Chloro[diferrocenyl(phenyl)phosphine]gold(I)

By Peter G. Jones, Cristina Freire Erdbrügger, Rainer Hohbein and Einhard Schwarzmann

Institut für Anorganische Chemie der Universität, Tammannstrasse 4, 3400 Göttingen,
Federal Republic of Germany

Abstract. $[AuCl\{P(C_6H_5)(C_{10}H_9Fe)_2\}]$, $M_r = 710\cdot5$, monoclinic, $P2_1/n$, $a = 10\cdot205$ (2), $b = 18\cdot014$ (4), $c = 12\cdot680$ (3) Å, $\beta = 98\cdot29$ (2)°, $V = 2306\cdot5$ Å³, $Z = 4$, $D_x = 2\cdot04$ Mg m⁻³, $\lambda(Mo\,K\alpha) = 0\cdot71069$ Å, $\mu = 7\cdot8$ mm⁻¹, $F(000) = 1368$, $T = 293$ K. The structure was refined to R 0·035 for 3523 unique observed reflections. The coordination geometry at the Au atom is linear, with Au–Cl 2·289 (2), Au–P 2·234 (2) Å, Cl–Au–P 176·1 (1)°. There are no unusually short intermolecular contacts. The Fe–C distances lie in the range 2·025 (8)–2·058 (8) Å, mean 2·041 (8) Å. The C_5H_4 rings of the ferrocene moieties are approximately eclipsed (rotated by 12, 14°, respectively).

Experimental. The title compound was obtained as a brown powder from the reaction of carbonylgold(I) chloride with diferrocenyl(phenyl)phosphine in benzene, and recrystallized from dichloromethane/petrol ether as orange to red plates. A full elemental analysis confirmed the expected composition.

A crystal 0·55 × 0·35 × 0·15 mm was mounted in a glass capillary. 5761 profile-fitted intensities (Clegg, 1981) were registered on a Stoe–Siemens four-circle diffractometer using monochromated Mo $K\alpha$ radiation ($2\theta_{max}$ 50°, quadrant $-h+k\pm l$ and some $+h$ equivalents). An absorption correction based on ψ scans was applied, with transmission factors 0·34 to 0·99. The intensities of 3 check reflections showed no significant variation. Merging equivalents gave 4045 unique reflections (R_{int} 0·026, index ranges h −12 to +12, k 0 to 21, l 0 to 15), 3530 with $F > 4\sigma(F)$. Cell constants were refined from 2θ values of 40 reflections in the range 20–23°.

The structure was solved by the heavy-atom method and subjected to full-matrix least-squares refinement on F. All non-H atoms were refined anisotropically; H atoms were included using a riding model. The final R value was 0·0352, wR 0·0412 for 280 parameters, 3523 reflections (seven low-angle reflections were omitted because of extinction effects). The weighting scheme was $w^{-1} = \sigma^2(F) + 0\cdot0004F^2$. Max. shift/e.s.d. = 0·003, max. residual electron density within ±1·0 e Å⁻³ near Au, $S = 1\cdot52$. The program system was *SHELX* (Sheldrick, 1986), locally modified by its

Table 1. *Atomic coordinates* (×10⁴) *and equivalent isotropic displacement parameters* (Å² × 10³)

	x	y	z	U_{eq}*
Au	4770·2 (2)	6930·5 (1)	1249·4 (2)	32 (1)
Cl	7033 (2)	6856 (1)	1522 (1)	47 (1)
Fe(1)	2078 (1)	8797·9 (5)	715 (1)	36 (1)
Fe(2)	2623 (1)	5743·2 (5)	−1123 (1)	32 (1)
P	2558 (2)	6918 (1)	997 (1)	30 (1)
C(11)	1892 (6)	6814 (3)	2248 (4)	32 (2)
C(12)	675 (6)	6480 (4)	2277 (5)	40 (2)
C(13)	165 (7)	6444 (4)	3221 (5)	45 (2)
C(14)	848 (8)	6749 (4)	4131 (5)	48 (3)
C(15)	2060 (8)	7083 (4)	4114 (5)	53 (3)
C(16)	2594 (7)	7109 (4)	3169 (5)	43 (2)
C(111)	1702 (6)	7710 (3)	390 (4)	31 (2)
C(112)	1972 (7)	8101 (3)	−552 (4)	41 (2)
C(113)	1031 (8)	8662 (4)	−784 (5)	60 (3)
C(114)	178 (8)	8646 (4)	−9 (6)	56 (3)
C(115)	559 (7)	8055 (3)	699 (5)	42 (2)
C(121)	3778 (9)	8896 (5)	1762 (7)	70 (3)
C(122)	3840 (8)	9371 (5)	890 (7)	65 (3)
C(123)	2776 (9)	9858 (4)	839 (7)	70 (3)
C(124)	2034 (9)	9712 (5)	1657 (7)	71 (3)
C(125)	2689 (10)	9113 (5)	2251 (6)	73 (3)
C(211)	1932 (6)	6138 (3)	203 (4)	30 (2)
C(212)	2432 (6)	5394 (3)	381 (4)	36 (2)
C(213)	1691 (7)	4931 (3)	−383 (5)	42 (2)
C(214)	759 (7)	5365 (4)	−1034 (5)	42 (2)
C(215)	882 (6)	6110 (3)	−667 (4)	33 (2)
C(221)	3970 (8)	6445 (4)	−1663 (6)	63 (3)
C(222)	2946 (10)	6250 (5)	−2504 (6)	74 (4)
C(223)	3004 (9)	5474 (5)	−2612 (5)	67 (3)
C(224)	3997 (8)	5204 (4)	−1840 (6)	61 (3)
C(225)	4599 (8)	5806 (5)	−1255 (6)	60 (3)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 2. *Selected bond lengths* (Å) *and valence angles* (°)

