

Preparation of a solid-state NMR sample

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Instrument: JEOL 300 MHz solid state

Probe: JEOL 3.2 mm MAS

Sample tube: NM-05410ST32 (Zirconia, ZrO₂); Order through parts@jeol.com

Sample volume: 27-50 mm³ (with and without spacers, respectively)

Starting your work

You must use iLab calendar to sign up for JEOL 300. After you made your time reservation, make sure to start Kiosk on your mobile device on time. You may start it up to 9 min before the time to 8 min after your start time. You don't need to be in the NMR room for that. If you did not start Kiosk, your reservation is cancelled and full amount is billed to your account as a "no-show" reservation. If "no show" happened by accident, you should make a new reservation, start the Kiosk, and contact NMR staff to avoid double billing.

Sample limitations

Density of your sample limits the maximum spinning speed for the tube:

	Material density, gram/cm ³	Maximum allowed speed, Hz
Organic compounds, polymers	1 - 2.5	22,000
organo-metallic	2.5 - 3	19,000
carbon, metals	3 - 4	17,000
ceramics, metals	>= 5	15,000

Tutorials

Watch the training tutorials for JEOL 300 MHz system on nmr.nd.edu.

Cleaning the sample tube

1. Disassemble the sample tube completely
2. Scratch out the old sample with the blue scraper tool
3. Soak all parts in water or water-alcohol mixture. The sleeve may be soaked in other solvents to dissolve the solid sample particles, if needed.
4. Clean the sleeve inside with a soft wire brush to make sure no particles are stuck to the inner surface
5. Check inside of the sleeve with a packing tool that the surface is smooth and was thoroughly cleaned up. Check inside of the inserts, top, and bottom caps for stuck particles.

6. Rinse all parts in alcohol and allow them air-dry

Packing the sample powder

NOTE: Sample must be completely dry as the sample tubes are NOT leak-proof.

- Clean black funnel with the wire brush
- Remove the spinning cap from the sample tube
- The bottom plug should be inside. Check that there is no gap between the bottom and a sleeve.
- Take out the spacers (use the needle tool - be careful not to scratch the sleeve and the spacer!)
- Place one spacer in if you intend to use spacers
- Drop the tube down the funnel
- Add sample powder
- Tap with the funnel to help direct the powder down
- Compress the powder with the blue tool **very slightly**, just enough for the tool to stop as it reaches the powder. **DO NOT PACK TIGHTLY!**
- Use just enough powder to have
 - if using spacers: the metallic strip fully inside the tube - the tube top should meet the colored portion.
 - if not using spacers: the blue strip inside, metallic strip fully outside the tube
- If you loaded too much sample, **DO NOT COMPACT POWDER FURTHER!** Instead, scratch out the excess powder with a scraper end of the blue tool.

Closing the tube

- If using spacers: Insert spacer on top of your sample, check depth for the cap with a blue tool.
NOTE: you should see entire metallic band of the tool out of the tube!
- Put in the spinning cap: push down with your fingers to fully insert. If there is a gap between the cap and a sleeve, you put in too much sample. You must open the tube and remove some powder!

Check the bottom cap

The paint for spinning speed detection should be black and coloring 1/2 of the circumference of the cap. If it is not good,

- wash it off with ethanol
- repaint with a black marker
- Give it 5 min to dry up before inserting in the spinner bore

Sample packing with a benchtop spinner

NOTE 1: The spinning is done in multiple steps with a gradually increasing spin rate to **pack the sample powder along the walls of the tube. You must spin at required speeds and for required time periods to correctly pack the sample.** If the sample was not packed well, the tube will be unbalanced and may explode at a high spin rate damaging the probe!

NOTE 2: Stand by while watching the tube to attain the requested speed: you need to witness the speed to overshoot the target and then begin drifting back. **Never turn on spinning and walk away!** Wait for the speed to settle and verify there are no fluctuations greater than 0.1%.

