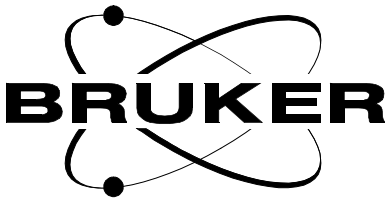

MAS Pneumatic Control Unit

User Manual

Version 003

BRUKER

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Contents

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Introduction 1

Introduction 1.1

This manual is intended to assist operators who have recently acquired a Pneumatic Unit (P/N H2620). Having read the manual the operator should

- be able to install the unit
- understand the basic functions of the hardware
- be able to use a set of basic software commands or keypad functions to operate the unit.

This manual is **not** intended to describe

- solid NMR or specifically MAS applications
- sample preparation
- MAS data analysis
- variable temperature work
- MAS probes

Manual Layout 1.2

The following section is intended to help the reader make the most use of this manual.

Chapter 2 describes the facilities that need to be installed in the laboratory prior to installation.

Chapter 3 describes the actual installation of the unit. If the installation has not yet been completed you are encouraged to read Chapter 4 and in particular Chapter 6 **prior** to installation as these describe the relevant hardware.

Chapter 4 describes the unit hardware and is essential reading if the operator is to fully understand how the unit operates and gain the maximum benefit from its use.

Chapter 5 describes the front panel LED and LCD displays. This information is required if the unit is to be operated in the Local mode. If the unit is to be operated in the Remote mode (i.e XWIN-NMR control) then this chapter is less important.

Chapter 6 describes the connections at the rear panel. If the unit is already installed then this chapter is less important. If any non standard configurations are to be contemplated then this chapter is essential.

Chapter 7 is the first Chapter to deal with the actual operation of the unit.There are 4 modes of operation and this chapter is essential if the operator is to choose the most appropriate.

Chapter 8 is for users opting to operate in the Local Manual Mode.

Chapter 9 is for users opting to operate in the Local Automatic Mode.

Chapter 10 is for users opting to operate in the Remote Manual Mode or the Remote Automatic Mode.

Finally Chapter 11 is a list of the principal specifications of the system.

Other Useful Manuals on the Subject of MAS *1.3*

While this manual concentrates on describing the operation of the MAS PU from a users perspective other manuals which contain useful information are listed below.

SB-MAS Operation Manual	P/N Z31401
High Resolution Magic Angle Spinning Spectroscopy	P/N B2265
AVANCE DMX/DSX Spectrometers, Solids Experiments User Manual	P/N H9321
High Resolution MAS Probes	P/N Z31391

Facility Requirements 2

Main Air Supply 2.1

Particulates: Oil Free Air with particle size no greater than 0.01 microns.

Dew Point: < -30°C.

Flow rate: This will depend to some extent on spin rates etc. but typically 4 m^3 / hr. (2.35 cu.ft./min.) flowing at greater than 6 bar (87 psi) will suffice.

Input pressure: Max.: 10 bar (145 psi)

Min.: 4 bar (58 psi)

Ideal: 6-8 bar (87-116 psi)

Control of Sample Temperature 2.2

The control of the sample temperature requires a BVT 2000 variable temperature unit and a 25l (0.88 cu.ft) liquid Nitrogen dewar. This is standard NMR equipment and not specific to MAS.

Nitrogen Supply for Low Temperature Work 2.3

For low temperature work the most common arrangement is to use a pressurized liquid Nitrogen tank with a Nitrogen boil-off device. A 60l (2.11 cu.ft.) gas cylinder (200 bar / 2900 psi) will typically last for 1 hour.

The various configurations for low temperature work are dealt with extensively in the manual ‘Avance DMX/DSX Spectrometers, Solids Experiments User Manual’ P/N H9321.

Ambient Laboratory Environment 2.4

No special requirements in respect of temperature and humidity are required.The PU will operate under standard laboratory conditions suitable for the NMR spectrometer.

Electrical Supply *2.5*

a.c. 230V 50/60 Hz single phase.

Maintenance *2.6*

The unit requires no special maintenance procedures. A well-controlled temperature and humidity environment in the laboratory will extend the life cycle of all parts. The quality of the Main Air into the rear of the PU is particularly important in preventing problems arising from impurities / particles etc.

Installation 3

Shipping Checklist 3.1

MAS Unit	H2620
Power cable	66384
AIR IN Tubing	10205
Trigger Cable	HZ 10022
User Manual	Z31047

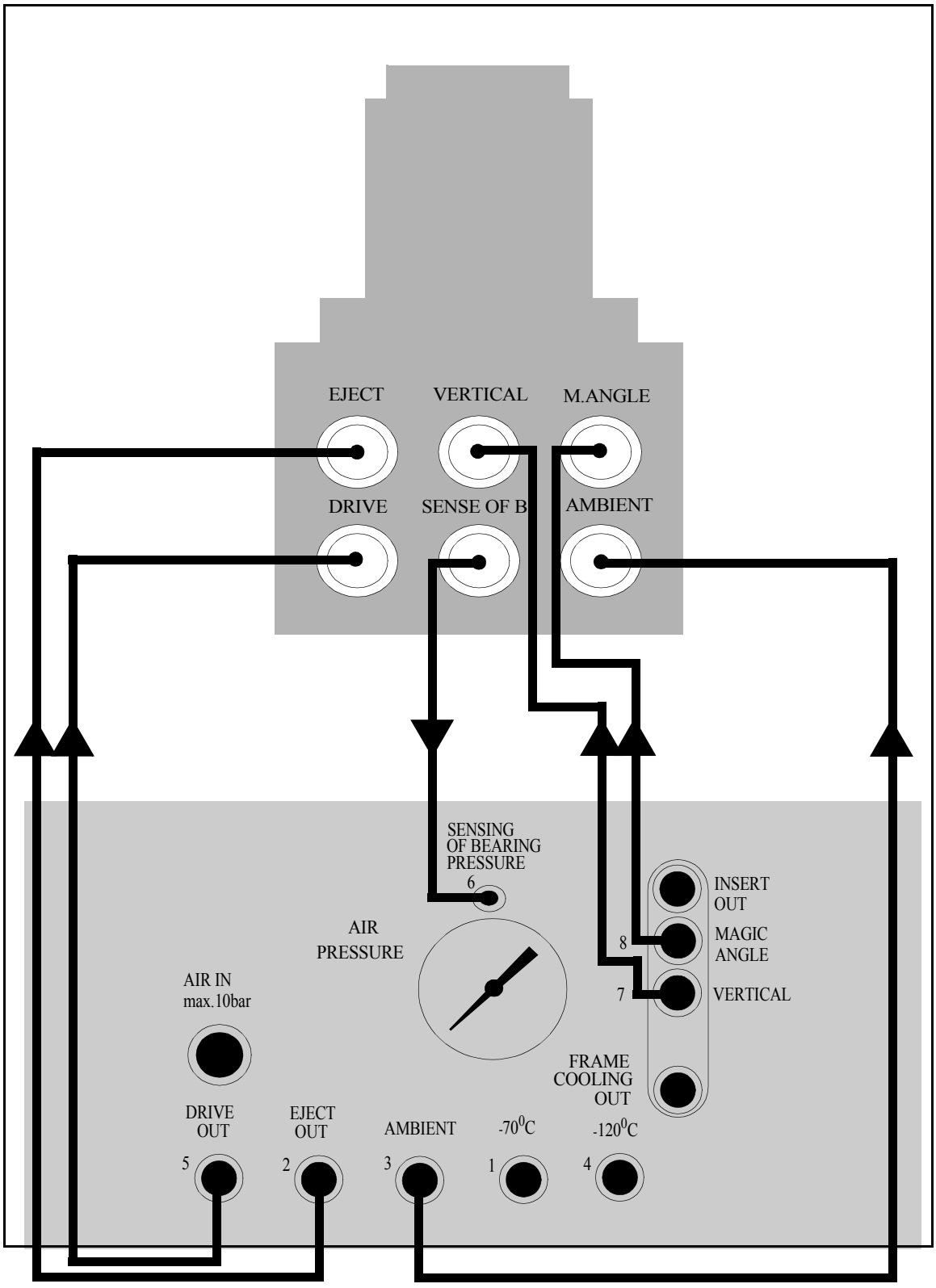
Useful Cable Part Numbers 3.2

RS232	HZ04055
Spin Cable	Z4035
Trigger to TCU	HZ10022

Procedure 3.3

Follow the procedure outlined below but DO NOT SWITCH THE UNIT ON at the mains until all other connections are complete. For an explanation of the unit hardware which might be helpful for the installation you should first refer to chapter 4 ***"Hardware: General Introduction and Functions"*** as well as chapter 6 ***"Back Panel Connections"***.

