Highly Stereoselective Co(III)-Catalyzed Three-Component C–H Bond Addition Cascade

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Abstract

A highly stereoselective three-component $C(sp^2)$–H bond addition across alkene and polarized π-bonds is reported for which Co(III) catalysis was shown to be much more effective than Rh(III). The reaction proceeds at ambient temperature with both aryl and alkyl enones employed as efficient coupling partners. Moreover, the reaction exhibits extremely broad scope with respect to the aldehyde input with electron rich and poor aromatic, alkenyl, and branched and unbranched alkyl aldehydes all coupling in good yield and with high diastereoselectivity. Multiple directing groups participate in this transformation, including pyrazole, pyridine, and imine functionalities. Both aromatic and alkenyl $C(sp^2)$–H bonds undergo the three-component addition cascade, and the alkenyl addition product can readily be converted to diastereomerically pure 5-membered lactones. Additionally, the first asymmetric reactions with Co(III)-catalyzed C–H functionalization are demonstrated with three-component C–H bond addition cascades employing *N-tert*-butanesulfinyl imines. These examples represent the first transition metal catalyzed C–H bond additions to *N-tert*-butanesulfinyl imines, which have proven to be versatile and extensively used intermediates for the asymmetric synthesis of amines.

Graphical abstract
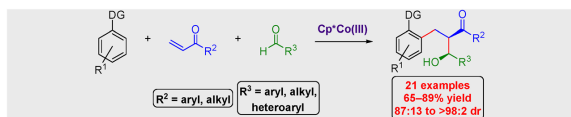
A highly stereoselective three-component $C(sp^2)$–H bond addition across alkene and polarized π-bonds is reported. Co(III) catalysis was much more effective than Rh(III) providing products in good yield and with high diastereoselectivity for an extremely broad range of aldehydes. Additionally, the first asymmetric reactions employing Co(III)-catalyzed C–H functionalization are reported for three-component C–H bond addition cascades with *N-tert*-butanesulfinyl imines.

Keywords

C–H activation; Homogeneous catalysis; Multicomponent reactions; Diastereoselectivity

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C–H functionalization has emerged as a core strategy to access synthetically useful structural motifs, many of which are found in drugs and natural products.[1,2] Of the many transition metal catalysts that facilitate this process, Rh(III) catalysts have been demonstrated to be among the most useful due to their unique reactivity and high functional group compatibility.[3] Following the seminal reports of Co(III)-catalyzed C–H functionalization by Kanai and Matsunaga in 2013,[5ac] a number of groups including our own have demonstrated that earth abundant Co(III) catalysts can provide analogous reactivity to their second row Rh(III) congeners.[4,5] For some transformations divergent reactivity is displayed between Cp*Co(III) and Cp*Rh(III) catalysts,[6] but significant differences in non-annulative redox-neutral couplings have yet to be reported. Recently, we described preliminary results on a three-component C–H functionalization cascade via Rh(III)-catalysis (Figure 1a).[7] Though the transformation proceeded in good yield, very poor diastereoselectivity was observed. After thorough investigation, only ethyl glyoxylate proved to be reactive in the three-component transformation (Figure 1a–b).

Here we report that Cp*Co(III)-catalysts not only facilitate three-component additions to a wide array of aldehydes, including electron poor and rich aromatic, alkenyl, and branched and unbranched alkyl derivatives, but that the transformation also proceeds with high diastereoselectivities ranging from 87:13 to >98:2 dr (Figure 1c).[8.9] Moreover, we disclose the first asymmetric C–H bond functionalization reaction employing Co(III) with three-component C–H bond addition cascades to *N-tert*-butanesulfinyl imines (Figure 1d).

