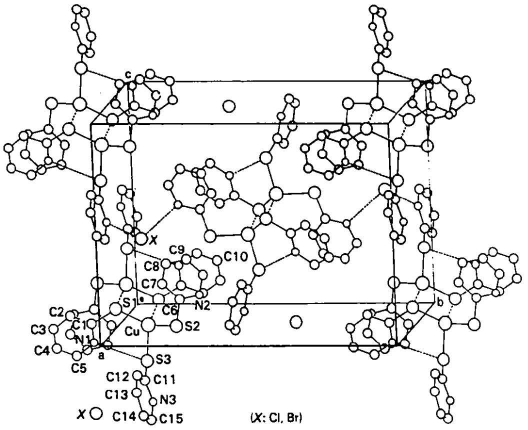

Fig. 2. Clinographic projection of the unit-cell contents of the title compounds.

References

Gagné, R. R., Kreh, R. P., Dodge, J. A., Marsh, R. E. & McCool, M. (1982). *Inorg. Chem.* **21**, 254–261.

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 71–151. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

Kokkou, S. C., Schramm, V. & Karagiannidis, P. (1985). *Acta Cryst.* C**41**, 1040–1043.

Main, P., Lessinger, L., Woolfson, M. M., Germain, G. & Declercq, J.-P. (1977). *MULTAN77. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Okaya, Y. & Knobler, C. B. (1964). *Acta Cryst.* **17**, 928–930.

Stewart, J. M., Machin, P. A., Dickinson, C. W., Ammon, H. L., Heck, H. & Flack, H. (1976). The *XRAY* system. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland, USA.

Summerville, R. H. & Hoffmann, R. (1976). *J. Am. Chem. Soc.* **98**, 7240–7254.

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Structure of 1,1-Dichloro-1-methyl-1λ^5-stibacyclohexane

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Abstract. $C_6H_{13}Cl_2Sb$, $M_r = 277{\cdot}8$, orthorhombic, *Pbcn*, $a = 10{\cdot}624\ (8)$, $b = 9{\cdot}094\ (6)$, $c = 10{\cdot}281\ (9)$ Å, $V = 993\ (1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}858$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 32{\cdot}7$ cm^{-1}, $F(000) = 536$, $T = 295$ K. $R = 0{\cdot}025$ for 709 observed reflections with $I > 2{\cdot}5\sigma(I)$. The six-membered stibacyclohexane ring is puckered into a chair conformation. The coordination of Sb is slightly distorted trigonal bipyramidal with the three C atoms in equatorial positions and the two Cl atoms in axial positions. The endocyclic C—Sb—C bond angle is 106·0 (2)° and significantly smaller than the exocyclic C—Sb—C bond angles of 127·0 (1)°.

Introduction. The present structure determination was carried out in relation to a study of the correlation of C—Sb—C bond angles in *cis*-diorganoantimony(V) compounds with ^{121}Sb Mössbauer spectra (Barbieri, Bertazzi, Gibb, Meinema & Noltes, 1979).

Experimental. Colorless needle-shaped crystal (0·12 × 0·12 × 0·82 mm) glued on top of a glass fiber. Enraf–Nonius CAD-4*F* diffractometer, Zr-filtered Mo*K*α radiation. 1344 reflections scanned, $\omega/2\theta$ scan, $\Delta\omega = (0{\cdot}60 + 0{\cdot}35\tan\theta)°$; $2{\cdot}2 < \theta < 27{\cdot}5°$; h: 0→13, k: 0→11, l 0→13. Two reference reflections (232: e.s.d. 1·4%; $\bar{2}3\bar{2}$: e.s.d. 1·0%), linear decay of 20% during 31 h of X-ray exposure time. Cell dimensions from the setting angles of eight reflections ($\theta \simeq 10{\cdot}5°$). An analysis of the lattice symmetry (Le Page, 1982) did not reveal any symmetry higher than *oP*. Space group *Pbcn* from the observed extinctions ($0kl$: $k = 2n+1$; $h0l$: $l = 2n+1$; $hk0$: $h+k = 2n+1$). Correction for Lp and linear decay but not for absorption. 1140 unique reflections of which 431 had $I < 2{\cdot}5\sigma(I)$. The structure was solved by standard Patterson and Fourier methods. Full-matrix least-squares refinement on F (excluding H atoms) with anisotropic thermal parameters converged to $R = 0{\cdot}0309$. An analysis of the resulting essentially planar six-membered-ring geometry showed some unsatisfactory short bonds (1·41–1·44 Å) involving

