The solid-state packing involves cation–anion H-bonding between amine groups and nitrate oxygen atoms [N(1)—O($7^{\rm i}$) = 2·897 (5); N(4)—O($6^{\rm ii}$) = 2·963 (6) Å]* to form zigzag chains of cations and anions which are related by translation along x. The cations of this chain are also directly related by H-bonding between an amine group and a carboxyl oxygen atom [N(4)—O($2^{\rm iii}$) = 2·904 (4) Å]. Another set of amine–carboxyl H-bonds [N(1)—O($4^{\rm iv}$) = 3·007 (5) Å] occurs between cations related by y translation. A bifurcated H-bond occurs between an imidazole NH group of one cation and carboxyl oxygen atoms of two different cations [N(3)—O($2^{\rm v}$) = 2·970 (5); N(3)—O($4^{\rm vi}$) = 2·888 (4) Å], which are related to the first by space-group symmetry coupled with x and y translations, respectively.

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* Symmetry code: (i) O(7) at x, y, z; (ii) O(6) at $x-1, y, z$; (iii) O(2) at $x-1, y, z$; (iv) O(4) at $x, y-1, z$; (v) O(2) at $1-x, \frac{1}{2}+y, -z$; (vi) O(4) at $-x, y-\frac{1}{2}, -z$.

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Structure of (Benzenethiolato)(2,3,7,8,12,13,17,18-octaethylporphinato)iron(III), $[Fe(C_{36}H_{44}N_4)(C_6H_5S)]$

BY KATHLEEN M. MILLER AND CHARLES E. STROUSE*

J. D. McCullough Laboratory for X-ray Crystallography, Department of Chemistry and Biochemistry, University of California, Los Angeles, California 90024, *USA*

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Abstract. M_r = 697·78, triclinic, $P\bar{1}$, a = 10·246 (4), b = 13·040 (4), c = 14·900 (5) Å, α = 107·82 (4), β = 73·00 (4), γ = 101·98 (5)°, V = 1797·12 Å^3, Z = 2, D_m = 1·30 (1), D_x = 1·29 Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, μ = 0·508 mm^{-1}, F(000) = 742, T = 117 K, R = 0·069, R_w = 0·076 for 4676 unique reflections. In the five-coordinate complex Fe(oep)(C_6H_5S) the Fe atom is displaced 0·466 (1) Å from the plane of the four N atoms and 0·512 (1) Å from the plane of the 24-atom porphyrin core. The Fe—S distance is 2·299 (3) Å and the average Fe—N distance is 2·057 (6) Å.

Introduction. Studies of the hemoprotein, cytochrome P-450, and of small-molecule analogues of the heme active site have suggested that a thiolate ligand is coordinated to the heme center of the enzyme (Blumberg & Peisach, 1971; Collman & Groh, 1982; Dolphin, 1979). Accurate and unambiguous structural parameters have been reported for only two thiolate-ligated ferric porphyrin complexes, Fe(ppixdme)(p-$NO_2C_6H_4S$) (Tang, Koch, Papaefthymiou, Foner, Frankel, Ibers & Holm, 1976) and $[Fe(tpp)(C_6H_5S)_2]^-$ (Byrn & Strouse, 1981) (ppixdme is protoporphyrin IX dimethyl ester and tpp is tetraphenylporphinato). Herein is reported the structure of a third iron porphyrin thiolate complex, Fe(oep)(C_6H_5S).

Experimental. A solution of 50 mg $[Fe(oep)]_2O$ in 20 ml benzene was stirred under dinitrogen with 12·5 ml 15% H_2SO_4(v/v). After one hour, 2·5 ml benzenethiol was added with rapid stirring. The organic phase was separated, and crystallization was achieved by slow evaporation under a stream of dinitrogen. Purple, rectangular parallelepiped single crystal, 0·20 × 0·25 × 0·28 mm. D_m by flotation in KI/water. Picker diffractometer equipped with a variable temperature device (Strouse, 1976), Zr filter. Unit-cell parameters by least-squares refinement of 10 reflections

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* To whom correspondence should be addressed.

