Standard Operating Procedure
^{19}F-NMR

Anasazi 90 MHZ NMR

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Sign-in to the NMR log book.

Georgia Gwinnett College

Shimming the instrument using DI water.

1) A) Load DI water standard sample (labeled as H_2O). B) Make sure to insert the tube halfway in the Teflon holder as shown. C) Note that the sample level needs to be up to the 'black mark' on the block.

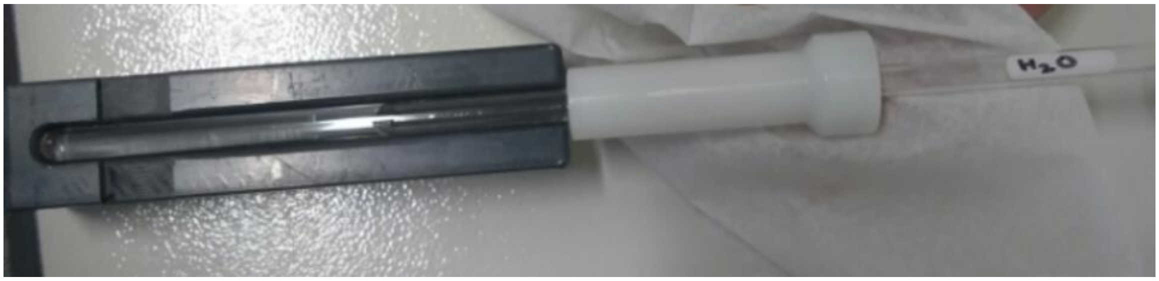

Figure 1. NMR tube inside the block. Measure the correct depth by slightly against the block. Don't touch the Teflon holder using bare hands. Always use Kim wipe.

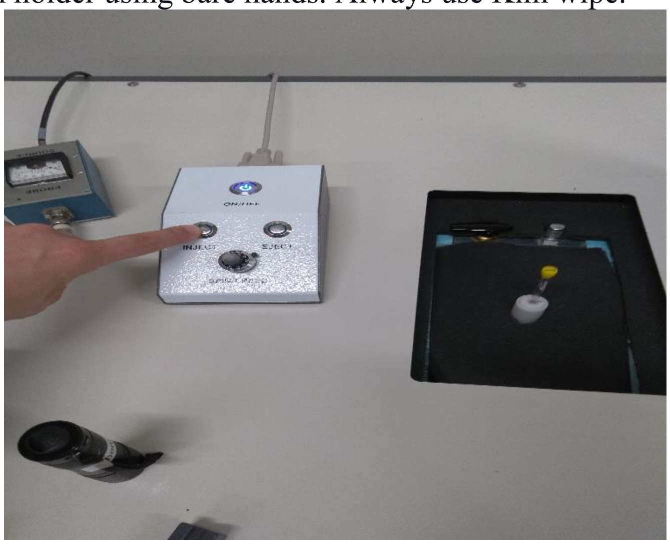

Figure 2. Insert the NMR tube with the Teflon holder (see on the right side of the figure) in the sample compartment. If the air is turned on you will see the NMR tube will bobble on the sample compartment. Click and hold the insert button for 15 seconds. Check the NMR tube is spinning using a light. You may draw a line across the NMR cap to easily see whether the NMR tube is spinning.

2) Open the NMR program (aii) on the computer

Figure 3. NMR software aii on the computer and NUTS program (NMR analyzing program)

3) Choose H1 nucleus (by default its program is set as H1 nucleus). Once you selected the nucleus you will see H1 on the bottom left side of the screen (see figure 5).

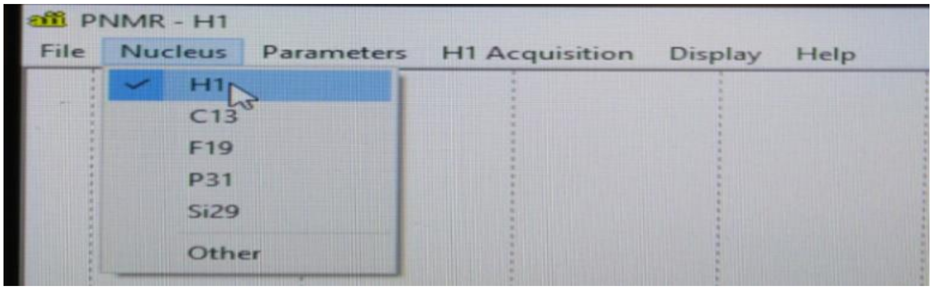

Figure 4. NMR aii menu, choose the nucleus H1.

4) Type ‘prep’ on the keyboard and press ENTER.

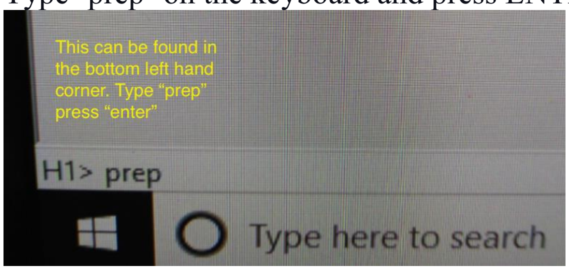

Figure 5. The appearance of the aii software after the selection of H1 nucleus and typing ‘prep’

5) NMR will start shimming (left side bottom of the screen will change green, indicating that that shimming process is in progress). After few minutes (or seconds) the left side bottom of the screen will change to yellow with an ATTENTION message about the spin rate (see Figure 6).

