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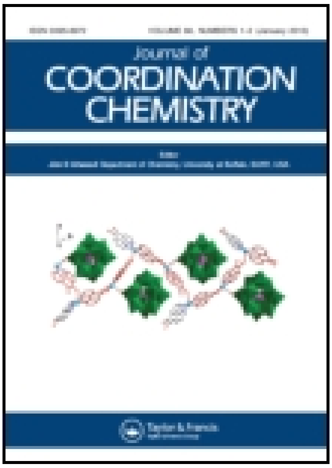

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Synthesis and characterization of aryltriphenylsilyl ethers and crystal structure of 2,4,6-tri-methylphenyl triphenylsilyl ether

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Synthesis and characterization of aryltriphenylsilyl ethers and crystal structure of 2,4,6-*tri*-methylphenyl triphenylsilyl ether

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Synthesis and structures of aryl triphenylsilyl ether derivatives are reported. Reactions of chlorotriphenylsilane, $(C_6H_5)_3SiCl$ (**1**), with sodium salts of sterically hindered phenol derivatives (**2a**–**2i**) were investigated. Aryltriphenylsilyl ethers $(C_6H_5)_3SiOAr$ (**3–8**) were obtained from these reactions. Structures of **3–8** were defined by IR, ^1H, ^{13}C, ^{29}Si NMR spectroscopy, elemental analysis, and ESI-MS spectra. The molecular structure of 2,4,6-*tri*-methylphenyl triphenylsilyl ethe (**6**) were determined by single-crystal X-ray diffraction.

Keywords: Aryltriphenylsilyl ether; Chlorotriphenylsilane; ^{29}Si NMR; Phenol

1. Introduction

Organosilicon chemistry is a rapidly developing field of science [1]. Organosilicon reagents have a range of applications in organic synthesis [2]. Silicon–oxygen bond-forming reactions are one of the most important reactions leading to useful siloxane polymers [3]. Monomeric organosilicon compounds are important in a range of applications apart from silicone chemistry, which is the largest consumer of silanes [4]. Polymers containing silicon–oxygen bonds in the main chain are important [5, 6] and silyl ethers are among the most widely used protecting groups for hydroxyl functionality in organic synthesis [7–9]. Silicon–oxygen compounds also find applications in stereoselective transformations of organic compounds [10]. They also play an important role in inorganic synthesis as precursors in the preparation of sol–gels and other condensed siloxane materials. Therefore, compounds containing a Si–O bond are important.

Generally, three approaches are used for the synthesis of silyl ethers. Silyl ethers are most commonly prepared from chlorosilanes ($R_{4-x}SiCl_x$) by reaction with alcohols or alkoxides under acidic or basic reaction conditions [11]. The second method involves

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cleavage of Si–N or Si–S bonds with alcohols or water and requires substrates that undergo such alcoholysis reactions. The third method, dehydrogenative coupling reaction between Si–H and O–H functional groups, is user friendly and proceeds under mild reaction conditions being catalyzed by various metal complexes [12].

Sterically hindered phenols are biologically active compounds [13]. Especially, 2,6-di-*tert*-butyl-4-methylphenol (butylated hydroxy toluene, BHT) derivatives are efficient antioxidants [14]. Here we report the synthesis and characterizations of aryl triphenylsilyl ethers via the reaction of chlorotriphenylsilane (**1**) with sterically hindered phenol derivatives. These compounds could find potential applications as effective antioxidants.

2. Experimental

All synthetic steps were carried out under N_2 or Ar in predried glassware using Schlenk techniques [15] with previously dried solvents. Tetrahydrofuran (THF), used as solvent, was distilled under argon from sodium benzophenone, prior to use. Starting materials were commercially available (Aldrich, Merck, Alfa Aesar, and Fluka) and used without purification. Reactions were monitored by using silica gel 60 F_{254} precoated Thin Layer Chromatography plates (Merck, Kieselgel 60, 0.25 mm thickness) and separating conditions were determined. The separation of products was carried out by column chromatography using silica gel (Merck, Kieselgel 60, 230–400 mesh; for 3 g crude mixture, 100 g silica gel was used in a column, 3 cm in diameter and 60 cm in length).

