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Syntheses and Structures of the Hexacoordinated Antimony Complexes: $Ph_3(C_2H_4O_2)Sb\cdots DMSO$, $(3\text{-}FC_6H_4)_3(C_2H_4O_2)Sb\cdots DMSO$, and $(4\text{-}MeC_6H_4)_3(C_6H_4O_2)Sb\cdots DMSO$

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Abstract—Complexes $Ph_3(C_2H_4O_2)Sb\cdots DMSO$ (**I**), $(3\text{-}FC_6H_4)_3(C_2H_4O_2)Sb\cdots DMSO$ (**II**), and $(4\text{-}MeC_6H_4)_3(C_6H_4O_2)Sb\cdots DMSO$ (**III**) are synthesized by the reactions of triarylantimony with ethylene glycol or pyrocatechol in the presence of an oxidant and dimethyl sulfoxide (DMSO). According to the X-ray diffraction analysis data, the Sb atoms in the complexes have the coordination of a distorted octahedron with the chelate cycle ($C_2H_4O_2$ or $C_6H_4O_2$) and two aryl groups in the equatorial plane, and the third aryl group and DMSO molecule occupy the axial positions. The CSbO axial angles are 173.59(5)° (**I**); 175.96(8)°, 175.93(8)° (**II**); and 174.07(8)° (**III**). The $Sb\cdots O{=}SMe_2$ distances (2.346(2)–2.407(2) Å) considerably exceed the sum of covalent radii of the atoms.

Keywords: triphenylantimony, tris(3-fluorophenyl)antimony, ethylene glycol, pyrocatechol, dimethyl sulfoxide, oxidation synthesis, hexacoordinated antimony(V) complexes, X-ray diffraction analysis

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INTRODUCTION

A significant number of structurally characterized organic complexes of hexacoordinated antimony(V) can be classified according to two types: compounds in which an increase in the coordination number is caused by the presence of the polydentate ligand [1–14] and complexes in which the coordination sphere of antimony contains a donor molecule along with the polydentate ligand [15–20]. In the complexes of the second type, a molecule of pentacoordinated antimony (with the configuration of a distorted square pyramid), which acts as a Lewis acid, and a molecule donating the electron pair can formally be distinguished. The length of the donor–acceptor $Sb\cdots L$ bond is determined by the nature of both the acceptor molecule and donor molecule L [21].

In order to establish various factors affecting the geometric parameters of the hexacoordinated antimony compounds with the additionally coordinated $Ar_3SbR\cdots L$ molecule (R is the bidentate organic ligand, and L is the donor molecule), we synthesized complexes $Ph_3(C_2H_4O_2)Sb\cdots OSMe_2$ (**I**), $(3\text{-}FC_6H_4)_3(C_2H_4O_2)Sb\cdots OSMe_2$ (**II**), and $(4\text{-}MeC_6H_4)_3(C_6H_4O_2)Sb\cdots OSMe_2$ (**III**) and studied their molecular and crystal structures.

EXPERIMENTAL

Synthesis of complex I. A 70% aqueous solution of *tert*-butyl hydroperoxide (73 mg, 0.57 mmol) was added with stirring to a solution of triphenylantimony (200 mg, 0.57 mmol), ethylene glycol (35 mg, 0.57 mmol), and DMSO (44 mg, 0.57 mmol) in benzene (10 mL). The solution was kept at 20°C for 24 h. After the slow evaporation of the solvent, the residue was recrystallized from a toluene–octane (3 : 1) mixture. The yield of colorless crystals of complex **I** was 178 mg (64%), mp = 101°C.

For $C_{22}H_{25}O_3SSb$		
Anal. calcd., %	C, 53.77	H, 5.09
Found, %	C, 53.59	H, 5.17

IR (ν, cm^{-1}): 2850, 2450, 1620, 1480, 1400, 1310, 1270, 1250, 1190, 1100, 1000, 980, 900, 880, 790, 750, 680, 630, 580, 470.

Compounds **II** and **III** were synthesized using a similar procedure.

Compound **II**: colorless transparent crystals, 82% yield, mp = 98°C.

