Cs(1), the six-coordination comprises the two waters, both 'inner' carboxylate oxygens [O(101) and O(102)] and the oxygens of two bridging carboxylate groups [O(111) and O(111)′]. The two caesium ions are separated by 4·093 (2) Å. This system of bonds accounts for the stability of the crystal structure, despite the inability of the metal ion to fit the ionophore cavity. The structure is also stabilized by interpolymer hydrogen-bonding interactions involving the water molecules and carboxylate oxygens, O*w*(1)···O(101), 2·94 Å ($\frac{1}{2}-x$, $-y$, $-\frac{1}{2}+z$); O*w*(1)···O(102), 2·79 Å ($\frac{1}{2}-x$, $-y$, $-1\frac{1}{2}+z$); O*w*(2)···O(111), 2·77 Å (x, $-\frac{1}{2}-y$, $-\frac{1}{2}+z$), O*w*(2)···O(102), 2·97 Å ($\frac{1}{2}-x$, $\frac{1}{2}+y$, $-2+z$). The bdda ligand itself is essentially planar [torsion angles C(1)—C(2)—O(72)—C(82), C(2)—O(72)—C(82)—C(92), C(72)—C(82)—C(92)—O(102), −179, −178, +8 (1)°; C(2)—C(1)—O(72)—C(81), C(1)—O(71)—C(81)—C(91), O(71)—C(81)—C(91)—O(101), −180, +177, −9 (1)°, respectively], consistent with observed conformations in both univalent and divalent bdda complexes (coordinated) and in ionic $(NH_4^+)_2(bdda)^{2-}$ (Kennard *et al.*, 1988). Fig. 2 is a view of the packing in the unit cell.

The authors thank the Chinese University of Hong Kong for the use of data collection facilities, the Queensland University of Technology and the University of Queensland for financial support.

References

GREEN, E. A., DUAX, W. L., SMITH, G. M. & WUDL, F. (1975). *J. Am. Chem. Soc.* **97**, 6689–6692.

IBERS, J. A. & HAMILTON, W. C. (1974). Editors. *International Tables for X-ray Crystallography*, Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

KENNARD, C. H. L., SMITH, G., O'REILLY, E. J., REYNOLDS, B. J. & MAK, T. C. W. (1988). *J. Chem. Soc. Dalton Trans.* pp. 2357–2362.

SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

SMITH, G., O'REILLY, E. J. & KENNARD, C. H. L. (1986). *Acta Cryst.* C**42**, 1329–1331.

SMITH, G., O'REILLY, E. J. & KENNARD, C. H. L. (1987). *Polyhedron*, **6**, 871–789.

Acta Cryst. (1989). C**45**, 1733–1736

Structure of $[N(C_4H_9)_4]_2[Fe(C_3H_7NO)_6][Fe_4Br_4S_4]_2$

BY AIDEEN C. M. YOUNG, MARC A. WALTERS AND JOHN C. DEWAN

Department of Chemistry, New York University, New York, NY 10003, *USA*

(*Received* 27 *October* 1988; *accepted* 14 *March* 1989)

Abstract. Bis(tetrabutylammonium) hexa(dimethylaminoformaldehyde)iron(2+) bis[tetrabromotetrakis-μ_3-thio-tetraferrate(2−)], $[N(C_4H_9)_4]_2[Fe(C_3H_7NO)_6][Fe_4Br_4S_4]_2$, M_r = 2321·8, monoclinic, $P2_1/n$, a = 10·840 (6), b = 19·860 (10), c = 21·180 (7) Å, β = 104·24 (4)°, V = 4420 (7) Å³, Z = 2, D_x = 1·745 g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ = 52·4 cm^{-1}, $F(000)$ = 2320, T = 203 K, final R = 0·050 for 5119 unique observed reflections with $I_o > 2\sigma(I_o)$. The $[Fe(C_3H_7NO)_6]^{2+}$ cation has crystallographically imposed $\bar{1}$ symmetry and the three unique Fe^{II}—O(DMF) distances are 2·092 (6), 2·142 (7) and 2·150 (8) Å. The Fe—S distances in $[Fe_4S_4Br_4]^{2-}$ range from 2·266 (2) to 2·284 (3) Å with the average being 2·275 Å. Similar values for Fe—Br are 2·326 (2), 2·346 (2) and 2·339 Å.

