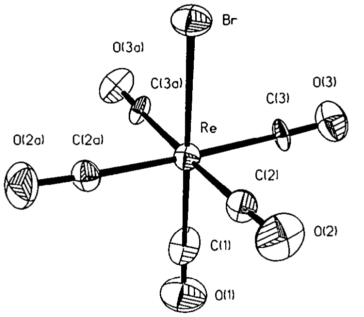

Fig. 1. A perspective view of the bromopentacarbonylrhenium(I) structure illustrating the atomic numbering scheme. Thermal ellipsoids have been drawn at the 50% probability level.

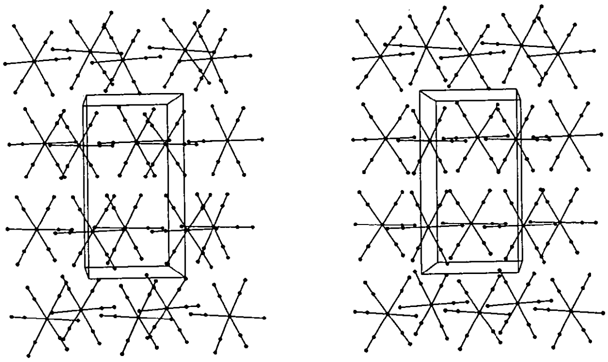

Fig. 2. A stereoview packing diagram projected down the crystallographic *a* axis.

Related literature. The reaction of Re dimers possessing *M*—*M* bonds with CO under pressure frequently results in the formation of monomeric octahedral complexes. The structure of the complex described here has been reported previously (Couldwell & Simpson, 1977). It is similar to that of an $ReCl_4(PMe_2Ph)_2$ complex (Aslanov, Mason, Wheeler & Whimp, 1970) and a related $ReCl_4(PEt_3)_2$ complex (Bucknor, Cotton, Falvello, Reid & Schmulbach, 1986).

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

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	*y*	*z*	U_{eq}
Re	1235 (1)	2500	9473 (1)	23 (1)
Br	2550 (2)	2500	6076 (3)	38 (1)
O(1)	− 267 (5)	2500	13431 (5)	58 (1)
O(2)	− 167 (5)	4443 (5)	7321 (5)	55 (1)
O(3)	2740 (5)	4404 (5)	11445 (5)	54 (1)
C(1)	280 (5)	2500	11913 (5)	33 (1)
C(2)	330 (5)	3719 (5)	8094 (5)	29 (1)
C(3)	2206 (5)	3718 (5)	10748 (5)	27 (1)

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

Re—Br	2.619 (2)	Re—C(1)	1.889 (5)
Re—C(2)	1.975 (5)	Re—C(3)	1.991 (6)
O(1)—C(1)	1.143 (6)	O(2)—C(2)	1.135 (8)
O(3)—C(3)	1.108 (8)		
Br—Re—C(1)	179.7 (2)	Br—Re—C(2)	88.7 (1)
C(1)—Re—C(2)	91.0 (2)	Br—Re—C(3)	88.4 (1)
C(1)—Re—C(3)	91.8 (2)	C(2)—Re—C(3)	88.6 (2)
C(2)—Re—C(2*A*)	91.9 (3)	C(3)—Re—C(2*A*)	177.1 (2)
C(3)—Re—C(3*A*)	90.8 (3)	Re—C(1)—O(1)	177.8 (5)
Re—C(2)—O(2)	178.0 (5)	Re—C(3)—O(3)	179.3 (5)

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Structure of [(1,2-η^2)-1,3,5,7-Cycloheptatetraene]bis(triphenylphosphine)platinum(0)

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Abstract. $[Pt\{(1,2\text{-}\eta^2)\text{-}C_7H_6\}\{P(C_6H_5)_3\}_2]$, (I), M_r = 809.82, monoclinic, $P2_1/c$, $a = 13.664\,(7)$, $b = 16.783\,(8)$, $c = 16.702\,(8)$ Å, $\beta = 111.68\,(4)°$, $V = 3559\,(3)$ Å^3, $Z = 4$, $D_x = 1.51$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 41.00$ cm^{-1}, $F(000) = 1608$, $T = 298$ K, $R = 0.0329$ and $wR = 0.0366$ for 3807 reflec-

