

GREAT 40/60

Gradient Amplifiers User Manual

Version 001



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This unit is not designed for any type of use which is not specifically described in this manual. Such use may be hazardous.

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Hardware description

1.1

Hardware description / Design

The 60A gradient system may be delivered in two different versions. One version for High Resolution and Diffusion NMR experiments in which only one GREAT 60A (W1209612) amplifier and one Master Unit (H9402) is needed. When the system is dedicated to micro imaging experiments, a complete cabinet containing 3 GREAT 60 (W1209612) amplifiers, one Master Unit (H9402) and one B0 Compensation Unit (W1212776) is delivered.

The Master Unit (Figure 1.1 below) contains the interface board between the GCU board located in the AQS rack and the amplifiers. This Unit is in charge of signal routing from the GCU to the amplifiers, setup parameter reading from the amplifier EPROM's and setup parameters writing from the keyboard input to the amplifier EPROM's. All the information through and from the Master Unit is in digital mode.





Figure 1.2. Zoom of the Master Unit upper part





Figure 1.3. Zoom of the Master Unit LED panel.

On the master Unit LED panel (Figure 1.3 above), there are four LED's checking the connections between the Master Unit output and Gradient amplifier input. The LED is green for each connected. For the X, Y, and Z connection a GREAT 1/40 or GREAT 1/60 may be connected whereas the Z_0 LED is on when the Z_0 compensation unit is connected. The yellow LED are blinking while the X, Y, Z or Z_0 channels are pulsing. The ERROR LED's are on only if the Master Unit has found an error status inside his own hardware or if there is a problem with the connected Unit (GREAT 1/40 or GREAT 1/60).

- 1. RS232 cable CCU-Master Unit Serial I/O (BBIS + EEPROM), 9 pin male/ 9 pin female, 6m : (HZ04053).
- 2. Master Unit Reset Button.
- 3. Cable data GCU-Master Unit, 50 pin SCSI, 7m. Shaped pulse (pulse length + pulse intensity in %) : (HZ04360).
- 4. Cable data Master Unit-GREAT 1/40 or 1/60 X channel, 68 pin SCSI, 1m (pulse + pre-emphasis + DC offset) : (HZ10202).
- 5. Cable data Master Unit-GREAT 1/40 or 1/60 Y channel, 68 pin SCSI, 1m (pulse + pre-emphasis + DC offset) : (HZ10202).
- 6. Cable data Master Unit-GREAT 1/40 or 1/60 Z channel, 68 pin SCSI, 1m (pulse + pre-emphasis + DC offset) : (HZ10202).
- Cable data Master Unit-Z₀ compensation Unit, 68 pin SCSI, 1m (Homo spoil from BSMS) : (HZ10202).
- 8. Master Unit ON/OFF Button.
- 9. Cable for PT100 thermocouple connection to measure the gradient coil temperature. The display is done on the LED. There is a temperature safety which cut the gradient pulse at a 50°C temperature. Part of the gradient cable (HZ0969 or W1212172 with the filter) or coming from BCU20 (W1210722) when external safety mode chosen.
- 10. Cable for temperature safety setting via BCU20. The temperature safety is set when the water temperature is higher than a fixed limitation (W1211022).
- 11. LED for Gradient coil temperature display.
- 12. LED Panel showing which channel is available (connected with amplifier ON) or not.
- 13. LED Panel showing Master Unit or Amplifier handling errors.
- 14. LED Panel whose LED's are blinking while pulsing.

The GREAT 60 is a amplifier which is able to deliver at the gradient coil from 0 to 60A currents. The amplifier is built in the manner that it is able to deliver respectively 10A, 20A, 30A, 40A, 50A and 60A with the highest dynamic available on a 16 bit DAC. The amplifier input comes from the Master Unit output in full digital mode. The GREAT amplifier contains the amplification steps and the current regu-



lation, the pre-emphasis circuits, the DC offset regulation and a correction loop for thermal DC offset level regulation. The front panel of a GREAT 1/60 is shown in Figure 1.4 below. The GREAT 1/40 amplifier is the lower current version of the GREAT 1/60. The electronic and mechanic characteristics of the GREAT 1/40 are the same as on the GREAT 1/60. There only difference is the performance of the power supply.





Figure 1.5. Zoom of the Great 1/60 (W1209612) upper part



- 15. TCU 0/5V signal used for external blanking (Burndy BNC connector part of HZ0969 Gradient cable). The BNC is connected on the gradient GATE connector and the birndy on the 28 pins Back panel BP1. (connection b for X channel nmrword 0 bit 32 / connection c for Y channel nmrword 0 bit 33 / connection d for Z channel nmrword 0 bit 34). For more information on the external blanking see section 1.4.
- 16. Gradient amplifier (+) output (may be Z⁺, Y⁺ or X⁺) depending on chosen gradient connector.
- 17. Ground.
- 18. Gradient amplifier (-) output (may be Z⁻, Y⁻ or X⁻⁾ depending on chosen gradient connector.
- Cable data from Master Unit (output 4, 5 or 6) depending on channel X, Y or Z choice. 68 pin SCSI, 1m (pulse + pre-emphasis + DC offset in digital mode) : (HZ10202).
- 20. Monitor output for pulse length/form control on oscilloscope (BNC connector). Maximum 10V output at full power e.g. if a current of 60A is sent to the gradient coil.
- 21. GREAT 1/60 amplifier ON/OFF Button.

22. LED showing the current pulsed gradient strength between -60 to +60A at full power on the 60A step.

Two other parts complete the gradient accessory : the gradient cable (HZ0969) and the filter box put in between the GREAT 1/60 and the gradient coil connector on the probe. The gradient cable and the filter box are shown in Figure 1.6 below.

Figure 1.6. Picture of the gradient cable used with the GREAT 1/40 and GREAT 1/60 amplifiers. The filter box shown on the picture is a part of the gradient cable accessory (HZ0969)









GREAT 1/60 High Resolution Installation

1.2

The GREAT 1/60 amplifier may be used by customers who are interested in self diffusion of molecules by diffusion coefficient measurements or DOSY experiments. It may be interesting in some cases to have Higher Field gradient strength (e.g. up to 100 G/cm) which may not be reached with the current 10A GREAT or 7A GAB gradient amplifier. In this case the GREAT 1/40 or GREAT 1/60 may be used on the 20 or 30A step as far as current 5mm probehead Z Gradient coils may not be damaged with currents lower than 25A. For such kind of High Resolution experiments we need 1 Master Unit (H9402), one gradient amplifier GREAT 1/60 (W1209612) or GREAT 1/40 (W1211690), 1 gradient cable (HZ0969) with its filter box, 1 RS232 cable (HZ04053), one 68 pin SCSI cable to connect the Master Unit Z output to the gradient amplifier input (HZ10202), 1 Burndy/BNC cable to connect the gradient amplifier GATE to the spectrometer back panel and 1 dummy load connector (HZ03940) for the PTS 100 connector on the GREAT front panel. The system looks like shown in Figure 1.7 below. The Master Unit and GREAT 1/40 or 1/60 system may be located inside the console if there is some room like in Two bay systems, or on the top of the console if the customer has a One bay system. There is no major gradient amplifier stability difference if the amplifier is located inside or outside the console if there is a thermal regulation in the spectrometer room.



Figure 1.7. High Resolution GREAT 1/60 Installation scheme on AV systems

GREAT 1/60 Diffusion / Micro Imaging Installation

The GREAT 60 Micro Imaging system is an accessory including a OneBay console inside of which you may find one Master Unit (H9402), three GREAT 1/40 or 1/60 Gradient amplifiers (W1209612) one for each X, Y and Z axis, an optional H0 compensation Unit (W1212776), the three axis gradient cable including the filter box (W1212172) and of course the BCU 20 temperature control unit (W1210722). Such a system combined with DIFF 30, DIFF 60 or Micro Imaging probeheads allows Diffusion experiments and NMR Image recording. The optional H0 compensation unit is necessary in some Imaging experiments where B0 field drift occurs. The BCU 20 temperature cooling unit is used to cool the gradient coil. The BCU 20 system may be driven directly by measuring the gradient coil temperature. This is achieved when the PT100 cable of the gradient cable is plugged on input 9 of the Master Unit. Otherwise it is possible to control the gradient coil temperature through the temperature changes of the BCU 20 cooling bath. This is achieved when the PT100 cable of the BCU 20 is plugged on input 10 of the Master Unit. You have to choose one of the two connections. In absence of a plug on one of the two inputs 9 or 10, it is not possible to start the gradient handling software Setpre. The temperature checking is necessary (when working in Diffusion or Micro Imaging Mode at gradient pulse currents higher than 30A) in order to avoid gradient coil burning when the gradient intensity becomes too high. The BCU 20 is equipped with an Eurotherm system driven by the XWINNMR software. The RS232 connection of the BCU 20 has to be plugged on one of the free tty's on the spectrometer console. The connection is activated by the cftg order. The BCU 20 Eurotherm is driven by the edtg software of XWINNMR. The edtg order opens a temperature control window in the same manner as edte does. You may found the same parameters and the same functions as in a classical edte window.

