

morphous replacement (MIR) phases were determined by matching histograms of electron density. Accurate metal cluster geometries and the associated errors in atomic positions can be determined from refinement against anomalous differences using normal scattering phases from a refined structure. When applied to MAD data collected on SIRHP, these methods confirmed the 4Fe-4S cluster asymmetry initially observed in the refined 1.6 Å resolution structure and resulted in a MAD-phased, experimental, electron-density map that is of better quality than the combined MAD/MIR map originally used to solve the structure.

PS02.04.14 SIMPROT: A PROGRAM IN DEVELOPMENT FOR CRYSTAL STRUCTURE DETERMINATION OF PROTEINS. F.R. Seljee, R. Peschar and H. Schenk, Laboratory for Crystallography, Amsterdam Institute for Molecular Studies (AIMS), University of Amsterdam, Nwe. Achtergracht 166, 1018 WV Amsterdam, The Netherlands

An overview will be presented of the Direct Methods program SIMPROT that is designed to deliver *ab initio* an initial model of a protein structure if isomorphous data sets are available, e.g. from SAS, MAD or SIR experiments. Basically, SIMPROT follows the same methodology as employed in the Direct Methods software package SIMPEL [1]. However, it has been suitably modified in accordance with the difference structure factor formalism that has been developed recently [2], [3], [4].

The latter formalism is based on using the difference between two isomorphous structure factors as variable in the derivation of the Joint Probability Distributions upon which Direct Methods are based. It has been shown that this approach leads to accurate phasesum invariant estimates. A graphical overview of SIMPROT is given and some preliminary results will be discussed.

References:

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Structure Determination Using Powder Data

MS02.05.01 SUPRAMOLECULAR STRUCTURES FROM HIGH RESOLUTION POWDER DIFFRACTION. R. E. Dinnebier, Lehrstuhl fuer Kristallographie, University of Bayreuth, 95440 Bayreuth, Germany

Over the past few years, the feasibility of determining crystal structures *ab initio* from powder diffraction data has been steadily improved. Although a number of complicated inorganic crystal structures have been solved by this method, very little has been done in the field of supramolecular structure determination, namely for organic- and organometallic compounds. Assuming the material itself is well crystallized, the use of Synchrotron radiation is necessary to get a resolution as high as possible over the entire angular range of the powder pattern. Besides the higher resolution, advances in the computational aspects of the problem are also crucial for structure determination. Especially the development of more sophisticated grid search techniques of molecular fragments, considering geometrical and physical aspects of the crystal, is an important step forward in finding the right solution. The structure solutions presented here stand for some of the most complicated organic and organometallic structures which have ever been solved *ab initio* from high resolution powder data. They include the high and the low temperature phase of the

Ru-sawhorse dimer $[\text{Ru}_2(\text{O}_2\text{PMe}_2)_2\text{CO}]_4\text{In}$ (1), the industrial important (Kolbe-Schmitt-synthesis) phenolates $\text{C}_6\text{H}_5\text{OA}$ (A= K, Rb, Cs) (2), $\text{C}_6\text{H}_5\text{OK}$ $2(\text{C}_6\text{H}_5\text{OH})$ (2), $\text{C}_6\text{H}_5\text{OK}$ $3(\text{C}_6\text{H}_5\text{OH})$ (2), Na-Para-Hydroxy-Benzoate $\text{NaC}_7\text{H}_5\text{O}_3$ (2), base free alkaline-cyclo-pentadienide $\text{C}_5\text{H}_5\text{A}$ (A=Li,Na,K,Rb) and the triclinic low temperature form of $\text{C}_{60}\text{Br}_{24}(\text{Br}_2)_2$ (3). In addition to well known techniques such as direct methods, difference Fourier synthesis, Patterson maps and Rietveld analysis, the newly proposed pseudo-atom method proved to be a very efficient tool to solve all structures containing well defined molecular fragments. In the case of $\text{C}_{60}\text{Br}_{24}(\text{Br}_2)_2$ the orientation of the well defined $\text{C}_{60}\text{Br}_{24}$ molecules in a triclinic distorted fcc lattice could be found unambiguously by maximizing of nearest neighbor distances. The structural motive of the high temperature form of the Ru-sawhorse dimer was found by conventional direct methods. The similarity criterion for the low temperature phase resulted in a restricted 4-dimensional grid search.

It can be shown that the precision is comparable to that achievable with single crystal techniques, and, therefore, allows for the interpretation of binding mechanism and reactions. Nevertheless, much more work is required in developing these structure solution methods further.

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MS02.05.02 SOLUTION AND REFINEMENT OF DRUG STRUCTURES FROM POWDER DATA. Kenneth Shankland, ISIS Facility, Rutherford Appleton Laboratory, Chilton, Didcot, Oxon OX11 0QX, U.K.

The vast majority of pharmaceuticals used in everyday life are moderately sized organic compounds. Usually, their molecular structures are known from single crystal X-ray experiments. However, polymorphism (which is frequently found in pharmaceuticals) often makes the determination of a particular crystal form very difficult. In such cases, obtaining a structure solution from powder data is an attractive option, but one which presents a considerable challenge given that most of the compounds of interest will crystallise in low symmetry space groups with large unit cells, leading to complex diffraction patterns with lots of peak overlap. Model building is usually precluded due to conformational flexibility in the molecules, so *ab initio* methods offer the best hope of a solution.

Computational strategies such as standard direct methods, combined maximum entropy / log-likelihood gain and optimal extraction of structure factors will be discussed, but a particular emphasis will be placed on using sample preparation and data collection strategies to maximise the chance of obtaining a structure solution.

MS02.05.03 ZEOLITE STRUCTURE DETERMINATION FROM POWDER DATA: COMPUTER-BASED INCORPORATION OF CHEMICAL INFORMATION. R.W. Grosse-Kunstleve, L.B. McCusker & Ch. Baerlocher, Laboratory of Crystallography, ETH Zentrum, CH-8092 Zurich.

Many technologically and industrially important materials, including zeolites, are synthesized and used in polycrystalline form. Since the crystal structures of such phases often determine their useful properties, it is essential that methods to study their structures are available.

The FOCUS method, which incorporates the use of chemical information into the structure determination process, has been developed. FOCUS combines automatic Fourier recycling (using