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

Basic Experiments User Guide

Version 002

The information in this manual may be altered without notice.

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DWG-Nr.: 002

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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 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 succested in this manual may not be suitable for all systems (e.g. Cryo probes) and could cause damage to the unit. Therfore 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.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

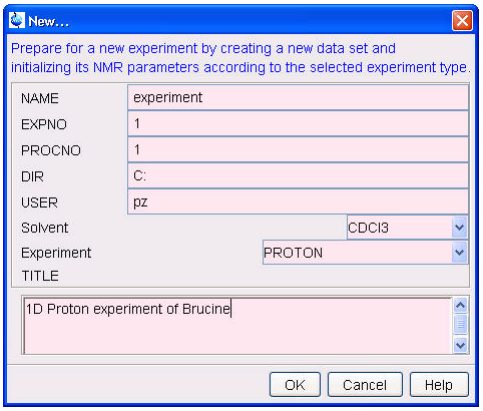
Sample:

30 mg Brucine in CDCl3

Experiment setup 2.2.1

1. Click on  and change the following parameters

Figure 2.1.



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

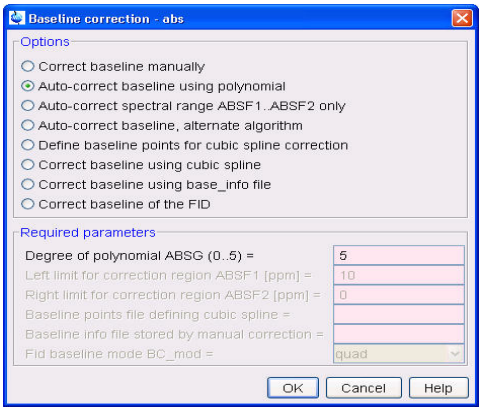
Acquisition *2.2.2*

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

Processing *2.2.3*

1. Process and Phase correct the spectrum
2. In the main menu click on '**Processing**' and select '**Baseline Correction**'

Figure 2.2.



3. Enable '**Auto-correct baseline using polynomial**'
4. Click on OK
5. Expand the spectrum (all peaks in display)
6. Click on 

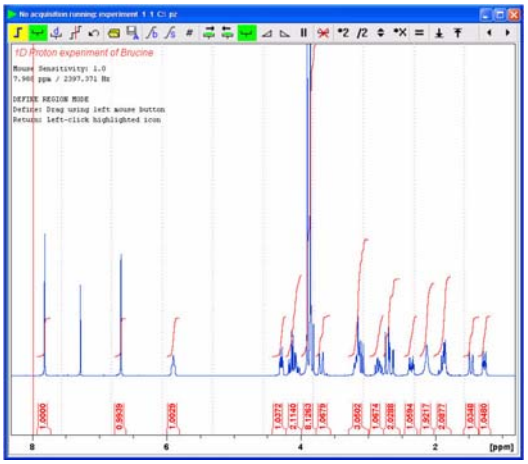
NOTE: As part of the automatic baseline correction (abs), the spectrum is integrated using the default parameters: azfe, azfw and isen. For a user defined integration, follow the steps below.

7. In the integration menu bar click on  to select all regions
8. In the integration menu bar click on  to delete the selected regions

Figure 2.3.

9. Click on OK
10. In the Integration menu bar click on 
11. 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
12. Repeat step 8 for the remainder of the peaks

Figure 2.4.

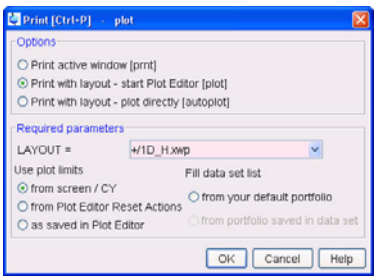


13. Click on  to save the integration region

Plotting the 1D Proton spectra *2.2.4*

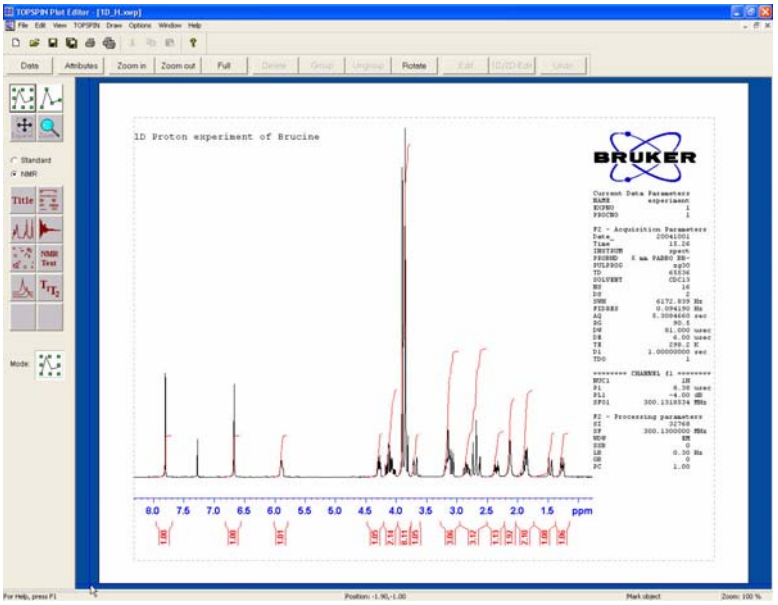
1. In the main menu click on '**File**' and select '**Print'** by clicking on it

Figure 2.5.



2. Enable '**Print with layout - start Plot Editor (plot)**'
3. Select the '**LAYOUT +/1D_H.xwp**'
4. Enable '**from screen/CY**'
5. Click on 

Figure 2.6.



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

1-D Carbon Experiment *2.3*

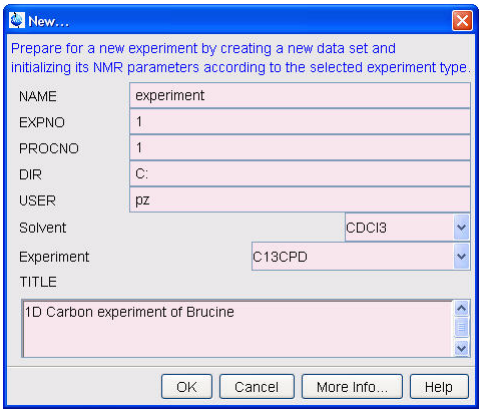
Sample:

30 mg Brucine in CDCl3

Experiment set up *2.3.1*

1. Click on  and change the following parameters

Figure 2.7.



2. Click on 
3. Insert the sample
4. Click on  to display the Lock display

5. In the lock display window click on  and select CDCl3
6. Tune the probe
7. Shim for best homogeneity
8. In the lock display window click on  to close the window
9. Select the '**AcquPars**' tab by clicking on it
10. Make the following change

NS = **128**

11. Click on  to read in the Prosol parameters

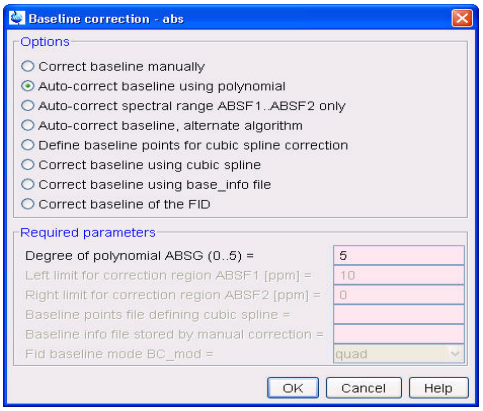
Acquisition *2.3.2*

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

Processing *2.3.3*

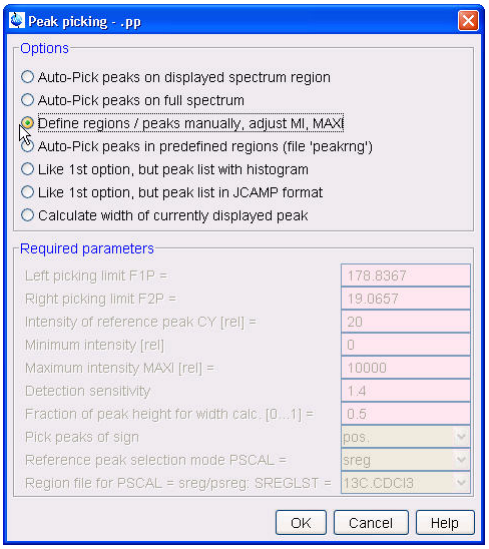
1. Process and Phase correct the spectrum
2. Type **abs**
3. In the main menu click on '**Processing**' and select '**Baseline Correction**'

Figure 2.8.



4. Enable '**Auto-correct baseline using polynomial**'
5. Click on 
6. Expand the spectrum (all peaks in display)
7. In the main menu click on '**Analysis**' and elect '**Peak Picking...[pp]**' by clicking on it

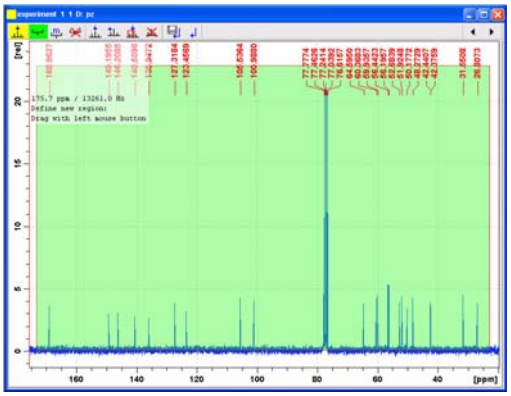
Figure 2.9.



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

9. Click on 

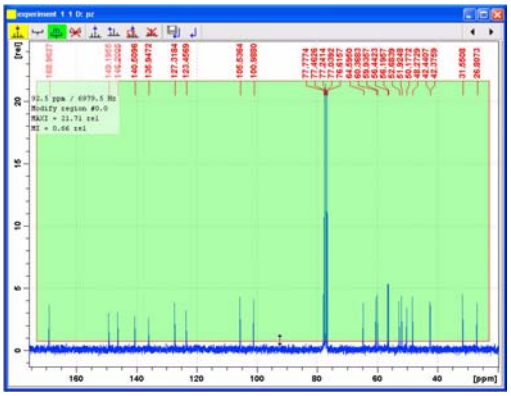
Figure 2.10. 1



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

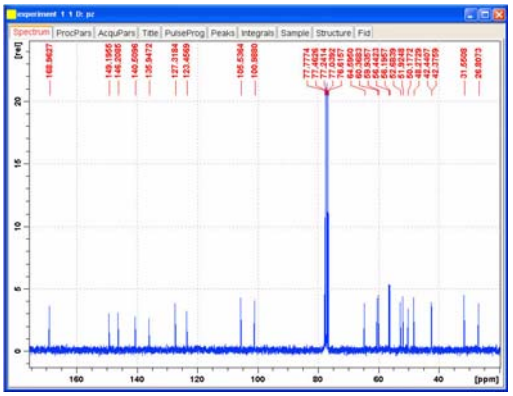
11. Click on 

Figure 2.11.



12. 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
13. Click on 
14. Click on 

Figure 2.12.

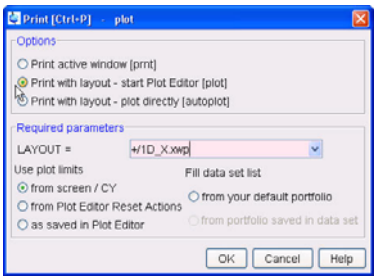


NOTE: To display the peak picking labels, right click inside the spectrum window and select ‘Display Properties’. Enable ‘Peak labels’ and click ‘OK’

Plotting the 1D Carbon spectrum *2.3.4*

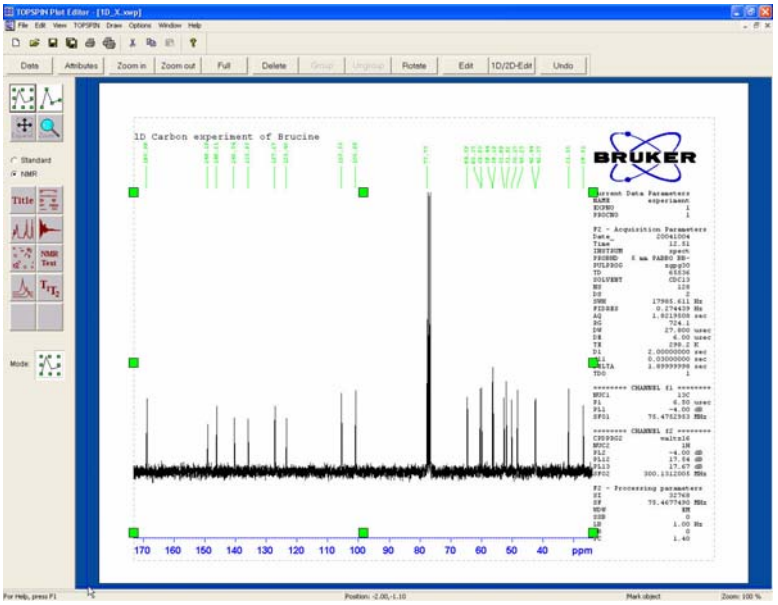
1. In the main menu click on ‘**File**’ and select ‘**Print**’ by clicking on it

Figure 2.13.



2. Enable ‘**Print with layout - start Plot Editor (plot)**’
3. Select the ‘**LAYOUT +/1D_X.xwp**’
4. Enable ‘**from screen/CY**’
5. Click on 

Figure 2.14.



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

DEPT-135 Experiment *2.4*

Sample:

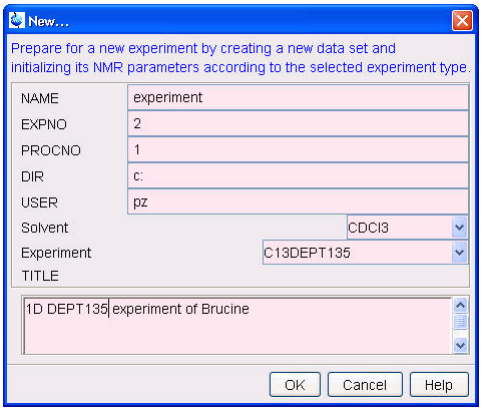
30 mg Brucine in CDCl3

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. Click on  and change the following parameters

Figure 2.15.



2. Click on OK

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

4. Mark the following change

NS = **64**

5. Click on  to read in the Prosol parameters

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

Acquisition *2.4.2*

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

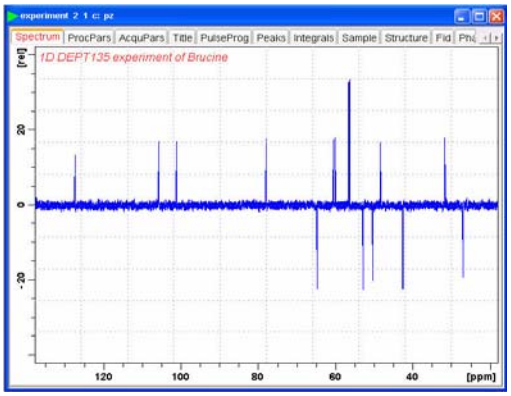
2. Click on  to start the acquisition

Processing *2.4.3*

1. Process and Phase correct the spectrum

2. Type **abs**

Figure 2.16.

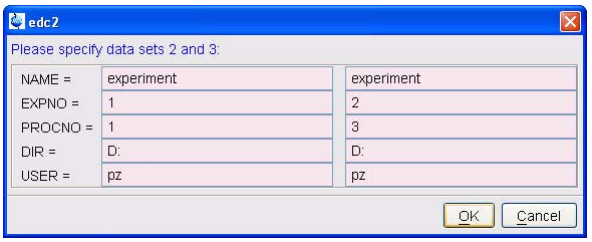


NOTE: To properly phase the DEPT135 spectrum be sure the CH2 are negative and the CH and CH2 are positive phased.

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

1. Type edc2 on the command line

Figure 2.17.

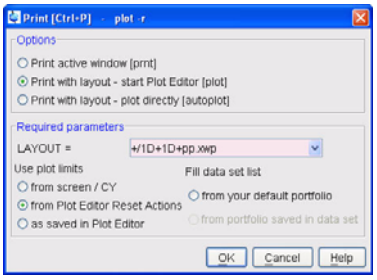


2. Enter the EXPNO and PROCNO of the 1D 13C spectrum into the first column (data set 2)

3. Click on 

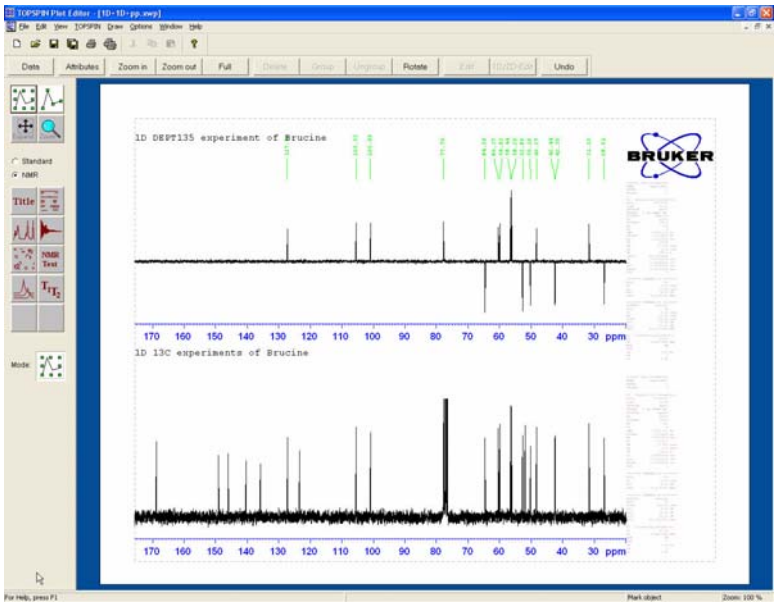
4. In the main menu click on '**File**' and select '**Print**' by clicking on it

Figure 2.18.



5. Enable '**Print with layout - start Plot Editor (plot)**'
6. Select the LAYOUT **+/1D+1D+pp.xwp**'
7. Enable '**from Plot Editor Reset Actions**'
8. Click on 

Figure 2.19.



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.

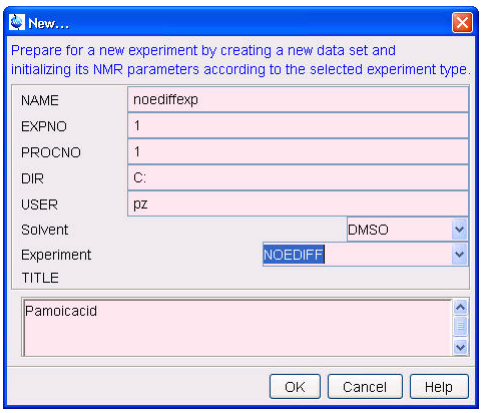
Sample:

40 mg Pamoic acid in DMSOd6

Preparation experiment 3.1.1

1. Click on  and change the following parameters

Figure 3.1.