Introduction. The iron–sulfur cubane-like core $[Fe_4S_4(SR)_4]^{2-}$ has been the subject of numerous studies concerned with the active sites of ferredoxins, nitrogenase and hydrogenase (Berg & Holm, 1982). We, and others, have recently shown that the complex $[Fe_4S_4Br_4]^{2-}$ can form from $FeBr_3$ and Na_2S in DMF solution *via* a self-assembly process that involves a sequence of dimerization and redox reactions (Rutchik, Kim & Walters, 1988; Müller & Schladerbeck, 1985; Müller, Schladerbeck & Bögge, 1985). We have attempted to utilize this type of reaction to synthesize heterobimetallic complexes. The compound reported here is an unanticipated product from that work.

Experimental. The title compound resulted from an attempt to synthesize a heteronuclear bimetallic compound containing Fe, Cl, Mn, S, CO and Bu_4N^+ in DMF. When the reaction solution was concentrated to minimum volume and left standing under a nitrogen atmosphere for several days a black crystalline product was obtained. This has been determined by X-ray crystallographic analysis to be an Fe–S

0108-2701/89/111733-04$03.00

cubane-like compound and is the subject of this report. The Mn, initially present in the reaction mixture as $Mn(CO)_5Br$, was not incorporated into this product which was the only solid isolated from the reaction mixture. Full details of the synthesis will be reported at a later date.

Details of the crystallography are as follows. Black crystal, dimensions 0·27 × 0·30 × 0·34 mm, monoclinic, space group $P2_1/n$ (No. 14), Rigaku AFC-6S four-circle diffractometer, liquid-nitrogen low-temperature device run at 203 K to prevent crystal from drying out. The crystal to detector distance was 285 mm, monochromated Mo $K\alpha$ radiation was employed, $\omega/2\theta$ scan technique, variable scan width where $\Delta\omega = (1·15 + 0·30\tan\theta)°$, scan rate 4° $\min^{-1}$ in ω. Weak reflections with $F < 10·0\sigma(F)$ were rescanned a maximum of two times and counts accumulated, stationary background counts were recorded at each side of the reflections. The ratio of peak: background counting time was 2:1. Lattice parameters were from 25 reflections having $30 < 2\theta < 35°$. An empirical absorption correction was applied using *DIFABS* (Walker & Stuart, 1983), transmission factors ranged from 0·84 to 1·22, three intensity standards measured every 150 data showed no decay. The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1985) followed by difference-Fourier syntheses. A total of 8017 unique reflections was measured to $2\theta = 50°$ (h 0→12, k 0→23, l −25 →24). The 5119 having $I_o > 2\sigma(I_o)$ were used in the structure refinement which was by full-matrix least squares on F (403 variables) using the *TEXSAN* crystallographic software package (Molecular Structure Corporation, 1985). The final data to parameter ratio was 12·7. All non-H atoms were refined anisotropically, methyl H atoms of the DMF ligands could not be located in final difference-Fourier maps and were ignored, other H atoms were placed in calculated positions (C—H = 0·95 Å) and assigned a temperature factor of 1·2 times B_{eq} of the atom to which they were bonded. Neutral-atom scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). Final $R = 0·050$, $wR = 0·060$, GOF = 1·45. Weights given by $w = 4F_o^2/\sigma^2(F_o^2)$. In the final cycle the maximum LS shift/e.s.d. was 0·02. The final difference-Fourier map maximum and minimum peaks were 0·96 and $-0·94$ e Å^{-3}, respectively. Table 1* gives atomic positional parameters, Table 2 bond lengths and angles. Fig. 1 shows the molecular geometry and atom-labeling scheme of the $[Fe_4Br_4S_4]^{2-}$ and $[Fe(C_3H_7NO)_6]^{2+}$ ions.

