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Copper-catalyzed synthesis of pyrido-fused quinazolinones from 2-aminoarylmethanols and isoquinolines or tetrahydroisoquinolines†

Thao T. Nguyen, ‡[a,b] Khang X. Nguyen, ‡[a,b] Phuc H. Pham, [a,b] Duc Ly, [a,b] Duyen K. Nguyen,[a,b] Khoa D. Nguyen,[a,b] Tung T. Nguyen *[a,b] and Nam T. S. Phan *[a,b]

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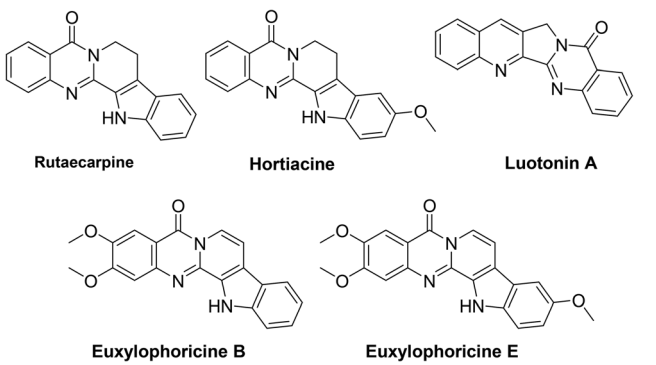
Pyrido-fused quinazolinones were synthesized *via* copper-catalyzed cascade C(sp^2)–H amination and annulation of 2-aminoarylmethanols with isoquinolines or pyridines. The transformation proceeded readily in the presence of a commercially available $CuCl_2$ catalyst with molecular oxygen as a green oxidant. Moreover, the dehydrogenative cross-coupling of 2-aminoarylmethanols with tetrahydroisoquinolines was explored, in which CuBr exhibited higher catalytic activity than $CuCl_2$. Broad substrate scope with good tolerance of functionalities was observed under the optimized reaction conditions. The bioactive naturally occurring alkaloid rutaecarpine could be obtained by this strategy. The remarkable feature of this protocol is that complicated heterocyclic structures are readily achieved in a single synthetic step from easily accessible reactants and catalysts. This pathway to pyrido-fused quinazolinones would be complementary to existing protocols.

Introduction

Quinazolinones are a significant class of N-heterocyclic compounds, frequently present in a wide range of pharmaceutically relevant natural products, drug candidates, agricultural chemicals, and functionalized organic materials.[1–3] Among numerous quinazolinone derivatives, fused quinazolinones play essential roles in drug discovery and development due to their versatile biological and pharmacological activities.[4–6] Representative fused quinazolinones in natural products and drug discovery research are illustrated in Scheme 1.[7–10] Classical synthetic strategies mainly relied on the reactions of electron-deficient 2-chloropyridines with substituted anthranilic acids, suffering from several disadvantages.[11] Metal-free protocols have been developed to access these structures.[12,13] Alternative pathways utilizing transition metal catalysts have been explored. Palladium-catalyzed transformations have been studied, including dearomatizing carbonylation protocols,[14] base-controlled carbonylative coupling followed by nucleophilic aromatic substitution,[15] C(sp^2)–H pyridocarbonylation with carbon monoxide,[16] direct carbonylation of C(sp^2)–H bonds with dimethylformamide,[17] and carbonylation/nucleophilic aromatic substitution reaction sequences.[18] Cheaper copper-based catalysts have been targeted for reactions, including domino transformations between 2′-haloacetophenones and 2-aminopyridines,[19] tandem reactions *via* oxidation, intramolecular cyclization and decarbonylation[20] (Scheme 2a), domino transformations from 2-bromopyridines and isatins[9] (Scheme 2b), and tandem C(sp^2)–H amination fol-



Scheme 1 Representative fused quinazolinones in natural products and drug discovery research.

