The Bruker DRX-500 NMR Spectrometer

Features:

- Temperature Control: Stable and Reproducible
- Linux computer, Dual Screen
- Automatic shimming
- New software (TopSpin)
- New BBO probe: excellent ^{13}C sensitivity, also ^{31}P, ^{11}B, ^{29}Si, ^{15}N, etc.
- Shaped Pulses: Selective 1D Experiments
- Gradients: Fast and Clean 2D Experiments

Magnet: Oxford 11.74 Tesla Superconducting Magnet, installed in the 1980's. This magnet drifts down in field and requires lowering the 1H frequency by 0.1 MHz every 18 months. We are now operating at 499.58 MHz, down from the original 500.13 MHz. The magnet drift means that the shim and lock settings change gradually and must re readjusted about once a week.

Console: The radio frequency electronics (transmitter and receiver) and the lock, shim and gradient controls are in a large two-bay cabinet, along with the temperature controller and a computer that runs the console. All of this was installed new in 1999, at a cost of $330,000. It has all of the modern NMR capabilities, including three RF channels, gradients, shaped (selective) pulses, and digital filtering.

Air Dryer: House air is dried by a two-column dessicant dryer mounted on the wall (large blue device on West wall). Every 5 minutes the two dessicant columns switch roles between active and regenerating, and it lets out a large noise. You'll get used to it.

Air Chiller: A low box on the floor in the NE corner of the lab cools the air to -40C and sends it through a thick hose (the "elephant's trunk") to the probe. This cold air is heated in the probe to the desired temperature (normally we use 298K or 25C) and this temperature is tightly controlled by a feedback loop. The variable temperature (VT) controller on the upper right side of the console displays the temperature in degrees Kelvin. This is the temperature of the air moving past the sample, measured by a thermocouple wire just below the sample. During rainy or humid weather the air dry sometimes lets enough moisture through to create ice buildup in the air chiller. This eventually blocks the passage of air and we have to warm it up. During the warm up and drying process, the sample temperature will be set higher, to 303K (30C). **Always check the sample temperature when you start.**

Computer: The "cockpit" consists of a dual flat-screen monitor, keyboard, mouse, Linux computer and the BSMS keyboard. The BSMS keyboard is really part of the console; it controls the sample eject / insert, the spinning, the shims and the lock. The single large knob allows you to adjust a number of parameters, including the shims, by first activating the parameter by pushing a button. The Linux computer was installed in December, 2009, along with a new

console computer and a new deuterium transmitter board. This allows us to use the new TopSpin software and to shim using automatic deuterium gradient shimming. The automatic shimming ("gradshim") maps the field homogeneity along the vertical (z) axis and corrects it automatically using the shims. NMR data is located at **/opt/topspin/data/<login>/nmr** where <login> is your login ID.

BBO probe: The broadband observe probe is also part of the December, 2009 upgrade. The inner (most sensitive) receiver coil is tunable to any nucleus ("broadband") and the outer coil is tuned to ^{1}H (500 MHz). The z-axis gradient makes the automatic gradient shimming possible. This probe has excellent ^{13}C sensitivity and is also very sensitive for observing other nuclei, such as ^{31}P, ^{11}B, ^{29}Si, ^{77}Se, ^{17}O and ^{15}N. Tuning and matching of the inner coil is accomplished by sliders, which slide up and down using a tool that hangs from the probe (see last page). The sliders define a number, each one clicking into place at a position from 0 to 9. Once the sliders are set to the rough numbers, fine tuning and matching can be done using the "**wobb**" command in TopSpin. This is done by sliding the last digit (rightmost) sliders up and down for the tune and match. These sliders are continuous and don't click into place at the digits. **Matching** moves the "V" pattern down to touch the bottom of the wobb display (ignore any right-left motion), and then **tuning** centers the "V" on the vertical line in the center of the display. The outer coil (^{1}H) does not change much from sample to sample so we don't tune and match it.

