Conditioning a Cold Probe

After a cold probe is cooled or if no experiments with 13C decoupling have been performed for several weeks it will be necessary to condition the probe. Failure to do so will lead to "decoupling noise" in your spectra. This noise is due to frozen air out gassing from the probe coil as high amounts of power are applied for 13C decoupling.

There are two things that need to be done in order to condition the probe.

1. Condition the probe by applying a large amount of power to the probe, but not too much to induce arcing.
2. Testing the probe to be sure that there is no "decoupling noise" present.

The above two items are identical except when conditioning the probe you may use an nt > 1 and for testing nt = 1.

Follow these instructions to condition the probe:

1. Load a gChsqc experiment
2. Set d1=1, at=0.1, pw=0, dres=1, dseq='garp1', ss=0, nt=1 (testing) or nt=user defined (conditioning)
3. Find the pwCref90 by multiplying pwC x compC.
4. Calculate the B1 field needed for decoupling.
    a. We need enough power to decouple 170 ppm for conditioning and garp1 decouples ~ 5X the bandwidth of a hard pulse. Thus we need enough power to achieve an excitation bandwidth of 170/5 x 13C freq. For example at 500 MHz we need a B1 field = 170/5 x 125 = 4250.
5. Calculate the power needed to achieve the B1 field calculated above using the attval command. To use attval enter the following onto the VNMRJ command line "attval(pwCref90, pwClvl)" substituting the appropriate values for pwCref90 calculated above and the pwClvl normally used for hard 13C pulses.
    a. The output from the attval command will be reported in the text output window. Scroll through the list and find what attenuator value will give a B1 (Hz) value closest to the B1 field determined earlier.

        i. Typical values are 46-48.
        ii. Set dpwr = value from attval
        iii. Set dmf = 1/90 deg pulse where the 90 deg pulse if from the attval output.
6. Setup an array of 10,000 steps by arraying d1 with a starting value of 1 and a step size of zero (thus all 10,000 will have d1=1).
    a. Note: Feel free to change the 10,000 steps to any value if you want to change the length of time of the test or conditioning, there is nothing magical about 10,000.
        i. With 10,000 steps the experiment time should be around 3 hours for each nt value.
7. For a test set nt=1 and type go. For conditioning set nt=user defined to adjust the time of the conditioning that you want. Typically an overnight run may be needed after a probe change, but a few hours should suffice if the probe has been cold for a while.
    a. Another idea is to run several 3 hour tests overnight in different jobs.
8. Now that the probe is conditioned we want to check that there is no “decoupling noise”. Make sure that you ran a conditioning experiment with nt=1
9. Issue the command “ft(‘noft’)”
10. Issue the command ff to see the full spectrum
11. Use the vertical cursors and the zoom button to zoom into the FID region so that no zeros from the zerofill are visible on the screen.
12. Issue the command “et_noise” from the VNMRJ command line
    a. This calculates the noise in each FID and reports the results to the Text Output window. It also displays each FID to the screen so you can get a quick visual analysis to determine if there is a lot of noise spikes in the FID’s.
        i. If this does not work properly make sure the celem value is equal to the array size and re-run et_noise.
13. Copy the content of the et_noise output to a text file
    a. Open a terminal and issue the command “vi filename” (filename can be whatever you want)
    b. Hit i to go to insert mode

    c. Go to the vnmrj window and place the cursor at the top left position of the output where it says “noise:spec …”
    d. Scroll to the bottom and click the mouse at the bottom right of the output while holding the shift key.
        i. At this point all the results should be highlighted
    e. Go back to the vi editor and click the middle mouse button. That will paste the results into the editor
    f. Hit the esc button to exit insert mode
    g. Hold shift and hit ZZ to save and exit the vi editor
14. From the terminal window type “gnuplot”
    a. Enter the following command into gnuplot to create a plot of FID noise versus FID number
    b. “plot ‘filename’ using 3:5”
15. A plot should be shown. If the plot has lots of scatter and the noise values are all around 1 then everything is fine. If some of the FID’s have noise values significantly higher than the baseline values than there is still decoupling noise and more conditioning is likely needed.
    a. Note that if the decoupling noise is found in the early FIDs and then was clean for the remainder of the FIDs everything may be fine.