Brucine (2,3-Dimethoxystrychnidin-10-one)

Brucine (2,3-Dimethoxystrychnidin-10-one) is an alkaloid, structurally related to strychnine, but less toxic. Figure 1 shows the ^{1}H NMR spectrum of a 50 mM brucine sample in $CDCl_3$ measured in a single scan taking 15 seconds to acquire.

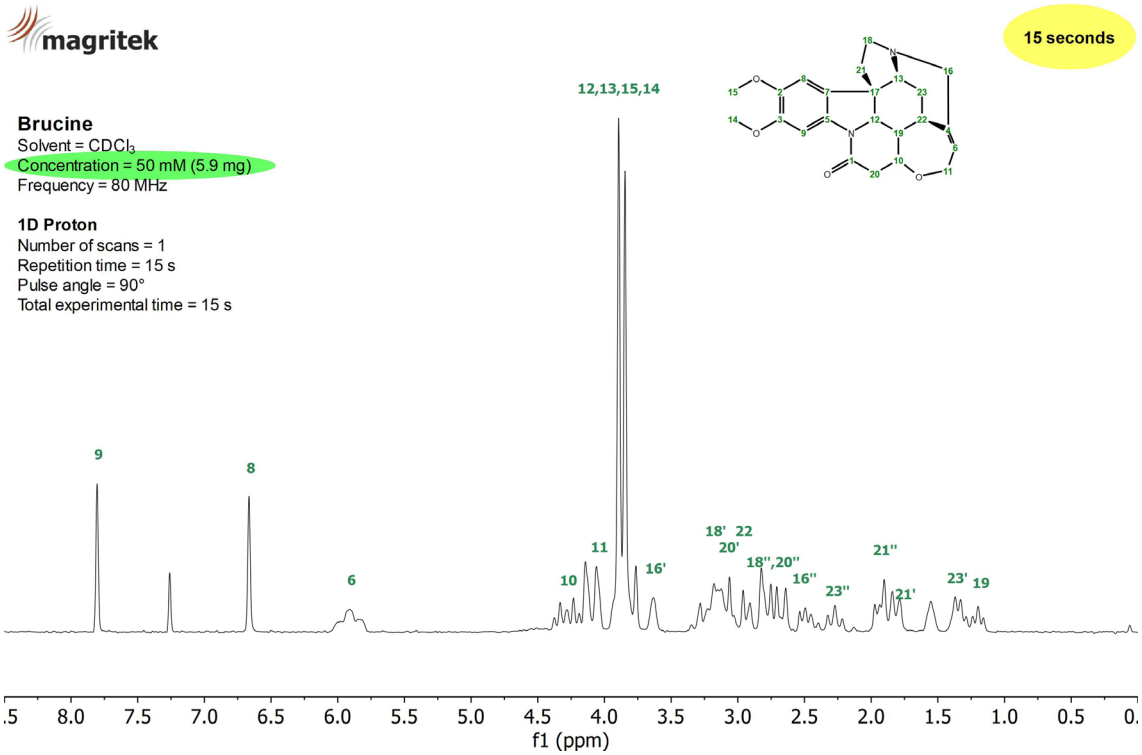

Figure 1: ^{1}H NMR spectrum of a 50 mM brucine sample in $CDCl_3$ measured on a Spinsolve 80 MHz system in a single scan.

2D COSY

The 2D COSY experiment allows one to identify coupled ^{1}H nuclei as they generate cross peaks out of the diagonal of the 2D data set. In Figure 2 a large number of cross peaks are well identifiable. For example, the protons at position 6 and 11 (light green) couple with each other. Furthermore, proton 19 couples with proton 10 (light blue), 12 (orange) and 20 (pink). In addition, the couplings between protons 8 and 9 (dark blue) as well as the couplings of protons 8 and 9 with protons 14 and 15 (dark green) can be observed nicely.

