The 1:1 adduct of antimony trifluoride with [1.5]dibenzothia-18-crown-6

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Key indicators

Single-crystal X-ray study
T = 123 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.019
wR factor = 0.045
Data-to-parameter ratio = 14.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound [systematic name: 6,7,9,10,12,13,15,16-octahydrodibenzo[n,q][1,4,7,10,13,16]pentaoxathiacyclooctadecine antimony(III) trifluoride clathrate], ($C_{20}H_{24}O_5S$)·SbF$_3$], the components are held together *via* electrostatic interactions between the antimony lone pair and the collective electron system of the macrocycle. The antimony trifluoride molecule is situated in a perching mode above the macrocyclic cavity, its coordination being completed by three crown ether O atoms at distances in the range 2.737 (1)–3.136 (1) Å. Neighboring 1:1 adducts are linked into chains *via* C—H···F hydrogen bonds.

Comment

For SbF$_3$ a number of complexes with crown ethers (CEs) have been synthesized and structurally characterized (Schafer *et al.*, 1990; Lipkowski *et al.*, 1996). They are perching complexes, where the neutral components are held together *via* electrostatic interactions between the antimony lone pair, pointed towards the center of the crown ring, and the collective electron system of the macrocyclic O atoms.
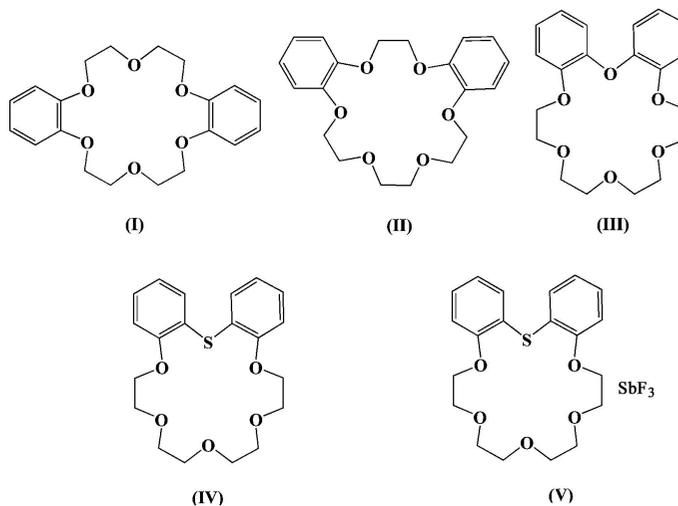
The peculiarities of the complex formation between antimony trifluoride and dibenzocrown ethers have not been studied yet. To fill this gap, we have studied the isomers of dibenzo-18-crown-6, (I)–(III), and 18-membered diphenyl sulfide CE, (IV). Under similar synthetic conditions, only crystals of complex (V) were suitable for X-ray examination. We report here the crystal structure of the title complex, (V).

It is known (Wolf *et al.*, 1987) that, when incorporated into a macrocyclic framework, sulfur prefers an exocyclic orientation of its lone pair and does not participate in the metal coordination (Campbell & Dalley, 1981; Campbell, Dalley & Simonsen, 1981; Campbell *et al.*, 1981a,b).
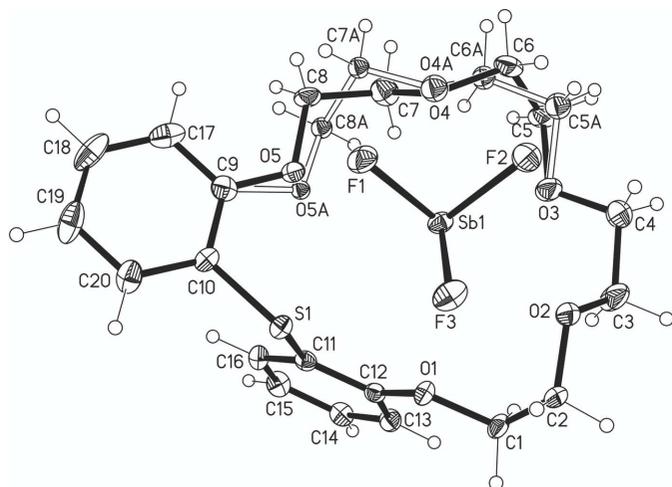
metal-organic papers

Figure 1
Top view of the title compound, with the atomic labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. The minor component of the disordered fragment is shown by open lines.
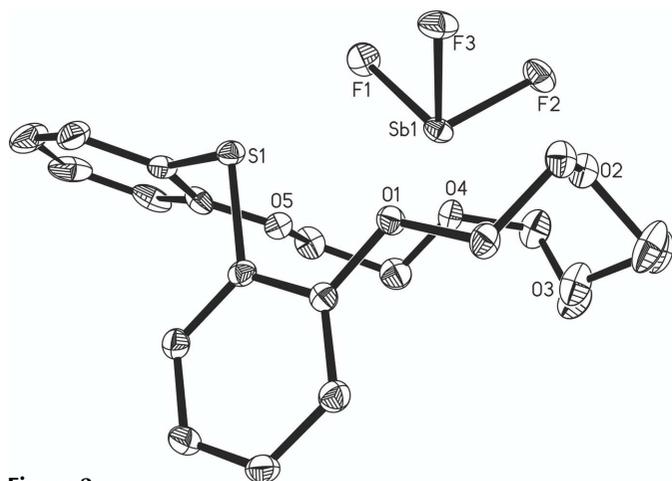

Figure 2
Side view of the title compound, with the atomic labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. The minor component of the disordered fragment and C-bound H atoms have been omitted for clarity.

