

Methyl 1H-indole-3-carboxylate Spinsolve 80

¹H Spectrum

The ¹H NMR spectrum of 250 mM Methyl 1H-indole-3-carboxylate in DMSO-*d*₆ measured in a single scan taking 10 s to acquire is shown in Figure 1.

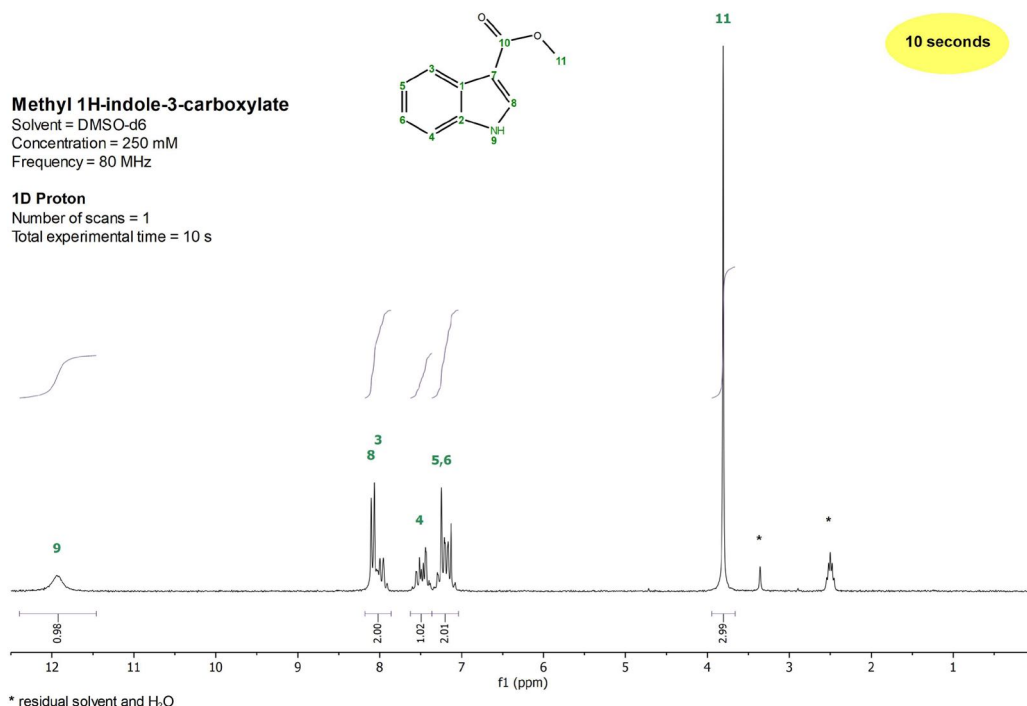


Figure 1: ¹H NMR spectrum of 250 mM Methyl 1H-indole-3-carboxylate in DMSO-*d*₆ measured on a Spinsolve Carbon 80 MHz in a single scan.

¹³C Spectra

Figure 2 shows the ¹³C NMR spectra of 0.8 M Methyl 1H-indole-3-carboxylate in DMSO-*d*₆ acquired using NOE and DEPT polarization transfer from ¹H to ¹³C and ¹H decoupling. The 1D Carbon experiment using NOE (top spectrum) is sensitive to all ¹³C nuclei in the sample. It clearly resolves the 10 expected resonances. The DEPT experiments show only ¹³C nuclei directly attached to ¹H and can be used for spectral editing. Since the peaks at 165, 136, 126 and 107 ppm do not show in the DEPT spectra they must belong to quaternary carbons. The DEPT-90 experiment gives only signal from CH groups, whilst the DEPT-45 and DEPT-135 give signals of CH, CH₂ and CH₃ groups, but the CH₂ groups appear as negative peaks in the DEPT-135.

