

## 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  $^1\text{H}$  NMR spectrum of a 250 mM Brucine sample in  $\text{CDCl}_3$  measured in a single scan taking 10 seconds to acquire.

### 1D Proton spectrum

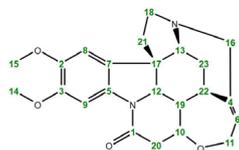


#### Brucine

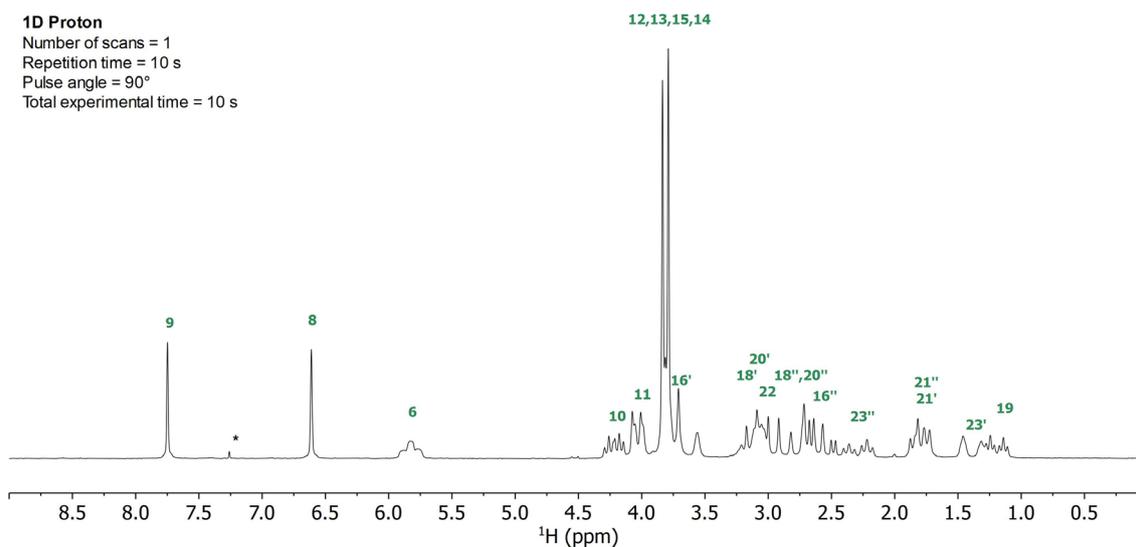
Solvent =  $\text{CDCl}_3$   
Concentration = 250 mM  
Frequency = 100 MHz

#### 1D Proton

Number of scans = 1  
Repetition time = 10 s  
Pulse angle =  $90^\circ$   
Total experimental time = 10 s



10 seconds



\* residual solvent

Figure 1:  $^1\text{H}$  NMR spectrum of a 250 mM Brucine sample in  $\text{CDCl}_3$  measured on a Spinsolve 100 MHz system in a single scan.

### 1D Carbon spectrum

Figure 2 shows the  $^{13}\text{C}$  NMR spectrum of 250 mM Brucine in  $\text{CDCl}_3$  acquired using NOE polarization transfer from  $^1\text{H}$  to  $^{13}\text{C}$  and  $^1\text{H}$  decoupling. The 1D Carbon experiment using NOE is sensitive to all  $^{13}\text{C}$  nuclei in the sample. It clearly resolves all the expected resonances.

