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μ -Oxido-bis[(chloroacetato- κ O)-triphenylantimony(V)]

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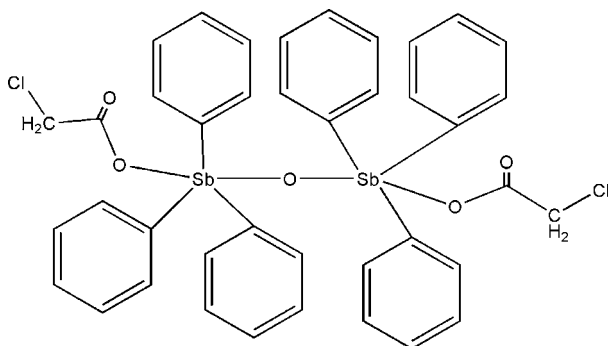
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 Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.011$ Å; R factor = 0.040; wR factor = 0.110; data-to-parameter ratio = 14.8.

The Sb atom in the centrosymmetric title complex, $[\text{Sb}_2(\text{C}_6\text{H}_5)_6(\text{C}_2\text{H}_2\text{ClO}_2)_2\text{O}]$, has a distorted trigonal-bipyramidal geometry. The bridging oxide O atom occupies one of the axial sites, while the O atom of the chloroacetate ligand occupies the other.

Related literature

For related literature, see: Gibbons & Sowerby (1998).



Experimental

Crystal data

$[\text{Sb}_2(\text{C}_6\text{H}_5)_6(\text{C}_2\text{H}_2\text{ClO}_2)_2\text{O}]$
 $M_r = 909.07$
 Monoclinic, $P2_1/c$
 $a = 10.4950$ (12) Å
 $b = 19.416$ (2) Å
 $c = 9.2584$ (10) Å
 $\beta = 95.383$ (2)°

$V = 1878.3$ (4) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 1.62$ mm⁻¹
 $T = 298$ (2) K
 $0.41 \times 0.18 \times 0.11$ mm

Data collection

Bruker SMART diffractometer
 Absorption correction: multi-scan
 (*SADABS*; Sheldrick, 1996)
 $T_{\min} = 0.556$, $T_{\max} = 0.842$

8838 measured reflections
 3305 independent reflections
 2465 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.035$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.109$
 $S = 1.00$
 3305 reflections
 223 parameters

6 restraints
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 1.14$ e Å⁻³
 $\Delta\rho_{\text{min}} = -1.07$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Sb1—O3	1.9503 (4)	O3—Sb1 ⁱ	1.9503 (4)
Sb1—O1	2.197 (4)		
O3—Sb1—O1	176.64 (11)	Sb1—O3—Sb1 ⁱ	180

 Symmetry code: (i) $-x, -y, -z + 1$.

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C17—H17 \cdots Cl1 ⁱⁱ	0.93	2.93	3.592 (8)	130

 Symmetry code: (ii) $x - 1, y, z$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 1997); software used to prepare material for publication: *SHELXTL*.

We acknowledge the National Natural Science Fund of China (grant No. 20771053) and the Natural Science Fund of Shandong Province (2005ZX09) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SG2199).

References

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supplementary materials

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μ -Oxido-bis[(chloroacetato- κ O)triphenylantimony(V)]

L. Quan, H. Yin and D. Wang

Comment

Organoantimony esters of carboxylic acids are widely used as biocides, as fungicides and, in industry, as homogeneous catalysts. We have therefore synthesized the title compound, (I), and present its crystal structure here. The molecular structure of (I) is shown in Fig. 1. For a related structure see bis(triphenylantimony) trifluoroacetate [Sb(C₆H₅)₃(O₂C₂F₃)₂O] (*M. N. Gibbons & Sowerby, 1998*).

The title compound comprises two (chloroacetyl)tris triphenylantimony units bridged by an oxygen dianion. Centrosymmetrically molecules are linked by short intermolecular C17—H17 \cdots Cl1 contacts [symmetry codes: $-1 + x, +y, +z$], and one-dimensional polymeric chains run parallel to the *a* axis, (Fig. 2.). The O—Sb—O angle is 176.64 (11)°. The Sb—O distances vary with the role oxygen atoms play in the structure, the terminal Sb1—O1 [2.197 (4) Å] bond being longer than bridging Sb1—O3 [1.9503 (4) Å] bond. (Table 1, Table 2.).

