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Structure of a Bis(η^4-exocyclic-1,3-diene)Fe(CO)$_3$ Complex

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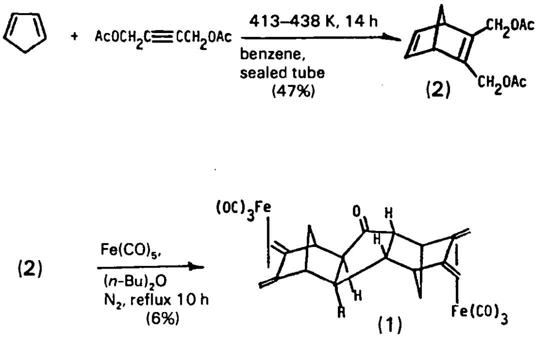
Abstract. *trans*-μ-(12–13-η:14–15-η-1,2,3,4,4a,4b,5,6,7,8,8a,9a-Dodecahydro-2,3,6,7-tetramethylene-1,4:5,8-dimethanofluoren-9-one)-bis(tricarbonyliron), [Fe$_2$(CO)$_6$(C$_{19}$H$_{20}$O)], $M_r = 544{\cdot}12$, triclinic, $P\bar{1}$, $a = 6{\cdot}940\,(1)$, $b = 12{\cdot}418\,(2)$, $c = 13{\cdot}894\,(2)$ Å, $\alpha = 93{\cdot}65\,(1)$, $\beta = 94{\cdot}50\,(1)$, $\gamma = 95{\cdot}31\,(1)°$, $V = 1185{\cdot}4\,(3)$ Å^3, $Z = 2$, $D_x = 1{\cdot}524$ g cm^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}71073$ Å, $\mu = 12{\cdot}64$ cm^{-1}, $F(000) = 556$, $T = 294$ K, $R = 0{\cdot}0412$ for 2432 reflections. The Fe^0 atoms exhibit distorted tetragonal pyramidal geometries with the bases defined by two C—O groups and the midpoints of the C=C bonds of the (*S-cis*)-butadiene moieties. The norbornene rings are fused to a planar cyclopentanone ring in an *exo–trans–exo* fashion.

Introduction. As part of a continuing program that is involved with the study of the stereochemistry and mechanism of thermal reactions of iron pentacarbonyl with substituted norbornadienes (Speert, Gelan, Anteunis, Marchand & Laszlo, 1973; Marchand & Hayes, 1977; Marchand, Goodin, Hossain & van der Helm, 1984; Marchand, Earlywine & Heeg, 1986), we have examined the corresponding reaction of 2,3-bis(acetoxy)norbornadiene with Fe(CO)$_5$. Reactions of this kind have been found typically to afford a variety of norbornadiene dimers and also to afford products that are derived *via* Fe(CO)$_5$-promoted coupling of the strained alkene substrate to carbon monoxide (Weissberger & Laszlo, 1976).

In the past, no effort has been made in our laboratory to isolate iron-containing intermediates and/or products that might result from these reactions. Instead, such Fe^0 complexes are routinely destroyed oxidatively by stirring the crude reaction product with a solution of ferric chloride in acetone (Marchand & Hayes, 1977; Marchand, Goodin, Hossain & van der Helm, 1984; Marchand, Earlywine & Heeg, 1986). However, when this procedure was applied to the product of the thermal reaction of 2,3-bis(acetoxy)norbornadiene with Fe(CO)$_5$, a pale yellow crystalline solid was obtained in low yield; this material proved to be the unusually stable bis(η^4-1,3-diene)Fe(CO)$_3$ complex (1).



