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# Synthesis of Fused *B*-containing Heterocyclic Compounds and Studied their Relevent Optical Properties

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**Abstract:** A series of fused 4,5-borazaropyrrolo[1,2-a]quinolines have been prepared from potassium organotrifluoroborates and substituted 1-(2-aminophenyl)pyrroles *via* C-B and N-B bonds formation directly. Target products were obtained in moderate to high yields under mild condition. Optical properties of some products were investigated by UV/Vis spectroscopy and spectrofluorometry. Moreover, the influence of structural modification on the spectroscopic and electronic properties was studied by DFT calculation.

Azaborines, as *B*-containing *N*-heterocyclic compounds,<sup>[1]</sup> have attracted much attentions in material science and medicinal chemistry.<sup>[2]</sup> Furthermore, azaborines with *B*-*N* unit as valuable building blocks exhibit a novel behavior in material science and biomedical research field,<sup>[3]</sup> such as fluorescent dyes<sup>[4]</sup> and biological labeling.<sup>[5]</sup> Moreover, the *B*-*N* unit has a strong dipole moment, and replacing C=C with *B*-*N* unit decreases the HOMO-LUMO gap, which often results in chemiluminescent materials.<sup>[6]</sup> So the photophysical properties of *B*-*N* containing heterocyclic compounds are distinctly different from those corresponding all-carbon aromatic systems. Parvez reported that phenanthrene analogues with *B*-*N* moieties could afford blue light emission with good quantum efficiencies.<sup>[7]</sup>

In past several decades, the synthesis of B-N containing heterocyclic compounds had become a popular strategy to increase structural diversity and provided new isosteric aromatic systems. Many procedures for azaborines construction have reported.<sup>[8]</sup> been ln 1959, the first example borazaronaphthalene was prepared by Dewar.<sup>[9]</sup> Subsequently, and Rombouts<sup>[11]</sup> respectively synthesized 2,1-Liu<sup>[10]</sup> borazaronaphthalenes and 2,1-borazaroquinolines which were performed with strict conditions or multiple steps (Scheme 1). Subsequently, significant progresses in this field had been achieved by the groups of Paetzold, Ashe, Piers, Cui, and so on.<sup>[12]</sup> Recently, Molander<sup>[13]</sup> described a new method to svnthesize borazaronaphthalenes from substituted 2aminostyrenes and potassium organotrifluoroborates (Scheme 1). However, the plain and gentle processes for B-N containing heterocycles formation were still very rare. Herein, we have developed a metal-free method to synthesize fused substituted 4,5-borazaropyrrolo[1,2-a]quinolines with 1-(2-aminophenyl)pyrroles and potassium organotrifluoroborates.

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Scheme 1. Synthesis of *B*-substituted azaborines.

With high interest in construction of azaborines, 1-(2-amino phenyl)pyrrole 1a and potassium phenyltrifluoroborate 2a were chosen as model substrates for our studies. In the presence of SiCl<sub>4</sub>, Et<sub>3</sub>N, and 1:1 mixture of PhMe/CH<sub>3</sub>CN at ambient temperature, the desired product 3aa was generated in 65% yield (Table 1, entry 1). Next, when the temperature was improved to 80 °C and the quantity of the Et<sub>3</sub>N was improved to 1.5 equiv. the vield was increased to 80% (entry 2). The solvents were evaluated and PhMe was proved to be the best solvent for this process with the vield of 89% (entry 8). It should be noted that the vield was slightly decreased with 1.5 equiv of SiCl<sub>4</sub> and 2.0 equiv of Et<sub>3</sub>N (entry 9). To our delight, the desired product 3aa was obtained in 96% yield when the reaction was carried out at 60 °C (entry 10). Subsequently, reactions were respectively conducted in O2 and Ar condition with yields of 92% and 93% (entry 11-12). After further scanning other fluorophiles, the improvements of the yield were not detected (entry 13-14). So the optimized reaction system was established as Table 1, entry 10.