Figure 3.1. Pneumatic connections for ambient temperature work with SB magnets.



For WB magnets the MAGIC ANGLE and VERTICAL connections are not required.

Pneumatic Connections for Ambient Temperature Work *3.4*

The figure on ***page 10*** shows the pneumatic connections that need to be made for ambient temperature work.

Make the pneumatic connections between the probe and the rear of PU before attaching the AIR IN

The air hoses are labelled using numbers that corresponds to the numbering used on the rear panel of the PU.

Note that the outputs MAGIC ANGLE and VERTICAL are required for standard bore magnets only. Wide bore magnets do not need these connections.

Finally attach the plastic tubing to the 'AIR IN max 10Bar' input of the rear panel. The pressure of the input should be between 6-10 Bar. The gauge at the rear of the panel should display the value to which the pressure is regulated inside the PU and so should remain constant at about 5.5 Bar even when the 'AIR IN' pressure is above this pressure. The 'Insert Out' connection is not required for SB magnets but is required for WB.

Electrical Connections *3.5*

If the PU is to be operated remotely via XWIN-NMR connect the RS232 port at the rear of the PU with the selected port on the CCU. The number of the selected TTY port on the CCU will be entered during the **cfmas** routine. TTY07 is standard but this can be defined by the user. If the PU is operated locally then this connection is not necessary.

Attach the TRIG output of the rear panel to the TRIG input (0 or 1) on the front panel of the TCU. This connection is only used if synchronization of the phase of the rotation with specific NMR pulse sequences is required.

Attach the SPIN rate cable to the base of the probe ensuring that the direction is correct according to the labels.

Temperature and Heater Connections *3.6*

Cooling of the probe assembly or frame is achieved via the Frame Cooling output of the PU rear panel which is attached to the base of the probe

For sample temperature control connect the heater cable and thermocouple coming from the rear of the VT unit to the base of probe.

Sample temperature is normally controlled using the Bearing gas output of the PU rear panel which is attached to the probe via the Ambient connection.

Three Bearing Gas outputs are available. The PU is programmed so that only one of these outputs can ever be physically opened at any one time.

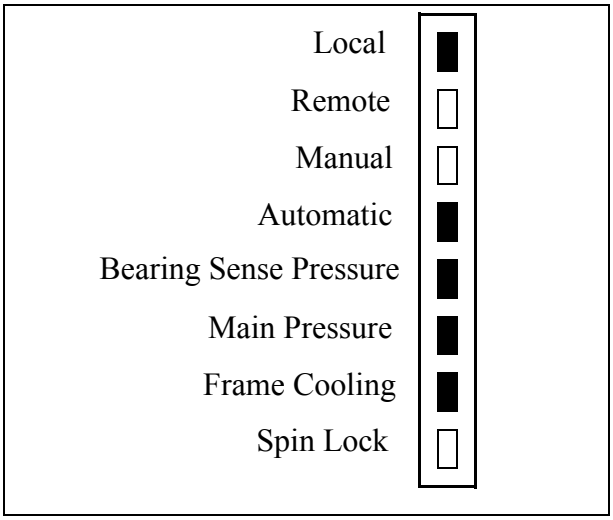
The 'Ambient' output is now standard and is normally connected directly to the probe base as in the figure on ***page 10***. However for low temperature work the Ambient output may be connected first to a heat exchanger and then applied to the probe base.

The other two outputs (-70°C,-120°C) are supplied to enable connections to be made with older models of heat exchangers.

Default Front Panel Display *3.7*

You can now switch the unit on. The front panel display should now be as the figure below:

Figure 3.2. Default Front Panel LED display after power on.



The unit always defaults to the Automatic mode, either Remote of Local depending upon the status stored at power off.

Adjustments to Eject Pressure *3.8*

This adjustment should only ever be necessary whenever the unit is first installed or a probe is changed. The adjustment can be made using a pneumatic outlet at the rear of the back panel labelled '**Eject Adjust'**

Turn the screw clockwise to reduce the pressure and anti clockwise to increase. A reason for adjusting the eject pressure might be if the pressure was insufficient to eject the rotor or if the rotor was removed too aggressively from the transfer system and in danger of being damaged against the top of the transfer system.

Hardware: General Introduction and Functions

4

Principle Functions 4.1

The principle functions of the MAS PU are to

- insert, eject and rotate NMR samples contained in specially designed rotors
- enable the sample stator to be tilted to the desired magic angle
- control the spin rate of the rotor and adjust pressure accordingly
- supply air to cool the probe assembly via the frame cooling.

While the principal functions are pneumatic there are also electronic signals such as control from XWIN-NMR via the CCU and also spin rate control. These features, as well as the principal pneumatic functions will be explained in the following sections.

Description of Rotor and Stator 4.2

Rotor: The sample is contained is a special tube which rotates at high speed and hence the name. The diameter of the rotor will be determined by the probe diameter and can currently be 2.5mm, 4mm or 7 mm see ***"Markings at base of rotors" on page 16***.

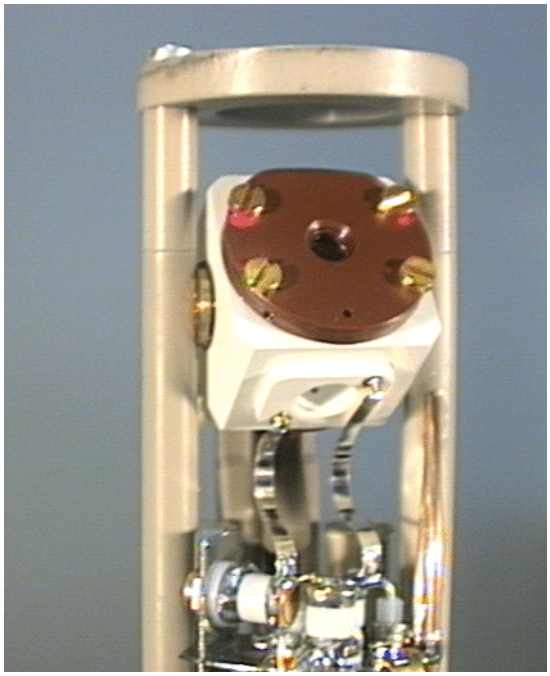
Stator: The rotor is spun inside a specially designed housing called a stator located at the top of the probe. This housing remains static during the rotation and hence the name. Pressure streams know as the Drive Pressure (DP) and Bearing Pressure (BP) are directed onto the rotor by means of the stator.

Two types of rotor material are normally used.

Silicon Nitride (grey in color)

Zirconia (white / yellow)

Figure 4.1. MAS Stator



Pneumatic Transfer System *4.3*

The transfer system is lowered into the top of the shim stack system. This connects the top of the magnet, where the rotor is inserted, to the stator located at the upper end of the probe.

Explanation of Drive Pressure (DP), Bearing Pressure (BP), Sense of Bearing Pressure *4.4*

A Bearing pressure is used to levitate the rotor within the stator on a cushion of air. This enables practically frictionless rotation in that there is no physical contact between the rotor and the stator. The actual rotation is achieved by the application of the Drive pressure. Successful stable rotation can only be achieved with a combination of the correct Bearing and Drive pressure which is controlled by the pneumatic unit. A sudden loss of Bearing Pressure is the most dangerous scenario in terms of rotor and stator damage as the levitation would be suddenly removed and the rotor would crash against the stator while still spinning. For this reason a Sense of Bearing measurement is used to ensure that the Bearing pressure is sufficiently high at all times. The Bearing Sense pressure sensor is located at the base of the probe. If insufficient BP is detected (for example due to a leak in an air hose) then the DP, which effectively rotates the sample, will be immediately reduced.

