

PINMRF

Bruker Avance / ARX NMR Spectrometers - Training Guide for Basic 1D NMR Spectroscopy

INCLUDING:

Avance DRX500-1 w/ 5mm TXI Cryoprobe – 367 WTHR

Avance DRX500-2 w/ 5mm TXI or BBO Probes – G43 RHPH

ARX400 w/ 5mm QNP Probe – 369 WTHR

ARX300 w/ 5mm QNP Probe – G43 RHPH

Avance DPX300 w/ 5mm QNP Probe – 408 HANS

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05-17-2009: Updated - JSH.

11-10-2006: Updated - JSH.

Basic Spectrometer Operation Guidelines – 1D Spectra

Bruker Avance / ARX NMR Spectrometers

PINMRF

Logging on to the SGI O2 computer and starting XWINNMR

1. Click on the icon with your login ID or enter your login ID in the login field
2. Enter your password in the password field.
3. After the window manager starts, you will see two windows on the screen: one is the "console" window and one is a UNIX shell window. Move the mouse pointer to the UNIX shell window and type `xwinnmr` to start the NMR program.
4. The NMR program will start and you will see the main window appear.

H1 experiment setup

5. Type `edc` (edit current dataset) to create a new dataset for your acquisition (or you can use the currently displayed dataset if you want to overwrite the existing data). When the `edc` window appears, type in the new dataset name in the NAME field and type in the desired experiment number in the EXPNO field (the PROCNO should be 1 and the DISK (/u), USER (your login ID) and TYPE (nmr) fields should not be changed) then click on SAVE to close the window.
NOTE: EXPNO can be any number but we suggest 1 for proton, 2 for carbon, 3 for DEPT, etc.
NOTE: for the NAME field, do not include spaces or special characters in the name – use only letters, numbers, and the – (dash) and _ (underscore) characters.
6. Type `rpar h1.prob.solv` to read the standard proton parameters. A window will appear listing the different sub-components of the parameter set; click the COPY ALL button to load all relevant parameters (OR, highlight the parameter types you want to load and then click COPY).

NOTES – Parameter set nomenclature:

The standard PINMRF parameter sets for Bruker spectrometers have filenames containing three fields separated by periods. The first field is for the nucleus and/or experiment, the second is for the sample probe, and the third is for the

solvent. All of the current standard parameter sets are listed on page 10 of this handout. Here are a few examples:

- h1.qnp.cdcl - H1 experiment, QNP probe, CDCl₃ solvent;
- c13.cryo.meth - C13 experiment, Cryoprobe, methanol-d4 solvent;
- c13dept.qnp.d2o - C13 DEPT-135 experiment, QNP probe, D₂O solvent;
- cosy.qnp.acet - H1 2D-COSY experiment, QNP probe, acetone-d6 solvent.

“Probe” field choices: cryo (500-1), bbo or txi (500-2), qnp (300’s, 400);

“Solvent” field choices: acet, cdcl, d2o, dmsol, meth.

NOTE – DRX500-2:

Check the note adjacent to the spectrometer to determine which probe is installed prior to reading the parameter set.

7. To examine the acquisition parameters you can use either the `eda` (edit acquisition parameters) or the `ased` (acquisition setup editor) commands. Each brings up a window; after examining and changing anything (if necessary), click the SAVE button to close the window and save the changes, or click the CANCEL button to close the window without saving any changes.

NOTE – DRX500-1:

Prior to acquiring any data using the cryoprobe, please check through the instructions on the yellow sheet to make sure that you know how to operate the system correctly!! If you are using a non-standard experiment, you must also check the Bruker publication Typical Pulses for the 5mm Cryoprobe, a copy of which is in the binder, to ensure that you will not exceed the probe’s power limits.

Changing samples, locking and shimming

8. Move the mouse cursor to the main window and type `lockdisp` to display the lock window. A sample of CDCl₃ resides in the magnet when the spectrometer is not in use, and this sample should be locked.

9. On the keypad, turn off LOCK (and SPIN, if spinning) function(s), then press the LIFT key

to eject the CDCl_3 standard sample. NOTE: the lift function on the ARX spectrometers requires the orange shift key to be depressed.