You should always finish the **benchtop** procedure at a maximum speed **exceeding** the desired speed to use in a **probe** by 2 kHz:

- if you want 10 kHz in a probe, spin at 12 kHz on a benchtop;
- for 17 kHz in the probe use 19 kHz on a benchtop

If the tube is not spinning at a stable rate, it is unbalanced, will get scratched, and may explode at high speeds damaging the probe. You MUST stop spinning and repack the sample.

Making sure the JEOL system is on

- **open N2 gas valve on a wall**
- spectrometer is always ON
- the laptop must be turned on and logged in
- MAS controller must be ON
- MASCONT software must be open
 - If you found MAS controller and/or laptop off: turn them on, launch MASCONT software and click Disconnect/Connect button.

Loading the benchtop spinner

- Take the probe out of the magnet
- Disconnect spinning and bearing gas lines from the probe
- Remove the caps from the benchtop spinner
- Put them on the probe gas inlets
- Attach gas lines to the benchtop spinner (according to color).
- Disconnect SPIN cable from the probe - attach to a spinner
- Open the spinner shield (rotate away)
- Wipe the sample tube with a kimwipe
- Check the top and bottom caps for gaps
- Do a scratch test
- Put the tube into the spinner with the spinning cap (brown) facing outside.
- Shake it a little to make sure the tube settles to the bottom of the bore.
- Close the spinner shield

- In the MASCONT window, check that you see the correct tube material shown in the "Sleeve" window (ZrO₂ for our tubes)
- Enter the File Name in the "Log" section as **[Your initials]_[experiment ID]_benchtop** and click "Start" to start recording the spinning log.

Connecting to a benchtop spinner from the computer

- Launch NoMachine software from the taskbar.
- Choose MAS controller/Spin control, click Connect
NOTE: If NoMachine does not work, proceed with using the laptop on a spectrometer console.

Adjusting light intensity for the speed measurement

IMPORTANT: Never touch the light adjustment knob while MASCONT is spinning in Auto mode!

- Make sure the MASCONT software is in a manual mode (default mode when stopped): click **Manual** button.
- Adjust Bearing pressure to 100 kPa (use slider, then arrows; 1kPa/click).
You should see the sample start spinning at low speed.
- If the sample does not spin, try these remedies (one after another):
 - Rotate light adjustment knob to have approximately middle position.
 - Set Bearing pressure to zero and take the sample out:
 - Verify that the flute (turbine) is facing outside.
 - Check if the flute and end cap are inserted tightly and there is no gap between them and a sleeve.
 - Wash off the paint on a bottom cap with ethanol and repaint the speed-counting strip.
NOTE: Let the marker dry up for 5 min before inserting the tube back into the spinner, otherwise you may transfer some marker paint on a bore of a spinner!
 - If you still cannot start spinning, try a standard sample.
 - If it does not spin either: the spinner bore needs cleaning —**contact NMR Center Director, Evgenii Kovrigin**
- If the sample started spinning in the previous step: now, adjust the Drive pressure to 4 kPa
- Wait 20 sec for the speed to stabilize (around 580 Hz)
- Rotate ("walk") the light adjustment knob in small increments (a few degrees) to find the detection region:
 - Walk to reach one end of the (to see the speed drop),
 - walk one step back (to see the speed restored),
 - walk to another end counting small steps till you see the speed drop again
 - step back one step
 - take 1/2 of the counted steps back (to set the knob near the middle of the maximum speed region)

- **This adjustment will need to be repeated at the high end of the speed range**

Spinning test at 800 Hz

- Start with low "Target Speed": 800 Hz
- Click "Auto"
- **Stand by while** watching the speed: you need to see it overshoot the target and then begin drifting back.
- The spinning rate should equilibrate at 800 Hz with Drive Pressure: 5, Bearing: 100
- Look at the log: **Desktop\Libraries\Documents\My Documents\MSCONT\Log** (the file refreshes approximately every two minutes only!)
- WAIT FIVE MINUTES and record the spin rate. It must be stable within <1% (8-4 Hz at 800 Hz)
- **IMPORTANT: If you see rate fluctuations of +/- 10% (50-100 Hz at 800 Hz) - the sample tube is unbalanced and touches the walls. It will be scratched and eventually explode. STOP IMMEDIATELY AND REPACK THE SAMPLE!**

