1D and 2D Experiments Step-by-Step Tutorial

Basic Experiments User Guide

Version 004

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This manual was written by

Peter Ziegler

Billerica, Massachusetts, USA

P/N: B4472
DWG-Nr.: 004

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Contents

Introduction 1

General 1.1

This manual was written for AVANCE systems running TopSpin and should be used as a guide through the set up process for some experiments. The success of running the experiments in this manual is under the assumption that all parameters have been entered in to the prosol table.

Disclaimer 1.2

This guide should only be used for its intended purpose as described in this manual. Use of the manual for any purpose other than that for which it is intended is taken only at the users own risk and invalidates any and all manufacturer warranties.

Some parameter values, specially power levels suggested in this manual may not be suitable for all systems (e.g. Cryo probes) and could cause damage to the unit. Therefore only persons schooled in the operation of the AVANCE systems should operate the unit.

Warnings and Notes 1.3

There are two types of information notices used in this manual. These notices highlight important information or warn the user of a potentially dangerous situation. The following notices will have the same level of importance throughout this manual.

Note: Indicates important information or helpful hints

WARNING: Indicates the possibility of severe personal injury, loss of life or equipment damage if the instructions are not followed.

Contact for Additional Technical Assistance 1.4

For further technical assistance on the BPSU36-2 unit, please do not hesitate to contact your nearest BRUKER dealer or contact us directly at:

BRUKER BioSpin Corporation
19 Fortune Drive, Manning Park
Billerica, MA 01821
USA

Phone: (978) 667-9580
FAX: (978) 667-2955
Email: applab@bruker-biospin.com
Internet: www.bruker-biospin.com

1-D Basic Experiments

2

Sample preparation 2.1

- Use clean and dry sample tubes
- Use medium to high quality sample tubes
- Always filter the sample solution
- Always use the same sample volume or solution height
- 5 mm tubes 0.5 ml or 5 cm
- 10 mm tubes 4 ml or 5 cm
- Use the sample depth gauge to adjust the sample depth (1.8 cm for older style probes, 2.0 cm for newer style probes)
- The sample tube should sit tightly inside the spinner
- Turn on lift air to insert the sample into the magnet
- Wipe the sample tube clean before inserting into magnet

1-D Proton Experiment 2.2

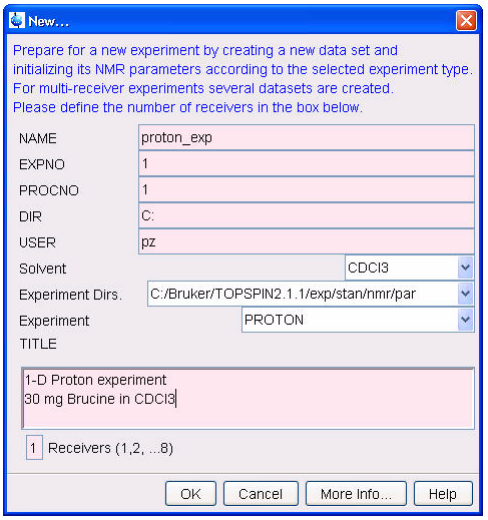
Sample:

30 mg Brucine in CDCl3

Experiment setup 2.2.1

1. Type **edc** and change the following parameters

Figure 2.1.



2. Click on OK
3. Insert the sample
4. Type **lock** and select CDCl3
5. Tune the probe
6. Shim for best homogeneity
7. Select the '**AcquPars**' tab by clicking on it
8. Click on  to read in the Prosol parameters

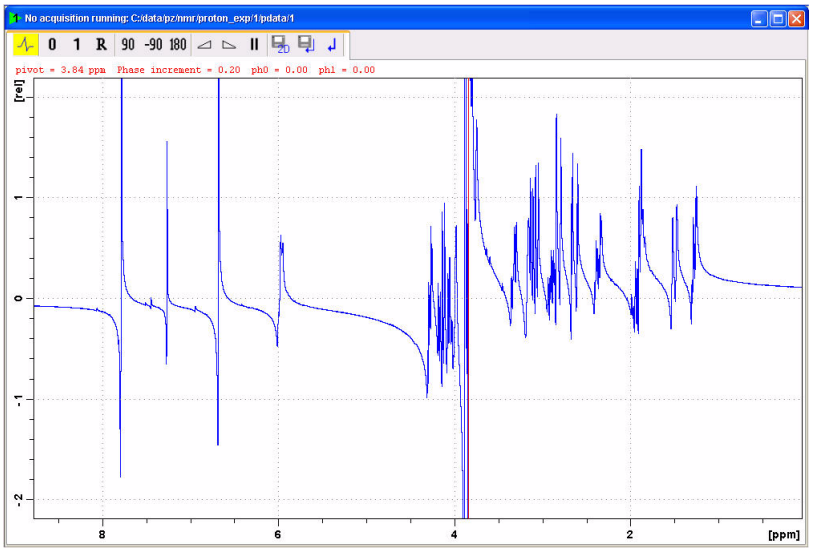
Acquisition *2.2.2*

1. Type **rga**
2. Type **zg** to start the acquisition

Processing *2.2.3*

1. Type **em** in the command line
2. Type **ft** in the command line
3. Expand the spectrum to include all peaks
4. Click on  for manually phasing the spectrum

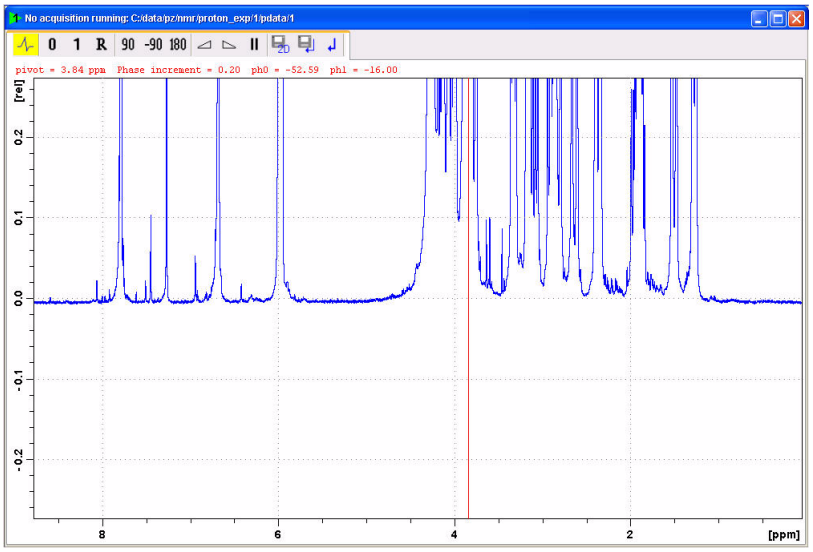
Figure 2.2.



5. Click on 0 and holding the left mouse button pressed move the mouse to adjust the zero order phase on the peak with the red cursor line (pivot point)

6. Click on 1 and holding the left mouse button pressed, move the mouse to adjust the first order phase on a peak distant from the pivot point

Figure 2.3.



NOTE: Increase the vertical scale for fine adjustment of the phase.

7. Click on  to store the phase values

8. Type **abs** for baseline correction

9. Expand the spectrum to include all peaks

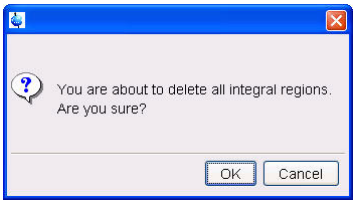
10. Click on 

NOTE: As part of the automatic baseline correction (abs), the spectrum is integrated using the default parameters: azfe, azfw and isen. For a user defined integration, follow the steps below.

11. In the integration menu bar click on  to select all regions

12. In the integration menu bar click on  to delete the selected regions

Figure 2.4.

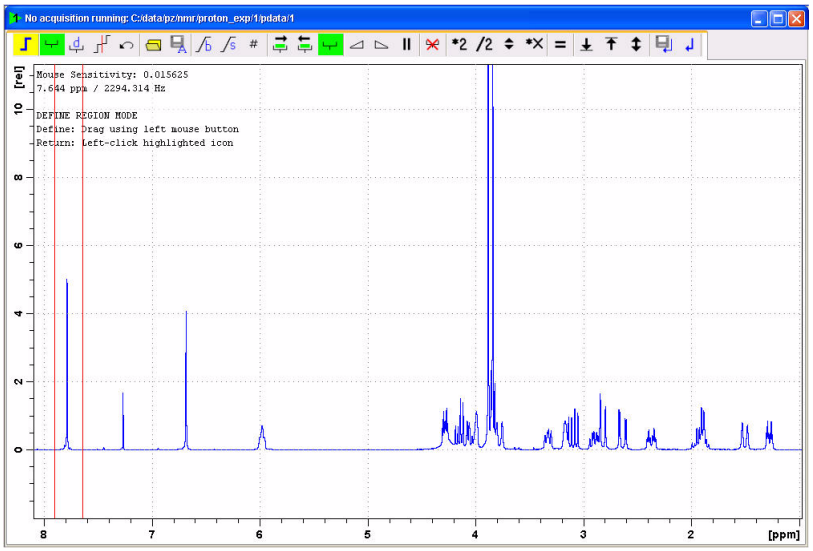


13. Click on 

14. In the Integration menu bar click on 

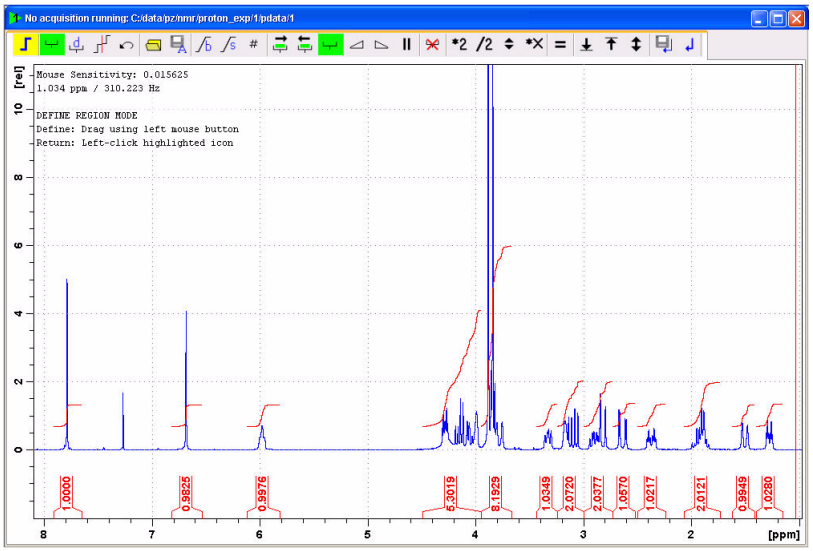
15. Set the cursor line, starting at the left of the spectrum, to the left of the first peak to be integrated, click the left mouse button and drag the cursor line to the right of the peak, then release the mouse button

Figure 2.5.



16. Repeat step15 for the remainder of the peaks

Figure 2.6.

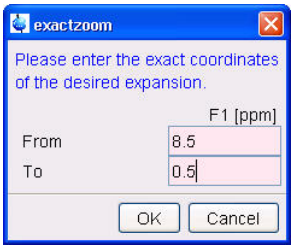


17. Click on  to save the integration region

Plotting the 1D Proton spectra *2.2.4*

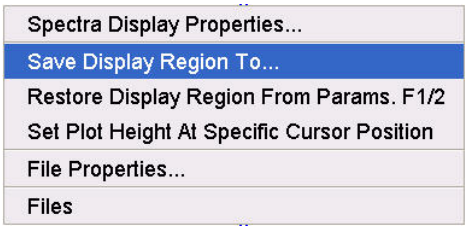
1. Expand the spectrum (all peaks in display)
2. Click on  to assign a exact expansion
3. Round off the F1 and f2 values to the nearest ppm

Figure 2.7.



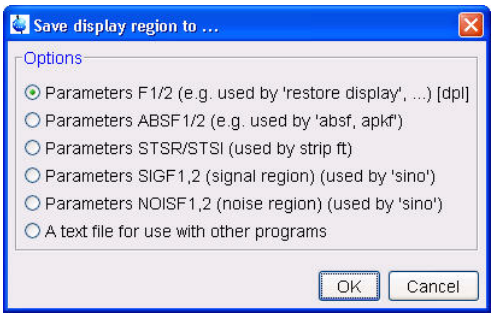
4. Click on 
5. Inside the spectrum window, click the right mouse button

Figure 2.8.



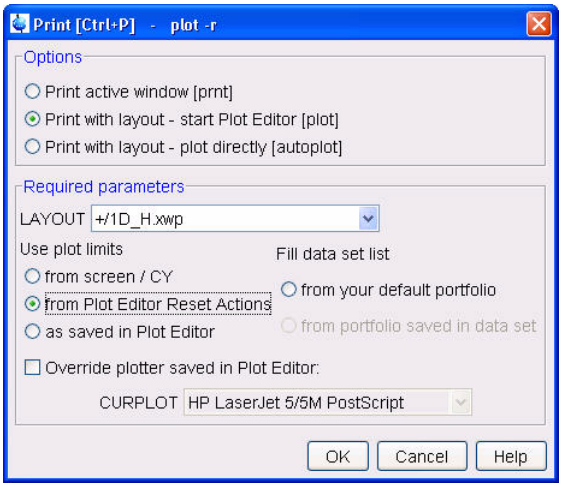
6. Select '**Save Display Region To**' by clicking on it

Figure 2.9.



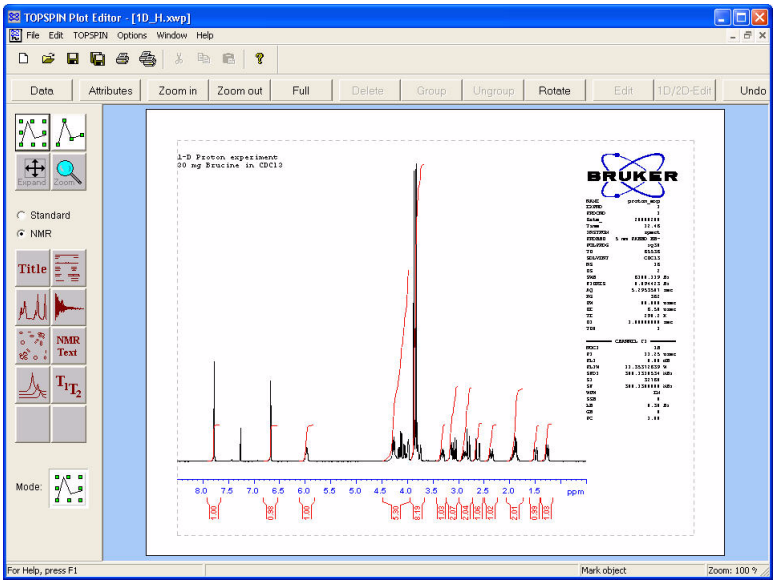
7. Enable '**Parameters F1/2(e.g. used by 'restore display')[dpl]**'
8. Click on OK
9. In the main menu click on '**File**' and select '**Print'** by clicking on it

Figure 2.10.



10. Enable '**Print with layout - start Plot Editor (plot)**'
11. Select the '**LAYOUT +/1D_H.xwp**'
12. Enable '**Plot Editor Reset Actions**'
13. Click on OK

Figure 2.11.



14. Click on '**File**' and select '**Print**' by clicking on it

1-D Carbon Experiment *2.3*

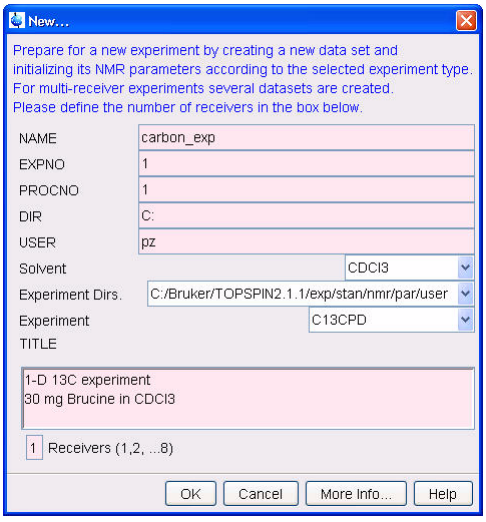
Sample:

30 mg Brucine in CDCl3

Experiment set up *2.3.1*

1. Type **edc** and change the following parameters

Figure 2.12.



2. Click on 

3. Insert the sample
4. Type **lock** and select CDCl3
5. Tune the probe
6. Shim for best homogeneity
7. Select the '**AcquPars**' tab by clicking on it
8. Make the following change

NS = **128**

9. Click on  to read in the Prosol parameters

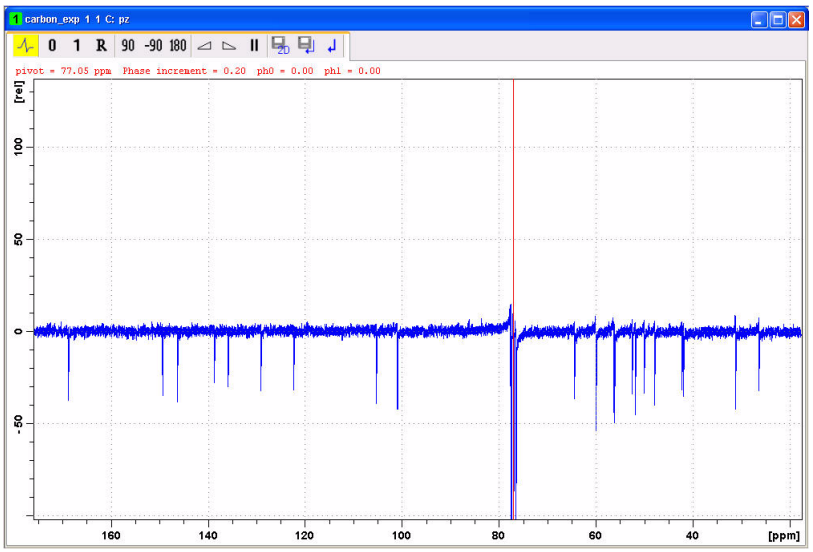
Acquisition *2.3.2*

1. Type **rga**
2. Type **zg** to start the acquisition

Processing *2.3.3*

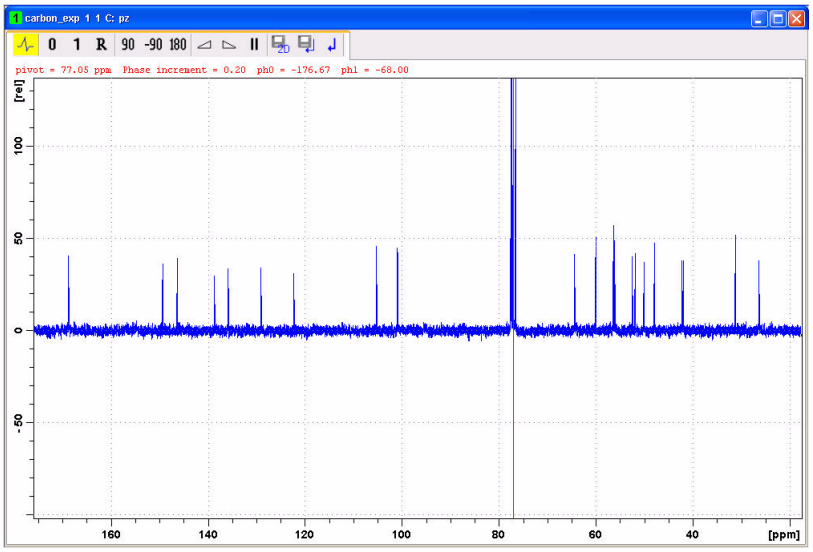
1. Type **em** in the command line
2. Type **ft** in the command line
3. Expand the spectrum to include all peaks
4. Click on  for manually phasing the spectrum

Figure 2.13.



5. Click on  and holding the left mouse button pressed move the mouse to adjust the zero order phase on the peak with the red cursor line (pivot point)
6. Click on  and holding the left mouse button pressed, move the mouse to adjust the first order phase on a peak distant from the pivot point

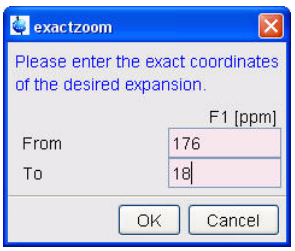
Figure 2.14.



NOTE: Increase the vertical scale for fine adjustment of the phase.

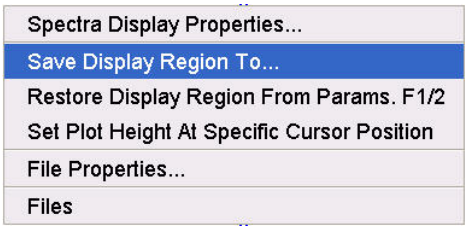
7. Click on  to store the phase values
8. Type **abs** for baseline correction
9. Expand the spectrum to include all peaks
10. Click on  to assign a exact expansion
11. Round off the F1 and f2 values to the nearest ppm

Figure 2.15.



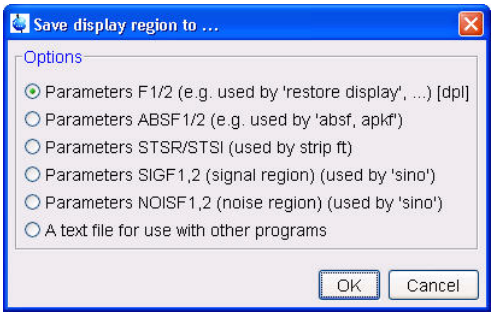
12. Click on 
13. Inside the spectrum window, click the right mouse button

Figure 2.16.



14. Select '**Save Display Region To**' by clicking on it

Figure 2.17.



15. Enable '**Parameters F1/2(e.g. used by 'restore display')[dpl]**'

16. Click on 

17. In the main menu click on '**Analysis**' and select '**Peak Picking[pp]**' by clicking on it

Figure 2.18.

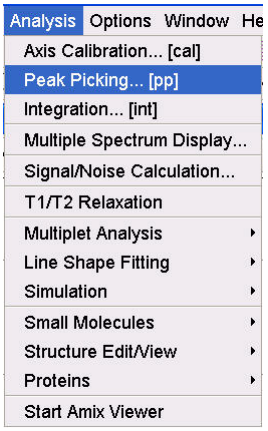
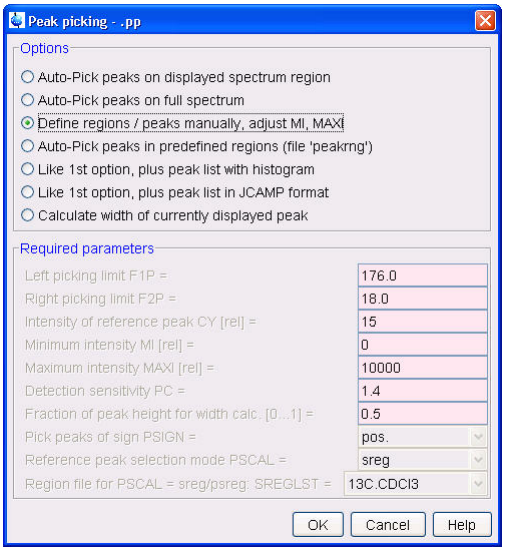


Figure 2.19.

