TXI Probe Characterization Sample
14 Jan 2008; DX

NMR Characterization Reagents for TXI CapNMR Probes
Vendor: Cambridge Isotope Labs

(1) Methanol-p_4 (^{13}C, 99%); Catalog #CLM-359

- 1 g is $125
- For a 200 mM stock solution in DMSO-d_6, a 5-mL volume contains 1 mmole of ^{13}C-methanol-p_4 (33 mg or 40.5 µL @ 25° C. This roughly costs $4 (plus the cost of the solvent).
- Seal remaining methanol-p_4 in a small vial using special black cap, and Parafilm; keep dry.

(2) Urea ($^{15}N_2$, 98%); Catalog #NLM-233

- 1 g is $166
- For a 200 mM stock solution in DMSO-d_6, a 5-mL volume contains 1 mmole of ^{15}N-urea (62 mg). This roughly costs $10.30 (plus the cost of the solvent).
- Keep powder dry.

The total cost of the 5-mL solution is less than $20.

Formula Summary

- Measure 41 µL of labeled methanol (using syringe) and add to 5-mL volumetric flask
- Weigh 62 mg (0.0620 g) of labeled urea (using analytical balance) and add to 5-mL volumetric flask
- Dissolve both to 5.00 mL of DMSO-d_6 in the 5-mL volumetric flask

Make 10 aliquots of about 0.5 mL each into small glass vials sealed with the "special" black caps and Parafilm.

The final solution is dissolved in DMSO-d_6 and contains (asterisks indicate isotopic label location):

- 200 mM ^{13}C-labeled methanol-p_4 (C^*H_3OH)
- 200 mM ^{15}N-labeled urea, which is H_2N^*-(C=O)-N^*H_2

Notes

- DMSO-d_6 is chosen as a solvent to disallow loss of the amino protons by exchange with the solvent.
- Store labeled reagents well-sealed in the refrigerator.