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IRON(III) NITRATE CATALYSED ONE-POT SYNTHESIS OF 2,3-DISUBSTITUTED QUINAZOLINONES BY COUPLING OF 2-BROMO BENZAMIDE, BENZALDEHYDE AND AMMONIA

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ABSTRACT
Heterocyclic compounds are commonly used Scaffolds on which pharmacophores are arranged to provide potent and selective drugs. This is especially true for six-membered ring heterocyclic compounds, which serve as the core components of many substances that possess a wide range of interesting biological activities. In this study, a series of Quinazolinone derivatives was designed and synthesized. The desired products were isolated in moderate to excellent yields in the presence of Iron(III) nitrate. The present protocol shows some specific advantages such as mildness, short reaction times. The chemical structures of the synthesized compounds were confirmed by ^{1}H NMR, ^{13}C NMR and mass spectral studies.

KEY WORDS: *Iron(III) nitrate, One-pot reaction, Quinazolinone, Synthesis*.

INTRODUCTION
Nitrogen-containing heterocycles are widely distributed in nature and are essential to life, playing a vital role in the metabolism of all living cells. Among those, Quinazolin-4(3*H*)-ones are also important building blocks in the synthesis of natural and pharmacological compounds.[I] which exhibits interesting pharmacological activities including antimalarial, antitumor, anticonvulsant, fungicidal, antimicrobial, and calcilytic activities.[II–VIII] These include kinase inhibition,[IX] anticancer,[X] antiinflammatory,[XI] antidiabetes, and anti-obesity activity.[XII] In addition more than 40 alkaloids containing a quinazolin- 4(3*H*)-one moiety have been isolated from natural source.[XIII] In addition, 4(3*H*)-quinazolinones are present in several bioactive natural products. [XIV-XV] For these reasons, their synthesis has received considerable attention.
Various approaches toward the synthesis of quinazolin-4(3*H*)-ones derivatives have been explored during the past years. One of the most common approaches is the cyclization of anthranilamides with aldehyde in the presence of various promoting agents, such as $NaHSO_3$,[XVI] p-toluenesulfonic acids/DDQ, [XVII] I_2, [XVIII] $CuCl_2$ (3.0 equiv),[XIX] and $FeCl_3$ (2.0 equiv).[XX] Some other methods include cyclization reaction of 2-amino benzamides with

substituted benzoyl chlorides in ionic liquid, [XXI] and cyclization of o-acylaminobenzamides, [XXII] 2-amino-benzonitrile,[XXIII] N-aryl orthanilamides,[XXIV] nitroenes.[XXV]

Very recently, several methods has been reported. Quinazolin-4-(*3H*)-one derivatives have been prepared from isatoic anhydride, ammonium acetate/ amines, and aldehydes in one-pot reaction catalyzed by $Ga(OTf)_3$ by Weike Su *et al*.[XXVI] Meng-Xia Liu *et al*. (2015) have developed $Y(OTf)_3$ catalyzed aerobic oxidative cyclization reaction for the selective synthesis of quinazolinones. [XXVII] Yu Tang *et al.* (2016) described the novel synthesis of quinazolinones by one-pot benign oxidative cyclization of alcohols with 2-aminobenzamides without catalyst under O_2. [XXVIII]Similarly, Adel S. El-Azab *et al*. (2016) studied the synthesis of new series of 2-substituted mercapto-4(3*H*)-quinazolinone.[XXIX] An efficient synthesis of quinazolinone derivatives has been performed from by the condensation of halide benzamide with amino acid using magnetically recyclable GO/Fe_3O_4–CuI as catalyst by Li-Yan Fan *et al.* (2016).[XXX] A convenient and transition-metal free protocol for quinazolinones synthesis with *o*-aminobenzamides and benzyl amines as substrates has been developed by Using H_2O_2 as the oxidant by Xiao Feng Wu *et al*. (2016). [XXXI] Quinazolinone derivatives bearing guanidinopropanoic acid derivatives were synthesized by Vijay Shankar Tiwari *et al*. (2016). [XXXII] A concise approach for the synthesis of Quinazolinones using an iron-catalyzed tandem reaction of 2-aminobenzamides with acyclic/cyclic 1,3-diketones via condensation, intramolecular nucleophilic addition in an aqueous solution of poly(ethylene glycol) under oxidant-free conditions has been developed by Guanshuo Shen *et al*. (2016). [XXXIII] Samira Karimpour *et al*. (2016) *r*eported the synthesis of Quinazolinones using nano-sized CuI particles under solvent-free conditions.[XXXIV] Thriveni *et al*. (2017) studied the synthesis of new series of Quinazolinone derivatives by one-pot reaction using a three-component condensation of anthranilic acid, amines, and ortho esters at room temperature under solvent-free conditions.[XXXV] Tandem cyclization of 2-halobenzoic acids with amidines provides a new facile quinazolinones using Cerium(III) chloride reported by Lalitha kumari *et al*. (2017).[XXXVI] An efficient protocol for the synthesis of Quinozolinones derivatives is achieved using BBr_3 as effective catalyst by Hari Krishna, *et al.,*[XXXVII] In this communication, we report a novel protocol for the selective synthesis of 2,3-disubstituted quinazolin-4(3*H*)-ones by using Iron(**III**) nitrate as effective catalyst.

