NMR Tips - Solvent Signal Suppression

Sometimes it is unavoidable to use extremely low sample concentrations because of a compound's poor solubility or low sample availability. Biomolecules may require using a protonated solvent, i.e. 90% H_2O + 10% D_2O to minimize deuterium exchange of OH or NH hydrogens.
All these situations will lead to one or more overwhelmingly tall solvent signals.

Please always record a routine proton spectrum first to avoid missing important data. If needed, I recommend trying one of the following solvent signal suppression techniques depending on a variety of circumstances. All are available through our spectrometers' IconNMR automation:

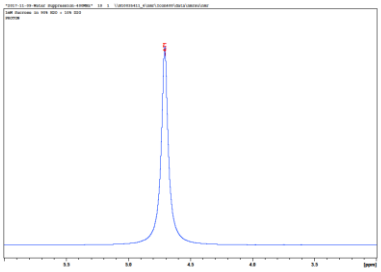
1. Suppression of one single signal

Option A: noesygppr1d (suppresses H_2O in H_2O/D_2O)

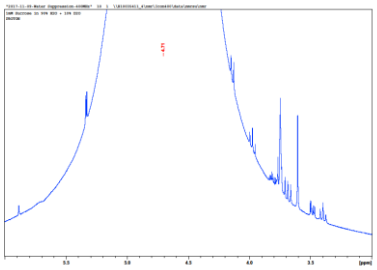
This technique is optimized for samples in H_2O/D_2O mixtures and aq. buffer solutions.
The solvent signal position is irradiated for a few seconds ("pre-saturated") to equalize its α and β spin populations. This will make the water hydrogens invisible to the NMR and will allow recording a clean ^1H spectrum of your molecule. Baseline correction is included.

Caution: OH and NH signals will also disappear!

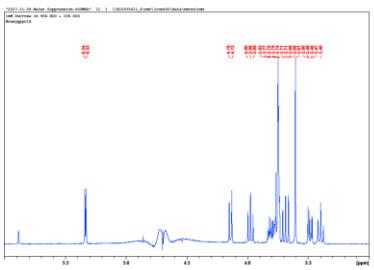
Test sample: 1 mM sucrose in 90% H_2O + 10% D_2O



no solvent suppression



no solvent suppression
1000 X vertical magnification



with solvent suppression

Option B: WATER (suppresses H_2O in organic solvents)

This technique can be used for samples in any deuterated solvent.
The experiment will first search for the tallest peak in the spectrum before suppressing it using the same technique as Option A. Baseline correction must be performed separately.

Caution: The tallest signal may not always be your solvent signal!
Verify with a separate regular proton experiment.

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2. Suppressing multiple solvent signals

These techniques work best for the suppression of two tall organic solvent signals.
They were developed for Liquid Chromatography samples which often contain residual LC solvents and can be adjusted to eliminate more than two signals.
They may also help when you have dilute wet samples, i.e. DMSO-d_6 + H_2O.
(Remember: All NMR solvents are hygroscopic and old solvent bottles may be contaminated.)

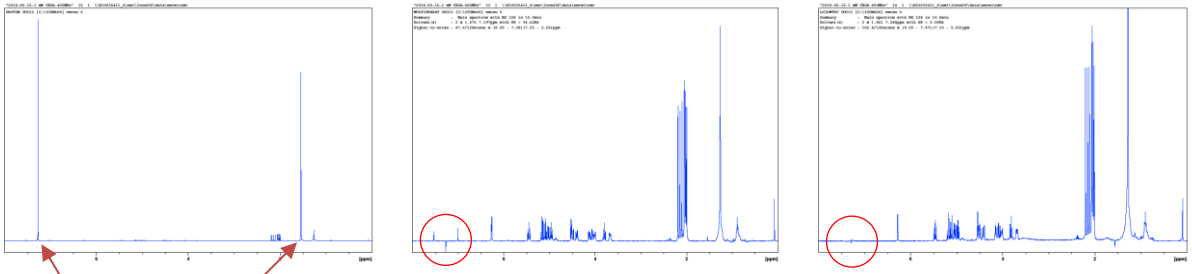
Option A: MULTIPRESAT (use with any NMR solvent)

This technique will first search for the **two** tallest proton signals, pre-saturate them and record a solvent signal free proton spectrum.