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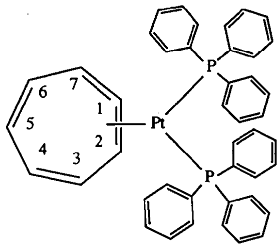
tions $[I \geq 3\sigma(I)]$. The Pt atom is involved in asymmetric bonding with the C and P atoms. Pt—C(1) is shorter than Pt—C(2) [2.000 (7) and 2.111 (8) Å]. This difference in bond lengths is the result of a difference in the bonding environments around the two C atoms. C(1) is slightly more electronegative than C(2) owing to an extra π bond around C(1). Consequently, C(1) exerts a slightly stronger *trans* effect than C(2) by lengthening bond Pt—P(2) [2.299 (2) Å compared to 2.287 (2) Å for Pt—P(1)]. The Pt atom lies in the plane of coordination which forms an angle of 131.5 (6)° with the plane of atoms C(1)—C(2)—C(3)—C(7). The torsion angle H(2)—C(2)—C(3)—H(3) is 83 (8)°, close to 90°, and therefore no coupling was found between H(2) and H(3) in 2D (COSY) ^{1}H NMR [Winchester & Jones (1985). *Organometallics*, **4**, 2228–2230]. This study not only confirms the allene form (1) rather than the carbene form (2) for the Pt complex of C_7H_6 in the solid state as well as in solution, but the torsion angle of nearly 90° between H(2) and H(3) explains why they are virtually uncoupled in the ^{1}H NMR spectrum.

Experimental. Crystals of (I) were obtained by slow evaporation from a mixture of THF (tetrahydrofuran) and hexane. The data crystal (a purple needle) had dimensions 0.18 × 0.20 × 0.29 mm. Data were collected at 298 K on a Siemens *R3m/E* diffractometer equipped with a graphite monochromator utilizing Mo $K\alpha$ radiation ($\lambda = 0.71069$ Å). 25 reflections with $20 \leq 2\theta \leq 22°$ were used to refine the cell parameters. 11 516 reflections (two equivalent sets) were collected using the ω-scan method ($h\ 0 \rightarrow 17$, $k\ -19 \rightarrow 19$, $l\ -19 \rightarrow 19$), 5540 unique reflections, $R_{\text{int}} = 0.0142$; 2θ range 3–50°, 1.2° ω-scan at 3–6° min^{-1}, depending on intensity. Four reflections (102, $\bar{1}\bar{6}6$, $\bar{2}\bar{1}0$, 060) were measured every 96 reflections to monitor instrument and crystal stability (maximum correction on I was < 1.032%). Absorption corrections were applied based on measured crystal faces using *SHELXTL* (Sheldrick, 1986); minimum and maximum transmission 0.350 and 0.438, respectively.



(I)

