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# Synthesis of Functionalized 2,3,4,5-Tetraphenylsilole Derivatives Through Hydrosilylation and Their Crystal Structures

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### SYNTHESIS OF FUNCTIONALIZED 2,3,4,5-TETRAPHENYLSILOLE DERIVATIVES THROUGH HYDROSILYLATION AND THEIR CRYSTAL STRUCTURES

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#### **GRAPHICAL ABSTRACT**



**Abstract** A series of new functionalized 2,3,4,5-tetraphenylsiloles were successfully synthesized in good yields by hydrosilylation reaction of 1-methyl-2,3,4,5-tetraphenyl-1H-silole with p-ethynyl benzene derivatives. This synthesis provides a convenient method of introducing reactive electron-withdrawing or electro-donating groups into siloles. All new siloles show aggregation-induced emission (AIE) effects.

Keywords Aggregation-induced emission; functionalized siloles; hydrosilylation

#### INTRODUCTION

2,3,4,5-Tetraphenyl siloles or silacyclopentadienes are a group of appealing molecules because of their opto-electronic properties, especially their aggregation-induced emission (AIE),<sup>[1–4]</sup> which is attributed to restricted intramolecular rotations of the peripheral phenyl rings against the central silole core. These molecules demonstrate potential applications in the field of organic-based devices, including light-emitting diodes and chemical sensors.<sup>[5–9]</sup>

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Recently, functionalized silole derivatives have attracted much attention because of their potential as chemosensors<sup>[10–13]</sup> and bioprobes.<sup>[14–18]</sup> However, the reported functionalized siloles are very limited, and the development of such new molecules is still highly desired. Herein, we report synthesis of a series of new siloles containing a functional styryl group at the silicon moiety via a convenient hydrosilylation reaction of 1-methyl-2,3,4,5-tetraphenylsilole (1) with *p*-ethynyl benzene derivatives. Amino- and carboxyl-group-substituted siloles were readily synthesized in good yields.

#### SYNTHESIS AND CRYSTAL STRUCTURES

The synthesis of functionalized 2,3,4,5-tetraphenylsiloles was carried out by hydrosilylation of 1-methyl-2,3,4,5-tetraphenylsilole (1) (Scheme 1). The introduction of amino, bromo, and carboxyl groups to siloles was accomplished by hydrosilylation reaction of 1-methyl-2,3,4,5-tetraphenylsilole with the corresponding functionalized phenylacetylene, respectively.

An initial test of hydrosilylation of **1** with 4-ethynylaniline was first performed in tetrahydrofuran (THF) with RhCl(PPh<sub>3</sub>)<sub>3</sub> as catalyst. However, after refluxing for 24 h, the process only produced target molecule (E)-1-methyl-1-(4-amino)styryl-2,3, 4,5-tetraphenylsilole (**2**) in 28% isolated yield. Prolonging the reaction time was ineffective for the reaction. By replacing RhCl(PPh<sub>3</sub>)<sub>3</sub> with chloroplatinic acid and using toluene as solvent, the hydrosilylation proceeded very well. The reaction at 70 °C for 24 h produced **1** in yields of 77% and 84% at 110 °C within 24 h, respectively. Similarly, (E)-1-methyl-1-(4-bromo)styryl-2,3,4,5-tetraphenylsilole (**3**) was prepared in 67% yield by hydrosilylation reaction of **1** with 1-bromo-4-ethynylbenzene in toluene for 24 h at 110 °C with chloroplatinic acid as catalyst.

However, the reaction of 1 with 4-ethynylbenzoic acid under the same condition used for synthesis of 2 and 3 produced only (E)-1-methyl-1-(4-carboxyl) styryl-2,3,4,5-tetraphenylsilole (4) in 54% yield, together with (Z)-4-(1,2-bis(1-methyl-2,3,4,5-tetraphenyl-1H-silol-1-yl)vinyl)benzoic acid (5) in a yield of 8% as a by-product. When the reaction was carried out at 70 °C, a lower temperature, the product 4 with an increased yield (68%) was obtained after 24 h. Further decrease



Scheme 1. Synthetic routes to functionalized 2,3,4,5-tetraphenylsilole derivatives.



Scheme 2. Probable route of producing the by-product 5.