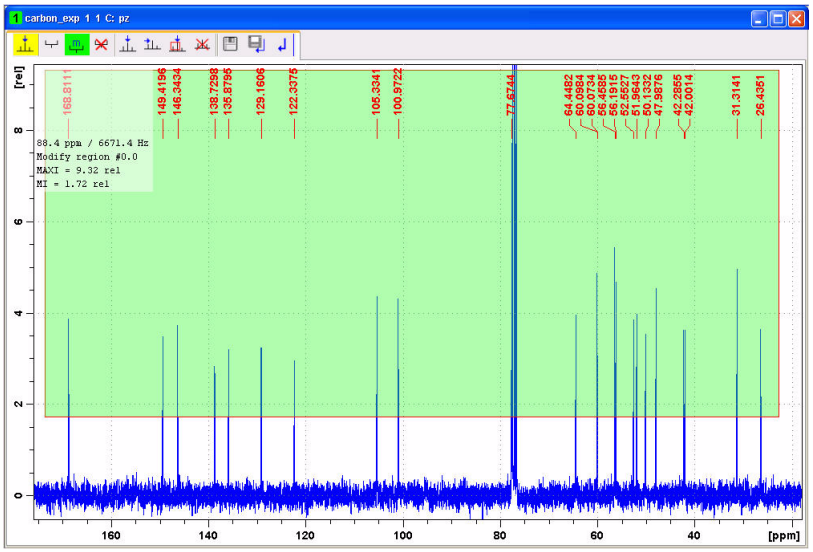


18. Enable '**Define regions / peaks manually, adjust MI, MAXI**'

19. Click on OK

20. Click the left mouse button and drag the cursor line from left to the right side of the spectrum

Figure 2.20. 1

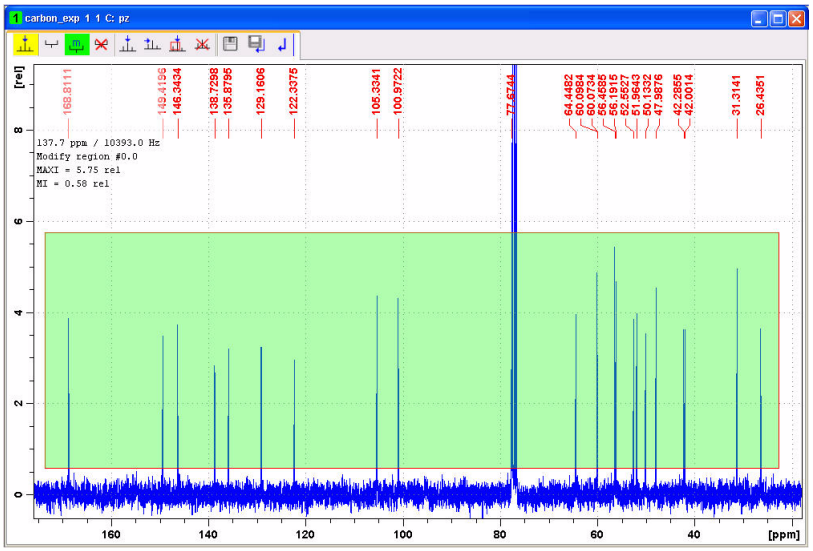


22. Click on  to manually adjust the minimum and maximum intensity levels

23. Click on the bottom line of the region box with the left mouse button and drag the line above the noise level, to set the minimum peak picking level

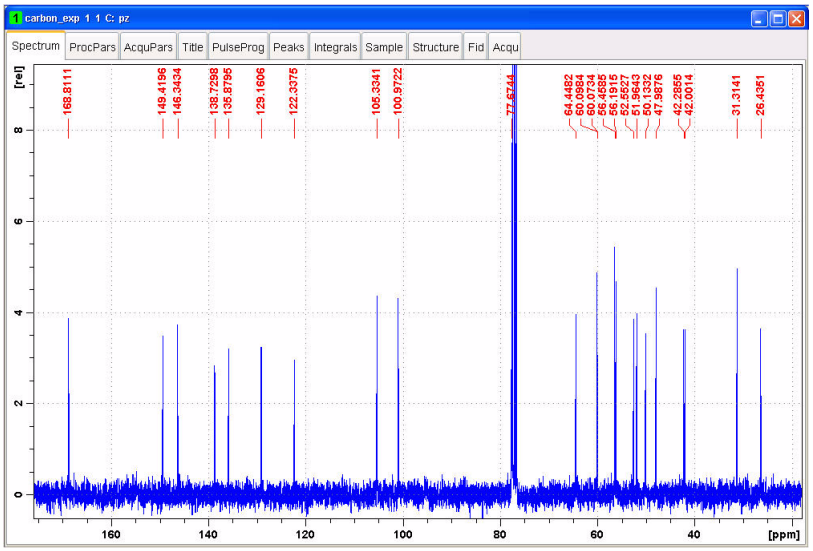
24. Click on the top line of the region box with the left mouse button and drag the line below unwanted peaks e.g. solvent peaks, to set the maximum peak picking level

Figure 2.21.



25. Click on  to store the peak picking values

Figure 2.22.

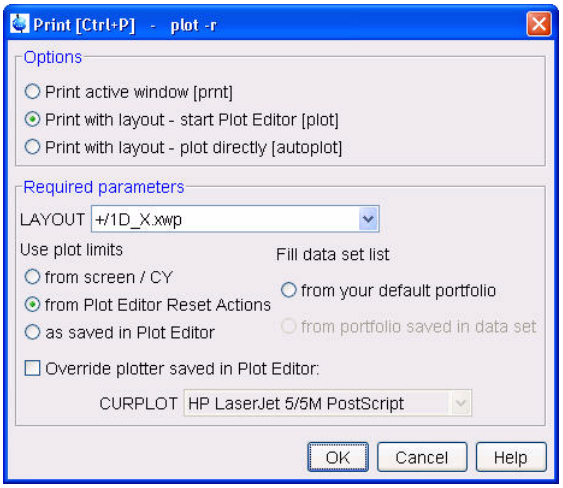


NOTE: To display the peak picking labels, right click inside the spectrum window and select ‘Display Properties’. Enable ‘Peak labels’ and click ‘OK’

Plotting the 1D Carbon spectrum *2.3.4*

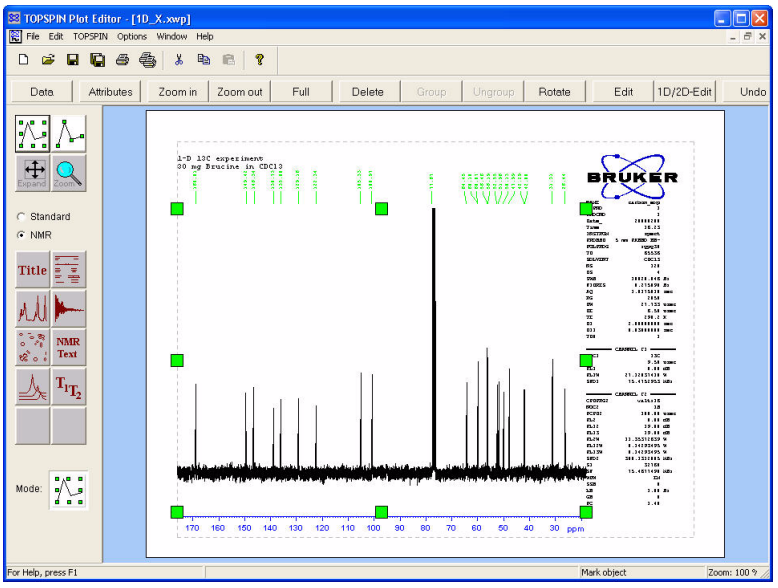
1. In the main menu click on '**File**' and select '**Print**' by clicking on it

Figure 2.23.



2. Enable '**Print with layout - start Plot Editor (plot)**'
3. Select '**LAYOUT +/1D_X.xwp**'
4. Enable '**Plot Editor Reset Actions**'
5. Click on OK

Figure 2.24.



6. Click on '**File**' and select '**Print**' by clicking on it

DEPT-135 Experiment 2.4

Sample:

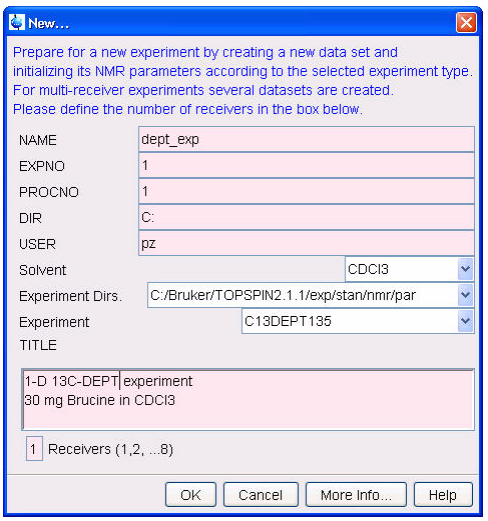
30 mg Brucine in CDCl3

Experiment set up 2.4.1

NOTE: This experiment usually follows a regular 1H decoupled 13C experiment. The result of a DEPT-135 experiment shows the CH and CH3 as positive and the CH2 as negative signals.

1. Type **edc** and change the following parameters

Figure 2.25.



2. Click on 

3. Insert the sample
4. Type **lock** and select CDCl3
5. Tune the probe
6. Shim for best homogeneity
7. Select the '**AcquPars**' tab by clicking on it
8. Make the following change

NS = **64**

9. Click on  to read in the Prosol parameters

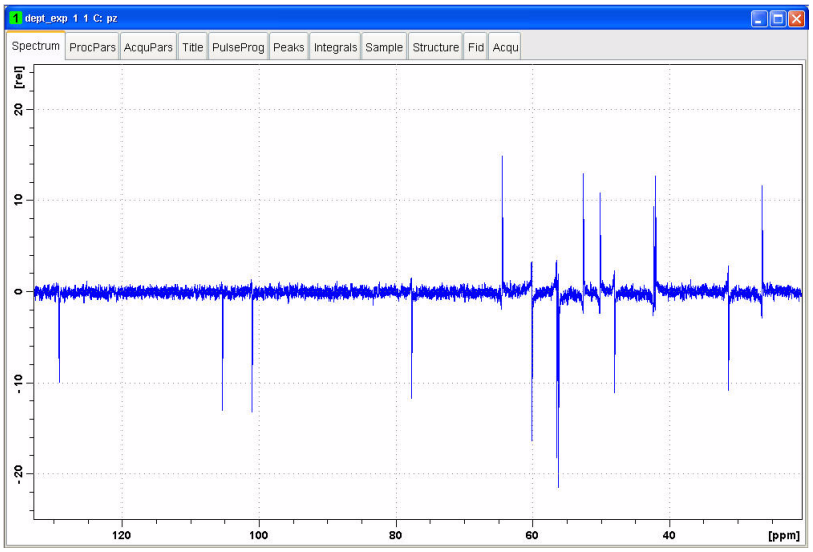
Acquisition 2.4.2

1. Type **rga**
2. Type **zg** to start the acquisition

Processing 2.4.3

1. Type **em** in the command line
2. Type **ft** in the command line

Figure 2.26. 3. Expand the spectrum to include all peaks



NOTE: Do to the fact that a DEPT135 spectrum contains negative and positive peaks, there is the possibility of getting phase results that are 180 degrees off. Instructions below assume that the left most line is a CH peak. This may not be the case, depending on the molecule under study.

4. Click on  for manually phasing the spectrum
5. Move the cursor line on to the left most peak
6. Click the right mouse button

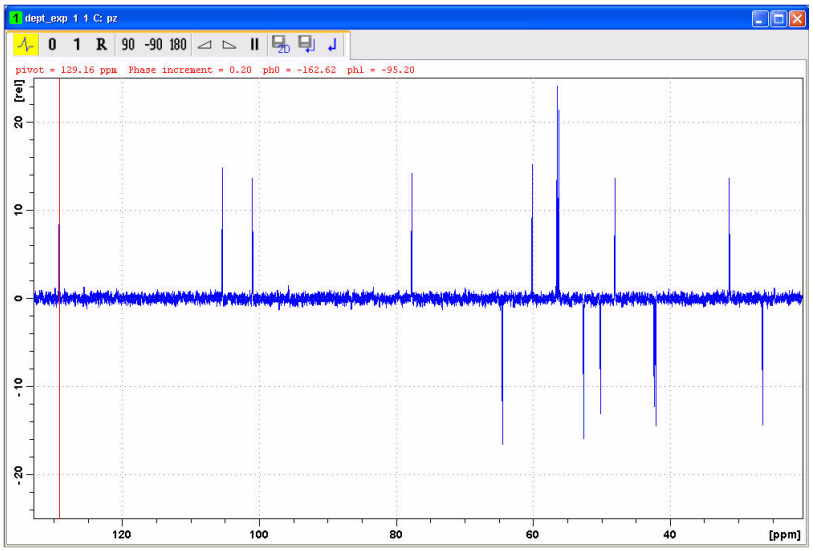
Figure 2.27.

7. Select '**Set Pivot Point**' by clicking on it

8. Click and hold on 0 with the left mouse button. Move the mouse up or down to adjust the zero order phase positive on the peak with the red cursor line (pivot point)

9. Click and hold on 1 with the left mouse button. Move the mouse up or down to adjust the first order phase on a peak distant from the pivot point taking in to consideration that this peak could be a CH2 resonance and therefor has to be phased negative

Figure 2.28.



NOTE: Increase the vertical scale for fine adjustment of the phase.

10. Click on  to store the phase values

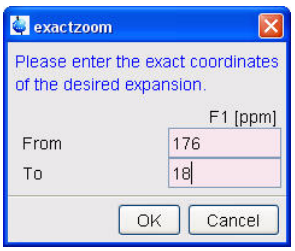
11. Type **abs** for baseline correction

12. Expand the spectrum to include all peaks

13. Click on  to assign a exact expansion

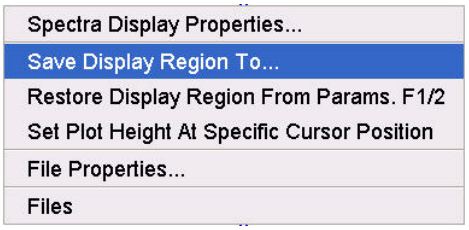
14. Round off the F1 and f2 values to the nearest ppm

Figure 2.29.



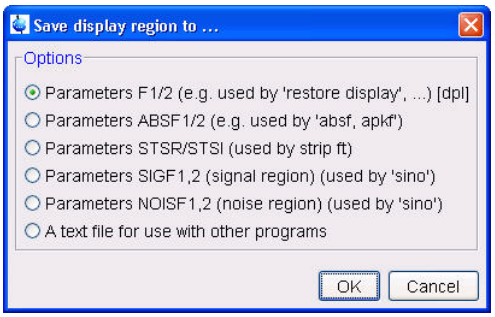
15. Click on OK
16. Inside the spectrum window, click the right mouse button

Figure 2.30.



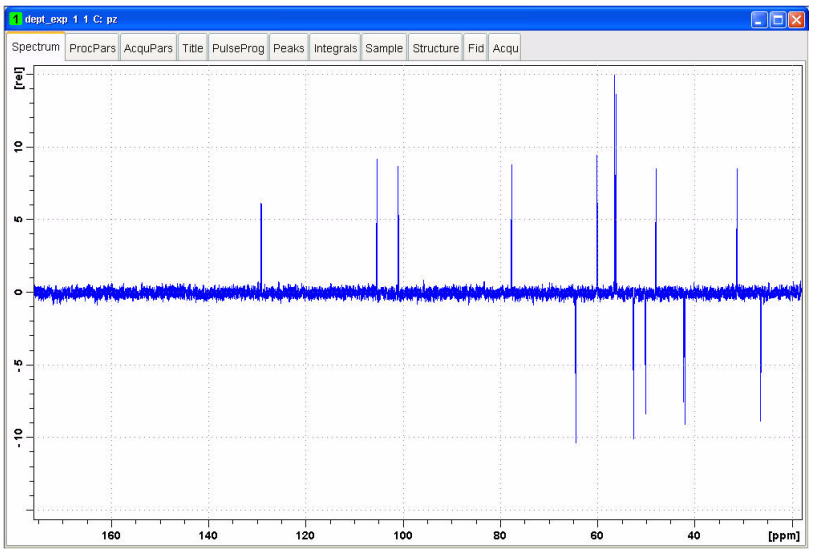
17. Select '**Save Display Region To**' by clicking on it

Figure 2.31.



18. Enable '**Parameters F1/2(e.g. used by 'restore display')[dpl]**'
19. Click on OK

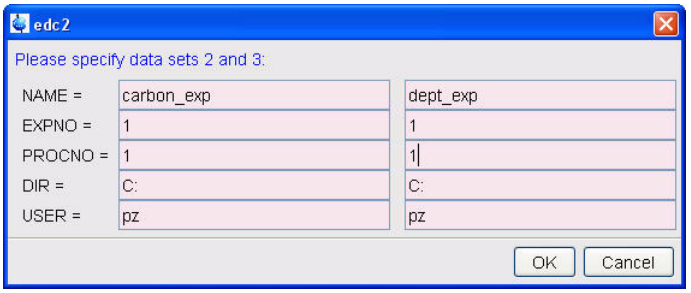
Figure 2.32.



Plotting the 1D Carbon and the DEPT135 spectra on the same page *2.4.4*

1. Type **edc2** on the command line

Figure 2.33.



2. Enter the EXPNO and PROCNO of the 1D 13C spectrum in to the first column of the edc2 window

EXPNO = **1**

PROCNO = **1**

3. Enter the EXPNO and PROCNO of the 1D DEPT135 spectrum in to the second column of the edc2 window

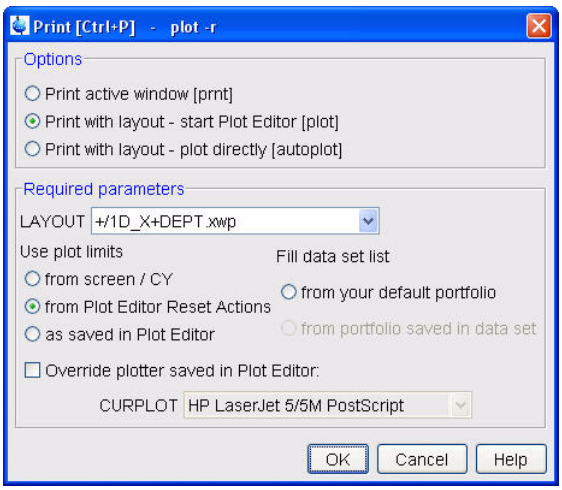
EXPNO = **2**

PROCNO = **1**

4. Click on OK

4. In the main menu click on '**File**' and select '**Print**' by clicking on it

Figure 2.34.



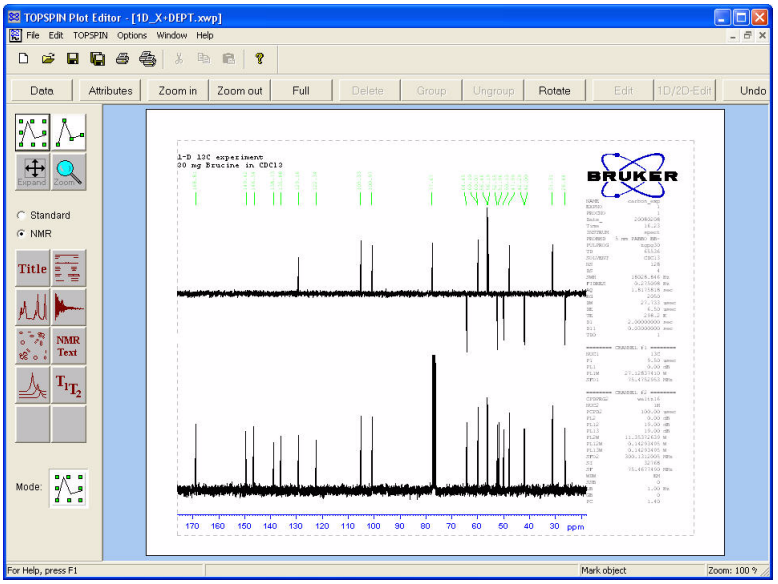
5. Enable '**Print with layout - start Plot Editor (plot)**'

6. Select the LAYOUT **+/1D_X+DEPT.xwp**'

7. Enable '**from Plot Editor Reset Actions**'

8. Click on OK

Figure 2.35.


TOPSPIN Plot Editor - [1D_X+DEPT.xwp]
File Edit TOPSPIN Options Window Help
Data Attributes Zoom in Zoom out Full Delete Group Ungroup Rotate Edit 1D/2D-Edit Undo
Expand Zoom
Standard
NMR
Title
NMR Text
Mode:
1-D 13C experiment
30 mg Brucine in CDCl3
BRUKER
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 ppm
For Help, press F1
Mark object
Zoom: 100 %

1-D NOE Difference Experiment

3

Introduction 3.1

The experiment in this chapter uses one frequency list and one presaturation power level. The data are collected using the noediff AU-program.

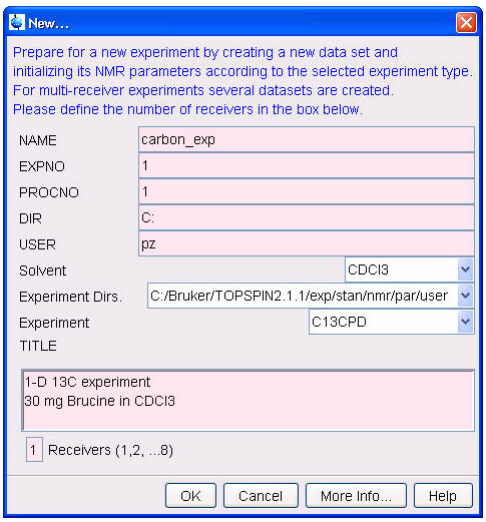
Sample:

40 mg Pamoic acid in DMSOd6

Preparation experiment 3.1.1

1. Type **edc** and change the following parameters

Figure 3.1.



2. Click on 

3. Insert the sample

4. Type **lock** and select DMSO
5. Turn the spinner off

NOTE: noe experiments should be run non spinning

6. Tune the probe
7. Shim for best homogeneity
8. Select the '**AcquPars**' tab by clicking on it
9. Make the following change

O2p [ppm] = **-4**

10. Click on  to read in the Prosol parameters
11. Select the '**ProcPars**' tab by clicking on it
12. Make the following change:

LB [Hz] = **1**

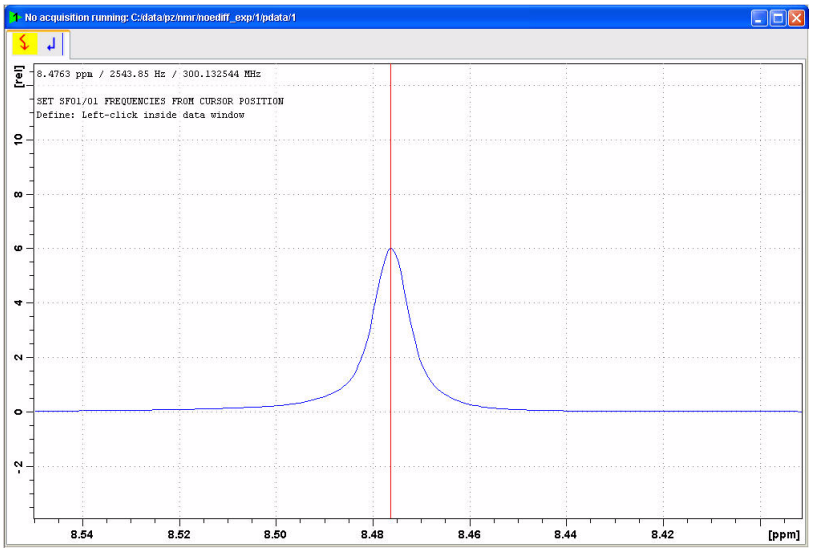
13. Type **rga**
14. Type **zg** to start the acquisition
15. Process and Phase correct the spectrum

Frequency list set up *3.1.2*

NOTE: Steps 1 through 5 are necessary to determine the correct power level (pl14) for presaturating the irradiation peak

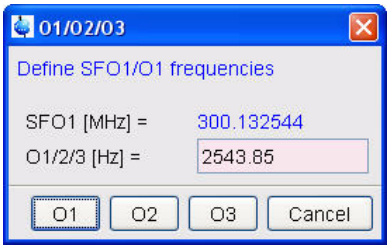
1. Expand the peak around 8.5 ppm
2. Click on 

Figure 3.2.



4. Move the cursor line to the center of the peak and click the left mouse button

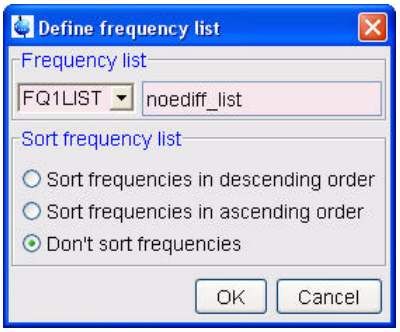
Figure 3.3.



5. Click on 

6. Click on 

Figure 3.4.



7. Select '**FQ1LIST**' and type a frequency list name (e.g. **noediff_list**)

8. Enable '**Don't sort frequencies**'

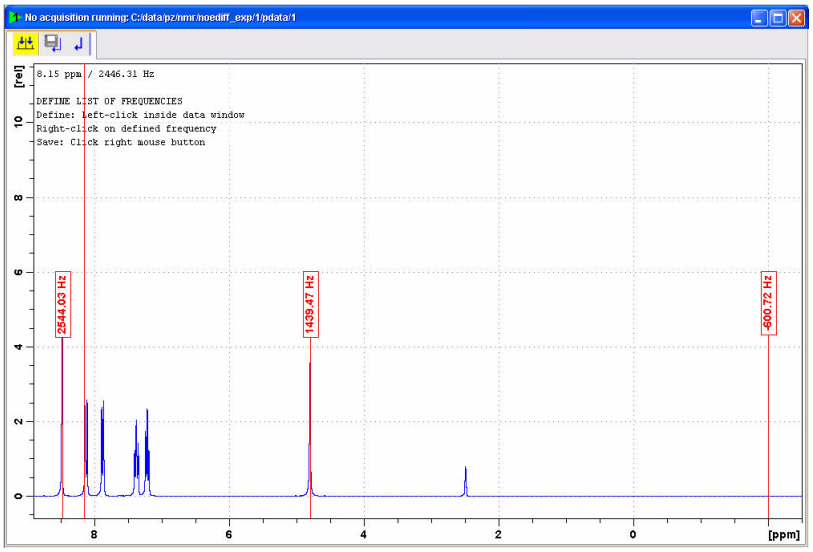
9. Click on 

10. Move the cursor line to –2ppm and click the left mouse button to assign the off resonance frequency

11. Expand the peak at 4.8 ppm

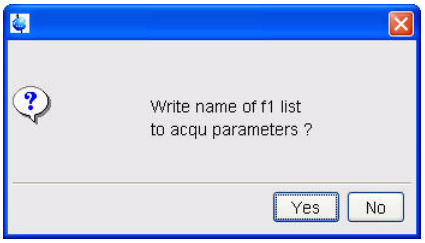
12. Move the cursor line to the center of the peak and click the left mouse button
13. Repeat steps11 through 12 to assign the frequency for the peak at 8.5ppm

Figure 3.5.



14. Click on  to save the frequency list

Figure 3.6.