For $C_{22}H_{22}F_3O_3SSb$

Anal. calcd., %	C, 48.44	H, 4.04
Found, %	C, 48.35	H, 4.11

IR (ν, cm^{-1}): 2830, 2670, 1590, 1460, 1400, 1260, 1205, 1060, 995, 940, 900, 850, 785, 680, 605, 440.

Compound **III**: colorless transparent crystals, 71% yield, mp = 159°C.

For $C_{29}H_{31}O_3SSb$

Anal. calcd., %	C, 59.90	H, 5.33
Found, %	C, 59.58	H, 5.93

IR (ν, cm^{-1}): 2880, 1590, 1475, 1420, 1400, 1390, 1325, 1250, 1185, 1100, 1070, 1020, 985, 940, 900, 865, 800, 740, 620, 578, 485.

The IR spectra of compounds **I–III** were recorded on a Shimadzu IRAffinity-1S IR spectrometer in KBr pellets in a range of 4000–400 cm^{-1}.

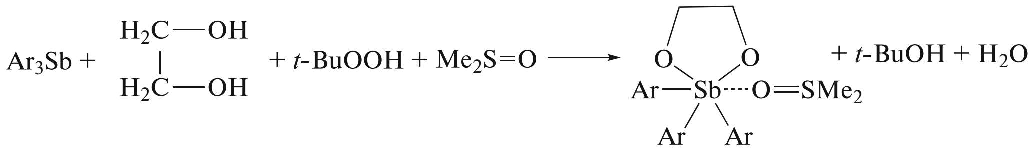
X-ray diffraction analyses of the crystals of complexes **I–III** were carried out on a D8 QUEST diffractometer (Bruker) (MoK_α radiation, λ = 0.71073 Å, graphite monochromator) at 296(2) K. Data were collected and edited, the unit cell parameters were refined, and an absorption correction was applied using the SMART and SAINT-Plus programs [22]. All calculations on structure determination and refinement were performed using the SHELXL/PC [23] and OLEX2 [24] programs. The structures were solved by a direct method and refined by least squares in the anisotropic approximation for non-hydrogen atoms. The crystallographic data and results for structure refinement are presented in Table 1. Selected bond lengths and bond angles are given in Table 2.

The full tables of coordinates of atoms, bond lengths, and bond angles were deposited with the Cambridge Crystallographic Data Centre (CIF files CCDC nos. 1576627 (**I**), 1581680 (**II**), and 1578531 (**III**); deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk/data_request/cif).

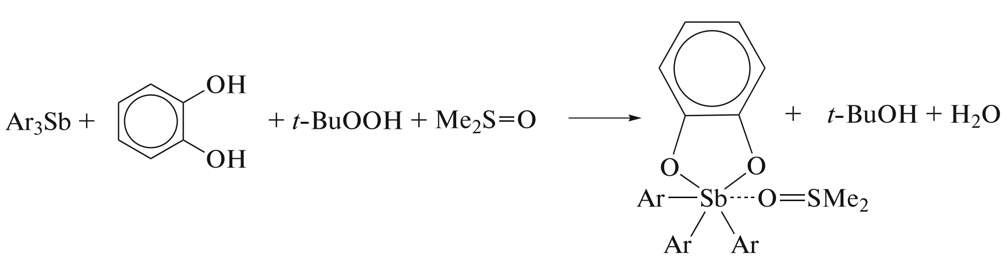
RESULTS AND DISCUSSION

It is known that the oxidation of triphenylantimony with *tert*-butyl hydroperoxide in the presence of acids HX (X = Cl, OR, OAr, OC(O)R, OSO_2R, and ON=CHR) in ether affords the Ph_3SbX_2 derivatives [25]. The complexes with the metallocycle [26, 27] or usual compounds Ph_3SbX_2 are formed [28] when bifunctional organic compounds are used in similar reactions. The introduction of a compound capable of acting as a *p*-donor ligand into the reaction mixture consisting of equimolar amounts of triarylantimony, 1,2-diol, and *tert*-butyl hydroperoxide results in the formation of the donor–acceptor complexes of hexacoordinated antimony [18].

We found that the reactions of triphenylantimony, tris(3-fluorophenyl)antimony, or tris(4-methylphenyl)antimony with ethylene glycol or pyrocatechol in the presence of *tert*-butyl hydroperoxide upon the addition of DMSO to a benzene solution occurred with the formation of the complexes containing the five-membered metallocycle and coordinated DMSO molecule.