2. Click on 
3. Insert the sample
4. Click on  to display the Lock display
5. In the lock display window click on  and select DMSO

6. Turn the spinner off

NOTE: noe experiments should be run non spinning

7. Shim for best homogeneity
8. In the lock display window click on  to close the window
9. Select the '**AcquPars**' tab by clicking on it
10. Make the following change:

O2p [ppm] = **-4**

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

LB [Hz] = **1**

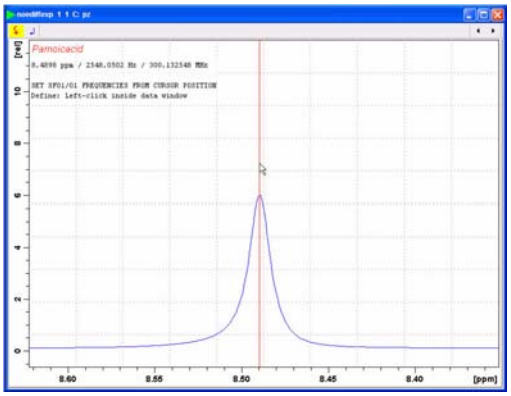
14. Tune the probe
15. In the main menu click on '**Spectrometer**', select '**Adjustment**' and click on '**Auto-adjust receiver gain**' or type **rga**
16. Click on  to start the acquisition
17. Process and Phase correct the spectrum

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

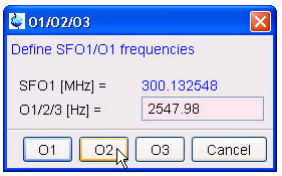
1. Expand the peak around 8.5 ppm
2. Click on 

Figure 3.2.



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

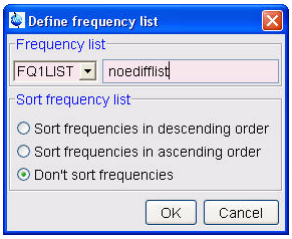
Figure 3.3.



5. Click on 

6. Click on 

Figure 3.4.



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

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

9. Click on 

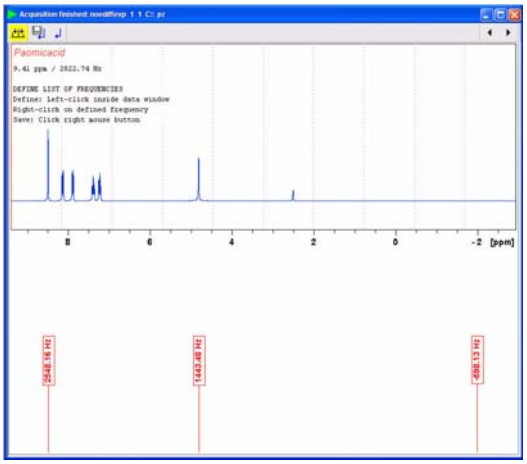
10. Move the cursor line to –2ppm and click the left mouse button to assign the off resonance frequency

11. Using the  tools to expand the peak at 8.5 ppm

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

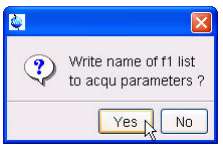
13. Repeat steps11 through 12 to assign the frequency for the peak at 4.8ppm

Figure 3.5.



14. Click on  to save the frequency list

Figure 3.6.



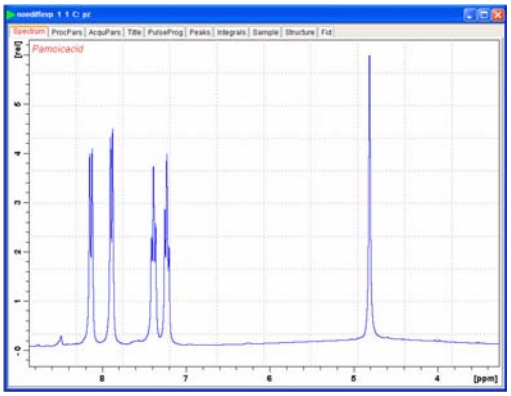
15. Click on 

Fine tuning *3.1.3*

1. Click on  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.

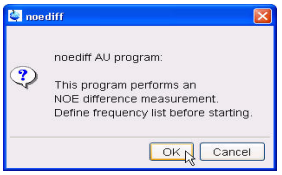
Figure 3.7.



Running the experiment *3.1.4*

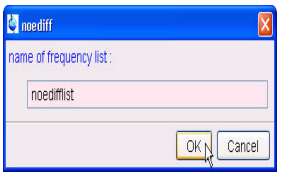
1. Type **noediff** on the command line

Figure 3.8.



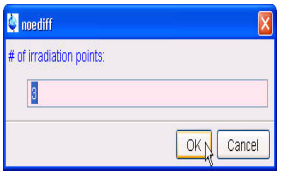
2. Click on OK

Figure 3.9.



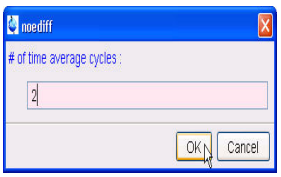
3. Click on OK

Figure 3.10.



4. Click on OK

Figure 3.11.

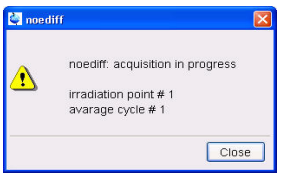


5 Change the # of time average cycles = **2**

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 again for a total of 16 scans on each experiment.

6 Click on OK

Figure 3.12.



Processing *3.1.5*

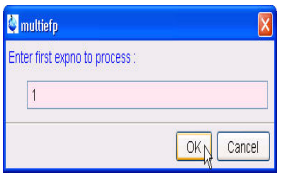
1.Start with experiment # 1

2. Type **ef**

3. Correct the phase very carefully

4. Type **multiefp**

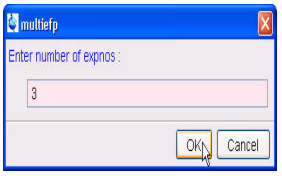
Figure 3.13.



5. Enter **1** for the first experiment number

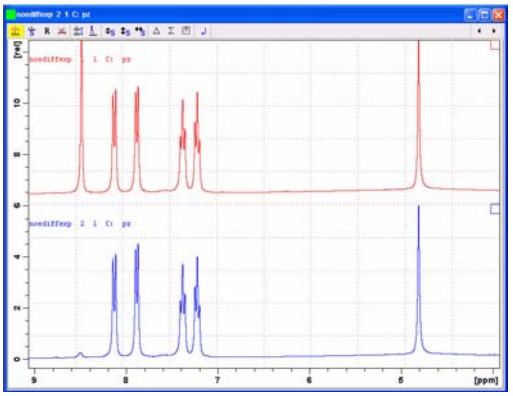
6. Click on OK

Figure 3.14.



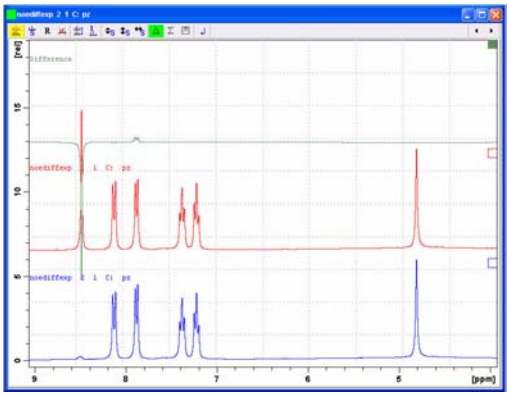
7. Enter **3** for the # of experiments
8. Click on OK
9. Drag experiment # 2 into the display window or type **re 2** in the command line
10. Click on
11. Drag experiment # 1 into the display window or type **re 1** in the command line

Figure 3.15.



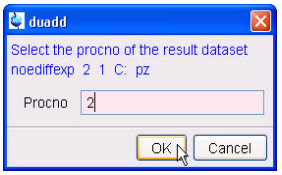
12. Click on

Figure 3.16.



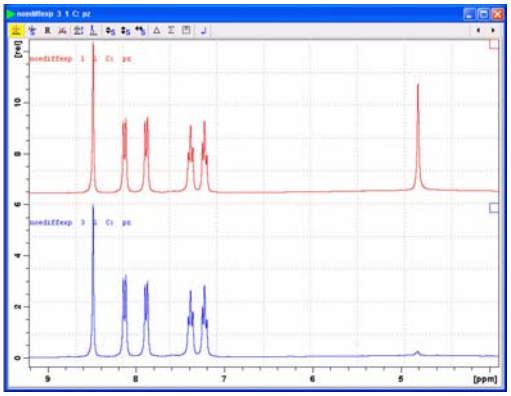
13. Click on

Figure 3.17.



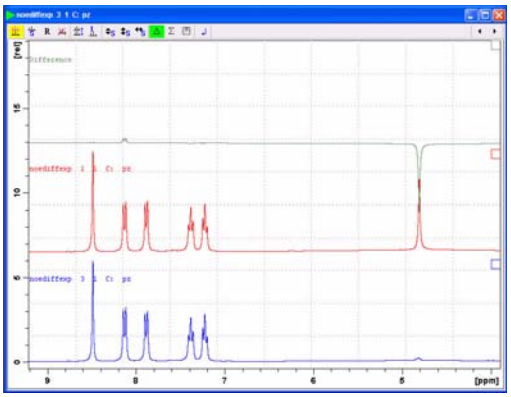
14. Enter **2** for the processing #
15. Click on OK
16. Click on 
17. Drag experiment # 3 into the display window or type **re 3** in the command line
18. Click on 
19. Drag experiment # 1 into the display window or type **re 1** in the command line

Figure 3.18.



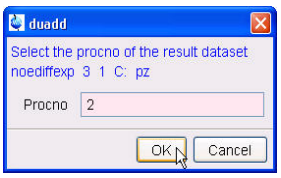
20. Click on 

Figure 3.19.



21. Click on 

Figure 3.20.



22. Enter **2** for the Procno
23. Click on OK
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. In the Integration menu bar click on  to define a integration region

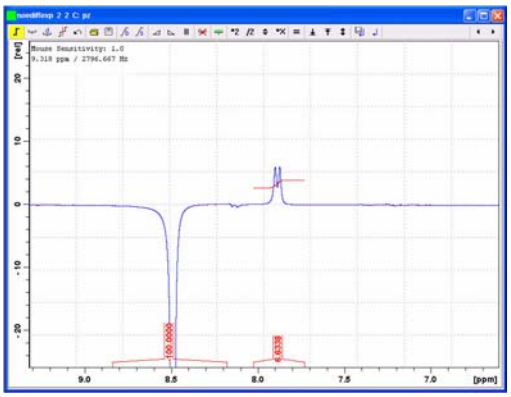
4. Define the regions by clicking the left mouse button and the use of the cursor lines

5. Click on  again

6. move the cursor line in to the region of the negative peak, click the right mouse button and select calibrate from the popup window

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

Figure 3.21.

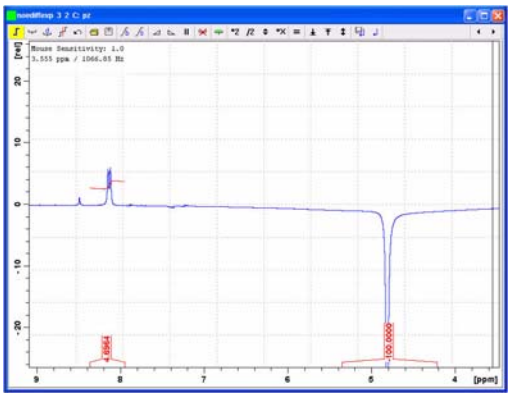


8. Click on 

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 3.22.



11. Click on 

1-D NOE Difference Experiment

Solvent Suppression Experiments

4

Introduction 4.1

Three different solvent suppression technics: Presaturation, Presaturation with composite pulses, WATERGATE and Excitation Sculpting are discussed in this chapter

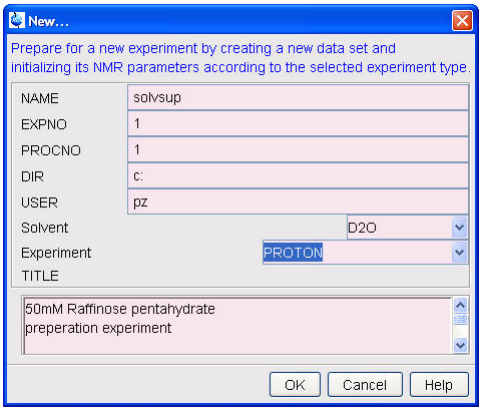
Sample:

50 mM Raffinose pentahydrate in 90% H2O / 10% D2O

Reference spectrum 4.1.1

1. Click on  and change the following parameters

Figure 4.1.



2. Click on 
3. Insert the sample
4. Click on  to display the Lock display
5. In the lock display window click on  and select D2O

6. Turn the spinner off

NOTE: solvent suppression experiments should be run non spinning

7. Tune the probe
8. Shim for best homogeneity
9. In the lock display window click on  to close the window
10. Select the '**AcquPars**' tab by clicking on it
11. Click on  to read in the Prosol parameters
12. Select the '**Spectrum**' tab by clicking on it

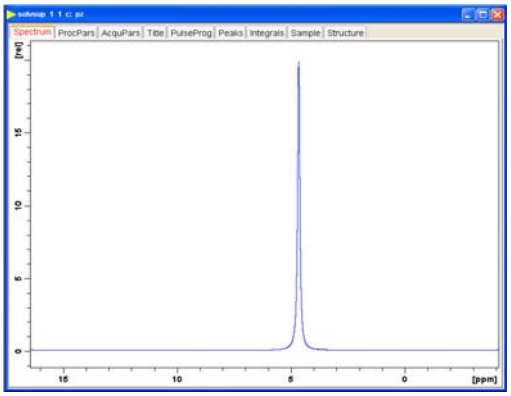
Acquisition *4.1.2*

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

Processing *4.1.3*

1. Process and Phase correct the spectrum

Figure 4.2.



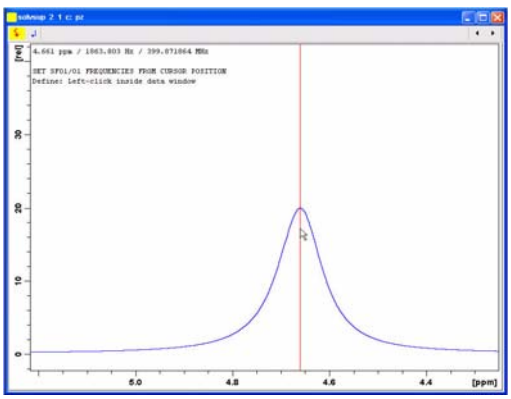
NOTE: Make sure that the SW is large enough to cover the entire Spectrum accounting for the position of O1. The presaturation is applied on resonance (at the O1 position) The power level for presaturation has to be known and entered into the Prosol parameters.

Presaturation *4.2*

Parameter set up *4.2.1*

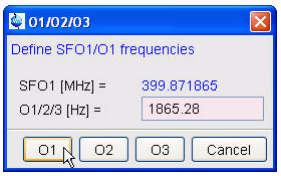
1. Type **wrpa 2** on the command line
2. Type **re 2** on the command line
3. Expand the Water signal at 4.8 ppm
4. Click on 

Figure 4.3.



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

Figure 4.4.



6. 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**

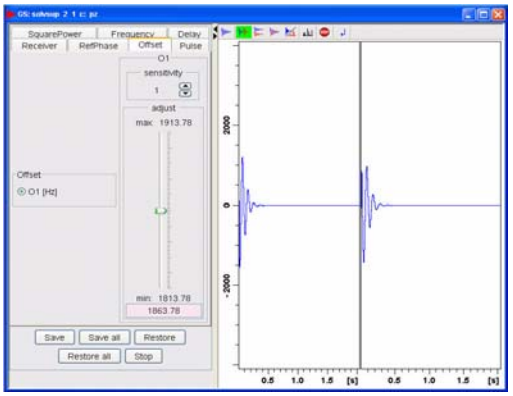
13. Select the '**Spectrum**' tab by clicking on it

Fine tuning *4.2.2*

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

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

Figure 4.5.



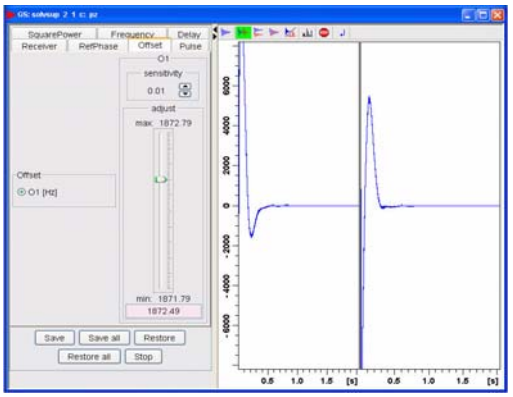
3. click on 

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

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

5. 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.



6. Click on 

7. Click on 

Acquisition *4.2.3*

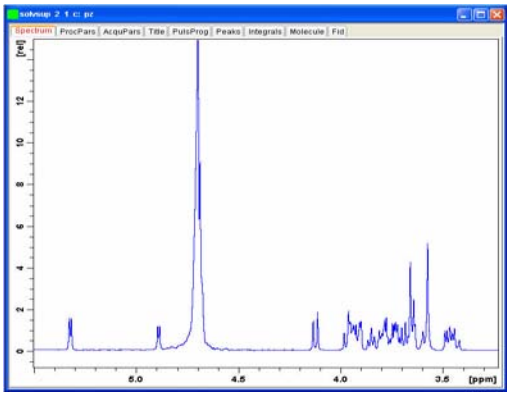
1. In the main menu click on ‘**Spectrometer**’, select ‘**Adjustment**’ and click on ‘**Auto-adjust receiver gain**’ or type **rga**

2. Click on  to start the acquisition

Processing *4.2.4*

1. Process and Phase correct the spectrum

Figure 4.7.



