Oral presentation

Implementing X-ray crystallography into courses at a student laboratory

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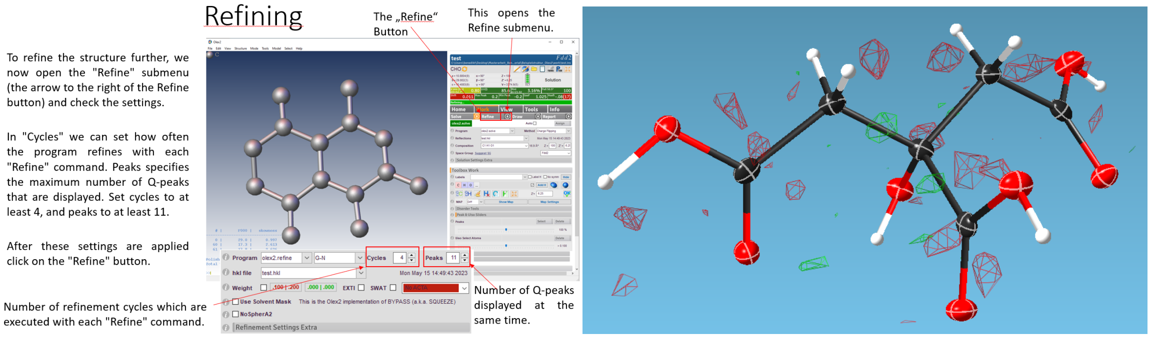
Single crystal X-ray diffraction is not only a powerful tool for structure determination but also an invaluable asset for education. Unlike many analytical methods, it inherently allows the visualization of chemical structures and give us a means by which "*we can now "see" the individual atoms and molecules*" as once stated by Sir W. H. Bragg[1] This unique capability makes X-ray diffraction an enticing addition to natural science teaching. There are practical limitations, as data collection requires specialised (and expensive) equipment as well as trained experts, both of which may not be available to schools. However, with already measured data available, solving and refining of crystal structures can be performed with and by students[2] with the help of graphical user interfaces.[3]

A module was created consisting of a didactically reduced introduction to the theoretical background of single crystal X-ray diffraction followed by course material consisting of an .ins and .hkl file from a sample dataset as well as a step-by-step guideline (**Figure 1** left). The material was designed to enable students to work as independently as possible from structure solution to refinement.

To further increase the students' motivation, the structure determination is best integrated into a practical lab course in which the substance under investigation is first isolated or synthesised by the students themselves. Ideally, substances that are chosen: (i) have a connection to everyday life such as citric acid (**Figure 1** right), sugars or acetylsalicylic acid and (ii) are known to repeatedly yield good quality single crystals when crystallised by students. When the course is held at the XLAB – Experimental Laboratory for Young People the students also visit the X-ray diffraction facilities of the Faculty of Chemistry at the University of Göttingen. They are shown how to select a single crystal with the aid of a microscope and how a measurement is set up combined with a short explanation on the essential parts of an X-ray diffractometer.

For the small molecule structures investigated with the students, structure solution is intentionally not performed with a current SHELXT[4] version but with direct methods in SHELXS (via ShelXle) or the charge-flipping method in Olex2. In this way, the students get a starting model consisting of only carbon atoms. They can then apply their knowledge of chemistry and the information gathered in the practical course (e.g. tests for functional groups) to assign the correct atom types, similar to a puzzle game. After each step of the refinement, students can evaluate the changes they made to the model based on the visualised residual density map and/or ADP shapes. As a final result they obtain a picture or short animation of a 3D molecular model that also crowns their efforts in the lab with success.



Refining

To refine the structure further, we now open the "Refine" submenu (the arrow to the right of the Refine button) and check the settings.

In "Cycles" we can set how often the program refines with each "Refine" command. Peaks specifies the maximum number of Q-peaks that are displayed. Set cycles to at least 4, and peaks to at least 11.

After these settings are applied click on the "Refine" button.

The „Refine" Button

This opens the Refine submenu.

Number of refinement cycles which are executed with each "Refine" command.

Number of Q-peaks displayed at the same time.

Program olex2.refine G-N
Cycles 4
Peaks 11
hkl file test.hkl
Mon May 15 14:49:43 2023
Use Solvent Mask This is the Olex2 implementation of BYPASS (a.k.a. SQUEEZE)

Figure 1. Left: Snapshot of the step-by-step guide. Right: Result of the structure determination of citric acid by students.

*[1] W. H. Bragg, Concerning The Nature Of Things: Six Lectures delivered at the Royal Institution, G. Bell And Sons Ltd., London, **1925**.*

*[2] a) B. F. Abrahams, C. J. Commons, R. E. Hill, T. A. Hudson, J. J. Jackowski, N. L. Peters, E. E. Rochette, R. S. Arlt, C. Walkear, J. Chem. Educ. **2023**, 100, 732–738. b) A. M. Beauparlant, C. T. Eagle, R. Mohseni, C. D. McMillen, J. Chem. Educ. **2023**, 100, 336–341.*

*[3] a) O. V. Dolomanov, L. J. Bourhis, R. J. Gildea, J. A. K. Howard, H. Puschmann, J. Appl. Cryst. **2009**, 42, 339–341. b) C. B. Hübschle, G. M. Sheldrick, B. Dittrich, J. Appl. Cryst. **2011**, 44, 1281–1284.*

*[4] G. M. Sheldrick, Acta Cryst. **2015**, A71, 3-8.*

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