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Molecular diversity of the reactions of MBH carbonates of isatins and various nucleophiles

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Full Research Paper

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Abstract

In this paper, the nucleophilic substitution reactions of various N- and P-containing nucleophiles to MBH carbonates of isatins were investigated. Diverse functionalized 3-substituted oxindole derivatives were successfully prepared in satisfactory yields and with high diastereoselectivity. In addition, the base-promoted dimerization of MBH carbonates of isatin afforded the ethylene-bridged bis(3-methylene)oxindole derivatives with nearly 4:1 diastereomeric ratios. The relative configurations of the various polycyclic compounds were clearly elucidated by determination of several single crystal structures.

Introduction

Isatins (indoline-2,3-diones) possessing an indole motif with a ketone and a γ-lactam moiety occur in numerous natural substances [1-4]. Isatins have many interesting aspects in organic reactions and potential applications. The versatile reactivity of isatins used both as an electrophiles and nucleophiles and their easy availability have made them to become valuable building blocks in organic synthesis. Nucleophilic additions or spiroannulation of the highly reactive carbonyl group at the C-3 position of isatins have various fascinating applications in organic synthesis, which allowed transformation of isatins into various biologically important 3-substituted and spirooxindole derivatives [5-7]. The unique molecular architecture and remarkable pharmacological activities have made spirooxindole one of the hottest synthetic targets with fruitful chemical and steric selectivity [8-15].

In recent years, the readily available Morita–Baylis–Hillman (MBH) carbonates of isatins have been recognized as one of the most powerful synthons for the construction of various spirooxindole skeletons [16-25]. MBH carbonates of isatins can be easily obtained via base-promoted MBH reactions of isatins with alkyl acrylates, acrylonitrile, and other activated alkenes [26-29]. Due to the stronger electron-withdrawing effect of the oxindole motif, MBH carbonates of isatins showed higher reactivity than that of the normal MBH carbonates of normal ketones [30-35]. In the presence of a Lewis base, MBH carbon-

ates of isatins can be easily converted to activated allylic ylides, which in turn can undergo allylic substitution and annulation reactions to give diverse 3-substituted and spirooxindole derivatives [36-41]. Inspired by these elegant synthetic methodologies and in continuation of our aim to develop domino reactions based on MBH carbonates of isatins for efficient construction of diverse polycyclic spiroindolinones [42-52], herein, we wish to report the nucleophilic substitution reactions of various N- and P-containing nucleophiles to MBH carbonates of isatins and convenient synthesis of diverse functionalized 3-substituted oxindole derivatives.

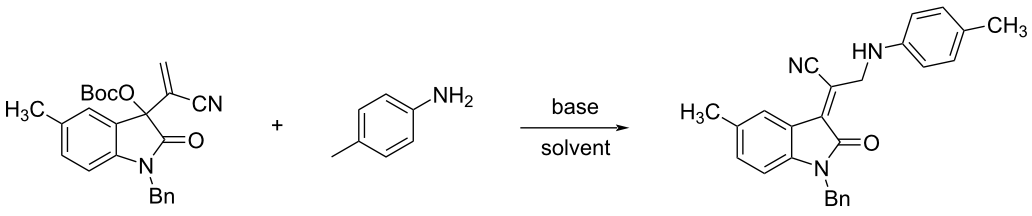
Results and Discussion

Initially, the reaction conditions were briefly examined by using MBH nitrile of isatin **1a** and *p*-toluidine (**2a**) as model reaction. It was found that the reaction did not proceed in DMF as solvent (Table 1). When the reaction was carried out in dichloromethane, dichloroethane, acetonitrile, and toluene, the expected product **3a** was produced in low yields (entries 2–5 in Table 1). The reaction in toluene at elevated temperature resulted in a slight increase of the yield (entries 6 and 7 in Table 1). In the presence of 20 mol % of DMAP, the reaction in toluene at room temperature afforded the product **3a** in 72% yield. However, increasing the amount of DMAP decreased the yield (entries 8–10 in Table 1). Additionally, stronger bases such as DABCO, DBU, triethylamine, and K_2CO_3 also resulted in the significant reduction of the yields. Therefore, the reaction of MBH nitrile of isatins and arylamines can be simply carried out in toluene at room temperature in the presence of a catalytic amount of DMAP.

With the optimized reaction conditions in hands, the scope of the reaction was investigated by using various MBH nitriles of isatins and aromatic amines in the reaction. The results are summarized in Scheme 1. All reactions proceeded smoothly to give the expected allylic S_N2-substituted products **3a–i** in satisfactory yields. Aromatic amines with various substituents could be employed in the reaction to give the expected products. *N*-Methylaniline and *n*-butylamine were also successfully converted to the desired products **3h** and **3i** in 72% and 63% yields, respectively. The structures of compounds **3a**–**i** were fully characterized by various spectroscopy techniques.

In order to show the universality of the substitution reaction, MBH maleimides of isatins **4** were also employed in the reaction. In the absence of any base as promoter, the reaction of

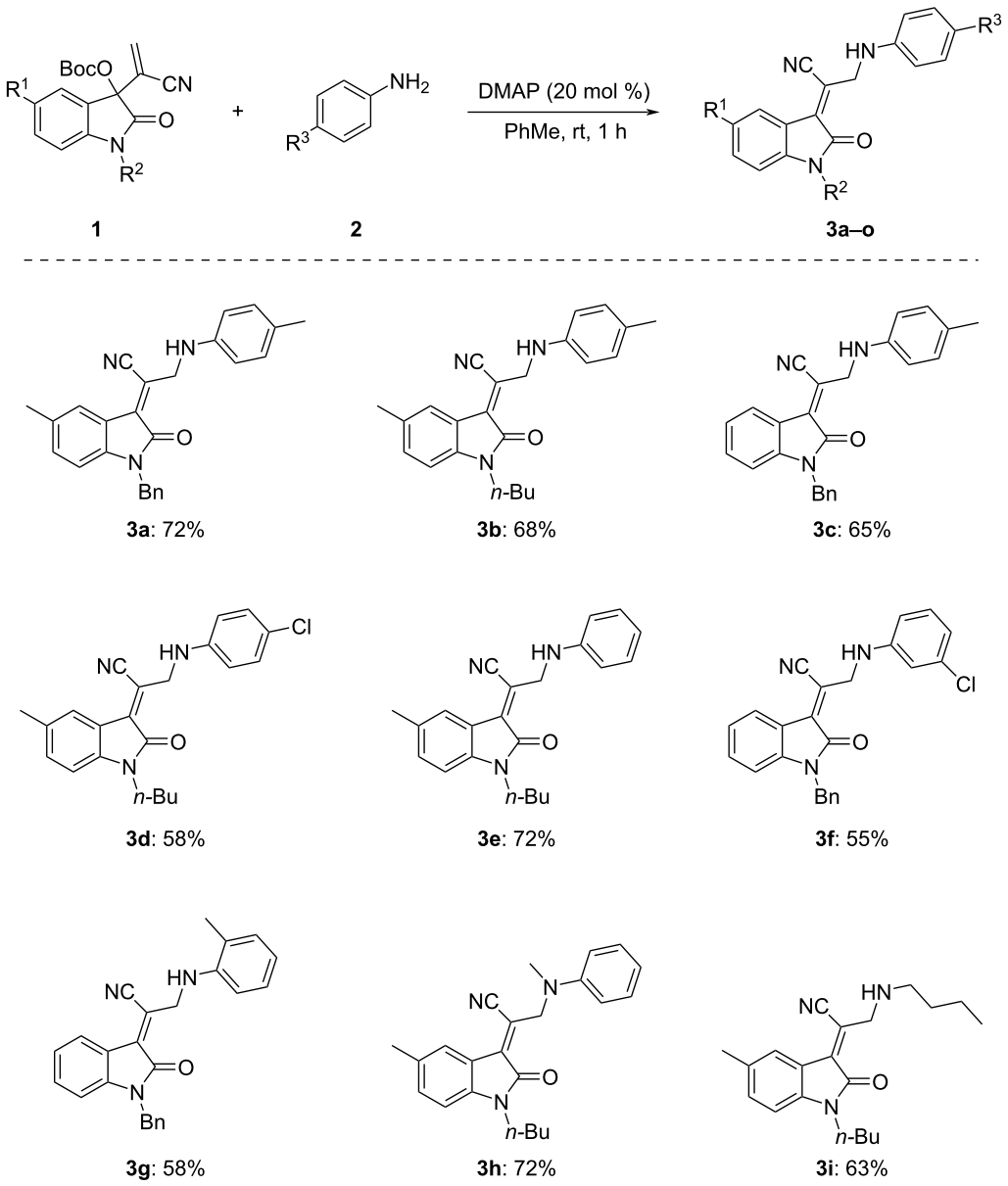
Table 1: Optimization of reaction conditions.[a]



1a **2a** **3a**

Entry	Base	Solvent	*T* [°C]	Time [h]	Yield [%][b]
1	–	DMF	rt	4	–
2	–	CH_2Cl_2	rt	4	12
3	–	DCE	rt	4	15
4	–	CH_3CN	rt	4	18
5	–	toluene	rt	4	20
6	–	toluene	60	4	39
7	–	toluene	80	4	47
8	DMAP (0.2 equiv)	toluene	rt	1	72
9	DMAP (0.3 equiv)	toluene	rt	4	45
10	DMAP (0.5 equiv)	toluene	rt	1	36
11	DABCO (0.2 equiv)	toluene	rt	1	<10
12	DBU (0.3 equiv)	toluene	rt	4	15
13	Et_3N (0.3 equiv)	toluene	rt	4	<10
14	K_2CO_3 (0.3 equiv)	toluene	rt	4	20

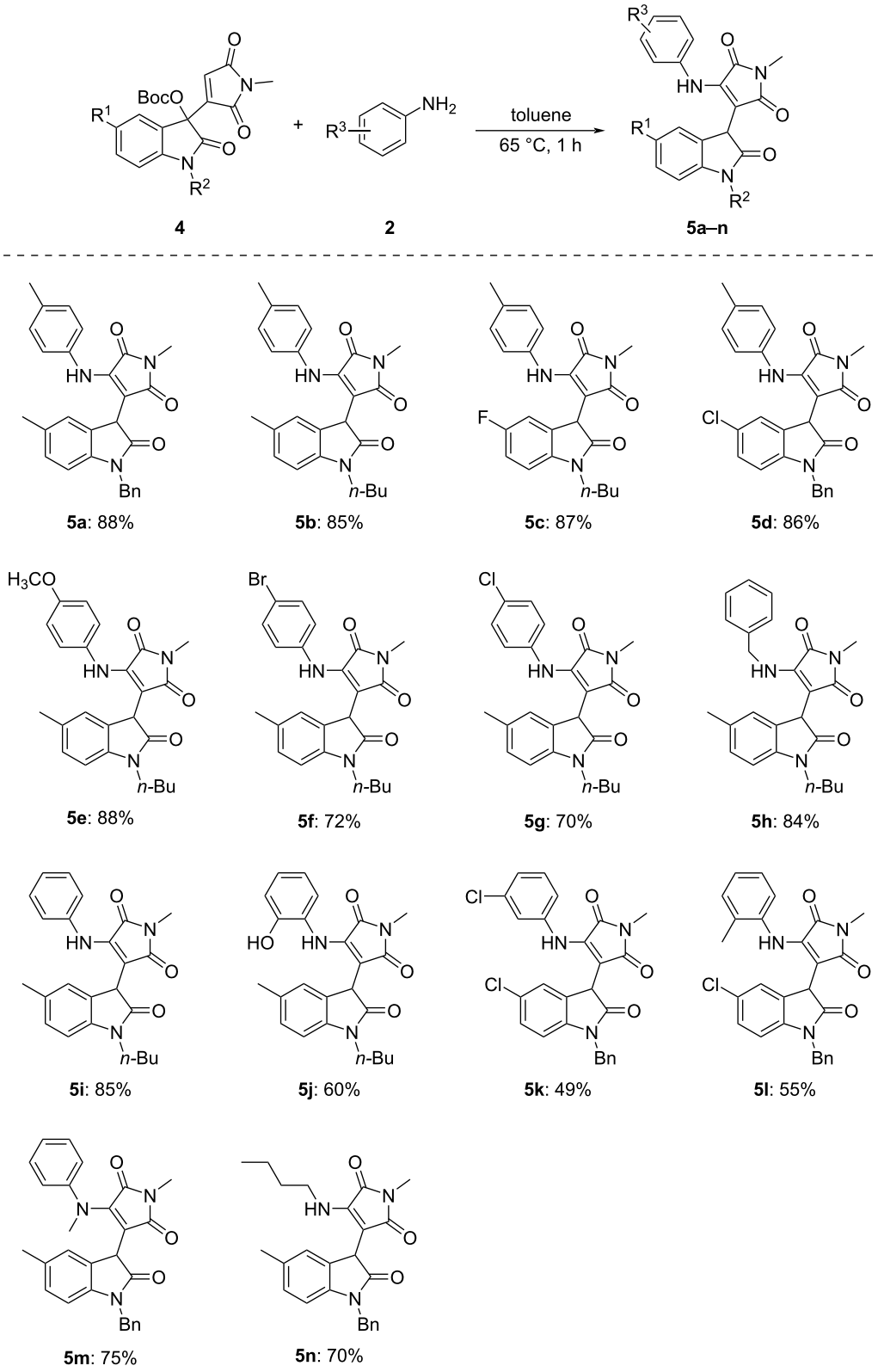
[a]Reaction conditions: MBH nitrile of isatin (0.1 mmol), *p*-toludine (0.1 mmol), solvent (5.0 mL); [b]Isolated yield.



