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# Sterically crowded aryloxide compounds of silicon

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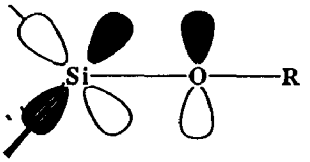
## Abstract

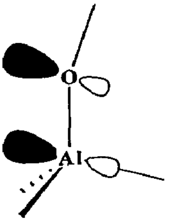
The interaction of one equivalent of NaBHT (BHT–H = butylated hydroxytoluene) with $Me_3SiCl$, $Me_2SiCl_2$ and $SiCl_4$ gives $Me_3Si(BHT)$ (**1**), $Me_2SiCl(BHT)$ (**2**) and $Cl_3Si(BHT)$ (**3**), respectively, which have been characterized by $^1H$ NMR, IR spectroscopy and elemental analysis. The molecular structures of **1** and **2** have been determined by X-ray crystallography. Compound **1**, space group $P2_1/a$, $a$ 9.129(10), $b$ 15.290(7), $c$ 13.196(8) Å, $\beta$ 95.15(6)°, $Z = 4$. Compound **2**, space group $P2_1/a$, $a$ 9.069(3), $b$ 15.189(4), $c$ 13.117(8) Å, $\beta$ 94.86(4)°, $Z = 4$. The presence of $\pi$-interaction between the oxygen lone pair and the aryl ring is proposed to account for the large Si–O–C angles observed; **1** 139.4(3)°, **2** 140.0(2)°.

## Introduction

There has been considerable discussion as to the magnitude of $\pi$-bonding between silicon and oxygen in alkoxy and aryloxy silanes $R_3SiOR'$. It has been proposed that the large Si–O–X angles observed in some alkoxy silanes (131–156°) [1,2,3] and disiloxanes (180°) [4] are due to the presence of significant $p_\pi$–$d_\pi$ bonding between oxygen and silicon [5,6,7] (I), which would be analogous to that found in transition metal alkoxide compounds.



(I)



(II)

We have recently reported that significant $\pi$-interactions are present between the oxygen lone pair and the Al–X $\sigma$ antibonding orbitals (II) in the monomeric four coordinate aluminum compounds $AlMe_2(BHT)PMe_3$ and $[AlMeCl_2(BHT)]^-$ (BHT = 2,6-di-t-butyl-4-methylphenol, BHT–H from the trivial name butylated hydroxytoluene) [8].

The structures of $AlMe_2(BHT)(PMe_3)$ and $[AlMeCl_2(BHT)]^-$ contain short Al–O bond distances (1.736(5), 1.713(4)Å), large Al–O–C angles (164.5(4), 164.0(3)°) and a highly distorted tetrahedral geometry around aluminum.

In order to examine the possibility that a similar bonding model, i.e., not involving *d* orbitals, applies to analogous silicon compounds, we have synthesized and structurally characterized the isoelectronic silicon compounds of the sterically crowded aryloxide BHT.

## Experimental

Microanalyses were performed by Oneida Research Services, Whitesboro, NY. Melting points were determined in sealed capillaries and are uncorrected. IR spectra (4000–700 $cm^{-1}$) were recorded on a Perkin Elmer 137 grating spectrometer as Nujols mulls. NMR spectra, in $C_6D_6$, were recorded on a Bruker AM-250 ($\delta$ in parts per million relative to $SiMe_4$). All manipulations were carried out under nitrogen. Solvents were dried, distilled, and degassed prior to use.

2,6-Di-t-butyl-4-methylphenol (BHT–H) (Aldrich) was sublimed prior to use. $Me_3SiCl$, $Me_2SiCl_2$, $SiCl_4$ (Aldrich) were used as received. NaBHT was prepared by the reaction of BHT–H with an excess of NaH in thf.

*$Me_3Si(BHT)$ (1).* To NaBHT (1.0 g, 4.13 mmol) in thf (50 ml) was added $Me_3SiCl$ (0.52 ml, 4.14 mmol). The solution immediately became cloudy. After stirring for 12 h the solvent was removed under vacuum and the resulting solid was sublimed in vacuo (110°C) to give X-ray quality crystals. Yield: 0.61 g, 50%. m.p. 119–121°C. IR: 1430(s), 1300(sh), 1285(sh), 1275(s), 1236(s), 1140(m) 1042(w), 970(m), 930(s), 900(sh), 854(s), 788(m), 768(br, w), 736(w) $cm^{-1}$. $^1H$ NMR ($\delta$): 7.16 (2H, s, $C_6H_2$), 2.22 (3H, s, $p$-$CH_3$), 1.46 (18H, s, $CCH_3$), 0.36 (9H, s, Si-$CH_3$). Anal. Found: C, 73.02; H, 10.89. $C_{18}H_{32}OSi$ calcd.: C, 73.90; H, 11.03%.

