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Copper(i) reagent-promoted hydroxytrifluoromethylation of enamides: flexible synthesis of substituted-3-hydroxy-2-aryl-3-(2,2,2-trifluoro-1-arylethyl)isoindolin-1-one†

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Introduction

Isoindoles are a series of notable nitrogen-containing compounds known for their bioactivity in nature.¹ In particular, 3-hydroxyisoindolin-1-ones such as **I** and **II** are the core structural motifs of several compounds of medicinal value (Fig. 1). 3-Hydroxy isoindolin-1-ones are known for their use as diuretic and anticancer drugs.² As substituted 3-benzylidene-2-arylisooindolin-1-ones have double bonds, we try to find a catalytic system for direct hydroxytrifluoromethylation of substituted 3-benzylidene-2-arylisooindolin-1-one.

Hydroxytrifluoromethylation of organic molecules has become a research focus in the field of organic synthesis for its unique biological activities.³ In 1991, Langlois and co-workers reported the first use of $\text{CF}_3\text{SO}_2\text{Na}$ as the trifluoromethyl radical source.⁴ Since then, a series of trifluoromethylation of olefins by using Langlois reagent has been published in the last

twenty years.⁵ In these reactions, $\text{CF}_3\text{SO}_2\text{Na}$ was excited by single electron oxidations to generate CF_3 free radicals. The oxidative partners included TBHP,⁶ $\text{K}_2\text{S}_2\text{O}_8$,⁷ $\text{PhI}(\text{OAc})_2$,⁸ DTBP,⁹ I_2O_5 ,¹⁰ metal (Cu, Mn),¹¹ photoinducers¹² and so on. There have only been several examples involving hydroxytrifluoromethylation of olefins to afford useful β -trifluoromethyl alcohols.¹³ Moreover, there were also studies of the metal-free-catalyzed hydroxytrifluoromethylation reactions of styrenes.¹⁴ Recently, manganese-catalyzed direct hydroxytrifluoromethylation reaction of styrene derivatives has been established.¹⁵ On the other hand, the CuCF_3 system has been used for the synthesis of direct hydroxytrifluoromethylation reaction.¹⁶ Visible light promoted C–F functionalization has been developed under mild reaction condition.¹⁷ Until now, hydroxytrifluoromethylation of enamides has not been reported. As part of our research on the transition metal-catalyzed free radical reaction of substituted 3-benzylidene-2-arylisooindolin-1-one,¹⁸ this communication reports the first example of hydroxytrifluoromethylation reaction of 3-benzylidene-2-arylisooindolin-1-one (the special structure of enamide) with sodium trifluoromethanesulfinate catalyzed by CuBr in the presence of $\text{K}_2\text{S}_2\text{O}_8$ (Scheme 1).

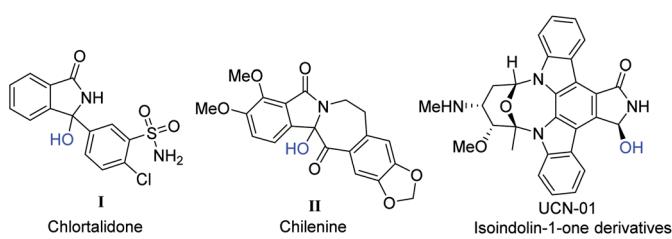


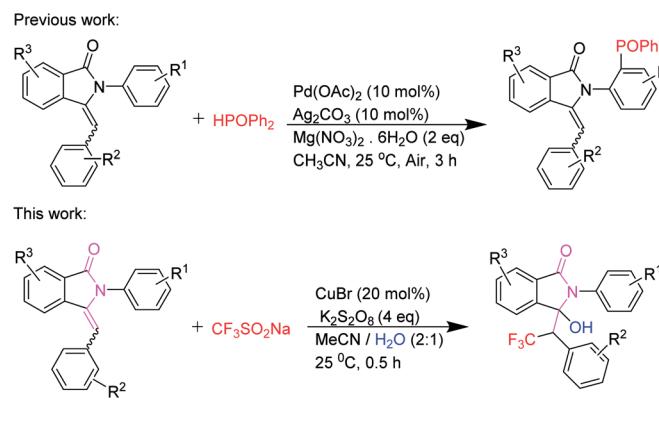
Fig. 1 Bioactive and drug value compounds containing 3-hydroxyisoindolin-1-one motifs.

