EUROPEAN **S**YNCHROTRON **R**ADIATION **F**ACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** Tocopherol transfer protein	**Experiment number**: 78
Beamline: BM14U	**Date of experiment**: from: 02052002 to:03052002	**Date of report**:
Shifts: 3	**Local contact(s)**: Dr. Hassan Belrhali (e-mail: belrhali@embl-grenoble.fr)	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

PD Dr. Achim Stocker

Uni Bern

Report:

S-SAD EXPERIMENT

At a minimum if you can provide the following statistics it would be really useful for us:
YELLOW fields not essential but useful, RED fields need your feedback

Experiment date	02 May 2002-03May 2002
Proposal code (BM14U### or MX###)	BM14U 14-U-45
Unit cell	a=60.34 Å, b=84.33 Å, c=87.06 Å, α =116.20, β=102.52, γ=100.07 degrees
Space group	P1
No. a.a. residues/monomer	397
No. Monomers/AU	3
No. sulphurs/monomer	30
Solvent content	
Disulphide bonds	none
Resolution data collected	1.9
Wavelength used for data collection	0.97950
Data collection strategy (crystal offset, use of kappa etc)	Oscillation ranges for each image ranged between 0.5 and 1.0 degrees with typical exposure times of 1 second per image. Crystal-to-detector distances varied from 130 mm to 250 mm.
Redundancy	4.7
Other anomalous scatterers present (known or unknown at experiment start	none

e.g. maybe you had other metals or ions such as Cd, Cl, Ca etc –or well bound sulphate ions)	
Software used for location of sulphurs	Twenty out of thirty expected selenium positions were determined using Shake-and-Bake version 2.1. These selenium positions were refined and phases were computed using SOLVE 2.01, resulting in a figure-of-merit of 0.34 at 2.25 Å resolution.
Software used for Refinement of sites/phasing/solvent flattening	A SAD data set was collected at the beamline X06SA at the Swiss Light Source (SLS) in Villigen. This data set was used to solve and refine the structure initially. Later, slightly higher resolution data were collected at the ID14-14 beamline at the ESRF Grenoble. Redundant data sets were collected at the peak wavelength which was determined experimentally by fluorescence scans. Data were integrated and scaled with MOSFLM/SCALA [37, 38] or XDS [39, 40]. Data collection statistics are given in Table 1. Solvent modification was effected with RESOLVE 2.01 [43] and increased the figure-of-merit to 0.58 at 2.05 Å resolution using the SLS data. The resulting electron density map was easily interpretable. Further density modification was carried out using ArpWarp [44] and concomitantly a partial model including sidechains was built automatically. The resulting model was completed using O [45]. Refinement was carried out with CNS [46] against the anomalous peak data. Care was taken to partition both Friedel mates either in the test or working set. Anomalous scattering factors for selenium were refined using CNS after the model had reached an R_{free} of 0.28. In the final stages of refinement NCS restrains were removed. This procedure was judged to be valid by the drop in both R and R_{free} by about 1%.
Experiment success /failure	Successful SAD experiment
Citation for work if published	Achim Stocker, Takashi Tomizaki, Clemens Schulze-Briese and Ulrich Baumann (2002): Crystal Structure of the Human Supernatant Protein Factor, *Structure*, 10, 1533–1540.

We are of course very happy if you have the time to provide full data processing/phasing and refinement statistics and any comments /thoughts on the experiment carried out.