

NMRFAM Spectrometer Guide

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This guide is designed to provide users of the DMX spectrometers at NMRFAM with a few basic instructions when getting started. It is not intended to be comprehensive, but rather to provide answers to the most common questions asked by new users and provide pointers to the other resources available to users. Please address questions or comments regarding the accuracy or usefulness of this document to Brian Volkman (volkman@nmrfam.wisc.edu).

Contents

Accessing the NMRFAM computers and spectrometer workstations	2
<i>OBTAINING AN ACCOUNT</i>	2
<i>THE NMRFAM WORKSTATIONS - <HOSTNAME>.NMRFAM.WISC.EDU</i>	2
<i>THE NMRFAM FILESYSTEM - TYPE DF TO SEE THE NAMES OF ALL AVAILABLE DISKS</i>	2
Major components of the DMX spectrometer	3
<i>MAGNET</i>	3
<i>PROBE</i>	3
<i>FILTERS</i>	3
<i>PREAMPLIFIER (HPPR)</i>	3
<i>HARDWARE CONSOLE</i>	3
<i>BSMS KEYPAD</i>	3
<i>SGI INDY COMPUTER</i>	3
Logging on the DMX spectrometer and starting XwinNMR software	4
Setting up the spectrometer for acquisition	4
<i>CHOICE OF PROBE</i>	4
<i>VARIABLE TEMPERATURE</i>	4
<i>SETTING TEMPERATURE</i>	4
<i>INSERTING THE SAMPLE IN THE MAGNET</i>	4
<i>THE DEUTERIUM LOCK</i>	4
<i>TUNING THE PROBE</i>	5
<i>MANUAL SHIMMING</i>	5
<i>GRADIENT SHIMMING</i>	5
Other sources of information	5

Accessing the NMRFAM computers and spectrometer workstations

Obtaining an account

Each of the five DMX spectrometers is operated via a Silicon Graphics (SGI) Indy workstation, which uses a UNIX operating system. A basic familiarity with UNIX will be essential to the efficient use of the spectrometer, as any manipulation of NMR data outside of the Bruker XwinNMR software (off-line processing, file transfer, data archival, etc.) will entail the use of UNIX commands.

All NMRFAM users will be given a computer account allowing them access to all of the Silicon Graphics workstations of the Facility. Frits Abildgaard is the System Administrator responsible for establishing computer accounts. Contact Frits directly by email (abild@nmrfam.wisc.edu) or ask any NMRFAM staffperson to arrange to have an account created. You will choose a *username* and *password* which will allow you to access the computers from the workstations at NMRFAM and remotely from computers off site over the Internet. **NOTE** - login to *banyo* (rlogin banyo) and change password with the command *passwd*.

The NMRFAM workstations - <hostname>.nmrfam.wisc.edu

Each SGI has a unique name which will allow you to use *telnet* or *FTP* clients to access them from remote locations, which is <hostname>.nmrfam.wisc.edu, where the <hostname> for the workstations dedicated to the DMX consoles are **pisa** (DMX400), **kerry** (DMX500i), **devon** (DMX500ii), **dexter** (DMX600) and **telemark** (DMX750). There are also a number of additional SGI workstations for which the naming convention is the same (<hostname>.nmrfam.wisc.edu).

The NMRFAM filesystem - type df to see the names of all available disks

Your **root directory** is specified by either **/usr1/people/<username>** or **/usr3/people/<username>**, depending upon your status as an nmrgroup member or outside user. This directory is provided for your general use, but **IS NOT TO BE USED FOR STORAGE OF NMR DATA FILES**, due to space limitations. A number of data disks are designated as working space for NMR data processing and analysis, all of which are named **/hostname_data** (e.g., /sussex_data, /hereford_data). These data disks are **NOT BACKED UP**, so unsaved data could be lost in the event of a hardware failure. In addition, files which are unused for more than **ONE WEEK** are automatically **DELETED** to prevent the rapid accumulation of files to the capacity of the disks. The exception is /watusi_data, which is a large (~40 GB) filesystem with a different type of quota system. See the NMRFAM intranet for details.

