Facile Synthesis of 2,3-Dihydrobenzofuran-3-ylacetic Acids by Novel Electrochemical Sequential Aryl Radical Cyclization–Carboxylation of 2-Allyloxybromobenzenes Using Methyl 4-*tert*-Butylbenzoate as an Electron-Transfer Mediator

Hisanori Senboku,* Jun-ya Michinishi, Shoji Hara

Laboratory of Organic Reaction, Division of Chemical Process Engineering, Graduate School of Engineering, Hokkaido University, Sapporo, Hokkaido 060-8628, Japan
Fax +81(11)7066555; E-mail: senboku@eng.hokudai.ac.jp
Received 29 March 2011

Abstract: Facile synthesis of 2,3-dihydrobenzofuran-3-ylacetic acids and related analogues was successfully carried out by a novel electrochemical aryl radical generation and its 5-*exo* cyclization followed by a carboxylation sequence of 2-allyloxybromobenzenes by using methyl 4-*tert*-butylbenzoate as an electron-transfer mediator.

Key words: electrochemical reduction, radical cyclization, fixation of carbon dioxide, radical-anion tandem reaction, 2,3-dihydrobenzofuran-3-ylacetic acids

Electroreductive generation of carbon radicals from organic halides is one of the useful and eco-friendly alternatives to conventional radical generation using toxic organotin reagents such as tributyltin hydride. Successful electroreductive generation of carbon-centered radicals without Bu_3SnH has been carried out by direct,[1,2] metal-complex-catalyzed,[3–6] or mediated[7–9] electrochemical reduction of organic halides and diazonium salts, and its application to radical cyclization reactions has also been performed to synthesize carbo- and heterocycles. On the other hand, the termination step of these radical reactions under electroreductive conditions usually proceeds by the reaction of anion species, which are produced by further one-electron reduction of the resulting cyclized radical. However, most of the termination steps in electroreductive radical reactions are merely protonation of the resulting anion, yielding protonated cyclic compounds, and little attention has been given to the use of the resulting anion for further reactions, especially carbon–carbon bond-forming reactions. While this sequential radical generation–cyclization can be achieved by the use of samarium(II) iodide, a representative one-electron reductant, followed by terminal anion reaction with electrophiles such as CO_2,[10,11] to the best of our knowledge, Ni-catalyzed tandem cyclization–carboxylation of unsaturated haloaryl ethers is the only example in electroreductive 'radical-type' cyclization followed by C–C bond formation with CO_2.[12] During the course of our studies in electroorganic synthesis,[13] we recently succeeded in selective generation of aryl radicals from 2-allyloxybromobenzenes by electrochemical reduction using methyl 4-*tert*-butylbenzoate as an electron-transfer mediator. We also found that thus-generated aryl radicals were applicable to radical cyclization, and after further one-electron reduction of the resulting cyclized radical, the resulting anion efficiently captured carbon dioxide to yield 3,4-dihydrobenzofuran-3-ylacetic acids in high selectivities and good yields. Herein, we report a facile synthesis of 3,4-dihydrobenzofuran-3-ylacetic acids and related analogues by novel electrochemical sequential aryl radical cyclization–carboxylation (ERCC) of 2-allyloxybromobenzenes using methyl 4-*tert*-butylbenzoate as an electron-transfer mediator.

