

FA5-MS01-O1

Resolution Bias Correction in EXPO2009. Angela Altomare^a, Corrado Cuocci^a, Carmelo Giocovazzo^{a,b}, Sabino Maggi^a, Anna Moliterni^a, Rosanna Rizzi^a. ^a*CNR-Institute of Crystallography (IC), via Amendola 122o, Bari 70126, Italy.* ^b*Dept. of Geomineralogy, University of Bari, via Orabona 4, Bari 70125, Italy.* E-mail: angela.altomare@ic.cnr.it

Fourier syntheses providing electron density maps always show series-termination errors due to the limited experimental resolution. The worse the resolution, the worse the quality of the map: peaks are misplaced with respect to the true positions, positive and negative peaks occur, the peak profile is deformed. Two new algorithms have been developed aimed at reducing the resolution bias. The first one works in direct space [1,2], the second one in reciprocal space [3]. They are able to improve both peak positions and peak form in such a way that the map better fits the real atomic electron density. The new procedures have been introduced in EXPO2009, the strengthened and updated version of EXPO [4] and applied to the electron density maps provided by Direct Methods. Organic as well as metallorganic powder structures with non-atomic resolution experimental data have been tested. In spite of the difficulties in structure solution by powder data, the new approaches succeeded in several cases which could not be solved without the resolution bias correction. The theory of the resolution bias correction and its application by EXPO2009 will be presented.

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Keywords: ab-initio powder structure determination; resolution; electronic density

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New Frontiers in Powder Diffraction Pattern Indexing: the Program N-TREOR09. Anna Moliterni^a, Angela Altomare^a, Gaetano Campi^a, Corrado Cuocci^a, Lars Eriksson^c, Carmelo Giocovazzo^b, Rosanna Rizzi^a, Per-Erik Werner^c. ^a*IC, Sede di Bari, via Amendola 122/o, 70126 Bari, Italy.* ^b*Dipartimento Geomineralogico, Università di Bari, Campus Universitario, Via Orabona 4, 70125 Bari, Italy.* ^c*Department of Structural Chemistry, Arrhenius Laboratory, University of Stockholm, S-106 91 Stockholm, Sweden.* E-mail: annagrazia.moliterni@ic.cnr.it

Despite its long history and the recent advances in theoretical methods, computer speed and experimental devices, powder pattern indexing can still be a challenge. More plausible

different unit cells are often suggested, among which the identification of the correct cell can be difficult. The cell reliability is generally assessed *via* historical figures of merit (i.e., M_{20} by De Wolff [1] and F_N by Smith & Snyder [2]) that sometimes fail. Usually the space group identification follows the indexing. We propose a new approach which simultaneously determines the unit cell and the extinction symbol by *N-TREOR09* (the evolved version of the indexing package *N-TREOR* [3]). A new figure of merit has been also introduced in *N-TREOR09*. It has been proved to be more powerful than M_{20} and combines the contribution by M_{20} with additional parameters like the number of unindexed lines, the agreement between the calculated and observed profiles, the degree of overlap in the pattern, the symmetry suggested by the automatic evaluation of the extinction group. *N-TREOR09* has been also enriched by an improved indexing procedure in the triclinic system and integrated into EXPO2009, the updated version of EXPO2004 [4]. The main features of *N-TREOR09* and its applications to a large set of test structures will be described.

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Keywords: powder indexing; space-group determination from powders; crystallographic computing

FA5-MS01-O3

Average and Local Structure of Laves Phase Deuterides YFe₂D_x. Joanna Ropka^a, Radovan Černý^a, Valerie Paul-Boncour^b, Michel Latroche^b, Thomas Proffen^c. ^a*Laboratory of Crystallography, University of Geneva, quai E.-Ansermet 24, CH-1211 Geneva, Switzerland.* ^b*CMTR, ICMPE, CNRS, 2 rue H. Dunant, 94320 Thiais Cedex, France.* ^c*Lujan neutron Scattering Center, LANL, Los Alamos, NM 87545, USA.*

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Between all cubic (C15) Laves phases, widely investigated for years, the YFe₂D_x system is especially interesting because of its magnetic properties. The structure of particular YFe₂D_x deuteride depends on deuterium content, likewise deuterium and magnetic ordering temperatures [1].

