



Efficient synthesis of polyfunctionalized carbazoles and pyrrolo[3,4-*c*]carbazoles via domino Diels–Alder reaction

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Abstract

The *p*-TsOH-catalyzed Diels–Alder reaction of 3-(indol-3-yl)maleimides with chalcone in toluene at 60 °C afforded two diastereoisomers of tetrahydropyrrolo[3,4-*c*]carbazoles, which can be dehydrogenated by DDQ oxidation in acetonitrile at room temperature to give the aromatized pyrrolo[3,4-*c*]carbazoles in high yields. On the other hand, the one-pot reaction of 3-(indol-3-yl)-1,3-diphenylpropan-1-ones with chalcones or benzylideneacetone in acetonitrile in the presence of *p*-TsOH and DDQ resulted in polyfunctionalized carbazoles in satisfactory yields. The reaction mechanism included the DDQ oxidative dehydrogenation of 3-(indol-3-yl)-1,3-diphenylpropan-1-ones to the corresponding 3-vinylindoles, their acid-catalyzed Diels–Alder reaction and sequential aromatization process.

Introduction

Carbazole is one of the most well-known privileged nitrogen-containing heterocycles. The carbazole skeleton is widely occurring in natural alkaloids and pharmacologically active compounds representing a broad spectrum of important bioactivities such as anticancer, antituberculosis, anti-protein kinase C, antipsychotic, and antioxidative activities [1-5]. For some examples, carprofen is a nonsteroidal anti-inflammatory pharmaceutical used to treat joint pain and postoperative pain [6] (Figure 1). Ellipticine was considered to be based mainly on DNA intercalation and topoisomerase II inhibition [7]. Midostaurin and carvediol have been approved by the FDA for

tumor therapy and treatment of congestive heart failure [8]. On the other hand, carbazole derivatives also have potential applications in optoelectronic materials, conducting polymers, and synthetic dyes [9-11]. Over the past decades, many efficient synthetic methodologies for functionalized carbazole derivatives have been successfully developed [12-18]. Because indoles are readily available materials, the direct extension of indoles to carbazole skeletons has a great advantage [19-27]. Therefore, the Diels–Alder reaction of activated 2-vinylindolines or 3-vinylindolines with diverse dienophiles has become the most attractive strategy for the synthesis of carbazole deriv-

atives [28–40]. In recent years, by using the one-pot domino synthetic strategy of in situ-generated 2-vinyl- or 3-vinylindolines and sequential Diels–Alder reaction with activated dienophiles, we have successfully developed several efficient synthetic protocols for diversely functionalized tetrahydrocarbazoles and the corresponding carbazole derivatives [41–47]. To further demonstrate the synthetic application of domino Diels–Alder reactions and in continuation of our aim to providing efficient domino reactions for the synthesis of biologically important carbazole derivatives [48–53], herein we wish to report the DDQ-mediated dehydrogenative Diels–Alder reaction of 3-(indol-3-yl)maleimides and benzoyl-substituted 3-ethylindoles with readily available chalcones for the convenient synthesis of polyfunctionalized carbazole derivatives.

Results and Discussion

According to our previously established reaction conditions for the preparation of spiro[indoline-3,5'-pyrrolo[3,4-*c*]carbazoles] [48], an equivalent amount of 3-(indol-3-yl)maleimide with chalcone was stirred in toluene at 60 °C for two hours in the presence of *p*-toluenesulfonic acid. After workup, two diastereoisomers **3a** and **3b** of tetrahydropyrrolo[3,4-*c*]carbazoles were successfully isolated in 18% and 71% yields, respectively (Scheme 1). It should be pointed out that nearly no reaction was

detected in the absence of *p*-toluenesulfonic acid. The structures of both products were fully characterized by various spectroscopy methods and confirmed by determination of their single crystal structures (Figure 2 and Figure 3).

From Figure 2, it can be seen that the phenyl group and the adjacent *p*-chlorobenzoyl group are *trans*-oriented. Additionally, the phenyl group is *cis*-oriented to the 1-benzylpyrrolidine-2,5-dione ring in compound **3a**. On the other hand, for compound **3b** (Figure 3), it can be seen that the phenyl group is *trans*-oriented to both, the *p*-chlorobenzoyl group and the 1-benzylpyrrolidine-2,5-dione ring in this compound. Thus, the isomers **3a** and **3b** are diastereoisomers. It is known that the starting chalcones usually have *E*-configuration. The phenyl group and *p*-chlorobenzoyl group still exist in the *trans*-position in both diastereoisomers **3a** and **3b** as in the starting chalcone. This result clearly showed that this acid-catalyzed cycloaddition reaction proceeded through a concerted Diels–Alder reaction mechanism.

