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Precision and accuracy of single crystal X-ray results

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Although everything seems to be already well known in the field of routine structural single crystal X-ray analysis and more than 1.1 mln organic, inorganic and macromolecular structures have been solved and refined so far, even commonly used approaches in X-ray diffraction and models of electron density applied should be critically re-evaluated from time to time. It is incredible that the Independent Atom Model (IAM) of electron density, effectively introduced a century ago, is still the most common model of electron density used in structural analysis. One would even say that its success has dominated the whole field of X-ray diffraction for the past century and for years now plays, in my opinion, quite a negative role. When IAM was introduced, Max von Laue, the Braggs, and their colleagues, were using home-made pieces of equipment which could have hardly supplied qualitative information on diffraction spots. In consequence, the errors associated with the model of electron density used were overshadowed by far larger diffraction hardware errors. However, within the past century, there has been an overwhelming progress in design and production of X-ray hardware which is made for needs of both small laboratories and large scale facilities. This progress in sophisticated X-ray hardware should also accelerate progress in the quality and complexity of models of electron density used to interpret experimental results. The use of the 100 years old IAM effectively proves that even with the most modern scientific tools, one can step backward and do ca. 100 years old crystallography.

In my presentation, I will discuss precision and accuracy of single crystal X-ray results obtained for multiple measurements of single crystals of oxalic acid as a function of resolution of X-ray data and quality of electron density model applied (IAM, multipole model (MM), Hirshfeld Atom Refinement (HAR) and Transferable Aspherical Atom Model of electron density (TAAM)). I will present a detailed comparison of structural, thermal and electronic parameters obtained for the same multiple diffraction data sets collected for single crystals of oxalic acid when different models of electron density are refined against collected intensities of reflections complemented by comparison to single crystal neutron diffraction and theoretical results[1]. Some practical suggestions will be presented how to estimate and improve the quality of single crystal X-ray diffraction structural results. Among others with the newer models, one can obtain more precise and accurate information on positions of H-atoms [2] or energy of intermolecular interactions in crystals.

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