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The Crystal and Molecular Structure of μ-Oxy-bis(triphenylazidoantimony), a Pentavalent Antimony Derivative with Metal–Oxygen–Metal Bonds.

BY GEORGE FERGUSON AND DONALD ROBERT RIDLEY
*Chemistry Department, The University, Guelph, Ontario, Canada N*1G 2*W*1

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Crystals of μ-oxy-bis(triphenylazidoantimony), $[(C_6H_5)_3SbN_3]_2O$, are monoclinic, space group $C2/c$ (C^6_{2h}) with four molecules in a unit cell of dimensions a = 12·633, b = 19·374, c = 14·178 Å and β = 105·4°. The structure was solved by the heavy-atom method and refined by least-squares procedures until convergence with R = 0·044 for 1982 reflexions greater than $3\sigma(I)$. The crystal structure is composed of discrete molecules lying on twofold (crystallographic) axes through the oxygen atom. The antimony atom in the asymmetric unit is in a slightly distorted trigonal bipyramidal environment with the bridging oxygen at one vertex and the azide group at the other. Main features of the molecular geometry include Sb–O(bridge) 1·985 (3), Sb–N(1) 2·236 (8), N(1)–N(2) 1·213 (12), N(2)–N(3) 1·116 (16), mean Sb–C 2·115 (8) Å, Sb–O–Sb 139·8 (4), Sb–N(1)–N(2) 118·3 (6), mean O–Sb–C 92·9 (3), and N(1)–N(2)–N(3) 176·8 (10)°.

Introduction

Infrared and conductance data of $XR_3Sb–O–SbR_3X$ (where $R = C_6H_5$ or CH_3 and X = halide, nitrate or pseudohalide) have been interpreted (Kolditz, Gitter & Rösel, 1962; Tranter, Addison & Sowerby, 1968; Doak, Long & Freedman, 1965; Goel & Ridley, 1971) in favour of non-ionic structures containing five-coordinated antimony. To elucidate the structure and the nature of the bonding in such systems we examined a number of derivatives and our preliminary results have been reported (Ferguson, Goel, March, Ridley & Prasad, 1971; March, Ferguson & Ridley, 1972). The azide derivative $[N_3(C_6H_5)_3Sb]_2O$ seemed particularly interesting in that although a few inorganic transition metal azide complexes have been carefully examined (*e.g.* $[N_3Co(NH_3)_5](N_3)_2$, Palenik, 1964; $[Cu(N_3)_2(NH_3)_2]$, Agrell, 1966; $[Cu(Et_2N.C_2H_4.NH.C_2H_4.NEt_2)N_3Br]$, Dori, 1968; $[Cu(N_3)_2(C_5H_5N)_2]$, Agrell, 1969; $[Zn(N_3)_2(C_5H_5N)_2]$, Agrell 1970; $[Zn(N_3)_2(NH_3)_2]$, Agrell & Vannerberg, 1971; $[Cu_2(N_3)_2\{[CH_2P(C_6H_5)_2]_2\}_3]$, Gaughan, Ziolo & Dori, 1971; $[As(C_6H_5)_4]_2[Pd_2(N_3)_6]$, Fehlhammer & Dahl, 1972), no structural determination of organometallic azide derivatives of group Vb elements has been reported.

Experimental

Needle-shaped crystals of $[N_3(C_6H_5)_3Sb]_2O$ were obtained by recrystallization from a chloroform–petroleum spirit mixture of a powder sample prepared by D. R. Ridley (Ridley, 1973). The crystal data are summarized in Table 1. Unit-cell and space-group data were obtained from rotation, Weissenberg and precession photographs taken with Cu $K\alpha$ radiation. The systematic absences (hkl absent if $h+k$ odd, $h0l$ absent if h odd and l odd) indicate space groups $C2/c$ or Cc. An analysis of E statistics indicated $C2/c$ as the correct choice; this was confirmed by the structure determination. Accurate cell parameters were obtained by a least-squares procedure applied to the setting angles of 12 high-order reflexions measured on our Hilger and Watts four-circle diffractometer. The intensities of all unique reflexions with 2θ (Cu $K\alpha$) $\le 140°$, corresponding to a minimum interplanar spacing of 0·82 Å, were measured with the diffractometer. The $\theta/2\theta$ scan technique was used with 1 second counts for 90 steps of 0·01° through the reflecting position. Stationary-crystal stationary-counter background measurements of 22·5 sec duration were measured at each end of the integrated scan. Three standard reflexions were monitored at two-hour intervals throughout the data collection. The maximum variation in intensity of standard reflexions was less than 2%. The crystal used in the data collection was approximately 0·10 × 0·12 mm in cross section and 0·32 mm long. Its faces were precisely indexed and absorption corrections were applied (Coppens, Leiserowitz & Rabinovich, 1965, program modified by R. F. Dellaca) to the intensity data which were then converted to structure amplitudes. The range of transmission coefficients in the absorption correction was 0·403 to 0·122. Reflexions with a net count less than 3σ above background $\{$where $\sigma(I) = [\text{scan} + \text{background} + (p\,.\,\text{Intensity})^2]^{1/2}\}$ were not included in the refinement; a value of p = 0·05 in the above expression gave what proved to be adequate weighting for the observed data; thus of the 2696 independent reflexion data obtained, 1982 were used in the refinement.

Table 1. *Crystal data*

μ-Oxy-bis(triphenylazidoantimony)
$C_{36}H_{30}N_6OSb_2$
M = 806·2
Monoclinic
a = 12·633 (2), b = 19·374 (4), c = 14·178 (5) Å, β = 105·4 (1)°, U = 3345·2 Å^3
D_m = 1·64, Z = 4, D_x = 1·66
$F(000)$ = 1592
Space group $C2/c$ (C^6_{2h}) (from analysis and E statistics)
Molecular symmetry 2 demanded
Linear absorption coefficient for X-rays (λ = 1·5418)
μ = 140·9 cm^{-1}

Structure determination and refinement

The coordinates of the antimony atom were determined from a three-dimensional Patterson synthesis and careful scrutiny of an electron-density distribution phased with the antimony-atom contributions revealed all N, O and C atoms. With all non-hydrogen atoms included in the structure-factor calculations, $R=\sum||F_o|-|F_c||/\sum|F_o|$ was 0·31.