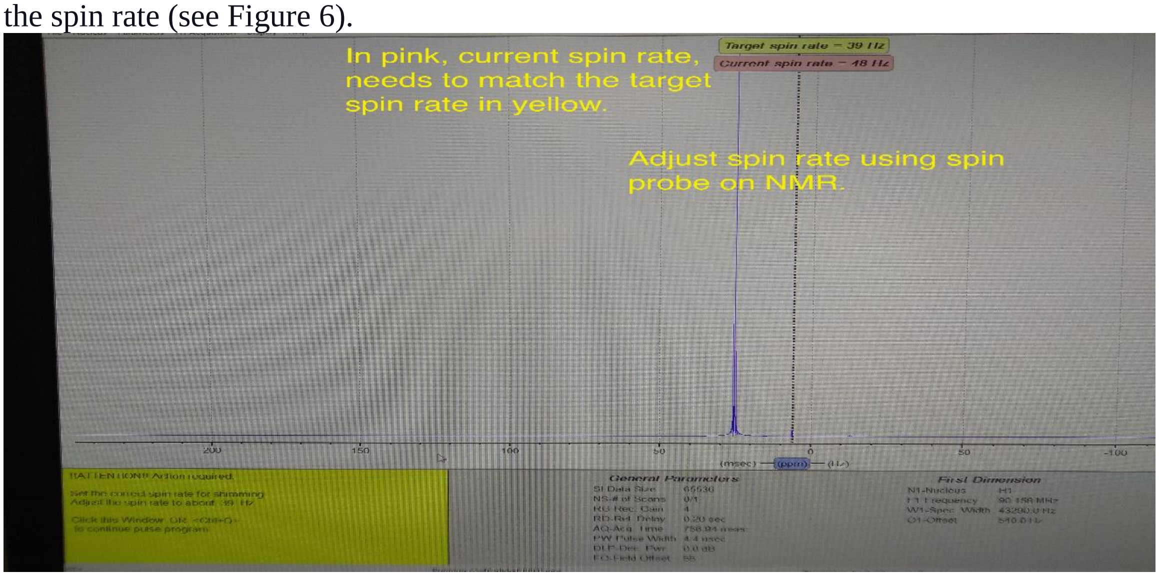

Figure 6. aii screen after the ATTN message, Target spin rate is 39 HZ (will appear in yellow) and current spin rate will appear in pink.

6) Adjust the spin rate if needed (**note: If the target spin rate is 39-40Hz and highlighted in green skip this step**) by turning the knob (decrease-counter clockwise and increase clockwise) as shown in Figure 7. Once the target spin rate is reached the pink color of the current spin rate will change to green (Figure 8). At that time lock the

spin rate by moving the black bar on the spin speed knob towards the right (see figure 7).

Figure 7. The control box has a knob to control the spin speed.

Figure 8. The current spin rate reached the targeted spin rate.

7) Click 'CNTRL Q' or click on the left bottom of the screen that is yellow (see figure 8). The screen will change as Figure 9. Wait for 3-5 minutes till the NMR complete the shimming process. Once the shimming process is complete the left bottom corner will change from green to grey.

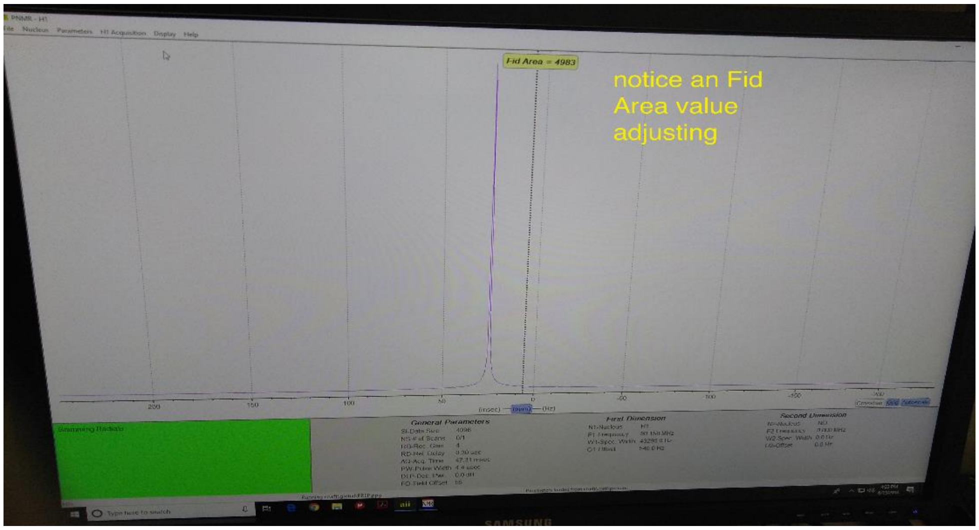

Figure 9. aii software screen showing the Shimming process in progress.

