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Lewis acid catalyzed spiro annulation of (Z)-3-amino-acrylates with 2-amino arylbenzamides: one-pot synthesis of pyrrole–quinazoline hybrids†

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The one-pot domino reaction of ethyl (Z)-3-amino-3-phenylacrylates with 2-amino-*N*-alkyl/arylbenzamides under Lewis acid catalysis was described as an effective way to construct novel spiro [pyrrole-3,2'-quinazoline] carboxylate derivatives. By combining substituted alkyl/aryl amides with spiro annulated 1*H*-pyrrole-2,3-diones, this method provides a novel way for producing spiro pyrrole derivatives in good to excellent yields. The current procedure has a number of benefits, including quicker reaction times, a broad tolerance range for functional groups, and the ability to synthesize 2,3-dihydroquinazolin-4(1*H*)-ones that are of biological importance and take part in organic transformations. This is the first use of molecular hybridization involving linking with pyrrole derivatives and dihydroquinazolin-4(1*H*)-ones.

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Introduction

Lewis acids are versatile catalysts in organic synthesis for carbon–carbon or carbon–nitrogen bond formation.¹ Among them, bismuth(III) salts are efficient catalysts in various transformations;² in particular, bismuth(III) triflate is the mainly proficient catalyst for diverse organic transformations.³ Predominantly, the annulation reactions of 2-amino *N*-arylbenzamides with alkynes,⁴ aldehydes,⁵ alcohols,⁶ ketones,⁷ imines⁸ or aryl halides⁹ also attracted more attention for C–N bond creation. Indeed, spiro annulation of ketones with 2-amino *N*-arylbenzamides in the presence of metal or non-metal catalysts has been playing a significant role in producing different cyclized products concerning two different pharmacophores. In particular, dihydroquinazolinones are more attractive scaffolds that have been established as key building blocks in diverse transformations. These occur in various natural products and in synthetic molecules that find applications in the pharmaceutical chemistry.^{10–17} Along with this, pyrrole is also one of the most significant heterocycles, for the reason that it exists in a large number of natural and synthetic compounds with significant pharmacological properties.^{18–22} Spirooxindole is a privileged scaffold commonly found in naturally occurring and synthetic biologically active compounds.²³ Some of the important drugs with dihydroquinazolinone pharmacophore, pyrrole and

spirooxindole dihydroquinazolin-4(1*H*)-one core structures are shown in Fig. 1.

In drug discovery, the molecular hybridization technique plays an important role in linking and expansion of different pharmacophores.²⁴ Despite the expansion of the spiro annulated reactions with 2-amino-*N*-arylbenzamides were explored. In the literature studies, several methods are available for the linkage of isatins with 2-amino-*N*-phenylbenzamides to produce spirooxindole quinazolinone derivatives.

Iron-catalyzed oxidative coupling of indoline-2-ones with aminobenzamides under iron chloride catalysis;²⁵ SPINOL-CPA catalyzed cyclization of 2-amino-*N*-substituted benzamides and isatins;²⁶ asymmetric cyclization reactions between *N*-substituted anthranilamides and isatins;²⁷ stannous chloride catalyzed reductive cyclization of 2-nitrobenzamides with isatins;²⁸ alum catalyzed regioselective reaction of isatoic

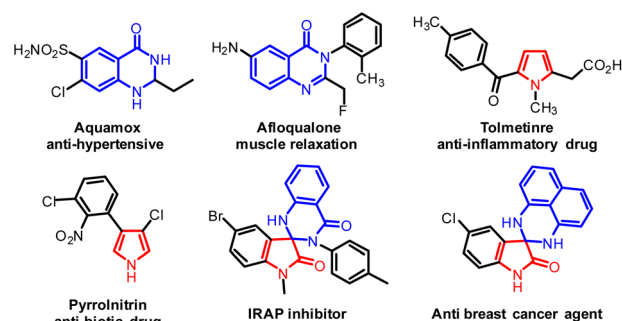


Fig. 1 Some of the pharmacologically important dihydroquinazolinones, pyrroles and spirooxindole dihydroquinazolinones.

