Journal of Organometallic Chemistry, 135 (1977) 351—360

REACTIONS OF METAL CARBONYL DERIVATIVES

XXI *. THE CRYSTAL AND MOLECULAR STRUCTURE OF μ-ETHYLTHIO-μ'-CARBONYLBIS(η-CYCLOPENTADIENYLCARBONYLIRON) (*Fe—Fe*) HEXAFLUOROANTIMONATE

ROBIN B. ENGLISH, LUIGI R. NASSIMBENI *,
Departments of Inorganic and Physical Chemistry, University of Cape Town, Private Bag, Rondebosch, Cape Town (Republic of South Africa)

and RAYMOND J. HAINES *
Department of Chemistry, University of Natal, Pietermaritzburg (Republic of South Africa)

(Received March 9th, 1977)

Summary

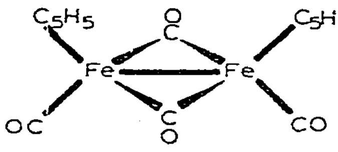
The solid state structure of μ-ethylthio-μ'-carbonylbis(η-cyclopentadienylcarbonyliron) hexafluoroantimonate has been determined by a single crystal X-ray analysis. Crystals of this compound are monoclinic, space group $c2/c$ with $a = 18.111(6)$, $b = 16.450(5)$, $c = 13.292(4)$ Å, $\beta = 92.2(1)°$, $Z = 8$. The determination was by Patterson and Fourier methods. A full matrix least-squares refinement resulted in a final R value of 0.044 for 1505 independent reflections. The two cyclopentadienyl groups are *cis* disposed with respect to the Fe—Fe vector while both the sulphur atom of the ethylthio group and the carbon atom of the bridging carbonyl group are equidistant from the two iron atoms. The latter are separated by a distance of 2.580(2) Å corresponding to a two electron Fe—Fe bond. The solution IR and NMR data are consistent with the cation retaining its *cis* structure in solution.

Introduction

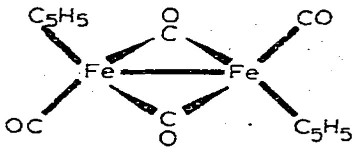
Considerable attention has been devoted to the study of the structural properties of $[\{Fe(\eta\text{-}C_5H_5)(CO)_2\}_2]$ [1—11]. Both the *cis* (I) and *trans* (II) isomers of this compound have been isolated and their crystal structures determined [6,7]. In solution this derivative is present as a mixture of these isomers, as established by IR and ^1H and ^{13}C NMR spectroscopy [5,7—11], with intercon-

---

* For Part XX, see ref. 1.

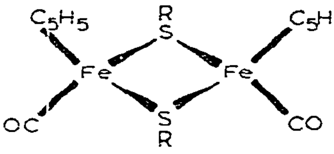


(I)

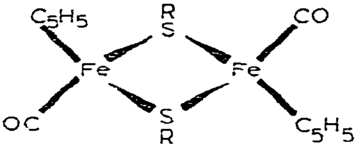


(II)

version of the two forms being extremely rapid at room temperature. This facile intramolecular rearrangement has been explained in terms of a pathway involving a non-bridged isomer as intermediate [10,11]. $[\{Fe(\eta\text{-}C_5H_5)(CO)(SR)\}_2]$ (R = alkyl or aryl group) can also be isolated in two isomeric forms, III and IV [12], but, in contrast to $[\{Fe(\eta\text{-}C_5H_5)(CO)_2\}_2]$, the *cis* isomer (III)

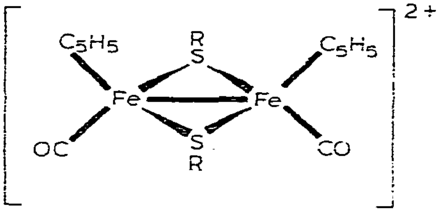


(III)



(IV)

is thermodynamically more stable. Further each isomer can exist independently in solution with elevated temperatures being required to convert the *trans* to the *cis* form. This conversion is thermally irreversible although the *trans* species can be produced from the *cis* by irradiation with UV light. The structural behaviour of the metal—metal bonded species $[\{Fe(\eta\text{-}C_5H_5)(CO)(SR)\}_2]^{2+}$ (V), obtained by two-electron oxidation of $[\{Fe(\eta\text{-}C_5H_5)(CO)(SR)\}_2]$ [13], has not been investigated in any detail but it is expected that interconversion of the *cis* and *trans* isomers will be more difficult than that for the two forms of $[\{Fe(\eta\text{-}C_5H_5)(CO)(SR)\}_2]$.



