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Iodine-Promoted Efficient Homocoupling of Arylboronic Acids in PEG-400 under Aerobic Conditions

Jincheng Mao,*[a] Qiongqiong Hua,[b] Guanlei Xie,[a] Zhigang Yao,[c] and Daqing Shi*[a]

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Iodine is able to catalyze the homocoupling of arylboronic acids in moderate to good yields in PEG-400 under aerobic conditions. This transition-metal-free catalytic system is readily available at low cost, which is practical and useful. It is noteworthy that a high activity of our catalytic system could be maintained under mild conditions (100 °C) by addition of water.

Introduction

The synthesis of biaryls through C–C bond-forming reactions is of great interest because of the prevalence of this structural motif in a myriad of bioactive important natural products and pharmaceutically interesting compounds.[1] Various biaryl coupling methods have thus been developed in the past decades.[2] Among them, cross-coupling between arylboronic acids and aryl halides has proved effective and has attracted much attention.[2a]

In contrast, there are very few preparations of symmetrical biaryls through homocoupling reactions,[2b,3] although symmetrical biaryl moieties are common in the structures of many natural products (Figure 1). Crisamicin A, for example, which has been isolated from a mud sample in the Philippines, was found to be active primarily towards Gram-positive bacteria.[4] Biphenomycin B, which was previously isolated from culture filtrates of *Streptomyces griseorubiginosus* No. 43708, was found to be a highly potent antibiotic against Gram-negative, β-lactam-resistant bacteria.[5] The discovery of these compounds' important pharmacological properties thus stimulated substantial interest in homocoupling chemistry.[6]

The procedures commonly used for the preparation of symmetrical biaryls rely on palladium-catalyzed dimerization of arylboronic acids in the presence of various ligands.[7] Studies of the possible mechanism have been performed to show the importance of oxidative coupling in the presence of dioxygen.[8] With the increasing demand for environmentally friendly methods, we[9a] and other groups[9b–9e] have found that the Pd-catalyzed oxidative dimerization of arylboronic acids or esters can be performed in aqueous media. In many cases, addition of certain oxidants was necessary to acquire high yields of biaryls.[10]

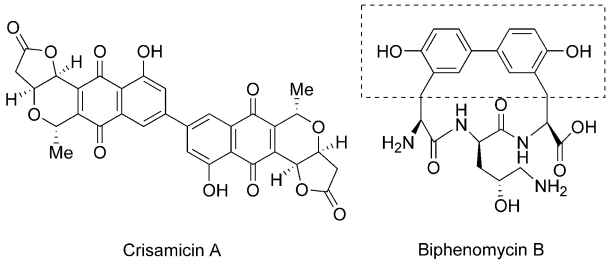

Figure 1. Symmetrical biaryl moieties in natural products.

Recently, Demir et al. reported that certain copper species are able to mediate the homocoupling of arylboronic acids in moderate to good yields.[11] Corma and co-workers described the supported-gold-catalyzed homocoupling of phenylboronic acid with high levels of conversion and selectivity.[12] Very recently, homocoupling of arylboronic acids mediated by Mn^{III}[13] and by $Ag_2O/CrCl_2$[14] has been reported. Although considerable efforts have been made, the development of other effective routes to these biaryls would be highly desirable.[15] In this context, iodine-mediated homocoupling of arylboronic acids under aerobic conditions will possibly provide a promising alternative.

