

# Heteronuclear Correlations

## Table of Contents

1. The XHCORR sequence
2. The COLOC sequence (COrrrelation via LOng range Coupling)
3. Heteronuclear 2D J-resolved Pulse Sequence
4. Contributors

The heteronuclear correlation of chemical shifts by scalar coupling derives from the existence of heteronuclear scalar coupling all owing a magnetization transfer from the more sensitive nucleus ( $^1\text{H}$ ) toward the less sensitive nucleus ( $^{13}\text{C}$ ). The model experiment is the correlation  $\delta(^{13}\text{C})/\delta(^1\text{H})$  which uses the large direct coupling  $^1J(^{13}\text{C}-^1\text{H})$  comprised between more 100 Hz and 200Hz and which allows to correlate the signals from a proton to that of a  $^{13}\text{C}$  to which it is bound (5).

## The XHCORR sequence

This sequence allows to correlate the signals of the  $^1\text{H}$  and of the  $^{13}\text{C}$  bound to each other (13). Spectrum 11 (XHCORR), shows the correlations for the dihydrofuran (Fig. 31) between bound carbons and protons. The chemical shifts of the protons of these molecules are in the table 2, those of the carbon are in the table 3.

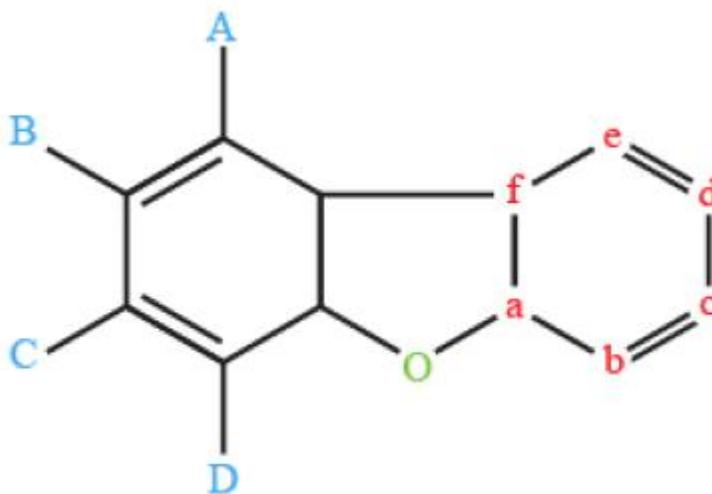


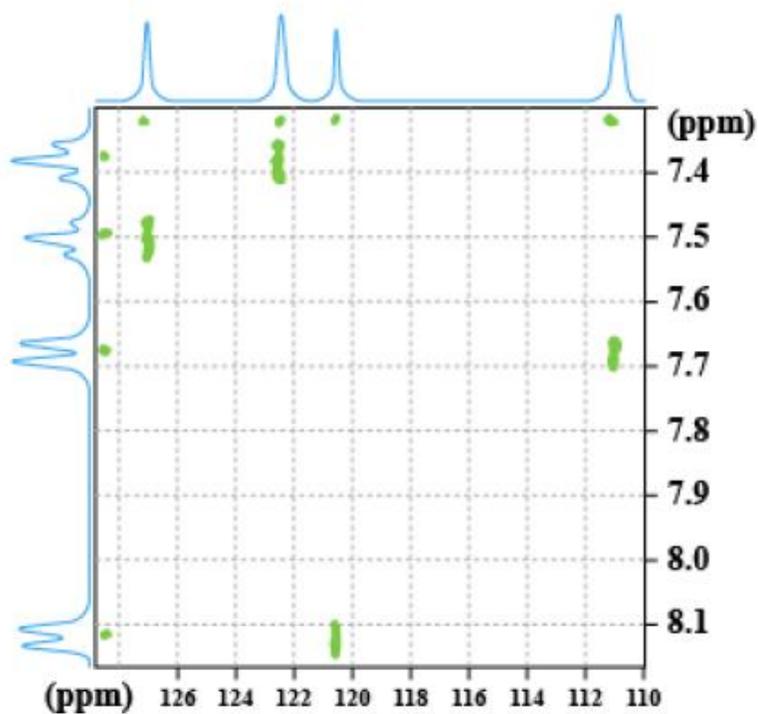
Fig. 31. The dibenzofuran (D.B.F.)

<sup>1</sup> H	Shift (ppm)
A	8.15
B	7.4
C	7.5
D	7.7

**Table 2:** DBF protons chemical shift.

<sup>13</sup> C	Shift (ppm)
a	156.2
b	111.6
c	127.0
d	122.6
e	120.6
f	124.2

**Table 3:** DBF carbons chemical shift



**Spectrum:** XHCORR of the DBF

Within the XHCORR pulse sequence (Fig. 25), transverse magnetization is caused by a  $\pi/2(^1H)$  impulsion which is evolving during the  $t_1$  period. The  $\pi$  impulsion ( $^{13}C$ ), located in the middle of this period refocuses the heteronuclear couplings.