(V)

It has been recently shown that $[\{Fe(\eta\text{-}C_5H_5)(CO)_2\}_2(SC_2H_5)]^+$ is readily decarbonylated to produce $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_3(SC_2H_5)]^+$ (Fig. 1) [14]. Because the latter can be regarded as being intermediate between $[\{Fe(\eta\text{-}C_5H_5)(CO)_2\}_2]$ and $[\{Fe(\eta\text{-}C_5H_5)(CO)(SC_2H_5)\}_2]^{2+}$, it was considered that a study of its structural properties in both solution and the solid state would be appropriate.

Results and discussion

Spectroscopic studies

$[Fe_2(\eta\text{-}C_5H_5)_2(CO)_3(SC_2H_5)]^+$ can exist in three possible isomeric forms for a planar FeSFe′C ring system. These are illustrated in Fig. 1. The *cis* isomers A and B will give rise to single cyclopentadienyl resonances in their NMR spectra whereas the *trans* isomer will afford two. Two infrared-active terminal C—O stretching modes are predicted for all three isomers which belong to the molecular point groups c_s, c_s, and c_1 respectively. However, in the case of the *cis* isomers, for parallel or near-parallel terminal carbonyl groups, the asymmetric mode will be very weak while for the *trans* isomer the symmetric mode will be of negligible intensity. Also, on the basis of the spectra of $[\{Fe(\eta\text{-}C_5H_5)(CO)_2\}_2]$ [5,7] and $[\{Fe(\eta\text{-}C_5H_5)(CO)(SR)\}_2]^n$ (n = 0, +1, and +2) [12,13], the frequency of the symmetric mode for the *cis* isomers is expected to be reasonably different to that of the asymmetric mode for the *trans* isomer. The IR spectrum of $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_3(SC_2H_5)]^+$ measured in CH_2Cl_2 as the tetrafluoroborate or hexafluoroantimonate salts, contains a strong terminal carbonyl stretching peak at 2032 cm^{-1}, a band of medium intensity at 1848 cm^{-1} assigned to the bridging carbonyls and a weak shoulder at 2004 cm^{-1}. The solid state spectrum reveals a similar band pattern in the C—O stretching region but with increased intensity of the peak at ca. 2000 cm^{-1}. The proton NMR spectrum of this species contains single cyclopentadienyl, methylene and methyl resonances down to temperatures of —60°C. This spectroscopic evidence is interpreted in terms of the cation existing as one of the *cis* isomeric forms A or B in both solution and the solid state. This contrasts with $[\{Fe(\eta\text{-}C_5H_5)(CO)_2\}_2]$ which, as described above, occurs as a mixture of its *cis* and *trans* isomers in solution in rapid equilibrium with each other [8]. Irradiation of $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_3(SC_2H_5)]BF_4$ in THF with UV light did not appear to convert it to the *trans* isomer, as determined by monitoring with IR, unlike that observed [12] for *cis*-$[\{Fe(\eta\text{-}C_5H_5)(CO)(SR)\}_2]$ under similar conditions.

X-Ray crystallographic studies

Crystals of $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_3(SC_2H_5)]SbF_6$ were chosen for the X-ray diffraction study. The molecular geometry of the cation and the atomic nomenclature utilised in its description are shown in Fig. 2; hydrogen atoms have been omitted for clarity. The structure is highly symmetrical with an approximate mirror plane through the bridging sulphur and carbon atoms and the methylene carbon of the —SC_2H_5 ligand. The Fe(1)C(3)Fe(2)S ring is slightly puckered (Table 1) with the dihedral angles between the planes defined by Fe(1)C(3)S and Fe(2)C(3)S and those defined by Fe(1)Fe(2)C(3) and Fe(1)Fe(2)S being 19.2 and 15.6° respectively. The essentially planar cyclopentadienyl rings (Table 1) are *cis* disposed with respect to the Fe—Fe vector but *trans* to the

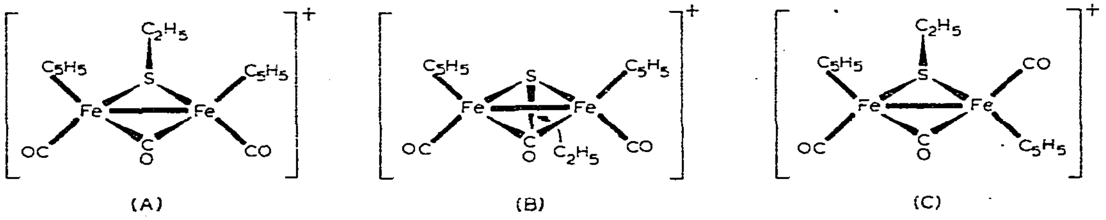


Fig. 1. Possible structures of $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_3(SC_2H_5)]^+$.

