

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

Basic Experiments User Guide

Version 002



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Contents

Introduction

General

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

1.1

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

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

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

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

1.4

1-D Basic Experiments

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.
	Mew
	Prepare for a new experiment by creating a new data set and initializing its NMR parameters according to the selected experiment type.
	NAME experiment
	EXPNO 1
	PROCNO 1
	DIR C:
	Experiment PROTON
	1D Proton experiment of Brucine
	OK Cancel Help
	2. Click on OK
	3. Insert the sample
	4. Click on 📅 to display the Lock display
	5. In the lock display window click on ${}_{\mathbf{R}}$ and select CDCl3
	6. Tune the probe
	7. Shim for best homogeneity
	8. In the lock display window click on \downarrow 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.
	 In the main menu click on 'Spectrometer', select 'Adjustment' and click c 'Auto-adjust receiver gain' or type rga
	2. Click on > to start the acquisition

1. Process and Phase correct the spectrum

2. In the main menu click on 'Processing' and select 'Baseline Correction'

Processing

2.2.3

🙀 Baseline correction - abs	(
Options	
O Correct baseline manually	
Auto-correct baseline using polynomial	
O Auto-correct spectral range ABSF1. ABSF2 only	
 Auto-correct baseline, alternate algorithm 	
O Define baseline points for cubic spline correction	
Correct baseline using cubic spline	
Correct baseline using base_info file	
Correct baseline using base_info file Correct baseline of the FID	
Correct baseline using base_info file Correct baseline of the FID Required parameters	
Correct baseline using base_info file Correct baseline of the FID Required parameters Degree of polynomial ABSG (05) = 5	
Correct baseline using base_info file Correct baseline of the FID Required parameters Degree of polynomial ABSG (05) = 5 Left limit for correction region ABSF1 [ppm] = 10	
Correct baseline using base_info file Correct baseline of the FID Required parameters Degree of polynomial ABSG (05) = Left limit for correction region ABSF1 [ppm] = Right limit for correction region ABSF2 [ppm] = 0	
Correct baseline using base_info file Correct baseline of the FID Required parameters Degree of polynomial ABSG (05) = Left limit for correction region ABSF1 [ppm] = Right limit for correction region ABSF2 [ppm] = Baseline points file defining cubic spline =	
Correct baseline using base_info file Correct baseline of the FID Required parameters Degree of polynomial ABSG (0.5) = Left limit for correction region ABSF1 [ppm] = Right limit for correction region ABSF2 [ppm] = Baseline points file defining cubic spline = Baseline info file stored by manual correction =	

- 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 \Re to delete the selected regions

Figure 2.3.



9. Click on OK

10. In the Integration menu bar click on 4

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.									
	In Acquisition (seein	g ingetiment 1 1	Ci pr						E 1	- 🔀
	፲ - 소 귀 ·	0 🖪 🖳 /	5 /s # 🛱 🛱 🗖		•2 /	2 0 .	× =	± 1	F 4	
	1D Proton expenn	nent of Brucine	19 F				-		-	
	House Sensitivity: 7.905 ppm / 2397.37	1.0 1 He								
	DEFINE REGION MODE Define: Drog using Return: Left-click	left mouse but highlighted ico	5.0EL							
				-						
		1.				r				
				1		5		11		
	2.0		7	11	.	,[er	
			=			1	11	1	11	
	11	1_	1	11/11	_1	Lall	N	1	11	_
	18	8	8		02		18 2	E		
	<u> </u>	8	<u>8</u>		, an		18/19	ųa,	33	
	-		6	4			-	2	(P	pm)
13. Cli	ck on	🗐 to	o save th	ie integ	grat	tion	re	gio	on	



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 OK



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

1-D Carbon Experiment

Sample:

30 mg Brucine in CDCl3

Experiment set up

2.3.1

2.3

1. Click on 📋 and change the following parameters

Figure 2.7.

	New					
	Prepare for a new initializing its NMR	experiment by parameters ac	creating a ne	w data e selec	i set and ted experime	ent type.
	NAME	experiment				
	EXPNO	1				
	PROCNO	1				
	DIR	C:				
	USER	pz				
	Solvent				CDCI3	~
	Experiment		C13CPI	5		~
	TITLE					
	1D Carbon expe	riment of Bruci	ne			 ×
		ОК	Cancel	Mor	e Info)	Help
2. Click	on OK					
3. Insert	the sample	e				
4. Click	on 📅 to	display	the Loo	ck d	isplay	

	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	
Accusicities		
Acquisition		:.3.Z
	1 In the main menu click on 'Spectrometer' select 'Adjustment' and clic	k on
	1. In the main menu click on 'Spectrometer', select 'Adjustment' and clic	;ł

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'



4. Enable 'Auto-correct baseline using polynomial'

5. Click on OK

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

Peak pickingpp	X
Options Auto-Pick peaks on displayed spectrum region Auto-Pick peaks on full spectrum Define regions / peaks manually, adjust MI, MA Auto-Pick peaks in predefined regions (file 'peak Like 1st option, but peak list with histogram Like 1st option, but peak list in JCAMP format	य akrng')
O Calculate width of currently displayed peak	
Left picking limit F1P = Right picking limit F2P = Intensity of reference peak CY [rel] = Minimum intensity [rel] Maximum intensity MAXI [rel] = Detection sensitivity Fraction of peak height for width calc. [01] = Pick peaks of sign Reference peak selection mode PSCAL =	178.8367 19.0657 20 0 10000 1.4 0.5 pos. ♥ sreg ♥

- 8. Enable 'Define regions / peaks manually, adjust MI, MAXI'
- 9. Click on OK





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.

