

UWB370B

ZG-22JAN92UD
GWW-14SEP88CD

Operation of the GN300WB Solids Console

Handout #370B

As with other lab instruments, this is only a reference guide. A basic understanding of the software and the system operation is a necessary prerequisite to using the solids accessories. Therefore, before you can be checked out to run solid samples, you must be checked out for solutions operation. This guide was written with the assumption that the user is already very familiar with normal solutions operation of the instrument.

There are many precautions that must be followed when running solids to prevent both damage to the instrument and injury to yourself. Study the section on precautions **VERY** carefully.

This handout includes sections which describe the changes necessary for observing different nuclei. These are included to give a better understanding of the instrument. HOWEVER, ALL PROBE AND NUCLEI CHANGES WILL BE DONE BY FACILITY PERSONNEL ONLY.

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Sample preparation

Most solid state NMR experiments need use the magic angle spinning (MAS) pencil probe which equipped with pencil like spinner or rotor.

The spinning rotor has a outer diameter of 7.5mm and has a sample volume about 200 mm³. You are recommended to have a full rotor of sample, however, experiments using smaller amount of sample still can be performed but with less sensitivity. For details of using the pencil probe, you are referred to handout #UWB374.

All samples should be in powder form and underpacked. No solution or gel type sample should be attempt to spin. Users should purchase their own spinner if they are going to use it regularly. However, user can borrow the spinner from the lab for uses and they are responsible if the rotor is damage because of the user's fault. Ordering information of rotor can be obtained from lab staff.

Precautions

PROBE DAMAGE WILL OCCUR IF ANY OF THE FOLLOWING PRECAUTIONS ARE NOT OBSERVED.

1. The transmitter pulse length (**P5** or **P2**) must **NEVER** be longer than 20 msec and should normally be set in the range of 1-10 msec. **ALWAYS** enter the necessary units following any numerical entry. **NEVER** assume that the default units are correct.
2. **ALWAYS** check for the right **SF** and **F2**. Any mistake to set SF or F2 off by 1MHZ will damage the amplifiers. **ALWAYS** make sure the spectrometer is setup for the nucleus you are observing.
3. **NEVER** give the **DN** command when using the decoupler for solids experiments. Instead, using either L1 (for 1PDNA experiment) or L2 (for CPBL experiment) to change decoupler power level.
4. Decoupler power is applied during the entire acquisition time (**AT**) for the CP and 1PDNA experiments. The decoupler is also on during the CP mixing time (**D2**), during the observe pulse (**P5**), and during the preacquisition delay (**A**), but these times are relatively short by comparison. **ALWAYS** check the acquisition time (**AT**) immediately before acquiring data and minimum **AT** should be used. If sample linewidths are on the order of 100 Hz, **AT** need only be about 10 msec. It is better to err on the side of poorer resolution than to take a chance on damaging the probe. (If zero filling was done once before the previous Fourier transform, the acquisition time will have doubled, and the **CB** command must be given to reset the data size.) The decoupler unit must **NEVER** be on for more than 50 msec total. If you need use longer acquisition time, you need lower the decoupling power.
5. The duty cycle =
$$\frac{\text{time transmitter is on}}{\text{total time for one sequence}}$$

This ratio should **NEVER** be greater than 10% for the decoupler transmitter.

6. **NEVER** start the experiment if you have anything that you are not sure of. Check with lab staff before you type **ZG**.

7. **NEVER** disconnect any cables (for tuning or any other purpose), nor change samples without first checking that:
 - a) all amplifiers are off
 - b) the instrument is not pulsing (check both the console lights and the computer monitor to verify this).
8. When reconnecting any cables, **ALWAYS** check the condition of the connector. If any problems are noted, please contact lab staff.
 - a) If the center contact is bent, it should either be straightened (i.e., disconnect the cable at both ends, move away from the magnet, and use pliers to gently press the contact back to the center), or not used until a replacement cable can be made.
 - b) If the cable end is loose, either tighten it (again away from the magnet), or wait for a replacement.

FAILURE TO OBSERVE THESE PRECAUTIONS COULD CAUSE PERSONAL INJURY AND/OR INSTRUMENT DAMAGE.

Sample placement in the probe

ALWAYS make sure the spectrometer is not pulsing when you change samples.

Before attempting to spin the rotor, inspect the outside for any scratches or abrasions. A scratched rotor may fail while spinning resulting in an explosive release of rotor shards and could cause major probe damage and personal injury. Do not use a rotor if it is found to be defective. Also, due to the ever present potential for rotor failure, never spin the rotor with the probe door open and always wear safety glasses when in the vicinity of the probe.

Make sure the end of the rotor is marked so the spinning speed can be detected by the spinning monitor.

