Table 1. *Atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

	Occupation	x	y	z	U_{eq}
Sb(1)	1.000	0.000	0.000	0.000	0.0462
Cl(2)	1.000	0.17224 (9)	0.0723 (1)	0.1408 (1)	0.0768
C(3)	0.500	0.047 (2)	0.0701 (7)	0.4880 (7)	0.0401
N(4)	0.500	0.1243 (5)	0.1864 (4)	0.4875 (6)	0.0534
C(5)	0.500	0.250 (1)	0.229 (1)	0.501 (2)	0.0700
C(6)	0.500	0.081 (1)	0.270 (1)	0.497 (2)	0.0678
H(51)		0.264 (5)	0.205 (7)	0.589 (7)	
H(52)		0.294 (7)	0.189 (9)	0.460 (9)	
H(53)		0.284 (5)	0.290 (5)	0.491 (6)	
H(61)		0.054 (5)	0.277 (5)	0.599 (6)	
H(62)		0.143 (5)	0.342 (6)	0.467 (5)	
H(63)		0.010 (6)	0.265 (6)	0.423 (8)	

Table 2. *Distances* (Å) *and angles* (°)

C(3)—N(4)	1.318 (5)	C(3)^i—C(3)—N(4)	149.8 (8)
C(3)—C(3)^{ii}	1.374 (9)	C(3)^i—C(3)—C(3)^{ii}	60.0 (4)
C(3)—C(3)^i	1.37 (1)		
N(4)—C(3)	1.318 (5)	C(5)—N(4)—C(3)	120.3 (3)
N(4)—C(5)	1.43 (1)	C(6)—N(4)—C(3)	119.4 (3)
N(4)—C(6)	1.43 (1)	C(6)—N(4)—C(5)	119.5 (3)
C(5)—H(53)	0.68 (3)	H(51)—C(5)—H(52)	86.1 (3)
C(5)—H(51)	0.96 (4)	H(51)—C(5)—H(53)	112 (1)
C(5)—H(52)	1.02 (6)	H(52)—C(5)—H(53)	109 (1)
C(5)—N(4)	1.43 (12)	N(4)—C(5)—H(52)	125 (3)
		N(4)—C(5)—H(53)	113 (1)
		N(4)—C(5)—H(51)	108.7 (8)
C(6)—H(62)	0.92 (3)	H(61)—C(6)—H(62)	110.4 (9)
C(6)—H(61)	1.07 (3)	H(63)—C(6)—H(62)	97.1 (3)
C(6)—H(63)	1.14 (4)	H(63)—C(6)—H(61)	107.1 (8)
C(6)—N(54)	1.43 (1)	H(4)—C(6)—H(62)	106.5 (5)
		N(4)—C(6)—H(61)	113.0 (8)
		N(4)—C(6)—H(63)	121 (1)
Sb(1)—Cl(2)	2.356 (1)	Cl(2)—Sb(1)—Cl(2)^i	89.972 (6)

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

$\Delta\rho_{max} = 0.85$, $\Delta\rho_{min} = -0.71$ e Å^{-3}, respectively. Extinction corrections were applied according to Zachariasen's (1968) formula; maximum extinction factor was 1.5; form-factor tables from *International Tables for X-ray Crystallography* (1962, Vol. III). All computations were performed on a MicroVAX II computer using a modified version of the program system *ATARI CRYSTAN*88 (1989). Positional parameters and the equivalent isotropic values of the anisotropic displacement factors are given in Table 1.* Bond lengths and bond angles are given in Table 2.

Related literature. The structure is strongly related to the structure of tris(dimethylamino)cyclopropenylium hexachloroniobate and -hexachlorotantalate with $\bar{c}_{Sb} = \frac{1}{2}c_{Nb,Ta}$ (Schäfer, Burzlaff, Grimmeiss & Weiss, 1992). The position of the organic cation results from the superposition of the unique cations in the hexachloroniobate and the hexachlorotantalate.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54767 (5 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Di-μ-chloro-bis[1,3-bis(di-*tert*-butylphosphino)propaneplatinum(II)] Tetrafluoroborate 0.8-Dichloromethane Solvate

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Abstract. $C_{38}H_{84}Cl_2P_4Pt_2^{2+}.2BF_4^-.0.8CH_2Cl_2$, M_r = 1367.7, triclinic, $P\bar{1}$, a = 11.191 (3), b = 11.512 (3), c = 13.134 (3) Å, α = 65.35 (2), β = 77.58 (2), γ = 66.31 (2)°, V = 1406.1 (6) Å^3, Z = 1, D_x = 1.615 Mg m^{-3}, F(000) = 677.6, λ(Mo $K\alpha$) = 0.7107 Å, μ = 5.36 mm^{-1}, T = 203 K, R = 0.042 for 5836 observed reflections. The two halves of the dimeric cation are related by a crystallographic centre of symmetry, the Pt atoms adopting a distorted square-planar geometry. The Pt—P distances