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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





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



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

DEPT-135 Experiment

Sample:

30 mg Brucine in CDCl3

Experiment set up

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

2.4

2.4.1

Image: Second			
Image: Second Secon	Processina	2 ,	4.3
I was a new sector of the sector experiment by creating a new sets as and infrastruct is x and x an		2. Click on 🕨 to start the acquisition	
 Image: Solution of the second part of the		1. In the main menu click on ' Spectrometer ', select 'Adjustment' and click on ' A to-adjust receiver gain' or type rga	۱u-
 I have experiment by creating a new data set and entitating its NMR percentages according to the selected experiment type in that its its NMR percentages according to the selected experiment type is a set of the s	Acquisition	2.4	1.2
Properties for a new experiment by creating a new data set and initializing its MMP parameters according to the selected experiment type intervent is selected experiment. The selected experiment type is selected experi		6. Select the 'Spectrum' tab by clicking on it	
Prepare for a new experiment by creating a new data set and initializing its NMP parameters according to the selected experiment type. NAME reperiment reproduct to the selected experiment type. PROCNO 1 DOR C: USER SOLVER TO COCISIVE For COCISIV		5. Click on to read in the Prosol parameters	
 New Prepare for a new experiment by creating a new data set and initializing its NMR parameters according to the selected experiment type. NAME experiment PROCNO 1 PROCNO 2 PROCNO 2 PROCNO 2 CC USER PZ CDCI3 CTIDEPT135 CTIDEPT135 CTIDEPT135 Concelled Help 2. Click on ok 3. Select the 'AcquPars' tab by clicking on it 4. Mark the following change		NS = 64	
 2. Click on OK 3. Select the 'AcquPars' tab by clicking on it 		4. Mark the following change	
Prepare for a new experiment by creating a new data set and initializing its NMR parameters according to the selected experiment type. NAME experiment EXPNO 2 PROCNO 1 DIR C: USER pZ Solvent CDCI3 V Experiment C13DEPT135 TITLE ID DEPT135[experiment of Brucine ID DEPT135] CK Cancel Help		3. Select the ' AcquPars ' tab by clicking on it	
Prepare for a new experiment by creating a new data set and initializing its NMR parameters according to the selected experiment type. NAME experiment EXPNO 2 PROCNO 1 DIR c: USER pz Solvent CDCI3 ¥ Experiment C13DEPT135 ¥ TITLE		2. Click on OK	
New Prepare for a new experiment by creating a new data set and initializing its NMR parameters according to the selected experiment type. NAME experiment EXPNO 2 PROCNO 1 DIR c: USER pz Solvent CDCI3 Experiment C13DEPT135 Y TITLE DDEPT135 experiment of Brucine Y		OK Cancel Help	
Prepare for a new experiment by creating a new data set and initializing its NMR parameters according to the selected experiment type. NAME experiment EXPNO 2 PROCNO 1 DIR c: USER pz Solvent CDCI3 ¥ Experiment C13DEPT135 ¥		1D DEPT135 experiment of Brucine	
Prepare for a new experiment by creating a new data set and Initializing its NMR parameters according to the selected experiment type. NAME experiment EXPNO 2 PROCNO 1 DIR c: USER pz Solvent CDCI3 V			
Prepare for a new experiment by creating a new data set and initializing its NMR parameters according to the selected experiment type. NAME experiment EXPNO 2 PROCNO 1 DIR c: USED rz			
New Prepare for a new experiment by creating a new data set and initializing its NMR parameters according to the selected experiment type. NAME EXPNO 2 1		DIR C:	
Prepare for a new experiment by creating a new data set and initializing its NMR parameters according to the selected experiment type. NAME experiment		EXPNO 2 PROCNO 1	
Prepare for a new experiment by creating a new data set and		Initializing its NMR parameters according to the selected experiment type. NAME experiment	
		Prepare for a new experiment by creating a new data set and	
i iguie 2.10.			

- 1. Process and Phase correct the spectrum
- 2. Type abs





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.

lease specify	/ data sets 2 and 3:	
NAME =	experiment	experiment
EXPNO =	1	2
PROCNO =	1	3
DIR =	D:	D:
USER =	pz	pz

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



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

🔄 Print [Ctrl+P] 🔗	plot -r		
Options Orint active win O Print with layout O Print with layout	dow (prnt) t - start Plot Ed t - plot directly	itor (plot) (autoplot)	
Required paramet	ers +/1D+1D+	qwx.qq	v
Use plot limits		Fill data set list	
from Plot Editor as saved in Plot	Y Réset Actions t Editor	o from your default p	ortfolio d in data s

- 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 OK





1-D NOE Difference Experiment

3.1

Introduction



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.

🌺 New	
Prepare for a new initializing its NMR	experiment by creating a new data set and parameters according to the selected experiment type.
NAME	noediffexp
EXPNO	1
PROCNO	1
DIR	C:
USER	pz
Solvent	DMSO 🛩
Experiment	NOEDIFF
TITLE	
Pamoicacid	
	OK Cancel Help
ick on OK	
sert the sampl	e

- 4. Click on 📅 to display the Lock display
- 5. In the lock display window click on 11 and select DMSO

6. Turn the spinner off



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 📢



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



	실 01/02/03	
	Define SFO1/O1 fre	quencies
	SFO1 [MHz] = O1/2/3 [Hz] =	300.132548 2547.98
	01 02	O3 Cancel
5. Click on 02		
6. Click on 📇		





- 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 Q Q Q Q Q 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



- 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.



Running the experiment

3.1.4

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



Figure 3.11.

# of time average cycles :	
2	

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.

💐 multiefp	
Enter first expno to proce	299 :
1	

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

Figure 3.14.

🍓 multiefp	
Enter number of expnos :	
3	

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





12. Click on Δ





13. Click on



elect the	procno of the result dataset
oediffexp	2 1 C: pz
Procno	2

- 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











Select the	procno of the result datase
noediffexp	3 1 C: pz
Procno	2
FIGUNO	2

22. Enter 2 for the Procno

23. Click on	OK
24. Click on	4

Integration

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



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



Solvent Suppression Experiments

4.1

Introduction



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

4.1.1

1. Click on 📋 and change the following parameters

Figure 4.1

2. 3. 4.

🥙 New			
Prepare for a ne initializing its NM	w experiment by creating a new R parameters according to the	v data set and selected experimer	nt type
NAME	solvsup		
EXPNO	1		
PROCNO	1		
DIR	C:		
USER	pz		
Solvent		D2O	*
Experiment TITLE	PROTO	N	~
50mM Raffino preperation e	se pentahydrate periment		~
	ОК	Cancel	Help
on OK]		
t the samp	le		
on 📅 t	o display the Loc	k display	
lock dien	low window click		nd

5. In the lock display window click on $\frac{1}{100}$ and select D2O

6. Turn the spinner off



1. Process and Phase correct the spectrum





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

4.2.1

Parameter set up

- 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.		
	2	01/02/03
	D	efine SF01/01 frequencies
		SFO1 [MHz] = 399.871865
		O1/2/3 [Hz] = 1865.28
		01 02 03 Cancel
6. Click on O1		
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 I 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		

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**





- 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



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



Presaturation with Composite Pulses

Parameter set up

- 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

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

4.3.2

4.3

4.3.1

1. Process and Phase correct the spectrum


Solvent suppression with WATERGATE

Parameter set up

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	<mark>⊥</mark> 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

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**

4.4.2

4.4

Fiaure	4.9.

