Table 2. *Geometry of the hydrogen bonds (e.s.d.'s in parentheses)*

It is assumed that e.s.d.'s for H atoms in calculated positions are 10 times those of the atoms to which they are attached.

N–H···Cl	N–H (Å)	H···Cl (Å)	N···Cl (Å)	N–H···Cl (°)
N(11)–H(11)···Cl(4^{i})	1·00 (4)	2·64 (5)	3·248 (4)	119 (3)
N(11)–H(11)···Cl(5^{i})		2·36 (4)	3·175 (4)	138 (3)
N(13)–H(13)···Cl(1^{ii})	1·00 (4)	2·24 (4)	3·216 (5)	164 (4)
N(21)–H(21)···Cl(4^{iii})	1·00 (5)	2·33 (5)	3·196 (5)	144 (4)
N(21)–H(21)···Cl(5^{iii})		2·64 (5)	3·368 (5)	130 (4)
N(23)–H(23)···Cl(1^{iv})	1·00 (5)	2·73 (5)	3·396 (5)	124 (4)
N(23)–H(23)···Cl(2^{v})		2·45 (5)	3·238 (5)	135 (4)

Symmetry code: (i) $1-x, 1-y, 1-z$; (ii) $\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z$; (iii) $\frac{1}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$; (iv) $x-\frac{1}{2}, \frac{1}{2}-y, \frac{1}{2}+z$; (v) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$.

exactly on the $y=0$ and $y=\frac{1}{2}$ planes form stacking columns running along the 2_1 axis. The distances between the rings are 3·221 (7) Å, which are thus markedly shorter than in the graphite structure (3·35 Å). Short C···C van der Waals contacts of 3·189 (8), 3·263 (8), 3·285 (8) and 3·339 (8) Å are observed. Research on the magnetic properties of these crystals and comparison with the magnetism of other polymeric chlorocuprates is planned.

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Structure of Hexacarbonyl-μ-ethoxymethylidyne-μ-[σ:1–2-η-(1-phenyl-1-propenyl)]-diiron(*Fe–Fe*), $C_{18}H_{14}Fe_2O_7$

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Abstract. $[Fe_2(CO)_6(C_9H_9)(C_3H_5O)]$, $M_r = 454{\cdot}0$, monoclinic, $P2_1/a$, $a = 16{\cdot}591\ (2)$, $b = 17{\cdot}140\ (3)$, $c = 6{\cdot}948\ (2)$ Å, $\beta = 100{\cdot}83\ (2)°$, $V = 1941\ (1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}55$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 1{\cdot}566$ mm^{-1}, $F(000) = 920$, room temperature, $R = 0{\cdot}049$ for 1235 observed reflections. The structure consists of an $Fe_2(CO)_6$ unit with 1-phenyl-1-propenyl and ethoxymethylidyne bridging groups. The first is σ bonded to an iron atom and unsymmetrically π bonded to the other iron atom, while the ethoxymethylidyne is symmetrically bonded to the two iron atoms. The methyl and phenyl groups attached to the vinyl group are in *cis* configuration. The Fe–Fe length is 2·549 (2) Å.

Introduction. A systematic study of carbon–carbon bond formation at diiron centres is being carried out by the authors' departments. In this paper it is shown that the reaction of $[Fe_2(CO)_6(\mu\text{-}CO)\{\mu\text{-}C(C_6H_5)\text{-}C(CH_3)H\}][P(C_6H_5)_4]$ (Lourichi & Mathieu, 1982) with a stoichiometric amount of $(C_2H_5)_3OBF_4$ at room temperature gives an almost quantitative yield of the title compound.

In order to confirm this result and compare the geometry of the title compound with that observed in $[Fe_2(CO)_6(\mu\text{-}COC_2H_5)\{\mu\text{-}C(C_6H_5)C(C_6H_5)H\}]$ (Ros, Yañez, Mathieu, Solans & Font-Altaba, 1985), an X-ray analysis was undertaken.