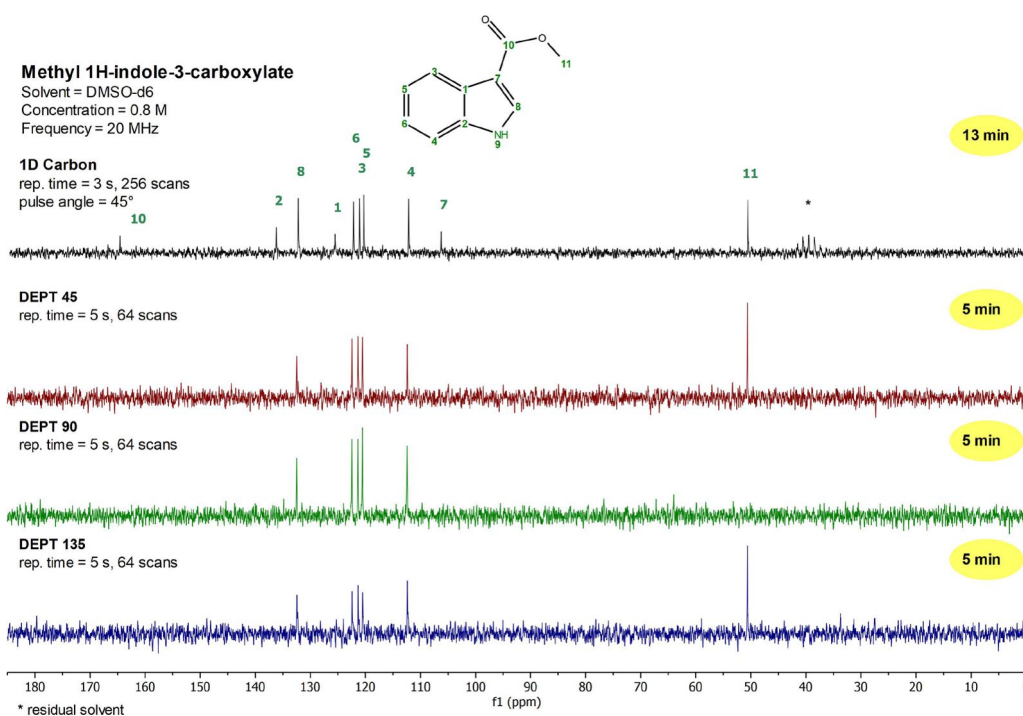


Figure 2: Carbon NMR spectra of 0.8 M Methyl 1H-indole-3-carboxylate in DMSO-*d*₆ measured on a Spinsolve Carbon 80 MHz using NOE (top) and DEPT 45, 90, 135 sequences.

2D COSY

The 2D COSY experiment allows one to identify coupled ^1H nuclei as they generate cross peaks out of the diagonal of the 2D data set. For example, the proton at position 8 couples to the NH group at position 9 (orange), whilst the aromatic protons at positions 3, 4, 5 and 6 couple to each other (green, cyan and blue).

Methyl 1H-indole-3-carboxylate

Solvent = DMSO- d_6
Concentration = 250 mM
Frequency = 80 MHz

COSY

Number of scans = 1
Total experimental time = 10 min

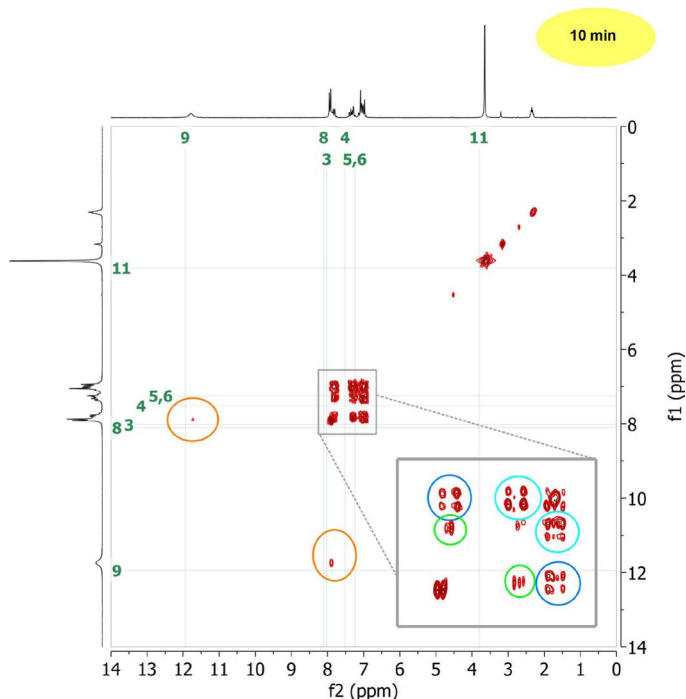
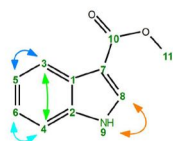


