Copper-Catalyzed Domino Reaction of 2-Haloanilines with Hydrazides: A New Route for the Synthesis of Benzo[*e*][1,2,4]triazine Derivatives

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Abstract: A copper-catalyzed domino reaction for the synthesis of benzo[*e*][1,2,4]triazine derivatives has been developed using readily available substituted 2-haloanilines and hydrazides as the starting materials. The procedure of the present method is mild, facile, and efficient.

Key words: benzo[*e*][1,2,4]triazine derivatives, 2-haloanilines, hydrazides, copper catalyst, domino reaction

1,2,4-Triazines are a well-known class of heterocyclic compounds and they have many applications including aza-nucleotides and -nucleosides,[2] herero Diels–Alder reactants,[3] ligands,[4] herbicidal,[5] metal ion adsorption.[6] Moreover, the most noteworthy property of 1,2,4-triazines is their biological activity, a large number of the related literature have been reported.[7] For example, 3-amino-1,2,4-benzotriazine-1,4-dioxide, (tirapazamine TPZ), one of the most promising prodrugs, is a bioreductive antitumor agent that is selectively toxic to hypoxic cells. Indeed, the study of TPZ has been a central occupation for many scientists.[8] Therefore, as the main body of TPZ, benzo[1,2,4]triazines have drawn extensive and enduring attentions.

In our continuous study on the synthesis of bioactive N-heterocycles, we have developed many simple routes to synthesize several compounds of N-heterocycles.[9] Accordingly, we directed our attention more commonly towards synthesizing benzo[1,2,4]triazine derivatives. To the best of our knowledge, several strategies for the synthesis of benzo[*e*][1,2,4]triazines have been reported. Typically, in 2006, Khodja et al. reported the two-step syntheses of 3-methyl and 3-phenyl-1,2,4-benzotriazines.[10a] This method was clean and not strenuous, however, it needed a reductive cyclization by PtO_2- or Pt/C-catalyzed hydrogenation. In 2010, Wang's group observed a pathway for the synthesis of 3-aryl-1,2,4-benzotriazine via rearrangement of bis(benzotriazol-1-yl)methylarenes.[10b] It is worth mentioning that this reaction also included two steps and required allylsamarium bromide as the catalyst. Other methods generally suffer from some limitations such as complicated procedures, harsh reaction conditions, and substrates that are not readily available.[10c–10h] In recent years, domino reactions have attracted a great deal of interest[11] for the reason that they have many advantages, such as effective, environmental friendly, and resource preservation, etc.[12] Herein, we wish to report an efficient copper-catalyzed domino strategy for the synthesis of benzo[*e*][1,2,4]triazine.

Initially, 2-iodoaniline (**1a**) and benzohydrazide (**2a**) were used as the model substrates to optimize the reaction conditions including catalysts, bases, solvents, and reaction temperatures under nitrogen. As shown in Table 1, four copper catalysts (0.1 equiv) were tested with two equivalents of Cs_2CO_3 (relative to the amount of 2-iodoaniline) as the base and DMF–1,4-dioxane as the solvent at 90 °C (Table 1, entries 1–4), and Cu_2O provided the highest yield (Table 1, entry 4). Other bases, KO*t*-Bu, K_2CO_3, pyridine, MeONa, and Bu_4NBr (Table 1, entries 5–9), were screened, and Cs_2CO_3 showed the best result (Table 1, compare entries 4, 5–9). The effect of solvents was also investigated, and DMF–1,4-dioxane (1:1) was the optimal solvent choice (Table 1, compare entries 4 and 10–13). We attempted different reaction temperatures (Table 1, entries 14 and 15), and 90 °C was chosen to be the best choice. In addition, to optimize further the reaction conditions, the same reaction was carried out under air, but it failed to improve the yield (Table 1, entry 17). However, the yield did not increase when the reaction time prolonged to eight hours (Table 1, entry 16).

Under the optimized reaction conditions, we then extended the scope of this reaction, and the results are illustrated in Table 2. Generally, the reaction of 2-iodoanilines with hydrazides proceeded smoothly and led to the desired product **3** with moderate yields. As shown in Table 2, a variety of 2-iodoanilines were efficient to give the desired benzo[*e*][1,2,4]triazine products (Table 2, entries 2–7). It is obvious that the electron density on 2-iodoanilines drastically affects the yields of this reaction (Table 2, compare entries 2–5 and 6, 7). Then the reaction scope of benzohydrazide was studied. From **2b** to **2i**, the electron-donating or electron-withdrawing groups on the benzene ring did not have distinct influence on the yields of **3** except **2e** and **2f**. In addition, 2-iodobenzohydrazide (**2k**), possibly because of its steric hindrance, gave only 22% yield of product with **1a** (Table 2, entry 17). In these experiments, we also tried the reaction of **1a** with furan-2-carbohydrazide (**2j**), and we got the desired product. Notably, there was a special case as shown in Scheme 1. The target product of