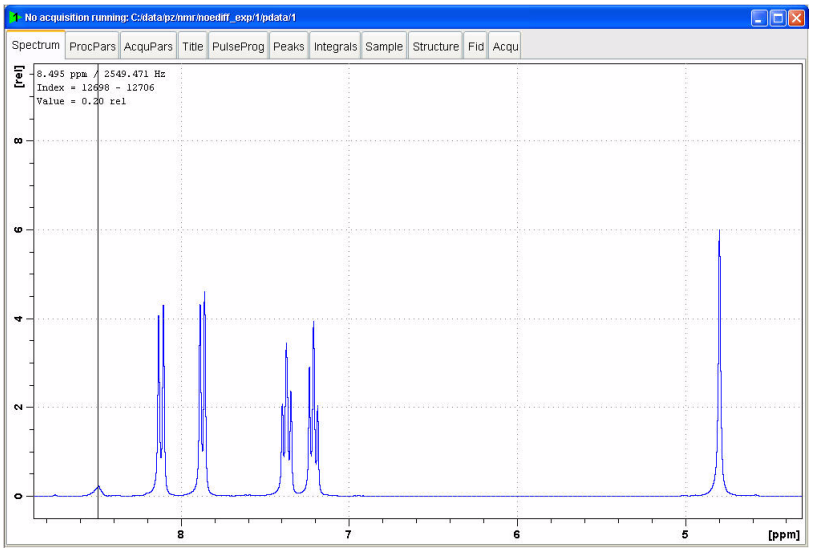
15. Click on 

Fine tuning *3.1.3*

1. Type **zg** to start the acquisition
2. Process and Phase correct the spectrum

NOTE: The irradiated signal at ~8.5 ppm (O2p = 8.5 ppm) should be almost completely suppressed as shown below. If necessary adjust pl14 and repeat steps 1 and 2 to optimize the suppression.

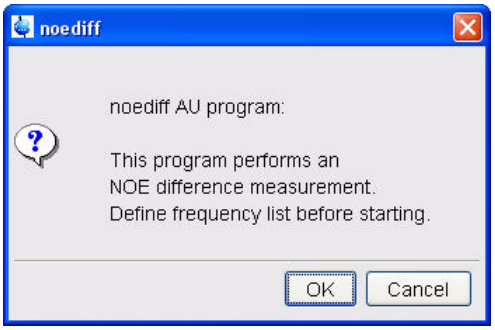
Figure 3.7.



Running the experiment 3.1.4

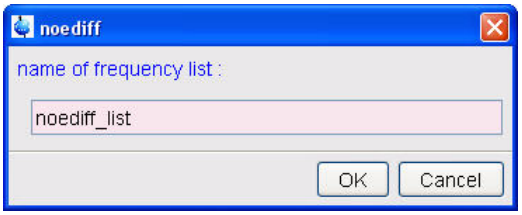
1. Type **noediff** on the command line

Figure 3.8.



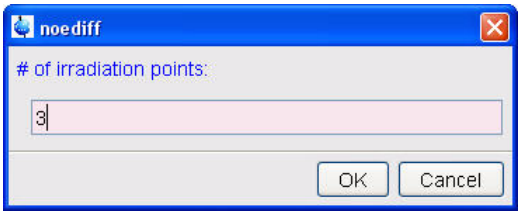
2. Click on OK

Figure 3.9.



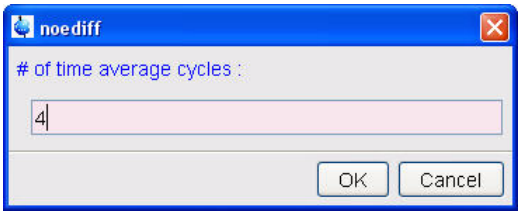
3. Click on OK

Figure 3.10.



4. Click on OK

Figure 3.11.

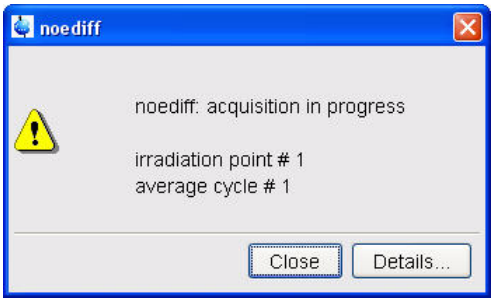


5 Change the # of time average cycles = **4**

NOTE: The experiment creates three data sets, one for each irradiation point in the list. It starts at the first irradiation and completes 8 scans for all the irradiation frequencies and then it loops through all three experiments 4 times for a total of 32 scans on each experiment.

6 Click on OK

Figure 3.12.



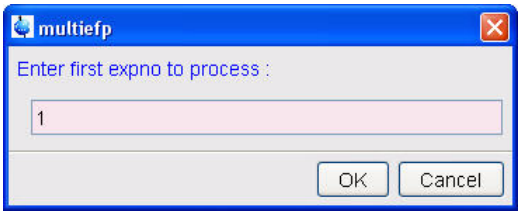
Processing *3.1.5*

1.Start with experiment # 1

2. Type **ef**

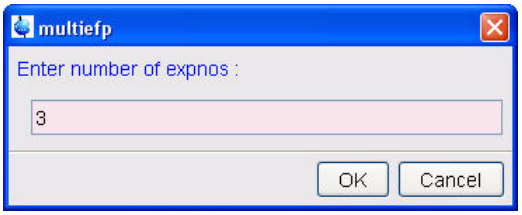
3. Correct the phase very carefully
4. Type **multiefp**

Figure 3.13.



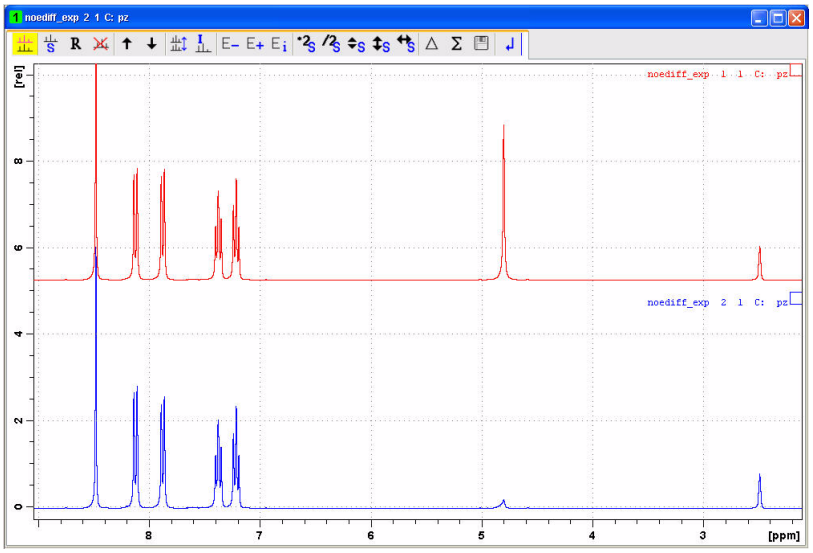
5. Enter **1** for the first experiment number
6. Click on OK

Figure 3.14.



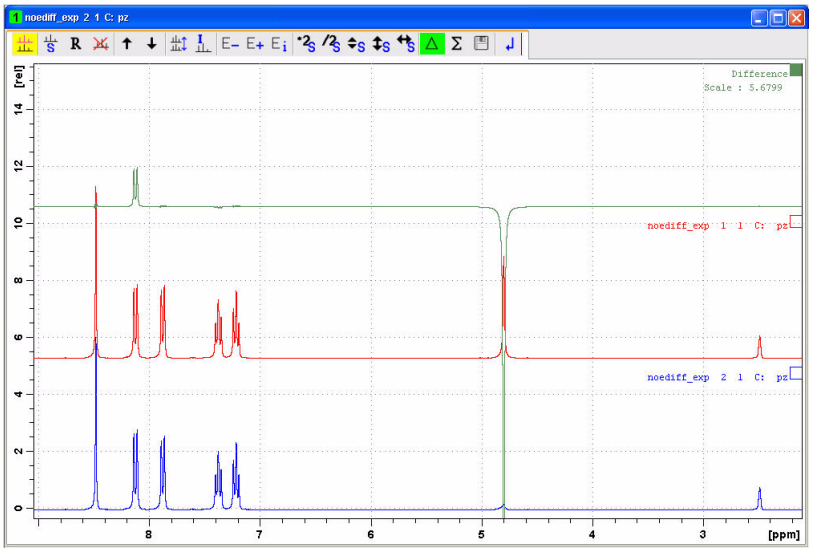
7. Enter **3** for the # of experiments
8. Click on OK
9. Drag experiment # 2 into the display window or type **re 2** in the command line
10. Click on 
11. Drag experiment # 1 into the display window or type **re 1** in the command line

Figure 3.15.



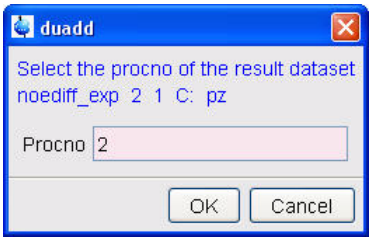
12. Click on 

Figure 3.16.



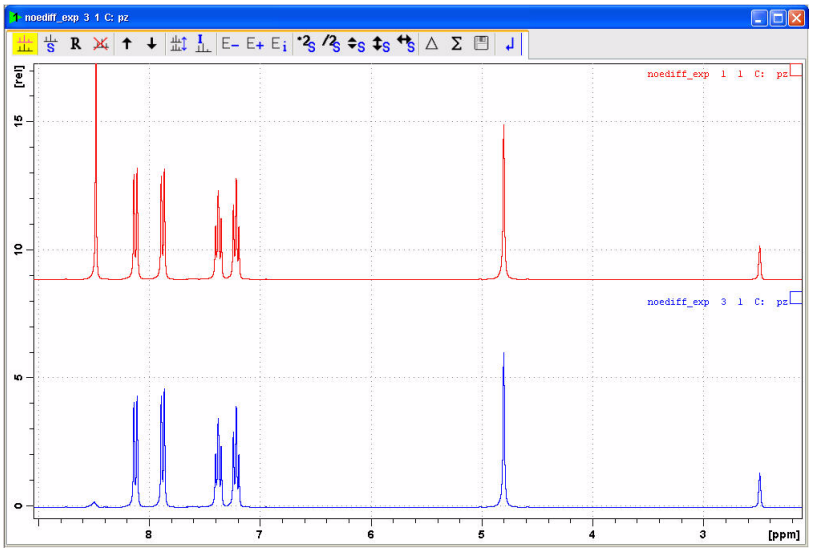
13. Click on 

Figure 3.17.



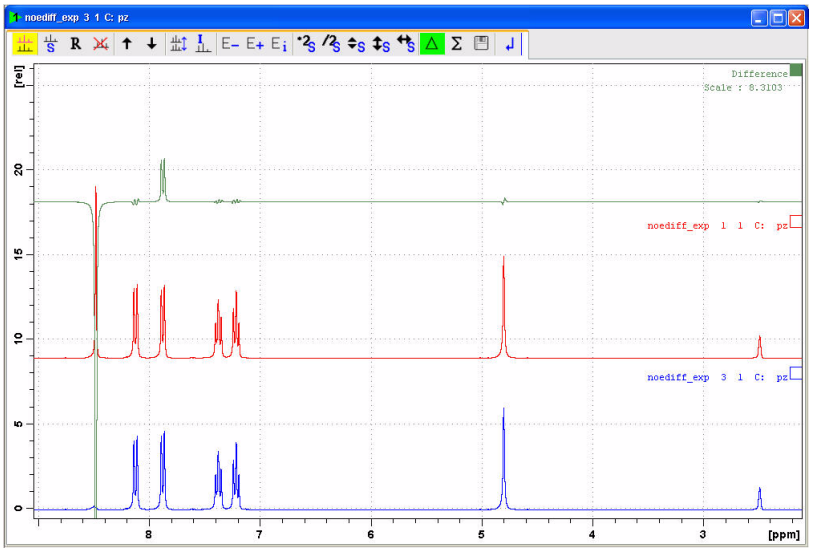
14. Enter **2** for the processing #
15. Click on 
16. Click on 
17. Drag experiment # 3 into the display window or type **re 3** in the command line
18. Click on 
19. Drag experiment # 1 into the display window or type **re 1** in the command line

Figure 3.18.



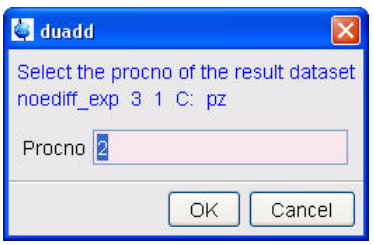
20. Click on 

Figure 3.19.



21. Click on 

Figure 3.20.



22. Enter **2** for the Procno

23. Click on 

24. Click on 

Integration *3.1.6*

1. Drag experiment # 2 processing # 2 into the display window or type **re 2 2** in the command line

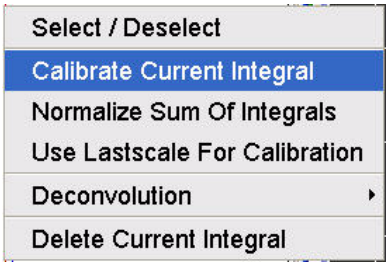
2. Click on 

3. Define the regions by clicking the left mouse button and the use of the cursor lines

4. move the cursor line in to the region of the negative peak

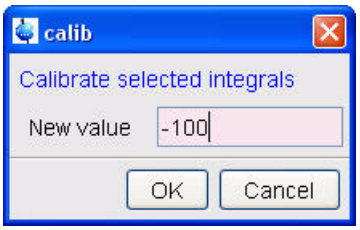
5. Click the right mouse button

Figure 3.21.



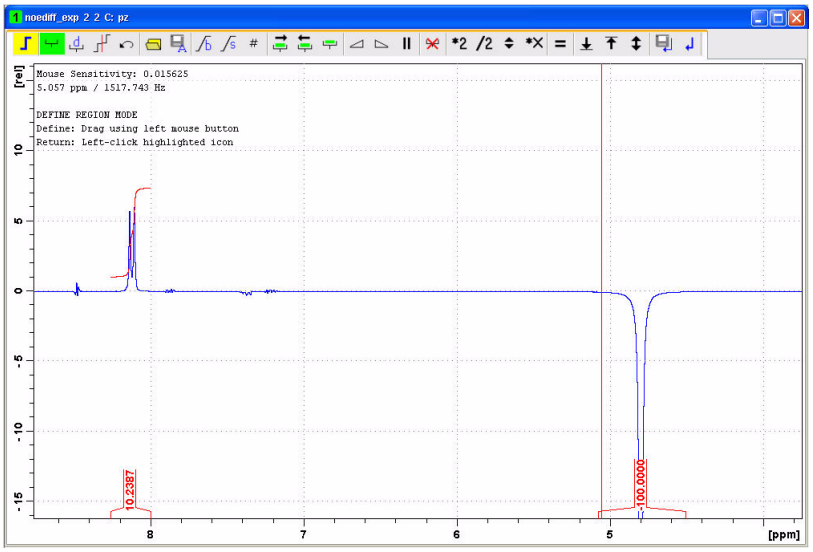
6. Select '**Calibrate Current Integral**'

Figure 3.22.



7. Change the value to **-100**

Figure 3.23.

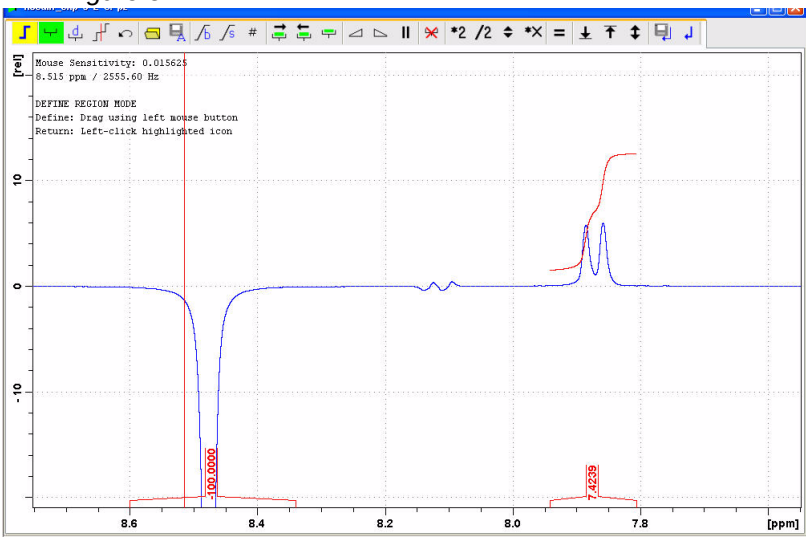


8. Click on 

9. Drag experiment # 3 processing # 2 into the display window or type **re 3 2** in the command line

10. Repeat steps 2 through 8

Figure 3.24.



11. Click on 

Solvent Suppression Experiments

4

Introduction 4.1

The following solvent suppression technics are discussed in this chapter. For single peak suppression: Presaturation, Presaturation with composite pulses, WATERGATE, Excitation Sculpting. For multiple peak suppression: WET.

Solvent suppression with Presaturation 4.2

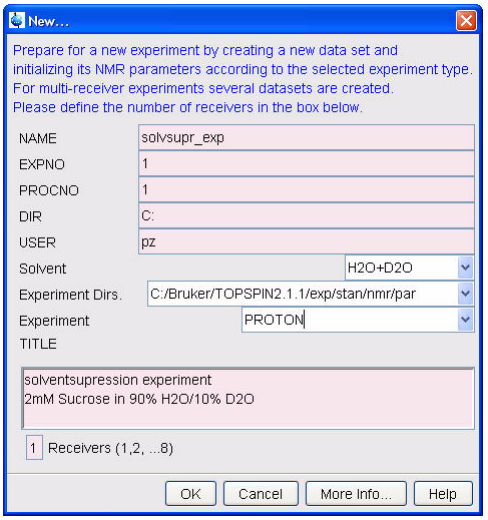
Sample:

2 mM Sucrose in 90% H2O / 10% D2O

Reference spectrum 4.2.1

1. Type **edc** and change the following parameters

Figure 4.1.



2. Click on OK
3. Insert the sample
4. Type **lock** and select H2O+D2O
5. Turn the spinner off

NOTE: Solvent suppression experiments should be run non spinning

6. Tune the probe
7. Shim for best homogeneity
8. Select the '**AcquPars**' tab by clicking on it
9. Make the following change

NS = **8**

10. Click on  to read in the Prosol parameters
12. Select the '**Spectrum**' tab by clicking on it

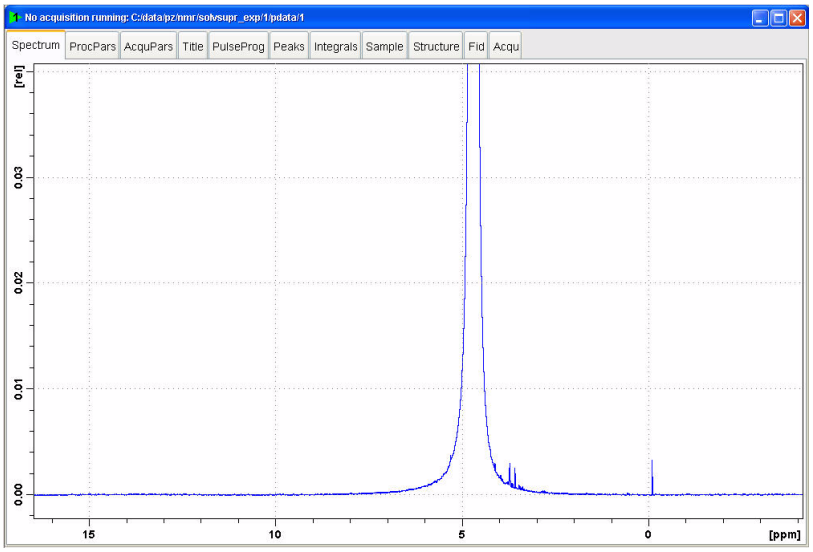
Acquisition *4.2.2*

1. Type **rga**
2. Type **zg** to start the acquisition

Processing 4.2.3

1. Process and phase correct the spectrum

Figure 4.2.

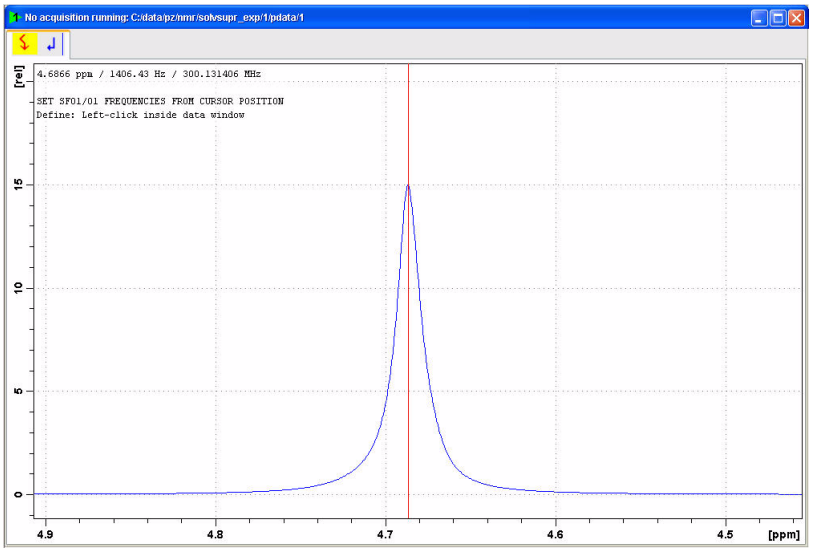


NOTE: Make sure that the SW is large enough to cover the entire Spectrum accounting for the position of O1. The presaturation is applied on resonance (at the O1 position) The power level for presaturation has to be known and entered into the Prosol parameters.

Parameter set up 4.2.4

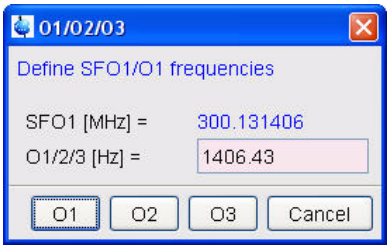
1. Type **wrpa 2** on the command line
2. Type **re 2** on the command line
3. Expand the Water signal at 4.8 ppm
4. Click on 

Figure 4.3.



5. Move the cursor line to the center of the peak and click the left mouse button

Figure 4.4.



5. Click on 

7. Select the '**AcquPars**' tab by clicking on it

8. Make the following changes:

PULPROG = **zgpr**

TD = **32k**

NS = **8**

DS = **4**

9. Click on  to display the pulsprogram parameters

10. Make the following changes:

D1 [s] = **2**

11. Select the '**ProcPar**' tab by clicking on it

12. Make the following changes:

SI = **16k**

13. Select the '**Spectrum**' tab by clicking on it

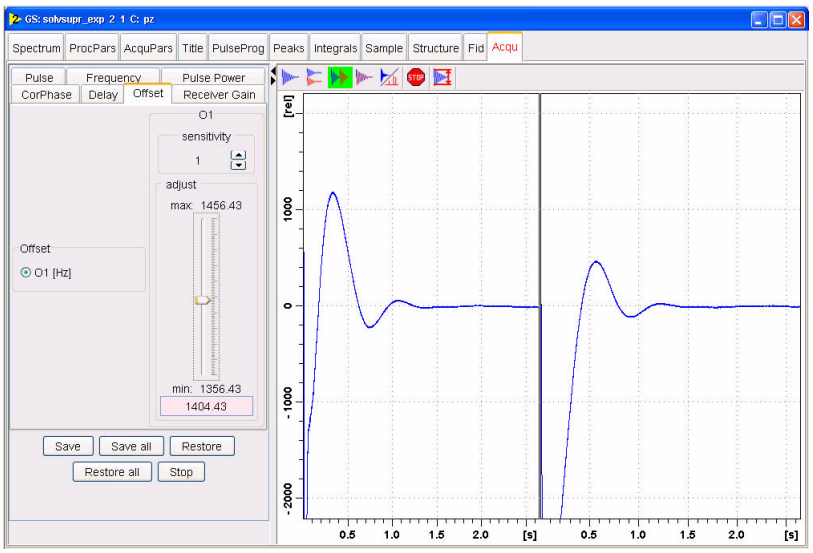
1. In the main menu click on '**Spectrometer**', select '**Adjustments**' and click on '**Auto-adjust receiver gain**' or type **rga**

2. Click on '**Spectrometer**' in the main menu bar, select '**Adjustments**' and click on '**Start acquisition, adjust params [gs]**' or type **gs**

3. click on 

4. Select the '**Offset**' tab

Figure 4.5.

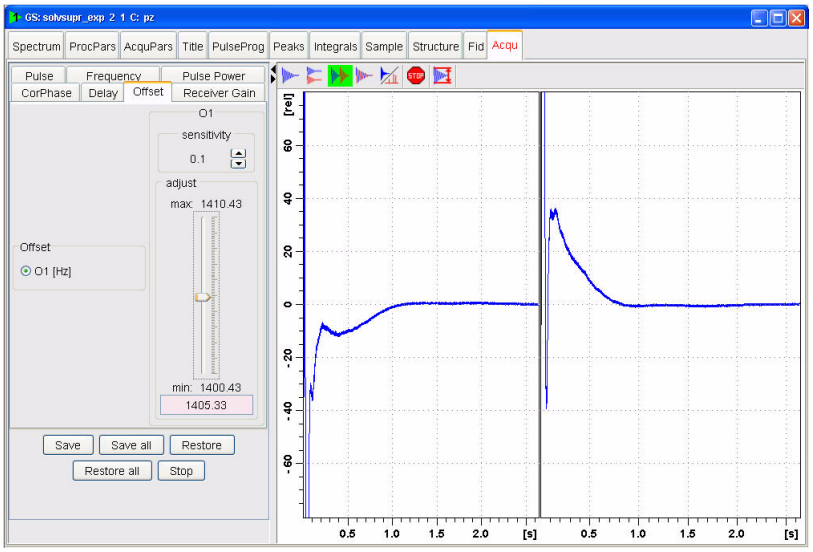


5. Change the O1 value by clicking just below or above the adjust slider

NOTE: for smaller changes, adjust the 'sensitivity' to smaller values.

6. Observe the fid area in the Acquisition information window for a smaller integration value and the FID to become a single line

Figure 4.6.



