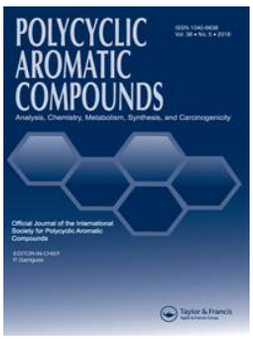

Polycyclic Aromatic Compounds

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A Simple and Efficient $[(n\text{-}Bu_3Sn)_2MO_4]_n$ Catalyzed Synthesis of Quinazolinones and Dihydroquinazolinones

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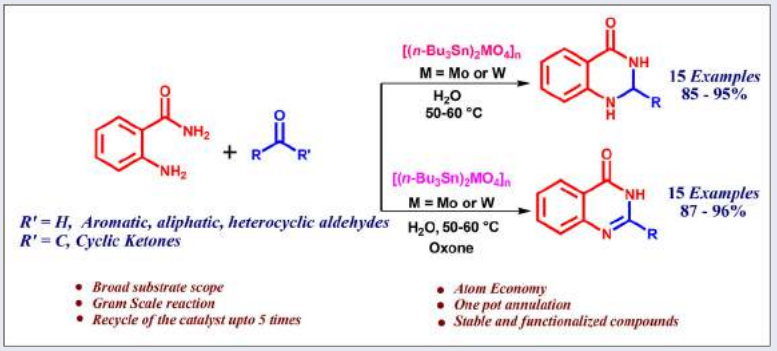
ABSTRACT

A novel unprecedented approach for the synthesis of various quinazolinones and dihydroquinazolinones has been using $[(n\text{-}Bu_3Sn)_2MO_4]_n$ as a catalyst. The reaction has been screened in various solvents and a gram scale experiment has also been demonstrated under given conditions. Further, the substrate scope of the reaction and the recyclability of the catalyst have also been studied.



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$[(n\text{-}Bu_3Sn)_2MO_4]_n$; dihydroquinazolinone; quinazolinone; recyclable catalyst

Introduction

Because heterocyclic chemistry is one of the emerging areas in organic chemistry, especially nitrogen-containing heterocycles have many applications in agro and pharmaceutical industry. Amongst them few compounds like quinazolinone and dihydroquinazolinone are present in many active pharmaceutical ingredients (APIs) as a core unit. However, these compounds were explored well in drug discovery unit to identify new chemical entities (NCEs) for early phase clinical studies (Phase I and Phase II). Moreover, from the literature it was found that the compounds containing dihydroquinazolinone units like quinethazone (**A**), fenquizone (**B**), benzouracil (**C**) and quinazolinone scaffolds like Febbrifugine (**D**), Luotonin A (**E**) and Rutaoarpine (**F**) are proved to be an excellent pharmacore units and also exhibit various therapeutic properties like anticancer,[1,2] antihypertension,[3] anticonvulsant,[4] antibacterial,[5,6] anti-inflammatory,[7,8] antianxietic activities,[9] and anti-diabatic (Figure 1).[10] Additionally, these core

