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Graphical Abstract

Practical approach to 2-thioxo-2,3-dihydroquinazolin-4(1*H*)-one *via* dithiocarbamate – anthranilic acid reaction

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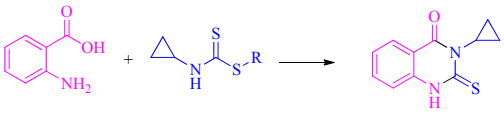

An efficient and straightforward method for the synthesis of 2-thioxo-2,3-dihydroquinazolin-4(1*H*)-one derivatives from the reaction of anthranilic acid derivatives with various dithiocarbamate derivatives has been successfully developed.

Original article

Practical approach to 2-thioxo-2,3-dihydroquinazolin-4(1*H*)-one *via* dithiocarbamate – anthranilic acid reaction

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ABSTRACT

A practical and straightforward protocol has been developed for the preparation of 2-thioxo-2,3-dihydroquinazolin-4(1*H*)-one derivatives from dithiocarbamate chemistry. The method involves the reaction of anthranilic acid derivatives (2-aminobenzoic acid, 2-aminobenzamide and isatoic anhydride) with various dithiocarbamate derivatives using ethanol as solvent. The main advantages of this protocol include practical simplicity, good to high yields, and ease of product isolation, purification and cheapness of the solvent.

1. Introduction

2-Thioxoquinazolin-4(1*H*)-ones are important heterocyclic compounds that widely present in natural products as well as medicinal, and pharmacological compounds [1]. In addition, several thioxoquinazolin analogues have been developed as antitumor, antibiotic, antidefibrillator and antipyretic agent (Fig.1). Furthermore, they display a broad range of applications for diabetes [2], cancer [3], and selective plant grow regulators [4,5]. Given the importance of these nitrogen heterocyclic compounds, the development of mild, high yielding and clean synthesis of these important compounds is daunting challenge and has been extensively investigated in literatures [6-12]. The classical methods for the synthesis of quinazolinedione ring system are the reaction of anthranilic acid and their derivatives with isothiocyanates or their equivalents [13-27]. However the difficult handling of unstable isothiocyanate as well as preparation of costly and toxic reagents, such as thiophosgene limits its practical applications. On the other hand, the dithiocarbamates are safe and highly efficient equivalent to isothiocyanate and were found to show broad application owing to their easy preparation in large scale from simple and readily available starting material [28-34]. In this context, our interest in green synthesis of dithiocarbamate derivatives [35,36], we aimed to develop an efficient and facile approach to synthesize a series of thioxoquinazolin-4(1*H*)-ones from dithiocarbamate derivatives under the mild and safe conditions.

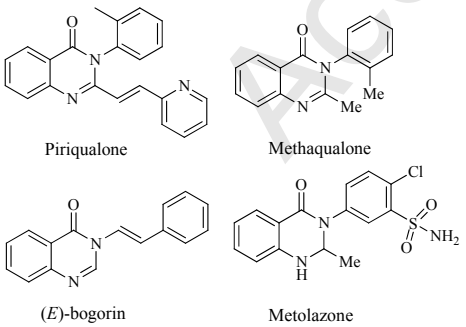

Fig. 1. Selected examples of quinazolin analogues with pharmacological activities.

2. Experimental

2.1 Materials and instrumentation

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All chemicals and solvents were commercially available. All products were confirmed by 1HNMR, FT-IR spectroscopy and mass spectrometry. ^{1}H NMR spectra were recorded on 500 or 300 MHz ^{1}H NMR, ^{13}C NMR 125.7 and 75 MHz NMR spectrometer using DMSO-d_6 as a solvent and chemical shifts have been expressed in (ppm) downfield from TMS. Water and ethanol were distilled before used. Melting points were recorded on Buchi 535 melting point apparatus and are uncorrected. FT-IR spectra were determined on a Bruker Vector-22 infrared spectrometer using KBr disks.

2.2. General procedure for the synthesis of 2-thioxo-2,3-dihydroquinazolin-4(1H)-one

To a well stirred solution of anthranilic acid **1** (0.5 mmol) and triethylamine (1.0 mmol) in ethanol (1 mL) was added dithiocarbamate **2** (0.5 mmol), and the reaction mixture was heated at 60 °C until the completion of the reaction (monitored by TLC). Once the reaction mixture has cooled to room temperature, resulted in the precipitation of a white solid, which was filtered, washed with water (10 mL) and recrystallized from ethanol or ethyl acetate to furnish pure 2-thioxoquinazolin-4(1*H*)-one derivatives without tedious work-up.

