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Electrochemical dehydrogenative cross-coupling of xanthenes with ketones

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A new external oxidant-free electrochemical dehydrogenative cross-coupling of xanthenes and ketones for the preparation of functionalized 9-alkyl-9*H*-xanthenes was developed. This method enables the fromation of a new $C(sp^3)$ - $C(sp^3)$ bond through release of H₂ as the major byproduct at room temperature, and features mild conditions, high atom economy, excellent functional-group tolerance, scalability and facile applications in pharmaceutical chemistry.

Functionalized xanthenes not only are valuable skeleton present in a broad range of pharmaceuticals and bioactive natural products but also are used as versatile fluorescent materials in catalytic chemistry (Figure 1).¹ For example, 1*H*-Indene-1,3(2*H*)-dione is known as important antitumor agents.^{1a} As a result, much efforts over the last few decades have been devoted to construct or modify the active compounds.²



Figure 1. Examples of important functionalized xanthenes.

Direct C-C coupling reaction which using oxygen as the oxidant has been regarded as a fascinating strategy to formation C-C bonds.^{3a-b} For instance, the group of Klussmann^{3c-e} reported a series of aerobic organocatalytic oxidative C-C bond formation reaction of xanthene derivatives with various C-nucleophiles and showed the

possible hydroperoxide intermediate in this transformation (Scheme 1a). Although these methods are efficient, the requirements for high pressure O_2 , longer reaction time, substrate scope and lower yields are drawbacks, which limited the application of this transformation. Therefore, it is attracting and promising to find a mild, efficient, eco-friendly, and safe synthetic strategy to construct this valuable chemical structure.⁴



Scheme 1. Electrochemical dehydrogenative cross-coupling reactions of xanthenes with nucleophiles.

In the past several years, electrochemical anodic oxidation has witnessed rapid development and attracted a great deal of interest in organic synthetic chemistry due to its highly efficient and environmentally benign features.⁵ In particular, electro-oxidative dehydrogenative cross-coupling reactions have been proven to be a promising and feasible alternative to construct new C-C bonds or C-X (X = N, O, etc...) bonds.⁶ For instance, in 2017, zeng and co-workers realized a ferrocene catalyzed intermolecular dehydrogenative reactions of xanthenes with *N*-alkoxyamides under electrochemical conditions for C-N bond construction (Scheme 1b).^{7a} To the best of our knowledge, the direct electrochemical for the construction $C(sp^3)$ - $C(sp^3)$ bond under oxidant-free conditions is rare and still a challenge in organic

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chemistry.⁸ With our continuing interest in green organic synthesis, here, we report a clean and practical electro-oxidative dehydrogenative cross-coupling of xanthenes with ketones to synthesize 9-alkyl substituted 9*H*-xanthenes under mild conditions without an external oxidant (Scheme 1c). This electrooxidative reaction enables the formation of C(sp³)-C(sp³) bond *via* a C-H functionalization process, which is characterized by its mild conditions, high atom economy, excellent functional-group tolerance and scalability.

Table 1 Screening of optimal reaction conditions.^a

ĺ	C(+)/PI(-), I = 5 mA 'Bu ₄ /NBF ₄ (0.05 M) 1a 2a C(+)/PI(-), I = 5 mA 'Bu ₄ /NBF ₄ (0.05 M) MSOH (2 equiv), MeCN, rt, 2 h	George Saa
Entry	Variation from the standard conditions	Yield (%) ^b
1	none	82
2	Pt(+)/C(-) instead of C(+)/Pt(-)	58
3	C(+)/C(-) instead of C(+)/Pt(-)	60
4	no electric current	0
5	ⁿ Bu ₄ NPF ₆ instead of ⁿ Bu ₄ NBF ₄	66
6	LiClO ₄ instead of ⁿ Bu ₄ NBF ₄	55
7	TBAI instead of ⁿ Bu ₄ NBF ₄	trace
8	TBAB instead of ⁿ Bu ₄ NBF ₄	50
9	without MsOH	0 ^c
10	<i>p</i> -TsOH instead of MsOH	73
11	BF ₃ ·Et ₂ O instead of MsOH	48
12	CH ₃ OH instead of MeCN	60
13	DMSO instead of MeCN	0
14	DCM instead of MeCN	30
15	Ar	80
16 ^d	none	78
17 ^e	none	80
^a Reaction conditions: 1a (0.25 mmol), 2a (0.5 mmol), carbon rod		
anode Pt plate cathode constant current = 5 mA MsOH (2)		

anode, Pt plate cathode, constant current = 5 mA, MsOH (2 equiv), $^{n}Bu_4NPF_6$ (0.4 mmol), MeCN (8 mL), undivided cell, at room temperature for 2 h. b Isolated yield. c 9*H*-xanthen-9-one (4) of 75% yield. d 1a (1g, 5.5 mol) for 18 h. e By IKA Electrasyn.

