

GOULD, R. O. & TAYLOR, P. (1985). *CALC*. Program for molecular geometry calculations. Univ. of Edinburgh, Scotland.

MALLINSON, P. D. & MUIR, K. W. (1985). *J. Appl. Cryst.* **18**, 51–53.

SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

WALKER, N. & STUART, D. (1983). *Acta Cryst.* **A39**, 158–166.

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Tetrakis(triphenylstibine)gold(I) Perchlorate

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Abstract. $[\text{Au}(\text{C}_{18}\text{H}_{15}\text{Sb})_4]\text{ClO}_4$, $M_r = 1708.6$, rhombohedral, $R\bar{3}$, $a = 14.5860$ (8), $c = 52.351$ (4) Å (hexagonal axes), $V = 9646$ Å³, $Z = 6$, $D_x = 1.765$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 4.0$ mm⁻¹, $F(000) = 4944$, $T = 293$ K, $R = 0.038$ for 3404 reflections. The Au atom and one Sb atom lie on the crystallographic threefold axis $\frac{2}{3}, \frac{1}{3}, z$; the coordination at the Au is almost exactly tetrahedral, with Au—Sb bond lengths of 2.658 (2) and 2.656 (2) Å, and Sb—Au—Sb bond angles of 108.8 (1) and 110.1 (1)°. The Cl atoms of the two independent perchlorate anions lie on special positions $\frac{1}{3}, \frac{2}{3}, \frac{1}{6}$ and 0,0,0 respectively; the site symmetry $\bar{3}$ is impossible for ordered perchlorate, and the O atoms are severely disordered.

Experimental. A colourless prism $0.55 \times 0.3 \times 0.2$ mm was mounted in a glass capillary. Intensities were registered to $2\theta_{\text{max}} = 50^\circ$ on a Stoe-Siemens four-circle diffractometer using monochromated Mo $K\alpha$ radiation; ω -scan technique. Of 7016 measured reflections, 3760 were unique ($R_{\text{int}} = 0.014$, index ranges $h - 17$ to 8, $k 0$ to 17, $l 0$ to 62) and 3404 with $F > 4\sigma(F)$ were considered observed. The cell constants were refined from $\pm\omega$ angles of 60 reflections in the 2θ range 20–23°. Three check reflections showed no significant intensity variation. An absorption correction based on ψ scans was applied, with transmission factors 0.62–0.85.

The structure was solved by the heavy-atom method and subjected to full-matrix least-squares refinement on F . The O atoms of the perchlorates were very badly resolved and are probably disordered over spheres with the Cl atoms as centres. Au, Sb and Cl atoms were refined anisotropically; idealized phenyl groups isotropically. The weighting scheme was $w^{-1} = \sigma^2(F) + 0.00015F^2$; final $R = 0.038$, with $wR = 0.042$; 76 parameters; $S 2.5$; maximum $\Delta/\sigma = 0.1$ for the disordered O atoms, other-

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters (Å² $\times 10^3$)

Equivalent isotropic U is defined as one third of the trace of the orthogonalized U_{ij} tensor

	x	y	z	U_{eq}
Au	6667	3333	5868.9 (1)	35 (1)
Sb(1)	6667	3333	5361.1 (1)	42 (1)
Sb(2)	5293.8 (3)	3895.0 (3)	6032.4 (1)	41 (1)
C(12)	8356 (4)	3650 (3)	4967 (1)	71 (2)
C(13)	9088	3445	4846	93 (3)
C(14)	9235	2623	4933	81 (2)
C(15)	8649	2006	5140	75 (2)
C(16)	7916	2210	5260	66 (2)
C(11)	7769	3032	5174	46 (1)
C(22)	4220 (3)	3878 (5)	6537 (1)	89 (3)
C(23)	4175	4066	6797	121 (4)
C(24)	5083	4450	6947	117 (4)
C(25)	6037	4647	6838	96 (3)
C(26)	6082	4459	6578	73 (2)
C(21)	5174	4075	6428	57 (2)
C(32)	3381 (4)	1688 (4)	5965 (1)	77 (2)
C(33)	2395	919	5873	96 (3)
C(34)	1739	1222	5750	81 (2)
C(35)	2070	2293	5719	75 (2)
C(36)	3056	3061	5811	60 (2)
C(31)	3712	2759	5934	47 (1)
C(42)	5708 (4)	5659 (4)	5653 (1)	66 (2)
C(43)	5667	6524	5553	79 (2)
C(44)	5239	7022	5698	70 (2)
C(45)	4852	6654	5943	78 (2)
C(46)	4893	5789	6043	56 (2)
C(41)	5321	5291	5898	43 (1)
Cl(1)	3333	6667	1667	77 (2)
Cl(2)	0	0	0	98 (2)
O(1)	4293 (14)	7169 (16)	1516 (3)	210 (7)
O(2)	9242 (20)	9213 (18)	174 (4)	303 (13)

wise $\Delta/\sigma = 0.04$; maximum, minimum $\Delta\rho = 1.5, -0.8$ e Å⁻³. Atomic scattering factors and f' , f'' values were taken from *International Tables for X-ray Crystallography*. (1974, Vol. IV). The program system used was Siemens *SHELXTL-Plus* (Sheldrick, 1989). Final atom coordinates are given in Table 1,* with derived bond lengths and angles in

