

# GREAT 1/10 and 3/10

## for TopSpin 2.0 pl3 User/Installation Manual

Version 001

Bruker **BioSpin** 

The information in this manual may be altered without notice.

BRUKER BIOSPIN accepts no responsibility for actions taken as a result of use of this manual. BRUKER BIOSPIN accepts no liability for any mistakes contained in the manual, leading to coincidental damage, whether during installation or operation of the instrument. Unauthorized reproduction of manual contents, without written permission from the publishers, or translation into another language, either in full or in part, is forbidden.

This manual was written by

Philippe Lux

© June 18, 2007: BRUKER BIOSPIN S.A. France Application & Spectroscopic development department

E-Mail address : philippe.lux@bruker.fr

P/N: Z31815 DWG-Nr.: Z4D10314/001

# Contents

	Contents
1	Introduction
1.1	Introduction
1.2	Disclaimer
1.3	Warnings and Notes
1.4	Contact for Additional Technical Assistance
2	Starting with the GREAT Gradient Amplifier
3	Great Calibration Operations11
4	GREAT Service Web Pages1
5	GRADIENT Strength Calibration
5.1	Gradient Profile Recording Procedure
6	GRADIENT Recovery/Pre-emphasis Test
6.1	Gradient Recovery Test Recording Procedure
6.2	Gradient Pre-emphasis Adjustment Procedure
7	GRADIENT Echo Stability Test
7.1	Gradient Stability Experiment Recording Procedure
8	Water Diffusion Coefficient Measurement Using a
	1D Gradient Echo Pulse Sequencer 35
9	Miscellaneous
9.1	The GCUTYPE and the GRADRES Parameters
92	The Meanings of the Blanking Orders 40
9.3	How Does the GREAT 10 Blanking Work with a 19" IPSO Com
	puter?
	Grad2.incl
9.4	Blanking Inhibition = On -> External Blanking Mode
9.5	Blanking Inhibition = Off -> Internal blanking mode
9.6	Blanking Inhibition = On -> External Blanking Mode with Pre- emphasis
9.7	Blanking Inhibition = Off -> Internal blanking mode with pre-em

98	phasis
0.0	Computer?
9.9	Contact for Further Questions? 47
10	1D Gradient "Shimming" Operations
10.1	1D Gradient Shimming Recording Procedure 50
11	3D Gradient "Shimmimg" Operation on X, Y, Z Gra-
	dient Coil55
11.1	3D Gradient Shimming Recording Procedure
12	3D Gradient "Shimming" Operation on Z Gradient
	Coil61
12.1	3D RCB Gradient Shimming Recording Procedure
13	Frequently Asked Questions (FAQ)65
13	Frequently Asked Questions (FAQ)65 I use a gradient pulse sequence and nothing happens on the lock level 65
13	Frequently Asked Questions (FAQ)65 I use a gradient pulse sequence and nothing happens on the lock level
13	Frequently Asked Questions (FAQ)65 I use a gradient pulse sequence and nothing happens on the lock level
13	Frequently Asked Questions (FAQ)
13 14	Frequently Asked Questions (FAQ)
13 14	Frequently Asked Questions (FAQ)       65         I use a gradient pulse sequence and nothing happens on the lock level.       65         I have no Gradient Pulse and I am sure that the coil is not damaged.       65         I have done the IPSO restart procedure but the GREAT does not pulse.       65         For all other questions, you can send an E-mail to the following address:       66         GREAT 1/10 - GREAT 3/10 Package       67         Figures       69

# Introduction

## Introduction

This manual is a part of the CD-ROM delivered with your GREAT amplifier in Adobe PDF format.

The CD-ROM contains parameter sets, pulse programs and automation programs used by the included test procedures.

- The pulse programs have to be placed in the pulse program library (import push button in the TopSpin edpul display).
- The automation programs have to be copied in the /src directory and compiled.
- The parameter sets have to be put in the parameter library (Import push button in the TopSpin edpar display).
- Run an expinstall procedure after file importation.

The part number for the CD-ROM is W1214559/ECL 00.

This manual applies for TopSpin version 2.0 pl 3 and higher.

### Disclaimer

1.2

GREAT amplifiers should only be used for their intended purpose as described in this manual. Use of GREAT amplifiers 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.

Service or maintenance work on GREAT amplifiers must be carried out by qualified personnel.

Only those persons schooled in the operation of GREAT amplifiers should use it.

Read this manual before operating GREAT. Pay particular attention to any safety related information.

## 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



## Contact for Additional Technical Assistance

For further technical assistance on GREAT, please do not hesitate to contact your nearest BRUKER dealer or contact us directly at:

BRUKER BioSpin SA 34 rue de l'industrie F-67160 Wissembourg France Phone: + 33 3 88 73 69 61 FAX: + 33 3 88 73 68 86 Email: didier.varney@bruker.fr Internet: www.bruker.fr

# Starting with the GREAT Gradient Amplifier



Figure 2.1. GREAT 1/10 Gradient Amplifier Front Panel



Figure 2.2. GREAT 3/10 Gradient Amplifier Front Panel



Figure 2.3. Connection Scheme of a GREAT 10 Amplifier to the 19" IPSO Unit

**GREAT LED block**: Green LED's show that the amplifier is operating whereas red LED's show that the amplifier is in fault status. If the LED's are red, go to the BGA Web page and perform **Restart Protection**.

**LVDS input connector**: Has to be connected to the GCONtroller (last FCONtroller in the IPSO rack) if the spectrometer has no digital pre-emphasis. Has to be connected to the Digital Pre-emphasis Processor (DPP) output if the spectrometer is equipped with digital pre-emphasis.

**GREAT output cable**: On the GREAT 1/10 you have one connector on the cable connected on the probe. On the GREAT 3/10, you have 3 connectors, one for each channel X, Y or Z.

**External TCU Blanking**: Blanking input's (T0 connector A6 for Z blanking for the GREAT 1/10 case and T0 connector A4, A5, A6 for X, Y, Z blanking for the GREAT 3/10). Note that in the case of the AQS IPSO, external blanking is not directly available. Special request has to be done for external blanking in this case.

**Monitor Output**: Analogical voltage output ( $R = 1 \Omega$ ) allowing gradient pulse display on external electronic devices like oscilloscopes.

**Ethernet connector**: This cable is connected on the HUB delivered with the spectrometer. During the "cf" spectrometer configuration procedure, the IPSO server attributes an IP address to the gradient amplifier device. Communication is by means of the BGA Web pages.

UPS and USB connectors: Not used on the actual spectrometer version.

**Coil Temperature connector**: Scheduled for future gradient coil temperature control operations.

On/Off button: Voltage On/off switch.

The AVANCE III spectrometer equipped with a GREAT 1/10 or GREAT 3/10 amplifier is able to work with or without the Digital Pre-emphasis Processor (DPP) board. The "cf" spectrometer configuration set the GREAT handling parameters automatically in the status in which the GREAT uses digital pre-emphasis. In this case the GCONtroller LVDS output has to be connected to the DPP LVDS input and the DPP LVDS output has to be connected to the LVDS GREAT input If you don't have any DPP board, you have to enter the **nodpp** order after each "cf" spectrometer configuration procedure. In this case the GCONtroller LVDS output has to be directly connected to the GREAT LVDS input.

In both cases, the GREAT amplifier blanking is managed either by the internal GCON blanking orders (ctrlgrad xx) or by the external T0 blanking words (signal A4, A5, A6 from IPSO A connector). The external blanking option does not exist on AQS IPSO systems. The blanking handling is chosen in the GREAT 10 service Web page with the **Blanking Inhibition function**. If this function is toggled to **Off**, then the internal GCON blanking is used. If this function is toggled to **On**, then the external blanking is only active.

# Great Calibration Operations

3

Open the BGA Web page by means of the "ha" order of the TopSpin interface or by opening a Web browser page. In the last case you have to know the BGA IP address. Enter the http://IP.address order into the Web browser to access to the BGA Web page.

On the home page select BGA Service Tools.

On the BGA Service Tools page, select Channel X, Channel Y, Channel Z.

The GREAT 1/10 and GREAT 3/10 gradient amplifiers are calibrated during the manufacturing test procedure by BRUKER. Nevertheless, the gradient amplifier offset may change with temperature. After the first time you switch the GREAT On/Off you have to push on the **configure** button. This operation calculates the gain applied to the offset correction loop.

It is **strongly recommended** each time you switch the GREAT power On/Off to run an auto offset calibration using the facility in the BGA Web page. The first operation to do, is to set the **Blanking Inhibition** to **on**. Remove the external blanking cables on the X,Y or Z connecters if they are connected. Run the offset calibration with the **execute** button. This operation may take less then one minute. At the end of the process you can check the offset value by means of the **refresh data** button. Connect the blanking cables again.

