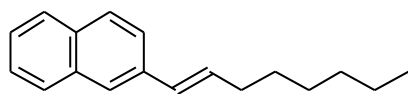


Supporting Information for Single Electron Transfer-Induced Cross-Coupling Reaction of Alkenyl Halides with Aryl Grignard Reagents

Eiji Shirakawa, Ryo Watabe, Takuya Murakami, and Tamio Hayashi

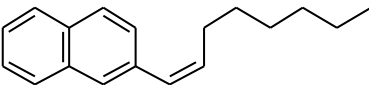
General Remarks. All manipulations of oxygen- and moisture-sensitive materials were conducted with a standard Schlenk technique under a nitrogen atmosphere. Nuclear magnetic resonance spectra were taken on a JEOL JNM LA600 spectrometer (^1H , 594 MHz and ^{13}C , 150 MHz) or a JEOL JNM LA500 spectrometer (^1H , 500 MHz) using tetramethylsilane (^1H , ^{13}C) as an internal standard. GC spectra were taken on Shimadzu GC-2014 equipped with capillary column ID-BPX5 (SGE). GC-MS spectra were taken on Shimadzu GCMS-QP5050A. High-resolution mass spectra were obtained with a Bruker Daltonics microTOF-Q spectrometer (ESI and APCI). Preparative recycling gel permeation chromatography (GPC) was performed with JAI LC-908 equipped with JAIGEL-1H and -2H using chloroform as an eluent. Unless otherwise noted, reagents were commercially available and used without further purification. Toluene and tetrahydrofuran (THF) were purified by passing through an alumina/catalyst column system (GlassContour Co.). Aryl Grignard reagents were prepared in THF from the corresponding aryl bromides and magnesium turning (99.95% purity trace metals basis, Aldrich Co., product number 403148). (*E*)-1-Iodo-1-octene,¹ (*Z*)-1-iodo-1-octene,² (*E*)- β -iodostyrene,² (*Z*)- β -iodostyrene,³ (*E*)-1-iodo-2-(4-methoxyphenyl)ethene,⁴ (*E*)-1-(4-chlorophenyl)-2-iodoethene,⁵ 2-iodo-1-octene,⁶ 2-iodo-3-methyl-2-butene,⁷ 1-iodocyclohexene,⁸ (*E*)-1-bromo-1-octene,⁹ (*E*)- β -bromostyrene,¹⁰ (*E*)-1-chloro-1-octene,¹¹ and (*E*)- β -chlorostyrene¹² were prepared according to literature methods.

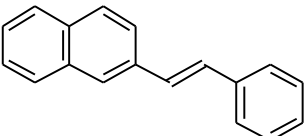
Coupling Reaction of Arylmagnesium Bromides with Alkenyl Halides (Scheme 1): General Procedure. From a THF solution of an arylmagnesium bromide (**1**: 0.30 mmol) placed in a 15 mL pressure-resistant tube (Ace Pressure Tube, Ace Glass 864804), most of the solvent was removed in vacuo at 0.3 mmHg for 20–30 min. To the residue was added successively toluene (2.0 mL), THF (97 μL , 1.2 mmol), and an alkenyl halide (**2**, **2'** or **2''**: 0.20 mmol), and the resulting mixture was stirred at 110 $^\circ\text{C}$ for 24 h. After cooling, the reaction mixture was quenched with a saturated NH_4Cl aqueous solution (2.0 mL) and extracted with Et_2O (10 mL $\times$ 3). The combined organic layer was dried over MgSO_4 , filtered, and concentrated in vacuo. The residue was subjected to silica gel chromatography (hexane, PTLC) to give the corresponding coupling product (**3**).