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^3$)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Pt	1048 (1)	−835 (1)	1910 (1)	45 (1)
P(1)	1877 (1)	−1636 (1)	1251 (1)	46 (1)
P(2)	2373 (2)	−246 (1)	3039 (1)	52 (1)
C(1)	−495 (6)	−1028 (4)	1301 (4)	62 (3)
C(2)	−429 (6)	−472 (4)	1916 (5)	65 (3)
C(3)	−875 (7)	−627 (5)	2550 (6)	73 (4)
C(4)	−1765 (9)	−1016 (6)	2403 (7)	92 (5)
C(5)	−2421 (8)	−1386 (8)	1633 (8)	114 (6)
C(6)	−2181 (8)	−1661 (8)	965 (7)	139 (7)
C(7)	−1186 (7)	−1608 (5)	899 (5)	86 (4)
C(11)	3053 (5)	−1253 (4)	1116 (4)	54 (3)
C(12)	3994 (5)	−1234 (4)	1794 (5)	61 (3)
C(13)	4851 (6)	−872 (6)	1746 (6)	89 (4)
C(14)	4773 (10)	−471 (8)	998 (8)	147 (7)
C(15)	3821 (10)	−459 (9)	320 (7)	163 (8)
C(16)	2964 (7)	−852 (5)	370 (5)	94 (4)
C(21)	2277 (5)	−2597 (4)	1793 (4)	50 (3)
C(22)	3134 (6)	−3028 (4)	1785 (5)	70 (4)
C(23)	3391 (7)	−3759 (5)	2220 (6)	91 (4)
C(24)	2802 (7)	−4035 (5)	2658 (6)	102 (4)
C(25)	1949 (8)	−3620 (5)	2661 (6)	86 (4)
C(26)	1691 (6)	−2896 (4)	2245 (4)	67 (3)
C(31)	1053 (5)	−1913 (4)	150 (4)	47 (3)
C(32)	1147 (6)	−2649 (4)	−208 (4)	66 (3)
C(33)	523 (7)	−2831 (5)	−1039 (5)	80 (4)
C(34)	−190 (6)	−2258 (5)	−1547 (4)	83 (4)
C(35)	−282 (6)	−1549 (5)	−1215 (4)	69 (3)
C(36)	342 (6)	−1375 (4)	−368 (4)	63 (3)
C(41)	3488 (6)	−872 (4)	3641 (4)	60 (3)
C(42)	3262 (7)	−1653 (4)	3810 (4)	74 (3)
C(43)	4070 (7)	−2180 (5)	4225 (5)	107 (5)
C(44)	5094 (7)	−1958 (6)	4440 (5)	111 (5)
C(45)	5326 (7)	−1191 (6)	4280 (5)	106 (5)
C(46)	4527 (6)	−648 (5)	3888 (5)	80 (4)
C(51)	2966 (5)	629 (4)	2749 (4)	56 (3)
C(52)	3503 (7)	1207 (5)	3335 (5)	88 (4)
C(53)	3953 (7)	1847 (5)	3096 (6)	99 (5)
C(54)	3860 (7)	1934 (5)	2261 (6)	97 (5)
C(55)	3342 (7)	1373 (5)	1667 (5)	92 (4)
C(56)	2889 (6)	723 (4)	1908 (5)	69 (3)
C(61)	1945 (5)	132 (4)	3890 (4)	58 (3)
C(62)	1233 (7)	741 (5)	3681 (5)	88 (4)
C(63)	813 (7)	1044 (5)	4264 (5)	87 (4)
C(64)	1133 (7)	699 (5)	5074 (5)	97 (5)
C(65)	1850 (8)	94 (5)	5292 (5)	106 (5)
C(66)	2254 (7)	−202 (5)	4704 (4)	80 (4)

The structure was solved by the heavy-atom method (Patterson in *SHELXTL*) from which the position of the Pt atom was obtained. The positions of the rest of the non-H atoms were obtained from a subsequent difference Fourier map. The structure was refined (*SHELX*76; Sheldrick, 1976) using cascade-matrix least squares. All phenyl H atoms were calculated in idealized positions; each was given a fixed isotropic thermal parameter equal to 1.2 times the equivalent isotropic thermal parameter of the C atom to which it is bonded. The allene H atoms were obtained from a difference Fourier map and refined with no constraints; H(6) was calculated in an idealized position but its thermal parameter was allowed to refine. All non-H atoms were refined with anisotropic thermal parameters. 436 parameters were refined and $\sum w(|F_o| - |F_c|)^2$ was minimized, $w = 1/(\sigma|F_o|)^2$, $\sigma(F_o) = 0.5\,kI^{-1/2}\,\{[\sigma(I)]^2 + (0.02I)^2\}^{1/2}$, I(intensity) = $(I_{\text{peak}} - I_{\text{background}})$(scan rate), and $\sigma(I) = (I_{\text{peak}} + I_{\text{background}})^{1/2}$ (scan rate), k is the correction for decay and Lp effects, 0.02 is a factor used to down-weight intense reflections and to account for

