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SOME REACTIONS OF 8,11,11-TRIMETHYL-11-SILABENZO[*b*]NAPHTHO-[2,3-*d*]THIOPHEN-6-ONE AT THE CARBONYL GROUP

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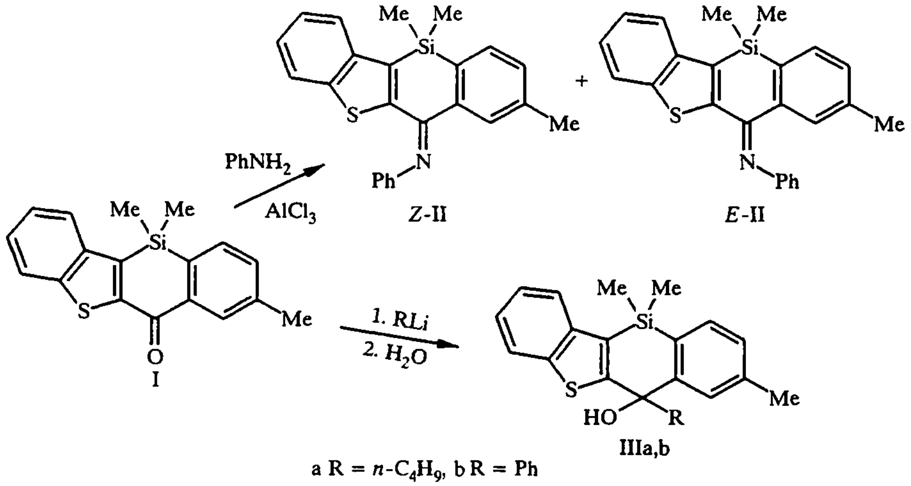
Reactions of C- and N-nucleophiles at the carbonyl group of 8,11,11-trimethyl-11-silabenzo-[b]naphtho[2,3-d]thiophen-6-one have been studied. PMR Spectroscopy showed that condensation with aniline gives a mixture of the Z- and E- isomers of the corresponding azomethine. Reaction with n-butyl and phenyllithium gave tertiary alcohols, the molecular structure of which was proved using X-ray crystallography.

Catalytic dehydrocycloisomerization of triorganosilyl substituted benzo[*b*]thiophenes gave the first representatives of a new class of heterocyclic compounds, *viz.* 11-silabenzo[*b*]naphtho[2,3-*d*]thiophenes [1, 2]. Their oxidation with $KMnO_4$ leads to 8,11,11-trimethyl-11-silabenzo[*b*]naphtho[2,3-*d*]thiophen-6-one (I) which is a promising compound for the construction of novel derivatives of the indicated system, particularly *via* reaction with C- and N-nucleophiles.

In this report we describe the reactions of compound I with aniline and with butyl- and phenyllithium. Bearing in mind that molecule I has a planar structure [3], it might be expected that the effect of steric factors can be ignored in the first approximation for these examples. Condensation with aniline was carried out in the presence



a R = n-C_4H_9, b R = Ph

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of BF_3 etherate, $ZnCl_2$, and $AlCl_3$. In this way, 8,11,11-trimethyl-6-phenylimino-11-silabenzo[*b*]naphtho[2,3-*d*]-thiophene (II) was prepared, the maximum yield being 50% and this was achieved when anhydrous $AlCl_3$ was used as catalyst. For the case of anhydrous $ZnCl_2$, the yield of compound II did not exceed 20% and, with the use of BF_3 etherate, only traces of the target product were identified using chromatography.

