Standard Operating Procedure
^{1}H-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.

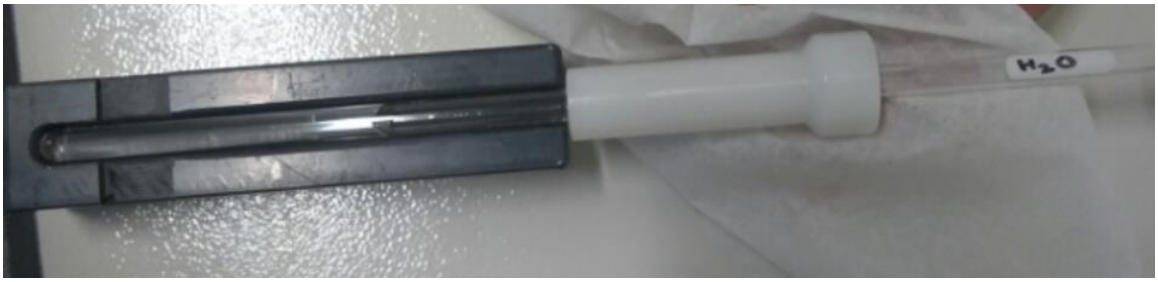

Figure 1. NMR tube inside the block. Measure the correct depth by slightly against the block. Don't touch the Teflon holder using bare hands. Always use Kim wipe.

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

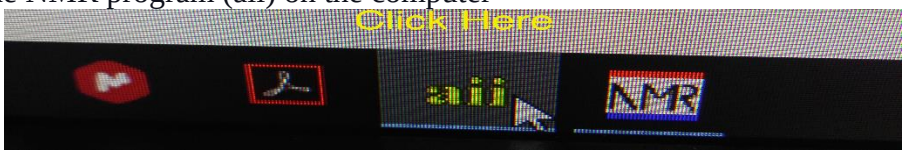

Figure 3. NMR software aii on the computer and NUTS program (NMR analyzing program)

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).

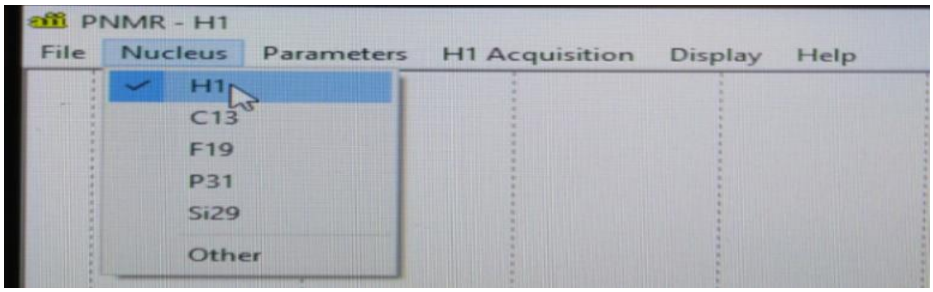

Figure 4. NMR aii menu, choose the nucleus H1.

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

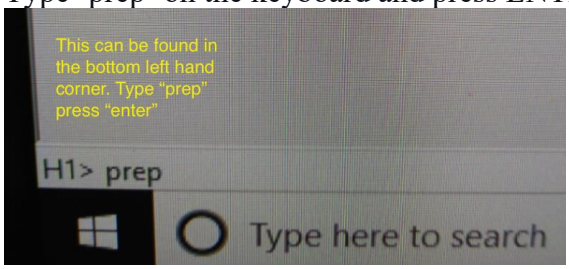

Figure 5. The appearance of the aii software after the selection of H1 nucleus and typing ‘prep’

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).

