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ARTICLE

Ruthenium(II)-catalyzed chemoselective deacylative annulation of 1,3-diones with sulfoxonium ylides *via* C–C bond activation

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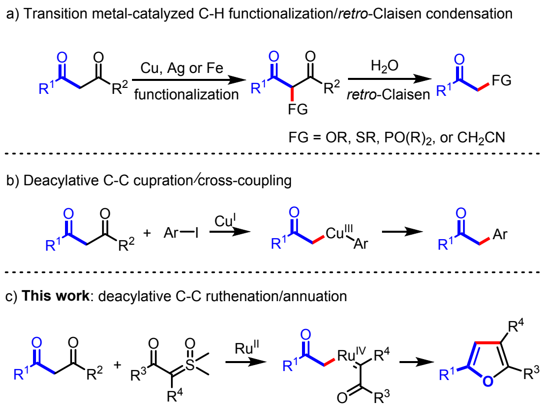
Si Wen, Weiwei Lv, Dan Ba, Jing Liu, and Guolin Cheng*

The first successful example of deacylative annulation of 1,3-diones with sulfoxonium ylides was achieved through Ru(II)-catalyzed C–C bond activation. The excellent chemoselectivity and broad substrate scope render this method a practical and versatile approach for the preparation of (hetero)aryl and alkenyl substituted furans, which are valuable units in many biologically active compounds and functional materials. A preliminary mechanistic study reveals that this process involves a deacylative α-ruthenation to generate the key alkyl Ru(II) intermediate with release of a benzoic acid fragment.

Introduction

Carbon–carbon bonds are the most extensive and basic chemical bonds in organic molecules. The selective cleavage of C–C bonds in a constructive manner enables the reconstitution of the molecular skeleton and introduction of new functional groups, thus attracting great attention.[1] In the past decade, the chemoselective C–C(CO) bond cleavage has been extensively studied by employing the strategies of chelation assistance,[2] ring-strain release,[3] and aromatization-driven.[4] However, the selective cleavage of unstrained C–C(CO) moieties without an auxiliary directing group still remains an unmet challenge[5,6] Recently, transition metal-catalyzed C–C(CO) bond functionalization of 1,3-diones have been achieved by Jiao,[6a] Bolm,[6b] Peng,[6c] and Wu.[6d] These methods are understood to proceed through oxidative α-functionalization of 1,3-diones, followed by *retro*-Claisen condensation to provide α-substituted ketones (Scheme 1a). However, Lei's work demonstrated an alternative reaction pathway for the C–C(CO) bond cleavage of 1,3-diones, in which the deacylative *α*-cupration could occur to form alkyl Cu(III) complexes that subsequently underwent cross-coupling to give α-aryl ketones (Scheme 1b).[7] Inspired by Lei's work, we questioned whether this open shell deacylative α-cupration mechanism might be translated to other transition metal, such as ruthenium, thereby delivering a catalytic formation of alkyl Ru intermediates that can be capture by suitable coupling partners. In continuation of our research on C–C(CO) bond cleavage reactions,[8] herein we disclose the first ruthenium-catalyzed deacylative annulation of 1,3-diones with sulfoxonium ylides (Scheme 1c). This method provides a practical and mild synthetic approach to substituted furans,[9] which are essential structural moieties in many biologically active compounds, natural products, and functional materials.[10]



Scheme 1 Transition metal-catalyzed C–C bond activation of 1,3-diones.

On the other hand, sulfoxonium ylides are readily available and bench-stable carbene precursors, which have been extensively explored for the transition metal-catalyzed functionalization of C–H bonds.[11] However, sulfoxonium ylide carbene-involved C–C bond functionalization has not yet been realized due to the challenging to control the chemoselectivity from the same starting materials.[12]

Results and discussion

We initiated our investigation on the model reaction of 1,3-diphenylpropane-1,3-dione **(1a)** with sulfoxonium ylide (**2a**) to optimize various reaction parameters. The results are summarized in Table 1. With $[RuCl_2(p\text{-cymene})]_2$ being a catalyst, $MesCO_2H$ being an additive, and Na_3PO_4 being a base, the desired reaction did occur in HFIP to afford the desired furan product (**3aa**) in 23% yield (entry 1). However, the C–H

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[a] *College of Materials Science & Engineering, Huaqiao University, Xiamen 361021, China. E-mail: glcheng@hqu.edu.cn*

