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Enantioselective Synthesis of 3-Alkynyl-3-hydroxyindolin-2-ones by Copper-Catalyzed Asymmetric Addition of Terminal Alkynes to Isatins

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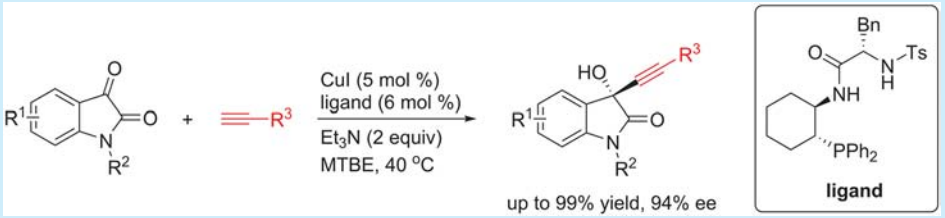
Supporting Information



ABSTRACT: A highly efficient copper-catalyzed asymmetric addition of terminal alkynes to isatins has been developed. In the presence of a catalytic amount of copper iodide and a chiral phosphine ligand, the reaction gave the corresponding chiral 3-alkynyl-3-hydroxyindolin-2-ones in high yields with high enantioselectivity. This methodology has a broad substrate scope, and the synthetic utility of the present protocol was further demonstrated by the transformation of chiral alkynylation products.

The chiral 3,3-disubstituted oxindole framework is a privileged motif that exits in various natural products and pharmaceutically active compounds.[1] As a class of important 3,3-disubstituted oxindoles, 3-substituted 3-hydroxyindolin-2-ones have attracted considerable attention, and much effort has been devoted to their asymmetric synthesis because of their diverse biological activities.[2] Many synthetic methods employing chiral organocatalysts[3] or metal complex catalysts[4] have been developed. Although there has been rapid advancement in the catalytic synthesis of chiral 3-substituted 3-hydroxyindolin-2-ones, the reported methods are mainly used for the synthesis of 3-alkyl-3-hydroxyindolin-2-ones and 3-aryl-3-hydroxyindolin-2-ones. The construction of chiral 3-alkynyl-3-hydroxyindolin-2-ones remains challenging.

Transition-metal-catalyzed asymmetric addition of terminal alkynes to unsaturated bonds is one of the most efficient methods of introducing alkynyl groups with high enantioselectivity in organic synthesis owing to its high synthetic utility with high atom efficiency.[5] Many novel protocols involving the use of transition-metal catalysts, such as iridium,[6] rhodium,[7] zinc,[8] cobalt,[9] and copper,[10] for the asymmetric addition of terminal alkynes to unsaturated bonds have been reported.

To construct chiral 3-alkynyl-3-hydroxyindolin-2-ones, transition-metal-catalyzed asymmetric addition of terminal alkynes to isatins should be one of most feasible methods. However, the successful examples are confined to the synthesis of achiral ones. For example, Hao and co-workers reported the addition of terminal alkynes to isatins, and a series of achiral 3-alkynyl-3-hydroxyindolin-2-ones were prepared in moderate yields in the presence of a zinc reagent as a promoter.[11] The yields of direct alkynylation of isatins were improved by use of a NHC-silver complex[12] or a bis-NHC-silver complex[13] as a catalyst. Recently, Nair and co-workers described a practical method for the synthesis of 3-alkynyl-3-hydroxyindolin-2-ones by using a catalytic amount of CuI/DBU in the addition of terminal alkynes to isatins.[14] These results led us to focus on the development of a chiral transition metal catalyst to realize the asymmetric version. Herein, we report a highly efficient copper-catalyzed asymmetric addition of terminal alkynes to isatins, which proceeds to provide 3-alkynyl-3-hydroxyindolin-2-ones in high yields with high enantioselectivity in the presence of copper iodide and a chiral phosphine ligand as a chiral catalyst.[15]

Initially, we selected 1-benzyl-7-(trifluoromethyl)isatin (**1a**) and phenylacetylene (**2a**) as the model starting materials to test the reaction. The reaction of **1a** and **2a** in the presence of 5 mol % of CuI with 6 mol % of (*R*)-binap in toluene at 60 °C for 24 h gave trace amounts of the desired product **3aa** (Table 1, entry 1). The use of ligand **L2** afforded **3aa** in 38% yield with poor enantioselectivity (Table 1, entry 2). We then turned to a kind of chiral cyclohexane-based phosphine ligands (Figure 1).[16]

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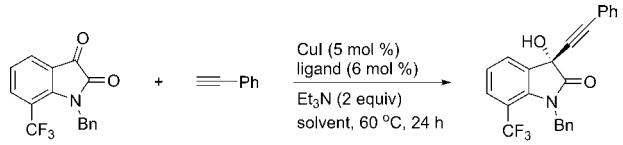
Table 1. Optimization of the Reaction Conditions[a]



entry	ligand	solvent	yield[b] (%)	ee[c] (%)
1	(*R*)-binap	toluene	trace	nd
2	L2	toluene	38	1
3	L3	toluene	29	29
4	L4	toluene	23	47
5	L5	toluene	26	58
6	L6	toluene	48	74
7[d]	L6	toluene	32	47
8[e]	L6	toluene	42	61
9[f]	L6	toluene	51	48
10	L6	$CHCl_3$	77	79
11	L6	CH_3CN	66	56
12	L6	1,4-dioxane	24	61
13	L6	THF	41	74
14	L6	MTBE	86	83
15[g]	L6	MTBE	87	92
16[h]	L6	MTBE	94	92

[a]Unless otherwise noted, the reactions were performed in a sealed tube with **1a** (0.2 mmol), **2a** (0.21 mmol), CuI (0.01 mmol), ligand (0.012 mmol), and Et_3N (0.4 mmol) in solvent (1.0 mL) at 60 °C for 24 h under N_2. [b]Isolated yields. [c]Determined by chiral HPLC analysis. [d]CuCl. [e]CuOAc. [f]CuOTf. [g]40 °C for 48 h. [h]40 °C for 72 h.

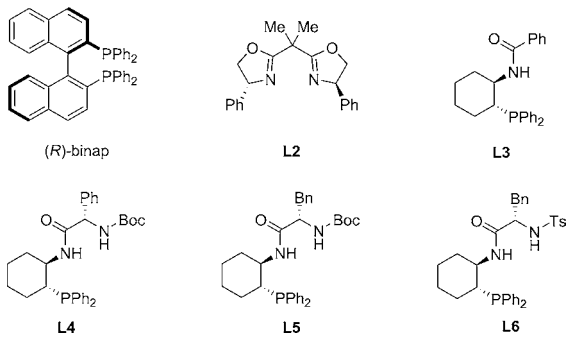
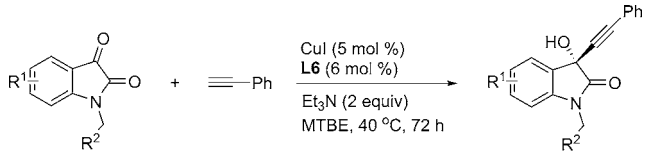


Figure 1. Structure of chiral ligands.

After screening of several chiral phosphine ligands, we were pleased to find that ligand **L6** was a good candidate giving **3aa** in 48% yield with 74% ee (Table 1, entries 3−6). Screening of copper salts revealed that copper iodide was the optimal one (Table 1, entries 7−9 vs entry 6). We investigated the effect of solvent. The use of methyl *tert*-butyl ether (MTBE) gave **3aa** in 86% yield with 83% ee (Table 1, entry 14 vs entries 10−13). These results indicated MTBE was a good solvent in this reaction. We also tested the effect of reaction temperature, and found that the reaction provided **3aa** in 87% yield with 92% ee when the reaction was carried out at 40 °C for 48 h (Table 1, entry 15). To our delight, the highest yield (94%) with highest enantioselectivity (92% ee) was obtained when the reaction was performed at 40 °C for 72 h (Table 1, entry16).

