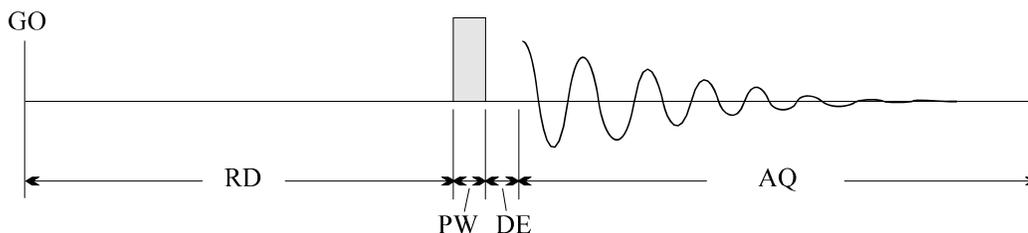


## 2. Bruker Acquisition Basics

by *cg fry*: created 2/17/94 – updated 12-Sep-00



BRUK-GO.CDR

### I. Description

[See the Primers chapter for more specific information on certain areas]

Acquisition on Bruker AM and AC spectrometers is based on the command **GO**. **GS** (go-setup), **ZG** (zero-go), and **CO** (continue) all use the **GO** command; **ZG** zeroes memory and coadds acquisitions until **NS** and **NE** are fulfilled, whereas **CO** coadds new acquisitions to the current memory. **GS** displays only the most recent acquisition.

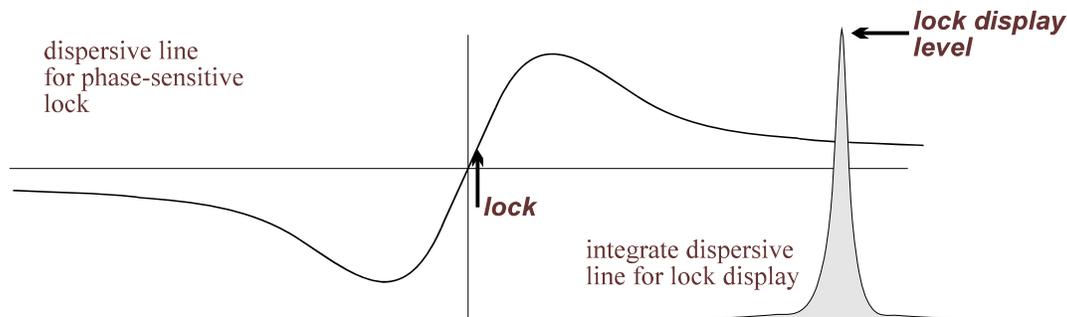
Four events are initiated when **GO** is issued, as shown in the figure above:

- i) The spectrometer waits a time RD seconds; relaxation delay.
- ii) A strong transverse rf pulse is applied with a duration PW  $\mu$ s.
- iii) A delay of duration DE  $\mu$ s occurs to allow for probe ringdown.
- iv) Acquisition begins, and lasts a time AQ seconds.

### II. Deuterium Lock

Locking on a deuterated solvent involves adjusting four spectrometer variables:

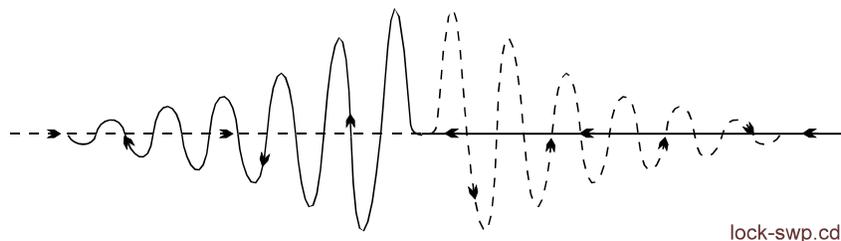
- i) Change **ZO shim (FIELD)** to find the lock signal. Deuterons track closely with protons, so  $\text{CDCl}_3$  will be 5.2 ppm downfield of  $(\text{CD}_3)_2\text{CO}$ . It should be clear (if you don't know, please ask!) that changing the magnetic field strength by 5.2 ppm will require a change in the spectrometer frequency by the same amount to keep a 10 ppm sweep width running from TMS at  $\delta=0$  ppm to  $\delta=10$  ppm.
- ii) The second adjustment involves the **lock phase**. The lock uses a *phase-sensitive* loop to keep the frequency of the spectrometer stable:



lock-phs.cdr

The lock in sweep mode on Bruker spectrometers should always have a positive initial slope.

