Technical Bulletin #C0060: gHMBC with CapNMR

This bulletin describes how to use the CapNMR probe for Ghmbc (^{1}H-^{13}C) on a Varian console/system.

Begin by loading an appropriate 'Proton' sequence. 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

Once there is a good Proton spectrum, type the macro 'Ghmbc' into the command prompt and this will load a slightly modified gHMBC pulse sequence. You can view the pulse sequence with a dps command. The sequence should look like the sequence in Figure 2.

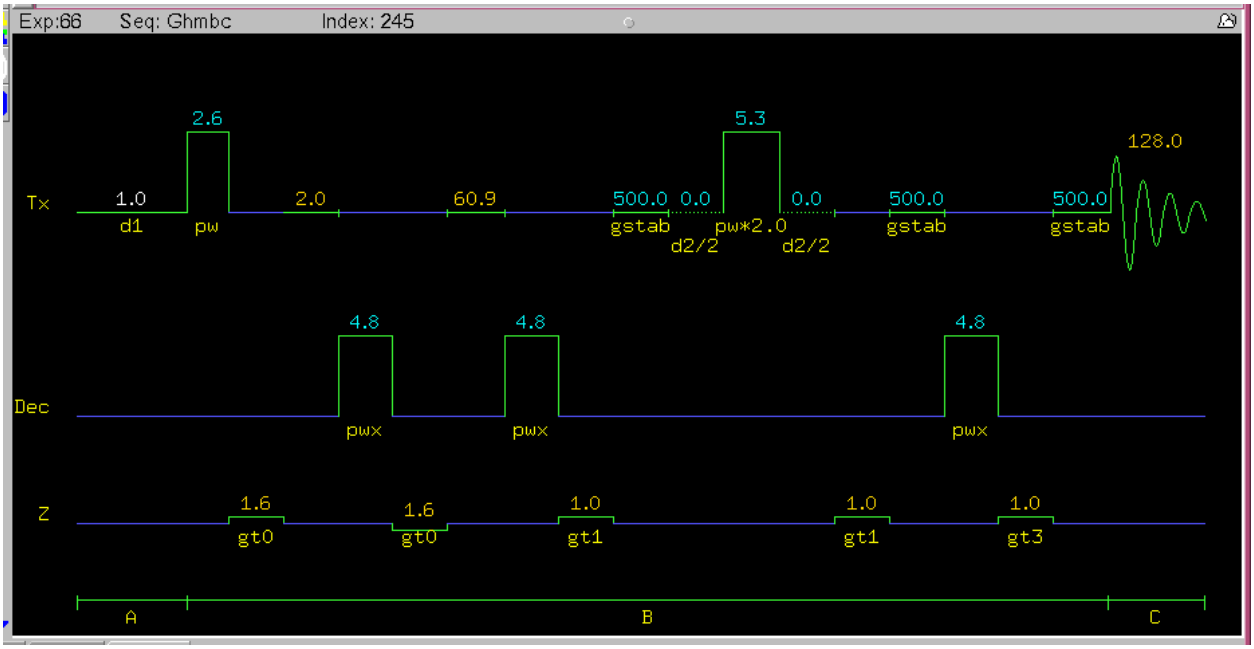

Figure 2. Ghmbc Pulse Sequence

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However, the pulse width is probe/console specific so your pulse width values (pw & pwx) should be different then the 2.6 and 4.8 values shown in Figure 2 and Figure 3. The pw90 for proton was calibrated with an array of the pw parameter in a Proton sequence. For tpwr=45, the pw90 for this probe was 2.6 microseconds. The pwx90 for carbon was calibrated with a pwxcal pulse sequence and for pwxlvl=45, the pwx90 for this probe was 4.8 microseconds.

Here is an example of the parameter set used for the Ghmbc pulse sequence. In order to view this window, click on Process and select Text Output and then type 'dg'.

