

Synthesis and Characterization of 9,9-diethyl-1-phenyl-1,9-dihydrofluoreno[2,3-*d*]imidazole-ended Fluorophores

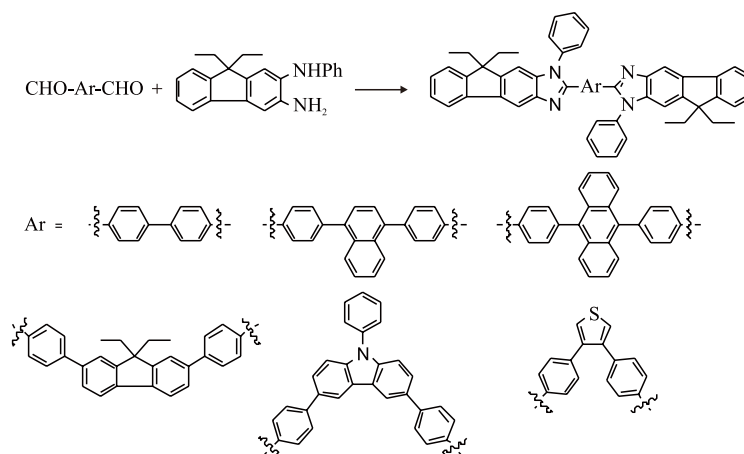
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Abstract: A series of π -conjugated compounds ending with 9,9-diethyl-1-phenyl-1,9-dihydrofluoreno[2,3-*d*]imidazole were conveniently synthesized by condensation of the key intermediate 9,9-diethyl-*N*²-phenyl-9H-fluorene-2,3-diamine with the corresponding symmetric aryl phthalaldehydes under very mild conditions. The structures of these compounds were confirmed by ¹H NMR, ¹³C NMR, and HRMS. Their UV-Vis spectroscopy data, fluorescent spectroscopy data, and further details of the electronic properties from cyclic voltammetry measurements and theoretical calculations were studied. Most compounds possess good fluorescence-emitting ability with quantum yield of fluorescence values in the region of 0.36-0.92 and display emission within 449-513 nm depending on the molecular nature.

Key words: fluorene; imidazole; synthesis; UV absorption; fluorescence

Graphical abstract



1 Introduction

Fluorene has been used as a functional building block in the fabrication of materials for applications in organic light-emitting diodes, field-effect transistors and organic solar cells (OSCs)^[1-4]. The molecular and photophysical properties of fluorenes can be tuned by structural modifications at the 2, 3, 6, 7, and 9- positions, which makes them one of the most

important classes of advanced materials being used in organic electronics^[5-9]. Fluorene derivatives have found application in electroluminescent devices as emitting materials due to their excellent electric or hole transporting ability, luminescence efficiency, thermal stability, and attractive optoelectronic properties^[10-17]. However, small-molecule fluorene derivatives are still rarely reported in the practical applications in OLEDs^[18-20].

Fluorescent characteristic relies largely on molecular structure and molecular assembly. Clarification the structure-property relationship of fluorescence would allow us to design more useful fluorescent reagents and probes which might be applied in other fields such as analytical and biological chemistry in the future.

Based on our continued research on fluor-

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eno[2,3-*d*]imidazole systems with specific optoelectronic properties^[21-23]. We reported the efficient synthesis and characterization of a series of compounds **3a-f** ending with 9,9-diethyl-1-phenyl-1,9-dihydrofluoreno[2,3-*d*]imidazole groups.

2 Experimental

2.1 Reagents and instrument

Commercially available starting chemicals were purchased and used as received without further purification. Thin-layer chromatography (TLC) was conducted on silica gel GF254 plates. ¹H NMR and ¹³C NMR spectra were recorded on a Bruker Avance (500 MHz) spectrometer in CDCl₃, unless otherwise noted. Chemical shifts were reported in the scale relative to CHCl₃ (7.26 ppm) for ¹H NMR, and to CDCl₃ (77.16 ppm) for ¹³C NMR, as internal references. High-resolution mass spectrometer (HRMS) spectra were recorded on Agilent 6540/6538. UV-Vis spectra were recorded on UV-2501Pc spectrophotometer. Fluorescent measurements were recorded on RF-5301 spectrophotometer.