Table 1. *Atom coordinates and equivalent isotropic temperature factors* (Å^2)

$$B_{eq} = \tfrac{8}{3}\pi^2\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Fe(1)	0·6076 (1)	0·28250 (6)	0·57030 (5)	2·32 (5)
Fe(2)	0·4649 (1)	0·19682 (6)	0·62553 (5)	2·43 (5)
Fe(3)	0·5377 (1)	0·31945 (6)	0·68161 (5)	2·41 (5)
Fe(4)	0·7153 (1)	0·22041 (6)	0·68708 (5)	2·51 (5)
Fe(5)	0·5	0·0	1·0	2·90 (7)
Br(1)	0·6441 (1)	0·32077 (5)	0·47176 (4)	3·59 (4)
Br(2)	0·30236 (9)	0·11582 (5)	0·60374 (5)	4·01 (4)
Br(3)	0·47747 (8)	0·40682 (5)	0·74177 (4)	3·61 (4)
Br(4)	0·9033 (1)	0·17853 (7)	0·75347 (6)	6·31 (6)
S(1)	0·6387 (2)	0·1702 (1)	0·5884 (1)	2·80 (9)
S(2)	0·5475 (2)	0·2183 (1)	0·7331 (1)	2·81 (9)
S(3)	0·7332 (2)	0·3308 (1)	0·6612 (1)	2·74 (8)
S(4)	0·4071 (2)	0·3006 (1)	0·5816 (1)	2·64 (8)
O(21)	0·3590 (7)	0·0372 (4)	0·9181 (4)	5·3 (4)
O(31)	0·6466 (6)	0·0278 (3)	0·9570 (3)	4·5 (3)
O(41)	0·5127 (7)	0·0992 (4)	1·0418 (4)	5·6 (4)
N(11)	0·3707 (5)	0·0912 (3)	0·3774 (3)	2·1 (3)
N(21)	0·2761 (7)	0·1028 (4)	0·8338 (4)	3·7 (3)
N(31)	0·8299 (7)	0·0277 (4)	0·9242 (3)	3·5 (3)
N(41)	0·5695 (7)	0·1835 (4)	1·1118 (4)	3·9 (4)
C(11)	0·3304 (7)	0·0421 (4)	0·4224 (3)	2·6 (3)
C(12)	0·1958 (8)	0·0156 (5)	0·4010 (4)	3·6 (4)
C(13)	0·1649 (8)	−0·0267 (5)	0·4545 (5)	3·6 (4)
C(14)	0·038 (1)	−0·0627 (6)	0·4317 (6)	5·9 (6)
C(15)	0·3517 (7)	0·0602 (4)	0·3102 (3)	2·4 (3)
C(16)	0·419 (1)	−0·0056 (5)	0·3079 (4)	3·7 (4)
C(17)	0·411 (1)	−0·0247 (6)	0·2378 (5)	5·3 (5)
C(18)	0·492 (1)	0·0195 (7)	0·2068 (5)	7·1 (7)
C(19)	0·5099 (7)	0·1074 (4)	0·4066 (4)	2·6 (3)
C(110)	0·5727 (8)	0·1562 (5)	0·3690 (4)	3·5 (4)
C(111)	0·7152 (8)	0·1546 (5)	0·3951 (5)	4·2 (4)
C(112)	0·7765 (9)	0·0928 (6)	0·3757 (5)	5·2 (5)
C(113)	0·2914 (7)	0·1551 (4)	0·3681 (4)	2·5 (3)
C(114)	0·2982 (8)	0·1964 (5)	0·4290 (4)	3·5 (4)
C(115)	0·187 (1)	0·2429 (5)	0·4201 (4)	4·0 (4)
C(116)	0·063 (1)	0·2080 (6)	0·4184 (5)	5·6 (6)
C(21)	0·355 (1)	0·0872 (6)	0·8919 (6)	5·3 (6)
C(22)	0·284 (1)	0·1708 (6)	0·8117 (6)	5·9 (6)
C(23)	0·187 (1)	0·0599 (6)	0·7946 (8)	9·0 (8)
C(31)	0·7412 (9)	−0·0013 (5)	0·9485 (4)	3·6 (4)
C(32)	0·938 (1)	−0·0101 (6)	0·9140 (5)	5·3 (5)
C(33)	0·817 (1)	0·0989 (5)	0·9049 (6)	5·5 (5)
C(41)	0·576 (1)	0·1211 (7)	1·0888 (6)	5·8 (6)
C(42)	0·493 (1)	0·2372 (6)	1·0789 (5)	5·5 (6)
C(43)	0·654 (1)	0·1983 (7)	1·1754 (5)	5·9 (6)

Discussion. The structure (Fig. 1) is composed of $[N(C_4H_9)_4]^+$ cations, $[Fe(DMF)_6]^{2+}$ complex cations and $[Fe_4Br_4S_4]^{2-}$ iron–sulfur clusters in a 2:1:2 ratio.

