Cross-Coupling

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Rhodium(I)-Catalyzed Regiospecific Dimerization of Aromatic Acids: Two Direct C−H Bond Activations in Water**

*Hang Gong, Huiying Zeng, Feng Zhou, and Chao-Jun Li**

***Abstract:** 2,2′-Diaryl acids are key building blocks for some of the most important and high-performance polymers such as polyesters and polyamides (imides), as well as structural motifs of MOFs (metal–organic frameworks) and biological compounds. In this study, a direct, regiospecific and practical dimerization of simple aromatic acids to generate 2,2′-diaryl acids has been discovered, which proceeds through two rhodium-catalyzed C−H activations in water. This reaction can be easily scaled up to gram level by using only 0.4–0.6 mol% of the rhodium catalyst. As a proof-of-concept, the natural product ellagic acid was synthesized in two steps by this method.*

2,2′-Diaryl acids are the key building blocks for some of the most important and high-performance polymers (high thermal stability, low coefficient of thermal expansion, and excellent mechanical/electrical properties; Figure 1a).[1] Moreover, these motifs also play an important role in the construction of MOFs (Figure 1b)[2] and are widely present in natural products (Figure 1c),[3] pharmaceutical agents (Figure 1d),[4] and C_2-symmetric ligands (Figure 1e).[5]

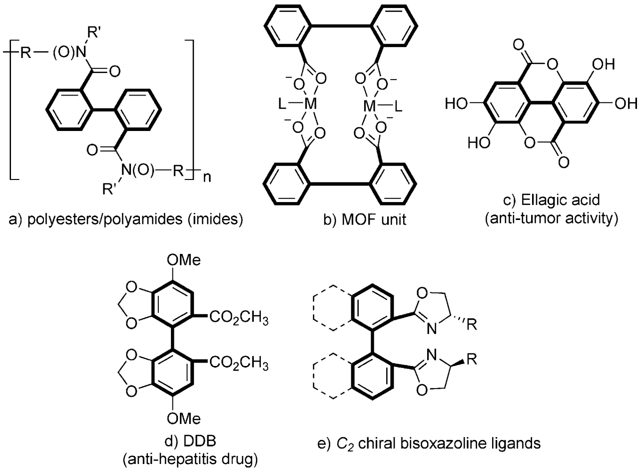
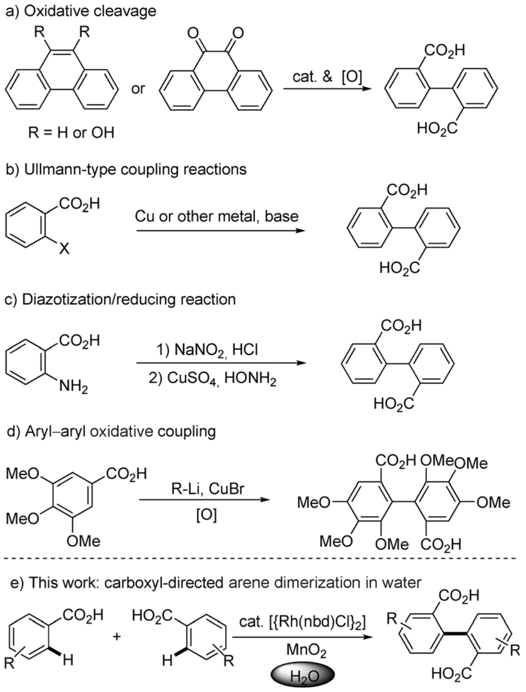

a) polyesters/polyamides (imides)
b) MOF unit
c) Ellagic acid (anti-tumor activity)
d) DDB (anti-hepatitis drug)
e) C_2 chiral bisoxazoline ligands

Figure 1. Representative compounds with a 2,2′-diaryl acids framework.


a) Oxidative cleavage
b) Ullmann-type coupling reactions
c) Diazotization/reducing reaction
d) Aryl–aryl oxidative coupling
e) This work: carboxyl-directed arene dimerization in water

Scheme 1. Strategies for the construction of diaryl acid motif. nbd = norbornadiene.