2.2 General procedure for the synthesis of compounds **3a-3f**

The compound 9,9-diethyl-N²-phenyl-9H-fluorene-2,3-diamine (1000 mg, 3.04 mmol) and the corresponding aldehyde (1.38 mmol) were dissolved in a mixed solvent of DMF and H₂O (*V*_{DMF}:*V*_{H₂O}=30:1), oxone (1.1 g, 1.8 mmol) was added, and the mixture was stirred at room temperature until the reaction was complete. Then the mixture was extracted with ethyl acetate, evaporated and purified with ethyl acetate and petroleum ether to afford the target compounds **3a-3f**.

2.2.1 4,4'-bis(9,9-diethyl-1-phenyl-1,9-dihydrofluoreno[2,3-*d*]imidazol-2-yl)-1,1'-biphenyl (**3a**)

Light yellow solid, Yield: 46.3%. M.p. = 303-305 °C; ¹H-NMR (500 MHz, CDCl₃) δ (ppm) 8.16 (s, 2H), 7.82 (d, *J* = 7.5 Hz, 2H), 7.63 (d, *J* = 8.4 Hz, 4H), 7.60-7.51 (m, 10H), 7.41 (d, *J* = 7.1 Hz, 4H), 7.38-7.33 (m, 2H), 7.32-7.28 (m, 4H), 7.11 (s, 2H), 2.08-1.91 (m, 8H), 0.31 (t, *J* = 7.3 Hz, 12H); ¹³C-NMR (125 MHz, CDCl₃) δ (ppm) 151.89, 149.55, 146.86, 142.85, 141.70, 140.75, 137.92, 137.62, 137.21, 130.05, 129.81, 129.40, 128.65, 127.60, 126.93, 126.82, 126.66, 122.79, 119.53, 110.19, 104.41, 55.83, 33.53, 8.54. HR-MS: *m/z* calcd for C₆₀H₅₀N₄ ([M+H]⁺) 827.4114, found 827.4101.

2.2.2 1,4-bis(4-(9,9-diethyl-1-phenyl-1,9-dihydrofluoreno[2,3-*d*]imidazol-2-yl)phenyl)naphthalene (**3b**)

Light yellow solid, Yield: 28.5%. M.p. = 305-

307 °C; ¹H-NMR (500 MHz, DMSO-*d*₆) δ (ppm) 8.08 (s, 2H), 7.83 (d, *J* = 2.8 Hz, 2H), 7.72 (d, *J* = 7.1 Hz, 2H), 7.60 (d, *J* = 7.6 Hz, 4H), 7.48 (t, *J* = 7.1 Hz, 4H), 7.44-7.40 (m, 2H), 7.39-7.29 (m, 12H), 7.25 (t, *J* = 6.5 Hz, 2H), 7.18 (d, *J* = 7.9 Hz, 4H), 7.03 (s, 2H), 1.90 (m, 8H), 0.21 (t, *J* = 7.3 Hz, 12H); ¹³C-NMR (125 MHz, CDCl₃) δ (ppm) 152.11, 149.57, 146.86, 142.87, 141.73, 141.69, 139.29, 137.92, 137.65, 137.25, 131.72, 130.11, 130.08, 129.29, 129.16, 128.71, 127.66, 126.95, 126.66, 126.43, 126.24, 126.15, 122.81, 119.56, 110.23, 104.47, 55.84, 33.54, 8.58.

2.2.3 9,10-bis(4-(9,9-diethyl-1-phenyl-1,9-dihydrofluoreno[2,3-*d*]imidazol-2-yl)phenyl)anthracene (**3c**)

White solid, Yield: 19%. M.p. = 305-308 °C; ¹H-NMR (500 MHz, DMSO-*d*₆) δ (ppm) 8.09 (d, *J* = 1.5 Hz, 2H), 7.91 (m, 4H), 7.86-7.83 (m, 6H), 7.60 (d, *J* = 7.6 Hz, 4H), 7.56 (s, 2H), 7.48 (t, *J* = 7.1 Hz, 4H), 7.44-7.40 (m, 2H), 7.39-7.29 (m, 12H), 7.25 (t, *J* = 6.5 Hz, 2H), 2.06-1.90 (m, 8H), 0.21 (t, *J* = 7.3 Hz, 12H); ¹³C-NMR (125 MHz, CDCl₃) δ (ppm) 159.13, 151.45, 150.70, 141.13, 138.64, 136.70, 135.84, 133.05, 130.89, 129.75, 129.36, 128.47, 128.41, 126.95, 126.56, 125.94, 123.42, 119.79, 118.39, 114.71, 104.23, 55.85, 33.51, 8.56.