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C(2) [in between C(1) and C(3)] and rather large bond angles at C(2) of 130° and C(3) of 133°. Inspection of the thermal ellipsoids showed a large component for C(2) in a direction perpendicular to the ring plane. It was concluded that C(2) was at the average position of the two positions [C(21) and C(22)] corresponding to a disorder model involving two six-membered rings in the chair conformation that are related by a crystallographic twofold axis that passes through C(3), C(4) and Sb. Refinement was continued with this disorder model assuming 1:1 disorder of the C(2) position only. H atoms were introduced at calculated positions conforming with the chair conformation of the ring and the disorder with an overall isotropic temperature factor ($U = 0{\cdot}07$ Å^2). Convergence was reached at $R = 0{\cdot}0254$ [$wR = 0{\cdot}0294$; $w = 1/\sigma^2(F)$; $S = 0{\cdot}91$; $(\Delta/\sigma)_{\max} = 0{\cdot}2$] for 709 observed reflections [$I > 2{\cdot}5\sigma(I)$] and 56 parameters. Residual density range: 0·60 (near Sb) and −0·50 e Å^{-3}. Scattering factors from Cromer & Mann (1968) and anomalous-dispersion corrections from Cromer & Liberman (1970). Calculations carried out on the Cyber 175 of the University of Utrecht with programs of the *SHELX*76 (Sheldrick, 1976) and *EUCLID* (Spek, 1982) packages.

Table 1. *Final atomic coordinates and equivalent isotropic thermal parameters for the non-*H *atoms*

The six-membered ring consists of the atom sequence Sb,C(1),-C(21),C(3),C(22^{i}) and C(1^{i}). Symmetry code: (i) $1-x$, y, $\frac{1}{2}-z$.

$$U_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	P.p.	$U_{\rm eq}$(Å^2)
Sb	0·5	0·60318 (5)	0·25	1·0	0·0505 (1)
Cl	0·3378 (1)	0·5952 (2)	0·0762 (2)	1·0	0·0948 (5)
C(1)	0·3859 (5)	0·7431 (6)	0·3643 (5)	1·0	0·071 (2)
C(21)	0·444 (2)	0·888 (2)	0·371 (1)	0·5*	0·093 (6)
C(22)	0·383 (1)	0·892 (2)	0·306 (1)	0·5*	0·077 (5)
C(3)	0·5	0·960 (1)	0·25	1·0	0·101 (4)
C(4)	0·5	0·3723 (7)	0·25	1·0	0·075 (3)

* Disordered position.

Table 2. *Bond distances* (Å), *angles* (°) *and torsion angles* (°) *for the non-*H *atoms*

Symmetry code: (i) $1-x, y, \frac{1}{2}-z$.

Sb–Cl	2·483 (3)	C(1)–C(21)	1·46 (2)
Sb–C(1)	2·114 (6)	C(22)–C(1)	1·48 (2)
Sb–C(4)	2·100 (7)	C(21)–C(3)	1·53 (2)
		C(3)–C(22)	1·50 (1)
Cl–Sb–Cl^{i}	176·65 (6)	Sb–C(1)–C(21)	109·1 (8)
Cl–Sb–C(4)	88·33 (4)	Sb–C(1)–C(22)	109·7 (5)
Cl–Sb–C(1)	91·1 (1)	C(1)–C(21)–C(3)	121·0 (9)
Cl–Sb–C(1^{i})	90·9 (1)	C(1)–C(22)–C(3)	120·9 (9)
C(1)–Sb–C(1^{i})	106·0 (2)	C(21)–C(3)–C(22^{i})	117·0 (10)
C(1)–Sb–C(4)	127·0 (1)		
C(4)–Sb–C(1)–C(21)	159·6 (6)	C(1)–C(21)–C(3)–C(22^{i})	−69 (2)
C(4)–Sb–C(1)–C(22)	−161·1 (5)	C(21)–C(1)–Sb–C(1^{i})	−20·4 (7)
Sb–C(1)–C(21)–C(3)	44 (2)	C(22)–C(1)–Sb–C(1′)	18·9 (6)
Sb–C(1)–C(22)–C(3)	−41 (1)		