7. Click on Save
8. Click on Stop

Figure 4.7.

9. Click on OK

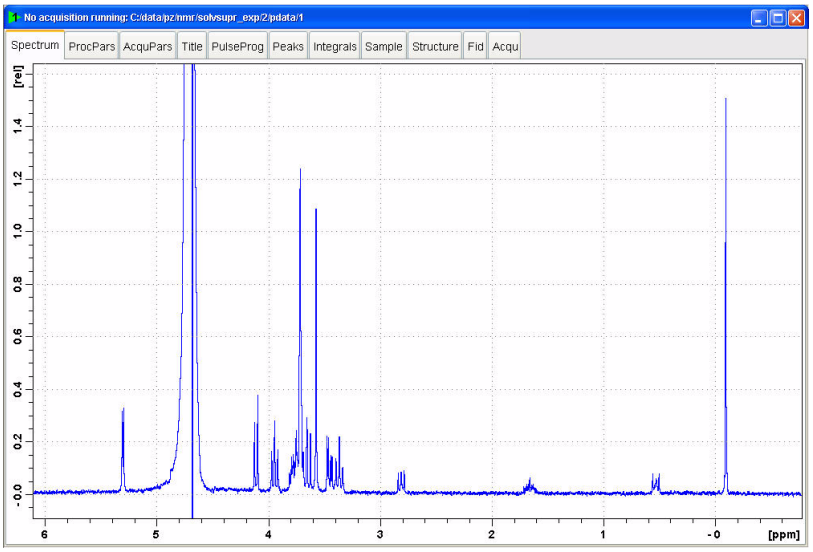
Acquisition *4.2.6*

1. Type **rga**
2. Type **zg** to start the acquisition

Processing *4.2.7*

1. Process and phase correct the spectrum

Figure 4.8.



Solvent suppression with Presaturation using Composite Pulses *4.3*

Parameter set up *4.3.1*

1. Follow the instructions in paragraph 4.2.1 through 4.2.5 step 9 in this chapter
2. Select the '**AcquPars**' tab by clicking on it
3. Make the following changes:

PULPROG = **zgcppr**

4. Select the '**Spectrum**' tab by clicking on it

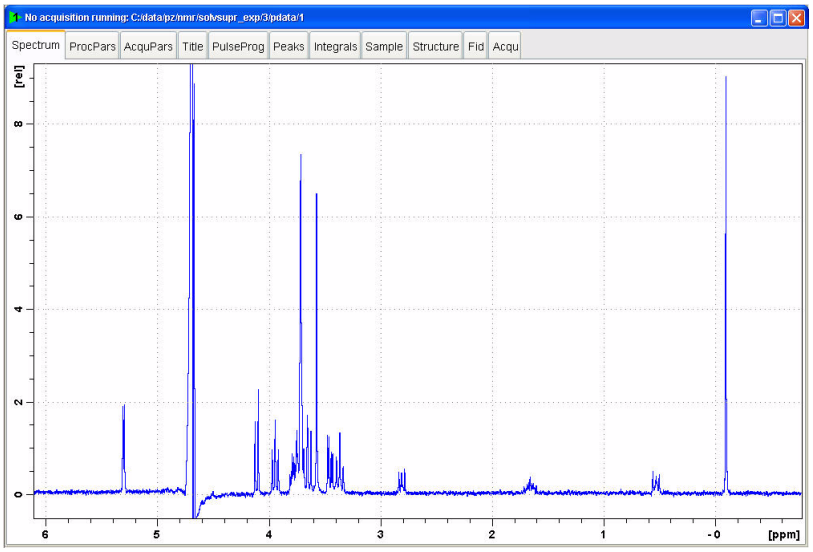
Acquisition *4.3.2*

1. Type **rga**
2. type **zg** to start the acquisition

Processing *4.3.3*

1. Process and phase correct the spectrum

Figure 4.9.



Solvent suppression with WATERGATE 4.4

Parameter set up 4.4.1

1. Follow the instructions in the paragraphs 4.2.1 through 4.2.5 step 9
2. Select the '**AcquPars**' tab by clicking on it
3. Make the following change

PULPROG = **p3919gp**

4. Click on  to display the pulsprogram parameters
5. Make the following changes:

D19 [s] = **0.00015**

GPZ1 [%] = **20**

6. Select the '**Spectrum**' tab by clicking on it

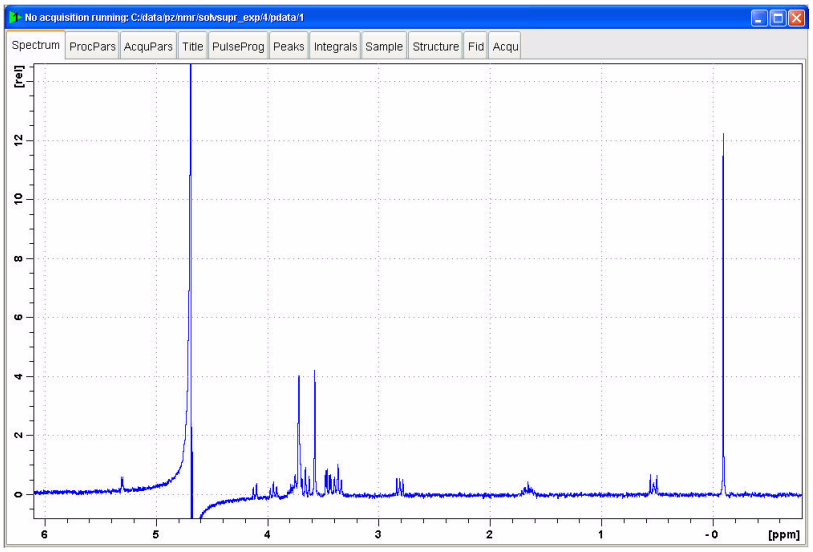
Acquisition 4.4.2

1. Type **rga**
2. Type **zg** to start the acquisition or

Processing 4.4.3

1. Process and phase correct the spectrum

Figure 4.10.



Solvent suppression with excitation sculpting *4.5*

Parameter se up *4.5.1*

1. Follow the instructions in the paragraphs 4.2.1 through 4.2.5 step 9
2. Select the '**AcquPars**' tab by clicking on it
3. Make the following changes:

PULPROG = **zgesgp**

4. Click on  to display the pulsprogram parameters
5. Make the following changes:

P12 [us] = **0.002**

SP! [dB] = calculate using the AU-program 'pulse' (e.g.**45**)

SPNAM1 = **Squa100.1000**

GPZ1 [%] = **31**

GPZ2 [%] = **11**

6. Select the '**Spectrum**' tab by clicking on it

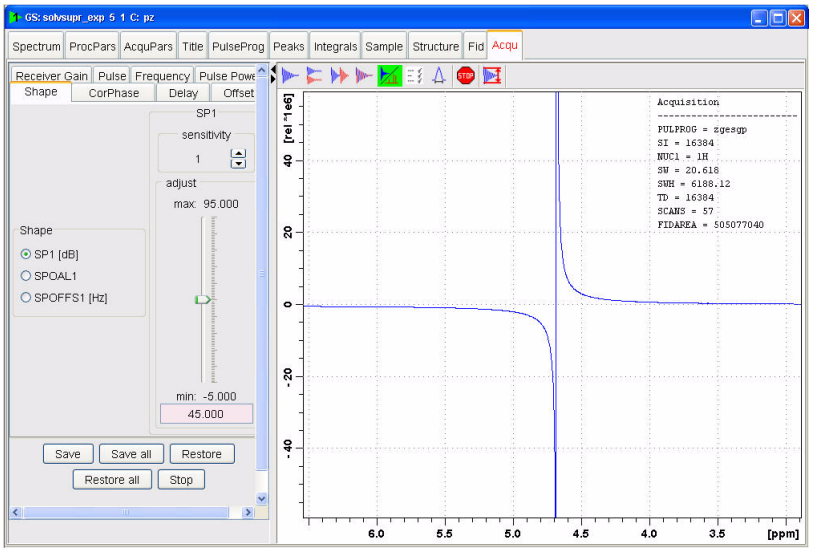
Fine Tuning *4.5.2*

1 Type **rga**

2. Type **gs**
3. Select the '**Shape**' tab
4. Enable '**SP1[dB]**'

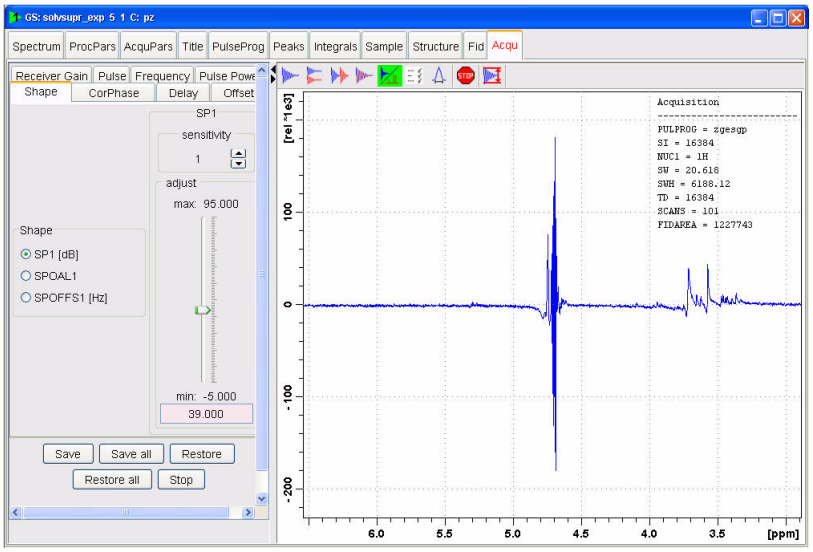
5. Click on 

Figure 4.11.



6. Move the slider to change the SP1 value for better solvent suppression

Figure 4.12.



7. Click on 

8. Click on 

Figure 4.13.

9. Click on OK

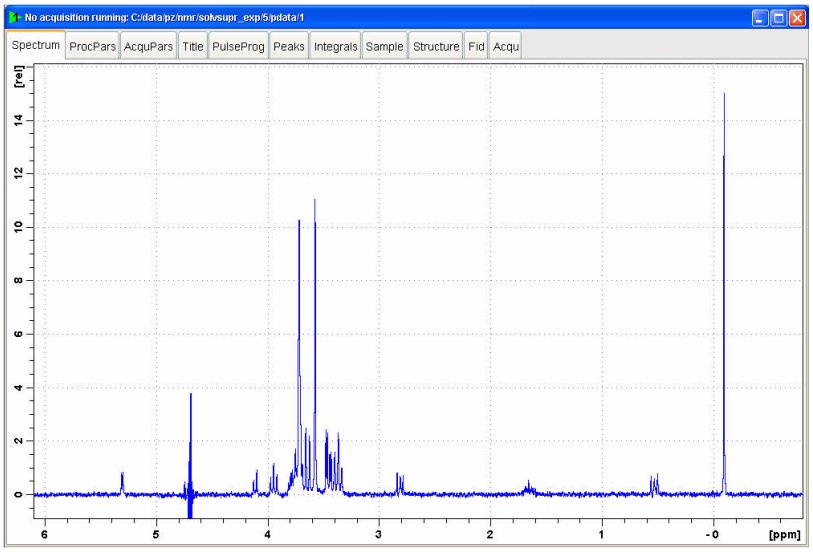
Acquisition 4.5.3

1. Type **rga**
2. Type **zg** to start the acquisition or

Processing 4.5.4

1. Process and phase correct the spectrum

Figure 4.14.



Solvent suppression with WET 4.6

Introduction 4.6.1

NOTE: To run this experiment, the WET standard pulse and power level for PSH3 have to be entered in to the prosol table. If there is no entry for PSH3, follow the steps below.

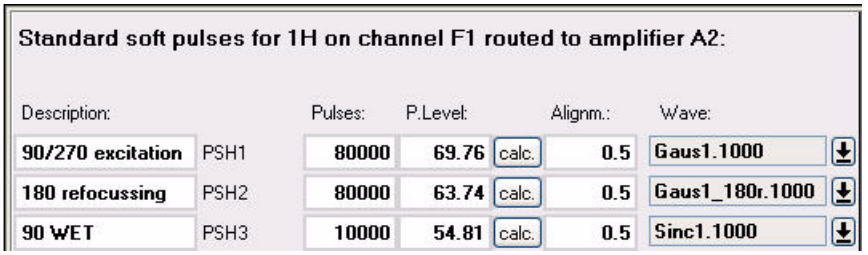
1. Type **edprosol** in the command line

2. Select Nucleus '**1H**'
3. Click on '**Standard Soft pulses**'
4. Enter the following parameters"

90 WET	pulses (PSH3)	=	**10'000**
	wave	=	**Sinc1.1000**

5. Click on calc. to calculate the 90 WET power level

Figure 4.15.



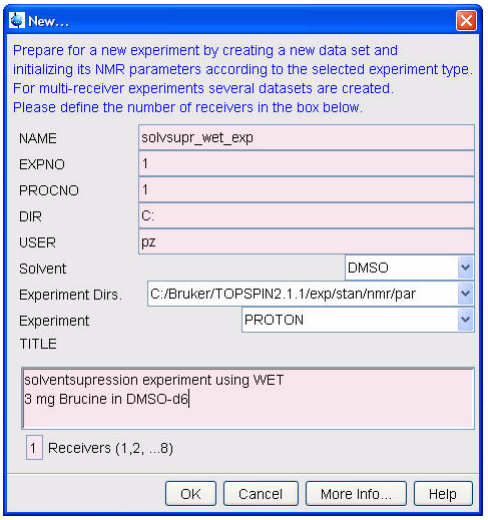
Sample:

3mg Brucine in DMSO d6

Reference spectrum *4.6.2*

1. Type **edc** and change the following parameters

Figure 4.16.



2. Click on OK
3. Insert the sample
4. Type **lock** and select H2O+D2O

5. Turn the spinner off

NOTE: solvent suppression experiments should be run non spinning

6. Tune the probe
7. Shim for best homogeneity
8. Select the '**AcquPars**' tab by clicking on it
9. Click on  to read in the Prosol parameters
10. Select the '**Spectrum**' tab by clicking on it

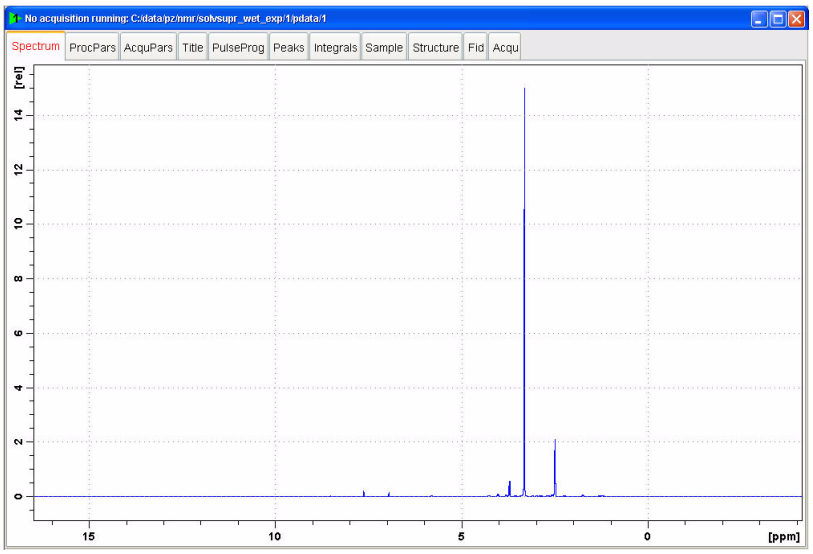
Acquisition *4.6.3*

1. Type **rga**
2. Type **zg** to start the acquisition or

Processing *4.6.4*

1. Process and phase correct the spectrum

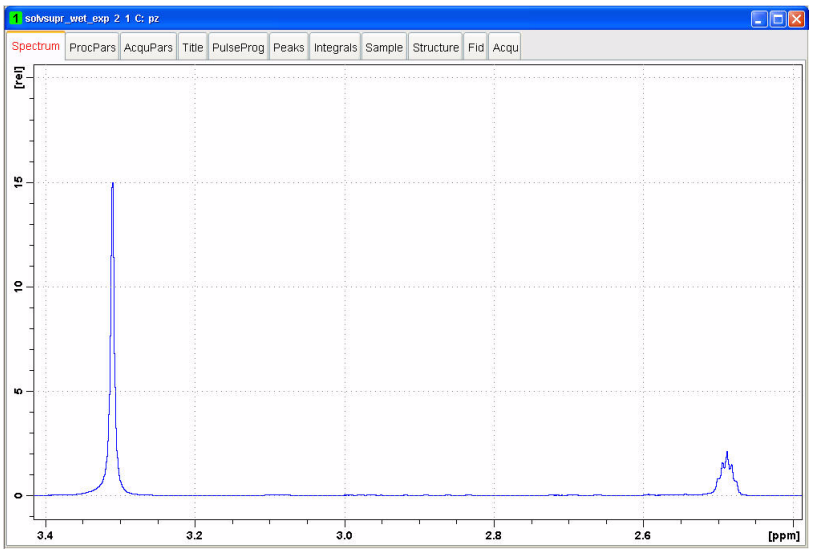
Figure 4.17.



Frequency list set up *4.6.5*

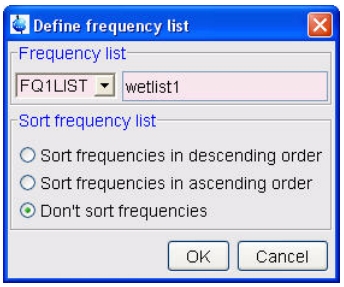
1. Type **wrpa 2** on the command line
2. Type **re 2** on the command line
3. Expand the spectrum to include both peaks for suppression

Figure 4.18.



4. Click on 

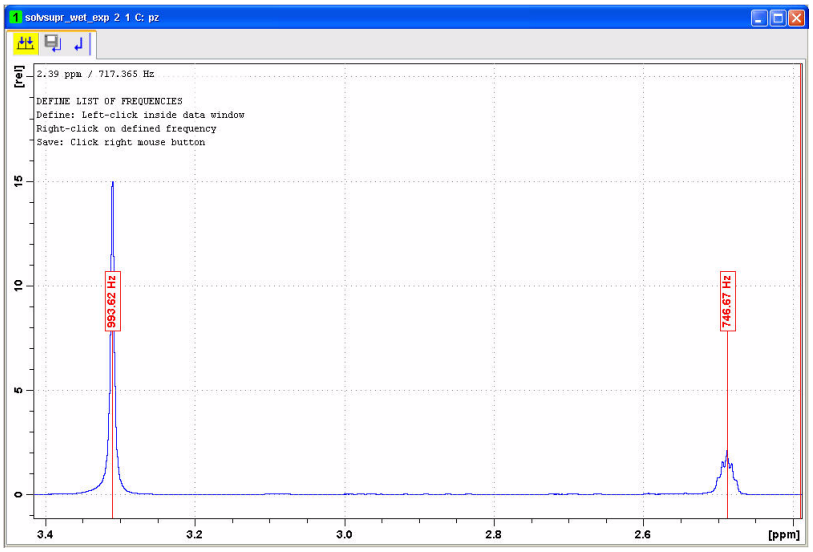
Figure 4.19.



5. Select '**FQ1LIST**' and type a frequency list name (e.g. **wetlist1**)
6. Enable '**Don't sort frequencies**'
7. Click on 

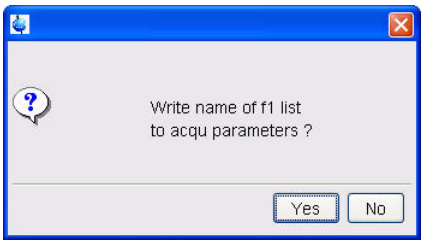
8. Move the cursor line to the center of the peak at 3.3 ppm and click the left mouse button
9. Move the cursor line to the center of the peak at 2.5 ppm and click the left mouse button

Figure 4.20.



10. Click on  to save the frequency list

Figure 4.21.



11. Click on 

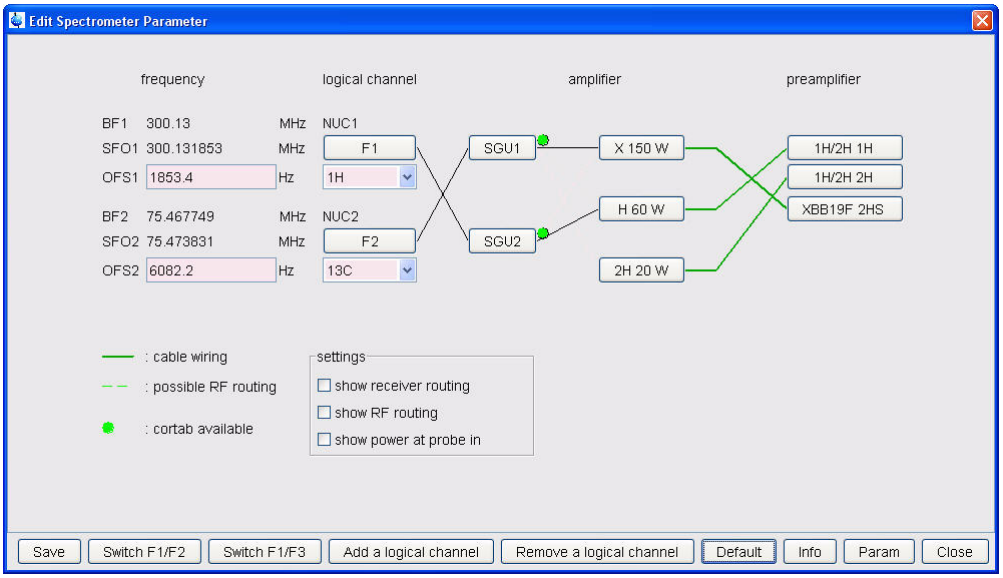
Setting up the acquisition parameters *4.6.6*

1. Select the '**AcquPars**' tab by clicking on it
2. Change the following parameter:

PULPROG = **wetdw**

3. Click on  to display the routing
4. Select '**13C**' for '**F2**'
5. Click on 

Figure 4.22.



6. Click on Save
7. Click on to display the pulsprogram parameters
8. Change the following acquisition parameters:

NS	=	**64**
DS	=	**16**
CPDPRG2	=	**garp**
GPZ21	=	**80**
GPZ22	=	**40**
GPZ23	=	**20**
GPZ24	=	**10**

9. Click on to read in the Prosol parameters
10. Select the '**Spectrum**' tab by clicking on it

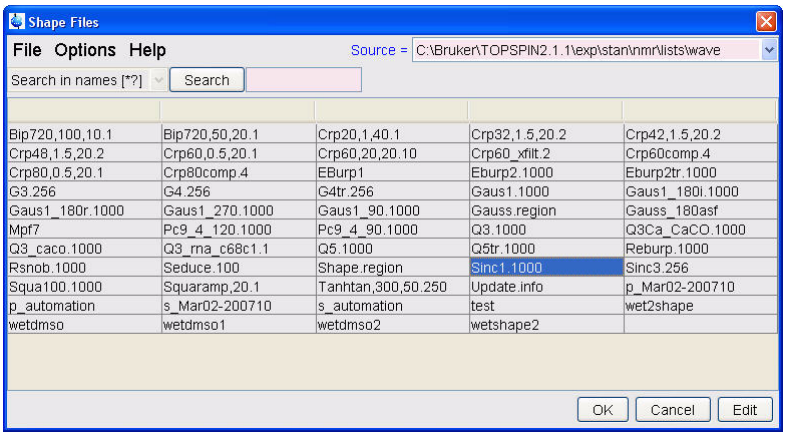
Selective pulses set up *4.6.7*

NOTE: One shaped pulse is created and can be tailored to select for a single or multiple resonances.

1. In the main menu click on '**Spectrometer**' and select '**Shape Tool**' or type **stdisp** in the command line

2. In the shape tool menu bar click on  and select **‘Shape’**

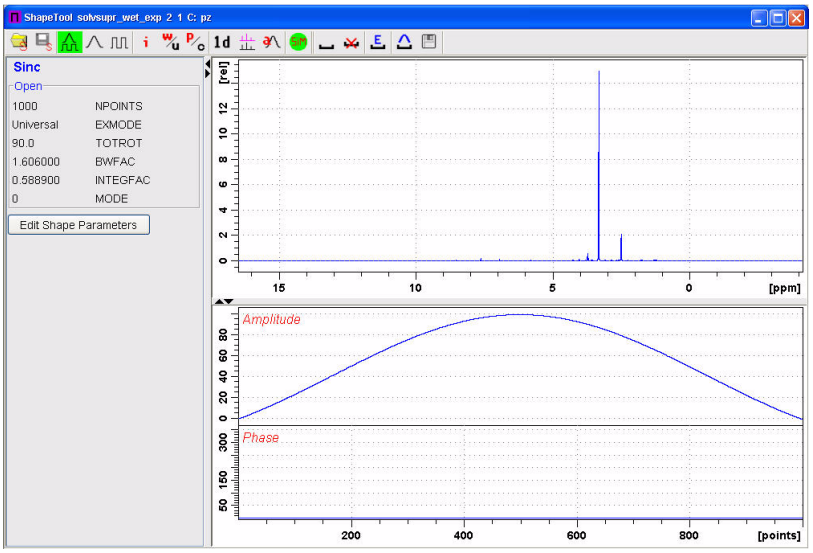
Figure 4.23.