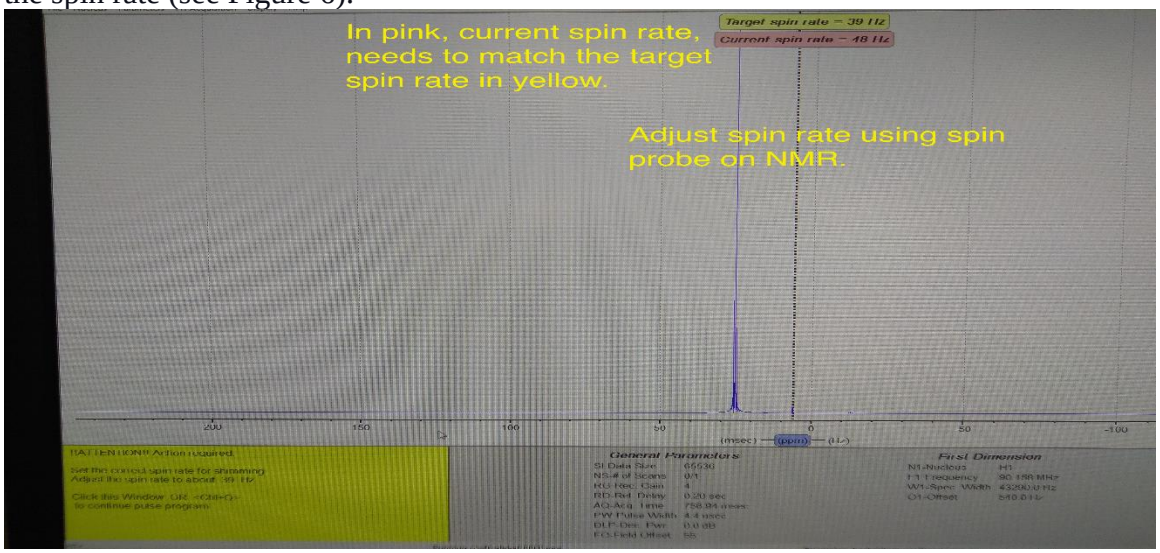

Figure 6. aii screen after the ATTN message, Target spin rate is 39 HZ (will appear in yellow) and current spin rate will appear in pink.

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.

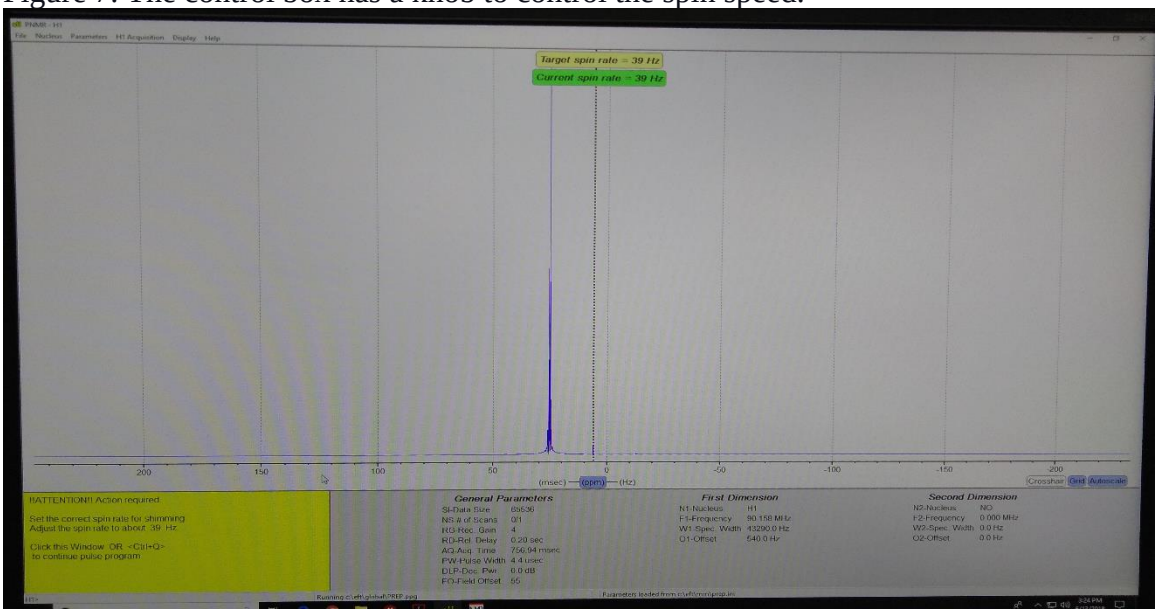

Figure 8. The current spin rate reached the targeted spin rate.

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.

