

The structure was determined by direct methods and refined by anisotropic full-matrix least squares on F . All C atoms were refined isotropically. All H atoms were located geometrically (C—H distance 0.98 Å) and not refined.

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Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55836 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SH1022]

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Structures of 1-Ferrocenyl-1-phenylethanol, Ferrocenyl(diphenyl)methanol and Ferrocene-1,1'-diylbis(diphenylmethanol)

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Abstract

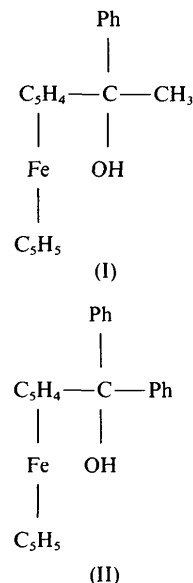
Racemic 1-ferrocenyl-1-phenylethanol, [(C₅H₅)Fe(C₅H₄)]CPhMeOH (I), crystallizes as discrete molecules which are not involved in hydrogen bonding; the shortest intermolecular O···O contact is 3.768(3) Å and the hydroxyl H atom is orientated towards the unsubstituted cyclopentadienyl ring. Ferrocenyl(diphenyl)methanol, [(C₅H₅)Fe(C₅H₄)]CPh₂OH (II) is hydrogen bonded to form centrosymmetric dimers with O···O 2.816(1) and H···O 2.52 Å. Ferrocene-1,1'-diylbis(diphenylmethanol), Fe[(C₅H₄)C(Ph)₂COH]₂ (III) crystallizes as a dimeric aggregate with the Fe atoms on twofold crystallographic symmetry axes and the four O atoms defin-

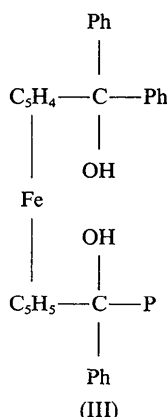
ing a folded trapezium with O···O 2.762(2), 2.714(2) and 2.865(2) Å. The four hydroxyl groups are disordered equally over two orientations such that there are two half-occupancy H-atom sites between each hydrogen-bonded oxygen pair.

Comment

Triphenylmethanol, Ph₃COH, crystallizes as hydrogen-bonded tetrameric aggregates with threefold crystallographic symmetry, each containing an approximately tetrahedral arrangement of O atoms with necessarily disordered hydroxyl H atoms (Ferguson, Gallagher, Glidewell, Low & Scrimgeour, 1992). These aggregates are significantly different from the cyclic tetrameric aggregates found in triphenylsilanol, Ph₃SiOH (Puff, Braun & Reuter, 1991), and in triphenylgermanol, Ph₃GeOH (Ferguson, Gallagher, Murphy, Spalding, Glidewell & Holden, 1992), whose graph set (Etter, MacDonald & Bernstein, 1990) is $R_4^4(8)$. Triphenylmethanol can act as a host towards guest molecules such as methanol and dimethyl sulfoxide (Weber, Skobridis & Goldberg, 1989) albeit with hydrogen-bonding arrangements wholly different from that in unsolvated Ph₃COH. The diol *m*-bis(diphenylhydroxymethyl)benzene, which forms similar host/guest aggregates with a range of different guest species, crystallizes as centrosymmetric dimers (Toda, Kai, Toyotaka, Yip & Mak, 1989) with graph set $R_2^2(16)$.

In order to assess the effect on the hydrogen-bonding patterns in the crystal lattice in alcohols of this general type by altering the steric demands at the central C atom, we have now determined the structures of racemic 1-ferrocenyl-1-phenylethanol, [(C₅H₅)Fe(C₅H₄)]CPhMeOH (I); ferrocenyl(diphenyl)methanol, [(C₅H₅)Fe(C₅H₄)]CPh₂OH (II); and ferrocene-1,1'-diylbis(diphenylmethanol), Fe[(C₅H₄)C(Ph)₂COH]₂ (III).