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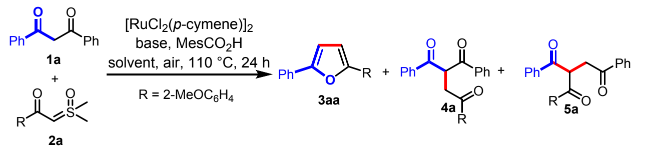
Table 1 Selected optimization studies[a]

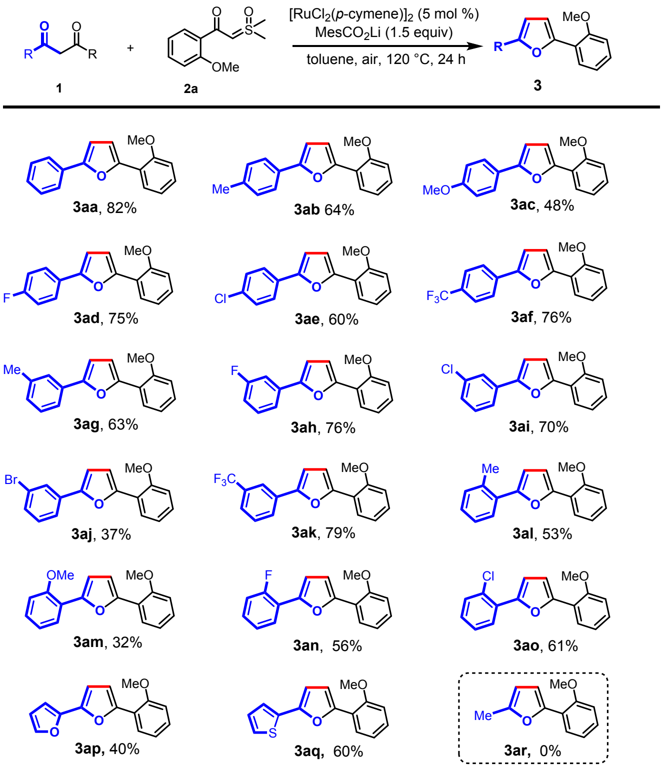


Entry	Solvent	Base	Yield (%)[b]	
			3aa	**4a+5a (4a:5a)**
1	HFIP	Na_3PO_4	23	45 (1:6)
2	^{i}PrOH	Na_3PO_4	26	8 (1:3)
3	DMF	Na_3PO_4	30	20 (1:3)
4	CH_3CN	Na_3PO_4	25	14 (1:2)
5	DCE	Na_3PO_4	15	24 (1:5)
6	toluene	Na_3PO_4	35	trace
7	toluene	Na_2CO_3	30	trace
8	toluene	K_2CO_3	20	trace
9	toluene	Cs_2CO_3	25	trace
10	toluene	$NaHCO_3$	24	0
11	toluene	KH_2PO_4	52	0
12	toluene	^{t}BuOLi	72	trace
13	toluene	-	10	0
14[c]	toluene	^{t}BuOLi	trace	0
15[c]	toluene	$MesCO_2Li$	71	trace
16[c,d]	toluene	$MesCO_2Li$	78	trace
17[c,e]	toluene	$MesCO_2Li$	40	trace
18[c,d,f]	toluene	$MesCO_2Li$	85(82)[g]	trace
19[c,d,h]	toluene	$MesCO_2Li$	66	trace
20[c,d,i]	toluene	$MesCO_2Li$	0	0

[a] Reaction conditions: except where otherwise noted, all of the reactions were performed with **1a** (0.1 mmol), **2a** (0.2 mmol), base (0.15 mmol), $MesCO_2H$ (0.15 mmol), and $[RuCl_2(p\text{-cymene})]_2$ (5 mol %) in solvent (1 mL) at 110 °C under air for 24 h. [b] The yields were determined by ^1H NMR analysis of the crude product using CH_2Br_2 as the internal standard. [c] without of $MesCO_2H$. [d] Reaction was carried out at 120 °C. [e] Reaction was carried out at 130 °C. [f] 2 mL of toluene was used. [g] Isolated yield. [h] 3 mL of toluene was used. [i] Without $[RuCl_2(p\text{-cymene})]_2$. HFIP = 1,1,1,3,3,3-hexafluoro-2-propanol. DMF = *N*,*N*-dimethylformamide. DCE = 1,2-dichloroethane. $MesCO_2H$ = 2,4,6-trimethylbenzoic acid.

