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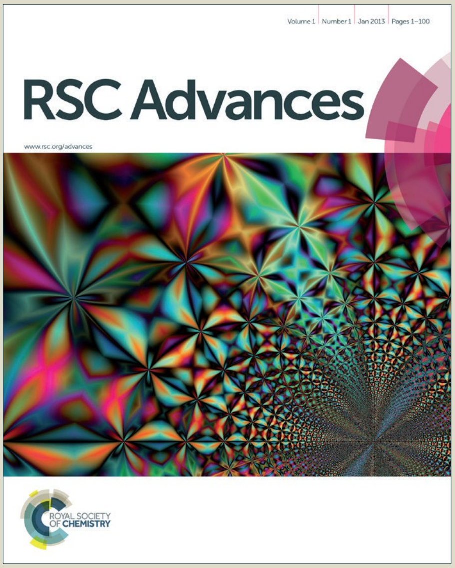

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Cobalt(III)-Catalyzed Synthesis of Pyrroles from Enamides and Alkynes

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An efficient and regioselective cobalt-catalyzed synthesis of multi-substituted pyrroles is reported by the use of readily available enamides and alkynes. The success of the process relies on the employment of a catalytic amount of Cp*Co(CO)I$_2$ together with CuO as the oxidant.

Pyrroles are important five-membered heterocycles and can be found as key structural units in biologically active natural products and diverse therapeutic agents.[1] They are also served as useful skeletons in many functional materials.[2] Consequently, tremendous efforts have been devoted to the approaches of this privileged heterocyclic core,[3] and transition metal-catalyzed reactions based on C-H cleavage take the prominent roles in the recent advances of pyrrole synthesis.[4] Fagnou[5a] and Glorius[5b] first reported the facile access of pyrroles independently from the annulation of alkynes with enamides by rhodium catalysis. In these transformations, the reaction performed without the prefunctionalization of the substrates, and the starting materials are low cost and ready availability. Later, this chemistry is successfully extended to the ruthenium catalysts by Wang,[6a] Ackermann,[6b] and Liu,[6c] and both N-acetyl substituted and N-unsubstituted pyrroles was accessed by slight modification of the reaction conditions. Very recently, Loh's and Guan's research groups, respectively, developed the palladium-catalyzed new method for the synthesis of pyrroles using enamides and alkynes as the starting materials.[7a,7b]

Despite these distinguished advances made in the development of new catalytic methods for the synthesis of pyrroles, there is still a primary demand for alternative approaches that involve cheap and environmentally friendly catalysts. In fact, the development of less expensive and environmentally more benign catalysts has become one of the major goals of organic synthesis. In this respect, cobalt-catalyzed reactions offer attractive prospects in terms of sustainable chemistry, and has been extensively investigated as an alternative catalyst in the field of direct C-H functionalizations.[8] Pioneering progress has been made by Murahashi,[9] Kisch,[10] Nakamura,[11] Kanai,[12] Daugulis,[13] and later advanced by the groups of Glorius,[14] Yoshikai,[15] Ackermann,[16] Ellman,[17] Song,[18] Chang,[19] Ge,[20] and others.[21] We recently reported an efficient and practical cobalt-catalyzed synthesis of pyrrolidinones from aliphatic amides and terminal alkynes.[22] In course of our continuing interest in direct functionalization of C-H bonds of enamides,[23] we report herein a cobalt-catalyzed new method for the construction of C-C and C-N bonds simultaneously, which features synthesis of multi-substituted pyrroles in high yields from readily available enamides[24] and alkynes. The success of the process relies on the employment of a catalytic amount of Cp*Co(CO)I$_2$ together with CuO as the oxidant.

