

[μ -o-Phenylenebis(diphenylphosphine)- $\kappa^2 P:P'$]bis[chlorogold(I)], dppbz(AuCl)₂

Ahmed A. Mohamed,^a Jeanette A. Krause Bauer,^b Alice E. Bruce^{a*} and Mitchell R. M. Bruce^a

^aDepartment of Chemistry, University of Maine, Orono, ME 04469-5706, USA, and

^bDepartment of Chemistry, University of Cincinnati, Cincinnati, OH 45221-0172, USA

Correspondence e-mail: abruce@maine.edu

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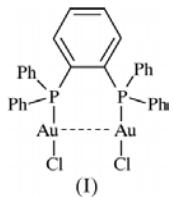
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The Au \cdots Au distance in the title compound, [Au₂Cl₂(C₃₀H₂₄P₂)], is 2.996 (1) Å, typical of an Au \cdots Au interaction. The two P–Au–Cl arms ‘cross’ at the Au centers, with a Cl–Au \cdots Au–Cl torsion angle of –63.92 (7)°. Only a small deviation from linearity is observed in the coordination around the Au atoms. Related phosphine–gold(I) chloride structures with intra- and intermolecular Au \cdots Au interactions are surveyed.

Comment

The chemistry of dinuclear gold(I)–phosphine complexes is quite extensive (Foley *et al.*, 1995; Brandys & Puddephatt, 2001; Khan *et al.*, 1989). Recently, we investigated photochemical isomerization in the dinuclear gold(I) complexes *cis*-Au₂X₂(dppee), where dppee is 1,2-bis(diphenylphosphino)ethylene and X is Cl, Br, I or SR, in which the Au atoms are constrained to be within bonding distance, *versus* *trans*-Au₂X₂(dppee), where an intramolecular approach is precluded (Foley *et al.*, 1995). We report here a dinuclear linear gold(I) complex, (I), of the potentially chelating ligand 1,2-bis(diphenylphosphino)benzene. In (I), the phenylene ring is rigid and hence *cis-trans* isomerization is precluded.



The crystal structure of (I) reveals that the two Au^I atoms have a linear coordination. The Au \cdots Au bond distance in compounds showing aurophilic interactions ranges from 2.8 Å (strong interaction) to >3.5 Å (weak interaction). The strength of this attraction has been determined experimentally to be *ca* 7–11 kcal mol^{−1} (1 kcal mol^{−1} = 4.184 kJ mol^{−1}) (Schmid-

baur, Graf & Muller, 1988; Narayanaswamy *et al.*, 1993). The Au \cdots Au distance of 2.996 (1) Å in (I) indicates a strong aurophilic attraction, similar to that found in [Au₂(S₂C₂B₁₀H₁₀)(dppbz)] [2.9771 (10) Å; Crespo *et al.*, 1997] and shorter than that in *cis*-(AuCl)₂dppee [3.05 (1) Å; Jones, 1980].

The Au–Cl bond lengths in (I) [2.293 (2) Å] are longer than the Au–P bonds [2.236 (2) Å] and fall within the expected range for similar phosphine–gold(I) complexes. Chemically equivalent bonds in the two P–Au–Cl arms are equivalent within experimental error. The two P–Au–Cl arms ‘cross’ at the Au centers, with a Cl–Au \cdots Au–Cl torsion angle of –63.92 (7)°. The hindered rotation about the PCCP linkage helps the two Au atoms to maintain the aurophilic interaction. The P–Au–Cl angle [173.21 (6)°] deviates only slightly from linearity. It is difficult to know whether this deviation is a result of Au \cdots Au bonding or crystal-packing forces. A survey of the Cambridge Structural Database (Version 5.23; Allen, 2002) revealed 20 phosphine–gold(I) chloride structures containing Au \cdots Au bonds and in these the P–Au–Cl angles average 174°, but there is no correlation between bond angle and Au \cdots Au bond distance. However, complexes without Au \cdots Au bonds have angles closer to 180°. For example, in Ph₃PAuCl and ⁱPr₃PAuCl, with no Au \cdots Au bond, the P–Au–Cl angles are 179.68° (Baenziger *et al.*, 1976) and 178.03° (Angermaier *et al.*, 1994), respectively.

Phosphine–gold(I) chloride complexes containing Au \cdots Au bonds can be grouped into two broad categories, *viz.* those with intramolecular Au \cdots Au interactions, as in (I), and those with intermolecular Au \cdots Au interactions. Complexes with intramolecular Au \cdots Au interactions have bis- or tris(phosphine) ligands that are linked and have restricted rotation about the phosphine-ligand backbone. Rotation is restricted by virtue of a rigid backbone, for example, in dppbz or *cis*-dppee, or by a small bite angle between P atoms that imposes steric restraints, as in Ph₂PNHPPPh₂ or Ph₂PCF₂PPPh₂. The intramolecular Au \cdots Au distances in these complexes range

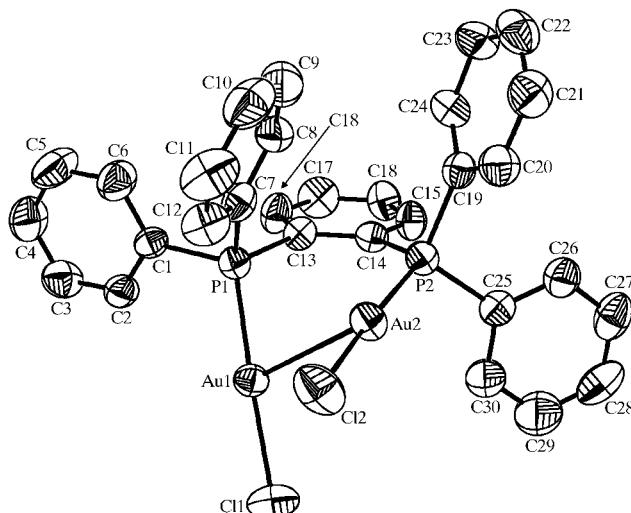


Figure 1

A view of the molecular structure of (I) with the atomic numbering scheme. Displacement ellipsoids are shown at the 50% probability level and H atoms have been omitted for clarity.

