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Expeditious Synthesis of 2-Phenylquinazolin-4-amines via a Fe/Cu Relay-Catalyzed Domino Strategy

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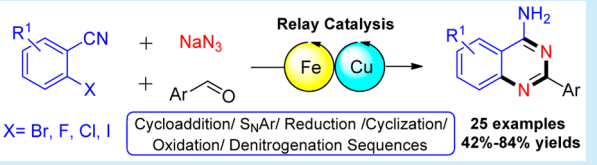
Supporting Information

ABSTRACT: A highly efficient Fe/Cu relay-catalyzed domino protocol has been developed for the synthesis of 2-phenylquinazolin-4-amines from commercially available *ortho*-halogenated benzonitriles, aldehydes, and sodium azide. This elegant domino process involved consecutive iron-mediated [3 + 2] cycloaddition, copper-catalyzed S_NAr, reduction, cyclization, oxidation, and copper-catalyzed denitrogenation sequences. The formed structure is the privileged core in drugs and bioactive molecules.



Quinazoline represents an important and abundant class of nitrogen-containing heterocycles.[1] In particular, as one of the diverse quinazoline derivatives, the 4-aminoquinazoline nucleus is exemplified as a privileged structure that exists in many pharmaceutical molecules and biologically active compounds,[2,3] such as erlotinib (**I**),[2a] geftinib (**II**),[2b] prazosin (**III**),[2c] and human adenosine A_3 receptor antagonist (**IV**)[2d] (Figure 1). In addition, 4-aminoquinazoline derivatives are often used as synthetic intermediates for the direct synthesis of biologically active molecules.[2d,3]

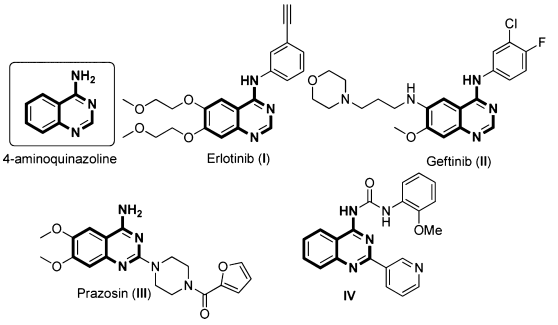
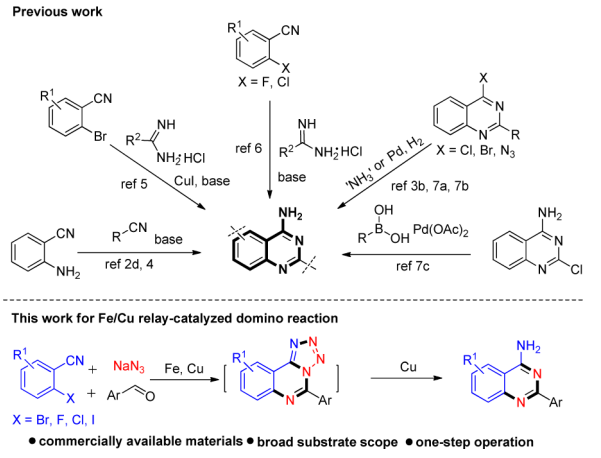


Figure 1. Selected drugs or biologically active compounds with a 4-aminoquinazoline moiety.

Because of their great value, the synthesis of 4-aminoquinazolines has gained much attention. The current synthetic methods of this skeleton are mainly summarized as the following three types: (i) the nucleophilic addition/cyclization reaction of anthranilonitrile with benzonitriles;[2d,4] (ii) the coupling/cyclization reaction of 2-bromobenzonitriles with amidines;[5] and (iii) the S_NAr/cyclization reaction of 2-fluorobenzonitriles with amidines;[6] Alternatively, 2-substituted 4-aminoquinazolines can be prepared by the decoration of the existing quinazoline nucleus[3b,7] (Scheme 1). Although these reactions provide efficient access to 4-aminoquinazolines, their applications are limited by a lack of suitable substrates, poor substitution diversity, and the requirement for harsh reaction conditions. Therefore, the development of effective new methods for the facile construction of 4-aminoquinazolines is highly desirable.