Two other parameters (the regulation current R and C parameters) may be different if the GREAT is used with X,Y,Z HR gradient coils or Z HR-MAS gradient coils. The default values (R = 74,9 % - C = 13,9 %) have to be used with probes having a Z gradient coil. In this case, you may start an acquisition using the **preempgp2.tst** pulse program (Standard <sup>1</sup>H acquisition parameters with GRD\_LENGTH (p16) = 2 ms, GRD\_WAVE\_FORM (GPNAM 1) = RECT.1, GRD\_STRENGTH (gpz1) = 50 %, GRD\_RECOVERY (d16) = 100 ms). Before starting the acquisition with "zg", you have to connect the GREAT Monitor Output (5) on an external electronic device like an oscilloscope. If the test is done when the GREAT is used together with the Digital Pre-emphasis Processor (DPP) board, be sure that all the pre-emphasis Gain values are set to 0 (see <u>Figure 6.4</u>, setpre section).

## **Great Calibration Operations**



Figure 3.1. Regulation current loop parameter settings. Pulse with an overshot (R = 74,9 % & C = 13,7 %)



Figure 3.2. Regulation current loop parameter settings. Pulse with an "undershot" (*R* = 43,1 % & *C* = 29,4 %)



*Figure 3.3. Regulation current loop parameter settings – The pulse shape edges are rectangular.* 

 $\rightarrow$  Correct values are R = 67,5 % & C = 17,6 %. For Z gradient coils of standard High Resolution probe.

## **Great Calibration Operations**

# **GREAT Service Web Pages**

4

The GREAT amplifiers new have a dedicated Web page. You can open this page either in the TopSpin interface using the "ha" order after selection of the BGA push button or via a Web browser via the BGA IP address attributed to the GREAT amplifier during the "cf" configuration procedure.

Hardware e	thernet addresses	
The hardware devices list and configured with a "W	ted below can be acce ÆB-Browser".	essed
Press the "Open" button f connection to this device Press the "Refetch addres addresses from DHCP set	for a browser with a sses" button to reload rver.	
Main Controller		
IPSO	149.236.99.253	Open
Digital Receiver Unit		
DRU1	149.236.99.89	Open
Amplifier		
BLA W1345083/0013	149.236.99.254	Open
Lock/Shim		
BSMS Z100818/0125	149.236.99.252	Open
Unknown Type		
BGA_W1213891_0014	149.236.99.243	Open
Refetch a	ddresses Print	Close

Figure 4.1. Access Window for the BGA Web Page using TopSpin hf Command.

The first BGA Web page which is displayed is the BGA Sevice Web page see *<u>Fig-ure 4.2.</u>* 

Eile Edit View	Go Bookmarks Tools	Mindow Help		
Back - Forwa	nd • 🙆 🔕	http://149.236.99.250/default.htm	V Search Print •	N
BRUKER	BGA Service Web			
14 internation	Bruker Gradient Device Information	Amplifier		
3A Reset	Nome	BGAC Grant 9-10		
and loss	Part number:	W1213762		
ert Log	Serial number:	0005		
A Service Report	Eot	0		
	Manufacturing location:	BFR		
content	Manufacturing date:	23/8/2006		
	BIS type:	BGA		
rverarie update				
	Software versions			
A Service Tools	Boot version:	20060124		
	Kernel version:	Windows CE 5.0		
	Annication vantion:	1.0 8 (20061023)		

Figure 4.2. Display of the BGA Service Web Page.

The **second BGA Web page** allows the user to reset the GREAT amplifier CPU in the same manner as an On/Off power switch without cutting power to the amplifier boards.

The **Event Log Web page** is for use by service personnel. On this page the last events resulting from orders coming from GREAT interface handling like offset changes, loop parameter changes, configuration operation etc. are summarized.

The **BGA Service Report Web page** is also for use by service personnel. It shows the status of the device. This Web page is used for troubleshooting only.

The **BIS Content Web page** shows all the parameters stored in the GREAT amplifier BIS, such as loop parameters, amplifier address, amplifier type etc.

The **Firmware Update Web page** is used for GREAT amplifier firmware updates. The actual CPU firmware boot and application version are displayed on the BGA Service Web page. As with all other spectrometer devices, if a new firmware is needed, it willd be delivered with the appropriate TopSpin version and can be downloaded using the firmware Update Web page shown in *Figure 4.3.* 



Figure 4.3. Display of the Firmware Update Web Page.

Select with the desired firmware version in the browser window and push the **Up-date** button. The procedure may take several minutes, do not interrupt it before the following message "**Firmware update was successful. The device will now restart**" is displayed.

The **BGA service Tools Web page** should be the much more used page as far as this page is used for:

**GREAT Channel Reset** (amplifier board protection): The GREAT amplifier includes a protection which puts the amplifier module in protective mode in order to ensure that no damage the probe occurs do to device mishandling. If the amplifier is in protective mode, you have to push the **Reset Protection** button to exit.

Auto Offset Calibration: When the GREAT is used together with the DPP, you have to toggle the Blanking Inhibition function to On. If you run an Auto Offset procedure for the first time you have to push the Configure button first and then the **Execute** button. Otherwise, you have only to push the **Execute** button. For further working this function has to be toggled to Off.

**Manual Offset correction**: Can be done by direct input of an integer value in the  $0 \le ADC$  offset  $\le 4096$ . The value corresponding to a current  $I \approx 0$  A should be found near an ADC value of 2048. To perform manual correction, you have to toggle the **Blanking Inhibition** function to **On**. Use the lock level which has to be optimized. If the adjustment is good enough you have to store the found ADC value by pushing on the store button. At the end of the operation don't forget to toggle the **Blanking Inhibition** function to **Off**.

**Loop Parameter Changing**: This is a manual operation. Use the standard preempgp2 pulse program and set the gradient strength (gpz1) to 50 %, the gradient length (p16) to 2 ms, the gradient recovery time (d16) to 100 ms and the gradient wave form (GPNAM1) to RECT.1. Connect an external electronic device like an oscilloscope on the GREAT monitor output. In "gs" mode, change the values of

R and C in the way to obtain a rectangular pulse on the display as shown in *Fig-ure 3.1.*, *Figure 3.2.* and *Figure 3.3.*).

**Configure Button**: This push button is used the first time you switch the power of your amplifier **On**. This order calibrates the gain of the offset correction loop. This value is directly stored.

Refresh Data: Display refresh order.

**Reset Protection**: Toggles the amplifier module from the Error status (if an error has occurred -> red LED) to the normal operating status (green LED).

**Blanking Threshold**: The blanking threshold function defines the current level at which the internal blanking switches the GREAT output MOS transistors **On/Off**. The default value is set to 0 for the positive and negative threshold. Changing this value may affect the pulse wave form and or the pre-emphasis correction current.



It is highly recommended to leave the positive/negative threshold set to the default values.

The **Service Command Web page** exists for the Z channel only if a GREAT 1/10 amplifier is used. In the case of the use of a GREAT 3/10 amplifier you have one Service Web page for each of the X, Y and Z channel (see *Figure 4.4.*).

18 (75)

		BGA Service Web	Mozilla		- E X
Eile Edit View	Go Bookmarks Tools Wine	dow Help			
Back - O	- 🗿 🙆 🗷	http://149.236.99.246/Service/SM	tain.htm	Search 3	Print -
BRUKER	BGA Service Web				
BGA information	Bruker Gradient An	nplifier			:
Channel Z	Service Command for	Channel Z			:
Back to main menu	Resistors	160 62.7 %	Change Loop Parameters		:
	Capacitors	42 16.5 %			:
	Othet	2250 9.9 %	Set Offset 0 <=	c= 4095	:
	ADC Offset value	2			
	AutoOffset	Execute	Stop		
	Calibration	Configure			:
	Current Parameters	Store			:
	Ampli Enable	On 💌			
	Secure Mode	Off -			
	Blanking Inhibition	Off •	# "On" is selected, the blanking from the Go	CON is inhibited	:
	Blanking Threshold	positive threshold: 0.000 A	Change Blanking Threshold	1	:
		negative threshold: 0.000 A		_	:
	Refresh data				
	Reset Protection				
	Bruker Access				:
					:
D DOVE Draker Breeze FA AV	Colors assessed				
Via 178 - 4 - 10					
100 CD 100 Edd	OEX Done				-0- 0.

Figure 4.4. Display of the Service Command Web page for the Z Channel of a GREAT 3/10 Amplifier.

