University of Delaware

The UD NMR Laboratory Application Notes 1.4

TXI Probe User's Quick Guide

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Sample handling

NMR Tube: The TXI probe is a 5mm triple resonance probe. Use only 5mm Wilmad 528 or higher tubes. Inspect the tube carefully before use. Carefully clean the tube and spinner with ChemWipses. Always check the sample depth and make sure that the depth is set to **20mm**

Temperature Control

Use **edte** to control the sample temperature. In the **edte** interface, when the probe heater is turned on (by clicking heater ON button), the BCU05 unit will start automatically. Set flow rate of the VT control to 673 l/h. The temperature control range should be kept with in the range of -10 to 80 C.

Probe Tuning

The TXI probe proton channel tuning is very sensitive upon the change of sample or solvent. Use **atmm manWbsw** to start a tuning interface. If a minimum of wobble curve is not displayed in the window, stop **atmm** and check parameter WBSW with **eda**. One may need to change WBSW to 10 or 20 MHz before a minimum point of the wobble curve can be displayed. When use command **atma**, make sure WBSW is set to at least 10 MHz.

Shimming

Always start with a good shim matrix by retrieving shim file with **rsh** *shimfilename*. The shim file is stored in the format of "txi_h2o_xxxxxx", where xxxxxx (ddmmyy) indicates the date the file was saved. Try to find most recent one for use. To find the txi probe related shim file, type **rsh txi*** , then chose the file by clicking on it. It is also good practice to save your own shin file. To do so, use **wsh** *myshimfilename*.
For the H_2O/D_2O sample, one should always use gradient shimming. Type **topshim** (**topshim 3d** for water sample) to start shimming. For common organic solvents, start with the same shim file as described above. Shimming manually or type **topshim** usually provides a satisfactory result.

Proton 90-degree pulse calibration for a H20/D2O sample

Make sure the probe is properly tuned and shimmed. Use **rpar PROTON all** to load the standard proton parameter. Type **gpro** to load standard power levels and pulse widths into the current experiment. Use **eda** to change pulse program from zg30 to zg. Set **td** to 4K, **ns** to 1, **ds** to 0, **rg** to 1, and o1 to 2820.5. Double the current value of p1. For example, if p1 is 8 usec., change it to 16 usec. In **gs** mode, watch H_2O FID while change pulse width p1 in a small step at the time (a few tens of one microsecond) until the beginning part of the FID reaches minimum. Half of the current p1 is the 90-degree pulse. Set value of p1 in current experiment. Once the hard pulse width is determined, use **edprosol** or **pulse** command to calculate the power levels corresponding to longer 90 pulses. For example, if the 90-degree pulse is 8 usec. at PL1 of 0, the TOCSY spin-lock pulse (30 usec.) can be calculated by typing **pulse 30u**. The **edprosol** provides a graphic interface for these calculations.

Common Experiment Files

The common acquisition files are listed in the following table.
General: Use **rpar** *filename* **all** to read the acquisition parameters into current experiment. Retrieve power levels and pulse widths from **prosol** using **gpro**. Use **ased** to edit proton 90-degree pulse widths. Use **eda** to check and edit NS, SW, SW1, O2, etc. In the **gs** mode, set appropriate **rg** before starting acquisition.
C13-HSQC/N15-HSQC: It is suggested set a large **ds** (32 or more), adjust shim z1 to restore any loss in lock level after experiment starts.

TXI Probe Common Acquisition Files

Experiment	File Name	Comment
Water suppression	txiwspr	Water suppression using pre-saturation
Water suppression	txiwswg	Water suppression using watergate
COSY	txicosyphwg	COSY with watergate
TOCSY	tximlevwg	TOCSY (mlev) with watergate
TOCSY	txidipsiwg	TOCSY(DIPSI) with water suppression
NOESY	txinoesygpwg	NOESY with watergate
ROESY	txiroesygpwg	ROESY with excitation sculpting
15N-HSQC	txin15hsqcf3	15N-HSQC using f3 channel
^{13}C-HSQC	txic13hsqcsisp	13C-HSQC (shaped pulse for all 180 pulse in f2 channel)