Varian VNMRS-500 MHz NMR Operating Procedures

1. Place **everything magnetic** (**keys, tools**, etc.) or media, that can be erased by magnetic fields (**ATM-cards, credit cards, USB-keys, cell-phones**) on the table next to the computer and **do not approach the instrument having any of these on you! Also don't approach any big pieces of magnetic metals (for example, chair, heavy tools) to NMR magnets, it can destroy very expensive magnets!**

2. Login

   - LINUX operating system

     User: [supervisor name]
     password: [you should have obtained it from your supervisor/research group]

3. Double-Click on Vnmrj icon in upper left area of the desktop

4. Click on *eject* in the START/STANDARD TAB to eject the standard sample and take it out of the instrument

5. Exchange the standard-tube with your own sample tube.

   - Make sure to employ the sample-depth adjustment tool to verify the correct position of your tube in the spinner

   - Wipe the lower part of the NMR-tube clean with a Kimwipe or paper-towel

   - This is also a good opportunity to check that you have enough solvent in your tube. The visibly part of your tube in the tool should be filled with liquid

6. Place the spinner with your sample in the instrument and click *insert* in the START/STANDARD TAB

7. Click on *Tools* in the upper line of the software and select *Locator*

   - Click on the MAGNIFYING GLASS and select *sort shimsets by probe and date*

   - Double-Click the newest shimset.

   - Close the locator window

8. Select the correct solvent for your sample in the START/STANDARD TAB

9. Click on *New Study* in the STUDY QUEUE PANEL

10. Enter a name for your sample in the field *Sample* in the START/STANDARD TAB

11. Uncheck the box *Autoplot* if you want to change details on your spectrum before print-out

12. Make sure *Shim* are checked

13. Don't check *Tune* for proton experiment (do it only for 13C, 31P, 19F or 2D NMR)

14. Select *Proton* from the EXPERIMENT SELECTOR PANEL, it will appear in the STUDY QUEUE PANEL

15. Click on green button *Submit* below the STUDY QUEUE PANEL and wait for message "Tune Probe Now / Start Acquisition with Acquire button" appeared

16. Then click on the ACQUIRE TAB below the black window

- Adjust S*pectral Width [ppm]* to your expected spectrum (normally 9.5 – -0.5)
- Select *Number of Scans* dependent on your sample amount (4-8 mg – 8 scans)
- If integration is important change *Relaxation Delay* to 5

17. Click on green button *Acquire* and wait until experiment will be finished. (If there will be red message "pneumatic pressure error" – go to START/SPIN-TEMP panel and click on button "Reset Pneumatic Router").

18. Then click on the PROCESS TAB below the black window and click on green button *Autoprocess* to see spectrum

19. Click on the 8th symbol (integration line) from the top on the right side of the screen. This is the integral button and allows you to switch through showing full integrals, showing partial integrals and no integrals. The symbol below (9th symbol) changes depending on the choice you made above
    - Click the 8th symbol until the 9th shows a red X above the integration line. Now click the 9th and you reset the integration
    - Click again on the 8th symbol until the 9th shows you a pair of scissors
    - Click on the scissors and then use left moue clicks in the spectrum to cut the integrals the way you want them
    - Now click on the first symbol showing a cursor line. This allows you again to place the red cursor line any place in the spectrum with a left click. A right click adds a second line
    - Place the left click cursor line on an integral whose value you know and click on the PROCESS TAB. You should be in PROCESS TAB/DEFAULT. Enter the integral value in the *Set Norm to* field and click on *Set Norm to*
20. Click on the 4th symbol from the bottom (orange line) to set a threshold for peak finding in the spectrum
21. Now you should be ready to print the spectrum under PROCESS TAB/PLOT by selecting Plot Spectrum, then Plot Spectrum Scale, Plot Peak Frequencies, Plot Integral Values, Plot Parameters and finally clicking on Plot page
22. Also save the spectrum by clicking on *File/Save as* in the menu bar on the top. You should automatically be in the home folder of your research group
23. When you are done repeat steps 4 to 6 and this time replace your sample with the standard-tube
24. Close Vnmrj NMR software at the end of your experiments. The last step is clicking on System in the topmost bar belonging to the LINUX-system and log out your user

