

## Shimming on the U400

Once you have logged onto the spectrometer *via* Reslog and inserted the sample (see handout UVU405, *U400 Sample Preparation, Positioning, and Insertion*),

### 1. Change Current Working Directory to Your fid Directory

**logon** <rtm>

enter your username at the prompt; change working directory to your fid directory

### 2. Retrieve a Standard Shim File for a Given Probe and Solvent

**rts('QUAD')** <rtm>

retrieve QUAD shimfile

**su** <rtm>

load shim values from shimfile to DACs

### 3. Select a Set of Standard Parameters for a Given Nucleus and Solvent

LC Main Menu

select main menu

LC 2:Setup

select setup menu

LC 1:H1,CDCl3 (for example)

select an appropriate set of standard parameters

**dg** <rtm>

display acquisition and processing parameters and make necessary changes.

### 4. Lock on the Sample

See handout UVU407 (*Locking on the U400*)

### 5. SHIM ON THE SAMPLE

LC SHIM

connect to interactive shim window

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#### Adjustment of parameters in the interactive shimming window:

Place the mouse pointer in a button (such as -1+) for the appropriate function (such as **Z1C**) and *left* click to *decrease* its value by the number of units selected, or *right* click to *increase* its value by the same amount.

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LC or RC **Z1C** -4+ button

adjust Z1C to maximize the lock level

LC or RC **Z2C** -4+ button

adjust Z2C to maximize the lock level.

LC or RC **Z1C** -1+ button

adjust Z1C to maximize the lock level

LC or RC **Z2C** -1+ button

adjust Z2C to maximize the lock level.

LC or RC **Z1** -16+ button

adjust Z1 to maximize the lock level

LC or RC **Z2** -64+ button

adjust Z2 to maximize the lock level.

LC or RC **Z1** -4+ button

adjust Z1 to maximize the lock level

- Repeat **Z1C** and **Z2C** shimming *interactively* (*i.e.*, if one is changed, the other should be readjusted) until maximum lock level is achieved. You may want to re-optimize the lock phase if large changes of these shim gradients have been made.
- Then, **Z1** and **Z2** are adjusted *interactively* until maximum lock level is reached (usually starting with -16+, then -4+).

c) Normally, **Z3** is not changed. If **Z3** is changed, **Z1** and **Z2** will need to be re-optimized.

NOTE: *No shimming on Z4 should be needed for routine experiments*, unless you are observing a sample with very different physical properties from those of the standard or a sample of unusually short length.

If Z4 shimming is required, change Z4 in one direction by 16 to 32 units and optimize Z1, Z2, and Z3. If the lock level increases, repeat Z4 shimming in the same direction (reduce the number of units being changed on Z4 as you are getting closer to its optimum) until maximum lock level is reached. If the lock level decreases, change Z4 in the opposite direction and optimize Z1, Z2, and Z3. The usual method of optimizing each shim gradient interactively based on the lock level does not apply to Z4 shimming, nor to shimming when the shims are very far from optimal.

In the case of Z4 shimming, it may be advantageous to use the FID area as the criterion instead of the lock level, since Z4 affects most strongly the beginning, or the larger end of the FID, which means that the FID area will be most sensitive to changes in Z4. See below for shimming on the FID.

When you finish shimming check that the lock level is between 40 - 90, then

LC Disconnect

disconnect from VNMR ACQUISITION window

**NOTE: You can insert the sample, lock and shim, then setup acquisition parameters, or insert the sample, setup acquisition parameters, then lock and shim. Both methods are correct, neither is better than the other; however, you must load a shim library before shimming or you will be wasting your time. You will have to decide which way you want to proceed with your own samples. However, pick a method and stick with it or you will forget to do something critical for your measurement.**

**Table 1. Locking Parameters and Standard Shim Files for the QUAD Probe** (11/12/93)

SOLVENT	CDCL <sub>3</sub>	C <sub>6</sub> D <sub>6</sub>	D <sub>2</sub> O	CD <sub>3</sub> OD	Ac-d <sub>6</sub>
Z0*	-23	-16	136	220	298
LOCK POWER	20 - 25	10 - 15	10	5 - 10	5 - 10
SHIM FILE	QUAD	QUAD	QUAD	QUAD	QUAD

\* Z0 will change with time.

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## Other Shimming Options

Three options are available for homogeneity adjustment on the U400: 1) manual emulation shimming, 2) interactive autoshim, and 3) background autoshim.

### 1. MANUAL EMULATION SHIMMING.

Manual emulation shimming is accessed with the *acqi* interactive VNMR ACQUISITION windows, which are not accessible when data acquisition is in progress. In this mode, homogeneity is adjusted manually while observing a lock level, real-time FID display, or real-time spectrum display on the screen (in the VNMR ACQUISITION window, not the main graphics window).

The procedure described on the previous page is for *MANUAL SHIMMING ON THE LOCK LEVEL*, and is the principle method of shimming for routine experiments. However, there will be times when another method, or combination of methods, of shimming will be preferred or even required.

**A. REAL-TIME SHIMMING ON THE FID.** This type of shimming is recommended only after manual shimming on the lock, either when the shims are close to optimum for fine tuning of the shims to improve the line shape, or when the shims are so far from optimum that the lock level is insensitive to changes of the shim gradients. It may also be used for Z4 shimming, preferably in conjunction with manual shimming on the lock.

After manual shimming on the lock,

LC Disconnect (in VNMR ACQUISITION window)

disconnect from VNMR ACQUISITION window

Select an appropriate set of standard parameters for a given nucleus and solvent. Usually, the 1H FID is used for fid-shimming because it is much stronger than that of the 13C. In fact, any nucleus that can give an FID with reasonable S/N in one scan will do.

