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Iridium(III)-Catalyzed Selective and Mild C-H Amidation of Cyclic *N*-Sulfonyl Ketimines with Organic Azides

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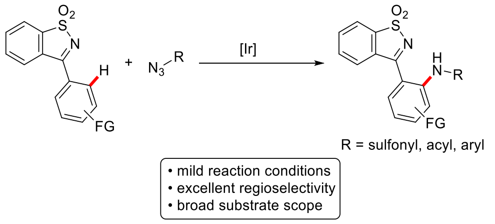
Abstract. A general protocol for iridium catalyzed direct C-H amidation of cyclic *N*-sulfonyl ketimines using sulfonyl, acyl and aryl azides as nitrogen source is reported herein. The reaction takes place at room temperature with acyl and aryl azides, while an elevated temperature needed with sulfonyl azides to furnish aminated sultams in excellent yields with complete chemo and regioselectivity, thus providing a robust and environmentally benign process to the synthesis of aminosultams.

Keywords: amidation; sultams; azides; C-H activation; Iridium

In modern synthetic chemistry transition metal catalyzed amination represents one of the significant and elemental C-N bond formation reactions.[1] By facilitating introduction of versatile synthetic amine fragments onto a great diversity of arenes and heteroarens, these reactions find immense applications in medicinal chemistry, agrochemistry, natural product synthesis and material chemistry.[2] Thus, it is not surprising that, in the past decades tremendous efforts have been paid by synthetic chemists to develop reliable and advanced synthetic routes to construct C-N bond.[3] Among them, cross-coupling reactions developed by Buchwald-Hartwig and Ullmann-Goldberg using palladium and copper respectively, have been studied extensively for efficient construction of C-N bonds.[4] Despite the efficiency and versatility, to react with amine source, these coupling reactions require pre-functionalized species such as aryl halides or pseudohalides. More recently, Chang's[5] and other groups[6] have developed amination methods using organic azides as amine source and widely applied them for an array of organic compounds, as azides are environmentally friendly reagents releasing non-toxic nitrogen gas as the only byproduct.

Being a ubiquitous motif in many pharmaceuticals and biologically active molecules, cyclic *N*-sulfonyl ketimines, are considered as an important class of sulfur and nitrogen containing heterocyclic molecules.[7] *N*-sulfonyl ketimines are valuable synthons to access a myriad of prevalent nitrogen containing molecules.[8] Although cyclic *N*-sulfonyl ketimines are commonly used building blocks[9] and chiral auxiliaries[10] in many organic transformations, their use as a directing group for C-H functionalization is still in a primitive stage. In contrast, acyclic *N*-sulfonyl ketimines are well explored as directing groups.[11] The limited exploration of these sultams as directing groups may be attributed to the lower coordination ability of nitrogen atom, due to the electron withdrawing effect of conjugated sulfonyl moiety.

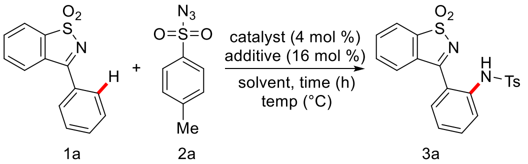
Though Li et al.,[12] reported the synthesis of indenamines from *N*-tosyl aldimines and internal alkynes by C-H activation using Ru catalyst, the first C-H functionalization of cyclic *N*-sulfonyl ketimines to form spirocyclic sultams with 1,3 dienes and alkynes was reported by Nishimura et al.,[13] and Dong et al.,[14] using Ir and Rh catalyst respectively. Later Wei group[15] demonstrated olefination, arylation, allylation and vinylation of sultam derivatives utilizing Rh catalysts and cyclic *N*-sulfonyl ketimines as effective directing groups. In early 2016, Cramer[16] reported the synthesis of chiral spirocyclic sultams using Rh complexes. These approaches are generally limited to C-C bond formation and only one example features C-N bond construction.[17] Our continuous research interest in C-N bond forming reactions,[18] prompted us to investigate cyclic *N*-sulfonyl ketimine assisted amidation of C-H bonds. Described herein is a Cp*Ir(III)-catalyzed mild and effective diect C-H amidation of sultam derivatives using organic azides, which furnishes monoamidation products in a highly chemo and regioselective manner(Scheme 1).



Scheme 1. Ir(III)-catalyzed directed C-H amidation

In preliminary experiments, readily accessible sultam derivative **1a**, was selected as the model substrate. Initial reaction of **1a** with *p*-toluenesulfonyl azide **2a**, in the presence of $[Cp^*IrCl_2]_2$ and $AgNTf_2$ in DCE at room temperature resulted in no conversion. To our surprise, when the reaction was heated at 60 ^{o}C, complete conversion was observed within 4 hours and product **3aa** was isolated in 80% yield, without the aid of any external oxidants (Table 1, entry 2). Other comparative catalysts Rh^{III}, Co^{III} were ineffective for this conversion, while Ru^{II} showed some activity, but in inferior effectiveness. Test reactions justified vital roles of the Iridium catalyst and the additive. The effects of reaction temperature and additives were also examined (see Supporting Information for details). It was found that decreasing the temperature resulted in lower conversion, while elevated temperatures furnished monoamidated sultam **3aa** as sole product in quantitative yield in shorter time (Table 1, entry 8). $AgNTf_2$ provided optimal results among a representative set of silver additives. With this exciting results in hand, a systematic screening of solvents was performed. The reaction displayed a prominent solvent dependence characteristic. While THF, 1,4-dioxane, DCM, t-AmOH, MeOH and acetone afforded secondary results compared to DCE. The solvents possessing coordinating ability, such as DMF and MeCN, completely retarded the reaction. To our delight, when reaction was performed in CF_3CH_2OH, product **3aa** was isolated in excellent yield (Table 1, entry 15). Related imidation reaction[19] was also tried in vain (See Supporting Information).

Table 1. Optimization of Reaction Conditions for Iridium Catalyzed Amidation of Cyclic *N*-sulfonyl Ketimines.[a]

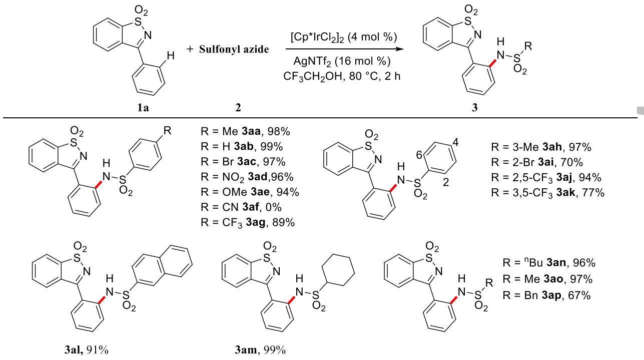


entry	catalyst	additive	temp (°C)	solvent	time (h)	yield (%)[b]
1	$[Cp^*IrCl_2]_2$	$AgNTf_2$	25	DCE	24	trace
2	$[Cp^*IrCl_2]_2$	$AgNTf_2$	60	DCE	4	80
3	$[Cp^*RhCl_2]_2$	$AgNTf_2$	60	DCE	24	NR
4	Ru(p-cym)$_2$Cl$_2$	$AgNTf_2$	60	DCE	24	35
5	-----------	$AgNTf_2$	60	DCE	24	NR
6	$[Cp^*IrCl_2]_2$	-------	60	DCE	24	NR
7	$[Cp^*IrCl_2]_2$	$AgNTf_2$	40	DCE	24	30
8	$[Cp^*IrCl_2]_2$	$AgNTf_2$	80	DCE	2	91
9	$[Cp^*IrCl_2]_2$	$AgSbF_6$	80	DCE	2	69
10	$[Cp^*IrCl_2]_2$	$AgBF_4$	80	DCE	2	60
11	$[Cp^*IrCl_2]_2$	$AgPF_6$	80	DCE	2	75
12	$[Cp^*IrCl_2]_2$	$AgNTf_2$	80	THF	2	62
13	$[Cp^*IrCl_2]_2$	$AgNTf_2$	80	Dioxane	2	58
14	$[Cp^*IrCl_2]_2$	$AgNTf_2$	80	DCM	2	80
15	$[Cp^*IrCl_2]_2$	$AgNTf_2$	80	CF_3CH_2OH	2	98

[a]Reaction conditions: To a mixture of catalyst (4 mol %), additive (16 mol%) were added **1a** (0.3 mmol), **2a** (0.36 mmol), solvent (0.5mL) and allowed to stir for particular time at indicated temperature under air atmosphere. [b]isolated yield.