Ar = Ph (**I**), 3-FC_6H_4 (**II**)



Ar = 4-$CH_3C_6H_4$ (**III**)

Compounds **I–III** were isolated as colorless crystals with low melting temperatures. They are highly soluble in aromatic solvents, and the solubility is aliphatic solvents is lower.

Table 1. Crystallographic data and experimental and structure refinement parameters for compounds **I–III**

Parameter	Value		
	I	**II**	**III**
FW	491.23	545.20	581.35
Crystal system	Triclinic	Triclinic	Monoclinic
T, K	296(2)	296(2)	296(2)
Space group			$P2_1/c$
Lattice parameters:			
a, Å	8.339(5)	9.267(7)	14.29(2)
b, Å	9.553(5)	13.997(13)	9.888(8)
c, Å	14.365(12)	17.441(12)	19.271(17)
α, deg	84.52(4)	98.20(4)	90.00
β, deg	89.28(3)	90.28(2)	93.37(5)
γ, deg	67.847(19)	95.92(3)	90.00
V, $Å^3$	1054.7(13)	2227(3)	2718(5)
Z	2	4	4
ρ_{calcd}, g/cm^3	1.547	1.626	1.421
μ_{Mo}, mm^{-1}	1.426	1.378	1.119
F(000)	496.0	1008	1184.0
Crystal size, mm	0.49 × 0.36 × 0.28	0.69 × 0.59 × 0.08	0.49 × 0.21 × 0.12
2θ, deg	5.56–84.46	2.923–36.319	5.9–51.4
Ranges of reflection indices	$-15 \leq h \leq 15$, $-18 \leq k \leq 18$, $-27 \leq l \leq 27$	$-15 \leq h \leq 15$, $-23 \leq k \leq 23$, $-29 \leq l \leq 29$	$-17 \leq h \leq 16$, $-12 \leq k \leq 12$, $-23 \leq l \leq 23$
Total number of reflections	95874	160169	22623
Independent reflections (R_{int})	14860 (0.0364)	21595 (0.0491)	5141 (0.0361)
Reflections with $F^2 > 2\sigma(F^2)$	12143	15210	4208
Number of refined parameters	247	565	312
GOOF	1.064	1.005	1.015
R factors for $F^2 > 2\sigma(F^2)$	$R_1 = 0.0373$, $wR_2 = 0.0988$	$R_1 = 0.0388$, $wR_2 = 0.0771$	$R_1 = 0.0252$, $wR_2 = 0.0551$
R factors for all reflections	$R_1 = 0.0516$, $wR_2 = 0.1064$	$R_1 = 0.0702$, $wR_2 = 0.0873$	$R_1 = 0.0385$, $wR_2 = 0.0603$
Residual electron density (min/max), $e/Å^3$	−1.68/3.26	−0.612/1.343	−0.41/0.62

The X-ray diffraction analyses of complexes **I–III** show that the coordination sphere of the antimony atoms represents a distorted octahedron, the equatorial plane of which is formed by the oxygen atoms of the chelate cycle ($C_2H_4O_2$ or $C_6H_4O_2$) and carbon atoms of two aryl groups (Ph, 3-FC_6H_4, or 4-MeC_6H_4). The axial positions are occupied by the carbon atom of the third aryl group and the oxygen atom of DMSO (Figs. 1–3). The crystal of compound **II** exhibits two types of crystallographically independent molecules (**A** and **B**). In each of them, the position of the fluorine atom in one of the aromatic rings is disordered over two positions. The antimony atoms deviate from the [OOCC] equatorial plane to the carbon atom by 0.288, 0.261, 0.261, and 0.279 Å in compounds **I**, **IIA**, **IIB**, and **III**, respectively.

The values of the CSbO axial angles (173.59(5)° in **I**, 175.97(7)° in **IIA**, 176.04(7)° in **IIB**, and 174.07(8)° in **III**) are smaller than the theoretical value. The sums of the OSbO, OSbC, and CSbC angles in the equatorial plane are 355.62(6)° in **I**, 356.37(8)° in **IIA**, 356.34(8)° in **IIB**, and 356.09(8)° in **III**.

