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trans-Chloro{3,4,5,6-tetrachloro-2-[(triphenylphosphine)aurio]phenyl}-bis(triphenylphosphine)platinum(II)

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Abstract. $C_{60}H_{45}AuCl_5P_3Pt$, triclinic, $P\bar{1}$, $a = 20{\cdot}039\,(3)$, $b = 13{\cdot}308\,(2)$, $c = 11{\cdot}699\,(1)$ Å, $\alpha = 67{\cdot}27\,(1)$, $\beta = 96{\cdot}67\,(1)$, $\gamma = 103{\cdot}27\,(1)^\circ$, $V = 2799{\cdot}4\,(7)$ Å^3, $Z = 2$, $D_c = 1{\cdot}69$ Mg m^{-3}, $\mu = 5{\cdot}68$ mm^{-1}. The positions of the Au and Pt atoms were determined by a Patterson synthesis, and those of the remaining non-H atoms using the *DIRDIF* program. The structure was refined by full-matrix least-squares methods to a final $R = 0{\cdot}035$ for 6622 observed reflections. The Pt atom has a planar four-coordination, with the triphenylphosphine groups in the *trans* position. The Au atom has a distorted linear coordination due to steric effects between the phenyl groups.

Introduction. The title compound was synthesized by Rosell & Sales (1981). Colourless prismatic crystals were obtained by evaporation from dichloromethane and methanol solutions. A crystal of 0·40 × 0·4 × 0·8 mm was used for crystal data and intensity measurements on a Syntex four-circle diffractometer. The unit cell was measured by automatically centring 25 independent reflections and refining the orientation matrix and unit-cell parameters by least squares. Intensities were collected with graphite-monochromated Mo $K\alpha$ radiation ($\lambda = 0{\cdot}71078$ Å). 8279 independent reflections were measured with the ω-scan technique up to $2\theta \leq 47^\circ$, 6222 of which were considered as observed by applying the condition $I \geq 2{\cdot}5\sigma(I)$. The data were collected with the crystal mounted along the long axis in order to minimize the absorption effects. Corrections were made for Lorentz–polarization effects but not for absorption.

The coordinates of Au and Pt atoms were obtained by means of a sharpened Patterson synthesis. The remainder of the non-H atoms were determined with the *DIRDIF* 80 system of computer programs (Beurskens, Bosman, Doesburg, Gould, Van den Hark & Prick, 1980).

The structure was isotropically and anisotropically refined by means of full-matrix least-squares methods with the *SHELX* 76 computer program (Sheldrick, 1976). The first refinement cycles employed isotropic thermal parameters, and the last refinement applied anisotropic thermal parameters for all non-H atoms. The function minimized was $\sum||F_o| - |F_c||^2$. The refinement was terminated at $R = 0{\cdot}035$, where R is defined as $\sum||F_o| - |F_c||/\sum|F_o|$.‡ A difference synthesis did not reveal the H atoms. The scattering factors used were taken from *International Tables for*

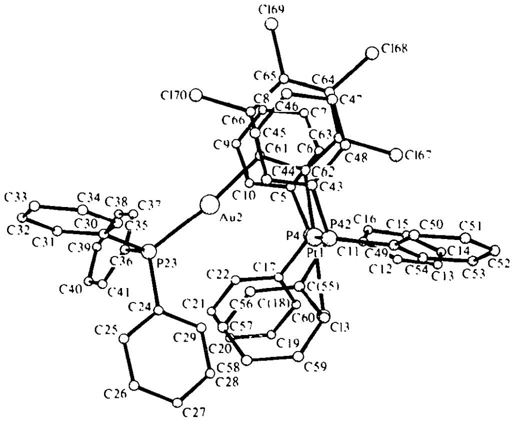

Fig. 1. View of the molecule with the numbering of atoms drawn by the program *PLUTO* 80 (W. D. S. Motherwell).