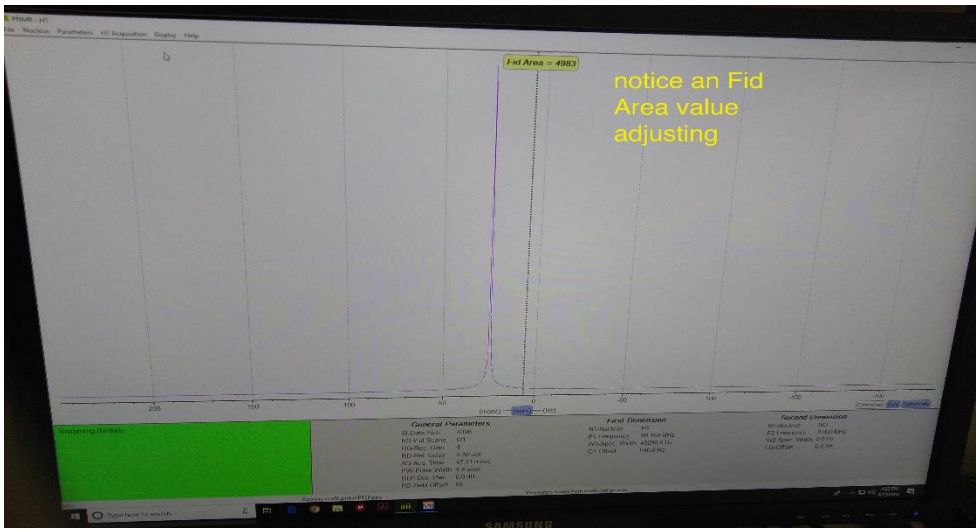

Figure 9. aii software screen showing the Shimming process in progress.

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 ^{1}H NMR of a sample

10) Insert the sample into the Teflon holder and inject it into the NMR sample chamber as described in step 1.
11) On the aii program screen from the top-down menu, click 'parameters' and select 'dilute sample ini' See Figure 10.

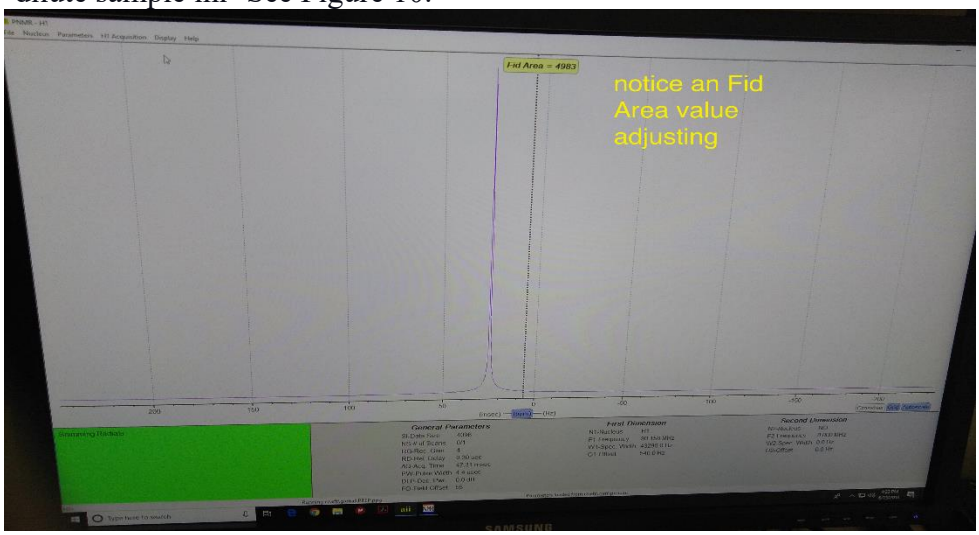

Figure 10. aii screen to select 'dilute sample ini'

12) From the top down menu on the aii screen select SHIM-optimize field homogeneity. See Figure 11.

