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Enantioselective Functionalization of Inactive sp^3 C–H Bonds Remote to Functional Group by Metal/Organo Cooperative Catalysis

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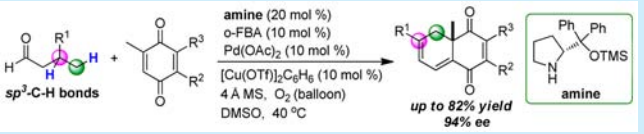
Supporting Information

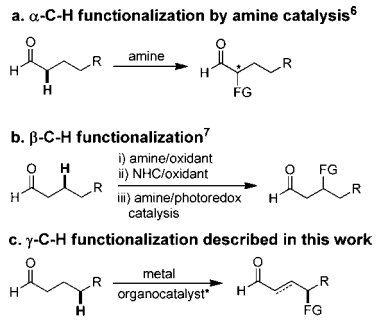
ABSTRACT: A metal/organo cooperative catalysis to enable the enantioselective functionalization of inactive C–H bonds γ to the formyl group in aliphatic aldehydes has been established. Instead of using enals as substrates in traditional organocatalytic cyclization reactions, the aliphatic aldehydes directly participated in [4 + 2] cyclization with quinone derivatives exploiting molecular oxygen as oxidants to afford optically active cyclic molecules with excellent levels of enantioselectivity. This method features a combination of pot, step, and atom economy.



The direct functionalization of inactive carbon–hydrogen bonds for building up structural complexity has been accepted as an ultimate goal in synthetic organic chemistry.[1] Over the last decades, great advances have been achieved in this field, leading to an explosive emergence of a tremendous number of atom-economy protocols,[2] some of which indeed enabled the synthesis of structurally complicated molecules to be more straightforward and efficient.[3] Despite these significant achievements, the catalytic enantioselective functionalization of C–H bonds has met with a great deal of challenges.[4] In particular, the asymmetric functionalization of carbon–hydrogen bonds in alkyl substituents located far away from functionalities appears to be even more challenging. Thus, very few successful examples describe the advances in this field.[5] Linear aliphatic aldehydes are feedstock chemicals and available abundantly. In fact, the organocatalytic stereoselective functionalization of active C–H bonds α to the formyl group is quite reasonable and has been well established.[6] Recently, the enantioselective introduction of functionalities at the carbon β to the carbonyl has also been enabled by asymmetric catalysis.[7] However, the direct functionalization of γ-C–H bonds in these molecules has rarely been described and therefore remains a formidable challenge. Herein, we present an enantioselective functionalization of γ-C–H bonds of aliphatic aldehydes enabled by metal/organo cooperative catalysis[8] (Scheme 1).

Basically, the aliphatic aldehyde **1** is able to undergo a condensation reaction with a chiral amine to form an enamine species **I**, which would then undergo a Saegusa-type oxidation reaction to generate an unsaturated iminium intermediate **II** in the presence of palladium(II) complex,[9] together with the generation of palladium(0), which could be oxidized into palladium(II) by suitable oxidants for the next catalytic cycle. The iminium intermediate **II** principally undergoes an isomerization to form a dienamine species **III**,[10] which would actually be able to participate in an asymmetric coupling reaction with electrophiles to generate either acyclic or cyclic enantioenriched compounds.[11] As such, the Pd/chiral amine combined catalysis would be a general strategy and provide a platform for the enantioselective functionalization of inactive C–H bonds in aliphatic aldehydes remote to the formyl functionality and therefore would enable the creation of new C–H bond activation-based asymmetric reactions (Scheme 2).

Scheme 1. Previous Asymmetric Functionalization of sp^3 C–H Bonds in Aliphatic Aldehydes



The feasibility of the general concept was examined by a reaction of 3-phenylbutanal (**1a**) with 2,6-dimethyl-1,4-benzoquinone (**4a**) in the presence of 10 mol % of $Pd(OAc)_2$ and 20 mol % of a chiral amine in DMSO under oxygen atmosphere at 40 °C (Table 1).[7c,9b] Unfortunately, the use of proline as an organocatalyst led to no reaction (entry 1). MacMillan's catalysts **6b** and **6c**[12] showed very low catalytic activity despite the fact that **6c** gave the desired bicyclic product **5a** in good