**Correct phase on lock sweep display  
(use Dual Display on SCM unit)**



Changes in solvent, temperature, and even in adjustments from poor to good shims can change the phase. *Good lock phase is critical to obtaining a stable, good lock, and thus a good shim and lineshape!* So you should adjust the lock phase after any such changes. Initially use the sweep display, but later carefully adjust the lock phase to maximize the lock amplitude.

- iii) The other two adjustments are the **lock gain** (lock amp on WP's) and **lock power**. Although the apparent effect of both controls is similar, to increase/decrease the lock intensity, the actual changes are fundamentally different. The **lock gain** is the lock receiver gain, changing nothing involved with the sample magnetization. The **lock gain** can therefore be changed at will with little danger.
- iv) The **lock power**, on the other hand, must be carefully adjusted to achieve the best lock performance. This control changes the radiofrequency (rf) power to the sample. Too little power will give a weak response, and a noisy lock. Too much power will cause saturation, which *must* be avoided. Proper settings can be found for any solvent using the following technique:

Increase/decrease the **lock power**, and watch for a "bounce" in the signal: the signal will go up/down in response to the change in power, but if it then rebounds (perhaps just slightly) down/up as in a bounce, the power is too high. Decrease the power until the bounce cannot be observed, and decrease the power another 20% to be safe.

Suggested settings for the **lock power** for the most common solvents are posted next to each AC spectrometer in the facility.

### III. Shimming

Once lock is achieved, the magnetic field inhomogeneity must be shimmed out. For experimental planning, one must keep in mind differences in solvents. Acetone-d<sub>6</sub> has a very narrow natural <sup>2</sup>H linewidth, and therefore gives good performance for shimming. DMSO-d<sub>6</sub> and pyridine-d<sub>7</sub> both have much broader natural <sup>2</sup>H linewidths, and are therefore relatively poor solvents for lock shimming. D<sub>2</sub>O can give very broad <sup>2</sup>H lines due to exchange. Of course, the last three solvents might be the only choices for solvation and cost reasons, but shimming on the FID might then be necessary if high-resolution <sup>1</sup>H spectra are needed.

A number of good discussions about shimming strategies are available. Since every change of sample requires shimming, every spectroscopist should take advantage of strategies to lessen the time spent shimming. Especially useful is G. A. Pearson, "Shimming an NMR Magnet," Chem. Dept., Univ. IA, Iowa City, IA 52252. Many practical examples are given that show how to visually determine which shim needs adjusting, and in which direction. These quick determinations can save much time at the spectrometer and lead to improved spectra. More detailed discussions of shimming can be found in Derome, p. 42-50, and in G. N. Chmurny and D. I. Hoult, "The Ancient and Honorable Art of Shimming," Concepts Magn. Reson. **2**, 131-149 (1990).

#### IV. Acquisition Parameters

The most important acquisition parameters are briefly described in this section. The mnemonics are specific to Bruker spectrometers:

**PW** – pulse width of **GQ** command (see Section I)

**RD** – relaxation delay

**DE** – probe/filter ringdown delay

**SW** – sweep width in Hz

**O1** – sets center of spectrum by changing absolute frequency

**DW** – dwell time in  $\mu\text{s}$ ; time per digitized point; set by  $\text{SW} \rightarrow \text{DW} = 1/2\text{SW}$