[(C₅H₅)Fe(C₅H₄)]CPhMeOH (I) (Fig. 1) crystallizes in the centrosymmetric space group $P2_1/n$ with one molecule in the asymmetric unit; consequently *R* and *S* enantiomers are present in equal numbers and related by crystallographic centres of inversion. The unsubstituted C₅H₅ ring is disordered over two sites with occupancies of 87 and 13% for the major and minor components. There is no hydrogen bonding in the crystal structure, presumably because the conformation adopted by the ferrocenyl, methyl and phenyl substituents in the solid state shields the hydroxyl group; the distance between the nearest hydroxyl O atoms O...O, 3.768(3) Å, precludes O—H...O hydrogen bonding. Instead the hydroxyl H atom (which was clearly located in difference maps) is orientated towards the C₅H₅ cyclopentadienyl ring with dimensions O—H 0.76 Å and C—O—H 105°.

Ferrocenyl(diphenyl)methanol (II) (Fig. 2) differs from the triphenylmethanol molecule by replacement of one of the phenyl groups by a ferrocenyl group. As in 1-ferrocenyl-1-phenylethanol (I) the hydroxyl H atom, the

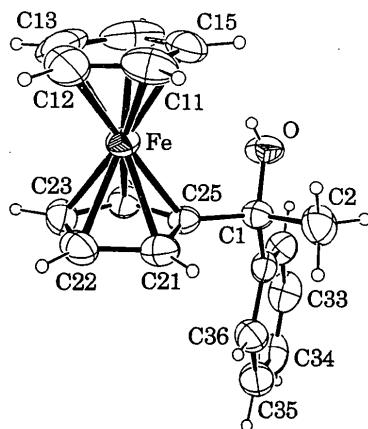


Fig. 1. A view of 1-ferrocenyl-1-phenylethanol (I). The non-H atoms are shown with thermal ellipsoids drawn at the 50% probability level. For clarity the minor cyclopentadienyl site has been omitted and the H atoms are drawn as small spheres of an arbitrary size.

position of which was clearly located from a difference map, is directed towards the unsubstituted C₅H₅ ring in the same molecule (Fig. 2). An unexpected feature in (II) is that the shortest O...O contact between adjacent molecules related by an inversion centre is 2.816(1) Å with O—H 0.76, H...O 2.52 Å, C—O—H 104, O—H...O 106°. The graph set for this centrosymmetric hydrogen-bonded motif is $R_2^2(4)$. It is of interest that the closest intermolecular approach in (I) [3.768(3) Å] is appreciably longer than in (II) [2.816(2) Å] although the steric congestion about the unique central C atom in (II) is apparently greater than in (I).

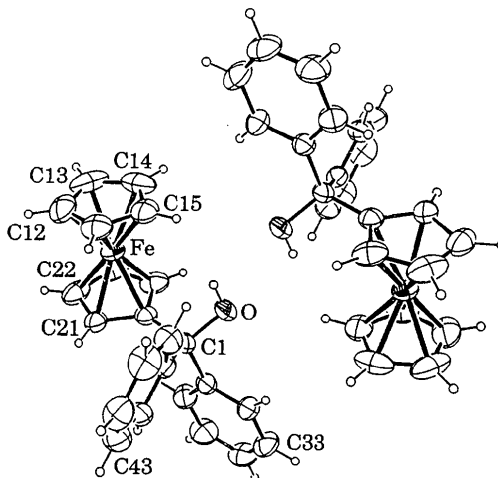


Fig. 2. A view of ferrocenyl(diphenyl)methanol (II) as the dimeric aggregate. Ellipsoid conditions are as for Fig. 1.

Ferrocene-1,1'-diylbis(diphenylmethanol) (III) differs from (I) and (II) in that it contains two carbinol moieties on the electroactive molecular core. The asymmetric unit in this structure comprises two independent half-molecules with both Fe atoms lying on a crystallographic twofold axis. The molecules are hydrogen bonded to form a dimeric structural motif, graph set $R_4^4(8)$ (Fig. 3) which is quite different from that found in triphenylmethanol. The O atoms form a flattened trapezium with O...O hydrogen-bond distances 2.762(2) (×2), 2.714(2) and 2.865(2) Å. Difference-density maps showed that in both molecules the hydroxyl H atom is disordered equally over two sites, each directed towards neighbouring hydroxyl O atoms as shown in Fig. 3. The O—H distances are in the range 0.60 to 0.70 Å while the C—O—H angles are between 116.3 and 125.7°. The positional disorder of the H atoms demands that the hydrogen-bonded motif describes either a clockwise or a counterclockwise pattern; hence each individual dimeric aggregate must be chiral, with equal numbers of the two enantiomers rendering the crystal as a whole achiral.