Results and Discussion

One simple way to address demands for recyclability and other environmental concerns is to immobilize a catalyst in a liquid phase by dissolving it in a nonvolatile liquid of low

[a] Key Laboratory of Organic Synthesis of Jiangsu Province, College of Chemistry, Chemical Engineering and Materials Science, Suzhou University,
Suzhou 215123, P. R.China
[b] Department of Chemistry, Xuzhou Normal University,
Xuzhou 221116, P. R. China
[c] Analytical & Testing Center, Suzhou University,
Suzhou 215123, P. R. China
Fax: +86-512-6588-0089
E-mail: jcmao@suda.edu.cn

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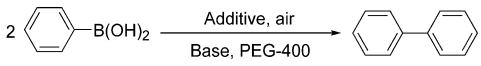
toxicity, such as a poly(ethylene)glycol (PEG).[16] Accordingly, we reported the first example of copper-powder-catalyzed reusable Suzuki–Miyaura coupling,[17] which proved capable of affording almost quantitative yields of coupling products from aryl iodides. In the presence of iodine as a necessary additive, it was also possible to perform smooth coupling reactions involving aryl bromides or chlorides. However, we do not know the exact role of the iodine in the mechanism, and so for this paper a series of experiments were conducted to try to make this clear.

The experimental data for the screening conditions are listed in Table 1. Initially, the homocoupling of phenylboronic acid was chosen as the model reaction. Iodine-catalyzed homocoupling reactions were conducted both under argon and in air (Table 1, Entries 1–2). As expected, the reaction under aerobic conditions gave a higher yield of the desired product (Entry 2). At the same time, TLC showed that the use of common organic solvents, such as dimethylformamide, dimethyl sulfoxide, dioxane, ethanol, and dichloromethane, did not afford the biphenyl. The blank experiment showed that without iodine, the reaction could not occur (Entry 3). Other halogenated reagents such as bromine and NBS (*N*-bromosuccinimide) were also investigated, but no catalytic reactions occurred (Entries 4–5). Without K_2CO_3, we also could not obtain the desired product (Entry 6).

Table 1. Screening of catalytic conditions for iodine-mediated homocoupling of phenylboronic acid.[a]



1a 2aa

Entry	Additive	Base	Temp. [°C]	Time [h]	Yield [%][b]
1[c]	I_2 (20%)	K_2CO_3	110	24	6
2	I_2 (20%)	K_2CO_3	110	24	11
3[d]	–	K_2CO_3	110	24	n. r.
4[d]	Br_2 (20%)	K_2CO_3	110	24	n. r.
5[d]	NBS (20%)	K_2CO_3	110	24	n. r.
6[d]	I_2 (20%)	–	110	24	n. r.
7	I_2 (20%)	KF	140	24	10
8[d]	I_2 (20%)	Cs_2CO_3	140	24	n. r.
9	I_2 (20%)	KOH	140	24	trace
10	I_2 (20%)	K_2CO_3	140	24	31
11	I_2 (20%)	K_3PO_4	140	24	30
12	I_2 (20%)	K_2CO_3	140	12	20
13	I_2 (30%)	K_2CO_3	140	24	42
14	I_2 (50%)	K_2CO_3	140	24	44
15[e]	I_2 (30%)	K_2CO_3	140	24	43
16[f]	I_2 (30%)	K_2CO_3	140	24	40
17	I_2 (30%)	K_2CO_3	140	48	54
18	I_2 (50%)	K_2CO_3	140	48	73
19	I_2 (100%)	K_2CO_3	140	48	97
20	I_2 (100%)	K_2CO_3	100	48	75
21[g]	I_2 (100%)	K_2CO_3	140	48	7

[a] Reaction conditions: **1a** (0.8 mmol), I_2 (20 mol-%), base (4.0 mmol), PEG-400 (2 mL), 100–140 °C, in air. [b] Isolated yield. [c] Under argon. [d] No reaction. [e] TBAB (10 mol-%) as the additional additive. [f] LiCl (10 mol-%) as the additional additive. [g] K_2CO_3 (0.8 mmol) was used.

We therefore performed the catalytic reactions at 140 °C. By screening different bases, including KF, Cs_2CO_3, KOH, K_2CO_3, and K_3PO_4 (Entries 7–11), we found that K_2CO_3 was the best one (Entry 10). Shortened times afforded disappointing results (Entry 12). Use of a higher loading of iodine was favorable for the synthesis of biphenyl (Entries 13–14), whereas with TBAB (nBu$_4$NBr) or LiCl as the additive we were unable to achieve enhanced results (Entries 15–16). Use of more iodine or of a longer reaction time (48 h) afforded better yields (Entries 17–19). Finally, use of 1 equiv. of iodine afforded biphenyl almost quantitatively (97% yield, Entry 19). With the same reaction time, reduction of the reaction temperature or of the amount of base both gave inferior results (Entries 20–21).

