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## PAPER

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### Rhodium(III)-catalyzed indole-directed carbenoid aryl C–H insertion/cyclization: access to 1,2benzocarbazoles†

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A rhodium(m)-catalyzed indole-directed aryl C–H bond carbenoid insertion cascade of 2-arylindoles with diazo compounds has been developed. This method provides a rapid access to 1,2-benzocarbazoles and isoquinoline-based polycyclic heteroaromatics with a broad range of functional group tolerance. The primary evaluation of the photoluminescence property of the novel extended  $\pi$ -systems indicated that these heteroarenes could be potentially used in the field of optoelectronic materials.

#### Introduction

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Fused polycyclic heteroaromatics (FPHs) have been referred to as privileged structures in complex natural products, pharmaceuticals and organic functional materials.1 Particularly, Nfused polycycles have attracted increasing interest for their diverse photophysical properties.<sup>2</sup> For example, carbazolebased FPHs (A, Fig. 1) could be employed to furnish green phosphorescent organic light-emitting diodes (OLED).<sup>3</sup> Pyranbased FPHs (B, Fig. 1)-containing molecular devices<sup>4</sup> exhibited red emission color. Moreover, spiro-annulated triarylaminebased polycyclic heteroarenes (C, Fig. 1)<sup>5</sup> could be used to assemble deep-blue OLED devices because these spiro molecules can provide exciton blocking and hole-injection properties due to their deep HOMO and high triplet energy level. Thus it can be seen that different core subunits generally exhibited particular optical and electroluminescent properties. However, in the past decades, although many structural skeletons have been well evaluated for their optoelectronic applications,6 1,2benzocarbazoles, especially for isoquinolinocarbazole derivative (D, Fig. 1) which possess excellent conformational rigidity property from the molecular backbone, have rarely been reported owing to the restriction of synthetic access to these compounds (D, Fig. 1).<sup>7</sup>

Recently, the 1,2-benzocarbazoles could be assembled relying on the construction of ring **A** and ring **B** through cascade

cyclic strategies of alkynes8 (Scheme 1a) and others,9 but most of these methods suffer from the disadvantage of requiring prefunctionalized alkynes or tedious steps. Therefore, developing more efficient method to construct 1,2-benzocarbazoles utilizing easily materials remains highly desirable and challenging. On the other hand, transition metal-catalyzed C-H carbenoid functionalization provides an atom- and stepeconomic approach to construct C-C bonds.<sup>10</sup> Moreover, the reactivity of acceptor moieties of carbenoids could also be fully utilized as an internal nucleophilic partner.<sup>11</sup> From the step economy as well as the functional group diversity point of view, cascade C-H carbenoid functionalization belongs to an innovative approach to complex heterocycles. For examples, we recently reported a Rh(III)-catalyzed relay Csp<sup>2</sup>-H bond carbenoid functionalization of N-aryl ketoimines, in which a nucleophilic enamine derived from imine/enamine-isomerization was trapped by an acceptor ketocarbonyl moiety of diazo compounds to assemble 1-azaphenalenes (1-APLEs). These 1-APLEs showed promising potential in optoelectronic applications due to their low-lying HOMO levels (Scheme 1b). 12 Encouraged by this work and in combination with our interest in developing cascade C-H carbenoid functionalization toward photoelectrically interesting  $\pi$ -conjugated molecule synthesis, we proposed that a 2-arylindole molecule which contains a nucleophilic directing indole group, could also possibly



Fig. 1 Selected examples of N-fused polycycles.

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Scheme 1 Aryl Csp<sup>2</sup>–H carbenoid functionalization strategies for assembling FPHs.

undergo relay carbenoid aryl C-H insertion/cyclization to furnish polycyclic heteroarenes through constructing ring **B** even ring  $C^{13}$  (Scheme 1c).

