# Article

# Synthesis and Fluorescent Properties of Tetra-Biphenyl N-Substituted Phthalimides

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A series of tetra(biphenyl-4-yl)phthalimide (TBPPI) derivatives with different *N*-substituents (*n*-butyl, phenyl, *p*-methyl phenyl, and *p*-acetyl phenyl moieties for compounds 7–10, respectively) were prepared to examine their fluorescent behavior under various conditions. The chemical structure of compound 7 has been successfully confirmed by single crystal X-ray diffraction analysis. The photoluminescence (PL) spectra in different ratios of  $CH_2Cl_2/EtOH$  mixture solutions revealed that compounds 7 and 8 exhibited both aggregation-induced emission (AIE) and aggregation-caused quenching (ACQ) behaviors, while compounds 9 and 10 displayed AIE and aggregation-induced emission enhancement (AIEE) properties, respectively.

Keywords: Tetra-biphenyl phthalimide; Photoluminescence spectrum; Aggregation-induced emission.

## INTRODUCTION

Organic semiconductors have drawn much attention from organic chemists in recent decades owing to their potential applications to inexpensive, lightweight, flexible, and large-area electronic devices such as organic light-emitting diodes (OLEDs).<sup>1</sup> Despite this promising potential for commercial optoelectronic applications, the fundamental intermolecular interactions of a single compound containing the phthalimide moiety both in dilute solution and in the solid state have not yet been fully investigated. It is well known that molecules of organic semiconductors commonly consist of planar aromatic rings.<sup>2</sup> For most organic semiconductors, the molecules are isolated and luminescent in dilute solution. However, when the molecules exist in the solid state, or the solution of the molecules is highly concentrated, the luminescence might be strongly quenched as a result of the aggregation of the molecules.<sup>3</sup> Such "aggregation-caused quenching" (ACQ) is believed to be due to the formation of  $\pi$ - $\pi$  interactions, in which the electrons can be delocalized over two or more molecules, leading to a long lifetime of excitons, red-shifting of emission, and low luminescence quantum efficiency.<sup>4</sup> Therefore, how to reduce the impact of the ACQ effect is a principal issue for real application of OLEDs.

Since 2001, Tang et al. have been reporting a series of unusual chromophores including silole and tetraphenylethene (TPE) derivatives (Figure 1) that are nonluminescent in dilute solutions but emit bright luminescence in their concentrated solution or cast films.<sup>5,6</sup> For example, dilute solutions of hexaphenylsilole (HPS) in good solvents such as acetonitrile and tetrahydrofuran (THF) are nonemissive because of the free rotation of the phenyl rings against the silole core, which can annihilate its excitons via a nonradiative pathway.<sup>5c</sup> However, when HPS molecules aggregate in the solid state, the restriction of intramolecular rotations (RIR) block the nonradiative pathway, thus making the molecules highly luminescent. They termed this abnormal phenomenon "aggregation-induced emission" (AIE), in which the  $\pi$ - $\pi$  interactions cannot be formed because of the heavy steric hindrance. Since AIE has high potential for the applications of OLEDs, efforts have been made to design and synthesize new AIE organic semiconductors.7 In 2010, Tang et al. utilized the AIE material 3TPETPA and the AIE enhancement (AIEE) material TTPEPy as emitting layers to fabricate a series of highly efficient OLEDs.<sup>6a,6b</sup> Because the hole mobility in most organic semiconductors is much higher than the electron mobility, an additional electron-transporting layer (ETL) is needed to balance the charge mobilities

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Fig. 1. Chemical structures and abbreviated notations of selected AIE and AIEE materials in the literature.

and enhance the performance of OLEDs.<sup>8</sup> To simplify the fabrication process and lower the construction cost of OLEDs, Tang *et al.* combined the electrontransporting group 2,5-diaryl-1,3,4-oxadiazole (Oxa) and the AIE chromophore TPE into a single molecule (TPE-Oxa).<sup>6c</sup> They demonstrated that the performance of OLEDs based on TPE-Oxa without the ETL was even better than that with the ETL. To the best of our knowledge, only a few AIE chromophores with electron-transporting groups have been reported so far.<sup>6c-6i</sup> Therefore, the design and synthesis of new AIE chromophores with electron-transporting groups for applications in OLEDs are extremely necessary.

In this study, we synthesized four electrondeficient chromophores, tetra(biphenyl-4-yl)phthalimide (TBPPI) derivatives (compounds 7–10), as shown in



Scheme 1. Synthetic routes to the tetra(biphenyl-4-yl) 1phthalimide derivatives.