SquarePower I Receiver RefPhas	e Offset Pulse Offset Pulse Offset Sensitivity 1 Call adjust max 1915.53	30 [146]	
Offset © O1 [Hz]	B	e	
Save Save	min: 1815.53 1865.53	8	

- 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



- 6. Click on Save
- 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





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



Parameter se up

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	t t	o read in the Prosol parameters				
4. Click on	Лt	o display the pulsprogram parameters				
5. Make the	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

4.5

4.5.1

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





Fine tuning

4.5.4

- 1. Select the 'AcquPars' tab by clicking on it
- 2. Click on **1** 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





T1 Experiment

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

5.1.1

1. Click on	
-------------	--

and set the following parameters:

Figure	5	1
riguio	υ.	

NAME	t1exp		
EXPNO	1		
PROCNO	1		
DIR	C:		
USER	pz		
Solvent		DMSO	*
Experiment		PROTONT1	~
T1 experimen	t of Pamoicacid		~
2		OK Cancel H	Help

- 4. Click on 📅 to display the Lock display
- 5. In the lock display window click on **1** and select DMSO

6. Turn the spinner off





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

13. Click on	Л	to display the pulsprogram parameters
14. Change I	D1[s] = 15
	_	

15. Click on to the right of the VDList name entry box

Figure 5.2.

Directory =C:\Bruker	\TOPSPIN1.3\exp\stan\nmr\list
noe	
preemp	
t1delay	
t1delay1	
t1list	
zfilter	
New file:	
	OK Cance

16. Select 't1delay' by clicking on it

click on OK



1. Type xf2 on the command line





Figure 5.5.		
	NMR Relaxation	n Guide
	Close	
	Extract Slice	Fitting Function Fitting Function Start Calculation



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	\sim	'Extract Slice'
----------------------------------	--------	-----------------

Figure 5.6.	
	C Extract a row from 26 data
	Fid or Spectrum must be extracted From the 2d relaxation data. This row should correspond to an experiment with the maximum or minimum delay time. All further data preparation will be done in respect to this row.
	FD) Spectrum Cancel
5. Click on	FID
Figure 5.7.	
•	M N
	Fid must be extracted From the 2d relaxation data. This Fid should correspond to an experiment with the maximum delay time. All further data preparation will be done in respect to this Fid.
	Fid Number =
	OK Cancel

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







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











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

Figure 5.	17.		
	lacation (Temp 1.1.C		8
Fit	ing type		
0	Area	Commit Peak-6 71-230 8999999999999	
6 0	16		-
Pea	# 1 at 8 490 ppm = 1.222s	gTransformsPhase	acutator
Pea	# 2 at 8.115 ppm + 636.409m		
T1 Pea	* 894.344m # 4 at 7.387 ppm	e - Derine Ranges Displa	y Report
T1 Pea	= 852.322m sk 5 at 7.228 ppm = 950.855m		\$
Pea T1	ak 6 at 4 804 ppm = 230.871m		icitur.
		8	
		0 2 4 6 8 10 12 [t(sec)] <	
24. In the	e Guic	de window. click on 🛅 'Display	Report'
			-
Figure 5.	18.		
	🛃 Repo	rt 🛛 🕅	
		Fitting report	
	Datase C:/da	t:	
	INTENS	SITY fit :	
	I[t]=	:I[0]+P*exp(-t/T1)	
	10 poi	nts for Peak 1, Peak Point at 8.490 ppm	
	neb uro		
	I[0] P	= 9.977e-001 = -1.938e+000	
	T1	= 1.232s	
	50	- 3.1042-003	
	ta	u ppm integral intensity	
	15.	000s 8.490 1.5411e+009 2.8169e+008	
	8. 4.	000s 8.490 1.5357e+009 2.58095e+008 000s 8.490 1.4242e+009 2.5884e+008	
	2.	000s 8.490 9.7524e+008 1.7187e+008 000s 8.490 2.8378e+008 3.8931e+007	
	500.	000m 8.490 -3.3963e+008 -8.1422e+007	
	250. 100.	000m 8.490 -7.8093e+008 -1.6368e+008 000m 8.490 -1.0842e+009 -2.2206e+008	
	50. 10	000m 8.490 -1.1956e+009 -2.4337e+008	
		·····································	
		Close Save as Print	
_			1
25. Click	on	Print	
26. Click	on 🔽	Close	
27 In the		a window aliak on Brint/Ex	Port
27. m me	Guid		pon
Figure 5	19		
i igule J.	10. [@p	rint/Export	
		There are the following options to survey the sub-sub-survey	
	4	 To the printer. Type Ctri/P To the printer. Type Ctri/P To a graphics file: Click File->Export, enter e.g. "curve.png" 	
		- To the Clipboard: Click Edit>Copy	
		ок	

28. Use the 'Ctrl' and the ' \mathbf{p} ' keys to print the data

Adding a New Nucleus

6.1

Observing 28Si



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 CDCI3 1. Click on 1 and change the following parameters Figure 6.1. New. Prepare for a new experiment by creating a new data set and initializing its NMR parameters according to the selected experiment type si29exp NAME EXPNO 1 PROCNO 1 DIR C: USER pz CDCI3 Solvent Experiment SI29IG TITLE inverse gated 29Si experiment of TMS v OK Cancel More Info.. Help 2. Click on OK 3. Insert the sample 4. Click on 📅 to display the Lock display 5. In the lock display window click on 11 and select CDCI3

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- 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.

•	No prosol parameterfile "29Si F1 A1" in
3	C/Bruker/TOPSPIN1.3/cont/instr/spect/prosol/34/CD



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.











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.





4. Click once on the coarse tuning + button



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





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



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



4. Type stop on the command line

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 **I** 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



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

Figure 6.11.





20. Click the right mouse button





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

Figure 6.14.



22. Enable 'Parameters F1/2 [dpl]'



24. Type paropt

Figure 6.15	5.	
	🥌 paropt	X
	Enter parameter to modify:	
	p1	
		<u>O</u> K <u>C</u> ancel
25. Enter p	1	

26. Click on OK

Figure 6.16.		
	🤹 paropt	
	Enter initial parameter value:	
	2	
		OK Cancel
27. Enter 2		
28. Click on	OK	
Figure 6.17.		
	🧟 paropt	
	Enter parameter increment:	
		OK Cancel
29. Enter 1		
20 Click on		
30. Click on	OK	
Figure 6.18.		
U	🧟 paropt	
	Enter # of experiments:	
	16	
		OK Cancel
31. Enter 16		<u>Q</u> K <u>C</u> ancel
31. Enter 16 32. Click on		OK Cancel



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.