[a]*Faculty of Chemical Engineering, Ho Chi Minh City University of Technology (HCMUT), 268 Ly Thuong Kiet, District 10, Ho Chi Minh City, Vietnam. E-mail: tungtn@hcmut.edu.vn, ptsnam@hcmut.edu.vn; Fax: (+84 8)38637504; Tel: (+84 8) 38647256 ext. 5681*

[b]*Vietnam National University Ho Chi Minh City, Linh Trung Ward, Thu Duc District, Ho Chi Minh City, Vietnam*

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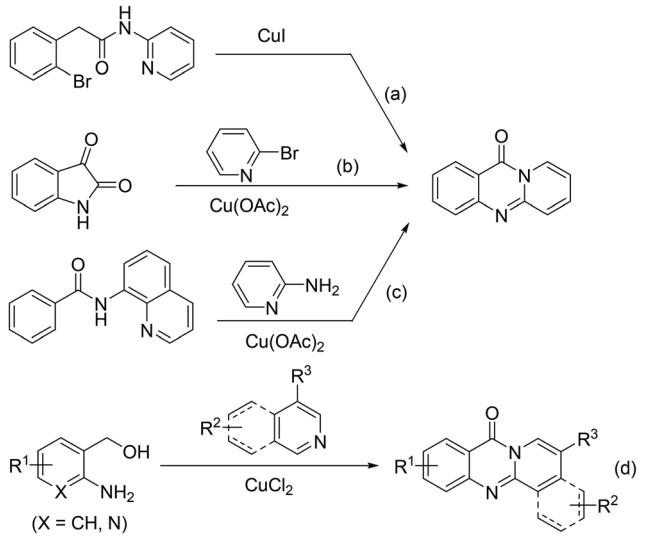
‡These authors contributed equally to this work.



Scheme 2 Copper-catalyzed synthesis of pyrido-fused quinazolinones.

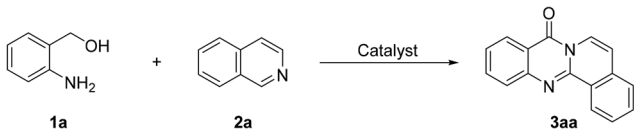
lowed by annulation[21] (Scheme 2c). Additionally, a cobalt nano-catalyst has been employed for the synthesis of ring-fused quinazolinones from aminoarylmethanols and cyclic amines.[22]

Direct C–H bond functionalization has been considered as an environmentally benign and robust method in organic synthesis, averting the necessity of pre-installation of a reactive functional group in coupling precursors.[23–25] This strategy typically reduces the number of transformations in multi-step syntheses which could involve complicated and laborious protection–deprotection sequences, thus offering the advantages of atom economy and waste minimization when compared to traditional synthetic approaches.[26–28] Cascade reactions have emerged as key steps in the efficient synthesis of complex structures, minimizing the amounts of waste generated by several chemical processes.[29,30] Therefore, cascade reactions *via* direct C–H bond amination would be desirable for the synthesis of nitrogen-containing heterocycles.[31] Over the last decade, numerous structures have been achieved through the C–H bond amination pathway, using different transition metal catalysts, including nickel,[32] rhodium,[33] iron,[34] manganese,[35] copper,[36] cobalt,[37] and palladium.[36] In this work, we would like to present the synthesis of pyrido-fused quinazolinones *via* copper-catalyzed cascade C(sp^2)–H amination and annulation of 2-aminoarylmethanols with isoquinolines or pyridines (Scheme 2d). A variety of pyrido-fused quinazolinone derivatives were obtained in the presence of a readily available copper(II) chloride catalyst, utilizing molecular oxygen as a green oxidant. This synthetic strategy would be complementary to previous approaches in the synthesis of these valuable heterocyclic structures.

Results and discussion

The research was commenced with the reaction of 2-aminobenzyl alcohol (**1a**) and isoquinoline (**2a**) to form 8*H*-isoquinolino[1,2-*b*]quinazolin-8-one (**3aa**) (Table 1). Initial studies indicated that the transformation required the presence of a

Table 1 Screening of conditions for the annulation of 2-aminobenzyl alcohol with isoquinoline[a]