All isolated new compounds have been characterized by elemental analysis, ^{1}H, ^{13}C, ^{29}Si NMR spectrometry, FT-IR, and mass spectrometry. Microanalysis was carried out on a LECO 932 CHNS-O apparatus. Melting points were measured in open capillary tubes with an Electrothermal-9200 melting point apparatus and were uncorrected. IR spectra were recorded on an ATI Unicam Mattson 1000 FT-IR spectrophotometer in KBr disks and were reported in cm^{-1}. ^{1}H, ^{13}C, and ^{29}Si NMR spectra were recorded in $CDCl_3$ using a Varian INOVA spectrometer operating at 500 MHz (^{1}H), 125.6 MHz (^{13}C), and 99.3 MHz (^{29}Si). The ^{1}H, ^{13}C, and ^{29}Si NMR chemical shifts were measured using $SiMe_4$ ($\delta = 0$) as an internal standard. Mass spectra were obtained by a Bruker MicROTOF LC/MS spectrometer using electrospray ionization (ESI) method.

2.1. *Synthesis and characterization of phenyl triphenylsilyl ether derivatives (3–8)*

2.1.1. Synthesis of 3. Small pieces of metallic sodium (0.15 g, 6.52 mM) were added to the solution of 2,4-*di*-methylphenol (**2a**) (0.48 g, 3.92 mM) in 25 mL of THF under Ar and the solution was stirred until cessation of hydrogen gas evolution, an indication of completion of the reaction. Excess sodium was removed by filtration and the solution of sodium-2,4-di-methylphenoxide was frozen with a liquid nitrogen–acetone mixture (−95 °C ± 5 °C). To this solution, chlorotriphenylsilane (1 g, 3.40 mM) in 10 mL of THF was slowly added and the resulting mixture was allowed to warm to ambient temperature with continued stirring. The reaction mixture was vigorously stirred at room temperature for 48 h, precipitated NaCl was filtered off, and solvent was removed under vacuum. The residue was chromatographed on silica gel: 100 g (eluent : acetone/*n*-hexane 1 : 2). 2,4-*di*-methylphenyl triphenylsilyl ether (**3**), as white crystals, was obtained in 11% yield, m.p. = 86–88 °C ($R_f = 0.879$ dichloromethane/*n*-hexane 1 : 2). IR (KBr, cm^{-1}): $\upsilon_{(\mathrm{CH\ aryl})} = 3066$,

$\upsilon_{(\mathrm{CH\ al})}=2916$, $\upsilon_{(\mathrm{CC\ aryl})}=1428$, $\upsilon_{(\mathrm{Si–C})}=1265$, $\upsilon_{(\mathrm{Si–O–C})}=1118$, 1027. NMR ($CDCl_3$, δ, ppm): ^{1}H, 2.10 (s, 3H, 4–CH_3), 2.15 (s, 3H, 2–CH_3), 6.48–6.83 (m, 3H, Ar–O), 7.56–7.59 (m, 15H, Ar–*H*). ^{13}C, 151.2, 135.8, 135.4, 134.0, 130.5, 130.2, 128.5, 127.9, 127.7, 126.9, 118.4, 20.5, 17.0. ^{29}Si, –15.23 (s). ESI-MS *m*/*z*: 403.2 $[M+Na]^+$. Found: C, 82.32; H, 6.5. Calcd (%) for $C_{26}H_{24}SiO$ (380.56): C, 82.10; H, 6.31.