The following scheme (see Figure 1.8) shows how the system works in Micro Imaging Mode. Note that the High Resolution case is a particular case of this scheme (in High Resolution and Diffusion only the Z channel is used).



Figure 1.8. Schematic way of working of the 40/60A gradient system in Micro Imaging Mode

In the picture of Figure 1.9 below is shown a complete so called "HMIMAG" Diffusion / Micro Imaging system in front and back view. On this light all the connections of a complete (including H0 compensation Unit) "HMIMAG" system are shown.





The following picture (Figure 1.10 below) shows the front panel of the optional H0 compensation unit. H0 compensation is necessary in some Micro Imaging experiments where B0 field drift is observed. The H0 compensation unit generates a small compensation pulse (~200mA) sent on the homospoil coil of the shim system.





- 23. To BSMS Homospoil.
- 24. To Master Unit 7 connector.
- 25. Monitor output for pulse length/form control on oscilloscope (BNC connector).

Hardware description



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External TCU blanking

1.4

The gradient pulses are generated by the GCU as numerical data and sent to the GREAT 60 amplifier through the Master Unit which makes the routing to the selected channel X, Y or Z. The pulse amplitude, the pulse shape and the pulse duration comes from the GCU. When no numerical data is sent from the GCU, there should be no current at the GREAT 60 amplifier output. Nevertheless some residual DC current exists at the GREAT 60 amplifier output as a consequence of thermal stability / tolerance of some electronic components. Thus it is necessary in order to avoid spectrometer stability perturbation to blank the amplifier output. This is done by electronic components (MOS) which are enabled or disabled through an external TCU blanking. The 5V TCU blanking level isolates the amplifier from the probe whereas the 0V TCU blanking level put the GREAT 60 amplification channel on line with the probe. Therefore the amplifier output is enabled while pulsing and disabled when no gradient pulse is sent to the probe.

In practice, we use in pulse programs the UNBLKGRAD, BLKGRAD, UNBLK-GRAMP, and BLKGRAMP orders for this purpose. The UNBLKGRAD / UNBLK-GRAMP orders enable pulsing whereas the BLKGRAD / BLKGRAMP disable pulsing and isolate the gradient coil from the GREAT amplifier. The BLKGRAMP and the UNBLKGRAMP are orders relative to X, Y and Z TCU external blanking whereas the BLK GRAD and the UNBLKGRAD orders add the Lock Hold external orders to the gradient amplifier blanking orders. The definition of the gradient blanking orders is the following :

- BLKGRAMP / UNBLKGRAMP: X, Y, Z Gradient blanking/unblanking nmrword 0 bit 32 for X channel, nmrword 0 bit 33 for Y channel and nmrword 0 bit 34 for Z channel.
- BLKGRAD / UNBLKGRAD: Combines Gradient blanking/unblanking and Lock Hold abilities (nmrword 3 bit 0).

The TCU nmrwords 0 bit 32, 33 and 34 are available on the AVANCE back panel 28 pin connector BP1 respectively at position b, c and d as shown in Figure 1.11.

TCU nmrwords 0 bit 32, 33 and 34 corresponding to X, Y and Z channel gradient blanking as found on the 28 pin back panel connector BP1

Figure 1.12. Burndy connector



- nmrword 0 bit 32 (BLK GRAD X)
- nmrword 0 bit 33 (BLK GRAD Y)
- nmrword 0 bit 34 (BLK GRAD Z)

HMIMAG Electronic characteristics

In this part of the manual are summarized some of the Electronic characteristics of the devices included in a "HMIMAG" Diffusion/Micro-Imaging system. You may also found some caution in handling with such a high gradient system.

Electronic characteristics :

GREAT Master Unit :

- Digital gradient pulse amplitude input from Gradient Control Unit (GCU). Use of gradient shape pulses with a maximal resolution of 6μs.
- Optically decoupled input.
- Digital gradient pulse amplitude output to gradient amplifiers and H0 compensation unit.
- Pulse duration control.
- Gradient coil temperature (directly measured at the coil vicinity or through temperature change in BCU 20 cooling bath).

GREAT 40/ GREAT 60 amplifiers :

- GCU real time driven gradient shape through 16 bit DAC.
- 40A / 60A maximum current in 10A steps for optimal digital resolution.
- Maximum voltage ± 100V.
- Pulse rise Time: 50 to 150µs depending on coil impedance.
- Duty cycle: 25% at 40A / 60A.
- Digital controlled pre-emphasis with 4 time constants.
- Digital controlled amplifier DC offset.
- Time average amplifier DC offset correction against temperature.
- TCU amplifier output blanking.
- Software controlled Impedance Matching, offset correction and pre-emphasis matching.

GREAT H0 compensation unit :

- Maximum current: 200 mA.
- Maximum voltage: ±110V.
- Pulse Rise Time: < 500ms on typical load (150 Ω , 100 mH).
- Dynamic range: ±200 mA by 6µA steps.
- Stability : $\pm 100 \mu$ V for 1 Minute, $\pm 100 \mu$ V for 1 hour (100 load), $\pm 500 \mu$ V for Δ T = 5K.

Caution: Please be very careful in the choice of the amplifier output current step. The software default value may be 60A. Short duty cycles (< 25%) and non adapted current levels may damage the filter and/or the gradient coil. This is especially true for High Resolution uses (Maximum current allowed : 20A).



GREAT ZO P/N: W1210128 ECL 00			
SPECIFICATIONS			
Power requipments	230V +/-10% 50Hz		
Power consumption	300W		
Weight	10kg		
Dimensions	445 / 484mm width (cabinet/front panel) 89mm height (5U unit) 460mm depth		
Software control	XWINNMR ("SETPRE" controlled)		
DIGITAL INPUTS AND OUTPUTS SIGNALS			
Gradient shape values	16 bits DAC controlled, GCU real time drived		
Preemphasis	Setting by RS232 0.2ms, 2ms, 20ms, 200ms ranges		
Current offset	Setting by RS232		
Analog and digital reset	Setting by RS232		
Amplifier ON/OFF	Setting by RS232		
Securities faults	Read by RS232 >I Temp Power supplies		
MAIN SPECIFICATIONS			
Maximum current	200mA		
Maximum voltage	+/-110V		
Rise time	<500µs on typical Load (150 Ohm ; 100mH)		
Stability	+/-100 μ V for 1 minute, +/-200 μ V for 1 hour (100 Ω load) +/- 500 μ V for Δ t = 5°C		
Dynamic range	+/-200mA by 6µA step		
Monitor output	10V for 200mA (load 1Mohm/22pF)		

Table 1.1. Specifications of the Great ZO

Hardware description



GREAT 60 Software commands

2.1

View of the interactive "Setpre" amplifier control module

The GREAT 1/60 amplifier is connected to the CCU through a RS232 Serial I/O cable. Commonly the connection bite is tty06. The "cf" command recognizes the presence of the 60A gradient amplifier as GREAT 3/60 as far as the software checks the presence of the Master Unit. In the "uxnmr.info" file the GREAT 1/60 is recognized as GREAT 3/60 amplifier. The XWINNMR software allows to handle some parameters of the amplifier through the "Setpre" order. When "Setpre" is entered on the keyboard, the widget shown in Figure 2.1 below is displayed on the screen.

ietpr2:)st	EA 351 ON, Gradient coi	l temperature: 0	degrees		•
<u>File</u> <u>E</u> dit	Channel Option	and an addition of		1	Help
20 A 🗧 🗆	Auto Offset	ш 🗇	2.30	▼ 1.00	*
Slow Base Preemp. Z	Time [ms]		17.988	♥ 0.100	*
20 ms 🔫	Gain [%]		1.76	▼ 1.00	*
Mid Base Preemp. Z	Time [ms]	(10)	0.2994	▼ 0.0100	
2 ms 🔫	Gain [%]		10.94	▼ 1.00	*
Fast Base Preemp. Z	Time [ms]		0.04991	▼ 0.00100	
0.2 ms	Gain [%]		6.99	▼ 1.00	
Impedance Load	Resistors [%]		87.1	▼ 0.5	
High 🥑	Capacitors [%]	<u> </u>	15.7	♥ 0.5	
Store	Recall	change	Undo	Clea	ur
(13)	(14)	15) (16	(17	

Figure 2.1. Screen display of the "Setpre" widget

The following functionalities are implemented in the "Setpre" software :

1. File : Read / Write pre-emphasis value files from disk under :

Scroll down menu/

<xwinnmr home>/exp/stan/nmr/parx/preemp/<probe number>/<name>

2. Edition of some parameters used for DOSY or Micro Imaging :

Scroll down menu



3. Channel selection when the gradient system has more then one GREAT 1/60 amplifier (default value: channel Z) :

Scroll down menu

4. Options menu : Handling of some GREAT functionalities e.g. Enable/Disable pre-emphasis circuit's :

Scroll down menu

5. GREAT 60 Amplifier step selection (10, 20, 30, 40, 50, or 60A) :

Scroll down menu

Gradient constant values set from user between 0 to 100 % sent to the amplifier a maximal current of 10A when 10A step is selected and 60A when 60A step is selected.



