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Silylene-spaced diphenylanthracene derivatives as blue-emitting materials

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Abstract

A novel series of blue emitting silylene-spaced diphenylanthracene derivatives have been synthesized and characterized. The rhodium-catalyzed hydrosilylation of bis[4-(dimethylsilyl)phenyl]anthracene **3**–**4** yielded stable 9,10-disubstituted (*E*)-divinylsilylene–diphenylanthracene products **7**–**10** and salt elimination reaction of bis[4-(chlorodimethylsilyl)phenyl]anthracene **5**–**6** gave 9,10-disubstituted disilyldiphenylanthracene compounds **11**–**14**. They are fluorescent in the blue region with good quantum efficiencies. The rhodium-catalyzed polyaddition including 2-*tert*-butyl-9,10-bis[4-(dimethylsilyl)phenyl]anthracene (**4**) afforded the nonconjugated copolymer **15**.
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Keywords: Electroluminescent; Silylene-spaced diphenylanthracene; Rhodium-catalyzed hydrosilylation

1. Introduction

Organic light-emitting devices (OLEDs) have attracted great attention in material science since the initial work was first reported in 1987 by Tang and Vanslyke because of their potential applications in full-color flat-panel displays [1]. For high-performance full-color displays, the development of three elemental colors of red-, green-, and blue-emitting materials with good color purity and high efficiency is necessary. After two decades of active research, a number of red, green, and blue emitters have been reported both in small molecular and polymer systems [2]. As compared to green OLEDs, blue and red OLEDs have to be improved in terms of color purity and efficiency [3]. Meanwhile, blue-emitting materials have still received considerable attention not only as a blue light source but also as a host for downhill energy transfer to green- or red-emitting materials [4]. Diphenylanthracene derivatives, in particular, have been often used as blue emitter in OLEDs, because they have shown to have good physical properties, such as maximum luminescence, power efficiency, current efficiency, and external quantum efficiency [5]. It is known that diphenylanthracene has high photoluminescence quantum yield, good electrochemical property and orthogonal structure between anthracene core and substituted peripheral phenyl ring [6]. Therefore, the devices containing diphenylanthracene as an emitter are expected to show higher electroluminescence efficiency, long lifetime, and deep blue color. Because of these advantages, diphenylanthracene became our blue luminophore of choice.

On the other hand, the introduction of a flexible spacer such as $SiMe_2$ between well-defined chromophores in a mononuclear compound [7] or a polymer chain [8] is current topic in luminescent materials. One advantage is that it facilitates the intramolecular photoinduced charge

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transfer process [9]. The other is that the silicon-based tetrahedral spacers could interrupt the π-conjugated chains, resulting in tuning the color of emitting materials by controlling conjugation length [8]. Recently, Luh et al. [10] reported that the intrachain chromophore interaction due to folding of copolymer has been observed in silylene–divinylene copolymers. We also reported various silylene-spaced divinyl- and trivinylarene fluorescent dyes [11]. To our knowledge, the introduction of $SiMe_2$ flexible spacer into diphenylanthracene derivatives has not reported yet. Only a few papers described 9,10-disilylanthracenes and their synthesis, structure, optical and electronic properties [12]. Especially, we chose pyrene and 9-phenylcarbazole units as energy matching substituents so that their absorption maxima can overlap with the emission of diphenylanthracene for facilitating intramolecular energy transfer [10b]. Moreover, we expected that the designed compound, in which carbazole moiety as a pendent group, which let the fabrication of OLEDs to be simplified by dual function (emitting and hole-transporting). A *tert*-butyl group is incorporated in the anthracene moiety not only to minimize aggregation of planar anthracene segments but also improve the solubility of the compounds in common solvents [4].

In this paper, we describe: (i) the synthesis and characterization of a blue emitting silylene-spaced diphenylanthracene derivatives; (ii) the optical properties of novel bis{[(aryl)dimethylsilyl]phenyl}-anthracene; and (iii) the electroluminescent properties of copolymer containing bis[(dimethylsilyl)phenyl]anthracene and divinylbenzene.

2. Results and discussion

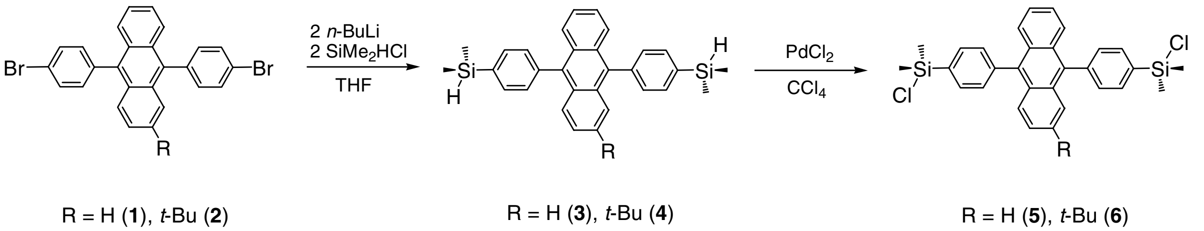
2.1. Synthesis

Scheme 1 illustrates the synthetic procedures for the preparation of bis[4-(di-methylsilyl)phenyl]anthracene **3**–**4** and bis[4-(chlorodimethylsilyl)phenyl]anthracene **5**–**6** derivatives. Dibromodiphenylanthracene starting materials **1**–**2** were synthesized by already known reduction procedure developed by Smet et al. [13] from anthraquinone and 2-*tert*-butylanthraquinone, respectively [4]. Thus, lithiation of dibromoanthracene with *n*-BuLi at −78 °C in THF followed by treatment with chlorodimethylsilane gave stable bis[4-(dimethylsilyl)phenyl]anthracene **3**–**4** in high yields. Compounds **3**–**4** are soluble in toluene and THF. The ^1H, ^{13}C, and ^{29}Si NMR spectra, and mass spectroscopy for **3**–**4** support the proposed structures. The ^1H NMR spectrum of **3** reveals the formation of silane viewed from the septet of Si–H of 4.59 ppm and doublet of $SiMe_2$ at 0.49 ppm ($^3J_{HH} = 3.9$ Hz). The ^{29}Si NMR spectrum of **3** shows a singlet at −23.5 ppm. This value is comparable to that observed for the 9,10-disilylanthracene [14]. Hydrogen–chlorine exchange of the disilyldiphenylanthracenes in the presence of a catalytic amount of palladium dichloride in carbon tetrachloride was accomplished by the similar method reported by Ishikawa and co-workers [15]. These exchange reaction gave moisture sensitive yellow compounds **5**–**6** in quantitative yield (Scheme 1). The chlorinated products **5**–**6** were readily isolated by extraction with toluene. But the hydrolytic lability of the Si–Cl bond precluded their isolation using chromatography.