Despite their importance, the synthetic methods for making 2,2′-diaryl acids are unsatisfactory. One of the most popular methods is the oxidative cleavage of phenanthrene,[6] phenanthrene-9,10-dione,[7] or phenanthrene-9,10-diol[8] (Scheme 1a). Various catalysts such as Pd/In, Ru, Os, or N-heterocyclic carbenes (NHC), along with a variety of oxidants including H_2O_2, KO_2, NaClO, $KMnO_4$, and air, have been reported for this method. However, the requirement of a pre-synthesized biaryl motif limits its practicability and generality. Another well-known method is the Ullmann-type coupling reaction (Scheme 1b),[9] however, the required halogen prefunctionalization of the aromatic acids as well as the intolerance of free acids under the classical Ullmann reaction conditions limit its applications to only a few diaryl acids. A third common strategy towards diaryl acids is the diazotization/coupling sequence, which requires multistep synthetic operations to generate the amino aromatic acids, and there are also the potential hazards of handling diazo compounds

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[*] Dr. H. Gong, Dr. H. Zeng, Dr. F. Zhou, Prof. Dr. C.-J. Li
Department of Chemistry and FQRNT Centre for Green Chemistry and Catalysis, McGill University
801 Sherbrooke St. W., Montreal, Quebec H3A 0B8 (Canada)
E-mail: cj.li@mcgill.ca

Dr. H. Gong
The Key Laboratory of Environmentally Friendly Chemistry and Application of the Ministry of Education, College of Chemistry, Xiangtan University, Xiangtan 411105 (China)

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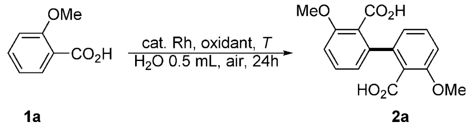
(Scheme 1 c).[10] In addition, oxidative coupling of stoichiometric amounts of lithium reagents (Scheme 1 d)[11] and the Suzuki cross-coupling reaction[12] have also been used to synthesize this motif. However, the required stoichiometric organolithium reagent as well as the necessary pre-synthesis of the haloaryl acids and carboxyl aryl boronic acids are shortcomings of these methods. In light of the principles of green and sustainable chemistry, a highly desirable alternative for the construction of diaryl acid moieties would involve two C−H activations. Despite of the obvious advantages, for example, shorter reaction steps and reduced waste, multiple obstacles such as unfavorable thermodynamics, the generally low reactivity of C−H bonds, and selectivity issues make this strategy particularly challenging.[13,14] To the best of our knowledge, no known general method for dimerization of simple aryl acids has been reported. As part of our continuous interest in C−H functionalizations[15] and aqueous reactions,[16] herein we describe an efficient, regiospecific, and general oxidative dimerization of simple aryl acids to generate a wide range of diaryl acids for the first time (Scheme 1 e).[17]

Our investigation commenced with the reaction of *o*-anisic acid using [RhH(Ph_3P)$_4$] (10 mol %) as the catalyst, MnO_2 as the oxidant, and water as the solvent at 150 °C under an atmosphere of air (Table 1). Gratifyingly, the desired homocoupling product 3,3′-dimethyl-2,2′-dibenzoic acid was formed in 56 % yield (entry 1). Encouraged by this result, various rhodium catalysts were examined (entries 2–4) and it was found that [{Rh(nbd)Cl}$_2$] (5 mol %) was the most effective, affording 85 % yield of the isolated diaryl acid (entry 4). Next, various oxidants were evaluated. Only a trace amount of the product was detected in most cases (entries 5–7) except for silver acetate, which gave a moderate yield (41 %; entry 7). Either decreasing the MnO_2 loading or lowering the reaction temperature resulted in lower yields (entries 8–10). When the amount of catalyst was further decreased to 2.5 mol %, a slightly lower yield (73 %) was obtained (entry 11). Finally, control experiments revealed that both the rhodium catalyst and MnO_2 are essential to this reaction (entries 12 and 13). By using the optimized reaction conditions [5 mol % [{Rh(nbd)Cl}$_2$], 3 equiv MnO_2, 150 °C under air in water for 24 h], the generality of this method was then explored.

Table 1: Selected optimization results.[a]



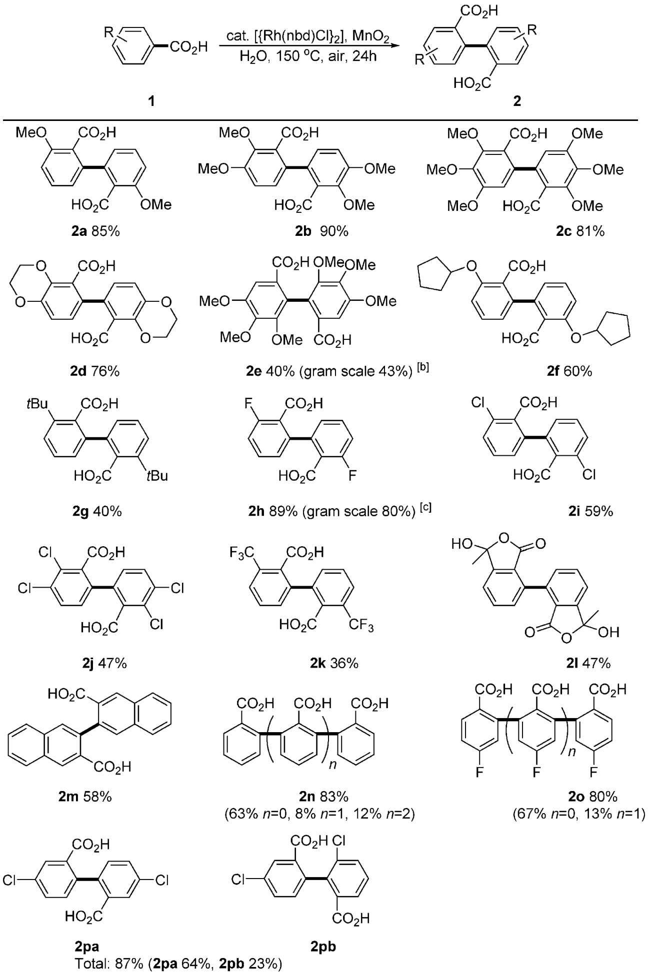
1a **2a**

Entry	Cat. (mol %)	Oxidant	*T* [°C]	Yield [%]
1	[RhH(Ph_3P)$_4$] (10)	MnO_2	150	56
2	[Rh_2(AcO)$_4$] (5)	MnO_2	150	20
3	[{Cp*$RhCl_2$}$_2$] (5)	MnO_2	150	60
4	[{Rh(nbd)Cl}$_2$] (5)	MnO_2	150	85
5	[{Rh(nbd)Cl}$_2$] (5)	$Na_2S_2O_8$	150	2
6	[{Rh(nbd)Cl}$_2$] (5)	$KMnO_4$	150	7
7	[{Rh(nbd)Cl}$_2$] (5)	AgOAc	150	41
8[b]	[{Rh(nbd)Cl}$_2$] (5)	MnO_2	150	53
9	[{Rh(nbd)Cl}$_2$] (5)	MnO_2	90	50
10	[{Rh(nbd)Cl}$_2$] (5)	MnO_2	120	75
11	[{Rh(nbd)Cl}$_2$] (2.5)	MnO_2	150	73
12	–	MnO_2	150	0
13	[{Rh(nbd)Cl}$_2$] (5)	–	150	0

[a] Unless otherwise noted, all reactions were conducted on a 0.2 mmol scale with 3 equiv of oxidant in a sealed tube in 0.5 mL H_2O. Yields are those of isolated products. For other results from the optimization of the reaction conditions please see the Supporting Information. [b] 1 equiv of MnO_2 was used. Cp* = C_5Me_5.

Gratifyingly, benzoic acid with electron-donating substitutents underwent the dehydrogenative coupling reaction in high yields (Table 2; **2a**–**d**). However, with 3,4,5-trimethoxybenzoic acid (**1e**), the reaction afforded the product in 40 % yield, presumably owing to the steric hindrance of the *meta*-methoxy group of **1e**. Importantly, this transformation was successful for gram-scale preparation, giving a comparable

Table 2: Scope of homodehydrogenative coupling reactions of aromatic carboxylic acids[a]

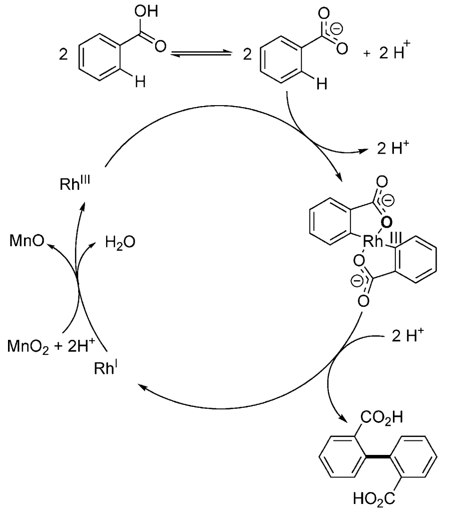


2a 85%
2b 90%
2c 81%
2d 76%
2e 40% (gram scale 43%) [b]
2f 60%
2g 40%
2h 89% (gram scale 80%) [c]
2i 59%
2j 47%
2k 36%
2l 47%
2m 58%
2n 83% (63% *n*=0, 8% *n*=1, 12% *n*=2)
2o 80% (67% *n*=0, 13% *n*=1)
2pa **2pb**
Total: 87% (**2pa** 64%, **2pb** 23%)

[a] All reactions were carried out with aromatic acid (0.2 mmol), [{Rh(nbd)Cl}$_2$] (5 mol %), and MnO_2 (3 equiv) in a sealed tube in 0.5 mL H_2O at 150 °C for 24 h unless otherwise stated. All yields are those for isolated products. [b] 1 gram (4.7 mmol) of aromatic acid, 0.6 mol % catalyst and 5 mL H_2O was used, reaction time = 72 h; [c] 1 gram (7.2 mmol) of aromatic acid, 0.4 mol % catalyst and 5 mL H_2O was used, reaction time = 72 h.

