BRUKER

BSMS

BSMS User Manual

Version 002

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Introduction 1

Overview 1.1

This manual describes how to use the **B**ruker **S**mart **M**agnet control **S**ystem (BSMS). The BSMS is a completely new Bruker unit that provides computer control of various functions associated with the magnet, magnetic field, and sample. The BSMS has the following subsystems, depending on the configuration of the particular spectrometer:

1. Sample control system (e.g., Lift and Spin).
2. Helium-level and optional Nitrogen-level measuring systems.
3. Room temperature shim control system.
4. Lock control system.
5. GRASP control system.

The BSMS is currently available for use with AMX, ARX, ASX, and AVANCE series spectrometers. A schematic diagram of the BSMS as part of an AMX spectrometer is shown in Figure 1 on page 6.

An overview of all BSMS subsystems is shown in Table 1 on page 7, and a typical configuration of the BSMS front panel is shown in Figure 1 on page 7.

Notation:

Throughout this manual, expressions in quotation marks and in bold italic letters (e.g., ***'4. Service'***) represent what is shown on the BSMS keyboard display. Expressions in square brackets and in bold capital italic letters (e.g., [***ENTER***]) represent keys on the BSMS keyboard.

Figure 1: The BSMS in the AMX Spectrometer

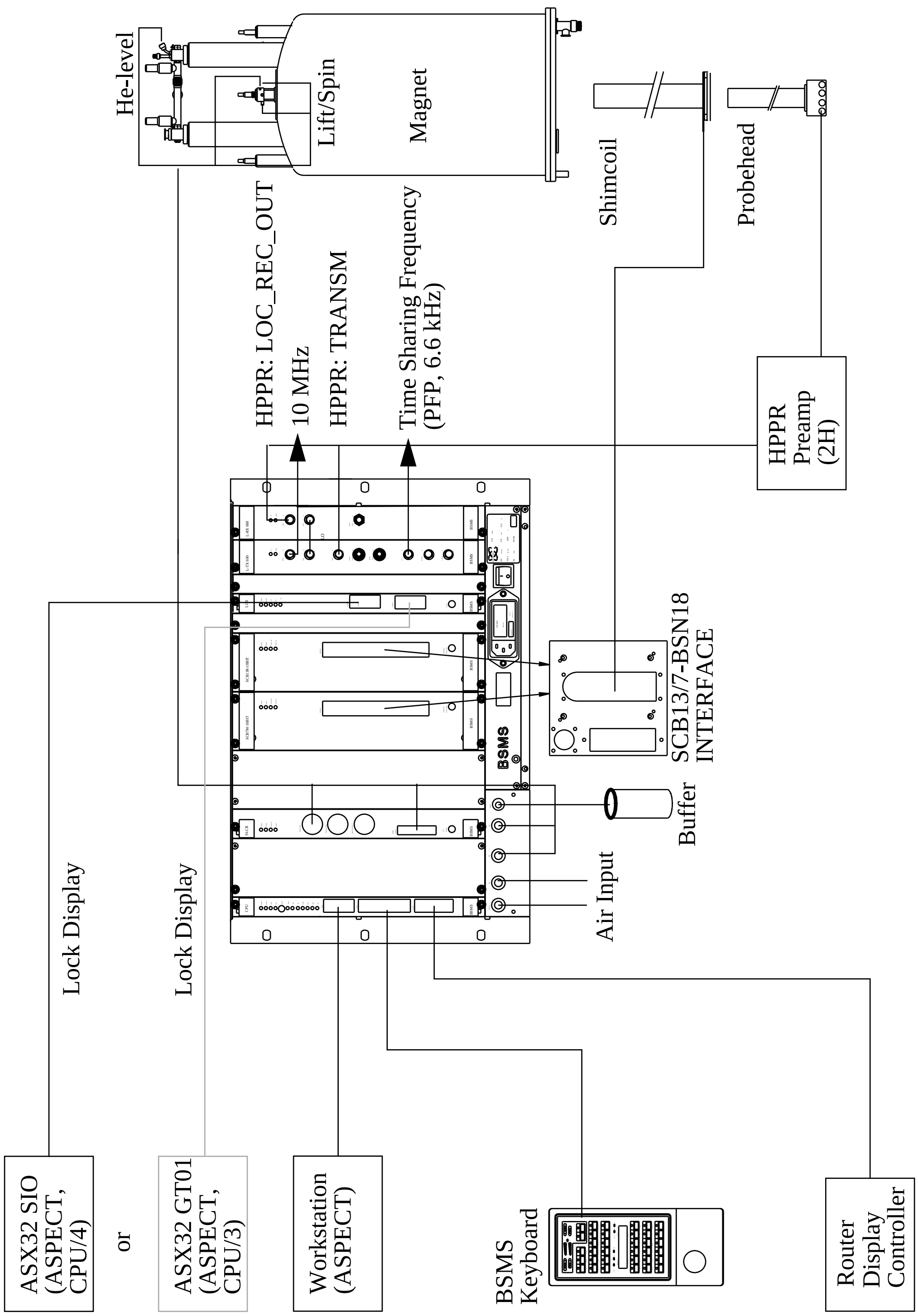
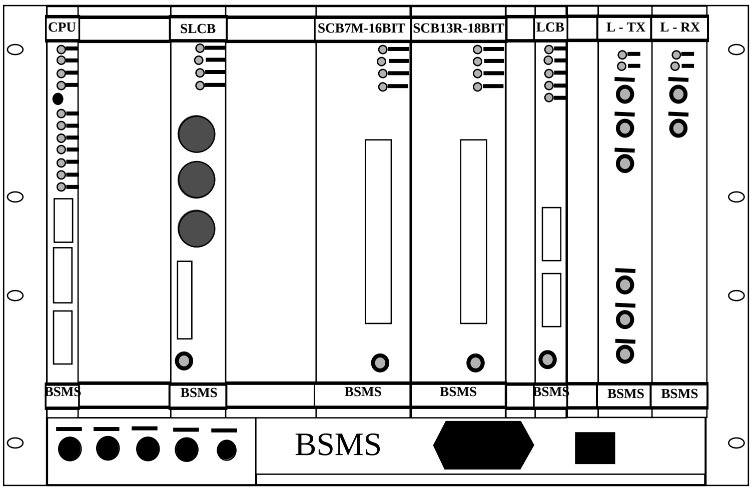


Table 1. Overview of BSMS Subsystems

Subsystem	Boards/Modules	Functions
Mainframe (see also BSMS Mainframe Manual)	Chassis Line Module USERBus VMEBus	19"-rack. Ventilation fan and main switch BSMS specific bus VME standard A16 A24 D16 bus
	Power Supply Module	Power supplies for all boards and devices
	CPU	BSMS master processor Connection for computer (ASPECT) and BSMS keyboard
	BSMS keyboard	User interface of BSMS. Includes router display
Sample and Level (see also BSMS Sample and Level Manual)	SLCB	Helium/nitrogen measuring system, lift and spin control
	Pneumatic Module	Contains all valves to control spin and lift
Shim (see also BSMS Shim Manual)	SCBxx R/M/L	Current sources for the room temperature shim system (BOSS)
GRASP (see also BSMS GRASP Manual)	SCBxx R/M	Current source and control of the Z-gradient on the room temperature shim system (BOSS)
Lock (see also BSMS (Daedalus) Lock Manual)	LCB	H0 power source and digital lock control
	L-TX	Lock transmitter (dependent on spectrometer frequency)
	L-RX	Lock receiver (dependent on spectrometer frequency)

Figure 1: Typical Configuration of the BSMS (Front View)



The BSMS can be operated independently of any computer. Its CPU possesses a non-volatile memory for program and parameter data. The software on all boards (CPU, BSMS keyboard, SCLB, SCB, and LCB) can be updated via the serial link.

Basic Operation 1.2

The keyboard is the primary user interface to the BSMS. All the functions controlled by the BSMS that are important for the general user can easily be accessed and manipulated from the BSMS keyboard. There are two versions of the keyboard: the HR-20 and the BOSS versions. These differ slightly in key layout and also in shim operation procedure. This manual is based primarily on the BOSS version; however, keys unique to the HR-20 version are described in Chapter 3 “Additional Key Description for Version HR-20” on page 21. Other instructions unique to the HR-20 keyboard are also included throughout this manual.

The BSMS keyboard, as shown in Figure 2 on page 10 and Figure 3 on page 11, has the following operation elements:

— An alphanumeric display with two adjacent 1x8-digit LED arrays.

— 35 keys, most with a second function and an indicator LED.

— A menu mode, to access functions that are not directly controlled by any of the 35 keys.

— One control knob, with an adjustable brake placed on the underside of the keyboard.

— 3 sample indicator LED’s (UP, DOWN, and MISSING).

— 2 function LED’s (2nd and MENU).

— One compact router display.

The layout of the BSMS keyboard is divided into different function sections:

— Sample functions ([***LIFT ON/OFF***], [***SPIN RATE***],…)

— Router functions ([***CHAN. SELECT***], [***TRANS P-DOWN***])

— Lock functions ([***FIELD***], [***LOCK PHASE***],…)

— He Level ([***HE-LEVEL***],…)

— Shim functions ([Z^0], [Z^1],…)

— General operation functions ([***DIFF. MODE***], [***STD BY***],…)

In general there are two types of keys:

1. “ON/OFF” keys toggle a function on and off. There is no accompanying message on the alphanumeric display. The LED of the key indicates the state of the function: a lit LED indicates the function is on, an unlit LED indicates the function is off, and a blinking LED indicates the function has not yet reached steady

state (for example, the LED of the [***LOCK ON/OFF***] key blinks during lock-in before the lock has been established). Two examples of “ON/OFF” keys are [***SPIN ON/OFF***] and [***SWEEP***].

2. “Data” keys are used to display the current value of a function and/or to change this value with the control knob. When a “Data” key is activated, the function name appears on the left-hand side of the display and the current value on the right-hand side. If the value of the selected function can be changed from the keyboard, immediately after the control knob is rotated, two numbers appear on the display: the previous value (PREVIOUS SET) on the left-hand side and the new value (ACTUAL) on the right-hand side. The previous value may be kept by pressing the same “Data” key again. The new value may be saved by pressing the [***STD BY***] key (see [***STD BY***] on page 18) or by pressing any other “Data” key. When the control knob is used to change the value of a function, a warning beep sounds if the end of the allowed range has been reached. Two examples of “Data” keys are [***SPIN RATE***] and [***LOCK PHASE***].

Some keys have a second function. To select this second function, press the orange [***2nd***] key and then the key above the desired function name. For example, to select [***AUTO PHASE***] press [***2nd***] and then [***LOCK PHASE***]. For more information on specific keys, please see Chapter 2 “Key Description” on page 13, and Chapter .

In addition, some functions that can be controlled by the BSMS keyboard are not directly accessible by any of the 35 keys. To monitor or change the values of these functions, the keyboard must be put into menu mode, as described in Chapter 4 “Menu Description” on page 23.

Finally, the control knob allows the user to change the value of a “Data”-key function once the key is activated, to move about in the menu mode, and to change the value of a menu-mode function once it is activated. The adjustable brake placed on the underside of the keyboard can be used to adjust how freely the control knob rotates.

