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Rapid Access to Spirocylic Oxindoles: Application of Asymmetric N-Heterocyclic Carbene-Catalyzed [3 + 3] Cycloaddition of Imines to Oxindole-Derived Enals

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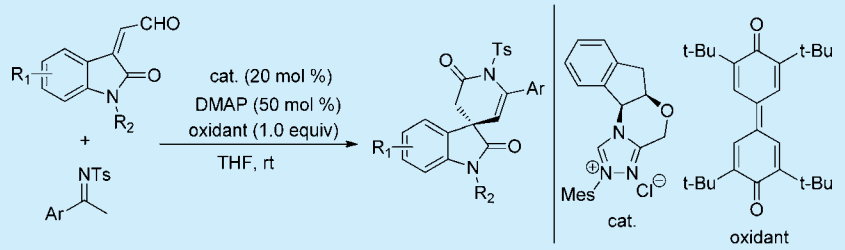
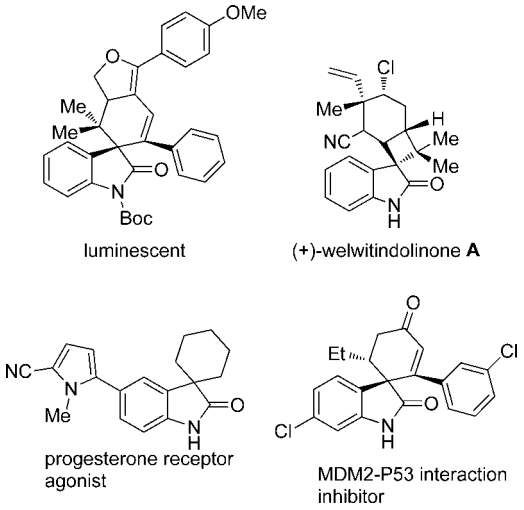
Supporting Information



ABSTRACT: A chiral N-heterocyclic carbene (NHC)-catalyzed [3 + 3] cycloaddition reaction of imines and oxindole-derived enals was developed for rapid access to spirocylic oxindoles. In most cases, the desired spirocylic oxindole products were obtained in high yields and excellent enantioselectivities with less than 1 h of reaction time.

Spirocyclic oxindoles represent important scaffolds in a large family of clinical pharmaceuticals and natural products such as luminescents, welwitindolinone A, progesterone receptor agonists, and MDM2-P53 interaction inhibitors (Scheme 1).[1] These compounds display a wide range of biological activities such as antimalarial, antituberculosis, growth hormone secretagogue, and antibacterial properties.[2] Consequently, many approaches have been developed for the efficient and stereoselective synthesis of this core motif. For example, the metal-catalyzed domino reaction, involving C−H activation, has been widely documented.[3] More recently, organocatalyzed reactions have emerged as attractively synthetic strategies by using cinchona alkaloid, phospine, or phosphoric acid as catalysts.[4] Although considerable progress has been described toward their syntheses, development of a novel method to efficiently synthesize this core structure remains valuable.

Scheme 1. Examples of Biological Active Spirocyclic Oxindoles



Over the past decade, NHCs have received significant interest in organocatalyzed reactions because of their special electronic characteristics.[5] Making use of intriguing organocatalytic activation of NHC for new bond formation opens up another avenue for the synthesis of target molecules. For instance, the traditional a^1-d^1 umpolung (benzoin condensation[6] and Stetter reaction[7]) and a^3-d^3 umpolung (homoenolate cycloaddition[8]) approaches have been well-documented. In particular, (oxidative) NHC organocatalysis has been extended to α-carbon[9] and β-carbon activation of saturated aldehydes, enals, ynals, α-bromo enals, and α,β-unsaturated esters. The formation of formal Michael acceptors is an important milestone in NHC organocatalysis,[10] which is different from the traditional umpolung reactions. The pioneering investigation was completed by Studer and De Sarkar by the addition of 1,3-dicarbonyl compounds to α,β-unsaturated acyl triazoliums generated from α,β-unsaturated aldehydes.[11] Alternately, Lupton and co-workers reported the NHC-mediated formation of Michael acceptors from α,β-unsaturated enol esters and acyl fluorides.[12] Chi, Scheidt, and Ye also documented the generation of α,β-unsaturated acyl azolium