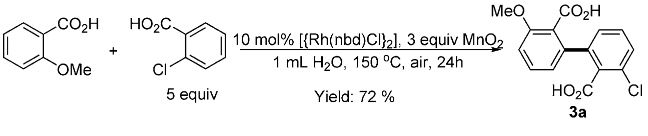
yield by using a much lower amount of catalyst (0.6 mol %) with a longer reaction time (72 h). For substrates bearing cyclopentyloxyl or *tert*-butyl groups, the reactions also proceeded smoothly to furnish the desired products **2f** (60 %) and **2g** (40 %), respectively. The slightly lower yields are most likely the result of their poor solubility in water. In contrast, for substrates bearing electron-withdrawing groups (**1i–l**), the reaction afforded the desired product in yields ranging from 30 to 59 % (**2i–l**); with *ortho*-fluorobenzoic acid (**1h**), the reaction yield increased to 89 %. Notably, the corresponding gram-scale synthesis of **2h** was achieved in 80 % yield by using 0.4 mol % of the rhodium catalyst. Interestingly, the substrate with a hemiacetal was also well tolerated (**2l**). Under the optimized reaction conditions, β-naphthoic acid (**1m**) could be converted into **2m** exclusively at the β-position. Interestingly, consecutive coupling products were observed in the absence of *ortho*-substituents (**2n–p**).

Based on our previous reports on rhodium-catalyzed carboxyl-directed C–H bond activation[15b] and other literature studies,[14] a plausible mechanism for this transformation is proposed in Scheme 2. We hypothesized that a rhodium(III)-initiated dual cyclometallation and a subsequent reductive elimination would produce the desired coupling product dicarboxylic acid and a rhodium(I) species, which was reoxidized to the active rhodium(III) catalyst by the oxidant MnO_2 (Scheme 2).



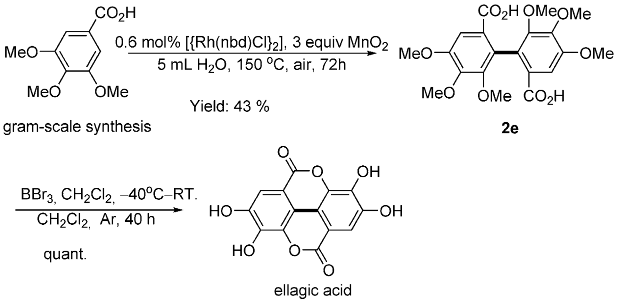
Scheme 2. Proposed mechanism for the rhodium-catalyzed aromatic dehydrogenative coupling strategy for the construction of the diaryl acid motif.

The cross-dehydrogenative coupling[18] reaction of aromatic acids was also examined briefly (Scheme 3), and it gave a good yield (72 %) of the isolated product when using the unoptimized reaction conditions. This result shows the potential extension of this method to the synthesis of nonsymmetric diaryl acids as well.



Scheme 3. Extension to nonsymmetric diaryl diacids.

The simplicity and convenience of this novel method to access diaryl diacids can be readily applied in the synthesis of natural products. As a proof-of-concept, the antitumor natural product ellagic acid can be effectively assembled from 3,4,5-trimethoxybenzoic acid in two steps on a gram scale (Scheme 4), although concise syntheses of ellagic acid have been reported previously.[19]



Scheme 4. Total synthesis of ellagic acid.

In conclusion, an efficient and practical synthetic approach towards highly important diaryl acids has been developed for the first time by using a rhodium-catalyzed oxidative homocoupling of simple aromatic acids in water. The reaction is environmentally friendly, operationally simple, not sensitive to air, and compatible with water. Additionally, this reaction can be easily scaled up to the gram-scale level with low rhodium catalyst loading (0.4–0.6 mol %). As an example, this method also provides an avenue for the rapid assembly of natural products such as ellagic acid, and unlocks the potential for the preparation of axial chiral compounds, which are widely used as chiral ligands in asymmetric catalysis. Further studies on the scope and mechanism of this method are in progress.

Experimental Section

A typical experimental procedure: A solution of an aromatic acid (0.2 mmol), [{Rh(nbd)Cl}$_2$] (4.6 mg, 0.01 mmol) and activated MnO_2 (purchased from Aldrich and used as received, 52.2 mg, 0.6 mmol) in distilled water (0.5 mL) was stirred in a sealed tube under an atmosphere of air at 150 °C for 24 h. The reaction mixture was then cooled to room temperature and acidified by dilute HCl to $pH < 3$, and then the solvent was evaporated in vacuo. The residue was purified by preparative thin-layer chromatography (TLC) on silica gel with diethyl ether containing an appropriate amount of formic acid to give the pure product.

Keywords: C–H activation · cross-coupling · rhodium · synthetic methods · water chemistry

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