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The Crystal and Molecular Structure of *O*-Ethylxanthato-bis(quinolin-8-olato)-antimony(III) and a Redetermination for Tris(*O*-ethylxanthato)antimony(III)

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Abstract

The crystal structures of the title compounds $Sb(C_9H_6NO)_2(S_2COC_2H_5)$ (1) and $Sb(S_2COC_2H_5)_3$ (2) have been determined by three dimensional X-ray diffraction techniques and refined by a least squares method; final R 0.049 for 2911 reflections $[I \geqslant 3\sigma(I)]$ for (1) and R 0.047, R_w 0.046 for 846 reflections $[I \geqslant 2\sigma(I)]$ for (2). Crystals of (1) are triclinic, space group $P1$, a = 10.825(2), b = 11.131(2), c = 8.911(1) Å, α = 109.45(1), β = 95.92(1) and γ = 93.02(1)° with Z = 2. Crystals of (2) are rhombohedral, space group $R\bar{3}$, a_{rhomb} = 10.138(3) Å and α = 103.43(2)°. The environment of the Sb atom in (1) is based on a pentagonal bipyramidal geometry consisting of the six donor atoms of the three chelating ligands and a stereochemically active lone-pair of electrons which occupies the remaining axial position. The xanthate ligand chelates the Sb atom almost symmetrically with two long Sb–S bonds of 3.059(2) and 3.171(2) Å. In contrast the xanthate ligands in (2) chelate the Sb atom with asymmetric Sb–S bonds of 2.511(2) and 3.002(3) Å.

Introduction

The reaction of $In(exa)_3$, $Pb(exa)_2$ and $Sn(exa)_4$ [exa = *O*-ethylxanthato] with oxine (8-hydroxyquinoline), hereafter abbreviated as oxinH, results in the substitution of one or more of the xanthate ligands by the oxin ligand to yield the following complexes: $In(exa)_2(oxin)$ [1], Pb(exa)(oxin) [2], and $Sn(exa)_2(oxin)_2$ [3] respectively. However, pure products could not be isolated from similar reactions involving Group V [1]. We have now isolated from the reaction products of $Sb(exa)_2Br$ [4] and the potassium salt, Koxin, the crystalline compound $Sb(exa)(oxin)_2$ [not the expected $Sb(exa)_2(oxin)$] and this has been characterized by single crystal structure analysis.

The structure of $Sb(exa)_3$ determined in 1961 [5] based on photographic data indicates dimensions, in particular those concerning the S_2CO moiety, which do not appear entirely consistent with recently determined Group V xanthates. We have redetermined this structure to enable a more satisfactory comparison with that of $Sb(exa)(oxin)_2$.

Experimental

Preparation

Sb(exa)(oxin)₂

To a stirred benzene solution of $Sb(exa)_2Br$ [4] (2.0 g, 50 ml) was added an aqueous solution of one mol equivalent potassium oxinate. (The potassium salt was prepared by stirring equimolar proportions of oxinH and potassium hydroxide in methanol, the solution was filtered and the resultant solid product was dissolved in water.) Stirring was continued for two hours, after which the benzene layer was separated and dried over anhydrous Na_2CO_3. The solvent was removed under reduced pressure and the resultant oil was taken up in dichloromethane and allowed to stand at 4 °C until a yellow solid was deposited. Characteristic absorptions were observed in the C–O and C–S regions of the infrared spectrum at 1270, 1240 and 1032 cm^{-1} respectively. Absorptions due to the coordinated oxin ligands occur at 1103(s), 822(m), 787(m), 752(s), 742(s), 719(s), 518(m), and 500(m) cm^{-1}.

Sb(exa)₃

$Sb(exa)_3$ was prepared as previously reported [5].

