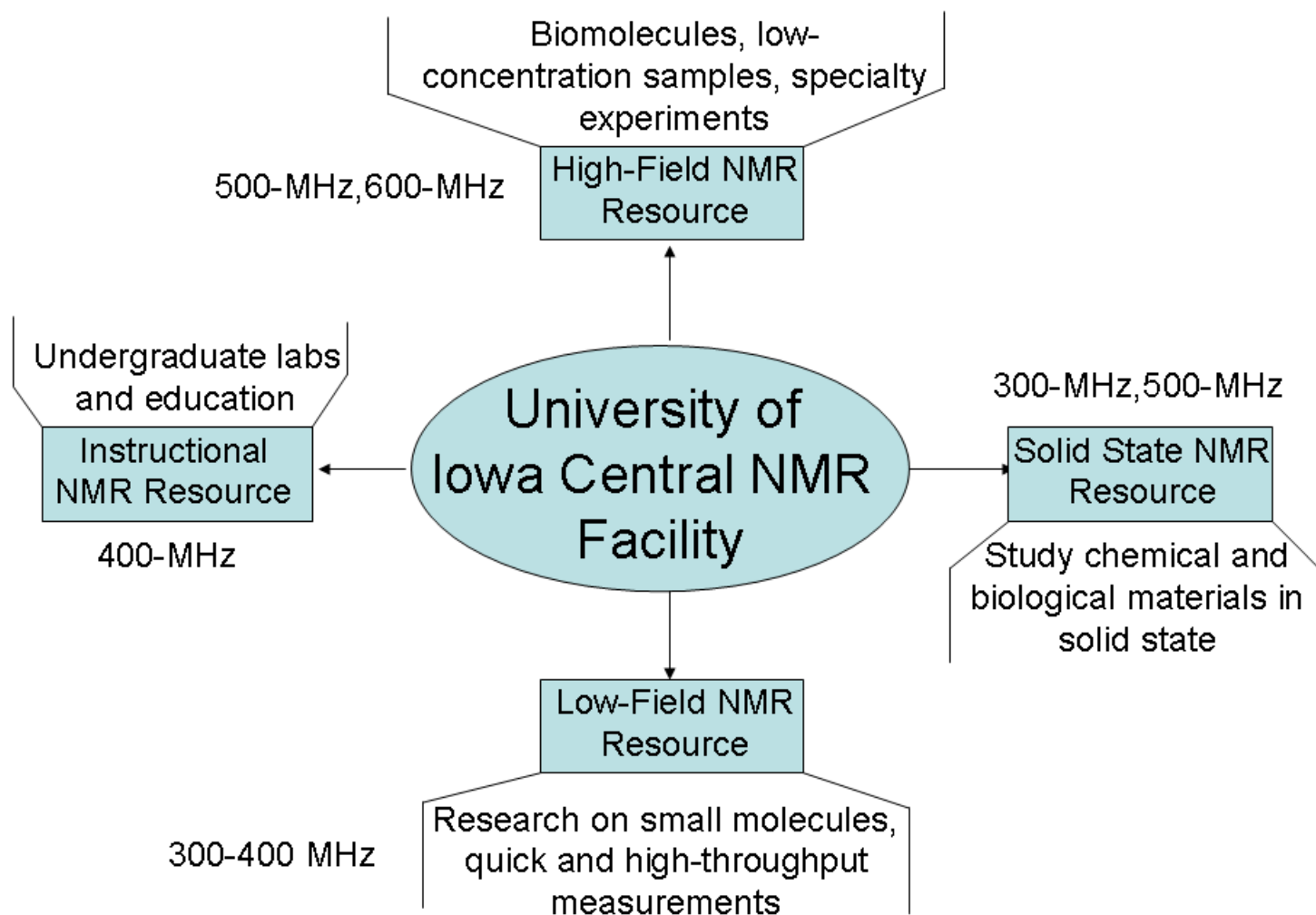


UI Central NMR Facility: General Information



Personnel: Santhana Velupillai, Fu Chen, Ryan Friedrich

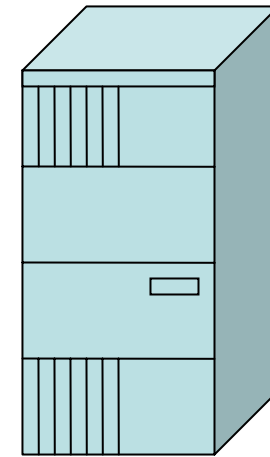
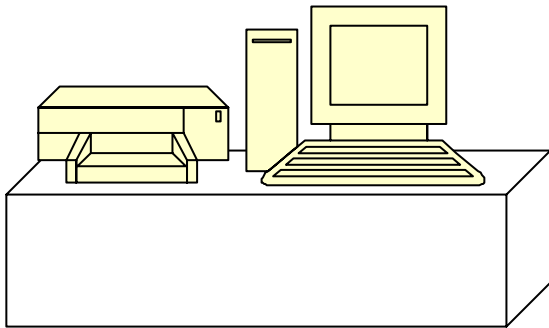
NMR Instruments: Similarities and Differences

Instrument/ Room	Frequency	Nature of Samples	Main Probe*	Additional Probes	Software
AV-300/ CB-W84	300 MHz	Solution	QNP/ ^1H - ^{13}C - ^{19}F - ^{31}P ; NTM, MS	DUAL/ ^1H - ^{13}C	TOPSPIN 1.3
DPX-300/ CB-W80	300 MHz	Solution	QNP/ ^1H - ^{13}C - ^{19}F - ^{31}P ; NTM; MS	DUAL/ ^1H - ^{13}C	TOPSPIN 1.3
FOUR-300/ CB-W275	300 MHz	Solution	DUAL/ ^1H - ^{13}C ; ATM, AGS	NONE	TOPSPIN 3.1
DRX-400/ CB-W84	400 MHz	Solution	BBO/Many Nuclei; MT, MS	BBI, Triple- Resonance	TOPSPIN 1.3
