Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Bis(dimethyloxonium) pentachloridoantimonate(III)

Wen-Jie Feng, Zhan Shu, Xin-Jü Ma and Zhi-Min Jin*

College of Pharmaceutical Sciences, Zhejiang University of Technology, Hangzhou 310014, People's Republic of China
Correspondence e-mail: apharm@sina.com

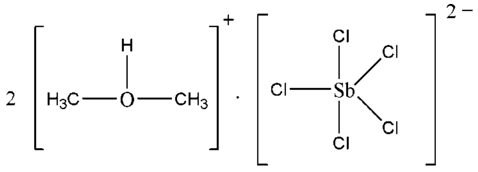
Received 30 July 2007; accepted 18 August 2007

Key indicators: single-crystal X-ray study; T = 223 K; mean σ(b–Cl) = 0.001 Å; R factor = 0.029; wR factor = 0.066; data-to-parameter ratio = 21.8.

The title compound, $(C_2H_7O)_2[SbCl_5]$, contains one-half of an $[SbCl_5]^{2-}$ anion lying on a mirror plane and one $C_2H_7O^+$ cation in the asymmetric unit. The ion pairs are linked togather by strong O—H···Cl hydrogen bonds. The $[SbCl_5]^{2-}$ anions are also linked through coordinated Sb—Cl bonds to form parallel chains.

Related literature

For related literature, see: Astruc *et al.* (1990); Bagno & Bukala (1990); Blake *et al.* (1990); Bujak & Zaleski (1998); Cai *et al.* (1995); Cheng & Niu (1995); Einstein & Jones (1973); Feng *et al.* (2007); Hall & Sowerby (1979); Ponikiewski & Rothenberger (2005); Rietz *et al.* (1978); Shikada *et al.* (1983); Spek (2003); Vojinovic *et al.* (2006); Wang *et al.* (2006); Wegman (1994); Zhou (2005).



Experimental

Crystal data

$(C_2H_7O)_2[SbCl_5]$
M_r = 393.16
Orthorhombic, *Pnma*
a = 8.5731 (15) Å
b = 11.858 (2) Å
c = 14.899 (3) Å
V = 1514.6 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.68 mm^{-1}
T = 223 (2) K
0.50 × 0.30 × 0.30 mm

Data collection

Bruker APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.397, T_{max} = 0.448
13634 measured reflections
1462 independent reflections
1408 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.066
S = 1.18
1462 reflections
67 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 1.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sb1—Cl3	2.4070 (11)	Sb1—Cl4	2.8847 (12)
Sb1—Cl2	2.4775 (12)	O1—C1	1.466 (5)
Sb1—Cl1	2.6110 (10)	O1—C2	1.480 (5)
Cl3—Sb1—Cl2	89.99 (4)	Cl2—Sb1—Cl4	171.81 (4)
Cl3—Sb1—Cl1	87.126 (19)	Cl1—Sb1—Cl4	90.65 (2)
Cl2—Sb1—Cl1	88.93 (2)	C1—O1—C2	113.8 (3)
Cl3—Sb1—Cl4	81.82 (4)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···Cl1	1.03 (5)	2.32 (5)	3.258 (4)	150 (3)

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2024).

References

Astruc, D., Lacoste, M. & Toupet, L. (1990). *J. Chem. Soc. Chem. Commun.* pp. 558–560.
Bagno, A. & Bukala, J. (1990). *J. Org. Chem.* **55**, 4284–4293.
Blake, A. J., Cradock, C. E. A. V., Ebsworth, E. A. V. & Franklin, K. C. (1990). *Angew. Chem. Int. Ed. Engl.* **29**, 76–78.
Bruker (2000). *SMART*, *SADABS*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bujak, M. & Zaleski, J. (1998). *Acta Cryst.* C**54**, 1773–1777.
Cai, G. Y., Liu, Z. M., Shi, R. M., He, C. Q., Yang, L. X., Sun, C. L. & Chang, Y. J. (1995). *Appl. Catal. A*, **125**, 29–38.
Cheng, Z. H. & Niu, Y. Q. (1995). *Coal Chem. Chem.* **71**, 31–34.
Einstein, F. W. B. & Jones, R. D. G. (1973). *Inorg. Chem.* **12**, 1690–1696.
Feng, W.-J., Wang, H.-B., Ma, X.-J., Li, H.-Y. & Jin, Z.-M. (2007). *Acta Cryst.* E**63**, m1786–m1787.
Hall, M. & Sowerby, D. B. (1979). *J. Chem. Soc. Chem. Commun.* pp. 1134–1135.
Ponikiewski, L. & Rothenberger, A. (2005). *Inorg. Chim. Acta*, **358**, 1322–1326.
Rietz, R. R., Edelstein, N. M., Ruben, H. W., Templeton, D. H. & Zalkin, A. (1978). *Inorg. Chem.* **17**, 653–658.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Shikada, T., Fujimoto, K. & Muyauchi, M. (1983). *Appl. Catal.* **7**, 361–368.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Vojinovic, K., Losehand, U. & Mitzel, N. W. (2004). *Dalton Trans.* **16**, 2578–2581.
Wang, L. G., Qi, Y., Wei, Y. X., Fang, D. R., Meng, S. H. & Liu, Z. M. (2006). *Catal. Lett.* **106**, 61–69.
Wegman, R. W. (1994). *J. Chem. Soc. Chem. Commun.* pp. 947–948.
Zhou, J. X. (2005). *Shanghai Chem. Ind.* **30**, 1–4.

