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Structure of 4-Methyl-1,2,6-tristibatricyclo[2.2.1.0^{2,6}]heptane, $C_5H_9Sb_3$

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Dedicated to Professor Dr Dr h.c. H. Behrens on the occasion of his 70*th birthday*

Abstract. $M_r = 434{\cdot}4$, monoclinic, $A2/m$, $a = 8{\cdot}574\,(3)$, $b = 9{\cdot}576\,(2)$, $c = 11{\cdot}354\,(3)$ Å, $\beta = 105{\cdot}26\,(2)°$, $V = 899{\cdot}4$ Å^3, $Z = 4$, $D_x = 3{\cdot}20$ g cm^{-3}, $\lambda(\mathrm{Ag}\ K\alpha) = 0{\cdot}55936$ Å, $\mu = 46{\cdot}4$ cm^{-1}, $F(000) = 768$, room temperature. Final $R = 0{\cdot}047$ for 681 unique reflections (unobserveds included). The molecule consists of a three-membered ring of Sb atoms [Sb—Sb 2·796 (3) and 2·817 (3) Å] and an organic group. All bond lengths and angles are in the expected range. In the crystal the molecules are mainly connected by intermolecular Sb···Sb contacts supplemented by van der Waals forces between methyl groups.

Introduction. The title compound is the first all-*cis* cyclic tristibine (Ellermann & Veit, 1982); thus it was necessary to correlate spectroscopic results with an X-ray structure determination.

Differences from the analogous arsenic compound (Thiele, Zoubek, Lindner & Ellermann, 1978), whose structure has already been determined, are important for further investigations.

Experimental. Light-red rhombic plates of crystals grown from tetrahydrofuran; crystal dimensions 0·32 × 0·22 × 0·14 mm; lattice parameters determined by a Guinier photograph, using refinement procedure *GIVER* (Krogmann, 1966); possible space groups: $A2$, Am, $A2/m$; $A2/m$ (non-standard setting of $C2/m$) gave the best agreement in the structure refinement. Intensities collected on a Philips PW 1100 diffractometer, using Ag $K\alpha$ radiation and a graphite monochromator, ω–2θ scans, scan width: $(1{\cdot}20 + 0{\cdot}2\tan\theta)°$, $2 \le 2\theta \le 18°$, 2861 reflections measured; symmetrically equivalent reflections averaged, 681 unique reflections (unobserveds included), index range $h \pm 9$, k 0/10, l 0/12; $R_{\mathrm{int}} = 0{\cdot}016$. Three standard reflections used for control of stability. Spherical absorption correction applied with $\mu R = 0{\cdot}54 \pm 0{\cdot}20$; Sb positions derived by Patterson techniques; a subsequent Fourier map revealed all C atoms, only some of the H atoms could be found in the ΔF map and no H atoms were included; final least-squares refinement on F used corrections for extinction and anomalous dispersion; weights $w = 1/\sigma^2$. Scattering factors taken from *International Tables for X-ray Crystallography* (1974).

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There was no change when using scattering factors for Sb^{3+} and Sb^{5+} instead of Sb^0. Final $R = 0{\cdot}047$, $wR = 0{\cdot}058$. The difference between R_{int} and wR can be explained by an insufficient absorption correction. $(\Delta/\sigma)_{max} = 0{\cdot}03$. Max. residual electron density $0{\cdot}48$ e Å^{-3}, min. residual electron density $-3{\cdot}4$, $-3{\cdot}2$ e Å^{-3} at the Sb atoms, next peak $-0{\cdot}9$ e Å^{-3}. For all computations the *CRYSTAN* system (Burzlaff, Böhme & Gomm, 1977) was used. Diagrams were drawn with *ORTEP*II (Johnson, 1977).

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

E.s.d.'s are given in parentheses.

	x	*y*	*z*	U_{eq}*
Sb(1)	3107 (1)	1471 (1)	728 (1)	639 (7)
Sb(2)	5386 (1)	0	2510 (1)	538 (8)
C(1)	1521 (16)	1310 (12)	1955 (12)	496 (75)
C(2)	3616 (21)	0	3544 (16)	449 (103)
C(3)	1839 (20)	0	2726 (16)	360 (94)
C(4)	700 (23)	0	3586 (18)	607 (124)

* U_{eq} = equivalent isotropic temperature factor, defined as $\frac{1}{3}$(trace of the orthogonalized U_{ij} tensor).

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

E.s.d.'s are given in parentheses.

Sb(1)–Sb(1')	2·817 (3)	C(2)–C(3)	1·56 (4)	
Sb(1)–Sb(2)	2·796 (3)	C(3)–C(4)	1·55 (6)	
Sb(1)–C(1)	2·19 (3)	Sb(1)···Sb(2)	3·966 (2)	I*
Sb(2)–C(2)	2·15 (4)	Sb(1)···Sb(1)	4·012 (3)	II*
C(1)–C(3)	1·51 (3)			

Sb(2)–Sb(1)–Sb(1')	59·74 (4)	Sb(2)–C(2)–C(3)	113·3 (7)
Sb(2)–Sb(1)–C(1)	86·44 (6)	C(1)–C(3)–C(1')	112·0 (8)
Sb(1')–Sb(1)–C(1)	85·97 (6)	C(1)–C(3)–C(2)	110·0 (8)
Sb(1)–Sb(2)–Sb(1')	60·51 (4)	C(1)–C(3)–C(4)	108·4 (8)
Sb(1)–Sb(2)–C(2)	86·26 (6)	C(2)–C(3)–C(4)	107·7 (8)
Sb(1)–C(1)–C(3)	112·2 (7)		

Symmetry code: (') $x, -y, z$.

* For intermolecular distances see Fig. 2.
I denotes equivalent position: $1-x, \frac{1}{2}-y, \frac{1}{2}-z$.
II denotes equivalent position: $1-x, y, -z$.

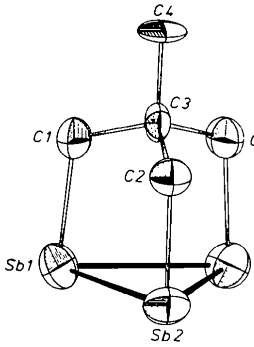

Fig. 1. The $CH_3C(CH_2Sb)_3$ molecule showing atom labelling and 50% probability thermal ellipsoids. Primed atoms are related to the corresponding unprimed ones by the mirror symmetry.

Discussion. Coordinates and equivalent isotropic temperature factors are given in Table 1.* A diagram of the molecule indicating the atomic-numbering scheme is shown in Fig. 1. Bond lengths and angles are in Table 2.

The constitution determined from spectroscopic data (Ellermann & Veit, 1982) could be verified by the X-ray structure analysis. In the free state the molecule is assumed to have symmetry $3m$ (C_{3v}); in the solid state the symmetry is reduced to m (C_s) (Fig. 1 and Table 2).

The three Sb atoms form an almost equilateral triangle, that is extended to a cage by the $CH_3C(CH_2)_3$ group. The Sb—Sb distances [2·796 (3) and 2·817 (3) Å] and Sb—C distances [2·15 (4) and 2·19 (3) Å] are in good agreement with the sum of covalent radii (Sb—Sb: 2·82, Sb—C: 2·18 Å) (Wells, 1975) and with experimental data. The angles involving the CH_2 groups are larger than tetrahedral.

The crystal structure can be described as zigzag chains built up of Sb triangles. The hydrocarbon groups of neighbouring molecules show opposite orientations to each other. The chains are connected such that Sb triangles are in contact with each other (Fig. 2). Thus a sequence of double layers is built up in the third direction. The Sb_3 groups form distorted wavy six-membered rings, which can be found in a regular shape in Sb metal (Barrett, Cucka & Haefner, 1963). The As homologue is almost isotypic (Thiele *et al.*, 1978).

