Organic LETTERS

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Fe-Catalyzed Novel Domino Isomerization/Cyclodehydration of Substituted 2-[(Indoline-3-ylidene)(methyl)]benzaldehyde Derivatives: An Efficient Approach toward Benzo[*b*]carbazole Derivatives

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Supporting Information

ABSTRACT: A new and efficient protocol to synthesize substituted benzo[*b*]carbazole derivatives has been demonstrated involving iron-catalyzed domino isomerization/cyclodehydration sequences from substituted 2-[(indoline-3-ylidene)(methyl)]benzaldehyde derivatives. The substrates could be easily made via Pd-catalyzed domino Heck−Suzuki coupling from 2-bromo-*N*-propargylanilide derivatives in high yields. Notably, the generality and efficiency of this two-stage domino strategy was further exemplified by the synthesis of a polycyclic benzofuran derivative.

The construction of polycyclic carbazoles has attracted considerable attention because of their remarkable biological and pharmacological activities and applications in material science.[1−3] Very recently, several benzo- and naphthocarbazole analogues have been explored as potential anticancer agents.[4] Numerous synthetic methods for the construction of these annulated carbazole ring systems have been developed in the past decades.[2] In particular, the synthesis of benzo[*b*]carbazole **A** framework and related structures received much attention because a number of biologically and pharmacologicaly active alkaloids contain this unit. For instance, naturally occurring alkaloids such as ellipticine **B** and 9-methoxyellipticine are isosteric with benzo[*b*]carbazole and have shown promising antitumor activity,[5] and few of them have been the subject of clinical trials.[6] Due to the structural similarity of 5*H*-benzo[*b*]carbazole to the ellipticines, this coplanar tetracyclic molecule appears to be an interesting lead structure for the development of novel antineoplastic agents.[7a]

For instances, carbazole **C** possesses cytostatic activity against leukemia type L 1210 cell culture,[7b] and **D** represents a potential bifunctional nucleic acid intercalating agent.[7c] Moreover, benzo[*b*]carbazole scaffolds such as **E** and related structures are widely used as molecular platforms for luminescent, hole-transporting, and host materials in organic light-emitting diodes (OLEDs).[8] Consequently, a number of useful synthetic strategies are available now for the synthesis of a benzo[*b*]carbazole scaffold.[2,9]

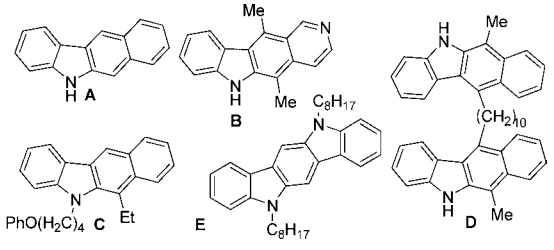
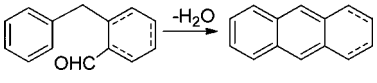


Figure 1. Structure of some important benzo[*b*]carbazole molecules.

Generally, these tetracyclic structures are commonly made from either prefunctionalized indoles[9] or naphathlene[10] derivatives. A few new approaches have been developed to access benzo[*b*]carbazole derivatives such as cycloaromatization of *N*-[2-(1-alkynyl)phenyl]ketenimines,[11a,b] intramolecular dehydro Diels−Alder reactions of *N*-(*o*-ethynyl)arylyanimides,[11c] Cu(II)-catalyzed annulation of 2-alkynylcarbazole-3-carbaldehydes,[11d] Pd(II)-catalyzed reaction between 2-alkynylbenzaldehydes and indoles,[11e] and cyclization of 2-ethynyl-*N*-triphenylphosphoranylidine anilines with α-diazoketones.[11f] However, many of these protocols have some limitations, such as lengthy synthetic sequences, harsh conditions, and low chemical yields with restricted substitution patterns. Accordingly, the development of new approaches for the construction of benzo[*b*]carbazole derivatives with specific substitution patterns is highly desirable.