A brief account of the structure of the $[Fe(DMF)_6]^{2+}$ dication, in the compound $[Fe(DMF)_6][Cl_2Fe(WS_4)]$, has been given but a full structure report has not appeared (Stremple, Baenziger & Coucouvanis, 1981). The synthesis of $[Fe(DMF)_6](ClO_4)_2$ has been reported (Gritzner, Linert & Gutmann, 1981). The Fe^{II} ion of the $[Fe(DMF)_6]^{2+}$ dication in the present structure is situated at a point of $\bar{1}$ symmetry and the dication therefore has this symmetry crystallographically imposed upon it. The three Fe—O(DMF) distances are 2·092 (6), 2·142 (7) and 2·150 (8) Å. The shorter distance in the present complex is comparable to the 2·082 (4) Å found in $(Ph_4P)_2[Fe^{II}(WS_4)_2(DMF)_2]$ (Stremple, Baenziger &

---

* Lists of anisotropic thermal parameters, structure factors, H-atom parameters, bond distances and angles involving H atoms, and intermolecular distances and a complete description of the structure determination have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52044 (91 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Interatomic distances* (Å) *and angles* (°) *involving the non*-H *atoms*

Fe(1)—S(4)	2·273 (3)	N(11)—C(113)	1·52 (1)
Fe(1)—S(1)	2·274 (3)	N(21)—C(21)	1·35 (1)
Fe(1)—S(3)	2·279 (3)	N(21)—C(23)	1·40 (1)
Fe(1)—Br(1)	2·344 (2)	N(21)—C(22)	1·44 (1)
Fe(2)—S(2)	2·274 (2)	N(31)—C(31)	1·33 (1)
Fe(2)—S(1)	2·274 (2)	N(31)—C(32)	1·45 (1)
Fe(2)—S(4)	2·284 (3)	N(31)—C(33)	1·47 (1)
Fe(2)—Br(2)	2·346 (2)	N(41)—C(41)	1·34 (1)
Fe(3)—S(4)	2·271 (3)	N(41)—C(42)	1·43 (1)
Fe(3)—S(2)	2·275 (3)	N(41)—C(43)	1·46 (1)
Fe(3)—S(3)	2·276 (3)	C(11)—C(12)	1·51 (1)
Fe(3)—Br(3)	2·339 (2)	C(12)—C(13)	1·51 (1)
Fe(4)—S(2)	2·266 (2)	C(13)—C(14)	1·51 (1)
Fe(4)—S(3)	2·279 (3)	C(15)—C(16)	1·50 (1)
Fe(4)—S(1)	2·280 (3)	C(16)—C(17)	1·52 (1)
Fe(4)—Br(4)	2·326 (2)	C(17)—C(18)	1·50 (2)
Fe(5)—O(31)	2·092 (6)	C(19)—C(110)	1·52 (1)
Fe(5)—O(31')	2·092 (6)	C(110)—C(111)	1·51 (1)
Fe(5)—O(21)	2·142 (7)	C(111)—C(112)	1·50 (1)
Fe(5)—O(21')	2·142 (7)	C(113)—C(114)	1·51 (1)
Fe(5)—O(41)	2·150 (8)	C(114)—C(115)	1·49 (1)
Fe(5)—O(41')	2·150 (8)	C(115)—C(116)	1·51 (1)
O(21)—C(21)	1·13 (1)	Fe(1)···Fe(2)	2·747 (2)
O(31)—C(31)	1·23 (1)	Fe(1)···Fe(3)	2·748 (2)
O(41)—C(41)	1·15 (1)	Fe(1)···Fe(4)	2·753 (2)
N(11)—C(11)	1·50 (1)	Fe(2)···Fe(3)	2·739 (2)
N(11)—C(19)	1·52 (1)	Fe(2)···Fe(4)	2·747 (2)
N(11)—C(15)	1·518 (9)	Fe(3)···Fe(4)	2·735 (2)