Table 2. Selected interatomic distances and bond angles in the structures of compounds **I–III**

Bond	*d*, Å	Angle	ω, deg
I			
Sb(1)–O(1)	2.020(2)	O(1)Sb(1)C(1)	93.61(8)
Sb(1)–O(2)	2.013(2)	O(1)Sb(1)C(11)	162.54(7)
Sb(1)–O(3)	2.407(2)	O(2)Sb(1)O(3)	80.12(8)
Sb(1)–C(1)	2.140(2)	O(2)Sb(1)C(1)	96.47(8)
Sb(1)–C(11)	2.155(2)	O(2)Sb(1)C(21)	161.39(7)
Sb(1)–C(21)	2.147(2)	C(1)Sb(1)O(3)	173.59(5)
S(1)–O(3)	1.507(2)	C(1)Sb(1)C(11)	100.76(7)
S(1)–C(9)	1.760(4)	C(21)Sb(1)O(3)	82.62(8)
S(1)–C(10)	1.771(3)	C(21)Sb(1)C(11)	98.38(7)
O(1)–C(7)	1.398(3)	O(3)S(1)C(9)	106.85(17)
O(2)–C(8)	1.408(3)	C(9)S(1)C(10)	98.4(2)
II			
Sb(1)–O(2)	2.004(2)	O(2)Sb(1)O(3)	81.58(9)
Sb(1)–O(3)	2.016(2)	O(3)Sb(1)C(1)	162.43(7)
Sb(1)–C(1)	2.145(2)	O(2)Sb(1)C(11)	162.68(7)
Sb(1)–C(11)	2.147(2)	C(1)Sb(1)C(11)	100.93(9)
Sb(1)–C(21)	2.150(2)	C(11)Sb(1)C(21)	99.58(9)
Sb(1)–O(1)	2.359(2)	C(21)Sb(1)O(1)	175.97(7)
S(1)–O(1)	1.526(2)	O(1)Sb(1)C(9)	106.39(12)
S(1)–C(9)	1.771(3)	O(1)S(1)C(10)	103.76(12)
S(1)–C(10)	1.773(3)	C(9)S(1)C(10)	98.28(17)
Sb(2)–O(6)	2.004(2)	S(1)O(1)Sb(1)	121.75(8)
Sb(2)–O(5)	2.018(2)	C(51)Sb(2)O(4)	176.04(7)
Sb(2)–C(51)	2.142(3)	O(5)Sb(2)C(31)	162.11(7)
Sb(2)–C(41)	2.147(2)	O(6)Sb(2)O(5)	81.38(9)
Sb(2)–C(31)	2.148(2)	O(6)Sb(2)C(51)	95.29(9)
Sb(2)–O(4)	2.360(2)	O(5)Sb(2)C(51)	99.63(9)
S(2)–O(4)	1.519(2)	O(6)Sb(2)C(41)	162.82(7)
S(2)–C(39)	1.767(3)	O(5)Sb(2)C(41)	86.67(9)
S(2)–C(40)	1.775(3)	C(51)Sb(2)C(41)	98.87(9)
III			
Sb(1)–O(3)	2.077(2)	O(3)Sb(1)C(1)	161.46(8)
Sb(1)–O(2)	2.066(2)	O(3)Sb(1)C(11)	94.11(10)
Sb(1)–O(1)	2.346(2)	O(2)Sb(1)O(3)	79.55(10)
Sb(1)–C(1)	2.155(3)	O(2)Sb(1)C(11)	93.74(11)
Sb(1)–C(11)	2.152(3)	O(2)Sb(1)C(21)	160.85(8)
Sb(1)–C(21)	2.155(3)	C(1)Sb(1)O(1)	83.62(10)
S(1)–O(1)	1.529(2)	C(1)Sb(1)C(21)	101.48(12)
S(1)–C(38)	1.772(3)	C(11)Sb(1)O(1)	174.07(8)
S(1)–C(37)	1.774(3)	C(11)Sb(1)C(1)	101.12(11)
O(3)–C(32)	1.354(3)	C(21)Sb(1)O(1)	82.50(10)
O(2)–C(31)	1.355(3)	O(1)S(1)C(38)	104.77(15)