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Scheme 2. General Strategy for the Enantioselective Functionalization C−H Bonds γ to the Formyl Group in Aliphatic Aldehydes

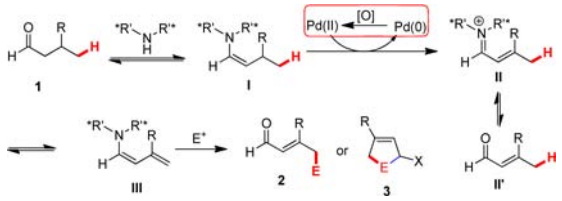
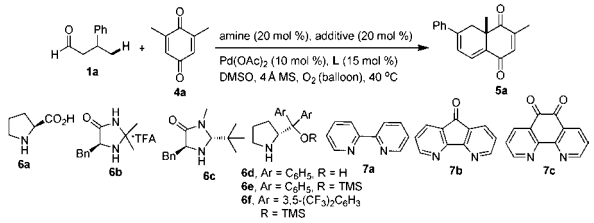


Table 1. Evaluation of Catalysts and Optimization of Reaction Conditions[a]



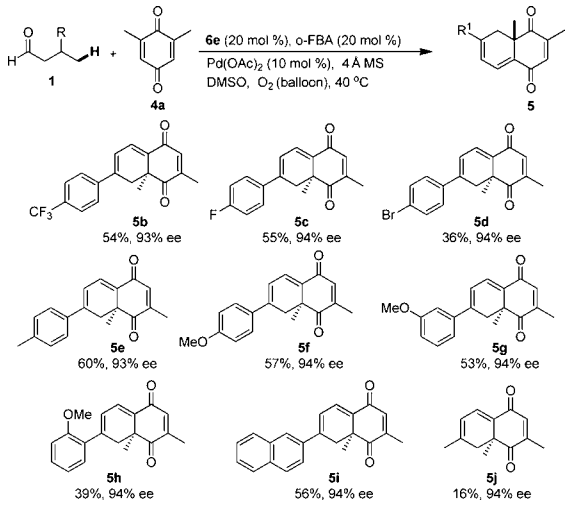
entry	ligand	amine	1a/4a	additive	yield[b] (%)	ee[c] (%)
1	none	**6a**	2/1	*o*-FBA	trace	n.d.
2	none	**6b**	2/1	*o*-FBA	3	82
3	none	**6c**	2/1	*o*-FBA	none	n.d.
4	none	**6d**	2/1	*o*-FBA	7	2
5	none	**6e**	2/1	*o*-FBA	37	90
6	none	**6f**	2/1	*o*-FBA	9	13
7	**7a**	**6e**	2/1	*o*-FBA	29	92
8	**7b**	**6e**	2/1	*o*-FBA	27	92
9	**7c**	**6e**	2/1	*o*-FBA	23	92
10[d]	none	**6e**	2/1	*o*-FBA	45	93
11[d]	none	**6e**	3/1	*o*-FBA	57	92
12[d]	none	**6e**	4/1	*o*-FBA	63	93
13[d,e]	none	**6e**	4/1	*o*-FBA	70 (62[f])	94 (91[f])
14[d,e]	none	**6e**	4/1	BA	63	94
15[d,e]	none	**6e**	4/1	AcOH	59	94

[a]Unless indicated otherwise, the reaction was performed with $Pd(OAc)_2$ (10 mol %), ligand (15 mol %), amine (20 mol %), additive (20 mol %), and 4 Å MS (100 mg) at 0.2 M in DMSO at 40 °C under O_2 (balloon) atmosphere. [b]Isolated yield. [c]Determined by HPLC, and the absolute configuration was assigned by comparing the optical rotation with the literature value.[11c] [d]The reaction was performed at 0.4 M. [e]10 mol % of additive. [f]The reaction was performed with **1a** on a 8.0 mmol scale. DMSO, dimethyl sulfoxide; TMS, trimethylsilyl; *o*-FBA, *o*-fluorobenzoic acid; BA, benzoic acid; AcOH, acetic acid; n.d., not determined.