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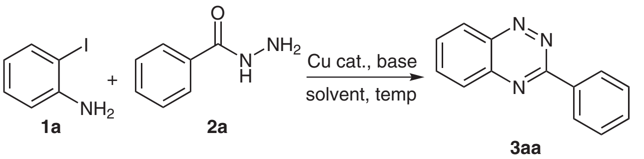
1g with **2a** was 7-bromo-3-phenylbenzo[*e*][1,2,4]triazine. However, by NMR, MS, and IR, the compound examined was 3-phenylbenzo[*e*][1,2,4]triazine (**3aa**), the substituent of Br queerly disappeared. The reason is not clear at the current stage, and investigations are ongoing in our laboratory.

Table 1 Optimization of Reaction Conditions with **1a** and **2a**[a]

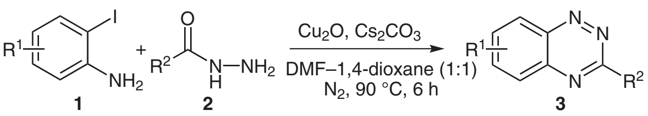


Entry	Catalyst	Base	Solvent	Temp (°C)	Yield (%)[b]
1	$Cu(OAc)_2$	Cs_2CO_3	DMF–1,4-dioxane (1:1)	90	39
2	CuBr	Cs_2CO_3	DMF–1,4-dioxane (1:1)	90	41
3	CuI	Cs_2CO_3	DMF–1,4-dioxane (1:1)	90	52
4	Cu_2O	Cs_2CO_3	DMF–1,4-dioxane (1:1)	90	75
5	Cu_2O	KO*t*-Bu	DMF–1,4-dioxane (1:1)	90	56
6	Cu_2O	K_2CO_3	DMF–1,4-dioxane (1:1)	90	34
7	Cu_2O	pyridine	DMF–1,4-dioxane (1:1)	90	n.r.[c]
8	Cu_2O	MeONa	DMF–1,4-dioxane (1:1)	90	40
9	Cu_2O	Bu_4NBr	DMF–1,4-dioxane (1:1)	90	n.r.[c]
10	Cu_2O	Cs_2CO_3	DMSO	90	50
11	Cu_2O	Cs_2CO_3	THF	90	21
12	Cu_2O	Cs_2CO_3	PhMe	90	n.r.[c]
13	Cu_2O	Cs_2CO_3	DMF	90	55
14	Cu_2O	Cs_2CO_3	DMF–1,4-dioxane (1:1)	80	37
15	Cu_2O	Cs_2CO_3	DMF–1,4-dioxane (1:1)	110	52
16	Cu_2O	Cs_2CO_3	DMF–1,4-dioxane (1:1)	90	75[d]
17	Cu_2O	Cs_2CO_3	DMF–1,4-dioxane (1:1)	90	36[e]

[a] Reaction conditions: **1a** (0.2 mmol), **2a** (0.3 mmol), base (0.4 mmol), catalyst (0.02 mmol) in 1 mL of solvent for 6 h under nitrogen atmosphere
[b] Isolated yield.
[c] n.r. = no reaction.
[d] Yield for a reaction time of 8 h.
[e] Under air.

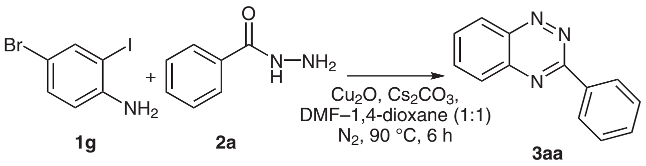
Encouraged by the results obtained, the reaction scope of 2-bromoanilines was studied. On the basis of the optimized reaction conditions above, we attempted to look for the optimal conditions of 2-bromoanilines with hydrazides. 2-Bromoaniline (**4a**) and benzohydrazide (**2a**) were used as the model substrates, and the procedure is illustrated in Table 3. The result of some different substrates is illustrated in Table 4.

