

Measurement of the Spin-Lattice Relaxation Time 'T₁'

All commands written in **bold type** are direct input at the input window prompt or a standard VNMR button. Note that the T₁ measurement (using the **dot1** macro) is available on all of the Facility Varian NMR spectrometers. See the Bruker 'inversion-recovery' handout for details on performing a T₁ measurement on the Bruker instruments.

Setup and data acquisition:

- Log onto the spectrometer and insert your sample into the magnet in the usual fashion.
- Choose the desired nucleus and solvent in the usual fashion. You may spin the sample but it is not necessary.
- Set the temperature to 20°C:
 - Type **temp=20 su**. If this doesn't work do the next step.
 - Type **temp**. In the Temperature Control Window select the 'Turn temperature control on at ...' radio button and use the slider bar to select the desired temperature.
- Lock the sample and check for lock power saturation. Do not saturate the lock channel.
- Tune the probe. Skip this step if using the Mercury 300.
- Shim the sample. If you are not spinning the sample you may wish to 'touch up' the x-y shims.
- Verify that the 90° pulse width (pw90) is accurate. The dot1 (do T₁) macro depends upon a properly calibrated pw90. If you don't know how or haven't done this in the past see our "How to calibrate a 90° pulse" handout.
- Run a normal 1D proton spectra then choose the desired spectral width using the cursors and press the **[Expand]** button. Type **movesw ga** and then wait for the experiment to complete.
- Type **dot1**. This will setup all of the parameters for a T₁ experiment including d1 (first sequence delay), pw (length of the rf pulse), p1 (first pulse width), nt (number of transients), and d2 (incremented delay in the 1st directly detected dimension).
 - You will be prompted for:
 - ENTER MINIMUM T1 EXPECTED (sec): insert a reasonable guess.
 - ENTER MAXIMUM T1 EXPECTED (sec): insert another reasonable value.
 - ENTER EXPERIMENT TIME (hours): insert your desired experimental time.
 - In the Text Window you will see something like:
 - STANDARD 1H OBSERVE
 - T1 EXPERIMENT
 - EXPERIMENT SET UP USING 15 TAU VALUES (this value will vary)
 - APPROXIMATE ACQUISITION TIME 1.59 HOURS (this value will vary)
 - Make the necessary adjustments (nt for example) to tailor the acquisition time to your needs.

- Type **gain?** Set the gain to the value returned in the Status Window (for example 20) by typing **gain=20**.
- Type **au**. (Submit experiment to acquisition and process data.)
 - You can view the experiment progress by typing **ds(1) vsadj wft dssh**. (Display 1st spectrum, Automatic vertical scale adjust, Weight and Fourier transform 1D data, Display stacked spectra horizontally).
- Type **da** (display acquisition parameter arrays) or **arraydim?** (display array dimension)

Processing the data:

- If arraydim=15, type **wft ds(15)**. This will perform a weighted Fourier transform of the last spectrum acquired.
- Phase the spectrum properly and select a threshold.
- Type **dll** to display an indexed line list in the Text Window or **ll** to send an indexed line list to the printer. We suggest a hard copy to make the selection process easier.
- Set the parameter 'npoint' to the number of peaks found. If the ll (line list) found 20 peaks type **npoint=20**. If a message appears in the Status Window 'Variable "npoint" doesn't exist. Type **create('npoint')**.
- From the line list, suppose you are interested in the T₁ values of peaks 3, 5, 12, 16, 17, and 20. Type **fp(3,5,12,16,17,20)**.
- To now perform the T₁ analysis type **t1**. The results will be displayed in the Text Window. If you would like a hardcopy of the T₁ analysis type **printt1**.