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55012 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA0090]

Table 2. Bond lengths (Å) and angles (°)

Au—Sb(1)	2.658 (2)	Au—Sb(2)	2.656 (2)
Sb(1)—C(11)	2.110 (6)	Sb(2)—C(21)	2.105 (6)
Sb(2)—C(31)	2.124 (4)	Sb(2)—C(41)	2.136 (6)
Sb(1)—Au—Sb(2)	108.8 (1)	Sb(2)—Au—Sb(2')	110.1 (1)
Au—Sb(1)—C(11)	117.7 (1)	C(11)—Sb(1)—C(11')	100.1 (2)
Au—Sb(2)—C(21)	118.5 (2)	Au—Sb(2)—C(31)	112.0 (2)
C(21)—Sb(2)—C(31)	102.1 (2)	Au—Sb(2)—C(41)	122.0 (1)
C(21)—Sb(2)—C(41)	99.7 (2)	C(31)—Sb(2)—C(41)	99.2 (2)

Symmetry code: (i) $-y, x-y, z$

Table 2. Fig. 1. shows the atomic numbering scheme of the title cation.

Related literature. For other salts of the same cation see Jones (1982) and Vicente, Arcas, Jones & Lautner (1990).

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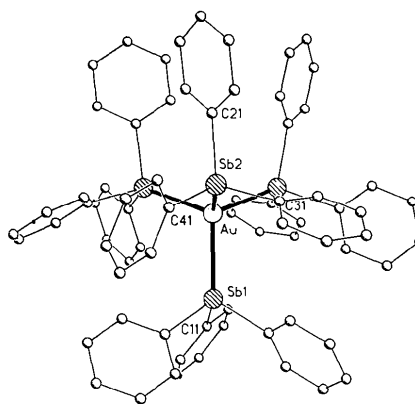


Fig. 1. The cation of the title compound, showing the numbering scheme of the asymmetric unit. Radii are arbitrary.

References

- JONES, P. G. (1982). *Z. Naturforsch. Teil B*, **37**, 937–940.
 SHELDRICK, G. M. (1989). *SHELXTL-Plus*. Version 4.1. Siemens Analytical X-ray Instruments, Inc., Madison, Wisconsin, USA.
 VICENTE, J., ARCAS, A., JONES, P. G. & LAUTNER, J. (1990). *J. Chem. Soc. Dalton Trans.* pp. 451–456.

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Structure of Chloro[*N*-(3-dimethylamino-1-propyl)salicylideneaminato]palladium(II)

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Abstract. C₁₂H₁₇ClN₂OPd, *M_r* = 347.135, orthorhombic, *Pbca*, *a* = 10.764 (1), *b* = 12.557 (4), *c* = 19.626 (2) Å, *V* = 2652.8 (9) Å³, *Z* = 8, *D_x* = 1.737 g cm⁻³, λ(Mo *Kα*₁) = 0.70926 Å, μ = 15.69 cm⁻¹, *F*(000) = 1392, *T* = 298 K, final *R* = 0.044 for 1769 reflections [*F_o* > 3σ(*F_o*)]. The coordination geometry around palladium in the title compound is almost square planar. The bond length of Pd—N2 [2.035 (7) Å] is shorter than that of Pd—N1 [2.101 (7) Å].

Experimental. The title compound [abbreviated as Pd(ONN)Cl] was synthesized as reported previously

(Oh, Yeo & Cho, 1991). Crystals suitable for X-ray work were obtained by allowing *n*-hexane to diffuse into a solution of Pd(ONN)Cl in CHCl₃. Orange crystal, 0.32 × 0.30 × 0.25 mm; Enraf–Nonius CAD-4 diffractometer; monochromated Mo *Kα* radiation; 25 reflections (12.0 < θ < 14.3°) used for measuring lattice parameters; ω/2θ scan, ω-scan width of (0.75 + 0.34tanθ)°; max. sinθ/λ = 0.5958 Å⁻¹; 0 ≤ *h* ≤ 12, 0 ≤ *k* ≤ 14, 0 ≤ *l* ≤ 23; 2663 unique reflections measured, 1769 observed [*I* > 3σ(*I*)]. The intensities of three standard reflections (143, 125, 108), recorded every 3 h of X-ray exposure, showed no significant variation. Lorentz–polarization correction; empirical absorption correction using *DIFABS* (Walker & Stuart, 1983) (min.

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