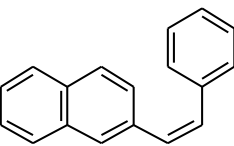


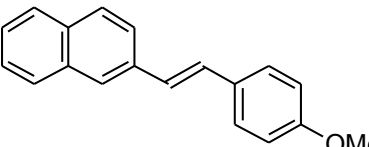
(*E*)-1-(2-Naphthyl)-1-octene ((*E*)-3am).¹³ A yellowish oil. ^1H NMR (594 MHz, CDCl_3) δ 0.90 (t, J = 7.1 Hz, 3 H), 1.27–1.42 (m, 6 H), 1.51 (quint, J = 7.5 Hz, 2 H), 2.26 (q, J = 7.3 Hz, 2 H), 6.36 (dt, J = 15.6, 6.8 Hz, 1 H), 6.54 (d, J = 15.6 Hz, 1 H), 7.40 (t, J = 7.1 Hz, 1 H), 7.44 (t, J = 7.5 Hz, 1 H), 7.58 (dd, J = 8.1, 1.3 Hz, 1 H), 7.67 (s, 1 H),

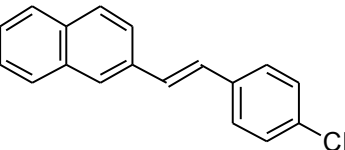
7.76 (d, $J = 8.9$ Hz, 1 H), 7.78 (d, $J = 8.9$ Hz, 2 H).

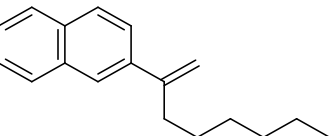
 **(Z)-1-(2-Naphthyl)-1-octene ((Z)-3am).** A yellowish oil. ^1H NMR (594 MHz, CDCl_3) δ 0.87 (t, $J = 7.1$ Hz, 3 H), 1.23–1.38 (m, 6 H), 1.49 (quint, $J = 7.6$ Hz, 2 H), 2.41 (qd, $J = 7.5, 2.0$ Hz, 2 H), 5.75 (dt, $J = 11.6, 7.1$ Hz, 1 H), 6.56 (d, $J = 11.5$ Hz, 1 H), 7.40–7.49 (m, 3 H), 7.72 (s, 1 H), 7.77–7.83 (m, 3 H). ^{13}C NMR (150 MHz, CDCl_3) δ 14.1, 22.6, 28.8, 29.0, 30.0, 31.7, 125.6, 126.0, 127.27, 127.34, 127.51, 127.55, 127.9, 128.7, 132.1, 133.3, 133.7, 135.4. HRMS (ESI) Calcd for $\text{C}_{18}\text{H}_{22}$: $[\text{M}+\text{Na}]^+$, 261.1614. Found: m/z 261.1618.

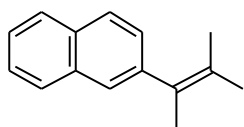
 **(E)-1-(2-Naphthyl)-2-phenylethene ((E)-3an).**¹⁴ A white solid. ^1H NMR (594 MHz, CDCl_3) δ 7.24 (d, $J = 16.3$ Hz, 1 H), 7.28 (t, $J = 7.1$ Hz, 1 H), 7.29 (d, $J = 16.3$ Hz, 1 H), 7.39 (t, $J = 7.8$ Hz, 2 H), 7.45 (t, $J = 6.8$ Hz, 1 H), 7.48 (t, $J = 6.8$ Hz, 1 H), 7.57 (d, $J = 7.5$ Hz, 2 H), 7.75 (dd, $J = 8.9, 2.0$ Hz, 1 H), 7.82 (d, $J = 7.5$ Hz, 1 H), 7.83 (d, $J = 8.1$ Hz, 1 H), 7.84 (d, $J = 8.1$ Hz, 1 H), 7.86 (s, 1 H).

 **(Z)-1-(2-Naphthyl)-2-phenylethene ((Z)-3an).**¹⁴ A white solid. ^1H NMR (594 MHz, CDCl_3) δ 6.69 (d, $J = 12.2$ Hz, 1 H), 6.76 (d, $J = 12.2$ Hz, 1 H), 7.17–7.24 (m, 3 H), 7.28 (dd, $J = 7.5, 1.4$ Hz, 2 H), 7.34 (dd, $J = 8.1, 1.4$ Hz, 1 H), 7.43 (dd, $J = 6.1, 3.4$ Hz, 2 H), 7.65 (d, $J = 8.9$ Hz, 1 H), 7.71 (dd, $J = 6.1, 3.4$ Hz, 1 H), 7.73 (s, 1 H), 7.77 (dd, $J = 6.1, 3.4$ Hz, 1 H).