The structure of azomethine II was confirmed by PMR data. Hence, in its spectrum there are multiplet signals for the aromatic protons in the region 6.89-8.40 ppm and a six proton singlet at 0.65 ppm for the $Si(CH_3)_2$ group. In the case of the 8-CH_3 group protons two broad signals are seen at 2.49-2.02 ppm, showing that compound II exists as a mixture of two isomers. The overall integrated intensity of these signals amounts to three protons and the ratio of their intensities is 63:37. Taking into account the magnetic anisotropic effect of the phenyl radical in the phenylimine fragment, the high field signal can be assigned to the *E*-isomer and the low field signal to the *Z*-isomer [4]. Increasing the temperature produces a typical dynamic NMR picture for these signals. At a temperature of 320 K they merge. Using the method proposed in [6] an estimate of the isomerization barrier $Z \rightleftarrows E$ at $T_c = 320$ K for exchange between two forms with different populations was carried out:

$$G^{\#}_{320}(E) = 15.3 \text{ kcal/mol}; \quad G^{\#}_{320}(Z) = 15.0 \text{ kcal/mol};$$
$$G^{\#}_{320}(E) - G^{\#}_{320}(Z) = 0.3 \text{ kcal/mol}.$$

The predominance of the *Z*-isomer in the azomethine II can be explained by the effects of electronic and steric factors [6].

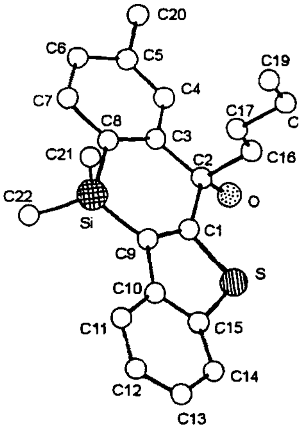

Fig. 1. Molecular structure of compound IIIa.

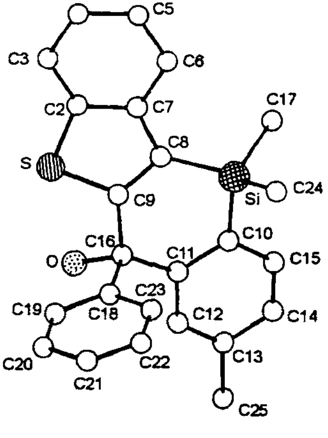

Fig. 2. Molecular structure of compound IIIb.

TABLE 1. Coordinates of Non-Hydrogen Atoms (×10^4) in Molecules of Compounds IIIa and IIIb

