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The Crystalline Sponge Method: Synthetic and Crystallographic Guidelines

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Synthetic and crystallographic guidelines for the crystalline sponge method based upon the analysis of rapidly-synthesized crystal sponges using high-flux synchrotron radiation are presented [1]. The synthetic procedure for the zinc-based metal-organic framework (MOF) used in preliminary crystal sponge reports [2, 3] has been modified to afford suitable sponges in 3 days instead of 2 weeks. These sponges were tested on some small molecules, with two being difficult cases for in-house diffraction analysis in regards to data quality and determination of space group. These issues were readily resolved by the use of high-flux synchrotron radiation with <1 hour data collection times. None of the structures required the use of electron density removal programs to treat solvent accessible voids. The crystalline sponge systems analyzed in this study are the highest-quality structures of this class presented to date that meets chemical crystallographic standards. As a result of these studies, a set of guidelines for the crystallographic process were developed, and the single crystal to single crystal transformation process for these systems was also analyzed. The presented guidelines will be helpful for those interested in using the crystalline sponge method to fully elucidate the structures of non-crystallizable compounds at in-house diffraction or synchrotron facilities, and will aid in obtaining data that meet the standards of chemical crystallography.

[1] *T. R. Ramadhar, S.-L. Zheng, Y.-S. Chen, et al., Submitted,* ***[2]*** *Y. Inokuma, S. Yoshioka, J. Ariyoshi, et al., Nature, 2013, 495, 461–466,* ***[3]*** *Y. Inokuma, S. Yoshioka, J. Ariyoshi, et al., Nature Protocols, 2014, 9, 246–252*

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