4 mm Drop-in (DI) Sample Packing Instructions and Sealing Cells

A. Sample Containers

The sample is contained in a cylindrical sample rotor with the ends sealed by end caps. A pair of DI-4 end caps consists of a front turbine cap and a rear tip cap. To accommodate the many types of samples and experimental conditions, a variety of rotors, end caps, and sealing cells are available. The optimum rotor and caps for a particular experiment depend on the following considerations: (1) sample availability; (2) significance of weak background signals from the caps; (3) temperature range of the experiment; (4) physical properties of the sample (e.g., wet, dry, air sensitive, etc.); (5) required spinning speed; and (6) the importance of optimizing microwave efficiency for DNP.

The DI rotors are manufactured with extremely close tolerances and will work with all the different types of DI end cap pairs for that size. End cap pairs are available in a variety of materials.

Refer to the NMR Probe and Rotors Materials Data on the Doty Scientific web site for more information on the properties of various rotors and turbine caps.

B. Sample Loading

A well balanced, uniformly packed sample is required to obtain optimum sample spinning and B_0 field homogeneity. In packing the rotor, symmetry is critical around the spinning axis of the sample. Variations in the packing density along the length of the rotor are not critical. If very high B_1 homogeneity is required, the sample needs to be restricted to the central half of the full sample length using special spacers or cells. A rotor with the sample packed preferentially toward one end will still spin well if the packing is radially symmetric. A well balanced sample can be obtained by hand packing if the material is kept uniform while filling the rotor. In many instances, better packing is achieved with the aid of a bench spinner. Examples of both methods are described below.

Sample Packing Tools

1. Sample Preparation

Most solids and wet samples can be packed uniformly into the rotors for spinning. Plastics can be machined into solid plugs. Irregular crystals or non-uniform powders are readily ground fine using a mortar and pestle, and fibers can be minced, ground, or even wound in such a way as to be radially symmetric. Spinning of granular samples may be improved by mixing the material with a fine powder lacking interfering NMR signals. Tissues and fibers may spin better when packed wet. Some other materials should be dried and ground fine for best packing. A small funnel cup, a tamp, a rotor scraper, and sample leveling tools are provided to assist in packing rotors.

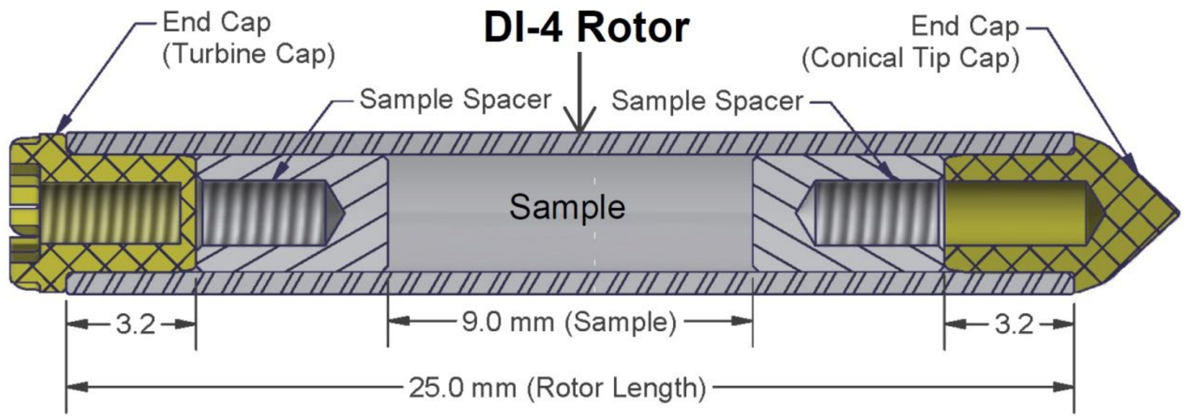
2. Hand Packing

Inspect the rotor for cracks. **Never use cracked rotors**. You should inspect the rotor with a magnifying glass. Sometimes cracks will show up with the following technique. Mark the rotor with a permanent marker. Wipe the color off with a towel wet with alcohol. The marker color may remain in a crack after cleaning.