AVIII-400/ CB-W444A	400 MHz	Solution	BBFO/Many Nuclei; ATM, AGS	BBI	TOPSPIN 2.1
AV-500/ CB-W275	500 MHz	Solution	BBFO Plus/Many Nuclei; ATM, AGS	LC-NMR (Not Installed)	TOPSPIN 2.1
AVIII-600/ CB-W77	600 MHz	Solution	1.7mm micro/ ^1H - ^{13}C - ^{15}N /Many Nuclei; ATM, AGS	TBI, PABBO, BBI	TOPSPIN 3.0
WB-300/ CB-W85	300 MHz	Solids	CPMAS 7mm/ Many Nuclei; MTM	CPMAS 4mm/ Many Nuclei	TecMag
AVIII-500/ CB-W275	500 MHz	Solids	CPMAS 4mm/ Many Nuclei	CPMAS 2.5mm/Many Nuclei; HRMAS 4mm/ ^1H - ^{13}C ; Triple-Resonance	TOPSPIN 3.0

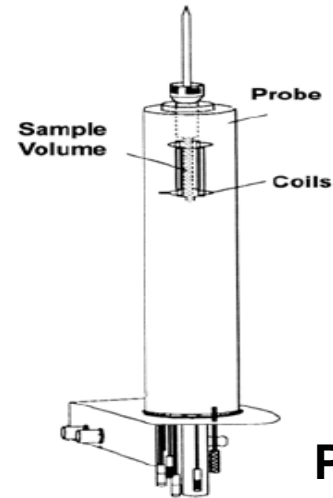
*NTM-No tuning and matching, MS-manual shimming, ATM-automatic tuning and matching, AGS- automatic gradient shimming, MTM-manual tuning and matching