**TD** – data acquisition size; number of points digitized;  $\text{TD}/2 = \# \text{ complex data pairs}$

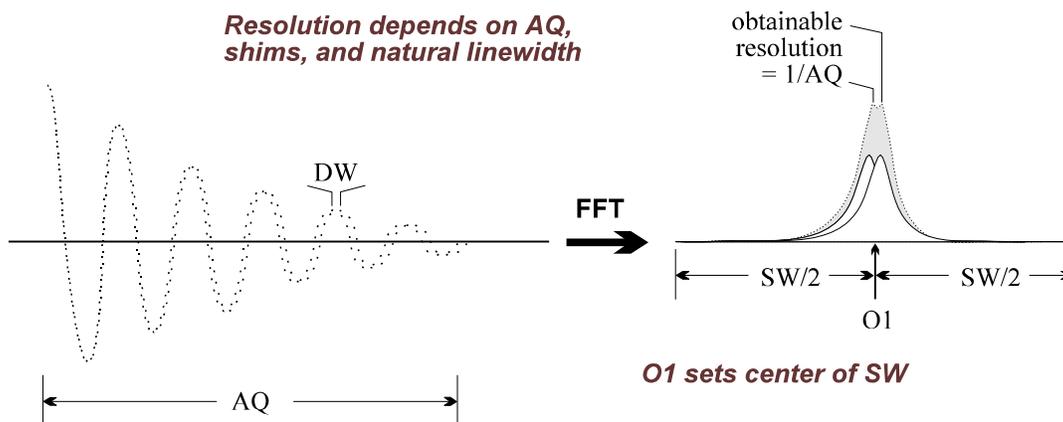
**SI** – Fourier transform size; zero fills if  $\text{TD} < \text{SI}$ ; only half SI used for real part, so

$$\# \text{ points in spectrum} = \text{SI}/2 \quad \text{digital resolution} = \frac{\text{sweep width}}{\# \text{ points in spectrum}} = \frac{\text{SW}}{\text{SI}/2} = \frac{\text{TD}}{\text{AQ} \cdot \text{SI}}$$

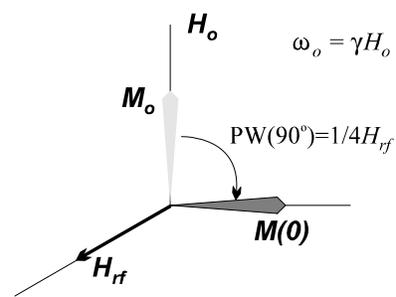
**AQ** – acquisition time; related to *obtainable resolution* (i.e., best resolution possible) unless sophisticated analytical procedures, such as linear prediction, are used. The digital resolution can be improved by zero-filling, but not the actual obtainable resolution (appropriate Gaussian multiplication perhaps can take advantage of one zero-fill, but further zero-fills will not help).

$$\text{obtainable resolution} = \frac{1}{\text{AQ}}$$

$$\text{AQ} = \text{DW} \cdot \text{TD} = \frac{\text{TD}}{2\text{SW}}$$



sw-aq-dw.cdr



pw90.cdr

### 3. Introduction to AC+ Spectrometers

by cg fry: created 1/10/94 – updated 03.Sept.2008

#### I. Summary of 1D Acquisitions on AC Spectrometers

The following list progresses similar to taking data on any sample. These are not inclusive for all occasions that occur in the laboratory, and cannot therefore replace an understanding of the basics of NMR. Many limitations will be self-imposed by the student that utilizes this list too strongly. That said, this section should provide a useful tool while learning to use Bruker AC spectrometers.