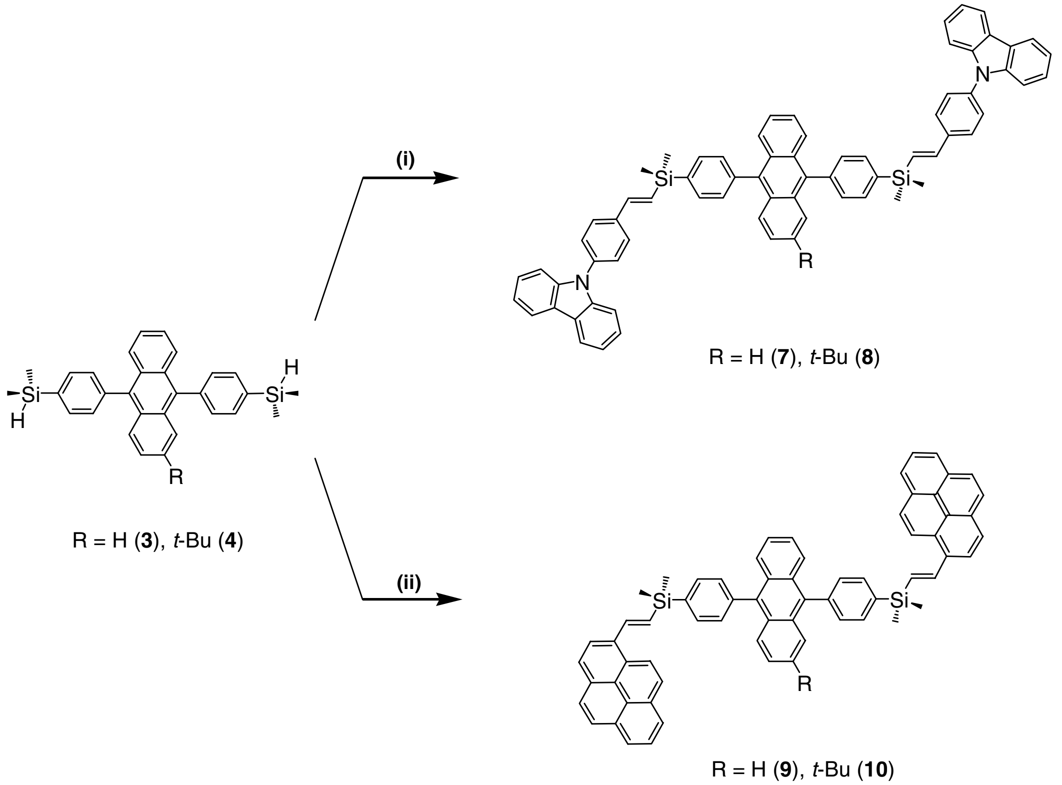
Spectroscopic data for **5**–**6** are completely consistent with their proposed structures. A parent ion in the mass spectrum of **5** was observed at *m*/*z* 514. The ^{29}Si NMR spectra of **5**–**6** exhibited one resonance at 15.2 ppm, significantly downfield shifted than those of their silane compounds **3**–**4**.

Many metal complexes were found to be efficient catalysts for the stereo-selective hydrosilylation of ethynyl derivatives. Among the metal catalysts, the rhodium catalyst exhibited the highest activity, completing the hydrosilylation under mild condition. The Rh-catalyzed hydrosilylation of hydrosilanes **3**–**4** with 1-ethynylpyrene and 1-carbazolyl-4-ethynylbenzene afforded the 9,10-disubstituted (*E*)-divinylsilyldiphenylanthracene products **7**–**10** in moderate yields (Scheme 2). These efficient regio- and stereo-selective polyaddition methods were developed by Mori et al. [16].

The *trans* configuration was readily assigned by the characteristic signals of the ^1H NMR spectra at 7.2–6.7 ppm with a large coupling constant of the vinyl group ($J = 18.9$–19.2 Hz). The absence of peaks at about 6.5 ppm with small coupling constant ($J \approx 15$ Hz) indicates that no *cis*-vinylene bonds were formed. The ^{29}Si NMR spectra of **7**–**10** showed one resonance at −15.2 to −16.6 ppm arising from the equivalent silicon atoms. Other synthetic utility of the hydrosilylation products was demonstrated by transformation of the representative products **5**–**6**. Treatment of chlorosilanes **5**–**6** with 1-carbazolyl-4-lithiobenzene and 1-lithiopyrene in THF afforded the 9,10-disubstituted disilyldiphenylanthracene compounds **11**–**14** which were stable enough to be isolated by silica gel chromatography (Scheme 3).



Scheme 1.

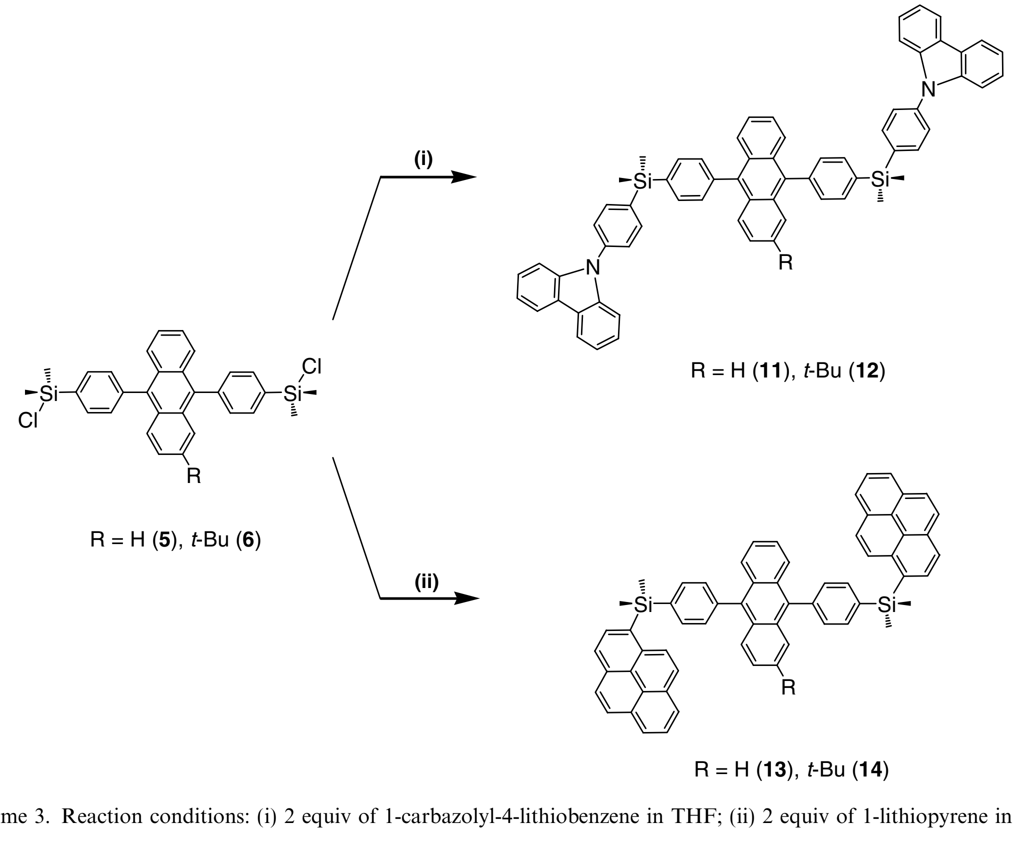


R = H (**3**), *t*-Bu (**4**)

R = H (**7**), *t*-Bu (**8**)

R = H (**9**), *t*-Bu (**10**)

Scheme 2. Reaction conditions: (i) 2 equiv of 1-carbazolyl-4-ethynylbenzene, $RhCl(PPh_3)_3$, NaI, THF, reflux, 12 h; (ii) 2 equiv of 1-ethynylpyrene, $RhCl(PPh_3)_3$, NaI, THF, reflux, 12 h.



