Fig. 2. Unit-cell packing diagram for $\{[(C_5H_5)FeP(C_6H_5)_3-(CO)NCC_5H_{11}CO_2][BF_4]\}_2$.

anisotropic, H atoms idealized (except for Hx which was found and refined) and updated (C—H = 0·96 Å, $U = 1\cdot2U$ of attached C); phenyl rings constrained to rigid hexagons, C—C = 1·395 Å. $R_F =$ 5·29%, $wR_F = 6\cdot54\%$, $S = 0\cdot35$, $w^{-1} = \sigma^2(F) + gF_o^2$, $g = 0\cdot0002$; $(\Delta/\sigma)_{max} = 0\cdot461$, $\Delta\rho_{max} = 0\cdot644$, $\Delta\rho_{min} = -0\cdot391$ e Å^{-3}; atomic scattering factors were from *International Tables for X-ray Crystallography* (1974); *SHELXTL* computer program (Sheldrick, 1984). Atomic and equivalent isotropic thermal

parameters are given in Table 1. Bond lengths and angles are given in Table 2.* Fig. 1 shows the molecular structure and Fig. 2 the unit-cell packing.

Related literature. To our knowledge no other structures of a cationic cyclopentadienyl iron complex with similar substitution have been previously reported.

References

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 99, 149. Birmingham; Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Macklin, P. D. (1988). PhD dissertation, The Pennsylvania State University, University Park, PA 16802, USA.

Sheldrick, G. M. (1984). *SHELXTL Users Manual*, version 4.1. Nicolet XRD Corp., Madison, WI, USA.

Structure of Bis(imidazole)(5,10,15,20-tetraphenylporphinato)iron(III) Bis(*cis*-1,2-dicyanoethylenedithiolato)cuprate(III) Tetrakis(tetrahydrofuran) Solvate

By Barbara R. Serr, Christine E. L. Headford, C. Michael Elliott and Oren P. Anderson*

Department of Chemistry, Colorado State University, Fort Collins, Colorado 80523, USA

Abstract. $[Fe(C_{44}H_{28}N_4)(C_3H_4N_2)_2][Cu(C_4N_2S_2)_2]\cdot 4C_4H_8O$, $M_r = 1437\cdot1$, triclinic, $P\bar{1}$, $a = 10\cdot012$ (4), $b = 11\cdot604$ (6), $c = 15\cdot802$ (7) Å, $\alpha = 71\cdot42$ (4), $\beta = 87\cdot12$ (3), $\gamma = 78\cdot45$ (4)°, $V = 1704$ (1) Å^3, $Z = 1$, $D_x = 1\cdot40$ g cm^{-3}, $\lambda(Mo\ K\alpha) = 0\cdot7107$ Å, $\mu = 6\cdot9$ cm^{-1}, $F(000) = 747$, $T = 143$ K, $R = 0\cdot063$ for 4935 unique observed reflections. Fe is six-coordinate, Fe—N(porphinato) (av.) = 1·99 (2), Fe—N(imidazole) = 1·981 (3) Å; Cu—S (av.) = 2·172 (8) Å.

Experimental. Fe(tpp) (Collman *et al.*, 1980) (0·025 g, 0·037 mmol) (tpp^{2-} = 5,10,15,20-tetraphenylporphinate) dissolved in 35 mL tetrahydrofuran. Solution of (tba)[Cu(mnt)$_2$] (Muetterties, 1961) (0·022 g, 0·037 mmol) (mnt^{2-} = *cis*-1,2-dicyanoethylenedithiolate, tba$^+$ = tetra-*n*-butylammonium) in 5 mL tetrahydrofuran added; reaction

mixture stirred for 3 h. Solution of imidazole (0·0025 g, 0·37 mmol) in 5 mL tetrahydrofuran added; vapor diffusion of *n*-hexane into the reaction solution yielded dark rectangular crystals.

Crystal size 0·32 × 0·32 × 0·18 mm. Nicolet *R3m* diffractometer, unit-cell constants from least-squares fit of setting angles for 25 reflections ($2\theta_{av} = 22\cdot59°$). Data collected (Wyckoff ω scans) to $(\sin\theta)/\lambda$ of 0·595 Å^{-1}, $-12 < h < 12$, $-14 < k < 14$, $0 < l < 19$. Three standard reflections (400, 070, 008) every 100, no significant variation; data corrected for Lorentz and polarization factors; no absorption correction applied due to low absorption coefficient; 6229 unique reflections, 4935 reflections with $F > 2\cdot5\sigma(F)$ observed, $R_{int} = 0\cdot0325$.