The data disks for offline processing are distinct from the disks on each of the spectrometer workstations. Data which is acquired on the **NMR spectrometers** is also **NOT BACKED UP** and will be automatically **DELETED** after a **TWO-WEEK** period of inactivity. The disks on the spectrometer workstations are known by two names, depending upon which computer you are currently logged into. When logged into a spectrometer workstation, the data disk for that machine is named **/u**, in keeping with the requirements of the data structure imposed by the Bruker XwinNMR software. NMR experiments created within the XwinNMR software are stored as subdirectories in the main data directory **/u/data/<username>/nmr**. Alternatively, from any other computer the same disk is named **/dmx###** (### = 400, 500I, 500ii, 600 or 750). For example, when logged into **kerry**, the console for the DMX500i spectrometer, your

data acquired on that machine is found in the directory `/u/data/<username>/nmr`, but from the computer `sussex`, the same directory is called `/dmx500i/data/<username>/nmr`. All of these disks are NFS-mounted and accessible from all of the NMRFAM workstations directly, with no need to FTP files between computers in the NMRFAM cluster.

Major components of the DMX spectrometer

Magnet

All of the magnets at NMRFAM are cryogen-cooled superconducting magnets which require regular filling with liquid nitrogen and liquid helium. Nitrogen is filled every Tuesday for all magnets, and helium fills are performed in the same maintenance period on a schedule which varies from magnet to magnet. Samples are lowered into the bore of the magnet on an air bearing which is automatically controlled.

Probe

The probe contains the circuitry for delivery of rf and gradient pulses into the sample as well as detection of NMR signals. Usually the probe will need to be tuned on each channel before setting up an NMR experiment. The NMRFAM WWW site contains a list of available probes for each spectrometer.

Filters

A number of filters may be needed between the preamp and probe to improve performance and reduce noise from crosstalk between different rf channels. The spectrometer should be configured with appropriate filters for the most routine types of experiments.

Preamplifier (HPPR)

The preamp sits next to the magnet and has an LED display which is used in probe tuning.

Hardware console

Most of the electronic components required for generating rf and gradient pulses and acquiring NMR data are contained in the hardware console. The DMX spectrometer is engineered to ideally allow the user to set up virtually any arrangement of multiple channel pulse scheme without the need to make changes to the physical setup of the hardware, thereby eliminating the need for users to access the console for wiring changes or other adjustments. There should be no need to open the doors of the hardware console during normal operation. Unexplained malfunctions occasionally necessitate resetting the acquisition computer in the console, but NMRFAM staff should be consulted before rebooting the spectrometer.

BSMS keypad

The BSMS keypad sits on the desktop next to the Indy workstation, and is used for a number of functions for controlling the deuterium lock and adjusting shims. It also contains LED displays for monitoring the rf amplifiers.

SGI Indy computer

The Indy which is located with the spectrometer is used to control acquisition of NMR experiments via a network connection to the acquisition computer in the hardware console and the XwinNMR software.

Logging on the DMX spectrometer and starting XwinNMR software

Type your *username* and *password* at the login screen on the Indy workstation to start your session. After the startup process is complete, find a text window and type **xwinnmr** or **uxnmr** to start the Bruker software. The graphics window which appears is the interface to the spectrometer from which all data acquisition will be controlled. The software is controlled through a variety of mouse-activated menus and buttons, as well as typed commands. Users of AMX series Bruker spectrometers will find a high degree of similarity in the interface and commands of the DMX software.

Setting up the spectrometer for acquisition

Choice of probe

Before inserting your sample in the magnet, confirm that the appropriate probe is installed, in terms of rf channels (^1H , ^{15}N , etc.), sample diameter (2.5mm, 5mm, 8mm etc.) and gradient capabilities. **DO NOT CHANGE THE PROBE** unless you have been given previous instruction - see Mark Anderson for training and details, or consult an NMRFAM staffperson if you are in need of hardware changes. Consult the NMRFAM WWW site for available probes.

Variable temperature

For many applications requiring temperature control, it is important that the air refrigeration unit be on, especially if temperature control below 25C is required. If necessary, turn on the cooling unit with switches on both the refrigeration unit and the controller (sits atop the refrigeration unit). Set the temperature of the cooling unit to at least $\sim 30\text{-}40^\circ$ below the desired sample temperature with the silver knob on control unit (e.g. set to -15C for sample temp of 25C). Additional control over VT capability can be gained by adjustment of the airflow (gas flowmeter on front of rf console), but this should generally be left at a setting of $\sim 500\text{l/hr}$ (top of ball at 5).

Setting temperature

The current temperature in the probe can be read from the VT controller in the rf console, and can be set to the desired temperature from within the XwinNMR program. Type **TE** to see the current set temp, and type a new value if desired. Then type **TESET** to send the updated value to the VT controller and move to the new set temp. It is normal for the controller to overshoot and correct when moving to a new temperature, but if a stable temperature is not reached within 5-10 minutes there may be a problem.