carbene insertion product (**4a**)[12a,b] and the C–C carbene insertion product (**5a**)[12b,c] were also obtained as an inseparable mixture in 45% combined yield with 1:6 chemoselectivity. Investigations on various solvents indicated that toluene performed better than others, affording (**3aa**) in a yield of 35% with excellent chemoselectivity (Table 1, entries 1–6). The use of a proper base was crucial for this reaction, and the exploration of different bases revealed that ^{t}BuOLi provided the best yield of 72% (entries 7–12). Only 10% yield of (**3aa**) was obtained in the absence of a base (entry 13). The $MesCO_2H$ additive was proved necessary to ensure the generation of (**3aa**). In the absence of $MesCO_2H$, trace of (**3aa**) was observed (entry 14). A comparative yield was observed when $MesCO_2Li$ was used instead of ^{t}BuOLi and $MesCO_2H$ (entry 15). The yield of (**3aa**) could be improved to 78% when the reaction was run at elevated temperature (entry 16). However, lower yield was obtained when the reaction was carried out at 130 °C (entry 17). Excitedly, when the solvent was increased to 2 mL, the desired product (**3aa**) was obtained in 85% yield (entry 18), but when the solvent was further increased to 3 mL, the yield of (**3aa**) was reduced to 66% (entry 19). Finally, in the absence of a catalyst, no desired product was observed (entry 20). Then, the optimized reaction conditions were identified, as follows: **1a** (0.1 mmol), **2a** (2 equiv), $[RuCl_2(p\text{-cymene})]_2$ (5 mol %), $MesCO_2Li$ (1.5 equiv) in toluene (2 mL) at 120 °C under air for 24 h (entry 18).

With the optimal conditions in hand, we turned our attention to the scope of 1,3-diones for this transformation (Scheme 2). It was found that the 1,3-diones bearing methyl, -methoxy, -halogen, and $-CF_3$ groups all could be smoothly carried out to afford the substituted furan products in moderate to good yields (**3aa–ao**). The structure of (**3ak**) was unambiguously verified by single-crystal X-ray diffraction.[13] The reactivity of this transformation was significantly influenced by the steric hindrance. For 1,3-diones with *ortho*-substituted phenyl rings (**3al–ao**) generally given lower yields of desired products than those with *meta*- and *para*-substituents (**3ab–af**). The electronic properties of the phenyl rings on 1,3-diones were observed to affect the reaction efficiency obviously. The substrates with electron-withdrawing groups (**3ad–af**, **3ah**, **3ai**, and **3ak**) given higher yields than those with electron-donating groups (**3ab**, **3ac**, and **3ag**). However, lower yield was observed when 1,3-dione with a bromo group was used (**3aj**). It was noteworthy that furan and thiophene ring were also tolerated, giving the desired products in moderate yields (**3ap, 3aq**), which could be expected to find wide applications in organic electronics.[10b,14] Finally, this reaction was not applied to pentane-2,4-dione (**3ar**).



Scheme 2 Scope of 1,3-diones. Reaction conditions: **1** (0.1 mmol), **2a** (0.2 mmol), $MesCO_2Li$ (0.15 mmol), and $[RuCl_2(p\text{-cymene})]_2$ (5 mol%) in toluene (2 mL) at 120 °C under air for 24 h.