The results of screening of an additive as an electron-transfer mediator for ERCC of 2-allyloxybromobenzene (**1a**) are summarized in Table 1. Electrolysis of **1a** was carried out using a test tube-like undivided cell equipped with a Pt plate cathode (2×2 cm^2) and an Mg rod anode (6 mm ϕ) in a DMF solution containing 0.1 M Bu_4NBF_4 as a supporting electrolyte in the presence of CO_2 at 0 °C.[14] When 3 F/mol of electricity was passed by 10 mA/cm^2 constant current without any additives, expected ERCC product **2a** was obtained as a mixture of directly carboxylated benzoic acid **3a** in moderate yield and low selectivity (Table 1, entry 1). We next tried phenanthrene[8] and 9,9-diethylfluorene,[9] which were reported to be effective for generation of aryl radicals from aryl halides, as additives. However, in both cases, carboxylic acids were obtained as a complex mixture, probably due to direct electrochemical carboxylation of additives (Table 1, entries 2 and 3). After several attempts, methyl benzoate was found to be favorable to yield ERCC product **2a** (Table 1, entry 4). Although the yield and selectivity were acceptable in this case, other carboxylic acids were also detected in its ^{1}H NMR as impurities, probably due to direct carboxylation of methyl benzoate. Finally, we found that methyl 4-*tert*-butylbenzoate (**4**) was effective, and expected **2a** was obtained in 54% isolated yield with high selectivity and no contaminants (Table 1, entry 5). We next investigated reaction conditions, including equivalents of mediator, current density, and electricity, using **1a** as a substrate, and the results are also summarized in Table 1. In contrast to the electrolysis at 20 mA/cm^2 with 3 F/mol of electricity in entry 6 (Table 1), electrolyses at 10 mA/

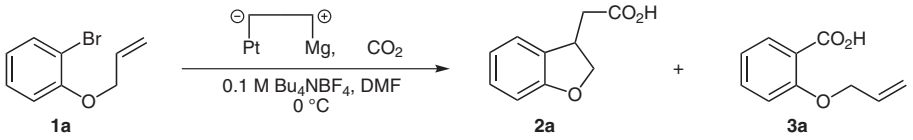
SYNLETT 2011, No. 11, pp 1567–1572
Advanced online publication: 15.06.2011
DOI: 10.1055/s-0030-1260794; Art ID: U02911ST

cm^2 and 50 mA/cm^2 current densities resulted in slight decreases of the yields of **2a** (Table 1, entries 5 and 7). Electron-transfer mediator **4**, methyl 4-*tert*-butylbenzoate, was able to be reduced to 0.5 equivalents at a current density of 20 mA/cm^2 without any decreases in yield or selectivity of **2a** (Table 1, entry 8). However, further reducing of the electron-transfer mediator resulted in lower product selectivities of **2a** toward **3a** with almost same product yields. Finally, when 7 F/mol of electricity was passed in a constant current electrolysis of **1a** at 20 mA/cm^2 in the presence of CO_2 and 0.5 equivalents of mediator **4**, conversion reached 85%, and the desired radical cyclization–carboxylation product, 2,3-dihydrobenzofuran-3-ylacetic acid (**2a**), was obtained in 82% yield with high selectively (Table 1, entry 9). No more electricity with a stable constant current, however, could not be supplied, probably due to precipitation of magnesium salts of both the products and produced oxalic acid, which could be observed as white precipitates at the cathode surface. It is noteworthy that a mediator **4** could be recovered quantitatively, and after purification by column chromatography, the recovered mediator **4** was reusable for the present reaction.

