Acta Cryst. (1994). C**50**, 39–40

Structure of Trichloro(triphenylphosphine)-gold(III), $[AuCl_3\{P(C_6H_5)_3\}]$

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(*Received* 2 *February* 1993; *accepted* 14 *June* 1993)

Abstract

The structure of the title compound displays the same *trans* effects and slight distortions from planarity as previously reported trihalide(trialkylphosphine)gold(III) structures.

Comment

Reaction of $Au(SPh)PPh_3$ with $PhI.Cl_2$ results in a yellow solid which when recrystallized by diffusion of ether into a dichloromethane solution, undergoes a disproportionation reaction. One of the products is structurally characterized here. The compound is shown in Fig. 1.

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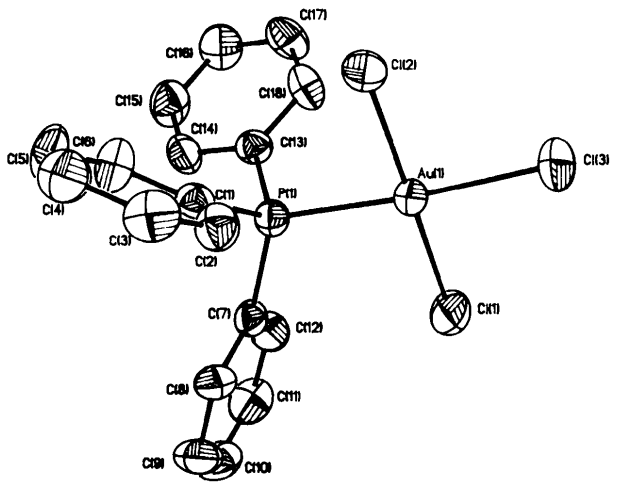

Fig. 1. A drawing of $[AuCl_3(PPh_3)]$ showing the atomic labeling scheme with thermal ellipsoids representing 50% probability.

The structure of $AuCl_3(PPh_3)$ has been reported previously (Bandoli, Clemente, Marangoni & Catalini, 1973) in the triclinic space group $P\bar{1}$. The structure presented here does not differ significantly from that reported. The longer Au—Cl distance of the chlorine *trans* to the phosphine and a slight deviation from planarity of ligands about the Au center are observed. $AuBr_3(PEt_3)$ (Eggleston, Chodosh, Hill & Girard, 1984) and $AuBr_3(PMe_3)$ (Perutz & Weisz, 1946) have also been structurally characterized. These show similar *trans* effects and deviations from planarity.

Experimental

Crystal data

$[AuCl_3(C_{18}H_{15}P)]$
M_r = 565.6
Monoclinic
$P2_1/n$
a = 9.881 (5) Å
b = 13.393 (6) Å
c = 14.396 (5) Å
β = 90.87 (3)°
V = 1905 (1) Å^3
Z = 4
D_x = 1.97 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 25–30°
μ = 8.213 mm^{-1}
T = 293 K
0.2 × 0.2 × 0.1 mm
Yellow
Crystal source: CH_2Cl_2/Et_2O

Data collection

Nicolet *R3m/E* diffractometer
Wycoff scans
Absorption correction: empirical
T_{min} = 0.652, T_{max} = 0.897
3839 measured reflections
3721 independent reflections
2521 observed reflections $[F_o^2 > 3\sigma(F_o)^2]$
θ_{max} = 22.5°
h = 0 → 12
k = 0 → 16
l = −18 → 18
3 standard reflections monitored every 97 reflections
intensity variation: <1%

Refinement

Refinement on F
R = 0.029
wR = 0.028
S = 1.25
2521 reflections
208 parameters
H-atom parameters not refined
$w = [\sigma^2(F_o) + 0.00019F_o^2]^{-1}$
$(\Delta/\sigma)_{max}$ = 0.019
$\Delta\rho_{max}$ = 0.71 e Å^{-3}
$\Delta\rho_{min}$ = −1.45 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

The program used to solve and refine the structure was *SHELXTL* (Sheldrick, 1985).