Experimental. A pale yellow crystal of dimensions $0{\cdot}35 \times 0{\cdot}10 \times 0{\cdot}175$ mm; Nicolet $R3m/\mu$ update of a $P2_1$ diffractometer; data collected in the Wyckoff mode ($4 \le 2\theta \le 45°$, 2θ fixed, ω varied), variable scan rate (4 to 29·3° min^{-1}), graphite-monochromated Mo $K\alpha$ radiation; lattice parameters from a least-squares

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Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^3$)

	x	y	z	U_{eq}*
Fe(1)	4126 (1)	2540 (1)	3977 (1)	49 (1)
Fe(2)	10165 (1)	3157 (1)	−2354 (1)	49 (1)
C(1)	2121 (7)	3193 (4)	3494 (3)	59 (2)
O(1)	872 (5)	3615 (3)	3164 (3)	88 (2)
C(2)	4399 (6)	3103 (3)	5205 (3)	57 (2)
O(2)	4608 (5)	3473 (3)	5979 (2)	83 (1)
C(3)	2662 (8)	1321 (4)	4071 (4)	77 (2)
O(3)	1713 (7)	526 (3)	4143 (3)	134 (2)
C(4)	9951 (7)	3650 (4)	−3537 (4)	63 (2)
O(4)	9785 (5)	3958 (3)	−4288 (2)	93 (2)
C(5)	12011 (7)	4073 (4)	−1737 (4)	64 (2)
O(5)	13214 (5)	4651 (3)	−1317 (3)	95 (2)
C(6)	11799 (7)	2161 (4)	−2537 (3)	69 (2)
O(6)	12806 (5)	1504 (3)	−2677 (3)	111 (2)
C(7)	5804 (5)	2942 (3)	2868 (3)	41 (1)
C(8)	6100 (5)	1891 (3)	3118 (3)	45 (1)
C(9)	5675 (5)	1161 (3)	2203 (3)	44 (1)
C(10)	7407 (5)	1475 (3)	1591 (3)	40 (1)
C(11)	7270 (5)	784 (3)	644 (3)	40 (1)
O(11)	7374 (4)	−182 (2)	574 (2)	50 (1)
C(12)	6998 (5)	1471 (3)	−213 (3)	36 (1)
C(13)	8827 (5)	1511 (3)	−802 (3)	43 (1)
C(14)	8291 (5)	2177 (3)	−1640 (3)	42 (1)
C(15)	8317 (5)	3251 (3)	−1247 (3)	40 (1)
C(16)	8856 (5)	3232 (3)	−174 (3)	40 (1)
C(17)	6998 (5)	2662 (3)	214 (2)	36 (1)
C(18)	7119 (5)	2647 (3)	1328 (3)	36 (1)
C(19)	5204 (5)	2843 (3)	1786 (3)	41 (1)
C(20)	4095 (5)	1708 (3)	1645 (3)	42 (1)
C(21)	10203 (5)	2325 (3)	−144 (3)	46 (1)
C(22)	6680 (6)	1714 (3)	4089 (3)	61 (2)
C(23)	6075 (6)	3819 (3)	3571 (3)	55 (2)
C(24)	7844 (6)	1926 (3)	−2640 (3)	57 (2)
C(25)	7946 (7)	4077 (4)	−1863 (3)	54 (2)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

