III. Performing ^1H T_1 Estimates

[updated: 13 July 2010]

1. Acquire a normal ^1H 1d spectrum, as described in Section I (sbs_1H1d).
2. Calibrate and set **pw90** , as described in Section II (sbs_1Hpw90).
3. The macro **T1est↵** will automatically setup parameters as follows.
   a) **p1=pw90*2 d1=10 dps↵**

      manual checks:

      set **nt=1 ss=0 vp=80 d2=0.01 ↵**

      ga↵ should show inverted magnetization everywhere.

      d2=10 ga↵ should show positive magnetization everywhere, except perhaps for solvent (or other very slow relaxing protons) peaks.

      Adjust **d2** to give crossovers for important multiplets (see 4 below).

   b) Using an array for **d2** is OK, but presents the problem of what to set **d1** to

   → **d2=0.1,0.25,0.4,0.6,0.9,1.3,2.0,3.5 d1=10 go↵ dsa↵** is usually a good setup for most compounds, but beware that **d1** may be too small, especially for samples sealed in non-O_2 containing atmospheres.

   Acquire with: **go↵** then **dsa↵**
4. Note the crossovers for each important multiplet, and note these points as $\mathbf{d2_{null}}$ times. Especially note the fastest and slowest relaxing protons of interest.
5. $T_1 \approx 1.4 \times \mathbf{d2_{null}}$ for each proton. If $T_1 > \mathbf{1/3 \times d1}$ = 3 sec, then **d1** was too short during the experiment. Repeat with a longer **d1** value.
6. See the T_1 section in VUG for more information, especially how to obtain quantitative T_1 values (rather than the estimates provided by $d2_{null}$). A brief outline on how to analyze and plot quantitative data in VNMR is:

   a) set **Th** (button on the **ds** menu) ;all peaks above line will be found by dpf
   b) **dpf↵** ;display peak frequencies
   c) **fp↵** ;writes list of peak amplitudes
   d) **t1↵** ;performs analysis and outputs T1 analysis
   e) **expl↵** ;displays a plot of the T1 fits
7. To return to normal acquisitions, set:

   T1off↵

   or equivalently: **p1=0 d2=0 vp=20 dps↵**

→ useful related commands: **da ff dc dg dssh dssl pl('all')**