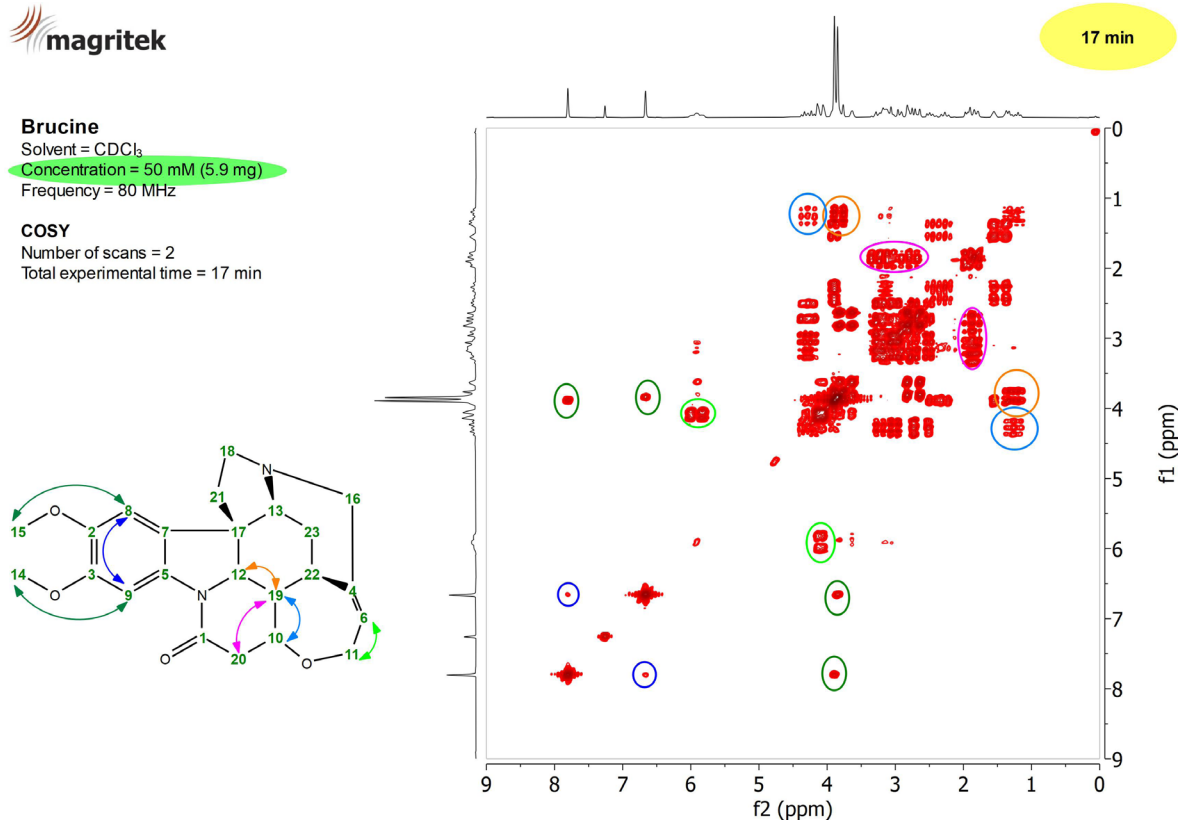

Figure 2: ^{1}H 2D COSY experiment of a 50 mM brucine sample in $CDCl_3$ acquired in 17 minutes on a Spinsolve 80 MHz system.

^{13}C Spectrum

Figure 3 shows the ^{13}C NMR spectrum of 1 M brucine in $CDCl_3$ acquired using NOE polarization transfer from ^{1}H to ^{13}C and ^{1}H decoupling. The 1D carbon experiment using NOE is sensitive to all ^{13}C nuclei in the sample. It clearly resolves all the expected resonances.