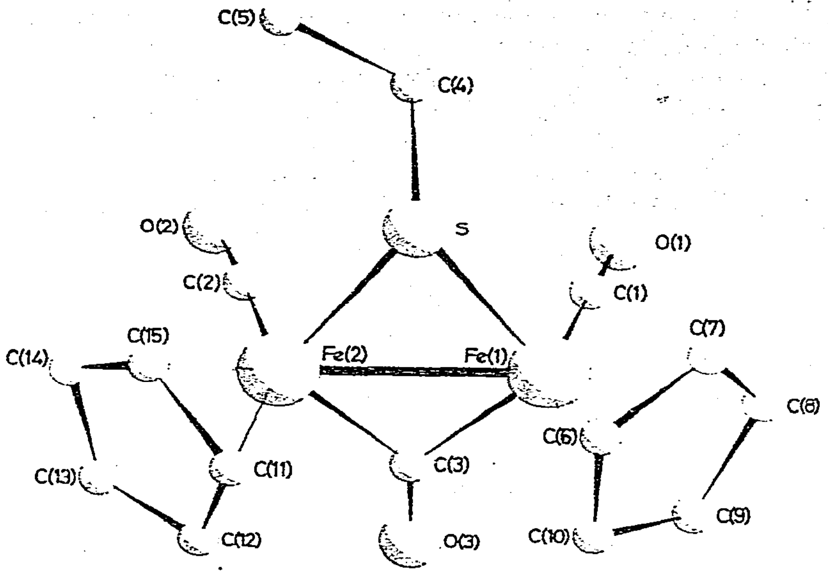


Fig. 2. The molecular geometry of $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_3(SC_2H_5)]^+$ showing the atomic number scheme used in the analysis; hydrogen atoms have been omitted for clarity but are numbered according to their parent carbon atom, i.e. H(6) is bonded to C(6) etc., except for the ethyl hydrogens where H(41) and H(42) are bonded to C(4) and H(51)—H(53) are bonded to C(5).

TABLE 1

LEAST—SQUARES PLANES AND THE PERPENDICULAR DISTANCES FROM EACH ATOM TO THE PLANE in Å

The equations of the planes are expressed in orthogonalized space as $lx + my + nz = P$. Atoms marked * were not included in the least-squares calculation.

Plane 1

$(17.349)x + (-1.408)y + (3.152)z = 3.369$

C(6)	0.014	C(8)	0.005	C(10)	—0.011
C(7)	—0.012	C(9)	0.004	Fe(1) *	1.733

Plane 2

$(0.267)x + (15.811)y + (3.653)z = 7.267$

C(11)	0.004	C(13)	—0.004	C(15)	—0.007
C(12)	0.000	C(14)	0.006	Fe(2) *	1.724

Plane 3

$(12.863)x + (10.040)y + (4.298)z = 10.875$

Fe(1)	—0.002	C(3)	0.008	S(1) *	0.467
Fe(2)	—0.002	O(3)	—0.005		

Plane 4

$(11.214)x + (8.633)y + (7.443)z = 12.373$

Fe(1)	0.0	S(1)	0.0	O(3) *	0.687
Fe(2)	0.0	C(3) *	0.396		

Angles between normals to planes

Plane 1	Plane 2	89.6°
Plane 3	Plane 4	15.2°

ethyl of the ethylthio group. The structure illustrated in Fig. 1B is thus that adopted by the $[SbF_6]^-$ salt of $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_3(SC_2H_5)]^+$ in the crystalline state. The coordination of each iron atom is approximately octahedral, assuming the convention that a cyclopentadienyl group occupies three coordination positions. The cation can thus be considered as two octahedra sharing an edge defined by the two vertices occupied by the bridging sulphur and carbon atoms.