Structure solved by direct methods (*RANT*); block-diagonal (max. 103 parameters/block, 436 parameters total, data/parameters = 11·3) weighted $\{w = [\sigma^2(F) + gF^2]^{-1}$, $g = 0\cdot0106\}$ least-squares refinement on *F*. H atoms in idealized positions

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *for* [Fe(tpp)(Him)₂][Cu(mnt)₂].4C₄H₈O

E.s.d.'s are in parentheses.

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Fe	0·5000	0·5000	0·5000	0·017 (1)
Cu	0·5000	0·5000	1·0000	0·032 (1)
N(1)	0·6045 (3)	0·3404 (3)	0·4865 (2)	0·021 (1)
N(2)	0·3474 (3)	0·4973 (3)	0·4258 (2)	0·018 (1)
N(3)	0·4135 (3)	0·4096 (3)	0·6100 (2)	0·020 (1)
N(4)	0·3848 (4)	0·2714 (3)	0·7378 (2)	0·029 (1)
N(5)	0·2722 (5)	0·8090 (4)	1·1947 (3)	0·052 (2)
N(6)	−0·0146 (4)	0·6340 (5)	1·1273 (3)	0·056 (2)
S(1)	0·5015 (1)	0·6183 (1)	1·0818 (1)	0·048 (1)
S(2)	0·2858 (1)	0·4918 (1)	1·0254 (1)	0·040 (1)
O(1)	0·9410 (13)	0·2317 (7)	0·1415 (5)	0·200 (6)
O(2)	0·3708 (4)	0·0751 (4)	0·8955 (2)	0·059 (2)
C(1)	0·7338 (4)	0·2795 (4)	0·5177 (3)	0·021 (1)
C(2)	0·7752 (4)	0·1758 (4)	0·4859 (3)	0·025 (1)
C(3)	0·6716 (4)	0·1729 (4)	0·4359 (3)	0·026 (1)
C(4)	0·5656 (4)	0·2754 (4)	0·4359 (3)	0·022 (1)
C(5)	0·4448 (4)	0·3073 (4)	0·3863 (3)	0·022 (1)
C(6)	0·4217 (4)	0·2245 (4)	0·3353 (3)	0·022 (1)
C(7)	0·4364 (5)	0·2598 (4)	0·2432 (3)	0·032 (2)
C(8)	0·4193 (5)	0·1827 (4)	0·1952 (3)	0·039 (2)
C(9)	0·3874 (5)	0·0690 (4)	0·2384 (3)	0·035 (2)
C(10)	0·3728 (5)	0·0325 (4)	0·3302 (3)	0·038 (2)
C(11)	0·3899 (5)	0·1090 (4)	0·3781 (3)	0·031 (2)
C(12)	0·3446 (4)	0·4109 (4)	0·3822 (3)	0·022 (1)
C(13)	0·2184 (4)	0·4428 (4)	0·3329 (3)	0·025 (1)
C(14)	0·1454 (4)	0·5455 (4)	0·3463 (3)	0·025 (1)
C(15)	0·2240 (4)	0·5805 (4)	0·4043 (3)	0·019 (1)
C(16)	0·1842 (4)	0·6842 (4)	0·4314 (3)	0·021 (1)
C(17)	0·0446 (4)	0·7596 (4)	0·4055 (3)	0·022 (1)
C(18)	0·0243 (5)	0·8750 (4)	0·3401 (3)	0·035 (2)
C(19)	−0·1062 (5)	0·9442 (4)	0·3176 (4)	0·046 (2)
C(20)	−0·2175 (5)	0·8987 (4)	0·3596 (3)	0·037 (2)
C(21)	−0·1994 (5)	0·7848 (5)	0·4250 (3)	0·037 (2)
C(22)	−0·0700 (5)	0·7159 (4)	0·4481 (3)	0·034 (2)
C(23)	0·4681 (4)	0·3030 (4)	0·6687 (3)	0·027 (1)
C(24)	0·2712 (5)	0·3619 (4)	0·7232 (3)	0·032 (2)
C(25)	0·2884 (4)	0·4477 (4)	0·6444 (3)	0·031 (2)
C(26)	0·3300 (5)	0·6504 (4)	1·1088 (3)	0·036 (2)
C(27)	0·2945 (5)	0·7380 (5)	1·1575 (3)	0·042 (2)
C(28)	0·2386 (5)	0·5955 (4)	1·0853 (3)	0·033 (2)
C(29)	0·0967 (5)	0·6181 (5)	1·1091 (3)	0·042 (2)
C(30)	1·0320 (11)	0·2671 (11)	0·2068 (9)	0·163 (8)
C(31)	0·8225 (9)	0·2456 (6)	0·1741 (4)	0·070 (3)
C(32)	0·8034 (7)	0·3367 (7)	0·2163 (4)	0·068 (3)
C(33)	0·9358 (6)	0·3210 (7)	0·2580 (5)	0·080 (3)
C(34)	0·3213 (9)	0·1136 (8)	0·9727 (4)	0·093 (4)
C(35)	0·1905 (8)	0·0746 (6)	0·9997 (4)	0·072 (3)
C(36)	0·1640 (7)	0·0103 (8)	0·9365 (5)	0·083 (4)
C(37)	0·2998 (6)	−0·0211 (6)	0·8986 (4)	0·062 (3)

