Organic & Biomolecular Chemistry

ROYAL SOCIETY OF CHEMISTRY

PAPER

View Article Online
View Journal | View Issue

Cite this: *Org. Biomol. Chem.*, 2021, **19**, 3479

Manganese-catalyzed dehydrogenative Csp^3–Csp^2 coupling of imidazo[1,2-*a*]pyridines with methyl ketones†

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Received 29th January 2021,
Accepted 19th March 2021

DOI: 10.1039/d1ob00169h

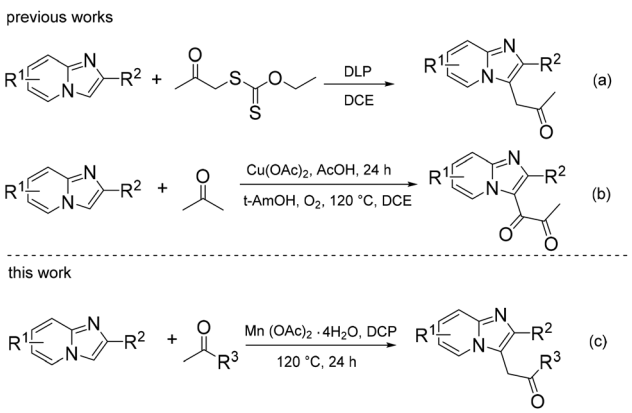
rsc.li/obc

A Mn(II)-catalyzed efficient C–H alkylation of imidazoheterocycles with methyl ketones has been developed *via* dehydrogenative C(sp^3)–C(sp^2) coupling which can serve as a novel approach toward hydrocarboxylated imidazolopyridines. The merit of this strategy is illustrated by excellent functional group tolerance and the use of cheap and readily available starting materials.

Introduction

Imidazo[1,2-*a*]pyridine and its derivatives are important nitrogen-containing heterocycles and have attracted great attention for their wide range of biological activities such as anticancer, antifungal, antiulcer, anti-inflammatory, antiprotozoal, antibacterial, analgesic, and anxioselective activities.[1] Moreover, some commercially available drug formulas contain the imidazo[1,2-*a*]pyridine motif such as zolpidem, alpidem, zolimidine, necopidem, and saripidem.[2] Due to the important values of imidazo[1,2-*a*]pyridine derivatives, a variety of synthetic strategies including condensation,[3] oxidative coupling[4] and decarboxylative coupling[5] have been developed for the synthesis and functionalization of imidazoheterocycles over the last decade.

Recently, C3–H alkylation *via* a radical process has become a viable method for the preparation of C3-alkylated imidazopyridines.[6] In 2016, Patel and co-workers reported the methylenation of imidazo[1,2-*a*]pyridines using H_2O_2 as a mild oxidant and DMSO (dimethyl sulfoxide) as the carbon synthon leading to the formation of 3,3′-bis(imidazopyridinyl)-methanes (Scheme 1a).[7] Li *et al.* and Miranda *et al.* independently developed a xanthate-based C3-alkylation of imidazopyridines using DLP (dilauroyl peroxide) as the initiator and oxidant in DCE.[8] Lu *et al.* described a $FeCl_3$-catalyzed tosylmethylation of imidazo[1,2-*a*]pyridines using TosMIC (*p*-toluenesulfonylmethyl isocyanides) in a mixture of H_2O and PEG400 (7 : 3).[9] These strategies are effective for assembling C3-alkylated imidazopyridines, although they sometimes suffer from poor reaction atom-economy and the requisition of pre-established functional groups or harsh conditions. Consequently, in view of green and sustainable chemistry, dehydrogenative Csp^3–Csp^2 coupling,[10] *via* the activation of the intrinsically less reactive C(sp^3)–H bond in aliphatic molecules, should be appealing enough for hydrocarboxylated imidazolopyridine constructions.[11] There have been a few methods reported to achieve direct coupling between imidazo[1,2-*a*]pyridines and the C(sp^3)–H bond among common aliphatic substances, such as acetonitriles,[12] alcohols and ethers.[13] However, the use of aliphatic ketones for alkylation remains a challenge. In 2015, Cao and co-workers reported a regioselective dicarbonylation of imidazo[1,2-*a*]pyridines by reacting with methyl ketones in toluene under an oxygen atmosphere (Scheme 1b);[14] in this approach, α-methyl was further oxidized to a carbonyl group to form 1,2-dicarbonylated products. In continuation of our research project on the synthesis and functionalization of imidazoheterocycles, herein, we report a novel and effective method to synthesize C3-alkylated imidazo[1,2-*a*]pyridines *via* direct cross-dehydrogenative coup-



Scheme 1 Synthesis of hydrocarbylated imidazo[1,2-*a*]pyridines.

