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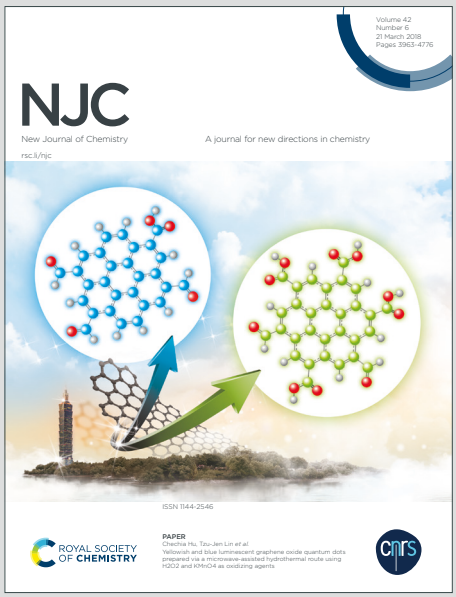

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ARTICLE

Regioselective Coupling of 2-Arylquinazolinone C-H with Aldehyde and Benzyl Alcohol under Oxidative Condition

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A direct and regioselective palladium catalyzed dehydrogenative coupling of aldehydes and benzyl alcohols with 2-aryl-quinazolinones C-H endowed with quinazolinone nucleus, which is one of the most fascinating and vital core units as an inherent directing group under oxidative condition was developed. This atom/step economic aroylation offers an attractive handle and very green approach for highly convergent and diversity-oriented synthetic modifications and construction of vital molecules of therapeutic interest.

The Synthetic manipulations that create new carbon-carbon bonds are among the most important transformations in organic chemistry, as they enable the design of artificial chemical structures essential to modern life. Cross coupling reactions catalyzed by transition metals have been developed and improved to a level that almost any structure can be prepared.[1-3] However, requirement of pre-functionalization of substrates, tedious purification stages and ultimately reduced overall material/atom and step economy in synthesis and chemical waste are associated with it. Current challenge is not only to develop new synthetic tools for performing a given transformation, but rather to perform it in a practical, environmental friendly, atom- and step-economical way. In past two decades, direct transformation of carbon-hydrogen (C–H) bond i.e. C–H activation have attracted much attention[4] and excellent achievements, and widespread applications have been made in the synthesis of pharmaceuticals, natural products, and agrochemicals. However, C–H bond activation still requires at least one functionalized partner to generate desired C–C bond. In this context, direct coupling of two C-H bonds under oxidative condition such as dehydrogenative coupling[5] pioneered by Murahashi[5p] and Li[5o] to form a new carbon-carbon bond is highly desirable as it address most of the above challenges.[6,7] Quinazolinone, one of the most fascinating and vital nucleus found in biologically active and medicinally important natural products and drug candidates, consequently received an intensive focus by synthetic and medicinal chemists (Figure 1).[8,9] Several marketed drugs and intermediates with range of biological and pharmacological activities containing quinazolinone as privileged core are known and making their synthetic modifications tempting. In addition, quinazolinone substrates are endowed with inherent

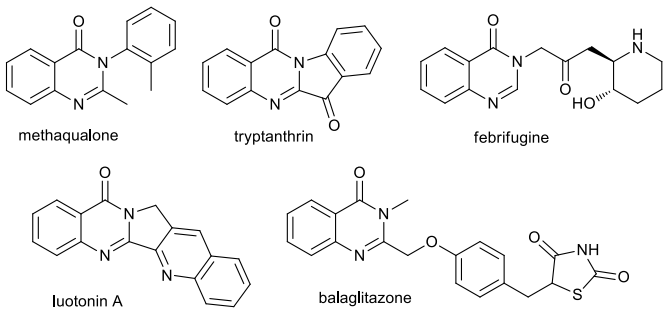
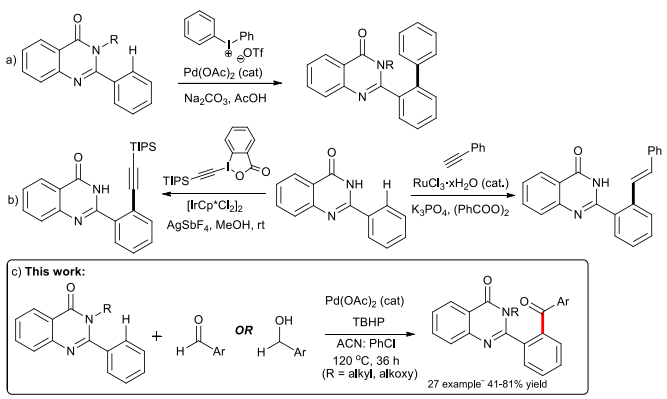