Under the optimized conditions, various arylboronic acids were employed in the homocoupling reactions; the results are summarized in Table 2. With 4-fluorophenylboronic acid as the substrate, an increase in the temperature from 140 °C to 150 °C led to an enhanced yield (Table 2, Entries 2–3). However, this modification was not suitable for the reaction of 4-chlorophenylboronic acid (Entries 4–5), and an even worse result was obtained with ethyl bromoacetate used as the additional oxidant (Entry 6), although for the reaction of 3-methoxyphenylboronic acid this oxidant was beneficial (Entries 7–8). Such irregularity has also been observed in Pd-catalyzed homocouplings.[10a] Performing of this reaction under oxygen gave a further improvement in the result (Entry 9).

With 2-methoxyphenylboronic acid as the substrate, lower yields of biaryl were obtained even at higher temperature (Entries 10–11), which is possibly due to the steric substituent effect. This explanation was further supported by the fact that the self-coupling product of 2-methylphenylboronic acid could not be acquired even if iodine (3 equiv.) was employed (Entry 12).

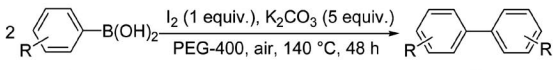
3-Formylphenylboronic acid gave a 50% yield of the corresponding biaryl (Entry 13).

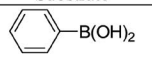
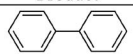
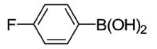
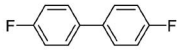
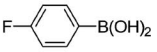
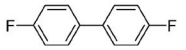
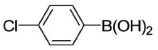
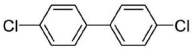
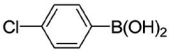
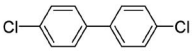
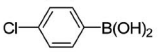
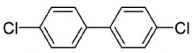
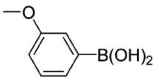
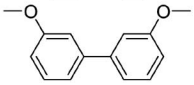
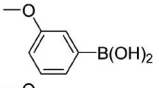
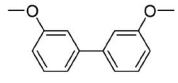
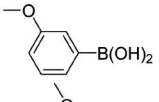
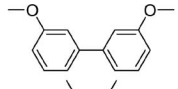
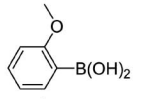
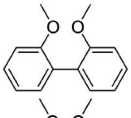
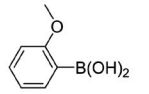
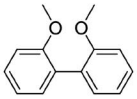
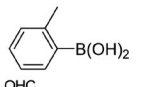
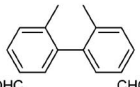
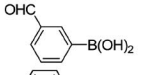
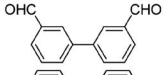
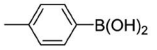
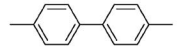
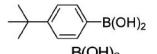
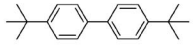
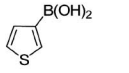
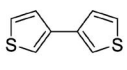
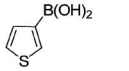
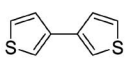
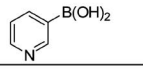
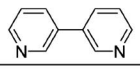
The homocoupling of *para*-substituted arylboronic acids gave good results (Entries 14–15), whereas with thiophen-3-ylboronic acid as the substrate only a moderate yield of the desired product was obtained (Entry 16). To our surprise, the same coupling reaction did not take place when carried out under oxygen (Entry 17). In addition, the self-coupling of 3-pyridinylboronic acid gave no product (Entry 18).