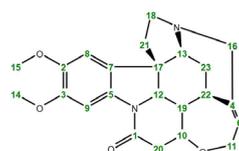


#### Brucine

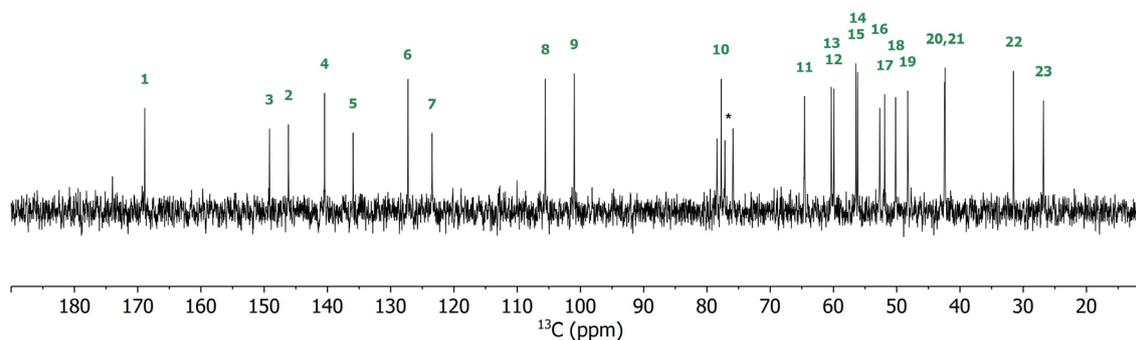
Solvent =  $\text{CDCl}_3$   
Concentration = 250 mM  
Frequency = 25 MHz

#### 1D Carbon

Number of scans = 512  
Repetition time = 3 s  
Pulse angle =  $45^\circ$   
Total experimental time = 26 min



26 min



\* residual solvent

Figure 2:  $^{13}\text{C}$  NMR spectrum of a 250 mM Brucine sample in  $\text{CDCl}_3$  measured on a Spinsolve 100 MHz system in 26 minutes.

## 2D COSY spectrum

The 2D COSY experiment allows one to identify coupled  $^1\text{H}$  nuclei as they generate cross peaks out of the diagonal of the 2D data set. In Figure 2 a large number of cross peaks can be nicely observed. 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 nicely observed.

