Positive Probability Ltd

Note N3: NMR – Resolving Long Range Coupling Constants

Introduction

Modern NMR spectrometers are capable of generating pulse sequences to suit almost any application. Consequently, it is possible to use sequences that will provide a complete coupling connectivity path to aid interpretation and it is rarely necessary to determine coupling constants and match them. Even so, there are applications where knowledge of coupling constants is useful. These may be obtained by using a special pulse sequence but they may also be obtained from a deconvolution to enhance resolution.

Data and Data Processing

This 1H example shows two protons from the spectrum of a steroid. The low field proton (left hand multiplet) has 4 couplings to other protons and there is some peak overlap. The high field multiplet also has 4 coupling constants but one is small and unresolved. The model was designed from the left and right peaks of the low field multiplet and this was used as the input to the ***ReSpect™***–based ***Sleuth™*** deconvolution program. The result was quantified so that the coupling constants could be determined.

Results and Discussion

Figure 1 shows the raw data, reconstruction, misfit, deconvolution and a deconvolution spike plot in which the spike heights are directly proportional to the found intensities.

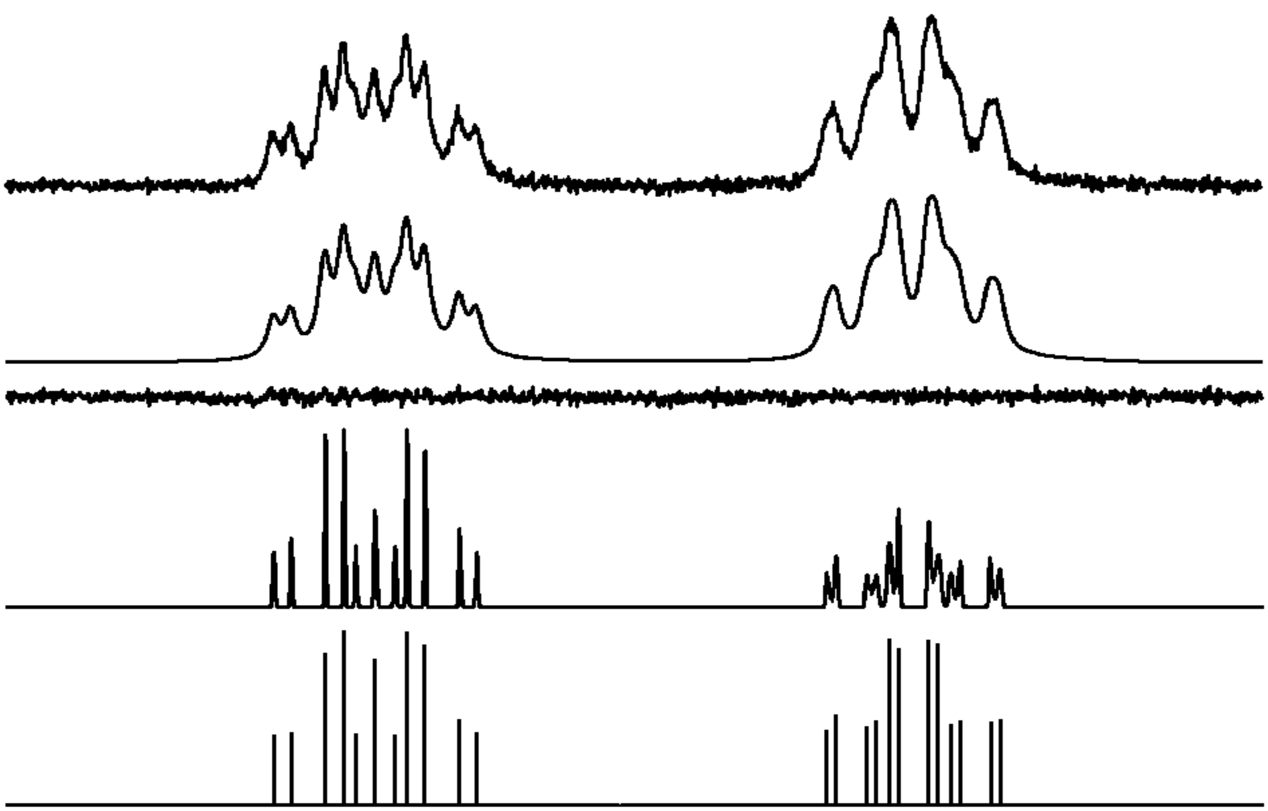

Figure 1. Top to bottom: raw data; reconstruction; misfit; deconvolution; spike plot

As can be seen, data reconstruction techniques efficiently separate signals from noise. The model was designed to fit the low field multiplet and the deconvolution for this proton is therefore sharp and clean. The deconvolved peaks for the high field proton are not so well resolved and this is for two reasons. Firstly, because the peak overlap is more severe and the certainty of peak positions and intensities is reduced. Tis is reflected in the width of the deconvolved signals. Secondly, the model was slightly too narrow for this proton and this also contributes to a broadening of the deconvolved signals. The spike plot shows the true intensity ratios of the peaks and the coupling patterns are obvious.

The fully quantified results are shown in the table below. Coupling constants are quoted in Hertz and the error bars are for one standard deviation. Proton A is at low field.

Found Coupling Constants

Proton A	Proton B
12.68± 0.17	14.70± 0.22
7.87± 0.16	6.03± 0.21
7.87± 0.16	6.03± 0.21
2.84± 0.17	1.35± 0.21

Note that the increased peak overlap for Proton B increases the uncertainty in peak position. There is therefore a consequent increase in the error bars.

Conclusions

This example has shown that fully quantified coupling constants may be extracted from data where peaks are severely overlapped without resorting to a second series of acquisitions.