Organic & Biomolecular Chemistry

PAPER

View Article Online
View Journal | View Issue

Cite this: *Org. Biomol. Chem.*, 2019, **17**, 6420

Dehydrogenative C(sp^3)–H bond functionalization of tetrahydroisoquinolines mediated by organic oxidants under mild conditions†

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Received 10th May 2019,
Accepted 17th June 2019

DOI: 10.1039/c9ob01090d

rsc.li/obc

The organocatalyzed Mannich reaction of unsubstituted and *N*-aryl-substituted tetrahydroisoquinolines (THIQs) and the Strecker reaction of several *N*-aryl-substituted THIQs through dehydrogenative C(sp3)–H bond functionalization (cross-dehydrogenative coupling) promoted by organic single electron oxidants DDQ and IBX are presented. The C–H oxidation/Mannich reaction of less reactive *N*-aryl substituted pyrrolidines is achieved *via* metal catalyzed photoredox catalysis. Operationally simple procedures provide desired products in an effective and time preserving manner.

Tetrahydroisoquinoline (THIQ) ring systems are present in numerous natural and synthetic organic compounds, many of which display useful and interesting biological activities (Fig. 1).[1] Covering a wide range of structural types, they are very attractive targets for synthesis and have stimulated the development of new synthetic approaches and methodologies. Recently, cross-dehydrogenative coupling (CDC) reactions have emerged as an effective tool for the functionalization of THIQs.

Since the seminal studies on cross-dehydrogenative coupling by Murahashi and Li through the activation of the α-C (sp^3)–H bond of tertiary amines,[2] tremendous progress has been made in the THIQ functionalizations mostly employing high-valent transition-metal catalysts combined with co-oxidants such as *tert*-butyl hydroperoxide (TBHP), H_2O_2, molecular oxygen, *etc.*[3,4] or utilizing visible light photoredox catalysis with transition metal complexes as catalysts.[5]

However, the use of organic oxidants possessing a high activity for C–H oxidation would be more desirable from the viewpoint of green and sustainable chemistry.[6] Moreover, metal impurities can be detrimental in pharmaceutically important intermediates and final products.[7] Hence, significant efforts have been made to carry out CDC reactions in the absence of metal catalysts. Visible light photoredox catalysis in the presence of organic photosenzitizers such as Eritrozine B,[8] eosin Y,[9] or carbon nitride[10] has proven to be a very effective tool for the functionalization of THIQs. Among organic oxidants, elemental iodine,[11] 2-iodoxybenzoic acid (IBX)[12] and hypervalent iodine(III)[13] reagents are notable examples in the oxidations and functionalizations of THIQs.

2,3-Dichloro-5,6-dicyano-1,4-benzoquinone (DDQ) as an organic oxidant with high oxidation potential has been extensively used in benzylic oxidation reactions and subsequent coupling reactions with various nucleophiles in the presence or in the absence of metal catalysts.[14] Stoichiometric DDQ had been used in tertiary amine C–H oxidations and the subsequent synthesis of vicinal diamines,[15] in the THIQ arylations,[16] Mukaiama/Manich type reactions of *N*-aryl pyrrolidines and silyl enol ethers[17] and in the intramolecular aza-Prins-type cyclizations.[18] Catalytic DDQ in the presence of catalytic amounts of AIBN was used in THIQ functionalizations by Prabhu's group, using oxygen as a stoichiometric oxidant.[19] A single example of the oxidative carbon–carbon bond-forming reaction of THIQ and acetone catalyzed by DDQ in the pres-

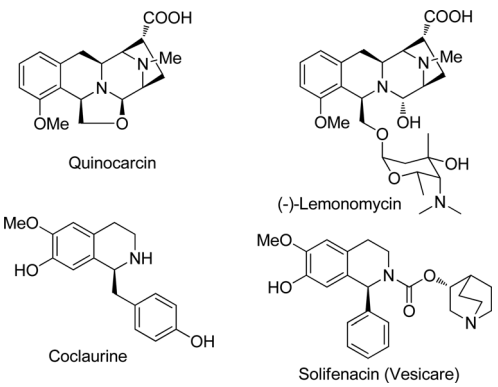

Fig. 1 Selected examples of bioactive molecules containing the THIQ motif.