Au–Cl	2·289 (2)	Au–P	2·234 (2)
Fe(1)–C(111)	2·028 (6)	Fe(1)–C(112)	2·029 (6)
Fe(1)–C(113)	2·056 (7)	Fe(1)–C(114)	2·041 (7)
Fe(1)–C(115)	2·046 (7)	Fe(1)–C(121)	2·034 (8)
Fe(1)–C(122)	2·058 (8)	Fe(1)–C(123)	2·037 (8)
Fe(1)–C(124)	2·039 (8)	Fe(1)–C(125)	2·038 (8)
Fe(2)–C(211)	2·042 (5)	Fe(2)–C(212)	2·043 (6)
Fe(2)–C(213)	2·045 (7)	Fe(2)–C(214)	2·039 (7)
Fe(2)–C(215)	2·056 (6)	Fe(2)–C(221)	2·055 (9)
Fe(2)–C(222)	2·043 (8)	Fe(2)–C(223)	2·040 (8)
Fe(2)–C(224)	2·025 (8)	Fe(2)–C(225)	2·050 (8)
P–C(11)	1·824 (6)	P–C(111)	1·787 (6)
P–C(211)	1·792 (6)		
Cl–Au–P	176·1 (1)	Au–P–C(11)	111·9 (2)
Au–P–C(111)	118·3 (2)	Au–P–C(211)	111·0 (2)
C(11)–P–C(111)	103·7 (3)	C(11)–P–C(211)	105·1 (3)
C(111)–P–C(211)	105·7 (2)		

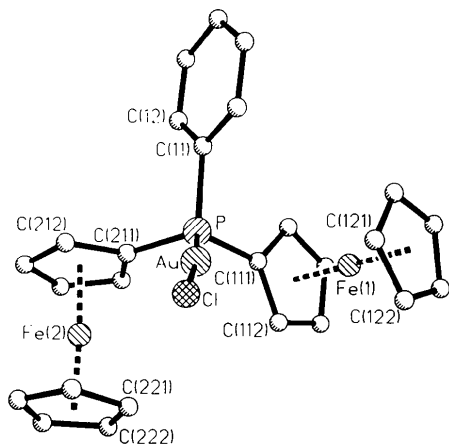author. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974).

Fig. 1. The molecule of the title compound in the crystal, showing the atom-labelling scheme. Radii arbitrary, H atoms omitted.

Final atomic coordinates are given in Table 1,* and selected geometry in Table 2. A perspective view of the title compound is shown in Fig. 1.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and all bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44849 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1988). C44, 1303–1305

Structure of (3,5-Dimethylpyridine)bis(1,10-phenanthroline)copper(II) Diperchlorate

BY D. Y. JETER AND W. J. CASTEEL JR

Department of Chemistry, Rhodes College, Memphis, TN 38112, USA

S. M. CONDREN, A. M. HOBSON AND T. E. STILES

Department of Chemistry, Christian Brothers College, Memphis, TN 38104, USA

AND A. W. CORDES

Department of Chemistry and Biochemistry, University of Arkansas, Fayetteville, AR 72701, USA

Abstract. $[Cu(C_7H_9N)(C_{12}H_8N_2)_2](ClO_4)_2$, $M_r = 730 \cdot 0$, monoclinic, $P2_1/n$, $a = 18 \cdot 430$ (3), $b = 9 \cdot 122$ (2), $c = 18 \cdot 744$ (8) Å, $\beta = 97 \cdot 92$ (3)°, $V = 3121$ (3) Å^3, $Z = 4$, $D_m = 1 \cdot 56$ (1), $D_x = 1 \cdot 55$ g cm^{-3}, $\lambda(Mo\ K\alpha) = 0 \cdot 71073$ Å, $\mu = 9 \cdot 3$ cm^{-1}, $F(000) = 1492$, $T = 293$ K, $R = 0 \cdot 081$ for 1312 unique observed reflections. The Cu-atom environment is approximately square pyramidal, with the apical position occupied by an N atom of a 1,10-phenanthroline (phen) molecule. The Cu atom is $0 \cdot 256$ (3) Å above the basal plane; the four basal N atoms are planar within $0 \cdot 10$ (2) Å. The Cu—N distances to the phen ligand in the basal plane are $2 \cdot 01$ (1), to the pyridine $2 \cdot 04$ (1) and to the phen which spans the basal-apical positions $2 \cdot 12$ (1) Å. The latter phen is folded [deviations from planarity of up to $0 \cdot 09$ (2) Å] while the basal phen is planar within $0 \cdot 03$ (2) Å.

Experimental. Complex (I) prepared by reaction of bis(1,10-phenanthroline)copper(II) perchlorate with