

1mm MAS sample packing & unpacking

I. Introduction

Sample packing is very important to achieve stable and safe sample spinning of 1 mm MAS. The caps are small therefore sometimes the caps fail to insert into the rotor sleeve properly. To avoid this, carefully designed packing tools are provided for 1 mm MAS.

II. Key points for successful sample packing and rotor maintenance.

1. Keep it clean.

The 1 mm MAS rotor is very small and thus even small dust particles may cause serious spinning problems. Care must be taken to keep not only sample tubes and caps clean but it also important to keep the sample packing tools clean. Dust-off and Kim-wipes are required to achieve this.

2. Avoid pressing both ends of the sample tube.

The caps are fragile. Pressing on both caps at the same time might break a cap. When pushing in a cap the other end of the rotor should be kept free. The rotor is clamped to the “sample tube affixing plate”. This will act like a circuit breaker; a too strong push force will cause the rotor to slip, instead of breaking the cap.

3. Insert caps properly.

Proper insertion of both caps is very important for safe spinning. Improper insertion may cause serious problem such as breaking the neck during sample spinning. If the bottom cap breaks during spinning, the probe might need to be fixed JEOL engineer or even to be sent back to factory.

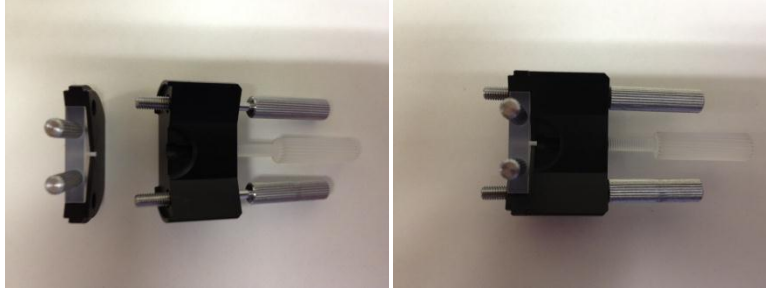
III. Packing a rotor

- A. Clean all tools carefully, dirt, dust, and lint will cause spinning problems and may damage the rotor, end caps or probe.
- B. Inspect all tools and parts with the microscope for dirt, dust, lint, and residual sample material. Re-clean the parts as necessary.
- C. Mount the rotor in the rotor packing holder. Tighten the screws to firmly hold the rotor in place.



D. Mounting the bottom cap

1. Press the rotor holder into the cap packing holder. Make sure the rotor position rod is screwed back and out of the way.



2. Open the vial containing the rotor caps and carefully dump them out on the microscope stage. Use a guard to prevent the caps from accidentally rolling off the stage.



3. Use the sticky tool to pick up the turbine cap and put it back in the vial. Cap the vial.

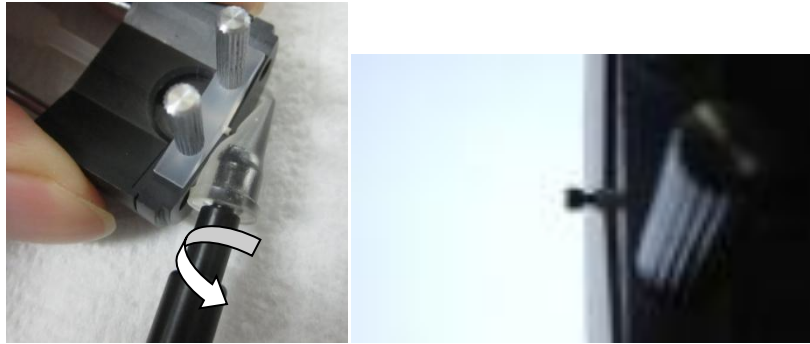


Keep the unused cap (turbine cap) into the vial again

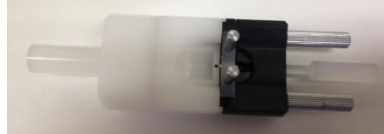
4. Use the sticky tool to pick up the bottom cap. Adjust the position of the cap so the flat end is against the sticky tool.



5. Under the microscope, inspect the cap for damage, dirt, or residue from the previous sample. Clean the cap is necessary.
6. Gently place the bottom cap in the end of the rotor.
7. Use a twisting motion to remove the sticky tool leaving the bottom cap partially inserted in the rotor.

