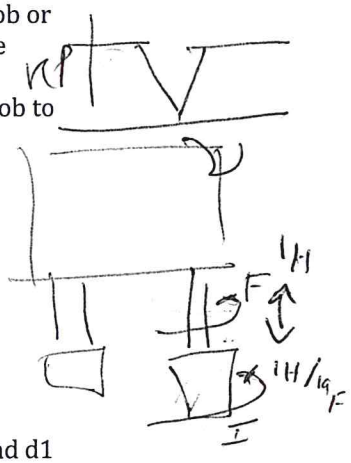
NMR Training

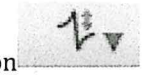
1) How and where to sign-up for instrumentation time **http://argenta2.rd.unr.edu**
    a. go over time rules listed on argenta2
    b. make sure netid is prefixed with **unr** when logging in to the instrument reservation system
2) Go over what nuclei can typically be ran on each NMR
    a. 400-MR $^{1}H/^{19}F/^{31}P$
    b. V400/~~V500~~ $^{1}H/^{19}F/^{13}C$
3) Go over magnet safety
    a. No magnetic items in the NMR room
    b. No ferromagnetic in NMR room
    c. Keep items like wallets and cellphone at least six feet away from the magnet
    d. No iPhone/ipads in the room during Helium fills (it has been shown that helium can penetrate the system clocks and render the devices dead)
    e. Inform the NMR staff if you have medical implants
4) Putting a sample in a turbine and correctly using the depth gauge
    a. Make sure you have at least three fingers of solvent or 760 microliters
5) Putting the sample/turbine into the magnet
    a. switch on top of magnet
    b. eject or insert button in VNMRJ parameters panel
    c. e or i in the command line
6) Go over the parts of the VNMRJ interface
    a. Experiments you'll typically use are: Proton, Carbon, gCOSY, ROESY, TOCSY, gHSQCAD, gHMBCAD
7) How to clear the automation queue in VNMRJ – makes a new folder to store data in
    a. Automation -> Background Acquisition -> New Background Run
8) How to load in the default set of shims in VNMRJ do this every time you run the NMR
    a. Tools -> Locator... ->double click on walkup_lk_d2o
    b. Close locator window and then type su followed by enter into the command line
9) How to tune the probe on the 400-mr and ~~v500~~: please note never to force the knob or barrel of the tuning stick. Tuning is a process of setting the NMR probe to the same frequency and impedance of the spectrometer
    a. Tune the barrel on the capacitor stick to adjust the frequency, turn the knob to adjust the impedance; note the barrel and knob are interactive
    b. In the command line type mtune
    c. Select the rf channel and nucleus you want to tune
        i. Channel 1: ^{1}H or ^{19}F
        ii. Channel 2: ^{13}C, ^{31}P, ^{15}N
    d. Click Start Probe tune button
    e. Click Autoscale button
    f. Adjust tuning knob and barrel on probe to get dip at correct frequency
    g. Click Stop Probe tune button
    h. Click Quit button



10) How to setup a ^{1}H 1D proton experiment: including when and why to change nt and d1
    a. New study then fill out sample name (letters, numbers, underscore, and dash only) and select the deuterated solvent you're working in
    b. Double click on the experiment you want, for example Proton, in the Study Queue and then adjust nt and d1
    c. nt is adjusted based off concentration (about 1 to 5 mM for ^{1}H, 50 to 100 mM for ^{13}C for quick runs)
    d. d1 is adjusted if your integrations do not come out to be whole numbers
11) How to submit a sample to the background
12) How to run multiple experiments on one sample
13) How to delete an experiment before submission

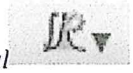
14) How to assess the quality of the shims (nl, dres on TMS or residual solvent proton)
    a. the TMS peak should be a sharp symmetrical peak with a linewidth of less than 1 Hz nonspinning and less than 0.5 Hz spinning at 20 Hz.
15) Manual locking of the NMR
    a. make sure Lock checkbox is unchecked
    b. click lock scan
    c. adjust z0 to be on resonance (sine wave becomes plateau)
    d. adjust lock gain and phase so that the lock level value is stable and between 20 and 70 – a minor fluctuation of a few units is fine
    e. do not adjust the lock phase
16) Manual shimming
    a. generally, just adjust z1 through z7
    b. if any shim is changes the value of the lock level significantly then start over from z1 again
17) How and where to save data to argenta
    a. /mnt/argenta/mr400/data/*PI*/*you*
18) How to load data in Mestrenova

    a. click the open document button
    b. navigate to your data folder on argenta
    c. open the FID file in your data folder
19) How to phase spectra in Mestrenova – ensuring the baseline is symmetrical around your peak



    a. click on the dropdown arrow on the phase button
    b. select manual correction
    c. left and right drag in the purple box until the spectrum is properly phasesd
20) How to reference spectra in Mestrenova: both off TMS and the residual solvent proton

    a. click on the dropdown arrow of the reference button and select *Reference*
    b. TMS peak – mouse over the peak until the peak turns red and click.
        i. Type 0 in the *New Shift* box
    c. Residual solvent peak
21) How to integrate the peaks in Mestrenova



    a. click on the dropdown arrow of the integrate button and select *Manual*
    b. drag over the peaks individually that you want to integrate
    c. set a specific integral to a known value
        i. mouse over the integral until it glows, then right click and select Edit Integral...
        ii. type in the new value in the **Normalized** box
22) How to label peaks with their chemical shifts in Mestrenova
    a. click on the dropdown arrow of the Peak Picking button and select *Peak by Peak*

    b. Mouse over the peaks that you want to label until the peak glows and then click
23) How to View tables for NMR data
    a. View → Tables...
24) How to measure coupling Constants
    a. Use crosshair tool and drag from the apex of one peak to the apex of another

25) How to predict chemical shifts based off a chemdraw structure in Mestrenova
    a. Draw molecule in Chemdraw
    b. Copy and paste molecule into a blank page in Mestrenova
    c. In Mestrenova Predict → 1H Spectrum
26) How to change display parameters in Mestrenova: for example spectrum color, font size, etc.....
27) How to save processed spectra in Mestrenova
28) How to print processed spectra in Mestrenova
29) How to copy and paste spectra in Mestrenova
30) How to create your data folders on argenta
31) Fill out instrument problem sheet on argenta
32) No food or drink in the lab
33) Point out the oxygen sensor in the lab. If it goes off leave the room immediately
34) In the event the building alarm goes off. Leave the room immediately
35) Run three checkout samples: $CHCl_3$ in acetone (manual shim less than 0.5 Hz spinning, less than 1 Hz nonspinning), low volume D_2O, DMSO
36) How to manually run a sample
    a. Forget about the study queue and don't use it
    b. Load in the parameters for a Proton by clicking Proton in the available experiments
    c. Tune the probe using the mtune macro
    d. Manually lock the NMR
    e. Manually shim the NMR
    f. Adjust nt and d1
    g. Type **ga** to go and acquire the date
    h. Save your data to argenta
37) You must run your first five samples in the presence of the SIL staff
    a. Sample 1 date: SS 1/30
    b. Sample 2 date: SS 1/30
    c. Sample 3 date:_____
    d. Sample 4 date:_____
    e. Sample 5 date:_____

_____

Trainee Signature

_____

Trainee Printed Name

Trainee Email: _____

Date Training completed: _____

_____

Trainer Signature

_____

Trainer Printed Name