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

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

	x	y	z	U_{eq}
Au(1)	0.4447 (1)	−0.0539 (1)	0.1402 (1)	0.034 (1)
Cl(1)	0.6507 (2)	0.0222 (2)	0.1365 (2)	0.062 (1)
Cl(2)	0.2367 (2)	−0.1281 (1)	0.1479 (1)	0.051 (1)

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Cl(3)	0.5326 (2)	−0.1960 (2)	0.0672 (1)	0.060 (1)
P(1)	0.3507 (2)	0.0788 (1)	0.2215 (1)	0.033 (1)
C(1)	0.1853 (6)	0.1107 (5)	0.1710 (5)	0.035 (2)
C(2)	0.1659 (7)	0.1029 (5)	0.0753 (5)	0.046 (2)
C(3)	0.0429 (7)	0.1285 (6)	0.0355 (5)	0.055 (3)
C(4)	−0.0613 (7)	0.1615 (6)	0.0903 (6)	0.059 (3)
C(5)	−0.0416 (7)	0.1693 (7)	0.1840 (7)	0.068 (3)
C(6)	0.0815 (7)	0.1445 (6)	0.2260 (5)	0.053 (3)
C(7)	0.4505 (6)	0.1928 (5)	0.2236 (4)	0.035 (2)
C(8)	0.4226 (6)	0.2679 (5)	0.1592 (5)	0.042 (2)
C(9)	0.4992 (8)	0.3573 (6)	0.1618 (6)	0.057 (3)
C(10)	0.5988 (8)	0.3685 (7)	0.2269 (6)	0.061 (3)
C(11)	0.6279 (7)	0.2958 (7)	0.2906 (6)	0.059 (3)
C(12)	0.5552 (6)	0.2059 (5)	0.2886 (5)	0.047 (3)
C(13)	0.3355 (6)	0.0396 (5)	0.3417 (5)	0.037 (2)
C(14)	0.2941 (7)	0.1108 (5)	0.4070 (5)	0.047 (3)
C(15)	0.2750 (8)	0.0835 (6)	0.4981 (5)	0.059 (3)
C(16)	0.2968 (8)	−0.0149 (7)	0.5257 (5)	0.060 (3)
C(17)	0.3386 (8)	−0.0850 (6)	0.4632 (5)	0.057 (3)
C(18)	0.3576 (7)	−0.0581 (5)	0.3705 (5)	0.047 (2)

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

Au(1)—Cl(1)	2.278 (2)	Au(1)—Cl(2)	2.287 (2)
Au(1)—Cl(3)	2.347 (2)	Au(1)—P(1)	2.329 (2)
P(1)—C(1)	1.828 (6)	P(1)—C(7)	1.818 (6)
P(1)—C(13)	1.817 (7)	C(1)—C(2)	1.392 (10)
C(1)—C(6)	1.382 (10)	C(2)—C(3)	1.379 (10)
C(3)—C(4)	1.379 (11)	C(4)—C(5)	1.364 (14)
C(5)—C(6)	1.390 (11)	C(7)—C(8)	1.392 (9)
C(7)—C(12)	1.394 (9)	C(8)—C(9)	1.418 (10)
C(9)—C(10)	1.357 (12)	C(10)—C(11)	1.366 (12)
C(11)—C(12)	1.402 (11)	C(13)—C(14)	1.404 (10)
C(13)—C(18)	1.389 (10)	C(14)—C(15)	1.378 (11)
C(15)—C(16)	1.392 (12)	C(16)—C(17)	1.368 (11)
C(17)—C(18)	1.398 (11)		
Cl(1)—Au(1)—Cl(2)	178.4 (1)	Cl(1)—Au(1)—Cl(3)	90.9 (1)
Cl(2)—Au(1)—Cl(3)	90.5 (1)	Cl(1)—Au(1)—P(1)	91.9 (1)
Cl(2)—Au(1)—P(1)	86.6 (1)	Cl(3)—Au(1)—P(1)	175.5 (1)
Au(1)—P(1)—C(1)	109.8 (2)	Au(1)—P(1)—C(7)	115.4 (2)
C(1)—P(1)—C(7)	107.0 (3)	Au(1)—P(1)—C(13)	107.3 (2)
C(1)—P(1)—C(13)	111.1 (3)	C(7)—P(1)—C(13)	106.2 (3)
P(1)—C(1)—C(2)	119.0 (5)	P(1)—C(1)—C(6)	121.0 (5)
C(2)—C(1)—C(6)	120.0 (6)	C(1)—C(2)—C(3)	120.0 (7)
C(2)—C(3)—C(4)	120.2 (7)	C(3)—C(4)—C(5)	119.6 (7)
C(4)—C(5)—C(6)	121.5 (8)	C(1)—C(6)—C(5)	118.8 (7)
P(1)—C(7)—C(8)	119.5 (5)	P(1)—C(7)—C(12)	120.9 (5)
C(8)—C(7)—C(12)	119.6 (6)	C(7)—C(8)—C(9)	119.5 (6)
C(8)—C(9)—C(10)	119.5 (7)	C(9)—C(10)—C(11)	121.9 (8)
C(10)—C(11)—C(12)	119.7 (7)	C(7)—C(12)—C(11)	119.7 (7)
P(1)—C(13)—C(14)	118.0 (5)	P(1)—C(13)—C(18)	122.8 (5)
C(14)—C(13)—C(18)	119.1 (6)	C(13)—C(14)—C(15)	120.2 (7)
C(14)—C(15)—C(16)	119.9 (7)	C(15)—C(16)—C(17)	120.7 (7)
C(16)—C(17)—C(18)	119.8 (7)	C(13)—C(18)—C(17)	120.3 (7)