(a)



Figure 1. (a) The x-ray single structures of 2 cocrystallized with toluene and (b) the single structures of 4 (hydrogen atoms were omitted for clarity).

of the reaction temperature to  $50 \,^{\circ}$ C afforded only **4** in 34% yield. The reason for formation of **5** was suggested as follows: During the hydrosilylation reaction of *p*-ethynylbenzoic acid and 1-methyl-2,3,4,5-tetraphenylsilole, the polymerization of *p*-ethynylbenzoic acid also took place. A great deal of polymer was formed and precipitated from the reaction mixture at the refluxing temperature of toluene, resulting in the change of ratio between *p*-ethynylbenzoic acid and 1-methyl-2,3,4,5-tetraphenylsilole; thus, the excessive silole **1** reacted further with the formed **4** to produce the by-product through dehydrogenative silylation (Scheme 2). The single-crystal structure of the by-product **5** (see supporting information) supports this hypothesis.

Single crystals of compounds 2 and 4 for x-ray diffraction analysis were grown from toluene/dichloromethane/petroleum ether mixtures. The crystal structures of 2 and 4 are shown in Fig. 1, and their crystal data and analysis parameters are summarized in Table 1. As shown in Fig. 1, the hydrosilylation catalyzed by chloroplatinic acid is a *trans*-addition reaction. Both 2 and 4 crystallized in the triclinic system space group P-1. Four substituted phenyls attached on the silole ring show a typical propeller model.

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Parameter	2	4
Formula	$C_{37}H_{31}NSi \cdot C_7H_8$	$C_{38}H_{30}O_2Si$
Formula weight	609.85	546.71
Crystal dimensions (mm)	0.40  imes 0.40  imes 0.20	0.35  imes 0.25  imes 0.07
Crystal description	Plate	Plate
Crystal color	Yellow	Yellow
Crystal system	Triclinic	Triclinic
Space group	P-1	P-1
a (Å)	11.8995(14)	6.463(3)
b (Å)	12.4232(16)	10.547(5)
c (Å)	13.3886(16)	26.024(12)
a (deg)	91.821(8)	96.082(7)
β (deg)	106.264(8)	95.058(6)
γ (deg)	113.046(8)	99.256(4)
$V (Å)^3$	1725.8(4)	1730.9(13)
Z	2	2
$D_{calcd} (g  cm^{-3})$	1.174	1.049
F(000)	648	576
tem (K)	173(2)	173(2)
Wavelength (Å)	1.54186	0.71073
Absorption coefficient $(mm)^{-1}$	0.826	0.096
$2\theta_{\text{max}}$ , deg (completeness)	68.22 (97.8%)	27.48 (99.5%)
Collected reflections	20191	6092
Unique reflections (R <sub>int</sub> )	6190 (0.0428)	6092(0.0000)
Data/restraints/parameters	6190/0/418	6092/0/372
$R_1$ , w $R_2$ [obs $I > 2\sigma(I)$ ]	0.0676, 0.1671	0.0972, 0.2704
$\mathbf{R}_1$ , w $\mathbf{R}_2$ (all data)	0.0834, 0.1783	0.1078, 0.2786
Goodness of fit, F <sup>2</sup>	1.072	1.099
Largest diff. peak and hole, $(e Å^{-3})$	0.692/-0.494	0.320/-0.355

Table 1. Summary of crystal data and intensity collection parameters for 2 and 4

The molecular stacking of compounds 2 and 4 along the *a* axis are shown in Figs. 2(a) and 2(b), respectively. For silole 2,  $\pi - \pi$  stacking was blocked by the noncoplanarity of peripheral phenyls attached on the silole ring. However, N-H… $\pi$  interactions arising from amino groups were observed. The interplane angle between the plane of H-N-H and the neighboring phenyls ring that attached on the silole ring is 68.64°, and the distance from the center of these two hydrogen atoms to the center of the phenyl ring is 2.824 Å, which is shorter than the limit of interaction (3.05 Å).<sup>[19]</sup> The stacking pattern of carboxyl-containing silole 4 is shown in Fig. 2(b). Every two molecules form a couple through the intermolecular hydrogen bonds.



Figure 2. (a) The molecular stacking for 2 along a axis (toluene molecules and hydrogen atoms were omitted to prevent overcrowding). (b) The molecular stacking for 4 along a axis (hydrogen atoms were omitted).

