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Iridium-catalysed direct sulfamidation of quinazolinones†

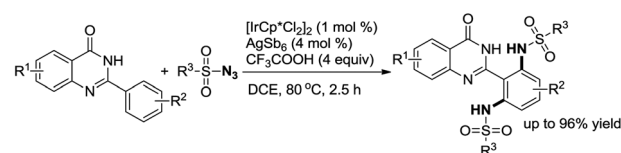
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Ir-catalysed direct sulfamidation of quinazolinones has been achieved. A series of *ortho*-diamided quinazolinones were obtained in up to 96% yields. This transformation could proceed smoothly with a low catalyst loading under mild conditions with nitrogen released as the sole byproduct. This approach potentially provides an environmentally benign sulfamidation process for atom/step economic syntheses of useful pharmaceutical molecules or important building blocks.

Over the past two decades, C–H bond functionalization has attracted considerable attention for its outstanding advantages in atom efficiency and synthesis step efficiency compared to the traditional cross-coupling reactions.¹ Prefunctionalization and multiple tedious purification steps can be avoided while enhancing the step economy of the transformation and decreasing the byproduct formation in these processes.² Therefore, direct functionalization of C–H bond has become an increasingly attractive strategy for the synthesis and derivatization of natural products and pharmaceutically relevant building blocks.³ However, controlling site selectivity is one of the challenges in C–H bond activation. In this regard, the strategy involving regioselective C–H bond activation assisted by directing group shows high potential. So far, various directing groups containing oxygen or nitrogen atoms have been developed successfully. As is well-known, quinazolinones are a significant class of compounds because of their pharmaceutical applications and biological activities. In particular, quinazolinones have been found to be with various bioactivities, such as antibacterial,⁴ antifungal,⁵ antimalarial,⁶ anticancer,⁷ antihypertensive,⁸ antitubercular,⁹ and anticonvulsant.¹⁰ Forward this direction, our group has developed a series of easy methods in modification of quinazolinones *via* metal-catalysed C–H bond functionalization, in which quinazolinones act as directing groups since there are two N atoms in these structures.¹¹ In addition, organic azide, as an internal oxidant and environmentally benign reagent, has been extensively explored in amidation reactions because nontoxic nitrogen gas is released as the only byproduct.¹² Recently, our group has developed an Ir-catalysed amidation of benzaldehydes with

sulfonyl azides as amino sources.¹³ In our continuing effort to develop green methods in C–H bond activation,¹⁴ herein, we disclose an Ir-catalysed direct amidation of 2-arylquinazolinones with sulfonyl azides as amino sources to produce *ortho*-diamided quinazolinones (Scheme 1), in which symmetrical di-amidated products was selectively obtained with high yields by controlling the amount of azide. This procedure could proceed smoothly with a low catalyst loading and tolerate various substituent groups with N₂ as the sole byproduct. The titled products are an important building block in organic synthesis and can be potentially converted into various highly valuable molecules *via* diverse transformations.

Initially, the amidation of 2-phenylquinazolin-4-(3*H*)-one (**1a**) with *para*-toluenesulfonyl azide (**2a**) (3.0 equiv.) was chosen as a model reaction to examine the impact of various parameters on the reaction (Table 1). The results revealed that *N,N'*-(2-(4-oxo-3,4-dihydroquinazolin-2-yl)-1,3-phenylene)bis(4-methyl benzenesulfonamide) (**3a**) was obtained as a main product in 96% yield in DCE at 80 °C when [IrCp*Cl₂]₂ (1 mol%) was used as a catalyst with AgSbF₆ (4 mol%), CF₃COOH (4 equiv.) under air (Table 1, entry 1). We tried the first set of conditions displayed in entry 1 of Table 1 firstly, and found they are the optimum reaction conditions fortunately. When the loading amount of **2a** was reduced to 2.0 or 1.0 equivalent, the reaction was complex and gave the mixture of the mono- (**4a**, Scheme 2a) and di-amidated products (**3a**). Only 75% and 60% yield of the target product **3a** was achieved when the reaction was carried out under O₂ and N₂ protection (Table 1, entries 2–3). Other metal salts, such as Pd(OAc)₂ and [RhCp*Cl₂]₂ were screened,

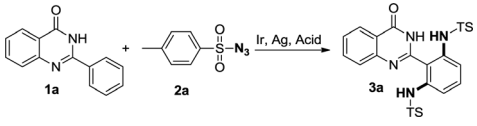


Scheme 1 Ir-catalysed direct amidation of 2-arylquinazolinones.

