

## Full Paper

### CPMAS Study of Ring Inversion of *cis*-decalin in the Solid State when Complexed with $\beta$ -cyclodextrin

CPMAS Study of Ring Inversion of *cis*-decalin

Helena Dodziuk<sup>A,B</sup> and Mariusz Pietrzak<sup>A</sup>

<sup>A</sup>Institute of Physical Chemistry, Polish Academy of Sciences, Kasprzaka 44/52, 01-224 Warsaw, Poland.

<sup>B</sup>Corresponding author. Email: dodziuk@ichf.edu.pl

## Experimental Section

$\beta$ -CD **1** was kindly provided by Wacker; *cis*-decalin **2** was purchased from Merck-Schuchardt. The **2@1** complex was crystallized from DMF using an excess of **2**. Colorless crystals precipitated from the solution stored at 313 K for several days were ground and placed in the 4mm rotor.

NMR spectra were recorded on a 500 MHz Bruker Avance II spectrometer operating at 125.77 MHz for <sup>13</sup>C. Standard <sup>13</sup>C CPMAS NMR method was used at the 10 and 3.7 kHz rotation speeds. Quantitative <sup>13</sup>C MAS spectrum was recorded using the one pulse experiment with inverse gated proton decoupling and delay time of 60 s. The spectra of liquid samples were measured in the rotor at the 3.7 kHz rotation speed.

Temperature control was achieved by using the <sup>207</sup>Pb chemical shift of Pb(NO<sub>3</sub>)<sub>2</sub> as a thermometer.<sup>[18]</sup> <sup>207</sup>Pb NMR spectra of the solid Pb(NO<sub>3</sub>)<sub>2</sub> were always recorded prior or directly after a measurement of the **2@1** complex using exactly the same conditions (set temperature, VT gas flow, rotation speed) in order to achieve stable temperature conditions, the measurements have been carried out after at least 30 minutes waiting before each experiment. At least 1 hour was allowed when changing the set temperature. Nevertheless, the distribution of temperatures inside the sample in the rotor was sometimes as high as 40°C at highest temperatures. Therefore, the temperatures shown in the figures are averaged values.

[18] a) G. Neue, C. Dybowski, *Solid State Nucl. Magn. Reson.* **1997**, 7, 333-336; b) T. Takahashi, H. Kawashima, H. Sugisawa, T. Baba, *Solid State Nucl. Magn. Reson.* **1999**, 15, 119-123; c) P. A. Beckmann, C. Dybowski, *J. Magn. Reson.* **2000**, 146, 379-380.