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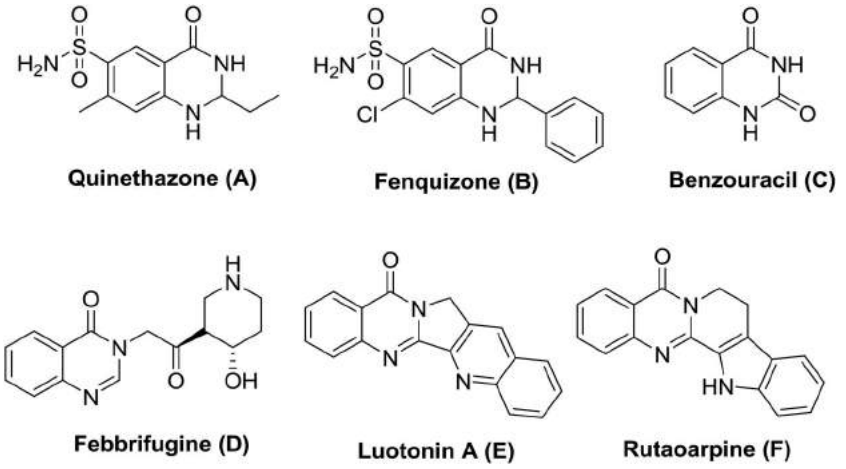
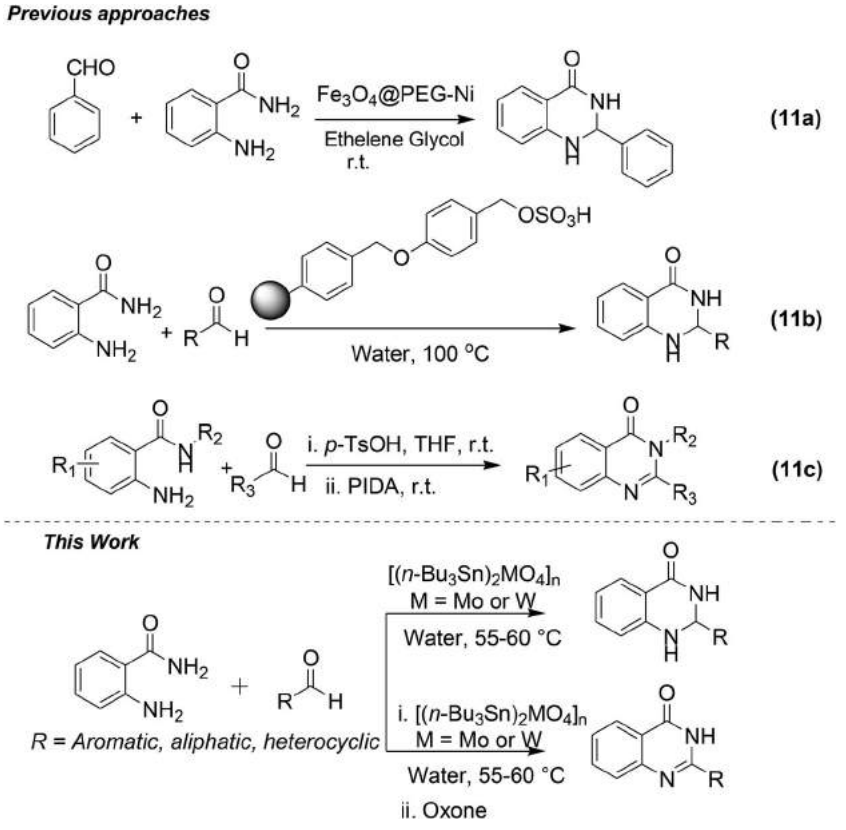


Figure 1. Some biologically important dihydroquinazolinone & quinazolinone compounds.



Scheme 1. Different synthetic approaches for quinazolinone derivatives

moieties also present in antidepressant, antihistamine, vasodilating agent molecules. Nonetheless, because of their biological activity, many methods have been reported for the synthesis of various dihydroquinazolinone derivatives[11–34] to provide good yields (Scheme 1). But each method has its own drawback like harsh reaction conditions, long reaction times, usage of expensive metals, multistep procedure, corrosive or non-benign catalyst, stoichiometric amount of catalyst, tedious

work-up and poor atom economy. Therefore, developing an environmentally benign method by using a novel catalyst in high yields is always challenging and noteworthy in synthetic organic chemistry.