Inserting the sample in the magnet

Place NMR tube in a plastic spinner and adjust the sample height with the clear plastic guide. The bottom of the sample tube should be at the 18mm mark for most 5mm dia. samples. Depress the **LIFT ON** button on the upper left corner of the BSMS keypad once to turn on the sample eject air, and again to lower sample after placing in the top of the magnet bore. Lights on the BSMS keypad will indicate whether the sample is up, down or missing.

The deuterium lock

Type **LOCKDISP** to bring up a window displaying the lock signal. type **LOCK** to bring up a choice of lock solvents. Click on appropriate solvent. This runs an automated lock-in routine. Some further adjustment may be necessary. In particular, adjust the **LOCK POWER** and **LOCK GAIN** (buttons on the BSMS keypad) so that the lock signal is in the top 1/3 of the lock display

window. Typical values for 90% H₂O/10% D₂O samples are -20 dB and 125 for lock power and gain, respectively. Also, check the **LOOP GAIN** by pressing the orange **2ND** and **z⁶** buttons simultaneously on the BSMS keypad. Use the keypad dial to select item #2, lock. Press the orange **2ND** button twice to see the current value of **LOOP GAIN**. It should be between ~ -40 - -20 dB, depending upon the s/N of the lock signal. Excessive values of **LOOP GAIN** (e.g. +15) can introduce significant noise into your spectrum due to lock instability.

Tuning the probe

Type **WOBB** to start the wobble sweep. Type **ACQU** to see the wobble curve on the screen. Change the nucleus by clicking the button onscreen labeled **WOBB-SW**. You will be prompted for the desired nucleus and sweep amplitude. You should see a V-shaped dip on the screen when wobb is running. Use the tuning tool to adjust the match and tune at the probe to center and adjust the dip to the bottom of the screen. Tuning knobs are labeled T and M and are color-coded to match the BNC connectors on the front of the probe for multichannel probes. If no V-shaped dip is observed, try enlarging the sweep amplitude, (parameter **WBSW**). **SPECIAL NOTE TO 750 USERS** - for tuning 1H, use the command **WOBB EXT50** and follow instructions on screen to first connect a 50Ω terminator to the preamp output, then connect to probe for normal tuning and matching.

Manual shimming

Manual shimming is performed with the BSMS keypad. Individual shims are selected by depressing combinations of buttons. For the axial shims, depress the on-axis button and then the desired shim (**z¹-z⁶**), and adjust the value with the dial, maximizing the lock display. For off-axis shims, press the desired button (x, y, etc.) and then either the **z⁰** button (for x shim, e.g.) or a higher order **z^x** button for combinations (xz, yz³, etc.). A simple auto-shimming feature is also available and controlled from the BSMS keypad.

Gradient shimming

Currently, samples in H₂O solution can use a gradient-assisted autoshimming feature if a gradient probe is being used. Hopefully the addition of hardware for fast ²H switching will allow gradient shimming of deuterated solvent samples as well. Both 1D and 3D gradient shimming are possible, and 3D shimming seems to perform significantly better than 1D, but a three-axis gradient probe is required for 3D shimming. Currently, the DMX500ii, DMX600 and DMX750 have 5mm triple-resonance probes with three-axis gradients. The DMX400 and DMX500i have Z-axis gradient probes only.

Type **GRADSHIM** to bring up the gradient shimming windows. Two or three new windows should appear. Toggle between 1D and 3D modes, select an appropriate control file, or create your own and press the RUN button. 1D shimming will generally take only a minute or two, while 3D shimming will take from 5-15 min.

Other sources of information

In addition to this guide, consult the many other help resources available at NMRFAM. Of primary use to users on the spectrometer will be the online XwinNMR manual which can be launched by clicking the **HELP** menu within the XwinNMR program. This launches a searchable hyper-linked version of the complete Bruker documentation for the XwinNMR software. For additional documentation on running the spectrometer, consult the AVANCE

USER'S GUIDE which should be located at the console of each spectrometer. Additional hardcopy documentation can be found in the high field library at NMRFAM.

For a wide variety of information on computer related issues (*e.g. data backup, available software, etc.*), type **info** at the UNIX prompt on any workstation to launch the Netscape web browser and access the **NMRFAM INTRANET** maintained by Frits Abildgaard (these pages are also accessible from the main NMRFAM www site, but are restricted to NMRFAM ip numbers; contact Frits Abildgaard [abild@nmrfam.wisc.edu] to have an outside computer added to the host list for remote access to the NMRFAM intranet web pages). Also consult the other sections of the NMRFAM web site at **www.nmrfam.wisc.edu**. A number of useful tips can be found on Ed Mooberry's web page (**www.nmrfam.wisc.edu/~esm**).