Isoamyl acetate

Spinsolve 80

^{1}H Spectrum

Isoamyl acetate ($C_7H_{14}O_2$) is mostly used as an artificial food flavor which smells similar to banana and pear. The ^{1}H NMR spectrum of 250 mM Isoamyl acetate in $CDCl_3$ measured in a single scan taking 10 seconds to acquire is shown in Figure 1. All peaks and ^{1}H-^{1}H couplings are well resolved and the integrals of the peaks correspond to the number of protons in the groups with very high accuracy.

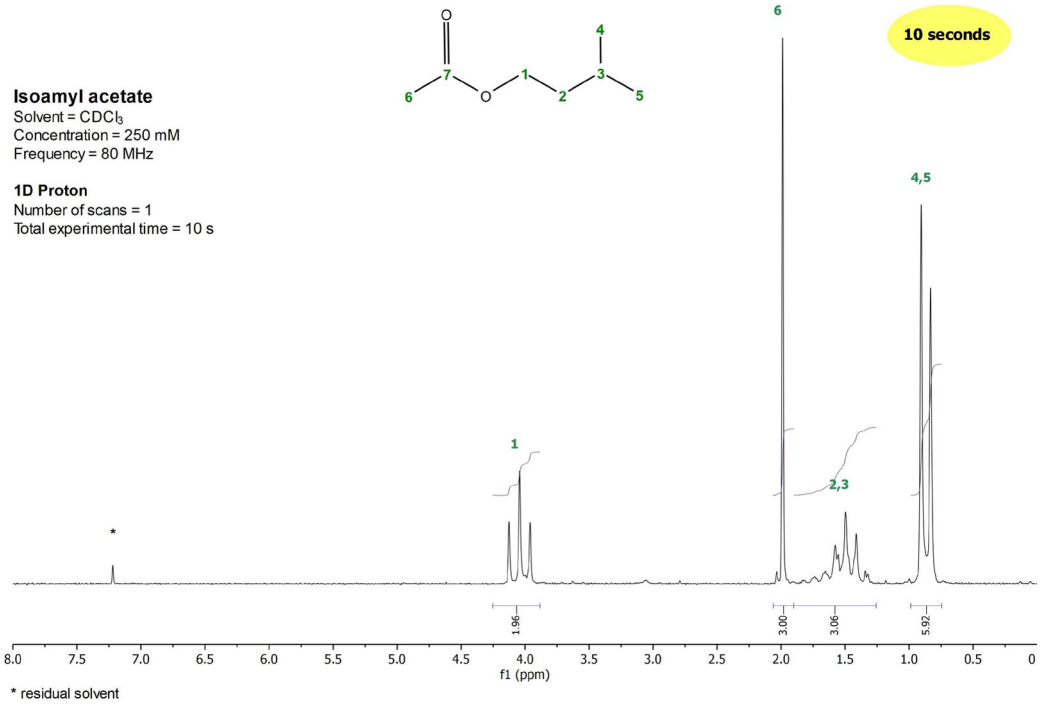

Figure 1: ^{1}H NMR spectrum of 250 mM Isoamyl acetate in $CDCl_3$ measured on a Spinsolve 80 MHz in a single scan.

^{13}C Spectra

Figure 2 shows the ^{13}C NMR spectra of 1 M Isoamyl acetate 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 171 ppm does not show in the DEPT spectra it must belong to a 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.