Subsequently, we tried to apply the I_2/K_2CO_3/PEG-400 system for the homocoupling of other aryl reagents. As shown in Scheme 1, no biphenyl was obtained when iodobenzene or trimethoxy(phenyl)silane were employed. However, this catalytic system was effective for various borane sources, such as phenylboronic esters and potassium trifluoro(phenyl)borate.

Couplings between two different arylboronic acids, to provide unsymmetrical biaryls, could be conducted smoothly under the standard conditions. As shown in Scheme 2, as well as the unsymmetrical products, self-coupling products of the two individual arylboronic acid components were also obtained, as would be expected and as was

Table 2. Iodine-promoted homocoupling of various arylboronic acids.[a]

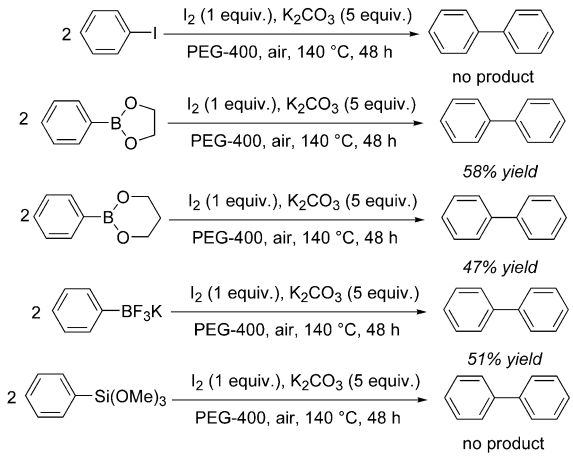


Entry	Substrate	Product		Yield [%][b]
1			**2aa**	97
2			**2bb**	56
3[c]			**2bb**	64
4			**2cc**	53
5[c]			**2cc**	36
6[d]			**2cc**	20
7			**2dd**	20
8[d]			**2dd**	43
9[e]			**2dd**	66
10			**2ee**	37
11[c]			**2ee**	< 10
12[f,g]			**2ff**	n. r.
13			**2gg**	50
14			**2hh**	91
15			**2ii**	> 99
16			**2jj**	50
17[e,g]			**2jj**	n. r.
18			**2kk**	trace

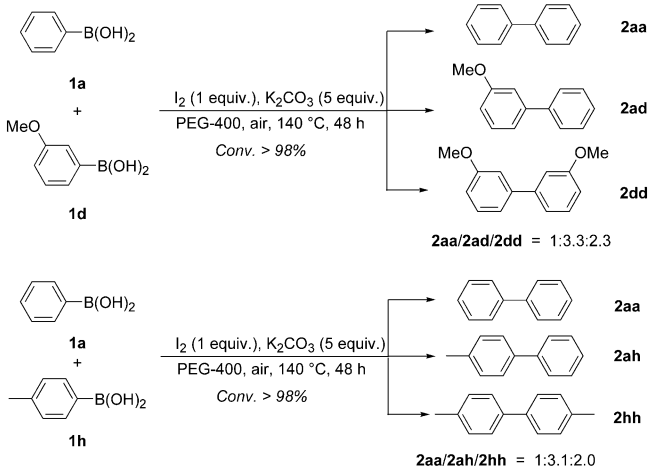
[a] Reaction conditions: **1** (0.8 mmol), I_2 (100 mol-%), K_2CO_3 (4.0 mmol), PEG-400 (2 mL), 140 °C, 48 h, in air. [b] Isolated yield. [c] Temperature 150 °C. [d] $BrCH_2COOEt$ (1 equiv.) as additive. [e] The catalytic reaction was performed under oxygen. [f] No reaction occurred even with iodine (8 equiv.). [g] No reaction.

confirmed by GC-MS. It is interesting to observe that the amount of cross-coupling product is approximately the sum of the amounts of the two different homocoupling products.