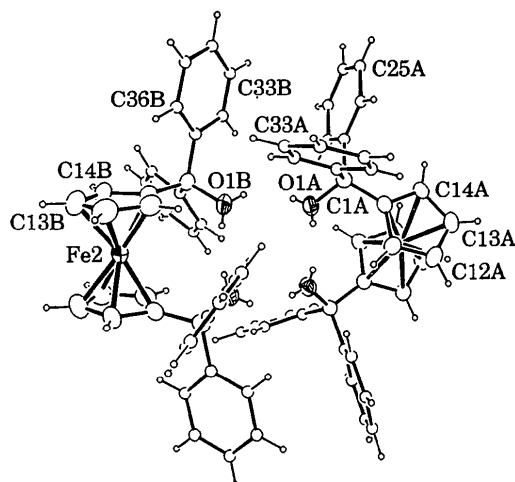


Fig. 3. A view of the two independent ferrocene-1,1'-diylbis(diphenylmethanol) molecules (III). The non-H atoms are depicted with thermal ellipsoids drawn at the 35% probability level. For clarity the H atoms are drawn as small spheres of an arbitrary size. A crystallographic twofold axis passes through the two Fe atoms.

In (I) the major conformer has a $3.4(3)^\circ$ angle between the C_5 planes of the ferrocene moiety which is within $8.8(3)^\circ$ of an eclipsed conformation [the relevant values for the minor isomer are $7(1)$ and $25(1)^\circ$, respectively]; in (II) the corresponding dihedral angle is $3.2(1)^\circ$ and the rotation of the C_5 planes out of an eclipsed conformation is $17.6(1)^\circ$. The two independent ferrocene moieties in (III) have $3.7(2)$ and $4.6(2)^\circ$ dihedral angles between their symmetry related C_5 planes (for molecules *A* and *B*, respectively). Although the C_5 rings are within $3.2(2)$ and $7.7(2)^\circ$ of being eclipsed, the conformation adopted is such that the exocyclic C atoms are rotated about a line joining the ring centroids through $-66.5(2)$ and $62.1(2)^\circ$ from an eclipsed conformation.

The molecular dimensions for (I), (II) and (III) are summarized in Table 4 and show no unusual features. There is no solvent of crystallization present in any of the lattices and an examination of the structures using *PLATON* (Spek, 1991) reveals no potential volume for any solvent molecules.

Experimental

Compound (I)

Crystal data

$C_{18}H_{18}FeO$

$M_r = 305.53$

Monoclinic

$P2_1/n$

$a = 13.7594(6) \text{ \AA}$

$b = 5.9786(3) \text{ \AA}$

$c = 17.8430(12) \text{ \AA}$

$\beta = 106.115(5)^\circ$

$V = 1410.12(13) \text{ \AA}^3$

$Z = 4$

$D_x = 1.439 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.70930 \text{ \AA}$

Cell parameters from 25 reflections

$\theta = 10.5\text{--}15.8^\circ$

$\mu = 1.06 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Block

$0.30 \times 0.25 \times 0.20 \text{ mm}$

Red-brown

Data collection

Enraf-Nonius CAD-4 diffractometer

$\omega/2\theta$ scans

Absorption correction:

empirical

$T_{\min} = 0.6940$, $T_{\max} =$

0.7878

5831 measured reflections

3069 independent reflections

2293 observed reflections

$[I_{\text{net}} > 3.0\sigma(I_{\text{net}})]$

$R_{\text{int}} = 0.012$

$\theta_{\text{max}} = 26.91^\circ$

$h = -17 \rightarrow 16$

$k = 0 \rightarrow 7$

$l = 0 \rightarrow 22$

3 standard reflections

frequency: 120 min

intensity variation: -2.0%

Refinement

Refinement on F^2

Final $R = 0.025$

$wR = 0.034$

$S = 1.25$

2293 reflections

197 parameters

O—H from Δ maps; C—H,

0.95 \AA riding

$w = 1/[\sigma^2(F) + 0.0004F^2]$

$(\Delta/\sigma)_{\text{max}} = 0.023$

$\Delta\rho_{\text{max}} = 0.23 \text{ e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.20 \text{ e \AA}^{-3}$

Extinction correction: Larson (1970)

Extinction coefficient:

10162 (3776)