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

Fe(1)–C(1)	1·782 (5)	Fe(1)–C(2)	1·791 (4)
Fe(1)–C(3)	1·761 (5)	Fe(1)–C(7)	2·062 (4)
Fe(1)–C(8)	2·071 (4)	Fe(2)–C(4)	1·790 (5)
Fe(2)–C(5)	1·763 (4)	Fe(2)–C(6)	1·773 (5)
Fe(2)–C(14)	2·054 (4)	Fe(2)–C(15)	2·083 (4)
C(7)–C(8)*	1·401 (5)	C(7)–C(19)	1·521 (5)
C(7)–C(23)	1·405 (5)	C(8)–C(9)	1·507 (5)
C(8)–C(22)	1·414 (5)	C(9)–C(10)	1·560 (5)
C(9)–C(20)	1·529 (5)	C(10)–C(11)	1·516 (5)
C(10)–C(18)	1·550 (5)	C(11)–O(11)	1·208 (5)
C(17)–C(18)	1·545 (5)		
C(1)–Fe(1)–C(2)	101·4 (2)	C(1)–Fe(1)–C(3)	92·4 (2)
C(2)–Fe(1)–C(3)	101·8 (2)	C(4)–Fe(2)–C(5)	102·8 (2)
C(4)–Fe(2)–C(6)	100·3 (2)	C(5)–Fe(2)–C(6)	92·0 (2)
C(8)–C(7)–C(19)	105·8 (3)	C(8)–C(9)–C(20)	102·6 (3)
C(8)–C(7)–C(23)	120·7 (3)	C(11)–C(10)–C(18)	106·6 (3)
C(7)–C(8)–C(9)	106·6 (3)	C(10)–C(11)–C(12)	111·0 (3)
C(7)–C(8)–C(22)	119·5 (3)	C(13)–C(12)–C(17)	103·5 (3)
C(8)–C(9)–C(10)	103·9 (3)	C(12)–C(13)–C(21)	99·9 (3)
C(9)–C(10)–C(18)	104·2 (3)	C(13)–C(14)–C(15)	106·2 (3)
C(10)–C(11)–O(11)	124·9 (3)	C(13)–C(14)–C(24)	133·6 (3)
O(11)–C(11)–C(12)	124·1 (3)	C(14)–C(15)–C(16)	106·4 (3)
C(11)–C(12)–C(17)	106·5 (3)	C(14)–C(15)–C(25)	119·6 (3)
C(12)–C(13)–C(14)	103·8 (3)	C(15)–C(16)–C(17)	103·8 (3)
C(15)–C(14)–C(24)	120·2 (4)	C(17)–C(16)–C(21)	100·9 (3)
C(12)–C(17)–C(16)	102·2 (3)	C(12)–C(17)–C(18)	107·6 (3)
C(10)–C(18)–C(19)	101·7 (3)	C(10)–C(18)–C(17)	108·0 (3)
C(7)–C(19)–C(18)	103·4 (3)	C(7)–C(19)–C(20)	102·2 (3)
C(13)–C(21)–C(16)	95·0 (3)	C(9)–C(20)–C(19)	94·4 (3)

* C–C distances shown for one half of the molecule.

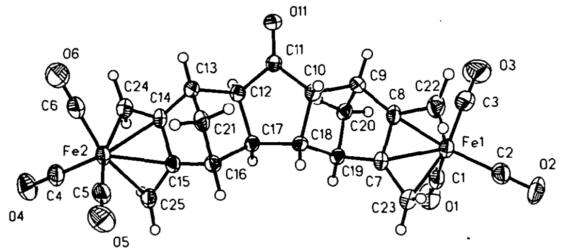

Fig. 1. Drawing of compound (1) with thermal ellipsoids drawn at the 30% probability level. H atoms are represented by spheres of arbitrary size.

refinement of 25 reflections ($31{\cdot}78 \le 2\theta \le 38{\cdot}50°$), angles measured by a centering routine; no systematic absences, Laue symmetry $\bar{1}$ and statistics consistent with space group $P\bar{1}$; monitored reflections $\bar{4}12$ and $\bar{2}15$ showed only statistical variation in intensities; 3097 independent reflections measured ($0 \le h \le 7$, $-13 \le k \le 13$, $-14 \le l \le 14$) with 2432 greater than $3\sigma(I)$; Lorentz and polarization corrections, ψ-scan empirical absorption correction (transmission factors 0·518 to 0·647); structure solved by direct-method techniques, block-cascade least-squares refinement, all H atoms located in a difference map and refined with a single isotropic thermal parameter; final $R = 0{\cdot}0412$, $wR = 0{\cdot}0334$ for 368 parameters ($R = 0{\cdot}0582$, $wR = 0{\cdot}0348$ for all data), $S = 1{\cdot}397$, $(\Delta/\sigma)_{max} = 0{\cdot}011$, $(\Delta/\sigma)_{av} = 0{\cdot}002$, largest peaks in the final difference map of 0·27 and $-0{\cdot}28\,e\,Å^{-3}$; $\sum w(|F_o| - |F_c|)^2$ minimized with $w = [\sigma^2(F_o) + 0{\cdot}00006F_o^2]^{-1}$. All computer programs supplied by Nicolet for Desktop 30 Microeclipse and Nova 4/C configuration (Nicolet Instrument Corporation, 1986); atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). Table 1 lists atomic positional parameters while Table 2 lists selected interatomic distances and valence angles.*

