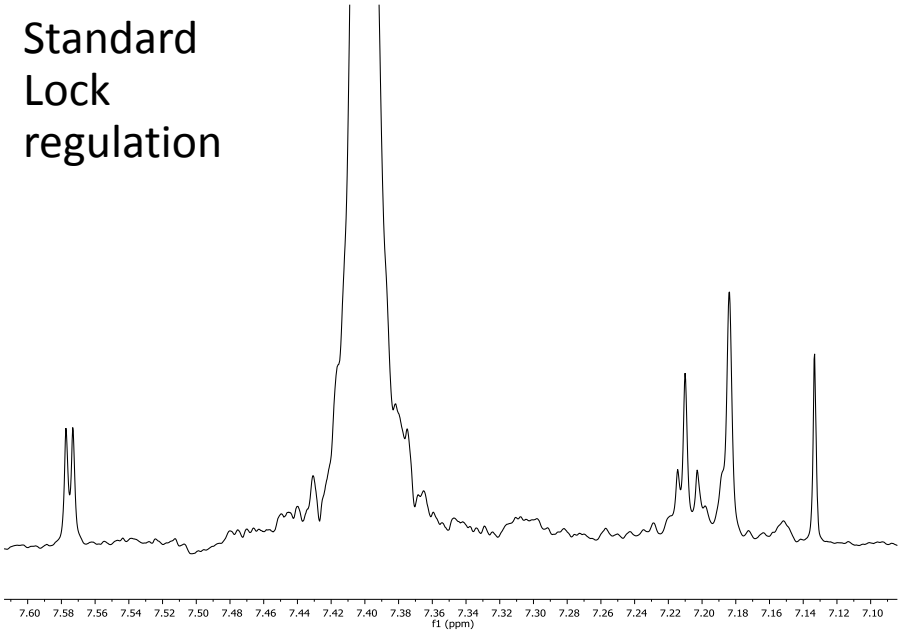
Experiment Setup for ^{2}H-free samples

Two options for running experiments on samples prepared in protio-solvents: (1) add a capillary with lock-solvent, or (2) run without lock.

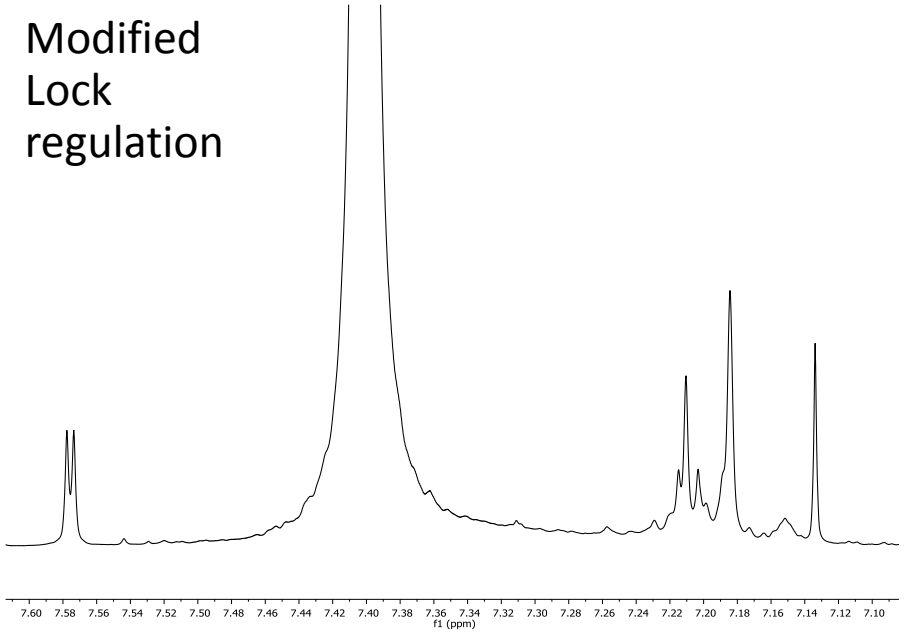
(1) Setting lock parameters for use with capillary insert.

Problem: lock parameters are set in the system based on the strength of the lock signal, if a capillary is used there are likely to be problems.

Standard Lock regulation



Modified Lock regulation



Solution: In Topspin, lock to the solvent in the capillary and run the *loopadj* command which optimises the lock regulation parameters. Save as a new solvent, e.g. c6d6_capillary.

If *loopadj* fails due to insufficient signal intensity, copy the parent solvent to a new solvent and modify the values manually – typically change loop time by -21, loop time by +4 and reduce the loop filter to about 21.

C_6D_6 → C_6D_6-capillary, loop gain 0 → -21, loop time 0.2 → 0.6, loop filter 300 → 20.

DMSO → DMSO-capillary, loop gain 5 → -17, loop time 0.25 → 0.65, loop filter 500 → 22.

Experiment Setup for ^{2}H-free samples

Two options for running experiments on samples prepared in protio-solvents: (1) add a capillary with lock-solvent, or (2) run without lock.

(2) Setting the automation to shim using the biggest ^{1}H solvent signal.

Problem: the system is setup to lock to a deuterium signal by default, and then shim using that signal, which is not possible if there is no deuterium in the sample.

Solution:

In the ICON NMR configuration select the solvent 'None' in Lock/Shim Options → Solvent/Probe Dependencies.

Set *Associated Shim Routine* to *XAU topshimfindsolvent2* (AU program from Bruker)
Set *Associated Lock Routine* to *LOCK-OFF*

What it does: The system does not try to lock to a deuterium signal. It then runs a ^{1}H spectrum, finds the biggest signal and uses that signal for 1D gradient shimming.

What it doesn't do: Optimise X-Y shims, lock (so there is no compensation for magnet drift or other magnetic field-related issues)

Tested on AVII+, AVIII running TS3.5, TS3.6