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Acta Cryst. (1994). D**50**, 233-236

Crystallization and preliminary X-ray diffraction analysis of tetra-heme cytochrome c_3 from sulfate- and nitrate-reducing *Desulfovibrio desulfuricans* ATCC 27774.

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(*Received* 30 *July* 1993; *accepted* 21 *October* 1993)

Abstract

Crystals of the tetra-heme cytochrome c_3 (M_r = 13 kDa, 107 residues, four heme groups) from sulfate- and nitrate-reducing *Desulfovibrio desulfuricans* ATCC 27774 have been obtained and crystallographically characterized. They belong to space group $P6_122$ with cell dimensions $a = b = 61.84\,(4)$ and $c = 109.7\,(2)$ Å, and $Z = 12$. Intensity data were initially collected on a FAST system with a rotating-anode X-ray source leading to a total of 22 592 observations, from which only 4930 were unique, in the resolution range 20.0–2.4 Å with an $R_{merge}(I)$ of 7.0%. Higher resolution data were measured on a FAST system at station 9.6 of the SRS (Daresbury, England), leading to 19 328 intensities, of which 11 179 were unique, in the resolution range 20.0–1.75 Å and an $R_{merge}(I)$ of 5.5%. Cross-rotation and translation functions were performed with *ALMN* and *TFSGEN* programs from the *CCP*4 suite. The packing of the molecules in the unit cell was checked with *TOM/FRODO*. Rigid-body refinement of the model and subsequent refinement using molecular dynamics were performed with *X-PLOR*, leading to a current R factor of 25.9%, for data up to 2.3 Å.

Introduction

Desulfovibrio desulfuricans ATCC 27774 is a bacterial strain belonging to the sulfate-reducing *Desulfovibrionaceae* family, with the unique capability of growing either in sulfate or in nitrate. These anions can be used as terminal acceptors of the electron-transport chain, while organic compounds or hydrogen are the initial electron donors for this obligate anaerobic family of bacteria (Postgate & Campbell, 1966; Peck, 1984). The metabolism of hydrogen is regulated by a reversible hydrogenase, which appears to be reduced or oxidized by the low-potential cytochrome c_3 (LeGall, Moura, Peck & Xavier, 1982; LeGall & Fauque, 1988). This is a *c*-type cytochrome which belongs to class III (Ambler, 1982; Moore & Pettigrew, 1990), which includes tri-heme, tetra-heme, octa-heme and also hexadeca-heme cytochromes. These are characterized by bishistidinyl iron coordination with 20–40 residues per heme. The characteristically very low redox potentials of these cytochromes, in the range −110 to −400 mV, differentiates them from class I (0 to +470 mV) or class II (−10 to +200 mV) cytochromes (Ambler, 1991; Moura, Costa, Liu, Moura & LeGall, 1991).

Three multi-heme *c*-type cytochromes were isolated from the soluble fraction of *D. desulfuricans* ATCC 27774 grown under nitrate- or sulfate-reducing conditions: *split-soret* cytochrome *c*, dodeca-heme cytochrome *c* and the tetra-heme cytochrome c_3. However, only the latter is similar to c_3 cytochromes found in other sulfate-reducing bacteria (Liu *et al.*, 1988).

The tetra-heme c_3 cytochromes have been intensively studied as they constitute very attractive models for intra- or inter-electron transfer molecules. Sequence homology for these four heme cytochromes (Meyer & Kamen, 1982, and references therein) can be as low as 30%, with conserved residues accounting mainly for the heme attachment. There are always 16 conserved residues of the 110–120 amino acids of the peptide chain, responsible for the covalent linkage to the hemes plus the histidinyl groups coordinating the four Fe atoms. Although the heme-heme arrangement found in the structures determined already seems to remain highly conserved in its architectural geometry, the wide range of redox potentials observed must be related to differences to be found in the three-dimensional molecular structures of the various c_3 cytochromes. Both X-ray diffraction analysis (Haser, Pierrot *et al.*, 1979; Higuchi, Kusunoki, Matsuura, Yasuoka & Kakudo, 1984; Kissinger, 1989; Morimoto, Tani, Okumura, Higuchi & Yasuoka, 1991; Matias, Frazão, Morais, Coll & Carrondo, 1993) and two-dimensional NMR (Coutinho, Turner, LeGall & Xavier, 1992; Turner, Salgueiro, LeGall & Xavier, 1992) techniques have been used to study the three-dimensional structures of these molecules. These structures were essential for the interpretation of abundant physicochemical data from Mössbauer spectroscopy, circular dichroism (CD), electron paramagnetic resonance (EPR), nuclear magnetic resonance (NMR) and from electrochemical methods (Moura *et al.*,

