

**Experiment title:**FUMARATE REDUCTASE, A MEMBRANE
PROTEIN COMPLEX FROM *Wollinella succinogenes***Experiment****number:**

LS-665

Beamline:

BM14

Date of experiment:

from: 26-Apr-97 7:00 to: 30-Apr-97 7:00

Date of report:

25-Aug-97

Shifts:

9

Local contact(s):

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*Received at ESRF:***28 AOUT 1997****Names and affiliations of applicants** (* indicates experimentalists):

C. Roy D. Lancaster* and Hartmut Michel,

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Germany.**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 dihaem 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 at least 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 $a = 87 \text{ \AA}$, $b = 190 \text{ \AA}$, $c = 119 \text{ \AA}$, with $\beta = 104.6^\circ$, the unit cell of form "B" is $a = 119 \text{ \AA}$, $b = 85 \text{ \AA}$, $c = 190 \text{ \AA}$, with $\beta = 96.4^\circ$. 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 (beam-)time-consuming, but still feasible.

During the beam time allocated to LS-665, the original two-wavelength experiment on a putative derivative had to be abandoned because of unexpected problems with the derivatization procedure. Instead, two native data sets were collected, one for each crystal form, to complement the one-wavelength putative derivative data collected under proposal LS-538. For crystal form “A”, 332 (0.5° oscillation) images from 5 crystals could be successfully merged to a data set of better than 2.8 Å resolution (83.1% complete) with R_{sym} for the intensities of symmetry-related reflections at 7.5% ($I/\sigma(I) = 16.4$). For crystal form “B”, 208 images from 6 crystals could be merged to a data set of better than 2.8 Å resolution (78.7% complete) with R_{sym} for the intensities of symmetry-related reflections at 8.9% ($I/\sigma(I) = 14.0$). The acquisition of this large number of high quality exposures was only possible because of the availability of the CCD detector at BM14, thus reducing reading-out time to a minimum. Many more images were collected from further crystals but these data could not be successfully merged to the data sets.