Error Messages and Troubleshooting 1.3

If the BSMS detects an error, an error message appears on the keyboard display and a warning beep sounds. Check the error message with the BSMS Service Tool (see Service Tools Manual).

Installation 1.4

Please read the manuals of the appropriate subsystems (e.g., Lock, Shim,…) for the proper installation procedures.

Figure 2: Layout of the BSMS Keyboard (Version BOSS)

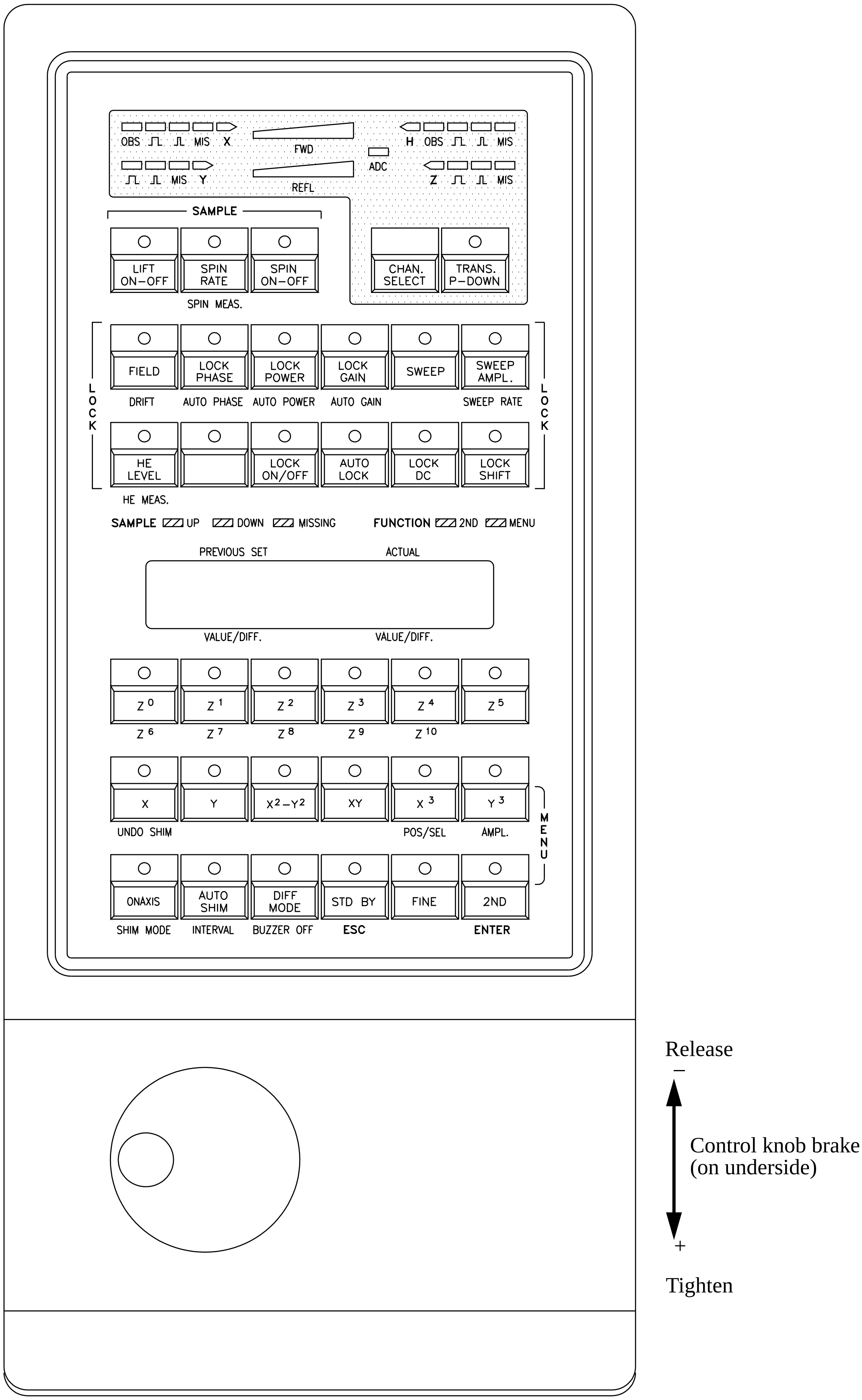
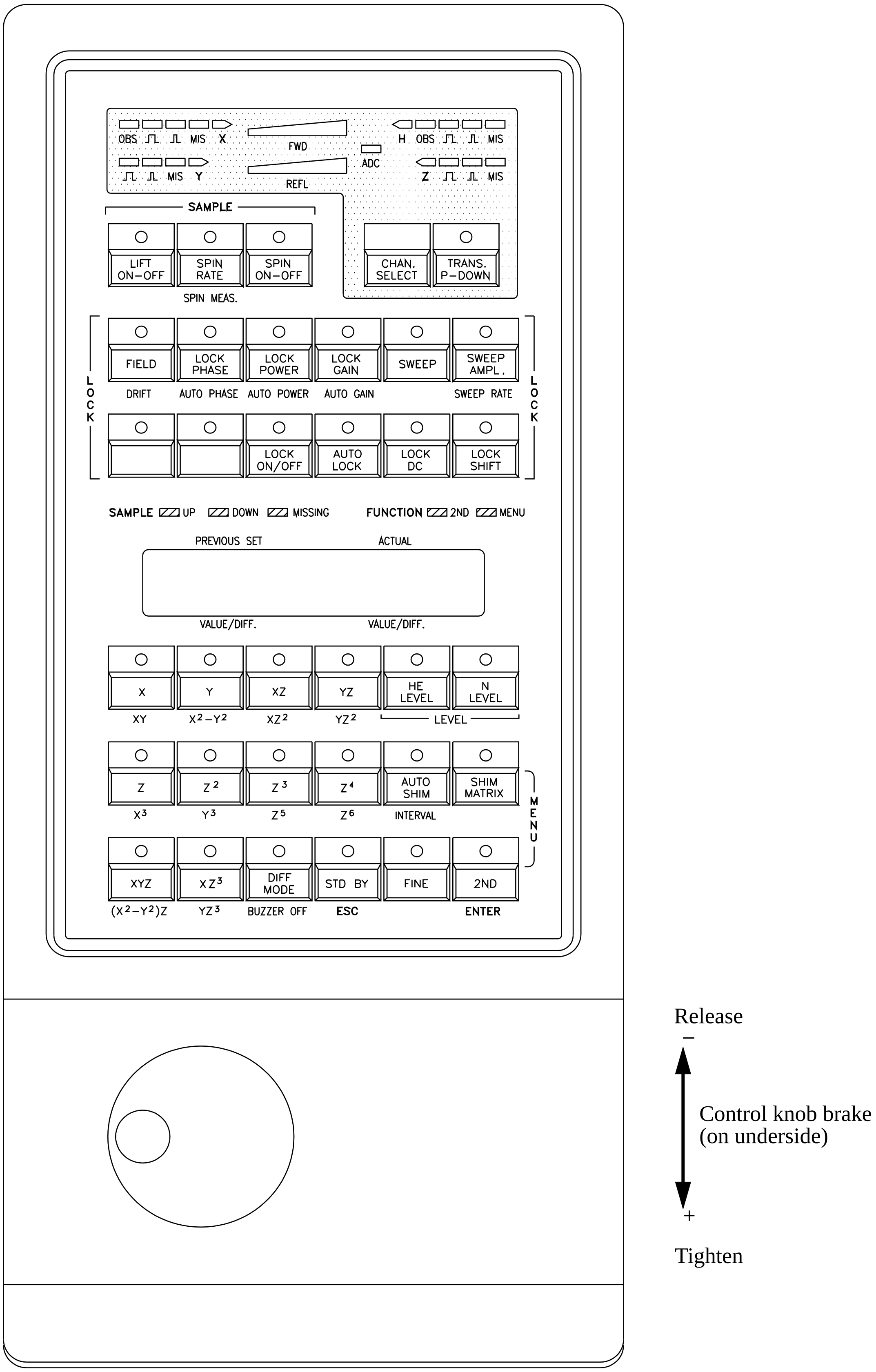


Figure 3: Layout of the BSMS Keyboard (Version HR-20)



Key Description 2

Introduction 2.1

The following is a list of functions of the “ON/OFF” and “Data” keys supported by BOSS keyboards. For keys with descriptions beginning “Enables user to set…”, once the function is activated by pressing the key, the current value of the function can be changed by rotating the control knob.

LIFT ON/OFF 2.2

Switches the sample lift on or off. This simply ejects or inserts the sample, unless sample spinning is activated. If the sample is spinning, pressing [***LIFT ON/OFF***] once will first stop the spinning and then eject the sample. Pressing [***LIFT ON/OFF***] a second time will first insert the sample and then restart the spinning.

SPINRATE 2.3

Enables user to set the rate (in Hz) of sample spinning (range: 7 Hz…Max Spinrate, where Max Spinrate is selected as discussed in ***‘1.6 Max Spinrate’*** on page 24).

SPIN MEAS 2.4

Shows the actual spinrate on the right-hand side of the alphanumeric display and the desired spinrate (as set by [***SPINRATE***]) on the left-hand side. The LED blinks if the actual spinrate deviates from the desired spinrate by more than 20% for a period of time lasting at least one minute. To disable the blinking LED, stop the sample spinning.

SPIN ON/OFF 2.5

Starts or stops the sample rotation. If the actual spinrate deviates from the desired spinrate by more than 5%, the LED starts blinking immediately.

FIELD 2.6

Enables user to set the H0 field (range: +/– 10,000 units). This function is enabled only if the lock is in field mode (see ***‘2.3 Shift/Field’*** on page 25). If the lock is in shift mode, [***FIELD***] can be used to display but not adjust the H0-field value. For optimum lock performance, [***FIELD***] and/or [***LOCK SHIFT***] must be adjusted so that the lock solvent signal is centered on the screen before lock-in.

DRIFT 2.7

Enables user to set the compensation for magnet drift (in [***FIELD***] units per day). This is useful if a long term measurement without lock is performed (for calibration procedure see Lock Operation on page 49). Once [***DRIFT***] is set to a non-zero value, magnet drift compensation occurs only when both lock and sweep are off.

LOCK PHASE 2.8

Enables user to set the phase of the lock receiver (range: 0°…359.9°, endless adjustment). For optimum lock performance, [***LOCK PHASE***] must be set to symmetrize the sweep wiggles seen before lock-in, or equivalently, to maximize the lock signal level observed after lock-in.

AUTO PHASE 2.9

Adjusts the lock phase automatically. This key is active only if lock is previously established. LED blinks during operation.

LOCK POWER 2.10

Enables user to set the output power of the lock transmitter (range: –60.0…0.0 dBm). This is the actual power of the pulses themselves and is independent of duty cycle. For optimum lock performance, [***LOCK POWER***] should be set approximately 6 dB below the minimum level at which the lock signal is saturated.

AUTO POWER 2.11

Automatically sets the lock power. This key is active only if lock is previously established. LED blinks during operation.

LOCK GAIN 2.12

Enables user to set lock receiver RF gain (range: 75.0…155.0 dB). This should be set high enough to ensure best utilization of the ADC, but not so high as to cause receiver gain overflow. Receiver gain overflow can be recognized by excessive noise in the lock signal, and a decrease in lock level with an increase in lock gain.