Experimental Section

Our initial investigation of non-activated aldehydes in the three-component coupling reaction began with the conditions previously developed for Rh(III) catalysis using ethyl glyoxylate (see Supplemental Optimization Table in Supporting Information).[7] Unfortunately, attempts at obtaining the desired alcohol products such as **4a** were unsuccessful leading only to high yields of the two-component product **5a** (Table 1). We next investigated Co(III)-catalysis and first evaluated Kanai and Matsunaga's preformed catalyst [Cp*Co(C_6H_6)][PF_6]$_2$ because this complex had previously been shown to facilitate C–H bond additions to electron deficient alkenes.[5ac] The desired product **4a** was formed in 65% yield and with high diastereoselectivity (95:5 dr) (entry 1, Table 1). Notably, for ethyl vinyl ketone (**2a**) a single regioisomer of the three-component product was obtained, establishing that equilibration to regioisomeric enolates does not occur after addition to the enone. In an effort to enhance the diastereoselectivity of this transformation we next ran the reaction at ambient temperature (23 °C), but noticed minimal conversion (entry 2). In contrast, employing the preformed catalyst [Cp*Co(C_6H_6)][B(C_6F_5)$_4$]$_2$ previously developed in our laboratory for the direct C–H bond addition to aldehydes,[5v] we found that the reaction proceeded at ambient temperature in 61% yield and with an improved 96:4 dr (entry 3). Exploring other acetate salts such as cesium or lithium acetate provided similar yields (entries 4–5). Furthermore, upon lowering the equivalents of benzaldehyde (**3a**) with LiOAc as the additive, the yield was maintained along with a slight improvement in diastereoselectivity (entry 6).

Employing the precatalyst [Cp*Co(CO)I$_2$] in conjunction with AgB(C$_6$F$_5$)$_4$ under the optimal conditions provided comparable yield and diastereoselectivity to that achieved with the preformed catalyst (entry 7). However, because the preformed catalyst [Cp*Co(C$_6$H$_6$)] [B(C$_6$F$_5$)$_4$)]$_2$ simplifies reaction set up and also is prepared without the use of any precious metals,[5v] this preformed catalyst was used in all subsequent experiments. Finally, when the precatalyst [Cp*RhCl$_2$]$_2$ with AgB(C$_6$F$_5$)$_4$ was used under the optimal reaction conditions for Co(III)-catalysis, the desired product **4a** was obtained in only trace amounts with high yields of the two-component addition product **5a** instead obtained (entry 8). This result highlights the vast superiority of Co(III) over Rh(III) catalysis for this three-component coupling reaction. When the Co(III) catalyst is absent no reaction is observed, while the removal of the LiOAc additive results in some product but with reduced yields and conversion for most substrates (see Supplemental Optimization Table).

After determining optimal reaction conditions, we next turned our attention to evaluating the scope of the three-component reaction (Table 2). Initially, a variety of enones were investigated. Phenyl vinyl ketone (**2b**) proved to be an effective coupling partner leading to high yields of products **4b** and **4c** with good diastereoselectivity. Methyl vinyl ketone also displayed good reactivity furnishing product **4d** in 69% yield and 95:5 dr. Following this, a series of aldehydes were then subjected to the three-component reaction. Electron-rich heteroaryl aldehydes coupled efficiently to give products **4e** and **4f** in good yields and with 98:2 dr. For aryl aldehydes, strong and weakly donating substituents provided products **4g** and **4j**, respectively, in good yield and with high diastereoselectivity. Benzaldehydes substituted with weakly and strongly electron deficient groups such as 4-bromo (**4h**), 4-methyl ester (**4i**), and 2-fluoro (**4k**) groups also coupled in good yields though with modestly lower diastereoselectivities. Use of the highly activated ethyl glyoxylate provided product **4l** in 51% yield and 75:25 dr, with the lower yield attributed to a competitive direct arene C–H bond addition to the aldehyde (see Supporting Information for details).

In addition to aryl and heteroaryl aldehydes, reactions with more challenging alkenyl and alkyl aldehydes proceeded smoothly. When crotonaldehyde was employed product **4m** was obtained in 66% yield and 96:4 dr. Importantly, competitive 1,4-addition into crotonaldehyde was not observed. The activated benzyloxy acetaldehyde displayed good reactivity providing product **4n** in 65% and 94:6 dr. Linear and branched alkyl aldehydes also underwent successful three-component coupling to give products **4o–s** in high yield and diastereoselectivity. These examples clearly demonstrate that self-condensation side reactions do not compete for enolizable alkyl aldehyde derivatives. Three-component addition product **4q** was isolated as a single diastereomer in 81% yield with the relative stereochemistry for this product rigorously established by X-ray crystallography.[10]

Different directing groups were also explored. In addition to pyrazole, pyridine was an effective directing group for the heteroaromatic C–H functionalization of isoquinolinones (Scheme 1). Notably, both aryl and alkyl aldehydes coupled efficiently to furnish products **4t** and **4u** in good yield and selectivity. Ketimine **1c** also directed the three-component C–H bond addition cascade (Scheme 2). Interestingly, the expected alcohol underwent an additional intramolecular cyclization to provide spirohemiaminal ether **6** in 95:5 dr.