Explanation of INSERT/EJECT and VERTICAL Commands *4.5*

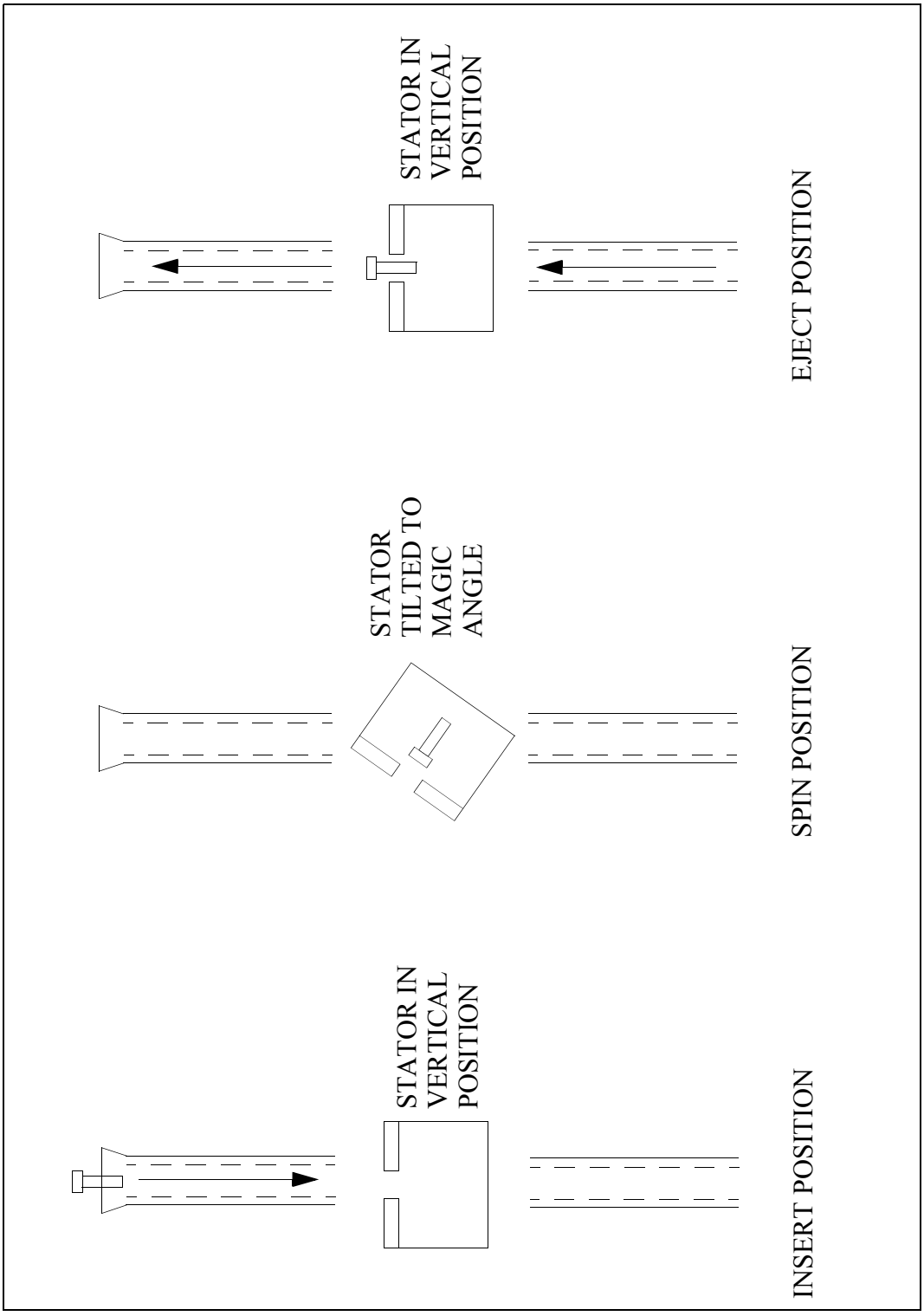


Figure 4.2. Schematic showing the relative positions of the stator for SB magnet

Note that the requirement that the stator be in the vertical position for insertion and ejection of the rotor is applicable to SB magnets only. The geometry of the probes in WB magnets renders this requirement unnecessary.

The rotor containing the sample is placed by the operator into the transfer system at the top of the magnet while the stator which holds the rotor during spinning is located some distance below at the top of the probe. The transfer system is used to transfer the rotor from the top of magnet to the stator and vice versa on a cushion of air.

The INSERT command will transfer the rotor from the top of magnet into the stator.

The EJECT command will transfer the rotor from the stator back to the top of magnet for removal by the operator.

Before a sample can be either inserted or ejected the stator must be in the vertical position as opposed to the magic angle position (this applies to standard bore magnets only!). The vertical operation is used to set the stator to the vertical position and is automatically implemented whenever the INSERT or EJECT command is entered.The EJECT command will apply the required pressure indefinitely until Stop is pressed. This is to ensure that the operator has sufficient time to remove the rotor from the top of magnet before the supporting air is removed.

Spin Rate Monitoring *4.6*

Figure 4.3. Markings at base of rotors

An infrared LED within the probe assembly emits a signal which is directed from the base of the probe to the base of the rotor by means of an optic fibre cable. A second optic fibre cable carries the reflected signal back from the base of the rotor to a detector. The frequency of the detected signal will depend on the speed of rotation and this signal is sent to the SPIN input at the PU rear panel. The PU processes the analogue voltage input which contains the frequency information and the spin rate is determined.

Front Panel LED & LCD Display, Arrow Keypad

5

Introduction 5.1

Regardless of the mode of operation an understanding of the front panel LED display is required. If the unit is to be operated in Remote (XWIN-NMR) mode then the front panel need only be referred to at initial setup.

LED Display 5.2

The front panel contains a set of 8 LEDs, the first 4 of which serve to display the current mode of operation. Four modes are possible

- LOCAL MANUAL
- LOCAL AUTOMATIC
- REMOTE MANUAL
- REMOTE AUTOMATIC

The current mode of operation can be set to all four possible modes using two keys on the main keypad namely the LOCAL / REMOTE key and the AUTO / MANUAL key.

When the unit is switched on or after a RESET the default mode is always AUTOMATIC as in ***Figure 5.1. on page 18***. Depending on the status at power off the PU will return to either LOCAL AUTOMATIC or REMOTE AUTOMATIC.

If the unit is to be operated under XWIN-NMR control then the REMOTE LED on the front panel must be on. This can be done by pressing the LOCAL / REMOTE key. Switching between REMOTE AUTOMATIC and REMOTE MANUAL can also be done from the XWIN-NMR window which is the interface used when operating in REMOTE mode.

BEARING SENSE PRESSURE: This LED indicates that the BP is sufficient to allow spinning to continue. The pressure is measured at the base of the probe and must be at least 50% of the actual BP itself.

MAIN PRESSURE: This LED indicates that the Pressure of the AIR IN at rear panel is greater than the minimum requirement of 4 Bar.

FRAME COOLING: This LED indicates that the pressure at the FRAME COOLING output is sufficiently high.

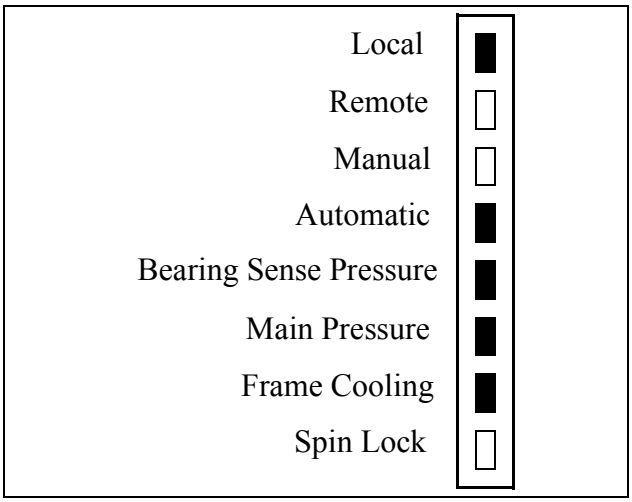


Figure 5.1. Front Panel LED display

SPIN LOCK: This LED, which operates in AUTOMATIC mode only, will light when a sufficiently stable spin rate has been detected. When the spin rate has reached the set value to within 5 Hz for a duration of greater than 5s then this LED will light. Since in MANUAL mode there is no set value for the spin rate, the Spin Lock LED has no function and will not light.

LCD Display and Arrow Keypad *5.3*

The LCD display is divided into a default Auto/Manual screen display as well as three pages called status pages 1, 2 and 3 respectively. The display enables the operator to view the status of various parameters. In conjunction with the arrow keypad the value of some of these parameters may be adjusted.