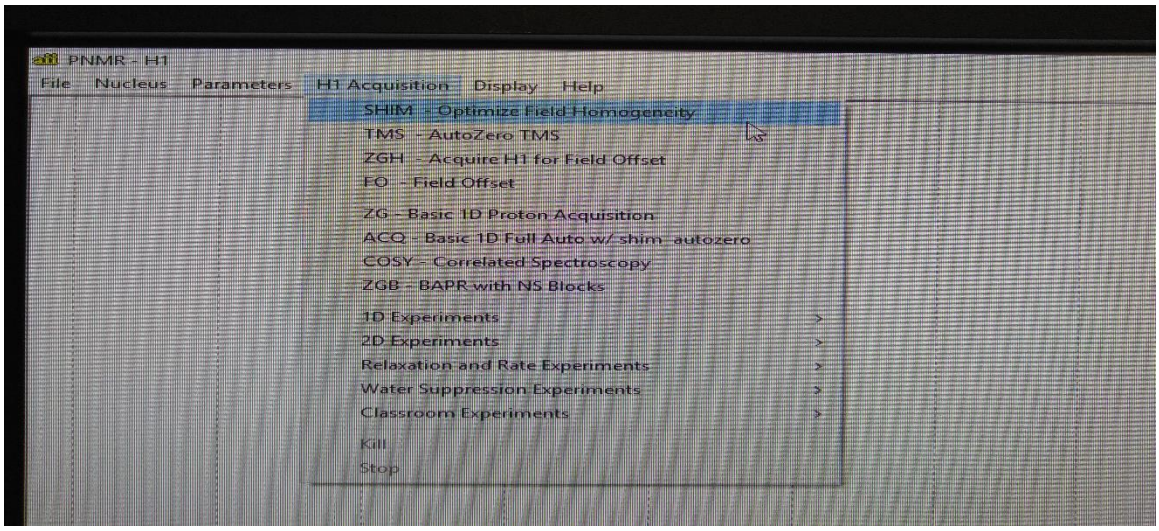

Figure 11. aii screen showing a selection of shim optimize field homogeneity.

13) A pop screen will appear asking to select SHIM or AUTO ZERO. Select SHIM (Auto zero can be adjusted manually when analyzing the spectrum).
14) After the left corner change from Green to Grey from the aii screen menu, click "ZG-Basic 1D proton acquisition". See Figure 12.

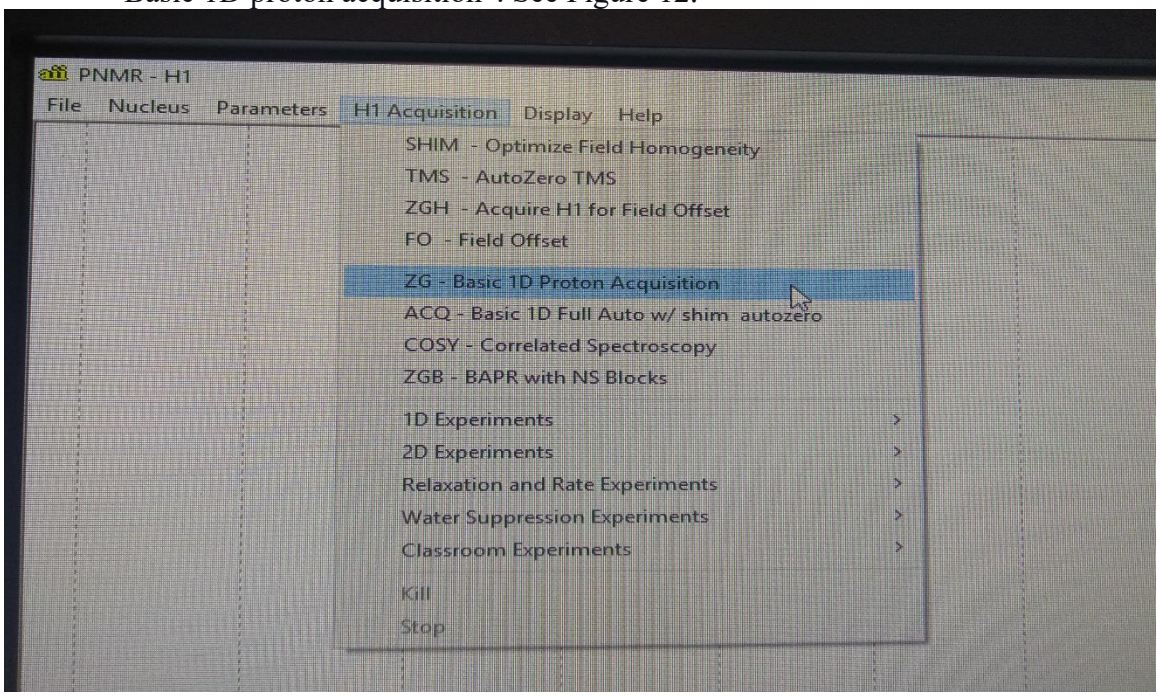

Figure 12. aii screen showing the selection of ZG-Basic 1D proton acquisition.

15) Immediately following the ZG protocol, a save menu will appear as shown in Figure 13. Save the incorrect file with name, date, etc.

