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μ-Oxido-bis[(chloroacetato-κO)-triphenylantimony(V)]

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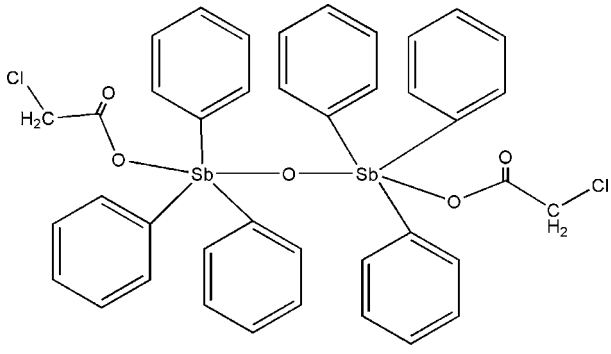
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.011 Å; R factor = 0.040; wR factor = 0.110; data-to-parameter ratio = 14.8.

The Sb atom in the centrosymmetric title complex, $[Sb_2(C_6H_5)_6(C_2H_2ClO_2)_2O]$, 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

$[Sb_2(C_6H_5)_6(C_2H_2ClO_2)_2O]$
M_r = 909.07
Monoclinic, $P2_1/c$
a = 10.4950 (12) Å
b = 19.416 (2) Å
c = 9.2584 (10) Å
β = 95.383 (2)°
V = 1878.3 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.62 mm^{-1}
T = 298 (2) K
0.41 × 0.18 × 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_{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
$\Delta\rho_{max}$ = 1.14 e Å^{-3}
$\Delta\rho_{min}$ = −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···A	D—H	H···A	D···A	D—H···A
C17—H17···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*.

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

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Gibbons, M. N. & Sowerby, D. B. (1998). *J. Organomet. Chem.* **555**, 271–278.
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Sheldrick, G. M. (1997). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

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supporting information

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μ-Oxido-bis[(chloroacetato-*κO*)triphenylantimony(V)]

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S1. 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 $[Sb(C_6H_5)_3(O_2C_2F_3)]_2O$ (*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···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,).

S2. Experimental

Chloroactic 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/di-chloromethane (1:2 *v*/*v*) solution over a period of a week (yield 85%. m.p. 470k). Anal. Calcd (%) for $C_{40}H_{34}O_5Sb_2Cl_2$(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

S3. 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_{iso}(H) = 1.2 U_{eq}(C, O) or 1.5 U_{eq}(C) for the methyl group.