With the optimized condition in hand, the scope of 1-(2aminophenyl)pyrrole was examined firstly and results were substituted illustrated in Table 2. A variety of 1-(2potassium aminophenyl)pyrroles reacted with phenyltrifluoroborates smoothly and furnished the desired products in high yields. Moreover, substituted groups, such as -Me, -OMe, -Cl, -F, on the aryl ring did not have significant influence on the formation of products 3ba-3ha. However, lower yield of 3ia was observed because of the strong electornwithdrawing nitrile group.

Then the synthesis of azaborines was further expanded to the range of substituted potassium aryltrifluoroborates (Table 3). The potassium aryltrifluoroborates bearing electron-donating and electron-withdrawing groups were tolerated well under the

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Table 1. Optimization of reaction conditions<sup>[a]</sup>.



Entry	Solvent	Fluorophile Et₃N (equiv) (equiv)		Temp (°C)	Yield (%) <sup>[b]</sup>
1	PhMe/CH₃CN (1:1)	SiCl <sub>4</sub> (2.0)	1.0	25	65
2	PhMe/CH <sub>3</sub> CN (1:1)	SiCl <sub>4</sub> (2.0)	1.5	80	80
3	CH₃CN	SiCl <sub>4</sub> (2.0)	1.5	80	84
4	Xylene	SiCl <sub>4</sub> (2.0)	1.5	80	56
5	PhCI	SiCl <sub>4</sub> (2.0)	1.5	80	72
6	DMF	SiCl <sub>4</sub> (2.0)	1.5	80	Trace
7	1,2-DCE	SiCl <sub>4</sub> (2.0)	1.5	80	34
8	PhMe	SiCl <sub>4</sub> (2.0)	1.5	80	89
9	PhMe	SiCl <sub>4</sub> (2.0)	2.0	80	80
10	PhMe	SiCl₄ (2.0)	1.5	60	96
11	PhMe	SiCl <sub>4</sub> (2.0)	1.5	60	93 <sup>[c]</sup>
12	PhMe	SiCl <sub>4</sub> (2.0)	1.5	60	92 <sup>[d]</sup>
13	PhMe	BCl <sub>3</sub> (1 M in CH <sub>2</sub> Cl <sub>2</sub> , 2.0)	1.5	60	73
14	PhMe	BF <sub>3</sub> ·OEt <sub>2</sub> (2.0)	1.5	60	40



Table 3. Scope of organotrifluoroborates.

> SiCl<sub>4</sub> (2 equiv) Et<sub>3</sub>N (1.5 equiv) PhMe

[a] Reaction conditions: **1a** (0.3 mmol), **2a** (0.33 mmol), Et<sub>3</sub>N (0.45 mmol), fluorophile (0.6 mmol), solvent (2 mL), 4 h, air. [b] Yields of isolated products. [c] Under  $O_2$  condition. [d] Under Ar condition. Entry in bold highlights optimized reaction conditions, and the reaction time was monitored by TLC.





60 °C, 4 h, air 1 3 MeO tBi NH ŇН ŇН 3dg (61%) 3bd (73%) 3ci (99%) 3bb (97%) 3fj (98%) 3fh (96%) 3ed (87%) 3eb (95%) QMe NH С 4aa (trace) 3he (87%)

optimized condition, generating desired products in moderate to good yields (**3ab,3ad-3an**). Obviously, the steric hindrance had a distinct impact on the reaction and the desired compound **3ac** was obtained with only 30% yield. Substrate **2k** with a strong electron-withdrawing group was not transformed to the desired product, which may be due to the electronic effect. The thienyl-substituted potassium trifluoroborate was also palyed well in this

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transformation, generating **3al** in 64% yield. What's more, aliphatic potassium trifluoroborates **2m-2o** were also explored in this process and gave products in 30-88% yields (**3am-3ao**).

In order to expand the substrate scope, further investigations were performed to evaluate substituted 1-(2-aminophenyl)pyrroles and potassium organotrifluoroborates (Table 4). The transformations were proceeded soomthly and desired products were obtained with high yields, especially **3bb**, **3ci**, and **3fj**. When more challenging substrate 2-(1*H*-indol-1-yl)aniline was subjected to standard condtions, only trace amount of **4aa** was detected.