WARNING : For High Resolution experiments set the amplifier calibration step to a maximum of 20A. All steps higher then 20A may damage the gradient coil.

6. GREAT 60 Amplifier auto offset adjustment done one the amplifier connected to the channel selected with the Scroll down menu 3 :

Toggle button

7. Offset adjustment slider and actual value display :

Slider

8. Pre-emphasis Time base (0.2, 2, 20 or 200ms) :

Scroll down menu

9. Impedance High / Low selection :

Toggle button

 Pre-emphasis Time constant and Gain selection for Slow, Middle and Fast Time base. The Time constant changes between 0 and the chosen Time base. The Gain value changes between -100 and +100% :

Slider

11. Amplifier regulation parameter matching :

Slider

12. Sensitivity buttons :

Toggle button

13. Storage of the displayed values on the amplifier EEPROM :

Button

14. Recall :

Button

15. Exchange :

Button

16. Undo allows to come back to the preceding values stored on the amplifier EEPROM :

Button

17. Clear : This Button set all values to 0 :

Button

Handling a GREAT 60 amplifier with "Setpre"

2.2

Before explaining how to handle the gradient amplifiers with the "Setpre" software, it is necessary to explain the way of working of the "Setpre" software. The Toggle buttons and the Scroll down menus are set on the "Setpre" interface. The Master Unit checks the values displayed on the interface every xx seconds and put the selection bytes accordingly to the values set by the user. The sliders are real time interactive commands as far as the values shown on the screen are transferred to the Master Unit as numerical data. This numerical data is then transferred to the DAC's of the GREAT 60 amplifiers. The pre-emphasis, the R and C loop parameters and the DC offset changes are managed in this way. With such kind of philosophy it is possible to adjust this parameters on the real time scale. The displayed values shown on the screen are only written on the GREAT 60 amplifier EEPROM by pushing the store button or by leaving the "Setpre" software. That is the reason : if you change some values without storage on the amplifier EEPROM, the undo button allows to come back to the last data stored on the EEPROM.



Storage of the **"Setpre**" screen display on the GREAT 60 amplifier EEPROM. The operation is also done when leaving the **"Setpre**" interface.

When you have changed some values on the "**Setpre**" screen display, a click on the undo button loads on the screen display the previous EEPROM stored values.



Undo

Call the last "**Setpre**" values stored on the GREAT 60 amplifier EEPROM.



Store the "Setpre" screen display values on the GREAT 60 amplifier EEPROM and read out the EEPROM values which are now displayed.



Put all "Setpre" screen display values to 0.



Impedance High / Low Toggle button. Has to be set on High Impedance. This menu is only available if you enable the Impedance & loop editing facility in the **option menu**.

Resistors [%]		87.1	V	0.5	
Capacitors [%]		15.7		0.5	

Figure 2.2. Resistors and Capacitors adjust panel

GREAT 60 amplifier regulation RC loop. The value of R and C may be adjusted between 10 and 100%. The values of this parameters are strongly depending on the inductance (L) of the gradient coil connected on the gradient amplifier. This RC regulation loop parameters have a direct effect on the pulse rise time/drop time and the symmetry of the Gradient pulse. Bad setting of one or both of those parameters may have the same effect as bad pre-emphasis setting on an NMR experiment using gradient pulsing. (see section <u>"Amplifier Control Loop Adjustment (R & C in "Setpre")" on page 23</u>).

Figure 2.3. Slow Base adjust panel

Slow Base Preemp. Z	Time (ms)	17.988	0.100 🛦
20 ms 🛥	Gain [%]	1.76	1.00 🔺

For the Slow, Middle and Fast pre-emphasis Time Base it is possible to choose between four time ranges (e.g. 0.2, 2, 20 and 200ms). Each Pre-emphasis circuit adds an exponential decay to the gradient shaped pulse whose amplitude depends on the Gain parameter and the decay on the time base. Of course in terms of analogical currents, the current really added to the gradient pulse depends on the choice of the R and C values put on the board. These last values are normally chosen in order to compensate Eddy currents on HR gradient coils, DIFF 30, DIFF 60 and Micro Imaging coils. Of course the values may be different between a Z HR gradient coil and a Micro Imaging coil. The way to set the pre-emphasis values in order to compensate as properly as possible the Eddy currents is shown in section 3.x. If DC is chosen, the pre-emphasis circuits are bypassed.

Figure 2.4. Currents and offset adjust panel



Three functions are shown in this picture:

• At the left, it is the amplifier step corresponding to the maximal current available at coil input when the gradient XWINNMR constant (cnst or gp) is set to 100%. In this shown example for cnst21 = 100% or gpz1 = 100%, we sent 20A on the gradient coil. Six steps are available: 10, 20, 30, 40, 50 and 60A.



Never forget that High Resolution gradient coils may be damaged by currents higher then 20A. Thus in High Resolution mode set the step button to 20A.

 In the middle there is the auto offset command. This command checks automatically the GREAT 60 output level in the absence of pulsing and compensate the DC level which may be exist at the amplifier output.



This order does only properly work if it is executed half an hour after turning the GREAT 60 amplifier ON. The amplifier must have reached his working temperature. An error message is displayed if the auto offset is started in the early minutes after turning the amplifier on.

 Sometimes it is necessary to optimize the GREAT 60 amplifier offset by hand. The slider allows to do that.

Preemphasis bypas:	s
BO compensation	
🛙 Bļanking	
Reset protection	
□ <u>O</u> ffset adjustment	
☑ Impedance&loop edit	iting

Option Scroll down menu :

•Pre-emphasis bypass: This mode allows to work without pre-emphasis. This mode may be used on some probes with gradient coils which generate small Eddy currents. This mode may be used when non square gradient pulses are sent to the gradient coil.

•B0 compensation: The B0 compensation is only used for Micro Imaging experiments.

•It is possible in the **"Setpre"** option window to turn ON/ OFF the gradient amplifier output. When the Blanking button is selected, the gradient output is on line with the

coil and you are able to adjust for example the DC offset in this mode. When the blanking button is not selected, the gradient amplifier output is isolated from the probe. Nevertheless, the TCU blanking/unblanking order found in pulse programs has priority.

- The Reset protection order makes a reset of the Master Unit in the same manner as the reset button of the Master Unit.
- In this menu the Offset adjustment order makes a DC auto offset correction on all channels recognized by the Master Unit. If more then one GREAT amplifier is connected, the DC auto offset is done on each amplifier.
- The Impedance and loop Editing order allows the display of the "Setpre" widget relative to Regulation loop parameter setting.

Channel Scroll down menu :

- ♀ ∑ preemphasis
 ♀ Y preemphasis
 ♥ Z preemphasis
 ◊ X B0 compensation
- ♦ Y B0 compensation
- ◊ Z B0 compensation

As far as the channels are independently managed by the Master Unit, the screen display menu shows only the parameters relative to the channel selected in the menu. The display shows, the parameters currently set on the chosen channel. If the store function was previously done, the parameters displayed on the screen are the same as the parameters written of the EEPROM of the corresponding GREAT amplifier. Otherwise not.

Grad. calib. const. [Hz/cm]
Grad. calib. const. [Gimm]
Gradient scaling factor
Rate to measure temperatur
Rate to check error status

Clear all preemphasis values

Edit Scroll down menu :

This window contains some dialog boxes where some parameters relative to gradient handling may be fixed.

•It is possible to put in the Gradient calibration constant in G/mm depending on the maximal current used on the GREAT amplifier and depending on the used coil. This parameter can be measured by means of an NMR experiment (see section 4.2). The Gradient calibration constant may be given in G/mm or Hz/cm. They are related by the following expression : $\Delta G = \Delta v (kHz) / (4.258 \text{ x R})$ where R is the sample height in cm. This parameter is used for the DOSY experiment.

ramp calculation in the DOSY experiment.

- The gradient scaling factor (value between 0 and 1) is used when gradient experiments use simultaneous X, Y and Z gradients for which the G_X, G_Y and G_Z field gradients should be identical. For such purpose it is necessary to use the Gradient scaling factor on each axis.
- The two following parameters allow to put in the time interval between the coil temperature measurements and the time between the **"Setpre"** software use to check up status errors in the Master Unit.
- The Clear all preamphasis values has the same effect than the Clear button but the operation is done simultaneously on all active channels.