E			GA Service	Web - Mozilla				
Elle Edit View (	<u>ao B</u> ookmarks <u>T</u> ools	Window Help						1
Back - Orward	Back       - O       O       Image: Antip://149.236.99.246/Service/SMain.htm         Image: Antip://149.236.99.246/Service/SMain.htm       Image: Antip://149.236.99.246/Service/SMain.htm							ati -
BRUKER 80	A Service Web	11/1						
BOA information	Bruker Gradient Amplifier							
Channel Z	LoopParamaters	for Channel Z						
Party In such states		Resistors				Capacito	rs	
and to the tree of	Current value	Value 160		Percentage 62.7 %	Current value	Vak 42	0	Percentage 16.5 %
	Now value	0 <= 160	<= 255	62.7 %	New value	0 42	<= 255	16.5 %
				Apply	/ data			

*Figure 4.5.* Display of the Window used for Loop Parameter Setting for the Z Channel of a GREAT 3/10 Amplifier.

File       Edit       View       Go       Bookmarks       Tools       Window Help         Back       Porward       Image: Search       Imag	Web - Mozilla	BGA Service Web
Back       Porward       Image: Image		Eile Edit View Go Bookmarks Tools Window Help
BOA Service Web BOA Information Chernel 2 Back to main meru Negative Threshold for the Zero detection Back to main meru Negative Threshold Current value 0,000 A Current value 0,000 A New value	ce/5Main.htm	Image: Service/
Book Information     Bruker Gradient Amplifier       Channel Z     Blanking Threshold for the Zero detection       Back to main meru     Negative Threshold Current value     Positive Threshold Current value       New value     0.000 A       New value     0.000 A		BRUKER BGA Service Web
Cherrel Z Blanking Threshold for the Zero detection  Negative Threshold Current value 0.000 A  New value 0.000 A  New value 0.000 cm125		Bruker Gradient Amplifier
Charmel 2 Blanking I hreshold for the Zero detection           Negative Threshold         Positive Threshold           Back to min meru         Negative Threshold         Current value         0.000 A           New value         1.25 cm + 10.000         cm 0         New value         0.000 A		Dially The hold of a Tarakanda
Back to main menu Current value New value 125 ce + 0,000 New value 0 cm 0 000 New value 0 cm 0 000 0 ce 125		Channel Z Blanking I hreshold for the Zero detection
Current value         0.000 A         Current value         0.000 A           New value         -1.25 cs - 0.000         cs 0         New value         0 cs 0.000 A	Positive Threshold	lack to main many Negative Threshold
New value -1.25 cs - 0.000 cs 0 New value 0 cs 0.000 cs 1.25	Current value 0.000 A	Current value 0.000 A
	New value 0 <= 0.000 <= 1.25	New value -1.25 <= - 0.000 <= 0

*Figure 4.6.* Display of the Window used for the internal GCON blanking threshold setting.

Note that the values are given in A. The negative threshold range is  $-1.25 \text{ A} \leq$ Threshold  $\leq 0 \text{ A}$ . The positive threshold range is  $0 \text{ A} \leq$  Threshold  $\leq 1.25 \text{ A}$ .

# **GRADIENT** Strength Calibration

The real gradient strength generated on a gradient coil by a gradient amplifier has to be known if diffusion experiments have to be carried out on a spectrometer. The value of the gradient strength  $G_{x,y,z}$  has to be known in G.cm<sup>-1</sup>.A<sup>-1</sup>. For X and Y gradient strength calibration a normal 5 mm diameter NMR tube can be used. For Z gradient strength calibration a spherical water sample, a Shigemi tube filled with less then 1 cm water or as in our case a 1.05 cm cylindrical sample tube put inside of a normal NMR tube.

Be sure that the center of the sample is located in the middle of the HF saddle coil. The **calibgp.tst** pulse program (see *Figure 5.1.*) is used. <sup>1</sup>H pulse length and power level are set as for gradient shiming profile recording. Use AQMOD = qsim and DIGMOD = analog. The spectral width may be set to 200 kHz digitized over 2 k data points. The total acquisition time is equal to 5.1 ms giving a defocusing gradient pulse length of aq/2 = 2.55 ms. The gradient wave form is automatically a square pulse, the gradient recovery delay (d16) is equal to 100 ms and the gradient strength is set between 5 and 100 %. It is possible to check with this test the real gradient system (GREAT amplifier + Z gradient coil) linearity by recording profiles between 5 and 100 % in 2,5 % steps. The gradient strength may be calculated on the gradient profile directly with the following formula or as the slope of the plot G = f(Gradient strength).

 $G = \Delta v / 4.258 \text{ x r}$ 

Where  $\Delta v$  is the profile width in kHz and r is the sample length in cm. G is given in G.cm<sup>-1</sup>.A<sup>-1</sup>. For a Z gradient coil of a HR probe used together with a GREAT amplifier, you may found  $5.3 \leq GZ \leq 5.8$  G.cm<sup>-1</sup>.A<sup>-1</sup>.



Figure 5.1. The calibgp Pulse Sequence used for Gradient Strength/linearity Measurement.



Figure 5.2. Gradient Profiles Recorded with a GREAT 3/10 Amplifier

The figure above shows gradient profiles recorded with a GREAT 3/10 amplifier on the Z channel used together with a 5 mm 1H/X Z Gradient BBO probe. The sample is a 1.05 cm height cylindrical glass tube put inside of a normal 5 mm NMR Willmad tube. The cylindrical glass sample is filled with pure non salty water. There is no deuterated solvent present. The profile are recorded without field lock. The lock signal sweep has to be set to Off.



Figure 5.3. Gradient Strength Linearity Plot G = f(Gradient Strength in %).

5.1

rpar CALIBGP.TST (all) Set pulprog to **calibgp.tst** p1 = pulse 90 at power level pl1 Set swh = 200000 Hz, AQMOD = qsim and DIGMOD = analog Set TD = 2 k data points and SI = 4 k data points. Set the number of scans NS = 2 Set GPNAM1 = RECT.1 Set the gradient recovery delay d16 = 100 µs Set the gradient strength gp21 to a value between 5 and 100% The acquisition time should be equal to 5 ms Record the gradient echo with "zg" Make a Fourier transformation with "ft" and calculate the magnitude component by use of the "mc" order.

For the gradient system linearity test, you have to record series of profiles with gradient strength set in a 5 to 100 % range in linear steps (2,5 % steps for example see *Figure 5.3.* 10).

## **GRADIENT Strength Calibration**

# GRADIENT Recovery/ Pre-emphasis Test

The gradient recovery test is part of the standard gradient amplifier test procedure. This test is used to evaluate the effect of Eddy currents occurring in gradient coils when the current is cut off. In the case of Z gradient coils built in standard HR probes, the Eddy currents are very low and pre-emphasis compensation is no longer needed (see *Figure 6.1.*). This is unfortunately not the case with Z gradient coils that are built in standard HR-MAS probes and X,Y,Z gradient coils that are built in standard HR probes. For an example of a gradient recovery test recorded without pre-emphasis with an HR-MAS probe see *Figure 6.2*.



Figure 6.1. Gradient recovery test recorded with a HR Z gradient probe.

The recovery delay changes in logarithmic steps starting at 10 ms up to 1 s. The first step was chosen equal to 10  $\mu$ s. The gradient pulse length (p16) is equal to 2 ms, the gradient wave form (GPNAM1) = RECT.1 and the gradient strength (gpz1) is set equal to 50 %.



Figure 6.2. The same test recorded with an HR-MAS Z gradient probe but the first step and the first increment are set to  $100 \ \mu$ s.

## Gradient Recovery Test Recording Procedure

6.1

For the HR experiment, use the doped water sample.

For the HR-MAS experiment, use the 2 mM sucrose in D<sub>2</sub>O rotor.

rpar PREEMPGP2.TST (all)

#### pulprog = preempgp2.tst

p1 = 90 degree pulse at power level pl1

O1 is put on resonance on the water peak

The spectral width SW\_p is set equal to 2,5 ppm for better peak resolution

Set TD = 16 k data points equal to SI

Set the number of scans NS = 1

The gradient pulse length is equal to 2 ms, the gradient strength is set to 50%, and the gradient wave form is RECT.1

Use the **paroptlog** automation program with logarithmic increments. The starting recovery delay d16 = 10  $\mu$ s (100  $\mu$ s for HR-MAS probes). The first increment is set equal to 10  $\mu$ s (100  $\mu$ s for HR-MAS probes) and the last value of d16 = 1 s. The multiplication factor depends on the number of experiments. For the here shown data 80 experiments are recorded. Thus the d16 multiplication factor is 1,17.

The gradient pre-emphasis test is done to match as good as possible resistance and capacities of 6 **identical** RC circuits in order to compensate the Eddy current effects on NMR FID's. For this purpose you have to record a set of 8 FID's with increasing recovery delays: 0,1 ms, 0,3 ms, 1 ms, 3 ms, 10 ms, 30 ms, 100 ms and 500 ms.