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intermediates from α,β-unsaturated carboxylic acid derivatives.[13] Following our interest in NHC organocatalysis methodology and the synthesis of spirocyclic oxindoles,[14] we herein report that, in the presence of TFA, NHC-catalyzed [3 + 3] cycloaddition reaction with imines and oxindole derived enals takes place to afford spirocyclic oxindoles.

The chemistry described in this paper builds on the investigation of exploring the NHC-catalyzed reactions of oxindole-derived enals with a variety of imines. In the previous studies, the formal Michael acceptor could be generated from the reaction of saturated enals and the NHC catalysts under appropriate conditions. In our report, the formal Michael acceptor generated from an oxindole-derived enal with NHC catalyst can be engaged in an enantiocontrolled Michael-addition type reaction with imines, which facilitates the formation of new six-membered lactams. The importance of this methodology is due to its great potential for accessing spirocyclic oxindole products.

One of the challenges of this reaction lies in the versatile reactivities of enals, which can undergo unwanted [3 + 2] cycloaddition reactions with imines catalyzed by NHC. Furthermore, imines, which are readily accessible substrates, can easily transfer to enamine under the action of base. This can, in turn, lead to the generation of β-quaternary carbon aldehyde by direct Michael addition with isatin-derived enals instead of a [3 + 3] cycloaddition reaction.

To investigate this unprecedented [3 + 3] cycloaddition reaction of isatin-derived enals and imines, we first surveyed the readily prepare disatin-derived enal **1a** and acetophone-derived imine **2a** in the presence of chiral *N*-mesityl-substituted triazolium salt **A**, different bases, solvents, and additives for optimization studies (Table 1). When oxidant **B** was used, the desired product was afforded in excellent enantioselectivity and moderate yield. Next, the use of different bases and solvents was evaluated for this reaction. DBU was found to slightly decrease the enantioselectivity (Table 1, entry 4). When Cs_2CO_3 was employed, the yield decreased dramatically even though there was no loss in enantioselectivity (Table 1, entry 5). We observed that the inorganic bases were deemed to be unsuitable for the reaction as they led to lower yields and enantioselectivities (Table 1, entry 6).

The screening of solvents revealed that THF was the most ideal (Table 1, entry 10), while CH_2Cl_2 gave the worst result with 21% yield and 97% ee with prolonged reaction time (18 h, Table 1, entry 9). Use of CH_3CN as solvent proceeded with 43% yield and enantioselectivity (97% ee), comparable to the results using THF with prolonged reaction time (15 h, Table 1, entry 8). It is particularly noteworthy that when the additive TFA was added in 2.5 mol % scale to this reaction the desired product could be afforded with slightly increased enantioselectivity (99% ee) and no loss in yield (Table 1, entry 10). It is probably ascribed to the role of TFA on forming Michael donor from imine to enamine and promoting the reaction.

