

Table 2. Geometric parameters (Å, °)

Ir—M1	2.090	Ir—M2	2.101
Ir—P1	2.346 (3)	Ir—P2	2.358 (3)
Ir—C35	2.191 (11)	Ir—C36	2.197 (12)
Ir—C39	2.223 (12)	Ir—C40	2.208 (12)
Fe—Cp1	1.630	Fe—Cp2	1.640
Fe—C1	2.002 (12)	Fe—C2	2.016 (13)
Fe—C3	2.006 (12)	Fe—C4	2.067 (14)
Fe—C5	2.039 (13)	Fe—C18	2.000 (11)
Fe—C19	2.019 (13)	Fe—C20	2.049 (14)
Fe—C21	2.068 (15)	Fe—C22	2.035 (11)
P1—C1	1.784 (11)	P1—C6	1.830 (11)
P1—C12	1.800 (12)	P2—C18	1.806 (13)
P2—C23	1.809 (13)	P2—C29	1.818 (11)
Ir...Fe	4.340 (2)		
M1—Ir—M2	83.4	M1—Ir—P1	172.8
M1—Ir—P2	87.9	M2—Ir—P1	89.6
M2—Ir—P2	170.8	P1—Ir—P2	99.2 (1)
Cp1—Fe—Cp2	178.9	Ir—P1—C1	119.1 (4)
Ir—P1—C6	112.2 (4)	Ir—P1—C12	113.1 (4)
C1—P1—C6	101.9 (5)	C1—P1—C12	104.9 (6)
C6—P1—C12	104.2 (5)	Ir—P2—C18	121.7 (4)
Ir—P2—C23	110.6 (4)	Ir—P2—C29	111.8 (4)
C18—P2—C23	103.1 (6)	C18—P2—C29	102.4 (5)
C23—P2—C29	105.7 (6)		

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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 71128 (36 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI1043]

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Ferrocenyl Ligands. Part 4. Structure of [1,1'-Bis(diphenylphosphino)-3,3'-bis(trimethylsilyl)ferrocene]dichlorocobalt†

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Abstract

1,1'-Bis(diphenylphosphino)-3,3'-bis(trimethylsilyl)-ferrocene acts as a bidentate ligand when reacted with CoCl_2 , producing a tetrahedral P_2Cl_2 coordination sphere that is significantly distorted. The influence of the trimethylsilyl groups in this distortion is considerable, restraining the ferrocenyl moiety from adopting the eclipsed configuration preferred for binding tetrahedral ions.

Comment

Recently we have embarked on a study of ferrocenyl ligands with a view to synthesizing novel tunable ligands (Houlton, Roberts, Silver & Drew 1990; Houlton, Ibrahim, Dilworth & Silver, 1990; Houlton, Ahmet, Miller, Silver & Slade, 1993). A part of this study is the investigation of sterically crowded ligands with the aim of introducing stereoselectivity to complex formation, and we have previously reported 1,1'-bis(diphenylphosphino)-3,3'-bis(trimethylsilyl)ferrocene (1) (Brown, Houlton, Roberts, Frampton & Silver, 1993). Here we report the crystal and molecular structure of the cobalt chloride complex of this ligand, (1) CoCl_2 , and discuss the effect of the 3,3'-substituents compared to complexes of the bis(diphenylphosphino)ferrocene (fdpp).

The structure of the parent ligand reveals a *cisoid* arrangement of the diphenylphosphino groups (Brown, Houlton, Roberts, Frampton & Silver,

† Dichloro- $2\kappa^2\text{Cl}$ -bis(μ -diphenyl[3-(trimethylsilyl)- $2\kappa\text{P}$:1(η^5)-cyclopentadienylidene]phosphine)-cobaltiron.