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Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic displacement coefficients* (Å^2 × 10^3)

	x	y	z	U_{eq}*
Pt(1)	−1006 (1)	326 (1)	1259 (1)	21 (1)
Cl(1)	−424 (2)	−1183 (2)	265 (1)	43 (1)
P(1)	−2285 (1)	−790 (1)	2581 (1)	23 (1)
P(2)	−1497 (1)	2080 (1)	1863 (1)	23 (1)
C(5)	−3269 (5)	9 (5)	3573 (4)	28 (2)
C(6)	−2667 (6)	718 (6)	3953 (4)	30 (2)
C(7)	−2715 (5)	2109 (5)	3028 (4)	28 (2)
C(1)	−3587 (6)	−863 (6)	1903 (5)	34 (2)
C(11)	−3103 (7)	−2004 (6)	1447 (6)	46 (3)
C(12)	−4085 (7)	516 (6)	950 (6)	43 (3)
C(13)	−4763 (6)	−1021 (7)	2760 (6)	48 (3)
C(2)	−1256 (6)	−2558 (5)	3488 (5)	33 (2)
C(21)	−560 (7)	−2399 (6)	4272 (5)	43 (3)
C(22)	−2110 (7)	−3410 (6)	4209 (5)	44 (3)
C(23)	−222 (6)	−3339 (6)	2807 (5)	40 (2)
C(3)	−45 (5)	2095 (6)	2360 (5)	31 (2)
C(31)	318 (7)	919 (7)	3483 (5)	44 (3)
C(32)	−377 (7)	3437 (7)	2545 (5)	41 (3)
C(33)	1171 (6)	1945 (6)	1540 (5)	39 (3)
C(4)	−2319 (6)	3767 (5)	715 (5)	30 (2)
C(41)	−3196 (7)	4869 (6)	1196 (6)	45 (3)
C(42)	−1345 (7)	4337 (6)	−171 (5)	40 (3)
C(43)	−3192 (7)	3526 (6)	127 (5)	41 (3)
B(1)	−3448 (7)	−2996 (7)	6946 (6)	41 (3)
F(21)	−3164 (6)	−1816 (6)	6405 (5)	82 (3)
F(22)	−4310 (5)	−2937 (7)	6294 (5)	83 (3)
F(23)	−2342 (4)	−4098 (5)	7056 (4)	74 (2)
F(24)	−4073 (5)	−3003 (5)	7976 (3)	73 (2)
C(11a)	−3438 (13)	3821 (13)	6527 (10)	79 (3)
Cl(3)	−2792 (4)	2294 (4)	7730 (3)	91 (2)
Cl(4)	−3216 (4)	3380 (4)	5335 (3)	97 (2)
Cl(3a)	−3567 (16)	3071 (16)	7613 (13)	93 (4)
Cl(4a)	−3415 (19)	3721 (20)	5521 (17)	127 (5)