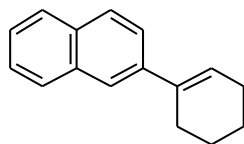
 **(E)-1-(4-Methoxyphenyl)-2-(2-naphthyl)ethene ((E)-3ao).**¹⁵ A white solid. ^1H NMR (594 MHz, CDCl_3) δ 3.85 (s, 3 H), 6.93 (d, $J = 8.1$ Hz, 2 H), 7.15 (d, $J = 16.3$ Hz, 1 H), 7.20 (d, $J = 16.3$ Hz, 1 H), 7.43 (t, $J = 7.5$ Hz, 1 H), 7.47 (t, $J = 7.1$ Hz, 1 H), 7.51 (d, $J = 8.9$ Hz, 2 H), 7.73 (d, $J = 8.9$ Hz, 1 H), 7.78–7.85 (m, 4 H).

 **(E)-1-(4-Chlorophenyl)-2-(2-naphthyl)ethene ((E)-3ap).**¹⁵ A white solid. ^1H NMR (594 MHz, CDCl_3) δ 7.18 (d, $J = 16.3$ Hz, 1 H), 7.25 (d, $J = 16.3$ Hz, 1 H), 7.35 (d, $J = 8.1$ Hz, 2 H), 7.44–7.51 (m, 2 H), 7.49 (d, $J = 8.1$ Hz, 2 H), 7.73 (d, $J = 8.1$ Hz, 1 H), 7.80–7.88 (m, 4 H).

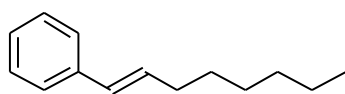
 **2-(2-Naphthyl)-1-octene (3aq).**¹⁶ A colorless oil. ^1H NMR (594 MHz, CDCl_3) δ 0.87 (t, $J = 6.8$ Hz, 3 H), 1.23–1.32 (m, 4 H), 1.36 (quint, $J = 7.2$ Hz, 2 H), 1.49 (quint, $J = 7.5$ Hz, 2 H), 2.61 (t, $J = 7.5$ Hz, 2 H), 5.16 (d, $J = 1.4$ Hz, 1 H), 5.40 (d, $J = 1.4$ Hz, 1 H), 7.44 (t, $J = 6.8$ Hz, 1 H), 7.47 (t, $J = 6.8$ Hz, 1 H), 7.57 (d, $J = 8.9$ Hz, 1 H), 7.77–7.85 (m, 4 H).



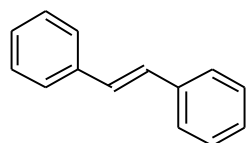
2-Methyl-3-(2-naphthyl)-2-butene (3ar).¹⁷ A colorless oil. ¹H NMR (594 MHz, CDCl₃) δ 1.64 (d, *J* = 1.4 Hz, 3 H), 1.86 (s, 3 H), 2.04 (s, 3 H), 7.29 (dd, *J* = 8.1, 1.4 Hz, 1 H), 7.42 (t, *J* = 6.8 Hz, 1 H), 7.45 (t, *J* = 6.8 Hz, 1 H), 7.56 (s, 1 H), 7.76–7.83 (m, 3 H).



