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

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Abstract

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 in acetonitrile in the presence of *p*-TsOH and DDQ resulted in polyfunctionalized carbazoles in satisfactory yields. The reaction mechanism included DDQ oxidative dehydrogenation of 3-(indol-3-yl)-1,3-diphenylpropan-1-ones to the corresponding 3-vinylindoles, its acid-catalyzed Diels-Alder reaction and sequential aromatization process.

Keywords

maleimides; chalcone; carbazole; 3-vinylindole; pyrrolo[3,4-*c*]carbazole; Diels-Alder reaction.

Introduction

Carbazole is one of the most well-known privileged nitrogen-containing heterocycle. The carbazole skeleton is widely occurring in natural alkaloids and pharmacologically active compounds representing broad spectrum of important bioactivities such as anticancer, antituberculosis, anti-protein kinase C, antipsychotic and antioxidant activities [1-5]. For some examples, Carprofen is a nonsteroidal anti-inflammatory pharmaceutical used to treat joint pain and postoperative pain [6] (Scheme 1). Ellipticine was considered to be based mainly on DNA intercalation and topoisomerase II inhibition [7]. Midostaurin and carvedilol have been approved by the FDA for therapy for tumor and 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 much more readily available material, the direct extension of indoles to carbazole skeletons has a great advantage [19-21]. Therefore, Diels-Alder reaction of active 2-vinylindolines or 3-vinylindolines with diverse dienophiles has become the most attractive strategy for the synthesis of many carbazole derivatives [22-34]. In recent years, by using the one-pot domino synthetic strategy of the *in situ* generated active 2-vinyl or 3-vinylindolines and sequential Diels-Alder reaction with active dienophiles, we have successfully developed several efficient synthetic protocols for diverse functionalized tetrahydrocarbazole and the corresponding carbazole derivatives [35-41]. For further demonstrating the synthetic applications of domino Diels-Alder reaction and in continuation our aim to providing efficient domino reaction for synthesis of biologically important carbazole derivatives [42-47], herein we wish to report DDQ

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.

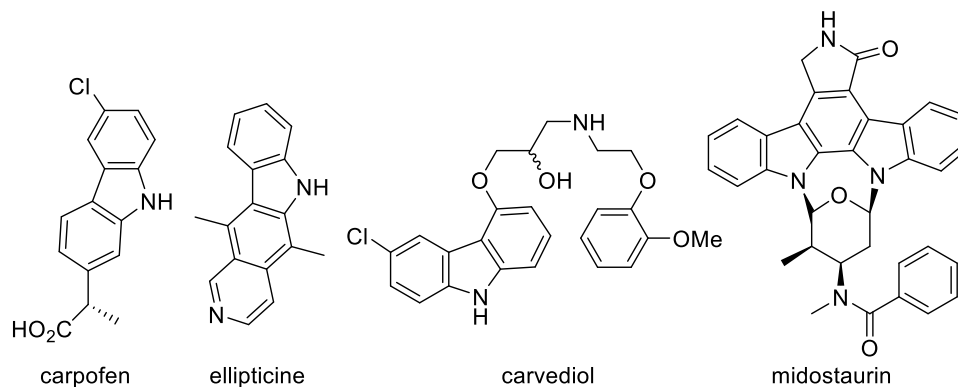
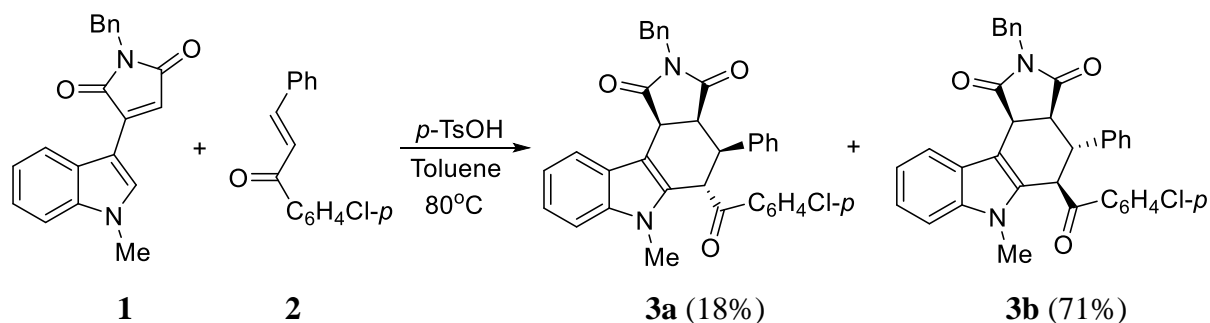