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

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

Pt(1)—Cl(1)	2.401 (2)	Pt(1)—P(1)	2.294 (2)
Pt(1)—P(2)	2.294 (2)	Pt(1)—Cl(1a)	2.405 (2)
Cl(1)—Pt(1a)	2.405 (2)	P(1)—C(5)	1.823 (6)
P(1)—C(1)	1.908 (9)	P(1)—C(2)	1.890 (5)
P(2)—C(7)	1.819 (5)	P(2)—C(3)	1.887 (8)
P(2)—C(4)	1.900 (5)	C(5)—C(6)	1.515 (11)
C(6)—C(7)	1.542 (7)	C(1)—C(11)	1.524 (12)
C(1)—C(12)	1.526 (8)	C(1)—C(13)	1.554 (9)
C(2)—C(21)	1.522 (12)	C(2)—C(22)	1.537 (10)
C(2)—C(23)	1.529 (9)	C(3)—C(31)	1.524 (8)
C(3)—C(32)	1.548 (11)	C(3)—C(33)	1.541 (8)
C(4)—C(41)	1.540 (9)	C(4)—C(42)	1.538 (9)
C(4)—C(43)	1.532 (12)		
Cl(1)—Pt(1)—Pt(1)	92.3 (1)	Cl(1)—Pt(1)—P(2)	168.5 (1)
P(1)—Pt(1)—P(2)	97.9 (1)	Cl(1)—Pt(1)—Cl(1a)	77.3 (1)
P(1)—Pt(1)—Cl(1a)	169.6 (1)	P(2)—Pt(1)—Cl(1a)	92.5 (1)
Pt(1)—Cl(1)—Pt(1a)	102.7 (1)	Pt(1)—P(1)—C(5)	116.2 (2)
Pt(1)—P(1)—C(1)	111.4 (2)	C(5)—P(1)—C(1)	101.9 (3)
Pt(1)—P(1)—C(2)	111.4 (2)	C(5)—P(1)—C(2)	104.6 (2)
C(1)—P(1)—C(2)	111.1 (2)	Pt(1)—P(2)—C(7)	115.6 (2)
Pt(1)—P(2)—C(3)	113.6 (2)	C(7)—P(2)—C(3)	104.1 (3)
Pt(1)—P(2)—C(4)	109.4 (2)	C(7)—P(2)—C(4)	101.8 (2)
C(3)—P(2)—C(4)	111.7 (3)	P(1)—C(5)—C(6)	117.0 (4)
C(5)—C(6)—C(7)	110.5 (5)	P(2)—C(7)—C(6)	116.7 (5)
P(1)—C(1)—C(11)	114.3 (4)	P(1)—C(1)—C(12)	106.7 (5)
C(11)—C(1)—C(12)	109.7 (6)	P(1)—C(1)—C(13)	109.9 (6)
C(11)—C(1)—C(13)	108.8 (6)	C(12)—C(1)—C(13)	107.2 (5)
P(1)—C(2)—C(21)	107.6 (4)	P(1)—C(2)—C(22)	111.2 (4)
C(21)—C(2)—C(22)	108.1 (5)	P(1)—C(2)—C(23)	112.9 (4)
C(21)—C(2)—C(23)	108.2 (5)	C(22)—C(2)—C(23)	108.7 (6)
Pt(2)—C(3)—C(31)	108.9 (5)	P(2)—C(3)—C(32)	111.0 (4)
C(31)—C(3)—C(32)	107.6 (6)	P(2)—C(3)—C(33)	113.5 (5)
C(31)—C(3)—C(33)	108.1 (4)	C(32)—C(3)—C(33)	107.5 (6)
P(2)—C(4)—C(41)	111.8 (4)	P(2)—C(4)—C(42)	113.3 (4)
C(41)—C(4)—C(42)	107.8 (5)	P(2)—C(4)—C(43)	107.7 (4)
C(41)—C(4)—C(43)	107.8 (5)	C(42)—C(4)—C(43)	108.4 (6)

are equivalent [2.294 (2), 2.294 (1) Å], as are the Pt—Cl distances [2.401 (2), 2.405 (2) Å]. The Cl(1)—Pt(1)—Cl(1a) angle is 77.3 (1)°.

Experimental. Pale yellow-green crystals were obtained as rectangular blocks by recrystallization of the crude compound from dichloromethane. Crystal size for data collection 0.33 × 0.25 × 0.15 mm, Siemens *Rm*/*V* diffractometer with graphite monochromator. Cell dimensions from 21 centred reflections with 2θ values 20 to 36°. Total of 6789 reflections measured to $2\theta = 55°$ using θ–2θ scans in the ranges $0 \leq h \leq 15$, $-15 \leq k \leq 15$ and $-18 \leq l \leq 18$. Numerical absorption correction based on face indexing was applied, minimum/maximum transmission 0.34/0.47. The data were corrected for a 2% decay in intensity as indicated by two reference reflections which were monitored periodically. Structure solution, refinement and graphics using *SHELXTL PLUS* (Sheldrick, 1988). Structure determined using direct methods, refined by full-matrix least squares on F with all non-H atoms refined with anisotropic thermal parameters. H atoms were included in the refinement in their calculated positions and allowed to ride on their respective C atoms with fixed isotoropic thermal parameters. There was evidence for disorder associated with the dichloromethane molecule which was best modelled by giving it a partial occupancy of 0.8.* 6445 independent reflections, $R_{int} = 0.021$, 5836 with $F > 4\sigma(F)$. At convergence $R = 0.042$, $wR = 0.054$ (on all data $R = 0.056$, $wR = 0.080$), $w = [\sigma^2(F) + 0.0021F^2]^{-1}$, $S = 1.03$ for 283 parameters, maxi-

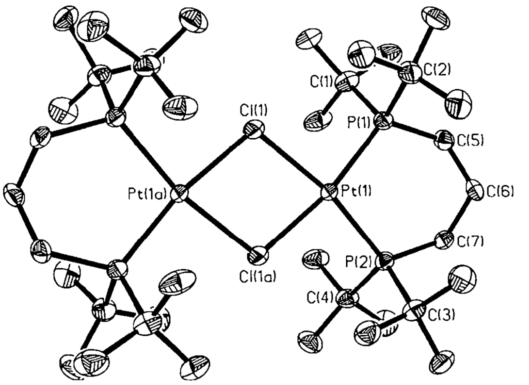

Fig. 1. Molecular structure of the title cation together with the numbering scheme used.