File Scroll down menu :

The orders in this menu allow to Read / Write and Convert pre-emphasis setup parameter files. The files are stored on the disk in the following directory:

/<xwinnmr home>/exp/stan/nmr/parx/preemp/<probe number>/default or <name>. The <probe number> should be the same number as defined by the XWINNMR edhead setup order (see XWINNMR User's Manual).

Installation Adjustments

Amplifier Control Loop Adjustment (R & C in "Setpre")

3.1

In this experiment the GREAT 1/60 amplifier output (channel X, Y and/or Z) is connected to the gradient coil of the probe and the GREAT 60 amplifier input is connected to connector 4, 5, and/or 6 of the Master Unit. Open the "Setpre" screen display (see <u>"Screen display of the "Setpre" widget." on page 17</u>) and enable the Impedance & loop Editing function of the Option Scroll down menu. For *High Resolution probes, the gradient maximal output power step selection has to be put on 20A*, whereas for Diffusion and Micro Imaging probes you may work at every step you wish. The GREAT 40 or GREAT 60 amplifier monitor output 20 (see <u>"Zoom of the Master Unit upper part" on page 5</u>) is connected either to an oscilloscope or on the spectrometer digitizer (SADC, HADC or FADC) inputs (channel A or channel B). Use the following pulse program to adjust the amplifier regulation resistance R and capacity C with the sliders 11 of the "Setpre" widget.

Table 3.1.	Gradient amplifier	r electronic puls	e test program

grad test el					
;avance version (00	0/05/18)	"d27=d29"			
Gradient amplifier Electronic pulse test					
; PHL (BBIO France)	-	1 ze			
;Variable d29 delay	y in between pulses.	d11 UNBLKGRAD			
	-	2 d1			
<pre>#include<avance.ind< pre=""></avance.ind<></pre>	c1>	DE1 DE2 DERX DEADC DEPA DE3			
<pre>#include<grad.incl:< pre=""></grad.incl:<></pre>	>	d27 DWELL_GEN			
		d27			
define delay rde1		p16:gp1			
define delay rde2		d29			
define delay rderx		p16:gp2			
define delay rdeado	c	aq			
define delay rdepa		rcyc=2			
		400m wr #0			
"rde1=de-de1"		d11 BLKGRAD			
"rde2=de-de2"		exit			
"rderx=de-derx"					
"rdeadc=de-deadc"		ph30=0			
"rdepa=de-depa"		ph31=0			
#define DE1 ((del rdel adc ph31 syrec)	;use gradient program GP1 = GP2 = RECT.1			
#define DE2 ((de2 rde2 ph30):f1	;gp1 z,y or x = 100% gp2 x,y or x = 0%			
#define DERX ((derx rderx RGP_RX_ON)	;NS=1			
#define DEADC ((deadc rdeadc RGP_ADC_ON)	;DS=0			
#define DEPA ((depa rdepa RGP_PA_ON)	;Acquisition mode=qf			
#define DE3 ((de)				

In this experiment d1 = 2s, d29 = 20ms and number of scans NS = 16. The gradient pulse length p16 = 5ms, the gradient strength $gp_{x,y,z} = 100\%$ and $gp_{x,y,z} = 0\%$, the gradient shape is a square (with a maximum number of points of 1248).

BIOSPIN

The time domain TD is 32 K, the spectral width is equal to the maximal allowed by the digitizer (e.g. 150 kHz for an SADC) to give the highest time domain resolution available (e.g. 3.3μ s in the case of an SADC). Of course the acquisition mode **AQMOD is set to qsim** and digitizer mode **DIGMOD is set to analog**. Start the pulse program is **gs** mode and match the amplifier regulation loop parameters R and C as good as possible to obtain a square gradient pulse with clean edges as shown in figure 3.1.below . R and C may be changed in the **"Setpre"** window with slider 11.

AV spectrometer screen display of the GREAT 60 amplifier as measured with the previous experiment at the monitor output 20, when using a 5mm TXI HR probe with Z gradient coil and the GREAT 60 on the 20A step. Left : R and C are not properly adjusted ; Right : Correct R and C matching.





Important : This experiment has to be done without pre-emphasis e.g. chose preemphasis bypass in the option scroll down menu and set the slow, mid and fast time base on DC. Do never forget that the amplifier regulation loop parameters may have the same effect as pre-emphasis sets on the NMR signal as far as both are exponential correction loops which may of course interact.

In the example shown in figure 3.1 above (e.g. for a **5 mm TXI probe equipped** with a **Z gradient coil**) the value found for **R is 87.8%** and the value found for **C** is **16.0%**. For Micro Imaging/Diff 30 probeheads, **R is near 75 to 78%** and **C is near 12 to 15%** depending on the wanted rise time.

Amplifier DC offset correction

3.2

The amplifier DC Offset correction may be done either by using the **automatic** software Offset adjustment of the "**Setpre**" window (all connected channels when using the **Offset adjustment** order of the Option Scroll Down Menu 4 or only the displayed channel when using the **Auto Offset Button** 6 or by using the **manual Software Offset** adjustment using the Offset Slider 7.



Important : Note that handling of preemphasis may change the GREAT 1/40 or 1/60 output DC level. Thus it is recommended to check each X, Y and Z channel DC Offset prior and after the preemphasis adjustment if the preemphasis circuits are bypassed then only one DC Offset correction step is needed.

Amplifier DC offset correction in presence of a Lock Signal

3.2.1

On production site, the amplifier DC Offset is matched as near as possible to 0 (on the μ V scale) at the 10A amplifier step with the built in potentiometers on the analogical side. Nevertheless, the room/console temperature differences may change slightly the DC Offset value. It is clear for conception reasons, that it is not possible to have the same Offset at the 10A step then at the 60A step. Thus it might be necessary to make a fine DC Offset adjustment by means of the software numerically.

For High Resolution probes or some Diffusion (DIFF 30) probes where the lock circuits are built in, we can use the lock level for DC Offset adjustment. Use the **blanking** order of the Option Scroll Down Menu in the **"Setpre"** window and enable the box. The GREAT 1/40 or 1/60 amplifier output is online with the gradient coil of the probe. By changing by hand the offset value with the Slider 7, you may see how the lock level drops or goes ahead. Note the lock level before blanking the amplifier and adjust either by hand or by using one of the Auto Offset adjustment orders. It is recommended to do a fine amplifier DC Offset adjustment by hand with Offset Slider 7 when you wish to record spectra needing positive and negative gradient pulses (see Section <u>"Gradient amplifier stability tests" on page 34</u>). Note and store the obtained value.

Amplifier DC offset correction without a Lock Signal

3.2.2

When no lock circuit is available on the probe (e.g. on some Diffusion and /Micro imaging probes) it is possible to do the automatic offset adjustment by operating in the same way as in section 3.2.1 with the difference that the DC level correction effect is no more visible. Only the change of the offset value on the **"Setpre"** Slider 7 widget shows the DC level correction effect. Nevertheless it is possible to do a fine DC level correction in the absence of lock signal. Put the amplifier in blanking mode with the **blanking order** of the Option Scroll Down Menu of **"Setpre"**. Start a **gs** ¹H single pulse spectrum recording on the 1% water sample (1% H2O + 99% D2O + 0.1 mg/ml GdCl3) using standard 1D parameters. In the acquisition window toggle from time domain to frequency domain (as for shimming) and observe the water peak height. As far as the GREAT 1/40 or 1/60 DC level generates a small field gradient in the gradient coil of the probe, this DC level acts on the spectrum as an additional shim coil. Thus optimize the water peak height/ width to obtain the biggest peak by handling the Offset Slider 7. Note and store the obtained value.

Pre-emphasis adjustment

The pre-emphasis circuits are built in the GREAT 1/40 or GREAT 1/60 amplifiers to compensate the Eddy currents generated in the gradient coil while pulsing. In presence of Eddy currents, an NMR signal may be disturbed even canceled. On newer gradient coils built in High Resolution, DIFF 30 and DIFF 60 probes the Eddy currents are very small but still exists. To observe the phenomenon and to adjust pre-emphasis time bases (Capacities) and Gain (Resistors), we observe a set of 8 FID's recorded after sending a gradient pulse to the probe. After the gradient pulse a 90° RF pulse is sent on the RF coils and the spectrum is observed.