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Table 1. *Positional parameters and U_{eq} (Å^2) values with estimated standard deviations for the non-hydrogen atoms*

$$U_{\rm eq} = \frac{1}{6\pi^2}\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}(\times 10^3)$
Fe	0·3033 (1)	1·1261 (1)	0·3043 (1)	20 (1)
Porphyrin core atoms				
C(1)	0·2352 (7)	1·0334 (6)	0·4825 (5)	19 (7)
C(2)	0·2560 (8)	1·0609 (6)	0·5802 (5)	25 (8)
C(3)	0·3457 (8)	1·1532 (6)	0·5893 (5)	24 (7)
C(4)	0·3815 (7)	1·1822 (6)	0·4984 (6)	24 (8)
C(5)	0·4741 (8)	1·2715 (6)	0·4783 (6)	25 (8)
C(6)	0·5201 (8)	1·2954 (6)	0·3891 (6)	30 (8)
C(7)	0·6269 (8)	1·3836 (6)	0·3677 (7)	37 (9)
C(8)	0·6518 (8)	1·3740 (7)	0·2714 (7)	41 (10)
C(9)	0·5597 (8)	1·2811 (7)	0·2340 (6)	35 (9)
C(10)	0·5484 (8)	1·2469 (7)	0·1395 (7)	39 (10)
C(11)	0·4535 (7)	1·1630 (6)	0·1026 (6)	26 (8)
C(12)	0·4412 (8)	1·1318 (7)	0·0024 (6)	30 (8)
C(13)	0·3376 (8)	1·0481 (7)	−0·0030 (6)	30 (8)
C(14)	0·2881 (8)	1·0270 (6)	0·0914 (5)	24 (8)
C(15)	0·1875 (8)	0·9449 (6)	0·1156 (5)	26 (8)
C(16)	0·1463 (7)	0·9157 (6)	0·2015 (5)	22 (7)
C(17)	0·0516 (7)	0·8216 (6)	0·2201 (6)	22 (8)
C(18)	0·0437 (7)	0·8217 (6)	0·3124 (5)	19 (7)
C(19)	0·1314 (7)	0·9161 (5)	0·3513 (5)	18 (7)
C(20)	0·1499 (7)	0·9467 (6)	0·4449 (5)	23 (7)
N(1)	0·3139 (6)	1·1090 (4)	0·4343 (4)	19 (6)
N(2)	0·4796 (6)	1·2348 (5)	0·3067 (5)	26 (6)
N(3)	0·3604 (6)	1·0979 (5)	0·1550 (4)	22 (6)
N(4)	0·1955 (6)	0·9723 (5)	0·2824 (4)	18 (6)
Ethyl atoms				
C(21)	0·1889 (8)	0·9956 (6)	0·6524 (5)	27 (8)
C(22)	0·2661 (9)	0·9006 (7)	0·6372 (6)	37 (9)
C(23)	0·4099 (8)	1·2103 (7)	0·6735 (6)	34 (8)
C(24)	0·5519 (9)	1·1782 (8)	0·6582 (6)	47 (10)
C(25)	0·6967 (9)	1·4657 (6)	0·4398 (7)	41 (9)
C(26)	0·8129 (9)	1·4217 (6)	0·4569 (7)	44 (10)
C(27)	0·7613 (13)	1·4397 (8)	0·2093 (8)	76 (14)
C(28)	0·6960 (14)	1·5038 (10)	0·1729 (10	110 (18)
C(29)	0·5261 (9)	1·1851 (7)	−0·0744 (6)	38 (9)
C(30)	0·4800 (10)	1·2920 (7)	−0·0664 (6)	45 (10)
C(31)	0·2781 (9)	0·9898 (7)	−0·0893 (5)	34 (8)
C(32)	0·1605 (9)	1·0451 (8)	−0·0872 (6)	46 (10)
C(33)	−0·0164 (8)	0·7392 (6)	0·1464 (5)	26 (8)
C(34)	0·0696 (10)	0·6513 (7)	0·0845 (7)	49 (10)
C(35)	−0·0367 (7)	0·7388 (6)	0·3656 (6)	26 (8)
C(36)	0·0517 (8)	0·6588 (6)	0·3666 (6)	34 (8)
Benzenethiolate atoms				
S	0·1336 (2)	1·2375 (2)	0·3607 (2)	31 (2)
C(37)	0·1668 (9)	1·3032 (7)	0·2673 (6)	36 (9)
C(38)	0·0984 (15)	1·2639 (9)	0·1946 (8)	88 (16)
C(39)	0·1152 (31)	1·3167 (19)	0·1260 (13)	174 (37)
C(40)	0·2073 (33)	1·4082 (21)	0·1270 (14)	171 (41)
C(41)	0·2780 (16)	1·4515 (12)	0·1965 (13)	113 (21)
C(42)	0·2581 (10)	1·3972 (8)	0·2697 (8)	61 (12)