Scheme 1: Reaction of arylamines and MBH carbonates of isatins. Reaction conditions: MBH carbonate of isatin (0.1 mmol), aromatic amine (0.1 mmol), DMAP (0.02 mmol, 20 mol %), toluene (5.0 mL), rt, 1 h; yields refer to isolated yields.

MBH maleimides of isatins with various aromatic amines in toluene at 65 °C gave the expected substitution products **5a**–**n** in high yields (Scheme 2). The substituents showed a marginal effect on the yields. In addition, a secondary arylamine such *N*-methylaniline and aliphatic amine (*n*-butylamine) also gave the expected products **5m** and **5n** in satisfactory yields. It should be pointed out that no base promoter is needed in the reaction, which is assumed to be due to the much higher reactivity of MBH maleimides of isatins compared to the above-mentioned MBH nitriles and formats of isatins. The single crystal structures of the compounds **5a** and **5j** were successfully determined (Figure 1 and Figure 2). From Figure 1 and Figure 2, it can be found that the C=C bond is located in the unit of the pyrrolidine-2,5-dione, while the scaffold of indolin-2-one is connected via a C–C single bond with the unit of the pyrrolidine-2,5-dione. Therefore, an allyl rearrangement must proceed in the reaction process, which is very different to that of the above mentioned reactions of MBH nitriles and formates of isatins.

It has been reported that triphenylphosphine can catalyze the cycloaddition reaction of MBH carbonates of isatins with some



Scheme 2: Reaction of arylamines and MBH maleimides of isatins. Reaction conditions: MBH maleimide of isatin (0.1 mmol), aromatic amine (0.1 mmol), toluene (5.0 mL), 65 °C, 1 h; yields refer to isolated yields.

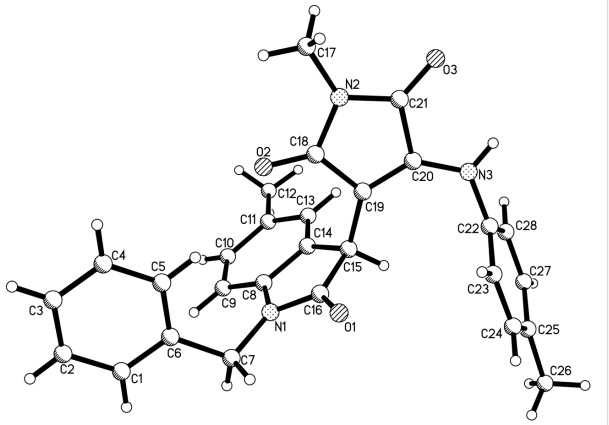


Figure 1: Single crystal structure of compound **5a**.

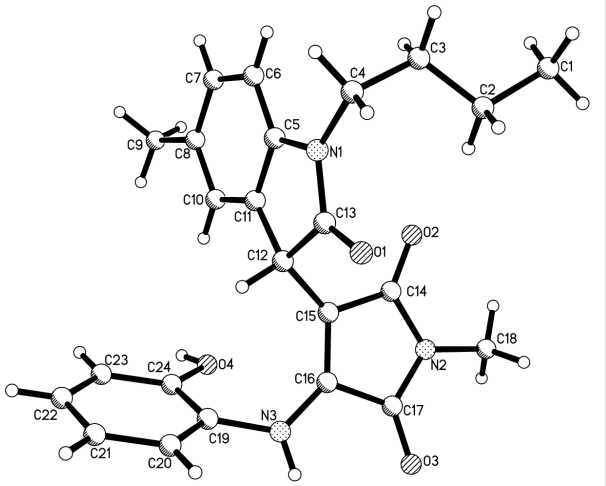
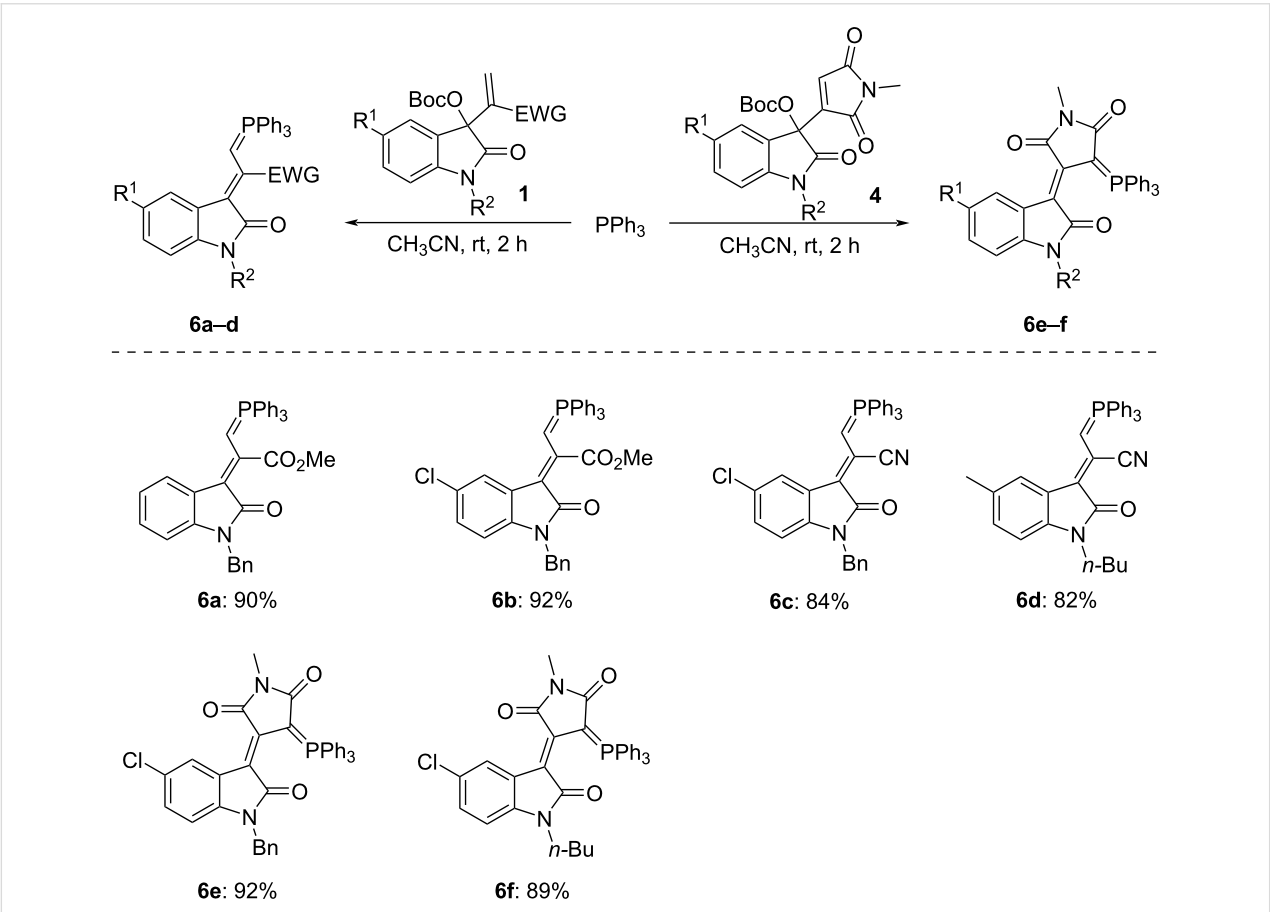


Figure 2: Single crystal structure of compound **5j**.

activated alkenes to give diverse spirooxindoles [38-41]. The reaction of triphenylphosphine with MBH nitriles of isatins in acetonitrile at room temperature quickly gave red solid products **6a–d** in high yields (Scheme 3). In this reaction, triphenylphosphine acted as a nucleophile to finish an allylic S_N2 reaction. The obtained triphenylphosphaneylidenes are stable, which



Scheme 3: Reactions of MBH carbonates of isatins and triphenylphosphine. Reaction conditions: MBH carbonate of isatin (0.1 mmol), triphenylphosphine (0.1 mmol), CH_3CN (5.0 mL), rt, 2 h; yields refer to isolated yields.

can be isolated and were fully characterized via various spectroscopy methods. The further annulation reaction did not proceed under the reaction conditions. The similar reaction of triphenylphosphine and MBH maleimides of isatins also resulted in the corresponding triphenylphosphaneylidenes **6e** and **6f** in satisfactory yields. The single crystal structure of the compound **6e** was determined by X-ray diffraction (Figure 3).

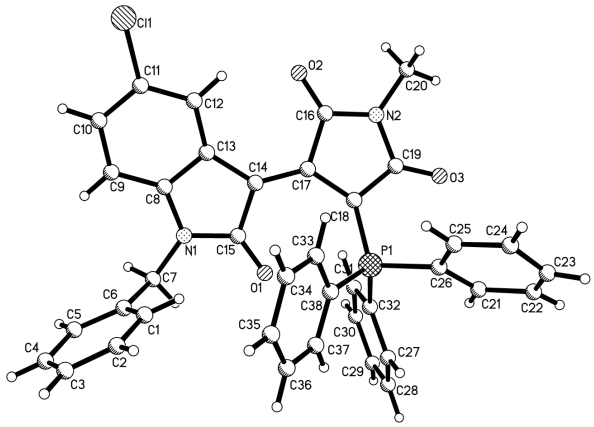
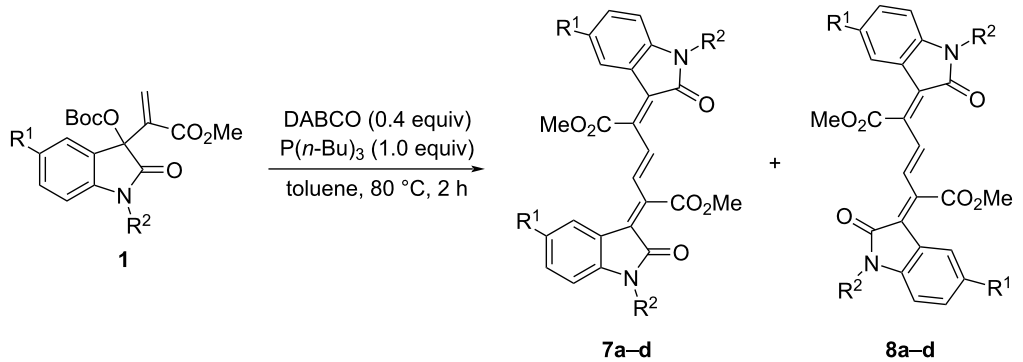


Figure 3: Single crystal structure of compound **6e**.

The similar reaction of more nucleophilic tri(*tert*-butyl)phosphine and MBH carbonate of isatin resulted in a mixture of products. After carefully screening the reaction conditions, we found that DABCO successfully promoted reaction of tri(*tert*-butyl)phosphine and MBH carbonates of isatins in toluene at 80 °C for three hours. These conditions resulted in mixtures of conjugated triene *cis/trans*-isomers **7a**–**d** and **8a**–**d** with nearly 4:1 ratios (Table 2). It should be pointed out that similar conjugated trienes have been obtained by DABCO-catalyzed dimerization of MBH carbonates of isatin in ODCB at high temperature (150 °C), which could be converted to bisspirooxindole derivatives via the isomerization of the double bond and a sequential thermal 6π-electrocyclic ring-closure process [30]. Here, the novel triene derivatives can be prepared under relative mild reaction conditions with good steric selectivity. The single crystal structures of compounds **7a** and **8a** were successfully determined by X-ray diffraction method (Figure 4 and Figure 5). From the crystal structures, it can be found that the compound **7a** has an *E,E,E*-configuration, while the compound **8a** has a *Z,E,E*-configuration.

On the basis of the above experiments and the previously published results [42-52], a plausible reaction mechanism was proposed in Scheme 4 to explain the formation of the various oxindole derivatives. At first, a Lewis base attack at the α-position of the MBH nitrile of isatin resulted in the intermediate **A** with elimination of carbon dioxide and *tert*-butoxide ion. Secondly, the product **3** was produced by the S_N2 substitution of the Lewis base by the arylamine. When MBH maleimides of isatin were used in the reaction, a direct Michael addition of the

Table 2: Dimerization of MBH carbonates of isatins.[a]



Entry	R^1	R^2	Compd	Yield [%][b]	Compd	Yield [%][b]
1	H	Bn	**7a**	63	**8a**	15
2	CH_3	*n*-Bu	**7b**	62	**8b**	14
3	Cl	*n*-Bu	**7c**	66	**8c**	12
4	F	Bn	**7d**	65	**8d**	13

[a]Reaction conditions: MBH carbonate of isatin (0.1 mmol), tri(*n*-butyl)phosphine (0.1 mmol), DABCO (0.04 mmol), toluene (5.0 mL), 80 °C, 2 h; [b]Isolated yield.

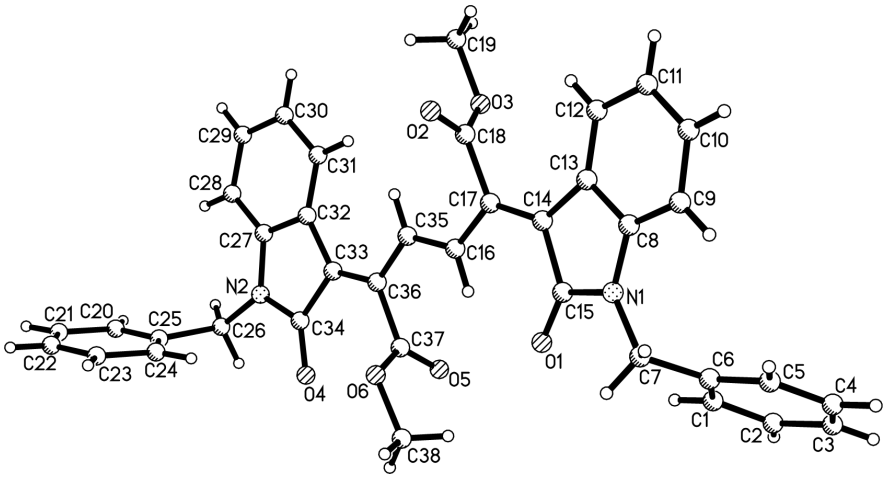


Figure 4: Single crystal structure of compound **7a**.

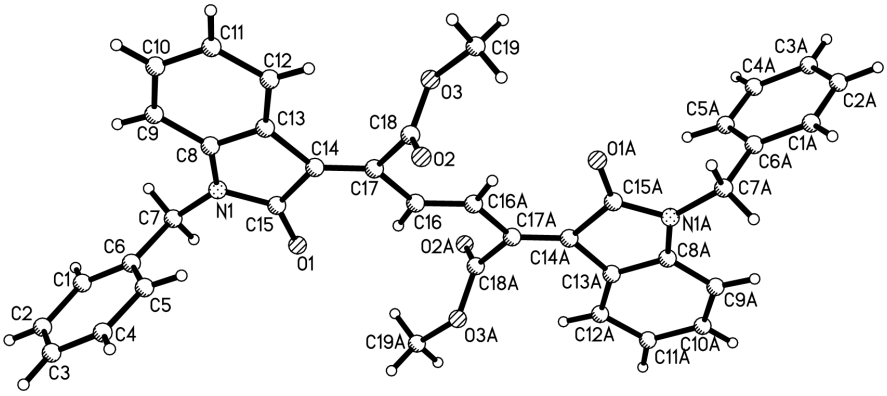
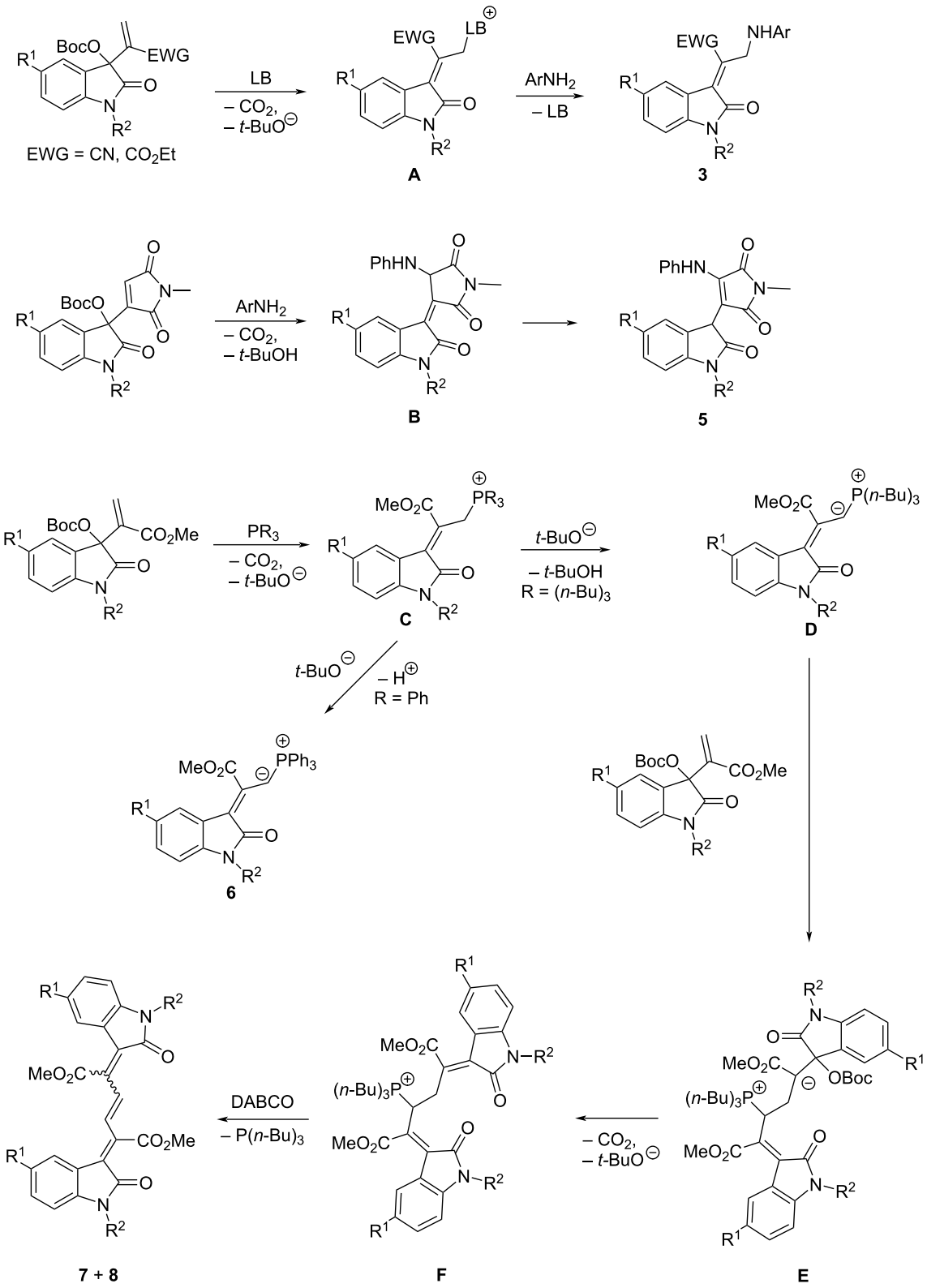