In the application of our methodology to two naphthylboronic acids (Scheme 3), GC-MS suggested that naphthalene was detectable besides the biaryl products. It was found that the ratios between the biaryl and naphthalene



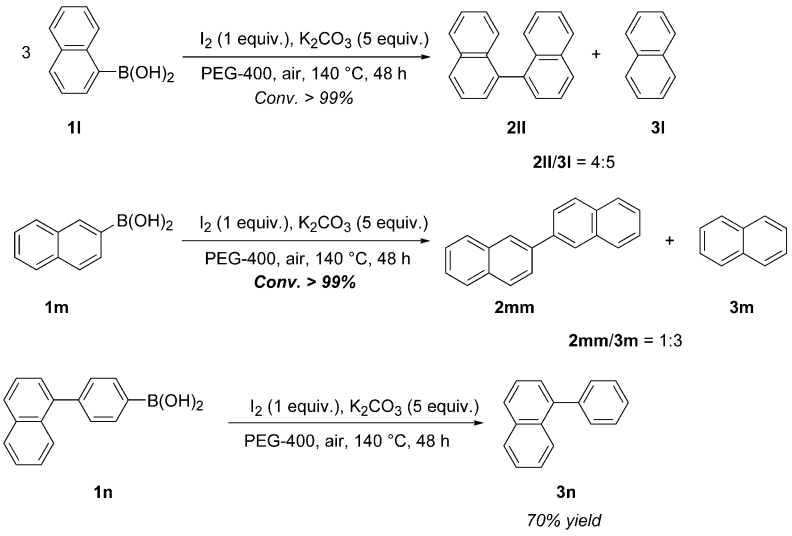
Scheme 1. Other aryl reagents investigated with the I_2/K_2CO_3/PEG-400 system.



Scheme 2. Unsymmetrical biaryls synthesized by our method.

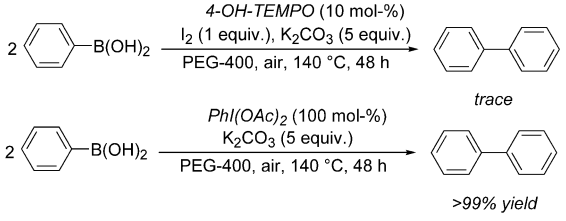
were different; we believe that this might be due to the difference between the activities of the α- and the β-positions on the naphthyl ring. Meanwhile, 4-(naphthalen-1-yl)phenylboronic acid was also employed in the homocoupling reaction. To our astonishment, only a trace of the desired self-coupling product was found, and 1-phenylnaphthalene was obtained in 70% yield. From these experimental data, we wondered whether the coupling reaction in the presence of the I_2/K_2CO_3/PEG-400 system proceeds through a radical mechanism.

In order to clarify the possible radical nature of this reaction, the homocoupling of phenylboronic acid with the I_2/K_2CO_3/PEG-400 system was conducted in the presence of 4-OH-TEMPO (4-hydroxy-2,2,6,6-tetramethylpiperidin-1-oxyl, 10 mol-%, Scheme 4). GC-MS showed that no biphenyl was obtained. This suggests that once the phenylboronic acid has decomposed to the phenyl radical, it will be immediately quenched by 4-OH-TEMPO. Actually, it has been reported that aryl radicals are generated from aryl-



Scheme 3. Homocoupling of several specific arylboronic acids with the I_2/K_2CO_3/PEG-400 system.

boronic acids in the presence of some oxidants.[13,18] In addition, Demir et al. observed that aryl radicals were obtained from arylhydrazines in the presence of the potassium permanganate/carboxylic acid/organic solvent system.[19]



Scheme 4. Experiments for consideration of the possible mechanism.

The question that then arises is: What is the oxidant in our catalytic reaction? Consequently, hypervalent iodine, such as iodobenzene diacetate, was employed to replace iodine. The result showed that biphenyl was obtained almost quantitatively, and so we propose that *iodine serves not only as the catalyst but also as the oxidant*. We speculate that iodine is probably transformed into some hypervalent iodine species in this particular solvent (PEG-400) in the presence of oxygen from the air and of K_2CO_3 in excess. In this way, the putatively generated oxidant would perhaps result in the decomposition of arylboronic acids at high temperature (140 °C), and the corresponding radicals could be produced. This could thus explain why the homocoupling products and the decomposition products were obtained during the self-coupling of arylboronic acids.