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.



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





3. Select Peak Power Parameters

Production Parameters	F	Peak	Power	
Sample Parameters Temperature Parameters	Nucleus:		Peak Power [W]	-
Coils Parameters	18	Ŧ	40.0	
Gradient System Parameto	2H	1±	30.0	
Probe Flow Parameters Tuning Matching Paramete	19F	1	40.0	
Peak Power Parameters	31P	14	110.0	
listory	87Rb]±	110.0	
	130]£	148.8	
	170	E	148.0	
	15H	1	180.0	
	148	1±	180.0	
	97Ma	Ŀ	180.0	
	none	1	0.0	
	none	Ŀ	0.0	
· · · · · · · · · · · · · · · · · · ·	none	14	0.0	100

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





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 Is

Figure 6.24.

🧶 GNU Shell				- 0
bash-2.0555 1s ampl_13C.1 ampl_13C.1.raw ampl_15N.1 ampl_15N.1.raw ampl_19F1.raw ampl_9F1.raw ampl_9F1.scale ampl_9H_1.scale ampl_9H_2.bash_2	amp1.1H.2.raw amp1.2H.3 amp1.2H.3.Mar18.2004 amp1.2H.3.raw amp1.3IP.1 amp2.13C.1 amp2.13C.1 amp2.13C.1.raw	amp2_13C_1.scale amp2_15N_1 amp2_15N_1.raw amp2_15N_1.scale amp2_19P_1 amp2_19P_1.raw amp2_11H_2 amp2_1H_2.raw	amp2_1H_2.scale amp2_31F_1 amp2_31F_1.raw amp2_31F_1.scale amp_table audit_cortab.txt	

19. Type cp amp1_13C_1 amp1_29Si_1

- 20. Type cp amp2_13C_1 amp2_29Si_1
- 21. Type Is 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"

Homonuclear Decoupling Experiment

Introduction		7.1
	Sample: 0.1% Ethylbenzene in CDCl3	
Preparation experim	nent	7.1.1
	1. Click on 📋 and change the following parameters	
	Figure 7.1.	
	2. Click on OK	
	3. Insert the sample	
	4. Click on 🙀 to display the Lock display	
	5. In the lock display window click on 10° and select CDCl3	
	 Tune the probe Shim for boat homogonoity 	
	8. In the lock display window click on the close the window	
	9. Select the 'AcquPars' tab by clicking on it	
	10 Click on H to read in the Prosol parameters	
	11.Make the following changes:	

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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







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



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

Homonuclear Decoupling Experiment





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.





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.

Please specify	data sets 2 and 3:	
NAME =	homodec	homodec
EXPNO =	2	2
PROCNO =	1	2
DIR =	C:	C:
USER =	pz	pz

4. Click on OK

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





- 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



10. Click anywhere on the reference spectrum



i iguio i . i z

Plot			
Xmin / Xmax:	13.943088	1	-5.735478
Ymin / Ymax:	-15614.382525	/ 32	7902.03302
🦵 Scale Bound	ling Box 🛛	Draw Bo:	around
Axes			
Left			Attributes
I✓ Bottom	I Right	_	
		— <u>]</u> —	
X-axis offset:	0.2	Y-axis	0.2
Grids	0	urve	
Comment of the local state			

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



Xmin / Xmax:	8.00000	1	1.000000
Ymin / Ymax	-15614.382525	1	327902.033022

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



- Unite	s X-Axes: @ppm CHz CP	oints
	Peak labels: I ppm C Hz C P	oints
Peal	<s< td=""><td></td></s<>	
Г	Show Peak Marks	
Г	Show Peak Labels	
	Text Format: X.2f	Attributes
Integ	grals	
Γ	Show Integrals	
	Ymin, Ymax2.82387e+008	, 5.92732e+009
•	Show Integral Labels	
	Text Format: %.1f	Attributes
	□ Labels above × axis	
Scal	ing Info	
-	-	1 A.M. A

15. Disable 'Show Integral Labels'

Figure 7.15.						
Integrals						
Ymin. Ymax: -2.82387e+008 , 5.92732e+009						
E Showl	☐ Show Integral Labels					
Text Format: %1f Attributes						
Lat	bels above X axis					
16. Click on Apply						
17. Click on OK						
18. Click on 1D/20	D-Edit					
Figure 7.16.						
Ŭ 🚺	dit Display Object					
	Scope: V Spectrum V Integral					
	$\leftrightarrow \uparrow \oplus \oplus \oplus \uparrow \leftrightarrow$					
	Exmand + 2 / 2 + 9 / 9					
	$\leftarrow \rightarrow \downarrow \uparrow \not \leftrightarrow$					
	Axis on Bottom TAxis on Top					
	Axis on Left Axis on Right X-Grid Y-Grid					
	□ Peaks □ Integrals					
	ppm v per cm: 0.331754					
	Middle V Offset: 4.5					
	Get Values Use Values					
19. Adjust the Y-scaling using the *2 /2 or 🔶 buttons						
20. Click on Close						
21. Repeat steps 8	– 8 through 20 on the decoupled spectrum					



22. Click on 'File' and select 'Save as'
Figure 7.18.



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



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

Gradient Shimming

1-D Proton gradient shimming

Sample:

2mM Sucrose in 10% D2O / 90% H2O

Parameter optimization

8.1.1

8.1

1. Click o	on 📋 and change the following parameters
Figure 8	.1.
	🔄 New 🔀
	Prepare for a new experiment by creating a new data set and initializing its NMR parameters according to the selected experiment type.
	NAME gradparopt
	EXPNO 1
	PROCNO 1
	USER Inmrsu
	Solvent D20
	Experiment gradshim1d1h
	OK Cancel More Info Help
2. Click	on OK
3. Insert	the sample
4. Turn t	he spinner off
5. Click d	on 🐨 to display the Lock display
6. In the	lock display window click on and select D2O
7. Tune 1	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 OK to overwrite this file
12. Click	on b to start the acquisition
13. Type	fmc (fourier transform, magnitude calculation)





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.





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.





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





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





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





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.