In addition to the functionalization of aryl C(sp^2)–H bonds, participation of alkenyl C(sp^2)–H bonds in the three-component coupling was also demonstrated (Scheme 3). Reaction of alkenyl pyrazole **1d** with ethyl vinyl ketone (**2a**) and benzaldehyde (**3a**) gave **4v** in 77% yield and with 87:13 diastereoselectivity. Further elaboration of **4v** was readily accomplished by ozonolysis to afford a lactol intermediate, which was conveniently oxidized with Fétizon's reagent to provide lactone **7** in diastereomerically pure form after chromatography (Scheme 4). This three-component coupling cascade consequently provides rapid access to gamma lactones, which are present in numerous drugs and natural products, from readily available α,β-unsaturated ketones and aldehydes.

A number of experiments were also conducted to provide insight into the mechanism of the Co(III)-catalyzed three-component reaction (Scheme 5). In the first mechanistic experiment, **5a** was subjected to the optimal reaction conditions in the presence of benzaldehyde (**3a**) (Scheme 5a). The alcohol product **4a** resulting from the three-component transformation was not observed and instead quantitative recovery of **5a** was obtained suggesting that upon release from the catalyst, **5a** does not participate in the catalytic cycle. Next, the three-component coupling of pyrazole **1e** with phenyl vinyl ketone (**2b**) and benzaldehyde (**3a**) was evaluated in the presence of **5a** (Scheme 5b). Under these conditions the expected alcohol product **4c** was formed in only a slightly slower yield than previously observed in the absence of **5a** (Table 2), and **5a** was recovered in 95% yield. This result conclusively indicates that the released conjugate addition product **5a** does not participate in the catalytic cycle, and therefore, the cobalt enolate that reacts with the aldehyde only arises through initial C–H bond addition to enone **2**. To evaluate whether or not alcohol **4a** exists in equilibrium with the conjugate addition product **5a**, alcohol **4a** was subjected to the optimal reaction conditions (Scheme 5c). Complete recovery of **4a** was obtained, and without any loss in stereochemical purity, thereby establishing irreversible formation of the alcohol.

A plausible mechanism for the reaction is depicted in Scheme 6. Coordination of the directing group to the cationic Co(III) catalyst, followed by directed C–H bond metallation provides the active cobaltacycle **8**. Conjugate addition of **8** into the enone coupling partner **2** provides the racemic cobalt enolate **9**.[11] Diastereoselective addition of the cobalt enolate **9** to aldehyde **3** via a chair transition state generates cobalt alkoxide **10**. Release of the product upon proto-demetallation yields the desired alcohol **4** while regenerating the cationic Co(III) catalyst. The high observed diastereoselectivity of this transformation could alternatively be explained by generation of a *Z*-Co(III) enolate that reacts with the aldehyde via a boat transition state with likely coordination of the directing group nitrogen. Mechanistic studies are planned that should provide better resolution of the reaction pathway and origin of stereoselectivity.

Following our investigation of aldehydes in the three-component reaction, we next explored additions to imines to provide access to branched amine products **12** (Scheme 7). Given the extensive use of nucleophilic additions to *N-tert*-butanesulfinyl imines for the asymmetric synthesis of amines,[12] we evaluated the viability of using *N-tert*-butanesulfinyl imines in this Co(III)-catalyzed three-component coupling process. In very promising preliminary results, asymmetric three-component addition to *N*-sulfinyl trifluoroacetaldimine **11a** gave the

amine product **12a** with high diastereoslectivity and with the absolute configuration rigorously established by X-Ray crystallography.[10] Furthermore, asymmetric three-component addition to sulfinyl imino ester **11b** proceeded to give α-amino ester **12b** also with high diastereoselectivity. To our knowledge these are the first asymmetric reactions employing Co(III)-catalyzed C–H functionalization and also represent the first examples of transition metal catalyzed C–H bond addition to *N-tert*-butanesulfinyl imines.

In summary, we have developed an efficient route for Co(III)-catalyzed three-component coupling of aromatic and alkenyl C(sp^2)–H bonds, enones, and aldehydes or *N-tert*-butanesulfinyl imines. Significantly, this method highlights major reactivity and stereoselectivity differences between Cp*Rh(III) and Cp*Co(III) catalyst systems. Here, we have showcased that Cp*Co(III)-three-component coupling is effective for aryl and alkyl enone coupling partners and displays extremely broad aldehyde scope. The 1-pyrazole, 2-pyridine, and imine directing groups each afforded products in good yield and with high diastereoselectivity. A cleavable alkenyl pyrazole can also be used in the three-component coupling to efficiently provide diastereomerically pure lactones. Additionally, we have provided preliminary data on an asymmetric variant of the reaction through the first C–H bond additions to *N-tert*-butanesulfinyl imines. Broadening enone scope by including β-substitution is currently underway as a means of introducing an additional stereocenter. Asymmetric additions to a range of *N-tert*-butanesulfinyl imines as well as chiral aldehydes are under active investigation, as is the development and evaluation of chiral Co(III) catalysts for this three-component cascade process.