Figure 3: COSY experiment of a 250 mM sample of Methyl 1H-indole-3-carboxylate in DMSO- d_6 acquired in 10 minutes on a Spinsolve Carbon 80 MHz.

2D J-Resolved

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.

Methyl 1H-indole-3-carboxylate

Solvent = DMSO- d_6
Concentration = 250 mM
Frequency = 80 MHz

JRES

Number of scans = 1
Total experimental time = 9 min

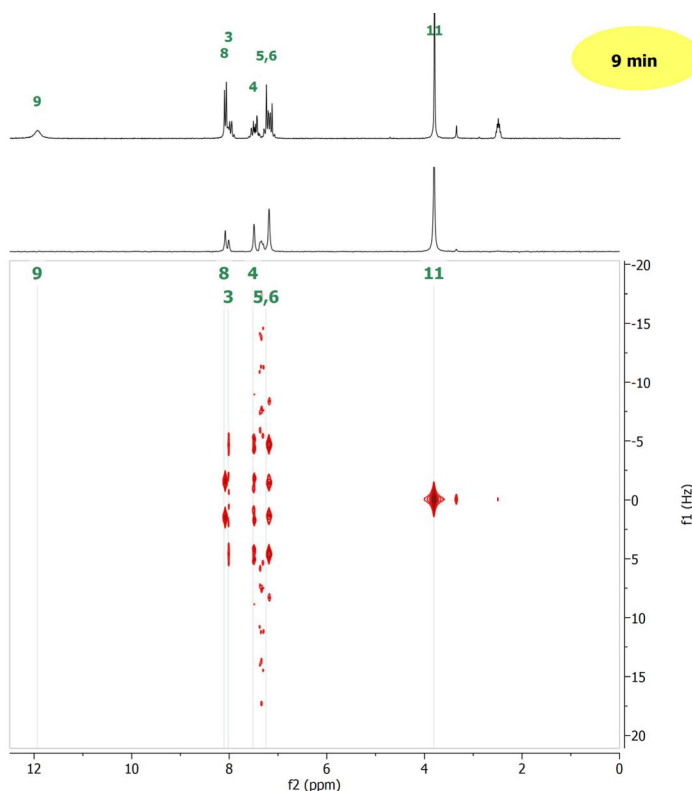
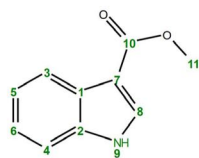


Figure 4: Homonuclear J-resolved spectrum of 250 mM Methyl 1H-indole-3-carboxylate in DMSO- d_6 acquired in 10 minutes on a Spinsolve Carbon 80 MHz.

HSQC-ME

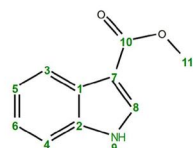
The HSQC is a powerful sequence widely used to correlate the ^1H with the one-bond coupled ^{13}C nuclei. The Spinsolve is equipped with a multiplicity edited version (HSQC-ME) of this method. It provides the editing power of the DEPT-135 sequence, which is useful to identify the signal of the CH_2 groups (blue) from the CH and CH_3 (red). Figure 5 shows the HSQC-ME spectrum of 0.8 M Methyl 1H-indole-3-carboxylate in $\text{DMSO}-d_6$ acquired in 17 minutes.

