

Synthesis of Stilbene and Distyrylbenzene Derivatives through Rhodium-Catalyzed *Ortho*-Olefination and Decarboxylation of Benzoic Acids

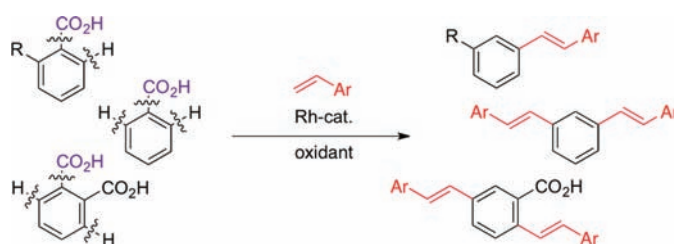
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ABSTRACT



Ortho-substituted benzoic acids efficiently undergo precisely ordered *ortho*-olefination/decarboxylation upon treatment with styrenes in the presence of a rhodium catalyst and silver salt oxidant to afford the corresponding *meta*-substituted stilbene derivatives. The selective syntheses of 1,3- and 1,4-distyrylbenzenes have also been realized through the reactions of simple benzoic acid and phthalic acid, respectively, with styrene under similar conditions.

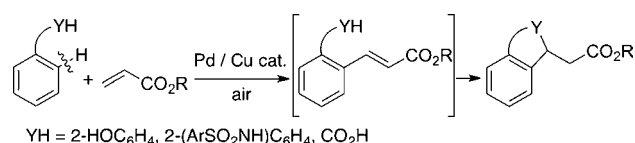
Since stilbene and distyrylbenzene structures are found in various natural products¹ and organic materials,² the development of methods for their effective construction has attracted considerable attention in organic synthesis. The conventional syntheses of such derivatives through transition-metal-catalyzed cross-coupling and Mizoroki–Heck reactions or Wittig-type reactions usually need complicated multisteps forming halogen- and phosphorus-containing byproducts.

(1) For recent examples, see: (a) Hua, X.; Fu, Y.-J.; Zu, Y.-G.; Wu, N.; Kong, Y.; Li, J.; Peng, X.; Efferth, T. *J. Pharm. Biomed. Anal.* **2010**, *52*, 273. (b) Kong, Y.; Fu, Y.-J.; Zu, Y.-G.; Liu, W.; Wang, W.; Hua, X.; Yang, M. *Food Chem.* **2010**, *117*, 152. (c) Vitrac, X.; Bornet, A.; Vanderlinde, R.; Valls, J.; Richard, T.; Delaunay, J.-C.; Méryllon, J.-M.; Teissédre, P.-L. *J. Agric. Food Chem.* **2005**, *53*, 5664. (d) Syah, Y. M.; Aminah, N. S.; Hakim, E. H.; Aimi, N.; Kitajima, M.; Takayama, H.; Achmad, S. A. *Phytochemistry* **2003**, *63*, 913.

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On the other hand, direct olefination reactions on aromatic rings involving regioselective C–H bond cleavage have been developed to provide more simple synthetic routes for vinylarene derivatives.³ As early examples, we reported that 2-phenylphenols (Scheme 1, YH = 2-HOC₆H₄),⁴ *N*-(aryl-

Scheme 1

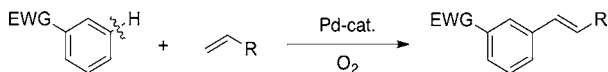


sulfonyl)-2-phenylanilines (YH = 2-(ArSO₂NH)C₆H₄),⁵ and benzoic acids (YH = CO₂H)⁵ undergo regioselective olefination at the *ortho*-position of YH upon treatment with an alkene such as acrylate ester and an appropriate oxidant under

palladium/copper catalysis. In each case, subsequent nucleophilic cyclization takes place to afford the corresponding heterocyclic product. Later, acetanilides,⁶ benzylamines,⁷ phenylacetic acids,⁸ 1-phenylprazoles,⁹ and 2-phenylethanol¹⁰ have also been shown to undergo direct *ortho*-olefination under palladium or rhodium catalysis.

Recently, Yu and co-workers reported the novel palladium-catalyzed *meta*-olefination of arenes possessing an electron-withdrawing group (Scheme 2).¹¹ In this case, however,

Scheme 2



considerable amounts of *para*-olefinated byproduct are also formed.