Figure 1. Selected bioactive quinazolinone drug and natural products.

directing group which can stimulate site selective C–H activation. Mhaske et. al disclosed quinazolinone directed C-H activation for monoarylation using diaryliodonium salts in presence of Pd-catalyst and Na_2CO_3 (Scheme 1, eq. a).[10] Back to back this, Hong and Park co-workers reported Pd-catalyzed and ligand assisted annulation of unmasked 2-arylquinazolin-4(3*H*)-ones with diaryliodonium salts via *C*-arylation followed by intramolecular *N*-arylation in presence of silver salt and base.[11] Later, Ru-catalyzed alkenylation of 2-aryl-quinazolin-



Scheme 1. Previous work and this work.

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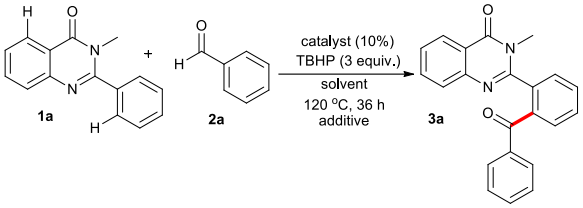
4(3*H*)-ones by coupling terminal alkynes as alkene partner (Scheme 1, eq. b)[12] and Ir-catalyzed alkynylation of 2-aryl-quinazolin-4(3*H*)-ones with TIPS-EBX as alkyne source (Scheme 1, eq. b)[13] have been reported. On the other hand, heck type coupling of quinazolinone, halogenation, amination, annulations and recently, oxidative annulations with aryl aldehyde and C-H sulfonylation as well as Rh-catalyzed annulation of quinazolinones with sulfoxonium ylidesare also reported.[14] Owing to the privileged biological and pharmacological importance of quinazolinone derivatives, and demand of direct coupling of two C-H bonds from atom/step-economy, convergent and diversity oriented synthesis point of view, herein, we have disclosed the straightforward and regio selective coupling of 2-aryl-quinazolinones C-H having quinazolinone as intrinsic directing group with aldehydes and benzyl alcohols under oxidative condition (Scheme 1, eq. c).

Our investigation commenced with the reaction of 2-phenyl-quinazolinone (**1a**) with benzaldehyde (**2a**) in the presence of $Pd(OAc)_2$ (5 mol%) and TBHP (3.0 equiv.) in DCE at 80 °C. After 36 h, we observed no recognizable change in the reaction mixture (table 1, entry 1). Hence, reaction was performed at 120 °C in sealed tube. After heating the reaction in sealed tube for 36 h, product **3a** was isolated in 25% yield using silica-gel column chromatography (table 1, entry 2). When reaction was performed using $PdCl_2$ as catalyst, results were not encouraging (table 1, entry 3). In order to optimize the reaction condition for better yield, various solvent, oxidizing agent were pursued. It was decided to perform the reaction in other solvent using $Pd(OAc)_2$ catalyst. Various solvents were screened (table 1, entries 4-8) but only ACN, and chlorobenzene found to give desired product in moderate yield (table 1, entry 6 & 8). Next, it was planned to screen combination of these solvents (table 1, entries 9-11) and found that acetonitrile:chlorobenzene (1:4) combination produced the product in good yield (entry 11). Replacement of oxidant with DTBP, $K_2S_2O_8$, $(NH_4)_2S_2O_8$ (entries 12-14) as well as performing the reaction in presence of some additives such as PPh_3, NaOAc, Na_2CO_3 did not turned on any expectations (entries 15-17). Decrease in the yield was observed by lowering the equivalents of oxidant (entry 18 & 19) and no substantial increase in yield was received after increasing oxidant equivalent (entry 20).

