Dichloro-1κCl,3κCl-bis[μ-methylenebis-(diphenylphosphine)]-1:2κ²P:P';2:3κ²P:P'-trigold(I) perchlorate

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Key indicators

Single-crystal X-ray study

$T = 293$ K

Mean σ(I–O) = 0.022 Å

R factor = 0.054

wR factor = 0.176

Data-to-parameter ratio = 21.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $[Au_3Cl_2(C_{25}H_{22}P_2)_2]ClO_4$, was synthesized from μ-bis(diphenylphosphinomethyl)dichlorodigold(I) and an excess of silver perchlorate in dichloromethane/methanol. Three Au atoms in a triangle are connected by two bridging bis(diphenylphosphino)methane ligands.

Comment

About 20 years ago, Uson *et al.* (1983) reported that a reaction of $Au_2(dppm)Cl_2$ [dppm = bis(diphenylphosphino)methane] with an equimolar amount of $AgClO_4$, followed by reaction with dppm and Au(tht)Cl (tht = tetrahydrothiophene), led to the title compound, (I), but no crystal structure was determined. In our efforts to prepare $[Au_2(dppm)]^{2+}$ as a precursor to polynuclear gold(I) complexes by removing chloride from $Au_2(dppm)Cl_2$ with an excess of $AgClO_4$, this same trinuclear complex was isolated. The crystal structure of this complex is reported here.

(I)
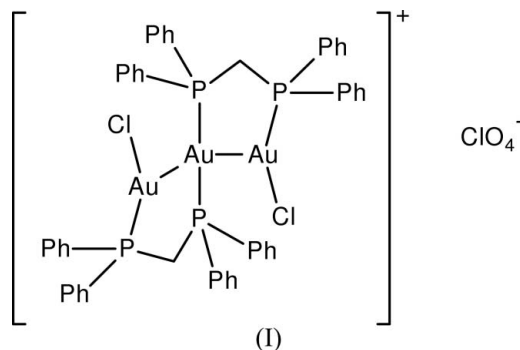
As shown in Fig. 1, Au2 is bonded to two P atoms, and the other two Au atoms are coordinated by one P and one Cl atom each, in a distorted linear arrangement (Table 1). The three Au^I atoms form a triangle, and the perchlorate anion is far from any metal center. The Au···Au separations are less than the van der Waals separation of *ca* 3.6 Å (White-Morris *et al.*, 2002). At these distances, aurophilic attractions are expected, leading to the triangular rather than a linear Au_3 structure.

Experimental

$Au_2(dppm)Cl_2$ (0.03 g, 0.035 mmol) was dissolved in dichloromethane (3 ml), to which was added dropwise a methanolic solution of $AgClO_4\cdot7H_2O$ (0.058 g, 0.17 mmol). After stirring for 3 h at room temperature, the AgCl precipitate was removed by filtration. The solvent was then removed *in vacuo* to give a yellow solid. The crude product was obtained by extraction with dichloromethane (yield 0.059 g, 78%). Block-shaped crystals were grown by slow evaporation of a solution in a mixture of acetone and ethanol (2:1 *v/v*).

Crystal data

[Au₃Cl₂(C₂₅H₂₂P₂)₂]ClO₄

$[Au_3Cl_2(C_{25}H_{22}P_2)_2]ClO_4$
$M_r = 1529.98$
Triclinic, $P\bar{1}$
$a = 9.8043\ (10)$ Å
$b = 15.1538\ (16)$ Å
$c = 18.8497\ (19)$ Å
$\alpha = 104.648\ (2)°$
$\beta = 102.068\ (2)°$
$\gamma = 101.456\ (2)°$
$V = 2554.3\ (5)$ Å³

$Z = 2$
$D_x = 1.989$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 4862
reflections
$\theta = 2.2–24.6°$
$\mu = 8.92$ mm⁻¹
$T = 293\ (2)$ K
Block, black
$0.4 \times 0.3 \times 0.15$ mm

