Macrocycle Refinement: Bambusuril Structures

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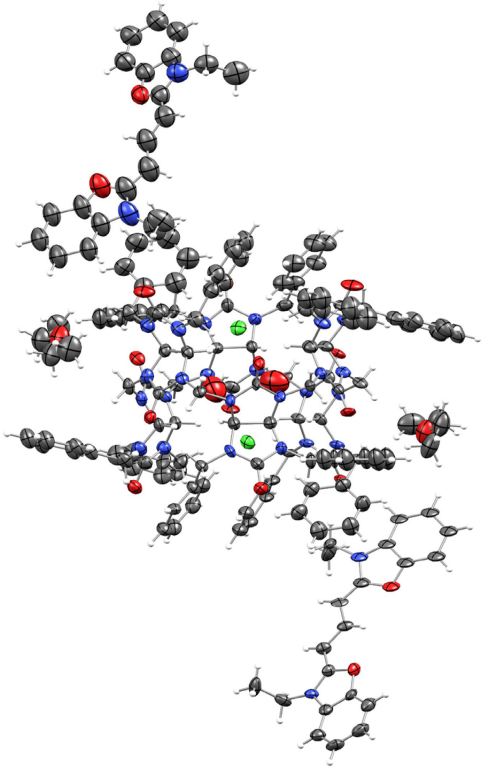
Supramolecular complexes formed by non-covalent interactions between two chemical building blocks, in particular non-rigid macrocycle hosts binding through weak interaction ions or other guests, are notoriously difficult to investigate by X-ray diffraction. The main reasons for this are the conformational flexibility of the macrocycle and inefficient packing, resulting in the inclusion of disordered solvent molecules. Massive or whole molecule disorder of the host-guest pair and solvent loss typically lead to non-ideal diffraction patterns. Yet, careful selection of crystallization conditions and mounting techniques can, to some extent, mitigate the problems.

Herein, we present examples of aforementioned conditions for macrocycle structures containing bambusuril. Bambusurils are made from derivatives of the bicyclic molecule glycoluril linked via methylene bridges, with repeat units ranging from 4 to 24. Owing to the binding affinity of the inner cavity, bambusurils can form host-guest complexes through non-covalent interactions. The structures discussed in this project include guest anions in the bambusuril cavity and a cationic organic dye molecule.

We optimized crystallization and developed techniques for crystal selection and mounting to better prevent solvent loss and thus preserve crystallinity. In addition, the structure analysis was performed using common techniques for the refinement of weakly diffracting structures with extensive disorder of large molecules or in voids. The strategies for the crystal selections and refinement on highly disordered structures will be discussed in detail.



Bambusuril macrocycle with anions, dyes, and solvent molecules. The SQUEEZE routine of PLATON[1] was applied to correct the data for unidentified solvent molecules.

1. A. L. Spek, *Acta Crystallogr. Sect. C* **2015**, 71, 9– 18.

Figure 1