Mo—C1	2.003 (6)	O2—C2	1.143 (8)
Mo—C2	2.016 (6)	O3—C3	1.129 (9)
Mo—C3	2.032 (7)	O4—C4	1.127 (8)
Mo—C4	2.042 (6)	O5—C11	1.389 (6)
P1—O5	1.598 (4)	O6—C21	1.419 (7)
P1—O6	1.605 (4)	O7—C31	1.391 (7)
P1—O7	1.610 (4)	O8—C41	1.415 (7)
P2—O8	1.600 (4)	O9—C51	1.413 (8)
P2—O9	1.612 (4)	O10—C61	1.394 (7)
P1—Mo—P2	89.55 (5)	O5—P1—O7	97.71 (23)
P1—Mo—C1	177.96 (18)	O6—P1—O7	103.97 (21)
P1—Mo—C2	94.57 (16)	Mo—P2—O8	115.43 (16)
P1—Mo—C3	88.51 (16)	Mo—P2—O9	120.40 (18)
P1—Mo—C4	89.08 (15)	Mo—P2—O10	118.51 (17)
P2—Mo—C1	88.49 (18)	O8—P2—O9	104.38 (24)
P2—Mo—C2	173.94 (17)	O8—P2—O10	96.86 (22)
P2—Mo—C3	88.17 (16)	O9—P2—O10	97.34 (24)
P2—Mo—C4	95.65 (15)	P1—O5—C11	130.7 (4)
C1—Mo—C2	87.42 (24)	P1—O6—C21	124.0 (4)
C1—Mo—C3	92.00 (23)	P1—O7—C31	125.3 (3)
C1—Mo—C4	90.54 (23)	P2—O8—C41	127.5 (4)
C2—Mo—C3	87.50 (24)	P2—O9—C51	122.4 (4)
C2—Mo—C4	88.87 (24)	P2—O10—C61	125.6 (4)
C3—Mo—C4	175.47 (22)	Mo—C1—O1	178.1 (5)
Mo—P1—O5	115.25 (17)	Mo—C2—O2	175.4 (5)
Mo—P1—O6	112.49 (14)	Mo—C3—O3	178.2 (5)
Mo—P1—O7	121.91 (14)	Mo—C4—O4	175.1 (5)
O5—P1—O6	102.97 (20)		

The space group was determined as either $P2_1$ or $P2_1/m$ from the systematic absences ($0k0$ absent if $k = 2n + 1$); $P2_1$ was chosen and confirmed by the analysis. All H atoms were clearly visible in difference maps calculated at intermediate stages of refinement; they were then positioned on geometric grounds (C—H 0.95 Å) and included as riding atoms in the structure-factor calculations. Space group $P2_1$ is chiral and calculations with the coordinates as listed and with their inverse, clearly showed that the correct enantiomer had been chosen for the crystal selected for the analysis; the R and wR values for the other enantiomer were higher (0.0306 and 0.0387 compared with 0.0275 and 0.0345, respectively). There is no solvent of crystallization present in the lattice and an examination of the crystal structure using *PLATON* (Spek, 1990) revealed no potential volume for any solvent molecules. Data collection and cell refinement were performed using *CAD*-4 *Software* (Enraf-Nonius, 1989). *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989) was used for data reduction, structure solution and refinement, and for preparation of the material for publication. The diagram was prepared using *ORTEP*II (Johnson, 1976).

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71838 (33 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1120]

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Hydrogen Bonding in Ferrocene Derivatives: Structure of the 1:1 Adduct of Ferrocenyl(diphenyl)methanol and Dimethyl Sulfoxide

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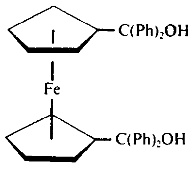
Abstract

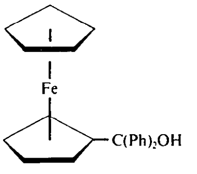
Ferrocenyl(diphenyl)methanol, $[(C_5H_5)Fe\{(C_5H_4)C(Ph_2)OH\}]$, forms a 1:1 adduct with dimethyl sulfoxide, $(CH_3)_2SO$. In this adduct, the hydrogen-bonded aggregate consists of one molecule of the ferrocene acting as hydrogen-bond donor and one molecule of DMSO acting as hydrogen-bond acceptor; the hydrogen-bonded O···O distance is 2.796 (2) Å.