AUTO GAIN 2.13

Automatically sets the lock gain. This key is active only if lock is previously established. LED blinks during operation.

SWEEP 2.14

Switches the H0-field sweep on or off. This key is active only if lock is ***not*** established.

SWEEP AMPL. 2.15

Enables user to set the amplitude of the H0-field sweep (range: 0.0…100.0 units). A sweep amplitude value of 100 causes the H0 field to be swept over approximately one third of the full [***FIELD***] range (i.e., approximately +/– 3,000 [***FIELD***] units).

SWEEP RATE 2.16

Enables user to set the rate of the H0-field sweep (range: 0.01…5.00 Hz). This is also the sweep rate observed in the lock display on the computer screen, and is independent of the sweep amplitude.

LOCK ON/OFF 2.17

Switches the lock on or off. LED blinks during lock-in procedure before the lock is established.

AUTO LOCK 2.18

Starts or stops automatic lock-in procedure. LED blinks during operation. During autolock, a ^{2}H (or ^{19}F, depending on the lock nucleus) spectrum of the lock solvent is acquired. This is referred to as the FFA-spectrum and is used to determine the correct field value. The field value is then adjusted accordingly, lock-in is begun, and the lock gain is adjusted automatically.

LOCK DC 2.19

Enables user to set the lock DC level (range: –100.0…+100.0 units). This simply sets where the lock signal, for a given lock power and gain, appears on the screen. The lock DC level can be shifted by +/– one-half screen height.

LOCK SHIFT 2.20

Enables user to set the frequency of the lock pulse in ppm of the basic deuterium frequency (range: –200.000…+200.000 ppm). This function is enabled only if the lock is in shift mode (see ***'2.3 Shift/Field'*** on page 25). If the lock is in field mode, [***LOCK SHIFT***] can be used to display but not adjust the frequency value. For optimum lock performance, [***LOCK SHIFT***] and/or [***FIELD***] must be adjusted so that the lock solvent signal is centered on the screen before lock-in.

HE-LEVEL 2.21

Displays the last two measured liquid helium levels, as determined either manually (with [***HE MEAS.***]) or automatically (see BSMS Sample and Level Manual).

HE MEAS. 2.22

Starts a manual liquid helium level measurement in the dewar. This takes about 12 s. Afterwards, the last measured level and the present level are shown on the display, in percent. This measurement resets the automatic He-level measurement function (which occurs every 26 hours). If the He level, as determined by this manual measurement, is too low **no** error message on the BSMS keyboard appears (see ***'9.6 Alarm-Level'*** on page 38).

Z^0...Z^{10}, X, Y, X^2–Y^2, XY, X^3, Y^3, ONAXIS 2.23

Enables user to set a value (range: +/– 130,000 units) for one of the available shims. The general procedure for selecting and adjusting a particular shim is as follows:

1. If an axial shim is desired, first press [***ONAXIS***] and then the desired key from the possible Z shims [Z^0],...[Z^{10}]. The shim value is adjusted by rotating the control knob.
2. Once the [***ONAXIS***] key is activated, to select a new axial shim it is only necessary to press the new Z key.
3. For a radial shim, first select the desired X/Y portion of the shim with one of the keys [***X***], [***Y***], [X^2–Y^2], [***XY***], [X^3],or [Y^3]. Then select the desired Z portion with one of the keys [Z^0],...[Z^{10}]. The selected shim and its present value are shown in the display window. As above, the shim value is adjusted by rotating the control knob.
4. To select a new radial shim with the same X/Y portion, it is only necessary to press the new Z key.

Examples:

Z^2: First press [***ONAXIS***], then [Z^2], and then adjust the shim value.
X: First press [***X***], then [Z^0], and then adjust the shim value.
XZ: (If X already selected as above) press [Z^1] and then adjust the shim value.

N.B.: Some shim systems do not support all shims that can be selected on the BSMS keyboard. A beep sounds when a shim is selected that is not supported by the particular shim system in use.

UNDO SHIM 2.24

Sets all shim values to the state existing after the last load of shim values from the computer, the last shim mode change (see [***SHIM MODE***]), or the last powerup of the BSMS, whichever occurred most recently.

POS/SEL, AMPL 2.25

These keys are to be used by a service engineer only. They are active only if your shim system supports a service shim mode (also referred to as a parameter shim mode). [***POS/SEL***] is used to select the current source number and [***AMPL***] to adjust the value of the selected current source.

SHIM MODE 2.26

Selects the shim mode. Possible modes are user, service, and install (see BSMS Shim Manual). For routine use, user mode should be selected.

AUTOSHIM 2.27

Activates the automatic shim routine which has the following algorithm (see ***'5. Shim-Ampl.'*** on page 29):

1. Find the next active shim, that is, one with a shim amplitude value not equal zero. Add the shim amplitude value to the current shim value.
2. Wait time set by [***INTERVAL***] (in seconds).
3. If the lock level increases or stays the same, return to step 1. If the lock level decreases, change the sign of the shim amplitude value of the selected shim and then return to step 1.
4. Repeat steps 1 to 3 until a break results. Possible break conditions are
   1. [***AUTOSHIM***] deactivated.
   2. Lock lost or deactivated.
   3. User selects any shim.
   4. Shim mode changed.
   5. Shim values loaded.
   6. Shim range changed.
   7. Any malfunction.

Only one break condition need be true for autoshimming to stop.

Note that the [***AUTOSHIM***] function is not active if lock is not established, all shim amplitude values equal zero, or shim mode is not set to User Mode. Also, if autoshimming stops because of break condition 2 (lock lost or deactivated), re-establishing the lock automatically restarts the autoshimming.

INTERVAL 2.28

Enables user to set the time (in seconds) between successive shim value changes during automatic shimming (see [***AUTOSHIM***]) (range: 1…30 s).

DIFF.MODE 2.29

Places the alphanumeric display in differential mode. When this mode is active, a Δ appears before the numbers shown in the display. Once the control knob is moved to adjust the current value of the selected function, two numbers appear in the display. The number to the right indicates the difference between the current value and the last stored value of the selected function. The number to the left indicates the difference between the last and the second-to-the-last stored values.

BUZZER OFF 2.30

Switches the buzzer off if the buzzer is on.

STD BY 2.31

Places the keyboard in standby mode. With this key, it is possible to finish and save an input without selecting another function.

FINE 2.32

Changes the sensitivity of the control knob. If the LED is on, the fine sensitivity is active. When coarse sensitivity is selected, the total change in function value per rotation of the control knob increases with increasing knob rotation rate. When fine sensitivity is selected, the total change in function value per rotation of the control knob is independent of knob rotation rate.

2nd 2.33

Selects the second function of a key. For example, to select [***AUTO PHASE***] first press the [***2nd***] key and then the [***LOCK PHASE***] key.

MENU 2.34

Simultaneously pressing [***2nd***] and [Y^3] activates the menu mode (see Menu Description on page 23).

ENTER 2.35

Enters successive sublevels of the menu and saves new function values. Only active in menu mode (see Menu Description on page 23).

ESC 2.36

Exits successive sublevels of the menu and quits a function without storing a new value. Only active in the menu mode (see Menu Description on page 23).

CHAN. SELECT 2.37

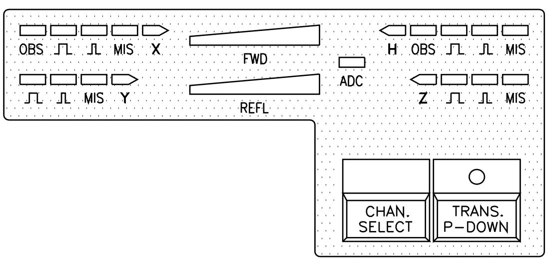
Selects the transmitter channel shown on the Router display (X, H, Y, or Z). Only active channels can be selected.

TRANS P-DOWN 2.38

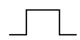
“Powers down” the transmitter system by disconnecting the RF from the transmitter input. Once powered down, the transmitter can be powered up only via computer or hardware reset. If the LED is lit the transmitter system is inactive.

Router Display 2.39

Figure 4: Layout of the Router Display



N.B.: This display is not supported from all systems.

H,X,Y,Z	Indicates selected channel.
OBS	Indicates observe channel.
	Indicates RF pulse in amplifier.
	Indicates RF pulse with corresponding output power exceeding a predefined level.
MIS	Mismatch display (SWR fault).
ADC	Indicates ADC active.
FWD	(Forward) Real time display of the forward power of the selected transmitter channel (linear scale).
REFL	(Reflected) Real time display of the reflected power of the selected transmitter channel (linear scale).

Chapter

Additional Key Description for Version HR-20 3

Introduction 3.1

The HR-20 keyboard differs from BOSS keyboards primarily with respect to the number of shims available and the cryogen level functions. There are several keys supported by BOSS keyboards but not by the HR-20 keyboard. These include [***UNDO SHIM***], [***POS/SEL***], [***AMPL.***], and [***ONAXIS***]. Other differences are listed below.

LEVEL 3.2

The second function of He- and N-level keys measures the actual level of the appropriate cryogen (see [***N MEAS.***] on page 21 and [***HE MEAS.***] on page 16). The N-level measurement system is optional.

N-LEVEL 3.3

Displays the last two measured nitrogen levels (see [***N MEAS.***] on page 21).

N MEAS. 3.4

Starts a nitrogen level measurement in the dewar and displays the last level measured as well as the present value.

$Z \ldots Z^6$, $X \ldots XZ^3$, $Y \ldots YZ^3$, $X^2{-}Y^2$, $(X^2{-}Y^2)Z$, XY, XYZ, X^3, Y^3 3.5

Enables user to set a shim value (range: +/– 130,000 units) of one of 20 possible shims.

MENU 3.6

Simultaneously pressing [***2nd***] and [***SHIM-MATRIX***] activates the menu mode (see Menu Description on page 23).

SHIM MATRIX 3.7

Not yet supported.

Menu Description

Introduction 4.1

The menu mode of the BSMS keyboard enables the user to select and adjust many functions that are not accessible by the “Data” and “ON/OFF” keys described in the previous two chapters. To enter and operate the menu only the following keys are necessary in addition to the control knob:

MENU

Simultaneously pressing [***2nd***] and [***Y³***] on BOSS keyboards (or [***2nd***] and [***SHIM-MATRIX***] on the HR-20 keyboard) places the keyboard in menu mode. The red “Function Menu” LED, located above the alphanumeric display, blinks when menu mode is active. Once in this mode, the [***2nd***] key becomes the [***ENTER***] key and the [***STD BY***] key becomes the [***ESC***] key.

ENTER

Goes to the next lower menu level or finishes and saves an input.

ESC

Leaves the selected menu level and goes to the next higher menu level, or quits an input without saving any change to the previous value.

Control Knob

By rotating the control knob it is possible to view all the menu items of a given level, and once a particular function is selected, to adjust the function value.

The menu is composed of the following submenus: ***‘1. Sample’***, ***‘2. Lock’***, ***‘3. Keyboard’***, ***‘4. Service’***, ***‘5. Shim-Ampl.’***, ***‘6. Shim-Sens.’***, ***‘7. GRASP’***, ***‘8. N-Function’***, ***‘9. He-Function’***, ***‘10. Shim-Ranges’***, and ***‘11. Shim-Current’***. Each of these submenus has several functions, which are described below.