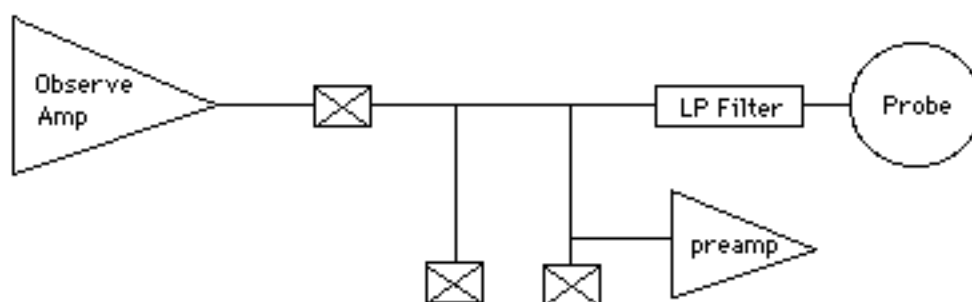
You merely need to place the rotor nose first into the probe, **CLOSE DOOR** and raise the probe into the magnet. Turn the bearing pressure up to about 17 psi and then bring the drive pressure up to about 10 psi. The rotor should begin spinning smoothly. If not, turn the driving pressure off and turn up to 10 psi quickly. If still not, you may need to repack the sample or see lab staff to check the rotor.

When increasing the spin rate, do so in small increments. The bearing can be kept at about 17-20 psi while the drive is increased for the desired spin rate. You should take a spectrum at 4 kHz to see weather the maximum speed is necessary or not. If necessary, set bearing to 20 psi and turn the driving gas to the highest pressure you can get (typically 30 psi and it amy vary). At this pressure, you should be able to reach 5.6 kHz spinning speed.

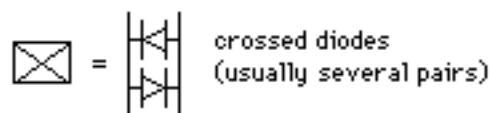
When stopping the rotor, leave the probe up in the magnet and slowly decrease the drive pressure so that the rotor does not suddenly crash to a halt. Remember that the rotor is ceramic and could explosively shatter. Keep the probe door closed and wear safety glasses! Check the spinning speed monitor, after it is below 100 Hz turn the bearing pressure to zero and then lower the probe and open the door. Remove the rotor from the probe by screwing the extractor tool partially into the endcap and pull it straight out. Be careful not to drop it! If drops, report to lab staff immediately, spinning a defected rotor will cause major probe damage.

Transcoupler

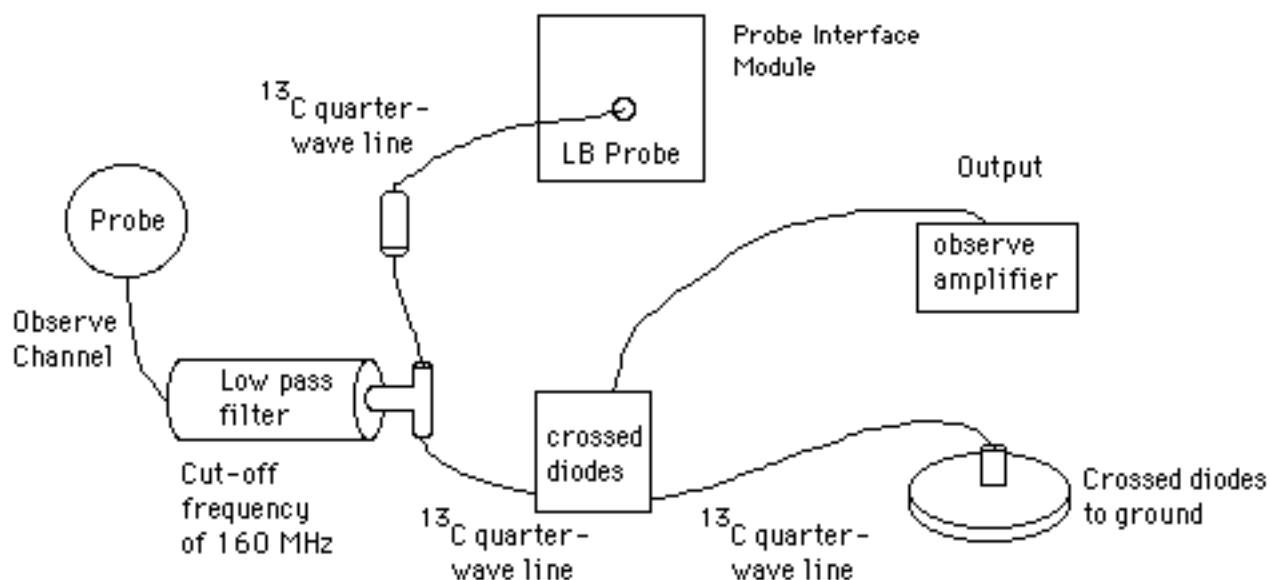
The function of a transcoupler is to isolate the transmitter from the receiver. The name " -transcoupler" comes from the particular arrangement of quarter-wave lines used in the solids experiment. It is important to understand the way the -transcoupler is configured, since changes need to be made when a different nucleus is to be observed. The basic circuit diagram is as follows:



where:



The quarter-wave lines have a band width of about $\pm 7\%$ of the rated frequency. A specific example of the -transcoupler in use for ^{13}C on the GN300 is given below:



When a change to a different nucleus is made, the quarter-wave lines in the -transcoupler must be changed. Be **VERY** careful when reconnecting the cables (see section on hardware precautions).