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (\AA^2) for (I)

$$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
Fe	0.286552 (22)	0.20604 (5)	0.076972 (16)	0.03530 (17)
O	0.09022 (13)	-0.0505 (3)	-0.06064 (10)	0.0486 (10)
C1	0.11628 (15)	0.1617 (3)	-0.08809 (12)	0.0349 (10)
C2	0.03753 (18)	0.3333 (4)	-0.08193 (14)	0.0488 (14)
C11	0.2308 (6)	0.3070 (7)	0.1668 (3)	0.060 (3)
C12	0.3343 (5)	0.2647 (15)	0.19439 (21)	0.083 (4)
C13	0.3490 (5)	0.0433 (18)	0.1788 (3)	0.091 (4)
C14	0.2557 (7)	-0.0535 (6)	0.1408 (3)	0.073 (3)
C15	0.18242 (23)	0.1115 (12)	0.13396 (18)	0.0522 (19)
C21	0.25511 (17)	0.4417 (4)	-0.00932 (12)	0.0405 (12)
C22	0.36090 (18)	0.4307 (4)	0.02694 (12)	0.0479 (13)
C23	0.39360 (17)	0.2121 (5)	0.01785 (13)	0.0483 (13)
C24	0.30801 (17)	0.0860 (4)	-0.02451 (12)	0.0411 (12)
C25	0.22180 (15)	0.2281 (4)	-0.04103 (11)	0.0328 (10)
C31	0.11470 (15)	0.1291 (4)	-0.17322 (11)	0.0350 (10)
C32	0.07987 (18)	-0.0649 (4)	-0.21391 (13)	0.0467 (13)
C33	0.08037 (20)	-0.0855 (5)	-0.29133 (15)	0.0582 (15)
C34	0.11442 (19)	0.0833 (6)	-0.32861 (13)	0.0583 (17)
C35	0.14936 (19)	0.2772 (5)	-0.28892 (14)	0.0548 (15)
C36	0.14887 (17)	0.3004 (4)	-0.21195 (13)	0.0451 (12)
C11A	0.2998 (23)	0.3065 (22)	0.1852 (11)	0.023 (4)
C12A	0.3697 (10)	0.135 (3)	0.1864 (12)	0.041 (6)
C13A	0.3253 (20)	-0.0614 (23)	0.1553 (13)	0.049 (7)
C14A	0.2102 (15)	-0.003 (5)	0.1312 (12)	0.046 (7)
C15A	0.2009 (15)	0.224 (5)	0.1513 (13)	0.033 (5)
HO1	0.105	-0.040	-0.017	0.0620

Compound (II)

Crystal data

$C_{23}H_{20}FeO$

$M_r = 368.25$

$D_x = 1.372 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

Triclinic

$P\bar{1}$
 $a = 7.8205$ (4) Å
 $b = 11.0274$ (4) Å
 $c = 11.7715$ (6) Å
 $\alpha = 105.707$ (4)°
 $\beta = 106.128$ (5)°
 $\gamma = 102.403$ (4)°
 $V = 891.45$ (7) Å³
 $Z = 2$

Data collection

Enraf-Nonius CAD-4
 diffractometer
 $\omega/2\theta$ scans
 Absorption correction:
 empirical
 $T_{\min} = 0.7666$, $T_{\max} =$
 0.8246
 5174 measured reflections
 5174 independent reflections

Refinement

Refinement on F
 Final $R = 0.030$
 $wR = 0.049$
 $S = 1.46$
 4294 reflections
 227 parameters
 O—H from Δ maps; C—H,
 0.95 Å riding
 $w = 1/[\sigma^2(F) + 0.0008F^2]$
 $(\Delta/\sigma)_{\max} < 0.001$

$\lambda = 0.70930$ Å
 Cell parameters from 25
 reflections
 $\theta = 9.50$ – 25.50°
 $\mu = 0.85$ mm⁻¹
 $T = 293$ K
 Block
 $0.45 \times 0.35 \times 0.30$ mm
 Orange

4294 observed reflections
 $[I_{\text{net}} > 3.0\sigma(I_{\text{net}})]$
 $\theta_{\max} = 29.92^\circ$
 $h = -10 \rightarrow 10$
 $k = 0 \rightarrow 15$
 $l = -16 \rightarrow 15$
 3 standard reflections
 frequency: 60 min
 intensity variation: none

$\Delta\rho_{\max} = 0.35$ e Å⁻³
 $\Delta\rho_{\min} = -0.25$ e Å⁻³
 Extinction correction: Larson
 (1970)
 Extinction coefficient:
 9806 (914)
 Atomic scattering factors
 from *International Tables*
 for *X-ray Crystallogra-*
phy (1974, Vol. IV, Table
 2.2B)