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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 42275 (5 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

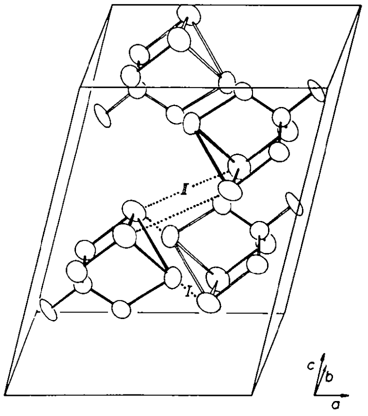

Fig. 2. Molecular packing in the crystal of $CH_3C(CH_2Sb)_3$.

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Redetermination and Absolute Configuration of Sodium Uranyl(VI) Triacetate

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Abstract. Sodium tris(acetato)dioxouranate(1−), $Na[UO_2(C_2H_3O_2)_3]$, $M_r = 470·15$, cubic, $P2_13$, $a = 10·689\,(2)$ Å, $V = 1221·3$ Å^3, $Z = 4$, $D_m = 2·562$, $D_x = 2·557\,(2)$ g cm^{-3}, Mo $K\alpha$, $\lambda(\alpha_1) = 0·70930$ Å, $\mu = 126·5$ cm^{-1}, $F(000) = 848$, $T = 294$ K, $R = 0·021$ for 1195 unique observed reflections. A determination of the anomalous-scattering term f'' for uranium and Mo $K\alpha$ radiation gives 9·7 (2) e per atom. Each uranyl ion [U—O(av.) = 1·758 (3) Å] lies on a threefold axis and is surrounded equatorially by six O atoms of three acetate groups [U—O(av.) = 2·464 (2) Å]. The absolute configuration determined by the anomalous X-ray scattering is correlated with the sign of the optical activity.

Introduction. The structure of sodium uranyl acetate was studied by Fankuchen (1935) and was determined more completely by Zachariasen & Plettinger (1959). We studied it again to obtain more accurate parameters for use in the analysis of experiments with synchrotron radiation (Templeton & Templeton, 1982). The purpose of these experiments was to measure anomalous-scattering terms and the effect of polarization on them. Incidental to this work we determined that the absolute configuration of the structure, if described in a right-handed coordinate system with the atomic parameters listed in Table 1, is that of a crystal for which the sign of the optical activity is negative.

Experimental. Crystals were made by dissolving reagent-grade uranyl acetate and sodium acetate in water, followed by slow evaporation. Crystal with 18 faces of forms {111}, {11$\bar{1}$} and {110}, 0·11 × 0·11 × 0·23 mm. D_m taken from Fankuchen (1935). Picker FACS-I diffractometer, graphite monochromator, θ–2θ scan; cell dimension from 12 reflections $42 < 2\theta < 48°$; analytical absorption correction, range 2·70 to 3·64; max. $(\sin\theta)/\lambda = 0·705$ Å^{-1}, h 0 to 15, k −15 to 15, l −15 to 0; three standard reflections, $\sigma = 1·3$, 1·0, 1·4%, no correction for decay; 3922 data, 1207 unique (including 12 observed less than background), $R_{\rm int}(I) = 0·030$; structure from Zachariasen & Plettinger (1959) refined on F, 62 parameters including f'' of U, anisotropic thermal parameters for all atoms except H, H atoms found in ΔF map and refined with isotropic thermal parameters and subject to restraints on H—H and C—H distances, $R = 0·021$ for 1195 reflections, $wR = 0·015$, $S = 1·01$, $w = [\sigma(F)]^{-2}$, derived from $\sigma^2(F^2) = [\sigma^2(F^2)$, counting statistics only, $+(0·014F^2)^2]$; max. $\Delta/\sigma = 0·10$; max. empirical isotropic correction for extinction 10% of F; max. and min. of ΔF synthesis 0·6 and −0·6 e Å^{-3}; atomic f including dispersion for neutral U, Na, O, C and spherical-bonded H from *International Tables for X-ray Crystallography* (1974); local unpublished programs and *ORTEP* (Johnson, 1965). Optical activity (positive rotation of 2 or 3°) was easy to observe in a well formed crystal of thickness 1·5 mm with a polarizing microscope illuminated with white light. This crystal was too large for reliable observation of the signs of Bijvoet differences. A small fragment was broken from a corner. Its diffraction intensities for several Bijvoet pairs, selected to be sensitive to configuration, were consistently reversed from those

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