doi:10.1107/S1600536807040901

supplementary materials

Acta Cryst. (2007). E**63**, m2411 [**doi:10.1107/S1600536807040901**]

Bis(dimethyloxonium) pentachloridoantimonate(III)

W.-J. Feng, Z. Shu, X.-J. Ma and Z.-M. Jin

Comment

Dimethyl ether (DME) can be used as a fuel with higher quality of heat in place of diesel oil, whereas its pollution is much lesser than the latter (Cai *et al.*, 1995; Wang *et al.*, 2006). It is also an important intermediate in fine chemical synthesis and is employed as aerosol, vesicant and low pressure refrigerant in many industries (Cheng & Niu, 1995; Zhou, 2005; Wegman, 1994; Shikada *et al.*, 1983; Bagno & Bukala, 1990). Several crystalline compounds containing DME have already been reported (Vojinovic *et al.*, 2006; Blake *et al.*, 1990; Astruc *et al.*, 1990; Ponikiewski & Rothenberger, 2005; Rietz *et al.*, 1978). Now we present here the structure of the title compound, (I).

An asymmetric unit of the title compound consists of a dimethyl ether oxonium (HDME) cation and a half $[SbCl_5]^{2-}$ anion lying on a mirror plane (Fig. 1); atoms Sb1/Cl2/Cl3/Cl4 lie in the plane. The HDME cations link to the $[SbCl_5]^{2-}$ anion by an O—H1···Cl1 hydrogen bond. The geometry of $[SbCl_5]^{2-}$ anion is distorted bipyramid. The geometrical arrangement of $[SbCl_5]^{2-}$ anion in general is like that of $[SbCl_5]^{2-}$ anion reported by Bujak (Bujak & Zaleski, 1998). The crystals of (I) and that reported by Bujak are allomers, which are similar in the crystalline structure of substances of different chemical composition. Furthermore, the coordinated Sb—Cl bond is worthy of note. A survey of some structures containing antimony and chlorine reveals that normal Sb—Cl bond lengths generally lie between 2.3 and 2.4 Å (Einstein & Jones, 1973) and those involved in bridging range from 2.8 and 3.0 Å (Hall & Sowerby, 1979; Feng *et al.*, 2007). It is noted that the upper limit for a Sb—Cl bond distance has been extended beyond 2.4 Å out and away, to 2.8847 (12) Å in the case of Sb1—Cl4 in (I).

In the crystal structure, the $[SbCl_5]^{2-}$ anions link to their neighbouring ones through coordinated bonds Sb1—Cl4 [3.295 (10) Å] forming chains and the $[SbCl_5]^{2-}$ chains are associated with HDME cations by O1—H1···Cl to strengthen the crystal structure (Fig. 2).

Experimental

The title compound was prepared by dissolving 1.5 g antimony trichloride in 10 ml absolute acetone, then adding 5 ml hydrochloride acid and 2 ml dimethylether to the solution. The solution was stirred and heated till turned clear. The reaction system was cooled slowly to room temperature. Crystals of (I) were formed by gradual evaporation of the solvents over a period of three weeks at 300 K. ^1H NMR (DMSO-d_6 / TMS): 2.55 (s, 6H, CH_3), 3.60 (s, 1H, OH).

Refinement

Two voids with volume 67 $Å^3$ were indicated by the program *PLATON* (Spek, 2003) which were ignored. H atom attached to O atom was deduced from a difference Fourier map, and incorporated in refinement freely. Others were placed in calculated positions and allowed to ride on their parent atoms at distances of 0.97 Å, with $U_{iso}(H) = 1.5\ U_{eq}(C)$. The final difference map showed a residual electron density of 1.26 e Å^{-3} lying 1.75 Å from Sb1 and was deemed meaningless.

