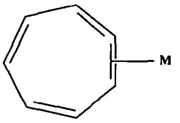
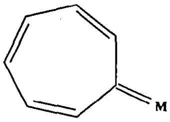
of nearly 90° are not coupled in the ^1H NMR spectrum, a fact that led to the misassignments of key H atoms [H(2) and H(7)] in an earlier publication (Winchester & Jones, 1985).



(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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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 $\bar{c}_{\mathrm{Sb}} = \frac{1}{2}c_{\mathrm{Nb,Ta}}$ (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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