1993) in contrast to the *trans* conformation for fdpp (Casellato, Ajó, Valle, Corrain, Longato & Graziani, 1988). This feature demonstrates the considerable geometric restrictions imposed by the Me₃Si groups. On complexation of the CoCl₂ group the ligand remains in essentially the same configuration as when uncoordinated. The P...P distance decreases on complexation from 3.96 to 3.78 Å, but this is still long compared to (fdpp)NiBr₂ (Butler, Cullen, Kim, Rettig & Trotter, 1985). The cyclopentadienyl rings are planar, inclined at a slight angle (2.7°), and staggered with respect to each other. This arrangement of the Cp rings is in marked contrast to fdpp when bound to tetrahedral Ni^{II}, which displays eclipsed rings (Butler, Cullen, Kim, Rettig & Trotter, 1985; Casellato, Ajó, Valle, Corrain, Longato & Graziani, 1988). This inability to adopt an eclipsed conformation probably accounts for the comparatively large difference in the phosphorus to metal bond lengths. The largest such difference observed in fdpp complexes is 0.032 Å for (fdpp)Mo(CO)₄, the smallest difference being within experimental error (Butler, Cullen, Kim, Rettig & Trotter, 1985). For (1)CoCl₂ this difference is considerably larger at 0.057 Å. Fig. 2 presents a profile view of the complex, indicating the displacements of the substituent groups relative to the ferrocenyl moiety and bond lengths for the coordination sphere. The iron–ring distances, also indicated, are at the larger end of the range observed for fdpp complexes, in keeping with tetrahedral coordination (Houlton, Ibrahim, Dilworth & Silver, 1990).

Surprisingly few tetrahedral cobalt complexes were found in the literature. Compared to di- μ -iodobis(iodotriphenylphosphine)cobalt(II) bis(benzene) (Gorter, Hinrichs, Reedijk, Rimbault, Pierrard & Hugel, 1985), (1)CoCl₂ exhibits both shorter and longer P—Co bond lengths [2.405 (2) and 2.348 (2) Å for (1)CoCl₂ *cf.* 2.3635 (8) Å] emphasizing the distorted nature of the coordination sphere. Co—Cl distances for (1)CoCl₂ are shorter than in the N₂Cl₂-coordinated dichlorophenyldi(2-pyridyl)phosphine-*N,N'*]cobalt(II) hemiethanolate complex (Ehrlich, Fronczek, Watkins, Newkome & Hager, 1984) and in the O₂Cl₂-coordinated complex dichlorobis(trimethylphosphine oxide)cobalt(II) (Edelmann & Behrens, 1986). The respective distances for these compounds are 2.221 (1) and 2.256 (2) Å, *cf.* an average 2.213 (2) Å for (1)CoCl₂.

We have reported a correlation between the ⁵⁷Fe Mössbauer parameters δ and Δ , and the metal-ion coordination geometry for the complexes of fdpp (Houlton, Ibrahim, Dilworth & Silver, 1990). Such studies of (1) showed Pd²⁺ complexation to decrease the isomer shift compared to the ligand itself, whereas Co²⁺ caused an increase. However, in contrast to fdpp, only a decrease in quadrupole splitting

was observed (Brown, Houlton, Roberts, Frampton & Silver, 1992). The structural analysis here shows the inability of (1) to adopt idealized ferrocenyl geometries on complexation, therefore, correlations between the hyperfine interactions of the ferrocenyl group and metal-ion coordination geometry would not be expected.

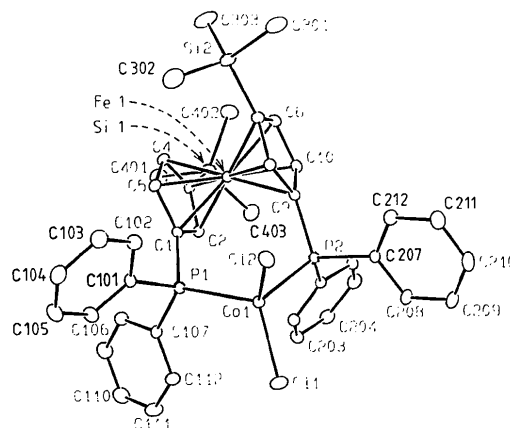


Fig. 1. Molecular structure of (1)CoCl₂ showing the labelling scheme, drawn using ORTEP (Johnson, 1965) with the thermal ellipsoids set at 0.10.