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‡ Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36662 (38 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Atomic parameters* ($\times 10^4$) *and equivalent isotropic thermal coefficients*

$B_{eq} = \frac{8}{3}\pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j$.

	x	y	z	B_{eq} (Å^2)
Pt(1)	7029 (1)	9293 (1)	6418 (1)	2·18 (1)
Au(2)	8262 (1)	7971 (1)	7784 (1)	2·85 (1)
Cl(3)	5953 (1)	18097 (2)	6251 (2)	3·49 (6)
P(4)	6698 (1)	9212 (2)	8301 (2)	2·41 (6)
C(5)	7380 (5)	9705 (8)	9260 (8)	3·0 (2)
C(6)	7614 (5)	10868 (9)	8913 (9)	3·6 (2)
C(7)	8180 (6)	11283 (10)	9557 (10)	4·5 (2)
C(8)	8506 (5)	10548 (12)	10544 (11)	5·1 (2)
C(9)	8265 (6)	9396 (11)	10908 (10)	5·1 (2)
C(10)	7700 (5)	8969 (9)	10263 (9)	4·1 (2)
C(11)	6076 (4)	10096 (8)	8057 (8)	2·9 (2)
C(12)	5717 (5)	10418 (9)	6932 (10)	4·1 (2)
C(13)	5220 (6)	11092(9)	6761 (12)	5·1 (2)
C(14)	5111 (6)	11423 (9)	7707 (13)	5·0 (2)
C(15)	5478 (6)	11069 (9)	8837 (13)	5·1 (2)
C(16)	5959 (5)	10410 (8)	9025 (9)	3·4 (2)
C(17)	6266 (5)	7823 (7)	9306 (8)	2·7 (2)
C(18)	5603 (5)	7614 (8)	9691 (9)	3·5 (2)
C(19)	5284 (6)	6525 (10)	10475 (10)	4·6 (2)
C(20)	5645 (6)	5673 (9)	10835 (10)	4·6 (2)
C(21)	6312 (6)	5878 (9)	10460 (10)	4·0 (2)
C(22)	6632 (5)	6956 (8)	9684 (10)	3·9 (2)
P(23)	8149 (1)	6075 (2)	8492 (3)	3·56 (6)
C(24)	7393 (5)	5275 (8)	7982 (9)	3·6 (2)
C(25)	7408 (6)	4263 (8)	7854 (11)	4·8 (2)
C(26)	6807 (7)	3691 (9)	7449 (12)	5·7 (2)
C(27)	6203 (6)	4114 (11)	7166 (13)	5·6 (2)
C(28)	6201 (5)	5116 (11)	7276 (12)	5·1 (2)
C(29)	6792 (5)	5709 (8)	7666 (9)	3·9 (2)
C(30)	8877 (5)	5681 (9)	8042 (12)	4·6 (2)
C(31)	9218 (7)	4877 (11)	8936 (14)	6·9 (2)
C(32)	9767 (7)	4616 (13)	8470 (17)	7·9 (2)
C(33)	9960 (8)	5169 (15)	7241 (19)	7·9 (2)
C(34)	9609 (7)	5945 (12)	6380 (16)	7·2 (2)
C(35)	9069 (6)	6219 (9)	6814 (12)	5·4 (2)
C(36)	8128 (5)	5529 (8)	10169 (10)	4·0 (2)
C(37)	7419 (6)	6252 (9)	10798 (10)	4·4 (2)
C(38)	8371 (6)	5867 (11)	12085 (11)	5·2 (2)
C(39)	8041 (6)	4779 (10)	12763 (11)	5·3 (2)
C(40)	7754 (7)	4074 (9)	12136 (11)	5·8 (2)
C(41)	7793 (7)	4440 (9)	10835 (11)	5·5 (2)
P(42)	7291 (1)	9555 (2)	4439 (2)	2·63 (6)
C(43)	8188 (5)	10129 (8)	4022 (8)	3·3 (2)
C(44)	8655 (5)	9465 (10)	4143 (10)	4·4 (2)
C(45)	9347 (6)	9971 (12)	3836 (12)	5·8 (2)
C(46)	9573 (6)	11088 (12)	3452 (12)	5·8 (2)
C(47)	9115 (7)	11756 (12)	3352 (12)	6·5 (2)
C(48)	8414 (5)	11283 (9)	3641 (10)	4·3 (2)
C(49)	6830 (5)	10543 (8)	3247 (8)	3·3 (2)
C(50)	7042 (6)	11014 (8)	1999 (9)	4·2 (2)
C(51)	6720 (6)	11787 (9)	1104 (10)	4·8 (2)
C(52)	6185 (7)	12121 (9)	1439 (11)	5·4 (2)
C(53)	5955 (6)	11646 (10)	2677 (12)	5·2 (2)
C(54)	6290 (5)	10853 (9)	3598 (10)	4·3 (2)
C(55)	7088 (5)	8296 (7)	4075 (8)	3·1 (2)
C(56)	7336 (6)	7373 (9)	4885 (9)	4·2 (2)
C(57)	7225 (7)	6397 (9)	4645 (11)	5·4 (2)
C(58)	6850 (7)	6346 (9)	3578 (10)	4·9 (2)
C(59)	6584 (7)	7259 (10)	2773 (10)	5·2 (2)
C(60)	6711 (5)	8251 (9)	3020 (10)	4·0 (2)
C(61)	8462 (5)	9673 (7)	7291 (8)	2·8 (2)
C(62)	7939 (4)	10247 (7)	6614 (8)	2·5 (2)
C(63)	8070 (5)	11389 (7)	6252 (8)	3·0 (2)
C(64)	8701 (5)	11997 (8)	6498 (9)	3·3 (2)
C(65)	9201 (5)	11411 (8)	7220 (10)	3·8 (2)
C(66)	9053 (5)	10254 (8)	7630 (8)	3·0 (2)
Cl(67)	7429 (1)	12119 (2)	5462 (2)	4·02 (6)
Cl(68)	8856 (2)	13425 (2)	5997 (3)	6·32 (6)
Cl(69)	9967 (2)	12141 (3)	7608 (4)	6·59 (6)
Cl(70)	9645 (1)	9542 (2)	8664 (3)	4·52 (6)

