Deuterium ^{2}H-NMR in the SCS NMR Lab – UI500NB Only

On the UI500NB Using Lock Channel (146 RAL)

A ^{2}H-NMR spectrum can be collected in two ways: use the lock channel, but it yields lower sensitivity; treat deuterium as a normal X-nucleus and collect the ^{2}H NMR spectrum on a broadband channel. Using the lock channel is quick and easy and typically recommended for most deuterium NMR experiments. However, if your sample concentration is low, performing the experiment on the UI600 using the AutoX probe is recommended.

Important Considerations

(1) Sample should be prepared in a non-deuterated solvent to avoid overflow of solvent signals. The ^{2}H isotopic peak of the non-deuterated solvent can often be observed and used as a reference signal.
(2) The natural abundance ^{2}H signals of the solvent are observable.
(3) The ^{2}H chemical shifts are equivalent to the ^{1}H chemical shifts.
(4) Deuterium NMR data are collected without locking (no lock signal possible). One strategy is to prepare a sample with a 90% protonated with 10% deuterated solvent (no compound needed), shim on this sample, then insert the real sample using the shimmed values just obtained. A better method is to perform ^{1}H gradient shimming on the sample (see instructions below). The only other option is to shim in FID mode where you observe shimming progress by monitoring the shape and appearance of the FID.

Using the lock channel on UI500NB spectrometer

(1) logon to UI500NB as usual
(2) The default probe on UI500NB is the 'hcn' probe, you can perform H1 gradient shimming on your sample as described on page 3 of the larger deuterium document.
    a. Another option is to simply bring a deuterated solvent blank, shim on that first, then replace the blank with your protonated solvent sample, and proceed to acquire.
(3) After shimming is done, turn the LOCK "OFF" in acqi window
(4) Re-cable the lock cable as follows (Figs. 1 and 5):

(a) Locate the lock cable labeled 'lock' (red tape). One end of the lock cable connects to the large, cylindrical H-2 lock filter (~ 1 foot long) located between the tan box and the Preamp stand (with the Tune Interface). The other end of the lock cable goes to the lock port on the probe.

(b) The top end of the cylindrical H-2 filter connects to a short cable on the side of the tan box (Fig 4). Disconnect this short cable from the top of the H-2 filter.

(c) Find a spare cable about 2 feet long on the floor next to the magnet leg.

(d) Connect this new cable to the top of the H-2 cylindrical filter.

(e) Connect the other end of this new cable to filter attached to port J5311 of the broadband side of the Preamp stand (the one with the Tune Interface panel in the middle; Fig 1). **BUT -**

<u>please note</u>, you need to disconnect the cable that is already there <u>and</u> the 'H-2 reject filter' which connects to the low-pass filter, which stays in place on port J5311. See Fig. 2.

To summarize: Connect the new, short cable to the lowpass filter already connected to J5311.

(h) This setup allows the observation of H-2 or lk signals through lock channel (lock coil).

(5) After setting up a normal proton spectrum, change all of these:

a. **tn='lk'** or **tn='H2' (either one works).**

b. **tpwr=45**

c. **pw=100**, nt=16 or more. Then, collect your H-2 spectrum.

When you are done, put everything back to its original configuration (Fig 4).

The ppm scale will be strange unless you change it. If you run with some $CDCl_3$ present, you can set that deuterium reference peak to 7.26 ppm. Otherwise, for other solvents, set the D to the H value for that particular solvent.

