

# User's Guide

## BRUKER NMR Spectrometers

DRX400 (SB040)      AMX300 (SB132)      DMX300 (SB132)

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### SAFETY PRECAUTIONS:

- ◆ Persons wearing **PACEMAKERS** may not enter the immediate area of the Magnet.
- ◆ No Steel objects (gas cylinders, storage dewars, tools, keys etc.) should come within one meter of the cryostat.
- ◆ Keep magnetic devices (e.g. floppy diskettes, credit cards) away from the cryostat.
- ◆ NMR sample tubes should be checked for mechanical defects (e.g. minor cracks).
- ◆ MSDS sheets are located in a binder in room SB134.
- ◆ Specific Safety information for the Instrumentation Laboratory (presence of Strong Magnetic and Radio Frequency Fields and CRYOGENIC fluids) is also posted in SB134 and SB 040.
- ◆ High electrical voltages are present in most of the major instruments in the laboratory.

### UNDERGRADUATE USE OF NMR SPECTROMETERS

As with any other Instrumentation Facility instrument, no undergraduate may operate NMR spectrometers without the prior approval of Dr. Yamdagni. Beyond this, the following restrictions also apply:

1. No undergraduate may use **DRX400** or **AMX300** NMR spectrometer.
2. Use of **DMX300** & **UGI400** NMR spectrometers may be allowed if the need to do so is a requirement of a CHEM 502 project or employment within a Departmental research group as a summer assistant.

### USE OF THE BRUKER DRX400 / AMX300 / DMX300

Only those who have been checked out according to the following procedure will be allowed to operate the **DRX400**, **AMX300** and **DMX300** systems.

- ◆ Attend a TWO hour **DRX400 / AMX300 / DMX300** operator training session (given periodically by Dr. Yamdagni).
- Return a complete NMR assignment
- Pass a Proficiency Test on Bruker **DMX300** system.

Failure to follow the above guidelines may result in the loss of user privileges.

### ACCESS

#### **DRX400 and AMX300:**

Please use the request sheets in SB134 (outside RY's office).

#### **DMX300:**

Online computer booking for  $^1\text{H}$  and  $^{13}\text{C}$  experiments.

## HARDWARE

### DRX400

Probes	BBI / Gradient	Inverse	Proton (inner coil)
	BBO / Gradient	Normal	Proton (outer coil)
	FH05	Normal	Proton (outer coil)
	BB10	Normal	Proton (outer coil)

Gradient Accessory

VT

Pentium PC with WINDOWS XP PRO

### AMX300

Probes	BBI5	Inverse	<sup>1</sup> H	inner coil
	FH05	Normal	<sup>1</sup> H	outer coil
	BB10	Normal	<sup>1</sup> H	outer coil
	BB20	Normal	<sup>1</sup> H	outer coil
	BL4	Solid		4 mm Rotor
	BL7	Solid		7 mm Rotor

VT

Silicon Graphics O2 with IRIS 6.5 ( UNIX )

### DMX300

Probes	TXI5	Inverse	<sup>1</sup> H	inner coil
			<sup>13</sup> C	outer coil

Silicon Graphics O2 with IRIS 6.5 ( UNIX )

## SOFTWARE

### DRX400

Enter	<b>group login name and password</b>	
double click	<b>clear</b>	
double click	<b>Xwin 3.1</b>	start xwin-nmr version 3.1
type	<b>lockdisp</b>	open lock display window

### AMX300 & DMX300

Enter	<b>group login name and password</b>	
click	<b>DESKTOP</b>	open a unix window
type	<b>xwinnmr</b>	start xwin-nmr version 2.5
type	<b>lockdisp</b>	open lock display window

**REMEMBER to LOGOUT after you are finished with your experiment(s).**

<b>FILE FORMAT</b>
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**DRX400:**

e:/data/group/nmr/filename/expno/procno/

example: e:/data/**D0911ry**/nmr/**91120ry1**/1/1/**AMX300:**

/ure/data/group/nmr/filename/expno/procno/

example: /ure/data/**A0911ry**/nmr/**91120ry1**/1/1/**DMX300:**

/dmx1/data/group/nmr/filename/expno/procno/

example: /dmx1/data/**M0911ry**/nmr/**91120ry1**/1/1/**ARCHIVING:**

NMR Data from all the NMR instruments are archived every month by Instrumentation Facility personnel.

