Catalyst-free, one pot and three-component synthesis of 4'-phenyl-1'H-spiro[indoline-3,2'-quinazolin]-2-ones and 2,4-diphenyl-1,2-dihydroquinazolines

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Graphical Abstract

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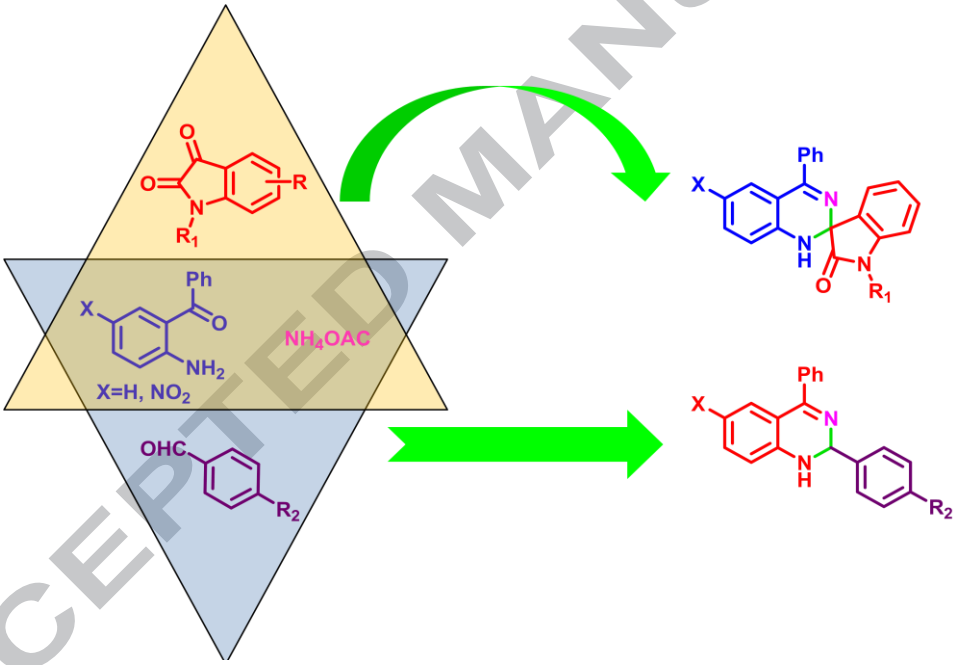

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Catalyst-free, one pot and three-component synthesis of 4'-phenyl-1'H-spiro[indoline-3,2'-quinazolin]-2-ones and 2,4-diphenyl-1,2-dihydroquinazolines

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ABSTRACT

A simple, green, efficient and three-component procedure has been developed for the synthesis of 4'-phenyl-1'H-spiro[indoline-3,2'-quinazolin]-2-ones (**4a-m** and **6a-g**) and 2,4-diphenyl-1,2-dihydroquinazolines (**8a-l**) by the reaction of 2-aminobenzophenones, isatins or aromatic benzaldehydes and ammonium acetate in excellent yields under catalyst-free conditions using ethanol as solvent. This method provides several advantages such as operational simplicity, higher yields, shorter reaction time and catalyst-free conditions with ethanol as a solvent makes the method eco-friendly as well as economical. All the 4'-phenyl-1'H-spiro[indoline-3,2'-quinazolin]-2-ones were tested for antimicrobial activity against both Gram-positive and Gram-negative bacterial strains including a fungal strain *Candida albicans* MTCC 3017. Among these, the compounds **4f**, **6a**, **6c** and **6g** showed appreciable antibacterial activity with MIC values 7.8 µg/ml selectively against Gram-positive bacteria, *Micrococcus luteus* MTCC 2470. On the other hand, compounds **4j**, **4m**, **6c** and **6g** showed good activity with MIC values ranging between 3.9 and 7.8 µg/ml against Gram-negative bacteria, *Klebsiella planticola* MTCC 530.