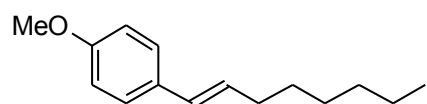
1-(2-Naphthyl)cyclohexene (3as).¹⁸ A white solid. ¹H NMR (594 MHz, CDCl₃) δ 1.71 (quint, *J* = 6.0 Hz, 2 H), 1.83 (quint, *J* = 6.0 Hz, 2 H), 2.24–2.30 (m, 2 H), 2.51–2.57 (m, 2 H), 6.27–6.31 (m, 1 H), 7.41 (t, *J* = 7.2 Hz, 1 H), 7.44 (t, *J* = 7.5 Hz, 1 H), 7.59 (dd, *J* = 8.5, 1.4 Hz, 1 H), 7.76 (d, *J* = 8.9 Hz, 1 H), 7.76 (s, 1 H), 7.79 (d, *J* = 7.4 Hz, 1 H), 7.80 (d, *J* = 8.2 Hz, 1 H).



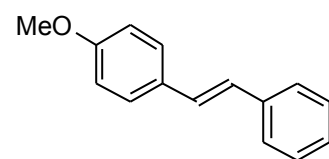
(*E*)-1-Phenyl-1-octene ((*E*)-3bm).¹³ A yellow oil. ¹H NMR (594 MHz, CDCl₃) δ 0.89 (t, *J* = 7.1 Hz, 3 H), 1.24–1.38 (m, 6 H), 1.46 (quint, *J* = 7.3 Hz, 2 H), 2.21 (q, *J* = 6.8 Hz, 2 H), 6.23 (dt, *J* = 15.6, 6.8 Hz, 1 H), 6.37 (d, *J* = 15.6 Hz, 1 H), 7.18 (t, *J* = 7.1 Hz, 1 H), 7.29 (t, *J* = 7.5 Hz, 2 H), 7.34 (d, *J* = 7.5 Hz, 2 H).



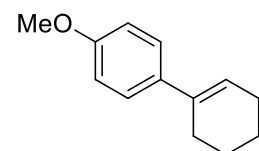
(*E*)-Stilbene ((*E*)-3bn).¹³ A white solid. ¹H NMR (594 MHz, CDCl₃) δ 7.12 (s, 2 H), 7.26 (t, *J* = 7.5 Hz, 2 H), 7.36 (t, *J* = 7.8 Hz, 4 H), 7.52 (d, *J* = 6.8 Hz, 4 H).



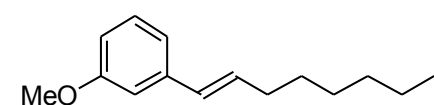
(*E*)-1-(4-Methoxyphenyl)-1-octene ((*E*)-3cm).¹⁹ A colorless oil. ¹H NMR (594 MHz, CDCl₃) δ 0.89 (t, *J* = 6.8 Hz, 3 H), 1.23–1.38 (m, 6 H), 1.45 (quint, *J* = 7.3 Hz, 2 H), 2.18 (q, *J* = 7.2 Hz, 2 H), 3.80 (s, 3 H), 6.08 (dt, *J* = 15.6, 6.8 Hz, 1 H), 6.32 (d, *J* = 15.6 Hz, 1 H), 6.83 (d, *J* = 8.9 Hz, 2 H), 7.27 (d, *J* = 8.9 Hz, 2 H).



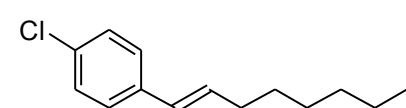
(*E*)-4-Methoxystilbene ((*E*)-3cn).¹³ A white solid. ¹H NMR (594 MHz, CDCl₃) δ 3.84 (s, 3 H), 6.91 (d, *J* = 8.8 Hz, 2 H), 6.98 (d, *J* = 16.3 Hz, 1 H), 7.07 (d, *J* = 16.3 Hz, 1 H), 7.24 (t, *J* = 7.5 Hz, 1 H), 7.35 (t, *J* = 7.8 Hz, 2 H), 7.46 (d, *J* = 8.9 Hz, 2 H), 7.49 (d, *J* = 7.5 Hz, 2 H).