Inverse Probes: An inverse probe has the ^{1}H coil on the inside, the most sensitive position, and the other coil on the outside. The "**nbb**" probe is an inverse broadband probe, with a tunable outer coil. Tuning is done by inserting long rods to set the rough frequency range, then turning knobs to fine tune. The inner coil is ^{1}H. The "**n3**" probe is an inverse probe with the outer coil double-tuned to ^{13}C and ^{15}N. Both probes have gradients on the x, y and z axes. These probes are for high ^{1}H sensitivity, especially for two-dimensional experiments like HSQC and HMBC. Probe changes must be done by NMR facility staff.

Overview of Bruker TopSpin Software

From top to bottom in the TopSpin window:

First Row: 9 Drop-Down Menus

Second Row: 29 Command Icons

Third Row: 22 Display Icons (*2, /2, *8, /8, etc.)
       Hold the cursor over any icon and a text explanation will appear

Display Frame: This is the main graphics frame. It is specific to your dataset, with the dataset name and experiment number at the top. Multiple datasets can be open at the same time, and they can be minimized to icons or maximized to the full graphics frame.

   Browser: The small triangles to the left of the display frame at the top open and close the browser. This is a file selection browser that allows you to look through your NMR data and select any dataset and drag to the display frame. You can open and close folders and their contents will be displayed. A specific experiment number (1, 2, ..) must be dragged into the display frame to select a dataset.

   Tabs: There are 10 tabs which open different things to be displayed in the graphics frame: **Spectrum** and **FID** are data displays, **Title** is a text editor to enter of change the title, **Peaks** and **Integrals** are data lists, and **ProcPars** and **AcquPars** are complete parameter lists.

Command Line Text Entry: A long pink box beneath the display frame is for entering text commands and parameters, just like in XWinNMR. You must click in the frame to activate it.

Acquisition Status Bar: At the bottom of the TopSpin window is a bar which contains, from left to right:

   Acquisition Status (scan number, total scans, time remaining)

   FID flash (a red FID icon flashes when an FID is being acquired)

   Lock (a miniature lock window, with the lock level scanning back and forth)

   Temperature (the sample temperature, from the variable temperature unit)

   Time (the time of day)

<u>**Reminder Sheet for Bruker DRX-500 Operation with TopSpin Software**</u>

I. Insert Sample, Lock and Shim.

1. <u>Login</u>: Enter your login name and password. *First login only:*
    a. Specify Printer Driver: Model: HP Color LaserJet 4550   Click **OK**.
    b. Click on: **Applications / System Tools / Terminal** (to bring up Linux window)

2. <u>Start NMR Software</u>: Click on: Applications / Bruker / TopSpin 1.3

    Maximize the TopSpin window by clicking on the ▣ at the upper right corner.

3. <u>Change Sample</u> (BSMS keyboard): **Lock Off**, **Spin Off**, **Lift On/Off**, remove standard ($CHCl_3$/$CDCl_3$) sample. Replace standard with your sample in the spinner, use the BBO Probe depth gauge to set the sample depth and clean with ethanol. Place sample at top of magnet bore.. **Lift On/Off**. When sample is down, **Spin On**.

4. <u>Auto Lock</u>: Enter **lock**, select solvent from list. Double-click on Lock icon [icon] or mini-

    lock display [Lock display icon] to bring up the full lock display on the screen.

5. If shims are bad, <u>Read in Shims</u>: **rsh current-bbo** (at TopSpin command line)

6. <u>Manually Shim X,Y Shims</u> (optional): (BSMS keyboard) **Spin Off. X, Z0** buttons; **Y, Z0** buttons; Turn **Fine** button off: **X, Z; Y, Z; X, Z2**; **Y, Z2**; **X2-Y2, Z0; XY, Z0**. Turn on **Fine** and **On-Axis** buttons. **Spin On**.