Presaturation with Composite Pulses *4.3*

Parameter set up *4.3.1*

1. Follow the instructions in paragraph 5.1.1 through 5.2.2 step 7 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

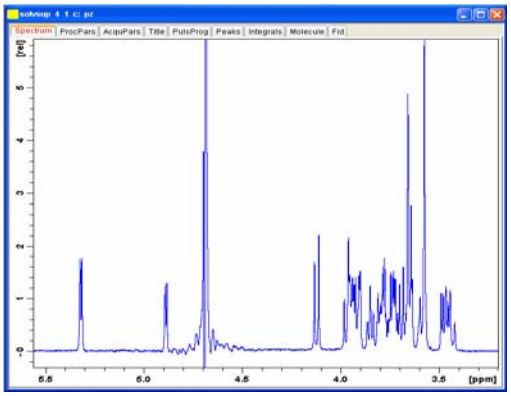
Acquisition *4.3.2*

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

Processing *4.3.3*

1. Process and Phase correct the spectrum

Figure 4.8.



Solvent suppression with WATERGATE *4.4*

Parameter set up *4.4.1*

1. Follow the instructions in the paragraphs 5.1.1 through 5.2.1 step 13
2. Make the following changes:

PULPROG = **p3919gp**

TD = **32k**

NS = **8**

DS = **4**

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

D1 [s] = **2**

D19 [s] = **0.00015**

GPZ1 [%] = **20**

5. Select the '**ProcPar**' tab by clicking on it
6. Make the following changes:

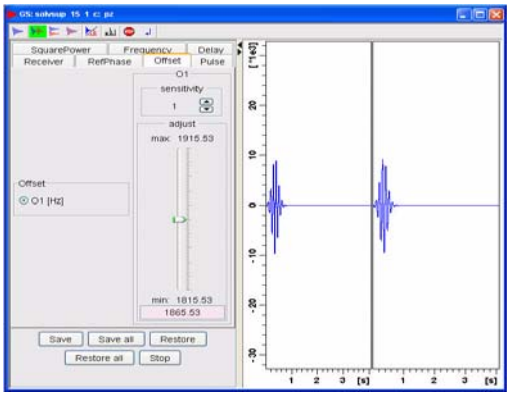
SI = **16k**

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

Fine tuning *4.4.2*

1. In the main menu click on '**Spectrometer**', select '**Adjustment**' and click on '**Auto-adjust receiver gain**' or type **rga**
2. In the main menu click on '**Spectrometer**', select '**Adjustment**' and click on '**Start acquisition, adjust params [gs]**' or type **gs**

Figure 4.9.



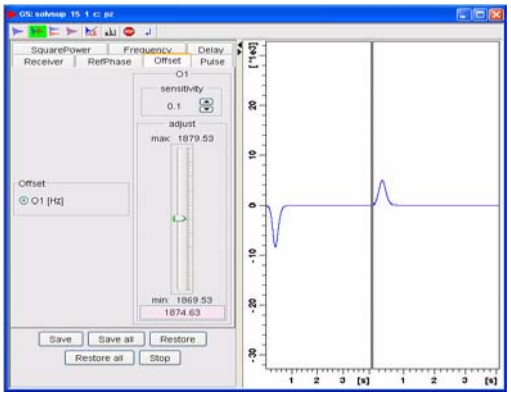
3. click on 

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

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

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

Figure 4.10.



6. Click on 

7. Select the ‘**Pulse**’ tab in the gs display window

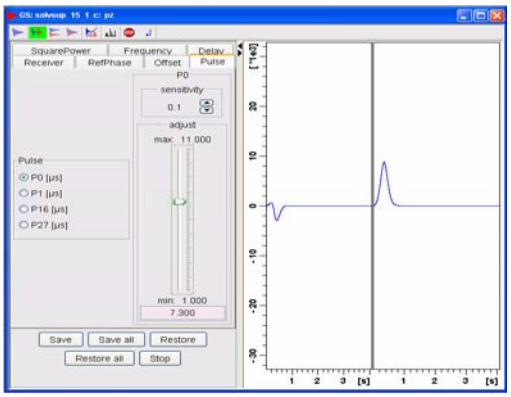
8. Enable **P0 [us]** by clicking on the radio button

9. Change the P0 value by clicking just below or above the adjust slider

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

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

Figure 4.11.



11. Click on Save

12. Click on Stop

Acquisition *4.4.3*

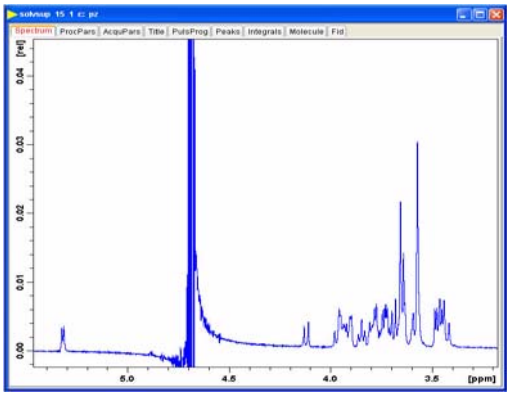
1. In the main menu click on ‘**Spectrometer**’, select ‘**Adjustment**’ and click on ‘**Auto-adjust receiver gain**’ or type **rga**

2. Click on ▶ to start the acquisition

Processing *4.4.4*

1. Process and Phase correct the spectrum

Figure 4.12.



Solvent suppression with excitation sculpting 4.5

Parameter se up 4.5.1

1. Follow the instructions in the paragraphs 5.1.1 through 5.2.1 step 13
2. Make the following changes:

PULPROG = **zgesgp**

TD = **32k**

NS = **8**

DS = **4**

3. Click on  to read in the Prosol parameters
4. Click on  to display the pulsprogram parameters
5. Make the following changes:

D1 [s] = **2**

GPZ1 [%] = **31**

GPZ2 [%] = **11**

6. Select the '**ProcPar**' tab by clicking on it
7. Make the following changes:

SI = **16k**

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

Acquisition 4.5.2

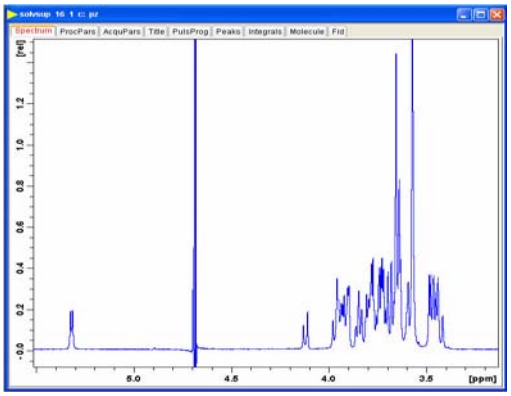
1 In the main menu click on '**Spectrometer**', select '**Adjustment**' and click on '**Auto-adjust receiver gain**' or type **rga**

2. Click on  to start the acquisition

Processing *4.5.3*

1. Process and Phase correct the spectrum

Figure 4.13.



Fine tuning *4.5.4*

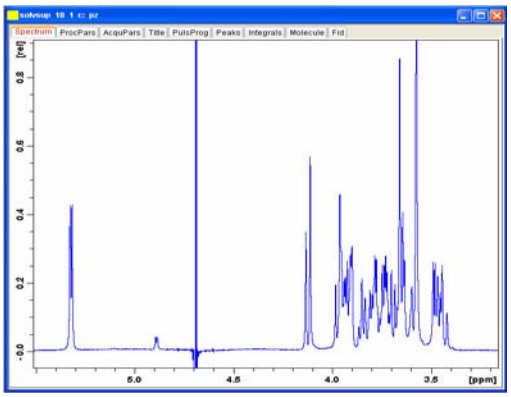
1. Select the '**AcquPars**' tab by clicking on it
2. Click on  to display the pulsprogram parameters
3. Make the following changes:

P12 [us] = **4000**

SP1 [dB] = calculate using the AU program 'calcpowlev'

4. In the main menu click on '**Spectrometer**', select '**Adjustment**' and click on '**Auto-adjust receiver gain**' or type **rga**
5. Click on  to start the acquisition
6. Process and Phase correct the spectrum

Figure 4.14.



T1 Experiment 5

Introduction 5.1

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

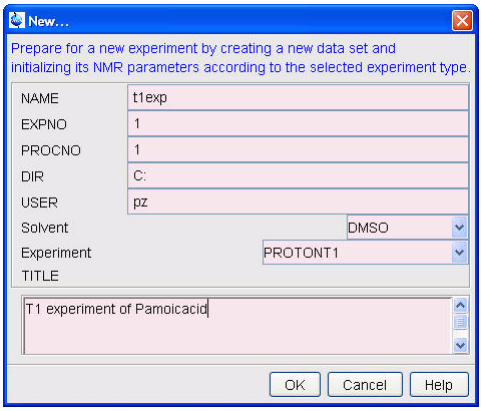
Sample:

30mg Pamoic acid in DMSOd6

Parameter set up 5.1.1

1. Click on  and set the following parameters:

Figure 5.1.



2. Click on 
3. Insert the sample
4. Click on  to display the Lock display
5. In the lock display window click on  and select DMSO

6. Turn the spinner off

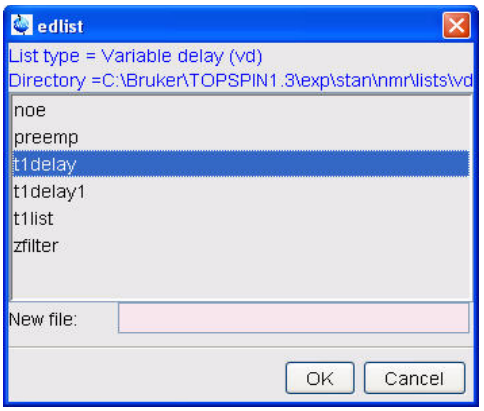
NOTE: T1 experiments should be run non spinning

7. Shim for best homogeneity
8. Tune the probe
9. In the lock display window click on  to close the window
10. Select the '**AcquPars**' tab by clicking on it
11. Click on  to read in the Prosol parameters
12. Change TD in F1 = **10**

NOTE: The value of TD in F1 is the number of delays used.

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

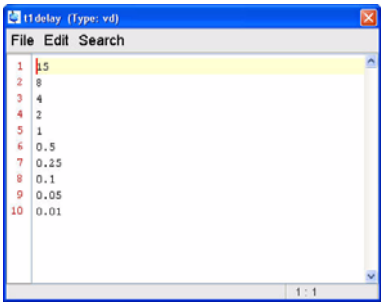
Figure 5.2.



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

click on 

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

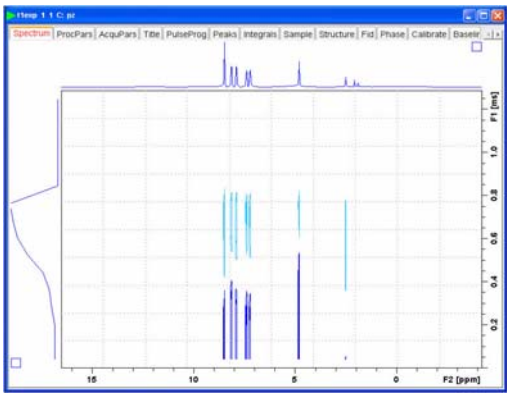
Acquisition *5.1.2*

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

Processing *5.1.3*

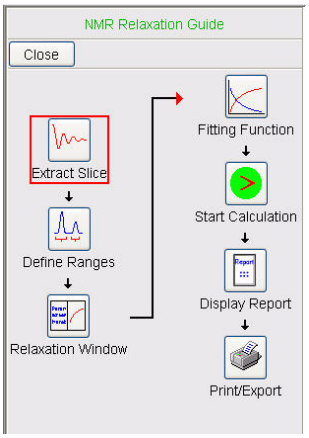
1. Type **xf2** on the command line

Figure 5.4.



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

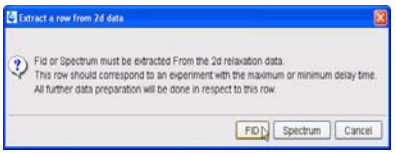
Figure 5.5.



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

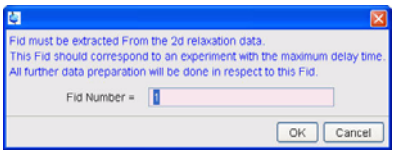
4. In the Guide window, click on  'Extract Slice'

Figure 5.6.



5. Click on 

Figure 5.7.



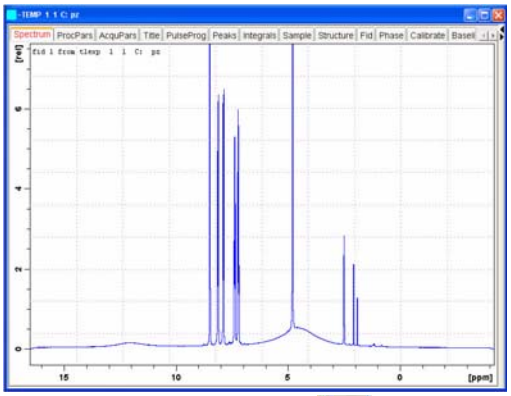
6. Select Fid Number = 1

7. Click on OK

NOTE: The Ft and Phase correction are done automatically. If the spectrum contains broad peaks see Figure 6.8, it may be necessary to perform a additional manual phase correction.

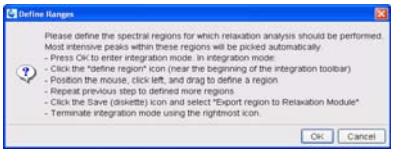
8. Check if phase is correct

Figure 5.8.



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

Figure 5.9.



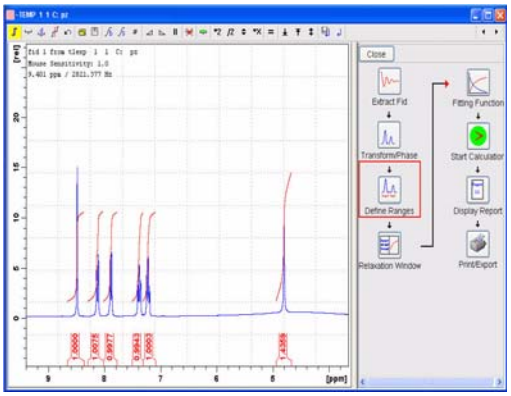
11. Click on OK

12. Click on  to define the regions

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

14. Click on  again

Figure 5.10.

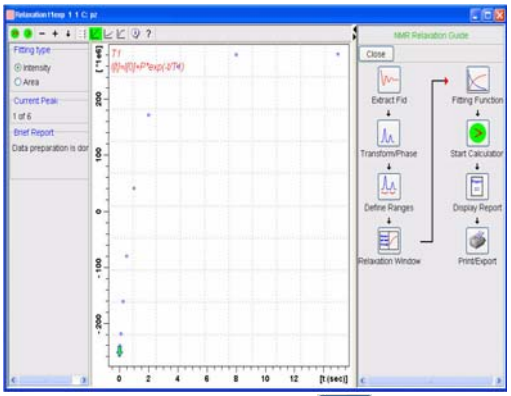


15. Click on 

Figure 5.11.

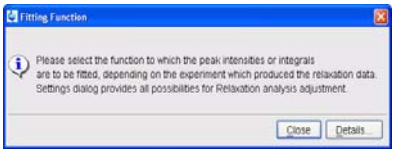
16. Select '**Export Region To Relaxation Module**' by clicking on it
17. In the Guide window, click on  'Relaxation Window'

Figure 5.12.



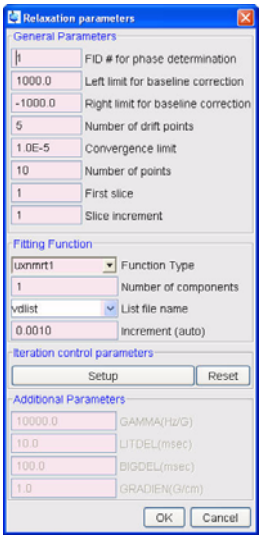
18. In the Guide window, click on  'Fitting Functions'

Figure 5.13.



19. Click on 

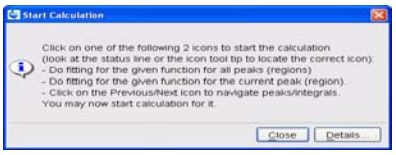
Figure 5.14.



20. Click on 

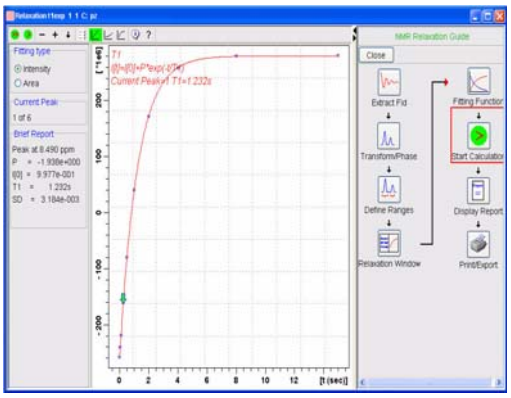
21. In the Guide window, click on  'Start Calculating'

Figure 5.15.



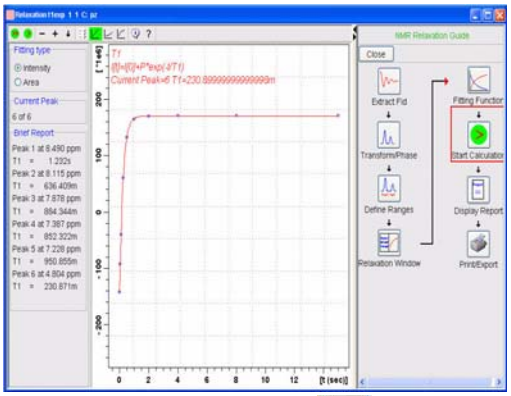
22. Click on 

Figure 5.16.



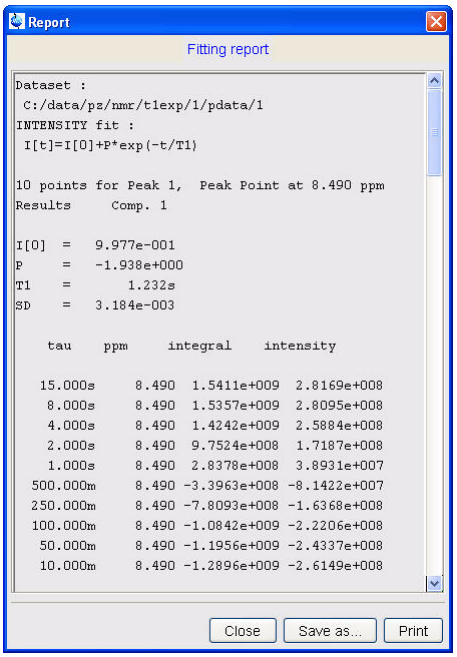
23. In the T1 data display window click on  to calculate all regions

Figure 5.17.



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

Figure 5.18.

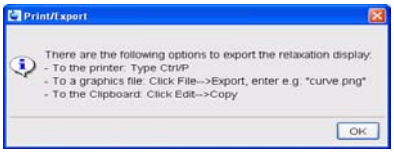


25. Click on 

26. Click on 

27. In the Guide window, click on  Print/Export

Figure 5.19.