<b>SETUP</b>
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[sample installation, spin and shimming]

remove		pressure plug from the top of magnet
press	<b>lock</b>	<b>off</b>
press	<b>spin</b>	<b>off</b>
press	<b>lift</b>	activate eject air
install	<b>sample</b>	36mm [minimum] solvent depth is required
press	<b>lift</b>	deactivate eject air
replace		pressure plug at the top of magnet
press	<b>spin</b>	<b>on</b>
type	<b>lock</b>	select solvent from the table
adjust	<b>lock power</b>	if required
adjust	<b>lock gain</b>	if required
adjust	<b>z<sup>1</sup>, z<sup>2</sup>, z<sup>3</sup></b>	for maximum homogeneity
press	<b>autoshim</b>	<b>z<sup>1</sup>, z<sup>2</sup>, z<sup>3</sup></b> shims will be optimized

<b>ACQUISITION</b>
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	type	<b>edc</b>	edit current data filename
	change	<b>filename</b>	format should be <b>YMMDDID#</b>
	change	<b>expno</b>	if necessary
	change	<b>procno</b>	if necessary
<b>OR</b>			
	type	<b>search</b>	open portfolio window
	choose	<b>filename</b>	recall an older file for parameter set
	type	<b>edc</b>	edit current data filename
	change	<b>filename</b>	format should be <b>YMMDDID#</b>
	change	<b>expno</b>	if necessary
	change	<b>procno</b>	if necessary
<b>DRX400</b>	type	<b>bbi</b> or <b>bbo</b>	click on experiment of interest
<b>AMX300</b>	type	<b>bbi5</b> or <b>fh05</b>	click on experiment of interest
<b>DMX300</b>	type	<b>txi5</b>	click on experiment of interest
	Choose	<b>copy all</b>	
	type	<b>ii</b>	initialize the hardware
	type	<b>setti</b>	enter a title for your experiment



<b>SOME COMMON COMMANDS &amp; PARAMETERS</b>
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<b>a</b>	go to acquisition window
<b>abs</b>	automatic baseline fix
<b>ased</b>	acquisition set up parameters
<b>bbi</b>	choose parameter set for BBI probe
<b>bbi5</b>	choose parameter set for BBI5 probe
<b>bbo</b>	choose parameter set for BBO probe
<b>cy</b>	vertical height for plot output
<b>dpa</b>	parameters for acquired data set
<b>eda</b>	acquisition parameters
<b>edc</b>	current parameters
<b>edg</b>	graphics parameters
<b>edo</b>	output parameters
<b>edp</b>	processing parameters
<b>edte</b>	variable temperature control
<b>efp</b>	em + ft + pk
<b>expt</b>	experiment time
<b>fh05</b>	choose parameter set for FH05 probe
<b>halt</b>	stop acquisition
<b>iexpno</b>	increase experiment number
<b>iprocno</b>	increase process number
<b>ii</b>	initialize hardware
<b>lb</b>	line broadening parameter
<b>lockdisp</b>	lock window
<b>lock</b>	automatic lock with choice of solvent
<b>multizg</b>	start a set of predefined experiments
<b>ns</b>	number of scans
<b>plot</b>	output to printer
<b>rga</b>	automatic receiver gain
<b>search</b>	access archived data
<b>setti</b>	write a title for current data set
<b>stop</b>	abort acquisition or wobble program
<b>txi5</b>	choose parameter set for txi probe
<b>view</b>	direct plot output to the screen
<b>wobb</b>	start probe coil tuning program
<b>xwinmr</b>	start nmr software
<b>zg</b>	zero memory and start acquisition