Selected interatomic distances and angles are tabulated in Tables 2 and 3 respectively. The Fe—Fe distance of 2.580(2) Å and the Fe—S—Fe bond angle of 71.7(1)° is consistent with the presence of an Fe—Fe two electron bond [15], in accordance with the magnetic properties of this complex. The two terminal carbonyls show considerable deviation from a possible parallel configuration as revealed by the C(1)—Fe—Fe and C(2)—Fe—Fe bond angles of 102.6(4) and 101.5(4)° respectively. This will explain the moderate intensity of the asymmetric C—O stretching mode in the solid state IR spectrum. Significantly the carbonyl groups in *cis*-$[\{Fe(\eta\text{-}C_5H_5)(CO)(SC_6H_5)\}_2]$ are, within experimental error, parallel [16] and, consistent with this, this compound [12] exhibits a single C—O stretching band in its IR spectrum. For comparison purposes, relevant bond lengths and angles have also been set out in Table 4 together with those of a number of structurally related derivatives. No deviations from established patterns are observed. Thus the Fe—S(bridg), Fe—CO(bridg), Fe—CO(term), and Fe—ct(cp) (ct(cp) = centroid of the cyclopentadienyl group) distances correspond with those found in similar compounds as do the Fe—S—Fe and Fe—C—Fe bridging angles.

A value of 82.6(5)° for the Fe—C—Fe bond angle is however worthy of comment. Considerable evidence is now available to suggest that bridging carbonyl groups will only span metal atoms which are separated by relatively short distances [21]. For instance while $[\{Co(CO)_4\}_2]$ with a Co—Co distance of 2.545(2) Å contains two symmetric bridging carbonyl groups [22], $[FeCo(CO)_8]^-$ with an Fe—Co distance of 2.585(3) Å contains a single asymmetric bridging carbonyl [23] and $[\{Fe(CO)_4\}_2]^{2-}$ with an Fe—Fe bond of 2.787(2) Å contains terminal

TABLE 2

BOND LENGTHS (Å) WITH e.s.d.'s IN PARENTHESES

Fe(1)—Fe(2)	2.580(2)		
Fe(1)—S(1)	2.202(3)	Fe(2)—S(1)	2.205(3)
Fe(1)—C(1)	1.77(1)	Fe(2)—C(2)	1.77(1)
Fe(1)—C(3)	1.96(1)	Fe(2)—C(3)	1.95(1)
Fe(1)—C(6)	2.10(1)	Fe(2)—C(11)	2.11(1)
Fe(1)—C(7)	2.13(1)	Fe(2)—C(12)	2.09(1)
Fe(1)—C(8)	2.10(1)	Fe(2)—C(13)	2.08(1)
Fe(1)—C(9)	2.09(1)	Fe(2)—C(14)	2.08(1)
Fe(1)—C(10)	2.11(1)	Fe(2)—C(15)	2.11(1)
S(1)—C(4)	1.84(1)	C(4)—C(5)	1.48(2)
C(1)—O(1)	1.14(1)	C(2)—O(2)	1.13(1)
C(3)—O(3)	1.16(1)		
C(6)—C(7)	1.43(2)	C(11)—C(12)	1.41(2)
C(6)—C(10)	1.39(2)	C(11)—C(15)	1.40(2)
C(7)—C(8)	1.40(2)	C(12)—C(13)	1.38(2)
C(8)—C(9)	1.40(2)	C(13)—C(14)	1.41(2)
C(9)—C(10)	1.41(2)	C(14)—C(15)	1.38(2)
Sb—F(mean)	1.855(20)		

TABLE 3

BOND ANGLES (°) WITH e.s.d.'s IN PARENTHESES

Fe(1)—S(1)—Fe(2)	71.7(1)		
Fe(1)—C(3)—Fe(2)	82.6(5)		
S(1)—Fe(1)—Fe(2)	54.2(1)	S(1)—Fe(2)—Fe(1)	54.1(1)
C(3)—Fe(1)—Fe(2)	48.5(3)	C(3)—Fe(2)—Fe(1)	48.9(3)
C(1)—Fe(1)—Fe(2)	102.6(4)	C(2)—Fe(2)—Fe(1)	101.5(4)
C(1)—Fe(1)—S(1)	96.8(4)	C(2)—Fe(2)—S(1)	94.2(3)
C(1)—Fe(1)—C(3)	87.4(5)	C(2)—Fe(2)—C(3)	88.4(5)
C(cp)—Fe(1)—C(cp)	(mean)	C(cp)—Fe(2)—C(cp)	(mean)
	39.0(4)		38.9(4)
Fe(1)—C(1)—O(1)	178(1)	Fe(2)—C(2)—O(2)	176(1)
Fe(1)—C(3)—O(3)	138(1)	Fe(2)—C(3)—O(3)	140(1)
Mean internal angle of cp ring bound to Fe(1)	108(1)	Mean internal angle of cp ring bound to Fe(2)	108(1)
Fe(1)—S(1)—C(4)	116.1(4)	Fe(2)—S(1)—C(4)	112.7(4)
S(1)—C(4)—C(5)	111(1)		
F—Sb—F(mean)	90.0(1)		
Torsion angles			
C(1)—Fe(1)—Fe(2)—C(2)	2.5		
C(1)—Fe(1)—C(3)—O(3)	72.2		
C(2)—Fe(2)—C(3)—O(3)	74.8		