Scheme 1. Synthetic Routes to 2-Substituted 4-Aminoquinazolines



Sodium azide (NaN_3), which was used as a convenient nitrogen source, has been widely applied in organic synthesis.[8−15] The common functions of NaN_3 mainly includes two types: (i) a 1,3-dipole to react with electron-deficient olefins,[8] alkynes,[9] or nitriles[10] and (ii) a coupling partner participating in copper-catalyzed S_NAr reactions.[11] Substantial progress has been made in developing domino reactions based on these two fundamental reactions involving NaN_3.[12−14] As part of our ongoing efforts toward developing novel copper-catalyzed domino reaction related to sodium azide,[15] herein we present a

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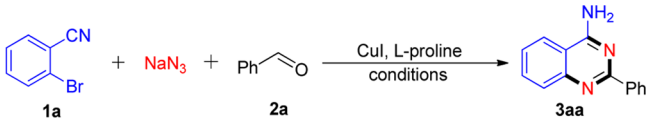
novel Fe/Cu relay-catalyzed domino strategy for the direct synthesis of pharmaceutically significant 2-phenylquinazolin-4-amine derives from commercially available *ortho*-halogenated benzonitriles, aldehydes, and sodium azide (Scheme 1).

To explore the feasibility of this domino protocol, our study commenced with *o*-bromobenzonitrile (**1a**), benzaldehyde (**2a**), and sodium azide as model substrates to optimize the reaction conditions. Initially, various Lewis acids were screened in view of their potential catalytic activity toward initial [3 + 2] cycloaddition of nitriles with NaN_3 according to the existing literature,[10] and $FeCl_3$ showed the highest efficiency in the presence of CuI/L-proline in DMF at 110 °C in a sealed vessel under air (Table 1, entries 1−9). Then several solvents were

Table 1. Optimization of the Reaction Conditions[a]



1a 2a 3aa

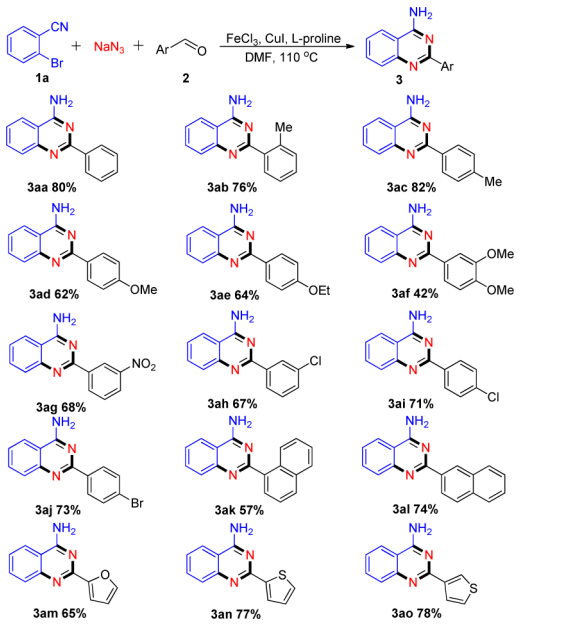
entry	catalyst	solvent	temp (°C)	yield[b] (%)
1	CAN	DMF	110	62
2	$FeCl_3$	DMF	110	71
3	$ZnCl_2$	DMF	110	42
4	$AlCl_3$	DMF	110	45
5	$InBr_3$	DMF	110	68
6	$ZnBr_2$	DMF	110	11
7	$Cu(OAc)_2$	DMF	110	trace
8	$Pd(OAc)_2$	DMF	110	trace
9	$AgNO_3$	DMF	110	trace
10	$FeCl_3$	DMSO	110	6
11	$FeCl_3$	1,4-dioxane	110	trace
12	$FeCl_3$	toluene	110	trace
13	$FeCl_3$	DMF	80	53
14	$FeCl_3$	DMF	100	64
15	$FeCl_3$	DMF	120	70
16		DMF	100	trace
17[c]	$FeCl_3$	DMF	110	trace
18[d]	$FeCl_3$	DMF	110	trace
19[e]	$FeCl_3$	DMF	110	80

[a]Reactions conditions: **1a** (0.5 mmol), **2a** (0.5 mmol), NaN_3 (2.0 mmol), CuI (10%), L-proline (20%), and catalyst (10%) were heated in 3 mL of solvent in a sealed vessel under air for 12 h. [b]Isolated yield. [c]Absence of CuI. [d]Absence of L-proline. [e]30 mol % of $FeCl_3$ was used.

