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Mohammad Ali Bodaghi Fard^a, Akbar Mobinikhaledi^a & Mahdia Hamidinasab^a

^a Department of Chemistry, Faculty of Sciences , Arak University , Arak , I. R. Iran Accepted author version posted online: 22 Oct 2013.Published online: 21 Nov 2013.



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An Efficient One-Pot Synthesis of 2,3-dihydroquinazolin-4(1H)-ones in Green Media

Mohammad Ali Bodaghi Fard, Akbar Mobinikhaledi, and Mahdia Hamidinasab

Department of Chemistry, Faculty of Sciences, Arak University, Arak, I. R. Iran

A series of mono- and disubstituted 2,3-dihydroquinazoline-4(1H)-ones have been synthesized in good yields by one-pot reaction using isatoic anhydride, ammonium acetate or amines and aldehydes in green and mild reaction conditions. The effects of various parameters such as the solvent, catalyst amount, reaction time, and temperature were examined. The protocol is simple, efficient, and environmentally benign and the catalyst can be recovered and reused without evident loss of reactivity.

Keywords dihydroquinazoline, green synthesis, ionic liquid, TBAB

1. INTRODUCTION

As a consequence of serious pollution problems, the adoption of "cleaner production" methods is an urgent priority.^[1] Organic solvents are volatile, most of them flammable, toxic, and harmful for environment. Thus, removing organic solvents in synthesis is very important in the drive towards environmentally friendly technologies.^[2,3] The potential of water or other nonclassical solvents such as ionic liquids has become highly relevant when designing organic syntheses. The use of these alternative reactions solvents can have inherent advantages such as enhanced rates of reaction or fewer or more readily isolated side products as well as facilitating product recovery.^[2,4] The use of ionic liquids as solvent and catalyst has attracted much attention in the context of green synthesis.^[5] However, the high cost of the most of the conventional room temperature ionic liquids and apprehension regarding the toxicity of some of them have led to the use of more benign salts in the molten state as practical alternatives. Tetra-n-butyl ammonium bromide (TBAB) and related ionic liquids have considerable interests as potential eco-friendly reagents due to their lack of measurable vapor pressure. These liquids are nonvolatile, nonflammable; possess high thermal and chemical stability; possess high ionic conductivity; and dissolve in vast range of organic and inorganic materials.^[6] Recently TBAB has been used as an extremely useful catalyst in various organic transformations.^[3,7] TBAB is also inexpensive, readily available and has inherent properties such as environmental compatibility, greater selectivity, operational simplicity, noncorrosive nature, and ease of reusability.

Mono- and disubstituted 2,3-dihydroquinazoline-4(*1H*)ones have a wide range of biological and pharmacological activities such as analgesic, antitumor, anticancer, diuretic and herbicide activities.^{[8–10}] In addition, these compounds can be easily oxidized to their quinazolin-4(*3H*)-one analogues,^[11] which also include important pharmacologically active compounds.^[12]

Several methods have been reported for the synthesis of 2,3-dihydroquinazolinones.^[13–16] However, these methods suffer from lengthy procedures and/or low yields, use of volatile and harmful solvents, and vigorous reaction conditions.^[13–16] Salehi and coworkers reported a new one-pot synthesis of these compounds using *p*-toluenesolfonic acid, silica sulfuric acid, alum, and montmorillonite K-10.^[17] Very recently, Su and coworkers reported efficient methods for the preparation of quinazolinones.^[18] However, the development of simpler and high yield approaches and milder reaction conditions toward this valuable nucleus is very desirable.

In continuation of our efforts to develop more versatile methodologies for the synthesis of heterocyclic compounds,^[19] herein we report a simple and efficient method for the selective one-pot preparation of mono- and disubstituted 2,3-dihydroquinazolin-4(*1H*)-ones.

EXPERIMENTAL

General

All products are known and characterized by comparison of their physical and spectroscopic data with those reported in literature. Melting points were obtained in open capillaries on an electrothermal 5000 digital apparatus and are not corrected. IR spectra were recorded on a galaxy series FT-IR 5000 spectrometer. NMR spectra were recorded on a Bruker 300 MHz spectrometer in DMSO-d₆ with TMS as an internal standard. Elemental analyses were measured by Vario EL equipment by Arak University. Progresses of the reactions were followed by

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Address correspondence to Mohammad Ali Bodaghi Fard, Department of Chemistry, Faculty of Sciences, Arak University, Arak 38156–879, I. R. Iran. E-mail: mbodaghi2007@yahoo.com



SCH. 1. A: Solvent-free, 105°C (oil bath), 40 mol% TBAB (0/128 g). B: Water, 70°C, 10 mol% TBAB (0/032 g).

dissolving a sample in ethyl acetate and monitoring by TLC using n-Hexane/EtOAc (2:1 v/v) as an eluent.