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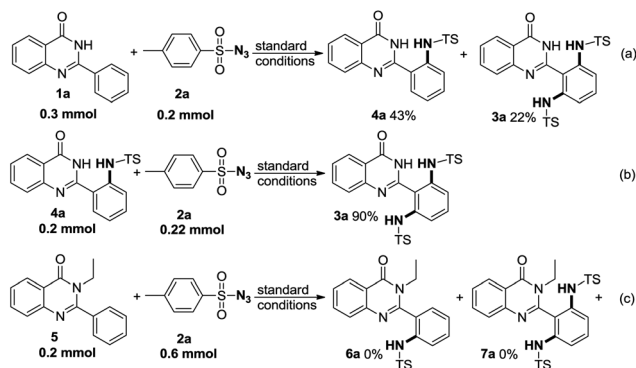
† Electronic supplementary information (ESI) available: General information, experimental procedures, copies of ¹H, ¹³C and ¹⁹F NMR spectra for products. See DOI: 10.1039/c8ra00524a



Table 1 Optimization of the reaction conditions^a


Entry	Catalyst	Silver	Acid	Solvent	Temp (°C)	t (h)	Yield ^b (%)
1	[IrCp*Cl ₂] ₂	AgSbF ₆	TFA	DCE	80	2.5	96
2 ^c	[IrCp*Cl ₂] ₂	AgSbF ₆	TFA	DCE	80	2.5	75
3 ^d	[IrCp*Cl ₂] ₂	AgSbF ₆	TFA	DCE	80	2.5	60
4 ^e	[IrCp*Cl ₂] ₂	AgSbF ₆	TFA	DCE	80	2.5	77
5	—	AgSbF ₆	TFA	DCE	80	2.5	nd
6	Pd(OAc) ₂	AgSbF ₆	TFA	DCE	80	2.5	nd
7	[RhCp*Cl ₂] ₂	AgSbF ₆	TFA	DCE	80	2.5	nd
8	[IrCp*Cl ₂] ₂	—	TFA	DCE	80	2.5	nd
9	[IrCp*Cl ₂] ₂	AgOAc	TFA	DCE	80	2.5	40
10	[IrCp*Cl ₂] ₂	Ag ₂ CO ₃	TFA	DCE	80	2.5	15
11	[IrCp*Cl ₂] ₂	AgNTf ₂	TFA	DCE	80	2.5	52
12	[IrCp*Cl ₂] ₂	AgSbF ₆	—	DCE	80	2.5	nd
13	[IrCp*Cl ₂] ₂	AgSbF ₆	AcOH	DCE	80	2.5	nd
14	[IrCp*Cl ₂] ₂	AgSbF ₆	PivOH	DCE	80	2.5	nd
15	[IrCp*Cl ₂] ₂	AgSbF ₆	TFA	DMF	80	2.5	nd
16	[IrCp*Cl ₂] ₂	AgSbF ₆	TFA	THF	80	2.5	nd
17	[IrCp*Cl ₂] ₂	AgSbF ₆	TFA	NMP	80	2.5	nd
18	[IrCp*Cl ₂] ₂	AgSbF ₆	TFA	DCE	90	2.5	86
19	[IrCp*Cl ₂] ₂	AgSbF ₆	TFA	DCE	70	2.5	70
20	[IrCp*Cl ₂] ₂	AgSbF ₆	TFA	DCE	80	3.0	88
21	[IrCp*Cl ₂] ₂	AgSbF ₆	TFA	DCE	80	2.0	81

^a Reaction conditions: **1a** (0.20 mmol), **2a** (0.60 mmol), Ir (1 mol%), Ag (4 mol%), acid (4.0 equiv.), solvent (2 mL). ^b Isolated yields. ^c O₂. ^d N₂. ^e Ir (0.5 mol%), Ag (2 mol%). nd = not detected.