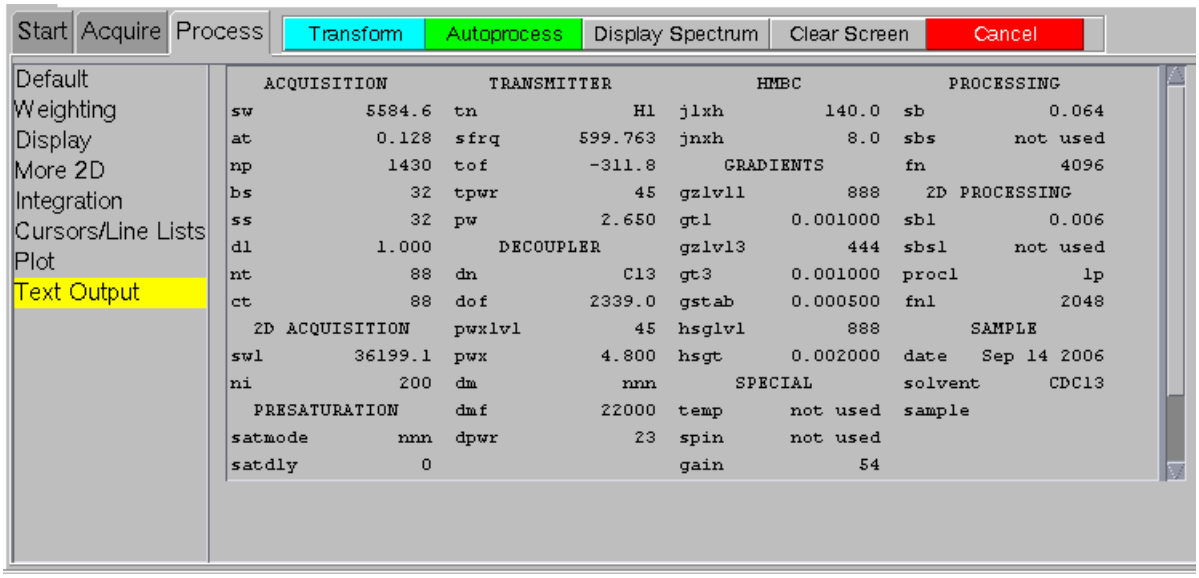

Figure 3. Ghmbc Parameters for the CapNMR Probe

IMPORTANT: GRADIENT POWER LEVELS
The CapNMR probe has a much stronger z-gradient then most standard tube probes. Most common tube probes are 50-80 gauss/cm at 10 amps while the CapNMR is commonly 350-400 gauss/cm at 10 amps. The default gzlvl1 and hsglvl values from Varian are 2000. You may have seen these values as high as 4500 for some tube probes. However, due to a more efficient gradient coil, not as much gradient power (gzlvl) is required for the same gradient field.

A standard gradient value (gzlvl1 & hsglvl) for the CapNMR probe is 700 to 900.
A standard gradient value (gzlvl3) for the CapNMR probe is 350 to 450.

These gradient and homospoil gradient values are automatically calculated from the gcal value in the probe file. The gcal value is calibrated at installation with a profile sequence, which performs a z gradient echo, and is saved in the probe file. Make sure your probe is set correctly (check with 'probe?') and that the 'probegcal' value is correct.

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Sample and Data Set:
The sample used was 10% (v/v) 2-ethyl-1-indanone in $CDCl_3$. 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 $CDCl_3$)**, which was injected and then pushed into a 5 microliter flowcell with more $CDCl_3$ solvent. This data was taken on a Varian Inova at 600 MHz with a TXI (Triple Inverse ^{1}H, ^{2}H, ^{13}C, ^{15}N) CapNMR probe with a 5 microliter enhanced flowcell in 4 hours.