Supplementary Material

Refer to Web version on PubMed Central for supplementary material.

Acknowledgements

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[10]. Synthetic details, characterization, and molecular structures obtained by X-ray structural analysis are described in the Supporting Information. CCDC 1471274 (**4q**) and 1471275 (**12a**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre.

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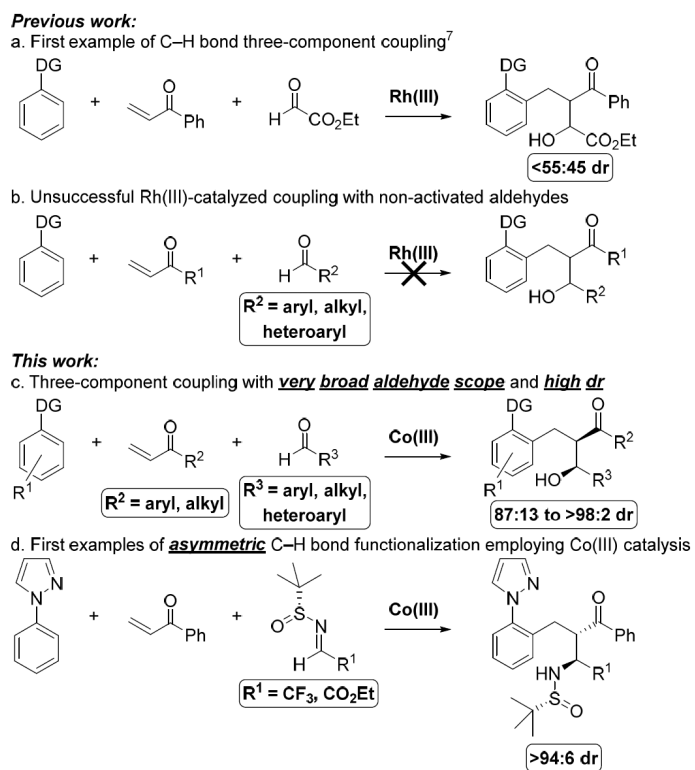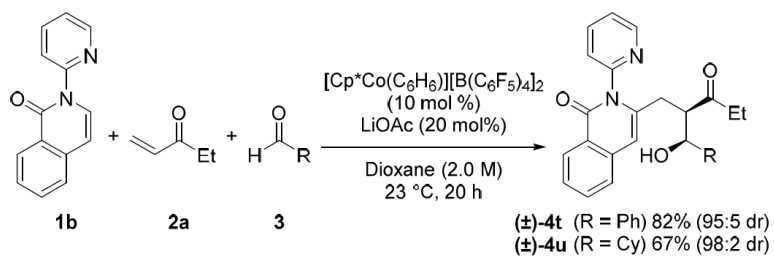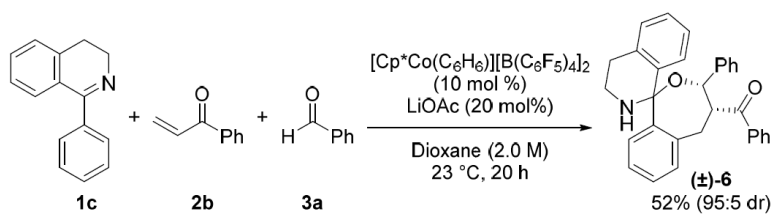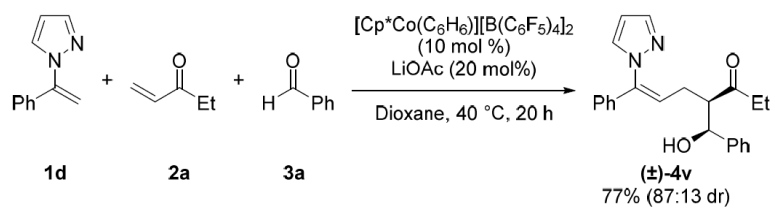
Figure 1.
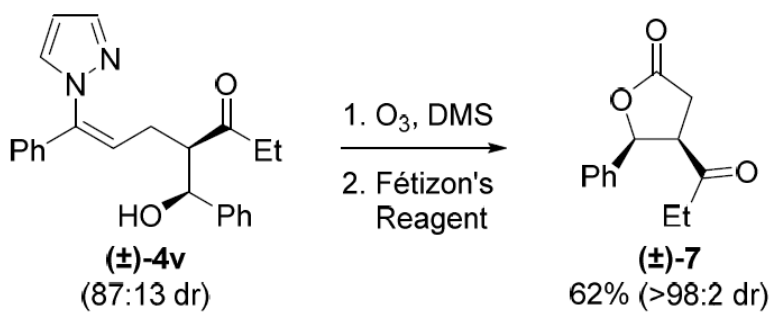C–H bond enabled three-component coupling.