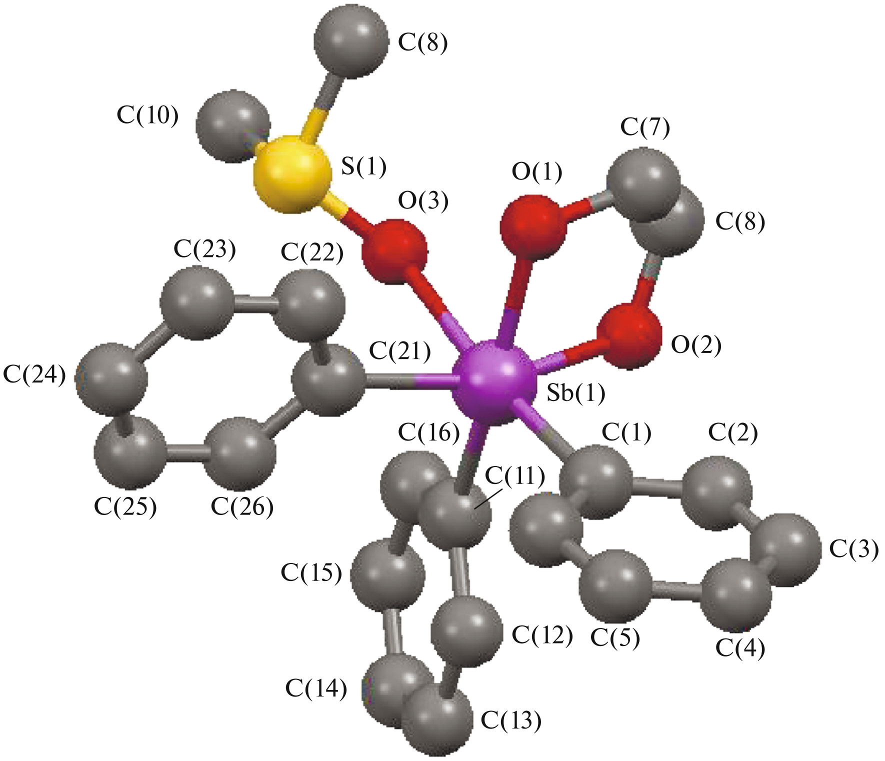


Fig. 1. General view of the molecule of compound **I** (hydrogen atoms are omitted).

The Sb—C bond lengths in complexes **I**, **IIA**, **IIB**, and **III** vary in the ranges 2.140(2)–2.155(2), 2.147(3)–2.154(3), 2.142(3)–2.147(3), and 2.152(3)–2.155(3) Å, respectively. No dependence of the bond length on its localization in the equatorial or axial position is observed. For example, the Sb—C axial bond is shorter than the equatorial distances in complex **I**, the axial and equatorial bonds are equal in length within the experimental inaccuracy in complexes **IIB** and **III**, and the axial bond is longer than the equatorial bonds in complex **IIA**.

The dithiolate ligand is asymmetrically coordinated to the antimony atom in complexes **I** and **II** (Sb—O 2.013(2) and 2.020(2), 2.005(2) and 2.016(2), 2.004(2) and 2.017(2) Å in compounds **I**, **IIA**, and **IIB**, respectively). The overall strength of ligand binding in complex **I** is lower than those in compounds **IIA** and **IIB**. The Sb—O bonds in the metallocycle of complex **III** (2.077(2), 2.066(2) Å) are somewhat longer than similar bonds in complexes **I** and **II**, which is consistent with published data [18].

The C—O distances in the five-membered metallocycles of complexes **I** and **II** are close to each other (1.398(3), 1.408(4) Å and 1.404(4), 1.406(3) Å; 1.404(4), 1.409(3) Å) and are slightly shorter than similar distances in the dithiolate complex $Ph_3(O_2C_2H_4)Sb\cdots ONC_5H_5$ (1.412(4), 1.419(6) Å) [18]. The C—O bonds in complex **III** are 1.354(3) and 1.355(3) Å, which almost coincides with the C—O bond lengths in complex $Ph_3(O_2C_6H_4)Sb\cdots OSMe_2$ (**IV**) (1.352(3) and 1.360(4) Å) [18].

Note that the configurations of the acceptor Ar_3SbR molecules are close to the square pyramidal one. The "size" of the sixth coordination site can be determined by the value of dihedral angles between the O—Sb—O and C—Sb—C planes (the O and C atoms are located in the equatorial plane of the complexes), which are 157.28°, 159.09°, and 158.12° in complexes **I**, **II**, and **III**, respectively.