[C—H = 0·96 Å, $U(\text{H}) = 1\cdot2 \times U_{iso}(\text{C})$].   Non-H atoms refined anisotropically. At convergence [$(\Delta/\sigma)_{max} = 0\cdot04$, $(\Delta/\sigma)_{mean} = 0\cdot006$ for last six cycles] $R = 0\cdot0634$, $wR = 0\cdot0835$, $S = 1\cdot75$, slope of normal probability plot = 1·40, $\Delta\rho_{max} = 0\cdot83$, $\Delta\rho_{min} = -0\cdot61$ e Å⁻³. Neutral-atom scattering factors and anomalous-dispersion corrections used (*International Tables for X-ray Crystallography*, 1974); all calculations performed using *SHELXTL* program library (Sheldrick, 1983). Table 1 gives atomic coordinates,

Table 2. *Selected bond lengths* (Å) *and bond angles* (°)

E.s.d.'s are in parentheses.

Fe—N(1)	2·006 (3)	C(15)—C(16)	1·382 (6)
Fe—N(2)	1·983 (3)	C(16)—C(1')	1·384 (7)
Fe—N(3)	1·981 (3)	N(3)—C(23)	1·318 (4)
N(1)—C(1)	1·376 (5)	N(3)—C(25)	1·387 (5)
N(1)—C(4)	1·376 (6)	N(4)—C(23)	1·342 (5)
N(2)—C(12)	1·390 (6)	N(4)—C(24)	1·358 (5)
N(2)—C(15)	1·389 (4)	C(24)—C(25)	1·352 (5)
C(1)—C(2)	1·426 (6)	Cu—S(1)	2·167 (2)
C(2)—C(3)	1·347 (7)	Cu—S(2)	2·177 (1)
C(3)—C(4)	1·427 (5)	S(1)—C(26)	1·744 (5)
C(4)—C(5)	1·395 (6)	S(2)—C(28)	1·737 (5)
C(5)—C(12)	1·390 (5)	C(26)—C(27)	1·441 (8)
C(12)—C(13)	1·438 (6)	C(27)—N(5)	1·138 (8)
C(13)—C(14)	1·340 (6)	C(28)—C(29)	1·445 (7)
C(14)—C(15)	1·436 (7)	C(29)—N(6)	1·130 (7)
		C—C(phenyl, av.)	1·38 (1)
N(1)—Fe—N(2)	91·2 (1)	C(12)—C(13)—C(14)	107·7 (4)
N(1)—Fe—N(3)	91·3 (1)	C(13)—C(14)—C(15)	107·6 (4)
N(2)—Fe—N(3)	90·9 (1)	C(14)—C(15)—C(16)	124·7 (3)
Fe—N(1)—C(1)	128·1 (3)	C(15)—C(16)—C(1')	123·5 (3)
Fe—N(1)—C(4)	126·2 (2)	N(3)—C(23)—N(4)	110·9 (3)
Fe—N(2)—C(12)	126·1 (2)	C(23)—N(4)—C(24)	107·9 (3)
Fe—N(2)—C(15)	128·3 (3)	N(4)—C(24)—C(25)	106·6 (4)
Fe—N(3)—C(23)	126·4 (3)	N(3)—C(25)—C(24)	108·9 (3)
Fe—N(3)—C(25)	127·8 (2)	C(23)—N(3)—C(25)	105·7 (3)
N(1)—C(1)—C(2)	110·0 (4)	S(1)—Cu—S(2)	92·8 (1)
N(1)—C(1)—C(16')	125·6 (4)	Cu—S(1)—C(26)	102·2 (2)
N(1)—C(4)—C(3)	110·2 (4)	Cu—S(2)—C(28)	102·0 (2)
N(1)—C(4)—C(5)	126·1 (4)	S(1)—C(26)—C(27)	115·9 (4)
C(1)—C(2)—C(3)	107·3 (3)	S(1)—C(26)—C(28)	121·0 (4)
C(2)—C(3)—C(4)	107·0 (4)	S(2)—C(28)—C(26)	121·4 (4)
C(3)—C(4)—C(5)	123·6 (4)	S(2)—C(28)—C(29)	116·6 (4)
C(5)—C(12)—C(13)	124·1 (4)	C(26)—C(27)—N(5)	176·7 (6)
N(2)—C(12)—C(5)	126·5 (4)	C(26)—C(28)—C(29)	122·0 (5)
N(2)—C(12)—C(13)	109·4 (3)	C(28)—C(26)—C(27)	123·0 (5)
N(2)—C(15)—C(14)	109·6 (4)	C(28)—C(29)—N(6)	178·8 (7)
N(2)—C(15)—C(16)	125·6 (4)	C—C—C(phenyl, av.)	120 (1)