- a) Unless you are immediately following another user, the spectrometer should be locked on a CDCl<sub>3</sub> standard. *Remove the CDCl<sub>3</sub> standard:*
  - ☞ Turn up the display intensity using knobs at the lower right corner of the display.
  - ☞ Use **CNTL-L** and **CNTL-D**, if needed, until the display has the proper information on screen.
  - ☞ Unlock and stop the spinner: *press* **LOCK** and **SPIN ON/OFF**; the light goes off for both buttons (this step is not really necessary; the lock system knows to do both automatically).
  - ☞ Eject the CDCl<sub>3</sub> standard sample: *press* **2<sup>ND</sup> LIFT**.
- b) *Insert your sample:*
  - ☞ Put the sample/tube into a sample spinner; lower it to the bottom of the depth gage, or if the solvent height is less than ~0.5ml (always use ≥ 0.35ml!!), center the solvent column on the rf center mark drawn in red on the depth gauge.
  - ☞ Clean the bottom of the tube using the ethanol and chemwipes: **do not get ethanol on the sample spinner (it will ruin it, at ~ \$75 each!)**.
  - ☞ Make sure the lift air is on: *if it is not, press* **2<sup>ND</sup> LIFT**.
  - ☞ Place the spinner+sample in the lift stream at the top of the magnet; make sure the tube is floating freely before letting go of it!
  - ☞ *Press* **LIFT OFF** and allow the sample to seat (you should hear a click as it seats).
  - ☞ *Press* **SPIN ON/OFF**; the sample should spin up, but after a ~5–20s delay.
- c) *Lock and shim the sample to a good line shape:*  
Follow section II below, and see the Primer on Shimming in Chap. 4.
- d) *Read in the proper jobfile, set RG, and acquire:*  
Follow section IV below: e.g., **RJ CDCL3.1DJ ↵ II ↵ ZG ↵ ^H RG ↵ change ↵ ZG ↵**
- e) *Save and Transfer data to the PC server, work up and plot:*  
Save data with the **WR filename.xxx ↵** command. Follow section V below (e.g., **^X TOPC filename.xxx ^X**). After step f), use the NUTS Cheat Sheet to work-up and plot the data.
- f) *Remove your sample, and lock on the CDCl<sub>3</sub> standard:*  
Follow section VI below; turn off the decoupler if needed using the **DO ↵** command.
- g) *Backup data:*  
Use Windows Explorer to copy data to a memory stick, or transfer it to your PC/Mac using the ftp server: <ftp://apollo.chem.wisc.edu/ac300>.

The primary functions for obtaining routine  $^1H$  spectra are contained in the Shim Control Module (SCM) and with preset job files that setup acquisition.

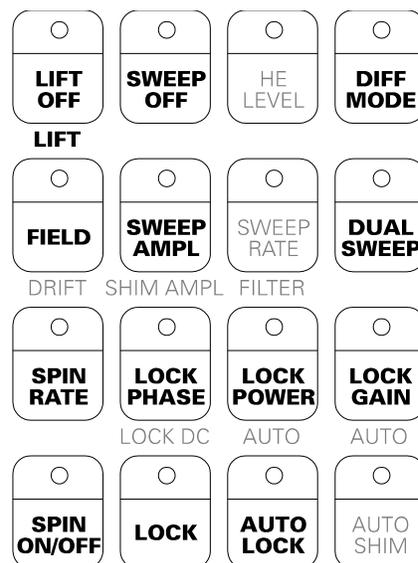
## II. Locking and Shimming with the SCM

- *Users should not use any controls except those shown darkened on the right!*

- <CNTL>-L toggles through various lock/data displays
- The FINE button should (nearly) always be on (lighted).
- Samples are ejected by pressing the orange 2ND button on the lower right, and the LIFT button. Insert the sample by pressing the LIFT OFF button.
- Reading in the facility shim file is recommended:

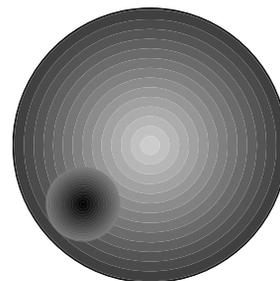
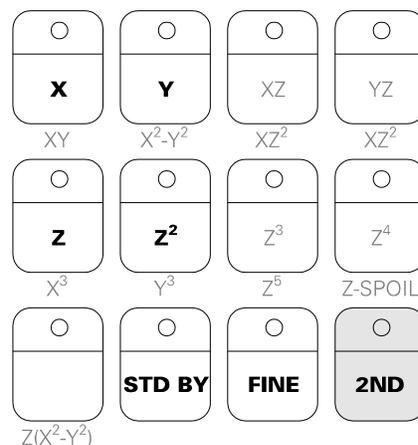
RSH *specname*.SHIM,↓ e.g., RSH ATHENA.SHIM,↓