10. Place your sample (CLEAN IT!!) in the spinner and use the depth gauge to check the sample depth. Place the spinner on the air column at the top of the magnet, and press the LIFT key again to insert the sample. Wait for the sample to seat, then press the SPIN button to start spinning.

11. Make sure the main window is highlighted and type `lock_solv`, where `solv` = `acet`, `cdcl`, `d2o`, `dms` or `meth`, according to the solvent you are using. Wait for lock to be established, and for any lights on the keypad to stop blinking.

12. Shim your sample using Z1, Z2 and Z3 shims. Adjust the LOCK GAIN and/or LOCK POWER as necessary. NOTE: on the DRX-500 keypads, the ON AXIS function must be on.

13. After shimming is completed click on the spectrum window to make it active.

H1 data acquisition

14. Type `acqu` to display the acquisition window (for FID display).

15. Type `rga` to set receiver gain - wait for message telling you it is finished.

16. Type `zg` to start acquisition. The command `halt` can be used to stop an acquisition before the requested number of scans (`ns`) is completed. NOTE: during a longer acquisition the command `tr` can be used to transfer the FID to disk. The `ef` command will then transform that FID and display the result. Use the `acqu` command again to return to the FID display.

C13 / other nuclei experiment setup, data acquisition

11. Use the `edc` command to create a new EXPNO for the C13 / other nucleus acquisition.

12. Type, for example, `rpar c13.prob.solv` to read the standard carbon-13 parameters, then click the COPY ALL button to load all relevant parameters (OR, highlight the parameter types you want to load and then click COPY). PINMRF standard experiments are listed on page 11.

13. To examine the acquisition parameters you can use either the `eda` (edit acquisition parameters) or the `ased` (acquisition setup editor) commands.

14. Type `acqu` to display the acquisition window (for FID display).

15. Type `rga` to set receiver gain - wait for message telling you it is finished.

NOTE: **do not** use the `rga` command when observing C13.

16. Type **zg** to start acquisition. The command **halt** can be used to stop an acquisition before the requested number of scans (**ns**) is completed. NOTE: during a longer acquisition the command **tr** can be used to transfer the FID to disk. The **ef** command will then transform that FID and display the result. Use the **acqu** command again to return to the FID display.

NOTE – DRX500-1:

16a. **Before** executing the **zg** command, type **crplock** to set the cryoprobe preamplifiers correctly for C13 observation. Then proceed as outlined above. NOTE: when you want to observe H1 again, you will need to type **crpon** prior to starting the proton acquisition.

Data processing and plotting

17. To examine the processing parameters you can use the **edp** (edit processing parameters) command. This brings up a window; after examining and changing anything (if necessary), click the SAVE button to close the window and save the changes, or click the CANCEL button to close the window without saving any changes. NOTE: the FID is always stored on disk so the **ef** command can be retyped at any time to generate a new spectrum after, for example, a processing parameter has been changed.

18. After acquisition is finished type **ef** to do the Fourier Transform. NOTE: after transformation the spectrometer will automatically switch to the spectrum display window.

19. **PHASING**: Using the left mouse button, click the PHASE button on the menu to the left of the spectrum display to enter the phasing routine. Click the BIGGEST button to apply a default phase correction using the biggest peak in the spectrum as the reference point. Then, move the mouse pointer over the PH0 button, hold down the left mouse button and move the mouse vertically up and down to adjust the 0-order phase correction. When the biggest peak is phased correctly, repeat the above process using the PH1 button, then iterate back and forth between PH0 and PH1 until the entire spectrum is phased correctly.

20. Click on the RETURN button to exit phasing; click the SAVE & RETURN choice that appears to save your phase corrections.

21. **PHASING – alternate**: type **apk** to carry out automatic phasing on the spectrum. This will not work unless the signal-to-noise ratio of the spectrum is good.

22. SPECTRUM MANIPULATION: To expand the spectrum and to move and/or change the expanded region, use the arrow buttons to the left of the spectrum display. These buttons have labels such as \uparrow , \rightarrow , etc. In addition, if the mouse is moved over the spectrum window and the left button clicked, the mouse pointer will jump onto the spectrum to give the chemical shift readout. The middle mouse button will then freeze the cursor in the desired location. Moving the mouse again will move a second cursor, and clicking the middle button again will expand the region between the cursors. Now click the left mouse button to release the cursor from the spectrum.