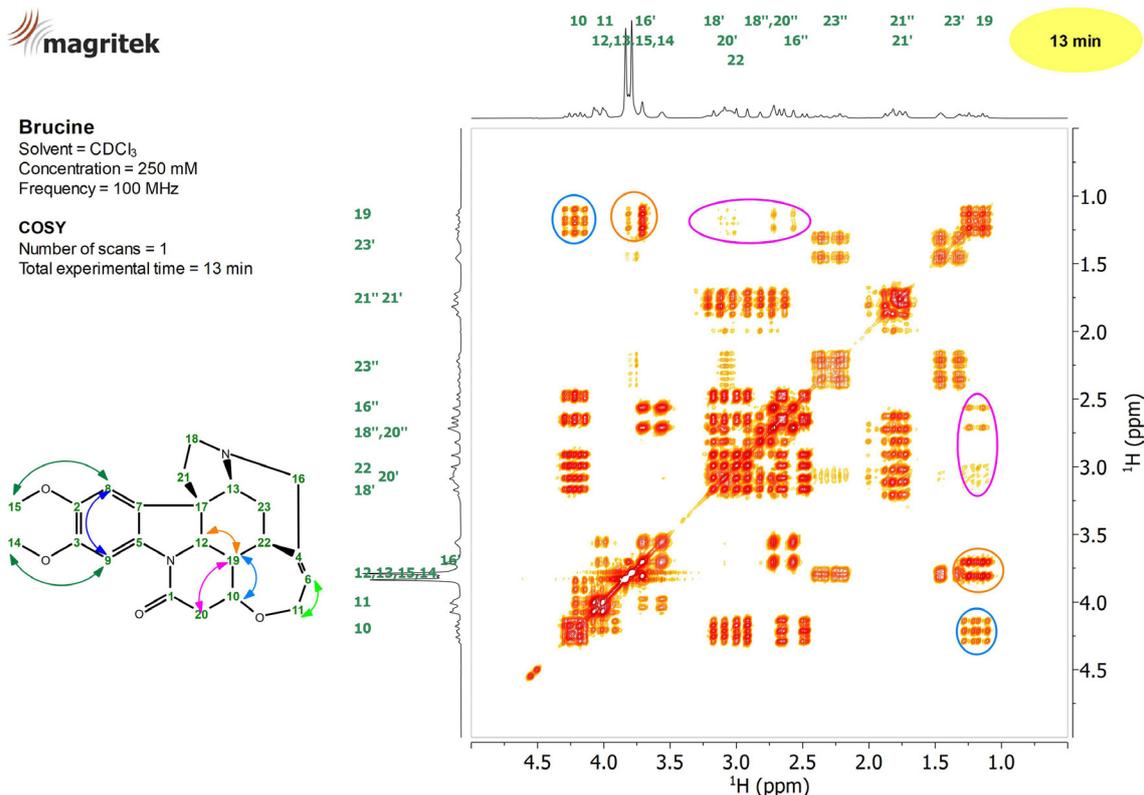
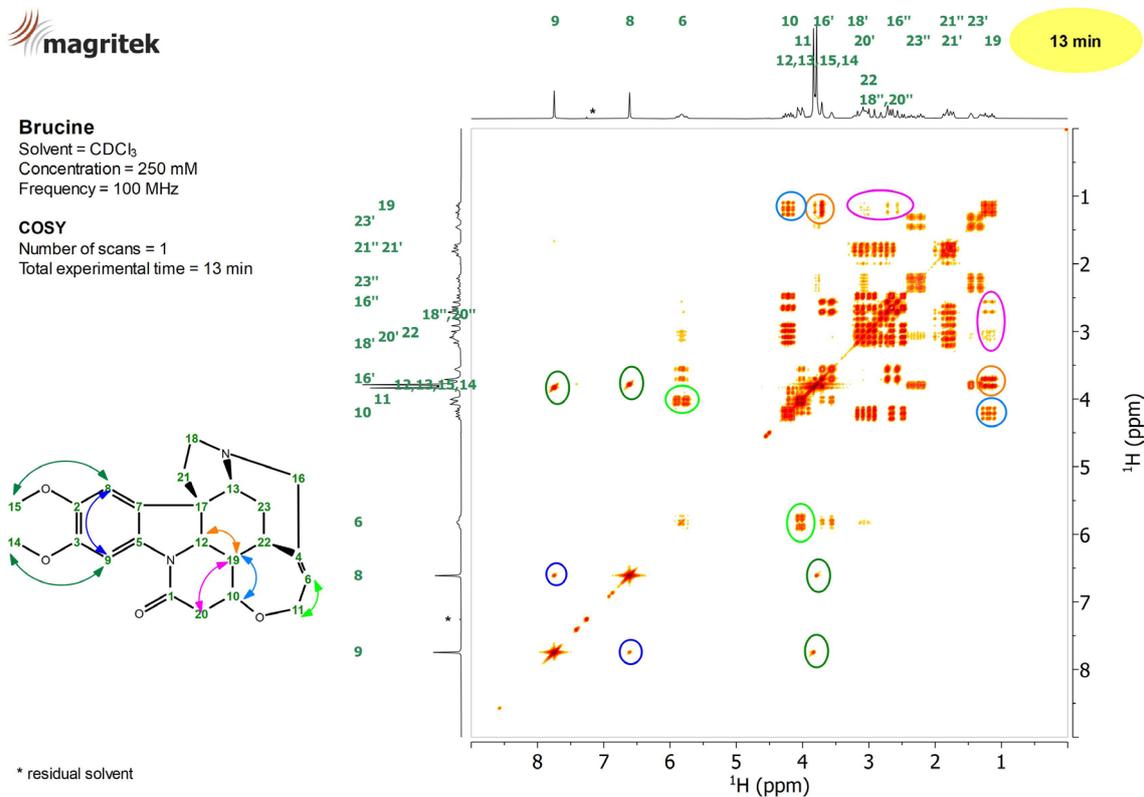


Figure 3:  $^1\text{H}$  2D COSY experiment of a 250 mM Brucine sample in  $\text{CDCl}_3$  acquired in 13 minutes on a Spinsolve 100 MHz system (top); zoom into the aliphatic region (0.5-5.0 ppm) of the  $^1\text{H}$  2D COSY spectrum which underlines the superb resolution.

## 2D JRES spectrum

This experiment is useful to identify the chemical groups generating a single line for each group by collapsing the J-coupling along the direct direction. The multiplets are generated along the vertical direction.