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†Electronic supplementary information (ESI) available. See DOI: 10.1039/c9ob01090d

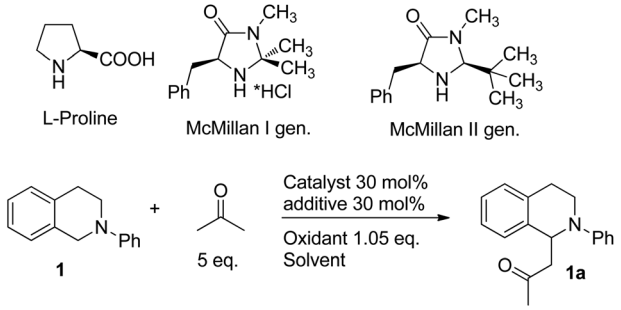
ence of an organocatalyst proceeding *via* an isolable iminium ion has been shown previously.[20]

Our aim was to develop a domino organocatalyzed Mannich reaction coupled with an organic oxidant promoted C–H oxidation of THIQs. As the single reported example[20] is limited to acetone as a nucleophile, systematic investigation about the reactivity, selectivity and substrate scope of these reactions is needed. Mannich products such as β-amino ketones and aldehydes are versatile synthetic intermediates for numerous pharmaceuticals and natural products and can be easily converted to 1,3-amino alcohols by reduction, or to Michael acceptors by elimination of the amine functionality.[21]

In order to make the reaction more practical and operationally simple, it is essential to activate ketones to their enolate form since ketones are less reactive pronucleophiles. Adding a catalytic amount of organocatalysts increases the reaction rate and adds to operational simplicity since organocatalysts are neither water nor oxygen sensitive. Herein we report a Mannich-type reaction of THIQs with ketones by employing organic oxidants and a catalytic amount of an amine catalyst. Short studies on the Strecker reaction of THIQs employing organic oxidants and metal complex promoted photoredox oxidation of *N*-aryl pyrrolidines are also shown.

Our investigation started with testing various organic oxidants in our envisaged reaction setup. Firstly, we tested DDQ as an oxidant of *N*-phenyl-tetrahydroisoquinoline **1** in the presence of 5 equivalents of acetone as a nucleophile with CH_3CN as a solvent. The reaction proceeds sluggishly to yield 10% of the desired target material after 48 h (Table 1, entry 1). Addition of 30 mol% of L-proline tremendously improved the reaction yield and 65% of the desired product was obtained after 48 h (Table 1, entry 2). It is known that acidic additives can increase the reaction rate of organocatalyzed reactions by increasing the rate of enamine formation and hydrolysis. Upon addition of 30 mol% of TFA as an additive, the reaction rate is dramatically increased, the reaction is completed in 2 hours, and the yield is improved to 78% (Table 1, entry 3). If the reaction was performed using acetone as a solvent, a slightly lower yield of the product is obtained (Table 1, entry 4). Several acidic additives such as AcOH and PTSA were tested but did not perform as good as TFA (Table 1, entries 5 and 6). Using Chloranil as an oxidant gave a slightly lower yield of 63% of the final product (Table 1, entry 7). Hypervalent iodine reagents did not give any product under optimized conditions (Table 1, entries 8–11). NHPI and *m*CPBA also did not promote oxidation steps in this reaction (Table 1, entries 12 and 13). In

Table 1 Optimization of the *N*-aryl substituted THIQ oxidation/organocatalyzed Mannich reaction[a]