Option B: lc1dwtdc (use with any NMR solvent)

The "WET" technique will first search for the **two** tallest proton signals, selectively excite them, and then de-phase their magnetization by applying gradient pulses.
All other hydrogen atoms will remain unaffected and can be measured by a detection pulse.
Additional carbon decoupling will remove the ^{13}C satellite peaks of the solvent(s) which may be taller than the signals of interest in micromolar solutions.

Test Sample: 1 mM cellobiose octaacetate in $CDCl_3$



without solvent suppression
$CHCl_3$ and H_2O signals
dominate the spectrum.

solvent suppression with
MULTIPRESAT.
Note the ^{13}C satellite peaks.

solvent suppression WET with
additional ^{13}C decoupling also
removes $CHCl_3$ satellite peaks.

Caution: Only use these techniques if you **know** you have **two** solvent signals!
Otherwise, the spectrometer will simply select the two tallest peaks in the spectrum.

Additional Considerations:
Some of the resulting automation spectra may have a distorted phase and/or baseline.
These distortions can be corrected with minor additional processing effort. (See the next page.)

Please contact me for further advice on making your spectra look more presentable.

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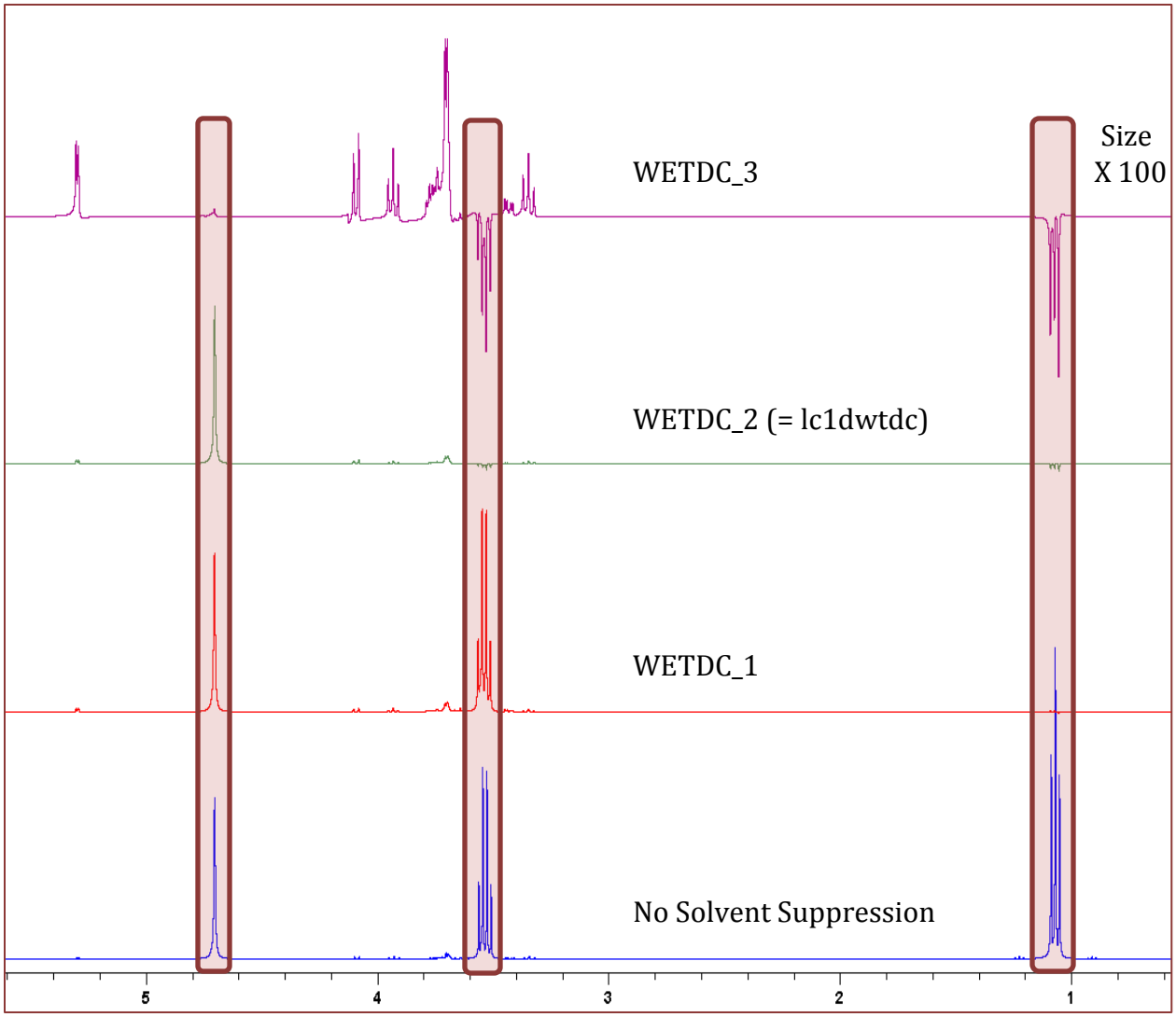
3. NEW: Select how many solvent signals you want to suppress!