*$Me_2ClSi(BHT)$ (2).* In an analogous manner to **1**, NaBHT (1.09 g, 4.50 mmol) and $Me_2SiCl_2$ (0.55 ml, 4.53 mmol), gave a product which sublimed in vacuo at 105°C. Recrystallization from toluene (−20°C) yielded X-ray quality crystals. Yield: 0.67 g, 47%. m.p. 116–118°C. IR 1625(br, w), 1430(s), 1307(w), 1286(s), 1235(s), 1148(m), 1047(w), 978(s), 948(s), 905(w), 875(m), 850(sh), 832(s), 782(w), 736(m) $cm^{-1}$. $^1H$ NMR ($\delta$); 7.13 (2H, s, $C_6H_2$), 2.18 (3H, s, $p$-$CH_3$), 1.45 (18H, s, $CCH_3$), 0.55 (6H, s, Si–$CH_3$). Anal. Found: C, 64.73; H, 9.19. $C_{17}H_{29}ClOSi$ calcd.: C, 65.25; H, 9.34%.

*$Cl_3Si(BHT)$ (3).* In an analogous manner to **1**, NaBHT (1.09 g, 4.50 mmol), and $SiCl_4$ (0.52 ml, 4.54 mmol) gave a product which has recrystallized from toluene (−20°C) Yield: 0.70g, 43%, m.p. 115–117°C. IR: 1638(br, w), 1600(w), 1316(w), 1275(m), 1220(s), 1205(sh), 1130(m), 1040(w), 1002(m), 958(w), 900(w), 873(w), 817(w, br), 773(w), 732(m) $cm^{-1}$. $^1H$ NMR ($\delta$): 7.09 (2H, s, $C_6H_2$), 2.10 (3H, s, $p$-$CH_3$), 1.44 (18H, s, $CCH_3$). Anal. Found: C, 50.74; H, 6.42. $C_{15}H_{23}Cl_3OSi$ calcd.: C, 50.92; H, 6.55%.

*X-ray crystallography.* All X-ray measurements were made on a Nicolet R3m/V four circle diffractometer, equipped with a LT-1 low-temperature device, operating in the $\theta$–$2\theta$ scan mode with graphite monochromated Mo-$K_\alpha$ radiation ($\lambda$ 0.71069

Table 1

Summary of X-ray diffraction data

| compound | $Me_3Si(BHT)$ | $Me_2ClSi(BHT)$ |
|---|---|---|
| formula | $C_{18}H_{32}OSi$ | $C_{17}H_{29}ClOSi$ |
| space group | $P21/a$ | $P2_1/a$ |
| $a$, Å | 9.129(10) | 9.070(3) |
| $b$, Å | 15.290(7) | 15.189(5) |
| $c$, Å | 13.196(8) | 13.117(9) |
| $\beta$, deg | 95.15(6) | 94.86(4) |
| $V$, $Å^3$ | 1834(2) | 1800(1) |
| $Z$ | 4 | 4 |
| $D$(calcd), $gcm^{-3}$ | 1.059 | 1.154 |
| cryst dimen. mm | 0.40×0.30×0.26 | 0.23×0.31×0.51 |
| temp. (°C) | −80 | −80 |
| radiation | Mo-$K_\alpha$ (0.71073 Å, graphite monochromator) | |
| $2\theta$ limit | 4.0–55.0 | 4.0–55.0 |
| no. of collected | 2703 | 4592 |
| no. of unique | 2402 | 4161 |
| obsd data | 1630 | 3310 |
| $R$ | 0.0643 | 0.0746 |
| $R_w$ | 0.0775 | 0.1341 |
| final residual, $eÅ^{-3}$ | 0.45 | 1.56 |