3. Select ‘**Sinc1.1000**’

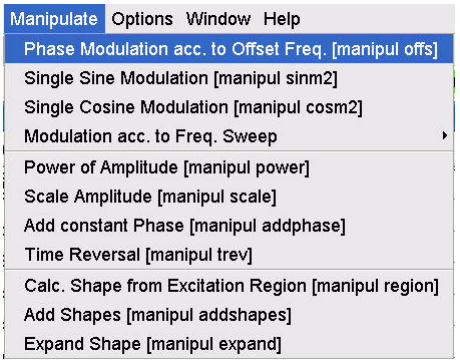
4. Click on 

Figure 4.24.



5. In the main menu click on ‘**Manipulate**’ and select ‘**Phase Modulation acc. to Offset Freq.**’ by clicking on it

Figure 4.25.



6. Enable '**Beginning at Phase 0 (ly->lz)**'

7. Enable '**Reference = O1from current Data Set**'

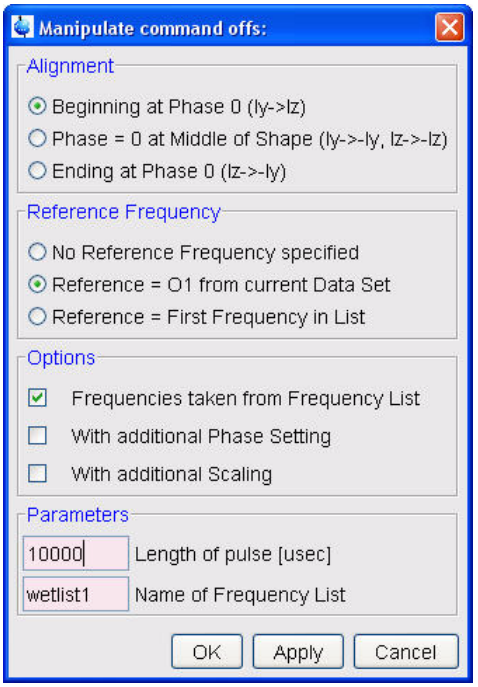
8. Check off '**Frequencies taken from Frequency List**'

9. Change Parameters:

Length of pulse (usec) = **10000**

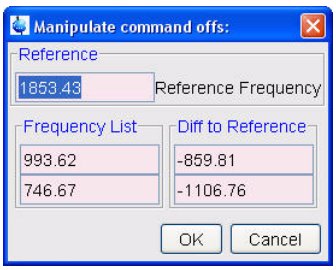
Name of Frequency List = **wetlist1**

Figure 4.26.



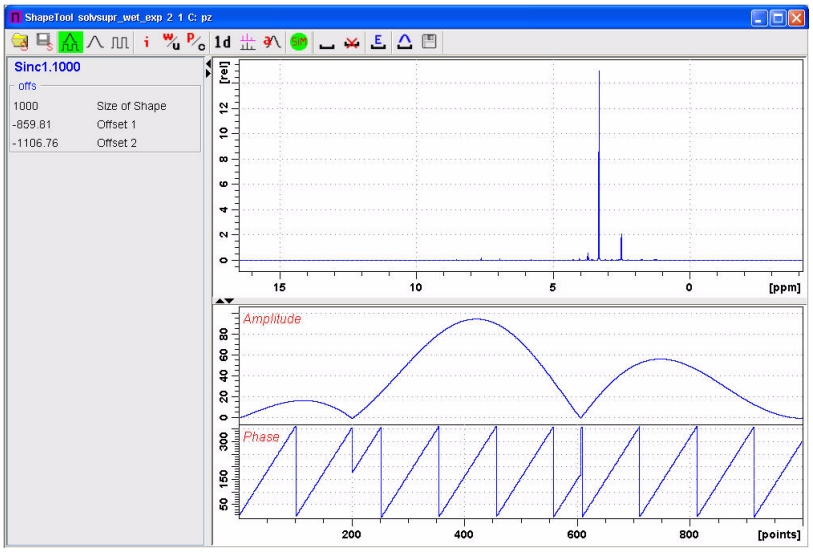
10. Click on 

Figure 4.27.



11. Click on 

Figure 4.28.



12. In the main menu click on '**Options**' and select '**Define Parameter Table**' by clicking on it

Figure 4.29.

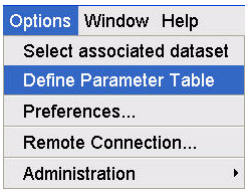
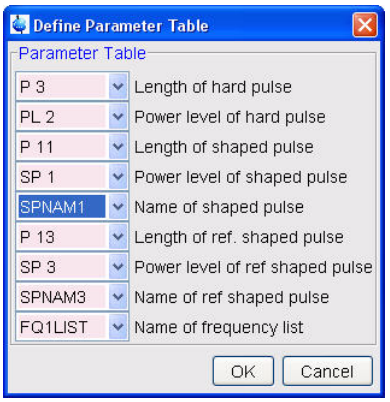


Figure 4.30.



13. Make the following changes:

Length of shaped pulse = **p11**

Power Level of shaped pulse = **SP1**

Name of shaped pulse = **SPNAM1**

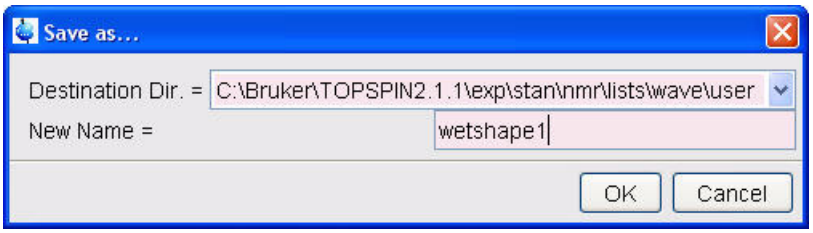
14. Click on 

15. Click on 

Figure 4.31.

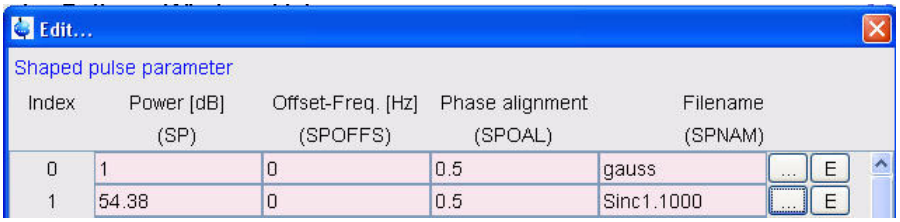
18. Type **wetshape1** in the File Name window
19. Click on OK

Figure 4.32.



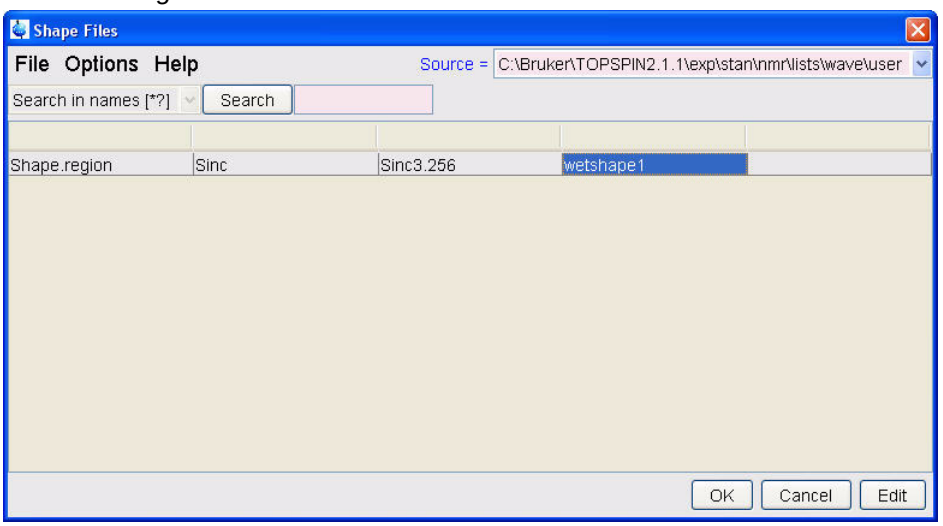
20. Click on OK
21. Click on to close the Shape Tool window
22. Type **shape** in the command line

Figure 4.33.



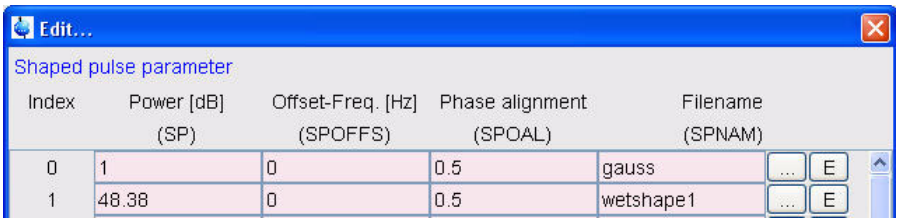
23. Click on to select SPNAM 1
24. Select the user directory in the '**Source**' window

Figure 4.34.



25. Select '**wetshape1**' from the list
26. Click on OK

Figure 4.35.



SP1 = power level adjusted to account for the number of frequency positions (see list below)

1 frequency = calibrated power level, e.g. 54.81 db
2 frequencies = calibrated level minus 6 dB, e.g. 48.81 db
3 frequencies = calibrated level minus 9.5 db, e.g. 45.31 db
4 frequencies = calibrated level minus 12 db, e.g. 42.81 db

27. Click on OK

Running the experiment *4.6.8*

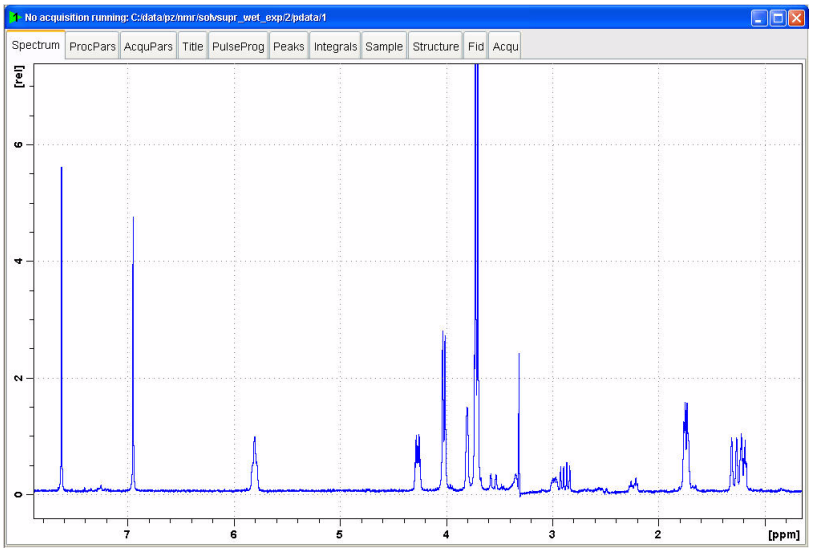
1. Type **lcwetset** in the command line

2. Tune the probe

To tune the probe a second time is necessary to tune the F2 frequency set to 13C for decoupling.

3. Type **rga** in the command line
4. Type **zg** in the command line
5. Type **ef** in the command line
6. Type **apk** in the command line or adjust the phase manually

Figure 4.36.



T1 Experiment 5

Introduction 5.1

The experiment discussed in this chapter is a Proton inversion recovery T1 using a variable delay list.

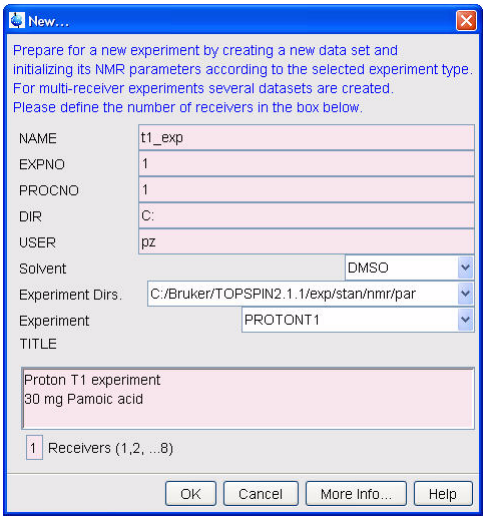
Sample:

30mg Pamoic acid in DMSOd6

Parameter set up 5.1.1

1. Type **edc** and change the following parameters

Figure 5.1.



2. Click on 

3. Insert the sample
4. Type **lock** and select DMSO

5. Turn the spinner off

NOTE: T1 experiments should be run non spinning

6. Tune the probe
7. Shim for best homogeneity
8. Select the '**AcquPars**' tab by clicking on it
9. Make the following change

TD (F1) = **10**

SW[ppm] (F2) = **6**

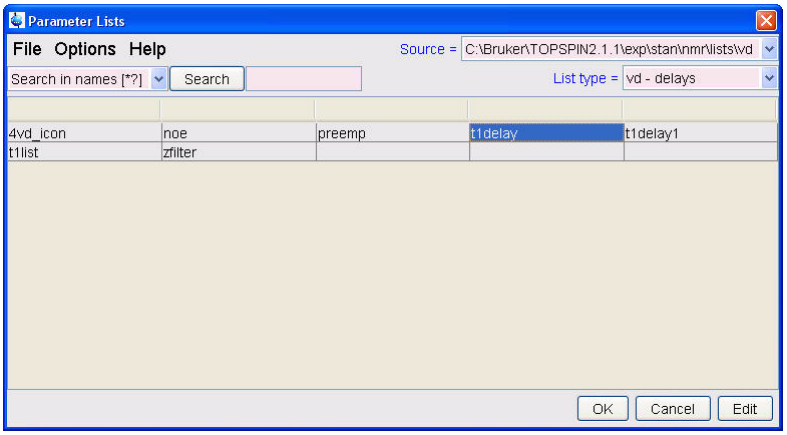
O1[ppm] = **6.5**

10. Click on  to read in the Prosol parameters

NOTE: The value of TD in F1 is the number of delays used. To get exact T1 results, the digital resolution in F2 may have to be adjusted by changing either the TD or SW.

13. Click on  to display the pulse program parameters
14. Change D1[s] = **15**
15. Click on  to the right of the VDList name entry box

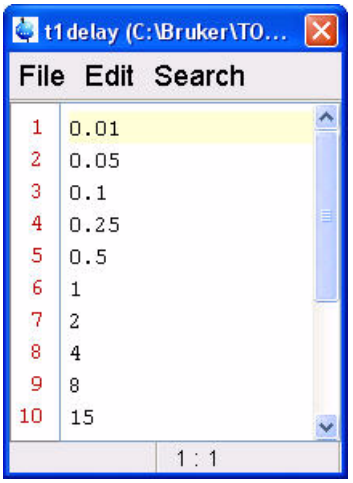
Figure 5.2.



16. Select '**t1delay**' by clicking on it

click on OK

Figure 5.3.



17. Enter the variable delay values as shown in Figure 6.3
18. Click on File and select '**Save**' by clicking on it
19. Click on File and select '**Close**' by clicking on it

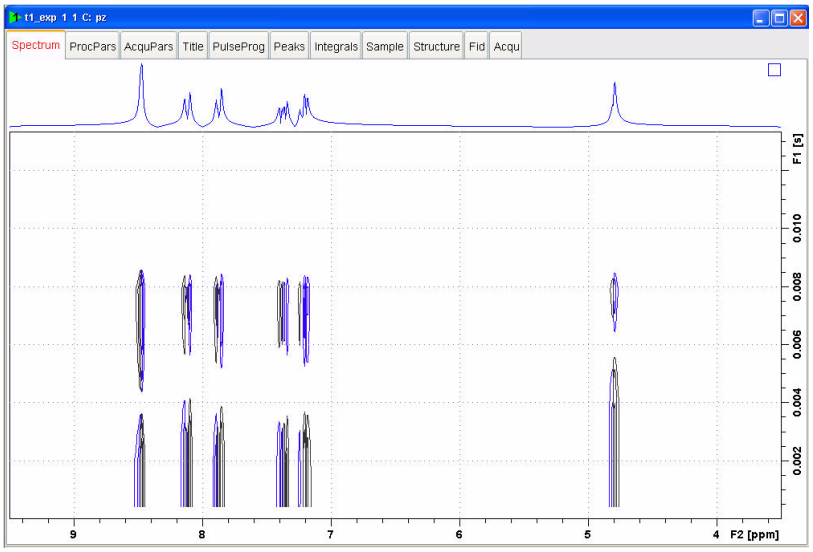
Acquisition *5.1.2*

1. Type **rga**
2. Type **zg** to start the acquisition

Processing *5.1.3*

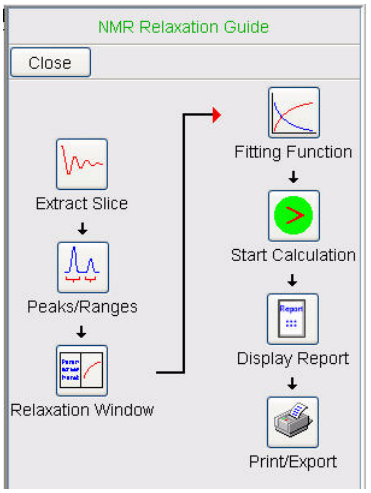
1. Type **xf2** on the command line

Figure 5.4.



2. Click on '**Analysis**' and Select '**T1/T2 Relaxation**' by clicking on it

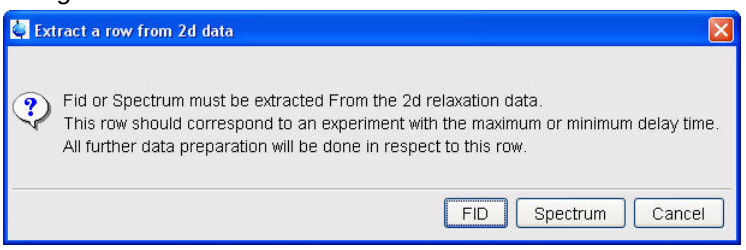
Figure 5.5.



NOTE: While executing the steps in the Guide, message windows will pop up. Please read each message thoroughly and follow the instructions in it.

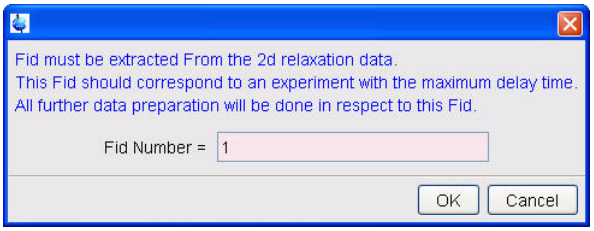
4. In the Guide window, click on  'Extract Slice'

Figure 5.6.



5. Click on FID

Figure 5.7.



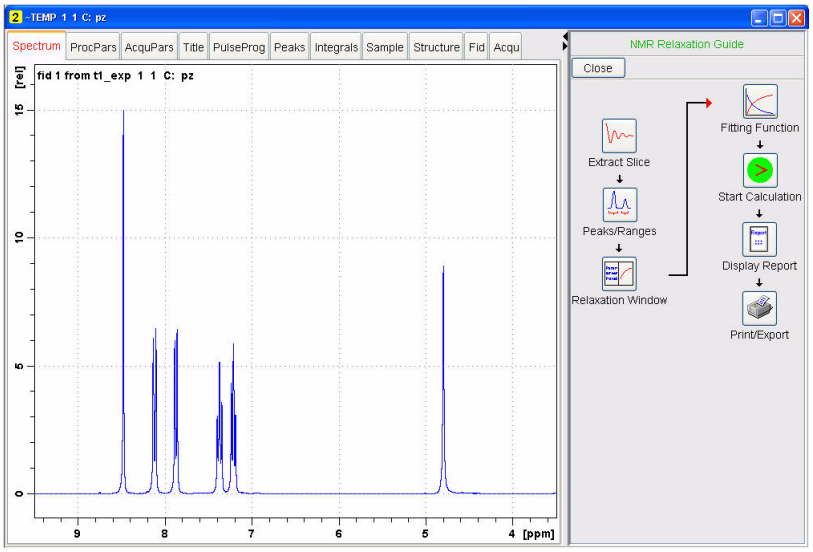
6. Select Fid Number = 1

7. Click on OK

NOTE: The Ft and Phase correction are done automatically. If the spectrum contains broad peaks see Figure 5.8, it may be necessary to perform a additional manual phase correction.

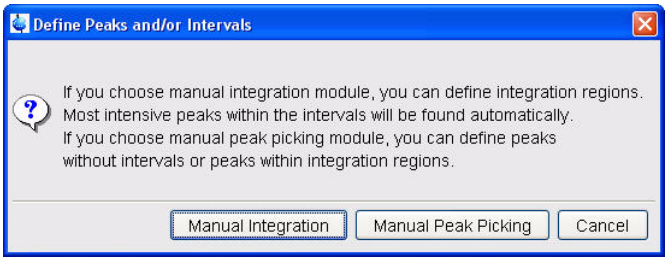
8. Check if phase is correct

Figure 5.8.



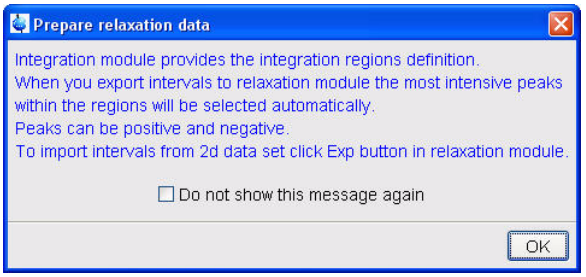
10. In the Guide window, click on  'Define Ranges

Figure 5.9.



11. Click on 

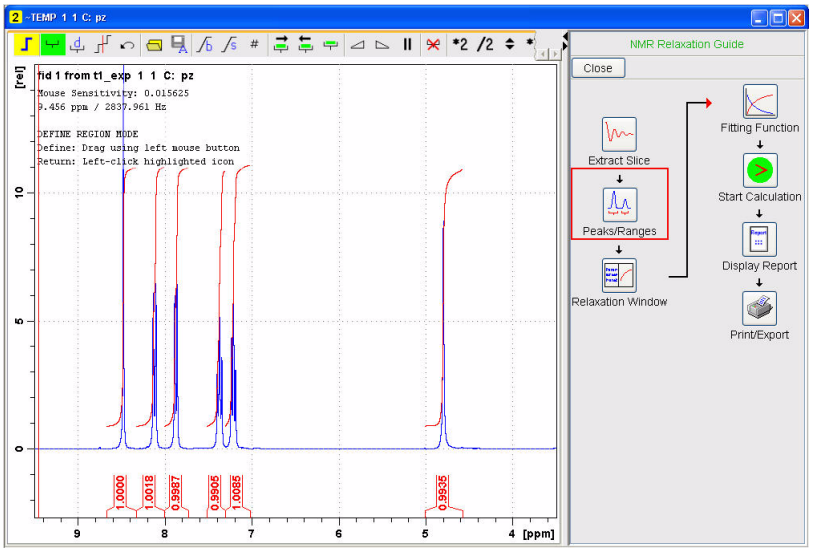
Figure 5.10.



12. Click on 

13. Define the regions by clicking the left mouse button and the use of the cursor lines

Figure 5.11.

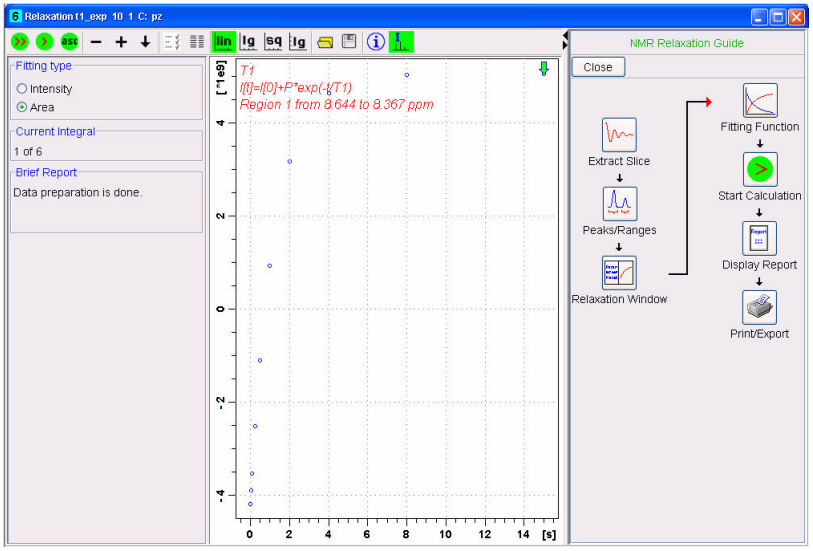


14. Click on 

Figure 5.12.

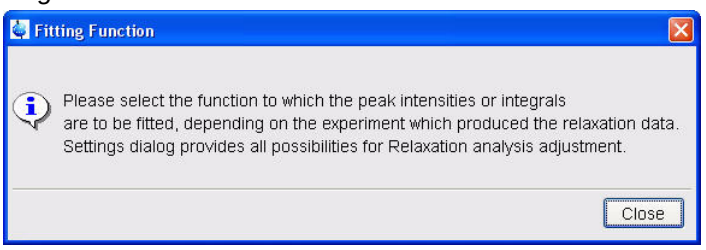
15. Select '**Export Region To Relaxation Module**' by clicking on it
16. In the Guide window, click on  'Relaxation Window'

Figure 5.13.



