



 ESRF	<b>Experiment title:</b> X-ray crystallographic study of the H <sup>+</sup> -ATPase from <i>N. crassa</i> with and without bound ligands	<b>Experiment number:</b> LS-1150
<b>Beamline:</b> ID14-2+1	<b>Date of experiment:</b> from: 20 Feb to: 21 Feb 2000	<b>Date of report:</b> 28 Feb 00
<b>Shifts:</b> 3+3	<b>Local contact(s):</b> Stefanie MONACO	<i>Received at ESRF:</i> <b>- 3 MAR. 2000</b>
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### Report:

In this round we were able to resume data collection from crystals soaked in various heavy atoms. As in the past, crystals were grown at UNC (Chapel Hill, NC, USA) and frozen in liquid nitrogen. Following a screening session on ID14-1 Feb. 6, 2000, a complete dataset was collected at ID14-2 from a tungstate-soaked crystal showing good resolution and promising isomorphism. As the measurements were carried out at the end of February 2000, we have not yet completed analysis of the data. However, the data quality of the potential tungstate derivative is the highest we have seen yet, and it is likely that this represents the first untwinned crystal observed.

Data extended to approximately 5.8 Å resolution (30% outer shell cut-off), and showed slightly higher resolution than in rounds I (6.8-7.2Å) and II (6.0 Å) (Table 1). The

completeness of the data is low in the two highest resolution shells (6.4-6.1 and 6.1-5.8 Å) due to anisotropy in the diffraction; all other resolution shells are >99% complete.

Table 1

Crystal	Resolution	R <sub>symm</sub>	Completeness	N(0.1)
D05 (Tungstate)	20-5.8 Å	0.066	0.89	0.094
	(6.1-5.8 Å)	(0.298)	(0.52)	

N(0.1) is the fraction of reflections with  $Z < 0.1$ . The number expected based on Wilson statistics is 0.095.

In addition to phasing by isomorphous replacement, a primary goal of the project has been to obtain data comparing different ligand-bound states of the ATPase. Crystals have previously been analyzed in the presence of vanadate. We have now obtained small crystals under identical conditions, but in the absence of vanadate. Although these diffracted poorly at ID14-1 (worse than 10 Å), they showed diffraction to 8 Å at the microfocus beamline (ID13).

The main priorities for the upcoming session are the analysis of further heavy atom soaks and the analysis of larger crystals grown in the presence and absence of vanadate.