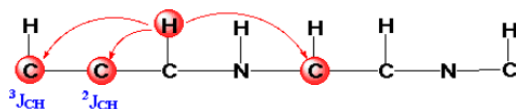


Measuring methods available and examples of their applications

2D HMBC (Heteronuclear Multiple-Bond Correlation spectroscopy)

HMBC detects heteronuclear correlations over longer ranges of about 2–4 bonds. The 2D HMBC experiment permits to obtain a 2D heteronuclear Chemical Shift correlation map between long-range coupled ^1H and heteronuclei (usually ^{13}C). It is widely used because it is based on proton-detection, offering high sensitivity in magnitude mode. In addition, long-range proton-carbon coupling constants can be measured from the resulting spectra.



The HMBC spectrum shows the typical 2D long-range correlation map. A cross-peaks means that the corresponding ^1H and heteronucleus are two- or three-bonds away. Residual direct connectivities are usually present as large doublets due to $^1\text{J}(\text{CH})$. A modification of the HMBC method is available, which allows suppression one-bond signals, leaving only the multiple-bond signals.

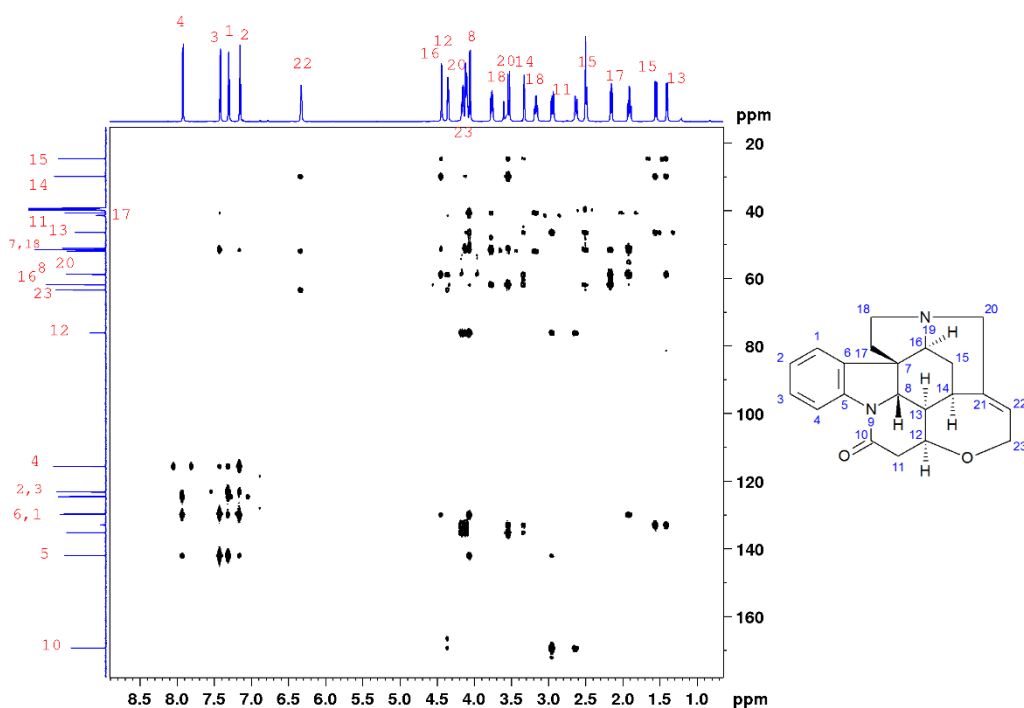


Fig. 1. Strychnine nitrate in DMSO- D_6 , ^1H , ^{13}C -HMBC optimized for $\text{J}(\text{CH})$ long range = 10 Hz in magnitude mode with assignment. Spectrometer: AVANCE III HD 700, Probehead: 5 mm PABBO BB-1H/D, Experiment time: 50 min