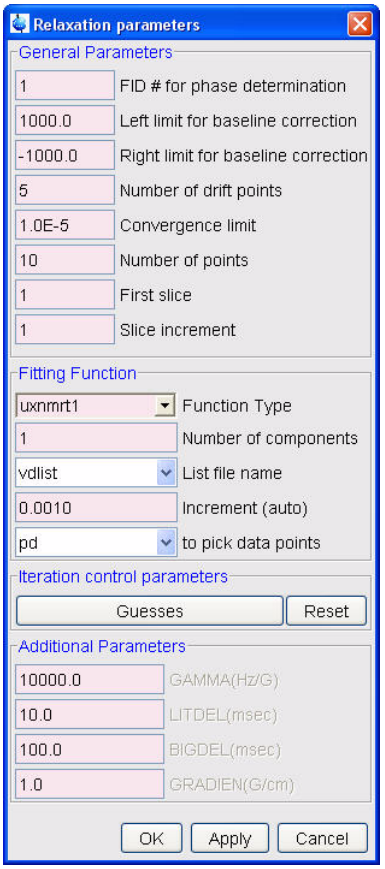
17. In the Guide window, click on  'Fitting Functions'

Figure 5.14.



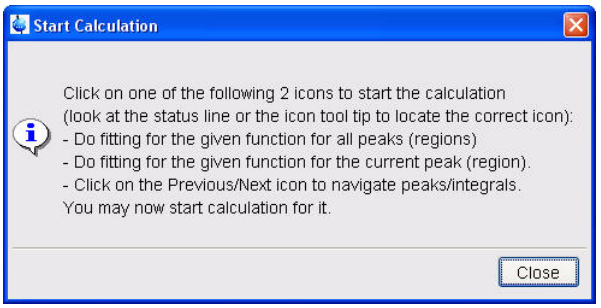
18. Click on Close

Figure 5.15.



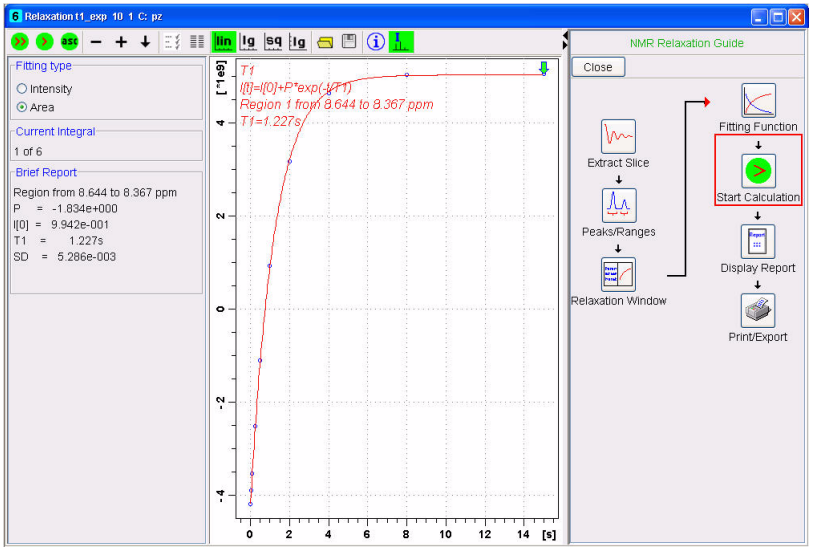
19. Click on OK
20. In the Guide window, click on  'Start Calculating'

Figure 5.16.



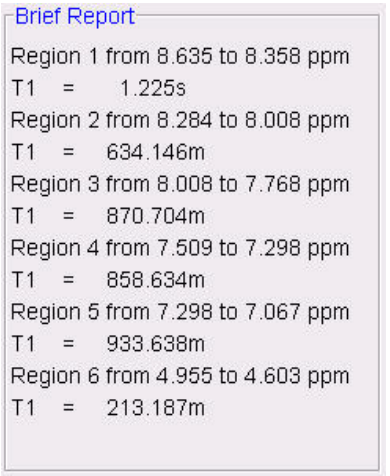
21. Click on 

Figure 5.17.



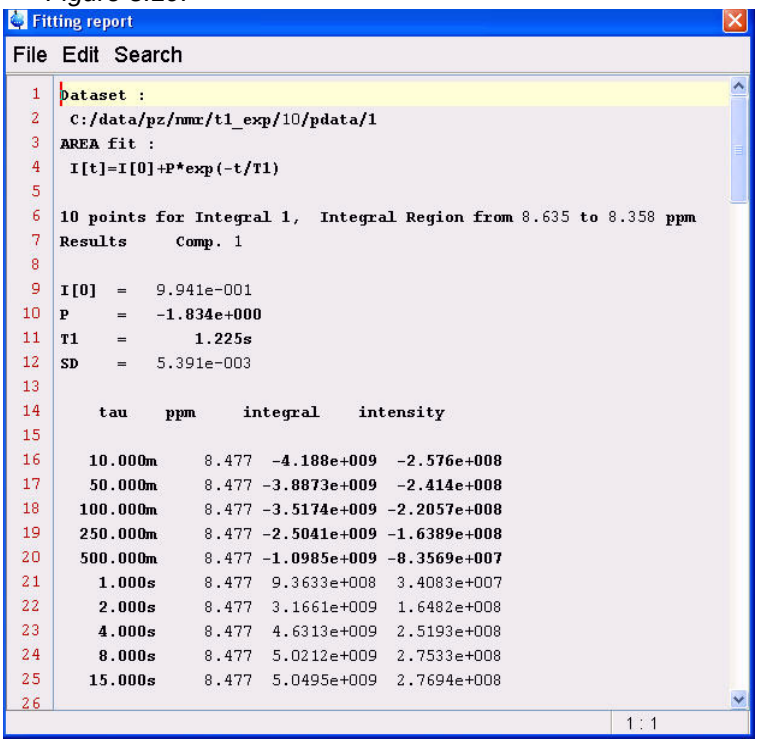
22. In the T1 data display window click on  to calculate all regions

Figure 5.18.



23. In the Guide window, click on  'Display Report'

Figure 5.19.



24. Click on '**File**' and select '**Close**'

Adding a New Nucleus

6

Observing 28Si 6.1

NOTE: Since there are different types of BB probes and system configurations, the below instructions are divided into sections. As an example, the nucleus 29Si is chosen.

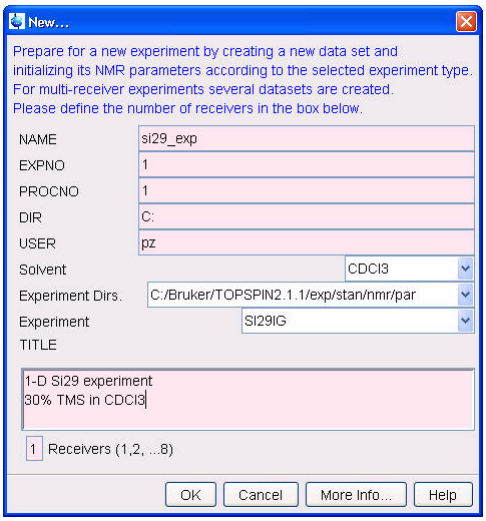
Preparation 6.1.1

Sample:

30% TMS in CDCl3

1. Type **edc** and change the following parameters

Figure 6.1.

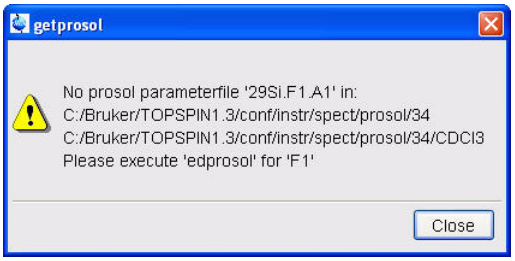


2. Click on 

3. Insert the sample

4. Type **lock** and select CDCl3
5. Tune the probe
6. Shim for best homogeneity
7. Select the '**AcquPars**' tab by clicking on it
8. Click on  to read in the Prosol parameters

Figure 6.2.



The error message appears only if the 90 deg. pulse in the prosol table of the new nucleus is set to zero. The proton parameters for decoupling on the other hand are copied into the new parameter set SI29IG

4. Click on 

NOTE: For the next steps the 90 deg. pulse and the corresponding power level of a nucleus close in frequency has to be known. In this case, the closest in frequency to 29Si is the nucleus 13C. The values can be found in the prosol table

5. Type **p1** on the command line
6. Enter the 13C 90 deg. transmitter pulse value
7. Type **pl1** on the command line
8. Enter the 13C 90 deg. transmitter power level value

BB-probe with ATM

1. Type **atmm** on the command line

NOTE: The manual probehead tuning/matching window (Figure 6.3) and the wobble curve (Figure 6.4) appears.

Figure 6.3.

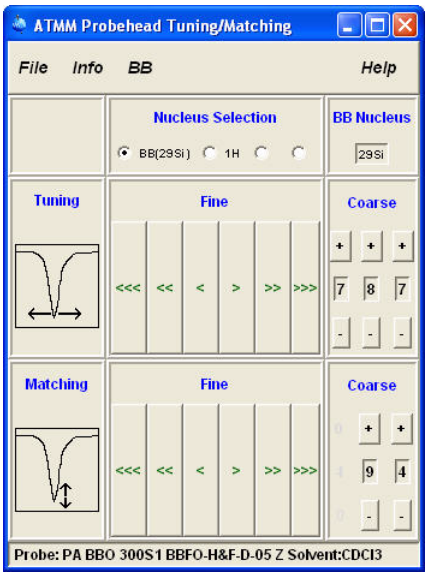
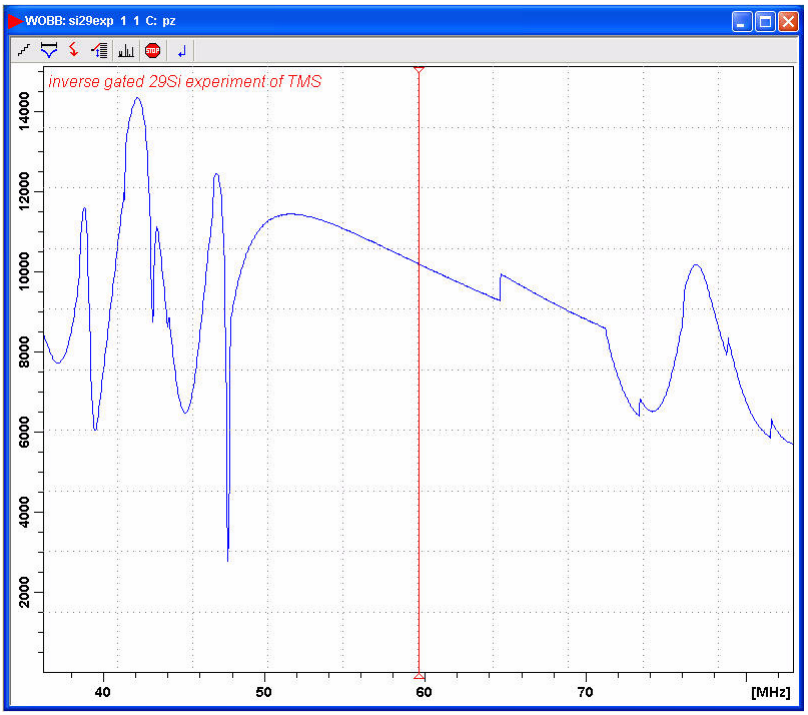


Figure 6.4.



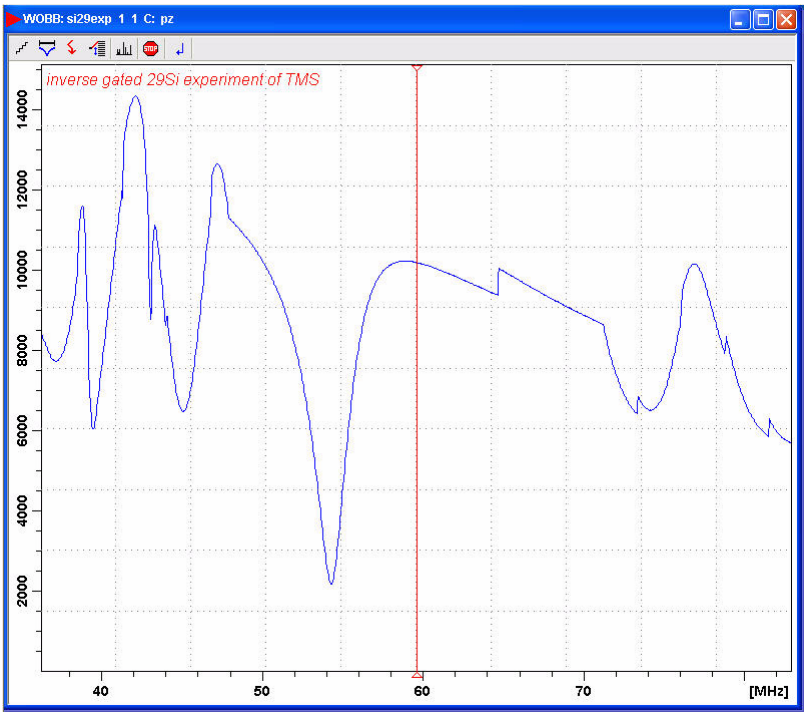
NOTE: The following steps below are executed in the atmm probehead tuning/ matching window.

2. Adjust the tuning by clicking on the >>> button

3. Repeat step 2 multiple times and watch the wobble curve moving towards the red line which indicates the correct frequency for 29Si

NOTE: If the curve does not reach the center (Figure 6.5) and the arrows are turning red, that means the fine tuning capacitor has reached the end. In this case the coarse tuning has to be changed.

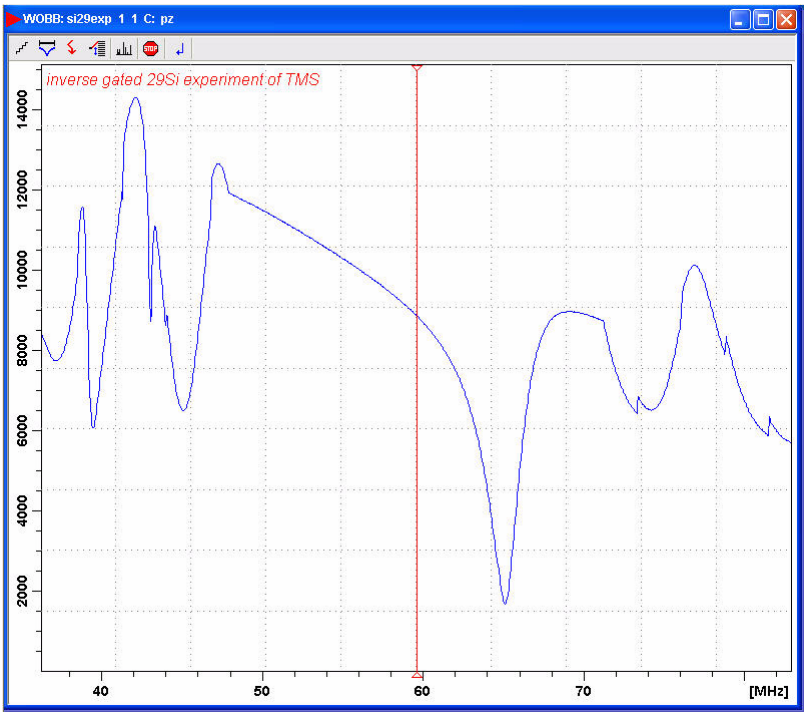
Figure 6.5.



4. Click once on the coarse tuning + button

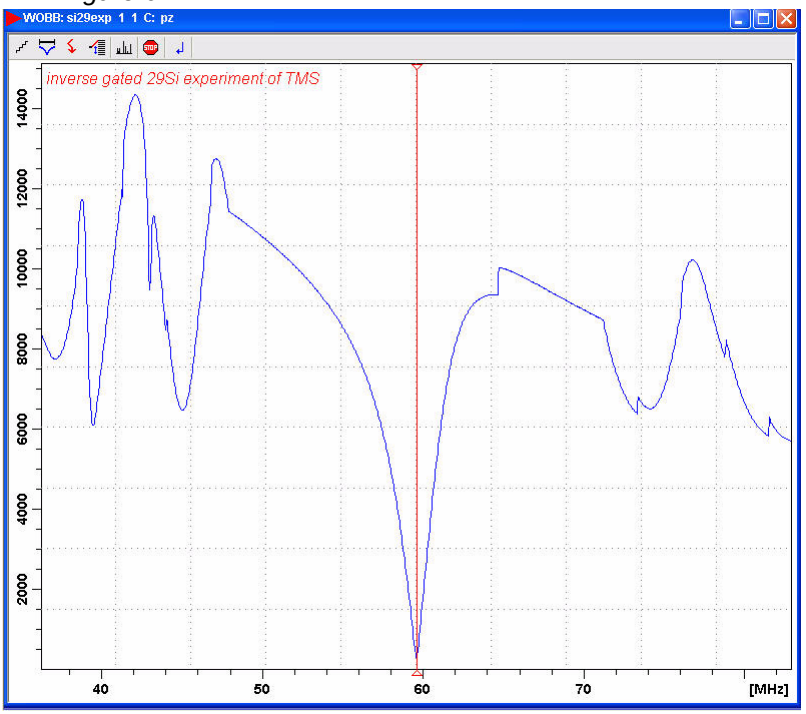
NOTE: The wobble curve jumps to the right side of the red line (Figure 6.6)

Figure 6.6.



5. Clicking on the coarse matching - button and the use of the <<< << < buttons, move the wobble curve on to of the red line (Figure 6.7)

Figure 6.7.



6. In the ATMM probehead/matching window click on '**File**' and select '**Save position**'

7. Click on '**File**' again and select '**Exit**'

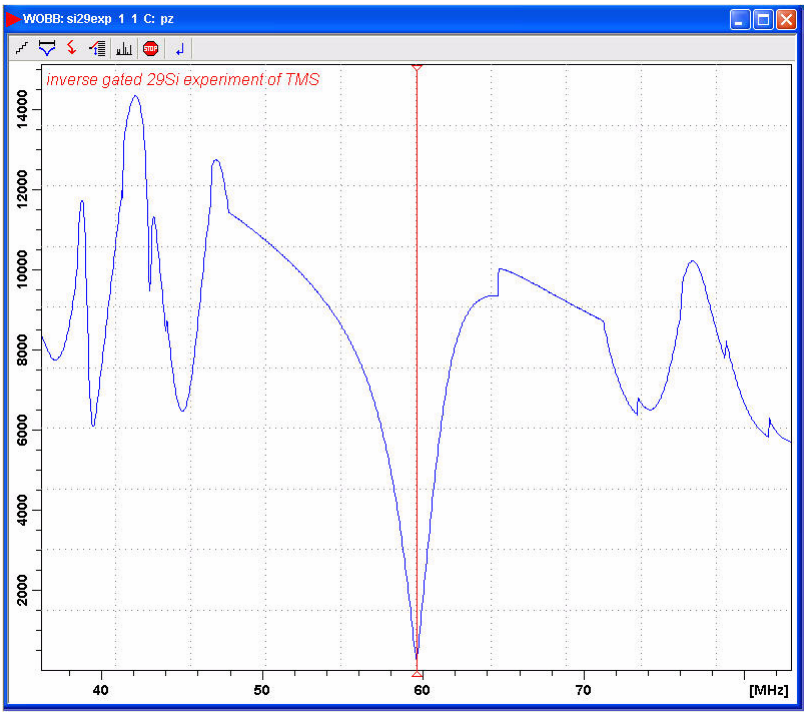
BB-probes without ATM

1. Using the sliders on the bottom of the probe dial in the tuning and matching numbers for 13C

2. Type **wobb** on the command line

3. Adjust the tuning and matching sliders on the bottom of the probe to move the wobble curve in to the red line

Figure 6.8.



4. Type **stop** on the command line

Determine the 90 deg. pulse length *6.1.3*

Systems without cortab files and power check turned off

NOTE: Power check is designed to protect probe from being damaged by excessing power. Since the transmitter power output over the whole frequency range is not perfectly linear, a procedure called cortab has to be performed on all observed nuclei. This requires special hardware and if it is all possible it should be done by a Bruker engineer. The cortab files are found in the directory [TopSpin home]/conf/instr/spect/cortab

1. Type **iexpno**
2. Select the '**AcquPars**' tab by clicking on it
3. Change AQ_mod = qsim
4. Click on  to display the pulse program parameters
5. Make the following changes:

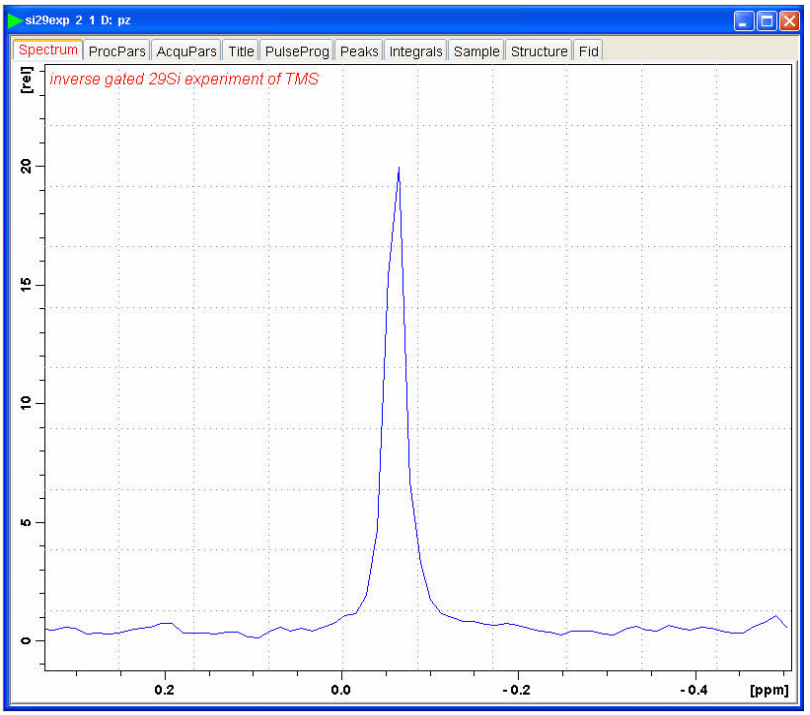
NS = **1**

DS = **0**

D1 = **60**

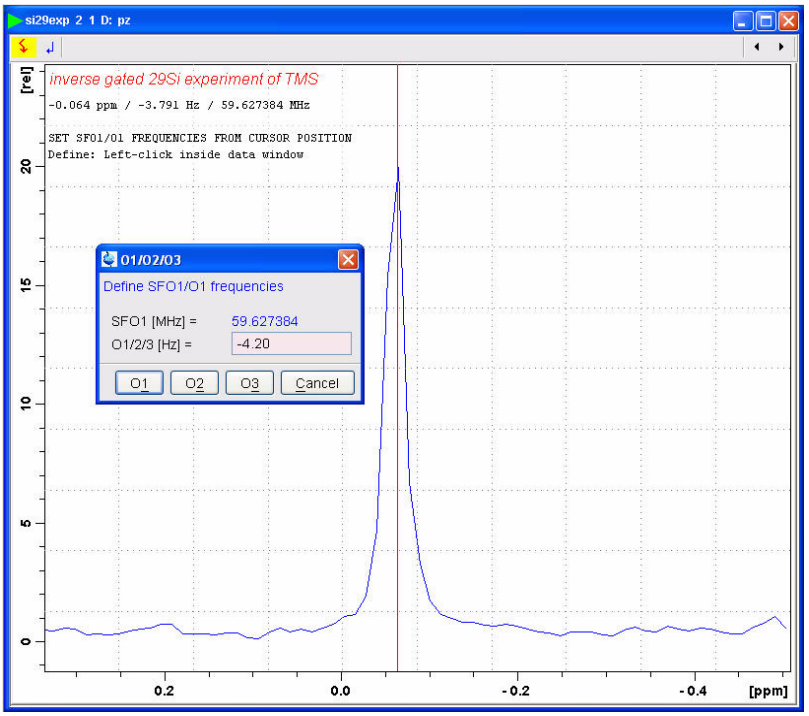
6. Select the '**Spectrum**' tab by clicking on it
7. Type **rga**
8. Type **zg** to start the acquisition
9. Process and Phase correct the spectrum
10. Expand the signal region at 0 ppm

Figure 6.9.



11. Click on 
12. Move the cursor line to the center of the peak and click the left mouse button

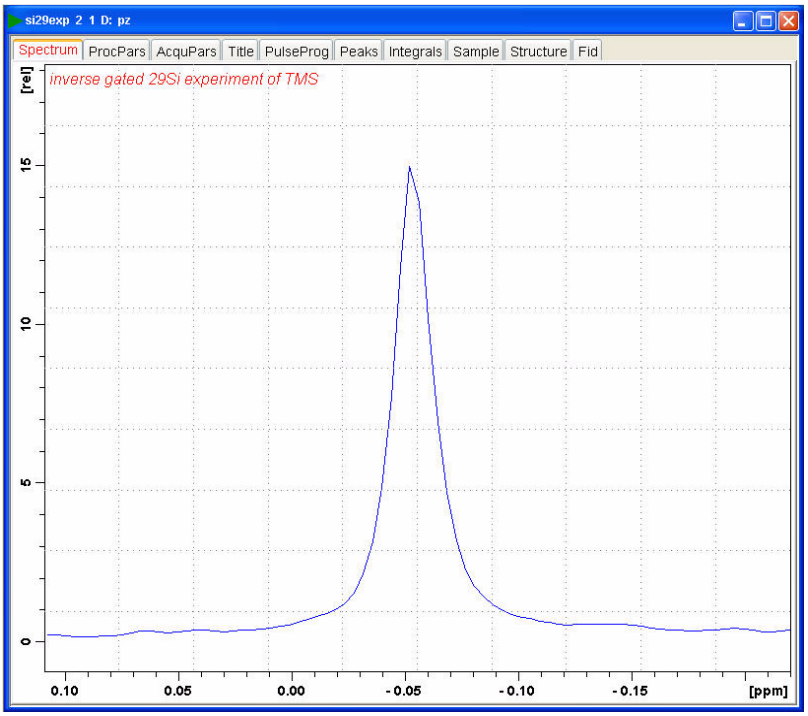
Figure 6.10.