(3) For selected reviews concerning C–H bond functionalization, see: (a) Satoh, T.; Miura, M. *Chem.–Eur. J.* **2010**, *16*, 11212. (b) Satoh, T.; Miura, M. *Synthesis* **2010**, 3395. (c) Karimi, B.; Behzadnia, H.; Elhamifar, D.; Akhavan, P. F.; Esfahani, F. K. *Synthesis* **2010**, 1399. (d) Colby, D. A.; Bergman, R. G.; Ellman, J. A. *Chem. Rev.* **2010**, *110*, 624. (e) Sun, C.-L.; Li, B.-J.; Shi, Z.-J. *Chem. Commun.* **2010**, *46*, 677. (f) Ackermann, L.; Vicente, R.; Kapdi, A. R. *Angew. Chem., Int. Ed.* **2009**, *48*, 9792. (g) Chen, X.; Engle, K. M.; Wang, D.-H.; Yu, J.-Q. *Angew. Chem., Int. Ed.* **2009**, *48*, 5094. (h) Daugulis, O.; Do, H.-Q.; Shabashov, D. *Acc. Chem. Res.* **2009**, *42*, 1074. (i) McGlacken, G. P.; Bateman, L. M. *Chem. Soc. Rev.* **2009**, *38*, 2447. (j) Kakiuchi, F.; Kochi, T. *Synthesis* **2008**, 3013. (k) Lewis, J. C.; Bergman, R. G.; Ellman, J. A. *Acc. Chem. Res.* **2008**, *41*, 1013. (l) Ferreira, E. M.; Zhang, H.; Stoltz, B. M. *Tetrahedron* **2008**, *64*, 5987. (m) Park, Y. J.; Park, J.-W.; Jun, C.-H. *Acc. Chem. Res.* **2008**, *41*, 222. (n) Beccalli, E. M.; Broggini, G.; Martinelli, M.; Sottocornola, S. *Chem. Rev.* **2007**, *107*, 5318. (o) Herrerias, C. I.; Yao, X.; Li, Z.; Li, C.-J. *Chem. Rev.* **2007**, *107*, 2546. (p) Alberico, D.; Scott, M. E.; Lautens, M. *Chem. Rev.* **2007**, *107*, 174. (q) Godula, K.; Sames, D. *Science* **2006**, *312*, 67. (r) Conley, B. L.; Tenn, W. J., III; Young, K. J. H.; Ganesh, S. K.; Meier, S. K.; Ziatdinov, V. R.; Mironov, O.; Oxgaard, J.; Gonzales, J.; Goddard, W. A., III; Periana, R. A. *J. Mol. Catal. A: Chem.* **2006**, *251*, 8. (s) Kakiuchi, F.; Chatani, N. *Adv. Synth. Catal.* **2003**, *345*, 1077. (t) Ritleng, V.; Sirlin, C.; Pfeffer, M. *Chem. Rev.* **2002**, *102*, 1731. (u) Kakiuchi, F.; Murai, S. *Acc. Chem. Res.* **2002**, *35*, 826. (v) Dyker, G. *Angew. Chem., Int. Ed.* **1999**, *38*, 1698. (w) Kakiuchi, F.; Murai, S. *Top. Organomet. Chem.* **1999**, *3*, 47. (x) Shilov, A. E.; Shul'pin, G. B. *Chem. Rev.* **1997**, *97*, 2879.

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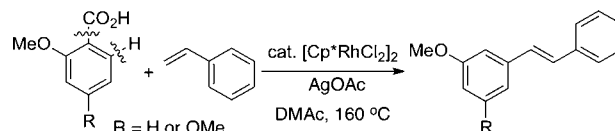
(9) Umeda, N.; Hirano, K.; Satoh, T.; Miura, M. *J. Org. Chem.* **2009**, *74*, 7094.

(10) Lu, Y.; Wang, D.-H.; Engle, K. M.; Yu, J.-Q. *J. Am. Chem. Soc.* **2010**, *132*, 5916.

(11) Zhang, Y.-H.; Shi, B.-F.; Yu, J.-Q. *J. Am. Chem. Soc.* **2009**, *131*, 5072.

In the course of our further study of the direct olefination of benzoic acids,¹² we unexpectedly observed that *meta*-methoxystilbenes were exclusively produced upon treatment of 2-methoxybenzoic acids with styrene under rhodium catalysis, accompanied by decarboxylation^{13–15} (Scheme 3).

Scheme 3



Note that methoxy-substituted stilbenes are of considerable interest due to their unique biological¹⁶ and photophysical and -chemical properties.¹⁷ The procedure was also found to be applicable to the syntheses of 1,3- and 1,4-distyrylbenzenes as well as a range of *meta*-substituted stilbenes. Expectedly, some distyrylbenzenes obtained have been found to show solid-state fluorescence. These new findings are described herein.