For initial optimization of the reaction conditions and the identification of the best cobalt source, *N*-(1-phenylvinyl)acetamide (**1a**) with 1,2-diphenylethyne (**2a**) were chosen as model substrates (Table 1). It was found that the previous reaction conditions for the cyclization of aliphatic amides and alkynes[22] failed to give the product (Table 1, entry 1). To our delight, treatment of **1a** (1.0 equiv) with 2a (1.5 equiv) in the presence of 10 mol % of Cp*Co(CO)I$_2$ and 2.2 equiv of Cu(OAc)$_2$ in CF$_3$CH$_2$OH at 100 °C for 8 h gave the desired *N*-acetyl substituted pyrrole **3aa** in 30% yield (Table 1, entry 2). The change of oxidant as silver salts showed poor effect on the promotion of the yield (Table 1, entries 3-4). It was found that a large amount of acetophenone, which was produced by the decomposition of enamide, was detected when Cu(OAc)$_2$ and silver salts were used as the oxidants. In

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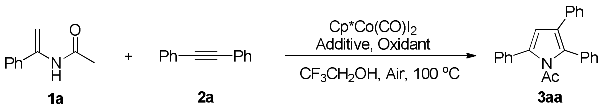
Table 1. Optimization of the Reaction Conditions[a]



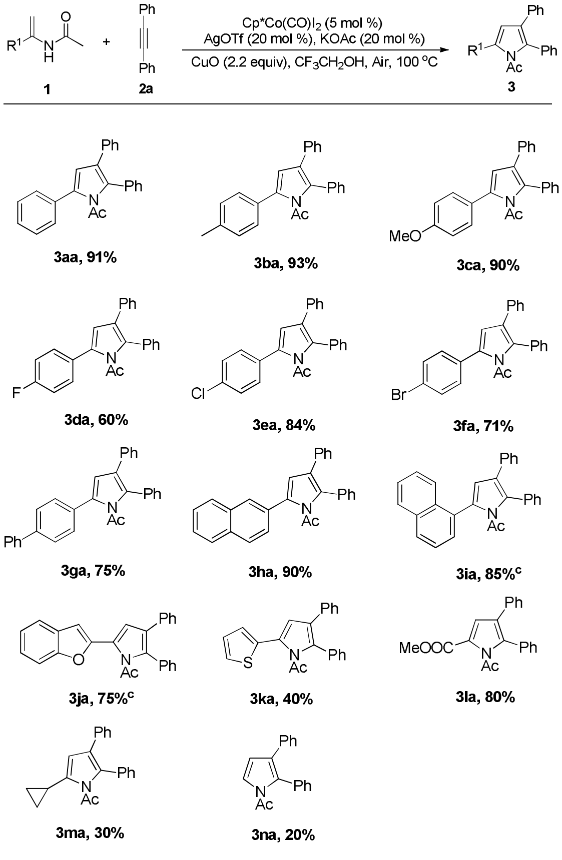
entry	Additive **1**	Additive **2**	Oxidant	Yield[b] (%)
1[c]	Na_2CO_3	TBAI	Ag_2CO_3	NR
2	AgOTf	KOAc	$Cu(OAc)_2$	30
3	AgOTf	KOAc	AgOAc	22
4	AgOTf	KOAc	Ag_2CO_3	16
5	AgOTf	KOAc	CuO	91
6	AgOTs	KOAc	CuO	45
7	$AgBF_4$	KOAc	CuO	NR
8	AgOTFA	KOAc	CuO	30
9	$AgSbF_6$	KOAc	CuO	40
10	AgOTf	NaOAc	CuO	79
11	AgOTf	LiOAc	CuO	64
12	-	KOAc	CuO	Trace
13	AgOTf	-	CuO	Trace

[a] Reactions were carried out by using **1a** (0.1 mmol), **2a** (0.15 mmol), $Cp^*Co(CO)I_2$ (10 mol %), oxidant (2.2 equiv), additive **1** (20 mol %), additive **2** (20 mol %), CF_3CH_2OH (1.0 mL), 100 °C, air, 8 h. [b] Isolated yield. [c] $Co(OAc)_2 \cdot 4H_2O$ (10 mol %), Ag_2CO_3 (4 equiv), TBAI (3 equiv), Na_2CO_3 (3 equiv), pyridine (2 equiv), $PhCF_3$ (1.0 mL), 100 °C. TBAI = Tetrabutylammonium Iodide.

contrast, CuO was compatible with the substrates and showed an obvious improvement of the reactivity to give the pyrrole product **3aa** in 91% yield (Table 1, entry 5). Among the several silver(I) salts tested as iodide abstractor from precatalyst $Cp^*Co(CO)I_2$,[12e] AgOTf showed the highest reactivity, albeit $AgBF_4$ was ineffective to the reaction (Table 1, entries 5-9). Furthermore, the counterion of acetate salts influenced the reaction significantly. For example, LiOAc and NaOAc promoted the reaction, but KOAc showed the best effect to give **3aa** in excellent yield (Table 1, entries 5, 10-11). The reaction in the absence of either KOAc or AgOTf gave a trace **3aa** product, indicating that both AgOTf and KOAc were essential to generate the active species of cationic catalytic Co(III) in situ (Table 1, entry 12-13).[12e] After screening the solvents, CF_3CH_2OH gave the best yields (see the Supporting Information for details).