Methyl 1H-indole-3-carboxylate

Solvent = CDCl_3
Concentration = 0.8 M
Frequency ^1H = 80 MHz

HSQC-ME

Number of scans = 4
Repetition time = 2 s
Number of steps = 64
Total experimental time = 17 min



Red = CH and CH_3

Blue = CH_2

* residual solvent

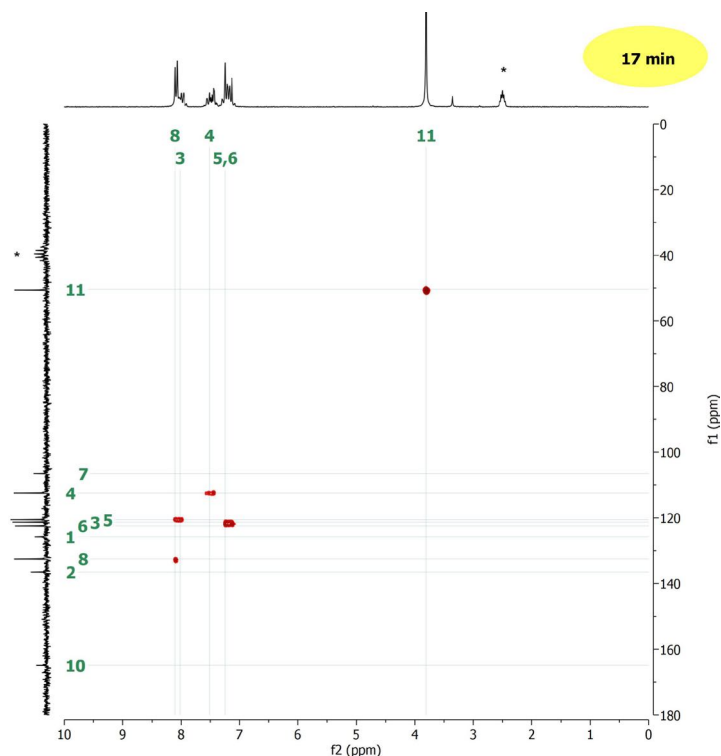


Figure 5: HSQC-ME spectrum of 0.8 M Methyl 1H-indole-3-carboxylate in $\text{DMSO}-d_6$ measured on a Spinsolve Carbon 80 MHz showing the correlation between the ^1H (horizontal) and ^{13}C (vertical) signals.

HMBC

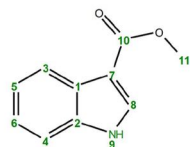
To obtain long-range ^1H - ^{13}C correlations through two or three bond couplings, the Heteronuclear Multiple Bond Correlation (HMBC) experiment can be used. Figure 6 shows, as an example, the long-range correlation of protons 11 with the carbon 10 (the sequence shows the correlation with quaternary carbons too). At the same time, it can be noticed that there is no correlation peak at the position of carbon 11, because there is no proton within four bonds to this carbon. The same exercise can be repeated for each proton signal along the horizontal scale to identify to which carbons they are long-range coupled.

Methyl 1H-indole-3-carboxylate

Solvent = $\text{DMSO}-d_6$
Concentration = 0.8 M
Frequency ^1H = 80 MHz

HMBC

Number of scans = 8
Repetition time = 2 s
Number of steps = 256
Total experimental time = 69 min