R = H (**5**), *t*-Bu (**6**)

R = H (**11**), *t*-Bu (**12**)

R = H (**13**), *t*-Bu (**14**)

Scheme 3. Reaction conditions: (i) 2 equiv of 1-carbazolyl-4-lithiobenzene in THF; (ii) 2 equiv of 1-lithiopyrene in THF.

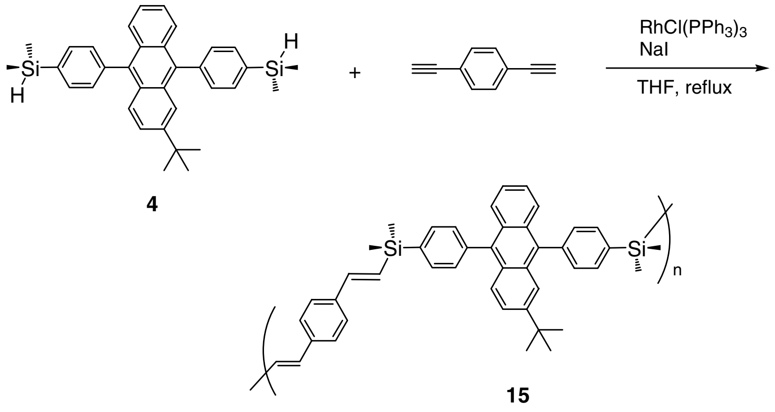
Incorporation of a *tert*-butyl group in the anthracene moiety dramatically improved the solubility of the compounds in common solvents. These new fluorophores have been fully characterized by NMR, mass, and elemental analysis. The ^{13}C NMR spectrum of **11** showed expected 18 resonances at 140.9–110.2 ppm due to the aromatic carbons. The ^{29}Si NMR spectrum of **11** showed one resonance at −10.7 ppm arising from the equivalent silicon atoms. The new fluorophore **11** also exhibits a molecular ion peak at m/z 928 in the mass spectrum.

We extended the hydrosilylation methodology to the reaction of 2-*tert*-butyl-9,10-bis[4-(dimethylsilyl)phenyl]-anthracene (**4**)to obtain copolymer. As expected, the rhodium-catalyzed hydrosilylation of **4** with 1,4-diethynylbenzene yielded copolymer **15** in 88% yield (Scheme 4). The purification process was that the polymer was dissolved in small amount of chloroform and precipitated in excess methanol several times.

The molecular weight (M_w) and the polydispersity of the polymer, determined by gel permeation chromatography (GPC) using THF as eluent and polystyrene as calibration standard, was in the range 200000 g/mol and 1.29, respectively. The polymer was soluble in common solvents such as THF, $CHCl_3$, and benzene. The ^{1}H NMR spectrum of **15** reveals characteristic two doublets at 7.06 and 6.74 ppm with a large coupling constant ($J = 18.6$ Hz) due to an inequivalent vinylic hydrogens, while the copolymer **15** shows no resonance attributed to the Si–H. The ^{29}Si NMR spectrum of **15** reveals a singlet resonance at −15.8 ppm, in accord with the upfield shift for the silylene-vinylene-bridge system which appears in the region from −12 to −30 ppm [17]. Such silylene-spaced vinylarene copolymers were recently reported by Mori et al. [16a] and Neckers and co-workers [18].

2.2. Optical properties

The UV–Vis absorption and photoluminescence spectra of all new compounds were obtained in CH_2Cl_2, with the λ_{max} values collected in Table 1. The absorption spectra and emission spectra of selective compounds **3**, **7**, **11**, **15**, and diphenylanthracene (**dpa**) are shown in Figs. 1 and 2, respectively. Intense absorption bands with a fine structure appear in the region of 250–420 nm. Three absorption maxima peaks at ca. 350–400 nm indicate the characteristic vibronic pattern attributed to the π → π* electronic transitions of diphenylanthracene and all the values are similar to that of diphenylanthracene. The absorption maxima of



Scheme 4.

Table 1
Physical data for the compounds **7**–**15** and **dpa**

Compound	UV[a] λ_{max} (nm)	PL[a] λ_{max} (nm)	PL in film λ_{max} (nm)	Φ_f[b]	HOMO[c] (eV)	LUMO (eV)	E_g[d] (eV)
7	328, 342, 358, 376, 397	419, 436	430	0.88	5.83	2.84	2.99
8	294, 322, 358, 377, 399	420, 433	429	0.90	5.85	2.88	2.97
9	290, 332, 357, 377, 398	429	515	0.87	5.81	2.89	2.92
10	299, 328, 357, 376, 398	427	502	0.92	5.80	2.87	2.93
11	293, 325, 358, 377, 397	420, 435	434	0.94	5.82	2.87	2.95
12	295, 324, 358, 376, 397	428	432	0.93	5.83	2.87	2.96
13	297, 338, 357, 376, 398	434	510	0.92	5.80	2.87	2.93
14	299, 335, 357, 377, 397	429	497	0.95	5.81	2.87	2.94
15	302, 374	430, 445	468	0.88	5.40	2.44	2.96
dpa	356, 375, 395	411, 432		0.95			

[a] Measured in CH_2Cl_2 solution.
[b] Determined by the dilution method.
[c] Determined using a photoelectron spectrometer except compound **15** (the HOMO level of compound **15** was determined by CV data).
[d] Calculated by optical band edge.

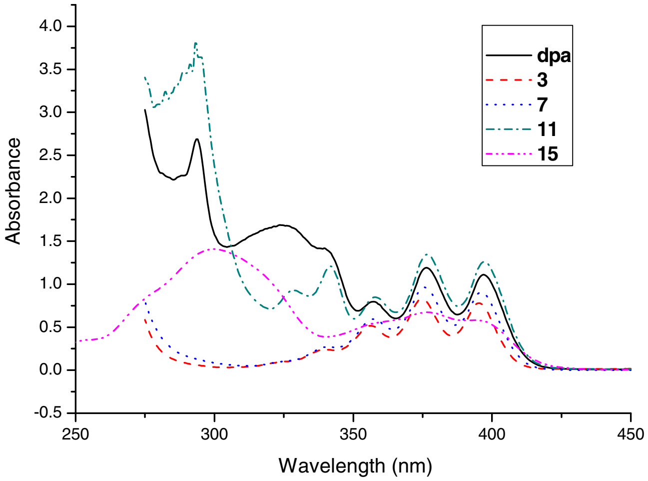


Fig. 1. The absorption spectra of **dpa**, **3**, **7**, **11**, and **15** in CH_2Cl_2.

