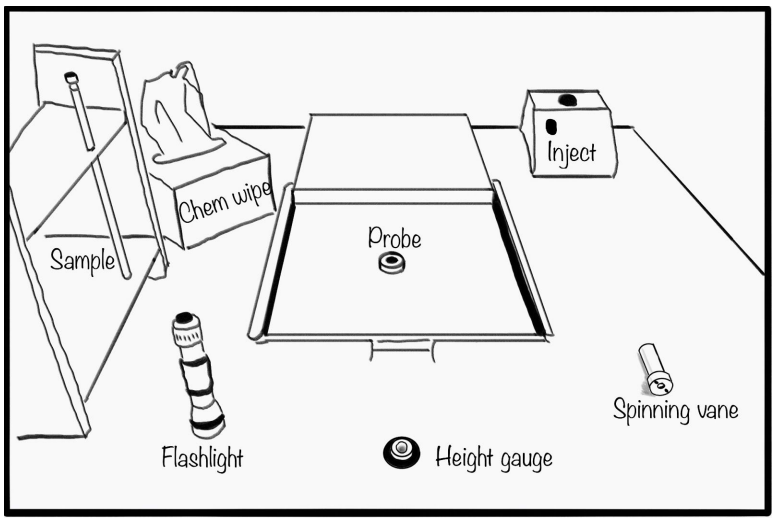
H1 NMR: Instructions to Acquire & Print

Inserting sample



1. **Locate** white cylinder (about the size of a thumb; called a **spinning vane**), optional: wipe with chem wipe.
2. Carefully insert desired sample **tube into** larger end of **spinning vane**.
3. Locate **heighth gauge** right in front of you, and **gently push** in the tube/vane.
4. Open the black plastic away from you
5. Hold down the "**inject**" button labeled on your right while lowering the tube/vane **small end down** (the intuitive direction) into the middle of the apparatus (the probe). There should be a click sound of the tube settling into place
6. **Check that it is spinning** by confirming that the mark on the cap is spinning / not visible, flashlight may be used. If needed, press eject and try again.

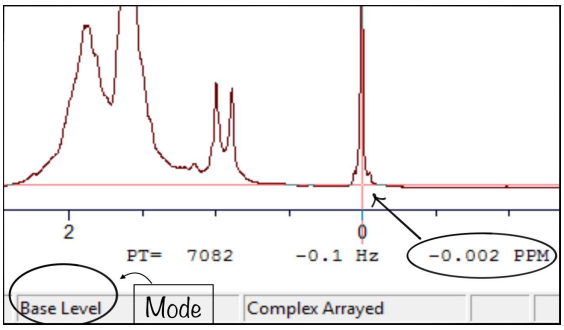
PNMR program

1. If needed, open PNMR program by selecting aii logo
2. Drop down the **Nucleus** menu, select **H1**
3. Drop down the **H1 Acquisition** menu, select **SHIM – Optimize Field Homogeneity**
   a dialogue box will open. **Select "Shim Only"** to optimize the instrument.
4. Be patient and **wait, do nothing** about a minute. Watch the lower left for the green box to disappear before continuing.

5. **H1 Acquisition:** select **ZG – Basic 1D Proton Acquisition.** This takes the spectrum.
6. Do not panic when asked for a file name. **Change nothing** to choose the **default file name** (pnmrfid) unless instructed otherwise. **Press Enter**.
7. Again wait a moment for the lower left green box to disappear.

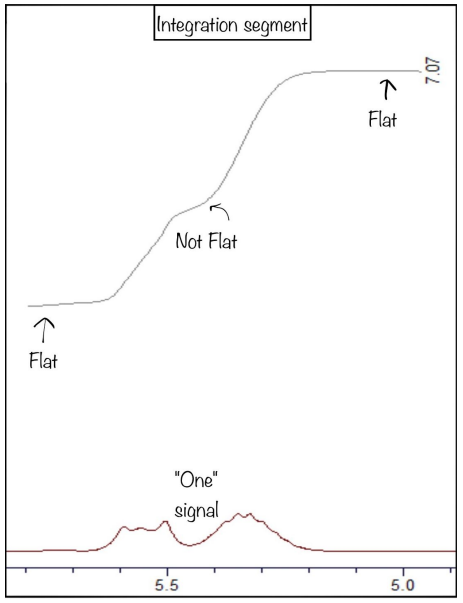
NUTS program

1. Alt-Tab to the NUTS program
2. Press **Ctrl F2** to open nmr file.
3. Don't search; **default file name** (pnmrfid) is already selected. Press Enter.
4. **Locate TMS** singlet on far right of spectrum to note it's value. (if peak's missing, ask)