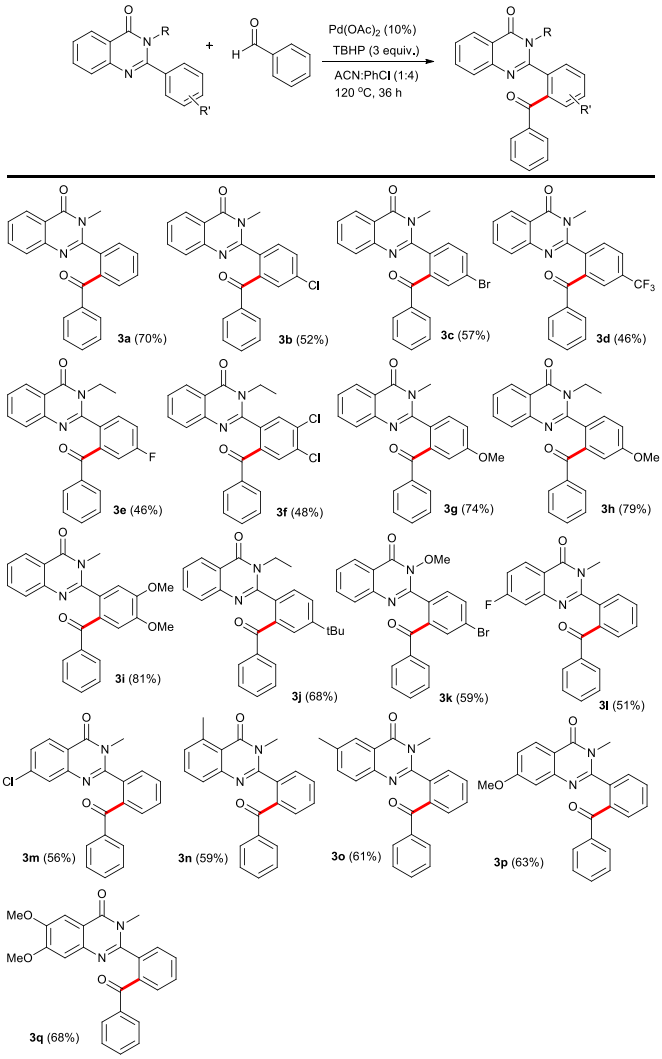
Having the optimized reaction condition, we investigated the reaction of several 2-aryl-quinazolinones (**1b-k**) with

Table 1. Optimization of the reaction conditions for Pd-catalyzed CDC reaction.[a]



entry	catalyst	solvent	Additive	yield[b]
1	$Pd(OAC)_2$	DCE[c]	-	trace
2	$Pd(OAC)_2$	DCE	-	25
3	$PdCl_2$	DCE		15
4	$Pd(OAC)_2$	THF	-	16
5	$Pd(OAC)_2$	dioxane	-	29
6	$Pd(OAC)_2$	ACN	-	43
7	$Pd(OAC)_2$	DMSO	-	nr
8	$Pd(OAC)_2$	PhCl	-	45
9	$Pd(OAC)_2$	ACN:PhCl (1:1)	-	60
10	$Pd(OAC)_2$	ACN:PhCl (4:1)	-	41
11	$Pd(OAC)_2$	ACN:PhCl (1:4)	-	70
12	$Pd(OAC)_2$	ACN:PhCl (1:4)	DTBP[d]	39
13	$Pd(OAC)_2$	ACN:PhCl (1:4)	$K_2S_2O_8$[d]	31
14	$Pd(OAC)_2$	ACN:PhCl (1:4)	$(NH_4)_2S_2O_8$[d]	36
15	$Pd(OAC)_2$	ACN:PhCl (1:4)	PPh_3[e]	31
16	$Pd(OAC)_2$	ACN:PhCl (1:4)	NaOAc[f]	38
17	$Pd(OAC)_2$	ACN:PhCl (1:4)	Na_2CO_3[f]	41
18[g]	$Pd(OAC)_2$	ACN:PhCl (1:4)	-	32
19[h]	$Pd(OAC)_2$	ACN:PhCl (1:4)	-	61
20[i]	$Pd(OAC)_2$	ACN:PhCl (1:4)	-	72