Exit Edi	it Setup	-			
	Shir	nmin	g Method		
C 3D	• 1D		C 1DSel.	C 10	D2H
	Cu	urren	t Probe		
No 1D 5 mm P	shim map ABBI 1H/I	exist D-BB	s for the cu Z-GRD Z8	rrent pro 20201/0	be 109
Create a	a shim ma	p usir	g Setup->:	Shim Ma	app
		Data	Set		
DISK D: USER gradshim					
FILENAME	gradshim	1d1h			
	Iterat	ion C	ontrol File	•	
FILENAME	deflt1d1h				
	lte	ratio	n Steps		
Step #1		highz	window:	26	
	Start G	radie	nt Shimmi	ng	
	Show Cu	irrent	Field Pro	files	

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.

CIUSE	Lait	MISC		
	S	him Map	ping Metho	d
C 3D	1	• 1D	C 1DSel.	C 1D2H
		Curre	nt Probe	
No 1E) shim	map exi	sts for the cur	rent probe
5 mm l	PABB	I 1H/D-B	B Z-GRD Z82	20201/009
		Parar	neter Set	
FILENAM	E grads	him1d1h.bbi		
18	10	Da	ita Set	
DISK D			USER gradshim	
FILENAM	e grad	dshim1d1	lh.bbi	
		Ech	o Times	
TE1 5			TE2 25	$\widehat{\mathcal{A}}$
	Sh	im Grou	p for Mappin	ng
SHIMGRO	OUP hig	hz		
SHIMS 7	z z 2 z 3	z4 z5		
		Start Sh	im Mapping	

- 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	Start Shim Mapping	
Figure 8.10.		
	👋 confirm 📃 🗖 🔀	
	Reference Shims should be stored in the file: C:/Bruker/TOPSPIN/exp/stan/nmr/lists/bsms/GSHIM	
11. Click on	ок	
Figure 8.11.		
	🔌 confirm 📃 🗖 🔀	
	The file used for Acquisition is: D:/data/gradshim/nmr/gradshim1d1h.bbi	

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.



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.



Figure	8.	1	4
--------	----	---	---

🏝 Shim Group Editor 📃 🗖	×
Close	
Shim Groups	
lowz - z z2	
midz - z z2 z3	
highz - z z2 z3 z4 z5	
nignzo - z zz z3 z4 z5 zo chim12 - z z2 z3 z4 y yz yz2 u yz yz2 w (y2.y2)	
shim19 - z z2 z3 z4 z5 x xz xz y z yz yz xy xyz (x2-y2) (x2-y	
shim20 - z z2 z3 z4 z5 z6 x xz xz2 y yz yz2 xy xyz (x2-y2) (x.	
shim27 - z z2 z3 z4 z5 x xz xz2 xz3 xz4 y yz yz2 yz3 yz4 xy :	
snim28 - z zz z3 z4 z5 z6 x xz xz2 xz3 xz4 y yz yz2 yz3 yz4 shim34 - z z2 z3 z4 z5 z6 x xz xz2 xz3 xz4 yz5 y yz yz2 yz3 yz4	
C	š
GROUP NAME midz4	
Available Shims Member Shims	_
z 🔺 z	
z2 z2	
23 23	
24 75	
ző	
×	
Z	
73	
	1
Edit New Delete Save Cancel	

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

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

Figure 8.15.	
	💐 Shim Iteration Editor
	Close
	Shim Iteration Editor
	Shim Groups
	Nov 222 midz - 222 33 higtrz - 222 33 4 25 higtrz - 222 33 4 25 shim12 - 222 33 4 45 shim12 - 222 33 4 5 xo xo x22 yy zy 22 xy (x2/y) shim19 - 222 33 4 45 xo xo x22 yy zy 22 xy xy (x2/y) shim20 - 222 33 4 45 xo xo x22 yy zy 22 xy xy (x2/y) shim20 - 222 33 4 45 xo xo x22 x3 x44 yy zy 22 ya shim20 - 222 33 4 45 xo xo x22 x3 x44 yy zy 22 ya shim20 - 222 32 4 45 xo xo x22 x3 x44 yy zy 22 ya shim20 - 222 32 4 45 xo xo x22 x3 x44 yy zy 22 ya shim20 - 222 32 4 5 xo xo z2 x3 x44 yy zy 22 ya shim20 - 222 32 4 5 xo xo z2 x3 x44 yy zy 22 ya shim20 - 222 32 4 5 xo xo z2 x3 x44 yy zy 22 ya shim20 - 222 32 4 5 xo xo z2 x3 x44 yy zy 22 ya shim20 - 22 x0
	Step #1 highz size: 26
	New Step Delete Step
2. Click on	lew

Figure 8.16.

Enter N	lew File Name
File: <mark>bbi1d1h</mark> / bbi1d1h defib1d1h	
defit1d2h defit1dsel defit1step defit2step	Cancel

- 3. Change the File Name to bbi1d1h
- 4. Click on OK
- 5. In the Shim Iteration Editor window enter the following parameters:

Step	#1	lowz	size	14
6. Click on	New Ste	p		
Step	#2	midz4	size	21
7. Click on	New Ste	p		
Step	#3	midz4	size	21
8. Click on	New Ste	p		
Step	#4	highz	size	25

Figure 8.17. Close Shim Iteration Editor Shim Groups New Open Save FILENAME bbi1d1h Step #1 lowz size: 14 size: 21 Step #2 midz4 size: 21 Step #3 midz4 Step #4 highz size: 25 × New Step Delete Step

9. Click on Save

10. Click on 'close'



S	himmin	g Method	
C 3D G	1D	C 1DSel.	1D
F	Curren	t Probe	
5 mm F	Z82020	H/D-BB Z-1	ΞRI
	Data	a Set	
DISK D:	υ	SER gradshim	
FILENAME	gradshir	m1d1h.bbi	
Iter	ration C	control File	•
FILENAME	bbi1d1h		
	Iteratio	n Steps	
Step #	¢1 lowz	window:	14
Step ≠	≠2 midz4	window:	21
Step #	≠3 midz4	window:	21
Step #	#4 highz	window:	25
Start	Gradie	nt Shimmi	ng
Show	Curren	t Field Pro	file
Show	Curren	t Field Pro	file

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





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



Sample:

3% CHCl3 in Acetone d6

Parameter	optim	ization
-----------	-------	---------

8.2.1

Figure	8.22.	
	New	
	Prepare for a initializing its I	new experiment by creating a new data set and MRR parameters according to the selected experiment type.
	NAME	grad2bparopt

1. Click on 📋 and change the following parameters

NAME	grad2hparop	pt			
EXPNO	1				
PROCNO	1				
DIR	D:				
USER	nmrsu				
Solvent				Acetone	*
Experiment		PROHU	MP		~
TITLE					
-					^
1					

- 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 $\frac{1}{2}$ 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 \square 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	<u>2H-Tx board</u>	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 'Spec	ctrum' tab by clicking on i	it
20. Click on 🕨 to	start the acquisition	

21. Type fmc (fourier transform, magnitude calculation)





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.





