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SILVER TRIFLATE CATALYSED ONE-POT SYNTHESIS OF 3-SUBSTITUTED QUINAZOLINONES BY THREE-COMPONENT COUPLING OF ANTHRANILIC ACID, AMINES AND ORTHO ESTERS AT ROOM TEMPERATURE UNDER SOLVENT-FREE CONDITIONS

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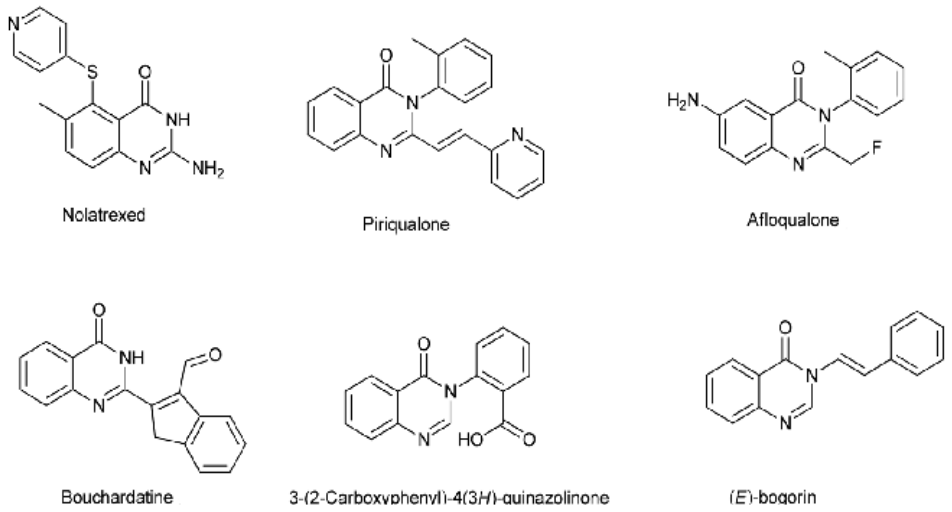
ABSTRACT
A series of 3-substituted quinazolinone derivatives have been synthesized in excellent yields by one-pot reaction using a three-component condensation of anthranilic acid, amines, and ortho esters at room temperature under solvent-free conditions. The reaction was efficiently promoted by AgoTf. All the products were identified by spectral (^{1}H NMR, ^{13}C NMR and mass) and analytical data.

KEYWORDS: One-pot reaction, room temperature, 3-Substituted Quinazolinone, Silver triflate.

INTRODUCTION
The exploration of heterocycles as privileged structures in drug discovery is an important major area in medicinal chemistry.[I] Among them, the quinazolinone ring system is a ubiquitous structural unit and important pharmacophore found in a number of alkaloids and many biologically active compounds. Quinazolinones have emerged as an important class of nitrogenated heterocycles that have attracted significant synthetic interest because of their pharmacological and therapeutic properties like anti-HIV,[II] anti-cancer,[III,IV] anti-tumour,[V] anti-inflammatory,[VI] anti-bacterial,[VII] anti-fungal,[VIII] antihypertensive[IX] and antimalarial activities.[X] etc. A small number of quinazolinones have been reported as potent chemotherapeutic agents in the treatment of tuberculosis. For example, 3-aryl-6,8-dichloro-2H-1,3-benzoxazine-2,4(3H)-diones and 3-arylquinazoline-2,4(1H,3H)-diones as antimycobacterial agents[XI] and quinazolinone derivatives as antitubercular agents.[XII] The antihyperlipidemic activities of these compounds were also investigated.[XIII] In addition to their occurrence in natural products, they also frequently appear in pharmaceutical agents for their applications as potent antagonistic receptors.[XIV] For example, Pegamine, isolated from *Peganum harmala*, has been found to possess cytotoxic activity.[XV] A few illustrative examples of quinazolinones core that show various pharmacological activities are listed in

Figure 1.



