

Technical Bulletin #C0010: Proton with CapNMR

This bulletin describes how to use the CapNMR probe for Proton on a Varian console/system.

Begin by loading an appropriate 'Proton' sequence. The Proton sequence should pull in the pulse width (pw) and transmitter power (tpwr) from the probe file. (If the Proton pulse sequence is not available, use 's2pul' and manually set the pw and tpwr. Make sure to use the appropriate power levels (see Figure 1). The solenoid microcoil in the CapNMR probe requires less transmitter power than traditional probes. The standard pulse width (pw90) is calibrated at installation for a transmitter power of 45 dB (tpwr=45).

Parameter	Varian Range	Bruker Range
Lock power	1 to 8	-55 to -45
Lock gain	34 to 48	110 to 130
Transmitter power level (dB)	40 to 45	15 to 20
90° Transmitter pulse width (µsec)	2.5 to 5	2.5 to 5
90° Decoupling pulse width (µsec)	5 to 12	7 to 12

Figure 1. Appropriate power levels for the CapNMR Probe

Type 'dps' to display the pulse sequence and make sure the sequence looks like Figure 2. Make sure the transmitter is set to proton (tn='H1'), the coil is properly tuned and matched, and the acquisition parameters (spectral window, number of points, etc...) are appropriate.

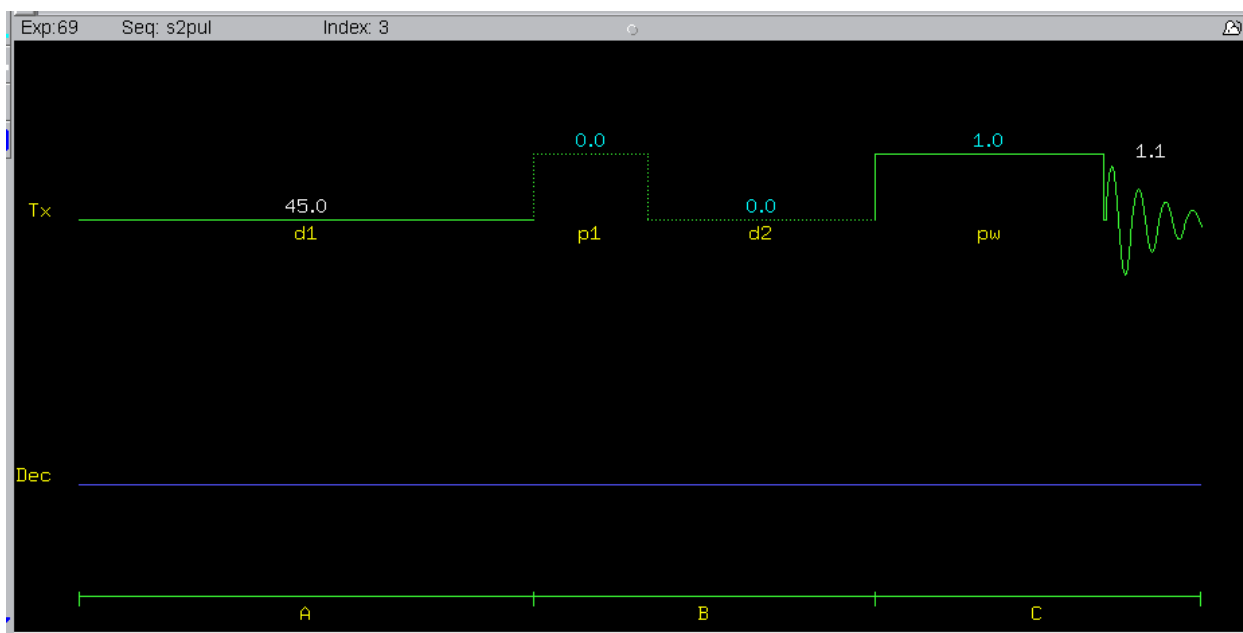


Figure 2. Proton Pulse Sequence

However, the pulse width is probe/console specific so your pulse width (pw) value should be different than the 1.0 value shown in Figure 2 and Figure 3. The pw90 for proton was calibrated with an array of the pw parameter in a Proton sequence and for tpwr=45, the pw90 for this probe was 2.6 microseconds.

