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Iodine-catalyzed oxidative system for cyclization of primary alcohols with *o*-aminobenzamides to quinazolinones using DMSO as the oxidant in dimethyl carbonate†

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The iodine catalyzed one-pot two-step oxidative system for cyclization of primary alcohols with *o*-aminobenzamides to quinazolinones using DMSO as the oxidant has been achieved, providing a convenient and efficient method for the synthesis of quinazolinones in good to excellent yields *via in situ* oxidation of primary alcohols to aldehydes. The reaction was carried out in the green solvent DMC, under atmospheric conditions. The procedure is suitable for aromatic or alkyl primary alcohols.

Introduction

Quinazoline and its derivatives exist in a variety of natural products and synthetic drugs[1] and play a significant role in medicinal chemistry for their biological activity, such as hypolipidemic,[2] anti-inflammatory,[3] anti-convulsant,[4] anti-ulcer,[5] and anti-cancer.[6] Some quinazolinones are currently attracting considerable attention due to their therapeutic value in the treatment of tuberculosis.[7]

There has been a long-standing interest in the development of new methods for the synthesis of quinazolinones due to their important applications.[8] A number of different strategies have been devised for condensation between aldehydes and *o*-aminobenzamides followed by oxidation of the aminal intermediate.[9] Stoichiometric or large excess amounts of oxidants, such as $KMnO_4$,[9a] $CuCl_2$,[9b] I_2,[9c] DDQ,[9d] and MnO_2,[9e] were required for this oxidation.

Due to copper-catalyzed Ullmann-type couplings making great achievements in recent years,[10] some quinazolinones were synthesized through the couplings between 2-halobenzoic acid derivatives and ammonia sources, including amidines,[10a] benzylamines[10b] and α-amino acids.[10c] Catalytic amounts of metal catalysts and stoichiometric amounts of bases were necessary for the reaction.

In order to explore a direct and convenient synthetic strategy for quinazolinones, chemists paid much attention to palladium-catalyzed carbonylation of aryl halides and C–H bonds, which represented a straightforward approach to carboxylic acids and their derivatives. Nitrogen-containing heterocycles of quinazolinones were synthesized through a palladium-catalyzed intramolecular or intermolecular carboxamidation procedure.[11]

In the past three years, noble metal catalyzed systems for N-alkylation of amides or sulfonamides with various alcohols based on catalytic hydrogen transfer reactions have been developed rapidly,[12] which provided a nice inspiration for chemists to construct N-containing heterocycles. In 2011, J. Zhou and Fang described a one-pot oxidative cyclization of primary alcohols with *o*-aminobenzamides to quinazolinones catalyzed by iridium under hydrogen transfer conditions.[13] Very recently, A. J. A. Watson and co-workers reported ruthenium-catalyzed hydrogen transfer for the conversion of alcohols into either 2,3-dihydro-quinazolines or quinazolines.[14] Both reactions contained the transformation of alcohols to aldehydes catalyzed by Ir or Ru complexes.

In view of the development of green chemistry, the enhancement in people's awareness of environmental protection in recent years and our continuous work on the C–S, C–N, and C–O bond forming reactions,[15] an efficient iodine catalyzed one-pot two-step oxidative system for cyclization of primary alcohols with *o*-aminobenzamides to quinazolinones was developed. The reaction was carried out in an environmentally benign solvent, dimethyl carbonate (DMC), in the presence of a mild oxidant, DMSO, using 5 mol% of iodine as a catalyst and without the need for oxygen and moisture-free reaction conditions (Scheme 1).

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Scheme 1 Molecular iodine catalyzed cyclization of primary alcohols with *o*-aminobenzamides to quinazolinones using DMSO as an oxidant.

Results and discussion

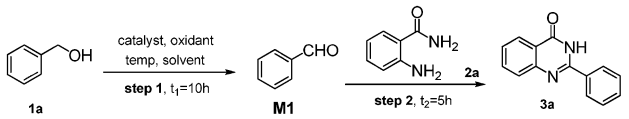
In recent years, molecular iodine has drawn considerable attention as an inexpensive, nontoxic, nonmetallic, and readily available catalyst for effecting various organic transformations.[16] However, to the best of our knowledge, there are no reports on the cyclization of primary alcohols with *o*-aminobenzamides to quinazolinones using an iodine–DMSO oxidative system under green and atmospheric conditions.

We initiated our study by examining the reaction of benzyl alcohol (**1a**) with *o*-aminobenzamide (**2a**) in a one-pot two-step manner. Benzyl alcohol was heated in DMSO at 100 °C for 10 h in the presence of 10 mol% of iodine. Then *o*-aminobenzamide was added to the reaction mixture. We were delighted to find that product **3a** was obtained with a 91% yield after column chromatography (Table 1, entry 1). Encouraged by this result, the cyclization of *o*-aminobenzamide and benzyl alcohol was optimized and the results are summarized in Table 1.

