5. LIFT AND SPIN

In the previous video, we created a new file location for the data we're preparing to collect. In this video we'll continue our preparations for data collection with the next two steps of the Basic Instructions: #3) properly placing our NMR sample into the magnetic field, and #4) optional spinning of the NMR tube inside of the probe.

This 4.7 T "family style" magnet is our most "dangerous" magnet in terms of stray field. This pine accordion fence sits on the 5 gauss line, which is also marked with masking tape on the floor around the magnet. Don't bring anything magnetic, electronic, or metallic into the magnetic field.

To insert the sample, number three on the instructions list, we'll need a depth measure and a spinner. There's a marking here for 5 mm outer diameter NMR tubes. That is the size you should be using. This black line marks where the coil is located inside the instrument. We have a qnp probe inside this magnet. It has two coils, an outer coil doubly tuned to ^{1}H and ^{2}H and an inner coil for the variable detection of ^{13}C, ^{31}P, or ^{19}F. We see two different markings on the depth measure that are centered the same, for the two different coils. Your sample should be both above and below the black markings. Or, in other words, the bottom of the glass tube and the meniscus at the top of the solution should be beyond both edges of the black markings. Otherwise, it will be hard to compensate for the magnetic field inhomogeneities caused by the jumps in magnetic susceptibility at the different liquid-air and liquid-solid interfaces. This will reduce the resolution and sensitivity of your NMR spectrum.

First I check that the spinner is clean. Since all NMR tubes arrive to the NMR lab thoroughly cleaned on the outside of the tube, this should not be a problem. However, it only takes one individual one time to arrive with a dirty tube, and you'll see this on the inside of the spinner. We have cotton swabs on the desktops to remove crud from the inside of the spinner before you put it into the magnet. This reduces down time on the spectrometer, keeps the instrumentation in good condition, and prevents your sample from having spinning problems.

If the glass sample tube sits too low, it may hit the probe and break. The spinner certainly won't sit in the right place for sample spinning. If it's placed too high, the solution might not reach the detector, and then we're doing an NMR experiment on air. So you do want to make sure that the tube is correctly placed into the spinner.

I place the depth measure on the table. The spinner only fits into it in one direction. The sample volume should be greater than 400 microliters, so that the sample height is c.a. 2 ½ - 3 fingers. Under conditions of sufficient sample volume, all I do is slide the sample down to the bottom, with the cap side up.

Next, leave the depth measure behind. We have cloths around for wiping the tubes and spinners in order to remove finger grease. If I touch my eyeglass lens, I leave a fingerprint that makes it difficult for me to see through my lens. When you touch the spinner, the fingerprint you leave behind can interfere with the optical eye's detection of the spinning frequency. And dust likes to stick to accumulated grease inside, blocking the tiny air holes used to spin the sample. It's much easier for you to wipe it now than for us to have down time on the spectrometer to remove the probe and the shim stack from the magnet to clean it from inside.

When I take the sample, I don't want to hold it by the spinner, I just wiped my finger prints off of it. Also, I don't want to hold it by the cap – the tube might fall. So, hold the assembly here.

We're now on step 3c of the instructions. Notice in the instructions the buttons in the pictures have labels – you can use that to figure out the location of the button on this BSMS keyboard. On the actual buttons that we use the most, the labels have been rubbed off through frequent use.

Press the lift button. This is the gray button in the upper left corner. The green light turns on. It takes a moment before we can hear or feel the lift air – so we can take our time to slowly and calmly walk to the magnet. Make sure you've removed your watch and emptied your pockets before you enter into the 5 gauss line. Once you can hear and feel the lift air, then it is safe to place the NMR tube with the spinner onto the cushion of air and let go without it crashing down.

The sample is sitting on a cushion of air. I press the lift button again. The green light is now off. As the lift air is turned off, the pressure is slowly lowered and the sample is gently placed into the coil at the center of the magnet. When the spectrometer recognizes that the sample is correctly positioned, the red "missing" light will change over to a green "down" light.

We now go to step number four of the instructions, which is optional. There is another button here labeled spin on/off. To spin the sample, I press the button and the green light will turn on. Air is blowing through tiny holes underneath the spinner in the room temperature shim stack. This causes the spinner to rotate around the z-axis. An optical eye can detect the spinning frequency by the black and white markings on the spinner.

When we spin in this way, any magnetic field inhomogeneities along the x-axis or along the y-axis are averaged over every full rotation. Thus, instead of trying to correct for spatial differences in the external magnetic field, we just smear them out. That simplifies our magnetic field corrections to a single axis, along Z. For standard pulse-acquire experiments on the 200 or 300 MHz spectrometers, we usually spin. The spin rate we use is 20 Hz. If we were to spin more slowly, we wouldn't average the x- and y-axes. If we were to spin the sample too rapidly, the vortex created in the center of the solution would make the situation worse than not spinning at all.

However, there are many instances were we choose NOT to spin. For instance, if we're running 2D spectra, or NOE measurements, or working with pulsed field gradients, or at higher fields, or using J-Young tubes – then we just skip the "spin" option and we'll use what is called "shimming" to correct for field inhomogeneities along the x- or y-axes.

Did you notice the spin button light? It was blinking and then it stopped blinking. The blinking light tells us that the spin button was activated, but the measured spin rate differs from what we requested by more than plus or minus 1 Hz.

To see what spinning frequency we requested, we can push this middle button labeled spin rate. It says 20 and the units are in Hz – meaning that we requested 20 rotation per second. In order to see the actual spinning rate, we'll use this orange button, which is labeled 2nd, in order to activate the second function of the spin rate button – the function written below the button, which is spin measure. Here, in red, it tells us on the left that we requested 20 Hz spinning for the sample, and here on the right it tells us that the actual spinning rate is 20 Hz. Since the actual spinning rate matches the requested spinning rate within plus or minus 1 Hz, the green light on the spin button is lit and is not blinking. To deactivate the spin rate/spin measure button, we press "stand by" here at the bottom.

In the next video we'll learn about the how and why of keeping our external magnetic field invariable and homogeneous during our NMR experiment – or in NMR jargon "lock" and "shim."