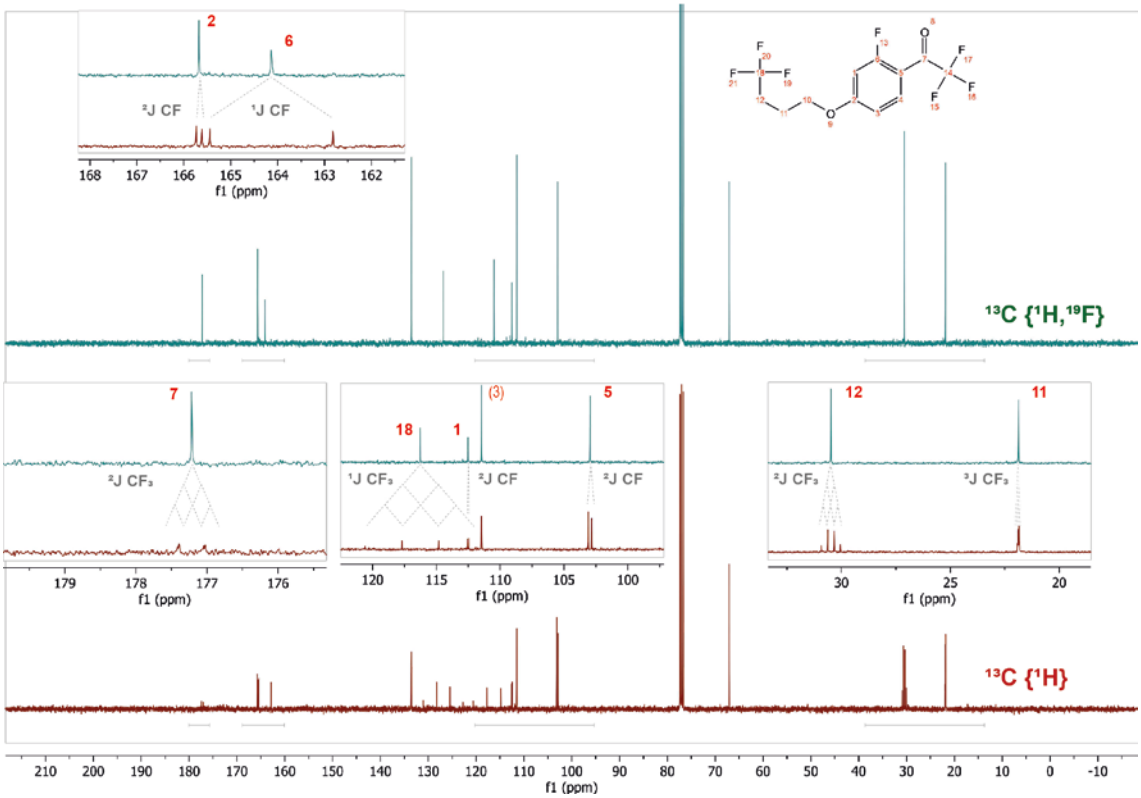
^{13}C NMR with ^{1}H and ^{19}F double decoupling

Note: This method is only available on the spectrometer **BCH1504R**

^{13}C spectra of fluorinated compounds appears with spited peaks as ^{19}F has a spin ½ and is 100% naturally abundant. This splitting is intense at 1 bond ($^{1}J^{19}F^{13}C$ = 250Hz) and is still visible 2 to 3 bonds away ($^{2}J^{19}F^{13}C$ = 50Hz).
It is therefore necessary to decouple ^{19}F to simplify ^{13}C spectra and to increase the signal to noise of ^{13}C peaks in the vicinity of ^{19}F sites.
Nevertheless, as ^{1}H need to be decoupled as well, a special triple channel probe capable to pulse at the same time on ^{1}H, ^{19}F and ^{13}C is needed. The NMR spectrometer located in BCH1504 (right) is equipped with such a probe.