Table 2 gives selected bond lengths and angles.* Fig 1 shows the structures of the [Fe(tpp)(Him)₂]⁺ cation (Him = imidazole) and the [Cu(mnt)₂]⁻ anion, as well as the numbering scheme used.

Related literature. The synthetic scheme was designed to allow redox reaction between Fe^{II}(tpp) and Cu^{III}(mnt)₂⁻; such reactions, in the presence of weak neutral bases, have yielded novel sulfur-bridged heterodinuclear iron(III)–copper(II) compounds (Serr, Headford, Elliott, Anderson, Spartalian & Hatfield, 1989). In the present case, however, the structure determination revealed that the added imidazole base displaced the sulfur bridge to yield the title compound. The structure consists of a discrete [Fe^{III}(tpp)(Him)₂]⁺ cation and an unreduced [Cu^{III}(mnt)₂]⁻ anion. Of the two occluded tetrahydrofuran molecules in the asymmetric unit, one is
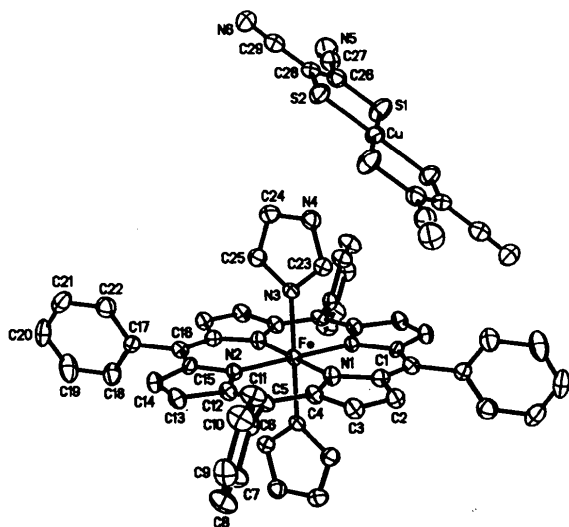
Fig. 1. The structure of the $[Fe(tpp)(Him)_2]^+$ cation and the $[Cu(mnt)_2]^-$ anion (50% probability thermal. ellipsoids). H atoms have been omitted for clarity.

between imidazole orientation and Fe spin state has been discussed (Scheidt & Chipman, 1986).

hydrogen bonded to the coordinated imidazole; in the other, O(1) and C(30) are subject to unresolvable disorder, which gives rise to high thermal parameters for those two atoms.

