

Cite This: Org. Lett. XXXX, XXX, XXX–XXX

# Successive Annulation to Fully Zigzag-Edged Polycyclic Heteroaromatic Hydrocarbons with Strong Blue–Green Electroluminescence

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**S** Supporting Information

ABSTRACT: A Brønsted-acid-promoted alkyne benzannulation approach was developed to synthesize the aminosubstituted dibenze[a,j]anthracence derivatives in excellent yields, which were directly converted to fully zigzag-edged polycyclic heteroaromatic hydrocarbons via a nitrogendirected electrophilic borylation. As the dopant in a blue-



green electroluminescent device, the resulted compound exhibited relatively high stability.

 ${f B}$  ecause polycyclic aromatic hydrocarbons (PAHs) are well-known as the key units and modeling compounds of conjugated polymers or graphene analogues, the construction of new PAH structures not only enlarges the organic semiconductor regimes, but also helpfully builds up the  $\pi$ extended materials. Typically, the electronic structure of PAHs were finely tailored via the molecular size, edge configuration, or embedding heteroatoms.<sup>1</sup> A zigzag-edged structure always possesses open-shell characters in the ground state, and metallic behaviors as theoretical evaluation,<sup>1c,2</sup> which have great potentials in spintronic devices.<sup>3</sup> Some annulation approaches to such types of structures have been widely developed, including Scholl reaction,<sup>1b,4</sup> Diels–Alder cycliza-tion,<sup>5</sup> Olefin metathesis cyclization,<sup>6</sup> etc. Of these, Lewis-/ Brønsted-acid-catalyzed alkyne benzannulation is a simply performed method.<sup>7</sup> Exploring cyclization to efficiently extend a structure with the retained functional groups (e.g., amino group), were extremely valuable for the formation of muchcomplex PAHs.

Replacing a C=C unit with an isoelectronic B–N moiety remarkably changes the electronic structure but exerts less influence on the geometric skeleton for a PAH molecule.<sup>8</sup> The nitrogen-directed electrophilic borylation has become an efficient cyclization to achieve an enlarged and fused skeleton. Given that the intrinsically different electronegativities of B and N atoms, embedding such polar BN units into a molecular backbone enables finely tuning the molecular physical properties, such as stacking alignments in solid states, optical absorption, and luminescence, which is extremely desirable for high performance in electronic devices.<sup>9</sup> Our group previously reported the first blue organic light-emitting diodes (OLEDs) based on B,N-heteroacene,<sup>10</sup> and Hatakeyama et al. developed a B,N-PAHs-based fluorescence and phosphorescent

OLEDs.<sup>11</sup> However, the corresponding investigation is in the early stages. Recently, our group and Hatakeyama et al. independently reported NBN-type heteroacenes.<sup>12</sup> Moreover, Wang et al. and Feng et al. constructed the extended structures via modification at the N atoms.<sup>13</sup> Currently, our effort focuses on the modification of the edge configuration of such NBNtype molecules, and further explores their electroluminescent behaviors.

In this work, we established an alkyne benzannulation via Brønsted acid activation to efficiently synthesize aminosubstituted dibenze [a,j] anthracence derivatives, which were further fused into a series of NBN-embedded PAHs via aminodirected electrophilic borylation. The resulted fully zigzagedged structures were clearly confirmed by NMR spectra, HRMS, and single-crystal analysis. Such PAHs exhibit highly reversible oxidation behaviors and strong luminescence. The blue-green electroluminescent device of such NBN-embedded PAHs was also investigated.

Starting from the key precursor 4',6'-bis(phenylethynyl)-[1,1':3',1''-terphenyl]-2,2''-diamine (3a) (detailed in the Supporting Information (SI)), the alkyne benzannulation of 3a was investigated (Scheme 1). By using a transition-metal Lewis acid catalyst, the main product with the imine-linked seven-member rings (4a-1) was isolated as a white solid in yields of 25%-55% (see Table S1 in the Supporting Information, entries 1-3). In a plausible cyclization mechanism (Figure S1 in the Supporting Information), as an example, a  $\pi$ -complex might be formed by binding the two Ag cations separately to the acetylene moieties of 3a. Following the conventional insertion, elimination, and isomerization

Received: April 28, 2019

Scheme 1. Synthetic Route to Dibenze[a,j]anthracence Derivatives



processes, **4a-1** was obtained.<sup>14</sup> When using ICl, the unresolved mixtures were formed (Table S1, entry 4).