The experiments (see *Figure 6.3.*) have been recorded with standard proton parameters on a 4 mm standard bore HR-MAS probe with Z gradient coil using the 2 mM sucrose in D2O rotor.



Figure 6.3. Gradient Pre-emphasis test **without pre-emphasis compensation** as obtained with an HR-MAS probe with square gradient pulses.

On this gradient system, the pre-emphasis correction functions are digital. Thus the 6 **identical** exponential correction functions are digits added to the gradient shape before analogical conversion. Each pre-emphasis has a maximal amplitude of 10 % of the maximal number of digits used for gradient shape definition. If the maximal number of digits for gradient amplitude definition is  $2^{16}$  (16 bytes of the used 20 bits DAC), the maximal intensity of each pre-emphasis function is  $0.1 \times 2^{16} = 6554$  data points. With a gradient pulse current of 5 A, each pre-emphasis overshot may reach ± 0.5 A.

Generating pre-emphasis in this way make us sure that the pre-emphasis over pulse ratio is constant and independent of the gradient strength. Nevertheless this way of working has one drawback, the integral of the gradient shape for a pre-emphased 10 A square pulse is smaller then the integral of the corresponding non-pre-emphased square pulse, as far as the 6 k data points of the 2<sup>16</sup> available for pre-emphasis function definition.

The pre-emphasis adjustment is done inside of the setpre interface (see <u>*Figure*</u> <u>6.4.</u>). The longest time constant in the example shown in <u>*Figure*</u> <u>6.3.</u> is smaller then 50 ms.

SetPre					ດ້ ດີ	$\boxtimes$
GREAT 3/10	D-E Z preen	nphasis 🗾 👻 🗟 😄 🗢 🖋 🤶 🔀	Probe te	mp.	314°C	
O Auto Of	fset [%]		25.60	•	0.01	-
Time Base #1	Time [ms]		0.00000	-	0.00001	-
0.2 ms 💌	Gain [%]		0.00	-	0.01	-
Time Base #2	Time [ms]		0.00000	-	0.00001	-
0.2 ms 💌	Gain [%]		0.00	-	0.01	-
Time Base #3	Time [ms]		0.00000	•	0.00001	4
0.2 ms 💌	Gain [%]		0.00	•	0.01	-
Time Base #4	Time [ms]		0.00000	-	0.00001	•
0.2 ms 💌	Gain [%]		0.00	-	0.01	-
Time Base #5	Time [ms]		0.00000	-	0.00001	4
0.2 ms 💌	Gain [%]		0.00	-	0.01	-
Time Base #6	Time [ms]		0.00000	•	0.00001	•
0.2 ms 💌	Gain [%]		0.00	-	0.01	-
			Prob	ehe	ead : 32	

*Figure 6.4.* Display of the Main Window of the "**setpre**" Interface Showing the Pre-emphasis Sliders used for Eddy Current Compensation.

If the pre-emphasis values are adjusted, the 8 FID display (see *Figure 6.3.*) will look like *Figure 6.5.*. The results should be the same at every gradient strength.



Figure 6.5. Gradient Pre-emphasis Test with Pre-emphasis Values Adjusted as Obtained with an HR-MAS Probe with Square Gradient Pulses.

If you use the pre-emphasis values found, you may obtain the following results for the gradient recovery test with an HR-MAS probe. *Figure 6.6.* needs to be compared with *Figure 6.2.*.



Figure 6.6. Gradient Recovery Test Recorded with an HR-MAS Z Gradient Probe

With the first step and the first increment set to 100  $\mu$ s. Pre-emphasis are adjusted.

## Gradient Pre-emphasis Adjustment Procedure

6.2

For the HR-MAS experiment, use the 2 mM Sucrose in D<sub>2</sub>O rotor

### rpar PREEMPGP.TST

#### pulprog = preempgp.tst

p1 = 90 degree pulse at power level pl1

O1 is put 2 kHz off resonance of the water peak

The spectral width SW\_p is set equal to 10 ppm

Set TD = 16 k data points and SI equal to 8 k data points

Set the number of scans NS = 1

The gradient pulse length is equal to 5 ms, the gradient strength is set to 50 %, the gradient wave form is RECT.1

Use the **preemp** variable delay list (VDLIST = preemp)

Set the AQMOD to qsim and the DIGMOD to analog.

The pre-emphasis starting base values for HR-MAS Z gradient probes are given in the following table. It is understood that the regulation current resistance and capacities are matched beforehand.

Time Base 1	20 ms	τ1	10 ms	Gain 1	0,85 %
Time Base 2	2 ms	τ2	1,2 ms	Gain 2	-1,00 %
Time Base 3	2 ms	τ3	0,5 ms	Gain 3	1,00 %
Time Base 4	0,2 ms	τ4	0,2 ms	Gain 4	8,50 %
Time Base 5	0,2 ms	τ5	0,1 ms	Gain 5	-5,00 %
Time Base 6	0,2 ms	τ6	0,0 ms	Gain 6	0,00 %

# GRADIENT Echo Stability Test

# 7

This experiment is used to test the gradient pulse reliability. For this purpose a bipolar gradient is sent on the gradient coil and the NMR peak intensity and phase standard deviation are measured for over 6 minutes (short time test) and the room temperature averaged over 1 hour. In order to avoid that lock instabilities influence the result it is recommended to choose the LOCK POWER in a –20 to –25 dB range and to use the **loopadj** automation program to set LOOP GAIN, LOOP TIME, LOOP FILTER, LOCK PHASE and LOCK GAIN. Typically values used with the doped water sample (0,1 mg/ml GdCl<sub>3</sub> in 1% H<sub>2</sub>O / 99% D<sub>2</sub>O ) are:

LOCK POWER: -25  $\leq$  LOCK POWER  $\leq$  -20

LOCK GAIN: 92  $\leq$  LOCK GAIN  $\leq$  102

LOOP GAIN: -2  $\leq$  LOOP GAIN  $\leq$  0

LOOP TIME: 0.2 s  $\leq$  LOOP TIME  $\leq$  0,25 s

LOOP FILTER:  $80 \le LOOP$  FILTER  $\le 120$ .

The tests are recorded using the **gradecho2d.tst** pulse program (see *Figure 7.1.*). The amplitude/phase standard deviation calculations are performed with the **gradstab\_test** automation program. Results obtained on a GREAT 3/10 for the 6 min short time test and the time average test are shown in *Figure 7.2.* and *Figure 7.3.* 



Figure 7.1. Pulse Sequence used for the Gradient Echo Short Time and Temperature Averaged Stability Test.

This sequence is also used for the water self diffusion coefficient measurement.

### Gradient Stability Experiment Recording Procedure

The experiment is recorded in 2D mode using the **gradecho2d.tst** pulse program.

For the HR experiment, use the doped water sample.

For the HR-MAS experiment, use the 2 mM Sucrose in D<sub>2</sub>O rotor.

Use standard 2D proton acquisition parameters -> rpar GRADECHO2D (all)

pulprog = gradecho2d.tst

p1 = 90 degree pulse at power level pl1

O1 is put on resonance on the water peak.

The spectral width SW\_p is set equal to 2,5 ppm for better peak resolution.

Set TD = 16 k data points equal to SI.

Set TD1 = 64 Words (short time test) and TD1 = 256 Words (time averaged test).

Set the number of scans NS = 1.

Set (d1 + aq) = 5 s for the short time test and (d1 + aq) = 15 s for the time averaged test.

The gradient pulse length is equal to 2 ms, the gradient strength is set to 25 %, the gradient wave form is RECT.1 and the recovery delay is set to 100  $\mu s.$ 

The spectra are processed with xf2 and phased

Run the **grad\_stab** automation program  $\rightarrow$  from 2D to 1D paropt type file. The grad\_stab automation program calculate the mean Intensity, the mean phase deviation and there respective standard deviation  $\sigma^2$ .



Figure 7.2. Gradient Echo Stability Test

Recorded over 6 mn (64 data points in  $f_1$ ), 1D resulting paropt type file.

**BRUKER BIOSPIN** 



Figure 7.3. Gradient Echo Time Averaged Test

Recorded over 1 hour (256 data points in  $f_1$ ), 1D resulting paropt type file.

When using a gradient echo sequence, the spectrometer stability results must be in the following ranges:

	Peak Intensity $\sigma^2$	Peak Phase $\sigma^2$
Short Time Stability	≤ 0,05 %	$\leq$ 0,5 deg.
Time Averaged Stability	≤ 0,10 %	≤ 1,0 deg.

Check your lock parameters to be sure that the field compensation loop works in the best conditions.

![](_page_32_Picture_7.jpeg)

Note that the here shown spectrometer stability during a gradient echo pulse sequence were obtained at room temperature without any sample temperature regulation.