2.2.4 2,2'-((9,9-diethyl-9H-fluorene-2,7-diyl)bis(4,1-phenylene))bis(9,9-diethyl-1-phenyl-1,9-dihydrofluoreno[2,3-*d*]imidazole) (**3d**)

Yellow solid, Yield: 40.4%. M.p. = 310-313 °C; ¹H-NMR (500 MHz, DMSO-*d*₆) δ (ppm) 8.19 (s, 2H), 7.83 (d, *J* = 7.4 Hz, 2H), 7.77 (d, *J* = 7.9 Hz, 2H), 7.66 (dd, *J* = 21.2, 8.4 Hz, 8H), 7.57 (dt, *J* = 18.5, 7.2 Hz, 10H), 7.45 (d, *J* = 7.2 Hz, 4H), 7.39-7.35 (m, 2H), 7.33-7.29 (m, 4H), 7.12 (s, 2H), 2.14-1.93 (m, 12H), 0.38 (t, *J* = 7.3 Hz, 6H), 0.32 (t, *J* = 7.3 Hz, 12H); ¹³C-NMR (125 MHz, CDCl₃) δ (ppm) 152.10, 150.97, 149.55, 146.78, 142.85, 142.21, 141.74, 140.84, 139.16, 137.89, 137.67, 137.32, 130.06, 129.78, 128.64, 127.66, 126.93, 126.88, 126.16, 122.78, 121.35, 120.17, 119.53, 110.16, 104.40, 57.96, 55.82, 33.54, 8.55. HR-MS: *m/z* calcd for C₇₇H₆₆N₄ ([M+H]⁺) 1047.5366, found 1047.5357.

2.2.5 2,2'-((9-phenyl-9H-carbazole-3,6-diyl)bis(4,1-phenylene))bis(9,9-diethyl-1-phenyl-1,9-dihydrofluoreno[2,3-*d*]imidazole) (**3e**)

Light yellow solid, Yield: 39.2%. M.p. = 303-306 °C; ¹H-NMR (500 MHz, DMSO-*d*₆) δ (ppm) 8.41 (d, *J* = 1.5 Hz, 2H), 8.18 (s, 2H), 7.83 (d, *J* = 7.5 Hz, 2H), 7.71-7.67 (m, 10H), 7.66-7.62 (m, 3H), 7.60 (d, *J* = 7.6 Hz, 5H), 7.56 (t, *J* = 7.3 Hz, 2H), 7.51 (t, *J* = 7.3 Hz, 1H), 7.48-7.45 (m, 6H), 7.39-7.35 (m, 2H), 7.33-7.28 (m, 4H), 7.13 (s, 2H), 2.02 (m, 8H), 0.32 (t, *J* = 7.3 Hz, 12H); ¹³C-NMR (125 MHz, CDCl₃) δ (ppm) 151.90,

149.56, 149.56, 146.86, 146.71, 142.87, 142.83, 142.48, 141.77, 141.69, 141.07, 140.74, 137.91, 137.84, 137.67, 137.61, 137.33, 137.18, 130.05, 129.83, 129.81, 128.64, 127.67, 127.59, 126.97, 126.93, 126.82, 122.79, 119.52, 110.33, 110.17, 110.12, 104.41, 55.82, 33.53, 8.55. HR-MS: m/z calcd for $C_{78}H_{61}N_5$ ($[M+H]^+$) 1068.5006, found 1068.4997.

2.2.6 3,4-bis(4-(9,9-diethyl-1-phenyl-1,9-dihydrofluoreno[2,3-*d*]imidazol-2-yl)phenyl)thiophene (3f)