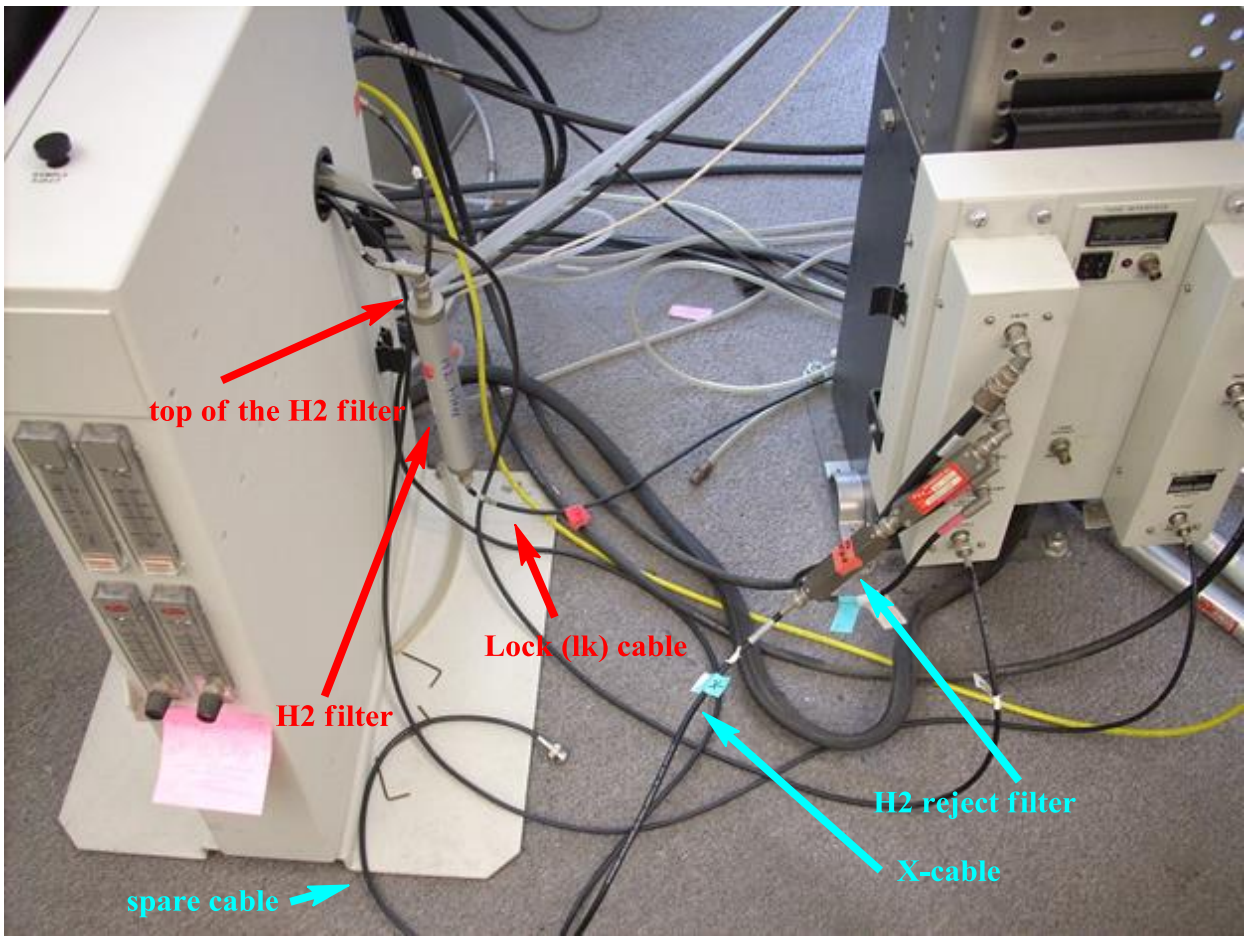

Fig 1. Original cable configuration of the UI500NB.

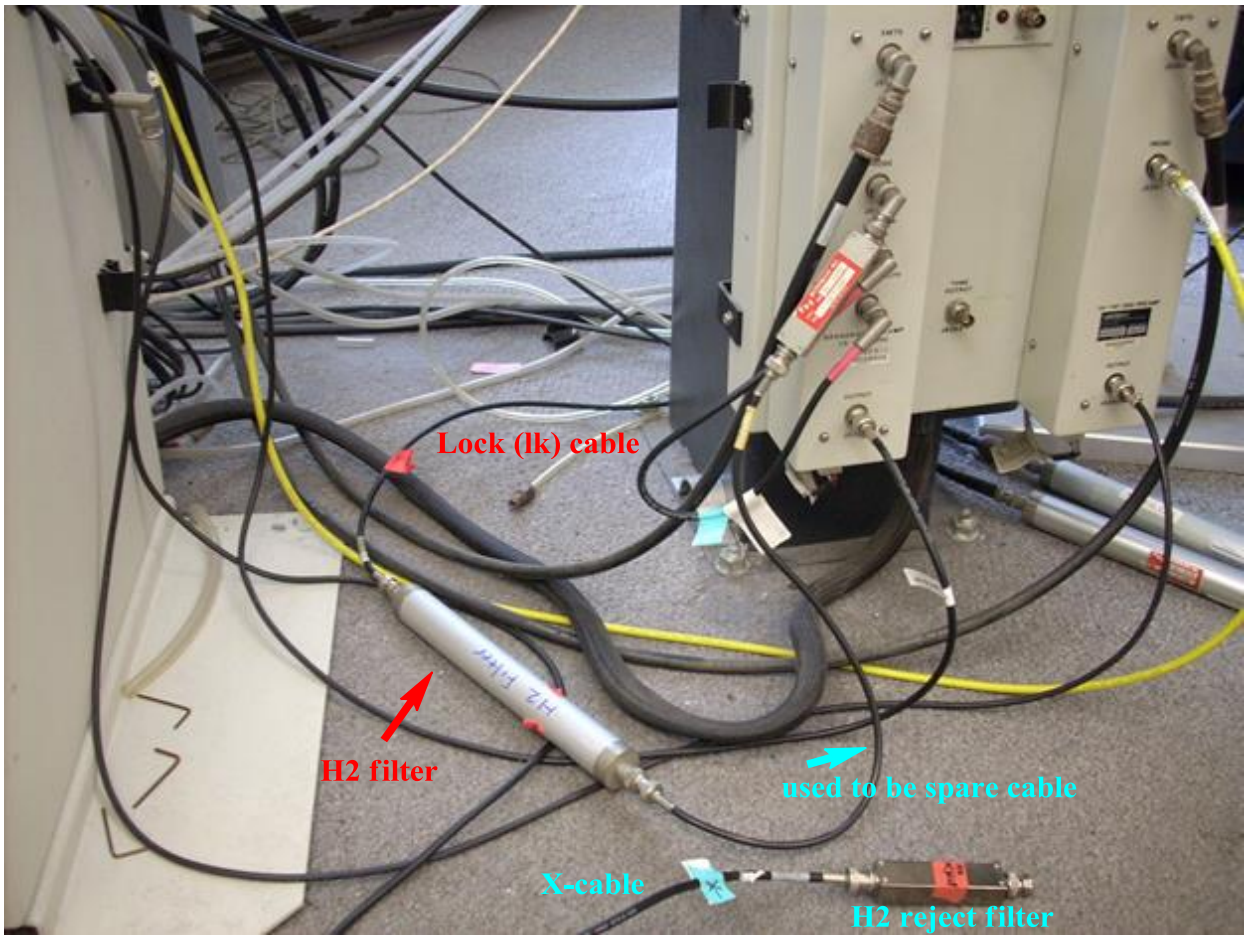

Fig 2. Configuration for deuterium cabling NMR on the UI500NB.