Table 2. *Bond distances* (Å) *and interbond angles* (°), *with estimated standard deviations*

Fe–S	2·299 (3)	N(1)–Fe–S	99·3 (2)
		N(2)–Fe–S	102·7 (2)
Fe–N(1)	2·052 (6)	N(3)–Fe–S	106·8 (2)
Fe–N(2)	2·054 (6)	N(4)–Fe–S	103·5 (2)
Fe–N(3)	2·062 (6)		
Fe–N(4)	2·059 (6)	N(1)–Fe–N(2)	87·5 (2)
		N(1)–Fe–N(4)	87·0 (2)
C(37)–S	1·766 (8)	N(2)–Fe–N(3)	87·0 (2)
		N(4)–Fe–N(3)	86·8 (2)
C(1)–N(1)	1·379 (9)	N(1)–Fe–N(3)	153·9 (2)
C(4)–N(1)	1·363 (9)	N(2)–Fe–N(4)	153·8 (2)
C(6)–N(2)	1·368 (10)		
C(9)–N(2)	1·373 (9)	C(37)–S–Fe	102·5 (3)
C(11)–N(3)	1·369 (9)		
C(14)–N(3)	1·375 (9)	C(4)–N(1)–Fe	126·0 (5)
C(16)–N(4)	1·381 (9)	C(1)–N(1)–Fe	127·1 (5)
C(19)–N(4)	1·374 (9)	C(6)–N(2)–Fe	124·9 (5)
		C(9)–N(2)–Fe	127·9 (5)
C(1)–C(20)	1·358 (10)	C(11)–N(3)–Fe	126·7 (5)
C(4)–C(5)	1·391 (10)	C(14)–N(3)–Fe	125·7 (5)
C(5)–C(6)	1·383 (11)	C(19)–N(4)–Fe	127·1 (5)
C(9)–C(10)	1·373 (11)	C(16)–N(4)–Fe	126·1 (5)
C(10)–C(11)	1·396 (11)		
C(14)–C(15)	1·378 (10)	C(4)–N(1)–C(1)	105·9 (6)
C(15)–C(16)	1·370 (10)	C(6)–N(2)–C(9)	105·5 (6)
C(19)–C(20)	1·387 (10)	C(11)–N(3)–C(14)	105·7 (6)
		C(19)–N(4)–C(16)	105·3 (6)
C(1)–C(2)	1·457 (10)		
C(3)–C(4)	1·441 (10)	C(20)–C(1)–N(1)	125·1 (7)
C(6)–C(7)	1·456 (10)	N(1)–C(4)–C(5)	124·7 (7)
C(8)–C(9)	1·445 (11)	N(2)–C(6)–C(5)	124·9 (7)
C(11)–C(12)	1·459 (11)	N(2)–C(9)–C(10)	124·3 (8)
C(13)–C(14)	1·440 (10)	N(3)–C(11)–C(10)	124·8 (7)
C(16)–C(17)	1·442 (10)	N(3)–C(14)–C(15)	123·6 (7)
C(18)–C(19)	1·438 (9)	C(15)–C(16)–N(4)	123·5 (7)
		N(4)–C(19)–C(20)	124·3 (6)
C(2)–C(3)	1·351 (10)		
C(7)–C(8)	1·352 (11)	N(1)–C(1)–C(2)	109·9 (6)
C(12)–C(13)	1·356 (11)	N(1)–C(4)–C(3)	110·6 (6)
C(17)–C(18)	1·353 (10)	N(2)–C(6)–C(7)	110·3 (7)
		N(2)–C(9)–C(8)	111·0 (7)
		N(3)–C(11)–C(12)	110·7 (7)
		N(3)–C(14)–C(13)	110·3 (7)
		N(4)–C(16)–C(17)	110·4 (6)
		N(4)–C(19)–C(18)	110·5 (6)

