Instructions for Conducting Probe Temperature Calibration

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I. Preparation
   A. Change from N_2 to air if cooling to temperatures below 0 °C
   B. Attach bucket to probe air coil if cooling and fill with necessary slurry
      1. Slurry temperatures should be at least 10 °C lower than temperature you are cooling to

II. Calibration
   A. Type **temp** <return> and set temperature by clicking to the L or R of scale
      1. L mouse click increases/decreases temp by 1 °C
      2. R mouse click increases/decreases temp by 0.1 °
      3. Close temp window
   B. Lock, shim with $CDCl_3$ sample still in instrument
   C. Eject (**e**) $CDCl_3$ standard and insert calibration standard and insert (**i**)
      1. Methanol is used for temperatures <30 °C
      2. Ethylene glycol is used fro temperatures >30 °C
   D. Type **nt=1**, then **ss=0**, and **np=32768** (or some number equal to 2^n, where n is an integer)
   E. Type **ga**
      1. If message of ADC overflow appears, several parameters can be adjusted
         a. type **tpwr=tpwr-12** <return> to lower transmission power
         b. type **gain=0** <return> to lower gain
   F. Place cursors on outside of peaks and type **movesw** <return>
   G. Once temperature has been reached, type **ga** <return>
   H. There should be two resonances, correspondiong to the methyl and hydroxyl protons. Zoom in on most downfield (hydroxyl) peak
      1. Place cursor on peak and type **nl** <return>
      2. Type **r1=cr** <return>
      3. Type **f full** <return>
   I. Zoom in on upfield peak
      1. Place cursor on peak and type **nl** <return>
      2. Type **delta=r1-cr** <return>
      3. Type **f full** <return>
   J. Type **tempcal('m')** when using methanol or **tempcal('e')** when using ethylene glycol; <return> and note the temperature
   K. Repeat steps F-J until temperature reading remains steady
   L. If the calibration temperature does not match that of the external reading, reset the temperature as in step A and recheck calibration until appropriate temperature is reached.