Probe tuning

Verify that all amplifiers are off and that you are not pulsing. Be sure that the correct capacitor insert is in place for the probe to be tunable to the desired nucleus. Type **N U** on the keyboard to select the observe nucleus, for example "C13" for carbon.

With the normal cable to the decoupler channel attached, connect the "TUNE" cable from the probe interface module to the observe channel of the probe. (It is not necessary to tune "through" the low pass filter). Identify the tune and match screw for observe (the two screw next to the observe cable connector) and the tune and match screw for the decouple (the two next to the decoupler cable connector). Type **X P** and increase the vertical display scale and the width until the dip is easily seen. Use the screw driver (made of teflon and copper) to change the tune and move the dip to the vertical line. Then change the match to maximize the dip. Recenter if necessary with the tuning. Repeat this procedure until no further improvement can be made. Then reconnect the normal observe cable.

Type **N U** to select the proton frequency "H1" and connect the "TUNE" cable from the probe interface module to the decoupler channel of the probe. Type **X P** and then tune and match as before using the decoupler wands. When finished, reconnect the normal decoupler cable and type **N U** to change back to observe nucleus.

If any large improvement has been made, the observe channel should be checked again. Likewise, if an additional improvement is then made on the observe channel, check the decoupler channel again, etc. Probe tuning may not be necessary when changing to another similar sample. Whenever in doubt, it is always best to check the tuning.

Power Equations

The following sections discuss amplifier selection and tuning. When the selected amps are properly tuned, both the forward and reflected power can be calculated with these equations.

Henry decouple and high band observe amp (40 dB insert):

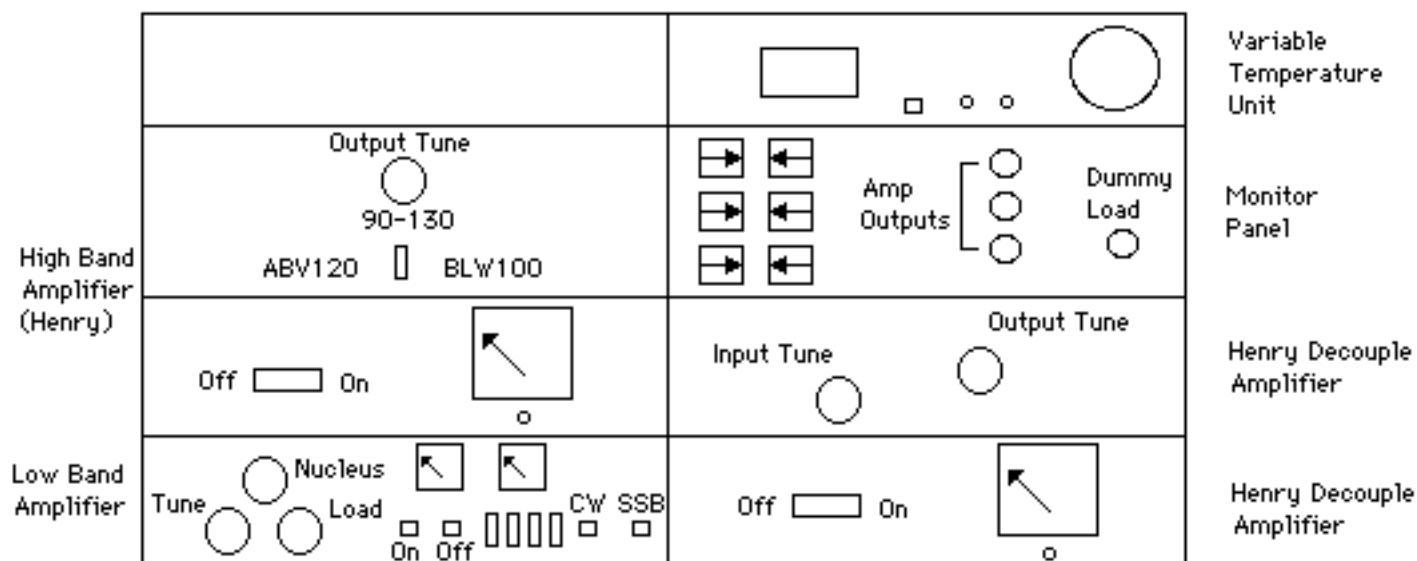
$$\text{Power} = 25 V_{PP}^2$$

Ehrhorn low band observe amp (43 dB insert):

$$\text{Power} = 49.88 V_{PP}^2$$

where V_{PP} = voltage peak to peak as read on the scope.

Observe amplifier selection



The Ehrhorn amplifier (referred to as low band amp in following instructions) will tune in the frequency range of 30-80 MHz. The Henry amplifier (high band amp) will tune in the range of 55-125 MHz. Select the low band amp for nuclei whose resonance frequency is between 30 and 80 MHz. The high band amp should be used for frequencies above 80 MHz. When using the low band amp, set the nucleus dial (selects frequency range) and the tune and load dials to values listed for the particular nucleus (see card on panel inside wooden cabinet door). The "HV" button should be in. When using the high band amp, select the frequency range with the dial.

