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Tris(*para*-Tolyl)- and Tris(4-Fluorophenyl)antimony Diaroxides: Syntheses and Structures

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Abstract—Bis(4-bromophenoxy)tris(*para*-tolyl)antimony (**I**), bis(4-nitrophenoxy)tris(*para*-tolyl)antimony (**II**), bis(4-nitrophenoxy)tris(4-fluorophenyl)antimony (**III**), bis(2,3,4,5,6-pentafluorophenoxy)tris(4-fluorophenyl)antimony (**IV**), and bis(2,3,4,5,6-pentachlorophenoxy)tris(4-fluorophenyl)antimony (**V**) (CIF files CCDC 1470829 (**I**), 1474589 (**II**), 1062337 (**III**), 1470476 (**IV**), and 1472954 (**V**)) are synthesized in high yields by the reactions of tris(*para*-tolyl)- and tris(4-fluorophenyl)antimony with 4-bromo-, 4-nitro-, 2,3,4,5,6-pentafluoro-, and 2,3,4,5,6-pentachlorophenol, respectively, in diethyl ether in the presence of *tert*-butyl hydroperoxide. The Sb atoms in compounds **I**–**V** have a distorted trigonal bipyramidal coordination with the aroxy groups in the axial positions (angles OSbO 174.08(11)°–179.4(5)°). The average Sb–C bond lengths in compounds **I**–**V** are similar and independent of the nature of the *para*-substituent in the aryl rings. The Sb–O distances are close to the sum of covalent radii of Sb and O atoms. Hydrogen bonds H···F are involved in the formation of the crystal structures of compounds **III**–**V**.

Keywords: diaroxide, tris(*para*-tolyl)antimony, tris(4-fluorophenyl)antimony, synthesis, structure

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INTRODUCTION

It is known that the trigonal bipyramidal coordination mode of the central atom in the aryl antimony(V) derivatives of the general formula Ar_3SbX_2, as a rule, is poorly distorted. Appreciable deviations of the bond angles at the antimony atom from theoretical values are mainly due to intramolecular interactions, for example, due to the tendency of the electronegative ligand X to additional coordination [1]. Some elongation of the Sb–O distances is observed in a series of compounds Ar_3SbX_2, where ligand X bonded to the antimony atom through the oxygen atom is a residue of alcohols, phenols, oximes, carboxylic acids, and sulfo acids. No regular influence of the nature of substituents in the aryl groups on the length of the Sb–C equatorial bonds was revealed. For example, in molecules of triarylantimony diacetates $Ar_3Sb[OC(O)CH_3]_2$, where Ar = C_6H_5 [2], $C_6H_4CH_3$-4, and $C_6H_4CF_3$-4 [3], the average Sb–C bond lengths in the phenyl and trifluoromethylphenyl derivatives are equal within the experimental inaccuracy and longer (by 0.01 Å) than those in the tolyl derivative. Note that the average Sb–O distances, on the contrary, coincide in the tolyl and trifluoromethylphenyl compounds and exceed that in the phenyl analog.

Continuing the studies of the influence of the nature of aroxyl and aryl ligands at the antimony atom on the geometric characteristics of triarylantimony diaroxide molecules, in this work we synthesized and structurally characterized bis(4-bromophenoxy)tris(*para*-tolyl)antimony (**I**), bis(4-nitrophenoxy)tris(*para*-tolyl)antimony (**II**), bis(4-nitrophenoxy)tris(4-fluorophenyl)antimony (**III**), bis(2,3,4,5,6-pentafluorophenoxy)tris(4-fluorophenyl)antimony (**IV**), and bis(2,3,4,5,6-pentachlorophenoxy)tris(4-fluorophenyl)antimony (**V**).

EXPERIMENTAL

Synthesis of complex I. A mixture of tris(*para*-tolyl)antimony (150 mg, 0.38 mmol), 4-bromophenol (131 mg, 0.76 mmol), and a 70% aqueous solution of *tert*-butyl hydroperoxide (49 mg, 0.38 mmol) in diethyl ether (30 mL) was stored for 24 h at 20°C. After the solvent removal, the solid residue was recrystallized from a toluene–octane (2 : 1 vol/vol) mixture. The yield of pale yellow crystals of compound **I** was 0.275 mg (98%), mp = 165°C.

For $C_{33}H_{29}O_2Br_2Sb$		
Anal. calcd., %:	C, 53.62;	H, 3.96.
Found, %:	C, 53.27;	H, 4.03.