a. ***Note: Torlon and GFT caps have some moisture absorption. It may be periodically necessary to bake out the turbine caps or tip caps at 50ºC for one hour. This is necessary if the caps become too tight.*** *(The opposite condition is much less likely. However, if one is in a very arid area or operating in a low moisture environment, the caps may have to be soaked in a liquid too make them tighter.)* ***Glass Filled Torlon (GFT)caps are used for extended temperatures.***

b. **Insertion of the Conical Tip Cap:** The proper cap insertion tools should be used to avoid damage to the conical tip cap. Gently insert a conical tip cap partly into one end (the painted end - if the rotor is painted) of the rotor. Then place the rotor with the tip cap into the conical tip insertion tool. Place the top insertion tool on the other end of the rotor. Press the rotor between the two insertion tools to fully seat the tip cap. The cap must be pressed in completely and squarely. *(There should not be a gap between the cap head and the rotor.)*

c. Optional: Sample spacers restrict the sample to the center of the coil area. If one chooses to use spacers, a spacer is required on each end. *(Sealing Cells which also restrict the sample will be discussed later.)* Insert a Sample Spacer, if desired, as shown in the figure below. Ensure that the spacer is placed such that it seats squarely against the inner end of the conical tip cap – Check the conical tip again to ensure it is still seated as described above.



Hand-packed solid sample in a DI 4mm rotor

d. Using the plastic funnel and spatula, fill the rotor about 30% full with the sample. It is critical that the sample be uniform. Loading of sensitive materials can be performed in a glove box, under special lighting, etc. With the tip cap in the conical tip insertion tool, lightly tap the bottom of the insertion tool squarely against a smooth, horizontal surface to cause the sample to settle.

e. The sample may now be packed more tightly by placing the tamping tool in the top of the rotor (which is still being held in the bottom insertion tool) and again gently tap.
Note: Using the tamp increases the concentration of the sample and therefore the S/N ratio, but it will also tend to accentuate imbalance in granular samples, or samples with irregular particle sizes.

f. Repeat steps 4 through 6 to fill the rotor, and then level the sample with the sample leveler.

g. Optional: Insert the second Sample Spacer as shown above. Take care that the threaded end faces outwards, necessary for ease of removal with the tool provided, and to ensure the sample stays uniformly packed.

h. **Alternative Packing Tips.** For best homogeneity, samples of limited quantity may be packed into small volume sample cells described in section E. Or they may be packed simply by partially filling the rotor and spinning. Centrifugal force will usually pack the sample against the wall in a well-balanced distribution. (Rotor spin packing is also useful with hydroscopic samples . Fill, spin, fill the vortex, spin… and repeat.)

i. ***Insertion and removal of DI-4 turbine caps is presented in the table in section D*** on the next page. **Note:** If you experience difficulty in cap removal, the caps can be more easily removed by cooling below -80°C. This can be done by placing the rotor in dry ice or liquid nitrogen for a few minutes. Remove the rotor from the cold bath, using a cloth or glove to protect the fingers, and immediately remove the turbine cap with the technique in ***D-2a*** while the rotor is still cold. Then the sample can be removed from the rotor. After the sample has been removed, the tip cap can then be removed as described in ***D-2b***

C. Sample Removal

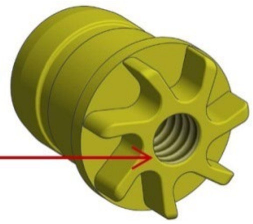
After the experiment, the turbine cap can be removed by the method in ***D-2a*** on the following page. The sample may be scraped out with the scraper or, in stubborn cases, dissolved. Standard turbine caps are designed to fit very tightly when new. They will loosen slightly with each use, especially if used much above room temperature.