#### SPECTRAL ANALYSIS

Ultraviolet (UV) absorption spectra and photoluminescence (PL) spectra of the functionalized siloles 2-4 in ethanol and ethanol/water mixture (1/9 v/v) were given in Fig. 3. UV spectra of each silole in ethanol/water mixtures are similar to that of its counterpart in ethanol solution. All siloles are strongly luminescent in ethanol/water mixtures but show no emission in ethanol solution. PL spectra experimentally illustrate that functionalized siloles also have the AIE feature. Compared with that of 1, the UV absorption maxima of the functionalized siloles shifted to longer wavelengths because of introduction of substituted styryl groups. However, these substituted groups have little influence on the PL spectra. All these functionalized siloles have emission maxima around 492 nm when excited at 365 nm.

UV-visible absorption and fluorescence emission spectra of these functionalized siloles in the solid state are shown in Fig. 4. The thin film was prepared by spin



**Figure 3.** (a) UV absorption spectra and (b) PL spectra of functionalized siloles in ethanol and ethanol/ water mixtures (1/9 v/v); concentrations of **1**, **2**, **3**, and **4** are 350  $\mu$ mol/L, 250  $\mu$ mol/L, 170  $\mu$ mol/L, and 160  $\mu$ mol/L, respectively.  $\lambda_{ex.} = 365$  nm.



Figure 4. (a) UV absorption spectra and (b) PL spectra of films of functionalized siloles on quartz plates, which were developed with THF solutions of 1, 2, 3, and 4.  $\lambda_{ex.} = 365$  nm.

coating on quartz plates from a silole solution in ethanol. The UV absorption spectra of the siloles in films are similar to those of their counterparts in ethanol/water mixtures, while the emission maxima of **2** and **4** are shifted to 467 nm, which is shorter by 25 nm than those of their counterpoints in solution. The same phenomenon of blue shift was also observed in crystal silole<sup>[20]</sup> as well as in the mixture of 40/60 acetone/water containing silole.<sup>[21,22]</sup> However, the emission maximum of **3** is at 487 nm and is only blue shifted 5 nm more than that of solution. The blue shift may be attributed to different intermolecular interactions in solid states. In ethanol/ water mixtures, the molecules of **2** and **4** aggregate loosely because of the hydrophobic interaction, but in the solid state they pack densely because of the N-H  $\cdots \pi$  interaction and hydrogen bonds, respectively. Therefore, the peak maxima of PL spectra of **2** and **4** appear at shorter wavelengths in the film state than in ethanol/ water mixtures. However, the main intermolecular force of **3** in solid state is merely van der Waals force, resulting in a very small blue shift.

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#### CONCLUSION

In conclusion, a series of functionalized siloles were synthesized in good yields by convenient hydrosilylation reaction of 1-methyl-2,3,4,5-tetraphenyl-1*H*-silole with corresponding *p*-ethynyl benzene derivatives. All new functionalized siloles are stable and show AIE character. The existence of the functional groups in siloles promises good potential to develop new silole-based functional materials for stimuli-responsive application.

### **EXPERIMENTAL**

Melting points were determined on a XT 5-2 microscope melting-point inspection apparatus, and the temperature was not calibrated. <sup>1</sup>H NMR and <sup>13</sup>C NMR spectra were measured on a Bruker Avance 400M spectrometer. High-resolution mass spectra (HRMS) were determined on a Water Micromass GCT mass spectrometer. High-resolution Fourier transform ion cyclotron resonance mass spectrometry (HR-FT-ICRMS) was performed on a Bruker Apex II FT-ICR mass spectrometer. The x-ray diffraction measurements for **2** and **5** were performed at 173 K with a Rigaku R-Axis Rapid diffractometer with a CuK $\alpha$  rotating anode generator (18 kW,  $\lambda = 1.54186$  Å), graphite monochromator, and an image plate area detector, while the measurement for **4** was performed at 173 K with a Rigaku Saturn 724 CCD diffractometer equipped with a sealed tube MoK $\alpha$  radiation source ( $\lambda = 0.71073$  Å) and graphite monochromator. UV absorption spectra were obtained on a Shimadzu UV-1601 spectrophotometer. PL spectra were performed on a Hitachi F-4500 fluorescence spectrophotometer.

Compound 1 was prepared according to the literature method.<sup>[23]</sup> Other chemical reagents were commercially available. Toluene was distilled from sodium benzophenone ketyl immediately prior to use. All other chemicals were used as received without further purification.