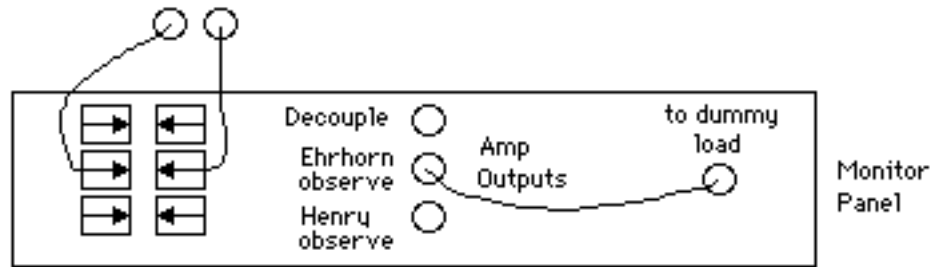
To operate the low band amp, press the red "ON" button and wait for the amplifier to warm up (the color of the meter light will change from red to green). Then press the "CW" button (now the color will change back to red). The "SSB" button can be used for higher power, but is usually not necessary. The amplifier can be put in the standby mode by pressing both the "CW" and "SSB" buttons lightly (the meter light will turn green). The power can then be shut off by pressing the green "OFF" button. When changing samples the amplifier should be off.

To operate the high band amp, press the red switch to "ON" or "OFF".

The following sections will instruct you in tuning the amplifiers and data acquisition, but you must remember whether the amplifiers should be on or off.

Tuning the observe amplifier

Verify that all amplifiers are off and that the instrument is not pulsing. Connect the output from the appropriate observe amplifier to the dummy load. Be sure that the input cable is connected to the appropriate observe amp. Also connect channels A and B of the scope to the monitor panel. The example given below shows connections when using the Erhorn (low band) amplifier.



Turn the scope on and turn off the room lights. Be sure that both channels of the scope are terminated with 50 ohms, then turn the power on for the observe amplifier. When using the low band amp, press the red "ON" button and wait several minutes for the amplifier to warm up (the color of the meter light will change from red to green at this point). Then press the "CW" button (now the color will change back to red). When the high band amp is used, merely press the red switch on and wait several minutes for the amplifier to come to temperature. Set all software parameters as follows:

C<control>**P** on, power level 2000 (if amplifier is not already well tuned, start at a much lower power level, tune, then increase gradually, tuning after each increment i.e., 300, 800, then 2000)
N U or **S F** (to set exact frequency for tuning the amplifier)
E X = 1PULS
P 2 = 10 μ sec
D 5 = 100 msec
C B = 1K
S W = +/- 10,000
A T = (check to be sure this is 25.6 msec)
N A = -1
Z G

Tune for a maximum display on channel A of the scope (the low band amp is tuned by both the "tune" and "load" dials, while the high band amp is tuned with only the "output tune" dial). Channel B will show the voltage proportional to the reflected power. There should be a factor of approximately ten difference in V_{for} and V_{ref} , i.e., $V_f/V_r > 10$. Type <control> **Q** to stop pulsing (verify by console lights that pulsing has stopped). Before disconnecting the cable, turn off the observe amplifier. The low band amp can just be put in the standby mode by pressing both the "CW" and "SSB" buttons lightly (the meter light turns green). Turn off the scope, and turn on the room lights if you are not going to tune the decouple amplifier. Then reconnect the appropriate observe amp output to the observe cable.

Tuning the high band Henry amplifier

First, you need change the input cable from the back of Erhorn to the input in the back of high band Henry amp. At the front panel, connect the high frequency observe to the dummy load. The rest tuning procedure is the same as the tuning of the Erhorn.

Tuning the decouple amplifier

This amplifier needs only to be checked about once a week. Verify that all amplifiers are off, and that the instrument is not pulsing. Connect the output from the Henry decouple amplifier

to the dummy load as in the previous section. Connect channels A and B of the scope to the appropriate places on the monitor panel. Turn on the scope and the amplifier (press the red switch on). Set the software parameters as follows:

F2 = (exact frequency for decoupling)
EX = 1PDNA
P2 = 1 μ sec
D5 = 100 msec
L1 = (set this decoupler power level to a value where you expect to see maximum cross polarization)
CB = 8 (not 8K)!!!
SW = +/- 20,000
AT = (check to be sure this is 100 μ sec.)
NA = -1
ZG

Tune for a maximum display on channel A of the scope with the "input tune" and "output tune" dials. Channel B will show the reflected power. Stop pulsing (verify). Turn off the decouple amplifier. Turn off the scope and turn on the room light. Reconnect the decouple amp output to the decouple cable.

