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To be cited as: Chem. Eur. J. 10.1002/chem.201603499

Link to VoR: http://dx.doi.org/10.1002/chem.201603499

Oxidative Annulation of Arenecarboxylic and Acrylic Acids with Alkynes Under Ambient Conditions Catalyzed by an Electron-Deficient Rhodium(III) Complex

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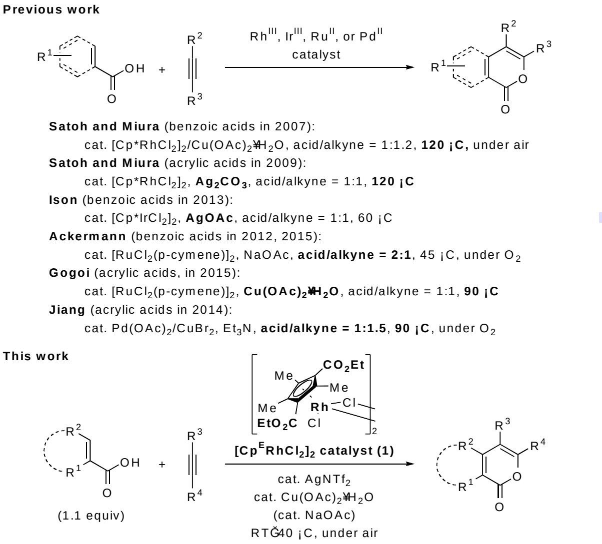
Abstract: It has been established that an electron-deficient Cp^E rhodium(III) complex catalyzes the oxidative [4+2] annulation of substituted arenecarboxylic and acrylic acids with alkynes under ambient conditions (at RT–40 °C, under air) without using excess amounts of substrates to produce the corresponding substituted isocoumarins and α-pyrones in high yields. Minor modification of reaction conditions depending on the coordination ability of alkynes realized the high efficiency.

Transition-metal catalyzed oxidative annulations with alkynes via cleavage of sp^2 C–H bonds are powerful methods for the construction of carbocycles and heterocycles.[1] For example, the oxidative [4+2] annulation of benzoic and acrylic acids with alkynes produces isocoumarins and α-pyrones,[2] respectively, in a highly step- and atom-economical manner (Scheme 1, top).[3–9] In 2007, Satoh and Miura developed the first example of the oxidative [4+2] annulation of benzoic acids with alkynes using a Cp* rhodium(III) complex as a catalyst, and $Cu(OAc)_2$•H_2O and air as oxidants.[3] They subsequently reported the annulation of acrylic acids instead of benzoic acids by using Ag_2CO_3 as an oxidant.[4] Not only rhodium(III) complex[5] but also iridium(III),[6] ruthenium(II),[7] and palladium(II)[8] complexes can catalyze this annulation, however, these reactions required high temperature (≥90 °C), stoichiometric metal oxidants, and/or excess amounts of substrates. Therefore, a new catalyst system, which shows the satisfyingly wide substrate scope under ambient conditions without using stoichiometric metal oxidants and excess amounts of substrates, remains a challenge. On the other hand, we reported that electron-deficient Cp^E rhodium(III) complex **1**[10a] showed high catalytic activity for oxidative coupling reactions via cleavage of sp^2 C–H bonds on electron-rich aromatic rings under ambient conditions (at RT, under air).[10,11] The observed high catalytic activity might be induced by the strong π-metal interaction between the electron-rich aromatic rings and the highly electrophilic rhodium in the transition state of the C–H bond cleavage. It was anticipated that an electron-rich benzoate salt would show high reactivity in the Cp^E rhodium(III)-catalyzed oxidative annulation with alkynes as a result of the strong π-metal interaction. In this paper, we succeeded this transformation under ambient conditions with the wide substrate scope by minor modification of reaction conditions depending on the coordination ability of alkynes (Scheme 1, bottom).