The Sb···O donor–acceptor bonds with the DMSO molecule are 2.407(2) Å in **I**, 2.359(2) and 2.360(2) Å in **IIA** and **IIB**, and 2.346(2) Å in **III**. The SbOS angles are 124.62(9)°, 121.8(1)°, 122.2(1)°, and 124.7(1)° in compounds **I**, **IIA**, **IIB**, and **III**, respectively.

An increase in the strength of donor molecule binding in complex **II** compared to that in complex **I** is evidently caused by the presence of the electronegative fluorine atoms in the aryl substituents, which enhances the acceptor ability of the antimony atom. On the contrary, the presence of the electron-donor methyl groups in the aromatic rings at the antimony atom in complex **III** decreases its acceptor properties, which leads to the elongation of the Sb···O bond in complex **III** compared to the phenyl complex of a similar structure (**IV**) (2.336(2) Å) [18]. The Sb···O distances in complexes $Ph_3(O_2C_2H_4)Sb\cdots ONC_5H_5$ (**V**) (2.377(3) Å) and **IV** (2.336(2) Å) [18] are shorter than

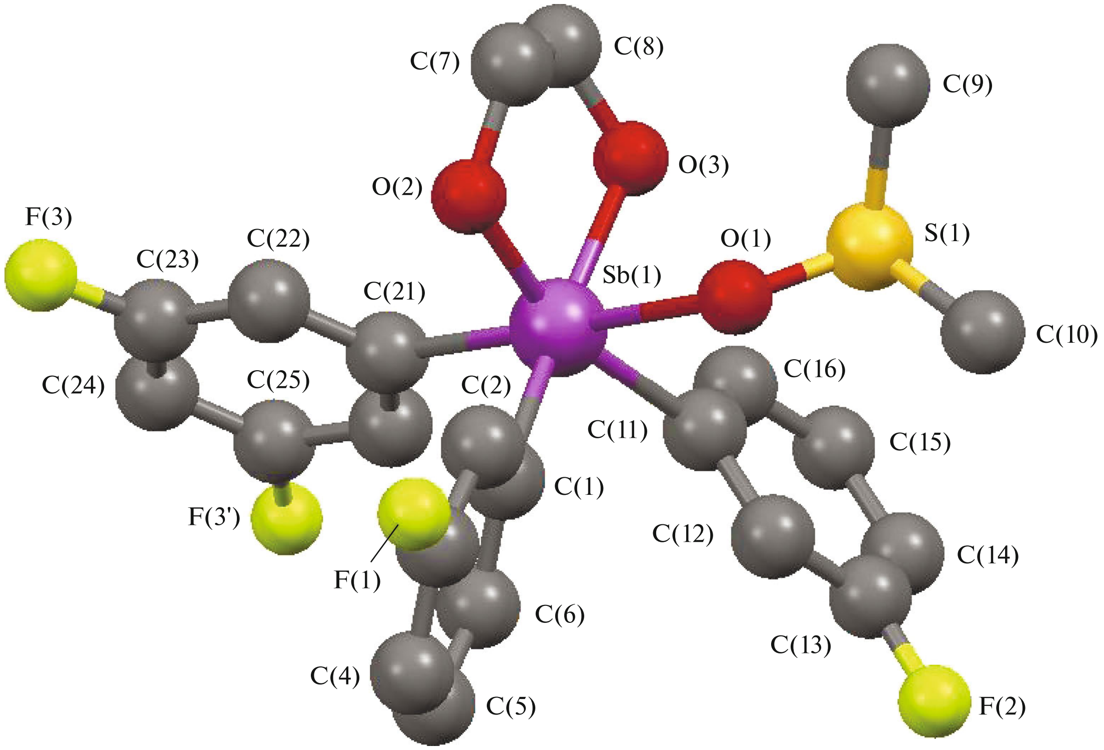


Fig. 2. General view of the molecule of compound **IIA**.