5. Click and **hold to drag cursor**; it's location gives the ppm value in the lower right.
6. When cursor is lined up with peak, the **ppm of TMS should be 0.00 +/- 0.05**.
7. If yes, proceed. If not, refer to troubleshooting on reverse of page.
8. Next steps are phasing (to **even out the baseline** of the spectrum), gives more accurate integration (aka number of protons per signal.)
   i. Type command "**zo**" to select areas for zoom routine mode. (>zo will appear in the command area bottom left, mode will change from "Base Level" to "zoom")
   ii. **Locate** a well defined peak on the far **left.**
   iii. **Select the region around the peak** (as if highlighting text) and Type "**1**"
   iv. Repeat on the far **right,** but Type **"2**"
   v. You've selected 2 regions to **adjust phasing**, aka getting both sides of the peak the same height above the baseline.
   vi. **Press Enter** to exit zoom.

   vii. Type command "**pe**" to enter phasing routine then **click and hold**, moving side to side until phasing adjusted. Right click and hold will allow adjustment of other peak.
   viii. **Press Enter** to exit phasing.. Next you'll flatten excess noise.
   ix. Type command "**fb**" to enter fit baseline routine. Pink stripes will appear; usually already correct.
   x. Clicking an area will add or remove a stripe. Check and remove any that are on a peak or large bit of noise, until they are only on "flat" parts of baseline, then **type** "**l**" as in Lucy.
   xi. **Press Enter.**
9. Next steps are to **integrate the peaks** (aka "choose values for peaks of interest").
   i. Type command "**id**" to display continuous integral, to ultimately divide by peak.
   ii. **Locate** a peak whose proton value you can guess; this is the reference for other peaks.
   iii. **Observe** the most horizontal part of the integral on either side of it, for most accurate count.
   iv. **Click** mouse once to bring up cursor line, then **click each side of peak**. (The continuous integral will have "disappeared." the "F" key toggles between continuous and segmented integrals. The "L" key will undo the last segment made. "C" will clear all segments made.)
   v. If needed **Click** the peak/segment and **type** "**v**". In the dialogue box enter a whole number proton value. If a prior group has already done this, the number will show.
   vi. To isolate protons count on remaining peaks, **press F**. Segment remaining integrals as above by observing flat areas between signals on continuous integral, toggle to segmented view and observe that the peaks are automatically integrated.
   vii. **Press Enter** to exit integral mode. Ctrl+I toggles the integral display in other modes.



10. Next steps **prepare view for printing**
   i. **Type "zo",** As before, click and drag to highlight region containing all peaks.
   ii. **Press Ctrl + E** to show expanded/zoomed view and **Press Enter** to exit zoom.
   iii. Optional: Use scroll bar on right to adjust to desired relative height of peaks.

iv. Drop down **File** menu, select **printer setup** only to check that orientation= **landscape.**
v. **Verify** that all looks well: flat baseline, good integral values displayed, all signals visible.

11. **Type** "**p**" to Print. If no longer in zoom or integral mode, type command "**pl**" to "plot."
12. Optional: Make copy for lab mate, write your name and the compound on your print out.

Troubleshooting

TMS peak is not at zero: Alt-Tab to PNMR program, type "FO", in dialogue box enter TMS peak noted above, including the sign, then set to 0. Repeat step 5, and continue.

Instructions are not working: You may be in the wrong mode. Either try pressing enter to exit the mode, check the bottom right; the mode you are in will be displayed.

Integrals are weird non whole numbers: Accept error up to +- 0.2 or 0.3; ratios will be increasingly imperfect further towards the left, and with taller signals. You can repeat integration step 5(v) with the same peak and try picking another whole number or instead choose a different peak for reference, repeating step 5(v). Be reasonable and think it through. There are only so many possible protons a signal can have, start with lower number and work your way up. Check that you haven't cut off part of the integral by selecting too close to the peak; all integral segments should start and end horizontal. Check that your baseline is the same height all above your spectrum, otherwise step 8 to redo phasing.