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Cobaltaelectro-Catalyzed Oxidative Allene Annulation by Electro-Removable Hydrazides

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An efficient C-H/N-H functionalization with allenes was enabled *via* versatile electro-oxidative cobalt catalysis. Thus, electrochemical C-H activations were accomplished with high levels of chemoselectivity and regioselectivity in an operationally simple undivided cell setup. The user-friendly nature of this protocol was highlighted by excellent functional group tolerance, electro-reductive removable hydrazide directing group and easy scalability. Experimental mechanistic studies were indicative of a facile BIES C-H cobaltation event.

Inexpensive cobalt-catalyzed oxidative C-H functionalization¹ becomes as an increasingly competent tool for the construction of C–C and C–Het bonds in modern synthetic organic chemistry. In this context, Daugulis uncovered an oxidative cobalt-catalyzed C-H/N-H functionalization with alkynes with the aid of chemical oxidants.² Thereafter, a variety of oxidative C-H activations were achieved via cobalt catalysis.³ Despite significant progress, these transformations generally required excess of chemical oxidants, such as silver(I),⁴ copper(II),⁵ cerium(IV),⁶ manganese(II/III),⁷ and molecular oxygen,⁸ among others. Therefore, byproduct formation was inevitable, which is jeopardizing the sustainable nature of the C-H activation strategy. By contrast, the Ackermann group devised the first electrochemical cobaltcatalyzed C-H activation in 2017.9 Thus, electrons were directly utilized as redox reagents¹⁰ to avoid the consumption

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of external oxidants and prevent the formation of undesired byproducts.¹¹

Allenes have been widely employed in molecular assembling,¹² functional material,¹³ medicines and natural products.¹⁴ Recently, significant advance has been made in the field of transition metal catalyzed C-H functionalization with allenes exploring, inter alia, iridium,15 rhodium,16 palladium,17 ruthenium,¹⁸ manganese,¹⁹ cobalt²⁰ and nickel²¹ catalyst. In this regard, the Zhai group very recently disclosed a novel cobalt-catalyzed trifunctionalization of allenes by a hydrazide directing group (Figure 1a).^{20b} Thus, various 3-acylquinolines were accessed step-economically with Ag₂CO₃ as the chemical co-oxidant. As a part of our program on sustainable cobaltcatalyzed C–H activation,^{8b, 11c, 22} we have now reported here in a novel electrochemical C-H annulation with allenes (Figure 1b). Notable features of our finding include (a) regioselective electrocatalytic C-H/N-H annulation with allenes, (b) inexpensive, Cp*-free cobalt catalyst, (c) operationally convenient undivided cell setup under mild conditions, (d) electro-removable hydrazide directing group,^{11c} and (e) experimental mechanistic studies towards electrocatalytic allene functionalization.



Figure 1. Cobaltaelectro-catalyzed C–H/N–H functionalization with allenes by electro-removable hydrazides

We started to investigate the cobaltaelectro-catalyzed oxidative C-H/N-H annuation with allenes utilizing hydrazide **1a** and diphenyl(propa-1,2-dien-1-yl)phosphine oxide **2a** as the model substrates(Table 1 and Table S1 in the Supporting

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Information).²³ To our delight, with an user-friendly undivided cell setup, the desired regioselective C-H annulation product 3aa was obtained in good yield when MeOH or TFE was used as solvent (entries 1-2). While other commonly used aprotic solvents, H_2O or HFIP led to no product formation or diminished yields. ²⁴ The yield was further improved when the reaction was performed at 40 °C (entry 3). Evaluation of alternative additives revealed that NaOAc was slightly better than NaOPiv and PivOH (entries 4-5). A lower yield was observed when a higher current of 4 mA was applied (entry 7). Control experiments demonstrated the key importance of the carboxylate additive, the cobalt catalyst and the electricity (entries 6, 8-9).

Table 1. Optimization of cobaltaelectro-catalyzed C-H activation with allene 2a

O H H H	Ме N< _{2-Ру} +	Ph ^{-P^{-O}} Ph ^{-Ph} 2a	ACC) ₂ (10 mol %) additive olvent, 15 h, ; Ar, 2.0 mA	Ph P Ph
Entry	Solvent	Additive	t (℃)	Yield (%) ^a
1	MeOH	NaOPiv	23	81
2	TFE	NaOPiv	23	84
3	TFE	NaOPiv	40	87
4	TFE	NaOAc	40	91
5	TFE	PivOH	40	81
6	TFE		40	48
7	TFE	NaOAc	40	69 ^{<i>b</i>}
8	TFE	NaOAc	40	c
9	TFE	NaOAc	40	Trace ^d

^aReaction conditions: 1a (0.55 mmol), 2a (0.50 mmol), Co(OAc)₂ (10 mol %), additive (2.0 equiv), solvent (3.5 mL), undivided cell, CCE at 2.0 mA, 15 h, Pt-plate cathode (1.0 × 1.0 cm), RVC anode (1.0 \times 1.5 cm), under Ar. ^b4.0 mA, 8.0 h. ^c Without cobalt. ^d Without electricity.