8) After the left corner changes from green to grey, the shimming process is complete. Remove the DI water sample from the sample chamber by pressing the ‘eject’ on the control box.
9) Remove the DI water sample from the Teflon holder (don’t touch the Teflon holder using bare hands, always use Kim wipe).

Recording ^{19}F NMR of a sample

10) Insert standard Fluorine sample (1% DCDBFE + 10% $CClF_3$ + 6 % TMS in acetone) into the Teflon holder and inject it into the NMR sample chamber as described in step 1.
11) Change the nucleus to F19 on aii computer screen (Figure 10)

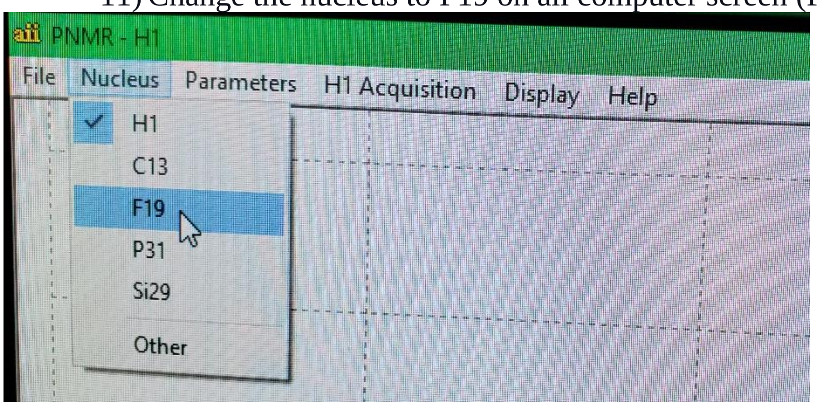

Figure 10. aii software screen showing changing to F19 nucleus.

12) Type F19tune and enter. Right, bottom of the screen will change from grey to yellow.
13) Remove the H probe located on the left side the NMR magnet. See Figure 11.

Figure 11. H probe

14) Connect to the source (it will be on the top of the NMR magnet, need to remove the cap). See Figure 12.

Figure 12. Probe source

15) Open the sample chamber, remove the grey sponge (see figure 13) and rotate probe marked ‘H’ clockwise using the red rod (see Figure 14) slowly to show the reading on the source to zero microamperes.

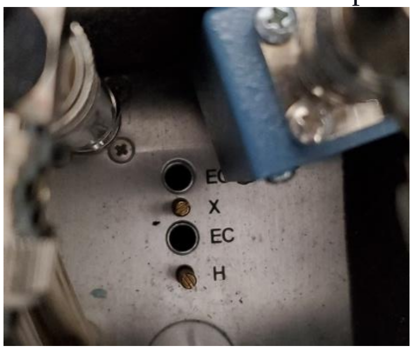

Figure 13. Inside the sample chamber.

Figure 14. Red rod for tuning the probe

16) Once the reading on the source is zero milli amperes, disconnect the H probe from the source (connect the cap), and connect it back to the original place. See figure 11.
17) Click enter
18) After the right-side corner of the aii screen change to grey, type F90cal
19) Type shim
20) Change the scan as needed
21) Type ZG. Immediately following the ZG protocol, a save menu will appear, Save the file in to correct folder with name, date, etc.

Analysis of the spectrum

22) Click NUTS program a screen will appear and click OK. ('NMR' on Figure 3 next to the aii program)
23) Type A1
24) Type AP
25) Type 'ID', an integral will appear on the screen above the spectrum. Move left side arrows to move or adjust the integral.
26) Bring the cursor next to the peak to be integrated, double click the left mouse button to appear green line (a red line will appear on the first click) move the cursor at the end of the peak, and single-click the left mouse button. Repeat this for all other peaks.
27) After integrating all peaks bring the cursor to the smallest peak. Click the left mouse button and the letter 'V' simultaneously. Type 1 in the highlighted blue box and click 'OK'.
28) Press 'enter' to exit from integration. Click 'CNTRL' I.
29) Type 'PP' for peak pick. If all peaks are not selected by the software, type 'DP' to manually pick the peaks. Bring the cursor to the peak that is not selected and then press enter.
30) Click print to print the spectrum
31) Click print page
32) Open paint in Microsoft programs from the control panel on the bottom left of the screen
33) CNTRL V
34) Export the file full ASC export.
35) Remove the sample and insert DI water sample into the sample chamber.
36) Complete the logbook.

To change back to ^{1}H Probe

37) Inject Propyl benzoate standard.
38) On aii screen, type H1tune and enter.
39) Follow steps 13 and 14. Open the sample chamber, remove the grey sponge (see figure 13) and rotate probe marked 'H' counter clockwise using the red rod (see Figure 14) slowly to show the reading on the source to zero microamperes.
40) Once the reading on the source is zero milli amperes, disconnect the H probe from the source (connect the cap), and connect it back to the original place. See figure 11.
41) Type H90cal and enter.
42) Type Shim.
43) Complete the logbook.