Using the Deuterium Broadline Solids Probe on the GN300WB

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I. GENERAL

The Deuterium Broadline probe is used to study the motion of deuterium nuclei in solids. The solid state spectra of deuterium nuclei can be very broad (>100kHz) and, therefore, fast digitization rates are needed. Spectra are normally recorded using the 2090 Digital Oscilloscope which can record spectra using a dwell time of 0.5 msec. The temperature range of the probe is -150 to 200 ^{o}C. Either a 5 mm or 10 mm sample coil can be used, depending on sensitivity requirements.

II. SAMPLE HOLDERS

Sample holders can be conveniently made from 5 or 10 mm NMR tubes. For added strength, a thick wall NMR tube should be used for the 5 mm sample holder. The total length should be 18 mm. A standard 10 mm NMR tube is strong enough to use for the 10 mm sample holder and should have a total length of 20 mm.

After packing your sample in the holder, use a strip of teflon tape to seal the open end.

III. SAMPLE INSERTION

1. Loosen the thumb screws and lower the probe.

2. Remove the RF shield "can".

3. Remove the insulating teflon cover.

4. Insert the teflon-taped end of your sample through the coil and center it.

5. Replace the teflon cover and the RF shield.

6. Raise the probe into the magnet and tighten the thumb screws.

7. Turn on the drive pressure to purge the sample chamber. Use about 5 psi (you may need to use a greater pressure for VT).

8. Tune the probe. Set LK = -, LT = -, IF = -1 dB.

IV. 90^o PULSE DETERMINATION

Solid state deuterium spectra are most often obtained using the quadrupolar echo sequence (see below); therefore, a precisely determined 90^o pulse is important. A good sample to use for this purpose is hexamethylbenzene-d_{18} which can be obtained from lab staff. This compound gives an intense and fairly narrow powder pattern. Therefore, the **1PULS** sequence can be used to determine the ^2H 90^o pulse. As usual, the signal (powder pattern) will flip up and down and have null crossings at the proper pulse angles.

V. QUADRUPOLAR ECHO PULSE SEQUENCE

The Quadrupolar Echo pulse, "QUADEC", is used to obtain wideline deuterium spectra. The echo refocuses after the receiver "dead time" thus allowing the first few points of a rapidly decaying FID to be recorded. These points are lost when **1PULS** is used which causes a distortion in the powder pattern. The sequence consists of the following:

```
QUADEC
QUADRUPOLAR ECHO

#  1:  D5, G
#  2:  P2, G
      XMTR PHASE:  A
#  3:  D8, G
#  4:  P2, G
      XMTR PHASE:  B
#  5:  D9, G
#  6:  A, G
      RCVR PHASE:  A
#  7:  D6   JUMP TO #   1

PHASE A=2*S    MODULO:  4
PHASE B=1   MODULO:  4
FCN #=      0
```

P2 is the 90^o pulse width and **D8** and **D9** are delay time. (For I = 1 nuclei, two 90^o pulses yield an echo.) Ideally, **D8** and **D9** should be the same, but they can be independently adjusted. For instance, you may want **D9** shorter than **D8** so that you can accurately determine the echo maximum and then left shift (**LS**) points in the FID up to it. The normal receiver delay time, **CR**, should be set to **DE**/**DW** = 1 and the receiver gating pulse, **RG**, should be set to its minimum

value of 1 msec. **P 2** can be adjusted slightly for individual samples so that both transitions are of equal intensity (using the wrong **P 2** can cause one transition to be more intense than the other.)

VI. T_1 MEASUREMENT VIA INVERSION RECOVERY/QUADRUPOLAR ECHO

This experiment, "T1QUEC", works just like a normal inversion recovery experiment except that a quadrupolar echo is used to generate the FID after the 180^{o}inversion pulse:

```
T1QUEC
INV. REC. T1 MEAS. W/ QUAD. ECHO

#   1:  D5, G
#   2:  P4, G
#   3:  D0, G
#   4:  P2, G
       XMTR PHASE:  A
#   5:  D8, G
#   6:  P2, G
       XMTR PHASE:  G
#   7:  D9, G
#   8:  A, G
       RCVR PHASE:  A
#   9:  D6   JUMP TO #   1

PHASE A=2*S     MODULO:  4
PHASE B=1    MODULO:  4
FCN #=       0
```

DO is the list of variable pulse delays to be used in acquiring successive spectra. Use **G S** (Go - Save) to acquire your data.

VII. LINESHAPE SYMMETRIZATION

Ideally, the deuterium powder pattern should be symmetrical if it is centered on resonance. However, this is often not the case. The powder pattern can be artificially symmetrized in the following manner:

1. Starting with the Real and Imaginary FID's in memory block 0 and a size of 4K (standard size when using the 2090 Digitizer), change **C B** to 2K. You will now see only the Real FID in memory block 0.
2. Now move to memory block 1 by typing "1". The Imaginary FID is located in this block.
3. Type "**ZER**" to zero the Imaginary FID.
4. Move back to memory block 0 by typing "0".
5. Change **C B** back to 4K. You will now have the Real and zeroed Imaginary FID's which can be worked up in the usual manner. This will yield a symmetric spectrum with increased

VIII. VARIABLE TEMPERATURE

The probe can be used to record spectra at temperatures between-150 and 200 ^{o}C. To obtain maximum efficiency of the heater/cooler system, connect the VT gas outlet from the air box to the inlet of the cooling coil then plug the heater drive gas outlet directly into the probe after it has been positioned inside the magnet. When you need to lower the probe, disconnect the heater line from the probe.

Set the desired temperature on the controller and place the drive gas coil into a coolant bath. You can use ice water, a dry ice slush, or liquid nitrogen depending on the temperature needed. Place a jackstand under the dewar used for the coolant bath. Of course, you do not need a coolant bath when above ambient temperatures are desired.

Wait for the temperature to equilibrate at the set-point and then **tune the probe**. Begin acquiring data. If you are using liquid nitrogen coolant, you will need to replenish it often. At lower temperatures, you will need to use increased flow rates, i.e., turn up the drive pressure.

When finished, remove the coolant from the drive gas coil and allow the probe to warm up to room temperature. The heater can be used to expedite this by setting the temperature to 20 ^{o}C and setting the power to no greater than 20%.

After the probe has been equilibrated at room temperature, disconnect the drive line from the base of the probe and lower it to retrieve your sample.