Experimental. Yellow prismatic crystals (0·1 × 0·1 × 0·2 mm) crystallized from dichloromethane/ethanol; Philips PW 1100 diffractometer, graphite monochromatized Mo $K\alpha$, cell parameters from 25 reflections ($4 \leq \theta \leq 9°$), ω-scan technique, scan width 1°, scan speed 0·03° s^{-1}. Three intensities measured every 2 h, no significant differences. 1325 reflections ($2 \leq \theta \leq 25°$); 1235 with $I \geq 2{\cdot}5\sigma(I)$; h,k,l range ±17,20,8. Structure solved by direct methods, Fe atoms from *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and remaining non-hydrogen atoms from *DIRDIF*81 (Beurskens, Bosman, Doesburg, Gould, van den Hark, Prick, Noordik, Beurskens & Parthasarathi, 1981). Full-matrix least-squares refinement (*SHELX*76; Sheldrick, 1976), $\sum w||F_o| - |F_c||^2$ minimized, $w = [\sigma^2(F_o) + 0{\cdot}00075|F_o|^2]^{-1}$, 13 H in computed positions, H(C5) by ΔF synthesis, overall isotropic temperature factor refined for H atoms, coordinates not refined. Final $R = 0{\cdot}049$, $wR = 0{\cdot}048$ for all observed reflections; f, f' and f'' from *International Tables for X-ray Crystallography* (1974); max. $\Delta/\sigma = 0{\cdot}9$ in U_{11} of C(1), max. and min. $\Delta\rho$ in final ΔF map $\pm 0{\cdot}3$ e Å^{-3}. IBM-4341 computer.

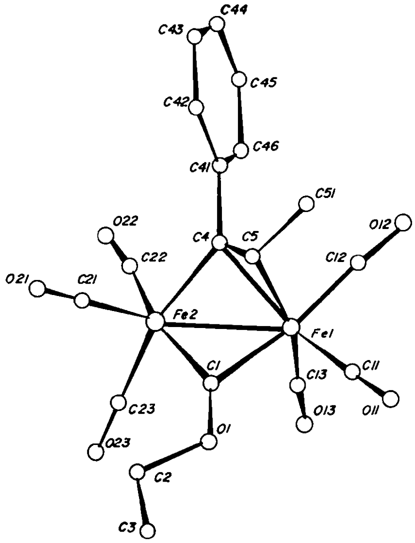

Fig. 1. View of the molecule showing the atom numbering.

Table 1. *Final atomic coordinates* (×10^4) *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)	6746 (1)	304 (1)	734 (2)	3·18 (8)
Fe(2)	6895 (1)	1783 (1)	874 (3)	3·47 (9)
C(1)	6105 (7)	1128 (7)	1426 (15)	3·6 (6)
O(1)	5393 (4)	1111 (5)	1918 (11)	4·2 (4)
C(2)	5049 (8)	1859 (8)	2511 (22)	6·2 (8)
C(3)	4299 (8)	1681 (8)	3244 (21)	6·5 (9)
C(4)	7675 (6)	993 (7)	2342 (14)	3·1 (5)
C(5)	7382 (7)	528 (7)	3725 (17)	3·5 (6)
C(51)	7875 (7)	−128 (7)	4883 (18)	4·9 (7)
C(41)	8563 (6)	999 (7)	2274 (16)	3·2 (5)
C(42)	9136 (7)	1139 (7)	3994 (17)	4·0 (6)
C(43)	9967 (7)	1157 (7)	3986 (21)	4·9 (7)
C(44)	10239 (8)	1041 (8)	2231 (23)	5·1 (7)
C(45)	9689 (8)	921 (7)	538 (19)	4·6 (7)
C(46)	8860 (7)	892 (6)	540 (17)	3·7 (6)
C(11)	6130 (8)	−417 (8)	1584 (18)	4·3 (7)
O(11)	5741 (6)	−880 (6)	2164 (14)	6·3 (6)
C(12)	7507 (8)	−370 (8)	179 (19)	4·1 (7)
O(12)	7957 (6)	−844 (5)	−127 (15)	6·4 (6)
C(13)	6263 (7)	413 (7)	−1787 (19)	4·3 (7)
O(13)	5935 (5)	449 (6)	−3389 (13)	6·1 (5)
C(21)	7042 (8)	2433 (8)	2941 (22)	4·9 (7)
O(21)	7123 (6)	2820 (6)	4276 (15)	7·5 (6)
C(22)	7657 (8)	2162 (8)	−434 (21)	5·5 (9)
O(22)	8092 (6)	2469 (7)	−1270 (15)	8·0 (7)
C(23)	6111 (8)	2292 (8)	−868 (20)	5·0 (8)
O(23)	5629 (6)	2609 (6)	−1914 (14)	7·5 (6)