Compound (III)

Crystal data

$C_{36}H_{30}FeO_2$
 $M_r = 550.47$
 Monoclinic
 $C2/c$
 $a = 13.2841$ (6) Å
 $b = 23.2856$ (12) Å
 $c = 18.8106$ (8) Å
 $\beta = 105.482$ (4)°
 $V = 5607.5$ (5) Å³
 $Z = 8$
 $D_x = 1.304$ Mg m⁻³

Data collection

Enraf-Nonius CAD-4
 diffractometer
 $\omega/2\theta$ scans
 Absorption correction:
 empirical
 $T_{\min} = 0.8333$, $T_{\max} =$
 0.8774
 6273 measured reflections
 6095 independent reflections
 4121 observed reflections
 $[I_{\text{net}} > 3.0\sigma(I_{\text{net}})]$

Refinement

Refinement on F
 Final $R = 0.036$
 $wR = 0.049$
 $S = 1.37$
 4121 reflections
 354 parameters
 C—H, 0.95 Å riding; O—H
 from Δ maps
 $w = 1/[\sigma^2(F) + 0.0006F^2]$
 $(\Delta/\sigma)_{\max} = 0.007$

Mo $K\alpha$ radiation

$\lambda = 0.70930$ Å
 Cell parameters from 25
 reflections
 $\theta = 10.00$ – 25.00°
 $\mu = 0.57$ mm⁻¹
 $T = 293$ K
 Block
 $0.75 \times 0.60 \times 0.60$ mm
 Orange

$R_{\text{int}} = 0.018$
 $\theta_{\max} = 26.91^\circ$
 $h = -16 \rightarrow 16$
 $k = 0 \rightarrow 29$
 $l = 0 \rightarrow 23$
 3 standard reflections
 frequency: 120 min
 intensity variation: -2.0%

$\Delta\rho_{\max} = 0.21$ e Å⁻³
 $\Delta\rho_{\min} = -0.28$ e Å⁻³
 Extinction correction: Larson
 (1970)
 Extinction coefficient:
 4275 (1274)
 Atomic scattering factors
 from *International Tables*
 for *X-ray Crystallogra-*
phy (1974, Vol. IV, Table
 2.2B)

Table 2. Fractional atomic coordinates and equivalent isotropic thermal parameters (Å²) for (II)

$$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
Fe	0.80686 (3)	0.347480 (17)	0.224884 (19)	0.03852 (13)
O	0.67955 (15)	0.58033 (11)	0.08724 (10)	0.0506 (6)
C1	0.83751 (17)	0.63797 (12)	0.20420 (12)	0.0352 (6)
C11	0.58540 (25)	0.31522 (18)	0.28581 (18)	0.0562 (10)
C12	0.6864 (3)	0.22922 (22)	0.30722 (21)	0.0678 (13)
C13	0.6845 (3)	0.15185 (17)	0.1896 (3)	0.0764 (16)
C14	0.5807 (3)	0.19263 (19)	0.09583 (19)	0.0677 (11)
C15	0.51897 (22)	0.29383 (17)	0.15655 (18)	0.0535 (9)
C21	1.01030 (19)	0.52109 (14)	0.34689 (15)	0.0432 (7)
C22	1.08970 (23)	0.41550 (18)	0.32494 (22)	0.0600 (11)
C23	1.0546 (3)	0.36274 (17)	0.19415 (23)	0.0648 (13)
C24	0.9547 (3)	0.43523 (16)	0.13356 (17)	0.0528 (9)
C25	0.92675 (18)	0.53351 (13)	0.22774 (13)	0.0373 (7)
C31	0.97241 (19)	0.74859 (13)	0.18654 (12)	0.0370 (6)
C32	0.90303 (22)	0.84081 (15)	0.14459 (14)	0.0457 (8)
C33	1.0218 (3)	0.94478 (16)	0.13074 (17)	0.0573 (11)
C34	1.2089 (3)	0.95820 (17)	0.15892 (17)	0.0617 (11)
C35	1.2783 (3)	0.86868 (20)	0.20082 (19)	0.0612 (11)
C36	1.16077 (22)	0.76284 (17)	0.21437 (16)	0.0507 (9)
C41	0.77634 (18)	0.69752 (13)	0.31412 (12)	0.0362 (7)
C42	0.90471 (21)	0.80034 (15)	0.42249 (14)	0.0452 (8)
C43	0.8520 (3)	0.85157 (17)	0.52458 (16)	0.0568 (11)
C44	0.6727 (3)	0.79912 (20)	0.52030 (19)	0.0612 (12)
C45	0.54613 (25)	0.69710 (21)	0.41465 (20)	0.0608 (12)
C46	0.59592 (21)	0.64693 (17)	0.31113 (17)	0.0499 (9)
HO1	0.640	0.509	0.083	0.0528