In another way, Brønsted acid was examined for activating the alkyne benzannulation of 3a. Upon the treatment of 3a with methylsulfonic acid (CH<sub>3</sub>SO<sub>3</sub>H) or trifluoroacetic acid (TFA) (2-20 equiv) at room temperature for 12 h, there is no annulated product formed, and only 3a was collected after workup with K<sub>2</sub>CO<sub>3</sub> aqueous solution. Under stronger conditions with trifluoromethanesulfonic acid (TfOH), a 2fold annulation smoothly occurred and resulted in compound 4a in a excellent yield. In order to make an insight into such Brønsted acidity-dependent cyclization, we first treated 3a with CH<sub>3</sub>SO<sub>3</sub>H or TFA, even in large excess amount, only leading to the protonation of amino groups of **3a**, verified by <sup>1</sup>H NMR spectra analysis (see Figure S2 in the Supporting Information), further treating such protonated 3a with catalytic-scale TfOH, 4a was quantitatively formed in 1 h (see Figure S3 in the Supporting Information). This phenomenon could be attributed to protonating the amino groups of 3a directly, which decreased the electronic density of the molecular skeleton; thus, a weaker Brønsted acid could not further drive an electrophilic cyclization. Under the same annulation conditions, the intermediates 3b and 3c with the different alkyl-substituted amino groups were also smoothly converted to 4b and 4c in good to high yields, respectively (Scheme 1). The chemical structures of all the resulting new compounds were fully characterized by <sup>1</sup>H, <sup>13</sup>C NMR spectra and HRMS spectra analysis. Notably, in <sup>1</sup>H NMR spectra of 4a-1 (in DMSO- $d_6$ ), the two multiple peaks centered at ~2.7 and 4.6 ppm are arising from the two methylene protons in each sevenmembered ring. Their different chemical environments are likely due to the conformational isomerism, which were further clarified by the temperature-dependent <sup>1</sup>H NMR spectra analysis (in DMSO- $d_6$ ) (see Figure S4 in the Supporting Information). The intensities of the two peaks gradually disappeared, and a new peak at ~3.0 ppm appeared with increasing temperature. Such phenomena were also found in some previous reports.<sup>15</sup> In  ${}^{13}$ C NMR spectra of 4a-4c, the acetylenic resonances at ~93 and 87 ppm in their precursors, 3a-3c, disappeared, essentially manifesting the successful 2fold alkyne benzannulation.

Apparently, the resulting 4a-4c bear two amine groups at the bay positions of dibenze [a,j] anthracence backbone, allow for substantial further modification. Here, we attempt to construct a fully zigzag-edged structure through the nitrogen-

directed borylation. As an example, 4a was combined with 3.0 equiv of BCl<sub>3</sub> in *o*-dichlorobenzene and heated up to 120 °C for a 12-h reaction. A NBN-embedded polycyclic aromatic hydrocarbons 5a were efficiently achieved in an extremely high yield. Similarly, the compounds 5b and 5c were also obtained as yellow solid in ~90% yield. Notably, trimethylsilyl (TMS) group as the directing and leaving groups is not required for this borylation (Scheme 2), which is likely attributed to the

Scheme 2. Synthesis of the Fully Zigzag-Edged PAHs 5a-5c



fact that the  $\pi$ -extended backbones of **4a**–**4c** are favorable for an electrophilic attack. Meanwhile, their fused skeletons definitely confine the rotation of the phenyl moieties bearing amino groups, which is also beneficial to the above cyclization. Their chemical structures were essentially confirmed by <sup>1</sup>H NMR, <sup>13</sup>C NMR, <sup>11</sup>B NMR, and HRMS spectra analysis. One set of simple chemical shifts in <sup>1</sup>H, <sup>13</sup>C NMR spectra revealed their highly symmetric structures. In <sup>1</sup>H NMR spectrum, the chemical shift of the amino hydrogen in the aromatic regions for **5a**, clearly revealed the NBN segment in a typical aromatic structure.

The structure of this type of molecule was further identified by X-ray crystallographic analysis. Single crystals of **5b** were achieved from the THF solution through slowly evaporating the solvent. It shows a relatively planar main backbone with a fully zigzag-edged periphery (Figure 1). The largest dihedral



Figure 1. Single-crystal structure and packing pattern of **5b** (hydrogen atoms omitted for clarity).

angle of 7.48° was observed among the fused rings. The bond lengths of B–N (1.445, 1.442 Å) are much shorter than a typical B–N single bond (1.58 Å), and similar to the B–N bond length (1.403 Å),<sup>10a,12b</sup> indicating the confirmation of double-bond character. Its crystal packing pattern consists of the slipped stacking columns in parallel arrangement. In each column, the adjacent molecules are alternatingly orientated toward reverse directions in a edge-to-edge alignment. The lack of  $\pi$ – $\pi$  stacking interactions is clearly defined by the packing of the aromatic ring above the alkyl chains of the neighboring molecule.