The optimization of the  $\Delta_1$  and  $\Delta_2$  delays allows the selection of the long range heteronuclear couplings, this means that instead of seeing the correlation between  $^{13}C$  and protons directly bound ( $H_B$ ), we favor the appearance of the correlations spots between  $^{13}C$  and non bound protons ( $H_{NB}$ ).

For example, for a coupling constant  $J=10$  Hz then  $\Delta_1=50$  ms and  $\Delta_2=33$  ms.

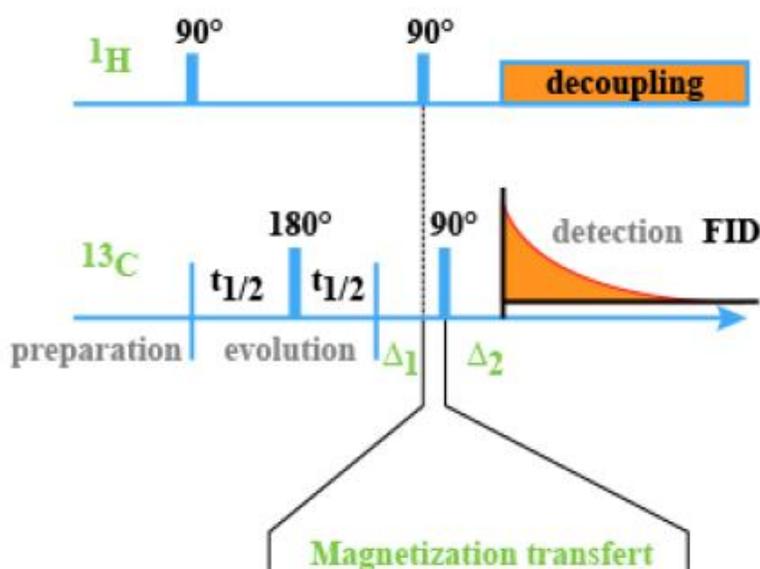


Fig. 25: The XHCORR pulse sequence.

## The COLOC sequence (CORrelation via LOnG range Coupling)

The sequence which is used (Fig. 26) allows generally a good correlation between the quaternary carbons and the neighbouring protons. On the spectrum 12 (COLOC), the correlation between the quaternary carbon ( $^{13}C = \delta = 156.2$  ppm) and its vicinal proton ( $\delta = 7.7$  ppm) is named Cq. However, it does not give good results for long range correlations involving protonated carbons (14). This observation led to write a pulse sequence named XC ORFE (X nucleus proton CORrelation with Fixed Evolution time). This one is very closed to the COLOC sequence.

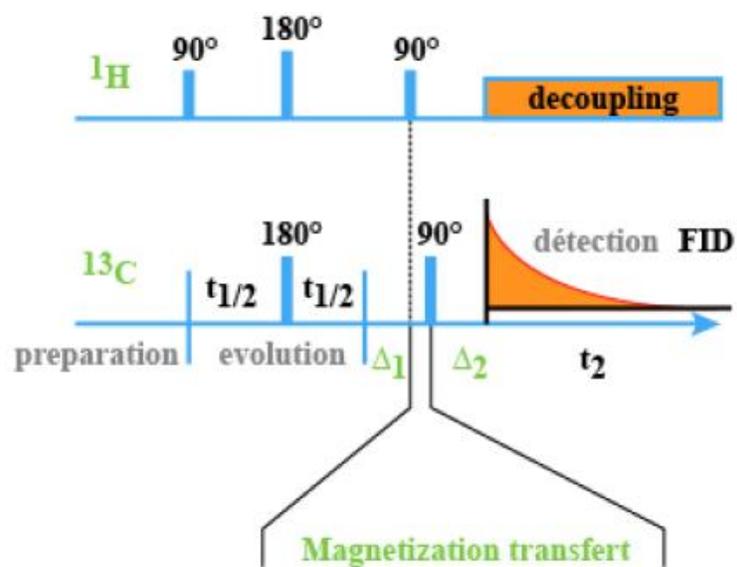
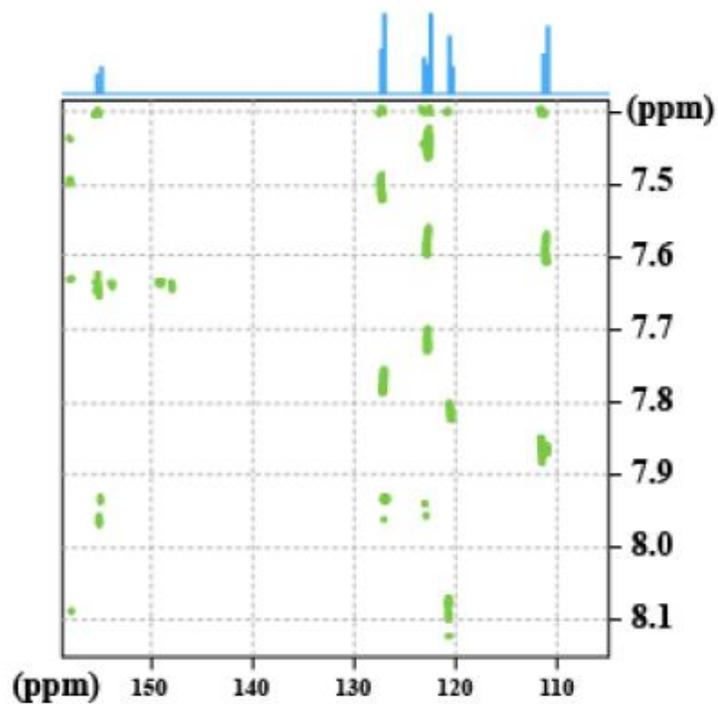