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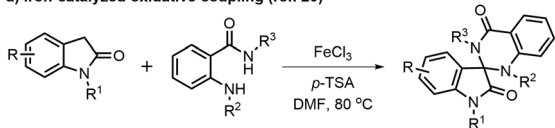
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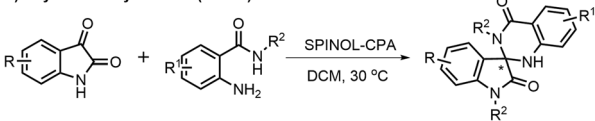


Previous work:

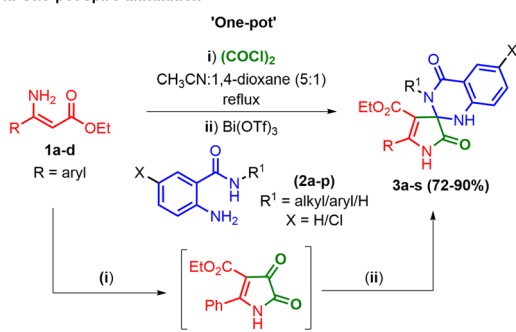
a) Iron-catalyzed oxidative coupling (ref. 20)



b) Asymmetric cyclization (ref. 21)



Present work: One-pot spiro annulation



Scheme 1 Synthesis of spiro [pyrrole-3,2'-quinazoline]carboxylates.

anhydride, isatins and aromatic/aliphatic amines;²⁹ one pot synthesis of spirooxindole dihydroquinazolinone derivatives under nano cerium oxide catalysis.³⁰ The synthesis of spirooxindole quinazoline derivatives uses all methods described in the literature; however, there is no literature on the synthesis of spiro[pyrrole-3,2'-quinazoline]carboxylates. Hybrids of quinazoline and pyrrole have not yet been reported.

This exacting reaction proceeds in two sequential steps; first, the cyclization of the (*Z*)-3-amino-3-phenylacrylates transpires to form 1*H*-pyrrole-2,3-diones,³¹ which are then converted to the desired products by spiro annulation with 2-amino-*N*-arylbenzamides (Scheme 1).

Moreover, one-pot synthesis reactions are helpful for avoiding several purification procedures at the same time and it can thus reduce chemical waste.³² The multicomponent reactions have attracted a high level of interest in the pharma industry due to their synthetic efficiency, high reactivity and atom economy.³³

Herein, we have established the first Lewis acid catalyzed one-pot spiro annulation of *in situ* generated 1*H*-pyrrole-2,3-diones from ethyl (*Z*)-3-amino-3-phenylacrylates and 2-amino-*N*-alkyl/aryl benzamides for the synthesis of *N*-heterocyclic spiro [pyrrole-3,2'-quinazoline]carboxylates, as shown in Scheme 1. The prominent features of this work include multicomponent one-pot reactions, novel spiro annulation under Lewis acid catalysis, the construction of a C–N framework, and abundant substrate scope with amides.

To the best of our knowledge, there is no precedent in the literature for using Lewis acids as a catalyst and ethyl (*Z*)-3-amino-3-phenylacrylates for the preparation of novel spiro [pyrrole-3,2'-quinazoline]carboxylates.

Results and discussion

Chemistry

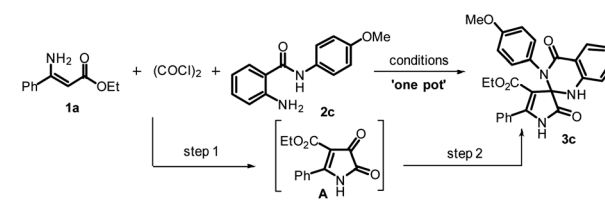
To begin the study for optimal conditions, ethyl (*Z*)-3-amino-3-phenylacrylate (**1a**), oxalyl chloride, and 2-amino-*N*-(4-methoxyphenyl) benzamides (**2c**) were taken as model substrates, and the results are mentioned in Table 1. In the beginning, **1a** was reacted with oxalyl chloride in dichloromethane at reflux, and we observed the formation of the intermediate ethyl 4,5-dioxo-2-phenyl-4,5-dihydro-1*H*-pyrrole-3-carboxylate (**A**) in 68% yield (entry 1, Table 1). To get a better yield of **A**, we carried out the reaction in various solvents using acetonitrile in combination with a solvent such as 1,4-dioxane, toluene, tetrahydrofuran, 1,2-dichloro ethane, and diethyl ether in 5 : 1 ratio (entries 2–6, Table 1).