The Bradsher reaction, involving Lewis or Brønsted acid catalyzed cyclodehydration of *o*-formyl/acyldiarylmethanes, offers a useful strategy to a wide array of polycyclic aromatic hydrocarbons (Scheme 1).[12]

Scheme 1. Bradsher Reaction for the Synthesis of Polynuclear Hydrocarbon

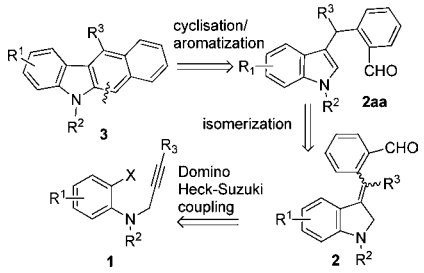


Received: February 27, 2014
Published: March 28, 2014

dx.doi.org/10.1021/ol500505k | *Org. Lett.* 2014, 16, 2166−2169

However, the difficulty of preparation, the starting materials, and harsh reaction conditions limit the synthesis of benzoannulated carbazole derivatives using this strategy. Considering our interest in the synthesis of heterocyclic compounds, we anticipated that the domino Heck–Suzuki coupling reaction[13] of 2-bromo-*N*-propargylanilide derivatives **1** with 2-formyl phenylboronic acid followed by isomerization could easily yield the Bradsher reaction precursor **2aa**. Finally, annulated carbazoles **3** could be achieved from **2aa** by a Bradsher reaction (Scheme 2). Herein, we report our results for the construction of libraries of benzo[*b*]carbazole derivatives.

Scheme 2. Synthetic Strategy to the Synthesis of Benzo[*b*]carabazole Derivatives

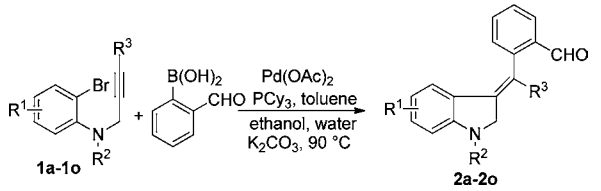


To test the above idea, we first attempted to prepare 2-[(indoline-3-ylidene)(methyl)]benzaldehyde **2a** via Heck–Suzuki coupling of 2-bromo-*N*-propargylanilide **1a** with 2-formylphenylboronic acid. Although the domino Heck–Suzuki coupling reaction is reported for the synthesis of 2-oxindoles derivatives,[13a−c] it has not been reported with 2-bromo-*N*-propargylanilide systems to synthesize 2,3-dihydroindole derivatives. Therefore, at first we began to optimize the domino Heck–Suzuki coupling between **1a** and 2-formylphenylboronic acid to find the best reaction conditions. After a great deal of study using various solvents, catalysts, bases, ligands, and temperatures (see the Supporting Information), we observed that 5 mol % of $Pd(OAc)_2$, 10 mol % of tricyclohexylphosphine (PCy_3), and 2.5 M K_2CO_3 in combination with ethanol and toluene at 90 °C gave the best results. Under these reaction conditions, the yield of the desired product **2a** was 85% (Table 1, entry 1). The reaction proceeds through an intramolecular *syn*-carbopalladation step via a 5-*exo*-*dig* process with the alkyne unit to give a σ-alkylpalladium(II) intermediate, and a subsequent intermolecular Suzuki coupling with phenylboronic acid derivatives gave the desired product **2a** in a stereoselective fashion.

A large number of substrates were prepared under the optimized reaction conditions; the results are shown in Table 1. This transformation was found to be very general, and the desired 2-[(indoline-3-ylidene)(methyl)]benzaldehydes **2a–o** were obtained in reasonably good yields. Aniline derivatives possessing electron-rich groups, such as methyl, and electron-poor groups, such as fluoride, were coupled under standard reaction conditions in very good yields (Table 1, entries 9–12).

Similarly, a wide number of functional groups on the aromatic ring of the alkyne terminus were studied. Aromatic rings possessing electron-donating groups such as *p*-Me and *p*-OMe (Table 1, entries 2, 3, 10, and 12) could be efficiently transformed to the desired product in good yields (68–78%). Alkynes bearing a number of electron-withdrawing groups such as *p*-COMe, *p*-CO_2Et, and *p*-Cl (Table 2, entries 4, 5, 9, and 13) were also investigated and gave the desired products in moderate to good yields. Notably, other aromatic rings connected to alkynes such as 3-thienyl, 4-biphenyl, and 2-naphthyl rings were also efficiently converted in this domino Heck–Suzuki coupling reaction and furnished the desired