28. Use the '**Ctrl**' and the '**p**' keys to print the data

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.

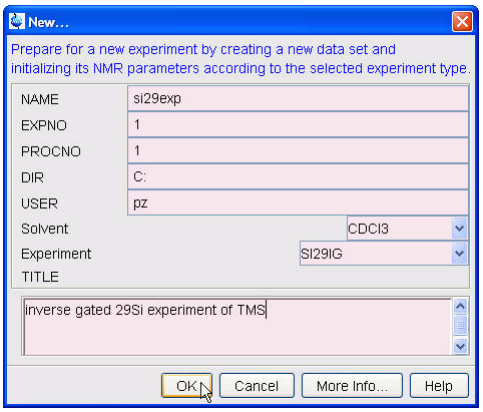
Preparation 6.1.1

Sample:

30% TMS in CDCl3

1. Click on  and change the following parameters

Figure 6.1.



2. Click on 
3. Insert the sample
4. Click on  to display the Lock display
5. In the lock display window click on  and select CDCl3

6. Shim for best homogeneity
7. In the lock display window click on  to close the window
8. Select the '**AcquPars**' tab by clicking on it
9. 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 Close

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 7.3) and the wobble curve (Figure 7.4) appears.

Figure 6.3.

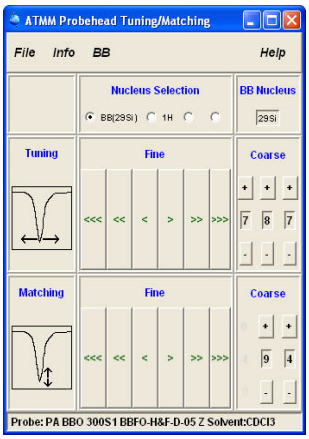
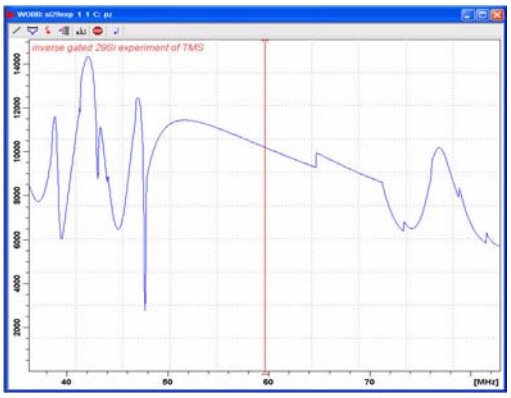


Figure 6.4.



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 7.5) and the arrows are turning red (Figure 7.6) that means the fine tuning capacitor has reached the end. In this case the coarse tuning has to be changed.

Figure 6.5.

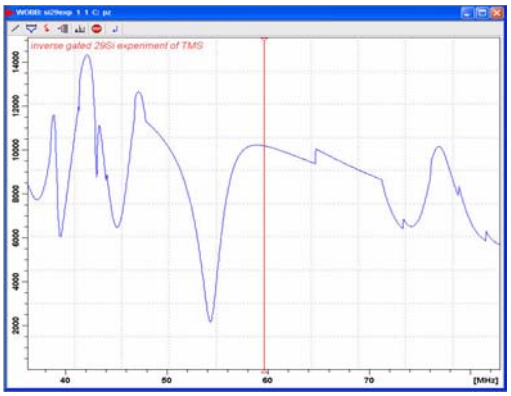
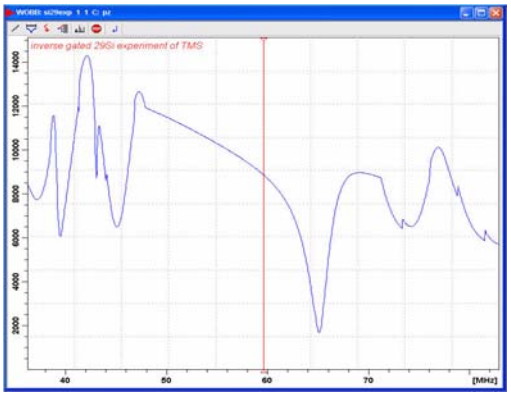


Figure 6.6.

4. Click once on the coarse tuning + button

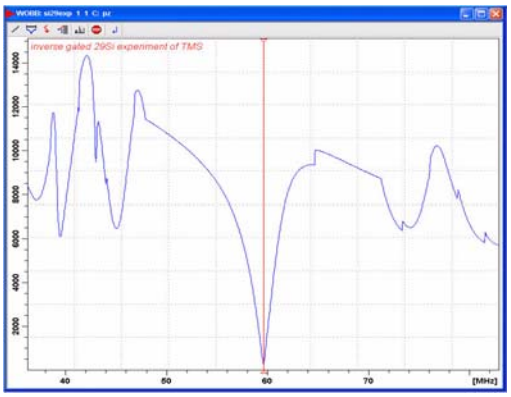
NOTE: The wobble curve jumps to the right side of the red line (Figure 7.7)

Figure 6.7.



5. Clicking on the coarse matching – button and the use of the <<< << < buttons, move the wobble curve on to of the red line (Figure 7.8)

Figure 6.8.



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

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

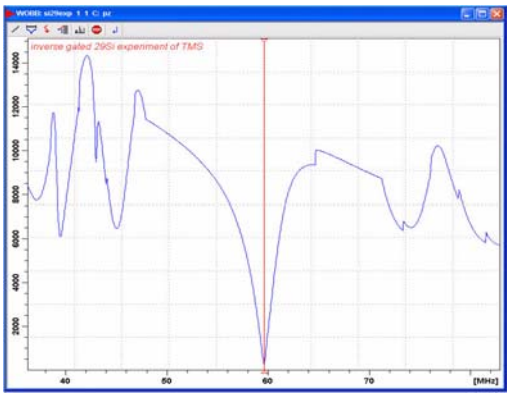
BB-probes without ATM

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 6.9.



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

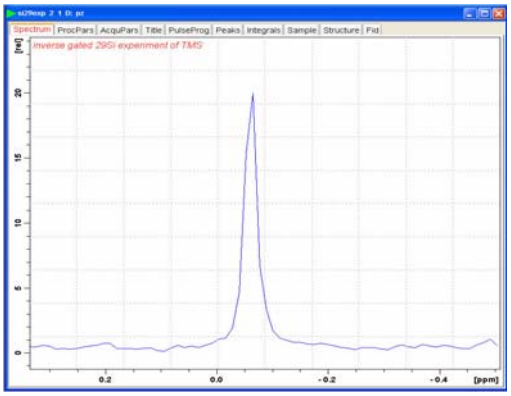
1. Type **iexpno**
2. Select the '**AcquPars**' tab by clicking on it
3. Change AQ_mod = qsim
4. Click on  to display the pulsprogram parameters
5. Make the following changes:

NS	=	1
DS	=	0
D1	=	60

6. Select the '**Spectrum**' tab by clicking on it
7. In the main menu click on '**Spectrometer**', select '**Adjustment**' and click on '**Auto-adjust receiver gain**' or type **rga**

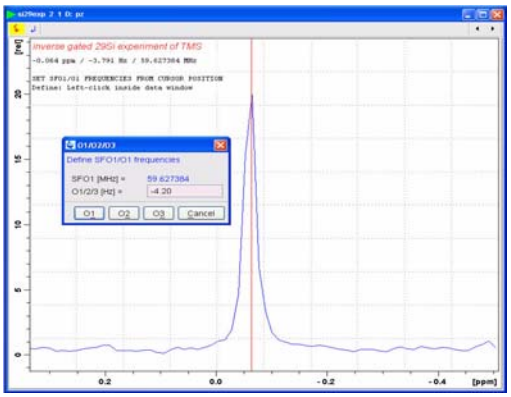
8. Click on  to start the acquisition
9. Process and Phase correct the spectrum
10. Expand the signal region at 0 ppm

Figure 6.10.



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

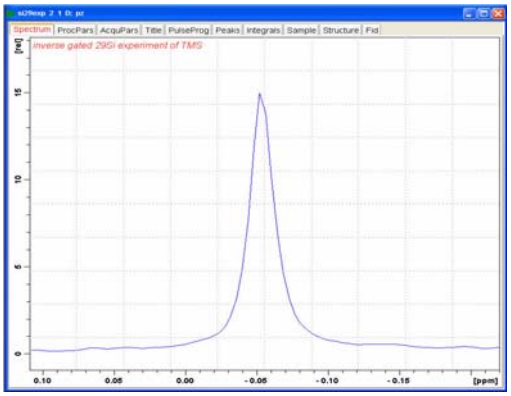
Figure 6.11.



13. Click on 

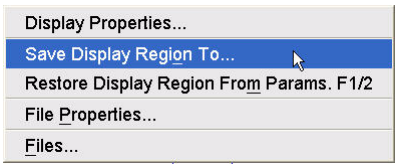
14. Type **swh 1000**
15. Type **td 8k**
16. Type **si 4k**
17. Click on  to start the acquisition
18. Process and Phase correct the spectrum
19. Expand the signal region at 0 ppm

Figure 6.12.



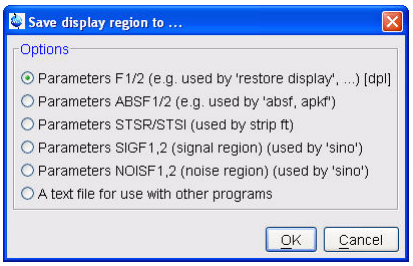
20. Click the right mouse button

Figure 6.13.



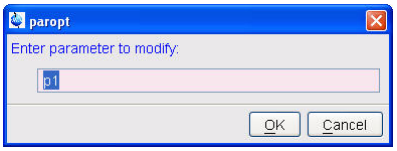
21. Select and click on '**Save Displayed Region To**'

Figure 6.14.



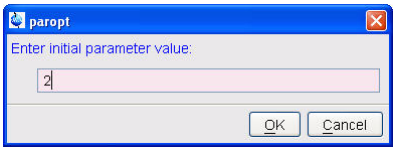
22. Enable '**Parameters F1/2 [dpl]**'
23. Click on OK
24. Type **paropt**

Figure 6.15.



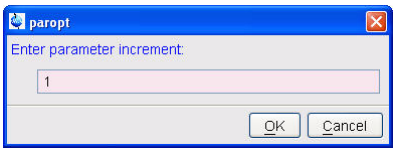
25. Enter **p1**
26. Click on OK

Figure 6.16.



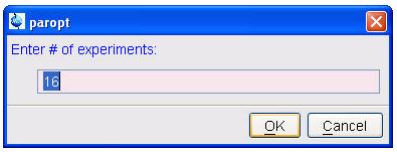
27. Enter **2**
28. Click on OK

Figure 6.17.



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

Figure 6.18.



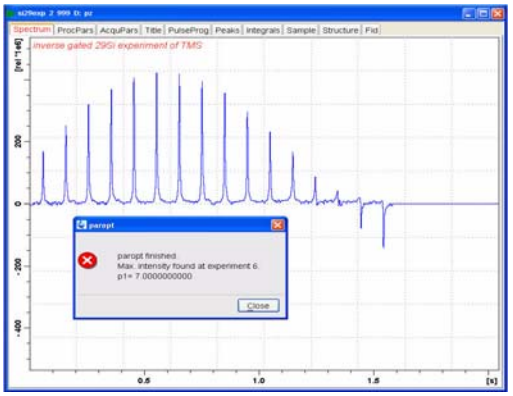
31. Enter 16
32. Click on OK

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 32 - 35below.

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 6.19.



32. Type **re 2 1**

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

34. Click on ▶ to start the acquisition

35. Type **efp**

Change p1 in small increments until the signal goes through a null. Simply divide the new value of the 360 deg. pulse by4. 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!

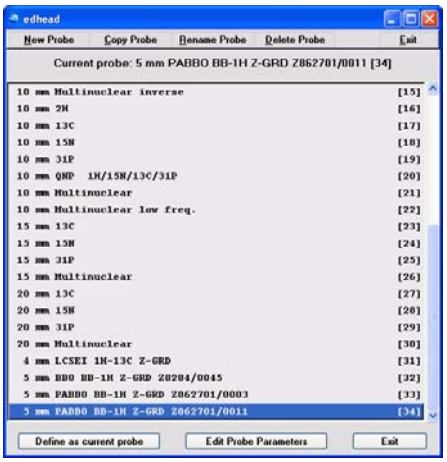
Systems with cortab and power check

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

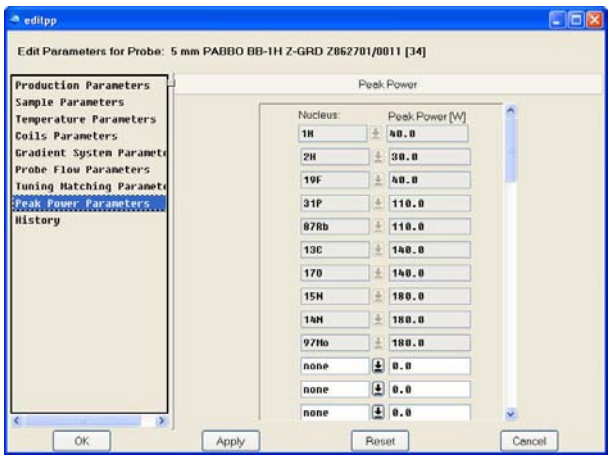
Figure 6.20.



2. Click on 

3. Select Peak Power Parameters

Figure 6.21.



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 6.22.

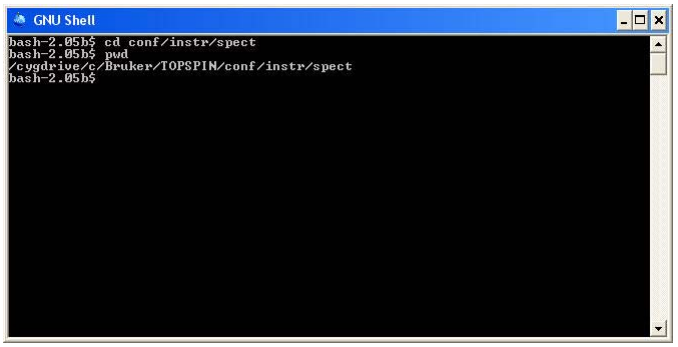
7. Click on Apply
8. Click on OK
9. Click on Exit

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 6.23.



14. Type **cd conf/instr/spect**
15. Type **pwd** to verify to be in the correct directory

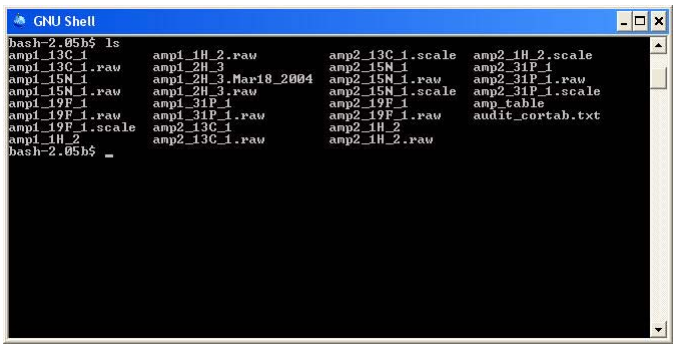
    c/Bruker/TOPSPIN/conf/instr/spect

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 6.24.



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 35 in the previous section, “System without cortab files and power check turned off”

Adding a New Nucleus

Homonuclear Decoupling Experiment

7

Introduction 7.1

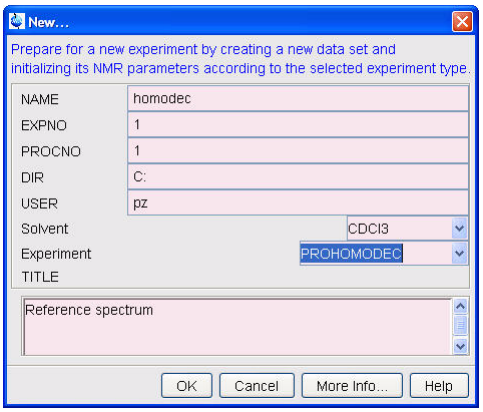
Sample:

0.1% Ethylbenzene in CDCl3

Preparation experiment 7.1.1

1. Click on  and change the following parameters

Figure 7.1.



2. Click on 
3. Insert the sample
4. Click on  to display the Lock display
5. In the lock display window click on  and select CDCl3
6. Tune the probe
7. Shim for best homogeneity
8. In the lock display window click on  to close the window
9. Select the '**AcquPars**' tab by clicking on it
10. Click on  to read in the Prosol parameters
11. Make the following changes:

PULPROG = **zg30**

NS = **8**

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

13. Make the following changes:

LB = **1**

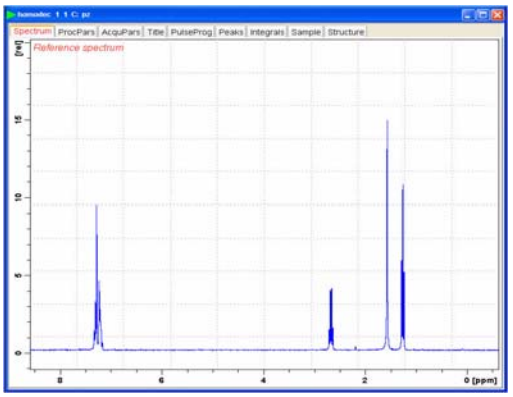
14. In the main menu bar, click on '**Spectrometer**', select '**Adjustment**' and click on '**Auto-adjust receiver gain**' or type **rga**

15. Click on  to start the acquisition

16. Process and Phase correct the spectrum

17. Type **abs**

Figure 7.2.



Parameter set up *7.1.2*

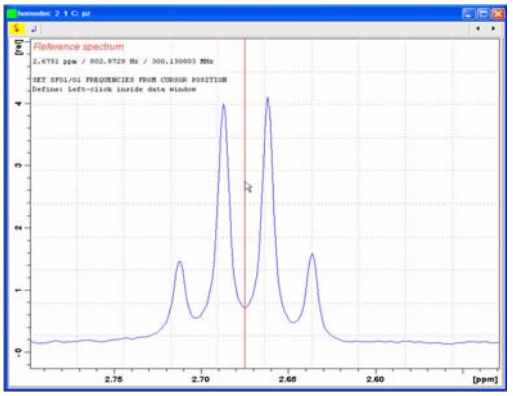
1. Type **wrpa 2** on the command line

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

3. Expand the quartet at 2.65ppm

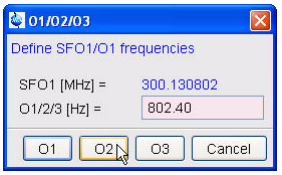
4. Click on 

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 O2
7. Select the '**AcquPars**' tab by clicking on it
8. Make the following changes:

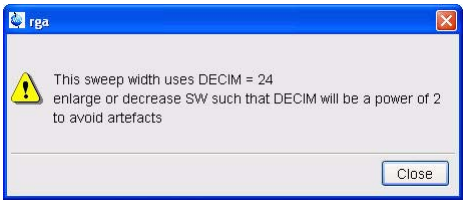
PULPROG = **zghd**

9. Select the '**Title**' tab by clicking on it
10. Change the title to: **Decoupled spectrum**

Acquisition *7.1.3*

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

Figure 7.5.

