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One-pot $B(C_6F_5)_3$ catalyzed cascade synthesis of 2-substituted-2,3-dihydroquinazolin-4(1*H*)-ones

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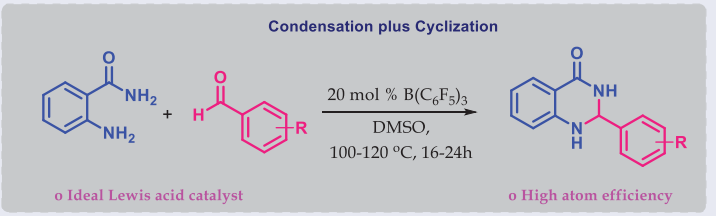
ABSTRACT

A new and efficient $B(C_6F_5)_3$ catalyzed domino strategy has been developed for the synthesis of 2-substituted quinazolinones. The reaction utilizes 2-aminobenzamide and aldehydes for a one-pot protocol. A wide range of substrate scope, functional group tolerance, and operational simplicity with excellent yield are synthetically useful features.

GRAPHICAL ABSTRACT



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KEYWORDS
Aldehyde; 2-aminobenzamide; quinazolinone; tris(pentafluorophenyl)borane

Introduction

Quinazolin-4-(3*H*)-one is a main structural unit found in several natural products and biologically potent compounds. Quinazolinones are considered as privileged structures due to their immanent nature and their function as pharmacophores in drugs.[1] Quinazolinones are known for their several pharmacological and biological activities including antimalarial,[2] anticancer,[3] anti-inflammatory,[4] antihypertensive,[5] and antituberculosis activities.[6] Owing to their notable significance, huge efforts have been made toward efficient and convenient strategies for the construction of the quinazoline skeleton from 2-aminobenzamide and aldehydes.[7–12] However, these strategies suffer from certain drawbacks including use of coupling agents/bases, use of heavy and expensive metal catalysts, harsh reaction conditions, use additives, low yields, stoichiometric or large excess amounts of toxic oxidants. Therefore, search for a simple and efficient cascade protocol to access quinazolinones from 2-aminobenzamide and aldehydes is highly desirable.

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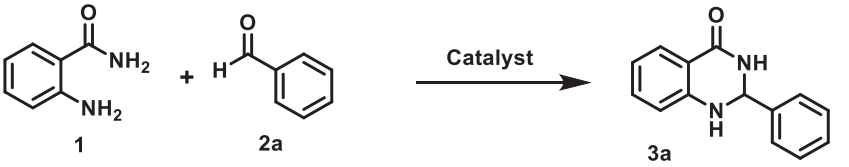
Table 1. Screening of catalyst for the synthesis of **3a**.



Entry	Catalyst (mol%)	Temp (°C)	Yield[a] (%)
1	–	100	00
2	$B(C_6F_5)_3$ (1)	100	20
3	$B(C_6F_5)_3$ (5)	100	25
4	$B(C_6F_5)_3$ (10)	100	45
5	$B(C_6F_5)_3$ (15)	100	60
6	$B(C_6F_5)_3$ (20)	100	80
7	$B(C_6F_5)_3$ (25)	100	80
8	Fe_2O_3 (20)	100	40
9	$ZnCl_2$ (20)	100	49
10	$BiCl_3$ (20)	100	58
11	$FeCl_3$ (20)	100	62

[a]Yield refers to an isolated yield after column chromatography.

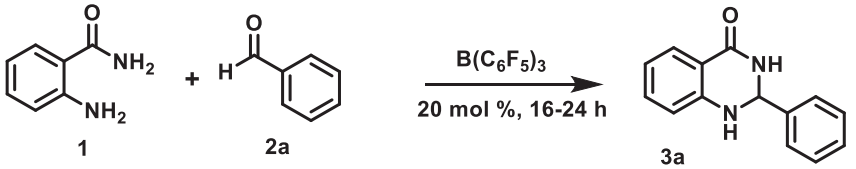
Moderate Lewis acidic nature of Tris(pentafluorophenyl)Borane (BCF) facilitates its usage in organic synthesis. In recent times, BCF has evolved as a mild, nontoxic, environmentally benign, moisture-tolerant, air-stable, heat-stable, inherently electrophilic, moderate and versatile Lewis acid imparting high chemo-, region- and stereoselectivity in many organic transformation.[13] Low catalytic loading and moisture-tolerance, makes BCF superior acid catalyst than traditional Lewis acids. Herein, we report one-pot $B(C_6F_5)_3$ catalyzed convenient synthetic protocol for the synthesis of 2-substituted-quinazolin-4-(3*H*)-ones from cost-effective and easily available 2-aminobenzamide and aldehydes in DMSO as a solvent.

