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Tetraphenylphosphonium dibromidoaurate(I)

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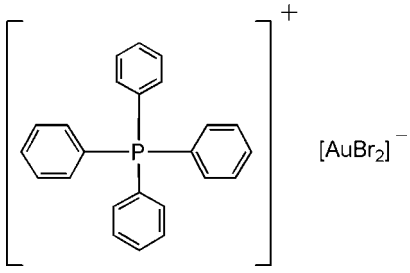
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.009 Å; R factor = 0.030; wR factor = 0.089; data-to-parameter ratio = 15.7.

In the structure of the title complex, $[(C_6H_5)_4P][AuBr_2]$, the $[Ph_4P]^+$ cations and the $[AuBr_2]^-$ anions are located on crystallographic twofold axes with no unusual contacts. The Au—Br distance of the virtually linear anion is 2.3370 (19) Å.

Related literature

The title compound was synthesized by adapted literature procedures (Braunstein & Clark, 1973; Buckley *et al.*, 1997). The Au—Br distance is similar to that of the $[n\text{-}Bu_4N]^+$ salt (Braunstein *et al.*, 1986).



Experimental

Crystal data

$(C_{24}H_{20}P)[AuBr_2]$
M_r = 696.14
Monoclinic, $P2/n$
a = 10.372 (4) Å
b = 7.602 (3) Å
c = 14.559 (9) Å
β = 92.05 (5)°
V = 1147.2 (10) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 9.97 mm^{-1}
T = 295 (2) K
0.25 × 0.20 × 0.15 mm

Data collection

Rigaku AFC-7*R* diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.189, T_{max} = 0.316
(expected range = 0.134–0.224)
2146 measured reflections
2029 independent reflections
1141 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
3 standard reflections
every 150 reflections
intensity decay: 2.7%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.089
S = 0.99
2029 reflections
129 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

Data collection: *MSC/AFC7 Diffractometer Control Software* (Molecular Structure Corporation, 1999); cell refinement: *MSC/AFC7 Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 2001); program(s) used to solve structure: *TEXSAN*; program(s) used to refine structure: *TEXSAN* and *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *TEXSAN* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2140).

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supplementary materials

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Tetraphenylphosphonium dibromidoaurate(I)

C. L. Noack, R. W. Buckley and P. C. Healy

Comment

As part of our studies on the synthesis, structural and spectroscopic characterization of gold(I) complexes, we synthesized the title complex, (I), by adaption of literature procedures (Buckley *et al.*, 1997; Braunstein & Clark, 1973).

The molecular structure of (I) is presented in Fig. 1. The $[Ph_4P]^+$ cations and the $[AuBr_2]^-$ anions are located on crystallographic twofold axes with no unusual contacts. The Au—Br distance of 2.3370 (19) Å is in good agreement with that of 2.376 (3) Å observed for the analogous $[n\text{-}Bu_4N]^+$ salt (Braunstein *et al.*, 1986). The Br—Au—Br angle is nearly linear (178.02 (7)°).

Experimental

Liquid bromine (3.0 ml) was added dropwise to a suspension of powdered metallic gold (2.0 g, 10.20 mmol) in a solution of tetraphenylphosphonium bromide (94.71 g, 10.20 mmol) in absolute ethanol (50 ml). The mixture was then stirred vigorously at room temperature for 2 h. More bromine (1.0 ml) was added and the resultant deep red solution stirred overnight at room temperature. The solution was then heated slowly to boiling to remove any excess bromine. Cooling to room temperature and filtration yielded the gold(III) complex $[PPh_4][AuBr_4]$ as a deep red solid (yield 8.01 g, 82%). $[PPh_4][AuBr_4]$ (2.0 g, 2.34 mmol) was then dissolved in warm (~320 K) absolute ethanol, followed by addition of acetone (10 ml) in 1 ml increments until the solution turned colourless. Cooling the solution to room temperature resulted in precipitation of colourless crystals of the title complex suitable for X-ray diffraction studies. Yield 1.38 g, 85%. m.p. 511–515 K. Analysis found: C 41.2, H 2.8%; calculated for $C_{24}H_{20}PAuBr_2$ C 41.4, H 2.9%.

Refinement

H atoms were constrained as riding atoms, fixed to their parent C atoms at a C—H distance of 0.95 Å. U_{iso}(H) values were set to $1.2U_{eq}$ of the parent atom.

Figures

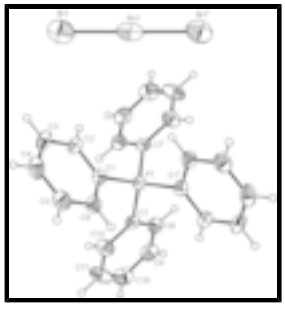

Fig. 1. View of the title compound with the atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level [Symmetry operation i): $-x + 3/2$, y, $-z + 1/2$.]