carbonyls only [23]. A Mn—Mn distance of 2.923(3) Å for [{Mn(CO)$_5$}$_2$] would also explain the absence of bridging carbonyls in this derivative [24]. It can be implied that the inability of carbonyl groups to bridge metal atoms separated by slightly longer distances is associated with a reluctance on the part of a bridging carbonyl to "open up" its M—C—M bond angle. By comparison the increase in the Fe—Fe distance of 0.81 Å from [Fe$_2$(η-C$_5$H$_5$)$_2$(CO)$_3$(SC$_2$H$_5$)]$^+$ to [{Fe(η-C$_5$H$_5$)(CO)(SC$_6$H$_5$)}$_2$] [16] is accompanied by an increase in the Fe—S—Fe bond

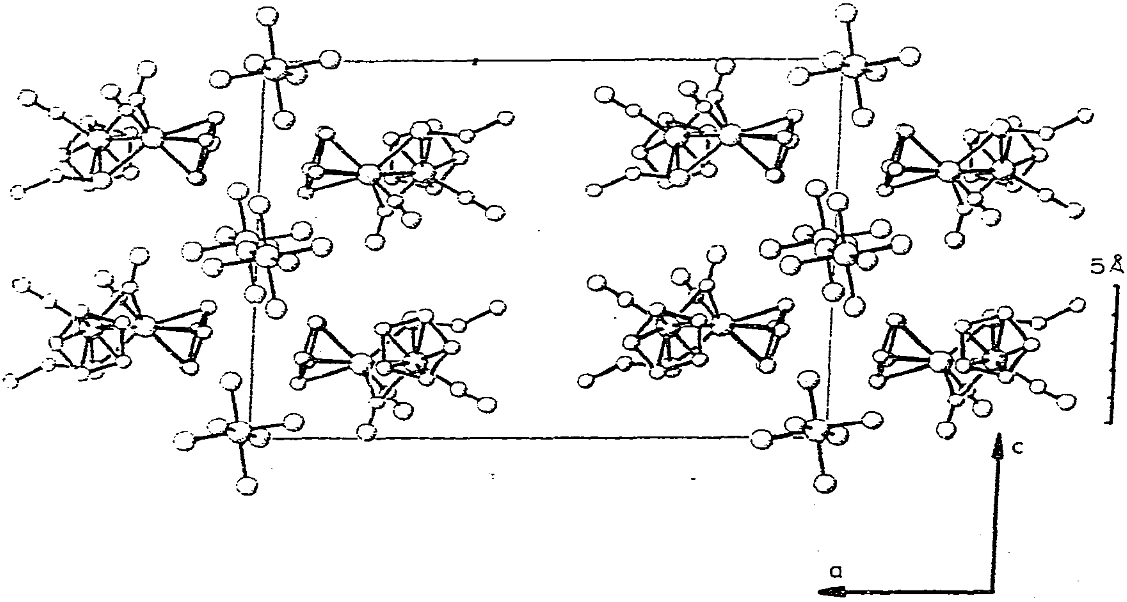


Fig. 3. The molecular packing projected onto (010).

angle of 26.3°. A similar increase in the Fe—C—Fe bond angle of $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_3(SC_2H_5)]^+$ would afford a value of ca. 109°. This is still somewhat less than that expected for an sp^2 hybridised carbon atom. A possible explanation for the apparent inability of bridging carbonyls to increase their bond angles beyond ca. 85° is that similar to that recognised for terminal carbonyls, these groups form stable bonds with transition metals by virtue of a strong π-interaction between the two and that this interaction decreases considerably on expansion of the M—C—M bond angle. This possibility needs further investigation.

Figure 3 shows the molecular packing in the unit cell. No intermolecular close contacts closer than 3.2 Å are observed.

Experimental

The compound was prepared as described previously [14].

Crystal data. $C_{15}H_{15}O_3F_6SFe_2Sb$, Mol. wt. 622.8, crystallises as dark green monoclinic needles with $a = 18.111(9)$, $b = 16.450(7)$, $c = 13.292(6)$ Å, $\beta = 92.2(2)°$, $v = 3957.13$ Å^3, $D_m = 2.10$ g cm^{-3} (by flotation; CH_2I_2/xylene), $D_c = 2.09$ g cm^{-3}, $\mu = 28.02$ cm^{-1}, $F(000) = 2416$, space group $c2/c$ (second setting), $Z = 8$.