7. <u>Automatic Shim</u>: Enter *gradshim*. In gradshim window, under **Data Set**, replace *gradshim* with your username in the **USER** box. Click on **Start Gradient Shimming**. The first time you will get the error message: *No source directory! Copy 'standard 1D' parameters.* Click on **Close**. When Shim Results / Z-axis Shim Maps is displayed, scroll up and check:

    Current Field Map Statistics
        RMS deviation…   **xxx**   Hz
    Predicted Field Map Statistics
        RMS deviation…   **yyy**   Hz

The "xxx" number is the starting quality of shimming, and the "yyy" is where you should be after one round of gradient shimming. A good "yyy" value (lower is better) is 0.12 Hz. Click **OK** to exit the **Shim Results** window. If desired, click again on **Start Gradient Shimming** to repeat the above procedure, up to three rounds total.

When finished, click Exit in the gradient shimming window. To return to your dataset, click on the ↵ arrow just to the right of the STOP sign [🛑] at the top of the display frame.

II. Acquire an FID.

1. Create Data Area: Click on **File** and **New** (leftmost drop-down menu) and edit the **Dataset Name** and **EXPNO** fields. Click on **Experiment** and scroll down to the bottom of the list. Select **std-1h-bbo** (for ^{1}H) or **std-13c-bbo** (for ^{13}C) or any other "std" experiment. Edit the **Title** box to enter a short description of the sample and experiment. Click **OK**.

2. (^{13}C Only) Tune and Match the Probe: Enter *wobb*. Adjust rightmost **Match** slider to get "V" pattern down to touch baseline, Adjust rightmost **Tune** slider to center "V" pattern on vertical line (see last page for diagram of BBO probehead). Enter *halt* when finished.

3. Adjust receiver gain: Enter *rga*   (wait for *rga: finished* at lower left)

4. Begin Data Acquisition: Enter *zg* (**Acquisition information** window shows scans and remaining time; **FID flash** marks individual FID recording) For ^{13}C, enter *tr*, *ef* and *apk* to process data during acquisition, enter *halt* and *efp* when satisfied.

5. Remove Sample and Insert Standard: Use the BSMS keyboard to turn off the Lock and Spin, turn on the Lift. Replace your sample with standard ($CHCl_3$/$CDCl_3$) sample in the spinner, adjust height and clean with ethanol. Insert the standard and lock on $CDCl_3$.

III. Processing Your Data.

1. Fourier Transform: **^{1}H**: Enter *sinm*, then *ft* (automatically switches display to spectrum window). **^{13}C**: Enter *ef*. **Both**: Enter *apk* to automatically correct phase.

2. Adjust Spectrum Display: Use icons in third row: to see full spectrum; ***2**, ***8**, **/2**, **/8** to change vertical scale; up and down arrows to move spectrum up and down. Click and drag up or down on icons   (vertical position) or   (vertical scale). To expand horizontally, simply position cursor (vertical line) and click and drag region to be expanded.

3. Manual Phase Correction (if needed): Click on   icon (second row, 10th from left). In the display frame at upper left, use the **0** and **1** icons for zero-order (at the pivot) and first-order (away from the pivot) corrections. The pivot is the vertical red cursor. Click ***2** a few times to exaggerate the phase errors, and expand the region of interest horizontally. To set the **pivot**: right click on a peak and left click on "**Set Pivot Point**". Click and hold on the "**0**" or "**1**" icon and drag up and down to adjust phase. Click on the   icon (save and return) when finished.

4. Set Reference Chemical Shift: Expand the area of the reference peak by clicking and dragging the vertical line. Click on the   icon (Spectrum calibration, second row 11th from

left). Position the cursor on the reference peak, left-click and enter the reference ppm value. Click **OK** and the [icon] (show full spectrum) icon.