Alumina, Kel-f, and Kalrez may be cleaned with virtually anything except very hot, concentrated, strong bases and Hydro-fluoric acid. Zirconia, macor, silicon nitride, Viton, Vespel, Aurum, and PEEK may be cleaned with most organic solvents, and cold, moderately concentrated (40%) strong acids and bases for several minutes. Hot soapy water often works best on the ceramics. Viton and Kalrez swell in acetone and some other organics. Torlon, Vespel and Aurum swell in water.

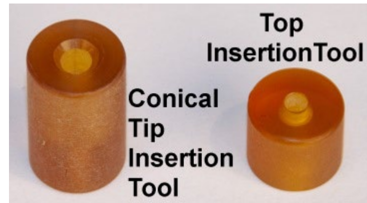
D. Insertion and Removal of DI-4 Turbine Caps

DI-4 Front Turbine Caps

Note how the turbine blades are connected to form a ring around the threaded hole.



Proper Insertion Tools for DI4 Caps



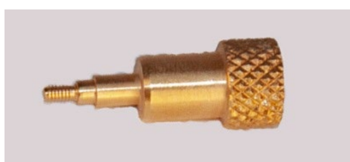
1. Proper Turbine Insertion:

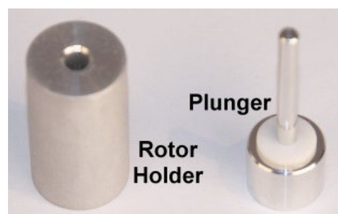
a. For the DI-4 front turbines, the proper cap insertion tools (shown above) should be used to avoid damage to the turbine or the conical tip cap. After filling the rotor (see prior sections), the rotor with the tip cap should be sitting in the conical insertion tool. Gently insert the turbine part way into the rotor. Now place the top insertion tool over the turbine cap and push the cap into the rotor.

Note: Kel-f turbine caps are softer than Torlon and GFT. Kel-F turbines should be inserted (and removed) using the brass removal tool shown below rather than the top insertion tool shown above.

b. If the cap does not fully seat *(there should not be a gap between the cap head and the rotor)*, some sample will need to be removed using the sample leveling tool. Remove the cap. Remove some sample with the leveling tool and reinsert the cap using the process described above.

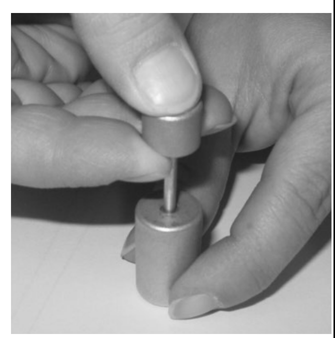
Proper Removal Tools





2. Proper DI-4 Cap Removal Technique

a. For turbine cap removal, the rotor should be held in the rotor holder (shown above). The rotor holder is easier to hold than the rotor, and protects the rotor while the cap is pulled out. The rotor is inserted into the rotor holder with the turbine end first.
(The rotor can only fit into the holder at its larger end.) The puller can then be threaded into the turbine. The turbine can be pulled out through the smaller end of the holder. It is important to pull the turbine straight out (not at an angle).



b. With the front turbine cap removed and the rotor emptied of all sample, the rear tip cap can be removed using the rotor holder and plunger. The rotor is inserted, (with the remaining rear tip end first) into the rotor holder. The plunger is inserted into the rotor. The tip cap is pressed out - passing through the smaller end of the rotor holder. *When the caps are new it may be necessary to cool them in liquid nitrogen to get them out. They may be very tight for the first use. Note section **B-2h** in Hand Packing on the prior page.*

There is a simple video on the use of packing tools. Note: In the video, JB mentions packing the sample, then removing the cap to see if there is a significant hole in the center of the rotor, and then he instructs filling the rotor more completely. This should only be needed when maximum sample is required. (Most of the time the sample is fine as first packed.) To access the video, click here.