Collection and reduction of data. Preliminary photography indicated the space group $c2/c$ or Cc. The former was chosen after inspection of the three dimensional Patterson map, and the refinement has vindicated this choice. Lattice constants were obtained from a least-squares analysis of the settings of 25 reflections measured on a Philips PW1100 four circle diffractometer. The intensities of 1929 reflections were measured on the diffractometer in the $\omega - 2\theta$ scan mode up to $2\theta = 40°$ (scan width 1.0°θ, scan speed 0.03 θ s^{-1}) using graphite-monochromated Mo-K_α radiation ($\lambda = 0.7107$ Å). Throughout the data collection, three reference reflections were recorded after every 52 reflections. Their intensities remained constant to within 4%. 306 reflections were rejected as being systematically absent or space group equivalent. With the criterion $I_{rel} > 2.0\sigma(I_{rel})$ for an observed reflection, a further 118 reflections were discarded as unobserved, leaving 1505 unique reflections to be employed in the determination of the structure. The data were recorded for Lorentz-polarization effects, but not for absorption, the maximum value of μR being 0.66 [25].

Structure determination and refinement. The Sb atom and the two Fe atoms were located from a Patterson map. A difference map based on the coordinates of these atoms (and assuming the space group $C2/c$) yielded the positions of the sulphur atom and six other light atoms. After eight cycles of full matrix least-squares refinement [26] all non-hydrogen atoms had been located and R had fallen to 0.11. A further four cycles of least-squares refinement based on all non-hydrogen atoms with the Sb and Fe atoms anisotropic resulted in an R of 0.07. A subsequent difference map revealed the positions of all but three hydrogen atoms. In a final four cycles of full-matrix least-squares refinement all non-hydrogen atoms were assigned anisotropic temperature factors. The hydrogen atoms were constrained to ride on their parent carbon atoms with C—H set at 1.08 Å, their positions being dictated by the geometry of the molecule. The isotropic temperature factors of the $—CH_2—$, $—CH_3$, and cyclopentadienyl

(continued on p. 360)

TABLE 4

COMPARISON OF BOND LENGTHS (Å) AND ANGLES (°) IN [$Fe_2(\eta\text{-}C_5H_5)_2(CO)_3(SC_2H_5)$]$SbF_6$ AND STRUCTURALLY RELATED COMPOUNDS [a]

Compound	Fe—Fe	Fe—S (av.)	Fe—C(bridg) (av.)	Fe—C(term) (av.)	Fe—ct(cp) (av.)	Fe—S—Fe (av.)	Fe—C(bridg)—Fe (av.)	X—Fe—X′ [b] (av.)
[$Fe_2(cp)_2(CO)_3(SEt)$]SbF_6	2.580(2)	2.204(3)	1.96(1)	1.77(1)	1.73(1)	71.7(1)	82.6(5)	101.5(3)
[$Fe_2(cp)_2(CO)_3(SO_2)$] [c,d]	2.597(1)	2.184(1)	1.932(4)	1.761(4)	1.736(1)	72.93(04)	84.45(15)	98.9(1)
[$Fe_2(cp)_2(CO)_3(GeMe_2)$] [e]	2.628(1)		1.913(6)	1.731(7)	1.758(8)		86.8(2)	85.9(2)
[$\{Fe(cp)(CO)_2\}_2$] (*cis*) [f]	2.531(2)		1.917(7)	1.745(8)	1.746(5)		82.6(3)	96.0(3)
[$\{Fe(cp)(CO)_2\}_2$] (*trans*) [g]	2.534(2)		1.914(5)	1.748(6)	1.754(6)		82.9(2)	97.1(4)
[$\{Fe(cp)(CO)(\mu\text{-}CNCH_3)\}_2$] [h]	2.538(1)		1.942(7)	1.714(8)	1.754(7)		81.9(3)	88.0(3)
[$\{Fe(cp)(CO)(SMe)\}_2$]BF_4 [i]	2.925(4)	2.234(4)		1.79(2)		81.8(1)		95.4(1)
[$\{Fe(cp)(CO)(SPh)\}_2$] [j,k]	3.39	2.262(6)		1.71(3)	1.71	98		81

[a] Ct(cp) signifies the centroid of the cyclopentadienyl group. Abbreviations: cp = η-C_5H_5; Me = CH_3; Et = C_2H_5; Ph = C_6H_5. [b] X, X′ = bridging atoms. [c] Data are for molecule A of the asymmetric unit. [d] Ref. 17. [e] Ref. 18. [f] Ref. 7. [g] Ref. 6. [h] Ref. 19. [i] Ref. 20. [j] Average values for the two independent molecules in the asymmetric unit. [k] Ref. 16.