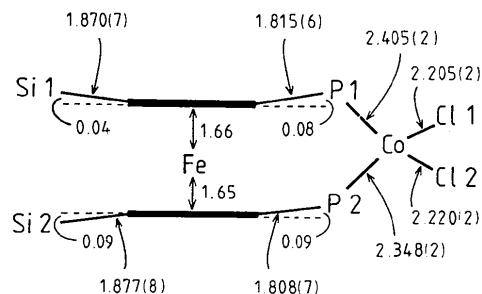


Fig. 2. Profile view of (1)CoCl₂ highlighting the P₂Cl₂ coordination sphere and the displacement of the substituent groups from the cyclopentadienyl rings.

Experimental

Crystal data

[CoFeCl₂(C₄₀H₄₄P₂Si₂)]

M_r = 828.59

Triclinic

P $\bar{1}$

a = 11.3947 (9) Å

b = 12.653 (1) Å

c = 16.872 (1) Å

α = 90.639 (7)°

β = 107.891 (6)°

γ = 114.817 (7)°

V = 2073.4 (3) Å³

Z = 2

D_x = 1.327 Mg m⁻³

Mo *K* α radiation

λ = 0.71069 Å

Cell parameters from 22

reflections

θ = 17–21°

μ = 1.0378 mm⁻¹

T = 293 K

Plate

0.25 × 0.16 × 0.06 mm

Green

Data collection

Enraf-Nonius CAD-4
diffractometer $\omega/2\theta$ scansAbsorption correction:
none

7552 measured reflections

7283 independent reflections

3331 observed reflections

 $[I > 3\sigma(I)]$

Refinement

Refinement on F Final $R = 0.041$ $wR = 0.046$ $S = 0.332$

3331 reflections

433 parameters

H-atom parameters not re-
fined $R_{\text{int}} = 0.042$ $\theta_{\text{max}} = 25^\circ$ $h = -13 \rightarrow 12$ $k = -15 \rightarrow 15$ $l = 0 \rightarrow 20$

3 standard reflections

frequency: 120 min

intensity variation: 2.7%

 $w = 1/[\sigma^2(F) + (0.02F)^2 + 1]$ $(\Delta/\sigma)_{\text{max}} < 0.01$ $\Delta\rho_{\text{max}} = 0.01 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\text{min}} = -0.01 \text{ e } \text{\AA}^{-3}$

Atomic scattering factors

from *International Tables*
for *X-ray Crystallography*
(1974, Vol. IV)Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (\AA^2)
$$B_{\text{eq}} = (4/3)[a^2 B(1,1) + b^2 B(2,2) + c^2 B(3,3) + ab(\cos \gamma)B(1,2) + ac(\cos \beta)B(1,3) + bc(\cos \alpha)B(2,3)].$$

