

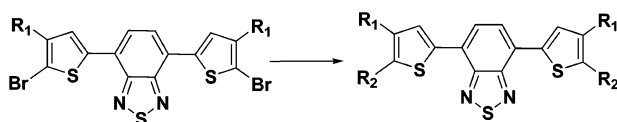
Amorphous Fluorescent Organic Emitters for Efficient Solution-Processed Pure Red Electroluminescence: Synthesis, Purification, Morphology, Solid-State Photoluminescence, and Device Characterizations

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$R_1 = \text{H}$, $R_2 = 2\text{-(9-carbazolyl)-9,9-dioctylfluorenyl}$ **1**

$R_1 = n\text{-hexyl}$, $R_2 = 3,5\text{-bis(naphthalen-1-yl)phenyl}$ **2**

New soluble and intrinsic amorphous red emitters **1** and **2** were obtained by Suzuki coupling. Due to the close molecular polarities, separation of target products from monocoupling impurities presents a challenge. The results showed highly pure **2** would be a potentially valuable fluorophore for solution-processed pure red electroluminescence in terms of visual sensitivity with respect to **1**.

Organic light-emitting materials and devices are continuing to advance toward practical applications since the early invention of efficient light-emitting diodes based on sublimable low molecular mass materials¹ and subsequent important observation of polymer electroluminescence (EL).² For the purposes of low cost device processing and large area display, solution-based technologies such as spin coating and printing techniques present distinct advantages. Along with this development, there is an ongoing interest in the synthesis of solution processible organic emitters.^{3,4} Among others, starburst and dendritic molecules have received particular attention since the well-defined multi-branched structures could not only effectively suppress inter-

molecular interactions and improve solid luminous efficiencies but also tend to induce a glassy state which is one of the crucial factors affecting the device durability. Highly efficient device performances have been observed based on such solution-processed fluorescent and phosphorescent emitters in the bulk and blend.^{4,5} With the latter, a small molecular charge-transporting host, 4,4'-bis(*N*-carbazolyl)-1,1'-biphenyl (CBP), is usually used.^{4,5b}

In the current scenario of materials development for solution processing, convenient and efficient syntheses of high-performance organic emitters with pure RGB emission are still desired for color display. For a material to possess solution processibility, a proper solubility and intrinsic glassy state are favored whether it be a host or dopant emitter. In the vacuum-deposited light-emitting devices (LEDs), the emitting materials are not necessary to be inherently amorphous, and those capable of forming molecular glasses under cooling from an isotropic melt are equally important. In this sense, the molecular design of solution processible emitters seems more demanding. Meanwhile, the synthesis might be troubled by purification. For instance, the potentially close molecular polarities between target products and reactants/reaction intermediates would likely render purification problematic by column separation, especially in synthesizing uniform multibranch molecules.⁶ In addition, for solution-processed molecular emitters, the tradeoff between morphological stabilities and solubilities has to be taken into consideration when solubilizing alkyl groups are introduced.

Nevertheless, despite the challenges that might be encountered, such as multistep reactions, purification, and yield concerns, it is worthwhile to explore new solution processible organic electroluminophores and synthetic possibilities to push them toward practical applications in virtue of the unique advantages they offer, such as potentially high materials purity and availability of reliable property–structure correlation with respect to polymers. We have become interested recently in solution-processed molecular materials⁷ and describe herein a new series of soluble and intrinsic amorphous red emitters **1** and **2**, based on double end-capped 4,7-di(thiophen-2-yl)-2,1,3-benzothiadiazole and 4,7-di(4-hexylthiophen-2-yl)-2,1,3-benzothiadiazole, respectively (Scheme 1).^{8,9} Due to electronic and structural reasons, pure red-emitting molecules and polymers are often susceptible to significant fluorescence quenching in the neat films. Interestingly, 2,1,3-benzothiadiazole was found to be an important and convenient class of electron acceptors and used to construct highly red photoemissive polymer solids for potential LED applications⁹ since the observation of high

