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# Articles and Statements

# Synthesis of 2,3-Disubstituted Quinazolinone Derivatives Using Silver Triflate as a Efficient Catalyst at Room Temperature

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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 Nitrogen containing hetero cycles, which serve as the core components of many substances that possess a wide range of interesting biological activities. A series of 2,3-disubstituted quinazolinone derivatives have been synthesized in excellent yields at room temperature. The reaction was efficiently promoted by AgoTf. This protocol is very simple and provides moderate yields. All the products were identified by spectral (<sup>1</sup>H NMR, <sup>13</sup>C NMR and mass) and analytical data.

Keywords: synthesis, 2,3-disubstituted quinazolinone, silver triflate, room temperature.

# 1. 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.

Literature survey revealed the versatile biological activities of quinazolinone derivatives (Büyüktimkin, 1985; Büyüktimkin et al., 1989). It has been established that quinazolinones possess antiviral (Mukherji et al., 1980), antifungal (Peet et al., 1986), antiallergic (Chaurasia, Sharma, 1982), antitumor (Li et al., 2012), antidiabetic activities (Chiou et al., 1996), coronary vasodilatory (Nagase et al., 2008), histamine receptor type-3 inverse agonism (Pandey, Lohani, 1979), anti cancer (Murugan et al., 2003; Shankar et al., 1984), anti-inflammatory (Shankar et al., 1985; Abdel-Aim's et al., 1994; Mohd, Shalini, 1998; Saravanan et al., 1998; Bhat et al., 2000) anti-tuberculosis (Kumar et al., 1970), CNS depressant activity (Tiwari, Pandey, 1975), anti-

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parkinsonism (Tiwardi, Rastogi, 1978; Srivastava et al., 1986) and bronchodilator activity (Rao, Bahekar, 1999).

	3-(5-phenyl-1, 3, 4-thiadiazol-2-yl)- 2-styrylquinazolin-4(3H)-one (CNS depressant activity)
F <sub>3</sub> C CONH <sub>2</sub> OH	8-(trifluoromethyl)-2, 3-dihydro-5- (4-hydroxyphenyl)-10- phenylimidazo [1, 2-c] quinazoline-7- carboxamide (anti-inflammatory activity)
	2-(2-(3,4,5-trimethoxybenzylidene)- 3-(4-nitrophenylamino)-3, 5, 6, 7, 8, 9-hexahydro-2H-thiazolo[2,3- ]quinazolin-5-yl)phenol (antioxidant activity)
	6-(3-(trifluoromethyl) thiopyran-1- yl) quinazoline-2, 4-diamine (antimalarial activity)
	4-(4-chlorocyclohexa-2,4-dienyl)- 3,4,5,6-tetrahydro-2-morpholino benzo [h]quinazoline (antileishmanial activity)
	6, 8-dibromo-2-phenyl benzoxazines (analgesic activity)

**Table 1.** Some of Quinazolinone derivatives with their biological activities



In view of their importance, many approaches have been developed for the preparation of quinazolinone derivatives. Several methods for the synthesis of 4(3H)-quinazolinones have been investigated in the past. Some common methods include the condensation of 2-aminobenzamides and substituted benzoyl chlorides or their equivalents in ionic liquids (Potewar et al., 2005; Wang et al., 2012), the tandem condensation and C–N cross coupling of 2-halobenzoicacids and amidines (Zhang et al., 2009), the cyclization of o-acylaminobenzamides (Armarego, 1979), 2-aminobenzonitrile (Bogert, Hand, 1902), N-aryl orthanilamides (Stephen, Wadge, 1956; Segarra et al., 1998), nitroenes (Akazome, 1995), and aza-Wittig reactions of a-azido-substituted aromatic imides (Takeuchi et al., 1989; Takeuchi et al., 1991). However, benzyl chlorides are carcinogenic alkylating

agents and poisonous lachrymators and the base K<sub>2</sub>CO<sub>3</sub> is needed to neutralize the hydrochloride produced during the reaction (Adib et al., 2011). Very recently, Ma developed a CuI-4-hydroxy-Lproline 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–ZnCl<sub>2</sub> mediated condensative cyclization (Xu et al., 2012). Unfortunately, both approaches lead to the generation of undesired byproducts, such as hydrogen halide which consumes K<sub>2</sub>CO<sub>3</sub> 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(3H)-quinazolinones in the presence of an equivalent amount of iodine as the catalyst (Dabiri et al., 2010). Recently, a series of Quinazolinone derivatives were synthesised from isatoic anhydride and benzimidamide using BBr<sub>3</sub> as a catalyst (Yedukondalu et al., 2017), and Tandem cyclization of 2-halobenzoic acids with amidines using Cerium(III) chloride as catalyst (Yedukondalu et al., 2017), one-pot reaction using a threecomponent condensation of anthranilic acid, amines, and ortho esters at room temperature under solvent-free conditions (Hari Krishna, Thriveni, 2017). Recently, transition-metal-catalyzed reactions have emerged as versatile tools for the construction of guinazolinones. For example, palladium-catalyzed carbonylation/cyclization cascades turned out to be an efficient approach toward quinazolinone derivatives (He et al., 2014; Wu et al., 2013). However, most of these procedures have some drawbacks, as they have a poor atom economy, use expensive chemicals. 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(3H)-quinazolinones using Silver triflate (10 mol%) as catalyst (Scheme I).

### 2. Materials and methods

Compounds were checked for their purity by TLC on silica gel G plates and spots were located by iodine vapours. The NMR spectra were measured with a 400 MHz Bruker Avance spectrometer at 400.1 and 100.6 MHz and spectra (<sup>1</sup>H NMR and <sup>13</sup>C NMR) were recorded using tetramethylsilane (TMS) in the solvent of  $CDCl_3$ -*d* or DMSO-*d*6 as the internal standard (<sup>1</sup>H NMR: TMS at 0.00 ppm,  $CDCl_3$  at 7.26 ppm, DMSO at 2.50 ppm. <sup>13</sup>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):

The catalyst Silver triflate (10 mol %) was added to a mixture of 2-aminobenzamide (2 mmol) in ethanol and aldehyde (3 mmol) at room temperature under  $N_2$  and the reaction mixture stirred for 4-6hrs. The progress of the reaction was monitored by TLC. After completion, the system was cooled to room temperature. The reaction mixture was diluted with water (15 ml), filter and dried over  $Na_2SO_4$  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 affords the product in 75-88 % yield. All the products were identified by spectral (<sup>1</sup>H NMR, <sup>13</sup>C NMR and mass) and analytical data

Scheme I: The synthetic route was depicted in Scheme I.



synthesis of 2,3-disubstituted 4(3H)-quinazolinones

Spectral data for selected compounds:

# 2,3-dimethylquinazolin-4(3H)-one (3a):



White solid, mp 110-111°C;

<sup>1</sup>**H** NMR ( $CDCl_3$ , 400 MHz):  $\delta$  2.63 (s, 3H), 3.62 (s, 3H), 7.44 (dt, *J*1 = 8.0 Hz, *J*2 =1.2 Hz, 1H), 7.61 (d, *J* = 8.0 Hz, 1H), 7.71 (dt, *J*1 = 8.0 Hz, *J*2 = 1.2 Hz, 1H), 8.25(dd, *J*1 = 8.0 Hz, *J*2 = 1.2 Hz, 1H) ppm;

<sup>13</sup>**C NMR** (CDCl<sub>3</sub>, 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]<sup>+</sup> calcd. for C10H10N2O 175.0866; found 175.0870.

# 3-benzyl-2-(4-oxopentyl)quinazolin-4(3H)-one (3b):

Pale yellow solid, mp 122-124°C;

<sup>1</sup>**H** NMR (CDCl<sub>3</sub>, 400 MHz):  $\delta = 2.04-2.08$  (m, 2H), 2.11 (s, 3H), 2.56 (t, J = 6.8 Hz, 2H), 2.74 (t, J = 7.6 Hz, 2H), 5.49 (s, 2H), 7.19 (d, J = 6.8 Hz, 2H), 7.27-7.36 (m, 3H), 7.47 (dt, J1 = 8.0 Hz, J2 = 0.8 Hz, 1H), 7.65 (d, J = 8.0 Hz, 1H), 7.75 (dt, J1 = 8.4 Hz, J2 = 1.6 Hz, 1H), 8.32 (dd, J1 = 8.0 Hz, J2 = 1.2 Hz, 1H); <sup>13</sup>**C** NMR (CDCl<sub>3</sub>, 100 MHz):  $\delta = 20.9$ , 30.1, 34.1, 42.3, 46.3, 120.4, 126.4, 126.6, 127.0, 127.2,127.6, 128.9, 134.4, 136.3, 147.2, 156.6, 162.6, 208.3;

**HRMS** (ESI): m/z [M+H]<sup>+</sup> calcd. for C20H20N2O2 321.1598; found 321.1605. **2-methyl-3-phenylquinazolin-4(3H)-one (3c):** 



White solid, mp 147-148°C; **'H NMR** (CDCl<sub>3</sub>, 400 MHz): δ = 2.25 (s, 3H), 7.27-7.28 (m, 2H), 7.46-7.59 (m, 4H), 7.68 (d, *J* = 7.6 Hz, 1H), 7.77 (dt, *J*1 = 8.4 Hz, *J*2 = 1.6 Hz, 1H), 8.28 (dd, *J*1 = 8.0 Hz, *J*2 = 1.2 Hz, 1H); **'3C NMR** (CDCl<sub>3</sub>, 100 MHz): δ 24.4, 120.8, 126.7, 126.8, 127.1, 128.0, 129.3, 130.0, 134.6, 137.8, 147.5, 154.2, 162.3; **HRMS** (ESI): m/z [M+H]<sup>+</sup> calcd. for C15H12N2O 237.1022; found 237.1028. 2-methyl-3-(o-tolyl)quinazolin-4(3H)-one (3d):



White solid, mp 119-121°C; **'H NMR** (CDCl<sub>3</sub>, 400 MHz):  $\delta$  = 2.13 (s, 3H), 2.19 (s, 3H), 7.16 (d, *J* = 7.6 Hz, 1H), 7.35-7.42 (m, 3H), 7.48 (t, *J* = 7.6 Hz, 1H), 7.69 (d, *J* = 8.0 Hz, 1H), 7.78 (dt, *J*1 = 8.4 Hz, *J*2 = 1.2 Hz, 1H), 8.29 (dd, *J*1 = 8.0 Hz, *J*2 = 0.8 Hz, 1H);

<sup>13</sup>**C NMR** (CDCl<sub>3</sub>, 100 MHz):  $\delta$  = 17.4, 23.9, 120.7, 126.6, 126.9, 127.1, 127.6, 127.9, 129.6,131.5, 134.6, 135.3, 136.8, 147.6, 154.3, 161.7;

HRMS (ESI): m/z [M+H]<sup>+</sup> calcd. for C16H14N2O 251.1179; found 251.1186.

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

White solid, mp 150-151°C; **'H NMR** (CDCl<sub>3</sub>, 400 MHz):  $\delta$  = 2.25 (s, 3H), 2.45 (s, 3H), 7.14 (d, *J* = 8.0 Hz, 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*1 = 8.0 Hz, *J*2 = 0.8 Hz, 1H), 8.26 (d, *J* = 8.0 Hz, 1H) ppm;

<sup>13</sup>**C NMR** (CDCl<sub>3</sub>, 100 MHz):  $\delta$  = 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]<sup>+</sup> calcd. for C16H14N2O 251.1179; found 251.1185. **3-(4-methoxyphenyl)-2-methylquinazolin-4(3H)-one (3f):** 

JCH2

White solid, mp 169-171°C; **'H NMR** (CDCl<sub>3</sub>, 400 MHz):  $\delta$  = 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), 7.67 (d, *J* = 8.0 Hz, 1H), 7.76 (dt, *J* = 8.4 Hz, *J* = 1.2 Hz, 1H), 8.26 (dd, *J* = 8.0 Hz, *J* = 0.8 Hz, 1H);

<sup>13</sup>**C NMR** (CDCl<sub>3</sub>, 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, 154.9, 159.9, 162.5; **HRMS** (ESI): m/z [M+H]<sup>+</sup> calcd. for C16H14N2O2 267.1128; found 267.1135.

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

White solid, mp 157-158°C;

<sup>1</sup>**H NMR** (CDCl<sub>3</sub>, 400 MHz):  $\delta = 2.25$  (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); <sup>13</sup>**C NMR** (CDCl<sub>3</sub>, 100 MHz):  $\delta = 24.4$ , 120.6, 126.8, 127.1, 129.6, 130.3, 134.8, 135.4, 136.2, 147.4, 153.7, 162.2; **HRMS** (ESI): m/z [M+H]<sup>+</sup> calcd. for C15H11ClN2O 271.0633; found 271.0639.

HRMS (ESI): m/z [M+H]<sup>+</sup> calcd. for C15H11ClN2O 271.0633; found 271.0639 3-(3-chlorophenyl)-2-methylquinazolin-4(*3H*)-one (3h):

White solid, mp 130-131°C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz):  $\delta = 2.27$  (s, 3H), 7.18-7.21 (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, J1 = 8.0 Hz, J2 = 0.8 Hz, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz):  $\delta = 24.4$ , 120.6, 126.6, 126.9, 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]<sup>+</sup> calcd. for C15H11ClN2O 271.0633; found 271.0641. **3-(3,4-dichlorophenyl)-2-methylquinazolin-4(3H)-one (3i):** 

White solid, mp 164-165°C; <sup>1</sup>**H NMR** (CDCl<sub>3</sub>, 400 MHz):  $\delta = 2.26$  (s, 3H), 7.13 (d, J = 8.8 Hz, 1H), 7.41 (s, 1H), 7.46 (t, J = 7.2 Hz, 1H), 7.61-7.68 (m, 2H), 7.77 (t, J = 7.6 Hz, 1H), 8.21 (d, J = 7.6 Hz, 1H) ppm; <sup>13</sup>**C NMR** (CDCl<sub>3</sub>, 100 MHz):  $\delta = 24.3$ , 120.2, 126.9, 126.9, 126.9, 127.6, 130.2, 131.6, 133.9,134.0, 134.9, 136.8, 147.2, 153.1, 161.9 ppm; **HRMS** (ESI): m/z [M+H]<sup>+</sup> calcd. for C15H10Cl2N2O 305.0243; found 305.0252.

### 4. Results and discussion

In our preliminarily investigation on the model reaction of 2-aminobenzamide and aldehyde, it was found that the reaction could be finished under very simple reaction conditions in the presence of Silver triflate as catalyst which gives the desired Quinazolinone product in good yield

2-aminobenzamide **1** with aldehyde **2** in the presence of Silver triflate catalyst proceeded rapidly in ethanol solvent at room temperature to afford **2**,**3**-disubstituted Quinazolinone (Scheme I). Any excess of Silver triflate (10 mol%) beyond this loading did not show any substantial improvement in the yield. So 10 mol% of Silver triflate chosen as the optimal loading of the catalyst. The reaction was carried out at room temperature. The results indicated that a wide range of structurally varied 2-aminobenzamide reacted smoothly to give the Quinazolinone in good yields (scheme **1**). Readily available starting materials and simple synthesizing procedures make this method very attractive and convenient for the synthesis of **2**,**3**-disubstituted Quinazolinone derivatives. Formation of products was confirmed by recording their <sup>1</sup>H NMR, <sup>13</sup>C, mass spetra.

# 5. Conclusion

In conclusion we have developed a simple methodology for the preparation of 2,3disubstituted Quinazolinone derivatives by using Silver triflate (10 mol%) as efficient catalyst. The notable features of this procedure are mild reaction conditions, good yield, enhanced rates and simplicity in operation, which make it a useful and attractive process for the synthesis of 2,3disubstituted quinazolinone derivatives. Thus, the developed methodology could be an alternative for the academic as well as industrial applications.

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