Table 2. *Bond lengths and angles with e.s.d.'s in parentheses*

Fe(2)–Fe(1)	2·549 (2)	O(1)–C(1)	1·290 (12)
C(1)–Fe(1)	1·884 (12)	C(2)–O(1)	1·493 (13)
C(5)–Fe(1)	2·181 (11)	C(3)–C(2)	1·463 (15)
C(4)–Fe(1)	2·092 (10)	C(51)–C(5)	1·528 (14)
C(11)–Fe(1)	1·774 (14)	C(4)–C(5)	1·403 (14)
C(12)–Fe(1)	1·805 (14)	C(41)–C(4)	1·483 (13)
C(13)–Fe(1)	1·793 (13)	O(11)–C(11)	1·142 (12)
C(1)–Fe(2)	1·821 (12)	O(12)–C(12)	1·150 (12)
C(4)–Fe(2)	2·014 (10)	O(13)–C(13)	1·145 (12)
C(21)–Fe(2)	1·798 (15)	O(21)–C(21)	1·127 (13)
C(22)–Fe(2)	1·810 (14)	O(22)–C(22)	1·136 (13)
C(23)–Fe(2)	1·824 (14)	O(23)–C(23)	1·115 (13)

C(5)–Fe(1)–C(1)	79·6 (4)	C(23)–Fe(2)–C(1)	89·0 (5)
C(4)–Fe(1)–C(1)	80·5 (4)	C(23)–Fe(2)–C(4)	165·5 (5)
C(4)–Fe(1)–C(5)	38·3 (4)	C(23)–Fe(2)–C(21)	101·9 (6)
C(11)–Fe(1)–C(1)	92·8 (5)	C(23)–Fe(2)–C(22)	88·5 (5)
C(11)–Fe(1)–C(5)	90·7 (5)	Fe(2)–C(1)–Fe(1)	86·9 (5)
C(11)–Fe(1)–C(4)	128·9 (5)	O(1)–C(1)–Fe(1)	129·7 (9)
C(12)–Fe(1)–C(1)	170·0 (5)	O(1)–C(1)–Fe(2)	143·3 (10)
C(12)–Fe(1)–C(5)	94·8 (5)	C(51)–C(5)–Fe(1)	119·2 (8)
C(12)–Fe(1)–C(4)	90·0 (5)	C(4)–C(5)–Fe(1)	67·4 (6)
C(12)–Fe(1)–C(11)	95·5 (5)	Fe(2)–C(4)–Fe(1)	76·7 (3)
C(13)–Fe(1)–C(1)	90·1 (5)	C(5)–C(4)–Fe(1)	74·3 (7)
C(13)–Fe(1)–C(5)	163·6 (5)	C(5)–C(4)–Fe(2)	117·1 (8)
C(13)–Fe(1)–C(4)	127·7 (5)	C(41)–C(4)–Fe(1)	128·7 (8)
C(13)–Fe(1)–C(11)	102·6 (5)	C(41)–C(4)–Fe(2)	121·8 (8)
C(13)–Fe(1)–C(12)	93·4 (5)	O(11)–C(11)–Fe(1)	178·8 (12)
C(4)–Fe(2)–C(1)	84·1 (5)	O(12)–C(12)–Fe(1)	174·7 (11)
C(21)–Fe(2)–C(1)	102·0 (5)	O(13)–C(13)–Fe(1)	176·6 (12)
C(21)–Fe(2)–C(4)	92·0 (5)	O(21)–C(21)–Fe(2)	177·4 (13)
C(22)–Fe(2)–C(1)	157·7 (6)	O(22)–C(22)–Fe(2)	173·1 (13)
C(22)–Fe(2)–C(4)	93·0 (5)	O(23)–C(23)–Fe(2)	179·1 (13)
C(22)–Fe(2)–C(21)	100·1 (6)		

