Running on samples in protonated solvents in ICON-NMR

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

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 account 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) Run a PROTON spectrum of the solvent for referencing purposes.

4) If you need a 1H spectrum, select **PROTON_1HSOLV/WaterSup1**. For 13C, select **C13CPD_1HSOLV**, or **C13CPD32_1HSOLV**. Normal 31P and 19F-detect experiments should be fine. See NMR manager to discuss options for 1H-13C or 1H-1H 2D if desired.

5) After your spectrum runs, you will need to reference on the solvent peak or on TMS if you have included it. Proton experiments will often need to be manually phased as well. (see subsequent pages).
Processing spectra with suppressed solvent peaks: Phasing

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

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.
Baseline Correction

1) Increase the vertical scale until the baseline is clearly visible.

2) Press \textbf{b} or click on the red arrow on the bottom of the \textbf{Auto Baseline Correction} icon under Processing and select \textbf{Baseline Correction}… Select different methods under \textbf{Method} and look at the blue baseline that has appeared along the bottom of the spectrum.

3) Adjust baseline parameters and/or method until the blue line matches the experimental baseline without under- or over-shooting anywhere. Click \textbf{OK}.
Referencing

1) Load all spectra collected for your sample into an Mnova document. It should all have been collected at the same time. Ideally, you will have run a PROTON experiment at the time that the rest of your data was acquired. If you included TMS or DSS this is even better.

2) With the PROTON spectrum shown, click on Reference under Analysis. Select your TMS/DSS peak if available and set to 0. Otherwise, select your solvent peak and set it to its expected shift.

3) Select Absolute Reference under Reference and select the now-referenced 1H spectrum in the drop-down for Use as Reference.

4) Check the rest of the spectra that correspond to the sample and click OK. This will reference the other spectra.
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