Discussion. Fig. 1 is a drawing of compound (1). The Fe atoms exhibit distorted tetragonal pyramidal geometries with the bases represented by two CO groups and the midpoints of the two C=C bonds of the (*S-cis*)-butadiene moieties. The basal atoms are planar (0·03 Å r.m.s.d.) in both Fe-atom geometries, and Fe(1) and Fe(2) are out of plane by 0·506 (1) and 0·522 (1) Å, respectively. The basal Fe—CO distances range from 1·761 (5) to 1·782 (5) Å while the apical distances are 1·791 (4) and 1·790 (5) Å. The distances from the Fe atoms to the midpoints of the C=C bonds

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* Lists of H-atom coordinates, anisotropic thermal parameters, all bond lengths and angles, least-squares planes and equations and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44659 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

range from 1·960 (4) to 1·985 (4) Å. The C atoms of the butadiene moieties are planar (0·0001 and 0·006 Å r.m.s.d.). Each butadiene plane is perpendicular [89·8 (1)°] to the basal plane of the tetragonal pyramid. Fe(2)—C(24), 2·110 (4) Å, is marginally shorter than the other three Fe–terminal C-atom distances [2·123 (4) to 2·130 (5) Å]. The terminal H atoms of the butadiene groups are not coplanar with these units, and the interplanar angles between the terminal H—C—H groups and the butadiene planes range from 37 (2) to 42 (2)°. The three C—C bonds of each butadiene moiety are statistically equivalent [1·401 (5) to 1·414 (5) Å]. Similar effects have been reported for other $Fe(CO)_3$–butadiene complexes (Cotton, Day, Frenz, Hardcastle & Troup, 1973; Meier, Cherpillod, Boschi, Roulet, Vogel, Mahaim, Pinkerton, Schwarzenbach & Chapuis, 1980; Meier, Pinkerton, Roulet, Vogel & Schwarzenbach, 1981; Pinkerton, Chapuis, Vogel, Hanisch, Narbel, Boschi & Roulet, 1979; Sustmann, Bohm & Saver, 1979). Least-squares planes and other interplanar angles are given in the supplementary material.

The cyclopentanone ring system is planar (0·03 Å r.m.s.d.) and the two norbornane rings are fused to it in an *exo–trans–exo* arrangement. The methylene bridges C(20) and C(21) are bent toward the cyclopentanone portion of the molecule as indicated by the non-equivalent interplanar angles, *e.g.* C(9)C(20)C(19) makes angles of 130·0 (3) and 121·3 (3)° with C(7)C(8)C(9)C(19) and C(9)C(10)C(18)C(19), respectively. All chemically equivalent bonds in the organic portion of the complex are statistically equivalent.

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Structure of Bis(oxamato-*O*,*O*′)lead(II) Monohydrate

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Abstract. $[Pb(C_2H_2NO_3)_2].H_2O$, $M_r = 401·3$, triclinic, $P\bar{1}$, $a = 6·727$ (1), $b = 7·293$ (1), $c = 9·814$ (1) Å, $\alpha = 81·70$ (1), $\beta = 75·93$ (1), $\gamma = 64·06$ (1)°, $V = 420$ Å^3, $Z = 2$, $D_x = 3·18$ Mg m^{-3}, $\lambda(\text{Ag } K\alpha)$, $\mu = 10·5$ mm^{-1}, $F(000) = 362$, $R = 0·027$, $wR = 0·031$, $S = 0·93$. The coordination polyhedron of the metal ion is a distorted pentagonal dipyramid. Each Pb^{2+} ion is bonded to seven O atoms with six belonging to four oxamate ions and one to the water molecule. The water molecule is also involved in three hydrogen bonds that reinforce the stability of the hydrate and the cohesion of the structure.

Introduction. This X-ray structure determination is part of a more general study on the interaction of metals with small ligands of the type *R*COCO*R*′ (*R* = OH, NH_2, *R*′ = OH, NH_2). Crystals of the title compound

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