IR (ν, cm^{-1}): 3064, 3020, 2970, 2951, 2918, 2862, 2810, 2727, 2654, 2642, 2605, 2584, 2555, 2515, 2418,

2384, 2308, 2285, 2254, 2204, 2189, 2144, 2088, 2077, 2042, 2015, 2007, 1992, 1967, 1923, 1903, 1888, 1867, 1828, 1791, 1749, 1716, 1575, 1560, 1479, 1456, 1436, 1419, 1394, 1361, 1338, 1313, 1247, 1211, 1188, 1159, 1118, 1093, 1068, 1037, 1014, 999, 966, 945, 927, 842, 823, 792, 732, 698, 640, 624, 586, 526, 501, 480, 462, 418.

Compounds **II**–**V** were synthesized similarly.

Compound **II**: 97% yield, yellow crystals, mp = 210°C.

For $C_{33}H_{29}N_2O_6Sb$		
Anal. calcd., %:	C, 59.04;	H, 4.36.
Found, %:	C, 58.93;	H, 4.53.

IR (ν, cm^{-1}): 3059, 3018, 2974, 2918, 2864, 2821, 2771, 2750, 2690, 2636, 2600, 2534, 2439, 2405, 2382, 2308, 2285, 2216, 1647, 1583, 1500, 1487, 1458, 1396, 1334, 1288, 1278, 1257, 1211, 1188, 1170, 1111, 1060, 1037, 1014, 1001, 943, 864, 846, 823, 804, 794, 756, 698, 653, 632, 584, 518, 478, 418.

Compound **III**: 61% yield, yellow crystals, mp = 188°C.

For $C_{30}H_{20}N_2O_6F_3Sb$		
Anal. calcd., %:	C, 52.73;	H, 2.96.
Found, %:	C, 52.49;	H, 3.02.

IR (ν, cm^{-1}): 3097, 3070, 3039, 2827, 1581, 1489, 1396, 1338, 1288, 1257, 1232, 1161, 1111, 1062, 1012, 866, 835, 819, 756, 694, 657, 634, 543, 511, 418.

Compound **IV**: 99% yield, colorless crystals, mp = 129°C.

For $C_{30}H_{12}O_2F_{13}Sb$		
Anal. calcd., %:	C, 46.60;	H, 1.57.
Found, %:	C, 46.45;	H, 1.63.

IR (ν, cm^{-1}): 3120, 3068, 2465, 1890, 1653, 1585, 1504, 1490, 1467, 1436, 1396, 1307, 1244, 1163, 1064, 1012, 991, 935, 825, 727, 628, 570, 509, 468, 420.

Compound **V**: 99% yield, colorless crystals, mp = 250°C (with decomp.).

For $C_{30}H_{12}O_2F_3Cl_{10}Sb$		
Anal. calcd., %:	C, 38.43;	H, 1.29.
Found, %:	C, 38.28;	H, 1.34.

IR (ν, cm^{-1}): 3101, 3068, 3035, 2960, 2908, 2843, 2781, 2727, 2654, 2561, 2517, 2449, 2360, 2331, 2266, 2225, 2177, 2040, 1581, 1531, 1487, 1388, 1355, 1303, 1230, 1222, 1163, 1130, 1068, 987, 935, 823, 773, 731, 715, 586, 511, 418.

The IR spectra of compounds **I**–**V** 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 compounds **I**–**V** were carried out on a D8 Quest diffractometer (Bruker) (MoK_α radiation, λ = 0.71073 Å, graphite monochromator). The data were collected and edited, the unit cell parameters were refined, and an absorption correction was applied using the SMART and SAINT-Plus programs [4]. All calculations on structure determination and refinement were performed using the SHELXL/PC [5] and OLEX2 programs [6]. The structures were solved by a direct method and refined by least squares in the anisotropic approximation for non-hydrogen atoms. The main crystallographic data and refinement results for the structures of compounds **I**–**V** are presented in Table 1. Selected bond lengths and bond angles are given in Table 2.

The full tables of atomic coordinates, bond lengths, and bond angles were deposited with the Cambridge Crystallographic Data Centre (CIF files CCDC 1470829 (**I**), 1474589 (**II**), 1062337 (**III**), 1470476 (**IV**), and 1472954 (**V**); deposit@ccdc.cam.ac.uk; http://www.ccdc.cam.ac.uk).