Satoh and Miura (benzoic acids in 2007):
cat. $[Cp^*RhCl_2]_2/Cu(OAc)_2$¥H_2O, acid/alkyne = 1:1.2, **120 ¡C,** under air
Satoh and Miura (acrylic acids in 2009):
cat. $[Cp^*RhCl_2]_2$, **Ag_2CO_3**, acid/alkyne = 1:1, **120 ¡C**
Ison (benzoic acids in 2013):
cat. $[Cp^*IrCl_2]_2$, **AgOAc**, acid/alkyne = 1:1, 60 ¡C
Ackermann (benzoic acids in 2012, 2015):
cat. $[RuCl_2(p\text{-cymene})]_2$, NaOAc, **acid/alkyne = 2:1**, 45 ¡C, under O_2
Gogoi (acrylic acids, in 2015):
cat. $[RuCl_2(p\text{-cymene})]_2$, **$Cu(OAc)_2$¥$H_2O$**, acid/alkyne = 1:1, **90 ¡C**
Jiang (acrylic acids in 2014):
cat. $Pd(OAc)_2/CuBr_2$, Et_3N, **acid/alkyne = 1:1.5**, **90 ¡C**, under O_2

Scheme 1. Transition-metal-catalyzed oxidative [4+2] annulations of benzoic and acrylic acids with alkynes.

First we screened the reaction conditions using 1.1 equiv of benzoic acid (**2a**) and 1.0 equiv of three types of alkynes, diphenylacetylene (**3a**), 1-phenyl-1-propyne (**3b**), and 6-dodecyne (**3c**). As a result, we established three reaction conditions A–C depending on the coordination ability of alkynes to the cationic rhodium (Table 1). When using weakly coordinating **3a**, the desired annulation proceeded in the presence of **1** (2.5 mol %), $AgNTf_2$ (12.5 mol %), and $Cu(OAc)_2$•H_2O (5 mol %) in $(CH_2Cl)_2$ at RT under air to give isocoumarin **4aa** in excellent yield (conditions A, entry 1). In sharp contrast, the use of $[Cp^*RhCl_2]_2$ instead of **1** afforded only a trace amount of **4aa** (entry 2). However, the reaction of moderately coordinating **3b** afforded **4ab** in low yield (entry 3) and that of strongly coordinating **3c** afforded only a trace amount of **4ac** (entry 4). On the other hand, the conditions B

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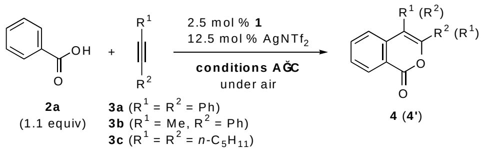
using moderately coordinating acetone as a solvent and an increasing amount of $Cu(OAc)_2{\bullet}H_2O$ (20 mol %) at 40 °C was found to be effective for **3b** (entry 6), while a slight decrease and modest increase of the product yield were observed for **3a** and **3c**, respectively (entries 5 and 7). Pleasingly, the addition of NaOAc (20 mol %) to conditions A significantly increased the yield of **4ac** (conditions C, entry 10), while this protocol was not effective for **3a** and **3b** (entries 8 and 9).

Table 1. Optimized conditions depending on coordination ability of alkynes **3**.[a]



conditions A: 5 mol % $Cu(OAc)_2$¥H_2O, $(CH_2Cl)_2$, RT
conditions B: 20 mol % $Cu(OAc)_2$¥H_2O, acetone, 40 ¡C
conditions C: 5 mol % $Cu(OAc)_2$¥H_2O, 20 mol % NaOAc, $(CH_2Cl)_2$, RT

Entry	Conditions	**3**	**4**	Yield [%][b]	r.s.[b]
1	A (24 h)	**3a**	**4aa**	97	–
2[c]	A (24 h)	**3a**	**4aa**	2	–
3	A (24 h)	**3b**	**4ab (4ab')**	36	89:11
4	A (24 h)	**3c**	**4ac**	3	–
5	B (72 h)	**3a**	**4aa**	95	–
6	B (72 h)	**3b**	**4ab (4ab')**	73	91:9
7	B (72 h)	**3c**	**4ac**	38	–
8	C (72 h)	**3a**	**4aa**	11	–
9	C (72 h)	**3b**	**4ab (4ab')**	40	88:12
10	C (72 h)	**3c**	**4ac**	87	–

[a] Conditions A: **1** (0.0050 mmol), $AgNTf_2$ (0.025 mmol), $Cu(OAc)_2{\bullet}H_2O$ (0.010 mmol), **2** (0.22 mmol), **3** (0.20 mmol), and $(CH_2Cl)_2$ (1.0 mL) were used. Conditions B: $Cu(OAc)_2{\bullet}H_2O$ (0.040 mmol) was used. Conditions C: NaOAc (0.040 mmol) was used. [b] Determined by ^1H NMR using 1,1,2,2-tetrachloroethane as an internal standard. [c] $[Cp^*RhCl_2]_2$ was used instead of **1**.