Other manipulations:

- *2, /2, *8, /8: click these buttons to adjust vertical scale up or down by factor 2 or 8;
- move the cursor over the spectrum, hold the middle mouse button down: move mouse left - right to scroll spectrum left - right;
- move the cursor over the spectrum, hold the right mouse button down: move mouse left - right to expand or contract spectrum.

23. CHEMICAL SHIFT REFERENCE: This can be used to correct the chemical shift of a known peak such as the solvent or TMS. Expand the region around the peak of interest, then left-click the CALIBRATE button to the left of the spectrum display. Move the cursor to the known peak and click the middle mouse button to set the chemical shift for this peak.

24. DEFINE PLOT REGION: To define a region for plotting, click on the DP1 button to the left of the spectrum display. A window will appear asking for the plot limits. If you want to plot the spectrum region as displayed, just press return twice; if you want to plot a specific region, enter the desired downfield and upfield plot limits and then y to the scale question, and that region will then be displayed.

25. If you want to expand the spectrum vertically for plotting, type **CY** and enter the desired value; the default is 12 - 15 cm.

26. TITLE: To enter a title for the plot, type **setti**. A text-editor window will open. This window uses a graphical editor. The default title will be shown. Move the mouse pointer to the editor window and edit the title as you would using any graphical text editor. When you are finished, move the mouse pointer to the FILE button in the upper left of the window. Hold down the left mouse button and move the pointer down the pop-up menu to the SAVE choice and then

release the button; this will save the text file. Repeat this process, and go to the EXIT choice to close the editor window.

27. **PEAK PICKING:** To check the peak picking, type **pps**. This will bring up a new window with the peaks listed. Click on the OK button to close this window. If you want to change the peak picking threshold, type **mi** and enter a new value in the window that appears. Repeat **pps** if necessary.

28. **INTEGRATION:** Click on the INTEGRATE button to the left of the spectrum window. Move the mouse pointer to the spectrum and click the left mouse button to put the cursor on the spectrum. Move the cursor to the downfield limit of integration and click the middle mouse button; move the cursor to the upfield limit and click the middle button again. Move the cursor back downfield a little and click the left mouse button to highlight this new integral trace (the trace will then show an asterisk at its end). Now move the pointer over to the left-side buttons and place the pointer over the BIAS button. Hold down the left mouse button and move the mouse up and down to adjust the bias (similar to how the phasing was done); repeat the process using the SLOPE button until the integral trace looks correct. NOTE: the bias effects the whole integral trace equally while the slope is frequency dependent with the downfield end of the trace as a reference point.

29. Move the pointer back to the spectrum and click to left mouse button to place the pointer on the spectrum. Now move the cursor on the spectrum to the most downfield break point desired in the integral trace. Click the middle mouse button to break the integral trace, move the mouse upfield the desired amount, and click the middle mouse button again to restart the integral trace. Repeat as necessary. When all the desired regions are defined, highlight one of a known area with the left mouse button, move the pointer to the left of the spectrum and click on the CALIBRATE button, then enter the desired integral area in the window that appears.

30. When the integration is complete, adjust the vertical scale of the integrals using the *2 or /2 buttons adjacent to the ALL button, then click on the RETURN button to exit; click the SAVE & RETURN choice that appears to save your integrals.

31. PLOTTING:

a) Prior to plotting the spectrum on paper, type **view** to see the plot output displayed on the screen (in a new window). Check that the plot is correct prior to plotting to paper. Click on the QUIT button in this new window to close the view window.

b) To plot the spectrum on paper using the default parameters, type **plot**. This will plot the spectrum, the axis, the integration (proton only), the peak picking, the title, and the parameters. If you want to alter this, type **edg** (edit graphics parameters). A window will appear with buttons for the choices regarding the items included in the plot; after examining and changing anything, click the SAVE button to close the window and save the changes, or click the CANCEL button to close the window without saving any changes. If you delete integration and/or peak picking, increase **CY** prior to issuing the plot command.