RESULTS AND DISCUSSION

Triarylantimony diaroxides were synthesized in high yields by the oxidative addition reaction [7] from triarylantimony and phenol in the presence of *tert*-butyl hydroperoxide in diethyl ether.

$$Ar_3Sb + 2HOAr' + t\text{-}BuOOH \rightarrow Ar_3Sb(OAr')_2 + t\text{-}BuOH + H_2O,$$

Ar = *p*-Tol; Ar' = C_6H_4Br-4 (**I**), $C_6H_4(NO_2$-4) (**II**),

Ar = 4-FC_6H_4; Ar' = $C_6H_4(NO_2$-4) (**III**), C_6F_5 (**IV**), C_6Cl_5 (**V**).

According to the X-ray diffraction data, in compounds **I**–**V** the antimony atoms have slightly distorted trigonal bipyramidal coordination with the oxygen atoms of the aroxy groups in the axial positions (Figs. 1–4). The crystals of compounds **III** and **IV** (Figs. 2, 3) contain two types of crystallographically independent molecules (**A** and **B**), the geometric parameters of which differ insignificantly. The aryl rings in the molecules of compounds **I**–**V** have a "propeller" conformation relative to the equatorial plane (C_3), and the values of the dihedral angles between the planes are different. In the molecules of compounds **IVA** and **IVB**, the planes of the C(1)–C(6) and C(61)–C(66) aryl groups almost coincide with the equatorial planes (the corresponding dihedral angles are 7.01° and 5.14°). The C(21)–C(26) and C(1)–C(6) planes in the molecules

Table 1. Crystallographic data and experimental and refinement parameters for the structures of compounds **I**–**V**

Parameter	Value				
	I	**II**	**III**	**IV**	**V**
FW	739.13	671.33	683.23	1546.33	468.82
Crystal system	Tetragonal	Monoclinic	Triclinic	Triclinic	Monoclinic
Space group	$P4_1$	$P2_1/c$	$P\bar{1}$	$P\bar{1}$	$C2/c$
a, Å	9.4445(4)	9.6944(4)	10.0009(18)	11.1514(7)	19.5509(6)
b, Å	9.4445(4)	25.6386(9)	10.2829(19)	11.4162(7)	16.2475(5)
c, Å	34.6574(19)	12.9758(5)	14.882(3)	12.9511(8)	10.8305(3)
α, deg	90.00	90.00	108.277(12)	104.365(2)	90.00
β, deg	90.00	108.604(2)	102.798(12)	112.838(2)	102.6680(10)
γ, deg	90.00	90.00	93.523(11)	99.514(3)	90.00
V, Å^3	3091.4(3)	3056.6(2)	1402.9(4)	1407.40(16)	3356.60(17)
Z	4	4	2	1	8
ρ_{calcd}, g/cm^3	1.588	1.459	1.617	1.8243	1.855
μ, mm^{-1}	3.504	0.949	1.050	1.095	1.665
F(000)	1456.0	1360.0	680.0	751.4	1824.0
Crystal size, mm	1.04 × 0.71 × 0.6	0.7 × 0.38 × 0.3	0.32 × 0.2 × 0.12	0.77 × 0.49 × 0.37	0.5 × 0.32 × 0.13
Range of data collection over 2θ, deg	6.54–40.44	6.64–55.78	4.22–53	5.6–77.84	6.88–53.54
Ranges of reflection indices	$-9 \le h \le 8$, $-9 \le k \le 9$, $-33 \le l \le 33$	$-12 \le h \le 12$, $-30 \le k \le 33$, $-17 \le l \le 15$	$-12 \le h \le 12$, $-12 \le k \le 12$, $-18 \le l \le 18$	$-19 \le h \le 19$, $-20 \le k \le 20$, $-22 \le l \le 22$	$-24 \le h \le 24$, $-20 \le k \le 20$, $-12 \le l \le 13$
Measured reflections	16749	12734	21764	137347	30117
Independent reflections	2939 ($R_{int} = 0.0394$)	6371 ($R_{int} = 0.0243$)	10830 ($R_{int} = 0.0523$)	31881 ($R_{int} = 0.0404$)	3565 ($R_{int} = 0.0287$)
Refinement variables	346	382	757	828	210
GOOF	1.095	1.118	0.975	1.109	1.051
R factors for $F^2 > 2\sigma(F^2)$	$R_1 = 0.0251$ $wR_2 = 0.0524$	$R_1 = 0.0451$ $wR_2 = 0.0864$	$R_1 = 0.0379$ $wR_2 = 0.0721$	$R_1 = 0.0528$ $wR_2 = 0.1219$	$R_1 = 0.0317$ $wR_2 = 0.0772$
R factors for all reflections	$R_1 = 0.0275$ $wR_2 = 0.0532$	$R_1 = 0.0670$ $wR_2 = 0.0931$	$R_1 = 0.0676$ $wR_2 = 0.0816$	$R_1 = 0.1090$ $wR_2 = 0.1643$	$R_1 = 0.0377$ $wR_2 = 0.0826$
Residual electron density (min/max), *e*/Å^3	0.27/–0.20	0.52/–0.50	0.86/–0.51	1.55/–1.05	0.63/–0.67