Comment

The diol ferrocene-1,1′-diylbis(diphenylmethanol), $[Fe\{(C_5H_4)CPh_2OH\}_2]$ (I), forms hydrogen-bonded adducts with a wide range of hydrogen-bond donor and acceptor molecules (Ferguson, Gallagher, Glidewell & Zakaria, 1993*a*; Glidewell, Ferguson, Lough & Zakaria, 1994). These adducts exhibit a very wide range of structural types and, for certain hydrogen-bond acceptors, several distinct adducts of different stoichiometries are formed. Thus, for example, (I) forms two adducts with dimethyl sulfoxide (DMSO) which have diol:DMSO ratios of 1:1 and 1:2, while of the adducts formed with 1,4-dioxan, those having diol:dioxan ratios of 1:1 and 1:2, have been characterized by X-ray methods (Glidewell, Ferguson, Lough & Zakaria, 1994). Although the mono-alcohol ferrocenyl(diphenyl)methanol, $[(C_5H_5)Fe\{(C_5H_4)C(Ph_2)OH\}]$ (II), forms fewer adducts than the diol (I), adducts with DMSO having alcohol:DMSO ratios of 2:1 and 1:1 have been identified; the structure of the 1:1 adduct has now been determined.



(I)



(II)

The structure of the 1:1 adduct formed between the ferrocenyl alcohol (II) and DMSO comprises finite hydrogen-bonded aggregates containing one molecule of each component in which the OH group of the ferrocene portion acts as hydrogen-bond donor and the O atom of the DMSO acts as hydrogen-bond acceptor (Fig. 1). Within the ferrocenyl moiety, the C—C bond lengths in the substituted cyclopentadienyl ring range from 1.403 (4) to 1.426 (3) Å [mean 1.420 (3) Å] while those in the unsubstituted ring lie in the range 1.384 (5) to 1.411 (6) Å [mean 1.397 (5) Å]. Such differences between the substituted and unsubstituted rings have been noted previously (Barnes, Bell, Glidewell & Howie, 1990). The other interatomic distances within the ferrocene fragment are typical of their types. The two C_5 rings are tilted from a parallel arrangement by 3.1 (1)° and twisted from an eclipsed conformation by 7.6 (1)°; near eclipsing of the two C_5 rings in hydrogen-bonded ferrocene derivatives appears to be the normal conformation (Ferguson, Gallagher, Glidewell & Zakaria, 1993*a*, 1993*b*, 1994; Gallagher, Ferguson, Glidewell & Zakaria, 1994; Glidewell, Ferguson, Lough & Zakaria, 1994). The conformation about the C1—C11 bond exocyclic to the C_5H_4 ring is such that the C1—C41 bond almost eclipses the C_5H_4 ring, while the projection of the C1—C31 bond is almost perpendicular to this ring.

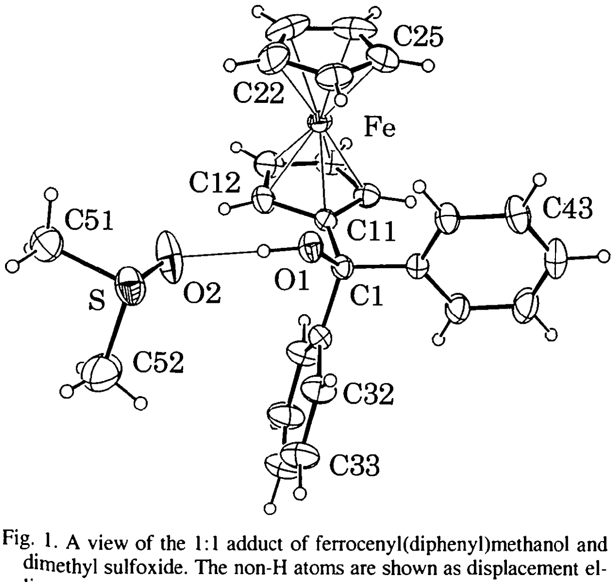


Fig. 1. A view of the 1:1 adduct of ferrocenyl(diphenyl)methanol and dimethyl sulfoxide. The non-H atoms are shown as displacement ellipsoids drawn at the 35% probability level. For clarity, H atoms are drawn as small spheres of arbitrary size.

