C44 .	0.2633 (4)	0.3884 (7)	0.1701 (5)	0.075 (3)
C45	0.2931 (4)	0.3646 (7)	0.1203 (5)	0.068 (3)
C46	0.3516 (3)	0.3984 (6)	0.1127 (4)	0.050 (2)
Sb1	0.15948 (3)	0.10900 (4)	0.09726 (3)	0.0542 (2)
F1	0.1044 (2)	0.0793 (4)	0.1543 (2)	0.085 (2)
F2	0.2199 (2)	0.1141 (5)	0.1598 (2)	0.093 (2)
F3	0.2138 (3)	0.1412 (7)	0.0404 (3)	0.143 (3)
F4	0.0989 (2)	0.1043 (5)	0.0355 (2)	0.092 (2)
F5	0.1738 (4)	−0.0164 (5)	0.0852 (4)	0.141 (3)
F6	0.1438 (3)	0.2347 (4)	0.1088 (3)	0.107 (2)

Table 2. *Selected geometric parameters (Å, °)*

Au1—P1	2.311 (2)	P2—C31	1.801 (8)
Au2—P2	2.307 (2)	P2—C41	1.818 (7)
Au1···Au2	6.911 (2)	C1—C2	1.525 (10)
P1—C1	1.818 (8)	C2—C3	1.519 (10)
P1—C11	1.802 (8)	C3—C4	1.507 (10)
P1—C21	1.817 (8)	C4—C5	1.497 (10)
P2—C6	1.804 (7)	C5—C6	1.535 (10)
P1—Au1—P1^i	174.2 (1)	C11—P1—C21	108.0 (3)
P2—Au2—P2^i	172.7 (1)	Au2—P2—C6	114.4 (2)
Au1—P1—C1	113.9 (3)	Au2—P2—C31	115.3 (3)
Au1—P1—C11	113.6 (3)	Au2—P2—C41	109.1 (2)
Au1—P1—C21	108.6 (3)	C6—P2—C31	104.8 (3)
C1—P1—C11	105.5 (3)	C6—P2—C41	107.4 (3)
C1—P1—C21	106.9 (4)	C31—P2—C41	105.3 (3)
P1^i—Au1—P1—C1	−50.9 (3)	C2—C3—C4—C5	−177.7 (7)
P2^i—Au2—P2—C6	−50.8 (3)	C3—C4—C5—C6	−177.6 (6)
Au1—P1—C1—C2	52.9 (6)	C4—C5—C6—P2	−75.9 (8)
P1—C1—C2—C3	−72.0 (9)	Au2—P2—C6—C5	51.2 (6)
C1—C2—C3—C4	−170.4 (7)		

Symmetry code: (i) $1 - x, y, \frac{1}{2} - z$.

It became obvious from examination of $\Delta\rho$ maps that there were disordered solvent molecules in the crystal lattice. The crystals prepared for the analysis had been grown from pure acetonitrile, which is thus the only choice, but the geometry pattern of the 'solvent' peaks in the difference map did not correspond with any ordered orientation of acetonitrile molecules. Accordingly, the contribution of the density of the disordered solvent molecules was subtracted from the measured structure factors using the *SQUEEZE* option (Spek, 1994) in *PLATON*95 (Spek, 1995*a*). This indicated that the occupancy of the disordered acetonitrile had a value of 0.8 in the asymmetric unit with a rough error estimate of <0.1. Subsequent refinement then converged with slightly lower *R* factors than when the disordered solvent had been allowed for as a set of 'carbon' peaks; there were no significant changes in the geometry of the gold-containing cation.

Data collection: *DIFRAC* with profile analysis (Gabe & White, 1993). Cell refinement: *DIFRAC*. Data reduction: *DATRD*2 in *NRCVAX*94 (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *SOLVER* in *NRCVAX*94. Program(s) used to refine structure: *NRCVAX*94; *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *NRCVAX*94; *ORTEP*II (Johnson, 1976) in *PLATON* (Spek, 1995*a*); *PLUTON* (Spek, 1995*b*). Software used to prepare material for publication: *NRCVAX*94; *SHELXL*93.