Discussion. Final atomic parameters are given in Table 1[*] and derived bond distances, bond angles and torsion angles in Table 2. Fig. 1 illustrates the molecular geometry and the atom numbering. The unit cell contains four molecules that are disordered according to the crystallographic twofold-axis site symmetry as shown in Fig. 2.

The five-coordination about Sb is slightly distorted bipyramidal with the apical SbCl bonds slightly tilted towards the methyl group. Sb lies exactly in the equatorial plane defined by the atoms C(1), C(1^{i}) and C(4). The observed distortion of the coordination polyhedron is of the non-Berry *b* type (Holmes, 1984).

The six-membered ring is in a chair conformation with average torsion angle 43·2 (5)°. The endocyclic C(1)SbC(1^{i}) = 106·0 (2)° bond angle is significantly

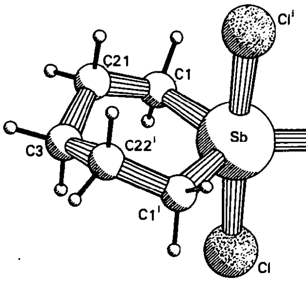

Fig. 1. Drawing of the molecule with adopted numbering scheme. The molecule is disordered according to the crystallographic twofold axis that passes through C(3), C(4) and Sb. For symmetry code see Table 1.

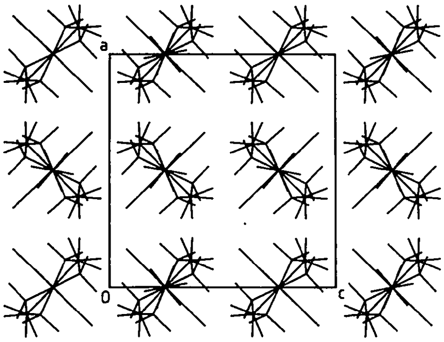

Fig. 2. View of the structure down the *b* axis, illustrating the mirror-plane type of disorder that conforms with the approximate *mm* symmetry present in the packing.

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[*] Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths involving H have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43989 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

smaller than the value of 120° reported for trimethylstibine dichloride (Wells, 1938) but larger than the corresponding value of 90·4 (8)° observed in the related five-membered-ring structure (Meinema, Noltes, Spek & Duisenberg, 1987).

No short intermolecular Sb···Cl contacts of the type observed in the related five-membered-ring compound are found.

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References

Barbieri, R., Bertazzi, N., Gibb, T. C., Meinema, H. A. & Noltes, J. G. (1979). *J. Chem. Soc. Dalton Trans.* pp. 1925–1928.

Cromer, D. T. & Liberman, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

Holmes, R. R. (1984). *Prog. Inorg. Chem.* **32**, 119–235.

Le Page, Y. (1982). *J. Appl. Cryst.* **15**, 255–259.

Meinema, H. A., Noltes, J. G., Spek, A. L. & Duisenberg, A. J. M. (1987). *J. Organomet. Chem.* To be submitted.

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

Spek, A. L. (1982). *Computational Crystallography*, edited by D. Sayre, p. 528. Oxford: Clarendon Press.

Wells, A. F. (1938). *Z. Kristallogr.* **99**, 367–377.