At first, several kinds of solvents were tested under an air atmosphere, a product was not detected without the addition of DMSO (Table 1, entries 2–5). Then 3 equivalents of DMSO were used as an oxidant for the cyclization in DMC and toluene, and yields of 76% and 78% for **3a** were obtained, respectively (Table 1, entries 6 and 7). To avoid the use of environmentally harmful solvents, the green solvent DMC was chosen for subsequent exploitation of optimal experimental conditions. Several oxidants such as O_2, H_2O_2, Oxone and TBHP were then tested for the reaction (Table 1, entries 8–11). It was found that in the presence of oxidants tested except for DMSO, the yield of the product decreased due to the peroxidation of benzyl alcohol to benzoic acid. The dosages of iodine and DMSO were also tested and it was found that 5 mol% iodine gave the highest yield of 93% in the presence of 5 equivalents of DMSO at 100 °C (Table 1, entries 12–16).

Based on this result, other iodine-containing non-metal catalysts such as NIS, TBAI, I_2O_5, and iodobenzene diacetate (DIB), were screened for the reaction. Only NIS showed the same catalytic activity as iodine (Table 1, entries 17 and 19–21). Furthermore, NBS was tested for the reaction, which can also form an oxidative system with DMSO and gave a slightly lower yield of 83% (Table 1, entry 18). The product and intermediate **M1** benzyl aldehyde were not formed without the presence of any catalyst under normal conditions (Table 1, entry 22). It was found that the desired product could also be obtained in a good yield by using 2 equivalents of iodine as an oxidant, which may prove that the aldehyde was formed *via in situ* oxidation of the primary alcohol by iodine (Table 1, entry 23).

Table 1 Optimization of reaction conditions for the synthesis of quinazolinones in one pot[a]



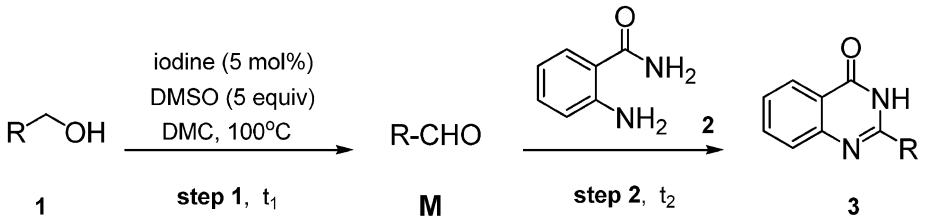
Entry	Catalyst (mmol)	Solvent	Oxidant (equiv.)	T (°C)	Yield[b] (%)
1	I_2	DMSO		100	91
2	I_2	DMF		100	0
3	I_2	DCE		100	0
4	I_2	Toluene		100	0
5	I_2	DMC		100	0
6	I_2	DMC	DMSO (3)	100	76
7	I_2	Toluene	DMSO (3)	100	78
8	I_2	DMC	O_2	100	Trace
9	I_2	DMC	H_2O_2 (3)	100	42
10	I_2	DMC	Oxone (3)	100	46
11	I_2	DMC	TBHP (3)	100	60
12	I_2	DMC	DMSO (4)	100	84
13	I_2	DMC	DMSO (5)	100	93
14[c]	I_2	DMC	DMSO (5)	100	93
15[d]	I_2	DMC	DMSO (5)	100	81
16[c]	I_2	DMC	DMSO (5)	80	85
17[c]	NIS	DMC	DMSO (5)	100	90
18	NBS	DMC	DMSO (5)	100	83
19	TBAI	DMC	DMSO (5)	100	0
20	I_2O_5	DMC	DMSO (5)	100	0
21	DIB	DMC	DMSO (5)	100	0
22		DMC	DMSO (5)	100	0
23	I_2 (2)	DMC	—	100	83

[a] Reaction conditions: **2a** (1 mmol), **1a** (1.2 mmol), catalyst (10 mol%), solvent (1 ml), in a sealed tube under an air atmosphere. [b] Yield of isolated product after column chromatography based on **2a**. [c] Iodine (5 mol%) was used. [d] Iodine (3 mol%) was used.