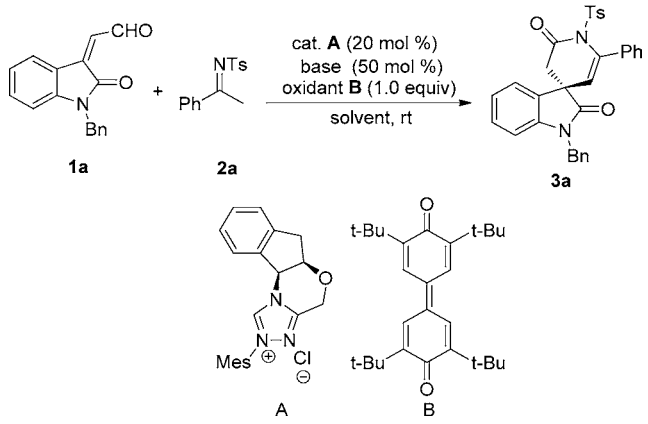
With the optimal reaction conditions established, the scope of this [3 + 3] cycloaddition was investigated (Scheme 2). The reaction proceeded smoothly to afford the desired products in high yields with excellent optical purity. The generality of [3 + 3] cycloaddition was investigated by using various isatin-derived enals and imines. The reaction proceeded smoothly for imines bearing electron-withdrawing groups on the aryl ring (Scheme 2, **3b**–**g**). Similarly, electron-donating groups (Scheme 2, **3h**,**i**) were also well tolerated, with 4-methoxyphenyl and 4-methylphenyl enals giving the highest enantioselectivity (99% ee), despite a relatively lower yield. 2-Thienyl-derived imine provided good yield and excellent enantioselectivity (Scheme 2, **3j**). The position of the substituent on the phenyl ring seemed to have little influence on reaction outcome (Scheme 2, **3b**,**e**,**f**).

We then varied the enal component of this reaction. Excellent yields and high enantioselectivities were obtained in most cases. The enal derived from *N*-methyl-protected isatin was well tolerated, affording good yield and the highest enantioselectivity (Scheme 2, **3p**). When enals drived from electron-withdrawing groups on the phenyl ring of isatin part were employed, the reaction proceeded smoothly regardless of the lower yield (Scheme 2, **3k**–**m**). Meanwhile, electron-donating group substituted substrates provided slightly increased yields with excellent enantioselectivities (Scheme 2, **3n**,**o**). The reactions typically took 1 h or less to reach completion.

To determine the stereochemistry of the spirocyclic oxindoles formed via the asymmetric NHC-catalyzed [3 + 3] cycloaddition with imines and oxindole-derived enals, the absolute configuration of the product **3o** was determined by X-ray crystallographic analysis (Figure 1, CCDC 1052886). The (1*S*,2*R*)-(+)-*cis*-*N*-mesityl-substituted triazolium salt prepared from (1*S*,2*R*)-(+)-*cis*-1-aminoindan-2-ol provided exclusively (*R*)-spiro[indoline-3,4′-pyridine]-2,2′-dione (**3o**).

In summary, we have described a rapid access to spirocyclic oxindoles via NHC-catalyzed [3 + 3] cycloaddition of imines with oxindole-derived enals. In most cases, the desired products were obtained in excellent enantioselectivities (96–99% ee) with less than 1 h reaction time. This protocol holds great

Table 1. Optimization of Reaction Conditions[a]



entry	base	solvent	additive	time (h)	yield[b] (%)	ee[c] (%)
1	K_2CO_3	THF		8	60	98
2	DIEPA	THF		2.5	58	89
3	DMAP	THF		1	80	96
4	DBU	THF		0.5	41	94
5	Cs_2CO_3	THF		2.5	37	98
6	NaOAc	THF		2	41	90
7	TEA	THF		2	72	97
8	K_2CO_3	CH_3CN		15	43	98
9	K_2CO_3	CH_2Cl_2		18	21	97
10[d]	**DMAP**	**THF**	**TFA**	**1**	**79**	**99**
11[d]	DMAP	THF	HAc	1	77	98

[a]Reaction conditions: enal **1** (0.15 mmol), imine **2** (0.1 mmol), catalyst (0.02 mmol), base (0.05 mmol), and oxidant (0.1 mmol) in solvent (1 mL). The mixture was stirred for 0.5–18 h at rt. [b]Yields of isolated product. [c]ee values determined by HPLC analysis on Chiralpak AD-H or IB column. [d]The additive was added in 2.5 mol %.