Table 2

Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement coefficients ($Å^2 \times 10^3$) of the non-hydogen atoms in $Me_3Si(BHT)$

| | $x$ | $y$ | $z$ | $U_{eq}$ [a] |
|---|---|---|---|---|
| Si(1) | 1902(2) | 3996(1) | 8123(1) | 26(1) |
| C(1) | 2875(7) | 3887(4) | 6946(4) | 39(2) |
| C(2) | 727(7) | 3015(3) | 8324(4) | 38(2) |
| C(3) | 3236(7) | 4026(4) | 9274(4) | 42(2) |
| O(1) | 841(4) | 4884(2) | 8070(2) | 23(1) |
| C(4) | 493(6) | 5595(3) | 7428(3) | 21(2) |
| C(5) | −794(6) | 5552(3) | 6745(3) | 19(2) |
| C(6) | −1056(6) | 6254(3) | 6065(3) | 26(2) |
| C(7) | −137(6) | 6975(3) | 6069(3) | 23(2) |
| C(8) | 1011(6) | 7027(3) | 6817(3) | 23(2) |
| C(9) | 1345(6) | 6364(3) | 7539(3) | 22(2) |
| C(10) | −1926(6) | 4790(3) | 6701(4) | 25(2) |
| C(11) | −3345(6) | 5013(4) | 6062(4) | 37(2) |
| C(12) | −1251(7) | 3989(3) | 6199(4) | 35(2) |
| C(13) | −2359(7) | 4577(4) | 7774(4) | 35(2) |
| C(14) | −444(7) | 7698(3) | 5306(4) | 33(2) |
| C(15) | 2576(6) | 6526(3) | 8399(3) | 23(2) |
| C(16) | 2976(6) | 7509(3) | 8510(4) | 31(2) |
| C(17) | 4005(6) | 6058(3) | 8141(4) | 31(2) |
| C(18) | 2110(7) | 6245(3) | 9439(4) | 33(2) |

[a] Equivalent isotropic $U$ defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

Table 3

Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement coefficients ($Å^2 \times 10^3$) of the non-hydrogen atoms in $Me_2ClSi(BHT)$.

| | $x$ | $y$ | $z$ | $U_{eq}$ [a] |
|---|---|---|---|---|
| Si(1) | 3113(1) | 8999(1) | 1863(1) | 24(1) |
| Cl(1B) | 2068 | 8904 | 3149 | 31(1) |
| C(1A) | 2121 | 8743 | 3104 | 28(1) |
| Cl(1A) | 4407 | 7926 | 1639 | 48(1) |
| C(1B) | 4261 | 7849 | 1868 | 30 |
| C(2) | 1766(4) | 9003(2) | 706(3) | 35(1) |
| O(1) | 4163(2) | 9867(1) | 1918(2) | 23(1) |
| C(3) | 4526(3) | 10583(2) | 2559(2) | 18(1) |
| C(4) | 5808(3) | 10536(2) | 3254(2) | 20(1) |
| C(5) | 6062(3) | 11236(2) | 3931(2) | 22(1) |
| C(6) | 5144(3) | 11969(2) | 3929(2) | 23(1) |
| C(7) | 3991(3) | 12025(2) | 3167(2) | 22(1) |
| C(8) | 3675(3) | 11362(2) | 2453(2) | 20(1) |
| C(9) | 6944(3) | 9774(2) | 3274(2) | 24(1) |
| C(10) | 8374(3) | 9998(2) | 3925(3) | 35(1) |
| C(11) | 6280(4) | 8960(2) | 3785(3) | 33(1) |
| C(12) | 7375(4) | 9564(2) | 2196(3) | 34(1) |
| C(13) | 5450(4) | 12700(2) | 4698(3) | 32(1) |
| C(14) | 2442(3) | 11526(2) | 1585(2) | 22(1) |
| C(15) | 1015(3) | 11049(2) | 1841(3) | 28(1) |
| C(16) | 2061(4) | 12507(2) | 1484(2) | 30(1) |
| C(17) | 2943(4) | 11246(2) | 541(2) | 30(1) |

[a] Equivalent isotropic $U$ defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

Å), following procedures previously described in detail [8]. The structure was solved and refined via direct methods and full matrix least squares in a routine manner. All non-hydrogen atoms, except those involved in disorder (vide infra), were refined anisotropically and, although most hydrogen atoms were located in difference maps, all were included in idealized positions ($U_{iso}(H) = 1.2\ U_{iso}(C)$; $d(C-H)$ 0.96 Å) for refinement.

