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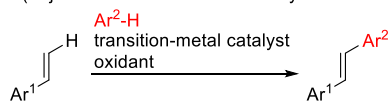
Metal- and Oxidant-Free Alkenyl C–H/Aromatic C–H Cross-Coupling Using Electrogenerated Iodosulfonium Ions

Ryutaro Hayashi^[a], Akihiro Shimizu^[a], Jonathan A. Davies^[b], Chris Willis^[b], and Jun-ichi Yoshida^{*[c]}

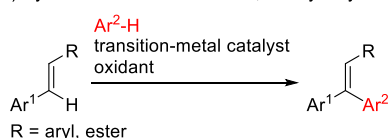
Abstract: A three-step transformation consisting of (1) addition of electrochemically generated iodosulfonium ions to vinylarenes to give (1-aryl-2-iodoethoxy)sulfonium ions, (2) nucleophilic substitution by subsequently added aromatic compounds to give 1,1-diaryl-2-iodoethane, and (3) elimination of HI with a base to give 1,1-diarylethenes was developed. The transformation serves as a powerful metal- and chemical-oxidant-free method for alkenyl C–H/aromatic C–H cross-coupling.

C–H/C–H cross-coupling serves as a powerful method for making C–C bonds and they are synthetically useful from viewpoints of atom-^[1] and step-economy.^[2] In particular, alkenyl C–H/aromatic C–H cross-coupling is useful for the introduction of alkenyl groups to aromatic compounds. The Fujiwara-Moritani reaction is an early example of a metal-catalyzed alkenyl C–H/aromatic C–H cross-coupling.^[3] Later, dehydrogenative Heck reactions were developed.^[4] In these approaches, 1,2-diarylethenes are often obtained as major products from vinylarenes (Scheme 1a).

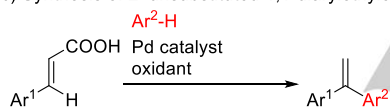
a) Synthesis of 1,2-diarylethenes
(Fujiwara-Moritani Reaction/Dehydrooxidative Heck Reaction)



b) Synthesis of 2-substituted 1,1-diarylethenes



c) Synthesis of 2-unsubstituted 1,1-diarylethenes



Scheme 1. Synthesis of Diarylethylene.

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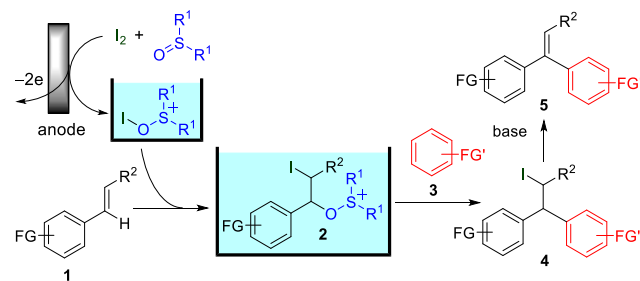
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Because many biologically active compounds contain the 1,1-diarylethylene structure,^[5] an efficient method for the synthesis of 1,1-diarylethenes has been highly desired. 1,1-Diarylethenes having an aryl or ester group at the 2-position were synthesized by metal-catalyzed alkenyl C–H/aromatic C–H cross-coupling of vinylarenes with aromatic compounds (Scheme 1b).^[6] However, the method for the synthesis of 2-unsubstituted 1,1-diarylethenes is limited to the decarboxylative cross-coupling of cinnamic acid derivatives and arenes (Scheme 1c)^[7] and cross-coupling of indolizine derivatives and vinylarenes.^[8] Therefore, the development of a more versatile method of alkenyl C–H/aromatic C–H cross-coupling to make 1,1-diarylethenes still remains a challenge.

Electrochemical oxidation serves as a powerful method for generating reactive cationic species under mild conditions without metal catalysts.^[9] Various electrochemical C–H bond transformations including C–H/C–H cross-coupling have been developed using the reactive cationic species.^[10] Recently, we reported the stabilized cation pool method, in which organic cations are electrochemically generated and accumulated in solutions in the presence of stabilizing agents.^[11] This methodology has enabled us to develop benzylic C–H/aromatic C–H coupling. In this case electrooxidatively generated benzyl cations are stabilized by sulfilimines. We envisaged that stabilized cation pools can also be generated by addition to alkenes. In fact, iodosulfonium ions electrooxidatively generated from I₂ and sulfoxides react with vinylarenes **1** to give β -iodoalkoxysulfonium ions **2** (Scheme 2).^[12] If ions **2** react as stabilized benzyl cations with aromatic compounds **3**, the resulting 1,1-diaryl-2-iodoethanes **4** could be easily transformed to 1,1-diarylethene **5** by treatment with a base. Thus, alkenyl C–H/aromatic C–H cross-coupling can be achieved. The concept works, and herein we report metal- and oxidant-free alkenyl C–H/aromatic C–H cross-coupling using electrogenerated iodosulfonium ions.

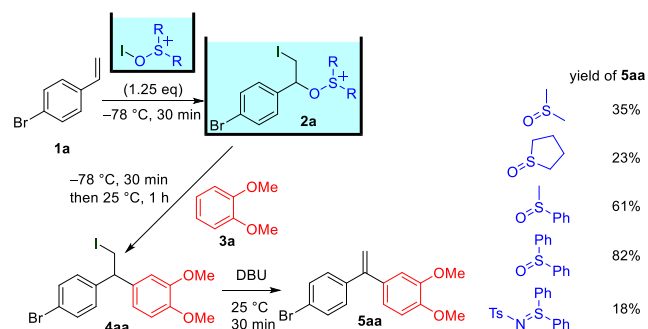


Scheme 2. Alkenyl C–H/Aromatic C–H Cross-Coupling via β -Iodoalkoxysulfonium Ion.

We first screened sulfoxides. Iodine was electrochemically oxidized in the presence of various sulfoxides in dichloromethane to generate the corresponding iodosulfonium ions. After the electrolysis was complete, 4-bromostyrene (**1a**) was added to the

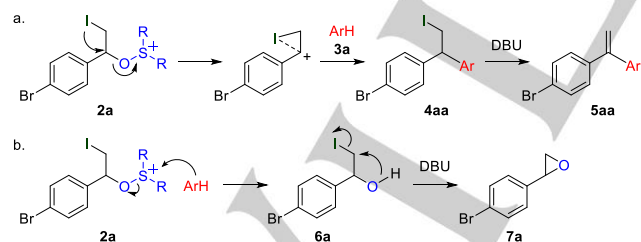
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anodic solution to generate β -iodoalkoxysulfonium ion **2a** (Scheme 3).



Scheme 3. Screening of sulfoxides. Iodine (0.5 mmol) was electrochemically oxidized in the presence of 2.0 mmol of sulfoxide or sulfilimine in a 0.3 M solution of Bu_4NBF_4 in CH_2Cl_2 at -78°C . After 3.0 *F* of electricity was applied, **1a** (0.4 mmol) was added. The resulting solution was treated with **2a** (2.0 mmol) and DBU (2.0 mmol). Isolated yields based on the amount of **1a** used are shown.

The reaction of **2a** with 1,2-dimethoxybenzene (**3a**) gave **4aa** presumably via a benzylic cation stabilized by intramolecular participation of iodine, indicating that the solution of **2a** serves as a stabilized cation pool for aromatic compounds. The subsequent reaction with DBU^[13] gave 1,1-diarylethylene **5aa** (Scheme 4a). When DMSO was used as a sulfoxide, **5aa** was obtained in 35% yield. In this case, the corresponding epoxide **7a** was also obtained in 13% yield probably due to attack on the sulfur atom of **2a** to give **6a**, which underwent cyclization by the action of DBU to give **7a** (Scheme 4b).^[14,15] To suppress the epoxide formation, we examined sulfoxides having bulky substituents. When tetramethylene sulfoxide was used, the yield of **5aa** decreased to 23%. However, the use of methyl phenyl sulfoxide gave rise to an increase in the yield to 61%. Diphenyl sulfoxide was more effective and gave **5aa** in 82% yield. On the other hand, the use of sulfilimine^[11] gave **5aa** in only 18% yield. Therefore, hereafter we used diphenyl sulfoxide.



Scheme 4. A Plausible Mechanism.

β -Iodoalkoxysulfonium ion **2a** derived from **1a** and diphenyl sulfoxide was successfully characterized by mass spectroscopy and ^1H and ^{13}C NMR analyses (See supporting information for details). There was a characteristic lower chemical shift of the benzylic proton of **2a** compared with that of 1-(4-bromophenyl)-2-iodoethanol (**6a**) (Figure 1). Variable-temperature ^1H NMR

analysis revealed that **2a** is stable at temperatures lower than -20°C (Figure S2). Notably, 1,1-diaryl-2-iodoethane **4aa** was isolated in 85% yield when DBU was not added.

Table 1. Scope of Nucleophiles

entry	substrate	t^1 (min)	product	yield
1	1a	30	5aa	82%
2	1b	30	5ba	74% ^{a)}
3	1c	30	5ca	64%
4	1d	90	5da	81%
5	1e	30	5ea	70%
6	1f	30	5fa	67%
7	(<i>Z</i>)- 1g	90	5ga	70% (<i>E/Z</i> = 28:72)
8	(<i>E</i>)- 1g	90	5ga	77% (<i>E</i> isomer only)
9	1h	30	5ha	51% ^{b)}
10	1i	30	5ia'	80% ^{c)}
11	1j	30	5ja	53% ^{d)}

1j (0.5 mmol) was electrochemically oxidized in the presence of 2.0 mmol of diphenyl sulfoxide in a 0.3 M solution of Bu_4NBF_4 in CH_2Cl_2 at -78°C using a divided cell under constant current conditions. After 3.0 *F* of electricity was applied, **1x** (0.4 mmol) was added to the resulting anodic solution. Then, **3a** (2.0 mmol) was added and the resulting **4xa** was treated with DBU (2.0 mmol). Isolated yields based on **1** are shown. a) Yields were determined by GC. b) After addition of DBU, the mixture was stirred at 35°C for 3 h. c) 10 equiv of DBU was used, and the mixture was stirred for 68 h. d) **1j** (0.2 mmol) was used.

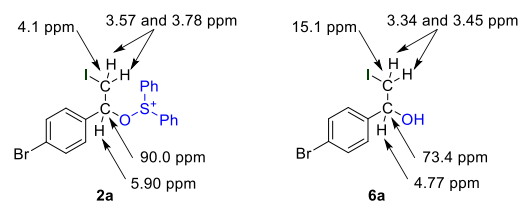
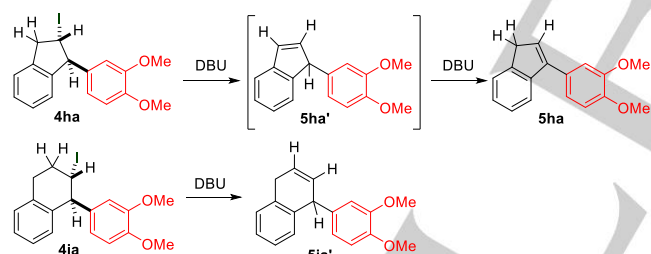


Figure 1. ^1H and ^{13}C NMR chemical shifts of **2a** and **6a**.^[16]

The present one-pot transformation is applicable to other vinylarenes bearing various functional groups as shown in Table 1. Styrene (**1b**) and styrenes having methyl, ester, and bromo groups on the aromatic ring gave the corresponding 1,1-diarylethylenes (entries 1–6). The reactions of substrates with trisubstituted alkenes was interesting. The reaction of (*Z*)-**1g** with **3a** gave (*Z*)-**5ga** as a major product and (*E*)-**5ga** as a minor product, while that of (*E*)-**1g** with **3a** gave (*E*)-**5ga** as a sole product. The present reaction can be applied to cyclic alkenes. Indene (**1h**), a 5-membered ring alkene, gave the corresponding 1,1-diarylethylene **5ha** via *trans*-**4ha**,^[17] while 1,2-dihydronaphthalene (**1i**), a 6-membered ring alkene, gave the alkene **5ia'** instead of the corresponding diarylethylene. The elimination of HI from **4ha** seemed to give **5ha'**, because the proton attached to the carbon bearing the dimethoxyphenyl group is *cis* to the iodine. However, rapid isomerization might take place to give **5ha**.^[18] In the case of **4ia**, **5ia'** was obtained as a sole product (Scheme 5). No isomerization of **5ia'** took place. The reaction of **1j** with two equivalents of iododiphenylsulfonium ion and **3a** gave the double arylated alkene **5ja**.



Scheme 5. Proposed Mechanism of the Elimination in the Reaction of Cyclic Alkenes.

