Ethyl crotonate

Spinsolve

^{1}H Spectra

Ethyl crotonate ($C_6H_{10}O_2$) is a colourless liquid at room temperature typically used as a solvent for cellulose esters as well as a plasticizer for acrylic resins. Figure 1 shows the ^{1}H NMR spectrum of 250 mM Ethyl crotonate in $CDCl_3$ measured in a single scan taking 15 seconds to acquire. The high resolution of the Spinsolve spectrometer can be particularly well appreciated in the multiplets of groups 1 and 3, where the small J-coupling between these groups can be well resolved.

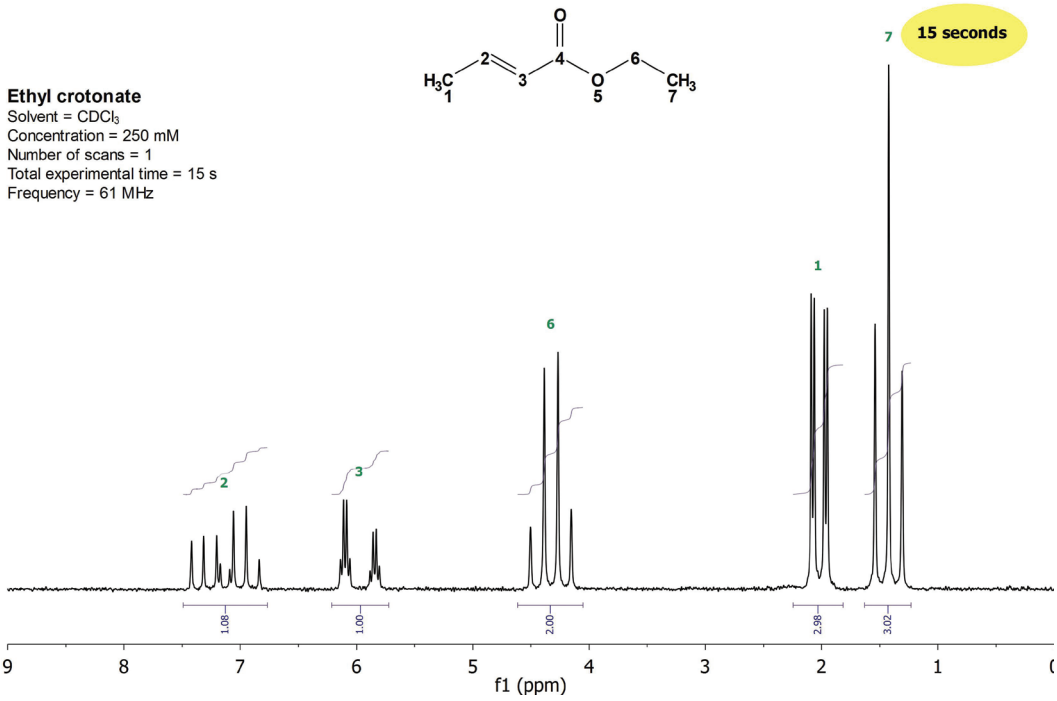

Figure 1: ^{1}H NMR spectrum of 250 mM Ethyl crotonate in $CDCl_3$ measured on a Spinsolve 60 MHz in a single scan. The integrals of the peaks correspond to the number of protons in the groups with very high accuracy.

^{13}C Spectra

Figure 2 shows the ^{13}C NMR spectra of 1 M Ethyl Crotonate in $CDCl_3$ acquired using NOE and DEPT polarization transfer from ^{1}H to ^{13}C and ^{1}H decoupling. The 1D Carbon experiment using NOE (top spectrum) is sensitive to all ^{13}C nuclei in the sample. It clearly resolves the 6 expected resonances. The DEPT experiments show only ^{13}C nuclei directly attached to ^{1}H and can be used for spectral editing. Since the peak at 167 ppm does not show in the DEPT spectra it must correspond to the quaternary carbon. The DEPT-90 experiment gives only signal from CH groups, whilst the DEPT-45 and DEPT-135 give signals of CH, CH_2 and CH_3 groups, but the CH_2 groups appear as negative peaks in the DEPT-135.