With the established conditions in hand, we next tested the scope of the reaction with respect to various sulfonyl azides using **1a** as the model substrate. Sulfonyl azides bearing both electron donating and electron withdrawing groups at *para-* position gave mono sulfamidated products in excellent yields except **2f** with p-cyano group.

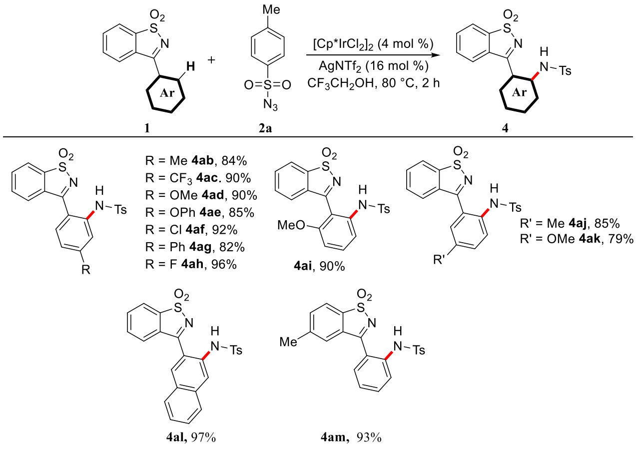
Table 2. Substrate Scope for Sulfonyl Azides with **1a** under Optimized Condition[a]



Under the optimized condition, 3-methylbenzenesulfonyl azide furnished product **3ah** in 97% yield. Arylsulfonyl azide having bromo group at *ortho* position could readily participate in this sulfamidation reaction, providing **3ai** in moderate yield. To our surprise, reaction with sulfonyl azide bearing trifluoromethyl groups at *ortho*- and *meta*- position, yielded **3aj** in 94% yield, while the substrate with the same substituent at 3- and 5- position provided **3ak** in only 77% yield. In addition, amidation reaction of **1a** with 2-naphthalenesulfonyl azide occurred efficiently to furnish corresponding aminosultam **3al** in high yield. Moreover, alkanesulfonyl azides were also compatible under the standard amidation condition.

We then set out to expand the scope of this transformation to substituted cyclic *N*-sulfonyl ketimines with respect to sulfonyl azide **2a**. We were pleased to observe that the amidation occurred smoothly irrespective of the position and electronic nature of substituents, thus giving rise to the corresponding C-6 sulfamidated sultams in high efficiency and regioselectivity. Indeed, substrates bearing methyl (**1b** and **1j**), alkoxy (**1d**, **1e**, 1**i** and 1**k**), trifluoromethyl (**1c**), phenyl (**1g**), chloro (**1f**) and fluoro (1**h**) groups at the C2-, 3-, 4-and 5- position of cyclic *N*-sulfonyl ketimines were all well suited for the present sulfamidation. Sultam bearing naphthyl group **1l**, also reacted effectively to produce **4al** in excellent yield. Furthermore ketimine derived from substituted saccharin **1m** was also suitable substrate for C-H amidation reaction, furnishing **4am** in 93% yield.

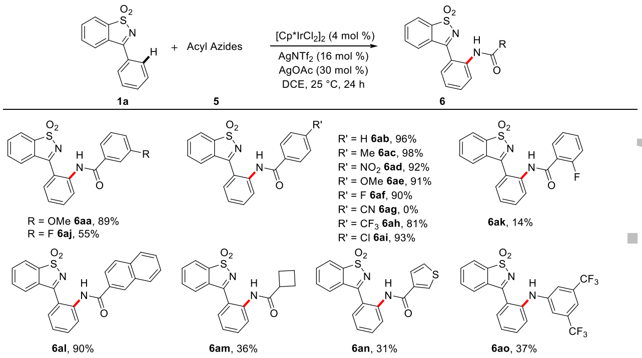
Table 3. Substrate Scope of Ir-Catalyzed C-H Amidation of Cyclic *N*-sulfonyl Ketimines[a]