Table 2. Selected bond lengths and bond angles in the structures of compounds **I**–**V**

Bond	*d*, Å	Angle	ω, deg
		I	
Sb(1)–O(1)	2.064(4)	O(1)Sb(1)C(1)	88.5(2)
Sb(1)–O(2)	2.044(4)	O(1)Sb(1)C(11)	85.71(19)
Sb(1)–C(1)	2.087(6)	O(1)Sb(1)C(21)	93.1(2)
Sb(1)–C(11)	2.098(7)	O(2)Sb(1)O(1)	179.21(17)
Sb(1)–C(21)	2.098(7)	O(2)Sb(1)C(1)	92.3(2)
Br(1)–C(34)	1.928(7)	O(2)Sb(1)C(11)	93.9(2)
Br(2)–C(44)	1.895(7)	O(2)Sb(1)C(21)	86.5(2)
O(1)–C(31)	1.344(7)	C(1)Sb(1)C(11)	122.7(3)
O(2)–C(41)	1.353(8)	C(1)Sb(1)C(21)	122.4(3)
C(14)–C(17)	1.517(1)	C(11)Sb(1)C(21)	114.8(3)
		II	
Sb(1)–O(1)	2.106(3)	O(1)Sb(1)C(1)	93.67(12)
Sb(1)–O(4)	2.095(3)	O(4)Sb(1)O(1)	177.69(11)
Sb(1)–C(21)	2.106(4)	O(4)Sb(1)C(21)	88.99(13)
Sb(1)–C(11)	2.103(3)	O(4)Sb(1)C(11)	93.12(13)
Sb(1)–C(1)	2.120(4)	O(4)Sb(1)C(1)	87.63(12)
C(34)–N(1)	1.451(6)	C(21)Sb(1)O(1)	88.71(14)
C(44)–N(2)	1.468(5)	C(21)Sb(1)C(1)	121.79(13)
O(4)–C(41)	1.314(4)	C(11)Sb(1)O(1)	88.18(12)
O(1)–C(31)	1.315(5)	C(11)Sb(1)C(21)	126.96(14)
C(14)–C(17)	1.520(6)	C(11)Sb(1)C(1)	111.25(14)
		IIIA	
Sb(1)–O(1)	2.078(9)	O(4)Sb(1)O(1)	178.4(5)
Sb(1)–O(4)	2.059(11)	C(11)Sb(1)O(1)	92.4(5)
Sb(1)–C(11)	2.047(15)	C(11)Sb(1)O(4)	86.3(5)
Sb(1)–C(1)	2.016(14)	C(1)Sb(1)O(1)	86.7(5)
Sb(1)–C(21)	2.006(16)	C(1)Sb(1)O(4)	93.0(5)
N(2)–C(44)	1.52(2)	C(1)Sb(1)C(11)	115.0(5)
C(34)–N(1)	1.42(2)	C(21)Sb(1)O(1)	90.3(5)
F(3)–C(24)	1.30(2)	C(21)Sb(1)O(4)	91.2(5)
F(2)–C(14)	1.31(2)	C(21)Sb(1)C(11)	121.7(6)
F(1)–C(4)	1.327(19)	C(21)Sb(1)C(1)	123.3(6)
		IIIB	
Sb(2)–O(7)	2.077(8)	O(7)Sb(2)C(71)	92.1(5)
Sb(2)–O(10)	2.062(11)	O(7)Sb(2)C(61)	87.1(5)
Sb(2)–C(71)	2.155(14)	O(7)Sb(2)C(51)	87.8(5)
Sb(2)–C(61)	2.177(12)	O(10)Sb(2)O(7)	179.4(5)
Sb(2)–C(51)	2.157(13)	O(10)Sb(2)C(71)	87.5(5)
F(5)–C(64)	1.402(18)	O(10)Sb(2)C(61)	93.4(6)
F(6)–C(74)	1.382(19)	O(10)Sb(2)C(51)	92.1(6)
F(4)–C(54)	1.41(2)	C(71)Sb(2)C(61)	114.2(6)
C(84)–N(3)	1.493(19)	C(71)Sb(2)C(51)	123.9(6)
N(4)–C(94)	1.42(2)	C(51)Sb(2)C(61)	121.8(6)