Experimental

Chloroacetic acid (0.02 g, 0.2 mmol) and sodium methoxide (0.4 ml, 0.2 mmol) was added to a stirred solution of oxygen bridged bis(chloro) tris triphenylantimony (0.16 g, 0.2 mmol) and toluene (20 ml). The reaction mixture was stirred at room temperature for 24 h. Crystals suitable for X-ray analysis were obtained by slow evaporation of a petroleum/dichloromethane (1:2 v/v) solution over a period of a week (yield 85%. m.p. 470k). Anal. Calcd (%) for C₄₀H₃₄O₅Sb₂Cl₂ (Mr = 909.08): C, 52.80; H, 3.72; O, 8.76; Cl, 7.76; Sb, 26.74. Found (%): C, 52.85; H, 3.77; O, 8.80; Cl, 7.80; Sb, 26.79

Refinement

The H atoms bound to aryl groups were idealized with a C—H = 0.93 Å. All other H atoms were also placed in idealized positions, with C—H = 0.97 Å, and treated as riding on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C}, \text{O})$ or $1.5 U_{\text{eq}}(\text{C})$ for the methyl group.

Figures

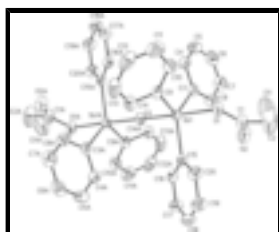


Fig. 1. The molecular structure, with atom labels and 50% probability displacement ellipsoids for non-H atoms.



Fig. 2. Crystal packing, showing a extensity polymer chain, linked by C17—H17...C11 hydrogen bonds (dashed lines).

μ -Oxido-bis[(chloroacetato- κ O)triphenylantimony(V)]

Crystal data

[Sb₂(C₆H₅)₆(C₂H₂Cl₁O₂)₂O]

$M_r = 909.07$

Monoclinic, $P2_1/c$

$a = 10.4950$ (12) Å

$b = 19.416$ (2) Å

$c = 9.2584$ (10) Å

$\beta = 95.383$ (2)°

$V = 1878.3$ (4) Å³

$Z = 2$

$F_{000} = 900$

$D_x = 1.607$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 3131 reflections

$\theta = 2.1$ – 24.7°

$\mu = 1.62$ mm⁻¹

$T = 298$ (2) K

Block, colourless

$0.41 \times 0.18 \times 0.11$ mm

Data collection

Bruker SMART
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298$ (2) K

φ and ω scans

Absorption correction: multi-scan
(SADABS; Sheldrick, 1996)

$T_{\min} = 0.556$, $T_{\max} = 0.842$

8838 measured reflections

3305 independent reflections

2465 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.035$

$\theta_{\text{max}} = 25.0^\circ$

$\theta_{\text{min}} = 2.0^\circ$

$h = -7 \rightarrow 12$

$k = -23 \rightarrow 21$

$l = -10 \rightarrow 11$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.040$

$wR(F^2) = 0.109$

$S = 1.00$

3305 reflections

223 parameters

6 restraints

Primary atom site location: structure-invariant direct
methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring
sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0489P)^2 + 4.2337P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$

