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Structure of μ-Oxo-bis[(2-hydroxyethanesulfonato)triphenylantimony(V)], $[Sb_2O(C_2H_5O_4S)_2(C_6H_5)_6]$

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Abstract. $M_r = 972{\cdot}39$, monoclinic, $P2_1/c$, $a = 10{\cdot}861$ (4), $b = 19{\cdot}916$ (6), $c = 9{\cdot}075$ (4) Å, $\beta = 94{\cdot}15$ (5)°, $U = 1958$ Å^3, $Z = 2$, $D_x = 1{\cdot}649$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 1{\cdot}56$ mm^{-1}, $F(000) = 972$, $T = 294$ (1) K. Final $R = 0{\cdot}032$ for 2727 unique observed X-ray diffractometer data. Two only slightly distorted trigonal-bipyramidal units (μ-O)—SbPh$_3$—O—S(O$_2$)C$_2$H$_4$OH with O atoms in apical and C(phenyl) atoms in equatorial positions are linked *via* a linear Sb—O—Sb bridge. The Sb—μ-O bond of 1·936 (1) Å is rather short. The Sb—O bond to the unidentately coordinated sulfonate group [2·276 (4) Å] appears to have appreciably high ionic character.

Introduction. The chemistry of triorgano-Group V element sulfonates is rather unexplored and structures of such compounds are unknown. We recently obtained for the first time, by reaction of Ph_3SbO (Ph = C_6H_5) with sulfonic acids in different solvents, antimony compounds which correspond to the formula $(Ph_3SbO_3SR)_2O$ (HO_3SR = sulfonic acid) having, according to spectroscopic data, an Sb—O—Sb bridge. Compounds of the type $(R_3SbX)_2O$ (R = organogroup, X = anionic ligand) may have a bent Sb—O—Sb bridge, as in $(Ph_3SbN_3)_2O$ (Sb—O—Sb:139·8°, Ferguson & Ridley, 1973), or a linear bridge as in $(Ph_3SbOOC_4H_9)_2O$ (Starikova, Shchegoleva, Trunov & Pokrovskaya, 1978). To obtain more facts about influences which determine the type of bridge in distiboxane and also to learn more about the coordination of the sulfonate group with organoelement moieties (a field which has also been neglected) we started to determine the crystal and molecular structure of organoantimony sulfonates and report here the structure of $(Ph_3SbO_3SCH_2CH_2OH)_2O$.

Experimental. $(Ph_3SbO_3SCH_2CH_2OH)_2O$ crystallizes from a solution of 10 mmol isethionic acid and 10 mmol Ph_3SbO in 250 ml acetone after standing for 1 d. Crystal size 0·32 × 0·29 × 0·10 mm, $\omega/2\theta$ scan, scan speed 0·04–0·01° s^{-1} in θ, Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation; lattice parameters from least-squares fit with 25 reflexions in range $9{\cdot}3 \le \theta \le 14{\cdot}0°$; three standard reflexions recorded every 4 h, only random deviations; 3799 reflexions, $1 \le \theta \le 25°$, $-10 \le h \le 10$, $0 \le k \le 23$, $0 \le l \le 12$; after averaging 3541 unique reflexions, 2727 with $I > 3\sigma(I)$, Lorentz–polarization correction and absorption correction *via* ψ scans; systematic absences: $(h0l)$ $l = 2n+1$, $(0k0)$ $k = 2n+1$, space group $P2_1/c$; structure solution *via* Patterson function, ΔF syntheses and full-matrix least-squares refinements with anisotropic temperature factors for all non-H atoms; complex neutral-atom scattering factors from Cromer (1974) and Cromer & Waber (1974). H atoms placed in geometrically calculated positions (C—H 1·08 Å) and not included in structure-factor calculation. Least-squares refinement based on F and 241 refined parameters, $R = 0{\cdot}032$, unit weights; max. Δ/σ for all parameters 0·02, av. $< 0{\cdot}00$; no significant features in final ΔF synthesis; programs: Enraf–Nonius *SDP* (1978), *ORTEP* (Johnson, 1976) and *POP*1 (van de Waal, 1976).

