Supporting Information

Photo-Induced Metal-Catalyst-Free Aromatic Finkelstein Reaction

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1. Supplementary Figures and Tables

Figure S1. LZC-4V photoreactor.

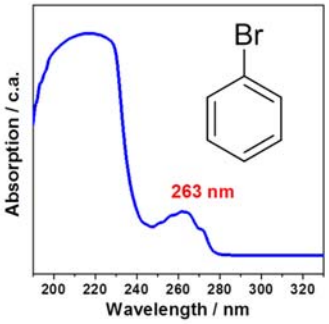


Figure S2. UV-vis absorption spectrum of bromobenzene in acetonitrile.

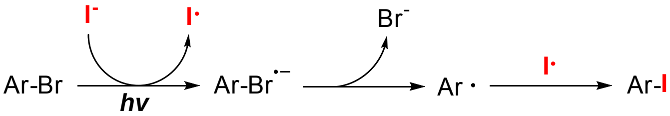
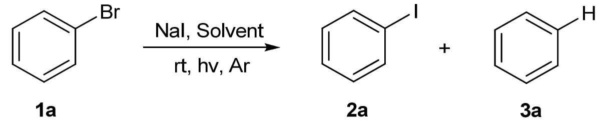


Figure S3. An alternative single electron transfer mechanism for the formation of the aryl radicals in competition with the homolysis route.

Table S1. Survey of various solvents.



Entry	Solvent (0.5 mL)	UV Cut-off Wavelength (nm)	2a GC Yield* (%)	3a GC Yield* (%)
1	acetonitrile	190	69	14
2	ethyl acetate	255	48	6
3	methanol	205	30	13
4	acetone	330	17 (69% recovery)	3
5	water	185	11	trace
6	n-hexane	195	42	7
7	cyclohexane	215	26	5
8	1,4-dioxane	230	4	18
9	tetrahydrofuran	245	6	69
10	hexafluorobenzene	280	1	n.d.

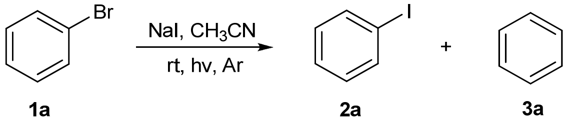
Reaction conditions: performed with **1a** (0.1 mmol) and NaI (0.2 mmol) in argon at room temperature under UV irradiation at an intensity of 4 mWcm^{-2} for 18 h. *Determined by gas chromatography–mass spectrometry (GC-MS) analysis of the crude reaction mixture using chlorobenzene as an internal standard.

Table S2. Effect of wavelength.

Entry	Solvent (0.5 mL)	UV Cut-off Filters (nm)	GC Yield* (%)
1	acetonitrile	none	69
2	acetonitrile	> 225	68
3	acetonitrile	> 250	47
4	acetonitrile	> 280	25
5	acetonitrile	> 320	19
6	acetonitrile	> 360	trace

Reaction conditions: performed with **1a** (0.1 mmol) and NaI (0.2 mmol) in argon at room temperature under UV irradiation at an intensity of 4 mWcm^{-2} for 18 h. *Determined by gas chromatography–mass spectrometry (GC-MS) analysis of the crude reaction mixture using chlorobenzene as an internal standard.

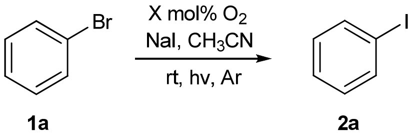
Table S3. Influence of reaction concentration on the selectivity.



Entry	NaI/Substrate (mmol)	MeCN (mL)	Light Intensity ($mWcm^{-2}$)	2a/3a*
1	0.2/0.1	0.25	4	6/1
2	0.2/0.1	0.5	4	5/1
3	0.2/0.1	1.0	4	4.5/1
4	0.2/0.1	4.0	4	2/1

Reaction conditions: performed with **1a** (0.1 mmol) and NaI (0.2 mmol) in argon at room temperature under UV irradiation for 18 h. *Determined by gas chromatography–mass spectrometry (GC-MS) analysis of the crude reaction mixture using chlorobenzene as an internal standard.

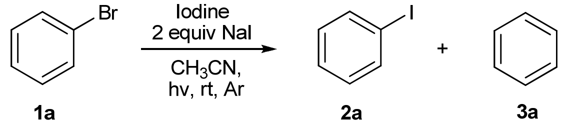
Table S4. Effect of oxygen.



Entry	O_2/Substrate	GC Yield (%)*
1	0 mol%	69
2	0.1 mol %	74
3	0.5 mol %	83
4	1.0 mol %	79
5	5.0 mol %	77
6	10.0 mol %	73
7	Air atmosphere (90 mol %)	61
8	Pure O_2 atmosphere (420 mol %)	25

Reaction conditions: performed with **1a** (0.1 mmol) and NaI (0.2 mmol) in acetonitrile (0.5 mL) at room temperature under UV irradiation at an intensity of 4 $mWcm^{-2}$ for 18 h. *Determined by gas chromatography–mass spectrometry (GC-MS) analysis of the crude reaction mixture using chlorobenzene as an internal standard.