By contrast, the  $\pi - \pi$  distance between the neighboring layers, with the distances of ~3.4–3.6 Å, were found in the recent reported NBN-containing molecules.<sup>13</sup> These intrinsic structural characters of **5b** in the solid state seems to endow such types of molecule with some unique photophysical properties. The optical spectra of **5a–5c** were characterized in dichloromethane (DCM) solution (see Figure 2a and Table 1). Their absorption spectra involving three groups of



**Figure 2.** (a) UV-vis absorption and fluorescence spectra of **5a**, **5b**, and **5c** in CH<sub>2</sub>Cl<sub>2</sub> (concentration =  $10^{-5}$  M), and (b) normalized cyclic voltammetry of **5a**, **5b**, and **5c** measured in CH<sub>2</sub>Cl<sub>2</sub> (0.1 M *n*-Bu<sub>4</sub>NPF<sub>6</sub>) at the scan rate of 0.1 V/s.

absorption bands exhibit vibronically split bands, which are associated with their rigid geometric structures, consistent with the crystallographic analysis. The main absorption bands in the ultraviolet regions were assigned to  $\pi - \pi^*$  transitions of their aromatic moieties. In the visible regions, the main absorption bands are arising from the transitions of HOMO to LUMO energy levels. The absorption maxima at  $\lambda_{abs} = 450$  nm for **5a** shows a slight blue shift, with respect to that of 454 nm for **5b** or **5c**, indicating that attaching alkyl chains of nitrogen atoms have less influence on their absorption behaviors. Such absorption maxima are significantly red-shifted over 50 nm, compared with the absorption maxima at  $\lambda_{abs} \approx 400$  nm for most reported NBN-containing PAHs, suggesting that **5a**-**5c** possess much larger  $\pi$ -extended backbones.

The emittance at  $\lambda_{em} = 459$  nm for 5a is slightly blue-shifted than those of 5b (464 nm) and 5c (474 nm). These fluorescence profiles show good mirror images of their corresponding absorption bands from 390 nm to 477 nm. The relatively small Stokes shifts of  $\sim$ 500 cm<sup>-1</sup> reflect their less-deformed geometric structures either in the ground or excited states, associated with their intrinsic rigid skeletons. Furthermore, these compounds exhibited intense blue-green emission with good fluorescence quantum yields ( $\Phi_{PL}$ ) (from 0.43 to 0.56), which is remarkably larger than the most of the reported B,N-embedded PAHs,<sup>11b,c,13a,16</sup> probably attributed to the backbones without efficient  $\pi$ -stacking interactions. 5a has a longer fluorescence lifetime than 5b or 5c (Table 1), probably due to the increased nonradiative fluorescence decay in the presence of longer alkyl chains.<sup>17</sup> Their absorption bands in thin films are similar to those in solution and showed the slight bathochromic shifts of  $\sim 8$  nm, relative to those in solution (Figure S6 in the Supporting Information). While, their emitting bands in film show red shifts of 48 nm for 5a, and 40 nm for 5b and 5c, respectively. Such phenomena manifested that the intermolecular interactions in solid states

Table 1. Photophysical and Electrochemical Data for 5a-5c

for such types of molecules strongly affect their excited states but not ground states, which also were observed in some other PAHs.<sup>18</sup> The fluorescence quantum yields of 5a-5c are much lower than those in solution (Table 1), which is probably due to the typical aggregation-caused quenching for a luminogen in solid state.

The electrochemical behaviors of 5a-5c were investigated by cyclic voltammetry (CV) (Figure 2b, Table 1). The reversible one-electron oxidation processes were observed at the redox peaks of 0.37 V for 5a, <0.46 V for 5b, and <0.47 V for 5c. After more than 10 cycles of cyclic voltammetry performance, there is no obvious change for these CV profiles, indicating that the generated radical cations are relatively stable under the experimental conditions (see Figure S8 in the Supporting Information). Meanwhile, those reported NBNcontaining PAHs always showed quasi-reversible or irreversible oxidation processes. Accordingly, the highest occupied molecular orbital (HOMO) energy levels of these compounds were calculated from the onsets of the first oxidation potentials as 5a (-5.17 eV) < 5b (-5.26 eV) < 5c (-5.27 eV). Such order is in good agreement with those results in our previous work (Table 1). Their density functional theory (DFT) calculations revealed that the HOMO and LUMO orbitals were almost located over the entire main backbones. A significant contribution from two N atoms but not B in HOMO, and the B atom, but relatively small parts of N atoms in LUMO, were observed (see Figure S9 in the Supporting Information, as well as Table 1).