Entry	Catalyst	Time (h)	Oxidant	Additive	Solvent	Yield[b] (%)
1	—	48	DDQ	—	CH_3CN	<10
2	L-Proline	48	DDQ	—	CH_3CN	65
3	L-Proline	2	DDQ	TFA	CH_3CN	78
4	L-Proline	2	DDQ	TFA	Acetone	72
5	L-Proline	4	DDQ	PTSA	CH_3CN	57
6	L-Proline	4	DDQ	AcOH	CH_3CN	61
7	L-Proline	4	Chloranil	TFA	CH_3CN	63
8	L-Proline	24	IBX	—	DMSO	<10
9	L-Proline	24	PIDA	—	CH_3CN	<10
11	L-Proline	24	PIFA	—	CH_3CN	—
12	L-Proline	24	NHPI	—	CH_3CN	—
13	L-Proline	24	*m*CPBA	—	CH_3CN	—
14	McMillan I gen.	4	DDQ	—	CH_3CN	65
15	McMillan II gen.	4	DDQ	TFA	CH_3CN	68
16	pyrrolidine	12	DDQ	TFA	CH_3CN	48
17	benzylamine	16	DDQ	TFA	CH_3CN	34

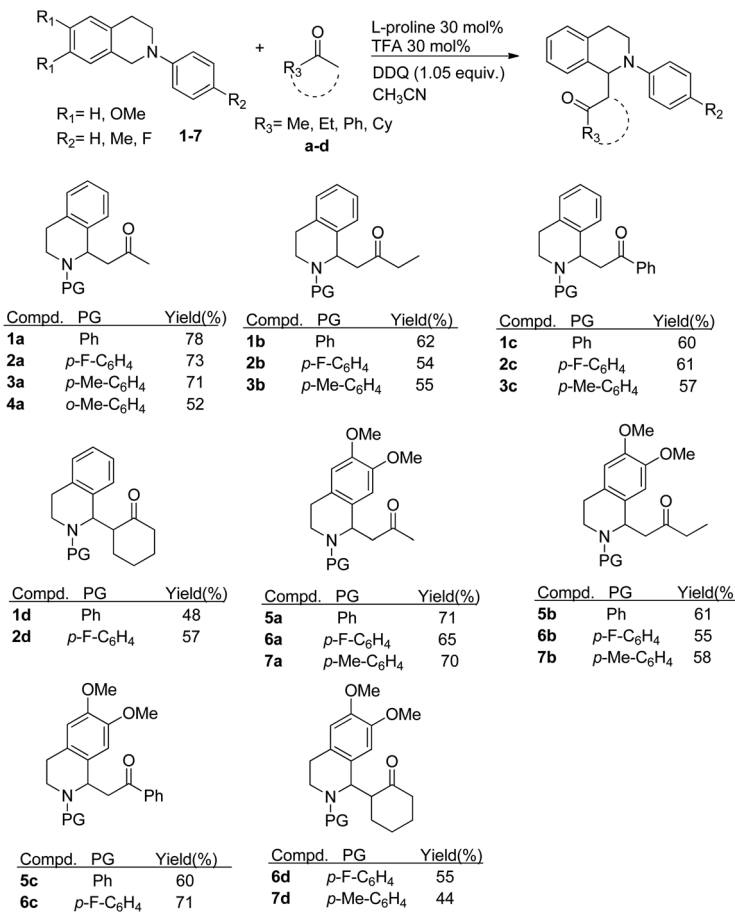
[a] Reaction conditions unless otherwise noted: Tetrahydroisoquinoline (0.25 mmol, 1 equiv.), oxidant (0.263 mmol, 1.05 equiv.), L-proline (0.075 mmol, 0.3 equiv.) and acetone (10 equiv.) were added to the solvent (2 mL) and stirred for a designated period of time. [b] Isolated yield after column chromatography. IBX = 2-Iodoxybenzoic acid; PIDA = phenyliododiacetate; PIFA = (bis(trifluoroacetoxy)iodo)benzene; NHPI = *N*-hydroxyphthalimide; *m*CPBA = *meta*-chloroperoxybenzoic acid.

the presence of McMillan type organocatalysts I and II with DDQ as an oxidant, the reaction proceeds to give 65% and 68% yield of the desired product, respectively (Table 1, entries 14 and 15). It is worth noting that 5–10% ee in the final product was observed with L-proline and McMillan type catalysts. Achiral secondary and primary amines such as pyrrolidine and benzylamine in the presence of TFA also give rise to the desired product, in 48% and 34% yield, respectively (Table 1, entries 16 and 17); in the absence of TFA, the reaction does not proceed.