3-Benzyl-2-thioxo2,3-dihydroquinazolin-4(1*H*)-one (**3a**): ^{1}H NMR (500 MHz, DMSO-d_6): δ 5.24 (s, 2H, CH_2), 7.19-7.27 (m, 7H, Ar-H), 7.74-781 (m, 2H), 12.01 (brs, 1H, NH); ^{13}C NMR (125 MHz, DMSO-d_6): δ 50.4, 116.3, 116.6, 124.9, 126.1, 127.8, 129.3, 140.8, 127.9, 135.8, 140.6, 159.7, 175.2.

3-Butyl-2-tioxo-2,3-dihydroquinazolin-4(1*H*)-one (**3b**): ^{1}H NMR (500 MHz, DMSO-d_6): δ 0.94 (t, 3H, J = 6.8 Hz), 1.31-1.42 (m, 2H), 1.63-1.72 (m, 2H), 4.40 (t, 2H, J = 6.8 Hz), 7.18-7.20 (m, 2H) 7.74 (t, 1H, J = 6.8 Hz), 7.96 (d, 1H, J = 6.8 Hz), 12.90 (brs, 1H, NH); IR (neat, cm^{-1}): 3250, 3144, 2955, 2935, 1652, 1626, 1538, 1490, 1340, 1272, 1184, 1128, 990, 798, 758, 690.

3-Phenyl-2-thioxo-2,3-dihydroquinazolin-4(1*H*)-one (**3c**): ^{1}H NMR (500 MHz, DMSO-d_6): δ 7.25–7.28 (m, 2H), 7.35 (t, 1H, J = 7.7 Hz), 7.40–7.96 (m, 6H), 13.03 (brs, 1H, NH); ^{13}C NMR (125 MHz, DMSO-d_6): δ 115.6, 116.1, 124.3, 127.4, 128.0, 128.8, 128.9, 135.5, 139.3, 139.6, 159.7, 176.0.

3-(4-Butylphenyl)-2-thioxo-2,3-dihydroquinazolin-4(1*H*)-one (**3d**): ^{1}H NMR (500 MHz, DMSO-d_6): δ 0.89 (t, 3H, J = 6.9 Hz), 1.32-1.34 (m, 2H), 1.57-158 (m, 2H), 2.46 (t, 2H, J= 6.7 Hz); 7.14-7.24 (m, 8H), 7.30-7.91 (m, 1H), 12.90 (brs, 1H, NH).

3-Cyclopropyl-2-tioxo-2,3-dihydroquinazolin-4(1H)-one (**3e**): ^{1}H NMR (500 MHz, DMSO-d_6): δ 1.15-1.17 (m, 4H), 2.82—2.83 (m, 1H), 7.33-7.36 (m, 2H), 7.72-7.92 (m, 2H), 12.76 (brs, 1H, NH).

3-Phenylpropyl-2-thioxoquinazolin-4(1*H*)-one (**3f**): ^{1}H NMR (500 MHz, DMSO-d_6): δ 2.65-2.66 (m, 2H), 3.32 (t, 2H, J = 7.1 Hz), 4.44 (t, 2H, J=6.9 Hz), 7.27-7.31 (m, 5H), 7.33-7.37 (m, 2H), 7.73-7.94 (m, 2H), 12.90 (brs, 1H, NH).

3-[(Furan-2-yl)methyl]-2,3-dihydro-2-thioxoquinazolin-4(1*H*)-one (**3g**): ^{1}H NMR (500 MHz, DMSO-d_6): δ 5.65 (s, 2H), 6.34-6.37 (m 2H), 7.34-7.39 (m, 2H), 7.53–7.97 (m, 3H, 12.08 (brs,1H, NH);

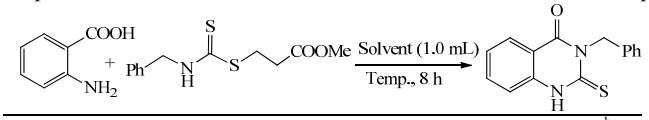
3-(3-Mthoxyphenyl)-2,3-dihydro-2-thioxoquinazolin-4(1*H*)-one (**3h**): ^{1}H NMR (500 MHz, DMSO-d_6): δ 3.74 (m, 3H), 6.88-7.04 (m, 3H), 7.20-7.35 (m, 3H), 7.43-7.94 (m, 2H), 13.01 (brs, 1H, NH). ^{13}C NMR (125 MHz, DMSO-d_6): δ 56.4, 116.2, 116.9, 124.8, 126.5, 127.4, 128.9, 130.4, 133.5, 136.1, 136.9, 138.7, 139.9, 154.1, 16.8, 177.6.