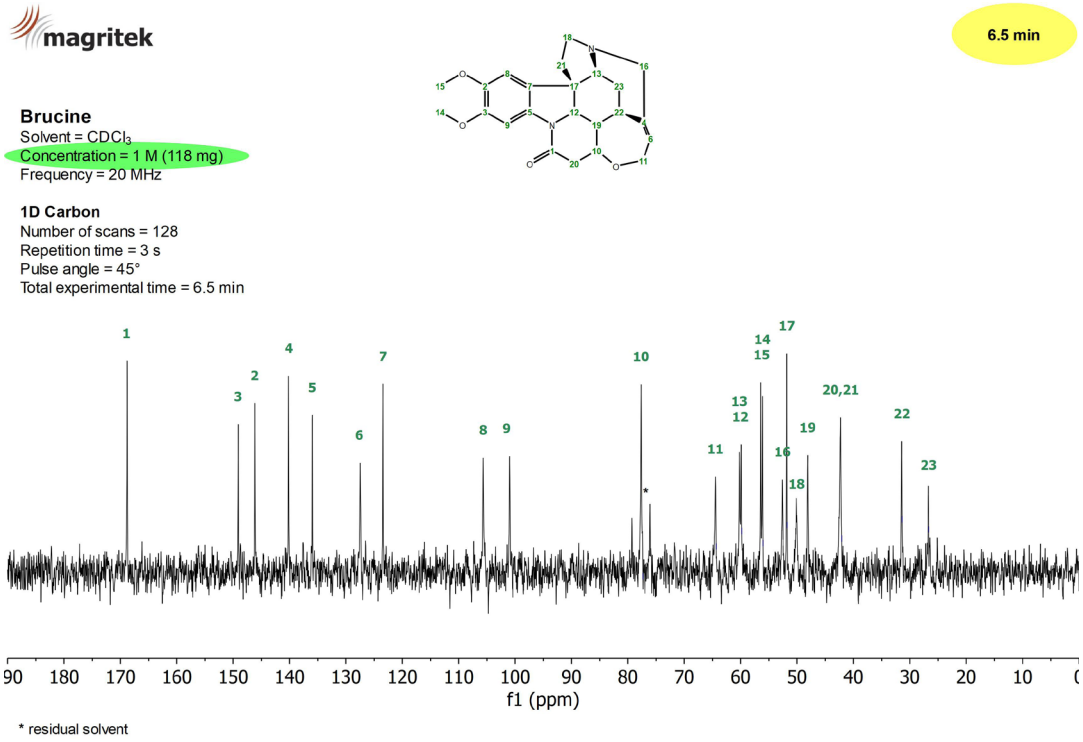

Figure 3: ^{13}C NMR spectrum of a 1 M brucine sample in $CDCl_3$ measured on a Spinsolve 80 MHz system in 6.5 minutes.

2D HSQC-ME and 2D HMBC

The HSQC and HMBC are powerful sequences widely used to correlate the one-bond coupled ^{1}H-^{13}C nuclei and long range ^{1}H-^{13}C, respectively. The Spinsolve is equipped with a multiplicity edited version (HSQC-ME) of this method. It provides the editing power of the DEPT-135 sequence, which is useful to differentiate between the signals of the CH_2 groups (blue) from the CH and CH_3 groups (red). Thanks to the higher sensitivity of these sequences, compared to direct detected ^{13}C experiments, samples with lower concentrations can be measured in relatively short times. Figure 4 shows the HSQC-ME (left) and HMBC (right) spectra of a 250 mM brucine sample in $CDCl_3$ acquired in 4 minutes and 34 minutes, respectively. The HSQC spectrum resolves all coupled ^{13}C and allows one to resolve the ^{1}H signals that overlap in the 1D proton spectrum. On the other hand, the HMBC shows the long-range correlations that are useful to identify and assign the different peaks in the spectrum. As an example, we marked on the HMBC spectrum the cross peaks of proton 8 with carbons 2, 3, 5, 7, 9 and 17 (the sequence shows the correlation with quaternary carbons, too).

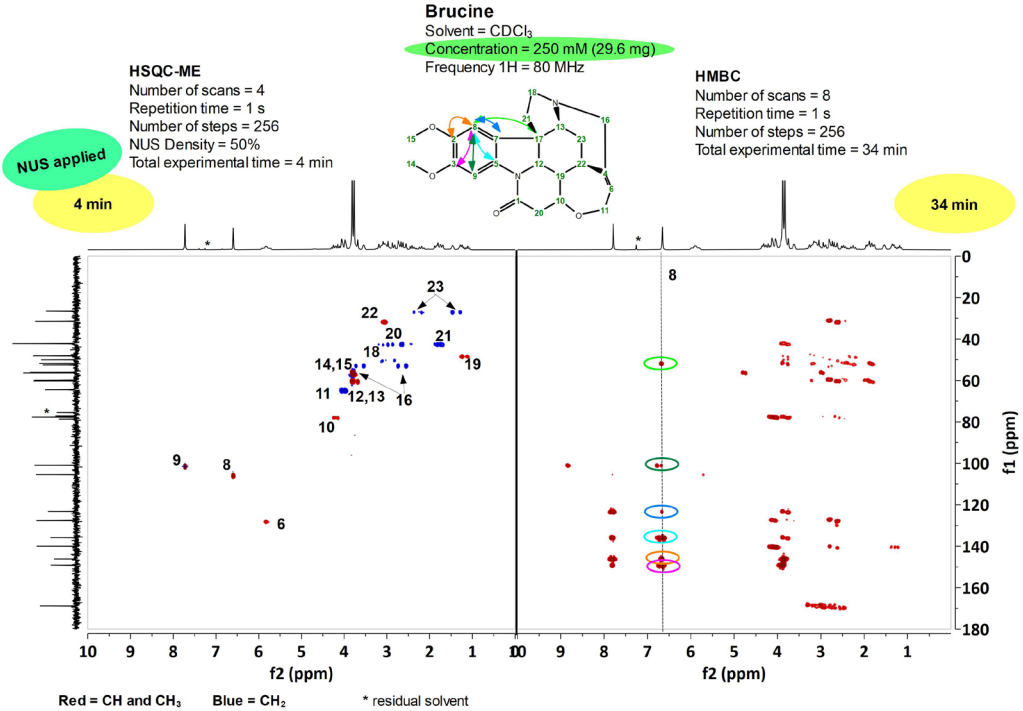

Figure 4: HSQC-ME (left) and HMBC (right) spectra of a 250 mM brucine sample in $CDCl_3$ showing the correlation between the ^{1}H (horizontal) and ^{13}C (vertical) signals.