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Electrostatic potential and electric field imaging by MEM powder diffraction dada analysis

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Accurate powder diffraction experiment by synchrotron radiation has made progress on the charge density study by the Maximum Entropy Method (MEM). So far, our developed analytical method which is the combination of the MEM and Rietveld refinement, so called MEM/Rietveld method, has been successfully applied to the structure analysis of novel nano materials, ferroelectric materials, manganites and superconductors by synchrotron radiation powder data[1,2]. The obtained MEM charge density enabled us structure refinement as well as observation of interplays between atoms and molecules, such as, bonding nature, charge transfer and etc. The electrostatic potential based on the experimental charge density should be more informative to see the interaction between atoms and molecules. Recently, we have succeeded in developing electrostatic potential and electric field imaging based on MEM charge density analysis[3]. The visualized electrostatic potential of the typical ferroelectric material, tetragonal $PbTiO_3$ has uncovered the feature of electronic polarization in Ti-O and Pb charge densities. In addition, the result shows very good agreement with that obtained from the ab initio calculation[4]. Very recently, we have also succeeded in visualization of charge order associated with orbital order in manganites[5] . Our new method is promising and extending potential of powder diffraction structure refinement. In the talk, the detail of the method and application to the study of the metal-insulator transition of α-$(BEDT\text{-}TTF)_2I_3$ will be presented.

1.M. Takata, et al., Z. Kristallogr. **216**,71-86(2001)
2.M. Takata, Acta Cryst. **A64**,232-245(2008)
3.H.Tanaka, et al., Phys. Rev. **B74**,172105 (2006).
4.R. E Cohen, Nature **358**,136-138(1992).
5.K. Kato, et al.,Phys.Rev.**B77**,081101(2008)

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MAD techniques applied to powder data: The method of the joint probability distribution functions

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The great improvement of the methods for ab initio structure solution from powder diffraction data (PDD) is due to the use of synchrotron radiation: it provides high quality diffraction data, sometimes simulating single crystal data. It also allows a fine wavelength tuning, so giving new emphasis to the Multiple wavelength Anomalous Dispersion (MAD) methods. Phase determination via MAD techniques is usually a three step procedure:
1) estimation of structure factor moduli of the anomalous scatterer substructure [1]
2) location of the anomalous scatterers [2] [3]
3) protein phase estimation [4]
Although with PDD the experimental measure of the Bijvoet differences is lost, MAD techniques can still be usefully applied [5]. The first step of our approach deals with the evaluation of the structure factor moduli |Foa|, given the prior knowledge of the moduli measured at two different wavelengths. Direct Methods or Patterson deconvolution techniques are used to locate the anomalous scatterer substructure. The phasing of the whole structure is performed by applying the method of the joint probability distribution functions given the substructure. The procedure has been implemented within the package EXPO [6]. Applications to synchrotron data are discussed.

[1] Burla et al. (2002) Acta Cryst. D58, 928-935
[2] Burla et al. (2003) Acta Cryst. D59, 662-669
[3] Burla et al. (2007) J. of Appl. Cryst. 40, 211-217
[4] Giacovazzo et al. (2004) Acta Cryst. D60, 73-82
[5] Helliwell et al. (2005) Acta Cryst. A61, 568-574
[6] Altomare et al. (2004) J. of Appl. Cryst. 37,1025-1028

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Towards routine refinement of hydrogenous materials by neutron powder diffraction

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We have embarked on a substantial programme of research tackling the fact that the accurate determination of the position of hydrogen in a material is usually regarded as a challenging issue unless large single crystals are available for neutron diffraction. We are tackling this problem using the capabilities offered by modern high flux neutron powder diffractometers, such as D20 at the ILL, which average incoherent scattering from hydrogen and allow, through sheer counting statistics, its contribution to the Bragg scattering to be adequately analysed, allowing for structure determination. Results will be presented from successful initial proof-of-concept experiments covering both low hydrogen content inorganic materials, for example in the critical area of water location (Henry et al, Chem Comm, 2008) and also high hydrogen content molecular complex materials in the topical area of temperature-dependent proton migration and transfer (Pulham, Wilson et al, in preparation). The move forward in our studies of hydrogenous compounds using neutron diffraction is demonstrated by this applicability to organic molecular systems; specifically hydrogen-bonded molecular complexes for which it is challenging to grow even X-ray sized single crystals but for which neutron data are vital. In reinforcing the importance of this area, we note not only the fact that powder studies of hydrogenous materials open up new regimes for multi-parameter