Three cycles of full-matrix least-squares refinement adjusted the positional and isotropic thermal parameters of the non-hydrogen atoms and an overall scale factor, and reduced R to 0·13. The function minimized was $\sum w(F_o-F_c)^2$ with w taken as the reciprocal of the variance in F as determined from the counting statistics. The scattering factors used in the calculations of F_c were those given in *International Tables for Crystallography* (1962) for Sb, O, N and C; the values for hydrogen were those of Stewart, Davidson & Simpson (1965). The non-hydrogen atoms were then allowed anisotropic motion and four cycles of least-squares refinement using a block-diagonal approximation to the matrix reduced R to 0·075. At this point a three-dimensional difference synthesis was computed and clearly revealed the positions of the 15 independent hydrogen atoms. These were then allowed for in subsequent least-squares calculations, but were not refined. The refinement converged adeqately in five further cycles of block-diagonal least squares at which point the ratio of shift/e.s.d. was 0·27; $R=0·044$, and $R'=\sum w(|F_o|-|F_c|)^2/\sum wF_o^2=0·065$ for 1982 reflexions. A difference synthesis computed at the conclusion of the refinement using all data, not just those greater than $3\sigma(I)$, confirmed the correctness of the refinement.

Table 2. *Fractional atomic coordinates with standard deviations (in parentheses)*

	x	y	z
Sb	0·13335 (4)	0·16298 (2)	0·21715 (4)
C(11)	0·0624 (7)	0·1499 (4)	0·0656 (7)
C(12)	−0·0091 (8)	0·0957 (4)	0·0339 (7)
C(13)	−0·0620 (9)	0·0875 (5)	−0·0649 (7)
C(14)	−0·0481 (9)	0·1354 (6)	−0·1302 (7)
C(15)	0·0212 (10)	0·1906 (6)	−0·1002 (8)
C(16)	0·0788 (9)	0·1972 (5)	−0·0023 (7)
C(21)	0·1398 (6)	0·2562 (4)	0·2950 (6)
C(22)	0·1466 (9)	0·3200 (4)	0·2520 (8)
C(23)	0·1458 (9)	0·3790 (5)	0·3080 (8)
C(24)	0·1390 (9)	0·3755 (5)	0·4014 (8)
C(25)	0·1334 (9)	0·3123 (5)	0·4437 (7)
C(26)	0·1342 (8)	0·2520 (4)	0·3902 (6)
C(31)	0·2293 (7)	0·0805 (4)	0·2949 (6)
C(32)	0·3427 (8)	0·0846 (4)	0·3242 (7)
C(33)	0·4018 (8)	0·0312 (5)	0·3762 (7)
C(34)	0·3513 (8)	−0·0253 (5)	0·4014 (7)
C(35)	0·2382 (8)	−0·0309 (4)	0·3724 (7)
C(36)	0·1769 (7)	0·0228 (4)	0·3177 (7)
O	0	0·1277 (4)	$\frac{1}{4}$
N(1)	0·2808 (6)	0·2055 (4)	0·1783 (5)
N(2)	0·3230 (7)	0·1711 (4)	0·1266 (6)
N(3)	0·3662 (10)	0·1404 (5)	0·0816 (9)
H(12)	−0·0323	0·0538	0·0538
H(13)	−0·1294	0·0606	−0·0689
H(14)	−0·1027	0·1369	−0·1960
H(15)	0·0048	0·2434	−0·1571
H(16)	0·1056	0·2367	0·0198
H(22)	0·1601	0·3140	0·1780
H(23)	0·1514	0·4211	0·2749
H(24)	0·1363	0·4230	0·4467
H(25)	0·1328	0·2994	0·5229
H(26)	0·1064	0·2067	0·4133
H(32)	0·3571	0·1250	0·2917
H(33)	0·4870	0·0304	0·4045
H(34)	0·4070	−0·0587	0·4539
H(35)	0·2084	−0·0781	0·4025
H(36)	0·0883	0·0078	0·2761

Table 3. *Anisotropic thermal parameters* ($\times 10^3$) *except* Sb ($\times 10^4$)

The temperature factor takes the form

$$\exp\left[-2\pi^2(h^2a^{*2}U_{11}+k^2b^{*2}U_{22}+l^2c^{*2}U_{33}+2klb^*c^*U_{23}+2lha^*c^*U_{31}+2hka^*b^*U_{12})\right].$$

Standard deviations are given in parentheses.

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Sb	606 (2)	581 (2)	593 (3)	−54 (6)	319 (5)	−30 (5)*
C(11)	68 (5)	69 (5)	71 (6)	−9 (8)	47 (9)	0 (8)
C(12)	91 (7)	71 (5)	86 (7)	−35 (10)	58 (11)	−21 (10)
C(13)	94 (8)	97 (7)	78 (7)	−53 (11)	39 (12)	−13 (12)
C(14)	92 (8)	122 (8)	65 (6)	−41 (11)	12 (11)	−15 (13)
C(15)	121 (9)	103 (7)	77 (7)	−59 (12)	35 (13)	22 (14)
C(16)	109 (8)	79 (6)	71 (6)	−52 (9)	23 (12)	−4 (12)
C(21)	62 (5)	68 (5)	64 (5)	−7 (8)	27 (8)	3 (8)
C(22)	105 (8)	65 (5)	77 (6)	−11 (9)	55 (12)	−4 (10)
C(23)	127 (9)	65 (6)	89 (8)	−11 (10)	69 (14)	9 (11)
C(24)	101 (8)	62 (6)	98 (8)	0 (10)	51 (12)	−21 (11)
C(25)	105 (8)	93 (7)	59 (6)	−20 (10)	38 (11)	−29 (12)
C(26)	84 (6)	65 (5)	63 (5)	−16 (8)	21 (9)	−13 (9)
C(31)	60 (5)	64 (5)	64 (5)	5 (8)	34 (8)	−13 (8)
C(32)	70 (6)	77 (6)	76 (6)	6 (9)	15 (9)	22 (9)
C(33)	80 (6)	85 (6)	85 (7)	24 (11)	−2 (11)	13 (11)
C(34)	89 (7)	83 (6)	79 (7)	43 (10)	6 (11)	1 (11)
C(35)	93 (7)	67 (5)	76 (6)	13 (9)	44 (11)	3 (9)
C(36)	76 (6)	64 (5)	78 (6)	8 (9)	32 (10)	14 (9)
O	60 (5)	64 (5)	69 (5)	0	31 (8)	0
N(1)	75 (5)	86 (5)	77 (5)	−30 (8)	52 (8)	2 (8)
N(2)	85 (5)	78 (5)	85 (6)	−13 (8)	63 (9)	2 (8)
N(3)	173 (12)	104 (9)	174 (11)	27 (14)	212 (20)	−56 (15)

