

User's Guide

BRUKER NMR Spectrometers

CFI600/RDQ400 (SB034) DRY400 (SB035) UGI400 (SA219)

Dr. R. Yamdagni
Department of Chemistry, University of Calgary

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 departmental 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 **CFI600, RDQ400, DRY400, DRX400 and AMX300** NMR spectrometers.
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 BRUKER CFI600, RDQ400, DRY400 & UGI400

Only those who have been checked out according to the following procedure will be allowed to operate the **CFI600, RDQ400, DRY400, DRX400 & AMX300** systems.

- ◆ Attend a TWO hour operator training session (given periodically by Dr. Yamdagni).
- ◆ Complete the NMR assignment
- ◆ Pass the Proficiency Test.

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

GENERAL NMR USAGE

Approach: Black Box vs. Knowledge based information.

Questions:

1. What information is needed for compound identification?
2. Is NMR the right technique for getting some of this information?
3. Which NMR experiments will provide the required information?
4. Which NMR instrument is best suited for those experiments?

In general, NMR experiments can provide the following information:

| | |
|---------------------|---|
| Nucleus environment | [Chemical Shift] |
| Quantitation | [Integration] |
| Neighboring nuclei | [Coupling constants] |
| Relaxation Times | [T1 and T2] |
| Correlation | [2D nmr: cosy, tocsy, hmqc, hmbc, hmhc etc.] |
| Stereo chemistry | [noe, noesy experiments] |
| Kinetics | [exsy experiments] |

HARDWARE:

Probes **CFI600** (BBO, SEF, TBI)
 RDQ400 (BBFO)
 DRY400 (BBO)
 UGI400 (QUAD)

SOFTWARE:

Windows XP platform **LOGIN:**
 Enter **group login name and password**
 Double click **topspin icon**
 type **lockdisp** open lock display window

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

FILE FORMAT:

example: **CFI600** **C0901ry / 90122ry1**
 RDQ400 **R0901ry / 90122ry1**
 DRY400 **Y0901ry / 90122ry1**
 UGI400 **U0901ry / 90122ry1**

ARCHIVING:

NMRs Data are archived regularly on the CDs by Instrumentation Facility personnel

SETUP: [sample installation, spin, lock and shimming]

| | | |
|---------|-----------------|--|
| press | lock | off |
| press | spin | off |
| press | lift | activate eject air |
| install | sample | 36mm [minimum] solvent depth is required |
| press | lift off | stop eject air |
| press | spin | on |

| | | | |
|--|------------------------|--|----------|
| type | edc | edit current data filename | |
| change | filename | format should be YMMDDID# | |
| change | expno | if necessary | |
| change | procno | if necessary | |
| type | bbo / sef / tbi | UGI400 click on experiment of interest | |
| type | bbfo | RDQ400 click on experiment of interest | |
| type | bbo | DRY400 click on experiment of interest | |
| type | quad | UGI400 click on experiment of interest | |
| choose | copy all | | |
| type | ii | initialize the hardware | |
| type | atma | enter a title for your experiment for CFI600, RDQ400 & DRY400 only. | B |
| type | lock | select solvent from the table | |
| adjust | lock power | if required | |
| adjust | lock gain | if required | |
| type | ts | for CFI600, RDQ400 & DRY400 only for UGI400 shim manually. | |
| press | autoshim | z¹, z², z³ shims will be optimized | |
| type | ased | acquisition parameters (or type eda) make changes if necessary | I |
| type | rga | automatic receiver gain determination | |
| type | ns | number of scans | I |
| type | expt | duration of the experiment | I |
| type | zg | to start acquisition | I |
| type | halt | to stop acquisition (do not use STOP) | I |
| set up other experiments (<i>if required</i>) for multizg operation at this stage | | | |
| type | iexpno | to create another experiment with same parameters recall a different set of parameter & enter title | |
| type | atma | automatic tuning for CFI600, RDQ400 & DRY400 only set expt (ns) and rga for each experiment before starting a series of experiments | |

type **multizg** to start a series of experiments on the same sample

PROCESSING:

| | | |
|--------------------|---|----------|
| dpa | only to check acquisition parameters | I |
| edp | processing parameters | B |
| lb | line broadening | |
| efp | em + ft + pk | |
| apk | phase correction (zero and first order) | I |
| abs | automatic baseline correction | |
| referencing | calibration | I |
| peak picking | | I |
| integration | | I |
| set plotting range | | I |
| print | select printer | |

COMMON COMMANDS:

| | |
|-----------------|---|
| a | go to acquisition window |
| abs | automatic baseline fix |
| ased | acquisition set up parameters |
| atma | automatic probe tuning |
| bbfo | parameter sets (RDQ400) |
| bbo | parameter sets (CFI600 & DRY400) |
| cy | vertical height for plot output |
| dpa | parameters for acquired data set |
| eda | acquisition parameters |
| edc | current parameters |
| edp | processing parameters |
| edte | variable temperature control |
| efp | em + ft + pk |
| expt | experiment time |
| halt | stop acquisition |
| iexpno | increase experiment number |
| iprocno | increase process number |
| ii | initialize hardware |
| lb | line broadening parameter |
| lockdisp | lock window |
| lock | automatic lock with choice of solvent |
| multizg | start a set of predefined experiments |
| ns | number of scans |
| print | output to printer |
| quad | parameter sets (UGI400) |
| rga | automatic receiver gain |
| rsh | recall a shim file |
| search | access archived data |
| sef | parameter sets (CFI600) |
| edti | write a title for current data set |
| stop | abort acquisition or wobble program |
| tbi | parameter sets (CFI600) |
| ts | automatic shimming using topshim |
| topspin | start NMR software |
| zg | zero memory and start acquisition |