enantioselectivity of 82% ee (entries 2 and 3). Surprisingly, both **6d** and **6f** showed very low catalytic activity and poor stereocontrol ability (entries 4 and 6), whereas the corresponding silyl ether derivative **6e** turned out to be the best co-catalyst,[11c] capable of delivering a much improved yield and enantiomeric excess (37% yield, 90% ee, entry 5). Previous findings suggested that the addition of bidentate nitrogeneous ligands would be able to accelerate the Pd(II)-catalyzed oxidation reactions.[13] Thus, typical ligands such as **7a−c** were examined for the reaction but resulted in diminished yields albeit with excellent levels of enantioselectivity (entries 7−9). Interestingly, a much enhanced yield could be obtained by conducting the reaction at a higher concentration (entry 10). Tuning the ratio of aldehyde **1a** to **4a** indicated that the presence of 4 equiv of aldehyde **1a** resulted in the generation of **5a** in fairly good yields and with excellent enantioselectivities (entries 10−12). The use of 10 mol % of acid additive allowed the yield to be greater, still with excellent levels of enantioselectivity (entry 13). Obviously, the acid additive to some degree exerted an impact on the reaction. For example, the replacement of *o*-fluorobenzoic acid with either benzoic acid or acetic acid gave a considerably lower yield, although the stereoselectivity was maintained (entry 13 vs 14 or 15). Notably, the gram-scale reaction of **1a** with **4a** gave consistent results (62% yield, 91% ee, entry 13).

Under the optimized reaction conditions, we next explored the substrate scope with respect to the aldehydes (Scheme 3). A

Scheme 3. Substrate Scope with Respect to Aldehydes[a]



[a]The reaction was performed with $Pd(OAc)_2$ (10 mol %), **6e** (20 mol %), *o*-FBA (10 mol %), and 4 Å MS (100 mg) at 0.4 M in DMSO at 40 °C under O_2 (balloon) atmosphere. Enantiomeric excesses were determined by HPLC, and the absolute configuration was assigned by comparing the optical rotation with the literature value.[11c]

range of 3-arylbutanals (**1b−i**) bearing either electronically deficient or rich aryl substituents were found to smoothly undergo the cascade reaction and afforded the desired products with an all-carbon quaternary stereogenic carbon in moderate to high yields (36−70%) and with high levels of enantioselectivities (93−94% ee). Although the substitution pattern has little effect on the stereoselectivity, it exerts great impact on the reaction conversion. Thus, the introduction of an *ortho*-substituent to the phenyl group led to a much diminished yield, but with a similar enantioselectivity in comparison with aldehydes bearing either *meta*- or *para*-substituted phenyl group (**5h** vs **5f** or **5g**). As for 3-methylbutanal (**1j**), the reaction resulted in a low yield but gave an excellent enantioselectivity (**5j**, 16%, 94% ee). The configuration of **5d** was assigned by comparing the optical rotation of the same compound that was identified by X-ray analysis.[11c]

Subsequently, the reaction conditions were expanded to naphthoquinone derivatives. Disappointingly, the asymmetric dehydrogenative cyclization of 3-phenylbutanal and 2-methyl-1,4-naphthoquinone (**4b**, MNQ) provided **5k** in a very low yield of 11% (Table 2, entry 1), suggesting that the reaction conditions required further optimization. Because the reaction conditions optimized above were efficient to activate the 3-phenylbutanal (Table 1), the low conversion of the whole dehydrogenative cycloaddition process[14] might be attributable to the low reactivity of 2-methyl-1,4-naphthoquinone (MNQ). As a

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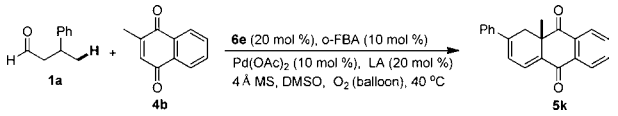
Table 2. Evaluation of Lewis Acid Catalysts for Reactions[a]



entry	LA	yield[b] (%)	ee[c] (%)
1	none	11	92
2[d]	(salen)CrCl	30	91
3	$Sc(OTf)_3$	30	94
4	$Mg(OTf)_2$	11	91
5	$Cu(OTf)_2$	38	91
6[d]	$[Cu(OTf)]_2C_6H_6$	54	91
7	$[Cu(OTf)]_2C_6H_6$	30	80