($9·50 \leq 2\theta \leq 19·78°$). θ–2θ scan, 2·0° min^{-1}, $2\theta_{\rm max} = 45°$ for the range $0 \leq h \leq 11$, $-14 \leq k \leq 13$, $-15 \leq l \leq 16$. Three reflections monitored every 100 reflections with no significant variation. 4676 unique data, 3100 with $I > 3\sigma(I)$, 001 reflection inaccurately measured and deleted, 1576 considered unobserved. Lorentz, polarization and absorption corrections applied (range 0·96 to 0·94). Structure solved by heavy-atom method. All non-hydrogen atoms located on Fourier and difference Fourier maps; H atoms located on difference Fourier maps or calculated. Anisotropic thermal parameters for all non-hydrogen atoms; isotropic thermal parameters for H fixed at $U_{\rm iso} = 0·0253$ Å^2. Full-matrix least-squares refinement based on F with max. $\sin\theta/\lambda = 0·54$ Å^{-1}. Refinement of 433 parameters converged to $R_w = 0·076$, $w = 1/\sigma^2(F_o)$, error of fit $S = 2·29$. $(\Delta/\sigma)_{\rm max} = 0·30$. Max. height 2·36 e Å^{-3} in final difference Fourier synthesis. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). All calculations performed on a UCLA Departmental DEC VAX 11/780 with the *UCLA Crystallographic Package* (1983) (locally edited versions of *CARESS, PROFILE, MULTAN, ORFLS, ORFFE, ABSORB* and *ORTEP*).

The largest features in the difference map were two peaks of height 2·36 and 1·02 e Å^{-3}, located near the C(27)–C(28) ethyl group; these C atoms also displayed large thermal parameters. This evidence suggests disorder of this moiety. Attempts to correct for the disorder with half-weighted models were unsuccessful. Disorder in peripheral porphyrin substituents has been observed in other structures (Hamor, Caughy & Hoard, 1965; Hoard, Hamor, Hamor & Caughy, 1965; Koenig, 1965; Little & Ibers, 1974).