Light yellow solid, Yield: 33.4%. M.p. = 245-248 °C; 1H -NMR (500 MHz, $CDCl_3$) δ (ppm) 8.14 (d, $J = 0.6$ Hz, 2H), 7.81 (d, $J = 7.5$ Hz, 2H), 7.63 (dd, $J = 8.0, 6.3$ Hz, 1H), 7.57 (dd, $J = 12.9, 5.1$ Hz, 1H), 7.53-7.49 (m, 4H), 7.45-7.39 (m, 6H), 7.38-7.32 (m, 7H), 7.29 (dt, $J = 6.4, 3.4$ Hz, 4H), 7.11-7.07 (m, 5H), 2.06-1.90 (m, 8H), 0.30 (t, $J = 7.3$ Hz, 12H); ^{13}C -NMR (125 MHz, $CDCl_3$) δ (ppm) 149.42, 147.88, 141.09, 140.04, 138.26, 136.91, 136.33, 134.56, 130.95, 129.67, 128.22, 126.73, 124.31, 123.28, 122.19, 121.66, 115.18, 57.33, 35.42, 8.93. HR-MS: m/z calcd for $C_{64}H_{52}N_4S$ ($[M+H]^+$) 909.3992, found 909.3983.

3 Results and discussion

3.1 Synthesis

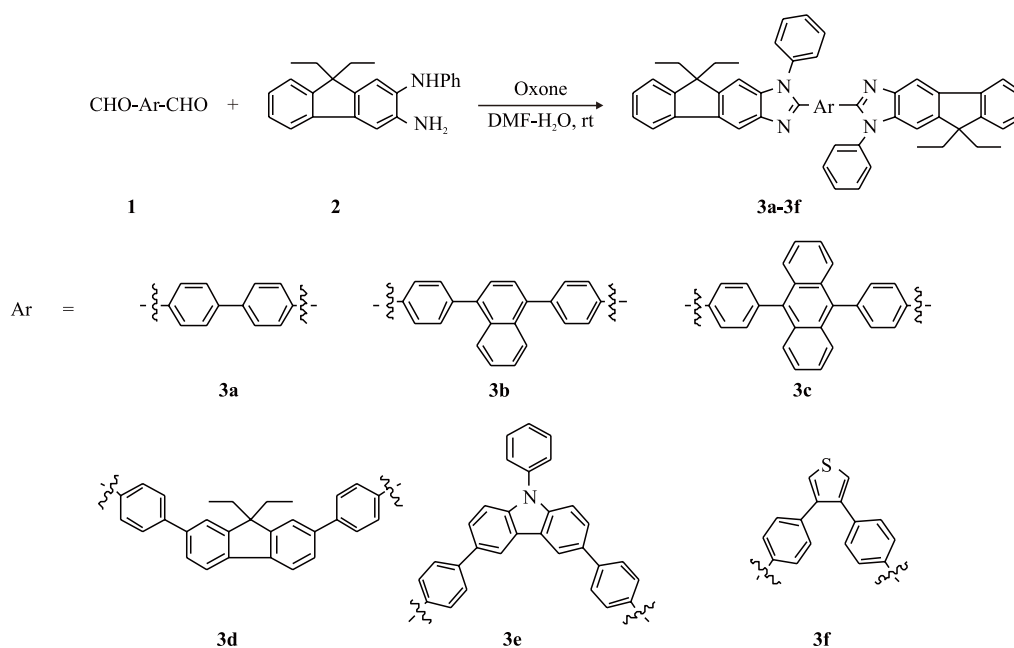
As shown in scheme 1, condensation of the corresponding symmetric aryl phthalaldehyde (**1**) with the key intermediate 9,9-diethyl-*N*²-phenyl-9H-fluorene-2,3-diamine (**2**)^[21] in wet DMF at room temperature under oxone-mediated oxidative conditions afforded the target compounds **3a-f** in 19%-46.3%

yields. All target compounds were isolated and purified by column chromatography on silica gel with high purity and their molecular structures were confirmed by 1H NMR spectroscopy, ^{13}C NMR spectroscopy, and HRMS, which are shown detailed in experiment section.

3.2 Optical properties

The UV-Vis absorption spectra of compounds **3a-f** were measured in CH_2Cl_2 and DMSO solutions with the concentration of 1×10^{-5} mol/dm³ at room temperature, and the optical characteristics are summarized in Table 1. All compounds displayed similar absorption characteristics, with two absorption peaks in the ranges of 250-277 and 345-376 nm, which could be attributed to different intramolecular π - π^* transitions. Compounds **3d** and **3e** with the absorption maxima at 366 and 376 nm, respectively, had longer absorption wavelengths, as the conjugated length extended, while, **3f** showed the shortest λ_{max} at 345 nm because of the increased steric hinderance and less planar structures. The UV-Vis absorption spectra measured in DMSO solutions indicated that all compounds showed small red shifts no more than 3 nm, compared with the corresponding spectra in CH_2Cl_2 , suggesting that the ground states of all these compounds are rather nonpolar in nature.