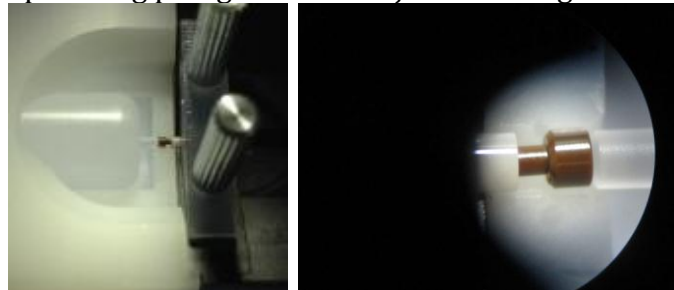


8. Connect the rotor holder and cap packing holder to the cap pressing tool and tighten the screws. Take care do not knock the bottom cap out of the rotor.

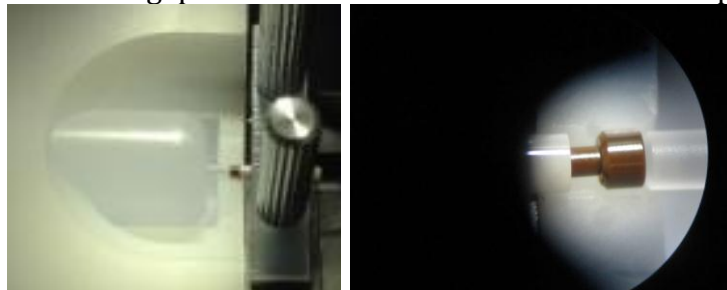


Release the center screw

9. Push in the cap seating plunger until it is just touching the bottom cap.



10. Use carefully dust-off to blow out any dirt or dust to prevent dust and dirt being trapped between the bottom cap and the rotor.
11. Under the microscope, press the cap into the rotor until it is firmly seated. It is critical that there no gaps between the rotor and the bottom cap.



Light from the bottom

12. Remove the cap pressing tool
13. Remove the rotor holder from the cap packing holder

E. Mounting the sample funnel

1. Remount the rotor holder on the cap packing holder so the bottom cap is towards the rotor position screw. The rotor positioning screw should not be touching the rotor.
2. Loosen the rotor holder clamp screws
3. Slide the rotor back so the end of the rotor is flush with the holder.
4. Tighten the screws to very loosely hold the rotor in place
5. Mount the sample funnel on the rotor holder - cap packing holder assemble and tighten the screws. Leave the clamp screws loosely holding the rotor in place.
6. Under the microscope turn the rotor positioning screw to move the rotor into the hole on the back of the sample funnel. Do not force the rotor into the sample funnel. If you meet resistance, stop, back out the rotor positioning screw, and reposition the sample funnel.



7. Tighten the rotor holder clamp screws.
8. Back out the rotor positioning screw so it is no longer touching the bottom cap.
9. Mount the sample funnel - rotor holder - cap packing holder assemble in the vertical holder.



10. Under the microscope inspect the sample funnel hole and make sure the hole in the rotor is not covered by the funnel.

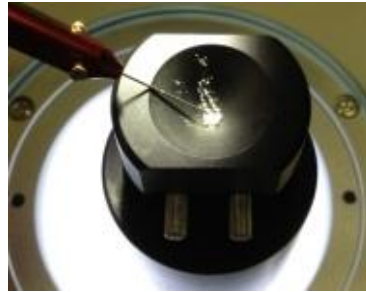
11. Check for gaps between the rotor and the funnel.

F. Packing the sample and adjusting the sample fill depth

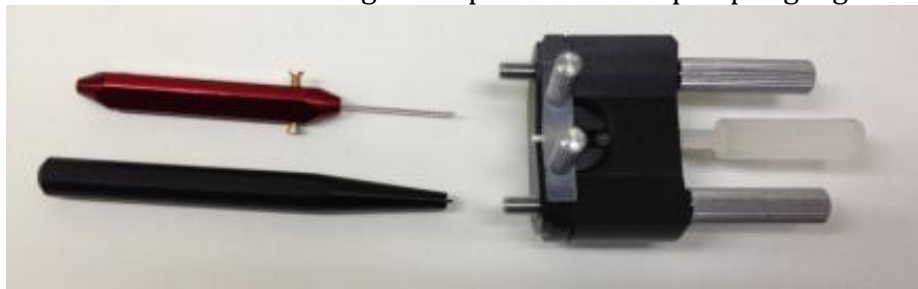
1. Prepare the sample for packing. Make sure it is finely ground and inspect it under the microscope for dirt and lint that may have accidentally gotten into the sample.
2. Transfer 1 to 3 mg of the sample to the sample funnel.