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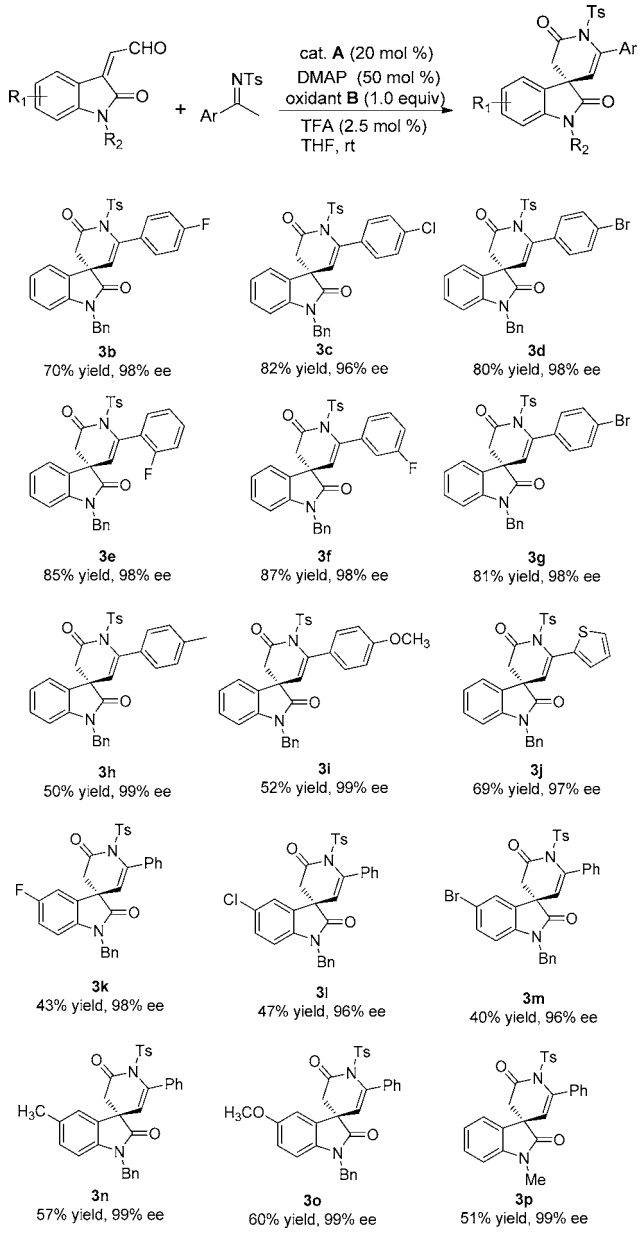
Scheme 2. Substrate Scope of Reactions



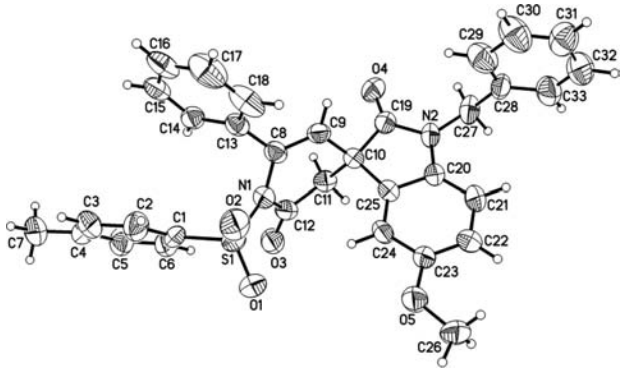


Figure 1. X-ray crystal structure of **3o**. Thermal ellipsoids were shown at 50% probability.

potential in the synthesis of biologically active spirocylic oxindole derivatives in high enantiomeric purity.

■ ASSOCIATED CONTENT

Supporting Information

Experimental procedures, spectral data, and X-ray data (CIF) for the products. The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.5b00726.

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Notes

The authors declare no competing financial interest.

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