Results and discussion

To demonstrate our methodology, we purchased BCF, substituted aromatic/hetero-aromatic aldehydes and 2-aminobenzamide commercially and used without any purification. Synthesis of quinazolin-4-(3*H*)-ones (**3a–j**) has been achieved from 2-aminobenzamide (**1**), and aldehyde (**2a–j**) in the presence of $B(C_6F_5)_3$ as a solid acid catalyst at 110–120 °C. The structure of products (**3a–j**) was confirmed by comparing spectroscopic data with literature values. To optimize reaction conditions, we initially examined the role of catalyst and screened several solid acid catalysts. 2-aminobenzamide (**1**), and benzaldehyde (**2a**) were selected as model starting materials and the reaction was carried out in DMSO at 100–120 °C using 20 mol % of $B(C_6F_5)_3$ as a catalyst. The progress of the reaction was checked by TLC and after 20 h the product **3a** was obtained in 80% yield (Table 1, entry 6). The formation of compound **3a** was confirmed by comparing ^1H, ^{13}C-NMR and mass spectroscopic data with literature data.

To know the effectiveness of BCF, we repeated the same reaction with 1, 5, 10, 15, 20, and 25 mol % of BCF and we got product **3a** in 80% yield after 20 h when 20 mol % of BCF was used as catalyst (Table 1, entry 6). To realize the efficiency of BCF, same reaction was repeated with different solid acid catalysts such as Fe_3O_4, $ZnCl_2$, $FeCl_3$,

Table 2. Screening of solvent for synthesis of **3a**.



Entry	Solvent	Temp (°C)	Yield[a] (%)
1	DMSO	100	80
2	THF	66	40
3	Toluene	110	28
4	CH_3CN	82	50
5	$CHCl_3$	50	35
6	EtOH	80	39
7	H_2O	100	n.d.
8	–	100	30

[a]Yield refers to an isolated yield after column chromatography.

$BiCl_3$, and $B(C_6F_5)_3$. Among these tested solid acid catalysts, BCF was found as excellent catalysts in terms of reaction time and chemical yield (Table 1, entry 6). To know the effect of solvent on chemical yield of this reaction, we performed same reaction with different solvents such as tetrahydrofuran, toluene, DMSO, acetonitrile, chloroform, ethanol, and water and found that DMSO provides the best results (Table 2, entry 1). Therefore, the use of 20 mol% of $B(C_6F_5)_3$ in DMSO is superior for this conversion.

Under the optimized reaction conditions, we synthesized several quinazolin-4-(3*H*)-ones (**3a–j**) in 62–92% yield. All mentioned reactions proceeded smoothly to give corresponding product in excellent yields and accommodated multifunctional aldehydes also. However, sterically hindered aldehyde (Table 3, entry 3e) gave poor yields in longer reaction times than those of unhindered aldehydes.

Based on the above results and literature reports,[14] a plausible mechanism for the synthesis of 2-substituted-2,3-dihydroquinazolin-4(1*H*)-ones is illustrated in Scheme 1. $B(C_6F_5)_3$ coordinates with the carbonyl oxygen of aldehyde and makes it more susceptible towards the nucleophilic attack by amino group of 2-aminobenzamide giving imine. Imine cyclizes in the presence of $B(C_6F_5)_3$ to give the final product (**3a–j**).