Atom	x	y	z
IIIa			
S	1505.7(5)	395(9)	4962.8(3)
Si	-728.5(5)	1492(1)	3686.2(4)
O	639(1)	-1986(2)	4579.5(9)
$C_{(1)}$	625(2)	283(3)	4399(1)
$C_{(2)}$	454(2)	-1034(3)	4331(1)
$C_{(3)}$	-424(2)	-1237(3)	3766(1)
$C_{(4)}$	-650(2)	-2507(3)	3625(1)
$C_{(5)}$	-1412(2)	-2834(4)	3283(1)
$C_{(6)}$	-1967(2)	-1862(4)	3096(2)
$C_{(7)}$	-1765(2)	612(4)	3226(1)
$C_{(8)}$	-977(2)	-239(3)	3561(1)
$C_{(9)}$	245(2)	1433(3)	4252(1)
$C_{(10)}$	693(2)	2474(3)	4592(1)
$C_{(11)}$	528(2)	3796(4)	4559(1)
$C_{(12)}$	1062(2)	4636(4)	4909(2)
$C_{(13)}$	1758(2)	4192(4)	5293(2)
$C_{(14)}$	1932(2)	2903(4)	5346(1)
$C_{(15)}$	1398(2)	2052(3)	4994(1)
$C_{(16)}$	1082(2)	-1334(3)	3771(1)
$C_{(17)}$	1002(2)	-515(4)	3240(1)
$C_{(18)}$	1632(2)	-807(4)	2899(2)
$C_{(19)}$	1524(3)	70(6)	2383(2)
$C_{(20)}$	-1622(3)	-4232(4)	3131(2)
$C_{(21)}$	-567(2)	2282(4)	3024(2)
$C_{(22)}$	-1571(2)	2354(4)	3921 (2)
IIIb			
S	4388(8)	4034(7)	8640(6)
Si	6435.4(3)	7911.2(8)	7634.3(7)
O	1823(2)	6301(2)	8947(2)
$C_{(2)}$	6338(3)	3644(3)	8924(2)
$C_{(3)}$	7126(4)	2344(3)	9410(3)
$C_{(4)}$	8698(4)	2242(3)	9530(3)
$C_{(5)}$	9448(4)	3410(4)	9171(3)
$C_{(6)}$	8662(3)	4696(3)	8723(2)
$C_{(7)}$	7064(3)	4847(3)	8585(2)
$C_{(8)}$	6032(3)	6082(3)	8107(2)
$C_{(9)}$	4575(3)	5791(2)	8066(2)
$C_{(10)}$	4457(3)	8910(3)	7453(2)
$C_{(11)}$	3144(3)	8287(3)	7399(2)
$C_{(12)}$	1739(3)	9139(3)	7170(3)
$C_{(13)}$	1559(3)	10568(3)	7011(3)
$C_{(14)}$	2841(4)	11180(3)	7013(3)
$C_{(15)}$	4245(4)	10363(3)	7321(3)
$C_{(16)}$	3154(3)	6712(3)	7543(2)
$C_{(17)}$	7404(4)	8462(3)	8723(3)
$C_{(18)}$	2984(3)	6358(3)	6402(2)
$C_{(19)}$	2074(3)	5372(3)	6319(2)
$C_{(20)}$	1988(3)	5018(3)	5302(3)