from 2.925 to 3.281 Å and the absolute values of the Cl—Au···Au—Cl torsion angles range from 30.51 to 76.41° (Schmidbaur, Graf & Muller, 1988; Schmidbaur, Reber *et al.*, 1988; Zank *et al.*, 1998; Lange *et al.*, 1997; Schmidbaur *et al.*, 1989; Jones & Bembenek, 1996; Bardaji *et al.*, 2000; Cerrada *et al.*, 2002; Stutzer *et al.*, 1992; Jones, 1980). Additional intermolecular Au···Au interactions may also be present.

Complexes in the second category (*i.e.* with intermolecular Au···Au interactions) are either mononuclear phosphine-gold(I) chloride complexes, or the phosphines are linked by conformationally flexible backbones, as in $\text{Ph}_2\text{P}(\text{CH}_2)_n\text{PPH}_2$, where $n = 2, 3, 5, 7$ and 8. The mononuclear complexes in this category form dimers or trimers *via* intermolecular Au···Au interactions if the substituents on P are not too bulky, *e.g.* $\text{Ph}(\text{CH}_3)_2\text{PAuCl}$ (Au···Au = 3.092 Å; Toronto *et al.*, 1996). Bisphosphine-gold(I) chloride complexes with flexible backbones tend to crystallize with the alkane backbone fully extended, and intermolecular Au···Au interactions link up the dinuclear gold complexes to form extended chains. It is interesting to note that, for complexes with intermolecular Au···Au interactions, the absolute value of the Cl—Au···Au—Cl torsion angles are larger, ranging from 94.2 to 135°, while the Au···Au interactions are also slightly longer on average, ranging from 3.061 to 3.233 Å (Hollatz *et al.*, 1999; Attar *et al.*, 1990; Balch & Fung, 1990; Van Calcar *et al.*, 1997; Banger *et al.*, 1999; Toronto *et al.*, 1996; Tzeng *et al.*, 1996; Schmidbaur *et al.*, 1992). The exceptions are for $n = 2$, which forms a dimer *via* intermolecular Au···Au bonds (3.189 Å), with Cl—Au···Au—Cl torsion angles of −74.87 and 82.44° (Bates & Waters, 1985), and $n = 4$ (Schmidbaur *et al.*, 1992) and $n = 6$ (Van Calcar *et al.*, 1995), in which no Au···Au interactions were found.

Experimental

1,2-Bis(diphenylphosphino)benzene was added to gold(I) thiodiethanol chloride, prepared *in situ*, in methanol (1:2 stoichiometric ratio), and a white precipitate formed immediately. The mixture was stirred for 1 h, filtered and washed with methanol. Colorless crystals of (I) were obtained by slow diffusion of diethyl ether into a dichloromethane solution of the title compound.

Crystal data

$[\text{Au}_2\text{Cl}_2(\text{C}_{30}\text{H}_{24}\text{P}_2)]$	Mo $K\alpha$ radiation
$M_r = 911.27$	Cell parameters from 5866 reflections
Orthorhombic, $Pbca$	$\theta = 2.4\text{--}28.3^\circ$
$a = 16.9558 (3)$ Å	$\mu = 8.83 \text{ mm}^{-1}$
$b = 18.1603 (2)$ Å	$T = 293 (2)$ K
$c = 22.22550 (10)$ Å	Needle, colorless
$V = 6843.73 (15)$ Å ³	$0.45 \times 0.12 \times 0.08$ mm
$Z = 8$	
$D_x = 1.769 \text{ Mg m}^{-3}$	

Table 1

Selected geometric parameters (Å, °).

Au1—P1	2.2408 (15)	Au2—P2	2.2360 (16)
Au1—Cl1	2.2921 (16)	Au2—Cl2	2.2925 (16)
Au1—Au2	2.9960 (3)		
P1—Au1—Cl1	173.07 (6)	P2—Au2—Cl2	173.21 (6)

Data collection

Bruker SMART 1K Platform CCD area-detector diffractometer	8478 independent reflections
ω scans	5769 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.061$
$T_{\text{min}} = 0.100$, $T_{\text{max}} = 0.493$	$\theta_{\text{max}} = 28.3^\circ$
45 724 measured reflections	$h = -18 \rightarrow 22$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.043$	$w = 1/[\sigma^2(F_o^2) + (0.0299P)^2]$
$wR(F^2) = 0.076$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{\text{max}} = 0.001$
8478 reflections	$\Delta\rho_{\text{max}} = 0.57 \text{ e } \text{\AA}^{-3}$
325 parameters	$\Delta\rho_{\text{min}} = -0.58 \text{ e } \text{\AA}^{-3}$

H-atom positions were calculated based on geometric criteria and H atoms were treated with a riding model (C—H = 0.93 Å), with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. Compound (I) crystallized in S-shaped layers with what appeared to be badly disordered Et_2O molecules. The solvent resided in the channels created by the layers. The crystallographic refinement was completed with the solvent contribution subtracted from the data using *SQUEEZE* (*PLATON*92; Spek, 1992).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Version 5.10; Siemens, 1996); program(s) used to refine structure: *SHELXTL* (Version 5.10); molecular graphics: *SHELXTL* (Version 5.03; Siemens, 1996); software used to prepare material for publication: *SHELXTL* (Version 5.03).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1679). Services for accessing these data are described at the back of the journal.

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metal-organic compounds

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