Module „NMR“ (MSc): Practical exercises

Sheet 10

10.1

The $^1$H and $^{13}$C$^1$H NMR spectra of (E)-4-methyl-4'-nitrostilbene (C$_{13}$H$_{13}$NO$_2$) recorded in acetone-$d_6$ solution at 298 K and 500 MHz are given below.

The $^1$H NMR spectrum has signals at $\delta$ 2.34, 7.23, 7.33, 7.48, 7.56, 7.83 and 8.22 ppm.

The $^{13}$C$^1$H NMR spectrum has signals at $\delta$ 21.3, 124.8, 126.2, 127.9, 128.0, 130.3, 134.1, 134.8, 139.5, 145.3 and 147.5 ppm.

The $^1$H-$^1$H COSY, $^1$H-$^{13}$C me-HSQC and $^1$H-$^{13}$C HMBC spectra are given on the following pages. Use these spectra to assign the $^1$H and $^{13}$C$^1$H resonances for this compound.
$^1$H NMR Spectrum
(Acetone-$d_6$, 500 MHz)

$^{13}$C$^1$H NMR Spectrum
(Acetone-$d_6$, 125 MHz)

$^1$H–$^1$H COSY Spectrum
(Acetone-$d_6$, 500 MHz)
Identify the following compound.
Molecular Formula: C_{14}H_{12}O_{2}
IR: 1720 cm^{-1}

{^1}H NMR Spectrum
(CDCl₃, 500 MHz)

{^1}H NMR Expansion
(CDCl₃, 500 MHz)

{^{13}}C{^{1}}H NMR Spectrum
(CDCl₃, 125 MHz)

{^{13}}C{^{1}}H NMR Expansion
(CDCl₃, 125 MHz)
$^1$H-$^1$H COSY Spectrum
(CDC$\text{I}_3$, 500 MHz)

Expansion A

Expansion B

$^1$H-$^1$H COSY Spectrum
Expansion A
Read the following publication and explain the relevance of the following NMR data:


- The tremendous advances in the frontiers of macromolecular and supramolecular science that would not have been accessible without the new solid-state NMR methodologies are elaborated and discussed by the Hansen, Graf, and Spiess.
- Both NMR and EPR (Electron Paramagnetic Resonance) provide similar information about structure and dynamics of polymers and supramolecular systems. The fundamental difference being that EPR is concerned with the magnetically induced splitting of electronic spin states, while NMR describes the splitting of nuclear spin states.
- Combination of spectroscopy, scattering, and computer simulation becomes a powerful tool in studying both structure and dynamics of the supramolecular systems.

Q1: Please compare NMR and EPR spectroscopy, e.g., in terms of 1) diamagnetic and paramagnetic molecules, 2) chemical shifts and g-factor, and 3) J coupling constant and hyperfine coupling constant.

Q2: The authors claimed that $^1$H chemical shifts could serve as convenient indicators of $\pi-\pi$ stacking. Please comment on this with the example given in Figures 20 and 21.

Q3: For soft matters which parameter is used to quantify the amplitude of rotational motions in general? How this parameter is defined?

Q4: Synthetic polypeptides containing proline provide a unique way of studying the interplay between hydrogen bonding and geometric packing effects. Why?

Q5: Why the PLP Cδ and PBLG amide C=O resonances show distinctly different temperature dependences (as shown in Figure 9b)?