$C_{(21)}$	2800(4)	5617(3)	4351(2)
$C_{(22)}$	3712(4)	6590(3)	4426(3)
$C_{(23)}$	3788(4)	6964(3)	5439(2)
$C_{(24)}$	7626(4)	8180(4)	6280(3)
$C_{(25)}$	24(4)	11456(4)	6712(3)

TABLE 2. Valence Angles (deg.) in the Molecule of Compound IIIa

Angle	ω	Angle	ω
$C_{(1)}$–S–$C_{(15)}$	91.4(1)	$C_{(4)}$–$C_{(5)}$–$C_{(6)}$	117.8(3)
$C_{(8)}$–Si–$C_{(9)}$	102.1(1)	$C_{(4)}$–$C_{(S)}$–$C_{(20)}$	120.4(3)
$C_{(8)}$–Si–$C_{(21)}$	111.0(2)	$C_{(6)}$–$C_{(5)}$–$C_{(20)}$	121.8(3)
$C_{(8)}$–Si–$C_{(22)}$	111.1(2)	$C_{(5)}$–$C_{(6)}$–$C_{(7)}$	121.1(3)
$C_{(9)}$–Si–$C_{(21)}$	111.3(2)	$C_{(6)}$–$C_{(7)}$–$C_{(8)}$	122.4(3)
$C_{(9)}$–Si–$C_{(22)}$	112.6(2)	$C_{(3)}$–$C_{(8)}$–$C_{(7)}$	116.1(3)
$C_{(21)}$–Si–$C_{(22)}$	108.7(2)	$C_{(1)}$–$C_{(9)}$–$C_{(10)}$	111.3(2)
$C_{(2)}$–$C_{(1)}$–$C_{(9)}$	131.0(2)	$C_{(9)}$–$C_{(10)}$–$C_{(11)}$	129.2(3)
O–$C_{(2)}$–$C_{(1)}$	108.5(2)	$C_{(9)}$–$C_{(10)}$–$C_{(15)}$	113.0(3)
O–$C_{(2)}$–$C_{(3)}$	109.9(2)	$C_{(11)}$–$C_{(10)}$–$C_{(15)}$	117.8(3)
O–$C_{(2)}$–$C_{(16)}$	103.3(2)	$C_{(10)}$–$C_{(11)}$–$C_{(12)}$	119.7(3)
$C_{(1)}$–$C_{(2)}$–$C_{(3)}$	115.3(2)	$C_{(11)}$–$C_{(12)}$–$C_{(13)}$	121.0(4)
$C_{(1)}$–$C_{(2)}$–$C_{(16)}$	109.8(3)	$C_{(12)}$–$C_{(13)}$–$C_{(14)}$	121.0(3)
$C_{(3)}$–$C_{(2)}$–$C_{(16)}$	109.4(2)	$C_{(13)}$–$C_{(14)}$–$C_{(15)}$	118.4(3)
$C_{(2)}$–$C_{(3)}$–$C_{(4)}$	115.9(3)	$C_{(10)}$–$C_{(15)}$–$C_{(14)}$	122.1(3)
$C_{(2)}$–$C_{(3)}$–$C_{(8)}$	124.0(3)	$C_{(2)}$–$C_{(16)}$–$C_{(17)}$	115.2(3)
$C_{(4)}$–$C_{(3)}$–$C_{(8)}$	120.0(2)	$C_{(16)}$–$C_{(17)}$–$C_{(18)}$	114.4(3)
$C_{(3)}$–$C_{(4)}$–$C_{(5)}$	122.4(3)	$C_{(17)}$–$C_{(18)}$–$C_{(19)}$	111.3(4)