Optimize the spectral window with the **movesw** command to include the signals of interest plus  $\approx 15\%$  of empty baseline.

The acquisition time **at** should be set so that the FID decays to zero at the end of the acquisition (typically 1 or 2 sec). To find out the minimum **at**, take a scan with the default **at** (4 sec) and type **df dscale('s') <rt>**, which will display the fid with a scale in seconds.

**at=\_\_\_\_\_ (minimum determined above)**

**d1=1 <rt>**

**ga <rt>**

**gf <rt>**

take a scan with current parameters

prepares current parameters for FID/spectrum display in *acqi*.

LC Connect

LC SHIM

LC FID

**The following is displayed:**

<b>16/16 bits</b>	<b>FID</b>	<b>Spectrum</b>
up	<b>Single/Summed</b>	<b>Grid on/off</b>
down	<b>fid level = #</b>	

The up and down buttons can be used to increase or decrease the vertical scale of the FID displayed. You can LC to toggle between **FID** and **Spectrum** (select FID), **Single** and **Summed** (select Single), **Grid on** and **off** (up to you).

Adjust the shim gradients as before, except that you are now observing the FID level and shape instead of the lock level. Unfortunately the usual bar graph and digital readout of the lock level are not available during FID shimming. The off-line analog lock level meter, however, is available and may be used to facilitate and speed up the shimming process, because its response time is much shorter and because most of the time, an improvement on the area and shape of the FID is accompanied by an improvement (or non-improvement, but not decrease) of the lock level.

When shimming is done,

LC Disconnect

disconnect from VNMR ACQUISITION window

**B. Real-time shimming on the spectrum.** This kind of shimming sounds good conceptually, because the final judgment on the shims *is* the spectrum and *not* the lock level or the FID. However, since one is judging the shims based on the line width and line shape, an *at* of around 10 seconds would be required for it to be meaningful (zero filling is not available), which will make the shimming a slow process, to say the least. Moreover, during spectrum-shimming, the lock level is not available as a criterion, and the spectral display is not interactive so a line width measurement is not possible. In practice, I would rather shim on the lock level, disconnect from the VNMR ACQUISITION window, take a scan and examine the spectrum, and shim again if necessary.

## 2) Interactive Autoshim.

Interactive autoshim is also accessed through the VNMR ACQUISITION window, and offers autoshimming on the lock level with a group of shim gradients (*i.e.* "Z1-Z2", "Z1-Z3", "Z1-Z4", "Z1,Z2,Z4", "Z1,X,Y,XZ,YZ", and "ALL NONSPINS") and a set of starting and ending criteria (*i.e.*, L>M, M>M, M>T, and T>T), both of which are selected by the operator. This mode of shimming could be beneficial when used after manual shimming on Z1C and Z2C, and on Z1F(-16+) and Z2F(-16+) to fine-tune the shims. Shimming on "Z1-Z2" or "Z1-Z3" with "M>M" will take 3 to 5 minutes, which is quite reasonable even for routine operations.

After manual shimming on the lock, and while you are still in the interactive shimming window,

LC SHIM:auto

select autoshim

RC HOLD on AUTO SHIM

display menu of autoshim methods.

Z1-Z2

For routine experiments, choose the

Z1-Z4

"Z1-Z2" or "Z1,Z2,Z3" shim method

Z1,X,Y,XZ,YZ

ALL NONSPINS

Z1,Z2,Z3

Z1,Z2,Z4

RC PULL on Z1-Z2, for example, then release

select an autoshim method

RC HOLD on QUALITY

display menu of shimming criteria.

L&gt;M

(L for Loose, M for Medium, and T

M&gt;M

for Tight) Choose "M&gt;M" for

M&gt;T

routine experiments, and "M&gt;T" or

T&gt;T

"T&gt;T" for high resolution work.

RC PULL on M&gt;M, then release

select "M&gt;M" shimming criterion

LC Start

start autoshimming routine

Once the autoshim is started, the Start button will change to Stop, and when the autoshim is finished, it will be changed back to Start again.

There is no time limit on this autoshim routine. If the lock level increases substantially and you wish to stop autoshimming,

LC Stop

stop autoshimming

It will cycle back to the most recent high value (but not necessarily the overall highest value). It might take a few minutes to stop after you have clicked this command, depending on which shimming criterion you chose. Do **not** attempt to do anything until Stop switches to Start. If it doesn't stop after 10 minutes, check with MSL staff.

### 3) **Background Autoshim.**

Background autoshim is controlled by the parameter *method* and the command *shim*. Unlike interactive autoshim, background autoshim allows shimming on the lock level *as well as on the FID*. It also has a wider range of shimming criteria (B, L, M, T, and E compared to L, M, and T for interactive autoshim) and a higher degree of flexibility and control in terms of customizing the shimmethod to fit a particular need. During routine operations, this method of shimming is most beneficial when used after manual shimming on the lock. Using the existing shimmethod 'fz123' (5-minute FID-shimming on Z1-Z3), for example, one can obtain a significant improvement on both the line width (a 0.05 to 0.1 Hz reduction is quite common with the 1% CHCl<sub>3</sub>/CDCl<sub>3</sub> standard) and the line shape.

Available shimmethods:

fz123 — 5 minutes, Z1, Z2, Z3 shimming on FID

fz12 — 5 minutes,

ft —

f

After manual shimming on the lock,

LC Disconnect

Select appropriate parameters if you haven't done so

Optimize sw and at

**method='fz123'** <rt>

**shim** <rt>

When done, the console will beep and the message "*Setup Complete*" will appear. You can then proceed with your experiment as usual.