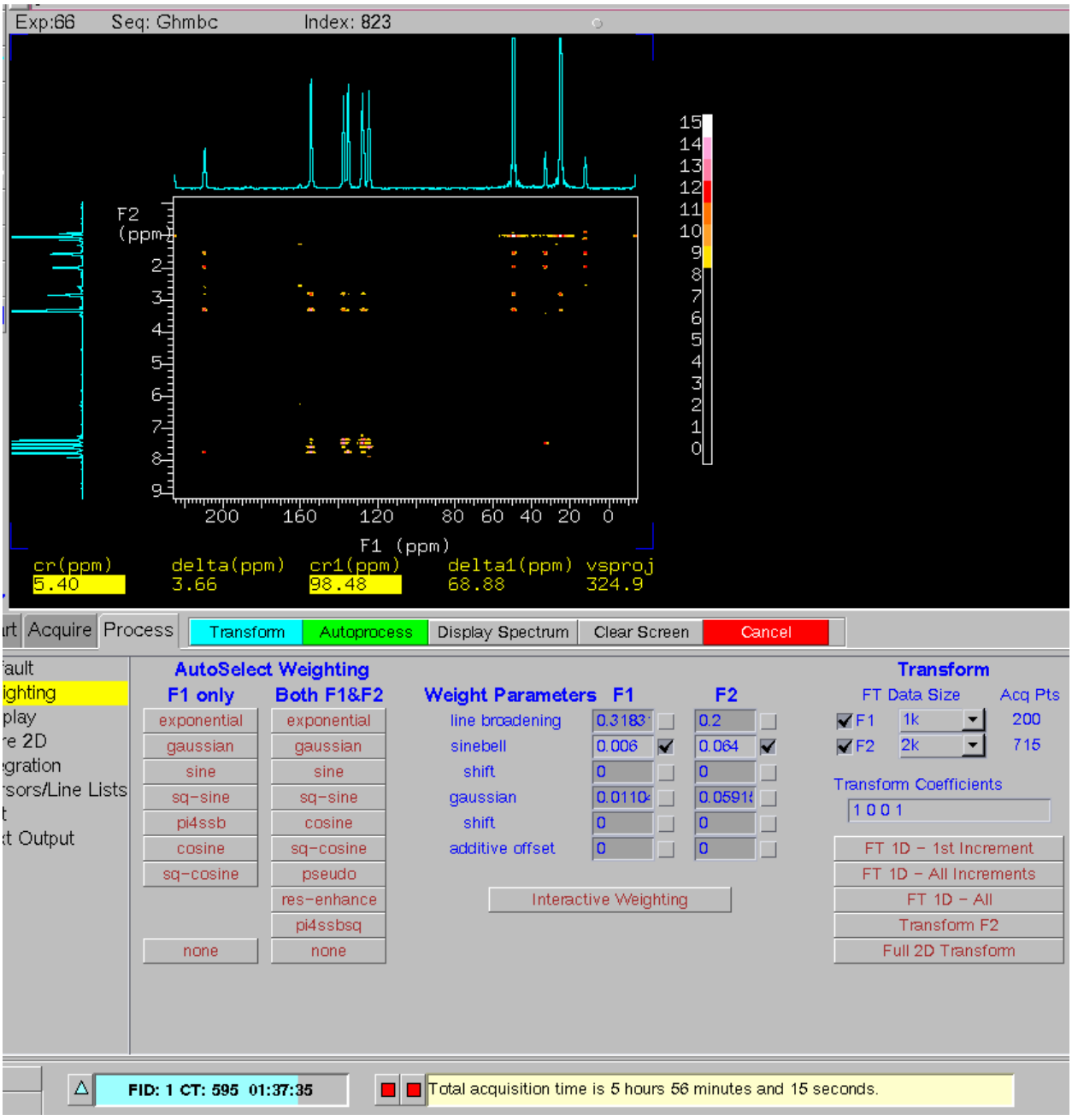

Figure 4. Ghmbc Spectrum from 10% 2-ethyl-1-indanone

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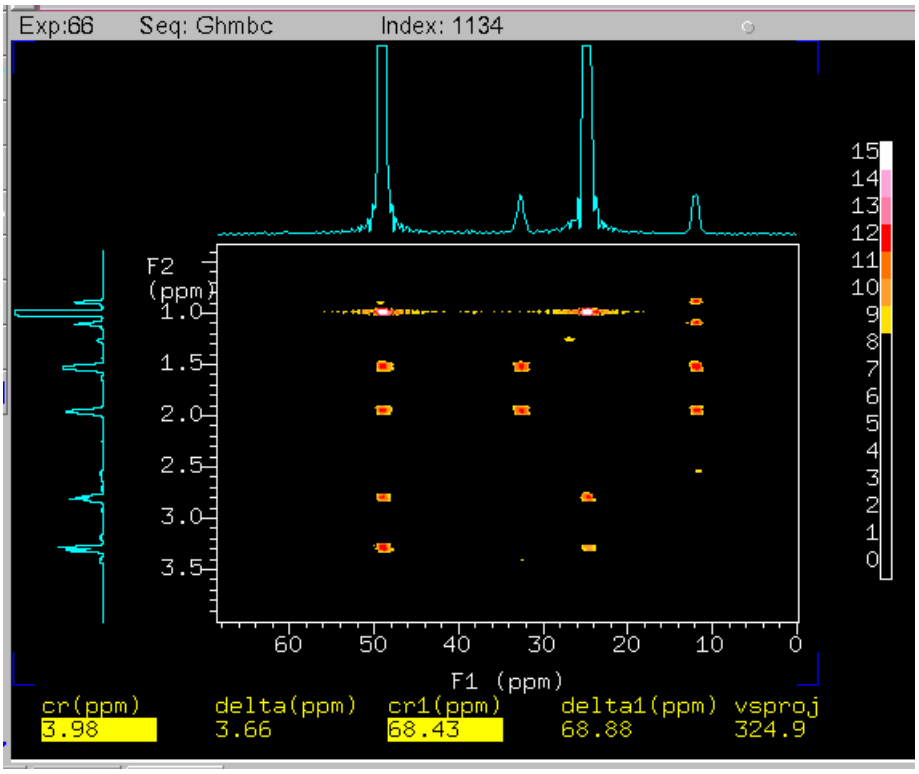

Figure 5. Ghmbc Spectrum (Aliphatic Region) from 10% 2-ethyl-1-indanone

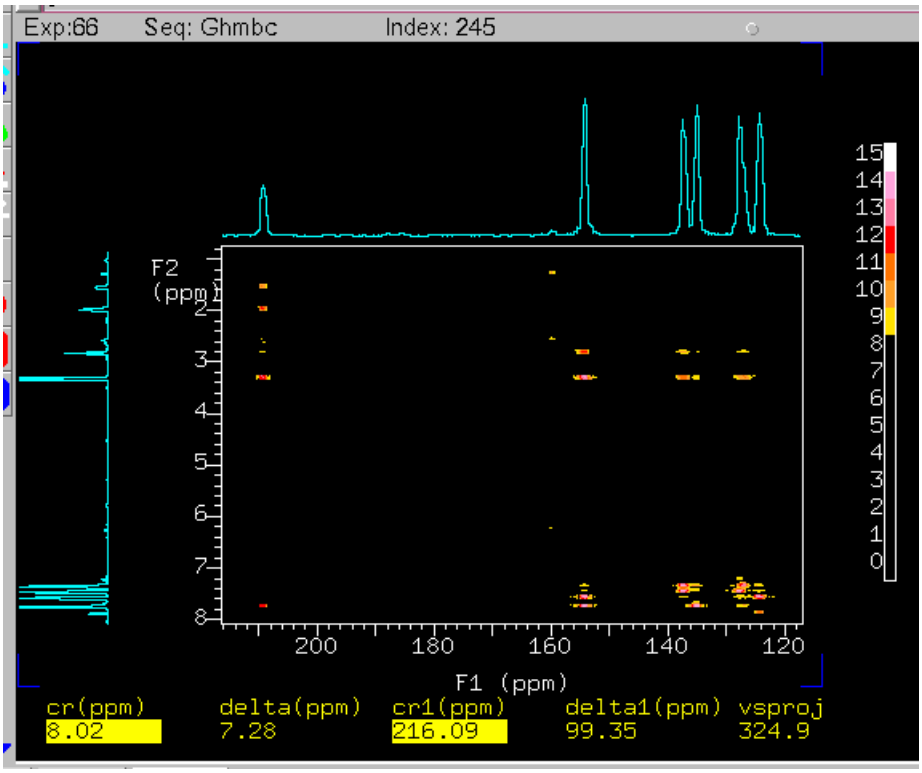

Figure 65. Ghmbc Spectrum (Aromatic Region) from 10% 2-ethyl-1-indanone

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Injection Method
A Hamilton Gastight 25-µL syringe was filled with clean $CDCl_3$ solvent and used to rinse the probe. Then, 5-µL of 10% 2-ethyl-1-indanone (519 µg; 3.2 µmol; v/v in $CDCl_3$) was drawn into the syringe and injected into the probe. Using the same syringe, 12 µL of clean $CDCl_3$ was picked up and injected into the probe to push the 5-µL sample into the NMR flow cell of the CapNMR probe. The 12-µL 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/)

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