Identification of light elements by long-wavelength crystallography

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The assignment of the element type to metal- or anion-binding sites in macromolecules when interpreting electron density maps is typically based on a combination of chemical and biological considerations. Bond lengths and angles, the coordination sphere, and crystallographic parameters like the B-factor from the structure refinement can provide indirect evidence, particularly at high resolution. However, at a lower resolution or in the case of mixed or partial occupancies this can lead to misinterpretation and wrong assignments. Anomalous diffraction can overcome this issue and provide direct evidence. The presence of a peak in the anomalous difference Fourier map above an absorption edge which disappears (or is significantly reduced in the case of L or M edges) when tuning the X-ray wavelength to below the edge provides the locations of a particular element [1]. The long-wavelength macromolecular crystallography beamline I23 at Diamond Light Source covers a significant larger wavelength range than any other beamline up $\lambda = 5.9$ Å opening this application to lighter elements of biological significance with access to the K absorption edges for elements like calcium, potassium, chlorine, sulfur, and phosphorus [2].

In my talk, I will present how we could overcome the challenges of long-wavelength crystallography to open these new opportunities to the user community and present several highlights [3,4,5]. While the beamline cannot access the absorption edges of sodium and magnesium, I will discuss how crystallography can help identify these elements.

[1] K. Handing et al. *Characterizing metal-binding sites in proteins with X-ray crystallography* Nature Protocols 13 (2018) 1062.
[2] A. Wagner et al. *In-vacuum long-wavelength macromolecular crystallography* Acta Crystallographica Section D Structural Biology 72 (2016) 430.
[3] A. Rozov et al. *Importance of potassium ions for ribosome structure and function revealed by long-wavelength X-ray diffraction* Nature Communications 10 (2019) 2519.
[4] P.S. Langan et al. *Anomalous X-ray diffraction studies of ion transport in K+ channels* Nature Communications 9 (2018) 4540.
[5] M. Herdman et al. *High-resolution mapping of metal ions reveals principles of surface layer assembly in Caulobacter crescentus cells* Structure 30 (2022) 215.