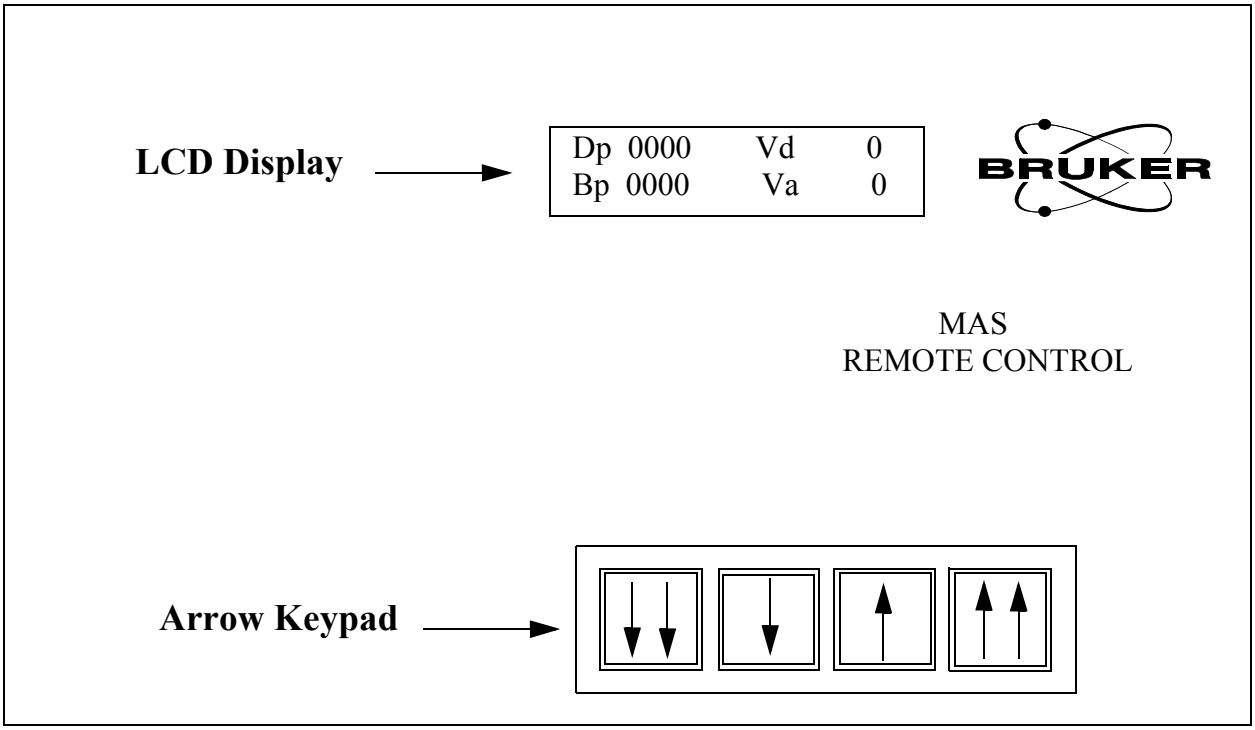
Note that status pages 2 and 3 are not normally required and should only be used for troubleshooting purposes.

Arrow Keypad *5.3.1*

The function of the arrow keypad will depend on which menu (Auto/Manual Screen Display, Status pages etc.) is current.

For standard operations the only parameters that need to be altered are the DP the BP and the spin rate. All of these are contained in the default LCD Auto/Manual Screen Display. Each of these parameters can be selected using the keypad. Once a function has been selected then the double arrows enables the operator to make coarse adjustments to an actual parameter value. The single arrows are used to make fine adjustments. All four arrows (up and down, single and double) are usable.

The functions of the arrow keys is different if the LCD menu is currently Status pages 1, 2 or 3. The double arrows enables the operator to scroll through the various status pages in either direction.The single up arrow is used to select which



parameter on a particular page is to be adjusted. The single down arrow is used to change the value of that parameter

LCD Auto Screen Display: *5.3.2*

BP: Bearing Pressure

DP: Drive Pressure

Vd: Desired spin rate

Va: Actual spin rate as measured by the optical sensor

Note: The LCD Auto Screen Display is the default screen display and in normal operation is the only display that needs to be adjusted and monitored.

Status Page 1 *5.3.3*

PHs: Probe size: Can be either 2.5, 4 or 7 mm

TYP: Type of magnet bore. Can be SB (standard bore) or WB (wide bore)

MAT: Can be ZnO (Zirconium Oxide) or SiN (Silicon Nitride).

Note: Status Page 1 is used to ensure that the PU is programmed with the appropriate hardware information. It needs only to be set once (assuming that the hardware is not changed!). The information will be stored even after a reset or power off.

Status Page 2 *5.3.4*

MA: status of valve controlling Magic Angle position. Can be either C (closed) or O (open). Open means that the stator is tilted to the Magic angle.

VE: status of valve controlling vertical positioning of stator. Can be either C (closed) or O (open). Open means that the stator is set to vertical position.

-70: status of valve controlling the -70°C Bearing pressure output at rear panel. Can be either C (closed) or O (open). Unless this output is being used it should normally be set to C.

-120: status of valve controlling the -120°C Bearing pressure output at rear panel. Can be either C (closed) or O (open). Unless this output is being used it should normally be set to C.

For all 4 valves above ‘Open’ means that the pressure is applied.

Status Page 3 *5.3.5*

IN: status of the valve controlling the Insert pressure. Can be either C (closed) or O (open).

MP: status of the valve controlling the Main pressure. Can be either C (closed) or O (open).

DP: status of the valve controlling the Drive pressure. Can be either C (closed) or O (open).

EJ: status of the valve controlling the Eject pressure. Can be either C (closed) or O (open).

For all 4 valves above ‘Open’ means that the pressure is applied.

Back Panel Connections

6

Description of Connections 6.1

Although not required to operate the unit the following sections have been included to explain the functions of the various inputs and outputs.

RS232

This is the connection to the CCU (standard port is TTY07). This connection enables remote control of the PU using XWIN-NMR. If the unit is to operated locally (either LOCAL MANUAL or LOCAL AUTOMATIC) then this connection is not required. The software needs to know which TTY port on the CCU is used and this information is given during the **cfmas** routine.

AIR IN max 10Bar

This is the main pneumatic pressure **input** to the PU and is the source of all other pressures that are used. This pressure is regulated to between 5 and 6 Bar internally inside the PU. This regulated pressure is then displayed on the circular pressure gauge of the rear-panel. Ideally the pressure applied to this input should be between 6-8 Bar. Although it is not ideal the unit may work with pressures as low as 4 Bar. As long as the pressure at the rear is greater than 4 Bar the MAIN PRESSURE LED at the front panel will light.

Sense of Bearing Pressure

This **input** serves to ensure that the pressure elevating the rotor (the BP) as detected at the probe base is sufficient. This is to ensure that above normal pressure losses do not occur along the plastic tubing connecting the PU Bearing Out connector and the probe. The sense of bearing pressure must be at least 50% (adjustable) of the Bearing Out pressure. If this is not the case then the Drive Pressure will be reduced.

Drive Out

Pressure **output** used to rotate the rotor.