Table 3. Fractional atomic coordinates and equivalent isotropic thermal parameters (Å²) for (III)

$$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
Fe1	0.00000	0.365480 (18)	0.25000	0.0349 (3)
O1A	0.05084 (15)	0.22975 (7)	0.19629 (9)	0.0535 (10)
C11A	-0.10339 (17)	0.32652 (10)	0.16306 (12)	0.0418 (11)
C12A	-0.13097 (19)	0.38478 (11)	0.16821 (13)	0.0500 (13)
C13A	-0.04583 (22)	0.41897 (10)	0.16176 (13)	0.0514 (15)
C14A	0.03433 (19)	0.38204 (10)	0.15238 (12)	0.0437 (13)
C15A	-0.00073 (17)	0.32427 (9)	0.15342 (11)	0.0366 (11)
C1A	0.05253 (17)	0.26957 (9)	0.13895 (11)	0.0373 (11)
C21A	0.16569 (18)	0.28055 (10)	0.13846 (13)	0.0443 (13)
C22A	0.24542 (22)	0.27318 (16)	0.20168 (16)	0.0765 (21)
C23A	0.3482 (3)	0.28287 (20)	0.20081 (23)	0.106 (3)
C24A	0.3722 (3)	0.29949 (18)	0.1378 (3)	0.093 (3)
C25A	0.2946 (3)	0.30684 (17)	0.07551 (21)	0.0865 (24)
C26A	0.19089 (22)	0.29750 (15)	0.07545 (16)	0.0692 (19)
C31A	-0.00877 (18)	0.24281 (10)	0.06505 (12)	0.0438 (12)
C32A	0.00569 (25)	0.18546 (12)	0.05098 (15)	0.0620 (16)
C33A	-0.0491 (3)	0.16084 (14)	-0.01549 (20)	0.0816 (24)
C34A	-0.1161 (3)	0.19305 (21)	-0.06793 (20)	0.094 (3)
C35A	-0.1293 (3)	0.24931 (21)	-0.05547 (18)	0.106 (3)
C36A	-0.0761 (3)	0.27397 (15)	0.01084 (16)	0.0783 (19)

Fe2	0.00000	-0.027433 (21)	0.25000	0.0525 (3)
O1B	0.08988 (15)	0.11400 (7)	0.22158 (11)	0.0653 (12)
C11B	0.01329 (23)	0.00908 (13)	0.15451 (15)	0.0599 (16)
C12B	-0.0127 (3)	-0.04931 (15)	0.14299 (19)	0.0785 (22)
C13B	0.0677 (3)	-0.08152 (13)	0.19150 (21)	0.0784 (23)
C14B	0.14239 (22)	-0.04303 (11)	0.23306 (16)	0.0598 (17)
C15B	0.10919 (20)	0.01393 (10)	0.21042 (13)	0.0484 (13)
C1B	0.16801 (19)	0.06976 (10)	0.23244 (14)	0.0501 (14)
C21B	0.23284 (20)	0.06863 (11)	0.31259 (15)	0.0521 (14)
C22B	0.20595 (22)	0.10055 (13)	0.36652 (18)	0.0661 (18)
C23B	0.2655 (3)	0.09817 (17)	0.43950 (20)	0.0885 (25)
C24B	0.3519 (3)	0.06354 (19)	0.45919 (19)	0.093 (3)
C25B	0.3808 (3)	0.03205 (16)	0.40646 (19)	0.0839 (21)
C26B	0.32353 (23)	0.03484 (13)	0.33397 (16)	0.0660 (17)
C31B	0.23728 (23)	0.08194 (12)	0.18059 (17)	0.0629 (18)
C32B	0.3100 (4)	0.12464 (18)	0.1970 (3)	0.111 (3)
C33B	0.3743 (5)	0.1361 (3)	0.1509 (4)	0.147 (5)
C34B	0.3656 (5)	0.1055 (3)	0.0884 (4)	0.134 (5)
C35B	0.2932 (5)	0.0644 (3)	0.0711 (3)	0.132 (4)
C36B	0.2293 (3)	0.05215 (18)	0.11698 (22)	0.099 (3)
HOA1	0.035	0.235	0.224	0.0642
HOA2	0.069	0.206	0.194	0.0642
HOB1	0.045	0.108	0.231	0.0770
HOB2	0.104	0.142	0.212	0.0770