Reminder of notation:

Expressions in quotation marks and in bold italic letters (e.g., ***‘4. Service***’) represent what is shown on the BSMS keyboard display. Expressions in square brackets and in bold capital italic letters (e.g., [***ENTER***]) indicate keys.

All submenus and functions marked with *****security code required** can be accessed only if the correct security code has been entered (see ***‘4.1 Sec.-Code’*** on page 28). ***These functions are for service only.***

1. Sample 4.2

Several functions for lift and spinner adjustment and calibration are implemented in this submenu. All typical values relate to a system pressure of approximately 5 bar and a shim upper part of type BST (Bruker Sample Transport system).

1.1 SpinCal 4.2.1

Performs an automatic calibration of the spin control. This calibration is needed if the spinrate fluctuation is unacceptable or if the maximum spinrate has been changed (see ***'1.6 Max Spinrate'*** on page 24).

1.2 Airflow 4.2.2

Enables user to set the air flow rate of the sample lift (range: 0…***'Max Airflow'*** value, typ.: 500 units). This should be chosen so that the sample floats on top of the BST.

1.3 Lift On/Off 4.2.3

Turns the sample lift on or off.

1.4 Max Airflow 4.2.4

*****security code required**

Enables user to set the maximum air flow rate of the sample lift (range: 0…1000 units, typ.: 600 units).

1.5 Lift Offset 4.2.5

*****security code required**

Enables user to set the air flow rate that ensures the sample falls gently into the turbine when sample lift is turned off (range: 0…***'Airflow'*** value, typ.: 150 units).

1.6 Max Spinrate 4.2.6

*****security code required**

Enables user to set the maximum spinrate (range: 4…100 Hz, typ.: 50 Hz (standard bore) or 30 Hz (wide bore)). After changing the maximum spinrate, it is necessary to do a spin calibration (see ***'1.1 SpinCal'*** on page 24).

2. Lock 4.3

Includes lock parameters which cannot be accessed by "Data" or "ON/OFF" keys.

2.1 Loop Gain 4.3.1

Enables user to set the PI control gain (range: –80…0 dB) of the regulator used once lock-in is achieved. A typical value is –35 dB. Higher (i.e., less negative) values may be used when the lock signal is strong. If the S/N of the lock signal is low, it is necessary to use a low gain value to avoid introducing noise modulation into the spectrum.

2.2 Loop Time 4.3.2

Enables user to set the PI control time constant (range: 0.001…1.000 s) of the regulator used once lock-in is achieved. A typical value is 0.05 s. In general, a longer time constant should be used for a noisier lock signal.

2.3 Shift/Field 4.3.3

Enables either field adjust or shift (i.e., frequency) adjust of the lock. Within this menu function, selecting ***'Field 0'*** activates the [***FIELD***] key, while selecting ***'Shift 1'*** activates the [***LOCK SHIFT***] key (default: ***'Field 0'***).

2.4 Display Mode 4.3.4

Enables user to select the lock display mode from one of the following:

Display mode name	Display mode number	Function
Re	0	Absorption signal (default)
Re Lp	1	Absorption signal (low-pass filtered)
Im	2	Dispersion signal
Cont.out	3	Regulator output
Re ex.	4	Absorption signal (8 * expanded amplitude)
Re LP ex	5	Absorption signal (low-pass filtered and 8 * expanded amplitude)
FFA Spec	6	Last FFA-spectrum
Cont. ex	7	Regulator output (expanded amplitude)
Reserve2	8	Reserved for further options

The absorption mode (Re 0) is appropriate for normal operation. The regulator output mode (Cont.out 3) can be used for observing the lock hold during GRASP experiments. Other modes are primarily for debugging purposes.

2.5 Z0-Comp. 4.3.5

Enables or disables Z0 compensation. Z0 compensation may be used in GRASP experiments to counteract any changes in H0 caused by the gradient pulses. This is an optional function and so is active only if installed.

2.6 RS-Baudrate 4.3.6

*****security code required**

Enables user to set the baudrate of the lock display serial link (optional). Within this function, the baudrate (in baud) is shown on the left-hand side of the display. The number on right-hand side is for orientation in the menu only.

3. Keyboard 4.4

Includes functions to control the keyboard and functions to display various software and hardware version numbers.

3.1 Lock Keyb. 4.4.1

Entering this function locks the BSMS keyboard (useful during a long experiment). ***'Keyboard locked'*** appears on the display and the control knob and all keys (except [***ESC***]) are disabled. To exit this mode, press [***ESC***].

3.2 Brightness 4.4.2

Enables user to set the brightness of the display and LED's (adjustable in 6 steps).

3.3 Displaytest 4.4.3

Tests keyboard display and all LED's (duration: approx. 6.5 s).

3.4 AppSW-Date 4.4.4

Displays the applications software date of the BSMS keyboard.

3.5 BootSW-Date 4.4.5

Displays the boot software date of the BSMS keyboard.

3.6 HW-Ver 4.4.6

Displays the hardware version of the BSMS keyboard controller board.

3.7 Disp.HW-Ver 4.4.7

Displays the hardware version of the BSMS keyboard display board.

4. Service 4.5

This submenu is for service only!

4.1 Sec.-Code 4.5.1

Enables user to enter the security code. When the security code is entered correctly, all keyboard submenus and functions are accessible.

Note: The current values of the functions accessible only with the proper security code are set by the service engineer. There should be no need to adjust these settings. If desired, however, the security code can be obtained by reading the Service Tool manual or by calling your Bruker Service Center.

4.2 Save Config 4.5.2

*****security code required**

Saves all configuration data of the BSMS to the CPU. After a reboot of the BSMS all saved configuration data are automatically written to the appropriate boards in the BSMS.

N.B.: Do not save the configuration if the BSMS shows any errors.

5. Shim-Ampl. 4.6

The shim amplitude and interval functions included in this submenu enable the user to define the algorithm used for autoshimming (see [***AUTOSHIM***] on page 17).

Ampl. Z, Z^2…XYZ^5, Z^0 4.6.1

Enables user to set the step size (shim amplitude) for each shim.

All to 0 4.6.2

Resets all shim amplitudes to zero.

Active? 4.6.3

Displays all shims that are active in the autoshim algorithm (i.e., whose step size is non-zero).

Interval 4.6.4

Has same function as the key (see [***INTERVAL***] on page 17).

Autoshim 4.6.5

Has same function as the key (see [***AUTOSHIM***] on page 17).

6. Shim-Sens. 4.7

The control knob sensitivity of each shim is adjustable (see [***FINE***] on page 18). The shim sensitivity is absolutely independent of the shim current sources.

Sens. Z, Z^2...XYZ^5, Z^0 4.7.1

Enables user to set a coarse and a fine control knob sensitivity for each shim. The user first selects the desired shim using the control knob and [***ENTER***], then selects coarse or fine with [***FINE***] (fine is selected when LED is lit), and then selects the sensitivity from the following list of possible values: 1, 2, 3, 5, 7, 10, 20, 30, 50, 70, 100, 200, 300, 500, 700, 1000, where larger numbers indicate increased knob sensitivity.

Defaults 4.7.2

Sets all shim sensitivities to their default values.

Save 4.7.3

Saves all shim sensitivities to the CPU of the BSMS.

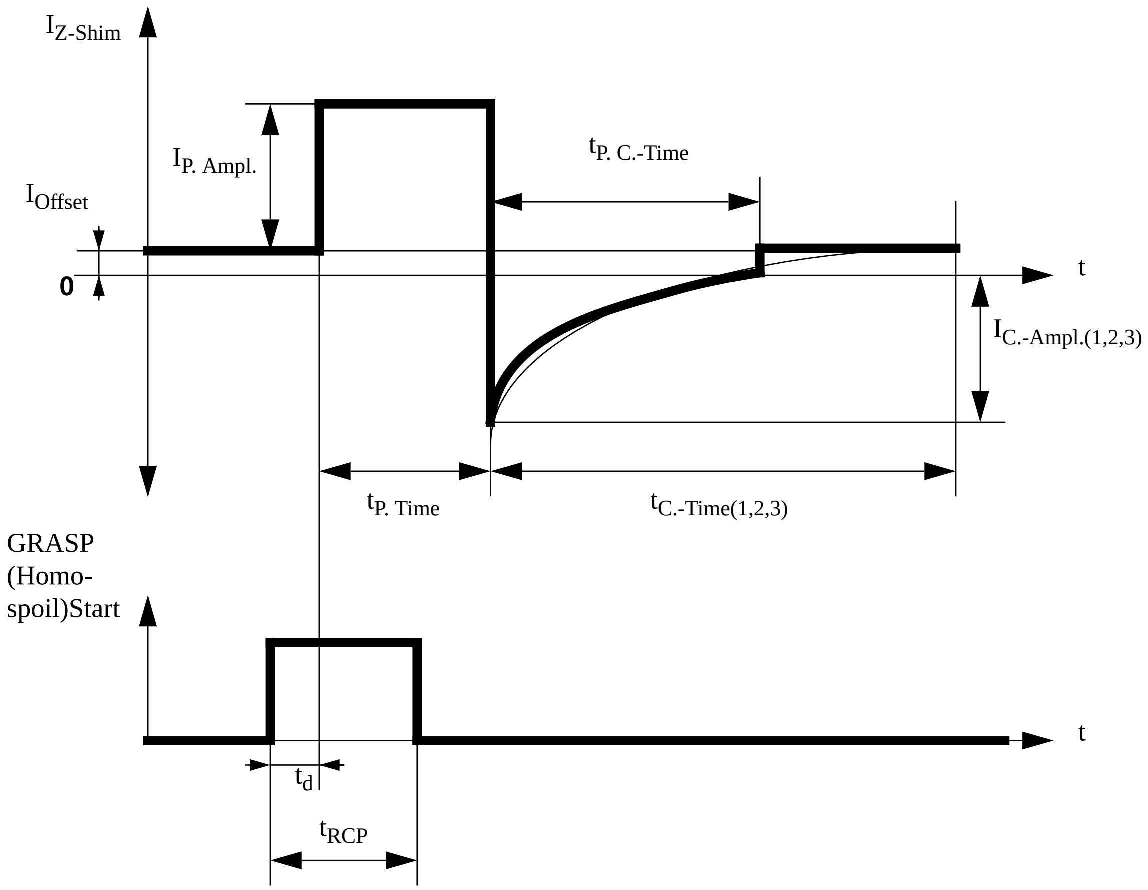
7. GRASP 4.8

GRASP (GRadient Assisted SPectroscopy) experiments utilizing the Z-shim coil can be set up and performed easily with the BSMS. Using the keyboard, the user may define up to 9 gradients with different amplitudes (***'7.2 P. Ampl.'***), durations (***'7.3 P. Time'***), and eddy-current compensation times (***'7.4 P. C.-Time'***). The user may also define one pulse shape (***'7.5 Shape'***), eddy-current compensation form (***'7.8 C.-Ampl.1'***,...***'7.13 C.-Time 3'***), and offset (***'7.14 Offset'***) to be applied to the gradients. Finally, a gradient sequence can composed from the previously defined pulses (***'7.6 Enter Seq.'***). It is also possible to define all GRASP parameters via computer. Each gradient is triggered with a GRASP (Homospoil) start pulse which is controlled by a real-time clock pulse (RCP). The maximum strength for each gradient is on the order of 2 Gauss/cm for BOSS1 and BOSS2 shim systems. This allows simple GRASP experiments such as COSY (see GRASP Operation manual) and HETCOR. Note, however, that this gradient strength is not sufficient for GRASP experiments on aqueous solutions if the coherence selection (and hence the water suppression) is based solely on gradients.