Several small peaks and troughs were noted around the Sb atom; we would have been surprised to find a completely flat topography as the least-squares program minimised $\sum w\Delta^2$ for data greater than $3\sigma(I)$ whereas in the difference synthesis all ΔF terms were given unit weights.

The final fractional coordinates of all the atoms and their thermal parameters are in Tables 2 and 3 respectively. Interatomic distances and angles with their e.s.d.'s are in Table 4. The observed and calculated structure amplitudes are in Table 5.

Table 4. *Interatomic distances and angles*

(*a*) Bonded distances with estimated standard deviations in parentheses

Sb——O	1·985 (3) Å	C(21)–C(22)	1·389 (12) Å
Sb——N(1)	2·236 (8)	C(22)–C(23)	1·393 (14)
Sb——C(11)	2·112 (9)	C(23)–C(24)	1·352 (17)
Sb——C(21)	2·107 (8)	C(24)–C(25)	1·374 (14)
Sb——C(31)	2·128 (7)	C(25)–C(26)	1·395 (13)
N(1)—N(2)	1·213 (12)	C(26)–C(21)	1·374 (13)
N(2)—N(3)	1·116 (16)	C(31)–C(32)	1·384 (12)
C(11)–C(12)	1·381 (12)	C(32)–C(33)	1·372 (13)
C(12)–C(13)	1·391 (13)	C(33)–C(34)	1·360 (14)
C(13)–C(14)	1·355 (16)	C(34)–C(35)	1·383 (14)
C(14)–C(15)	1·375 (16)	C(35)–C(36)	1·401 (12)
C(15)–C(16)	1·391 (13)	C(36)–C(31)	1·381 (12)
C(16)–C(11)	1·385 (14)		

(*b*) Valency angles with estimated standard deviations in parentheses

Sb——O——Sb	139·8 (4)°
C(11)–Sb——O	92·3 (3)
C(21)–Sb——O	95·1 (3)
C(31)–Sb——O	91·3 (3)
C(11)–Sb——C(21)	125·6 (3)
C(21)–Sb——C(31)	115·8 (3)
C(11)–Sb——C(31)	117·8 (3)
N(1)—Sb——O	178·3 (3)
Sb——N(1)—N(2)	118·3 (6)
Sb——N(1)—N(3)	119·5 (4)
N(1)—N(2)—N(3)	176·8 (10)
Sb——C(11)—C(12)	119·2 (7)
Sb——C(11)–C(16)	121·8 (6)
C(12)–C(11)–C(16)	118·9 (8)
C(11)–C(12)–C(13)	120·9 (9)
C(12)–C(13)–C(14)	119·6 (9)
C(13)–C(14)–C(15)	120·5 (9)
C(14)–C(15)–C(16)	119·8 (10)
C(15)–C(16)–C(11)	119·0 (10)
Sb——C(21)–C(22)	122·1 (7)
Sb——C(21)–C(26)	117·3 (6)
C(22)–C(21)–C(26)	120·6 (8)
C(21)–C(22)–C(23)	118·0 (10)
C(22)–C(23)–C(24)	122·0 (9)
C(23)–C(24)–C(25)	119·7 (9)
C(24)–C(25)–C(26)	120·0 (9)
C(25)–C(26)–C(21)	119·7 (8)
Sb——C(31)–C(32)	120·9 (6)
Sb——C(31)–C(36)	119·1 (6)
C(32)–C(31)–C(36)	119·9 (7)
C(31)–C(32)–C(33)	119·3 (9)
C(32)–C(33)–C(34)	121·5 (9)
C(33)–C(34)–C(35)	120·3 (9)
C(34)–C(35)–C(36)	118·8 (9)
C(35)–C(36)–C(31)	120·1 (8)

Table 4 (*cont.*)

(*c*) Some intramolecular non-bonded distances (Å)

(i) Within the asymmetric unit

Sb···C(12)	3·034	O·····C(36)	2·985
Sb···C(16)	3·075	N(1)···C(11)	2·997
Sb···C(22)	3·079	N(1)···C(21)	2·905
Sb···C(26)	2·997	N(1)···C(31)	3·097
Sb···C(32)	3·077	N(1)···C(16)	3·102
Sb···C(36)	3·048	N(1)···C(22)	3·135
Sb···N(2)	3·007	N(2)···C(11)	3·199
C(11)···C(21)	3·753	N(2)···C(31)	3·417
C(11)···C(31)	3·630	N(2)···C(16)	3·180
C(21)···C(31)	3·587	N(2)···C(32)	3·215
O···C(11)	2·956		
O···C(21)	3·021		
O···C(31)	2·941		
O···C(12)	3·099		
O···C(26)	3·291		

(ii) Between the two halves of the molecule

Sb···Sb	3·727	C(22)···C(22)	3·690
Sb···C(26)	3·739	C(22)···C(21)	3·713
C(11)···C(26)	3·363	C(22)···C(23)	3·742
C(12)···C(26)	3·706	C(23)···C(23)	3·612
C(12)···C(36)	3·646	C(26)···C(21)	3·749
C(16)···C(26)	3·629	C(33)···N(3)	3·535
C(16)···C(25)	3·746	C(32)···N(3)	3·724

(*d*) Intermolecular contacts < 3·7 Å

N(1)···C(15)$^{\mathrm{i}}$	3·607	N(3)····C(34)$^{\mathrm{iii}}$	3·360
N(2)···C(15)$^{\mathrm{i}}$	3·405	N(3)····C(35)$^{\mathrm{iii}}$	3·657
N(2)···C(16)$^{\mathrm{i}}$	3·510	C(13)···C(12)$^{\mathrm{iv}}$	3·659
N(3)···C(15)$^{\mathrm{ii}}$	3·551		
N(3)···C(16)$^{\mathrm{ii}}$	3·472		

The superscripts refer to the following equivalent positions:

i	$\frac{1}{2}-x$	$\frac{1}{2}-y$	$-z$
ii	$\frac{1}{2}+x$	$\frac{1}{2}-y$	$\frac{1}{2}+z$
iii	x	$-y$	$\frac{1}{2}+z$
iv	$-x$	$-y$	$-z$

which should be applied to the coordinates of the second atom.