Scheme 1. The chemical structure of compound 7 was successfully confirmed by single-crystal X-ray diffraction analysis. In order to understand the AIE (or AIEE) properties of TBPPIs, their fluorescent behavior in different ratios of  $CH_2Cl_2/EtOH$  mixture was investigated. Their photoluminescence (PL) spectra showed that compounds 7 and 8 exhibited both AIE and ACQ behaviors while compounds 9 and 10 displayed AIE and AIEE properties, respectively.

### **RESULTS AND DISCUSSION**

The idea of designing the target materials, namely compounds 7–10 (Scheme 1), for this work is as follows: in order to understand how the optical properties of the tetra-biphenyl isoindole-dione structure could be influenced by the various substituents on the nitrogen atom, n-butyl moiety was adopted as the alkyl electrondonating group on 7, phenyl moiety as the aryl electrondonating group on 8, p-methyl phenyl moiety as the aryl electron-donating group on 9, and p-acetyl phenyl moiety as the aryl electron-withdrawing group on 10. Thus, the target molecules 7-10 were synthesized based on the methodology outlined in Scheme 1. According to the procedure described in the literature.<sup>9,10</sup> periodination of phthalic anhydride was carried out in concentrated sulfuric acid to give tetraiodophthalic anhydride (2). The reaction reported by Golub et al. was then applied for the conversion of the anhydride 2 to compounds 3-**6**.<sup>11</sup> Finally, Suzuki coupling reaction of tetraiodophthalimides 3-6 with biphenyl-4-boronic acid furnished the target compounds 7–10, respectively.

It is noteworthy that a single crystal of compound 7 was obtained, and the structure of which was confirmed by X-ray crystallography (Figure 2(a)). The Tetra-biphenyl Phthalimides

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Fig. 2. Crystal structure of compound 7: (a) single molecule and (b) the dimer packing. Carbon in gray, hydrogen in white, oxygen in red, and nitrogen in blue. Space-filling molecular model presents examples of  $\pi$ -stacking dimers for clarity.

dihedral angles between the phthalimide and substituted phenyl rings, R1, R2, R3, and R4, were found to be as large as 51.5°, 61.1°, 66.3°, and 64.9°, respectively, demonstrating that there is steric hindrance between the phthalimide core and the phenyl rings. The crystal structure of compound **7** adopts antiparallel  $\pi$ -stacks of dimers with a short intermolecular distance of 3.73 Å, which indicates the existence of intermolecular interactions. These antiparalleldimers reveal that the electronrich phenyl ring of phthalimide faces the electron-poor maleinimide of phthalimide, as shown in Figure 2(b).

To investigate the optical properties of TBPPIs, the fluorescent behaviors of 7-10 in different conditions were measured using the method reported in the literature.<sup>12</sup> Compounds 7–10 dissolved well in good solvents such as CH<sub>2</sub>Cl<sub>2</sub> and THF, and the fluorescence quantum yields of these target molecules were obtained (see Supporting information). In 10 µM of a pure CH<sub>2</sub>Cl<sub>2</sub> solution, compound 7 was weakly fluorescent with the maximum intensity ( $\lambda_{max}$ ) at 455 nm and quantum yield  $(\Phi_{\rm F})$  as low as 17%. The PL intensity and quantum yields ( $\Phi_{\rm F}$ ) of compound 7 were enhanced (Figure 3) when the volume fraction of the nonsolvent, EtOH, was increased from 0% (E0) to 45% (E45), while these values were decreased as the amount of EtOH was larger than 60% (E60). The  $\lambda_{max}$  values of compound 7 were red-shifted gradually from 455 (E0) to 512 nm (E90) as the ratio of EtOH was increased. These results indicated that the fluctuating  $\Phi_{\rm F}$  values and red-shifted luminescence of compound 7 were due to the combined effects of AIE and ACQ. In a good solvent, compound 7 was weakly fluorescent due to the de-excitation of excitons by free rotations of the biphenyl rings against



Fig. 3. Fluorescent character of compound 7. (a) PL spectra in CH<sub>2</sub>Cl<sub>2</sub>/EtOH mixture  $(10^{-5} \text{ M})$  excited at 300 nm. (b) Quantum yield in CH<sub>2</sub>Cl<sub>2</sub>/EtOH mixture. (c) Photograph taken under UV illumination.

the phthalimide core. As the ratio of EtOH was increased, the molecules of compound 7 were closer to each other such that the intramolecular rotations of biphenyl rings were restricted and the  $\Phi_F$  values were enhanced.

At the same time, the molecules formed antiparallel dimers progressively owing to the  $\pi$ - $\pi$  interactions (Figure 2(b)), which caused the ACQ effect and the redshift of the emission.