On the basis of our experiments and previous reports,<sup>[14]</sup> a plausible mechanism for the approach is outlined in Scheme 2. Firstly, the potassium phenyltrifluoroborate is transformed to corresponding active organodichloroborane I with SiCl<sub>4</sub>. Then, intermediate II is formed from the reaction of **1a** and I via *B-N* bond formation.<sup>[15]</sup> Finally, the intermediate II is converted to desired product **3aa** by attack of pyrrole double bond to boron.

To probe the optical properties of these products, we chose product **3fj** as model substrate for this experiment. Firstly, spectral characteristics of **3fj** were investigated in several solvents: PhMe, THF, DCM, MeCN, and EtOH. The result was summarized in Figure 1. As demonstrated in Figure 1,



Scheme 2. Proposed mechanism.







Figure 2. Normalized absorption and fluorescence emission spectra of complexes 3aa, 3am, 3bd, 3ca, 3ci, 3ed, 3fj, and 3ia in MeCN at 10.0  $\mu$ M.

Table 5. Optical properties of 3aa, 3am, 3bd, 3ca, 3ci, 3ed, 3fj, 3ia and 5aa<sup>[a]</sup>.

Compound	λ <sub>abs</sub> (nm)	$\lambda_{em}$ (nm)	ε (M <sup>-1</sup> cm <sup>-1</sup> )	$\Phi^{[b]}$	Stokes shift (nm)
3aa	317	396	19800	0.46	79
3am	314	342	17500	0.42	28
3bd	325	400	17700	0.48	75
3ca	332	416	28700	0.55	84
3ci	332	406	25300	0.57	74
3ed	324	392	15200	0.42	68
3fj	328	434	32900	0.38	113
3ia	334	384	18900	0.39	50
5aa	357	450	33300	0.55	93

[a] Photophysical properties were measured in MeCN at 10.0  $\mu M.$  [b] The fluorescence quantum yields were calculated in MeCN using quinine sulfate in 0.05 M  $H_2SO_4$  as the standard.

the product **3fj** showed an absorption maximum at 328 nm and solvents had no influence on UV absorption band of **3fj**. Nevertheless, the increasing polarity of solvents led to a red-shift in the fluorescence emission bands. For example, the emission maximum of **3fj** in PhMe emerged at 401 nm and red-shifted to 433 nm in MeCN.

Subsequently, absorption and fluorescence emission spectra of **3aa**, **3am**, **3bd**, **3ca**, **3ci**, **3ed**, **3fj** and **3ia** were also evaluated in MeCN. As shown in Table 5 and Figure 2, compounds **3bd**, **3ca**, **3ci**, and **3fj** exhibited bathochromic shifts compared with



Figure 3. The relative energies of the frontier orbitals of 3aa, 3ca, 3ia, 3am, 3ci, 3fj, 3bd and 3ed, calculated with B3LYP/6-31G(d) level.

compound **3aa** and these compounds showed strong blue fluorescence at 400-434 nm. In absorption and emission bands, **3fj** with electron-withdrawing group showed 11 nm and 38 nm red-shifts respectively, which was consistent with a smaller HOMO-LUMO gap in **3fj** vs **3aa** in figure 3. Fluorescence quantum yields of these products were also detected and the quantum yields were moderate (0.38-0.57) in MeCN. At the same time, the photophysical properties of 4-phenylpyrrolo[1,2-*a*]quinoline **5aa**<sup>[16]</sup> were investigated which showed more obvious redshift compared with **3aa**.

To investigate the influence of structural modification on the spectroscopic and electronic properties of the boron-complexes, ground-state geometry optimizations were performed by DFT with B3LYP functional and 6-31(G) basis set by using the Gaussian 09 software package.<sup>[17]</sup> The PBE0 functional was preferred for using in time-dependent density functional theory (TD-DFT) calculations with 6-311+G(d,p) basis sets. Figure 4 and Figure 1S showed the frontier molecular orbitals of **3ia**, **3fj**,



Figure 4. The nodal patterns of HOMO-1, HOMO, LUMO and LUMO+1 of 3ia, 3fj, 3bd and 3ed, calculated with B3LYP/6-31G(d) level.