MAKE SURE TO WRITE YOUR NAME AND EXPERIMENT INTO THE LOG-BOOK !

ps. For 13C NMR experiment select *Carbon* from EXPERIMENT SELECTOR PANEL at step 14, for COSY select (HH)gCOSY, for 31P or 19F NMR go to Liquids/Std 1D/*Phosphorous* or *Fluorine*, for NOESY– to Liquids/(HH)Homo 2D/*NOESY*, etc.

Varian INOVA-300 MHz NMR Operating Procedures

The Varian 300 MHz operation is similar to Varian 500 MHz operation but **cannot be used with full automation** (*New Study, Submit, Find Z0, Gradient Autoshim buttons* will not work properly!)

So, go by steps 1-7 (preparation) and 18-24 (processing/plot) of the 500 MHz protocol, but instead of steps 8-17 (for lock, shimming and acquisition), do following:

1. *For lock*, go to START/SHIMS panel. First step is to increase or decrease your **Z0** to find signal of lock.
2. *For shimming*, you will need to modify two parameters **Z1C and Z2C**. As you did for the lock, add +/- 1 to see the effects on your lock. The goal is to maximize the lock for your sample. Once your lock signal is the maximum it can reach, you are ready to run your sample.
3. Select the correct solvent for your sample in the START/STANDARD TAB
4. Enter a name for your sample in the field *Sample* in the START/STANDARD TAB
5. Select *Proton* from the EXPERIMENT SELECTOR PANEL
6. Then click on the ACQUIRE TAB below the black window
   - Adjust S*pectral Width [ppm]* to your expected spectrum
   - Select *Number of Scans* dependent on your sample amount
   - If integration is important change *Relaxation Delay* to 5
7. Click on green button *Acquire* and wait until experiment will be finished.
8. Save the spectrum by clicking on *File/Save as* in the menu bar on the top

Bruker Fourier-300 MHz NMR Operating Procedures

It is easy to use Bruker NMR instrument with the sample changer:

1. Wipe the lower part of the NMR-tube clean with a Kimwipe or paper-towel
2. Make sure to employ the sample-depth adjustment tool to verify the correct position of your tube in the spinner.
3. Place the spinner with your sample in the 16 position sample changer
4. Click on *Change User* button in the ICON-NMR software and login as your lab user
5. Select lines corresponding position of your samples, choose the name of directory for your experiment recording, number of experiment in this directory, solvent, experiment (usually, proton), title, etc.
6. *Submit* your experiments, each proton NMR experiment with full automation (sample load, rotation, solvent lock, shimming, acquisition, processing) will run ~7 minutes
7. Once experiment is finished, login as *Processing Only* user (with no password). Go to your Windows directory and copy your NMR spectra to your USB key.
8. Remove your samples (you can spin the sample changer slowly) and delete lines for finished experiments
9. **Don't close the TopSpin or ICON-NMR software** after your experiments

HOW TO REMOVE STUCK SAMPLE

If you have red color at the sample changer, and your sample is not at the top, it means the sample was not ejected.

1. Turn off the blue button on the sample changer
2. Carefully remove the 16 position sample changer from the magnet
3. Go to *Processing Only* account
4. Open the main TopSpin 3.2 window (see icons at the bottom of Windows 7)
5. Go to Acquire/Sample menu at the top
6. Turn on sample lift air (**ej**)
7. Your sample will be ejected, and you can remove it now
8. Turn off sample lift air (**ij**)
9. Don't close main TopSpin 3.2 window, just minimize it, and open ICON-NMR window now
10. Place the 16 position sample changer back on the magnet, spin it slowly to find proper position of the sample changer
11. Turn on the blue button on the sample changer and wait until light will be green
12. Run your new NMR experiment in the regular way