

Analysing complex mixtures using Pure shift DOSY

Product used : Nuclear Magnetic Resonance (NMR)

Diffusion-ordered spectroscopy (DOSY) is a powerful NMR method for the analysis of mixtures. In DOSY, signals in the NMR spectrum are resolved according to the measured diffusion coefficient for each signal, yielding a 2D spectrum which has chemical shift along the x-axis and diffusion coefficient along the y-axis.

Unfortunately, overlap between signals in the NMR spectrum can significantly limit the resolution in the diffusion dimension of DOSY spectra. This is a particular problem in ^1H spectra, where broader lines due to J -splittings mean that overlap is common. Fig. 1 shows a ^1H spectrum of *L*-menthol and *R*-(+)-limonene. The region of 1.3-2.2 ppm (red box), in particular, is heavily overlapped.

Recently, a new class of so-called "Pure shift" experiments has emerged that greatly reduces signal overlap by removing J -splittings in the spectrum to give single lines instead of broad multiplets for each peak. Fig. 2 shows the region of 1.3-2.2 ppm collected using a standard ^1H experiment (top) and a Pure shift (PSYCHE) experiment (bottom).

Pure shift and DOSY methods can be combined to enable the measurement of Pure shift DOSY spectra with much higher resolution in the diffusion dimension than conventional DOSY spectra. Fig. 3 shows the Pure shift DOSY spectrum of the same mixture. The 2D spectrum is shown on the left and the 1D slices of each component on the right. The signals of *L*-menthol and *R*-(+)-limonene have been completely resolved.

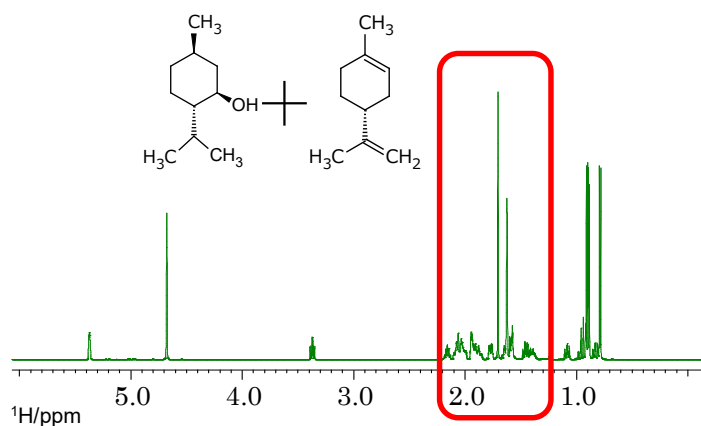


Fig. 1: ^1H spectrum of *L*-menthol and *R*-(+)-limonene

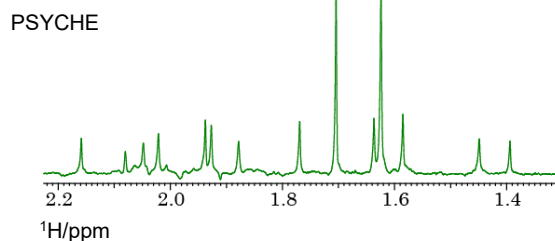
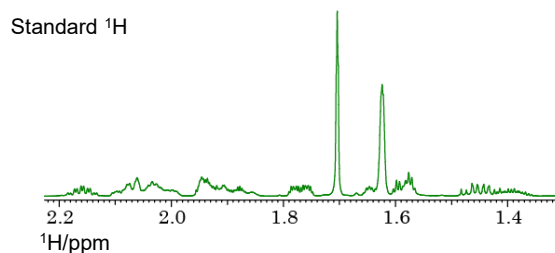
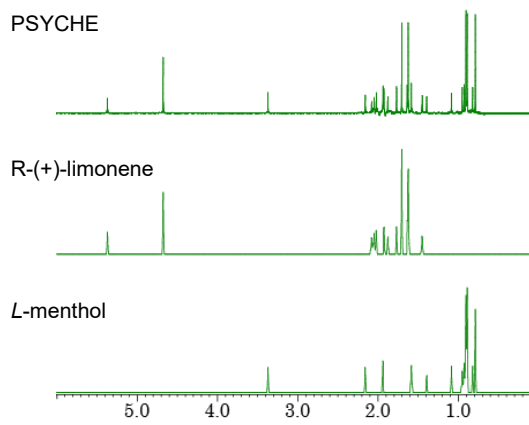
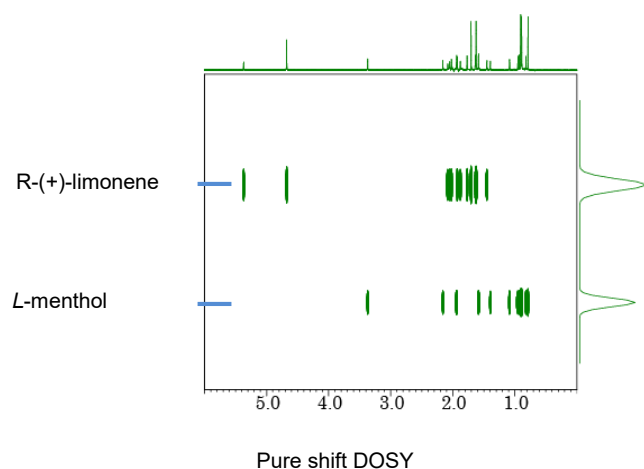


Fig. 2: Application of PSYCHE Pure shift method significantly reduces signal overlap



PSYCHE spectrum of the mixture and slices for each component

Fig. 3: Pure shift DOSY spectrum of *L*-menthol and *R*-(+)-limonene

Certain products in this brochure are controlled under the "Foreign Exchange and Foreign Trade Law" of Japan in compliance with international security export control. JEOL Ltd. must provide the Japanese Government with "End-user's Statement of Assurance" and "End-use Certificate" in order to obtain the export license needed for export from Japan. If the product to be exported is in this category, the end user will be asked to fill in these certificate forms.

Copyright © 2020 JEOL Ltd.