X-ray Crystallography (1974) and the anomalous-scattering factors from Cromer & Liberman (1970). Fig. 1 shows a view of the molecule and the numbering of atoms. Table 1 shows the final atomic parameters and the equivalent isotropic thermal coefficients.

Discussion. Figs. 2 and 3 show bond distances and angles. The Au atom is found in a distorted linear coordination. The bond angle C(61)—Au(2)—P(23) [173·3 (2)°] is smaller than the value observed in chloro(triphenylphosphine)gold(I) [179·63 (8)°] (Baenziger, Bennett & Soboroff, 1976) or in methyl(triphenylphosphine)gold(I) [179·1 (8)°] (Gavens, Guy, Mays & Sheldrick, 1977). It seems to be due to steric hindrance between C(24), C(29) and C(17), C(22) in the phenyl groups [C(29)···C(21) interatomic distance is 3·45 (1) Å]. The C—Au bond distance (Table 2) is intermediate between the C(sp^3)—Au and the C(sp)—Au bond distance, while the P—Au bond distance is longer than the values observed in the literature (Table 2). The torsion angles P—Au—C(61)—C are −165·3 (7)° for C=C(62) and 11·7 (7)° for C=C(66). From these data it is inferred that π interaction does not exist between C and Au atoms.

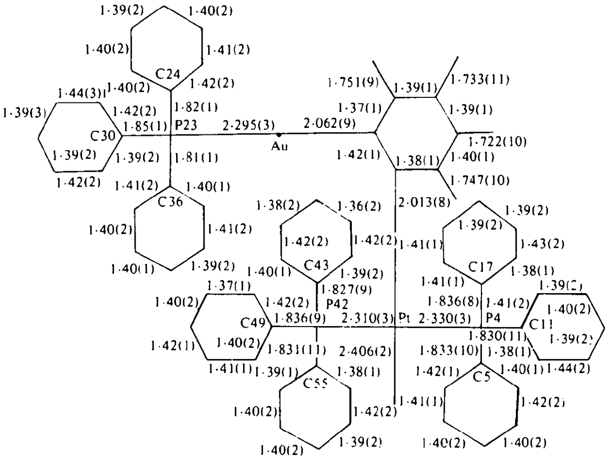

Fig. 2. Bond distances in Å.