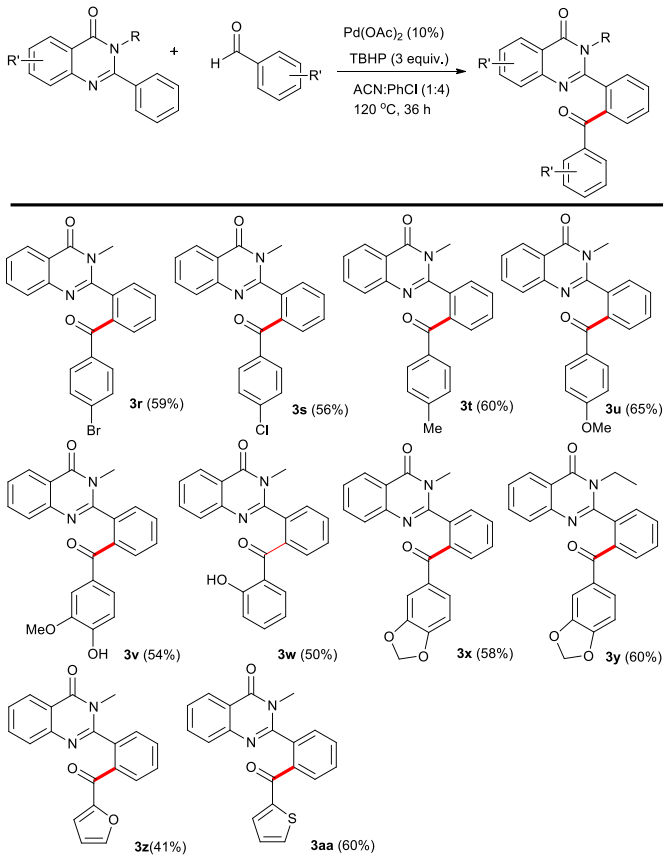
[a]Conditions: 1a (1.0 mmol, 1.0 equiv.), 2a (2.0 equiv.), catalyst (10 mol%), oxidant (3 equiv.), solvent (3.0 mL), 120 °C, 36 h. [b]isolated yields. [c]refluxed at 80 °C. [d]as oxidant (3.0 equiv.) instead of TBHP. [e]as additive (20 mol%). [f]as additive (3.0 equiv.). [g]1.0 equiv. of TBHP. [h]2.0 equiv. of TBHP. [i]4.0 equiv. of TBHP.



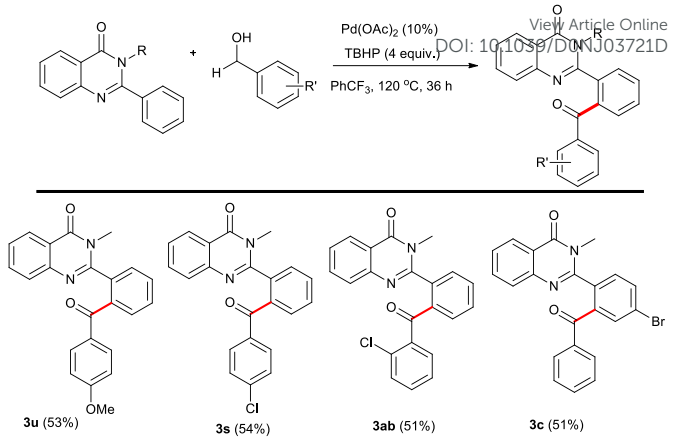
Scheme 2. Scope of 2-aryl-quinazolinones.