However, all these reactions provided the intermediate **A** exclusively, and the best yield was obtained in acetonitrile. To achieve the spiro[pyrrole-3,2'-quinazoline]-4-carboxylate **3c** in one pot, we added 2-amino-*N*-arylbenzamide (**2c**) to the same reaction mixture, but no formation of **3c** was observed. In view of this unsuccessful result in attaining **3c**, the approach of the reaction was modified. Primarily, the formation of **A** under appropriate conditions (entry 6, Table 1) was recognized by thin-layer chromatography (TLC), and then, 10 mol% of Bi(OTf)₃ was added along with **2c** and stirred for 2 h. To our delight, the formation of **3c** was observed in 90% yield (entry 7, Table 1). Next, we also studied the reaction with different catalysts in the optimized solvent medium, and the results are shown in Table 1. In a while, the replacement of Bi(OTf)₃ with other Lewis acids in step 2 showed that indium triflate, copper triflate, zinc chloride, iodine, europium triflate, indium chloride, zinc triflate, ferric chloride, and BF₃·OEt₂ also gave the product **3c** in yields of 82, 78, 75, 75, 74, 70, 68, 65 and 60%, respectively (entries 8–16, Table 1). In addition, we varied the loading of the catalyst Bi(OTf)₃, and the reactions were conducted with 5 and 20 mol% of the catalyst, and a moderate yield of **3c** was observed with a decrease in the catalyst loading, while no significant effect was observed on the product yield with an increasing amount of catalyst (entries 17–18, Table 1). From these observations, Bi(OTf)₃ (10 mol%) is the best catalyst, and CH₃CN-1,4-dioxane is the most suitable solvent medium for this transformation. Hence, the conditions used in entry 7 (Table 1) were found to be most favorable to exploring the preparation of differently substituted spiro[pyrrole-3,2'-quinazoline]-4-carboxylate from the reaction of (*Z*)-3-amino-acrylate with a 2-amino arylbenzamide.

From the optimal reaction conditions (entry 7, Table 1), we next turned our interest to evaluate the overview of this conversion with various 2-amino-*N*-alkyl/arylbenzamides (**2a–p**) examined, and the results are shown in Table 2.

Pleasingly, a wide range of 2-amino-*N*-alkyl/arylbenzamides are well companionable with the present transformation to provide the corresponding spiro [pyrrole-3,2'-quinazoline]carboxylates in good yields. 2-Amino-*N*-arylbenzamides having electron donating groups **2b** to **2d** were well reacted with **1a** and oxalyl chloride to give the products