GF thanks NSERC (Canada) for research grants. TRS thanks Johnson Matthey PLC for the loan of gold salts.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1344). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2-Ferrocenyl-3-phenylquinoxaline

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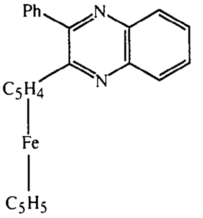
Abstract

In the title molecule, $[Fe(C_5H_5)(C_{19}H_{13}N_2)]$, the heterocyclic ring is almost coplanar with the cyclopentadienyl ring to which it is bonded [dihedral angle 4.1 (1)°]; the phenyl ring is almost orthogonal to the heterocyclic ring [dihedral angle 87.6 (1)°].

Comment

Following the observation of a very wide range of hydrogen-bonding patterns in adducts of ferrocenyl alcohols with amines, where the ferrocene acts as a hydrogen-bond donor (Ferguson, Gallagher, Glidewell & Zakaria, 1993; Glidewell, Ferguson, Lough & Zakaria, 1994; Ferguson, Glidewell, Lewis & Zakaria, 1995), we embarked on a study of functionalized ferrocenes which are potentially strong hydrogen-bond acceptors. We have recently reported the structure of one such ferrocene, 2-ferrocenylquinoxaline (Ferguson, Glidewell & Scott, 1995), and here we report the structure of a related compound, 2-ferrocenyl-3-phenylquinoxaline, (I), in which the features of interest, apart from confirmation of the molecular constitution, include the relative orientations of the ferrocenyl, phenyl and quinoxaline portions of the molecule and the nature of any intermolecular interactions.



(I)

Within the ferrocene fragment, the mean C—C distances in the substituted and unsubstituted cyclopentadienyl rings are 1.420 (11) and 1.404 (9) Å, respectively; similarly, the corresponding mean Fe—C distances for the two rings are identical within experimental error [2.038 (5) and 2.039 (7) Å]. The quinoxaline fragment shows marked bond fixation of the naphthalene type, as also observed both in 2-ferrocenylquinoxaline (Ferguson, Glidewell & Scott, 1995) and in hexachloroquinoxaline (Vermeulen & Huiszoon, 1979): in (I), the C5—C6 and C9—C10 bonds are significantly longer than the C6—C7 and C8—C9 bonds, and the N1—C10 and N4—C5 bonds are significantly longer than the N1—C2 and C3—N4 bonds.

The internal angles at the N atoms, C2—N1—C10 and C3—N4—C5, are fairly typical of those found in simple pyrazines and quinoxalines; however, none of the N—C—C angles in the pyrazino ring shows the large enhancement over 120° typical of such angles, and observed for the N4—C3—C2 angle [123.6 (2)°] in 2-ferrocenylquinoxaline (Ferguson, Glidewell & Scott, 1995). In contrast, the C—C—C angles exterior to the pyrazino ring both have values significantly greater than 120°, so that the N1—C2—C11 and N4—C3—C31 angles are correspondingly reduced; these data clearly indicate the repulsive intramolecular interaction between the adjacent ferrocenyl and phenyl substituents.

The quinoxaline ring is almost coplanar with the C1*n* (*n* = 1–5) ring of the ferrocene fragment (Fig. 1); the dihedral angle between these two rings is 4.1 (1)°. Similar near coplanarity has been observed both in 2-ferrocenylquinoxaline and in a bisquinolylferrocene (Gelin & Thummel, 1992). The phenyl ring, on the other hand, is almost orthogonal to the quinoxaline ring with a dihedral angle between these two planes of 87.6 (1)°. Inspection of a space-filling diagram of the molecular structure shows (i) that the phenyl group is sterically constrained by the presence of the ferrocene group to lie almost orthogonal to the quinoxaline, and (ii) that H12 (bonded to C12) on the ferrocene lies close to the shielding cone of the phenyl ring; the closest intramolecular distances involving H12 and the phenyl ring are H12···C31 2.54, H12···H36 2.76 and H12···C32 2.85 Å, but the ^{1}H and ^{13}C NMR spectra of (I) show no evidence for unusual shielding of H12 or C12.

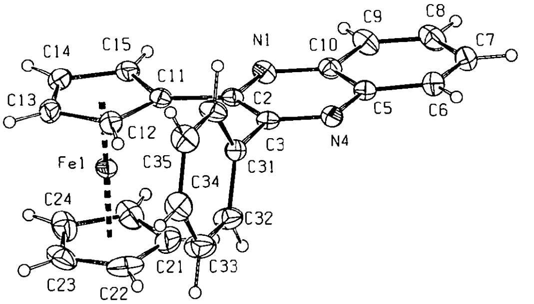


Fig. 1. A view of the *S* enantiomer of (I) with our numbering scheme (except for C25 and C36). Ellipsoids are drawn at the 30% probability level.