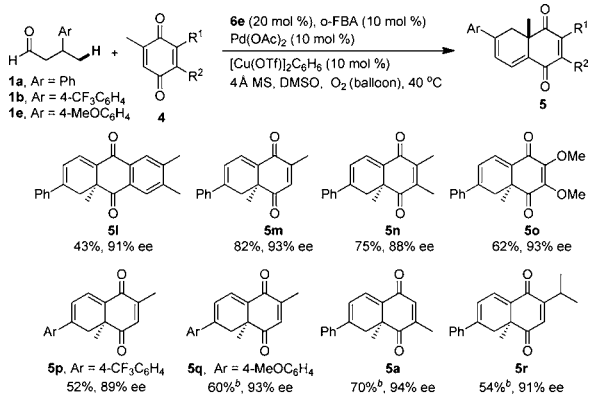
[a]Unless indicated otherwise, the reaction was performed with LA (20 mol %) under standard conditions. [b]Isolated yield. [c]Determined by HPLC using a chiral stationary phase. [d]10 mol % of LA.

consequence, a variety of Lewis acids were examined to activate MNQ (Table 2). Indeed, the use of (salen)CrCl as a co-catalyst was able to significantly improve the yield of **5k** to 30%, and the enantioselectivity could be obtained in 91% ee (entry 2). The evaluation of some other typical Lewis acids found that either Cu(I) or Cu(II) turned out to be a more efficient activator of MNQ and allowed the reaction to give a much enhanced yield, and more significantly, the stereoselectivity was maintained (entries 3−7). In particular, the use of a benzene complex of copper(I) trifluoromethanesulfonate ($[Cu(OTf)]_2C_6H_6$) as a co-catalyst led to a much improved yield with 91% ee (entry 6). However, the addition of additional amounts of Lewis acid to the reaction was obviously deleterious to control the stereoselectivity, and even the yield was surprisingly sacrificed (entry 7).

The ternary catalyst system optimized was then applied to the reaction of different quinone derivatives (Scheme 4). Indeed, the 2,6,7-trimethyl-1,4-naphthoquinone (**4c**) smoothly underwent the asymmetric dehydrogenative cycloaddition to furnish the corresponding polycyclic product **5l** in a moderate yield of 43% and with high enantioselectivity of 91% ee. Notably, 2,5-dimethyl-1,4-benzoquinone (**4d**) turned out to be a more reactive dienophile and was thus able to provide the product **5m** in an 82% yield and with 93% ee. The installation of substituents to benzoquinones was nicely tolerated to generate chiral bicyclic products in high yields and high levels of enantioselectivity, as exemplified by **5n** and **5o**. Interestingly, the aldehydes bearing an electronically neutral and deficient aryl group underwent a cleaner reaction under the assistance of the copper catalyst to give **5k** (entry 6, Table 2) and **5p** (Scheme 4) in higher yields, respectively, whereas a lower yield was provided in the presence of Lewis acid co-catalyst when the aldehyde with an electronically rich aryl substituent was used, as indicated by results related to **5q** (also see Table S1, Supporting Information). Moreover, both 2,6-dimethylbenzoquinone and 2-isopropyl-5-methylbenzoquinone were able to participate in a cleaner asymmetric dehydrogenative cycloaddition in the absence of copper Lewis acid and furnished **5a** and **5r** in good yields and excellent enantioselectivity (also see Table S1, Supporting Information). These unusually low yields in the cases with Lewis acid cocatalyst might arise from the aerobic oxidation of unsaturated aldehydes **II′** generated from Saegusa-type oxidation (Scheme 2) into carboxylic acid catalyzed by the copper complex[15] to compete against the desired asymmetric cycloaddition and thereby resulted in some side reactions to erode the yields of cycloaddition products.

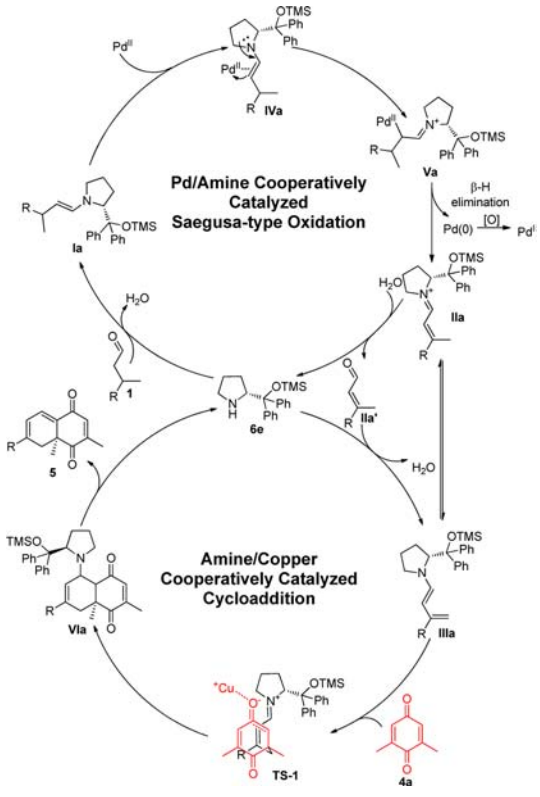
Basically, the asymmetric dehydrogenative cycloaddition proceeds via two individual reaction sequences.[9b] As shown in Scheme 5, the amine catalyst **6e** initially reacted with an aldehyde **1** to generate **Ia**, which undergoes the Saegusa-type oxidation via key intermediates **IVa** and **Va** to give an unsaturated iminium species **IIa**. Meanwhile, the palladium(0) species generated from the β-H elimination in this Saegusa-type oxidation was oxidized

Scheme 4. Substrate Scope of Dehydrogenative Cycloaddition Catalyzed by a Ternary System[a]



[a]Unless indicated otherwise, the reaction was performed with $Pd(OAc)_2$ (10 mol %), **6e** (20 mol %), o-FBA (10 mol %), $[Cu(OTf)]_2Ph$ (10 mol %), and 4 Å MS (100 mg) at 0.4 M in DMSO at 40 °C under O_2 (balloon) atmosphere. Isolated yield is shown. Enantiomeric excesses were determined by HPLC using a chiral stationary phase. [b]In the absence of $[Cu(OTf)]_2C_6H_6$.

Scheme 5. Proposed Reaction Mechanism for the Asymmetric Dehydrogenative Cycloaddition Reaction



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to catalytically active palladium(II) in an oxygen atmosphere.[16] The iminium **IIa** could be hydrolyzed into an unsaturated aldehyde **IIa′** that would be able to react again with the chiral amine **6e** to generate a dienamine **IIIa**. Alternatively, the intermediate **IIa** might be possible to directly isomerize into the dienamine **IIIa**. Subsequently, the [4 + 2] cycloaddition reaction of the dienamine **IIIa** with the quinone dienophile such as **4a** occurs[10] wherein the addition of the γ-carbon atom of dienamine **IIIa** to quinone **4a** results in the formation of an intermediate **VIa** via a zwitterionic transition state **TS-1**,[11c] in which the negative charge of quinone is mainly localized on the oxygen atom and is stabilized either by imine cation or the coordination to copper(I) trifluoromethanesulfonate ($[Cu(OTf)]_2C_6H_6$) (in the reactions using LA as co-catalyst). Finally, the intermediate **VIa** undergoes elimination to generate the product **5** and release the catalyst **6e**.

In summary, we have developed a metal/organo cooperative catalytic strategy to enable the enantioselective functionalization of C−H bonds in alkyl substituents of aldehydes remote to the formyl functionality. As such, the enolizable aliphatic aldehydes densely bearing sp^3 C−H bonds were able to directly undergo the asymmetric dehydrogenative [4 + 2] cyclization reaction to generate chiral bicyclic compounds in good yields and with excellent enantioselectivities. In comparison with classical processes, this method essentially avoids the use of highly reactive enals that are basically prepared from saturated aldehydes and, hence, features the combination of pot, step, and atom economy. More importantly, the concept presented herein indicates a new and robust pathway to functionalize inactive C−H bonds in aliphatic carbonyls.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.5b02653.

Experimental details and characterization data (PDF)

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Notes

The authors declare no competing financial interest.

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