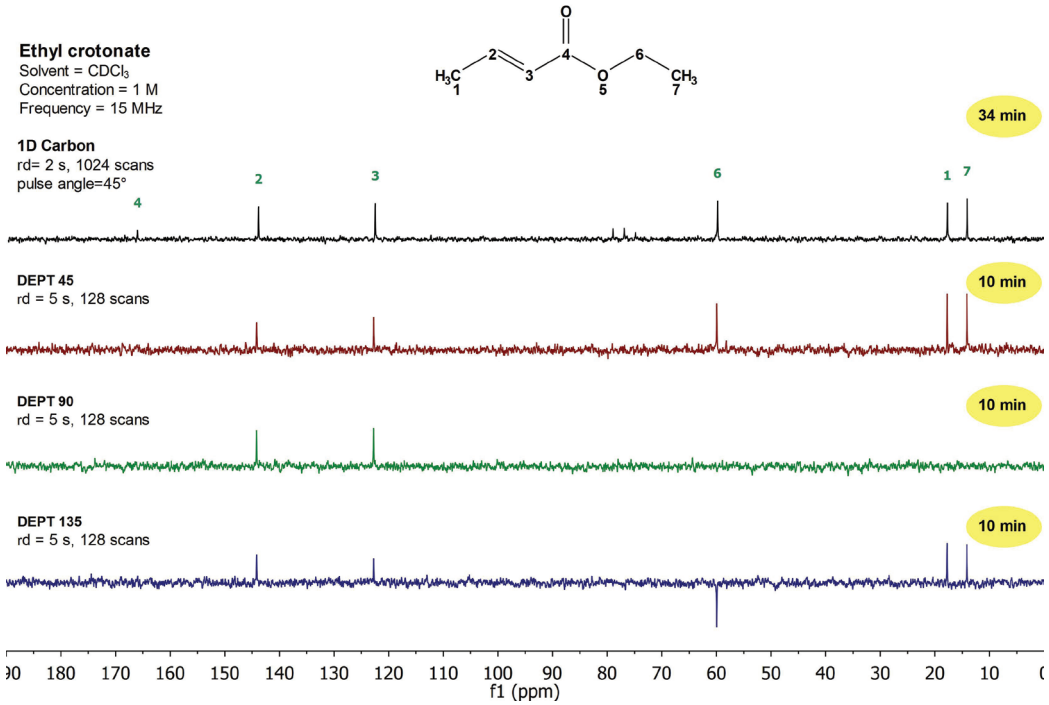

Figure 2: Carbon NMR spectra of 1 M Ethyl crotonate in $CDCl_3$ measured on a Spinsolve 60 MHz using NOE (top) and DEPT 45, 90 and 135 sequences.

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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. For example, the methyl group at position 1 couples to the CH group at position 2 (blue) and 3 (green).On the other hand, the methyl group at position 7 couples to the CH_2 group at positions 6 (violet). Furthermore, the coupling between the neighbour CH groups 2 and 3 is marked in red.