We next examined the substrate scope of various enamides under the optimized reaction conditions as shown in Scheme 1. In general, the transformation displayed high functional group tolerance and proved to be a reliable methodology for preparation of multisubstituted pyrroles. Both the electron-rich and electron-deficient enamides showed good reactivity to give the corresponding pyrroles in good yields (**3aa-3ga**). Naphthylenamides and 2-benzofuranyl enamide participated in the reaction smoothly to give the desired pyrroles in high yields (**3ha-3ja**). Thienylenamide underwent the reaction to give the thienylsubstituted pyrrole **3ka**. Electron-deficient enamide showed good reactivity to give the corresponding pyrrole **3la** in good yield. Alkyl substituted enamide afforded relatively lower yield (**3ma**) due to the heavy decomposition. A variety of functional groups, including alkyl, methoxyl, phenyl, fluoro, chloro, bromo, and ester, were well tolerated in the reaction, which may allow the further construction of complex molecules. The unstable *N*-vinylacetamide **3n** carried out the reaction to provide 2,3-substituted pyrrole **3na** but in low yield.

Scheme 1. Scope of Enamides in the Synthesis of Substituted Pyrroles[a,b]



[a] Reactions were carried out by enamides **1** (0.1 mmol), diphenyl-acetylene **2a** (0.15 mmol), $Cp^*Co(CO)I_2$ (10 mol %), AgOTf (20 mol %), KOAc (20 mol %), CuO (2.2 equiv) and CF_3CH_2OH (1.0 mL) at 100 °C in air for 8 h. [b] Isolated yield. [c] Air was replaced by N_2.

The reactivity of a range of functionally diverse alkynes was investigated as shown in Scheme 2. Symmetrical diaryl internal

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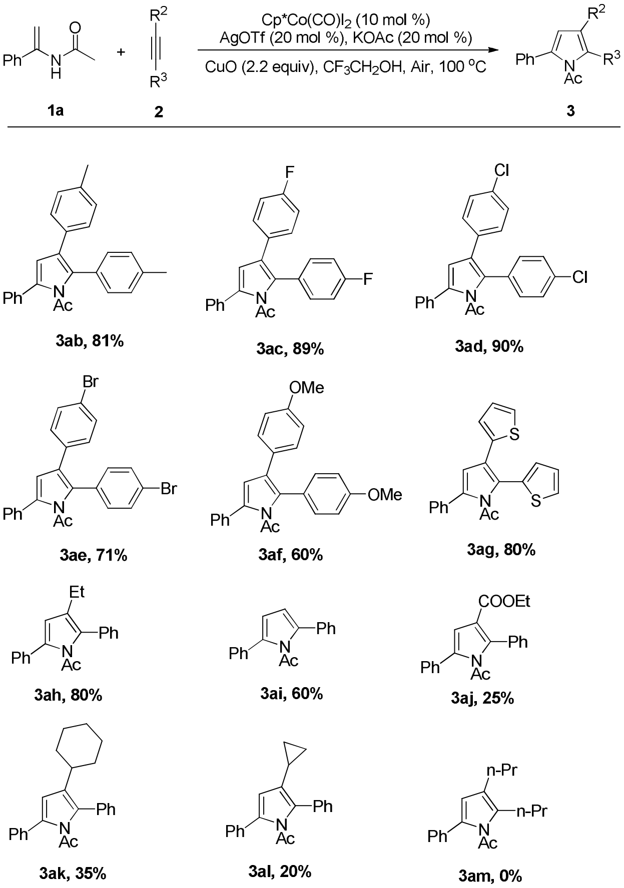
alkynes with either electron-donating or electron-withdrawing groups participated in the reaction smoothly to afford pyrrole **3ab-3af** in moderate to excellent yields. Notably, 1,2-di(thiophen-2-yl)ethyne was tolerated to give the thiophene containinge pyrrole **3ag** in good yield. Comparing with the palladium catalytic system, [7a, 7b] this cobalt catalytic system presented excellent regioselectivity. For example, when aryl alkyl-disubstituted alkynes were used as the substrates, the C-C bond formation always took place at the alkyl-substituted carbon of alkynes and the C-N bond generated between the nitrogen and aryl-substituted carbon of alkynes. Other regioisomers were not observed in these reactions. The transformation was sensitive to the hindrance of the alkynes. The steric alkyl aryl alkynes showed the less reactivity to give the corresponding pyrroles in lower yields (**3ak, 3al**). Interestingly, trimethyl(phenylethynyl)silane participated in the reaction but finally gave the TMS-cleavage pyrrole **3ai** in 60% yield. Less reactivity of ethyl 3-phenylpropiolate was observed and the pyrrole **3aj** was obtained in low yield. The aliphatic internal alkyne was inactive.