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†Electronic supplementary information (ESI) available. See DOI: 10.1039/d1ob00169h

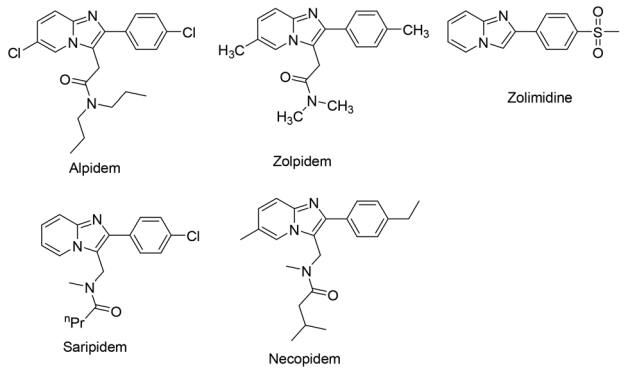


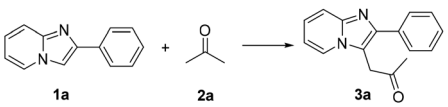
Fig. 1 Imidazo[1,2-*a*]pyridine-containing drugs.

ling of imidazoheterocycles with methyl ketones. This alkylation reaction is catalyzed by $Mn(OAc)_2{\cdot}4H_2O$, using DCP as the oxidant and without additional solvent (Fig. 1).

Results and discussion

Initially, 2-phenylimidazo[1,2-*a*]pyridine (**1a**) and acetone (**2a**) were chosen as the model substrates to optimize the reaction conditions (Table 1). The reaction of **1a** and **2a** in the presence of DTBP (di-*tert*-butyl peroxide) (3.0 equiv.) was performed under air at 120 °C for 24h, and the desired product 1-(2-phenylimidazo[1,2-*a*]pyridine-3-yl)propan-2-one (**3a**) was obtained in 42% isolated yield (Table 1, entry 1). In light of the above results, various peroxides including CHP (cumene hydroperoxide), DCP (dicumyl peroxide), TBHP (*tert*-butyl hydroperoxide) (70% in water), TBPB (*tert*-butyl perbenzoate), and DLP (dilauroyl peroxide) were used under identical reaction conditions (Table 1, entries 2–6); DCP showed a higher efficiency for this reaction than other peroxides. The amount of oxidant was screened, and the results showed that 3.0 equiv. of DCP provided **3a** in better yield (Table 1, entries 7 and 8). To further improve the reaction efficiency, several metal catalysts such as $CuCl_2$, CuCl, $FeCl_3$, $Mn(OAc)_2{\cdot}4H_2O$, $Ni(OAc)_2{\cdot}4H_2O$, $Mn(OAc)_2$ and $MnCl_2$ were tested in this reaction (Table 1, entries 9–16), and $Mn(OAc)_2{\cdot}4H_2O$ seemed to be the most appropriate for this transformation. The effect of the solvent on the reaction was also taken into account. Different solvents such as DMSO, DMF, EtOH, and chlorobenzene were investigated, and all gave inferior results (Table 1, entries 17–20). On increasing the reaction temperature to 130 °C, there was no significant change in the yield of **3a**. A lower yield of 62% was obtained when the temperature was decreased to 100 °C (Table 1, entries 21 and

Table 1 Optimization of the reaction conditions[a,b]



Entry	Oxidant (equiv.)	Catalyst	Solvent	Temp. (°C)	Yield[b] (%)
1	DTBP (3.0)	—	—	120	42
2	H_2O_2 (30%) (3.0)	—	—	120	Trace
3	DCP (3.0)	—	—	120	55
4	CHP (3.0)	—	—	120	37
5	TBHP (70%) (3.0)	—	—	120	26
6	DLP (3.0)	—	—	120	48
7	DCP (2.0)	—	—	120	47
8	DCP (4.0)	—	—	120	54
9	DCP (3.0)	$CuCl_2$	—	120	35
10	DCP (3.0)	CuCl	—	120	34
11	DCP (3.0)	$FeCl_3$	—	120	Trace
12	DCP (3.0)	$MnCl_2$	—	120	67
13	DCP (3.0)	$Mn(OAc)_2$	—	120	74
14	DCP (3.0)	$Mn(OAc)_2{\cdot}4H_2O$	—	120	78
15	DCP (3.0)	$Mn(OAc)_3{\cdot}2H_2O$	—	120	73
16	DCP (3.0)	$Ni(OAc)_2{\cdot}4H_2O$	—	120	70
17[c]	DCP (3.0)	$Mn(OAc)_2{\cdot}4H_2O$	DMSO	120	18
18[c]	DCP (3.0)	$Mn(OAc)_2{\cdot}4H_2O$	DMF	120	Trace
19[c]	DCP (3.0)	$Mn(OAc)_2{\cdot}4H_2O$	EtOH	120	35
20[c]	DCP (3.0)	$Mn(OAc)_2{\cdot}4H_2O$	C_6H_5Cl	120	29
21	DCP (3.0)	$Mn(OAc)_2{\cdot}4H_2O$	—	130	76
22	DCP (3.0)	$Mn(OAc)_2{\cdot}4H_2O$	—	100	62
23[d]	DCP (3.0)	$Mn(OAc)_2{\cdot}4H_2O$	—	120	69
24[e]	DCP (3.0)	$Mn(OAc)_2{\cdot}4H_2O$	—	120	76
25[f]	DCP (3.0)	$Mn(OAc)_2{\cdot}4H_2O$	—	120	70
26[g]	DCP (3.0)	$Mn(OAc)_2{\cdot}4H_2O$	—	120	72
27[h]	DCP (3.0)	$Mn(OAc)_2{\cdot}4H_2O$	—	120	78

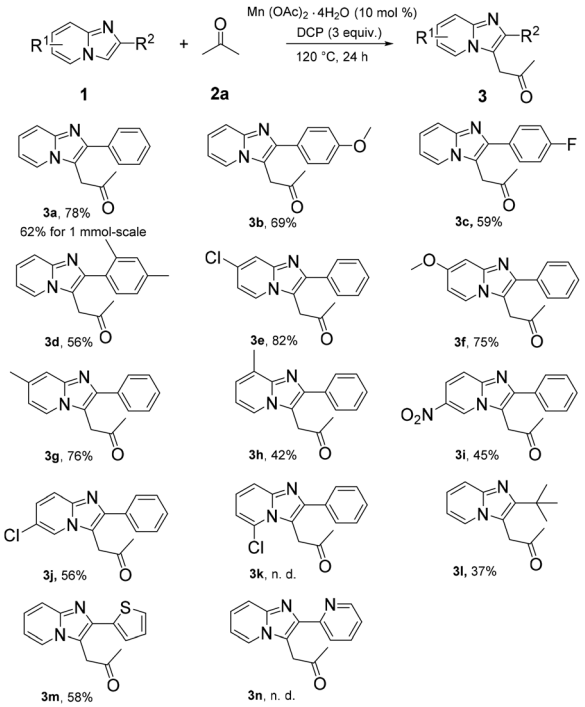
[a] Reaction conditions: **1a** (0.2 mmol), **2a** (2 mL), oxidant (3 equiv.), catalyst (0.02 mmol) and solvent (2.0 mL), 120 °C, 24 h, under air. [b] Isolated yield. [c] **1a** (0.2 mmol), **2a** (0.6 mmol), and solvent (2.0 mL). [d] Catalyst (0.01 mmol). [e] Catalyst (0.04 mmol). [f] 18 h. [g] 30 h. [h] Under N_2.

22). The most appropriate amount of $Mn(OAc)_2{\cdot}4H_2O$ was 10 mol% (Table 1, entries 14, 23 and 24). When the reaction was carried out for 18 h or 30 h, the yield of **3a** decreased (Table 1, entries 25 and 26). In addition, the result had no obvious difference when the reaction was performed in N_2 (Table 1, entry 27). Finally, the optimized reaction conditions were achieved using 10 mol% of $Mn(OAc)_2{\cdot}4H_2O$ and 3.0 equiv. of DCP at 120 °C for 24 h under air.