Table 1 Optimization of reaction conditions^a


| Entry | Reaction conditions for step 1 solvent (5 : 1) | Reaction conditions for step 2 Lewis acid (mol%) | % yield ^e | |
|-----------------|--|--|----------------------|-----------|
| | | | A | 3c |
| 1 | DCM : 1,4-dioxane | — | 68 | — |
| 2 | Toluene : 1,4-dioxane | — | 75 | — |
| 3 | THF : 1,4-dioxane | — | 78 | — |
| 4 | DCE : 1,4-dioxane | — | 81 | — |
| 5 | Ether : 1,4-dioxane | — | 86 | — |
| 6 | CH ₃ CN : 1,4-dioxane | — | 95 | — |
| 7 ^b | CH ₃ CN : 1,4-dioxane | Bi(OTf) ₃ (10) | Trace | 90 |
| 8 | CH ₃ CN : 1,4-dioxane | In(OTf) ₃ (10) | Trace | 82 |
| 9 | CH ₃ CN : 1,4-dioxane | Cu(OTf) ₂ (10) | 17 | 78 |
| 10 | CH ₃ CN : 1,4-dioxane | ZnCl ₂ (10) | 20 | 75 |
| 11 | CH ₃ CN : 1,4-dioxane | I ₂ (10) | 20 | 75 |
| 12 | CH ₃ CN : 1,4-dioxane | Eu(OTf) ₂ (10) | 20 | 74 |
| 13 | CH ₃ CN : 1,4-dioxane | InCl ₃ (10) | 25 | 70 |
| 14 | CH ₃ CN : 1,4-dioxane | Zn(OTf) ₂ (10) | 27 | 68 |
| 15 | CH ₃ CN : 1,4-dioxane | FeCl ₃ (10) | 27 | 65 |
| 16 | CH ₃ CN : 1,4-dioxane | BF ₃ ·OEt ₂ (10) | 33 | 60 |
| 17 ^c | CH ₃ CN : 1,4-dioxane | Bi(OTf) ₃ (5) | 15 | 78 |
| 18 ^d | CH ₃ CN : 1,4-dioxane | Bi(OTf) ₃ (20) | Trace | 90 |

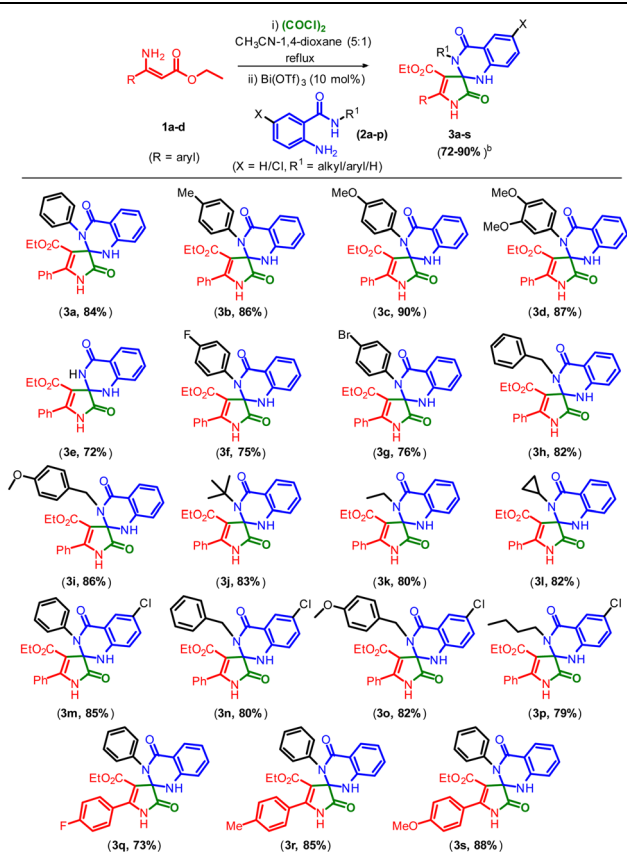
^a Reaction conditions: all the reactions were performed using **1** (1.0 mmol), oxalyl chloride (1.15 mmol), **2c** (1.0 mmol) and Lewis acid (10 mol%) in 1.0 mL of the solvent at reflux for 2–4 h. ^b For entries 7–16, after confirming the formation of **A** by TLC, 10 mol% of Lewis acid and **2c** (1.0 mmol) (step 2) were added and stirred for 2–4 h. ^c Bi(OTf)₃ (5 mol%). ^d Bi(OTf)₃ (20 mol%). ^e Isolated yields.

