

**MS30-1-1 Benefits of Cu K $\beta$  radiation for the structure determination of sponge crystals**

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**Abstract**

The Crystalline Sponge method enables the structural elucidation of scarce, small organic analytes and hard-to-crystallize compounds. By diffusion of solvent and analyte molecules into the cavities of a metal-organic framework (the crystalline 'sponge') and molecular orientation by the MOF's functional groups, an analyte becomes part of the crystal structure and thus observable through X-ray diffraction methods.[1]

Unfortunately, many problems inherent to the structural elucidation of MOFs limit the possibilities of this method. These include weak host-guest interaction, incomplete diffusion, twinning, disorder of both analyte and solvent and sample decay. As much as the physical and chemical aspects of the method have recently been improved, the crystallographic aspects pose challenging problems onto the method. As we have recently adopted the use of the very rarely used Cu K $\beta$  radiation in our X-ray department and found promising results in the analysis of our small molecule data, we also applied Cu K $\beta$  radiation to the Crystal sponge method and compared it to the more commonly used wavelength Cu K $\alpha$  in a total of six comparison experiments. Cu K $\beta$  inherits a shorter wavelength than Cu K $\alpha$  radiation (1.39222 Å compared to 1.54187 Å) and thus results in up to 38 % more unique reflections and up to 136 % total reflections. This is mainly due to the maximum resolution of 0.72 Å is accessible for Cu K $\beta$  radiation, compared to 0.80 Å for Cu K $\alpha$  radiation. Less absorption by consistently 25 % in absorption coefficient for Cu K $\beta$  leads to less background and absorption damage.[2] This results in better crystallographic models for the Cu K $\beta$  data sets: Significantly lower quality parameters were obtained in every Cu K $\beta$  experiment. Bond precision was mainly better and the compared residual electron density maps (Fig. 1) look much cleaner. Additionally, more solvent positions could be modelled in the Cu K $\beta$  data sets. In total, way fewer restraints and no constraints had to be used in the models collected using Cu K $\beta$  radiation. Our findings suggest a routine use of Cu K $\beta$  radiation – not only for the crystalline sponge method. The same advantages are to be expected in general for the structural elucidations of metal-organic frameworks but also standard small molecule crystallography.

**References**

[1] Hoshino, M., Khutia, A., Xing, H., Inokuma, Y. & Fujita, M. (2016). *IUCrJ*. 3, 139–151

[2] Meurer, F., von Essen, C., Kuehn, C., Puschmann, H., Bodensteiner, M. (2022). *IUCrJ* 9, DOI: <https://doi.org/10.1107/S2052252522002147>

Cu K $\alpha$  (left) and Cu K $\beta$  (right) of the same sponge