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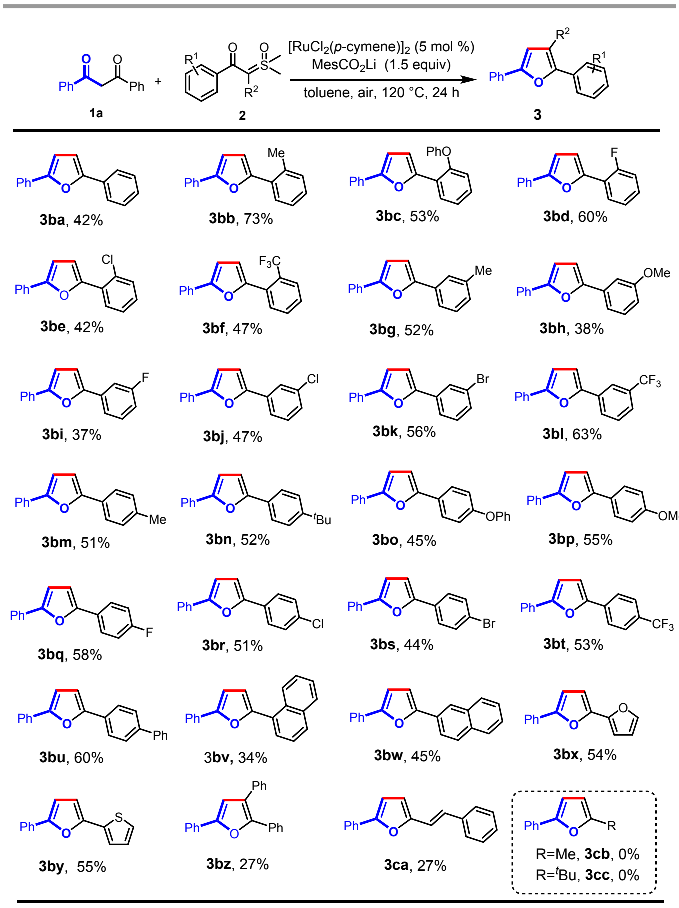
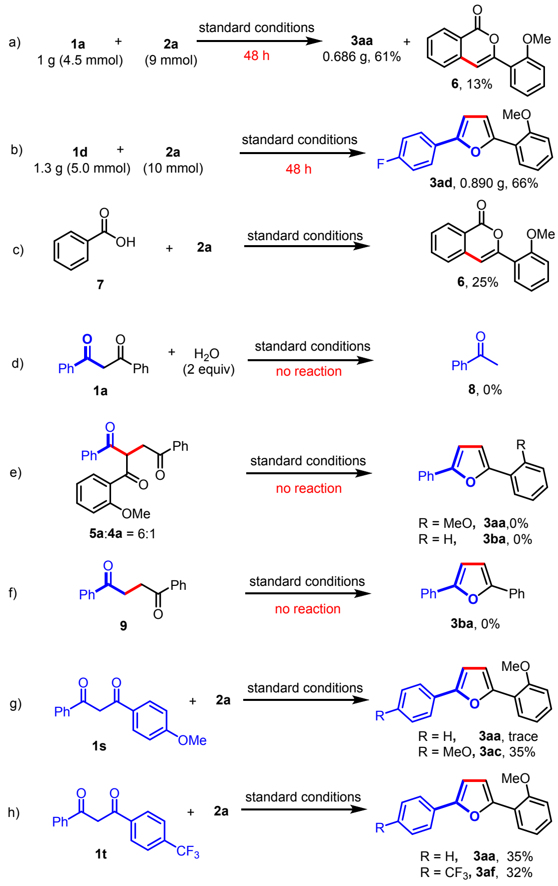


Scheme 3 Scope of sulfoxonium ylides. Reaction conditions: **1a** (0.1 mmol), **2** (0.2 mmol), $MesCO_2Li$ (0.15 mmol), and $[RuCl_2(p\text{-cymene})]_2$ (5 mol%) in toluene (2 mL) at 120 °C under air for 24 h.

Next, we further investigated the reaction of 1,3-diphenylpropane-1,3-dione with a variety of aroyl sulfoxonium ylides under the optimal reaction conditions (Scheme 3). Various valuable functional groups were tolerated, such as methyl, phenyl, phenoxyl, methoxyl, halogen, and trifluoromethyl. The reactivity was not sensitive to the steric hindrance and electronic properties of the phenyl rings on the sulfoxonium ylides. For the *ortho*-position of phenyl ring, having electron-donating moieties (**3ba**–**bc**) and electron-withdrawing groups (**3bd**–**bf**), gave the desired products in moderate to good yields. The substrates with *meta*- and *para*-substituted phenyl ring could be smoothly converted into the desired products in moderate yields (**3bg**–**bt**). Then, a phenyl group was introduced in the *para*-position which formed the tetra(aryl ring)-containing product (**3bu**) in 60% yield. In addition, 1-naphthalenyl (**3bv**), 2-naphthalenyl (**3bw**), 2-furyl (**3bx**), and 2-thienyl (**3by**) substrates were also tolerated, giving the corresponding products in the yields of 34%, 45%, 54%, and 55%, respectively. Importantly, triphenyl substituted furan (**3bz**) could also be obtained using α-phenyl sulfoxonium ylide as substrate, albeit in low yield due to the increased bulkiness. Alkenoyl sulfoxonium ylide was also a capable substrate, giving 2-alkenyl substituted furan (**3ca**) in 27% yield. The aryl/alkenyl groups in conjugation with the carbonyl are indispensable moieties for the successful formation of the corresponding furans, and the sulfoxonium ylides with alkanoyl groups failed to provide the desired products (**3cb** and **3cc**).

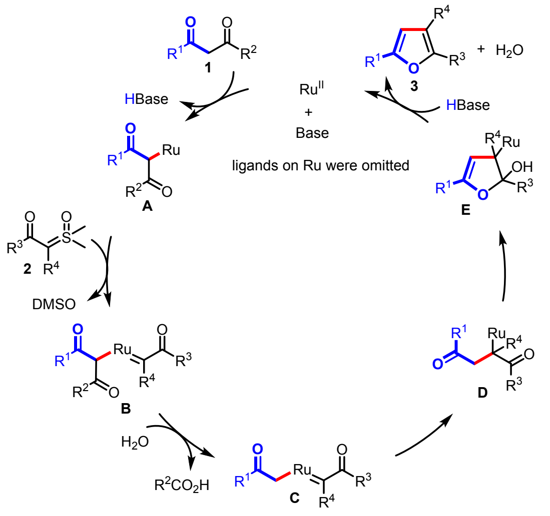


Scheme 4 Gram-scale reactions and control experiments.

A gram-scale experiment of this deacylative annulation was demonstrated employing (**1a**) and (**2a**) as model substrates; the product (**3aa**) was obtained in 61% yield, along with an isocoumarin byproduct (**6**) (Scheme 4a). The 5 mmol scale experiment of (**1d**) and (**2a**) could give the product (**3ad**) in 66% yield (Scheme 4b). Ackermann recently reported a Ru(II)/Ag(I)-catalyzed C–H activation/annulation of benzoic acids with sulfoxonium ylides for the synthesis of isocoumarins.[11f] Indeed, we notice that isocoumarin (**6**) could also be generated in our silver-free conditions in 25% yield from benzoic acid (**7**) and (**2a**) (Scheme 4c). These results indicated that benzoic acid might be generated during the C–C bond cleavage process.

To further understand the reaction mechanism, the reaction of 1,3-dione (**1a**) and H_2O was investigated under the standard reaction conditions. No reaction was observed, which indicated that the Ru(II)-catalyzed C–C activation could not occur in the absence of a sulfoxonium ylide (Scheme 4d). Furthermore, the mixture of **5a** and **4a** (6:1) could not afford furan products (**3aa** or **3ba**) under the standard reaction conditions (Scheme 4e). To rule out the possibility that Paal–Knorr furan synthesis is involved in our transformation, we also prepared 1,4-diphenylbutane-1,4-dione (**9**); however, no desired product (**3ba**) was obtained when this 1,4-dione was subjected to the standard reaction conditions (Scheme 4f). Finally, the intramolecular competitive reactions of unsymmetrical 1,3-diones (**1s**) or (**1t**) with (**2a**) indicated that the chemoselectivity of this reaction was affected by the electron density of aryl-group, and the C–C bond cleavage tend to occur at the less electron-rich moieties (Scheme 4g and 4h). The electron-deficient carbonyls are more likely to be attacked by a nucleophile, such as H_2O, which may induce the subsequent C–C bond cleavage.

On the basis of these results, we proposed that the reaction would proceed as showed in the Scheme 5. The transformation begins with the generation of Ru complex (**A**) under basic condition, which is subsequently captured by sulfoxonium ylide to form Ru carbene complex (**B**). Then, the C–C bond activation occurs in the presence of H_2O, giving Ru complex (**C**). Migratory insertion of (**C**) affords intermediate (**D**). Finally, intramolecular annuation of (**D**) results in intermediate (**E**), following by β-O elimination to finish the furan products (**3**) and regenerate the Ru(II) catalyst.



Scheme 5 Plausible catalytic cycle.

Conclusions

In conclusion, we have demonstrated the first example of Ru(II)-catalyzed chemoselective deacylative annulation of 1,3-diones with sulfoxonium ylides. A series of substituted furans have been synthesized in reasonable yields from this novel method. This protocol that use of unstrained C–C(CO) bond as nucleophiles in transition metal-catalyzed cross couplings should be expected to find wide implications. More work to better understand the mechanistic information of this strategy is currently underway

Conflicts of interest

There are no conflicts to declare.

Acknowledgements

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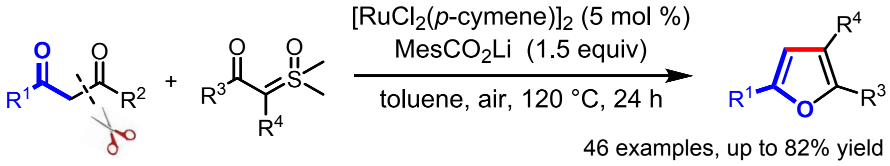

The highly chemoselective Ru(II)-catalyzed deacylative annulation of 1,3-diones with sulfoxonium ylides was achieved to deliver (hetero)aryl substituted furans.