Here is an example of the parameter set used for the Proton pulse width calibration (Note: There is a very long delay (d1=45) and the pulse width (pw) is arrayed. In order to view this window, click on Process and select Text Output and then type 'dg').

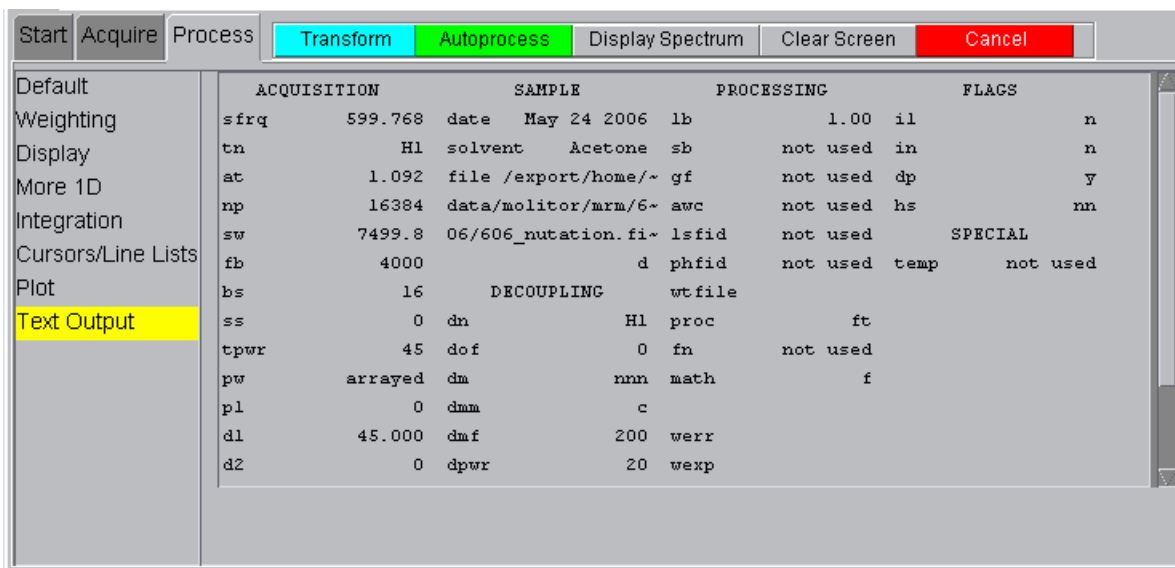
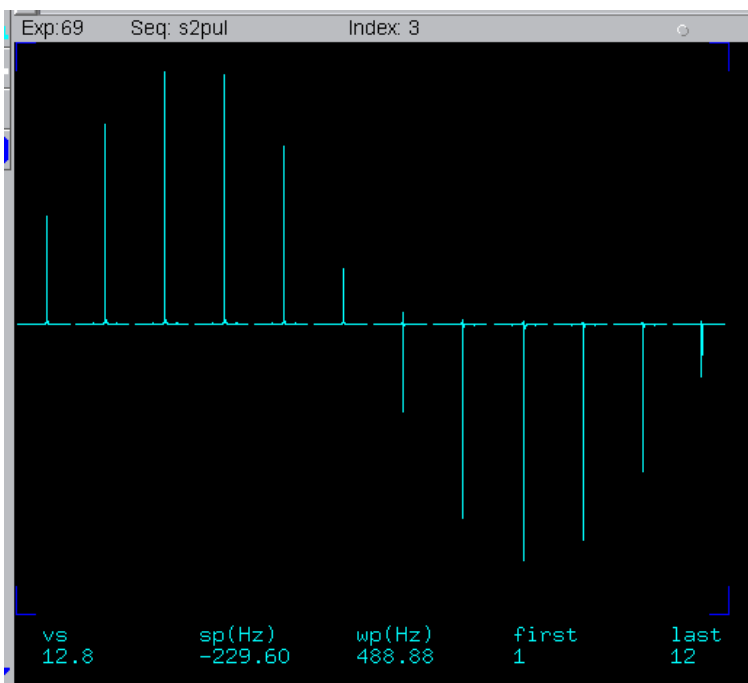