Table S5. Effect of iodine.



Entry	NaI (mmol)	I_2 /Substrate	GC Yield (%)*	2a/3a
1	0.2	none	69	5/1
2	0.2	0.5 mol%	80	7/1
3	0.2	2.5 mol%	87	10/1
4	0.2	10 mol%	91	18/1
5	0.2	25 mol%	87	25/1
6	0.2	100 mol%	82	>30/1
7	0	10 mol%	<5	
8	0	100 mol%	26	
9	0	none	n.d.	**3a:**7%

Reaction conditions: performed with **1a** (0.1 mmol) and NaI (0.2 mmol) in acetonitrile (0.5 mL) and pure argon at room temperature under UV irradiation at an intensity of 4 $mWcm^{-2}$ for 18 h. *Determined by GC-MS analysis of the crude reaction mixture using chlorobenzene as an internal standard.

2. Procedure Details

All commercially available compounds were purchased from Sigma-Aldrich, Strem and Across Company, and used without further purification. All reactions were carried out in airtight quartz tubes filled with ultra-pure argon (>99.999%) unless otherwise noted. NMR spectra were recorded on Varian VNMRS 500 spectrometer with proton resonances at 500 MHz and carbon resonances at 126 MHz, respectively. Chemical shifts are reported in parts per million (ppm). The solvent residual peaks, e.g., of chloroform ($CDCl_3$: δ 7.26 ppm and δ 77.0 ppm), were used as references. Data are reported as following: chemical shift, multiplicity (s = singlet, d = doublet, dd = doublet of doublets, t = triplet, q = quartet, m = multiplet, br = broad signal) and integration. In most cases, the aryl iodide product also contained a small amount of unreacted aryl bromide starting material due to the separation difficulties. Flash column chromatography was performed with E. Merck silica gel 60 (230–400 mesh) or SORBENT silica gel 30-60 µm. Analytical thin layer chromatography (TLC) was performed using Merck silica gel 60 F254 pre-coated plates (0.25 mm). GC-MS was recorded on an Agilent 5975 GC-MS instrument (EI).

A typical experimental procedure as follows: aryl bromide (0.1 mmol), NaI (0.2 mmol) and iodine (0.01 mmol) were added into 0.5 mL of acetonitrile. The airtight quartz tube (10 mL) containing these reactants and solvent was evacuated by four freeze-pump-thaw cycles and back-filled with ultra-purified argon (> 99.999%) prior to use. The reaction was stirred at 20 °C under UV irradiation (254 nm) at an intensity of 4.0 $mWcm^{-2}$ by using a standard LZC-4V photoreactor from Luzchem Company for 36h. When the reaction was complete, the resulting suspension was diluted with sodium thiosulfate aqueous solution (0.01 M, 10 mL) to reduce excess iodine and then extracted with ethyl ether (3 × 15 mL). The combined organic phases were dried by Na_2SO_4, concentrated, and the residue was purified by flash chromatography on silica gel to provide the desired product. For (*E*)-β-bromostyrene substrate, the evaporation temperature during the workup must be kept below 20 °C to avoid the formation of α, β-addition products.

3. Calculation of Apparent Quantum Efficiency (A. Q. E)

In principle, it takes one photon to cleave one C-Br bond to form one target product. The energy of one photon (E_{photon}) with wavelength of λ_{inc} (nm) is calculated using the following equation:

$$E_{\text{photon}} = \frac{hc}{\lambda_{\text{inc}}} = \frac{6.63\times10^{-34}\,\text{Js}\times3\times10^{8}\,\text{ms}^{-1}}{254\times10^{-9}\,\text{m}} = 7.8\times10^{-19}\,\text{J}$$

where h (J·s) is Planck's constant, c ($m\cdot s^{-1}$) is the speed of light and λ_{inc} (m) is the wavelength of the incident light. And the total energy of the incident monochromatic light (E_{total}) is calculated using the following equation:

$$E_{\text{total}} = PSt = 4\times10^{-3}\,\text{Wcm}^{-2}\times0.8\text{cm}^{-2}\times18\times3600\,\text{s} = 2.07\times10^{2}\,\text{J}$$

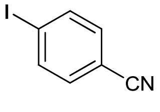
where P ($W\cdot cm^{-2}$) is the power density of the incident light, S (cm^2) is the irradiation area and t (s) is the photoreaction time. The total number of incident photons can be obtained through the following equation:

$$\text{Number of incident photons} = \frac{E_{\text{total}}}{E_{\text{photo}}} = \frac{2.07\times10^{2}\,\text{J}}{7.8\times10^{-19}\,\text{J}} = 2.65\times10^{20} = 0.44\,\text{mmol}$$

Since it is difficult to directly determine the number of reacted electrons via experimental methods, as a result, the apparent quantum yield (A.Q.Y.) is defined as follow:

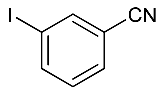
$$\text{A.Q.Y.}(\%) = \frac{\text{Number of produced iodobenzene}}{\text{Number of incident photons}}\times100\% = \frac{0.091\,\text{mmol}}{0.44\,\text{mmol}}\times100\% = 20.7\%$$