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

Pt—P(1)	2.287 (2)	Pt—P(2)	2.299 (2)
Pt—C(1)	2.000 (7)	Pt—C(2)	2.111 (8)
P(1)—C(11)	1.820 (8)	P(1)—C(21)	1.832 (6)
P(1)—C(31)	1.825 (6)	P(2)—C(41)	1.818 (7)
P(2)—C(51)	1.826 (7)	P(2)—C(61)	1.839 (8)
C(1)—C(2)	1.365 (11)	C(1)—C(7)	1.350 (10)
C(2)—C(3)	1.429 (15)	C(3)—C(4)	1.321 (15)
C(4)—C(5)	1.411 (15)	C(5)—C(6)	1.355 (20)
C(6)—C(7)	1.407 (15)	C(11)—C(12)	1.364 (8)
C(11)—C(16)	1.381 (12)	C(12)—C(13)	1.348 (12)
C(13)—C(14)	1.387 (17)	C(14)—C(15)	1.373 (15)
C(15)—C(16)	1.373 (18)	C(21)—C(22)	1.381 (11)
C(21)—C(26)	1.382 (12)	C(22)—C(23)	1.402 (11)
C(23)—C(24)	1.353 (15)	C(24)—C(25)	1.360 (14)
C(25)—C(26)	1.379 (11)	C(31)—C(32)	1.399 (10)
C(31)—C(36)	1.375 (9)	C(32)—C(33)	1.368 (9)
C(33)—C(34)	1.409 (10)	C(34)—C(35)	1.339 (12)
C(35)—C(36)	1.386 (9)	C(41)—C(42)	1.399 (10)
C(41)—C(46)	1.377 (11)	C(42)—C(43)	1.384 (11)
C(43)—C(44)	1.362 (14)	C(44)—C(45)	1.376 (14)
C(45)—C(46)	1.385 (12)	C(51)—C(52)	1.381 (10)
C(51)—C(56)	1.377 (11)	C(52)—C(53)	1.367 (13)
C(53)—C(54)	1.361 (14)	C(54)—C(55)	1.361 (11)
C(55)—C(56)	1.386 (12)	C(61)—C(62)	1.364 (11)
C(61)—C(66)	1.385 (10)	C(62)—C(63)	1.394 (15)
C(63)—C(64)	1.385 (12)	C(64)—C(65)	1.363 (13)
C(65)—C(66)	1.384 (15)		

P(1)—Pt—P(2)	105.5 (1)	P(1)—Pt—C(1)	106.0 (2)
P(2)—Pt—C(1)	148.2 (2)	P(1)—Pt—C(2)	144.7 (2)
P(2)—Pt—C(2)	109.7 (2)	C(1)—Pt—C(2)	38.7 (3)
Pt—P(1)—C(11)	118.0 (2)	Pt—P(1)—C(21)	113.0 (2)
C(11)—P(1)—C(21)	104.9 (3)	Pt—P(1)—C(31)	113.8 (2)
C(11)—P(1)—C(31)	102.1 (3)	C(21)—P(1)—C(31)	103.5 (3)
Pt—P(2)—C(41)	116.7 (2)	Pt—P(2)—C(51)	114.9 (2)
C(41)—P(2)—C(51)	104.4 (3)	Pt—P(2)—C(61)	113.8 (2)
C(41)—P(2)—C(61)	102.2 (3)	C(51)—P(2)—C(61)	103.1 (3)
Pt—C(1)—C(2)	75.0 (4)	Pt—C(1)—C(7)	141.5 (6)
C(2)—C(1)—C(7)	134.9 (9)	Pt—C(2)—C(1)	66.3 (5)
Pt—C(2)—C(3)	128.4 (5)	C(1)—C(2)—C(3)	120.4 (7)
C(2)—C(3)—C(4)	124.6 (9)	C(3)—C(4)—C(5)	128.1 (12)
C(4)—C(5)—C(6)	129.4 (11)	C(5)—C(6)—C(7)	125.3 (9)
C(1)—C(7)—C(6)	121.7 (9)	P(1)—C(11)—C(12)	120.6 (6)
P(1)—C(11)—C(16)	119.7 (5)	C(12)—C(11)—C(16)	118.7 (7)
C(11)—C(12)—C(13)	122.2 (7)	C(12)—C(13)—C(14)	119.5 (8)
C(13)—C(14)—C(15)	118.9 (12)	C(14)—C(15)—C(16)	120.8 (12)
C(11)—C(16)—C(15)	119.7 (8)	P(1)—C(21)—C(22)	123.8 (6)
P(1)—C(21)—C(26)	117.6 (5)	C(22)—C(21)—C(26)	118.7 (6)
C(21)—C(22)—C(23)	120.2 (8)	C(22)—C(23)—C(24)	119.6 (8)
C(23)—C(24)—C(25)	120.6 (8)	C(24)—C(25)—C(26)	120.5 (10)
C(21)—C(26)—C(25)	120.3 (8)	P(1)—C(31)—C(32)	122.4 (4)
P(1)—C(31)—C(36)	119.8 (5)	C(32)—C(31)—C(36)	117.8 (5)
C(31)—C(32)—C(33)	120.7 (6)	C(32)—C(33)—C(34)	119.5 (7)
C(33)—C(34)—C(35)	120.5 (6)	C(34)—C(35)—C(36)	119.6 (6)
C(31)—C(36)—C(35)	122.0 (7)	P(2)—C(41)—C(42)	116.8 (5)
P(2)—C(41)—C(46)	124.6 (6)	C(41)—C(42)—C(43)	120.3 (8)
C(42)—C(41)—C(46)	118.4 (7)	C(43)—C(44)—C(45)	119.5 (8)
C(42)—C(43)—C(44)	120.6 (9)	C(41)—C(46)—C(45)	120.5 (8)
C(44)—C(45)—C(46)	120.6 (9)	P(2)—C(51)—C(56)	119.2 (5)
P(2)—C(51)—C(52)	123.3 (6)	C(51)—C(52)—C(53)	121.5 (8)
C(52)—C(51)—C(56)	117.5 (7)	C(53)—C(54)—C(55)	119.9 (9)
C(52)—C(53)—C(54)	120.1 (8)	C(51)—C(56)—C(55)	120.9 (7)
C(54)—C(55)—C(56)	120.0 (9)	P(2)—C(61)—C(66)	123.4 (6)
P(2)—C(61)—C(62)	117.5 (6)	C(61)—C(62)—C(63)	122.2 (7)
C(62)—C(61)—C(66)	118.9 (8)	C(63)—C(64)—C(65)	120.5 (10)
C(62)—C(63)—C(64)	117.8 (8)	C(61)—C(66)—C(65)	119.6 (8)
C(64)—C(65)—C(66)	120.9 (8)		

