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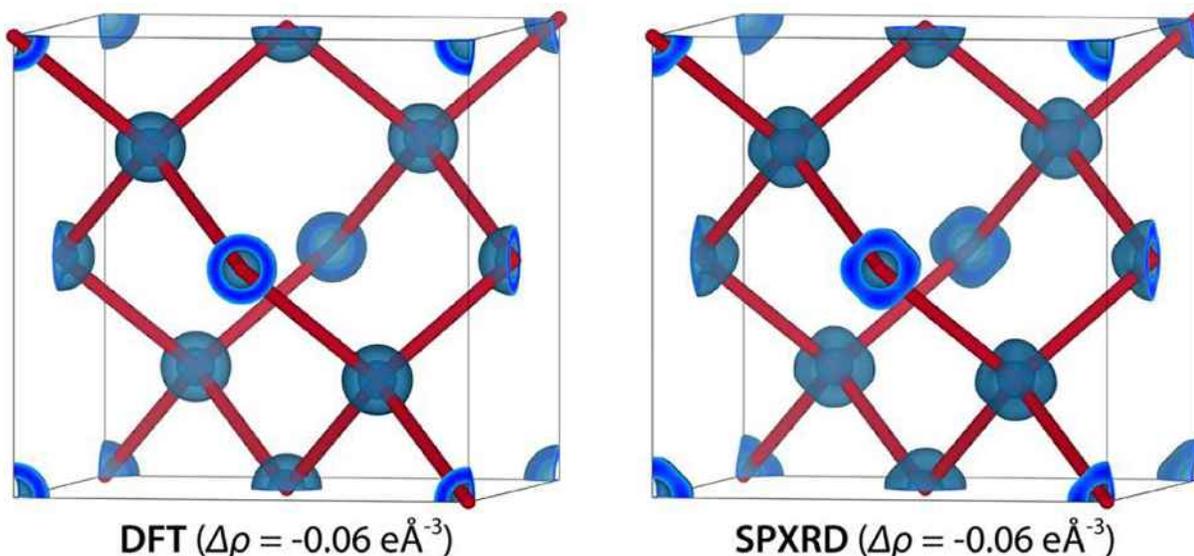
Accurate Charge Densities from Synchrotron Powder Diffraction

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Advancements within the field of synchrotron powder X-ray diffraction (SPXRD) have rendered it a viable approach for probing subtle electronic features. It is especially powerful for highly crystalline inorganic extended materials, resolving severe extinction issues conventionally encountered with single-crystal diffraction. Furthermore, SPXRD data exhibit markedly reduced absorption and may be collected in a single exposure. The latter prevents systematic errors from merging a multitude of detector frames, each possessing a slightly different scale factor. These experimental advantages are counterbalanced by a more complicated data analysis, where the key problems are the inherent challenges of overlapping reflections and background subtraction. To evaluate the performance of SPXRD and to test the methodologies for estimating charge densities (CDs), we use benchmark data on diamond collected to low d-spacing. [1] The critical step is the recovery of observed structure factors from the powder pattern. This is the focal point of the present study, scrutinizing several traditional and novel approaches that deviate strongly in terms of model complexity and structure-dependency. All recovered sets of structure factors are evaluated with respect to their capability to determine the true atomic displacement parameter and to estimate the CD by both multipolar modelling and maximum entropy reconstruction. The data are of such exceptional quality that they even reveal how the innermost electron density responds to the formation of covalent bonding (figure below). Supporting their huge success over the last decades, only the Rietveld-based approaches are capable of quantifying this fine feature. The study of core polarization has emerged as a new frontier in chemical bonding studies, and the relevant experimental information may be reliably accessed by SPRXD for highly crystalline materials.

[1] N. Bindzus et al. (2014) *Acta Cryst. A* 70, 39-48



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