Given the promising emitting properties, the electroluminescent (EL) behaviors of **5b** as an example, was explored as the emitting dopant for the fabrication of an EL device with the structure of ITO/MoO<sub>3</sub>/NPB/**5b**/Alq<sub>3</sub>/Liq/Al (Figure 3a;



Figure 3. Electroluminescent (EL) device properties: (a) the structure, luminescent spectra and CIE coordinates (inset); (b) voltage-current/brightness plots.

the details of the device fabrication are given in the Supporting Information). The EL spectra of the device displays two emission peaks at 471 and 500 nm (see Figure 3), typically belonging to blue–green light with CIE (0.22, 0.42). Without device optimization, a low turn-on voltage of 2.8 V was observed. Moreover, a maximum luminescence of 8095 cd/m<sup>2</sup> was achieved at 8.0 V, suggesting that the device, on the basis

	UV-vis <sup>a</sup>		Fluorescence			Electrochemistry			Calculation <sup>c</sup>	
	$\lambda_{\max}$ (nm)	$\log \varepsilon$	$\lambda_{\max}$ (nm)	$\tau$ (ns)	$\Phi_{\mathrm{PL}}{}^{b}$	$E_{\rm HUMO}^{c}$ (eV)	$E_{\rm LUMO}^{d}$ (eV)	$E_{ox}^{e}(V)$	HOMO (eV)	LUMO (eV)
5a	450	4.43	459	7.9	0.56/0.06	-5.17	-2.47	0.37	-4.89	-1.49
5b	454	4.52	464	6.7	0.49/0.04	-5.26	-2.61	0.46	-4.85	-1.46
5c	454	4.59	467	6.2	0.43/0.05	-5.27	-2.62	0.47	-4.81	-1.42

 ${}^{a}10^{-5}$  M in dichloromethane (DCM) at 298 K. <sup>b</sup>Absolute values. <sup>c</sup>Calculated from the first oxidation peak using  $E_{HOMO} = -E_{ox} - 4.80$  eV. <sup>d</sup>Estimated according to  $E_{LUMO} = E_{HOMO} + E_{g}$ . <sup>e</sup>The first oxidation potential, estimated using the tangent method.

## Table 2. Electroluminescent (EL) Device Performance

CIE

					C	ш
voltage (V)	luminescence, $L (cd/m^2)$	luminous efficiency, $\eta_{\rm c}~({\rm cd/A})$	power efficiency, $\eta_{\rm p}~(\rm lm/W)$	external quantum efficiency, EQE (%)	x	у
2.8	0.64	0.90	1.01	0.43	0.24	0.42
4.0	47.88	1.16	0.91	0.59	0.21	0.40
7.0	2678.00	1.32	0.59	0.56	0.22	0.42
8.0	8095.00	1.10	0.43	0.49	0.22	0.42

of such types of molecule, has excellent stability, even under a higher working voltage, thereby delivering a relatively high fluorescent brightness (Table 2). The maximum external quantum efficiency of 0.59% is still much lower than the theoretically evaluated value of ~5% for an organic emitting material with singlet excitation used, which might be further improved by the rational molecular modification and optimization of device fabrication.

In conclusion, we have constructed a highly efficient alkyne benzannulation access to prepare the amino-substituted dibenze[a,j]anthracence derivatives upon the activation of Brønsted acid. These derivatives were concisely transformed to new types of NBN-embedded PAHs through a nitrogendirected electrophilic borylation. The fully zigzag-edged structure for such types of molecules was essentially confirmed by single-crystal X-ray analysis. Their strong photoluminescence and highly reversible oxidation processes were also clearly manifested. The blue–green electroluminescent device using **Sb** as a dopant, exhibits a low relative low turn-on voltage of 2.8 V, and a strong luminescence up to 8095 cd/m<sup>2</sup>, demonstrating its excellent semiconducting characters. The reported successive annulation might be widely suitable for the formation of many complex PAHs with promising properties.

### ASSOCIATED CONTENT

# **Supporting Information**

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.or-glett.9b01487.

Spectroscopic data for new compounds, experimental and computational details, crystal structural data (PDF)

## **Accession Codes**

CCDC 1912074 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data\_request/cif, or by emailing data\_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: + 44 1223 336033.

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#### Notes

The authors declare no competing financial interest.

## ACKNOWLEDGMENTS

We thank Shanghai Committee of Science and Technology for financial support within Project No. 16JC1400703. Furthermore, this work was financially supported by National Natural Science Foundation of China (Nos. 21574080, 21774072, 21720102002). Open Project Program of the State Key Laboratory of Inorganic Synthesis and Preparative Chemistry (No. 2019-01, Jilin University). We acknowledge the Instrumental Analysis Center of SJTU for the NMR, singlecrystal, and HRMS measurements.

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