Synthesis of substituted dihydrobenzofuranylacetic acids **2** from bromobenzenes **1** having methyl groups on the allyloxy group as substrates was next investigated, and the results are summarized in Table 2. The present ERCC was found to be also applicable to the synthesis of substituted benzofuranylacetic acids **2**. From 2-allyloxybromobenzenes **1b** and **1c** having methyl groups at their allylic position, the corresponding aryl radicals could also be generated with high selectivity, and efficiently underwent sequential radical cyclization–carboxylation through one-electron reduction to yield the corresponding 2,3-dihydrobenzofuran-3-ylacetic acids **2b** and **2c** in 85% and 79% yields, respectively (Table 2, entries 1 and 2). The product **2b** was obtained as a 9:1 mixture of diastereomers. Recrystallization from hexane gave the major product as a single isomer, whose stereochemistry analyzed by ^{1}H NMR NOESY revealed it to be a *trans* isomer of 2,3-dihydro-2-methylbenzofuran-3-ylacetic acid (*trans*-**2b**). In a similar manner, the present ERCC reaction of bromide **1d** having a methyl group at the internal carbon of the C–C double bond provided the corresponding dihydrobenzofuranylacetic acid **2d** in 78% yield (Table 2, entry 3). In all cases, aryl radical cyclization of ERCC proceeded *exo*-selectively, and no *endo*-cyclization products were observed. On the other hand, methyl groups at the terminal carbon of the C–C double bond in the allyloxy group in **1** strongly affected the yields of ERCC product **2**. When crotyloxybromobenzene (**1e**) was subjected to the ERCC reaction, desired product **2e** was selectively obtained in only 48% yield along with 3-ethyl-2,3-dihydrobenzofuran (**5**) and a formylated, instead of carboxylated, product **6** in 15% and 4% ^{1}H NMR yields, respectively (Table 2, entry 4 and Figure 1). Similar results were obtained when prenyloxy derivative **1f** having two methyl groups at the terminal carbon was used as a substrate. Desired ERCC product **2f** was selectively obtained in only 33% yield along with 3-isopropyl-2,3-dihydrobenzofuran (**7**) and a formylated **8** in 25% and 3% ^{1}H NMR yields, respectively (Table 2, entry 7 and Figure 1). In contrast to the results for **1e**, the yield of the desired product **2f** became lower than that of **2e**, and the yield of protonated product **7** increased.

These results would be due to the relative stability of anion intermediates **A**, **B**, and **C**, which were generated by

Table 1 Screening of Reaction Conditions in ERCC of **1a**



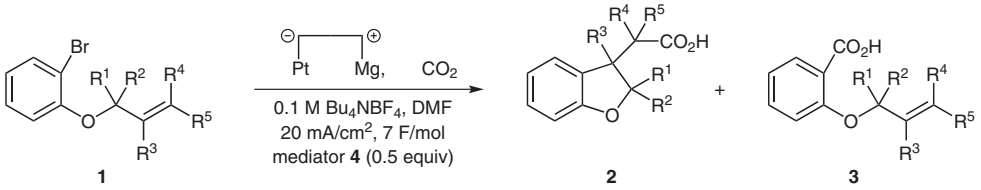
Entry	Additive (equiv)	Current density [mA/cm^2]	Electricity [F/mol]	Yield (%)[a] and ratio (**2a**/**3a**)[b]	Conversion (%)[b]
1	none	10	3	56[b] (62:38)	75
2	phenanthrene (2)	10	3	a complex mixture	44
3	9,9-diethylfluorene (2)	10	3	a complex mixture	69
4	methyl benzoate (2)	10	3	71[b] (97:3)	73
5	methyl 4-*tert*-butylbenzoate (2)	10	3	54 (98:2)	64
6	methyl 4-*tert*-butylbenzoate (2)	20	3	59 (98:2)	68
7	methyl 4-*tert*-butylbenzoate (2)	50	3	55 (98:2)	62
8	methyl 4-*tert*-butylbenzoate (0.5)	20	3	59 (99:1)	69
9	methyl 4-*tert*-butylbenzoate (0.5)	20	7	82 (98:2)	85

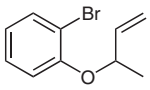
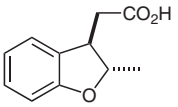
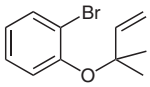
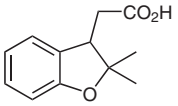
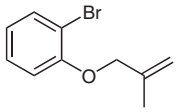
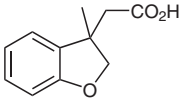
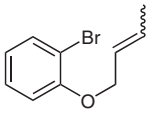
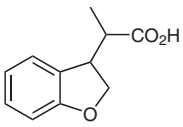
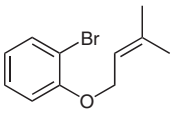
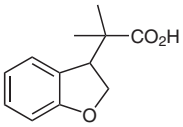
[a] Isolated yield.
[b] Determined by ^{1}H NMR.

