Observation of NOE by HSQC-NOESY

Product used : Nuclear Magnetic Resonance (NMR)

NOE (Nuclear Overhauser Effect) correlations comprise important information to estimate internuclear distance and determine structure. However, NOE correlation peaks are very weak compared with diagonal peaks in 2D NOESY. For this reason, it is difficult to observe NOE correlation peaks in the vicinity of much larger diagonal peaks. In such cases, HSQC-NOESY can alleviate the problem as it does not detect diagonal peaks. On the other hand, it is important to consider its low sensitivity.

Fig. 1 shows an expansion of NOESY spectrum of 45 mg diphenyl(2,4,6-trimethybenzol)phosphine oxide in $CDCl_3$. Only H3/H1 correlation is clearly observed in the aromatic region. It is difficult to say whether or not there is a correlation between H1 and H4.

Fig. 2 shows a pulse sequence of ^{1}H-^{13}C HSQC-NOESY. This experiment does not employ ^{13}C decoupling during the acquisition time, and hence HSQC correlations are observed as doublets due to $^{1}J_{CH}$. On the other hand, positive NOE correlations are observed with opposite phase compared to HSQC correlations. This makes discrimination of HSQC and HSQC-NOESY peaks possible and minimizes overlaps.

The HSQC-NOESY spectrum of the same sample is shown in Fig. 3. In addition to the correlation peak H4/H1 that could not be observed in the NOESY spectrum because of the signal overlap with the diagonal signals, the correlation of H3/H3 with identical chemical shifts can also be observed. See Fig. 4 for the structural formula of diphenyl(2,4,6-trimethybenzol)phosphine oxide and the NOE correlations observed in the HSQC-NOESY spectrum.

If ^{13}C decoupling was applied, H4/H1 and H3/H3 NOE correlations would be overlapped with the relatively strong HSQC correlations.

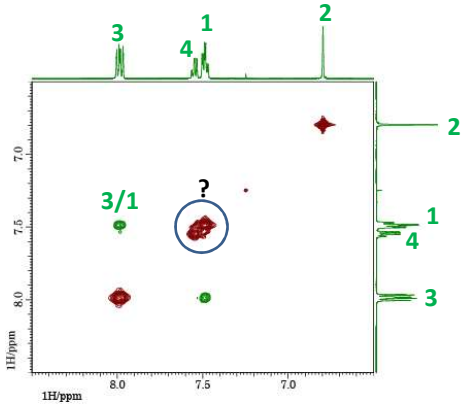

Fig. 1: Expansion of ^{1}H-^{1}H NOESY

Scans = 4, Y points = 256, mixing time = 3 s, exp. time = ca. 2.75h

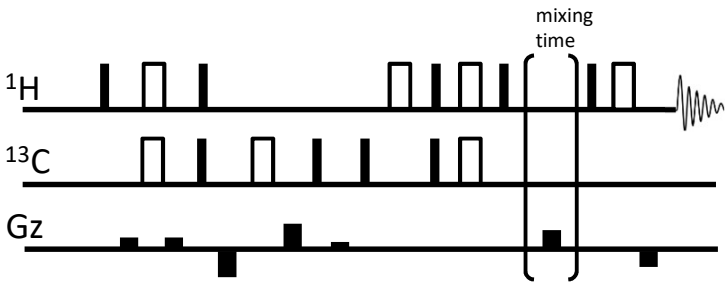

Fig. 2: Pulse sequence of HSQC-NOESY

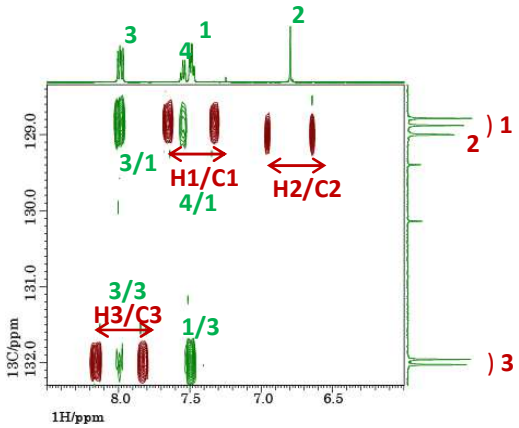

Fig. 3: Expansion of HSQC-NOESY

※C_1 and C_3 signals are doublets due to ^{31}P ($^{2}J_{PC}$ and $^{3}J_{PC}$)

● HSQC correlation、● Positive NOE correlation

Scans = 32, Y points = 256, mixing time = 3 s, exp. time = ca. 22 h

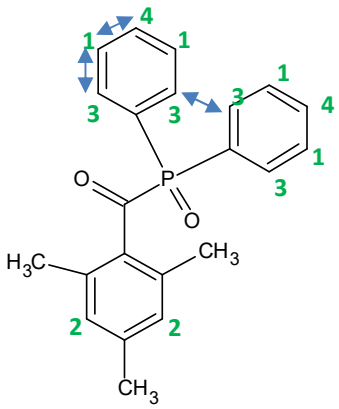

Fig. 4: NOE Correlations indicated in Fig.3

instrument : JNM-ECZ500R, ROYALPROBE™ HFX

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