## **GRADIENT Echo Stability Test**

## Water Diffusion Coefficient Measurement Using a 1D Gradient Echo Pulse Sequencer

# 8

This is the final gradient amplifier test. This test is working if the pre-emphasis are properly adjusted, if the gradient amplifier gradient echo stability is inside the defined stability range, if the gradient amplifier accuracy test is found into the specified range and finally if the gradient amplifier offset is properly adjusted. The test is done on the doped water sample (0,1 mg/ml GdCl<sub>3</sub> in 1% Water + 99% D<sub>2</sub>O) at 25 C. We use the gradient echo pulse sequence **gradecho2.tst** or the gradient Hahn Echo **gradecho\_180.tst** pulse sequence with a  $\delta$  of 1,6 ms and a  $\Delta$  of 30 ms. The gradient wave form used is a square and the gradient recovery delay is set to 100 µs. The test is recorded in paropt mode using the "**paroptlin**" automation program with the gradient strength (gpz<sub>1</sub> or gpy<sub>1</sub> or gpx<sub>1</sub>) changing in 2,5% steps starting at 0% up to 100%. The following exponential decreasing curve should be obtained for the gradient echo experiment (see *Figure 8.1.*).

![](_page_34_Picture_3.jpeg)

Figure 8.1. Water Self Diffusion Curve Obtained on the Doped Water Sample at 25 °C.

![](_page_35_Figure_1.jpeg)

Figure 8.2. Water Self Diffusion Curve Obtained on the Doped Water Sample at 25 °C.

If we plot for both experiments log  $(I/I_0) = f(G^2)$  you should obtained the straight lines shown in *Figure 8.3.* and *Figure 8.4.* respectively for the gradient echo and the gradient Hahn Echo experiments. The water self diffusion coefficient calculated on the basis of the straight line slope should be found in a range between 2,1 x  $10^{-9}$  and 2,3 x  $10^{-9}$  m<sup>2</sup>.s<sup>-1</sup>. The calculation assume that for gradient square pulses:

 $\mathsf{D}|| = -\mathsf{Slope} / \gamma_{\mathsf{H}}^2 \,\delta^2 \,(\Delta - \delta/3)$


Figure 8.3. Plot of  $log(I/I_0) = f(G^2)$  Obtained with the Gradient Echo Experiment.



Figure 8.4. Plot of log(1/10) = f(G2) Obtained with the Gradient Hahn Echo Experiment.

The two plots should give nearly the same results. In the example shown here, the value of the water self diffusion coefficient calculated with the data of the plot shown in <u>*Figure 8.3.*</u> is equal to  $2,28 \times 10^{-9} \text{ m}^2.\text{s}^{-1}$  and the value of the water self diffusion coefficient calculated with the data of the plot shown in <u>*Figure 8.4.*</u> is equal to  $2,11 \times 10^{-9} \text{ m}^2.\text{s}^{-1}$ .

## Miscellaneous

9.1

#### The GCUTYPE and the GRADRES Parameters

As shown in part 1 of this manual, the GREAT 1/10 and the GREAT 3/10 amplifiers have been designed to be able to work with an internal blanking and with an external 0/-5 V blanking signal (T0 signals available on the IPSO front Panel). The GCON board LVDS output has to be connected on the DPP board LVDS input and the DPP board LVDS output on the GREAT LVDS input. In this way, it is possible to work with or without pre-emphasis and with the external or internal blanking. If a GREAT amplifier is detected by the Hardware, the spectrometer assume that there are both a GREAT and a DPP board inside. The software set in the *I* <disk>/topspin/conf/instr/spect /uxnmr.par file the GCUTYPE parameter to the value 11 and the GRADRES (Gradient pulse resolution) to 5 ms (default value). The value GRADRES defines the number of pre-emphasis correction functions.

GRADRES = 2  $\mu$ s -> the GREAT works with all 6 pre-emphasis functions disabled in the same way as the GAB.

GRADRES = 5  $\mu$ s -> the GREAT works with 3 of the 6 pre-emphasis functions enabled, the other 3 may not be used.

GRADRES = 10  $\mu$ s -> the GREAT works with all the 6 pre-emphasis functions enabled.

The system uses the following orders of the Grad.incl file to manage GREAT amplifier blanking/unblanking. The external blanking orders (T0 word bit 32, 33 and 34) are **only available on 19" IPSO computers**. For TopSpin 2.0 pl3 software and higher versions, external blanking is no longer needed. TopSpin is able to handle the GCON outcoming ctrlgrad xx orders properly. Thus all the existing pulse programs using gradient pulsing mat be used if the external blanking cables are connected or not. External blanking is used if the **blanking inhibition** function of the GREAT Web page is toggled to **On**. If the **blanking inhibition** function of the GREAT Web page is toggled to **Off**, the internal blanking coming from the GCON is used and the external blanking is disabled.

#### Grad.incl

9.1.1

....

#define UNBLKGRAD setnmr 3|0 setnmr 0|32|33|34 ctrlgrad 0 #define BLKGRAD setnmr 3^0 setnmr 0^32^33^34 ctrlgrad 7 On the 19" IPSO computer, 3 blanking signals (0, -5V) are available:

nmr word 0 bit 32 for the X channel (Signal 4 of the IPSO A connector) nmr word 0 bit 33 for the Y channel (Signal 5 of the IPSO A connector) nmr word 0 bit 34 for the Z channel (Signal 6 of the IPSO A connector)

On the AQS IPSO computer, the external blanking signals are no longer available. If an AQS IPSO computer is used with a GREAT 10 amplifier, the gradient amplifier blanking is handled by the GCON out coming ctrlgrad xx orders if a DPP board is present or not.

The following table shows the syntax and meaning of the ctrlgrad xx blanking orders.

ctrlgrad	Binary	В0	Z	Y	X
0	0000	UNBLK	UNBLK	UNBLK	UNBLK
1	0001	UNBLK	UNBLK	UNBLK	BLK
2	0010	UNBLK	UNBLK	BLK	UNBLK
3	0011	UNBLK	UNBLK	BLK	BLK
4	0100	UNBLK	BLK	UNBLK	UNBLK
5	0101	UNBLK	BLK	UNBLK	BLK
6	0110	UNBLK	BLK	BLK	UNBLK
7	0111	UNBLK	BLK	BLK	BLK
8	1000	BLK	UNBLK	UNBLK	UNBLK
9	1001	BLK	UNBLK	UNBLK	BLK
10	1010	BLK	UNBLK	BLK	UNBLK
11	1011	BLK	UNBLK	BLK	BLK
12	1100	BLK	BLK	UNBLK	UNBLK
13	1101	BLK	BLK	UNBLK	BLK
14	1110	BLK	BLK	BLK	UNBLK
15	1111	BLK	BLK	BLK	BLK

UNBLK means that the channel is unblanked.

BLK means that the channel is blanked -> the gradient amplifier channel output is isolated from the probe (Auto offset should be not possible if the **Blanking Inhibi-tion** function is set to **Off**).

#### How Does the GREAT 10 Blanking Work with a 19" IPSO Computer?

9.3

There is only one way to select the kind of blanking the user want to use: the **blanking inhibition** function of the GREAT 10 channel hosted on the BGA Web page shown in *Figure 4.4.*, *Figure 4.5.*, *Figure 4.6.*. It is possible to test the two ways of blanking of a GREAT amplifier by using a digital oscilloscope. On channel 1, connect the GREAT X, Y or Z channel Monitor output. On channel 2, connect the X, Y or Z external blanking input coming from connector A. On channel 3, connect the TP 42 position (inside of the GREAT) corresponding to the logical state of the blanking MOS transistor (see Gradient Manual on the BASH).

For this test you can use a modified gradient include file **Grad2.incl** together with a modified version of the **gradecho2.tst** as shown below.

Grad2.incl

9.3.1

#define UNBLKGRAD setnmr 3 0 ctrlgrad 0
#define BLKGRAD setnmr 3^0 ctrlgrad 7
#define UNBLKGRAMP setnmr 0 32 33 34
#define BLKGRAMP setnmr 0^32^33^34
;gradecho2.tst
;avance-version (00/02/07)
;pulseprogram for test gradient and preemphasis adjustment
;
;\$CLASS=HighRes
;\$DIM=1D
;\$TYPE=
;\$SUBTYPE=
;\$COMMENT=
;\$OWNER=bruker
#include <avance.incl></avance.incl>
#include <grad2.incl></grad2.incl>
"d11=30m"
1 ze
2 d1
5m UNBLKGRAMP

1m UNBLKGRAD p1:f1 ph1:r p16:gp1 d16 p16:gp1\*-1 d16 1m BLKGRAD 50u BLKGRAMP go=2 ph31 wr #0 d11 exit ph1=0 ph31=0 ;pl1: f1 channel - power level for pulse (default) ;p1: f1 channel - 90 degree high power pulse ;p16: homospoil/gradient pulse ;d1: relaxation delay; 1-5 \* T1 ;d11: delay for disk I/O [30 msec]

;d16: delay for homospoil/gradient recovery ;use gradient: gp 1

;\$Id: preempgp2,v 1.8 2005/11/10 12:17:01 ber Exp \$

9.4

#### Blanking Inhibition = On -> External Blanking Mode

The screen display shown below shows how the external blanking works. The ctrlgrad 0 and ctrlgrad 7 orders are sent from the GCON but they are ignored by the GREAT 10 amplifier.