Next, we examined the reactions of β -iodoalkoxysulfonium ion **2a** with various aromatic nucleophiles (Table 2). 1,2-Dimethoxybenzene (**3a**) and 1,4-dimethoxybenzene (**3c**) gave **5aa** and **5ac**, respectively in good yields, while 1,3-dimethoxybenzene (**3b**) gave **5ab** in a poor yield. Anisole (**3d**) gave the 1,1-diarylethylene as a mixture of two isomers, while 2-iodoanisole (**3e**) gave **5ae** as a single isomer. Xylene (**3f**) and 1-methylnaphthalene (**3g**) gave the corresponding 1,1-diarylethylenes **5af** and **5ag**, respectively. Heterocyclic compounds such as *N*-tosylpyrrole (**3h**) and bromothiophene (**3i**) gave the corresponding 1,1-diarylethylenes **5ah** and **5ai**, respectively.

Table 2. Scope of Nucleophiles

entry	Ar-H	product	yield
1			82%
2			32%
3			61%
4			70% <i>o,p</i> = 11:89
5			69%
6			74% ^{a)}
7			80%
8			62%
9			64%

I_2 (0.5 mmol) was electrochemically oxidized in the presence of 2.0 mmol of diphenyl sulfide in a 0.3 M solution of Bu_4NBF_4 in CH_2Cl_2 at -78°C . After 3.0 F of electricity was applied, **1a** (0.4 mmol) was added to the resulting solution, followed by the treatment with **3** (2.0 mmol) and DBU (2.0 mmol). Isolated yield based on **1a** used is shown. a) After the addition of **3f** (5 mL), the mixture was stirred at -20°C for 42 h then at 25°C for 1 h.

In conclusion, we have developed metal-free alkenyl C–H/aromatic C–H cross-coupling using iododiphenylsulfonium ion which can be easily generated from I_2 and diphenyl sulfide using the electrochemical method. Notably, the present transformation can be performed in one-pot without isolating the intermediates. We hope that the present method will be applied to synthesis of various 1,1-diarylethylenes which serve as useful building blocks for making biologically interesting compounds.

Experimental Section

In the anodic chamber of an H-type divided cell were placed iodine (63.4 mg, 0.25 mmol), diphenylsulfide (404 mg, 1.00 mmol), and 0.3 M Bu₄NBF₄/CH₂Cl₂ (10.0 mL). In the cathodic chamber were placed trifluoromethanesulfonic acid (80 μL), 0.3 M Bu₄NBF₄/CH₂Cl₂ (10.0 mL). The constant current electrolysis (8.0 mA) was carried out at -78 °C with magnetic stirring until 3.0 F of electricity was consumed. To the anodic chamber was added a solution of styrene substrates **1** (0.40 mmol) in CH₂Cl₂ (0.5 mL), and to the cathodic chamber 0.5 mL of CH₂Cl₂ was added at -78 °C. After the solution was stirred for 30 min at -78 °C, to the anodic chamber was added a solution of 1,2-dimethoxybenzene (**3a**) (254 μL, 2.00 mmol) in CH₂Cl₂ (0.5 mL), and to the cathodic chamber 0.5 mL of CH₂Cl₂ was added at -78 °C. The solution was stirred for 30 min at -78 °C and for 1 h at 25 °C. Then DBU (300 μL) was added to both the anodic and cathodic chambers, and the resulting mixture was stirred for additional 30 min. The solution in the anodic chamber was collected and the solvent was removed under reduced pressure. The crude product was purified by flash chromatography and GPC to obtain the coupling product **5xa**.

Acknowledgements

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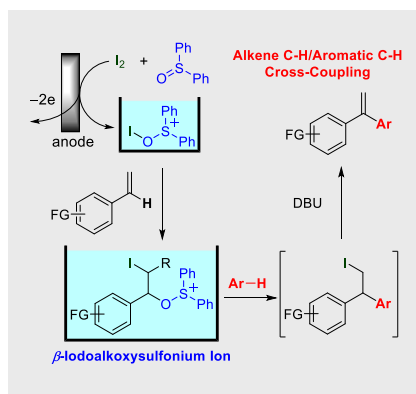
Keywords: C–H/C–H cross-coupling • benzyl cations • C–H functionalization • electrochemistry • oxidation

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Entry for the Table of Contents

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A three-step transformation consisting of (1) addition of electrochemically generated iodosulfonium ions to vinylarenes, (2) nucleophilic substitution by subsequently added aromatic compounds, and (3) elimination of HI with a base enabled metal- and oxidant-free alkenyl C–H/aromatic C–H cross-coupling. The present one-pot transformation provides an easy access to various 1,1-diarylethylenes.



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