The acid-catalyzed Diels–Alder reaction afforded a mixture of two diastereoisomers, which decreased the synthetic value of the reaction. Thus, after the first step reaction, a DDQ dehydrogenation reaction was carried out in acetonitrile at room temper-

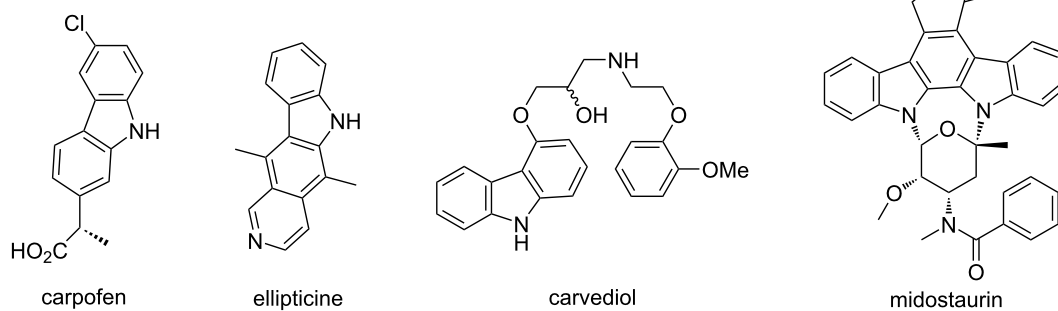
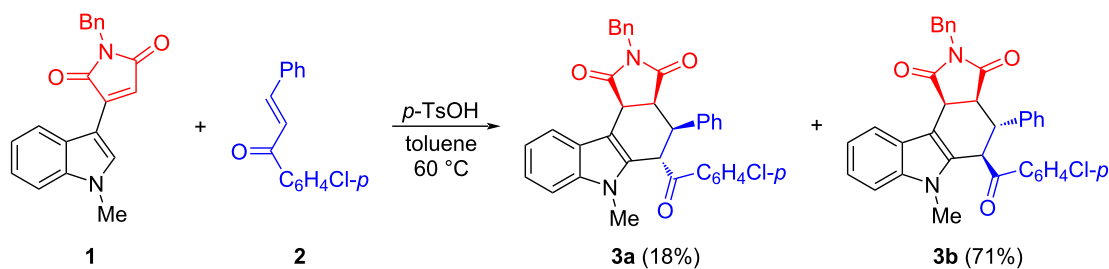


Figure 1: Representative bioactive carbazole derivatives.



Scheme 1: Synthesis of tetrahydropyrrolo[3,4-*c*]carbazoles **3a** and **3b**.

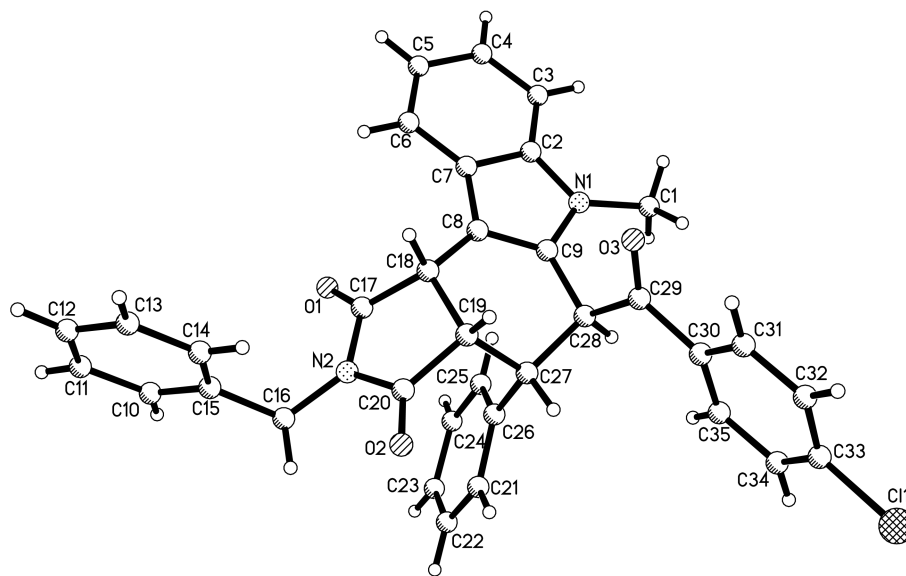


Figure 2: Single crystal structure of the isomer **3a**.