Our numbering scheme is shown in Fig. 1, a stereo pair produced with the aid of *ORTEP* (Johnson, 1965). The arrangement of the molecules in the unit cell is in Fig. 2.

Discussion

Our analysis establishes that $[N_3(C_6H_5)_3Sb]_2O$ is non-ionic with pentacoordinated antimony. The molecule (Fig. 1) possesses a (crystallographic) twofold axis through the oxygen atom. The antimony atom in the asymmetric unit is in a slightly distorted trigonal bipyramidal environment with the bridging oxygen at one apex and the azide group at the other. The angle O–Sb–N(1) is 178·3 (3)°. The phenyl carbon atoms bonded to antimony are distorted slightly out of the equatorial plane away from the bridging oxygen in such a way that the antimony atom is 0·11 Å above the plane of C(11), C(21), C(31), resulting in a mean O–Sb–C(phenyl) angle of 92·9 (3)°.

The Sb–O bond length [1·985 (3) Å] is significantly shorter than values reported in a number of antimony derivatives, *e.g.* 2·084 (7) Å in tetraphenylantimony hydroxide (Beauchamp, Bennett & Cotton, 1969), 2·039 (8) and 2·027 (8) Å in dimethoxytriphenylantimony, and 2·061 (7) Å in methoxytetraphenylantimony (Shen, McEwen, La Placa, Hamilton & Wolf, 1968). It is, however, very similar to the Sb–O distance (1·99 (2) Å) in $Cl_5Sb–OP(CH_3)_3$ (Brändén & Lindqvist, 1961) in which equal attraction of electrons on both sides of the bridging oxygen was implied from theoretical calculations. The relatively short Sb–O distance in the

Table 5. *Observed structure amplitudes and calculated structure factors*

Reflexions with a net count of less than 3σ above background are marked with an asterisk.

$C_{36}H_{30}N_6OSb_2$

K	L	OBS	CALC
**** H = 0 ****			
0	-14	58	57
0	-12	88	89
0	-10	99	96
0	-8	24	26
0	-6	137	140
0	-2	328	356
0	4	38	95
2	-8	13*	4
2	-7	27	19
2	0	217	240
2	1	317	351
2	2	124	133
2	3	264	290
2	4	145	148
2	5	118	125
2	6	40	39
2	9	36	38
2	10	34	37
2	11	58	67
2	12	33	30
2	13	62	52
2	14	32*	31
2	16	5*	18
4	-7	34	34
4	-6	91	91
4	0	276	295
4	1	211	230
4	2	147	155
4	3	125	129
4	4	149	151
4	5	94	86
4	8	27	25
4	9	42	41
4	10	9*	12
4	11	53	53
4	12	40	37
4	13	67	63
4	14	44	43
4	16	22*	20
6	-13	12*	8
6	-11	10*	5
6	0	326	345
6	1	22	22
6	2	228	247
6	3	10*	5
6	4	122	127
6	5	109	112
6	6	98	101
6	7	40	42
6	8	27	27
6	9	16	15
6	10	44	42
6	12	65	58
6	14	63	58
6	15	5*	8
6	16	28	30
8	0	78	78
8	1	224	236
8	2	40	38
8	3	143	144
8	4	92	83
8	5	118	121
8	6	2*	4
8	7	30	32
8	8	9*	11
8	9	42	42
8	10	9*	5
8	11	51	48
8	12	17*	14
8	13	68	63
8	14	23*	20
8	15	39	39
10	0	131	137
10	1	168	169
10	2	138	139
10	3	94	98
10	4	45	48
10	5	20	18
10	6	70	69
10	7	13	10
10	8	11*	7
10	9	35	34
10	10	31	30
10	11	62	64
10	12	47	46
10	13	49	44
10	14	39	35
10	15	27	31
12	0	173	170
12	1	59	58
12	2	156	158
12	3	14	15
12	4	117	113
12	5	55	54
12	6	50	47
12	7	27	26
12	8	23	23
12	9	14	12
12	10	34	34
12	11	9*	2
12	12	56	52
12	14	37	40
14	-7	8*	4
14	-4	9*	1
14	0	60	57
14	1	159	157
14	2	32	30
14	3	96	92
14	5	59	55
14	6	11*	10
14	8	15*	18
14	9	25	25
14	10	12*	11
14	11	44	41
14	12	21*	13
14	13	32	35
16	-1	45	43
16	0	86	78
16	2	69	64
16	3	37	34
16	4	46	41
16	5	27	26
16	6	14*	12
16	7	8*	15
16	8	12*	9
16	9	7*	3
16	11	20	20
16	12	22	24
18	-10	5*	12
18	0	34	35
18	1	49	48
18	2	69	69
18	3	21	16
18	4	52	53
18	5	27	23
18	6	23*	17
20	-8	4*	1
20	-4	5*	4
20	0	19	15
20	1	65	65
20	2	21	19
20	3	42	45
20	5	25	27
20	6	8*	2
20	7	6*	9
22	0	37	43
22	1	24	25
22	2	30	36
22	3	19	23
22	4	11*	18
22	6	9*	6
**** H = 1 ****			
1	-15	31	32
1	-14	36	37
1	-13	73	78
1	-12	68	56
1	-11	94	97
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17	-3	57	50
17	-2	9*	3
17	-1	52	53
17	0	10*	7
17	1	40	42
17	2	5*	5
17	3	22	29
17	4	9*	2
17	5	6*	3
19	-7	16*	18
19	-6	23	23
19	-5	23	24
19	-4	29	33
19	-3	51	29
19	-2	40	43
19	-1	24	26
19	0	42	46
19	1	17*	23
19	2	24	27
19	3	12*	11
21	-2	17	29
**** H = 8 ****			
0	-14	63	53
0	-12	77	67
0	-10	31	37
0	-8	45	50
0	-6	14*	14
0	-4	38	36
0	-2	150	143
0	0	196	193
0	2	152	161
0	4	141	139
0	6	90	86
0	8	31	27
0	10	20	14
2	-14	13	25
2	-13	52	50
2	-12	33	34
2	-11	62	65
2	-10	35	33
2	-9	56	56
2	-8	29	27
2	-7	55	52
2	-6	25	25
2	-3	69	64
2	-2	45	81
2	-1	123	122
2	0	79	79
2	1	135	133
2	2	64	67
2	3	149	147
2	4	63	63
2	5	101	101
2	6	33	32
2	7	54	53
2	8	11*	10
2	9	13*	8
2	11	14*	17
4	-14	19	23
4	-13	40	46
4	-12	34	35
4	-11	67	67
4	-10	52	53
4	-9	62	56
4	-8	63	58
4	-7	34	34
4	-6	16*	11
4	-5	19	16
4	-4	53	44
4	-3	80	79
4	-2	80	78
4	-1	96	83
4	0	76	71
4	1	117	117
4	2	93	94
4	3	124	119
4	4	130	99
4	5	41	43
4	6	34	33
4	7	33	36
4	8	12*	12
4	9	2*	8
4	10	13*	4
4	11	8*	13
6	-12	57	59
6	-11	10*	3
6	-10	72	73
6	-9	14*	11
6	-8	57	55
6	-7	7*	9
6	-6	5*	5
6	-5	12*	13
6	-4	64	62
6	-3	9*	6
6	-2	134	132
6	-1	33	30
6	0	135	134
6	1	44	45
6	2	116	113
6	3	18	20
6	4	93	92
6	5	13*	7
6	6	64	66
6	7	15*	7
6	8	19	24
6	9	2*	13
8	-13	54	55
8	-12	14*	13
8	-11	66	69
8	-10	8*	5
8	-9	61	63
8	-8	32	33
8	-7	74	20
8	-6	28	24
8	-5	3*	9
8	-4	2*	4
8	-3	115	116
8	-2	10*	12
8	-1	109	135
8	0	24	20
8	1	31	76
8	2	43	43
8	3	85	82
8	4	46	45
8	5	59	61
8	6	23	24
8	7	34	39
10	-12	40	38
10	-11	51	52
10	-10	51	52
10	-9	55	54
10	-8	27	25
10	-7	34	33
10	-6	5*	4
10	-5	14*	13