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Discussion. The structure of the title compound is shown in Fig. 1 and in a stereoview in Fig. 2. Positional parameters and equivalent values of the anisotropic temperature factors are given in Table 1,[*] bond lengths and angles in Table 2.

The unit cell contains two centrosymmetrical molecules with a linear Sb—O—Sb bridge as the central part. The O atom of this bridge resides on a center of symmetry. The atoms bound to Sb form a slightly distorted trigonal bipyramid with the three C(phenyl) atoms in the equatorial plane and the two O atoms in the apical positions. Through the center of symmetry the phenyl rings at the Sb atoms are in staggered positions. The dihedral angles between the least-squares planes through the phenyl groups and the plane perpendicular to the Sb(1)—O(5) direction are 4·5 (5), 118·2 (5) and 38·7 (5)°. The sulfonate group is unidentately coordinated. From a comparison of the bond lengths with those in sulfonic acid esters [S—O:1·602 (8), S=O:1·431 (9), 1·426 (8) Å, Wieczorek, Bokii & Struchkov, 1975; S—O:1·589 (2), S=O:1·431 (9), 1·426 (8) Å, Williams, Ando, Bloor & Hursthouse, 1979] it can be inferred that the Sb—O(1) bond has an appreciably high ionic character. This corresponds (i) with the fact that the Sb(1)—O(1) bond is about 0·24 (1) Å longer than in other comparable Sb compounds (Shen, McEwen, La Placa, Hamilton &

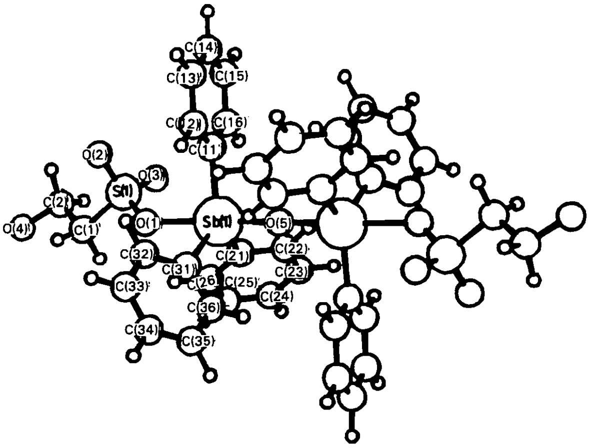

Fig. 1. General view of the molecule, showing atom numbering.

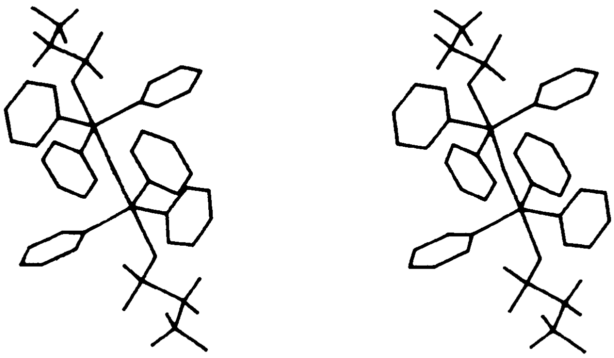

Fig. 2. Stereoscopic view of the molecule.

Table 1. *Fractional atomic coordinates and equivalent isotropic B values* (Å^2)