NMR Equipment

Computer & Peripherals

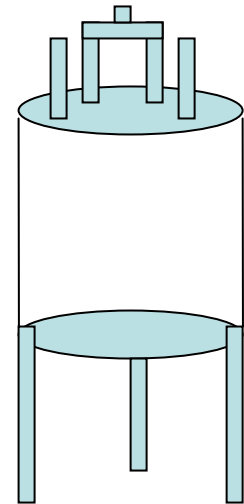


RF Console

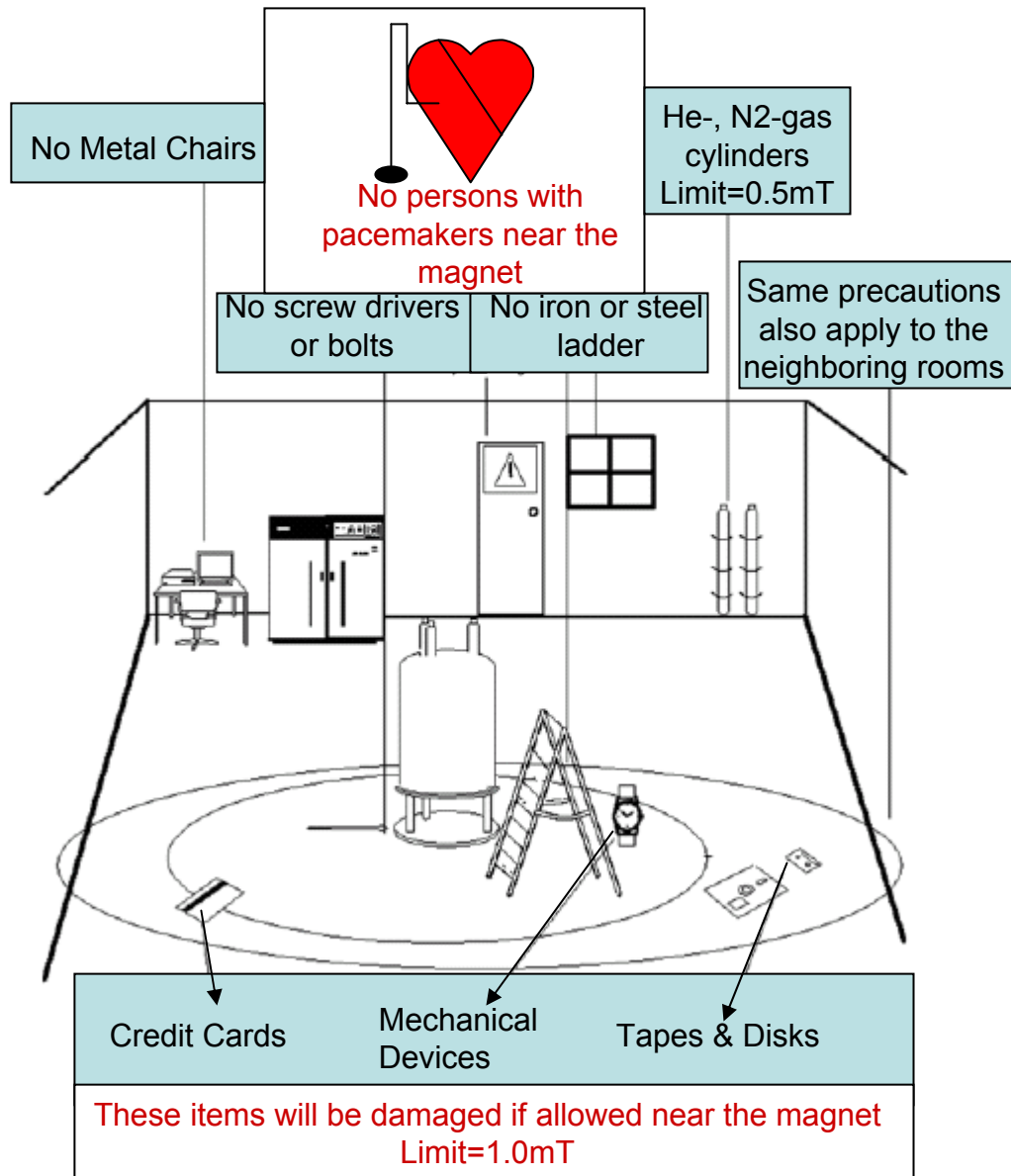


Probe

Magnet



Safety First !!!