Figure 9.1. GREAT 10 Z channel External blanking during a gradient echo experiment.

The **blanking inhibition** function is set to **On**. The violet trace is measured at Z monitor output, the green trace is measured at Z channel blanking input and the yellow trace is measured at the TP 42 position on the GREAT interface board.

### Blanking Inhibition = Off -> Internal blanking mode

The screen display shown in *Figure 9.2.* shows how the internal blanking works. The setnmr 0 bit 32,33 and 34 signals are sent on the T0 A connector but they are ignored by the GREAT 10 amplifier.



*Figure 9.2. GREAT 10 Z channel Internal blanking during a gradient echo experiment.* 

The **blanking Inhibition** function is set to **Off**. The violet trace is measured at Z monitor output, the green trace is measured at Z channel blanking input and the yellow trace is measured at the TP 42 position on the GREAT interface board.

9.5

### Blanking Inhibition = On -> External Blanking Mode with Pre-emphasis 9.6

The screen display shown in *Figure 9.3.* shows how the external blanking works. The ctrlgrad 0 and ctrlgrad 7 orders are sent from the GCON but they are ignored by the GREAT 10 amplifier. For this test the following pre-emphasis values have been used and the second delay used before the BLKGRAD order in the **gradecho2.tst** pulse program has been set to 100  $\mu$ s. As may be seen on the display (see *Figure 9.3.*), the external blanking cut off the pre-emphasis signal before its end.

Time Base 1 = 0.2 ms – Time Constant 1 = 0.1 ms – Gain 1 = 10 %Time Base 2 = 0.2 ms – Time Constant 2 = 0.1 ms – Gain 2 = 10 %Time Base 3 = 0.2 ms – Time Constant 3 = 0.1 ms – Gain 3 = 10 %



Figure 9.3. GREAT 10 Z channel External blanking during a gradient echo experiment.

The **blanking Inhibition** function is set to **On**. The violet trace is measured at Z monitor output, the green trace is measured at Z channel blanking input and the yellow trace is measured at the TP 42 position on the GREAT interface board.

#### Blanking Inhibition = Off -> Internal blanking mode with pre-emphasis 9.7

The screen display shown in *Figure 9.4.* shows how the internal blanking works. The setnmr 0 bit 32,33 and 34 signals are sent on the T0 A connector but they are ignored by the GREAT 10 amplifier. For this test the following pre-emphasis values have been used and the delays used before the BLKGRAD/UNBLKGRAD orders in the **gradecho2.tst** pulse program have been set to 100  $\mu$ s. As may be seen on the display see *Figure 9.3.*, the external blanking cut off the pre-emphasis signal before its end.

Time Base 1 = 0,2 ms – Time Constant 1 = 0,1 ms – Gain 1 = 10 % Time Base 2 = 0,2 ms – Time Constant 2 = 0,1 ms – Gain 2 = 10 % Time Base 3 = 0,2 ms – Time Constant 3 = 0,1 ms – Gain 3 = 10 %



Figure 9.4. GREAT 10 Z channel External blanking during a gradient echo experiment.

The **blanking Inhibition** function is set to **Off**. The violet trace is measured at Z monitor output, the green trace is measured at Z channel blanking input and the yellow trace is measured at the TP 42 position on the GREAT interface board.

#### How Does the GREAT 10 Blanking Work with an AQS IPSO Computer? 9.8

In this case, the **blanking inhibition** function has to be set to **Off**. Thus only the internal blanking is operating.

**Contact for Further Questions?** 

For further question, please send an E-mail to the following address: philippe.lux@bruker.fr

9.9

## 1D Gradient "Shimming" Operations

# 10

In modern NMR spectroscopy, the Gradient "Shimming" is a procedure which allows to correct the possible  $B_0$  field homogeneity around the sample volume. It can be done by hand or by using a gradient echo experiment (*Figure 10.1.*) called the gradient shimming procedure ("Gradient Shimming: Principles and Practical Aspects, M. Weiger, D. Moskau, R. Kerssebaum & W.E. Hull edited by BRUKER BIOSPIN"). In 1D gradient shimming we use two gradient coil Z profiles recorded the first with an echo time of 5 ms and the second one with an echo time of 25 ms. The comparison of both allow the calculation of factors related to  $B_0$  non homogeneity. The procedure changes the values of the Z,  $Z^2$ ,  $Z^3$ ,  $Z^4$  and  $Z^5$  shims in order to correct the homogeneity.



Figure 10.1. Gradient Echo pulse scheme as used for 1D gradient Shimming. Only the Z shims are matched with this experiment.

#### 1D Gradient Shimming Recording Procedure

rpar gradshim1d1H (all)

The pulse program must be imgegp1d

RG = 128 and p0 = p90 at power level pl1 when using  $1 \% H_2O$ 

RG = 32 and p0 = 2  $\mu$ s at pl1 = 16 dB when using 90 % H<sub>2</sub>O.

SWh must be 40000 Hz for an acquisition time of 6 ms (TD2 = 512 W)

Number of scans = 1

Use the **gradshim** order on the TopSpin user interface to define the shim set which has to be matched and the number of iterations.

If there is no field Map present for the used probe, go in the **gradshim** set up menu and choice the shim set corresponding to your system (including or not shim  $Z^6$ ) and **start Field Mapping**.



Note that on the Gradient amplifier Web page on the Service Tool Display, the Blanking Inhibition function has to be toggled to Off.

If the shim mapping procedure is finished (needs more or less half an hour), you may obtain shim profiles as shown in *Figure 10.2.*.



Figure 10.2. 1D Shim Map Resulting from the Shim Mapping Setup Procedure.

There should be one 1D field map set for each probe. The maps are used for further gradient shimming with the same probe.

The red curve is the Z profile, the dark green curve is the  $Z^2$  profile, the grey curve is the  $Z^3$  profile, the blue curve is the  $Z^4$  profile and finally, the green curve is the  $Z^5$  profile. The Z shim maps shown in *Figure 10.2.* have been recorded on the 1% doped water sample on an AVANCE II+ 400 MHz equipped with a 5 mm TXI <sup>1</sup>H/ <sup>13</sup>C/<sup>15</sup>N X, Y, Z gradient probe. The experiment have been done with the standard gradshim1d1H parameters by setting p0 to a 90 degree flip angle at power level pl1 and using a receiver gain RG = 128 (in order to obtain a good S/N on the Z dimension profile).

Gradient shimming can now been done. Currently you may use the parameter sets shown on the **gradshim** display of *Figure 10.3.*. Two iteration may respectively recorded on highZ (Z,  $Z^2$ ,  $Z^3$ ,  $Z^4$ ,  $Z^5$ ) and midZ (Z,  $Z^2$ ,  $Z^3$ ) to obtain a good shim values set as see *Figure 10.4.*.



Figure 10.3. Display of the Main window used for 1D gradient shimming.

Generally one experiment with two iterations (highZ and midZ) are sufficient to obtain a correct shim set.



Figure 10.4. Z profile obtained after the two steps of 1D gradient shimming

Recorded according experiment settings of *Figure 10.3.* 

The spectra shown in <u>Figure 10.5.</u> and <u>Figure 10.6.</u> have been respectively obtained before and after 1D gradient shimming of the 5 mm TXI  $^{1}$ H/ $^{13}$ C/ $^{15}$ N X, Y, Z gradients probe. On the spectrum shown in <u>Figure 10.5.</u> a shim set of another probe was used as starting value. The spectrum shown in <u>Figure 10.6.</u> was obtained after the two 1D/ $^{1}$ H gradient shimming steps.



Figure 10.5. <sup>1</sup>H Spectrum of the doped water sample

(0.1 mg/ml GdCl<sub>3</sub> in 1%  $H_2O + D_2O$ ) obtained using a shim set coming from another probe.



Figure 10.6. 1H Spectrum of the doped water sample

(0,1 Mg/ml Gdcl<sub>3</sub> In 1%  $H_2$ o +  $D_2$ o) Obtained After The Two 1d/<sup>1</sup>h Gradient Shimming Iterations.