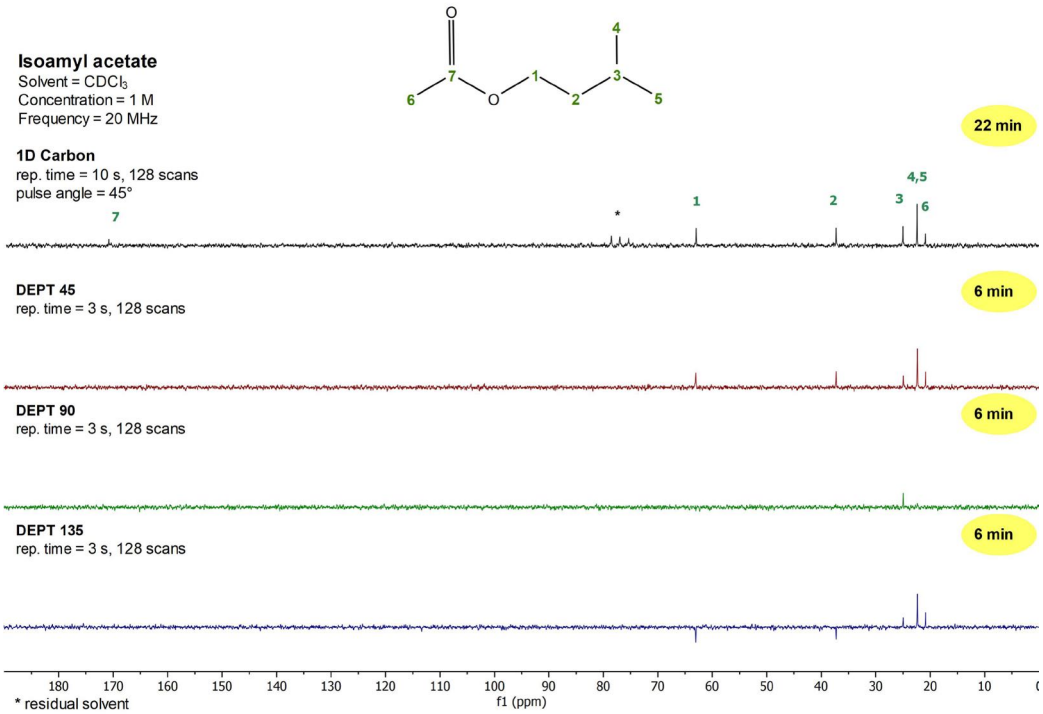

Figure 2: Carbon NMR spectra of 1 M Isoamyl acetate in $CDCl_3$ measured on a Spinsolve 80 MHz using NOE (top) and DEPT 45, 90 and 135 sequences.

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2D COSY

The 2D COSY experiment allows one to identify coupled ^1H nuclei as they generate cross peaks out of the diagonal of the 2D data set. Marked in blue are highlighted the couplings between the CH proton at positions 3 with the CH_3 protons in positions 4 and 5. In addition the cross peaks between the CH_2 protons in positions 1 and 2 can be observed (orange).