Synthesis of 3-Substituted Quinazolinone derivatives has been extensively investigated.[XVI] Quinazolinone derivatives have become especially noteworthy in recent years due to their wide spectrum of biological activity.[XVII–XXIII] On the basis of biological and medicinal importance, the synthesis and bioactivity of the quinazolinone nucleus have got impetus along with the chemists and biologists in recent years. Owing to the excellent biological properties of 3-Substituted Quinazolinone derivatives, particularly, 3-substituted quinazolinone, numerous synthetic methods have been found in the book chapters and in recent literature reviews.[XXIV, XXV]

Several methods involve cyclization of *o*-acylaminobenzamide,[XXVI] amination of benzoxazin-4-one,[XXVII] multicomponent reactions (MCRs) among isatoic anhydride, amine with aldehyde,[XXVIII] benzyl halide[XXIX] or orthoester.[XXX] In addition, transition metal catalysts such as copper,[XXXI] ruthenium,[XXXII] iridium[XXXIII] and palladium[XXXIV] have also been applied to the synthesis of quinazolinones through a one-pot oxidative cyclization of primary alcohols with *o*-aminobenzamides as well as through a domino process of *N*-arylation followed by condensative[XXXV] cyclization. Another extensively studied strategy was a sometimes metal-free, one-pot protocol which involves a cyclocondensation of anthranilamides with aldehydes followed by a subsequent oxidant-mediated dehydrogenation process.[XXXVI] Various non-metal oxidants such as DDQ,[XXXVIIa] I_2, [XXXVIIf] O_2 [XXXVIIb] as well as metal ones such as $CuCl_2$, [XXXVIIc] $KMnO_4$,[XXXVIIe] have been applied to realize the second oxidative step. There are several methods for the synthesis of quinazolinones.[XXXVIII] Recently, Das et al., have demonstrated a green process for the synthesis of 3-substituted quinazolinone, which is limited in that only phenyl R groups are tolerated.[XXXIX] More recently, Liu et al. reported another procedure for the synthesis of these compounds through a two-step reaction in the presence of $P(PhO)_3$/anhydrous pyridine under microwave irradiation at 250^0C.[XL]

Although these strategies has some practical advantages and potential applications, many of the existing methods have the disadvantages such as long reaction times, low yields, harsh reaction conditions, difficult work-up and use of environmentally toxic reagents or media. The demand for increasingly clean and efficient chemical synthesis is important from both economic and environmental points of view.[XL1] In this communication, we report a novel

protocol for the selective synthesis of 3-substituted quinazolin-4(3H)-ones by using silver triflate as effective catalyst.

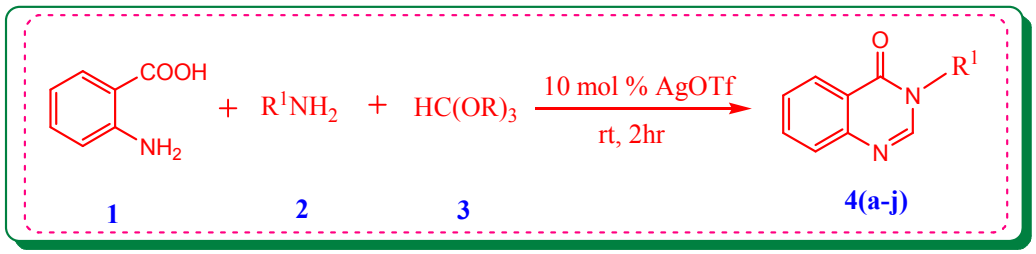
MATERIALS AND METHODS

Compounds were checked for their purity by TLC on silica gel G plates and spots were located by iodine vapors. The NMR spectra were measured with a 400 MHz Bruker Avance spectrometer at 400.1 and 100.6 MHz and 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 3-Substituted Quinazolinone derivatives 4(a-j):

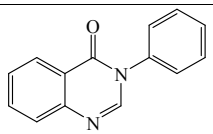
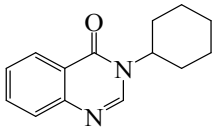
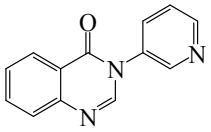
A mixture of anthranilic acid **1** (10 mmol), an amine **2** (12 mmol), an orthoester **3** (12 mmol), and AgOTf (1 mmol, 10 mol %) was stirred at room temperature for 2hrs. The progress of the reaction was monitored by TLC. After completion, the system was cooled to room temperature. The product **4** was obtained through simple filtering, and recrystallized from ethanol. All the products were identified by spectral (^{1}H NMR, ^{13}C NMR and mass) and analytical data.

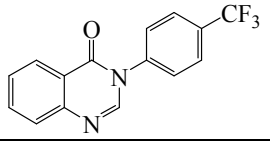
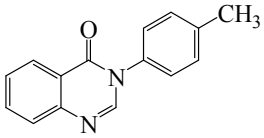
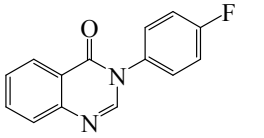
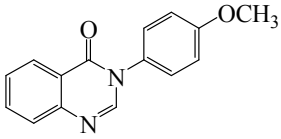
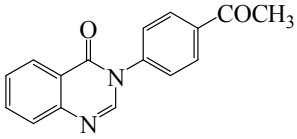
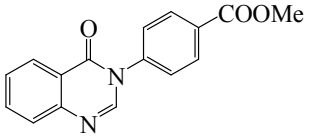
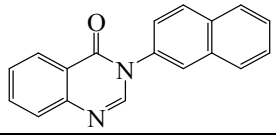
Scheme I: The synthetic route was depicted in Scheme I.



Synthesis of 3-Substituted Quinazolinone derivatives

Table 1: Synthesis of 3-Substituted Quinazolinone derivatives 4(a-j)

Entry	Product	Yield(%)
1		84
2		81
3		84

4		87
5		85
6		86
7		84
8		85
9		85
10		88

Spectral data for selected compounds:

3-Phenylquinazolin-4(3*H*)-one: (3a)

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.38 (ddd, J = 8.0, 1.5, 0.7 Hz, 1H), 8.14 (s, 1H), 7.89 –7.73 (m, 2H), 7.61–7.48 (m, 4H), 7.46–7.38 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.63, 147.76, 145.98, 137.38, 134.48, 129.53, 129.01, 127.54, 127.37, 127.06, 126.90, 122.32.

3-Cyclohexylquinazolin-4(3*H*)-one (3b):

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.31 (d, *J* = 8.0 Hz, 1H), 8.10 (s, 1H), 7.74-7.68 (m, 2H), 7.48 (t, *J* = 7.6 Hz, 1H), 4.82-4.76 (m, 1H), 1.99-1.90 (m, 4H), 1.78 (d, *J* = 13.2 Hz, 1H), 1.67-1.58 (m, 2H), 1.54-1.45 (m, 2H), 1.28-1.19 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.77, 147.61, 143.99, 134.17, 127.34, 127.16, 127.03, 122.03, 53.45, 32.68 (2C), 25.99 (2C), 25.37.

3-(Pyridin-3-yl)quinazolin-4(*3H*)-one (3c):

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.74 (d, *J* = 6.4 Hz, 2H), 8.36 (d, *J* = 7.6 Hz, 1H), 8.12 (s, 1H), 7.86-7.76 (m, 3H), 7.57-7.51 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.69, 150.25, 147.86, 147.63, 145.27, 135.09, 134.92, 128.24, 127.89, 127.29, 124.16, 124.07, 122.17.

3-(4-(Trifluoromethyl)phenyl)quinazolin-4(3*H*)-one (3d):

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.36 (ddd, *J* = 8.0, 1.5, 0.6 Hz, 1H), 8.10 (s, 1H), 7.87 –7.72 (m, 4H), 7.64 – 7.51 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.27, 147.61, 145.07, 140.36, 134.85, 131.19, 128, 127.66, 127.47, 127.14, 126.77, 123.50, 122.07.

3-*p*-Tolylquinazolin-4(3*H*)-one (3e): ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.37 (d, *J* = 8.0 Hz, 1H), 8.11 (s, 1H), 7.82-7.49 (m, 2H), 7.58 (t, *J* = 7.2 Hz, 1H), 7.36-7.28 (m, 4H), 2.43 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.02, 148.04, 146.42, 139.37, 135.05, 134.63, 130.37, 127.69, 127.31, 126.86, 122.54, 21.34.

3-(4-Fluorophenyl)quinazolin-4(3*H*)-one (3f): ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.36 (d, *J* = 7.6 Hz, 1H), 8.09 (s, 1H), 7.82-7.76 (m, 2H), 7.57 (t, *J* = 7.6 Hz, 1H), 7.43-7.21 (m, 2H), 7.26 (t, *J* = 8.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.97, 160.91, 147.95, 145.96, 134.82, 133.53, 129.08 (d, *J* = 8.7 Hz, 2C), 127.90, 127.77, 127.28, 122.38, 116.92.

3-(4-Methoxyphenyl)quinazolin-4(3*H*)-one (3g): ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.37 (dd, *J*1 = 0.4 Hz, *J*2 = 8.0 Hz, 1H), 8.12 (s, 1H), 7.80-7.77 (m, 2H), 7.56 (t, *J* = 6.4 Hz, 1H), 7.34 (d, *J* = 9.2 Hz, 2H), 7.05 (d, *J* = 8.8 Hz, 2H), 3.87 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.03, 148.00, 147.45, 146.59, 134.66, 131.27, 128.27, 127.72, 127.65, 127.29, 122.47, 114.96, 55.74.

3-(4-Acetylphenyl)quinazolin-4(3*H*)-one (3h):

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.33 (d, J=8.8, 1H), 8.11 (s, 1H), 8.09-7.97 (m, 2H), 7.84-7.49 (m, 5H), 2.62 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.52, 160.50, 147.78, 145.42, 138.42, 137.81, 134.72, 131.47, 129.87, 128.79, 127.78, 127.61, 127.04, 126.68, 122.08, 26.63.

Methyl 4-(4-oxoquinazolin-3(4*H*)-yl)benzoate (3i):

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.38 (dd, *J*1 = 1.2 Hz, *J*2 = 8.0 Hz, 1H), 8.24-8.21 (m, 2H), 8.12 (s, 1H), 7.86-7.77 (m, 2H), 7.59-7.53 (m, 3H), 3.97 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.10, 160.59, 147.79, 145.44, 141.32, 134.98, 131.11, 130.86, 128.06, 127.81, 127.35, 127.12, 122.31, 52.63.

3-(Naphthalen-2-yl)quinazolin-4(3*H*)-one (3j):

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.42 (d, *J* = 7.6 Hz, 1H), 8.21 (s, 1H), 8.04-7.81 (m, 6H), 7.59-7.52 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.07, 148.01, 146.34, 135.19, 134.73, 133.45, 133.13, 129.68, 128.23, 127.98, 127.80, 127.73, 127.36, 127.27, 127.17, 125.73, 124.74, 122.49.

RESULTS AND DISCUSSION

In our preliminarily investigation on the model reaction, it was found that the reaction could be finished under very simple reaction conditions in the presence of Silver triflate which gives the desired 3-Substituted Quinazolinone derivatives in good yield. Silver triflate can efficiently catalyze a one-pot synthesis of 3-substituted quinazolinones via a three-component condensation of anthranilic acid, amines, and ortho esters (**Scheme 1**). The reaction was carried out at room temperature under solvent-free conditions and the results are summarized in **Table 1.**

CONCLUSION

The present work concludes that AgOTf has been employed as a novel and efficient catalyst for the synthesis of 3-Substituted Quinazolinone derivatives. 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 3-substituted quinazolinone derivatives.

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