32. PLOTTING - alternate:

a) Instead of using the “built-in” plot routines, you can use XwinPlot to plot your spectra. After defining the plot region, etc., as in steps 22 -26, type **xwinplot**. This will open a new window with the XwinPlot editor displaying your spectrum. Consult the XwinPlot manual for XwinPlot-specific commands.

Finishing up with XWINNMR and logging off the SGI O2 computer

33. Click on the LOCK DISP icon or on the border of the lock window to open the window or bring it to the front.

34. Remove your sample and replace it with the CDCl₃ standard following steps 9 & 10 above.

35. Type **standard** to set up the spectrometer for the standard CDCl₃ sample.

36. Touch up the shims of the CDCl₃ standard sample following step 11 above.

37. Click the QUIT button in the lower right corner of the lock window to close the lock window.

38. If you have been running spectra other than proton, use the **edc** command (step 8) to read a proton dataset, and then type **ii** to initialize the spectrometer back to proton.

39. Type **exit** to leave the NMR program. A confirmation window will pop up - READ IT! If it asks "Do you want to leave XWINNMR?", click the OK button. If it says "Processes are still active - Kill them?", click the CANCEL button. Go back to the XWINNMR window and double

check that the lock window is not still running and hidden somewhere. Type `lockdisp` to re-display the lock window, then click the QUIT button in the lower right corner of the lock window to close the lock window. Type `exit` again - if processes are still active, call PINMRF staff for assistance.

40. Move the mouse pointer to the background of the UNIX desktop and hold down the right mouse button. A desktop menu will appear. Keep the right mouse button depressed and move the cursor to the LOG OUT choice, then release the right mouse button. A logout screen will appear: click YES with the left mouse button to logout from UNIX.

Supplemental Instructions

S1. USING THE SAMPLE TEMPERATURE CONTROLLER: To regulate the sample temperature between room temperature and ca. 100 deg. C (DRX500-1: 55 deg. C), within the limits of your solvent, you may use the sample temperature controller. Type `edte` to open the temperature-control window. To change the temperature set point, click the CHANGE button next to the “Target temp.” readout. In the pop-up window, enter the new set point (**in degrees Kelvin!**) in the “Sample target temp” field, then click APPLY and OK to load the new set point. Next, set the heater power limit by clicking on the SET MAX.. button adjacent to the “Heater” readout. In the pop-up window, set the heater power limit value either by typing it in the field or adjusting the slider, then click on APPLY and OK to load the new heater limit. If the heater is not on, click the ON/OFF button on the left side of the “Heater” display to cycle the heater on and off as necessary (ARX spectrometers: use the physical “Heater” button on the front panel of the temperature control unit for this function – a red light will illuminate when the heater is enabled). Turn off the heater when you are finished with your experiments; on the DRX500-1, leave the unit set to 298K with the heater enabled. To close the edte window, left-click the “FILE” pulldown in the upper left of the window, and go to the “EXIT” choice in the menu.

NOTE: to regulate the sample temperature below room temperature, on the DRX500 spectrometers you may turn on the cooling unit located near the magnet. This will allow sample temperatures to be regulated down to ca. 0 deg. C. For lower temperatures, or on the 300/400 spectrometers, liquid nitrogen must be used for cooling. Please contact PINMRF staff for assistance with the liquid-nitrogen cooling equipment.

XWINNMR

Commonly Used Keyboard Commands

NOTE THAT ALMOST ALL COMMANDS ARE AVAILABLE FROM THE PULL-DOWN MENUS AT THE TOP OF THE SPECTRUM DISPLAY

Parameter Setup

edc - edit current dataset - reads existing and/or creates new datasets
eda - edit acquisition parameters
ased, edasp - edit acquisition parameters - pulse program driven or nucleus-related
gpro - update current experiment with current probe's pulse calibration parameters
edte - set up temperature controller parameters
edp - edit processing parameters
edg - edit graphics parameters - controls plot output attributes
wrpa - copy current dataset to a new one - specify new name and experiment number
re - read new dataset - specify new name and experiment number

