MS46 Reproducibility in crystallography

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The 'colouring' problem in the delafossite $PdRhO_2$ vanished?
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Abstract
Outstanding properties of metallic delafossites , e.g. $PtCoO_2$, $PdCoO_2$, $PdRhO_2$, are largely attributed to exceptional quality of single crystals which are typically obtained without special efforts [1]. X-ray diffraction experiments on single crystals of very high quality containing heavy elements are typically suffering from severe extinction effects. The pronounced anisotropic structure is well reflected in a plate-like shape of the crystals. The associated severe absorption effects need to be taken into account with great care. Fortunately, we have been able to optimize crystal shape via micro-structuring using focused ion beam (FIB) techniques. Without any particularly sophisticated additional data treatment the structure refinement for $PdRhO_2$ revealed truly surprising results [2]. The anticipated "colouring" problem seems not to exist, as among the four obvious possibilities to occupy the two metal sites the 'correct' one clearly gives the best fit to the experimental data. In order to check if this unexpected result is just accidental, e.g. a consequence of remaining systematic errors in the data, several statistical and analytical tests were applied in order to unravel associated bias in the final structural model. Ultimate goal are clear indicators which are helping to improve the significance of experimentally derived parameters and to establish an approach to identify and experimentally minimize contributions from systematic errors in diffraction data.

References
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[2] P. Kushwaha et al., *Cryst. Growth Des.* **17** (2017), 4144. DOI: 10.1021/acs.cgd.7b00418