one-electron reduction of the radicals produced by aryl radical cyclization (Figure 1). Anion **A**, generated from **1a** by aryl radical cyclization followed by one-electron reduction of the resulting cyclized radical, is a primary anion, which is sufficiently stable to react with carbon dioxide selectively yielding **2a** as a sole cyclized product. On the other hand, anions **B** and **C**, generated in a similar manner from **1e** and **1f**, respectively, are secondary and tertiary anions, which are relatively unstable compared with primary anion **A**. Therefore, side reactions, such as proton abstraction from tetrabutylammonium cation and reaction with DMF, used as a solvent, would also take place competitively to provide protonation products **5** and **7** and formylation products **6** and **8**, respectively. Basicity of tertiary anion **C** would be, in general, stronger than that of secondary anion **B**, resulting in the increase in yield of protonated product **7** in contrast to that of **5**. On the other hand, nucleophilicity of tertiary anion **C** would be lower than that of secondary anion **B** because of its bulkiness, resulting in the decrease in yield of carboxylated product **2f** compared to that of **2e**.

To improve the yield and selectivity of the ERCC product **2e**, the reaction of **1e** was carried out at lower temperatures, and the results are also summarized in Table 2 (entries 5 and 6). When the ERCC reaction was performed at –20 °C, the yield of protonated product **5** decreased, and

Table 2 Synthesis of 2,3-Dihydrobenzohuran-3-ylacetic Acids **2** by the Present ERCC



Entry	Substrate **1**	Product **2**	Temp (°C)	Yield (%)[a] and ratio (**2**/**3**)[b]	Conversion (%)[b]
1[c]	 **1b**	 **2b** (dr = 9:1)[b,d]	0	85 [90] (97:3)	94
2[e]	 **1c**	 **2c**	0	80 [89] (98:2)	90
3	 **1d**	 **2d**	0	78 [92] (98:2)	85
4[f]	 **1e**	 **2e**[j]	0	48 [56] (>99:1)	86
5[g]			–20	49 [79] (>99:1)	71
6[h,i]			–40	31 [86] (>99:1)	36
7[k,l]	 **1f**	 **2f**	0	33 [45] (>99:1)	73

[a] Isolated yields. The yields based on reacted **1** are shown in brackets.
[b] Determined by ^{1}H NMR.
[c] 10 F/mol of electricity was passed.
[d] Diastereomer ratio is shown as dr. Major isomer is shown.
[e] 9.2 F/mol of electricity was passed.
[f] Compounds **5** and **6** were also obtained in 15% and 4% ^{1}H NMR yields, respectively.
[g] Compounds **5** and **6** were also obtained in 10% and 3% ^{1}H NMR yields, respectively.
[h] 3.2 F/mol of electricity was passed.
[i] Compounds **5** and **6** were also obtained in 3% and 1% ^{1}H NMR yields, respectively.
[j] Compound **2e** consists of a 6:4 mixture of diastereomers.
[k] 6.2 F/mol of electricity was passed.
[l] Compounds **7** and **8** were also obtained in 25% and 3% ^{1}H NMR yields, respectively.

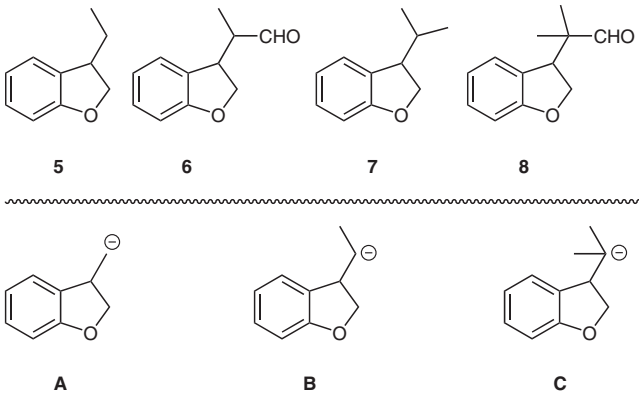


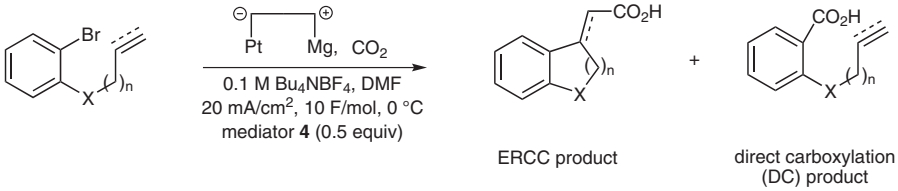
Figure 1 Byproducts in ERCC of **1e** and **1f** and anion intermediate