substitution on 2-aryl ring as well as with *N*-methyl, *N*-ethyl and *N*-methoxy quinazolinones in order to assess the scope of the optimized cross-dehydrogenative coupling reaction (Scheme 2). Consequently, 2-aryl-quinazolinones bearing electron attracting groups such as -Cl, -Br, -F, -CF_3 (**1b-f**) with *N*-methyl and *N*-ethyl group afforded the corresponding desired products in moderate yields (46-57%). *N*-Methoxy-2-Aryl-quinazolinone also produced desired product (**3k**) in 59% yield. Under similar conditions, 2-aryl-quinazolinones holding electron releasing groups such as alkyl and alkoxy groups (**1g-j**) reacted effectively with benzaldehyde to providing corresponding desired acylated products (**3g-j**) in good yields (68-81%). Interestingly, with unsymmetrical aryl ring, acylation occurs selectively at less hindered side only (**3f** and **3j**). Next, the effect of substitution on aryl ring of quinazolinone core structure was demonstrated (Scheme 3). Quinazolinone having -fluoro and -chloro group as well as methyl group produced the desired products (**3l, 3m, 3n** and **3o**) in good to moderate yield (51, 56, 59 and 61% respectively), whereas quinazolinones having methoxy group delivered the product (**3p** and **3q**) in 63 and 68% yield respectively.

Further, the scope of aryl aldehyde was investigated (Scheme 3). Treatment of aryl-aldehydes having electron attracting and releasing groups proceeded to afford desired product (**3r-y**) in good to moderate yields (50-65%). Arylaldehydes having hydroxy group (-OH) at 2 or 4 position gave expected product



Scheme 3. Scope of aryl-aldehydes and quinazolinones.



Scheme 4. Oxidative coupling of benzyl alcohols with quinazolinones.

(**3v** and **3w**) in moderate yield (50 and 54% respectively). Other than substituted benzaldehydes, 2-furylaldehyde and 2-thienylaldehyde also reacted to affording corresponding product (**3z** and **3aa**) in moderate yield (41 and 60% respectively).

Considering that the C-H coupling of benzyl alcohol under oxidative condition,[15] the behaviour of our substrate under similar condition with benzyl alcohol was planned (Scheme 4). When 2-phenyl-quinazolinone (**1a**) was allowed to react with benzyl alcohol (**4a**) under our optimized condition, desired acylated product 3a was observed in low yield (35%). Reaction was also performed in other solvents such as DCE, THF, DMSO, and toluene but no significant improvement in yield was observed. Next, we conducted reaction in trifluorotoluene as solvent and 4.0 equivalent of TBHP. To our delight reaction went smoothly to give desired product **3a** in 61% yield. Some more benzyl alcohols were treated under similar condition with 2-phenyl-quinazolinone (**1a**) to give corresponding desired product (**3u**, **3s**, **3ab**, **3c**) in good to moderate yields (Scheme 4). When our optimized condition was tested on NH-quinazolinone (2-phenylquinazolin-4(3*H*)-one), we observed complex reaction mixture (multiple spots).

In order to trace the mechanistic path of our developed reactions, some control experiments were planned (Scheme 5). We performed the radical trapping experiment during the reaction of 2-aryl-quinazolinone **1a** with benzaldehyde **2a** in the presence of varied equivalents of classical radical scavengers 3,5-di-*tert*-butyl-4-hydroxytoluene (BHT) and (2,2,6,6-tetramethylpiperidin-1-yl)oxyl (TEMPO). Observations of these experiments revealed the rapid decrease in yield of product **3a** in the presence of 2−4 equivalents of the radical scavengers individually to the reaction under standard condition. Expected radical trapped adduct product (**5**) generated by interaction of acyl radical with TEMPO was isolated and characterized using ^{1}H, ^{13}C and HRMS data. When reaction was performed with benzyl alcohol in presence of TEMPO (4.0 equiv.), TEMPO trapped adduct was formed and no desired product formation was observed. When reaction was performed in absence of TBHP, with 10 mol% as well as 1.0 equivalent of palladium catalyst, no product formation was

