ESRF
experimental report

Experiment title:

pl3 Suc-l

Experimment number:

LS49C

Beamline(s) used:
BLA/ID2

Date and time of experiment
***from:* 22/8/94**
to: 24/8/94

Local contact(s):
B. Rasmussen

Date of report:
18/8/94

received

Name and affiliation of applicants (please mark experimentalists whith an asterisk)

Prof. LN. Johnson
Dr. Martin Noble*
Dr. Jane Endicott*

Experiment report ***(If this work has been published, please give reference and abstract):***

Wavelength: 0.906 Å
Slit Size: 100 µm
Xtal-film distance: 420 mm
Main beam coordinates X= 149.8 mm Y=149.3 mrn (Denzo refined parameters)
Solution at edge of detector 2.7 Å
Oscilation range 2°
Exposure time: Between 5 and 20 seconds
Cell parameters: 60.78 60.78 265.8 90 90 120
space group: $P6_522$
Typical crystal size 30 µm x 30 µm x 200 µm
Total number of frames collected 124
Ion chamber current range: 25-40

Data sets produced

1) p13 sucl Wild type
Max resolution: 27 Å
Numer of crisstal: 6
Completeness to max resolution: 87.5%
Overall Merging R-factor (intensities): 10.4%
Completeness in final resolution shell (2.82-2.70 Å): 90,2%
Merging **R-factor in** highest resolution shell: 47,1 %
Number of frames used in dataset: 15

2 13 SUC1 seleno methionine
Max resolutiom 28 Å
N,B. No diffration from these crystals could be observed using an in-house rotating anode source.
Number of crystals: **8**
Completeness to max resolution: 75.8%
Overall **Merging** R-factor (intensities): 12,6%
Completeness in final resolution shell (2.82-2.70 Å) 81.2 %
Merging R-facror in highest resolution shell: 43,0%
Number of frames used in dataset 12

MFID between wild type and seleno, methionine dataset: 0.142 (on F)

experimental report: Experiment number LS-89C

Comments:

Support and help on the **beam** line were exellent Crystals suffered quickly from radiation damage: typicalIy the third exposures of any give spot on the crystal could not be included in the final datasets. For the smaller seleno-methionime crystals, even the second frames scaled poorly together with the other data. Sealing together of wild type **and seleno-methionine data** showed some strange behaviour at low resolution. This may be **influenced by** 1) large **beam stop** shadow, and **2)** possible non-linear response of detector at high intensity

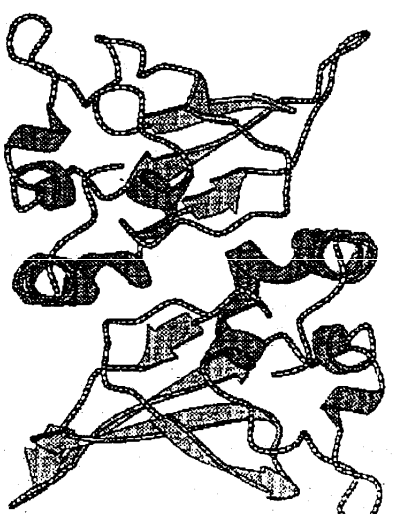
Suggestions:

1) Visiting scicntists should be strongly encouraged to investigate cryo-conditions for their protein to best rake advantage of **the high brilliance beam** line

2] A range **of media should be made available** for the backing Up of data. We had to hunt hard to **find a suitable DAT tape dive to retrieve our frames.**

Result:

The seleno methionine positions were discovered automatically by the direct methods options of SHELX using FPH-FP coefficients from the data collected in Grenoble. Phases from this derivative could then be used to cross phase difference fourier from heavy atom soaked datasets collected elsewhere and correctly identify sites in these other datasets. MIR phases guided principality by the seleno-methionine derivative have now been used to succefulIy solve the structure of p 13 suc 1, which is currently under refinement whith an R-factor of 20% to 2.7 A resolution. This excellent result could not have been achicved without the intense. reliable beam of the ESRF.



Cartoon of p13 suc-1: