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Synthesis and Structure of Tetra- and Triphenylantimony Organosulfonates

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Abstract—Tetraphenylantimony 2-naphthalenesulfonate (**I**), tetraphenylantimony phenylmethanesulfonate (**II**), and bis(tetraphenylantimony) sulfate (**III**) were synthesized via reaction of pentaphenylantimony with 2-naphthalenesulfonic acid, triphenylantimony bis(phenylmethanesulfonate), and triphenylantimony sulfate, respectively, in toluene. Triphenylantimony bis(phenylmethanesulfonate) (**IV**) was synthesized from triphenylstibine, phenylmethanesulfonic acid, and hydrogen peroxide (taken in the molar ratio of 1 : 2 : 1). Organoantimony betaine $Ph_3\overset{+}{Sb}CH(Ph)SO_2\overset{-}{O}$ (**V**) was obtained by reacting triphenylstibine with phenylmethanesulfonyl chloride in the presence of hydrogen peroxide. The structures of the compounds synthesized were determined using X-ray diffraction analysis. The Sb atoms in **I**–**IV** have distorted trigonal bipyramidal coordination. The Sb–C and Sb–O distances are equal to 2.100(4)–2.140(3) and 2.644(2) Å in molecule **I**, 2.105(2)–2.131(2) and 2.699(1) Å in **II**, 2.096(6)–2.169(6) and 2.101(6), 2.271(3) Å in **III**, and 2.101(3)–2.102(5) and 2.111(2) Å in **IV**. In **V**, the Sb atoms have close tetrahedral (2.105–2.115 Å) and remote (2.65, 3.019 Å) octahedral environment (the oxygen atoms of sulfo group of their own and neighboring molecules). Two betaine molecules are joined through the O atoms of the sulfo groups of bidentate phenylmethanesulfonate ligands to form a centrosymmetrical dimer. The intermolecular C(1)–H(1)··O(1) hydrogen bonds (3.10 Å) are formed that strengthen the dimer.

Pentaorganylantimony is known to react with antimony derivatives R_3SbX_2, where R is Ar or Alk and X is the acid residue, (toluene, 1 h, 90°C) and form R_4SbX compounds in a high yield [1–18]. In the course of our studies in this field of chemistry of antimony compounds, we established that an increase in the size of substituent X hinders this reaction. Thus, triphenylantimony bis(2-naphthalenesulfonate) fails to react with pentaphenylantimony under the mentioned conditions, although the reaction between pentaphenylantimony and 2-naphthalenesulfonic acid gives tetraphenylantimony 2-naphthalenesulfonate (**I**) in 80% yield. Bulky arenesulfonate ligands at the antimony atom are likely to hinder the formation of a labile ionic complex, which, as we suggest, forms at the first stage of the reaction and decomposes further to yield tetraphenylantimony arenesulfonate:

$$Ph_5Sb + Ph_3Sb(OSO_2Ar)_2$$
$$\longrightarrow [Ph_4Sb]^+[Ph_4Sb(OSO_2Ar)_2]^- \longrightarrow 2Ph_4SbOSO_2Ar.$$

Thus, tetraphenylantimony phenylmethanesulfonate (**II**) was the only product (yield 97%) of reaction of pentaphenylantimony with triphenylantimony bis(phenylmethanesulfonate):

$$Ph_5Sb + Ph_3Sb(OSO_2CH_2Ph)_2 \longrightarrow 2Ph_4SbOSO_2CH_2Ph.$$

(**II**)

Bis(tetraphenylantimony) sulfate (**III**) was synthesized in the yield of 53% using similar scheme (toluene, 4 h, 90°C):

$$Ph_5Sb + Ph_3SbSO_4 \longrightarrow (Ph_4Sb)_2SO_4.$$

(**III**)

The yield of complex **III** obtained from bisulfate and pentaphenylantimony under these conditions was higher (79%):

$$Ph_5Sb + Ph_4SbOSO_3H \longrightarrow (Ph_4Sb)_2SO_4 + PhH.$$

(**III**)

The antimony compounds Ph_3SbX_2 with symmetrical structure were synthesized from triphenylstibine, sulfonic acid, and hydrogen peroxide via the reactions of oxidative addition:

$$Ph_3Sb + 2HOSO_2R + H_2O_2$$
$$\longrightarrow Ph_3Sb(OSO_2R)_2 + 2H_2O.$$

In this reaction, triphenylantimony bis(phenylmethanesulfonate) (**IV**) was obtained in the yield of 91%; however, the yields of triphenylantimony bis(2-naphthalenesulfonate) and triphenylantimony bis(1-naphthalenesulfonate) were 46 and 25%, respectively, which is likely to be due to the increasing size of the organic fragment of arenesulfonate ligand.

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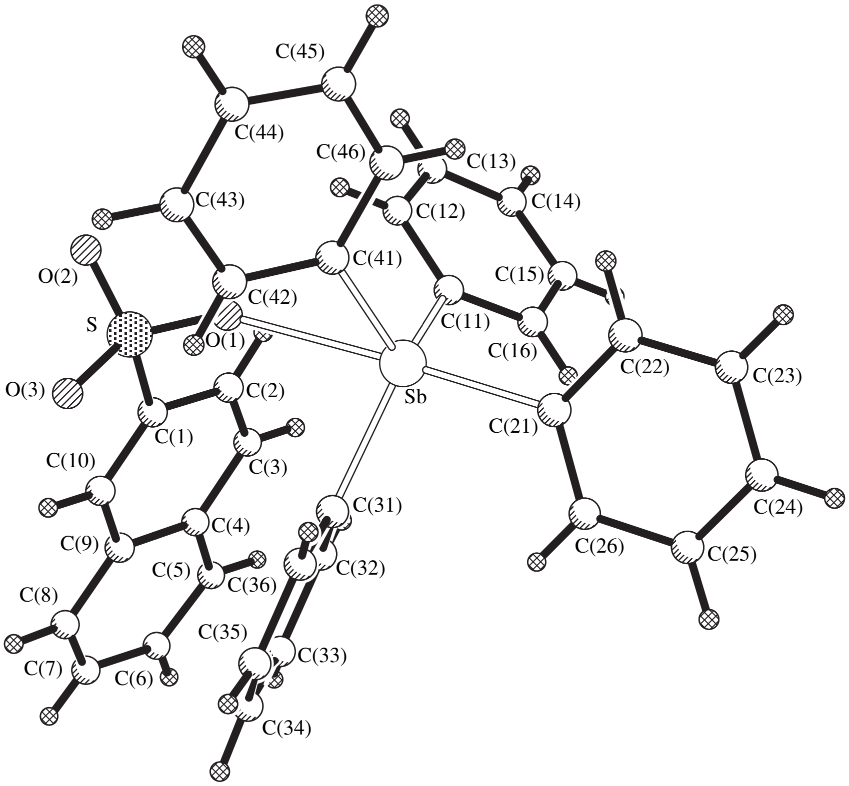

Fig. 1. Structure of tetraphenylantimony 2-naphthalenesulfonate (**I**).