Setup ^{13}C {^{1}H,^{19}F} 1d NMR experiment : (See as well manual experiment setup)

- Load your sample

* Start by acquiring a ^{1}H spectrum (useful 1) to check your samples 2) to cross reference the spectrum)

- Command : **newnmr** (create a ^{1}H dataset)
- Command : **atma** (tuning) ; **rsh** (read standard shim file); **lock** (solvent); **topshim** (shim)
- Command : **zg** (start the experiment)

* Setup ^{19}F experiment

- Command : **edc** (create a new dataset); **rpar refe*** (read pre-set experiment) → choose **refe_1d_19F_zg_{1H}** in user
- Command : **atma** (^{19}F tuning as it was not done before)
- Command : **zg** (start the experiment)

* Setup ^{13}C {^{1}H,^{19}F} experiment

- Command : **edc** (create a new dataset); **rpar refe*** (read pre-set experiment) → choose **refe_1d_13C_zg_{1H,19F}**
- Command : **atma** (^{13}C tuning as it was not done before)
- Command: **o3p** set the carrier of ^{19}F as centred as possible on your ^{19}F peaks (default is -100ppm) (see notes)
- Command : **zg** (start the experiment)

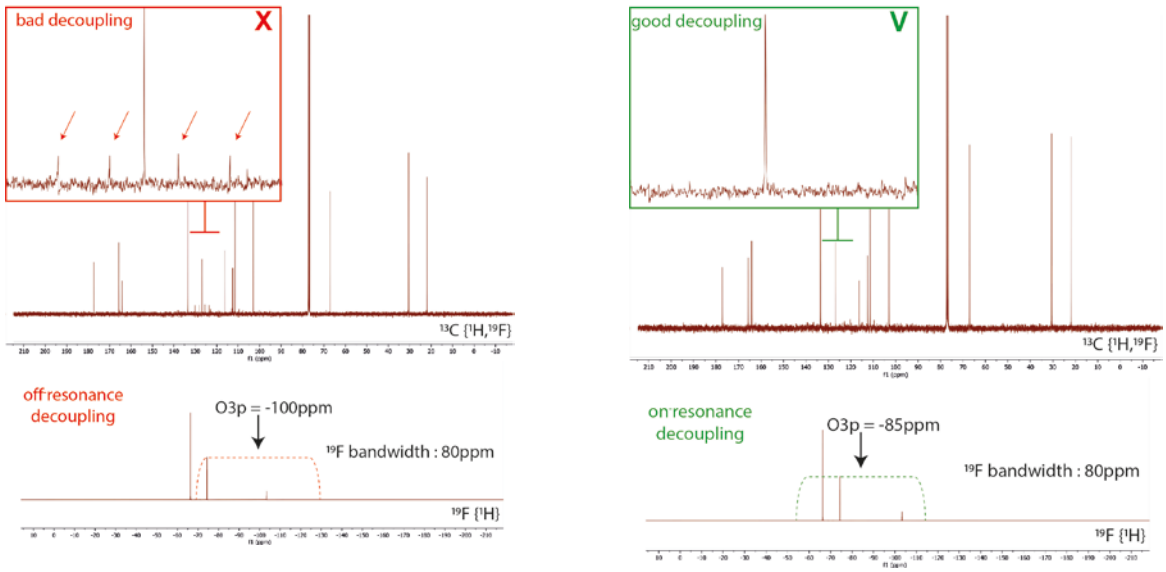
You can eventually change some parameters:

- **ns** : change number of scans.
  Rem : The signal to noise (SNR) accumulate proportionally to the square root of the number of scans (ns)
  Ex: if 1024 scans are needed to have a SNR of 5, 4096 scans will be needed to have a SNR of 10

- **O1P** and **SW** : the centre and the width (in ppm) of the spectral window

Note on ^{19}F decoupling bandwidth :