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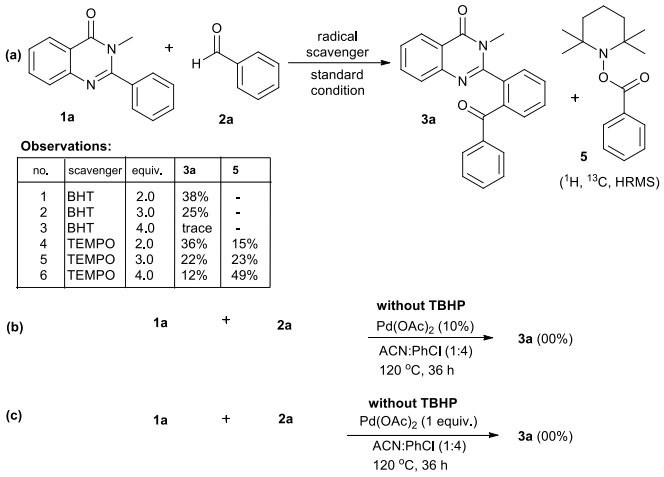
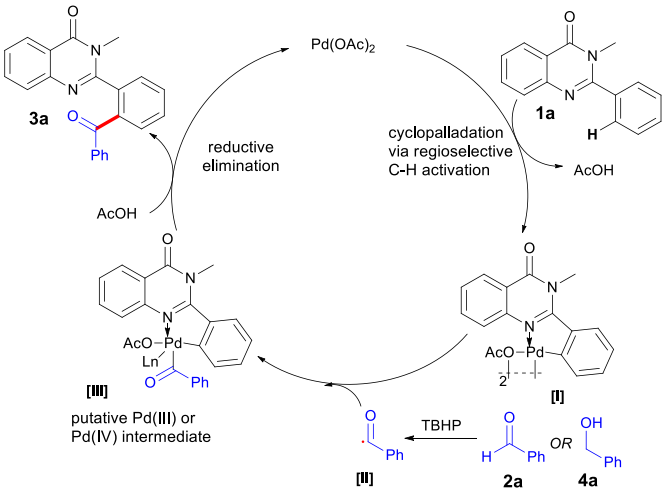


no.	scavenger	equiv.	3a	5
1	BHT	2.0	38%	-
2	BHT	3.0	25%	-
3	BHT	4.0	trace	-
4	TEMPO	2.0	36%	15%
5	TEMPO	3.0	22%	23%
6	TEMPO	4.0	12%	49%

Scheme 5. Studies for tracing mechanistic path.



Scheme 6. Plausible mechanistic path.

observed. These observations strongly support that a radical process is involved in the reaction.

Based on these observations and previous literature[16], plausible reaction mechanistic path for the present reaction has been proposed (Scheme 6). Initially, dimeric cyclopalladation(II) intermediate (**I**) forms via quinazolinone nitrogen chelation assisted regioselective C-H bond activation. Aroyl radical (**II**) generates by the interaction of aldehyde or benzyl alcohol with THBP radical. Interaction of this radical intermediate (**II**) with cyclopalladation(II) intermediate (**I**) which realize oxidation of palladium(II) to putative Pd(III)[16] or Pd(IV)[16] intermediate species (**III**) which upon reductive elimination releases desired product as well as Pd(II)-catalyst for next cycle.

Conclusions

In conclusion, palladium catalyzed regioselective dehydrogenative coupling of 2-arylquinazoline-4-one C–H with aldehyde and oxidative coupling with benzyl alcohol has been developed. Looking the assurance of functionalization of ketone group and the medicinal and pharmacological significance of the quinazolinone motif, atom and step efficacy, attractive substrate scope and good yields makes this direct aroylation protocol more enticing and prominent alternative for synthetic modifications and drug design.

Conflicts of interest

"There are no conflicts to declare".

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