In the ferrocene fragment, the mean value of the torsion angles C1*n*—*Cg*1—*Cg*2—C2*n* is −6.5 (3)° (*Cg*1 and *Cg*2 are the centroids of the two rings) indicating an average conformation of the two cyclopentadiene rings which is close to eclipsed, as typically found in monosubstituted ferrocenes. The displacement parameters of the atoms C2*n* (*n* = 1–5) indicate a significant torsional motion of this ring about the local fivefold axis; similarly, the displacement parameters of the phenyl ring indicate a torsional oscillation about the C3—C31 bond. The angle *Cg*1—Fe—*Cg*2 is 179.1 (1)°, while the dihedral angle between the best planes through C1*n* and C2*n* is 0.9 (2)°.

The molecules are packed such that C33—H33 of the molecule at (x, y, z) points towards the C5—C10 bond in the molecule at $(\frac{1}{2} - x, \frac{1}{2} - y, -z)$, and C35—H35 in the molecule at (x, y, z) points at the C11—C15 bond in the molecule at $(1 - x, -y, -z)$, but none of these contacts is significantly shorter than the sum of van der Waals radii (Bondi, 1964).

Experimental

Compound (I) was prepared by the reaction of 1-ferrocenyl-2-phenylethanedione with 1,2-diaminobenzene in the absence of solvent, followed by chromatography on alumina with light petroleum (b.p. 313–333 K) as eluent. Crystals suitable for single-crystal diffraction were grown by slow evaporation of a solution in hexane/dichloromethane.

Crystal data

[Fe(C$_5$H$_5$)(C$_{19}$H$_{13}$N$_2$)]
$M_r = 390.25$
Monoclinic
$C2/c$
$a = 20.455$ (2) Å
$b = 8.5280$ (8) Å
$c = 20.338$ (2) Å
$\beta = 92.237$ (7)°
$V = 3545.0$ (5) Å^3
$Z = 8$
$D_x = 1.462$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 11.5\text{–}15.8°$
$\mu = 0.861$ mm^{-1}
$T = 294$ (1) K
Plate
0.42 × 0.41 × 0.07 mm
Red

Data collection

Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: three ψ scans at 4° steps (North, Phillips & Mathews, 1968)
$T_{\min} = 0.638$, $T_{\max} = 0.820$
3942 measured reflections
3845 independent reflections
2751 observed reflections [$I > 2\sigma(I)$]

$R_{\rm int} = 0.020$
$\theta_{\max} = 26.91°$
$h = -26 \rightarrow 26$
$k = 0 \rightarrow 10$
$l = 0 \rightarrow 25$
3 standard reflections frequency: 120 min intensity decay: 1.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0340$
$wR(F^2) = 0.0883$
$S = 1.012$
3845 reflections
244 parameters
H atoms riding [*SHELXL*93 (Sheldrick, 1993) defaults, C—H = 0.93 Å]

