



Full paper/Mémoire

Three-component one-pot synthesis of 2,3-dihydroquinazolin-4(1H)-one derivatives in 2,2,2-trifluoroethanol

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ABSTRACT

Trifluoroethanol (TFE) is found to be an efficient and recyclable medium in promoting one-pot, three-component coupling reactions of isatoic anhydride, aldehyde and ammonium acetate or primary amine to afford the corresponding 2,3-dihydroquinazolin-4(1H)-one derivatives in high yields. The solvent (TFE) can be readily separated from reaction products and recovered in excellent purity for direct reuse.

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1. Introduction

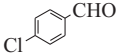
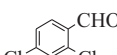
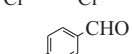
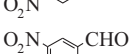
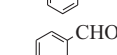
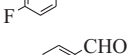
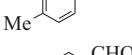
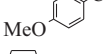
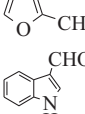
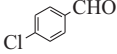
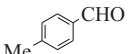
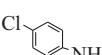
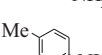
Fluorinated solvents have recently received a great deal of attention as potential new media for organic synthesis [1]. Owing to their unique physicochemical properties (high hydrogen bonding donor ability, nonvolatility, nonflammability, polarity, high ionizing power, and low nucleophilicity), fluorinated alcohols modify the course of reactions when they are used as solvents, allowing reactions which usually require the use of added reagents or metal catalysts to be carried out under neutral and mild conditions [2–16]. Therefore, today they have marched far beyond this border, showing their significant role in controlling the reaction as powerful reaction media. Reactions in fluorinated solvents are generally selective and without effluents, allowing thus a facile isolation of the product and a recovery of the solvent by distillation. In order to take advantage of these properties, we have studied bond cleavage and formation reactions facilitated by the acidic character and strong hydrogen bond donor ability of fluorinated alcohols. In a continuation of our work on the application of fluorinated alcohols in several organic transformations [17–22], we have developed an

efficient synthesis of 2,3-dihydroquinazolin-4(1H)-one derivatives under reflux conditions in 2,2,2-trifluoroethanol without the use of a catalyst or any other additives. 2,3-dihydroquinazolinones are very important compounds partially because of their pharmacological properties which include wide applications in medicinal chemistry; notable among them are antifertility, antibacterial, antitumor, antitremor, antifungal, and mono-amine oxidase inhibition [23–26]. In addition, 2,3-dihydroquinazolinone derivatives have recently been evaluated as antagonists of various biological receptors, such as 5-HT_{5A} related diseases [27], calcitonin gene-related peptide [28], and vasopressin V3 receptors [29]. Despite 2,3-dihydroquinazolinone usage in pharmaceutical and other industries, comparatively few methods for their preparation have been reported. In accordance with the significance of 2,3-dihydroquinazolinone, several synthetic methods have been developed for the construction of this kind of fused heterocycles from suitable precursors [30–45]. Very recently, Lee et al. reported a one-pot synthesis of 2,3-dihydroquinazolin-4(1H)-ones using ethylenediamine diacetate in aqueous media under reflux condition [46]. However, some of these procedures have certain limitations such as harsh reaction conditions, use of expensive acid catalysts in organic solvents, long reaction time, tedious work-up, and low yields. Thus, the development of novel methods for the

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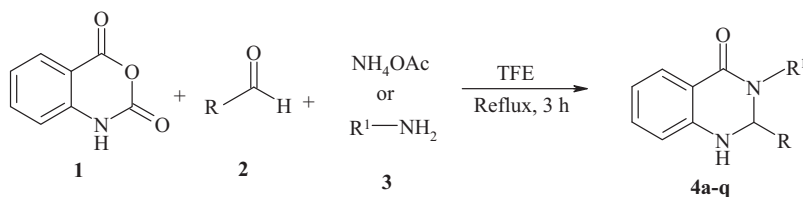
Table 1
Synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones in TFE.

