to propagate phase information from those reflexions 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 PRPP AMIDOTRANSFERASE. by J.L.Smith*, E.J.Zaluzec, and J.-P.Wery, Dept.of Biological Sciences, Purdue Univ., West Lafayette, IN 47907 USA, and Y.Satow, Faculty of Pharmaceutical Science, University of Tokyo, Bunkyo-ku, Tokyo, Japan

Glutamine PRPP amidotransferase is a unusual FeS protein that catalyzes the first step of *de novo* purine biosynthesis. The enzyme is a tetramer or 50-kDa subunits; each subunit contains one Fe_4S_4 cluster. MAD phasing was based on K-edge anomalous scattering by Fe. Multiwavelength data to 3.0A spacings were measured on imaging plates at beamline 14A at the Photon Factory. The asymmeric unit of the crystals contains one tetrameric molecule. Positions of the Fe_4 clusters were initially determined using data to 5.5A spacings, and individual Fe atoms were located by analysis of partial-structure Fourier maps at 3.0A resolution. Experimental phases to 3.0A were derived from the multiple |Fobs| by a probability treatment similar to the Blow-Crick analysis of erros in isomorphous replacement (Pahker, Smith & Hendrickson(1990) Acta Cryst. A46, 537-540). 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was developed for automatic mask generation and rapid, automatic phase refinement (J.T.Bolin, S.W.Muchmore & J.L.Smith, Unpublished), which allowed us to experiment with the phase refinement protocols until we found the most interpretable map.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 3.0A electron density was of excellent quality and was easily interpreted for the structure.

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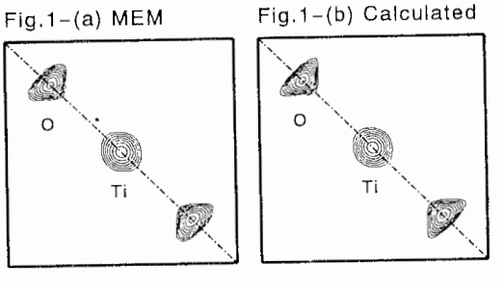
PS-02.02.05 ANHARMONIC THERMAL VIBRATION OF RUTILE (TiO_2) DETERMINED FROM NUCLEAR DENSITY DISTRIBUTION OF MAXIMUM ENTROPY METHOD ANALYSIS By S. Kumazawa*; Department of Physics, Science University of Tokyo, Japan and M. Takata & M. Sakata; Department of Applied Physics, Nagoya University, Japan

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 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 (TiO_2) were analysed 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, Larsen, Kubota & Iversen; (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 analysed. 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 0.30477, which shows excellent agreement with the previous result of Rietveld refinement by Howard, Sabime & Dickson; (1991) Acta Cryst. **B47**, 462-468, i.e. 0.30478 (6). The higher moments which are related to thermal behaviors 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 terms and that O atoms had not only 4th order but also 3rd order anharmonicities, which cause substantial skewness 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 refinemnet are shown for (002) plane, which are beautifully agreed.

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



PS-02.02.06 Determination of the Anharmonicity Constant of GaAs by Means of the Bijvoet- Relation of the weak (666) Reflection

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