General Procedure for One-pot Synthesis of 2,3-dihydroquinazolin-4(1H)-ones (Method A)

A well powdered mixture of isatoic anhydride (1 mmol), ammonium acetate (2 mmol) or primary amine (1.2 mmol), aldehyde (1 mmol), and TBAB (0.4 mmol) was added to a screw-capped vial containing magnetic stirring bar and heated at 105°C in a preheated oil bath for appropriate time. After completion of reaction, as indicated by TLC, the mixture was cooled to room temperature and washed with cold water. The solid residue was extracted with ethanol or acetone (2×10 mL). The further purified products were obtained by recrystalization from ethanol-water (4:1).

General Procedure for One-pot Synthesis of 2,3-dihydroquinazolin-4(1H)-ones (Method B)

To a mixture of isatoic anhydrides (1 mmol), ammonium acetate (2 mmol) or primary amine (1.2 mmol) and aldehyde

TABLE 1 Optimization of reaction condition

Entry	Solvent	Catalyst	Temperature (°C)	Yield (%) ^a
1	Solvent-free	None	25	0
2	Solvent-free	None	105	10
3	Solvent-free	TBAB (20%)	105	81
4	Solvent-free	TBAB (40%)	105	90
5	CHCl ₃	TBAB (10%)	Reflux	10
6	CH ₃ CN	TBAB (10%)	Reflux	25
7	DMF	TBAB (10%)	Reflux	15
8	Toluene	TBAB (10%)	Reflux	15
9	C ₂ H ₅ OH	TBAB (10%)	Reflux	57
10	H_2O	TBAB (10%)	25	48
11	H_2O	TBAB (10%)	70	89, 87, 89 ^b
12	H_2O	None	Reflux	18

^aIsolated yields.

^bCatalyst reused for three consecutive reactions.

(1 mmol) in water (5 mL), TBAB (0.1 mmol) was added. The reaction mixture was stirred at 70° C for the appropriate time. After completion of the reaction, as indicated by TLC, the reaction mixture was cooled to room temperature and filtered to afford the crude products. The further purified products were obtained by recrystalization from ethanol-water (4:1). In addition, the filtrate could be also reused for the next batch reaction.

Selected Characterization Data for Products

4b: Mp: 232–234°C. IR (KBr): 3314, 3195 (NH), 1659 (C=O) cm⁻¹. ¹H NMR (300 MHz, DMSO- d_6): $\delta = 8.23$ (s, 1 H), 7.60 (d, J = 7.7 Hz, 1 H), 7.37 (d, J = 8.1 Hz, 2 H), 7.17 – 7.25 (m, 3 H), 7.05 (s, 1 H), 6.14 (d, J = 8.1 Hz, 1 H), 6.66 (t, J = 7.7 Hz, 1 H), 5.70 (s, 1 H), 2.28 (s, 3 H). ¹³C NMR (75 MHz, DMSO- d_6): $\delta = 163.7$, 147.6, 138.9, 137.7, 133.2, 128.8, 127.5, 126.8, 117.3, 115.2, 114.6, 66.7, 21.8. Anal. Calcd. for C₁₅H₁₄N₂O: C 75.61, H 5.92, N 11.76; Found: C, 75.49; H, 5.99; N, 11.68.

4c: Mp: 198–200°C, IR(KBr): 3310, 3191(NH), 1655(C=O). ¹H NMR (300 MHz, DMSO-d₆): $\delta = 8.35$ (s, 1 H), 7.60 (d, J = 7.8 Hz, 1 H), 7.27 (t, J = 7.8 Hz, 1 H), 7.15 (s, 1 H), 6.74 (d, J = 8.2 Hz, 1 H), 6.68(t, J = 7.8 Hz, 1 H), 5.76 (s, 1 H). ¹³C NMR (75 MHz, DMSO-d₆): $\delta = 163.5$, 147.7, 140.2, 133.4, 131.2, 128.8, 128.3, 127.4, 117.3, 115.0, 114.5, 66.3. Anal. Calcd. for C₁₄H₁₁BrN₂O: C, 55.47; H, 3.66; N, 9.24; Found: C, 55.53; H, 3.71; N, 9.30.

