

Photochemical and Electrochemical C–N Borylation of Arylhydrazines

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H.Z. conceived the project and directed the research. L.D. and L.S. performed the experiments. L.D., L.S. and H.Z. discussed the results and wrote the paper. L.D. and L.S. contributed equally to this work.

Supplementary Information

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1. General information

Unless otherwise noted, materials were obtained from commercial suppliers and used without further purification. 4-Acetamido phenylhydrazine hydrochloride was prepared according to the procedures reported in the literature.^[1] CH₃CN was purified by distillation with calcium hydride. The instrument for electrolysis was dual display potentiostat (DJS-292B). The anode electrode and cathode electrode were both Pt plates. The photoreactors used in this research were bought from Wuhan JinBoTianHua Instrument (blue LEDs, λ_{\max} = 450 nm, light intensity = 64.8 mw/cm², 5 W for every light bulb; the distance between the lamp (without filter) and the test tube (borosilicate glass) is around 0.8 cm; every test tube was irradiated by one light bulb from the bottom). Analytical thin-layer chromatography (TLC) was performed using Huang Hai HSGF254 (0.2 mm) precoated plates. The developed chromatogram was analyzed by UV lamp (254 nm). Flash column chromatography was performed with silica gel (200–300 mesh). Gas chromatography (GC) analysis was conducted on a Agilent 7024A instrument equipped with a HP-5 column (30 m × 0.25 mm, Hewlett-Packard) with biphenyl as an internal standard. GCMS analysis was conducted on a Shimadzu GCMS-QP2010 instrument equipped with a Restec-5HT column (30 m × 0.25 mm, Hewlett-Packard). Nuclear magnetic resonance (NMR) spectra were recorded on Bruker Advance III (400 MHz) spectrometers with tetramethylsilane as an internal standard. High resolution mass spectra (HRMS) were obtained from Agilent 6545 Q-TOF LCMS with electrospray ionization (ESI). Chemical shifts for ¹H NMR were expressed in parts per million (ppm) relative to tetramethylsilane (δ 0.00 ppm) or residual peak of CDCl₃ (δ 7.26 ppm). Chemical shifts for ¹³C NMR were expressed in ppm relative to CDCl₃ (δ 77.16 ppm). Data was reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, dd = doublet of doublets, t = triplet, m = multiplet), coupling constant (Hz), and integration.

2. Photochemical and electrochemical C–N borylation of arylhydrazines



entry	scale	Photochemical	Electrochemical
1	0.2 or 0.3 mmol	77% (isolated)	58% (isolated)
2	1.0 mmol	71% (isolated)	50% (isolated)
3	7.0 mmol	60% (isolated)	56% (isolated)

General procedure for photochemical C–N borylation of arylhydrazines: In an oven-dried 50 mL tube equipped with a magnetic stirring bar, arylhydrazine hydrochloride (0.2 mmol), B₂pin₂ (1.0 mmol, 259.1 mg), NaHCO₃ (0.5 mmol, 42.0 mg), Eosin B (3 mol%, 4.1 mg) and CH₃CN (1.0 mL) were added. The reaction mixture was stirred at room temperature under air atmosphere and irradiated by 5 W blue LED for 10 h. When the reaction was completed, the mixture was diluted with brine and extracted with ethyl acetate three times. The combined organic layers were dried over anhydrous Na₂SO₄. The solvent was removed and the pure product was obtained by column chromatography on silica gel using petroleum ether and ethyl acetate as the eluent.

The detailed procedure for the synthesis of 2a (1.0 mmol scale): In an oven-dried 50 mL tube equipped with a magnetic stirring bar, **1a** (1.0 mmol, 150.6 mg), B₂pin₂ (5 mmol, 1295.5 mg), NaHCO₃ (2.5 mmol, 210.0 mg), Eosin B (3 mol%, 20.8 mg) and CH₃CN (5.0 mL) were added. The reaction mixture was stirred at room temperature under air atmosphere and irradiated by 5 W blue LED for 24 h. When the reaction was completed, the mixture was diluted with brine and extracted with ethyl acetate three times. The combined organic layers were dried over anhydrous Na₂SO₄. The solvent was removed and the yellow oil product was obtained by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 500 : 1) in 71% isolated yield (145.2 mg).

The detailed procedure for the synthesis of 2a (gram scale): In an oven-dried 100 mL round bottom flask equipped with a magnetic stirring bar, **1a** (7.0 mmol, 1.0543 g), B₂pin₂ (35 mmol, 9.0692 g), NaHCO₃ (17.5 mmol, 1.4700 g), Eosin B (3 mol%, 145.6 mg) and CH₃CN (35.0 mL) were added. The reaction mixture was stirred at room temperature under air atmosphere and irradiated by blue light band for 7 days. When the reaction was completed, the mixture was filtered. The solvent was removed and the yellow oil product was obtained by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 500 : 1) in 60% isolated yield (0.8557 g).

General procedure for electrochemical C–N borylation of arylhydrazines: In an oven-dried undivided three-necked flask (25 mL) equipped with a stir bar, arylhydrazine hydrochloride (0.3 mmol), B₂pin₂ (1.5 mmol, 388.6 mg), NaHCO₃ (0.6 mmol, 50.4 mg), Et₃N (0.39 mmol, 39.8 mg), ⁿBu₄NI (0.3 mmol, 113.0 mg) and CH₃CN (8.0 mL) were added. The flask was equipped with two

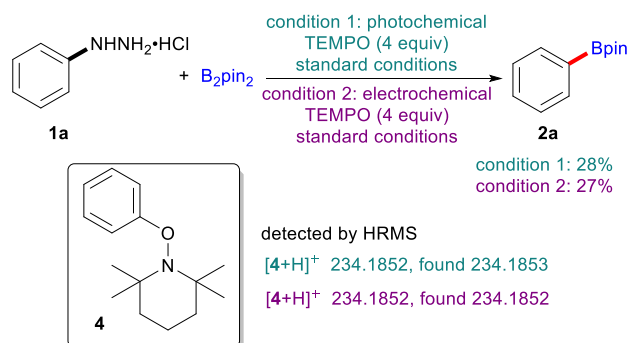
platinum electrodes (15 mm × 15 mm × 0.3 mm) as cathode and anode. The reaction mixture was stirred under air atmosphere for 15 min and electrolyzed at a constant current of 10 mA at room temperature for 4 h. The solvent was removed and the pure product was obtained by column chromatography on silica gel using petroleum ether and ethyl acetate as the eluent.

The detailed procedure for the synthesis of 2a (1.0 mmol scale): In an oven-dried undivided three-necked flask (50 mL) equipped with a stir bar, **1a** (1.0 mmol, 150.6 mg), B₂pin₂ (5.0 mmol, 1295.5 mg), NaHCO₃ (2.0 mmol, 168.0 mg), Et₃N (1.3 mmol, 132.8 mg), ⁿBu₄NI (1.0 mmol, 376.9 mg) and CH₃CN (12.0 mL) were added. The flask was equipped with two platinum electrodes (15 mm × 15 mm × 0.3 mm) as cathode and anode. The reaction mixture was stirred under air atmosphere for 20 min and electrolyzed at a constant current of 10 mA at room temperature for 13 h. The solvent was removed and the yellow oil product was obtained by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 500 : 1) in 50% isolated yield (102.6 mg).

The detailed procedure for the synthesis of 2a (gram scale): In an oven-dried 250 mL beaker equipped with a stir bar, **1a** (7.0 mmol, 1.0543 g), B₂pin₂ (35.0 mmol, 9.0692 g), NaHCO₃ (14.0 mmol, 1.1760 g), Et₃N (9.1 mmol, 0.9301 g), ⁿBu₄NI (7.0 mmol, 2.6384 g) and CH₃CN (186.0 mL) were added. The flask was equipped with two platinum electrodes (15 mm × 15 mm × 0.3 mm) as cathode and anode. The reaction mixture was stirred under air atmosphere for 30 min and electrolyzed at a constant current of 10 mA at room temperature for 4 days. The solvent was removed and the yellow oil product was obtained by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 500 : 1) in 56% isolated yield (0.8002 g).

3. Mechanism studies

Radical trapping experiments

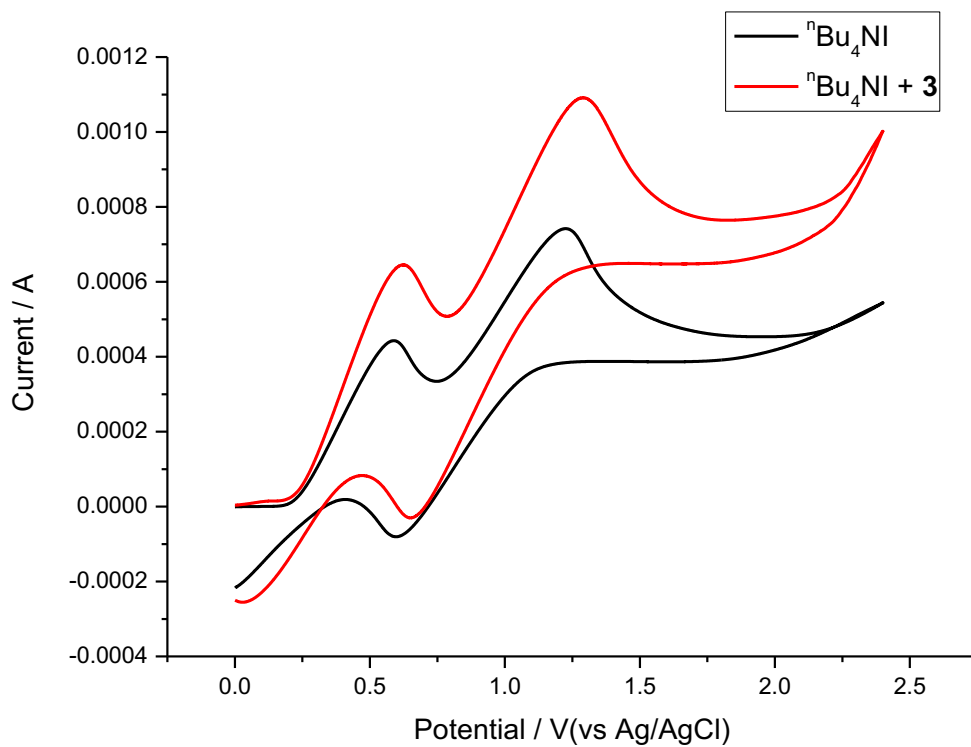


In an oven-dried 50 mL tube equipped with a magnetic stirring bar, **1a** (0.2 mmol, 30.1 mg), B₂pin₂ (1.0 mmol, 259.1 mg), NaHCO₃ (0.5 mmol, 42.0 mg), Eosin B (3 mol%, 4.1 mg), TEMPO (0.8 mmol, 127.5 mg) and CH₃CN (1.0 mL) were added. The reaction mixture was stirred at room temperature under air atmosphere and irradiated by 5 W blue LED for 10 h. The yield of product **2a** was determined by GC and calibrated using biphenyl as internal standard. Meanwhile, the adduct **4** could be detected in the reaction solution by HRMS.

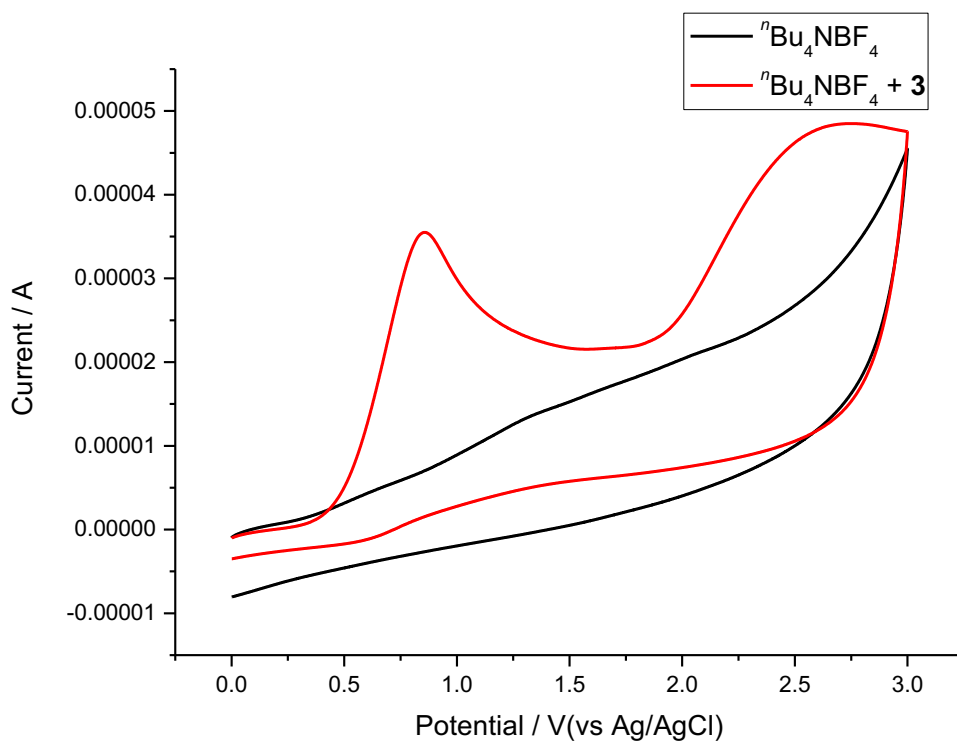
In an oven-dried undivided three-necked flask (25 mL) equipped with a stir bar, **1a** (0.3 mmol, 45.1 mg), B₂pin₂ (1.5 mmol, 388.6 mg), NaHCO₃ (0.6 mmol, 50.4 mg), TEMPO (1.2 mmol, 191.3 mg), Et₃N (0.39 mmol, 39.8 mg), ⁿBu₄NI (0.3 mmol, 113.0 mg) and CH₃CN (8.0 mL) were added. The flask was equipped with two platinum electrodes (15 mm × 15 mm × 0.3 mm) as cathode and anode. The reaction mixture was stirred under air atmosphere for 15 min and electrolyzed at a constant current of 10 mA at room temperature for 4 h. The yield of product **2a** was determined by GC and calibrated using biphenyl as internal standard. Meanwhile, the adduct **4** could be detected in the reaction solution by HRMS.

Cyclic voltammetry experiments

Cyclic voltammetry was performed in a three-electrode cell connected to a Schlenk line at room temperature. The working electrode was a platinum disk electrode, the counter electrode a platinum wire. The reference was an Ag/AgCl electrode submerged in saturated aqueous KCl solution and separated from reaction by a salt bridge. 10.0 mL of CH₃CN containing 0.01 M ⁿBu₄NI or ⁿBu₄NBF₄ were poured into the undivided three-necked flask (25 mL). The scan rate was 0.1 V/s, ranging from 0 V to 3.0 V. The peak potentials vs. Ag/AgCl for used. Two oxidation peaks of ⁿBu₄NI could be observed at 0.59 V and 1.23 V (above, black line) and the oxidation peaks of phenylhydrazine **3** and ⁿBu₄NI mixed solution were 0.62 V and 1.29 V (above, red line). There was no oxidation peak for ⁿBu₄NBF₄ (below, black line) while the oxidation peak of phenylhydrazine **3** was 0.86 V (below, red line).



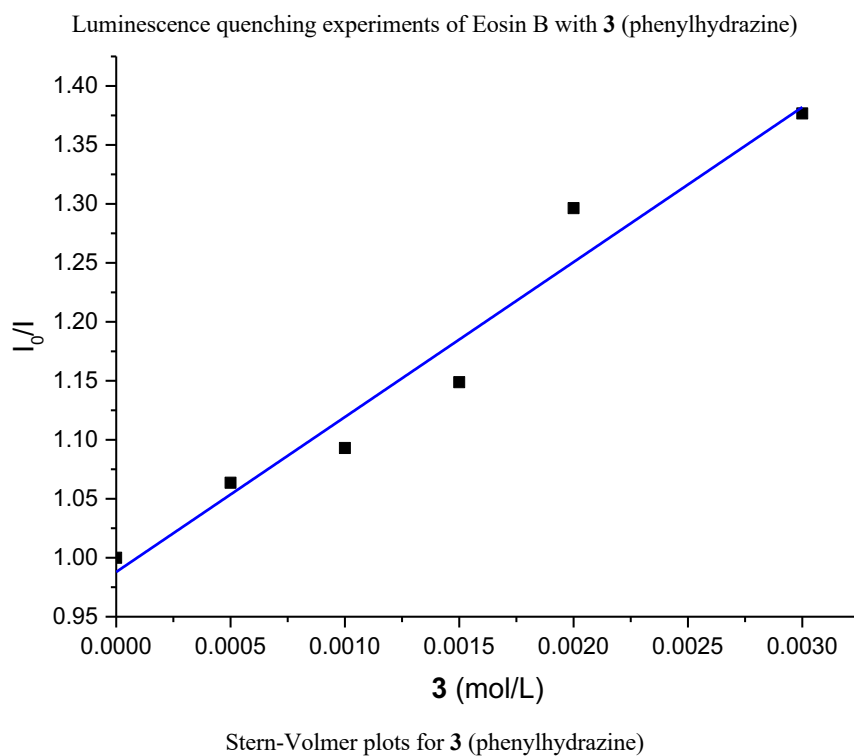
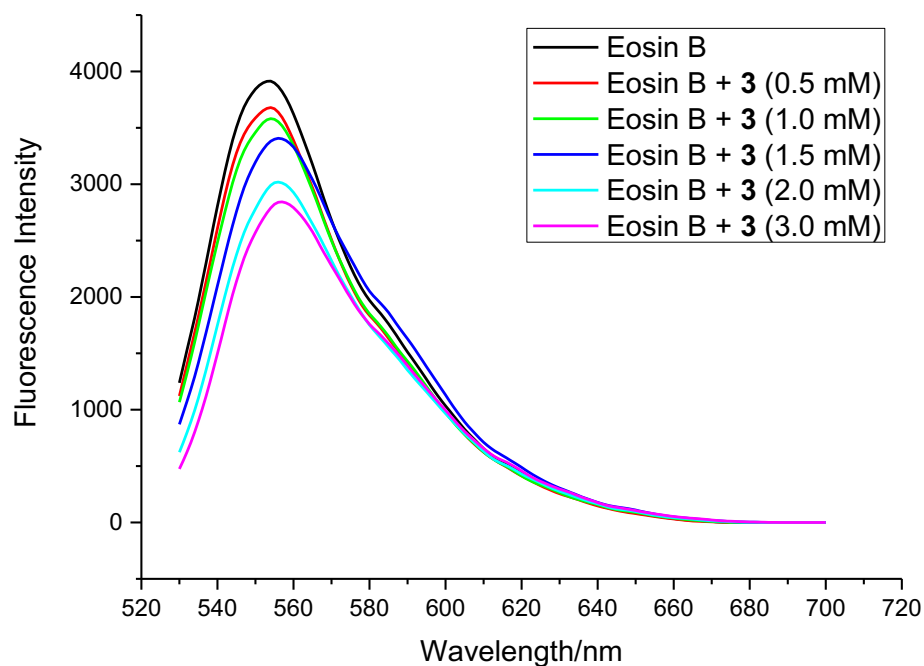
Cyclic voltammograms of $n\text{Bu}_4\text{NI}$ and phenylhydrazine **3** (0.05 mmol) 0-3 V



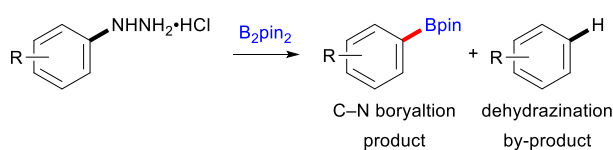
Cyclic voltammograms of $n\text{Bu}_4\text{NBF}_4$ and phenylhydrazine **3** (0.05 mmol) 0-3 V

Luminescence quenching experiments

Emission intensities were recorded using a FL970 Spectrophotometer. First, all Eosin B solutions were excited at 530 nm and the emission intensity at 556 nm was observed. In a typical experiment, the emission spectrum of a 5×10^{-5} M solution of Eosin B and different concentration of phenylhydrazine **3** in anhydrous CH_3CN in 10 mm path length quartz cuvette was collected.



By-product analysis experiments



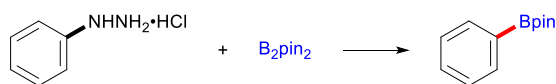
entry	R	Photochemical	Electrochemical
1	2,4-Me ₂	8% by-product (GC)	29% by-product (GC)
2	4- ^t Bu	8% by-product(GC)	24% by-product (GC)

In an oven-dried 50 mL tube equipped with a magnetic stirring bar, **1e** (0.2 mmol, 35.1 mg) or **1h** (0.2 mmol, 42.2 mg), B₂pin₂ (1.0 mmol, 259.1 mg), NaHCO₃ (0.5 mmol, 42.0 mg), Eosin B (3 mol%, 4.1 mg) and CH₃CN (1.0 mL) were added. The reaction mixture was stirred at room temperature under air atmosphere and irradiated by 5 W blue LED for 10 h. The yield of by-product *m*-xylene or *tert*-butylbenzene was determined by GC and calibrated using biphenyl as internal standard.

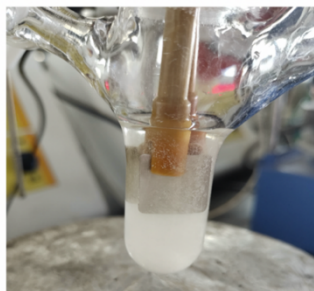
In an oven-dried undivided three-necked flask (25 mL) equipped with a stir bar, **1e** (0.3 mmol, 52.6 mg) or **1h** (0.3 mmol, 63.3 mg), B₂pin₂ (1.5 mmol, 388.6 mg), NaHCO₃ (0.6 mmol, 50.4 mg), Et₃N (0.39 mmol, 39.8 mg), ⁿBu₄NI (0.3 mmol, 113.0 mg) and CH₃CN (8.0 mL) were added. The flask was equipped with two platinum electrodes (15 mm × 15 mm × 0.3 mm) as cathode and anode. The reaction mixture was stirred under air atmosphere for 15 min and electrolyzed at a constant current of 10 mA at room temperature for 4 h. The yield of by-product *m*-xylene or *tert*-butylbenzene was determined by GC and calibrated using biphenyl as internal standard.

In general, higher yields were obtained under photochemical conditions than electrochemical conditions. We have briefly explored the reason for this phenomenon by examining the reaction systems of 2,4-dimethyl phenylhydrazine hydrochloride **1e** (80% yield under photochemical condition; 58% yield under electrochemical condition) and 4-*tert*-butyl phenylhydrazine hydrochloride **1h** (80% yield under photochemical condition; 58% yield under electrochemical condition) under photochemical and electrochemical conditions. For the reactions of **1e**, 8% yield of dehydrazination by-product (1,3-dimethylbenzene) was observed under photochemical condition while 29% yield of 1,3-dimethylbenzene was obtained under electrochemical condition. For the reactions of **1h**, 8% yield of dehydrazination by-product (*tert*-butylbenzene) was observed under photochemical condition while 24% yield of *tert*-butylbenzene was obtained under electrochemical condition. Therefore, we assume that the generally lower yields obtained under electrochemical condition might attribute to the obvious dehydrazination side reaction in electrochemical process.

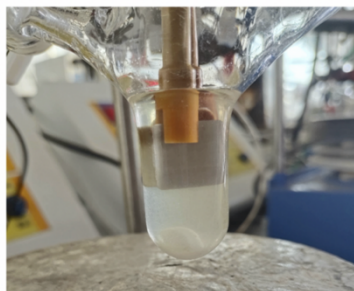
Base effect experiments



entry	NaHCO_3	NEt_3	product
1	✓	✗	88% (GC)(Photochemical)
2	✓	✓	72% (GC)(Photochemical)
3	✗	✓	66% (GC)(Photochemical)
4	✓	✓	68% (GC)(Electrochemical)
5	✗	✓	61% (GC)(Electrochemical)
6	✓	✗	53% (GC)(Electrochemical)



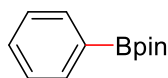
Without Et_3N , after 15 min stirring



Adding Et_3N , after 15 min stirring

NaHCO_3 as the base showed higher efficiency than NaHCO_3 and Et_3N combined in photochemical reaction while the reverse effect was observed in electrochemical reaction. Electrochemical reaction needs higher requirement for the solubility of the substrate. Et_3N could neutralize phenylhydrazine hydrochloride fast, to promote the electron transformation between electrode and phenylhydrazine. Therefore, we conclude that the faster generation of phenylhydrazine from the phenylhydrazine hydrochloride with the aid of Et_3N is beneficial for electrochemical reaction.

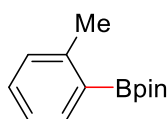
4. Analytical data of products



2a

4,4,5,5-Tetramethyl-2-phenyl-1,3,2-dioxaborolane (2a)^[2]

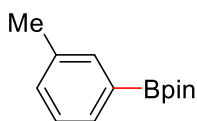
Photochemical condition (31.6 mg, 77%) / Electrochemical condition (35.7 mg, 58%). ¹H NMR (400 MHz, CDCl₃) δ 7.84 (d, *J* = 7.6 Hz, 2H), 7.50-7.46 (m, 1H), 7.41-7.38 (m, 2H), 1.37 (s, 12H); ¹³C NMR (100 MHz, CDCl₃) δ 134.8, 131.3, 127.8, 83.8, 24.9.



2b

4,4,5,5-Tetramethyl-2-(*o*-tolyl)-1,3,2-dioxaborolane (2b)^[2]

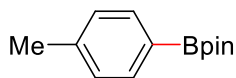
Photochemical condition (28.7 mg, 66%) / Electrochemical condition (29.6 mg, 45%). ¹H NMR (400 MHz, CDCl₃) δ 7.80 (d, *J* = 7.6 Hz, 1H), 7.37-7.33 (m, 1H), 7.21-7.18 (m, 2H), 2.58 (s, 3H), 1.38 (s, 12H); ¹³C NMR (100 MHz, CDCl₃) δ 144.9, 135.9, 130.9, 129.8, 124.8, 83.4, 24.9, 22.3.



2c

4,4,5,5-Tetramethyl-2-(*m*-tolyl)-1,3,2-dioxaborolane (2c)^[3]

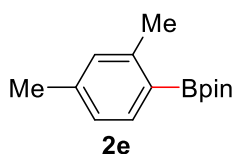
Photochemical condition (22.8 mg, 52%) / Electrochemical condition (28.2 mg, 43%). ¹H NMR (400 MHz, CDCl₃) δ 7.64-7.59 (m, 2H), 7.28-7.25 (m, 2H), 2.35 (s, 3H), 1.34 (s, 12H); ¹³C NMR (100 MHz, CDCl₃) δ 137.2, 135.4, 132.1, 131.8, 127.7, 83.7, 24.9, 21.3.



2d

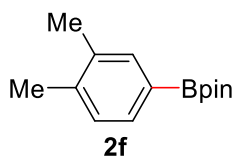
4,4,5,5-Tetramethyl-2-(*p*-tolyl)-1,3,2-dioxaborolane (2d)^[2]

Photochemical condition (31.3 mg, 72%) / Electrochemical condition (37.4 mg, 57%). ¹H NMR (400 MHz, CDCl₃) δ 7.71 (d, *J* = 7.6 Hz, 2H), 7.18 (d, *J* = 7.6 Hz, 2H), 2.36 (s, 3H), 1.33 (s, 12H); ¹³C NMR (100 MHz, CDCl₃) δ 141.5, 134.9, 128.6, 83.7, 24.9, 21.8.



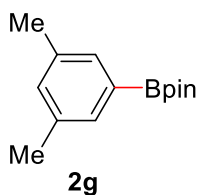
2-(2,4-Dimethylphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (2e)^[3]

Photochemical condition (37.2 mg, 80%) / Electrochemical condition (40.3 mg, 58%). ¹H NMR (400 MHz, CDCl₃) δ 7.67 (d, *J* = 7.6 Hz, 1H), 6.98-6.97 (m, 2H), 2.50 (s, 3H), 2.30 (s, 3H), 1.32 (s, 12H); ¹³C NMR (100 MHz, CDCl₃) δ 145.0, 140.9, 136.2, 130.8, 125.6, 83.3, 24.9, 22.2, 21.6.



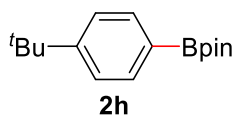
2-(3,4-Dimethylphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (2f)^[4]

Photochemical condition (36.1 mg, 78%) / Electrochemical condition (40.4 mg, 58%). ¹H NMR (400 MHz, CDCl₃) δ 7.58 (s, 1H), 7.55 (d, *J* = 7.6 Hz, 1H), 7.15 (d, *J* = 7.2 Hz, 1H), 2.28 (s, 3H), 2.27 (s, 3H), 1.34 (s, 12H); ¹³C NMR (100 MHz, CDCl₃) δ 140.2, 136.0, 135.9, 132.4, 129.2, 83.6, 24.9, 20.0, 19.5.



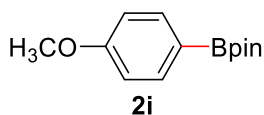
2-(3,5-Dimethylphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (2g)^[3]

Photochemical condition (26.8 mg, 58%) / Electrochemical condition (25.8 mg, 37%). ¹H NMR (400 MHz, CDCl₃) δ 7.44 (s, 2H), 7.10 (s, 1H), 2.32 (s, 6H), 1.34 (s, 12H); ¹³C NMR (100 MHz, CDCl₃) δ 137.2, 133.0, 132.4, 83.7, 24.9, 21.2.



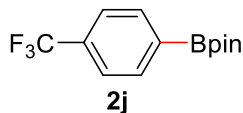
2-(4-(*tert*-Butyl)phenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (2h)^[2]

Photochemical condition (41.5 mg, 80%) / Electrochemical condition (45.3 mg, 58%). ¹H NMR (400 MHz, CDCl₃) δ 7.76 (d, *J* = 8.0 Hz, 2H), 7.41 (d, *J* = 8.4 Hz, 2H), 1.33 (s, 12H), 1.32 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 154.5, 134.7, 124.7, 83.6, 34.9, 31.2, 24.9.



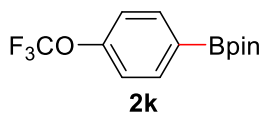
2-(4-Methoxyphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (2i)^[2]

Photochemical condition (24.5 mg, 52%) / Electrochemical condition (43.6 mg, 62%). ¹H NMR (400 MHz, CDCl₃) δ 7.75 (d, *J* = 8.4 Hz, 2H), 6.90 (d, *J* = 8.8 Hz, 2H), 3.83 (s, 3H), 1.33 (s, 12H); ¹³C NMR (100 MHz, CDCl₃) δ 161.5, 136.5, 114.4, 86.4, 54.7, 26.7.



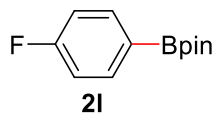
4,4,5,5-Tetramethyl-2-(4-(trifluoromethyl)phenyl)-1,3,2-dioxaborolane (2j)^[2]

Photochemical condition (33.8 mg, 62%) / Electrochemical condition (40.0 mg, 49%). ¹H NMR (400 MHz, CDCl₃) δ 7.93 (d, *J* = 8.0 Hz, 2H), 7.60 (d, *J* = 8.0 Hz, 2H), 1.34 (s, 12H); ¹³C NMR (100 MHz, CDCl₃) δ 135.1, 132.9 (q, *J* = 32.0 Hz), 124.3 (q, *J* = 4.0 Hz), 124.2 (q, *J* = 270.0 Hz), 84.2, 24.7.



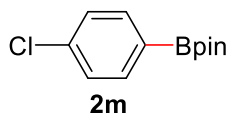
4,4,5,5-Tetramethyl-2-(4-(trifluoromethoxy)phenyl)-1,3,2-dioxaborolane (2k)^[2]

Photochemical condition (18.6 mg, 32%) / Electrochemical condition (34.4 mg, 40%). ¹H NMR (400 MHz, CDCl₃) δ 7.84 (d, *J* = 8.0 Hz, 2H), 7.20 (d, *J* = 8.0 Hz, 2H), 1.34 (s, 12H); ¹³C NMR (100 MHz, CDCl₃) δ 151.7, 136.5, 120.5 (q, *J* = 256.0 Hz), 119.9, 84.1, 24.8.



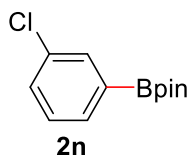
2-(4-Fluorophenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (2l)^[2]

Photochemical condition (9.8 mg, 22%) / Electrochemical condition (16.1 mg, 24%). ¹H NMR (400 MHz, CDCl₃) δ 7.81-7.78 (m, 2H), 7.07-7.02 (m, 2H), 1.34 (s, 12H); ¹³C NMR (100 MHz, CDCl₃) δ 165.0 (d, *J* = 249.0 Hz), 137.0 (d, *J* = 8.0 Hz), 114.9 (d, *J* = 20.0 Hz), 83.9, 24.9.



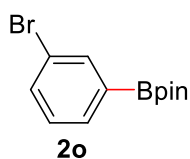
2-(4-Chlorophenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (2m)^[2]

Photochemical condition (17.7 mg, 37%) / Electrochemical condition (20.1 mg, 28%). ¹H NMR (400 MHz, CDCl₃) δ 7.73 (d, *J* = 8.4 Hz, 2H), 7.34 (d, *J* = 8.4 Hz, 2H), 1.34 (s, 12H); ¹³C NMR (100 MHz, CDCl₃) δ 137.6, 136.2, 128.0, 84.0, 24.9.



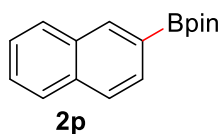
2-(3-Chlorophenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (2n)^[2]

Photochemical condition (35.6 mg, 75%) / Electrochemical condition (35.1 mg, 49%). ¹H NMR (400 MHz, CDCl₃) δ 7.78 (s, 1H), 7.66 (d, *J* = 7.6 Hz, 1H), 7.44-7.41 (m, 1H), 7.32-7.28 (m, 1H), 1.34 (s, 12H); ¹³C NMR (100 MHz, CDCl₃) δ 134.6, 134.1, 132.7, 131.3, 129.2, 84.2, 24.9.



2-(3-Bromophenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (2o)^[5]

Photochemical condition (40.5 mg, 72%) / Electrochemical condition (33.9 mg, 40%). ¹H NMR (400 MHz, CDCl₃) δ 7.94 (s, 1H), 7.71 (d, *J* = 7.2 Hz, 1H), 7.57 (d, *J* = 8.0 Hz, 1H), 7.25-7.21 (m, 1H), 1.34 (s, 12H); ¹³C NMR (100 MHz, CDCl₃) δ 137.5, 134.2, 133.1, 129.5, 122.5, 84.2, 24.9.



4,4,5,5-Tetramethyl-2-(naphthalen-2-yl)-1,3,2-dioxaborolane (2p)^[3]

Photochemical condition (25.5 mg, 50%) / Electrochemical condition (32.1 mg, 42%). ¹H NMR (400 MHz, CDCl₃) δ 8.38 (s, 1H), 7.89-7.81 (m, 4H), 7.52-7.45 (m, 2H), 1.39 (s, 12H); ¹³C NMR (100 MHz, CDCl₃) δ 136.3, 135.1, 132.9, 130.4, 128.7, 127.8, 127.0, 125.9, 84.0, 25.0.

5. Reference

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6. NMR spectra of products