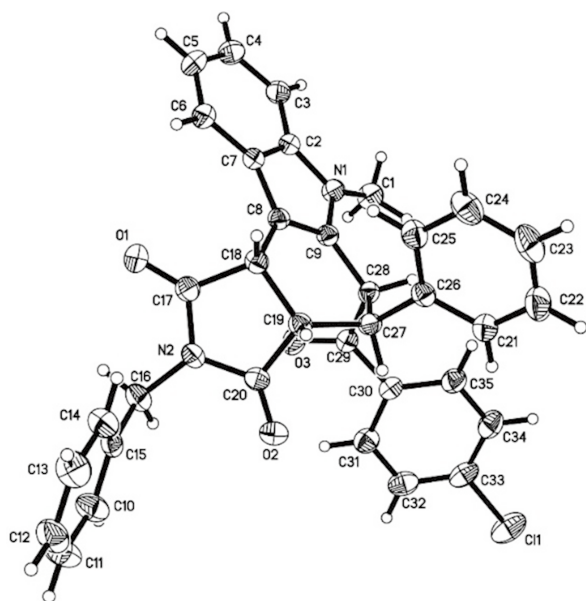
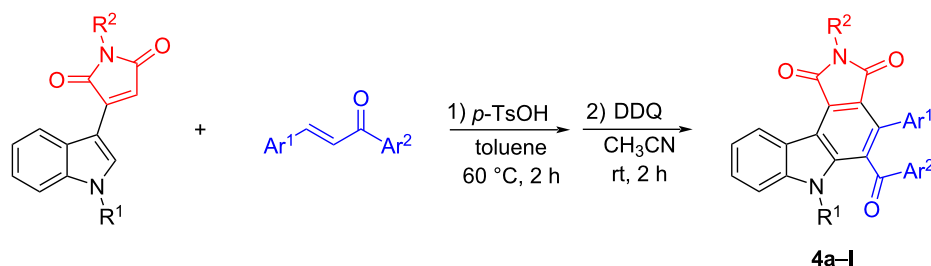


Figure 3: Single crystal structure of the isomer **3b**.

ature. A series of aromatized pyrrolo[3,4-*c*]carbazoles **4a–l** were successfully synthesized by the one-pot two-step reaction and the results are summarized in Table 1. All reactions proceeded smoothly to give the corresponding pyrrolo[3,4-*c*]carbazoles **4a–l** in satisfactory yields. Indole itself and *N*-methylindole could also be successfully employed in the reaction. The *N*-Me, *N*-Ph, and *N*-Bn substitution in the maleimide moiety showed only a marginal effect on the reaction outcome. Various chalcones with electron-donating methyl and

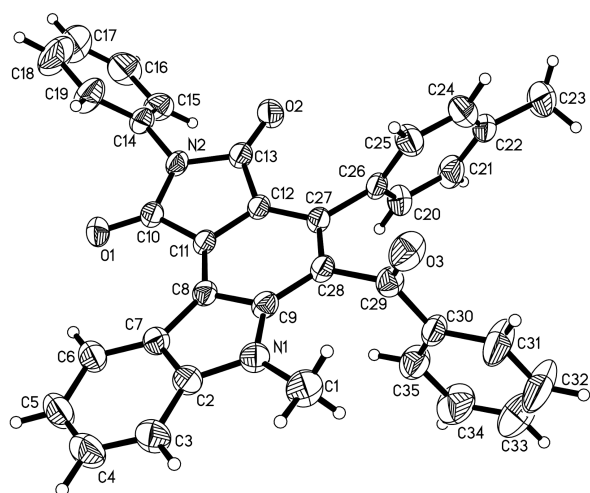
methoxy groups and electron-withdrawing *m*-chloro and *p*-chloro substituents gave the products in good yields. However, the nitro-substituted chalcone gave the product **4h** in a slightly lower yield. The structures of the pyrrolo[3,4-*c*]carbazoles **4a–l** were established by various spectroscopy methods. Further, the single crystal structure of compound **4g** was determined by X-ray diffraction (Figure 4). It can be seen that the ring of pyrrolo[3,4-*c*]carbazole exists in a slightly twisted plane. The dihedral angles of the phenyl and the benzoyl group to the central benzene ring are 72.018° and 88.402°.