Data Acquisition

lockdisp - display lock window
lock - start autolocking routine using Bruker default parameters
rpar - read parameter set
rsh - read shim set
wsh - save shim set - enter a unique name in the menu
ii - initialize interfaces
acqu - display acquisition window - shows FID on screen
wobb - start wobble routine for probe tuning
rga - set receiver gain automatically
ns - number of scans
2s ns - display number of scans stored with current dataset
zg - zero current data and start acquisition
go - start data acquisition
tr - transfer FID to disk for processing
halt - halt data acquisition after next scan
stop - stop data acquisition immediately
standard - set up spectrometer for CDCl₃ standard sample

Data Acquisition - DRX500-1 - Cryoprobe Specific

crpon - enable cold preamplifier for H1 observation
crplock - enable warm preamplifier for C13 observation
cwobb - start wobble routine for cryoprobe tuning

Custom Acquisition AU Programs for Advanced 1D Spectra

deptjsh - turn current C13 dataset into DEPT experiment and acquire

homodecjsh - turn current H1 dataset into a homodecoupling experiment and acquire

noediffjsh - turn current H1 dataset into a 1D nOe-difference experiment and acquire

presatjsh - turn current H1 dataset into a presaturation experiment and acquire

Data Processing and Plotting

setti - enter title for plot

ft - Fourier transformation

ef - exponential multiplication and Fourier transformation

efp - carry out “ef” plus apply existing phase corrections to spectrum

multiefp - carry out “efp” on multiple spectra in sequential experiment numbers

lb - controls amount of exponential multiplication

sref - set chemical shift scale reference using TMS or default solvent parameters

nzp - number of data points to zero at start of FID

zp - zero nzp points at start of FID

basl - enter baseline correction routine

pscal - define plot vertical scaling method

cy - plot vertical scaling

cx - plot horizontal scaling

mi - threshold for peak picking

pps - peak picking with output on screen

pp - peak picking with output on paper

view - view plot output on screen

plot - plot spectrum on paper

dual - enter dual display mode - EXPNO2 and PROCNO2 parameters must be correct in the menu

xwinplot - start XwinPlot standalone program using current dataset

xwp - start XwinPlot standalone program using current plot region

Data Handling

dir - show a listing of your experiment names

dirf - show a listing of all 1D FID files (includes experiment names and numbers)

dirser - show a listing of all 2D SER files (includes experiment names and numbers)

dirs - show a listing of all 1D spectra (includes experiment names and numbers)

dir2d - show a listing of all 2D spectra (includes experiment names and numbers)

dels - delete 1D processed spectra (FID's are not deleted)

del2d - delete 2D processed spectra (SER files are not deleted)

Notes

Bruker Avance / ARX Spectrometer Parameter Sets - 07-01-2006

NOTE: “solv” = solvent = acet, cdcl, d2o, dmsu, or meth (for C₆D₆ use cdcl).

NOTE: some parameter sets do not include a “solv” field in their filename.

Parameter Sets

DRX-500-1 (cryoprobe)

h1.cryo.solv
presat.cryo.d2o
c13.cryo.solv
c13dept.cryo.solv
cosy.cryo.solv
tocsy.cryo.solv
noesy.cryo.solv
hmqc.cryo.solv
hmbc.cryo.solv

DRX-500-2 (bbo or txi probe)

h1.bbo.solv	h1.txi.solv
presat.bbo.d2o	presat.txi.d2o
c13.bbo.solv	c13.txi.solv
c13dept.bbo.solv	c13dept.txi.solv
cosy.bbo.solv	cosy.txi.solv
tocsy.bbo.solv	tocsy.txi.solv
noesy.bbo.solv	noesy.txi.solv
hetcor.bbo.solv	hmqc.txi.solv
p31.bbo	hmbc.txi.solv
p31nd.bbo	
si29.bbo	
ga69.bbo	
ga71.bbo	
n15.bbo	

ARX-400, DPX-300, ARX-300 (qnp probe)

h1.qnp.solv
c13.qnp.solv
c13dept.qnp.solv
f19.qnp.cdcl
p31.qnp.cdcl
p31nd.qnp.cdcl
cosy.qnp.solv
tocsy.qnp.solv
noesy.qnp.solv
hetcor.qnp.solv