When the amount of EtOH was less than 45%, AIE was dominant, and therefore the luminescence was enhanced and red-shifted. However, ACQ dominated when the amount of EtOH was over 60%. The green emission of E90 was similar to that of the cast film under UV irradiation, which demonstrated that such emission was from the antiparallel dimers of compound 7.

The variations in wavelength of the PL spectra and  $\Phi_{\rm F}$  values of compound 8 (Figure 4) show similar tendency as those of compounds 9 and 10 (Figures 5 and 6), indicating that the fluorescent behavior of compound 8 at various ratios of CH<sub>2</sub>Cl<sub>2</sub>/EtOH mixture was also controlled by AIE. In contrast to compound 7, compounds 8–10 do not show any shift in wavelength of the PL spectra with the amount of EtOH; at the same time, their  $\Phi_{\rm F}$  values were enhanced with increasing fraction of EtOH. We therefore can conclude from the above discussion that compounds 8–10 were AIE and AIEE active, respectively. It is speculated that compound 7 shows AIE and ACQ simultaneously such that



Fig. 4. Fluorescent character of compound **8**. (a) PL spectra in  $CH_2Cl_2/EtOH$  mixture  $(10^{-5} \text{ M})$  excited at 300 nm. (b) Quantum yield in  $CH_2Cl_2/EtOH$  mixture. (c) Photograph taken under UV illumination.



Fig. 5. Fluorescent character of compound 9. (a) PL spectra in CH<sub>2</sub>Cl<sub>2</sub>/EtOH mixture (10<sup>-5</sup> M) excited at 300 nm. (b) Quantum yield in CH<sub>2</sub>Cl<sub>2</sub>/EtOH mixture. (c) Photograph taken under UV illumination.



Fig. 6. Fluorescent character of compound 10. (a) PL spectra in  $CH_2Cl_2/EtOH$  mixture ( $10^{-5}$  M) excited at 300 nm. (b) Quantum yield in  $CH_2Cl_2/EtOH$  mixture. (c) Photograph taken under UV illumination.

its emission spectrum is much broader than those of the other compounds. The signal at 520 nm in Figure 6 (a) was probably caused by the acetyl group in compound **10**.

### CONCLUSIONS

In conclusion, we have synthesized four new *N*-substituted phthalimide derivatives (7–10) by the traditional Suzuki coupling method. The PL spectra demonstrated that compound 7 presented both AIE and ACQ behaviors; however, compounds 8–10 displayed AIE and AIEE properties, respectively. Since compound 7 could reveal excellent intermolecular  $\pi$ – $\pi$  stacking (Figure 2), it exhibited different fluorescent properties from those of compounds 8–10. Further studies of the electroluminescence properties of compounds 7–10 are in progress. The performances of these compounds on the application to OLEDs are also under investigation.

# EXPERIMENTAL

#### Materials

Phthalic anhydride and  $K_2CO_3$  were purchased from Showa Co. Ltd. and used as received. Periodic acid, *p*-toluidine, iodine, and biphenyl-4-boronic acid were purchased from Acros and used without further purification. 4-Aminoacetophenone and *n*-butylamine were from Alfa Aesar and used as received. Palladium(II) acetate was from Lancaster Synthesis. Dimethyl formamide was obtained from TEDIA as ACS-grade solvent.

All reactions were carried out in a round-bottom flask or a sealed tube. When necessary, moisturesensitive solvents were dried in accordance with standard procedures and transferred via a syringe. Crude product solutions were dried on Na<sub>2</sub>SO<sub>4</sub>, filtered, and then concentrated with a rotary evaporator below 40°C at ~30 Torr. Flash column chromatography was performed using 230–400 mesh silicagel. TLC was performed on silica gel sheets with an organic binder and detected by 0.5% phosphor-molybdic acid solution in 95% ethanol. The quantum yields of the compounds in different solvents were calculated using rhodamine as reference.

#### Synthesis of tetraiodophthalic anhydride (2)

To a solution of periodic acid (5.40 g, 23.70 mmol) in concentrated  $H_2SO_4$  (60 mL) was

added iodine (18 g, 70.89 mmol). After the mixture was stirred for 30 min, phthalic anhydride (3.0 g, 20.25 mmol) was slowly added. The reaction mixture was stirred at room temperature for 24 h, after which it was heated up to 110°C and stirred for another 24 h. The mixture was cooled to room temperature and then poured onto ice-water to form a precipitate, which was then collected by suction filtration. The collected solid was thoroughly washed with methanol to remove the iodine residue. The crude product was finally recrystallized from THF/hexanes to give the pure compound 2 (5.9 g, 45%). mp 324–325°C (lit.[11, 12] 327–331°C); IR (KBr): 1859, 1815, 1784, 1776, 1755, 1520, 1329, 1292, 1187, 929, 628 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, Me<sub>2</sub>SO- $d_6$ ): no signal: <sup>13</sup>C NMR (75 MHz, Me<sub>2</sub>SO- $d_6$ ): 168.16, 139.76, 126.56, 106.62.