- Magnet
 - Large attractive force on ferromagnetic objects
 - Large content of liquid nitrogen and helium
- Cryogenics
 - Possible magnet quench
- Electrical
 - Similar to any other laboratory equipment
- Chemical
 - Do not prepare your samples in the NMR laboratory

Hands-on NMR Spectroscopy: Av300

Logbook Entry: Policies

- Enter in the logbook before you start on the instrument
- Use military time format (e.g. 16:00 for 4:00pm)
- Write the account number legibly
- Include anything unusual (breakdowns errors, problems pertaining to spinning, shimming etc.) during the instrument operation in logbook entry
- Never use an instrument without making an entry in the logbook

IMPORTANT: NEVER GET ON AN INSTRUMENT IF YOU ARE NOT SPECIFICALLY TRAINED TO OPERATE THE INSTRUMENT BY ONE OF THE STAFF MEMBERS

Sample insertion: Step-by-Step

- Remove cap from NMR
- Open Topspin 1.3 program
- Drag 1H folder from your research group subdirectory (in the "NMR Data Browser" screen) to the "Bruker TOPSPIN 1.3" window
- Turn off LOCK on BSMS panel
- Turn off sample SPIN on BSMS panel if it is on (it should be off already if the standard sample is in the instrument)
- Turn on sample LIFT on BSMS panel (standard sample will float at the top of the magnet)

CAUTION: SAMPLE BREAKAGE RISK, MAKE SURE THE MAGNET CAP HAS BEEN TAKEN OFF THE MAGNET BEFORE TURNING ON THE SAMPLE LIFT

Sample insertion: Step-by-Step

- Take standard sample out of the magnet straight
- Replace your sample into the spinner; make sure to use the depth gauge
- Wipe the NMR tube and spinner with a kimwipe
- Insert sample straight into the magnet
- Turn off sample LIFT on BSMS panel

EXERCISE CAUTION WHEN REMOVING AND INSERTING SAMPLES IN THE MAGNET (STRAIGHT, NOT IN AN ANGLE)

Preparation for Data Collection: Step-by-Step

- Go to the "file" tab in the "Bruker TOPSPIN 1.3" window and choose "new"
- Name the file (do NOT use long name or any special characters), choose the exp # and solvent type. Click "ok"
- Type **rsh std.shim** in the "Bruker TOPSPIN 1.3" window
- Turn on SPIN on BSMS panel
- Click on the "lock display" icon in the "Bruker TOPSPIN 1.3" window
- Click on the "lock current sample" icon in the "lock display" window
- Double click on the solvent that you are using and wait for the message box in the "Bruker TOPSPIN 1.3" window to say "lock finished"

Preparation for Data Collection: Step-by-Step

- Turn on Z1 on BSMS panel and turn wheel to adjust signal to highest level
- Turn on Z2 on BSMS panel and turn wheel to adjust signal to highest level
- Repeat the last two steps until signal is at highest level possible (always end with adjusting Z1). If signal goes off screen, press the LOCK GAIN button on the BSMS panel and turn the wheel until you can see signal again then turn on Z1 again and continue shimming.
- Press STDBY button on BSMS panel

Data Acquisition Step-by-Step

- Type **ns** in "Bruker TOPSPIN 1.3" window (start with 16 scans)
- Type **ds** in "Bruker TOPSPIN 1.3" window and set it 0 (zero)
- Type **d1** in "Bruker TOPSPIN 1.3" window and set it to 0.01
- Type **ii** in "Bruker TOPSPIN 1.3" window
- Type **rga;zg;efp;apk** in "Bruker TOPSPIN 1.3" window and wait for spectrum to appear

Data Processing: Step-by-Step

- Click on the "spectrum calibration" icon in the "Bruker TOPSPIN 1.3" window and choose the solvent peak (e.g. set chloroform to 7.26)
- Click on the "interactive integration" icon in the "Bruker TOPSPIN 1.3" window, then click on the "define integral regions" icon on the submenu and choose the desired integration areas. Click on the "return, save changes" icon when finished
- Click on the "manual peak picking" icon in the "Bruker TOPSPIN 1.3" window and label the desired peaks and then click on the "return, save changes" icon
- Print the data