Table 2. (Contd.)

Bond	*d*, Å	Angle	ω, deg
		IVA	
Sb(1)–O(1)	2.075(4)	C(21)Sb(1)O(1)	87.98(18)
Sb(1)–C(21)	2.099(6)	C(1)Sb(1)O(1)	91.15(18)
Sb(1)–C(1)	2.057(5)	C(1)Sb(1)C(21)	119.9(2)
Sb(1)–O(2)	2.091(3)	O(2)Sb(1)O(1)	178.67(14)
Sb(1)–C(11)	2.081(5)	O(2)Sb(1)C(21)	91.31(17)
O(1)–C(31)	1.321(5)	O(2)Sb(1)C(1)	90.19(16)
O(2)–C(41)	1.334(6)	C(11)Sb(1)O(1)	92.82(18)
F(8)–C(36)	1.342(8)	C(11)Sb(1)C(21)	118.8(2)
F(6)–C(34)	1.363(6)	C(11)Sb(1)C(1)	121.2(2)
F(5)–C(33)	1.353(7)	C(11)Sb(1)O(2)	86.54(15)
		IVB	
Sb(2)–O(3)	2.092(4)	O(4)Sb(2)O(3)	177.03(13)
Sb(2)–O(4)	2.070(3)	C(51)Sb(2)O(3)	90.57(19)
Sb(2)–C(51)	2.091(5)	C(51)Sb(2)O(4)	87.18(17)
Sb(2)–C(61)	2.144(6)	C(61)Sb(2)O(3)	90.54(18)
Sb(2)–C(71)	2.114(7)	C(61)Sb(2)O(4)	92.29(16)
O(3)–C(81)	1.333(8)	C(61)Sb(2)C(51)	121.7(2)
O(4)–C(91)	1.334(6)	C(71)Sb(2)O(3)	87.9(2)
F(17)–C(82)	1.409(8)	C(71)Sb(2)O(4)	91.46(19)
F(19)–C(84)	1.347(8)	C(71)Sb(2)C(51)	117.5(2)
F(20)–C(85)	1.318(8)	C(71)Sb(2)C(61)	120.7(2)
		V	
Sb(1)–O(1)	2.101(2)	O(1')Sb(1)O(1)	174.08(11)
Sb(1)–O(1')	2.101(2)	C(1')Sb(1)O(1')	89.50(10)
Sb(1)–C(1)	2.099(3)	C(1')Sb(1)O(1)	93.79(10)
Sb(1)–C(1')	2.099(3)	C(1)Sb(1)O(1)	89.50(10)
Sb(1)–C(11)	2.097(4)	C(1)Sb(1)O(1')	93.79(10)
Cl(3)–C(24)	1.729(3)	C(1')Sb(1)C(1)	112.49(17)
Cl(4)–C(25)	1.717(3)	C(11)Sb(1)O(1')	87.04(6)
F(1)–C(4)	1.360(4)	C(11)Sb(1)O(1)	87.04(6)
F(2)–C(14)	1.356(5)	C(11)Sb(1)C(1)	123.75(8)
O(1)–C(21)	1.323(4)	C(11)Sb(1)C(1')	123.76(8)