Data collection

Bruker SMART CCD area-detector
diffractometer
φ and ω scans
Absorption correction: multi-scan
(*SADABS*; Bruker, 2003)
$T_{min} = 0.050$, $T_{max} = 0.262$
13943 measured reflections

9809 independent reflections
7107 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 26.0°$
$h = -12 \rightarrow 11$
$k = -18 \rightarrow 8$
$l = -21 \rightarrow 23$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.176$
$S = 1.03$
9809 reflections
461 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1057P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.010$
$\Delta\rho_{max} = 2.18$ e Å⁻³
$\Delta\rho_{min} = -1.04$ e Å⁻³

Table 1
Selected geometric parameters (Å, °).

Au1—P1	2.230 (3)	Au2—P4	2.311 (3)
Au1—Cl2	2.286 (3)	Au2—Au3	3.1738 (7)
Au1—Au2	3.0883 (7)	Au3—P3	2.243 (3)
Au2—P2	2.304 (3)	Au3—Cl1	2.285 (3)
P1—Au1—Cl2	173.19 (11)	Au1—Au2—Au3	66.846 (16)
P2—Au2—P4	166.55 (10)	P3—Au3—Cl1	173.15 (11)

All H atoms were treated as riding, with phenyl C—H distances of 0.93 Å and methylene C—H distances of 0.97 Å, and with $U_{iso}(H) = 1.2U_{eq}(C)$ for phenyl groups and $1.5U_{eq}(C)$ for methylene groups. All phenyl groups and the perchlorate anion are likely to be disordered, and a combination of constraints and restraints was used; separate disorder components could not be satisfactorily resolved. The largest difference-map peak is 0.86 Å from atom Au3 and the deepest hole is 0.61 Å from atom C27.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2001); software used to prepare material for publication: *SHELXTL*.
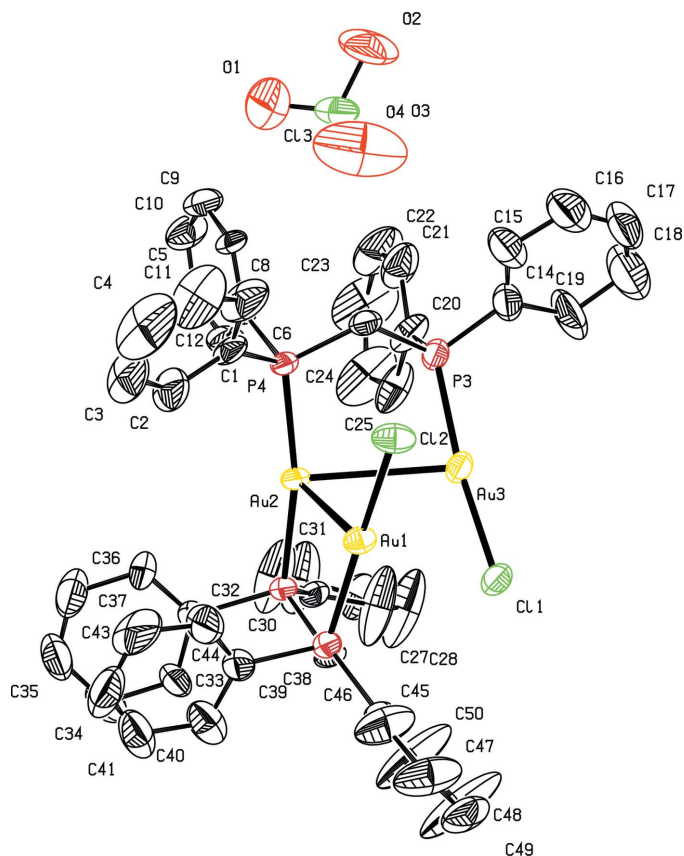

Figure 1
The structure of the asymmetric unit. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity.

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