After all of the non-hydrogen atoms in compound **2** were located and refined anisotropically, C(1a) and Cl(1a) refined to non-positive definite thermal parameters, and a difference map revealed peaks in the appropriate location for a Cl atom attached to Si near C(1a), and a C atom attached to Si near Cl(1a). Refinement of the occupancy of C(1a) and the position nearby assigned as Cl(1b) indicated a disorder between C and Cl. The site occupancy factor for C(1a) and Cl(1b) was allowed to refine freely resulting in a 74.64% (C(1a)) 25.36% (Cl(1b)) occupancy. C(1b) and Cl(1a) were therefore refined with occupancy 25.36% and 74.64% respectively. All disordered atoms were refined isotropically. Hydrogen atoms on C(1a) and C(1b) were included in idealized positions with the appropriate site occupancy.

A crystal data summary is given in Table 1; fractional atomic coordinates are listed in Tables 2 and 3; bond lengths and angles are given in Tables 4 and 5. Tables of anisotropic thermal parameters, hydrogen parameters, and observed and calculated structure factors are available from the authors (A.R.B.).

## Results and discussion

Interaction of one equivalent of NaBHT with $Me_3SiCl$, $Me_2SiCl_2$ and $SiCl_4$ gives $Me_3Si(BHT)$ (**1**), $Me_2ClSi(BHT)$ (**2**) and $Cl_3Si(BHT)$ (**3**) respectively as colorless sublimable solids. The $^1H$ NMR and analytical data for all the compounds is consistent with their formulation. The structures of **1** and **2** have been determined by X-ray crystallography.

The molecular structures of **1** and **2** are shown in Fig. 1 and 2, respectively; selected bond lengths and angles are given in Tables 4 and 5. The structures consist of discrete monomeric units. The geometry around Si in both compounds is essentially tetrahedral, with the sum of the X–Si–Y angles, not including oxygen (326.7° (**1**), 320.2° (**2**)) being close to that for tetrahedron (328.5°). Similar geometries have been observed in silicon compounds where no $\pi$-bonding is present [9,10,11].

The Si–O–C angles (139.4(3)° (**1**), 140.0(2)° (**2**)) are larger than observed for silyl ethers (120–131°) [9,10,11]. They are, however, similar to the values predicted for $R_3SiOPh$ (R = Me, Et, Pr) from dipole moment measurements (140°) [12]. This would suggest that the increase in the Si–O–C angle is perhaps due to the presence of the phenyl ring and not to any $\pi$-interaction with the Si–X $\sigma^\star$ orbital or steric effects of the *ortho* t-butyl groups. The Si–O bond distances (1.666(4) Å (**1**), and 1.624(2) Å (**2**)) are larger than or within the range previously observed for Si–O bonds, 1.612–1.640 Å [9,10,11].

We propose that, unlike the analogous aluminum compounds [5], there is no significant $\pi$-interaction between the oxygen lone pair orbitals and the Si–X $\sigma^\star$ orbitals in these aryloxide compounds of silicon. It should be noted that this does not preclude the presence of a $\pi$-interaction involving the Si 3*d* orbits [6]. The large

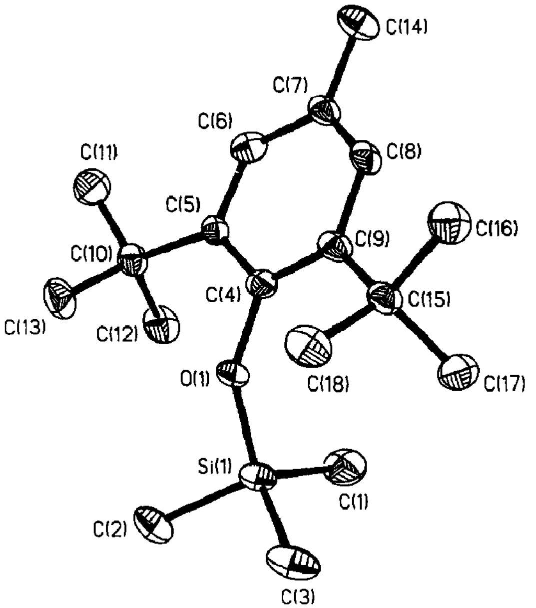


Fig. 1. The structure of $Me_3Si(BHT)$. Thermal ellipsoids are drawn at the 50% level, and the hydrogen atoms are omitted for clarity.

