Smith, D. C., Miskowski, V. M., Mason, W. R. & Gray, H. B. (1990). *J. Am. Chem. Soc.* **112**, 3752–3767.
Stiegman, A. E., Rice, S. F., Gray, H. B. & Miskowski, V. M. (1987). *Inorg. Chem.* **26**, 1112–1116.
Wong, Y.-S. & Lippard, S. J. (1977). *J. Chem. Soc. Chem. Commun.* pp. 824–825.

Acta Cryst. (1993). C**49**, 1602–1603

Chloro(dimethylphenylphosphine)gold(I)

P. D. Cookson and E. R. T. Tiekink

Department of Chemistry, University of Adelaide, Adelaide, SA 5001, Australia

(*Received* 7 *January* 1992; *accepted* 15 *March* 1993)

Abstract

Two molecules of [(Me_2Ph)PAuCl] comprise the asymmetric unit; these differ in the relative orientations of the phosphine-bound substituents. The Au—P bond distances are 2.214 (6) and 2.205 (5) Å for molecules (1) and (2), respectively; the Au—Cl distances are 2.276 (6) and 2.273 (6) Å and the P—Au—Cl angles are 177.2 (3) and 175.4 (2)°, respectively.

Comment

The geometries found for the independent Au atoms in [(Me_2Ph)PAuCl] are as found in related derivatives. Of interest is the systematic variation of the Au—P bond distances in complexes of general formula [R_3PAuCl]. Whereas the Au—Cl bond distances are equal within experimental error in the series of complexes R = *cyclo*-C_6H_{11} (Muir, Muir, Pulgar, Jones & Sheldrick, 1985), R = *o*-MeC_6H_4 (Harker & Tiekink, 1990), R = Ph (Baenziger, Bennett & Soboroff, 1976), R = Et (Tiekink, 1989) and R_3 = Me_2Ph, the trend in the Au—P bond distances decreases in this order, *i.e.* Au—P 2.242 (4) for R = *cyclo*-C_6H_{11}, 2.243 (2) for R = *o*-MeC_6H_4, 2.235 (3) for R = Ph, 2.232 (9) and 2.231 (8) (two molecules) for R = Et, and 2.214 (6) and 2.205 (5) Å (two molecules) for R_3 = Me_2Ph; the relatively high experimental errors not withstanding. This trend follows that predicted from cone-angle considerations (Tolman, 1977).

The structure is molecular, there being no significant intermolecular contacts except for an Au(1)···Au(2) interaction of 3.262 (2) Å. The two independent molecules differ from each other in the relative orientations of the phosphine-bound substituents, as shown by the torsion angles Cl(n)—Au(n)—P(n)—C(n2),C(n3),C(n11) of −47, −165 and 72° for n = 1, and −178, 68 and −56° for n = 2, respectively.

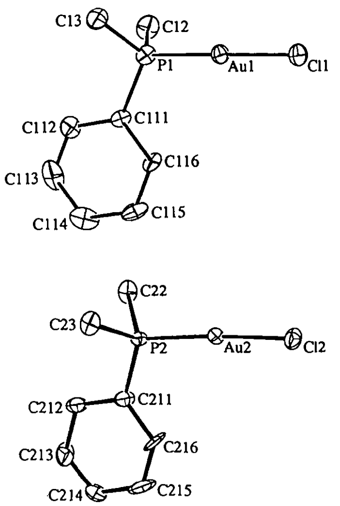

Fig. 1. Molecular structure of [(Me_2Ph)PAuCl] showing the crystallographic numbering scheme; diagram drawn at 30% probability levels (Johnson, 1976).