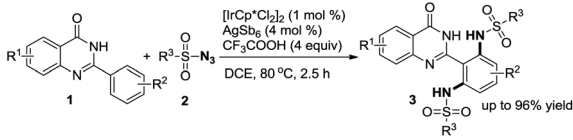
Scheme 2 Exploration of the reaction mechanism.

showing that the reaction did not proceed without Ir catalyst and the yield of **3a** was decreased when the loading of catalyst was reduced to 0.5 mol% (Table 1, entry 1 vs. entries 4–7). Moreover, AgSbF₆ was shown to favor this reaction than other Ag salts (Table 1, entry 1 vs. entries 8–11). TFA was chosen to be better acid (Table 1, entry 1 vs. entries 12–14). On the other hand, DCE was demonstrated to be the best solvent (Table 1, entry 1 vs. entries 15–17). The yield of **3a** decreased when the reaction temperature and reaction time were changed (Table 1, entries 18–21). Based on the results, under the optimum reaction conditions, the target product **3a** was obtained in 96% yield

in DCE at 80 °C when [IrCp*Cl₂]₂ (1 mol%) was used as a catalyst with AgSbF₆ (4 mol%), CF₃COOH (4 equiv.) under air (Table 1, entry 1).

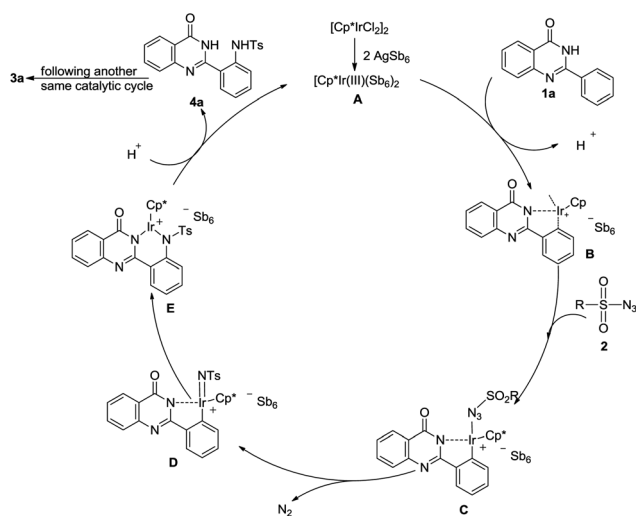
With the optimized reaction conditions in hand, the scope of the substrates was examined (Table 2). First, toluenesulfonyl azide (**2a**) reacted smoothly with 2-phenylquinazolin-4-(3*H*)-one (**1a**) and its derivatives (**1b–1i**) to give **3a–3i** in good to excellent yields (60–96%). Halogen groups, such as F, Cl, and methoxyl at the 5- or 6-position of quinazolinones provided the corresponding products **3b–3d** in 86%, 78% and 92% yields, respectively, which indicated that the electron density on the moiety of the quinazolinones did not significantly influence the efficiency of the reaction. F group at the *meta*- and *para*-position of 2-phenyl in 2-aryl-quinazolin-4-(3*H*)-one provided the corresponding products **3e–3f** in 65% and 94% yields. Other groups, such as Cl, Br, trifluoromethyl, methyl, *t*-butyl, and methoxyl could be well tolerated and gave the corresponding products in satisfactory yields (**3g–3l**) (60–96%). However, no product was obtained with functional groups such as –CN, –NO₂, and –OH in 2-aryl-quinazolin-4-(3*H*)-one. 2-Phenylquinazolin-4-(3*H*)-one (**1a**) also reacted smoothly with substituted azides (**2b–2d**) to give the desired products **3m–3o** in 80–89% yields. It can be seen that the electron density on the aromatic moiety of the azides did not significantly influence the efficiency of the reaction, and alkyl azide could also be tolerated in this procedure.