2.1.2. Synthesis of 4. 3,4-*di*-methylphenol (**2b**) (0.48 g, 3.92 mM), Na (0.15 g, 6.52 mM), and **1** (1 g, 3.40 mM) were used for the preparation of **4** as for **3**. The residue was chromatographed (silica gel: 100 g, eluent : dichloromethane/*n*-hexane 1 : 1) giving 3,4-*di*-methylphenyl triphenylsilyl ether (**4**) in 19% yield, m.p. = 56–58 °C ($R_f = 0.657$ dichloromethane/*n*-hexane 1 : 3). IR (KBr, cm^{-1}): $\upsilon_{(\mathrm{CH\ aryl})}=3066$, $\upsilon_{(\mathrm{CH\ al})}=2920$, $\upsilon_{(\mathrm{CC\ aryl})}=1427$, $\upsilon_{(\mathrm{Si–C})}=1257$, $\upsilon_{(\mathrm{Si–O–C})}=1116$. NMR ($CDCl_3$, δ, ppm): ^{1}H, 2.26 (s, 3H, 4–CH_3), 2.13 (s, 3H, 3–CH_3), 6.61–6.89 (m, 3H, Ar–O), 7.38–7.50 (m, 15H, Ar–*H*). ^{13}C, 152.9, 137.5, 135.7, 135.5, 135.2, 133.8, 130.2, 130.1, 129.8, 129.7, 127.9, 127.7, 116.8, 121.2, 19.8, 18.8. ^{29}Si, –15.15 (s). ESI-MS *m*/*z*: Found: C, 82.24; H, 6.53. Calcd (%) for $C_{26}H_{24}SiO$ (380.56): C, 82.10; H, 6.31.

2.1.3. Synthesis of 5. 2,6-*di*-methylphenol (**2c**) (0.41 g, 3.36 mM), Na (0.15 g, 6.52 mM), and **1** (1 g, 3.40 mM) were used for the preparation of **5** as for **3**. The residue was chromatographed (eluent : dichloromethane/*n*-hexane 1 : 2) and 2,6-*di*-methylphenyl triphenylsilyl ether (**5**) was obtained in 11% yield, m.p. = 91–93 °C ($R_f = 0.540$ acetone/*n*-hexane 1 : 2) IR (KBr, cm^{-1}): $\upsilon_{(\mathrm{CH\ aryl})}=3067$, $\upsilon_{(\mathrm{CH\ al})}=2915$, $\upsilon_{(\mathrm{CC\ aryl})}=1427$, $\upsilon_{(\mathrm{Si–C})}=1268$, $\upsilon_{(\mathrm{Si–O–C})}=1116$, 1028. NMR ($CDCl_3$, δ, ppm): ^{1}H, δ 2.21 (s, 6H, 2,6-di-CH_3), 6.81–6.95 (m, 3H, Ar–O); 7.44–7.71 (m, 15H, Ar–*H*). ^{13}C, 152.2, 135.5, 135.2, 134.1, 130.2, 129.8, 127.7, 121.8, 18.54. ^{29}Si, –16.50 (s). ESI-MS *m*/*z*: 403.2 $[M+Na]^+$. C, 82.10; H, 6.31. Found: C, 82.08; H, 6.48. Calcd (%) for $C_{26}H_{24}SiO$ (380.56): C, 82.10; H, 6.31.