tested (Table 1, entries 10−12), and DMF proved to be the most effective solvent (Table 1, compare entries 2 and 10−12). Increasing or decreasing the temperature of the reaction did not lead to any further improvements in the yield (Table 1, entries 13−15). A control experiment confirmed that $FeCl_3$, CuI, and L-proline are indispensable elements in our catalytic system (Table 1, entries 16−18). Slightly improved efficiency was observed when the loading of $FeCl_3$ was increased from 10 to 30 mol % (Table 1, entry 19). Overall, the optimized reaction conditions were identified as **1a** (0.5 mmol), 1.0 equiv of **2a**, 4.0 equiv of sodium azide, 30 mol % of $FeCl_3$, 10 mol % of CuI, and 20 mol % of L-proline in 3 mL of DMF at 110 °C in a sealed vessel under air.

With the optimal reaction conditions in hand, we next investigated the scope of the domino process. A variety of aromatic aldehydes bearing different substituents were tested, and the results are summarized in Scheme 2. It was found that the transformation was very general; electron-neutral (4-H, 2-Me, 4-Me), electron-donating (4-OMe, 4-OEt, 3,4-$(OMe)_2$), and

Scheme 2. Scope of Aryl Aldehydes[a,b]



[a]Reaction conditions: **1a** (0.5 mmol), **2** (0.5 mmol), NaN_3 (2.0 mmol), $FeCl_3$ (0.15 mmol), CuI (0.05 mmol), and L-proline (0.1 mmol) in DMF (3 mL) at 110 °C in a sealed vessel under air for 12 h. [b]Isolated yields.

electron-deficient (3-NO_2) groups were well tolerated, giving the corresponding products in moderate to good yields (42%−82%, **3aa**−**ag**). To our delight, the optimized conditions were mild enough to allow halo-substituted substrates (67%−73%, **3ah**−**aj**), which provided the possibility for further functionalization. Furthermore, sterically hindered substrates such as 1-naphthaldehyde and 2-naphthaldehyde were also found to be suitable for this transformation (**3ak**−**al**, 57% and 74%). Meanwhile, the optimized conditions could be applied to heteroaryl aldehydes including furan-2-carbaldehyde, thiophene-2-carbaldehyde, and thiophene-3-carbaldehyde (**3am**−**ao**, 65%−78%). Furthermore, the structure of **3aa** was unambiguously determined by X-ray crystallographic analysis (see the Supporting Information).

To further expand the scope of the substrates, a variety of *ortho*-halogenated benzonitriles and aryl aldehydes were then examined. Gratifyingly, electron-neutral (4-Me, 5-Me) groups on the phenyl rings of 2-bromobenzonitriles were compatible and provided the corresponding products in moderate to good yields (Scheme 3, 53−84%, **3ba**−**cg**). Halogen-substituted 2-bromobenzonitriles (5-F, 5-Cl) also afforded the desired products in moderate yields (Scheme 3, 45% and 67%, **3da** and **3ea**). In addition, other *ortho*-halogenated benzonitriles such as 2-fluorobenzonitrile, 2-chlorobenzonitrile, and 2-iodobenzonitrile all also exhibit good reactivity under the optimized conditions (Scheme 3, 74−82%, **3aa**−**aa**).

Notably, this method could also be successfully applied in the convenient synthesis of 1-(2-methoxyphenyl)-3-(2-(pyridin-3-yl)quinazolin-4-yl)urea (**IV**), which is a potent and selective human adenosine A_3 receptor antagonist demonstrated by van Muijlwijk-Koezen.[2d] As shown in Scheme 4, the reaction of *o*-bromobenzonitrile (**1a**) with sodium azide and nicotinaldehyde occurred smoothly under the standard conditions to afford the corresponding products **3ap** in 77% yield. The product **3ap** was