With the techniques WETDC_1, WETDC_2, and WETDC_3 you can automatically suppress up to three solvent signals. This is especially useful if you have impurities of ethanol, ethyl acetate or DMF in your sample or if you are using solvent mixtures:

Test Sample: 1 mM Sucrose + 100 mM Ethanol in D2O



The top spectrum has been magnified by a factor of 100
to show the efficient suppression of all three ethanol ^{1}H signals.

Caution:

1. The NMR will select the tallest peaks in the spectrum. Make sure they really are solvent peaks.
2. Signals close to the solvent peaks will also be reduced in size or disappear.

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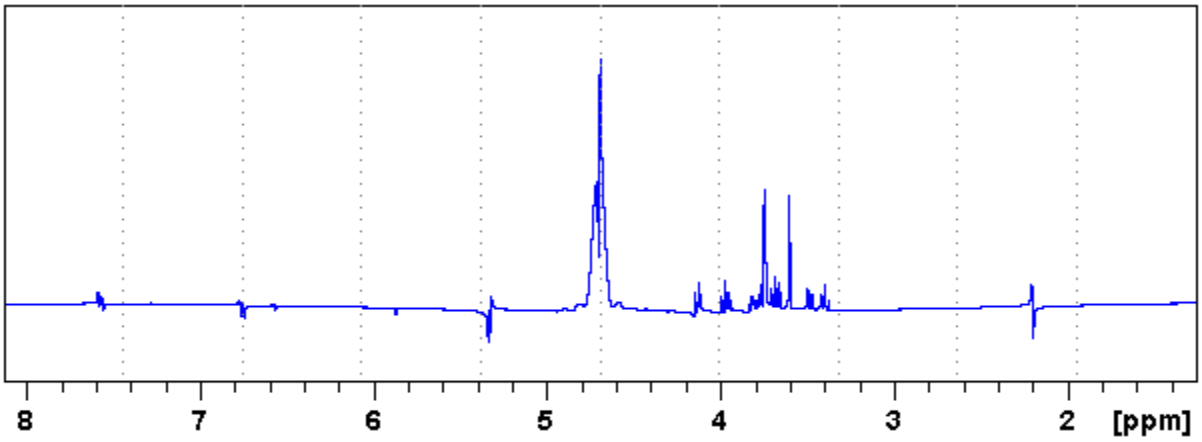
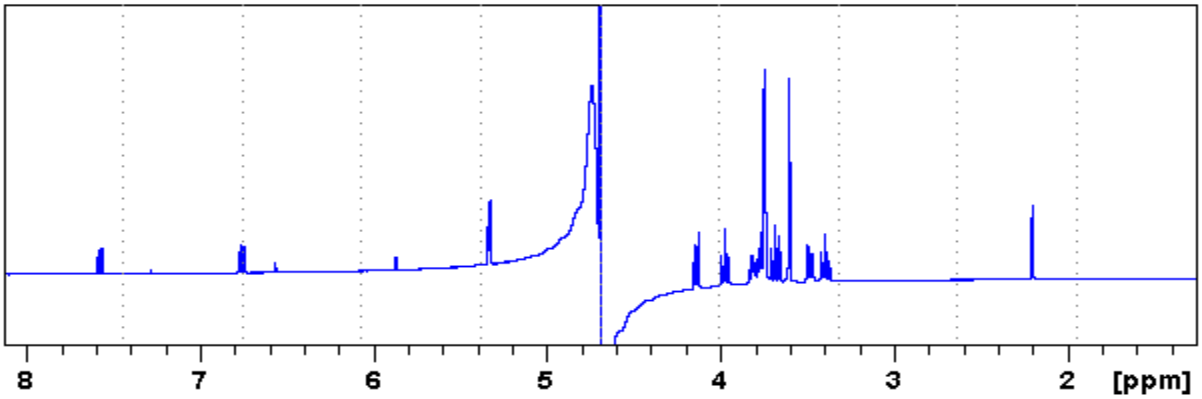
4. Minimizing distortions in Solvent Suppression Spectra