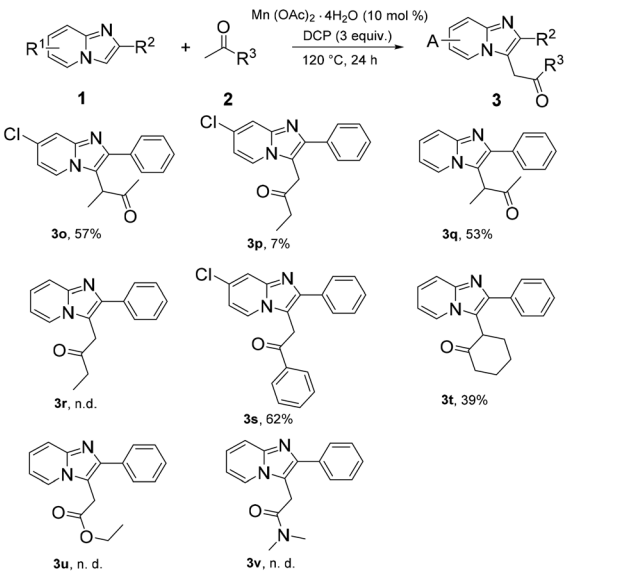
After optimizing the reaction conditions, we turned our attention toward the substrate scope of imidazo[1,2-*a*]pyridines; the results are summarized in Scheme 2. Imidazo[1,2-*a*]pyridine having an electron donating group (p-OMe) on the C-2 phenyl ring reacted smoothly with acetone, and afforded the product **3b** in 69% yield. The substrate with an electron withdrawing group (p-F) on the C-2 phenyl ring of imidazo[1,2-*a*]pyridine also delivered the corresponding product **3c** in 59% yield. 2-(2,4-Dimethylphenyl)imidazo[1,2-*a*]pyridine was also tolerated in this reaction, affording **3d** in 56% yield. Subsequently, the effect of the substituents on the pyridine ring was explored. The performance of the C-7 substituents on imidazo[1,2-*a*]pyridines was better than those at other positions in this reaction. The substrate bearing an electron-withdrawing substituent such as –Cl at the C-7 position of imidazo[1,2-*a*]pyridine reacted with **2a** to produce **3e** in 82% yield, and the substrates with electron-donating substituents such as –OMe and –Me at the C-7 position of imidazo[1,2-*a*]pyridines proceeded well in the reaction, giving the corresponding products **3f** and **3g** in yields of 75% and 76%, respectively. The substrates having other substituents (8-Me, 6-Cl, and 6-NO_2) on the pyridine ring of imidazo[1,2-*a*]pyridines furnished the desired products (**3h–3j**) in 42–56% yields. Moreover, when the phenyl ring was replaced with *tert*-butyl, the reactions proceeded to give the product **3l** in 37% yield. Imidazo[1,2-*a*]pyridine with C-2 thienyl performed well in the reaction, and gave **3m** in 58% yield. However, the desired product **3n** was not observed when the thiophene ring was replaced with electron-deficient pyridine.

Subsequently, the scope of ketones was explored, and the results are summarized in Scheme 3. 7-Chloro-2-phenylimidazo[1,2-*a*]pyridine was used to react with butanone, and two different products were separated by column chromatography. The C-3 butanone cross-coupling product **3o** was obtained in 57% yield, and the C-1 product **3p** was separated only in 7% yield. Butanone reacted with **1a** and gave the C-3 product **3q** in 53% yield, but the C-1 product **3r** was not observed in the reaction mixture. Acetophenone performed well in this optimized reaction and gave the desired product **3s** in 62% yield. Cyclohexanone was tolerated in this reaction and provided the product **3t** in 39% yield. However, the desired products **3u** and **3v** were not detected when the reactions were carried out using ethyl acetate and *N*,*N*-dimethylacetamide as partners with **1a**.

