

Instructions for MAS Sample Packing

(1) We suggest spin testing empty rotor before packing – this allows one to determine if unstable spinning is due to packing – hence requiring sample to be repacked.

NOTE: To optimize signal and RF homogeneity, sample should be centered in the rotor

NOTE: Only handle rotors using Kimwipes, finger grease will affect how rotor spins.

(2) Determine type of spacers to be used: Teflon/Kel-F/PTFE, unless ^{19}F spectrum to be collected, then we suggest a non $^{13}\text{C}/^{19}\text{F}$ containing material such as boron nitride.

(3) Calculate bottom and top spacer lengths needed to center sample via:

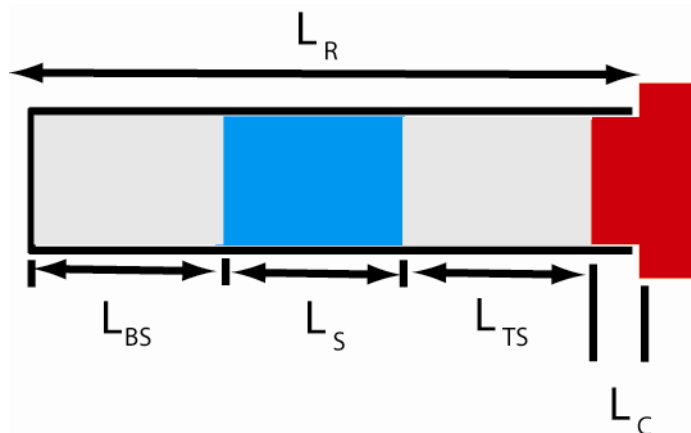


Figure 1: Typical Solid-State NMR Rotor Diagram

Total length of rotor measured without caps: $L_R - L_S = L_{BS} + L_{TS}$, where

L_R = Inner length of rotor

L_C = Length of end cap that will be inside rotor

$L_{BS} + L_{TS}$ = Length of Bottom and Top Spacers

(4) Weigh the rotor, endcap(s), bottom and top spacers.

NOTE: When packing sample, if static electricity is an issue try using glove bag filled with Argon gas. Argon helps dissipate static electricity, making it easy to handle lyophilized powders.

(5) Weigh sample in rotor and subtract off weight measured above to obtain actual sample mass.

NOTE: Frictional forces during spinning increase the temperature inside the rotor and can denature protein samples. Therefore, try to minimize the time that sample is spent spinning above 3.5kHz. (see: Seth A. McNeill; Gor'kov, P. L.; Struppe, J.;

Rotor Volume and Max Speeds

Bruker

3.2 mm – volume = 30 μ L, max speed = 24 kHz

4 mm – volume = 80 μ L, max speed = 15 kHz

Revolution NMR

3.2 mm –

thin-wall – volume = 36 μ L, max speed = 18 kHz

thick wall – volume = 22 μ L, max speed = 25 kHz

Bruker

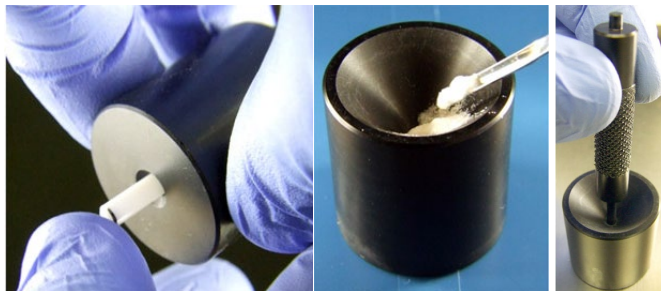
Rotor	length (mm)	max speed	volume
4.0	13	14 kHz	80 μ L
3.2	8	24 kHz	30 μ L
1.9	11	42 kHz	10 μ L
1.3	7.7	67 kHz	1.5 μ L
0.7	5	111 kHz	0.35 μ L

Phoenix

rotor	length (mm)	max speed	volume
3.2	11.6	24 kHz	36 μ L
3.2	11.6	18 kHz	22 μ L
2.5	10	30 kHz	18 μ L
1.6	10	40 kHz	8 μ L