To further expand the scope of this domino Diels–Alder reaction, another kind of 3-vinylindoles was employed in the one-pot reaction. First, the 3-(indol-3-yl)-1,3-diphenylpropan-1-ones prepared through Friedel–Crafts alkylation of indole with chalcones, were oxidized by DDQ in acetonitrile to generate in situ the expected active diene, indole-substituted chalcones. Then, the *p*-TsOH-catalyzed Diels–Alder reaction of indole-chalcones with second chalcones and sequential aromatization through DDQ dehydrogenation resulted in the polyfunctionalized carbazoles **6a–l** in good yields (Table 2). Additionally, the similar reaction with benzylideneacetone gave the desired carbazoles **6m** and **6n** albeit in significantly lower yields. Thus, this one-pot domino reaction successfully constructed carbazoles with four substituents on the benzene ring from the corresponding indole derivatives with two molecules of chalcones. It should be pointed out that the Diels–Alder reaction resulted in a complex mixture comprising four diastereoisomers of the tetrahydrocarbazoles, which was very difficult to

Table 1: Synthesis of pyrrolo[3,4-c]carbazoles **4a–l**.^a

| entry | compound | R ¹ | R ² | Ar ¹ | Ar ² | yield (%) ^b |
|-------|-----------|-----------------|-----------------|---|--|------------------------|
| 1 | 4a | H | Ph | <i>p</i> -CH ₃ C ₆ H ₄ | C ₆ H ₅ | 87 |
| 2 | 4b | CH ₃ | CH ₃ | <i>p</i> -ClC ₆ H ₄ | C ₆ H ₅ | 89 |
| 3 | 4c | CH ₃ | CH ₃ | C ₆ H ₅ | <i>p</i> -CH ₃ C ₆ H ₅ | 92 |
| 4 | 4d | CH ₃ | CH ₃ | <i>p</i> -CH ₃ C ₆ H ₄ | <i>p</i> -CH ₃ C ₆ H ₄ | 91 |
| 5 | 4e | CH ₃ | Ph | C ₆ H ₅ | CH ₃ | 85 |
| 6 | 4f | CH ₃ | Ph | C ₆ H ₅ | C ₆ H ₅ | 84 |
| 7 | 4g | CH ₃ | Ph | <i>p</i> -CH ₃ C ₆ H ₄ | C ₆ H ₅ | 93 |
| 8 | 4h | CH ₃ | Ph | <i>m</i> -NO ₂ C ₆ H ₄ | <i>p</i> -CH ₃ C ₆ H ₄ | 76 |
| 9 | 4i | CH ₃ | Ph | <i>p</i> -CH ₃ C ₆ H ₄ | <i>p</i> -CH ₃ OC ₆ H ₄ | 86 |
| 10 | 4j | CH ₃ | Ph | <i>p</i> -CH ₃ C ₆ H ₄ | <i>m</i> -ClC ₆ H ₄ | 84 |
| 11 | 4k | CH ₃ | Bn | <i>p</i> -CH ₃ C ₆ H ₄ | C ₆ H ₅ | 93 |
| 12 | 4l | CH ₃ | Bn | C ₆ H ₅ | <i>p</i> -ClC ₆ H ₄ | 87 |

^aReaction conditions: 1) 3-(indol-3-yl)maleimide (1.0 mmol), chalcone (1.0 mmol), toluene (10.0 mL), *p*-TsOH (0.2 mmol), 80 °C, 2 h; 2) DDQ (1.2 mmol), CH₃CN (10.0 mL), rt, 2 h. ^bIsolated yields.