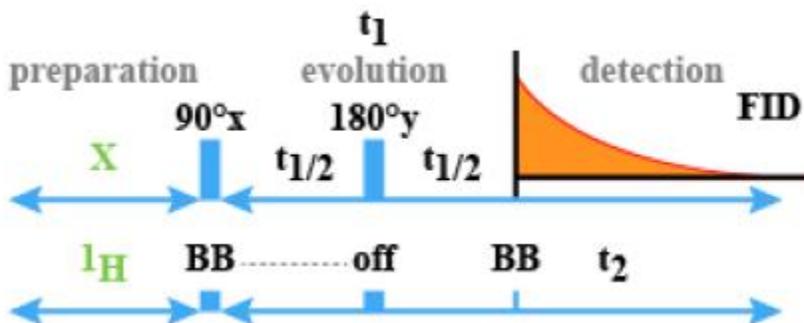
Fig. 26: The COLOC pulse sequence (14).



Spectrum 12: COLOC of DBF

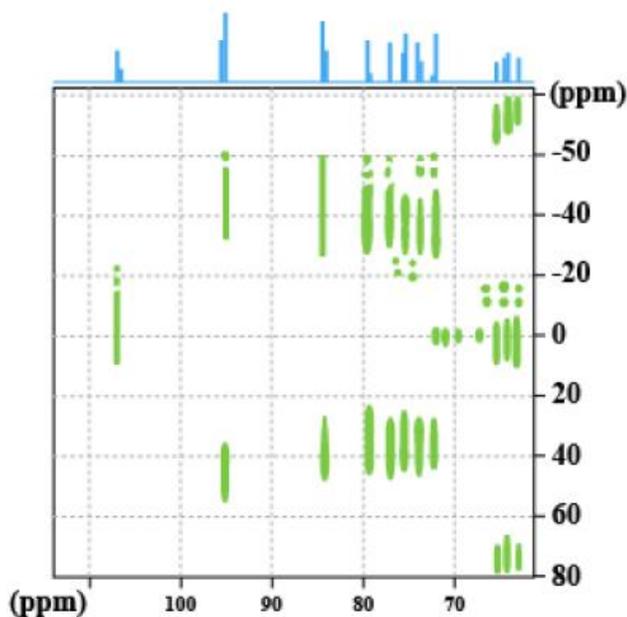
## Heteronuclear 2D J-resolved Pulse Sequence

In this experiment we observe the coupling  $J$  between the X nuclei ( $^{13}\text{C}$ ,  $^{15}\text{N}$ ,  $^{31}\text{P}$  ...etc ) and the protons which are bound to them (Fig. 27). A coupled normal 1D spectrum XH may be very difficult to explain. This is due to the intercrossing of the multiplets (12). For simplifying the spectra, we will spread the coupling data in the F1 dimension and the informations arising from the chemical shifts in the F<sub>2</sub> dimension.



**Fig. 27:** The 2D J-resolved heteronuclear pulse sequence.

The results are given for the saccharine (Fig. 29). The F<sub>2</sub> carbon (108 ppm) is a quaternary one because we may observe only one spot devoid of coupling. The G, carbon (95 ppm) is a CH because there are two correlation spots. Moreover, the coupling constant between this  $^{13}\text{C}$  and this  $^1\text{H}$  is of 80 Hz approximately (Spectrum 13 and 14).



**Spectrum 13:** Heteronuclear 2D J-resolved

The Heteronuclear correlation of chemical shifts in inverse detection. What is the reverse detection? The reverse detection or indirect detection affords the NMR parameters (chemical shifts,  $t_1$ , ...) of a weakly sensitive X nucleus by observing it through a much more sensitive nucleus (generally the proton) (1.5).

## Contributors

- Engineer of Research. Marc Bria (University of Lille1)
- The virtual NMR Spectrometer

Source:[http://chemwiki.ucdavis.edu/Physical\\_Chemistry/Spectroscopy/Magnetic\\_Resonance\\_Spectroscopies/Nuclear\\_Magnetic\\_Resonance/NMR%3A\\_Experimental/2D\\_NMR/Heteronuclear\\_Correlations](http://chemwiki.ucdavis.edu/Physical_Chemistry/Spectroscopy/Magnetic_Resonance_Spectroscopies/Nuclear_Magnetic_Resonance/NMR%3A_Experimental/2D_NMR/Heteronuclear_Correlations)