of compounds **I** and **II** are nearly perpendicular to the planes (C_3) (the corresponding dihedral angles are 73.12° and 80.66°). The sums of the CSbC equatorial angles are close to the theoretical value, whereas individual values can differ substantially from 120° (122.7(3)°, 122.4(3)°, 114.8(3)° in **I**; 121.79(13)°, 126.96(14)°, 111.25(14)° in **II**; 115.0(5)°, 121.7(6)°, 123.3(6)° in **IIIA**; 114.2(6)°, 123.9(6)°, 121.8(6)° in **IIIB**; 119.9(2)°, 118.8(2)°, 121.2(2)° in **IVA**; 121.7(2)°, 117.5(2)°, 120.7(2)° in **IVB**; 112.49(17)°, 123.75(8)°, 123.76(8)° in **V**). The antimony atoms shift from the equatorial plane (C_3) by 0.034 (**I**), 0.003 (**II**), 0.008 (**IIIA**), 0.038 (**IIIB**), 0.024 (**IVA**), and 0.012 Å (**IVB**). The metal atom in a molecule of compound **V** lies in the equatorial plane. The angles between the axial and equatorial substituents CSbO vary within 85.7(2)°–93.9(2)° (**I**), 87.63(12)°–93.67(12)° (**II**), 86.3(5)°–93.0(5)° (**IIIA**), 87.1(5)°–93.4(6)° (**IIIB**), 86.54(15)°–92.82(18)° (**IVA**), 87.18(17)°–92.29(16)° (**IVB**), and 87.04(6)°–93.79(10)° (**V**).

The average Sb–C bond lengths in the molecules of compounds **I**–**V** differ slightly (2.094(7) for **I**, 2.110(1)

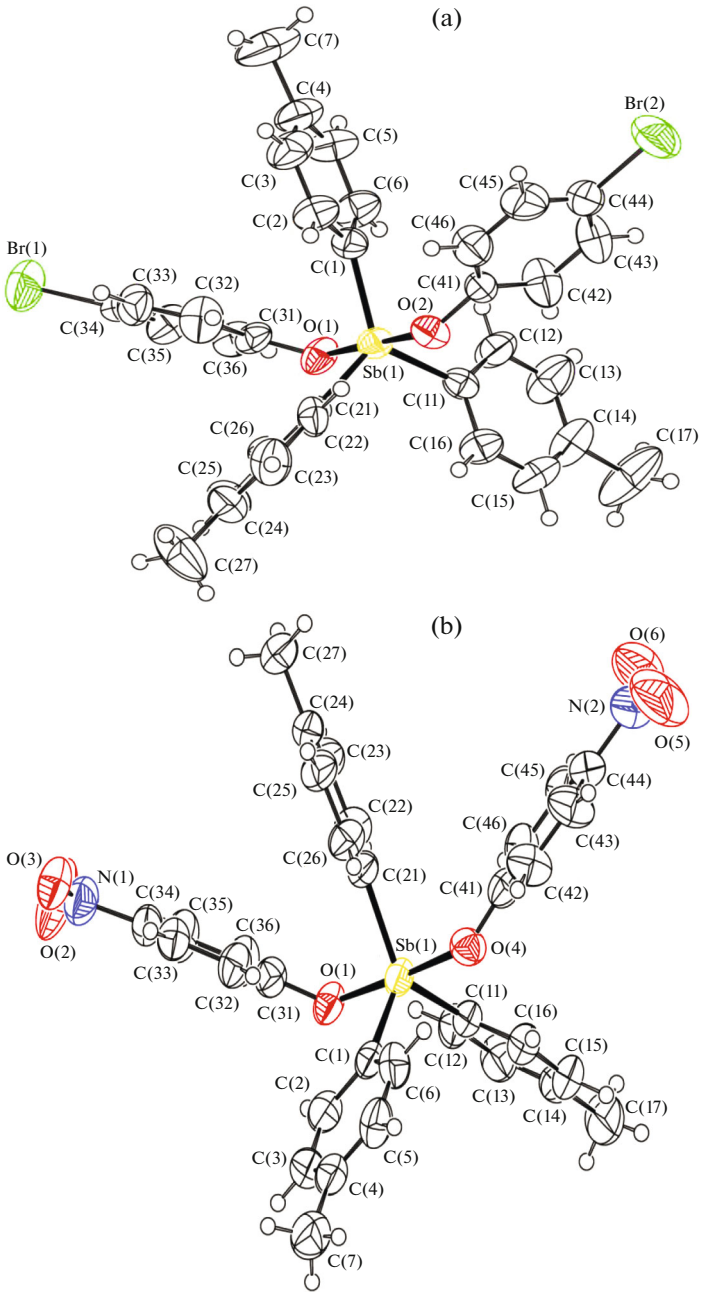

Fig. 1. Molecular structures of compounds (a) **I** and (b) **II**.

for **II**, 2.093(2) for **III**, 2.097(5) for **IV**, and 2.098(3) Å for **V**). The average Sb–O bond lengths are 2.054(4) (**I**), 2.100(3) (**II**), 2.069(9) (**III**), 2.082(4) (**IV**), and 2.101(2) Å (**V**).