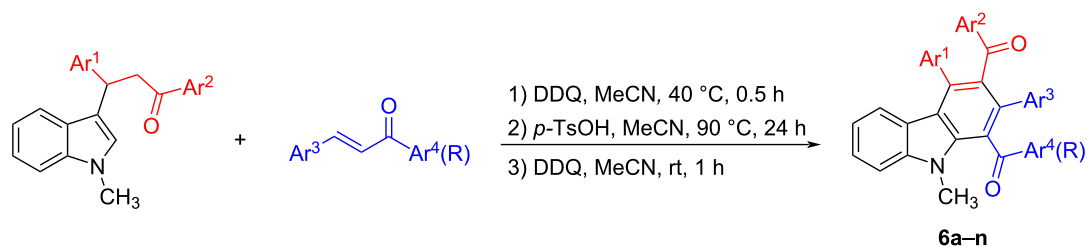
**Figure 4:** Single crystal structure of the isomer **4g**.

separate. After oxidation with DDQ, the aromatized carbazole derivatives **6a–n** were easily obtained as single products in good yields. The chemical structures of the carbazoles were fully characterized by ¹H NMR, ¹³C NMR, IR, and HRMS spectra.

To explain the formation of the products, a plausible reaction mechanism was proposed in Scheme 2 on the basis of the previously reported reaction [48,53]. Firstly, the DDQ oxidative dehydrogenation of 3-(indol-3-yl)-1,3-diphenylpropan-1-one gave the expected indole-substituted chalcone **A**, which comprises the desired 3-vinylindole scaffold as the reactive diene. In the meantime, the carbonyl group of the chalcone is protonated to give the activated dienophile in the presence of *p*-toluenesulfonic acid. Secondly, the Diels–Alder reaction of indole-chalcone **A** with the dienophile results in the tetrahydrocarbazole **B** having an exocyclic C=C bond. Thirdly, a new tetrahydrocarbazole intermediate **C** is formed by a 1,3-H shifting process. The resulting tetrahydrocarbazole intermediate (**C**) might be a mixture of several possible diastereoisomers because it has four substituents on the cyclohexenyl ring. After a further DDQ oxidation, the aromatized carbazole **6** is successfully produced as the final product.

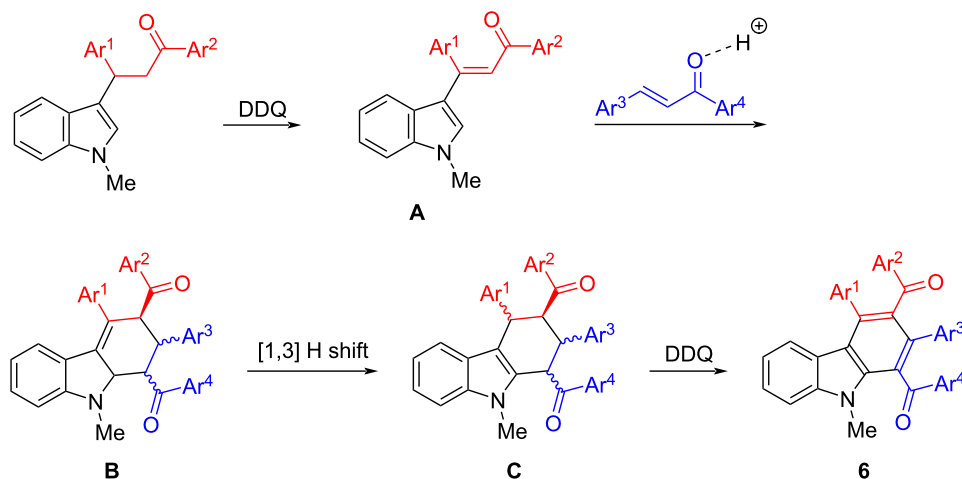
Conclusion

In summary, we have investigated the domino Diels–Alder reaction of 3-(indol-3-yl)maleimides and in situ-generated indole-chalcones with dienophilic chalcones. This one-pot two-step reaction successfully provided the polyfunctionalized carbazole derivatives in an extremely simple and highly effi-