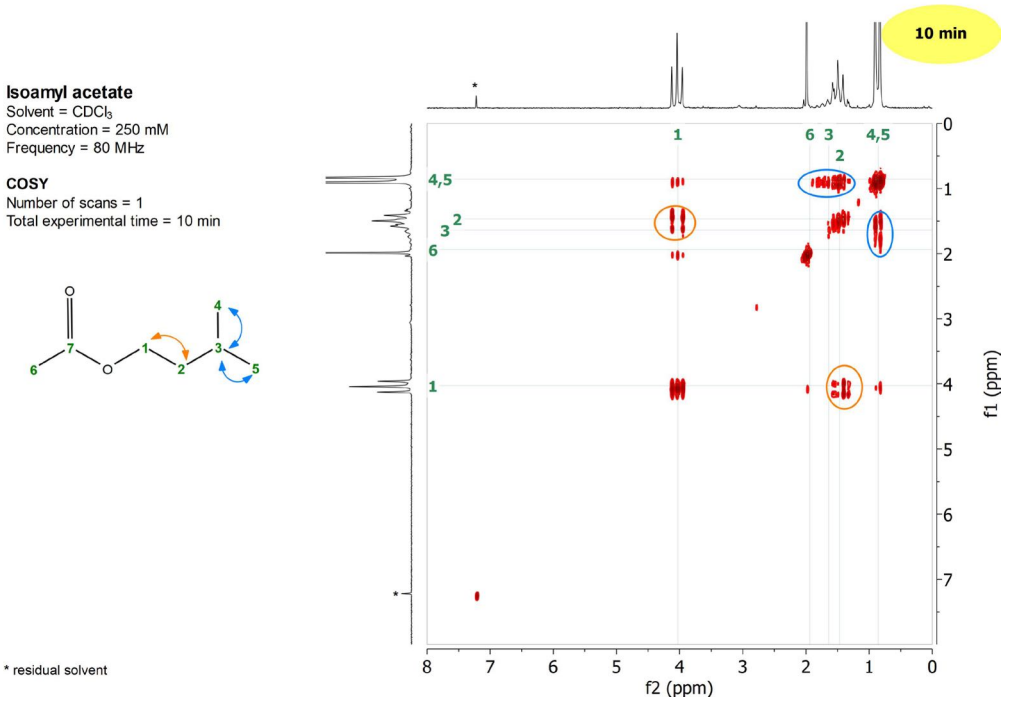

Figure 3: COSY experiment of a 250 mM Isoamyl acetate sample acquired in 10 minutes on a Spinsolve 80 MHz system.

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.

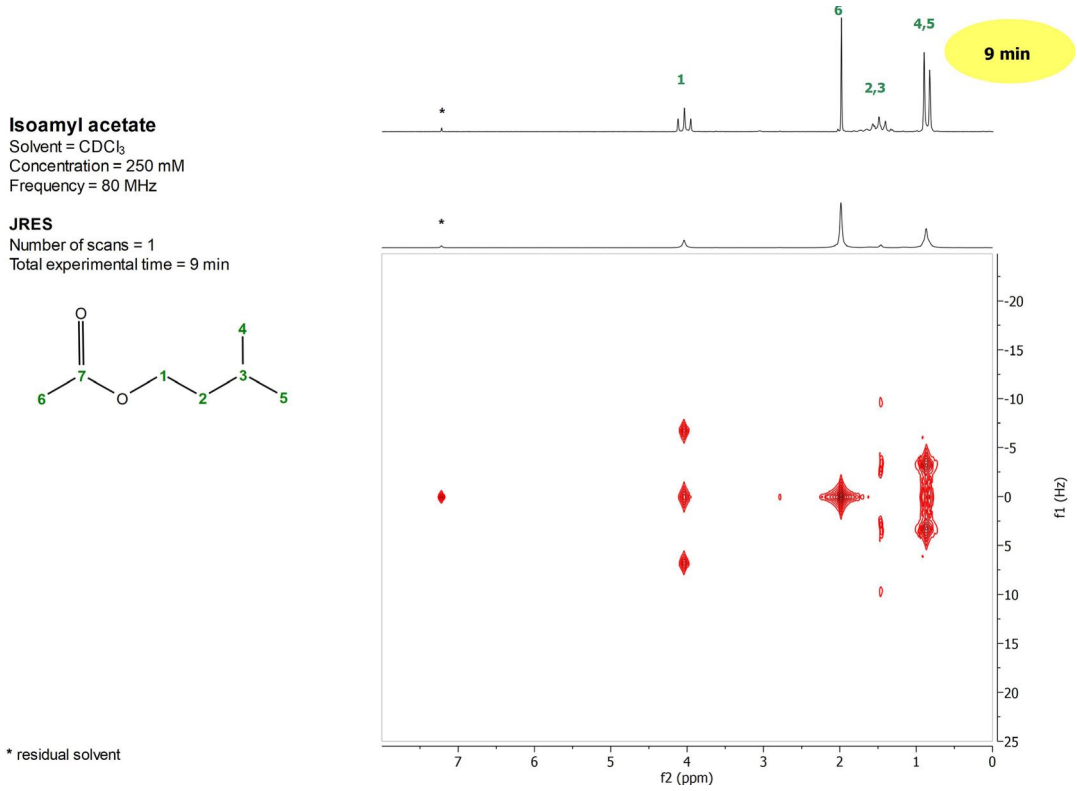

Figure 4: Homonuclear J-resolved spectrum of 250 mM Isoamyl acetate in $CDCl_3$ on a 80 MHz system.