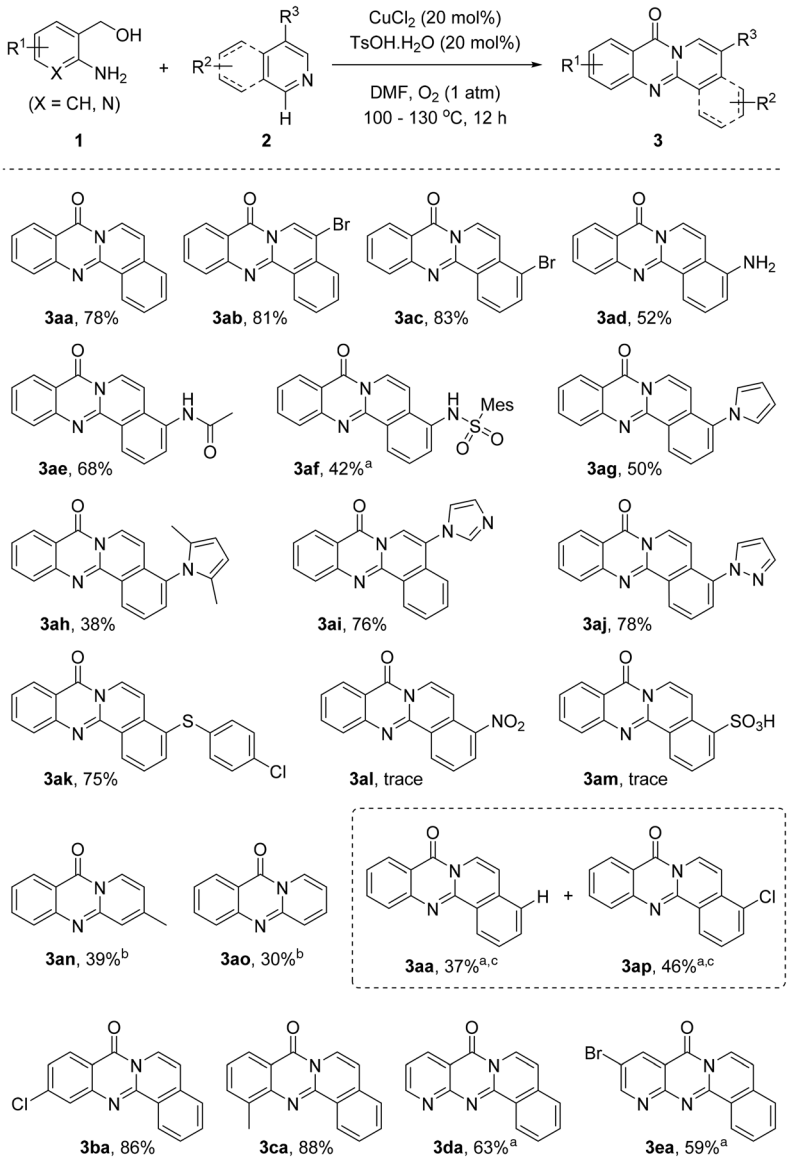


1a **2a** **3aa**

Entry	Temperature (°C)	Catalyst (mol%)	**1a** : **2a** (mol : mol)	Additive (equiv.)	Solvent (mL)	Yield[b] (%)
1	80	$Cu(OAc)_2$ (20%)	1 : 3	TsOH·H_2O (1.5)	DMF (0.5 mL)	27
2	100	$Cu(OAc)_2$ (20%)	1 : 3	TsOH·H_2O (1.5)	DMF (0.5 mL)	52
3	120	$Cu(OAc)_2$ (20%)	1 : 3	TsOH·H_2O (1.5)	DMF (0.5 mL)	51
4	100	—	1 : 3	TsOH·H_2O (1.5)	DMF (0.5 mL)	0
5	100	$CuCl_2$ (20%)	1 : 3	TsOH·H_2O (1.5)	DMF (0.5 mL)	64
6	100	$CuBr_2$ (20%)	1 : 3	TsOH·H_2O (1.5)	DMF (0.5 mL)	62
7	100	CuBr (20%)	1 : 3	TsOH·H_2O (1.5)	DMF (0.5 mL)	61
8	100	CuCl (20%)	1 : 3	TsOH·H_2O (1.5)	DMF (0.5 mL)	63
9	100	$CuCl_2$ (20%)	1 : 2.5	TsOH·H_2O (1.5)	DMF (0.5 mL)	58
10	100	$CuCl_2$ (20%)	1 : 3	—	DMF (0.5 mL)	6
11	100	$CuCl_2$ (20%)	1 : 3	HCOOH (1.5)	DMF (0.5 mL)	37
12	100	$CuCl_2$ (20%)	1 : 3	AcOH (1.5)	DMF (0.5 mL)	46
13	100	$CuCl_2$ (20%)	1 : 3	CH_3SO_3H (1.5)	DMF (0.5 mL)	65
14	100	$CuCl_2$ (20%)	1 : 3	TsOH·H_2O (0.2)	DMF (0.5 mL)	68
15	100	$CuCl_2$ (20%)	1 : 3	TsOH·H_2O (0.2)	DMAc (0.5 mL)	67
16	100	$CuCl_2$ (20%)	1 : 3	TsOH·H_2O (0.2)	NMP (0.5 mL)	64
17	100	$CuCl_2$ (20%)	1 : 3	TsOH·H_2O (0.2)	DMSO (0.5 mL)	58
18	100	$CuCl_2$ (20%)	1 : 3	TsOH·H_2O (0.2)	DMF (1.5 mL)	81