We commenced the study by taking 9H-xanthene (1a) as the representative substrate and cyclopentanone (2a) as the partner, employing our previously optimized conditions for cross-coupling reactions under electrooxidative condition. As indicated in Table 1, the electrolyses was carried out in a single compartment cell under constant-current condition (5 mA) using carbon rod as working electrode and platinum plate as cathode electrode. As expected, 82% yield of 2-(9H-xanthen-9-yl)cyclopentan-1-one (3aa) was obtained when the reaction was happen in the presence of ⁿBu₄NBF₄ as the electrolyte, CH₃CN as the solvent and MsOH as additive at room temperature (entry 1). Two other combinations of the electrodes were attempted and when carbon rod anode was replaced by Pt plate or the Pt plate cathode was replaced by carbon rod, the efficiency of this transformation was decreased (entries 2-3). However, no desired product could be obtained without electric current (entry 4). The choice of electrolyte has also affected the reaction efficiency. When we switched from ⁿBu₄NBF₄ to nBu₄NPF₆,

the reaction proceeded smoothly and gave the desire product 3aa with lower yield (66%) (entry 5). Additionally, 10ther/electrolytes, such as LiClO₄, TBAI and TBAB, were also investigated. As compared with ammonium salts electrolyte, LiClO₄ has showed decreased reaction efficiency due to its low conductivity in the reaction (entry 6). Notably, TBAI as the electrolyte failed to deliver the desired product 3aa (entry 7), while 50% yield of product 3aa was isolated when TBAB as the electrolyte (entry 8). Unfortunately, the desired product could not be detected under no MsOH condition, and gave 75% yield oxidation by-product 9H-xanthen-9-one (4) (entry 9). In addition, other additive acids, such as p-TsOH and BF₃·C₂H₅O were examined for this transformation, but they were shown to be less effective (entries 10 and 11). In this transformation, MsOH may promote the enol isomerization of ketones, which in turn facilitates the nucleophilic reaction.^{3c-e} Other solvents, namely CH₃OH, DMSO and DCM, were also investigated and MeCN was found to be the optimal one (entry 1 vs entries 12-14). Almost the same yield can be retained when the reaction worked at argon atmosphere (entry 15). Notably, a 5.5 mmol scale reaction was conducted and 3aa could be obtained in 78% yield (entry 16). The same result (80%) was also obtained by the use of IKA Electrasyn 2.0 equipment (entry 17).

With the optimized reaction conditions in hand, we next investigated the substrate scope of this electrochemical crossdehydrogenative coupling reaction and studied different ketones (Scheme 2). Different substituted ketones, including cyclic ketones, heterocyclic ketones, α , β -unsaturated ketones, open-chain ketones, 1,3-diketones and acetophenones, proved compatible with the reaction conditions, and gave the corresponding products 3ab-az and **3aA-aC** in moderate to good yields. Cyclic ketones cyclohexanone 2b and cycloheptanone 2c were performed smoothly with 9H-xanthene (1a) to afford the corresponding 9ketone substituted xanthene derivatives 3ab and 3ac in 68% and 83% yields, respectively. To our surprise, 12-membered cyclic ketone cyclododecanone 2d was also tolerated and afford the 2-(9H-xanthen-9-yl)cyclododecan-1-one 3ad in 70% yield. Different substituted cyclic ketones reacted with 9H-xanthenes 1a, affording the corresponding 9-keto xanthenes **3ae-ag** in synthetically useful yields (40-69%). For heterocyclic ketone substrates 2h and 2i, corresponding products were isolated with moderate yields (Products 3ah and 3ai). Benzocyclones 2j-20 were successfully cross-dehydrogenative coupling with 9H-xanthene leading to 3aj-ao in 40-71% yields. To our delight, two cyclic α , β -unsaturated ketones, cyclopent-2-en-1-one 2p and cyclohex-2-en-1-one 2q participated well. It is noteworthy that acetophenones 2r-t, which carry electron-rich group (Me) or electron-poor substituents (such as Cl and NO₂ groups) furnished products **3ar-at**, albeit in 38-51% yields. Among these transformations, open-chain ketones 2i-2y were suitable for the electrochemical oxidative conditions, which provided the desired products **3ai-ay** in 49-80% yields. For example, phosphate ester substrate diethyl (2-oxo-2phenylethyl)phosphonate 2w was translated to the corresponding (2-oxo-2-phenyl-1-(9H-xanthen-9product diethyl yl)ethyl)phosphonate 3aw in 80% yield. In addition, 1,3-diones (2z and 2A) and keto esters (2B and 2C) were also studied, providing the corresponding xanthene derivatives 3az and 3aA-aC in 51-86% yields. Notably, three drug molecules 5α -Dihydroprogesterone **2D**, Progesterone 2E and Canrenone 2F were compatible with the

