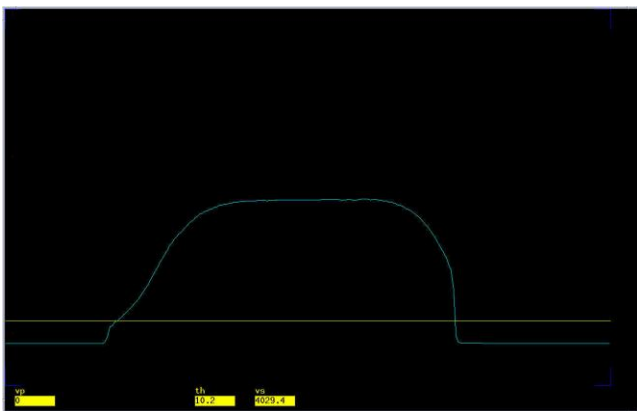


Sample Positioning in the spinner

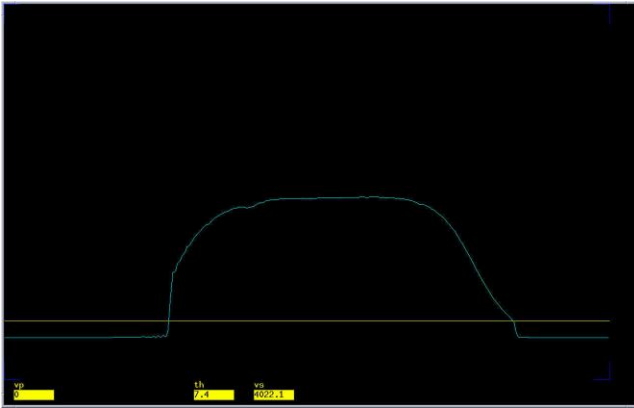
As a first step in obtaining good NMR spectrum the NMR tube containing the sample should be carefully prepared and positioned in the spinner.

- (1) It is desirable to use Shigemi tube for all samples in aqueous solvent (90% H₂O/10%D₂O).
- (2) The sample height in the tube could be 18-20mm. If sample volume is limited one could go down to 16mm height but harder to position and shim. More than 20mm height defeats the benefits of Shigemi tube.
- (3) Make sure there are no bubbles while placing the plunger. Also, there should not be liquid above the plunger.
- (4) Use the depth gauge to position the sample in the spinner. On cold probes usually the sample sits a bit higher. The coil mark in the depth gauge is 16mm. For a 20mm sample height you may place the tube so that the top is 3mm above and bottom is 1mm below the marks. The fine adjustment can be done using the gmapsys experiment used to create gradient shim map.
- (5) Insert the sample in the magnet tune and find lock and check non-spin shims and Z1-Z2 manually.
- (6) Start gmapsys and set parameters for 1H gradient map: gzlvl=4000, d3[2]=2.5ms, pw=1, tpwr=40, gain=8, d1=3, nt=1. In VNMR use the go dssh button, in vnmrj use Acquire Trial spectrum button. There will be two profile look at the second one for symmetric shape.
- (7) Say, sample is 2mm up than from the center, the profile will look like (at Inova600):

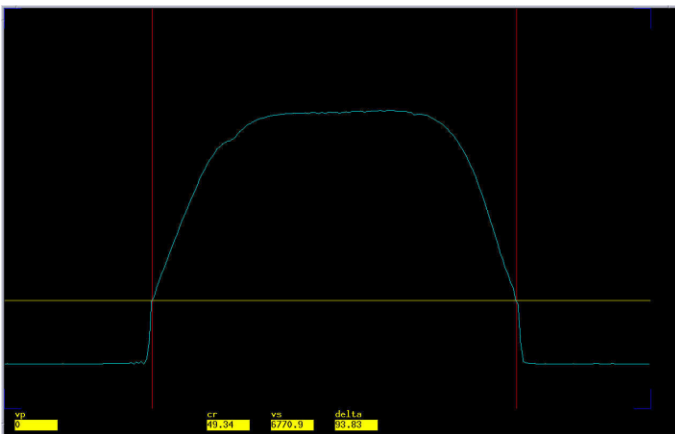


On the left the sample top-plunger interface is visible, on the right the bottom of sample and plug interface is not in line with the left. Move the sample down to align them.

(8) Sample is now pushed down but 2mm too far down



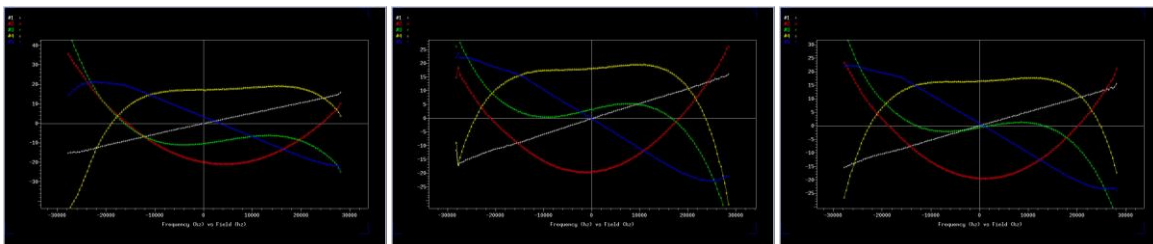
(9) The sample is pulled up a bit and centered:



Both cut of points in the profile is symmetric. A map can be made now and it is better to set $gsize=5$ and do shim all the 5 -Z shims. Also, it is useful to set $ss=-1$ so that the changes in shim current stabilizes. Before making the map set $gmap_z1z4='y'$ which would come in handy while shimming – will shim first $z1-z4$ and then $z1-z5$.

(10) Once shimming is done check Z0 again – find the on-res condition for lock and adjust phase, touch $z1$ and $z2$ shims.

(11)The following are shim maps for the three sample position – see the $z2$ curve and see how it is sensitive to the position and a centered parabola is obtained when sample is centered.



2mm up

2mm down

Centered