methyl (–CH₃, **3b**), methoxy (–OCH₃, **3c**) and di-methoxy (3,4-OCH₃, **3d**) in 86, 90, and 87% yields, respectively. Interestingly, having free amide functionality compound **2e** underwent reaction smoothly to provide the corresponding product **3e** in a 72% yield. 2-Amino-*N*-arylbenzamides **2f** and **2g** which bear halogens were also found to be equally consenting in reaction with **1a** and oxalyl chloride under similar reaction conditions to give the products fluoro (–F, **3f**) and bromo (–Br, **3g**) in 75 and 76% yields, respectively. In addition, 2-amino-*N*-alkylbenzamides benzyl (**2h**), 4-OCH₃ benzyl (**2i**), *tert*-butyl (**2j**), ethyl (**2k**) and cyclopropyl (**2l**) were also well tolerated to react with **1a** and oxalyl chloride under optimal conditions to give the corresponding products **3h**, **3i**, **3j**, **3k** and **3l** in 82, 86, 83, 80, and 82% yields, respectively. Further, other 5-Cl substituted 2-amino-*N*-aryl/alkyl benzamides such as phenyl (**2m**), benzyl (**2n**), 4-OCH₃ benzyl (**2o**) and *n*-butyl (**2p**) were found to react with **1a** and oxalyl chloride to give the desired products **3m**, **3n**, **3o** and **3p** in 85, 80, 82 and 79% yields, respectively. To further expand the substrate scope, the reactivity of diversely substituted phenyl (4-F, **1b**), (4-CH₃, **1c**) and (4-OCH₃, **1d**) was well tolerated to react with oxalyl chloride under optimized conditions and followed by a reaction with **2a** to obtain the corresponding products **3q**, **3r** and **3s** in 73, 85, and 88% yields, respectively.

To analyze the expediency of this method, a gram-scale reaction of compound **1a** with **2p** was carried out under standard reaction conditions (Scheme 2a). To our delight, the formation of **3p** was observed in 76% yield, and moreover, it was further utilized for the synthetic transformations, as shown in Scheme 2b. Palladium-catalyzed C–C coupling reaction of compound **3p** with phenylboronic acid **4** in toluene : water (1 : 1) as the solvent medium delivered the ethyl 3'-butyl-2,4'-dioxo-5,6'-diphenyl-1,2,3',4'-tetrahydro-1'*H*-spiro[pyrrole-3,2'-quinazoline]-4-carboxylate **5** in 82% yield.³⁴ Next, we turned our attention towards the generality of this transformation with *o*-phenylenediamine **6** in the presence of standard reaction conditions. Delightfully, the reaction progressed well and gave the ethyl 2-phenyl-1*H*-pyrrolo[2,3-*b*]quinoxaline-3-carboxylate **7** in an 88% yield (Scheme 2c).

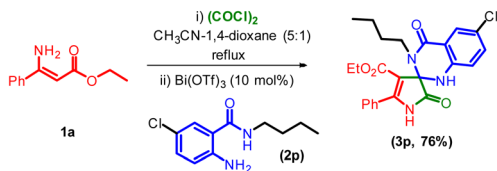
The obtained results from control experiments are shown in Scheme 3. In this observation, when the reaction was carried out with CH₃CN-1,4-dioxane as a solvent medium, we got intermediate **A** from the reaction of ethyl (*Z*)-3-amino-3-phenylacrylate **1a** and oxalyl chloride (Scheme 3, eqn (1)). Then intermediate **A** was transformed into ethyl 2,4'-dioxo-3',5'-diphenyl-1,2,3',4'-tetrahydro-1'*H*-spiro[pyrrole-3,2'-quinazoline]-4-carboxylate **3a** when it reacted with 2-amino-*N*-phenylbenzamide **2a** under Lewis acid catalysis (Scheme 3, eqn (2)).