Often the residual solvent signal will distort the baseline and may make it difficult to correctly phase and integrate the spectrum:

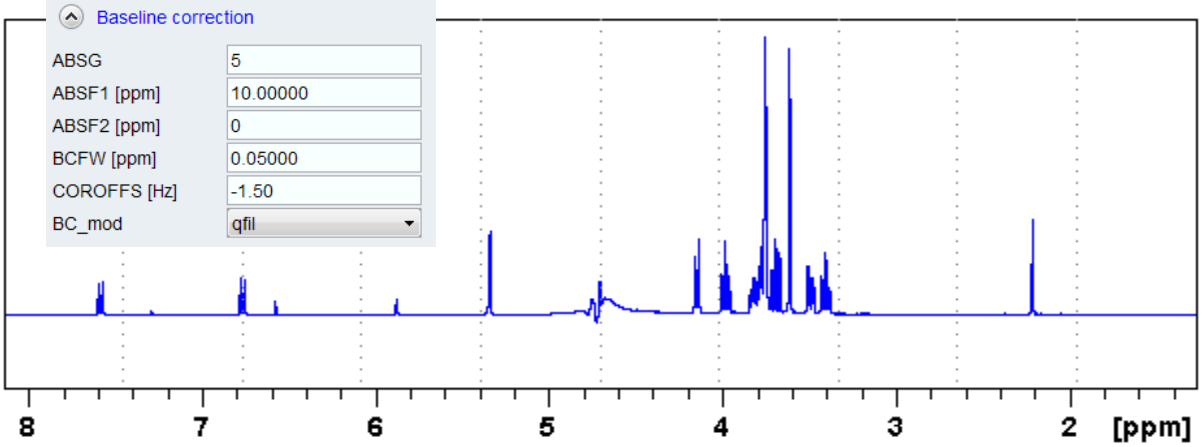


The solution is to apply a filter to the solvent signal:

Step 1: Enter the command "**EF**" and correct the phase of your signals. Ignore the solvent!



Step 2: Set the following parameters in the ProcPars section, then type "**EFP**":



Adjust the **COROFFS** parameter, type "**EFP**", and repeat until the spectrum looks best. Finish with "ABS" or "BAS" to correct the baseline.

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