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Structural Characteristics of Aryloxybismuthanes Stabilized by Hypervalent Bond Formation. Synthesis, Incorporation of 4-Methoxyphenol through Hydrogen Bonding, and Crystal Supramolecularity

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10-Phenoxyphenothiabismine 5,5-dioxide (**2a**) and related compounds **2b**,**c**, the first example of an air-stable aryloxybismuthane whose bismuth−oxygen bond is not incorporated into a ring system, were synthesized and fully characterized. Substitution of the OAr group in **2** by phenols in solution revealed two substitution tendencies. The substitution with 2-methoxyphenol proceeded preferentially due to intramolecular coordination of the 2-methoxy oxygen atom with the bismuth atom in 10-(2-methoxyphenoxy)phenothiabismine 5,5-dioxide (**2b**). Furthermore, the substitution is more favorable with more acidic phenols, which is in accord with the apicophilicity observed at an apical position in hypervalent compounds. 10-(4-Methoxyphenoxy)phenothiabismine 5,5-dioxide (**2c**) was found to form 10-(4-methoxyphenoxy)phenothiabismine 5,5-dioxide·4-methoxyphenol 1:1 complex (**3**) through hydrogen bonding between the OH group of 4-methoxyphenol and the aryloxide oxygen atom of **2c**. Such complex formation may be the initial step of the substitution reactions. Comparison of the X-ray crystal structures of **2b**, **2c**, and **3** shows that these bismuthanes adopt various association modes, which might show potential utility of bismuth−oxygen coordinate bonds in the construction of three-dimensional organometallic systems.

Introduction

Bismuth triaryloxides are attracting interest as potential precursors for bismuth-containing oxide superconductors, and several compounds of this class have been characterized by X-ray crystallography. For example, bismuth tris(2,6-dimethylphenoxide)[1] adopts a monomeric structure due to the steric bulk of the aryloxide ligands, while bismuth tris(pentafluorophenoxide)[2] is an aryloxide-bridged dimer. Bismuth tris(2-methoxyethoxide)[3,4] comprises a one-dimensional alkoxide-bridged chain. These findings show that the association mode of bismuth triaryloxides and trialkoxides is affected by the aryloxy or alkoxy ligand. This suggests that the aryloxy- and alkoxybismuthanes, $R_nBi(OR')_{3-n}$ ($n = 1, 2$), will show similar structure effects. To our knowledge, however, examples of X-ray crystallographic studies of an aryloxy-[5] or alkoxybismuthane[6] are few in number, although several synthetic studies have been reported.[7] Diethyl(pentafluorophenoxy)bismuthane[5] is the sole example in aryloxybismuthane characterized by a crystal structure determination. This compound was reported to be quite air-sensitive and to form C_6F_5O-bridged chain polymers. Herein, we describe synthesis, substitution at the bismuth center in solution, and association in the solid state of aryloxybismuthanes **2** that are stabilized by hypervalent bond formation.[8] These are the first examples of air-stable aryloxybismuthanes whose bismuth−oxygen bond is not incorporated into a ring system.[6]

Results and Discussion

Aryloxybismuthanes **2** were synthesized by the reaction of 10-chlorophenothiabismine 5,5-dioxide (**1**)[9] with the corresponding lithium aryloxide (1.2 equiv) in THF

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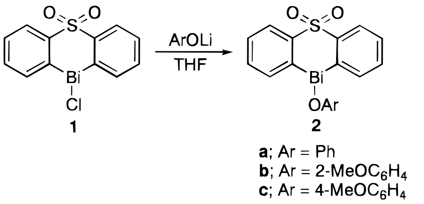
(8) By using hypervalent bond formation we have recently succeeded in the first synthesis of an optically pure iodobismuthane: Murafuji, T.; Satoh, K.; Sugihara, Y.; Azuma, N. *Organometallics* **1998**, *17*, 1711.

10.1021/om9908534 CCC: $19.00

at −20 °C (eq 1).

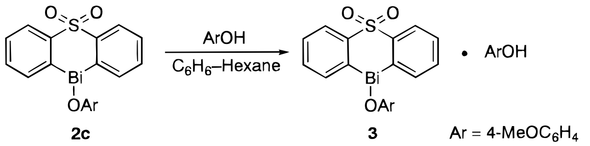


(1)

After an aqueous workup of the reaction mixture with brine at room temperature, the organic layer was concentrated to leave an oily residue, which was crystallized from benzene/hexane (3:1) to give the aryloxybismuthanes **2** in 60−70% yield. Unlike the usual aryloxybismuthanes, such as diphenylphenoxybismuthane,[7c] which shows moisture sensitivity, the bismuthanes **2** are air-stable and unaffected by the aqueous workup. This enhanced stability may be attributed to the hypervalent bond formation involving the sulfonyl oxygen, bismuth, and Bi-aryloxy oxygen atoms.

The aryoxybismuthanes **2** were found to undergo substitution at the bismuth atom by phenols in solution. An experiment in which **2c** was allowed to react with 2-methoxyphenol (**2c**/ArOH = 1.0:1.7 equiv) produced **2b** (**2b**/**2c** = 85:15), while a similar experiment, the reaction of **2b** with 4-methoxyphenol (**2b**/ArOH = 1.0: 1.7 equiv), proceeded with difficulty (**2b**/**2c** = 60:40). Such a pronounced difference in reactivity between **2b** and **2c** toward substitution at bismuth possibly is due to intramolecular coordination of the 2-methoxy oxygen atom with the bismuth atom to form a five-membered chelate ring in **2b**. Similar reactions of **2a** with 4-methoxyphenol (**2a**/ArOH = 1.0:1.0 equiv) and of **2c** with phenol (**2c**/ArOH = 1.0:1.0 equiv) resulted **2a**/**2c** ratios in solution of ca. 61:39 and 62:38, respectively. This result indicates that the substitution at the bismuth atom is more favorable with more acidic phenols, which is in accord with the tendency that a more electron-withdrawing substituent prefers the apical position at the hypervalent center.

Isolation of the substitution product was carried out. Thus, when a mixture of **2a** and excess 2-methoxyphenol (1.7 equiv) in benzene/hexane (5:1) was kept at room temperature, preferential crystallization of **2b** occurred. Reaction of **2c** with an excess of 2-methoxyphenol under similar conditions also afforded **2b**. In reactions of **2b** with 4-methoxyphenol or phenol, solid **2b** preferentially separated. Upon mixing of **2a** and an excess of 4-methoxyphenol, a mixture of a 1:1 complex, 10-(4-methoxyphenoxy)phenothiabismine 5,5-dioxide·4-methoxyphenol **3** and **2c** separated. The preferential crystallization of **2c** is considered to be due to the difference in solubility between **2a** and **2c**. Eventually, it was found that complex **3** is formed when a mixture of **2c** with an excess of 4-methoxyphenol (1.7 equiv) in benzene/hexane (5:1) is stored at room temperature; such complex formation is specific for **3** (eq 2).



(2)

Bismuthane **2c** (mp 178−180 °C) and 4-methoxyphenol (mp 55−57 °C) are colorless crystals, while complex **3** (mp 116−118 °C) is pale yellow. The UV/vis and ^{1}H NMR spectra of **3** are additive of those of **2c** and 4-methoxyphenol, respectively. The IR spectrum of solid **3** measured in KBr shows a broad absorption band due to the OH stretching vibration (3200 cm^{-1}), which is shifted by 150 cm^{-1} to lower wavenumber compared with that of 4-methoxyphenol (Figure 1). This suggests that complex **3** is hydrogen bonded in the solid state, but that it dissociates into **2c** and 4-methoxyphenol in solution.

To clarify how the hydroxyl group of 4-methoxyphenol participates in such complex formation through hydrogen bonding, an X-ray structure analysis of **3** was carried out (Figure 2, Tables 1 and 2). Despite the presence of the Lewis basic sulfonyl oxygen atoms, hydrogen bonding to the aryloxide oxygen atom O(3) was observed. In addition, the distance between the O(3) and hydroxyl O(5) atoms [2.78(1) Å] is in excellent agreement with that (ca. 2.78 Å) estimated by the empirical correlation with the wavenumber of the OH stretching vibration observed in the IR spectra.[10] Complex **3** is considered to represent the initial step of the substitution reaction in solution. The intramolecular distance between the bismuth atom and the sulfonyl oxygen atom O(1) [2.775(7) Å] is within the sum of the van der Waals radii (3.72 Å), which suggests the formation of a hypervalent bond over the O(1), Bi, and O(3) atoms. Furthermore, the intermolecular Bi···O(1) distance, 3.412(6) Å, indicates that complex **3** is present as a dimer (Scheme 1).

An X-ray structure analysis revealed that **2c** has a dimeric structure through intermolecular interaction between the bismuth and aryloxide oxygen O(3) atoms. The intermolecular Bi···O(3) distance is 2.787(9) Å, which is comparable to the intramolecular Bi···O(1) distance of 2.765(9) Å and is much shorter than the intermolecular Bi···O(1) distance in **3** (Table 2). Comparison of the crystal structure of **3** with that of **2c** shows the difference in the coordination mode between these bismuth centers brought about by the incorporation of 4-methoxyphenol (Scheme 1). While bismuthane **2c** constitutes a dimeric structure by the self-assembly through the coordinate bond between the bismuth and aryloxide oxygen atoms, **3** alters the coordination of the bismuth center to the sulfonyl oxygen atom, due to incorporation of 4-methoxyphenol as a guest molecule with hydrogen bonding. In both structures the electron-withdrawing ability of the aryloxide oxygen apparently is enhanced by the coordination bond with the bismuth atom or the hydrogen bond, to strengthen the respective hypervalent bonds.

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(9) Suzuki, H.; Murafuji, T.; Azuma, N. *J. Chem. Soc.*, *Perkin Trans. 1* **1992**, 1593. Hypervalent bond formation by an intramolecular coordination of a sulfonyl oxygen atom with a bismuth atom: (a) Suzuki, H.; Murafuji, T. *J. Chem. Soc.*, *Chem. Commun.* **1992**, 1143. (b) Suzuki, H.; Murafuji, T.; Azuma, N. *J. Chem. Soc.*, *Perkin Trans. 1* **1993**, 1169.

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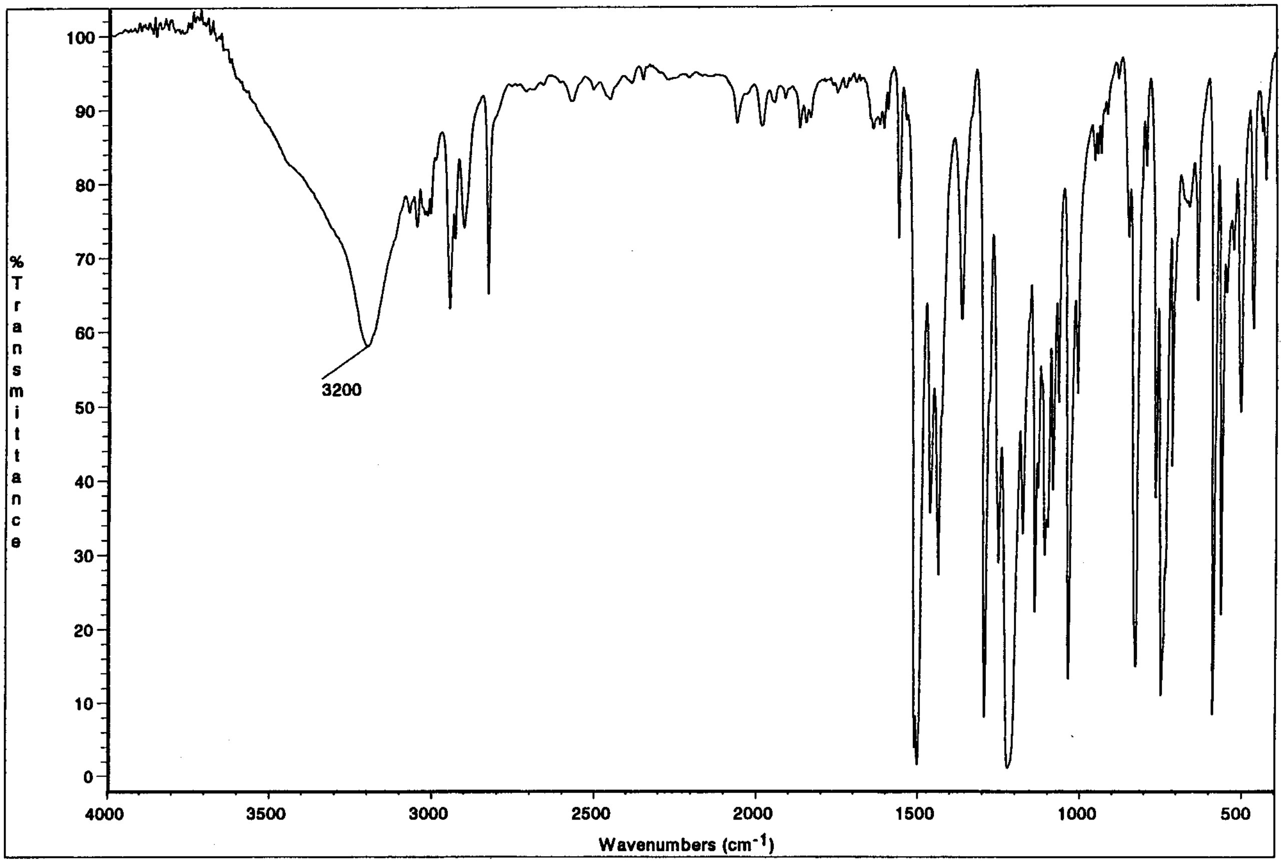

Figure 1. IR spectrum of **3** in KBr.

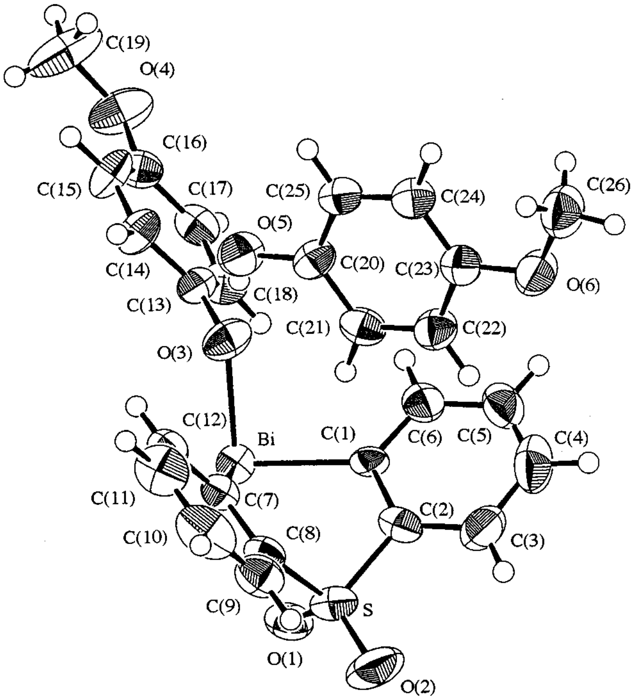

Figure 2. ORTEP drawing of the molecular structure of **3** with 50% probability ellipsoids.

Bismuthane **2b** was found to construct a supramolecular structure[11] in the crystal in which the bismuth center adopts a high coordination geometry[12] with five Bi−O coordinate bonds (Figure 3, Table 2 and Scheme 1). In addition to the intramolecular coordination of the sulfonyl oxygen O(2) atom [Bi···O(2), 2.743(5) Å], the 2-methoxy oxygen atom O(4) also coordinates with the bismuth atom in the same molecule [Bi···O(4), 2.833(4) Å]. Furthermore, two oxygen atoms, that is, the aryloxide oxygen O(3) and sulfonyl oxygen O(2) atoms, also participate in intermolecular Bi−O association with distances of 3.122(4) and 3.367(4) Å, respectively. The hypervalent bond O(2)−Bi−O(3) [153.3(1)°] in **2b** is slightly bent compared with that of O(1)−Bi−O(3) [158.9(3)°] in **2c**, reflecting the high coordination center of the bismuth atom in **2b**.

Experimental Section

General Comments. All reactions were carried out under argon. Diethyl ether and THF were distilled from calcium hydride under nitrogen before use. Bismuth(III) chloride was purified by refluxing with thionyl chloride. ^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ on a Bruker AVANCE 400S (400 MHz) spectrometer with tetramethylsilane as an internal standard. UV/vis spectra were measured in $CHCl_3$ on a HITACHI 220A spectrophotometer. IR spectra were obtained as KBr pellets on a Nicolet Impact 410 spectrophotometer.

10-Aryloxyphenothiabismine 5,5-Dioxide (2). General Procedure. To a solution of 10-chlorophenothiabismine 5,5-dioxide **1**[9] (460 mg, 1 mmol) in THF (5 mL) was added at −20 °C a solution of LiOAr (1.2 mmol) prepared from ArOH (1.2 mmol) and *n*-butyllithium (1.2 mmol) in the same solvent (5 mL), and the mixture was stirred for 30 min, during which

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[11] For recent work on the construction of a three-dimensional organometallic system containing a bismuth center: Genge, A. R. J.; Levason W.; Reid, G. *Chem. Commun.* **1998**, 2159.

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Table 1. Crystal and Structure Determination Data for 2b, 2c, and 3

	2b	2c	3
formula	$C_{19}H_{15}BiO_4S$	$C_{19}H_{15}BiO_4S$	$C_{26}H_{23}BiO_6S$
fw	548.37	548.37	672.50
a, Å	10.864(2)	24.177(9)	17.817(6)
b, Å	11.460(3)	7.174(2)	8.443(2)
c, Å	8.481(2)	21.940(6)	18.232(5)
α, deg	109.37(2)		
β, deg	110.76(1)	108.32(2)	117.71(2)
γ, deg	97.50(2)		
V, Å^3	893.9(4)	3612(2)	2428(1)
cryst color and shape	colorless and prismatic	colorless and prismatic	pale yellow and plate
cryst syst	triclinic	monoclinic	monoclinic
space group	$P\bar{1}$ (No. 2)	$C2/c$ (No. 15)	$P2_1/n$ (No. 14)
Z	2	8	4
F(000)	520	2080	1304
D_{calcd}, g cm^{-3}	2.037	2.016	1.839
cryst dimens, mm	0.50 × 0.45 × 0.20	0.30 × 0.25 × 0.07	0.07 × 0.25 × 0.35
μ, cm^{-1}	99.64	98.62	73.60
collection range	$2\theta_{max} = 63.0°$	$2\theta_{max} = 55.0°$	$2\theta_{max} = 55.0°$
scan rate, deg min^{-1}	16.0	8.0	16.0
scan width, deg	$1.47 + 0.30 \tan \theta$	$1.52 + 0.30 \tan \theta$	$1.21 + 0.30 \tan \theta$
no. of unique reflns	4134	4477	5965
no. of obsd data	4101 [$I > 0.00\sigma(I)$]	2571 [$I > 2.00\sigma(I)$]	3211 [$I > 2.00\sigma(I)$]
no. of variables	226	226	310
range of transmn factors	0.41−1.00	0.32−1.00	0.45−1.00
R^a	0.052	0.054	0.050
$R_w{}^a$	0.068	0.047	0.041
max peak in final Fourier map, e Å^{-3}	2.01	1.87	1.39
min peak in final Fourier map, e Å^{-3}	−1.87	−2.04	−1.45
goodness of fit	1.52	1.72	1.27

[a] $R = \sum||F_o| - |F_c||/\sum|F_o|$, $R_w = [(\sum w(|F_o| - |F_c|)^2/\sum wF_o{}^2)]^{1/2}$; $w = 1/\sigma^2(F_o) = 4F_o{}^2/\sigma^2(F_o{}^2)$.

Table 2. Selected Bond Lengths (Å) and Angles (deg) for 2b, 2c, and 3, with esd's in Parentheses

2b		2c		3	
		Bond Lengths			
Bi−O(3)	2.176(4)	Bi−O(3)	2.169(9)	Bi−O(3)	2.153(7)
Bi−C(2)	2.308(5)	Bi−C(2)	2.28(1)	Bi−C(1)	2.25(1)
Bi−C(8)	2.278(6)	Bi−C(8)	2.27(1)	Bi−C(7)	2.25(1)
S−O(2)	1.454(5)	S−O(1)	1.445(9)	S−O(1)	1.458(7)
S−O(1)	1.432(5)	S−O(2)	1.441(9)	S−O(2)	1.426(8)
Bi···O(2)	2.743(5)	Bi···O(1)	2.765(9)	Bi···O(1)	2.775(7)
Bi···O(4)	2.833(4)				
		Intermolecular Distances			
Bi···O(3)	3.122(4)	Bi···O(3)	2.787(9)	Bi···O(1)	3.412(6)
Bi···O(2)	3.367(4)			O(3)···O(5)	2.78(1)
		Bond Angles			
O(3)−Bi−C(2)	98.5(2)	O(3)−Bi−C(2)	94.6(4)	O(3)−Bi−C(1)	90.3(3)
O(3)−Bi−C(8)	83.7(2)	O(3)−Bi−C(8)	94.4(4)	O(3)−Bi−C(7)	86.3(3)
C(2)−Bi−C(8)	84.1(2)	C(2)−Bi−C(8)	87.2(4)	C(1)−Bi−C(7)	86.4(3)
O(2)−Bi−O(3)	153.3(1)	O(1)−Bi−O(3)	158.9(3)	O(1)−Bi−O(3)	151.6(2)

time the temperature was raised to ambient. After addition of brine (5 mL), the mixture was diluted with ethyl acetate (10 mL), and the organic layer was separated, dried over anhydrous sodium sulfate, and evaporated to leave crude **2**. Recrystallization from benzene/hexane (3:1) gave pure product as colorless crystals.

10-Phenoxyphenothiabismine 5,5-Dioxide (2a): yield 66%; mp 203−206 °C; ^{1}H NMR δ 6.85−6.90 (3H, m, OArH), 7.30 (2H, t, J 7.3, OArH), 7.45 (2H, t, J 7.3, ArH), 7.70 (2H, t, J 6.7, ArH), 8.35 (2H, d, J 7.9, ArH), 8.54 (2H, d, J 7.3, ArH); IR (cm^{-1}) 1588, 1485, 1288, 1237, 1142, 760, 740. Anal. Calcd for $C_{18}H_{13}BiO_3S$: C, 41.71; H, 2.53. Found: C, 41.89; H, 2.62.

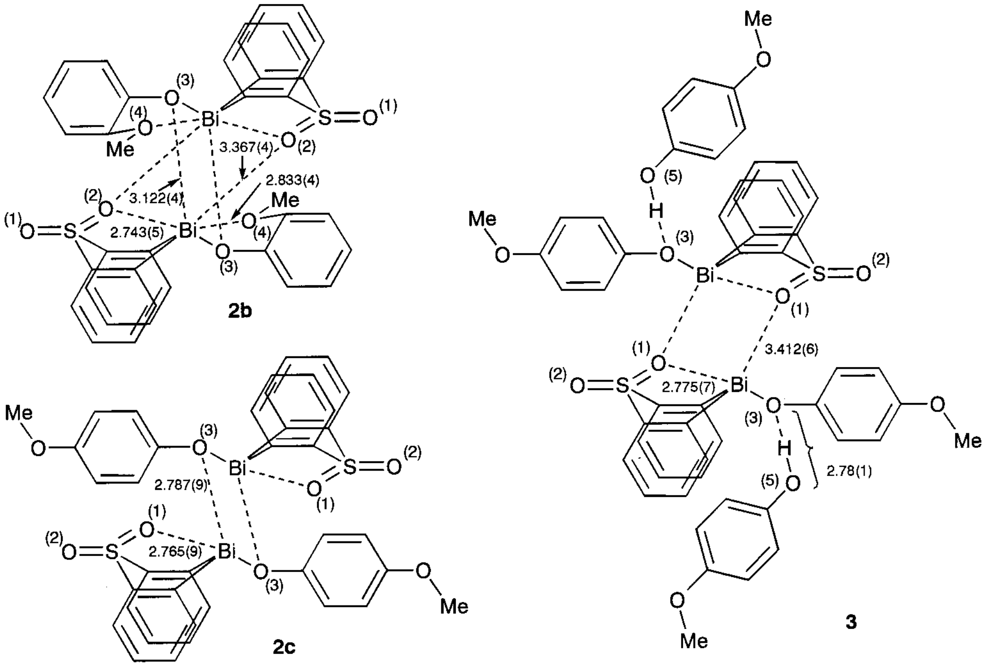
10-(2-Methoxyphenoxy)phenothiabismine 5,5-Dioxide (2b): yield 70%; mp 224−227 °C; ^{1}H NMR δ 4.05 (3H, s, Me), 6.76−6.80 (1H, m, OArH), 6.94−6.98 (3H, m, OArH), 7.41 (2H, t, J 7.6, ArH), 7.68 (2H, t, J 7.4, ArH), 8.32 (2H, d, J 6.9, ArH), 8.64 (2H, d, J 6.7, ArH); ^{13}C NMR δ 55.15, 111.13, 118.15, 120.95, 122.26, 128.31, 128.37, 134.51, 134.91, 141.06, 149.48, 150.75, 187.05; IR (cm^{-1}) 1489, 1285, 1276, 1259, 1143, 1110, 762, 738. Anal. Calcd for $C_{19}H_{15}BiO_4S$: C, 41.62; H, 2.76. Found: C, 41.68; H, 2.87.

10-(4-Methoxyphenoxy)phenothiabismine 5,5-Dioxide (2c): yield 60%; mp 178−180 °C; ^{1}H NMR δ 3.79 (3H, s, Me), 6.85 (4H, m, OArH), 7.44 (2H, t, J 7.4, ArH), 7.70 (2H, t, J 7.4, ArH), 8.34 (2H, d, J 7.1, ArH), 8.52 (2H, d, J 7.1, ArH); ^{13}C NMR δ 55.9. 114.8, 120.2, 128.6, 128.7, 134.6, 135.0, 140.6, 153.0, 156.5, 183.6; UV/vis: λ_{max}/nm (log ϵ) 284 (2.75); IR (cm^{-1}) 1497, 1303, 1228, 1216, 1142, 1030, 836, 765, 738. Anal. Calcd for $C_{19}H_{15}BiO_4S$: C, 41.62; H, 2.76. Found: C, 41.27; H, 2.73.

Substitution of 2 with Phenols. General Procedure. Typical example is exemplified by the experiment of **2a** with 4-methoxyphenol. Aryloxybismuthane **2a** (8.08 mg, 0.0156 mmol) and 4-methoxyphenol (1.93 mg, 0.0156 mmol) were disolved in $CDCl_3$ (0.75 mL), and the ^{1}H NMR spectrum of the solution was measured immediately at room temperature. The ratio of **2a**:**2c** was estimated from that of the integration of peak areas for the methyl protons of 4-methoxyphenol and **2c**.

10-(4-Methoxyphenoxy)phenothiabismine 5,5-Dioxide· 4-Methoxyphenol 1:1 Complex (3). Aryloxybismuthane **2c** (1.097 g, 2 mmol) and 4-methoxyphenol (422 mg, 3.4 mmol) were dissolved in benzene (10 mL), and after addition of

Scheme 1



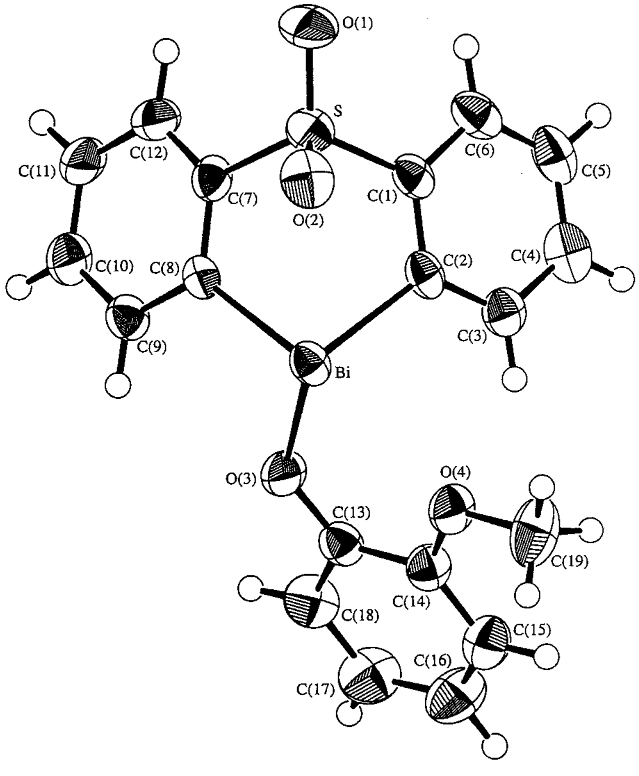


Figure 3. ORTEP drawing of the molecular structure of **2b** with 50% probability ellipsoids.

hexane (2 mL), the mixture was allowed to stand for 2 days at room temperature. Complex **3** gradually separated as pale yellow crystals, which were filtered and collected: yield 75%; mp 116−118 °C; ^{1}H NMR δ 3.76 (3H, s, Me), 3.79 (3H, s, Me), 4.50 (1H, s, OH), 6.78 (4H, m, OArH), 6.85 (4H, m, OArH), 7.44 (2H, t, *J* 7.4, ArH), 7.70 (2H, t, *J* 7.4, ArH), 8.34 (2H, d, *J* 7.1, ArH), 8.52 (2H, d, *J* 7.1, ArH); UV/vis λ_{max}/nm (log ϵ) 288 (2.92); IR (cm^{-1}) 3200, 1509, 1464, 1437, 1293, 1213, 1140, 1110, 1037, 828, 747. Anal. Calcd for $C_{26}H_{23}BiO_6S$: C, 46.44; H, 3.45. Found: C, 46.71; H, 3.46.

X-ray Crystallography. All measurements were made on a Rigaku AFC5R diffractometer with graphite-monochromated Mo Kα radiation (λ = 0.71069 Å) and a 12 kW rotating anode generator. The data were collected at 298 K using the ω−2θ scan technique. The data were corrected for Lorentz and polarization effects. The structures were solved by the Patterson method.[13] The non-hydrogen atoms were refined anisotropically. Neutral-atom scattering factors were taken from Cromer and Waber.[14] Anomalous dispersion effects were included in F_{calcd};[15] the values for $\Delta f'$ and $\Delta f''$ were those of Cromer.[16] All calculations were performed using the TEXSAN[17] crystallographic software package of Molecular Structure Corporation.

Acknowledgment. This work was supported by The Research Fund Grant-in-Aid for Exploratory Research (No. 09874134) and for Scientific Research on Priority Area (A) (Nos. 10146235 and 11120237) from the Ministry of Education, Science, Sports and Culture, Japan.

Supporting Information Available: X-ray data for **2b**, **2c**, and **3**. This material is available free of charge via the Internet at http://pubs.acs.org.

OM9908534

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