5. Integrate: Click on the [icon] (**Interactive integration**, second row, 14th from left) icon. Expand the region of interest and adjust the vertical scale. In the data display frame, click on the second icon from the left [icon] (Define new region using cursor – toggle). Now click and drag horizontally to select each integral region. Select an integral to normalize by right-clicking within the region of the integral and selecting **Calibrate**. Enter the desired integral value and click **OK**. You can select all integrals and adjust their vertical scale. Under the **View** drop-down menu (top of TopSpin window) you can select **Display Properties** and turn off the integrals and/or integral regions. Click on the [icon] (Save and Return) icon to exit the integration mode.

9. Peak Picking: Click on the [icon] (**Manual Peak Picking**) icon in the second row. Select both the horizontal and vertical limits by clicking and dragging a rectangular box with the mouse. All peaks within this region and having a peak height within the vertical regions will be picked. The parameter **PC** (enter **pc** at command box) can be made smaller to get missed peaks to be selected. You can manually add any missed peaks by clicking on the [icon] (**Define peak manually**) icon in the upper left of the display frame. Then use the vertical cursor to select peaks with the left mouse button. Click on the [icon] (Save and Return) icon to exit peak picking mode.

## IV. Plotting Your Spectrum.

1. Plot as Spectrum Appears in the Display Frame. Click on **File / Print** (top row, left) and check the button for "**Print Active Window**". Click **OK**, **Print** and **OK**.

2. Plot Using the Plot Editor. Click on File / Print (top row, left) and check the button for "Print with Layout – start Plot Editor". This brings up the plot editor with a specific layout of objects. The layout can be changed and saved as your preferred layout.

3. Exit TopSpin and Log Out. Your data is already saved and you can return to it at any time. Click on **File** and **Exit**, then **OK** to leave **TopSpin**.

4. Exit from Linux. At the top left of the screen, click on **Actions** and **Log Out**. Confirm by clicking on **OK**. Your billing session is now ended.

BBO (Broadband Observe) Probe
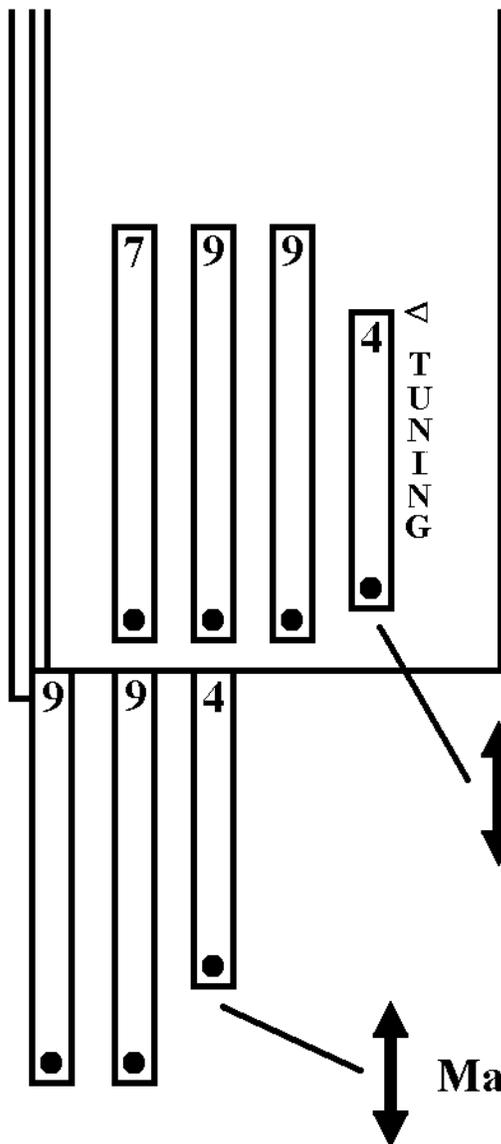
	Tune	Match
^{13}C	799.4	99.4
^{31}P	899.6	99.6
^{15}N	778.2	86.3
^{11}B	898.2	99.4

TUNING

7 9 9

4

9 9 4

Tune ^{13}C

Match ^{13}C

Tuning Tool