



Experiment title: INIREG	Experiment number: LS 1817	
Beamline: ID14-1 ID14-4 ID14-1	Date of experiment: from: 4 Oct. 2000 to: 5 Oct. 2000 from: 29 Jan. 2001 to: 29 Jan. 2001 from: 31 Jan. 2001 to: 1 Feb. 2001	Date of report: 26.02.2001
Shifts: 3 2 3	Local contact(s): Stéphanie Monaco Raimond Ravelli Steffie Arzt	<i>Received at ESRF:</i>
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Report:

Toulouse group:

1- FixJN soaked with beryll fluoride (ID14-1: 3×3 sec/2.5°)

Cell parameters: 32.0 Å, 42.4 Å, 44.6 Å, 93.7°, 103.0°, 102.0° (P1)

Completeness: 97.1%

Multiplicity: 2.0

Resolution limit: 1.9 Å

Rsym = 4.1%

2- PhoPN pH 7 + Mn²⁺ (ID14-1: 3×3 sec/0.5°)

Cell parameters: 45.7 Å, 45.7 Å, 134.8 Å (P4₁2₁2)

Completeness: 92.0 %

Multiplicity: 6.1

Resolution limit: 1.6 Å

Rsym = 9.1%

3- PhoPN pH 7 + Mg²⁺ (ID14-1: 3×3 sec/0.5°)

Cell parameters: 46.0 Å, 46.0 Å, 135.7 Å (P4₁2₁2)

Resolution limit: 1.9 Å

Problems with data processing, unexpected high mosaicity value

4- PhoPN pH 7 soaked with phosphoramidate n°1 (ID14-4: 1×1 sec/0.5°)

Cell parameters: 45.6 Å, 45.6 Å, 134.2 Å (P4₁2₁2)

Completeness: 99.5 %

Multiplicity: 3.7

Resolution limit: 1.5 Å

Rsym = 7.6%

5- PhoPN pH 7 soaked with phosphoramidate n°2 (ID14-4: 1×1 sec/0.5°)

Cell parameters: 45.6 Å, 45.6 Å, 134.2 Å (P4₁2₁2)

Completeness: 99.5 %

Multiplicity: 6.3

Resolution limit: 1.5 Å

Rsym = 9.0%

6- TorD native (ID14-4: 1×10 sec/0.5°)

Cell parameters: 66.0 Å, 93.4 Å, 95.1 Å (P2₁2₁2₁)

Completeness: 98.7 %

Multiplicity: 3.7

Resolution limit: 2.4 Å

Rsym = 3.3%

7- OXA 10 class D β-lactamase. Native pH 6.0 (ID14-1: 3×6 sec/1°)

Cell parameters: 66.8 Å, 82.5 Å, 101.8 Å, 90°, 95.5°, 90° (P2₁)

Completeness: 97.8 %

Multiplicity: 2.5

Resolution limit: 1.9 Å

Rsym = 8.5%

8- OXA 10 class D β-lactamase. Native pH 6.5 (ID14-1: 3×1.5 sec/1°)

Cell parameters: 66.7 Å, 82.5 Å, 101.7 Å, 90°, 95.5°, 90° (P2₁)

Completeness: 98.3 %

Multiplicity: 2.6

Resolution limit: 1.7 Å

Rsym = 6.8%

9- OXA 10 class D β-lactamase. Complex with 6α-hydroxypropyl penicillanate n°1 (ID14-4: 1×3 sec/0.75°)

Cell parameters: 66.6 Å, 82.5 Å, 101.7 Å, 90°, 95.4°, 90° (P2₁)

Completeness: 98.3 %

Multiplicity: 2.5

Resolution limit: 1.7 Å

Rsym = 7.0%

10- OXA 10 class D β-lactamase. Complex with 6α-hydroxyoctyl penicillanate n°1 (ID14-4: 1×1 sec/0.75°)

Cell parameters: 66.6 Å, 82.8 Å, 101.4 Å, 90°, 95.5°, 90° (P₂)

Completeness: 98.8 %

Multiplicity: 3.0

Resolution limit: 1.7 Å

R_{sym} = 10.1%

11- OXA 10 class D β-lactamase. Complex with 6β-hydroxyethyl penicillanate n°1 (ID14-1: high resolution data set 3×2 sec/1°; low resolution data set 3×0.6 sec/1.5°)

Cell parameters: 66.4 Å, 82.4 Å, 101.4 Å, 90°, 95.3°, 90° (P₂)

Completeness: 96.2 %

Multiplicity: 2.3

Resolution limit: 1.5 Å

R_{sym} = 12.0%

12- OXA 10 class D β-lactamase. Complex with WL n°1 (ID14-1: 3×4 sec/1°)