2.1.4. Synthesis of 6. 2,4,6-*tri*-methylphenol (**2d**) (0.55 g, 4.04 mM), Na (0.18 g, 7.82 mM) and **1** (1 g, 3.40 mM) were used for the preparation of **6** as for **3**. The residue was chromatographed (eluent : dicholoromethane/*n*-hexane 1 : 1) to give 2,4,6-*tri*-methylphenyl triphenylsilyl ether (**6**) in 49% yield, m.p. = 119–120 °C ($R_f = 0.658$ dichloromethane/*n*-hexane 1 : 2). IR (KBr, cm^{-1}): $\upsilon_{(\mathrm{CH\ aryl})}=3045$, $\upsilon_{(\mathrm{CH\ al})}=2909$, $\upsilon_{(\mathrm{CC\ aryl})}=1426$, $\upsilon_{(\mathrm{Si–C})}=1234$, $\upsilon_{(\mathrm{Si–O–C})}=1116$. NMR ($CDCl_3$, δ, ppm): ^{1}H, δ 1.92 (s, 6H, 2,6-*di*-CH_3), 2.23 (s, 3H, 4-CH_3), 6.72 (s, 2H, ArO–H), 7.28–7.67 (m, 15H, Ar–*H*), ^{13}C, 149.9, 135.5, 135.3, 134.3, 130.8, 129.2, 128.4, 127.6, 20.5, 18.4. ^{29}Si, –16.81 (s). ESI-MS *m*/*z*: 395 $[M^+]$. Found: C, 82.18; H, 6.61 %. Calcd (%) for $C_{27}H_{27}SiO$ (395.59): C, 82.15; H, 6.59.

2.1.5. Synthesis of 7. 4-*tert*-butyl-2-methylphenol (**2f**) (0.56 g, 3.41 mM), Na (0.16 g, 6.95 mM), and **1** (1 g, 3.40 mM) were used for the preparation of **7** as for **3**. The residue was chromatographed (eluent : dichloromethane/*n*-hexane 1 : 1) giving 4-*tert*-butyl-2-methylphenyl triphenylsilyl ether (**7**) in 35% yield, m.p. = 127–128 °C ($R_f = 0.682$ dichloromethane/*n*-hexane 1 : 2). IR (KBr, cm^{-1}): $\upsilon_{(\mathrm{CH\ aryl})}=3067$, $\upsilon_{(\mathrm{CH\ al})}=2960$, $\upsilon_{(\mathrm{CC\ aryl})}=1428$, $\upsilon_{(\mathrm{Si–C})}=1255$, $\upsilon_{(\mathrm{Si–O–C})}=1116$. NMR ($CDCl_3$, δ, ppm): ^{1}H, δ 1.14 (s, 9H, 4-Bu^t), 2.20 (s, 3H, 2-CH_3), 6.71–6.79 (m, 3H, Ar–O) 7.27–7.59 (m, 15H, Ar–*H*). ^{13}C, 151.1, 143.9, 135.9, 135.7, 135.5, 135.2, 135.0, 130.2, 127.9, 127.8, 127.7, 123.2, 117.9, 34.0, 31.5, 17.4. ^{29}Si, –15.62 (s). ESI-MS *m/z*: 445.2 $[M+Na]^+$. Found: C, 82.52; H, 7.27. Calcd (%) for $C_{29}H_{30}SiO$ (422.64): C, 82.34; H, 7.09.

2.1.6. Synthesis of 8. 2-*tert*-butyl-4-methylphenol (**2g**) (0.63 g, 3.83 mM), Na (0.17 g, 7.39 mM), and **1** (1 g, 3.40 mM) were used for the preparation of **8** as for **3**. The residue was chromatographed (eluent : dichloromethane/*n*-hexane 1 : 1) and 2-*tert*-butyl-4-methylbutylphenyl triphenylsilyl ether (**8**) was obtained in 32% yield, m.p. = 114–116 °C ($R_f = 0.676$ dichloromethane/*n*-hexane 1 : 2). IR (KBr, cm^{-1}): $\upsilon_{(CH\ aryl)} = 3047$, $\upsilon_{(CH\ al)} = 2945$, $\upsilon_{(CC\ aryl)} = 1427$, $\upsilon_{(Si–C)} = 1231$, $\upsilon_{(Si–O–C)} = 1115$. NMR ($CDCl_3$, δ, ppm): ^{1}H, 1.59 (s, 9H, 2-Bu^t), 2.35 (s, 3H, 4-CH_3), 6.77 (s, 3H, Ar–O), 7.24–7.85 (m, 15H, Ar), ^{13}C, 151.7, 139.0, 135.5, 135.2, 133.8, 130.2, 130.0, 128.0, 127.7, 126.9, 119.8, 34.7, 29.8, 20.9. ^{29}Si, –15.40. ESI-MS *m/z*: 445.2 $[M+Na]^+$. Found: C, 82.54; H, 7.32 %. Calcd (%) for $C_{29}H_{30}SiO$ (422.64): C, 82.34; H, 7.09.