#### Results and discussion

To achieve this reaction, we commenced our study with 2-phenylindole (1a) and 2-diazo-3-oxobutanoic ethyl ester (2a) as a model reaction (Table 1). The effect of base on the cascade aryl C-H bond carbenoid insertion was first evaluated using  $[Cp*RhCl_2]_2$  (5 mol%) as a catalyst in combination with cocatalyst  $Cu(OAc)_2$  (50 mol%) and additive KOAc (2.0 equiv.) in CH<sub>3</sub>CN under an Ar atmosphere at 80 °C for 8 h (entries 1-4), and we quickly found that this catalytic system produced the major 5-ethoxycarbonyl-6-methyl-1,2-benzocarbazole 3a under basic condition.<sup>14</sup> Among them, Na<sub>2</sub>CO<sub>3</sub> provided 3a (62% yield) and isoquinoline-based carbazole 4a (13% yield) (entry 2). Although the competitive aryl C-H carbenoid insertion led to poor chemoselectivity under Rh(III)/Cu(II) system, this positive result still encouraged us to conduct further optimization of the reaction conditions for achieving excellent selectivity. Low oxidation state copper(1) salt chemoselectively afforded 3a in 84% yield under NaOAc/Na2CO3 system, and no 4a was observed (compare entries 5-9 with 10). Interestingly, switching copper(I) cocatalysts into silver salts resulted in the formation of the major product 4a when employing 2,2,2-trifluoroethanol (TFE) as solvent in the absence of  $Na_2CO_3$  (entries 11–13). In particular, Rh(III)/AgOAc/PivOH system significantly increased the chemoselectivity of this transformation at 120 °C and provided 45% yield of 4a (compare entries 11-13 with 14). When other alcohols such as *i*-PrOH were employed as solvents, no desired 3a and 4a were observed (entry 15).

With the optimized reaction conditions in hand, the scope of 2-arylindoles was then examined by employing 2-diazo-3-oxobutanoic ethyl ester (2a) as the coupling partner. As shown in Scheme 2, a variety of 2-arylindoles were smoothly converted to the corresponding multisubstituted 1,2-benzocarbazole derivatives in good to excellent yields. The substituents on the

#### Table 1 Optimization of the reaction parameters<sup>a</sup>



					Yield $(\%)^k$	
Entry	Cocatalyst	Additive	Base	Solvent	3a/4a	
1	Cu(OAc)	KOAc	_	CH <sub>2</sub> CN	0/0	
2	$Cu(OAc)_2$	KOAc	Na <sub>2</sub> CO <sub>3</sub>	CH <sub>3</sub> CN	62/13	
3	$Cu(OAc)_2$	KOAc	NaHCO <sub>3</sub>	CH <sub>3</sub> CN	53/19	
1	$Cu(OAc)_2$	KOAc	KHCO <sub>3</sub>	CH <sub>3</sub> CN	48/28	
5	CuOAc	KOAc	Na <sub>2</sub> CO <sub>3</sub>	CH <sub>3</sub> CN	61/7	
5	CuBr	KOAc	Na <sub>2</sub> CO <sub>3</sub>	CH <sub>3</sub> CN	68/5	
7	CuI	KOAc	Na <sub>2</sub> CO <sub>3</sub>	CH <sub>3</sub> CN	48/0	
3	CuCN	KOAc	Na <sub>2</sub> CO <sub>3</sub>	CH <sub>3</sub> CN	67/20	
)	CuCl	CsOAc	Na <sub>2</sub> CO <sub>3</sub>	CH <sub>3</sub> CN	58/18	
10	CuCl	NaOAc	Na <sub>2</sub> CO <sub>3</sub>	CH <sub>3</sub> CN	84/0	
1	$Ag(NTf)_2$	AcOH	_	TFE	$31/41^{c}$	
12	AgOAc	AcOH	_	TFE	28/39 <sup>c</sup>	
13	AgOAc	PhCO <sub>2</sub> H	_	TFE	29/35 <sup>c</sup>	
4	AgOAc	PivOH		TFE	$8/45^{c}$	
15	AgOAc	PivOH	_	<i>i</i> -PrOH	$0/0^{c}$	
	-					