instrument instability. Final $R = 0.0329$, $wR = 0.0366$ ($R_{\text{all}} = 0.0613$, $wR_{\text{all}} = 0.0449$) for 3807 reflections having $I \geq 3\sigma(I)$, and goodness of fit = 1.22. Maximum $\Delta/\sigma = 0.02$ in the final refinement cycle and the minimum and maximum peaks in the ΔF map were -0.91 and $1.47\ \text{e}\ \text{Å}^{-3}$, respectively. The highest peak in the ΔF map was 1.11 Å from the Pt atom and thus was attributed to its anisotropy. The linear absorption coefficient was calculated using values from *International Tables for X-ray Crystallography* (1974, Vol. IV, p.55). Scattering factors for non-H atoms were taken from Cromer & Mann (1968) with anomalous-dispersion corrections from Cromer & Liberman (1970), while those of H atoms were from Stewart, Davidson & Simpson (1965). The positional parameters and the equivalent isotropic thermal parameters of the non-H atoms are listed in Table 1,* bond lengths and angles are in Table 2. The thermal ellipsoid drawing (*SHELXTL*, Sheldrick, 1986) of the molecule with the atom labelling scheme is given in Fig. 1.

Related literature. Depending on the metal and its ligands, theory predicts that transition-metal complexes of monocyclic C_7H_6 may exist in either an allene form (1) (Winchester, 1985) or a carbene form (2). The carbene form has been unequivocally confirmed for the F_p^+ (F_p = dicarbonyl-η^5-cyclopentadienyliron) complex in both solution (Allison, Kawada & Jones, 1978) and in the crystal (Riley, Davis, Allison & Jones, 1980). In contrast, NMR indicates an allene form for the bistriphenylphosphine Pt complex in solution (Winchester & Jones, 1985). However, to date there has been no evidence to support this structure in the crystal. This study confirms that the preferred form for the bistriphenylphosphine Pt complex in the crystal is also the allene. This study also explains why H(2) and H(3) with a torsion angle