TABLE 5

FRACTIONAL ATOMIC COORDINATES AND THEIR e.s.d.'s (× 10^4) AND ANISOTROPIC TEMPERATURE FACTORS AND THEIR e.s.d.'s (× 10^3)

Atom	x/a	y/b	z/c	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Sb	−182(0)	2564(1)	10196(1)	40(1)	61(1)	41(1)	13(0)	6(0)	4(0)
Fe(1)	1893(1)	4983(1)	7991(1)	32(1)	38(1)	36(1)	−1(1)	3(1)	1(1)
Fe(2)	2828(1)	3802(1)	7953(1)	36(1)	38(1)	33(1)	−1(1)	0(1)	2(1)
F(1)	761(4)	2197(6)	9549(6)	56(5)	140(7)	109(6)	4(5)	20(4)	18(5)
F(2)	−10(4)	2419(6)	11183(5)	96(6)	235(12)	51(5)	38(6)	−10(4)	37(7)
F(3)	−1125(4)	2970(5)	10053(5)	60(5)	123(7)	70(5)	4(5)	9(4)	31(5)
F(4)	−368(5)	2761(6)	8448(5)	128(7)	168(9)	46(5)	24(5)	11(5)	43(6)
F(5)	−556(5)	1542(5)	9664(8)	109(7)	89(7)	198(10)	9(7)	17(6)	−26(6)
F(6)	178(5)	3622(5)	9953(7)	137(8)	94(7)	139(8)	−13(6)	20(6)	−36(6)
S(1)	2753(2)	4824(2)	6881(2)	44(2)	54(2)	38(3)	8(2)	9(1)	7(2)
O(1)	2653(5)	6193(5)	9244(7)	92(7)	54(6)	87(7)	−12(5)	−17(5)	−10(6)
O(2)	4096(5)	4444(6)	9087(7)	65(7)	89(7)	81(7)	7(6)	−22(5)	−19(6)
O(3)	2042(4)	4006(5)	9823(6)	79(6)	82(7)	28(5)	6(5)	13(4)	5(5)
C(1)	2370(6)	5716(8)	8746(9)	45(8)	45(8)	69(9)	−3(7)	15(7)	3(6)
C(2)	3590(7)	4216(7)	8651(8)	45(8)	56(8)	37(7)	21(6)	12(6)	9(6)
C(3)	2192(6)	4174(7)	9009(8)	40(7)	53(8)	29(7)	−4(6)	6(6)	5(6)
C(4)	3550(6)	5515(8)	7002(10)	53(9)	65(9)	85(10)	45(8)	20(7)	2(7)
C(5)	4207(7)	5150(9)	6545(11)	46(9)	123(14)	101(12)	48(10)	20(8)	12(9)
C(6)	1072(6)	4659(9)	6911(10)	43(8)	72(11)	61(10)	−25(8)	−11(7)	7(7)
C(7)	1122(6)	5523(9)	6945(10)	41(8)	73(11)	55(9)	9(8)	3(6)	10(7)
C(8)	971(6)	5760(8)	7931(11)	48(8)	48(9)	80(10)	−2(8)	−7(7)	4(7)
C(9)	813(5)	5061(9)	8489(9)	30(7)	88(11)	55(8)	−1(9)	14(6)	2(7)
C(10)	865(6)	4381(7)	7849(11)	28(7)	49(9)	86(10)	−10(9)	−11(7)	10(6)
C(11)	2247(10)	2925(9)	7083(14)	84(13)	61(10)	106(14)	−41(10)	−44(11)	25(9)
C(12)	2291(10)	2686(8)	8100(15)	93(13)	41(9)	124(15)	−29(9)	52(11)	−15(8)
C(13)	3029(9)	2605(7)	8388(10)	79(10)	32(8)	81(10)	7(7)	−13(9)	13(7)
C(14)	3449(7)	2797(7)	7553(11)	62(9)	45(8)	80(10)	−10(7)	10(9)	10(7)
C(15)	2970(11)	2981(8)	6758(9)	142(15)	48(9)	37(8)	−7(7)	−9(10)	36(10)

TABLE 6

FRACTIONAL ATOMIC COORDINATES OF THE HYDROGEN ATOMS (X 10^4)

