

U400 Sample Preparation, Positioning, and Insertion

SAMPLE PREPARATION

You must use a 528PP Wilmad tube (7 or 8 inches). There is an advantage to the longer tube on this instrument since the 7 inch tubes are going to sit low in the spinner. Any tape, parafilm or improper sealing of an NMR tube may prohibit proper positioning of the tube in the spinner and may also cause spinning problems.

In preparing the sample, it is very important to have a solvent height of 50 mm, or 2 inches, in the NMR tube in order to minimize inhomogeneity. This height is measured from the bottom of the NMR tube to the meniscus and is approximately 0.7 ml in volume. A sample that is 10 mm shorter or higher than 50 mm may be tolerable, but if you attempt to achieve higher sensitivity by concentrating your sample down to 30 mm or less, you are making a big mistake! The loss in sensitivity due to poor shimming on a short sample (which is inevitable and will result in broader line widths and poorer line shapes) will always outweigh any gain from a higher concentration. Having more solvent than you need in the tube is less of a problem, except that substantial shimming may be required as the standard shim values are determined with the 50-mm sample height and may not accommodate unusually short or long samples. (Samples that are longer than 50 mm should be avoided in VT experiments to minimize temperature gradients over the length of the sample.) Reshimming the instrument for a different sample height ($50 \pm >10$ mm) can be very time consuming. It is, therefore, strongly recommended that you always prepare your samples for this instrument in the same fashion and to a set height of 50 mm.

Finally, filter and/or degas the sample if necessary.

SAMPLE POSITIONING

The correct drawing to position your sample in the spinner is located on the wooden divider between the magnet and the two-bay console as well as on the ladder. Using the drawing on the left of the diagram, place the spinner edge on the top of the drawing, and match the bottom of the NMR tube to the bottom of the tube in the drawing (133mm). When positioning the sample, make sure that your eyes are at the same level as the bottom of the tube in the drawing. Otherwise, the sample could be mis-positioned by as much as 5 mm. Ask the MSL staff or your trainer for a demonstration of this the first time you train on this instrument. If you do not position the NMR tube to within 1 mm of the position on the drawing, the standard shim libraries may not be very close and a substantial amount of shimming may be required. A few seconds spent here can save you many frustrating minutes in shimming.

SAMPLE INSERTION

After you have logged onto the spectrometer *via* Reslog:

LC **C**onnect (in the VNMR ACQUISITION window on the right side of the screen)

LC **e**ject

Carefully climb the ladder and remove the sample from the top of the upper barrel of the magnet. Avoid touching the narrow part of the spinner to keep it clean and prevent spinning problems. Position your sample

in the spinner (see above) and place it back on the top of the upper barrel of the magnet (with the eject air still on). Make sure that the spinner does not get caught on the lip of the spin stack. (If this happens, the sample

will hang momentarily when the eject air goes off, then fall suddenly and usually break in the probe when it hits the bottom.) Return to the console,

LC **insert**

and listen for two clicking sounds as the eject air goes off and the sample slowly drops in. Then

LC **Disconnect** (in the VNMR ACQUISITION window)

NOTE:

1) Sample ejection and insertion can also be accomplished outside of the VNMR ACQUISITION window by using the *e* and *i* commands respectively.

2) For routine operations, the spin rate should be set to 20 rps for all samples. If your sample won't spin, first, check and make sure that the **SPIN:on/off** in the VNMR ACQUISITION window is on "**on**", then type "**spin=20** <rtn>" and "**spin** <rtn>". (This can also be used to regulate sample spinning when the sample is spinning too fast.) If this doesn't work, eject your sample and check the NMR tube for deformity. If it is OK, clean the tube and the spinner with ethanol, being careful not to dissolve the white dots on the side of the spinner. (The ethanol is located either in the hood or by the console with the standard samples.) If everything fails, call MSL staff for help.