[a] Reaction conditions: 2-aminobenzyl alcohol (0.1 mmol; 12 h; reactor flushed with oxygen). TsOH: *p*-toluenesulfonic acid; DMF: *N*,*N*-dimethylformamide; DMSO: dimethyl sulfoxide; DMAc: *N*,*N*-dimethylacetamide; and NMP: *N*-methyl-2-pyrrolidone. [b] GC yield.

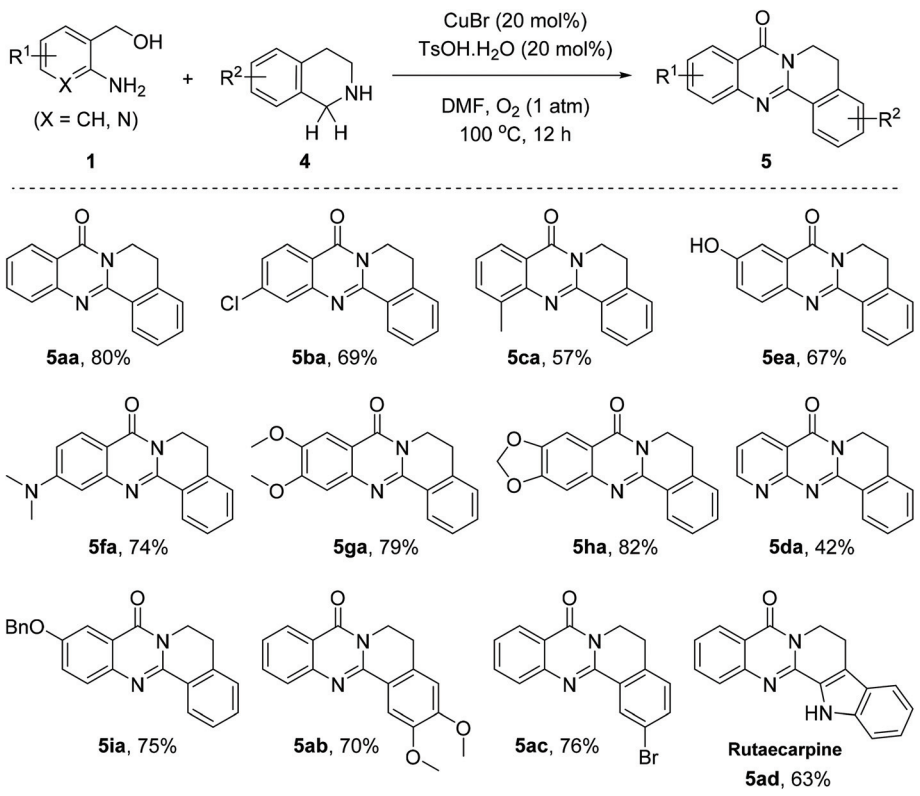
copper catalyst, an acidic additive, and molecular oxygen as an oxidant. Primarily, the reaction conditions were screened to improve the yield of **3aa**, regarding temperature, catalyst, additive, reactant molar ratio, and solvent (see Table S1† for detailed data). Conducting the reaction at various temperatures revealed that the best yield was obtained at 100 °C (entry 2). No trace amounts of the pyrido-fused quinazolinone product were detected in the absence of any catalyst (entry 4). A series of copper salts were employed for the reaction, and 20 mol% $CuCl_2$ exhibited the best performance with 64% yield of **3aa** being achieved (entry 5). Increasing or decreasing the amount of $CuCl_2$ resulted in lower yields of the desired product. Additionally, several iron salts were tested, offering lower activity than $CuCl_2$. Furthermore, the reactant molar ratio significantly influenced the transformation, and using 3 equivalents of **2a** offered the best result. Only 6% yield was obtained in the absence of any acidic additive (entry 10). Several organic acids were tested, and utilizing 20 mol% *p*-toluenesulfonic acid led to 68% yield (entry 14). Both increasing and decreasing the amount of the additive displayed a negative impact on the transformation. Moreover, by changing the solvent and solvent volume, the yield of **3aa** could be enhanced to 81% (entry 18). It should be noted that the reaction conducted under air afforded 37% yield while no product was detected for the experiment carried out under argon.