^{19}F decoupling bandwidth do not cover the entire ^{19}F spectral width (as ^{19}F spectral width is large in ppm and thus in Hz). It cover only about 80 ppm in the current setup.
Setting o3p, the ^{19}F carrier frequency, centred on the ^{19}F peaks will improve its decoupling efficiency

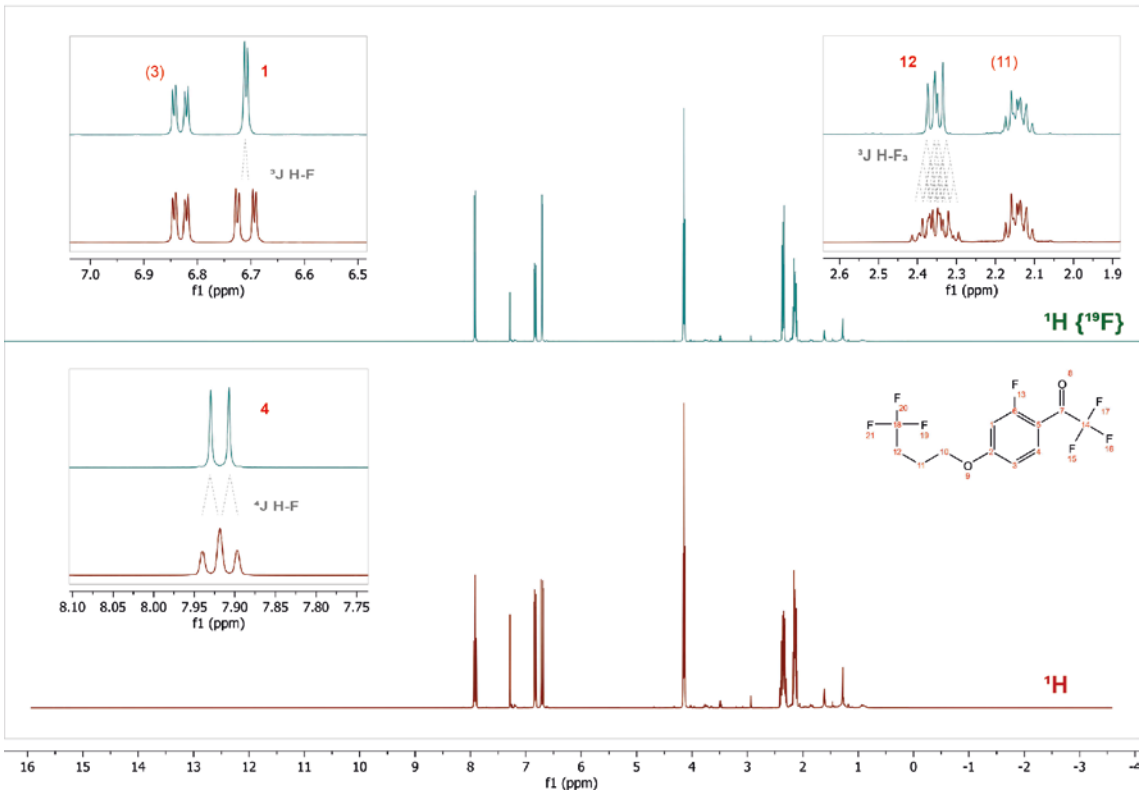


^{1}H ^{19}F decoupled:

It is as well possible simplify ^{1}H 1d spectra of fluorinated compounds decoupling ^{19}F. This experiment can be done on any standard NMR probe.

* Setup ^{1}H {^{19}F} experiment

- Command : **edc** (create a new dataset); **rpar refe*** (read pre-set experiment) → choose **refe_1d_1H_zg_{19F}**
- Command: **o2p** set the carrier of ^{19}F as centred as possible on your ^{19}F peaks (default is -100ppm) (see notes)
- Command : **zg** (start the experiment)



^{19}F ^{13}C HSQC: (see dedicated SOP)