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



Exit Ed	it Setup		
	Shimn	ning Method	
C 3D	C 1D	C 1DSel.	ID2H
	Curr	ent Probe	
No 1D2 5 mm F	H shim map PABBI 1H/D-I	exists for the cu BB Z-GRD Z82	irrent prob 0201/0099
Create	a shim map u	using Setup->S	him Mappi
	D	ata Set	
DISK D:		USER gradshim	
FILENAME	gradshim1d:	2h	
	Iteratio	n Control File	
FILENAME	deflt1d2h		
	ltera	tion Steps	
	Step #1 m	nidz window:	22
	Start Grad	dient Shimmin	g
	Show Curr	ent Field Profi	les

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.

🂐 Shim M	łapping		
Close	Edit Misc		
	Shim Ma	pping Metho	d
C 3D	C 1D	C 1DSel.	ID2H
	Curr	ent Probe	
No 1D2 5 mm F	H shim map ABBI 1H/D-E	exists for the o BB Z-GRD Z8	current probe 20201/0099.
	Para	meter Set	
FILENAM	gradshim1d2h.b	bi	
-	D	ata Set	
DISK D:		USER gradshim	
FILENAM	gradshim1c	12h.bbi	
	Ecl	no Times	
TE1 5		TE2 125	
	Shim Gro	up for Mappi	ng
SHIMGRO	OUP highz		
SHIMS Z	z2 z3 z4 z5		
2	Start S	him Mapping	

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	Start Shim Mapping
Figure 8.27.	
	🔌 confirm
	Reference Shims should be stored in the file: C:/Bruker/TOPSPIN/exp/stan/nmr/lists/bsms/GSHIM
11. Click on	ок
Figure 8.28.	
	💐 confirm 📃 🗖 🔀
	The file used for Acquisition is: D:/data/gradshim/nmr/gradshim1d1h.bbi

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.









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'





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

8.31.	
	🗢 Shim Group Editor
	Close
	Shim Groups
	0 vor. 2 z 2 midz. 2 z 2 z 3 z 4 highz - 2 z 2 z 3 z 4 z 5 highz - 2 z 2 z 3 z 4 z 5 shim 12 - z 2 z 3 z 4 z 5 z 6 shim 19 - z 2 z 3 z 4 z 5 x x z 2 y y z 2 x y x (x2-y2) shim 19 - z 2 z 3 z 4 z 5 x x z 2 y y z 2 x y x (x2-y2) (x2-y shim 20 - z 2 z 3 z 4 z 5 x x z 2 y z y z 2 x y x (x2-y2) (x2-y shim 27 - z 2 z 3 z 4 z 5 x x z 2 x 2 x 3 x 4 y z y z 2 x 3 y x (x2-y2) (x2-y) shim 28 - z 2 z 3 z 4 z 5 x x x z 2 x 3 x 4 y z y z 2 x 3 y z 4 y z 2 x 3 y z 4 y z 2 x 3 y z 4 y z 2 x 3 y z 4 y z 2 x 3 y z 4 y z 2 x 3 y z 4 y z 2 x 3 y z 4 y z 2 x 3 y z 4 y z 2 x 3 y z 4 y z 2 x 3 y z 4 y z 2 x 3 y z 4 z 5 x y z 2 x 3 y z 4 y z 2 x 3 y z 4 y z 2 x 3 y z 4 y z 2 x 3 y z 4 y z 2 x 3 y z 4 y z 2 y z 3 y z 4 z 5 y z
	Available Shims Member Shims
	Z A Z A Z2 Z3 Z3 Z3 Z5 Z6 X Z4 Z6 X X X V2 V V V
	Edit New Delete Save Cancel

Figure 8.31.



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'



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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 Ste	q		
Step	#2	midz	size	18
7. Click on	New Ste	p		
Step	#3	midz4	size	22

Figure 8.34.



9. Click on 'close'

Figure 8.35.	
1	👋 Gradient Shimming 📃 🗖 🔀
	Exit Edit Setup
	Shimming Method C 3D C 1D C 1D5el. @ 1D2H
	Current Probe 5 mm PABBI 1H/D-BB Z-GRD Z820201/0099
	Data Set DISK D: USER gradshim FILENAME gradshim1d2h.bbi
	Iteration Control File
	Iteration Steps Step #1 lowz window: 14
	Step #2 midz window: 18
	Start Gradient Shimming
	Show Current Field Profiles

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



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.

Shim Results					
		Z-axis Field	Maps		
30.0			1		
20.0					
10.0 -	Y			-	
0.0 -	1			4	
-10.0 -				-	
-20.0 -	4		_	-	
-30.0	0.0	0.5	1.0		
Peak deviation 0.30 Hz					
Shim New Change	Old				
z 1178 <= 2 + z2767 <= .45 + z3147 <= .40 + z4 1071 <= .670 z5 1585 <= +	1176 -722 -107 + 401 1585				
ок					
Click on or					

Figure 8.36.

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.





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

BRUKER BIOSPIN

-	Shimming for Automation 📃 🗖 🔀
	Close Edit
	Shimming Method
	Z820201/0099
	Data Set
	Iteration Control File
	FILENAME bbi1d2h *
	Iteration Steps
	Step #1 lowz Window: 14
	Step #2 midz Window: 18 Step #3 midz4 Window: 22
	Store for Automation
6.Click on	Store for Automation



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

BRUKER BIOSPIN

8.3.1

1. Click on	and change the following parameters
Figure 8.39).

EXPNO 1 PROCNO 1 DIR D: USER nmrsu Solvent D20 Experiment PROTON TITLE PROTON	EXPNO 1 PROCNO 1 DIR D: USER nmrsu Solvent D2 Experiment PROTON TITLE D	NAME	grad3drcb		
PROCNO 1 DIR D: USER nmrsu Solvent D20 Experiment PROTON TITLE PROTON	PROCNO 1 DIR D: USER nmrsu Solvent D2r Experiment PROTON TITLE D2r	EXPNO	1		
DIR D: USER nmrsu Solvent D20 Experiment PROTON TITLE PROTON	DR D: USER nmrsu Solvent D2r Experiment PROTON TITLE D2r	PROCNO	1		
USER nmrsu Solvent D2O Experiment PROTON TITLE PROTON	USER nmrsu Solvent D2: Experiment PROTON TITLE	DIR	D:		
Solvent D20 Experiment PROTON TITLE	Solvent D2: Experiment PROTON TITLE	USER	nmrsu		
Experiment PROTON	Experiment PROTON	Solvent			D20
		Experiment TITLE		PROTON	

- 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 11 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

Capacitum (Capacitum	ProcPars AcquPars Title PutseProg Peaks Integrals Sampa 	e Structure Pid
}		
5		-
7		-
Ş		
3		-
	Finne 79-711 5.911 200 (1 of 92)	









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.