Experimental

Crystal data

[$AuCl(C_8H_{11}P)$]
M_r = 370.6
Orthorhombic
$P2_12_12_1$
a = 12.639 (4) Å
b = 16.931 (6) Å
c = 9.458 (3) Å
V = 2024 (1) Å^3
Z = 8
D_x = 2.432 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 8.0-12.8°
μ = 148.550 cm^{-1}
T = 295 K
Block
0.27 × 0.27 × 0.14 mm
Colourless

Data collection

AFC-6R diffractometer
$\omega/2\text{-}\theta$ scans
Absorption correction: refined from ΔF (Walker & Stuart, 1983)
$T_{\min}$ = 0.945, $T_{\max}$ = 1.058
3015 measured reflections
2143 independent reflections
1608 observed reflections [$I > 3.0\sigma(I)$]
$R_{\rm int}$ = 4.58
$\theta_{\max}$ = 25.3°
$h = -1 \rightarrow 15$
$k = 0 \rightarrow 18$
$l = -11 \rightarrow 0$
3 standard reflections monitored every 400 reflections intensity variation: −2.26%

Refinement

Refinement on F
Final R = 0.0350
wR = 0.0390
S = 2.380
1608 reflections
199 parameters
H atoms included in calculated positions
Weighting scheme based on measured e.s.d.'s
$(\Delta/\sigma)_{\max}$ = 0.0070
$\Delta\rho_{\max}$ = 0.93 e Å^{-3}
$\Delta\rho_{\min}$ = −0.98 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Acta Crystallographica Section C
ISSN 0108-2701 ©1993

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1992). Cell refinement: *MSC/AFC Diffractometer Control software*. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1992). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1986). Program(s) used to refine structure: *TEXSAN LS*. Software used to prepare material for publication: *TEXSAN FINISH*.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å²)

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

	x	y	z	$U_{\rm eq}$
Au(1)	0.54801 (7)	0.35615 (5)	0.5251 (1)	0.0403
Au(2)	0.43693 (7)	0.32211 (5)	0.21977 (10)	0.0337
Cl(1)	0.6889 (5)	0.2949 (4)	0.4228 (8)	0.0626
Cl(2)	0.4269 (5)	0.4549 (3)	0.1866 (7)	0.0502
P(1)	0.4152 (5)	0.4157 (4)	0.6343 (6)	0.0376
P(2)	0.4450 (5)	0.1923 (3)	0.2333 (6)	0.0310
C(12)	0.377 (2)	0.372 (2)	0.801 (3)	0.0550
C(13)	0.296 (2)	0.415 (1)	0.542 (3)	0.0679
C(22)	0.459 (2)	0.154 (1)	0.405 (2)	0.0451
C(23)	0.561 (2)	0.155 (1)	0.152 (2)	0.0439
C(111)	0.442 (2)	0.516 (1)	0.677 (2)	0.0275
C(112)	0.362 (2)	0.568 (1)	0.712 (3)	0.0447
C(113)	0.384 (2)	0.642 (2)	0.747 (3)	0.0662
C(114)	0.487 (2)	0.670 (2)	0.745 (2)	0.0541
C(115)	0.568 (2)	0.620 (2)	0.709 (3)	0.0538
C(116)	0.545 (2)	0.544 (1)	0.675 (2)	0.0339
C(211)	0.334 (2)	0.144 (1)	0.159 (3)	0.0426
C(212)	0.329 (2)	0.060 (1)	0.165 (3)	0.0490
C(213)	0.246 (2)	0.025 (1)	0.093 (3)	0.0630
C(214)	0.174 (2)	0.065 (2)	0.027 (4)	0.0771
C(215)	0.182 (2)	0.147 (2)	0.018 (3)	0.0740
C(216)	0.262 (2)	0.184 (1)	0.078 (4)	0.0742

Table 2. *Geometric parameters* (Å, °)

