Poster

Creation and Analysis of a Single Crystal X-ray Diffraction Experiment Benchmark Database

D. Brüx[1], F. Kleemiss[1]

[1]*Institute of Inorganic Chemistry, RWTH Aachen University, Landoltweg 1a, 52074 Aachen, Germany*

daniel.bruex@rwth-aachen.de

Classical methods for the determination of crystal structures from single crystals rely on the independent atom model (IAM), disregarding the error it comes along with. Modern state-of-the-art instrumental techniques and precision allow us to determine the molecular electron density in crystals with high accuracy, making the IAM shortcomings undisregardable [1]. By relying on more accurate electron density models, methods of aspherical refinement improve the modeling of crystal structures. The Hirshfeld atom refinement (HAR) has proven to be a powerful tool in this regard [2]. When applied, tailor-made scattering factors are calculated on the fly for each atom in the crystal structure using quantum chemical *ab-initio* methods. This underlying step of calculating the electron density is regulated by a set of parameters for the calculation. At this point, we refer to the HAR implementation of *Olex2*: *NoSpherA2*, which already offers a variety of parameters for the refinement [3]. The choice of those can have a significant impact on the refined crystal structure, but distinct and extensive studies in this regard lack so far.

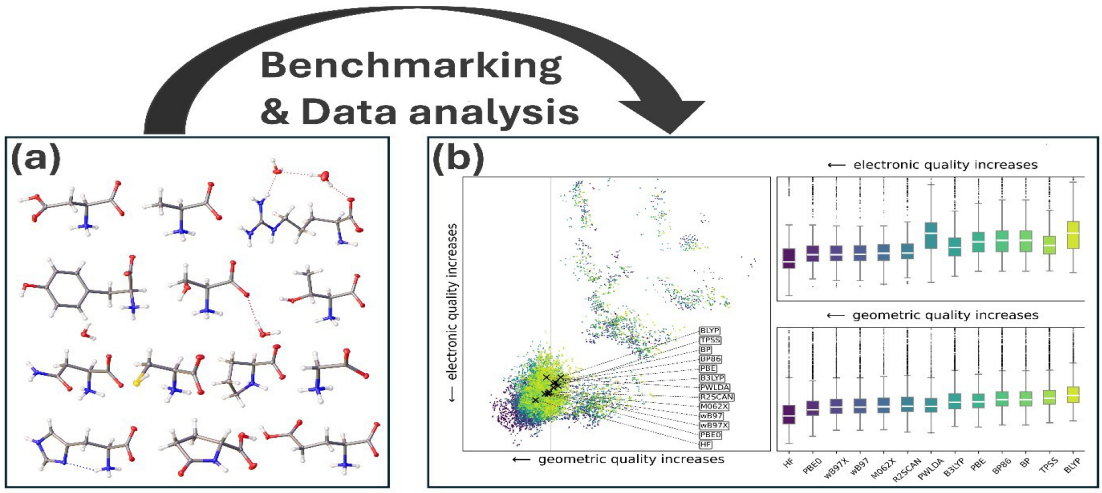

Figure 1. Workflow of the Benchmark: Creating a set of high-resolution crystal structures (a); Evaluating the whole dimensional space of all parameter combinations (b).

In this work, we present a benchmark database of single crystal X-ray diffraction data of small molecules. For this, datasets of 14 amino acid were used to systematically evaluate the influence of the different input parameters on the results obtained by HAR. The latter includes the basis set, DFT method, non-local dispersion correction, solvent model, convergence criteria, and integration accuracy. Each dataset was refined with all possible permutations of the input parameters. The results were analyzed according to electronic and structural quality indicators. The quality of the refinement outcome for small polar molecules demonstrates a strong dependency on the choice of the input parameters, showcasing the importance of a systematic evaluation of the refinement parameters. Revealing, that especially unexpected results can arise when evaluating the complete parameter space, which also gives the possibility to draw conclusions about the method itself.

[1] Sanjuan-Szklarz, W. F., Woińska, M., Domagała, S., Dominiak, P. M., Grabowsky, S., Jayatilaka, D., Gutmann, M. & Woźniak, K. (2020) *IUCrJ*, **7**, 920-933.

[2] Capelli, S. C., Burgi, H.-B., Dittrich, B., Grabowsky, S. & Jayatilaka, D. (2014) *IUCrJ*, **1**, 361-379.

[3] Kleemiss, F., Dolomanov, O. V., Bodensteiner, M., Peyerimhoff, N., Midgley, L., Bourhis, L. J., Geoni, A., Malaspina, L. A., Jayatilaka, D., Spencer, J. L., White, F., Grundkötter-Stock, B., Steinhauer, S., Lentz, D., Puschmann, H., Grabowsky, S. (2021) *Chem. Sci.* **12**, 1675