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Results and discussion

When the model reaction of 3-benzylidene-2-phenylisoindolin-1-one (**1a**) with sodium trifluoromethanesulfinate (**2**) was performed in $\text{CH}_3\text{CN}/\text{H}_2\text{O}$ in the presence of oxidants such as TBHP, DTBP, $\text{Mn}(\text{OAc})_3$ and $\text{PhI}(\text{OAc})_2$, no desired products were obtained (Table 1, entries 1, 2, 3 and 4). After the addition of $\text{K}_2\text{S}_2\text{O}_8$ (4 eq.), the reaction proceeded smoothly to afford the desired product, 3-hydroxy-2-phenyl-3-(2,2,2-trifluoro-1-phenylethyl)isoindolin-1-one (**3a**) in 48% yield (Table 1, entry 5). Further investigation of copper catalysts, the yield of **3a** was



improved to 72% when we used CuBr as the catalyst (Table 1, entries 9). On the other hand, when we use FeCl_3 in place of CuBr, the reaction afforded the desired product in a lower yield (Table 1, entry 12). By screening polar mixed solvents such as DMSO/H₂O, DMF/H₂O, THF/H₂O, acetone/H₂O, and a representative nonpolar solvent, toluene (Table 1, entries 15–19), we found that CH₃CN/H₂O (2 : 1) works best for the reaction. Apart from the above-mentioned factors, the effects of catalyst loading, reaction temperature and time were also investigated, and the optimal reaction conditions were determined to be room temperature reaction for 0.5 h in air atmosphere, with the addition of 20 mol% CuBr as catalyst, K₂S₂O₈ as single electron oxidation reagent and CH₃CN/H₂O as solvent (Table 1, entries 20–27).

With the promising results obtained in the model reaction, we subsequently examined the substrate scope of 3-benzylidene-2-arylisodolin-1-one under the optimized reaction conditions (20 mol% CuBr as catalyst, and K₂S₂O₈ as

Table 1 Optimization of the reaction conditions^a

Entry	Catalyst (%)	Oxidant	Temperature	Solvent (66.7%)	Yield ^b
1		TBHP	25 °C	CH ₃ CN	N.D
2		DTBP	25 °C	CH ₃ CN	N.D
3		Mn(OAc) ₃	25 °C	CH ₃ CN	N.D
4		PhI(OAc) ₂	25 °C	CH ₃ CN	N.D
5		K ₂ S ₂ O ₈	25 °C	CH ₃ CN	48%
6	CuO (20)	K ₂ S ₂ O ₈	25 °C	CH ₃ CN	30%
7	Cu(OAc) ₂ (20)	K ₂ S ₂ O ₈	25 °C	CH ₃ CN	55%
8	CuCl (20)	K ₂ S ₂ O ₈	25 °C	CH ₃ CN	60%
9	CuBr (20)	K ₂ S ₂ O ₈	25 °C	CH ₃ CN	72%
10	CuI (20)	K ₂ S ₂ O ₈	25 °C	CH ₃ CN	28%
11	CuBr ₂ (20)	K ₂ S ₂ O ₈	25 °C	CH ₃ CN	50%
12	FeCl ₃ (20)	K ₂ S ₂ O ₈	25 °C	CH ₃ CN	45%
13	Ag ₂ CO ₃ (20)	K ₂ S ₂ O ₈	25 °C	CH ₃ CN	48%
14	CuBr (20)	K ₂ S ₂ O ₈	25 °C	CH ₃ CN	48%
15	CuBr (20)	K ₂ S ₂ O ₈	25 °C	DMSO	6%
16	CuBr (20)	K ₂ S ₂ O ₈	25 °C	DMF	10%
17	CuBr (20)	K ₂ S ₂ O ₈	25 °C	THF	37%
18	CuBr (20)	K ₂ S ₂ O ₈	25 °C	Acetone	55%
19	CuBr (20)	K ₂ S ₂ O ₈	25 °C	Toluene	0%
20	CuBr (20)	K ₂ S ₂ O ₈	50 °C	CH ₃ CN	65%
21	CuBr (20)	K ₂ S ₂ O ₈	80 °C	CH ₃ CN	60%
22	CuBr (20)	K ₂ S ₂ O ₈	10 °C	CH ₃ CN	66%
23 ^c	CuBr (20)	K ₂ S ₂ O ₈	25 °C	CH ₃ CN	35%
23 ^d	CuBr (20)	K ₂ S ₂ O ₈	25 °C	CH ₃ CN	28%
23 ^e	CuBr (20)	K ₂ S ₂ O ₈	25 °C	CH ₃ CN	70%
24 ^f	CuBr (20)	K ₂ S ₂ O ₈	25 °C	CH ₃ CN	46%
25 ^g	CuBr (20)	K ₂ S ₂ O ₈	25 °C	CH ₃ CN	70%
26 ^h	CuBr (20)	K ₂ S ₂ O ₈	25 °C	CH ₃ CN	70%
27	CuBr (10)	K ₂ S ₂ O ₈	25 °C	CH ₃ CN	60%

^a Reaction conditions: **1a** (1 mmol), **2**, (3 mmol), CuBr, (0.2 mmol), K₂S₂O₈ (4 mmol), solvent (10 ml), at 25 °C in air atmosphere, 30 min. ^b Yields are given for isolated products. ^c CH₃CN/H₂O = 5/1. ^d CH₃CN/H₂O = 1/1. ^e K₂S₂O₈ (5 mmol) was added. ^f K₂S₂O₈ (3 mmol) was added. ^g 1 h. ^h In argon atmosphere.



oxidant in $\text{CH}_3\text{CN}/\text{H}_2\text{O}$ (2 : 1) at 25 °C, for 0.5 h in air atmosphere).

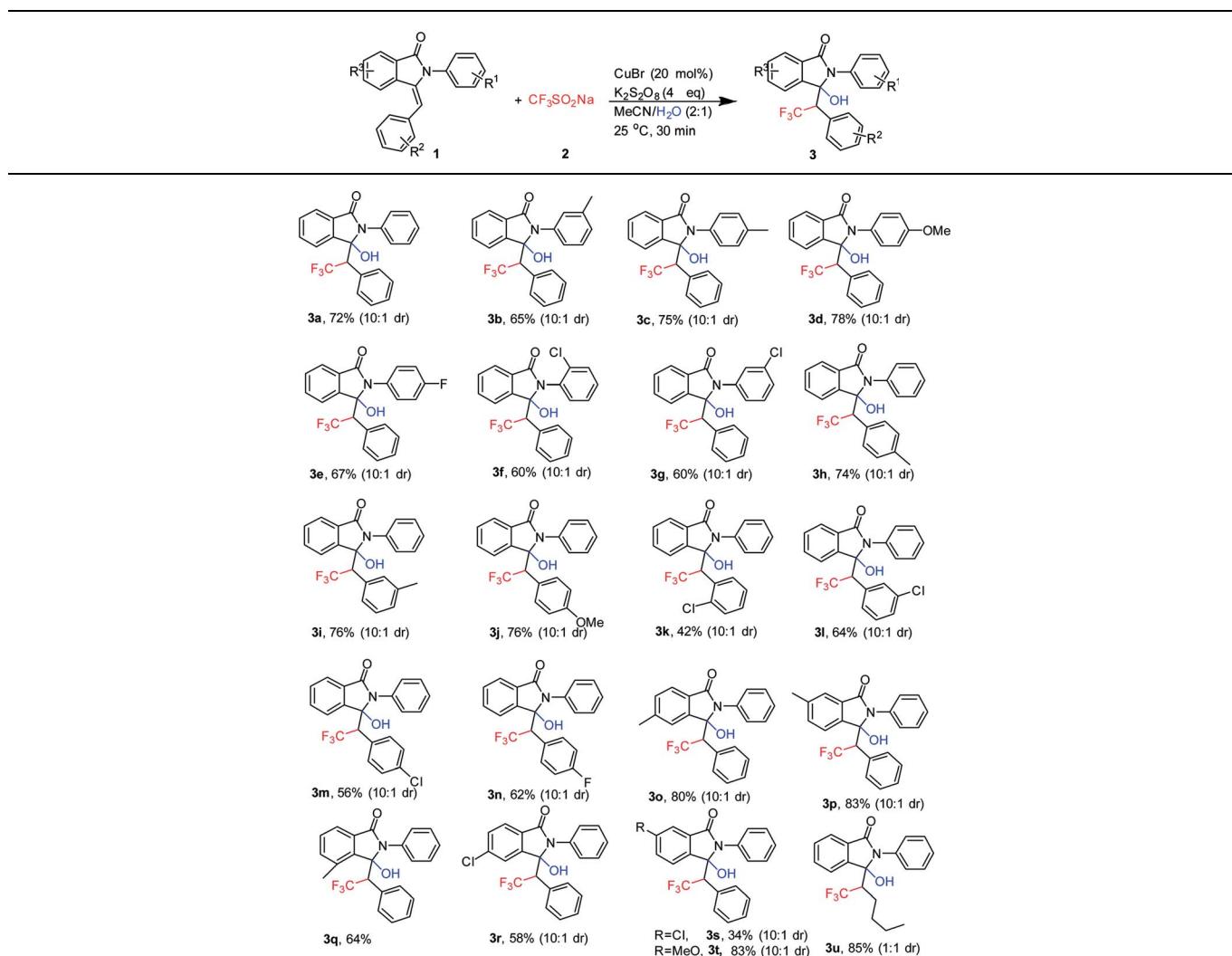
As shown in Table 2, electron-donating substituents such as methyl and methoxy groups on the aryl ring of substituted 3-benzylidene-2-arylisindolin-1-one (**1**) facilitated the reaction to afford the hydroxytrifluoromethylation products (**3**) in moderate to good yields (Table 2, 65–83%, **3a**–**3d**, **3h**–**3j**, **3o**–**3p**, and **3t**). On the contrary, electron-withdrawing groups such as F and Cl were unfavorable for the reaction and led to lower yields (Table 2, 34–67%, **3e**–**3g**, **3k**–**3n** and **3r**–**3s**). We also found that when the substrate was 3-pentylidene-2-phenylisoindolin-1-one, the target product (**3u**) was in 85% yield but diastereomeric ratio is 1 : 1.

In order to understand the reaction mechanism, following control experiments were carried out. We repeated the reaction in the presence of radical quencher 2,2,6,6-tetramethylpiperidin-1-oxyl (TEMPO) and none of **3a** was

obtained (Scheme 2a). The result suggested that free radical were probably generated during the reaction. Furthermore, **3a** was also not detected when the reaction was performed with the addition of butylated hydroxytoluene (BHT, 3.0 equiv.) under the standard conditions (Scheme 2b). Trifluoromethylation products was obtained when 1,1-diyldibenzene and Langlois reagent were carried out in standard condition (Scheme 2c). On the other hand, neither aryl amine nor benzylamine substrate produced the *ortho*-position C–H activated products (Scheme 2d).¹⁸ These results indicated that the reaction is only suitable for enamine substrates which have enough electron cloud density.