Having optimized the reaction conditions, a variety of primary alcohols were tested to determine the scope and limitations of the method. Results listed in Table 2 demonstrate that most of the primary alcohols **1** tested underwent smooth transformation to afford the corresponding quinazolinone **3** in high yields. In general, benzylic alcohols bearing electron-donating groups on the benzene ring were more reactive than those with electron-withdrawing groups (Table 2, entries 1–11). Heteroaryl primary alcohols proceeded well for the present procedure (Table 2, entries 12 and 13). We are delighted to disclose that satisfactory yields were also obtained from cyclization of *o*-aminobenzamide with cinnamic alcohol and phenethyl alcohol (Table 2, entries 11 and 14). The alkyl primary alcohol, butyl alcohol, could also be transformed to the desired product with 10 mol% PTSA as an additive (Table 2, entry 15).

To explore the catalytic mechanism, a series of blank and parallel experiments were tested for the cyclization of *o*-aminobenzamide and benzyl aldehyde **M1**, and the results are listed in Table 3. It was found that without the presence of DMSO, the use of 10 mol% of iodine as a catalyst leads to the cyclization product in a low yield (16%) (Table 3, entry 1). In

Table 2 Iodine catalyzed cyclization of primary alcohols with *o*-aminobenzamides to quinazolinones under the optimum conditions[a]



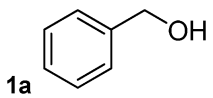
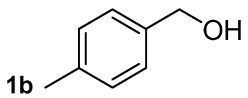
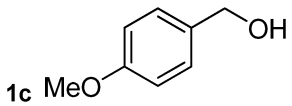
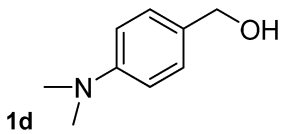
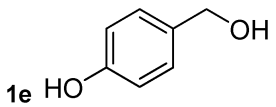
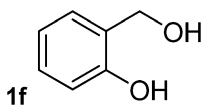
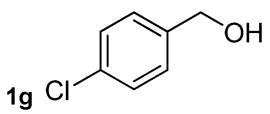
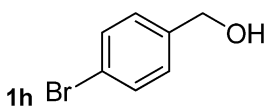
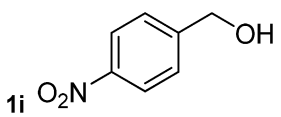
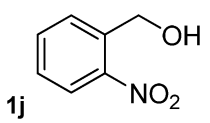
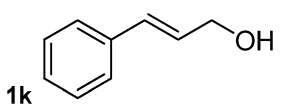
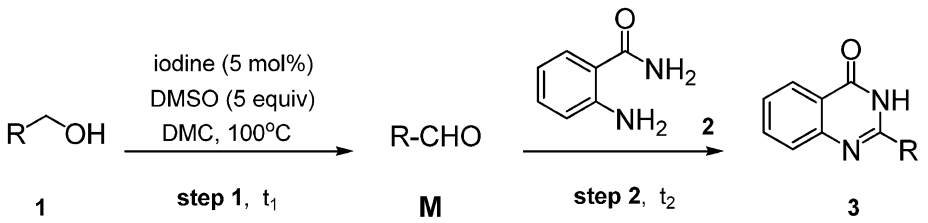
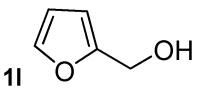
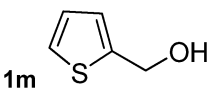
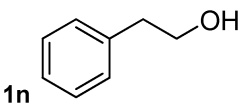
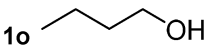
Entry	RCH_2OH	t_1 (h)	t_2 (h)	Yield[b] (%)
1	 **1a**	10	5	93
2	 **1b**	10	5	93
3	 **1c**	7	5	95
4	 **1d**	8	5	94
5	 **1e**	10	5	90
6	 **1f**	10	6	87
7	 **1g**	14	5	90
8	 **1h**	14	5	89
9[c]	 **1i**	18	4	57
10[c]	 **1j**	18	4	55
11[e]	 **1k**	15	6	76

Table 2 (*Continued*)

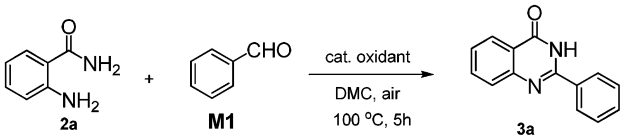


Entry	RCH_2OH	t_1 (h)	t_2 (h)	Yield[b] (%)
12	 1l	12	6	83
13	 1m	18	8	77
14[ce]	 1n	15	8	51
15[d]	 1o	15	10	41

[a] Reaction conditions: 2 (1 mmol), 1 (1.2 mmol), iodine (0.05 mmol, 5 mol%), DMSO (5 mmol) and DMC (1 ml), at 100 °C. [b] Isolated yield based on substrate 2. [c] Iodine (10 mol%) was used. [d] PTSA (10 mol%) was added as an additive. [e] Under a nitrogen atmosphere.

the presence of 10 mol% of HCl, KI can catalyze the reaction with DMSO as an oxidant (Table 3, entry 4). In the absence of HCl or DMSO, no product was detected with KI as a catalyst (Table 3, entries 2 and 3). The reaction also proceeded well in the presence of HI and DMSO without I_2 or KI (Table 3, entry 5). In a recent article, we have demonstrated that DMSO could convert HI to I_2.[15d] These results suggest the essential role of iodine and DMSO for the cyclization step of the present procedure.