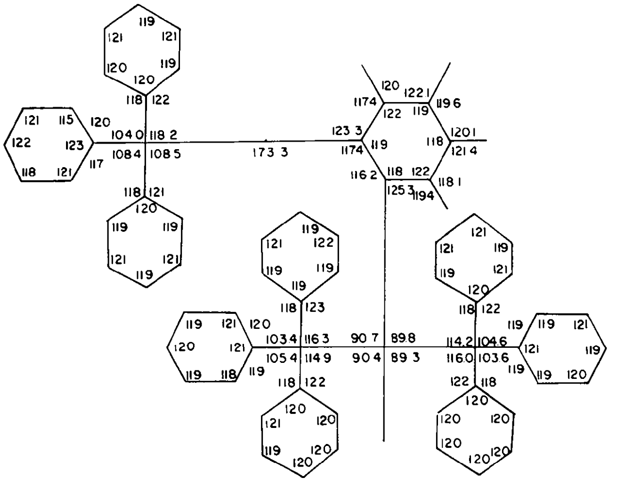

Fig. 3. Bond angles in degrees [e.s.d.'s: C—C—*R* (*R* = C or P) ≤ 1°; C—P—C < 0·6°, C—C—Cl < 0·7°; and *A*—*R*—*B* (*R* = Au or Pt) < 0·3°].

Table 2. *Comparison of bond distances* (Å) *of several related compounds*

	Title compound		Related compounds	References
C—Au	2·062 (9)	C(sp^3)—Au	2·124 (28)	
		C(sp)—Au	1·85(4)—1·93 (2)	(*b*, *c*)
P—Au	2·295 (3)		2·212—2·279 (8)	(*a*, *c*, *d*, *e*)
Cl—Pt	2·406 (2)		2·296 (5)—2·45 (1)	(*f*, *g*)
C—Pt	2·013 (8)		2·04 (18)*	(*f*, *g*, *h*, *i*, *j*, *k*, *l*)
P—Pt	2·320 (6)*		2·30 (7)*	(*f*, *g*, *h*, *i*, *j*, *k*, *l*)

References: (*a*) Gavens *et al.* (1977). (*b*) Corfield & Shearer (1967). (*c*) Bellon, Manassero & Sansoni (1969). (*d*) Baenziger *et al.* (1976). (*e*) Clegg (1976). (*f*) Chiesi Villa, Gaetani Manfredotti, Guastini, Carusi, Furlani & Russo (1977*a*). (*g*) Chiesi Villa, Gaetani Manfredotti, Guastini, Carusi, Furlani & Russo (1977*b*). (*h*) O'Flynn & McDonald (1976). (*i*) Chiesi Villa, Gaetani Manfredotti & Guastini (1976). (*j*) Del Pra, Zanotti, Bardi, Belluco & Michelin (1979). (*k*) Bonamico, Dessy, Fares, Russo & Scaramuzza (1977). (*l*) Chiesi Villa, Gaetani Manfredotti, Guastini & Russo (1977).

* Mean value.

The Pt atom displays a planar four-coordination. The largest deviation from the mean plane is 0·141 (6) Å and corresponds to the P(4) atom. The Pt—Cl, Pt—C and Pt—P distances are similar to those obtained in other compounds with Pt—Cl, Pt—C(sp^2), Pt—C(sp) and Pt—P distances (Table 2). The torsion angles P—Pt(1)—C(62)—C are: 87·2 (7) [P(4),C(61)]; −86·2 (7) [P(4),C(63)]; −99·8 (7) [P(42),C(61)]; and 86·5(7)° [P(42),C(63)] respectively. Consequently, there are no π interactions between the C and Pt atoms.

The P atoms display a distorted tetragonal coordination with C—P—*M* (*M* = metal) mean angle equal to 114 (3)° (range: 108·5–118·2°). This value is 114·6° in the compounds mentioned in Table 2 (range: 103·4–108·4°) (mean value in the literature 104·7°, range: 101·6–108·6°).

The benzene ring of the tetrachlorophenyl group is planar within experimental error [largest atom-to-mean-plane deviation = −0·04 (2) Å in C(61) and 0·04 (2) Å in C(66)]. The bond distances C(61)—C(62) = 1·42 (1), C(66)—Cl(70) = 1·751 (9) and C(63)—Cl(67) = 1·747 (10) Å are larger than C(ar)—C(ar) = 1·397 Å and C(ar)—Cl = 1·716 Å [1·705 (6)–1·730 (7) Å] (Solans, Gali, Miravitlles & Font-Altaba, 1978; Gali, Solans, Miravitlles, Font-Altaba & Armet, 1978; Miravitlles, Solans, Germain & Declercq, 1979), while the Au or Pt—C—C angles are smaller than 120°. These differences are due to steric hindrance between the Cl atoms and the Pt^{II} and Au^{I} ions. Cl···Cl mean distance = 3·12, Cl···Pt = 3·408, Cl···Au = 3·384 and Pt···Au = 3·265 Å. The atom-to-benzene-mean-plane deviations are Cl(67) 0·13, Cl(68) −0·04, Cl(69) 0·02, Cl(70) 0·25, Pt 0·218 and Au −0·114 Å.