With the optimal reaction conditions in hand (Table 1, entry 16), we investigated the isatin scope, the results of which are summarized in Table 2. It was found that the reaction conditions were suitable for a variety of substituted isatins,

Table 2. Substrate Scope of Isatins[a]



entry	R^1, R^2	yield[b] (%)	ee[c] (%)
1	7-CF_3, Ph (**3aa**)	94	92
2	7-CF_3, 4-MeC_6H_5 (**3ba**)	90	91
3	7-CF_3, 4-$^tBuC_6H_5$ (**3ca**)	92	92
4	7-CF_3, 4-FC_6H_5 (**3da**)	97	92
5	7-CF_3, 4-ClC_6H_5 (**3ea**)	96	92
6	7-CF_3, 4-BrC_6H_5 (**3fa**)	88	90
7	7-CF_3, 4-CNC_6H_5 (**3ga**)	92	90
8	7-CF_3, naphthyl (**3ha**)	91	90
9	7-CF_3, H (**3ia**)	96	92
10	7-CF_3, Me (**3ja**)	98	93
11	7-CF_3, ^{n}Pr (**3ka**)	97	93
12	6-CF_3, Ph (**3la**)	91	89
13	5-CF_3, Ph (**3ma**)	90	93
14[d]	5-CO_2Me, Ph (**3na**)	78	80
15[d]	5-NO_2, Ph (**3oa**)	93	84
16	5-F, Ph (**3pa**)	87	82
17	5-Br, Ph (**3qa**)	78	86
18	H, Ph (**3ra**)	16	85
19	5-MeO, Ph (**3sa**)	33	87

[a]Unless otherwise noted, the reactions were performed in a sealed tube with **1** (0.2 mmol), **2a** (0.21 mmol), CuI (0.01 mmol), **L6** (0.012 mmol), and Et_3N (0.4 mmol) in MTBE (1.0 mL) at 40 °C for 72 h under N_2. [b]Isolated yields. [c]Determined by chiral HPLC analysis. [d]$CHCl_3$ was used instead of MTBE.

providing the corresponding products in good to excellent yields with high enantioselectivities (Table 2, entries 1−17). For example, the reaction of **1a** and **2a** in the presence of 5 mol % of CuI and 6 mol % of **L6** in MTBE at 40 °C for 72 h gave **3aa** in 94% yield with 92% ee (Table 2, entry1). The compounds of 7-CF_3-substituted isatins reacted with **2a** to give the products in high yields with high enantioselectivities (Table 2, entries 1−11). A wide range of functional groups are tolerated in the present reaction, including halogens (entries 4−6, 16, and 17), nitrile (entry 7), ester (entry 14), and nitro (entry 15). The reaction of isatins with different electron-withdrawing substituents on the 5-position and **2a** also provided the corresponding products in high yields with good to high enantioselectivities (Table 2, entries 13−17). Although the reaction gave products **3ra** and **3sa** in good enantioselectivities when isatins **1r** and **1s** were used, the yields are low (Table 2, entries 18 and 19). The absolute configuration of the alkynylation product was confirmed to be *S* by means of X-ray diffraction analysis of a single crystal of **3qa** (Figure 2).[17]

The scope of terminal alkynes was also examined. As shown in Table 3, several terminal alkyne derivatives can be employed to this reaction giving the corresponding products in good to excellent yields with high enantioselectivities (Table 3, entries 1−12). The phenylacetylenes bearing electron-donating groups or electron-withdrawing groups reacted with **1a** to provide the products in high yields with high enantioselectivities (Table 3, entries 2−9). It was found that the reactions of **1a** and terminal alkynes with naphthyl, thionyl, and cyclohexenyl groups