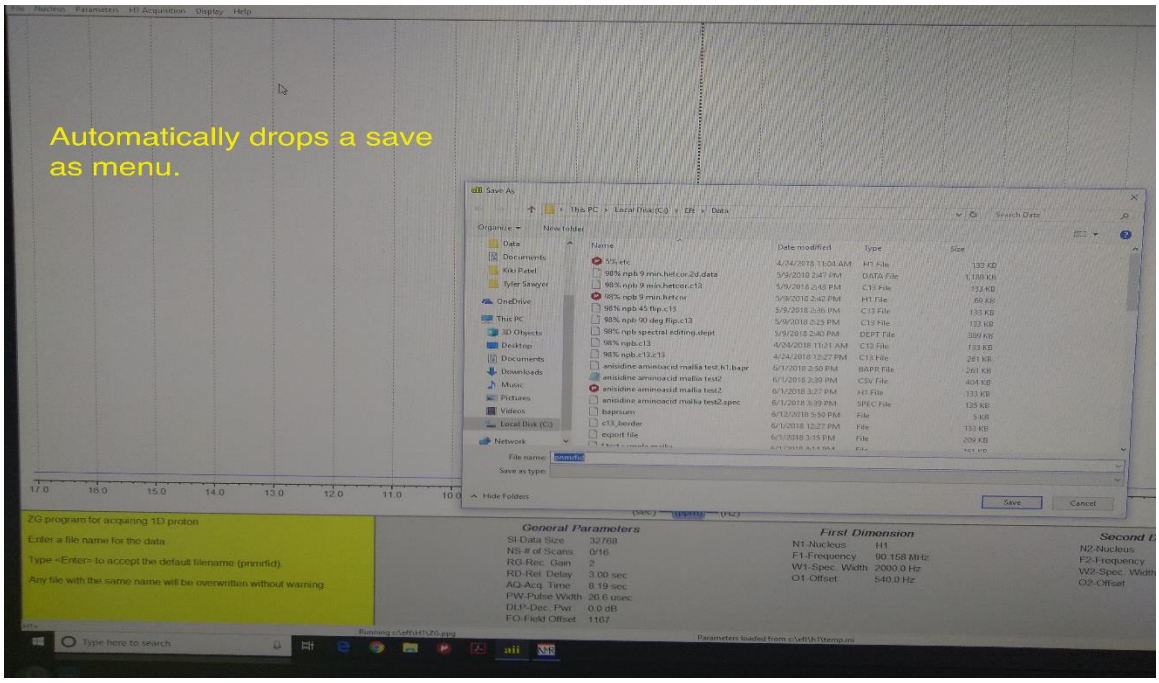

Figure 13. aii screen showing the pop-up regarding saving of the file.

16) Don't touch the keyboard or screen if there is a green highlight on the left bottom side of the screen. When the green changes to grey on the left bottom side of the screen the measurement is completed.

Analysis of the spectrum

17) Click NUTS program a screen will appear and click OK. ('NMR' on Figure 3 next to the aii program)
18) Click 'CNTRL' and F2 together, and open the file from the computer and click 'OK'.
19) A) Double click the left mouse button, to open the zoom mode (you will a cross wire cursor on the screen). B) Single-click the left mouse button and drag the highlighter over the peaks of interest (left to right). C) Click once on the right mouse button to select the shaded region. D) Press 'enter' to exit from zoom (cross wire cursor will disappear). (See Figures 14 and 15).

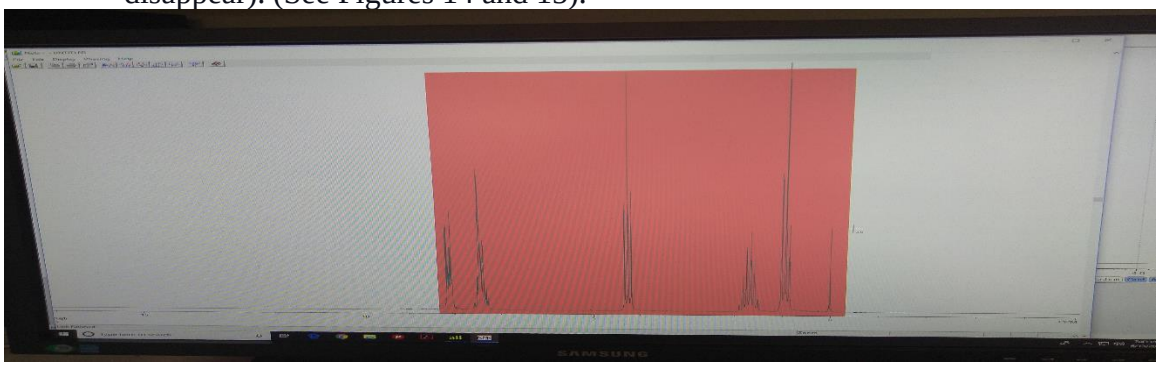

Figure 14. NUTS screen showing the zoom-in highlighted peaks.

