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Lower resolution Laue neutron data yield correct nanocluster formulations

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The renaissance in Laue studies - at neutron sources - provides us with access to single crystal neutron diffraction data for synthetic compounds without requiring synthesis of prohibitively large amounts of compound or improbably large crystals. Such neutron diffraction studies provide vital data where proof of the presence or absence of hydrogen in particular locations is required and which cannot validly be proved by X-ray studies. Since the commissioning of KOALA at OPAL in 2009[1] we have obtained numerous data sets which demonstrate the vital importance of measuring data even where the extent of the diffraction pattern is at relatively low resolution - especially when compared to that obtainable for the same compound with X-rays. In the Laue experiment performed with a fixed radius detector, data reduction is only feasible for crystals in the "goldilocks" zone – where the unit cell is relatively large for the detector, a correspondingly low resolution diffraction pattern in which adjacent spots are less affected by overlap will yield more data against which a structure can be refined than a pattern of higher resolution – one where neighbouring spots overlap rendering both unusable (in our current methodology). Analogous application of powder neutron diffraction in such determinations is also considered. Single crystal neutron diffraction studies of several important compounds (up to 5KDa see figure below)[2] in which precise determination of hydride content by neutron diffraction was pivotal to the final formulation will be presented. The neutron data sets typically possess 20% or fewer unique data at substantially “lower resolution” than the corresponding X-ray data sets. Careful refinement clearly reveals chemical detail which is typically unexplored in related X-ray diffraction studies reporting high profile chemistry despite the synthetic route being one which hydride ought to be considered/excluded in product formulation.

[1] *A.J. Edwards, Aust. J. Chem. 2011, 64, 869-872,* ***[2]*** *A.J. Edwards, manuscript in preparation*

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