Crystallography

Sb(exa)(oxin)₂

A crystal suitable for X-ray analysis was obtained by the slow evaporation of a dichloromethane solution. Preliminary photographic work indicated that the only symmetry present, $\bar{1}$, was that required by Friedel's law and successful structure analysis was

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achieved in the $P\bar{1}$ space group. Accurate cell parameters and an orientation matrix were obtained by a least squares procedure on the setting angles of 25 carefully centred reflections. Intensity data were collected on an Enraf-Nonius four circle CAD-4F diffractometer fitted with CuK$\bar{\alpha}$ (nickel filtered) radiation. The ω:2θ scan technique was used to measure the intensities of 4958 reflections for which $1° \leqslant \theta \leqslant 75°$. Using the $I \geqslant 3\sigma(I)$ criterion of observability on 4129 unique reflections there were 2911 reflections remaining which were used in the subsequent solution and refinement of the structure. No decomposition of the crystal occurred during the data collection. Correction was made for Lorentz and polarization effects and for absorption [6a] (max. and min. transmission factors 0.4753 and 0.2588).

Crystal Data

Sb(exa)(oxin)$_2$, $C_{21}H_{17}N_2O_3S_2Sb$, M = 531.3, Triclinic, $P\bar{1}$, C_i^1, No. 2), a = 10.825(2), b = 11.131(2), c = 8.911(1) Å, α = 109.45(1), β = 95.92(1) and γ = 93.02(1)°, U = 1002.7 Å^3, D_{calc} = 1.760 Mg m^{-3}, Z = 2, $F(000)$ = 528, CuK$\bar{\alpha}$ (nickel filtered) λ = 1.5418 Å, μ = 13.23 mm^{-1}, no. parameters/no. variables = 11.

The position of the Sb atom was determined from a three-dimensional Patterson synthesis. All non-hydrogen atoms were located in subsequent difference maps. The structure was refined using a full-matrix least squares method in which the function $\Sigma w\Delta^2$ was minimized, where w was the weight applied to each reflection and $\Delta = ||F_o| - |F_c||$. Refinement with individual isotropic (R 0.075) and then with anisotropic thermal parameters converged with R 0.047, where $R = \Sigma\Delta/\Sigma F$. Hydrogen atoms were not included in the model and since analysis of variance showed no special features unit weights were employed throughout refinement. A final difference map revealed a maximum residual electron density peak of approximately 1.0 eÅ^{-3} in the vicinity of the Sb atom.

Sb(exa)$_3$

Suitable crystals for X-ray analysis were grown by the slow evaporation of a benzene/hexane solution of the compound. Preliminary photographic and diffractometer studies confirmed that Sb(exa)$_3$ crystallizes in the $R\bar{3}$ space group as previously reported [5].

Intensity data for 2404 reflections were collected as for Sb(exa)(oxin)$_2$ with CuK$\bar{\alpha}$ radiation. The $I \geqslant 2\sigma(I)$ criterion of observability on 1299 unique reflections yielded 846 reflections which were used in the subsequent refinement. No significant decomposition of the crystal occurred during the data collection and correction was made for Lorentz and polarization effects and for absorption [6a].

Crystal Data

Sb(exa)$_3$, $C_9H_{15}O_3S_6Sb$, M = 485.3, Trigonal, $R\bar{3}$ (C_{3i}^2, No. 148), a_{rhomb} = 10.138(3) Å, α = 103.43(2)°, U = 939.6, D_{calc} = 1.715 Mg m^{-3}, Z = 2, $F(000)$ = 528, CuK$\bar{\alpha}$ (nickel filtered), λ = 1.5418 Å, μ = 18.02 mm^{-1}, no. parameters/no. variables = 15.

The Patterson synthesis indicated the position of the Sb atom to be at a site of symmetry 3, and subsequent difference maps enabled the location of all other non-hydrogen atoms comprising the xanthate moiety. The structure was refined as for Sb(exa)(oxin)$_2$ with the use of anisotropic thermal parameters for all non-hydrogen atoms. Hydrogen atoms were included in the model at their calculated positions and the weighting scheme, $w = [\sigma^2(F) + 0.0005|F_o|^2]^{-1}$ was applied. At convergence the values of R and R_w were 0.047 and 0.046 respectively, where $R_w = [\Sigma w\Delta^2/\Sigma wF^2]$. A final difference map revealed a maximum residual electron density peak of less than 0.5 eÅ^{-3}.