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71402 (26 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR1064]

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Acta Cryst. (1994). C**50**, 40–41

Structure of Dichlorobis(μ-hydroxo)-bis(μ_3-oxo)octaphenyltetratin(IV), $[Sn_4Cl_2(O)_2(OH)_2(C_6H_5)_8]$

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(*Received* 23 *November* 1992; *accepted* 14 *June* 1993)

Abstract

The structure of the title compound, dichloro-1κCl,3κCl-di-μ-hydroxo-1:2κ^2O;3:4κ^2O-di-μ_3-oxo-1:2:4κ^3O;2:3:4κ^3O-octaphenyl-1κ^2C,2κ^2C,3κ^2C,-4κ^2C-*quadro*-tetratin(IV), consists of an almost planar array of four Sn^{IV} atoms bridged by two O^{2-} and two OH^- ligands. Each Sn atom is bonded to two phenyl groups and the two terminal Sn atoms of the array are bonded to Cl^- ligands. In this way, each Sn atom possesses a rather distorted trigonal bipyramidal coordination geometry.

Comment

$[Sn(Cl)Ph_2(\mu\text{-}O)(\mu\text{-}OH)SnPh_2]_2$ was obtained as a contaminant in the preparation of $[Ph_2SnCl\{\mu\text{-}CH_2\text{-}P(O)Ph_2\}]_2$ from $[Ph_2SnCl_2]$ and $[Li\{CH_2P(O)Ph_2\}]$, believed to occur due to the presence of traces of

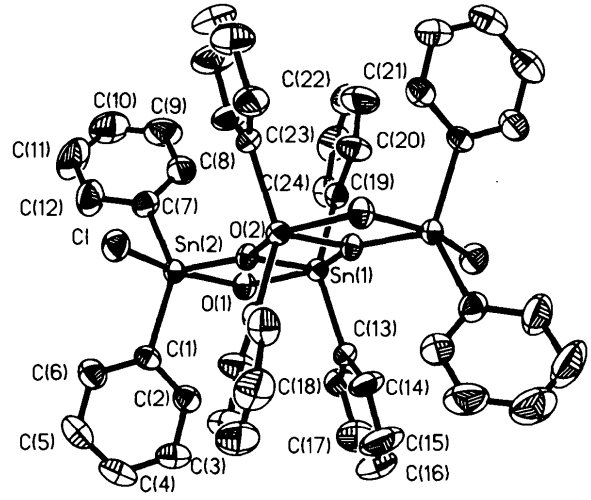

Fig. 1. View of $[Sn(Cl)Ph_2(\mu\text{-}O)(\mu\text{-}OH)SnPh_2]_2$. H atoms have been omitted; thermal ellipsoids have been drawn at the 50% probability level.