$\Delta\rho_{\text{max}} = 1.14$ e Å⁻³

$\Delta\rho_{\text{min}} = -1.07$ e Å⁻³

Extinction correction: none

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Sb1	0.14913 (3)	0.054058 (18)	0.45933 (4)	0.04187 (15)
Cl1	0.5107 (3)	0.17116 (18)	0.2357 (4)	0.1409 (11)
O1	0.3199 (4)	0.1102 (2)	0.4046 (5)	0.0581 (11)
O2	0.2708 (6)	0.2089 (3)	0.5109 (6)	0.0856 (16)
O3	0.0000	0.0000	0.5000	0.0530 (15)
C1	0.3341 (7)	0.1745 (4)	0.4368 (8)	0.0679 (19)
C2	0.4414 (10)	0.2112 (5)	0.3674 (12)	0.127 (4)
H2A	0.5079	0.2221	0.4440	0.152*
H2B	0.4076	0.2546	0.3286	0.152*
C3	0.2216 (5)	0.0580 (3)	0.6778 (6)	0.0455 (13)
C4	0.2782 (7)	-0.0004 (4)	0.7389 (8)	0.0693 (19)
H4	0.2832	-0.0400	0.6834	0.083*
C5	0.3272 (8)	-0.0002 (4)	0.8811 (9)	0.082 (2)
H5	0.3673	-0.0394	0.9211	0.099*
C6	0.3175 (8)	0.0574 (5)	0.9647 (8)	0.081 (2)
H6	0.3492	0.0569	1.0619	0.098*
C7	0.2617 (8)	0.1152 (4)	0.9058 (8)	0.076 (2)
H7	0.2561	0.1544	0.9625	0.092*
C8	0.2134 (7)	0.1160 (3)	0.7628 (7)	0.0642 (18)
H8	0.1751	0.1558	0.7230	0.077*
C9	0.2230 (6)	-0.0235 (3)	0.3337 (6)	0.0448 (14)
C10	0.3500 (7)	-0.0424 (4)	0.3467 (8)	0.0679 (19)
H10	0.4074	-0.0195	0.4127	0.082*
C11	0.3925 (9)	-0.0937 (4)	0.2649 (11)	0.093 (3)
H11	0.4781	-0.1067	0.2769	0.112*
C12	0.3100 (10)	-0.1264 (4)	0.1649 (10)	0.088 (3)
H12	0.3399	-0.1609	0.1071	0.106*
C13	0.1843 (9)	-0.1089 (4)	0.1497 (9)	0.088 (3)
H13	0.1280	-0.1316	0.0821	0.105*
C14	0.1402 (7)	-0.0574 (3)	0.2346 (7)	0.0609 (17)
H14	0.0539	-0.0456	0.2247	0.073*
C15	0.0381 (5)	0.1302 (3)	0.3458 (6)	0.0446 (13)
C16	-0.0780 (6)	0.1476 (3)	0.3891 (7)	0.0554 (16)
H16	-0.1081	0.1257	0.4686	0.066*
C17	-0.1512 (7)	0.1976 (3)	0.3149 (9)	0.070 (2)
H17	-0.2291	0.2106	0.3467	0.084*
C18	-0.1096 (8)	0.2278 (4)	0.1957 (9)	0.074 (2)
H18	-0.1608	0.2602	0.1441	0.088*
C19	0.0063 (8)	0.2112 (4)	0.1511 (8)	0.074 (2)
H19	0.0357	0.2328	0.0710	0.088*
C20	0.0798 (7)	0.1616 (3)	0.2265 (7)	0.0619 (17)
H20	0.1586	0.1494	0.1959	0.074*

supplementary materials

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sb1	0.0420 (2)	0.0387 (2)	0.0444 (2)	-0.00671 (18)	0.00144 (15)	-0.00121 (18)
C11	0.1011 (19)	0.184 (3)	0.144 (3)	-0.051 (2)	0.0457 (18)	-0.015 (2)
O1	0.059 (3)	0.048 (2)	0.068 (3)	-0.019 (2)	0.011 (2)	-0.002 (2)
O2	0.099 (4)	0.067 (3)	0.094 (4)	-0.022 (3)	0.029 (3)	-0.015 (3)
O3	0.041 (3)	0.052 (3)	0.066 (4)	-0.016 (3)	0.006 (3)	0.003 (3)
C1	0.062 (4)	0.068 (5)	0.075 (5)	-0.025 (4)	0.014 (4)	-0.007 (4)
C2	0.125 (7)	0.114 (6)	0.152 (8)	-0.045 (6)	0.065 (6)	-0.035 (6)
C3	0.047 (3)	0.046 (3)	0.043 (3)	-0.013 (3)	0.003 (3)	-0.001 (3)
C4	0.085 (5)	0.051 (4)	0.069 (5)	-0.003 (4)	-0.011 (4)	0.003 (3)
C5	0.095 (6)	0.078 (5)	0.070 (5)	0.004 (5)	-0.018 (4)	0.023 (4)
C6	0.089 (6)	0.104 (7)	0.049 (5)	-0.023 (5)	-0.008 (4)	0.007 (5)
C7	0.098 (6)	0.082 (5)	0.049 (5)	-0.007 (5)	0.008 (4)	-0.015 (4)
C8	0.079 (5)	0.056 (4)	0.058 (5)	0.005 (4)	0.010 (4)	-0.003 (3)
C9	0.051 (4)	0.041 (3)	0.044 (3)	-0.009 (3)	0.011 (3)	-0.003 (3)
C10	0.056 (4)	0.070 (5)	0.076 (5)	0.000 (4)	0.001 (4)	-0.010 (4)
C11	0.082 (6)	0.075 (6)	0.128 (8)	0.024 (5)	0.032 (6)	-0.006 (5)
C12	0.115 (7)	0.055 (4)	0.102 (7)	0.003 (5)	0.047 (6)	-0.018 (4)
C13	0.102 (7)	0.073 (5)	0.090 (6)	-0.029 (5)	0.020 (5)	-0.037 (5)
C14	0.059 (4)	0.060 (4)	0.064 (4)	-0.010 (3)	0.008 (3)	-0.010 (3)
C15	0.049 (3)	0.039 (3)	0.046 (4)	-0.003 (3)	-0.002 (3)	-0.002 (3)
C16	0.061 (4)	0.043 (3)	0.061 (4)	-0.006 (3)	0.000 (3)	0.003 (3)
C17	0.065 (5)	0.059 (4)	0.083 (6)	0.013 (4)	-0.007 (4)	-0.003 (4)
C18	0.089 (6)	0.055 (4)	0.072 (5)	0.011 (4)	-0.021 (4)	0.007 (4)
C19	0.095 (6)	0.069 (5)	0.055 (5)	-0.002 (4)	-0.002 (4)	0.014 (4)
C20	0.080 (5)	0.063 (4)	0.044 (4)	0.000 (4)	0.008 (3)	0.010 (3)

