SHORT-FORMAT PAPERS

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Chloro[tris(*o*-tolyl)phosphine]gold(I)

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Abstract. [Au{$P(C_7H_7)_3$}Cl], $M_r = 536{\cdot}8$, monoclinic, $C2/c$, $a = 24{\cdot}114$ (2), $b = 12{\cdot}305$ (1), $c = 15{\cdot}834$ (1) Å, $\beta = 125{\cdot}07$ (1)°, $V = 3845$ (1) Å^3, $D_x = 1{\cdot}855$ Mg m^{-3} for $Z = 8$, Mo $K\bar{\alpha}$ radiation, $\lambda = 0{\cdot}7107$ Å, $\mu = 7{\cdot}820$ mm^{-1}, $F(000) = 2064$, $T = 293$ (2) K, $R = 0{\cdot}036$ for 2106 observed reflections. The Au atom exists in a linear geometry defined by a Cl atom, Au—Cl 2·281 (3) Å, and a P atom, Au—P 2·243 (2) Å, such that Cl—Au—P is 179·4 (1)°. The dihedral angles formed between the three phosphine-bound *o*-tolyl groups are 81·5, 81·1 and 83·3°.

Experimental. The title compound was prepared from the reaction of $HAuCl_4$ and tris(*o*-tolyl)phosphine (Eastman) using the literature method (Al-Saády, McAuliffe, Parish & Sandbank, 1985). Crystals were obtained from the slow evaporation of an ethanol/$CHCl_3$ solution; m.p. 556–558 K. Enraf–Nonius CAD-4F diffractometer controlled by a PDP8/A computer, graphite-monochromated Mo $K\bar{\alpha}$ radiation; $\omega/2\theta$ scan technique. Cell parameters on crystal 0·42 × 0·15 × 0·12 mm by least squares on 25 reflections ($10 \le \theta \le 13°$) (Boer & Duisenberg, 1984). Analytical absorption correction applied; max. and min. transmission factors 0·376 and 0·159 (Sheldrick, 1976). Total of 3982 reflections ($1{\cdot}5 \le \theta \le 25{\cdot}0°$) measured in the range $-28 \le h \le 28$, $0 \le k \le 14$, $-18 \le l \le 1$. No significant variation in the net intensities of two reference reflections ($\bar{1}1,\bar{3},3$, $2\bar{6}2$) measured every 7200 s. 3392 unique reflections (R_{int} 0·043) and 2106 satisfied $I \ge 2{\cdot}5\sigma(I)$. Structure solved by Patterson method, full-matrix least-squares refinement of 218 parameters based on F (Sheldrick, 1976). Anisotropic thermal parameters for non-H atoms and H atoms included in their calculated positions (C—H 0·97 Å). At convergence $R = 0{\cdot}036$, $wR = 0{\cdot}037$, $w = 1{\cdot}78/[\sigma^2(F) + 0{\cdot}0009F^2]$, $S = 1{\cdot}64$, $(\Delta/\sigma)_{max} \le 0{\cdot}001$, $(\Delta\rho)_{max} = 1{\cdot}43$, $(\Delta\rho)_{min} = -1{\cdot}64$ e Å^{-3}; no extinction correction. Scattering factors for H, C, Cl and P given in *SHELX*76 (Sheldrick, 1976) and those for neutral Au corrected for f' and f'' from *International Tables for X-ray Crystallography* (1974). All calculations on SUN4/280 computer system. Atomic parameters are given in Table 1, selected interatomic parameters in Table 2† and the numbering scheme used is shown in

† Lists of structure factors, thermal parameters, H-atom parameters, mean-plane data and all bond distances and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52829 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional atomic coordinates and* B_{eq} *values* (Å^2)

$B_{eq} = 8\pi^2/3(U_{11} + U_{22} + U_{33})$.

	x	y	z	B_{eq}
Au	0·40854 (2)	0·24209 (2)	0·43744 (3)	2·97
Cl	0·4729 (1)	0·2401 (2)	0·6134 (2)	4·07
P	0·3460 (1)	0·2452 (2)	0·2645 (2)	2·70
C(11)	0·3783 (4)	0·3365 (7)	0·2099 (7)	2·67
C(12)	0·3978 (5)	0·4434 (8)	0·2479 (7)	3·34
C(121)	0·3996 (6)	0·4873 (8)	0·3380 (9)	5·22
C(13)	0·4164 (5)	0·5108 (8)	0·1980 (9)	3·96
C(14)	0·4187 (5)	0·4749 (9)	0·1173 (9)	4·57
C(15)	0·4005 (5)	0·3690 (9)	0·0838 (9)	4·61
C(16)	0·3807 (5)	0·3024 (8)	0·1305 (7)	3·58
C(21)	0·3404 (4)	0·1132 (6)	0·2089 (7)	2·64
C(22)	0·3992 (5)	0·0516 (7)	0·2418 (8)	3·53
C(221)	0·4691 (5)	0·0836 (9)	0·3331 (9)	4·62
C(23)	0·3910 (6)	−0·0461 (7)	0·1921 (8)	4·37
C(24)	0·3285 (6)	−0·0843 (7)	0·1119 (8)	4·03
C(25)	0·2719 (6)	−0·0238 (8)	0·0790 (9)	4·19
C(26)	0·2773 (5)	0·0734 (7)	0·1261 (8)	3·62
C(31)	0·2582 (4)	0·2894 (7)	0·2035 (7)	2·56
C(32)	0·2188 (5)	0·2482 (7)	0·2365 (8)	3·75
C(321)	0·2460 (5)	0·1674 (9)	0·3226 (9)	5·15
C(33)	0·1533 (4)	0·2858 (8)	0·1859 (7)	3·67
C(34)	0·1258 (5)	0·3596 (8)	0·1070 (8)	3·69
C(35)	0·1636 (5)	0·4005 (8)	0·0753 (8)	3·60
C(36)	0·2303 (5)	0·3634 (7)	0·1234 (7)	3·27