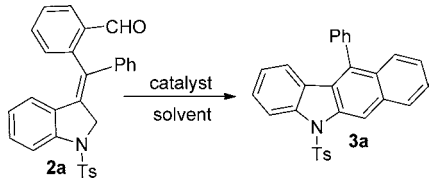
Table 1. Preparation of Substrates via Domino Heck–Suzuki Reaction



entry	R^1	R^2	R^3	time (h)	yield[b] (%)	(*E*/*Z*)
1	H	Ts	Ph	2	**2a**	85 (1:0)
2	H	Ts	*p*-MeC_6H_4	2.5	**2b**	78 (1:0)
3	H	Ts	*p*-$MeOC_6H_4$	3	**2c**	76 (1:0)
4	H	Ts	*p*-$MeCOC_6H_4$	5	**2d**	54 (1:1)
5	H	Ts	*p*-$EtO_2CC_6H_4$	5	**2e**	60 (3:1)
6	H	Ts	3-thienyl	4	**2f**	86 (0:1)
7	H	Ts	4-biphenyl	4	**2g**	74 (1:0)
8	H	Ts	1-naphthyl	5	**2h**	75 (1:0)
9	*p*-Me	Ts	*p*-ClC_6H_4	4	**2i**	70 (1:0)
10	*p*-Me	Ts	*p*-$MeOC_6H_4$	4	**2j**	73 (1:0)
11	*p*-F	Ts	Ph	5	**2k**	63 (1:0)
12	*p*-F	Ts	*p*-MeC_6H_4	5	**2l**	68 (5:1)
13	H	Ms	*p*-ClC_6H_4	3	**2m**	75 (6:1)
14	H	Ts	Me	2	**2n**	83 (1:0)
15	H	Ts	$(CH_2)_3CH_3$	2	**2o**	57 (1:0)

[a]Reaction conditions: substrate **1** (0.5 mmol), 2-formylphenylboronic acid (0.75 mmol), $Pd(OAc)_2$ (5 mol %), PCy_3 (10 mol %), toluene (2 mL), ethanol (2 mL), and 2.5 M K_2CO_3. [b]Isolated yield.

Table 2. Optimization of Reaction Conditions for the Domino Isomerisation/Cyclodehydration Process



entry	catalyst	solvent	temp (°C)	time (h)	yield[b] (%)
1	$FeCl_3$	CH_2Cl_2	40	6	70
2	$FeCl_3$	$ClCH_2CH_2Cl$	rt	3	95
3	$FeCl_3$	CH_3CN	80	8	48
4	$FeCl_3$	$MeNO_2$	60	5	78
5	$FeCl_3$	toluene	60	6	83
6	$FeCl_3$	tetrahydrofuran	60	6	nr
7	$FeCl_3$,$6H_2O$	$ClCH_2CH_2Cl$	60	1	90
8	$FeBr_3$	$ClCH_2CH_2Cl$	60	0.5	63
9	$InCl_3$	$ClCH_2CH_2Cl$	60	1.5	90
10	$In(OTf)_3$	$ClCH_2CH_2Cl$	60	6	78
11	Ag(OTf)	$ClCH_2CH_2Cl$	60	1.5	90
12	$AlCl_3$	$ClCH_2CH_2Cl$	85	5	50[c]
13	$ZnCl_2$	$ClCH_2CH_2Cl$	85	5	30
14	CuCl	$ClCH_2CH_2Cl$	85	6	nr
15	*p*-TsOH	$ClCH_2CH_2Cl$	60	5	85
16	TfOH	$ClCH_2CH_2Cl$	60	1.5	90

[a]Reaction conditions: substrate **2a** (0.21 mmol), solvent (2 mL), catalyst (10 mol %). [b]Isolated pure yield. [c]2.5 equiv of $AlCl_3$ used.

dx.doi.org/10.1021/ol500505k | *Org. Lett.* 2014, 16, 2166–2169

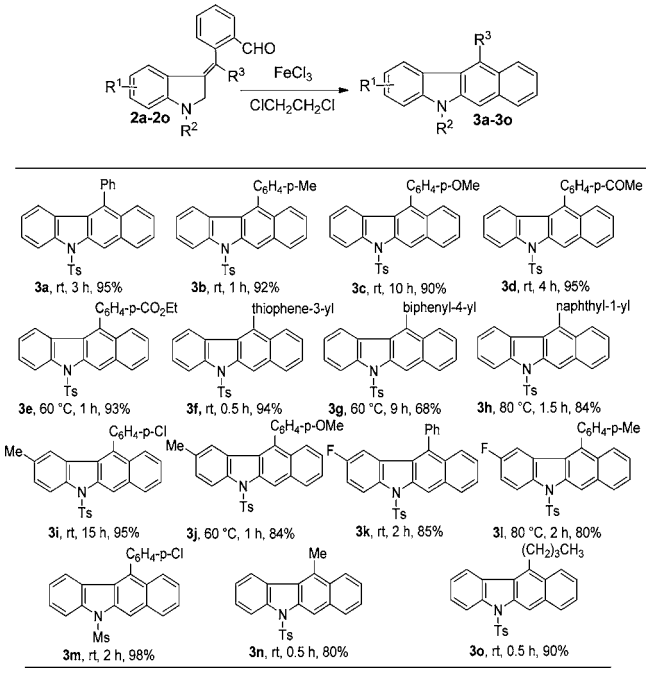
products in very good yields of 86%, 74%, and 75%, respectively (Table 2, entries 6–8). Interestingly, this strategy was also applicable to the alkyne unit bearing alkyl groups such as -Me and -Bu and afforded the desired product in 83% and 57% yields, respectively (Table 1, entries 14 and 15). In most cases, the desired compounds were isolated as a single isomer, and in a few cases, a mixture of stereoisomers was obtained (Table 2, entries 4, 5, 12, and 13). The next step was conducted without separation of the isomers.