Figs. 1 and 2 show the top and side *ORTEP-3* (Farrugia, 1997) views, respectively, of the title compound. The antimony trifluoride molecule is situated in a perching mode above the macrocyclic cavity and deviates 1.273 (2) Å from the least-square plane through the six (5O + 1S) heteroatoms, which are coplanar within 0.480 Å. The antimony first coordination sphere comprises three covalently bound F atoms at distances of 1.928 (1)–1.942 (1) Å, two CE O atoms, O2 and O4, at approximately equal distances of 2.737 (1) and 2.801 (9) Å, and atom O3 at a distance of 3.136 (1) Å.

The twist angle between the benzene rings in (V) is 85.06 (6)° [for comparison, the same angle is 69.2 (5)° in (III) in its complex with dithiouracil (Ganin *et al.*, 2005) and 87.5 (1)° in molecule (III) itself (Kotlyar *et al.*, 2005)].

Neighboring complexes, *via* C—H⋯F hydrogen bonds (Table 2), are linked into chains running along the [010] direction (Fig. 3).

Experimental

To a stirred solution of antimony trifluoride (89 mg, 0.5 mmol) in methanol (10 ml) a solution of (IV) (188 mg, 0.5 mmol) in methanol (10 ml) was added at a room temperature. The reaction mixture was allowed to stand until crystals were deposited. Transparent colorless crystals of (V), suitable for X-ray investigation [m.p. 463 K (decomposition)], were obtained by evaporation of a methanol solution. Analysis calculated for $C_{20}H_{24}F_3O_5SSb$: C 39.24, H 3.95, S 5.24%; found: C 39.66, H 4.04, S 5.40%.

Crystal data

$(C_{20}H_{24}O_5S)[SbF_3]$	$Z = 4$
$M_r = 555.20$	$D_x = 1.726$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 10.598$ (2) Å	$\mu = 1.44$ mm^{-1}
$b = 16.293$ (2) Å	$T = 123$ (2) K
$c = 13.286$ (2) Å	Prism, yellow
$\beta = 111.760$ (9)°	$0.15 \times 0.12 \times 0.10$ mm
$V = 2136.32$ (6) Å^3	

Data collection

Nonius KappaCCD area-detector diffractometer	4190 independent reflections
φ–ω scans	3952 reflections with $I > 2\sigma(I)$
Absorption correction: none	$R_{int} = 0.026$
16182 measured reflections	$\theta_{max} = 26.0°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0225P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.019$	$+ 2.0901P]$
$wR(F^2) = 0.046$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.07$	$(\Delta/\sigma)_{max} = 0.001$
4190 reflections	$\Delta\rho_{max} = 0.36$ e Å^{-3}
296 parameters	$\Delta\rho_{min} = -0.42$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Sb1—F2	1.9277 (11)	Sb1—F1	1.9418 (11)
Sb1—F3	1.9299 (11)		
F2—Sb1—F3	88.00 (5)	F3—Sb1—F1	87.01 (5)
F2—Sb1—F1	88.83 (5)	C10—S1—C11	103.83 (8)
O1—C1—C2—O2	−66.91 (18)	O4—C7—C8—O5	−72.4 (6)
C1—C2—O2—C3	−77.18 (19)	C7—C8—O5—C9	−176.7 (3)
C2—O2—C3—C4	176.81 (16)	C8—O5—C9—C10	−170.4 (2)
O2—C3—C4—O3	−70.5 (2)	O5—C9—C10—S1	2.8 (3)
C3—C4—O3—C5	−173.0 (2)	C9—C10—S1—C11	−76.52 (16)
C4—O3—C5—C6	−99.1 (3)	C10—S1—C11—C12	158.81 (14)
O3—C5—C6—O4	−61.7 (6)	S1—C11—C12—O1	−4.0 (2)
C5—C6—O4—C7	−63.3 (9)	C11—C12—O1—C1	−179.65 (15)
C6—O4—C7—C8	−163.8 (6)	C12—O1—C1—C2	160.99 (14)

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C1—H1A⋯F1^i	0.99	2.45	3.368 (2)	155
C13—H13A⋯F1^i	0.95	2.40	3.244 (2)	147

Symmetry code: (i) $-x + 1, y - \frac{1}{2}, -z + \frac{1}{2}$.

Atoms O4, O5, C5, C6, C7 and C8 are disordered over two positions, with site occupancies of 0.628 (1) and 0.372 (1). The minor

component was refined in an isotropic approximation. All H atoms were placed in calculated positions, with C—H distances of 0.95 or 0.99 Å, and were treated using a riding-model approximation, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *COLLECT* and *DENZO* (Otwinowski & Minor, 1997); data reduction: *COLLECT* and *DENZO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

Figure 3
Part of the crystal structure of (V), showing the linking of the complexes into chains *via* C—H···F hydrogen bonds. The F atom marked with a hash (#) is at the symmetry position $(1 - x, y - \frac{1}{2}, -z + \frac{1}{2})$.

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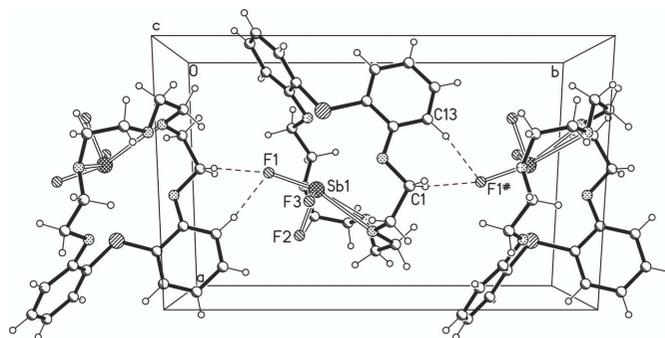Wolf, R. E. Jr, Hartman, J. R., Storey, J. M. E., Foxman, B. M. & Cooper, S. R. (1987). *J. Am. Chem. Soc.* **109**, 4328–4335.