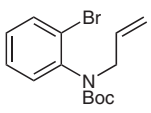
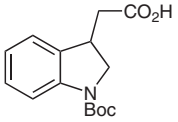
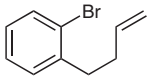
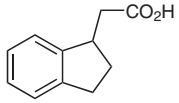
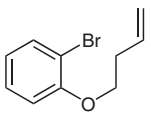
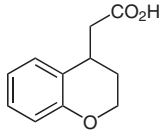
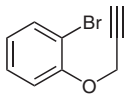
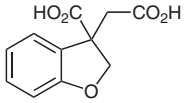
the yield of **2e** based on reacted **1e** increased (Table 2, entry 5) in contrast to the results at 0 °C (Table 2, entry 4). It was found that lowering the temperature to –40 °C was effective for improving the yield and selectivity of ERCC product **2e**. Although only 3.2 F/mol of electricity could be passed, probably due to passivation by precipitation of magnesium salts on the cathode surface, the yield of **2e** based on reacted **1e** reached 86%, and the yields of **5** and **6** successfully decreased (Table 2, entry 6). These results indicated that reaction temperature strongly affected yields of the ERCC product when a substrate has alkyl substituents at the terminal carbon of the allyloxy group. It is thought that when the reaction using **1e** was carried out at a lower temperature, sufficient stability of anion intermediate **B** to react selectively with carbon dioxide could be achieved, providing **2e** in higher yields. By lowering the reaction temperature, a higher concentration of carbon dioxide than that at 0 °C would be realized, and acceleration of the reaction of anion **B** with carbon dioxide would also be a reasonable explanation to improve the yield and selectivity of the product **2e**.

The present ERCC reaction was also applicable to constructing not only dihydrobenzofurans but also indane, indoline, and chromane skeletons followed by carboxylation, yielding acetic acids substituted by those. The results are summarized in Table 3.

ERCC of bromobenzene **9** having an *N*-allyl group on the phenyl ring provided indolin-3-ylacetic acid (**10**) in 40% yield with high product selectivity (Table 3, entry 1). Car-

Table 3 Synthesis of Acetic Acid Analogues by the Present ERCC



Entry	Substrate	ERCC product	Yield (%)[a] and ratio (ERCC/DC)[b]	Conversion (%)[b]
1[c]	 **9**	 **10**	40 [67] (>99:1)	60
2	 **11**	 **12**	68 [74] (94:6)	92
3	 **13**	 **14**	58 [91] (98:2)	64
4[d]	 **15**	 **16**	60 [85] (>99:1)	71