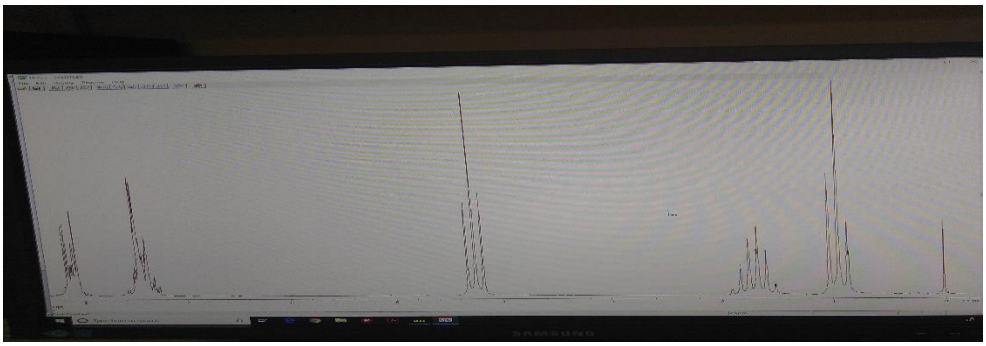

Figure 15. NUTS screen showing zoomed-in NMR peaks.

20) Move the cursor to the top of the most right side peak (TMS peak), a) left-click, and type the letter 'O' simultaneously. A Box will pop up, put 'zero' into the blue highlighted box, and press OK. See Figure 16.

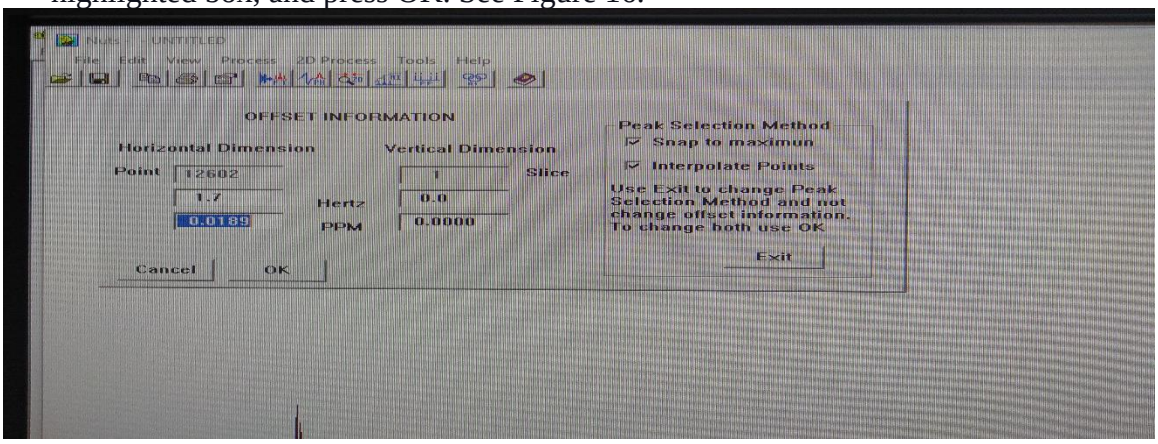

Figure 16. Selecting the reference.

21) Type 'ID', an integral will appear on the screen above the spectrum. Move left side arrows to move or adjust the integral. See Figure 17.

