

Peter C. Healy

School of Science, Griffith University, Nathan,
Brisbane 4111, Australia

Correspondence e-mail: p.healy@griffith.edu.au

Key indicators

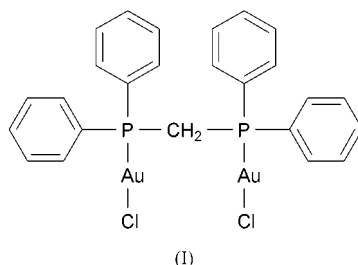
Single-crystal X-ray study
 $T = 295\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.020\text{ \AA}$
 R factor = 0.034
 wR factor = 0.096
Data-to-parameter ratio = 16.1For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.A new polymorph of μ -bis(diphenylphos-
phino)methane- $\kappa^2 P:P'$ -bis[chlorogold(I)]

The title compound, $[(\text{AuCl})_2(\text{dppm})]$ ($\text{dppm} = \text{Ph}_2\text{P}(\text{CH}_2)\text{PPh}_2$), has been found to crystallize as a new triclinic polymorph. Unlike the previously reported monoclinic form, there are no aurophilic $\text{Au}\cdots\text{Au}$ interactions between the Au(I) atoms.

Received 13 October 2003
Accepted 3 November 2003
Online 8 November 2003

Comment

The structure of the title compound, (I), was first determined by Schmidbaur *et al.* (1977) [monoclinic, space group $C2/c$, $a = 22.31(1)\text{ \AA}$, $b = 7.215(7)\text{ \AA}$, $c = 18.12(1)\text{ \AA}$ and $\beta = 120.43(8)^\circ$]. This structure, (Ia), consists of discrete molecules of $[(\text{AuCl})_2(\text{dppm})]$ disposed about the crystallographic twofold axis with a conformational structure in which the $\text{Au}-\text{P}\cdots\text{P}-\text{Au}$ torsion angle is $67(1)^\circ$, yielding an intramolecular $\text{Au}\cdots\text{Au}$ distance of $3.351(2)\text{ \AA}$. This distance has subsequently been classified as an intramolecular $\text{Au}^I\cdots\text{Au}^I$ aurophilic interaction, facilitated by the small bite of the dppm ligand (*e.g.* Van Calcar *et al.*, 1997).



The complex also crystallizes in the triclinic space group $P\bar{1}$, as discrete molecules with one complete molecule constituting the asymmetric unit [structure (Ib); Figs. 1 and 2]. The most significant difference between the structures of the two polymorphs is that, in (Ib), there are no $\text{Au}^I\cdots\text{Au}^I$ aurophilic interactions. In (Ib), the $\text{Au}-\text{P}\cdots\text{P}-\text{Au}$ torsion angle is $124.3(1)^\circ$, yielding an $\text{Au}1\cdots\text{Au}2$ distance of $5.617(3)\text{ \AA}$, while all intermolecular $\text{Au}\cdots\text{Au}$ distances are greater than 4 \AA . The Au atoms are located above the plane of a phenyl ring on the opposite P atom, suggesting that $\text{Au}\cdots\pi$ interactions may be an important factor in the determination of the conformational structure of this polymorph.

There are no unusual features of the gold coordination geometry of (Ib), with $\text{Au}-\text{P} = 2.233(3)$ and $2.228(2)\text{ \AA}$, $\text{Au}-\text{Cl} = 2.276(3)$ and $2.278(3)\text{ \AA}$, and $\text{P}-\text{Au}-\text{Cl} = 177.4(1)$ and $175.5(1)^\circ$ [*cf.* (Ia), $\text{Au}-\text{P} = 2.238(1)\text{ \AA}$, $\text{Au}-\text{Cl} = 2.288(1)\text{ \AA}$ and $\text{P}-\text{Au}-\text{Cl} = 175.2(2)^\circ$]. The absence of aurophilic interactions in (Ib), however, is consistent with the observed increase in the $\text{P}-\text{C}-\text{P}$ ligand bite angle from $116(1)^\circ$ in (Ia) to $119.7(5)^\circ$ in (Ib).