The scattering factors for H, C, N, O and S were those collected by Sheldrick [7] while the scattering factors for neutral Sb were those listed in ref. 6b, the values being corrected for anomalous dispersion [6c]. Structure determination and refinement were performed using the SHELX-76 program system [7] on the University of Melbourne's CYBER 170-730 computer system.

Fractional atomic coordinates for Sb(exa)(oxin)$_2$ and Sb(exa)$_3$ are given in Tables I and II respectively and the numbering schemes used are shown in Figs. 1 and 3. Interatomic parameters for Sb(exa)(oxin)$_2$ are listed in Tables III and IV while those for Sb(exa)$_3$ are given in Table V. A listing of the anisotropic thermal parameters and of the observed and calculated structure factors have been deposited with the Editor.

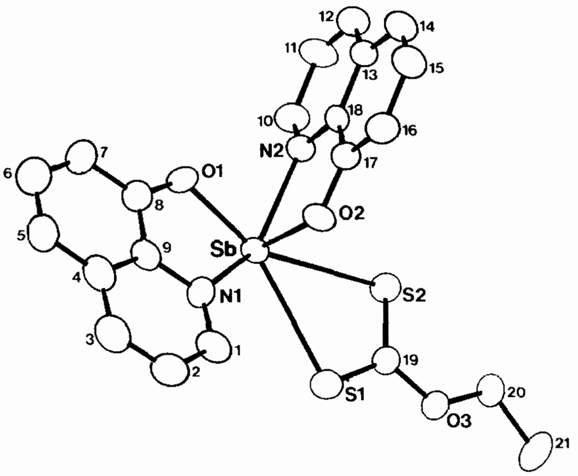

Fig. 1. The numbering scheme used for Sb(exa)(oxin)$_2$. The atoms N(1), O(1), N(2), S(1), and S(2) approximate a pentagonal plane and O(2) occupies an axial position. Atoms otherwise not indicated are carbons.

TABLE I. Fractional Atomic Coordinates for Sb(exa)(oxin)$_2$ (Estimated Standard Deviations in Parentheses in this and all Subsequent Tables).

Atom	x/a	y/b	z/c
Sb	0.1277(1)	0.0300(1)	0.1733(1)
O(1)	0.2741(5)	0.1771(5)	0.2401(7)
N(1)	0.0787(6)	0.1911(6)	0.4046(8)
C(1)	−0.0137(8)	0.1929(8)	0.4929(11)
C(2)	−0.0383(9)	0.3047(9)	0.6119(12)
C(3)	0.0380(9)	0.4149(9)	0.6459(12)
C(4)	0.1426(9)	0.4142(8)	0.5586(12)
C(5)	0.2267(9)	0.5224(8)	0.5854(13)
C(6)	0.3241(9)	0.5132(9)	0.4948(13)
C(7)	0.3417(8)	0.3947(9)	0.3751(13)
C(8)	0.2595(8)	0.2876(8)	0.3462(12)
C(9)	0.1588(7)	0.2975(7)	0.4381(10)
O(2)	0.1970(5)	−0.0384(5)	0.3446(7)
N(2)	0.3001(6)	−0.0808(6)	0.0752(8)
C(10)	0.3425(8)	−0.1029(9)	−0.0631(12)
C(11)	0.4562(9)	−0.1609(9)	−0.0936(13)
C(12)	0.5230(8)	−0.1911(8)	0.0252(13)
C(13)	0.4770(7)	−0.1703(7)	0.1726(11)
C(14)	0.5353(8)	−0.2015(8)	0.3047(13)
C(15)	0.4819(9)	−0.1786(9)	0.4410(13)
C(16)	0.3648(8)	−0.1233(9)	0.4602(11)
C(17)	0.3070(7)	−0.0914(7)	0.3342(9)
C(18)	0.3615(7)	−0.1143(7)	0.1922(10)
S(1)	−0.1261(2)	−0.0849(2)	0.2084(3)
S(2)	0.0219(2)	−0.2565(2)	−0.0253(3)
C(19)	−0.1019(7)	−0.2288(7)	0.0770(10)
O(3)	−0.1923(5)	−0.3209(5)	0.0595(8)
C(20)	−0.1828(9)	−0.4503(8)	−0.0474(13)
C(21)	−0.3012(10)	−0.5293(10)	−0.0385(17)