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electrochemical oxidative conditions, and led to the complex compounds **3aD-aF** in 60%, 67% and 62% yields, respectively.

Table 2. Substrate scope of ketones (2).^a



^{*a*} Reaction conditions: carbon rod anode, Pt plate cathode, constant current = 5 mA, **1a** (0.25 mmol), **2** (0.5 mmol), MsOH (2 equiv), ${}^{n}Bu_{4}NBF_{4}$ (0.4 mmol), MeCN (8 mL), undivided cell, at room temperature for 2 h.

We also studied the scope for electrochemical crossdehydrogenative coupling reactions of with xanthenes **1** using cyclopentanone **2a** as the coupling partner and the results was shown in Table 3. In general, both electron-donating and electronwithdrawing substituents, such as Me, Cl, OH, OMe and F groups, on the benzene ring of xanthenes were well-tolerated in this reaction. For example, xanthene **1d** bearing an unprotected hydroxyl group (was also tolerated leading to 2-(2-hydroxy-9Hxanthen-9-yl)cyclopentan-1-one **3da** in 42% yield. Similarly, substrates with naphthalene ring showed tolerance for the optimized conditions (Products **3fa-ha**). We also found the reaction between 2-fluoro-9H-xanthene **1i** and 1,3-diphenylpropane-1,3dione **2A** was also worked well, furnished the 2_{7} was also worked well, furnished the 2_{7} with a standard the st

Table 3. Substrate scope of xanthenes (1).^a



^{*a*} Reaction conditions: carbon rod anode, Pt plate cathode, constant current = 5 mA, **1** (0.25 mmol), **2a** (0.5 mmol), MsOH (2 equiv), ⁿBu₄NBF₄ (0.4 mmol), MeCN (8 mL), undivided cell, at room temperature for 2 h.

Based on the previous reports, CV results and our experimental results, a plausible reaction mechanism for the electrochemical synthesis of xanthenes was proposed in Scheme $3^{.3, 9\cdot 10}$ The initial step involves losing one electron from the xanthene forming a short-lived radical cation **A** as the first intermediate. Since two oxidation peak is observed in the cyclic voltammetric curve (See Figure S2). Intermediate **C** is formed from **A** *via* twice SET process. The stabilized carbocation C will react with the nucleophilic enol **D** which formation from 2a in the presence of MsOH to yield product **3aa**. At the cathode, H⁺ are reduced to generate H₂, obviating the need for electron and proton acceptors for the electrochemical dehydrogenative cross-coupling reaction.



Scheme 3. Possible reaction mechanism.

In summary, we have established an electro-oxidative dehydrogenative cross-coupling of xanthenes with ketones to produce the important valuable molecules under green, simple, and mild conditions. This reaction avoided the use of external oxidants and H₂ was the only byproduct. The developed methodology is able to accommodate a wide variety of ketones, including cyclic ketones, heterocyclic ketones, α , β -unsaturated ketones, open-chain ketones, 1,3-diketones, β -keto esters, acetophenones even drug ketone

molecules with a α -C(sp³)-H bond. We have also proposed a possible mechanism based on experimental evidence. Further studies involving the application of the developed electro-oxidative methodology to the synthesis of heterocyclic molecules are ongoing in our laboratory.

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Conflicts of interest

The authors declare no conflict of interest.

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