MATERIALS AND METHODS

Melting points were determined in open-end capillaries and are uncorrected. Compounds were checked for their purity by TLC on silica gel G plates and spots were located by iodine vapors. The IR spectra were recorded on Perkin-Elmer spectrum RX IFT-IR System using KBr pellets. Elemental analyses of the newly synthesized compounds were carried out on Perkin Elmer model 2400 C H N analyzer. All the compounds gave satisfactory elemental analysis within ±0.4% of theoretical values. Ultra sonication was performed using BANDELIN SONOREX ® (Germany) 4D ultrasound cleaner with a frequency of 50 KHz and an output power of 480 W. The flask was located at the maximum energy area in the cleaner and addition or removal of water was used to control the temperature of the water bath.

All reactions were carried out under argon in oven-dried glassware with magnetic stirring. Unless otherwise noted, all materials were obtained from commercial suppliers and were used without further purification. All solvents were reagent grade. Unless otherwise noted, organic extracts were dried with anhydrous Na_2SO_4, filtered through a fritted glass funnel, and concentrated with a rotary evaporator (20–30 Torr). Flash chromatography was performed with silica gel (200–300 mesh) by using the mobile phase indicated. The NMR spectra were measured with a 400 MHz Bruker Avance spectrometer at 400.1 and 100.6

MHz, for ^{1}H for ^{13}C, respectively, in $CDCl_3$ solution with tetramethylsilane as internal standard. Chemical shifts are given in ppm (δ) and are referenced to the residual proton resonances of the solvents. Proton and carbon magnetic resonance spectra (^{1}H NMR and ^{13}C NMR) were recorded using tetramethylsilane (TMS) in the solvent of $CDCl_3$-*d* or DMSO-*d*6 as the internal standard (^{1}H NMR: TMS at 0.00 ppm, $CDCl_3$ at 7.26 ppm, DMSO at 2.50 ppm; ^{13}C NMR: $CDCl_3$ at 77.16 ppm, DMSO at 40.00 ppm.

General Procedure for the synthesis of 2,3-disubstituted Quinazolinone derivatives 3(a-i):

To a mixture of 2-bromobenzamide compound (80 mg, 0.4 mmol), benzaldehyde (82 mL, 0.8 mmol), Ferric nitrate nonahydrate (0.08 mmol) in DMSO (2 mL) was added 26% aqueous ammonia (0.5 mL) in a round bottom flask. The mixture was stirred at 80-100^{0}C for 6-8 hrs. A錯er being cooled to room temperature, the resulting mixture was quenched with NH_4Cl solution and extracted with ethyl acetate. The combined organic layer was washed with H_2O and brine, and organic layer was dried over Na_2SO_4, filtered, and concentrated under reduced pressure to give the crude product. The crude compound was purified through the silica gel column chromatography affords the quinazolinone in 70-78% yield. The structure were established by spectral (IR, ^{1}H NMR, ^{13}C NMR and mass) and analytical data

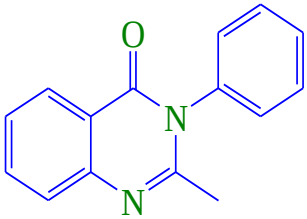
Scheme I:

The synthetic route was depicted in scheme I. Quinazolinone compounds 3(a-i) were synthesised in single step (one-pot reaction). The compounds 3(a-j) were obtained in moderate yields.

Scheme I: Synthesis of 2,3-disubstituted Quinazolinone derivatives

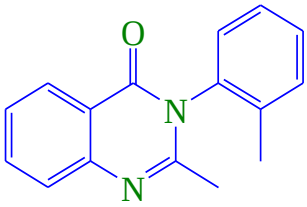
Spectral data for selected compounds:

2-methyl-3-phenylquinazolin-4(*3H*)-one (3a):



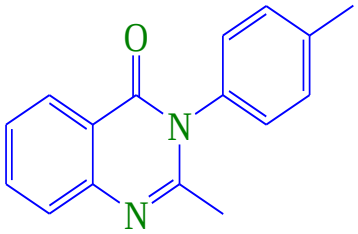
^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.28 (1H, dd, *J* = 1.0 Hz, *J* = 7.5 Hz), 7.79- 7.76 (1H, m), 7.70 (1H, d, *J* = 8.0 Hz), 7.59-7.51 (3H, m), 7.49- 7.46 (1H, m), 7.29- 7.27 (2H, m), 2.26 (3H, s);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 162.3, 154.3, 147.4, 137.8, 134.6, 130.0, 129.3, 128.0, 127.1, 126.75, 126.7, 120.8, 24.4;
HRMS (ESI-MS) cald. for C15H12N2O (M+Na) 259.0848, found 259.0846.

2-methyl-3-(*o*-tolyl)quinazolin-4(*3H*)-one (3b):



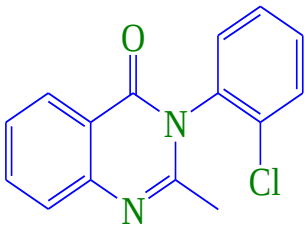
^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.18 (1H, d, *J* = 8.0 Hz), 7.65 (1H, t, *J* = 8.0 Hz), 7.58 (1H, d, *J* = 8.0 Hz), 7.36 (1H, t, *J* = 7.6 Hz), 7.30- 7.23 (3H, m), 7.05 (1H, d, *J* = 7.2 Hz), 2.08 (3H, s), 2.02 (3H, s);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 161.0, 153.7, 147.1, 136.2, 134.7, 134.0, 130.9, 128.9, 127.3, 127.0, 126.5, 126.2, 126.0, 120.1, 23.2, 16.8;
HRMS (ESI-MS) cald. for C16H14N2O (M+H) 251.1184, found 251.1181.

2-methyl-3-(p-tolyl)quinazolin-4(*3H*)-one (3c):



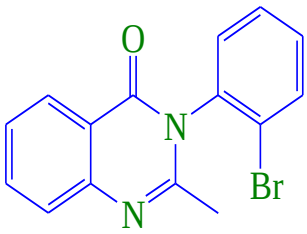
^{1}H NMR ($CDCl_3$, 400 MHz): δ 2.25 (s, 3H), 2.46 (s, 3H), 7.14 (d, *J* = 8.0Hz, 2H), 7.35 (d, *J* = 7.6 Hz, 2H), 7.46 (t, *J* = 7.6 Hz, 1H), 7.67 (d, *J* = 8.4 Hz, 1H), 7.76 (dt, *J1* = 8.0 Hz, *J2* = 0.8 Hz, 1H), 8.27 (d, *J* = 8.0 Hz, 1H) ppm;
^{13}C NMR ($CDCl_3$, 100 MHz): δ 21.3, 24.4, 120.8, 126.6, 126.7, 127.1, 127.7, 130.6, 134.5, 135.1, 139.3, 147.5, 154.5, 162.4;
HRMS (ESI): m/z $[M+H]^+$ calcd. for C16H14N2O 251.1179; found 251.1185

3-(2-chlorophenyl)-2-methylquinazolin-4(*3H*)-one (3d):



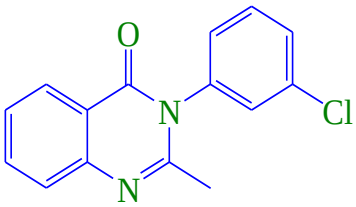
^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.31 (1H, dd, *J* = 1.2 Hz, *J* = 8.0 Hz), 7.83- 7.79 (1H, m), 7.72 (1H, d, *J* = 8.0 Hz), 7.66- 7.62 (1H, m), 7.52- 7.47 (3H, m), 7.38- 7.35 (1H, m), 2.25 (3H, s);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 161.5, 153.7, 147.5, 135.5, 134.8, 132.6, 130.85, 130.81, 129.9, 128.4, 127.2, 126.9, 126.8, 120.6, 23.6;
HRMS (ESI): m/z=271(M+H), positive mode.

3-(2-Bromo)-2-methylquinazolin-4(*3H*)-one (3e):



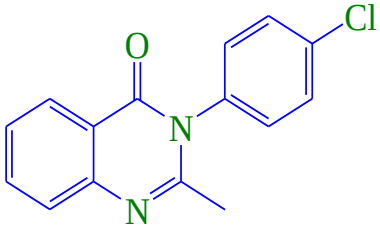
^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.31 (2H, d, *J* = 7.6 Hz), 7.81 (1H, t, *J* = 8.0 Hz), 7.72 (1H, d, *J* = 7.6 Hz), 7.57- 7.49 (2H, m), 7.42 (1H, t, *J* = 8.0 Hz), 7.38 (1H, d, *J* = 8.0 Hz), 2.25 (3H, s);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 161.4, 153.6, 147.6, 137.2, 134.8, 134.0, 130.9, 129.9, 129.1, 127.2, 126.9, 126.7, 122.9, 120.6, 23.7;
HRMS (ESI-MS) cald. for C15H11 79BrN2O (M+H) 315.0133, found 315.0128;
C15H11 81BrN2O (M+H) 317.0113, found 317.0109.

3-(3-chlorophenyl)-2-methylquinazolin-4(*3H*)-one (3f):



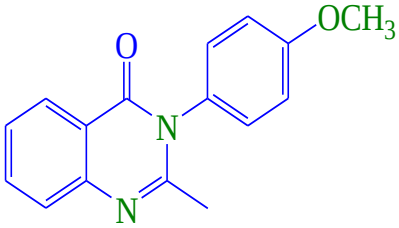
^{1}H NMR ($CDCl_3$, 400 MHz): δ 2.27 (s, 3H), 7.18-7.20 (m, 1H), 7.31 (s,1H), 7.46-7.51 (m, 3H), 7.68 (d, *J* = 8.4 Hz, 1H), 7.79 (dt, *J1* = 8.0 Hz, *J2* =1.2 Hz, 1H), 8.26 (dd, *J*1 = 8.0 Hz, *J*2 = 0.8 Hz, 1H);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 24.4, 120.6, 126.6, 126.8, 126.9, 127.1, 128.6, 129.7, 131.0, 134.8, 135.6, 138.8, 147.4, 153.5, 162.1;
HRMS (ESI): m/z $[M+H]^+$ calcd. for C15H11ClN2O 271.0633; found 271.0641.

3-(4-chlorophenyl)-2-methylquinazolin-4(*3H*)-one (3g):



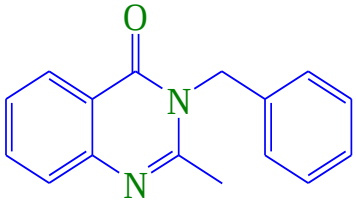
^{1}H NMR ($CDCl_3$, 400 MHz): δ 2.25 (s, 3H), 7.22 (d, *J* = 8.4 Hz, 2H), 7.48 (t, d = 8.4 Hz, 1H), 7.54 (d, *J* = 8.4 Hz, 2H), 7.68 (d, *J* = 8.0 Hz, 1H), 7.78 (t, *J* = 7.2 Hz, 1H), 8.26 (d, *J* = 8.0 Hz, 1H);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 24.4, 120.6, 126.8, 127.1, 129.5, 130.3, 134.8, 135.4, 136.2, 147.4, 153.7, 162.2;
HRMS (ESI): m/z $[M+H]^+$ calcd. for C15H11ClN2O 271.0633; found 271.0639.

3-(4-methoxyphenyl)-2-methylquinazolin-4(*3H*)-one (3h):



^{1}H NMR ($CDCl_3$, 400 MHz): δ 2.26 (s, 3H), 3.87 (s, 3H), 7.05 (d, *J* =8.8 Hz, 2H), 7.17 (d, *J* = 8.8 Hz, 2H), 7.46 (t, *J* = 7.6 Hz, 1H), 7.66 (d, *J* = 8.0 Hz, 1H), 7.76 (dt, *J1* = 8.4 Hz, *J2* = 1.2 Hz, 1H), 8.26 (dd, *J*1 = 8.0 Hz, *J*2 = 0.8 Hz, 1H);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 24.4, 55.6, 115.1, 120.8, 126.6, 126.7, 127.1, 129.0, 130.2, 134.5, 147.5, 154.8, 159.9, 162.6;
HRMS (ESI): m/z $[M+H]^+$ calcd. for C16H14N2O2 267.1128; found 267.1135.

3-benzyl-2-methylquinazolin-4(*3H*)-one (3i):



^{1}H NMR ($CDCl_3$, 400 MHz): δ 2.59 (s, 3H), 5.44 (s, 2H), 7.24 (d, *J* = 7.2Hz, 2H), 7.31-7.38 (m, 3H), 7.51 (t, *J* = 7.6 Hz, 1H), 7.67 (d, *J* = 8.0 Hz, 1H), 7.78 (dt, *J1* = 8.4 Hz, *J2* = 1.2 Hz, 1H), 8.35 (dd, *J*1 = 8.0 Hz, *J*2 = 0.8 Hz,1H);
^{13}C NMR ($CDCl_3$, 100 MHz): δ 23.4, 47.1, 120.2, 126.5, 126.6, 126.7, 127.1, 127.7, 128.9, 134.4, 135.8, 147.3, 154.6, 162.4;
HRMS (ESI): m/z $[M+H]^+$ calcd. for C16H14N2O 251.1179; found 251.1185

RESULTS AND DISCUSSION

In our preliminarily investigation on the model reaction of 2-aminobenzamide, aldehyde, and aqueous ammonia, it was found that the reaction could be finished under very simple reaction conditions in the presence of Ferric nitrate as catalyst. In order to investigate the scope of the reaction, different substituted aromatic ring in 2-aminobenzamide were employed and it was clear that yield is not affected by the position of the substituent on aromatic ring. Ferric nitrate can efficiently catalyze a one-pot synthesis of 2,3-disubstituted quinazolinones via a three-component condensation of 2-bromobenzamide with aldehydes and aqueous ammonia under air (**Scheme 1**). The reaction was carried out under very simple reaction conditions which gives the desired 2,3-disubstituted Quinazolinone derivatives in good yield. These methods are more convenient and reactions can be carried out in higher yield. In such consequence we have developed a new protocol for the preparation of 2,3-disubstituted quinazolinones with short times and high yields. Formation of products was confirmed by recording their ^{1}H NMR, ^{13}C, mass spetra.

CONCLUSION

In summary, we have developed a convenient and rapid synthetic route to 2,3-disubstituted quinazolinones via ferric nitrate catalyzed tandem reaction of 2-bromobenzamides with aldehydes and aqueous ammonia under air. The present protocol exhibits good functional

group tolerance, readily available and inexpensive starting materials, and operational simplicity. To further explore novel synthetic approaches toward other nitrogen-containing heterocyclic compounds is underway in our laboratory.

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