After having a series of key substrates **2a–o**, next we focused on the development of reaction conditions for the isomerization of **2a** as the model substrate. Because of our ongoing interest in the area of development of new reactions catalyzed by iron(III) salts,[14] we first investigated the isomerization of **2a** to **2aa** in the presence of iron(III) chloride.

Serendipitously, we observed that the formation of benzo[*b*]carbazole **3a** occurred in one pot via domino isomerization/cyclodehydration sequences in 70% yield when the substrate **2a** was treated with 10 mol % of $FeCl_3$ in dichloromethane at 40 °C (Table 2, entry 1). Inspired by this result, some reaction parameters such as solvent effects and role of catalysts were systematically investigated for the purpose of improving the yield. The results are summarized in Table 2. To our delight, the yield of **3a** was significantly increased to 95% when the reaction was carried out in 1,2-dichloroethane at room temperature in 3 h (Table 2, entry 2). Further study showed that MeCN, $MeNO_2$, and toluene were less efficient (Table 2, entries 3–5), while in THF the reaction did not proceed at all, even after heating at 60 °C (Table 2, entry 6). Coordinating solvents may have reduced the activity of iron(III) and, hence, were not suitable for this reaction. Next, different metal catalysts such as hydrated $FeCl_3$, $FeBr_3$, $InCl_3$, $In(OTf)_3$, and AgOTf were also evaluated for this process using the substrate **2a** in 1,2-dichloroethane. All these catalysts showed high catalytic activity, but with regard to temperature and yields, they were inferior to anhydrous $FeCl_3$. Lewis acids such as $ZnCl_2$ gave only 30% yield of the desired product (Table 2, entry 13), whereas catalytic anhydrous $AlCl_3$ (10 mol %) did not furnish any product but gave 50% yield in the presence of 2.5 equiv of $AlCl_3$ (Table 2, Entry12). Brønsted acids such as *p*-TsOH and TfOH (Table 2, entries 15 and 16) worked at 60 °C and afforded lower yields of 85% and 90%, respectively. The reaction did not proceed in the absence of catalyst and was sluggish in reducing the amount of catalyst loading. Thus, $FeCl_3$ (10 mol %) in 1,2-dichloroethane was defined as the standard reaction conditions for the additional study.

To demonstrate the generality of this new reaction, substrates **2b–o** were examined under the optimized reaction conditions. As shown in Scheme 3, the transformation proceeded quite smoothly and afforded the desired benzo[*b*]-carbazole derivatives in excellent yields (68–98%). The reaction was found to tolerate various functional groups such as -Me, -OMe, -COMe, $-CO_2Et$, and -Cl at the *para*-position of the aromatic ring (R^3) of 2,3-dihydroindole derivatives and afforded the corresponding products **3b–e,i,l,m** in 80–98% yields. Both electron-donating groups such as *p*-Me and electron-withdrawing groups such as *p*-F on the phenyl ring of the aniline moiety could be well-tolerated in this transformation and afforded the corresponding products in 80–95% (Scheme 3, **3i–l**). Therefore, the yields of the products are not significantly sensitive to the electronic effects of the different groups; however, heating is required in a few cases for completion the reactions (Scheme 3, **3e,g,h,j,l**).