We established that reaction of triphenylstibine with phenylmethanesulfonyl chloride in the presence of hydrogen peroxide follows a different course and results in organoantimony betaine (**V**):

$$\mathrm{Ph_3Sb + ClSO_2CH_2Ph \xrightarrow{H_2O_2} Ph_3\overset{+}{S}bCH(Ph)SO_2\bar{O}}. \qquad (\mathbf{V})$$

According to X-ray diffraction data, the antimony atoms in molecules **I** and **II** have heavily distorted trigonal bipyramidal coordinations with the axial sulfonate ligands (Figs. 1, 2). The Sb–C_{eq} bond lengths and $OSbO_{ax}$ angle in compounds **I** (2.100(4), 2.104(4), 2.114(4) Å; 177.2(1)°) and **II** (2.105(2), 2.105(2), 2.105(2) Å; 177.04(5)°) are almost equal. In structures **I** and **II**, the Sb–C_{ax} and Sb–O bond lengths are somewhat different (2.140(3), 2.644(2) Å in **I** and 2.131(2), 2.699(1) Å in **II**). When analyzing the geometrical parameters of structurally characterized tetraphenylantimony sulfonates (Table 1), one can see that the Sb–O distances vary within wide intervals, the longest distance being found in compound **II**.

In complex **III** (Fig. 3), the equatorial CSbC angles (111.8(3)°–124.9(3)°) are close to 120°; the sums of the equatorial angles are equal to 358.3° (Sb(1)) and 357.6° (Sb(2)); the Sb–O (2.248(3) and 2.271(3) Å) distances are close to the analogous distances in tetraphenylantimony oximates [9, 13, 16, 17] and tetraphenylantimony aroxides [19–22], in which this bond can be considered as a low-polarity covalent bond.

Note that in antimony derivatives Ar_4SbX (X is the residue of a strong acid), the Sb–X bond is, as a rule, an ionic bond. Thus, the crystal of triphenylantimony bisulfate consists of tetraphenylstibonium cations and hydroxysulfate anions [23]. The replacement of acid hydrogen atom in this compound by the electron-donor group Ph_4Sb decreases the accepting properties of the sulfate group, which is followed by a decrease in the Sb–O bond polarity and in its strengthening. A shortening of the Sb(1)–O(4) and Sb(2)–O(1) bonds in **III** makes the Ph_4Sb fragments approach the sulfur atom and, as a consequence, makes the O(2)···Sb(2) and O(3)···Sb(1) distances reduce to 3.737(4) and 3.411(4) Å, respectively. The former value insignificantly exceeds the sum of the van der Waals radii of the oxygen and sulfur atoms, while the latter makes 92% of this sum, thus suggesting the donor–acceptor interaction between the O(3) and Sb(1) atoms.

Alkanesulfonate groups in molecule **IV** (Fig. 4) are in the axial position, while the Ph substituents are in the equatorial positions. The sum of the equatorial bond angles is equal to 360°, the OSbO angle is equal to 175.8(1)°. Thus, in molecule **IV**, the Sb atom has a distorted trigonal bipyramidal coordination. The Sb–O (2.101(3), 2.102(5) Å) and Sb–C (2.111 (2) Å) bond lengths are standard for antimony(V) compounds of the

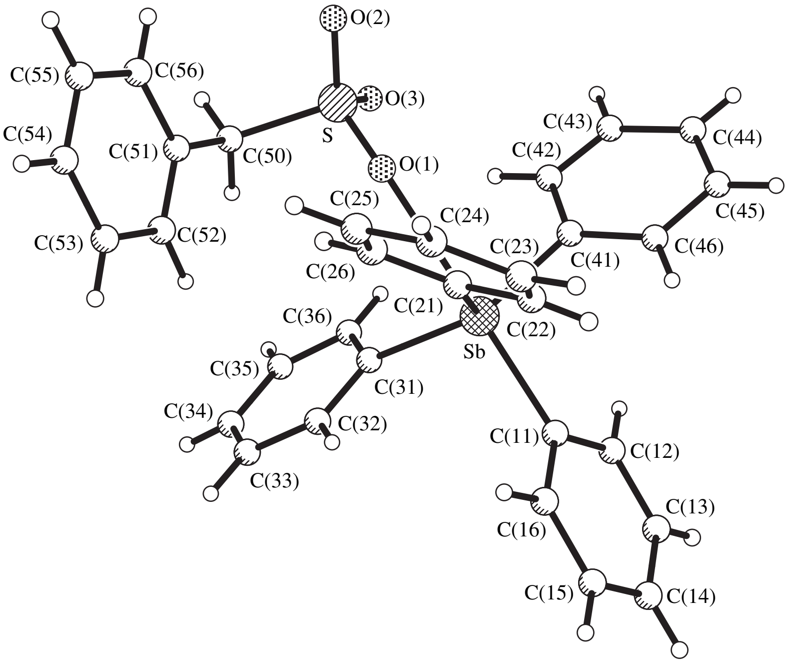

Fig. 2. Structure of tetraphenylantimony phenylmethanesulfonate (**II**).

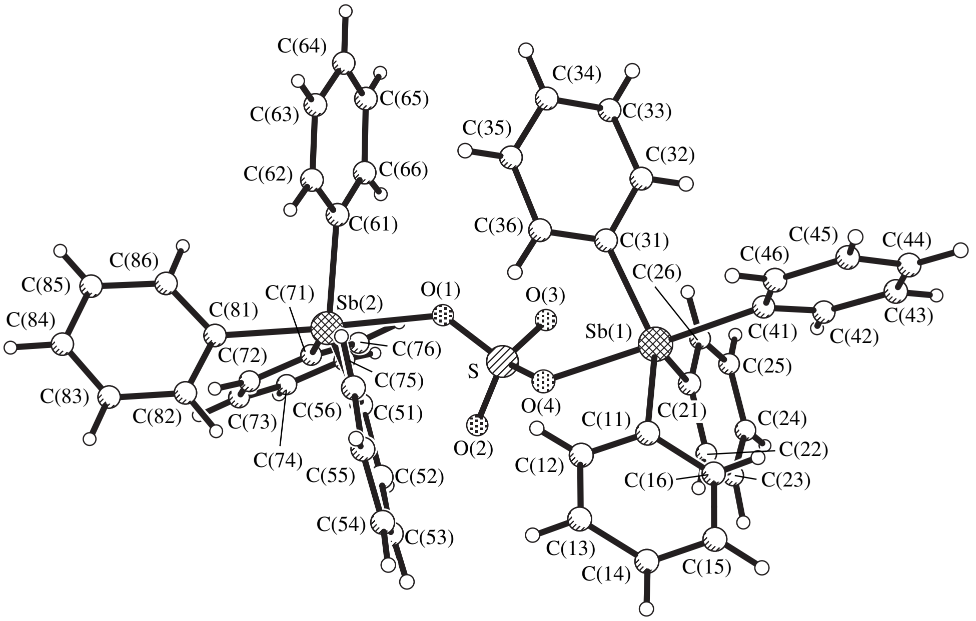

Fig. 3. Structure of bis(tetraphenylantimony) sulfate (**III**).

Ar_3SbX_2 type [24]. Molecule **IV** has its own symmetry C_2 with a twofold axis passing through the Sb, C(31), and C(34) atoms.

According to elemental and X-ray diffraction analyses data, betaine **V** is the crystal solvate $Ph_3SbCH(Ph)SO_3 \cdot C_7H_8$. The antimony atom in this compound has a distorted tetrahedral coordination (Fig. 5). The CSbC angles vary within the 98.7(1)°–122.0(1)° range. Two molecules **V** are joined through the hydrogen bond between the hydrogen atom of the methine group and the oxygen atom of the sulfonate ligand, this O atom being coordinated to the Sb atom of another molecule (the Sb–O(1)' distance is equal to 3.019(2) Å). In addition, the distance between O(3) and Sb atoms in molecule **V** is equal to 2.988(2) Å. The length of one of the S–O bonds in the sulfo group

Table 1. Selected bond lengths and angles in tetraphenylantimony sulfonates with the general formula Ph_4SbOSO_2R

R	Bond lengths, Å			Angle $OSbC_{ax}$, deg
	Sb–C_{eq}	Sb–C_{ax}	Sb–O	
$PhCH_2$	2.105(2)	2.131(2)	2.699(1)	177.04(5)
	2.105(2)			
	2.105(2)			
C_6H_3(COOH)-3, (OH)-4 [2]	2.092(3)	2.118(3)	2.691(3)	174.5(1)
	2.097(3)			
	2.118(3)			
2-$C_{10}H_7$	2.100(4)	2.140(3)	2.644(2)	177.2(1)
	2.104(4)			
	2.114(4)			
C_6H_4Me-4 [1]	2.099(4)	2.129(4)	2.588(4)	178.2(2)
	2.101(4)			
	2.119(4)			
C_6H_5 [25]	2.104(5)	2.131(5)	2.506(4)	176.0(2)
	2.108(6)			
	2.111(6)			
$C_6H_3Me_2$-2,4 [3]	2.101(6)	2.131(6)	2.464(4)	175.2(2)
	2.102(6)			
	2.111(7)			
$OSbPh_4$	2.096(6)			
	2.118(6)	2.178(6)	2.248(3)	174.8(2)
	2.109(6)			
	2.101(6)			
	2.124(5)			
	2.095(3)	2.169(6)	2.271(3)	174.1(2)

(S–O(2), 1.433(2) Å) noticeably differs from those of the remaining two bonds (S–O(1, 3), 1.446(2), 1.454(2) Å, respectively), which can be explained only by an additional interaction of the O(1, 3) atoms with the Sb and H atoms of the methine group.

Thus, in complex **V**, the coordination number of the central atom is increased to six. This suggestion is supported by the fact that the values of the C(21)SbO(3) and C(41)SbO(1)' angles (164.26(9)° and 175.66(9)°, respectively) are close to the values of angles in octahedral complexes.

EXPERIMENTAL

Synthesis of complex I. A mixture of 0.50 g of pentaphenylantimony and 0.20 g of 2-naphthalenesulfonic acid in 20 ml of toluene was kept at 20°C for 1 h; the solvent was removed; the residue was recrystallized from water. The yield was 0.50 g (80%); mp 199°C.

Synthesis of complex II. A mixture of 1.00 g of pentaphenylantimony, 1.37 g of triphenylantimony bis(phenylmethanesulfonate) and 20 ml of toluene was kept at 90°C for 1 h. After cooling, the colorless crystals were filtered off and dried. The yield was 2.16 g (91%); mp 196°C.

Synthesis of complex III. a) A mixture of 1.00 g of pentaphenylantimony and 0.89 g of triphenylantimony sulfate in 15 ml of toluene was heated at 90°C for 4 h. The solvent was removed. The product was recrystallized from water. The yield of colorless crystals **III** was 1.00 g (53%); mp 230°C.

b) A mixture of 1.00 g of pentaphenylantimony and 1.04 g of tetraphenylantimony bisulfate in 10 ml of toluene was heated at 90°C for 1 h. The solvent was removed. The yield of crystal complex **III** was 1.50 g (79%); mp 230°C.

Synthesis of complex IV. To a mixture of 0.50 g of triphenylantimony and 0.48 g of phenylmethanesulfonic acid in 20 ml of ether, 0.16 ml of 30% hydrogen peroxide solution was added. After 12 h, the solvent was removed; the residue was recrystallized from toluene. The yield of colorless crystals **IV** was 0.70 g (71%), mp 193°C.

Triphenylantimony bis(1-naphthalenesulfonate) was synthesized by a procedure similar to that for complex **IV**. The yield was 25%; mp 221°C.

Triphenylantimony bis(2-naphthalenesulfonate) was synthesized using procedures similar to those used in the synthesis of complex **IV**. The yield was 46%; mp 227°C.

Synthesis of complex V. To a mixture of 0.50 g of triphenystibine and 0.28 g of phenylmethanesulfonyl chloride in 20 ml of diethyl ether, 0.16 ml of 30% hydrogen peroxide solution was added. The reaction mixture was kept at 20°C for 12 h; the solvent was removed; the residue was recrystallized from toluene. The yield was 0.90 g (91%); mp 232°C (with decomposition).

X-ray diffraction analysis of natural-faceted single crystals of complexes **I**–**V** was carried out on a Bruker SMART 1000 CCD diffractometer (λMoK_α radiation). Structures **I**–**V** were solved by direct methods and refined by the least-squares method in an anisotropic approximation for non-hydrogen atoms. Positions of the hydrogen atoms were calculated geometrically and included in the refinement in the rider model.

Table 2. Crystallographic data and results of refinement of structures **I**–**V**

Parameter	Value				
	I	**II**	**III**	**IV**	**V**
Empirical formula	$C_{34}H_{27}O_3SSb$	$C_{31}H_{27}O_3SSb$	$C_{48}H_{40}O_4SSb_2$	$C_{32}H_{29}O_6S_2Sb$	$C_{32}H_{29}O_3SSb$
M	637.37	601.34	956.36	695.42	615.36
T, K	293(2)	293(2)	296(2)	297(2)	296(2)
Crystal system	Orthorhombic	Triclinic	Monoclinic	Monoclinic	Triclinic
Space group	$P2_12_12_1$	$P\bar{1}$	$P2_1/c$	$C2/c$	$P\bar{1}$
Unit cell parameters:					
a, Å	10.1258(12)	9.278(1)	12.645(3)	19.296(3)	8.618(1)
b, Å	15.8458(19)	10.680(1)	26.714(5)	9.405(1)	9.923(1)
c, Å	17.687(2)	13.918(2)	12.299(2)	17.343(2)	16.233(2)
α, deg	90	96.737(2)	90		87.729(2)
β, deg	90	99.606(2)	97.293(5)	99.782(3)	88.857(2)
γ, deg	90	98.345(2)	90		86.001(2)
V, Å^3	2837.8(6)	1331.2(3)	4121.0(14)	3101.6(7)	1383.5(3)
Z	4	2	4	4	2
ρ(alcd), g/cm^3	1.492	1.500	1.541	1.489	1.477
μ_{Mo}, mm^{-1}	1.079	1.145	1.405	1.066	1.104
F(000)	1288	608	1912	1408	624
Crystal form (size, mm)	Prism (0.15 × 0.20 × 0.25)	Prism (0.20 × 0.25 × 0.28)	Prism (0.10 × 0.15 × 0.25)	Prism (0.20 × 0.20 × 0.05)	Prism (0.20 × 0.20 × 0.20)
θ, deg	1.73–30.00	3.00–29.00	1.79–20.06	2.14–24.05	3.03–26.03
Range of reflection indices	$-12 \le h \le 14$ $-20 \le k \le 22$ $-24 \le l \le 23$	$-12 \le h \le 12$ $-14 \le k \le 14$ $-18 \le l \le 18$	$-12 \le h \le 8$ $-25 \le k \le 25$ $-11 \le l \le 11$	$-21 \le h \le 22$ $-10 \le k \le 10$ $-16 \le l \le 19$	$-10 \le h \le 10$ $-12 \le k \le 12$ $-19 \le l \le 20$
Total number of reflections	20681	13858	12316	7155	14750
Total number of unique reflections	8051 (R_{int} = 0.0528)	6996 (R_{int} = 0.0251)	3860 (R_{int} = 0.0338)	2447 (R_{int} = 0.0773)	5442 (R_{int} = 0.0528)
Reflections with $I > 2\sigma(I)$	4352	6096	2952	1866	3814
Number of refined parameters	353	325	496	187	325
GOOF	0.789	0.980	0.899	1.009	0.877
R-factors for all reflections	R_1 = 0.0723 wR_2 = 0.0667	R_1 = 0.0333 wR_2 = 0.0745	R_1 = 0.0423 wR_2 = 0.0571	R_1 = 0.0687 wR_2 = 0.0972	R_1 = 0.0615 wR_2 = 0.0865
Final R-factor ($I > 2\sigma(I)$)	R_1 = 0.0334 wR_2 = 0.0599	R_1 = 0.0283 wR_2 = 0.0728	R_1 = 0.0277 wR_2 = 0.0541	R_1 = 0.0446 wR_2 = 0.0871	R_1 = 0.0388 wR_2 = 0.0809
Residual electron density (min/max), e/Å^3	0.362/0.350	0.412/0.963	0.271/0.382	0.356/0.492	0.379/0.573

Table 3. Coordinates of atoms ($\times 10^4$) and their isotropic equivalent thermal parameters in structures **I–V**

Atom	x	y	z	U_{eq}, Å^2
I				
Sb	1710.3(2)	6898.8(1)	3439.6(1)	41.64(7)
S	1512(1)	4396.1(6)	3687.6(6)	58.4(3)
O(1)	8539(3)	10239(1)	1606(2)	65.8(7)
O(2)	8936(4)	8797(2)	1818(3)	169(2)
O(3)	9114(3)	9379(3)	570(3)	140(2)
C(1)	6784(5)	9178(2)	1166(2)	47.4(8)
C(2)	5871(5)	9399(2)	1745(2)	64(1)
C(3)	4564(5)	9287(3)	1636(3)	72(1)
C(4)	4068(5)	8922(3)	959(3)	56(1)
C(5)	2707(5)	8759(3)	839(4)	80(2)
C(6)	2298(5)	8367(3)	188(4)	94(2)
C(7)	3202(6)	8123(3)	–348(3)	89(2)
C(8)	4526(5)	8283(3)	–267(3)	71(1)
C(9)	4992(4)	8682(2)	390(2)	51(1)
C(10)	6348(4)	8833(2)	513(2)	52(1)
C(11)	2789(4)	6611(2)	2456(2)	40.8(9)
C(12)	2410(4)	5973(2)	1966(2)	58(1)
C(13)	3100(5)	5845(2)	1307(2)	63(1)
C(14)	4159(5)	6333(3)	1140(2)	70(1)
C(15)	4544(3)	6975(3)	1611(3)	58(1)
C(16)	3849(4)	7113(2)	2273(2)	49(1)
C(21)	2011(3)	8234(2)	3490(2)	44.9(8)
C(22)	1401(4)	8772(2)	2995(2)	53(1)
C(23)	1644(5)	9631(2)	3023(2)	64(1)
C(24)	2493(5)	9949(3)	3547(3)	78(1)
C(25)	3123(7)	9415(3)	4041(2)	89(2)
C(26)	2863(5)	8556(3)	4019(2)	71(1)
C(31)	2469(4)	6557(2)	4510(2)	47(1)
C(32)	3672(4)	6175(2)	4588(3)	63(1)
C(33)	4135(5)	5994(3)	5309(3)	85(2)
C(34)	3392(8)	6198(3)	5921(3)	87(2)
C(35)	2184(6)	6568(3)	5839(3)	80(2)
C(36)	1727(5)	6762(2)	5135(2)	64(1)
C(41)	–333(3)	6705(2)	3333(2)	40.8(9)
C(42)	–982(4)	6205(2)	3847(2)	48(1)
C(43)	–2341(5)	6084(3)	3760(3)	61(1)
C(44)	–2997(4)	6447(3)	3161(3)	63(1)
C(45)	–2333(4)	6952(4)	2655(2)	62(1)
C(46)	–993(4)	7073(3)	2734(2)	52(1)
II				
Sb	6613.46(9)	9748.10(8)	7412.99(6)	37.89(2)
S	3940.4(4)	6278.6(4)	7120.9(3)	47.9(1)
O(1)	4916(1)	7494(1)	7479(1)	52.0(3)
O(2)	2970(1)	5949(1)	7798(1)	76.9(4)
O(3)	3183(2)	6203(1)	6118(1)	82.3(5)
C(11)	7966(2)	11490(1)	7286(1)	42.3(3)
C(12)	7609(2)	12108(2)	6474(1)	51.6(4)
C(13)	8526(2)	13189(2)	6333(2)	65.0(5)
C(14)	9801(2)	13662(2)	7007(2)	64.8(5)
C(15)	10159(2)	13065(2)	7809(2)	64.4(5)
C(16)	9257(2)	11983(2)	7957(1)	54.8(4)
C(21)	6894(2)	9801(1)	8951(1)	41.7(3)
C(22)	6930(2)	10971(2)	9514(1)	57.7(4)
C(23)	7114(2)	11063(2)	10527(1)	67.3(5)
C(24)	7281(2)	10006(2)	10971(1)	67.2(6)
C(25)	7254(2)	8853(2)	10424(1)	67.5(5)
C(26)	7048(2)	8730(2)	9396(1)	55.1(4)
C(31)	7708(2)	8390(1)	6744(1)	42.7(3)
C(32)	9127(2)	8348(2)	7219(2)	61.5(5)
C(33)	9931(2)	7477(2)	6831(2)	76.8(6)
C(34)	9338(2)	6685(2)	5978(2)	72.4(5)
C(35)	7931(3)	6720(2)	5499(1)	76.9(6)
C(36)	7084(2)	7574(2)	5887(1)	59.8(5)
C(41)	4531(2)	10127(1)	6771(1)	38.6(3)
C(42)	3453(2)	9246(2)	6135(1)	53.0(4)
C(43)	2086(2)	9593(2)	5802(1)	65.0(5)
C(44)	1808(2)	10795(2)	6110(2)	62.8(5)
C(45)	2896(2)	11669(2)	6739(1)	56.6(4)
C(46)	4258(2)	11342(2)	7059(1)	47.8(4)
C(50)	5125(2)	5091(2)	7073(1)	63.2(5)
C(51)	6015(2)	4960(1)	8056(1)	51.6(4)
C(52)	7474(2)	5499(2)	8335(2)	79.6(7)
C(53)	8268(2)	5371(2)	9301(2)	96.0(9)
C(54)	7555(3)	4671(2)	9880(2)	84.1(7)
C(55)	6123(2)	4126(2)	9608(2)	70.0(5)
C(56)	5350(2)	4280(2)	8704(1)	57.9(5)
III				
Sb(1)	2074(1)	6218(1)	3909(1)	41(1)
Sb(2)	2485(1)	4042(1)	2269(1)	37(1)
S	3069(1)	5339(1)	2317(1)	39(1)
O(1)	2367(3)	4887(1)	2081(3)	42(1)
O(2)	4171(3)	5198(1)	2339(3)	54(1)
O(3)	2711(3)	5719(1)	1524(3)	55(1)
O(4)	2898(3)	5518(1)	3446(3)	41(1)
C(11)	2765(5)	6016(2)	5508(5)	41(2)
C(12)	2930(5)	5528(2)	5850(5)	49(2)
C(13)	3400(5)	5431(2)	6906(5)	58(2)
C(14)	3718(5)	5814(3)	7609(5)	52(2)
C(15)	3561(5)	6301(3)	7272(6)	61(2)
C(16)	3071(5)	6400(2)	6221(5)	52(2)
C(21)	3126(6)	6649(2)	3091(5)	47(2)
C(22)	4097(7)	6774(3)	3623(6)	75(2)
C(23)	4747(7)	7097(3)	3146(9)	99(3)
C(24)	4428(9)	7303(3)	2166(10)	98(3)
C(25)	3471(8)	7175(3)	1608(7)	86(3)
C(26)	2804(6)	6840(2)	2069(6)	67(2)

Table 3. (Contd.)

Atom	*x*	*y*	*z*	U_{eq}, Å^2	Atom	*x*	*y*	*z*	U_{eq}, Å^2
		III					**IV**		
C(31)	687(5)	5870(2)	3150(5)	44(2)	C(21)	4143(2)	3025(4)	1849(2)	38(1)
C(32)	–57(6)	6157(3)	2487(6)	61(2)	C(22)	4031(2)	3078(4)	1039(2)	59(1)
C(33)	–968(7)	5932(4)	1988(6)	82(2)	C(23)	3477(2)	2358(5)	617(2)	74(2)
C(34)	–1185(7)	5449(4)	2163(8)	94(3)	C(24)	3043(2)	1576(5)	984(3)	71(1)
C(35)	–489(8)	5170(3)	2833(8)	93(3)	C(25)	3141(2)	1522(5)	1772(2)	65(1)
C(36)	468(6)	5376(3)	3321(6)	73(2)	C(26)	3689(2)	2260(4)	2216(2)	56(1)
C(41)	1219(5)	6856(2)	4471(5)	46(2)	C(31)	5000	6344(5)	2500	55(2)
C(42)	1415(6)	7340(3)	4207(5)	67(2)	C(32)	5544(3)	7030(5)	2926(3)	108(2)
C(43)	886(7)	7731(3)	4682(8)	86(3)	C(33)	5531(4)	8522(6)	2923(5)	154(3)
C(44)	185(8)	7635(3)	5380(6)	80(3)	C(34)	5000	9210(9)	2500	151(4)
C(45)	–36(7)	7153(3)	5637(6)	85(3)			**V**		
C(46)	488(6)	6771(2)	5190(6)	73(2)	Sb	5329.1(2)	6250.8(2)	6495.4(1)	38.77(6)
C(51)	3024(5)	4196(2)	3922(4)	39(2)	S	6125.4(9)	3234.9(7)	5988.0(5)	41.6(2)
C(52)	4043(6)	4344(2)	4260(5)	51(2)	O(1)	6352(2)	3588(2)	5124(1)	51.5(6)
C(53)	4424(6)	4355(2)	5364(6)	64(2)	O(2)	5747(3)	1866(2)	6155(1)	58.8(7)
C(54)	3805(8)	4212(2)	6115(6)	70(2)	O(3)	7351(2)	3680(2)	6494(1)	55.0(6)
C(55)	2780(8)	4070(3)	5813(6)	80(2)	C(1)	4435(3)	4306(3)	6295(2)	37.4(8)
C(56)	2363(6)	4066(2)	4693(5)	63(2)	C(11)	3528(3)	3836(3)	7033(2)	38.0(8)
C(61)	861(5)	4101(2)	1683(5)	38(2)	C(12)	1935(4)	3776(3)	6977(2)	47.8(9)
C(62)	112(6)	3870(2)	2220(5)	52(2)	C(13)	1056(4)	3323(3)	7644(2)	61(1)
C(63)	–935(6)	3888(2)	1848(6)	62(2)	C(14)	1749(4)	2948(3)	8373(2)	67(1)
C(64)	–1282(5)	4131(2)	867(6)	56(2)	C(15)	3355(4)	2986(3)	8434(2)	61(1)
C(65)	–549(6)	4347(2)	301(5)	60(2)	C(16)	4218(4)	3435(3)	7762(2)	52(1)
C(66)	518(6)	4346(2)	719(5)	48(2)	C(21)	3510(3)	7741(3)	6705(2)	41(1)
C(71)	3511(5)	4029(2)	1028(4)	37(2)	C(22)	1955(4)	7496(3)	6652(2)	52(1)
C(72)	4039(5)	3592(2)	871(5)	45(2)	C(23)	824(4)	8467(3)	6854(2)	62(1)
C(73)	4701(5)	3561(3)	66(6)	59(2)	C(24)	1237(4)	9702(3)	7106(2)	64(1)
C(74)	4810(6)	3967(3)	–605(5)	61(2)	C(25)	2762(4)	9962(3)	7144(2)	63(1)
C(75)	4285(6)	4396(3)	–468(5)	59(2)	C(26)	3902(4)	8981(3)	6956(2)	58(1)
C(76)	3610(5)	4433(2)	331(5)	49(2)	C(31)	6991(3)	7084(3)	5697(2)	41(1)
C(81)	2470(5)	3241(2)	2557(5)	44(2)	C(32)	6698(4)	8407(3)	5393(2)	53(1)
C(82)	2986(5)	3014(3)	3482(5)	57(2)	C(33)	7840(4)	9018(4)	4933(2)	65(1)
C(83)	2970(6)	2507(3)	3630(7)	75(2)	C(34)	9235(4)	8339(4)	4770(2)	62(1)
C(84)	2447(7)	2207(3)	2868(9)	89(3)	C(35)	9518(4)	7028(4)	5060(2)	57(1)
C(85)	1913(7)	2409(3)	1930(7)	89(3)	C(36)	8407(4)	6393(3)	5524(2)	50(1)
C(86)	1933(6)	2923(3)	1775(6)	68(2)	C(41)	6342(4)	6176(3)	7674(2)	47(1)
		IV			C(42)	7955(4)	5970(3)	7766(2)	61(1)
Sb	5000	4108.5(4)	2500	37.2(1)	C(43)	8580(5)	5994(4)	8536(2)	82(1)
S(1)	4041(1)	4899(1)	3901(1)	66.8(3)	C(44)	7618(5)	6202(4)	9217(3)	93(2)
O(1)	4533(1)	4025(3)	3515(1)	51.9(7)	C(45)	6047(5)	6405(4)	9132(2)	87(1)
O(2)	4392(2)	6106(3)	4269(2)	110(1)	C(46)	5397(5)	6405(4)	8363(2)	69(1)
O(3)	3412(2)	5140(4)	3366(2)	113(1)	C(51)	7388(4)	925(4)	9092(2)	170(3)
C(1)	3859(3)	3712(5)	4650(2)	92(2)	C(52)	7750(4)	2258(4)	9162(3)	125(2)
C(11)	3613(2)	2265(5)	4360(2)	77(2)	C(53)	8119(5)	2724(4)	9926(3)	181(3)
C(12)	4106(3)	1187(5)	4374(3)	90(2)	C(54)	8127(5)	1856(6)	10619(3)	161(3)
C(13)	3923(4)	–158(7)	4074(3)	122(2)	C(55)	7765(6)	522(6)	10549(2)	249(5)
C(14)	3248(4)	–376(8)	3754(4)	144(3)	C(56)	7395(5)	57(4)	9786(3)	288(6)
C(15)	2741(3)	617(8)	3751(4)	143(3)	C(57)	7073(8)	384(8)	8284(4)	187(3)
C(16)	2924(3)	2006(7)	4058(3)	121(2)	H(1)	3780(30)	4470(30)	5784(16)	45

Table 4. Selected bond lengths and angles in structures **I**–**V**

Bond	*d*, Å	Angle	ω, deg	Bond	*d*, Å	Angle	ω, deg
I				**III**			
Sb–C(41)	2.100(4)	C(41)SbC(11)	113.9(1)	S–O(1)	1.504(4)	O(1)SO(4)	106.4(2)
Sb–C(11)	2.104(4)	C(41)SbC(31)	113.6(2)	S–O(2)	1.440(4)	C(61)Sb(2)C(51)	120.1(2)
Sb–C(31)	2.114(4)	C(11)SbC(31)	119.7(1)	S–O(3)	1.442(4)	C(51)Sb(2)C(71)	123.1(2)
Sb–C(21)	2.140(3)	C(41)SbC(21)	106.7(1)	S–O(4)	1.511(4)	C(61)Sb(2)C(71)	114.6(2)
S–O(2)	1.381(3)	C(11)SbC(21)	100.1(1)			SO(1)Sb(2)	138.3(2)
S–O(1)	1.434(2)	C(31)SbC(21)	99.5(1)	**IV**			
S–O(3)	1.459(4)	O(2)SO(1)	113.2(3)	Sb–C(21)	2.101(3)	C(21)SbC(21)'	122.0(2)
S–C(1)	1.779(5)	O(2)SO(3)	115.3(3)	Sb–C(21)'	2.101(3)	C(21)SbC(31)	119.0(1)
C(1)–C(10)	1.352(5)	O(1)SO(3)	109.1(2)	Sb–C(31)	2.102(5)	C(21)'SbC(31)	119.0(1)
C(1)–C(2)	1.423(5)	O(2)SC(1)	106.2(2)	Sb–O(1)'	2.111(2)	C(21)SbO(1)'	86.7(1)
C(2)–C(3)	1.350(5)	O(1)SC(1)	105.6(2)	Sb–O(1)	2.111(2)	C(21)'SbO(1)'	91.2(1)
C(3)–C(4)	1.421(6)	O(3)SC(1)	106.7(2)	S(1)–O(3)	1.416(3)	C(31)SbO(1)'	92.12(7)
C(4)–C(5)	1.419(6)	C(10)C(1)C(2)	120.1(4)	S(1)–O(2)	1.417(3)	C(21)SbO(1)	91.2(1)
C(4)–C(9)	1.426(6)	C(10)C(1)S	121.3(3)	S(1)–O(1)	1.496(3)	C(21)'SbO(1)	86.7(1)
C(5)–C(6)	1.373(7)	C(2)C(1)S	118.5(3)	S(1)–C(1)	1.792(5)	C(31)SbO(1)	92.12(7)
C(6)–C(7)	1.373(7)	C(3)C(2)C(1)	120.2(4)	C(1)–C(11)	1.500(7)	O(1)'SbO(1)	175.8(1)
C(7)–C(8)	1.372(6)	C(2)C(3)C(4)	121.3(4)	C(11)–C(16)	1.366(7)	O(3)S(1)O(2)	117.2(2)
C(8)–C(9)	1.404(5)	C(5)C(4)C(3)	122.9(5)	C(11)–C(12)	1.387(7)	O(3)S(1)O(1)	109.4(2)
C(9)–C(10)	1.410(5)	C(5)C(4)C(9)	118.9(5)	C(12)–C(13)	1.390(7)	O(2)S(1)O(1)	110.5(2)
II				C(13)–C(14)	1.343(9)	O(3)S(1)C(1)	108.8(2)
Sb–C(31)	2.105(2)	C(31)SbC(21)	112.78(6)	C(14)–C(15)	1.35(1)	O(2)S(1)C(1)	108.1(2)
Sb–C(21)	2.105(2)	C(31)SbC(41)	123.43(5)	C(15)–C(16)	1.432(9)	O(1)S(1)C(1)	101.5(2)
Sb–C(41)	2.105(2)	C(21)SbC(41)	113.37(6)	C(21)–C(26)	1.372(5)	S(1)O(1)Sb	138.3(2)
Sb–C(11)	2.131(2)	C(31)SbC(11)	101.23(6)	C(21)–C(22)	1.385(5)	C(11)C(1)S(1)	114.3(3)
Sb–O(1)	2.699(1)	C(21)SbC(11)	102.01(6)	**V**			
S–O(3)	1.442(2)	C(41)SbC(11)	99.38(6)	Sb–C(31)	2.103(3)	C(31)SbC(21)	108.7(1)
S–O(2)	1.446(2)	C(31)SbO(1)	76.52(5)	Sb–C(21)	2.111(3)	C(31)SbC(41)	105.0(1)
S–O(1)	1.453(1)	C(21)SbO(1)	80.71(5)	Sb–C(41)	2.115(3)	C(21)SbC(41)	98.7(1)
S–C(50)	1.798(2)	C(41)SbO(1)	80.46(5)	Sb–C(1)	2.166(3)	C(31)SbC(1)	122.0(1)
C(11)–C(16)	1.383(2)	C(11)SbO(1)	177.04(5)	Sb–O(3)	2.988(2)	C(21)SbC(1)	111.4(1)
C(11)–C(12)	1.391(2)	O(3)SO(2)	114.02(9)	Sb–O(1)'	3.019(2)	C(41)SbC(1)	108.4(1)
C(12)–C(13)	1.385(2)	O(3)SO(1)	112.67(8)	S–O(2)	1.433(2)	C(31)SbO(3)	87.07(9)
C(13)–C(14)	1.371(3)	O(2)SO(1)	111.73(8)	S–O(1)	1.446(2)	C(21)SbO(3)	164.26(9)
C(14)–C(15)	1.366(3)	O(3)SC(50)	104.59(9)	S–O(3)	1.454(2)	C(41)SbO(3)	77.06(9)
C(15)–C(16)	1.383(3)	O(2)SC(50)	107.00(9)	S–C(1)	1.816(3)	C(1)SbO(3)	57.19(9)
C(21)–C(26)	1.378(2)	O(1)SC(50)	106.07(8)	C(1)–C(11)	1.494(4)	C(31)SbO(1)'	78.50(9)
C(21)–C(22)	1.388(2)	SO(1)Sb	158.54(8)	C(11)–C(16)	1.368(4)	C(21)SbO(1)'	77.61(9)
III				C(11)–C(12)	1.382(4)	C(41)SbO(1)'	175.66(9)
Sb(1)–C(31)	2.096(6)	C(31)Sb(1)C(21)	124.9(3)	C(12)–C(13)	1.387(4)	C(1)SbO(1)'	71.29(8)
Sb(1)–C(11)	2.118(6)	C(21)Sb(1)C(11)	111.8(3)	C(13)–C(14)	1.363(5)	O(3)SbO(1)'	105.92(6)
Sb(1)–C(21)	2.109(6)	C(31)Sb(1)C(11)	121.6(2)	C(14)–C(15)	1.393(5)	O(2)SO(1)	114.4(1)
Sb(1)–C(41)	2.178(6)	C(41)Sb(1)O(4)	174.8(2)	C(15)–C(16)	1.384(4)	O(2)SO(3)	114.7(1)
Sb(1)–O(4)	2.248(3)	SO(4)Sb(1)	128.5(2)	C(21)–C(26)	1.377(4)	O(1)SO(3)	112.4(1)
Sb(2)–C(51)	2.101(6)	O(2)SO(3)	114.6(2)	C(21)–C(22)	1.383(4)	O(2)SC(1)	106.7(1)
Sb(2)–C(81)	2.169(6)	O(3)SO(1)	107.9(2)	C(22)–C(23)	1.366(4)	O(1)SC(1)	104.1(1)
Sb(2)–C(61)	2.095(6)	O(3)SO(4)	109.2(2)	C(23)–C(24)	1.379(5)	O(3)SC(1)	103.1(1)
Sb(2)–C(71)	2.124(5)	O(2)SO(1)	110.0(2)	C(24)–C(25)	1.360(5)	SO(3)Sb	83.06(9)
Sb(2)–O(1)	2.271(3)	O(2)SO(4)	108.5(2)	C(25)–C(26)	1.372(4)	SC(1)Sb	104.8(1)

Hydrogen bond parameters in crystal **V**

Bond	Distances, Å			Angle CHP, deg
	C–H	H···O	C···O	
C(1)–H(1)···O(1)	1.02(3)	2.37(3)	3.100(3)	128(2)

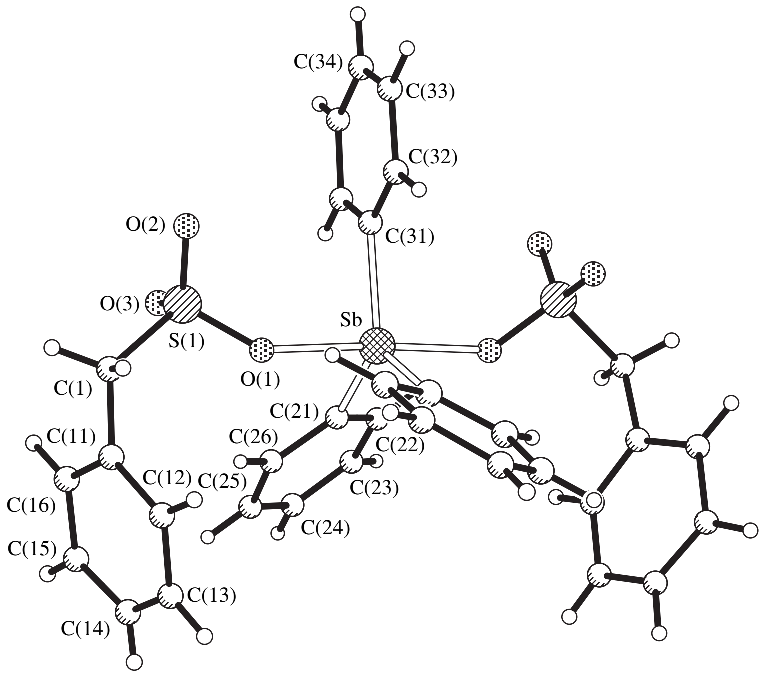

Fig. 4. Structure of triphenylantimony bis(phenylmethanesulfonate) (**IV**).

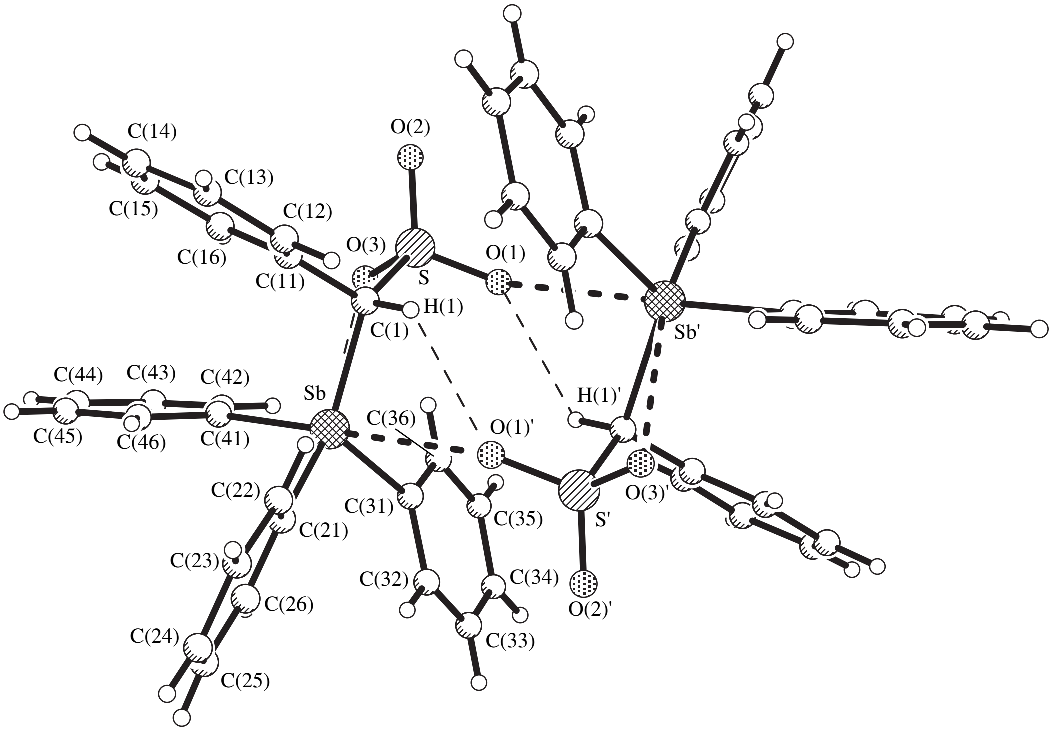

Fig. 5. Molecular structure of compound **V**.

Data collection and processing and refinement of the unit cell parameters were carried out using the SMART and SAINT-*Plus* programs [25]. All the calculations for the structure determination and refinement were performed using the SHELXTL/PC programs [26].

Selected crystallographic data and the results of refinement of structures **I**–**V** are presented in Table 2, coordinates of atoms are given in Table 3, and bond lengths and angles are listed in Table 4.

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