$w = 1/[\sigma^2(F_o^2) + (0.0491P)^2 + 0.1242P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = -0.001$
$\Delta\rho_{\max} = 0.276$ e Å^{-3}
$\Delta\rho_{\min} = -0.312$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Fe1	0.42851 (1)	0.04454 (4)	0.11897 (1)	0.03234 (10)
N1	0.35043 (9)	0.4338 (2)	0.15955 (8)	0.0361 (4)
C2	0.37272 (9)	0.3878 (2)	0.10233 (9)	0.0306 (4)
C3	0.34182 (9)	0.4421 (2)	0.04158 (9)	0.0311 (4)
N4	0.29202 (8)	0.5396 (2)	0.04077 (8)	0.0358 (4)
C5	0.26949 (10)	0.5886 (2)	0.09955 (10)	0.0332 (5)
C6	0.21703 (11)	0.6954 (3)	0.10085 (12)	0.0433 (5)
C7	0.19368 (11)	0.7412 (3)	0.15998 (13)	0.0482 (6)
C8	0.22150 (12)	0.6847 (3)	0.21893 (12)	0.0483 (6)
C9	0.27309 (12)	0.5836 (3)	0.21931 (11)	0.0431 (6)
C10	0.29880 (10)	0.5336 (3)	0.15921 (10)	0.0348 (4)
C11	0.43017 (10)	0.2827 (2)	0.10657 (10)	0.0336 (5)
C12	0.46549 (10)	0.2050 (3)	0.05652 (11)	0.0375 (5)
C13	0.51676 (10)	0.1174 (3)	0.08745 (12)	0.0436 (5)
C14	0.51413 (11)	0.1385 (3)	0.15582 (12)	0.0453 (6)
C15	0.46104 (11)	0.2385 (3)	0.16838 (11)	0.0394 (5)
C21	0.33343 (12)	−0.0256 (3)	0.1218 (2)	0.0626 (8)
C22	0.3638 (2)	−0.0909 (4)	0.0665 (2)	0.0664 (8)
C23	0.41639 (15)	−0.1823 (3)	0.0899 (2)	0.0594 (7)
C24	0.41921 (13)	−0.1750 (3)	0.15853 (14)	0.0542 (7)
C25	0.36809 (13)	−0.0774 (3)	0.17821 (14)	0.0558 (7)
C31	0.36339 (10)	0.3930 (3)	−0.02468 (10)	0.0326 (4)
C32	0.33421 (12)	0.2672 (3)	−0.05722 (12)	0.0491 (6)
C33	0.35304 (13)	0.2252 (4)	−0.11929 (13)	0.0593 (7)
C34	0.40098 (12)	0.3075 (3)	−0.14920 (12)	0.0541 (7)
C35	0.43074 (12)	0.4324 (3)	−0.11762 (12)	0.0508 (6)
C36	0.41201 (12)	0.4750 (3)	−0.05546 (11)	0.0434 (5)

Table 2. *Selected geometric parameters (Å, °)*

The pseudo-atoms *Cg*1 and *Cg*2 are the centroids of the two five-membered rings.

Fe1—*Cg*1	1.642 (1)	N4—C5	1.364 (3)
Fe1—*Cg*2	1.653 (1)	C5—C6	1.409 (3)
N1—C2	1.326 (2)	C5—C10	1.412 (3)
N1—C10	1.356 (3)	C6—C7	1.368 (3)
C2—C3	1.442 (3)	C7—C8	1.393 (3)
C2—C11	1.478 (3)	C8—C9	1.363 (3)
C3—N4	1.315 (3)	C9—C10	1.415 (3)
C3—C31	1.494 (3)		
*Cg*1—Fe1—*Cg*2	179.1 (1)	N4—C5—C10	120.3 (2)
C2—N1—C10	118.4 (2)	C6—C5—C10	119.8 (2)
N1—C2—C3	120.2 (2)	C7—C6—C5	119.6 (2)
N1—C2—C11	115.4 (2)	C6—C7—C8	120.9 (2)
C3—C2—C11	124.4 (2)	C9—C8—C7	120.9 (2)
N4—C3—C2	121.8 (2)	C8—C9—C10	120.0 (2)
N4—C3—C31	114.9 (2)	N1—C10—C5	121.1 (2)
C2—C3—C31	123.2 (2)	N1—C10—C9	120.0 (2)
C3—N4—C5	118.1 (2)	C5—C10—C9	118.8 (2)
N4—C5—C6	119.9 (2)		
C11—*Cg*1—*Cg*2—C21	−6.9	C14—*Cg*1—*Cg*2—C24	−6.4
C12—*Cg*1—*Cg*2—C22	−6.4	C15—*Cg*1—*Cg*2—C25	−5.9
C13—*Cg*1—*Cg*2—C23	−6.7		

Compound (I) crystallized in the monoclinic system; space groups *C*2/*c* or *Cc* were indicated by the systematic absences. *C*2/*c* was chosen and confirmed by the successful refinement. The structure was solved by the Patterson heavy-atom method. Examination of the structure with *PLATON* (Spek, 1995) showed that there were no solvent-accessible voids anywhere in the crystal lattice.

Data collection: *CAD*-4-*PC* (Enraf–Nonius, 1992). Cell refinement: *SET*4 and *CELDIM* (Enraf–Nonius, 1991). Data reduction: *DATRD*2 in *NRCVAX*94 (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to refine structure: *NRCVAX*94 and *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *NRCVAX*94 and *PLATON* (Spek, 1995). Software used to prepare material for publication: *NRCVAX*94 and *SHELXL*93.