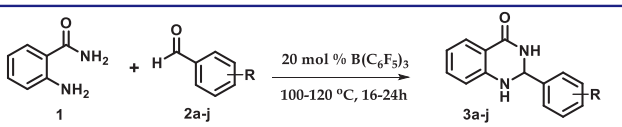
Antioxidant and antibacterial activity

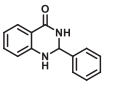
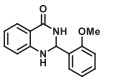
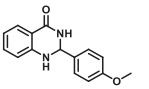
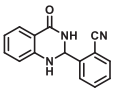
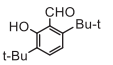
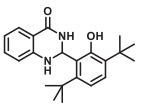
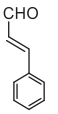
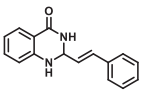
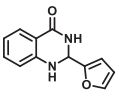
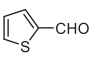
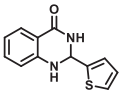
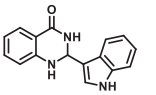
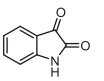
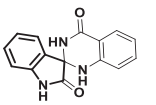
Above prepared 2-substituted-2,3-dihydroquinazolin-4(1*H*)-ones (**3a–j**) were evaluated for their antioxidant (DPPH and hydroxy radical scavenging assay) and antibacterial potency against *Escherichia coli* and *Staphylococcus aureus* and the results are shown in Table 4. Among these compounds, **3i** and **3j** depicted excellent antioxidant activity while compounds **3c** and **3h** effectively inhibited *S. Aureus* and *E. coli* respectively.

Experimental

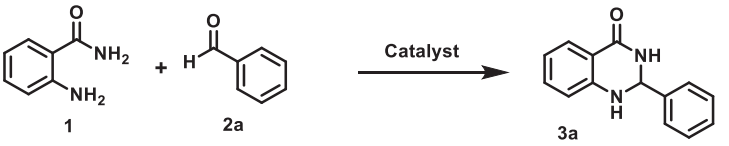
General procedure for the synthesis of 2-phenyl-2,3-dihydroquinazolin-4(1*H*)-one (3a): A mixture of 2-aminobenzamide (**1**) (0.136 mg, 1 mmol), $B(C_6H_5)_3$ (20 mol %), and benzaldehyde (**2a**) (0.60 mg, 5 mmol) in DMSO (2 − 4 mL) was heated at

Table 3. Synthesis of 2-substituted quinazolin-4-(3*H*)-ones (**3a–j**) under $B(C_6F_5)_3$ catalysis.

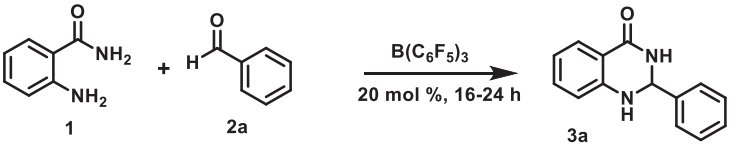


Entry	Aldehyde	Product	Time (h)	Yield[a] (%)
3a			20	80
3b			18	85
3c			19	82
3d			17	90
3e			24	62
3f			22	72
3g			16	92
3h			21	76
3i			23	70
3j			21	75

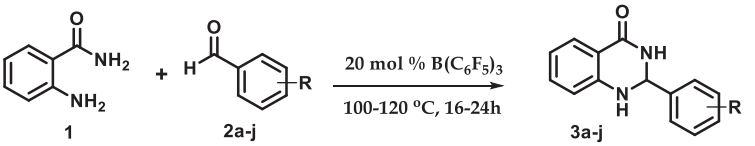
[a]Yield refers to an isolated yield after column chromatography.



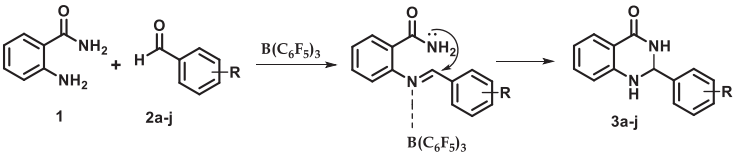
Screening of catalyst for synthesis of **3a**.



Screening of solvent for synthesis of **3a**.