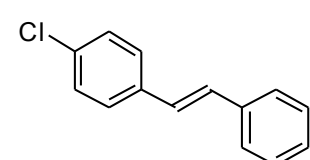
1-(4-Methoxyphenyl)cyclohexene (3cs).²⁰ A yellowish oil. ¹H NMR (594 MHz, CDCl₃) δ 1.65 (quint, *J* = 6.1 Hz, 2 H), 1.77 (quint, *J* = 6.0 Hz, 2 H), 2.16–2.22 (m, 2 H), 2.35–2.42 (m, 2 H), 3.80 (s, 3 H), 6.01–6.05 (m, 1 H), 6.85 (d, *J* = 8.9 Hz, 2 H), 7.32 (d, *J* = 8.9 Hz, 2 H).



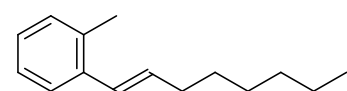
(E)-1-(3-Methoxyphenyl)-1-octene ((E)-3dm). A yellow oil. ^1H NMR (594 MHz, CDCl_3) δ 0.89 (t, $J = 6.8$ Hz, 3 H), 1.24–1.38 (m, 6 H), 1.46 (quint, $J = 7.5$ Hz, 2 H), 2.20 (q, $J = 7.2$ Hz, 2 H), 3.81 (s, 3 H), 6.22 (dt, $J = 15.6, 6.8$ Hz, 1 H), 6.35 (d, $J = 15.6$ Hz, 1 H), 6.75 (dd, $J = 7.8, 2.4$ Hz, 1 H), 6.88 (d, $J = 2.0$ Hz, 1 H), 6.94 (d, $J = 8.1$ Hz, 1 H), 7.20 (t, $J = 7.8$ Hz, 1 H). ^{13}C NMR (150 MHz, CDCl_3) δ 14.1, 22.6, 28.9, 29.3, 31.7, 33.0, 55.2, 111.3, 112.4, 118.6, 129.4, 129.6, 131.6, 139.5, 159.8. HRMS (APCI) Calcd for $\text{C}_{15}\text{H}_{22}\text{O}$: $[\text{M}+\text{H}]^+$, 219.1743. Found: m/z 219.1737.



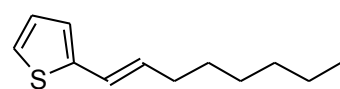
(E)-1-(4-Chlorophenyl)-1-octene ((E)-3em).¹⁹ A yellow oil. ^1H NMR (594 MHz, CDCl_3) δ 0.90 (t, $J = 6.8$ Hz, 3 H), 1.24–1.39 (m, 6 H), 1.46 (quint, $J = 7.5$ Hz, 2 H), 2.20 (q, $J = 7.2$ Hz, 2 H), 6.21 (dt, $J = 15.7, 6.8$ Hz, 1 H), 6.33 (d, $J = 15.6$ Hz, 1 H), 7.22–7.30 (m, 4 H).



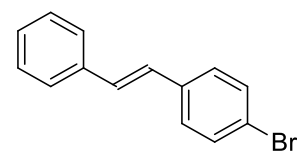
(E)-4-Chlorostilbene ((E)-3en).²¹ A white solid. ^1H NMR (594 MHz, CDCl_3) δ 7.05 (d, $J = 16.3$ Hz, 1 H), 7.09 (d, $J = 16.3$ Hz, 1 H), 7.27 (t, $J = 7.5$ Hz, 1 H), 7.32 (d, $J = 8.2$ Hz, 2 H), 7.36 (t, $J = 7.8$ Hz, 2 H), 7.44 (d, $J = 8.8$ Hz, 2 H), 7.50 (d, $J = 7.5$ Hz, 2 H).



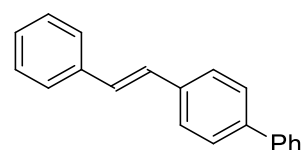
(E)-1-(2-Methylphenyl)-1-octene ((E)-3fm).¹⁹ A yellowish oil. ^1H NMR (594 MHz, CDCl_3) δ 0.90 (t, $J = 6.8$ Hz, 3 H), 1.24–1.40 (m, 6 H), 1.47 (quint, $J = 7.3$ Hz, 2 H), 2.23 (q, $J = 7.3$ Hz, 2 H), 2.33 (s, 3 H), 6.09 (dt, $J = 15.6, 7.1$ Hz, 1 H), 6.56 (d, $J = 15.6$ Hz, 1 H), 7.07–7.18 (m, 3 H), 7.41 (d, $J = 7.5$ Hz, 1 H).