In each of the 8 FID's the time between the end of the gradient pulse and the 90° RF pulse is different. The pulse program used for this purpose is the following :



Figure 3.2. GRD Pulse delay diagram



Table 3.2. Pulse program for preemphasis adjustment



The pre-emphasis adjustment is done in **gs** mode on the 1% water sample (1% H2O + 99% D2O + 0.1mg/ml GdCl3). Use for this experiment standard 1D parameters normally used for ¹H spectra recording. Set the acquisition time to 500ms, a repetition time of 2s, an RF pulse flip angle of 5 to 10° to decrease T_1 relaxation effects while observing the FID's. Put the spectrum Offset O1 approximately 2-3kHz away from the NMR peak resonance frequency. Use a gradient square pulse of about 30 to 50ms length. The gradient shape names GPNAM1 = RECT.1

or every other square pulse shape (according to the fact that the GCU resolution is 4μ s). On High Resolution probes, use a **gradient strength of 70% on the 20A step** (G+ " 70 G/cm). On DIFF 30 / DIFF 60 probes, use a **gradient strength of 50% on the 40A step** (G+ " 600G/cm / " 1200G/cm). Chose VDLIST = preemp (e.g. 0.1ms, 0.3ms, 1ms, 3ms, 10ms, 30ms, 100ms, 500ms).

The pre-emphasis are adjusted by using the Time Base Scroll Down Menu for the Slow, Mid and Fast Base circuits. The Time Base is changed by the **Time Sliders** 10 (variation of the circuit capacity) whereas the gain is changed by the **Gain Sliders** 10 (variation of the circuit resistance).

After starting the **gs** command for spectra recording, put the Slow Time Base on 200ms, the Mid. Time Base on 20ms and the Fast Time Base on 2ms. Put the Time Sliders in the middle of the scale e.g. 100ms, 10ms and 1ms respectively for the Slow, Mid and Fast Time Bases. Put all the Gain Sliders to 0. On the screen display FID nr. 8 and 7 are the reference FID's whose amplitude/phase are near the FID amplitude/phase of the water sample recorded in such conditions in absence of gradient pulsing.

Now adjust the gain of **the longest time constant** to optimize the decay of FID number 6. Meanwhile observe FID number 5. If FID nr. 6 reaches his optimum amplitude before FID nr. 5, then proceed to adjust the gain until FID nr. 5 is optimal. Reduce the time constant until FID nr. 6 is undistorted and iterate the procedure until FID nr. 6 and FID nr. 5 are similar to the reference FID's. If on the other hand, FID 5 becomes undistorted before FID nr. 6, then increase the time constant to optimize FID nr. 6 and reduce the gain to improve FID nr. 5. Iterate until both FID's are optimal.

Repeat this procedure for FID nr. 4 and FID nr. 3 using **the middle time constant** and then repeat this procedure for FID nr. 2 and FID nr. 1 using **the slow time constant** until all the four FID's looks like the reference FID's.

It should be possible that the adjustment of the slow and middle time constants have a distortion and/or an effect on the amplitude of other FID's. If it is the case iterate the whole procedure again in order to obtain 8 FID's whose amplitude/ phase are as near as possible (see <u>"Pre-emphasis adjustment" on page 28</u>).

Pre-emphasis adjustment : Experience after Eddy current compensation. The variable delay list was : 0.1, 0.3, 1, 3, 10, 30, 100 and 500ms on an HR probe. The GREAT 1/60 is used on the 20A step. GRD pulse length = 30ms at ~70G/cm.

Installation Adjustments







Installation Tests

Gradient Recovery test

In this experiment we check if the pre-emphasis adjustment setup is correct for NMR experiments using gradient square pulses. We use a pulse sequence in which a gradient square pulse is sent to the probe prior to the RF pulse and the acquisition. The delay between the end of the gradient square pulse and the beginning of the RF pulse (e.g. the gradient recovery delay) changes during the experiment (see pulse program below). We check the NMR water peak amplitude/ phase variation with the recovery delay changes. For this purpose, run the **preempgp2** pulse program in **paropt** mode on the 1% water sample (1% H2O + 99% D2O + 0.1mg/ml GdCl3).

Table 4.1.	Pulse	program	for	preem	ohasis
	1 0100	program	101	proonin	110010

;preempgp2	ph1=0
;avance-version (00/02/07)	ph31=0
;pulseprogram for preemphasis adjustment	
	<pre>;pl1 : f1 channel - power level for pulse (default)</pre>
<pre>#include <avance.incl></avance.incl></pre>	;p1 : f1 channel - 90 degree high power pulse
<pre>#include <grad.incl></grad.incl></pre>	;p16: homospoil/gradient pulse
	:d1 : relaxation delay: 1-5 * T1
1 ze	:d11: delay for disk I/O [30 msec]
5011 UNBLKGRAD	:d16: delay for homospoil/gradient recovery
2 d1	use gradient : gn 1
2 di	g_{μ}
210.921	;;iu: pieempgp2;v 1.0 2000/05/08 11:40:50 eng Exp ;
d16	
pl phl	
go=2 ph31	
d11 BLKGRAD	
wr #0	
exit	

In this experiment standard proton NMR parameters are used : the relaxation delay d1 = 2-3s, the RF ¹H pulse has a flip angle of 90°, the number of scans = 1, the number of data points = 16K and the spectral width SW ~10ppm. To define the gradient pulse, set the GPNAM1 parameter to RECT.1 or all other rectangular pulses shape (with the condition that the pulse length / number of points in the shape is greater than 4µs). The gradient pulse length is set to 5ms and the gradient strength gp1x,y,z to 50% on the 20A step when working with a HR probe, 25% on the 40A step when working with a DIFF 30/60 probe and a GREAT 40 amplifier or 12.5% on the 60A step when working with a DIFF 30/60 probe and a GREAT 60 amplifier. The gradient strength becomes 50G/cm on HR probes and 450 G/cm on a DIFF 30/60 probe. Run the experiment in paropt mode using the paroptlog automation program. Answer the questions as follows :

Enter parameter to modify : d16

Enter initial parameter value : 0.0001

Enter initial increment : 0.0001

- Enter final parameter value : 1
- Enter # of experiments : 100

If the Eddy currents are properly compensated, the differences between the initial water peak amplitude and phase (e.g. the ¹H spectrum is recorded 100µs after the 5ms square gradient pulse) and the final water peak amplitude and phase (e.g. the ¹H spectrum is recorded 1s after the 5ms square gradient pulse) should be minimal. The resulting spectra obtained after good pre-emphasis values adjustment are shown in figure 4.1 below :

Gradient Recovery test recorded with a 5ms square gradient pulse sent on the gradient coil of a 5mm TXI Z Gradient probe. The GREAT 60 is used on the 20A step. Gradient strength = 50% e.g. 50 G/cm. The GREAT 60 amplifier is unblanked/blanked respectively at the beginning and the end of the pulse sequence and the pre-emphasis are adjusted in the previously explained manner. The recovery delay changes in logarithmic steps starting at 100µs up to 1s.



Gradient strength calibration

The gradient strength is calibrated by measuring the profile of a phantom of known spatial dimensions in the presence of a gradient and noting the spectral width of the profile. In practice a 5mm (inner tube diameter = 0.42mm) doped water sample is adequate for X and Y profiles. The spectral width of the obtained profile gives at a given gradient pulse strength $g_{X'Y'Z}$ in A, the field gradient $G_{X'Y'Z} = g_{X'Y'Z} \times d_{X'Y'Z}$. The relationship between the measured profile spectral width and the gradient strength is given by :

 $g_{x,y,z} = \Delta v_{x,y,z} / (d_{x,y,z} x 4.258) \text{ G.cm}^{-1}$ [4.2.1] where Δv is given in kHz and d in cm.

For the Z gradient strength profile a 2.5mm spherical insert filled with 50μ l water may be used as well as a Shigemi Tube containing 2mm water height. The insert or the water sample must be put in the middle of the RF coil. Use the sample tube depth gauge. The accurate position of the insert is reached when the gradient profile is perfectly centered in the spectral window. The limitation of the gradient profile measurement method is the spectral width covered with the spectrometer digitizer (150kHz with SADC digitizer and 1MHz with HADC+ digitizer).



4.2

If D_2O is missed for practical reasons it is possible to work without lock signal with the lock sweep switched off.

In both cases, X and Y or Z profile measurements, use the **calibgp** pulse program. Initially use $g_{xyyz} = 10\%$ on 20A step with an HR probe (e.g. 5% on 40A step or 3.33% on 60A step with DIFF 30 or DIFF 60 probes). With such small gradient strength, choose a 50kHz spectral width, a time domain TD = 256 or 512k, an accurate 90° RF pulse, a correct d1 and a number of scans NS = 4. Put AQMOD to qsim and DIGMOD = analog as far as DQD may not operate at such high spectral width. Acquire the echo signal, Fourier Transform it with fmc to obtain a magnitude spectrum. You should obtain the gradient profile shown in figure 4.2 below obtained with a spherical insert of 0.25cm along the Z Axis of an HR 5mm TXI Z gradient coil.