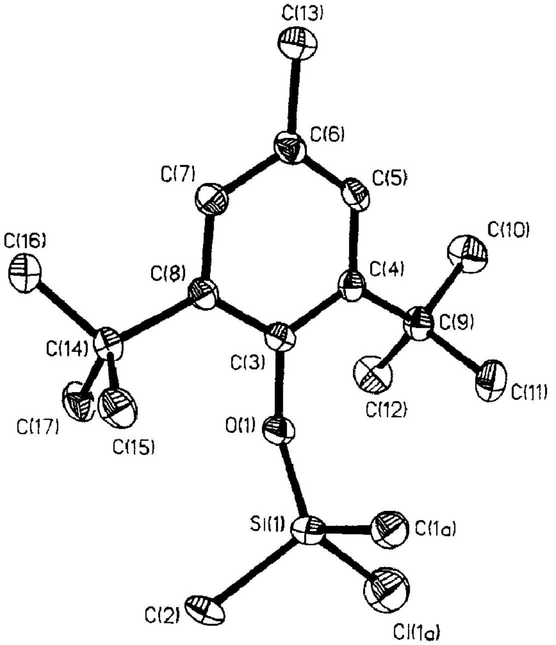


Fig. 2. The structure of Me$_2$ClSi(BHT). Thermal ellipsoids are drawn at the 50% level, and the hydrogen atoms are omitted for clarity. The position of C(1) and Cl(1) are disordered, only the more abundant (75%) structure is shown.

Table 4

Bond lengths (Å) and bond angles (deg) in Me$_3$Si(BHT)

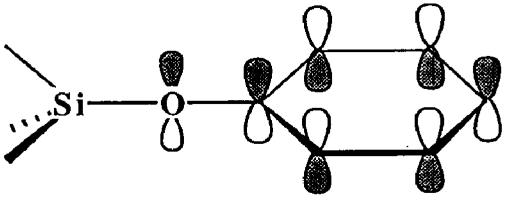
| | | | |
|---|---|---|---|
| Si(1)-C(1) | 1.866 (6) | Si(1)-C(2) | 1.877 (6) |
| Si(1)-C(3) | 1.860 (6) | Si(1)-O(1) | 1.666 (4) |
| O(1)-C(4) | 1.399 (6) | C(4)-C(5) | 1.416 (7) |
| C(4)-C(9) | 1.410 (7) | C(5)-C(6) | 1.406 (7) |
| C(5)-C(10) | 1.556 (7) | C(6)-C(7) | 1.386 (7) |
| C(7)-C(8) | 1.375 (7) | C(7)-C(14) | 1.505 (7) |
| C(8)-C(9) | 1.406 (7) | C(9)-C(15) | 1.543 (7) |
| C(10)-C(11) | 1.520 (7) | C(10)-C(12) | 1.547 (7) |
| C(10)-C(13) | 1.538 (7) | C(15)-C(16) | 1.551 (7) |
| C(15)-C(17) | 1.552 (8) | C(15)-C(18) | 1.535 (7) |
| C(1)-Si(1)-C(2) | 111.6(3) | C(1)-Si(1)-C(3) | 110.9(3) |
| C(2)-Si(1)-C(3) | 104.2(3) | C(1)-Si(1)-O(1) | 110.9(2) |
| C(2)-Si(1)-O(1) | 108.6(2) | C(3)-Si(1)-O(1) | 110.5(2) |
| Si(1)-O(1)-C(4) | 139.4(3) | O(1)-C(4)-C(5) | 118.6(4) |
| O(1)-C(4)-C(9) | 119.5(4) | C(5)-C(4)-C(9) | 121.6(4) |
| C(4)-C(5)-C(6) | 117.0(4) | C(4)-C(5)-C(10) | 124.7(4) |
| C(6)-C(5)-C(10) | 118.3(4) | C(5)-C(6)-C(7) | 122.5(5) |
| C(6)-C(7)-C(8) | 118.1(4) | C(6)-C(7)-C(14) | 120.4(4) |
| C(8)-C(7)-C(14) | 121.5(5) | C(7)-C(8)-C(9) | 123.3(5) |
| C(4)-C(9)-C(8) | 116.5(4) | C(4)-C(9)-C(15) | 124.6(4) |
| C(8)-C(9)-C(15) | 118.9(4) | C(5)-C(10)-C(11) | 112.5(4) |
| C(5)-C(10)-C(12) | 108.7(4) | C(11)-C(10)-C(12) | 107.0(4) |
| C(5)-C(10)-C(13) | 110.5(4) | C(11)-C(10)-C(13) | 106.4(4) |
| C(12)-C(10)-C(13) | 111.8(4) | C(9)-C(15)-C(16) | 112.0(4) |
| C(9)-C(15)-C(17) | 109.6(4) | C(16)-C(15)-C(17) | 105.8(4) |
| C(9)-C(15)-C(18) | 111.8(4) | C(16)-C(15)-C(18) | 105.6(4) |
| C(17)-C(15)-C(18) | 111.9(4) | | |