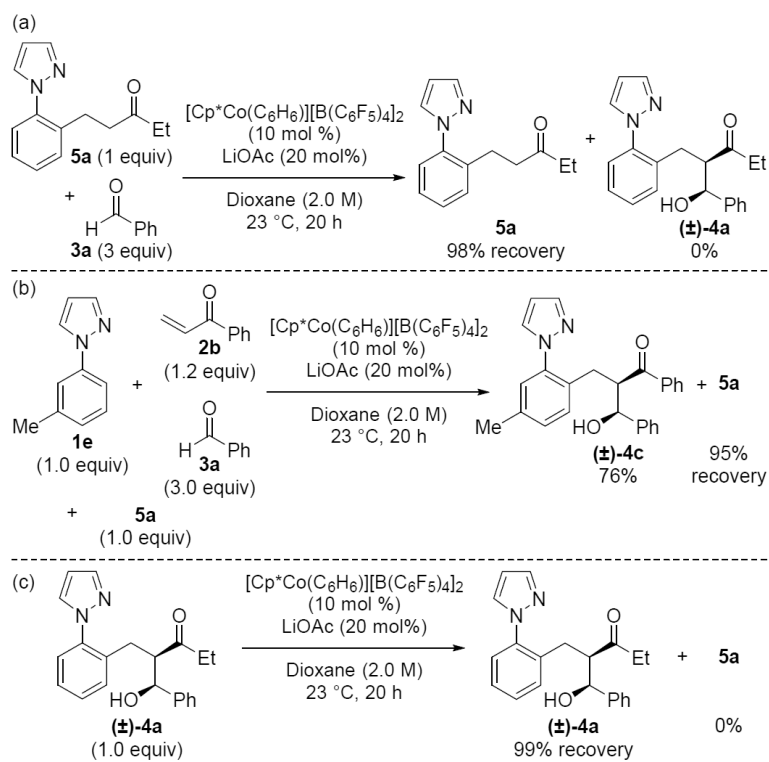**Scheme 1.**
Three-component coupling with pyridylisoquinolinone.

Scheme 2.
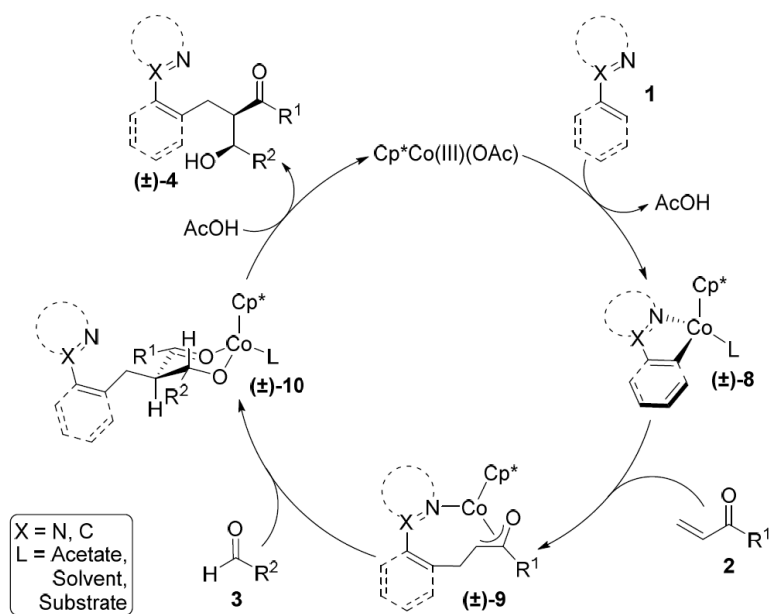Use of ketimine directing group to access a spirohemiaminal ether.
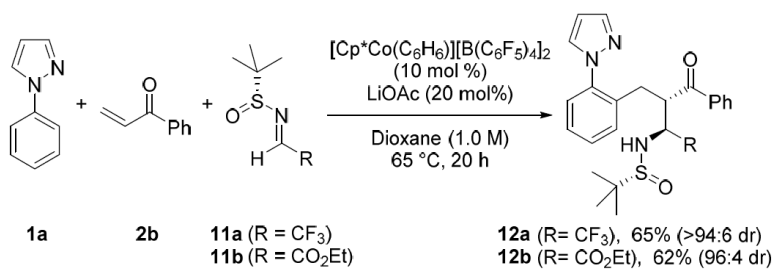
Scheme 3.
Alkenyl C–H bond three-component coupling.

