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# Palladium-catalyzed synthesis of novel trifluoromethylated quinazolinone, *N*-arylquinazoline and *N*-benzylquinazoline derivatives

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# ABSTRACT

A simple and palladium-catalyzed procedure for synthesis of a novel series of potentially biologically active trifluoromethyl-substituted quinazolinones and *N*-arylquinazoline derivatives via condensation-cyclization reaction of 2-aminobenzamide, 2-amino-N'-arylbenzimidamides and 2-amino-N'-benzylbenzimidamides with trifluoroacetimidoyl chlorides has been developed. noteworthy, this investigation showed the possible of transition-metal-catalyzed activation of trifluoroacetimidoyl chlorides as a carbon trifluoromethylated source for the synthesis of quinazolines and quinazolinone derivatives in good to excellent yields.

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Quinazoline and quinazolinones form a main group of pharmaceutical heterocyclic derivatives that have a considerable place in medicinal and pesticide chemistry [1], like analgesic, anti-inflammatory, anti-hypertensive, anti-tubercular, anti-bacterial and anti-viral activities [2]. Also, quinazolines are extensively used as sedative [3a], anti-bacterial [3b,c], anti-diabetic [3d], anti-inflammatory [3e], anti-viral [3f] and anti-tumor agents [3g].

4-Anilinoquinazoline derivatives are a group of potentially high selective anti-cancer reagents for their powerful ability to prevent several receptor tyrosine kinases, such as EGFR, NGFR, and VEGFR-2, which often over-expresses or deregulates in many solid tumors [4]. For example, compounds A and B, establish a main subgroup of kinase inhibitor anti-cancer reagents and exhibited dihydrofolate reductase inhibition respectively. Also, KSP ATPase inhibitor C shows antimitotic activity and now is in phase II clinical trials as a potential anti-cancer chemotherapeutic factor, while methaqualone D is a clinically used sedative hypnotic drug (Fig. 1) [5]. According to the above and from our point of view, fluorinated quinazolinones and quinazoline derivatives are very more important relative to the non-fluorinated quinazolinones and quinazolines due to the fluorine atom effects. Many synthetic efforts have been made for its construction starting from a variety of substrates

\* Corresponding author. E-mail address: darehkordi@vru.ac.ir (A. Darehkordi). [6], among which 2-aminobenzamide is probably the most typical one.

One of the general procedure for the synthesis of quinazolinone derivatives is commonly reaction of 2-aminobenzamide with either carboxylic acid derivatives under harsh conditions [7], or aldehydes and followed by oxidation using strong oxidants [8]. However, in this procedure in general excess amounts of oxidants should be used and produces a large amount of waste, therefore, requiring tedious purification processes [9]. To overcome these drawbacks such as low yields, multistep reactions, or harsh reaction conditions, latter investigations gave improved methods using Vilsmeier reagent, which are more eco-friendly, without any by-products [10]. On the other hand heterogeneously-catalyzed processes have a notable role in prevent of the harmful process, declining the waste manufactures, avoiding the calamitous solvents usage, and crude catalysts separation and recycling [9].

For example, *N*-substituted quinazolinones have been synthesized from anthranilamide derivatives using dicumyl peroxide (DCP) as the methyl source. This reaction is a tandem *N*-methylation-sp<sup>3</sup> C—H amination-oxidation process with use of a copper catalyst [6]. Also, the Ding and Fu groups individually reported that copper-catalyzed *N*-arylation of *o*-bromobenzoic acid derivatives with amidines and following that intramolecular reaction could produce 2-substituted and 2,3-disubstituted quinazolinones [11].

Quinazolinones have been synthesized from reaction of 2aminobenzamide, aryl bromides, and carbon monoxide in present







Fig. 1. Structures of some biologically important 4-anilinoquinazolines and quinazolinones.

of palladium as a catalyst in good yields [12]. Also, palladiumcatalyzed three-component carbonylative reaction of trifluoroacetimidoyl chlorides and amines resulted to synthesis of 2-(trifluoromethyl)quinazolin-4(3H)-one derivatives [13].

Reaction 4-halo- or 4-mercaptoquinazolines with aromatic amines [14], 4(3H)-quinazolone with aromatic amine hydrochlorides in the presence of phosphorus pentoxide and dimethylcyclohexylamine [15], desulfurization of 4-phenylaminoquinazol-2thione using Raney nickel [16], and 2-aminobenzonitrile with aniline derivatives using AlCl<sub>3</sub>, and then reaction of the products with formic acid [17] are some of the approaches reported for synthesis of substituted 4-aryl amino quinazolines.

Fluorine is an element which has very more important role in pharmaceutical and organic chemistry, because, it donates special characteristics to the organic compounds and therefore change their physicochemical and biological properties [18]. The trifluoromethyl substituent ( $CF_3$ ) is one of the most prevalent fluorinated groups in drug chemistry, agricultural, and material sciences [19]. Because it provides simultaneously high lipophilicity, a high electron density and a steric demand similar to that of the isopropyl group [20]. recently, synthesis of new organofluorine compounds via reactions of trifluoromethylation have been extensively investigated [21].

Some drugs can results side effects such as gastric irritation, ulceration, and hemorrhage. In this regard, the functionalized of molecule with fluorine atoms has emerged as a reliable method to eliminate the harmful mentioned. The presence of fluorine atoms changes the physical-chemical properties of the molecules to which they are attached, thus modify cell permeability and decrease metabolic degradation, and also result to a longer half-life [22].

Due to the above mentioned and in follow of our studies interest on synthesis of trifluoromethylated heterocycles via palladium catalyzed reactions [23], quinazolinone and quinazoline derivatives [24,25], herein, we report palladium-catalyzed synthesis of trifluoromethylated quinazolinones and quinazoline derivatives from reaction 2-aminobenzamide and 2-amino-N'-arylbenzimidamides or 2-amino-N'-benzylbenzimidamides with trifluoroacetimidoyl chlorides with use of Pd(PPh<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub> and Et<sub>3</sub>N respectively (Scheme 1).

2,2,2-Trifluoro-*N*-arylacetimidoyl chlorides **1** have been synthesized by reaction of trifluoroacetic acid, primary aryl amines, and triphenylphosphine in  $CCl_4$  and triethylamine. Work-up and distillation of the mixture reaction gave the target trifluoroacetimidoyl chlorides in good to excellent yields (Scheme 2) [21].

Then in other to the synthesis of quinazolinone derivatives, we carried out condensation-cyclization reaction between 2aminobenzamide **2a** and trifluoroacetimidoyl chlorides **1**. To gain the optimized conditions, reaction of 2,2,2-trifluoro-N-(p-tolyl)



**Scheme 1.** Synthesis of 3-aryl-2-(trifluoromethyl)quinazolin-4(3H)-one 3 and *N*-aryl-2-(trifluoromethyl)quinazolin-4-amine or *N*-benzyl-2-(trifluoromethyl)quinazolin-4-amine derivatives **5**.



Scheme 2. Preparation of 2,2,2-trifluoroacetimidoyl chloride derivatives 1.

acetimidoyl chloride **1a** and 2-aminobenzamide **2a** was selected as the model reaction. Different base, catalysts and solvents were used at room temperature and reflux conditions. The results have been showed in Table 1.

Without use of any catalyst, with both room temperature and reflux conditions in presence of various solvents and base, no desired 3-(p-tolyl)-2-(trifluoromethyl)quinazolin-4(3H)-one 3a was observed after 24 h, based on the TLC (*n*-Hexane: EtOAc 3:1) (Table 1, entries 1–3 and 5–6). To evaluate the catalytic activity of TiO<sub>2</sub> for the synthesis of quinazolinone, catalytic amounts of TiO<sub>2</sub> were added to the reaction mixture and only a trace amount of 3a was obtained in presence of NaH or Et<sub>3</sub>N in CH<sub>3</sub>CN under reflux conditions (Table 1, entry 7-8). But, the yield of the desired product 3a was significantly improved in shorter reaction time when catalytic amounts (5 mol%) of Pd(PPh<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub> were added to the reaction mixture in presence of Et<sub>3</sub>N in CH<sub>3</sub>CN under reflux conditions (Table 1, entry 4). Therefore, the optimal reaction conditions are 1 equiv of 1a and 1 equiv 2a in the presence of 5 mol % Pd(PPh<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub>, 2 mmol Et<sub>3</sub>N in CH<sub>3</sub>CN at the reflux conditions for 3 h (Table 1, entry 4). With determining of optimized reaction conditions, the scope and generality of reaction were investigated and the representative results are shown in table 2. The reactions carried out smoothly under optimized conditions. After the end of reaction (monitored by TLC), the catalyst was easily removed by filtration. Therefore, a simple work-up produced the target products **3a-f** in good to excellent yields.

Also, in order to expanding the scope of reaction, in addition of 2-aminobenzamide **2a**, 2-amino-*N*-phenylbenzamide **2b**, 2-amino-*N*-methylbenzamide **2d**, examined under the optimized conditions. In these cases also we did not see significant difference in reaction time, yield and elimination of amine group, resulted to formation of 3-aryl-2-(trifluoromethyl) quinazolin-4(3H)-one derivatives **3a-f** (Scheme 3).

In order to the investigation of electronic effects on the reaction time and yield, various imidoyl chlorides contains electron withdrawing and electron donating groups, were used (Table 2). The reaction proceeds without significant difference in reaction time or yield. Therefore, with suitable reaction conditions, synthesis of

#### Table 1

Optimization of conditions reaction for synthesis the 3-(p-tolyl)-2-(trifluoromethyl)quinazolin-4(3H)-one 3a.



Entry	Catalyst	Base	Solvents	T(°C)	Yield (%)
1	-	Et₃N	Toluene	rt	-
2	-	Et <sub>3</sub> N	Toluene	reflux	-
3	-	Et₃N	THF	rt	-
4	$Pd(PPh_3)_2Cl_2^a$	Et₃N	CH₃CN	reflux	85
5	-	K <sub>2</sub> CO <sub>3</sub>	CH₃CN	rt	-
6	-	K <sub>2</sub> CO <sub>3</sub>	CH₃CN	reflux	-
7	TiO <sub>2</sub> <sup>b</sup>	Et₃N	CH <sub>3</sub> CN	reflux	Trace
8	TiO <sub>2</sub> <sup>c</sup>	NaH	CH <sub>3</sub> CN	reflux	Trace

<sup>a</sup> Conditions: **1a** (1 mmol), **2a** (1 mmol) Et<sub>3</sub>N (2 mmol) and Pd(PPh<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub> (5 mol %) in CH<sub>3</sub>CN at reflux.

<sup>b</sup> Conditions: **1a** (1 mmol), **2a** (1mmmol) Et<sub>3</sub>N (2 mmol) and TiO<sub>2</sub> (5 mol %) in CH<sub>3</sub>CN at reflux.

<sup>c</sup> Conditions: **1a** (1 mmol), **2a** (1mmmol) NaH (2 mmol) and TiO<sub>2</sub> (5 mol %) in CH<sub>3</sub>CN at reflux.

Table 2

Synthesis of 3-phenyl-2-(trifluoromethyl)quinazolin-4(3H)-one derivatives 3a-f under optimized conditions.<sup>a</sup>





 $^a$  Conditions: 1 (1 mmol), 2a (1 mmol)  $Et_3N$  (2 mmol) and  $Pd(PPh_3)_2Cl_2$  (5 mol%) in CH\_3CN at reflux.



Scheme 3. Synthesis of 3-phenyl-2-(trifluoromethyl)quinazolin-4(3H)-one derivatives 3a-f using 2-aminobenzamides 2.

trifluoromethyl-substituted quinazolinone could be accessed by this transformation in good to excellent yields.

A possible mechanism for this reaction is proposed in Scheme 4.

According to the obtained results, the above mechanism can be proposed for the synthesis of trifluoromethyl-substituted quinazolinone derivatives from reaction of the 2-aminobenzamide **2a** and trifluoroacetimidoyl chlorides **1** in the presence of  $Pd(PPh_3)_2$ -Cl<sub>2</sub>, as a heterogeneous catalyst (Scheme 4). According to the literature [26–29], firstly, Pd(II) becomes to Pd(0) which activates the trifluoroacetimidoyl chlorides **1a-f** to produce palladium imidoyl complex **A**. Then this activated complex reacts with 2-aminobenzamide **2a** and produce organopalladium compound **B**. In continue, organopalladium compound **B** under a reduction-elimination reac-



**Scheme 4.** A possible mechanism for the synthesis of 3-aryl-2-(trifluoromethyl) quinazolin-4(3H)-one derivatives (**3a-f**).



**Scheme 5.** Synthesis of 2-amino-N'-phenylbenzimidamide and 2-amino-N'-ben-zylbenzimidamides derivatives **4**.



Scheme 6. Synthesis of N-(p-tolyl)-2-(trifluoromethyl)quinazolin-4-amine 5a

tion results to 2-(2,2,2-trifluoro-*N*'-arylacetimidamido)benzamide **C** which tautomerizes to compound **D**. Finally, intermolecular nucleophilic attack of NH- group and elimination of NH<sub>3</sub> molecule, to afford the desired 3-aryl-2-(trifluoromethyl)quinazolin-4(3H)- one derivatives **3a-f**.

Possessing accepted a new method for synthesis of trifluoromethylated arylquinazolinones **3a-f** from the reaction of 2-aminobenzamide **2a** with imidoyl chlorides **1**, we were then investigated in whether this procedure could be used in the synthesis of *N*-aryl-2-(trifluoromethyl)quinazolins or *N*-benzyl-2-(trifluoromethyl)quinazolines by replacing 2-aminobenzamide **2a** with 2-amino-N'-arylbenzimidamides or 2-amino-N'-benzylbenz-imidamides **4a-1**. For this purpose, in the first, 2-amino-N'-arylbenzimidamides or 2-amino-N'-benzylbenz-imidamides or 2-amino-N'-benzylbenzimidamides **4a-1**. are obtained via reaction of *o*-aminobenzonitrile and aniline or benzyl amine derivatives in the presence of AlCl<sub>3</sub> at 150–180 °C according to the literature reported (Scheme 5) [30].

To examine our idea, firstly, we reacted 2-amino-N'-(p-tolyl) benzimidamide **4a** and N-(3,4-dimethylphenyl)-2,2,2-trifluoroacetimidoyl chloride **1d** in presence of Pd(PPh<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub> as catalyst and Et<sub>3</sub>N base in CH<sub>3</sub>CN under reflux conditions (Same conditions for synthesis of quinazolinones). After elimination of arylamine molecule related to N-(3,4-dimethylphenyl)-2,2,2-trifluoroacetimidoyl chloride **1d**, N-(p-tolyl)-2-(trifluoromethyl)quinazolin-4-amine **5a** was produced (Scheme 6).

As mentioned in Scheme 4 quinazolinone derivatives **3a-f** were obtained from reaction of 2-aminobenzamide with 2,2,2-trifluoroacetimidoyl chlorides after elimination of NH<sub>3</sub> molecule. In this reaction NH3 molecule eliminates from 2-aminobenzamide due to the electron-withdrawing effect of carbonyl group. But when used 2-amino-N'-arylbenzimidamides or 2-amino-N'-benzylbenzimidamides **4** instead of 2-aminobenzamide **2a**, 3,4-dimethylaniline group related to N-(3,4-dimethylphenyl)-2,2,2-trifluoroacetimidoyl chloride **1d** was eliminated and therefore arylaminequinazolines or benzylaminequinazolines were produced. In order to generalize the optimum conditions and method, various 2-amino-N'-arylbenzimidamide or 2-amino-N'-benzylbenzimidamides **4** were used as substrates.

The results summarized in table 3, indicate the effect of the substituent attached on the *N*-aryl or *N*-benzyl rings ( $R_3$ ) in this reaction. As shown in Table 3, 2-amino-N'-arylbenzimidamide or 2-amino-N'-benzylbenzimidamide derivatives **4** bearing Me, OMe, Et, 3,4-di Me, 2,4,6-tri Me and 2-Me on the phenyl and 4-Me on the benzyl moiety reacted at shorter time and higher yield.

A possible mechanism for this reaction is proposed in Scheme 7.

#### Table 3

Synthesis of *N*-aryl-2-(trifluoromethyl)quinazolin-4-amine and *N*-benzyl-2-(trifluoromethyl)quinazolin-4-amine derivatives under optimized conditions.<sup>a</sup>



Compound	R <sub>3</sub>	Product	Yield (%)
5a	4-Me-Ph	NH	87
5b	4-OMe-Ph	MeO CF <sub>3</sub>	87
		NH NH	
5c	3,4-di Me-Ph	N CF3	80
5d	3-Et-Ph	N N CF3	85
		NH	
5e	4-F-Ph	F CF3	75
5f	4-Cl-Ph	CI NH	78
5g	4-Br-Ph	Br	80
-		NH	
5h	3-Br-Ph	Br NH	80
51	2 Me Ph	N N CF <sub>3</sub>	80
51	2-1VIC-1'II	NH	ου

Table 3 (continued)



 $^a$  Conditions: 1d (1 mmol), 4 (1 mmol) Et\_3N (2 mmol) and Pd(PPh\_3)\_2Cl\_2(5 mol%) in CH\_3CN at reflux.



**Scheme 7.** A possible mechanism for the synthesis of *N*-aryl-2-(trifluoromethyl) quinazolin-4-amine derivatives **5**.

According to the literature [25–28], firstly, Pd(II) becomes to Pd (0) which activates the trifluoroacetimidoyl chlorides **1d** to produce palladium imidoyl complex **A**. Then this activated complex reacts with 2-amino-N'-arylbenzimidamide or 2-amino-N'-benzylbenzimidamides **4** and produce organopalladium compound **B**. Then, organopalladium compound **B** under a reduction-elimination reaction results to intermediate **C** which tautomerizes to compound **D**. Finally, intermolecular nucleophilic attack of second amino group and then elimination of 3,4-dimethylaniline group, to afford the desired *N*-aryl-2-(trifluoromethyl)quinazolin-4-amine derivatives **5a-I** [25,29].

In summary, we have demonstrated an efficient and direct method for the synthesis of structurally diverse and biologically important novel trifluoromethylated quinazolinone, *N*-arylquinazoline and *N*-benzylquinazoline derivatives via Palladium-catalyzed cyclocondensation of trifluoroacethymidoyl chlorides with 2-aminobenzamide, 2-amino-N'-arylbenzimidamides or 2-amino-N'-benzylbenzimidamides in good to excellent yields. The synthetic protocol features good functional group tolerance, and has potential to be used to synthesis of biologically active molecules.

# **Declaration of Competing Interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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#### Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.tetlet.2021.153053.

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