#### (E)-1-Methyl-1-(4-amino)styryl-2,3,4,5-tetraphenylsilole (2)

Compound 1 (0.24 g, 0.6 mmol), p-ethynylaniline (0.07 g, 0.6 mmol), H<sub>2</sub>PtCl<sub>6</sub>· H<sub>2</sub>O (20  $\mu$ L of 0.05 g·mL<sup>-1</sup> isopropanol solution,  $1.93 \times 10^{-9}$  mol), and toluene (20 mL) were mixed under an N<sub>2</sub> atmosphere and refluxed for 24 h. After cooling to room temperature, the mixture was concentrated under reduced pressure. The residue was subjected to column chromatography on a silica gel with petroleum ether/ethyl acetate mixture (3/1 v/v) as an eluent, followed by the further recrystallization from a hexane/Tetrahydrofuran (THF) mixture to give 0.259 g (0.5 mmol) of **2** in 84% yield as a yellow-green solid. Mp 80–82 °C; <sup>1</sup>H NMR (400 MHz, acetone-*d*<sub>6</sub>)  $\delta$  7.24 (d, 2H, *J* = 8), 7.10–7.00 (m, 17H), 6.89–6.86 (m, 4H), 6.62 (d, 2H, *J* = 8), 6.30 (d, 1H, *J* = 19), 4.90 (s, 2H), 0.61 (s, 3H). <sup>13</sup>C NMR (400 MHz, acetone-*d*<sub>6</sub>)  $\delta$  155.612, 150.352, 149.815, 141.731, 140,625, 140.028, 130.687, 129.752, 128.912, 128.694, 128.550, 128.318, 127.845, 127.122, 126.527, 126.435, 114.818, 114.693, -5.886. HRMS (EI): calcd. for C<sub>37</sub>H<sub>31</sub>SiN: 517.2226. Found: 517.2231.

#### (E)-1-Methyl-1-(4-bromo)styryl-2,3,4,5-tetraphenylsilole (3)

Compound 3 was synthesized in the same manner as described for 2 using p-ethynyl benzoyl bromine instead of p-ethynylaniline in 67% yield as a yellow-green

solid. Mp 186–188 °C; <sup>1</sup>H NMR (400 MHz, acetone- $d_6$ )  $\delta$  7.53 (d, 2H, J=8), 7.48 (d, 2H, J=8), 7.21(d, 1H, J=19), 7.15–7.00 (m, 16H), 6.89–6.88 (m, 4H), 6.80 (d, 1H, J=19), 0.66 (s, 3H). <sup>13</sup>C NMR (400 MHz, acetone- $d_6$ )  $\delta$  156.260, 147.755, 141.122, 140.382, 139.905, 132.584, 130.699, 129.789, 129.425, 128.810, 128.383, 127.275, 126.627, 124.202, -6.129. HRMS (EI): calcd. for C<sub>37</sub>H<sub>31</sub>Si<sup>79</sup>Br: 580.1222. Found: 580.1230. calcd. for C<sub>37</sub>H<sub>31</sub>Si<sup>81</sup>Br: 582.1201. Found: 582.1211.

#### (E)-1-Methyl-1-(4-carboxyl)styryl-2,3,4,5-tetraphenylsilole (4)

Compound **4** was synthesized in a manner similar to that described for **2** by the reaction of p-ethynylbenzoic acid and **1** at 70 °C in 68% yield as a yellow-green solid. Mp 234–236 °C; <sup>1</sup>H NMR (400 MHz, DMSO- $d_6$ )  $\delta$  13.0 (s, 1H), 7.90 (d, 2H, J=8), 7.66 (d, 2H, J=8), 7.23 (d, 1H, J=19), 7.12–7.00 (m, 12H), 6.93–6.91 (m, 4.5H), 6.87–6.84 (m, 4.5H), 0.65 (s, 3H). <sup>13</sup>C NMR (300 MHz, acetone- $d_6$ )  $\delta$  166.953, 154.995, 146.995, 141.183, 139.735, 138.519, 130.743, 129.761, 129.420, 128.073, 127.617, 126.846, 126.533, 125.852, 125.267, -6.483. HR-FT-ICRMS m/z calcd. for C<sub>38</sub>H<sub>29</sub>O<sub>2</sub>Si: 545.1942 [M – H]<sup>-</sup>, Found: 545.1939.

#### Supplementary Material

CCDC 792769 (compound 2), 792768 (compound 4), and 792770 (compound 5) contain the supplementary crystallographic data for this article. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html

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