* residual solvent

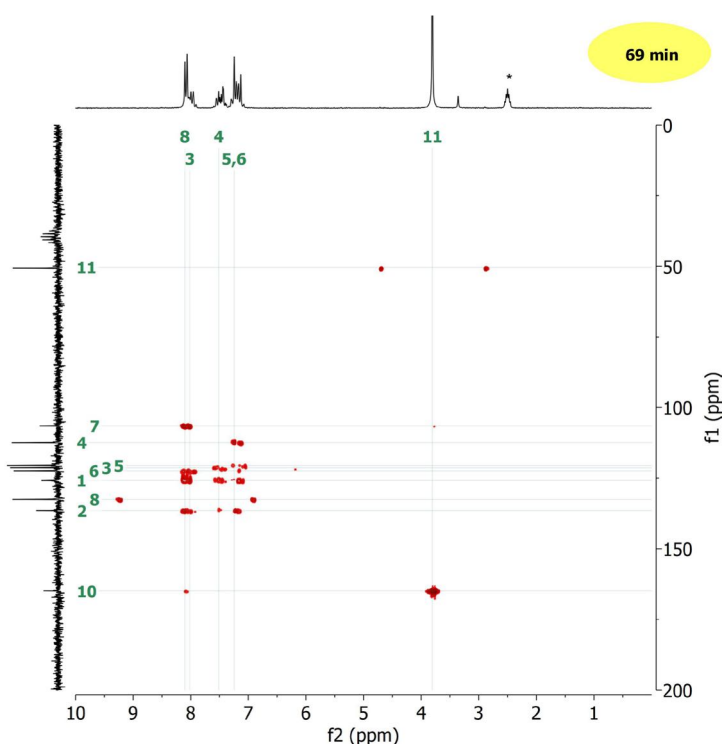


Figure 6: HMBC spectrum of 0.8 M Methyl 1H-indole-3-carboxylate in $\text{DMSO}-d_6$ measured on a Spinsolve Carbon 80 MHz showing the long-range couplings between the ^1H (horizontal) and ^{13}C (vertical) signals.

T_1 proton relaxation

This experiment is useful to measure the T_1 relaxation time of each chemical group. Figure 7 shows the T_1 build up curves for the different protons (color coded) in Methyl 1H-indole-3-carboxylate. The T_1 values obtained by fitting the build-up curves with single exponential functions are shown next to the build-up curves. The remarkable quality of the fits demonstrates the high signal-to-noise and reproducibility of the Spinsolve spectrometer. Note that for the methyl group H11 bi-exponential behavior could be observed due to internal rotation and reorientation as known from literature.^[1]

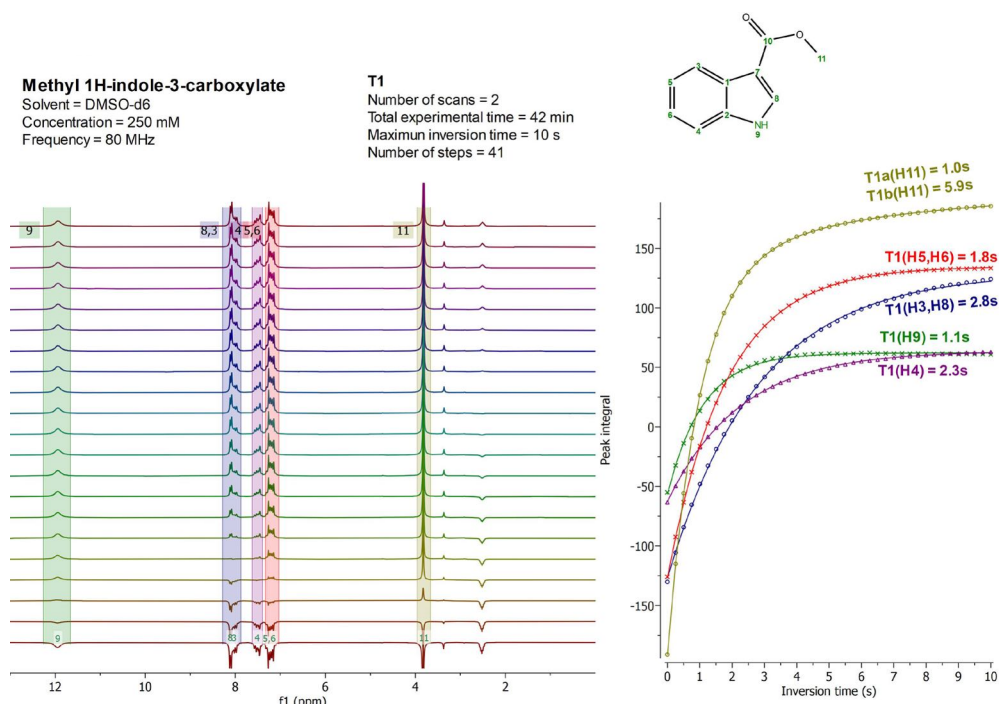


Figure 7: Proton T_1 relaxation measurement done on 250 mM Methyl 1H-indole-3-carboxylate dissolved in DMSO- d_6 with a Spinsolve 80 MHz system.