Table 2 Synthesis of Benzo[*e*][1,2,4]triazines from 2-Iodoanilines and Hydrazides[a,13]



Entry	R^1	R^2	Product	Yield (%)[b]
1	**1a** H	**2a** Ph	**3aa**	75
2	**1b** 4-Me	**2a** Ph	**3ba**	57
3	**1c** 4-Et	**2a** Ph	**3ca**	56
4	**1d** 4-*i*-Pr	**2a** Ph	**3da**	56
5	**1e** 4,5-Me_2	**2a** Ph	**3ea**	64
6	**1f** 4-Cl	**2a** Ph	**3fa**	43
7	**1g** 4-Br	**2a** Ph	**3aa**	30
8	**1a** H	**2b** 4-MeC_6H_4	**3ab**	52
9	**1a** H	**2c** 3-MeC_6H_4	**3ac**	54
10	**1a** H	**2d** 3,5-$Me_2C_6H_3$	**3ad**	56
11	**1a** H	**2e** 4-$MeOC_6H_4$	**3ae**	40
12	**1a** H	**2f** 3,4-$(MeO)_2C_6H_4$	**3af**	36
13	**1a** H	**2g** 3-ClC_6H_4	**3ag**	65
14	**1a** H	**2h** 4-ClC_6H_4	**3ah**	62
15	**1a** H	**2i** 3-$O_2NC_6H_4$	**3ai**	61
16	**1a** H	**2j** furan-2-yl	**3aj**	46
17	**1a** H	**2k** 2-IC_6H_4	**3ak**	22
18	**1b** 4-Me	**2b** 4-MeC_6H_4	**3bb**	52
19	**1b** 4-Me	**2g** 3-ClC_6H_4	**3bg**	65
20	**1e** 4,5-Me_2	**2h** 4-ClC_6H_4	**3eh**	60
21	**1a** H	**2l** *i*-Bu	–	0

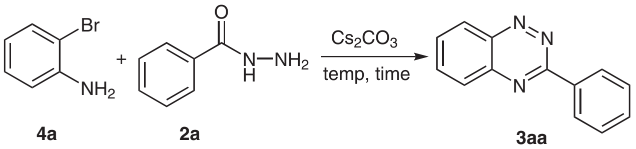
[a] Reaction conditions: **1** (0.2 mmol), **2** (0.3 mmol), Cs_2CO_3 (0.4 mmol), Cu_2O (0.02 mmol), N_2, DMF (0.5 mL) with 1,4-dioxane (0.5 mL), 90 °C, 6 h.
[b] Isolated yield after column chromatography.



Scheme 1 A special case

It is obvious from Table 2 and Table 4 that the result of 2-iodoanilines was superior to 2-bromoanilines in this reaction. On the other hand, the reaction of 2-bromoanilines with hydrazines explored the scope of this protocol. The structure of compound **3bg** (Figure 1) was confirmed by single-crystal X-ray analysis.

Table 3 Screening the Impact of Quantity of Base, Temperature, and Time[a]



4a **2a** **3aa**

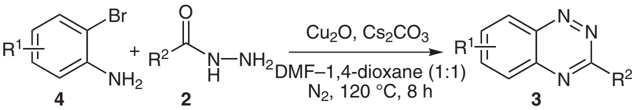
Entry	Cs_2CO_3 (equiv)	Temp (°C)	Time (h)	Yield (%)[b]
1	2.0	90	6	0
2	3.0	90	6	0
3	3.0	120	6	10
4	3.0	120	8	17
5	4.0	120	8	19
6	6.0	120	8	34 (18)[c]
7	6.0	120	10	33
8	8.0	120	8	34

[a] Reaction conditions: **4a** (0.2 mmol), **2a** (0.3 mmol), Cs_2CO_3, Cu_2O (0.02 mmol), N_2, DMF (0.5 mL) with 1,4-dioxane (0.5 mL), temp, time.

[b] Isolated yield after column chromatography.

[c] Yield for a reaction time of 6 h.

Table 4 Synthesis of Benzo[*e*][1,2,4]triazines from 2-Bromoanilines and Hydrazides[a]



4 **2** **3**

Entry	R^1	R^2	Product	Yield (%)[b]
1	**4a** H	**2a**	**3aa**	34
2	**4b** 4-Me	**2a**	**3ba**	25
3	**4c** 4-Cl	**2a**	**3fa**	4
4	**4b** 4-Me	**2b**	**3bb**	20
5	**4b** 4-Me	**2g**	**3bg**	10

[a] Reaction conditions: **4** (0.2 mmol), **2** (0.3 mmol), Cs_2CO_3 (1.2 mmol), Cu_2O (0.02 mmol), N_2, DMF (0.5 mL) with 1,4-dioxane (0.5 mL), 120 °C, 8 h.

[b] Isolated yield after column chromatography.