Figure 1 Representative bioactive carbazole derivatives

Results and Discussion

According to our previously established reaction conditions for the preparation of spiro[indoline-3,5'-pyrrolo[3,4-*c*]carbazoles] [42], an equivalent amount of 3-(indol-3-yl)maleimide with chalcone was stirred in toluene at 60 °C for two hours. After workup, two diastereoisomers **3a** and **3b** of tetrahydropyrrolo[3,4-*c*]carbazoles were successfully isolated in 18% and 71% yields, respectively (Scheme 1). Their structures were fully characterized by various spectroscopy and were confirmed by determining of the single crystal structures (Fig. 1-2). From the Fig. 1, it can be seen that the phenyl group exist on the *cis*-positions of the ring of 1-benzylpyrrolidine-2,5-dione and the *trans*-the *p*-chlorobenzoyl group in the diastereoisomer **3a**. On the other hand, from Fig. 2, it can be seen that the phenyl group exist on the *trans*-positions of the *p*-chlorobenzoyl group and the ring of 1-benzylpyrrolidine-2,5-dione in compound **3b**. Thus, the isomers **3a** and **3b** are belonging to the diastereoisomers. It is known that the starting chalcones usually have *E*-configuration. The phenyl group and *p*-

chlorobenzoyl group still exist on *trans*-position in both diastereoisomers **3a** and **3b** as **that** in the starting chalcone. This result clearly showed that this acid catalyzed cycloaddition reaction proceeded with a concerted Diels-Alder reaction mechanism.



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

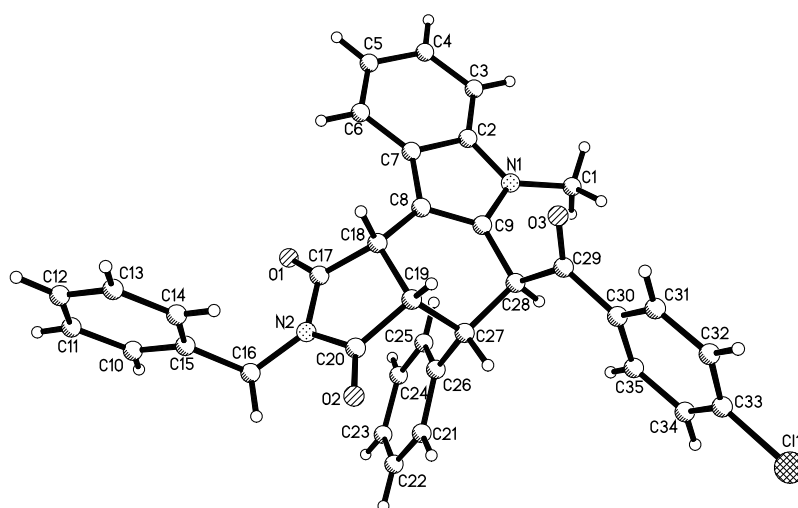


Fig. 1 Single crystal structure of the isomer **3a**

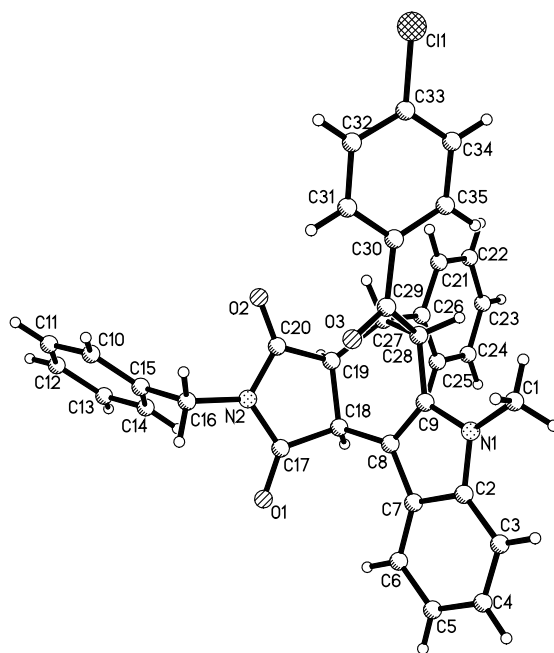
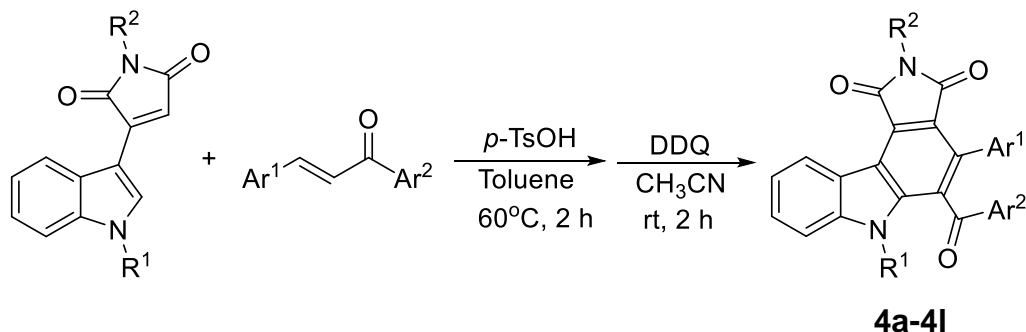


Fig. 2 Single crystal structure of the isomer **3b**

The acid-catalyzed Diels-Alder reaction afforded mixture of the two diastereoisomers with lower molecular ratio, which decreased the synthetic value of the reaction. Thus, after finishing the first step reaction, a further DDQ dehydrogenated reaction was carried out in acetonitrile at room temperature. A series of aromatized pyrrolo[3,4-*c*]carbazoles **4a-4l** were successfully synthesized by the one-pot two-step reaction. The results are summarized in Table 1. All reaction proceeded smoothly to give the pyrrolo[3,4-*c*]carbazoles **4a-4l** in satisfactory yields. Indole itself and *N*-methylindole can be successfully employed in the reaction. The *N*-Me, *N*-Ph and *N*-Bn in the moiety of maleimide showed very marginal effect on the reaction. Various chalcones with normal electron-donating methyl, methoxy groups and electron-withdrawing *m*-chloro, *p*-chloro groups gave the products in good yields. However, the nitro-substituted chalcone gave the product **4h** in littler lower yields. The structures of the pyrrolo[3,4-*c*]carbazoles **4a-4l** were established by various spectroscopy. The single crystal structure of the compound **4g** was determined by X-ray diffraction method (Fig. 3). It can be seen that the ring of pyrrolo[3,4-*c*]carbazole exists in a slightly

twisted one plane. The dihedral angles of phenyl group and the benzoyl group to the central benzene ring are 72.018° and 88.402°.

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



Entry	Compd	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	C ₆ H ₅	92
4	4d	CH ₃	Ph	<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>m</i> -ClC ₆ H ₄	86
10	4j	CH ₃	Ph	<i>p</i> -CH ₃ OC ₆ H ₄	<i>p</i> -CH ₃ OC 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

a. Reaction 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 hrs; 2. DDQ (1.2 mmol), CH₃CN (10.0 mL), rt, 2 hrs. b. Isolated yields.

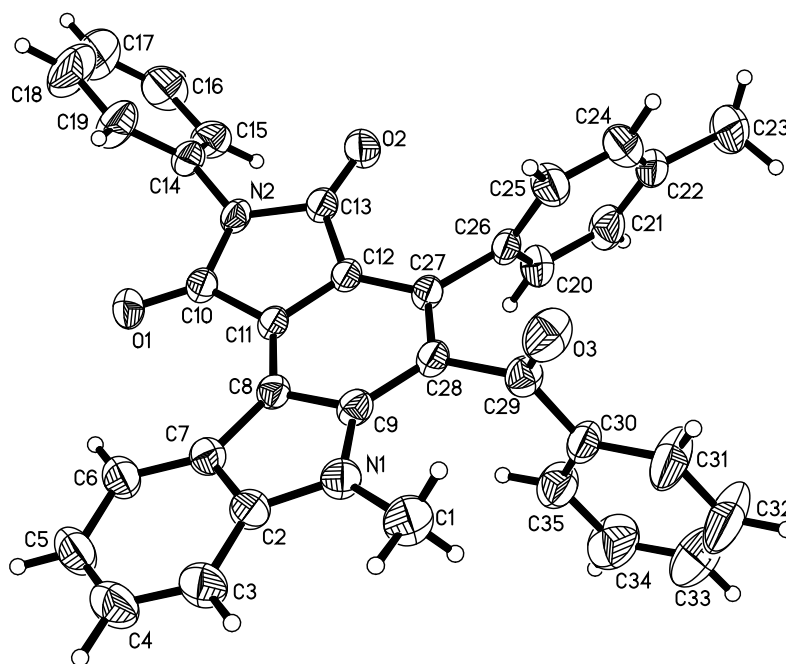


Fig. 3 Single crystal structure of the isomer **4g**

For developing the variety of this domino Diels-Alder reaction, another kind of 3-vinylindoles were also employed in the one-pot reaction. 3-(Indol-3-yl)-1,3-diphenylpropan-1-ones were firstly oxidized by DDQ in acetonitrile to generate *in situ* the expected active diene, indole-chalcones. Then, *p*-TsOH catalyzed Diels-Alder reaction of indole-chalcones with second chalcones and sequential aromatization process with DDQ dehydrogenation resulted in the polyfunctionalized carbazoles **6a-6l** in good yields (Table 2). Additionally, the similar reaction benzylideneacetone gave the desired carbazoles **6m** and **6n** in lower yields. Because the starting material 3-(Indol-3-yl)-1,3-diphenylpropan-1-ones was previous prepared by acid catalyzed Friedel-Crafts alkylation of indole with chalcone. Thus, this one-pot domino reaction successfully constructed carbazoles with four substituents on the benzene from indole with two molecules of chalcones. It should be pointed that the Diels-Alder reaction resulted in the several diastereoisomers of tetrahydrocarbazoles, which was very difficult to separate out. After oxidation with DDQ, the aromatized carbazole derivatives

6a-6m were easily obtained as one product in good yields. The chemical structures of the carbazoles were fully characterized by ^1H , ^{13}C , IR and HRMS spectra.

Table 2 Synthesis of the polysubstituted carbazoles **6a-6l**

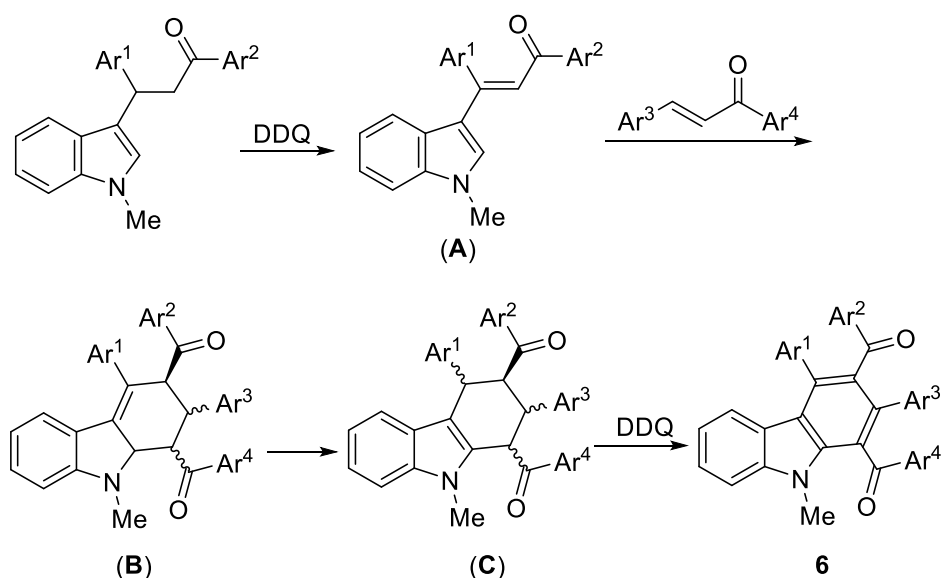


Entry	Compd	Ar ¹	Ar ²	Ar ³	Ar ⁴	Yield (%) ^b
1	6a	C ₆ H ₅	C ₆ H ₅	C ₆ H ₅	C ₆ H ₅	61
2	6b	<i>p</i> -CH ₃ C ₆ H ₄	<i>p</i> -CH ₃ C ₆ H ₄	<i>p</i> -CH ₃ C ₆ H ₄	<i>p</i> -CH ₃ C ₆ H ₄	63
3	6c	<i>p</i> -CH ₃ C ₆ H ₄	<i>p</i> -CH ₃ C ₆ H ₄	<i>p</i> -CH ₃ C ₆ H ₄	<i>p</i> -ClC ₆ H ₄	68
4	6d	<i>p</i> -CH ₃ C ₆ H ₄	<i>p</i> -CH ₃ C ₆ H ₄	<i>p</i> -CH ₃ OC ₆ H ₄	<i>o</i> -CH ₃ C ₆ H ₄	64
5	6e	<i>p</i> -CH ₃ C ₆ H ₄	<i>p</i> -CH ₃ OC ₆ H ₄	<i>p</i> -CH ₃ OC ₆ H ₄	<i>o</i> -CH ₃ C ₆ H ₄	62
6	6f	<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	<i>p</i> -CH ₃ OC ₆ H ₄	66
7	6g	<i>p</i> -CH ₃ C ₆ H ₄	<i>p</i> -ClC ₆ H ₄	C ₆ H ₅	C ₆ H ₅	59
8	6h	<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	<i>p</i> -ClC ₆ H ₄	60
9	6i	<i>p</i> -CH ₃ C ₆ H ₄	<i>p</i> -ClC ₆ H ₄	<i>p</i> -CH ₃ OC ₆ H ₄	<i>p</i> -NO ₂ C ₆ H ₄	69
10	6j	<i>p</i> -CH ₃ C ₆ H ₄	<i>p</i> -ClC ₆ H ₄	<i>p</i> -ClC ₆ H ₄	<i>p</i> -CH ₃ OC ₆ H ₄	63
11	6k	<i>p</i> -ClC ₆ H ₄	<i>p</i> -CH ₃ OC ₆ H ₄	<i>p</i> -CH ₃ C ₆ H ₄	<i>m</i> -ClC ₆ H ₄	61
12	6l	<i>p</i> -ClC ₆ H ₄	<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	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

a. Reaction 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, 4h; 3. DDQ (1.2 mmol), rt, 1h; b. Isolated yields.

For explaining the formation of the various compounds, a plausible reaction mechanism was proposed in Scheme 1 on the basis of the previously reported reaction [42, 47]. Firstly, the DDQ oxidative dehydrogenation of 3-(indol-3-yl)-1,3-

diphenylpropan-1-one gave the expected indole-chalcone (**A**), which has the typical scaffold of 3-vinylindole as a reactive diene. Secondly, a Diels-Alder reaction of indole-chalcone with the added dienophilic chalcone resulted in a tetrahydrocarbazole (**B**) with an exocyclic C=C bond. Thirdly, a new tetrahydrocarbazole intermediate (**C**) was formed by the 1,3-H shifting process. The resulting tetrahydrocarbazole intermediate (**C**) might have several possible diastereoisomers because it has four substituents on the cyclohexenyl ring. After further DDQ oxidation, the aromatized carbazole **6** was successfully produced as the final product.



Scheme 2 proposed domino reaction mechanism for the carbazoles **6**

Conclusion

In summary, we have investigated the one-pot domino Diels-Alder of 3-(indol-3-yl)maleimides and in situ generated indole-chalcone with chalcones. This reaction successfully provided extremely simple and high efficient protocol for polyfunctionalized carbazole derivatives. This reaction has the advantages of using readily available starting reagents, simple manufacture, high efficiency and atomic economy. The unusual feature of this reaction is normal electron-demand Diels-Alder

reaction between electron-deficient dienes (3-(indol-3-yl)maleimides and indole-chalcone to electron-deficient dienophilic chalcone. 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-4l: To a round 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 80 °C for two hours. After removing the solvent by rotatory evaporation, DDQ (1.2 mmol) and acetonitrile (10.0 mL) was added. 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 (V/V = 8:1) as eluent to give the pure product for analysis.

5-Benzoyl-2-phenyl-4-(*p*-tolyl)pyrrolo[3,4-*c*]carbazole-1,3(2*H*,6*H*)-dione (4a): green solid, 87%, m.p. 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; MS (*m/z*): HRMS (ESI-TOF) Calcd. for C₃₄H₂₂NaN₂O₃ ([M+Na]⁺): 529.1523, Found: 529.1512.

2. General procedure for the preparation of the carbazoles 6a-6n: To a round flask was 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 half hour. Then,

chalcone (0.5 mmol) and *p*-toluenesulfonic acid (0.06 mmol) was added. The solution was refluxed for four hours. After cooling to room temperature, DDQ (1.2 mmol) was added. 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 (V/V/V = 20: 1:5) to give pure product for analysis.

(9-Methyl-2,4-diphenyl-9H-carbazole-1,3-diyl)bis(phenylmethanone) (6a): White solid, 61%, m.p. 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; MS (*m/z*): HRMS (ESI) Calcd. for C₃₉H₂₇NO₂ ([M+Na]⁺): 564.1934, found: 564.1926.

Supporting Information

Characterization data and ¹H, ¹³C, HRMS spectra of the compounds are available. The crystallographic data of the compounds **3a** (CCDC 2099074), **3b** (CCDC 2099075), **4g** (CCDC 2099076) have been deposited at the Cambridge Crystallographic Database Center (<http://www.ccdc.cam.ac.uk>)

Acknowledgements

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