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Extracting structural information from protein powder diffraction data

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It is sometimes difficult and/or time-consuming to obtain crystals of suitable size for single crystal X-ray diffraction measurements. However if a microcrystalline material can be obtained, it is still possible to perform powder diffraction experiments. Although the collapse of the three-dimensional reciprocal space into a one-dimensional powder diffraction pattern gives rise to a severe loss of information, the availability of high-resolution powder diffractometers and synchrotron sources enables the recording of high-quality powder patterns from proteins. From these spectra, a set of valid intensities can be extracted and/or Rietveld refinement can be carried out [1-2]. It is thus possible to investigate the binding of ligands or heavy atoms to biological macromolecules [3]. We present here results obtained recently in this field. Emphasis will be put on low-resolution phase determination by the methods of isomorphous replacement and contrast variation.
By using extracted intensities from a series of powder diffraction patterns, the location of heavy-atom is possible and a low resolution structure envelope could even been obtained for some small proteins. We present here data for PPE (porcine pancreatic elastase) in which the radiation-induced anisotropic lattice expansion was used as a way to uncorrelate the intensities of overlapping peaks.
On the other hand, contrast variation by solvent exchange is commonly used in small-angle scattering studies, both with X-rays and neutrons, and allows ab initio phasing and molecular envelope determination [2]. We present here our first results on using the solvent contrast variation method in powder diffraction studies.

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Crystallographic study and comparisons of compounds $A_2Sr_2B_2C_2O_X$

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The structural study of compounds, produced by heating of mixtures corresponding to the general chemical type of the type $A_2Sr_2B_2C_2O_X$, where A is Bi, B is Ba or Cd or combinations of them and C is Cu are presented in this paper. Powder mixtures with suitable proportions of Bi_2O_3, $SrCO_3$, CdO, BaO and CuO were prepared and heated in air, at a temperature of 650°C, until 900°C with a step of 30°C for 24h or more. After the powdering, we measured the samples taking XRD measurements with using a two cycle diffractometer with Bragg-Bretano geometry and CuKa radiation. The crystalline phases were characterized, using the PDF2 database and the help of bibliography. All the unit cell constants and crystal structure parameters were refined by the using of Powder Profile Analysis (Rietveld's method).The refinement took place starting step by step and finding the phases that represent. The crystallographic study of the samples and the exactly determination of the structures and phase percentages is the final result.