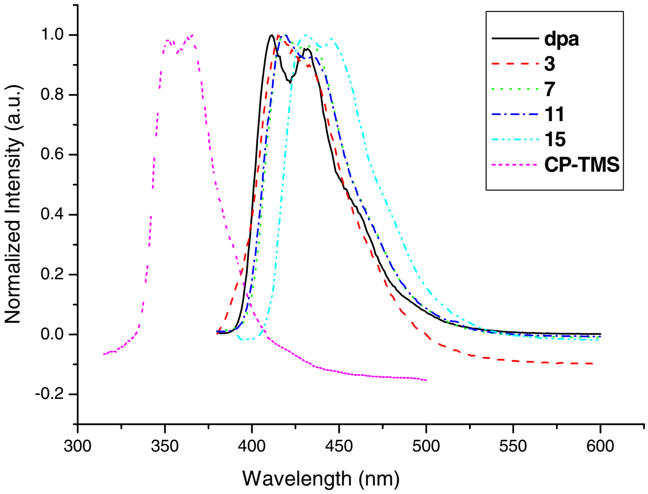


Fig. 2. The emission spectra of **dpa**, **3**, **7**, **11**, **15** and **CP-TMS** in CH_2Cl_2.

the longest wavelength are somewhat red-shifted (2–4 nm) compared to that of diphenylanthracene.

Compounds **7**–**14** exhibit intense fluorescence emission in the blue wavelength region in dichloromethane solution and the emission maxima are summarized in Table 1. The similar values of λ_{em} maxima to diphenylanthracene which are in the range 420–430 nm indicate that the π-conjugation was effectively interrupted by the silicon spacer. The same emission spectra are obtained irrespective of the applied excited wavelengths, indicating that downhill relaxation to the lowest excited state is efficient [17]. There are small hypsochromic shifts of compounds (**8**, **10**, **12**, and **14**) incorporated *tert*-butyl in the anthracene moiety with respect to their analogues because of minimizing aggregation of planar anthracene segments. No residual emission spectra of pyrene [19] and 9-phenylcarbazole [20] substituents in the range of 350–400 nm, prove that their emission maxima overlap with the absorption of diphenylanthracene pursuing intramolecular energy transfer. In the case of solid PL, the compounds with 9-phenylcarbazole substituents showed coincident emission wavelength with solution PL. However, the compounds with pyrene substituents were extremely bathochromic shifted (∼70 nm) in solid state resulted in strong π–π stacking ability. The maximum for the solution and solid state photoluminescence of the polymer **15** appear at 430 and 468 nm, respectively. The red shift from that of the solution can be attributed either to the difference in the energy transfer processes between the film and the solution due to the presence of rotation of the chromophore, or to the effect of packing and local geometry of the polymers.

The PL quantum yield is relatively high, which is comparable to those of diphenylanthracene ($\Phi_f = 0.95$) or 9,10-disilylanthracenes ($\Phi_f = 0.90$–0.97) reported by Matsumoto et al. [12a]. The highest occupied molecular orbital (HOMO) and lowest unoccupied molecular orbital (LUMO) of these silylene-spaced diphenylanthracene derivatives **7**–**14** are listed in Table 1. The HOMO was determined using a ultraviolet photoelectron spectrometer and LUMO was calculated by the optical band gap estimated from the onset wavelength (the lowest-energy absorption edge) of the UV–Vis absorption spectrum ($E_g = 1240\ \text{nm}/\lambda_{onset}$) [21]. The HOMO and LUMO energy of these products are at ca. 5.80–5.85 and 2.84–2.89 eV, respectively.

2.3. Electrochemistry

As shown in Fig. 3, the electrochemical characteristic of polymer **15** was investigated by the cyclic voltammetry (CV) method which was measured in THF containing 0.1 M tetrabutylammonium perchlorate (TBAP). Cyclic voltammetry was employed to evaluate the ionization potentials (i.e., hole-injection ability) and the redox stability of our copolymer. The CV curve was referenced to an Ag/AgCl reference electrode. The first oxidation potential was used to determine HOMO energy level and used to obtain LUMO energy level with the absorption band edge

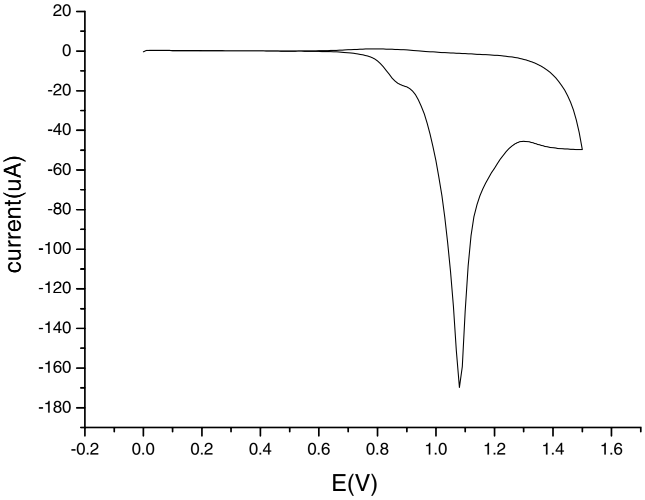


Fig. 3. CV characteristics of polymer **15**.

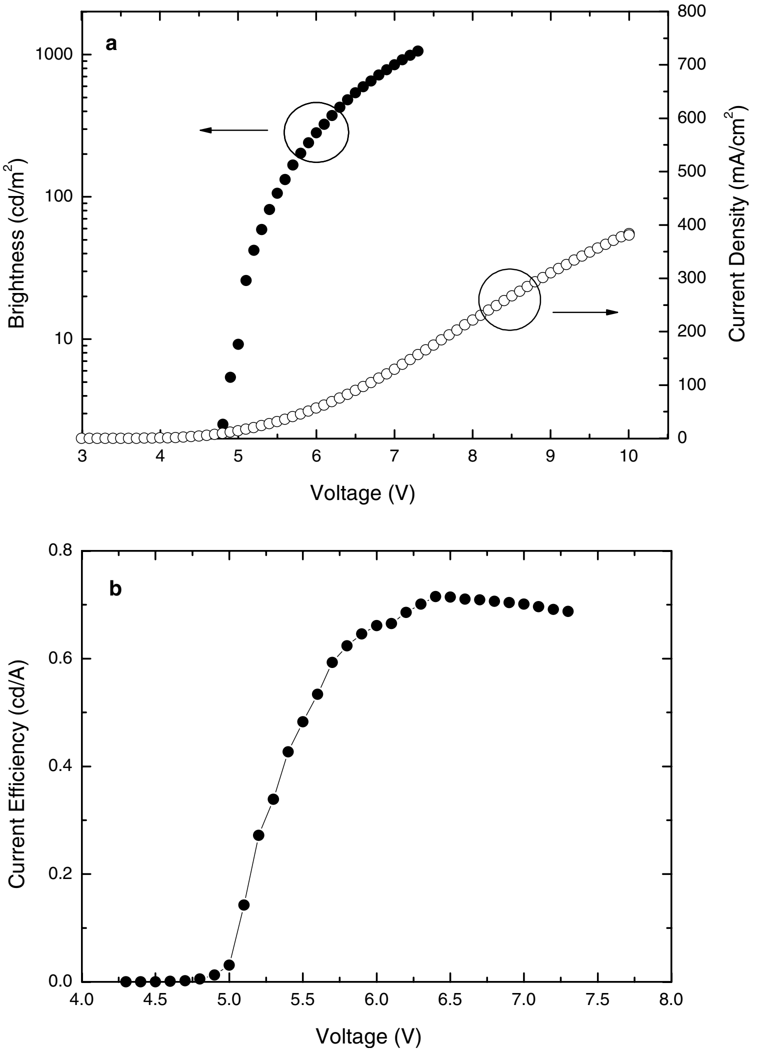


Fig. 4. (a) Current density and luminescence (brightness) vs. voltage and (b) current efficiency vs. voltage characteristics of the EL device with polymer **15**.