Geometric parameters (\AA , $^\circ$)

Sb1—O3	1.9503 (4)	C9—C14	1.371 (9)
Sb1—C3	2.095 (6)	C9—C10	1.377 (9)
Sb1—C9	2.095 (6)	C10—C11	1.353 (10)
Sb1—C15	2.101 (6)	C10—H10	0.9300
Sb1—O1	2.197 (4)	C11—C12	1.364 (12)
C11—C2	1.670 (9)	C11—H11	0.9300
O1—C1	1.289 (8)	C12—C13	1.357 (11)
O2—C1	1.201 (8)	C12—H12	0.9300
O3—Sb1 ⁱ	1.9503 (4)	C13—C14	1.379 (9)
C1—C2	1.525 (10)	C13—H13	0.9300
C2—H2A	0.9700	C14—H14	0.9300
C2—H2B	0.9700	C15—C16	1.361 (8)
C3—C4	1.377 (8)	C15—C20	1.368 (8)
C3—C8	1.382 (8)	C16—C17	1.381 (9)
C4—C5	1.367 (10)	C16—H16	0.9300
C4—H4	0.9300	C17—C18	1.358 (10)

C5—C6	1.370 (11)	C17—H17	0.9300
C5—H5	0.9300	C18—C19	1.360 (10)
C6—C7	1.357 (10)	C18—H18	0.9300
C6—H6	0.9300	C19—C20	1.382 (9)
C7—C8	1.372 (10)	C19—H19	0.9300
C7—H7	0.9300	C20—H20	0.9300
C8—H8	0.9300		
O3—Sb1—C3	93.36 (15)	C3—C8—H8	119.9
O3—Sb1—C9	93.77 (15)	C14—C9—C10	118.6 (6)
C3—Sb1—C9	116.3 (2)	C14—C9—Sb1	118.2 (5)
O3—Sb1—C15	93.31 (15)	C10—C9—Sb1	123.3 (5)
C3—Sb1—C15	126.3 (2)	C11—C10—C9	121.0 (7)
C9—Sb1—C15	116.4 (2)	C11—C10—H10	119.5
O3—Sb1—O1	176.64 (11)	C9—C10—H10	119.5
C3—Sb1—O1	88.53 (19)	C10—C11—C12	120.1 (8)
C9—Sb1—O1	82.89 (19)	C10—C11—H11	120.0
C15—Sb1—O1	87.80 (19)	C12—C11—H11	120.0
C1—O1—Sb1	120.4 (4)	C13—C12—C11	120.2 (7)
Sb1—O3—Sb1 ⁱ	180.00 (2)	C13—C12—H12	119.9
O2—C1—O1	127.7 (6)	C11—C12—H12	119.9
O2—C1—C2	116.8 (7)	C12—C13—C14	119.8 (8)
O1—C1—C2	115.4 (7)	C12—C13—H13	120.1
C1—C2—C11	118.7 (7)	C14—C13—H13	120.1
C1—C2—H2A	107.6	C9—C14—C13	120.3 (7)
C11—C2—H2A	107.6	C9—C14—H14	119.9
C1—C2—H2B	107.6	C13—C14—H14	119.9
C11—C2—H2B	107.6	C16—C15—C20	119.3 (6)
H2A—C2—H2B	107.1	C16—C15—Sb1	119.8 (4)
C4—C3—C8	119.0 (6)	C20—C15—Sb1	120.9 (5)
C4—C3—Sb1	118.1 (5)	C15—C16—C17	120.0 (6)
C8—C3—Sb1	122.9 (5)	C15—C16—H16	120.0
C5—C4—C3	120.2 (7)	C17—C16—H16	120.0
C5—C4—H4	119.9	C18—C17—C16	120.1 (7)
C3—C4—H4	119.9	C18—C17—H17	119.9
C4—C5—C6	120.3 (7)	C16—C17—H17	119.9
C4—C5—H5	119.8	C17—C18—C19	120.6 (7)
C6—C5—H5	119.8	C17—C18—H18	119.7
C7—C6—C5	120.1 (7)	C19—C18—H18	119.7
C7—C6—H6	120.0	C18—C19—C20	119.0 (7)
C5—C6—H6	120.0	C18—C19—H19	120.5
C6—C7—C8	120.2 (7)	C20—C19—H19	120.5
C6—C7—H7	119.9	C15—C20—C19	120.9 (7)
C8—C7—H7	119.9	C15—C20—H20	119.6
C7—C8—C3	120.2 (7)	C19—C20—H20	119.6
C7—C8—H8	119.9		

