



	<b>Experiment title:</b> The Structure Elucidation of the Membrane Intrinsic Protein Complex Photosystem I by X-Ray Crystallographic Methods	<b>Experiment number:</b> LS-653
<b>Beamline:</b> ID2-BL4	<b>Date of experiment:</b> from: 30/01/97                      to:            03/02/97	<b>Date of report:</b> <b>26/08/97</b>
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**Report:**

Crystal structure analysis of photosystem I (PSI) from *Synechococcum elongatus* at 4 Å resolution revealed many important structural features of this complex (**Krauß** et al., 1996; Schubert et al., 1997) as iron sulfur clusters, **α-helices** including interhelical links and chlorophyll *a* cofactors of the electron transfer system and the core antenna of PSI. The resulting model indicated a surprising similarity between the reaction centre core of PSI and purple bacterial reaction centres.

Although individual PSI crystals show X-ray diffraction to 3 Å resolution, an electron map

at the corresponding resolution is not available at present. The limitations in the attainable maximum resolution of the structure analysis are caused by the relatively large mosaicity ( $> 0.6''$ ) of the crystals coupled to large unit cell constants (space group  $P6_3$ ,  $a = b = 286 \text{ \AA}$ ,  $c = 167 \text{ \AA}$  at 277 IS) and by the short lifetime of the crystals in the intense X-ray beam at synchrotron sources, especially at the High Brilliance Beam Line ID2 at ESRF. A significant progress was achieved during our last beam time at ID2, when we could collect the first data sets under cryogenic conditions. The lifetime of the PSI crystals in the beam increased remarkably under these conditions: native crystals could be irradiated about six hours, heavy atom derivative crystals two to three hours at one position until significant loss of diffraction quality was observed. Because of the length of the crystals (about 1 mm) and the small slit size used for the primary beam (ranging from  $150 \times 150 \text{ \mu m}^2$  to  $100 \times 100 \text{ \mu m}^2$ ) data could be collected from different positions of the crystals.

Data were collected by the conventional rotation method using a monochromatic X-ray beam ( $\lambda = 0.99 \text{ \AA}$ ) and a Marresearch image plate detector (diameter 300 mm). The rotational increment per image was  $0.4^\circ$ . Surprisingly, the crystal symmetry changed upon freezing from the hexagonal space group  $P6_3$  to monoclinic  $P2_1$ , with  $a = 277 \text{ \AA}$ ,  $b = 165 \text{ \AA}$ ,  $c = 283 \text{ \AA}$  and  $\beta = 119^\circ$ . Partial data sets were collected from two native crystals, two mercury and two gold derivative crystals, which are suitable up to  $3.2 \text{ \AA}$  resolution. For the merged data sets, which are between 65 % and 80 % complete even in the highest resolution shells, the mean multiplicity ranges from 2.2 to 2.7, less than 8 % of the measured reflections have  $I/\sigma(I) < 3.0$  and the  $R_{\text{sym}}$  values are below 20 % (highest resolution shells).

## References

- Krauß**, N., Schubert, W.-D., Klukas, O., Fromme, P., Witt, H.T. & Saenger, W. (1996) Photosystem I at 4 A Resolution Represents the First Structural Model of Joint Photosynthetic Reaction Centre and a Core Antenna System. *Nut. Struct. Biol.* **3**,965-973.
- Schubert, W.-D., Klukas, O., **Krauß**, N., Saenger, W., Fromme, P. & Witt, H.T. (1997) Photosystem I: of *Synechococcus elongatus* at 4 A Resolution: Comprehensive Structure Analysis. *J. Mol. Biol.*, in press.