GF thanks NSERC (Canada) for research grants.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1042). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1-Ferrocenyl-2-phenylethanedione

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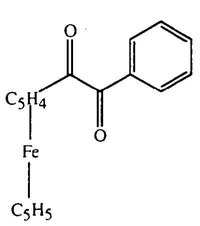
Abstract

The title compound, $[Fe(C_5H_5)(C_{13}H_9O_2)]$, crystallizes as chiral molecules in space group $P2_12_12_1$; in the crystal chosen for structure analysis, the molecules have the *S* configuration. The molecules are linked by weak C—H···O hydrogen bonds into chains parallel to the *b* axis and by C—H···π(C=C) interactions into chains parallel to the *a* axis.

Comment

Ferrocenyl-1,2-diketones Fc–CO–CO–*R* [Fc = (C_5H_5)-$Fe(C_5H_4)$] are important synthetic intermediates. As part of a wider study of these materials, we now report the structure of a representative example, 1-ferrocenyl-2-phenylethanedione, Fc–CO–CO–Ph (ferrocenylphenylglyoxal), (I).



(I)

1-Ferrocenyl-2-phenylethanedione crystallizes in the non-centrosymmetric space group $P2_12_12_1$ with a single molecule in the asymmetric unit (Fig. 1). Within these molecules the fragment C11—C1—O1 is almost coplanar with the substituted cyclopentadienyl ring C1*n* (*n* = 1–5) (Table 2), as typically found in simple acyl ferrocenes Fc–CO–*R* (Gyepes & Hanic, 1975; Sato, Iwai, Sano & Konno, 1984; Barnes, Bell, Glidewell & Howie, 1990); similarly, the fragment C31—C2—O2 is almost coplanar with the phenyl ring (Table 2). The mean value of the C1*n*—*Cg*1—*Cg*2—C2*n* twist angle (*n* = 1–5) is −20.0 (4)°, essentially midway between the value for complete eclipsing of the rings, 0.0°, and the value for perfect staggering, 36.0° (*Cg*1 and *Cg*2 are the ring centroids).

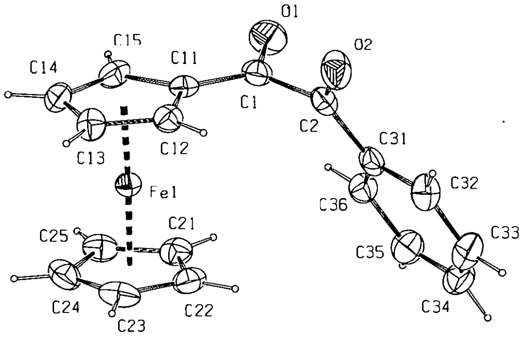


Fig. 1. A view of the *S* enantiomer of (I) with our numbering scheme. Ellipsoids are drawn at the 30% probability level.

The torsion angle O1—C1—C2—O2 is 109.1 (5)°, so that the projections of the two carbonyl groups onto the plane normal to C1—C2 are almost orthogonal. Benzil, Ph–CO–CO–Ph, exhibits a similar value, 111.6°, for this torsion angle in the solid state (Brown & Sadanaga, 1965; More, Odou & Lefebvre, 1987), and similar torsion angles have been found for a range of 4,4′-disubstituted benzils $(XC_6H_4CO)_2$ (Kimura, McCluney & Watson, 1979; Crowley, Balanson & Mayerle, 1983). However, butane-2,3-dione, CH_3–CO–CO–CH_3 (Eriks, Hayden, Yang & Chan, 1983) and its 1,4-bis-diazo derivative (Hope & Black, 1972) are both centrosymmetric molecules in the solid state ($P2_1/n$, Z = 2) and hence adopt the *trans*-planar C_{2h} conformation; similarly, this conformer was the sole rotamer found in the gas phase even at 798 K (Danielson & Hedberg, 1979). The conformation found for (I) corresponds to molecular point group C_1, so that the molecules are chiral in the solid state, and all have the same handedness in $P2_12_12_1$; in the crystal chosen for study, the molecules have the *S* configuration. In solution, however, the NMR spectra indicate that *R*/*S* interconversion is fast at room temperature, so that crystallization in $P2_12_12_1$ represents a case of spontaneous resolution, or 'conglomerate crystallization'