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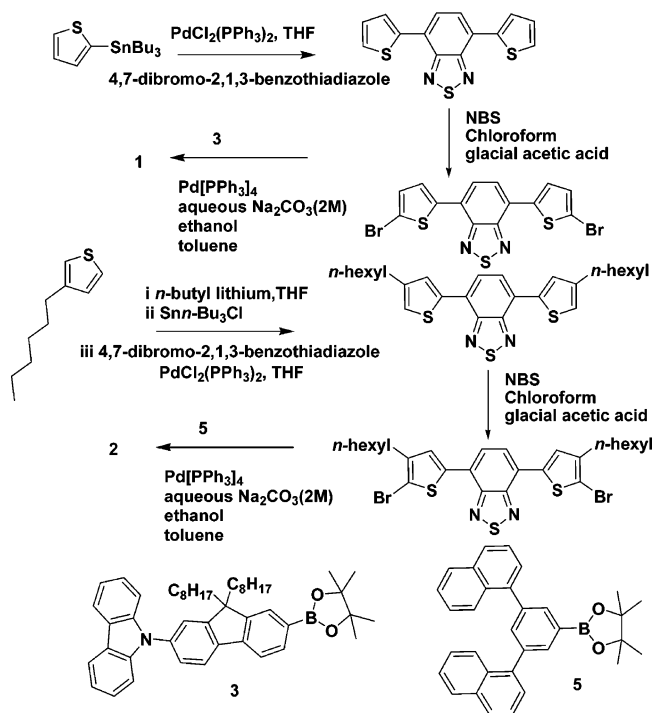
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SCHEME 1. Synthetic Routes to Red Emitters **1** and **2**

PL efficiencies of relevant fluorophores in solution.¹⁰ Small molecular benzothiadiazole-based derivatives¹¹ were later utilized in vacuum-deposited LEDs with high brightness.^{11a,b} However, efficient deep red EL devices based on them have yet to be fully explored. On the other hand, the lack of inherent amorphous morphology, that is, the glassy state comes upon cooling a melt, as noted, would prevent some of them from being favorable emitters for the purpose of solution processing. This might be part of the reason solution-processed molecular analogues remain scanty.¹²

The end group of 2-(carbazoyl-9-yl)-9,9-dioctylfluorenyl was successfully used to increase the solubility and morphological stability of iridium complexes previously.^{7a} 3,5-Bis(1-naphthyl)-phenyl was very conveniently available and was a potentially highly useful building block for constructing stable molecular glasses.¹³ In this work, they were used in an attempt not only to improve the morphological stabilities of the resultant target products but also to tune the emitting color in the red region and reduce solid PL quenching. The present study of low-polarity **1** and **2**, obtained via a double “symmetrical” substitution, was in a good approximation to the synthesis of multi-branched organic emitters and intended to give an opportunity to address a number of issues, such as synthesis, purification, solubility, morphological stability, and film-forming properties,

as well as materials functions. The results showed that **2** could be a potentially useful solution processible fluorophore for pure red LEDs.