Shim Files

DRX-500-1 (cryoprobe)

shims.cryo

DRX-500-2 (bbo or txi probe)

shims.bbo	shims.txi
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ARX-400, DPX-300, ARX-300 (qnp probe)

shims.qnp

Probe Tuning Guidelines – Various Probes
Bruker Avance / ARX NMR Spectrometers
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Spectrometer setup - H1 tuning

1. Run a quick proton experiment to determine that the sample is correct.
2. We will tune the H1 coil first. Turn off the sample spinning.
3. Type `acqu` to go to the acquisition window, then type `wobb` to start the probe tuning ("wobble") routine.

NOTE – DRX500-1:

Use the command `cwobb` to start the probe tuning routine.

Tuning the H1 coil - all probes

4. The computer screen will show a trace with a dip in it; hopefully the dip will be close to the correct frequency in the center of the screen. If there is no dip, click the WOBB-SW button to the left of the screen, then enter a new value in the correct field of the screen that pops up. The default SW is 4 MHz.; try 12 or 16 MHz. if it needs changing.
5. When you have confirmed that there is a dip present, go to the magnet and adjust the yellow or gold-colored tune and match screws under the probe to bring the minimum of the dip to the center of the screen and also as close to the bottom of the display as possible. NOTE: the location of the dip left to right is referred to as the "tune" and the depth of the dip (closeness to the baseline) is referred to as the "match." The tune screw will tend to move the dip left - right and the match screw will improve the depth of the dip. The display on the preamp housing also shows the quality of the probe tuning and can be used in conjunction with or instead of the computer screen display.
6. When the tuning is OK, type `halt` to stop the wobble routine.
7. Restart sample spinning if desired. NOTE: leave spinning off for 2D experiments.
8. Re-acquire the proton spectrum if desired.

Spectrometer setup - C13 and N15 tuning of the Cryoprobe and TXI probe

1. Carry out steps 1 - 8 above to ensure the H1 coil is tuned.
2. Use the `edc` command to read or create a new dataset.
3. Use the `rpar` command to read the correct parameter set for the X-nucleus you want to observe, then type `ii` to initialize the spectrometer. Turn off sample spinning.
4. Type `acqu` to go to the acquisition window, then type `wobb` (`cwobb` on DRX500-1) to start the probe tuning ("wobble") routine.

Tuning the X-nucleus coils - Cryoprobe and TXI probe

5. Proceed as above for H1 tuning, using the blue tune and match screws for tuning C13 and the red tune and match screws for tuning N15.
6. When the tuning is OK, type `halt` to stop the wobble routine.
7. Restart sample spinning if desired. NOTE: leave spinning off for 2D experiments.

Spectrometer setup - X-nucleus tuning of the BBO probe (DRX500-2)

1. Carry out steps 1 - 8 above to ensure the H1 coil is tuned.
2. Use the `edc` command to read or create a new dataset.
3. Use the `rpar` command to read the correct parameter set for the X-nucleus you want to observe, then type `ii` to initialize the spectrometer.
4. Type `acqu` to go to the acquisition window, then type `wobb` to start the probe tuning ("wobble") routine.

Tuning the X-nucleus coil - BBO probe

5. X-nucleus coil tuning is done using gold-colored sliders visible on the bottom of the probe. The sliders are numbered, and there is a directory of numbers for the tune and match settings for most common X-nuclei hanging below the probe. Confirm that the tune and match settings are correct for the X-nucleus you want to observe.
6. The computer screen will show a trace with a dip in it; hopefully the dip will be close to the correct frequency in the center of the screen. If there is no dip, click the WOBB-SW button to

the left of the screen, then enter a new value in the correct field of the screen that pops up. The default SW is 4 MHz.; try 12 or 16 MHz. if it needs changing.