- For samples with a change in solvent:
  - a) turn LOCK off
  - b) change LOCK POWER to appropriate setting (see table next page)
  - c) SWEEP AMPL does not work on Athena. Set FIELD and LOCK PHASE as listed on the console
  - d) adjust the LOCK PHASE if necessary; DUAL SWEEP can help here
  - e) press AUTO LOCK and wait for LOCK GAIN to stop blinking
  - f) press LOCK; check LOCK POWER
  - g) adjust LOCK GAIN to get signal in top fifth of screen
  - h) adjust LOCK PHASE just as you would any shim (no interactions occur; but LOCK PHASE should be readjusted whenever  $Z^2$  is moved substantially)
  - i) shim with Z and  $Z^2$  shims; turn off spinning and adjust X and Y shims if spinning sidebands are large (other shims should not need to be adjusted)
- Shim settings can be reset to PREVIOUS SET value by repressing the current shim key. E.g., start in Z at -140, and realize after going to -80 that you're going in the wrong direction. Pressing the Z key again will reset the Z shim back to -140.



PREVIOUS SET

ACTUAL



### III. Job Files and Lock Power

Deuterated Solvent	Job File	Lock Power		$^1H$ $\delta$ (ppm)*	$^{13}C$ $\delta$ (ppm)*
		unlocked	locked		
acetone	ACETONE.1DJ	25	15	2.2	30.2 205.1
acetonitrile	CD3CN.1DJ	20.0	10.0	2.0	0.3 117.2
methanol	CD3OD.1DJ	20.0	10.0	3.5	49.3
deuterated water	D2O.1DJ	25.0	15.0	4.8	
dichloromethane	CD2CL2.1DJ			5.3	54.2
dimethylsulfoxide	DMSO.1DJ	35.0	35.0	2.6	39.5
chloroform	CDCL3.1DJ	35.0	25.0	7.3	77.7
benzene	C6D6.1DJ	22.0	12.0	7.4	128.7

\*from Bruker Almanac 1994, p. 119-120.

Other solvents can be run using one of the job files listed above. The key to the selection is getting the LOCK POWER set correctly. For toluene-d<sub>8</sub>, pyridine-d<sub>5</sub> and acetic acid-d<sub>4</sub> use C6D6.1DJ. Ask staff if you need to use another solvent.

### IV. Data Collection

- A. Read in a job file for your solvent; use a job name in the table above: e.g., for CDCl<sub>3</sub>, enter **RJ CDCL3.1DJ** . Type **II** to initialize the interface (i.e., set the hardware up correctly).
- B. Set the receiver gain, **RG**, by watching carefully the size of the 1st FID following a **ZG**.
  - i. Type **ZG**. If the 1st FID is larger than  $\pm 1$  division on the AC/AM display, stop the acquisition using **<CNTL>H** . Increase **RG**, and redo the **ZG**, again watching carefully the size of the 1st FID.
  - ii. If the 1st FID is much smaller than  $\pm 1$  division, increase **RG**.
  - iii. Automatic gain adjustment can also be used with **RGA**. This method is not recommended for various reasons too detailed to go into at this point (ask if you really want to know).
- C. Use EP mode to adjust the sweep width and offset using **<CNTL>O** (see next page)
- D. Set the number of scans, **NS**, to a multiple of 8. Collect data by using zero-go, **ZG**.
- E. Write the data to the spectrometer hard disk using the write command: **WR filename.xxx**.
- F. Transfer the data to the network server via NMRLink (see next section).
- G. Log usage on the Chronos PC Excel spreadsheet.

### V. File Transfer: NMRLINK

(usually FID's are saved; RE FID1 will read back the FID after an FT; spectrum is SPC1)

- **To send a file to the network:**
  - i) take data as normal (**ZG**, then **WR filename.xxx**)
  - ii) type **<CNTL>X** to go into the "2nd session" (displayed only on the LCD display)
  - iii) enter **topc filename.xxx**
  - iv) type **<CNTL>X** to go back to the 1st session and DISNMR
- **To retrieve a file from the network back to the spectrometer**
  - same as sending, but use **frompc filename.xxx** instead of the topc command

## VI. Departure

- A. Following Section II, insert the lock standard and relock.
- B. Turn the screen intensity down.
- C. Clean up the console area.
- D. Log time spent on the spectrometer on the Excel spreadsheet on Chronos; note any problems in the spreadsheet such as difficult spinning, ejecting sample, spinning sidebands etc. Any problems such as a computer crash, disk full, or sample breaking, find a TA or NMR staff immediately.