Table 5 (*cont.*)

K	L	OBS	CALC
10	-4	39	38
10	-3	30	33
10	-2	69	69
10	-1	64	62
10	0	68	67
10	1	97	98
10	2	68	66
10	3	74	72
10	4	43	44
10	5	37	37
10	6	30	28
10	7	17	15
10	8	15*	15
10	9	7*	2
12	-10	48	47
12	-9	17*	17
12	-8	45	40
12	-6	21	24
12	-5	9*	13
12	-4	34	37
12	-3	13*	10
12	-2	67	64
12	-1	25	23
12	0	78	78
12	1	39	36
12	2	78	80
12	3	27	25
12	4	66	59
12	5	17	14
12	6	32	35
12	7	10*	9
12	8	11*	13
12	9	10*	7
14	-9	44	40
14	-8	4*	2
14	-7	22	24
14	-6	9*	9
14	-5	7*	6
14	-4	17*	15
14	-3	39	35
14	-2	24	20
14	-1	54	51
14	0	27	26
14	1	58	60
14	2	10*	3
14	3	53	54
14	5	48	52
14	7	14*	18
16	-9	9*	21
16	-8	3*	21
16	-7	10*	12
16	-6	12*	14
16	-5	6*	11
16	-4	12*	12
16	-3	22	24
16	-2	23	21
16	-1	26	28
16	0	32	31
16	1	27	30
16	2	39	46
16	3	21	26
16	4	33	35
16	5	11*	18
16	6	10*	17
18	-8	21	21
18	-7	6*	5
18	-5	4*	1
18	-4	22	21
18	-3	11*	6
18	-2	24	32
18	-1	22	20
18	0	30	34
18	1	17*	19
18	2	25	35
18	3	7*	13
20	-4	3*	4
20	-3	17	20
20	-2	4*	2
20	-1	21	27
**** H = 9 ****			
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1	-11	40	43
1	-10	49	49
1	-9	64	70
1	-8	54	51
1	-7	38	35
1	-6	51	50
1	-5	63	51
1	-4	32	28
1	-3	30	32
1	-2	14*	11
1	-1	47	43
1	0	33	31
1	1	38	36
1	2	43	52
1	3	71	70
1	4	61	60
1	5	73	70
1	6	42	42
1	7	51	54
1	8	33	30
1	9	34	38
1	10	13*	14
3	-12	62	62
3	-11	9*	5
3	-10	92	90
3	-9	15*	16
3	-8	105	103
3	-7	24	23
3	-6	107	105
3	-4	51	52
3	-3	6*	4
3	-2	4*	3
3	-1	10*	15
3	0	41	43
3	1	33	27
3	2	35	43
3	3	12*	17
3	4	73	90
3	6	7	28
3	7	14*	4
3	8	60	61
3	9	13*	5
3	10	23	29
5	-13	37	36
5	-12	27	30
5	-11	64	67
5	-10	27	28
5	-9	31	27
5	-8	17*	14
5	-7	75	71
5	-6	47	40
5	-5	106	106
5	-4	43	43
5	-3	37	36
5	-2	12*	12
5	-1	33	27
5	0	12*	6
5	1	57	56
5	2	12*	12
5	3	74	72
5	4	29	30
5	5	72	73
5	6	54	52
5	7	75	73
5	8	23	21
5	9	42	44
5	10	9*	10
7	-12	20	23
7	-11	53	45
7	-10	37	41
7	-9	82	82
7	-8	50	54
7	-7	78	75
7	-6	65	60
7	-5	65	62
7	-4	35	37
7	-3	51	49
7	-2	16*	14
7	-1	29	26
7	0	9*	11
7	1	33	35
7	2	51	46
7	3	94	84
7	4	46	50
7	5	52	53
7	6	47	45
7	7	44	45
7	8	31	34
7	9	26	34
9	-11	9*	10
9	-10	72	71
9	-9	6*	4
9	-8	74	72
9	-6	42	44
9	-5	25	24
9	-4	54	50
9	-3	7*	7
9	-2	11*	5
9	-1	23	22
9	0	36	34
9	1	9*	3
9	2	59	57
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11	-8	16*	14
11	-7	72	69
11	-6	27	24
11	-5	70	78
11	-4	9*	10
11	-3	14	15
11	-2	13*	15
11	-1	30	31
11	0	23	22
11	1	44	46
11	2	8*	7
11	3	35	40
11	4	17*	11
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11	6	9*	10
11	7	29	37
13	-9	40	39
13	-8	48	47
13	-7	55	45
13	-6	47	50
13	-5	28	27
13	-4	25	26
13	-3	17	15
13	-1	11*	6
13	0	11*	13
13	1	24	27
13	2	32	32
13	3	27	23
13	4	32	30
13	5	26	31
13	6	14*	23
13	7	15*	19
15	11	7*	13
15	-9	3*	13
15	-7	3*	22
15	-6	51	43
15	-5	15*	11
15	-4	34	37
15	-3	12*	3
15	-2	16*	5
15	-1	5*	7
15	0	15	21
15	1	15	15
15	2	26	25
15	3	17	19
15	4	36	42
17	-8	10*	9
17	-7	33	40
17	-6	4*	6
17	-4	31	34
