

Insert Gel into Tapered Gel NMR Sample Tube

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- Prepare gels as describe by Yizhou Liu et al.¹
- Wash gels to remove excess monomers.
 - Place gels in 1:1 solution of acetone and methanol for 5 hours.
 - Place gels in chloroform overnight. Repeat the following day.
 - Allow gels to air dry on a glass surface.
 - Store gels for future use.
- Dissolve analyte in a small vial with 300uL of Chloroform-d.
 - Vial should be long enough to allow gel to expand (~4cm).
- Insert a washed and dried gel into the vial.
 - Close cap tightly.
- Place the vial on its side to allow the solution to be absorbed by the gel.
 - This can take several hours.
- Once the solution is absorbed and the gel appears swollen, it can be inserted into the wide section of the Tapered Gel NMR Sample Tube (Fig. 1).²
 - Insert the collar³ (Fig. 2) on to the top of the Tapered Gel NMR Sample Tube.
 - Insert funnel⁴ (Fig. 3) into the other end of the collar as shown in Fig. 4.
 - Using a pair of tweezers, insert gel into the funnel.
 - To minimize the gel from adhering to the glass funnel, place a few drops of chloroform-d into the funnel.
 - Use the wide end of the rod⁵ (Fig. 5) to push the gel into the wide section of the Tapered Gel NMR Sample Tube ensuring the gel is inside the tube by a few millimeters.
 - Place a drop of chloroform-d in the wide end of the Tapered Gel NMR Sample Tube to remove air and plug using the tip of a micro stopper.⁶
 - Cap the narrow end of the Tapered Gel NMR Sample Tube.
- Run NMR experiments.
- Insert sample into the narrow section of the Tapered Gel NMR Sample Tube.
 - Place the collar on to the top of the Tapered Gel NMR Sample Tube to protect it.
 - Use the wide end of the rod to push the gel through the taper until the gel is a few millimeters from the end of the Tapered Gel NMR Sample Tube.
 - Place a drop of chloroform-d in the narrow end of the Tapered Gel NMR Sample Tube to remove air and plug using the tip of a micro stopper.

¹ J. Am. Chem. Soc. 2016, 138 (30), pp 9548–9556

² <http://newera-spectro.com/tapered-gel-nmr-sample-tubes>

³ New Era NE-314-5

⁴ New Era NE-204

⁵ New Era NE-315-3.2/4.2

⁶ Sigma-Aldrich Red-rubber micro stoppers Z165204-12EA

- Cap the narrow end of the Tapered Gel NMR Sample Tube.
- Run NMR experiments
- Extract Gel out of the Tapered Gel NMR Sample Tube.
 - Place the collar on to the top of the Tapered Gel NMR Sample Tube to protect it.
 - Insert the wide end of the rod into the wide section of the Tapered Gel NMR Sample.
 - Push the gel all the way down until the rod touched the 'taper' section of the Tapered Gel NMR Sample Tube.
 - Remove the rod and insert the narrow section of it into the wide section of the Tapered Gel NMR Sample Tube.
 - Push the gel out of the narrow section of the Tapered Gel NMR Sample Tube and onto a glass surface.



Fig. 1



Fig. 2



Fig. 3



Fig. 4



Fig. 5