Discussion. Atomic coordinates and equivalent isotropic temperature factors are listed in Table 1 and bond lengths and angles are given in Table 2.* An *ORTEP* drawing is shown in Fig. 1 with the atom-numbering scheme, and a packing diagram is displayed in Fig. 2. The bond distances and bond angles within the porphyrin core are similar to those of other reported octaethylporphyrin structures (Cullen & Meyer, 1976; Einstein & Willis, 1978; Masuda, Taga, Osaki, Sugimoto, Yoshida & Ogoshi, 1980; Scheidt & Geiger, 1982; Takenaka, Sasada, Watanabe, Ogoshi & Yoshida, 1972). The FeN_4S moiety has the pyramidal structure found for other five-coordinate, high-spin Fe^{III} porphyrin complexes (Hoard, Cohen & Glick, 1967; Hoffman, Collins, Day & Fleischer, 1972). The Fe—N distances range from 2·052 (6) to 2·062 (6) Å, with a mean distance of 2·057 (6) Å. The Fe atom is displaced 0·466 (1) Å from the N_4 plane and 0·512 (1) Å from the mean porphyrin plane toward the axial thiolate ligand. These values are in the range reported for other five-coordinate, Fe^{III} porphyrin complexes (Cullen & Meyer, 1976; Hoard, 1971; Scheidt, 1977; Scheidt & Reed, 1981), and, in particular, are similar to the distances reported for the five-coordinate Fe^{III} porphyrin thiolate complex, Fe(ppixdme)(p-$NO_2C_6H_4S$) (Tang, Koch, Papaefthymiou, Foner, Frankel, Ibers & Holm, 1976). In this complex, the average Fe—N distance is 2·064 (18) Å and the Fe displacements from the N_4 plane and the mean porphyrin plane are 0·434 and 0·448 Å, respectively. The Fe—S bond distance of 2·299 (3) Å in the title complex is 0·025 Å shorter than the corresponding distance in Fe(ppixdme)(p-$NO_2C_6H_4S$). The large thermal parameters observed for the benzenethiolate C atoms in Fe(oep)(C_6H_5S) reflect the freedom of motion of this axial ligand within the crystal lattice and do not appear to be associated with the disorder of the C(27)—C(28) ethyl group.

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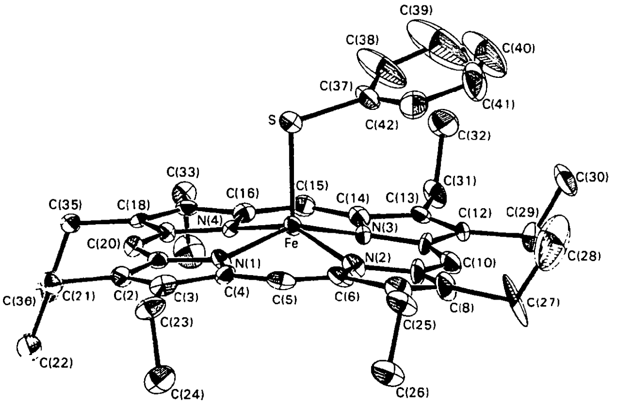

Fig. 1. Molecular structure showing the numbering scheme (50% probability ellipsoids). H atoms and selected atom labels have been omitted for clarity.

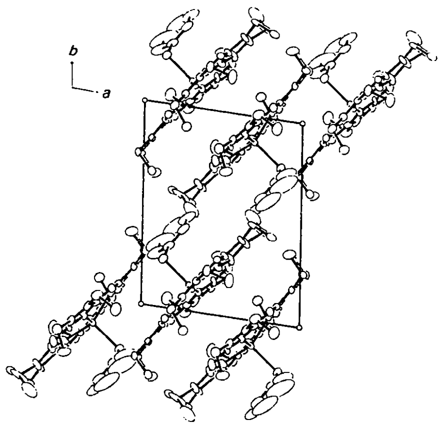

Fig. 2. Packing diagram down the c axis, with the b axis vertical.

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* Lists of structure factors, anisotropic thermal parameters, H-atom positions, porphyrin, ethyl, and phenyl bond distances and bond angles, and perpendicular displacements from the 24-atom porphyrin-core least-squares plane have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39373 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of *mer*-Trichlorotris(pyridine)rhodium(III), $[RhCl_3(C_5H_5N)_3]$*