TABLE II. Fractional Atomic Coordinates for Sb(exa)$_3$.

Atom	x/a	y/b	z/c
Sb	0.1500(1)	0.1500(1)	0.1500(1)
S(1)	0.1200(2)	0.3241(2)	0.3532(2)
S(2)	0.0238(2)	0.2954(3)	−0.0507(3)
C(1)	0.1962(8)	0.3773(8)	−0.0088(8)
O(1)	0.2537(6)	0.4671(6)	−0.0700(5)
C(2)	0.1623(11)	0.4957(10)	−0.1905(10)
C(3)	0.2561(12)	0.5802(10)	−0.2532(10)

TABLE III. Interatomic Distances (Å) for Sb(exa)(oxin)$_2$.

Atoms	Distance	Atoms	Distance
Sb–S(1)	3.059(2)	S(1)–C(19)	1.692(8)
Sb–S(2)	3.171(2)	S(2)–C(19)	1.682(8)
Sb–O(1)	2.105(5)	C(19)–O(3)	1.338(9)
Sb–N(1)	2.368(7)	O(3)–C(20)	1.454(11)
Sb–O(2)	2.018(6)	C(20)–C(21)	1.540(14)
Sb–N(2)	2.373(7)	S(1)···S(2)	2.986(3)
N(1)–C(1)	1.332(11)	N(2)–C(10)	1.310(12)
C(1)–C(2)	1.399(13)	C(10)–C(11)	1.429(13)
C(2)–C(3)	1.369(13)	C(11)–C(12)	1.364(14)
C(3)–C(4)	1.437(14)	C(12)–C(13)	1.404(14)
C(4)–C(5)	1.409(12)	C(13)–C(14)	1.431(13)
C(5)–C(6)	1.381(14)	C(14)–C(15)	1.352(15)
C(6)–C(7)	1.430(14)	C(15)–C(16)	1.439(13)
C(7)–C(8)	1.384(12)	C(16)–C(17)	1.386(12)
C(8)–C(9)	1.417(12)	C(17)–C(18)	1.403(11)
C(9)–N(1)	1.359(10)	C(18)–N(2)	1.345(10)
C(9)–C(4)	1.416(12)	C(18)–C(13)	1.427(11)
C(8)–O(1)	1.307(11)	C(17)–O(2)	1.355(9)
N(1)···O(1)	2.678(9)	N(2)···O(2)	2.662(9)

Results and Discussion

Sb(exa)(oxin)$_2$

The central metal atom is chelated by the two oxinate ligands and the xanthate ligand, as is shown in Fig. 1, with each of the chelating moieties being essentially planar. The planes defined by the xanthate ligand and the oxine ligand oxin (**1**), are almost coplanar (dihedral angle 4.7°) and both of the planes are almost perpendicular to the plane defined by oxin (2), both dihedral angles being 88.3°. The Sb environment is best described as a derivative of the pentagonal bipyramid geometry with the atoms S1, S2, O1, N1, and N2 constituting an approximate pentagonal plane; deviations from the plane of best fit: S1, −0.007(2), S2 0.029(2), O1 0.294(6), N1 −0.193(7) and N2 −0.400(7) Å. The O2 atom and a stereochemically active lone pair of electrons occupy the axial positions. The Sb atom lies 0.233(1) Å out of the equatorial plane in the direction of the lone pair. The overall geometry is similar to those observed in the $[Sb(C_2O_4)_3]^{3-}$ anions of $K_3[Sb(C_2O_4)_3]$ and $(NH_4)_3[Sb(C_2O_4)_3]$ [8], in $Sb[S_2P(C_6H_5)_2]_3$ [9] and $Sb(S_2COC_2H_5)_3\cdot 0.5C_{10}H_8N_2$, [10]. The latter has been described as adopting 'stereochemistry C', i.e. intermediate between the two limiting geometries pentagonal bipyramidal and capped trigonal prismatic [10].