2.2. *X-ray crystallography*

Crystallographic data were collected on a Bruker SMART APEXII diffractometer using MoKα radiation ($\lambda = 0.71073$ Å). Absorption correction by multi-scan was applied [16], the structure was solved by direct methods and refined by full-matrix least squares against F^2 using all data [17]. All non-H atoms were refined anisotropically. Hydrogen positions were calculated geometrically at 0.95 Å (CH) and 0.98 Å (CH_3) from the parent carbon; a riding model was used during the refinement process, and the U_{iso} (H) values were constrained to

Table 1. Crystallographic data of **6**.

	6
Empirical formula	$C_{27}H_{26}OSi$
Fw	394.57
T (K)	120(2)
Crystal system	Triclinic
Space group	P−1
a (Å)	10.2419(3)
b (Å)	11.3407(3)
c (Å)	11.4646(5)
α (°)	109.9160(10)
β (°)	103.7490(10)
γ (°)	109.9990(10)
V (Å^3)	1077.81(6)
Z	2
μ(mm^{-1}) (Mo K$_\alpha$)	0.124
Reflection collected	14,791
Reflection total	3793
Reflection unique	3557
Parameters	265
R_{int} (merging R value)	0.0177
θ_{max} (°)	25.03
T_{min}/T_{max}	0.9449/0.9689
Goodness-of-fit on F^2	1.063
R [$F^2 > 2\sigma(F^2)$]	0.0324
wR	0.0830

be 1.2 U_{eq} (CH) and 1.5 U_{eq} (CH_3). The general-purpose crystallographic tool PLATON [18] was used for structure analysis and presentation of the results. The figure was drawn with DIAMOND (Version 3.1) [19]. Crystal structure and refinement data of **6** are summarized in table 1.

3. Results and discussion

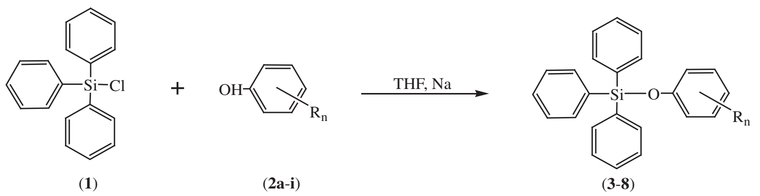
Reactions of chlorotriphenylsilane with an equimolar amount of sodium salts of sterically hindered phenols (**2a**-**2i**) in THF gave aryl triphenylsilyl ether derivatives (**3–8**) (table 2). Products were isolated from the reaction mixtures by column chromatography. The structures of **3–8** identified by ^{1}H, ^{13}C, ^{29}Si NMR, ESI-MS, elemental analyses, and FT-IR spectroscopy were consistent with the analytical and spectroscopic data (mentioned in the Experimental section). White solid **3–8** were stable in air and water.

Compounds **3–8** were obtained in low yield, from 11 to 49%. The nature of solvent and reagents affects the yields which were low, perhaps a result of losses during purification of the silyl ethers by column chromatography.

Compound **1** was also reacted with 2-*tert*-butyl-6-methylphenol (**2g**), 2,6-di-*tert*-butyl-4-methylphenol (**2h**), and 2,4,6-tri-*tert*-butylphenol (**2i**). But, pure and defined compounds could not be obtained from these reactions; steric hindrance plays an important role in these reactions.

Although **6** was synthesized by others with high yield in the presence of $(B(C_6F_5)_3$ as catalyst, the structural elucidation was given with spectroscopic data only [20]. In this study, the same compound was prepared without catalyst in moderate yield and structural characterization was done by X-ray diffraction.