For interest in green chemistry, we then began to explore the couplings in aqueous solution at lower temperature (100 °C), and the relationship between volume ratio (PEG-400/water) and the yield of biphenyl is shown in Table 3. It was found that when only water was used, no product was obtained, which may be attributed to the poor solubility of the phenylboronic acid in water (Table 3, Entry 1). With increasing PEG-400/H_2O ratio, greater amounts of product were obtained up to a ratio of 5:1, at which we obtained our best result, in the form of a 94% yield (Entry 5). However, further increases in the ratio resulted in poorer results (Entries 6–7). We can thus see that appropriate amounts of water might accelerate the self-coupling reactions.

Table 3. Homocoupling of phenylboronic acid in aqueous solutions.[a]

Entry	Solvent [v/v]	Temp. [°C]	Yield [%][b]
1	H_2O	100	trace
2	PEG-400/H_2O (1:2)	100	12
3	PEG-400/H_2O (1:1)	100	24
4	PEG-400/H_2O (2:1)	100	72
5	PEG-400/H_2O (5:1)	100	94
6	PEG-400/H_2O (10:1)	100	67
7	PEG-400/H_2O (20:1)	100	58

[a] Reaction conditions: **1a** (0.8 mmol), I_2 (100 mol-%), K_2CO_3 (2.0 mmol), solvent (2 mL), 100 °C, 48 h, in air. [b] Isolated yield.

Conclusions

We showed that iodine was able to promote the homocoupling of arylboronic acids in PEG-400 under aerobic conditions in moderate to good yields. In comparison with previously reported systems, this transition-metal-free catalytic system is readily available and at low cost, unlike palladium catalyst systems, which has particular relevance to the fields of drug discovery and manufacture. It is noteworthy that addition of water endowed our catalytic system with a high activity under milder conditions. Further work is in progress in this laboratory with the aims of extending the application of these readily available catalytic systems in other coupling transformations and of studying the mechanisms of such iodine-mediated homocoupling reactions.

Experimental Section

General Remarks: All reactions were carried out in air. All arylboronic acids or esters and other chemical reagents were purchased

from Aldrich or Alfa. In order to evade issues of metal contamination, extensive experiments were performed in new flasks with new stirring bars and new caps. The most important observation is that all reagents including iodine, arylboronic acids, K_2CO_3, and PEG-400 were also examined by ICP-MS and were not found to contain any metals such as Pd, Cu, etc. Flash column chromatography was performed with silica gel (300–400 mesh). Analytical thin-layer chromatography was performed with glass plates precoated with 200–400 mesh silica gel impregnated with a fluorescent indicator (254 nm). NMR spectra were measured in $CDCl_3$ with a Varian Inova 400 NMR spectrometer (400 MHz or 300 MHz) using TMS as an internal reference. Products were characterized by comparison of ^1H and ^{13}C NMR spectroscopic data with those in the literatures.

Typical Experimental Procedure for Iodine-Mediated Homocoupling of Arylboronic Acids: A capped tube (10 mL) was charged with potassium carbonate (4.0 mmol), and the arylboronic acid (0.8 mmol), in PEG-400 (2 mL) as solvent, and iodine (0.4 mmol) were then added under air. The tube was sealed under air, and the mixture was heated to 140 °C and stirred for 48 h. After cooling to room temperature, the mixture was diluted with water, and the combined aqueous phases were extracted three times with ethyl acetate. The organic layers were combined, dried with Na_2SO_4, and concentrated to yield the crude product, which was further purified by silica gel chromatography with petroleum ether as eluent to provide the desired product.

Supporting Information (see footnote on the first page of this article): ^1H, and ^{13}C NMR spectra of the coupling products.

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