Tetraphenylphosphonium dibromidoaurate(I)

Crystal data

$(C_{24}H_{20}P)[AuBr_2]$	F_{000} = 656
M_r = 696.14	D_x = 2.015 Mg m^{-3}
Monoclinic, $P2/n$	Mo $K\alpha$ radiation λ = 0.7107 Å
Hall symbol: -P 2yac	Cell parameters from 17 reflections
a = 10.372 (4) Å	θ = 19.5–20.0º
b = 7.602 (3) Å	μ = 9.97 mm^{-1}
c = 14.559 (9) Å	T = 295 (2) K
β = 92.05 (5)º	Block, colourless
V = 1147.2 (10) Å^3	0.25 × 0.20 × 0.15 mm
Z = 2	

Data collection

Rigaku AFC-7R diffractometer	R_{int} = 0.031
Radiation source: Rigaku rotating anode	θ_{max} = 25.1º
Monochromator: graphite	θ_{min} = 2.4º
T = 293(5) K	h = 0→12
$\omega/2\theta$ scans	k = 0→9
Absorption correction: ψ scan (North *et al.*, 1968)	l = −17→17
T_{min} = 0.189, T_{max} = 0.316	3 standard reflections
2146 measured reflections	every 150 reflections
2029 independent reflections	intensity decay: 2.7%
1141 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.030	$w = 1/[\sigma^2(F_o^2) + (0.0398P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.089	$(\Delta/\sigma)_{max} < 0.001$
S = 0.99	$\Delta\rho_{max}$ = 0.53 e Å^{-3}
2029 reflections	$\Delta\rho_{min}$ = −0.48 e Å^{-3}
129 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001Fc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0051 (4)
Secondary atom site location: difference Fourier map	

Special details

Experimental. The scan width was (1.68 + 0.35tanθ)° with an ω scan speed of 32° per minute (up to 4 scans to achieve I/σ(I) > 15). Stationary background counts were recorded at each end of the scan, and the scan time:background time ratio was 2:1.

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The observed criterion of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating –*R*-factor-obs *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and *R*-factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
P1	0.25000	0.9998 (3)	0.25000	0.0381 (7)
C1	0.2830 (5)	0.8562 (6)	0.3460 (4)	0.0394 (19)
C2	0.3963 (5)	0.8672 (7)	0.3983 (4)	0.0486 (19)
C3	0.4191 (6)	0.7473 (10)	0.4688 (5)	0.063 (3)
C4	0.3294 (8)	0.6203 (9)	0.4871 (5)	0.067 (3)
C5	0.2177 (7)	0.6088 (9)	0.4346 (5)	0.062 (3)
C6	0.1932 (6)	0.7254 (8)	0.3640 (4)	0.051 (2)
C7	0.1167 (5)	1.1411 (6)	0.2700 (4)	0.0391 (19)
C8	0.0934 (6)	1.2824 (8)	0.2107 (4)	0.056 (2)
C9	−0.0045 (7)	1.3976 (8)	0.2281 (5)	0.067 (3)
C10	−0.0828 (7)	1.3741 (9)	0.3009 (6)	0.076 (3)
C11	−0.0622 (7)	1.2337 (11)	0.3585 (6)	0.083 (3)
C12	0.0379 (6)	1.1178 (8)	0.3439 (5)	0.061 (2)
Au1	0.75000	0.82371 (5)	0.25000	0.0857 (2)
Br1	0.77409 (10)	0.81831 (11)	0.41295 (8)	0.1106 (5)
H2	0.45730	0.95610	0.38610	0.0600*
H3	0.49720	0.75270	0.50490	0.0750*
H4	0.34470	0.54050	0.53660	0.0800*
H5	0.15740	0.51900	0.44700	0.0730*
H6	0.11560	0.71680	0.32790	0.0610*
H8	0.14540	1.29870	0.15690	0.0680*
H9	−0.01900	1.49660	0.18950	0.0820*
H10	0.05220	1.02040	0.38350	0.0730*
H11	−0.11650	1.21470	0.41130	0.0970*
H12	−0.15260	1.45590	0.31200	0.0890*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0383 (10)	0.0387 (11)	0.0374 (13)	0.0000	0.0020 (9)	0.0000