Figures

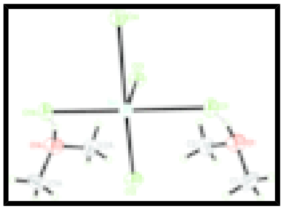


Fig. 1. The molecular structure of (I) plotted with 30% probability displacement ellipsoids; the symmetry related atoms are labeled with A. The hydrogn bonds are illustrated as dashed line.

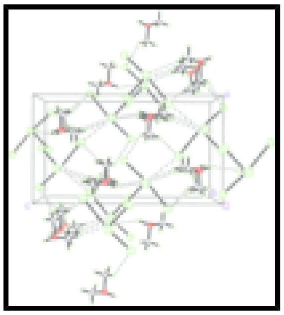


Fig. 2. The packing diagram of (I) viewed down the *b* axis. Hydrogen bonds are illustrated by dashed lines.

Bis(dimethyloxonium) pentachloridoantimonate(III)

Crystal data

$(C_2H_7O)_2[SbCl_5]$	$F_{000} = 760.0$
$M_r = 393.16$	$D_x = 1.724$ Mg m^{-3}
Orthorhombic, *Pnma*	Mo *K*α radiation λ = 0.71070 Å
Hall symbol: -P 2ac 2n	Cell parameters from 5451 reflections
a = 8.5731 (15) Å	θ = 3.2–25.3°
b = 11.858 (2) Å	μ = 2.68 mm^{-1}
c = 14.899 (3) Å	*T* = 223 (2) K
V = 1514.6 (5) Å^3	Block, colourless
Z = 4	0.50 × 0.30 × 0.30 mm

Data collection

Bruker APEX area-detector diffractometer	1462 independent reflections
Radiation source: fine-focus sealed tube	1408 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.034$
T = 223(2) K	$\theta_{max} = 25.4$°
φ and ω scans	$\theta_{min} = 3.2$°
Absorption correction: multi-scan (SADABS; Bruker, 2000)	$h = -10\rightarrow10$
$T_{min} = 0.397$, $T_{max} = 0.448$	$k = -14\rightarrow12$

13634 measured reflections	$l = -16 \rightarrow 17$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.029$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.066$	$w = 1/[\sigma^2(F_o^2) + (0.0272P)^2 + 1.5075P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.18$	$(\Delta/\sigma)_{max} = 0.001$
1462 reflections	$\Delta\rho_{max} = 1.26$ e Å^{-3}
67 parameters	$\Delta\rho_{min} = -0.48$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Sb1	0.80313 (3)	0.7500	0.602413 (18)	0.03087 (12)
Cl1	0.81044 (10)	0.53013 (7)	0.59403 (6)	0.0446 (2)
Cl2	0.60634 (14)	0.7500	0.48064 (8)	0.0487 (3)
Cl3	1.00870 (13)	0.7500	0.49238 (7)	0.0419 (3)
Cl4	1.06504 (14)	0.7500	0.72397 (8)	0.0437 (3)
O1	0.8051 (3)	0.4769 (3)	0.3795 (2)	0.0707 (9)
C1	0.6423 (5)	0.4401 (4)	0.3866 (3)	0.0746 (14)
H1C	0.6068	0.4128	0.3288	0.112*
H1D	0.5779	0.5031	0.4053	0.112*
H1E	0.6346	0.3800	0.4306	0.112*
C2	0.8291 (5)	0.5704 (4)	0.3151 (3)	0.0618 (11)
H2A	0.7672	0.6349	0.3333	0.093*
H2B	0.7974	0.5463	0.2556	0.093*
H2C	0.9386	0.5911	0.3142	0.093*
H1	0.848 (5)	0.500 (3)	0.441 (3)	0.074*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sb1	0.03218 (19)	0.03316 (19)	0.02726 (19)	0.000	0.00403 (11)	0.000
Cl1	0.0512 (5)	0.0344 (5)	0.0483 (5)	−0.0028 (3)	0.0036 (4)	0.0002 (4)
Cl2	0.0398 (6)	0.0594 (7)	0.0470 (7)	0.000	−0.0087 (5)	0.000
Cl3	0.0392 (6)	0.0542 (7)	0.0323 (6)	0.000	0.0098 (5)	0.000
Cl4	0.0496 (7)	0.0451 (7)	0.0363 (6)	0.000	−0.0014 (5)	0.000
O1	0.0618 (19)	0.086 (2)	0.0644 (19)	−0.0030 (15)	−0.0056 (14)	−0.0033 (18)
C1	0.052 (2)	0.084 (3)	0.088 (3)	−0.019 (2)	0.006 (2)	−0.035 (3)
C2	0.081 (3)	0.063 (3)	0.042 (2)	0.007 (2)	−0.003 (2)	−0.0039 (19)