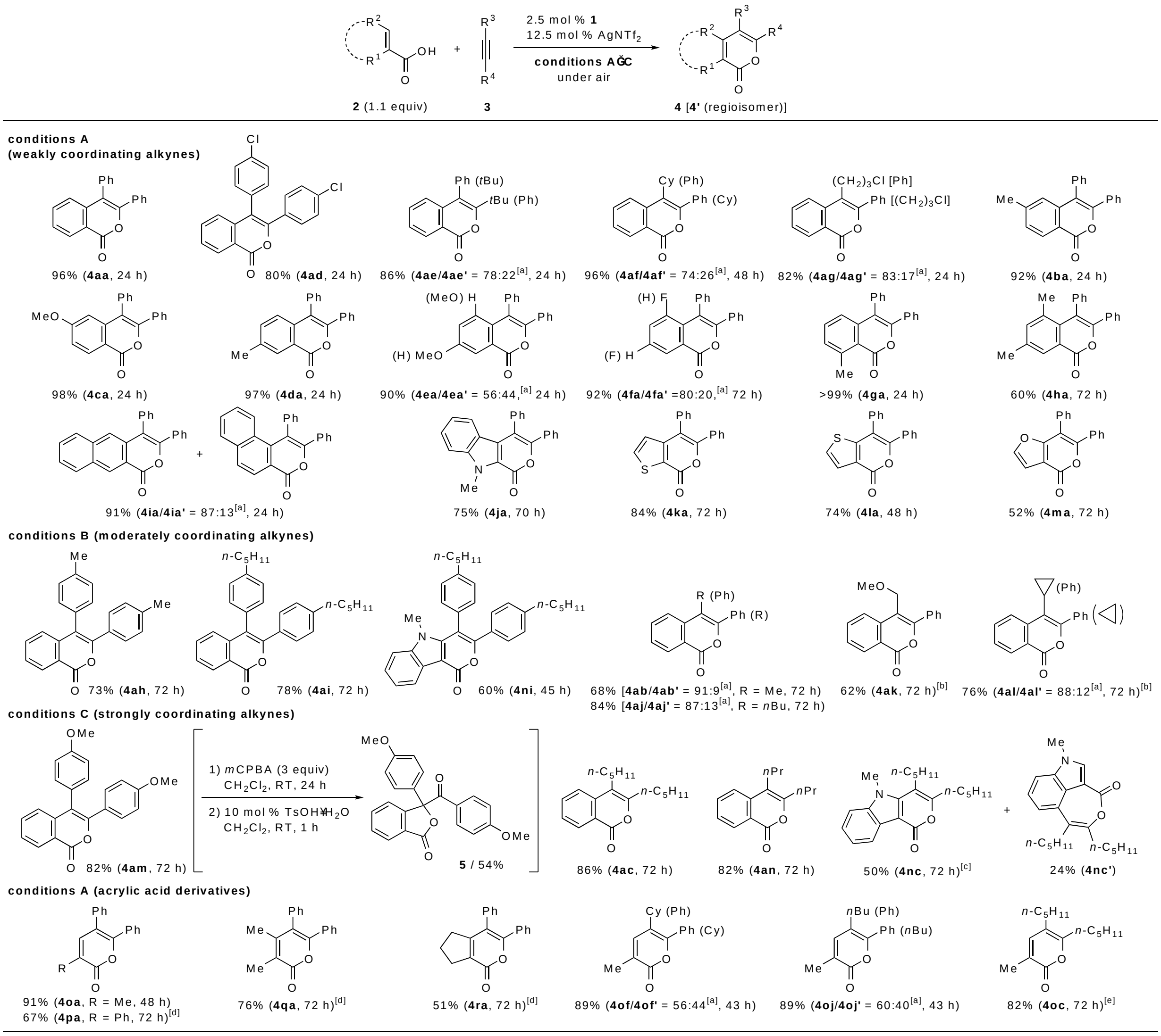
Wide substrate scope was realized by employing the above protocols (Scheme 2). With respect to weakly coordinating alkynes under the conditions A, not only **3a** but also electron-deficient diarylacetylene **3d** reacted with **2a** to afford **4ad** in high yield. The reactions of **2a** with unsymmetrical alkynes bearing *tert*-butyl (**3e**), cyclohexyl (**3f**), and chloropropyl (**3g**) groups afforded **4ae**–**ag** in high yields. In these reactions, the opposite regioselectivities were observed between **3e** and **3f**,**g**. With respect to benzoic acids, *para*, *meta*-, and *ortho*-substituted ones **2b**–**g** could equally participate in this reaction regardless of their electron densities, although regioselectivities were poor (**4ea**/**4ea'**) to moderate (**4fa**/**4fa'**). Importantly, sterically hindered 3,5-dimethylbenzoic acid (**2h**) also reacted with **3a** to give **4ha** in good yield. In the reaction of 2-naphthalenecarboxylic acid (**2i**), the C–H bond at the 3-position was cleaved in preference to the 1-position to give **4ia** as a major product. Not only benzoic acids but also indole-, thiophene-, and furan-carboxylic acids **2j**–**m** reacted with **3a** under the conditions A to give **4ja**–**ma** in good to high yields. With respect to moderately coordinating alkynes under the conditions B, electron-rich diarylacetylenes **3h** and **3i** reacted with **2a** and **2n** to give **4ah**, **4ai**, and **4ni** in good yields. With respect to unsymmetrical alkynes, not only 1-phenyl-1-propyne (**3b**) but also hexyl (**3j**), methoxymethyl (**3k**), and cyclopropyl (**3l**) substituted phenylacetylenes reacted with **2a** to give **4aj**–**l** in good yields with good to perfect regioselectivities, although high catalyst loadings were required for **3k** and **3l**. Highly electron-rich diarylacetylene **3m**, and dialkyl acetylenes **3c** and **3n** reacted with **2a** under the conditions C to give isocoumarins **4am**, **4ac**, and **4an** in high yields. Importantly, thus obtained electron-rich isocoumarin **4am** could be converted to γ-lactone **5** by oxidative rearrangement.[12] Interestingly, the reaction of **2n** with **3c** afforded oxepin **4nc'** as well as α-pyrone **4nc**.