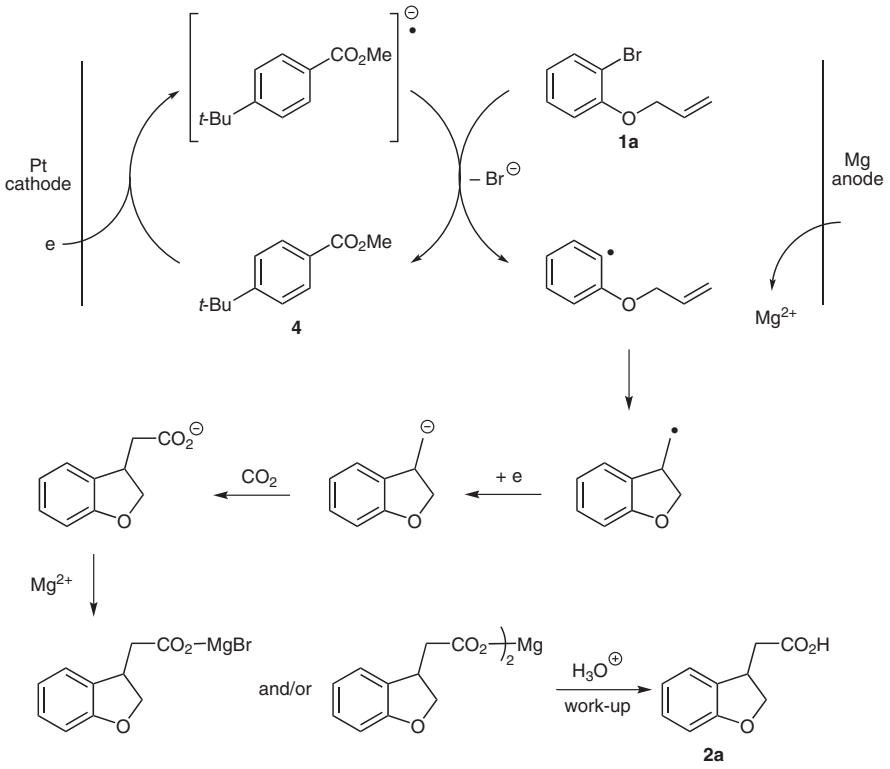
[a] Isolated yields. The yields based on reacted substrate are shown in brackets.
[b] Determined by ^{1}H NMR.
[c] 5.7 F/mol of electricity was passed with 10 mA/cm^2 of current density.
[d] 3 F/mol of electricity was passed.

bocycles could also be constructed by the present ERCC reaction. Thus, indan-1-ylacetic acid (**12**) was successfully obtained in 68% yield from 2-(3-butenyl)bromobenzene (**11**) through cyclopentane formation by aryl radical cyclization followed by a capture of carbon dioxide by the resulting anion species (Table 3, entry 2). Formation of a six-membered ring by ERCC yielding a chromane skeleton was also carried out. When 2-(3-butenyloxy)bromobenzene (**13**) was subjected to ERCC reaction under similar conditions, the expected six-membered ring could be constructed, and the subsequent carboxylation also took place efficiently to yield chroman-4-lyacetic acid (**14**) in 58% yield with high product selectivity (Table 3, entry 3). 2-(2-Propynyloxy)bromobenzene (**15**) was finally tried as a substrate to investigate the possibility of a C–C triple bond as a radical acceptor yielding α,β-unsaturated carboxylic acid. Electrolysis of **15** under similar conditions with 3 F/mol of electricity provided not α,β-unsaturated carboxylic acid but dicarboxylic acid **16** as a sole product (Table 3, entry 4). The formation of dicarboxylic acid **16** would be a result of overcarboxylation of early-produced α,β-unsaturated carboxylic acid at the β-position. A similar β-position-selective carboxylation of ethyl cinnamate by using Mg metal yielding diethyl 2-phenylsuccinate after esterification has recently been reported.[15]

From the viewpoint of reaction pathways, cyclic voltammetry (CV) of **1a** and **4** was carried out.[14] In CV of methyl 4-*tert*-butylbenzoate (**4**) alone in DMF, a reversible reduction peak appeared at –2.9 V vs. Ag/Ag^+. On the other hand, one irreversible reduction peak was observed at –3.2 V vs. Ag/Ag^+ in CV of 2-allyloxybromobenzene (**1a**) in DMF. When CV of **4** was carried out in the presence of 0.5 equivalents of **1a**, an increase in reduction peak current and a decrease in oxidation peak current of **4** were observed. Moreover, when an additional 0.5 equivalents of **1a** (totally a stoichiometric amount for **4**) was added to the electrolyte solution, CV of **4** resulted in a further increase in reduction peak current and a disappearance of the oxidation peak of **4**.[14] These results indicate that methyl 4-*tert*-butylbenzoate (**4**) works as an electron-transfer mediator for highly selective generation of aryl radicals from aryl bromides under electroreductive conditions.