Table 2 Scope of substrates ^{a,b}


Entry	1	2	3	Yield ^b (%)
1	1a, R ¹ = H; R ² = H	2a, R ³ = 4-CH ₃ C ₆ H ₄	3a	96
2	1b, R ¹ = 5-F; R ² = H	2a, R ³ = 4-CH ₃ C ₆ H ₄	3b	86
3	1c, R ¹ = 6-Cl; R ² = H	2a, R ³ = 4-CH ₃ C ₆ H ₄	3c	78
4	1d, R ¹ = 6-OCH ₃ ; R ² = H	2a, R ³ = 4-CH ₃ C ₆ H ₄	3d	92
5	1e, R ¹ = H; R ² = 3-F	2a, R ³ = 4-CH ₃ C ₆ H ₄	3e	65
6	1f, R ¹ = H; R ² = 4-F	2a, R ³ = 4-CH ₃ C ₆ H ₄	3f	94
7	1g, R ¹ = H; R ² = 4-Cl	2a, R ³ = 4-CH ₃ C ₆ H ₄	3g	60
8	1h, R ¹ = H; R ² = 4-Br	2a, R ³ = 4-CH ₃ C ₆ H ₄	3h	87
9	1i, R ¹ = H; R ² = 4-CF ₃	2a, R ³ = 4-CH ₃ C ₆ H ₄	3i	96
10	1j, R ¹ = H; R ² = 4-CH ₃	2a, R ³ = 4-CH ₃ C ₆ H ₄	3j	70
11	1k, R ¹ = H; R ² = 4- <i>t</i> -Bu	2a, R ³ = 4-CH ₃ C ₆ H ₄	3k	91
12	1l, R ¹ = H; R ² = 4-OCH ₃	2a, R ³ = 4-CH ₃ C ₆ H ₄	3l	90
13	1a, R ¹ = H; R ² = H	2b, R ³ = Ph	3m	83
14	1a, R ¹ = H; R ² = H	2c, R ³ = 4-CH ₃ OC ₆ H ₄	3n	89
15	1a, R ¹ = H; R ² = H	2d, R ³ = CH ₃	3o	80

^a Reaction conditions: **1** (0.20 mmol), **2** (0.6 mmol), Ir (1 mol%), Ag (4 mol%), TFA (4.0 equiv.), solvent (2 mL). ^b Isolated yields.



Scheme 3 The proposed reaction mechanism.

To clarify the reaction mechanism, some control experiments were carried out (Scheme 2). First, 4-methyl-*N*-(2-(4-oxo-3,4-dihydroquinazolin-2-yl)phenyl)benzenesulfonamide (**4a**) and **3a** were obtained in 43% and 22% yields when **1a** (0.3 mmol) was treated with **2a** (0.2 mmol) under the optimized conditions (Scheme 2a). Additionally, **4a** (0.2 mmol) could react with **2a** (0.22 mmol) to give the expected product **3a** in 90% yield (Scheme 2b). To confirm which N atom of quinazolinones (**1a**) was coordinating with Ir(III), NH-protected quinazolinone (**5**) was introduced to the reaction. No target product (**6a** or **7a**) was obtained (Scheme 2c). These results suggested that NH of quinazolinones (**1a**) played as a directing group in the catalytic

cycle, and compound **4a** was the key intermediate in this reaction.

Based on the results obtained and the literatures,¹⁵ a plausible reaction pathway for this Ir(III)-catalysed amidation reaction was proposed and shown in Scheme 3. Initially, the dimeric precursor [IrCp*Cl₂]₂ was converted into a cationic species **A** by the aid of silver salt. The five-membered iridacycle **B** with one vacant accessible site was formed by the coordination of the iridium atom with the nitrogen atom, and subsequently an electrophilic attack at the *ortho*-position carbon atom. Then, intermediate **C** was formed through interaction of azide with the cationic metal center. It was proposed that intermediate **D** from complex **C** occurred in an oxidative manner to release the N₂ molecule. A new C–N bond was formed by insertion of the N–Ts imines species into a new iridacycle **E**. The compound **E** was protodemetalated to deliver the sulfamidated product **4a**. Finally, the desired product **3a** was generated *via* another same catalytic cycle from compound **4a**.

Conclusions

In summary, we have demonstrated an Ir-catalysed direct amidation of 2-arylquinazolinones with sulfonyl azides as amino sources to produce a series of *ortho*-diamided quinazolinones. This transformation involves two C–N bonds formation with low catalyst loading under mild conditions and wide functional group tolerance. Moreover, this approach provided a fast-track strategy for atom/step economical syntheses of useful pharmaceutical molecules with N₂ as the sole byproduct. The products obtained are important building blocks in organic synthesis and could be easily converted into various highly valuable



molecules *via* diverse transformations. Further study on the application of this reaction is ongoing in our laboratory.

Conflicts of interest

There are no conflicts to declare.

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