Cell parameters: 66.4 Å, 82.6 Å, 101.8 Å, 90°, 95.0°, 90° (P₂)

Completeness: 97.3 %

Multiplicity: 2.5

Resolution limit: 1.8 Å

R_{sym} = 4.9%

13- OXA 10 class D β-lactamase. Complex with 6α-hydroxyoctyl penicillanate n°2 (ID14-1: 3×4 sec/1°)

Cell parameters: 66.3 Å, 82.2 Å, 101.3 Å, 90°, 95.5°, 90° (P₂)

Completeness: 92.1 %

Multiplicity: 2.5

Resolution limit: 1.4 Å

R_{sym} = 5.8%

14- Several small crystals or crystals that diffract only very weakly or not at all on a laboratory X-ray source were tested: single mutant of FixJN, complex formed between CheY2 and P2, UMP Kinase, complex between CheY and FliM. These tests were of importance to orient further crystallization experiments.

Palaiseau group:

15- Native P12K (ID14-1: 2x3 sec/0.5° for high resolution + 1 pass for low resolution 3x0.5 sec/0.5°)

Cell parameters: 66.3 Å, 82.2 Å, 101.3 Å, 90°, 95.5°, 90° (P₂)

Completeness: 92.1 %

Multiplicity: 2.5

Resolution limit: 1.4 Å

R_{sym} = 5.8%

16- eIF5B- trigonal form (ID14-1: 3x1 sec/0.5°)

Cell parameters: 134.95 Å, 134.95 Å, 116.47 Å (P₃)

Completeness: 95.2 %

Multiplicity: 2
Resolution limit: 2.7 Å
Rsym = 4.8% (36.1%)

17- eIF5B- TMLA (ID14-1: 3x5 sec/0.5°)
Cell parameters: 135.4 Å, 135.4 Å, 115.9 Å (P3)
Completeness: 61 %
Multiplicity: 1.3
Resolution limit: 3.9 Å
Rsym = 6.6% (32.8%)

18- eIF5B- KAuCl4 (ID14-1: 3x3 sec/0.5°)
Cell parameters: 135.25 Å, 135.25 Å, 115.7 Å (P3)
Completeness: 83.4 %
Multiplicity: 1.7
Resolution limit: 3.5 Å
Rsym = 4.3% (22%)

Further analysis of the data showed that the trigonal crystals of eIF5B are twinned along both a,b* (near 50%) and a,b (about 12 %). The data could therefore not be detwinned and up to now, we were unable to solve the structure.*

20-IF2 from yeast mitochondria with tRNA (ID14-1: 3x3 sec/1°)
Cell parameters: 32 Å, 236.5 Å, 45.45 Å, 90°, 110.7°, 90° (P2₁)
Completeness: %
Multiplicity:
Resolution limit: Å
Rsym =

21-D-tyr-tRNA deacylase (ID14-4: three passes: 2sec/0.7°; 1sec/0.7°, 1sec/0.7° with 80% beam attenuation)
Cell parameters: 119 Å, 83.4 Å, 83.9 Å, 90°, 128.45°, 90° (C2)
Resolution limit: 1.5 Å
Rsyms were in the range 7-10 % whatever the resolution. We have not yet understood the origin of this problem.

22-D-tyr PCMBS (ID14-4: 1 sec/1°)
Cell parameters: 119.3 Å, 82.7 Å, 83.5 Å, 90°, 128.25°, 90° (C2)
Completeness: 100 %
Multiplicity: 4.8
Resolution limit: 2 Å
Rsym: 13.7 %; same problem as above, ex: 22 % at 2 Å, 14 % at 6 Å

23-D-Tyr PtCl4 (ID14-4: 1 sec/1°)
Cell parameters: 119.3 Å, 82.7 Å, 83.5 Å, 90°, 128.25°, 90° (C2)

Completeness: 100 %

Multiplicity: 4.8

Resolution limit: 2 Å

Rsym: 13.7 %; same problem as above, ex: 22 % at 2 Å, 14 % at 6 Å

24-D-Tyr SmSO4 (ID14-4: 1 sec/1°)

Cell parameters: 118.4 Å, 83.2 Å, 83.6 Å, 90°, 128.3°, 90° (C2)

Completeness: 98.8 %

Multiplicity: 2.1

Resolution limit: 2 Å

Rsym: 9.8 %; same problem as above.

25-IF2 from yeast mitochondria with tRNA (ID14-4: 1 sec/0.5°)

230 frames collected to 4 Å resolution. We were unable to scale the data together.

26-eIF5B - monoclinic form (ID14-4: 1 sec/0.3°)

Cell parameters: 154 Å, 367 Å, 112 Å, 90°, 97.1°, 90° (C2)

400 frames collected to 3.5 Å resolution. No satisfying processing could be obtained.