Probable reaction pathways of the present ERCC reaction are shown in Scheme 1. A one-electron reduction of aryl halide selectively occurs by a radical anion of mediator **4** to generate aryl radical. In the absence of mediator **4**, two-electron reduction of aryl halide competitively occurs to generate the corresponding aryl anion, providing directly carboxylated benzoic acid. On the other hand, dissolution of an Mg anode as magnesium ion proceeds to result in prevention of any species from oxidizing at the anode.[13,16,17]

In conclusion, facile synthesis of 2,3-dihydrobenzofuran-3-ylacetic acids and related analogues was achieved by a novel electrochemical aryl radical cyclization–carboxylation sequence (ERCC) of 2-allyloxybromobenzenes by using methyl 4-*tert*-butylbenzoate as an electron-transfer mediator. The results indicate that the present ERCC will become a powerful tool for the synthesis of 2,3-dihydrobenzofuran-3-ylacetic acids and related analogues by



Scheme 1 Reaction pathways

using CO_2 without metal reagents, such as Bu_3SnH and SmI_2.

Supporting Information for this article is available online at http://www.thieme-connect.com/ejournals/toc/synlett.

Acknowledgment

H.S. deeply appreciates partial support by Grant-in-Aid for Scientific Research (B: No. 22360327) from the Japan Society for the Promotion of Science.

References and Notes

(1) (a) Grimshaw, J.; Trocha-Grimshaw, J. *Tetrahedron Lett.* **1974**, *15*, 993. (b) Grimshaw, J.; Haslett, R. J.; Trocha-Grimshaw, J. *J. Chem. Soc., Perkin Trans. 1* **1977**, 2448. (c) Grimshaw, J.; Mannus, D. *J. Chem. Soc., Perkin Trans. 1* **1977**, 2456. (d) Grimshaw, J.; Hamilton, R.; Trocha-Grimshaw, J. *J. Chem. Soc., Perkin Trans. 1* **1982**, 229. (e) Donnelly, S.; Grimshaw, J.; Trocha-Grimshaw, J. *J. Chem. Soc., Chem. Commun.* **1994**, 2171.

(2) LeStrat, F.; Murphy, J. A.; Hughes, M. *Org. Lett.* **2002**, *4*, 2735.

(3) (a) Ozaki, S.; Matsushita, H.; Ohmori, H. *J. Chem. Soc., Chem. Commun.* **1992**, 1120. (b) Ozaki, S.; Matsushita, H.; Ohmori, H. *J. Chem. Soc., Perkin Trans. 1* **1993**, 2339. (c) Ozaki, S.; Horiguchi, J.; Matsushita, H.; Ohmori, H. *Tetrahedron Lett.* **1994**, *35*, 725.

(4) Ihara, M.; Katsumata, A.; Setsu, F.; Tokunaga, Y.; Fukumoto, K. *J. Org. Chem.* **1996**, *61*, 677.

(5) (a) Olivero, S.; Clinet, J. C.; Duñach, E. *Tetrahedron Lett.* **1995**, *36*, 4429. (b) Clinet, J. C.; Duñach, E. *J. Organomet. Chem.* **1995**, *503*, C48. (c) Gómez, M.; Muller, G.; Penyella, D.; Rocamora, M.; Duñach, E.; Olivero, S.; Clinet, J. C. *Organometallics* **1997**, *16*, 5900. (d) Olivero, S.; Rolland, J. P.; Dunãch, E. *Organometallics* **1998**, *17*, 3747.

(6) (a) Torii, S.; Inokuchi, T.; Yukawa, T. *J. Org. Chem.* **1985**, *50*, 5875. (b) Inokuchi, T.; Kawafuchi, H.; Aoki, K.; Yoshida, A.; Torii, S. *Bull. Chem. Soc. Jpn.* **1994**, *67*, 595.

(7) Munusamy, R.; Dhathathreyan, K. S.; Balasubramanian, K. K.; Venkatachalam, C. H. *J. Chem. Soc., Perkin Trans. 2* **2001**, 1154.