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1991, and references therein). In addition, three-dimensional computational calculations were used to propose models for the complexes of c_3 cytochromes with their redox partners ferredoxin (Cambillau, Frey, Mosse, Guerlesquin & Bruschi, 1988), rubredoxin (Stewart *et al.*, 1989) and with the *in vitro* partner flavodoxin (Stewart *et al.*, 1988; Palma *et al.*, 1993), as well as to study the electron transfer between the different redox centres.

Crystal forms of tri-heme (Haser, Payan, Bache, Bruschi & LeGall, 1979), tetra-heme (Frey, Haser, Pierrot, Bruschi & LeGall, 1976; Bando *et al.*, 1979; Sieker, Jensen & LeGall, 1986; Kissinger, 1989; Morimoto *et al.*, 1991; Matias *et al.*, 1993) and octa-heme (Sieker *et al.*, 1986; Czejk *et al.*, 1992) c_3 cytochromes have been reported in the literature, but only the structures of some tetra-heme cytochromes have been solved, namely those from *Desulfomicrobium baculatus* Norway 4 (Haser, Pierrot *et al.*, 1979), *Desulfovibrio vulgaris* Miyazaki F (Higuchi *et al.*, 1984), *Desulfovibrio gigas* (Kissinger, 1989) and *Desulfovibrio vulgaris* Hildenborough (Morimoto *et al.*, 1991; Matias *et al.*, 1993). Here we report results on the crystallization of another tetra-heme cytochrome c_3, from *Desulfovibrio desulfuricans* ATCC 27774, and its structure determination using the molecular-replacement technique.

Materials and methods

Protein biosynthesis, extraction, purification and crystallization

The composition of media used for growing nitrate- or sulfate-respiring bacteria of *Desulfovibrio desulfuricans* ATCC 27774 has been described in the literature by Liu & Peck (1981) and Peck (1984), as well as the preparation and fractionation of cell extracts, isolation and purification of tetra-heme cytochrome c_3 by Liu *et al.* (1988). Crystals of cytochrome c_3 were obtained by the vapour-diffusion method using sitting drops and ammonium sulfate as the precipitating agent. Crystals were obtained by equilibrating drops containing 6 µl of a protein solution of 13 mg ml^{-1} in 0.1 *M* Tris-HCl buffer (pH 7.6), plus 4 µl of 3.5 *M* ammonium sulfate solution in 0.05 *M* sodium acetate buffer (pH 4.0) and 2 µl of a 4.0 *M* ammonium sulfate solution, against 3 ml of 3.5 *M* ammonium sulfate in 0.05 *M* sodium acetate buffer (pH 4.0). Crystals appeared after 4–5 d at room temperature and continued to grow up to a maximum size of *ca* 1.0 × 0.6 × 0.6 mm.

Crystallographic diffraction data collection and processing

An initial X-ray diffraction data set to 2.4 Å was collected in our laboratory on an Enraf-Nonius FAST area-detector diffractometer using graphite-monochromated Cu $K\alpha$ radiation, λ = 1.5418 Å, from an Enraf-Nonius FR571 rotating-anode generator. The determination of the space group, the unit-cell dimensions and the intensity-data measurements were carried out online using the program *MADNES* (Pflugrath & Messerschmidt, 1989). The cell parameters reported correspond to the mean and e.s.d. values calculated from the refined values during data collection. A second data set to 1.75 Å was collected at station 9.6 of the SRS (Daresbury, England), with synchrotron radiation, λ = 0.89 Å, also using an Enraf-Nonius FAST area-detector diffractometer. Intensity data were collected and processed offline with the program *MADNES* (Pflugrath & Messerschmidt, 1989). Individual intensities for each of the data sets were scaled, merged and statistically improved with programs *AGROVATA*, *ROTAVATA* and *TRUNCATE* from *CCP*4 suite (SERC Daresbury Laboratory, 1979).