[1] Blicharska, B.; Hertz, H. G.; Versmold, H. *J. Magn. Resonance* 1979, 33, 531.

T_2 proton relaxation

This experiment uses a CPMG sequence to allow the protons to relax with the transverse relaxation time, T_2 , and acquires only the signals during the last echo. To acquire the full data set it is necessary to repeat the experiment incrementing the duration of the CPMG module by increasing the number of echoes generated during this period. The T_2 values are obtained by fitting the peak integrals of each group as a function of the CPMG evolution time. The figure shows the T_2 decay curves for the different protons in Methyl 1H-indole-3-carboxylate (color coded). Note that for the methyl group H11 bi-exponential behavior could be observed due to internal rotation and reorientation as known from literature.^[1]

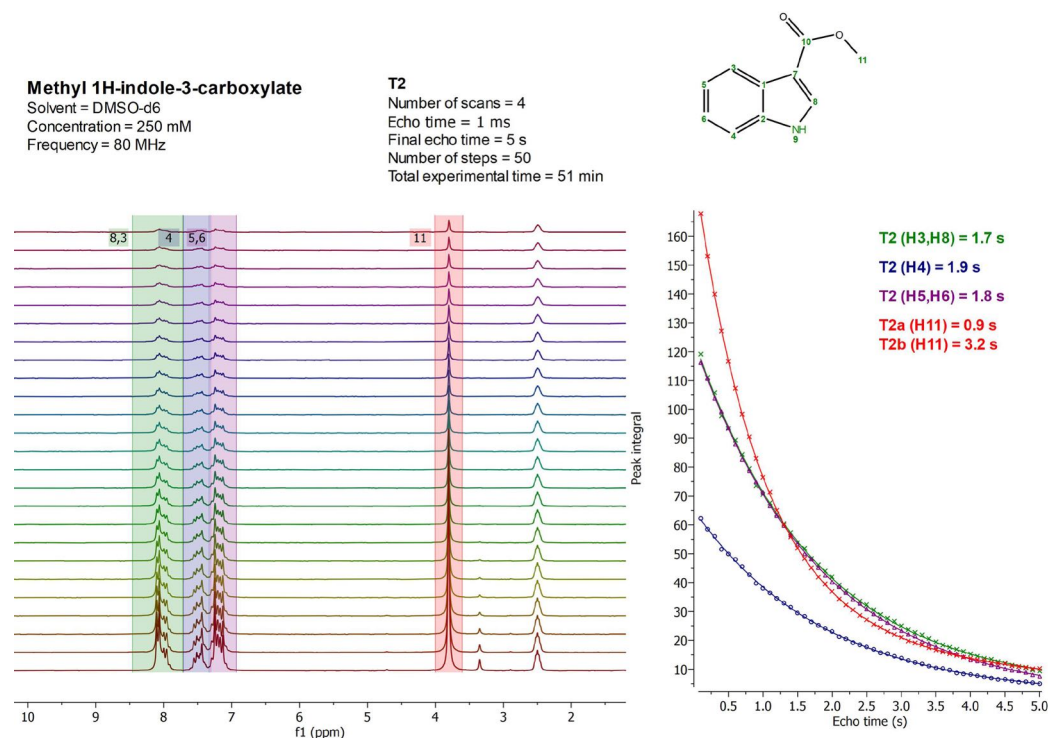


Figure 8: Proton T_2 relaxation curves measured for 250 mM Methyl 1H-indole-3-carboxylate dissolved in DMSO- d_6 with a Spinsolve 80 MHz system.

[1] Blicharska, B.; Hertz, H. G.; Versmold, H. *J. Magn. Resonance* 1979, 33, 531.