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 Isoamyl acetate in $CDCl_3$ acquired in 35 minutes.

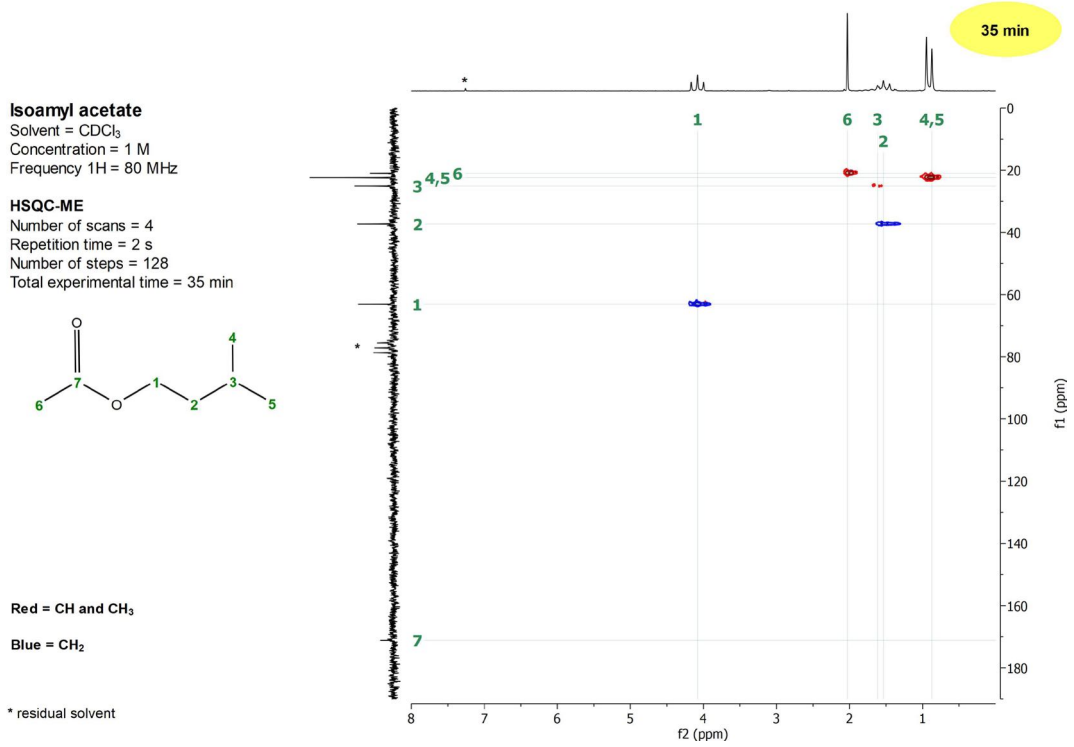

Figure 5: HSQC-ME spectrum of 1 M Isoamyl acetate showing the correlation between the ^{1}H (horizontal) and ^{13}C (vertical) signals on a Spinsolve 80 MHz.

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 1 with carbons 2, 3 and 7 (the sequence shows the correlation with quaternary carbons too). Here it is to note that there is ^{3}J correlation with carbon 3 (purple) and carbon 7 (green) as well as ^{2}J correlation with carbon 2 (light blue). The same exercise can be repeated for each proton signal along the horizontal scale to identify which carbon are long-range coupled.