Notably, non-fused α-pyrones could also be prepared under the conditions A by the oxidative annulation of acrylic acid derivatives with alkynes regardless of their coordination abilities (Scheme 2). Various 2-substituted and 2,3-disubstituted acrylic acids **2o**–**r** reacted with **3a** to give α-pyrones **4oa**–**ra** in good to high yields, although high catalyst loadings were required for **2p**–**r**. Unsymmetrical alkynes **3f** and **3j** could also react with **2o** to give **4of**/**4of'** and **4oj**/**4oj'** in high yields, while the regioselectivities were low. Furthermore, highly coordinating dialkylacetylene **3c** reacted with **2o** by using 20 mol % of $Cu(OAc)_2$ to give **4oc** in high yield presumably due to possible bidentate coordination of **2o** to rhodium.

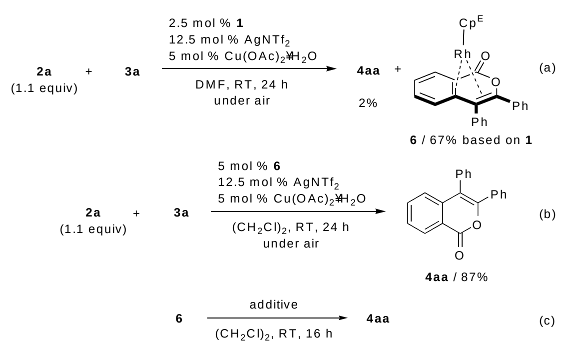
In the course of the reaction optimization, we found that the reaction of **2a** with **3a** in DMF resulted in low substrate conversion and afforded isocoumarin-coordinating rhodium(I) complex **6** (Scheme 3a). The reaction of **2a** with **3a** using a catalytic amount of **6** instead of **1** afforded **4aa** in high yield (Scheme 3b). These results indicate that **6** is the intermediate of the present oxidative [4+2] annulation and DMF deters oxidative decomplexation of **6**. Thus, the oxidative decomplexation of **6** was examined by using various oxidants and additives (Scheme 3c). Surprisingly no reaction was observed using the copper(II) oxidant under argon (entry 1). Instead, the reaction with $NaNTf_2$ under air afforded **4aa** in 25% yield (entry 2). The use of both the copper(II) oxidant and $NaNTf_2$ under argon afforded **4aa** in the highest yield (entry 3). These facts suggest that coordination of NTf_2^- to rhodium may accelerate the oxidative decomplexation. Contrary to the oxidative decomplexation of an isocoumarin-ruthenium(0) complex reported by Ackermann,[7d] the use of acetic acid under air was ineffective (entry 4).