Table 4. Summary of dimension ranges (\AA , $^\circ$) for (I)–(III)

	(I)	(II)	(III)
Fe—C _{cp}	2.029(4)–2.053(3)	2.037(2)–2.057(2)	2.033(3)–2.052(2)
Mean	2.043(3)	2.048(2)	2.040(3)
C _{cp} —C _{cp}	1.406(4)–1.425(3)	1.388(3)–1.430(2)	1.405(5)–1.426(3)
Mean	1.419(3)	1.414(3)	1.418(4)
C _{cp} —C _{sp³}	1.515(3)	1.517(2)	1.516(3), 1.517(3)
C _{sp³} —C _{sp³}	1.518(3)	—	—
C _{ar} —C _{sp³}	1.526(3)	1.533(2)–1.534(2)	1.524(3)–1.543(3)
C _{sp³} —O	1.441(3)	1.439(2)	1.427(3)–1.438(3)
C _{ar} —C _{ar}	1.362(4)–1.389(3)	1.363(4)–1.398(2)	1.334(10)–1.405(4)
Mean	1.380(3)	1.384(3)	1.374(6)
O—C—C _{sp³}	108.69(17)	—	—
O—C—C _{ar}	109.46(17)	105.1(1), 110.4(1)	106.0(2)–107.9(2)
O—C—C _{cp}	106.83(17)	109.9(1)	107.9(2), 110.4(2)

1-Ferrocenyl-1-phenylethanol (I) was obtained from the reaction of acetylferrocene with phenyllithium, and crystals were grown by slow evaporation of a solution in CH₂Cl₂. Ferrocenyl(diphenyl)methanol (II) was obtained from the reaction of benzoylferrocene with phenyllithium; crystals were obtained by slow evaporation of a solution in CH₂Cl₂. Ferrocene-1,1'-diylbis(diphenylmethanol) (III) was obtained from the reaction of 1,1'-dibenzoylferrocene with phenyllithium; crystals were grown by slow evaporation of a solution in dichloromethane/petroleum (b.p. 313–333 K).

Data collection and cell refinement: Enraf-Nonius CAD-4 software. Data reduction, program used to solve and refine structure, software used to prepare material for publication: NRCVAX (Gabe, Le Page, Charland, Lee & White, 1989).

The space group for (I) was determined unambiguously from the systematic absences ($h0l$ absent if $h+l = 2n+1$, $0k0$ absent if $k = 2n+1$) as $P2_1/n$. Molecule (II) crystallized in the triclinic system; space group $P1$ was assumed and confirmed by the analysis. The systematic absences for (III) (hkl absent if $h+k = 2n+1$, $h0l$ absent if $l = 2n+1$) allow the space group to be either $C2/c$ or Cc ; the former was assumed and confirmed by the analysis. In molecule (I) it was clear from early difference maps that the unsubstituted C₅ ring was disordered over two sites; occupancies of 0.87 and 0.13 were estimated from Fourier and difference Fourier maps. In all three structures, the H atoms attached to the C atoms were clearly visible in difference maps [except for the minor C₅ ring of (I)]; they were positioned geometrically (C—H 0.95 \AA) and included as riding atoms in the structure factor

calculations. The hydroxyl H atoms [disordered equally in (III) over two sites] were included in the structure factor calculations at the positions obtained from difference maps. The ORTEP diagrams were prepared using ORTEPII (Johnson, 1976).

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55804 (77 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: L11034]

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Structure of Dimethyl 9-Methyl-9,10-dihydro-9,10-ethenoanthracene-11,12-dicarboxylate

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Abstract

There are two molecules per asymmetric unit and the two ester groups have different orientations in each of them; the C=C—C=O torsion angles are 1.9 and 2.4(6) $^\circ$ for the groups remote from and 91.7 and -91.4(5) $^\circ$ for the