<sup>*a*</sup> Unless otherwise noted, all the reactions were carried out using 2-phenylindole (**1a**) (0.20 mmol) and diazo compound (**2a**) (0.40 mmol) with  $[Cp*RhCl_2]_2$  catalysts (0.01 mmol, 5 mol%) in the presence of cocatalyst (copper salts: 0.10 mmol, 50 mol%; silver salts: 0.06 mmol, 30 mol%), base (0.40 mmol, 2.0 equiv.) and additives (0.06 mmol, 30 mol%) in solvent (2.0 mL) at 80 °C for 8 h under Ar in a sealed reaction tube. Followed by flash chromatography on SiO<sub>2</sub>. <sup>*b*</sup> Isolated yield. <sup>*c*</sup> The reaction temperature was 120 °C; 0.80 mmol of diazo compound **2a** was used.

indole ring and the 2-indoly phenyl ring apparently exerts different electronic effects on the reactivity (3a-3w). For example, electron-rich substituents (Me, OMe) provided the corresponding 1,2-benzocarbazoles in 61–82% yields (**3b**, **3c**, **3h**, **3l**, **3n** and **3t**). On the contrary, electron-poor substituent such as CO<sub>2</sub>Et, CN, NO<sub>2</sub> and CF<sub>3</sub> *etc.* showed poorer reactivity and afforded the target compounds in 35–58% yields (**3f**, **3g**, **3j**, **3r** and **3s**). It is worth noting that the reaction is sensitive to the substituent position in the indole rings. 5- or 6-Substituted 2-phenylindoles showed good reactivity (**3b–3j**), but 7-methyl 2-phenylindole did not furnish the desired carbazole, possibly due to steric factors (**3k**).

Moreover, the present cascade aryl C–H carbenoid insertion protocol was subsequently applied to a wide range of  $\alpha$ -acyldiazo compounds with particular 2-phenylindole (1a) as the coupling partner. As anticipated,  $\alpha$ -alkylacyl,  $\alpha$ -alkoxymethylacyl and  $\alpha$ -homoallylic acyl substituted diazoacetates could easily enable assembly of 5-ethoxycarbonyl-1,2benzocarbazoles in 59–79% yields (3x-3az). Meanwhile, linear  $\alpha$ -acyl diazoketones produced the corresponding 1-indolo[2,1-*a*] isoquinolin-5-yl-ethanones 3bz and 3cz in 63% and 78% yields, respectively, but cyclic  $\alpha$ -acyl diazoketones produced the desired 11*H*-benzo[*a*]carbazole 3dz in 63% yield. If the indole



Scheme 2 Substrate scope. <sup>a</sup> For the synthetic procedure of product 3: all the reactions were carried out using 2-arylindoles (1) (0.20 mmol) and diazo compounds (2) (0.40 mmol) with [Cp\*RhCl<sub>2</sub>]<sub>2</sub> (5 mol%) in the presence of CuCl (50 mol%), NaOAc (30 mol%) and Na<sub>2</sub>CO<sub>3</sub> (2.0 equiv.) in acetonitrile (2.0 mL) at 80 °C for 8 h under Ar in a sealed reaction tube. <sup>b</sup> For the synthetic procedure of product 4: all the reactions were carried out using 1 (0.20 mmol) and 2 (0.80 mmol) with [Cp\*RhCl<sub>2</sub>]<sub>2</sub> (5 mol%) in the presence of AgOAc (30 mol%), PivOH (2.0 equiv.) in CF<sub>3</sub>CH<sub>2</sub>OH (2.0 mL) at 120 °C for 24 h under Ar in a sealed reaction tube. <sup>c</sup> Isolated yield. <sup>d</sup> Cu(OAc)<sub>2</sub> (50 mol%) was used.

nitrogen atom was blocked with a methyl group, this transformation did not occur (3ez).

Next, Rh(III)/Ag(I) system catalyzed double aryl C–H carbenoid insertion cyclization between 2-arylindoles and 2-diazo-3oxobutanoic ethyl ester (2a) was further investigated. As shown in Scheme 2 (4a–4g), various methyl, alkoxy and halosubstituted 2-aryl-indoles could be smoothly employed to furnish isoquinolinocarbazoles (4a–4g) in acceptable yields (33– 56%).<sup>12</sup> It should be noted that around 5% yields of 1,2-benzocarbazoles (3a–3c, 3m, 3p, 3fz and 3gz) were observed.