Gradient strength calibration curves obtained with the Z Gradient coil of a 5mm TXI probe using the GREAT 1/60 amplifier on the 20A step and a 0.25cm Spherical Water insert (with 50ml pure water inside).

1: $gp_z 1 = 60\%$, 2: $gp_z 1 = 50\%$, 3: $gp_z 1 = 40\%$, 4: $gp_z 1 = 30\%$, 5: $gp_z 1 = 20\%$ and 6: $gp_z 1 = 10\%$.





The experiment can be done with positive G+ or negative G- gradients. The following table summarizes measured positive and negative Z gradient field strength as they were measured on the gradient coil of a 5mm TXI probe when using the GREAT 1/60 amplifier on the 20A step. The gradient strength g_z is given in G/cm and calculated using the formula **[4.2.1]**. The Z gradient system linearity (e.g. Z channel of the amplifier and Gradient coil) may be appreciated on the plot shown in Figure 4.3 below.

Plot of the Gradient strength $g_z = G_z^+ x dz$ (in G.cm⁻¹) against the square pulse gradient amplitude defined by the software ($gp_z 1 = G^+$) when using a Z coil of a 5mm TXI probe. The GREAT 60 amplifier is used on the 20A step. The sample is a 0.25cm spherical water plug.

Installation Tests



Figure 4.3. Plot of the Gradient Strength

Positive/Negative Gradient pulse accuracy

Some experiments use gradient echo sequences in which a Positive Gradient Field is used for spin defocusing and a Negative Gradient Field is used for spin refocusing. All echo/anti-echo pulse sequences as well as some diffusion experiments use such kind of Gradient Fields. These kind of experiments are quite sensitive against the Positive/Negative Gradient Pulse Amplitude especially on the Z axis // to the B₀ field. The following test allows the checking and the matching of some parameters in order to obtain the best Positive/Negative Gradient Pulse Amplitude accuracy. For this purpose, use a classical gradient spin echo sequence as shown in figure 4.4 on the doped water sample (0.1mg/ml GdCl3 + 1% H₂O in D₂O).





To start the experiment set $G_{x,y}^+$ or $_z = -G_{x,y}^-$ or $_z$. Start a **paropt** experiment and set the parameters as following :

Enter parameter to modify : gp_{x'y'z}2 or cnst22 depending on the pulse program syntax,

Enter the initial value : $gp_{x,y,z}^2 = (-gp_{x,y,z}^2 + gp(W) range / 2)$

Enter parameter increment : -0.003%

Enter # of experiments : 33

4.3

(W) gp _{range} = 33 x 0.003. The value of the parameter increment is the smallest step which may be defined with the used 16 bits DAC. As may be seen on figure 4.5, small gradient strength changes ($\Delta G = |G^+ - G^-| \le 0.01$ %) induce a loss of more then 10% of signal refocused in the here used conditions (e.g. 5ms square gradient pulse for defocusing and refocusing). Normally if all the amplifier parameters are accurately set, you should found that:

|G-|=|G+|±0.006: the error is smaller than twice the gradient resolution

The parameters which may influence this test are :

 bad GREAT 60 DC offset matching. Don't forget that pre-emphasis corrections change the DC current level,

Positive/Negative Gradients accuracy tests recorded with the GREAT 60 amplifier on the 20A step and the Z gradient coil of a 5mm TXI probe.





Figure 4.6. $gp_z 1 = 20\%$ and $-19.955 \le gp_z 2 \le -20.045\%$ in -0.003% steps (e.g. around 20 G/cm)



Gradient amplifier stability tests

With the gradient amplifier stability we check the way in which the gradient amplifier is able to sent reproducible pulses to the coil during an NMR experiment. This test allows to check the amplifier DC matching done by the user on the spectrometer (see section "Amplifier DC offset correction" on page 24) and the efficiency of the amplifier DC offset thermal correction. On the production site the thermal behavior of a GREAT 1/40 or 1/60 amplifier is matched in a temperature range of 18 < T _{G opt.} < 27 C (where T _{G opt.} is the optimal room temperature in which high gradient output stability is reached). The principle of this test is to reproduce N times the same pulse sequence (see Figure 4.6) in proton NMR spectroscopy using the doped water sample (0.1mg/ml Gadolinium Chloride in 1% $H_2O + 99\% D_2O$). The test may be done in **paropt** mode or by using the tests included in the BRUKER HWT test procedure. The results have to be compared with the spectrometer stability test when the gradient amplifier is blanked or the gradient cable unplugged. When N is equal to 32 (using a repetition time of 10s) we obtain the so called short time stability test which allows to check the amplifier DC offset matching. When N is equal to 256 (using a repetition time of 15s) we obtain the so called time averaged stability test which allows to check if the temperature gradient output stability is good enough.

G+ / G- Gradient Echo pulse sequence used for gradient amplifier stability tests. The spectra are recorded using 5ms 20 G/cm gradient square pulses on a 5mm High Resolution probehead.



Figure 4.7. Gradient Echo pulse sequence

Short time gradient stability test - Number of scans N = 32

4.4.1

In this experiment, the number of scans N is equal to 32, a repetition delay of 10s (relaxation + acquisition time) is used. The total experiment time is 5mn 20s. The experiment is done with the 10A, 20A, 30A and 60A steps. The gradient strength is maintained unchanged e.g. 40%, 20%, 13.222% and 6.666% respectively. The spectrum shown in figure 4.7 is obtained in these conditions using a 5ms 20G/cm gradient square pulse echo on the 10A step.

Gradient Echo G+/G- short time stability test using the gradient echo sequence recorded on the 1% water sample ($0.1mg/ml \ GdCl_3 + 1\% \ OH_2 \ in \ 0D_2$). Gradient Pulse length = 5ms - Gradient shape = square - Recovery delay = 0.0001s Gradient strength = 20 G/cm.



Figure 4.8. Gradient Echo time stability

Gradient pulse amplitude stability results obtained on the 10A, 20A, 30A and 60A when using the Gradient echo G+/G- sequence using 5ms square pulses at 20 G/cm are summarized in the following table :

Table 4.2.	Short Time	stability test
------------	------------	----------------

	Stability test std Dev. Factor (*)
Gradient echo expt. At 20 G/cm on 10 A step	2.1
Gradient echo expt. At 20 G/cm on 20 A step	1.8
Gradient echo expt. At 20 G/cm on 30 A step	2.3
Gradient echo expt. At 20 G/cm on 60 A step	2.9

(*) The stability test std Dev. Factor = Water peak Int. Std Dev. (Grad. Echo) / Water peak Int. Std Dev. (without Grad.)

The water peak intensity standard deviation calculated over 32 gradient echo experiments must be smaller than three times the water peak intensity standard deviation calculated over 32 single pulse experiments (without gradients). Note that on the 60A step, the stability decreases. This is a consequence of less resolution in the gradient pulse / pre-emphasis / DC offset digital definition (Resolution = 300μ A on the 10A step, 1.8 mA on the 60A step).

Time averaged gradient stability test - Number of scans N = 256	4.4.2
---	-------

In this experiment, the number of scans N is equal to 256, a repetition delay of 15s (relaxation + acquisition time) is used. The total experiment time is 1h4mn. The experiment is done with the 20A step (can be done on all the other steps using the same experimental conditions described here). The gradient strength is

set to 20%. The spectrum shown in figure 4.8 below is obtained in these conditions using a 5ms 20G/cm gradient square pulse echo on the 20A step.

Gradient Echo G+/G- time averaged stability test using the gradient echo sequence recorded on the 1% water sample (0.1 mg/ml $GdCl_3 + 1\% OH_2$ in $0D_2$). Gradient Pulse length = 5ms - Gradient shape = square - Recovery delay = 0.0001s Gradient strength = 20 G/cm.





Table 4.3. Average Time stability test

	Stability test std Dev. Factor (*)
Gradient echo expt. At 20 G/cm on 20 A step	2.0

(*) The stability test std Dev. Factor = Water peak Int. Std Dev. (Grad. Echo) / Water peak Int. Std Dev. (without Grad.)

The water peak intensity standard deviation calculated over 256 gradient echo experiments must be smaller than three times the water peak intensity standard deviation calculated over 256 single pulse experiments (without gradients) recorded in the same conditions (room temperature etc...).

Water self diffusion coefficient measurement

4.5

This last gradient amplifier test is a basic application of diffusion spectroscopy. This test may be done on 5mm High Resolution and Diffusion Probeheads (DIFF 30 / DIFF 60). The water self diffusion coefficient is measured using an echo experiment using gradients during the echo time as shown in Figure 4.9 below. The choice of the gradient pulse length δ and of the evolution time Δ is critical and may be done properly. If δ and Δ are too short or too long, the diffusion curve is not properly defined and the water self diffusion coefficient calculated on the basis of the experimental data points (Water peak Intensity = f (gradient strength)) may be not correct. It is also important as for every NMR experiment that the $\pi/2$ and π proton pulse length are accurately set. In the example shown here, we have used the doped water sample (0.1 mg/ml Gadolinium Chloride + 1% H2O + 99% D2O) and a 5mm HR probehead (100 G/cm maximal gradient strength).