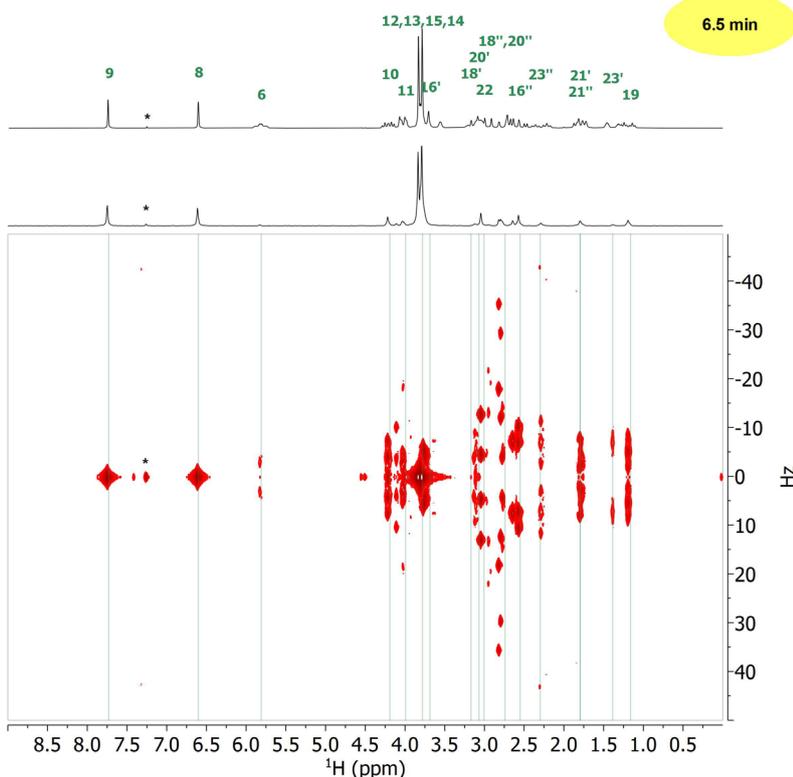
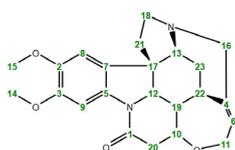


### Brucine

Solvent = CDCl<sub>3</sub>  
Concentration = 250 mM  
Frequency 1H = 100 MHz

### JRES

Number of scans = 1  
Total experimental time = 6.5 min



\* residual solvent

Figure 4: Homonuclear J-resolved (JRES) spectrum of 250 mM Brucine in CDCl<sub>3</sub> on a Spinsolve 100 MHz.

## 2D HSQC-ME

The HSQC is a powerful sequence widely used to correlate the <sup>1</sup>H with the one-bond coupled <sup>13</sup>C nuclei. 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<sub>2</sub> groups (blue) from the CH and CH<sub>3</sub> groups (red). Figure 5 shows the HSQC-ME spectrum of a 250 mM Brucine sample in CDCl<sub>3</sub> acquired in 1 minute. The measurement time was optimized applying NUS (non uniform sampling).

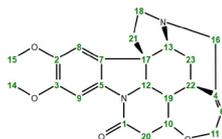


### Brucine

Solvent = CDCl<sub>3</sub>  
Concentration = 250 mM  
Frequency 1H = 100 MHz

### HSQC-ME

Number of scans = 2  
Repetition time = 0.5 s  
Number of steps = 256  
NUS = 25%  
Total experimental time = 1 min