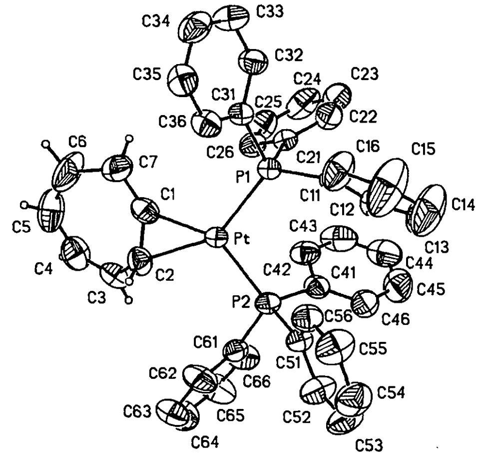


Fig. 1. Molecular structure of (I), with 50% probability ellipsoids, showing the atom-numbering scheme (for clarity phenyl H atoms are not included).

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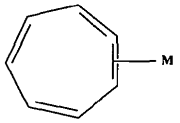
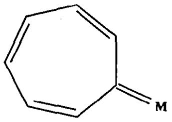
* Lists of anisotropic thermal parameters, H-atom positional parameters, bond lengths and angles involving H atoms, and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54686 (38 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST0542]

of nearly 90° are not coupled in the ^1H NMR spectrum, a fact that led to the misassignments of key H atoms [H(2) and H(7)] in an earlier publication (Winchester & Jones, 1985).



(1) Allene



(2) Carbene

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Structure of 1,2,3-Tris(dimethylamino)cyclopropenylium Hexachloroantimonate(V)

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Abstract. $[C_9H_{18}N_3][SbCl_6]$, $M_r = 502.73$, trigonal, $R\bar{3}$, $a = 12.8388$ (3), $c = 9.6665$ (8) Å, $V = 1380$ Å^3, $Z = 3$, $D_x = 1.815$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0.70926$ Å, $\mu = 23.808$ cm^{-1}, $F(000) = 738$, room temperature, final $R = 0.024$ for F and 549 reflections. The crystal structure consists of layers parallel to the *ab* plane. The organic cation is fixed between two octahedra perpendicular to this plane; the two possible orientations are occupied statistically as already demanded by space group symmetry. There was no evidence for doubling of the *c* parameter.

Experimental. A solution of 3 mmol tris(dimethylamino)cyclopropenylium chloride in 30 ml CH_2Cl_2 was added to a solution of 3 mmol $SbCl_5$ in 15 ml CH_2Cl_2. Stirring the mixture led to a violet fall-out of the title compound. Recrystallization in a mixture of 15 ml CH_3CN and 0.5 ml $SOCl_2$ led to needles of the title compound. The specimen used for the structure determination had dimensions 0.18 × 0.18 × 0.36 mm.

All measurements were performed on a PW 1100 instrument rebuilt and equipped with additional facilities (Gomm, 1992). ω–2θ scan, graphite-monochromated Mo $K\alpha$ radiation, modified Lehmann–Larsen profile analysis; lattice parameters derived from 33 reflections with $3 \leq \theta \leq 8°$; absorption correction by using a modified version of *CAMEL JOCKEY* (Flack, 1975) based on empirical ψ-scan data, max. and min. correction factors 1.36 and 1.70; intensities collected for $-15 \leq h \leq 15$, $-15 \leq k \leq 15$, $-11 \leq l \leq 11$, $\theta_{\max} = 25°$; six standard reflections, no significant variation; 3259 reflections measured, 549 unique reflections, no unobserved reflections omitted; $R_{\rm int}$ based on F is 0.0214.

The Sb and Cl atoms were determined using the Patterson method. The rest of the non-H atoms were determined from a Fourier map. A subsequent difference Fourier map revealed all H atoms. Full-matrix least squares based on F; weights derived from experimental standard deviations $w = 1/\sigma(F)$. In the final stage, anisotropic displacement parameters were used for all non-H atoms and isotropic displacement parameters for the H atoms. Final $R = 0.024$, $wR = 0.026$, $S = 1.69$, $(\Delta/\sigma)_{\max} = 0.01$. Maximum and minimum electron density residuals are

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