7. When you have confirmed that there is a dip present, go to the magnet and adjust the gold tune and match sliders under the probe to bring the minimum of the dip to the center of the screen and also as close to the bottom of the display as possible. NOTE: the location of the dip left to right is referred to as the "tune" and the depth of the dip (closeness to the baseline) is referred to as the "match." The tune slider(s) will tend to move the dip left - right and the match slider(s) will improve the depth of the dip. The display on the preamp housing also shows the quality of the probe tuning and can be used in conjunction with or instead of the computer screen display.
8. When the tuning is OK, type `halt` to stop the wobble routine.
9. Be sure to tune the X-nucleus coil back to C13 before you leave the spectrometer.

Spectrometer setup - F19/P31/C13 tuning of the QNP probe

1. Carry out steps 1 - 8 above to ensure the H1 coil is tuned.
2. Use the `edc` command to read or create a new dataset.
3. Use the `rpar` command to read the correct parameter set for the X-nucleus you want to observe, then type `ii` to initialize the spectrometer.
4. Type `acqu` to go to the acquisition window, then type `wobb` to start the probe tuning ("wobble") routine.

Tuning the F19/P31/C13 -nucleus coil - QNP probe

5. Proceed as above for H1 tuning, using the red tune and match screws.
6. When the tuning is OK, type `halt` to stop the wobble routine.

NOTE - tuning of QNP probes:

As a general rule, the QNP probes will not need tuning by the user – these directions are included here for completeness only. Please be very careful if you decide to optimize the tuning on the QNP probes.

Bruker Avance / ARX Spectrometers - Probe Tuning Matrix

Which experiments in general will require probe tuning?

Type of Experiment	ARX/DPX-300/400	DRX-500-2		DRX-500-1
	QNP probe	TXI probe	BBO probe	Cryoprobe
1D proton	No	No	No	No
1D fluorine	No	N/A	N/A	N/A
1D phosphorus	No	N/A	Yes	N/A
1D carbon	No	Yes	Yes	Yes
1D other nuclei	N/A	N/A	Yes	N/A
1D DEPT, presat., etc.	Possibly	Yes	Yes	Yes
2D COSY	No	Yes	No	Yes
2D TOCSY, NOESY, etc.	Possibly	Yes	Yes	Yes
2D 1H obs. X dec. (HMQC)	N/A	Yes	Yes	Yes
2D X obs. 1H dec. (HXCO)	Possibly	Yes	Yes	Yes
3D, 4D any	N/A	Yes	Yes	Yes

When tuning is required all relevant nuclei should be tuned, e.g., both ^1H and ^{13}C for a carbon-13 acquisition with ^1H decoupling.

Instrumental Overview – Approximate Sensitivity (S/N), 5mm Probes

Console/Probe	^1H sensitivity	^{13}C sensitivity
Varian 300's, quad-nucleus probes	100:1	90:1
ARX/DPX-300's, QNP probe	130:1	120:1
ARX-400, QNP probe	230:1	190:1
DRX-500-1, Cryoprobe	3800:1	200:1*
DRX-500-2, TXI probe	800:1	90:1*
DRX500-2. BBO probe	350:1	300:1

*These probes will show a background signal or rolling baseline when used for ^{13}C acquisition.

**Checkout Practical Exam Requirements -
Bruker Avance / ARX NMR Spectrometers
PINMRF**

Prior to performing this practical exam you must first pass a written exam. The written exam will test you on material in this document as well as spectrometer-related matters.

This is an outline of the tasks you will be required to perform correctly in order to pass the checkout practical exam. You will be allowed up to 30 minutes to complete the following tasks. You may use any training handouts and notes you have. Standard samples will be used for the checkout and will be provided by the NMR lab. PINMRF staff may, at their discretion, observe you performing all or part of the checkout exam.

Test Sample - 10% Ethylbenzene (or other sample) in CDCl₃

- login to computer and start XWINNMR, open lock display
- remove CDCl₃ standard, insert new sample, lock and shim
- create new dataset
- check ¹H probe tuning
- run ¹H spectrum, process, integrate and plot
- set up new dataset for ¹³C spectrum
- check ¹³C probe tuning
- run ¹³C spectrum, process and plot

Standard Sample - 100% CDCl₃

- remove test sample and replace with CDCl₃ standard sample, lock and shim
- initialize spectrometer to ¹H
- shutdown XWINNMR and logout from UNIX