	x	y	z	B_{eq}
Co1	0.55941 (8)	0.37002 (7)	0.25986 (6)	3.01 (2)
Fe1	0.25477 (8)	0.02738 (7)	0.21318 (6)	2.72 (2)
Cl1	0.6747 (2)	0.5489 (2)	0.3346 (1)	5.22 (5)
Cl2	0.6572 (2)	0.3331 (2)	0.1747 (1)	4.72 (4)
P1	0.3207 (2)	0.3183 (1)	0.1860 (1)	2.87 (4)
P2	0.5594 (2)	0.2292 (1)	0.3493 (1)	2.81 (4)
Si1	-0.0141 (2)	-0.0972 (2)	0.2966 (1)	4.36 (5)
Si2	0.2888 (2)	-0.1610 (2)	0.0703 (2)	5.20 (6)
C1	0.2055 (5)	0.1645 (5)	0.1805 (4)	2.8 (1)
C2	0.1663 (5)	0.1201 (5)	0.2507 (4)	3.1 (2)
C3	0.0705 (6)	-0.0012 (5)	0.2276 (4)	3.3 (2)
C4	0.0520 (6)	-0.0305 (6)	0.1423 (4)	3.6 (2)
C5	0.1328 (6)	0.0681 (5)	0.1126 (4)	3.6 (2)
C6	0.2973 (6)	-0.1125 (5)	0.2435 (5)	3.8 (2)
C7	0.3417 (6)	-0.0700 (5)	0.1749 (4)	3.7 (2)
C8	0.4387 (5)	0.0513 (5)	0.2040 (4)	3.1 (1)
C9	0.4532 (5)	0.0817 (5)	0.2898 (4)	2.7 (1)
C10	0.3639 (6)	-0.0211 (5)	0.3132 (4)	3.3 (2)
C101	0.2980 (6)	0.3449 (5)	0.0777 (4)	3.3 (2)
C102	0.3399 (7)	0.2906 (6)	0.0280 (5)	4.6 (2)
C103	0.3397 (7)	0.3194 (7)	-0.0502 (4)	5.5 (2)
C104	0.2955 (8)	0.4015 (7)	-0.0799 (5)	5.7 (2)
C105	0.2538 (8)	0.4558 (7)	-0.0323 (5)	6.0 (2)
C106	0.2551 (7)	0.4271 (6)	0.0470 (4)	4.7 (2)
C107	0.2364 (6)	0.3907 (5)	0.2242 (4)	3.2 (2)
C108	0.0940 (7)	0.3412 (6)	0.1981 (5)	4.8 (2)
C109	0.0278 (7)	0.3953 (7)	0.2262 (6)	6.3 (2)
C110	0.1036 (7)	0.5003 (7)	0.2798 (6)	7.0 (3)
C111	0.2412 (8)	0.5496 (7)	0.3051 (6)	7.4 (3)
C112	0.3107 (7)	0.4971 (6)	0.2788 (5)	5.2 (2)
C201	0.4992 (6)	0.2348 (5)	0.4363 (4)	2.9 (1)
C202	0.4475 (6)	0.3140 (5)	0.4428 (4)	3.6 (2)
C203	0.3958 (6)	0.3199 (6)	0.5056 (5)	4.4 (2)
C204	0.3979 (7)	0.2450 (7)	0.5650 (4)	4.9 (2)
C205	0.4516 (7)	0.1672 (7)	0.5608 (4)	5.0 (2)
C206	0.5031 (6)	0.1625 (5)	0.4977 (4)	3.6 (2)
C207	0.7321 (6)	0.2418 (5)	0.3976 (4)	3.2 (2)
C208	0.8224 (6)	0.3239 (6)	0.4695 (5)	4.9 (2)
C209	0.9575 (7)	0.3450 (7)	0.5010 (5)	5.8 (2)
C210	1.0036 (7)	0.2850 (7)	0.4628 (6)	6.5 (3)
C211	0.9176 (8)	0.2042 (8)	0.3918 (7)	8.2 (3)
C212	0.7813 (7)	0.1817 (7)	0.3588 (5)	5.8 (2)

C301	0.4273 (9)	-0.2016 (9)	0.0759 (7)	9.6 (3)
C302	0.264 (1)	-0.075 (1)	-0.0166 (6)	9.7 (4)
C303	0.129 (1)	-0.2958 (9)	0.0549 (7)	8.9 (4)
C401	-0.1925 (7)	-0.1131 (8)	0.2664 (6)	6.8 (3)
C402	-0.0156 (8)	-0.2435 (7)	0.2828 (6)	6.4 (3)
C403	0.0834 (8)	-0.0229 (8)	0.4089 (5)	6.3 (3)

Table 2. Geometric parameters (\AA , $^\circ$)