(E)-1-(2-Thienyl)-1-octene ((E)-3gm).¹⁹ A yellow oil. ^1H NMR (594 MHz, CDCl_3) δ 0.89 (t, $J = 6.8$ Hz, 3 H), 1.24–1.37 (m, 6 H), 1.45 (quint, $J = 7.5$ Hz, 2 H), 2.17 (qd, $J = 7.2, 1.4$ Hz, 2 H), 6.07 (dt, $J = 15.6, 7.2$ Hz, 1 H), 6.50 (d, $J = 15.6$ Hz, 1 H), 6.86 (d, $J = 3.4$ Hz, 1 H), 6.93 (dd, $J = 4.6, 3.5$ Hz, 1 H), 7.08 (d, $J = 4.8$ Hz, 1 H).



(E)-4-Bromostilbene ((E)-3bt).²² A white solid. ^1H NMR (594 MHz, CDCl_3) δ 7.04 (d, $J = 16.3$ Hz, 1 H), 7.10 (d, $J = 16.3$ Hz, 1 H), 7.25–7.30 (m, 1 H), 7.34–7.41 (m, 4 H), 7.48 (d, $J = 8.1$ Hz, 2 H), 7.51 (d, $J = 8.2$ Hz, 2 H).



(E)-4-Phenylstilbene ((E)-3'bt).²³ A white solid. ^1H NMR (594 MHz, CDCl_3) δ 7.16 (s, 2 H), 7.27 (t, $J = 7.5$ Hz, 1 H), 7.33–7.40 (m, 3 H), 7.45 (t, $J = 7.5$ Hz, 2 H), 7.54 (d, $J = 7.5$ Hz, 2 H), 7.58–7.65 (m, 6 H).

Coupling Reaction of 4-Methoxyphenylmagnesium Bromide (1c) with 1-Iodocyclohexene (2s) (eqn (1)). The reaction was conducted according to the General Procedure using **1c** (0.88 M, 0.34 mL, 0.30 mmol) and **2s** (41.6 mg, 0.20 mmol). After cooling, the reaction mixture was quenched with a saturated NH₄Cl aqueous solution (2.0 mL), and then diluted with Et₂O (4 mL). An aliquot of the organic layer was subjected to GC analysis to determine the yield and the isomer ratio of (1-cyclohexenyl)toluenes (**4s**) to be 0.04% and *o*:*m*:*p* = 50:12:38. The GC sample was returned to the residue, and the reaction mixture was extracted with Et₂O (10 mL x 3). The combined organic layer was dried over MgSO₄, filtered, and concentrated in vacuo. The residue was subjected to silica gel chromatography (hexane, PTLC) to give the coupling product (**3cs**: 92% yield).