Table 2. Preparations of silyl ether derivatives.



Compound	R_n	M.p. (°C)	Yield (%)	R_f	Chromatographic eluent ratios
3	2,4-Dimethyl	86–88	11	0.879 [CH_2Cl_2-*n*-hexane (1 : 2)]	Acetone$_2$-*n*-hexane (1 : 2)
4	3,4-Dimethyl	56–58	19	0.657 [CH_2Cl_2-*n*-hexane (1 : 3)]	CH_2Cl_2-*n*-hexane (1 : 1)
5	2,6-Dimethyl	91–93	11	0.540 [Acetone$_2$-*n*-hexane (1 : 2)]	CH_2Cl_2-*n*-hexane (1 : 2)
6	2,4,6-Dimethyl	119–120	49	0.658 [CH_2Cl_2-*n*-hexane (1 : 2)]	CH_2Cl_2-*n*-hexane (1 : 1)
7	4-*tert*-Butyl-2-dimethyl	127–128	35	0.682 [CH_2Cl_2-*n*-hexane (1 : 2)]	CH_2Cl_2-*n*-hexane (1 : 1)
8	2-*tert*-Butyl-4-dimethyl	114–116	32	0.676 [CH_2Cl_2-*n*-hexane (1 : 2)]	CH_2Cl_2-*n*-hexane (1 : 1)

3.1. *Spectroscopic studies*

The aryl and alkyl C–H stretching frequencies of **3–8** occur at 3045–3067 cm^{-1} and 2915–2960 cm^{-1}, respectively. Si–C bonds had strong infrared absorbances at 1231–1268 cm^{-1} [21–23]. These derivatives also showed strong intensity absorptions at 1115–1118 from (Si–O–C) stretching vibrations. These data are in agreement with the literature [23–25].

^{1}H–, ^{13}C–, ^{29}Si–NMR data provided strong evidence for the structures of **3–8**. In ^{1}H–NMR spectra all CH_3 protons were singlets. The phenoxy protons were at $\delta = 6.48$–6.79, $\delta = 6.61$–6.89, $\delta = 6.81$–6.95, and $\delta = 6.71$–6.79 as multiplets for **3**, **4**, **5,** and **7**, respectively. The phenoxy protons were at $\delta = 7.56$–7.59, $\delta = 7.38$–7.50, $\delta = 7.44$–7.71, $\delta = 7.28$–7.67, $\delta = 7.27$–7.69, and $\delta = 7.24$–7.85 as multiplets for **3**, **4**, **5**, **6**, **7,** and **8**, respectively. The methyl protons resonate at $\delta = 2.10$ (4-CH_3), 2.15 (2–CH_3) (in a 1 : 1 ratio); 2.26 (4–CH_3), 2.13 (3–CH_3) (in a 1 : 1 ratio), and 2.21 (2,6-*di*–CH_3) as singlet for **3**, **4,** and **5**, respectively.

The methyl protons resonate at $\delta = 2.23$ (2–CH_3) and 1.92 (4–CH_3) (in a 2 : 1 ratio) for **6**. For **7**, the protons of the *tert*-butyl group at the para position and methyl group at the ortho position gave singlets at $\delta = 1.14$ and 2.20 (in a 3 : 1 ratio), respectively. The protons of the methyl at the para position and *tert*-butyl group at the ortho position in **8** gave singlets at $\delta = 2.35$ and 1.59 (in a 1 : 3 ratio), respectively. These data were in agreement with the literature [26, 27].

The ^{29}Si NMR spectra of these compounds contain signals characteristic of silyl ether derivatives between −15.15 and −16.81 ppm [28] (a sharp single peak at −15.23 for **3**, −15.15 for **4**, −16.50 for **5**, −16.81 for **6**, −15.62 for **7,** and −15.40 for **8)**.