(8) (a) Kurono, N.; Honda, E.; Komatsu, F.; Orito, K.; Tokuda, M. *Chem. Lett.* **2003**, *32*, 720. (b) Kurono, N.; Honda, E.; Komatsu, F.; Orito, K.; Tokuda, M. *Tetrahedron* **2004**, *60*, 1791.

(9) At the present stage, they concluded that 9,9-diethylfluorene does not work as a 'typical' mediator in their electroreductive radical generation.[9d] See: (a) Nakagawa, Y.; Mitsudo, K.; Tanaka, H. *The 89th Annual Meeting of the Chemical Society of Japan* Funabashi, March 27th–30th, **2009**, Abstr. No. 1F4-34. (b) Mitsudo, K.; Nakagawa, Y.; Tanaka, H. *The 216th Electrochemical Society Meeting* Vienna, Austria, Oct. 4th–9th, **2009**, Abstr. No. 1644. (c) Mizukawa, Z.; Nakagawa, Y.; Mitsudo, K.; Tanaka, H.; *The 77th Annual Meeting of the Electrochemical Society of Japan* Toyama, March 29th–31st, **2010**, Abstr. No. 1J08. (d) Mitsudo, J.; Nakagawa, Y.; Mizukawa, J.; Suga, S.; Akaba, R.; Tanaka, H. *The 217th Electrochemical Society Meeting* Vancouver, BC, Canada, April 25th–30th, **2010**, Abstr. No. 818.

(10) Molander, G. A.; Harris, C. R. *Chem. Rev.* **1996**, *96*, 307.

(11) Representative papers: (a) Curran, D. P.; Totleben, M. J. *J. Am. Chem. Soc.* **1992**, *114*, 6050. (b) Molander, G. A.; McKie, J. A. *J. Org. Chem.* **1992**, *57*, 3132. (c) Nomoto, A.; Koji, Y.; Shiino, G.; Tomisaka, Y.; Mitani, I.; Tatsumi, M.; Ogawa, A. *Tetrahedron Lett.* **2010**, *51*, 6580.

(12) Olivero, S.; Duñach, E. *Eur. J. Org. Chem.* **1999**, 1885.

(13) Recent papers: (a) Senboku, H.; Yamauchi, Y.; Fukuhara, T.; Hara, S. *Electrochemistry* **2006**, *74*, 612. (b) Senboku, H.; Takahashi, M.; Fukuhara, T.; Hara, S. *Chem. Lett.* **2007**, *36*, 228. (c) Yamauchi, Y.; Fukuhara, T.; Hara, S.; Senboku, H. *Synlett* **2008**, 438. (d) Yamauchi, Y.; Sakai, K.; Fukuhara, T.; Hara, S.; Senboku, H. *Synthesis* **2009**, 3375. (e) Yamauchi, Y.; Hara, S.; Senboku, H. *Tetrahedron* **2010**, *66*, 473. (f) Senboku, H.; Nakahara, K.; Fukuhara, T.; Hara, S. *Tetrahedron Lett.* **2010**, *51*, 435. (g) Ohkoshi, M.; Michinishi, J.; Hara, S.; Senboku, H. *Tetrahedron* **2010**, *66*, 7732.

(14) See Supporting Information.

(15) Maekawa, H.; Murakami, T.; Miyazaki, T.; Nishiguchi, I. *Chem. Lett.* **2011**, *40*, 368; and references cited therein.

(16) (a) Silvestri, G.; Gambino, S.; Filardo, G.; Gulotta, A. *Angew. Chem., Int. Ed. Engl.* **1984**, *23*, 979. (b) Silvestri, G.; Gambino, S.; Filardo, G. *Acta Chem. Scand.* **1991**, *45*, 987.

(17) (a) Sock, O.; Troupel, M.; Périchon, J. *Tetrahedron Lett.* **1985**, *26*, 1509. (b) Chaussard, J.; Folest, J. C.; Nédélec, J. Y.; Périchon, J.; Sibille, S.; Troupel, M. *Synthesis* **1990**, 369.