The timing and parameters of a GRASP pulse are shown in Figure 5 on page 32. The GRASP start pulse is provided by an RCP (this trigger pulse must be delivered to the front panel of the SCB13R). The actual gradient pulse begins after a fixed 150 µs trigger delay and lasts for a time defined by ***'7.3 P. Time'***, which is independent of the length of the RCP. At the completion of the gradient pulse, the compensation (preemphasis) pulse begins. There is only one compensation pulse form, defined by three exponential functions. The decay times of these three exponentials are defined by ***'7.9 C.-Time 1'***, ***'7.11 C.-Time 2'***, and ***'7.13 C.-Time 3'***. The amplitudes are defined by ***'7.8 C.-Ampl.1'***, ***'7.10 C.-Ampl.2'***, and ***'7.12 C.-Ampl.3'***, where these amplitudes are expressed as negative percentages of the preceding gradient pulse amplitude. The length of the compensation pulse is defined by ***'7.4 P. C.-Time'***. At the end of this time, the compensation amplitude is set to zero.

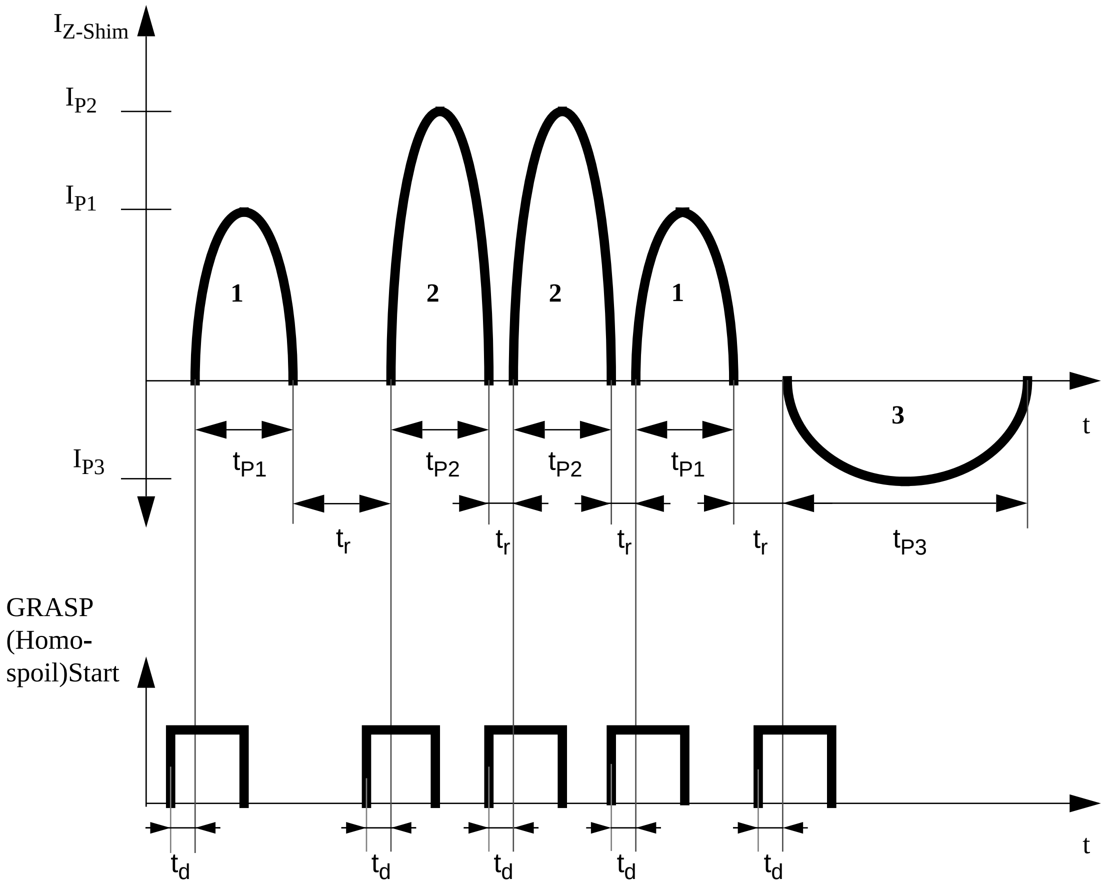
Note that the combination of shaped gradient pulse and compensation pulse is made up of ≤223 sample points each with duration ≥100 µs. Thus, if a gradient and compensation pulse pair lasts ≤22.3 ms, it will be made up of ≤223 sample points of 100 µs duration. On the other hand, if it lasts >22.3 ms, it will be made up of 223 sample points of >100 µs duration, where these pulses last an integral number of microseconds.

Figure 5: Timing of a GRASP Pulse



$I_{P.\ Ampl.}$:	Gradient amplitude (max. +/–1 A, resolution: 12 bit).
$t_{P.\ Time}$:	Gradient length (min. 300 µs, max. 0.1 s) sample point length: 100 µs if ($t_{P.\ Time} + t_{P.\ C.\text{-}Time}$) ≤ 22.3 ms >100 µs (resolution: 1 µs) if ($t_{P.\ Time} + t_{P.\ C.\text{-}Time}$) > 22.3 ms
$t_{P.\ C.\text{-}Time}$:	Compensation pulse length. The compensation amplitude is set directly to 0 at the end of this time.
$t_{C.\text{-}Time(1,2,3)}$:	Decay times of the three exponential functions which make up the compensation pulse form.
$I_{C.\text{-}Ampl.(1,2,3)}$:	Amplitudes of the three exponential functions which make up the compensation pulse form. These are expressed as negative percentages of $I_{P.\ Ampl.}$ of the immediately preceding gradient pulse.
I_{Offset}:	Offset of the GRASP hardware (see Sample and Spectrometer Setup in the GRASP Operation manual).
t_d:	150 µs fixed trigger delay.
t_{RCP}:	Duration of the GRASP Start Pulse (min. 300 µs). The start pulse must be delivered to the SCB13R front panel.
N.B.:	***The duration of the GRASP Start Pulse does not correspond to the actual duration of the gradient pulse.***

Figure 6: Hypothetical GRASP Sequence (1-2-2-1-3)



I_{P1}, t_{P1}:	Amplitude and duration of gradient with index 1.
I_{P2}, t_{P2}:	Amplitude and duration of gradient with index 2.
I_{P3}, t_{P3}:	Amplitude and duration of gradient with index 3.
t_d:	150 µs fixed trigger delay.
t_r:	150 µs minimum recovery time.

7.1 Sel. Pulse 4.8.1

Enables user to select the index number of the gradient to be manipulated or defined. When using the keyboard, the user can define up to 9 different gradients.

7.2 P. Ampl. 4.8.2

Enables user to set the amplitude (in % of the maximum current) and sign of the selected gradient, indicated by the index number on the left-hand side of the display (range: –100%…+100%).

7.3 P. Time 4.8.3

Enables user to set the duration (in µs) of the selected gradient, indicated by the index number on the left-hand side of the display (range: 300…100,000 µs).

7.4 P. C.-Time 4.8.4

Enables user to set the duration (in µs) of the compensation pulse following the selected gradient, indicated by the index number on the left-hand side of the display (range: 0…1,000,000 µs). This time merely chooses what portion (starting from the beginning) of the compensation pulse form will be used with the selected gradient. This pulse provides simple preemphasis to compensate for eddy currents.

7.5 Shape 4.8.5

Enables user to set the shape to be used for **all** gradient pulses. Currently, there are five implemented shapes with index numbers as shown below:

0. Square
1. Sine
2. Trapezoid (1:6:1 ratio)
3. Triangle (symmetrical)
4. Gauss

7.6 Enter Seq. 4.8.6

Enables user to define the sequence of gradients. The index numbers in the sequence correspond to the gradients defined previously, and the end of the sequence must be marked by the flag ‘***End***’. Note that all gradients of the sequence have the same shape. Due to limitations of the alphanumeric display, the sequence defined here may contain only up to 7 gradients. The character ‘**_**’, which appears on the display as well as in the following example, indicates cursor position.

EXAMPLE: GRASP Sequence 1-2-2-1-3 (see Hypothetical GRASP Sequence (1-2-2-1-3) on page 33)

1. Enter the menu ([***2nd***] and [***Y***^{3}] for BOSS keyboards).
2. Enter the GRASP submenu and the Enter Sequence function (***‘7. GRASP’***, [***ENTER***], ***‘7.6 Enter Seq.’***, [***ENTER***],‘***_ 1***’).
3. Select ‘***1***’ with the control knob and enter it with [***ENTER***] (‘***_ 1***’, [***ENTER***], ‘***1< End***’).
4. Select and enter ‘***2***’ (‘***1_ 2***’, [***ENTER***], ‘***12< End***’).
5. Select and enter ‘***2***’ (‘***12_ 2***’, [***ENTER***], ‘***122< End***’).
6. Select and enter ‘***1***’ (‘***122_ 1***’, [***ENTER***], ‘***1221< End***’).
7. Select and enter ‘***3***’ (‘***1221_ 3***’, [***ENTER***], ‘***12213< End***’).

8. Select and enter '***End***' ('***12213< End***', [***ENTER***], ***'7.6 Enter Seq.'***).
9. Leave the menu. ([***ESC***], [***ESC***], '***Standby***').

Note that in the Enter Sequence function, pressing [***ESC***] once moves the cursor one step to the left until it is on the first position of the sequence. When the cursor is at the first position, pressing [***ESC***] quits the Enter Sequence function without saving any changes. Similarly, [***ENTER***] can be used to move the cursor one step to the right until it is on the last position of the sequence (which must be the end-of-sequence flag '***End***'). When the cursor is at the last position, pressing [***ENTER***] exits the Enter Sequence function, saving all changes. Thus, to edit a gradient sequence in this function, use [***ENTER***] and [***ESC***] to position the cursor, the control knob to select new gradient index numbers or the end-of-sequence flag, [***ENTER***] to exit and save the new sequence, and [***ESC***] to quit without saving the new sequence.

7.7 Reset Seq. 4.8.7

Resets the internal sequence pointer to the first index of the sequence.

7.8 C.-Ampl.1 4.8.8

Enables user to set the amplitude (in negative % of the gradient pulse amplitude) of the first compensation set (range: –100%…+100%). Here, for example, +100% corresponds to a compensation set amplitude equal in magnitude but ***opposite*** in sign to the immediately preceding gradient pulse. Thus, in general, a positive value should be chosen.

7.9 C.-Time 1 4.8.9

Enables user to set the time constant (in µs) of the first compensation set (range: 0…100,000 µs).

7.10 C.-Ampl.2 4.8.10

Enables user to set the amplitude (in negative % of the gradient pulse amplitude) of the second compensation set (range: –100%…+100%).

7.11 C.-Time 2 4.8.11

Enables user to set the time constant (in µs) of the second compensation set (range: 0…100,000 µs).

