Efficient synthesis of mono- and disubstituted 2,3-dihydroquinazolin-4(1*H*)-ones using aluminum methanesulfonate as a reusable catalyst

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Abstract A wide range of mono- and disubstituted 2,3-dihydroquinazolin-4(1*H*)ones were synthesized via a one-pot three components condensation of isatoic anhydride, aldehydes, and ammonium salts or primary amines in the presence of aluminum methanesulfonate in EtOH/H₂O solution. The catalyst is reusable and could be recycled for several runs without any distinct decrease in its efficiency. A plausible mechanism for this one-pot three components reaction was proposed.

Keywords 2,3-Dihydroquinazolin-4(1H)-ones · Aluminum methanesulfonate · Multicomponent reactions · Isatoic anhydride

Introduction

2,3-Dihydroquinazolinone derivatives have been reported to possess a wide range of pharmacological activities such as anti-inflammatory and analgesic activities [1], and antitumor [2], anticancer [3], antibacterial [4], and diuretic activities [5]. In addition, these compounds as chemical intermediates can easily be oxidized to their corresponding quinazolin-4(3H)-ones [6], which are also important biologically active heterocyclic compounds [7, 8].

Several methods have been reported for the synthesis of 2,3-dihydroquinazolinones. Among them, the most direct procedure includes condensation of the appropriate derivatives of anthranilamide with an aldehyde or ketone in the presence of Brønsted acid or Lewis acid [9–15]. However, these methods suffer from low

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yields and/or vigorous conditions. The structures of products are limited. Other synthetic methods for access to this bicyclic system are not general in scope [16, 17]. Therefore, simple, efficient and environmentally benign procedures for synthesizing 2,3-dihydroquinazolin-4(1H)-ones are still in demand.

One-pot multi-component reactions (MCRs) are very important and attractive subjects in organic synthesis due to the formation of carbon-carbon and carbonhetero atom bonds. They are performed without needing to isolate any intermediate during their process, which saves time, energy and raw materials. Very recently, Dabiri and Salehi first reported that 2,3-dihydroquinazolin-4(1H)-ones could be synthesized from a novel one-pot three components condensation of isatoic anhydride, an aromatic aldehyde, and a primary amine or ammonium salts [18, 19]. Since then, some other catalysts such as p-TsOH [20], acetic acid [21], Zn(PFO)₂ [22], Ga(OTf)₃ [23], ionic liquid [24, 25], montmorillonite K-10 [26, 27], Al(H₂PO₄)₃ [28], I₂ [29, 30], amberlyst-15 [31], silica sulfuric acid [32], silica-bonded S-sulfonic acid [33], Al/Al₂O₃ and Fe₃O₄ nanoparticles [34, 35], copolymer-PTSA [36], MCM-41-SO₃H [37], SiO₂-FeCl₃ [38] and silica-bonded *N*-propylsulfamic acid [39] have also been reported. However, these methodologies suffer from at least one of the following disadvantages: long reaction time, high temperature, and costly reagents. Now, during our continuous study on green and efficient MCRs, we report here a one-pot three components synthesis of 2,3-dihydroquinazolin-4(1H)-ones from isatoic anhydride, aldehydes, and ammonium salts or primary amines in the presence of 5 mol% (based on aldehyde) aluminum methanesulfonate (Al(MS)₃·4H₂O) in EtOH/H₂O solution (Scheme 1). Metal methanesulfonates $(M(MS)_x \cdot aH_2O)$ as water-tolerant Lewis acid catalysts have been well documented in recent years [40-45]. The new synthetic protocol enlarges the application scope of metal methanesulfonates in organic synthesis.



Scheme 1 Al(MS)3·4H2O-catalyzed synthesis of mono- and disubstituted 2,3-dihydroquinazolin-4(1*H*)- ones in ethanol aqueous solution

Experimental

General

Melting points were determined using a RD-II micromelting point apparatus. Infrared spectra were recorded on a Scimitar 2000 series Fourier Transform instrument of VARIAN (KBr diluted pellets). ¹H NMR and ¹³C NMR spectra were recorded on a Bruker ARX-500 spectrometer in DMSO- d_6 using TMS as an internal standard. Mass spectra were obtained with an Agilent 1100 series LC/MSD VL ESI instrument. Elemental analyses were carried out on an EA 2400II elemental analyzer (PerkinElmer).