1 and **2** were readily synthesized by reaction of 4,7-di(5-bromothiophen-2-yl)-2,1,3-benzothiadiazole and 4,7-di(5-bromo-4-hexylthiophen-2-yl)-2,1,3-benzothiadiazole^{8,9} with boronic acids **3** and **5**, respectively, via conventional Suzuki coupling.¹⁴ **3** was prepared with improved yield according to a previously described procedure.^{7a} Compared to the diphenylamino analogue, the rigid structure of the carbazoyl moiety is more liable to afford a glassy state.¹⁵ In addition, under the same Ullmann reaction conditions, 9H-carbazole seemed to react more efficiently with 2,7-dibromo-9,9-dioctylfluorene than diphenylamine. The latter usually undergoes effective amination using iodides and requires more demanding conditions.¹⁶ 3,5-Bis(1-naphthyl)phenyl bromide **4** was readily isolated from the coupling reaction of 1-naphthalenylboronic acid and 1,3,5-tribromobenzene, based on the method by Whitaker and McMahon.¹³ Subsequent lithiation and boronation of **4** yielded **5**. However, the isolation of **1** and **2** from monocoupling products initially seemed a challenge and improbable by column chromatography as thin layer chromatography (TLC) revealed they have very similar R_{f} values with a variety of eluents. Fortunately, in the case of **1**, it was found that the monocoupling impurity is significantly soluble in petroleum ether (PE) while the biscoupling product is much less. Finally, simple washing of the mixture in refluxing PE following column chromatography led to easy separation. In the case of **2**, a different strategy was adopted as the monocoupling product showed decreased (but still appreciable) solubility in PE. By continuously lowering polarity of the mixed eluent PE/ CH_2Cl_2 , it happened, eventually, that the monocoupling product could be constantly eluted off while **2** was almost unaffected on the column. With elution under a slight overpressure, the separation turned out efficient. The biscoupling product was then obtained smoothly by increasing the eluent polarity. *The partial solubility of the monocoupling products in petroleum ether is believed to be crucial for the effective purification of 1 and 2.* The identity and high purity of target products were thus confirmed by thin film chromatography (TLC), ^1H NMR, microanalysis, and MALDI-TOF mass measurement. They showed excellent solubility in common solvents such as dichloromethane and toluene. For instance, 40 mg of **1** was easily soluble in 1 mL of toluene *without saturation*. Generally, a higher boiling point solvent is beneficial for the film homogeneity of solution-processed light-emitting devices.

MALDI-TOF mass spectrometry revealed different ionization behavior of **1** and **2** using dithranol as matrix. A dominant peak at m/z 1408.8 (100%) was observed for **1**, corresponding to the molecular weight of a singly protonated species, implying the probable protonation of a carbazoyl moiety under high-energy conditions,^{7a} whereas the one at m/z 1124.44 (100%) was due apparently to parent **2** losing an electron.

The solution and solid UV-vis and PL spectra of **1** and **2** are shown in Figure 1, and data are summarized in Table 1. Both compounds are highly red emissive solids. The solid emission maxima occurred at 672 nm in **1** and at 630 nm with

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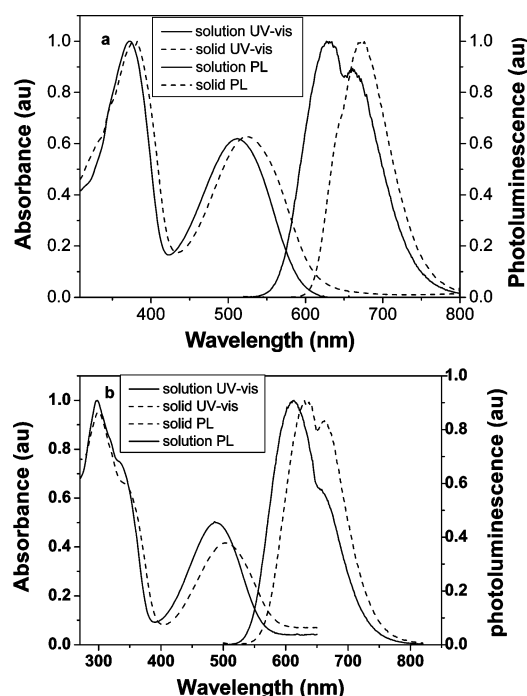


FIGURE 1. The UV-vis absorption and photoluminescence spectra of **1** (a) and **2** (b) in CH_2Cl_2 solutions and in the films on quartz.