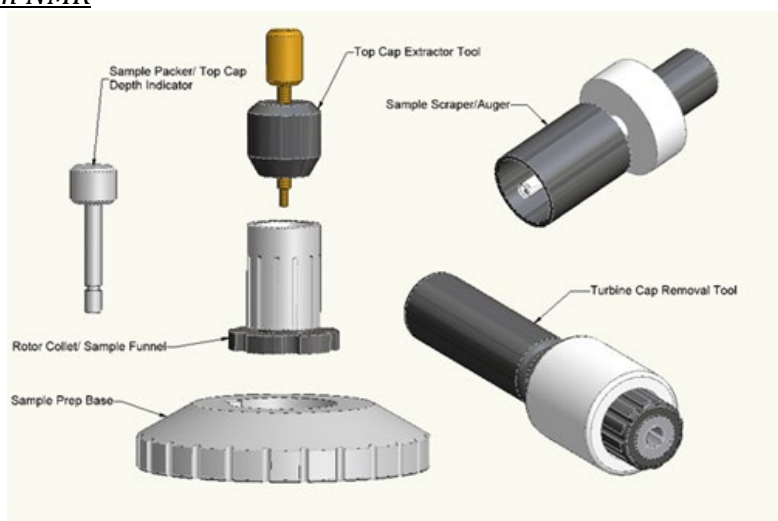
Sample Packing tools:

Bruker



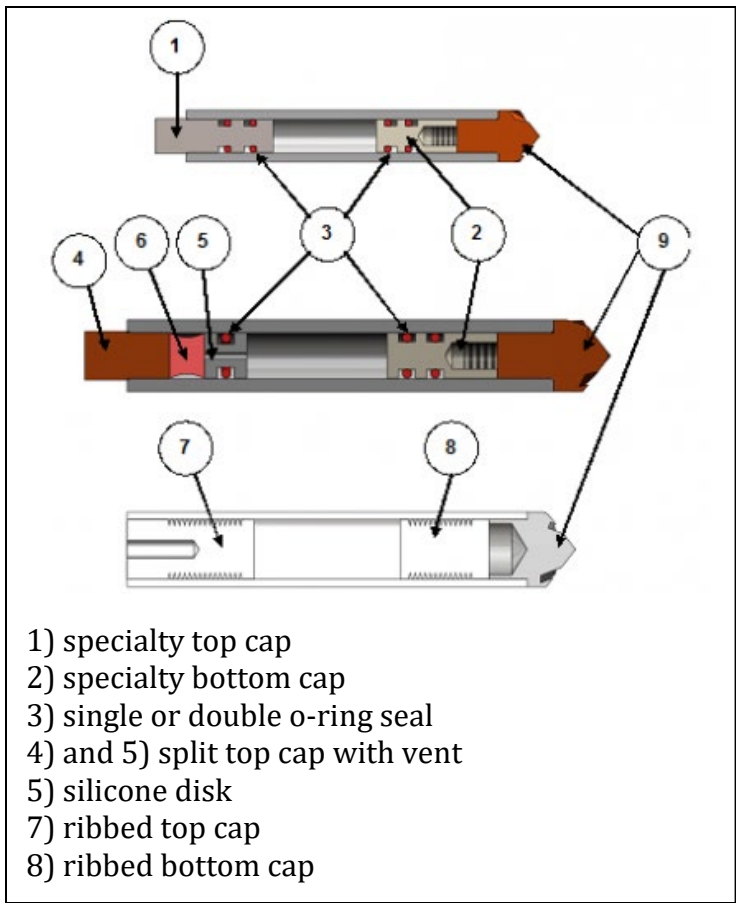
<http://chem.ch.huji.ac.il/nmr/preparation/preparation.html>

Revolution NMR



<https://revolutionnmr.com/products/>

Revolution NMR sealing for semi-liquid and air-sensitive samples



<https://revolutionnrmr.com/products/>