Synthesis and characterization of $M(MS)_x \cdot aH_2O$

 $M(MS)_x \cdot aH_2O$ were synthesized according to Ref. [41]. They were characterized by IR and Thermogravimetry (TG) analysis. Taking Al(MS)₃·4H₂O as an example, IR data: v_{as} (SO₃) 1,189 cm⁻¹, v_s (SO₃) 1,061 cm⁻¹, v_{as} (CH₃) 2,941 cm⁻¹, δ_s (CH₃) 1,384 cm⁻¹. TG curves were monitored on a Perkin-Elmer Pyris 1 TGA (sample mass is about 2.5 mg). Experiments were conducted in dynamic Air atmosphere (20 mL/min) at a heating rate of 20 °C min⁻¹ from 30 to 850 °C. Crystal water's weight losing ratio is 17.9%, number of crystal water is 4, starting decomposition temperature is 200 °C, and weight content of pyrolytic product is 13.2%.

General procedure for preparation of 2,3-dihydroquinazolin-4(1*H*)-ones catalyzed by $Al(MS)_3 \cdot 4H_2O$

A stirred mixture of isatoic anhydride (0.915 g, 5.5 mmol), aldehyde (5 mmol), ammonium salt or primary amine (5.5 mmol) and $Al(MS)_3 \cdot 4H_2O$ (0.096 g, 0.25 mmol) in 5 mL EtOH/H₂O (1/1 (v/v)) was refluxed for an appropriate time as indicated in Tables 3 and 4. When the reaction completed as monitored by TLC, the reaction mixture was cooled to room temperature. The corresponding pure product was obtained by simple filtering, washed with 3 × 10 mL 50% ethanol aqueous solution for three times, and recrystallized from EtOH. The filtrate containing the catalyst was evaporated under reduced pressure to give the recovered catalyst, which could be reused without further treatment. The products were characterized by IR, ¹H NMR, LC/MS, and elemental analysis. Spectral data for some new compounds:

2,3-Dihydro-2-(2-chlorophenyl)-3-phenyl-quinazolin-4(1*H*)-one (**6c**): white crystal, IR (KBr): 3,309, 3,070, 2,361, 1,637, 1,606, 1,491, 1,452, 758 cm⁻¹; ¹H NMR (500 MHz, DMSO- d_6): δ 7.77 (dd, J = 1.3, 6.4 Hz, 1H), 7.60–7.58 (m, 1H), 7.46 (d, J = 2.5 Hz, 1H), 7.39–7.37 (m, 1H), 7.31–7.27 (m, 5H), 7.21–7.17 (m, 3H), 6.79–6.74 (m, 2H), 6.60 (d, J = 2.7 Hz, 1H); ¹³C NMR (125 MHz, DMSO- d_6): δ 162.2, 146.0, 140.1, 136.9, 133.8, 131.3, 130.3, 129.8, 128.6, 128.2, 127.8, 127.4, 126.9, 126.6, 117.6, 114.8, 114.6, 70.2; MS (ESI): m/z (%) 335 (M + H⁺, 100), 336 (23), 337 (36), 338 (8), 315 (6); Anal. Calcd. for C₂₀H₁₅N₂OCl: C, 71.74; H, 4.52; N, 8.37. Found: C, 71.59; H, 4.61; N, 8.46.

2,3-Dihydro-2-(4-nitrophenyl)-3-methyl-quinazolin-4(1*H*)-one (**6k**): yellow crystal, IR (KBr): 3,309, 2,360, 1,644, 1,599, 1,521, 1,457, 1,407, 763 cm⁻¹; ¹H NMR (500 MHz, DMSO-*d*₆): δ 8.74 (s, 1H), 8.37 (d, *J* = 8.5 Hz, 3H), 8.20 (d, *J* = 8.6 Hz, 2H), 7.76 (d, *J* = 6.9 Hz, 1H), 7.53 (t, *J* = 6.9 Hz, 1H), 7.35 (t, *J* = 7.4 Hz, 1H), 7.27 (d, *J* = 7.8 Hz, 1H), 2.80 (d, *J* = 4.6 Hz, 3H); ¹³C NMR (125 MHz, DMSO-*d*₆): δ 160.2, 149.0, 148.2, 141.1, 131.2, 129.9, 129.7, 129.2, 126.5, 124.0, 119.0, 70.1, 26.1; MS (ESI): *m*/*z* (%) 284 (M + H⁺, 100), 282 (22); Anal. Calcd. for C₁₅H₁₃N₃O₃: C, 63.60; H, 4.62; N, 14.83. Found: C, 63.78; H, 4.70; N, 14.69.