a shoulder at 662 nm in **2**, coherent with the stronger electron-donating capacity of 2-(carbazoyl-9-yl)-9,9-dioctylfluorenyls. It is interesting to note that **1** and **2** revealed a rather high absolute solid PL efficiency of ca. 0.30 and 0.39, respectively, measured in the integrating sphere under direct excitation within the charge transfer absorptive band with a 488 nm argon laser.¹⁷ The values are almost half of those obtained under a 325 nm He-Cd laser excitation, while the emission spectra remained independent of the excitation wavelengths. Compared to a prototype dopant red emitter DCJTb,¹⁸ **1** and **2** exhibited much improved solid PL efficiencies, which could probably be related to efficient energy transfer from energy-harvesting end groups to central planar-emitting cores¹⁹ and certain solid-state molecular packing/morphology imposed by the end groups of 2-(9-carbazoyl)-9,9-dioctylfluorenyls and 3,5-bis(1-naphthyl)phenyls capable of preventing severe PL quenching.^{7a,13}

1 and **2** featured a broad emission with an unusual well-formed lower energy shoulder in solution, which cannot be attributed to the potential residual impurities in the samples based on the spectral shape and no “detectable” evidence of impurities by our current analytical methods. The same phenomenon occurred in some DCM-type red-light-emitting dyes and requires further understanding.²⁰

To probe the intrinsic solid morphology, the sample was carefully dried to avoid heating before DSC measurements. **1** and **2** showed remarkable thermal stabilities with decomposition temperatures well over 400 °C. DSC measurements on the as-

prepared samples revealed distinct glass transitions with a T_g of 72 °C for **1** and 82 °C for **2**, demonstrating the strong capacity of the current end groups to impose an inherent glassy state in these seemingly simple π -conjugated molecular systems. Notably, in both cases, neither crystallization nor melting was observed upon heating until 300 °C (Figure 2). The thermal processes were also confirmed by hot-stage polarized optical microscopy. The intrinsic morphology stability, together with attractive solubility and solid PL properties, enabled **1** and **2** to be desired red host/dopant emitters in solution-processed blended devices to prevent phase segregation which would become serious during the solvent evaporation process.

Initially, single-layered electroluminescent devices [ITO/PEDOT:PSS/1(**2**)/Ba/Al] were studied. **1** and **2** had onset voltages of 4.8–4.9 V at 1 cd/m^2 and maximal external device efficiencies of 0.66–0.88% with deep red emission. The relatively inefficient devices could be attributed to unbalanced charge injection and transport considering the high PL efficiencies in the neat films. The insertion of a hole transporting/electron blocking (HT/EB) PVK layer exerted only a limited effect on the device efficiencies.

MEH-PPV and P-PPV are well-studied yellow and green emissive polymers and showed few structural defects. Their solid emission spectra overlap with the optical absorptions of **1** and **2**, implying potential energy transfer from the polymers to red emitters when blended. The double-layered devices [ITO/PEDOT:PSS/PVK/1:MEH-PPV(80:20 wt/wt)/Ba/Al] and [ITO/PEDOT:PSS/PVK/2:P-PPV(64:36 wt/wt)/Ba/Al] showed much improved EL characteristics: $V_{\text{onset}} = 3.8$ V, $L_{\text{max}} = 890$ cd/m^2 , $\eta^{\text{ext}}_{\text{max}} = 2.1\%$, $\lambda_{\text{max}} = 665$ nm with CIE coordinates (0.69, 0.30) for **1**; and $V_{\text{onset}} = 4.3$ V, $L_{\text{max}} = 1960$ cd/m^2 , $\eta^{\text{ext}}_{\text{max}} = 1.82\%$, $\lambda_{\text{max}} = 637$ nm with CIE coordinates (0.654, 0.345) for **2**. At a current density of 32 mA/m^2 , $\eta^{\text{ext}} = 1.6\%$, $V = 7.2$ V, and $L = 394$ cd/m^2 for **2**, while $\eta^{\text{ext}} = 1.67\%$, $V = 6.7$ V and $L = 118$ cd/m^2 for **1** (Figure 2). Due to better visual sensitivity, the current efficiency of **2** was 1.23 cd/A with respect to 0.37 cd/A of **1**, while their external device efficiencies were very close under the same current. The diminished solid PL quenching made possible a high concentration of the red dyes into polymers. The significant enhancement in EL performances certainly resulted from more balanced charge injection and transport in the blends and the possibility of confined energy transfer from MEH-PPV and P-PPV to red host emitters in the presence of a HT/EB PVK layer. In the case of **2**, EL characteristics of the unoptimized as-blended device can compare well with one of the most efficient red electrofluorescent polymers, PFO-DHTBT10,^{9c} and with a 4,7-di(thiophen-2-yl)-2,1,3-benzothiadiazole-cored glass nematic liquid crystal lightly doped into an oligo(fluorene) host.^{12b} With the latter devices, higher working voltages were observed.^{9c,12b} Thus, via green-emitting P-PPV, efficient pure red electroluminescence was realized. Further device optimization might be directed to be compatible to the processing conditions of potential blue and green emitters.