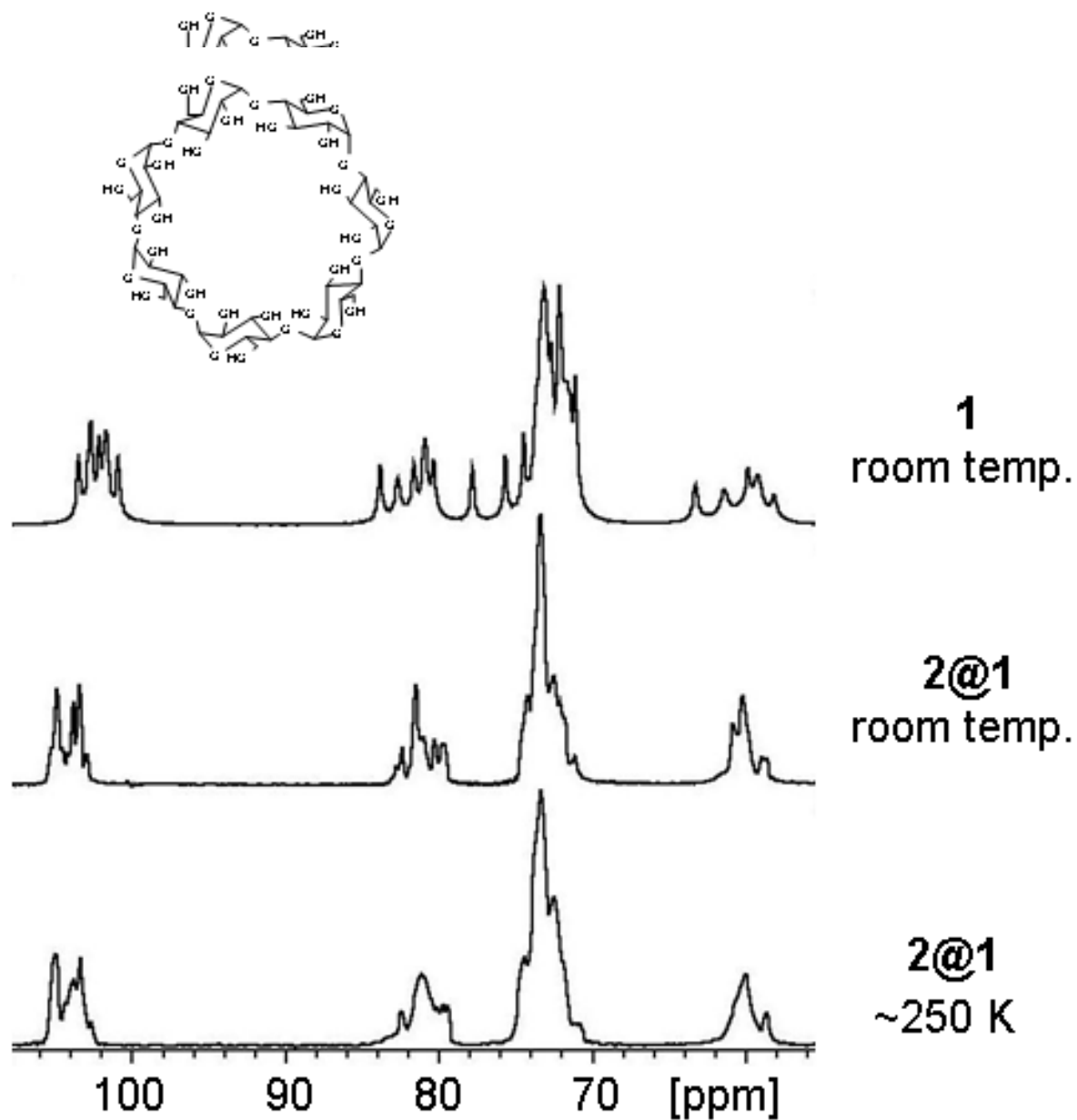


Figure S1.  $^{13}\text{C}$  CPMAS NMR spectra of the free **1** and the **2@1** complex ( $\beta$ -CD part) at room and low temperature.