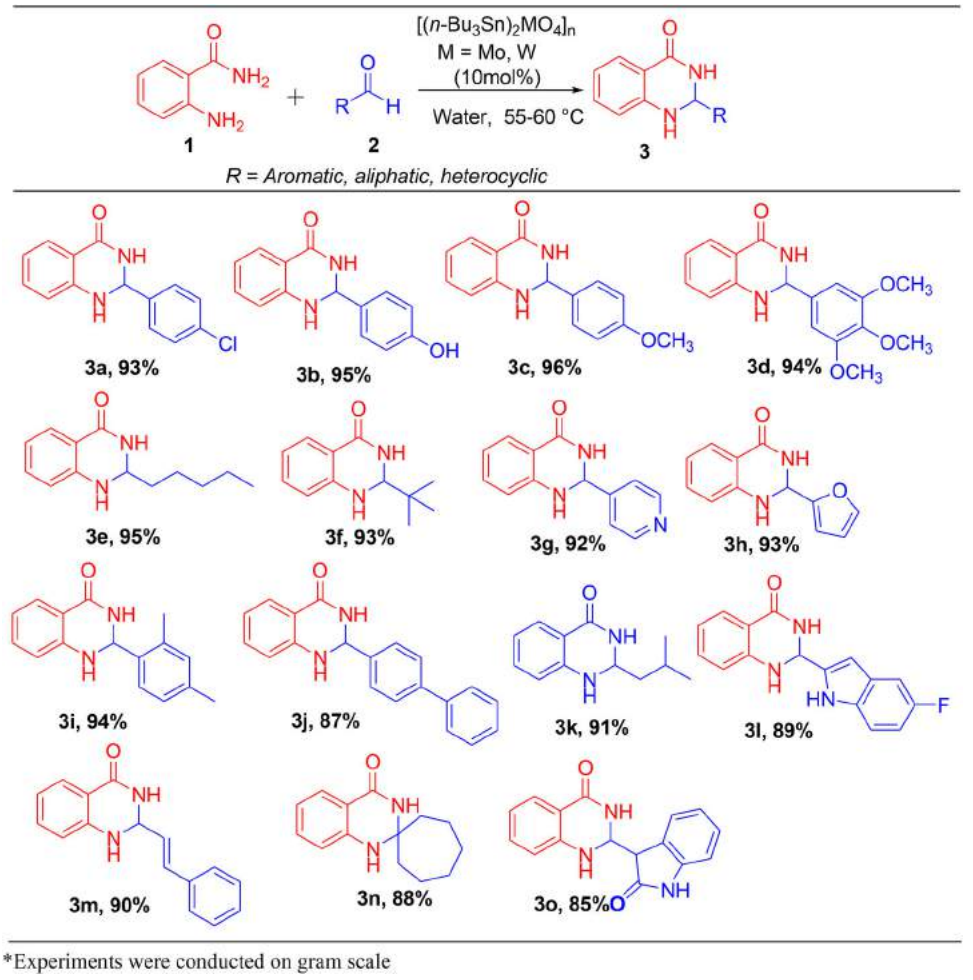
Results and discussion

In continuation of our earlier methods for the synthesis of various medicinally important heterocyclic compounds[35–41] here in we report the synthesis of different quinazolinones and dihydroquinazolinones from 2-aminobenzamide using $[(n\text{-}Bu_3Sn)_2MO_4]_n$ as a novel catalyst. However, from the literature it is found that tri-*n*-butyltin derivatives were prepared by Fischer and coworkers.[31,42–44] These compounds, having the general formula $[(R_3Sn)_2MO_4]$ [R = Me, Et, *n*-Pr, *n*-Bu and phenyl (Ph); M = Mo and W], which are formed through the coordination of the tetrahedral $[MO_4]^{2-}$ subunits and $[R_3Sn]^+$ as the linking spacers. To the best of our knowledge, very few synthetic transformations such as epoxidation of olefins, sulfoxidation, and *N*-oxidation of primary aromatic amines were reported using this catalyst.[31,43,45] However, here in, we would like to explore this catalyst for the synthesis of quinazolinone compounds. Accordingly, we have prepared two catalysts $(Bu_3Sn)_2MoO_4$ and $(Bu_3Sn)_2WO_4$ and their catalytic performance was also studied in the synthesis of quinazolinone by using 2-aminobenzamide and 4-chloro benzaldehyde as a model substrates. The reaction conditions were optimized by screening the solvents and in various reaction parameters such as temperature, time, and quantity of catalyst by using the model substrates. The reaction was screened in various solvents like DCM, THF, DMF, 1,4-dioxane, CH_3CN, toluene, ethanol and methanol. To our delight, the reaction was progressed well in polar protic solvents like ethanol, methanol-water than non-polar and polar aprotic solvents. In the view of green chemistry aspects, we have chosen water as the best solvent to get good yields (Table 1). Further, the influence of the temperature was also studied in different temperatures. The reaction was slow at room temperature and under reflux conditions; the reaction was completed to get a product in 93% yield. Moreover, the reaction was also checked in various stoichiometric amounts of the catalyst, and after fine-tuning, 10% of the catalyst provided good yields. Subsequently, the substrate scope of the reaction with different aldehydes was also studied. Gratifyingly, the reaction proceeded well with all types of aldehydes like aryl, *n*-hexyl, *tert*-butyl, cinnamyl, biphenyl, isobutyl, indole moiteis to get quantitative yields (Scheme 2). Moreover, the reaction was further checked with ketones like cycloheptanone, isatin and the reaction was progressed well. Nevertheless, to understand the reaction on a higher scale, we have also demonstrated the reaction on a 10 g scale and gratifyingly we could amenable to reproduce the similar

Table 1. Reaction optimization conditions for the synthesis of dihydroquinazolinones.

Entry	Solvent	Time (h)	Yield (%)	Catalyst (mole %)
1*	DCM	6	52	10
2*	THF	6	43	10
3*	DMF	6	40	10
4*	1,4-Dioxane	6	80	10
5*	CH_3CN	6	76	10
6*	Toluene	6	60	10
7*	Ethanol	1	92	10
8*	Methanol	1	91	10
9*	Water	0.5	93	10
10	Water, RT	20	85	10
11	Water, 55-60 °C	0.75	93	10
12	Water, 55-60 °C	1.5	90	5
13	Water, 55-60 °C	0.25	93	15
14	Water, 55-60 °C	0.25	93	20

*Reactions were performed under reflux conditions.