TABLE 3. Valence Angles (deg.) in the Molecule of Compound IIIb

Angle	ω	Angle	ω
$C_{(2)}$–S–$C_{(9)}$	91.3(1)	$C_{(11)}$–$C_{(12)}$–$C_{(13)}$	123.2(3)
$C_{(8)}$–Si–$C_{(10)}$	102.1(1)	$C_{(12)}$–$C_{(13)}$–$C_{(14)}$	117.8(3)
$C_{(8)}$–Si–$C_{(17)}$	111.9(1)	$C_{(12)}$–$C_{(13)}$–$C_{(25)}$	121.3(3)
$C_{(8)}$–Si–$C_{(24)}$	111.9(1)	$C_{(14)}$–$C_{(13)}$–$C_{(25)}$	120.9(3)
$C_{(10)}$–Si–$C_{(17)}$	110.2(1)	$C_{(13)}$–$C_{(14)}$–$C_{(15)}$	120.0(3)
$C_{(10)}$–Si–$C_{(24)}$	111.2(1)	$C_{(10)}$–$C_{(15)}$–$C_{(14)}$	122.8(3)
$C_{(17)}$–Si–$C_{(24)}$	109.4(2)	O–$C_{(16)}$–$C_{(9)}$	107.6(2)
$C_{(3)}$–$C_{(2)}$–$C_{(7)}$	122.5(3)	O–$C_{(16)}$–$C_{(11)}$	109.2(2)
$C_{(2)}$–$C_{(3)}$–$C_{(4)}$	118.0(3)	O–$C_{(16)}$–$C_{(18)}$	105.8(2)
$C_{(3)}$–$C_{(4)}$–$C_{(5)}$	120.5(3)	$C_{(9)}$–$C_{(16)}$–$C_{(11)}$	115.2(2)
$C_{(4)}$–$C_{(5)}$–$C_{(6)}$	121.4(3)	$C_{(9)}$–$C_{(16)}$–$C_{(18)}$	108.0(2)
$C_{(5)}$–$C_{(6)}$–$C_{(7)}$	119.9(3)	$C_{(11)}$–$C_{(16)}$–$C_{(18)}$	110.5(2)
$C_{(2)}$–$C_{(7)}$–$C_{(6)}$	117.6(2)	$C_{(16)}$–$C_{(18)}$–$C_{(19)}$	121.0(2)
$C_{(2)}$–$C_{(7)}$–$C_{(8)}$	113.3(2)	$C_{(16)}$–$C_{(18)}$–$C_{(23)}$	121.0(3)
$C_{(6)}$–$C_{(7)}$–$C_{(8)}$	129.0(3)	$C_{(19)}$–$C_{(18)}$–$C_{(23)}$	118.0(3)
$C_{(7)}$–$C_{(8)}$–$C_{(9)}$	111.6(2)	$C_{(18)}$–$C_{(19)}$–$C_{(20)}$	120.4(3)
$C_{(8)}$–$C_{(9)}$–$C_{(16)}$	130.6(2)	$C_{(19)}$–$C_{(20)}$–$C_{(21)}$	121.3(3)
$C_{(11)}$–$C_{(10)}$–$C_{(15)}$	117.1(2)	$C_{(20)}$–$C_{(21)}$–$C_{(22)}$	118.7(3)
$C_{(10)}$–$C_{(11)}$–$C_{(12)}$	119.0(2)	$C_{(21)}$–$C_{(22)}$–$C_{(23)}$	120.4(3)
$C_{(10)}$–$C_{(11)}$–$C_{(16)}$	124.3(2)	$C_{(18)}$–$C_{(23)}$–$C_{(22)}$	121.2(3)
$C_{12)}$–$C_{(11)}$–$C_{(16)}$	116.8(2)		