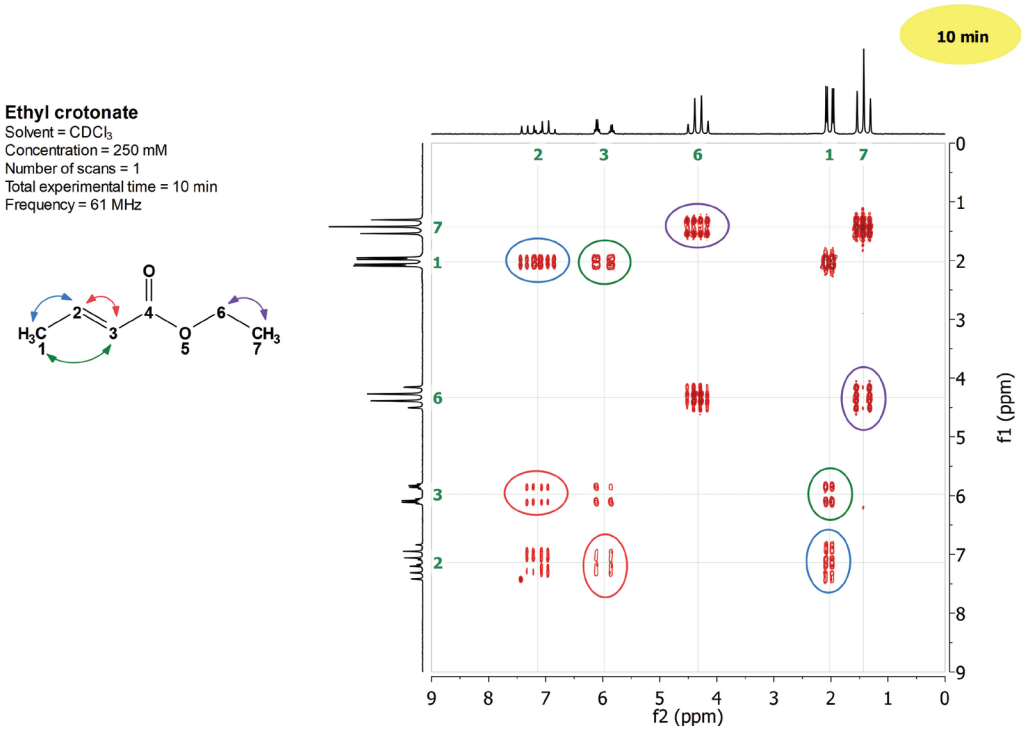

Figure 3: COSY experiment of a 250 mM Ethyl crotonate sample acquired in 10 minutes on a Spinsolve 60.

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.

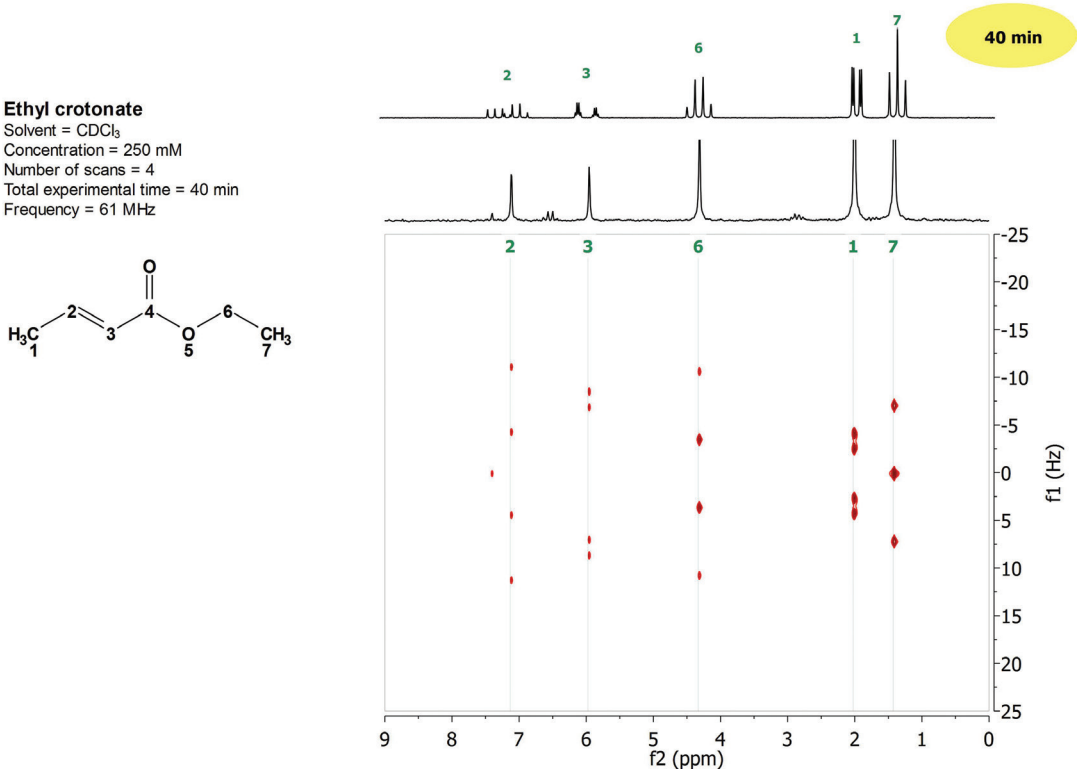

Figure 4: Homonuclear J-resolved spectrum of 250 mM Ethyl crotonate in $CDCl_3$.