$$B_{eq} = \tfrac{4}{3}\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Sb(1)	0·14258 (3)	0·04966 (2)	0·08059 (4)	2·43 (1)
S(1)	0·3902 (1)	0·15667 (7)	0·1339 (2)	3·08 (3)
O(1)	0·3153 (3)	0·1001 (2)	0·1859 (4)	3·41 (8)
O(2)	0·4909 (4)	0·1320 (2)	0·0546 (5)	4·5 (1)
O(3)	0·3149 (4)	0·2065 (2)	0·0577 (5)	4·5 (1)
O(4)	0·6079 (4)	0·2726 (2)	0·3941 (5)	4·5 (1)
O(5)	0·000	0·000	0·000	4·0 (1)
C(1)	0·4563 (5)	0·1926 (3)	0·3022 (6)	3·6 (1)
C(2)	0·5321 (6)	0·2538 (4)	0·2656 (7)	4·9 (1)
C(11)	0·2255 (5)	0·0556 (3)	−0·1205 (6)	3·1 (1)
C(12)	0·2985 (7)	0·0025 (4)	−0·1632 (8)	5·0 (2)
C(13)	0·3479 (8)	0·0070 (4)	−0·3016 (9)	6·7 (2)
C(14)	0·3223 (8)	0·0619 (5)	−0·3926 (8)	6·6 (2)
C(15)	0·2521 (7)	0·1155 (4)	−0·3472 (8)	6·0 (2)
C(16)	0·2014 (6)	0·1124 (4)	−0·2075 (7)	4·5 (1)
C(21)	0·0406 (5)	0·1312 (3)	0·1545 (6)	2·7 (1)
C(22)	−0·0729 (5)	0·1444 (3)	0·0825 (7)	3·7 (1)
C(23)	−0·1451 (6)	0·1975 (4)	0·1337 (8)	4·8 (2)
C(24)	−0·1016 (7)	0·2338 (3)	0·2566 (7)	4·7 (1)
C(25)	0·0119 (6)	0·2197 (3)	0·3290 (7)	4·3 (1)
C(26)	0·0862 (6)	0·1685 (3)	0·2775 (6)	3·5 (1)
C(31)	0·1932 (5)	−0·0233 (3)	0·2398 (6)	2·9 (1)
C(32)	0·3120 (6)	−0·0500 (3)	0·2553 (7)	4·1 (1)
C(33)	0·3354 (7)	−0·1007 (3)	0·3605 (8)	5·5 (2)
C(34)	0·2449 (8)	−0·1228 (4)	0·4461 (8)	5·9 (2)
C(35)	0·1276 (8)	−0·0955 (4)	0·4298 (8)	6·2 (2)
C(36)	0·1004 (6)	−0·0448 (4)	0·3251 (7)	4·5 (1)

Table 2. *Bond lengths* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

Sb(1)—C(11)	2·096 (5)	S(1)—O(2)	1·438 (4)
Sb(1)—C(21)	2·103 (5)	S(1)—O(3)	1·431 (4)
Sb(1)—C(31)	2·095 (5)	S(1)—C(1)	1·789 (6)
Sb(1)—O(1)	2·276 (4)	C(1)—C(2)	1·522 (9)
Sb(1)—O(5)	1·936 (1)	C(2)—O(4)	1·428 (8)
S(1)—O(1)	1·487 (4)		
C(11)—Sb(1)—C(21)	120·2 (2)	O(1)—S(1)—C(1)	103·1 (3)
C(11)—Sb(1)—C(31)	122·2 (2)	O(2)—S(1)—O(3)	115·1 (3)
C(11)—Sb(1)—O(1)	87·0 (2)	O(2)—S(1)—C(1)	106·9 (3)
C(11)—Sb(1)—O(5)	94·7 (1)	O(3)—S(1)—C(1)	108·4 (3)
C(21)—Sb(1)—C(31)	115·7 (2)	Sb(1)—C(11)—C(12)	119·2 (4)
C(21)—Sb(1)—O(1)	87·8 (2)	Sb(1)—C(11)—C(16)	117·7 (4)
C(21)—Sb(1)—O(5)	95·2 (1)	Sb(1)—C(21)—C(22)	118·0 (4)
C(31)—Sb(1)—O(1)	81·3 (2)	Sb(1)—C(21)—C(26)	120·0 (4)
C(31)—Sb(1)—O(5)	93·9 (1)	Sb(1)—C(31)—C(32)	122·0 (4)
O(1)—Sb(1)—O(5)	175·2 (1)	Sb(1)—C(31)—C(36)	115·8 (4)
Sb(1)—O(1)—S(1)	130·9 (2)	S(1)—C(1)—C(2)	108·9 (4)
O(1)—S(1)—O(2)	110·7 (2)	C(1)—C(2)—O(4)	108·7 (5)
O(1)—S(1)—O(3)	111·8 (2)	Sb(1)—O(5)—Sb(1′)	180

The C—C bond lengths and C—C—C angles in the phenyl groups range from 1·37 (1) to 1·420 (9) Å |mean: 1·39 (1) Å| and from 117·6 (6) to 123·1 (5)° |mean: 120·0 (7)°| respectively.