Next we turned our attention to exploring the compatibility of acyl azides for our reaction. Under the optimized amidation conditions for sulfonyl azides, acyl azide **5a** was unreactive. Pleasingly, the reactivity of acyl azide was dramatically enhanced when an oxidant AgOAc was added to the reaction performed in DCE at room temperature to give amidated compound **6aa** in 89% yield after 24 h. Only DCE and AgOAc furnished optimal results among a representative set of solvents and oxidants screened (see the Supporting Information). With this optimized iridium catalytic system, we tested scope of acyl azides with cyclic *N*-sulfonyl ketimines.

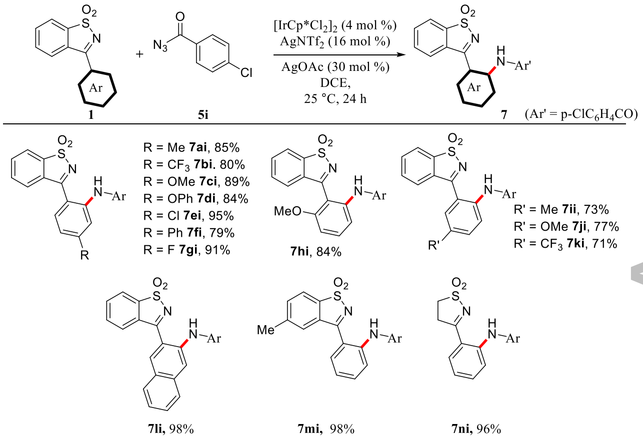
An array of *para*-, *meta*- and *ortho*-substituted azides **5** bearing electron-withdrawing or electron-donating groups were successfully converted into desired amide products **6**. In addition, reaction of **1a** with acyl azide derived from naphthalene occurred efficiently to furnish **6al** in high yield. Moreover, the acyl azides of cyclobutane and 3-thiazole displayed only moderate efficiencies in present catalytic system. Even aryl azide, derived from 3,5-bis(trifluoromethyl)benzene furnished the corresponding aminated product **6ao** albeit in moderate yield.

Table 4. Scope of acyl azides[a]