Scheme 3 Annulation of 2-aminoarylmethanols with isoquinolines and pyridines. Reaction conditions: 2-aminoarylmethanols **1** (0.1 mmol, 1.0 equiv.); isoquinolines **2** (0.3 mmol, 3.0 equiv.); $CuCl_2$ (20 mol%), $TsOH{\cdot}H_2O$ (20 mol%); DMF (1.5 mL), 100 °C; 12 h. [a] Reaction was performed at 120 °C. [b] Reaction was performed at 130 °C. [c] Isoquinoline-4-boronic acid pinacol ester (0.2 mmol, 2.0 equiv.) and $CuCl_2$ (1.2 equiv.) were used. Yields are isolated yields. Product isolation is based on the combination of 2 parallel reaction tubes.

With these results in mind, we subsequently explored the generality of the protocol in the synthesis of pyrido-fused quinazolinones (Scheme 3). First, a series of isoquinolines and pyridines were examined for the transformation with 2-aminobenzyl alcohol. Under standard conditions, **3aa** was obtained in 78% isolated yield. Bromo-substituted isoquinolines readily reacted with 2-aminobenzyl alcohol, producing **3ab** and **3ac** in 81% and 83% yields, respectively. Isoquinolin-5-amine and its derivatives were less reactive towards the transformation, though **3ad**, **3ae**, and **3af** were generated in 52%, 68%, and 42% yields, respectively. Similarly, the reaction of pyrrolyl-substituted isoquinolines proceeded with difficulty, affording **3ag** and **3ah** in 50% and 38% yields, respectively. In contrast, pyrazolyl-substituted isoquinolines are more reactive, with **3ai** and **3aj** being obtained in 76% and 78% yields, respectively. Compared to other reactants, 5-((4-chlorophenyl)thio)isoquinoline emerged as the most reactive candidate, forming **3ak** in 75% yield. Nitro- and sulfonic acid-substituted isoquinolines and pyridines exhibited lower reactivity in this transformation, presumably due to their low basicity (**3al**, **3am**, **3an**, and **3ao**). Interestingly, utilizing 4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)isoquinoline in the presence of excess $CuCl_2$ led to a mixture of **3aa** and **3ap** in 37% and 46% yields, respectively. In the next series of experiments, several derivatives of 2-aminobenzyl alcohol were used for annulation with isoquinoline, affording the corresponding heterocyclic products in high yields (**3ba**, **3ca**, **3da**, and **3ea**).