Molecular-replacement studies

The coordinates of *Desulfovibrio vulgaris* Miyazaki F (Higuchi *et al.*, 1984) were extracted from the Protein Data Bank (Bernstein *et al.*, 1977), translated to obtain their centre of masses as the origin and used as search model for molecular-replacement purposes.

Molecular-replacement calculations were performed using programs from the *CCP*4 suite (SERC Daresbury Laboratory, 1979). Structure factors were calculated at 3.0 Å using atomic coordinates and temperature factors with *GENSFC* incorporating the *FFT* program, for an orthogonal cell in *P*1 symmetry, and dimensions twice those for each spacial direction of the model. Normalized structure-factor amplitudes for both the search molecule and the native data were obtained with *ECALC*. Cross-rotation searches were performed with *ALMN*, based on Crowther's fast-rotation function. Maps were calculated using 2.5° steps in each of the Eulerian angles, with Bessel functions of order up to 60. Maps were investigated in the resolution range 20.0–3.0 Å and integration Patterson radii of 15, 18, 20, 22, 25 and 30 Å.

For translation-function calculations the search model was placed in a cell of dimensions identical to the crystal unit cell. *GENSFC* calculated structure factors with *P*1 symmetry from the atomic coordinates with temperature factors. Partial structure factors were calculated to 2.0 Å for each symmetry-related molecule in Laue group 622 using the *PREPARE*, *COLLATE* and *MERGE* steps. As no space-group translations were applied, the partial structure factors could be used for translation functions in both $P6_122$ and $P6_522$. Maps were calculated in space groups $P6_122$ and $P6_522$ using data between 20.0 and 4.0 Å, and between 20.0 and 3.0 Å, by the programs *TFSGEN-FFT*, and solution analysis was performed by *MAPSIG*.

Packing of the final solution was visually verified on a graphics workstation with the programs *TOM/FRODO* (Cambillau & Horjales, 1987; Roussel, Fontecilla-Camps & Cambillau, 1990), and *MOLPACK* (Wang, Driessen & Tickle, 1991).

Results and discussion

Crystal data

The hexagonal prismatic crystals were found to belong to the hexagonal space group $P6_122$ or $P6_522$, with cell dimensions $a = b = 61.84\,(4)$, $c = 109.7\,(2)$ Å, and containing a solvent fraction of 47% assuming one molecule per asymmetric unit ($Z = 12$).

Diffraction data

Intensity data were initially collected from a rotating-anode source leading to a total of 22 592 observations of 4930 unique reflections in the resolution range 20.0–2.4 Å with an $R_{merge}(I)$ of 7.0%. The unique data set comprised 93.0% of the theoretically predicted reflections, with 79.7% of the amplitudes having $I > 3\sigma(I)$.

A higher resolution data set was measured with synchrotron radiation leading to 19 328 intensities, merged into 11 179 unique amplitudes and comprising 84.8% of the theoretically predicted reflections in the resolution range 20.0–1.75 Å. The $R_{merge}(I)$ obtained was 5.5%, with 81.9% of the amplitudes having $I > 3\sigma(I)$.

Search and refinement models

All c_3 cytochromes contain 16 obligatory homologous residues, as a result of the cysteinic covalent bonding of the peptide chain to the hemes plus the axial histidinic iron linking. In addition, it seems that seven other amino acids are consistently present in all known sequences of this family (Meyer & Kamen, 1982), which implies a general basic homology of *ca* 22% among all c_3 cytochromes. For molecular-replacement purposes the cytochrome c_3 from *D. vulgaris* Miyazaki F was used as the search model. This is 45% homologous to cytochrome c_3 from *D. desulfuricans* ATCC 27774, and its structural parameters [available from the Protein Data Bank (Bernstein *et al.*, 1977)] have been refined (Higuchi *et al.*, 1984) to R = 19% from data up to 1.8 Å resolution.