# General procedure for the synthesis of compounds 3–6

To a mixture of tetraiodophthalic anhydride (2, 1.0 g, 1.53 mmol) and the corresponding alkyl or aryl amine (1.84 mmol) in DMF (10 mL) was added acetic acid (50 mL). The reaction mixture was stirred at reflux (~150°C) for 24 h, and then cooled to room temperature. The mixture was poured onto ice–water to form a precipitate, which was then collected by suction filtration. The crude product was finally recrystallized to give the corresponding pure tetraiodophthalimide.

**Compound 3** Yellow solid; 83% yield; mp 244–246°C; IR (KBr): 3064, 2960, 2859, 1768, 1749, 1704, 1438, 1393, 626 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, Me<sub>2</sub>SO-*d*<sub>6</sub>):  $\delta$  3.53 (t, *J* = 7.2 Hz, 2H), 1.54–1.43 (m, 2H), 1.26–1.12(m, 2H), 0.87 (t, *J* = 7.5 Hz, 3H); <sup>13</sup>C NMR (75 MHz, Me<sub>2</sub>SO-*d*<sub>6</sub>):  $\delta$  164.25, 135.54, 134.34, 104.00, 38.23, 29.64, 19.50, 13.50. HRMS calcd. for C<sub>12</sub>H<sub>9</sub>I<sub>4</sub>NO<sub>2</sub>: 706.6812; found: 706.6815.

**Compound 4** Yellow solid; 81% yield; mp 309–310°C; IR (KBr): 3065, 1715, 1598, 1494, 1401, 1384, 1291, 1161, 1109, 1099, 889, 748, 733, 632 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, Me<sub>2</sub>SO- $d_6$ ):  $\delta$  7.55–7.38 (m, 5H); <sup>13</sup>C NMR (75 MHz, Me<sub>2</sub>SO- $d_6$ ):  $\delta$  163.49, 135.97, 134.34, 132.09, 128.89, 128.53, 127.69, 104.64. HRMS calcd. for C<sub>14</sub>H<sub>5</sub>I<sub>4</sub>NO<sub>2</sub>: 726.6499; found: 726.6495.

**Compound 5** Yellow solid; 83% yield; mp 305–306°C; IR (KBr): 3066, 2976, 2854, 1829, 1789, 1758, 1718, 1389, 629 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, Me<sub>2</sub>SO-*d*<sub>6</sub>):  $\delta$  7.33–7.24 (m, 4H), 2.36 (s, 3H); <sup>13</sup>C NMR (75 MHz, Me<sub>2</sub>SO-*d*<sub>6</sub>):  $\delta$  163.56, 138.04, 136.06, 134.30, 129.45, 129.34, 127.43, 104.66, 20.82. HRMS calcd. for  $C_{15}H_7I_4NO_2$ : 740.6656; found: 740.6647.

**Compound 6** Yellow solid; 78% yield; mp 311–313°C; IR (KBr): 3072, 2361, 1755, 1718, 1672, 1409, 1374, 1229, 1180, 1131, 1100, 883, 636 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, Me<sub>2</sub>SO-*d*<sub>6</sub>):  $\delta$  8.09 (d, *J* = 7.8 Hz, 2H), 7.58 (d, *J* = 7.8 Hz, 2H), 2.63 (s, 3H); <sup>13</sup>C NMR (75 MHz, Me<sub>2</sub>SO-*d*<sub>6</sub>):  $\delta$  197.40, 163.08, 136.23, 136.17, 136.08, 134.23, 128.73, 127.43, 104.84, 26.91. HRMS calcd. for HRMS calcd. for C<sub>16</sub>H<sub>7</sub>I<sub>4</sub>NO<sub>3</sub>: 768.6605; found: 768.6608.

# General procedure for the synthesis of compounds 7-10

A mixture of biphenyl-4-boronic acid (0.32 g, palladium(II) 1.62 mmol). acetate (2.24 mg, 0.013 mmol),  $K_2CO_3$  (0.3 g, 2.16 mmol), and the corresponding tetraiodophthalimide (0.71 mmol) in DMF (5 mL) in a sealed tube was heated at 115°C for 60 h. After the reaction mixture was cooled to room temperature, saturated aqueous NaCl solution (10 mL) was added. The mixture was extracted with CH<sub>2</sub>Cl<sub>2</sub>  $(30 \text{ mL} \times 3)$ . The organic layers were combined, dried (Na<sub>2</sub>SO<sub>4</sub>), and concentrated under reduced pressure. The residue was purified with flash column chromatography (*n*-hexanes:EtOAc = 8:1) to give the corresponding tetra-(biphen-4-yl)phthalimide.