Co1—Cl1	2.205 (2)	Si1—C401	1.857 (9)
Co1—Cl2	2.220 (2)	Si1—C402	1.856 (9)
Co1—P1	2.405 (2)	Si1—C403	1.871 (9)
Co1—P2	2.348 (2)	Si1—C3	1.870 (7)
Fe1—C1	2.068 (6)	Si2—C301	1.83 (1)
Fe1—C2	2.041 (6)	Si2—C302	1.86 (1)
Fe1—C3	2.068 (7)	Si2—C303	1.84 (1)
Fe1—C4	2.041 (7)	Si2—C7	1.877 (8)
Fe1—C5	2.061 (7)	P1—C1	1.815 (6)
Fe1—C6	2.054 (7)	P1—C101	1.821 (7)
Fe1—C7	2.078 (7)	P1—C107	1.812 (7)
Fe1—C8	2.043 (6)	P2—C9	1.808 (7)
Fe1—C9	2.034 (6)	P2—C201	1.813 (7)
Fe1—C10	2.036 (7)	P2—C207	1.820 (7)
C1—C2	1.431 (9)	C6—C7	1.43 (1)
C2—C3	1.421 (9)	C7—C8	1.430 (9)
C3—C4	1.409 (9)	C8—C9	1.435 (9)
C4—C5	1.410 (9)	C9—C10	1.426 (9)
C1—C5	1.423 (9)	C6—C10	1.41 (1)
C101—C102	1.38 (1)	C201—C202	1.377 (9)
C102—C103	1.37 (1)	C202—C203	1.38 (1)
C103—C104	1.37 (1)	C203—C204	1.39 (1)
C104—C105	1.36 (1)	C204—C205	1.37 (1)
C105—C106	1.39 (1)	C205—C206	1.38 (1)
C101—C106	1.37 (1)	C201—C206	1.393 (9)
C107—C108	1.39 (1)	C207—C208	1.38 (1)
C108—C109	1.38 (1)	C208—C209	1.37 (1)
C109—C110	1.36 (1)	C209—C210	1.34 (1)
C110—C111	1.34 (1)	C210—C211	1.36 (1)
C111—C112	1.38 (1)	C211—C212	1.38 (1)
C107—C112	1.38 (1)	C207—C212	1.38 (1)
Cl1—Co1—Cl2	114.32 (9)	Cl2—Co1—P1	111.45 (8)
Cl1—Co1—P1	113.13 (8)	Cl2—Co1—P2	101.51 (8)
Cl1—Co1—P2	109.99 (9)	P1—Co1—P2	105.41 (7)

The data were collected using the *FLAT* method (Enraf-Nonius, 1990) in which each collection was carried out at an azimuthal angle Ψ calculated to minimize absorption by minimizing the path of the X-rays through the crystal. The structure was determined by Patterson methods using *SHELXS86* (Sheldrick, 1986) and refined by least squares using anisotropic thermal parameters for all non-H atoms. All H atoms were included in calculated positions, riding on their respective bonded atoms. Killean & Lawrence (1969) weights were used in the refinement. The calculations were performed on a VAX computer using *SHELXS86* and *MolEN* (Fair, 1990). Diagrams were drawn with *ORTEP* (Johnson, 1965) and *PLUTO* (Motherwell & Clegg, 1978).

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, complete geometry, least-squares-planes data and r.m.s. amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71130 (76 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HU1021]

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Acta Cryst. (1993). **C49**, 1619–1621

Structure of $\text{Ru}_4(\text{CO})_8(\text{O}_2\text{CCH}_2\text{CH}=\text{CH}_2)_4(\text{NMe}_3)_2$

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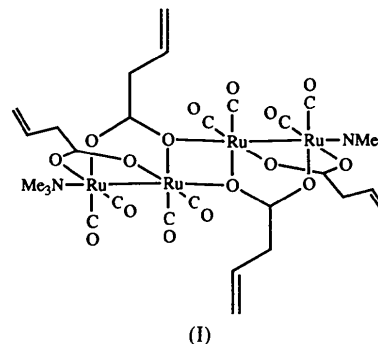
Abstract

The tetranuclear ruthenium carboxylate complex, bis(μ_3 -3-butenato)-bis(μ -3-butenato)-octacarbonylbis(trimethylamine)tetraruthenium(2 *Ru*—*Ru*), consists of a dimer of diruthenium carboxylates, $\text{Ru}_2(\text{CO})_4(\text{O}_2\text{CCH}_2\text{CH}=\text{CH}_2)_2$, held together by O-atom bridge bonds. The two terminal Ru atoms complete their six-coordination with trimethylamine ligands.