Scheme 4.
Employing a cleavable directing group for further functionalization.

Scheme 5.
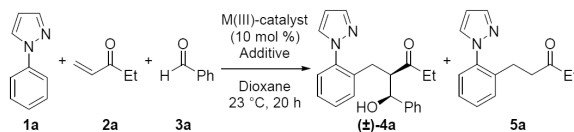Mechanistic Experiments.

Scheme 6.
Proposed mechanism for the Co(III)-catalyzed three-component transformation.

Scheme 7.
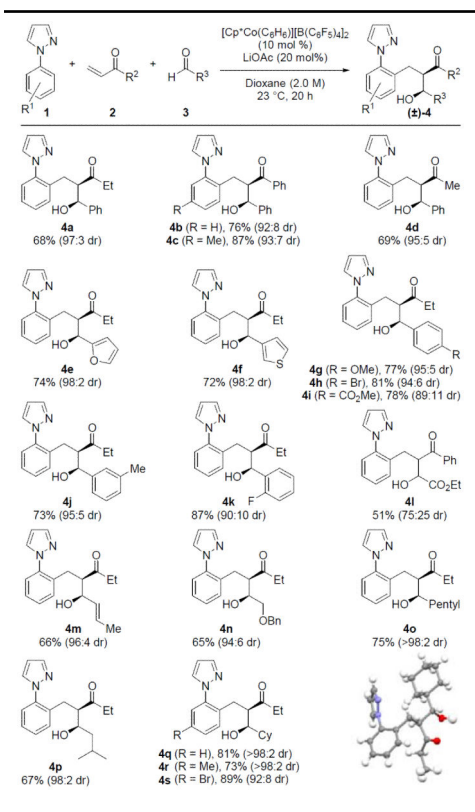Three-component coupling with *N-tert*-butanesulfinyl imines.

Table 1

Optimization for the three-component transformation.[a,b]

Entry	M(III)-catalyst	Additive (mol %)	3a (equiv)	Yield 4a[b]	Yield 5a[b]
1[c]	[Cp*Co(C6H6)][PF6]2	KOAc (20)	5	65% (95:5 dr)	21%
2	[Cp*Co(C6H6)][PF6]2	KOAc (20)	5	<5%	<5%
3	[Cp*Co(C6H6)][B(C6F5)4]2	KOAc (20)	5	61% (96:4 dr)	33%
4	[Cp*Co(C6H6)][B(C6F5)4]2	CsOAc (20)	5	57% (96:4 dr)	43%
5	[Cp*Co(C6H6)][B(C6F5)4]2	LiOAc (20)	5	63% (96:4 dr)	37%
6	Cp*Co(C6H6)][B(C6F5)4]2	LiOAc (20)	3	70% (97:3 dr)	27%
7[d]	[Cp*Co(CO)I2] / AgB(C6F5)4	LiOAc (20)	3	63% (97:3 dr)	32%
8[d,e]	[Cp*RhCl2]2 / AgB(C6F5)4	LiOAc (20)	3	<5%	77%

[a] Conditions: **1a** (1.0 equiv), **2a** (1.2 equiv), **3a** (3–5 equiv) for 20 h in 1,4-dioxane (2.0 M).

[b] Determined by ^{1}H NMR analysis relative to 1,3,5-trimethoxybenzene as an external standard.

[c] Reaction run at 50 °C.

[d] 20 mol % of AgB(C6F5)4.

[e] 5 mol % of [Cp*RhCl2]2 dimer.

Table 2

Scope for the Co(III)-catalyzed three-component transformation.[a,b]