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2,2′:6′,2″-Terpyridyl–Aquaisothiocyanatotriphenyltin(IV)(1:1)*

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Abstract. $C_{15}H_{11}N_3.C_{19}H_{17}NOSSn$, triclinic, $P\bar{1}$, a = 10·326 (2), b = 10·739 (2), c = 15·880 (2) Å, α = 105·04 (1), β = 97·14 (1), γ = 111·5 (1)°, Z = 2, D_c = 1·427, D_m = 1·432 g cm^{-3}. The final residual is R_F = 0·026 on 4581 observed reflections at 296 K. The Sn atom is coordinated to the NCS group, the three phenyl groups and the water-molecule O atom in a trigonal-bipyramidal arrangement. The terpyridyl group is hydrogen-bonded to the water-molecule O atom.

Introduction. Complex formation between neutral, planar tridentate ligands and tin tetrachloride, mono-organotin(IV) chlorides and diorganotin(IV) chlorides is well known (Fergusson, Roper & Wilkins, 1965; May & Curran, 1972; Pelizzi & Pelizzi, 1976; Einstein & Penfold, 1966). In particular, the chelating agent 2,2′:6′,2″-terpyridyl forms complexes with all these Sn^{IV} species, behaving as a tridentate in each case. Seven-coordination at the Sn atom has been proposed in several instances (May & Curran, 1972; Pelizzi & Pelizzi, 1976) and confirmed by crystal-structure analysis of the compound diisothiocyanatodimethyl(terpyridyl)tin(IV) (Naik & Scheidt, 1973). However, no seven-coordinate Sn^{IV} complex with three Sn—C bonds has yet been characterized. Recently, the complex of triphenyltin chloride with 2,2′:6′,2″-terpyridyl was found to have a most unusual structure, in which the terpyridyl moiety was not bound directly to the Sn atom at all, but hydrogen-bonded to a coordinated water molecule (Prasad, Lee, Le Page & Smith, 1982).

Corresponding chloro and isothiocyanato organotin complexes often have different structures, *e.g.* the 2,2′-bipyridyl complexes of diphenyltin dichloride and diphenyltin diisothiocyanate both have approximately octahedral structures, but in the former, the phenyl groups are *trans* to each other (Harrison, King & Richards, 1974) whereas in the latter complex, the hydrocarbons are *cis* to each other (Gabe, Prasad, Le Page & Smith, 1982). Consequently, it was considered worthwhile to synthesize the isothiocyanato analogue of the complex between triphenyltin chloride and 2,2′:6′,2″-terpyridyl.

Triphenyltin chloride was a gift from M & T Chemicals. An ethanolic solution of triphenyltin isothiocyanate was prepared from the chloride by a metathetical reaction in ethanol and the precipitated potassium chloride was filtered off. The complex was obtained in good yield by mixing hot ethanolic solutions containing stoichiometric quantities of triphenyltin isothiocyanate and 2,2′:6′,2″-terpyridine. White crystals of the complex were obtained by recrystallization from ethanol (m.p. 391–393 K). Microanalyses were carried out by the Australian Microanalytical service, CSIRO, Melbourne, Australia. Found (%): C 61·88, H 4·29, N 8·46, Sn 18·6; calculated (%) for $C_{34}H_{28}N_4OSSn$: C 61·93, H 4·28, N 8·50, Sn 18·0.

The diffraction intensities of a 0·35 × 0·35 × 0·35 mm crystal were measured at room temperature with graphite-monochromatized Mo $K\alpha$ radiation generated at 50 kV, 16 mA, using the $\theta/2\theta$ scan technique with line-profile analysis (Grant & Gabe, 1978). A total of

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