The most rich phase of this system under ambient condition - YFe₂D_{4.2} - is formed as a cubic (*Fd3m*) and monoclinic (*Pc*) above and below deuterium ordering temperature, respectively. The deuteriums in disordered (cubic) structure partially occupies 3 sites, while occupancies in fully ordered monoclinic structure equal to 1 for almost all of 18 deuterium sites. Deuterium atoms make polyhedras (1 tetrahedron, 7 trigonal bipyramids - fig.1) around iron with distances Fe-D between 1.62-1.83 Å. [2]

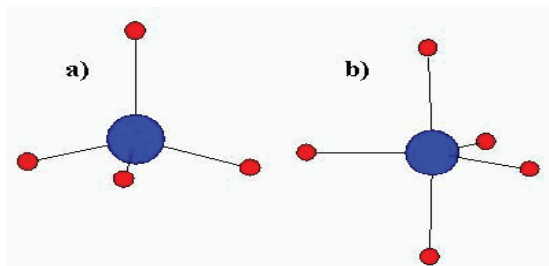


Fig.1. Coordinates polyhedra around Fe atoms: a) tetrahedron, b) trigonal bipyramid

The Rietveld analysis gives us information about changes of average structures (cubic \leftrightarrow monoclinic) but Pair distribution Function (PDF) methods let us describe the local configuration of deuterium atoms which stays almost the same in order and disordered phases (fig.2). Neutron Time-of-Flight (ToF) data were collected at IPNS, Argonne, and Lujan Center, Los Alamos on series of samples with different deuterium content. Each sample was measured below and above the temperature of deuterium ordering. The local deuterium configuration in disordered phase have been modeled by DISCUS using Revers Monte Carlo technique [3].

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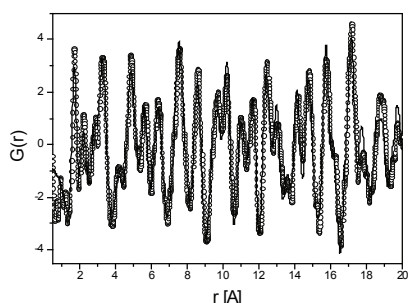


Figure 2. Observed PDF (points) of $\text{YFe}_2\text{D}_{4.2}$ in disordered state and modeled (solid line) by same local order of deuterium atoms around iron as in the ordered phase.

FA5-MS01-O4

Structural Investigations of LnBO_3 (Ln= Y, La, Nd, Sm, Eu, Gd, Dy, Ho, Er, Yb, Lu) by Rietveld Method. Semih Seyyidoglu^a, Katrin Hoffmann^b, Barbara Albert^b, Aysen Yilmaz^a. ^aDepartment of Chemistry, Middle East Technical University, 06531, Ankara, Turkey. ^bEduard-Zintl-Institut für Anorganische und Physikalische Chemie, Petersenstr. 18, 64287, Darmstadt, Germany.

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Numerous efforts have been devoted to the structure determination of these compounds in literature. While LaBO_3 and NdBO_3 exhibit the aragonite type (orthorhombic Pnma) structures [1-2], the structure vaterite type rare earth borates (Y, Gd-Yb) are still under investigation since the structure of these compounds are directly related with their synthesis method. The structure of GdBO_3 were defined in rhombohedral space group R32 by Lin and coworkers [3]. While in Sheptyakov's work[4], Eu doped yttrium orthoborate structure is monoclinic space group C2/c, Chadeyron produced YBO_3 single crystals with P63/m space group [5]. Since there is no systematic structural analysis work of whole rare earth borate series, in this work we synthesized and solved their structure by using Rietveld Method [6] with GSAS program[7]. We prepared LnBO_3 (Ln=Y, La, Nd, Sm, Eu, Gd, Dy, Ho, Er, Tm, Yb, Lu) powder samples by using Ln_2O_3 and H_3BO_3 (ratio=1:2) heated at 900 °C for 10 hour and 1000 °C for 5. Then, their XRD patterns were collected on a PANalytical X'PERT PRO diffractometer equipped with PIXCEL detector. Among these rare earths, LaBO_3 and NdBO_3 were solved based on Pnma orthorhombic structure. The crystal structure of YBO_3 , DyBO_3 and HoBO_3 were C2/c monoclinic and SmBO_3 showed P-1 triclinic structure. The structure of TbBO_3 , TmBO_3 and YbBO_3 were solved based on P21/m.

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Keywords: inorganic solid state chemistry; boron compounds; rietveld structural analysis

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Metal and Chemical Hydrides: Highlights and Pitfalls of Powder Diffraction. Radovan Černý. Laboratory of Crystallography, University of Geneva, 24, quai Ernest-Ansermet, CH-1211 Geneva 4, Switzerland.

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The methods of structural characterization of metal and chemical hydrides are reviewed (see also [1]). It is shown that powder diffraction is essential component of hydrides research where the structural characterization is currently undertaken by X-ray and neutron diffraction. In the case of chemical hydrides like borohydrides of light alkaline metals/earths X-ray diffraction alone can provide the structural parameters with sufficient accuracy. A crystallographer analyzing hydrides has to face numerous crystallographic challenges which include complex structures, superstructures, pseudo-symmetries, twinning,