Diethyl phtalate

Diethylphtalate ($C_{12}H_{14}O_4$) is used as a plasticizer, detergent base, solvent and aerosol spray in hundreds of products, such as plastic packaging films, cosmetic formulations, toys and personal care products. Figure 1 shows the $^1$H NMR spectrum of 250 mM diethylphtalate in CDCl$_3$ measured in a single scan taking 10 seconds to acquire. The high resolution of the Spinsolve spectrometer can be particularly well appreciated in the multiplets of the ethyl group (9-12), where the small J-coupling between the methyl and the methylene can be well resolved.

![H NMR spectrum of 250 mM diethylphtalate in CDCl$_3$](image)

Figure 1: $^1$H NMR spectrum of 250 mM diethylphtalate in CDCl$_3$ measured on a Spinsolve 80 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 diethylphtalate 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 molecule is symmetric). The DEPT experiments show only $^{13}$C nuclei directly attached to $^1$H and can be used for spectral editing. Since the peaks at 132 and 167 ppm do not show in the DEPT spectra, they must correspond to quaternary carbons 7,8 and 2,3. The DEPT-90 experiment gives only signals 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.

![Carbon NMR spectra of 1 M diethylphtalate in CDCl$_3$](image)

Figure 2: Carbon NMR spectra of 1 M diethylphtalate in CDCl$_3$ measured on a Spinsolve 80 MHz using NOE (top) and DEPT 45, 90, and 135 sequences.
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 at position 9 (or 11) couples to the methylene at position 10 (or 12).

![COSY experiment of a 250 mM diethylphthalate sample acquired in 10 minutes on a Spinsolve 80 MHz.](image)

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.

![J-Resolved experiment of a 250 mM diethylphthalate sample acquired in 9 minutes on a Spinsolve 80 MHz.](image)
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 diethylphthalate in CDCl$_3$ acquired in 18 minutes.

**HSQC-ME**

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 proton 9 and 11 with carbons 10 and 12 (the sequence shows the correlation with quaternary carbons too). The same exercise can be repeated for each proton signal along the horizontal scale to identify which carbon are long-range coupled.

**HMBC**

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**$T_1$ 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 diethylphthalate. 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 diethylphthalate dissolved in CDCl$_3$ on a Spinsolve 80 MHz.](image)

**$T_2$ 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 diethylphthalate (color coded).

![Figure 8: Proton $T_2$ relaxation curves measured for 250 mM diethylphthalate dissolved in CDCl$_3$ on a Spinsolve 80 MHz.](image)