S(4)—Fe(1)—S(1)	104·04 (9)	Fe(3)—S(3)—Fe(1)	74·22 (8)
S(4)—Fe(1)—S(3)	103·45 (9)	Fe(3)—S(3)—Fe(4)	73·80 (7)
S(4)—Fe(1)—Br(1)	114·68 (8)	Fe(1)—S(3)—Fe(4)	74·33 (8)
S(1)—Fe(1)—S(3)	103·7 (1)	Fe(3)—S(4)—Fe(1)	74·42 (8)
S(1)—Fe(1)—Br(1)	114·71 (7)	Fe(3)—S(4)—Fe(2)	73·94 (8)
S(3)—Fe(1)—Br(1)	114·80 (8)	Fe(1)—S(4)—Fe(2)	74·14 (8)
S(2)—Fe(2)—S(1)	103·6 (1)	C(21)—O(21)—Fe(5)	129 (1)
S(2)—Fe(2)—S(4)	103·9 (1)	C(31)—O(31)—Fe(5)	133·7 (6)
S(2)—Fe(2)—Br(2)	114·68 (8)	C(41)—O(41)—Fe(5)	132 (1)
S(1)—Fe(2)—S(4)	103·68 (9)	C(11)—N(11)—C(19)	107·1 (6)
S(1)—Fe(2)—Br(2)	114·73 (8)	C(11)—N(11)—C(15)	109·8 (6)
S(4)—Fe(2)—Br(2)	114·89 (8)	C(11)—N(11)—C(113)	112·5 (6)
S(4)—Fe(3)—S(2)	104·3 (1)	C(19)—N(11)—C(15)	111·1 (5)
S(4)—Fe(3)—S(3)	103·61 (9)	C(19)—N(11)—C(113)	110·5 (6)
S(4)—Fe(3)—Br(3)	115·93 (8)	C(15)—N(11)—C(113)	106·0 (5)
S(2)—Fe(3)—S(3)	104·02 (9)	C(21)—N(21)—C(23)	126 (1)
S(2)—Fe(3)—Br(3)	112·36 (8)	C(21)—N(21)—C(22)	116 (1)
S(3)—Fe(3)—Br(3)	115·31 (8)	C(23)—N(21)—C(22)	118 (1)
S(2)—Fe(4)—S(3)	104·22 (9)	C(31)—N(31)—C(32)	121·4 (8)
S(2)—Fe(4)—S(1)	103·6 (1)	C(31)—N(31)—C(33)	119·8 (8)
S(2)—Fe(4)—Br(4)	114·22 (8)	C(32)—N(31)—C(33)	118·8 (8)
S(3)—Fe(4)—S(1)	103·52 (9)	C(41)—N(41)—C(42)	126 (1)
S(3)—Fe(4)—Br(4)	112·05 (8)	C(41)—N(41)—C(43)	116 (1)
S(1)—Fe(4)—Br(4)	117·74 (9)	C(42)—N(41)—C(43)	117·4 (8)
O(31)—Fe(5)—O(31')	180	N(11)—C(11)—C(12)	116·4 (6)
O(31)—Fe(5)—O(21)	91·3 (3)	C(11)—C(12)—C(13)	110·1 (7)
O(31)—Fe(5)—O(21')	88·7 (3)	C(12)—C(13)—C(14)	111·9 (8)
O(31)—Fe(5)—O(41)	87·6 (3)	C(16)—C(15)—N(11)	115·3 (6)
O(31)—Fe(5)—O(41')	92·4 (3)	C(15)—C(16)—C(17)	109·7 (8)
O(21)—Fe(5)—O(21')	180	C(18)—C(17)—C(16)	113 (1)
O(21)—Fe(5)—O(41)	88·7 (3)	N(11)—C(19)—C(110)	116·8 (6)
O(21)—Fe(5)—O(41')	91·3 (3)	C(111)—C(110)—C(19)	110·2 (7)
O(41)—Fe(5)—O(41')	180	C(112)—C(111)—C(110)	113·5 (8)
Fe(1)—S(1)—Fe(2)	74·32 (7)	C(114)—C(113)—N(11)	115·8 (7)
Fe(1)—S(1)—Fe(4)	74·40 (8)	C(115)—C(114)—C(113)	111·1 (7)
Fe(2)—S(1)—Fe(4)	74·21 (8)	C(114)—C(115)—C(116)	114·0 (9)
Fe(4)—S(2)—Fe(2)	74·49 (8)	O(21)—C(21)—N(21)	126 (1)
Fe(4)—S(2)—Fe(3)	74·09 (7)	O(31)—C(31)—N(31)	124 (1)
Fe(2)—S(2)—Fe(3)	74·06 (8)	O(41)—C(41)—N(41)	127 (1)

Symmetry code: (i) $1 - x, -y, 2 - z$.