Finally, the photo-physical properties, quantum yields and fluorescence lifetime spectrometer of partial molecules were explored systematically. The data of absorption and photo-luminescence wavelength, the molar absorption coefficients ( $\varepsilon$ )



Fig. 2 UV-vis absorption (a) and emission spectra (b) of the compounds 3a, 4a, 4c, 4f, 4g and 5a in dichloromethane solutions( $10^{-5}$  M).



Fig. 3 Emission color change of 3a, 4a, 4c, 4f, 4g, and 5a in dichloromethane ( $10^{-5}$  M) upon excitation at 365 nm.

and the excited state lifetimes ( $\tau$ ) as well as quantum yields ( $\Phi$ ) of the selected compounds **3a**, **4a**, **4c**, **4f**, **4g** and **5a**<sup>15</sup> in CH<sub>2</sub>Cl<sub>2</sub> are listed in Table S-1.† As shown in Fig. 2, the emission spectra of **3a** and **5a** showed a peak at 374 nm and 386 nm, respectively. In contrast, the other compounds exhibited obvious bathochromic shifts (521–539 nm) in emission.<sup>16</sup>

Their emission colour change in dichloromethane  $(10^{-5} \text{ M})$ under UV excitation (365 nm) further indicated that **3a** and **5a** showed blue light, while the others exhibited yellow light under UV excitation (Fig. 3). These results demonstrated molecules **4a**, **4c**, **4f** and **4g** were potential for furnishing white OLED devices.<sup>17</sup>

Several control experiments were performed to further explore the possible mechanism (Scheme 3). First, the H/D exchange of 2-phenylindole **1a** was conducted in a Rh(m)/ Cu(I)/CH<sub>3</sub>OD or Rh(m)/Ag(I)/AcOD system in the absence of diazo compounds, and 36% and 13% deuterium incorporation at the 2-indolylphenyl ring C2- and C6-position was observed, respectively (eqn (1) and (2)). And also, the H/D exchange of C3position from indole ring occurred possibly due to that indole C3-position possesses strong nucleophilic properties and easily attacks rhodium cation followed by metal protonation. On the contrary, no H/D exchange of *N*-methyl-2-phenylindole **1y** was observed under the same conditions (eqn (3)), this fact implied that the indole NH moiety played a key role of chelation in



Scheme 3 Preliminary mechanistic studies.



promoting 2-indolylphenyl ring C–H bond cleavage. Subsequently, treating 1,2-benzocarbazole **3a** with diazo compound **2a** under a Rh(III)Ag(I)/PivOH system did not furnish the corresponding isoquinoline-based carbazole **4a**. This control experiment also demonstrated that 1,2-benzocarbazole **3a** was not the intermediate to form product **4a** (eqn (4)). Finally, the competitive isotope effect ( $k_{\rm H}/k_{\rm D} = 2.7$ ) further indicated that 2-indolylphenyl C–H bond-breaking was possibly involved in the rate-limiting step of 1,2-benzocarbazole-forming reaction (eqn (5)) (see ESI† for more details).

A proposed catalytic cycle is shown in Scheme 4. The initial N-coordination of 2-phenylindole **1a** to Rh(m) catalyst followed by 2-indolylphenyl C-H bond activation afforded rhodacycle intermediate **A** to generate Rh-carbene species **B**, which would undergo migratory insertion and protonolysis to form rhodacycle intermediate **C** and complex **D**, respectively.<sup>18,19</sup>

Subsequently, Rh(m)/Cu(1) system catalyzed cyclization of **D** to furnish 1,2-benzocarbazole **3a**, in which Cu(1) salts played a role of Lewis acid to activate keto-carbonyl group of **F**. Meanwhile, complex **D** could also further undergo the secondary indole-directed phenyl C-H carbenoid insertion/cyclization cascade under Rh(m)/Ag catalytic system to produce isoquinoline-based carbazole **4a**.

### Conclusions

In summary, we have developed a Rh(III)-catalyzed, indoledirected phenyl C–H bond carbenoid insertion/cyclization of 2-phenylindoles<sup>20</sup> with alpha-acyl diazocompounds<sup>21</sup> for rapid assembly of 1,2-benzocarbazoles and isoquinoline-based carbazoles. Further exploring the photoelectronic performance of isoquinoline-based carbazoles demonstrated that these compounds could be further assembled within promising optoelectronic devices.

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