HSQC-ME

The HSQC is a powerful sequence widely used to correlate the ^{1}H 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 1 M Ethyl crotonate in $CDCl_3$ acquired in 35 minutes.

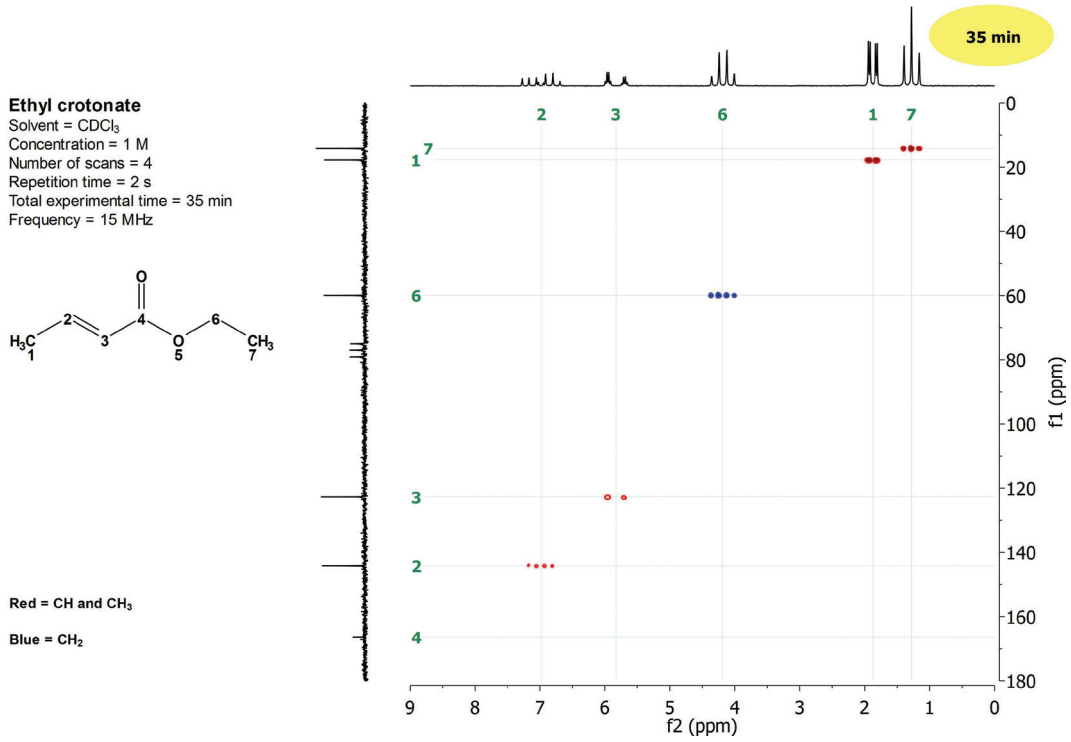

Figure 5: HSQC-me spectrum of 1 M Ethyl crotonate showing the correlation between the ^{1}H (horizontal) and ^{13}C (vertical) signals.

HMBC

To obtain long-range ^{1}H-^{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 6 with carbons 4 and 7 (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 6. The same exercise can be repeated for each proton signal along the horizontal scale to identify which carbon are long-range coupled.