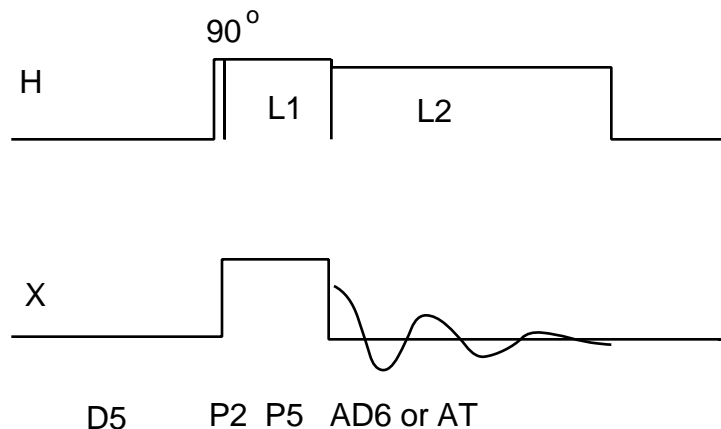
Data acquisition and processing with cross polarization

Most solid state NMR experiments of organic compounds are carried out using the cross polarization method because of the abundance of proton. In cross polarization experiment, the proton magnetization is transferred to the observe nucleus and thus the signal is enhanced. To select the cross polarization experiment (with bilevel decoupling), type **EX = CPBL**. The pulse sequence for this basic experiment follows:

```
CPBL
CROSS POLARIZATION
W/BILEVEL DECOUPLING

#      1:      D5
#      2:      D2, D, C, L1
#      3:      P5, D, C, L1, G
#                   XMTR      PHASE: A
#                   DEC        PHASE: B
#      4:      A, D, C, L2,G
#                   DEC        PHASE: B
#      5:      D6, D, C, L2 JUMP TO # 1
#                   DEC        PHASE: B

PHASE      A=2*S      MODULO: 4
PHASE      B=2*S+1    MODULO: 4
```



D5 is the typical "post-acquisition" delay (actually written first in most pulse sequences). For a CP experiment, the maximum sensitivity in the least time is seen when the delay is set equal to $1.25 T_1$ of protons in the sample. **D2** is the proton 90° pulse (enter in μsec). **P5** is the length of time the observe transmitter is on (typically 2-10 msec), and this is the cross polarization time. **D6 = AT** which is the acquisition time. Keep in mind that all values entered must also be within set limits (see section on precautions). **L1** is decoupler power level for the cross polarization and **L2** is the decoupling power level for the acquisition (typically 75-78 dB). A larger dB value indicates more decoupling power.

The preceding steps only select the pulse sequence and set parameters defined in the sequence. To turn the CPMAS module on (i.e., transmit using the high-power solids amplifiers), type **C<control>P**, answer **Y** or **N** to turn the module on, and enter a power level of **2000** (actual power is proportional to this number). To turn on the decoupler transmitter, type **DC**, answer **Y** or **N** to turn it on, select an output level of \emptyset dB with heterodecoupling and a modulation mode of 4(CW) (**NEVER type DN**). Use L1 or L2 to change decoupler power.

Before acquiring data, check to be sure that the proper amplifiers are set and turned on. The following parameters should also be set to the indicated values for a CP experiment:

- NA - number of scans**
- CB - be sure to use the smallest possible data size**
- F2 - set to frequency of protons in sample**
- SF - set to frequency of observe nucleus**
- QP - on = 1**
- AB - on**
- IF - Bessel, 3 dB attenuation**
- LT - on for all nuclei except those which are close in frequency to the lock (i.e., ^{17}O)**
- TC - off**

Data processing is very similar to a normal solutions experiment. The **GM** command can be used instead of **EM** to apodize the FID by Gaussian multiplication. The line broadening value is still defined with the **LB** command. If zero-filling is desired, the **ZF** command should be used immediately prior to transformation (i.e., **ZG,SA,BC,GM,ZF,FT...**). If data has been acquired with too short of a preacquisition delay (example: **DE<20 μsec**), the data block can be shifted to the left by one point with the **LS** command. The **FU** command can be used to adjust the first few data points in the FID if pulse breakthrough or receiver gating is a big problem. Only experienced users should attempt to use this command and it is accurate only to the degree that the user can

extrapolate the undistorted data backwards to the beginning of the FID. Refer to the software manual or see lab staff for details.

Many of the values for parameters used will not be printed in the Zeta list. The observe power level used (i.e., **C** <control> **P** level of **2000**) is plotted under OBS LO PWR on the list. Values for **QP**, **AB**, **IF**, **GN**, **TC**, and **LT** are not plotted. Most of the data processing information is not included (examples: **ZF**, **LS**, **FU** and any baseline correction routines like **BF** and **IC**). The shim values are not saved with the data, nor are they plotted in the Zeta list. No indication of which amplifier was used will be listed.

There are some other problems that the user should be aware of. In some instances, the number of acquisitions will not be included in a Zeta list. This problem can be avoided by always using **GS** to obtain data. By recalling the FID immediately after acquisition completion with **GA**, the correct **NA** count will also be recalled. This value can be retrieved from saved data with the <control> **D** command only. The values for **NA** and **CR** do not get reloaded when a data file is recalled from the disk. If any zero-filling was done, the initial data size should be noted on the spectrum, since the Zeta list will show only the final data size before transformation. If only the transformed data was saved, the initial data size cannot be calculated from stored parameters.