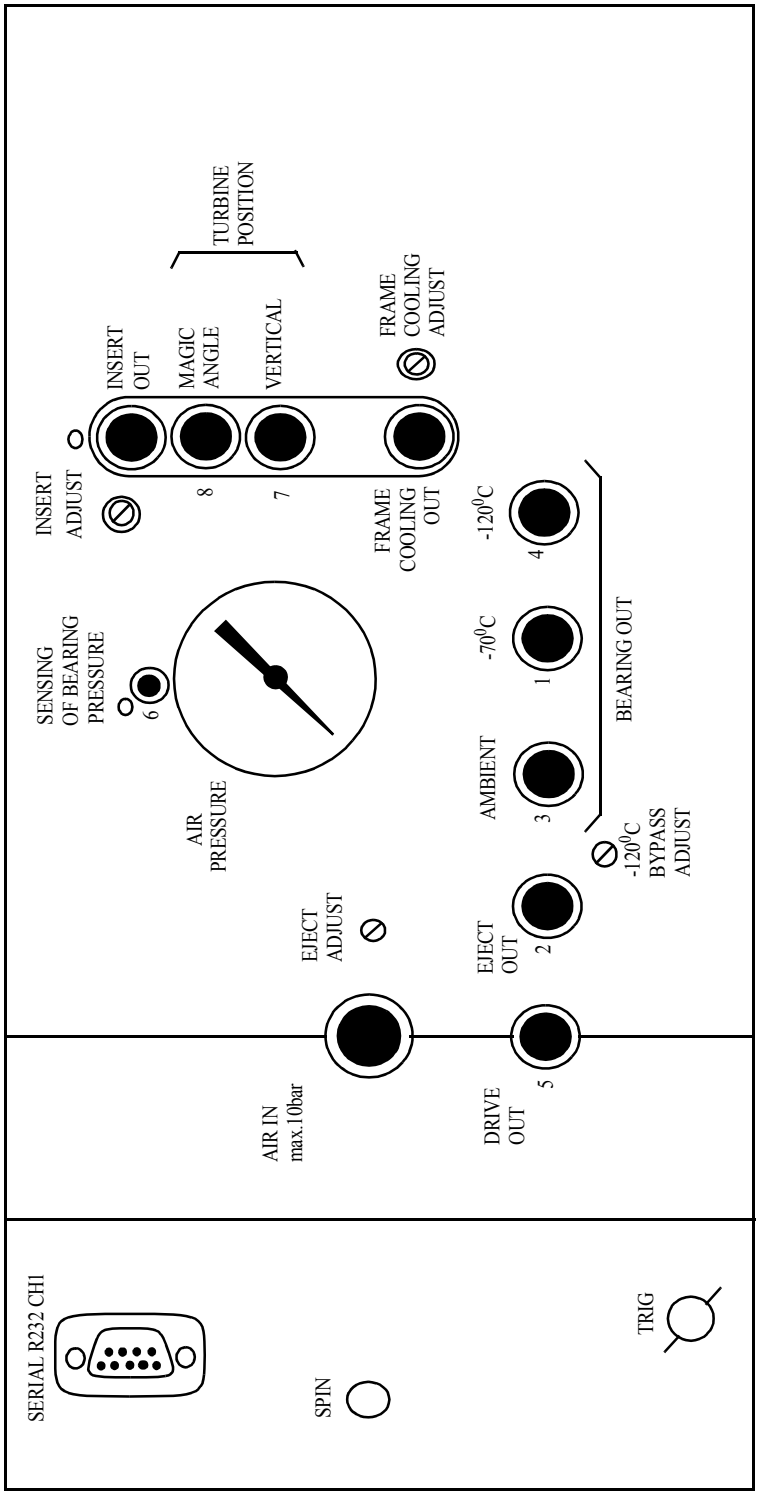


Figure 6.1. Backpanel connections

Insert Out

Not connected in standard configuration with SB magnet. If a Sample Changer System (BACS) is used with a SB magnet, or if a WB magnet is used then this connection is required.

Insert Adjust

Not required in normal operation for SB magnet. Will require initial adjustment at installation with WB magnets.

Eject Out

Pressure **output** which ejects the rotor from the stator.

Eject Adjust

Enables the operator to adjust the eject output pressure. This adjustment should only ever be necessary at installation or whenever a probe is changed. Turn the screw clockwise to reduce the air flow and anti clockwise to increase.

Frame Cooling Out

Pressure **output** which cools the probe assembly.

Frame Cooling Adjust

Enables the operator to adjust the cooling rate of the probe assembly. This adjustment should only ever be necessary whenever the operating temperature is changed. Turn the screw clockwise to reduce the air flow and anti clockwise to increase.

Bearing Out

Ambient, -70°C, -120°C: One of these three **output**s will be connected to either a heat exchanger or directly to the probe. The PU is programmed so that only one of the 3 output valves can ever be open at any given time. In the standard configuration the Ambient output is used. The -70°C, -120°C outputs have been made available to make the current PU model compatible with older heat exchangers units but are not normally used.

Magic Angle

Output that tilts the stator to the desired angle.

Vertical

Output that switches the stator back to the vertical position to enable the rotor to be ejected or a new rotor inserted. This output is required for standard bore magnets only.

120C Bypass Adjust:

Not required in normal operation.

Spin

Electrical **Input** signal used to monitor the spin rate of the rotor.

Trig

Electrical **Output** to TCU. This is a TTL 5V signal that replicates the spinning of the rotor and can be used to synchronize the phase of the rotation with specific NMR pulse sequences.

General Modes of Operation 7

Introduction 7.1

An understanding of the various modes of operation is essential if the operator is to properly use the PU. Four modes of operation are possible. They are

- Local Automatic
- Local Manual
- Remote Automatic
- Remote Manual

The current mode of operation at any given time can be seen from the front panel LED display.

Local Manual Mode 7.2

In this mode the user adjusts the DP and BP from the front panel to achieve the appropriate spin rate. This requires a knowledge of appropriate settings and is recommended for more experienced operators only. Although more difficult to use one advantage of operating in the manual mode is that max spin rates which are set in automatic mode may be bypassed. Furthermore if the system is using non standard probes or rotors then there may be a case for operating in this mode since the standard algorithms may not be the most suitable.

Note that in this mode the PU must be programmed with information regarding the magnet bore (SB or WB) so that the Vertical command is correctly implemented.

Local Automatic Mode 7.3

This is a simpler mode of operation by which the user enters the desired spin rate on the front panel and the PU selects and implements the appropriate DP and BP based on type of magnet, rotor and probe. **Unless the user wishes to implement some non-standard procedure or is operating with non-standard probes etc. LOCAL AUTOMATIC mode is recommended over LOCAL MANUAL mode**.

An essential feature of this mode is that the PU must first be programmed with information regarding the hardware such as magnet bore, probe size etc. Otherwise unsuitable combinations of DP and BP may be applied (see ***"Status Page 1" on page 19***

Remote (XWIN-NMR) Manual and Automatic Mode *7.4*

This is probably the most user-friendly mode of operation and as with the local mode, two possibilities exist, either manual or automatic. The commands are entered in the XWIN-NMR control window as opposed to controlling the operation from the PU front panel. **Once again unless the user wishes to implement some non-standard procedure or is operating with non-standard probes etc. REMOTE AUTOMATIC mode is recommended over REMOTE MANUAL mode**.

As with Local Automatic mode, with Remote Automatic mode the user simply enters the desired spin rate and the PU selects and implements the appropriate DP and BP based on type of magnet, rotor and probe. In Remote Manual mode the user adjusts the DP and BP from the XWIN-NMR control window to achieve the appropriate spin rate.

The hardware communication required to enable the software control the instrumentation is achieved via the rs232 port at the rear panel of the PU. The PU must always be operated in the Remote mode when operated from XWIN-NMR.

Programming the PU to Match the Hardware *7.5*

As explained in previous sections if the PU is operated in either LOCAL AUTOMATIC mode or REMOTE AUTOMATIC mode the PU needs to know some information regarding the type of magnet, rotor and probe etc. The following possibilities exist, although not every combination is supported by the hardware.

probe size	2.5 / BL4 / BL7 / DB7
MAGNET TYPE	SB / WB
Rotor Material	SiN / ZnO

The above information is required so that the PU implements certain max values of spin rate for safety reasons and also attempts to achieve stable spinning using suitable combinations of DP and BP based on established algorithms.

Local Manual Mode 8

Introduction 8.1

This chapter is intended to provide all the necessary information if the PU is to be operated in the LOCAL MANUAL mode. It begins with a general explanation of the keypad and then continues with a step by step guide of spinning the sample. If you are unsure of which mode of operation is most suitable you should refer back to Chapter 7 ***"General Modes of Operation" on page 25***.

Keypad Control 8.2

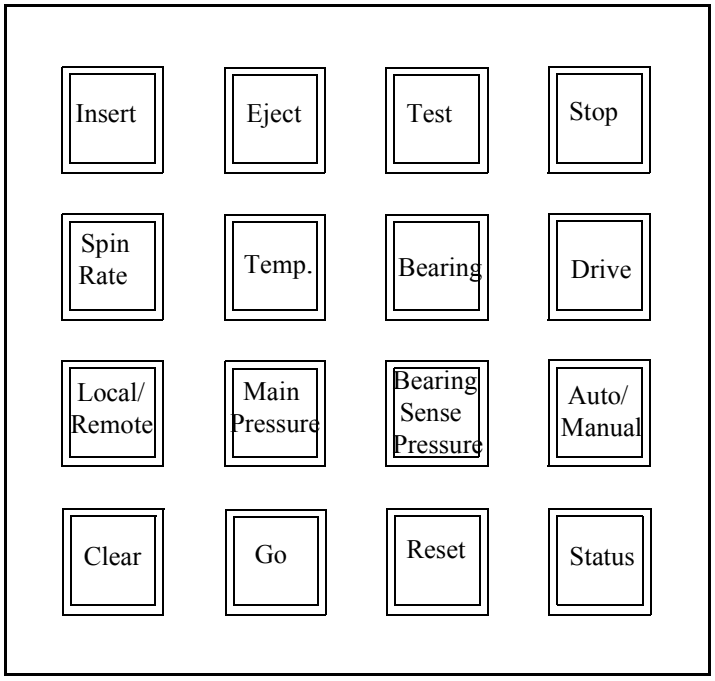


Figure 8.1. Front Panel Keypad

INSERT

This activates the pressure to insert the rotor and will continue indefinitely until STOP is pressed when operating in manual mode. The stator will also tilt to the magic angle position (SB magnets only) after pressing STOP.

EJECT

Tilts the stator to the vertical position (SB magnets only) and applies pressure to eject the rotor. This pressure will continue indefinitely until the operator presses STOP.This is to allow the operator sufficient time to remove the rotor from the top of the transfer system.

TEST

Displays date of current Firmware

STOP

Has two modes of operation.

If the sample is spinning then it stops spinning by setting the BP and DP to zero in a regulated way.

Alternatively if either the INSERT or EJECT key was last pressed, the applied pressure is removed.

SPIN RATE

No function. The spin rate can not be set directly in local manual mode

It can only be observed in the LCD Manual Screen Display.