from UV–Vis absorption spectra. These energy levels of polymer **15** are shown in Table 1.

2.4. Electroluminescent device

The electroluminescence (EL) device fabricated through vapor-deposition using the small molecular diphenylanthracene derivatives **7**–**14** showed the recrystallization during the device operation, which is a typical problem of an organic molecular EL device. The device with diphenylanthracene was also known to have poor film-forming property [22]. Therefore, a EL device based on a polymer containing diphenylanthracene is expected to have good device properties. The devices were prepared in configuration of ITO/PEDOT (65 nm)/polymer **15** (50 nm)/BaF_2 (2 nm)/Ca (50 nm)/Al (300 nm). Comparing the relative HOMO (5.4 eV) and LUMO (2.4 eV) energy levels of the emitting polymer **15** with other layer sequences, the energy levels seem to be appropriate to show good EL characteristics. The EL characteristics of the present OLED are shown in Fig. 4(a) and (b) for current density and luminescence verse voltage (*I*–*V*–*L*) and current efficiency verse voltage, respectively. Fig. 4(a) shows that the device with polymer **15** as an emitting layer exhibits moderate turn-on voltage of 4.7 V (defined as 1 cd/m^2) and maximum brightness of about 1000 cd/m^2. The maximum current efficiency of device is about 0.7 cd/A from Fig. 4(b).

In conclusion, we were easily able to introduce a flexible silylene-space into blue emitting diphenylanthracene derivatives using the rhodium catalyzed hydrosilylation and salt elimination reaction. These novel compounds **3**–**15** exhibit intense fluorescence emission in the blue wavelength region. Electroluminescent devices based on copolymer **15** was fabricated and showed good EL characteristics. Further study with **4** as blue emitting copolymer including the rigid segment will be explored. Device optimizations are under investigation.

3. Experimental

All experiments were performed under a nitrogen atmosphere in a vacuum atmosphere drybox or by standard schlenk techniques. THF and ether were distilled from sodium benzophenone and carbon tetrachloride was distilled from P_2O_5. ^{1}H, ^{13}C NMR and ^{29}Si NMR spectra were recorded on a Varian Mercury 300 spectrometer. The absorption and photoluminescence spectra were recorded on a Perkin–Elmer Lambda 2S UV–Vis spectrophotometer and a Perkin–Elmer LS fluorescence spectrometer, respectively. The fluorescence quantum yields of compounds **7**–**15** were determined relative to 9,10-diphenylanthracene in CH_2Cl_2 at 298 K ($\Phi_f = 0.95$) by the dilution method [23]. The quantum yields were calculated using previously reported procedures [24]. Ultraviolet photoelectron spectroscopy (UPS) was measured by means of an AC-2 machine. All starting materials were purchased from either Aldrich or Strem and used without further purification. 1,4-Diethynylbenzene [25], 1-ethynylpyrene [26], 1-carbazolyl-4-bromobenzene [27], 1-carbazolyl-4-ethynylbenzene [28], $RhCl(PPh_3)_3$ [29], 9,10-bis(4-bromo-phenyl)anthracene (**1**), and 2-*tert*-butyl-9,10-bis(4-bromophenyl)anthracene (**2**) [4] were prepared according to the literature.

3.1. 9,10-Bis[4-(dimethylsilyl)phenyl]anthracene (3)

To a stirred THF solution (30 mL) of 9,10-dibromodiphenylanthracene, **1** (1.65 g, 3.38 mmol) was added a solution of *n*-BuLi (5.0 mL, 1.6 M in hexane) at −78 °C and stirred for 30 min at that temperature. To this solution was added chlorodimethylsilane (0.9 mL, 8.11 mmol) at −78 °C. The reaction mixture was slowly warmed to ambient temperature and stirred for 12 h. The solvent was removed under reduced pressure. H_2O and CH_2Cl_2 were added to the resulting mixture. Then the product was extracted with CH_2Cl_2 (20 mL). After evaporation, the compound **3** was isolated as a white solid in 92% yield. M.p.: 91–93 °C. ^{1}H NMR ($CDCl_3$): δ 7.78 (d, 4H, $J = 7.2$ Hz, *Ph*), 7.70 (dd, 4H, $J = 6.9$ Hz, $J = 3.3$ Hz, *anth*), 7.48 (d, 4H, $J = 7.2$ Hz, *Ph*), 7.32 (dd, 4H, $J = 6.9$ Hz, $J = 3.3$ Hz, *anth*), 4.59 (sept, 2H, $J = 3.9$ Hz, Si–*H*), 0.49 (d, 12H, $J = 3.9$ Hz, Si–C*H*$_3$). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 140.1, 136.8, 134.2, 130.9, 129.9, 127.2, 125.5, 124.8, −3.5. ^{29}Si{^{1}H} NMR ($CDCl_3$): δ −23.5. MS: *m*/*z* 446 [M^+]. Anal. Calc. for $C_{30}H_{30}Si_2$: C, 80.66; H, 6.77. Found: C, 80.48; H, 6.62%.

3.2. 2-tert-Butyl-9,10-bis[4-(dimethylsilyl)phenyl]-anthracene (4)

Compound **4** was prepared using the same procedure as described for **3** in 87% yield. M.p.: 85 °C. ^{1}H NMR ($CDCl_3$): δ 7.80–7.77 (m, 4H, *Ph*), 7.72–7.62 (m, 4H, *anth*), 7.51–7.44 (m, 5H, *Ph* and *anth*), 7.33–7.30 (m, 2H, *anth*), 4.61 (m, 2H, Si–*H*), 1.27 (s, 9H, *t-Bu*), 0.50 (m, 12H, Si–C*H*$_3$). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 147.3, 140.2, 139.3, 136.9, 136.3, 134.2, 131.7, 130.9, 129.9, 129.6, 128.5, 127.2, 126.7, 125.3, 124.5, 121.6, 118.6, 117.8, 35.0, 31.0, −3.6. ^{29}Si{^{1}H} NMR ($CDCl_3$): δ −23.3. MS: *m*/*z* 502 [M^+]. Anal. Calc. for $C_{34}H_{38}Si_2$: C, 81.21; H, 7.62. Found: C, 81.01; H, 7.51%.