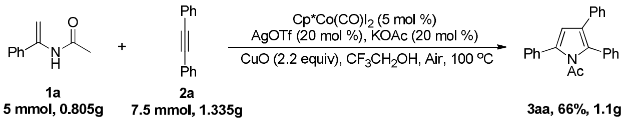
Scheme 2. Scope of Alkyne in the Synthesis of Multisubstitueted Pyrroles [a,b]



[a] Reactions were carried out by using **1a** (0.1 mmol), **2** (0.15 mmol), $Cp^*Co(CO)I_2$ (10 mol %), AgOTf (20 mol %), KOAc (20 mol %), CuO (2.2 equiv), CF_3CH_2OH (1.0 mL), 100 °C, air, 8 h. [b] Isolated yield.

This procedure could be scaled up with equal ease to a 5 mmol scale and afforded the product in 66% yield. (Scheme 3)

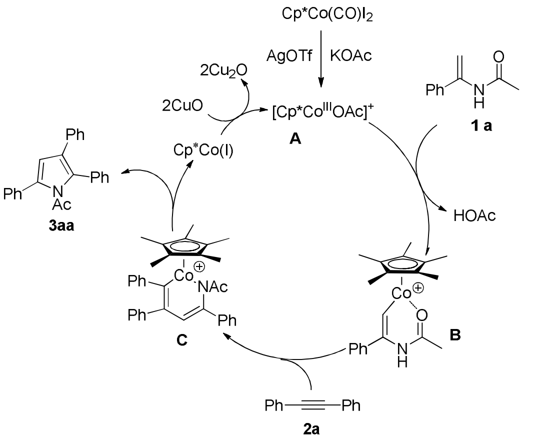
Scheme 3. Gram-Scale Reaction of 1a[a,b]



[a] Reactions were carried out by using **1a** (5 mmol), **2a** (7.5 mmol), $Cp^*Co(CO)I_2$ (10 mol %), AgOTf (20 mol %), KOAc (20 mol %), CuO (2.2 equiv), CF_3CH_2OH (5.0 mL), 100 °C, air, 8 h. [b] Isolated yield.

Based on the observed results and previous reports, [12e] a plausible catalytic cycle is proposed as shown in Scheme 4. Iodide abstraction from precatalyst $Cp^*Co(CO)I_2$ by AgOTf and ligand exchange generates the catalyst **A**, which undergoes metalation at the vinyl C-H with the assistance of amide group to give the cobaltacyclic complex **B**. The subsequent migratory insertion of alkynes into cobaltacycle **B** forms the intermediate **C**, which undergoes the reductive elimination to give the pyrrole product and liberate Co(I) complex. The oxidation of Co(I) by CuO regenerates Co(III) catalyst to fulfill the catalytic cycle.

Scheme 4. Proposed Mechanism



In conclusion, we have developed an efficient cobalt-catalyzed new method for the synthesis of multi-substituted pyrroles from readily available enamides and alkynes. In this transformation, carbon-carbon and carbon-nitrogen bonds are simultaneously constructed and a range of synthetically useful alkynes were tolerated to give the diverse pyrroles in moderate to high yields. Comparing with the palladium catalytic system, this cobalt catalytic system presented excellent regioselectivity. Further studies to explore cobalt-catalyzed C-H functionalization to construct the heterocycles are ongoing in our laboratory.

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