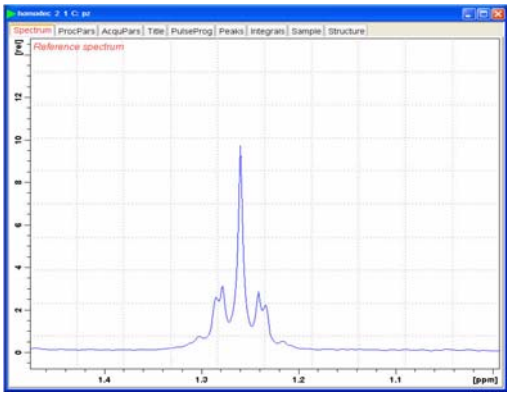


2. Adjust the sweep width if necessary
3. Click on ▶ to start the acquisition

Processing *7.1.4*

1. Process and Phase correct the spectrum
2. Type **abs**
3. Expand the peak at 1.25ppm

Figure 7.6.

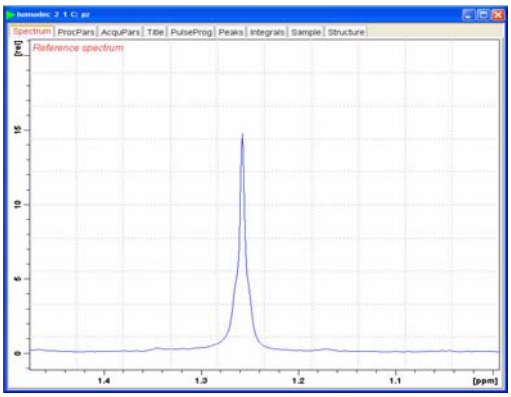


NOTE: This peak is partially collapsed triplet that represents the methyl protons. Increasing the decoupling power level will result in a single peak.

Fine tuning *7.1.5*

1. Type **pl24** on the command line
2. Lower the value by **2**
3. Repeat steps 9 through 13
4. If necessary repeat step 13 through 16

Figure 7.7.

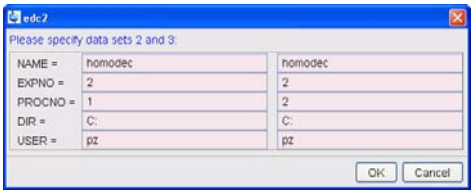


CAUSHION: Increasing the decoupling power level in small steps until the peak is fully decoupled. To much power can cause damage.

Plotting the reference and decoupled spectrum on the same page *7.1.6*

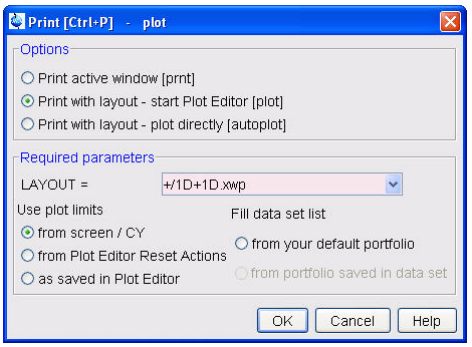
1. Display the reference spectrum
2. Type **edc2**
3, Specify data set 2 as the decoupled spectrum

Figure 7.8.



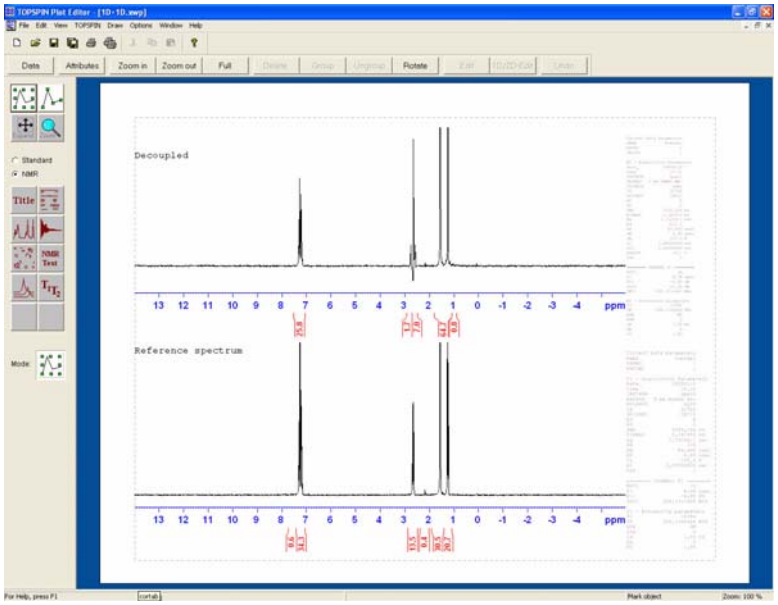
4. Click on OK
5.In the main menu click on '**File**' and select '**Print**'

Figure 7.9.



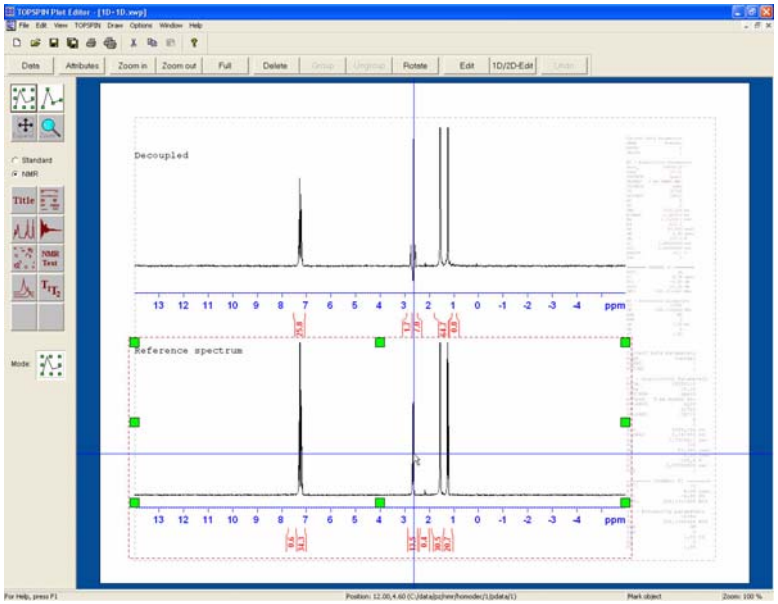
6. In Options select '**Print with Layout - start Plot Editor [plot]**'
7. In Required Parameters select: '**LAYOUT = +/1D + 1D.xwp**'
8. Enable '**from screen/CY**'
9. Click on OK

Figure 7.10.



10. Click anywhere on the reference spectrum

Figure 7.11.



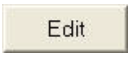
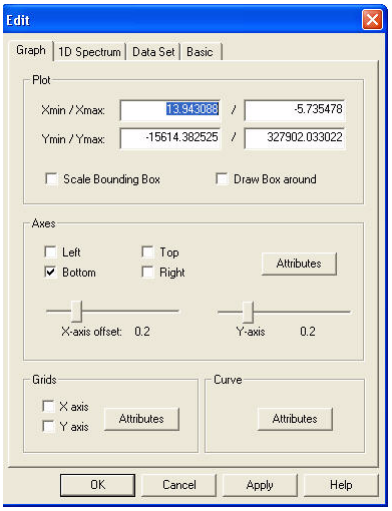
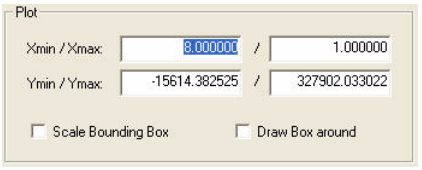
11. Click on 

Figure 7.12.



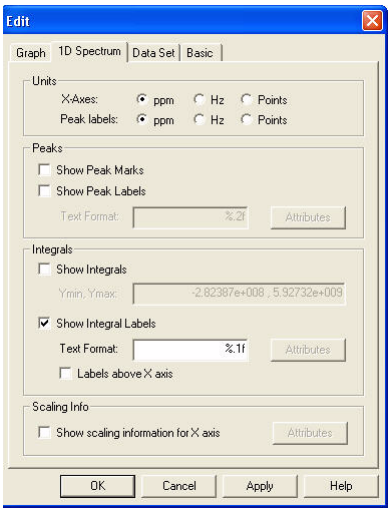
12. Select the '**Graph**' tab by clicking on it
13. Change the '**Xmin/Xmax**' to **8 / 1**

Figure 7.13.



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

Figure 7.14.

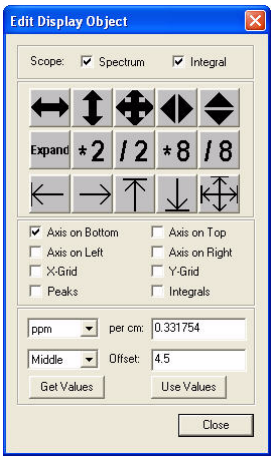


15. Disable '**Show Integral Labels**'

Figure 7.15.

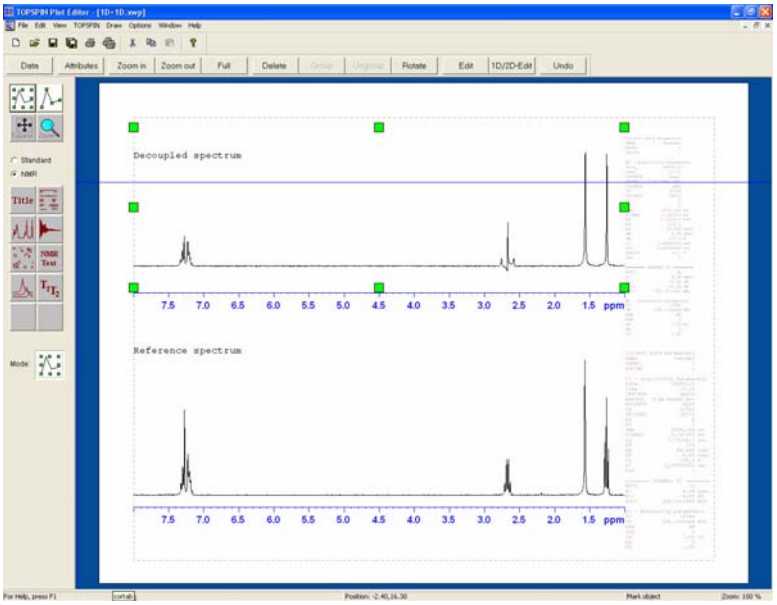
16. Click on Apply
17. Click on OK
18. Click on 1D/2D-Edit

Figure 7.16.



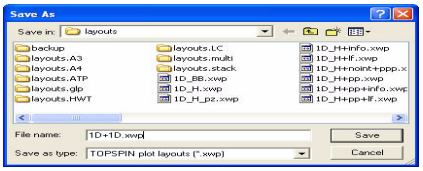
19. Adjust the Y-scaling using the *2 /2 or  buttons
20. Click on Close
21. Repeat steps 8 through 20 on the decoupled spectrum

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

Gradient Shimming 8

1-D Proton gradient shimming 8.1

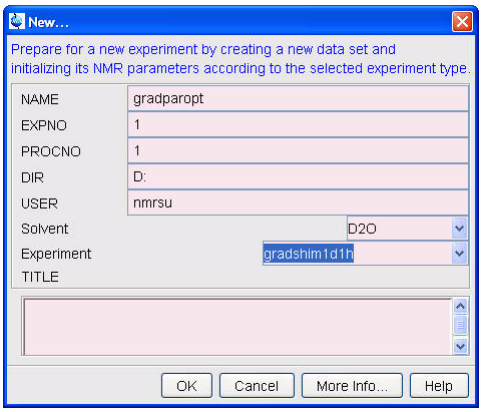
Sample:

2mM Sucrose in 10% D2O / 90% H2O

Parameter optimization 8.1.1

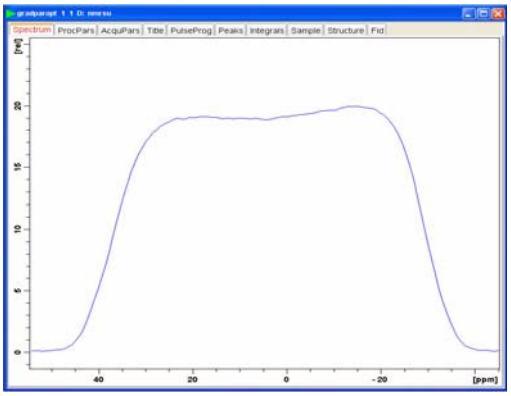
1. Click on  and change the following parameters

Figure 8.1.



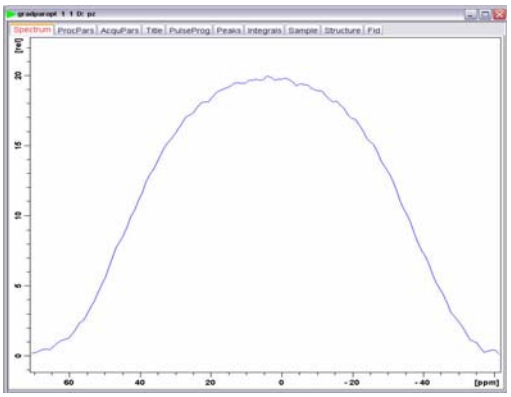
2. Click on 
3. Insert the sample
4. Turn the spinner off
5. Click on  to display the Lock display
6. In the lock display window click on  and select D2O
7. Tune the probe
8. Shim for best homogeneity
9. In the lock display window click on  to close the window
10. Type **wsh GSHIM**
11. Click on  to overwrite this file
12. Click on  to start the acquisition
13. Type **fmc** (fourier transform, magnitude calculation)

Figure 8.2.



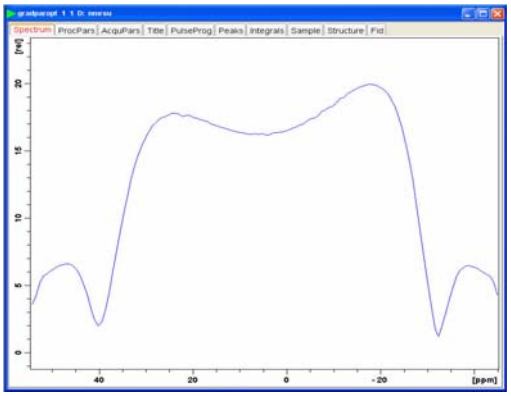
The image in Figure 10.2 is typical for a BBI (1Hcoil on the inside) probe on 2mM Sucrose in 90% H2O, 10% D2O and indicates that the parameters and Hardware are ok. The image should fill about 80% of the display. Proceed with step 14.

Figure 8.3.



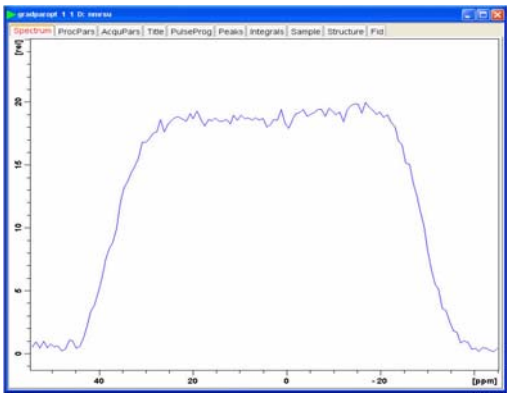
The image in Figure 10.3 is typical for a BBO (1H coil on the outside) probe on 2mM Sucrose in 90% H2O, 10% D2O and indicates that the parameters and Hardware are ok. The image should fill about 80% of the display. Proceed with step 14.

Figure 8.4.



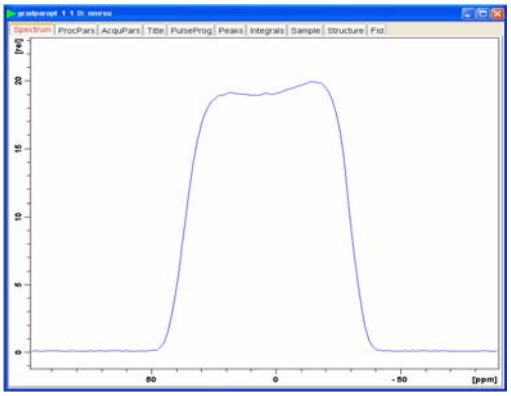
The image in Figure 10.4 is the result of to high of a receiver gain. Make the necessary adjustment.

Figure 8.5.



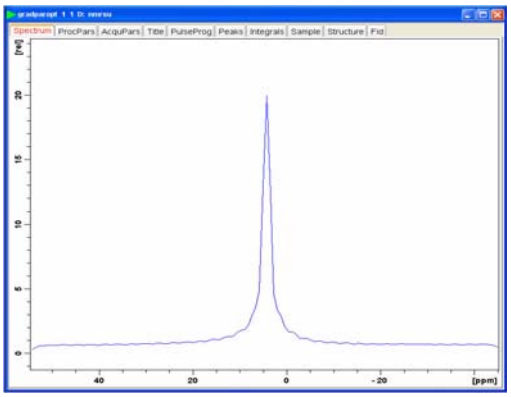
The image in Figure 10.5 is the result of the signal to weak. (pl1 or probe not tuned). Make the necessary adjustment.

Figure 8.6.



The image in Figure 10.6 is the result of sweep width to big or wrong gradient ratio. Make the necessary adjustment.

Figure 8.7.



The image in Figure 10.7 is a result of missing gradients. Check the gradient cable.

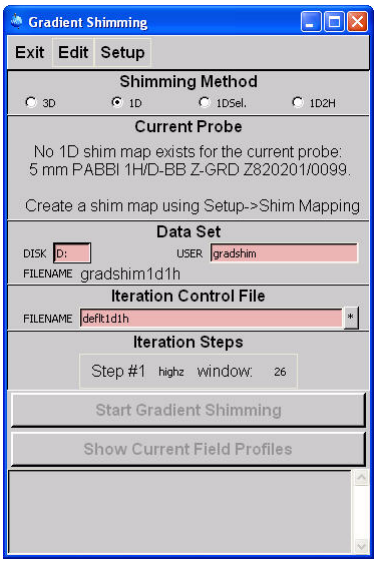
14. Type **wpar gradshim1d1h.bbi**

This parameter file is now specific for the type of probe head used. Other probe heads could have the extension like.qnp,.bbo, etc.