Coucouvanis, 1981) and the 2·07 (1) Å observed in the compound Fe^{II}(phthalocyanato)(CO)(DMF).-DMF (Calderazzo, Pampaloni, Vitali, Pelizzi, Collamati, Frediani & Serra, 1980) as well as the 2·098 (9) Å reported for the Fe^{II}—O(DMF) distance in $[Fe(DMF)_6][Cl_2Fe(WS_4)]$ (Stremple, Baenziger & Coucouvanis, 1981). The only other Fe—O(DMF) distance to be reported occurs in the Fe^{III} complex $Fe(DMF)(phenanthroline)Cl_3$ and, as might be expected, is shorter than all the reported Fe^{II}—O(DMF) distances at 2·03 (1) Å (Reiff, Witten, Mottle, Brennan & Garafalo, 1983).

The Fe—S distances in the $[Fe_4Br_4S_4]^{2-}$ cluster range from 2·266 (2) to 2·284 (3) Å and average 2·275 Å. This compares with the average Fe—S distance of 2·278 Å in $(Ph_4P)_2[Fe_4Br_4S_4]$ (Müller, Schladerbeck & Bögge, 1985). Each Fe atom in the cluster is bonded to one terminal Br^- ligand where the Fe—Br distances range from 2·326 (2) to 2·346 (2) Å and average 2·339 Å. This average is again similar to the 2·342 Å average seen in $(Ph_4P)_2$-$[Fe_4Br_4S_4]$. Distances within the remainder of the structure are as expected.

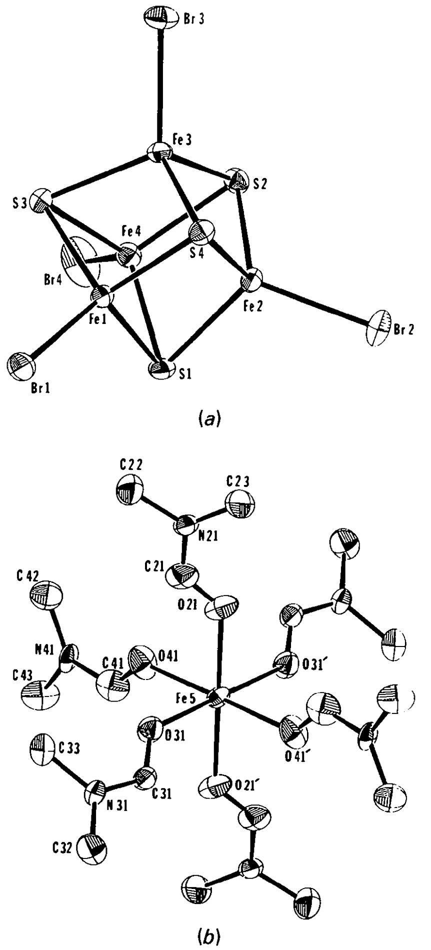

Fig. 1. *ORTEP*II diagram (Johnson, 1976) showing the (*a*) $[Fe_4Br_4S_4]^{2-}$ and (*b*) $[Fe(C_3H_7NO)_6]^{2+}$ ions of the structure along with the atom-labeling scheme and 30% probability thermal ellipsoids. Primed atoms of the cation are related to those unprimed by a center of symmetry at Fe(5). H atoms are omitted.

Funds to purchase the X-ray diffraction equipment were provided by NSF Grant CHE 87-11307.

References

BERG, J. M. & HOLM, R. H. (1982). *Iron–Sulfur Proteins*, Vol. 4, edited by T. G. SPIRO, pp. 1–66, and references therein. New York: John Wiley.

CALDERAZZO, F., PAMPALONI, G., VITALI, D., PELIZZI, G., COLLAMATI, I., FREDIANI, S. & SERRA, A. M. (1980). *J. Organomet. Chem.* **191**, 217–242.

GRITZNER, G., LINERT, W. & GUTMANN, V. (1981). *J. Inorg. Nucl. Chem.* **43**, 1193–1199.

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

JOHNSON, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Molecular Structure Corporation (1985). *TEXSAN–TEXRAY. Structure Analysis Package*. MSC, 3200A Research Forest Drive, The Woodlands, TX 77381, USA.

MÜLLER, A. & SCHLADERBECK, N. (1985). *Chimia*, **39**, 23–24.