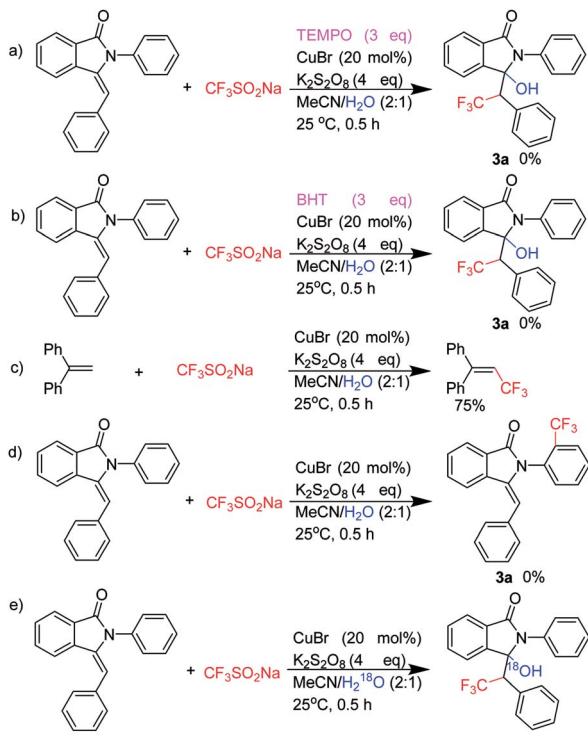
As the hydroxytrifluoromethylation always took place under argon atmosphere in the above experiments, we wondered whether the reaction would proceed if isotopically labelled molecular H_2^{18}O was used. Hence, we did further reactions (Scheme 2e). Surprisingly, the corresponding ^{18}O -containing

Table 2 Scope studies of 3-hydroxy-2-phenyl-3-(2,2,2-trifluoro-1-phenylethyl)isoindolin-1-one^a



^a Reaction conditions: **1** (1 mmol), **2** (3 mmol), CuBr, (0.2 mmol), $\text{K}_2\text{S}_2\text{O}_8$ (4 mmol), $\text{CH}_3\text{CN}/\text{H}_2\text{O}$ 2 : 1 (10 ml), at 25 °C in air atmosphere, 30 min. Yield of isolated products are given.



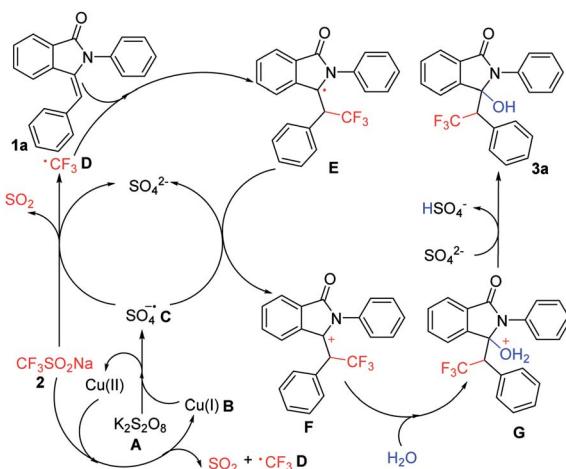


Scheme 2 Control experiments.

product **4a** was obtained in 70%. These results further indicated that the oxygen source of this reaction is derived from H_2O rather than oxygen gas.

On the basis of the mechanistic studies and experimental results, a plausible mechanism is proposed in Scheme 3.

Initially, the $\text{K}_2\text{S}_2\text{O}_8$ (**A**) was excited by $\text{Cu}(\text{i})$ (**B**) to generate the intermediate SO_4^{2-} radical anions (**C**), which then reacted with $\text{CF}_3\text{SO}_2\text{Na}$ (**2**) to form trifluoromethyl free radical (**D**). **D** underwent addition with substrate (**1a**) to form key radical intermediate **E**. Thereafter, the radical intermediate **E** was oxidized by SO_4^{2-} radical anions (**C**) which can regenerate SO_4^{2-} to produce the cation intermediate **F**. Due to the presence of H_2O , the cation intermediate **F** underwent nucleophilic



Scheme 3 Proposed reaction mechanism.

addition to generate the corresponding intermediate **G**. The cation intermediate **G** underwent removing protons to generate the corresponding product **3a** and HSO_4^- . Finally, $\text{Cu}(\text{II})$ was reduced to $\text{Cu}(\text{i})$ by $\text{CF}_3\text{SO}_2\text{Na}$ (**2**) to complete the catalytic cycle.

Conclusions

In summary, we have developed a novel catalytic system for direct hydroxytrifluoromethylation of substituted 3-benzylidene-2-aryloindolin-1-ones *via* a radical pathway. The reaction has a high regioselectivity as the CF_3 free radical is prone to attacking a double bond rather than the aryl. The method has a broad scope and offers a good yield. The corresponding products are potentially useful in drug discovery.

Conflicts of interest

There are no conflicts to declare.

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