Shim Mapping *8.1.2*

1. Type **gradshim**

Figure 8.8.



2. Enable Shimming Method '**1D'**

3. In the Data Set section under Disk, type the path where the nmr data are normally stored

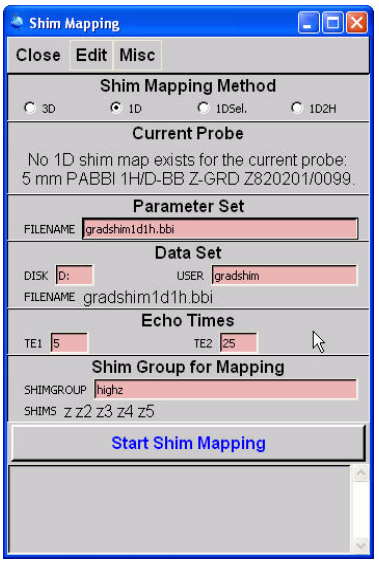
If the gradshim window is open for the first time, the default path for the Disk is 'u'. Do not forget to change the path to where the data are normally stored, e.g. for Windows: C:, D:, or C:\Bruker\TOPSPIN and for LINUX: /opt or /opt/topspin.

4. In the Data Set section under USER use the default name **gradshim**

To avoid permission problems in using the gradient shimming, it is desired for a multi user environment to use the USER “gradshim”.

5. Click on the ‘**Setup**’ tab and select ‘**Shim Mapping**’

Figure 8.9.



6. Enable Shimming Method ‘**1D’**

7. In the Parameter Set section change the FILENAME to: **gradshim1d1h.bbi**

The file name for the parameter set should correspond with the probe head in use, see previous section 10.1.1 Parameter Optimization, steps 1 through 13.

8. In the Data Set section under Disk, type the path where the nmr data are normally stored

If the gradshim window is open for the first time, the default path for the Disk is ‘u’. Do not forget to change the path to where the data are normally stored, e.g. for Windows: C:, D:, or C:\Bruker\TOPSPIN and for LINUX: /opt or /opt/topspin.

9. In the Data Set section under USER use the default name **gradshim**

To avoid permission problems in using the gradient shimming, it is desired for a multi user environment to use the USER gradshim.

10. Click on 

Figure 8.10.

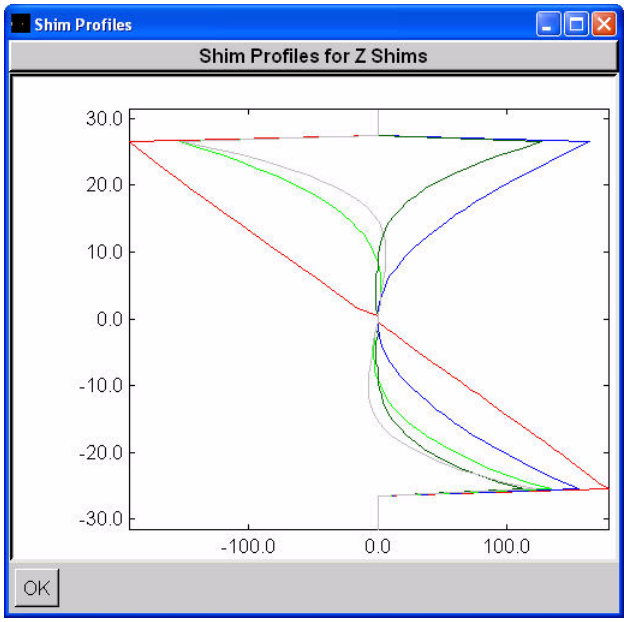
11. Click on 

Figure 8.11.

12. Click on OK

The Shim Mapping starts now and the control windows are inactive. On completion of mapping, the Shim Profile window appears. In a good map the functions (lines) for Z1 - Z5 should be smooth and symmetrical. Note the number on the y axis where the functions become discontinuous, approximately +/- 26 in Figure 10.12. This number will define the maximum window size in the iteration control file.

Figure 8.12.



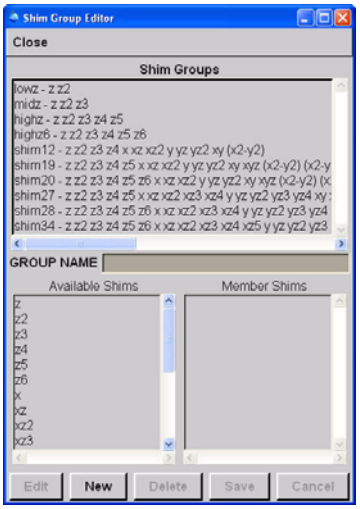
13. Click on OK

14. Click on '**close**' to close the Shim Mapping window

Shim Groups setup *8.1.3*

1. Click on the '**Edit**' tab and select '**Shim Groups**'

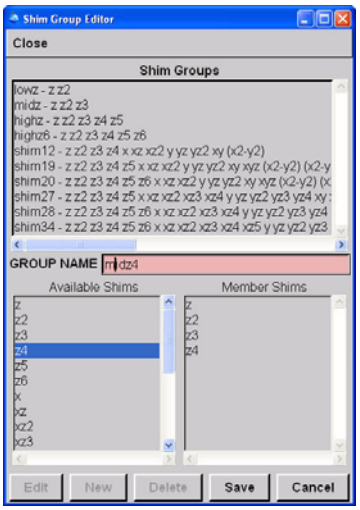
Figure 8.13. -



A list of default Shim Groups are visible in the Shim Group window. If it is desired to create a new Shim Group, follow the steps below.

2. Click on **New**

Figure 8.14.



3. Type **midz4** in to the GROUP NAME window

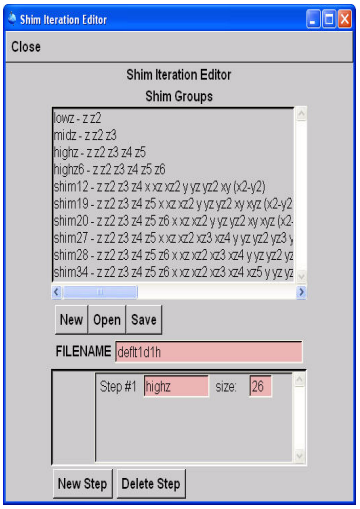
4. Write **Z**, **Z2**, **Z3** and **Z4** in to the Member Shims window by clicking on the corresponded shims in the Available Shims window

5. Click on **Save**

Iteration Control set up *8.1.4*

1. In the Gradshim window Click on the '**Edit**' tab and select '**Iteration Control**'

Figure 8.15.



2. Click on 

Figure 8.16.

3. Change the File Name to **bbi1d1h**
4. Click on 

5. In the Shim Iteration Editor window enter the following parameters:

   Step #1 lowz size **14**

6. Click on 

   Step #2 midz4 size **21**

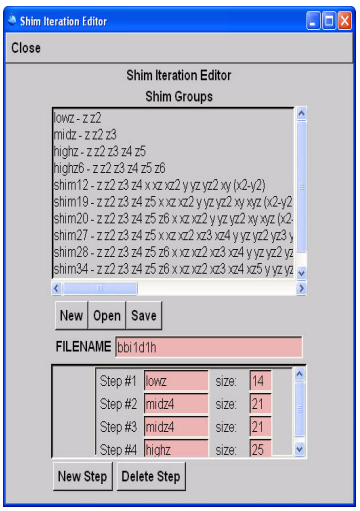
7. Click on 

   Step #3 midz4 size **21**

8. Click on 

   Step #4 highz size **25**

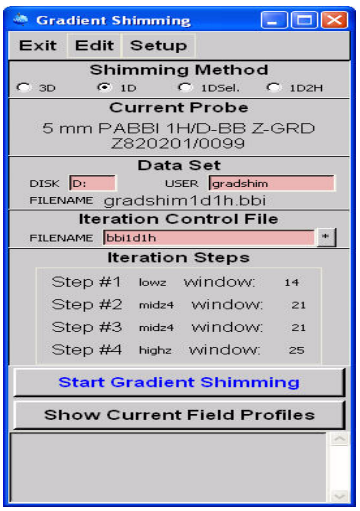
Figure 8.17.



9. Click on 

10. Click on '**close**'

Figure 8.18.



10. Click on '**Exit**' in the Gradient Shimming window

1D-1H Gradient Shimming *8.1.5*

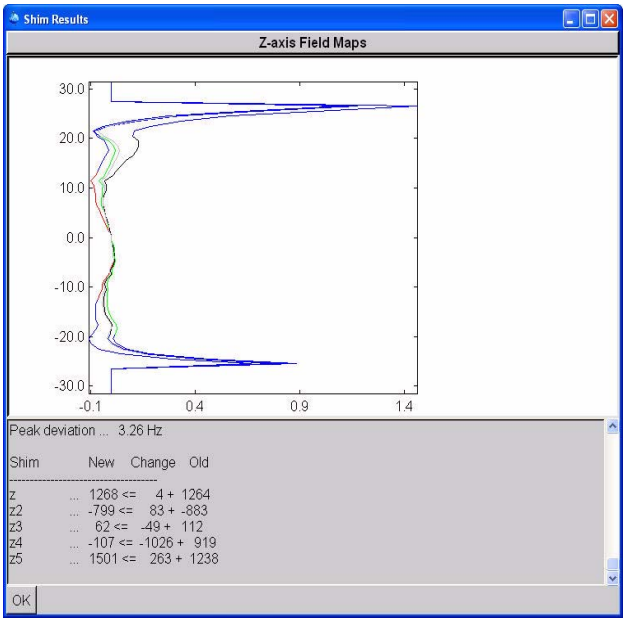
To a sure that the gradient shimming is working, a solvent suppression experiment such as pre saturation should be performed after the gradient shimming,

see Chapter 5 in this manual. The splitting of the anomeric proton peak at 5.3 ppm can be measured.

1. Type **gradshim**
2. Enable the Shimming Method '**1D**'
3. Click on Start Gradient Shimming

Gradient shimming can be executed repeatedly until there is convergence of the shim values. In general, changes to the shim values on the order of <+/- 5 for z and z2, <+/- 50 for z3. <+/- 200 for z4 and z5 indicates convergence.

Figure 8.19.



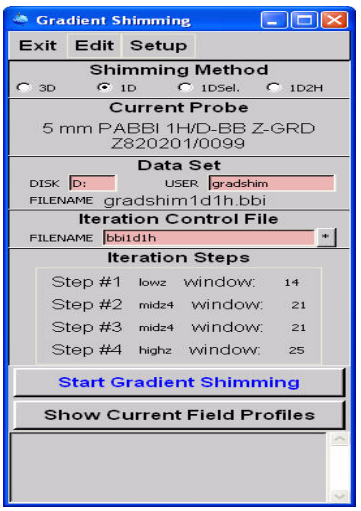
2. Click on OK
3. Close the Gradient Shimming window by selecting the '**Exit**' tab

To use the Proton Gradient Shimming in ICONNMR make sure that 1D Proton Gradient Shimming set up was performed and working. then follow the steps below.

1. Type **gradshim**

2. Enable Shimming Method '**1D'**

Figure 8.20.

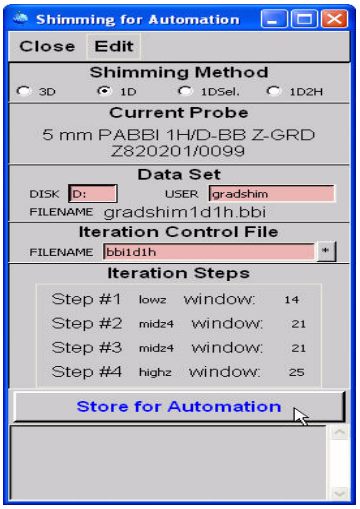


Check that all the parameters are correct.

3. Click on the '**Setup**' tab and select '**Automation**'

4. Enable the Shimming Method '**1D'** and check that all parameters are the same as in the Gradient Shimming window

Figure 8.21.



5.Click on 

Only one method is available to use in Automation. In this case 1D Proton Gradient Shimming is assigned for Automation.

6. Click on '**Close**'

7. Click on '**Exit**' in the Gradient Shimming window

1D Deuterium Gradient Shimming *8.2*

A TH-Tx board or a 2H-Lockswitch unit are required to perform Deuterium gradient shimming

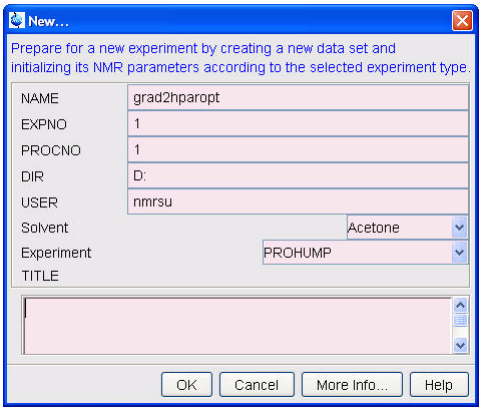
Sample:

3% CHCl3 in Acetone d6

Parameter optimization *8.2.1*

1. Click on  and change the following parameters

Figure 8.22.



2. Click on OK
3. Type **getprosol**
4. Insert the sample
5. Turn the spinner off
6. Click on  to display the Lock display
7. In the lock display window click on  and select Acetone
8. Tune the probe
9. Shim for best homogeneity
10. In the lock display window click on  to close the window
11. Type **wsh GSHIM**
12. Click on OK to overwrite this file
13. Type **iexpno**
14. Type **rpar gradshim1d2h all**
15. Select the '**AcquPars**' tab by clicking on it
16. Change the following parameter:

    O1P [ppm] = **2.03**

17. Click on  to display the pulsprogram parameters

The default parameter set gradshim1d2h has to be optimized for the use of either the 2H-Tx board or the 2H-Lockswitch unit. Select the corresponding parameters in step 14.

18. Make the following changes:

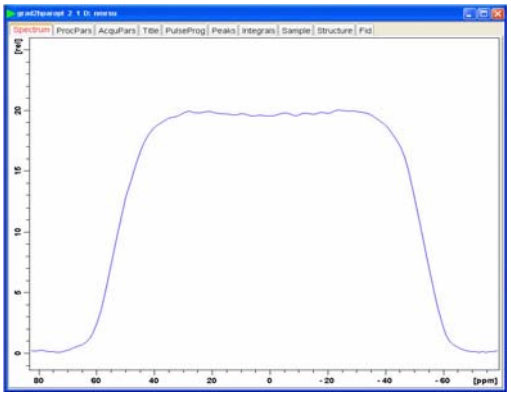
Parameters	2H-Tx board	2H-Lockswitch unit
TD	**512**	**512**
NS	**64**	**64**
DS	**4**	**4**
SWH	**9980**	**9980**
RG	**256**	**256**
D1	**0.05**	**0.05**
P1	**100**	**100**
PL1	**-6**	**6**
GPZ1	**6**	**6**
GPZ2	**-10**	**-10**

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

20. Click on  to start the acquisition

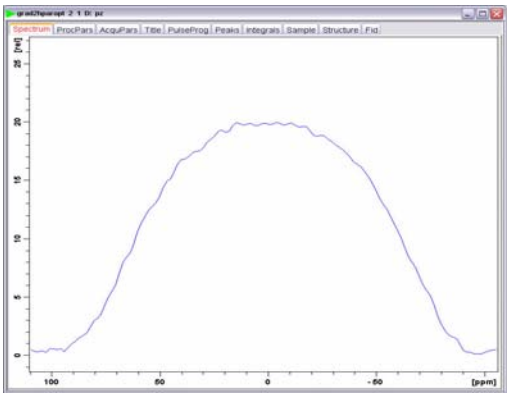
21. Type **fmc** (fourier transform, magnitude calculation)

Figure 8.23.



The image in Figure 10.22 is typical for a BBI probe(1H/2H coil on the inside) on 3% CHCl3 in Acetone d6 and indicates that the parameters and Hardware are ok. The image should fill about 80% of the display. Proceed with step 19.

Figure 8.24.



The image in Figure 10.23 is typical for a BBO probe (1H/2H coil on the outside) on 3% CHCl3 in Acetone d6 and indicates that the parameters and Hardware are ok. The image should fill about 80% of the display. Proceed with step 19.

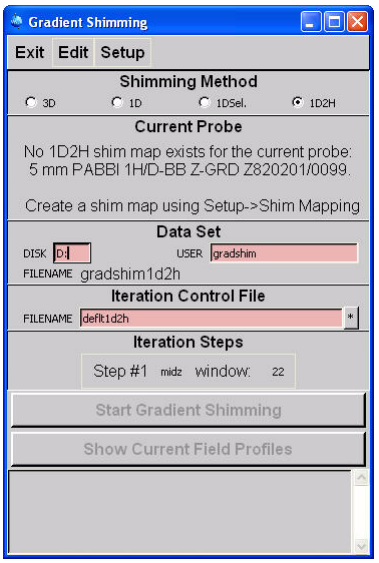
19. Type **wpar gradshim1d2h.bbi all**

This parameter file is now specific for the type of probe head used. Other probe heads could have the extension like.qnp,.bbo, etc.

Shim Mapping *8.2.2*

1. Type **gradshim**

Figure 8.25.



2. Enable Shimming Method '**1D2H'**

3. In the Data Set section under Disk, type the path where the nmr data are normally stored

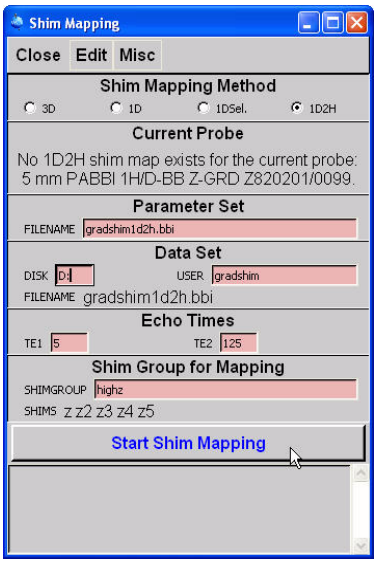
If the gradshim window is open for the first time, the default path for the Disk is 'u'. Do not forget to change the path to where the data are normally stored, e.g. for Windows: C:, D:, or C:\Bruker\TOPSPIN and for LINUX: /opt or /opt/topspin.

4. In the Data Set section under USER use the default name “gradshim”

To avoid permission problems in using the gradient shimming, it is desired for a multi user environment to use the USER “gradshim”.

5. Click on the ‘**Setup**’ tab and select ‘**Shim Mapping**’

Figure 8.26.