The S—O distance in the hydrogen-bonded DMSO molecule is 1.488 (2) Å, towards the lower end of the range of 1.471-1.531 Å recorded (Schomburg, Blaschette & Schulte-Körne, 1990) for free and complexed DMSO, and very significantly shorter than the S—O distance [1.531 (5) Å corrected for libration, 1.521 (5) Å uncorrected] in DMSO itself (Thomas, Shoemaker & Eriks, 1966). The S—C distances [1.773 (3) and 1.776 (3) Å], the C—S—C angle [97.4 (2)°] and the O—S—C angles [105.0 (1) and 106.6 (2)°] are all near the middle of the ranges reported previously [1.730-1.821 Å, 92.1-100.1° and 103.5-107.5°, respectively (Schomburg, Blaschette & Schulte-Körne, 1990)].

The hydrogen-bonded O···O distance is 2.796 (2) Å, indicative of a fairly weak hydrogen bond although perhaps marginally stronger than those [2.816 (1) Å] in the dimeric form of solvent-free (II) (Ferguson, Gallagher, Glidewell & Zakaria, 1993*b*); the O—H···O angle is 172.3° (hydroxyl H-atom coordinates from a difference map). The hydrogen bonding in this adduct of ferrocenyl(diphenyl)methanol and DMSO is thus the simplest that could be envisaged, with a single hydrogen-bond motif described by the graph set *D* (Etter, 1990; Etter, MacDonald & Bernstein, 1990). A similar motif occurs in the 1:2 adduct of DMSO with tetraphenylethane-1,2-diol, $Ph_2C(OH)C(OH)Ph_2$ (Bond, Bourne, Nassimbeni & Toda, 1989), although in that adduct, half of the DMSO molecules exhibit orientational disorder; the two orientations have common sites for the C and O atoms, but different sites for the S atoms. The same type of DMSO disorder occurs in $(Ph_3COH)_2$.DMSO (Weber, Skobridis & Goldberg, 1989). In the present adduct, the DMSO molecules are fully ordered. The hydrogen-bonding pattern in $(Ph_3COH)_2$.DMSO provides a plausible model for the structure of $(II)_2$.DMSO, with the O atom of the DMSO acting as hydrogen-bond acceptor from two alcohol molecules. The 1:1 and 1:2 adducts formed by DMSO with the ferrocenediol (I) have not yet been characterized structurally because crystals of a quality appropriate for single-crystal X-ray diffraction study have not so far been obtained. Any structural predictions can only be speculative since the structures of 1:1 and 1:2 adducts formed by DMSO with a series of linearly condensed polythiophenes show simple *D* motifs for the 1:2 adducts, but a variety of structures for the 1:1 adducts (Mazaki, Hayashi & Kobayashi, 1992).

Experimental

Crystals were obtained by slow evaporation of a solution of ferrocenyl(diphenyl)methanol (Ferguson, Gallagher, Glidewell & Zakaria, 1994) in dry DMSO. As the dried crystals rapidly lost transparency when exposed to air, they were stored under DMSO until required and then coated with epoxy resin prior to X-ray examination.

Crystal data

$[Fe(C_5H_5)(C_{18}H_{15}O)].C_2H_6OS$
$M_r = 446.38$
Triclinic
$P\overline{1}$
$a = 7.7174$ (9) Å
$b = 9.5225$ (9) Å
$c = 16.1768$ (19) Å
$\alpha = 99.311$ (8)°
$\beta = 102.708$ (10)°
$\gamma = 103.949$ (9)°
$V = 1096.14$ (21) Å^3

$Z = 2$
$D_x = 1.352$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71067$ Å
Cell parameters from 25 reflections
$\theta = 11.50$-$19.00°$
$\mu = 0.79$ mm^{-1}
$T = 293$ K
Block
0.68 × 0.49 × 0.44 mm
Orange-yellow

Data collection

Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: empirical
$T_{min} = 0.691$, $T_{max} = 0.742$
4755 measured reflections
4755 independent reflections

3903 observed reflections $[I_{net} > 3.0\sigma(I_{net})]$
$\theta_{max} = 26.90°$
$h = -9 \rightarrow 9$
$k = 0 \rightarrow 12$
$l = -20 \rightarrow 20$
3 standard reflections
frequency: 120 min
intensity variation: 4.0%

Refinement

Refinement on F
$R = 0.034$
$wR = 0.052$
$S = 1.45$
3903 reflections
264 parameters
$w = 1/[\sigma^2(F) + 0.0008F^2]$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.37$ e Å^{-3}

