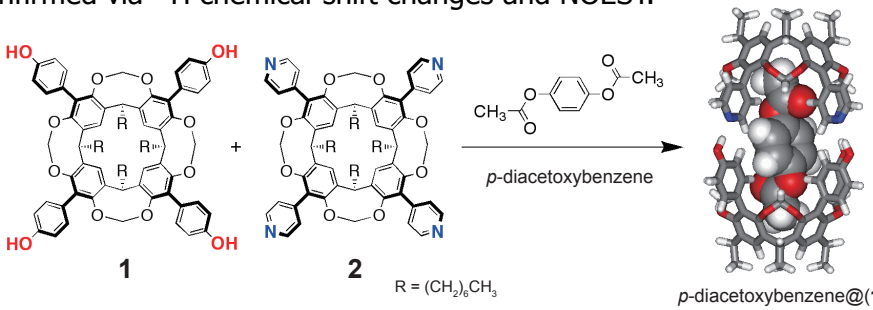
# Application of DOSY: Analysis of guest encapsulation

Guest encapsulation of host compounds like cyclodextrin has been studied through various analytical methods. Among them, NMR spectroscopy is mainly used to confirm complex formation via chemical-shift changes and to clarify steric structures using difference NOE and NOESY. By contrast, DOSY separates NMR spectra of mixture compounds with respect to their diffusion coefficients, and here its application to guest encapsulation is introduced.

* The present study is collaboration with Prof. K. Kobayashi of Shizuoka University.

Tetrakis(4-hydroxyphenyl)-cavitand (**1**) and tetra(4-pyridyl)-cavitand (**2**) self-assembly into a heterodimeric capsule (**1·2**) via hydrogen bonds in chloroform solution. This heterodimeric capsule possesses internal nanospace and encapsulates a guest molecule. For example, addition of 1 equiv of *p*-diacetoxybenzene to a capsule **1·2** forms 1:1 guest-encapsulating **1·2**, namely, *p*-diacetoxybenzene@(**1·2**). This guest encapsulation to the internal nano-space was confirmed via $^1$H chemical-shift changes and NOESY.




**< Analysis of DOSY spectrum >**

Figure 1 shows a DOSY spectrum of 1:1 mixture solution of *p*-diacetoxybenzene@(**1·2**) and *p*-diacetoxybenzene, manifesting two components having substantially different diffusion coefficients; free *p*-diacetoxybenzene shows a large coefficient (upper in the spectrum) while *p*-diacetoxybenzene@(**1·2**) a small coefficient (lower in the spectrum). The almost identical coefficients of encapsulated *p*-diacetoxybenzene and heterodimeric capsule **1·2** firmly prove their encapsulation. Also, two distinct components of *p*-diacetoxybenzene inside and outside of the capsule suggest that their exchange should be slow on the NMR time scale and that *p*-diacetoxybenzene@(**1·2**) be stable.

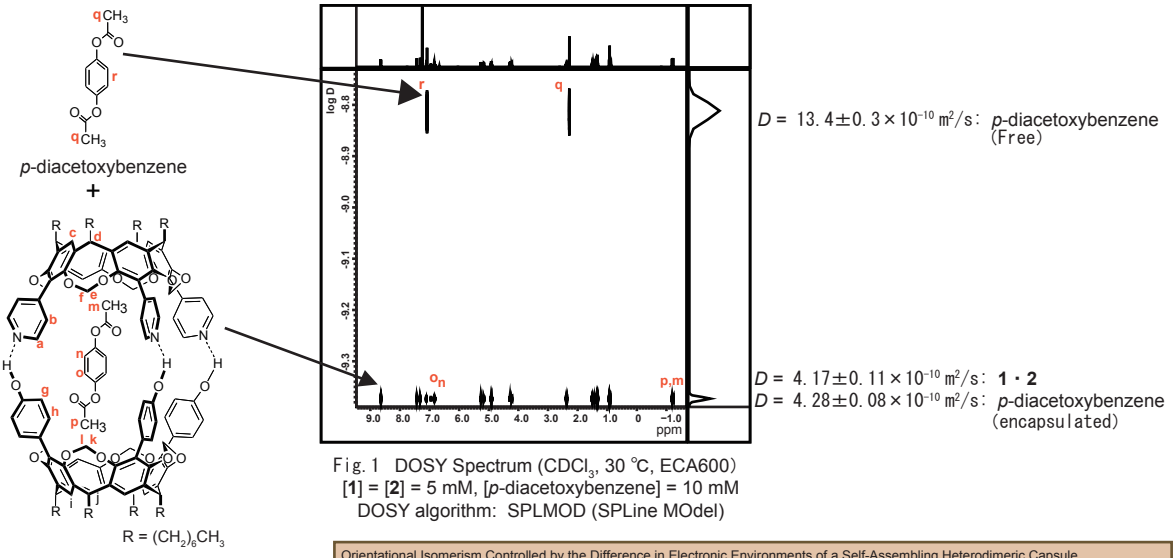



Fig. 1 DOSY Spectrum ($CDCl_3$, 30 ℃, ECA600)
[**1**] = [**2**] = 5 mM, [*p*-diacetoxybenzene] = 10 mM
DOSY algorithm: SPLMOD (SPLine MOdel)

Orientational Isomerism Controlled by the Difference in Electronic Environments of a Self-Assembling Heterodimeric Capsule
Kenji Kobayashi, Ryosuke Kitagawa, Yoshifumi Yamada, Masamichi Yamanaka, Takako Suematsu, Yoshihisa Sei, and Kentaro Yamaguchi
*J.Org.Chem.* **2007**, *72*, 3242-3246.

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