3.3. 9,10-Bis[4-(chlorodimethylsilyl)phenyl]anthracene (5)

To a shlenk flask charged with 9,10-bis[4-(dimethylsilyl)phenyl]anthracene (**3**) (0.55 g, 1.23 mmol) and a catalytic amount of $PdCl_2$ (0.011 g, 0.062 mmol) was added distilled carbon tetrachloride (30 mL). The mixture was refluxed for 1 h under an Ar atmosphere. After cooling, the solution was evaporated and the toluene was added. The suspension was formed. The solution was filtered through well-dried celite pad and drying under reduced pressure afforded the moisture sensitive pale yellow solid in 81% yield. ^{1}H NMR (C_6D_6): δ 7.82 (d, 4H, $J = 6.9$ Hz, *Ph*), 7.68 (dd, 4H, $J = 7.2$, $J = 3.3$ Hz, *anth*), 7.43 (d, 4H, $J = 6.9$ Hz, *Ph*), 7.11 (dd, 4H, $J = 7.2$ Hz, $J = 3.3$ Hz, *anth*), 0.56 (s, 12H, Si–C*H*$_3$). ^{13}C{^{1}H} NMR (C_6D_6): δ 141.8, 137.3, 135.6, 133.7, 131.4, 130.4, 128.9, 126.4, 2.1. ^{29}Si{^{1}H} NMR ($CDCl_3$): δ 15.2. MS: *m*/*z* 514 [M^+]. Anal. Calc. for $C_{30}H_{28}Cl_2Si_2$: C, 69.88; H, 5.47. Found: C, 69.65; H, 5.35%.

3.4. 2-tert-Butyl-9,10-bis[4-(chlorodimethylsilyl)phenyl]-anthracene (6)

Compound **6** was prepared using the same procedure as described for **5** in 77% yield. ^{1}H NMR ($CDCl_3$): δ 7.87–7.85 (m, 4H, *Ph*), 7.66–7.43 (m, 9H, *Ph* and *anth*), 7.32-7.29 (m, 2H, *anth*), 1.26 (s, 9H, *t-Bu*), 0.83 (s, 12H, Si–C*H*$_3$). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 147.3, 141.5, 140.5, 138.8, 137.0, 136.4, 135.3, 133.2, 130.9, 130.1, 129.9, 128.5, 127.9, 126.1, 124.9, 122.4, 35.0, 31.0, 1.8. ^{29}Si{^{1}H} NMR ($CDCl_3$): δ 15.1. MS: *m*/*z* 570 [M^+]. Anal. Calc. for $C_{34}H_{36}Cl_2Si_2$: C, 71.43; H, 6.35. Found: C, 71.25; H, 6.22%.

3.5. 9,10-Bis{[4-(4-carbazolyl)phenylvinyldimethylsilyl]-phenyl}anthracene (7)

To a mixture of 9,10-bis[4-(dimethylsilyl)phenyl]anthracene (**3**) (0.45 g, 1.0 mmol), 1-carbazolyl-4-ethynylbenzene (0.59 g, 2.2 mmol), $RhCl(PPh_3)_3$ (0.002 g, 0.1 mol%), and NaI (0.015 g, 5 mol%) was added THF (25 mL). The mixture was refluxed for 12 h under inert atmosphere. The solution was dried in vacuo. Pure product **7** was isolated by silica gel chromatography (eluent: methylene chloride/hexane 1:3) as a white solid in 61% yield. M.p.: 221–223 °C. ^{1}H NMR ($CDCl_3$): δ 8.16 (d, 4H, $J = 7.5$ Hz, *Ph*), 7.85 (d, 4H, $J = 7.5$ Hz, *Ph*), 7.79–7.72 (m, 8H, *Ph* and *anth*), 7.59 (d, 4H, $J = 7.5$ Hz, *Ph*), 7.53 (d, 4H, $J = 7.5$ Hz, *Ph*), 7.44–7.26 (m, 16H, *Ph* and *anth*), 7.20 (d, 2H, $J = 19.2$ Hz, Ph–C*H*=), 6.74 (d, 2H, $J = 19.2$ Hz, Si–C*H*=), 0.63 (s, 12H, Si–C*H*$_3$). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 140.9, 140.0, 137.6, 137.2, 134.0, 131.1, 130.7, 129.9, 128.1, 127.1, 126.4, 125.8, 125.5, 124.8, 123.6, 120.5, 120.3, 119.7, 110.9, 109.6, −2.2. ^{29}Si{^{1}H} NMR ($CDCl_3$):

δ −15.2. MS: *m/z* 980 [M$^+$]. Anal. Calc. for $C_{70}H_{56}N_2Si_2$: C, 85.67; H, 5.75. Found: C, 85.42; H, 5.61%.

*3.6. 2-tert-Butyl-9,10-bis{[4-(4-carbazolyl)phenylvinyldimethylsilyl]phenyl}anthracene (**8**)*

Compound **8** was prepared using the same procedure as described for **7** except 2-*tert*-butyl-9,10-bis[4-(dimethylsilyl)phenyl]anthracene (**4**) was used instead of 9,10-bis[4-(dimethylsilyl)phenyl]anthracene (**3**). The pure product **8** was isolated by silica gel chromatography (eluent: methylene chloride/hexane 1:3) as a pale yellow solid in 65% yield. M.p.: 194–195 °C. ^{1}H NMR ($CDCl_3$): δ 8.15 (d, 4H, $J = 7.5$ Hz, *Ph*), 7.92–7.28 (m, 35H, *Ph* and *anth*), 7.19 (d, 2H, $J = 19.2$ Hz, Ph–C*H*=), 6.84 (d, 2H, $J = 19.2$ Hz, Si–C*H*=), 1.26 (s, 9H, *t-Bu*), 0.62 (s, 12H, Si–C*H*$_3$). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 148.1, 147.2, 140.7, 137.3, 136.9, 134.0, 131.1, 130.0, 129.6, 128.6, 127.4, 127.0, 126.4, 126.0, 125.1, 123.7, 122.8, 121.6, 120.8, 120.5, 118.8, 118.2, 116.4, 114.8, 112.6, 110.3, 35.1, 29.8, −2.0. ^{29}Si{^{1}H} NMR ($CDCl_3$): δ −15.6. MS: *m/z* 1036 [M$^+$]. Anal. Calc. for $C_{74}H_{64}N_2Si_2$: C, 85.67; H, 6.22. Found: C, 85.42; H, 6.08%.