Atom	*x/a*	*y/b*	*z/c*	Atom	*x/a*	*y/b*	*z/c*
H(41)	3674(6)	5646(8)	7786(10)	H(9)	668(5)	5037(9)	9269(9)
H(42)	3417(6)	6073(8)	6607(10)	H(10)	767(6)	3753(7)	8040(11)
H(51)	4642(7)	5596(9)	6497(11)	H(11)	1753(10)	3035(9)	6622(14)
H(52)	4028(7)	4964(9)	5795(11)	H(12)	1823(10)	2590(8)	8567(15)
H(53)	4412(7)	4628(9)	6962(11)	H(13)	3236(9)	2427(9)	9129(10)
H(6)	1181(6)	4291(9)	6260(10)	H(14)	4045(7)	2800(7)	7543(11)
H(7)	1256(6)	5924(9)	6335(10)	H(15)	3127(11)	3145(8)	6009(9)
H(8)	972(6)	6375(8)	8216(11)				

hydrogens were refined as three independent variables and converged to values of $U = 0.073$, 0.089, and 0.095 Å^2 respectively. The refinement converged to $R = 0.044$ and $R_w = 0.040$ with a weighting scheme $w = 0.5785/(\sigma^2F + 0.000965 F^2)$.

The final atomic coordinates and temperature factors are given in Tables 5 and 6. The observed and calculated structure factors and an analysis of variance computed after the final cycle are available, from the authors;

All calculations were carried out on a UNIVAC 1106 computer at the University of Cape Town.

Acknowledgement

The authors express their gratitude to the South African Council for Scientific and Industrial Research for the diffractometer data collection and also, together with the University of Cape Town, for financial support.

References

1 F.A. Cotton, H. Stammreich and G. Wilkinson, J. Inorg. Nucl. Chem., 9 (1959) 3.
2 K. Noack, J. Inorg. Nucl. Chem., 25 (1963) 1383.
3 R.D. Fischer, A. Vogler and K. Noack, J. Organometal. Chem., 7 (1967) 135.
4 F.A. Cotton and G. Yagupsky, Inorg. Chem., 6 (1967) 15.
5 A.R. Manning, J. Chem. Soc. A, (1968) 1319.
6 R.F. Bryan and P.T. Greene, J. Chem. Soc. A, (1970) 3064.
7 R.F. Bryan, P.T. Greene, M.J. Newlands and D.S. Field, J. Chem. Soc. A, (1970) 3068.
8 J.G. Bullitt, F.A. Cotton and T.J. Marks, Inorg. Chem., 11 (1972) 671.
9 O.A. Gansow, A.R. Burke and W.D. Vernon, J. Amer. Chem. Soc., 94 (1972) 2550.
10 R.M. Kirchner, T.J. Marks, J.S. Kristoff and J.A. Ibers, J. Amer. Chem. Soc., 95 (1973) 6602.
11 F.A. Cotton and R.D. Adams, J. Amer. Chem. Soc., 95 (1973) 6589.
12 R.J. Haines, J.A. de Beer and R. Greatrex, J. Organometal. Chem., 85 (1975) 89.
13 J.A. de Beer, R.J. Haines, R. Greatrex and J.A. van Wyk, J. Chem. Soc. Dalton, (1973) 2341.
14 R.B. English, R.J. Haines and C.R. Nolte, J. Chem. Soc. Dalton, (1975) 1030.
15 L.F. Dahl, E. Rodulfode Gil and R.D. Feltham, J. Amer. Chem. Soc., 91 (1969) 1653.
16 G. Ferguson, C. Hannaway and K.M.S. Islam, Chem. Commun., (1968) 1165.
17 M.R. Churchill and K.L. Kalra, Inorg. Chem., 12 (1973) 1650.
18 R.D. Adams, M.D. Brice and F.A. Cotton, Inorg. Chem., 13 (1974) 1080.
19 F.A. Cotton and B.A. Frenz, Inorg. Chem., 13 (1974) 253.
20 N.G. Connelly and L.F. Dahl, J. Amer. Chem. Soc., 92 (1970) 7472.
21 F.A. Cotton and J.M. Troup, J. Amer. Chem. Soc., 96 (1974) 5070.
22 G.G. Summer, H.P. Klug and L.E. Alexander, Acta Cryst., 17 (1964) 732.
23 H.B. Chin, M.B. Smith, R.D. Wilson and R. Bau, J. Amer. Chem. Soc., 96 (1974) 5285.
24 L.F. Dahl and R.E. Rundle, Acta Cryst., 16 (1963) 419.
25 "International Tables for X-ray Crystallography", 1967, Vol. 11, 2nd ed., Kynoch Press, Birmingham, pp. 291—313.
26 G.M. Sheldrick, Shel-X Program System, to be published.