MÜLLER, A., SCHLADERBECK, N. & BÖGGE, H. (1985). *Chimia*, **39**, 24–25.

REIFF, W. M., WITTEN, E. H., MOTTLE, K., BRENNAN, T. F. & GARAFALO, A. R. (1983). *Inorg. Chim. Acta*, **77**, L27–L30.

RUTCHIK, S., KIM, S. & WALTERS, M. A. (1988). *Inorg. Chem.* **27**, 1513–1515.

SHELDRICK, G. M. (1985). *SHELXS86*. In *Crystallographic Computing* 3, edited by G. M. SHELDRICK, C. KRÜGER & R. GODDARD, pp. 175–189. Oxford Univ. Press.

STREMPLE, P., BAENZIGER, N. C. & COUCOUVANIS, D. (1981). *J. Am. Chem. Soc.* **103**, 4601–4603.

WALKER, N. & STUART, D. (1983). *Acta Cryst.* A**39**, 158–166.

Acta Cryst. (1989). C**45**, 1736–1738

Structure of Ethyl 4-Methyl-2-oxo-3-phenyl-2,3-dihydro-5-benzofurancarboxylate

BY R. GHOSH AND P. ROYCHOWDHURY*

Department of Physics (X-ray Laboratory), University College of Science,
*92 Acharys Prafulla Chandra Road, Calcutta-*700 009, *India*

AND P. MAULIK

Biophysics Division, Department of Medicine, Boston Univeristy School of Medicine, Boston, MA 02118, *USA*

(*Received* 13 *February* 1989; *accepted* 29 *March* 1989)

Abstract. $C_{18}H_{16}O_4$, $M_r = 296{\cdot}322$, monoclinic, $C2/c$, $a = 22{\cdot}92\,(3)$, $b = 8{\cdot}06\,(2)$, $c = 22{\cdot}72\,(2)$ Å, $\beta = 133{\cdot}24\,(3)°$, $V = 3057{\cdot}61\,(3)$ Å^3, $Z = 8$, $D_m = 1{\cdot}30$, $D_x = 1{\cdot}29$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}7135$ Å, $\mu = 0{\cdot}85$ cm^{-1}, $F(000) = 1248{\cdot}0$, $T = 298\,(1)$ K, final $R = 0{\cdot}067$ for 1536 observed reflections. The structure has two phenyl rings (I,III), one of which (III) is fused to the furan ring (II). Ring I is perpendicular to III and nearly perpendicular to II, which is in a twist conformation. The C(5)—C(9) single bond is shortened [1·491 (10) Å] owing to the adjacent double bonds.

Introduction. The crystal and molecular structure of the title compound was solved as a part of our program of structure analyses of a group of benzofuran derivatives.

Experimental. Crystals from ethanol; density measured by flotation in benzene–bromoform, crystal size 0·35 × 0·45 × 0·45 mm; intensity data collected on an Enraf–Nonius CAD-4 diffractometer using graphite-monochromatized Mo $K\alpha$ radiation; cell parameters refined by least-squares fitting of the setting angles of 25 high-angle reflections ($14 \leq \theta \leq 18°$). 2087 independent reflections collected ($-21 \leq h \leq 23$, $0 \leq k \leq 9$, $-22 \leq l \leq 23$; $2 \leq \theta \leq 60°$), correction for Lp but not for absorption; space group $C2/c$ or Cc (hkl, $h+k$ odd; $h0l$, h and l odd; the space group was shown to be $C2/c$ by the structure analysis); structure solved by *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978); R value was 0·089 after full-matrix least-squares refinement using the program *SHELX*76 (Sheldrick, 1976) of all the non-H atoms with anisotropic thermal parameters; all the H atoms located from ΔF synthesis. Each H atom was assigned the isotropic temperature factor of the attached non-H atom; final two cycles of refinement of the positional and anisotropic thermal parameters of the non-H atoms keeping all the parameters of the H atoms fixed with 1536 observed reflections [$I \geq 2{\cdot}5\sigma(I)$] led to a final $R = 0{\cdot}067$, $S = 1{\cdot}88$, number of parameters refined = 199; $\Delta/\sigma < 0{\cdot}05$ in final refinement; residual $\Delta\rho = 0{\cdot}6$ e Å^{-3}. Atomic scattering factors from *Interna-*

0108-2701/89/111736-03$03.00