2,3-Dihydro-2-phenyl-3-n-propyl-quinazolin-4(1*H*)-one (**6p**): white crystal, IR (KBr): 3,303, 3,065, 2,361, 1,630, 1,588, 1,507, 1,458, 748 cm⁻¹; ¹H NMR (500 MHz, DMSO-*d*₆): δ 7.65 (dd, *J* = 1.2, 6.5 Hz, 1H), 7.34–7.27 (m, 6H), 7.18 (dt, *J* = 1.5, 6.8 Hz, 1H), 6.66–6.62 (m, 2H), 5.83 (d, *J* = 2.5 Hz, 1H), 3.86–3.81 (m, 1H), 2.75–2.69 (m, 1H), 1.63–1.41 (m, 2H), 0.82 (t, *J* = 7.4 Hz, 3H); ¹³C NMR (125 MHz, DMSO-*d*₆): δ 162.1, 146.2, 141.2, 133.0, 128.4, 128.2, 127.3, 126.0, 117.0, 115.0, 114.2, 70.1, 46.0, 20.7, 11.1; MS (ESI): *m/z* (%) 267 (M + H⁺, 100), 268 (19); Anal. Calcd. for C₁₇H₁₈N₂O: C, 76.66; H, 6.82; N, 10.52. Found: C, 76.81; H, 6.74; N, 10.41.

2,3-Dihydro-2-phenyl-3-benzyl-quinazolin-4(1*H*)-one (**6r**): white crystal, IR (KBr): 3,401, 3,023, 2,361, 1,633, 1,579, 1,502, 1,447, 749 cm⁻¹; ¹H NMR (500 MHz, DMSO- d_6): δ 7.70 (dd, J = 1.4, 6.4 Hz, 1H), 7.36–7.20 (m, 12H), 6.69–6.63 (m, 2H), 5.74 (d, J = 2.6 Hz, 1H), 5.34 (d, J = 15.4 Hz, 1H); 3.83 (d, J = 15.4 Hz, 1H); ¹³C NMR (125 MHz, DMSO- d_6): δ 162.3, 146.2, 140.5, 137.4, 133.3, 128.5, 128.4, 127.6, 127.4, 127.1, 126.1, 117.2, 114.6, 114.3, 69.8, 47.1; MS (ESI): m/z (%) 315 (M + H⁺, 100), 316 (24); Anal. Calcd. for C₂₁H₁₈N₂O: C, 80.22; H, 5.78; N, 8.91. Found: C, 80.39; H, 5.67; N, 8.99.

Results and discussion

At the outset, we investigated the catalytic activity of different $M(MS)_x \cdot aH_2O$ (0.25 mmol) using the model reaction of isatoic anhydride (5.5 mmol), benzaldehyde (5 mmol) and ammonium acetate (5.5 mmol) in EtOH (5 mL) under reflux with vigorous stirring for 2 h. The results are summarized in Table 1. After several trials, it was found that the catalytic activity of Al(MS)₃·4H₂O was similar to that of transition metal methanesulfonates, rare earth metal methanesulfonates, alkaline earth metal methanesulfonates, and alkali metal methanesulfonate. They all gave good yields, which were above 70%. We chose Al(MS)₃·4H₂O as the most promising catalyst because it is economical and it gave a higher yield. When no catalyst was used, a low yield of the product was produced (Table 1, entry 18).

Next, we examined the above reaction in different solvents under reflux for 2 h (Table 2). Only H_2O and EtOH, the clean solvent and relatively benign organic solvent, were investigated to standardize the reaction condition (Table 2, entries 1–5). It seems that EtOH/H₂O (1/1 (v/v)) system is the best for the catalytic reactions in terms of yield, environmental protection, and product isolation (Table 2, entry 4).

