


Running on samples in protonated solvents in ICON-NMR

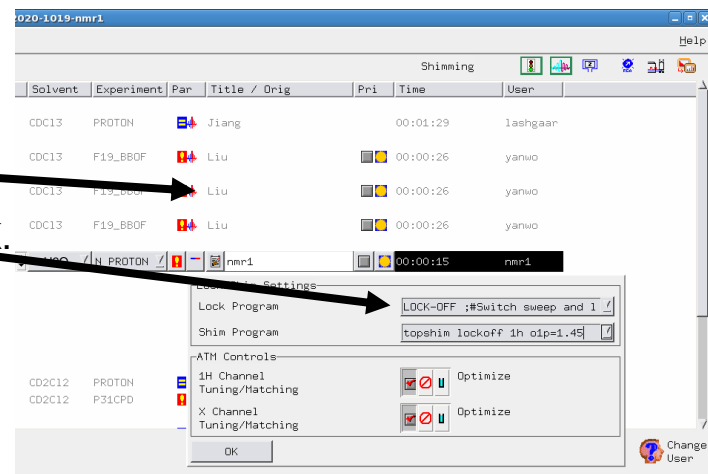
1) Select your solvent from the drop-down list. Take note of whether it's the H or D version!

2) Switch off lock. (ONLY NECESSARY FOR AV400)

- Click on the lock/shim options icon:  (see me if your username doesn't have access to this feature)
- Select "LOCK-OFF" from the Lock Program drop-down. Press OK.
- DO NOT change anything under ATM Controls!

3) Select "PROTON_1HSOLV, C13CPD_1HSOLV, or C13CPD32_1HSOLV" instead of the normal experiments. Normal ^{31}P and ^{19}F -detect experiments should also be fine. See NMR manager to discuss options for ^1H - ^{13}C or ^1H - ^1H 2D if desired.

4) After your spectrum runs, you will need to reference on the (suppressed, for ^1H) solvent peak or TMS if you have included it. Proton experiments will often need to be manually phased as well.

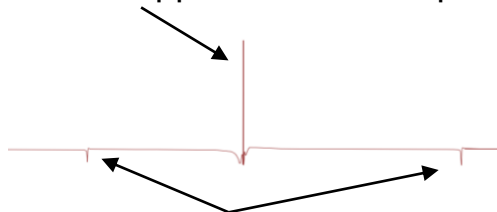


WARNING: Running without a deuterium lock may result in undesired artifacts in your spectrum! Likewise, solvent suppression will suppress any peaks of interest near the solvent peak in addition to the solvent peak!

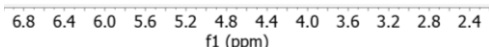
Processing spectra with suppressed solvent peaks

Autophasing routines may prioritize your residual solvent signal! In that case, you will need to do manual phasing.

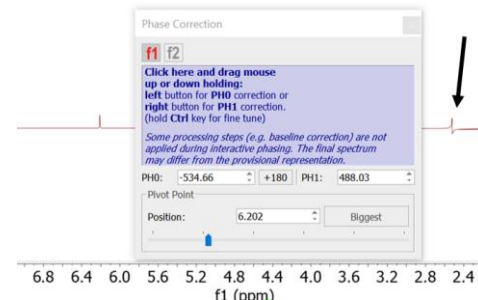
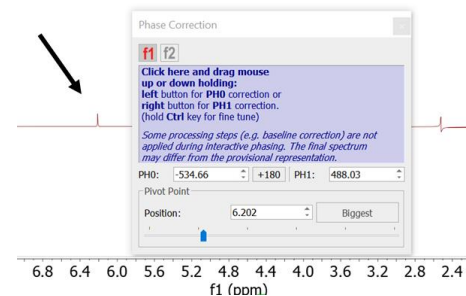
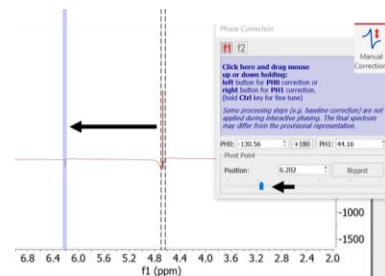
“phased” suppressed solvent peak



out-of-phase signal of interest

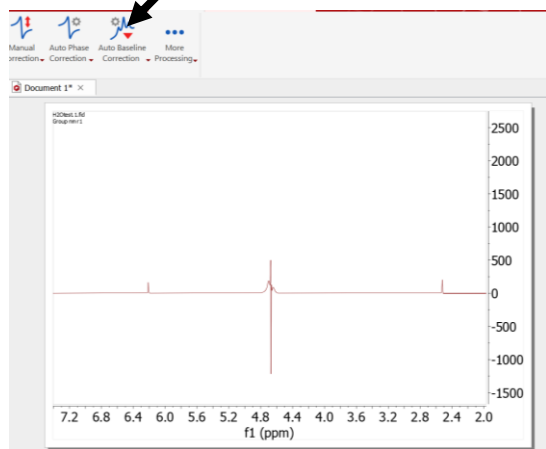


- 1) Click on “Manual Correction” under “Processing.” Use the slider to move the purple bar from the solvent peak to one of your peaks of interest
- 2) Holding your left mouse button in the purple window, drag the mouse until the peak you selected is in-phase (positive and symmetric). This is “0th order” phasing because it applies to all peaks equally.
- 3) Holding your right mouse button in the purple window, drag the mouse until a (non-solvent) peak far from the earlier peak is also in-phase. This is “1st order” phase correction which is linearly dependent on frequency.

