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Collecting 3D Electron Diffraction Data for Crystal Structure Determination. Sven Hovmöller[a], Daliang Zhang[a], Daniel Grüner[a], Xiaodong Zou[a], Peter Oleynikov[a]. [a]*Structural Chemistry, Stockholm University, Sweden.*
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Possible methods for collecting complete 3D electron diffraction data from crystals much smaller than 1μm^3 are described. Data can be collected on a CCD camera and automatically extracted by computer. The critical question of data quality is addressed – can electron diffraction data compete with X-ray diffraction data in terms of resolution, completeness and quality of intensities?

Keywords: microcrystals; electron diffraction; electron microscopy

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Structure Determination of IM-5 by Electron Microscopy Alone. Junliang Sun[a,b], Zhanbing He[a], Sven Hovmöller[a], Xiaodong Zou[a,b], Fabian Gramm[c], Christian Baerlocher[c], Lynne B. McCusker[c]. [a]*Structural Chemistry, Stockholm University, Sweden.* [b]*Berzelii Centre EXSELENT on Porous Materials, Stockholm University, Sweden.* [c]*Laboratory of Crystallography, ETH Zurich, Switzerland.*
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IM-5 structure was first reported in 1998.[1] Due to the complexity and impurities, it could not be solved by X-ray diffraction alone until eight years later Baerlocher et. al. solved it in a charge-flipping structure solution algorithm by combining powder X-ray diffraction and electron microscopy.[2] Electron microscopy combined with image processing has been proved to be a powerful tool for the structure determination.[3] To test the limitation of electron crystallography, the IM-5 structure was redetermined by the electron microscopy technique alone. The unit cell was obtained by tilt series of selected area electron diffraction (SAED) patterns. Combining reflection conditions from SAED patterns and projection symmetries from high resolution transmission electron microscopy (HRTEM) images, the space group was determined as *Cmcm*. A 3D potential map was reconstructed from HRTEM images along three main zone axes shown below, from which all 24 unique Si positions were obtained. After adding oxygen atoms between each Si-Si pairs, distance least-squares refinement was done by DLS-76.[4] The final structure model deviates on average by 0.16Å for Si and 0.31Å for O from that refined using X-ray powder diffraction data.

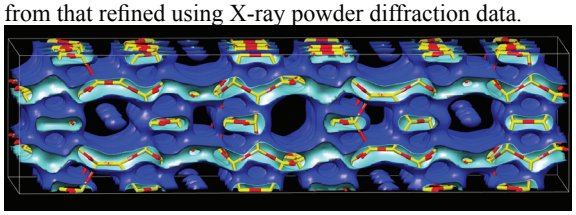

Figure 1. A 3D potential map reconstructed from 144 reflections. The Si net is superimposed. Dark blue is outside and light blue inside the walls.

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Keywords: electron crystallography; zeolite structures; image processing theory

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Application of Automated Diffraction Tomography to Structural Solution of Inorganic Phases: Charoite. Entico Mugnaioli[a], Ute Kolb[a], Ira Rozhdestvenskaya[b,c], Wulf Depmeier[c], Michael Czank[c]. [a]*Insitute for Physical Chemistry, Johannes Gutenberg University Mainz, Germany,* [b]*Saint Petersburg State University, Dept. of Crystallography, Saint Petersburg, Russia,* [c]*Institute of Geowissenschaften, Dept. of Crystallography, Christian-Albrechts-University, Kiel, Germany.*
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The investigation of the nano scale is nowadays one of the most challenging borders for crystallography. Electron diffraction is able to give high resolution structural data from single nano-crystals, but is often biased due to strong aberration of intensities and the difficulties to acquire rich data sets from manual tilt. Automated diffraction tomography (ADT) [1,2] complies a powerful and quick tool for a more reliable, complete and reproducible electron diffraction data collection from single nano-crystals. ADT data sets allow a 3D reconstruction and visualization of reciprocal space, an unambiguously cell parameters determination and a rich quasi-kinematical intensities collection, sufficient for ab initio structural solution [3]. On of the most challenging subject analyzed until now is charoite.
Charoite is a highly appreciated semi-precious stone of violet color. It was first found in the Murun alkaline massif (Siberia, Russia) more than 50 years ago. Space group P2/m was supposed from X-ray powder diffraction [4], and a structural model in which tubular dreier single chains are the main building units was proposed on the basis of high resolution transmission electron microscopy [5].
With ADT we were able to detect unambiguously two different polytypes with β=96° and β=90°. A structurally undisturbed small single fiber (200 nm in diameter) of polytype β=90° was chosen for intensities collection. 8495 reflections were acquired up to 0.7 Å resolution. Space group $P2_1/m$ was determined by intensities distribution and the structure was solved ab initio by direct methods and refined by Fourier maps and least squares. The new structure consists of 90 independent atoms, 53 of which are oxygen's. A refinement of the occupancies was also performed and the resulting crystal chemical formula is $(K_{13.4}Sr_{1.4})_{14.8}(Ca_{26.2}Na_{5.8})_{32}Si_{70}(O,OH)_{180}(OH,F)_{4.0}*4.8H_2O$.

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Keywords: electron diffraction tomography; structure analysis; charoite

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***"Ab initio"* Structure Solution of Organic Materials from Electron Diffraction to Mography Data.**

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Electron diffraction has been always an attractive method to solve crystal structures, firstly because of its fine lateral resolution (the method can easily probe volumes down to several tenth of nanometers, thus making nano phases as well as agglomerated materials assessable), secondly electrons have different scattering factors than X-rays, which allow to "see" relatively light elements in the presence of heavy atoms.
One of the major complications in the electron diffraction data processing is the presence of dynamical effects, which severely modify the data, and hamper structure determination. These difficulties are not so strong for organic materials, which typically diffract in "quasi-kinematical" manner.
Although use of electron diffraction data for structure solution of organic materials promises obvious benefits, it has been rarely used in the past [1, 2]. Recently a new method of collecting and processing of electron diffraction data was developed (ADT) [3, 4]. Practically the method allows sampling of up to 2/3 of reciprocal space from a single nano crystal. This data is powerful enough to give a solution of the structure directly from the raw data. Several organic structures of difference classes of compounds were solved from the ADT data "ab initio" using SIR08 [5].

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