Fluorine experiments (Vnmrj 2.3A)

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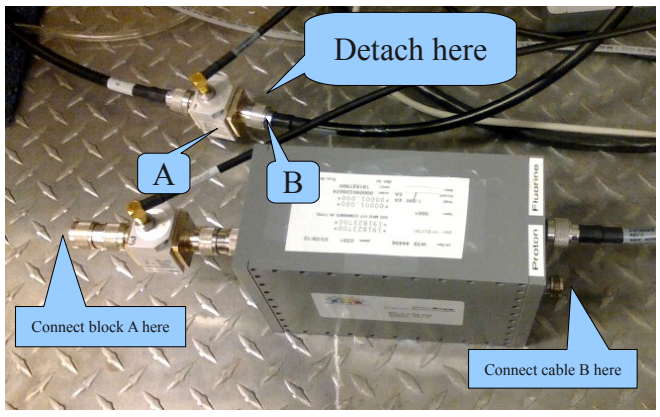
H{F}, F{H}, FH-HOESY, FH-HETCOR and C{F,H}

Experiments that require simultaneous pulsing at the proton and fluorine frequencies require a spectrometer with two high frequency channels and a probe that can be doubly tuned to ^{1}H and ^{19}F. The experiments are: proton detection with fluorine decoupling (H{F}), fluorine detection with proton decoupling (F{H}), fluorine-proton 2D heteronuclear Overhauser correlations (FH-HOESY), fluorine-proton 2D heteronuclear correlations (FH-HETCOR) and carbon detection with simultaneous proton and fluorine broadband decoupling (C{F,H}). In our facility only *Tellurium* can perform these experiments and they require that the HFX probe be installed. Only the Facility staff is authorized to change probes. Please make an appointment with one of the staff members to have the probe changed and for more information.

Proceed as follows:

1. Install the HFX probe (staff).
2. Change the filter configuration (staff).
3. Change the probe parameter in vnmrj (`probe='HFX_W015'`).
4. Load new shims (`setlock`) and lock and shim as usual.
5. Manually tune the probe if needed. Remember ch1=H1, ch3=F19, ch2=C13. (staff).
6. Select the experiment from the menu.
7. Always run `UMsetprobe` after an experiment is selected from the menu or the vertical panel.
8. Modify any parameters and click **[Acquire]**.



Default configuration

HFX configuration

When you are done:

1. Change the probe parameter in vnmrj (probe='`OneNMR_W010`').
2. Install default probe and change filter configuration.
3. Re-index protune.

X{F}, X{H}

Heteronuclear detection (^{13}C, ^{31}P, ^{11}B, etc) with fluorine broadband decoupling can be done in any fluorine capable spectrometer (*Gallium*, *Cobalt*, *Tellurium*). The decoupling scheme used (waltz16 or garp) is not efficient for very wide spectral widths but is fine for proton decoupling or for less than 10 ppm in fluorine. Fluorine decoupling with wide spectral widths works much better in *Tellurium*; see below. Heteronuclear detection with proton decoupling can also be done in this way.

1. Shim, lock, tune as usual.
2. Load parameters for X.
3. Set decoupler nucleus: `dn='F19'` or `dn='H1'`.
4. Type `UMsetbbdec(`*`offset_ppm`*`)`, where *offset_ppm* is the chemical shift in ppm at the center of the F19 or H1 region to decouple.
5. Larger decoupling widths can be achieved with garp: set `dmm='g'`, `dseq='garp'`.
6. Nuclei with negative magnetogyric ratio (^{15}N, ^{29}Si, ^{113}Cd, ^{119}Sn) exhibit a negative Overhauser effect that can reduce the signal intensity. Set `dm='nny'` to eliminate it.
7. Click **[Acquire]**.

Note: the parameters for F19 bb decoupling (pwx, pwxlvl) must exist in the probe file.

^{19}F-^{13}C-correlations.

Fluorine-Carbon correlations can be done in any fluorine capable spectrometer (*Gallium*, *Cobalt*, *Tellurium*) but the best results are obtained in *Tellurium* (no probe change required).

1. Shim, lock, tune as usual.
2. Load parameters for ^{19}F, take a quick spectrum, optimize spectral width and take a new spectrum.
3. From the main menu select **Experiments > Convert current parameters to do... > CRISIS2 Experiments > gc2hsqc**.
4. Check / adjust parameters (number of transients, C13 spectral width, etc) and click **[Acquire]**.

Note: “Crisis2” experiments work much better than other common experiments like hsqc or hmbc for F19 correlations.

C13{F19} on *Tellurium*

Larger broadband decoupling widths can be achieved with a Wurst scheme.

1. Load parameters for C13. If using the HFX probe, set `rfchannel='213'` and use channel 3 for decoupling.
2. Set `tn='H1' dn='F19'`
3. To make the Wurst waveform, use `makeFHdecshape(`*`bandwidth, maxjhf, dutycycle`*`)` where:

bandwidth is the spectral width to decouple in Hz;
maxjhf is the maximum coupling constant expected, and
dutycycle=1. For example: `makeFHdecshape.(40000, 280, 1)`

4. Use `setDECoffset(`*ppm*`)` to set the decoupler offset (the center of the region to decouple in ppm).
5. Set `dm='yyy'  dmm='p'`
6. Change the observe nucleus back to C13: `tn='C13'`.
7. Adjust any other parameters and click [Acquire].

C13{H1,F19} on *Tellurium* (HFX probe required)

It is possible to have simultaneous proton and fluorine decoupling with a reduced F19 decoupling region of about 10-15 ppm. Ask the staff for more details.

1. Follow instructions for C13{F19} to setup bb dec shape with shorter bandwidth (5kHz), dutyc=0.4. Record the parameters. Perhaps do the same for F19{H1}?
2. Load parameters for C13, set `rfchannel='213'`
3. Enter parameters from step 1 into channel 3 (F19), `dm2='nny', dmm='ccp'`

A brief manual can be found in the spectrometers in /vnmr/manual/F19-H1.pdf