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Ab initio structure solution from powder diffraction data using charge flipping

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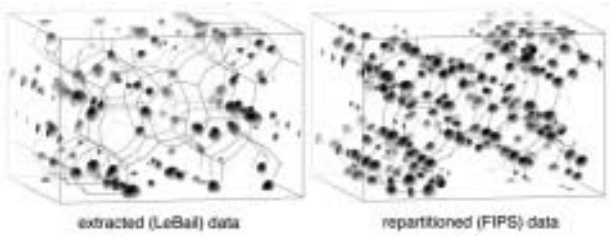
The algorithm dubbed charge flipping (CF) is an alternative method for structure solution that uses an iterative procedure to reconstruct approximate electron densities obtained from diffraction data [1,2]. The CF algorithm is highly successful for medium-sized structures, and seems to work best when large areas of ''empty space'' are present. This criterion of ''emptiness'' is also fulfilled by zeolites, so an investigation of the potential of the CF algorithm for solving complex zeolite structures from powder diffraction data was undertaken.

Real powder data suffer from limited resolution in reciprocal space and overlap of reflections in the diffraction pattern. In a series of tests, the tolerance of the CF procedures to these limitations was probed. For example, both simulated and real data for the zeolite ZSM-5 (*Pnma*, a=20.1Å, b=19.7Å, c=13.1Å, 38 atoms in the asymmetric unit) were studied.

For simulated single crystal data with d(min) = 1.1 Å, CF worked very well. Even when the intensities of 506 groups of overlapping reflections were equipartitioned (1428 of 2188 reflections), a good solution resulted, but when the overlap was increased to 1755 reflections, **no** solution was obtained. However, when these overlapping intensities were repartitioned using the FIPS (fast iterative Patterson squaring [3]) algorithm, all 38 atoms were found. Using real data with d(min) = 0.98Å and FWHM ranging from 0.05 to 0.09°2θ, the following electron density maps were obtained:



For the FIPS repartitioning, 471 overlap groups (criterion for overlap: 0.2* FWHM) were formed and 2630 out of a total of 3052 reflections were repartitioned.

These results show that the CF algorithm works well with good powder data and minimal overlap, even for limited resolution. Futhermore, for data with significant overlap, repartitioning using FIPS can also yield good results. However, the usual convergence tests did not work for the powder data. The Fourier map shown on the right was the best among a series of trials, and had to be selected by visual inspection.

A number of adaptations to the CF algorithm have been implemented in order to make it work with real powder diffraction data from materials of unknown structure.

[1] Oszlányi, G. and Sütö, A. (2004). *Acta Cryst*. A60, 134-141
[2] Oszlányi, G. and Sütö, A. (2005). *Acta Cryst*. A61, 1347-152
[3] Estermann, M.A. and Gramlich, V. (19993). *J. Appl. Cryst.* 26, 396-404.

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Studies of Two Polymorphic Systems: 2-Chloro-4-nitrobenzoic Acid and (2-Furyl) oxoacetamide

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In 1951, it was shown by Ebert and Gottlieb that infrared spectrometry could be a useful tool for the study of polymorphism in organic compounds [1]. One of the examples of its use was 2-chloro-4-nitrobenzoic acid. That compound was obtained in two polymorphic forms: Modification I was produced by crystallization from benzene and Modification II formed from the slowly cooled melt of the acid. The two modifications can easily be distinguished by their infrared spectra. Further investigations on this polymorphic system were reported in 1987 by Kuhnert-Brandstätter and Riedmann [2]. They used Kofler thermomicroscopy, differential scanning calorimetry (DSC) and infrared spectra to identify and characterize the two crystals forms. Here, we report for the first time, on the crystal structures of the two polymorphs of 2-chloro-4-nitrobenzoic acid. The crystal structures were solved by single crystal X-ray methods. We also reinvestigated and characterized the two forms by powder diffraction, FT-IR and thermal analysis techniques. The polymorphism of (2-furyl) oxoacetamide was discovered in 1988 by Kellner, Kuhnert-Brandstätter and Malissa [3]. The commercial product exists in a low-temperature phase (Form 2) and can be transformed by heating into high-temperature modification (Form 1). The third form can be observed by slow cooling of the melt on the hot-stage, and it transforms to the form1 above 75°C. We have solved the crystal structures of Form 1 and 2: one by single crystal method and the second from synchrotron powder diffraction. These polymorphic forms also were studied by thermal methods, FT-IR and powder diffraction.

[1] Ebert, A. A., Jr. and Gottlieb, H. B., *J. Am. Chem. Soc*.,1952, 74, 2806-2810.
[2] Kuhnert-Brandstätter, M; Riedmann, M., *Microchimica Acta*., 1987, 2, 107-120.
[3] Kuhnert-Brandstätter, M; Riedmann, M., *Microchimica Acta*., 1988, 3, 153-165.