## 11

## 3D Gradient "Shimmimg" Operation on X, Y, Z Gradient Coil

The 1D gradient shimming procedure is shown in chapter 9 of this manual. It is possible to achieve gradient shimming on all shims even the planar shims X, Y and there combinations. The procedure is called 3D gradient shimming (*Figure* <u>11.1</u>.). This time we record a 3D image of the water sample homogeneity inside of the RF coil volume. The experiment is recorded in 3D mode and needs three dimensional gradient pulse generation. The experiment uses X and Y gradients for phase encoding and the Z gradient for frequency encoding (Z profile). The sequence shown in *Figure* <u>11.1</u>. is used. For each of the 32 X gradient pulse strength increment  $-2.5 \le G_X \le 2.5$  G.cm<sup>-1</sup>, 32 Y gradient pulse strength increment transformation of the 1024 recorded echo's leads to a 3D image of the field homogeneity of the sample volume inside of the RF coil.





All the X, Y, Z shims and there combinations are matched with this experiment.

#### 3D Gradient Shimming Recording Procedure

#### rpar gradshimdata (all)

The pulse program must be imgegp3d

RG = 128 and p0 = p90 at power level pl1 when using 1 %  $H_2O$ 

RG = 32 and p0 = 2  $\mu$ s at pl1 = 16 dB when using 90 % H<sub>2</sub>O.

SWh must be 40000 Hz for an acquisition time of 3 ms (TD2 = 256 W)

Number of scans = 1

Use the **gradshim** order on the TopSpin user interface to define the shim set which has to be matched and the number of iterations.

If there is no field Map present for the used probe, go in the **gradshim** set up menu and choice the shim set corresponding to your system (including or not shim  $Z^6$ ) and **start Field Mapping**.

X gradient strength: -5.557  $< G_X < 5.557 \%$ 

Y gradient strength: -5.557 <  $G_Y$  < 5.557 %

Z gradients:  $G_{Z1}$  = 3.11 % and  $G_{Z2}$  = -6.23 %



Note that on the Gradient amplifier Web page on the Service Tool Display, the Blanking Inhibition function has to be toggled to Off.

If the shim mapping procedure is finished (needs more or less half an hour), you may obtain shim profiles as shown in *Figure 11.2.* 



Figure 11.2. Shim Map resulting from the Shim Mapping Set Up Procedure.

There should be one 3D field map set for each probe. The maps are used for further gradient shimming with the same probe.

The red curve is the Z profile, the dark green curve is the Z<sup>2</sup> profile, the green curve is the Z<sup>3</sup> profile, the blue curve is the Z<sup>4</sup> profile and finally, the gray curve is the Z<sup>5</sup> profile. The Z shim maps shown in *Figure 11.2.* have been recorded on the 2 mM Sucrose sample in water (90% H<sub>2</sub>O/10% D<sub>2</sub>O) on an AVANCE II+ 400 MHz equipped with a 5 mm TXI <sup>1</sup>H/<sup>13</sup>C/<sup>15</sup>N X, Y, Z gradient probe. The experiment have been done with the standard **gradshimdata** parameters by setting p0 to 2  $\mu$ s at power level pl1= 12 dB and using a receiver gain RG = 32 (in order to obtain a good S/N on the Z dimension profile and avoid saturation).

Gradient shimming can now been done. Currently you may use the parameter sets shown on the **gradshim** display for 3D gradient shimming. The shim set selection depends on your shim system (BOSS 1 or BOSS 2). The number of gradient shimming iterations depends how far away the shim values of the starting shim set are against the good values. With a maximum of **four iterations** you should obtain a good shim set. A view of the obtained 3D image as well as two projections are shown on *Figure 11.4.* 

With the shim set obtained after the two iteration 3D gradient shimming you should obtain a proton sucrose spectrum with water pre-saturation which should look like the spectrum shown in *Figure 11.5.* 



Figure 11.3. Z Field maps obtained after two 3D gradient shimming iterations.

Figure 11.3: The probe which had to be shimmed was a 5 mm TXI X, Y, Z Gradient Coil and the starting shim was used with a 5 mm BBO Z Gradient probe.



Figure 11.4. A) Three dimensional image of the water sample

Contained inside the RF coil after 3D Fourier Transformation of the gradient echo FID's; B) F1-F2 projection of the 3D image and C) F2-F3 projection of the 3D image.



Figure 11.5. <sup>1</sup>H proton spectrum with water pre-saturation obtained with a 2 mM

Sucrose sample in water (90 %  $H_2O$  + 10 %  $D_2O$ ) using the shim set found with the 3D gradient shimming experiment.

## 12

## 3D Gradient "Shimming" Operation on Z Gradient Coil

The 3D gradient shimming procedure used for probes having an X, Y, Z gradient coil is shown in chapter 10 of this manual. It is possible to achieve gradient shimming on all shims even the planar shims X, Y and there combinations on a current Z gradient coil probe. The procedure is called 3D RCB gradient shimming. This time we record a 3D image of the water sample homogeneity inside of the RF coil volume. The experiment is recorded in 3D mode and needs three dimensional gradient pulse generation. The experiment uses X and Y gradients for phase encoding and the Z gradient for frequency encoding (Z profile). The sequence shown in *Figure 12.1.* is used. For each of the 32 X gradient pulse strength increment  $-2.5 \le G_X \le 2.5$  G.cm<sup>-1</sup>, 32 Y gradient pulse strength increments  $-2.5 \le G_Y \le 2.5$  G.cm<sup>-1</sup> and the Z profile are recorded. The X and Y gradient pulses are generated on with the shim coils using the BSMS shim current generation. The 3D Fourier transformation of the 1024 recorded echo's leads to a 3D image of the field homogeneity of the sample volume inside of the RF coil.



Figure 12.1. Gradient Echo pulse scheme as used for 3D RCB gradient Shimming.

All the X, Y, Z shims and there combinations are matched with this experiment.

rpar gradshim3Drcb (all)

The pulse program must be imrcpgegp3d

RG = 128 and p0 = p90 at power level pl1 when using  $1 \% H_2O$ 

RG = 32 and p0 = 2  $\mu$ s at pl1 = 16 dB when using 90 % H<sub>2</sub>O.

SWh must be 40000 Hz for an acquisition time of 3 ms (TD2 = 256 W)

Number of scans = 1

Use the **gradshim** order on the TopSpin user interface to define the shim set which has to be matched and the number of iterations.

If there is no field Map present for the used probe, go in the **gradshim** set up menu and choice the shim set corresponding to your system (including or not shim  $Z^6$ ) and **start Field Mapping**.

Z gradients:  $G_{Z1}$  = 3.11 % and  $G_{Z2}$  = -6.23 %



Note that on the Gradient amplifier Web page on the Service Tool Display, the Blanking Inhibition function has to be toggled to Off.

Gradient shimming can now been done. Currently you may use the parameter sets shown on the **gradshim** display generally used for the 3D RCB gradient shimming. One or two iterations are necessary on shim 19 (BOSS 1: all shims with the exception of Z6) or on shim 27 (BOSS 2: all shims with the exception of Z6) should be sufficient to obtain a good shim set. The criteria to stop or continue the iteration procedure depends on the flatness of the final field map obtained at the end of each step. If the Z field profile looks like in *Figure 12.2.* a), you need to run further steps until the field map looks like the profile shown in *Figure 12.2.* b).



Figure 12.2. Z Field maps obtained after two 3D gradient shimming iterations.

The probe which had to be shimmed was a 5 mm TXI X, Y, Z Gradient Coil and the starting shim was used with a 5 mm BBO Z Gradient probe.



Figure 12.3. A) Three dimensional image of the water sample

contained inside the RF coil after 3D Fourier Transformation of the gradient echo FID's; B) F1-F2 diagonal map extracted from the 3D image and C) F2-F3 diagonal map extracted from the 3D image.

## Frequently Asked Questions (FAQ)

## 13

## I use a gradient pulse sequence and nothing happens on the lock level.

Be sure that the gradient strength parameters relative to the used channel X, Y, Z are different of 0.

If the gradient strength are properly set, go to BGA Service Tools and check if the used **amplifier module** is set to **on**.

Be sure that the Blanking Inhibition is set to off.

If all the parameters are correct put the **Blanking Inhibition** to **on** and change the offset DAC value to another value then 2048 (between 0 and 4096). Push the **Set DAC value** button. If the lock level moves then go to the next step. If nothing happens on the lock level, the gradient coil is damaged -> Sent the probe back to your service office.

#### I have no Gradient Pulse and I am sure that the coil is not damaged.

Sometimes bad data bytes are transferred from the GCONtroller and/or the DPP board to the GRADIENT amplifier DAC.

Switch the GREAT power supply **On/Off** with button (10).

Restart the IPSO with the red button on the IPSO computer Front Panel.