1. Introduction

Quinazolines are an important class of heterocycles found in a wide range of natural products and pharmaceuticals and exhibit several biological activities including antibacterial,[1] antitumor,[2] antiplasmodial,[3] anti-inflammatory,[4] antiviral[5] and anti-oxidant[6] activities, apart from their usage as photo-chemotherapeutic agents,[7] DNA-gyrase, PDE5, EGFR tyrosine kinase inhibitors,[8] T-type calcium channel[9] and CB2 receptor are familiar targets of various quinazoline derivatives.[10] This scaffold is also the building block for many naturally occurring alkaloids such as Bouchardatia neurococca, Bacillus cereus, Peganum nigellastrum and Dichroa febrifuga.[11]

The spirooxindole unit is a privileged heterocyclic moiety present in a large number of alkaloids and natural products such as spirotryprostatin A and B. These two natural alkaloids isolated from the fermentation broth of Aspergillus fumigatus, has been identified as inhibitors of microtubule assembly,[12] pteropodine act as positive modulators of muscarinic M(1) and 5-HT(2) receptors[13] and alstonisine (figure 1). A spirooxindole system is also a core scaffold of many synthetic pharmaceuticals with a wide range of biological applications such as antimicrobial,[14] antitumor,[15] antibiotic[16] and inhibitors of the human NK-1 receptor.[17] Due to the significant biological activity of these spiroxindoles, it is required to find new and simple synthetic methods for the preparation of new substituted spirooxindoles.

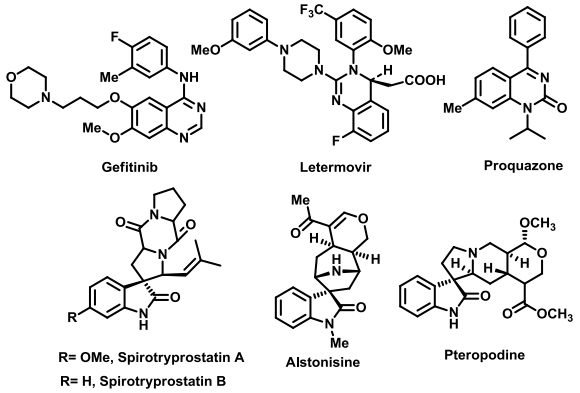


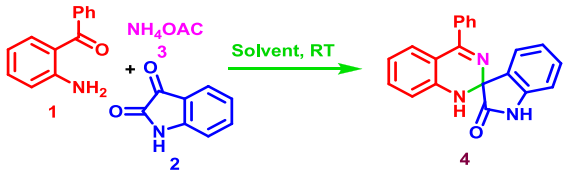
Figure 1. Biologically and pharmaceutically important quinazoline and spirocyclic oxindoles

Owing to extensive applications of diverse quinazoline derivatives in various fields, several methods have been reported such as copper-catalyzed cascade coupling of 2-bromobenzaldehyde with acetamidine hydrochloride,[18a] copper-catalyzed Ullmann N-arylation coupling,[18b,c] photochemical method,[18d] tandem reaction from 2-aminobenzophenones and benzylic amines,[18e,f] maltose-urea-NH_4Cl mixture as a solvent synthesis,[18g] copper-catalyzed alkynylation and cyclization of N-phenylbenzamidines,[18h] the condensation of aldehydes with 2-aminobenzylamine using sodium hypochlorite[18i] or MnO_2[18j] as

oxidant and microwave promoted synthesis.[18k,l,m,n] Inspite of their remarkable biological activities, only few methods for the synthesis of 4'-phenyl-1'H-spiro[indoline-3,2'-quinazolin]-2-one derivatives have been reported in the literature.[19] However, these methods suffer from one or more disadvantages such as use of catalysts, lower yields and difficult operation.

In the course of our interest to develop environmentally benign methods for the synthesis of bioactive compounds[20] we herein report a facile synthesis of 4'-phenyl-1'H-spiro[indoline-3,2'-quinazolin]-2-ones and 2,4-diphenyl-1,2-dihydroquinazolines using 2-amino benzophenones, isatins or aromatic benzaldehydes and ammonium acetate in ethanol under catalyst-free conditions.