E. Sealing Cells.

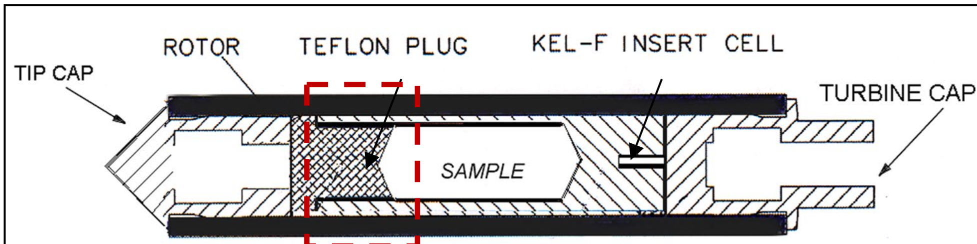
1. Sample Cell Overview: Sealing cells are useful for many reasons. They can seal reference samples that may need to be run again months after initial use. They can be used for samples with limited volumes. They can be used to store spectrometer set-up standards, freeing up rotors and caps for use with many different cells instead of single packed samples. <u>Sealing cells may be used with wet or dry samples</u>.

Generally sealing cells are made of Kel-F and Plugs are made of Teflon. Ultem cells with PVC plugs are also available for ^{19}F probes, The figure below shows a sealing cell and its plug.

Plug (Left) and Sealing Cell (Right)

Use with Puller # 01003

To avoid the sealing cell expanding outward while spinning – which could allow leakage, the cell with the plug inserted must fit snugly into the rotor. To ensure this, nominal, oversize and undersize plugs are provided. Usually nominal plugs fit best. The fit should be checked as follows: slide the cell with the plug inserted into the rotor. If the cell is loose at the plug area *(Note the dashed box area below)*, an oversize plug may be put in place of the nominal plug. If the plug is too tight you may need to use an under size plug. The plug may be pulled out using your fingernails or by using small pieces of sandpaper to hold the cell and the plug while pulling the plug out. If necessary, use a screwdriver blade or a sharp knife to pry out the plug – taking care not to damage the plug.



Sealing cell and rotor end caps in correct position inside the rotor.

2. Sample Cell Operation: Fill the cell with your liquid sample to the very top. While slightly squeezing the cell, push the plug into the top of the cell and release the squeeze. This technique helps in filling the cell without trapping an air bubble. Insert the cell into the rotor, plug-end in first. The closed end of the cell has a threaded hole to facilitate removal from the rotor with a puller.

To seal a liquid sample, the plugs are made such that they fit snugly into the cells, because of slight variations (± 8 μm) in their size during manufacture, this may cause the filled and sealed cell to fit too tightly into the rotor.. In such a case the sealing cell may be sanded with a fine (600 grit or higher) clean sandpaper. Caution sanding away too much may cause the cell to fit loosely in the rotor and expand during spinning causing a leak.

After inserting the sealing cell, insert the conical tip cap from the bottom end – i.e. the end closer to the plug. Use the insertion tools and the procedure in section ***B-2a***. Ensure that the tip cap is fully inserted and seats firmly and squarely against the end of the rotor. Next, insert the turbine cap into the rotor from the end closer to the back of the sealing cell, as shown in the figure above. Insert the turbine cap and insure that it is fully inserted and seats firmly and squarely against the end of the rotor using the procedure in ***D-1*** <u>for the appropriate turbine.</u>

To remove the sealing cell from the rotor, first pull the turbine cap out using the tools provided. Use the correct threaded Spacer Removal Tool (shown previously) to remove the sealing cell. **The vacuum created inside the rotor can sometimes suck the sealing cell plug back into the rotor and cause your sample to spill. To avoid this, follow these instructions carefully.** Screw the tool into the end of the sealing cell and gently pull it out untill the plug gets close to the end of the rotor – **PAUSE** at this point do not pull it further. The approximate pausing position is shown in the last figure. Again note that the vacuum created inside the rotor can sometimes suck the plug back into the rotor and cause your sample to spill. Thus wait for a few minutes to let the vacuum decay, then gently pull the entire cell out. (You can also try other techniques such as slowly rotating the cell as you pull it or moving it back and forth while pulling it out to eliminate the vacuum effect.)

To allow the vacuum to decay, pause at approximately this position for a few minutes.