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 <rtn>`
   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') <rtn>`
   retrieve QUAD shimfile

   `su <rtn>`
   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,CDC13 (for example)`  select an appropriate set of standard parameters
   `dg <rtn>`
   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

   ************************************************************************************
   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.
   ************************************************************************************

   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.

   a) 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.
   b) 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

**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)

<table>
<thead>
<tr>
<th>SOLVENT</th>
<th>CDCl3</th>
<th>C6D6</th>
<th>D2O</th>
<th>CD3OD</th>
<th>Ac-d6</th>
</tr>
</thead>
<tbody>
<tr>
<td>Z0*</td>
<td>-23</td>
<td>-16</td>
<td>136</td>
<td>220</td>
<td>298</td>
</tr>
<tr>
<td>LOCK POWER</td>
<td>20 - 25</td>
<td>10 - 15</td>
<td>10</td>
<td>5 - 10</td>
<td>5 - 10</td>
</tr>
<tr>
<td>SHIM FILE</td>
<td>QUAD</td>
<td>QUAD</td>
<td>QUAD</td>
<td>QUAD</td>
<td>QUAD</td>
</tr>
</tbody>
</table>

* Z0 will change with time.

**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 ≈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') <rtn>**, which will display the fid with a scale in seconds.

- `at=______` (minimum determined above)
- `d1=1 <rtn>`
- `ga <rtn>`
- `gf <rtn>`

LC **Connect**
LC **SHIM**
LC **FID**

The following is displayed:

<table>
<thead>
<tr>
<th>16/16 bits</th>
<th>FID</th>
<th>Spectrum</th>
</tr>
</thead>
<tbody>
<tr>
<td>up</td>
<td>Single/Summed</td>
<td>Grid on/off</td>
</tr>
<tr>
<td>down</td>
<td>fid level = #</td>
<td></td>
</tr>
</tbody>
</table>

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,
**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 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 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.
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% CHCl3/CDCl3 standard) and the line shape.

Available shimmethods:

- **fz123** — 5 minutes, Z1, Z2, Z3 shimming on FID
- **fz12** — 5 minutes,
- **ftt** —
- **f**

After manual shimming on the lock,

`LC Disconnect`

Select appropriate parameters if you haven't done so

`Optimize sw and at`

**method**='fz123' `<rtn>`

**shim** `<rtn>`

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