Optimizing parameters for cross polarization

First, MAKE SURE that the parameters for the cross polarization is corrected as described in the last section.

1. Shim values: Load in the appropriate shims from the shim library. The linewidth of solid spectra is much broader than solution spectra. Thus, the experiment is performed without using the lock and usually fine tuning of the shims is not necessary.
2. Hartmann-Hahn match: The cross polarization only occurs at so-called Hartmann-Hahn match between the observe power and the decoupler power. It is easiest to keep the observe transmitter power level (i.e., **C**<control>**P** level = **2000**) constant and vary the **L1**. For most standard sample, the FID can be seen in single scan. Start with **D2** = **5** μ S, **P5**=**3mS** find the Hartmann-Hahn match to maximize the FID by change **L1**. (from **L1**=**73**, to **L1**=**80** depending on the observe nucleus)
3. Proton 90° pulse lengths: Find the proton 90° pulse lengths (**D2**) for **L1** levels that meets the Hartmann-Hahn match. Using **D2**=**2** μ S and **NA**=**4**, take a spectrum and phase it. Then vary D2 at 3 μ S a step and process the data using the same phase parameters. Find the D2 values for the 180° pulse node and the 360° pulse node. Divide the 360° node D2 by 4 and add another 0.5 μ S (due to the amplifier rising time) to obtain the 90° degree pulselength for the proton.
4. Cross polarization time: Typical 1 mS **P5** should be sufficient to the cross polarization. To detect observing nucleus that is remote to protons, **P5** should be longer but should not exceed 10 mS.
5. L2 decouple level: Start with L2 3 dB smaller than L1, the Hartmann-Hahn match and use a small block size (**AT**≤25.6 msec), increase the value for **L2** by 1 or 2 dB. If the linewidth is not significantly narrowed, it means the previous used decoupler power is sufficient. If it does narrow the linewidth, increase the L2 slowly until the proton is fully decoupled. If

"splats" are seen in the FID, stop the acquisition immediately by **Cntrl Q Q Q** and lower the decoupling power.

Spinning Angle Adjustment

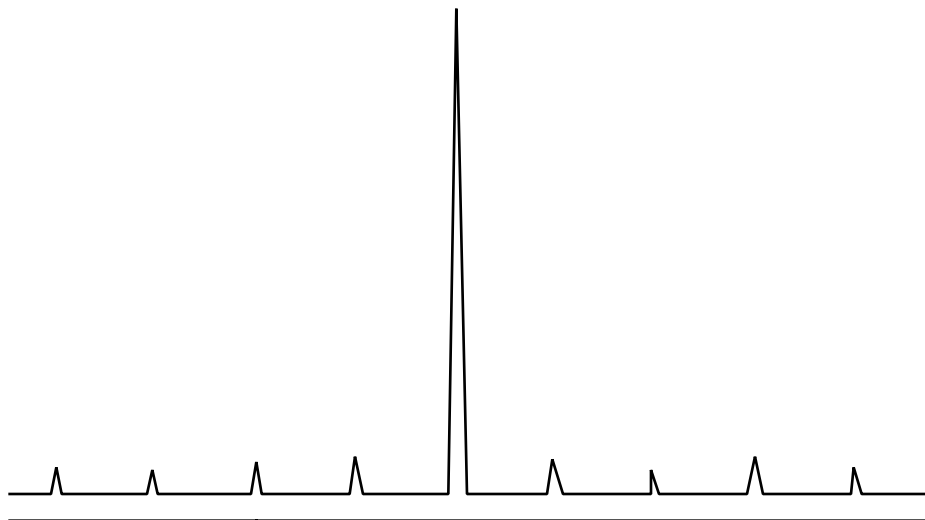
The spinning angle has to be checked once a week to make sure it is at the magic angle and whether the adjustment is needed. $K^{79}\text{Br}$ is used to make the adjustment.

The following example of angle adjustment and spin rate measurement will work well for ^{13}C samples: Put the Carbon standard sample (HexaMethyl Benzene HMB which is mixed with KBr) It is not necessary to retune the probe to observe ^{79}Br (the signal will be so strong that the 270 KHz difference in frequencies will not matter) if the spectrometer is already setup to observe ^{13}C . Enter the following parameters:

```

NU = BR79
EX = 1PULS
P2 = 2 µsec
D5 = 100 msec
CB = 4K
SW = +/- 50,000
NA = 32
GN = 100
CR = 1.8
RG = 15

```



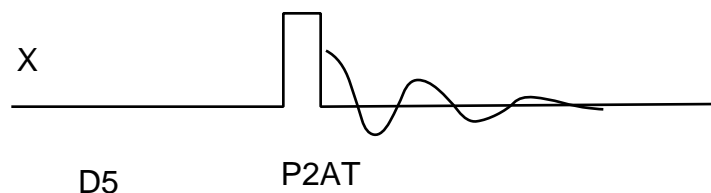
Acquire and process the data and you will notice a large first order phase correction, due to the large CR value. If the spinning angle is near the magic angle, numerous spinning sideband should be seen in the spectrum. The procedure to adjust to the magic angle is maximizing the sideband intensity. To do this interactively, **LI=ZGBCEMFTPSWT** with LB=10, WT=5 sec and NA=16, type **AU=-1** to start automatic acquiring and processing with about every 5 seconds. During the 5 second period, turn the angle adjust wind at the bottom of the probe while watching the change of the spinning sideband height. After the height of the spinning sideband are maximized, the spinning angle should be at the magic angle and type **Cntrl H** to stop the acquisition.