Extinction correction: Larson (1970)
Extinction coefficient: 1604 (398)
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

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

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

	x	y	z	U_{eq}
Fe	0.94246 (4)	0.63757 (3)	0.852215 (19)	0.04294 (18)
*Cp*1†	1.06278	0.52057	0.83366	
*Cp*2†	0.82584	0.75879	0.86986	
O1	0.62969 (19)	0.34995 (16)	0.67799 (9)	0.0460 (8)
C1	0.7534 (3)	0.29815 (21)	0.73730 (12)	0.0368 (10)
C11	0.9205 (3)	0.42521 (20)	0.79211 (12)	0.0382 (10)
C12	1.0334 (3)	0.53129 (25)	0.75892 (15)	0.0526 (12)
C13	1.1864 (3)	0.6214 (3)	0.82909 (19)	0.0634 (15)
C14	1.1687 (3)	0.5733 (3)	0.90511 (17)	0.0593 (13)
C15	1.0049 (3)	0.45164 (22)	0.88308 (13)	0.0458 (11)
C21	0.6887 (4)	0.6743 (3)	0.82198 (20)	0.0673 (16)
C22	0.8185 (5)	0.7789 (3)	0.79813 (22)	0.0792 (21)
C23	0.9593 (4)	0.8563 (3)	0.8742 (3)	0.094 (3)
C24	0.9142 (5)	0.7981 (4)	0.94406 (23)	0.0929 (23)
C25	0.7485 (5)	0.6863 (3)	0.91095 (21)	0.0758 (20)
C31	0.8165 (3)	0.18008 (22)	0.68516 (12)	0.0410 (11)
C32	0.6851 (4)	0.0700 (3)	0.61840 (17)	0.0632 (15)
C33	0.7358 (5)	−0.0419 (3)	0.57155 (18)	0.0773 (18)
C34	0.9169 (5)	−0.0437 (3)	0.58935 (20)	0.0777 (19)
C35	1.0475 (4)	0.0628 (3)	0.65524 (20)	0.0761 (18)
C36	0.9971 (3)	0.1747 (3)	0.70237 (16)	0.0579 (13)
C41	0.6417 (3)	0.22968 (21)	0.79612 (12)	0.0388 (9)
C42	0.5061 (3)	0.2873 (3)	0.81899 (14)	0.0481 (12)
C43	0.4139 (3)	0.2313 (3)	0.87661 (16)	0.0623 (16)
C44	0.4539 (4)	0.1169 (3)	0.91060 (17)	0.0690 (16)
C45	0.5859 (4)	0.0566 (3)	0.88737 (18)	0.0728 (19)
C46	0.6808 (4)	0.1140 (3)	0.83092 (16)	0.0601 (15)
S	0.66160 (9)	0.41418 (9)	0.46329 (5)	0.0700 (4)
O2	0.7892 (3)	0.4561 (3)	0.55258 (13)	0.1038 (19)
C51	0.7375 (4)	0.5638 (3)	0.41465 (18)	0.0707 (17)
C52	0.7353 (6)	0.2804 (4)	0.4011 (3)	0.109 (3)
HO1	0.694	0.386	0.639	0.067 (7)

† *Cp*1 and *Cp*2 are the centroids of the rings C11-C15 and C21-C25.

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

Fe—*Cp*1	1.645	C11—C12	1.423 (3)
Fe—*Cp*2	1.652	C11—C15	1.426 (3)
Fe—C11	2.0469 (18)	C12—C13	1.423 (3)
Fe—C12	2.0269 (21)	C13—C14	1.403 (4)
Fe—C13	2.0344 (23)	C14—C15	1.425 (3)
Fe—C14	2.0452 (23)	C21—C22	1.399 (4)
Fe—C15	2.0512 (20)	C21—C25	1.388 (5)
Fe—C21	2.040 (3)	C22—C23	1.403 (5)
Fe—C22	2.031 (3)	C23—C24	1.411 (6)
Fe—C23	2.023 (3)	C24—C25	1.384 (5)
Fe—C24	2.040 (3)	S—O2	1.488 (2)
Fe—C25	2.044 (3)	S—C51	1.776 (3)
O1—C1	1.433 (2)	S—C52	1.773 (4)
C1—C11	1.520 (3)	O1—HO1	0.948
C1—C31	1.533 (3)	O1···O2	2.796 (2)
C1—C41	1.538 (3)	O2···HO1	1.854
*Cp*1—Fe—*Cp*2	178.29	C11—C1—C41	109.95 (15)
C1—O1—HO1	107.2	C31—C1—C41	110.46 (15)
O1—C1—C11	111.12 (15)	O2—S—C51	104.98 (14)
O1—C1—C31	108.82 (15)	O2—S—C52	106.61 (19)
O1—C1—C41	106.30 (14)	C51—S—C52	97.42 (16)
C11—C1—C31	110.12 (15)	O1—HO1—O2	172.3