The photoluminescence (PL) spectra were recorded upon excitation at each excitation wavelength (λ_{ex}) in CH_2Cl_2 solution (1×10^{-7} M) at room temperature. In CH_2Cl_2 solution, the fluorescence spectra of compounds **3a** and **3b** were very similar with emission peaks at 475 nm. The fluorescence spectra



Scheme 1 Synthesis of conjugated compounds **3a-f**

Table 1 Optical properties and Φ_{FL} of compounds **3a-f**

Compound	λ_{abs}/nm^a		λ_{ex}/nm^b		λ_{em}/nm^c		Stokes shift/nm ^d		Φ_{FL}^e
	CH ₂ Cl ₂	DMSO	CH ₂ Cl ₂	DMSO	CH ₂ Cl ₂	DMSO	CH ₂ Cl ₂	DMSO	
3a	277,361	277,363	371	372	475	478	104	106	0.91
3b	250,351	252,354	352	355	475	477	123	122	0.56
3c	250,354	251,355	355	356	449	451	94	95	0.36
3d	256,366	258,367	367	368	502	507	135	139	0.51
3e	260,376	261,378	376	376	513	515	137	139	0.52
3f	261,345	262,346	356	357	449	453	93	96	0.41

^aUV-Vis absorption wavelength in CH₂Cl₂ or in DMSO at room temperature

^bExcitation wavelengths in CH₂Cl₂ or in DMSO at room temperature

^cFluorescence wavelengths in dichloromethane or in DMSO at room temperature

^dStokes shift in dichloromethane or in DMSO at room temperature

^eFluorescence quantum yields, measured in CH₂Cl₂ solution using a 0.2 M H₂SO₄ solution of quinine sulfate ($\Phi_{FL} = 0.55$) as a reference

of **3d** (peaked at 502 nm), and **3e** (peaked at 513 nm) were both red-shifted relative to that of **3a** due to the elongation of the conjugation length. However, the anthracene inserted derivative (**3c**) with emission peak at 449 nm was blue-shifted by 26 nm relative to **3a**, this might be attributed to the highly twisted conformation between the anthracene and the 9,9-diethyl-1,2-diphenyl-1,9-dihydrofluoreno[2,3-*d*]imidazole units. Similar to that of **3c**, the derivative **3f** showed blue emission, with the emission peak at 449 nm. In addition, the solvent effect on the fluorescence characteristics was also observed. All compounds were slightly red-shifted in DMSO relative to those in CH₂Cl₂.

Quantum yields were calculated on the basis of quinine sulfate ($\Phi_{FL} = 0.55$). **3a** showed the highest quantum yield in solutions and relatively lower quantum yields were observed for all the other compounds and compounds **3c** and **3f** showed the lowest emission intensities maybe because of the increased torsional strain by inserting an anthracene or 1,2-thiophene into biphenyl. In all, the emission efficiency in dilute solution largely depends on the molecular structure and can be tuned by structural modification.

3.3 Electrochemical properties

The redox potentials of all compounds were determined by cyclic voltammetry (CV) measurements which were carried out in a three-electrode cell setup with 0.1 M tetrabutylammonium perchlorate (Bu₄NClO₄) as a supporting electrolyte in anhydrous CH₂Cl₂ to probe the electrochemical behavior of the materials. As seen from Table 2, the onset potentials of oxidation (E^{ox}) of these compounds were at about 0.92-0.97 eV. The band gaps (E_g) of about 2.88-3.12 eV were estimated from the optical edge. Compounds **3c** and **3f** exhibited a relatively larger band gap by comparison with those of compounds **3a-b**, **3d-e**, which derived

from the more distorted π -conjugation structures. The LUMO energy levels for compounds **3a-f** were determined to be around 2.42-2.67 eV by combining the HOMO energy levels together with the band gaps.