The film homogeneities of spin-coated films of **1**, MEH-PPV, **1**:MEH-PPV(80:20 wt/wt), **2**, P-PPV, and **2**:P-PPV (64:36 wt/wt) from toluene solution were studied by atomic force microscopy (AFM). The corresponding surface root-mean-square roughnesses were found to be 0.30, 0.53, 0.34, 0.27, 0.66, and 0.50 nm, respectively. The excellent solubility, amorphous nature, and compatibility of **1** and **2** precluded any potential phase segregation in the blends.

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TABLE 1. UV-vis and PL Spectral Data

	UV-vis ($\lambda_{\text{max}}^{\text{abs}}$, nm)		PL ($\lambda_{\text{max}}^{\text{em}}$, nm, quantum yield)		HOMO (eV) ^d	LUMO (eV) ^e
	solution	solid	solution	solid		
1	372, 512	382, 524	627 (655 ^a , 0.93 ^b)	672 (–, 0.30 ^c)	–5.12	–3.06
2	298, 486	300, 503	613 (656 ^a , 0.58 ^b)	630 (662 ^a , 0.39 ^c)	–5.36	–3.18

^a Shoulder. ^b Quantum yields were measured in CH₂Cl₂ (1.0 × 10^{–6} mol L^{–1}) using rhodamine B in ethanol as the reference ($\phi_r = 0.64$).¹⁷ ^c Excited with 488 nm laser. ^d Derived from onset oxidation voltages using Ag/AgCl as reference electrode and ferrocene as internal standard. ^e Derived from optical band gap.

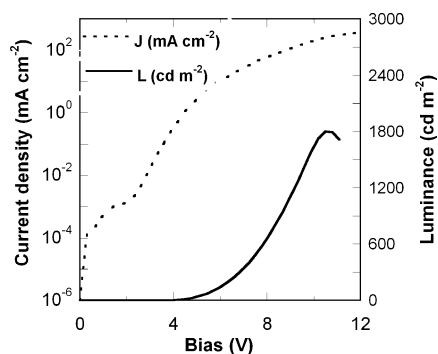


FIGURE 2. *J*–*V*–*L* characteristics of [ITO/PEDOT:PSS/PVK/2:P-PPV(64:36 wt/wt)/Ba/Al].

In summary, a new series of solution processible red emitters were synthesized via a symmetric double Suzuki coupling. The solubility of the monocoupling impurities was shown to be critical to purification. The desirable solubility, morphological stability, solid-state photoluminescence, and as-blended device characteristics, as well as the eventual feasibility of synthesis and purification, make **2** a promising molecular material for high-performance solution-processed pure red electroluminescence. Regarding the prominent importance of solution processible organic emitters for practical applications, attention is called to renewed and innovative molecular designs and synthetic methodologies to afford rapid, convenient, and high-yield syntheses and high materials purity. Intensive efforts along with this guideline are currently underway in our laboratory.