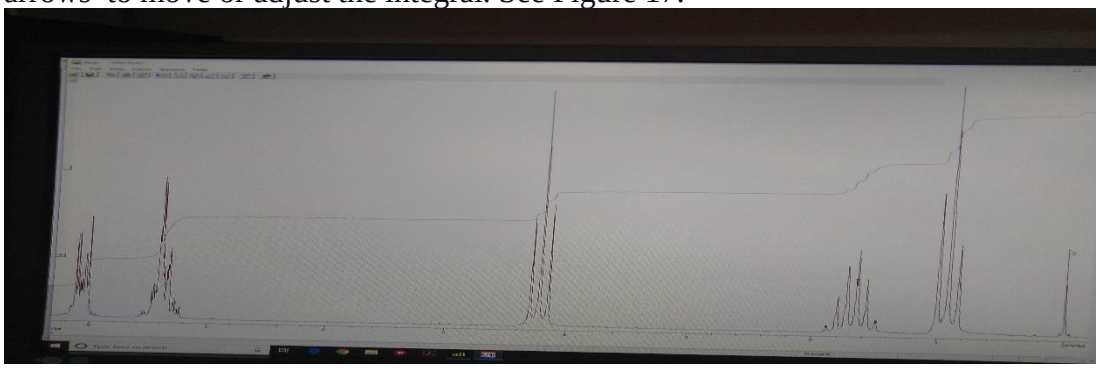

Figure 17. NUTS screen showing the integral on the spectrum.

22) 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. See Figure 18.

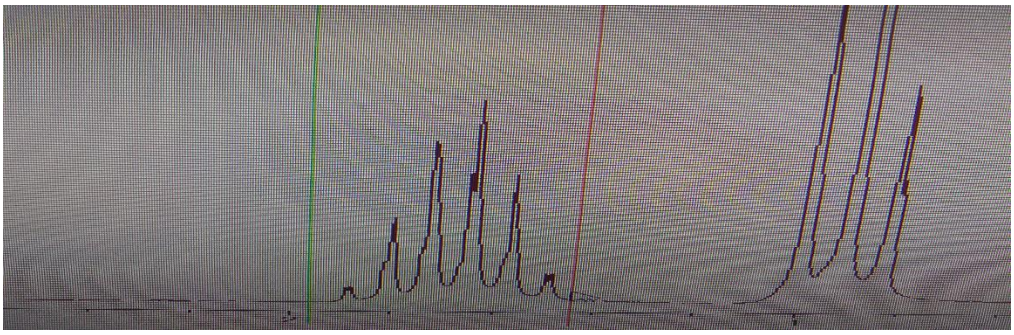

Figure 18. The NUTS program showing the integration process.

23) After integrating all peaks (except TMS), 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'. See Figure 19.

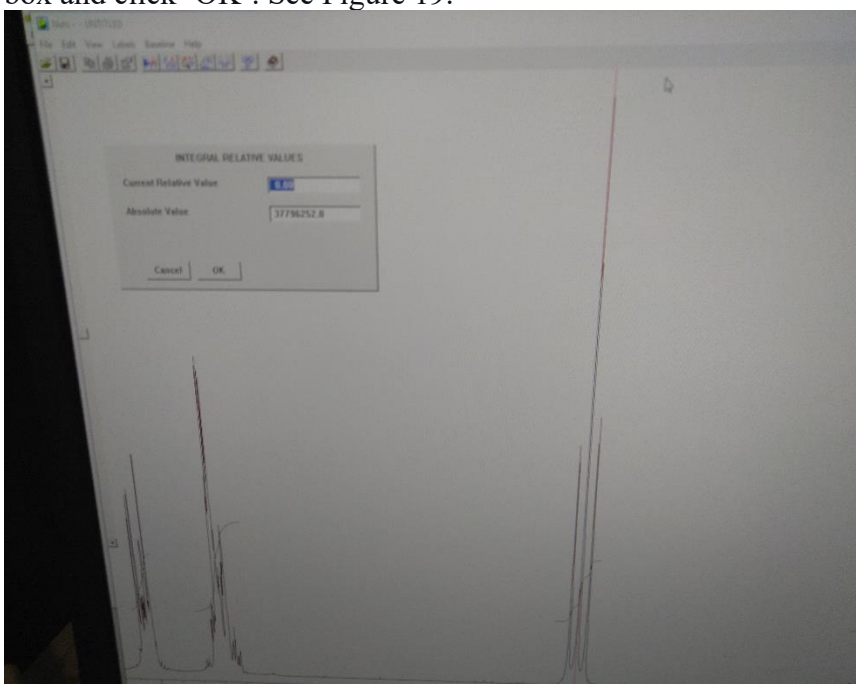

Figure 19. NUTS program screen showing the addition of integration value.

24) Press 'enter' to exit from integration. Click 'CNTRL' I.
25) 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.
26) Click print to print the spectrum
27) Click print page
28) Open paint in Microsoft programs from the control panel on the bottom left of the screen
29) CNTRL V
30) Export the file full ASC export.
31) Remove the sample and insert DI water sample into the sample chamber.
32) Complete the logbook.