17	-3	19	17
17	-2	4*	4
17	0	6*	3
17	1	19	22
17	2	13*	10
17	3	20	31
19	-4	15*	19
19	-3	3*	5
19	-2	5*	6
19	-1	5*	3
**** H = 10 ****			
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0	-10	46	46
0	-8	50	59
0	-6	113	105
0	-4	124	116
0	-2	70	73
0	0	39	86
0	2	37	38
0	4	40	37
0	6	30	31
0	8	42	45
2	-11	30	29
2	-10	35	33
2	-9	50	48
2	-8	34	35
2	-7	74	70
2	-6	51	49
2	-5	84	81
2	-4	54	55
2	-3	105	102
2	-2	37	37
2	-1	58	54
2	0	34	29
2	1	16*	15
2	2	19	16
2	3	5*	5
2	5	27	25
2	6	14*	16
2	7	47	47
2	8	15*	14
2	9	27	41
4	-12	7*	7
4	-11	21	25
4	-10	32	34
4	-9	54	57
4	-8	33	34
4	-7	45	43
4	-6	47	45
4	-5	72	71
4	-4	65	61
4	-3	76	72
4	-2	53	47
4	-1	54	57
4	0	39	37
4	1	21	21
4	2	6*	6
4	3	25	26
4	4	27	24
4	5	11	15
4	6	13*	19
4	7	33	35
4	8	25	27
4	9	21	17
4	10	7*	4
4	11	57	56
6	-9	6*	5
6	-8	79	81
6	-6	116	111
6	-5	21	20
6	-4	123	120
6	-2	93	89
6	-1	15*	10
6	0	44	44
6	1	7*	13
6	2	18	13
6	4	30	31
6	5	10*	9
6	6	36	35
6	7	7*	3
6	8	32	36
8	-11	21	21
8	-10	31	31
8	-9	46	47
8	-8	33	29
8	-7	51	53
8	-6	27	26
8	-5	121	115
8	-4	41	39
8	-3	100	97
8	-2	31	29
8	-1	50	55
8	0	10*	6
8	1	14*	12
8	2	10*	5
8	3	19	15
8	5	27	24
8	6	8*	9
8	7	29	36
8	8	12*	14
10	-10	10*	16
10	-9	27	28
10	-8	31	31
10	-7	49	49
10	-6	57	53
10	-5	41	39
10	-4	58	57
10	-3	40	44
10	-2	45	47
10	-1	52	51
10	0	33	34
10	1	28	27
10	2	15*	5
10	3	12*	1
10	4	14*	12
10	5	14*	15
10	6	19	21
10	7	14*	13
12	-8	43	41
12	-7	5*	9
12	-6	55	55
12	-5	29	27
12	-4	45	47
12	-3	4*	7
12	-2	44	42
12	-1	21	17
12	0	43	39
12	1	14	19
12	2	14*	13
12	3	8*	1
12	4	10*	11
12	5	4*	4
12	6	10*	18
14	-10	3*	2
14	-7	9*	40
14	-6	10*	12
14	-5	54	50
14	-4	15*	16
14	-3	60	58
14	-2	15*	14
14	-1	41	42
14	0	11*	6
14	1	19	19
14	3	2*	1
14	4	11*	8
16	-8	27	27
16	-7	26	26
16	-6	25	31
16	-5	27	29
16	-4	35	36
16	-3	26	27
16	-2	27	29
16	-1	21	19
16	0	15*	14
**** H = 11 ****			
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1	-11	34	39
1	-10	7*	12
1	-9	12*	11
1	-8	11*	10
1	-7	39	35
1	-6	22	21
1	-5	66	64
1	-4	45	45
1	-3	74	86
1	-2	52	49
1	-1	97	90
1	0	45	44
1	1	51	54
1	2	35	32
1	3	35	32
1	4	8*	4
1	5	19	16
1	6	7*	4
3	-11	11*	7
3	-10	23	21
3	-9	11*	12
3	-8	14*	7
3	-6	56	43
3	-5	13*	2
3	-4	63	77
3	-3	20	13
3	-2	37	43
3	0	45	48
3	2	74	70
3	3	11*	4
3	4	20	23
3	7	2*	1
5	-11	19	20
5	-10	14*	4
5	-9	15*	6
5	-8	16*	14
5	-7	7*	12
5	-6	30	30
5	-5	51	49
5	-4	16*	21
5	-3	79	73
5	-2	30	33
5	-1	75	73
5	0	24	25
5	1	61	62
5	2	14*	18
5	3	41	39
5	4	22	23
5	6	3*	1
5	7	10*	4
7	-10	9*	3
7	-9	4*	9
7	-8	13*	10
7	-7	27	25
7	-6	13*	14
7	-5	53	55
7	-4	32	33
7	-3	49	50
7	-2	30	39
7	-1	56	51
7	0	50	47
7	1	55	56
7	2	35	34
7	3	30	33
7	4	16*	19
7	5	13*	14
7	6	5*	2
7	7	9*	1
9	-8	9*	6
9	-7	5*	3
9	-6	36	37
9	-5	2*	15
9	-4	50	53
9	-3	2*	5
9	-2	68	70
9	-1	5*	5
9	0	77	80
9	1	23	19
9	2	49	51
9	4	20	22
9	5	9*	1
9	6	5*	3
11	-9	5*	2
11	-7	4*	11
11	-6	17*	15
11	-5	36	38
11	-4	21	20
11	-3	57	52