*3.7. 9,10-Bis{[4-(pyrenyl)vinyldimethylsilyl]phenyl}anthracene (**9**)*

Compound **9** was prepared using the same procedure as described for **7** except 1-ethynylpyrene was used instead of 1-carbazolyl-4-ethynylbenzene. Pure **9** was isolated by silica gel chromatography (eluent: methylene chloride/hexane 1:3) as a white solid in 70% yield. M.p.: 237–238 °C. ^{1}H NMR ($CDCl_3$): δ 8.47 (d, 2H, $J = 9.3$ Hz, *pyr*), 8.37 (d, 2H, $J = 8.1$ Hz, *pyr*), 8.25–8.00 (m, 16H, *pyr*), 7.93 (d, 4H, $J = 7.8$ Hz, *Ph*), 7.78 (dd, 4H, $J = 6.9$ Hz, $J = 3.3$ Hz, *anth*), 7.57 (d, 4H, $J = 7.8$ Hz, *Ph*), 7.36 (dd, 4H, $J = 6.9$ Hz, $J = 3.3$ Hz, *anth*), 7.18 (d, 2H, $J = 18.9$ Hz, Ph–C*H*=), 7.84 (d, 2H, $J = 18.9$ Hz, Si–C*H*=), 0.72 (s, 12H, Si–C*H*$_3$). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 142.4, 140.1, 137.7, 137.2, 134.3, 133.0, 132.2, 131.6, 131.4, 131.3, 131.0, 130.0, 128.4, 128.2, 127.7, 127.5, 127.2, 126.1, 125.5, 125.3, 125.1, 124.7, 123.0, 122.2, 120.6, 118.4, 117.2, 112.6, −2.8. ^{29}Si{^{1}H} NMR ($CDCl_3$): δ −16.3. MS: *m/z* 898 [M$^+$]. Anal. Calc. for $C_{66}H_{50}Si_2$: C, 88.15; H, 5.60. Found: C, 88.02; H, 5.51%.

*3.8. 2-tert-Butyl-9,10-bis{[4-(pyrenyl)vinyldimethylsilyl]phenyl}anthracene (**10**)*

Compound **10** was prepared using the same procedure as described for **7** except 1-ethynylpyrene, and 2-*tert*-butyl-9,10-bis[4-(dimethylsilyl)phenyl]anthracene (**4**) were used instead of 1-carbazolyl-4-ethynylbenzene, and 9,10-bis[4-(dimethylsilyl)phenyl]anthracene (**3**), respectively. Pure **10** was isolated by silica gel chromatography (eluent: methylene chloride/hexane 1:3) as a pale yellow solid in 72% yield. M.p.: 222–225 °C. ^{1}H NMR ($CDCl_3$): δ 8.47 (d, 2H, $J = 9.0$ Hz, *pyr*), 8.36 (d, 2H, $J = 7.8$ Hz, *pyr*), 8.20–7.91 (m, 20H, *pyr*), 7.77 (dd, 4H, $J = 6.9$ Hz, $J = 3.3$ Hz, *anth*), 7.56 (d, 4H, $J = 7.8$ Hz, *Ph*), 7.34 (dd, 4H, $J = 6.9$ Hz, $J = 3.3$ Hz, *anth*), 7.18 (d, 2H, $J = 18.9$ Hz, Ph–C*H*=), 6.78 (d, 2H, $J = 18.9$ Hz, Si–C*H*=), 1.27 (s, 9H, *t-Bu*), 0.73 (s, 12H, Si–C*H*$_3$). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 151.0, 142.7, 140.7, 140.2, 139.6, 138.8, 137.5, 134.1, 134.0, 132.9, 132.0, 131.6, 131.3, 131.0, 129.9, 129.7, 129.2, 128.6, 128.2, 127.8, 127.6, 127.1, 126.8, 126.2, 125.6, 125.3, 125.1, 124.0, 122.8, 120.4, 118.2, 112.7, 34.9, 31.0, −2.4. ^{29}Si{^{1}H} NMR ($CDCl_3$): δ −16.6. MS: *m/z* 954 [M$^+$]. Anal. Calc. for $C_{62}H_{46}Si_2$: C, 88.00; H, 6.12. Found: C, 87.78; H, 6.01%.

*3.9. 9,10-Bis{[4-(4-carbazolyl)phenyldimethylsilyl]phenyl}anthracene (**11**)*

To a stirred THF solution (25 mL) of 1-carbazolyl-4-bromobenzene (0.69 g, 2.1 mmol) was added *n*-BuLi (1.5 mL, 2.4 mmol, 1.6 M in hexane) at −78 °C. The solution was stirred for 30 min at that temperature and then added 9,10-bis[4-(chlorodimethylsilyl)phenyl]anthracene (**5**) (0.49 g, 0.95 mmol) dissolved in THF (10 mL) via cannula. The solution was slowly warmed to room temperature and stirred another 8 h. H_2O and brine was added to the solution and the product was extracted with CH_2Cl_2. Pure **11** was isolated by silica gel chromatography (eluent: methylene chloride/hexane 1:5) as a white solid in 45% yield. M.p.: 205–206 °C. ^{1}H NMR ($CDCl_3$): δ 8.16 (d, 4H, $J = 7.2$ Hz, *Ph*), 7.90 (d, 4H, $J = 7.2$ Hz, *Ph*), 7.85 (d, 4H, $J = 7.2$ Hz, *Ph*), 7.79–7.72 (m, 4H, *anth*), 7.65 (d, 4H, $J = 7.2$ Hz, *Ph*), 7.55–7.29 (m, 20H, *Ph* and *anth*), 0.78 (s, 12H, Si–C*H*$_3$). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 140.9, 140.2, 138.7, 137.7, 137.1, 136.9, 136.0, 134.2, 131.2, 130.9, 129.9, 127.2, 126.3, 125.7, 124.9, 123.6, 120.5, 110.2, −2.1. ^{29}Si{^{1}H} NMR ($CDCl_3$): δ −10.7. MS: *m/z* 928 [M$^+$]. Anal. Calc. for $C_{66}H_{52}N_2Si_2$: C, 85.30; H, 5.64. Found: C, 85.12; H, 5.55%.

*3.10. 2-tert-Butyl-9,10-bis{[4-(4-carbazolyl)phenyldimethylsilyl]phenyl}anthracene (**12**)*

Compound **12** was prepared using the same procedure as described for **11** except 2-*tert*-butyl-9,10-bis[4-(chlorodimethylsilyl)phenyl]anthracene (**6**) was used instead of 9,10-bis[4-(chlorodimethylsilyl)phenyl]anthracene (**5**). Pure **12** was isolated by silica gel chromatography (eluent: methylene chloride/hexane 1:5) as a pale yellow solid in 42% yield. M.p.: 192–194 °C. ^{1}H NMR ($CDCl_3$): δ 8.17 (d, 4H, $J = 7.2$ Hz, *Ph*), 7.92–7.28 (m, 35H, *Ph* and *anth*), 1.26 (s, 9H, *t-Bu*), 0.79 (s, 12H, Si–C*H*$_3$). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 140.9, 140.2, 138.7, 137.7, 137.1, 136.9, 136.0, 134.2, 132.6, 131.2, 130.9, 130.5, 129.9, 128.2, 127.2, 126.3, 125.7, 124.9, 123.6, 122.6, 120.5, 119.6, 117.4, 110.2, 35.0, 30.9, −2.2. ^{29}Si{^{1}H} NMR ($CDCl_3$): δ −10.9. MS: *m/z* 984 [M$^+$]. Anal. Calc. for $C_{70}H_{60}N_2Si_2$: C, 85.32; H, 6.14. Found: C, 85.13; H, 6.03%.