Discussion. The structure consists of discrete dinuclear molecules linked by van der Waals forces. A view of the molecule with the numbering of atoms is shown in Fig. 1. Atom parameters are in Table 1* and selected bond distances and angles are in Table 2. The molecule contains two organic bridges: 1-phenyl-1-propenyl and ethoxymethylidyne groups. The first is σ bonded to Fe(2) with an Fe(2)—C(4) distance of 2·014 (10) Å and unsymmetrically π bonded to Fe(1) with Fe(1)—C(4) 0·089 (21) Å shorter than Fe(1)—C(5), as is usual in other known complexes of this type (Iggo, Mays, Raithby & Hendrick, 1983 and references therein). The phenyl and methyl groups are in *cis* configuration, forming a torsion angle of −15·4 (9)°, similar to that observed in the 1,2-diphenylvinyl complex (Ros *et al.*, 1985), where this angle is −13·1°. The substitution of one phenyl by a methyl group in this ligand produces a shortening of the C(4)—C(5) [1·40 (1) and 1·47 (3) Å in the 1-phenyl-1-propenyl and 1,2-diphenylvinyl complexes, respectively] and Fe(1)—C(4) bond distances [2·092 (10) and 2·15 (2) Å, respectively], giving the ligand a geometry intermediate between that in the 1,2-diphenylvinyl complex and that in $Fe_2(CO)_5\{\mu\text{-}C(OC_2H_5)C[C(O)OCH_3]C[C(O)OCH_3]\}\{\mu\text{-}C(C_6H_5)\text{-}C(C_6H_5)H\}$ (Ros, Commenges, Mathieu, Solans & Font-Altaba, 1984).

The bridging ethoxymethylidyne group is bonded almost symmetrically to Fe(1) and Fe(2) and is situated on the opposite side to the vinyl group, minimizing interactions, as in the 1,2-diphenylvinyl complex.

The Fe—Fe bond length of 2·549 (2) Å is close to the Fe—Fe distances found for most structures containing the $Fe_2(CO)_6$ moiety (Krüger, Barnett & Brauer, 1978) and the bond distances and angles of each $Fe(CO)_3$ moiety have values typical of those reported in the literature.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and full bond length and angle data have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42232 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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The Structures of 2′-[1-(2-Pyridyl)ethylidene]-3-azabicyclo[3.2.2]nonane-3-carbothiohydrazide, $C_{16}H_{22}N_4S$, and -3-carboselenohydrazide, $C_{16}H_{22}N_4Se$*

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Abstract. $C_{16}H_{22}N_4S$ (I): $M_r = 302{\cdot}4$, monoclinic, Cc, $a = 6{\cdot}433$ (1), $b = 21{\cdot}796$ (3), $c = 11{\cdot}533$ (2) Å, $\beta = 90{\cdot}48$ (1)°, $V = 1616{\cdot}9$ Å^3, $Z = 4$, D_m(flotation) = 1·23 (1), $D_x = 1{\cdot}243$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}7107$ Å, $\mu = 1{\cdot}297$ mm^{-1}, $F(000) = 648{\cdot}0$, $T = 293$ K, $R = 0{\cdot}049$ for 991 observed reflections. $C_{16}H_{22}N_4Se$ (II): $M_r = 349{\cdot}3$, triclinic, $P\bar{1}$, $a = 8{\cdot}462$ (1), $b = 9{\cdot}178$ (1),

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