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Table 2. *Selected interatomic distances* (Å) *and bond angles* (°)

Au—Cl	2·281 (3)	Au—P	2·243 (2)
P—C(11)	1·840 (9)	P—C(21)	1·815 (8)
P—C(31)	1·835 (8)		
Cl—Au—P	179·4 (1)	Au—P—C(11)	114.5 (3)
Au—P—C(21)	112·8 (3)	Au—P—C(31)	114·2 (3)
C(11)—P—C(21)	104·7 (4)	C(11)—P—C(31)	104·1 (4)
C(21)—P—C(31)	105·6 (4)		

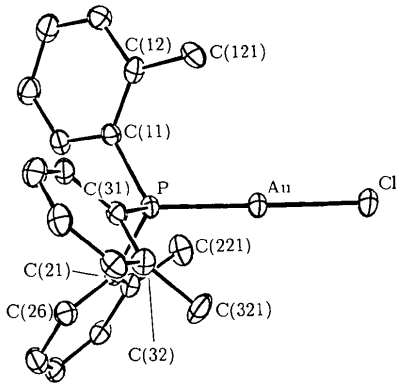

Fig. 1. Molecular structure and numbering scheme for $[(C_{21}H_{21}P)AuCl]$ (Johnson, 1971).

Fig. 1 which is drawn with *ORTEP* (Johnson, 1971) with 25% probability ellipsoids.

Related literature. As expected from cone-angle considerations (Tolman, 1977), the Au—P and Au—Cl bond distances in [(*o*-tolyl)$_3$PAuCl] with a cone angle of 194° are comparable to those found for [(cyclohexyl)$_3$PAuCl] of 2·242 (4) and 2·279 (5) Å with a cone angle of 170° (Muir, Muir, Pulgar, Jones & Sheldrick, 1985). In contrast, [(Et)$_3$PAuCl], with a cone angle of 132°, has Au—P and Au—Cl of 2·232 (9) and 2·305 (8) Å, respectively (Tiekink, 1989).

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Structure of a New Iodobismuthate: Tetra(*n*-butyl)ammonium 1,2;1,2;1,2;2,3;2,3;2,3-Hexa-μ-iodo-1,1,1,3,3,3-hexaiodotribismuthate(III) (3:1)

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Abstract. $(C_{16}H_{36}N)_3Bi_3I_{12}$, $M_r = 2877{\cdot}20$, monoclinic, $P2_1/c$, $a = 17{\cdot}796$ (4), $b = 20{\cdot}565$ (6), $c = 23{\cdot}414$ (8) Å, $\beta = 100{\cdot}25$ (2)°, $V = 8432$ (4) Å^3, $Z = 4$, $D_x = 2{\cdot}266$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 10{\cdot}61$ mm^{-1}, $F(000) = 5208$, room temperature, final $R = 0{\cdot}140$ for 4850 independent observed reflections with $F_o > 3\sigma(F_o)$. The compound contains a novel $Bi_3I_{12}^{3-}$ anion, consisting of three face-sharing $[BiI_6]$ octahedra. The bridging Bi—I bond lengths average 3·078 Å (inner Bi atom) and 3·330 Å (outer Bi atoms), whereas the bonds to terminal I atoms average 2·910 Å. Bond-length constraints were applied to the refinement of the tetrabutylammonium cation C atoms.

Experimental. Orange, plate-like needles were prepared from equimolar amounts of $[(n\text{-}C_4H_9)_4N]I$ and BiI_3, dissolved in hot absolute ethanol, by slow cooling. One of the largest available crystals (0·008 × 0·13 × 0·59 mm) was mounted on a Nicolet *P3/F* four-circle diffractometer with graphite monochromator. ω-scan data, 0·9° width, 2–8° min^{-1}, $2\theta < 45°$, $(\sin\theta)/\lambda < 0{\cdot}538$ Å^{-1} (no observed diffracted intensity beyond that), h: 0 to 19, k: 0 to 22, l: −25

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