Figure 5: Single crystal structure of compound **8a**.

arylamine to the C=C bond of the maleimide unit and sequential elimination of carbon dioxide and *tert*-butoxide ion gives the intermediate **B**, which in turn undergoes an allylic rearrangement to afford the product **5**. In this process, no extra addition of Lewis base is needed. When triphenylphosphine or tri(*n*-butyl)phosphine were involved in the reaction, the similar S_N2' substitution of tri(*n*-butyl)phosphine with the elimination of carbon dioxide and *tert*-butoxide anion gives the phosphonium salt **C**. Then, the deprotonation of phosphonium salt **C** by the *tert*-butoxide anion affords the zwitterionic intermediate **D**. In the case of triphenylphosphine, the similar zwitterionic intermediate **D** is stable and could be isolated as the product **6**. In the case of tri(*n*-butyl)phosphine, Michael addition of the more activated zwitterionic intermediate **D** to MBH formate of isatin resulted in the intermediate **E**. Then, the elimination of carbon dioxide and *tert*-butoxide anion gives the intermediate **F**. Finally, the base-promoted elimination of tri(*n*-butyl)phosphine resulted in the *Z*/*E* isomers of the triene derivatives **7** or **8**.

Conclusion

In summary, we have investigated nucleophilic substitution reactions of various N- and P-containing nucleophiles to MBH carbonates of isatins. It is interesting to find that diverse functionalized 3-substituted oxindole derivatives were successfully prepared in satisfactory yields and with high diastereoselectivity. In addition, the base-promoted dimerization of MBH carbonates of isatin afforded the ethylene-bridged bis(3-methylene)oxindole derivatives with nearly 4:1 ratios. This reaction not only clarified the essence of the substitution reaction of MBH carbonates of isatin with various N-, P-containing nucleophiles, but also led to the development of a synthetic protocol



Scheme 4: Proposed reaction mechanism for the various compounds.

for the convenient synthesis of 30 diversely substituted oxindole derivatives.

Supporting Information

The crystallographic data of compounds **5a** (CCDC 2390713), **5j** (CCDC 2390714), **6e** (CCDC 2390715), **7a** (CCDC 2390716) and **8a** (CCDC 2390717) have been deposited at the Cambridge Crystallographic Database Center.

Supporting Information File 1

Experimental procedures, ^{1}H, ^{13}C NMR, and HRMS spectra for all new compounds.
[https://www.beilstein-journals.org/bjoc/content/supplementary/1860-5397-21-21-S1.pdf]

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Author Contributions

Zi-Ying Xiao: investigation. Jing Sun: data curation. Chao-Guo Yan: supervision; writing – review & editing.

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Data Availability Statement

All data that supports the findings of this study is available in the published article and/or the supporting information of this article.

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