7.12 C.-Ampl.3 4.8.12

Enables user to set the amplitude (in negative % of the gradient pulse amplitude) of the third compensation set (range: –100%…+100%).

7.13 C.-Time 3 4.8.13

Enables user to set the time constant (in µs) of the third compensation set (range: 0…100,000 µs).

7.14 Offset 4.8.14

Enables user to set the compensation for the DC offset (in DAC units) between the GRASP and regular shim mode of the Z shim (range: –500…+500) (see Sample and Spectrometer Setup in the GRASP Operation manual).

8. N-Function 4.9

Includes several functions for the (optional) Nitrogen level measurement. For the HR-20 keyboard, access to this entire submenu requires the correct security code.

8.1 N-Level 4.9.1

Measures the liquid nitrogen level (in %). This optional function is available only on BOSS keyboards. The HR-20 keyboard provides the [***N-LEVEL***] key instead of this menu function (see [***N-LEVEL***] on page 21).

8.2 Fill 4.9.2

*****security code required**

Measures the liquid nitrogen level on line during the fill-up procedure (measurement interval: 0.25 s)

8.3 0% 4.9.3

*****security code required**

Calibrates the nitrogen level measurement system for 0% nitrogen level (see installation of SLCB).

8.4 100% 4.9.4

*****security code required**

Calibrates the nitrogen level measurement system for 100% nitrogen level (see installation of SLCB).

8.5 Voltage 4.9.5

*****security code required**

Displays the nitrogen level sensor measurement voltage (range: 0…–8 V, resolution:10 mV).

9. He-Function 4.10

*****security code required**

Includes several functions for adjusting and configuring the He level measurement system.

9.1 Fill 4.10.1

*****security code required**

Measures He level on line during the fill-up procedure. Note that this measurement indicates tendency only (accuracy: +/–10%). The fill measurement current is 1.25 times the measurement current, and the measurement interval is 7 s.

9.2 0% 4.10.2

*****security code required**

Calibrates He-level measurement system for 0% He level (see SLCB installation).

9.3 100% 4.10.3

*****security code required**

Calibrates He-level measurement system for 100% He level (see SLCB installation).

9.4 De-Ice 4.10.4

*****security code required**

De-ices the He-level sensor (de-ice current: 200 mA).

9.5 Meas.Curr. 4.10.5

*****security code required**

Sets the He-level sensor measurement current (default: 100 mA).

9.6 Alarm-Level 4.10.6

*****security code required**

Enables service engineer to set the He level below which the buzzer sounds and an error message appears on the display (see magnet manual, default value: 100%). This supervisor function is active during an automatic He-level measurement, but not a manual He-level measurement (see also [***HE MEAS.***] on page 16).

10. Shim-Ranges 4.11

*****security code required**

All shim current sources have a range switch which enables the service engineer to select the actual current corresponding to the current source value. A few of these range switches can be controlled from the BSMS keyboard; however, most must be varied by a hardware change to the appropriate SCB (for more information see Shim Manual). Note that for the HR-20 keyboard, the shim ranges are labeled ***'RangeZ…XYZ, X^3, Y^3'***.

Range 1…39 4.11.1

*****security code required**

Enables service engineer to select the shim current source number whose range is to be changed or read. For those ranges that can be changed from the keyboard, the values 0, 1, 2, and 3 may be chosen. Those that cannot be changed from the keyboard have possible values 0 and 1. The value presently selected can be read from the keyboard.

11. Shim-Current 4.12

*****security code required**

Enables service engineer to measure all shim currents. Note that for the HR-20 keyboard, the shim current sources are labeled ***'Curr.Z**…**XYZ, X^3, Y^3'***.

Curr. 1…39 4.12.1

*****security code required**

Displays the actual current of the selected current source (resolution: 1 mA, accuracy: 10 mA!).

Chapter

Sample Positioning 5

Introduction 5.1

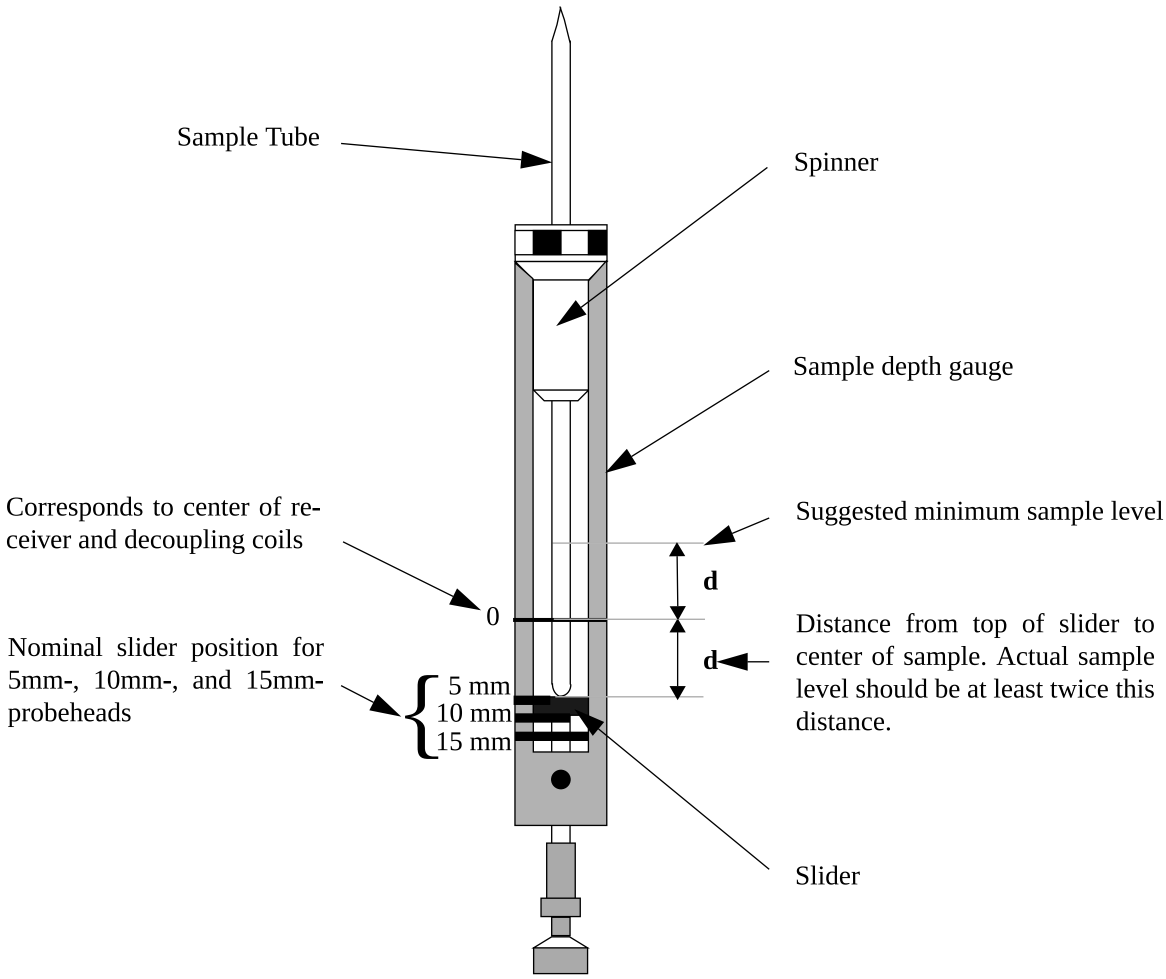
The following is intended to be a practical guide for adjusting sample position.

When the sample tube, held by the spinner, is inserted into the magnet, it is lowered gently until the spinner lands in the turbine. The sample tube then extends below the spinner and turbine, towards the most homogeneous region of the magnetic field. Similarly, once in the magnet, the probehead extends up from the bottom of the magnet so that its receiver coil and decoupling coils are centered with respect to this homogeneous region. In order for the sample itself to be positioned optimally with respect to the coils of the probehead, the sample tube position in the spinner must be carefully adjusted. Although coil sizes vary from probehead to probehead, the distance from the spinner to the coil centers is the same for each probehead. Thus, provided all sample tubes are filled to the same level, it is possible to use the same sample tube/spinner position for all samples and probeheads designed for a given sample tube diameter.

Adjustment Guide 5.2

A sample depth gauge is provided to assist the user in correctly positioning the sample tube with respect to the spinner. Its use is explained in Figure 7 on page 42. Note that if the sample tube does not extend far enough below the spinner, most of the sample will remain above the probehead coils where it cannot be detected in the NMR experiment. On the other hand, if the sample tube extends too far below the spinner, the tube bottom may touch the probehead insert and so interfere with sample spinning.

Figure 7: Sample Tube Depth Adjustment



•Adjust the slider to the heavy black line corresponding to the sample tube diameter ***as indicated on the probehead label***.

•Seat the spinner on top of the depth gauge as shown.

•Carefully push the sample tube through the spinner until the bottom just touches the top of the slider.

•For example:

Probehead label: ∅ = 5 mm.
Slider position: approx. at the 5 mm label.
(**d** should be at least 15 mm and is typically 20 mm).

Shim Operation 6

Introduction 6.1

The following is intended to be a practical guide for adjusting the room temperature shim system (BOSS). The purpose of shimming is to maximize the magnetic field homogeneity, which depends somewhat on probehead and sample geometry. In general, it is necessary to shim the magnetic field after each probehead change, sample change, and occasionally between changes to correct for any system drifts.

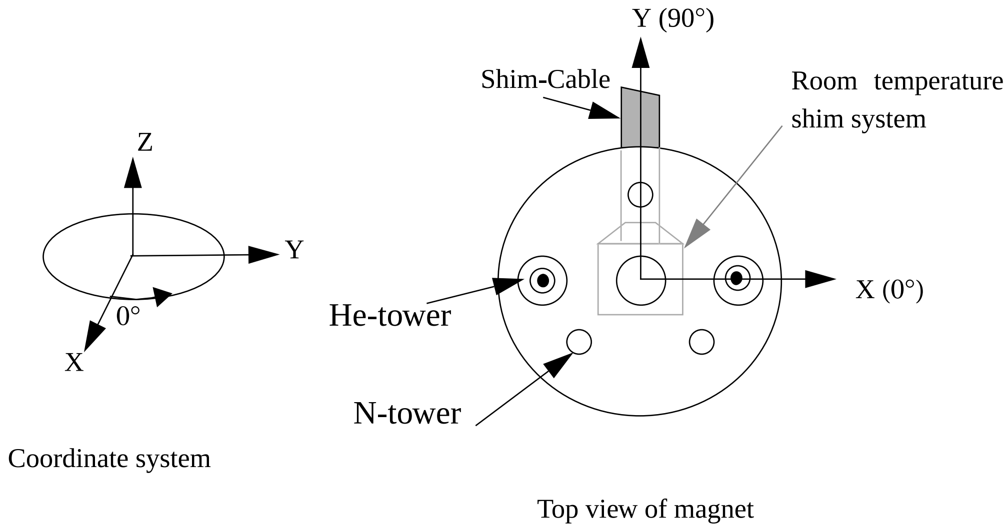
Optimal shim settings may vary substantially from probehead to probehead; however, provided the probehead is always positioned the same in the magnet and the sample is always positioned the same with respect to the receiver coil, the shim values for a given probehead will be fairly reproducible. Thus, shimming time can be greatly reduced if the shim settings for each probehead are stored as a shim file on the computer (see UXNMR Manual, read/write shim values). When the probehead is changed, the shim file for the new probehead can be read in and then final adjustments can be made to these shim values to correct for system drifts, and to account for the geometry of the particular sample being used.