2. Results and discussion



Scheme 1. Model Reaction

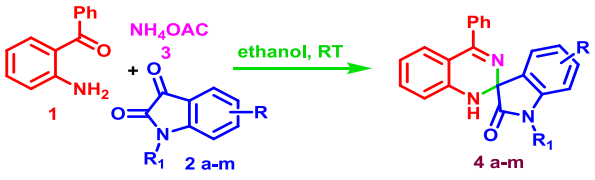
For the synthesis of 4'-phenyl-1'H-spiro[indoline-3,2'-quinazolin]-2-one (**4**), 2-amino benzophenone (**1**), isatin (**2**) and ammonium acetate (3) were selected as model reactants during the optimization process. Initially, this transformation was carried out in water without any catalyst at room temperature and under refluxing conditions and the reaction was monitored by TLC (Scheme 1). It was observed that the reaction did not proceed even until 360 min (Table 1, entries 1 and 2). When the same reaction was carried out using various solvents such as CH3CN, MeOH and EtOH at room temperature, the desired transformation was accomplished in the good yields 62%, 90% and 95%, respectively (Table 1, entries 3-5). Therefore, EtOH was found to be a suitable solvent that provides higher yields (95%) in 50 min at room temperature and further increase of time did not improve the yields. The high yield obtained (95%) in EtOH prompted its selection for catalyst-free conditions.

Table 1. Optimization of reaction conditions.

Entry	Solvent	Temperature (°C)	Time (min)	Yield[a] (%)
1	water		360	--
2	water	reflux	360	--
3	CH_3CN	rt	60	62
4	MeOH	rt	60	90
5	EtOH	rt	50	95

[a]isolated yields

The scope and generality of the present protocol was then examined by employing various substituted isatins (Scheme 2) and the results are summarized in Table 2. The reaction tolerates both electron withdrawing as well as donating substituents on the isatin component without any significant deviation in yields (entries 4b-j). Similarly the N-protected isatins also afforded the corresponding products in excellent yields (entries **4k-m**).

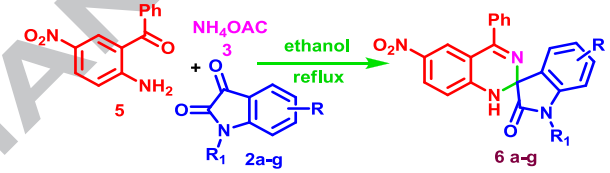


Scheme 2. Synthesis of 4'-phenyl-1'H-spiro[indoline-3,2'-quinazolin]-2-one derivatives

Table 2. Synthesis of 4'-phenyl-1'H-spiro[indoline-3,2'-quinazolin]-2-one derivatives.

Entry	R	R_1	Time (min)	Yield[a] (%)
4a	H	H	50	94
4b	5-F	H	60	92
4c	7-F	H	60	92
4d	5-Cl	H	60	91
4e	5-Br	H	60	88
4f	5-I	H	60	87
4g	5-Me	H	60	88
4h	5,7-Di Me	H	90	84
4i	5-OMe	H	70	87
4j	5-NO_2	H	60	92
4k	H	Benzyl	70	88
4l	5-Cl	Benzyl	70	87
4m	5-Br	Benzyl	70	85

[a]isolated yields



Scheme 3. Synthesis of 6'-nitro-4'-phenyl-1'H-spiro[indoline-3,2'-quinazolin]-2-one derivatives

For the synthesis of 6'-nitro-4'-phenyl-1'H-spiro[indoline-3,2'-quinazolin]-2-one (**6a**), a reaction was performed using 2-amino-5-nitro benzophenone (**5**), isatin (**2**) and ammonium acetate (**3**) in EtOH at room temperature, traces of the product was found even until 6 hours. Later, this reaction was carried out under reflux conditions and the desired transformation provided the product in very good yield. A series of 6'-nitro-4'-phenyl-1'H-spiro[indoline-3,2'-quinazolin]-2-one derivatives were synthesized under the above optimized conditions (Scheme 3). In this case, the presence of an electron withdrawing group at position 5 of the 2-aminobenzophenone, slowed down the reaction resulting in comparably longer reaction time and lower yields (Table 3, entries **6a-6g**).

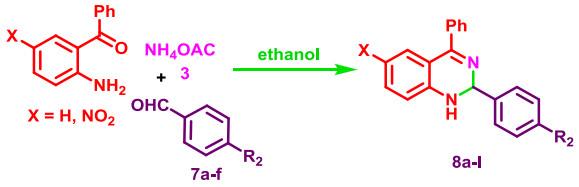
Table 3. Synthesis of 6'-nitro-4'-phenyl-1'H-spiro[indoline-3,2'-quinazolin]-2-one derivatives.