2-Thioxo-3-(*p*-tolyl)-2,3-dihydro-2-thioxoquinazolin-4(1*H*)-one (**3i**) ^{1}H NMR (500 MHz, DMSO-d_6): δ 2.37 (s, 3H), 7.10-7.25 (m, 2H), 7.26-7.38 (m, 3H), 7.45-7.95 (m, 3H), 12.06 (brs, 1H, NH). ^{13}C NMR (125 MHz, DMSO-d_6): δ 18.1, 114.8, 115.9, 128.0, 128.7, 129.1, 129.7, 130.9, 132.4, 135.5, 138.4, 139.38, 160.2, 175.8.

3. Results and discussion

Initially, we have investigated the condensation reaction of anthranilic acid **1** and dithiocarbamate **2** [36] as a model reaction in water (Table 1). The results indicate that when the condensation reaction was carried out at room temperature and 100 ^{o}C, it gave lower yields of product even after prolonged reaction time (Table 1, entries 1-2). On the other hand, when the model reaction was carried out in the presence of the triethylamine at reflux condition, the desired product was obtained in moderate yields (58%). A detailed inspection of the reaction showed that other base such as NaOH, KOH, and Na_2CO_3 did not improve the yield of the reaction. Moreover, different organic solvents were further studied. As shown in Table 1, the nonpolar solvents such as CH_2Cl_2 and toluene gave only moderate yields of the products (58% and 55%, respectively), the polar solvents (CH_3CN and DMF) gave much better yields than that of water. However, ethanol can give an excellent isolated yield (95%) for this reaction (Table 1, entry 10) and appeared to be the best media for this reaction in term of yields and product separation. Under optimum conditions, the reaction time was also studied (Table 1, entries 15-18). About 8 hours of reaction was sufficient for the completion of reaction. After the completion of the reaction, the reaction mixture was solidified and could be monitored visually. So, the reaction mixture was allowed to cool to room temperature, and the product was isolated by filtration after water addition.

Table 1
Optimization of reaction condition for the formation of 2-thioxoquinazolin-4(1*H*)-one.[a]



Entry	Solvent	Temp. (°C)	Promoter	Yield (%)[b]
1	H_2O	r.t.	-	Trace
2	H_2O	100	-	Trace
3	H_2O	100	Et_3N	58

4	H_2O	100	NaOH	35
5	H_2O	100	KOH	38
6	H_2O	100	Na_2CO_3	42
7	H_2O	100	PTSA	30
8	H_2O	100	HCl	45
9	CH_3CN	80	Et_3N	76
10	EtOH	60	Et_3N	95
11	Toluene	100	Et_3N	55
12	DMF	100	Et_3N	83
13	DCM	40	Et_3N	58
14	PEG	60	Et_3N	74
15	EtOH	60	Et_3N	40 (2 h)
16	EtOH	60	Et_3N	65 (4 h)
17	EtOH	60	Et_3N	80 (6 h)
18	EtOH	60	Et_3N	95 (12 h)

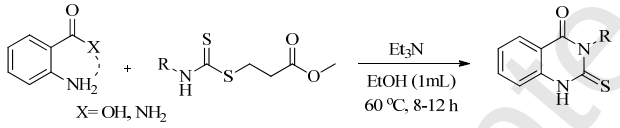
[a] Reaction conditions: anthranilic acid (0.5 mmol) and promoter (1.0 mmol) in solvent (1 mL) was added dithiocarbamate **2** (0.5 mmol) and the mixture was stirred.
[b] Isolated yields.

Encouraged by the initial success, we applied the optimal condition to the synthesis of a series of substituted 2-thioxoquinazolin-4(1*H*)-one and the results were found in Table 2. Various aliphatic and aromatic dithiocarbamates **2** were synthesized from the corresponding amines, carbon disulfide and Michael acceptor and were evaluated for the condensation reaction with anthranilic acid **1**. Various dithiocarbamates prepared from different primary aliphatic amines such as benzylamine, butyl amine, 1-phenylethylamine, 2-phenylethylamine, 3-phenylpropylamine and cyclopropylamine reacted with 2-aminobenzoic acid, smoothly under optimized condition to give the corresponding 2-thioxoquinazolin-4(1*H*)-one in good to excellent yield.