Nucleophilic addition of BuLi and PhLi to the carbonyl group of compound I occurs in quite high yields (62 and 64% respectively). Since compounds IIIa,b can be obtained as well defined crystals and bearing in mind the paucity of known 11-silabenzo[*b*]naphtho[2,3-*d*]thiophene derivatives, it was our aim to study their structures by X-ray analysis. The results obtained are given in Figures 1 and 2 and in Tables 1-4. The geometric parameters observed were close to standard values [7].

In the mass spectra of compounds IIIa,b, the dissociative ionization was characterized by very low intensity elimination of a CH_3 group from the molecular ions, the maximal intensities (100%) being for peaks with *m/z* 309 $[M-C_4H_9]^+$ for compound IIIa and $[M-C_6H_5]^+$ for IIIb.

TABLE 4. Bond Lengths (Å) in Molecules of Compounds IIIa and IIIb

Bond	*d*	Bond	*d*
IIIa		IIIb	
S–C$_{(1)}$	1.750(2)	S–C$_{(2)}$	1.741(3)
S–C$_{(15)}$	1.735(2)	S–C$_{(9)}$	1.749(2)
Si–C$_{(8)}$	1.855(4)	Si–C$_{(8)}$	1.855(3)
Si–C$_{(9)}$	1.854(3)	Si–C$_{(10)}$	1.861(3)
Si–C$_{(21)}$	1.870(4)	Si–C$_{(17)}$	1.860(4)
Si–C$_{(22)}$	1.864(4)	Si–C$_{(24)}$	1.858(3)
O–C$_{(2)}$	1.443(4)	O–C$_{(16)}$	1.440(3)
C$_{(1)}$–C$_{(2)}$	1.510(4)	C$_{(2)}$–C$_{(3)}$	1.388(4)
C$_{(1)}$–C$_{(9)}$	1.359(4)	C$_{(2)}$–C$_{(7)}$	1.402(4)
C$_{(2)}$–C$_{(3)}$	1.531(4)	C$_{(3)}$–C$_{(4)}$	1.385(5)
C$_{(2)}$–C$_{(16)}$	1.544(5)	C$_{(4)}$–C$_{(5)}$	1.386(5)
C$_{(3)}$–C$_{(4)}$	1.392(5)	C$_{(5)}$–C$_{(6)}$	1.366(4)
C$_{(3)}$–C$_{(8)}$	1.397(4)	C$_{(6)}$–C$_{(7)}$	1.408(4)
C$_{(4)}$–C$_{(5)}$	1.380(4)	C$_{(7)}$–C$_{(8)}$	1.439(3)
C$_{(5)}$–C$_{(6)}$	1.372(5)	C$_{(8)}$–C$_{(9)}$	1.363(4)
C$_{(5)}$–C$_{(20)}$	1.519(6)	C$_{(9)}$–C$_{(16)}$	1.518(3)
C$_{(6)}$–C$_{(7)}$	1.360(6)	C$_{(10)}$–C$_{(11)}$	1.406(4)
C$_{(7)}$–C$_{(8)}$	1.421(4)	C$_{(10)}$–C$_{(15)}$	1.396(4)
C$_{(9)}$–C$_{(10)}$	1.452(4)	C$_{(11)}$–C$_{(12)}$	1.390(3)
C$_{(10)}$–C$_{(11)}$	1.400(5)	C$_{(11)}$–C$_{(16)}$	1.529(4)
C$_{(10)}$–C$_{(15)}$	1.403(4)	C$_{(12)}$–C$_{(13)}$	1.370(4)
C$_{(11)}$–C$_{(12)}$	1.382(5)	C$_{(13)}$–C$_{(14)}$	1.388(5)
C$_{(12)}$–C$_{(13)}$	1.378(5)	C$_{(13)}$–C$_{(25)}$	1.511(4)
C$_{(13)}$–C$_{(14)}$	1.370(5)	C$_{(14)}$–C$_{(15)}$	1.372(4)
C$_{(14)}$–C$_{(15)}$	1.390(4)	C$_{(16)}$–C$_{(18)}$	1.533(4)
C$_{(16)}$–C$_{(17)}$	1.515(5)	C$_{(18)}$–C$_{(19)}$	1.384(4)
C$_{(17)}$–C$_{(18)}$	1.511(6)	C$_{(18)}$–C$_{(23)}$	1.376(4)
C$_{(18)}$–C$_{(19)}$	1.518(6)	C$_{(19)}$–C$_{(20)}$	1.373(5)
		C$_{(20)}$–C$_{(21)}$	1.366(4)
		C$_{(21)}$–C$_{(22)}$	1.371(5)
		C$_{(22)}$–C$_{(23)}$	1.376(5)

EXPERIMENTAL

IR Spectra were recorded on an IR-435 spectrometer for KBr tablets. PMR Spectra were obtained on a Bruker WM-400 instrument using $CDCl_3$ solvent and TMS internal standard. Mass spectra were taken on a Kratos MS-25 RFA instrument . TLC was carried out on Silufol UV-254 bound layer plates. Column chromatography was carried on a column (2 × 40 cm) filled with 100/160 μ silica gel. The synthesis of the starting material I was reported in [1, 2].

X-Ray Structural Investigation of Compounds IIIa,b. Cell parameters and reflection intensities for crystals of IIIa and IIIb were measured on an Enraf Nonius CAD-4, four circle automatic diffractometer ($\lambda MoK\alpha$, graphite monochromator, $\theta/2\theta$ scanning to θ_{max} =25°). The structure was solved by a direct method using the MULTAN program (SDP program set). Refinement of the positional and thermal parameters for non-hydrogen atoms was carried out in the anisotropic, full matrix approximation. Hydrogen atoms were localized by Fourier synthesis and refined in the isotropic approximation.

Compound IIIa. Monoclinic crystals. At 20°C a = 16.6681, b = 10.3918, c = 24.0908 Å, β = 103.82°, V = 4052.1 Å^3, Z = 4, space group $P2_1/N$. R factor 0.03934.

Compound IIIb. Crystals assigned to a triclinic system. At 20°C a = 8.7782, b = 9.9158, c = 12.1617 Å, α = 76.65, β = 84.83, γ = 80.82°, V = 1015.2 Å^3, Z = 2, space group P-1, final factor 0.03836.