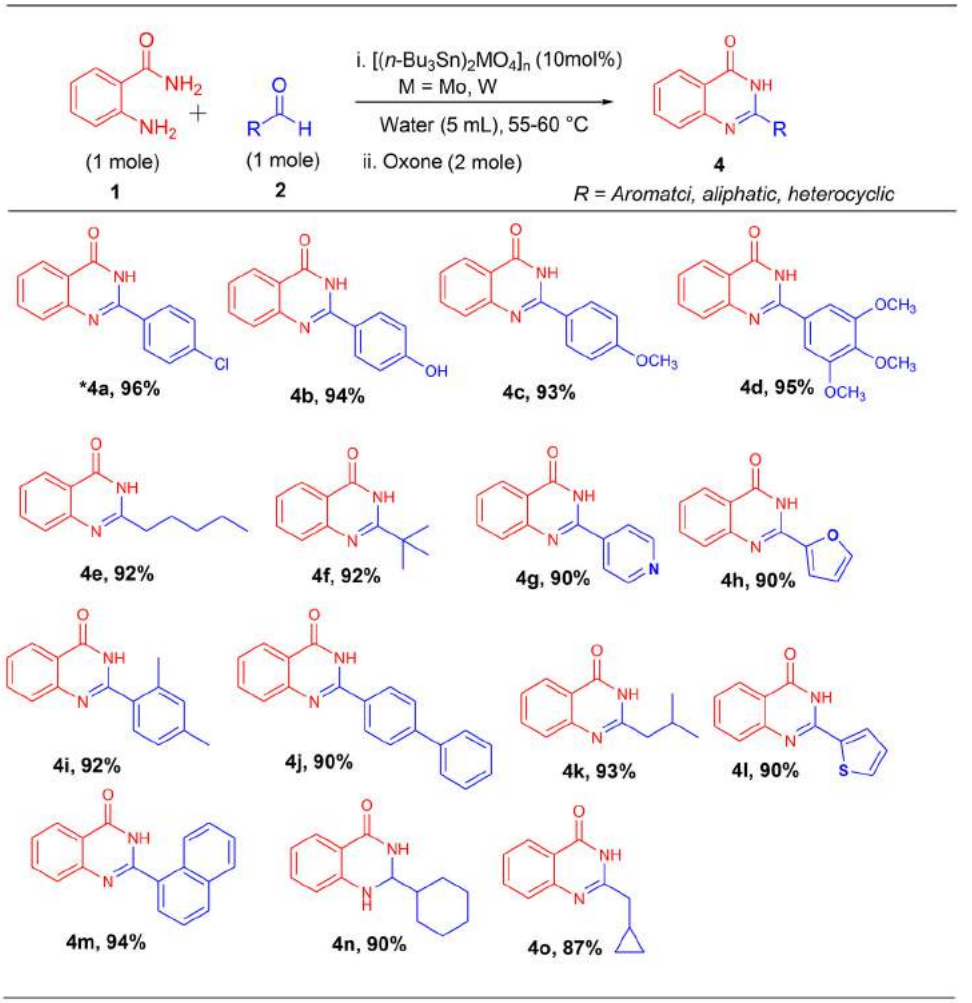


Scheme 2. Substrate scope of the reaction
*Experiments were conducted on gram scale

Table 2. Screening of oxidants.

Entry	Oxidant	Equivalents	Time (h)	Yield (%)
1	Oxone	1.0	2	80
2	Oxone	1.5	1.5	90
3	Oxone	2.0	1	91
4	PIDA	2.0	2	84
5	TBHP	2.0	1.5	88
6	H_2O_2	2.0	3	78
7	$K_2S_2O_8$	2.0	1.5	83

results (Scheme 2, entries 12, 13, 14, 15). Indeed, the auto-oxidation of dihydroquinazolinone was performed in presence of atmospheric oxygen but the reaction was not successful. Subsequently, we had drawn our attention to screen with various oxidizing agents like oxone, PIDA, TBHP, H_2O_2 and $K_2S_2O_8$. To our delight, we found that oxone was affording good yields (Table 2). Furthermore, the substrate scope of the reaction was also studied and pleasingly aryl, aliphatic, heterocyclic and cyclic aldehydes provided quantitative yields (Scheme 3). Finally, the recyclability of the catalyst was also studied in both the cases and could produce the similar catalytic activity up to five cycles (Figures 2 and 3).