**Table 6.** Calculated excited wavelength ( $\lambda$ ) and oscillator strengths (f) for component **3fj** of selected transition energies.

Compound	State <sup>[a]</sup>	E(eV)	$\lambda$ (nm) $f^{[b]}$	Orbitals (coefficient) <sup>[c]</sup>
3fj	S <sub>1</sub>	3.7787	328.11 0.2023	H>L(97%)
	S <sub>2</sub>	4.1886	296.00 0.1317	H-1>L(42%), H>L+1(52%)
	S <sub>3</sub>	4.4603	277.97 0.3126	H-1>L(52%), H>L+1(41%)

[a] Excited state. [b] Oscillator strength. [c] MOs involved in the transitions. Calculation was performed at the PBE0/6-311+G(d,p) level in MeCN.

3bd, 3ed, and 3aa, 3ca, 3am, 3ci, respectively. As can be deduced from Figure 4, for these complexes, LUMO electronic distribution are almost identically global, whereas HOMO electronic distribution is mainly on the large π-system. In case of 3fj, which (trifluoromethyl)benzene group is introduced at the boron atom, HOMO is localised on the  $\pi$ -system and HOMO-1 electronic distribution could extend to the (trifluoromethyl)benzene group. And the HOMO-LUMO energy of 3fj is the lowest from Figure 3. For complex 3fj, the transitions with relatively stronger oscillator strength between the first and third are mixed HOMO→LUMO and HOMO-1→LUMO, HOMO→ LUMO+1 transitions. It is also apparent that the third lowest transition is rather close-lying in energy/wavelength.

Multicenter bond order<sup>[18]</sup> could be used to evaluate aromaticity and its positive value indicates that the ring would have aromaticity (Table S1). The computational results show that the values of ring 1 and 3 are positive, suggesting the fivemembered ring 1 and six-membered ring 3 are aromatic nucleus with conjugated  $\pi$  electronic structure. While, the ring 2 is also one aromatic ring with small positive value. Finally, the three rings (ring 1, 2 and 3) would form one large conjugate structure.

In conclusion, we have described a synthetic approach to 4,5borazaropyrrolo[1,2-a]quinolines via cyclization of 1-(2aminophenyl)pyrroles and organotrifluoroborates in a facile procedure. In this transformation, a wide functional group can be tolerated and desired products are obtained in moderate to good yields. Meanwhile, the optical properties and DFT calculation about *B*-containing products are investigated gaining a moderate result. Furthermore, the study allows us to find that chemical modification at the boron atom leads to corresponding blueshifts or red-shifts in the absorption and emission bands. Due to attractive spectral characteristics, resultant azaborines have tremendous potential applications in biotechnology and material science.

#### **Experimental Section**

#### General procedure for the synthesis of 4,5-Borazaropyrrolo[1,2a]quinolines derivatives:

2-(1*H*-pyrrol-1-yl)aniline **1a** (1 equiv, 0.3 mmol), potassium organotrifluoroborate **2a** (1.1 equiv, 0.33 mmol), Et<sub>3</sub>N (1.5 equiv, 0.45 mmol), SiCl<sub>4</sub> (2.0 equiv, 0.6 mmol), toluene (2 mL) were added to a screw-cap vial with a stir bar. The reaction mixture was heated to 60 °C under vigorous stirring for 4 h. Then the reaction mixture was cooled to room temperature and extracted with ethyl acetate and water. The combined organic phase was dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>. The solvent

was evaporated in vacuo and the crude product was purified by column chromatography, eluting with petroleum ether/EtOAc (20:1) to afford the desired **3aa**.

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A new *B*-containing heterocyclic compound ! A series of azaborines were prepared through 1-(2-aminophenyl)pyrroles and organotrifluoroborates in one-pot manner. The optical properties of these azaborines were investigated and the attractive photornetrics could be applied in many scientific fields.

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