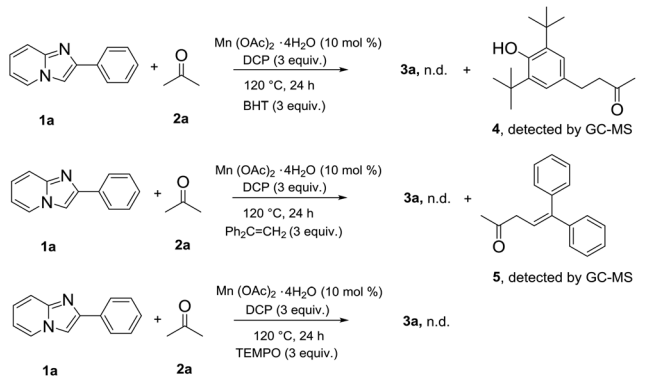
In order to gain further insight into the mechanistic pathway of the reaction, control experiments were carried out (Scheme 4). When 3 equiv. of butylated hydroxytoluene (BHT), ethene-1,1-diyldibenzene and 2,2,6,6-tetramethylpiperidine-1-oxyl (TEMPO) were added to the reaction mixture under the standard conditions, the desired product **3a** was not detected in these reaction mixtures. Notably, the radical scavenger suppressed the reaction, and the acetone radical was captured by BHT and ethene-1,1-diyldibenzene offered products **4** and **5**



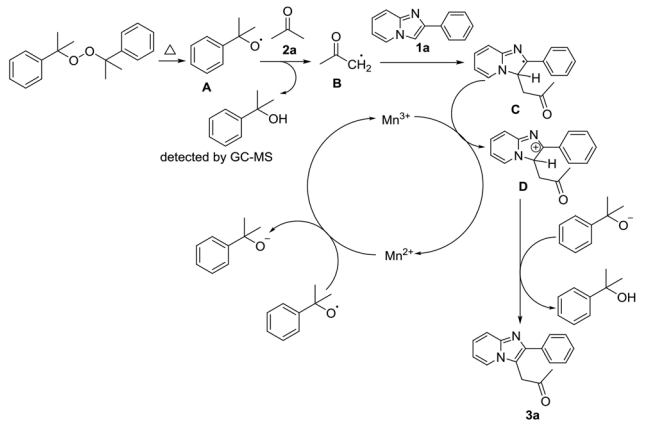
Scheme 2 Substrate scope of imidazo[1,2-*a*]pyridines. Conditions: **1** (0.20 mmol), **2a** (2.0 mL), catalyst (0.02 mmol), and DCP (0.60 mmol), 120 °C, 24 h, under air. Isolated yield.



Scheme 3 Substrate scope of imidazo[1,2-*a*]pyridines and methyl ketones. Conditions: **1** (0.20 mmol), **2** (2.0 mL), catalyst (0.02 mmol), and DCP (0.60 mmol), 120 °C, 24 h, under air. Isolated yield.



Scheme 4 Control experiments for the investigation of the mechanism.



Scheme 5 Proposed reaction mechanism.

(detected by GC-MS). These results illustrated that a radical process is possibly involved in the reaction.

On the basis of the above experimental results and previous studies,[15] a plausible mechanism is depicted in Scheme 5. Initially, homolysis of DCP produced the radical intermediate **A**, and the acetone radical **B** was generated by the abstraction of hydrogen from acetone by **A**. **B** coupled with **1a** to produce the C3-functionalized intermediate **C**. On the other hand, Mn^{2+} was oxidized to Mn^{3+} by **A**, and the imidazopyridine radical **C** was oxidized by Mn^{3+} to generate the carbocation **D** and regenerated Mn^{2+}. Finally, the C3–H elimination of the intermediate **D** led to the product **3a**.

Conclusions

In conclusion, a highly efficient manganese-catalyzed method has been developed for the synthesis of C-3 hydrocarboxylated imidazolopyridines. In this process, the C(sp^3)–H bond in acetone was activated by DCP and the acetone radical coupled directly with imidazopyridine compounds, avoiding the preparation of halogenated hydrocarbon compounds which are usually used in conventional alkylation reactions. Both electron-donating and electron-withdrawing groups at different positions of imidazo[1,2-*a*]pyridines are tolerated in the reaction, and they provide the target alkylation products in moderate to good yields. This method provides a potential route for highly regioselective functionalization of imidazopyridines that may find wide applications in pharmaceutical synthesis.

Conflicts of interest

The authors declare no competing financial interest.

Acknowledgements

We thank the National Natural Science Foundation of China (No. 21362022) and the Natural Science Foundation of Jiangxi Province (No. 20192BAB203006) for financial support.

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