Table 2: Synthesis of the polysubstituted carbazoles **6a–n**.^a

| entry | compound | Ar ¹ | Ar ² | Ar ³ | Ar ⁴ (R) | yield (%) ^b |
|-------|-----------|--|---|--|--|------------------------|
| 1 | 6a | C ₆ H ₅ | C ₆ H ₅ | C ₆ H ₅ | C ₆ H ₅ | 61 |
| 2 | 6b | <i>p</i> -CH ₃ C ₆ H ₄ | <i>p</i> -CH ₃ C ₆ H ₄ | <i>p</i> -CH ₃ C ₆ H ₄ | <i>p</i> -CH ₃ C ₆ H ₄ | 63 |
| 3 | 6c | <i>p</i> -CH ₃ C ₆ H ₄ | <i>p</i> -CH ₃ C ₆ H ₄ | <i>p</i> -ClC ₆ H ₄ | <i>p</i> -CH ₃ C ₆ H ₄ | 68 |
| 4 | 6d | <i>p</i> -CH ₃ C ₆ H ₄ | <i>p</i> -CH ₃ C ₆ H ₄ | <i>o</i> -CH ₃ C ₆ H ₄ | <i>p</i> -CH ₃ OC ₆ H ₄ | 64 |
| 5 | 6e | <i>p</i> -CH ₃ OC ₆ H ₄ | <i>p</i> -CH ₃ C ₆ H ₄ | <i>o</i> -CH ₃ C ₆ H ₄ | <i>p</i> -CH ₃ OC ₆ H ₄ | 62 |
| 6 | 6f | <i>p</i> -CH ₃ OC ₆ H ₄ | <i>p</i> -CH ₃ C ₆ H ₄ | <i>p</i> -CH ₃ OC ₆ H ₄ | <i>p</i> -ClC ₆ H ₄ | 66 |
| 7 | 6g | <i>p</i> -ClC ₆ H ₄ | <i>p</i> -CH ₃ C ₆ H ₄ | C ₆ H ₅ | C ₆ H ₅ | 59 |
| 8 | 6h | <i>p</i> -ClC ₆ H ₄ | <i>p</i> -CH ₃ C ₆ H ₄ | <i>p</i> -ClC ₆ H ₄ | <i>p</i> -CH ₃ C ₆ H ₄ | 60 |
| 9 | 6i | <i>p</i> -ClC ₆ H ₄ | <i>p</i> -CH ₃ C ₆ H ₄ | <i>p</i> -NO ₂ C ₆ H ₄ | <i>p</i> -CH ₃ OC ₆ H ₄ | 69 |
| 10 | 6j | <i>o</i> -ClC ₆ H ₄ | <i>p</i> -CH ₃ C ₆ H ₄ | <i>p</i> -CH ₃ OC ₆ H ₄ | <i>p</i> -ClC ₆ H ₄ | 63 |
| 11 | 6k | <i>p</i> -CH ₃ OC ₆ H ₄ | <i>p</i> -ClC ₆ H ₄ | <i>m</i> -ClC ₆ H ₄ | <i>p</i> -CH ₃ C ₆ H ₄ | 61 |
| 12 | 6l | <i>p</i> -CH ₃ OC ₆ H ₄ | <i>p</i> -ClC ₆ H ₄ | <i>p</i> -CH ₃ OC ₆ H ₄ | <i>p</i> -ClC ₆ H ₄ | 62 |
| 13 | 6m | C ₆ H ₅ | C ₆ H ₅ | C ₆ H ₅ | CH ₃ | 31 |
| 14 | 6n | <i>p</i> -ClC ₆ H ₄ | <i>p</i> -CH ₃ C ₆ H ₄ | C ₆ H ₅ | CH ₃ | 32 |

^aReaction conditions: 1) 3-(indol-3-yl)-1,3-diphenylpropan-1-one (0.6 mmol), chalcone (0.5 mmol), DDQ (0.72 mmol), MeCN (15.0 mL), rt, 0.5 h; 2) *p*-TsOH (0.06 mmol), reflux, 4 h; 3) DDQ (0.6 mmol), rt, 1 h. ^bIsolated yields.

**Scheme 2:** Proposed domino reaction mechanism for the formation of carbazoles **6**.

cient fashion. This protocol has the advantages of using readily available starting reagents, simple manufacture, high efficiency and atomic economy. The unusual feature of this reaction is the normal electron-demand Diels–Alder reaction between elec-

tron-deficient dienes such as 3-(indol-3-yl)maleimides and indole-chalcone to electron-deficient dienophilic chalcones. The potential applications of this reaction in organic and medicinal chemistry might be significant.

Experimental

1. General procedure for the preparation of the carbazoles

4a–l: To a round-bottomed flask was added 3-(indol-3-yl)maleimide (1.0 mmol), chalcone (1.0 mmol), *p*-toluenesulfonic acid (0.2 mmol), and toluene (10.0 mL). The solution was heated to 60 °C for two hours. After removing the solvent by rotatory evaporation, DDQ (1.2 mmol) and acetonitrile (10.0 mL) were added and the mixture was stirred at room temperature for two hours. After removing the solvent, the residue was subjected to column chromatography with petroleum ether and ethyl acetate 8:1 (v/v) as eluent to give the pure products for analysis.