Spinning at 1600 Hz

- Stop spinning
 - wait for all readings to zero
 - Set 1600 Hz
 - Click Auto
 - **Stand by while** watching the speed: you need to see it overshoot the target and then begin drifting back.
 - Wait until spin rate equilibrates at 1600+/-2 (takes several minutes)
 - Stable parameters: Drive 9, Bearing 100
 - Wait 5 minutes
- NOTE: This rate is most difficult for the spinner to maintain. It may overshoot to almost 3000 and then comes back - this is OK.

Spin at 3000 Hz

- Stop spinning
- wait for all readings to zero
- set 3000 Hz
- **Stand by while** watching the speed: you need to see it overshoot the target and then begin drifting back.
- stable readings: 3000 Hz +/- 1-2 (0.1%), Drive 31, Bearing 100
- wait 5 minutes

Spin at 6000 Hz

1. Stop spinning
2. wait for all readings to zero
3. set 6000 Hz

4. **Stand by while** watching the speed: you need to see it overshoot the target and then begin drifting back.
5. stable at 6000 Hz+/- 6 (0.1%), Drive 77 , Bearing 150
6. wait 5 minutes

Spin at 9000 Hz

1. Stop spinning
2. wait for all readings go to zero
3. set 9000 Hz
4. **Stand by while** watching the speed: you need to see it overshoot the target and then begin drifting back.
5. as the speed is reached, check light adjustment using the following protocol (no need to wait for full stabilization):
 - a. **Switch to Manual mode:** click Manual button
 - b. Walk the light adjustment knob looking at speed to find the middle of the detection range
 - c. **Return to Auto mode:** click Auto
6. stable at 9000 Hz+/- 10 (0.1%), Drive 126 , Bearing 150
7. wait 5 minutes

Spin at 12 000 Hz (working spin rate in the probe will be 10 000 Hz)

1. At this high speed—no need to stop spinning
2. adjust spin rate to 12,000 Hz, hit **Enter**
3. **Stand by while** watching the speed: you need to see it overshoot the target and then begin drifting back.
4. stable parameters: 12,000 Hz+/- 1-2 Hz (0.1%), Drive 172 , Bearing 249
5. wait 5 minutes

IMPORTANT: If you see rate fluctuations of > 0.2% (24 Hz) - the sample tube is unbalanced and touches the walls. It will be scratched and eventually explode. STOP IMMEDIATELY AND REPACK THE SAMPLE!

Spin at 15 000 Hz and 19 000 Hz (for working spin rate in the probe of 17 000 Hz)

1. At this high speed—no need to stop spinning
2. adjust spin rate, hit **Enter**
3. **Stand by while** watching the speed: you need to see it overshoot the target and then begin drifting back.
4. watch for stability within 0.1-0.2%

Finishing the benchtop spinner procedure

- Stop spinning

- Click [Stop] in the "Log" section to stop recording the spinning log. It is viewable in **Desktop\Libraries\Documents\My Documents\MSCONT\Log**
- Disconnect hoses and the SPIN controller cable and reconnect to the probe
- Take sample out of benchtop spinner
- Proceed with **Solid State Experiment** protocol

For a repeated test of a well-packed sample intended to spin at 10 kHz

- Start with 800 Hz, verify stable speed
- Jump without stopping to 5kHz, verify stable speed
- Jump to 10 kHz , verify stable speed
- Jump to 12 kHz, verify stable speed
- Go to the probe

For a repeated test of a well-packed sample intended to spin at 17 kHz

- Start with 800 Hz, verify stable speed
- Jump without stopping to 5kHz, verify stable speed
- Jump to 10 kHz, verify stable speed
- Jump to 14 kHz, verify stable speed
- Jump to 17 kHz, verify stable speed
- Jump to 19 kHz, verify stable speed
- Go to the probe