Entry	Aldehyde	Amine	Product	Yield %
1		NH ₄ OAc	4a	95
2		NH ₄ OAc	4b	97
3		NH ₄ OAc	4c	80
4		NH ₄ OAc	4d	85
5		NH ₄ OAc	4e	92
6		NH ₄ OAc	4f	90
7		NH ₄ OAc	4g	90
8		NH ₄ OAc	4h	95
9		NH ₄ OAc	4i	90
10		NH ₄ OAc	4j	85
11		NH ₄ OAc	4k	92
12		NH ₄ OAc	4l	85
13			4m	85
14			4n	90
15			4o	85
16			4p	85
17			4p	90

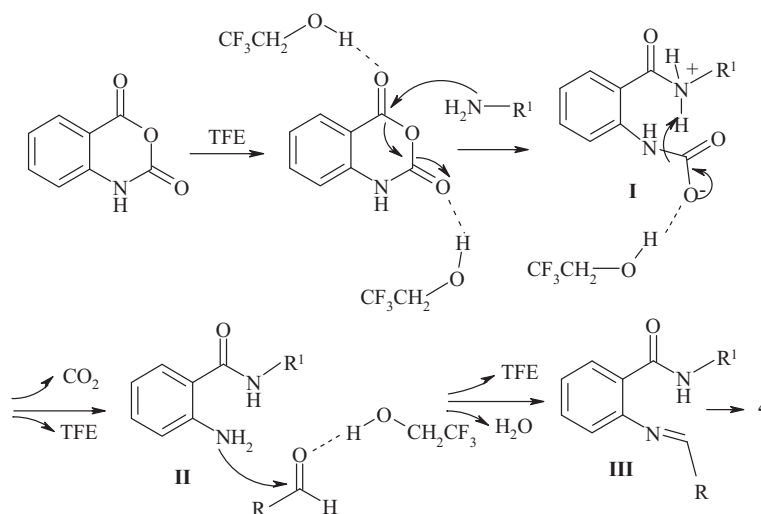
synthesis of dihydroquinazolin-4(1*H*)-ones is of great importance because of their potential biological and pharmaceutical activities. The present investigation describes a facile preparation of 2,3-dihydroquinazolinone derivatives via one-pot condensation of isatoic anhydride and aldehydes with NH₄OAc or primary amine in Trifluoroethanol (TFE) (Scheme 1).

2. Results and discussion

In an initial endeavor, the reaction was carried out by simply mixing isatoic anhydride, benzaldehyde and NH₄OAc (Table 1, entry 1) in trifluoroethanol and refluxing the resulting mixture for 3 hours. The corresponding 2,3-dihydroquinazolin-4(1*H*)-one **4a** was



Scheme 1. One-pot three-component condensation of isatoic anhydride, aldehydes and NH₄OAc or primary amine in TFE.



Scheme 2. Proposed mechanism for the synthesis of 2,3-dihydroquinazolin-4(1H)-ones.

obtained in high yield (95%). The application of this procedure to a variety of substrates (aldehydes and amines) was investigated and the results are summarized in Table 1. Various aldehydes, such as aromatic and heteroaromatic (**2a** to **2q**), could also participate in the reaction with isatoic anhydride (**1**) and NH_4OAc or primary amine to give the corresponding 2,3-dihydroquinazolin-4(1H)-one products **4a** to **4q** in good yields. As shown in Table 1, the effect of electronic nature of the substituents on the aromatic ring did not show strong influence on reaction yields. However, ortho-substituted aromatic aldehydes turned out to be reluctant to undergo smooth reaction probably because of steric hindrance (Table 1, entries 3,4). Furthermore, acid sensitive aldehydes worked well without any decomposition or polymerization under these reaction conditions (entry 11). To expand the scope of amine substrates, ammonium acetate and primary aromatic amines including aniline, *p*-toluidine, and 4-chloroaniline were applied to this protocol. In all cases, the desired reactions took place successfully to afford a series of 2,3-dihydroquinazolin-4(1H)-one (**4m–4q**) in good yields. The experimental procedure is very efficient, convenient, rapid and has the ability to tolerate a variety of other functional groups, such as alkyl, methoxyl, nitro, and halides under these reaction conditions. One of the major advantages of this protocol is the isolation and purification of the products, which have been achieved by simple filtration and crystallization of the crude products. The results illustrate the high ability of this method for the synthesis of substituted 2,3-dihydroquinazolinones with different groups. Interestingly, the reaction did not proceed to completion when either ethanol or water alone was used as solvent, even at reflux conditions. After the reaction, TFE can be easily separated (by distillation) and reused without decrease in its activity. For example, the reaction of isatoic anhydride, benzaldehyde and NH_4OAc (Table 1, entry 1) afforded the corresponding 2,3-dihydroquinazolin-4(1H)-one derivative in 95%, 95%, 92%, 90% and 90% isolated yield over five cycles. When we carried out the

reaction in TFE at room temperature, the reaction proceeded very slowly to give very poor yields.

A tentative mechanism for the formation of 2,3-dihydroquinazolin-4(1H)-ones was proposed (Scheme 2). In this process, the TFE acts as Brønsted acid [47] and plays a significant role in increasing the electrophilic character of the electrophiles. The hydrogen bond donor ability might not be important in this case. Actually, the hydrogen bond donating ability of these solvents drops as temperature rises owing to the fact that hydrogen bond formation is exothermic [48,49]. The polar transition state of the reaction could be stabilized well by the high ionizing solvent TFE. First, the isatoic anhydride is activated by TFE followed by the *N*-nucleophilic amine attacks on the carbonyl to form intermediate **I**. Then, decarboxylation occurs resulting in generation of 2-amino-*N*-substituted-benzamide (**II**). Subsequently, the reaction of activated aldehyde with **II** proceeds to afford intermediate **III** that is converted to product **4** via an intramolecular cyclization.

3. Conclusions

In summary, we have developed efficient synthesis of 2,3-dihydroquinazolin-4(1H)-one derivatives via one-pot condensation of isatoic anhydride, aldehydes and NH_4OAc in TFE without using any catalyst or additives. In contrast to the existing methods using potentially hazardous catalysts/additives, this new method offers the following competitive advantages:

- avoiding the use of any base, metal or Lewis acid catalyst;
- short reaction time;
- ease of product isolation/purification by non-aqueous work-up;
- high chemoselectivity;
- no side reaction;
- low costs and simplicity in process and handling. The recovered TFE can be reusable. Further studies and

efforts to extend the scope of this method for other useful reactions are currently underway.

4. Experimental

4.1. General procedure

Isatoic anhydride (1 mmol), aldehydes (1 mmol) and NH_4OAc (1 mmol) were dissolved in TFE (2 mL) and was refluxed for the stipulated time. The progress of the reaction is monitored by TLC. After completion of the reaction, the corresponding solid product **4** was obtained through simple filtering, and recrystallized from ethanol to yield the highly pure 2,3-dihydroquinazolin-4(1H)-one derivatives. The physical data (mp, IR, NMR) of known compounds were found to be identical with those reported in the literature. Spectroscopic data for selected examples are shown below.

2,3-Dihydro-2-phenylquinazolin-4(1H)-one (4a); white solid, mp 225–227 °C (Lit.[44] mp 218–220 °C); IR (KBr): 3302, 3176, 3062, 1653, 1610, 1508, 1482; ^1H NMR (500 MHz, $\text{DMSO}-d_6$): δ = 5.76 (s, 1H), 6.68 (t, J = 7.4 Hz, 1H), 6.76 (d, J = 8.09 Hz, 1H), 7.10 (br s, 1H, NH), 7.25 (t, J = 7.3 Hz, 1H), 7.33–7.41 (m, 3H), 7.50 (d, J = 7.44 Hz, 2H), 7.62 (d, J = 7.7 Hz, 1H), 8.28 (br s, 1H, NH); ^{13}C NMR ($\text{DMSO}-d_6$, 100 MHz): δ = 67.4, 115.2, 115.8, 117.9, 127.7, 128.2, 129.1, 129.3, 134.1, 142.5, 148.7, 164.4.