Weak intermolecular hydrogen bonds H(7A)···O(1) (2.59 Å) are observed in the crystals of compound **I**. The nitro groups participate in the formation of the crystal structure of compound **II** to form intermolecular hydrogen bonds H(46)···O(3) (2.55 Å) and H(43)···N(1) (2.69 Å). Intermolecular contacts H···O(NO_2) (2.53–2.70 Å) and H···F (2.63, 2.56 Å) are observed in the crystals of compound **III**. The structural organization of the crystals of compounds **IV** and **V** is mainly caused by intermolecular interactions H···F (2.47–2.67 Å) and H···Cl (2.79, 2.93 Å), respectively.

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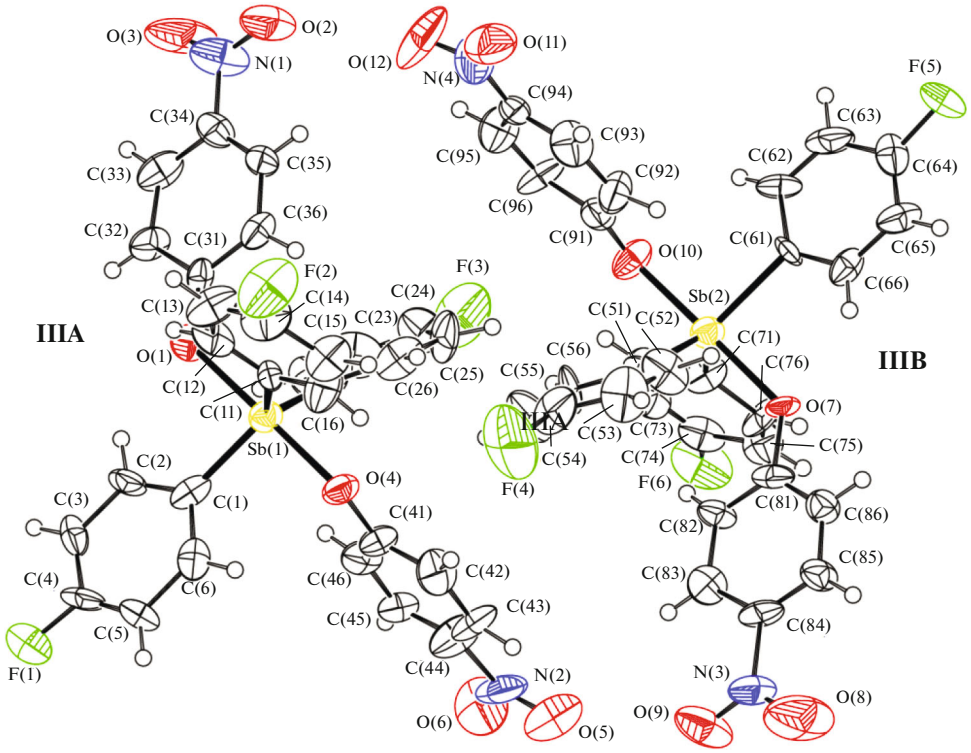

Fig. 2. Molecular structures of compounds **IIIA** and **IIIB**.