Wait IPSO stabilisation.

Do a "cf" spectrometer configuration procedure.

Do a GREAT offset calibration on the used gradient channel.

Try pulsing again, the gradient amplifier should work. If it is not the case, please go to the next step.

## I have done the IPSO restart procedure but the GREAT does not pulse.

If you use the DPP together with the GREAT amplifier, switch the IPSO computer and the GREAT amplifier **Off**.

Connect the GCONtroller LVDS output directly on the GREAT LVDS input.

Switch the GREAT amplifier and the IPSO computer **On**.

Do a "cf" spectrometer configuration procedure.

Enter the **nodpp** order in the TopSpin interface.

Do a GREAT offset calibration on the used gradient channel.

Try pulsing again, the gradient amplifier should work. If it is not the case, please go to the next step.

If the amplifier works, the Digital Pre-emphasis Processor (DPP) does not work properly -> call your service office.

If the amplifier does not pulse after all the tests, please call your service office.

## For all other questions, you can send an E-mail to the following address:

philippe.lux@bruker.fr.

## *GREAT 1/10 - GREAT 3/10 Package*

## 14

The GREAT 1/10 or GREAT 3/10 package consists of:

- GREAT 1/10 (W1213891) or GREAT 3/10 (W1213762) amplifier.
- 1 Ethernet cable 2m length (85997).
- 1 LVDS cable 0,5 m length (HZ13656).
- 1 LVDS cable 1 m length (86868) or 2 m length (87925).
- 1 Digital Pre-emphasis Processor (DPP) (H12513F1).
- 1 CD rom containing the User and Installation Manual, test parameters, pulse programs and automation programs (W1214559/EC00).
- 1 gradient amplifier output cable (W1213791) for GREAT 1/10 or (W1213792) for GREAT 3/10.
- 1 blanking cable with 6 blanking output's (HZ14004).

### GREAT 1/10 - GREAT 3/10 Package

## Figures

1 Introd	uction 5
2 Startii	ng with the GREAT Gradient Amplifier 7
Figure 2.1. Figure 2.2. Figure 2.3.	GREAT 1/10 Gradient Amplifier Front Panel
3 Great	Calibration Operations 11
Figure 3.1.	Regulation current loop parameter settings. Pulse with an over- shot ( $R = 74.9 \% \& C = 13.7 \%$ )
Figure 3.2.	Regulation current loop parameter settings. Pulse with an "un- dershot" ( $R = 43.1 \% \& C = 29.4 \%$ )
Figure 3.3.	Regulation current loop parameter settings – The pulse shape edges are rectangular
4 GREA	T Service Web Pages 15
Figure 4.1.	Access Window for the BGA Web Page using TopSpin hf Com- mand
Figure 4.2.	Display of the BGA Service Web Page
Figure 4.3.	Display of the Firmware Update Web Page17
Figure 4.4.	Display of the Service Command Web page for the Z Channel of a GREAT 3/10 Amplifier
Figure 4.5.	Display of the Window used for Loop Parameter Setting for the Z Channel of a GREAT 3/10 Amplifier
Figure 4.6.	Display of the Window used for the internal GCON blanking threshold setting
5 GRAD	IENT Strength Calibration 21
Figure 5.1.	The calibgp Pulse Sequence used for Gradient Strength/linear- ity Measurement
Figure 5.2.	Gradient Profiles Recorded with a GREAT 3/10 Amplifier22
Figure 5.3.	Gradient Strength Linearity Plot G = f(Gradient Strength in %).
6 GRAD	IENT Recovery/Pre-emphasis Test 25
Figure 6.1.	Gradient recovery test recorded with a HR Z gradient probe
Figure 6.2.	The same test recorded with an HR-MAS Z gradient probe but
Figure 6.3.	Gradient Pre-emphasis test without pre-emphasis compensa- tion as obtained with an HR-MAS probe with square gradient
	-

Figure 6.4.	pulses Display of the Main Window of the "setpre" Interface Showin the Pre-emphasis Sliders used for Eddy Current Compensatio	. 27 Ig on. 28
Figure 6.5.	Gradient Pre-emphasis Test with Pre-emphasis Values Adjust as Obtained with an HR-MAS Probe with Square Gradient Pr	. 20 ted uls- 28
Figure 6.6.	Gradient Recovery Test Recorded with an HR-MAS Z Gradie Probe	. 20 ent . 29
7 GRAD	IENT Echo Stability Test	31
Figure 7.1.	Pulse Sequence used for the Gradient Echo Short Time and	
Figuro 7.2	Temperature Averaged Stability Test.	. 31
Figure 7.3.	Gradient Echo Time Averaged Test	. 33
8 Water	Diffusion Coefficient Measurement Using a 1D	
Gradie	ent Echo Pulse Sequencer	35
Figure 8.1.	Water Self Diffusion Curve Obtained on the Doped Water Sa	3m- 35
Figure 8.2.	Water Self Diffusion Curve Obtained on the Doped Water Sa	. 55 am-
-	ple at 25 °C.	. 36
Figure 8.3.	Plot of log(I/I0) = f(G2) Obtained with the Gradient Echo Exp iment.	)er- . 37
Figure 8.4.	Plot of log(I/I0) = f(G2) Obtained with the Gradient Hahn Ech Experiment.	וס . 37
9 Misce	llaneous	39
Figure 9.1.	GREAT 10 Z channel External blanking during a gradient ec	ho ⊿3
Figure 9.2.	GREAT 10 Z channel Internal blanking during a gradient ech	10
Figure 9.3.	GREAT 10 Z channel External blanking during a gradient ec	. 44 ho
U U	experiment	. 45
Figure 9.4.	GREAT 10 Z channel External blanking during a gradient ec experiment.	ho . 46
10 1D Gra	adient "Shimming" Operations	49
Figure 10.1.	Gradient Echo pulse scheme as used for 1D gradient Shimmi	ng.
	Only the Z shims are matched with this experiment.	. 49
Figure 10.2.	1D Shim Map Resulting from the Shim Mapping Setup Proce dure	}- 51
Figure 10.3.	Display of the Main window used for 1D gradient shimming.	52
Figure 10.4.	Z profile obtained after the two steps of 1D gradient shimmir	וg . 52
Figure 10.5.	1H Spectrum of the doped water sample	. 53
Figure 10.6.	1H Spectrum of the doped water sample	. 54

11 3D Gradient "Shimmimg" Operation on X, Y, Z Gradient

### Coil55

Figure 11.1.	Gradient Echo pulse scheme as used for 3D gradient Shimmir	ng. 55
Figure 11.2.	Shim Map resulting from the Shim Mapping Set Up Procedur	e.
Figure 11.3.	Z Field maps obtained after two 3D gradient shimming itera- tions.	.58
Figure 11.4.	A) Three dimensional image of the water sample	.58
Figure 11.5.	1H proton spectrum with water pre-saturation obtained with a mM	a 2 .59
12 3D Gra	adient "Shimming" Operation on Z Gradient Coil	61
<b>12 3D Gra</b> Figure 12.1.	adient "Shimming" Operation on Z Gradient Coil Gradient Echo pulse scheme as used for 3D RCB gradient Shimming.	<b>61</b> .61
<b>12 3D Gra</b> Figure 12.1. Figure 12.2.	adient "Shimming" Operation on Z Gradient Coil Gradient Echo pulse scheme as used for 3D RCB gradient Shimming. Z Field maps obtained after two 3D gradient shimming itera- tions.	<b>61</b> .61 .63
<b>12 3D Gra</b> Figure 12.1. Figure 12.2. Figure 12.3.	adient "Shimming" Operation on Z Gradient Coil Gradient Echo pulse scheme as used for 3D RCB gradient Shimming. Z Field maps obtained after two 3D gradient shimming itera- tions. A) Three dimensional image of the water sample	<b>61</b> .61 .63 .63

## 14 GREAT 1/10 - GREAT 3/10 Package 67

## Figures
## Tables

1	Introduction	5
2	Starting with the GREAT Gradient Amplifier	7
3	Great Calibration Operations	11
4	GREAT Service Web Pages	15
5	GRADIENT Strength Calibration	21
6	GRADIENT Recovery/Pre-emphasis Test	25
7	GRADIENT Echo Stability Test	31
8	<i>Water Diffusion Coefficient Measurement Using a 1D</i> <i>Gradient Echo Pulse Sequencer 35</i>	
9	Miscellaneous	39
10	1D Gradient "Shimming" Operations	49
11	3D Gradient "Shimmimg" Operation on X, Y, Z Gradie Coil 55	nt
12	3D Gradient "Shimming" Operation on Z Gradient Coi 61	I
13	Frequently Asked Questions (FAQ)	65
14	GREAT 1/10 - GREAT 3/10 Package	67

## Tables

## Notes: