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BORON TRIBROMIDE CATALYSED FACILE AND EFFICIENT SYNTHESIS OF 2,3-DISUBSTITUTED QUINAZOLINONE DERIVATIVES

Chikkanti Jaganmohan^{a,b}, Vinti SP.Rao^a, Shiva Prakash.P^a, Vinay Kumar K.P^a, Sandeep Mohanty^a, Jaydeep Kumar^a, Venkateswara Rao B^b and Akula Raghunadh^a

^aDr. Reddy's Laboratories Limited, API Plant, IDA Bollaram, Hyderabad, India;

^bDepartment of Organic Chemistry & Chemistry of Foods, Drugs and Water Analysis, Andhra University, Visakhapatnam, Andhra Pradesh, India;

*Corresponding Author E-mail:- chikkanti2018jagan@gmail.com

ABSTRACT

A Simple, convenient synthetic protocols have been developed for the synthesis of 2,3-disubstituted Quinazolinone derivatives using Borontribromide as efficient catalyst. This reaction proceeds under mild conditions. This method was found to be better method giving high yields. The present method shows some advantages such as short reaction times and enhanced selectivity. The chemical Structures of the Compounds are confirmed by ¹H NMR & ¹³C NMR, Mass spectral data.

KEY WORDS: Borontribromide, 2-amino-N-substituted benzamide, Synthesis, 2,3-disubstituted quinazolinone.

INTRODUCTION

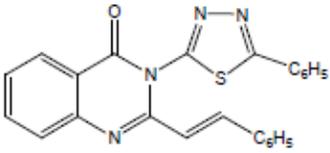
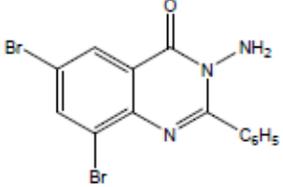
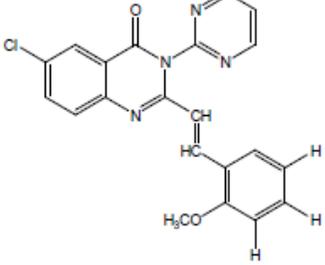
The chemistry of heterocyclic compounds represents half of all organic chemistry research worldwide. In particular, heterocyclic structures form the basis of many pharmaceutical and other bioactive products. Heterocyclic compounds form the basis of many pharmaceutical, agrochemical and veterinary products. Among a wide variety of nitrogen heterocyclic moieties that have been explored for developing pharmaceutically useful molecules, quinazolinone plays an important role in medicinal chemistry and subsequently have emerged as a pharmacophore that possess a diversity of useful biological activities. The pharmacodynamic versatility of quinazolin-4-one moiety has been documented not only in many of its synthetic derivatives but also in several naturally occurring alkaloids isolated from animals, plants and microorganisms.

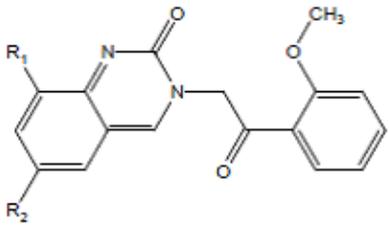
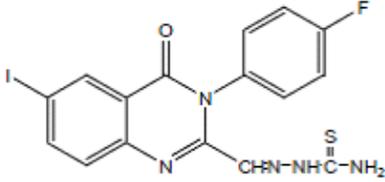
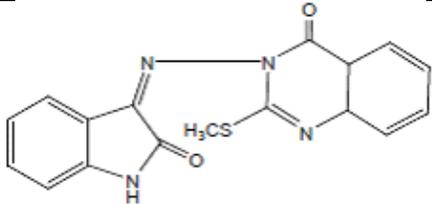
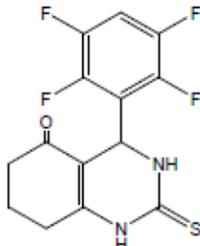
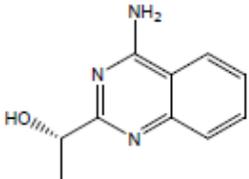
Quinazolinones are one of the classes of fused heterocycles that are of considerable interest [1]. Construction of small molecule mimics of biological structures is a key

contribution of organic chemistry to the discovery of new pharmaceuticals with the wide range of biological activities. The stability of quinazolin-4-one nucleus has inspired medicinal chemists to introduce many bioactive moieties to this nucleus to synthesize new potential medicinal agents [2]. Quinazolinone nucleus is also considered as one of the well-established peptidomimetic scaffold [3]. 4(3*H*)-quinazolinones are most prevalent, either as intermediates or as natural products in many proposed biosynthetic pathways. Quinazolines are classes of fused heterocycles that are of considerable interest because of the diverse range of their biological properties. Quinazolin-4(3*H*)-ones are displaying a broad spectrum of biological and pharmacological activities such as anti-fungal [4], anti-tumour [5], hypotensive [6], anti-cancer [7, 8], anti-HIV [9], anti-inflammatory [10], anti-bacterial [11] etc.

In addition to their occurrence in natural products, they also frequently appear in pharmaceutical agents for their applications as potent antagonistic receptors [12]. For example, Pegamine, isolated from *Peganum harmala*, has been found to possess cytotoxic activity [13] and NPS 53574, a new calcilytic template for blocking calcium receptor (CaR) activity, is proven to be capable of treating osteoporosis [14]. Both of these two biologically active compounds possess the common quinazolinone skeleton in their respective structure. Some of Quinazolinone derivatives with their biological activities were shown in **Table 1**.

Table 1: Some of Quinazolinone derivatives with their biological activities

	<p>3-(5-phenyl-1,3,4-thiadiazol-2-yl)-2-styrylquinazolin-4(3<i>H</i>)-one (CNS depressant activity)</p>
	<p>6,8-dibromo-2-phenyl benzoxazines (analgesic activity)</p>
	<p>2-(2-methoxystyryl)-6-chloro-3-(pyrimidin-2-yl) quinazolin-4(3<i>H</i>)-One (antileukemic activity)</p>

	3-(2-(2-methoxyphenyl)-2-oxoethyl)quinazolinone derivatives (anticoccidial activity)
	1-((3-(4-fluorophenyl)-3,4-dihydro-6-iodo-4-oxoquinazolin-2-yl)methylene)thiosemicarbazides (anticonvulsant activity)
	3-(2-oxoindolin-3-ylideneamino)-2-(methylthio)quinazolin-4(3H,4aH,8aH)-one (anti HIV activity)
	4-(2,3,5,6-tetrafluorophenyl)-1,2,3,4,7,8-hexahydro-2-thioxoquinazolin-5(6H)-one (antifungal activity)
	(S)-1-(4-Aminoquinazolin-2-yl)ethanol (animutagenic activity)

Considering the significance of this class of compounds, many efforts have been devoted to explore the methodologies for the construction of 4(3*H*)-quinazolinone skeletons. Several methods for the synthesis of 4(3*H*)-quinazolinones have been investigated in the past. The representative methods involve cyclization of *o*-acyl aminobenzamide [15], amination of benzoxazin-4-one [16], multicomponent reactions (MCRs) among isatoic anhydride, amine with aldehyde [17], benzyl halide [18] or orthoester [19]. Some common methods include the condensation of 2-aminobenzamides and substituted benzoyl chlorides or their equivalents in ionic liquids [20], the tandem condensation and C–N cross coupling of 2-halobenzoic acids and amidines [21]. The condensation of 2-aminobenzamides and substituted benzoyl chlorides or their equivalents in ionic liquids,[22] the tandem condensation and C–N cross coupling of 2-halobenzoic acids and amidines,[23] the cyclization of *o*-acylaminobenzamides,[24] 2-amino-benzonitrile,[25]

N-aryl orthanilamides,[26] nitroenes,[27] and aza-Wittig reactions of α -azido-substituted aromatic imides.[28] However, benzyl chlorides are carcinogenic alkylating agents and poisonous lachrymators and the base K_2CO_3 is needed to neutralize the hydrochloride produced during the reaction.[29] Very recently, Ma developed a CuI–4-hydroxy-L-proline catalyzed coupling involving N-substituted o-bromobenzamides and formamide or other amides to afford 3-substituted quinazolinones directly, or 2,3-disubstituted quinazolinones via a HMDS–ZnCl₂ mediated condensative cyclization.[30] Unfortunately, both approaches lead to the generation of undesired byproducts, such as hydrogen halide which consumes K_2CO_3 making the overall process less efficient in terms of atom economy. Dabiri et al. reported a one-pot three-component route to synthesize 2,3-disubstituted 4(3*H*)-quinazolinones in the presence of an equivalent amount of iodine as the catalyst.[31] Recently, a series of Quinazolinone derivatives were synthesised from isatoic anhydride and benzimidamide using BBr_3 as a catalyst,[32] and Tandem cyclization of 2-halobenzoic acids with amidines using Cerium(III) chloride as catalyst,[33] one-pot reaction using a three-component condensation of anthranilic acid, amines, and ortho esters at room temperature under solvent-free conditions.[34] Recently, transition-metal-catalyzed reactions have emerged as versatile tools for the construction of quinazolinones. For example, palladium-catalyzed carbonylation/cyclization cascades turned out to be an efficient approach toward quinazolinone derivatives.[35]

However, most of these procedures have some drawbacks, as they have a poor atom economy, use environmentally toxic reagents or media, use expensive chemicals, and often involve harsh reaction conditions, unsatisfactory yields and most seriously, the use of the stoichiometric oxidants or heavy metal reagents. Therefore, the development of more practical and efficient approaches toward quinazolinone derivatives remains an attractive task for organic chemists. Herein we report an efficient synthesis of 4(3*H*)-quinazolinones using BBr_3 as catalyst (**Scheme I**).

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. 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 1H 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 (1H 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

(¹H NMR: TMS at 0.00 ppm, CDCl₃ at 7.26 ppm, DMSO at 2.50 ppm; ¹³C NMR: CDCl₃ at 77.16 ppm, DMSO at 40.00 ppm.

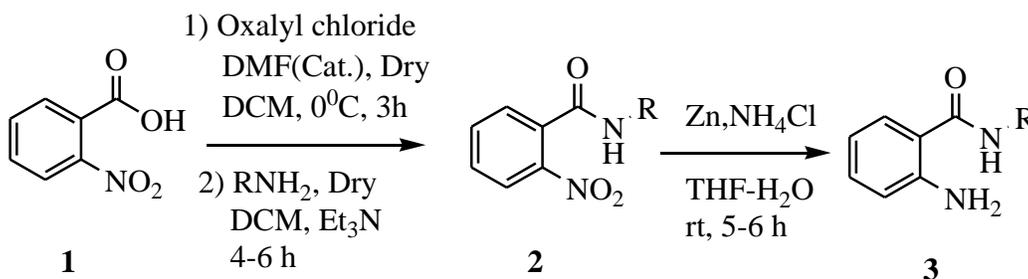
(I) General procedure for synthesis of starting material:

(a) General procedure for synthesis of 2-nitro *N*-substituted benzamide (2):

To 2-nitrobenzoic acid (**1**) (6 mmol), oxalyl chloride (8 ml) in dry DCM (10 ml) was added for 3 hrs at room temperature and stirred in round bottom flask. Next, the resultant acid chloride was added to the substituted amines (4 mmol) and triethylamine (10 ml). It was stirred for another 4-6 hrs at room temperature to furnish the respective 2-nitro-*N*-(substituted)benzamides (**2**). (**Scheme 1**)

(b) General procedure for synthesis of 2-amino-*N*-substituted benzamide (3):

2-Nitro-*N*-(substituted)benzamide(**2**) (2.2 mmol) was dissolved in THF-H₂O solution (5:1, 20 ml). To this solution, NH₄Cl (6.6 mmol) and Zn dust (17.6 mmol) were added. The reaction mixture was stirred at room temperature for 5-6 hrs. After the completion of the reaction, reaction mixture was filtered and extracted with ethyl acetate and evaporated to dryness. The residue was purified by column chromatography on silica gel to give compound **3**. (**Scheme 1**)



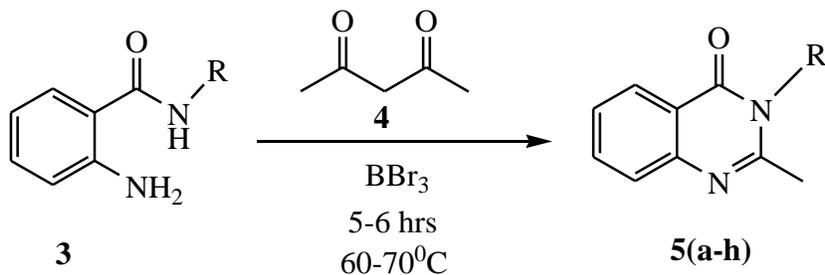
Scheme 1

(II) General procedure for synthesis of target compound:

(a) General procedure for synthesis of 2,3-disubstituted Quinazolinone derivatives 5(a-h):

To a solution of 2-amino *N*-substituted benzamide (**3**) (2 mmol) in ethanol, pentane-2,4-dione (**4**) (3 mmol), BBr₃ catalyst (0.2 mmol) in dichloromethane was added at room temperature under N₂ atmosphere. The reaction mixture was stirred at 60-70°C for 5-6 hrs. After completion of reaction (monitored by TLC), the reaction mixture was quenched with water (8 ml). The organic layer was dried over anhydrous Na₂SO₄, filtered, and concentrated under reduced pressure to give the crude product. The crude compound was purified through the silica gel column chromatography using ethyl acetate and hexane

(30:70) as eluent. The structure were established by spectral (IR, ^1H NMR, ^{13}C NMR and mass) and analytical data. (**Scheme 2**) Quinazolinone compounds 5(a-h) were obtained in moderate yields. The synthetic route was depicted in **Scheme 2**.



Scheme 2

Spectral data for selected compounds:

2,3-dimethylquinazolin-4(3H)-one (5a)

White solid, mp 110-111°C;

^1H NMR (CDCl₃, 400 MHz): δ 2.63 (s, 3H), 3.63 (s, 3H), 7.44 (dt, $J = 8.0$ Hz, 1H), 7.61 (d, $J = 8.0$ Hz, 1H), 7.71 (dt, $J = 8.0$ Hz, 1H), 8.25 (dd, $J = 8.0$ Hz, 1H) ppm;

^{13}C NMR (CDCl₃, 100 MHz): δ 23.6, 31.1, 120.2, 126.5, 126.5, 126.8, 134.3, 147.1, 154.6, 162.3 ppm;

HRMS (ESI): m/z [M+H]⁺ calcd. for C₁₀H₁₀N₂O 175.0866; found 175.0870.

2-Methyl-3-phenylquinazolin-4(3H)-one (5b)

brown solid, m.p. = 130-132 °C ;

^1H NMR (CDCl₃, 400 MHz): δ 8.28 (dd, $J = 7.5$ Hz, 1H), 7.79- 7.76 (m, 1H), 7.70 (d, $J = 8.0$ Hz, 1H), 7.59-7.51 (m, 3H), 7.49- 7.46 (m, 1H), 7.29- 7.27 (m, 2H), 2.26 (s, 3H);

^{13}C NMR (CDCl₃, 100 MHz): δ 24.4, 120.8, 126.7, 126.75, 127.1, 128.0, 129.3, 130.0, 134.6, 137.8, 147.4, 154.3, 162.3.

HRMS (ESI-MS) cald. for C₁₅H₁₂N₂O (M+Na) 259.0848, found 259.0846.

2-methyl-3-(o-tolyl)quinazolin-4(3H)-one (5c)

yellow solid; ; m.p. = 112°C ;

^1H NMR (CDCl₃, 400 MHz): δ 8.18 (d, $J = 8.0$ Hz, 1H), 7.66 (t, $J = 8.0$ Hz, 1H), 7.59 (d, $J = 8.0$ Hz, 1H), 7.36 (t, $J = 7.6$ Hz, 1H), 7.30- 7.23 (m, 3H), 7.05 (d, $J = 7.2$ Hz, 1H), 2.08 (s, 1H), 2.02 (s, 1H);

^{13}C NMR (CDCl₃, 100 MHz): δ 16.8, 23.3, 120.1, 126.0, 126.2, 126.5, 127.0, 127.3, 128.9, 130.9, 134.0, 134.7, 136.2, 147.0, 153.7, 161.0,

HRMS (ESI-MS) cald. for C₁₆H₁₄N₂O (M+H) 251.1184,found 251.1181.

2-methyl-3-(p-tolyl)quinazolin-4(3H)-one (5d)

White solid, mp 150-151°C;

¹H NMR (CDCl₃, 400 MHz): δ = 2.25 (s, 3H), 2.45 (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, *J* = 8.0 Hz, 1H), 8.27 (d, *J* = 8.0 Hz, 1H);

¹³C NMR (CDCl₃, 100 MHz): δ = 21.3, 24.4, 120.8, 126.5, 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-bromophenyl)-2-methylquinazolin-4(3H)-one (5e)

white solid; m.p. = 144°C;

¹H NMR (CDCl₃, 400 MHz): δ 8.31 (d, *J* = 7.6 Hz, 1H), 7.81 (t, *J* = 8.0 Hz, 1H), 7.72 (d, *J* = 7.6 Hz, 1H), 7.57- 7.49 (m, 1H), 7.42 (t, *J* = 8.0Hz, 1H), 7.38 (d, *J* = 8.0 Hz, 1H), 2.25 (s, 3H);

¹³C NMR (CDCl₃, 100 MHz): δ 23.7, 120.6, 122.9, 126.7, 126.9, 127.2, 129.1, 129.9, 130.9, 134.0, 134.8, 137.2, 147.6, 153.6, 161.4.

HRMS (ESI-MS) cald. for C₁₅H₁₁79BrN₂O (M+H) 315.0133, found 315.0128;

C₁₅H₁₁81BrN₂O (M+H) 317.0113, found 317.0109.

3-(2-chlorophenyl)-2-methylquinazolin-4(3H)-one (5f)

yellow solid; m.p. = 98-100°C;

¹H NMR (CDCl₃, 400 MHz): δ 8.31 (dd, *J* = 8.0 Hz, 1H), 7.83- 7.79 (m, 1H), 7.72 (d, *J* = 8.0 Hz, 1H), 7.66- 7.62 (m, 1H), 7.52- 7.47 (m, 3H), 7.38- 7.35 (m, 1H), 2.25 (s, 3H);

¹³C NMR (CDCl₃, 100 MHz): δ 23.6, 120.6, 126.8, 126.9, 127.2, 128.4, 129.9, 130.81, 130.85, 132.6, 134.8, 135.5, 147.5, 153.7, 161.5.

HRMS (ESI-MS) *m/z*=271 (M+H), positive mode.

3-(4-chlorophenyl)-2-methylquinazolin-4(3H)-one (5g)

White solid, mp 157-158°C;

¹H NMR (CDCl₃, 400 MHz): δ = 2.25 (s, 3H), 7.22 (d, *J* = 8.4 Hz, 2H), 7.48 (t, *J* = 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);

¹³C NMR (CDCl₃, 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 (5h)

White solid, mp 169-171°C;

¹H NMR (CDCl₃, 400 MHz): δ = 2.26 (s, 3H), 3.88 (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, *J* = 8.4 Hz, 1H), 8.26 (dd, *J* = 8.0Hz, 1H);

¹³C NMR (CDCl₃, 100 MHz): δ = 24.4, 55.5, 115.2, 120.8, 126.6, 126.7, 127.1, 129.0, 130.2, 134.5, 147.5, 154.8, 159.9, 162.5;

HRMS (ESI): *m/z* [M+H]⁺ calcd. for C₁₆H₁₄N₂O₂ 267.1128; found 267.1135.

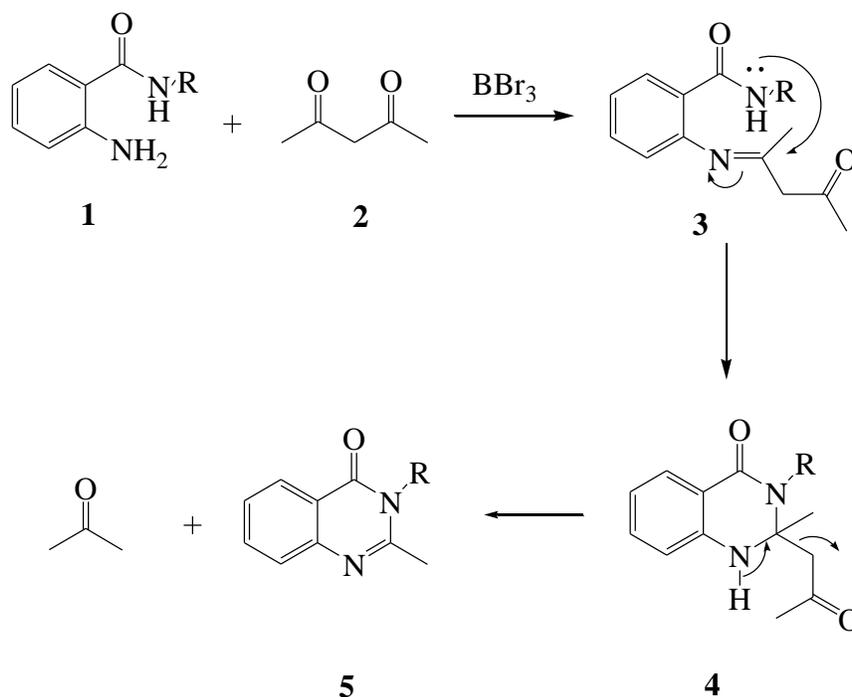
CONCLUSION

In summary, we have developed a convenient and rapid synthetic route to 2,3-disubstituted quinazolinones *via* Boron tribromide catalyzed tandem reaction of 2-amino

N-substituted benzamide and pentane-2,4-dione. The present protocol exhibits good functional group tolerance, readily available and inexpensive starting materials, and operational simplicity. The reaction proceeds under mild conditions with good to excellent yields. The advantage of this method are extremely mild reaction conditions, short reaction time, high yield. To further explore novel synthetic approaches toward other nitrogen-containing heterocyclic compounds is underway in our laboratory.

Based on the results obtained above, a proposed reaction mechanism was shown in (Scheme 3) for the formation of 2,3-disubstituted quinazolinone derivatives. The Lewis acid BBr_3 catalyzed condensation reaction of 2-amino N-substituted benzamide **1** with 1,3-diketone **2** would take place to generate a ketimine intermediate **3**. Then, the intramolecular nucleophilic addition of **3** would produce adduct **4**. The C-C bond cleavage reaction would finally occur to generate the desired product **5**.

Mechanism



Scheme 3

ACKNOWLEDGEMENT

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