ESRF	**Experiment title:** BAG-Frankfurt	**Experiment number**: LS-2189
Beamline: ID14EH1	**Date of experiment**: from: 15.02.03 to: 17.02.03	**Date of report**: 30.07.03
Shifts: 5/6	**Local contact(s)**: Sigrid Kocielski	*Received at ESRF:*

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Report:

Sodium-Proton Antiporter NhaA from *E. coli* (C. Hunte, E. Screpanti)

Three shifts of the beamtime were used for work towards structure determination of NhaA, the main Na^+/H^+ antiporter of *Escherichia coli*. The crystals are generally smaller than 0.2 x 0.1 x 0.1 mm^3 and diffraction cannot be tested at home radiation sources. Cryo-conditions have been successfully established and during the last period crystallization conditions have been identified, which improved the resolution limit to 4-5 Å. Based on this information crystallization conditions were further optimized and 30 crystals of refined conditions were tested during this beamtime. Again, improved crystallization conditions were found and for the first time complete data sets of four different conditions could be collected (3.9-4.0 Å resolution, 89- 95 % completeness) using an exposure time of 120 seconds per frame. The space group P21 was confirmed with ~ a= 109 Å, b= 123 Å, c=121 Å. However, there is a considerable variation in unit cell dimensions between crystals and the angle beta is very close to 90 °. Therefore, space group determination is still ambiguous. Another round of

crystal optimization will be conducted and transferred to crystallization of selenomethionine-NhaA for phase determination.

Quinol:Fumarate-Reductase from *Campylobacter jejuni* (M. Mileni, C. R. D. Lancaster)

Quinol:fumarate reductase (QFR), couples the reduction of fumarate to succinate to the oxidation of quinol to quinone, in a reaction opposite to that catalysed by mitochondrial complex II (succinate dehydrogenase). QFR from *Campylobacter jejuni* consists of three protein subunits, FrdA, FrdB, and FrdC. Crystals of this bioenergetically important membrane protein complex have recently been obtained. During the shift available for this subproject, one data set could be collected (see Table 1) at T = 4°C from just one crystal of space group P1 with unit cell dimensions of a = 130.1 Å, b = 130.9 Å, c = 164.2 Å, α = 108.6°, β = 90.6°, and γ = 118.5°.

Table 1. Diffraction data collected at ESRF ID14-EH1 on a crystal of *C. jejuni* QFR.

	resol. range [Å]	measured reflections	unique reflections	complete [%]	R_{sym} [%]
13_2b	30.0-3.90	237,657	80,480	99.1	10.7
	4.04-3.90		*8,034*	*98.7*	*34.2*

UMP from *Aquifex aeolicus* (G. Peng, C. R. D. Lancaster)

During the shift available to this project, a data set could be collected from a crystal of UMP, a membrane protein of currently unknown identity from *Aquifex aeolicus* (cf. Table 2). The crystal of space group $P2_12_12_1$, with unit cell dimensions a = 104.6 Å, b = 117.7 Å, and c = 192.7 Å, diffracted X-rays to 4.0 Å, but the data was limited to 5.0 Å because of the high mosaicity of 1.6°.

Table 2. Diffraction data collected at ESRF ID14-EH1 on a crystal of *A. aeolicus* UMP.

	resol. range [Å]	measured reflections	unique reflections	complete [%]	R_{sym} [%]
U1-P2_D5_4	30.0-5.00	44,385	9,113	84.3	10.9
	5.18-5.00		*778*	*73.4*	*40.8*

One shift was lost due to shutter problems.