The asymmetric unit consists of molecules of Sb(exa)(oxin)$_2$, two of which comprise the unit cell contents, as is shown in Fig. 2. There are significant intermolecular interactions involving the Sb, S and C atoms, Table VI. The close approach of the two molecules is surprising since the repulsive effect of the lone pair of electrons on the Sb atom, which is implied by the arrangement of the donor atoms in the form of a pentagonal pyramid, would be expected to preclude the close approach of another molecule. The geometry of these intermolecular contacts is, however, such that the repulsive effect of the lone pair of electrons is substantially reduced. It is suggested that the tetragonal pyramid formed by the

TABLE IV. Bond Angles (°) for Sb(exa)(oxin)$_2$.

Atoms	Angle	Atoms	Angle
S(1)–Sb–S(2)	57.24(6)	O(1)–Sb–N(1)	73.3(2)
S(1)–Sb–O(1)	152.7(2)	N(1)–Sb–O(2)	80.1(2)
S(1)–Sb–N(1)	79.5(2)	N(1)–Sb–N(2)	139.8(2)
S(1)–Sb–O(2)	85.7(2)	O(2)–Sb–N(2)	74.2(2)
S(1)–Sb–N(2)	127.6(2)	Sb–S(1)–C(19)	90.9(3)
S(2)–Sb–O(1)	149.8(2)	Sb–S(2)–C(19)	87.3(3)
S(2)–Sb–N(1)	135.4(2)	S(1)–C(19)–S(2)	124.4(5)
S(2)–Sb–O(2)	86.1(2)	S(1)–C(19)–O(3)	113.5(6)
S(2)–Sb–N(2)	73.3(2)	S(2)–C(19)–O(3)	122.1(6)
O(1)–Sb–N(1)	73.3(2)	C(19)–O(3)–C(20)	119.6(7)
O(1)–Sb–O(2)	91.7(2)	O(3)–C(20)–C(21)	105.5(8)
Sb–N(1)–C(1)	130.7(5)	Sb–N(2)–C(10)	129.9(6)
Sb–N(1)–C(9)	109.4(5)	Sb–N(2)–C(18)	109.2(5)
N(1)–C(1)–C(2)	122.1(8)	N(2)–C(10)–C(11)	121.3(9)
N(1)–C(9)–C(4)	121.9(7)	N(2)–C(18)–C(13)	121.4(7)
C(1)–C(2)–C(3)	119.8(9)	C(10)–C(11)–C(12)	119.1(9)
C(2)–C(3)–C(4)	119.4(9)	C(11)–C(12)–C(13)	120.0(8)
C(3)–C(4)–C(5)	123.7(9)	C(12)–C(13)–C(14)	125.7(7)
C(3)–C(4)–C(9)	116.9(8)	C(12)–C(13)–C(18)	117.3(8)
C(4)–C(5)–C(6)	119.7(9)	C(13)–C(14)–C(15)	120.8(8)
C(5)–C(4)–C(9)	119.4(9)	C(14)–C(13)–C(18)	117.0(8)
C(5)–C(6)–C(7)	120.9(8)	C(14)–C(15)–C(16)	122.3(9)
C(6)–C(7)–C(8)	120.3(9)	C(15)–C(16)–C(17)	118.0(8)
C(7)–C(8)–O(1)	122.0(8)	C(16)–C(17)–O(2)	121.1(7)
C(7)–C(8)–C(9)	118.8(8)	C(16)–C(17)–C(18)	120.3(7)
O(1)–C(8)–C(9)	119.2(7)	O(2)–C(17)–C(18)	118.6(7)

TABLE V. Bond Distances (Å) and Bond Angles (°) for Sb(exa)$_3$.