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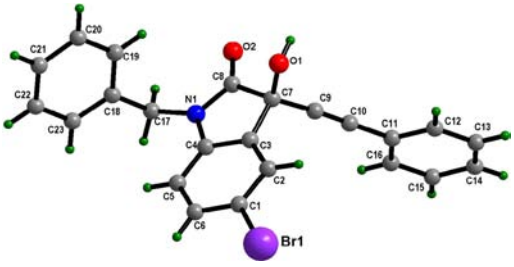
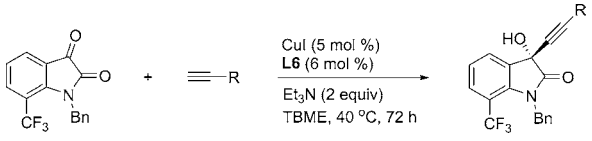


Figure 2. X-ray structure of **3qa**.

Table 3. Substrate Scope of Terminal Alkynes[a]



1a 2 3

entry	R	yield[b] (%)	ee[c] (%)
1	Ph (**3aa**)	94	92
2	2-MeOC$_6$H$_4$ (**3ab**)	99	93
3	2-MeC$_6$H$_4$ (**3ac**)	82	85
4	3-MeC$_6$H$_4$ (**3ad**)	95	94
5	4-MeC$_6$H$_4$ (**3ae**)	93	93
6	4-MeOC$_6$H$_4$ (**3af**)	78	89
7	4-EtC$_6$H$_4$ (**3ag**)	96	92
8	4-^{t}BuC$_6$H$_4$ (**3ah**)	82	91
9	4-FC$_6$H$_4$ (**3ai**)	81	88
10	1-naphthyl (**3aj**)	95	89
11	2-thionyl (**3ak**)	93	85
12	2-cyclohexenyl (**3al**)	96	91
13	^{n}Bu (**3am**)	28	84

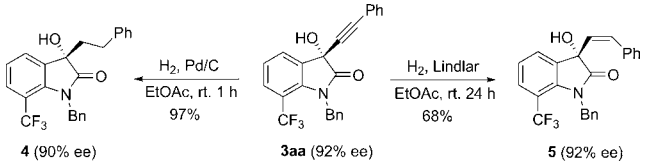
[a]The reactions were performed in a sealed tube with **1a** (0.2 mmol), **2** (0.21 mmol), CuI (0.01 mmol), **L6** (0.012 mmol), and Et_3N (0.4 mmol) in MTBE (1.0 mL) at 40 °C for 72 h under N_2. [b]Isolated yields. [c]Determined by chiral HPLC analysis.

proceeded well to give the products in high yields with high enantioselectivities (Table 3, entries 10−12). However, the reaction of **1a** and 1-hexyne afforded product **3am** in low yield with good enantioselectivity (Table 3, entry 13).

The obtained chiral 3-alkynyl-3-hydroxyindolin-2-ones **3** by the present methodology can be used as versatile intermediates and building blocks in organic synthesis. For example, the reduction of product **3aa** with Pd/C provided **4** in 97% yield with 90% ee, while reduction with Lindlar catalyst afforded **5** in 68% yield with 92% ee (Scheme 1).

In conclusion, we have developed a highly efficient copper-catalyzed asymmetric addition of terminal alkynes to isatins. The chiral 3-alkynyl-3-hydroxyindolin-2-ones were obtained in good to excellent yields with high enantioselectivities by using the combination of CuI and a chiral phosphine ligand **L6**. A variety of functional groups including halogens, ester, nitro, and

Scheme 1. Transformation of Product 3aa



4 (90% ee) **3aa** (92% ee) **5** (92% ee)

nitrile are tolerated in the present reaction. The synthetic utility of present protocol was further demonstrated by the efficient transformation of the enantiomerically enriched 3-alkynyl-3-hydroxyindolin-2-ones.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.6b00971.

Procedure, NMR spectra, X-ray data, and analytical data for all new compounds (PDF)

X-ray data for **3qa** (CIF)

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Author Contributions

‖N.X. and D.-W.G. contributed equally.

Notes

The authors declare no competing financial interest.

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(17) CCDC 1470366 (**3qa**) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre.

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