EUROPEAN SYNCHROTRON RADIATION FACILITY

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 original report should be sent, together with 5 reduced (A4) copies, to the User Office.

In addition, please send a copy of your file as an e-mail attachment to reports@esrf.fr**,** using the number of your experiment to name your file. This will enable us to process your report for the ESRF Annual Report.

Reports accompanying requests for additional beam time

If your report is to support **a new proposal**, the orginal report form should be sent with the new proposal form, and a copy of your report should be attached to each copy of your proposal. 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.

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- bear in mind that the report will be reduced to 71% of its original size. A type-face such as "Times", 14 points, with a 1.5 line spacing between lines for the text, produces a report which can be read easily.

ESRF	**Experiment title:** St. Andrews Dundee BAG	**Experiment number**: LS-1951
Beamline: ID29	**Date of experiment**: from: 24th MARCH 2001 to:25th MARCH 2001	**Date of report**: 21st August 2001
Shifts: 3	**Local contact(s)**: Dr. Gordon Leonard	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

William N. Hunter*, Charles S. Bond*, Lauris E. Kemp*, Mads Gabrielsen*

Report:

Isoprenoid biosynthesis

Three-wavelength MAD experiments were carried out on two crystal forms of a selenomethionine derivative of 2C-methylerythritol synthase (MECPS). A XANES scan was recorded and used to identify the wavelengths to be used. Form I ($P2_12_12$, 54 x 114 x 88Å) diffracts to 2.1Å, form II ($I2_13$, a=144Å) only to 3.0Å. Form II suffered severe radiation damage during the experiment so really only the f" data is acceptable (Rsym 7.8%, 100% complete and redundancy of 8). The lesson we have learned is that we need to attenuate more on this beamline. The form I crystal gave three wavelengths with greater then 96% completeness, and Rsym's between 5 and 5.4% with redundancy of greater than 2. The use of direct methods (SnB, SHELXD) has identified plausible Se positions in form I which unfortunately do not produce an interpretable electron density map. There appears to be

some useful phase information but we need to obtain further derivative data. The low redundancy of the orthorhombic data may be contributing to the poor phasing observed.

Our previous MAD experiment on a selenomethionine derivative of 4-diphosphocytidyl-2C-methyl-D-erythritol synthetase (DMES) failed to identify the Se positions. (see previous report on BM14). We prepared a potential platinum derivative of this protein then carried out a XANES scan which showed the presence of the metal and selected wavelengths for data collection. We screened several crystals searching for an appropriate sample though in all cases the diffraction quality was poor. Resolution tests of a new crystal form (small needles) of DMES showed that it was inferior to the tetragonal blocks first obtained and only diffracted to between 4 and 5Å.

Studies of DNA/metal complexes with Hjc, a DNA resolvase

A number of candidate crystals of an Hjc-Holliday Junction complex have been tested. One crystal form gave 7Å diffraction which could be indexed as I cubic, a = 143 Å. This corresponds to crystals which provided 3.5 Å data collected previously on native Hjc and, while these cocrystals stain positive for DNA (ethidium bromide), it is as yet uncertain whether they represent the true complex. Further studies must be undertaken. Data collection on a candidate Hjc-Mg^{2+} complex yielded low quality data (due to poor crystal quality) but which nevertheless indicated the absence of Mg^{2+} once the refinement was completed.

Ser/Thr Protein kinase

Three large (0.3mm) beautiful (absolutely gorgeous) cubic blocks of the stress-activated protein kinase (MAPKAPK2) each grown under different conditions were tested and only diffract to 6Å. This is equivalent to the diffraction limit observed in-house therefore no datasets were recorded.