4k: Mp: 227–229°C. IR (KBr): 3291, 3191 (NH), 1655 (C=O) cm⁻¹. ¹H NMR (300 MHz, DMSO-d₆): $\delta = 8.09$ (s, 1 H), 7.59 (d, J = 7.5 Hz, 1 H), 7.21–7.30 (m, 3 H), 6.93 (s, 1 H), 6.63–6.71 (m, 4 H), 5.62 (s, 1 H), 2.86 (s, 6 H). ¹³C NMR (75 MHz, DMSO-d₆): $\delta = 162.8$, 147.9, 133.1, 131.8, 128.7, 127.6, 117.3, 115.1, 114.3, 113.8, 67.1, 41.7 Anal. Calcd. for C₁₆H₁₇N₃O: C, 71.89; H, 6.41; N, 15.72; Found: C, 71.81; H, 6.47; N, 15.67.

4i: Mp: 191–193°C. IR (KBr): 3299, 3184 (NH), 1659 (C=O) cm⁻¹. ¹H NMR (300 MHz, DMSO-d6): δ = 8.20 (s, 1 H), 7.61 (d, *J* = 7.7 Hz, 1 H), 7.42 (d, *J* = 8.6 Hz, 2 H), 7.24 (t, *J* = 7.2 Hz, 1 H), 7.02 (s, 1 H), 6.95 (d, *J* = 8.6 Hz, 2 H), 6.74 (d, *J* = 8.1 Hz, 1 H), 6.67 (t, *J* = 7.2 Hz, 1 H), 5.70 (s, 1 H), 3.75 (s, 3 H). ¹³C NMR (75 MHz, DMSO-d₆): δ = 164.1,

Entry 1	R ¹ C ₆ H ₅			Method A Time Yield (min) (%) ^a		Method B Time Yield (min) (%) ^a		Mp (°C)	Ref
		NH ₄ OAc or R ² NH ₂ NH ₄ OAc	Product 4a						
				30	90	50	89	217–219	17c
2	$4-MeC_6H_4$	NH ₄ OAc	4b	30	91	50	87	232-234	17c
3	$4-BrC_6H_4$	NH ₄ OAc	4c	35	88	60	89	198-200	
4	$4-ClC_6H_4$	NH ₄ OAc	4d	35	87	60	87	206-208	18a
5	$4-NO_2C_6H_4$	NH ₄ OAc	4e	40	81	75	78	214-216	18a
6	$3-NO_2C_6H_4$	NH ₄ OAc	4f	40	79	75	79	261-218	18a
7	$2-NO_2C_6H_4$	NH ₄ OAc	4g	40	71	75	67	190-193	18a
8	$4-OHC_6H_4$	NH ₄ OAc	4h	30	89	45	83	277-279	18a
9	4-OMeC ₆ H ₄	NH ₄ OAc	4i	30	91	45	90	191–193	18a
10	$4-FC_6H_4$	NH ₄ OAc	4j	35	86	50	87	200-202	18a
11	$4-N(Me)_2C_6H_4$	NH ₄ OAc	4k	25	92	40	90	227-229	18a
12	$3,4-(OMe)_2C_6H_3$	NH ₄ OAc	41	25	91	40	89	212-214	17c
13	2-Furyl	NH ₄ OAc	4m	35	82	55	73	165-167	18a
14	2-Pyridyl	NH ₄ OAc	4n	35	85	55	77	184–186	18a
15	C_6H_5	PhNH ₂	4o	35	88	60	87	205-207	17c
16	$4-NO_2C_6H_4$	$PhNH_2$	4p	40	83	60	86	196–197	17c
17	$3-NO_2C_6H_4$	$PhNH_2$	4q	40	85	60	82	187–189	17c
18	$4-ClC_6H_4$	PhNH ₂	4r	35	90	55	89	216-218	17c
19	$4-ClC_6H_4$	PrNH ₂	4s	35	91	60	90	173-174	
20	$4-NO_2C_6H_4$	$PrNH_2$	4t	40	89	60	87	121-123	18a

 TABLE 2

 One-pot synthesis of 2 3-dihydroquinazolin-4(1H)-ones promoted by TBAB

^aIsolated yield.