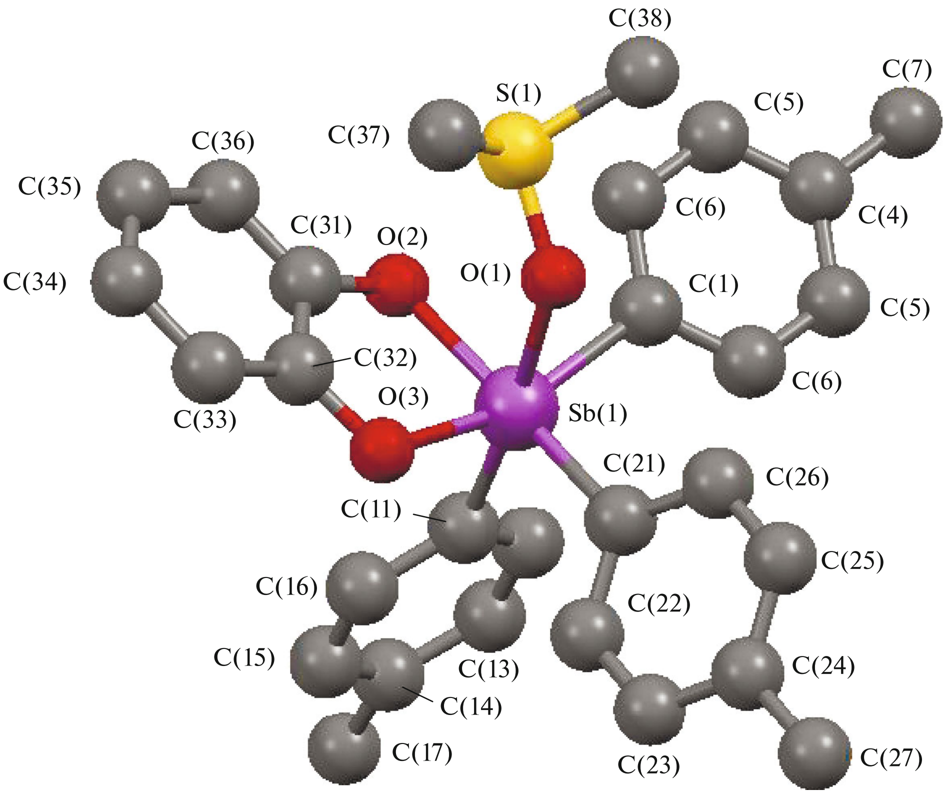


Fig. 3. General view of the molecule of compound **III**.

those in complex **I**, which shows that the strength of the intermolecular interactions depends on the nature of the donor molecule and also on the environment of the metal atom as a whole. The shortening of the Sb···O distance in compound **V** compared to a similar distance in complex $Ph_3(O_2C_6H_4)Sb\cdots ONC_5H_5$ was explained [18] by stronger acceptor properties of the *o*-OC_6H_4O group.

The coordination of the DMSO molecule with the antimony atoms in the acceptor Ar_3SbR molecules results in some elongation of the O–S bonds to 1.507(2) Å in **I**, 1.526(2) in **IIA**, 1.519(2) Å in **IIB**, and 1.529(2) Å in **III**, exceeding a similar distance in the molecule of uncoordinated DMSO (1.497 Å [29]). The S–C distances in complexes **I**, **II**, and **III** (1.760(4), 1.771(3); 1.767(3)–1.773(3); 1.772(3), 1.774(3) Å) also differ from similar distances in uncoordinated DMSO (1.809 Å [29]).

In the crystals of complexes **I** and **III**, the molecules are joined into dimers to form macrocycles by weak hydrogen bonds O···H–C, where O is the oxygen atom of the chelate ligand and H is the hydrogen atom of the methyl group of DMSO (O···H is 2.60 and 2.69 Å in **I** and **III**, respectively). The dimers form piles oriented along the crystallographic axis *b*. The structural organization in complex **II** is more complicated, because intermolecular contacts of both H···O and H···F types take place.

Thus, donor–acceptor complexes **I–III** are formed by the acceptor molecule Ar_3SbR in which the antimony atom has the coordination of a distorted tetragonal pyramid and by the DMSO donor molecule added from the side of the pyramid base. An analysis of the geometric characteristics of complexes **I–III** shows that the strength of the donor–acceptor interactions Sb···O is primarily determined by the electronic factors, namely, by the donor and acceptor properties of the aryl and chelate ligands at the antimony atom.

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