Density functional theory (DFT) calculations have also been applied to characterize the three-dimensional geometries and the frontier molecular orbital energy levels of **3a-f** at the B3LYP/6-31G* level by using the Gaussian 03 program. The orbital plots of the HOMO-LUMO are illustrated in Fig.1. In most of the above compounds (**3a-b** and **3d-e**), the HOMOs were located on the entire molecular backbones consisting of the two fluorenoimidazole skeletons and the bridging aryl ring. Their LUMOs, however, were mainly concentrated on the interconnected aryl ring. In the case of compound **3c**, the anthracene was nearly perpendicular to the 9,9-diethyl-1,2-diphenyl-1,9-dihydrofluoreno[2,3-*d*]imidazole units, the HOMO and LUMO were localized mainly on the anthracene as expected. For compound **3f**, the HOMO was mainly constituted by one fluorenoimidazole skeleton and a bridging phenyl ring, however, LUMO was localized on the other side.

Table 2 Optical properties of compounds **3a-3f**

Compound	HOMO/eV ^a	LUMO/eV ^b	E^{ox}/V^c	E_g/eV^d
3a	-5.56	-2.58	0.96	2.98
3b	-5.52	-2.42	0.92	3.10
3c	-5.54	-2.42	0.94	3.12
3d	-5.55	-2.67	0.95	2.88
3e	-5.52	-2.58	0.92	2.94
3f	-5.57	-2.46	0.97	3.11

^a HOMO energy levels derived from the oxidation potential using HOMO = $-E^{ox} - 4.60$, the redox potentials were measured with cyclic voltammetry system

^b LUMO energy levels were deduced using the formula LUMO = HOMO + E_g

^c Onset of oxidation potentials. E^{ox} vs Fc+/Fc estimated by CV

^d $E_g = 1240/UV(\text{onset})$. UVs(onset) were estimated from the onset of the absorption spectra

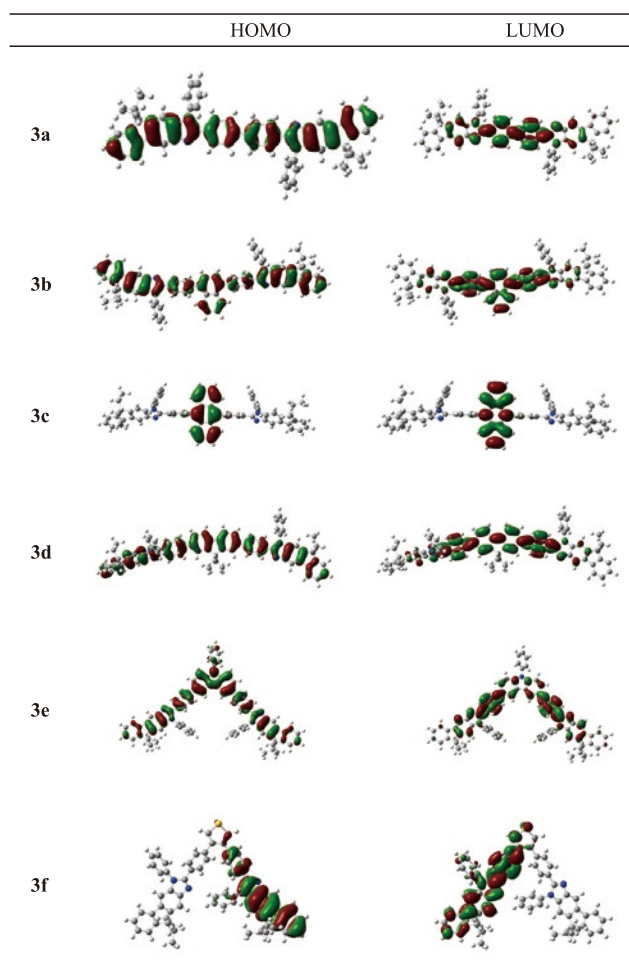


Fig.1 HOMO and LUMO electronic density distributions of compounds 3a-3f

4 Conclusions

The synthesis and structure-property relationship of a series of π -conjugated compounds ending with 9,9-diethyl-1-phenyl-1,9-dihydrofluoreno[2,3-*d*]imidazole groups was studied in detail. The absorption spectra and fluorescence characteristics show dependence on their molecular structure and the luminescent efficiencies can be tuned by structural modification. This study demonstrates that systematic study of these organic emitters can provide an efficient way to produce excellent candidates for the potential applications in optoelectronics. Further research regarding the incorporation of these systems usable as organic light-emitting diodes will be reported in due course.

Conflict of interest

All authors declare that there are no competing interests.

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