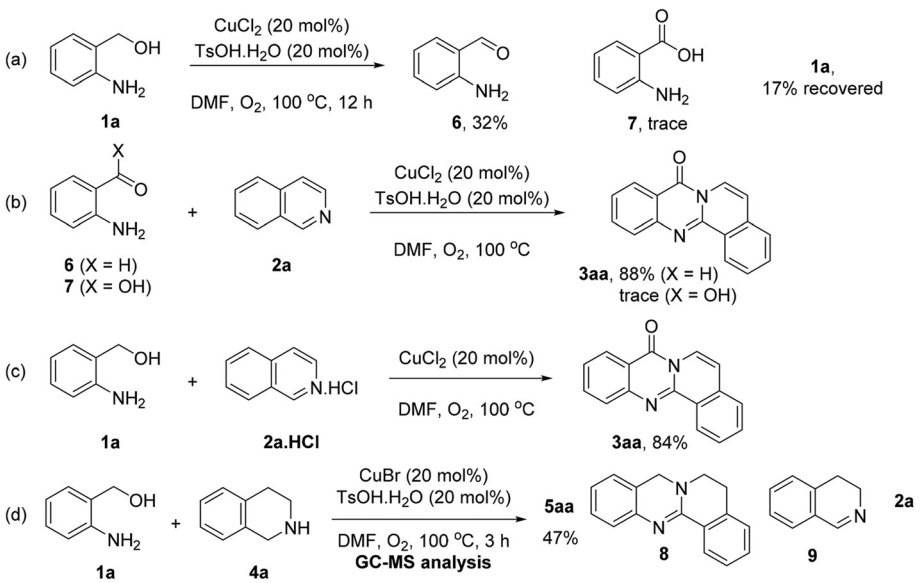
Additionally, the dehydrogenative cross-coupling of 2-aminoarylmethanols with tetrahydroisoquinolines was investigated (Scheme 4). Initially, the annulation of 2-aminobenzyl alcohol (**1a**) and tetrahydroisoquinoline (**4a**) to form 5,6-dihydro-8*H*-isoquinolino[1,2-*b*]quinazolin-8-one (**5aa**) was addressed. The reaction conditions were screened to maximize the yield of **5aa**, concerning temperature, catalyst, additive, reactant molar ratio, and solvent (see Table S2† for detailed data). Different from the formation of **3aa**, CuBr should be used instead of $CuCl_2$ for the synthesis of **5aa.** The best result was achieved for the reaction conducted in DMF at 100 °C for 12 h under oxygen, utilizing 3 equivalents of tetrahydroisoquinolines, in the presence of 20 mol% CuBr and 20 mol% $TsOH{\cdot}H_2O$. Under these conditions, 86% GC yield of **5aa** was detected (entry 42, Table S2†), and 80% isolated yield of **5aa** was achieved. Several derivatives of **5aa** were also synthesized utilizing the standard reaction conditions (Scheme 3). Substituted 2-aminobenzyl alcohols were reactive in the reaction with **4a**, producing the corresponding products in high yields (**5ba**, **5ca**, **5ea**, **5fa**, **5ga**, **5ha** and **5ia**). (2-Aminopyridin-3-yl)methanol was less reactive than **1a**, though **5da** was



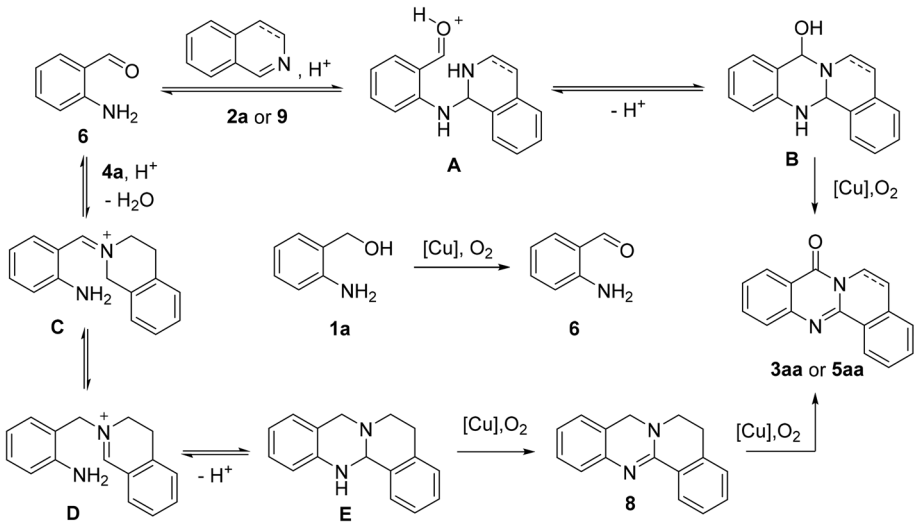
Scheme 4 Annulation of 2-aminoarylmethanols with tetrahydroisoquinolines. Reaction conditions: 2-aminoarylmethanols **1** (0.1 mmol, 1.0 equiv.); tetrahydroisoquinolines **4** (0.3 mmol, 3.0 equiv.); CuBr (20 mol%); $TsOH{\cdot}H_2O$ (20 mol%); DMF (0.5 mL); 100 °C; 12 h. Yields are isolated yields. Product isolation is based on the combination of 2 parallel reaction tubes.

obtained in 42% yield. The reaction of **1a** and 6,7-dimethoxy-1,2,3,4-tetrahydroisoquinoline hydrochloride proceeded to 70% yield of **5ab** under standard conditions. Interestingly, following this protocol, bioactive naturally occurring alkaloid rutaecarpine (**5ad**) was obtained in 63% yield. These observations were comparable to those previously reported in the literature, where a nanocobalt catalyst was employed for similar transformations.[22]