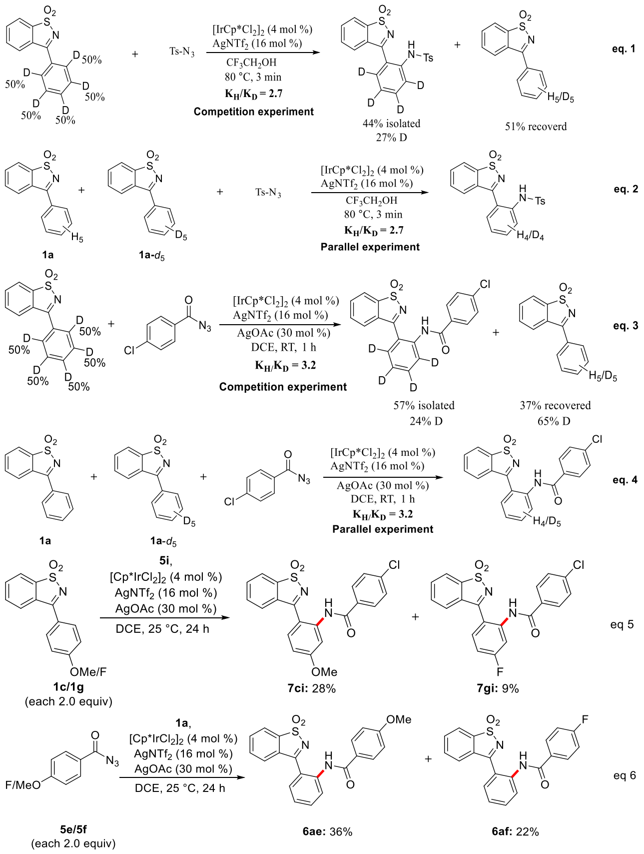


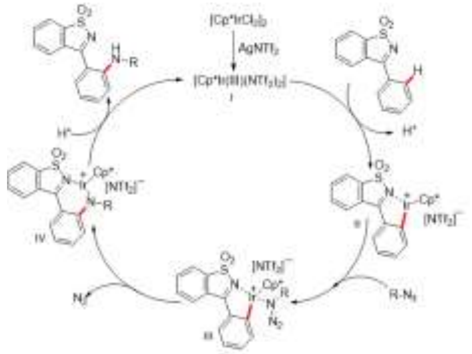
To probe the generality of cyclic *N*-sulfonyl ketimines towards amidation, a set of derivatized sultams **1** were tested. The *para*- and *ortho*-substituted sultams were selectively transformed into corresponding amide products **7ai-hi**. The catalytic system exhibited phenomenal chemo selectivity and hence tolerated a plethora of valuable functional groups such as Cl, F, OMe, OPh, Ph and CF_3. Even under steric control, *meta*- substituted sultams furnished **7ii-ki** in moderate yield with an excellent site selectivity. Under the optimized conditions, sultam with naphthyl group, scaccharin derived ketimine and cyclic ketimine reacted exceptionally well to yield respective aminosultams **7li**, **7mi** and **7ni**.

Table 5. Scope of sultams with acyl azide **5i**[a]



Finally, to acquire some insights into mechanistic pathway of our current amidation reaction, intermolecular competition and parallel experiments between **1a** and its penta-deuterated analog **1a-*d*$_5$** with tosyl azide **2a** (eq. 1 and 2) as well as acyl azide **5i** (eq. 3 and 4) were carried out separately. The kinetic isotope effect values (KIE) of 2.7 and 3.2 were observed, after the reactions were stirred for 3 min and 60 min, respectively. Notable KIE values for C(sp^2)-H amidation suggest that C-H bond cleavage is plausibly involved in the rate determining step.[20] Intermolecular competition experiments with various substituted sultams **1** (eq. 5) and acyl azides **5** (eq. 6), unveiled electron rich sultams and acyl azides to be intrinsically reactive substrates. The relatively faster rate of acyl azide **5e** may be due to more electron richness of **5e** compared to **5f**. Based on these studies and previous results,[5, 21] a plausible pathway for amidation reaction is proposed as shown in scheme 2. Initially, the dimeric iridium species forms a cationic species I with AgNTf$_2$, which in turn initiates the directed C6-H bond activation of cyclic *N*-sulfonyl ketimines via the formation of five membered cyclometallated iridium complex by chelation assistance of iminyl nitrogen atom on sultams. Then the interaction of azide with iridium metal centre of II generates III, which forms IV upon migratory insertion of an imido group into Iridium-Carbon bond with simultaneous expulsion of N$_2$. The complex IV, after protodemetallation furnishes amidated product with the regeneration of active catalyst.





Scheme 2. Proposed Plausible Mechanism for the Iridium-Catalyzed Amidation of Sultams.

In conclusion, we have described an iridium-catalyzed mild and regioselective amidation protocol for cyclic *N*-sulfonyl ketimines with a range of organic azides to furnish the amidated products in moderate to excellent yields, which offers an efficient route to the synthesis of biologically relevant aminosultam compounds.

Experimental Section

To a screw capped vial with stir bar were added cyclic *N*-sulfonyl ketimine (**1a**, 0.12 mmol), sulfonyl azide (**2**, 0.14 mmol), [IrCp*Cl$_2$]$_2$ (3.9 mg, 0.004 mmol, 4 mol %), AgNTf$_2$ (7.6 mg, 0.019 mmol, 16 mol %) and 2, 2, 2-trifluoroethanol (0.5 ml) under atmospheric conditions. The reaction mixture was stirred at 80 °C for 2 h, filtered through a pad of celite and then washed with dichloromethane (10 mL x 3). Organic solvents were removed under reduced pressure and the residue was purified by chromatography on silica gel (*n*-hexane/EtOAc) to give the desired product **3**.

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