02-Methods for Structure Determination and Analysis, Computing and Graphics

to propagate phase information from those reflections where it is strongly indicated to those where it is essentially absent. Maximum-entropy extrapolation will perform this task in an optimal fashion.

MS-02.02.04 DETERMINATION OF THE STRUCTURE OF A LARGE PROTEIN BY MAD PHASING: GLUTAMINE FRPP AMIDOTRANSFERASE, by J.L. Smith, E.J. Zaluske, and J.-F. Wery, Dept. of Biological Sciences, Purdue Univ., West Lafayette, IN 47907 USA, and Y. Shukow, Faculty of Pharmaceutical Science, University of Tokyo, Bunkyo-ku, Tokyo, Japan

Glutamine FRPP amidotransferase is a unusual FeS protein that catalyzes the first step of de novo purine biosynthesis. The enzyme is a tetramer of 50–90 kDa subunits; each subunit contains one FeS₄ cluster. MAD phasing was based on X-ray anomalous scattering by Fe. Multil wavelength data to 3.0Å spacings were measured on imaging plates at beamline 14A at the Photon Factory. The asymmetric unit of the crystals consists one tetrameric molecule. Positions of the Fe₄ clusters were initially determined using data to 5.0Å spacings, and individual Fe atoms were located by analysis of partial structure Fourier maps at 3.0Å resolution. Experimental phases to 3.0Å were derived from the multiple IFoal by a probability treatment similar to the Blow-Crick analysis of errors in isomorphous replacement (Pabke, Smith & Hendre16ou1990 Acta Cryst. A46, 758–758). Once the Fe partial structure was known, phase probability expressions were computed for all experimental observations. The resulting phase coefficients were combined for all observations of each unique reflection without regard to the crystal, asymmetric unit or time of the measurement. The final MAD phase set was used as a starting point for phase refinement by local symmetry averaging and solvent flattening. A procedure was developed for automatic mask generation and rapid, automatic phase refinement (J.T. Bom, S.W. Muchmore & J.L. Smith, unpublished), which allowed us to experiment with the phase refinement protocols until we found the most interpretable map. The final 3.0Å electron density was of excellent quality and was easily interpreted for the structure.

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PS-02.02.05 ANHARMONIC THERMAL VIBRATION OF RUTILE (TiO₂) DETERMINED FROM NUCLEAR DENSITY DISTRIBUTION OF MAXIMUM ENTROPY METHOD ANALYSIS, by S. Kumazawa, Department of Physics, Science University of Tokyo, Japan and M. Takata & M. Sekata, Department of Applied Physics, Nagoya University, Japan

Maximum Entropy Method (MEM) analysis enables us to have an electron density distribution which is consistent with the observed structure factors and least biased with the not-observed structure factors when it is applied to X-ray diffraction data. By applying it to neutron diffraction data, a nuclear density distribution can be obtained, which is considered to describe the thermal smearing of the nuclei. Such a thermal smearing must be influenced by all kinds of thermal vibrations regardless of harmonic and anharmonic vibrations. It is, therefore, possible to determine thermal parameters of the constituent atoms by the analysis of the nuclear density distributions obtained by the MEM analysis. In this work, the MEM nuclear density distribution of rutile (TiO₂) were analyzed by least square refinement and thermal parameters for an effective one particle potential (OPP) were determined. This is a second example of determining the thermal parameters from the nuclear density distribution. First example was done by Takata, Sakata, Lassen, Kubota & Inversi (1992) Inaugural Conference, AsCA'92 in the case of Be. In this case, Be atom is located at a special position and it was not necessary to determine the atomic position beforehand. In rutile case, x-coordinate of oxygen has to be determined before the thermal parameters are analyzed. In the present study, the atomic position was defined and determined as the position for which the first order moment of nuclear density becomes zero. The obtained x-coordinate was 0.30417, which shows excellent agreement with the previous results of Rietveld refinement by Howard, Sabine & Dickson (1991) Acta Cryst. B47, 453–486, i.e., 0.30478 (6). The higher moments which are related to thermal vibrations are also calculated, which is very helpful to consider what kind of OPP model to be employed for thermal parameters analysis. The least square refinement was successfully performed and thermal parameters are obtained as well as the harmonic ones. More specifically to say about anharmonicities of rutile, it was found that Ti atoms had the 4th order anharmonic term and that O atoms had not only 4th order but also 3rd order anharmonicities, which cause substantial smearing of O atoms in rutile. In Fig. 1, both (a) the MEM nuclear densities and (b) the calculated densities from the final thermal parameters determined by the present least square refinement are shown for (001) plane, which are beautifully agreed.

Fig. 1-(a) MEM
Fig.1-(a) NBM
Fig.1-(b) Calculated

PS-02.02.06 Determination of the Anharmonicity Constant of GaAs by Means of the Bijvoet Relation of the weak (066) Reflection, by U. Pietzsch and K. Tasche, Fachbereich Physik der Universität Potsdam, Am Neuen Palais 10, D-14476 Potsdam, Germany

Due to the influence of anomalous dispersion the weak (066) and (4-h, 4-h, 0) reflections of the zinc-blende structure differ from each other. At large scattering vector this difference, scaled by the Bijvoet relation