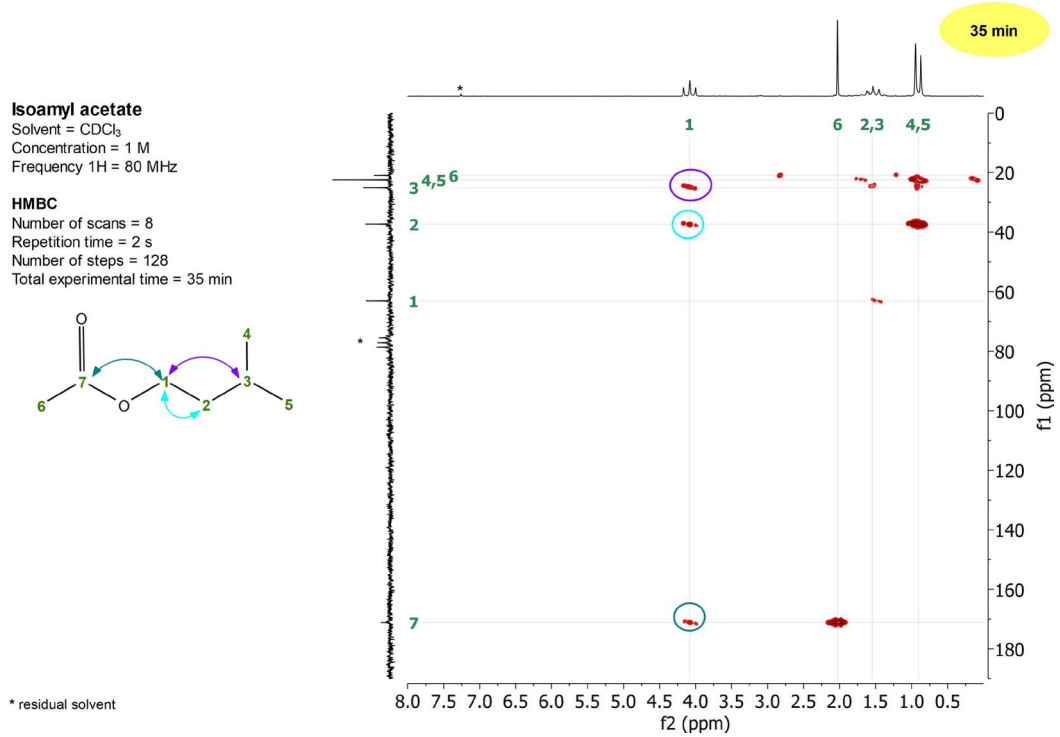

Figure 6: HMBC spectrum of 1 M Isoamyl acetate on a Spinsolve 80 MHz showing the long range couplings between ^{1}H and ^{13}C nuclei.

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 Isoamyl acetate. 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.