Atoms	Distance	Atoms	Angle
Sb–S(1)	2.511(2)	Sb–S(1)–C(1)	92.9(3)
Sb–S(2)	3.002(3)	Sb–S(2)–C(1)	78.1(3)
S(1)–C(1)	1.719(8)	S(1)–C(1)–S(2)	123.8(5)
S(2)–C(1)	1.656(8)	S(1)–C(1)–O(1)	111.2(5)
C(1)–O(1)	1.318(9)	S(2)–C(1)–O(1)	125.1(6)
O(1)–C(2)	1.483(11)	C(1)–O(1)–C(2)	119.8(7)
C(2)–C(3)	1.482(14)	O(1)–C(2)–C(3)	108.1(8)

Atoms	Angle
S(1)–Sb–S(2)	64.65(6)
S(1)–Sb–S(1)^{I*}	88.48(7)
S(1)–Sb–S(2)^{I*}	153.11(8)
S(2)–Sb–S(1)^{I*}	89.81(7)
S(2)–Sb–S(2)^{I*}	113.54(8)

* Where 1 refers to the symmetry relation z, x, y.

S1, S2, C19 and Sb′ atoms, [where Sb′ is related by the symmetry operation $(\bar{x}, \bar{y}, \bar{z})$] encapsulates the lone pair of electrons, as is highlighted in Fig. 2, and therefore dimishes the repulsive effects between the molecules. This encapsulation of the lone pair of electrons would also provide an explanation for the lenghtening of the Sb–S bonds.

Sb(exa)$_3$

Sb(exa)$_3$ is isomorphous with the As analogue, As(exa)$_3$, [5, 11], Fig. 3. The xanthate ligands in Sb(exa)$_3$ chelate the metal centre with inequivalent Sb–S bond lengths, 2.511(2) and 3.002(3) Å and this asymmetry is reflected in the associated C–S bond lengths of 1.719(8) and 1.656(8) Å, respectively.

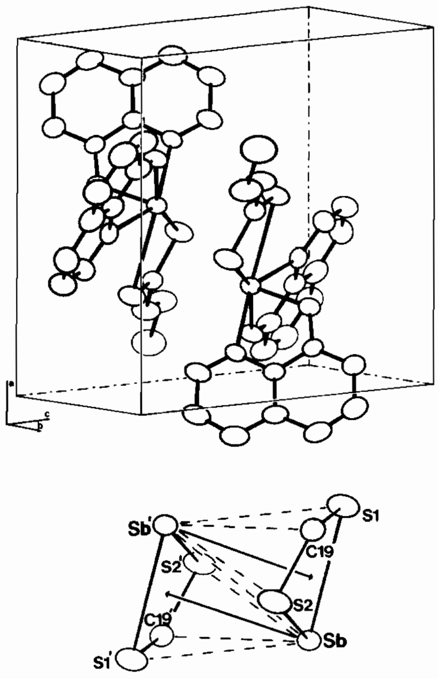

Fig. 2. Unit cell contents of Sb(exa)(oxin)$_2$, with the two centrosymmetrically related SbS$_2$C units shown in the lower figure. The arrows represent the proposed direction of the lone pair of electrons.