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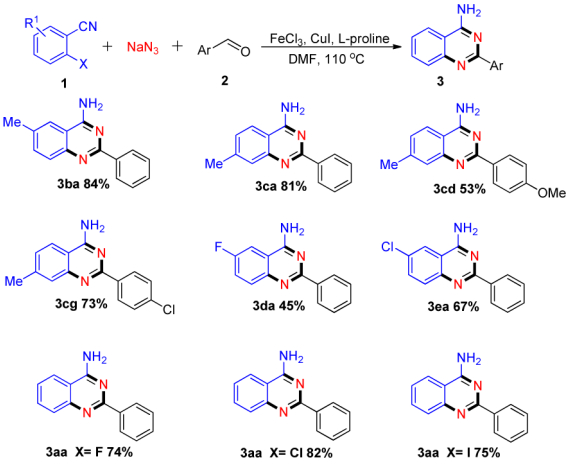
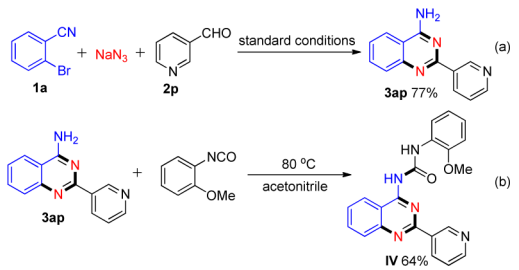
Scheme 3. Scope of *o*-Halogenated Benzonitriles and Aryl Aldehydes[a,b]



[a]Reaction conditions: **1** (0.5 mmol), **2a** (0.5 mmol), NaN_3 (2.0 mmol), $FeCl_3$ (0.15 mmol), CuI (0.05 mmol), and L-proline (0.1 mmol) in DMF (3 mL) at 110 °C in a sealed vessel under air for 12 h. [b]Isolated yields.

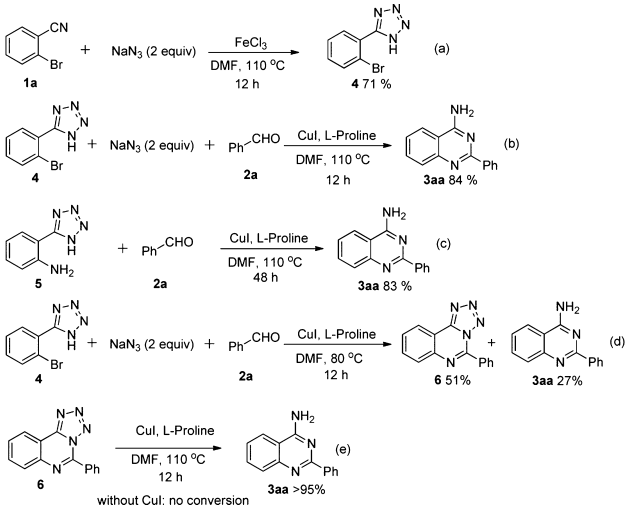
Scheme 4. Synthetic Application



subsequently transformed to pharmaceutically active molecular **IV** according to the reported procedure.[2d]