In order to gain mechanistic insights into the C–H bond cleavage step using **1**, we conducted the deuterium-labeling experiments (Scheme 4). The reaction of benzoic acid-d_5 (**2a**-[D_5]) with **3a** under the conditions A furnished **4aa**-[D_5] (Scheme 4a). As almost no loss of deuterium from **4aa**-[D_5] was observed, the metalation of the benzoic acid was found to be irreversible. The intermolecular competition experiments between **2a** and **2a**-[D_5] in the presence of **3a** (conditions A) or **3c** (conditions C) revealed large DKIE values of 8.1 and 6.7, respectively (Scheme 4b).[13] These facts suggest that the rate-determining step involves the C-H bond cleavage.

A plausible mechanism for the oxidative [4+2] annulation of **2** with **3** is shown in Scheme 5. First, a cationic rhodium species **A** is likely generated by the reaction of **1**, $AgNTf_2$, and $Cu(OAc)_2$. Next, coordination of **2** through deprotonation forms rhodium(III) carboxylate **B**. Cyclometalation through cleavage of the *ortho*-C–H bond furnishes rhodacycle **C**. Coordination and subsequent insertion of **3** to **C** give rhodacycle **D**. When using benzoic acid (**2a**), bulky substituents (R^1) of alkynes reside in α-position to rhodium in order to avoid steric repulsion to the aromatic hydrogen in **D'**. On the contrary, when using methacrylic acid

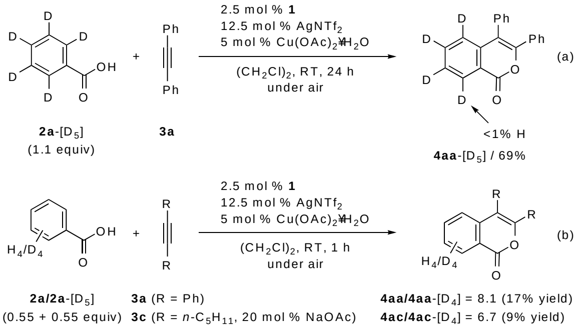


Scheme 2. Substrate scope. Conditions A–C: see Table 1. [a] Ratios of regioisomers. [b] **1** (5 mol %), $AgNTf_2$ (25 mol %), and $Cu(OAc)_2$•H_2O (40 mol %) were used. [c] At 40 °C. [d] **1** (5 mol %), $AgNTf_2$ (25 mol %), and $Cu(OAc)_2$•H_2O (10 mol %) were used. [e] $Cu(OAc)_2$•H_2O (20 mol %) was used.