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[*] Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39901 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Wolf, 1968) and (ii) that the angle O(2)—S—O(3) of 115·1 (3)° is smaller than the corresponding angle in sulfonic acid esters [120·0 (1)°, Wieczorek *et al.*, 1975; 119·2 (5)°, Williams *et al.*, 1979] but larger than in ionic sulfonates [112·2 (8)°, Couldwell, Prout, Robey, Taylor & Rossotti, 1978].

H of the isethionate hydroxy group, which is clearly represented by ν(OH) at 3380 cm^{-1} in the IR spectrum (same position as in sodium isethionate), was not localized. A short intermolecular O(4)···O(2)(x, $\frac{1}{2}-y$, $\frac{1}{2}+z$) distance of 2·760 (6) Å could be correlated with a hydrogen bridge between these atoms. The existence of a hydrogen bridge is also indicated by the position and broadness of ν(OH).

A linear Sb—O—Sb bridge between pentacoordinated Sb atoms was also found in μ-oxo-bis(*tert*-butylperoxotriphenylantimony) (Starikova *et al.*, 1978) and in μ-oxo-bis[tris(*p*-chlorophenyl)(1,1,1-trifluoro-2,4-pentanedionato-*O,O′*)antimony(V)]–chloroform (1/2), in which Sb, however, is hexacoordinated (Ebina, Ouchi, Yoshino, Sato & Saito, 1978). The Sb—μ-O bond lengths in these compounds are comparable [this work: 1·936 (1) Å; $(\text{Ph}_3\text{SbOO-}tert\text{-C}_4\text{H}_9)_2\text{O}$: 1·974 (2) Å; $[(p\text{-ClC}_6\text{H}_4)_3\text{SbCF}_3\text{C(O)CH(CO)CH}_3]_2$ $0{\cdot}2\text{CHCl}_3$: 1·943 Å, $\sigma < 0{\cdot}0004$ Å].

The interesting question of whether a bent or a linear Sb—O—Sb bridge should be expected in a certain distiboxane still cannot be answered. Deviations of the Sb—O—Sb angle in distiboxanes from the tetrahedral angle have been correlated with intramolecular repulsions between the two halves of the specific molecules (Ferguson & Ridley, 1973). For linear bridges interaction of the electron pairs of O with empty *d* orbitals of Sb was discussed (Starikova *et al.*, 1978; see also March & Ferguson, 1975). This interpretation would be consistent with the observation of rather short Sb—μ-O bonds in the compounds with linear Sb—O—Sb bridges.

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Structure of Acetato(acetylacetonato)diaquacalcium, $[\text{Ca(CH}_3\text{COO)(C}_5\text{H}_7\text{O}_2\text{)(H}_2\text{O)}_2]$

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Abstract. $M_r = 234{\cdot}27$, $P\bar{1}$, $a = 6{\cdot}524$ (1), $b = 6{\cdot}851$ (1), $c = 12{\cdot}504$ (1) Å, $\alpha = 87{\cdot}70$ (1), $\beta = 81{\cdot}42$ (1), $\gamma = 71{\cdot}21$ (1)°, $Z = 2$, $V = 523{\cdot}2$ (1) Å^3, D_m(298 K) = 1·48 g cm^{-3}, D_x(140 K) = 1·49 g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 5{\cdot}74$ cm^{-1}, $T = 140$ K, $F(000) = 208$, 1953 unique observed reflections, $R = 0{\cdot}031$. A distorted dodecahedral arrangement of eight O atoms surrounds each Ca^{2+} ion. The ligation (average Ca—O distances in parentheses) consists of one chelating acetylacetonate (2·390 Å), two waters (2·498 Å), two monodentate acetates (2·349 Å) and one bidentate acetate (2·563 Å) which provides a cross-link to a centrosymmetrically related chain.

Introduction. The crystal structure determination of the title compound was undertaken to check the stoichiometry of the crystals, which were known to contain acetylacetonate yet differ from the previously determined structure of $[\text{Ca(acetylacetonate)}_2(\text{H}_2\text{O})_2]\text{.H}_2\text{O}$ (Sahbari & Olmstead, 1983). The phosphorescence and

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