Thereafter, to verify the robustness and versatility of the cobaltaelectro-catalyzed C-H annulation approach, a variety of substituted hydrazides 1a were tested under the optimal reaction condition being identified (Scheme 1). Thus, the benzhydrazides bearing electron-donating (e.g. methyl, ipropyl, methoxy, benzyloxy, methylthio) or electron-deficient (e.g. fluoro, chloro, trifluoromethyl, benzyloxy) substituents in the para position were efficiently transformed within the allene annulation manifold (3aa-3na). A set of synthetically useful electrophilic functional groups, such as iodo, bromo, cyano, ketone, amide and ester, was fully compatible, thus enabling further diversification of thus-obtained products. Exclusive site selectivity was observed for the metasubstituted hydrazides, thus only the less sterically hindered o-C-H bond was exclusively functionalized (30a-3ra). Moreover, the ortho-substituted hydrazides also proved to be amenable substrates, albeit gave slightly lower yields, compared with their para- or meta- analogues (3sa-3wa). In addition, arenes bearing multiple electron-rich substituents also furnished the corresponding isoquinolin-1(2H)-one derivatives 3xa-3za with high efficacy.



^a 5 mmol scale 67%; ^b 20 mol % Co(OAc)

Scheme 1. Scope with respect to hydrazides 1

Next, the scope for the differently substituted allenes 2 was investigated for this transformation (Scheme 2). Generally, an excellent regio- and chemo-selectivity was observed, thus the annulation exclusively occurred at the terminal position of the allenes 2. Apart from diphenylphosphine oxide substituted allene 1a, diethyl phosphonate, arene and ester substituted allenes 2b-2f also proved to be viable coupling partners, furnishing the corresponding products in moderate to good yields. In addition, the challenging cyclonona-1,2-diene 2g also displayed good reactivity in this transformation, with two regioisomers observed in a 2.7:1 ratio.23

Finally, the practical use of our electrocatalytic annulation protocol was further illustrated with its easy scalability. Thus, product 3aa was prepared in a comparable yield in a gramscale reaction with an operationally simple undivided cell setup (3aa in Scheme 1). In addition, following our previous reports,^{11c} the *N*-methyl pyridine motif in annulation products 3 was smoothly removed by cathodic electro-reduction via Sml₂ catalysis in an operationally simple undivided cell (Scheme 3).²⁴



Inspired by the outstanding versatility of the cobaltcatalyzed electrooxidative C-H/N-H activation with allenes, a

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set of experiments were performed to rationalize its mechanism(Scheme S1 in the Supporting Information).²³ To this end, electron-rich hydrazide **1b** exhibited better reactivity in an intermolecular competition experiment, which may indicated that a base-assisted internal electrophilic type substitution (BIES) metalation is operative.²⁵ However, this result can also be rationalized by preferential binding of the electron-rich ligand to cobalt(III) species. Moreover, electron withdrawing trifluromethyl substituted allene 2f displayed a relatively lower reactivity as compared with its analogue 2e. Interestingly, no H/D scrambling was observed in either the product 3aa or the re-isolated hydrazide 1a when dueterated methanol was used as a co-solvent. Furthermore, a minor kinetic isotope effect (KIE) was obtained by a parallel experiment, which is indicative of a facile C-H cleavage process.



Scheme 3. Electro-reductive removal of directing group

Next, a series of cyclic voltammetric analyses was performed to investigate the electrochemical C–H activation (Figure 2). Hydrazide **1a** exhibited oxidative peaks at 1.0 V_{SCE} and 1.3 V_{SCE} with an onset potential at 0.76 V_{SCE} (curve b, Figure 2), while no obvious oxidation peaks were observed for allene **2a** (curve c, Figure 2). Notably, the onset potential for oxidation of cobalt(II) precatalyst shifted from 0.77 V_{SCE} (curve d, Figure 2) to 0.60 V_{SCE} and an obvious catalytic current was detected in the presence of hydrazide **1a** (curve e, Figure 2). These observations clearly indicated substrate **1a** being coordinated to cobalt(II), which could facilitate the key anodic oxidation toward cobalt(III) carboxylate complex. No significant quenching was observed for the introduction of allene **2a** into the mixture (curve f, Figure 2).

Based on the above mentioned mechanistic findings and our previous reports, a plausible catalytic cycle was proposed as depicted in Scheme 4. The electrooxidative C–H activation was initiated *via* the anodic oxidation, which was followed by a carboxylated assisted BIES C–H cobaltation to furnish cobalt (III) complex **7**. Then, the regioselective allene insertion and subsequent reductive elimination delivered cobalt(I) complex **10** and the exo-methylene isoquinolone **9**, which could transformed to the desired product **3** *via* isomerization. Next, the catalytically competent cobalt(III) complex **6** was reformed by the key anode oxidation to close the catalytic cycle. Overall, the coboltaelectrocatalysis strategy avoided the consumption of chemical oxidants and molecular hydrogen was released as the only byproduct.



Figure 2. Cyclic voltammograms of n-Bu₄NPF₆ (0.1 M) solutions in MeOH at 0.1 V/s. [Co] = Co(OAc)₂.²³



Scheme 4. Plausible catalytic cycle

In conclusion, we have devised a highly atom-economical electocatalytic C–H annulation with allenes. Thus, C–H/N–H annulations were achieved with remarkable levels of regioand chemo-selectivity *via* an earth-abundant Cp*-free cobalt catalyst. The synthetic power of this protocol was further illustrated by the excellent functional group tolerance, electroreductive removable hydrazide directing group, and easy-scalability. Detailed experimental mechanistic studies revealed a facile carboxylate-assisted BIES C–H cleavage event.

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Conflicts of interest

There are no conflicts to declare.

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