*Conducted on 10 g scale

Scheme 3. Substrate scope of the reaction
*Conducted on 10 g scale

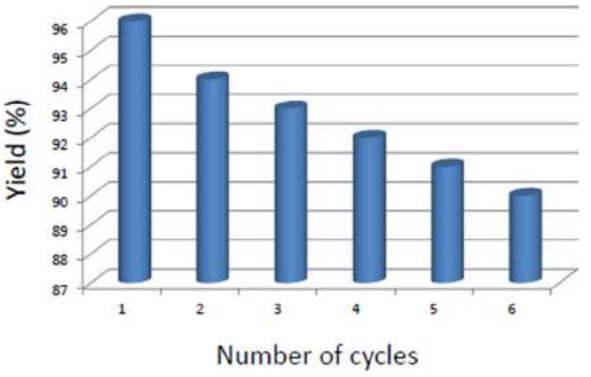


Figure 2. Recycle of catalyst $[(n\text{-}Bu_3Sn)_2MO_4]_n$.

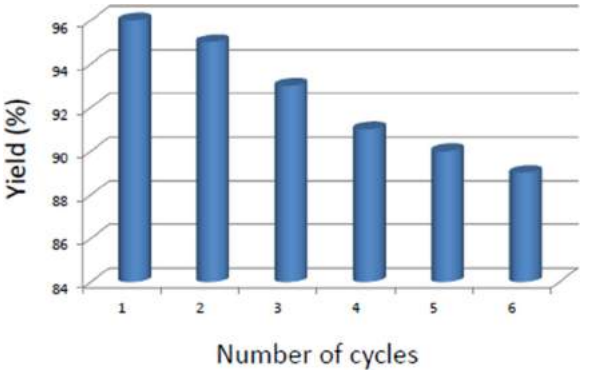
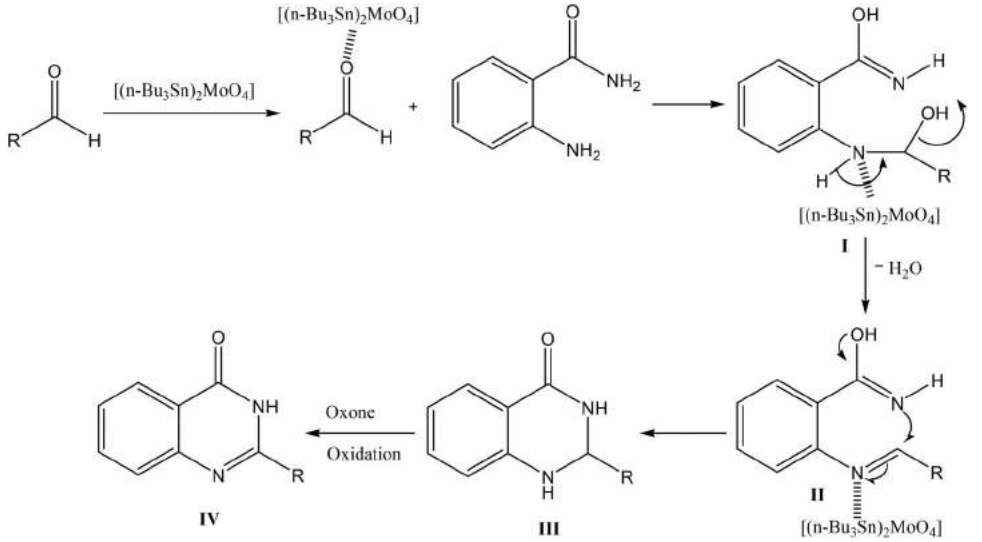


Figure 3. Recycle of catalyst $[(n\text{-}Bu_3Sn)_2MO_4]_n$.



Scheme 4. Plausible mechanism for the synthesis of quinazolinone

In the first step, the catalyst $[(n\text{-}Bu_3Sn)_2MO_4]_n$ act as Lewis acid and activates the aldehyde by coordinating with aldehyde. In the second step, the amine group of the anthranilamide reacts with activated aldehyde with the loss of water molecule to form a Schiff base II *via* intermediate I. In the third step, II undergoes intramolecular cyclization to form intermediate III and in the final step, the oxidation of III in presence of oxone afforded the desired quinazolinone (Scheme 4).[46,47]

Conclusions

In summary, we have successfully demonstrated the synthesis of various quinazolinones and dihydroquinazolinones by using $[(n\text{-}Bu_3Sn)_2MO_4]_n$ as a novel catalyst to get good yields. This is an atom economic, environmentally benign approach. Further, the substrate scope of the reaction and the recyclability of the catalyst was also well demonstrated. Finally, molybdenum and tungsten-based coordination polymers were found to be the most effective, and their efficiency was almost equal. The synthetic utility of this catalyst for other transformations are in progress in our laboratory.

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