Synthesis of 2-substitutedquinazolin-4-(3H)-ones (**3a-j**) under $B(C_6F_5)_3$ catalysis



Scheme 1. Plausible mechanism for formation of **3a–j.**

$100-120\,^{\circ}C$ for $16-24\,h$ in a sealed tube. After cooling, the reaction mixture was poured into water and extracted with EtOAc. The organic layer was washed with brine, dried over $MgSO_4$, and concentrated in vacuum. The residue was purified by flash column chromatography (silica gel, hexanes/EtOAc) to give desired product. The R_f value for this compound is 0.5. White solid (90 mg (80%) yield), m.p. 100–102 °C TLC $R_f =$ 0.5 (EtOAc:Hexane = 2:8); IR ν_{max} (KBr, cm^{-1}): 1620, 1662, 2990, 3165, 3190, 3320; ^1H NMR (400 MHz, $CDCl_3$ and DMSO-d_6): δ 5.77 (1H, s), 6.74 (2H, t), 7.12 (1H, s), 7.25–7.40 (4H, t), 7.50–7.64 (3H, dd), 8.30 (1H, s); ^{13}CMR (100 MHz, $CDCl_3$ and DMSO-d_6): δ 67.06, 114.89, 115.44, 117.60, 127.35,127.84, 128.81, 128.94, 133.79, 142.11, 148.35; MS (ESI): m/z 225 $[M+H]^+$; Anal. Calcd for $C_{14}H_{12}N_2O$: C, 74.98%, H, 5.39%, N, 12.49%; Found: C, 74.87%, H, 5.26%, N, 12.34% (Supplementary material).

2-(4-Methoxyphenyl)-2,3-dihydroquinazolin-4(1*H*)-one (3c): White solid (93 mg (82%) yield), m.p. 125–127 °C, TLC $R_f = 0.6$ (EtOAc: Hexane = 2:8); IR ν_{max} (KBr, cm^{-1}): 1059, 1475, 1600, 1680, 2925, 3190, 3310, 3445; ^1H NMR (400 MHz, $CDCl_3$ and DMSO-d_6): δ 3.76 (3H, s), 5.71 (1H, s), 6.64–6.92 (5H, m) , 7.19–7.22 (1H, t), 7.42–7.45 (2H, d) , 7.63–7.65 (1H, d), 8.06 (1H, s); ^{13}CNMR (100 MHz, $CDCl_3$ and DMSO-d_6): δ 55.48, 67.10, 113.91, 114.79, 114.82, 117.57, 127.75,128.67, 133.47, 133.57,148.45, 159.98,

Table 4. Antioxidant and antibacterial potency of compounds (**3a–j**).

		Antimicrobial activity (mm)		Antioxidant activity	
Entry	Compound	*E. coli*	*S. aureus*	DPPH	OH
1	**3a**	–	–	09.25 ± 0.85	08.17 ± 0.87
2	**3b**	02	02	19.01 ± 0.71	3.39 ± 0.77
3	**3c**	03	04	11.25 ± 0.69	19.89 ± 0.15
4	**3d**	–	–	10.99 ± 0.25	19.89 ± 0.15
5	**3e**	04	–	02.87 ± 0.65	33.25 ± 0.96
6	**3f**	–	–	07.29 ± 0.67	0.98 ± 0.01
7	**3g**	06	–	25.67 ± 0.44	3.71 ± 0.65
8	**3h**	08	03	0.87 ± 0.45	14.12 ± 0.15
9	**3i**	–	–	36.87 ± 0.87	27.19 ± 0.34
10	**3j**	–	–	41.25 ± 0.98	26.87 ± 0.89

164.36; MS (ESI): *m/z* 255 $[M+H]^+$; Anal. Calcd for $C_{15}H_{14}N_2O_2$: C, 70.85%, H, 5.55%, N, 11.02%; Found: C, 70.62%, H, 5.44%, N, 10.94%.

Conclusion

In conclusion, we have developed an BCF catalyzed direct one pot, simple, efficient, synthetic protocol for the synthesis of 2-substituted-2,3-dihydroquinazolin-4(1*H*)-ones from 2-aminobenzamide and aldehyde in good to excellent yields. High yields, easy workup, high atom-economy, low cost and easy handling of the catalyst are important highlights of this protocol.

Acknowledgments

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