13. Click on 

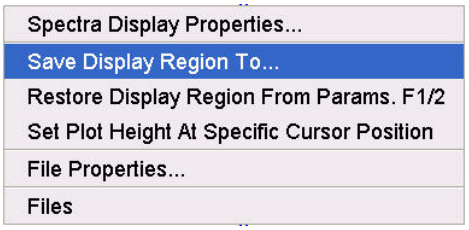
14. Type **swh 1000**
15. Type **td 8k**
16. Type **si 4k**
17. Type **zg** to start the acquisition
18. Process and Phase correct the spectrum
19. Expand the signal region at 0 ppm

Figure 6.11.



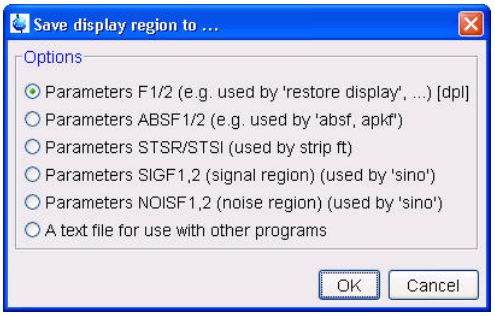
20. Click the right mouse button

Figure 6.12.



21. Select and click on '**Save Displayed Region To**'

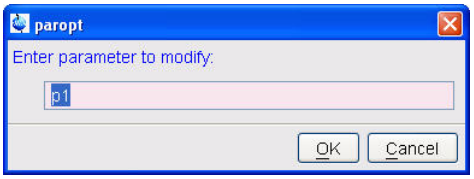
Figure 6.13.



22. Enable '**Parameters F1/2 [dpl]**'
23. Click on 

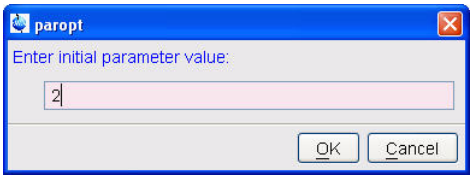
24. Type **paropt**

Figure 6.14.



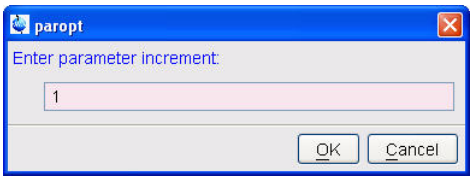
25. Enter **p1**
26. Click on OK

Figure 6.15.



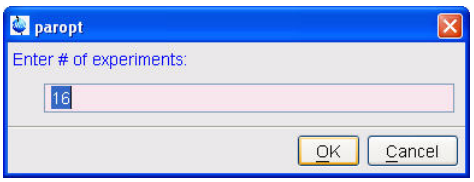
27. Enter **2**
28. Click on OK

Figure 6.16.



29. Enter **1**
30. Click on OK

Figure 6.17.



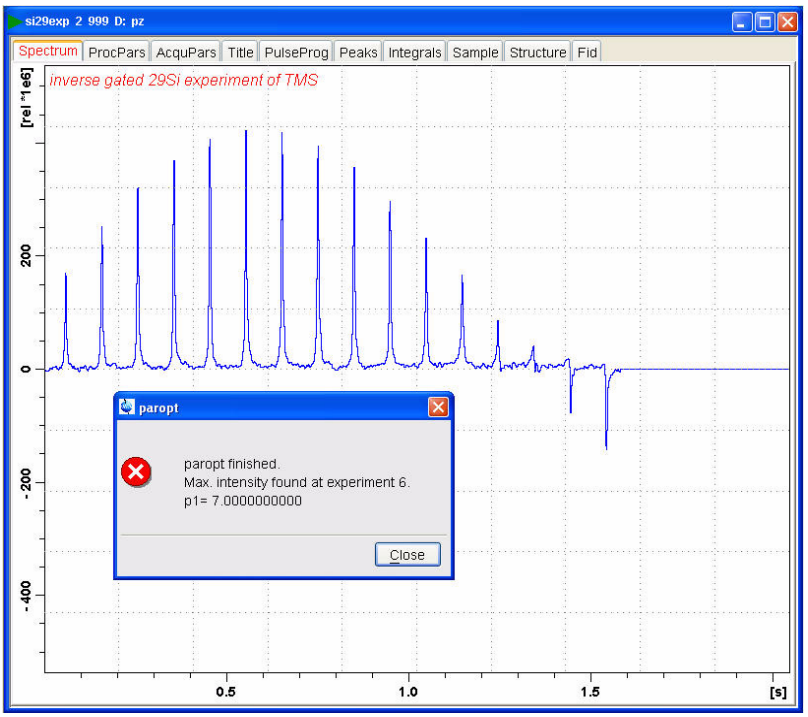
31. Enter 16
32. Click on OK

The AU program paropt is starting the acquisition now and the result is displayed in the window of the processing number 999. On the end of the acquisition the programs preforms a peak picking and determine the tallest peak in the array. A

pop up window appears with value of the 90 degree pulse length for 29Si. Write this value down! To obtain a more accurate value, follow the steps 33 - 36 below.

WARNING: IF THE 90 DEG. PULSE LENGTH IS LESS THEN 5 USEC FOR A 5MM PROBES AND LESS THEN 10 USEC FOR A 10 MM PROBE, THERE IS A RISK OF ARCING. TO PREVENT ARKING, CHANGE PL1 TO A HIGHER DB VALUE AND REPEAT STEPS 24 THROUGH 35.

Figure 6.18.



33. Type **re 2 1**

34. Type **p1** and change the value to be a 360 deg. pulse (multiply the value observed in paropt by 4)

35. Type **zg** to start the acquisition

36. Type **efp**

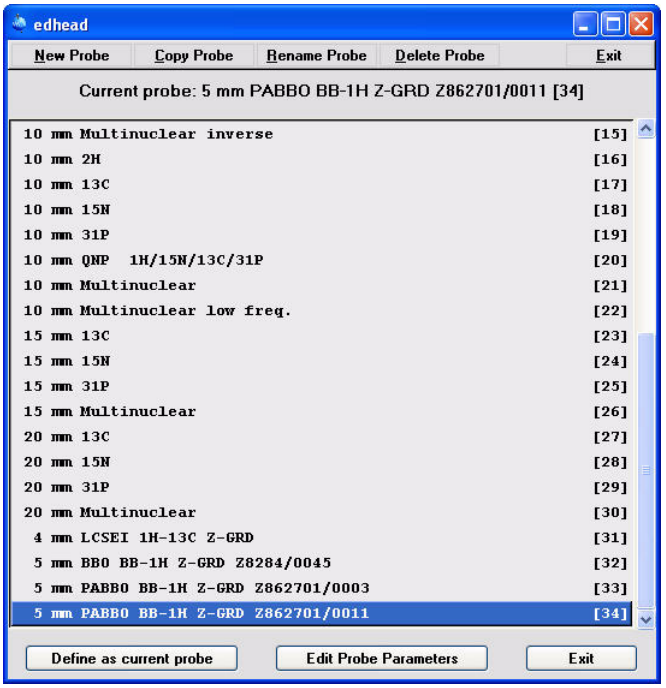
Change p1 in small increments until the signal goes through a null. Simply divide the new value of the 360 deg. pulse by 4. This will be the exact 90 degree pulse for observing 29Si.
IMPORTAND: ENTER THIS VALUE AND THE POWER LEVEL IN TO THE PROSOL PARAMETERS TABLE!

Systems with cortab and power check

NOTE: Power check is designed to protect probe from being damaged by excessing power. Since the transmitter power output over the whole frequency range is not perfectly linear, a procedure called cortab has to be performed on all observed nuclei. This requires special hardware and if it is all possible it should be done by a Bruker engineer. A work around for this procedure, is to copy the existing cortab files of a nucleus which is CLOSE in frequency to the new nucleus. Follow exactly the steps below.

1, Type **edhead** on the command line

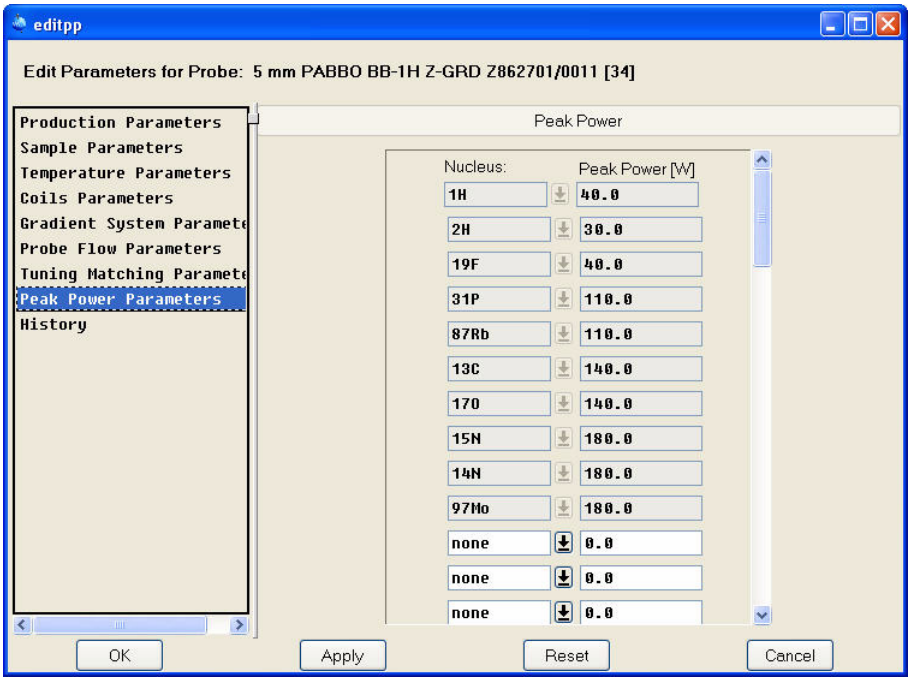
Figure 6.19.



2. Click on 

3. Select Peak Power Parameters

Figure 6.20.



4. Click on the 

5. Select 29Si from the nuclei list

6. In the Peak Power [W] window for the 29Si nucleus, enter the same value as for 13C

Figure 6.21.

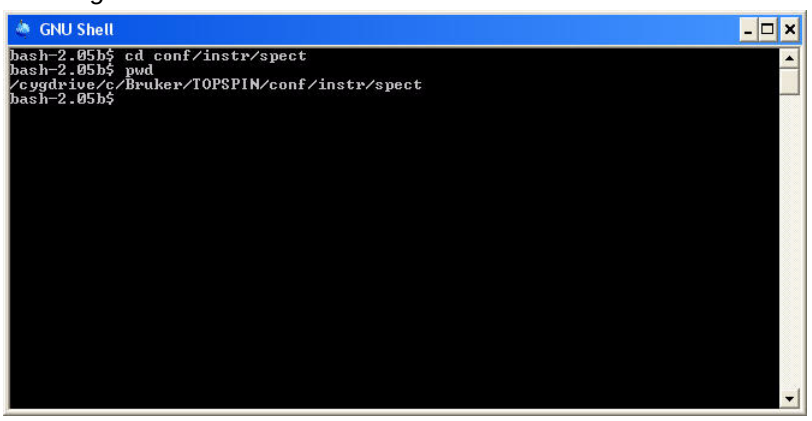
7. Click on Apply
8. Click on OK
9. Click on Exit

The next steps it is necessary to login as the NMR superuser, to avoid permission problems.

Windows XP

10. In the Windows Desktop click on '**start**'
11. Select '**Programs**'
12. Select '**Bruker TOPSPIN**'
13. Select and click on '**GNU shell**'

Figure 6.22.



14. Type **cd conf/instr/spect**

15.Type **pwd** to verify to be in the correct directory

c/Bruker/TOPSPIN/conf/instr/spect

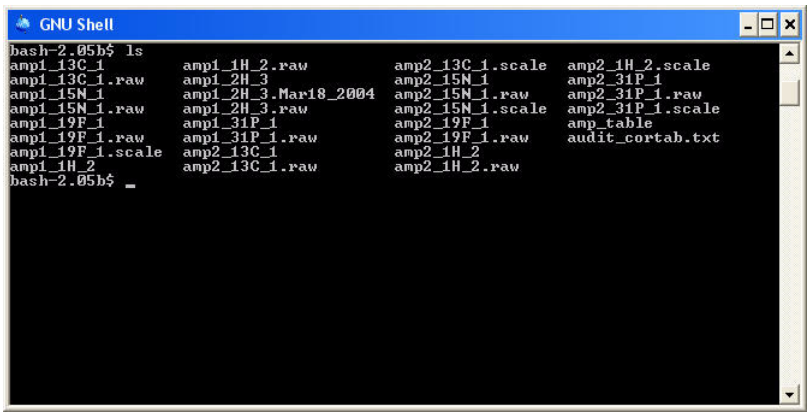
16. Type **cp -R cortab cortab.bkp**

This creates a backup directory of cortab, in case something goes wrong.

17. Type **cd cortab**

18. Type **ls**

Figure 6.23.



19. Type **cp amp1_13C_1 amp1_29Si_1**

20. Type **cp amp2_13C_1 amp2_29Si_1**

21. Type **ls** to verify the copied cortab files

22. Close the GNU shell window

23. To determine the 90 degree pulse length follow step 1 through 36 in the previous section, “6.1.3 System without cortab files and power check turned off”

Homonuclear Decoupling Experiment

7

Introduction 7.1

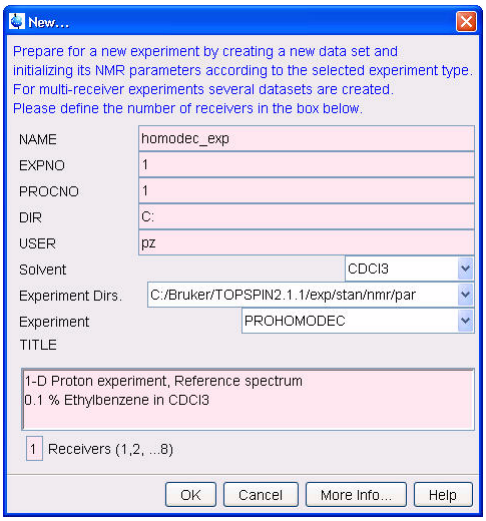
Sample:

0.1% Ethylbenzene in CDCl3

Preparation experiment 7.1.1

1. Type **edc** and change the following parameters

Figure 7.1.



2. Click on 

3. Insert the sample
4. Type **lock** and select CDCl3
5. Tune the probe
6. Shim for best homogeneity
7. Select the '**AcquPars**' tab by clicking on it
8. Click on  to read in the Prosol parameters

9.Make the following changes:

PULPROG = **zg30**

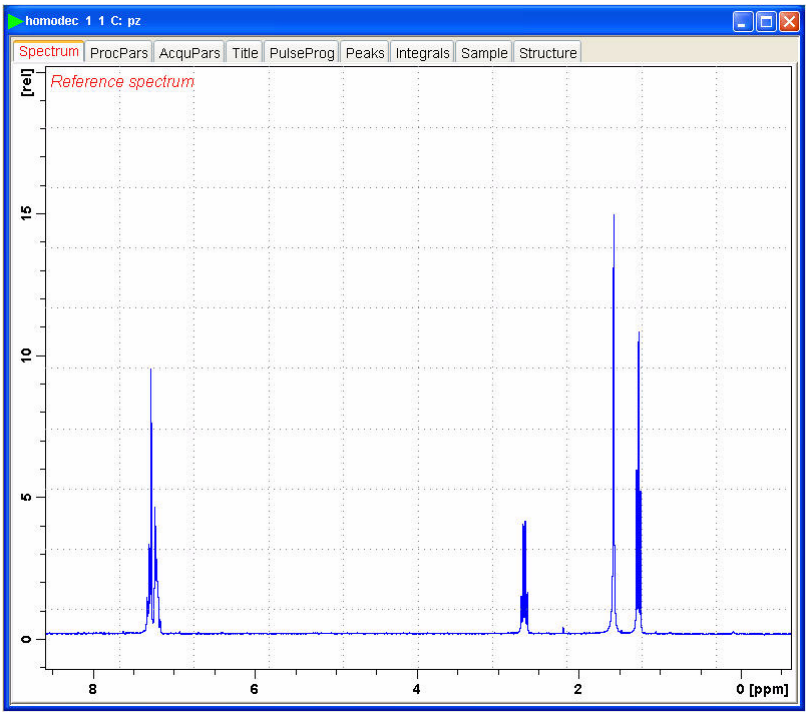
NS = **8**

10. Select the '**ProcPars**' tab by clicking on it
11. Make the following changes:

LB = **1**

12. Type **rga**
13. Type **zg** to start the acquisition
14. Process and Phase correct the spectrum
15. Type **abs**

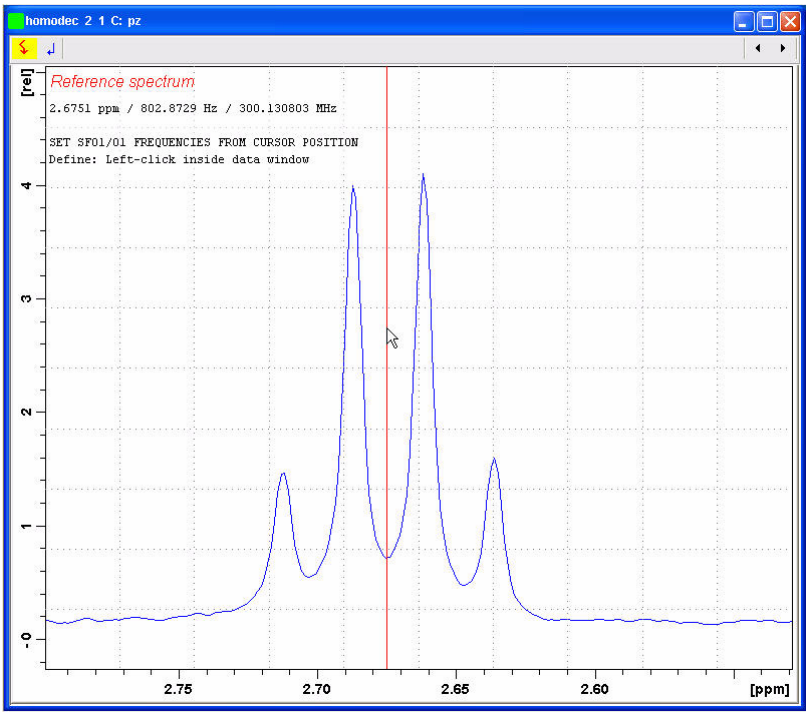
Figure 7.2.



Parameter set up *7.1.2*

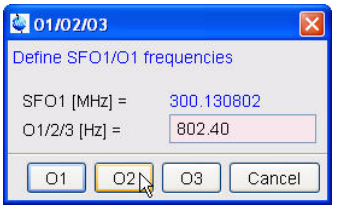
1. Type **wrpa 2** on the command line
2. Type **re 2** on the command line
3. Expand the quartet at 2.65ppm
4. Click on 

Figure 7.3.



5. Move the cursor line to the center of the peak and click the left mouse button

Figure 7.4.



6. Click on 

7. Select the '**AcquPars**' tab by clicking on it

8. Make the following changes:

PULPROG = **zghd**

9. Select the '**Title**' tab by clicking on it

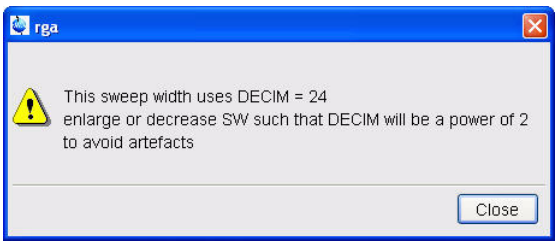
10. Change the title to:

1-D Homonuclear decoupling experiment
0.1 % Ethylbenzene in CDCl3

Acquisition *7.1.3*

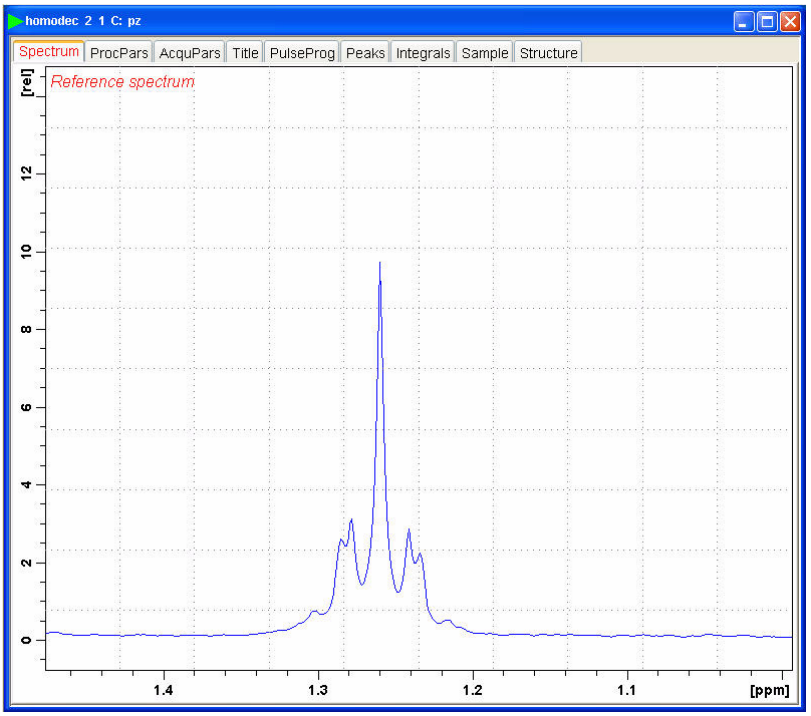
1. Type **rga**

Figure 7.5.



2. Adjust the sweep width if necessary
3. Type **rga**
5. Process and Phase correct the spectrum
6. Type **abs**
7. Expand the peak at 1.25ppm

Figure 7.6.

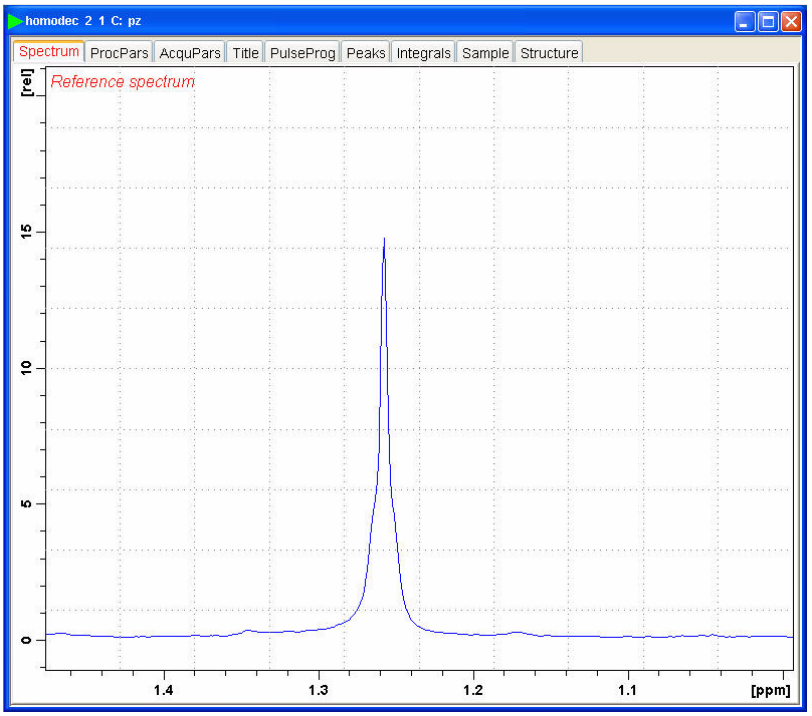


NOTE: This peak is partially collapsed triplet that represents the methyl protons. Increasing the decoupling power level will result in a single peak.

Fine tuning 7.1.4

1. Type **pl24** on the command line
2. Lower the value by **2**
3. Repeat steps 3 through 6
4. If necessary repeat this steps

Figure 7.7.

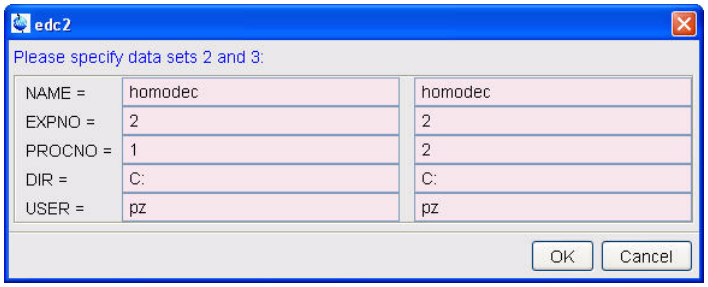


CAUSHION: Increasing the decoupling power level in small steps until the peak is fully decoupled. To much power can cause damage.

Plotting the reference and decoupled spectrum on the same page 7.1.5

1. Display the reference spectrum
2. Type **edc2**
3, Specify data set 2 as the decoupled spectrum

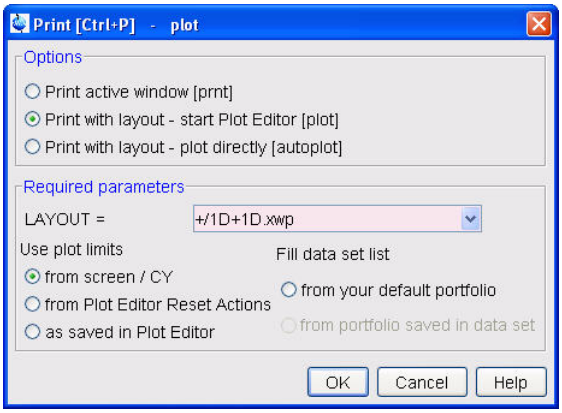
Figure 7.8.