The authentic sample of an isomeric mixture of *o*-, *m*-, and *p*-(1-cyclohexenyl)toluenes (**4s**) for the GC analysis was obtained by the reaction of bis(1-cyclohexenecarbonyl) peroxide in toluene.²⁴ Thus, to toluene (2 mL) was added bis(1-cyclohexenecarbonyl) peroxide (25.0 mg, 0.10 mmol), and the resulting mixture was stirred at 80 °C for 24 h. After cooling, the reaction mixture was quenched with a saturated NH₄Cl aqueous solution (2.0 mL). GC analysis showed that the crude mixture contains *o*-, *m*-, and *p*-(1-cyclohexenyl)toluenes in 50:20:30 ratio. PTLC and GPC purification gave an analytically pure sample (0.9 mg, 3% yield) in 32:28:40 of *o*:*m*:*p* ratio (determined by GC). The isomers were characterized by GC, GC-MS and ¹H NMR (500 MHz: the spectrum is attached). The chemical shifts of *o*- and *p*-(1-cyclohexenyl)toluenes [*o*-(1-cyclohexenyl)toluene: δ 2.28 (s, 3 H), 5.54 (quint, *J* = 1.8 Hz, 1 H); *p*-(1-cyclohexenyl)toluene: δ 2.34 (s, 3 H), 6.09 (quint, *J* = 1.9 Hz, 1 H)] are consistent with the previous report.²⁵ *m*-(1-Cyclohexenyl)toluene, which has never been reported on literature, has peaks at 2.33 ppm (s, 3 H) and 6.08 ppm (quint, *J* = 1.9 Hz, 1 H). On GC, *o*-isomer appears the earliest and *m*-isomer the latest. GC-MS (EI) analysis showed that each isomer has the parent ion peak (*m/z* = 172) and fragment ion peaks (*m/z* = 157, 129, 115, 91).

Coupling Reaction of 2-Naphthylmagnesium Bromide with (E)-1-Iodo-1-octene in the Presence of a Substoichiometric Amount of Lithium Naphthalenide (Scheme 3). From a THF solution of 2-naphthylmagnesium bromide (**1a**: 0.88 M, 0.34 mL, 0.30 mmol), most of the solvent was removed in vacuo at 0.3 mmHg for 30 min. To the residue was added successively toluene (2.0 mL) and (E)-1-iodo-1-octene ((E)-**2m**: 47.6 mg, 0.20 mmol). After dropwise addition of a THF solution of lithium naphthalenide (**5**: 0.50 M, 0.080 mL, 0.040 mmol),²⁶ the resulting mixture was stirred at 80 °C for 12 h. After cooling, the reaction mixture was quenched with a saturated NH₄Cl aqueous solution (2.0 mL) and extracted with Et₂O (10 mL x 3). The combined organic layer was dried over MgSO₄, filtered, and concentrated in vacuo. Nitromethane was added to the crude mixture as an internal standard for ¹H NMR analysis. The conversion of (E)-**2m** and the yield of (E)-**3am** was determined by ¹H NMR analysis.

Competition Reaction between Alkenyl Iodides toward Phenylmagnesium Bromide (Scheme 4). The reaction was conducted according to the General Procedure using **1b** (0.88 M, 0.34 mL, 0.30 mmol), (E)-**2m** (23.8 mg, 0.10 mmol), and (E)-**2n** (23.0 mg, 0.10 mmol) at

110 °C for 6 h. After cooling, the reaction mixture was quenched with a saturated NH₄Cl aqueous solution (2.0 mL) and then diluted with Et₂O (4 mL). Decane was added to the reaction mixture as an internal standard for GC analysis, and an aliquot of the organic layer was subjected to GC analysis. The conversions of (*E*)-**2m** and (*E*)-**2n** and the yields of (*E*)-**3bm** and (*E*)-**3bn** were determined by GC analysis.

Competition Reaction between Arylmagnesium Bromides toward an (*E*)-1-Halo-1-octene (Scheme 5). The reaction was conducted according to the General Procedure using **1h** (0.92 M, 0.16 mL, 0.15 mmol), **1f** (0.93 M, 0.16 mL, 0.15 mmol) and (*E*)-**2m** (47.6 mg, 0.20 mmol) or (*E*)-**2'm** (38.2 mg, 0.20 mmol) at 80 °C for the time specified in Scheme 5. After cooling, the reaction mixture was quenched with a saturated NH₄Cl aqueous solution (2.0 mL) and extracted with Et₂O (10 mL x 3). The combined organic layer was dried over MgSO₄, filtered, and concentrated in vacuo. Nitromethane was added to the crude mixture as an internal standard for ¹H NMR analysis. The conversion of the alkenyl halide and the yields of (*E*)-**3hm** and (*E*)-**3fm** were determined by ¹H NMR analysis.

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NMR Spectra of the Products