7.In the Parameter Set section change the FILENAME to: **gradshim1d2h.bbi**

The file name for the parameter set should correspond with the probe head in use, see previous section 10.2.1 Parameter Optimization, steps 1 through 19.

8. In the Data Set section under Disk, type the path where the nmr data are normally stored

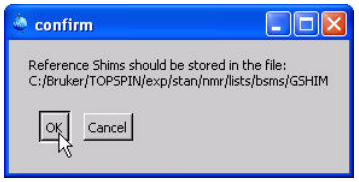
If the gradshim window is open for the first time, the default path for the Disk is ‘u’. Do not forget to change the path to where the data are normally stored, e.g. for Windows: C:, D:, or C:\Bruker\TOPSPIN and for LINUX: /opt or /opt/topspin.

9. In the Data Set section under USER use the default name **gradshim**

To avoid permission problems in using the gradient shimming, it is desired for a multi user environment to use the USER gradshim.

10. Click on 

Figure 8.27.



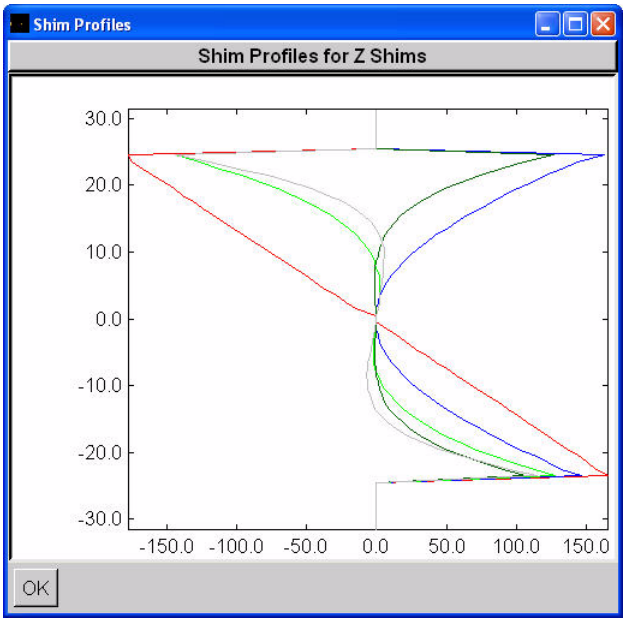
11. Click on 

Figure 8.28.

12. Click on OK

The Shim Mapping starts now and the control windows are inactive. On completion of mapping, the Shim Profile window appears. In a good map the functions (lines) for Z1 - Z5 should be smooth and symmetrical. However since this is a 2H Shim map depending on the instrument and probehead, the lines could be more noisy then the 1H Shimmap. Note the number on the y axis where the functions become dicontenious, approximately +/- 25 in Figure 10.28. This number will define the maximum window size in the iteration control file.

Figure 8.29.



13. Click on OK

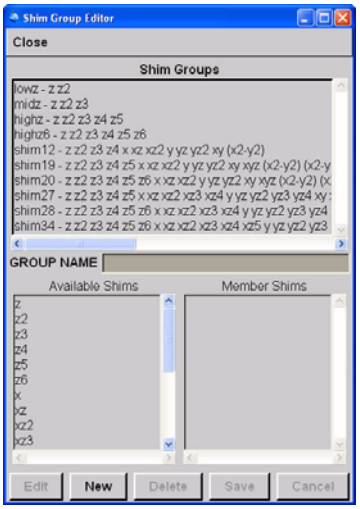
14. Click on '**close**' to close the Shim Mapping window

Shim Groups setup *8.2.3*

If a Shim Group midz4 has been already added to the list during the 1D Proton Gradient shimming set up, then skip this section and proceed with 10.2.4 Iteration Control set up.

1. Click on the '**Edit**' tab and select '**Shim Groups**'

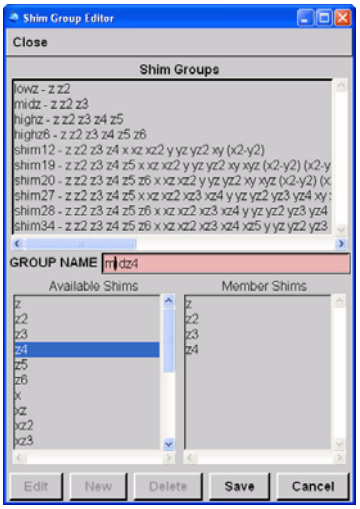
Figure 8.30. -



A list of default Shim Groups are visible in the Shim Group window. If it is desired to create a new Shim Group, follow the steps below.

2. Click on New

Figure 8.31.



3. Type **midz4** in to the GROUP NAME window

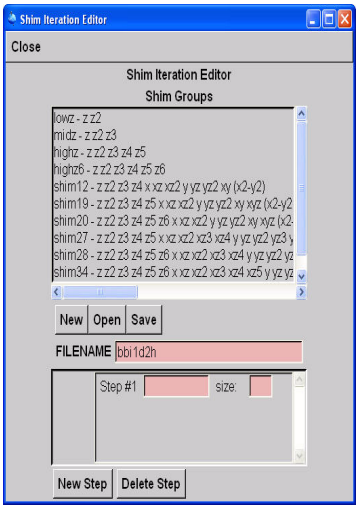
4. Write **Z**, **Z2**, **Z3** and **Z4** in to the Member Shims window by clicking on the corresponded shims in the Available Shims window

5. Click on Save

Iteration Control set up *8.2.4*

1. In the Gradshim window Click on the '**Edit**' tab and select '**Iteration Control**'

Figure 8.32.



2. Click on New

Figure 8.33.

3. Change the File Name to **bbi1d2h**
4. Click on OK
5. In the Shim Iteration Editor window enter the following parameters:

   Step #1 lowz size **14**

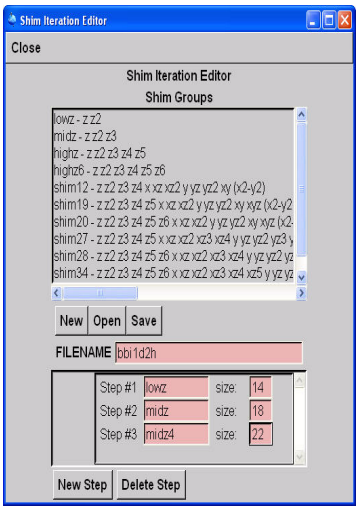
6. Click on New Step

   Step #2 midz size **18**

7. Click on New Step

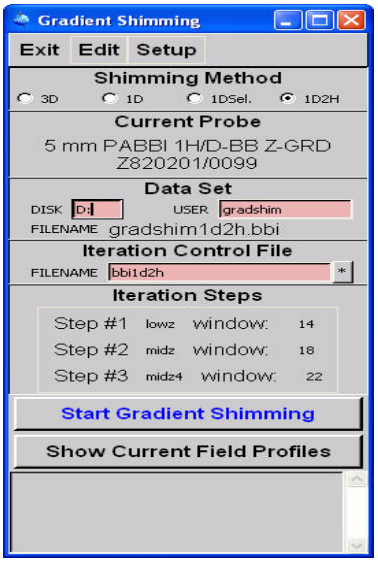
   Step #3 midz4 size **22**

Figure 8.34.



8. Click on Save
9. Click on '**close**'

Figure 8.35.



10. Click on '**Exit**' in the Gradient Shimming window

NOTE: during the gradshim parameter set up the lock was deactivated. To regain the lock, read in a regular 1D proton spectrum. Since we started in the set up for deuterium gradient shimming with a proton parameter set, follow the instructions below.

11. Type **re 1**

12. Type **II** for initializing the interface

The lock should be activated now.

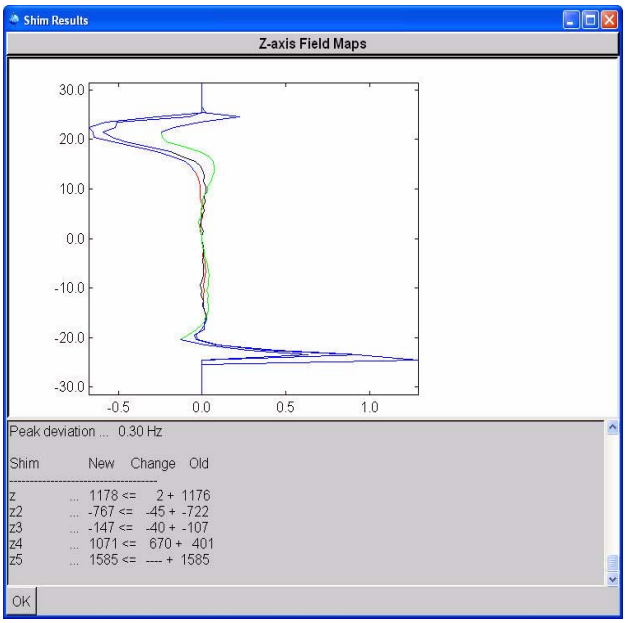
1D-2H Gradient Shimming *8.2.5*

To a sure that the gradient shimming is working, the hump test experiment using the 3% CHCl3 in Acetone d6 sample, should be performed after the gradient shimming.

1. Type **gradshim**
2. Enable the Shimming Method '**1D2H**'
1. Click on Start Gradient Shimming

Gradient shimming can be executed repeatedly until there is convergence of the shim values. In general, changes to the shim values on the order of <+/- 5 for z and z2, <+/- 50 for z3. <+/- 200 for z4 and z5 indicates convergence.

Figure 8.36.



2. Click on OK

3. Close the Gradient Shimming window by selecting the '**Exit**' tab

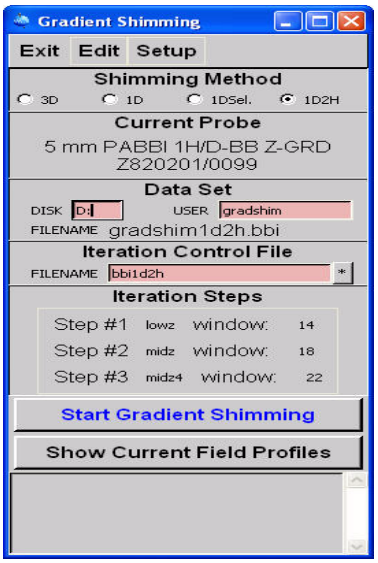
Automation *8.2.6*

NOTE: To use the Deuterium Gradient Shimming in ICONNMR make sure that 1D Deuterium Gradient Shimming set up was performed and working. and then follow the steps below.

1. Type **gradshim**

2. Enable the Shimming Method '**1D2H**'

Figure 8.37.

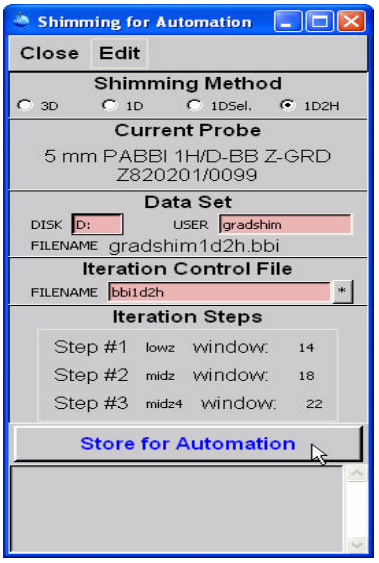


NOTE: Check that all the parameters are correct.

3. Click on the '**Setup**' tab and select '**Automation**'

4. Enable the Shimming Method '**1D2H**' and check that all parameters are the same as in the Gradient Shimming window

Figure 8.38.



5.Click on 

Only one method is available to use in Automation. In this case 1D Deuterium Gradient Shimming is assigned for Automation.

6. Click on '**Close**' in the Shimming for Automation window

7. Click on '**Exit**' in the Gradient Shimming window

3D RCB Gradient Shimming *8.3*

A TH-Tx board or a 2H-lockswitch unit are required to perform 3D RCB gradient shimming

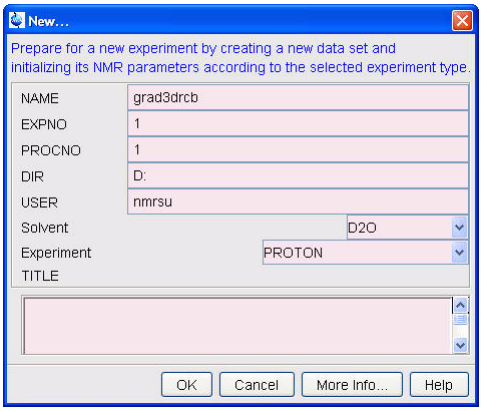
Sample:

2mM Sucrose in 10% D2O / 90% H2O

Parameter optimization *8.3.1*

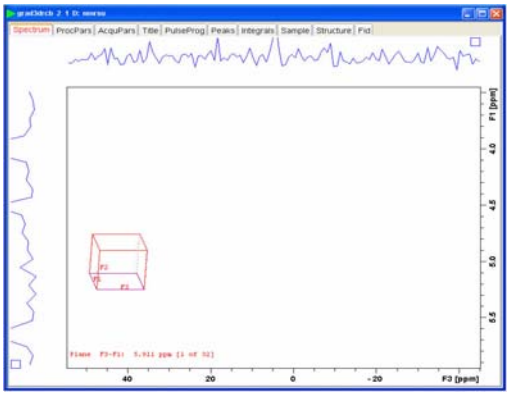
1. Click on  and change the following parameters

Figure 8.39.



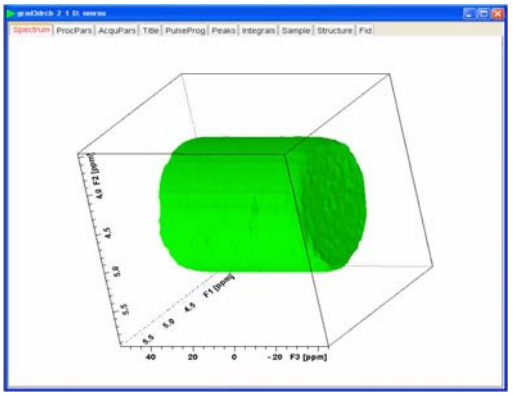
2. Click on OK
3. Type **getprosol**
4. Insert the sample
5. Turn the spinner off
6. Click on  to display the Lock display
7. In the lock display window click on  and select D2O
8. Tune the probe
9. Shim for best homogeneity
10. In the lock display window click on  to close the window
11. Type **wsh GSHIM**
12. Click on OK to overwrite this file
13. Type **iexpno**
14. Type **rpar gradshimrcb3d all**
15. Type **xaua**
16. Type **tf3**
17. Type **tf2**
18. Type **tf1**
19. Type **re 2**

Figure 8.40.



20. In the main menu, click on 

Figure 8.41.



Results of a successful RCB gradient profile experiment are shown in figure 10.41. Note that the image fills much of the box. Parameters which can be tweaked to improve the image are RG (increase to e.g. 64), P0 (increase to e.g. 2.5 usec) and SWH (decrease to e.g. 30000 Hz)

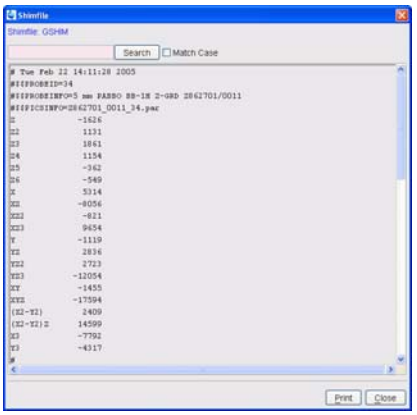
21.Type **wpar gradshimrcb3d.bbi all**

This parameter file is now specific for the type of probe head used. Other probe heads could have the extension like.qnp, bbo, etc.

Shim Mapping *8.3.2*

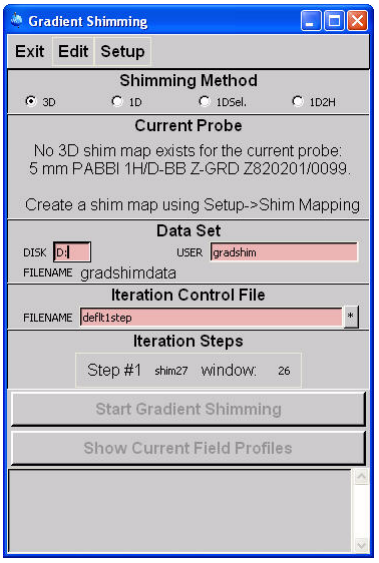
1. Type **vish GSHIM**

Figure 8.42.



2. Count the number of shims displayed and write the number down
3. Type **gradshim**

Figure 8.43.



4. Enable Shimming Method '**3D'**

5. In the Data Set section under Disk, type the path where the nmr data are normally stored

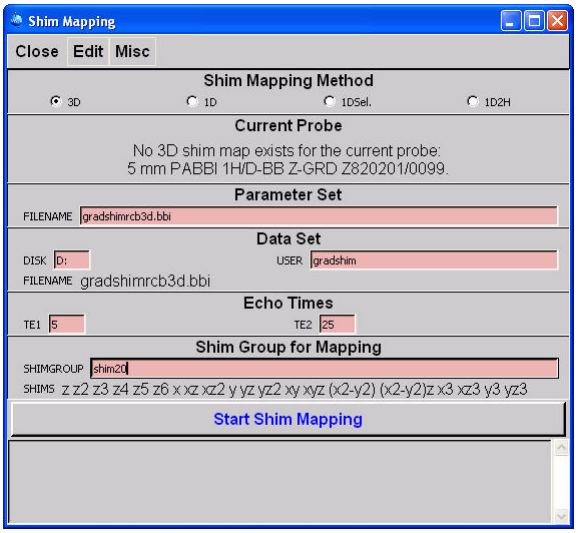
If the gradshim window is open for the first time, the default path for the Disk is 'u'. Do not forget to change the path to where the data are normally stored, e.g. for Windows: C:, D:, or C:\Bruker\TOPSPIN and for LINUX: /opt or /opt/topspin.

6. In the Data Set section under USER use the default name **gradshim**

To avoid permission problems in using the gradient shimming, it is desired for a multi user environment to use the USER "gradshim".

7. Click on the '**Setup**' tab and select '**Shim Mapping**'

Figure 8.44.



8.In the Parameter Set section change the FILENAME to: **gradshimrcb3d.bbi**

The file name for the parameter set should correspond with the probe head in use, see previous section 10.2.1 Parameter Optimization, steps 1 through 19.

9. In the Data Set section under Disk, type the path where the nmr data are normally stored

If the gradshim window is open for the first time, the default path for the Disk is ‘u’. Do not forget to change the path to where the data are normally stored, e.g. for Windows: C:, D:, or C:\Bruker\TOPSPIN and for LINUX: /opt or /opt/topspin.