TEMP

Please note that this key should not normally be activated during an experiment unless the temperature needs to be changed. If the output is to be changed then for safety reasons it is essential that all three outputs are connected correctly.

This key controls the three possible outputs Ambient, -70°C, -120°C.

Press Temp to activate.

The current status of the temp output is shown in the LCD message

ACTUAL -->AMBIENT

Use the single down arrow to scroll through and select the desired output. All other three keypads have no function in this mode.

Press TEMP again to activate the change.

BEARING

Allows the user to vary the Bearing pressure (BP). After activating this key the user is prompted to use the up/down arrows to adjust the BP. On touching any arrow the user is sent into the manual screen display and the current BP displayed. A horizontal arrow will signify that the BP can be adjusted. All 4 up/down arrow keys may be used. Coarse changes are made with the double arrow keys while single arrow keys will make fine adjustments.

DRIVE

Allows the user to vary the Drive pressure (DP). After activating this key the user is prompted to use the up/down arrows to adjust the DP. On touching any arrow the user is sent into the manual screen display and the current DP displayed. A horizontal arrow will signify that the DP can be adjusted. All 4 up/down arrow keys may be used. Coarse changes are made with the double arrow keys while single

arrow keys will make fine adjustments. Note that in Manual mode spinning will commence as soon as a DP is set.

Warning: Always apply the BP before setting the DP. This will ensure that the rotor is levitated before spinning commences

LOCAL/REMOTE

Switches between local (keypad control) and remote (XWIN-NMR control).This will alter the display on the LED front panel.

MAIN PRESSURE

Displays the Main Pressure as measured at the rear of the PU.

BEARING SENSE PRESSURE

Displays the Bearing Pressure as measured at the base of the probe.

AUTO/MANUAL

Toggles between Automatic and Manual mode. This will alter the display on the LED front panel.

CLEAR

No function in manual mode.

GO

No function in manual mode. As soon as the DP and the BP are present spinning will commence independent of whether the GO key has been pressed or not.

RESET

Resets the unit. A reset will immediately remove the DP but maintain the BP. Do not use this key under normal circumstances.

STATUS

Enables the user to view and edit selected parameters.

The LCD display is divided into a default auto screen display as well as three pages called status pages 1, 2 and 3 respectively. The explanation of these parameters was described in 5.3 ***"LCD Display and Arrow Keypad" on page 18*** and will not be repeated here. If you are unsure of the meaning of these parameters you should refer back to this section.

Only page 1 is required to be set by the operator. Pages 2 and 3 would normally only be used to troubleshoot the instrument.

Operating in the LOCAL MANUAL Mode *8.3*

For the purposes of this guide a typical experiment using a 7 mm. probe in a standard bore magnet with a spin rate of 5 kHz has been chosen. However if the sample size or spin rate are different then the operator can make the appropriate adjustments.

Table 8.1. ***"PH MAS200 - 400 SB BL4" on page 30*** shows typical values of BP and DP and resulting typical spin rates for 4 mm diameter rotors in a SB magnet while Table 8.2. ***"PH MAS200-400SB BL7" on page 31*** shows the equivalent pressures for 7 mm rotors. The listed values are average values given for guidance only. Deviation for these values due to variations in production are likely. Operation in this LOCAL MANUAL mode will require an amount of trial and error to establish the precise combination of DP and BP to achieve the required spin rates.

As you can see the settings and resulting spin rates are entirely dependent on whether the probe is 4mm or 7 mm. Since in the MANUAL mode the sample is spun entirely according to the DP and BP as set by the operator, no algorithms are used and hence it is not necessary to program the PU with information regarding the probe size and type as regards choosing the correct algorithms. However it is important that the entries in Status Page 1 are correct so that safe max pressures appropriate to the hardware are applied. Furthermore the PU needs to know whether the magnet is SB or WB for correct application of the vertical commands.

In MANUAL mode no max. spin rates are set but max. values of DP and BP are, which will effectively limit the spin rate.

Although not strictly necessary it will be seen in the description below that to achieve a spin rate of 5 kHz the DP and BP are not set to 2500 and 1070 in one single step. Instead they are set to (somewhat) arbitrary intermediate values before the final values are set. This is good practice and merely ensures that the desired spin rate is approached gradually avoiding major overshoot and possible damage.

Warning: Always apply BP before setting the DP. This will ensure that the rotor is levitated before spinning commences.

Table 8.1. PH MAS200 - 400 SB BL4

BP (mbar)	DP (mbar)	Approx. Spin rate (kHz)
2300	280	4
2400	370	5
2500	510	6
2800	790	8
3000	1180	10
3000	1820	13
3000	2280	15

Table 8.2. PH MAS200-400SB BL7

BP (mbar)	DP (mbar)	Approx. Spin rate (kHz)
1500	480	3
2000	740	4
2500	1070	5
3000	1380	6
3000	1820	7

Step by Step Guide to Spin the Sample *8.4*

Warning: Always apply BP before setting the DP. This will ensure that the rotor is levitated before spinning commences.

1. Switch on the PU unit and press the Auto/Manual key on the front panel to switch to manual mode. This will be confirmed by the front panel LED display.
2. Place the sample at the top of the transfer tube having first pressed the INSERT key. (for SB magnets the INSERT command will ensure that the stator is in the vertical position)
3. After a few seconds press STOP. The rotor is now in place inside the stator.
4. Press BEARING to set the BP. The message '*use arrow keys to set Bearing*' will be displayed. The LCD auto display will be as follows:

   SPIN Dp 0mB

   0 Bp**>** 0mB

   The arrow immediately to the right of Bp indicates that the bearing pressure may now be adjusted.
5. Use the arrow keypad on the front panel to increase the BP to 500.
6. Press the DRIVE key. The LCD display will now be as below.

   SPIN Dp> 0mB

   0 Bp 500mB

   The arrow immediately to the right of Dp indicates that the Drive pressure may now be adjusted
7. Use the arrow keypad on the front panel to increase the DP. The SPIN display will also increase. Continue to increase the DP until the SPIN rate is between 500 Hz and 1000 Hz.

   The display will now be approximately as below

SPIN	Dp>	90mB
500	Bp	500mB

8. Press BEARING to further adjust the BP and increase it to 2500.
9. Press Drive to further adjust the DP and increase it to 1070.

   The SPIN should now increase to approximately 5kHz and the display will be as below:

SPIN	Dp>	1070mB
5000	Bp	2500mB

10. The procedure is then to make further adjustments to the DP and BP until the required SPIN is achieved. This may require some experience and for this reason the MANUAL mode is not always recommended.
11. When the experiment is finished press STOP. This will reduce the BP and the DP in a regulated manner.
12. Press EJECT and the rotor will be lifted to the top of the transfer tube.
13. Remove the rotor and press STOP. This will turn off the eject pressure and keep the stator in the vertical position.

Local Automatic Mode

9

Introduction 9.1

This chapter is intended to provide all the necessary information if the PU is to be operated in the LOCAL AUTOMATIC mode. It begins with a general explanation of the keypad and then continues with a step by step guide of spinning the sample. If you are unsure of which mode of operation is most suitable you should refer back to chapter 7 ***"General Modes of Operation" on page 25***.

Keypad Control 9.2

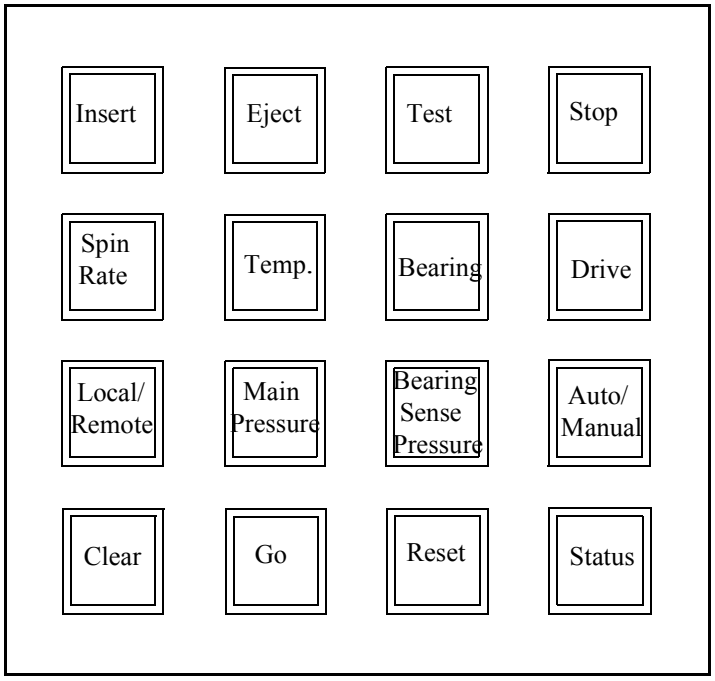


Figure 9.1. Front Panel Keypad

INSERT

This activates the pressure to insert the rotor and will either continue for 10s before being automatically cutoff or will cutoff as soon as the Stop key is pressed. The stator will also be automatically tilted to the magic angle position (for SB magnets only).