Figure 3. Proton Parameters for the CapNMR Probe

This will give a natural nutation of the proton signal. (Absolute intensity mode is required aig='ai'.) During this procedure, make sure to use the tpwr is the same and the tpwr in your desired experiments. The CapNMR can sometimes have a very short pulse width of less than 2 μ sec at tpwr=45. If a longer pulse width is desired, just decrease the tpwr until you receive the desired pulse width.



Sample and Data Set:

The sample used was 10% (v/v) 2-ethyl-1-indanone in CDCl₃. Pure 2-ethyl-1-indanone is a clear, brown liquid with a T1 relaxation of approximately 0.8 seconds. This data set was taken on a **5-μL of 10% 2-ethyl-1-indanone (519 μg; 3.2 μmol; v/v in CDCl₃)**, which was injected and then pushed into a 5 microliter flowcell with more CDCl₃ solvent. This data was taken on a Varian Inova at 600 MHz with a TXI (Triple Inverse ¹H, ²H, ¹³C, ¹⁵N) CapNMR probe with a 5 microliter enhanced flowcell in less than 1 minute.

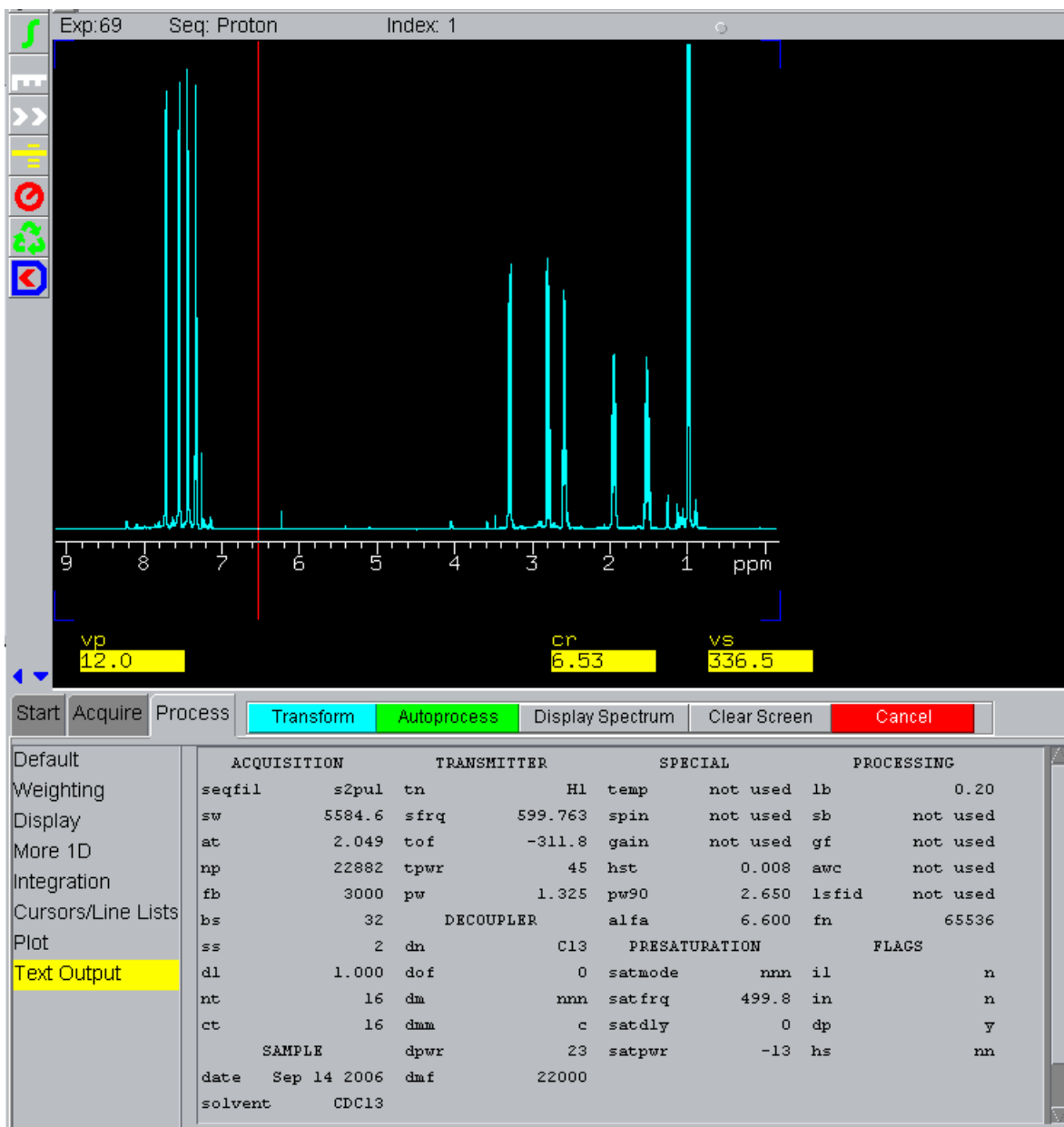


Figure 4. Proton Spectrum from 10% 2-ethyl-1-indanone

**Sample: 5 μ L injection of
10% 2-ethyl-1-indanone
[519 μ g; 3.2 μ mol] in CDCl_3**

Time: less than 1 minute
(16 scans; 55 sec)

S/N of aliphatic peaks ~1180

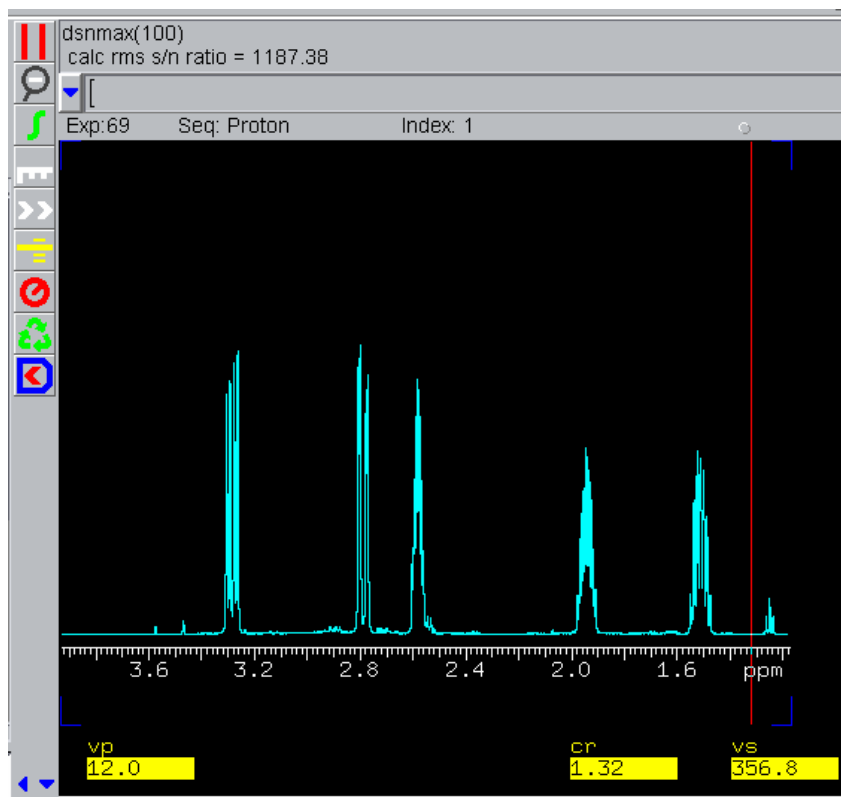


Figure 5. S/N from the aliphatic region of 10% 2-ethyl-1-indanone

**Sample: 5 μ L injection of
10% 2-ethyl-1-indanone
[519 μ g; 3.2 μ mol]
in CDCl_3**

Time: less than 1 minute
(16 scans; 55 sec)