Symmetry codes: (i) $-x, -y, -z+1$.

supplementary materials

Hydrogen-bond geometry (Å, °)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C17-H17\cdots C11^{ii}$	0.93	2.93	3.592 (8)	130

Symmetry codes: (ii) $x-1, y, z$.

Fig. 1

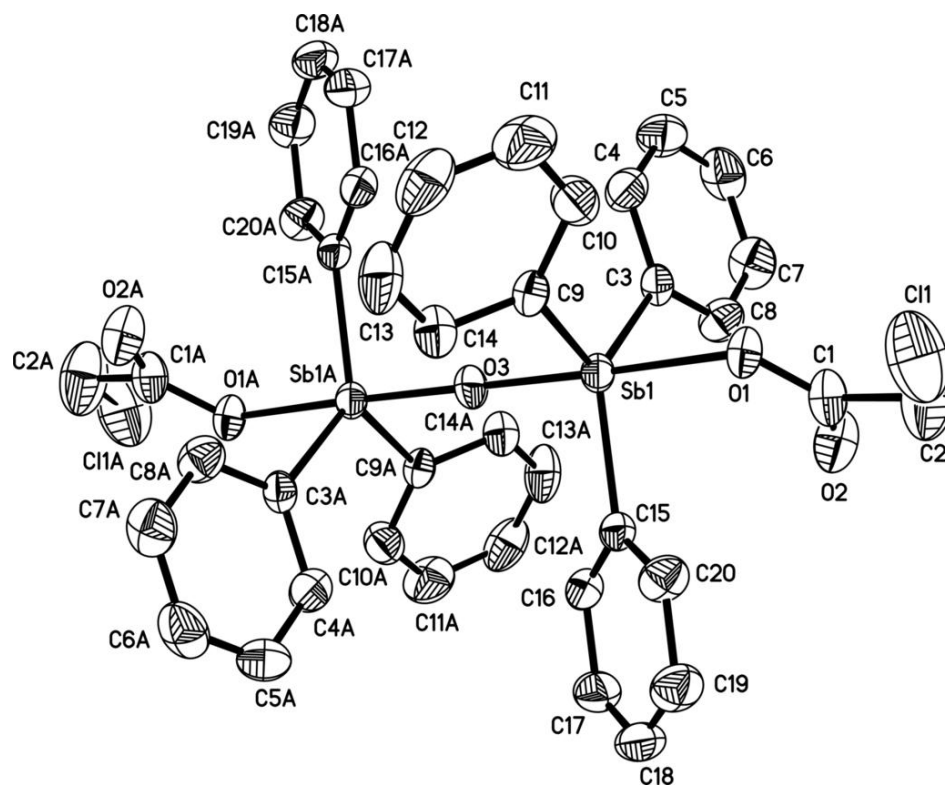


Fig. 2