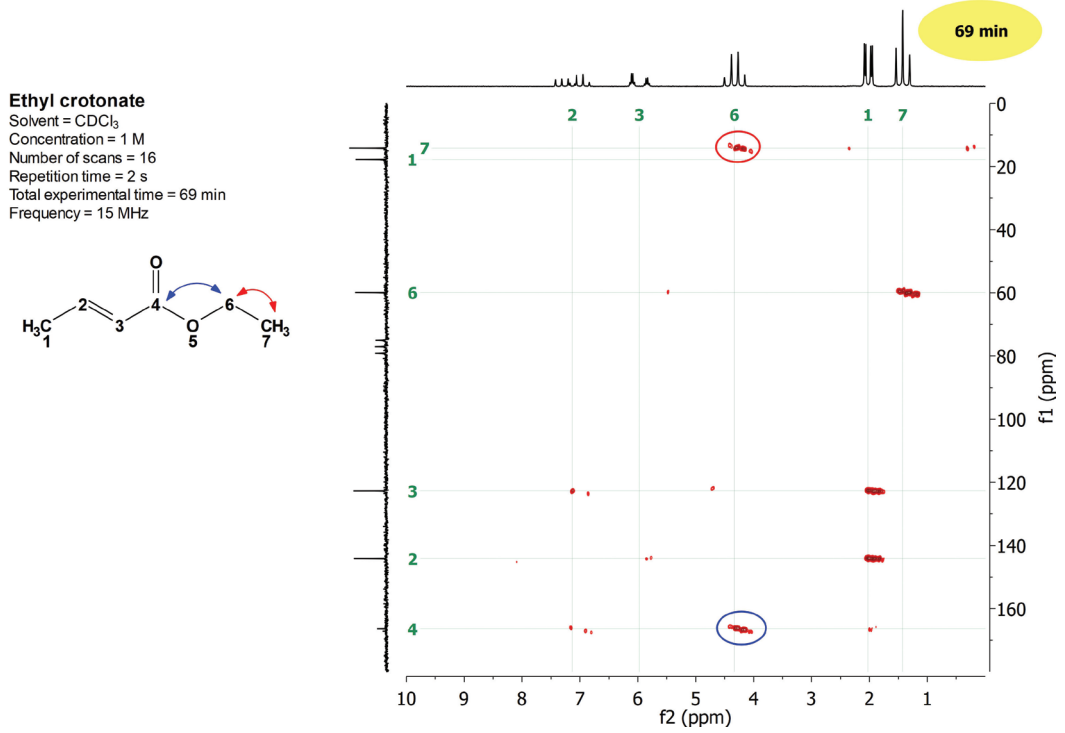

Figure 6: HMBC spectrum of 1 M Ethyl crotonate showing the long range couplings between ^{1}H and ^{13}C nuclei.

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 Ethyl crotonate. 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 demonstrate the high signal-to-noise and reproducibility of the Spinsolve spectrometer.