Comment

Dodecacarbonyltriruthenium reacts with several carboxylic acids to provide polymeric compounds $[\text{Ru}(\text{CO})_2(\text{RCO}_2)]_n$. Subsequent reactions of these polymeric derivatives with various Lewis bases result in the formation of dimeric carboxylate complexes of the type $\text{Ru}_2(\text{CO})_4(\text{RCO}_2)_2L_2$ (Crooks, Johnson, Lewis, Williams & Gamlen, 1969). The X-ray structure of $(\text{CH}_3\text{CO}_2)_2\text{Ru}_2(\text{CO})_4(\text{C}_5\text{H}_5\text{N})_2$, as well as that of an analogous osmium complex, $(\text{CH}_3\text{CO}_2)_2\text{Os}_2(\text{CO})_6$, have been

determined (Kilbourn, 1971; Bullitt & Cotton, 1971). The overall structures of these complexes possess sixfold coordination about the metal atoms with *cis*-bridging acetate groups. We have isolated crystals of a ruthenium carboxylate complex (I) containing features of both the



polymeric and dimeric carboxylate derivatives from the reaction of $\text{Ru}_3(\text{CO})_{12}$ and 1-butenic acid in the presence of trimethylamine *N*-oxide. The structure reported in this work is similar to that reported for the tetranuclear compound $(\text{CH}_3\text{CO}_2)_4\text{Ru}_4(\text{CO})_8\{\text{P}[(\text{CH}_2)_3\text{CH}_3]_3\}_2$ (Bianchi, Frediani, Nardelli & Pelizzi, 1981).

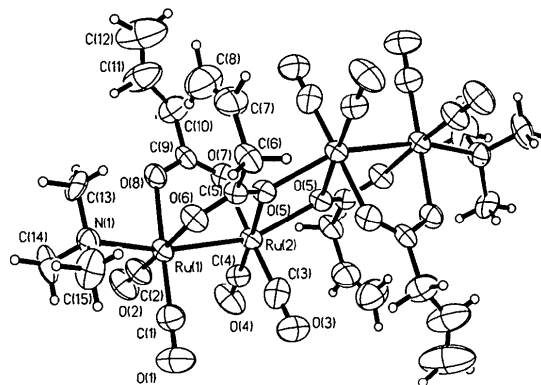


Fig. 1. Thermal ellipsoid plot (50% probability) of $\text{Ru}_4(\text{CO})_8(\text{O}_2\text{CCH}_2\text{CH}=\text{CH}_2)_4(\text{NMe}_3)_2$. H atoms have been drawn as spheres with arbitrary radii. For clarity only one of the atoms of each of the disordered atomic pairs [C(13), C(13'); C(14), C(14'); C(15), C(15')] is displayed.

Experimental

Crystal data

$[\text{Ru}_4(\text{C}_4\text{H}_5\text{O}_2)_4(\text{C}_3\text{H}_9\text{N})_2(\text{CO})_8]$
 $M_r = 1086.91$
 Monoclinic
 $P2_1/c$
 $a = 7.628 (2) \text{ \AA}$
 $b = 19.526 (4) \text{ \AA}$
 $c = 13.120 (3) \text{ \AA}$

$D_x = 1.863 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 $\lambda = 0.71073 \text{ \AA}$
 Cell parameters from 25 reflections
 $\theta = 19.89\text{--}34.23^\circ$
 $\mu = 1.568 \text{ mm}^{-1}$
 $T = 197 \text{ K}$