10. In the Data Set section under USER use the default name **gradshim**

To avoid permission problems in using the gradient shimming, it is desired for a multi user environment to use the USER gradshim.

11. In the Shim Group for Mapping section under SHIMGROUP type the Shim Group name corresponding to the number of shims written down in step 2 (in this example the Shim Group name is shim20)

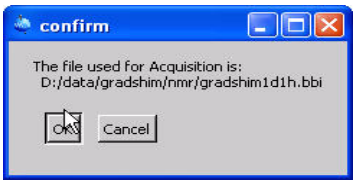
The number of shims is dependent on the system and magnet

12. Click on 

Figure 8.45.

13. Click on 

Figure 8.46.

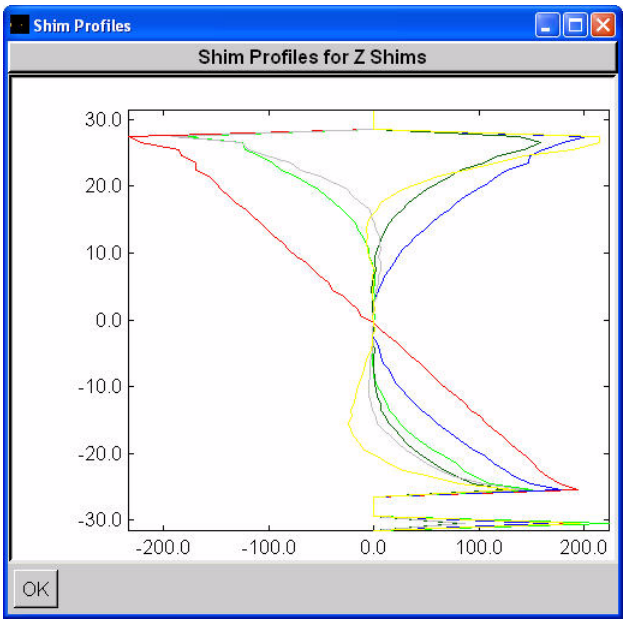


14. Click on 

The Shim Mapping starts now and the control windows are inactive. This task takes approximately 2-3 hours. On completion of mapping, the Shim Profile win-

dow appears. Only the profiles of the Z-shims mapped will be displayed. There is no way of observing the profiles of the off-axis shims. Note the number on the y axis where the functions become dicontenious, approximately +/- 25 in Figure 10.47. This number will define the maximum window size in the iteration control file.

Figure 8.47.



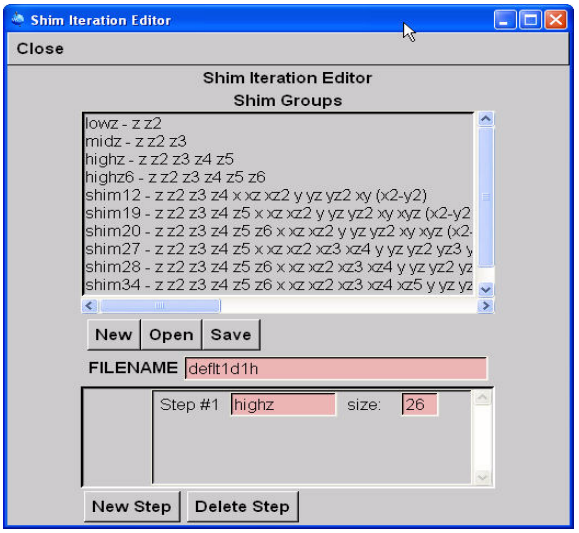
13. Click on OK

14. Click on '**close**' to close the Shim Mapping window

Iteration Control set up *8.3.3*

1. In the Gradshim window Click on the '**Edit**' tab and select '**Iteration Control**'

Figure 8.48.



2. Click on 

Figure 8.49.

3. Change the File Name to **bbi4step**

4. Click on 

5. In the Shim Iteration Editor window enter the following parameters:

Step #1 shim12 size **16**

6. Click on 

Step #2 shim19 size **20**

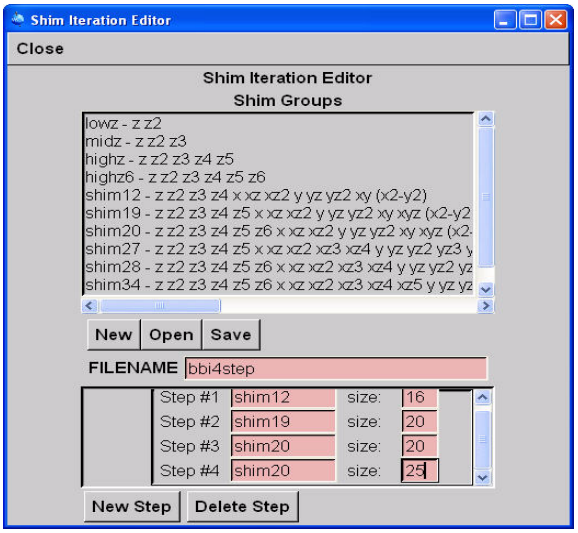
7. Click on 

Step #3 shim20 size **20**

8. Click on 

Step #4 shim20 size **25**

Figure 8.50.

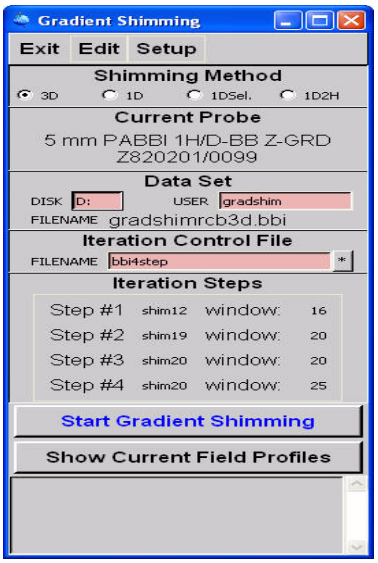


The shim group selected depends upon the system. The group selection as shown in the picture 10.50 is for a BOSS I system with 20 shims. For a Boss II system with 28 or 34 shims you may want to use 5 steps for shimming using shim 27 or shim 33 as the last two steps.

9. Click on Save

10. Click on '**close**'

Figure 8.51.

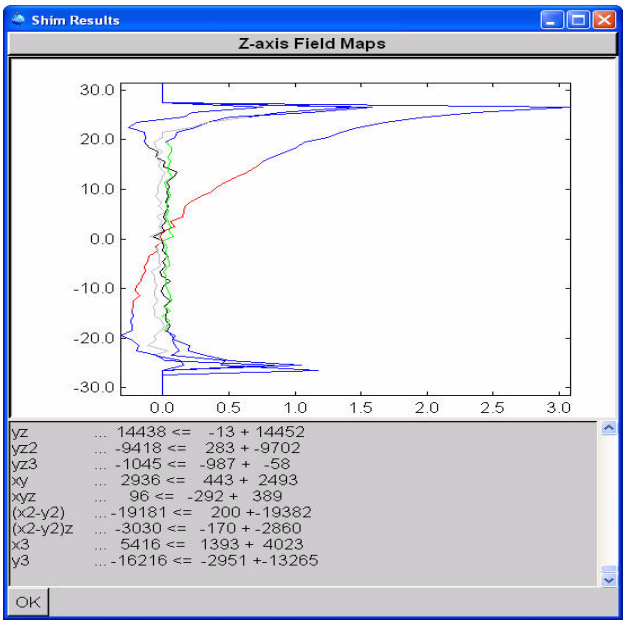


11. Click on '**Exit**' in the Gradient Shimming window

To a sure that the gradient shimming is working, a solvent suppression experiment such as presaturation should be performed after the gradient shimming, see Chapter 5 in this manual. The splitting of the anomeric proton peak at 5.3 ppm can be measured.

1. Type **re 1**
2. Type **gradshim**
2. Enable the Shimming Method '**3D**'
3. Click on Start Gradient Shimming

Figure 8.52.



2. Click on OK
3. Close the Gradient Shimming window by selecting the '**Exit**' tab

2D Basic Experiments

9

2-D gradient COSY 9.1

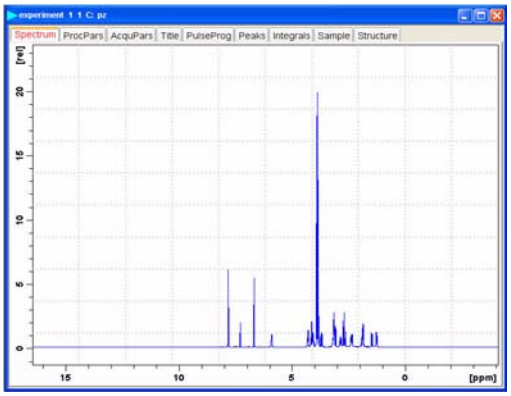
Sample:

30 mg Brucine in CDCl3

Preparation experiment 9.1.1

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

Figure 9.1.



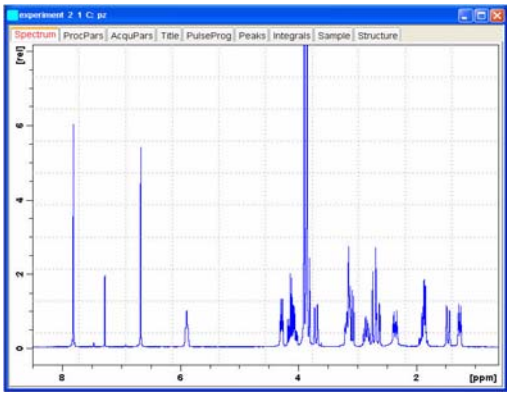
2. Type **wrpa 2** on the command line

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

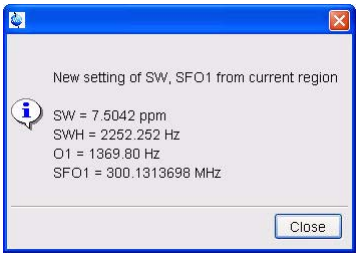
NOTE: You may exclude the solvent peak, if it falls outside of the region of interest.

Figure 9.2.



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

Figure 9.3.



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 Close

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

Setting up the COSY experiment *9.1.2*

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] = value from step 6 (Preparation experiment 10.1.1)

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

O1 [Hz] = value from step 7 (Preparation experiment 10.1.1)

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] = value from step 9 (Preparation experiment 10.1.1)

SR [F1] = value from step 9 (Preparation experiment 10.1.1)

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

9. Make the following changes:

2-D gradient COSY experiment of Brucine

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

Acquisition *9.1.3*

NOTE: The following steps 1 through 3 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

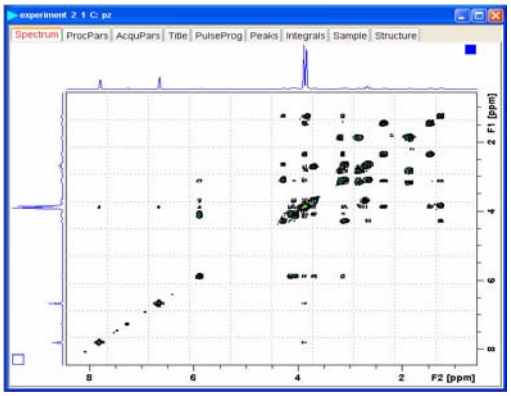
4. Click on  to start the acquisition

Processing *9.1.4*

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

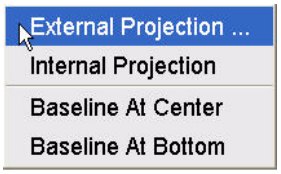
Figure 9.4.



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

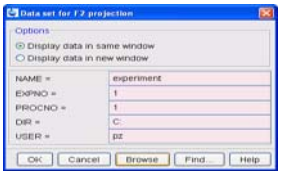
3. Click the right mouse button inside the F2 projection

Figure 9.5.



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

Figure 9.6.



5. Make the following changes:

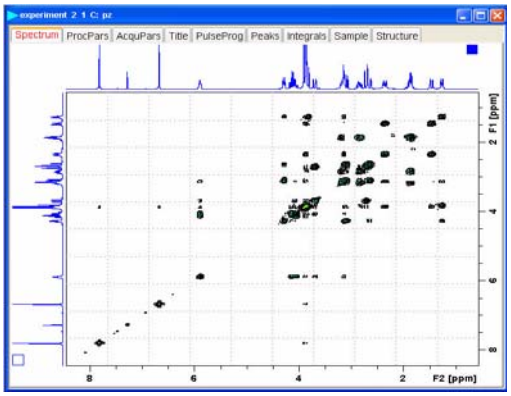
EXPNO = **1** (Experiment number of the 1-D Preparation experiment)

6. Click on OK

7. Click the right mouse button inside the F1 projection

8. Repeat steps 3 through 7

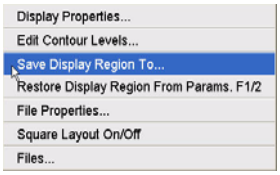
Figure 9.7.



Plotting *9.1.5*

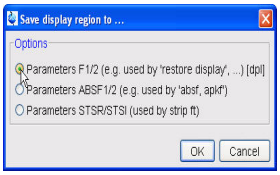
1. Use the *2 /2 *8 /8 buttons to adjust for a suitable contour level
2. Click the right mouse button inside the 2-D contour display

Figure 9.8.



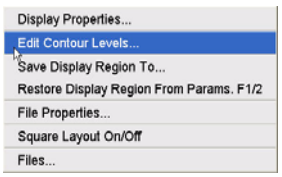
3. Select '**Save Displayed Region To...**' by clicking on it

Figure 9.9.



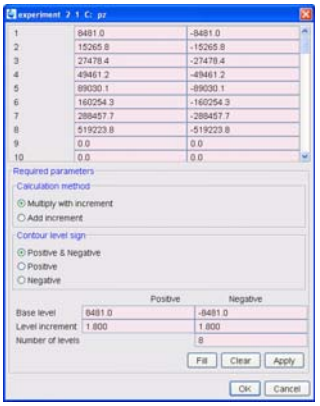
4. Select '**Parameters F1/2 [dpl]**' by enabling the radio button
5. Click on OK
6. Click the right mouse button inside the 2-D contour display

Figure 9.10.



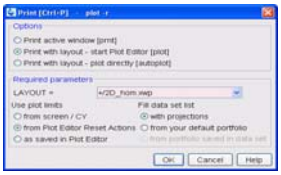
7. Select '**Edit Contour Levels**' by clicking on it

Figure 9.11.



8. Click on Apply
9. Click on OK
10. In the main menu click on '**File**'
11. Select '**Print**' by clicking on it

Figure 9.12.



12. Enable the following options:

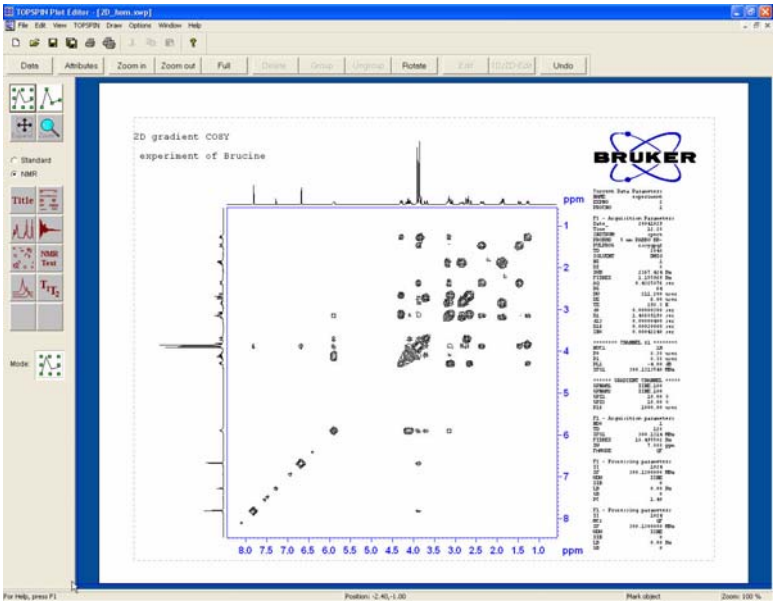
    Print with layout-start Plot Editor

    from Plot Editor Reset Actions

    with projections

13. Select LAYOUT = **+/2D_hom.xwp**
14. Click on OK

Figure 9.13.



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

2-D phase sensitive NOESY experiment *9.2*

Sample:

30 mg Brucine in CDCl3

Preparation experiment *9.2.1*

1. Follow the instructions in 10.1.1 Preparation experiment, steps 1 through 9

Setting up the NOESY experiment *9.2.2*

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] = value from step 6 (Preparation experiment 10.1.1)

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

O1 [Hz] = value from step 7 (Preparation experiment 10.1.1)

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] = **0.7**

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

9. Make the following changes:

SR [F2] = value from step 9 (Preparation experiment 10.1.1)

SR [F1] = value from step 9 (Preparation experiment 10.1.1)

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 experiment of Brucine

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

Acquisition *9.2.3*

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

2. Click on  to start the acquisition

Processing *9.2.4*

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.

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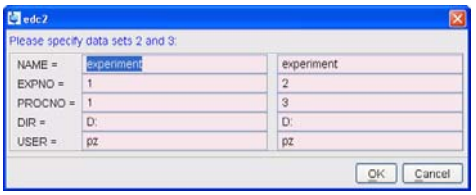
1. Type **edc2**

Figure 9.14.

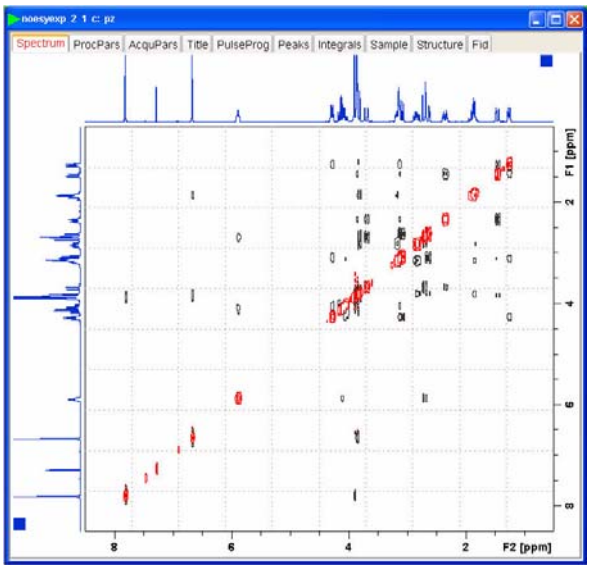


2. Enter the EXPNO and PROCNO of the Preparation experiment 10.1.1 into the first and second column (data set 2 and 3)

3. Click on OK

4. Type **xaup**

Figure 9.15.



Notes: