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Synthesis and Structural Characterization of a Homoleptic Bismuth Arenethiolate

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Introduction

Current interest in bismuth thiolates derives in part from their utility as fungicides,[2] antitumor agents,[3] vulcanization catalysts,[4] and analytical reagents.[5] Although homoleptic Bi(III) compounds of the genre $Bi(SR)_3$ have been prepared previously,[6] X-ray structural data are available only for bidentate SN (pyridinethiolate)[7] and SS (dithiocarbamate) ligands.[8] Herein we report the preparation and X-ray crystal structure of $[Bi(SAr')_3]$ (**1**; Ar′ = 2,4,6-*t*-$Bu_3C_6H_2$). To our knowledge, this represents the first X-ray structural information on a monodentate tris thiolate of Bi(III).

Experimental Section

General Procedures. All manipulations were performed under a dry, oxygen-free dinitrogen or argon atmosphere using standard Schlenk techniques or in a Vacuum Atmospheres HE-493 drybox. The solvents toluene, hexane, and diethyl ether were freshly distilled from sodium/benzophenone prior to use. ^1H NMR spectra were recorded on a General Electric QE 300 instrument operating at 300.17 MHz and are referenced to Me_4Si (0.0 ppm). The C_6D_6 solvent was dried over 4 Å molecular sieves prior to use. Mass spectra (EI and CI) were recorded on a Bell and Howell 21–491 instrument and elemental analyses were determined on a Perkin-Elmer 2400 analyzer. A solution of *tert*-butyllithium in THF was purchased from Aldrich and titrated prior to use. The $BiCl_3$ was procured commercially (Aldrich) and the thiol, HSC_6H_2(2,4,6-*t*-Bu_3) was prepared as described in the literature.[9]

Preparation of Li(SC_6H_2(2,4,6-*t*-Bu_3)). One equivalent of *t*-BuLi (0.36 mmol, 0.16 mL of 2.24 M solution in THF) was added via cannula to a stirred solution of HSC_6H_2(2,4,6-*t*-Bu_3) (0.10 g, 0.36 mmol) in 50 mL of THF at −78 °C. The clear solution was allowed to warm to 25 °C, stirred for an additional 2 h, and then used in the following preparation.

Preparation of Bi(SC_6H_2(2,4,6-*t*-Bu_3))$_3$ (1). Three equivalents of $LiSC_6H_2$(2,4,6-*t*-Bu_3) (8.4 mL, 11.9 M solution in THF) were added to a stirred solution of $BiCl_3$ (0.030 g, 0.10 mmol) in 50 mL of THF at −78 °C. The dark orange reaction mixture was allowed to warm slowly to 25 °C and then stirred at this temperature for 12 h. The solvent and volatiles were removed under reduced pressure and the resulting orange residue was extracted with hexane (40 mL). The white precipitate (tested positive for LiCl) was filtered off and the filtrate was concentrated in vacuo to a volume of 20 mL. Cooling of this solution to −20 °C (24 h) produced 0.08 g (77% yield) of orange crystalline **1**. Mp: 105–110 °C dec. ^1H NMR (C_6D_6, 25 °C, 300.15 MHz): δ 7.54 (6 H, s, *m*-H ring), 1.63 (54 H, s, *o*-*t*-Bu), 1.29 (27 H, s, *p*-*t*-Bu). Anal. Calcd for $C_{54}H_{87}BiS_3$: C, 62.26; H, 8.42. Found: C, 62.02; H, 8.21. MS (EI and CI): *m/e* 554, $[(2,4,6\text{-}t\text{-}Bu_3C_6H_2)_2S_2]^+$.

Table I. Crystallographic Data for $Bi(SAr')_3$ (**1**; Ar′ = 2,4,6-*t*-$Bu_3C_6H_2$)

color	orange	γ, deg	91.981(9)
formula	$C_{54}H_{87}BiS_3$	V, Å^3	5775(5)
fw	1941.6	d(calc), g cm^{-3}	1.202
cryst dimens, mm	0.31 × 0.08 × 0.98	Z	4
cryst syst	triclinic	radiation	Mo Kα
space group	$P\bar{1}$	tot. no. of reflcns	10 603
a, Å	17.214(2)	no. of tot. reflcns obsd	5033
b, Å	17.742(2)	σ test	$F_o > 6\sigma(F_o)$
c, Å	20.984(2)	R^a	0.0582
α, deg	99.335(8)	$R_w{}^b$	0.0604
β, deg	114.264(9)		

[a] $R = \sum||F_o| - |F_c|\sum|F_o|$. [b] $R_w = [\bar{\omega}(|F_o| - |F_c|)^2/\sum\bar{\omega}F_o^2]^{1/2}$.

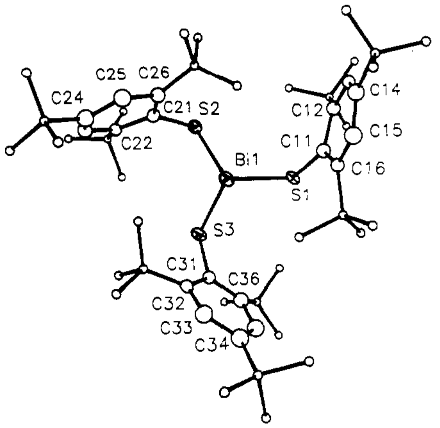

Figure 1. ORTEP view of molecule A of compound **1**.

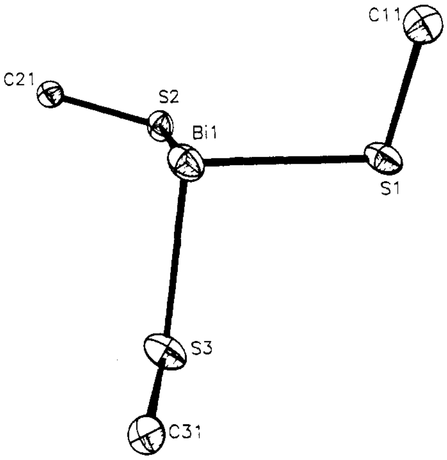

Figure 2. View emphasizing the geometry around Bi for molecule A of compound **1**.

X-ray Crystal Structure of 1. Crystals of **1** suitable for X-ray diffraction experiments were grown from hexane solution at −20 °C over a 2-week period. Details of the crystal data and a summary of intensity data collection parameters for **1** are presented in Table I. The crystals were mounted in thin-walled glass capillaries and sealed under argon. The unit cell parameters were obtained by centering 25 reflections having 2θ values >35°. The triclinic space group $P\bar{1}$ was established on the basis of systematic absences. The data were collected on an Enraf-Nonius CAD-4 diffractometer (25 °C) in the $+h,\pm k,\pm l$ quadrant with 2θ values between 2 and 40° in a manner described previously.[10] The intensity standards indicated a <1% decrease in intensity over the course of data collection and hence, no correction was applied. A ψ scan of three reflections having χ values between 80 and 90° showed a minimum transmission of 41% and a maximum transmission of 98%. An empirical absorption correction was applied. The observed structure factors of

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Table II. Atomic Positional Parameters for Bi(SAr′)$_3$ (**1**; A′ = 2,4,6-*t*-Bu$_3$C$_6$H$_2$)

atom	*x*/*a*	*y*/*b*	*z*/*c*	*U*(eqv)	atom	*x*/*a*	*y*/*b*	*z*/*c*	*U*(eqv)
Bi(1)	0.22234(7)	0.31226(6)	0.27710(6)	0.042(5)	C(241)	0.419(2)	0.037(2)	0.194(2)	0.060(8)
Bi(2)	0.09931(7)	0.19158(6)	0.73807(6)	0.049(3)	C(242)	0.510(2)	0.040(2)	0.206(2)	0.087(4)
S(1)	0.1871(4)	0.4447(4)	0.3199(4)	0.040(6)	C(243)	0.366(2)	0.049(2)	0.119(2)	0.081(2)
S(2)	0.3554(4)	0.3025(4)	0.3867(4)	0.040(3)	C(244)	0.384(3)	−0.039(2)	0.204(2)	0.152(61)
S(3)	0.1190(5)	0.2318(4)	0.3061(4)	0.047(1)	C(261)	0.464(2)	0.323(1)	0.292(1)	0.040(7)
S(4)	0.1062(5)	0.1385(4)	0.8468(4)	0.056(12)	C(262)	0.398(2)	0.377(2)	0.262(2)	0.071(23)
S(5)	0.2488(4)	0.2614(4)	0.8084(4)	0.045(5)	C(263)	0.529(2)	0.357(2)	0.368(1)	0.057(6)
S(6)	0.0004(4)	0.2956(4)	0.7383(4)	0.042(3)	C(264)	0.515(2)	0.315(2)	0.245(2)	0.100(15)
C(11)	0.229(2)	0.502(1)	0.276(1)	0.040(7)	C(321)	0.090(2)	0.102(2)	0.169(2)	0.056(8)
C(12)	0.311(2)	0.553(1)	0.317(1)	0.039(7)	C(322)	0.084(2)	0.053(2)	0.222(2)	0.080(25)
C(13)	0.352(1)	0.579(1)	0.281(1)	0.030(6)	C(323)	0.187(2)	0.124(2)	0.191(1)	0.059(5)
C(14)	0.322(2)	0.560(2)	0.209(1)	0.048(7)	C(324)	0.065(2)	0.042(2)	0.097(2)	0.087(11)
C(15)	0.242(2)	0.525(2)	0.171(2)	0.055(8)	C(340)	−0.145(5)	0.216(5)	−0.005(5)	0.076(28)
C(16)	0.193(2)	0.497(1)	0.203(1)	0.033(6)	C(342)	−0.240(4)	0.205(4)	−0.004(3)	0.058(17)
C(21)	0.396(2)	0.226(1)	0.343(1)	0.036(7)	C(343)	−0.143(5)	0.304(5)	−0.007(4)	0.105(27)
C(22)	0.390(1)	0.149(1)	0.356(1)	0.030(6)	C(344)	−0.142(5)	0.152(5)	−0.054(4)	0.109(27)
C(23)	0.398(2)	0.094(2)	0.311(1)	0.042(7)	C(341)	−0.158(4)	0.229(3)	0.019(3)	0.030(17)
C(24)	0.413(2)	0.103(2)	0.252(2)	0.052(8)	C(345)	−0.115(5)	0.254(5)	−0.032(4)	0.088(23)
C(25)	0.431(2)	0.176(2)	0.246(1)	0.054(8)	C(346)	−0.224(5)	0.289(4)	0.019(4)	0.100(25)
C(26)	0.426(2)	0.241(1)	0.293(1)	0.040(7)	C(347)	−0.211(5)	0.147(5)	−0.024(4)	0.115(28)
C(31)	0.028(2)	0.214(1)	0.221(1)	0.033(6)	C(361)	−0.070(2)	0.293(2)	0.276(2)	0.073(38)
C(32)	0.032(1)	0.165(1)	0.161(1)	0.031(6)	C(362)	−0.060(2)	0.234(2)	0.321(2)	0.104(26)
C(33)	−0.022(2)	0.173(2)	0.100(2)	0.055(8)	C(363)	−0.012(2)	0.368(2)	0.320(2)	0.142(74)
C(34)	−0.091(2)	0.218(2)	0.088(2)	0.059(8)	C(364)	−0.159(2)	0.312(2)	0.253(2)	0.120(48)
C(35)	−0.096(2)	0.258(2)	0.148(2)	0.057(8)	C(421)	0.228(2)	0.005(2)	0.849(2)	0.052(12)
C(36)	−0.041(2)	0.256(2)	0.215(1)	0.047(7)	C(422)	0.181(2)	−0.000(2)	0.770(2)	0.080(41)
C(41)	0.216(2)	0.147(1)	0.902(1)	0.045(7)	C(423)	0.298(2)	−0.046(2)	0.858(2)	0.118(66)
C(42)	0.264(2)	0.090(2)	0.887(2)	0.055(8)	C(424)	0.175(2)	−0.027(2)	0.885(2)	0.097(47)
C(43)	0.354(2)	0.110(2)	0.921(2)	0.063(8)	C(441)	0.490(2)	0.197(2)	0.997(2)	0.057(48)
C(44)	0.397(2)	0.177(2)	0.967(1)	0.051(8)	C(442)	0.531(2)	0.146(3)	0.960(2)	0.207(164)
C(45)	0.344(2)	0.224(2)	0.987(2)	0.059(8)	C(443)	0.509(3)	0.269(3)	0.983(2)	0.164(21)
C(46)	0.253(2)	0.211(2)	0.956(1)	0.046(7)	C(444)	0.530(2)	0.198(3)	1.070(2)	0.165(119)
C(51)	0.275(2)	0.310(1)	0.750(1)	0.041(7)	C(461)	0.205(3)	0.260(2)	0.992(2)	0.091(32)
C(52)	0.294(2)	0.272(1)	0.696(1)	0.042(7)	C(462)	0.146(2)	0.214(2)	1.009(2)	0.088(27)
C(53)	0.290(2)	0.308(2)	0.642(1)	0.052(8)	C(463)	0.154(2)	0.309(2)	0.945(2)	0.094(7)
C(54)	0.279(2)	0.382(2)	0.643(1)	0.047(7)	C(464)	0.267(2)	0.315(2)	1.061(2)	0.136(45)
C(55)	0.280(1)	0.423(1)	0.702(1)	0.036(6)	C(521)	0.323(2)	0.187(2)	0.692(2)	0.054(8)
C(56)	0.276(2)	0.389(1)	0.758(1)	0.035(6)	C(522)	0.382(3)	0.171(2)	0.764(2)	0.139(49)
C(61)	−0.085(2)	0.259(1)	0.657(1)	0.038(7)	C(523)	0.373(3)	0.168(2)	0.644(2)	0.157(26)
C(62)	−0.082(2)	0.275(1)	0.594(1)	0.045(7)	C(254)	0.250(3)	0.125(2)	0.663(3)	0.186(106)
C(63)	−0.148(2)	0.235(1)	0.525(1)	0.045(7)	C(541)	0.278(2)	0.419(2)	0.582(2)	0.084(12)
C(64)	−0.208(2)	0.183(2)	0.526(2)	0.053(8)	C(542)	0.364(2)	0.421(2)	0.581(2)	0.138(20)
C(65)	−0.216(2)	0.175(2)	0.586(2)	0.063(9)	C(543)	0.256(3)	0.503(2)	0.588(2)	0.159(39)
C(66)	−0.155(2)	0.214(2)	0.651(2)	0.052(8)	C(544)	0.219(3)	0.370(2)	0.509(2)	0.135(13)
C(121)	0.343(2)	0.579(1)	0.399(1)	0.039(7)	C(561)	0.283(2)	0.442(2)	0.826(2)	0.082(46)
C(122)	0.388(2)	0.515(1)	0.436(1)	0.050(13)	C(562)	0.353(2)	0.423(2)	0.896(1)	0.062(17)
C(123)	0.406(2)	0.654(2)	0.423(2)	0.078(8)	C(563)	0.195(3)	0.446(2)	0.826(2)	0.091(38)
C(124)	0.271(2)	0.607(2)	0.421(1)	0.056(21)	C(564)	0.308(3)	0.522(2)	0.821(2)	0.147(29)
C(141)	0.380(2)	0.593(2)	0.175(2)	0.068(9)	C(621)	−0.019(2)	0.337(2)	0.588(1)	0.042(7)
C(142)	0.377(2)	0.682(2)	0.182(2)	0.146(40)	C(622)	−0.019(2)	0.417(2)	0.635(2)	0.080(20)
C(143)	0.348(3)	0.553(3)	0.098(2)	0.205(25)	C(623)	−0.047(2)	0.355(2)	0.512(2)	0.088(23)
C(144)	0.470(3)	0.573(2)	0.204(2)	0.147(17)	C(624)	0.069(2)	0.310(2)	0.608(2)	0.074(6)
C(161)	0.094(2)	0.470(2)	0.157(2)	0.053(12)	C(641)	−0.276(2)	0.143(2)	0.452(2)	0.055(9)
C(162)	0.042(2)	0.506(2)	0.192(2)	0.072(20)	C(642)	−0.318(3)	0.068(3)	0.456(2)	0.171(21)
C(163)	0.069(2)	0.491(2)	0.084(2)	0.092(33)	C(643)	−0.346(3)	0.189(3)	0.423(3)	0.225(29)
C(164)	0.075(2)	0.381(2)	0.140(2)	0.080(9)	C(644)	−0.244(3)	0.124(3)	0.399(2)	0.180(23)
C(221)	0.382(2)	0.129(2)	0.422(1)	0.040(7)	C(661)	−0.174(2)	0.206(2)	0.721(2)	0.061(29)
C(222)	0.442(2)	0.181(2)	0.490(1)	0.056(16)	C(662)	−0.108(2)	0.157(2)	0.769(2)	0.075(17)
C(223)	0.290(2)	0.128(2)	0.413(2)	0.080(14)	C(663)	−0.264(2)	0.158(2)	0.694(2)	0.126(16)
C(224)	0.406(2)	0.053(2)	0.434(2)	0.082(20)	C(664)	−0.175(2)	0.282(2)	0.760(2)	0.069(12)

equivalent reflections were averaged. Data with intensities less than $3.0\sigma(I)$ and $(\sin\theta)/\lambda$ less than 0.10 were excluded, and a weighting scheme $([\sigma F]^2 + 0.000625F^2]^{-1})$ was used in the final stages of the refinement, unless stated otherwise. The data were corrected for Lorentz and polarization effects. The bismuth atoms were found using a Patterson search, and subsequent location of the other non-hydrogen atoms was achieved using difference Fourier maps. The hydrogen atoms were not located; however, their contributions were included in the final stages of the refinement. The final R and R_w values were 0.0582 and 0.0604, respectively. All calculations were performed on a MICROVAX 3100 Computer using the SHELX programs.[11]

Results and Discussion

In order to minimize the chance of oligomerization or decomposition of the Bi(III) thiolate, it was desirable to employ a bulky ligand array. The ligand of choice was the arenethiolate [SAr′]$^-$ (Ar′ = 2,4,6-*t*-Bu$_3$C$_6$H$_2$) since it has proved to be effective for the stabilization of e.g. low-coordinate Zn(II), Cd(II), and Hg(II) derivatives.[12] The requisite thiol, Ar′SH, was prepared according to the method of Rundel[9] and converted to the

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Table III. Selected Bond Distances (Å) and Angles (deg) for $\mathrm{Bi(SAr')_3}$ (**1**; Ar′ = 2,4,6-*t*-$\mathrm{Bu_3C_6H_2}$)[a]

Molecule A	
Bi(1)–S(1) 2.558 (7)	Bi(1)–S(3) 2.556 (7)
Bi(1)–S(2) 2.562 (7)	
S(1)–Bi(1)–S(2) 99.1 (2)	S(2)–Bi(1)–S(3) 96.2 (2)
S(1)–Bi(1)–S(3) 97.6 (2)	
Molecule B	
Bi(2)–S(4) 2.569 (8)	Bi(2)–S(6) 2.555 (7)
Bi(2)–S(5) 2.554 (7)	
S(4)–Bi(2)–S(5) 90.3 (2)	S(5)–Bi(2)–S(6) 104.5 (2)
S(4)–Bi(2)–S(6) 100.0 (2)	

[a] Estimated standard deviations are given in parentheses.

corresponding lithio derivative LiSAr′ by treatment with *t*-BuLi in THF solution. In turn, the metathetical reaction of LiSAr′ with $\mathrm{BiCl_3}$ in the same solvent afforded an 77% yield of orange, crystalline $\mathrm{Bi(SAr')_3}$ (**1**).

$$\mathrm{BiCl_3 + 3LiSAr'} \xrightarrow[-3\,\mathrm{LiCl}]{} \underset{\mathbf{1}}{[\mathrm{Bi(SAr')_3}]}$$

The elemental analysis data for **1** are consistent with the composition $[\mathrm{Bi(SAr')_3}]$. Unfortunately, however, it proved impossible to identify **1** by mass spectroscopy. The highest *m*/*e* peak in either EI or CI experiments corresponds to the molecular ion for the known compound Ar′SSAr′.[9] Inferentially, **1** undergoes facile reductive elimination of disulfide in the vapor phase. Solution phase ^{1}H and NMR data (Experimental Section) are indicative of the presence of only one type of $\mathrm{SC_6H_2}$(2,4,6-*t*-$\mathrm{Bu_3}$) moiety.

Since the structure of **1** could not be established definitively on the basis of spectroscopic data, it was expedient to conduct an X-ray diffraction study. The solid state of **1** consists of discrete molecules and there are no unusually short intermolecular contacts. There are two very similar but independent molecules, A and B, in the asymmetric unit. Molecule A is illustrated in Figures 1 and 2. Atomic positional parameters and selected interatomic distances and angles for both molecules are assembled in Table II and III, respectively. Each molecule features a pyramidal $\mathrm{BiS_3}$ core, and the Bi–S bond distances for both molecules are equal within experimental error. The main distinction between the two molecules lies in the relative orientations of the aryl rings and the fact that the average S–Bi–S bond angle in molecule A is slightly smaller (~0.7°) than that in molecule B. As in the case of, e.g., $\mathrm{Bi(OC_6H_3(2,6\text{-}Me_2))_3}$,[13] the packing of bulky aryl rings in the crystal lattice plays a major role in determining the observed conformation. However, despite the presence of the bulky arenethiolate ligands, steric strain is not evident in the metrical parameters of the $\mathrm{BiS_3}$ core. Thus, the sum of angles at Bi in **1** (292.9(2)° in molecule A and 294.8(2)° in molecule B) are less than those in $[\mathrm{BiMes_3}]$ (308.1°)[14] or $\mathrm{Bi(C_6H_2(2,4,6(CF_3)_3))_3}$ (317.9°)[15] and comparable to those in the homoleptic bismuth amides $[\mathrm{Bi(NPh_2)_3}]$ (296 and 297° for two independent molecules)[16] and $[\mathrm{Bi(NMe_2)_3}]$ (292.8°).[17] The average Bi–S bond distances in molecules A and B of **1** are identical (2.559(8) Å) and very similar to those in the oxadithiabismocane $[\mathrm{C_6H_5Bi(SCH_2CH_2OCH_2CH_2S)}]$ (average 2.581(3) Å)[18] and for the in-plane Bi–S distance in $[\mathrm{Bi(2\text{-}SC_5H_3N\text{-}3\text{-}SiMe_3)}]$ (2.626(8) Å).[7]

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Supplementary Material Available: Tables of interatomic distances and angles, fractional coordinates for hydrogen atoms, anisotropic thermal parameters and a stereoscopic view of the unit cell (15 pages). Ordering information is given on any current masthead page.

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