After the optimal conditions were found, the scope of the reaction was investigated. *N*-Phenyl, *N*-*p*-tolyl, *N*-*p*-C_6H_4-F- or *N*-*o*-tolyl substituted THIQs were tested. *N*-Aryl groups stabilize reactive intermediates in cross-dehydrogenative couplings,[22] and these tertiary substituted substrates proved to be the best choice in this reaction setup.

As the nucleophilic partners several ketones were tested: acetone, ethyl methyl ketone, acetophenone and cyclohexanone (Scheme 1). In all cases, good yields were obtained; reactions with acetone proceeded most efficiently giving the best yields among the tested ketones. Ethyl methyl ketone having two enolizable positions reacts exclusively at the less substituted side of the molecule to give products in very good yields (Scheme 1, products **1–3b** and **5–7b**). Acetophenone reacts efficiently as well giving very good yields of products in all cases (Scheme 1, products **1–3c** and **5,6c**). Cyclohexanone reacts a bit slower compared to other tested ketones giving desired products **1,2d** and **6,7d** in approximately 1 : 1.3 diastereomeric ratio (see Scheme 1 and the ESI†). *o*-Tolyl substituted THIQs reacted only with acetone (product **4a**, Scheme 1) and did not react with any other nucleophile under optimized conditions, most probably due to the increased sterical hindrance close to the reaction center. All the aryl substituted tertiary THIQs reacted very efficiently; however, it is often required to remove the protecting group of the amine to have unsubstituted secondary amine for the synthetic or biomedical purposes.

Unprotected THIQ **8a** did not react under optimized conditions using DDQ as an oxidant. After oxidant screening tests, it was found that IBX is an effective oxidant of unprotected THIQs with DMSO as a solvent. It was possible to isolate intermediary imines **9a** and **9b** under these conditions in 69% and 78%, respectively. Isolated imines from both tetrahydroisoquinoline **8a** and 6,7-dimethoxy-tetrahydroisoquinoline **8b** reacted with ketones under Mannich reaction conditions (enamine formed from ketone and a catalytic amount of



Scheme 1 Organocatalyzed, DDQ promoted, THIQ oxidation/Mannich reaction.

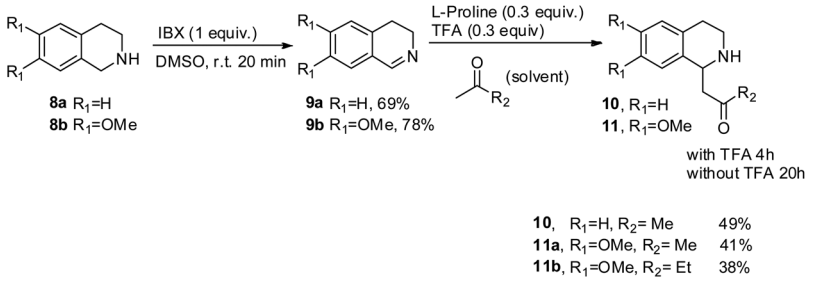
L-proline) to give moderate yields of desired products **10a** and **11a,b** (Scheme 2). A one pot reaction was attempted but did not proceed at all: upon addition of ketones, catalyst and additive to DMSO solution of imine formed *in situ* from THIQ **8a** and IBX, the Mannich reaction did not take place. Oxidation of **8a** with IBX also proceeds in MeOH at 60 °C, to give iminium ion **9a**. Upon addition of acetone, L-proline and additive to this reaction mixture, no reaction occurs. Optimization of this reaction and making it a one pot or tandem process is the subject of our continued interest, as it is known that tandem reactions could be far more effective and time saving omitting isolation of intermediates.

The plausible mechanism of the THIQ C–H oxidation/Mannich reaction is shown in Scheme 3. DDQ promoted oxidations are thought to begin with single electron oxidation of tertiaty amines.[23] In the next step upon hydride abstraction, an iminium ion is formed. Counterion exchange with TFA may take place giving iminium ion **I** which undergoes nucleophilic attack from enamine **II** formed from ketone and L-proline. Mannich adduct **III** undergoes hydrolysis with water to give the desired product, recovering a L-proline catalyst, which enters the next cycle (Scheme 3).

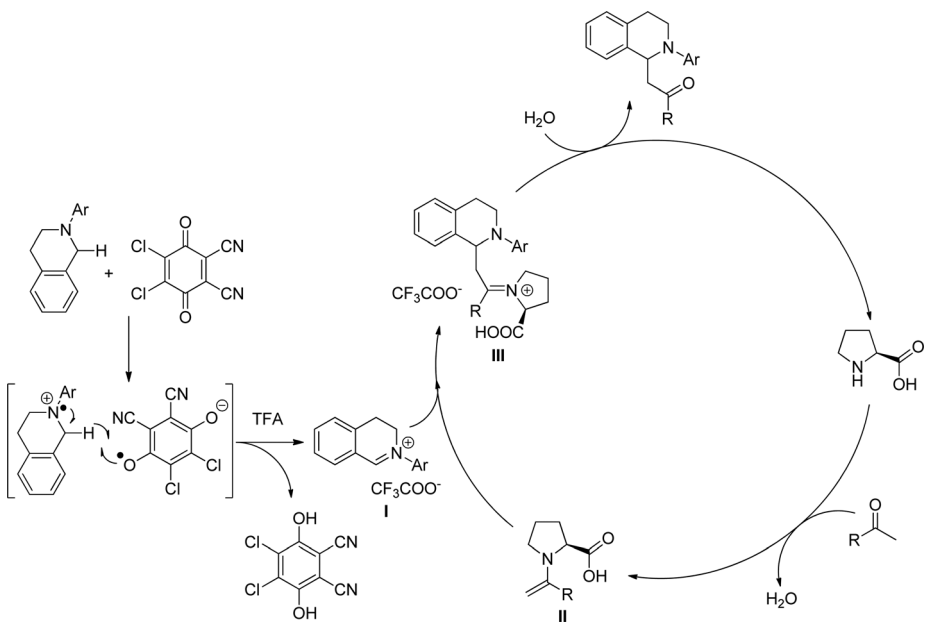
DDQ promoted C–H oxidation of THIQs/Strecker reaction

As the extension of the methodology of using organic oxidants for CDC functionalizations of THIQs we also tested DDQ as an oxidant in the C–H oxidation/Strecker reaction using TMSCN as the source of CN^- ions. α-Cyanations of THIQs using 2,2,6,6-tetramethylpiperidine *N*-oxide fluoroborate salt as an oxidant are known[24] while DDQ has been used as an oxidant in the α-cyanation of allyl ethers.[25]

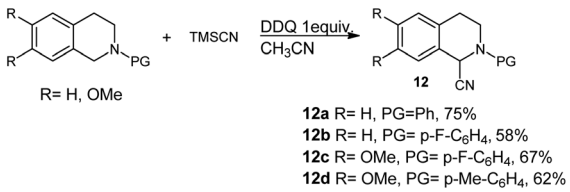
DDQ oxidation of *N*-aryl substituted THIQs proceeded very efficiently providing imine *in situ*; upon imine formation completion as confirmed by TLC, TMSCN is added to the reaction mixture to provide the Strecker product in good yields (Scheme 4). Reduction of Strecker adducts might provide



Scheme 2 C–H Oxidation/Mannich reaction of unprotected THIQs.



Scheme 3 Plausible mechanism of the DDQ promoted C–H oxidation/L-proline catalyzed Mannich reaction.