Finish Up: Step-by-Step

- Turn off SPIN and LOCK
- Turn on sample LIFT on BSMS panel (your sample will float at the top of the magnet)
- Take your sample out of the magnet
- Insert standard sample into the spinner; make sure to use the depth gauge
- Wipe the NMR tube and spinner with a kimwipe
- Insert the sample straight into the magnet
- Turn off sample LIFT on BSMS panel
- Replace cap on NMR

Finish Up: Step-by-Step

- Click on the "lock display" icon in the "Bruker TOPSPIN 1.3" window
- Click on the "lock current sample" icon in the "lock display" window
- Select D₂O (LOCK on D₂O)
- Enter the end time in the logbook
- Verify that the magnet bore is closed with cap

Data collection: ^{13}C , ^{31}P and ^{19}F

- Always run a quick ^1H spectrum before collecting data for any other nuclei
- Drag 13C, 31P or 19F (e.g. 13C for carbon-13 spectrum) folder from your research group subdirectory from the "NMR Data Browser" screen to the "Bruker TOPSPIN 1.3" window
- Type **ns** in "Bruker TOPSPIN 1.3" window; the value of this parameter depends upon the nucleus and sample concentration, and 128, 64 and 32 are common starting points for ^{13}C , ^{31}P and ^{19}F respectively
- Type **ds** in "Bruker TOPSPIN 1.3" window and set it to at least 4

Data Collection: ^{13}C , ^{31}P and ^{19}F

- Type **d1** in "Bruker TOPSPIN 1.3" window set it accordingly: ^{13}C , 1s; ^{31}P , 2s; ^{19}F , 5s
- Type **ii** in "Bruker TOPSPIN 1.3" window
- Type **rga;zg;efp;apk** in "Bruker TOPSPIN 1.3" window and wait for spectrum to appear
- If the signal-to-noise-ratio (SNR) is poor, set a larger value for **ns** and use the command sequence "**tr;efp**", to evaluate SNR, and stop the data collection using the command **halt**
- It is common to do overnight runs for ^{13}C , if the concentration is low (<5mg)

Data Processing: ^{13}C , ^{31}P and ^{19}F

- Data processing steps are very similar to those of ^1H data
- Tips for ^{13}C , ^{31}P and ^{19}F :
 - a) phase: it is quite often necessary **phc0** and **phc1** have to be adjusted iteratively
 - b) enter the command, **abs** (automatic baseline correction) to correct non-flat baseline

chkshm

ii
rga
ns 1
ds 0
zg
efp
apk

ii— Initialize Instrument; recommended when sample and or nucleus of observation changes

rga— Adjust receiver gain automatically

ns 1— Set the number of scans to 1

ds 0— Set dummy scans to 0

zg— Zero Go; Overwrite the existing data with the "new" data

efp— em+ft+pk; exponential window multiplication, Fourier transformation, followed by phase adjustment with "phc0" and "phc1"

apk— Automatic phase correction; may not be adequate for some data

runpro

zg
efp
apk

zg— Zero Go; Overwrite the existing data with the "new" data

efp— em+ft+pk; exponential window multiplication, Fourier transformation, followed by phase adjustment with "phc0" and "phc1"

apk— Automatic phase correction; may not be adequate for some data

Hands-on NMR Spectroscopy: DPX-300

Operation of the Spectrometer

- Logbook entry (same as Av300)
- Sample Insertion: Step-by-Step
 - the standard file name is 1H-QNP, in
stead of 1H (13C, 31P, and 19F are different
accordingly)
- Preparation for Data Collection: Step-by-Step
(same as Av300)
- Data Acquisition: Step-by-Step (same as Av300)
- Data processing: Step-by-Step (same as Av300)
- Finish-up: Step-by-Step (same as Av300)