Shimfile		
Shimfle, GSH		
	Search Match Case	
# Tue Feb 2	2 14:11:28 2005	
SICEPOBEID-	34	
#ISPROBELN	On5 mm FABBO 88-18 2-GRD 2862701/001:	1
#ISPICSINF:	#2862701_0011_34.per	
10000000	-1626	
22	1131	
123	1861	
24	1154	
25	-362	
26	-549	
x	5314	
202	-8056	
222	-021	
2023	9654	
T.	-1119	
12	2836	
722	2723	
123	-12054	
27	-1455	
XYZ	-17594	
(22-22)	2409	
(32-32)2	14599	
20	-7791	
13	-4317	
(H)		
<		,
		Pret Close

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

					کار کار ک
Exit	Edit	Setup			
		Shir	nming Me	ethod	
• 3D)	C 1D	С	1DSel.	C 1D2H
		C	urrent Pro	obe	
No 5 m	3D s im PA	him map BBI 1H/	exists for D-BB Z-G	the curr RD Z82	ent probe: 0201/0099.
Crea	ate a :	shim ma	p using Se	ətup->Sl	nim Mapping
<u> </u>			Data Set	t	
DISK	D:		USER G	pradshim	
FILEN.	AME GI	radshim	data		
	_	Iterat	ion Cont	rol File	
FILEN.	AME de	eflt1step			*
		lte	ration St	eps	
	ŝ	Step #1	shim27 Wİ	ndow:	26
		Start G	radient S	himmin	g
	S	how Cu	Irrent Fie	ld Profi	les

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
Iguit	0.77

Shim Mapping			
Close Edit Mis	c		
	Shim Ma	pping Method	
SD	C 1D	C 1DSel.	C 1D2H
	Curr	ent Probe	
۱ 5	o 3D shim map ex mm PABBI 1H/D-E	ists for the current p B Z-GRD Z820201/	robe: /0099.
	Para	meter Set	
FILENAME gradshimrcb	3d.bbi		
	D	ata Set	
DISK D:		USER gradshim	
FILENAME gradshir	ircb3d.bbi		
	Ech	no Times	
TE1 5		TE2 25	
	Shim Gro	up for Mapping	
SHIMGROUP shim20	E - 6 y	2000-6212162	
5mim5 Z ZZ ZS Z4 Z	5 ZO X XZ XZZ Y YZ YZ	2 XY XYZ (XZ-YZ) (XZ-	yzjz X5 X25 y5 y23
	Start Sl	nim Mapping	

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	Start Shim Mapping
Figure 8.45.	confirm Confirm Confirm C:/Bruker/TOPSPIN/exp/stan/nmr/lists/bsms/GSHIM Concel Cancel
13. Click on	ок
Figure 8.46.	Cancel
14. Click on	ок

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.



Iteration Control set up

8.3.3

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

Figure 8.48.

🍓 Shim Ite	eration Editor	
Close	М	
	Shim Iteration Editor	
	Shim Groups	
	lowz - z z2	^
	midz - z z2 z3	
	nignz - z zz z3 z4 z5 biobz6 - z z2 z3 z4 z5 z6	
	shim12 - 7 72 73 74 x x7 x72 y y7 y72 xy (x2-y2)	
	shim19 - z z2 z3 z4 z5 x xz xz2 y yz yz2 xy xyz (x2-y2	
	shim20 - z z2 z3 z4 z5 z6 x xz xz2 y yz yz2 xy xyz (x2-	
	shim27 - z z2 z3 z4 z5 x xz xz2 xz3 xz4 y yz yz2 yz3 y	
	shim28 - z z2 z3 z4 z5 z6 x xz xz xz3 xz4 y yz yz2 yz	
	Shiimisa - 2 22 23 24 25 26 x x2 x22 x23 x24 x25 y y2 y2	~
		<u>_</u>
	New Open Save	
	FILENAME defit1d1h	
	Step #1 highz size: 26	
	New Step Delete Step	<u> </u>
lick on	New	

Figure 8.49.

🌢 fileselect	
Enter New	/ File Name
File: bbi4step	
/ bbild1h bbild2h deflt1d1h deflt1d2h deflt1d2e deflt1step deflt2step deflt2step deflt5step	Cancel
	×

- 3. Change the File Name to bbi4step
- 4. Click on OK
- 5. In the Shim Iteration Editor window enter the following parameters:

Step	#1	shim12	size	16
6. Click on	New Ste	p		
Step	#2	shim19	size	20
7. Click on	New Ste	р		
Step	#3	shim20	size	20
8. Click on	New Ste	р		
Step	#4	shim20	size	25

Liguro	0 6 7
гюле	0:30
1 19010	0.00.