Table 5

Bond lengths (Å) and bond angles (deg) in $Me_2ClSi(BHT)$.

| | | | |
|---|---|---|---|
| Si(1)-Cl(1B) | 2.009 (2) | Si(1)-C(1A) | 1.965 (2) |
| Si(1)-Cl(1A) | 2.045 (2) | Si(1)-C(1B) | 2.034 (2) |
| Si(1)-C(2) | 1.865 (4) | Si(1)-O(1) | 1.624 (2) |
| O(1)-C(3) | 1.396 (3) | C(3)-C(4) | 1.418 (4) |
| C(3)-C(8) | 1.413 (4) | C(4)-C(5) | 1.392 (4) |
| C(4)-C(9) | 1.549 (4) | C(5)-C(6) | 1.390 (4) |
| C(6)-C(7) | 1.386 (4) | C(6)-C(13) | 1.510 (4) |
| C(7)-C(8) | 1.389 (4) | C(8)-C(14) | 1.547 (4) |
| C(9)-C(10) | 1.530 (4) | C(9)-C(11) | 1.552 (5) |
| C(9)-C(12) | 1.531 (5) | C(14)-C(15) | 1.545 (4) |
| C(14)-C(16) | 1.532 (4) | C(14)-C(17) | 1.539 (4) |
| C(1A)-Si(1)-Cl(1A) | 105.7(1) | Cl(1B)-Si(1)-C(1B) | 102.3(1) |
| Cl(1B)-Si(1)-C(2) | 111.1(1) | C(1A)-Si(1)-C(2) | 111.2(1) |
| Cl(1A)-Si(1)-C(2) | 103.3(1) | C(1B)-Si(1)-C(2) | 107.8(1) |
| Cl(1B)-Si(1)-O(1) | 109.7(1) | C(1A)-Si(1)-O(1) | 115.6(1) |
| Cl(1A)-Si(1)-O(1) | 108.1(1) | C(1B)-Si(1)-O(1) | 113.5(1) |
| C(2)-Si(1)-O(1) | 112.0(1) | Si(1)-O(1)-C(3) | 140.0(2) |
| O(1)-C(3)-C(4) | 119.1(2) | O(1)-C(3)-C(8) | 119.7(2) |
| C(4)-C(3)-C(8) | 121.1(2) | C(3)-C(4)-C(5) | 117.0(3) |
| C(3)-C(4)-C(9) | 123.8(2) | C(5)-C(4)-C(9) | 119.2(2) |
| C(4)-C(5)-C(6) | 123.0(3) | C(5)-C(6)-C(7) | 117.8(3) |
| C(5)-C(6)-C(13) | 120.8(3) | C(7)-C(6)-C(13) | 121.4(3) |
| C(6)-C(7)-C(8) | 122.8(3) | C(3)-C(8)-C(7) | 117.5(2) |
| C(3)-C(8)-C(14) | 124.1(2) | C(7)-C(8)-C(14) | 118.3(3) |
| C(4)-C(9)-C(10) | 111.9(3) | C(4)-C(9)-C(11) | 108.7(2) |
| C(10)-C(9)-C(11) | 106.1(3) | C(4)-C(9)-C(12) | 111.2(2) |
| C(10)-C(9)-C(12) | 106.9(3) | C(11)-C(9)-C(12) | 111.9(3) |
| C(8)-C(14)-C(15) | 109.3(2) | C(8)-C(14)-C(16) | 111.4(2) |
| C(15)-C(14)-C(16) | 106.7(3) | C(8)-C(14)-C(17) | 111.1(2) |
| C(15)-C(14)-C(17) | 112.3(3) | C(16)-C(14)-C(17) | 105.9(2) |

Si–O–C angles observed for **1** and **2** could, however, arise primarily from $\pi$-delocalization of the aryloxide lone pair perpendicular to the aryl ring and the aryl ring $\pi^*$ orbital (III).



(III)

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