C1	0.049 (3)	0.031 (3)	0.038 (4)	0.006 (2)	0.000 (3)	0.000 (2)
C2	0.051 (3)	0.046 (3)	0.048 (4)	0.002 (3)	−0.009 (3)	−0.001 (3)
C3	0.072 (5)	0.067 (4)	0.049 (5)	0.011 (4)	−0.020 (4)	0.014 (4)
C4	0.101 (6)	0.053 (4)	0.046 (4)	0.023 (4)	0.006 (4)	0.009 (3)
C5	0.072 (5)	0.054 (4)	0.061 (5)	−0.002 (3)	0.010 (4)	0.013 (4)
C6	0.054 (4)	0.051 (4)	0.047 (4)	−0.004 (3)	0.000 (3)	0.007 (3)
C7	0.038 (3)	0.034 (3)	0.045 (4)	0.000 (2)	−0.003 (3)	−0.002 (3)
C8	0.054 (4)	0.058 (4)	0.055 (4)	0.005 (3)	−0.001 (3)	0.008 (3)
C9	0.070 (5)	0.048 (3)	0.081 (6)	0.011 (3)	−0.016 (4)	0.001 (4)
C10	0.065 (5)	0.059 (4)	0.105 (7)	0.014 (3)	0.001 (5)	−0.023 (4)
C11	0.067 (5)	0.069 (5)	0.117 (7)	0.013 (4)	0.042 (5)	−0.010 (5)
C12	0.063 (4)	0.048 (3)	0.072 (5)	0.002 (3)	0.017 (4)	0.008 (3)
Au1	0.0670 (3)	0.0462 (2)	0.1450 (6)	0.0000	0.0207 (3)	0.0000
Br1	0.1107 (7)	0.0801 (6)	0.1423 (10)	−0.0118 (5)	0.0247 (6)	0.0203 (6)

Geometric parameters (Å, °)

Au1—Br1	2.3770 (19)	C8—C9	1.371 (9)
Au1—Br1^{i}	2.3770 (19)	C9—C10	1.370 (11)
P1—C1	1.797 (6)	C10—C11	1.369 (11)
P1—C7^{ii}	1.783 (5)	C11—C12	1.384 (10)
P1—C7	1.783 (5)	C2—H2	0.9500
P1—C1^{ii}	1.797 (6)	C3—H3	0.9500
C1—C6	1.394 (8)	C4—H4	0.9500
C1—C2	1.380 (8)	C5—H5	0.9500
C2—C3	1.387 (9)	C6—H6	0.9500
C3—C4	1.374 (10)	C8—H8	0.9700
C4—C5	1.368 (11)	C9—H9	0.9500
C5—C6	1.374 (9)	C10—H12	0.9700
C7—C8	1.394 (8)	C11—H11	0.9800
C7—C12	1.386 (9)	C12—H10	0.9500
Au1···C10^{iii}	3.892 (7)	C12···Au1^{ii}	3.936 (7)
Au1···C11^{iv}	3.973 (9)	C12···C6	3.398 (9)
Au1···C12^{iv}	3.936 (7)	C1···H6^{ii}	2.9700
Au1···C10^{v}	3.892 (7)	C1···H10	2.7700
Au1···C11^{ii}	3.973 (9)	C4···H11^{x}	2.9800
Au1···C9^{v}	4.140 (7)	C5···H11^{x}	2.8500
Au1···C12^{ii}	3.936 (7)	C5···H8^{v}	3.0800
Au1···C9^{iii}	4.140 (7)	C6···H10	2.7000
Au1···H9^{iii}	3.5800	C7···H2^{ii}	2.7600
Au1···H12^{iii}	3.1000	C7···H8^{ii}	2.9100
Au1···H9^{v}	3.5800	C8···H2^{ii}	2.8900
Au1···H12^{v}	3.1000	C9···H6^{xi}	3.0700
Br1···C12^{iv}	3.725 (7)	H2···C8^{ii}	2.8900
Br1···C11^{iv}	3.685 (9)	H2···C7^{ii}	2.7600