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* The Cl atoms were disordered over two sites with occupancies 0.6 and 0.2.

mum $\Delta/\sigma = 0.041$, $\Delta\rho_{\max} = 3.63$ e Å^{-3} at a distance of 0.94 Å from the Pt atom, $\Delta\rho_{\min} = -4.61$ e Å^{-3}. Scattering factors for all atoms from *SHELXTL PLUS*. Atomic parameters are given in Table 1,* and selected bond distances and bond angles are listed in Table 2. Fig. 1 shows the cation together with the numbering scheme used.

Related literature. The related mononuclear compound [Pt{’Bu$_2$P(CH$_2$)$_3$P’Bu$_2$}Cl$_2$] has been structurally characterized (Harada, Kai, Yasuoka & Kasai, 1979). The expected differences associated with the Pt-ligand parameters on going to the dinuclear, Cl-bridged complex cation are observed. Thus the Pt—P and Pt—Cl distances are lengthened and the Cl—Pt—Cl angle is substantially reduced. Furthermore, the correlation between equivalence of Pt—Cl distances with equivalence of the P(1)—Pt(1)—Cl(1a) and P(2)—Pt(1)—Cl(1) angles (Robertson & Wickramasinghe, 1987) is maintained.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond distances and angles including the anion have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54792 (28 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI0109]

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Structure of Tris(dicyclohexylphenylphosphine)gold(I) Perchlorate

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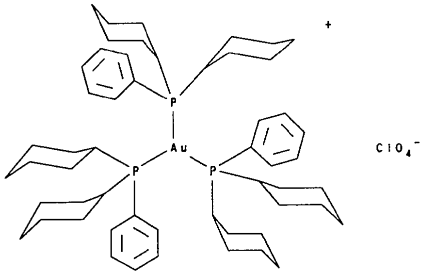
Abstract. [Au{P(C$_6$H$_{11}$)$_2$(C$_6$H$_5$)}$_3$][ClO$_4$], M_r = 1119.59, trigonal, $R\bar{3}c$, a = 13.192 (5), c = 52.83 (2) Å, V = 7962 (9) Å^3, Z = 6, D_x = 1.401 g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, μ = 29.5 cm^{-1}, F(000) = 3468, T = 296 K, final R = 0.047 for 1321 unique observed reflections. The Au atom in the anion and the perchlorate ion in [Au(Cy$_2$PhP)$_3$][ClO$_4$] lie on a threefold axis. The complex has an almost ideal trigonal-planar geometry, with an Au—P distance of 2.421 (3) Å, P—Au—P angles of 119.9 (3)°, and the Au atom only 0.08 (2) Å out of the plane defined by the three P atoms.

Experimental. To a beaker of 40 ml ethanol were added 0.20 mmol of [Cy$_2$PhPAuCl], prepared following the procedure used by Sutton (Sutton, McGusty, Walz & Di Martino, 1972). A stoichiometric amount of ammonium perchlorate and a sixfold excess of Cy$_2$PhP (Strem) were then added under nitrogen atmosphere in a glove bag. After stirring for about 20 min the beaker was removed from the glove bag and a white solid (NH$_4$Cl) was filtered from solution. After some time colorless crystals formed in solution and were isolated by vacuum filtration. Analysis for C$_{54}$H$_{81}$AuClO$_4$P$_3$: Calc. C 57.93, H 7.29%; found, C 58.02, H 7.29%.



Small rectangular plate, size and D_m not measured, Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$, ω–2θ scan, scan width (0.9 + 0.34tanθ)°, $2\theta_{\max}$ = 57°, lattice parameters from least-squares fit of 24 reflections in the range $12 < \theta < 15°$. Conditions limiting reflections hkl ($-h + k + l = 3n$); $00l$ ($l = 6n$); with $\underline{h} \le 17$, $\underline{k} \le 17$, $l \le 71$; 3 standard reflections, (3,4,$\overline{22}$, 4,$\bar{7}$,$\overline{22}$, $\bar{7}$,4,16), total intensity loss 0.7% during 17.1 h exposure, total reflections measured 2768, no equivalent reflections measured, 2224 of these unique, 1321 with $F^2 > 2\sigma^2(F_o)$, $\sigma(F_o) = \{[\sigma^2(I_{\text{raw}}) + (0.04F_o{}^2)^2]^{1/2}/2F_o\}$.

0108-2701/92/050915-03$06.00