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Chromatography in the NMR tube

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Recent developments in nuclecar magnetic resonance (NMR) equipment permit the spatially resolved observation of NMR signals on routine instruments that are part of virtually every chemistry laboratory. Specifically, a series of thin (~ 1 mm) horizontal slices may be excited and recorded of the sample instead of the ~20 mm bulk volume within the rf coil. Through referencing with solutions of known concentrations, local concentrations can be determined. We have recently applied slice-selective NMR spectroscopy to a range of chemical problems: A first example was the monitoring of the anisotropic swelling of cross-linked polymers in organic solvents and determination of the homogeneity of anisotropy across the polymer. Further applications were a "single-shot" NMR titration, where the signal of the first component was resolved over a concentration gradient of the "titrated" component, and a reaction monitoring, where two reagents diffuse towards each other in the sample tube. Here, we present another application: “chromatography in the NMR tube” (a glass tube with Ø = 5 mm): Two compounds that may differ in molecular size or polarity diffuse downwards through a polymeric matrix (which may be cross-linked polystyrene or even silicone grease) at their individual rates and appear in the slices at the bottom with individual concentrations. Although no complete separation of the compounds is achieved, signal assignment is facilitated, and diffusion coefficients may be calculated as an alternative to the DOSY method.

Biography

Michael John completed his PhD with Horst Kessler at the Technical University of Munich in 2004, and then spent 2 years with Gottfried Otting at the Australian National University, Canberra. Since 2007, he is Lecturer and Director of the NMR facility at the University of Göttingen. His list of publications includes more than 60 papers in peer-reviewed journals and 25 conference contributions.

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