(12) Compared with benzoic acids, heteroarene carboxylic acids tend to readily lose their carboxyl group. For our recent studies on the decarboxylative arylation and vinylation of heteroarene carboxylic acids, see: (a) Miyasaka, M.; Fukushima, A.; Satoh, T.; Hirano, K.; Miura, M. *Chem.–Eur. J.* **2009**, *15*, 3674. (b) Nakano, M.; Tsurugi, H.; Satoh, T.; Miura, M. *Org. Lett.* **2008**, *10*, 1851. (c) Maehara, A.; Tsurugi, H.; Satoh, T.; Miura, M. *Org. Lett.* **2008**, *10*, 1159. (d) Okazawa, T.; Satoh, T.; Miura, M.; Nomura, M. *J. Am. Chem. Soc.* **2002**, *124*, 5286.

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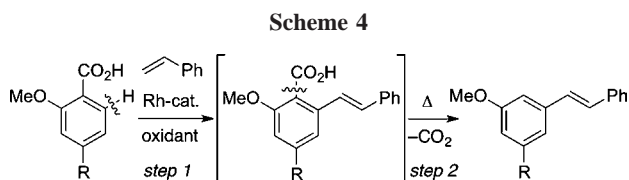
(15) For protodecarboxylation of benzoic acids, see: (a) Cornella, J.; Sanchez, C.; Banawa, D.; Larrosa, I. *Chem. Commun.* **2009**, 7176. (b) Goossen, L. J.; Linder, C.; Rodriguez, N.; Lange, P. P.; Fromm, A. *Chem. Commun.* **2009**, 7173. (c) Goossen, L. J.; Thiel, W. R.; Rodriguez, N.; Linder, C.; Melzer, B. *Adv. Synth. Catal.* **2007**, *349*, 2241. (d) Dickstein, J. S.; Mulrooney, C. A.; O'Brien, E. M.; Morgan, B. J.; Kozlowski, M. C. *Org. Lett.* **2007**, *9*, 2441.

(16) For recent example, see: (a) Mazué, F.; Colin, D.; Gobbo, J.; Wegner, M.; Rescifina, A.; Spatafora, C.; Fasseur, D.; Delmas, D.; Meunier, P.; Tringali, C.; Latruffe, N. *Eur. J. Med. Chem.* **2010**, *45*, 2972. (b) Wang, T. T. Y.; Schoene, N. W.; Kim, Y. S.; Mizuno, C. S.; Rimando, A. M. *Mol. Nutr. Food Res.* **2010**, *54*, 335. (c) Weng, C.-J.; Wu, C.-F.; Huang, H.-W.; Wu, C.-H.; Ho, C.-T.; Yen, G.-C. *J. Agric. Food Chem.* **2010**, *58*, 2886. (d) Sarpietro, M. G.; Ottimo, S.; Giuffrida, M. C.; Spatafora, C.; Tringali, C.; Castelli, F. *Int. J. Pharm.* **2010**, *388*, 144.

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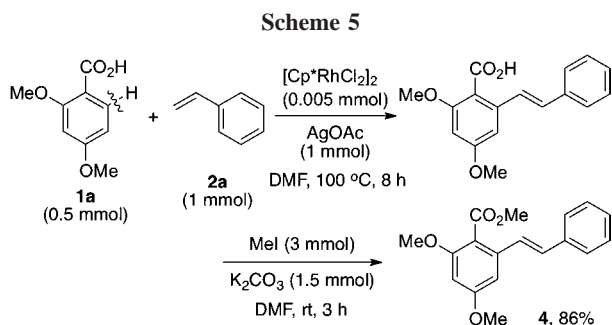
In an initial attempt, the reaction of 2,4-dimethoxybenzoic acid (**1a**) with styrene (**2a**) (2 equiv) was conducted under different conditions (Table S1 in the Supporting Information). Although $[\text{Cp}^*\text{RhCl}_2]_2$ (1 mol %) and AgOAc (2–3 equiv) were found to be suitable choices for the catalyst and oxidant, respectively, the yield of 3,5-dimethoxystilbene (**3a**) was moderate (~50%).

The reaction appears to proceed via (i) the regioselective olefination at the 6-position of 2-methoxybenzoic acids under rhodium catalysis (Scheme 4, step 1) and (ii) the subsequent



decarboxylation of resulting 2-methoxy-6-styrylbenzoic acids (step 2).

It was revealed that a relatively high temperature (160 °C) was needed for not step 1 but step 2. Thus, the olefination of **1a** with **2a** proceeded smoothly even at 100 °C to produce the corresponding 2-stilbenecarboxylic acid, which was subsequently methyl-esterified for quantification (Scheme 5).