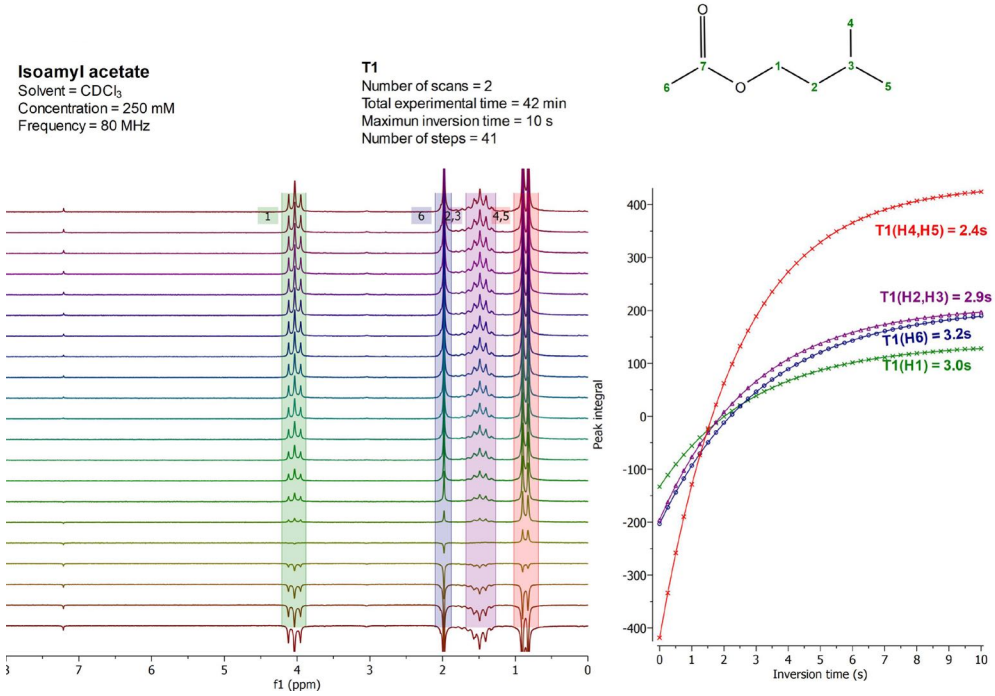

Figure 7: Proton T_1 relaxation measurement done on 250 mM Isoamyl acetate dissolved in $CDCl_3$ with a Spinsolve 80 MHz.

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 Isoamyl acetate (color coded).

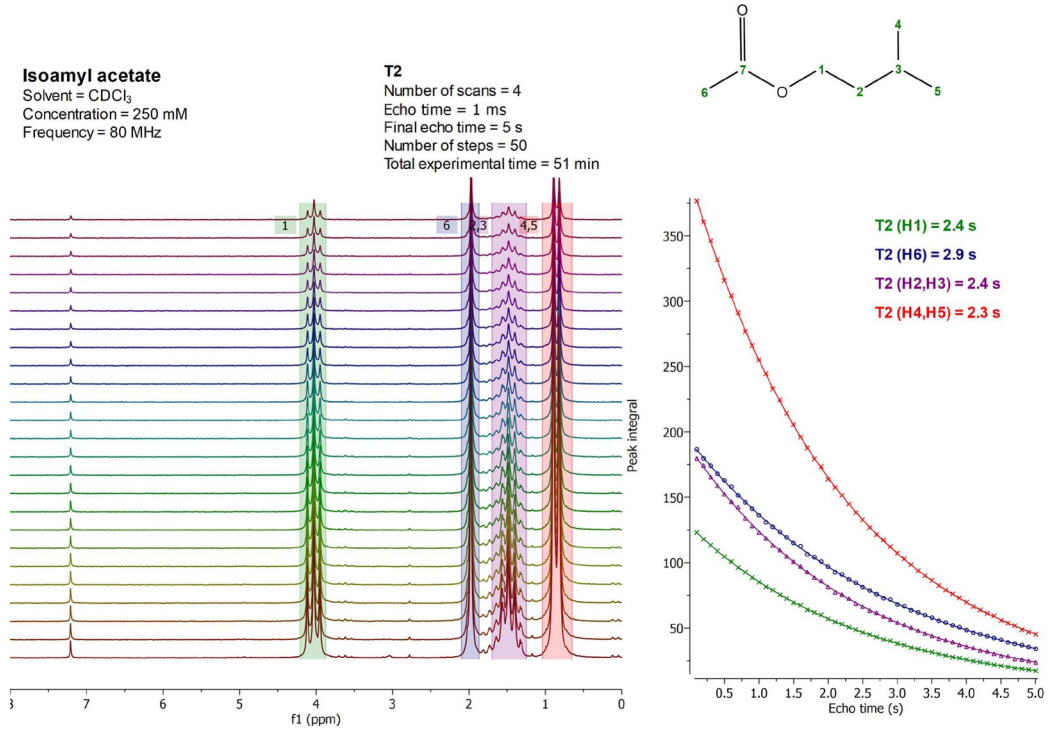

Figure 8: Proton T_2 relaxation curves measured for 250 mM Isoamyl acetate dissolved in $CDCl_3$ with a Spinsolve 80 MHz system.