Entry	R	R_1	Time (min)	Yield[a] (%)
6a	H	H	150	88
6b	5-F	H	150	86
6c	5-Cl	H	160	85
6d	5-Me	H	170	85
6e	5-OMe	H	170	83
6f	5-NO_2	H	160	85
6g	5-Br	Benzyl	180	82

[a]isolated yields

Based on the interesting results obtained using the above discussed method for the synthesis of 4'-phenyl-1'H-spiro[indoline-3,2'-quinazolin]-2-one derivatives (**4a-m** and **6a-g**), it was considered of interest to perform the reaction using aromatic benzaldehydes as reported by Boulcina and co-

workers[19b] (Scheme 4). In this regard, benzaldehydes possesing both electron donating as well as electron withdrawing substituents have been employed. It was observed that the synthesis of 2,4-diphenyl-1,2-dihydroquinazoline derivatives (**8a-f**) using this method requires slightly longer times when compared to the synthesis of **4a-4m**. Whereas 6-nitro-2,4-diphenyl-1,2-dihydroquinazolines (**8g-l**) were obtained under reflux conditions and the time required is similar to that for the synthesis of **6a-6g**. Interestingly, the aromatic benzaldehydes with both electron donating as well as electron withdrawing substituents reacted well to provide excellent yields of the corresponding products and similar to the isatins, benzaldehydes afford the dihydro product exclusively.



Scheme 4. Synthesis of 2,4-diphenyl-1,2-dihydroquinazoline derivatives.

Table 4. 2,4-diphenyl-1,2-dihydroquinazoline derivatives.

Entry	X	R_2	Time (min)	Condition	Yield[a] (%)
8a	H	4-Cl	90	rt	89
8b	H	4-Br	90	rt	87
8c	H	4-NO_2	70	rt	92
8d	H	3-NO_2	90	rt	85
8e	H	4-OMe	80	rt	90
8f	H	3,4-DiOMe	90	rt	88
8g	NO_2	4-Cl	150	reflux	81
8h	NO_2	4-Br	150	reflux	80
8i	NO_2	4-NO_2	130	reflux	85
8j	NO_2	3-NO_2	160	reflux	78
8k	NO_2	4-OMe	140	reflux	82
8l	NO_2	3,4-DiOMe	150	reflux	79

[a]isolated yields

After employing isatins and benzaldehydes, we turned our attention towards the use of activated ketones like benzil and ethyl pyruvate. It was observed that the reaction did not proceed until 6 hours even under refluxing conditions.

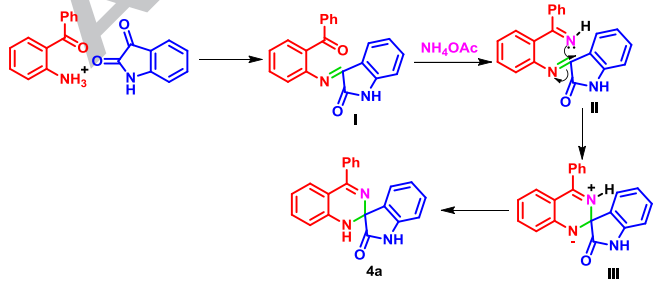


Figure 2. Plausble mechanism for the formation of 4'-phenyl-1'H-spiro[indoline-3,2'-quinazolin]-2-one

The plausible mechanism for the formation of 4'-phenyl-1'H-spiro[indoline-3,2'-quinazolin]-2-one from a three component reaction of isatin, 2-aminobenzophenone and ammonium acetate is outlined in the Figure 2. The reaction is presumed to proceed with the formation of a ketoimine (**I**) from 2-aminobenzophenone and isatin. Later ammonium acetate reacts with the keto group of 2-aminobenzophenone to form a diimine (**II**) with the expulsion of water and acetic acid. The imine carbon which is susceptible for a nucleophilic attack was then attacked by the adjacent nitrogen atom to form carbanion intermediate (**III**) which undergoes intramolecular cyclization to form **4a.**