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Bis[trichloro(diméthylsulfoxyde)platinate(II)] de Tétraammineplatine(II)

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Abstract. $[Pt(NH_3)_4][PtCl_3(CH_3)_2SO]_2$, $M_r = 1022{\cdot}4$, monoclinic, $C2/c$, $a = 21{\cdot}438$ (4), $b = 7{\cdot}098$ (2), $c = 14{\cdot}655$ (3) Å, $\beta = 102{\cdot}55$ (1)°, $V = 2176{\cdot}6$ (9) Å^3, $Z = 4$, $D_x = 3{\cdot}12$ Mg m^{-3}, $\lambda(\text{Mo } K\bar{\alpha}) = 0{\cdot}7107$ Å, $\mu = 20{\cdot}4$ mm^{-1}, $F(000) = 1840$, $T = 295$ (1) K. $R = 0{\cdot}040$ for 2162 independent reflections [$I > 3\sigma(I)$]. The structure contains $[Pt(NH_3)_4]^{2+}$ and $[PtCl_3(CH_3)_2SO]^-$ ions. The coordination around the two Pt atoms is square planar. Bond distances and angles agree with previously published values. The structure can be regarded as constituted of layers parallel to the (100) plane. In each layer, there is a sheet of cations between two sheets of anions. Anions and cations belonging to the same layer are linked together by N—H···O and N—H···Cl hydrogen bonds.

Introduction. La détermination de la structure cristalline du composé décrit dans le présent mémoire s'inscrit dans le cadre de l'étude des composés de coordination formés par le platine(II) avec l'ammoniac et les amines aliphatiques. Elle fait suite à celle du bis[trichloro(diméthylsulfoxyde)platinate(II)] de bis(propanediamine-1,3)platine(II) (Viossat, Toffoli, Khodadad & Rodier, 1987*a*). Pour obtenir le produit utilisé, nous avons d'abord préparé $K[PtCl_3(CH_3)_2SO]$ selon la méthode indiquée par Kukushkin, Vyazmenskii & Zorina (1968) et reprise par Melanson, Hubert & Rochon (1976). Le trichloro(diméthylsulfoxyde)platinate(II) de potassium, pris à l'état cristallisé, est ensuite ajouté en quantité stoechiométrique à une solution aqueuse de chlorure de tétraammineplatine(II). Il se forme des cristaux orangés que l'on sépare par filtration, lave à l'éthanol et sèche à l'air. La détermination de leurs teneurs en platine, carbone, azote et hydrogène a donné des résultats en bon accord avec la formule indiquée ci-dessus.

Partie expérimentale. Cristal prismatique: 0,12 × 0,15 × 0,35 mm. Dimensions de la maille déterminées sur monocristal avec 25 réflexions telles que $5{,}65 \le \theta \le 17{,}75°$. Diffractomètre Enraf–Nonius CAD-4. $0{,}049 \le (\sin\theta)/\lambda \le 0{,}661$ Å^{-1}; $-28 \le h \le 26$; $0 \le k \le 19$; $0 \le l \le 19$. Balayage $\theta/2\theta$ d'amplitude $s(°) = 0{,}90 + 0{,}35\,\mathrm{tg}\theta$. Réflexions de contrôle: 14,0,0, $\bar{4}\bar{2}\bar{4}$ et $\bar{5}\bar{1}0$. $\sigma(I)/I$ (réflexions de contrôle): 0,0025. 2625 réflexions indépendantes mesurées, 463 réflexions inobservées [$I \le 3\sigma(I)$]. Méthode de l'atome lourd et calcul de la densité électronique. H non localisés. Corrections d'absorption selon Walker & Stuart (1983). Affinement basé sur les F. Facteurs de diffusion des *International Tables for X-ray Crystallography* (1974). Paramètres affinés: x, y, z et β_{ij} de Pt, Cl, S, O, N et C. $wR = 0{,}052$, $w = 1/\sigma^2(F)$, $S = 1{,}99$, $(\Delta/\sigma)_{\max} = 0{,}05$, $|\Delta\rho|_{\max} = 1{,}6$ (4) e Å^{-3}. Programmes de calcul du système *SDP* (B. A. Frenz & Associates, Inc., 1982). Ordinateur PDP 11/44.

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