The BOSS shim system consists of a number of shim coils arranged in the room temperature bore of the magnet. During shimming, the currents in these shim coils are adjusted so that the small magnetic field gradients produced cancel the residual inhomogeneity of the main magnetic field (H0) as completely as possible. Other shim systems have different configurations, so it may be possible to select a shim on the BSMS keyboard which is not supported by your particular shim system (see BSMS Shim Manual, BOSS). When this happens, a beep sounds. The shim currents can be controlled either by the BSMS keyboard or by the computer.

Basic shimming is performed while observing the lock signal (either ^{2}H or ^{19}F) on the computer screen. Either the ringing resonance observed during the field sweep, or the actual locked signal can be monitored. In the first case, each shim is adjusted in the direction of increasing signal amplitude and decay time. In the second, shims are adjusted simply to increase signal amplitude (lock level). Finest shimming is accomplished while observing the ^{1}H FID and its transformed spectrum.

The field axes used to designate the different shim coils are defined such that the Z direction lies along the sample tube axis (i.e., along H0). X and Y lie in the transverse plane with Y oriented along the shim cable as shown in Figure 8 on page 44.

Figure 8: Orientation of the Room Temperature Shim System



There are two categories of shims: radial and axial (or onaxis). Shims with names containing the letters “X” or “Y” are called radial shims. The gradients produced by radial shims are averaged by sample spinning, so these shims are adjusted without sample rotation. Radial shims affect the observed lineshape when the sample is not spinning, and the size of spinning sidebands when it is.

The pure Z shims (e.g., Z, Z^2, Z^3, Z^4…) are the axial shims. The gradients produced by axial shims are not averaged by sample spinning, so axial shims affect the observed lineshape whether or not the sample is spinning.

Shimming on the Lock Signal 6.2

When the spectrometer is locked (see Lock Operation on page 49), the vertical offset of the lock trace on the graphics display corresponds to the amplitude of the lock substance signal, assuming constant lock DC, gain, and power levels. The lock level, then, serves as useful guide for basic shim adjustment. The goal in shimming on the lock signal is to adjust the shims so that the lock trace appears as high on the graphics display as possible. This lock level corresponds to the highest possible lock substance signal amplitude.

Shimming on the FID (Free Induction Decay) 6.3

Optimizing Resolution and Lineshape 6.3.1

To obtain the highest possible resolution, additional fine adjustment of the shims is carried out while observing the ^{1}H FID. This is particularly important for the final adjustments of the Z and Z^2 shims. These corrections are most easily done on a sample that gives a single line in the NMR spectrum. Examples include chloroform, TMS, or benzene (the “lineshape” sample consists of 10% CHCL3 and 1%TMS). With experience, however, shimming on other samples (such as the ortho-dichloro benzene test sample) is also possible.

The shape of the FID, and especially the beginning of the FID, indicates the shape of the transformed signal line, while the length of the FID tail is important to the overall resolution. For good line shape and high resolution, the shim controls must be adjusted so that the FID envelope is truly exponential with the longest possible decay time. The decay time limits the resolution in the transformed spectrum.

Minimizing of Spinning Sidebands (SSB) 6.3.2

Occasionally, one sees modulations on the FID envelope that, upon Fourier transformation, produce spinning sidebands (SSB) in the NMR spectrum. These undesirable SSB can often be reduced by shimming on the FID, especially when the spectrum has a strong single line. If SSB are present, the FID appears to be amplitude modulated with the spin rotation frequency. Thus, any FID modulation due to SSB will change with the spinning rate. The Fourier transformed spectrum contains sidebands symmetrically placed about the parent spectral peak, separated from the parent peak by the spinning frequency and integral multiples of it. Increasing the spinning rate will increase this separation and also decrease the amplitude of the sidebands. In general, SSB can be reduced by adjusting the radial shims with the following shim procedure (see Adjusting the Radial (X/Y) Shims (No Sample Rotation)).

Adjusting the Radial (X/Y) Shims (No Sample Rotation) 6.4

Adjust the radial shims in the following order (see How to Obtain the Optimum Shim Settings on page 46). Note that although Z and Z^2 are not radial shims, it is necessary for them to be set correctly before adjusting the radial shims.

1. Stop the sample rotation if necessary. The system must be locked (lock on).
2. Z
3. Z^2, Z
4. X, Y, Z
5. XZ, YZ, X, Y
6. XZ^2, YZ^2, XZ, YZ, X, Y
7. $(X^2–Y^2)$, XY, X, Y

Repeat steps Figure 2 through Figure 7 several times until no further increase in the lock line position is observed. Higher order shims (XZ^3, YZ^3,…) may be adjusted slightly. After adjusting the radial shims, check the onaxis shims.

Adjusting the Onaxis (Z) Shims (with Sample Rotation) 6.5

Adjust the onaxis shims in the following order (see How to Obtain the Optimum Shim Settings on page 46).

1. Set the spinrate to ~25 and start sample rotation. The system must be locked (lock on).
2. Z
3. Z^2, Z
4. Z^3, Z^2, Z
5. Z^4, Z^3, Z^2, Z
6. Z^5, Z^4, Z^3, Z^2, Z

Repeat steps Figure 2 through Figure 6 several times until no further increase in the Lock line position is observed. The transformed ^{1}H spectrum can be used to determine better which onaxis shim must be adjusted. Note that when sample rotation is stopped, the lock level should not drop significantly. If it does, then it is necessary to re-adjust the radial shims.

How to Obtain the Optimum Shim Settings 6.6

In general, it is possible to optimize the radial shims by adjusting each one individually to maximize the field homogeneity. Once all the radial shims have been adjusted, it is possible to correct for any inter-shim interactions by re-adjusting each radial shim individually, as indicated above in Adjusting the Radial (X/Y) Shims (No Sample Rotation).

On the other hand, the axial shims are best optimized in an iterative manner. For example, first Z and Z^2 may be adjusted as follows:

1. Adjust Z to obtain the highest lock level.
2. Note the present value of Z and Z^2. Change the latter value by a small amount. The lock level may change.
3. Return to Z and re-adjust this to maximize the lock level. If this new maximum level is higher than the maximum in step Figure 1 , continue changing Z^2 in this direction and maximizing the lock level by re-adjusting Z. If the new maximum is lower than the maximum in step Figure 1 , reset Z and Z^2 to their values in step Figure 2 , and try changing Z^2 in the opposite direction.
4. Continue this iterative procedure until the maximum lock level is found.

Once Z and Z^2 have been adjusted, Z^3, Z^4, Z^5,… are added to the loop one at a time. For example, to add Z^3 to the loop, note the values of Z, Z^2, and Z^3. Change Z^3 a small amount in one direction and then maximize the lock level using Z^2 and Z. If this new maximum is an overall improvement, continue changing Z^3 in this direction and maximizing the lock level with Z^2 and Z. If it is not, reset Z, Z^2, and Z^3 and try changing Z^3 in the opposite direction. Once Z, Z^2, and Z^3 have been adjusted successfully to obtain the highest lock level possible, Z^4 can be added to the loop, and so forth.

Between these steps it is very useful to analyze the transformed ^{1}H spectrum. The lineshape will give an indication as to which onaxis shim must be adjusted, as shown in Figure 9 .

Figure 9: Influence of the Onaxis Shims on the Spectral Lineshape

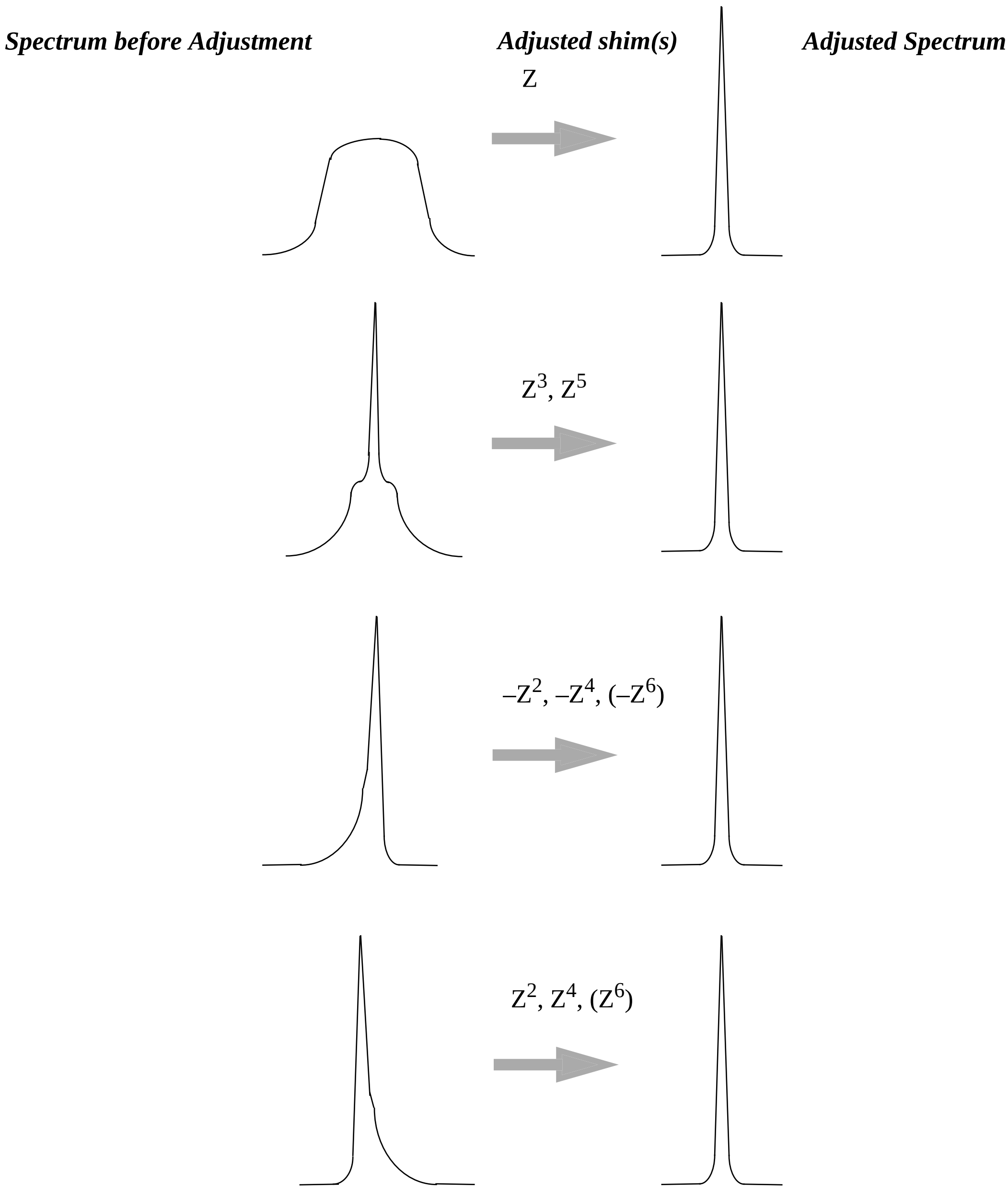
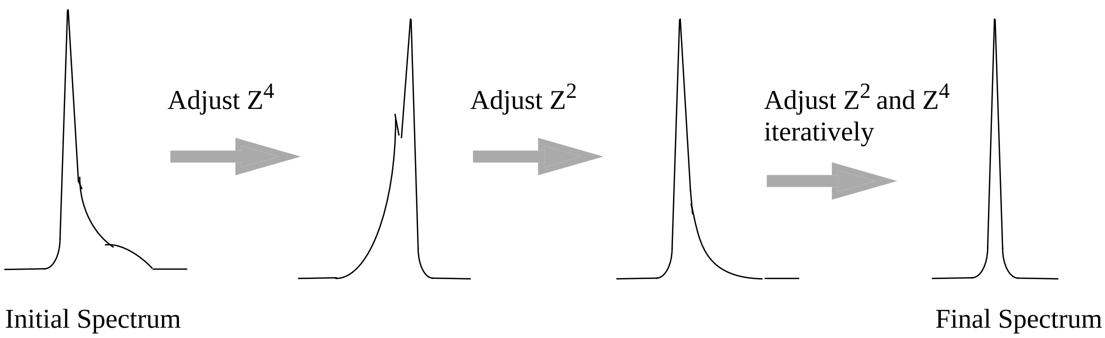


Figure 10: Example of an Onaxis Shim Adjustment



When to Re-Shim 6.7

Sample changes, probehead changes, drift of the magnet or environmental changes will make it necessary to re-shim the system.