3. Under the microscope, use the blunt end of a 0.48 mm drill to pack the sample into the rotor. Tamp the sample gently. Take care do not break the drill in the rotor.



4. Fill the rotor completely. Packing in small amounts will improve the packing and final results.
5. Remove the sample funnel - rotor holder - cap packing holder assemble from the vertical holder
6. Remove the sample funnel
7. Clean the end of the rotor. You may use dust-off, but be careful do not lose the sample out of the rotor.
8. Under the microscope, drill out about 1 mm of the sample with a 0.48 mm drill. Do this in small amounts checking the depth with the cap depth gauge.



9. Clean the end of the rotor to remove any extra sample.

G. Mounting the turbine cap

1. Open the vial containing the turbine cap and carefully dump it out on the microscope stage. Use a guard to prevent the cap from accidentally rolling off the stage.
2. Use the sticky tool to pick up the turbine cap. Adjust the position of the cap so the flat end is against the sticky tool.

Never try to pick the caps by finger. It would damage the caps, especially turbine caps.

3. Under the microscope, inspect the cap for damage, dirt, or residue from the previous sample. Clean the cap is necessary.
4. Gently place the turbine cap in the end of the rotor.
5. Use a twisting motion to remove the sticky tool leaving the turbine cap partially inserted in the rotor.
6. Connect the rotor holder and cap packing holder to the cap pressing tool and tighten the screws. Take care do not knock the turbine cap out of the rotor.
7. Push in the cap seating plunger until it is just touching the turbine cap.

Release the center screw

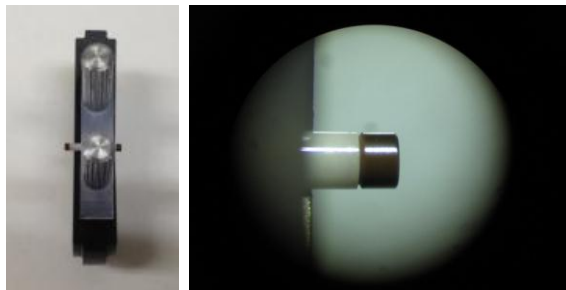
8. Use carefully dust-off to blow out any dirt or dust to prevent dust and dirt being trapped between the bottom cap and the rotor.
9. Under the microscope, press the cap into the rotor until it is firmly seated. It is critical that there no gaps between the rotor and the turbine cap.

Light from the bottom

10. Remove the cap pressing tool
11. Remove the rotor holder from the cap packing holder

H. Final rotor inspection

1. Under the microscope at the highest magnification inspect the rotor and end caps for gaps between the rotor and cap. Check for gaps from at least 4 directions. If there is a gap remount the holder on the cap packing holder and cap pressing tool and reseat the end cap. If necessary remove the cap and try to reinstall the cap without a gap.



2. Clean the rotor with dust-off to remove all dirt, dust and lint from the area of the end caps.
3. Remove the rotor from the rotor holder.
4. Under the microscope re-inspect the rotor to ensure that everything is correct, there no gaps or dirt.

Clean up the outside of rotor and the bottom surface of the bottom cap by kim-wipe.

Keep the sample into the plastic bag to avoid the damage on the turbine cap.

I. Loading the rotor in the probe

1. Remove the probe cap.
2. Open the drive and bearing air to ~ 100 kPa for 30 seconds to blow out dirt, dust, or lint that may have gotten into the probe.

At first we need 5mins blow out.

3. Close the drive and bearing air.
4. Pick up the rotor with the plastic forceps. Make sure the bottom cap is pointed away from the forceps.



5. Insert the rotor in the probe stator. You may find tilting the probe back makes this operation easier.
6. Tap the probe to help drop the rotor completely into the stator.
7. Lower the rotor clip to keep the rotor in place.
8. Put the probe cap on.
9. Set the spinning speed and turn on the drive and bearing air by clicking the Auto button
10. The rotor should start spinning and you should not hear air hissing.