Control experiments were performed to probe the elementary steps of the transformation (Scheme 5). A plausible reaction pathway for the formation of (tetrahydro)isoquinolino-fused quinazolinone is proposed in Scheme 6. The active intermediate was believed to be 2-aminobenzaldehyde (**6**), which was formed *via* copper-catalyzed oxidation of the alcohol moiety under atmospheric oxygen.[38,39] The product **3aa** was formed *via* annulation reaction between 2-aminobenzaldehyde (**6**) and isoquinoline (**2a**), followed by oxidation to furnish the quinazolinone ring.[36] A similar pathway for the formation of **5aa** from tetrahydroisoquinoline (**4a**) is also possible, with the cyclic imine **9** being the active intermediate.[40] In both cases, the significant basicity of isoquinoline or the dihydroisoquinoline nitrogen triggered the Csp^2–H amination step. This was activated by a proton source, as evidenced from the control reaction using isoquinoline hydrochloride (Scheme 5c). An alternative mechanism for the formation of **5aa** is also provided, due to the observation of an amidine intermediate by



Scheme 5 Control experiments.



Scheme 6 Proposed reaction pathway.

GC-MS during the reaction progress. The cyclic amidine **8** was susceptible to oxidation when exposed to an oxygen atmosphere.[41] Additionally, the reaction of **6** with **2a** in the absence of the copper catalyst did not result in any trace amount of **3aa**. In contrast, **5aa** was detected by GC-MS for the reaction of **6** with **4a** in the absence of the copper catalyst. In both cases, intermediates **B** and **E** were not detected by GC-MS under these conditions due to their low stability. These observations imply that the conversion of **B** to **3aa** required the copper catalyst, while **E** could be converted to **5aa** without the copper species. Nevertheless, the presence of the copper catalyst accelerated the formation of **5aa** from **E**. Indeed, further investigation is needed to elucidate the mechanism of the transformation.

Conclusions

In summary, a new route to pyrido-fused quinazolinones *via* copper-catalyzed cascade C(sp^2)–H amination and annulation of 2-aminoarylmethanols with isoquinolines or pyridines was developed. The transformation proceeded readily in the presence of a commercially available copper salt catalyst, utilizing molecular oxygen as a green oxidant. Among a series of copper salts, $CuCl_2$ offered the best catalytic activity. An acidic additive was required, and *p*-toluenesulfonic acid monohydrate emerged as the best candidate for the formation of the pyrido-fused quinazolinones. Broad substrate scope with good tolerance of functionalities was observed under the optimized reaction conditions. Moreover, the dehydrogenative cross-coupling of 2-aminoarylmethanols with tetrahydroisoquinolines was investigated under similar conditions, in which CuBr exhibited higher catalytic activity than $CuCl_2$. Several ring-fused quinazolinones were obtained following this protocol. The remarkable feature of this protocol is that complicated heterocyclic structures are readily achieved in a single synthetic step from easily accessible reactants and catalysts. This pathway to pyrido-fused quinazolinones would be complementary to existing methods, and would be significant to pharmaceutical chemistry, materials science, and industrial chemistry.

Experimental

In a representative experiment, to a 12 mL screw-cap vial was added isoquinoline (0.3 mmol, 3.0 equiv.), $CuCl_2$ (20 mol%), $TsOH{\cdot}H_2O$ (20 mol%) and DMF (1.5 mL). The reaction tube was flushed with molecular oxygen, tightly capped and stirred at room temperature for 10 min. Then, 2-aminobenzyl alcohol (0.1 mmol, 1.0 equiv.) was added in three portions and the resulting mixture was stirred at 100 °C for 12 h. Upon completion of the reaction, the mixture was cooled to room temperature and diphenyl ether (17.0 mg, 0.1 mmol) as an internal standard was added. The organic components were subsequently extracted into ethyl acetate (2.0 mL), washed with $NaHCO_3$ solution (5% in water, 1.0 mL) and brine (1.0 mL), dried over anhydrous Na_2SO_4, and analyzed by GC with reference to diphenyl ether. To isolate the pyrido-fused quinazolinone product, the combined organic extracts were concentrated *in vacuo* and purified by column chromatography on silica gel with a hexane/ethyl acetate solvent system to afford the pure product. The product identity was further confirmed by GC-MS, ^{1}H NMR and ^{13}C NMR.

Conflicts of interest

There are no conflicts to declare.

Acknowledgements

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