O O O O O O O O O O O O O O O O O O O	PhCHO + NH4OAc —	$\frac{M(MS)_x \cdot aH_2O}{EtOH, reflux}$	• NH NH H Ph
Entry	$M(MS)_x \cdot aH_2O$		Yield (%)
1	Al(MS) ₃ ·4H ₂ O		88
2	Fe(MS) ₂ ·4H ₂ O		87
3	Cu(MS)2·4H2O		85
4	$Cd(MS)_2 \cdot 2H_2O$		80
5	Zn(MS) ₂ ·4H ₂ O		80
6	$Mn(MS)_2 \cdot 2H_2O$		79
7	Ni(MS) ₂ ·4H ₂ O		70
8	Ce(MS) ₃ ·2H ₂ O		83
9	La(MS) ₃ ·2H ₂ O		82
10	Pr(MS) ₃ ·2H ₂ O		80
11	Yb(MS) ₃ ·3H ₂ O		76
12	$Nd(MS)_3 \cdot 2H_2O$		71
13	$Sr(MS)_2 \cdot 3H_2O$		81
14	Ba(MS) ₂		78
15	Ca(MS) ₂		76
16	$Mg(MS)_2 \cdot 2H_2O$		74
17	Li(MS)·2H ₂ O		81
18	None		39

Table 1 Catalytic activity of $M(MS)_x \cdot aH_2O$ in the reaction of isatoic anhydride, benzaldehyde, and ammonium acetate

After reaction, the resulted mixture was washed with ethanol aqueous solution. The catalyst remaining in the aqueous phase could be recovered by evaporating the filtrate. $Al(MS)_3 \cdot 4H_2O$ could be reused several times without further treatment. In order to demonstrate the water-tolerant ability of $Al(MS)_3 \cdot 4H_2O$, the recovered catalyst was washed with acetone and tested by IR and TG. The results showed that the spectra data of the recovered catalyst were nearly consistent with that of the fresh catalyst. The results are indicated in Table 2, entry 4. In this case, no distinct loss of activity was observed after four successive runs.

The optimal amount of solvent was also investigated (Table 2, entries 4, 6 and 7). The results show 5 mL EtOH/H₂O (1/1; v/v) was suitable to facilitate the reaction. Further studies showed that the amount of $Al(MS)_3 \cdot 4H_2O$ affected the reaction significantly (Table 2, entries 4, 8–10). The product yields enhanced along with the increase in the amount of catalyst. A satisfactory yield was achieved in the presence of 5 mol% $Al(MS)_3 \cdot 4H_2O$, while 10 mol% catalyst did not bring an obviously better yield.

	O O O O O O O O	$H_4OAc \longrightarrow$ Al(MS) ₃ •4H ₂ O	O NH NH H Ph
Entry	Solvent (mL)	Amount of catalyst (mol%)	Yield (%)
1	None	5	74 ^a
2	H ₂ O (5.0)	5	65
3	EtOH (5.0)	5	88
4	EtOH $(2.5) + H_2O(2.5)$	5	88, 86, 84, 81 ^b
5	EtOH $(1.5) + H_2O(3.5)$	5	83
6	EtOH $(1.5) + H_2O(1.5)$	5	83
7	EtOH $(4.0) + H_2O(4.0)$	5	72
8	EtOH $(2.5) + H_2O(2.5)$	1	73
9	EtOH $(2.5) + H_2O(2.5)$	3	80
10	EtOH (2.5) + H_2O (2.5)	10	89

 Table 2
 Condensation of isatoic anhydride, benzaldehyde, and ammonium acetate under various reaction conditions

^a Yield refers to without solvent at 80 °C

^b Catalyst was reused four times

With the optimized conditions in hand, we explored the scope of the one-pot three components cyclocondensation reaction. Ammonium carbonate and ammonium acetate were employed as the source of ammonia, which was mixed with isatoic anhydride and aldehydes in the presence of $Al(MS)_3$ ·4H₂O to give the corresponding 2-substituted-2,3-dihydroquinazolin-4(1*H*)-ones under the conditions (Table 3). Whether the aromatic ring was bearing electron-withdrawing or electrondonating groups, the corresponding 2-aryl-2,3-dihydroquinazolin-4(1*H*)-ones derivatives were obtained in good yields. However, no desired products were produced when NH₄Cl was used. In the case of aliphatic aldehyde such as propionaldehyde, a trace of product was yielded under these conditions (Table 3, entry 10).