	01-1					
	Shi	Chim Crown	altor			
l	-2	Shim Group	s		_	
IOWZ - Z Z	IOWZ - Z ZZ					
highz - z	72 73 74 75	5				
highz6 - :	z z2 z3 z4 :	z5 z6				
shim12 -	z z2 z3 z4	x xz xz2 y yz y	rz2 xy (x	2-y2)		
shim19 - z z2 z3 z4 z5 x xz xz2 y yz yz2 xy xyz (x2-y2						
shim20 - z z2 z3 z4 z5 z6 x xz xz2 y yz yz xy xyz (x2-						
shim20 -	z z2 z3 z4	z5 z6 x xz xz2 z5 x xz xz2 xz2	3 yz yz yz z	2 xy xyz () 17 v72 v7:	3.	
shim20 - shim27 - shim28 -	z z2 z3 z4 z z2 z3 z4 z z2 z3 z4	z5 z6 x xz xz/ z5 x xz xz xz2 xz z5 z6 x xz xz/	: y yz yz, 3 xz4 y y 2 xz3 xz4	2 xy xy2 () /z yz2 yz: · y yz yz2	3 y V2	
shim20 - shim27 - shim28 - shim34 -	z z2 z3 z4 z z2 z3 z4 z z2 z3 z4 z z2 z3 z4 z z2 z3 z4	z5 z6 x xz xz2 z5 x xz xz2 xz z5 z6 x xz xz2 z5 z6 x xz xz2	3 xz4 y 3 xz4 y 2 xz3 xz4 2 xz3 xz4 2 xz3 xz4	2 xy xyz () /z yz2 yz: · y yz yz2 · xz5 y yz	3 y yz yz ↓	
shim20 - shim27 - shim28 - shim34 -	z z2 z3 z4 z z2 z3 z4 z z2 z3 z4 z z2 z3 z4 z z2 z3 z4	25 26 x x2 x22 25 x x2 x2 x22 x2 25 26 x x2 x2 x22 25 26 x x2 x22	: y yz yz 3 xz4 y y 2 xz3 xz4 2 xz3 xz4	z xy xyz () /z yz2 yz: · y yz yz2 · xz5 y yz	√2. 3 y yz yz ↓	
shim20 - shim27 - shim28 - shim34 -	z z2 z3 z4 z z2 z3 z4 z z2 z3 z4 z z2 z3 z4	25 26 x x2 x2 25 x x2 x2 x2 25 z6 x x2 x2 x2 25 z6 x x2 x2 25 z6 x x2 x2 25 x6 x x2 x2	: y yz yz 3 xz4 y y 2 xz3 xz4 2 xz3 xz4 2 xz3 xz4	2 xy xyz () /z yz2 yz y yz yz2 xz5 y yz	√2. yz yz ↓	
shim20 - shim27 - shim28 - shim34 -	z z2 z3 z4 z z2 z3 z4 z z2 z3 z4 z z2 z3 z4 Open Sa ME bbi4s	25 26 x x2 x2 25 x x2 x2 x2 25 z6 x x2 x2	: y yz yz 3 xz4 y y 2 xz3 xz4 2 xz3 xz4 2 xz3 xz4	2 xy xyz () rz yz2 yz: · y yz yz2 · xz5 y yz	√2. 3 y y2 y2 >	
shim20 - shim27 - shim28 - shim34 - New FILENA	z z z z3 z4 z z2 z3 z4 z z2 z3 z4 z z2 z3 z4 z z2 z3 z4 Open Sa ME bbi4s	25 26 x x2 x2 25 x x2 x2 x2 25 x6 x x2 x2 25 26 x x2 x2 25 26 x x2 x2 ave	size:	2 xy xy2 () yz yz2 yz2 y yz yz2 xz5 y yz	√2 yz yz →	
shim20 - shim27 - shim28 - shim34 - < New FILENA	z z2 z3 z4 z z2 z3 z4 z z2 z3 z4 z z2 z3 z4 Open Sa ME bbi4s Step #1 Step #2	125 26 x x2 x22 125 x x2 x22 x2 125 26 x x2 x22 125 26 x x2 x22 125 26 x x2 x2 125 26 x x2 x2 x2 x2 x2 x2 125 26 x x2 x	size: size:	2 xy xy2 () yz yz2 yz2 y yz yz2 xz5 y yz 16 16 20	2- 3 y y2 y2 y2	
shim27 - shim27 - shim38 - shim34 - < New FILENA	z z2 z3 z4 z z2 z3 z4 z z2 z3 z4 z z2 z3 z4 m Open Sz ME bbi4s Step #1 Step #2 Step #3	1 25 26 x x2 x22 x2 1 25 x x2 x2 x2 x2 1 25 26 x x2 x2 x2 1 25 26 x x2 x22 1 25 26 x x2 x2 x2 1 25 26 x x2 x2 x2 x2 x2 x2 1 25 x x2 x	size: size: size:	2 xy xy2 () rz yz2 yz2 y yz yz2 xz5 y yz 16 20 20	2. 3 y y2 y2 ♥	



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.



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.



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

2D Basic Experiments

Sample:

30 mg Brucine in CDCl3

Preparation experiment

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.

9.1.1



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





- 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)

BRUKER BIOSPIN

- 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





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.

Options Display data i O Display data i	n same window n new window	
NAME =	experiment	
EXPNO =	1	
PROCNO = 1		
DIR = C		
USER -	DZ	

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



Plotting

- 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.

C	Display Properties
E	dit Contour Levels
50	ave Display Region To
F	Restore Display Region From Params. F1/2
F	ile Properties
s	quare Layout On/Off
F	iles

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

	Casperiment 2	1 C: p7	
	1	8481.0	-8481.0
	2	15265.8	15265.8
	3	27478.4	-27478.4
	4	49461.2	-49461.2
	5	89030.1	-89030.1
	6	160254.3	-160254.3
	7	288457.7	-288457.7
	8	519223.8	-519223.8
	9	0.0	0.0
	10	0.0	0.0
	Required param	eters	
	Calculation met	hod	
	Multiply with Add increment	increment cf	
	Contage interesting	2	
	Contour level s	on .	
	Postive & N O Postive O Negative	sgative	
	and the second s	Poste	Normative
	Raselevel	8481.0	-6481.0
	Level increment	1.800	1.800
	Number of level	5	8
	April 1 and 1 and 1 and 1 and 1 and 1		
			Len Creat T yobh
	ļ.		OK Cancel
. Click on Apple . Click on	oly K menu	click on	'File'

12. Enable the following options:

Print with layout-start Plot Editor from Plot Editor Reset Actions with projections 13. Select LAYOUT =

+/2D_hom.xwp

OK Cancel Hels

14. Click on OK



15. In the Plot Editor's main menu, click in 'File'

16. Select 'Print' by clicking on it

2-D phase sensitive NOESY experiment

Sample:

30 mg Brucine in CDCI3

Preparation experiment

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

Setting up the NOESY experiment

- 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

9.2

9.2.1

9.2.2

4. Make the following changes:

		•	0
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	U I	to read	in the Prosol parameters
6. Click on	Л	to displa	ay the pulsprogram parameters
7. Make the	follo	wing cha	anges:
D1 [s]	=	2	
D8 [s]	=	0.7	
8. Select the	e ' Prc	ocPar' ta	ab by clicking on it
9. Make the	follo	wing cha	anges:
SR [F2]		=	value from step 9 (Preparation experiment 10.1.1)
SR [F1]		=	value from step 9 (Preparation experiment 10.1.1)
PHC0 [degr	ee] (F	=1)=	90
PHC1 [degr	ee] (F	=1)=	-180
FCOR (F1)		=	1
10. Select th	ne ' Ti	tle ' tab	by clicking on it
11. Make th	e follo	owing cl	nanges:
2-D p	hase	sensit	ive NOESY experiment of Brucine
12. Select th	ne ' S p	oectrun	n' 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.

1. Type edc2



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: