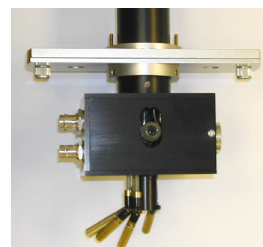
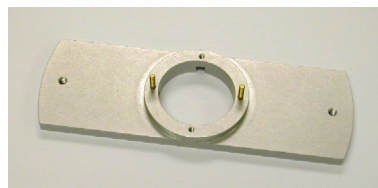


## Technical Bulletin J0140 – Vertical Probe Positioning and Pulse Width Calibration for Bruker Spectrometers

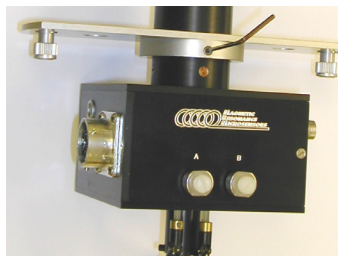
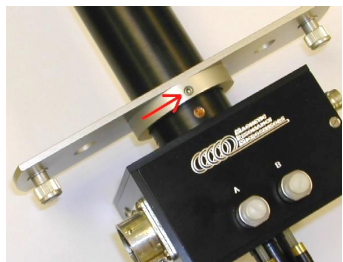
*This procedure was designed from methods developed on a Bruker Avance spectrometer with XWINNMR 2.0 or higher, and this procedure may vary if you have a different spectrometer or older software.*

### Mounting & Securing the probe:

There is an adapter plate for all Bruker CapNMR probes that screw into the shim stack *before* the probe is put into the magnet. This adapter plate screws into the shim stack as



any other Bruker probe mounting plate would. Then, position the probe into the magnet. The probe's mounting collar will attach to the Bruker adapter plate by two spring-loaded screws built into the mounting collar. Once the mounting collar is connected to the adapter plate, these are three 'set screws' around the base of the



mounting collar that set the vertical and rotational dimensions of the probe. The probe is delivered with a 5/64" beryllium-copper (non-magnetic) Allen wrench for tightening these three setscrews.

### Step 1) Setup the spectrometer

Load the standard proton single pulse experiment, or type edc, rpar select proton and [Copy All].

Type eda to view acquisition parameters and change the following settings:

- pulprog = zg
- ns = 1
- d1 = 1
- pl1 = 15 [Important\*]

\* The CapNMR probe has a much more sensitive coil than standard 5 mm probes. In order to protect the probe, do not use a power level (pl#) lower than 15 dB.

(For most Bruker 600 magnets, there is generally a little less than 1 cm between the top of the probe and the bottom of the mounting bracket holding the probe.) Inject your sample into the CapNMR probe. For further directions on sample injection, fluidic connections, and tubing, please see the 'Fluidic Tutorial' technical bulletins in the knowledge base at [www.microNMR.com](http://www.microNMR.com).

## Step 2) Tuning & Match the CapNMR Probe

Take an initial single scan spectrum to make sure the spectrum is set up correctly.

Click on [Wobb] to start up the tuning interface – (default window is 4ppm)

- Type acqu to view impedance dip
- Twist the knobs on the bottom of the CapNMR probe to tune and match
  - The knobs are labeled on the underside of the probe box.

If you have difficulty finding the impedance dip, you can widen the acqu window by:

Click on [Wobb-SW] for tuning interface that prompts you for window

- Do *not* change MHz (hit enter to continue)
- Enter a larger swept width (i.e. 20 ppm)

However, once you have the dip close, return the viewing window to 4 MHz using the same process to touch up the tuning and matching.

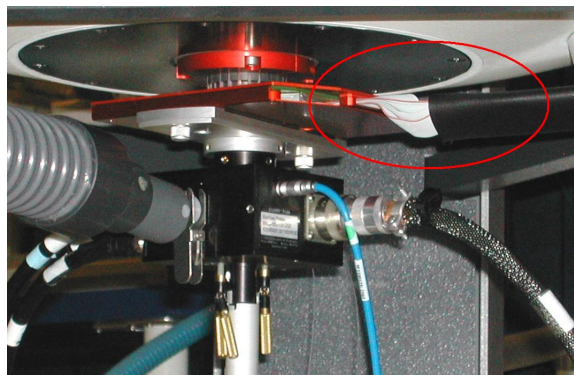
When properly tuned and matched, it is important to stop the acquisitions before returning to the standard window. Click [Stop] in the middle of left menu, and then click on [Return] at the bottom of the left menu. This will stop the WOBB and return you to the standard window with an idle system.

Take a single scan proton spectrum with a zg pulse program and set your cursors to the desired window and click on [SW-SFO1] to set the desired spectral window and proton transmitter offset frequency (o1p).

## Step 3) Lock Channel

The lock channel is just as sensitive as the proton so less lock power is required. Generally, a lock power of  $-55$  to  $-45$  is used (minimum =  $-60$ ). However, this allows you to turn the lock gain as high as you see fit to get a respectable lock signal.

Before testing the vertical position, ensure the X- and Y-axis of the probe are aligned axes of the shim stack. (The axes on the magnet do not matter.) Locate the shim cable coming off the shim stack (circled in red). This is generally the Y axis of the shims stack so the X axis would be perfectly perpendicular to that shim cable. Now that the X and Y axes of the shims stack are defined, align the sides of the probe to be parallel and perpendicular to those X & Y coordinates on the shim stack. It does not matter which side of the probe aligns with which coordinate as long as the probe box sides are parallel to one of the shim stack axes. This ensures that the horizontal solenoid coil is in a pure X or Y dimension of space and that will make shimming easier.



Once you have the field resonance set, balance the phase, and turn the lock on. Take a single scan spectrum and look for your solvent peak (this will most likely be quite poor, half height > 8 Hz). Change the scale to Hz with the [PPM/Hz] button and take careful notice of where the solvent is.

Turn the lock AND drift off. Then, select Z1 on the BSMS panel and increase Z1 by +7,000 points. Leave lock and drift off and take another spectrum. Look for how far the peak drifts from the previous value. (If there are strange artifacts in your baseline, make sure the drift is turned off.) If the peak drifted to the right (down field) after an increase in Z1, then the probe generally has to be moved up. However, if the peak drifted to the left (up field) after the Z1 increase, then the probe generally has to be moved down. Sometimes the shim polarity can be reversed and may need to be moved the other way so move the probe a few millimeters in one direction and make a judgment call from the difference in your next adjustment.

Before making another change, move Z1 back to its original value and turn drift back on (leave lock off) to confirm the field is still in range and the phase hasn't changed. If they have, optimize them. Take a single scan and take note of your solvent peak (it will have drifted since last time).

Next, turn the drift off and make another +7000 point adjustment to Z1. Leave lock and drift off and take another spectrum. The amount of drift should have decreased since you last moved the probe. If the difference of the drift increased, then the probe was moved the wrong way. The closer the probe is to its appropriate vertical position, the less the solvent peak will drift with a Z1 shim change.

Continue this process of:

- 1) Turn Z1 shim back to original value
- 2) Turn drift on and check phase (leaving lock off)
- 3) Take a single scan spectrum and note position of solvent peak
- 4) Turn drift off
- 5) Make a large change in Z1 shim
- 6) Take a single scan spectrum and note the how much the solvent peak drifted
- 7) Make a judgment call from the amount of drift as to which way to push/pull probe.

When the probe is properly positioned, the *solvent peak should drift less than 10 Hz* and should appear to change amplitude but not drift. At this time, ensure the X and Y axis of the probe is still appropriately aligned with the shim stack cable and then tighten the three shoulder screws in the mounting plate that anchor the X, Y and Z orientation of the probe in the magnet.

## **Proton Pulse Width Calibration**

*Using 5% CHCl<sub>3</sub> (chloroform-p6) in acetone-d<sub>6</sub>*

First, optimize a 1H parameter set (eda or ased) for the observation of the 5% chloroform (protonated) in acetone-d<sub>6</sub> line shape sample.

Second, type edc or iexpno to open a new experiment. In eda, select zg as your pulprog for a full 90 degree pulse. Save and close. Type ased in the command line to check your acquisition parameters. Start with an appropriate P1 (proton p90) of around 4 usec and ensure the PL1 (power level for the proton p90) is 15 dB or higher, and o1p is set to a standard window like 0-10 ppm). Take a single scan spectrum to ensure the experiment is properly setup.

Next, type paropt and XWINNMR will query you for parameters to the array it is about to setup. (Popt is another way of running an array and suggest using it if you are familiar with the function.) Enter p1 for the parameter to array, 20 for the numbers of experiments and 1 usec for the initial value and 1 usec for the increments on the arrayed parameter. The array should automatically start. Watch for the maximums, nulls, and minimums. First maximum = 90o / First Null = 180o / First minimum = 270o / Second null = 360o pulses

## **Carbon (or X Channel) Pulse Width Calibration**

*Using 5% CHCl<sub>3</sub> (chloroform-p6) in acetone-d<sub>6</sub>*

First, optimize a 1H parameters set from your proton pulse width calibration. Make sure SR = 0 before setting O1P. Type edasp and turn on your carbon (or X Channel) decoupler.

Second, type edc or iexpno to open another new experiment. In eda, select decp90 as your pulprog for a indirect 90 degree pulse. Save and close. Type ased in the command line to check your acquisition parameters. Set your P1 (proton p90) and PL1 (power level for the proton p90), and o1p from the values used/learned from your proton pulse width calibration. Set o2p to 77 ppm, CNST2 to 216, and if the remarks for automatic calculation in the pulse programs were not removed during XWINNMR install, set D2 to 3.34 ms. Set D1 to 10 s, DS = 0, NS = 1, and RG to (~ 265 or 512). Set P3 (Carbon 90o) to 0 (for a indirect 90o pulse) and PL2 (power level for the carbon p90) to 15 dB. (Just like the proton power level, the carbon power level, p13, should not be set to anything lower then 15 dB.) Take one scan and process. Phase, one satellite should be up, the other down.

Next, type paropt and XWINNMR will query you for parameters to the array it is about to setup. (Popt is another way of running an array and suggest using it if you are familiar with the function.) Enter p3 for the parameter to array, 20 for the numbers of experiments and 1 usec for the initial value and 1 usec for the increments on the arrayed parameter. The array should automatically start. The 13C p90 is where the satellites cancel, and when you exceeded p90, the satellites will flip. Since experiment is indirect

detection, the nutation follows the current pattern. First maximum =  $0^\circ$  / First Null =  $90^\circ$   
/ First minimum =  $180^\circ$  / Second null =  $270^\circ$  pulses