Figure 4.10. Sequence for self diffusion coefficient measurements using a Hahn Echo and symmetric gradients during the echo time



The spectrum shown in Figure 4.10 is obtained by plotting the water peak intensity against the gradient strength. The gradient strength changes in 250mA steps starting at 0A up to 20A. Two square gradient pulses of 800µs duration are used. The gradient recovery delay is set to 100µs and the echo time Δ is equal to 30ms. The experiment shown here is recorded on a 5mm TXI probehead equipped with a Z gradient coil. When using DIFF 30 or DIFF 60 probeheads, shorter δ and Δ have to be used. It is recommended to work on 10 or 20A step (remember that for 10A gradient pulses the gradients strength is 300 G/cm on a DIFF 30 and 600 G/cm on a DIFF 60 probehead).

Water peak intensity decreasing as function of the gradient pulses strength in the Stejskal & Tanner experiment when δ = 800µs and Δ = 30 ms. The experiment is recorded at 300K.



In the Stejskal & Tanner experiment, the self diffusion coefficient of a molecule can be calculated on the basis of Magnetization changes as a function of the square gradient pulses :

$$M_z = M_0 * \exp(-D_{||} \gamma^2 \delta^2 (\Delta - \delta/3) G^2)$$

By plotting the Magnetization (which is proportional to the measured water peak intensity I) against G², we obtain a straight line (figure 4.11.a) and the self diffusion coefficient may be calculated from the slope of the straight line. We should obtain for the water self diffusion coefficient at 303K: 2.59 *10⁻⁹ m²/s ⁽¹⁾. The plot of the calculated water peak intensity against G using the experimental self diffusion coefficient D_{||} deduced from the straight line plot should fit properly the experimental points (Figure 4.11). To obtain good results you may calibrate the gradient strength in G/cm of your gradient coil (see section<u>"Gradient strength calibration</u>).

<u>Left</u> : Plot of the water peak intensity as a function of G², <u>Right</u> : Plot of the calculated and experimental water peak intensity as a function of the square gradient pulse strength G in G/cm. For both plots, dots are experimental water peak intensities whereas the full lines are calculated data. We have measured for the water at 300K a self diffusion coefficient $D_{\rm H} = 2.53 * 10^{-9} \, {\rm m}^2/{\rm s}$.



Figure 4.12. Plots of the water

Appendix

Gradient Pulse Programming

5.1



Please read the manual TOPSIN "Pulse program reference" for more information.

For historical reasons, two different gradients generation syntaxes have been used for gradient pulses in pulse programs: the gs and gp philosophies. In earlier times there was a third one using the **:h** order which was designing the BSMS homospoil gradient pulse. In this chapter we try to give an overview of how to generate gradient pulses in the gs and gp syntaxes used up to now. The basic working of the GCU is to sent some numerical data to the GREAT amplifier. This numerical data is converted to an analogical current inside the amplifier by a 16 bytes DAC. This analogical current is added to the pre-emphasis and DC offset coming from the Master Unit which sent numerical data to the pre-emphasis and offset DAC located inside of the GREAT amplifier. A gradient pulse from the GCU may be presented in his basic form as :

delay: ngrad gradient duration delay: ngrad

Together with this orders, the GCU uses a gradient shape defined by GRDPROG = $\langle gradname \rangle$ which defines on which X, Y or Z axis the gradient is sent, the gradient amplitude (from -100 to 100%) and the gradient shape if the gradient is not a square pulse. The gradient file used in this case is found in the :

/<xwinnmr home>/exp/stan/nmr/lists/gp/ directory with <name> = <gradname>. The <gradname> file is written as follows :

The first three values correspond to the amplitude of the 3 axis gradients as respectively G_x , G_y and G_z . In this example, a square gradient whose amplitude corresponds to 20% of maximal available amplitude is sent to the gradient coil.

As was shown in part <u>"External TCU blanking" on page 13</u>, it is possible to blank/unblank the GREAT amplifier output. If the blanking (GATE connector of the GREAT 1/40 or 1/60) is not connected, the GREAT amplifier is online with the probe. It is possible to work properly in this way. Nevertheless, GREAT amplifier blanking is possible. We use for this purpose the BLKGRAD, UNBLKGRAD, BLK-GRAMP and UNBLGRAMP orders defined. The blanking/unblanking orders may be used before the first gradient pulse and after the last gradient pulse on each X, Y, Z channel. The gradient sequence is written as following :

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^{(0) |(0) |(20)} {(0) |(0) |(0)}

#include <Grad.incl>

delay1 UNBLKGRAD delay: ngrad gradient duration delay: ngrad delay2 BLKGRAD

In this sequence, delay $1 = 4\mu s$ or higher, delay $= 2\mu s$ or higher and delay 2 = gradient recovery time depending if pre-emphasis are needed or not. For square $pulses on a GREAT 60 this delay is equal to <math>50\mu s$ or higher. If the gradient coil of the probe needs with the gradient amplifier higher pre-emphasis base times, the blanking/unblanking orders have to be used in an other way e.g. at the beginning and the end of the pulse sequence:

#include <Grad.incl>

1 ze 2 d1 BLKGRAD delay1 UNBLKGRAD delay: ngrad gradient duration delay: ngrad delay2

go= 2 ph31 d11 BLKGRAD

In this sequence, the position of the GREAT amplifier blanking in the pulse sequence allows to work with higher pre-emphasis time bases. In absence of a pulse sequence involving gradient pulses, the GREAT amplifier output is isolated from the gradient coil of the probe. When the GREAT blanking is operated in this way, the gradient amplifier works in the same manner as a GREAT 1/10 or a GREAT 3/10 amplifier.

The BLKGRAD and UNBLKGRAD orders combine the Lock Hold orders and the gradient blanking orders as defined in the <Grad.incl> file.

Table 5.1. Gradient include file

;Grad2.incl - include file for Gradient Spectroscopy ; for TCU3 ;
;avance-version (00/07/27)
;blank/unblank gradient amplifier and turn lock-hold on/off #define BLKGRAD setnmr3^0 setnmr0^34^32^33 #define UNBLKGRAD setnmr3/0 setnmr0/34/32/33
;blank/unblank gradient amplifier #define BLKGRAMP setnmr0^34^32^33 #define UNBLKGRAMP setnmr0 34 32 33

In this syntax, the Lock Hold order is set by nmrword 3 byte 0, the blanking of the Z gradient by nmrword 0 byte 34, the blanking of the Y gradient by nmrword 0 byte 33 and the blanking of the X gradient by nmrword 0 byte 32.

This was the second way of defining GCU gradient pulses in a pulse program. This philosophy uses the gradient pulse definition statement found in the **<Grad.incl>** file located in the **/<xwinnmr Path>/exp/stan/nmr/lists/pp/** directory. The statement is the following :

#define GRADIENT(ampl) p16:ngrad #define GRADIENT2(ampl) p19:ngrad

With this syntax, p16 designs the pulse length of the GRADIENT() pulse and p19 designs the pulse length of GRADIENT2(). The **gs** syntax uses gradient definition files located in the gp directory. To generate a square pulse, the gradient shape of the GRADIENT pulse is defined as **GRDPROG=1squa.r** for example and the gradient shape of the GRADIENT2 pulse is defined as **GRDPROG2**. Such a gradient programming file to design **one** square pulse is written in the following manner:

p16 { (0) | (0) | (0) + (cnst21) } 40u { (0) | (0) | (0) } { (0) | (0) | (0) }

In this **gp** file example, the GCU generates a square pulse on the Z channel whose amplitude is cnst21. The constant cnst21 is a float number between -100.0 and +100.0%. As far as the gradient amplifier is equipped with a 16 bytes Digital/ Analog Converter (DAC), **the gradient pulse maximal amplitude resolution** given in % of the maximal available current is: **0.003%**.

