Microsymposia

The EXPO package [1] is a computing program able to successfully provide the structure solution by starting from minimal information: the experimental powder diffraction pattern and the chemical formula of the compound to investigate. EXPO can be used for solving organic, inorganic, metalorganic structures for a large variety of applications. Automation and efficiency are suitably combined in EXPO for performing all the steps of the solution process: indexation, space group determination, intensity extraction, structure solution, model optimization, Rietveld refinement.

Automation. Default strategies, identified as the most effective, are selected for carrying out the solution process automatically and quickly. They may fail when the experimental data resolution is bad and/or the structure complexity is remarkable. In these cases, EXPO can promptly switch to appropriate strategies by profiting by a user friendly graphical interface.

Efficiency. Innovative theories and computing procedures aiming at making straightforward all the steps of the solution process are integrated in EXPO and widely tested. EXPO is continuously updated and optimized in terms of both computing efficiency and graphical performances.

In EXPO the two stages based *ab initio* approach (in the first stage the intensities are extracted; in the second, the reflections are phased by Direct Methods) [2] is the automatic choice. The model provided by Direct Methods is usually partial and approximate because of well known unavoidable problems in powder diffraction (overlapping, background, preferred orientation). It can be optimized by default [3], [4] and/or non-default strategies [5], [6] which are able to reduce the errors depending on the limited experimental resolution.

A very recent study has regarded a new and more effective figure of merit [7], alternative to the classical one. It is able to pick up the phasing trial corresponding to the best solution among several feasible ones.

Non ab initio method, requesting the knowledge of the expected molecular geometry and based on Simulated Annealing technique [8, 9], can optionally be attempted particularly in case of solution of organic compounds.

Examples of successful automatic runs by EXPO will be discussed in addition to available special strategies.

[1] A. Altomare, M. Camalli, C. Cuocci, C. Giacovazzo, A. Moliterni, R. Rizzi, *J. Appl. Cryst.* **2009**, *42*, 1197-1206. [2] R. Caliandro, C. Giacovazzo, R. Rizzi, **2008** in *Powder Diffraction Theory and Practice*, ed. by R.E. Dinnebier and S.J.L. Billinge, RCSPublishing. [3] A. Altomare, C. Cuocci, C. Giacovazzo, G.S. Kamel, A. Moliterni, R. Rizzi, *Acta Cryst.* **2008**, *A64*, 326-336. [4] A. Altomare, C. Cuocci, C. Giacovazzo, A. Moliterni, R. Rizzi, *J. Appl. Cryst.* **2008**, *41*, 592-599. [5] A. Altomare, C. Cuocci, C. Giacovazzo, A. Moliterni, R. Rizzi, *Acta Cryst.* **2009**, *A65*, 183-189. [6] A. Altomare, C. Cuocci, C. Giacovazzo, A. Moliterni, R. Rizzi, *J. Appl. Cryst.* **2010**, *43*, 798-804. [7] A. Altomare, C. Cuocci, C. Giacovazzo, A. Moliterni, R. Rizzi, *J. Appl. Cryst.* **In Press.** [8] A. Altomare, R. Caliandro, C. Giacovazzo, A. Moliterni, R. Rizzi, *J. Appl. Cryst.* **2003**, *36*, 230-238. [9] A. Altomare, C. Cuocci, C. Giacovazzo, A. Moliterni, R. Rizzi, *J. Appl. Cryst.* **2008**, *41*, 56-61.

Keywords: powder diffraction, structure solution, software

MS.95.3

Pushing the limits of powder charge flipping

Lynne B. McCusker,[a] Christian Baerlocher,[a] Lukas Palatinus,[b] [a]*Laboratory of Crystallography, ETH Zurich, (Switzerland).* [b]*Institute of Physics, Academy of Sciences, (Czech Republic).* E-mail: mccusker@mat.ethz.ch

In 2004 Oszlányi and Sütö introduced a new way of determining crystal structures from single-crystal diffraction data that they termed charge flipping [1]. The algorithm is an elegant one, based on a very simple perturbation (charge flipping) of electron-density maps that are generated during the structure solution process. Initially, this charge-flipping algorithm was viewed as a curiosity, but in a surprisingly short period of time the approach found application in areas where traditional methods flounder. One of these was X-ray powder diffraction (XPD).