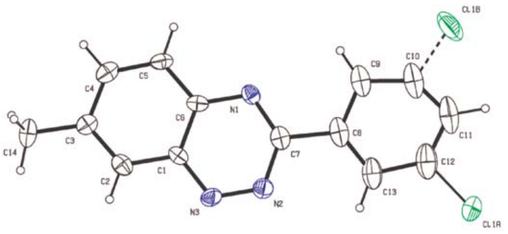
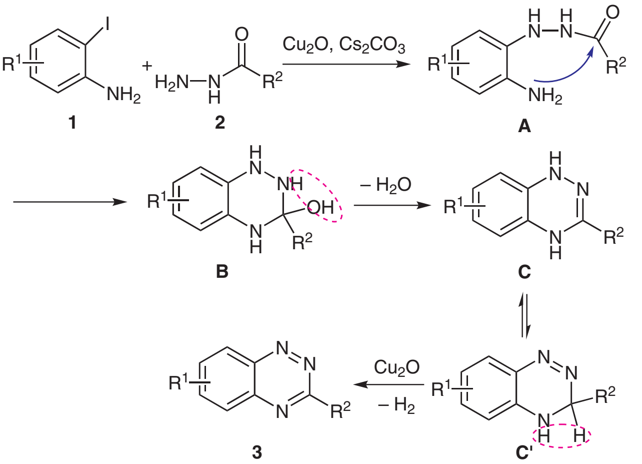


Figure 1 X-ray crystal structure of **3bg**

Nevertheless, the yields of all products are not very high. It is enlightening to recall that the coupling of hydrazides with aryl iodides more easily forms N-arylated products, and the yields of N′-arylated products are moderate or slightly low.[14]

A plausible mechanism is proposed in Scheme 2 that is based on previous reports[15] combined with our experimental results. Firstly, the Ullmann-type coupling of 2-iodoaniline **1** with hydrazide **2** provides **A**. Then NH_2 attacks the carbonyl group affording the intermediate **B**, which would lose H_2O to form **C**. As a result of tending to conjugation, the C=N bond of **C** may shift to the N=N bond forming **C′**. Finally, oxidative aromatization[16] with removal of hydrogen from **C′** in the presence of Cu_2O would yield the target product **3**.



Scheme 2 Proposed mechanism

In summary, we have developed a simple and efficient copper-catalyzed method for the synthesis of benzo[*e*][1,2,4]triazine derivatives. The protocol uses cheap and less toxic Cu_2O as the catalyst, readily available 2-haloanilines and hydrazides as the starting materials, and the reaction conditions are mild. Many substrates are suitable for this procedure, affording various benzo[*e*][1,2,4]triazine derivatives in moderate yields. This strategy may be very useful for the synthesis of benzo[*e*][1,2,4]triazines and corresponding natural products. A more detailed investigation on the mechanism and scope of this reaction as well as the utilization of this reaction in the synthesis of complex molecules is ongoing in our laboratory.

Supporting Information for this article is available online at http://www.thieme-connect.com/ejournals/toc/synlett.

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References and Notes

(1) These authors contributed equally to this work.

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(13) **Typical Procedure for the Synthesis of Benzo[*e*][1,2,4]triazine**
An oven-dried Schlenk tube was charged with Cu_2O (2.9 mg, 0.02 mmol), Cs_2CO_3 (130.4 mg, 0.4 mmol), **1a** (44 mg, 0.20 mmol) and **2a** (41 mg, 0.3 mmol). The Schlenk tube was sealed, evacuated, and backfilled with nitrogen (3 cycles). Then DMF (0.5 mL) and 1,4-dioxane (0.5 mL) were added to the reaction tube. The reaction was stirred at 90 °C under N_2 for 6 h. After cooling to r.t. the solvent was diluted with EtOAc (3×10 mL), washed with brine (3×10 mL), and dried over anhyd Na_2SO_4. After the solvent was evaporated in vacuo, the residues were purified by column chromatography, eluting with PE–EtOAc (20:1) to afford 31 mg (75%) of 3-phenylbenzo[*e*][1,2,4]triazine (**3aa**) as a yellow solid, mp 119–121 °C. ^1H NMR (400 MHz, $CDCl_3$): $\delta = 8.77–8.74$ (m, 2 H), 8.51 (dd, $J = 8.4, 0.8$ Hz, 1 H), 8.07 (d, $J = 8.8$ Hz, 1 H), 7.96–7.92 (m, 1 H), 7.83–7.78 (m, 1 H), 7.59–7.57 (m, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 160.0$, 146.6, 141.2, 135.8, 135.6, 131.6, 130.3, 129.7, 129.3, 129.1, 128.9. IR (neat): 3368, 3060, 1506, 1329, 1016, 771, 699 cm^{-1}. HRMS: m/z calcd for $C_{13}H_{10}N_3$ $[M + H]^+$: 208.0870; found: 208.0871.

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