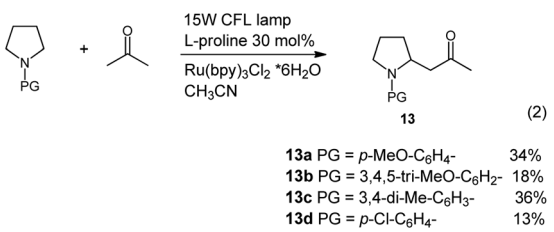


Scheme 4 DDQ promoted C–H oxidation/Strecker reaction of *N*-aryl substituted THIQs.

vicinal diamines, potentially pharmaceutically important compounds.

CDC coupling of pyrrolidines

Most of the CDC coupling methodologies (*vide supra*) are still limited to activated amines such as tetrahydroisoquinolines or benzylamines, and only a few examples have been reported using pyrrolidine or piperidine derivatives.[17,26] Thus, it is very challenging to develop an oxidative Mannich reaction of non-activated tertiary amines. *N*-Aryl substituted pyrrolidines or piperidines did not react under optimized conditions of the DDQ or IBX promoted C–H oxidation/Mannich reaction. No reaction was observed also when other organic oxidants: *m*CPBA, PIDA, PIFA or NHPI were tested. Under conditions of metal catalyzed photoredox catalysis, using $[Ru(bpy)_3]Cl_2$ in the presence of L-proline and irradiating with a 15 W CFL lamp, the reaction with acetone proceeded to give products in low yields after few days of reaction time (see the ESI†) (Scheme 5). Yields up to 36% were obtained, the main issue being the loss of activity of the catalyst after 12–24 h. The $[Ru(bpz)_3][PF6]_2$ catalyst was tested as well but did not provide better yields of the product **13a** compared to the $[Ru(bpy)_3]Cl_2$ catalyst. The 8 W CFL lamp did not provide better yields and **13a** was isolated in 15% yield after 4 days of reaction. The mechanism of this type of reaction is very well known and has been previously described.[5b] The reaction proceeds *via* single electron transfer (SET) from the *N*-aryl substituted tertiary amine to $Ru(bpy)_3^{2+*}$ species formed upon irradiation of the $Ru(bpy)_3^{2+}$ catalyst. An amine radical cation that is formed loses hydride to give an iminium ion that undergoes nucleophilic attack by the enamine species present in the reaction mixture and formed from ketone and the secondary amine



Scheme 5 Visible-light-promoted asymmetric cross-dehydrogenative coupling of tertiary amines to ketones.

catalyst. Ru^+ species are reoxidized by molecular oxygen *in situ* to reenter the catalytic cycle.

A number of pyrrolidine substrates possessing different aryl protecting groups have also been tested as well as N-Ph-piperidine but in all cases desired reactions were not observed (see ESI, Scheme S1†). We are currently working on the further improvements of this reaction and we will report results in due course.

Conclusion

We have shown that organic oxidants, DDQ and IBX, can be successfully used in the C(sp3)–H bond oxidation/organocatalyzed Mannich reaction of tertiary *N*-aryl-substituted and *N*-unsubstituted THIQs in an effective manner. The use of an organocatalyst and an acidic additive in the Mannich reaction of *in situ* formed iminium ions and ketones tremendously improved the reaction times and yields of this domino process. Besides the Mannich reaction, Strecker type addition of TMSCN to iminium ions (formed *in situ*) can also be performed with tertiary aryl substituted THIQs using DDQ as an oxidant. Less reactive *N*-aryl pyrrolidines did not undergo oxidation reactions with organic oxidants. However, under conditions of metal complex catalyzed photoredox reactions, C–H oxidation and the subsequent organocatalyzed Mannich reaction take place. These procedures represent a powerful method to form new carbon–carbon bonds directly from two different C–H bonds under oxidative conditions.

Conflicts of interest

There are no conflicts to declare.

Acknowledgements

This work was supported by the Ministry of Education, Science and Technology of the Republic of Serbia (Grant No. 172020). We gratefully acknowledge COST action CA15106: C–H Activation in Organic Synthesis (CHAOS) for support.

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