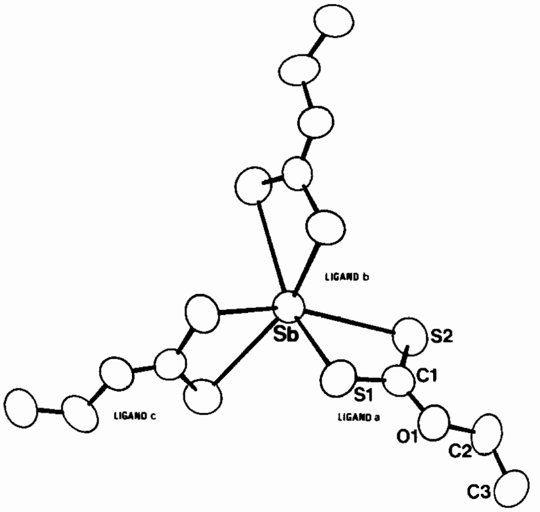

Fig. 3. The numbering scheme used for Sb(exa)$_3$, ligands b and c are related to ligand a by the symmetry operations *z*, *x*, *y* and *y*, *z*, *x* respectively.

TABLE VI. Important Intermolecular Distances (Å) less than the Van Der Waals Radii (Å) for Sb(exa)(oxin)$_2$.*

Atoms	Distance	Sum of van der Waals radii [15]
Sb···Sb'	3.781(1)	4.4
Sb···S(1)'	3.649(3)	4.0
Sb···S(2)'	3.595(2)	4.0
Sb···C(19)'	3.630(8)	3.9

*Primed atoms related by the symmetry operation $\bar{x}$, $\bar{y}$, $\bar{z}$.

TABLE VII. A Comparison of Sb–S and Associated C–S Distances (Å) in Sb(III) Xanthates.

Structure	Sb–S	C–S	Ref.
Sb(exa)$_3$			This work
	2.511(2)	1.719(8)	
	3.002(3)	1.656(8)	
Sb(iprxa)$_3$			[16]
	2.508(1)	1.731(5)	
	3.006(1)	1.655(5)	
[Sb(exa)$_2$Br]$_n$			[4]
	2.513(5)	1.732(16)	
	2.685(4)	1.678(15)	
	2.493(5)	1.719(15)	
	2.993(4)	1.683(16)	
[Sb(exa)$_3$·0.5(4,4'-bipy)]			[10]
	2.477(2)	1.738(5)	
	3.091(2)	1.647(5)	
	2.615(2)	1.719(5)	
	2.892(2)	1.663(5)	
	2.612(2)	1.731(5)	
	2.878(2)	1.665(5)	
Sb(exa)(oxin)$_2$			This work
	3.059(2)	1.692(8)	
	3.171(2)	1.682(8)	

The sulphur atoms involved in the short Sb–S distances form an equilateral triangle, the plane of which is 2.265(3) Å from a second equilateral triangle formed by the sulphur atoms associated with the weaker Sb–S interactions. As a consequence of the restricted bite distance of the xanthate ligands, one triangle is rotated 41.61(6)° relative to the other away from the ideal octahedral angle of 60°. The asymmetry of the Sb–S bond lengths is believed to arise as a result of a stereochemically active lone pair of electrons protruding, along the three-fold axis, through the triangular array formed by the sulphur atoms involved in the weak Sb–S bonds.

Conclusion

The mode of coordination of the xanthate ligand in Sb(exa)(oxin)$_2$ is unique amongst the structures of Sb(III) xanthates so far reported. This can be seen from Table VII which shows that the usual gross asymmetry of the Sb–S bond lengths observed for these xanthates is not found in Sb(exa)(oxin)$_2$ in which both sulphur atoms of the xanthate moiety form two long and essentially equivalent Sb–S bonds. The elongation of the Sb–S bonds has been ascribed to the intermolecular interactions mentioned above. The reduced asymmetry of the Sb–S bonds is also reflected in the associated C–S bonds which are equal within experimental error. The overall geometry of the xanthate ligand observed in Sb(exa)(oxin)$_2$ resembles that found for the sodium [12], potassium [13], and tetraethylammonium [14] salts of the ethylxanthate ligand, however, since the Sb–S distances [3.059(2) and 3.171(2) Å] are well within the sum of the van der Waals radii for these atoms (4.0 Å [15]), the Sb(exa)(oxin)$_2$ complex must be considered to be molecular.

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