2-(4-Chlorophenyl)-2,3-dihydroquinazolin-4(1H)-one (4b); white solid, m.p 201–203 °C (Lit.[43] 198–200 °C); IR (KBr): 3307, 3186, 3025, 1667, 1651, 1607, 1504, 1483, 1383, 1292, 1133 cm^{-1} . ^1H NMR ($\text{DMSO}-d_6$, 400 MHz): δ = 5.77 (s, 1H), 6.70 (t, J = 8.1 Hz, 1H), 6.95 (d, J = 6.4 Hz, 1H), 7.15 (br s, 1H, NH), 7.22–7.47 (m, 3H), 7.51 (d, J = 8.8 Hz, 2H), 7.61 (dd, J_1 = 7.8 Hz, J_2 = 1.3 Hz, 1H), 8.34 (br s, 1H, NH); ^{13}C NMR ($\text{DMSO}-d_6$, 100 MHz): δ = 65.8, 114.4, 115.1, 117.27, 127.3, 128.3, 128.7, 132.9, 133.3, 140.7, 147.7, 163.4.

2,4-Dichloro-2,3-dihydroquinazolin-4(1H)-one (4d); white solid, m.p 182–184 °C (Lit.[44] 181–185 °C); IR (KBr): 3337, 3179, 3025, 1661 cm^{-1} . ^1H NMR ($\text{DMSO}-d_6$, 400 MHz): δ = 6.1 (s, 1H), 6.71 (t, J = 8.1 Hz, 1H), 6.93 (d, J = 6.4 Hz, 1H), 7.04 (br s, 1H, NH), 7.24–7.29 (t, J = 7.5 Hz, 1H), 7.47–7.50 (m, 1H), 7.65–7.68 (m, 3H), 8.25 (br s, 1H, NH). ^{13}C NMR ($\text{DMSO}-d_6$, 100 MHz): δ = 63.3, 114.6, 114.7, 117.6, 127.4, 128.6, 128.9, 130.9, 132.9, 133.5, 133.9, 136.9, 147.5, 163.6.

2-(4-Bromophenyl)-2,3-dihydroquinazolin-4(1H)-one (4g); white solid, m.p 201–203 °C (Lit.[43] 202–204 °C); IR (KBr): 3307, 3188, 3025, 1665, 1651, 1605, 1504, 1480, 1430, 1382 cm^{-1} . ^1H NMR ($\text{DMSO}-d_6$, 400 MHz): δ = 5.76 (s, 1H), 6.67–6.77 (m, 2H), 7.15 (s, 1H, NH), 7.25 (dt, J_1 = 7.7 Hz, J_2 = 1.5 Hz, 1H), 7.45 (d, J = 8.6 Hz, 2H), 7.58–7.62 (m, 3H), 8.35 (s, 1H, NH); ^{13}C NMR ($\text{DMSO}-d_6$, 100 MHz): δ = 65.8, 114.4, 114.9, 117.3, 121.5, 127.3, 129.1, 131.2, 133.4, 141.1, 147.6, 163.5.

2-(4-Methylphenyl)-2,3-dihydroquinazolin-4(1H)-one (4i); white solid, m.p 227–228 °C (Lit.[43] 233–234 °C); IR (KBr): 3310, 3190, 3015, 1667, 1655, 1606, 1507, 1482 cm^{-1} . ^1H NMR ($\text{DMSO}-d_6$, 400 MHz): δ = 2.30 (s, 3H), 5.90 (s, 1H), 6.67 (t, J = 7.3 Hz, 1H), 6.75 (d, J = 8.2 Hz, 1H), 7.06 (br s, 1H, NH), 7.19 (d, J = 7.8 Hz, 2H), 7.22–7.26 (m, 1H), 7.38–7.62 (m, 3H), 8.25 (br s, 1H, NH);

^{13}C NMR ($\text{DMSO}-d_6$, 100 MHz): δ = 20.7, 66.4, 114.9, 117.1, 126.7, 127.3, 128.7, 133.2, 137.2, 137.7, 138.7, 147.9, 163.6.

2-(4-Methoxyphenyl)-2,3-dihydroquinazolin-4(1H)-one (4j); white solid, m.p 179–180 °C (Lit.[43] 178–180 °C); IR (KBr): 3296, 3174, 2832, 1650, 1608, 1503, 1482, 1386, 1301, 1240 cm^{-1} . ^1H NMR ($\text{DMSO}-d_6$, 400 MHz): δ = 3.83 (s, 3H), 5.79 (s, 1H), 6.76 (t, J = 7.4 Hz, 1H), 6.83 (d, J = 7.6 Hz, 1H), 7.04 (dt, J_1 = 8.6 Hz, J_2 = 2.0 Hz, 2H), 7.10 (br s, 1H, NH), 7.30–7.35 (m, 1H), 7.51–7.71 (m, 3H), 8.28 (br s, 1H, NH); ^{13}C NMR ($\text{DMSO}-d_6$, 100 MHz): δ = 55.2, 66.3, 113.6, 114.4, 115.1, 117.07, 127.3, 128.2, 133.2, 133.4, 148.0, 159.5, 163.6.

2,3-Dihydro-3-phenyl-2-(4-chlorophenyl)quinazolin-4(1H)-one (4n); white solid, m.p 216–218 °C (Lit.[45] 216–217 °C); IR (KBr): 3294, 3025, 1631, 1613, 1506, 1486, 1414, 1385, 1312, 1161, 1088, 1014 cm^{-1} . ^1H NMR ($\text{DMSO}-d_6$, 400 MHz): δ = 6.33 (d, J = 2.7 Hz, 1H), 6.74 (t, J = 7.4 Hz, 1H), 6.77 (d, J = 7.8 Hz, 1H), 7.18–7.22 (m, 1H), 7.25–7.29 (m, 3H), 7.31–7.42 (m, 6H), 7.67–7.73 (m, 2H); ^{13}C NMR ($\text{DMSO}-d_6$, 100 MHz): δ = 72.1, 114.8, 115.3, 117.7, 126.1, 126.2, 128.1, 128.4, 128.5, 128.6, 132.9, 133.8, 139.6, 140.5, 146.4, 162.1.

2,3-Dihydro-3-phenyl-2-(4-methylphenyl)quinazolin-4(1H)-one (4o); white solid, m.p 222–224 °C (Lit.[43] 223–226 °C); IR (KBr): 3296, 3015, 1633, 1611, 1586, 1505, 1486 cm^{-1} . ^1H NMR ($\text{DMSO}-d_6$, 400 MHz): δ = 2.22 (s, 3H), 6.24 (d, J = 2.5 Hz, 1H), 6.71 (t, J = 7.6 Hz, 1H), 6.76 (d, J = 8.1 Hz, 1H), 7.10 (d, J = 7.8 Hz, 2H), 7.16–7.21 (m, 1H), 7.25–7.35 (m, 7H), 7.61–7.74 (m, 2H); ^{13}C NMR ($\text{DMSO}-d_6$, 100 MHz): δ = 20.6, 72.4, 114.8, 115.4, 117.4, 126.1, 126.1, 126.4, 127.9, 128.5, 128.9, 133.7, 137.5, 137.8, 141.1, 146.5, 162.2.

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