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$ScRh_3B_x$(x=0.0-1.0) has been investigated recently as an ultra-hard material. Crystal structure refinements and electron density analyses of this material were carried out by synchrotron X-ray powder diffraction. The powder diffraction data were collected using Multi-Detector System powder diffractometer at the BL-4B2 experimental station of the Photon Factory. The crystal structure refinements were performed using the Rietveld method and the electron density maps were calculated with the Maximum Entropy Method (MEM). The results of the refinements show that the crystal structure of $ScRh_3B_x$ is cubic with Pm3m space group, which has same atomic arrangement with perovskite structure. The lattice constant increases linearly according to the increase of B amount. In the electron density maps obtained by MEM analysis, electron density raises are obviously observed between B and Rh atoms. The rises of electron density show the existence of covalent bond between B and Rh atom. In spite of the liner increase of lattice constant according to the increase of B amount, the hardness of this series of compounds have a minimum between 0.4 and 0.7 of B contents. This change of hardness is supposed to be related to the amounts of the covalent bond in the crystal structure. The bond character of this series of compounds is also discussed based on the results of electron density analyses.

Keywords: borides, electron density, powder diffraction

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Structure Determination of a Novel Protein by Sulphur SAD using Novel Crystal mounting Method

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A crystal mounting technique was developed for the sulphur SAD method using longer wavelength X-rays. This technique is novel in that the a nylon loop is glued directly onto the tip of the micropipette and fixed as if the micropipette tip is located in the loop, so the solution caught in the loop can be aspirated through the micropipette just before flash freezing. Using this technique, the cryo-buffer and cryoloop can be removed easily before data collection to eliminate their X-ray absorption. The structures of novel proteins were solved using this technique in combination with chromium radiation. In the case of PH1109 from *P. horikoshii*, 90% of all residues were built automatically by *RESOLVE* using this technique, but only 76% were built for the dataset obtained using the standard loop. These results indicated that our crystal mounting technique was superior to the standard loop mounting method for the measurement of small anomalous differences, and yielded good results in sulphur substructure solution and phasing. We will demonstrate that the sulphur SAD method with a chromium source is more practical for macromolecular structure determination using our crystal mounting technique.

Keywords: sulphur, SAD, crystal mounting method

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IL MILIONE: A Complete Package for a Global Phasing, from Powders to Proteins

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IL MILIONE is a multipurpose compact, user friendly, efficient package for the global phasing of the crystal structures. The following tasks can be accomplished:

a) phasing and refining powder data. The program EXPO2004 [1] has been incorporated;

b) ab initio crystal structure solution of small, medium and macromolecules. The program SIR2004 [2] has been incorporated. Structures can be solved both by Patterson and Direct Methods (resolution up to 1.4-1.5Å, up to 2000 atoms in the asymmetric unit)

c) a new molecular replacement routine has been incorporated;

d) SAD-MAD, SIR-MIR, SIRAS-MIRAS cases can be faced. The new method provides quite simple and effective formulas both for locating heavy-atom/anomalous-scatterer substructures, and for phasing reflections ([3], [4]).

The program is highly automatic and suitable for high throughput crystallographic. Results of numerous applications will be shown.

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Keywords: structure determination, crystallografic software, protein crystallography

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***OASIS-2004* and Difficult SAD Phasing**

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OASIS [1] is a direct-method program for resolving the phase ambiguity in single-wavelength anomalous diffraction (SAD) and in single isomorphous replacement (SIR) of proteins. The new version, OASIS-2004 includes algorithms for automatically tuning the scaling factor associated to the lack-of-closure error and for dynamically incorporating known structure fragment(s) in the iterative direct-method phasing. Details of the phasing strategy will be described. Application to SAD data from a series of known as well as originally unknown proteins will be given. The data sets were collected either with synchrotron radiation or with in-house sources (Cr-Kα and Cu-Kα) X-rays. Among the applications, an originally unknown protein with more than a thousand amino acids in the asymmetric unit has been solved with Cr-Kα sulfur-SAD data. Good quality phases have been successfully derived from sulfur-SAD data at the Bijvoet ratio $<|\Delta F|>/<F>$ lower than 0.6%. In all cases the combination of programs OASIS-2004, DM, RESOLVE-BUILD and ARP/wARP enabled automatic structure analysis from *ab initio* SAD phasing to model building. All resulted in a model containing more than 90% of the content of the asymmetric unit.

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Keywords: SAD phasing, direct methods, proteins

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A Novel Method to Prepare Iodine Derivatives for In-house Phasing

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We developed novel procedures for efficient preparation of iodine derivatives of protein crystals that are most effectively employed for in-house phase determination. In this procedure, target native crystals are exposed by gaseous iodine. In the crystals, hypoiodous acids are