

# CRYSTALLOGRAPHIC INSIGHT INTO DESOLVATION PROCESS OF CUCURBITURIL CRYSTALS

*Oksana Danylyuk<sup>a</sup>, Volodymyr Sashuk<sup>a</sup> and Vladimir Fedin<sup>b</sup>*

<sup>a</sup>*Institute of Physical Chemistry, Polish Academy of Sciences, Kasprzaka 44/52, 01-224 Warszawa, Poland; odanylyuk@ichf.edu.pl.*

<sup>b</sup>*Nikolaev Institute of Inorganic Chemistry, Siberian Branch of the Russian Academy of Sciences, 3 Acad. Lavrentiev. Ave., 600090 Novosibirsk, Russian Federation.*

The retention of crystallinity upon desolvation of molecular crystals and even metal-organic frameworks is not common, as the molecules are rigidly and densely packed in the crystals and the original framework usually collapses once solvent is removed from the structure. However, in rare cases the host framework remains substantially unaffected by solvent (guest) removal yielding structure with open channels or discrete lattice voids that can show permanent porosity<sup>1</sup>. Furthermore, sometimes happens, the desolvation process proceeds as single-crystal to single-crystal transformation resulting in distortion and sliding of the structure, changes in conformation, coordination modes and/or space group. Such behavior is classified as flexible and dynamic motions<sup>2</sup>.

Here we would like to present the crystallographic study on the desolvation process of two crystalline materials: magnesium ion complexed cucurbit[6]uril and cucurbit[5]uril hydrochloride hydrate. The crystal structure of the magnesium ion complexed cucurbit[6]uril reveals the presence of large water-filled channels that collapse due to water loss at ambient conditions. The structure determination of the desolvated system shows the unit cell volume difference between the initial crystal and that exposed to ambient conditions of 13.8%. Interestingly, the drastic shrinkage of the channels is accompanied by the change in the coordination environment of the magnesium ion. The cucurbit[5]uril hydrochloride hydrate (space group  $P2_1/c$ ) transforms into thermodynamically stable form (space group  $Pnma$ ) with the loss of 8 water molecules per cucurbit[5]uril at ambient conditions *via* a single-crystal to single-crystal mechanism.

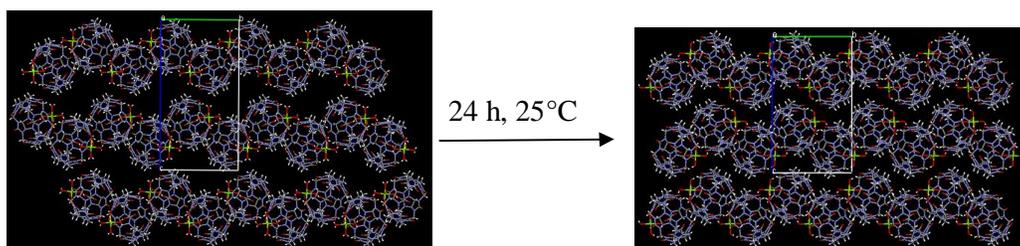


Fig. 1 Crystal packing of the magnesium complexed cucurbit[6]uril structure before and after desolvation (water molecules omitted for clarity).

1. Barbour L. J. *Chem. Commun.* 1163 (2006).
2. Kitagawa S., Uemura K. *Chem. Soc. Rev.* 109 (2005).