

Report of Experiment MX608 at ID14-1 on 27th February 2008

The beamline worked properly during most of the shifts assigned to our group. We used the sample changer, without major problems, though sometimes an error about “not properly vial fitting” appeared. It was probably due to presence of ice in the sample changer because we used only Hampton research *HP* cap and vials.

The assistance of our local contact, Dr. Juan Sanchez-Weatherby, who was precious and present, was very helpful.

We used DNA both for testing and data collection, but in a few cases we collected the data manually and processed the frames using *mosflm*, since DNA failed in finding the cell.

The beam intensity was fine during the whole beamtime as well as the detector and all the software of the network.

We tested 60 crystals and performed 7 data collections of crystals belonging to 4 different projects.

1) Project on a human Glutathione Transferase from Pi class in complex with antitumoral compound NBDHEX. We collected three data sets with the following statistics:

<i>Co-crystallization - GTPi – ID14-1</i>						
Space group	C2					
Unit cell dimensions	77.423	89.349	68.914	90.000	97.901	90.000
	<i>Overall</i>	<i>InnerShell</i>				
Low resolution limit	30.0	-				
High resolution limit	1.9	1.97				
R _{merge}	0.050	0.159				
Multiplicity	4.7	-				
Mean((I)/sd(I))	20.49	11.01				
Completeness	99.8	98.8				
Chi square	1.086	1.483				
<i>Soak 1 - GTPi – ID14-1</i>						
Space group	C2					
Unit cell dimensions	78.361	89.446	69.134	90.000	98.245	90.000
	<i>Overall</i>	<i>InnerShell</i>	<i>OuterShell</i>			
Low resolution limit	30.0	48.51	2.95			
High resolution limit	1.5	1.55	1.50			
R _{merge}	0.092	0.123	-			
Multiplicity	4.3	-	-			
Mean((I)/sd(I))	21.7	13.43	-			
Completeness	98.1	82.4	-			
Chi square	0.99	1.653	-			
<i>Soak 2 - GTPi – ID14-1</i>						
Space group	C2					
Unit cell dimensions	78.587	89.347	69.264	90.000	98.355	90.000
	<i>Overall</i>	<i>InnerShell</i>	<i>OuterShell</i>			
Low resolution limit	30.0	48.51	2.95			
High resolution limit	1.55	1.61	1.55			
R _{merge}	0.053	0.26	-			
Multiplicity	2.6	-	-			
Mean((I)/sd(I))	9.49	5.06	-			
Completeness	98.1	82.4	-			
Chi square	1.39	1.642	-			

2) Project on Serine hydroxymethyl transferase from *Methanococcus jannaschii*, a divergent SHMT seemingly binding RNA.

We collected data on two crystals, one native and the other obtained co-crystallizing with glycine.

The structures, using the high diffraction dataset, have been solved by MR using the program phaser. The structures are currently under manual building, but improvement of crystal quality is a work in progress.

native dataset:		co-crystallization dataset:	
space group	P212121	space group	P212121
cell	86.593 110.154 110.714	cell	86.593 110.154 110.714
resolution	30.0-3.4 Å	resolution	30.0-2.9 Å
completeness	98%	completeness	98%
I/sigma	9.49	I/sigma	10.72
Redundancy	3.2	Redundancy	2.8
Rmerge	0.083	Rmerge	0.067

3) *Leishmania major* Trypanothione reductase, a key enzyme in the trypanothione-based redox metabolism of pathogenic trypanosomes. We collected one data set of the native recombinant enzyme. We tested some heavy metal soaks but they diffracted at 7.0 Å. Both co-crystallization trial and other soaks with some inhibitors are in progress.

native - Tr1 - ID14-1		
	Overall	InnerShell
Space group	P4	
Unit cell dimensions	103.391 103.391 191.832 90 90 90	
Low resolution limit	30.0	48.51
High resolution limit	2.8	1.55
R _{merge}	0.096	0.626
Multiplicity	3.8	3.5
Mean((I)/sd(I))	10.2	8.9
Completeness	99.1	98.0

4) Adult worm *Schistosoma mansoni* Trx is a project from the structural genomics of this pernicious human parasite. We had already solved the structure of the isoform expressed by the larval stage, and now we focussed on the adult isoform. We collected 1 data set at 1.6Å from the only crystal coming from a large robotized screening. MR was performed with MolRep using the larval protein as a search model and model building and refinement are under progress.

Here are the final statistic with scala:

Summary data for Project: trx_wt,		
	Overall	OuterShell
space group	P212121	
Unit cell dimensions	a=53.03, b=53.57, c=109.64Å	
Low resolution limit	46.78	1.69
High resolution limit	1.60	1.60
Rmerge	0.109	0.490
Rmeas (all I+ & I-)	0.118	0.529
Rpim (all I+ & I-)	0.044	0.199
Total no. observations	97542	14015
Total no. unique	14235	2020
Mean((I)/sd(I))	14.6	4.7
Completeness	99.9	99.5
Multiplicity	6.9	6.9