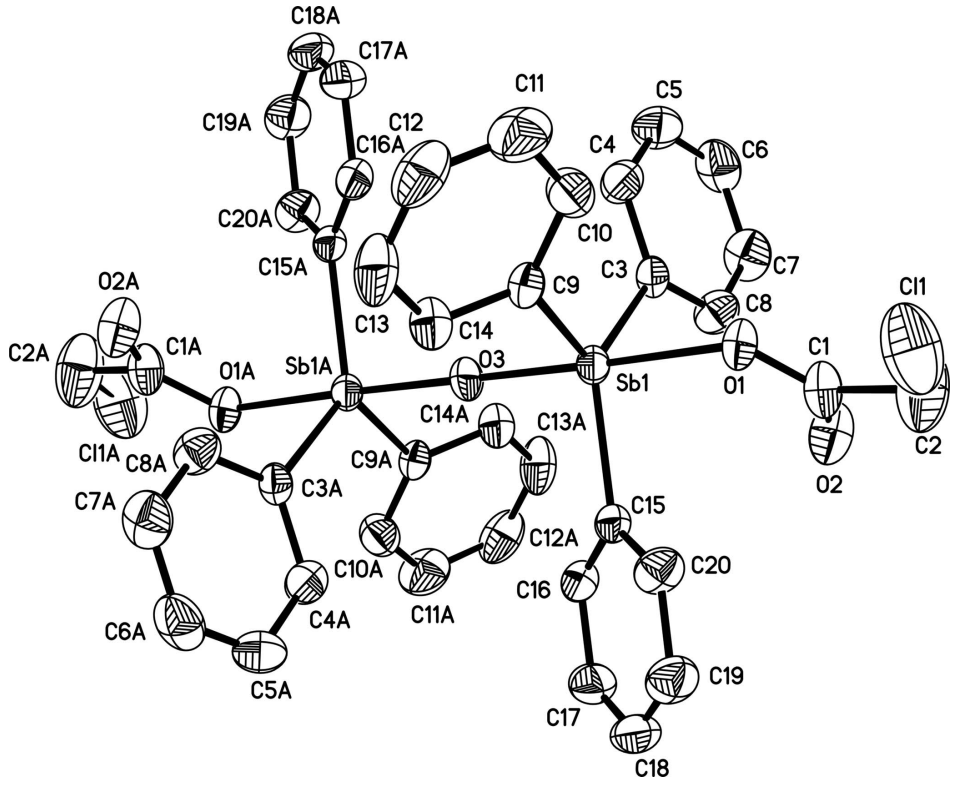

Figure 1

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

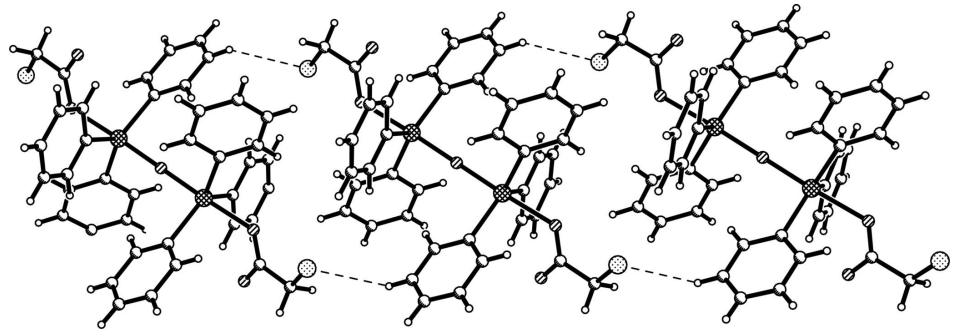

Figure 2

Crystal packing, showing a extensity polymer chain, linked by C17—H17···Cl1 hydrogen bonds (dashed lines).

μ-Oxido-bis[(chloroacetato-*κO*)triphenylantimony(V)]

Crystal data

$[Sb_2(C_6H_5)_6(C_2H_2ClO_2)_2O]$
$M_r = 909.07$
Monoclinic, $P2_1/c$
$a = 10.4950\ (12)$ Å
$b = 19.416\ (2)$ Å
$c = 9.2584\ (10)$ Å
$\beta = 95.383\ (2)°$
$V = 1878.3\ (4)$ Å^3
$Z = 2$
$F(000) = 900$
$D_x = 1.607$ 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}
$T = 298$ K
Block, colourless
0.41 × 0.18 × 0.11 mm

Data collection

Bruker SMART diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
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_{int} = 0.035
θ_{max} = 25.0°, θ_{min} = 2.0°
$h = -7 \rightarrow 12$
$k = -23 \rightarrow 21$
$l = -10 \rightarrow 11$

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 $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 1.14 e Å^{-3}
$\Delta\rho_{min}$ = −1.07 e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{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*

Atomic displacement parameters (Å²)

	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)
Cl1	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 (Å, º)

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)
Cl1—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—Cl1	118.7 (7)	C14—C13—H13	120.1
C1—C2—H2A	107.6	C9—C14—C13	120.3 (7)
Cl1—C2—H2A	107.6	C9—C14—H14	119.9
C1—C2—H2B	107.6	C13—C14—H14	119.9
Cl1—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 code: (i) $-x, -y, -z+1$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C17—H17···Cl1^{ii}	0.93	2.93	3.592 (8)	130

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