¹⁵N HMBC

¹H-¹⁵N-HMBC is basically the same as the proton-carbon correlation HMBC experiment, but as a result you obtain the ¹H-¹⁵N-correlation. In contrast to a ¹⁵N-detected NMR spectrum it is much more sensitive. Thus you need less time for a measurement. In one example (molecular weight 300 g/mol, concentration 30 mg/ml) it was possible to obtain a good quality ¹H-¹⁵N-HMBC-spectrum within 60 min. In order to obtain a ¹⁵N-NMR spectrum under the same conditions you would have to measure overnight. Moreover, the ¹H-¹⁵N-HMBC-spectrum provides additional information for structure elucidation. Therefore, a ¹H-¹⁵N-HMBC-spectrum is preferable over a ¹⁵N-NMR spectrum in most cases.

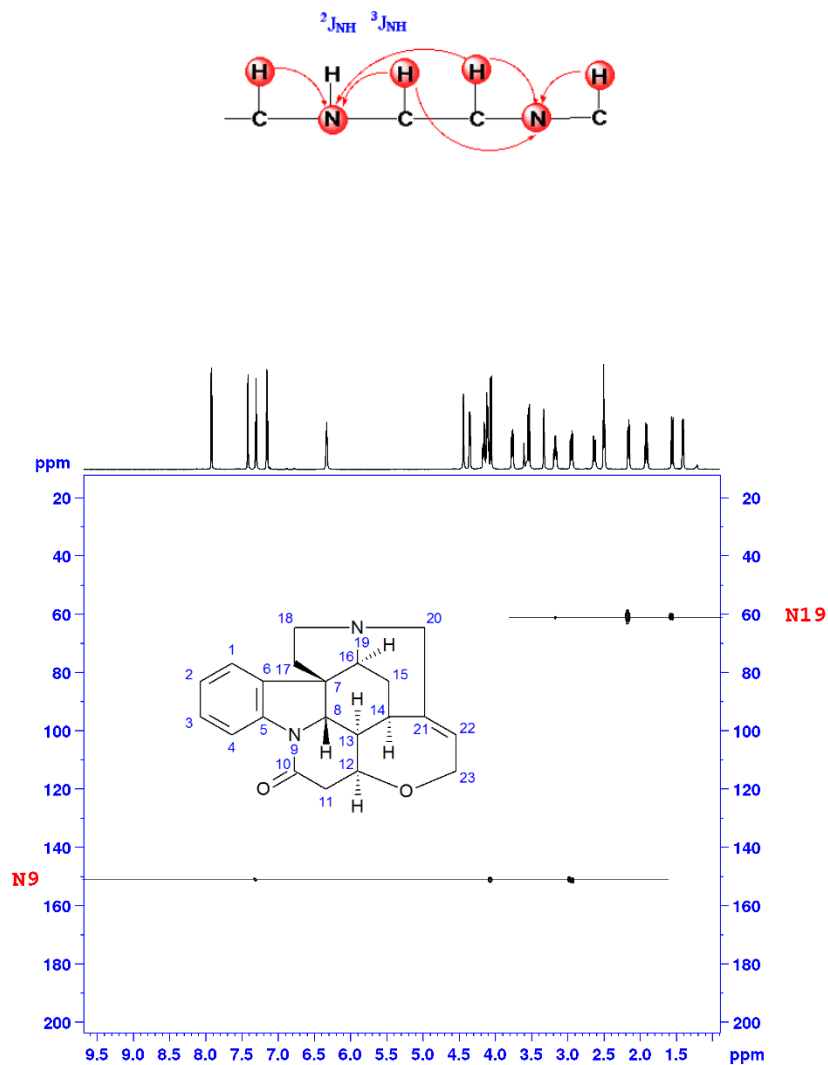


Fig. 2. Strychnine nitrate in DMSO-D₆, ¹H, ¹⁵N-HMBC with assignment, Spectrometer: AVANCE III HD 700B, Probehead: 5 mm CPTXO ¹³C/¹⁵N-¹H/D with z gradients, Experiment time: 70 min