5-Benzoyl-2-phenyl-4-(*p*-tolyl)pyrrolo[3,4-*c*]carbazole-

1,3(2*H*,6*H*)-dione (4a): green solid, 440 mg, 87%; mp 234–236 °C; ¹H NMR (400 MHz, CDCl₃) δ 9.24 (s, 1H, ArH), 9.15 (d, *J* = 8.0 Hz, 1H, ArH), 7.60–7.56 (m, 1H, ArH), 7.53–7.44 (m, 7H, ArH), 7.42–7.31 (m, 3H, ArH), 7.21–7.14 (m, 4H, ArH), 6.92 (d, *J* = 7.6 Hz, 2H, ArH), 2.18 (s, 3H, CH₃); ¹³C {¹H} NMR (100 MHz, CDCl₃) δ 198.0, 167.3, 142.9, 141.7, 138.2, 137.6, 137.2, 132.9, 131.9, 131.7, 130.7, 129.1, 129.0, 128.8, 128.2, 127.9, 127.8, 127.7, 126.7, 126.1, 125.6, 121.7, 120.7, 120.3, 119.2, 111.3, 21.1; IR (KBr) ν: 2988, 1786, 1734, 1611, 1485, 1456, 1357, 1314, 1185, 1021, 988, 786, 734 cm⁻¹; HRMS–ESI–TOF (*m/z*): [M + Na]⁺ calcd for C₃₄H₂₂NaN₂O₃, 529.1523; found, 529.1512.

2. General procedure for the preparation of carbazoles

6a–n: To a round-bottomed flask were added 3-(indol-3-yl)-1,3-diphenylpropan-1-one (0.6 mmol), DDQ (0.72 mmol), and acetonitrile (15.0 mL). The mixture was stirred at room temperature for 30 min. Then, the chalcone (0.5 mmol) and *p*-toluenesulfonic acid (0.06 mmol) were added and the solution was refluxed for four hours. After cooling to room temperature, DDQ (0.6 mmol) was added and the mixture was stirred at room temperature for one hour. After removing the solvent by rotatory evaporation at reduced pressure, the residue was subjected to column chromatography with a mixture of petroleum ether, ethyl acetate and methylene dichloride 20:1:5 (v/v/v) to give the pure products for analysis.

(9-Methyl-2,4-diphenyl-9*H*-carbazole-1,3-diyl)bis(phenyl-

methanone) (6a): White solid, 165 mg, 61%; mp 249–251 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.64 (d, *J* = 7.6 Hz, 3H, ArH), 7.43 (t, *J* = 7.8 Hz, 2H, ArH), 7.39–7.34 (m, 6H, ArH), 7.28–7.24 (m, 3H, ArH), 7.09 (t, *J* = 7.6 Hz, 2H, ArH), 7.01 (s, 2H, ArH), 6.96 (t, *J* = 7.8 Hz, 2H, ArH), 6.84 (d, *J* = 6.8 Hz, 4H, ArH), 3.64 (s, 3H, CH₃); ¹³C NMR (400 MHz, CDCl₃) δ 198.5, 198.4, 142.4, 138.9, 138.7, 137.6, 137.5, 136.8, 135.9, 135.2, 133.3, 132.2, 131.5, 131.4, 129.5, 129.2, 128.3, 128.2, 127.8, 127.6, 127.0, 126.9, 126.4, 122.4, 122.0, 121.8, 121.6, 119.6, 108.7, 32.1; IR (KBr) ν: 3057, 3023, 2907, 2360, 2339,

1720, 1605, 1482, 1320, 1267, 1172, 1009, 936, 805, 743, 612, 447 cm⁻¹; HRMS–ESI (*m/z*): [M + Na]⁺ calcd for C₃₉H₂₇NO₂, 564.1934; found, 564.1926.

The crystallographic data of the compounds **3a** (CCDC 2099074), **3b** (CCDC 2099075), and **4g** (CCDC 2099076) have been deposited at the Cambridge Crystallographic Database Centre (<http://www.ccdc.cam.ac.uk>).

Supporting Information

Supporting Information File 1

Characterization data and ¹H NMR, ¹³C NMR, and HRMS spectra of the synthesized compounds.

[<https://www.beilstein-journals.org/bjoc/content/supplementary/1860-5397-17-159-S1.pdf>]

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Preprint

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