The original algorithm was adapted to accommodate powder diffraction data by two different groups in 2006 [2] and 2007 [3]. The latter includes a second perturbation of the electron-density map based on histogram matching [4] prior to the repartitioning of the intensities of overlapping reflections, and has been implemented in the program *Superflip* [5]. Although diverse structures have been solved automatically with this program, its limits have not yet been reached. By including information from electron microscopy (EM) experiments in different ways, three particularly complex zeolite structures [6,7,8] could be solved. At least part of the algorithm's strength lies in the fact that it operates in both real and reciprocal space, so manipulation in both is possible.

In the first of the zeolite structures, starting phases for the charge-flipping algorithm were generated using an approximate (but incorrect) model derived from several high-resolution transmission electron microscopy (HRTEM) images. In the second case, a structure envelope [9] was generated from a single lower resolution HRTEM image and imposed in real space to enforce the channel system. In the third case, reflection intensities from selected area electron diffraction patterns were used to pre-partition the intensities of reflections overlapping in the powder diffraction pattern.

It has also been shown that precession electron diffraction (PED) data can be used to improve the quality of the intensities extracted from a powder diffraction pattern (by identifying and eliminating weaker reflections) and to generate some better starting phases (by applying charge flipping to 2-dimensional PED data) [10]. In light of the improvements arising from the use of 2-dimensional EM data to supplement the powder diffraction data, an alternative approach using 2-dimensional subsets of the XPD data has recently been devised [11].

Further automation incorporating some of these developments into the software can be easily envisioned.

[1] G. Oszlányi, A. Sütö, *Acta Crystallogr. A* **2004**, *60*, 134-141. [2] J.S. Wu, K. Leinenweber, J.C.H. Spence, M. O'Keeffe, *Nature Mater*. **2006**, *5*, 647-652. [3] C. Baerlocher, L.B. McCusker, L. Palatinus, *Z. Kristallogr*. **2007**, *222*, 47-53. [4] K.Y.J. Zhang, P. Main, *Acta Crystallogr. A*, **1990**, *46*, 41-46. [5] L. Palatinus, G. Chapuis, *J. Appl. Crystallogr*. **2007**, *40*, 786-790. [6] Ch. Baerlocher, F. Gramm, L. Massüger, L.B. McCusker, Z.B. He, S. Hovmöller, X.D. Zou, *Science*, **2007**, *315*, 1113-1116. [7] C. Baerlocher, D. Xie, L.B. McCusker, S.J. Hwang, I.Y. Chan, K. Ong, A.W. Burton, S.I. Zones, *Nature Mater*. **2008**, *7*, 631-635. [8] J.L. Sun, C. Bonneau, A. Cantin, A. Corma, M.J. Diaz-Cabañas, M. Moliner, D.L. Zhang, M.R. Li, X. D. Zou, *Nature*, **2009**, *458*, 1154-1157. [9] S. Brenner, L.B. McCusker, Ch. Baerlocher, *J. Appl. Crystallogr*. **1997**, *30*, 1167-1172. [10] D. Xie, Ch. Baerlocher, L.B. McCusker, *J. Appl. Crystallogr*. **2008**, *41*, 1115-1121. [11] D. Xie, Ch. Baerlocher, L.B. McCusker, submitted.

Keywords: powder diffraction, charge flipping, structure determination

MS.95.4

Algorithm and performance of a new powder indexing software *Conograph*

Ryoko Oishi-Tomiyasu, Takashi Kamiyama, *Institute of Materials Structure Science, High Energy Accelerator Research Organization (Japan).* E-mail: ryoko.tomiyasu@kek.jp