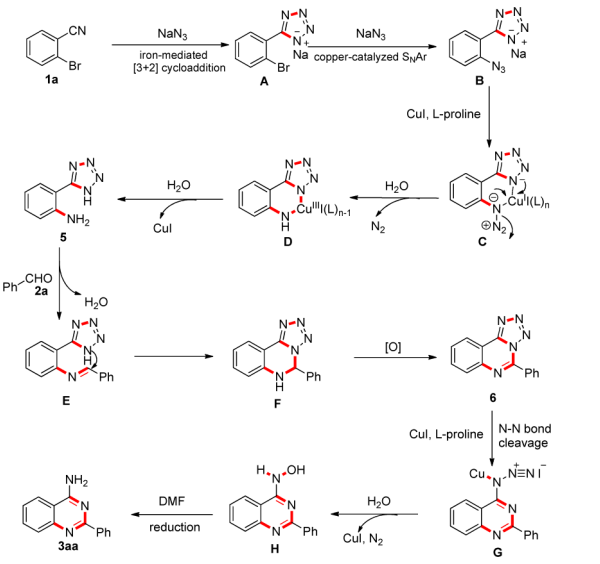
Having established the scope of our new domino reaction, we turned our attention to evaluate the reaction mechanism. We initially investigated the reaction of *o*-bromobenzonitrile (**1a**) with sodium azide (2 equiv) in DMF in the presence of $FeCl_3$ at 110 °C for 12 h, which gave 5-(2-bromophenyl)-1*H*-tetrazole (**4**) in 71% yield (Scheme 5a). When 5-(2-bromophenyl)-1*H*-tetrazole (**4**) was treated with benzaldehyde (**2a**) and NaN_3 (2 equiv) in the presence of CuI in DMF at 110 °C in a sealed vessel under air for 12 h, the target product 2-phenylquinazolin-4-amine (**3aa**) was isolated in 84% yield (Scheme 5b). Furthermore, the reactions of 2-(1*H*-tetrazol-5-yl)aniline (**5**) and benzaldehyde (**2a**) were conducted under standard conditions, and the desired product **3aa** was obtained in 83% yield (Scheme 5c). When 5-(2-bromophenyl)-1*H*-tetrazole (**4**) was treated with benzaldehyde (**2a**) and NaN_3 (2 equiv) in the presence of CuI and L-proline in DMF at 80 °C for 6 h, 5-phenyltetrazolo[1,5-*c*]quinazoline (**6**) and 2-phenylquinazolin-4-amine (**3aa**) were obtained in 51% and 27% yields, respectively (Scheme 5d). Next, when 5-phenyltetrazolo[1,5-*c*]quinazoline (**6**) was heated at 110 °C for 12 h in DMF in the presence of CuI and L-proline, the substrate could be converted to the desired product **3aa** in almost quantitative yield (Scheme 5e). Taken together, these control experiments clearly demonstrated that 5-(2-bromophenyl)-1*H*-tetrazole (**4**), 2-(1*H*-tetrazol-5-yl)aniline (**5**), and 5-phenyltetrazolo[1,5-*c*]quinazoline (**6**) may be key intermediates in this reaction.

Scheme 5. Control Experiments



On the basis of the above observations and literature precedent,[10−19] a possible reaction mechanism of this transformation was represented in Scheme 6. Initially, the sodium 5-

Scheme 6. Possible Mechanism



(2-bromophenyl)tetrazol-1-ide (**A**) was generated though an iron-mediated [3 + 2] cycloaddition of *o*-bromobenzonitrile (**1a**) with NaN_3.[10] Subsequently, intermediate **A** would undergo a copper-catalyzed S_NAr with NaN_3 to afford intermediate **B** in the light of the *ortho*-substituent effect.[10,16] Coordination of azide to copper, followed by an electrocyclization with the concomitant release of N_2, would give the Cu(III) complex **D**,[17] which would undergo a reduction with the aid of trace H_2O in DMF to give intermediate 2-(1*H*-tetrazol-5-yl)aniline (**5**).[11c−e] Next, 2-(1*H*-1,2,3-triazol-5-yl)aniline (**5**) could easily condense with benzaldehyde (**2a**) to give imine intermediate **E**. Then intramolecular nucleophilic attack of nitrogen to imine in **E** followed by oxidative dehydrogenation led to **F**. Eventually, the target product **3aa** was obtained after final cooper-catalyzed denitrogenation process.[18] It is also possible that 5-phenyltetrazolo[1,5-*c*]quinazoline (**6**) and 2-(1*H*-tetrazol-5-yl)aniline (**5**) could be formed via a synergistic oxidation−

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reduction reaction between intermediates **B** and **F**.[19] Further mechanistic studies of the detailed process of reduction and oxidation in this reaction system are in progress.

In conclusion, we have developed a highly efficient Fe/Cu relay-catalyzed domino reaction for the facile synthesis of pharmaceutically significant 2-phenylquinazolin-4-amines from commercially available *ortho*-halogenated benzonitriles, aldehydes, and sodium azide. This elegant domino process involved consecutive iron-mediated [3 + 2] cycloaddition, copper-catalyzed S_NAr, reduction, cyclization, oxidation, and copper-catalyzed denitrogenation sequences. Notably, sodium azide acted as dual nitrogen source in the construction of these fused N-heterocycles. Moreover, the free NH_2 generated from this reaction can be utilized for further manipulation. Application of this self-sequence strategy utilizing NaN_3 as a simple nitrogen donor for the synthesis of other fascinating N-heterocycles are underway in our laboratory.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.5b02020.

Crystallographic data of **3aa** (CIF)

Experimental procedures, product characterizations, and copies of the ^1H and ^{13}C NMR spectra(PDF)

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Notes

The authors declare no competing financial interest.

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