Red = CH and CH<sub>3</sub>

Blue = CH<sub>2</sub>

\* residual solvent

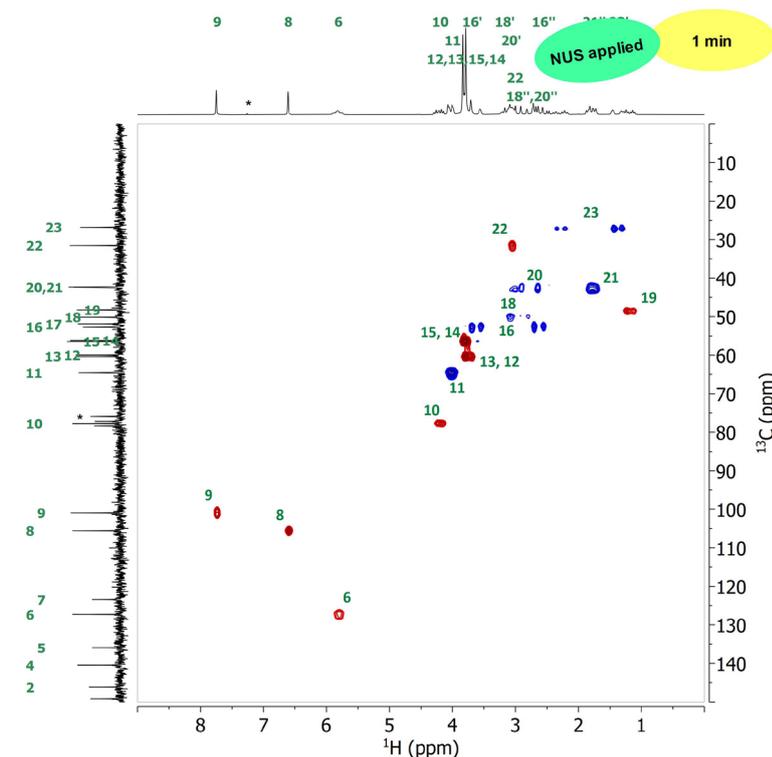


Figure 5: HSQC-ME spectrum of a 250 mM Brucine sample in CDCl<sub>3</sub> showing the correlation between the <sup>1</sup>H (horizontal) and <sup>13</sup>C (vertical) signals.

## 2D HMBC

To obtain long-range  $^1\text{H}$ - $^{13}\text{C}$  correlations through two or three bond couplings, the Heteronuclear Multiple Bond Correlation (HMBC) experiment can be used. Figure 6 shows the long-range correlation of proton 8 with carbons 2, 3, 5, 7, 9 and 17 (the sequence shows the correlation with quaternary carbons, too).

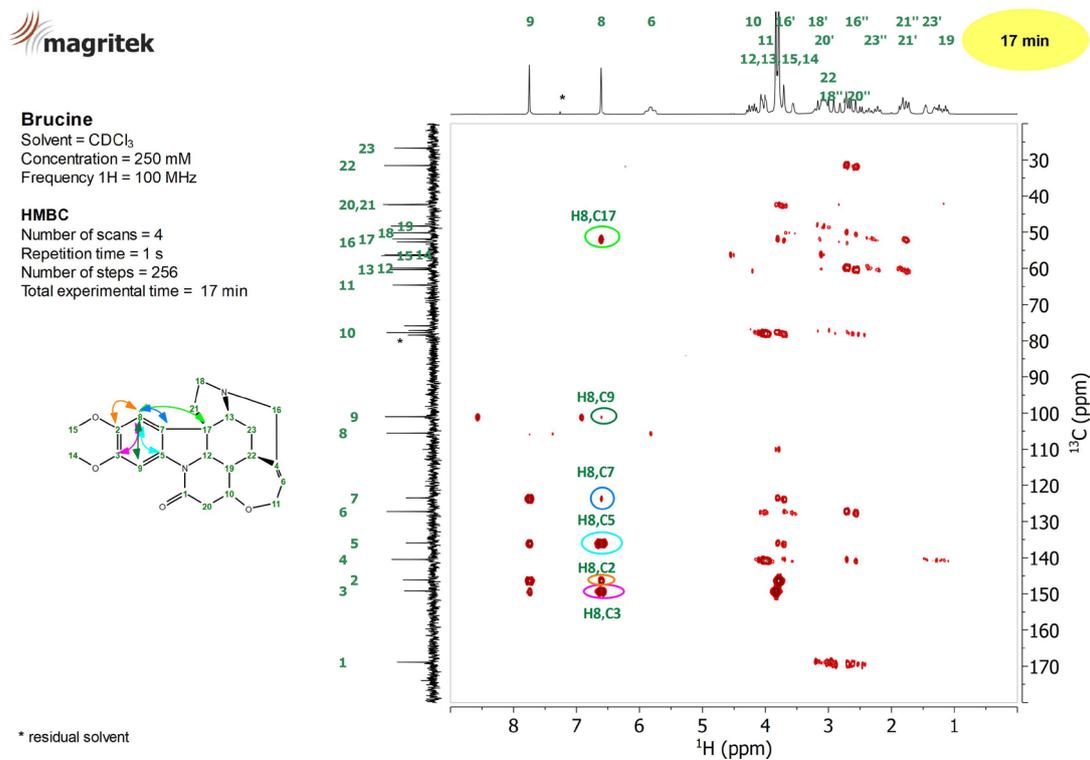


Figure 6: HMBC spectrum of a 250 mM Brucine sample in  $\text{CDCl}_3$  showing the long-range couplings between  $^1\text{H}$  and  $^{13}\text{C}$  nuclei.