

Fluorine experiments (Vnmrj 3.2A)

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Proton detection with fluorine decoupling: $^1\text{H}\{^{19}\text{F}\}$

Fluorine detection with proton decoupling: $^{19}\text{F}\{^1\text{H}\}$

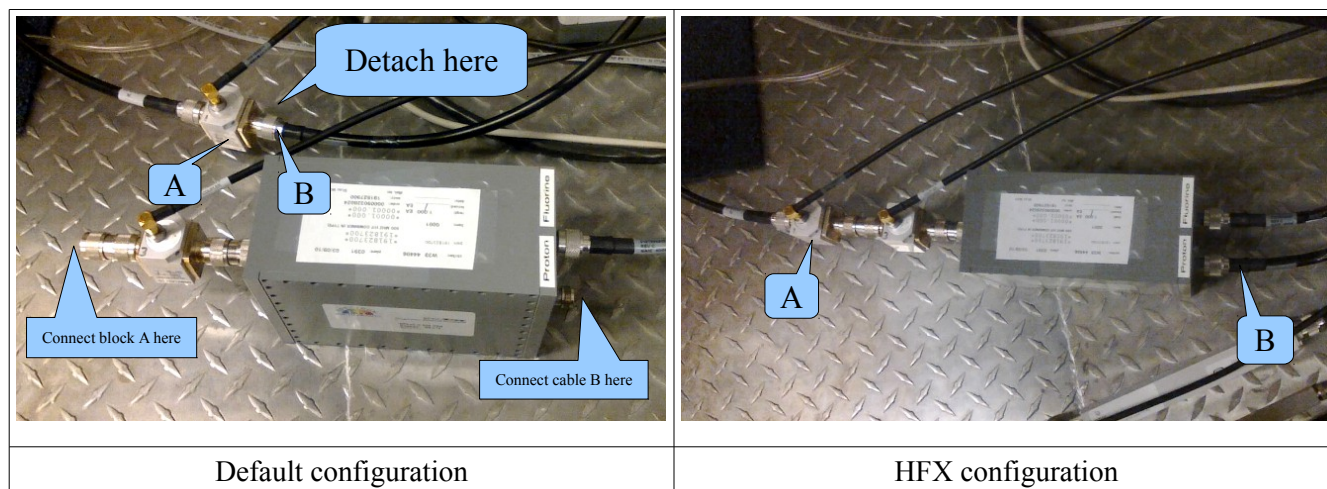
Fluorine-proton 2D heteronuclear Overhauser correlations: FH-HOESY

Fluorine-proton 2D heteronuclear correlations: FH-HETCOR

Thanks to the pulse sequences of Vnmrj 3.1, these experiments can be carried out in spectrometers that have only one high frequency channel. The only requirement is that the probe can be double-tuned to both ^1H and ^{19}F . In our Facility, *Cobalt* with its default QUAD probe and *Tellurium* with the HFX probe can perform these experiments. *Cobalt* doesn't require a probe change or re-tuning and the experiments are ready to run; all you need is to select the desired experiment and run it, but the performance is not as good as that of the HFX probe in *Tellurium*. On the other hand, *Tellurium*, with its HFX probe has much better sensitivity and fluorine decoupling range, but you will need to make an appointment with one of the staff members to have the probe changed. Only the Facility staff is authorized to change probes. Note that the sensitivity of the 1D experiments with decoupling is about 60% of the non-decoupled experiment. The default parameters provide a decoupling bandwidth of 8 kHz or about 16 to 20 ppm for both proton and fluorine. A brief manual can be found in the spectrometers in /vnmr/manual/F19-H1.pdf.

If using *Tellurium*, 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' and UMsetprobe) (staff).
4. Manually tune the probe if needed. Remember that ch1=H1, ch3=F19, ch2=C13. (staff).
5. Type UMsetprobe.
6. Load new shims (setlock) and lock and shim as usual.
7. Select the experiment from the menu Experiments > 19F-1H experiments.
8. In the Acquire > Defaults panel modify any desired parameter and enter the chemical shift of the center of the region to decouple in the Decoupler offset field. Click **Acquire**.



When you are done in *Tellurium*:

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

If using *Cobalt*, proceed as follows (no probe change, tuning or filters are needed):

1. Load new shims (setlock) and lock and shim as usual.
2. Select the experiment from the menu **Experiments > 19F-1H experiments**.
3. In the **Acquire > Defaults** panel modify any desired parameter and enter the chemical shift of the center of the region to decouple in the **Decoupler offset** field. Click **[Acquire]**.

Carbon-Fluorine 2D heteronuclear 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 is 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.

Heteronuclear detection with fluorine decoupling: X $\{^{19}\text{F}\}$

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 macro UMsetbbdec sets up a Wurst-40 adiabatic decoupling scheme that is effective over very large spectral widths like those common in fluorine spectra.

1. Shim, lock, tune as usual (see **Note 1**).
2. Load parameters for the X nucleus.
3. Set decoupler nucleus: dn='F19'. (see **Note 2**)
4. Type UMsetbbdec (ppm1, ppm2), where ppm1 and ppm2 define the boundaries to decouple. For example, to decouple from -70 ppm to -100 ppm type UMsetbbdec (-70, -100).
5. 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.
6. Click **[Acquire]**.

Note 1: If you are not using a deuterated solvent, you will have to shim with gradient shimming and the ^1H map (Acquisition > Do Gradient Shimming.. > Use ^1H Gradient Map); thus the probe has to be tuned to proton during shimming and tuned to fluorine after shimming. If your solvent is deuterated, gradient shimming will use the lock signal to tune and it doesn't matter if it is already tuned to fluorine. Remember that the probe has to be tuned to both nuclei of interest (X and ^{19}F) before starting the acquisition.

Note 2: If you are using the HFX probe in *Tellurium* in 3-channel configuration, fluorine is in channel 3 (the second decoupler). Set `dn2='F19', UMsetbbdec(-70, -100, 280, 1, 3)`.

Note 3: The parameters for F19 broadband decoupling (`pwx, pwxlvl`) must exist in the probe file.

Carbon detection with proton and fluorine decoupling in *Tellurium*: $^{13}\text{C}\{^1\text{H}, ^{19}\text{F}\}$ (HFX probe required)

It is possible to have simultaneous proton and fluorine decoupling in a carbon spectrum. However, if the fluorine spectrum has a very wide spectral width, it may be impossible to decouple the entire region in a single carbon spectrum and two or more ^{13}C spectra may need to be run where separate F19 regions are decoupled. This experiment can only be done in *Tellurium* with the HFX probe. Ask the staff to schedule a probe change.

1. Install and tune HFX probe and setup for 3 channel configuration (staff).
2. Load parameters for Carbon.
3. Type `dn2='F19'` to set the nucleus for the second decoupler (the third channel).
4. Type `UMsetbbdec(-60, -90, 280, 1, 3)`. The first two parameters define the F19 region to decouple in ppm, the third parameter is the maximum C-F coupling constant to decouple in Hertz, the fourth parameter should be 1 for this kind of experiment, and the last parameter should be 3 to select the third channel (F19 in 3-channel configuration) for decoupling.