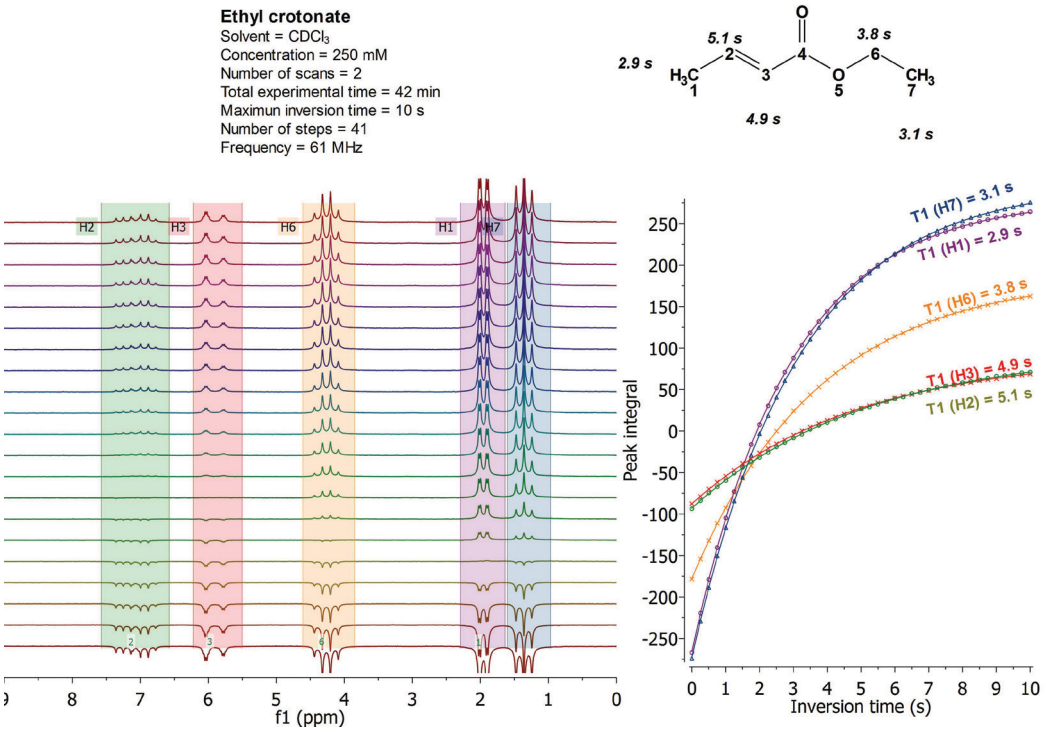

Figure 7: Proton T_1 relaxation measurement done on 250 mM Ethyl crotonate dissolved in $CDCl_3$.

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. Figure 8 shows the T_2 decay curves for the different protons in Ethyl crotonate (color coded).

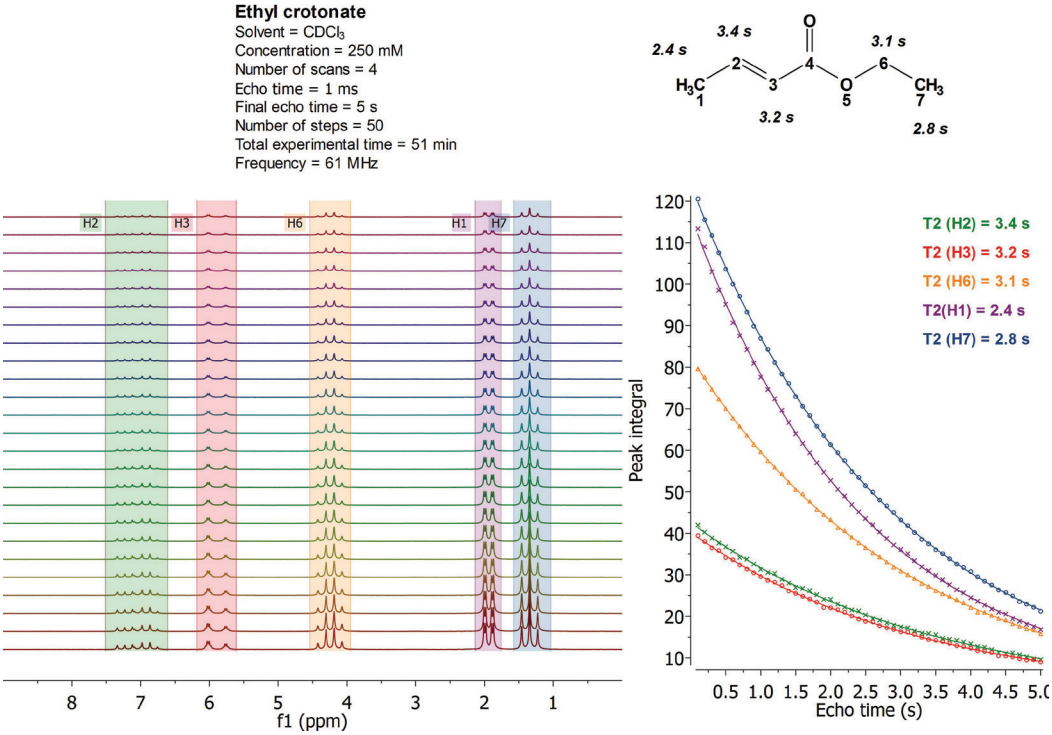

Figure 8: Proton T_2 relaxation curves measured for 250 mM Ethyl crotonate dissolved in $CDCl_3$.