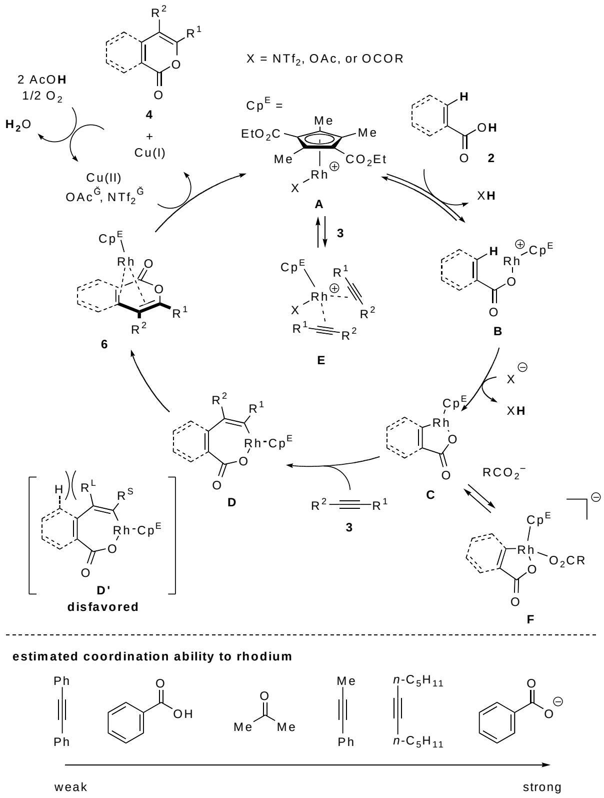
Entry	Additive	Yield [%]
1	$Cu(OAc)_2$¥H_2O (2 equiv), under Ar	<5
2	$NaNTf_2$ (2 equiv), under air	25
3	$Cu(OAc)_2$¥H_2O (2 equiv), $NaNTf_2$ (2 equiv), under Ar	40
4	AcOH (50 equiv), under air	9

Scheme 3. (a) Isolation, (b) catalytic activity, and (c) oxidative decomplexation of isocoumarin-coordinating Cp^ERh^I complex **6**.



Scheme 4. Deuterium-labeling experiments.

(**2o**), steric hindrance in **D’** may be small, therefore, the reactions of **2o** with unsymmetrical alkynes resulted in low regioselectivity. From **D**, C–O reductive elimination affords rhodium(I) complex **6**. This rhodium(I) complex **6** is oxidized by copper(II) oxidant in the presence of the counteranion NTf_2^- to regenerate the active rhodium(III) complex **A** and the resulting copper(I) is reoxidized by molecular oxygen in air.



Scheme 5. Plausible mechanism for the formation of **4**.

Beneficial effects of minor modification of reaction conditions can be explained as follows. Coordination of two molecules of strongly coordinating alkynes to **A** giving inactive species **E** results in low conversion of **2** under the conditions A. When adding NaOAc (conditions C), strong coordination of in situ generated carboxylate anion to **A** accelerates the reactions with the strongly coordinating alkynes, although this carboxylate anion may coordinate to **C** giving inactive species **F**, which inhibits the coordination of weakly coordinating alkynes. When using moderately coordinating alkynes, the use of moderately coordinating acetone as a solvent at slightly elevated temperature (conditions B) facilitates ligand exchange through equilibration between intermediates **A** and **E**, and **C** and **F** via acetone-coordinating rhodium species.[14]

In conclusion, we have established that an electron-deficient Cp^{E} rhodium(III) complex catalyzes the oxidative [4+2] annulation of substituted arenecarboxylic and acrylic acids with alkynes under ambient conditions (at RT–40 °C, under air) without using excess amounts of substrates to produce the corresponding substituted isocoumarins and α-pyrones in high yields. Minor modification of reaction conditions depending on the coordination ability of alkynes realized the high efficiency.

Acknowledgements

This work was supported partly by ACT-C from JST (Japan), Grants-in-Aid for Scientific Research (Nos. 26102004 and 25105714) from MEXT (Japan), and a Grant-in-Aid for Research Activity Start-up (No. 15H06201) from JSPS (Japan). We are grateful to Umicore for generous support in supplying the rhodium complex.

Keywords: Alkynes • Annulation • Isocoumarins • α-Pyrones • Rhodium

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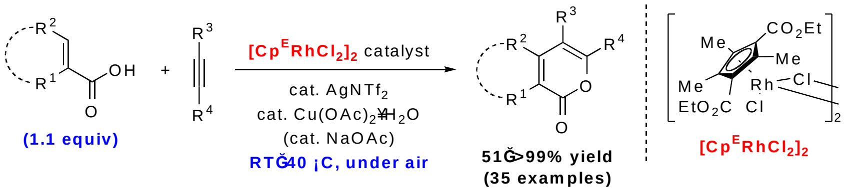

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Page No. – Page No.

Oxidative Annulation of Arenecarboxylic and Acrylic Acids with Alkynes Under Ambient Conditions Catalyzed by an Electron-Deficient Rhodium(III) Complex