Br1···H11^{iv}	3.2200	H4···Br1^{vi}	3.0900
Br1···H4^{vi}	3.0900	H6···C1^{ii}	2.9700
Br1···H12^{iii}	3.2300	H6···H10	2.5400
C2···C8^{ii}	3.536 (8)	H6···C9^{xii}	3.0700
C6···C12	3.398 (9)	H8···C7^{ii}	2.9100
C6···C6^{ii}	3.562 (9)	H8···C5^{vii}	3.0800
C8···C8^{ii}	3.405 (9)	H9···Au1^{viii}	3.5800
C8···C2^{ii}	3.536 (8)	H9···Au1^{vii}	3.5800
C9···Au1^{vii}	4.140 (7)	H10···C6	2.7000
C9···Au1^{viii}	4.140 (7)	H10···C1	2.7700
C10···Au1^{viii}	3.892 (7)	H10···H6	2.5400
C10···Au1^{vii}	3.892 (7)	H11···C4^{x}	2.9800
C11···Au1^{ix}	3.973 (9)	H11···C5^{x}	2.8500
C11···Br1^{ix}	3.685 (9)	H11···Br1^{ix}	3.2200
C11···Au1^{ii}	3.973 (9)	H12···Au1^{vii}	3.1000
C12···Au1^{ix}	3.936 (7)	H12···Au1^{viii}	3.1000
C12···Br1^{ix}	3.725 (7)	H12···Br1^{viii}	3.2300
Br1—Au1—Br1^{i}	178.02 (7)	C7—C12—C11	120.3 (6)
C1—P1—C7	111.5 (3)	C1—C2—H2	120.00
C1—P1—C7^{ii}	111.5 (2)	C3—C2—H2	121.00
C1^{ii}—P1—C7	111.5 (2)	C4—C3—H3	120.00
C1—P1—C1^{ii}	105.2 (3)	C2—C3—H3	120.00
C1^{ii}—P1—C7^{ii}	111.5 (3)	C3—C4—H4	120.00
C7—P1—C7^{ii}	105.9 (3)	C5—C4—H4	120.00
P1—C1—C2	121.8 (4)	C6—C5—H5	120.00
P1—C1—C6	118.0 (4)	C4—C5—H5	119.00
C2—C1—C6	120.1 (5)	C1—C6—H6	120.00
C1—C2—C3	119.2 (5)	C5—C6—H6	120.00
C2—C3—C4	120.5 (6)	C9—C8—H8	120.00
C3—C4—C5	120.2 (7)	C7—C8—H8	120.00
C4—C5—C6	120.5 (6)	C8—C9—H9	120.00
C1—C6—C5	119.6 (6)	C10—C9—H9	118.00
P1—C7—C12	122.2 (4)	C11—C10—H12	120.00
C8—C7—C12	119.0 (5)	C9—C10—H12	121.00
P1—C7—C8	118.8 (4)	C10—C11—H11	121.00
C7—C8—C9	119.4 (6)	C12—C11—H11	119.00
C8—C9—C10	121.5 (6)	C7—C12—H10	119.00
C9—C10—C11	119.5 (7)	C11—C12—H10	120.00
C10—C11—C12	120.2 (7)		
C7—P1—C1—C2	−114.7 (5)	P1—C1—C6—C5	177.4 (5)
C7—P1—C1—C6	68.6 (5)	C2—C1—C6—C5	0.6 (9)
C1^{ii}—P1—C1—C2	124.4 (5)	C1—C2—C3—C4	−0.7 (10)
C1^{ii}—P1—C1—C6	−52.4 (5)	C2—C3—C4—C5	1.3 (11)

C7^{ii}—P1—C1—C2	3.4 (5)	C3—C4—C5—C6	−0.9 (11)
C7^{ii}—P1—C1—C6	−173.3 (4)	C4—C5—C6—C1	0.0 (10)
C1—P1—C7—C8	168.0 (4)	P1—C7—C8—C9	−176.5 (5)
C1—P1—C7—C12	−10.2 (6)	C12—C7—C8—C9	1.7 (9)
C1^{ii}—P1—C7—C8	−74.8 (5)	P1—C7—C12—C11	178.0 (5)
C1^{ii}—P1—C7—C12	107.0 (5)	C8—C7—C12—C11	−0.2 (9)
C7^{ii}—P1—C7—C8	46.6 (5)	C7—C8—C9—C10	−2.1 (10)
C7^{ii}—P1—C7—C12	−131.6 (5)	C8—C9—C10—C11	0.8 (11)
P1—C1—C2—C3	−176.9 (5)	C9—C10—C11—C12	0.8 (12)
C6—C1—C2—C3	−0.2 (9)	C10—C11—C12—C7	−1.1 (11)

Symmetry codes: (i) −*x*+3/2, *y*, −*z*+1/2; (ii) −*x*+1/2, *y*, −*z*+1/2; (iii) *x*+1, *y*−1, *z*; (iv) *x*+1, *y*, *z*; (v) −*x*+1/2, *y*−1, −*z*+1/2; (vi) −*x*+1, −*y*+1, −*z*+1; (vii) −*x*+1/2, *y*+1, −*z*+1/2; (viii) *x*−1, *y*+1, *z*; (ix) *x*−1, *y*, *z*; (x) −*x*, −*y*+2, −*z*+1; (xi) *x*, *y*+1, *z*; (xii) *x*, *y*−1, *z*.

Fig. 1