ESRF	**Experiment title:** X-Ray Structure Analysis of the Membrane Protein Complex Fumarate Reductase from *Wollinella succinogenes*	**Experiment number:** LS-538
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Report:

Fumarate reductase (menaquinol:fumarate oxidoreductase) from *Wollinella succinogenes* consists of three subunits, FrdA, FrdB, and FrdC, with a total molecular weight of 130 kDa. FrdC (30 kDa) is a diheme cytochrome b, which anchors the enzyme in the membrane and contains the site for menaquinol oxidation. FrdA (73 kDa) contains covalently bound FAD, a trinuclear [3Fe-4S] iron sulfur centre, and carries the site of fumarate reduction. FrdB (27 kDa) contains binuclear [2Fe-2S] and tetranuclear [4Fe-4S] iron-sulfur centres. This enzyme is currently the best investigated system involved in anaerobic respiration.

Crystals of this bioenergetically important 130 kDa membrane protein complex diffract up to 2.1 Å resolution and have two different unit cells, both of the monoclinic space group $P2_1$. The unit cell of crystal form "A" is a = 87 Å, b = 190 Å, c = 119 Å,

with $\alpha = \gamma = 90°$, and $\beta = 104.6°$, the unit cell of crystal form "B" is a = 119 Å, b = 85 Å, c = 190 Å, with $\alpha = \gamma = 90°$, and $\beta = 96.4°$. Assuming a solvent content of 65%, there are four complexes per unit cell and thus two complexes in the asymmetric units of both unit cells. However many crystals contain both unit cells, and single crystals of both unit cells are not distinguishable morphologically. This makes data collection from several crystals time-consuming, but still feasible.

During the beam time allocated to LS-538, approximately 1100 0.5° oscillation images (including test exposures) to high resolution limits of 3.1 Å were collected from 22 crystals. The acquisition of this large number of exposures was only possible because of the availability of the CCD detector at BM14, thus reducing reading-out time to a minimum. Whenever possible, crystals were translated up to two times after the recording of 20-40 images each, because of deterioration of the diffraction pattern due to radiation damage. Considerable care was taken to set the crystals, which is difficult as the crystallographic axes are not straightforwardly deduced from the crystal morphology. In exceptional cases, the "inverse beam" data collection strategy was used in an attempt to obtain useful anomalous measurements. However, efficient sampling of reciprocal space was thwarted by occasional problems with the goniometer phi motor control!

Of crystal form "A", only 299 of 623 processed (HKL, Otwinowski & Minor) images from four crystals could be successfully merged to a partial data set of 167,788 measured reflections between 25 Å and 3.1 Å (45,891 unique, 70.4% complete), with R_{sym} for the intensities of symmetry-related reflections at 6.1%.

Processing of 272 images of crystal form "B" is currently in progress.

In the case of another putative derivative, 162 of 234 measured images could be merged to a partial data set of 49.2% completeness (15-3.5 A, 61870 measurements, 22329 unique) with R_{sym} (I) at 5.3%.