The way to write a gradient pulse in a pulse program is shown in pre-emphasis test pulse program **preempgs2** :

Table 5.2. preempgs2 pulse program

;preempgs2	ph1=0
favance-version	pist=0
;pulseprogram for preemphasis adjustment	
	;pl1 : f1 channel - power level for pulse (default)
<pre>#include <avance.incl></avance.incl></pre>	;p1 : f1 channel - 90 degree high power pulse
<pre>#include <grad.incl></grad.incl></pre>	:p16: homospoil/gradient pulse
1 78	d1 · relaxation delay: 1-5 * T1
2 41	dif. dolow for homograpil/gradient regenery
2 01	juid: delay for nomosport/gradient recovery
50u UNBLKGRAD	
GRADIENT(cnst21)	;use gradient program (GRDPROG) : 1squa
d16	
4u BLKGRAD	;use gradient : cnst21
p1 ph1	
go=2 ph31	
wr #0	
exit	

In this pulse program, the gradient is defined by the GRADIENT(cnst21) order which is a gradient pulse whose **length is p16**, whose **amplitude is cnst21** and whose shape is **GRDPROG = 1squa**. The delay d16 is normally the gradient recovery delay (in HR the default value is 100µs). The d16 delay allows pre-emphasis to be applied if there are some. In addition d16 must be higher then 10μ s. If you use the consecutive gradients, they must be separated by a delay d16 higher than 10μ s. This is a software limitation. In the case of use of two gradient pulses in pulse program it is possible to define the second gradient pulse as **GRADIENT(cnst22)**. The same pulse length may be used or **GRADIENT2(cnst22)** if an other pulse length is needed. **The GRADIENT(cnst22)** is a gradient pulse whose length is p16, whose amplitude is cnst22 (between -100.0 and +100.0%) and whose shape is GRDPROG whereas the **GRADIENT2(cnst22)** is a gradient

pulse whose length is p19, whose amplitude is cnst22 and whose shape is GRD-PROG.

In the case of the use of two gradients (two square gradient pulses for example), the GRDPROG becomes :

p16 { (0) | (0) | (0) + (cnst21) } 40u { (0) | (0) | (0) } { (0) | (0) | (0) } p16 { (0) | (0) | (0) + (cnst22) } 40u { (0) | (0) | (0) } { (0) | (0) | (0) }

With this syntax it is possible to define other gradient pulse shapes than the square. The GCU includes assembler files which generate sine functions over a defined number of points. The number ratio between the pulse length and the number of points must be > 4µs otherwise a GCU error message is displayed. The following gradient file 1sine.r is given as example:

```
p16 { (0) | (0) | (0) + sine(cnst21,100) }
40u { (0) | (0) | (0) }
{ (0) | (0) | (0) }
```

The square and the sine gradient shape are the most commonly used gradient pulse shapes. Thus for the major NMR experiments using gradient pulses, the square and sine shapes are sufficient. The GCU compiler is able to generate some other internal functions (see XWINNMR acquisition Manual p 277) like linear ramps from -100 to +100% (rd1, rd2, rd3), cosine function (cos), gaussian function (gauss) and plusminus (alternative gradient strength sign change).

The gp gradient syntax

5.1.2

The gp syntax is the newest programming way of gradient pulses. With this syntax a gradient pulse is directly defined inside of the pulse program as **gradient pulse length: gpn** where 0 < n < 31. In a same data set it is possible to define and use up to 32 different gradient shapes. The gradient pulse length may be **pn** associated to a gradient shape **GPNAMEn**. The gradient shape is designed in the same manner as RF shapes using the "**stdisp**" XWINNMR order. The "**stdisp**" generated gradient pulse shapes have to be stored in the **<xwinnmr Pathway>/exp/stan/nmr/lists/gp** directory instead of the **/wave** directory used for RF pulse shapes. With this syntax the gradient recovery test pulse program preempgs2 becomes preempgp2 and is written in the following manner :

Table 5.3.	preempgs2	pulse	program	with gp	syntax
------------	-----------	-------	---------	---------	--------

;preempgp2	d16
;avance-version (00/02/07)	4u BLKGRAD
;pulseprogram for preemphasis adjustment	p1 ph1
	$\sigma o=2 ph31$
<pre>#include <avance.incl></avance.incl></pre>	wr #0
<pre>#include <grad.incl></grad.incl></pre>	exit
1 ze	
2 d1	ph1=0
50u UNBLKGRAD	ph31=0
p16:gp1	
<pre>;pl1 : f1 channel - power level for pulse (default)</pre>	;d16: delay for homospoil/gradient recovery
;p1 : f1 channel - 90 degree high power pulse	;d11: delay for disk I/O [30 msec]
<pre>;p16: homospoil/gradient pulse</pre>	;use gradient : gp 1
;d1 : relaxation delay; 1-5 * T1	;\$Id: preempgp2,v 1.6 2000/05/08 11:40:56 eng Exp \$



The gradient pulse shape name is given by the parameter **GP n or GPNAME n** when the gpn order is used in the pulse program. The shapes shown in figure 2.2 below can be used as gradient shape pulses. Of course you may define and use your own gradient shape pulse. There is only one condition to take into account while designing gradient shape pulse: the Pulse Length / Number of points ratio must be > 4μ s.



Figure 5.1. 90% amplitude square gradient pulse defined over 248 points (the gradient pulse length must be ³ 1ms)





In the **gp** syntax, the gradient pulse length is defined by the order **pn**, the gradient shape by the order **GP n** or **GPNAMEn** relative to the **:gpn** order found in the pulse program. The gradient amplitude is defined by the gpn_x , gpn_y and gpn_z orders (see figure 2.3 below) relative to the 3 X, Y and Z gradient orientations. Do not forget that the maximal gradient strength is given by:

 $G_{x,y,z} = gpn_{x,y,z} x Amplitude$

where **Amplitude** is the maximal shape amplitude in % as defined in the shape tool.

Figure 5.3. Gradient parameters needed for gradient pulse handling in the **gp** syntax as displayed in the XWINNMR "ased" screen display

			====== GRADIENT CHANNEL ======
GPNAM1	RECT.1		file name for gp1
GPX1	0.00	x	x-gradient strength 1
GPY1	0,00	x	y-gradient strength 1
GPZ1	100.00	x	z-gradient strength 1
P16	800.00	usec	homospoil/gradient pulse

Handling gradient ramps

Recording 1D experiments with changing gradient strength (e.g. gradient echo, gradient spin echo, diffusion coefficient measurements) is easily done by running a **paropt** like automation program in which **either cnst21 or gpn** $_{x_1y_1z}$ may be changed. The situation is different in the case of 2D experiments like DOSY, where the **cnst21 and gpn** $_{x_1y_1z}$ parameters can not be changed during the 2D map recording. The situation is the same in Micro Imaging experiments where gradient pulse strength have to be changed in 3D data sets. For this purpose it is possible to use the **"ramp"** facilities of the GCU compiler. The easiest way to define gradient ramps is to use the GCU compiler multiplication facility. It is possible to multiply a gradient pulse statement by numbers or functions as is shown in the two following lines :

- The gradient pulse is multiplied by a constant: p16: gp1*0.75
- The gradient pulse is multiplied by an internal function :

4 p16: gp1*plusminus

..... igrad plusminus

lo to 4 times td1

This function can be used in 2D Echo/Anti echo gradient experiments as far as **plusminus** contains 2 values +1 and -1. Thus the gradient amplitude changes from +G to -G every two igrad statements.

```
4 p16: gp1*sin(100)
```

•••••

.

.....

igrad sin

lo to 4 times 100

In this case the gradient pulse amplitude is **multiplied by one of the 100 points** defining the sin(100) function every igrad statement.

5.1.3

• The gradient pulse is multiplied by an external function :

The last case mentioned here (e.g. gradient pulse statement multiplication by an external function) is the most interesting facility as far as it is possible to define all kinds of ramps and functions for gradient pulse amplitude modulation in multi dimensional experiments. This means that the gradient shaped pulse amplitude may be modulated by some other shaped functions during 2D and 3D acquisition pulse sequences. The principle of gradient pulse statement multiplication is applied in this case ; but this time the multiplier is an external function which may be designed with the shape tool ("stdisp" order in XWINNMR) and stored in the /<xwinnmr path>/exp/stan/nmr/lists/gp directory. For example we generate the following linear amplitude ramp function (see figure 2.4 below) stored in the gp directory with <name>=ramp. It is possible to use this ramp function to multiply a gradient statement. The following order/definition statements have to be used in the pulse program:

#include <Grad.incl>
#include <Avance.incl>
define list<gradient> gramp=<ramp>
 1 ze
 2 d1
 3
4 p16: gp1*gramp
 go=2
 d1 wr #0 id0 igrad gramp
 lo to 3 times td1
As shown in figure 2.4 below, the ampliance of the second secon

As shown in figure 2.4 below, the amplitude of every point defining the shape of the gradient pulse is multiplied by the ramp amplitude which is incremented by every igrad statement. Note that the **dgrad** (decrement grad) statement is also available on the internal GCU compiler.



Figure 5.4. <u>Sine shape of the gradient pulse used in the preceding pulse sequence GPNAM1 = sine.124 with a maximal amplitude of 100%</u>



Figure 5.5. Gradient ramp function incremented every igrad statement



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