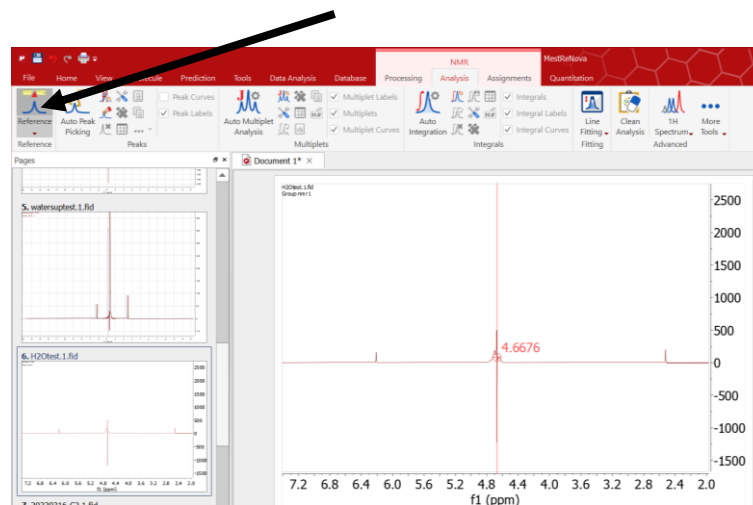


Processing spectra with suppressed solvent peaks

- 4) Baseline Correct: Click on “Auto Baseline Correction” under “Processing” to adjust your baseline, which the solvent peak may have disrupted.



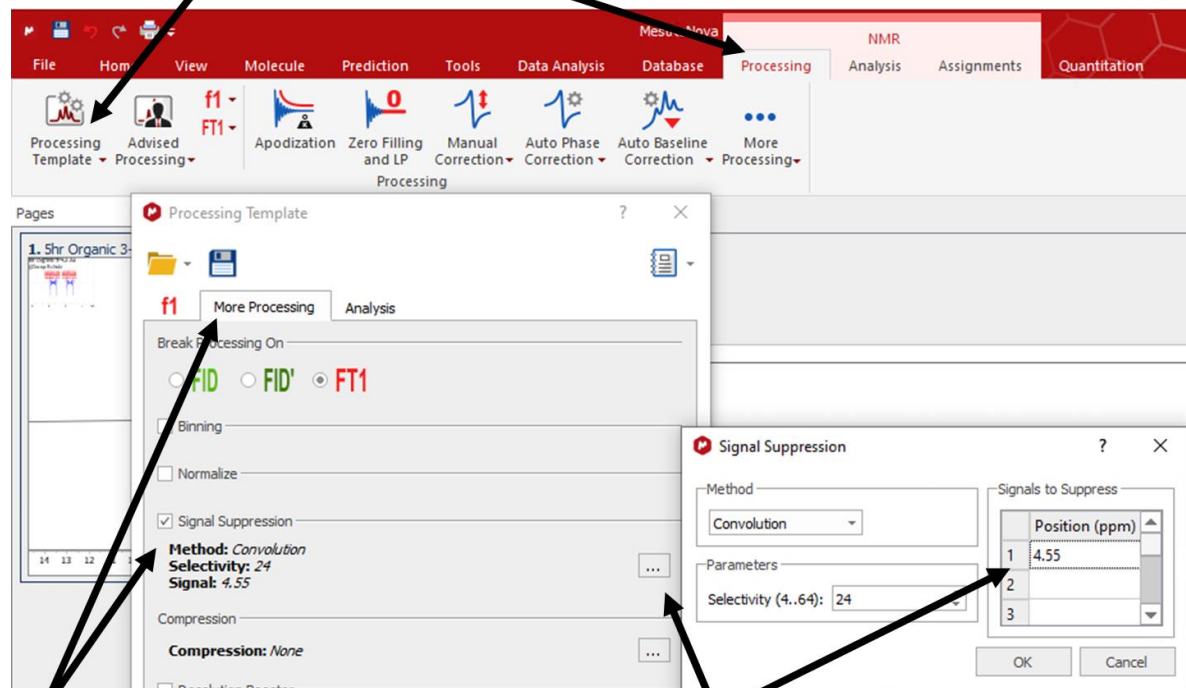
- 5) Reference: Click on “Reference” under “Analysis” and click the sharp part of your (suppressed) solvent peak. Set it equal to the known shift of your solvent. Note that protonated solvents can have slightly distinct shifts from their deuterated versions!



Suppressing protonated solvent peaks in MNova

If desired, solvent peaks can be removed by signal suppression in MNova. This is mostly “cosmetic” and other signals near the solvent peak will also be removed.

- 1) Processing tab
- 2) Processing template



- 3) “More processing”/ Select “Signal Suppression”

- 4) Open up options, confirm there is an entry near solvent peak

Referencing unlocked spectra

If you do not have a good solvent peak to reference on (say, for a ^{19}F or ^{31}P experiment), you should indirectly reference from a corresponding ^1H spectrum:

- 1) Submit a PROTON_1HSOLV experiment along with your X-detect experiment. These need to run back-to-back!
- 2) Reference the ^1H spectrum on the solvent or ideally TMS, noting the chemical shift change upon referencing (for example, $\Delta\text{ppm} = +0.3 \text{ ppm}$).
- 3) Reference a peak in the X-detect experiment by changing it by the same Δppm as for the ^1H referencing.



/opt/nmrdata/nmr	1HSOLV_referencetest	3	H2O	Water, non-de	N PROTON_1HSOLV	★
/opt/nmrdata/nmr	1HSOLV_referencetest	4	H2O	Water, non-de	N C13CPD32_1HSOLV	★