On the other hand, we investigated the synthesis of 2,3-disubstituted-2,3dihydroquinazolin-4(1H)-ones from isatoic anhydride, aldehydes and primary amines under the optimized conditions (Table 4). All the reactions proceeded smoothly. Aromatic aldehydes carrying either electron-withdrawing or electrondonating groups afforded the desired products in moderate to high yields. However, only trace products were isolated when propionaldehyde and cinnamaldehyde were used (Table 4, entries 6 and 7). Aromatic and aliphatic amines worked well under the reaction conditions.

A plausible mechanism for this one-pot three components reaction was proposed. The first step may involve the condensation of isatoic anhydride activated by Al(MS)₃·4H₂O with ammonium salts/primary amine, then followed by decarboxylation to yield the corresponding 2-aminobenzamide. Next step, imine intermediate

	O N H O +	(1 R ₁ CHO +	NH ₄) ₂ CO ₃ or - NH ₄ OAc	Al(MS) ₃ •4 EtOF	¹ H ₂ O (5 mol% ¹ /H ₂ O (1/1)	⁽⁶⁾	NH NH NH R1
	1	2					3a-3j
Entry	R ₁	Ammonium sal	t Time (h)	Product	Yield (%)	Mp (°C)	
						Found	Lit.
1	C ₆ H ₅	(NH ₄) ₂ CO ₃	1.0	3a	91	224–226	218–220 [26]
2	$3-O_2NC_6H_4$	$(NH_4)_2CO_3$	1.0	3b	93	190–192	190–192 [46]
3	$4-O_2NC_6H_4$	$(NH_4)_2CO_3$	4.5	3c	94	198–200	198–200 [46]
4	$2-ClC_6H_4$	$(NH_4)_2CO_3$	0.5	3d	90	203-205	203–205 [46]
5	$4-ClC_6H_4$	$(NH_4)_2CO_3$	0.5	3e	88	210-211	205–206 [23]
6	2,4-Cl ₂ C ₆ H ₃	$(NH_4)_2CO_3$	3.0	3f	95	165–167	167–169 [<mark>46</mark>]
7	4-MeC ₆ H ₄	$(NH_4)_2CO_3$	1.0	3g	82	221-222	225-227 [10]
8	4-MeOC ₆ H ₄	$(NH_4)_2CO_3$	1.0	3h	91	182-184	178–180 [26]
9	4-(Me) ₂ NC ₆ H ₄	$(NH_4)_2CO_3$	1.0	3i	80	208-210	206–208 [46]
10	CH ₃ CH ₂	$(NH_4)_2CO_3$	4.0	3ј	Trace	-	-
11	C ₆ H ₅	NH ₄ OAc	2.0	3a	88	224-226	218–220 [<mark>26</mark>]
12	3-O2NC6H4	NH ₄ OAc	5.0	3b	93	190–192	190–192 [<mark>46</mark>]
13	$4-O_2NC_6H_4$	NH ₄ OAc	6.5	3c	95	198-200	198–200 [<mark>46</mark>]
14	2-ClC ₆ H ₄	NH ₄ OAc	2.0	3d	96	202-203	203-205 [46]
15	4-ClC ₆ H ₄	NH ₄ OAc	1.0	3e	80	210-211	205–206 [23]
16	2,4-Cl ₂ C ₆ H ₃	NH ₄ OAc	6.0	3f	92	165–167	167–169 [<mark>46</mark>]
17	4-MeC ₆ H ₄	NH ₄ OAc	1.0	3g	60	221-222	225–227 [10]
18	4-MeOC ₆ H ₄	NH ₄ OAc	1.0	3h	80	182–184	178–180 [26]
19	$4-(Me)_2NC_6H_4$	NH ₄ OAc	1.5	3i	90	206–208	206–208 [46]

Reaction conditions: isatoic anhydride (5.5 mmol), aromatic aldehydes (5 mmol), ammonium salts (5.5 mmol), Al(MS)₃·4H₂O (5 mol%), EtOH/H₂O (5 mL, 1/1; v/v), under reflux

could be obtained by addition of 2-aminobenzamide with aldehydes promoted by $Al(MS)_3 \cdot 4H_2O$. Subsequently, imine intermediate undergoes cyclization to afford the 2,3-dihydroquinazolin-4(1*H*)-ones. Similar mechanisms of this reaction have been reported and supported before by Dabiri et al. [18] and Wang et al. [22].

