

Pre-Saturation of Two Peaks – Double Solvent Suppression

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You should be familiar with the solvent suppression (pre-saturation) experiment before proceeding.

To eliminate two peaks in a proton spectrum (typically solvent peaks), a double pre-saturation method is employed. This technique utilizes two channels of the NMR console.

Protocol

This description uses THF- d_8 as a solvent example with its two peaks present at 1.73 ppm and 3.58 ppm. To suppress (pre-saturate) both peaks:

- (1) Make sure the decoupler nucleus is set to hydrogen (**dn = 'H1'**), then collect a normal proton spectrum. Next, find the dof values of the two solvent peaks:
 - a. Set the cursor at one peak position (e.g. 1.73 ppm), type **nl sd** and a dof offset value is given on the command line; record this dof value (probably by writing it down on paper).
 - b. Set the cursor at the other peak position (e.g. 3.58 ppm), type **nl sd**, and again a dof offset value is given on the command line; record this dof value (probably by writing it down on paper).
- (2) Move the spectrum to another window by typing **jexp2** then **mf(1,2)** (assuming the normal proton experiment was collected in **exp1**). In the new experimental window, type **wft**, then **presat** to load the presat experiment. Look carefully at the satfrq value listed in the presat table and confirm that it is set to the last dof value found previously. If satfrq is not set equal to the last (2nd) dof value, set it now.
 - a. Next, set the corresponding satpwr and satdly valves by typing **satpwr= 0** (or a number up to 10) and set **satdly = 1** (or a number up to 5) depending on the intensity of the solvent peaks. You may need to adjust these values by trial and error based on the residual solvent signal.
 - b. Again, be sure **dn='H1'**, set **dof = the other dof offset value found previously**. This solvent peak is about to be suppressed (presaturated) by channel 2 of the NMR console. Type **dm='ynn' dmm='ccc'** and **dpwr=10** (or a number up to 20), and **dmf=200**.
 - c. **Note:** Pay attention to the power used (**satpwr** and **dpwr**) in the pre-saturation. **DO NOT** exceed the values mentioned above, or the probe can be permanently damaged. **Repair could cost your group \$3000, and weeks of down time.**
- (3) Collect a proton spectrum. This proton spectrum should have both solvent peaks suppressed. It is normal for the residual peaks to look like “glitches” and not phase properly.

In the future, we'll have the ability to use excitation sculpting sequences to pre-saturate multiple peaks. Please keep in touch with the staff for developments.