Other pulse sequences for solids

Only the CPBL experiment has been described so far. This is not always the best choice for a solids experiment. For example, cross polarization would not work if there is no proton in the sample. This section describes other pulse sequences available on the instrument. The CPBL sequence is also included again, here, for comparison with other pulse sequences. This handout contains only superficial descriptions, and the literature should be consulted for more details. Most pulse sequences will already be in the software library but some may need to be added with the **N X** command depending upon the current version of software in use. Be sure you are familiar with the operation of this command before attempting to use it on your own.

One pulse (1PULS):

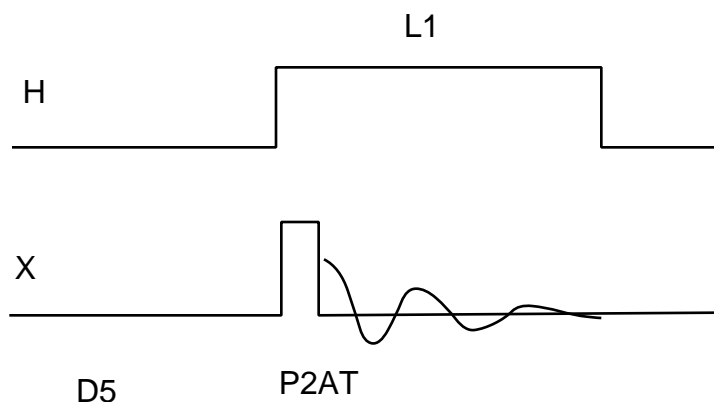
Experiments on samples where few protons (if any) are present can be done with only magic angle spinning and the simple 1PULS sequence.



P2 is the pulse length and usually a 60° degree pulse length is a good choice. D5 is the acquisition delay and should be about $1.5T_1$ long. For most of the quadrupolar nucleus, $I > 1/2$ e.g. ^{17}O , ^{27}Al etc, the spin lattice relaxation is very short and D5 of 300-500mS should be sufficient. However, for nucleus with $I=1/2$, like ^{13}C , ^{29}Si , T_1 can be very long (from few sec to minutes) and long D5 should be used.

One pulse, decoupler on during acquisition (1PDNA):

When proton decoupling is needed, the 1PDNA experiment can be used.



In this experiment, because the decoupler is turned on for the entire acquisition period, the choice of the **AT**, **D5** and **L1** parameters needs to be checked. **AT** is the time that decoupler is turned on and it should be less than 50 mS. The ratio of **AT** and **D5** is the duty cycle and it should not exceed 10%. **L1** is the decoupling power and it should be less than the maximum power it allows which varies with the observed nucleus.

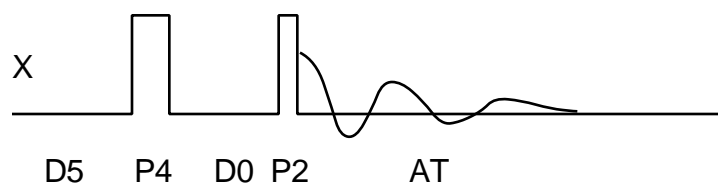
Cross polarization with bilevel decoupling (CPBL):

This sequence is used for low γ nuclei with long relaxation times. When the Hartmann-Hahn condition is satisfied, a signal enhancement equal to γ_H/γ_X could theoretically be obtained (in practice this ratio is smaller, but still significant). Also, the recycle time depends only on the relaxation of the proton spins. The pulse sequence that follows uses one power level for the proton 90° pulse and CP time, and a higher power level for decoupling during the acquisition.

Refer to the CP sections in this handout for instructions on optimizing parameters, data acquisition, and processing.

Regular T_1 inversion (T1IRX):

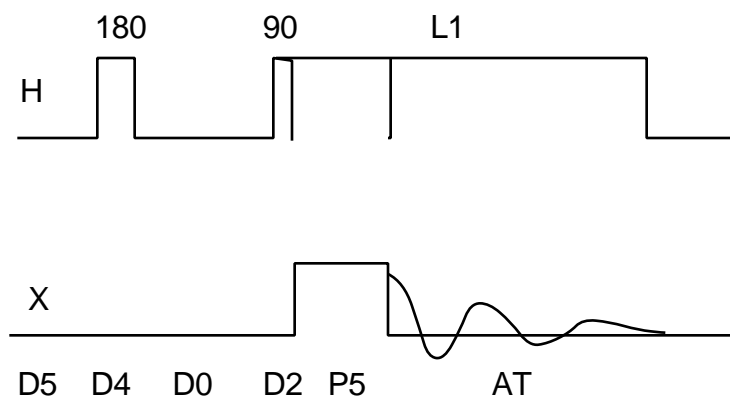
This pulse sequence should be used for solids in place of T1IR.