4. Click on OK

5.In the main menu click on '**File**' and select '**Print**'

Figure 7.9.



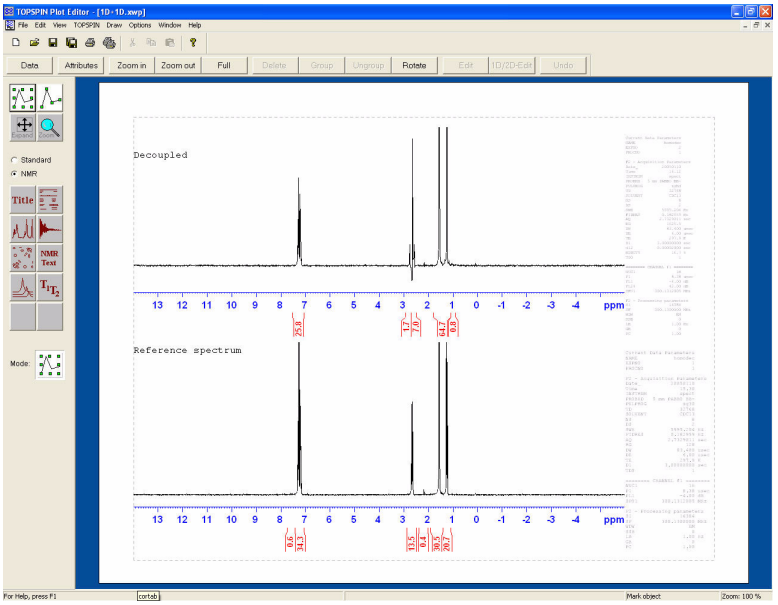
6. In Options select '**Print with Layout - start Plot Editor [plot]**'

7. In Required Parameters select: '**LAYOUT = +/1D + 1D.xwp**'

8. Enable '**from screen/CY**'

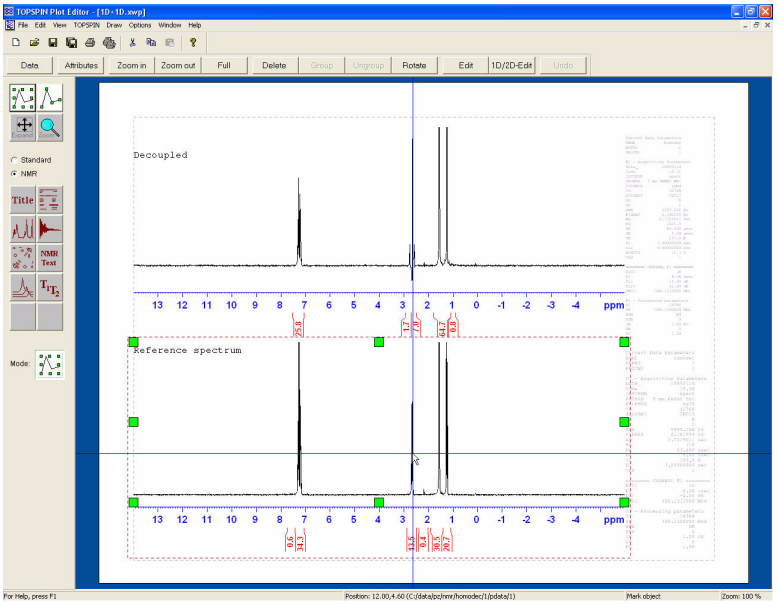
9. Click on OK

Figure 7.10.



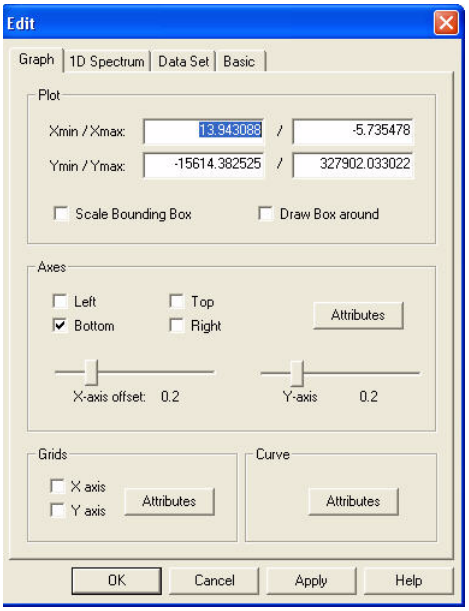
10. Click anywhere on the reference spectrum

Figure 7.11.



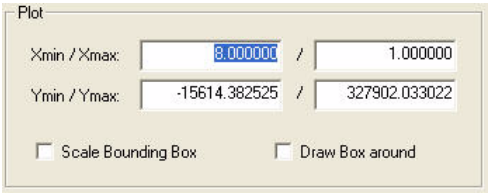
11. Click on 

Figure 7.12.



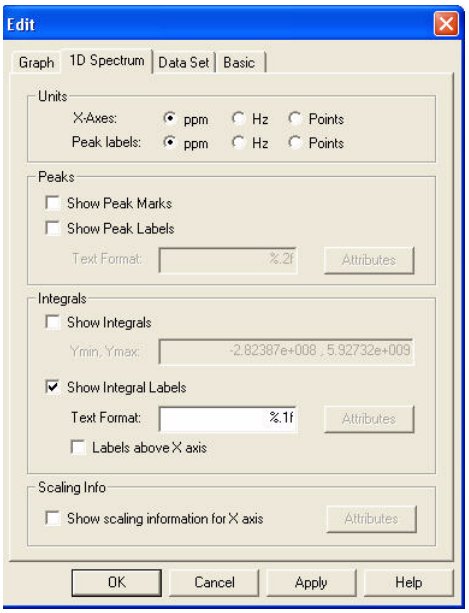
12. Select the '**Graph**' tab by clicking on it
13. Change the '**Xmin/Xmax**' to **8 / 1**

Figure 7.13.



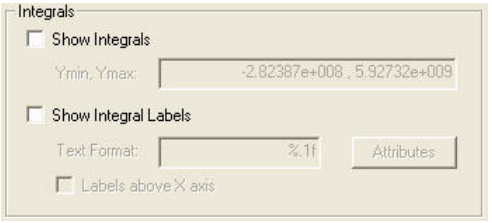
14. Select the '**1D Spectrum**' tab by clicking on it

Figure 7.14.



15. Disable '**Show Integral Labels**'

Figure 7.15.

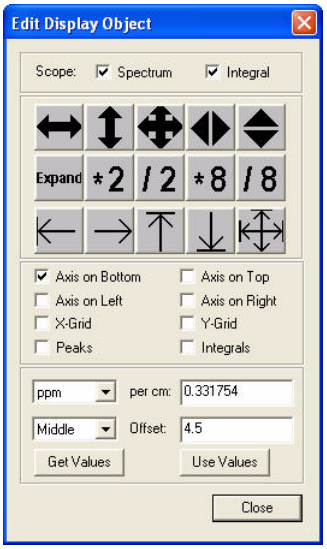


16. Click on 

17. Click on 

18. Click on 

Figure 7.16.

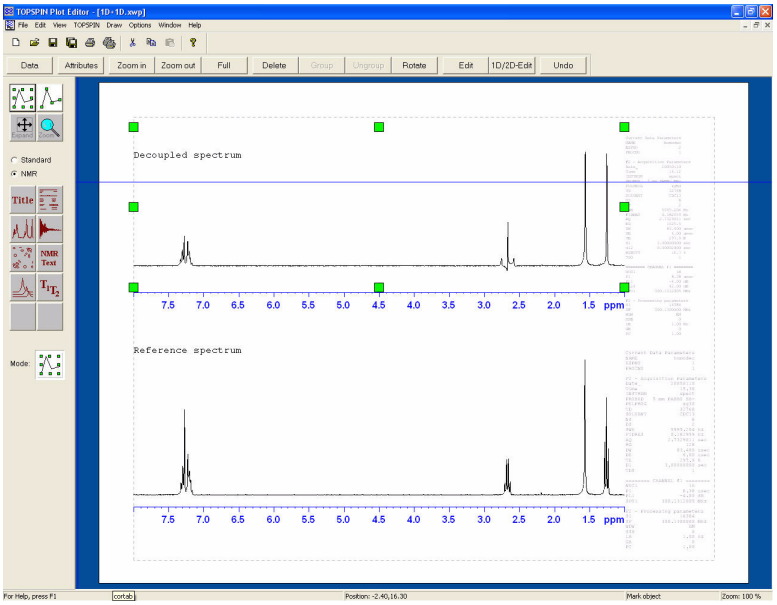


19. Adjust the Y-scaling using the 

or  buttons
20. Click on 

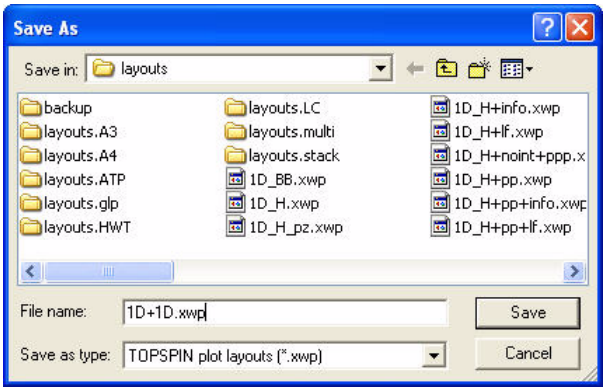
21. Repeat steps 10 through 20 on the decoupled spectrum

Figure 7.17.



22. Click on '**File**' and select '**Save as**'

Figure 7.18.



23. Type new File name (**e.g. 1D+1D_homodec.swp**)

NOTE: Store all new layouts in [TOPSPIN home]\plot\layouts directory

24. Click on '**File**' and select '**Print**' by clicking on it

2D Basic Experiments

8

2-D gradient COSY 8.1

Sample:

30 mg Brucine in CDCl3

Preparation experiment 8.1.1

1. Run a 1D Proton spectrum, following the instructions in the Step-by-Step Tutorial, Basic Experiments User Guide, 1-D Proton Experiment, 2.2

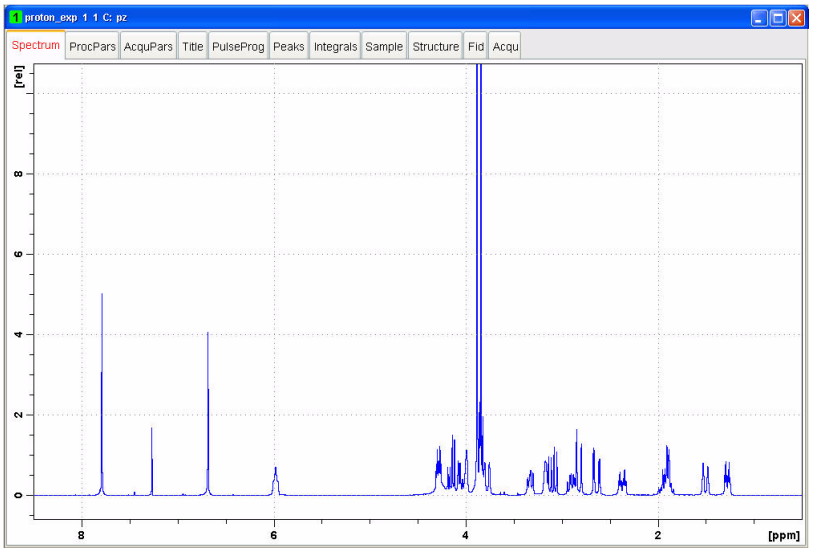
2. Type **wrpa 2** on the command line

3. Type **re 2**

4. Expand the spectrum to display all peaks, leaving ca. 0.5 ppm of baseline on either side of the spectrum

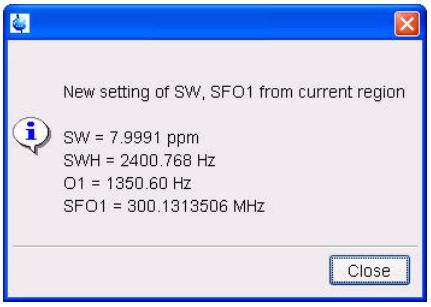
NOTE: You may exclude the solvent peak, if it falls outside of the region of interest.

Figure 8.1.



5. Click on  to set the sweep width and the O1 frequency of the displayed region

Figure 8.2.



6. Write down the value of SW, rounding off to the nearest 1/10th of a ppm
7. Write down the value of O1, rounding off to the nearest Hz
8. Click on 

9. Type **sr** and write down the exact value

Setting up the COSY experiment *8.1.2*

1. Type **rpar COSYGPSW all**

2. Turn the spinner off

NOTE: 2-D experiments should be run non spinning

3.Select the '**AcquPars**' tab by clicking on it

4. Make the following changes:

SW [F2] = e.g. **8** (value from step 6, Preparation experiment 8.1.1)

SW [F1] = same exact value as SW (F2)

O1 [Hz] = e.g. **1351** (value from step 7, Preparation experiment 8.1.1)

5. Click on  to read in the Prosol parameters

6. Select the '**ProcPar**' tab by clicking on it

7. Make the following changes:

SR [F2] = e.g. **0** (value from step 9, Preparation experiment 8.1.1)

8. Select the '**Title**' tab by clicking on it

9. Make the following changes:

2-D gradient COSY experiment
30 mg Brucine in CDCl3

10. Select the '**Spectrum**' tab by clicking on it

Acquisition *8.1.3*

NOTE: The following steps 1 through 4 are necessary to determine the exact receiver gain

1. Type **pulprog zg** on the command line

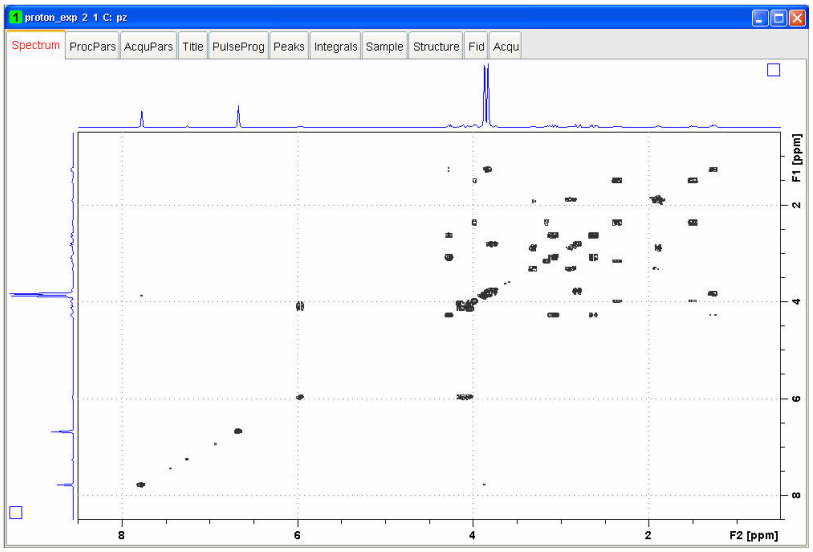
2. In the main menu click on '**Spectrometer**', select '**Adjustment**' and click on '**Auto-adjust receiver gain**' or type **rga**

3. Type **pulprog cosygpqf** on the command line

4. Type **zg** to start the acquisition

1. Type **xfb** on the command line to process the 2-D data
2. Type **sym** on the command line to symmetrize the 2-D data

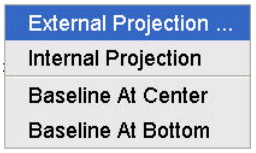
Figure 8.3.



NOTE To display the higher resolution external projections, follow the steps 3 through 8 below

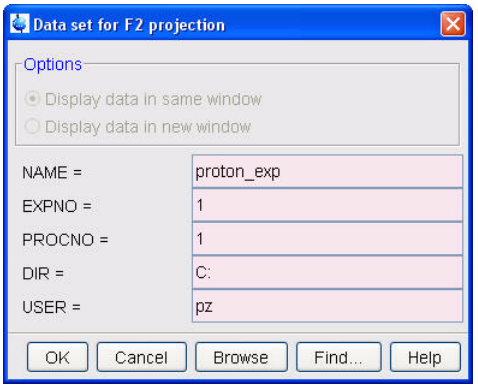
3. Click the right mouse button inside the F2 projection

Figure 8.4.



4. Select '**External Projection**' by clicking on it

Figure 8.5.



5. Make the following changes:

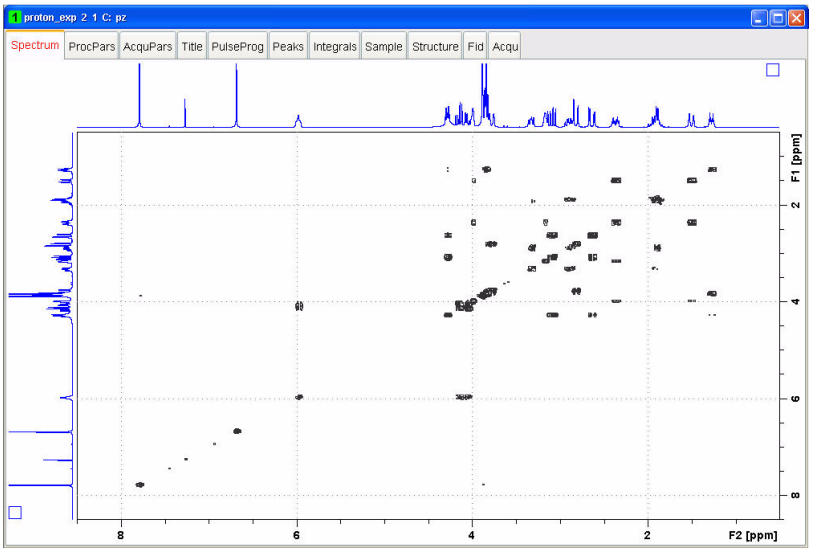
EXPNO = 1 (Experiment number of the 1-D Preparation experiment)

6. Click on OK

7. Click the right mouse button inside the F1 projection

8. Repeat steps 3 through 7

Figure 8.6.

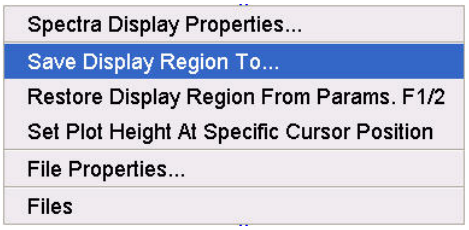


Plotting *8.1.5*

1. Use the *2 /2 *8 /8 buttons to adjust for a suitable contour level

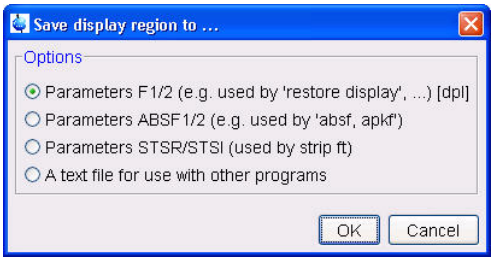
2. Click the right mouse button inside the 2-D contour display

Figure 8.7.



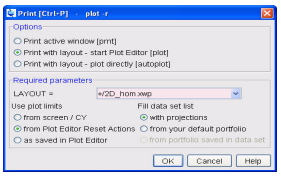
3. Select '**Save Displayed Region To...**' by clicking on it

Figure 8.8.



4. Select '**Parameters F1/2 [dpl]**' by enabling the radio button
5. Click on OK
6. In the main menu click on '**File**'
7. Select '**Print**' by clicking on it

Figure 8.9.



8. Enable the following options:

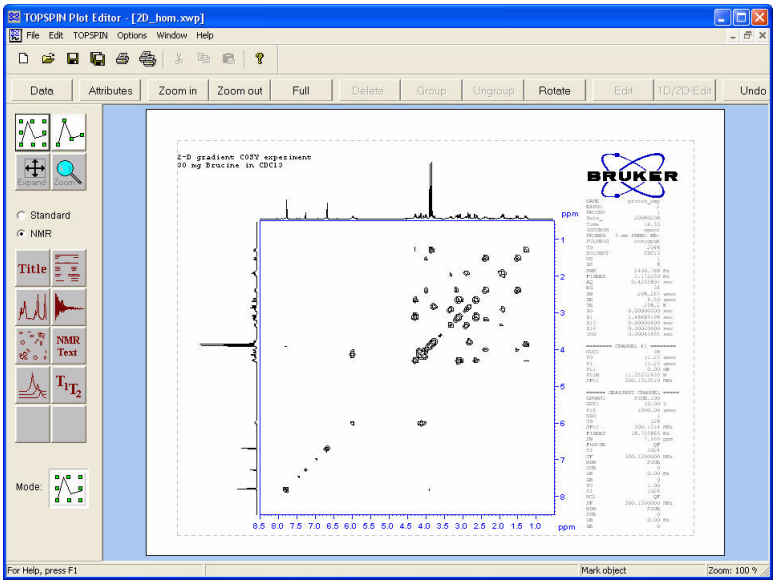
   Print with layout-start Plot Editor

   from Plot Editor Reset Actions

   with projections

9. Select LAYOUT = **+/2D_hom.xwp**
10. Click on OK

Figure 8.10.



15. In the Plot Editor's main menu, click in '**File**'
16. Select '**Print**' by clicking on it

2-D phase sensitive NOESY experiment *8.2*

Sample:

30 mg Brucine in CDCl3

Preparation experiment *8.2.1*

1. Follow the instructions in 8.1.1 Preparation experiment, steps 1 through 9

Setting up the NOESY experiment *8.2.2*

1. Type **rpar NOESYPHSW all**
2. Turn the spinner off

NOTE: 2-D experiments should be run non spinning

3.Select the '**AcquPars**' tab by clicking on it

4. Make the following changes:

NS = **8**

TD (F1) = **128**

SW [F2] = e.g. **8** (value from step 6, Preparation experiment 9.1.1)

SW [F1] = same exact value as SW (F2)

O1 [Hz] = e.g. **1351** (value from step 7, Preparation experiment 9.1.1)

5. Click on  to read in the Prosol parameters

6. Click on  to display the pulsprogram parameters

7. Make the following changes:

D1 [s] = **2**

D8 [s] = **0.700**

NOTE: The mixing time D8 is dependent on the size of the Molecule and the magnetic strength. It can vary from a large Molecule to a small one from 100 ms to 800 ms.

8. Select the '**ProcPar**' tab by clicking on it

9. Make the following changes:

SR [F2] = e.g. **0** (value from step 9, Preparation experiment 8.1.1)

SR [F1] = e.g. **0** (value from step 9, Preparation experiment 8.1.1)

PHC0 [degree] (F1)= **90**

PHC1 [degree] (F1)= **-180**

FCOR (F1) = **1**

10. Select the '**Title'** tab by clicking on it

11. Make the following changes:

2-D phase sensitive NOESY
30 mg Brucine in CDCl3

12. Select the '**Spectrum**' tab by clicking on it

Acquisition *8.2.3*

1. Type **rga**

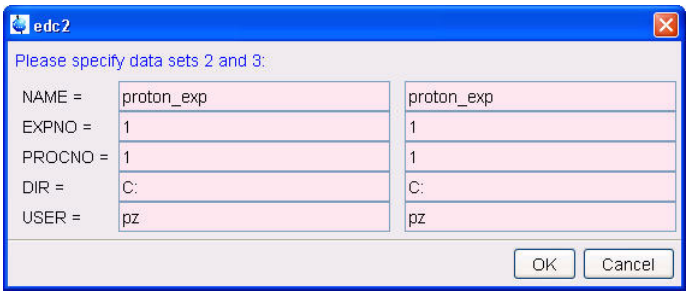
2. Type **zg** to start the acquisition

Processing *8.2.4*

The standard Bruker parameter sets are optimized to run under complete automation through the use of AU programs. The name of the AU program is entered in the acquisition (eda) and processing (edp) parameter lists, as AUNM. To start the acquisition, the command xaua may be used. For executing the processing AU program the command xaup may be used.

1. Type **edc2**

Figure 8.11.

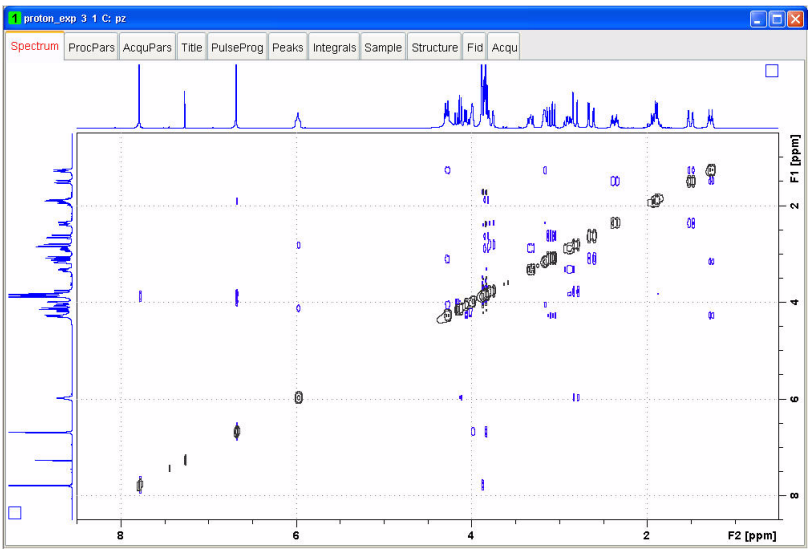


2. Enter the EXPNO and PROCNO of the Preparation experiment 10.1.1 into the first and second column (data set 2 and 3)

3. Click on OK

4. Type **xaup**

Figure 8.12.



Notes: