Organometallic Chemistry

Synthesis, structure, and thermal destruction of aroxytetraphenylstiboranes

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A series of aroxytetraphenylstiboranes, Ph_4SbOAr, were obtained by the reaction of pentaphenylstiborane with phenols at ~20 °C. The thermolysis of these compounds gives *O*- or *o*-*C*-phenylation products. The thermolysis of stiboranes, which incorporate aryl groups containing electron-withdrawing substituents (Ar = 2,4-Br_2, 2,4-Cl_2, 2-NO_2, 4-OPh) produces predominantly simple diaryl ethers of asymmetric structure in 58 %, 90 %, 32 %, and 60 % yields, respectively.

Key words: aroxytetraphenylstiboranes, thermal decomposition; X-ray study.

Phenoxytetraphenylstiborane obtained by treatment of pentaphenylstiborane with phenol undergoes decomposition when heated to 180 °C to give triphenylstiborane and diphenyl ether in quantitative yields.[1] Obviously, this reductive elimination can serve as a base for creating a method for the synthesis of asymmetric diaryl ethers from aroxytetraphenylstiboranes and similar compounds. In order to determine whether aryl derivatives of Sb^V can be used in organic synthesis, we obtained a series of hitherto unknown aroxy-derivatives of tetraphenylstibonium and analyzed the products of their thermal decomposition.

Aroxytetraphenylstiboranes were synthesized by reactions of pentaphenylstiborane with phenols in toluene at room temperature. The reaction occurred much more readily when the number of electron-withdrawing substituents in the starting phenol increased, which is probably due to a simultaneous increase in its acid properties.

$$Ph_5Sb + ArOH \longrightarrow \underset{\mathbf{1-19}}{Ph_4SbOAr} + PhH$$

Stiboranes **1**—**19** synthesized are colorless crystalline compounds (excepting the nitro derivatives, which are yellow) soluble in aromatic hydrocarbons and in polar organic solvents. The highest melting point is observed

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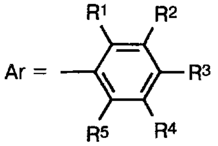
Table 1. Properties and elemental analysis data for aroxytetraphenylstiboranes of general formula Ph_4SbOAr



Compound	R^1	R^2	R^3	R^4	R^5	M.p. /°C	Yield (%)	Found/Calculated (%) C	Found/Calculated (%) H	Molecular formula
1	Br	H	NO_2	H	Br	288	94	49.55 49.59	3.00 3.03	$C_{30}H_{22}Br_2NO_3Sb$
2	H	H	I	H	H	142	91	55.44 55.47	3.67 3.70	$C_{30}H_{24}IOSb$
3	H	H	OMe	H	H	142	90	67.11 67.27	4.78 4.88	$C_{31}H_{27}O_2Sb$
4	Br	H	Br	H	H	163	95	52.76 52.86	3.35 3.38	$C_{30}H_{23}Br_2OSb$
5	H	H	NO_2	H	H	151	85	63.31 63.38	4.20 4.23	$C_{30}H_{24}NO_3Sb$
6	NO_2	H	NO_2	H	H	188	88	58.68 58.73	3.73 3.75	$C_{30}H_{23}N_2O_5Sb$
7	NO_2	H	H	H	H	156	95			$C_{30}H_{24}NO_3Sb$*
8	H	H	C(O)H	H	H	173	89			$C_{31}H_{25}O_2Sb$*
9	Br	H	C(O)H	H	H	153	86	58.95 59.05	3.78 3.81	$C_{31}H_{24}BrO_2Sb$
10	Br	H	Bu^t	H	Br	195	92	55.12 55.36	4.11 4.21	$C_{34}H_{31}Br_2OSb$
11	Br	H	Me	H	Br	195	90	53.47 53.53	3.51 3.60	$C_{31}H_{25}Br_2OSb$
12	Cl	H	H	H	Cl	223	93	60.59 60.81	3.77 3.89	$C_{30}H_{23}Cl_2OSb$
13	Cl	H	Cl	H	H	163	78	60.49 60.81	3.81 3.89	$C_{30}H_{23}Cl_2OSb$
14	H	H	Cl	H	H	130	75	64.53 64.57	4.28 4.30	$C_{30}H_{24}ClOSb$
15	H	H	Bu^t	H	H	105	72	70.26 70.47	5.65 5.70	$C_{34}H_{33}OSb$
16	Bu^t	H	Bu^t	H	H	135	67	71.69 71.81	6.41 6.46	$C_{38}H_{41}OSb$
17	Br	Me	Bu^t	H	Bu^t	192	81	64.07 64.29	5.62 5.77	$C_{39}H_{42}BrOSb$
18	Br	H	Bu^t	H	Bu^t	205	80	63.55 63.87	5.54 5.60	$C_{38}H_{40}BrOSb$
19	Br	H	Br	H	Me	211	92	53.41 53.53	3.48 3.60	$C_{31}H_{25}Br_2OSb$

Note. The structures of the compounds were also confirmed by their reactions with HCl in ethanol at ~20 °C resulting in quantitative formation of tetraphenylstibonium chloride and substituted phenols. * According to X-ray diffraction analysis.

in the case of 2,6-dibromo-4-nitrophenoxytetraphenylstiborane (**1**, Table 1).

We also obtained Sb^V compounds of similar structure by treating tetraphenylstibonium bromide with sodium phenolates or with phenols in the presence of an acceptor of hydrogen bromide in benzene or dioxane. However, in this case the yields of the target products were lower and did not exceed 70 %.

$$Ph_4SbBr + ArONa \longrightarrow Ph_4SbOAr + NaBr$$

$$Ph_4SbBr + ArOH \xrightarrow{Py} Ph_4SbOAr + HBr \cdot Py$$

Table 2. Properties and elemental analysis data for products of thermal decomposition of aroxytetraphenylstiboranes Ph_4SbOAr (220 °C, 2 h)

Starting compounds	Thermolysis products	Yield (%)	Found / Calculated (%)		Molecular formula
			C	H	
4	2,4-Dibromophenoxybenzene (colorless viscous liquid)	58	43.53 43.90	2.16 2.44	$C_{12}H_8Br_2O$
7	2-Nitrophenoxybenzene (viscous liquid)	32	66.40 66.98	3.89 4.19	$C_{12}H_9NO_3$
	2-Nitrophenol	22			
8	4-Oxophenoxybenzene (colorless viscous liquid)	60	78.47 78.79	4.88 5.05	$C_{13}H_{10}O_2$
13	2,4-Dichlorophenoxybenzene (colorless viscous liquid)	90	59.93 60.25	3.28 3.35	$C_{12}H_8Cl_2O$

A modification of the latter synthetic method involving treatment of aqueous solutions of a phenol and a tetraphenylstibonium halide with an amine makes it possible to simplify the procedure for synthesizing aroxytetraphenylstiboranes and to obtain a chemically pure compound after filtration and drying.

The thermal decomposition of aroxytetraphenylstiboranes was carried out in the absence of a solvent or in toluene at temperatures above the decomposition points. It was shown that the direction of the thermolysis of the above compounds is determined by the nature and positions of substituents at ArO. For example, if the aroxy group contains electron-withdrawing substituents, asymmetric diaryl ethers are formed. In some cases, the reaction products contain not only a diether but also the corresponding phenol, as in the thermolysis of 2-nitrophenoxytetraphenylstiborane **7** (Table 2).

$$Ph_4SbOAr \xrightarrow{\Delta} PhOAr$$

It should be noted that the *O*- and *o*-*C*-phenylation of phenols described previously for Bi^V derivatives[2—4] resulted in somewhat lower yields of products in the case of organoantimony compounds.

The structures of 2-nitrophenoxytetraphenylstiborane (**7**) and *p*-formylphenoxytetraphenylstiborane (**8**) were confirmed by X-ray diffraction analysis. The Sb atoms in molecules **7** and **8** (Figs. 1 and 2, respectively) have a trigonal-bipyramidal coordination typical of pentacoordinated Sb^V (the sums of angles in the equatorial plane are 357.6° and 357.2°, while the axial angles are 177.4(2)° and 171.8(1)° for **7** and **8**, respectively). As calculations show,[5,6] this type of coordination is somewhat more beneficial energetically than the tetragonal-pyramidal coordination. The latter type has been observed[7,8] only in the crystalline structure of nonsolvated pentaphenylstiborane. On the other hand, antimony has the usual trigonal-bipyramidal coordination in a crystal solvate of this compound with cyclohexane, $Ph_5Sb \cdot 0.5C_6H_{12}$.[9]

The peculiarities of the structure of trigonal-bipyramidal complexes of group V elements are nicely described by the valent shell electron pair repulsion theory (VSEPR).[10,11] In accordance with one of the bases of this theory, the most electronegative substituents occupy the axial positions in the pentagonal-bipyramidal environment of the central atom. The equatorial positions in complexes **7** and **8** are occupied by phenyl substituents (the C(Ph)—Sb—C(Ph) angles between them are within 114.3(1)—124.0(1)° and 116.6(2)—122.5(2)° for **7** and **8**, respectively), while the axial positions contain the

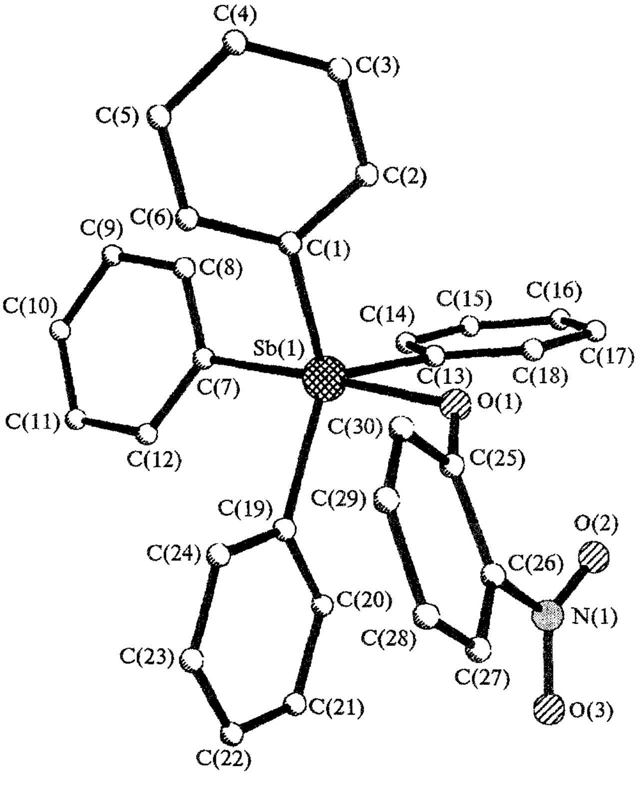


Fig. 1. Structure of molecule **7**.

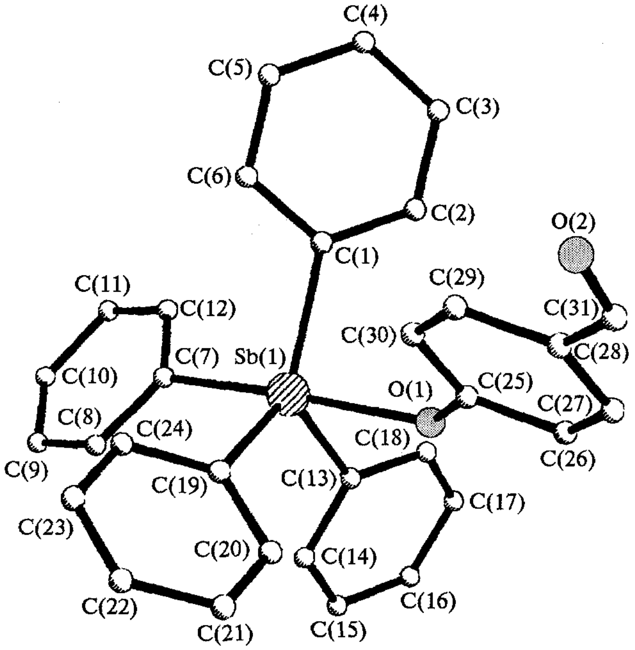


Fig. 2. Structure of molecule **8**.

most electronegative substituents. In fact, the relatively more electronegative (with respect to the unsubstituted phenyl ring) *o*-nitrophenoxy group (in molecule **7**) and *p*-formylphenoxy group (in molecule **8**) are in the axial positions. As expected, all angles between the most electronegative axial ligand and the equatorial phenyl rings are markedly less than 90° (the O_{ax}—Sb—C_{eq} angles are 83.6(1)—86.3(1)° and 80.8(1)—87.1(2)° in structures **7** and **8**, respectively). Within the VSEPR theory, this is explained by repulsion of the more bulky electron pairs of the valent shells of the C atoms of the axial and equatorial phenyl rings. Naturally, if the axial positions contain groups with equal electronegativities (*e.g.*, in $Ph_3(SbOMe)_2$),[12] all angles between the axial and equatorial substituents are close to 90° (the O_{ax}—Sb—C_{eq} angle is within 85.5(4)—93.4(4)°). However, if the substituents at the axial positions differ (as in molecules **7** and **8**), the equatorial substituents deviate towards the most electronegative axial substituent (all bond angles O_{ax}—Sb—C_{eq} in the structure of Ph_4SbOMe are also within 81.5(5)—89.5(4)°).[12]

The Sb—C_{eq} distances in both structures are shorter than Sb—C_{ax} (2.115(4)—2.123(3) and 2.181(5) Å in molecule **7**; 2.118(4)—2.139(5) and 2.188(5) Å in molecule **8**, respectively). The O—C(Ph) bond lengths are the same within the experimental error (1.317(6) and 1.321(5) Å for **7** and **8**, respectively). Thus, neither the position nor the nature of the substituent in the phenoxy group substantially affects the O—C(Ph) bond. The Sb—O distance in molecule **7** is somewhat greater (by 0.019 Å) than that in **8**, which agrees with the smaller Sb—O—C(Ph) angle in **7** (the Sb—O bond lengths in structures **7** and **8** are 2.221(4) and 2.202(3) Å, while the Sb—O—C(Ph) angles are 123.8(3)° and 129.6(3)°, respectively).

Experimental

^1H NMR spectra were recorded on a Tesla BS-567A NMR spectrometer (100 MHz). Acetone-d_6 or $CDCl_3$ were used as solvents, and HMDS served as the internal standard. IR spectra were recorded on a UR-20 spectrophotometer (suspensions in Vaseline oil, liquid films, or solutions in $CHCl_3$ between KBr plates).

Aroxytetraphenylstiboranes were synthesized in evacuated glass tubes according to the following procedures.

A. A mixture of pentaphenylstiborane (5 mmol) and 2-nitrophenol (5 mmol) in toluene (10 mL) was kept for 24 h at ~20 °C. The crystals that formed were washed with hexane and dried.

Table 3. Atomic coordinates (×10^4) and their equivalent isotropic temperature factors (×10^3) in structure **7**

Atom	*x*	*y*	*z*	U_{iso}/Å^2	Atom	*x*	*y*	*z*	U_{iso}/Å^2
Sb(1)	1813(1)	8918(1)	7784(1)	39(1)	C(14)	4363(5)	8115(5)	7762(4)	59(2)
N(1)	3490(5)	12787(4)	7038(4)	73(2)	C(15)	5448(5)	7875(6)	7397(4)	71(2)
O(1)	1552(3)	9891(3)	6500(2)	51(1)	C(16)	5670(5)	8134(5)	6480(5)	68(2)
O(2)	3995(5)	12111(5)	6558(5)	126(3)	C(17)	4838(6)	8662(6)	5913(4)	73(3)
O(3)	4279(5)	13986(4)	7619(4)	103(2)	C(18)	3756(5)	8925(5)	6283(4)	59(2)
C(1)	−360(4)	7349(4)	6868(3)	46(1)	C(19)	2261(4)	10875(4)	8839(3)	39(1)
C(2)	−748(7)	6806(6)	5774(4)	87(2)	C(20)	3727(4)	11868(4)	9299(3)	48(2)
C(3)	−2129(8)	5620(8)	5203(5)	115(3)	C(21)	4045(4)	13147(4)	10014(4)	55(2)
C(4)	−3074(6)	5093(5)	5739(5)	85(3)	C(22)	2918(5)	13433(4)	10244(4)	58(2)
C(5)	−2691(5)	5672(6)	6834(5)	72(2)	C(23)	1454(5)	12453(5)	9777(3)	57(2)
C(6)	−1339(5)	6820(5)	7408(4)	58(2)	C(24)	1123(4)	11170(4)	9083(3)	48(2)
C(7)	2031(4)	8019(4)	9085(4)	46(2)	C(25)	1007(4)	10814(4)	6537(3)	46(2)
C(8)	1577(5)	6608(4)	8837(4)	55(2)	C(26)	1900(4)	12254(4)	6842(3)	49(2)
C(9)	1657(6)	5993(5)	9640(5)	70(2)	C(27)	1293(6)	13210(5)	6990(4)	67(2)
C(10)	2209(6)	6810(6)	10717(5)	70(3)	C(28)	−187(7)	12783(6)	6787(5)	76(3)
C(11)	2667(5)	8221(5)	10989(4)	61(2)	C(29)	−1104(5)	11387(6)	6442(4)	71(2)
C(12)	2575(4)	8820(4)	10175(3)	51(2)	C(30)	−533(5)	10430(5)	6320(4)	59(2)
C(13)	3522(4)	8652(4)	7223(3)	46(2)					

Table 4. Atomic coordinates ($\times 10^4$) and their equivalent isotropic temperature factors ($\times 10^3$) in structure **8**

Atom	x	y	z	U_{iso}/Å^2	Atom	x	y	z	U_{iso}/Å^2
Sb(1)	3002(1)	2426(1)	8389(1)	38(1)	C(15)	7118(6)	3966(4)	8767(4)	60(2)
O(1)	4297(4)	1294(2)	8809(2)	50(1)	C(16)	7617(6)	3916(4)	9514(4)	62(2)
O(2)	1842(6)	−2466(2)	8087(4)	84(2)	C(17)	6835(7)	3426(4)	9964(3)	66(2)
C(1)	1280(5)	1988(3)	9017(3)	42(1)	C(18)	5581(6)	2960(4)	9650(3)	55(2)
C(2)	1662(6)	1549(4)	9675(3)	58(2)	C(19)	3009(6)	1933(3)	7297(3)	45(2)
C(3)	526(8)	1220(4)	10047(4)	69(2)	C(20)	4213(6)	1417(3)	7133(3)	57(2)
C(4)	−1019(7)	1332(4)	9752(4)	69(2)	C(21)	4168(8)	1084(4)	6436(4)	70(2)
C(5)	−1384(6)	1746(4)	9080(4)	61(2)	C(22)	2960(10)	1247(5)	5882(4)	81(3)
C(6)	−235(6)	2095(3)	8712(3)	50(2)	C(23)	1797(9)	1736(4)	6040(4)	76(3)
C(7)	1977(5)	3653(3)	8066(3)	43(1)	C(24)	1785(7)	2094(4)	6746(3)	58(2)
C(8)	2307(6)	4072(3)	7437(3)	55(2)	C(25)	3934(5)	489(3)	8713(3)	40(1)
C(9)	1951(7)	4928(4)	7343(4)	65(2)	C(26)	5019(5)	−125(3)	8993(3)	47(2)
C(10)	1277(7)	5355(3)	7868(4)	65(2)	C(27)	4709(5)	−970(3)	8881(3)	48(2)
C(11)	928(7)	4939(3)	8491(4)	63(2)	C(28)	3306(5)	−1249(3)	8504(3)	41(1)
C(12)	1288(6)	4103(3)	8594(3)	52(2)	C(29)	2216(5)	−636(3)	8233(3)	46(2)
C(13)	5047(5)	3011(3)	8899(3)	41(1)	C(30)	2510(6)	199(3)	8329(3)	50(2)
C(14)	5838(5)	3523(4)	8455(3)	52(2)	C(31)	3006(7)	−2143(4)	8390(3)	60(2)

Table 5. Main bond lengths (d) in molecules **7** and **8**

Molecule 7		Molecule 8	
Bond	d/Å	Bond	d/Å
Sb(1)—O(1)	2.221(4)	Sb(1)—O(1)	2.202(3)
Sb(1)—C(1)	2.123(3)	Sb(1)—C(1)	2.139(5)
Sb(1)—C(7)	2.181(5)	Sb(1)—C(7)	2.188(5)
Sb(1)—C(13)	2.121(5)	Sb(1)—C(13)	2.117(4)
Sb(1)—C(19)	2.115(4)	Sb(1)—C(19)	2.121(5)
O(1)—C(25)	1.317(6)	O(1)—C(25)	1.321(5)
N(1)—O(2)	1.197(8)	O(2)—C(31)	1.206(8)
N(1)—O(3)	1.220(5)		
N(1)—C(26)	1.449(6)		

B. A solution of tetraphenylstibonium bromide (5 mmol) and sodium 2-nitrophenolate (5 mmol) in dioxane (10 mL) was kept at ~20 °C. The solvent was removed *in vacuo*. The residue was repeatedly extracted with hot benzene, whereupon crystals of **7** precipitated, yield 62 %.

C. A solution of 2-nitrophenol (5 mmol) and pyridine (6 mmol) in water (20 mL) was added with stirring to a hot solution of tetraphenylstibonium bromide (5 mmol) in water (100 mL). The mixture was kept for 1 h at 90 °C and cooled. The resulting crystals were filtered off, washed with water, and dried, yield 70 %.

Thermal decomposition of aroxytetraphenylstiboranes was carried out in a П-shaped evacuated glass setup. Compound **8** (5 mmol) was heated for 2 h at 220 °C. The liquid that condensed in the cooled part of the setup was chromatographed on Al$_2$O$_3$ (hexane as the eluent) to give 3 mmol (60 %) of 4-oxophenoxybenzene as a syrupy liquid [IR, ν/cm^{-1}: 1780, 1665, 1260, 1025; ^{1}H NMR (acetone-d$_6$), δ: 9.94 (s, 1 H, C(O)H); 7.9—6.9 (m, 9 H, Ar)] and 1.8 mmol (36 %) of 4-oxyphenol [m.p. 117 °C; IR (Vaseline oil), ν/cm^{-1}: 3550, 1630, 1560; ^{1}H NMR (acetone-d$_6$), δ: 9.82 (s, 1 H, C(O)H); 7.77 (s, 2 H, Ar); 6.96 (s, 2 H, Ar)].

X-Ray diffraction experiments were carried out on a Siemens P3/PC automatic four-circle diffractometer (λ(Mo-Kα), graphite monochromator, θ/2θ-scanning). The crystals of compound **7** are triclinic, and those of compound **8** are monoclinic; at 20 °C, a = 10.194(4) and 8.827(2) Å, b = 10.963(4) and

Table 6. Main bond angles (ω) in molecules **7** and **8**

Molecule 7		Molecule 8	
Angle	ω/deg	Angle	ω/deg
O(1)—Sb(1)—C(1)	86.3(1)	O(1)—Sb(1)—C(1)	85.8(1)
O(1)—Sb(1)—C(7)	177.4(2)	O(1)—Sb(1)—C(7)	171.8(1)
C(1)—Sb(1)—C(7)	93.8(1)	C(1)—Sb(1)—C(7)	97.6(2)
O(1)—Sb(1)—C(13)	84.6(2)	O(1)—Sb(1)—C(13)	80.8(1)
C(1)—Sb(1)—C(13)	114.3(1)	C(1)—Sb(1)—C(13)	122.6(2)
C(7)—Sb(1)—C(13)	97.6(2)	C(7)—Sb(1)—C(13)	91.2(2)
O(1)—Sb(1)—C(19)	83.6(1)	O(1)—Sb(1)—C(19)	87.1(2)
C(1)—Sb(1)—C(19)	124.0(2)	C(1)—Sb(1)—C(19)	118.1(2)
C(7)—Sb(1)—C(19)	94.2(2)	C(7)—Sb(1)—C(19)	97.8(2)
C(13)—Sb(1)—C(19)	119.3(1)	C(13)—Sb(1)—C(19)	116.6(2)
Sb(1)—O(1)—C(25)	123.8(3)	Sb(1)—O(1)—C(25)	129.6(3)
O(2)—N(1)—O(3)	120.8(5)	O(2)—C(31)—C(28)	127.2(5)
O(2)—N(1)—C(26)	120.4(4)		

15.844(5) Å, c = 13.291(5) and 18.041(5) Å, α = 102.69(2)°, β = 102.18(2)° and 98.32(2)°, γ = 111.43(1)°, V = 1278(2) and 2497(1) A^3, space groups $P\bar{1}$ (Z = 2) and $P2_1/c$ (Z = 4), d_{calc} = 1.477 and 1.467 g cm^{-3} for **7** and **8**, respectively. Both structures were solved by the direct method and refined by the full-matrix least-squares method in the anisotropic approximation to R = 0.052, R_w = 0.067 for 4360 reflections with $I > 3\sigma(I)$ for **7** and to R = 0.043, R_w = 0.059 for 4516 reflections with $I > 3\sigma(I)$ for **8**. The H atoms in both structures were localized on a difference synthesis and refined isotropically. The calculations were carried out on an IBM PC computer using SHELXTL PLUS programs.[13] Atomic coordinates and their equivalent temperature factors in structures **7** and **8** are given in Tables 3 and 4, respectively. Main bond lengths and bond angles are presented in Tables 5 and 6.

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