It applies a "regular" 180° pulse instead of the composite pulse included in T1IR. The sequence is useful for T_1 calculations on samples where cross polarization is not necessary.

Proton T_1 via cross polarization (PT1CP):

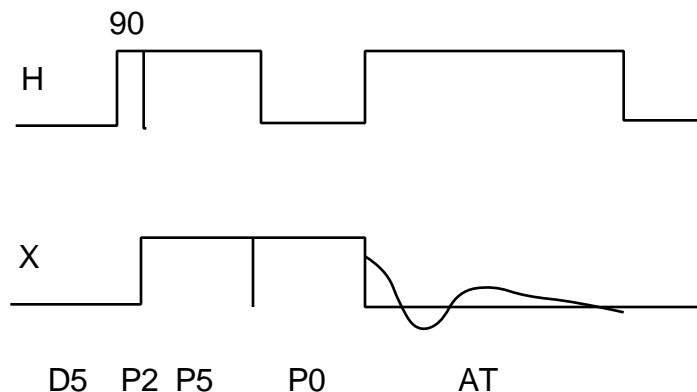
The proton T_1 can be determined using cross polarization (with bilevel decoupling) when a proton 180° pulse and a variable delay () are inserted before the proton 90° pulse. The value for the delay can be varied and T_1 can be determined in a manner analogous to that used for T1IR.



In the following sequence, **D0** is the variable delay.

Observe T_1 (XT1RHO):

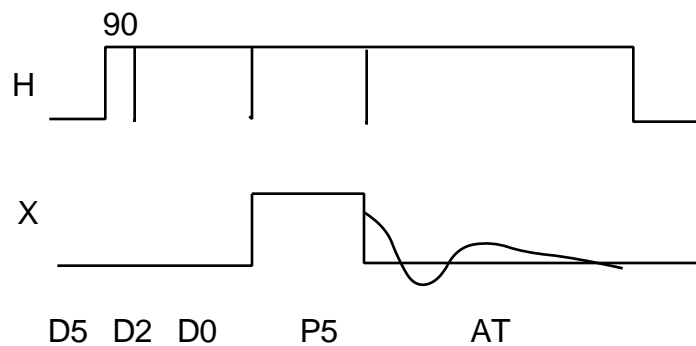
By inserting a variable pulse length of duration after the CP time and before the acquisition, T_1 (spin-lattice relaxation time in rotating frame) can be measured for the observe nucleus. This variable pulse length is entered as **P0**.



Because the observe amplifier is turned on during the **P0** period, the maximum **P0** should be less than 50 mS or you need to turn down the observe amplifier power level using the **C^P** command.

Proton T_1 via observe nucleus (PT1RHO):

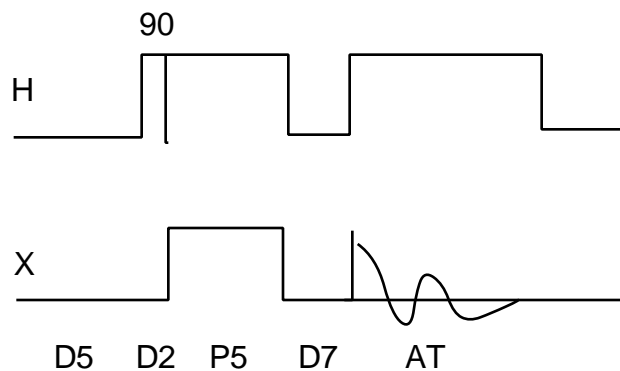
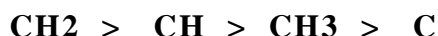
By inserting a variable delay (during which time the decoupler is on) after the proton 90-degree pulse and before the CP time, T_1 of the protons can be measured indirectly by observing the



change in intensity of the observe signal. This variable delay is entered as **D0**. Because the decoupler is turned on during **D0**, the length of **D0** should be included to calculate the duration and the duty cycle of the decoupler.

Interrupted decoupling or dipolar dephasing (ID):

This pulse sequence will give a spectrum where the signals from carbons (or other nuclei) with attached protons are suppressed to certain degree depending on the the number of proton it attaches.. A brief delay (**D7**) without proton decoupling is inserted between the cross polarization time and the acquisition time with full proton decoupling. For carbon, the delay should be set between 2-150 μ sec. During this delay time the carbon spins will dephase under the influence of the proton dipolar field, but those with attached protons will dephase very quickly compared to those without. The relation of how fast the signal decays and the number of attached proton is the following:



This experiment works best for rigid systems. Imperfections can be reduced by adjusting the delay interval.

Variable temperature operation

For the variable temperature operation using the pencil MAS probe, users are referred to handout #UWB374 which has a temperature range -150 °C to 250 °C.

For the variable temperature operation using the wideline deuterium probe, users are referred to handout # UWB373.

Shut down procedure

Turn all solids amplifiers off. Be sure you are not pulsing. Lower the probe and remove your sample. Close the sample door and place the probe back in the magnet. Shut down the rest of the system as instructed in the solutions manual.