to treat powder data is illustrated on a series of inorganic structures of variable complexity.

Keywords: direct methods, powder data, inorganic crystals

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Combining X-ray powder diffraction and electron microscopy to solve complex structures

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When a complex structure of a polycrystalline material cannot be solved from X-ray powder diffraction (XPD) or electron microscopy data alone, combining the two techniques might succeed. Phase information derived from high-resolution transmission electron microscopy (HRTEM) images can be combined with XPD intensities to facilitate structure solution. Alternatively, the single-crystal intensities from electron diffraction (ED) data can be used to improve the estimate of the relative intensities of reflections that overlap in the XPD pattern. The first approach has already proven to be a powerful one, but it requires HRTEM images, which are not always easy to obtain. The second is the subject here. Tests show that intensities from conventional selected area electron diffraction patterns are not very reliable because of multiple scattering effects, so they cannot be used directly to repartition the intensities of overlapping reflections. However, the precession electron diffraction (PED) technique reduces these effects. The PED intensities are still distorted, so we took a very conservative approach, using them simply to identify the weak reflections. These reflections were then eliminated from the XPD intensity extraction. Initial tests of the feasibility of this approach were performed on the zeolite ZSM-5 using the powder charge flipping (pCF) program and simulated ED patterns along 3 zone axes. Then real PED data were used. In both cases, the final pCF electron density maps revealed the positions of all 12 Si and 26 O atoms in the framework structure. This is not the case when the original XPD data without weak reflection elimination are used. It appears that even this simple modification of the dataset has a significant impact on structure solution.

Keywords: electron crystallography, powder diffraction, structure determination

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Powder diffraction, electron microscopy, focus, charge flipping and zeolites

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As a result of impressive methodological advances in recent years, the determination of a crystal structure from powder diffraction data is no longer a rarity. However, some zeolite structures remain intractable. To address such problems, the powder diffraction data can be supplemented with information from other sources. Both the zeolite-specific structure-solution program Focus and the more generally applicable powder charge-flipping (pCF) algorithm have been adapted to accommodate such data combinations, and this has allowed the complex structures of TNU-9 (24 Si atoms), IM-5 (24 Si atoms) and SSZ-74 (23 Si atoms) to be solved. The key in all cases was the combination of high-resolution powder diffraction data with information derived from high-resolution transmission electron microscopy (HRTEM) images. For TNU-9, Focus was used to combine the reflection intensities extracted from the powder diffraction pattern with phases and a structure envelope derived from HRTEM images. For the other two, the pCF algorithm implemented in Superflip was used to effect the combination. Both algorithms work in both direct and reciprocal space, so they are particularly well-suited for bringing data from different sources together, whether the data be in diffraction or real space. Adding even a very limited amount of phase information to the initial (usually random) phases in either algorithm, can make the difference between solving and not solving a structure, and this proved to be the case for TNU-9, IM-5 and SSZ-74. With 23-24 Si atoms in the asymmetric unit, these are by far the most complex zeolite framework structures known. The approach used to elucidate their structures is not only generally applicable, but many extensions to include other types of data can be envisioned.

Keywords: powder diffraction, electron microscopy, charge flipping

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First success in direct evaluation of electron density distribution of polymer by X- N method

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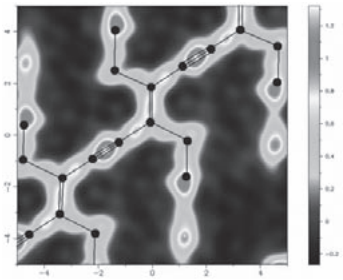
Bonded electron density distribution along the skeletal chains of polydiacetylene single crystal has been successfully and quantitatively evaluated for the first time on the basis of the organizedly combined X-ray diffraction and neutron diffraction data (X-N method). The application of X-X method, where only the X-ray diffraction data were used in an approximation, resulted in the abnormal distribution of electrons on the triple bond. This comes from ambiguity of atomic positions and an overestimation of temperature factors in the X-ray method since the atomic positions and thermal parameters were approximated from the information on the center of gravity of electron density clouds. Contrarily the neutron method gave us the exact positions and thermal parameters of atomic nuclei themselves. The X-N method was applied also to the low-molecular-weight model compounds to get the reasonable bonded electron density distributions. The thus evaluated electron density distributions were compared with those calculated by density functional theory.



Keywords: electron density distribution, polymer, X-N method