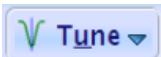
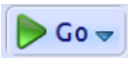


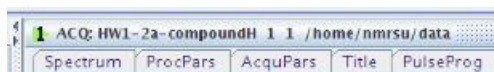
Acquiring ^{29}Si 1D Experiments on the Bruker NMR

by Monika Ivancic, PhD (October, 2020)

1. Start by acquiring a ^1H 1D experiment (uvm.1H1D) (see Bruker Speedy setup guide) Even if you may not need the data, a ^1H 1D will be the best indicator of shim quality. Also, one can reference X-nuclei spectra from a properly referenced ^1H spectrum that was acquired under identical lock conditions (so same lock and shim), which can only be true in the same session. You may use NS = 8, which will take less time.
2. Create a new dataset (type 'new' to get GUI up), set experiment number to 2, then for experiment, choose 'uvm.29Si' (no ^1H decoupling) OR 'uvm.29Si.dec1H' (with ^1H decoupling), depending on whether or not you want the ^1H s decoupled. You may acquire both versions and compare the differences.
3. Tune the probe to ^{29}Si by typing 'atma' or clicking . The system will tune ^{29}Si at about 99.36MHz. It may or may not retune ^1H after. Please be patient, it's important for the process to finish.
4. ^{29}Si is not very sensitive, so most likely you don't need to optimize gain. Typically for ^{29}Si , RG = 203 (highest value possible).
5. Acquire your data by typing 'zg' or clicking . This is a 32 scan experiment, although your compound may need more scans. Once acquisition is complete, type 'efp' followed by 'apk'. Because NMR tubes are made with glass and glass contains Silicon, there will be a large, broad ^{29}Si glass peak in the upfield region of your spectrum (at about -114ppm). If your compound peaks lie in the same or similar region, they should be much sharper than the glass peak, and typically can be distinguished.

The default experiment uses a large sweep width (SW = 399.4ppm) and is centered at O1 = 39.5ppm. You may find it important to reduce this SW, in case your peak has finer coupling that wouldn't be picked up with this large SW.

6. You are going to keep the data that you acquired in 5. Copy the dataset (includes parameters AND data) into another experiment by typing 'wra 3' (copy into experiment 3) and then you need to go into that experiment by typing 're 3'. At this point it will look identical, but above your data and tabs, you will see what experiment number you're in.




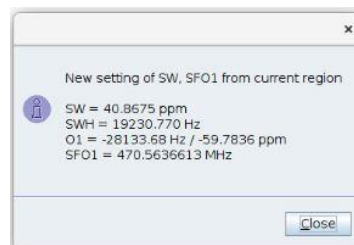
Here is an example of being in exp 1:

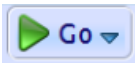
7. Adjust the sweep width to be about 100ppm surrounding your peak. If you have more than one peak, make sure the new sweep width includes all the peaks with enough baseline (~10ppm or more) to the left of the leftmost and to the right of the rightmost

peaks. Using the mouse, left-click at the starting point (and hold) until the end point. The zoomed in spectrum will appear when you release.

Be careful here, since SW and AQ (acquisition time) are inversely proportional to each other. When reducing SW to 100ppm, AQ = 3.3sec, however if you reduce SW even more, say 50ppm, AQ = 6.6 sec. Your acquisition time will increase with decreasing SW, and the experiment takes longer to acquire. If AQ ends up large, you may always reduce it (to 2-3 sec) after the SW/O1 adjustment.

8. Then click the  icon, which adjusts the SW and O1 parameters. You will get a message like this:



9. Re-acquire the data with 'zg' or , this data will have the new SW and O1. Then process with 'efp' and use 'apk' to phase data.

10. If you followed the above instructions, you will have 3 datasets:

Exp 1 – a ^1H 1D

Exp 2 – a ^{29}Si with a large SW

Exp 3 – a ^{29}Si with the smaller adjusted SW

11. If you acquired a 'uvm.29Si' in the above instructions, you may repeat with a 'uvm.29Si.dec1H'. By comparing the two, you will be able to determine whether or not ^1H s are splitting the ^{29}Si peak.