EJECT

Tilts the stator to the vertical (for SB miscounts only) and applies pressure to eject the rotor The pressure will continue indefinitely until the operator presses STOP.This is to allow the operator sufficient time to remove the rotor from the top of the transfer system.

TEST

Displays date of current Firmware

STOP

Has two modes of operation.

If the sample is spinning then it stops spinning by setting the BP and DP to zero in a regulated way.

Alternatively if the INSERT or EJECT key was last pressed, the applied pressure is removed.

SPIN RATE

Enables the operator to set the desired spin rate Vd on the LCD display.

TEMP

Please note that this key should not normally be activated during an experiment unless the temperature needs to be changed. If the output is to be changed then for safety reasons it is essential that all three outputs are connected correctly.

This key controls the three possible outputs Ambient, -70°C, -120°C.

Press Temp to activate.

The current status of the temp output is shown in the LCD message

ACTUAL -->AMBIENT

Use THE SINGLE arrow down to scroll through and select the desired output. All other three keypads have no function.

Press TEMP again to activate the change.

BEARING

Allows the user to set the start-up Bearing pressure using the arrow keys.

DRIVE

No function in this mode.

LOCAL/REMOTE

Switches between local (keypad control) and remote (XWIN-NMR control).This will alter the display on the LED front panel.

MAIN PRESSURE

Displays the Main Pressure as measured at the rear of the PU.

BEARING SENSE PRESSURE

Displays the Bearing Pressure as measured at the base of the probe

AUTO/MANUAL

Toggles between Automatic and Manual mode. This will alter the display on the LED front panel.

CLEAR

No function in this mode.

GO

Starts the spinning by applying the BP and DP appropriate to the desired spin rate.

RESET

Resets the unit. Do not use this key under normal circumstances.

STATUS

Enables the user to view and edit selected parameters.

The LCD display is divided into a default auto screen display as well as three pages called status pages 1, 2 and 3 respectively. The explanation of these parameters was described in 5.3 ***"LCD Display and Arrow Keypad" on page 18*** and will not be repeated here. If you are unsure of the meaning of these parameters you should refer back to this Chapter.

Only page 1 is required to be set by the operator. Pages 2 and 3 would normally only be used to troubleshoot the instrument. If page 1 is not set correctly then the PU will have difficulty selecting the algorithms appropriate to the specific hardware set-up.

Operating in the LOCAL AUTOMATIC Mode *9.3*

In order for the correct algorithms to be applied the PU requires information regarding the type of magnet, rotor and probe.

The following possibilities exist.

probe size	2.5 / BL4 / BL7 / DB7
MAGNET TYPE	SB / WB
Rotor Material	SiN / ZrO

Note that only combinations of 'probe' and 'diameter' that correspond to actual existing hardware can be selected.

Once this information has been entered the only other information that needs to be entered is the desired spin rate Vd. With the appropriate hardware information the PU can implement certain max values of spin rate for safety reasons and also attempt to achieve stable spinning using suitable combinations of DP and BP based on established algorithms.

For the purposes of this guide we have chosen a typical experiment using a 7 mm. probe in a standard bore magnet with a rotor material of Silicon Nitride. However if the probe size, magnet type or rotor material are different then the operator must make the appropriate adjustments.

Step by step guide to spin the sample *9.4*

1. Switch the unit to Local Automatic mode. This will be confirmed by the front panel LED display.
2. The LCD display should be as below

Dp	0	Vd>	0000
Bp	0	Va	0000

The display shows that DP and BP are both set at zero and that the desired spin rate (Vd) and the actual spin rate (Va) are consequently also zero.

3. Place the sample at the top of the transfer tube having first pressed the INSERT key. (for SB magnets the INSERT command will ensure that the stator is in the vertical position).
4. After a few seconds the insert pressure will be removed automatically. The rotor is now in place inside the stator and the stator is automatically tilted to the magic angle.
5. Press STATUS on the front panel keypad. The display changes to

PHs	TYP	MAT
>4	WB	ZnO

This is Status Page 1 and the meaning of the parameters are as follows.

PHs: Probe size: Currently set to 4 mm

TYP: Type of magnet bore. Currently set tor WB

MAT: Rotor material Currently set to ZnO (Zirconium Oxide)

The values above are the default settings of the unit which are loaded after a power off or a reset.

The arrow immediately to the left of the 4 indicates that the probe size may be altered by using the arrow keys.

6. Use the change value arrow (down arrow) to set the PHs to a value of 7. The display should now be as below:

PHs	TYP	MAT
>7	WB	ZnO

7. Press the cursor arrow (up arrow) to enable the magnet type be adjusted. The display should now be as below.

PHs	TYP	MAT
7	>WB	ZnO

8. Press the change value arrow (down arrow) to change the magnet type to SB. The display should now be as below.

PHs	TYP	MAT
7	>SB	ZnO

9. Finally press the cursor arrow (up arrow) to enable the rotor material be adjusted. The display should now be as below

PHs	TYP	MAT
7	SB	>ZnO

10. Press the change value arrow (down arrow) to change the material to SiN. The display should now be as below.

PHs	TYP	MAT
7	SB	>SiN

The PU is now able to apply the correct algorithms appropriate to the hardware.

11. Press SPIN RATE on the front panel keypad and use the arrow keys to set a desired spin rate Vd.
12. Press GO

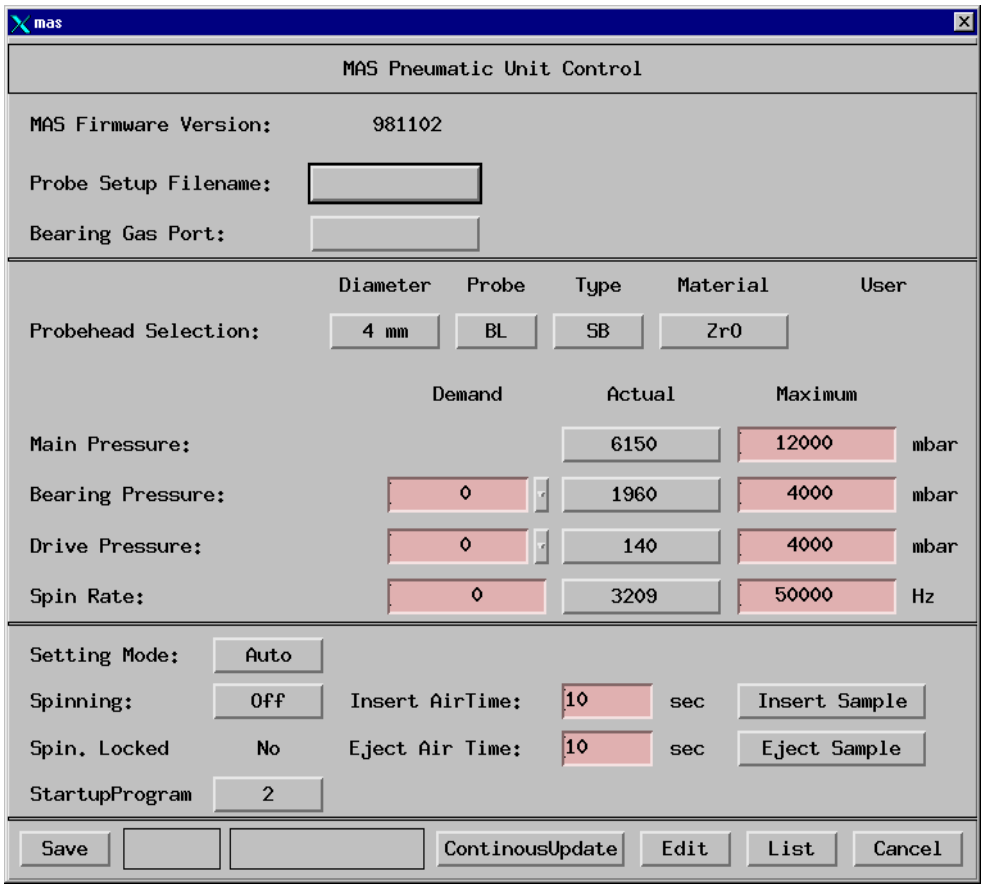
    The PU will then gradually increase the applied BP and DP. The display will show the actual spin rate Va increasing until it reaches the required value of Vd. When this has been achieved the Spin Lock LED on the front panel will light.
13. When the experiment is finished press STOP. This will reduce the BP and the DP in a regulated manner.
14. Press EJECT and the sample will be lifted to the top of the transfer tube.
15. Remove the rotor and press STOP. This will turn off the eject pressure.