Data collection and cell refinement: *CAD*-4 *Software* (Enraf-Nonius, 1989). Data reduction, structure solution and refinement, preparation of material for publication: *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). The adduct between (II) and DMSO crystallized in the triclinic system; space group $P\overline{1}$ was assumed and confirmed by the refinement. All non-H atoms were refined by full-matrix least-squares calculations. All H atoms were clearly visible in difference maps; those bonded to C atoms were positioned geometrically (C—H 0.95 Å) and included as riding atoms in the structure-factor calculations. The hydroxyl H atom was positioned at the coordinates obtained from a difference map and allowed to refine isotropically. The displacement-ellipsoid diagram (Fig. 1) was prepared using *ORTEP*II (Johnson, 1976). Examination of the structure with *PLATON* (Spek, 1992) showed that there were no solvent-accessible voids in the crystal lattice.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1165). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1994). C**50**, 681-683

[Pd{P($C_5H_{10}N$)Ph_2}$_2$$Cl_2$]

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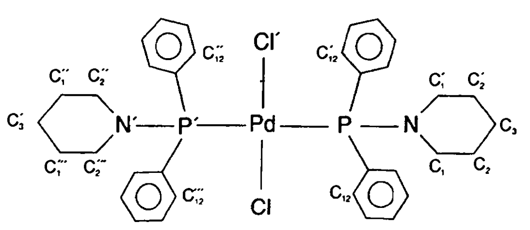
Abstract

The diphenyl(piperidino)phosphine ligands in the structure of dichlorobis[diphenyl(piperidino)phosphine]palladium, [$PdCl_2(C_{17}H_{20}NP)_2$], coordinate to the Pd atom in a *trans* arrangement and the two Cl ligands complete the square-planar Pd coordination sphere. This arrangement is required by the crystallographically imposed $2/m$ symmetry that the molecule possesses. The Pd atom sits on a centre of inversion with the P, N and C(*para*) atoms of the piperidine group being bisected by a mirror plane. The Cl atoms define a twofold axis.

Comment

We have been studying the bidentate ligand bis(diphenylphosphino)amine, $Ph_2PN(H)PPh_2$, and its derivatives. In structural studies on complexes of bis(diphenylphosphino)amine ligands (Browning, Farrar & Frankel, 1992), the observed P—N bond lengths are significantly less than the values associated with P—N single bonds (1.77 Å) (Greenwood & Earnshaw, 1984) or the sum of the covalent radii of the P and N atoms (1.84 Å) (Shriver, Atkins & Langford, 1990). The short P—N bond lengths and the observed planarity at the N centres in the bis(diphenylphosphino)amine ligands have been the basis for proposed P—N π bonding. Our theoretical calculations on these ligands suggest that the P—N bond order is one (Browning, Farrar & Peterson, 1991). In an attempt to obtain a better estimate of a P—N single-bond length for the bisphosphinoamine ligands, we undertook the structural characterization of a complex containing bis[diphenyl(piperidino)phosphine] ligands, $Ph_2PNC_5H_{10}$. The chair conformation of the piperidine ring forces the N atom towards pyramidal geometry and thus disrupts any significant π bonding.

Bond lengths and angles for the molecule are listed in Table 2. All intermolecular contacts are greater than the sum of the van der Waals radii of the respective atom pairs. Fig. 1 is a view of the molecule (1) with H atoms omitted for clarity.



(1)

The piperidine ring is in a chair conformation with the lone pair of the N atom oriented *anti* to the Pd—P bond. Steric demands would favour the piperidine and phenyl rings to mesh forming a propeller-type arrangement, as is seen for triarylphosphines (Corbridge, 1974). The piperidine group is instead bracketed by the two phenyl groups. This

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