Table 2. Shim Adjustments Suggested Following a System Change

Action	Shims to adjust
Sample change	Z, Z^2
Probehead change	onaxis and radial
Environment change	onaxis and radial
Periodical (each two weeks) when no change occurs	onaxis or Z, Z^2

Lock Operation

Manual Lock-In 7.1

The first step in manually locking on a solvent when the correct field value is not known is to search for the lock signal. One approach to finding the lock signal is to set the sweep amplitude to the maximum (100), increase the lock power (e.g., to 0 dBm), and increase the lock gain (e.g., to 120 dB). The lock DC should be set to approximately –75 and the sweep rate to 0.2 Hz. Adjust the field value until the lock signal is approximately centered on the screen, and then begin to reduce the sweep amplitude. If the signal disappears from the screen during this process, it may be brought back by re-adjusting the field value. Eventually, the lock signal should be centered on the screen with the sweep amplitude reduced to a value in the range of 2 to 5. The lock power and gain should also be reduced to a level suitable to the particular solvent. Finally, the lock phase must be adjusted. The phase is optimized when the amplitude of the sweep wiggles is the same for both directions of the field sweep. If the wiggles in one direction are larger than those in the other, adjust the lock phase to correct the imbalance. Having the correct phase is important to achieving lock-in.

Caution: Sidebands

It may be difficult, especially if the lock signal is very narrow, to observe the lock signal when the sweep amplitude is fully open, despite the high power and gain settings suggested above. If this is the case, reduce the sweep amplitude. However, be warned that before locking in on an unfamiliar solvent, it is important to verify that the lock signal observed is the parent signal and not a sideband. Although it is possible to lock on a sideband, the poorer signal-to-noise ratio of the sideband will result in a poorer overall lock performance. One way to verify that the lock signal is not a sideband, once the lock signal is centered on the screen, is to set the field value to +/–5300 units (for a standard bore magnet, more for a wide bore magnet). After changing the field value it is necessary to wait a few seconds as the actual magnetic field follows slowly (due to eddy-current effects). If the original signal was indeed the parent signal, the signal observed now is a sideband and has a much lower signal amplitude. Be sure to lock on the signal with the highest amplitude.

A second caution is that optimum lock performance will only be achieved if the lock power level is set somewhat below saturation (as described below). Thus, when using lock solvents which saturate easily (e.g., Acetone-d6), the lock power should be set rather low, ideally around –20 dBm.

Once the sweep wiggles of the parent signal are centered on the screen, have the correct phase, and are at least 1/3 the height of the screen, lock-in may be started by pressing the [***LOCK ON/OFF***] key. If the wiggles are too small adjust the lock gain to compensate. A strong regulator is used for the first moment of lock-in to establish the correct field value. If lock-in is successful, a second regulator then automatically takes over, the [***LOCK ON/OFF***] LED stops blinking, and the lock power is reduced. This second regulator uses the parameters (described below) set from the BSMS keyboard or computer.

Once lock-in is achieved, the overall lock results can be improved by adjusting the lock phase to produce the maximum signal amplitude.

Optimal Operation with the Digital Lock 7.2

One advantage of the digital lock system provided by the BSMS is that the user is no longer restricted to adjusting the field value to find the lock signal. It is now also possible to adjust the actual frequency of the lock channel. This is advantageous because it allows very nearly the same magnetic field (H0) value to be used for all lock solvents. When the same H0 value is used, the absolute frequency of the reference (e.g., TMS) signal remains approximately the same, regardless of the solvent, and thus spectral referencing is no longer solvent dependent. In addition, if the absolute frequency of the TMS signal no longer varies from sample to sample, it now makes sense to define the offset frequencies of the observe and decouple channels in terms of ppm rather than Hz. This is helpful to the chemist who is used to thinking of chemical shifts in terms of ppm and not Hz, and who would know the offset frequencies in ppm appropriate for a particular sample. From the BSMS keyboard itself it is possible to adjust the frequency of the lock channel by first placing the keyboard in shift mode (see ***'2.3 Shift/Field'*** on page 25), pressing the [***LOCK SHIFT***] key, and then selecting the frequency (in ppm) with the control knob.

A second advantage of the digital lock is that it allows the user to optimize the second regulator used to control H0 once lock-in has been achieved. Currently, there are two lock parameters (loop gain and loop time) available in the menu mode of the BSMS keyboard, which enable the user to control the behavior of this regulator. The following briefly describes how to set these lock parameters, in addition to the standard lock parameters, for the best lock results.

During shimming, these lock parameters are not terribly important. It is important, however, to set the lock power approximately 6 to 10 dB under saturation and to optimize the lock phase.

During critical NMR experiments (e.g., difference experiments), it is very important to have good shim values and optimal lock parameters to ensure good field stability. The most important indicator of an optimal lock parameter set is a high signal-to-noise ratio of the lock signal. To achieve this, first the lock power should be set as high as possible and yet not so high as to cause saturation. Increase the lock power in small steps and observe the lock line on the screen. The lock level should increase steadily in response to the increase in power level; when it no longer increases, or even begins to decrease, saturation has been reached. Depending on the lock solvent, this may happen rather quickly (e.g., at approximately –30 dBm for Acetone). The optimum lock power level is a few dB below saturation.

It is also important to choose the best lock receiver gain (lock gain). In general, if the lock DC is set appropriately (i.e., at approximately –75) it is sufficient to set the lock gain so that the lock line is in the upper part of the screen. The goal here is to best use the ranges of the A/D converter and the signal processor. This occurs when the lock gain is set as high as possible without causing receiver gain overflow, which can be recognized by the presence of a very noisy lock signal, and a decrease in lock level with a further increase in lock gain.

Finally, the regulator should be optimized using loop gain and loop time (see ***'2.1 Loop Gain'*** and ***'2.2 Loop Time'*** on page 25). A large (i.e., less negative) loop gain value enables a better field disturbance compensation, which is what is desired.

However, if the signal-to-noise ratio of the lock signal is not sufficient, too high a loop gain causes the H0 field to be noise modulated. When this occurs, the lock line oscillates visibly on the screen. Of course, this noise modulation then shows up in the NMR spectrum, which is highly undesirable. Thus, a useful rule of thumb is that the better the signal-to-noise ratio of the lock signal is, the higher the loop gain may be set. A typical loop gain setting is –35 dB. For optimum regulator performance, though, the loop gain cannot be set independently of the loop time. A typical loop time value is 0.05 s and, in general, longer loop times are necessary for lock signals with poorer signal-to-noise ratios.

Lock settings appropriate for various conditions are listed below in Table 3, Table 4, and Table 5. The settings shown in Table 3 are appropriate for a lock signal with quite a high signal-to-noise ratio, those in Table 4 are appropriate for a lock signal with a fairly poor signal-to-noise ratio, and those in Table 5 cause the regulator to behave the same as that of the old analog lock system.

One final comment is in order. If two different lock solvents yield lock signals having the same screen line position (lock level) but with a different lock gain and power setting used for each, then the system signal-to-noise ratio varies inversely with respect to the lock gain. For example, if one solvent requires 10 dB more gain than the other to achieve the same signal level, the corresponding signal-to-noise ratio is l0 dB less than that for the other solvent.

Table 3. Lock Settings for a Hump Test

Lock settings with a very high loop gain and good signal-to-noise ratio (AMX600)			
Lock Power	–30	dBm	Saturation
Lock Gain	90	dB	
Loop Gain	–10	dB	
Loop Time	0.004	s	

Table 4. Lock Settings for a 2D Experiment with H_2O Suppression

2mMol Lysozyme in 90% H_2O/ 10%D_2O (AMX600)			
Lock Power	–10	dBm	Saturation
Lock Gain	108.6	dB	
Loop Gain	–40	dB	
Loop Time	0.13	s	

Table 5. Default Lock Settings

Corresponding to the old analog lock		
Loop Gain	–32	dB
Loop Time	0.136	s

Drift Calibration Procedure 7.3

The [***DRIFT***] function enables compensation of the magnetic field drift (in field units per day) during a long term measurement performed without lock. In order for [***DRIFT***] to provide the correct compensation, it is necessary to calibrate the magnetic field drift as follows:

1. Set the drift to zero ([***2nd***], [***DRIFT***], and choose 0 with the control knob).
2. Insert a sample with a strong lock signal.
3. Switch lock off ([***LOCK ON/OFF***]).
4. Switch sweep on ([***SWEEP***]).
5. Adjust the H0 field value until the sweep wiggles are centered on the screen ([***FIELD***]).
6. Press [***STD BY***] and wait for 24 hours without any action on spectrometer.
7. After 24 hours, enter diff-mode ([***DIFF.MODE***]) and select [***FIELD***].
8. Adjust the field value to return the sweep wiggles to the center of the screen as in step 5.. Notice the Δ value displayed on the right-hand side of the display.
9. Select drift ([***2nd***], [***DRIFT***]) and set this parameter to the Δ value found in step 8. with the same polarity. This completes the drift adjustment and further corrections are usually not necessary.
10. To save the drift value, first select the menu on the keyboard ([***2nd***] and [***Y^3***]).
11. Enter the security code (***‘4. Service’***, [***ENTER***], ***‘4.1 Sec.-Code’***, [***ENTER***], enter the code with control knob and [***ENTER***], a beep sounds if the code is correct, [***ESC***], and you are now in the submenu ***‘4. Service’***).
12. Save the drift by saving the BSMS configuration (***‘4. Service’***, [***ENTER***], ***‘4.2 Save Config’***, [***ENTER***], you hear a beep and the message ‘***Done***’ appears).
13. Leave the menu ([***ESC***], [***ESC***], ‘***Standby***’).

Once [***DRIFT***] has been set to a non-zero value, magnetic field drift compensation occurs when both lock and sweep are off.

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