ESRF	**Experiment title:** Physical Estimation of Triplet Phases for Discriminating Between Similar Structure Models	**Experiment number:** HS-37
Beamline: SNBL BMOI	**Date of Experiment:** from: 23.10.96 07:00 to: 28.10.96 07:00	**Date of Report:** 14.08.97
Shifts: 15	**Local contact(s):** K. Knudsen/ E. Weckert	*Received at ESRF :*

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Report:

Background

Physically estimated triplet phases (PETP) from three-beam interference experiments can be used to discriminate between several possible and closely related models of a structure. This novel application of PETP's has been explored in a study of the complex a-D-glucose NaCl H,O (2:1:1) in space group $P3_1$, with V = 4.180 Å^3. A structure of the complex has been published (Model CA) [1]. However, there exists one alternate solution (Model GE) [2]. We have refined both models against their original data sets, and identified the differences in structure. The primary difference is an interchange of the Cl and water 0 positions, which is accompanied by small, mainly translatory displacements of the glucose rings, and a reorientation of the water molecules and one of the glucose OH groups, leading to a reversal of the polarity in chains of H-bonds along the polar axis. The changes in structure imply significant changes in phase for about 50% of the structure factors. In contrast, triplet phases being sensitive to the model are extremely scarce, thus, among 2 x 10^6 calculated triplet-phase invariants, only about 1100 with amplitudes suitable for phase measurements had $|\Delta\Phi_3| > 30^o$. In the great majority of cases where model-sensitive single phases are involved, they occur in pairs, each with an intermodel $|\Delta\phi| \sim 180^o$, thus rendering $\Delta\Phi_3 \sim 0$. The triplets with the best model discriminatory properties were combinations of one sensitive and two insensitive single phases. By measuring several model-sensitive triplet-phase invariants and comparing with those calculated for the relined models CA and GE we expected initially to be able to identify one correct model of the pair.

Work in October, 1996: ESRF Experiment HS-3 7, and (in part) SNBL Experiment 01-02-46

Three-beam intensity profiles were collected for four different crystals of the complex. As this concludes the investigation, we give here a summary of all experiments and some results. Thirteen crystals, all cut from one large single crystal of a-D-glucose NaCl H_2O (2:1:1), have been subjected to physical phase estimation which included studies of model-sensitive triplet phases. Each phase assignment was based on the intensity profiles mapped out in Ψ-scans of the pair of triplets -H, L, H-L and H, -L, -H+L, corresponding to phases $+\Phi_3$ and $-\Phi_3$, respectively. Two distinct right-handed cell matrices are possible in this space group, the (incorrect) alternative being (b, a, -c). It was ascertained that the choice of unit cell of the crystals was internally consistent and in agreement with the indexing of the native data sets CA and GE. Each intensity profile was scanned from 5 to 50 times depending on the particular experimental conditions. From 8 to 43 triplets were examined for each crystal specimen. The mapping of a total 309 pairs of three-beam interference profiles included 89 different triplets.

Results

The estimated triplet phases have been compared with the parent values calculated for the refined structure models CA and GE. A statistical analysis of the results provides highly significant evidence that two structures are present, but do not coexist in the same crystal. Six crystals confirm model CA, seven crystal fit the GE model. The estimated random error in the phase assignments is 19.7'; for all crystals the model with the largest mean variance can be rejected at a significance level $p << 0.001$. Intensity profiles for two triplets, one model insensitive with $|\Delta\Phi_3| \sim 20^\circ$ (a), and one model sensitive with, $|\Delta\Phi_3| \sim 100^\circ$ (*b*), are shown in Fig. 1 for both structure models. Note the reversed asymmetry in the profiles of Fig. *1(b)*.

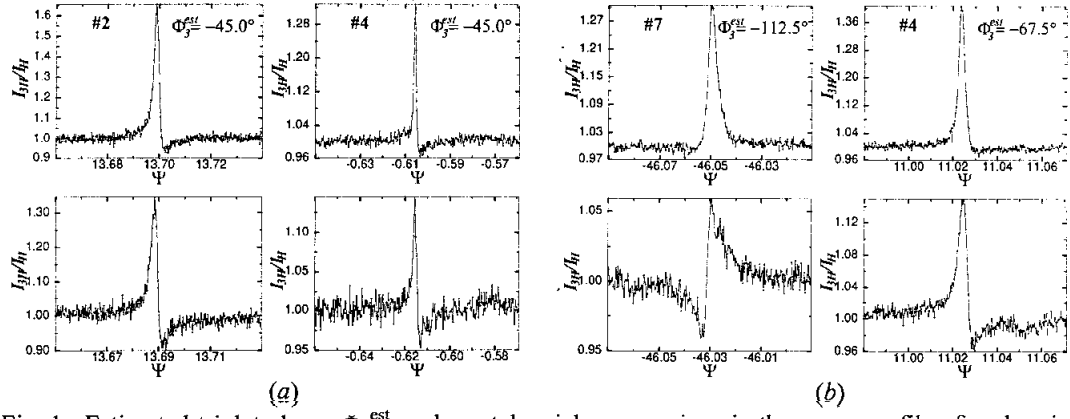

Fig. 1. Estimated triplet phase, Φ_3^{est}, and crystal serial no. are given in the upper profile of each pair. (*a*) Triplet 3 0 -4/ -2 4 -1/ -1 -4 5, $\Phi_3^{CA} = -48^\circ$ (Cryst. #2), $\Phi_3^{GE} = -65^\circ$ (Cryst. #4). (*b*) Triplet -2 -2 5/ -6 3 5/ 8 -1 -10, $\Phi_3^{CA} = -144^\circ$ (Cryst. #7), $\Phi_3^{GE} = -47^\circ$ (Cryst. #4).

The 'P-scans were carried out on a six-circle Huber diffractometer (Prof. Hummer, University of Karlsruhe) located on the Swiss-Norwegian Beamline, ESRF. A full account of this work will be published [3].

[1] Ferguson, G., Kaitner, B., Connett, B.E. & Rendle, F. (1991). *Acta Cryst.* B47,479-484.

[2] Fröhlich, R. (1989). Personal communication.

[3] Mathiesen, R.H., Mo, F., Eikenes, A., Nyborg, T. & Larsen, H.B. (1997). Submitted to *Acta Cryst.* Ser. A.