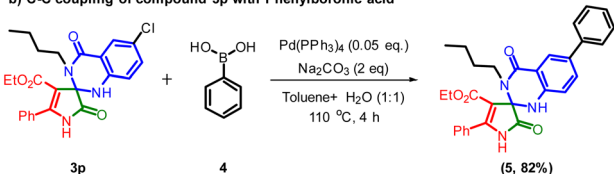
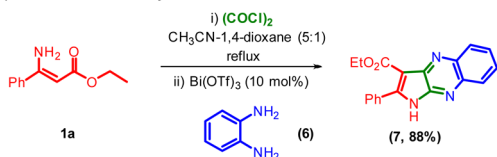
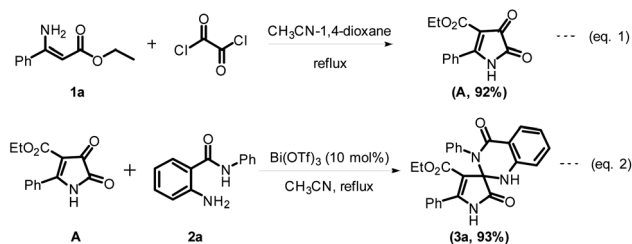
Table 2 Scope of 2-amino *N*-alkyl/arylbenzamides^a

^a Reaction conditions: all the reactions were performed using **1** (1 equiv.) with oxalyl chloride (1 equiv.) in CH₃CN-1,4-dioxane (5 : 1) and then **2** (1 equiv.) and Bi(OTf)₃ (10 mol%) at reflux for 4–8 h. ^b Isolated yields.

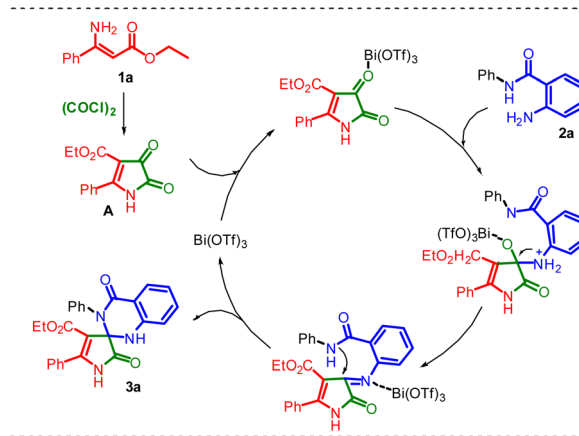
a) Gram scale reaction



b) C-C coupling of compound 3p with Phenylboronic acid

c) Reaction with *o*-PhenylenediamineScheme 2 (a) Gram scale synthesis of **3p**; (b) synthetic application of compound **3p**; (c) reaction of compound **3a** with *o*-phenylenediamine.

Scheme 3 Study of control experiments.

Scheme 4 Plausible mechanism for the formation of ethyl 2,4'-dioxo-3',5-diphenyl-1,2,3',4'-tetrahydro-1'H-spiro[pyrrole-3,2'-quinazoline]-4-carboxylate **3a**.

The noteworthy observation in this study is that the compound **1a** transformed into **3a** when **1a** reacted with oxalyl chloride and then into **2a** under Lewis acid catalysis.

On the basis of the results illustrated above and previous experiments, we suggest a feasible mechanism for the spiro annulation of ethyl (*Z*)-3-amino-3-phenylacrylate **1a** and 2-amino *N*-phenylbenzamide **2a**, as shown in Scheme 4. Initially, the intermediate compound **A** is formed by the cyclization of **1a** and oxalyl chloride in the presence of the solvent medium. Then, the compound **3a** was generated from the compound **A** by spiro annulation with **2a** in the presence of Lewis acid catalysis. This route for the formation of **3a** from compound **A** probably involves some transient stages.

Conclusions

In conclusion, we have demonstrated a highly efficient Lewis acid promoted spiro annulation for the one-pot synthesis of spiro[pyrrole-3,2'-quinazoline]-4-carboxylate derivatives using ethyl-(*Z*)-3-amino-3-phenylacrylate with oxalyl chloride and 2-amino-*N*-alkyl/arylbenzamides. This work established the first application of hybridization of pyrroles with dihydroquinazolinones by spiro annulation. The short reaction times and uncomplicated reaction conditions make this one pot method particularly attractive for the efficient preparation of biologically and medicinally interesting spiro[pyrrole-3,2'-quinazoline]-4-carboxylate molecules.



Conflicts of interest

The authors declare that they have no conflict of interest.

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