Au(1)—Cl(1)	2.276 (6)	P(1)—C(13)	1.74 (2)
Au(1)—P(1)	2.214 (6)	P(1)—C(111)	1.77 (2)
Au(2)—Cl(2)	2.273 (6)	P(2)—C(22)	1.76 (2)
Au(2)—P(2)	2.205 (5)	P(2)—C(23)	1.77 (2)
P(1)—C(12)	1.81 (2)	P(2)—C(211)	1.77 (2)
Cl(1)—Au(1)—P(1)	177.2 (3)	C(13)—P(1)—C(111)	106 (1)
Cl(2)—Au(2)—P(2)	175.4 (2)	Au(2)—P(2)—C(22)	115.2 (7)
Au(1)—P(1)—C(12)	114.9 (9)	Au(2)—P(2)—C(23)	111.6 (8)
Au(1)—P(1)—C(13)	114.9 (9)	Au(2)—P(2)—C(211)	113.7 (8)
Au(1)—P(1)—C(111)	113.4 (8)	C(22)—P(2)—C(23)	100 (1)
C(12)—P(1)—C(13)	101 (1)	C(22)—P(2)—C(211)	106 (1)
C(12)—P(1)—C(111)	104 (1)	C(23)—P(2)—C(211)	108 (1)

[$(\mathrm{Me_2Ph})\mathrm{PAuCl}$] was prepared according to the literature procedure (Al-Saády, McAuliffe, Parish & Sandbank, 1985). Scans of (0.94 + 0.35tanθ)° were made at 32° min^{-1} in ω. The ratio of peak to background counting times was 2:1. The absolute configuration was determined by reversing the signs of the reflections (other hand: $R = 0.047$ and $wR = 0.050$).

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 71195 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1052]

References

Al-Saády, A. K., McAuliffe, C. A., Parish, R. V. & Sandbank, J. A. (1985). *Inorg. Synth.* **23**, 191-194.

Baenziger, N. C., Bennett, W. E. & Soboroff, D. M. (1976). *Acta Cryst.* B**32**, 962-963.

Harker, C. S. W. & Tiekink, E. R. T. (1990). *Acta Cryst.* C**46**, 1546-1547.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Molecular Structure Corporation (1992). *TEXSAN. Single Crystal Structure Analysis Package*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Molecular Structure Corporation (1992). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Muir, J. A., Muir, M. M., Pulgar, L. B., Jones, P. G. & Sheldrick, G. M. (1985). *Acta Cryst.* C**41**, 1174-1176.

Sheldrick, G. M. (1986). *SHELXS86. Program for the Solution of Crystal Structures*. Univ. of Göttingen, Germany.

Tiekink, E. R. T. (1989). *Acta Cryst.* C**45**, 1233-1234.

Tolman, C. A. (1977). *Chem. Rev.* **77**, 313-348.

Walker, N. & Stuart, D. (1983). *Acta Cryst.* A**39**, 158-166.

Acta Cryst. (1993). C49, 1603–1606

Structure of (η^5-Cyclopentadienyl)bis(dimethylphenylphosphine)palladium(II) Perchlorate

LJUBICA MANOJLOVIĆ-MUIR, RONALD J. CROSS AND ROBERT W. HOYLE

Department of Chemistry, University of Glasgow, Glasgow G12 8QQ, Scotland

(*Received* 23 *October* 1992; *accepted* 11 *March* 1993)

Abstract

In the crystal structure of $[\mathrm{Pd}(\eta^5\text{-}\mathrm{C_5H_5})(\mathrm{PMe_2Ph})_2]\mathrm{ClO_4}$ the asymmetric unit contains two cations and two anions, one cation adopting a nearly eclipsed, and the other a staggered orientation of the $\mathrm{C_5H_5}$ ring with respect to the perpendicular $\mathrm{Pd(PMe_2Ph)_2}$ fragment. The coordination geometry around the metal atom is distorted square planar in both rotamers. The Pd—P distances are 2.250 (2)–2.253 (2) Å and the Pd—C distances vary from 2.252 (8)–2.343 (6) Å.

Comment

Interest in transition-metal complexes $[M(\eta^5\text{-}\mathrm{C_5H_5})L_x]$ stems from realization that binding to a ML_x fragment with metal-atom orbitals of less than cylindrical distribution causes the $\mathrm{C_5H_5}$ ring to lose its D_{5h} symmetry.

Significant variations in the C—M and C—C bond lengths have been observed in the complexes of the