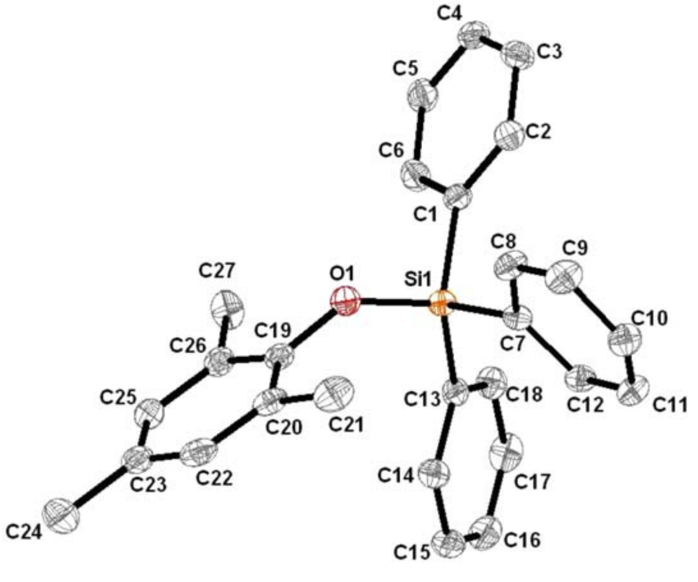


Figure 1. View of the molecular structure for **6** with the atom-numbering scheme. Displacement ellipsoids are drawn at 30% probability level. Hydrogens have been omitted for clarity. Selected bond parameters: Si–O: 1.6517 (10) Å; Si–C1: 1.8666(14) Å; Si–C7: 1.8697(14) Å; SiC13: 1.8681(14) Å; O–C19: 1.3814(17) Å; C1–Si–C13: 112.83(6)°; C7–Si–C13: 109.04(6)°; C1–Si–C7: 110.63(6)°; C1–Si–O: 105.12(6)°; C7–Si–O: 108.70(6)°; C13–Si–O: 110.41(6)°; SiO–C19: 136.79(9)°.

*X-ray crystal structure of **6**.* The data collection and refinement parameters of **6** are presented in table 1. The molecular structure is shown in figure 1. Compound **6** shows a distorted tetrahedral coordination sphere around Si. Selected bond parameters also are given in figure 1. The three SiC bond lengths were 1.8666(14)–1.8697(14) Å and the tetrahedral C–Si–C bond angles were 109.04(6)°, 110.63(6)°, and 112.83(6)°. The Si–O distance is 1.6517(10) Å. The O–Si–C bond angle values were 105.12(6)°, 108.70(6)°, and 110.41(6)°. These values are close to those reported for triphenylsilanol [29–32]. The sterical hindrance between the tris-methylphenoxy-substituent and the three phenyl groups is reflected by a large Si–O–C19 bond angle of 136.79(9)°, comparable to those reported in the ether isopropenyloxy[tris(pentafluorophenyl)]silane (138.9(1)°) [33] and tert-butoxytriphenylsilane (135.97 (12)°) [34], but larger than those reported in the 2-(methoxy)ethoxy (triphenyl)silane (123.08°) [35].

Close investigation of crystal structures of **6** shows many intermolecular interactions where separations between donors and acceptors are less than 3.2 Å. These intermolecular π–π interactions are predominantly between phenyl groups.

4. Conclusion

Reactions of chlorotriphenylsilane with sterically hindered phenols were investigated at room temperature. Five silyl ether derivatives (**3**, **4**, **5**, **7**, and **8**) from these reactions were prepared at room temperature. Structure of **6** was clarified with X-ray diffraction.

Sterically hindered phenols are efficient antioxidants. Separately, silyl ether derivatives are used as protecting groups in organic synthesis. Thus, these compounds could find applications as effective antioxidants and protecting groups.

Supplementary material

CCDC-793401 contains the supplementary crystallographic data for compound **6**. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

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