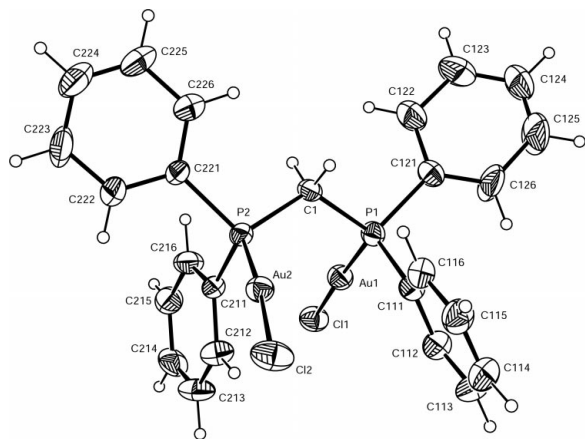


Figure 1
View of (Ib), with the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level.

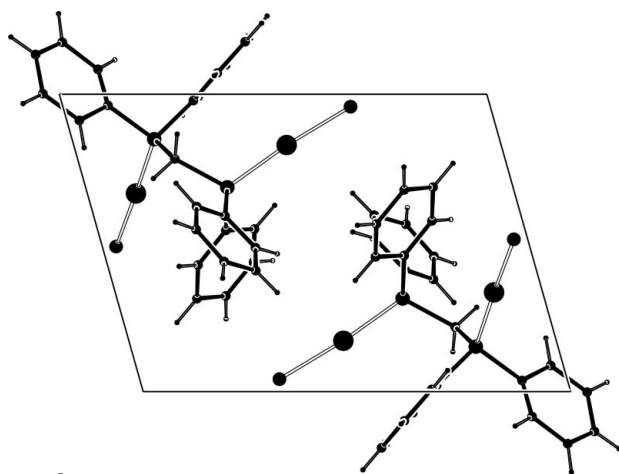


Figure 2
Crystal packing diagram for (Ib), viewed down the *a* axis, with *b* horizontal and *c* vertical.

Experimental

(NBu₄)[AuCl₄] (29 mg, 0.049 mmol) and bis(diphenylphosphino)methane (19 mg, 0.049 mmol) were suspended in a solution of concentrated HCl (0.2 ml) in dimethylformamide (5 ml). The mixture was gently warmed to give a clear pale-yellow solution, which quickly faded to give a colorless solution. Cooling this solution to room temperature followed by slow evaporation of the solvent over a period of several days yielded well formed colorless needle-like crystals of (Ib); m.p. 538–541 K. Analysis found: C 35.80, H 2.65%; calculated for C₂₅H₂₂Au₂Cl₂P₂: C 35.36, H 2.61%.

Crystal data

[Au₂Cl₂(C₂₅H₂₂P₂)]
M_r = 849.21
 Triclinic, *P*1
a = 10.912 (5) Å
b = 13.116 (5) Å
c = 9.856 (4) Å
 α = 102.81 (3)°
 β = 107.19 (3)°
 γ = 96.88 (4)°
V = 1287.8 (10) Å³

Z = 2
D_x = 2.190 Mg m⁻³
 Mo K α radiation
 Cell parameters from 25 reflections
 θ = 12.6–16.1°
 μ = 11.72 mm⁻¹
T = 295 K
 Needle, colorless
 0.40 × 0.25 × 0.20 mm

Data collection

Rigaku AFC-7R diffractometer
 ω -2 θ scans
 Absorption correction: ψ scan
 (North *et al.*, 1968)
 T_{\min} = 0.041, T_{\max} = 0.096
 5139 measured reflections
 4519 independent reflections
 3666 reflections with $I > 2\sigma(I)$