XWIN-NMR Control 10

Introduction 10.1

This chapter describes the XWIN-NMR interface and specifically the various fields in the MAS control window. The PU must always be operated in the Remote mode when operated from XWIN-NMR. Although there are two modes of operation REMOTE MANUAL and REMOTE AUTOMATIC, they both use the same interface and they will be dealt with together in this chapter. If you are unsure of the distinction between REMOTE MANUAL and REMOTE AUTOMATIC then refer back to chapter 7 ***"General Modes of Operation" on page 25***. The XWIN-NMR window is evoked by typing '**mas**' and the screen display is shown below.

Figure 10.1. MAS XWIN-NMR *control window*



Description of various fields. *10.2*

MAS Firmware Version

Displays date of current firmware.

Probe set-up Filename

Clicking on this enables a set of probe specific parameters to be loaded automatically. The source files are stored in u/exp/stan/nmr/lists/mas/filename

Bearing Gas Port

Can be set to Ambient / -70°C / -120°C /. This controls the output used to control the sample temperature. Default setting is Ambient which is the standard configuration.

Probehead Selection

Enables the Diameter, Type of probe, type of magnet and rotor material to be set. This will dictate the algorithms that are used to spin the sample. Note that only combinations of 'probe' and 'diameter' that correspond to actual existing hardware can be selected. When the control window is first opened the current status of the parameters is read from the PU.

Main Pressure, Bearing Pressure, Drive Pressure, Spin Rate

Displays values of these parameters. Note that for these to be current the **'continuous update'** field at the base of the display must be clicked. The Demand (desired) spin rate can be entered and the appropriate DP and BP will be automatically set. This corresponds to operating in the Automatic mode. Alternatively the DP and the BP can be set and the rotor will spin accordingly. This corresponds to operating in the Manual mode.

Setting Mode

Can be set to 'auto' or 'manual'.

Spinning

Can be set to 'on' or 'off'. Note that you should wait 10s after the 'insert sample' command before setting the spin to 'on'.

Spin Locked

Will be set to 'yes' whenever a stable spin rate has been detected. When the spin rate has reached the set value to within 5 Hz for a duration of greater than 5s then Spin Locked will switch to 'yes'. This is only applicable to Automatic mode because there is no set spin rate in Manual mode.

Insert Air Time

Not adjustable and set permanently to 10s.

Eject Air Time

Set with the '**cfmas**' command. Default is 10s.

Insert Sample

Inserts the sample

Eject Sample

Ejects the sample

Continuous Update

This will ensure that the displayed parameters are current.

Cancel

Exit the control window

Baseline commands *10.3*

The following commands may be entered directly onto the XWIN-NMR baseline without entering the control window evoked by the command '**mas**'.

cfmas configure the PU (This should only be necessary at installation)

masr set the spinning rate

masg start spinning

mash stop spinning

mase eject sample

masi insert sample

masrmon Used to produce an on-screen display of the current spin rate for ease of monitoring. The operator can also dump the spin rate to a log file which can be inspected later.

Operating in the REMOTE MANUAL Mode *10.4*

Note that in this mode the user adjusts the DP and BP to achieve the appropriate spin rate. This requires a knowledge of appropriate settings and is recommended for more experienced operators only. Although more difficult to use, one advantage of operating in the manual mode is that max spin rates which are set in automatic mode may be bypassed (Max. values of DP and BP are set however). Furthermore if the system is using non standard probes or rotors then there may be a case for operating in this mode. **However unless you have a specific reason for operating in the MANUAL mode you are advised to move directly to the later section which will provide the same guide operating in the much simpler REMOTE AUTOMATIC mode.**

Step by Step Guide to Spin the Sample *10.5*

Warning: Always apply the BP before setting the DP. This will ensure that the rotor is levitated before spinning commences

1. Ensure that the PU is operating in Remote mode.This can be achieved by toggling the Local / Remote key on the front panel keypad.
2. Enter the command '**mas**' to call up the Mas window.
3. Enter the correct details under 'Probehead Selection'. If available load an existing parameter set by clicking on 'Probe Setup Filename'. Alternatively enter the diameter, Probe, magnet type and rotor material under the 'Probehead Selection' fields.
4. Under Bearing Gas Port enter the appropriate output (Ambient, -70°C, -120°C) that is to be used for the BP. In the standard configuration the 'Ambient' output is used.
5. Set the 'Setting Mode' to 'Manual'.
6. Place the rotor into the top of the transfer system having first clicked on 'Insert Sample'.
7. Click on 'Demand Bearing Pressure' and enter the desired value
8. Click on 'Demand Drive Pressure' and enter the desired value.
9. Click on 'Continuous update'

The 'Actual Bearing Pressure' and 'Actual Drive Pressure' values will be seen to increase as will the 'Actual Spin Rate'. They will be updated every 1 second as long as the 'Continuous update' field has been clicked.

10. To alter the spin speed in manual mode the 'Demand Bearing Pressure' and the 'Demand Drive Pressure' need to be adjusted.
11. When the experiment is completed click on 'Spinning' and set it to 'Off'.
12. Click on 'Eject Sample' and remove the rotor.
13. Click on 'Cancel' to exit the menu.

The spin rate may be monitored **during** an experiment but with the main Control window closed by clicking on 'Cancel' and entering the command '**masrmon'.**

Operating in the REMOTE AUTOMATIC Mode *10.6*

For the successful spinning of the sample it is vital that the correct entries be made in the 'Probehead Selection' fields. This is so that the algorithms most appropriate to the chosen hardware are selected. For ease of use a standard set of parameters can be stored under a 'Probe Setup Filename'.

Step by Step Guide to Spin the Sample *10.7*

1. Ensure that the PU is operating in Remote mode.This can be achieved by toggling the Local / Remote key on the front panel keypad.
2. Enter the command '**mas**' to call up the Mas window.
3. If available load an existing parameter set by clicking on 'Probe Setup Filename'. Alternatively enter the diameter, Probe, magnet type and rotor material under the 'Probehead Selection' fields.

4. Under Bearing Gas Port enter the output (Ambient, -70°C, -120°C) that is to be used for the BP. In the standard configuration the ‘Ambient’ output is used.
5. Set the ‘Setting Mode’ to ‘Auto’.
6. Place the rotor into the top of the transfer system having first clicked on ‘Insert Sample’.
7. Click on ‘Spin Rate’ and enter the desired speed.
8. Click on ‘Spinning’ and set to ‘on’.
9. Click on ‘Continuous update’

The ‘Actual Bearing Pressure’ and ‘Actual Drive Pressure’ values will be seen to increase as will the ‘Actual Spin Rate’. They will be updated every 1 second as long as the ‘Continuous update’ field has been clicked.

10. The ‘Spin Locked’ field will be set to ‘Yes’ when a sufficiently stable spin rate has been detected. This is when the spin rate has reached the set value to within 5 Hz for a duration of greater than 5s.
11. When the experiment is completed click on ‘Spinning’ and set it to ‘Off’.
12. Click on ‘Eject Sample’ and remove the rotor.
13. Click on ‘Cancel’ to exit the menu.

The spin rate may be monitored **during** an experiment but with the main Control window closed by clicking on ‘Cancel’ and entering the command ‘**masrmon’.**

System Specifications 11

Spin Rates Accuracy and Stability 11.1

± 5 Hz for 4 mm rotors at less than 15 kHz.

± 2 Hz for 7 mm rotors at less than 5 kHz.

± 10 Hz for 2.5 mm rotors at less than 35 kHz.

Temperature Range 11.2

The SB MAS probe is designed for VT operation from -80°C to +120°C.

Note that for high temperatures special rotors and in particular rotor caps are required.

Maximum Spin Rate 11.3

This is rotor/ probe diameter and magnet bore dependent and is limited to ensure that no damage is done to either the sample or the probe.

The SB MAS probe is designed for MAS experiments at spin rates up to 7kHz with 7mm outer diameter rotors, up to 15kHz with 4mm outer diameter rotors and up to 35kHz with 2.5mm outer diameter rotors .The max spin rate is only operational when using automatic mode.

When operating in manual mode there is not an explicit max spin rate however there is a max allowed BP and DP which will effectively establish a max spin rate.

The above spin rates can only be achieved with a well regulated air supply and normal laboratory conditions.

Available Probes and Rotors 11.4

Various configurations are available depending on the application

Range of suitable probe size	BL2.5 / BL4 / BL7 / DB7
Range of suitable rotors:	2.5mm, 4mm, 7 mm
Rotor Material	SiN / ZnO

Figures

Figures

Tables

Tables