11	-2	26	25
11	-1	70	72
11	0	12*	15
11	1	56	57
11	2	8*	16
11	3	32	37
11	4	6*	1
11	5	13*	11
13	-7	19	21
13	-6	22	19
13	-5	23	28
13	-4	35	38
13	-3	30	34
13	-2	34	37
13	-1	41	42
13	0	41	42
13	1	29	35
13	2	25	28
13	3	7*	18
15	-7	11*	4
15	-6	15*	20
15	-5	12*	12
15	-4	26	29
15	-3	17	16
15	-2	34	30
15	0	28	38
15	1	7*	10
**** H = 12 ****			
0	-10	57	53
0	-8	46	46
0	-6	7*	16
0	-4	18	20
0	-2	41	38
0	0	55	54
0	2	63	60
0	4	41	41
0	6	27	36
2	-10	31	33
2	-9	50	49
2	-8	29	25
2	-7	32	33
2	-6	6*	3
2	-5	7*	8
2	-4	15*	5
2	-3	25	26
2	-2	23	26
2	-1	32	32
2	0	39	40
2	1	53	52
2	2	31	30
2	3	52	52
2	4	16*	17
2	5	34	38
2	6	11*	10
4	-10	32	35
4	-9	49	47
4	-8	21	22
4	-7	42	43
4	-6	12*	15
4	-5	8*	3
4	-3	26	26
4	-2	12*	11
4	-1	52	40
4	0	37	29
4	1	59	56
4	2	41	38
4	3	45	46
4	4	20	23
4	5	23	30
4	6	12*	17
6	-9	16*	11
6	-8	57	55
6	-7	19	12
6	-6	23	24
6	-4	7*	5
6	-3	8*	3
6	-2	26	25
6	0	48	50
6	2	59	60
6	3	3*	8
6	4	43	50
6	5	12*	3
8	-8	21	21
8	-7	32	35
8	-6	12*	7
8	-5	6*	5
8	-4	9*	10
8	-3	12*	14
8	-2	5*	11
8	-1	35	35
8	0	15*	14
8	1	48	47
8	2	25	22
8	3	42	45
8	4	10*	18
8	5	5*	35
10	-7	4*	24
10	-6	13*	13
10	-4	17	5
10	-3	21	21
10	-1	30	30
10	0	30	31
10	1	34	36
10	2	23	26
10	3	21	32
10	4	4*	25
12	-9	3*	10
12	-8	16	15
12	-7	12*	13
12	-6	3*	4
12	-5	6*	3
12	-4	3*	11
12	-2	14	20
12	-1	9*	5
12	0	31	35
12	1	11*	10
12	2	24	38
14	-4	6*	3
14	-3	11*	10
14	-2	11*	9
14	-1	18	22
**** H = 13 ****			
1	-8	36	32
1	-7	58	56
1	-6	38	36
1	-5	56	54
1	-4	26	25
1	-3	28	26
1	-2	25	21
1	1	10*	6
1	2	20	22
1	3	18	21
1	4	9*	13
1	5	16	27
3	-8	62	59
3	-7	11*	6
3	-6	67	64
3	-5	5*	10
3	-4	41	39
3	-3	6*	6
3	-2	21	19
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3	2	24	28
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3	4	16*	26
5	-7	61	60
5	-6	15*	18
5	-5	44	44
5	-4	17*	15
5	-3	24	23
5	-2	8*	4
5	-1	18	10
5	0	7*	6
5	1	11*	11
5	2	11*	14
5	3	13*	24
5	4	8*	19
7	-10	2*	24
7	-9	2*	35
7	-8	4*	32
7	-7	10*	44
7	-6	37	31
7	-5	35	35
7	-4	20	21
7	-3	15	24
7	-2	16	14
7	1	11*	15
7	3	20	28
9	-10	27	29
9	-9	10*	2
9	-8	37	39
9	-7	5*	13
9	-6	36	39
9	-5	4*	5
9	-4	23	24
9	-2	6*	12
9	-1	12*	7
9	0	4*	4
9	2	12*	16
11	-7	34	35
11	-6	3*	10
11	-5	26	28
11	-4	2*	5
11	-3	13*	15
11	-2	3*	4
11	0	7*	2
**** H = 14 ****			
0	-8	5*	26
0	-6	61	59
0	-2	50	50
0	0	39	42
2	-10	1*	2
2	-9	7*	20
2	-6	15	16
2	-5	47	49
2	-4	25	24
2	-3	45	44
2	-2	24	25
2	-1	37	36
2	0	18	21
2	1	16	20
2	2	4*	8
4	-10	1*	13
4	-8	7*	17
4	-7	26	28
4	-6	30	30
4	-5	37	36
4	-4	26	29
4	-3	41	38
4	-2	29	28
4	-1	34	32
4	0	17	20
4	1	12*	15
6	-9	5*	5
6	-8	27	27
6	-6	39	40
6	-5	4*	7
6	-4	47	48
6	-3	7*	5
6	-2	38	37
6	-1	9*	10
6	0	14*	20
8	-8	4*	11
8	-7	32	32
8	-6	6*	8
8	-5	34	39
8	-4	12*	15
8	-3	35	34
8	-2	12*	11
8	-1	23	26
**** H = 15 ****			
1	-8	6*	1
1	-7	4*	8
1	-5	21	19
1	-4	10*	12
1	-3	23	26
1	-2	15	18
1	-1	29	36
3	-6	14	11
3	-4	27	26
3	-3	4*	1
3	-2	34	30
5	-5	16	19
5	-4	8*	6