S/N of aromatic peaks ~1880

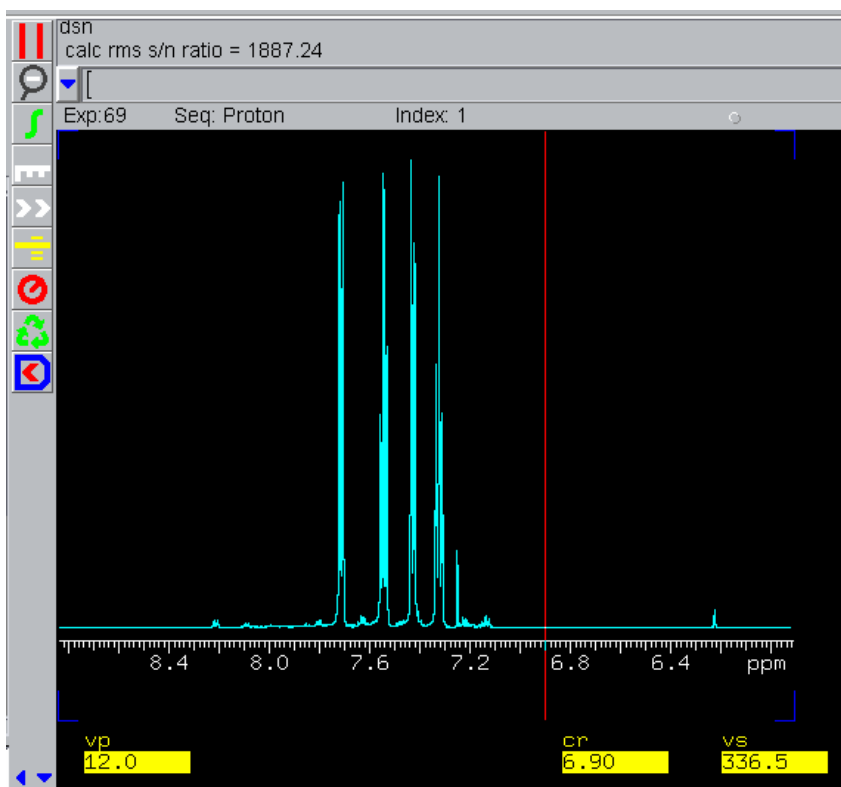


Figure 6. S/N from the aromatic region of 10% 2-ethyl-1-indanone

Note: There are many ways to take S/N but the two most common ways are dsn and dsnmax. The 'dsn' command will take the relative S/N of the current cursor position while the dsnmax will analyze a region for the best S/N. The method used for the aliphatic region was dsnmax(100) which compared 100 Hz windows within the noise region. For dsnmax, the cursor must be placed on the very right-hand side on the screen as the macro measure up field from the cursor.

Injection Method

A Hamilton Gastight 25- μ L syringe was filled with clean CDCl_3 solvent and used to rinse the probe. Then, 5- μ L of 10% 2-ethyl-1-indanone (519 μ g; 3.2 μ mol; v/v in CDCl_3) was drawn into the syringe and injected into the probe. Using the same syringe, 12 μ L of clean CDCl_3 was picked up and injected into the probe to push the 5- μ L sample into the NMR flow cell of the CapNMR probe. The 12- μ L Push Volume was calibrated in advance of sample injection.

Screenshots

All of the screenshots shown in this Technical Bulletin are from a Varian Inova console running Varian VnmrJ 2.1B software on a Unix workstation. The magnet was an Oxford 600 MHz magnet. (For more information on the Varian VnmrJ software, see <http://www.varianinc.com/cgi-bin/nav?products/nmr/>)