

Application for fluorine compounds

Product used : Nuclear Magnetic Resonance (NMR)

ROYAL probe™ HFX can simultaneously irradiate ^1H , ^{19}F , and ^{13}C (or other X-nuclei) even in a basic console with basic two-channel console, and is a versatile probe that can measure a wide-variety of nuclei at high sensitivity. Here we introduce some useful experiments for fluorine-containing compounds that can be run on conjunction with JNM-ECZ400S equipped with ROYALPROBE™ HFX.

Data 1 : Triple-resonance measurement of ^{13}C data.

^{13}C signals of fluorinated compounds are typically split by the large J_{CF} coupling. These splittings significantly decrease sensitivity and complicate data analysis. $^{13}\text{C}\{^1\text{H}, ^{19}\text{F}\}$ spectra can be most easily analyzed and another advantage is the full sensitivity as shown in Fig.1.

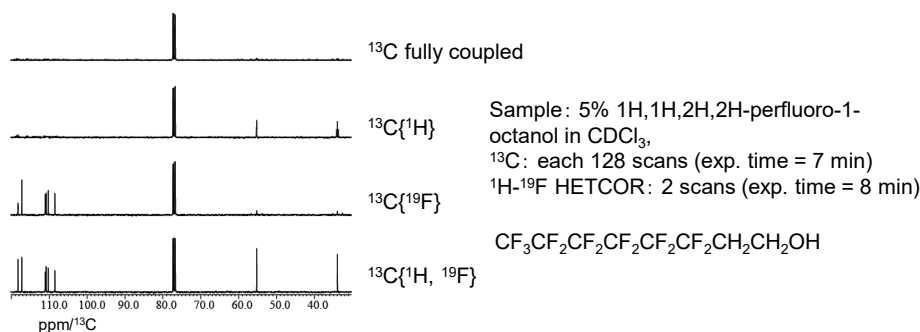


Fig. 1: Comparison of ^{13}C , $^{13}\text{C}\{^1\text{H}\}$, $^{13}\text{C}\{^{19}\text{F}\}$ and $^{13}\text{C}\{^1\text{H}, ^{19}\text{F}\}$ spectra

Data 2 : ^1H - ^{19}F HETCOR

^1H - ^{19}F HETCOR (HETeronuclear CORrelation) is a very useful experiment for structure elucidation (Fig.2).

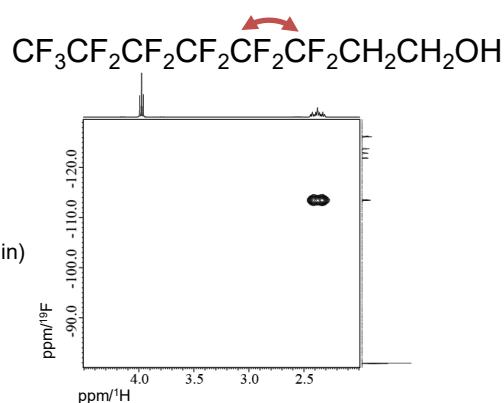


Fig. 2: ^1H - ^{19}F HETCOR spectrum

Data 3: 1D ^1H - ^{19}F HOESY

Trifluridine is a trifluoromethyl derivative of deoxyuridine that is used as an antineoplastic and antiviral agent. It is not easy to observe the correlation signal between ^1H and ^{19}F in this compound, since the 4-bond ^1H - ^{19}F J -coupling is very small. 1D ^1H - ^{19}F HOESY (Fig. 3) is an effective method to selectively observe signal of protons which are spatially close to fluorine atoms within a couple of minutes. 2D ^1H - ^{19}F HOESY can also provide similar information, but it takes several hours to collect 2D data. Fig. 4 shows that the proton atom observed at 8.65 ppm is spatially close to the CF_3 group.

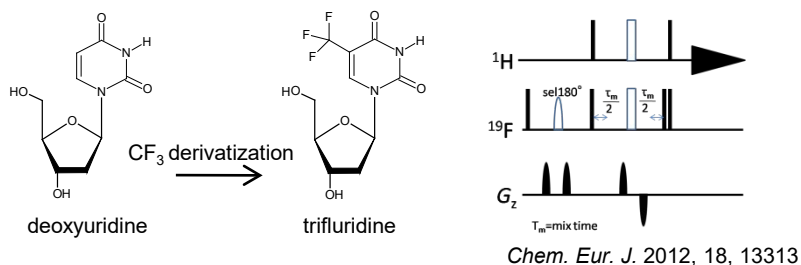


Fig. 3: pulse sequence of 1D ^1H - ^{19}F HOESY

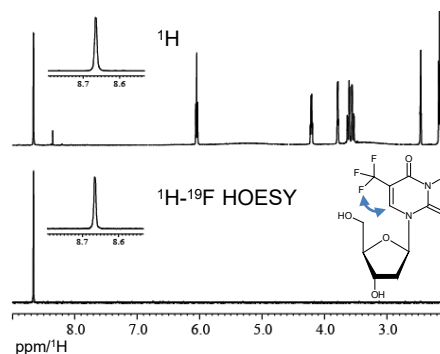


Fig. 4: 1D ^1H - ^{19}F HOESY spectrum
Sample: 100 mM Trifluridine in DMSO- d_6
Upper spectrum: ^1H , 8 scans (exp. time = 1 min),
Lower: HOESY, 16 scans (exp. time = 1 min),
mix_time = 1s

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