The synthesized 4'-phenyl-1'H-spiro[indoline-3,2'-quinazolin]-2-one derivatives were evaluated for their antibacterial activity using well diffusion method against both Gram-positive bacterial strains such as *Staphylococcus aureus* MTCC 96, *Bacillus subtilis* MTCC 121, *Staphylococcus aureus* MLS16 MTCC 2940 and *Micrococcus luteus* MTCC 2470 as well as Gram-negative bacterial strains such as *Klebsiella planticola* MTCC 530, *Escherichia coli* MTCC 739 and *Pseudomonas aeruginosa* MTCC 2453. The minimum inhibitory concentration (MIC) values are summarized in Table 5 and were compared with ciprofloxacin. From the results it is evident that, some of the compounds were selectively active against one Gram-positive bacteria, *Micrococcus luteus* MTCC 2470 and one Gram-negative bacteria, *Klebsiella planticola* MTCC 530. Compounds **4j** and **6c** exhibited potent antimicrobial activity with MIC value 3.9 µg/ml and **4m** and **6g** showed good antimicrobial activity with MIC value 7.8 µg/mL against *Klebsiella planticola* MTCC 530. Compounds **4f**, **6a**, **6c** and **6g** showed appreciable antibacterial activity with MIC values 7.8 µg/ml against *Micrococcus luteus* MTCC 2470. Compounds **4b**, **4d** and **6a** showed the MIC value of 15.6 µg/mL against *Klebsiella planticola* MTCC 530. The compounds **4a**, **4b**, **4d**, **4e**, **4h** and **4j** exhibited moderate activity with MIC values 31.2 µg/ml against *Micrococcus luteus* MTCC 2470. In addition, all the compounds were evaluated for their antifungal potential against the fungal strain *Candida albicans* MTCC 3017 in comparison with miconazole as standard drug. In this case, only compound **6c** showed appreciable MIC value of 31.2 µg/mL against the tested fungal strain.

Table 5. Antimicrobial activity of the compounds against different microbial strains

Compound	Minimum Inhibitory Concentration (MIC, µg/ml)		
	M.l[a]	*K.p*[b]	*C.a*[c]
4a	31.2	62.5	-
4b	31.2	15.6	62.5
4d	31.2	15.6	-
4e	31.2	-	-
4f	7.8	-	-
4h	31.2	-	-
4j	31.2	3.9	-
4m	-	7.8	-
6a	7.8	15.6	-
6c	7.8	3.9	31.2
6d	-	-	62.5
6g	7.8	7.8	-
Miconazole	-	-	7.8
Ciprofloxacin	0.9	0.9	-

[a]Micrococcus luteus MTCC 2470, [b]Klebsiella planticola MTCC 530, [c]Candida albicans MTCC 3017.

Conclusion

In conclusion, a simple, mild, efficient and environmentally benign method for the synthesis of 4'-phenyl-1'H-spiro[indoline-3,2'-quinazolin]-2-ones and 2,4-diphenyl-1,2-dihydroquinazolines has been developed without using any catalyst in ethanol. The advantages of this method include its simplicity of operation, cleaner reactions, absence of side products and higher yields. Furthermore, the spiroquinazolines have been screened for their antimicrobial activities. Among these, compounds **4f**, **6a**, **6c** and **6g** showed selective activity against Gram-positive bacteria. Whereas compounds **4j**, **4m**, **6c** and **6g** showed potent antimicrobial activity against Gram-negative bacteria.

General procedure for the synthesis of 4'-phenyl-1'H-spiro[indoline-3,2'-quinazolin]-2-one derivatives (4a-m) and 2,4-diphenyl-1,2-dihydroquinazoline (8a-f)

A mixture of 2-amino benzophenone (1 mmol), corresponding isatin or aldehyde (1 mmol) and ammonium acetate (2 mmol) in ethanol (5 mL) was stirred at room temperature. The progress of reaction was monitored by TLC. After completion of the reaction ice-cold water was added and stirred for a while. The solid product obtained was filtered and washed with water. This was further purified by crystallisation from ethanol or by short column chromatography on silica gel using ethyl acetate–petroleum ether as the eluent.

General procedure for the synthesis of 6'-nitro-4'-phenyl-1'H-spiro[indoline-3,2'-quinazolin]-2-one derivatives (6a-g) and 6-nitro-2,4-diphenyl-1,2-dihydroquinazolines (8g-l)

A mixture of 2-amino-5-nitro benzophenone (1 mmol), corresponding isatin or aldehyde (1 mmol) and ammonium acetate (2 mmol) in ethanol (5 mL) was was refluxed. The progress of reaction was monitored by TLC. After completion of the reaction the reaction mixture was cooled and ice-cold water was added and stirred for a while. The solid product obtained was filtered and washed with water. This was further purified by crystallisation from ethanol.

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