To our delight, the stepwise heating, at 120 °C for 8 h (olefination) and then at 160 °C for 4 h (decarboxylation), significantly increased the yield of **3a**. It was further improved by the addition of K_2CO_3 and AgOAc¹⁵ at the second step up to 85% (Table 1, entry 1). Under similar conditions, 2-methoxy- (**1b**), 2-acetyl-amino- (**1c**), 2-fluoro- (**1d**), and 2-phenyl- (**1e**) benzoic acids also underwent decarboxylative olefination to afford the corresponding *meta*-substituted stilbenes **3b–e** (entries 2–5). 2-Styrylnaphthalene (**3f**) could be synthesized effectively from 1-naphthoic acid (**1f**) and **2a** (entry 6). Substituted styrenes **2b–d** also reacted with **1a** to give substituted stilbenes **3g–i** (entries 7–9). It should be noted that the thus obtained 3,5,4'-trimethoxystilbene (**3g**), an analog of naturally occurring resveratrol,¹⁸ is of particular interest for its biological activities including anticancer properties.¹⁶

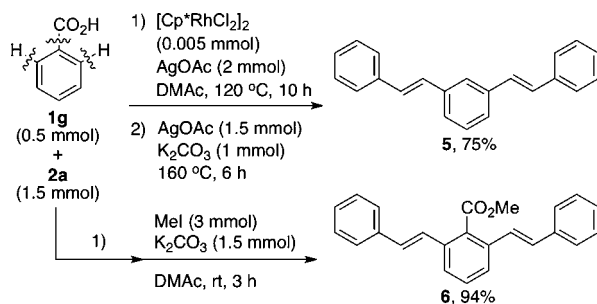
Table 1. Reaction of Benzoic and Naphthoic Acids **1** with Styrenes **2**^a

entry	1	2	product, % yield ^b
1			 3a , 85 (80)
2			 3b , 71 (66)
3			 3c , 74 (66)
4			 3d , 74 (66)
5			 3e , 55 (54)
6			 3f , (70)
7			 3g : R = OMe, 75 (73)
8			3h : R = Bu ^t , 81 (67)
9			3i : R = Cl, 76 (65)

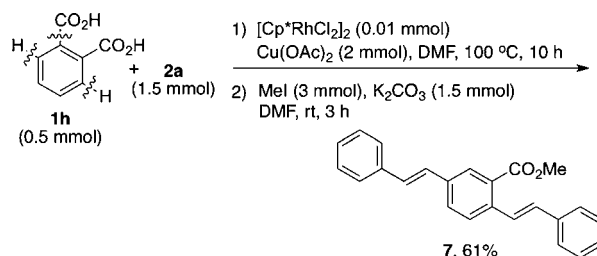
^a Reaction conditions: (1) **1** (0.5 mmol), **2** (1 mmol), $[\text{Cp}^*\text{RhCl}_2]_2$ (0.005 mmol), AgOAc (1–1.5 mmol), DMAc (2.5 mL) at 120 °C under N_2 for 8 h; (2) With the addition of AgOAc (1 mmol) and K_2CO_3 (1 mmol) at 160 °C under N_2 for 4 h. ^b GC yield based on the amount of **1** used. Value in parentheses indicates yield after isolation.

Unsubstituted benzoic acid (**1g**) also underwent the present decarboxylative olefination upon treatment with **2a** under the stepwise heating conditions (Scheme 6). In this case, double olefination took place at the 2- and 6-positions to form 1,3-distyrylbenzene (**5**)¹⁹ selectively. As in the reaction of **1a** (Scheme 5), the intermediary formation of 2,6-distyrylben-

Scheme 6



Scheme 7



zoic acid was confirmed and quantitated after esterification to the methyl ester (**6**) (Scheme 6).

Interestingly, a 1,4-distyrylbenzene derivative, which is of importance in organic materials field,²⁰ could be obtained through the reaction of phthalic acid (**1h**) with **2a** (Scheme 7). Thus, treatment of **1h** with **2a** in the presence of $[\text{Cp}^*\text{RhCl}_2]_2$ and $\text{Cu}(\text{OAc})_2 \cdot 2\text{H}_2\text{O}$, as a catalyst and an oxidant, respectively, in DMF at 100 °C for 10 h followed by methyl esterification resulted in the selective formation of methyl 2,5-distyrylbenzoate (**7**).

Some obtained distyrylbenzenes showed solid-state fluorescence as described above. Notably, **6** exhibited a relatively strong emission compared to a typical emitter, anthracene,

by a factor of 2.2 ($\lambda_{\text{emis}} = 413$ nm, Figure S1 in the Supporting Information).

In summary, we have succeeded in preparing a series of *meta*-substituted stilbenes as well as 1,3- and 1,4-distyrylbenzene derivatives from readily available benzoic acids and styrenes through precisely ordered *ortho*-olefination/decarboxylation under rhodium catalysis. In these reactions, the carboxyl function acts as a unique, removable directing group. Work is underway toward further development of relevant reactions around the key functional group.

Acknowledgment. This work was partly supported by Grants-in-Aid from the Ministry of Education, Culture, Sports, Science and Technology, Japan.

Supporting Information Available: Additional results, standard experimental procedure, and characterization data of products. This material is available free of charge via the Internet at <http://pubs.acs.org>.

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