BY K. R. ACHARYA, S. S. TAVALE AND T. N. GURU ROW

National Chemical Laboratory, Pune-411 008, *India*

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Abstract. $M_r = 443{\cdot}45$, monoclinic, $P2_1/n$, $a = 9{\cdot}008\ (2)$, $b = 12{\cdot}457\ (2)$, $c = 15{\cdot}445\ (2)$ Å, $\beta = 90{\cdot}87\ (2)°$, $V = 1732{\cdot}9$ Å^3, $Z = 4$, D_m(flotation) = 1·63, $D_x = 1{\cdot}65$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}7107$ Å, $\mu = 13{\cdot}74$ mm^{-1}, $F(000) = 888{\cdot}0$, $T = 293$ K, $R = 0{\cdot}052$ for 1171 reflections. The rhodium atom has nearly regular octahedral coordination with three meridional chlorines with bond lengths ⟨Rh—Cl⟩ = 2·334 (5) and ⟨Rh—N⟩ = 2·057 (13) Å. The three pyridine moieties are planar and are nearly perpendicular to each other. The molecules are held together by van der Waals forces.

Introduction. Rhodium complexes with pyridine have not been well studied in spite of their growing importance as homogeneous catalysts, *e.g.* for carbonylation of nitro compounds (Hammond & Franco, 1974). It is often desirable to use catalysts with well defined structures so that a suitable mechanism for the reaction can be evolved. In this context, the structure of $Rh(py)_3Cl_3$ has been studied.

Experimental. Synthesis from $RhCl_3.3H_2O$ and pyridine (Chaudhari & Rode, 1983); crystal (orange colour, needle shape) approximate dimensions 0·18 × 0·15 × 0·25 mm used for data collection; lattice parameters from 25 reflections ($12 < 2\theta < 35°$); intensity data collected on Enraf–Nonius CAD-4F-11M single-crystal X-ray diffractometer; graphite-monochromated Mo $K\alpha$ radiation; 3 standard reflections (060, 402, $0\bar{4}8$), ≤ 5% intensity variation; $\omega/2\theta$ scan mode; scan speed 1° min^{-1}; $\theta \leq 24°$; 3054 reflections collected, 1171 judged significant ($|F_o| \geq 3\sigma|F_o|$); h 0–10, k 0–13, l 0–±17; no correction for absorption; structure solved by direct methods using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978); Fourier map based on rhodium peak and four other peaks which formed the right kind of geometry revealed the entire structure. Full-matrix refinement of scale factor, positional and anisotropic thermal parameters (isotropic thermal parameters for H atoms, located from difference map) converged to $R = 0{\cdot}052$ and $R_w = 0{\cdot}054$; $\sum w(|F_o| - |F_c|)^2$ minimized, $w = (18{\cdot}0 + 1{\cdot}0|F_o| + 0{\cdot}014|F_o|^2)^{-1}$; atomic scattering factors from *International Tables for X-ray Crystallography* (1974); max. $\Delta/\sigma = 0{\cdot}1$, final $\Delta\rho$ excursions $< |0{\cdot}3|$ e Å^{-3}; *LALS* (Gantzel, Sparks & Trueblood, 1961) used for refinement.

Discussion. The atomic parameters with their standard deviations for non-H atoms are given in Table 1. Bond lengths and angles are given in Table 2.† Fig. 1 gives a perspective view (*ORTEP*; Johnson, 1965) of the molecule along with the numbering of atoms. The rhodium has a nearly regular octahedral coordination with three meridional chlorines. The ⟨Rh—Cl⟩ distance of 2·334 (5) Å and ⟨Rh—N(py)⟩ distance of 2·057 (13) Å agree well with those reported (Rozière, Lehmann & Potier, 1979; Murray-Rust, 1977).This coordination around rhodium particularly resembles that in trichloro(dimethyl sulfoxide)bis(pyridine)-rhodium (Colamarino & Orioli, 1976) with ⟨Rh—Cl⟩ distance of 2·334 (5) Å and ⟨Rh—N(py)⟩ distance of 2·052 (13) Å, the averages being over the two independent molecules. The planar pyridine moieties are nearly perpendicular to each other and their geometry is in good agreement with the structure of *trans*-dichlorotetrakis(pyridine)rhodium(III)

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* NCL Communication No. 3421.

† Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares-planes' information have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39349 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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