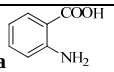
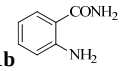
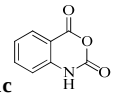
Furthermore, dithiocarbamates derived from primary aryl amines such as aniline, 4-methoxyaniline, 4-butylaniline and 4-chloroaniline were also found to react with 2-aminobenzoic acid to afford the desired products in good yield (Table 2). In a similar manner, we also studied the reaction of dithiocarbamates derived from secondary amines, such as pyrrolidine and diethylamine under the same reaction conditions. However, this condensation reaction between 2-aminobenzoic acid and dithiocarbamates did not take place and only starting materials were recovered. As an extension of this work, we carried out the reaction in 2-aminobenzamide and isatoic anhydride with dithiocarbamates using identical strategy. As outlined in Table 2, these gave the corresponding 2-thioxoquinazolin-4(1*H*)-one **3** in good yield.

Table 2

The preparation of 2-thioxo-2,3-dihydroquinazolin-4(1*H*)-one derivatives from dithiocarbamates.[a]

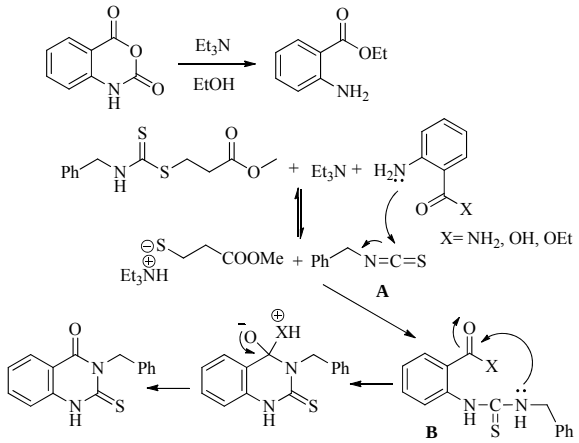


1 **2** **3**

Entry	**1**	R	**3**	Yield (%)[b]	Mp. Found	Mp. Rep.
1	 **1a**	$C_6H_5CH_2$-	**3a**	95	249-250	251-253[17]
2	 **1b**	$C_6H_5CH_2$-	**3a**	93	249-250	251-253[17]
3	 **1c**	$C_6H_5CH_2$-	**3a**	78	249-250	251-253[17]
4	**1a**	Bu-	**3b**	87	169-170	172[37]
5	**1a**	C_6H_5-	**3c**	90	305-307	305-306[38]
6	**1b**	C_6H_5-	**3c**	85	305-307	305-306[38]
7	**1c**	C_6H_5-	**3c**	80	305-307	305-306[38]
8	**1a**	4-Bu-C_6H_4-	**3d**	68	280-282	
9	**1b**	4-Bu-C_6H_4-	**3d**	71	280-282	
10	**1a**		**3e**	75	183-185	
11	**1b**		**3e**	83	183-185	
12	**1a**	$C_6H_5(CH_2)_3$-	**3f**	86	240-242	248-250[13]
13	**1b**	$C_6H_5(CH_2)_3$-	**3f**	93	240-242	248-250[13]
14	**1a**		**3g**	84	216-218	218-219[40]
15	**1a**	3-MeO-C_6H_4-	**3h**	84	251-253	248-249[39]
16	**1a**	4-$CH_3C_6H_4$-	**3i**	88	294-297	295-296[40]

[a] Reaction conditions: anthranilic acid (0.5 mmol) and triethylamine (1.0 mmol) in ethanol (1 mL) was added dithiocarbamate (0.5 mmol) and stirred at 60 ºC.
[b] isolated yields.

Based on our observations, a possible mechanism is depicted in Scheme 1. Treatment of dithiocarbamate with an excess of trimethylamine in refluxing ethanol resulted in isothiocyanate intermediate **A**, and then a second addition between **A** and anthranilic acid derivatives (2-aminobenzoic acid, 2-aminobenzamide and methyl anthranilate that *in situ* generated from the reaction of isatoic anhydride with ethanol in the presence of Et_3N) occurred, leading to thiourea intermediate **B**, which underwent intramolecular cyclization and subsequent deamination to furnish the dihydroquinazolin-4(1*H*)-one products.



Scheme 1. Suggested mechanism for the dihydroquinazolin-4(1*H*)-one synthesis from dithiocarbamate – anthranilic acid derivatives.

4. Conclusion

In summary, we have demonstrated a conceptually practical synthesis of extremely useful 2-thioxo-2,3-dihydroquinazolin-4(1*H*)-one derivatives from readily available dithiocarbamates and 2-aminobenzoic acid in good to excellent yields. This approach used a wide variety of dithiocarbamates prepared from commercially available primary amine substrates in large scale. The products can be isolated by a simple filtration of the reaction mixture without chromatography.

Acknowledgment

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