Conclusions

In conclusion, we have described an efficient strategy for the synthesis of mono- and disubstituted 2,3-dihydroquinazolin-4(1H)-ones via a one-pot three components

		R ₁ CHO +	R ₂ NH ₂ -	Al(MS)3•4 EtOF	4H ₂ O (5 mol% 4/H ₂ O (1/1)	$\xrightarrow{O}_{N} \xrightarrow{R_2}_{N} \xrightarrow{R_1}_{R_1}$		
	1	4	5				6a-6r	
Entry	R ₁	R ₂	Time (h)	Product	Yield (%)	Mp (°C)		
						Found	Reported	
1	C ₆ H ₅	C ₆ H ₅	1.5	6a	90	216-217	214–215 [23]	
2	$3-O_2NC_6H_4$	C_6H_5	6.0	6b	95	185–186	186–188 [<mark>26</mark>]	
3	$2-ClC_6H_4$	C_6H_5	4.0	6c	93	211-212	_	
4	$4-ClC_6H_4$	C_6H_5	2.0	6d	74	223-225	219–220 [23]	
5	4-MeOC ₆ H ₄	C_6H_5	1.5	6e	82	203-204	204–205 [22]	
6	CH ₃ CH ₂	C_6H_5	6.0	6f	Trace	-	-	
7	C ₆ H ₅ CH=CH	C ₆ H ₅	6.0	6g	Trace	-	-	
8	C ₆ H ₅	4-ClC ₆ H ₄	1.5	6h	60	208-209	210–212 [22]	
9	C ₆ H ₅	$4\text{-}CH_3C_6H_4$	1.5	6i	76	196–197	196–199 [22]	
10	C ₆ H ₅	Me	7.0	6j	54	159–160	165 [<mark>26</mark>]	
11	$4-O_2NC_6H_4$	Me	1.0	6k	87	200-202	_	
12	$4-ClC_6H_4$	Me	7.0	61	33	190–191	190 [<mark>26</mark>]	
13	C ₆ H ₅	Et	6.0	6m	85	130-131	134–137 [<mark>26</mark>]	
14	$3-O_2NC_6H_4$	Et	2.0	6n	73	170-171	176–178 [<mark>26</mark>]	
15	$4-ClC_6H_4$	Et	6.0	60	34	130-132	132–135 [<mark>26</mark>]	
16	C ₆ H ₅	n-Pr	6.5	6р	51	120-121	_	
17	C ₆ H ₅	n-Bu	5.0	6q	92	127-128	120–122 [22]	
18	C ₆ H ₅	$C_6H_5CH_2$	6.0	6r	88	158-159	-	

Table 4	Al(MS)3·4H2O-catalyzed	synthesis of	2,3-disut	ostituted-2,3-d	íhydroquinazo	lin-4(1H)-one	s by
he reaction of isatoic anhydride, aldehydes, and primary amines							

Reaction conditions: isatoic anhydride (5.5 mmol), aldehydes (5 mmol), amines (5.5 mmol), $Al(MS)_3$ ·4H₂O (5 mol%), EtOH/H₂O (5 mL, 1/1; v/v), under reflux

condensation of isatoic anhydride, aldehydes, and ammonium salts or primary amines using the inexpensive, water-tolerant, and recyclable $Al(MS)_3 \cdot 4H_2O$ as a catalyst in EtOH/H₂O solution. The method offers several advantages including mild reaction conditions, simple work-up, easy recovery and reusable catalyst, and extensive application scope. Furthermore, the novel catalyst is expected to contribute the development of a more benign reaction in water or aqueous media, and the related work is underway in our laboratory.

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