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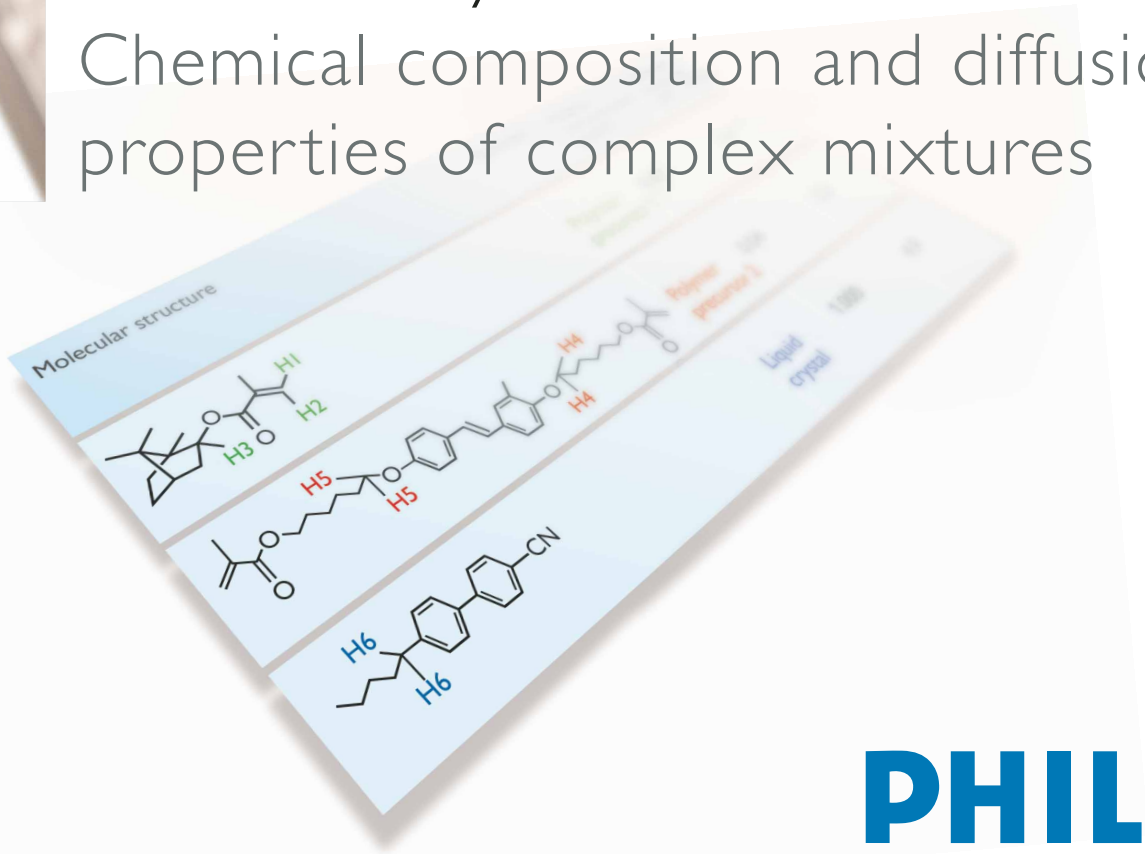
Molecular characterization and quantification of mixture components are essential analyses in chemical research and production environments. Similarly, absolute determination of diffusion constants of mixture components is often needed to solve problems of a more physical nature. Both can be assessed most adequately with NMR (Nuclear Magnetic Resonance) spectroscopy. This is exemplified for a mixture of liquid crystal (LC) material and UV-curable polymer precursors as used for the production of new-generation LC displays.

A NMR study

Chemical composition and diffusion properties of complex mixtures



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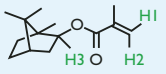
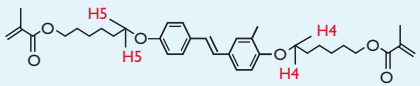
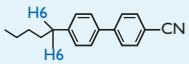
Molecular structure	Function	Relative concentration (mol/l)	Diffusion constant (10^{-11} m^2 s^{-1})
	Polymer precursor 1	0.87	5.4
	Polymer precursor 2	0.04	2.2
	Liquid crystal	1.000	4.9

Table 1: Chemical structures, relative concentrations, and diffusion constants of the LC/polymer mixture components as determined by NMR spectroscopy. Atoms labelled H1, H2, H3, H4, H5 and H6 correspond to the similarly labelled ^{1}H-NMR signals in figure 1.

Fig. 1: ^{1}H-NMR spectra of the LC/polymer mixture and the individual mixture components. ^{1}H-NMR signals labelled H1, H2, H3, H4, H5, and H6 correspond to the similarly labelled atoms in the chemical structures shown in table 1.

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New-generation LC displays

In the ongoing race to make larger, thinner, and cheaper LC displays, there is a clear trend towards a minimum number of production steps. Philips has developed a new LCD manufacturing technology that uses a single substrate with a stack of tailored organic layers. To create these so-called paintable displays, a substrate is coated with a mixture consisting of a LC material and polymer forming materials. This mixture is UV-cured in such a way that the switchable LC material ends up below a sheet of polymer. The process that causes this phase separation is called photo-enforced stratification. In order to optimise the production process of these displays, mathematical models have been generated for computer simulation of the phase separation process. In these models, concentrations and diffusion constants of all constituents in the LC/polymer mixture are critical input parameters and have to be determined experimentally.

NMR

Many atomic nuclei absorb electromagnetic waves with frequencies corresponding to the radio-frequency region of the electromagnetic spectrum (10^6 – 10^9 MHz). Absorption of radio waves by nuclei is only possible in the presence of a magnetic field. This phenomenon is called Nuclear Magnetic Resonance (NMR). NMR absorption spectra of nuclei, like visual-light absorption spectra of electrons, are highly indicative of the chemical composition of the sample in question. At present, NMR is the most commonly used analytical technique in organic chemistry.

Chemical composition of the LC/polymer mixture

The complete chemical composition can be determined from standard NMR spectra of the LC/polymer mixture, like the ^{1}H-NMR spectrum shown in fig.1. The established chemical structures of the three mixture constituents are listed in table 1.

Diffusion constants

Diffusion constants can be measured with a more complicated version of the NMR experiment. For this experiment a position-dependent magnetic field ("gradient") needs to be applied to the sample. The peak intensities in this type of NMR spectrum depend on the movement of the molecules in the gradient. For the determination of diffusion constants, a series of NMR spectra is acquired, in which the gradient strength is increased for each spectrum. Using data processing, the series of spectra can be transformed into a so-called diffusion-ordered two-dimensional NMR spectrum (fig. 2). The vertical axis directly represents the diffusion coefficient. The absolute diffusion constants of all mixture components can be determined after assignment of the NMR peaks. In this way quantitative information about concentrations and diffusivities of all mixture components is obtained.

Fig. 2: Contour plot of the diffusion-weighted two-dimensional NMR spectrum. The individual NMR spectra of the three mixture components (one-dimensional horizontal cross sections along the colored lines) can be clearly resolved.