The drive pressure must not be exceeded 20 kPa.

11. Stop the spinning and wait for the rotor to stop.
12. Mount the probe in the magnet and run the desired NMR experiments.

Keep watching the spinning frequency and drive pressure. If the required drive pressure is 15% higher than the written value, stop the spinning and check the rotor again.

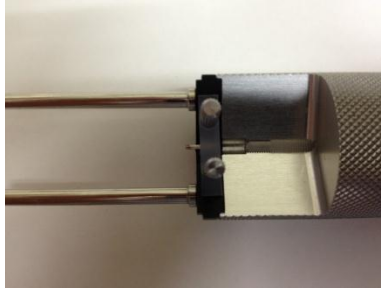
IV. Unpacking the sample

A. Removing the rotor from the probe

1. Turn off spinner and wait for spinner to stop
2. Lower probe and remove cap
3. Slide rotor clip out of way
4. Remove rotor by tilting the probe and tapping or sucking out the rotor with a 10 cc syringe

B. Removing a rotor cap

1. Mount and clamp the rotor in the cap tweezers guide and rotor holder. About 2 to 3 mm of the rotor body should be exposed on the outside of the rotor holder. You may use the clamp bar with the rubber tip.



2. Using the microscope carefully slide the cap tweezers over the rotor until it is touching the rotor holder
3. Place one side of the cap tweezers against the rotor
4. Carefully slide the cap tweezers away from the holder until it engages the groove between the cap and the rotor
5. Close the cap tweezers engaging the groove on both sides of the rotor.



6. Use freeze spray for 10 to 15 seconds to cool the rotor and cap tweezers. This makes removing the cap easier and helps prevent damage to the caps.
7. Pull the cap out of the rotor using the cap tweezers.
8. Transfer the cap from the removal tool to a vial and close the vial.

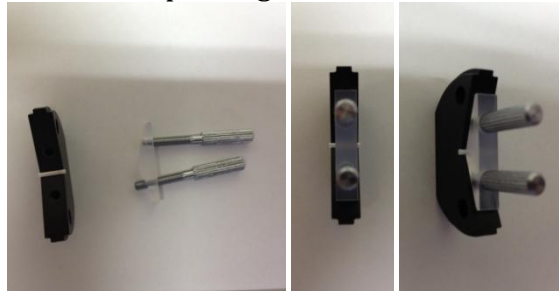
C. Removing the remaining rotor cap

1. Reverse the rotor in the cap tweezers guide and rotor holder.
2. About 2 to 3 mm of the rotor body should be exposed on the outside of the rotor holder
3. Using the microscope carefully slide the cap tweezers over the rotor until it is touching the rotor holder
4. Place one side of the cap tweezers against the rotor
5. Carefully slide the cap tweezers away from the holder until it engages the groove between the cap and the rotor
6. Close the cap tweezers engaging the groove on both sides of the rotor.
7. Use freeze spray for 10 to 15 seconds to cool the rotor and cap tweezers. This makes removing the cap easier.
8. Pull the cap out of the rotor using the cap tweezers.
9. Transfer the cap from the cap tweezers to a vial and close the vial.

10. Remove the rotor from the cap tweezers guide and rotor holder.

D. Removing the sample from the rotor

1. Mount the rotor in the rotor packing holder.



2. Under the microscope use a 0.48mm drill mounted in the holder to carefully drill out the sample. DO NOT apply too much pressure when drilling and take care so the drill does not break off in the rotor damaging the rotor



3. Slide the drill back and forth in the rotor to clean out the sample completely.

4. Use dust-off or equivalent to blow off any remaining sample.

5. Cut a 4 to 6 cm narrow triangle from a Kim-wipe. Make the cut across the fiber grain of the Kim-wipe



6. Twist the Kim-wipe into a thread.



7. Under the Microscope, thread the twisted Kim-wipe through the rotor and carefully pull the Kim-wipe through the rotor. Additional twisting may be necessary to pass the Kim-wipe through the rotor. With practice you should be able to pull +85% of the Kim-wipe through the rotor.



8. Using the microscope inspect the bore of the rotor to make sure it is clean.
9. Remove the rotor from the holder and put in the vial with the end caps. Cap the vial.