## VII. Adjusting for SW and O1

The following notes summarize commands to allow quick manual modifications of **SW** and **O1** to get appropriate spectra for special cases:

- Suppose you want to look at a portion of the normal spectrum with high digital resolution (e.g., the aromatic region of ODCB). Take the normal spectrum, and then follow the steps shown below to narrow the sweep width to obtain just that region.
- Suppose instead that you have an acid group or heteroatom that causes chemical shifts outside the normal 0-10 ppm region. In these cases, you would see “folded” peaks in spectra obtained with SW=10ppm (2500 Hz on Phoenix); see the spectra on the next page. Folded peaks will almost always have unusual phasing that cannot be corrected with normal phase corrections in NUTS.

- A. For folded peaks, double or triple the sweep width, SW. On the AC-300, 6000 Hz = 20 ppm, so use SW=12000 up to 100000. Retake the spectrum using **ZG**.
- B. Perform an FT at the spectrometer, by entering **FT** .
- C. Go into EP mode by entering **EP<RET>** .
- D. Note in EP mode that the region of text has changed from the top of the screen to the bottom. EP mode is now ready to accept a variety of commands; use the following two (see also Primers Chapter):

**P** - phase adjust:

- i. expand using B-knob on a large peak on one side of spectrum
- ii. enter phase mode by entering **P** (do not press <RET>)
- iii. correct 0-order with C-knob
- iv. move to large peak furthest away and correct 1st-order with D-knob
- v. use **M** to save

A-knob moves spectrum horizontally

B-knob expands/contracts region on screen

C-knob performs 0-order phase correction (<CNTL>C reverses)

D-knob performs 1st-order phase correction (<CNTL>D reverses)

**M** will memorize phase adjustment and return to EP mode

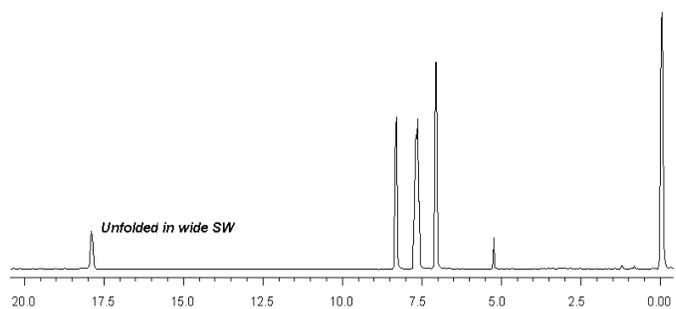
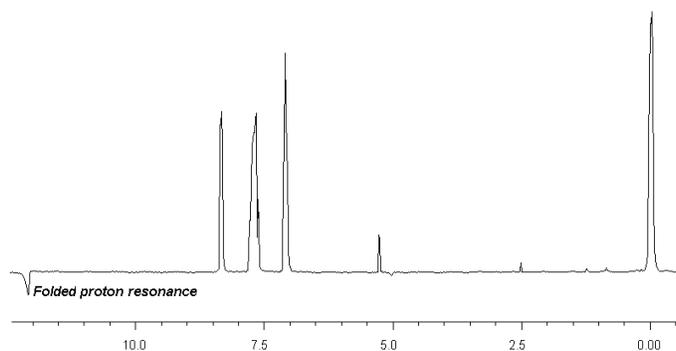
<RET> will ignore phase adjustments and return to EP mode

<CNTL>**O** - automatic SW and O1 redefinition

- i. expand using B-knob to region you want as full spectrum
- ii. move cursor to center of screen using C,D-knobs
- iii. press <CNTL>**O**. The computer will set new SW and O1 values.

- E. Press <RET> to exit EP mode, and enter **ZG** to take new spectra.

### VIII. Spectra showing folding and unfolding of deshielded proton.



### IX. Caveats

- A. Watch the lock power: if it's too high, saturation will occur, and poor line shapes will likely result (you can check this by watching that lock signal increases consistently, and doesn't "bounce", as the lock power is increased).
- B. The hard disks on the AC spectrometers fill multiple times per year. If you get a DISK FULL error message, see NMR Director or a TA to clear space. Always make backups immediately!