Experimental Section

Synthesis of 1. Pd(PPh₃)₄ (90 mg, 8.0 mmol) was added to a mixture of 4,7-bis(5-bromothiophen-2-yl)-2,1,3-benzothiadiazole (0.24 g, 0.53 mmol) and **3** in toluene (30 mL), aqueous Na₂CO₃ (2 M, 3 mL), and ethanol (3 mL) under an inert atmosphere of nitrogen. The reaction was heated at 90 °C overnight. After being cooled to room temperature, distilled water was added. The organic layer was separated, dried over MgSO₄, filtered, and concentrated under reduced pressure. The residue was subject to flash chromatography using silica gel and petroleum ether/CH₂Cl₂ (4:1 v/v) as the eluent to obtain **1** containing a certain amount of monocoupling

impurity. Subsequently, the crude product was dissolved in minimum CH₂Cl₂ and precipitated by addition of petroleum ether. The whole mixture was then heated to reflux, cooled, filtered, and washed with petroleum ether to afford **1** as a pure dark red solid (yield 0.49 g, 67%). For the purposes of TGA and DSC measurements, the sample was carefully dried to remove any residual aliphatic organic solvent used in the workup procedure and to avoid heating. The absence of solvents was confirmed by ¹H NMR prior to analytical characterizations: ¹H NMR (300 MHz, CDCl₃, ppm) δ 0.78–0.83 (m, 20H), 1.13–1.26 (m, 40H), 2.05–2.11 (m, 8H), 7.29–7.35 (m, 4H), 7.40–7.47 (m, 8H), 7.53–7.58 (m, 6H), 7.73 (s, 2H), 7.76–7.84 (m, 4H), 7.93 (d, 2H, *J* = 8.55 Hz), 7.97 (s, 2H), 8.17–8.21 (m, 6H). Anal. Calcd for C₉₆H₁₀₂N₄S₃: C, 81.89; H, 7.30; N, 3.98. Found: C, 81.44; H, 7.16; N, 4.06. MALDI-TOF: *m/z* 1408.8(100%) MH⁺ (calcd 1407.7).

Synthesis of 2. This compound was synthesized by a method similar to that for **1**, using 4,7-bis(5-bromo-4-hexylthiophen-2-yl)-2,1,3-benzothiadiazole and **5** instead. The crude product was subject to column chromatography using silica gel and graded petroleum ether/CH₂Cl₂ eluents from 10:1 to 6:1 (v/v) to afford **2** as a dark red solid (yield 76%): ¹H NMR (300 MHz, CDCl₃, ppm) δ 0.82 (t, 6H, *J* = 6.96 Hz), 1.24–1.27 (m, 12H), 1.33–1.41 (m, 4H), 1.77 (q, 4H, *J* = 7.44 Hz), 2.89 (t, 4H, *J* = 7.98 Hz), 7.49–7.60 (m, 16H), 7.66 (t, 2H, *J* = 1.59 Hz), 7.77 (d, 1H, *J* = 1.59 Hz), 7.85 (s, 2H), 7.88–7.95 (m, 8H), 8.05 (s, 2H), 8.13–8.16 (m, 4H). Anal. Calcd for C₇₈H₆₄N₂S₃: C, 83.23; H, 5.73; N, 2.49. Found: C, 83.12; H, 6.18; N, 2.56. MALDI-TOF: *m/z* 1124.44 (100%) M⁺ (calcd 1124.42).

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Supporting Information Available: Experimental details, ¹H NMR, MALDI-TOF mass spectra, TGA and DSC characterizations, PL spectra of MEH-PPV and P-PPV in the thin films, EL spectra and luminance–current density–voltage characteristics of as-blended devices, AFM images of spin-coated films of **1**, **2**, and **1**:MEH-PPV and **2**:P-PPV blends. This material is available free of charge via the Internet at <http://pubs.acs.org>.

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