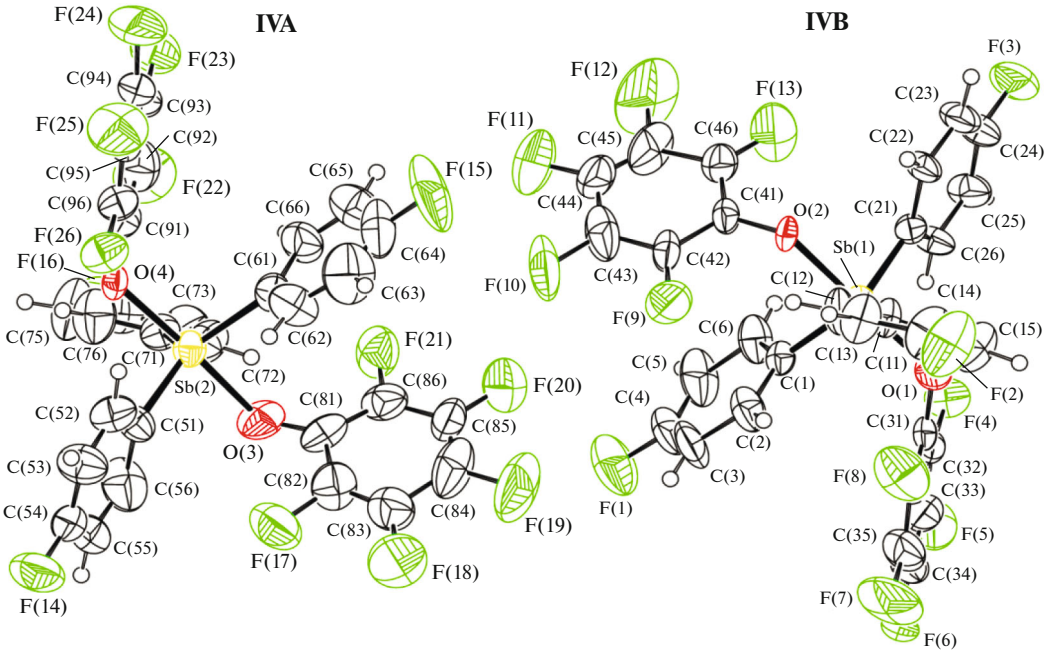

Fig. 3. Molecular structures of compounds **IVA** and **IVB**.

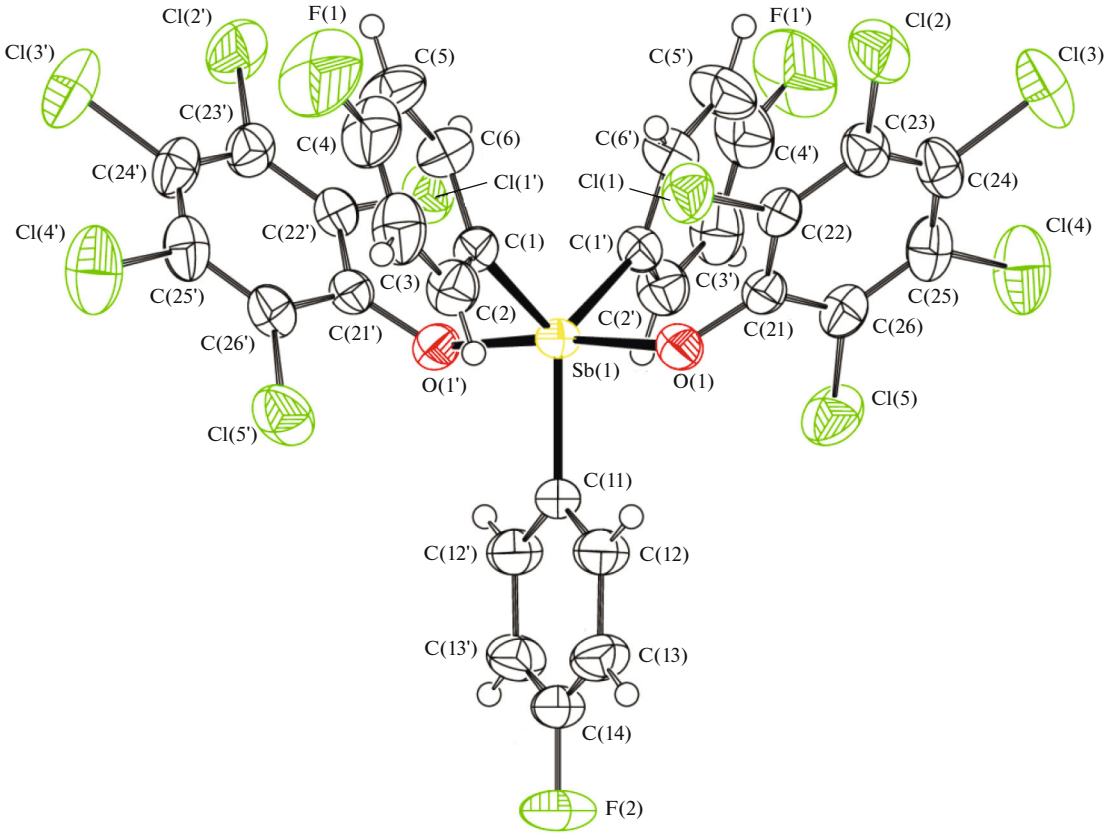

Fig. 4. Molecular structure of compound **V**.

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