R_{int} = 0.017
 θ_{max} = 25.0°
 h = -12 → 12
 k = -15 → 15
 l = -5 → 11
 3 standard reflections
 every 150 reflections
 intensity decay: 1.2%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)]$ = 0.034
 $wR(F^2)$ = 0.096
 S = 1.06
 4519 reflections
 281 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0408P)^2 + 7.2469P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}}$ = 0.001
 $\Delta\rho_{\text{max}}$ = 1.31 e Å⁻³
 $\Delta\rho_{\text{min}}$ = -1.33 e Å⁻³
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.00070 (19)

Table 1

Selected geometric parameters (Å, °).

Au1—Cl1	2.276 (3)	P1—C111	1.803 (11)
Au1—P1	2.233 (3)	P1—C121	1.812 (10)
Au2—Cl2	2.278 (3)	P2—C1	1.821 (9)
Au2—P2	2.228 (2)	P2—C211	1.813 (9)
P1—C1	1.820 (9)	P2—C221	1.808 (9)
Cl1—Au1—P1	177.44 (11)	C1—P2—C221	103.4 (4)
Cl2—Au2—P2	175.48 (10)	C211—P2—C221	104.5 (4)
Au1—P1—C1	112.9 (3)	P1—C1—P2	119.7 (5)
Au1—P1—C111	114.7 (3)	P1—C111—C112	119.6 (8)
Au1—P1—C121	113.4 (3)	P1—C111—C116	121.6 (8)
C1—P1—C111	108.1 (5)	P1—C121—C126	123.3 (14)
C1—P1—C121	102.5 (5)	P1—C121—C122	119.6 (9)
C111—P1—C121	104.1 (5)	P2—C211—C212	119.8 (7)
Au2—P2—C1	115.7 (3)	P2—C211—C216	120.6 (8)
Au2—P2—C211	112.5 (3)	P2—C221—C222	119.1 (9)
Au2—P2—C221	112.6 (3)	P2—C221—C226	124.0 (8)
C1—P2—C211	107.1 (4)		

H atoms were constrained in the riding model approximation, fixed to their parent C atoms at a C—H distance of 0.95 Å, and $U_{\text{iso}}(\text{H})$ values were set to 1.2 U_{eq} of the parent atom. The maximum and minimum residual electron densities were located within 1 Å of atoms Au1 and Au2. The magnitude and anisotropy of the displacement ellipsoids of phenyl rings 12*n* and 22*n* ($n = 1-6$) are consistent with a measure of disorder in these rings.

Data collection: *MSC/AFC-7 Diffractometer Control Software for Windows* (Molecular Structure Corporation, 1999); cell refinement: *MSC/AFC-7 Diffractometer Control Software for Windows*; data reduction: *TEXSAN for Windows* (Molecular Structure Corporation, 1997–2001); program(s) used to solve structure: *TEXSAN for Windows*; program(s) used to refine structure: *TEXSAN for Windows* and *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *TEXSAN for Windows* and *PLATON* (Spek, 1980–2001).

The author thanks Griffith University for financial assistance.

References

- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
 Molecular Structure Corporation (1999). *MSC/AFC-7 Diffractometer Control Software for Windows*. Version 1.02. MSC, 9009 New Trails Drive, The Woodlands, TX 77381, USA.

Molecular Structure Corporation (1997–2001). *TEXSAN for Windows*. Version 1.06. MSC, 9009 New Trails Drive, The Woodlands, TX 77381, USA.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
Schmidbaur, H., Wohlleben, A., Wagner, F., Orama, O. & Huttner, G. (1977). *Chem. Ber.* **110**, 1748–1754.

Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (1980–2001). *PLATON for Windows*. Version 121201. University of Utrecht, The Netherlands.
Van Calcar, P. M., Olmstead, M. M. & Balch, A. L. (1997). *Inorg. Chem.* **36**, 5231–5238.