Scheme 3. Iron-Catalyzed Synthesis of Substituted Benzo[*b*]carbazole Derivatives[a]

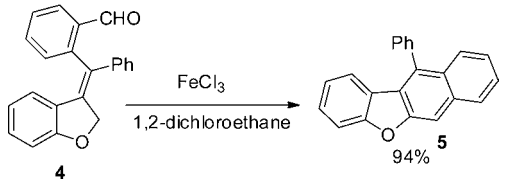


[a]Reaction conditions: substrates **2** (0.21 mmol), $FeCl_3$ (10 mol %), and 1,2-dichloroethane (2 mL).

Next, the present protocol was extended to the other aromatics and heteroaromatic ring at the olefinic center (R^3), and they also worked efficiently and gave good to excellent yield of the desired products such as 3-thienyl (Scheme 3, **3f**, 94%), *p*-biphenyl (Scheme 3, **3g**, 68%), and 1-naphthyl (Scheme 3, **3h**, 84%). Similarly, this strategy was also applicable to the alkyl group bearing 2,3-dihydroindole derivatives such as **2n** and **2o** and gave the target benzo[*b*]carbazole **3n** and **3o** in 80% and 90% yields, respectively (Scheme 3).

Finally, the generality of this strategy was also verified by the synthesis of fused benzofuran derivative **5** in 94% yield from the Heck–Suzuki coupling product **4** (Scheme 4).

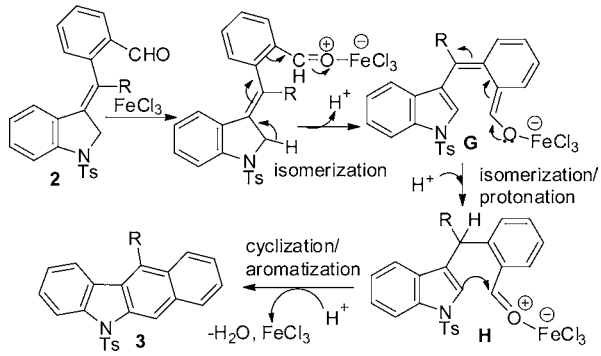
Scheme 4. Synthesis of Polycyclic Benzofuran Derivative



Based on our experimental observations and following the mechanism of the Bradsher reaction,[12] a plausible reaction pathway is delineated in Scheme 5. The isomerization reaction did not work in the absence of catalyst and substrate without bearing a 2-formyl group in **2**. Therefore, it was concluded that isomerization leading to the intermediate **G** must be driven by complexation of the carbonyl group with $FeCl_3$. Then, isomerization and protonation of **G**, furnishing the intermediate **H**, followed by intramolecular Fridel–Crafts alkylation and subsequent aromatization by dehydration of water, afforded the benzo[*b*]carbazole derivatives **3**. All the structures of benzo-[*b*]carbazoles **3a–n** and benzofuran derivative **5** were

dx.doi.org/10.1021/ol500505k | *Org. Lett.* 2014, 16, 2166–2169

Scheme 5. Plausible Mechanism for the Isomerization/Cyclization/Aromatization Sequence



characterized by ^{1}H NMR, ^{13}C NMR, and HRMS. The product **3b** (CCDC No. 987775) was confirmed by its X-ray structure (Figure 2).

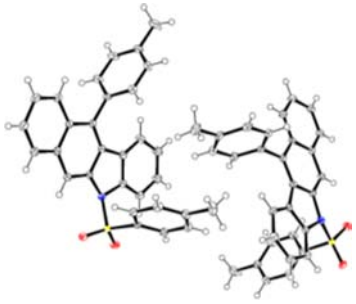


Figure 2. X-ray crystallographic structure of compound **3b**.

In summary, we have developed an iron(III)-catalyzed novel and efficient strategy for the synthesis of libraries of structurally diverse benzo[*b*]carbazole heterocycles via domino isomerization/cyclization/aromatization reaction. A wide range of substrates were investigated to show the generality of the process. The advantages of this new method are the ease of preparation of substrates, operational simplicity, high atom-economy, mild conditions, and use of inexpensive and environmentally friendly $FeCl_3$ (10 mol %) as the catalyst. Moreover, this strategy could also be extended to the synthesis of a polycylic benzofuran molecule. Further mechanistic investition and synthetic application are underway in our laboratory.

■ ASSOCIATED CONTENT

Supporting Information

Experimental procedure, copies of NMR spectra, and X-ray crystallographic data of **3b** (CIF). This material is available for free of charge via the Internet at http://pubs.acs.org.

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Notes

The authors declare no competing financial interest.

■ ACKNOWLEDGMENTS

K.P., K.B., S.J., and S.S. are thankful to the CSIR, New Delhi, India, for their fellowships.

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