147.1, 139.8, 133.1, 130.9, 128.6, 127.9, 127.1, 117.2, 115.3, 114.7, 66.7, 56.3. Anal. Calcd. for $C_{15}H_{14}N_2O_2$: C, 70.86; H, 5.51; N, 11.02; Found: C, 70.92; H, 5.55; N, 10.98.

40: Mp: 205–207°C. IR (KBr): 3307 (NH), 1657 (C=O) cm⁻¹. ¹H NMR (300 MHz, DMSO-d₆): δ = 7.73 (d, *J* = 7.5 Hz, 1 H), 7.67 (s, 1 H), 7.27–7.47 (m, 11 H), 6.75 (m, 2 H), 6.29 (s, 1 H). Anal. Calcd. For C₂₀H₁₆N₂O: C, 79.98; H, 5.37; N, 9.33; Found: C, 79.91; H, 5.46; N, 9.38.

4s: MP: 173–174°C. IR (KBr): 3303 (NH), 1662 (C=O) cm⁻¹. ¹H NMR (300 MHz, DMSO-d₆): δ = 7.20–7.33 (m, 5 H), 7.18 (s, 1 H), 7.58–7.61 (m, 1 H), 6.68 (t, *J* = 7.5 Hz, 1 H), 6.64 (d, *J* = 7.2 Hz, 1 H), 5.86 (s, 1 H), 3.80–3.85 (m, 1 H), 2.70–2.75 (m, 1 H), 1.42–1.55 (m, 1 H), 0.82 (t, *J* = 6 Hz, 3H). ¹³C NMR (75 MHz, DMSO-d₆): δ = 162.6, 146.5, 140.7, 133.6, 133.3, 128.9, 128.4, 127.9, 117.7, 115.5, 114.8, 69.7, 46.6, 21.3, 11.6. Anal. Calcd. for C₁₇H₁₇ClN₂O: C, 67.88; H, 5.70; N, 9.31; Found: C, 67.77; H, 5.79; N, 9.43.

RESULTS AND DISCUSSION

After many examinations, we found that the TBAB promoted efficiently synthesis of 2,3-dihydroquinazolin-4(1H)-ones in green and mild condition. This one-pot method involves three component reactions of isatoic anhydride, ammonium acetate

or primary amines, and different aldehydes in molten TBAB or in aqueous media with the use of catalytic amount of TBAB (Scheme 1). We optimized the reactions condition using isatoic anhydride, ammonium acetate and benzaldehyde in the presence of TBAB (Table 1). Results showed the efficient catalytic activity (Table 1, entries 2, 4, 11, and 12) and reusability (Table 1, entry 11) of TBAB. After optimization we studied the scope of this reaction. Generally, the cyclocondensation reactions proceeded well and afforded the desired products in good to excellent yields. The reaction was compatible with a variety of aryl aldehydes and heterocyclic aldehydes (Table 2).

On the other hand, we investigated the synthesis of 2,3disubstituted 2,3-dihydroquinazolin-4(1H)-ones from isatoic anhydride, primary amines and aryl aldehydes under optimized reaction condition and the disubstituted products of **40-4t** were obtained in high yields. (Table 1, entries 15–20). The method A has often better yields and shorter reaction times but the method B has a very simple workup procedure.

A tentative mechanism for the formation of 2,3-dihydroquinazolin-4(1H)-ones was proposed (Scheme 2). At the first step the condensation of isatoic anhydride with ammonia followed by decarboxylation could be produced antranilamide **5**. The intermediate imine **6**, could be obtained by the addition



SCH. 2.

of **5** with aldehydes promoted by TBAB. Imine **6** can undergo a cyclization to afford the dihydroquinazolinone product **4** (Scheme 2).

CONCLUSION

In conclusion, we described an efficient selective synthesis of 2,3-dihydroquinazolin-4(IH)-ones by the condensation reaction of isatoic anhydride, ammonium acetate or primary amine and an aldehyde using TBAB as a solvent and catalyst in good to excellent yields. Simple operations, short reaction time, environmental compatibility, availability and reusability of catalyst, and mild reaction conditions are key features of this new protocol.

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