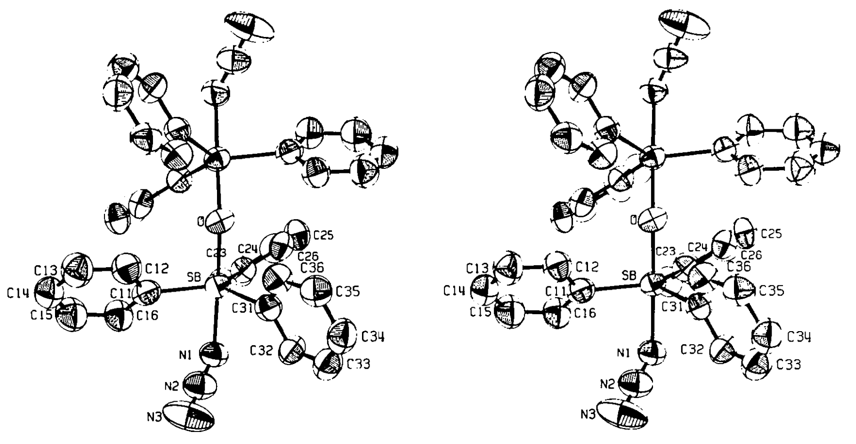

Fig. 1. Stereoscopic drawing of the molecule of μ-oxy-bis(triphenylazidoantimony). The thermal ellipsoids are drawn at the 50% probability level.

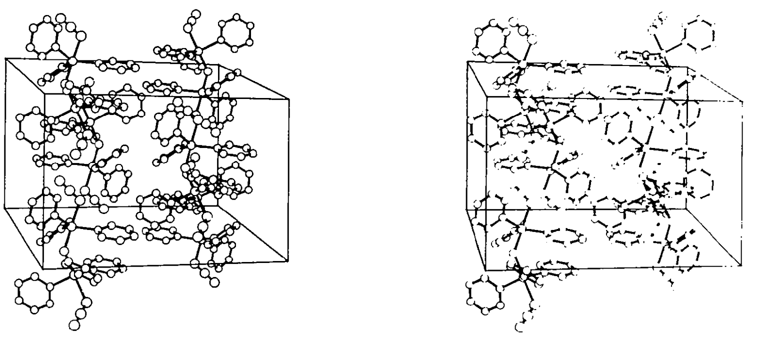

Fig. 2. Stereoscopic diagram showing the molecular packing in the unit cell. The origin of the cell is at the lower right-rear corner with **a** ↑, **b** ←, and **c** out of the page.

present structure necessitates the expansion of the Sb–O–Sb angle to 139·8 (4)° from the normal tetrahedral angle in order to alleviate steric interactions between phenyl rings in the two halves of the molecule (see below); in di-μ-ethoxybistetrachloroantimony, where no steric overcrowding occurs, the Sb–O–Sb angle is 110·1 (1)° (Preiss, 1968). The Sb–O distance would also indicate little ionic character, in accord with the compound's relatively low melting point.

The mean Sb–C(phenyl) bond length (2·115 (9) Å) is similar to the mean values found in related derivatives *e.g.* 2·120 (12) Å in dimethoxytriphenylantimony (Shen, McEwen, La Placa, Hamilton & Wolf, 1968). The C–Sb–C angles range from 115·8 to 125·6°; the deviations from 120° may be attributed to steric interactions of the planar phenyl rings which are rotated about the Sb–C(phenyl) bonds in such a way as to minimize intra- and intermolecular interactions. Phenyl rings 1 and 3 (Fig. 1) are rotated through 51 and 71° respectively in the same sense from the plane through C(11), C(21) and C(31), while ring 2 is rotated 37° in the opposite direction. The intramolecular interactions occuring between the two halves of the molecule are listed in Table 4; the intramolecular overcrowding between ring 2 and ring 2′ (its twofold-axis relation) would have been prohibitive in the absence of an angle increase at the oxygen atom.

The Sb–N(1) distance is 2·236 (8) Å; no other antimony–azide distances have previously been reported. The nearly linear azide group (N(1)–N(2)–N(3) angle 176·8 (9)°) is oriented *trans* with respect to the O–Sb′ bond in the symmetry related portion of the molecule and angle Sb–N(1)–N(2) is 118·3 (6)°. The N–N bonds in the azide are asymmetric, N(1)–N(2) 1·213 (12) Å and N(2)–N(3) 1·116 (6) Å, in agreement with values reported for the corresponding bonds, in azidopentamminecobalt(III) azide [1·208 (7) and 1·145 (7)] (Palenik, 1964); in diazidodiaminecopper(II) [1·186 (11) and 1·139 (13)] (Agrell, 1966); in diazido-μ-1,2-bis(diphenylphosphino)ethane-bis-[1,2-bis(diphenylphosphino)ethane]-dicopper(II) [1·196 (8) and 1·076 (18)] (Gaughan, Ziola & Dori, 1971); and in di-μ-azido-bis[diazidopalladate(II)] anion (1·205 (12) and 1·139 (11) for terminal azide grops, and 1·239 (14) and 1·142 (13) Å for the bridging azide group) (Fehlhammer & Dahl, 1972).

The shorter distances between molecules are listed in Table 4 and correspond to van der Waals distances; Fig 2 gives a view of the molecular packing. The shortest intermolecular contact occurs between N(3), the terminal azide nitrogen atom, and C(34) of a neighbouring phenyl ring (N···C 3·360 Å). It is possible that the small deviation of the N(1)–N(2)–N(3) angle from linearity (3·2°) occurs to allow a slightly better packing arrangement.

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