Comparison of the recently determined amino-acid sequence of cytochrome c_3 from *D. desulfuricans* ATCC 27774 (J. Van Beeuman and coworkers, University of Ghent, unpublished results), with that of *D. vulgaris* Miyazaki F (Shinkai, Hase, Yagi & Matsubara, 1980), reveals that both cytochromes have the same length of 107 amino acids; nevertheless the differences in the numbering of the obligatory histidine-iron bonding residues would imply at least one insertion and one deletion within the peptide chain. For refinement of the molecular-replacement solution, a model was obtained by aligning both primary structures and replacing all mutated amino acids from the search model by the corresponding ones in *D. desulfuricans* ATCC 27774, using initially a zero occupancy factor for all new added atoms (dummy atoms). Residues corresponding to insertions or deletions were not included in the model but enabled the definition of the segmented peptide chains that were used in a multiple rigid-body refinement.

Rotation function

Cross-rotation calculations were carried out using three initial orientations of the model, the orientation of Miyazaki's structure and two other arbitrary rotations through Eulerians [38, 79, 333°] and [9, 42, 312°]. Different radii of Patterson integration and resolution data up to 3.0 Å were used in the calculations. A consistent main peak emerged from most of the runs, with heights about five times the root mean square of the maps, for Patterson radii in range of 20-30 Å and clearly above all noise peaks. The best result was obtained for an integration to 30 Å at Eulerian angles of $[\alpha, \beta, \gamma]$ = [11.3, 81.5, 63.0°] from Miyazaki's orientation, with a signal of 5.7σ and a 1.3σ difference relative to the first noise peak.

Translation function

Having found the rotation-function solution, translation-function calculations were performed in both possible space groups $P6_122$ and $P6_522$, for the two resolution cut-off ranges of 20.0-3.0 and 20.0-4.0 Å. While in space group $P6_122$ a consistent solution for both calculations clearly emerged above all other features (about 11σ and a difference of 3.5σ relative to first noise peak) at fractional coordinates of $[u, v, w]$ = [0.64, 0.86, 0.34], in space group $P6_522$ not only was there no peak clearly above the root mean square of the maps (typical values for noise peaks are *ca* $4\text{-}5\sigma$ with differences of 0.1σ) but also the two resolution cut-offs originated different rankings for common noise peaks.

Packing verification

A visual verification of the correctness of the molecular-replacement solution was obtained. The 12 molecules packed well in terms of sensible van der Waals contacts both within the unit cell and also with neighbouring crystallographic cells, suggesting that the model used for Patterson search was indeed similar to the crystallographic structure.

Initial refinement

Refinement of the search model was initially performed as a unique rigid body using *X-PLOR* (Brünger, 1992). Data between 8.0 and 3.5 Å were used for ten cycles but no decrease in either the R factor or the total term of empirical energy was observed. Further rigid-body refinement was undertaken using 12 rigid bodies corresponding to the four heme groups plus eight segments of peptide chain, originating from modelled insertions and deletions. After 90 cycles of refinement a decrease in R from 51.7 to 48.5% was observed. Further atomic refinement with data between 8.0 and 3.0 Å led to an R factor of 36% after 554 cycles. The current R factor for all data between 8.0 and 2.3 Å reached 25.9%.

The authors are grateful to Dr P. Rizkallah and to Dr M. Papiz for the supporting the X-ray diffraction data collection and processing at station 9.6, SRS (Daresbury, England), to Professor J. Van Beeuman for the amino-acid sequence of *D. desulfuricans* ATCC 27774 prior to publication, to Professor J. LeGall and C. Costa for providing the purified protein and also to Professors I. M. Moura and J. J. G. Moura for useful discussions. The bacteria were grown by the staff of the Fermentation Plant at the University of Georgia, USA. This work was supported by JNICT grant PBIC/C/BIO/1277/92 and JNICT fellowship BD/2155/92-IF to JM.

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Acta Cryst. (1994). D**50**, 236

Refined structure of cadmium-substituted concanavalin A at 2.0 Å resolution. Erratum.

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(*Received* 6 *December* 1993)

Abstract

An error in the paper by Naismith, Habash, Harrop, Helliwell, Hunter, Wan, Weisgerber, Kalb & Yariv [*Acta Cryst.* (1993), D**49**, 561-571] is corrected. The first sentence of the caption for Fig. 6 on p. 568 should read: The *S*1 (Cd^{2+}) and *S*2 (Ca^{2+}) metal sites.

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All relevant information is given in the *Abstract*.

Acta Crystallographica Section D
ISSN 0907-4449 © 1994