8,11,11-Trimethyl-6-phenylimino-11-silabenzo[*b*]naphtho[2,3-*d*]thiophene (II). A mixture of compound I (0.31 g, 1.0 mmol), aniline (4 ml, 3.9 g, 42 mmol), and $AlCl_3$ (0.15 g, 1.1 mmol) was held for 1 h at 120-125°C. After cooling, the reaction mixture was treated with aqueous ammonia, extracted with chloroform, the extract dried over $MgSO_4$, and evaporated. The residue was chromatographed on a column (eluent hexane–ethyl acetate, 4:1) to give azomethine II (0.2 g, 50%) as yellow crystals; mp 118-119°C (ethanol); R_f 0.83 (hexane–ethyl acetate, 4:1). IR spectrum: 820, 1250 ($Si(CH_3)_2$); 1610 cm^{-1} (C=N). PMR spectrum: 1.65 (6H, s, $SiCH_3$); 2.02 and 2.49 (3H, two br. s, 63:37, CCH_3); 6.89-8.40 ppm (12H, m, H_{arom}). Mass spectrum (I_{rel}, %): 383 M^+ (100), 368 $[M-CH_3]^+$ (28). Found, %: C 75.22; H 5.81; N 3.61; S 8.99. $C_{24}H_{21}NSSi$. Calculated, %: C 75.19; H 5.48; N 3.65; S 8.95.

Compound II (0.08 g, 20%) was prepared similarly from compound I (0.16 g, 0.05 mmol) and freshly distilled aniline (2 g, 21 mmol) in the presence of sublimed $ZnCl_2$ (0.08 g, 0.06 mmol).

6-Butyl-8,11,11-trimethyl-11-silabenzo[*b*]naphtho[2,3-*d*]thiophen-6-ol (IIIa). Butyllithium (0.2 g, 3 mmol) in hexane was added to a solution of compound I (1.0 g, 3 mmol) in absolute ether (50 ml) under an argon stream at -20°C. The reaction product was stirred for 20 min at -20°C, then for 2 h at 20°C, and hydrolyzed using a saturated solution of NH_4Cl (100 ml). The organic layer was dried with $MgSO_4$ and evaporated. The residue (1.2 g) was chromatographed on a column using hexane–ethyl acetate (5:1) as eluent to give IIIa (0.74 g, 62%) as colorless crystals; mp 125-126°C (heptane). R_f 0.49 (hexane–ethyl acetate, 5:1). IR spectrum: 820, 1250 ($Si(CH_3)_2$), 3500 cm^{-1} (OH). Mass spectrum (I_{rel}, %): 366 M^+ (38), 309 $[M-C_4H_9]^+$ (100), 351 ($[M-CH_3]^+$ (11), 348 $[M-H_2O]^+$ (28). Found, %: C 72.21; H 7.20; S 8.61. $C_{22}H_{26}OSSi$. Calculated, %: C 72.13; H 7.10; S 8.74.

8,11,11-Trimethyl-6-phenyl-11-silabenzo[*b*]naphtho[2,3-*d*]thiophen-6-ol (IIIb). A solution of compound I (0.5 g, 1.6 mmol) in ether (50 ml) was added to phenyllithium (prepared from lithium (0.3 g, 42 mmol) and bromobenzene (3.2 g, 21 mmol) in absolute ether (50 ml)) under an argon stream at 0°C. The reaction mixture was refluxed for 2 h using a reflux condenser, cooled, and hydrolyzed using a saturated, aqueous solution of NH_4Cl (50 ml). The organic layer was dried with $MgSO_4$ and the residue, after evaporation, was crystallized from heptane to give compound IIIb (0.4 g, 64%) as colorless crystals; mp 203-204°C and R_f 0.5 (hexane–ethyl acetate, 5:1). IR spectrum: 830, 1255 ($Si(CH_3)_2$), 3500 cm^{-1} (OH). Mass spectrum (I_{rel}, %): 386 M^+ (79), 371 $[M-CH_3]^+$ (10), 368 $[M-18]^+$ (42), 309 $[M-C_6H_5]^+$ (100). Found, %: 74.55; H 5.72; S 8.31. $C_{24}H_{22}OSSi$. Calculated, %: C 74.61; H 5.69; S 8.29.

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