Geometric parameters (Å, °)

Sb1—Cl3	2.4070 (11)	O1—H1	1.03 (5)
Sb1—Cl2	2.4775 (12)	C1—H1C	0.9700
Sb1—Cl1	2.6110 (10)	C1—H1D	0.9700
Sb1—Cl1^i	2.6110 (10)	C1—H1E	0.9700
Sb1—Cl4	2.8847 (12)	C2—H2A	0.9700
O1—C1	1.466 (5)	C2—H2B	0.9700
O1—C2	1.480 (5)	C2—H2C	0.9700
Cl3—Sb1—Cl2	89.99 (4)	O1—C1—H1C	109.5
Cl3—Sb1—Cl1	87.126 (19)	O1—C1—H1D	109.5
Cl2—Sb1—Cl1	88.93 (2)	H1C—C1—H1D	109.5
Cl3—Sb1—Cl1^i	87.126 (19)	O1—C1—H1E	109.5
Cl2—Sb1—Cl1^i	88.930 (19)	H1C—C1—H1E	109.5
Cl1—Sb1—Cl1^i	173.87 (4)	H1D—C1—H1E	109.5
Cl3—Sb1—Cl4	81.82 (4)	O1—C2—H2A	109.5
Cl2—Sb1—Cl4	171.81 (4)	O1—C2—H2B	109.5
Cl1—Sb1—Cl4	90.65 (2)	H2A—C2—H2B	109.5
Cl1^i—Sb1—Cl4	90.65 (2)	O1—C2—H2C	109.5
C1—O1—C2	113.8 (3)	H2A—C2—H2C	109.5
C1—O1—H1	111 (2)	H2B—C2—H2C	109.5
C2—O1—H1	109 (2)		

Symmetry codes: (i) x, $-y+3/2$, z.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···Cl1	1.03 (5)	2.32 (5)	3.258 (4)	150 (3)

Fig. 1

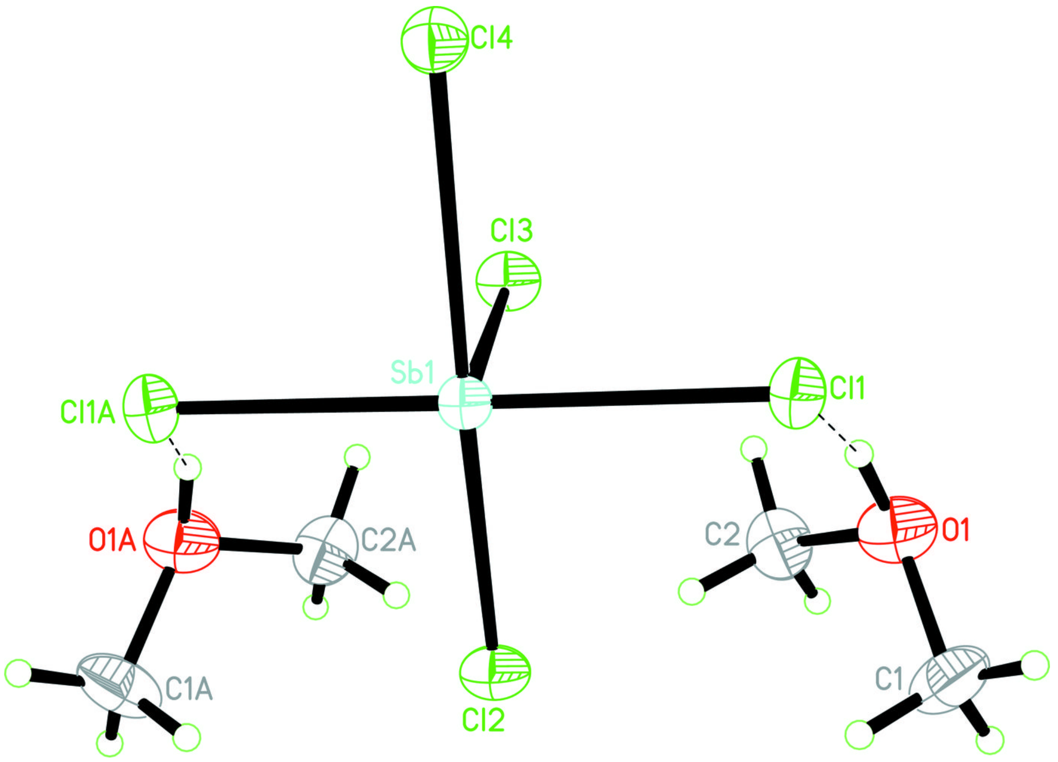


Fig. 2