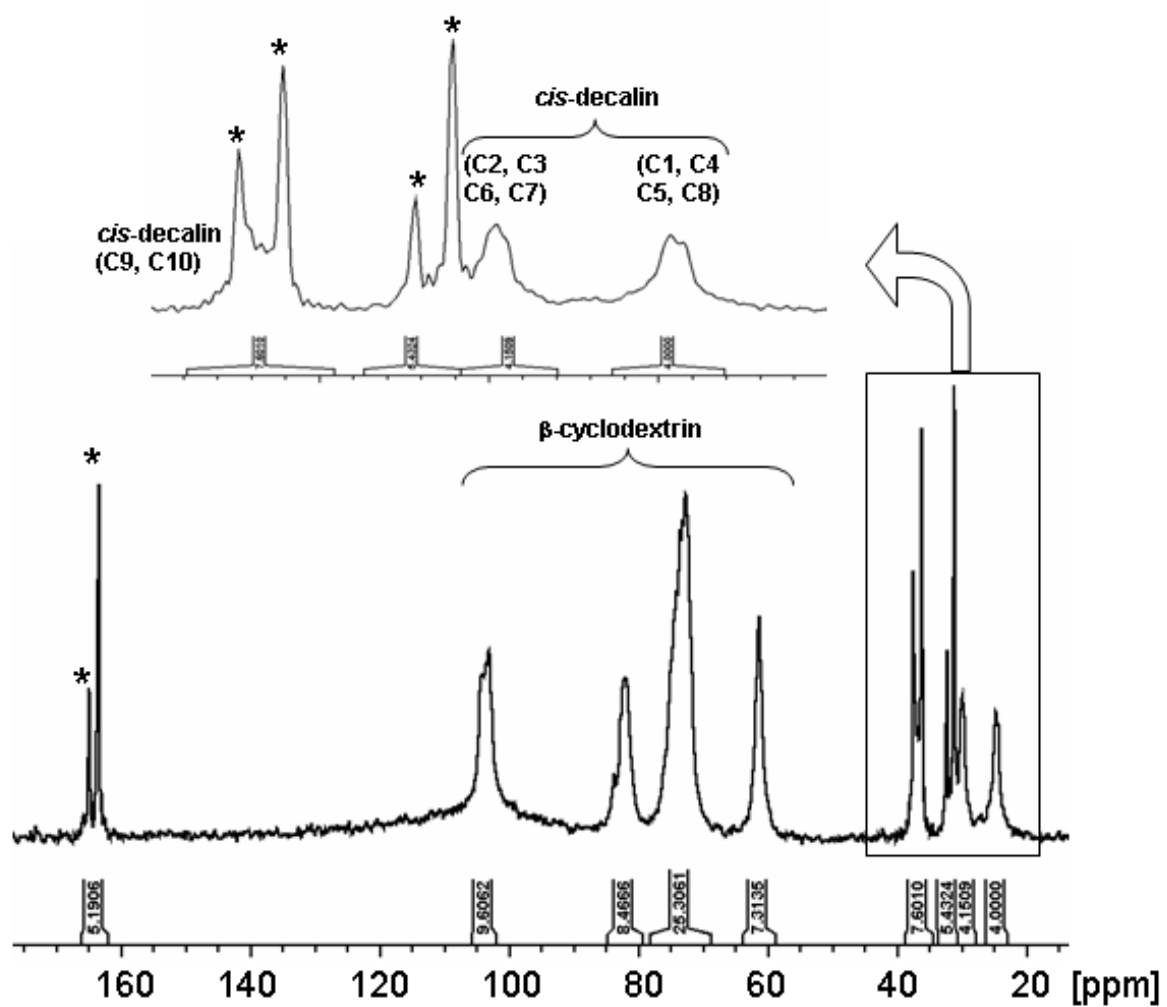


Figure S2. Quantitative  $^{13}\text{C}$  MAS NMR spectrum of the 2@1 complex. Signals labelled with asterisks belong to DMF.

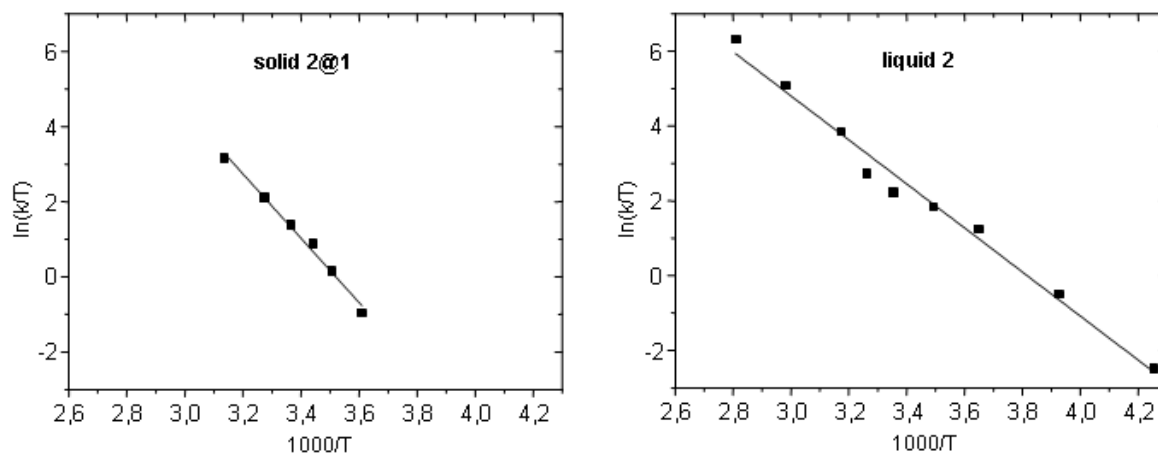


Figure S3. Eyring plots for the solid **2@1** complex and for liquid **2**.