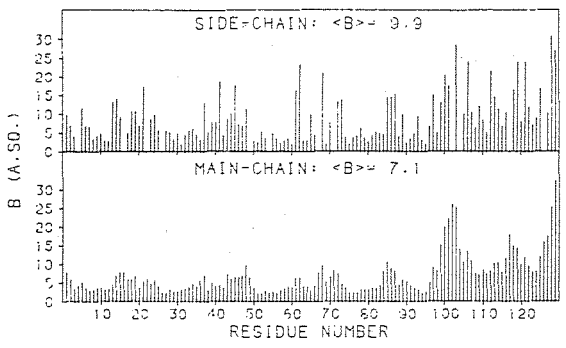
01.5-1 SRLSQ REFINEMENT OF TRICLINIC LYSOZYME. by M. Ramanadham, Neutron Physics Division, Bhabha Atomic Research Centre, Bombay 400085, India, and, L.C. Sieker and L.H. Jensen, Department of Biological Structure, University of Washington, Seattle, WA 98195, USA.

The x-ray structure refinement of triclinic hen egg-white lysozyme by the method of stereochemically restrained least-squares (SRLSQ) at 2Å resolution has recently been completed. The 1270-atom structure (1001 protein non-hydrogen atoms, 5 NO_3^- groups and 249 'solvent' oxygen atoms) was refined to an R-value of 0.124 using 7075 independent x-ray structure amplitudes with $10 > d \geq 1.97$Å. The refinement had also resulted in an excellent agreement between the model and the ideal geometries. The ΔF map based on the final model had no gross unexplained features. During the first phase, no model corrections were made outside the refinement process. Though the R-value came down to 0.169, gross orientational errors still remained in the model in the residue range 100-105, and in the side chains of some of the ARG, ASP, ASN, GLU and GLN residues. During the final phase, model corrections were made after every few cycles of refinement with the help of a Vector General interactive graphics system, the FRODO program and the Fourier maps computed using the PROTEIN package. The relative weights of the observations and the restraints were adjusted from time to time with the help of various $\sum \omega \Delta^2$ terms. Only mild restraints were applied on B-values. The B-value distributions, shown in the Figure below, are generally in good agreement with the environments of various residues and the secondary structural features of the protein molecule. The removal of the planar restraints from the peptide linkages during the final stages of refinement did not have any adverse effect on the model. In fact, it revealed an error in the residue range 117-118, which was subsequently corrected. This, and another error at 102-103 region could be corrected only when the R-value came down to about 0.14. Some portions of the protein had indications of the existence of static disorder. This and other aspects will be analysed during the refinement at 1.5Å resolution, which is being taken up presently.



MODEL PLAM46: B-VALUE DISTRIBUTIONS: <B>= 8.5

01.6-1 THE EVALUATION OF SOLVENT AND SALT DENSITY IN PROTEIN CRYSTALLOGRAPHY: A NEUTRON STUDY. By B.P. Schoenborn and X. Cheng, Department of Biology, Center for Structual Biology, Brookhaven National Laboratory, Upton, NY 11973.

In protein crystallography, it has been customary to omit the low order data in refinement procedures. This data contains, however, important data about the gross features of the unit cell content and particularly the scattering density of the solvent, i.e. solvent structure. In order to use the low order Bragg reflections, a solvent evaluation procedure has been developed that allows the description of the low order structure factors as a combination of solvent and protein terms. This allow the use of all observed SF in a least squares refinement. Coupled with the measurement of the crystal density by a density gradient technique, the evaluation of the solvent scattering makes it possible to determine the amount of salt present in the solvent space.

01.6-2 NEUTRON STRUCTURE ANALYSIS OF PLASTOCYANIN. By W.B. Church, T.P.J. Garrett, H.C. Freeman, Department of Inorganic Chemistry, University of Sydney, Sydney 2006, Australia, and B.P. Schoenborn, Biology Department, Brookhaven National Laboratory, Upton, New York 11973, USA.

Plastocyanin is a 10,500d protein containing one 'type I' copper atom, which is essential for the protein's biological role as an electron carrier. The structure of Cu(II)-plastocyanin from poplar leaves has previously been solved and refined using X-ray data at a resolution of 1.6Å. The intramolecular hydrogen bonds which can be inferred from the distances between pairs of non-hydrogen atoms include several that stabilize the protein configuration near the Cu site. Of particular interest is a N-H...S bond to a Cu-binding cysteine side-chain, which appears to be analogous to such bonds at the active sites of a number of other metalloproteins. The possible importance of hydrogen bonding in relation to the electron transfer function of plastocyanin has generated an interest in the experimental determination of the hydrogen atom positions. Neutron diffraction data for deuterated poplar plastocyanin have been measured at the Brookhaven National Laboratory High-Flux Beam Reactor. The intensities of 64% of the reflections to 1.7Å are significant [$I \geq 2\sigma(I)$]. Even the initial nuclear scattering density difference maps provided evidence for well-ordered hydrogen atoms. In this paper, we report progress in the neutron structure analysis of the deuterated protein and in a parallel refinement of the structure using new X-ray counter data to 1.6Å resolution.