A number of related compounds have been studied: (tba)[Cu(mnt)$_2$] (Forrester, Zalkin & Templeton, 1964); [Fe(tpp)(Him)$_2$]Cl (Collins, Countryman & Hoard, 1972); [Fe(oep)(2-MeHim)$_2$]ClO$_4$ (Geiger, Lee & Scheidt, 1984; oep = 2,3,7,8,12,13,17,18-octaethylporphinate); [Fe(tpp)(2-MeHim)$_2$]ClO$_4$ (Kirner & Hoard, 1978); {K(cryptand[2.2.2])}-[Fe(tpp)(4-MeHim)$_2$] (Quinn, Strouse & Valentine, 1983); [Fe(tpp)(1-Meim)$_2$] (Steffen, Chun & Hoard, 1978); [Fe(tpp)(Hbim)$_2$]ClO$_4$ (Levan & Strouse, 1983; Hbim = benzimidazole). The metric parameters for the title compound suggest that Fe is low spin (Scheidt & Reed, 1981). The relationship

References

Collins, D. M., Countryman, R. & Hoard, J. L. (1972). *J. Am. Chem. Soc.* **94**, 2066–2072.

Collman, J. P., Brauman, J. D., Doxsee, K. M., Halber, T. R., Bunnenber, E., Linder, R. E., Lamar, G. N., Grandio, J. D., Lang, G. & Spartalian, K. (1980). *J. Am. Chem. Soc.* **102**, 4182–4192.

Forrester, J. D., Zalkin, A. & Templeton, D. H. (1964). *Inorg. Chem.* **3**, 1507–1515.

Geiger, D. K., Lee, Y. J. & Scheidt, W. R. (1984). *J. Am. Chem. Soc.* **106**, 6339–6343.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Kirner, J. F. & Hoard, J. L. (1978). 175th Natl Meet. Am. Chem. Soc., Anaheim, California, USA, 13–17 March, Abstracts.

Levan, K. R. & Strouse, C. E. (1983). Am. Crystallogr. Assoc. Summer Meet., Snowmass, Colorado, USA, 1–5 August, Abstract H1.

Muetterties, E. L. (1961). Editor. *Inorganic Syntheses*, Vol. X, p 13. New York: McGraw-Hill.

Quinn, R., Strouse, C. E. & Valentine, J. S. (1983). *Inorg. Chem.* **22**, 3934–3940.

Scheidt, W. R. & Chipman, G. M. (1986). *J. Am. Chem. Soc.* **108**, 1163–1167.

Scheidt, W. R. & Reed, C. A. (1981). *Chem. Rev.* **81**, 543–555.

Serr, B. R., Headford, C. E. L., Elliott, C. M., Anderson, O. P., Spartalian, K. & Hatfield, W. E. (1989). In preparation.

Sheldrick, G. M. (1983). *SHELXTL*. Revision 5. Nicolet XRD Corporation, Madison, Wisconsin, USA.

Steffen, W. L., Chun, H. K. & Hoard, J. L. (1978). 175th Natl Meet. Am. Chem. Soc., Anaheim, California, USA, 13–17 March, Abstracts.

Methyl 2-[(2,6-Dimethoxyphenyl)ethynyl]-3-methoxybenzoate

By Kevin L. Evans, Gregory W. Horn, Frank R. Fronczek and Richard D. Gandour*

Department of Chemistry, Louisiana State University, Baton Rouge, LA 70803-1804, USA

Abstract. $C_{19}H_{18}O_5$, $M_r = 326.4$, orthorhombic, $Pna2_1$, $a = 19.071$ (2), $b = 7.7090$ (11), $c = 11.3771$ (12) Å, $V = 1672.6$ (6) Å^3, $Z = 4$, $D_x = 1.296$ g cm^{-3} at 298 (1) K, $\lambda(Cu\ K\alpha) = 1.54184$ Å, $\mu = 7.36$ cm^{-1}, $F(000) = 688$, 1811 unique data measured, final $R = 0.031$ for 1745 reflections with $I > 3.0\sigma(I)$. Maximum deviations from planarity of the two aromatic rings are 0.0149 (13) Å for the ring

* To whom correspondence should be addressed.