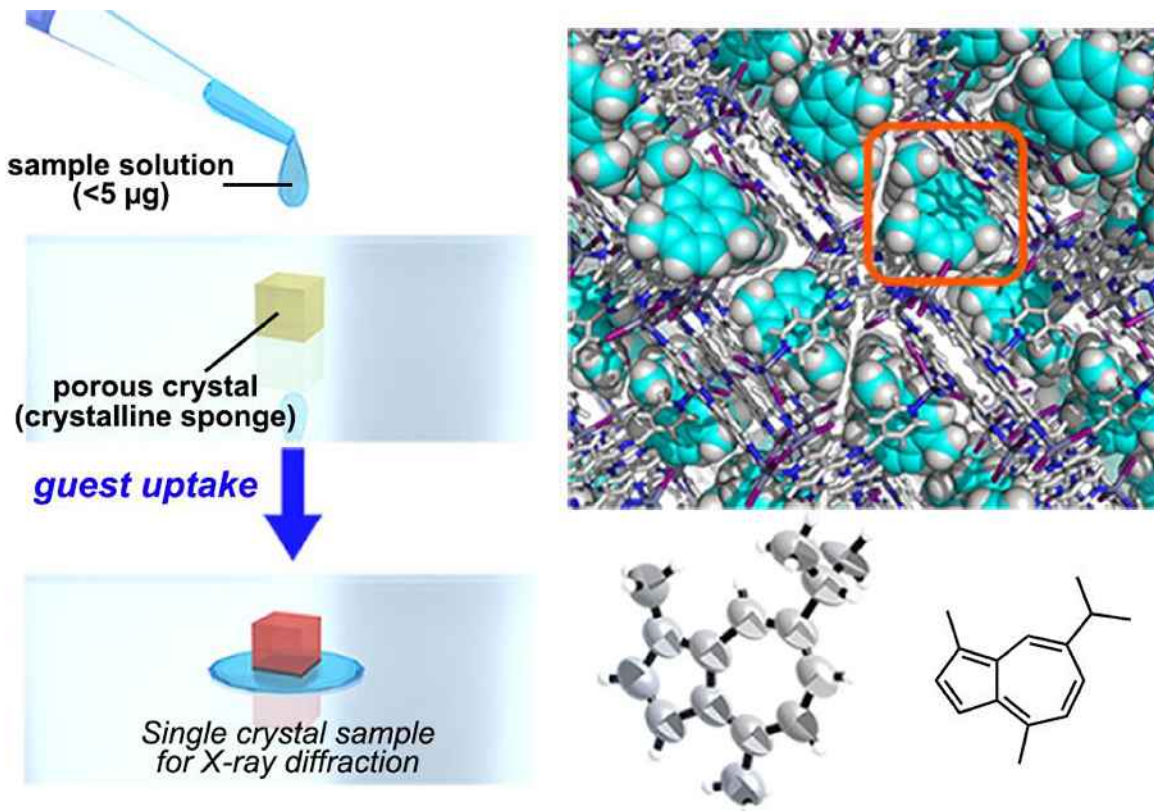


Single Crystal X-ray Analysis of Trace Compounds Using Porous Network Crystals

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Crystallization of compounds has been one of the major drawbacks of single crystal X-ray analysis. The situation becomes more difficult when we deal with volatile, oily, and trace amount of samples. We have developed a new method to prepare a single crystal sample for X-ray measurement from non-crystalline compounds on nanogram-to-microgram scale using porous coordination network crystals. Here porous crystals are used as crystalline sponges that can accommodate and three-dimensionally align the compounds in a single-crystal to single-crystal fashion. In this method, a crystalline sponge is soaked in a solution containing ~5 μg of target samples. Upon slow evaporation of solvent, target compounds are accommodated and concentrated in crystal so that they are oriented along the pores with high occupancies. Thus, the structures of target compounds are analysed along with the coordination framework without crystallization of the samples. Moreover, since single crystal samples can be prepared using one tiny crystal, molecular structure of 50 ng of non-crystalline compound has been successfully determined by this method. In this presentation, we will show a variety of structures determined by using this new method, so-called crystalline sponge method.

[1] Y. Inokuma, S. Yoshioka, J. Ariyoshi et al., *Nature*, 2013, 495, 461-466, [2] Corrigendum: Y. Inokuma, S. Yoshioka, J. Ariyoshi et al., *Nature*, 2013, 501, 262, [3] Y. Inokuma, S. Yoshioka, J. Ariyoshi et al., *Nature Protocols*, 2014, 9, 246-252.



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