**Compound** 7 Yellow solid; 36% yield; mp 282–283°C; IR (KBr): 3072, 2361, 1755, 1718, 1672, 1409, 1374, 1229, 1180, 1162, 1100, 883, 636 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, Me<sub>2</sub>SO-*d*<sub>6</sub>):  $\delta$  7.59–6.86 (m, 36 H), 3.60 (t, *J* = 7.5 Hz, 2H), 1.64–1.60 (m, 2H), 1.36–1.29 (m, 2H), 0.90 (t, *J* = 7.5 Hz, 3H); <sup>13</sup>C NMR (75 MHz, Me<sub>2</sub>SO-*d*<sub>6</sub>):  $\delta$  167.61, 147.66, 140.75, 140.36, 140.05, 139.28, 138.9, 137.30, 134.76, 131.46, 130.65, 128.76, 127.35, 127.17, 126.92, 126.13, 125.82, 30.69, 20.39, 13.80. ESI-MS calcd. for C<sub>60</sub>H<sub>46</sub>NO<sub>2</sub>: 812.3523; found: 812.3520.

**Compound 8** Yellow solid; 34% yield; mp 337–338°C; IR (KBr): 3070, 3026, 2922, 1769, 1722, 1693, 1680, 1598, 1513, 1485, 1366, 1310, 1156, 1114, 1070, 748, 729, 695 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, Me<sub>2</sub>SO-*d*<sub>6</sub>):  $\delta$  7.56–7.18 (m, 39 H), 6.90 (d, *J* = 8.4 Hz, 2H); <sup>13</sup>C NMR (75 MHz, Me<sub>2</sub>SO-*d*<sub>6</sub>):  $\delta$  166.49, 148.27, 140.71, 140.31, 140.14, 139.89, 139.02, 138.42, 137.20, 134.59, 131.79, 131.44, 130.66, 128.89, 128.75, 128.08, 127.37, 127.15, 126.92, 126.18, 125.87. ESI-MS calcd. for C<sub>62</sub>H<sub>42</sub>NO<sub>2</sub>: 832.3210; found: 832.3247.

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Compound 9 Yellow solid; 35% yield; mp 333-334°C;IR (KBr): 3065, 2921, 2360, 1771, 1720, 1600, 1513, 1487, 1371, 1124, 1007, 697 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, Me<sub>2</sub>SO-d<sub>6</sub>): δ 7.56–6.89 (m, 40 H), 2.33 (s. 3H): <sup>13</sup>C NMR (75 MHz, Me<sub>2</sub>SO- $d_6$ ):  $\delta$  166.61. 148.11, 140.68, 140.25, 140.04, 139.76, 138.90, 137.92, 137.19, 134.55, 131.42, 130.65, 130.20, 129.49, 128.73, 128.08, 127.66, 127.34, 127.13, 126.88, 126.68, 126.40, 126.13, 125.84, 21.27. ESI-MS calcd. for C<sub>63</sub>H<sub>44</sub>NO<sub>2</sub>: 846.3367; found: 846.3406.

**Compound 10** Yellow solid; 36% yield; mp 252–254°C; IR (KBr): 3066, 3028, 2358, 1723, 1681, 1599, 1516, 1487, 1359, 1268, 1178, 838, 764, 696 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, Me<sub>2</sub>SO-*d*<sub>6</sub>):  $\delta$  7.87–6.98 (m, 40 H), 2.47 (s, 3H); <sup>13</sup>C NMR (75 MHz, Me<sub>2</sub>SO-*d*<sub>6</sub>):  $\delta$  196.94, 167.94, 143.73, 141.99, 141.21, 140.30, 140.10, 139.70, 139.59, 139.47, 138.73, 138.22, 137.78, 137.48, 135.24, 132.36, 132.08, 131.00, 130.68, 129.75, 129.39, 129.31, 128.40, 127.85, 127.16, 126.93, 126.72, 126.64, 126.49, 126.11, 125.66, 125.31, 119.09, 26.86. ESI-MS calcd. for C<sub>64</sub>H<sub>44</sub>NO<sub>3</sub>: 874.3316; found: 874.3347.

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### **Supporting information**

Additional supporting information is available in the online version of this article.

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