Table 3 Cyclization of *o*-aminobenzamide with benzyl aldehyde (step 2)[a]



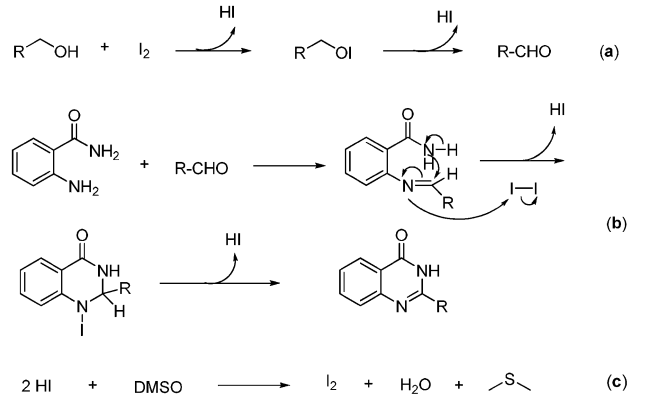
Entry	Catalyst	Acid	Oxidant	Yield[b] (%)
1	I_2			16
2	KI		DMSO	0
3	KI	HCl		0
4	KI	HCl	DMSO	93
5		HI	DMSO	95

[a] Reaction conditions: catalyst (10 mol%), acid (20%, 10 mol%), DMSO (3 equiv.), DMC (2 ml). [b] Yield of isolated product after column chromatography.

Therefore, a plausible reaction mechanism for the iodine catalyzed one-pot two-step oxidative system using DMSO as an oxidant is depicted in Scheme 2. In the first step, the alcohol was oxidized to the corresponding aldehyde with iodine as the oxidant (Scheme 2a). The *in situ* generated aldehyde then reacted with *o*-aminobenzamide to form an imine intermediate. In the presence of iodine, the imine intermediate cyclized to form a N-iodinated species. Elimination of HI produced the final product (Scheme 2b). Iodine can be regenerated in both of the two steps *via* the oxidation of HI with DMSO as the oxidant (Scheme 2c).

Conclusions

In conclusion, a convenient and efficient iodine catalyzed one-pot two-step oxidative system for the synthesis of quinazolinones from the cyclization of primary alcohols with *o*-aminobenzamides using DMSO as the oxidant has been achieved. The present procedure is not moisture sensitive and is carried out in a green solvent, DMC, with high product yields. Moreover, the procedure is suitable for aromatic or alkyl primary alcohols.



Scheme 2 Proposed mechanism for iodine-catalyzed cyclization of a primary alcohol and *o*-aminobenzamide.

Experimental

All chemicals (AR grade) were obtained from commercial sources and were used without further purification. Petroleum ether (PE) refers to the fraction boiling in the 60–90 °C range. The progress of the reactions was monitored by TLC (silica gel, Polygram SILG/UV 254 plates). Column chromatography was performed on Silicycle silica gel (200–300 mesh). Melting points were obtained using Yamato melting point apparatus Model MP-21 and are uncorrected. IR spectra were recorded on a Shimadzu spectrophotometer using KBr discs. ^{1}H and ^{13}C NMR spectra were obtained using a Bruker DRX 500 (500 MHz) spectrometer in $CDCl_3$ or DMSO-d_6 with TMS as the internal standard. All the products are known compounds and they were identified by comparison of their physical and spectral data with those reported in the literature.

A mixture of primary alcohol **1** (1.2 mmol), and DMSO (5 mmol) was dissolved in DMC (1 ml) at 100 °C in a sealed tube, then iodine (0.05 mmol, 5 mol%) was added. The reaction proceeded under an air atmosphere for the indicated time. Then the reaction solution was cooled to room temperature and *o*-aminobenzamide (1 mmol) was added. The reaction proceeded for the indicated time until complete consumption of the starting material, as monitored by TLC, was achieved. The solution was diluted with DMC (5 ml), washed with H_2O (3 × 5 mL), and then the organic layer was separated and concentrated under vacuum and the crude product was purified by column chromatography (PE : EtOAc, 1 : 1) or recrystallization (PE : EtOAc, 3 : 1) to provide the analytically pure product **3**.

Acknowledgements

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