3.11. *9,10-Bis[4-(pyrenyldimethylsilyl)phenyl]anthracene (**13**)*

Compound **13** was prepared using the same procedure as described for **11** except 1-bromopyrene was used instead of 1-carbazolyl-4-bromobenzene. Pure **13** was isolated by silica gel chromatography (eluent: methylene chloride/hexane 1:5) as a white solid in 50% yield. M.p.: 207 °C. ^{1}H NMR ($CDCl_3$): δ 8.38–7.99 (m, 18H, *pyr*), 7.82 (d, 4H, $J = 8.1$ Hz, *Ph*), 7.72 (dd, 4H, $J = 6.9$ Hz, $J = 3.3$ Hz, *anth*), 7.46 (d, 4H, $J = 8.1$ Hz, *Ph*), 7.33 (dd, 4H, $J = 6.9$ Hz, $J = 3.3$ Hz, *anth*), 0.98 (s, 12H, Si–CH_3). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 147.0, 140.7, 140.0, 138.1, 136.9, 136.6, 136.1, 134.7, 134.1, 133.4, 133.2, 132.6, 131.4, 130.8, 130.0, 129.6, 128.5, 128.2, 127.6, 127.2, 126.0, 125.5, 125.0, 124.1, −0.5. ^{29}Si{^{1}H} NMR ($CDCl_3$): δ −12.7. MS: *m/z* 846 [M$^+$]. Anal. Calc. for $C_{62}H_{46}Si_2$: C, 87.90; H, 5.47. Found: C, 87.72; H, 5.36%.

3.12. *2-tert-Butyl-9,10-bis[4-(pyrenyldimethylsilyl)phenyl]-anthracene (**14**)*

Compound **14** was prepared using the same procedure as described for **11** except 1-bromopyrene, and 2-*tert*-butyl-9,10-bis[4-(chlorodimethylsilyl)phenyl]anthracene (**6**) were used instead of 1-carbazolyl-4-bromobenzene, and 9,10-bis[4-(chlorodimethylsilyl)phenyl]anthracene (**5**), respectively. Pure **14** was isolated by silica gel chromatography (eluent: methylene chloride/hexane 1:5) as a pale yellow solid in 46% yield. M.p.: 198 °C. ^{1}H NMR ($CDCl_3$): δ 8.47 (d, 2H, $J = 9.3$ Hz, *pyr*), 8.37 (d, 2H, $J = 8.1$ Hz, *pyr*), 8.25–8.00 (m, 15H, *pyr*), 7.93 (d, 4H, $J = 7.8$ Hz, *Ph*), 7.78 (dd, 4H, $J = 6.9$ Hz, $J = 3.3$ Hz, *anth*), 7.57 (d, 4H, $J = 7.8$ Hz, *Ph*), 7.36 (dd, 4H, $J = 6.9$ Hz, $J = 3.3$ Hz, *anth*), 1.29 (s, 9H, *t-Bu*), 1.00 (s, 12H, Si–CH_3). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 147.3, 140.2, 140.0, 138.1, 136.9, 136.6, 136.1, 134.7, 134.1, 133.4, 133.2, 132.6, 132.2, 131.4, 130.8, 130.4, 130.0, 129.6, 129.2, 128.5, 128.2, 127.6, 127.2, 126.0, 125.5, 125.0, 124.1, 123.6, 123.0, 118.6, 35.1, 30.8, −0.2. ^{29}Si{^{1}H} NMR ($CDCl_3$): δ −12.8. MS: *m/z* 902 [M$^+$]. Anal. Calc. for $C_{66}H_{54}Si_2$: C, 87.76; H, 6.03. Found: C, 87.58; H, 5.92%.

3.13. *Polymer **15***

To a mixture of 2-*tert*-butyl-9,10-bis[4-(dimethylsilyl)-phenyl]anthracene (**4**) (0.55 g, 1.1 mmol) and 1,4-diethynylbenzene (0.14 g, 1.1 mmol) was combined with RhCl(PPh_3)$_3$ (0.002 g, 0.1 mol%), and NaI (0.015 g, 5 mol%) in THF (5 mL). The mixture was refluxed for 8 h under inert atmosphere. After evaporation, the polymerization mixture was dissolved in 5 mL of chloroform and the solution was poured into a large amount of methanol with vigorous stirring to form a precipitate. It was filtered with a glass frit, washing one more time with methanol, and dried under reduced pressure afforded the pale yellow polymer **15** in 88% yield. ^{1}H NMR ($CDCl_3$): δ 7.78–7.41 (m, 15H), 7.30 (dd, 2H, $J = 6.9$ Hz, $J = 3.3$ Hz, *anth*), 7.06 (d, 1H, $J = 18.6$ Hz, Ph–C*H*=), 6.74 (d, 1H, $J = 18.6$ Hz, Si–C*H*=), 1.26 (s, 9H, *t-Bu*), 0.57 (s, 12H, Si–CH_3). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 147.4, 140.1, 138.2, 137.5, 137.3, 136.9, 136.7, 134.3, 133.0, 132.2, 131.6, 131.4, 131.3, 131.0, 130.1, 129.9, 129.6, 128.5, 127.0, 126.1, 125.5, 125.3, 125.1, 124.7, 123.0, 35.1, 31.0, −2.2. ^{29}Si{^{1}H} NMR ($CDCl_3$): δ −16.5. $M_w = 200000$, $M_w/M_n = 1.29$.

3.14. *CV measurements*

The cyclic voltammetry experiment of polymer **15** was performed with a BAS-100 electrochemical analyzer. All measurements were carried out using a glassy carbon working electrode, a platinum auxiliary electrode and a non-aqueous Ag/$AgNO_3$ reference electrode. All experiments were performed at room temperature using 0.1 M tetrabutylammonium hexaflurophosphate (TPAP) as supporting electrolyte and THF as solvent.

3.15. *Fabrication of electroluminescent devices*

The ITO electrode was cleaned by sonication in a detergent solution for 4 min and washed with distilled water. Further sonication in 1,1,1-trichloroethane for 3 min was done before blowing dry under nitrogen. The devices were prepared as a sequence of ITO/PEDOT (65 nm)/EML (50 nm)/BaF_2 (2 nm)/Ca (50 nm)/Al (300 nm). The hole transporting material, poly(3,4-ethylene dioxythiophene)–poly(styrene sulfonic acid) (PEDOT, Baytron-P), was purchased from Bayer. PEDOT was first spin coated onto a pre-cleaned ITO glass and baked at 110 °C for 10 min. A solution of polymer **15** in toluene as an emission layer (EML) was spin-coated onto the surface of PEDOT. This film was baked at 130 °C for 1 h for removing the solvent. The BaF_2/Ca/Al cathode was deposited on the emitting polymer by thermal evaporation with a metal mask. The luminescence–voltage (*L*–*V*), current–voltage (*I*–*V*) and current efficiency–voltage characteristics were measured using a Keithley 238 source measurement unit and PR650 (Photo Research Corp.).

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2006.01.016.

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