4. Analytical Data

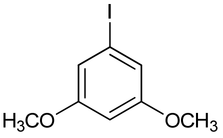


4-Iodo-benzonitrile (2b). Flash chromatography afforded the product as white solid (yield: 93%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.85 (d, *J* = 8.5 Hz, 2H), 7.37 (d, *J* = 8.5 Hz, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 138.5, 133.2, 118.2, 111.8, 100.3. The ^{1}H and ^{13}C data were in agreement with previously published data.[1]

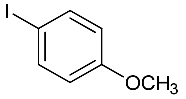
Gram-Scale Synthesis of 2b. 4-bromo-benzonitrile (1.092 g, 6 mmol), NaI (1.8 g, 12 mmol) and I_2 (0.152 g, 0.6 mmol) were added into 30 mL of acetonitrile. The airtight round-bottom quartz flask (75 ml) containing these reactants and solvent was evacuated by four freeze-pump-thaw cycles and back-filled with ultra-purified argon (> 99.999%) prior to use. The reaction was stirred at 20 °C under UV irradiation at an intensity of 4.0 $mWcm^{-2}$ by using a standard LZC-4V photoreactor from Luzchem Company for 72h. After reaction, the resulting suspension was diluted with sodium thiosulfate aqueous solution (0.2 M, 30 mL) to reduce excess iodine and then extracted with ethyl ether (3 × 30 mL). The combined organic phases were dried by Na_2SO_4, concentrated, and the residue was purified by flash chromatography on silica gel. The total amount of product is 1.232 gram with 86% yield (96% purity).



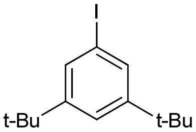
3-Iodo-Benzonitrile (2c). Flash chromatography afforded the product as colorless solid (yield: 76%). ^1H NMR (500 MHz, $CDCl_3$) δ 7.99 (t, *J* = 1.5 Hz, 1H), 7.95 (m, 1H), 7.66-7.60 (m, 1H), 7.22 (t, *J* = 7.9 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 141.9, 140.5, 131.2, 130.5, 117.1, 114.3, 93.9. The ^1H data was in agreement with previously published data.[2]



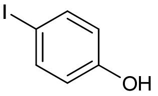
1-Iodo-3, 5-dimethoxybenzene (2d). Flash chromatography afforded the product as white solid (yield: 91%). ^1H NMR (500 MHz, $CDCl_3$) δ 6.86 (d, *J* = 2.2 Hz, 2H), 6.39 (t, *J* = 2.2 Hz, 1H), 3.76 (s, 6H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 161.1, 115.8, 100.7, 94.1, 55.5. The ^1H and ^{13}C data were in agreement with previously published data.[3]



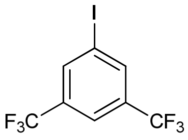
4-Iodoanisole (2e). Flash chromatography afforded the product as off-white solid (yield: 85%). ^1H NMR (500 MHz, $CDCl_3$) δ 7.55 (d, *J* = 9.0 Hz, 2H), 6.67 (d, *J* = 8.9 Hz, 2H), 3.77 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 159.5, 138.2, 116.4, 82.7, 55.3. The ^1H and ^{13}C data were in agreement with previously published data.[4]



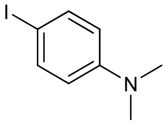
1-Iodo-3,5-ditert-butylbenzene (2f). Flash chromatography afforded the product as colorless solid (yield: 89%). ^1H NMR (500 MHz, $CDCl_3$) δ 7.53 (d, *J* = 1.7 Hz, 2H), 7.36 (t, *J* = 1.6 Hz, 1H), 1.30 (s, 18H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 153.1, 131.8, 121.8, 94.8, 34.9, 31.3. The ^1H and ^{13}C data were in agreement with previously published data.[5]



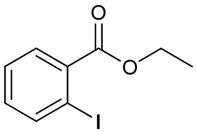
4-Iodophenol (2g). Flash chromatography afforded the product as light yellow solid (yield: 88%). ^1H NMR (500 MHz, $CDCl_3$) δ 7.52 (d, *J* = 8.8 Hz, 2H), 6.63 (d, *J* = 8.8 Hz, 2H), 4.84 (s, br, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 155.4, 138.5, 117.8, 82.7. The ^1H and ^{13}C data were in agreement with previously published data.[6]



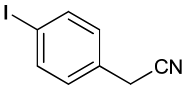
1,3-Bis(trifluoromethyl)-5-iodobenzene (2h). Flash chromatography afforded the product as colorless liquid (yield: 76%). ^{1}H NMR (500 MHz, CDCl$_3$) δ 8.15 (s, 2H), 7.85 (s, 1H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 137.7 (s), 133.0 (q, J = 33.8 Hz), 122.2 (q, J = 273.1 Hz), 121.8-121.7 (m), 93.9 (s). The ^{1}H and ^{13}C data were in agreement with previously published data.[7]



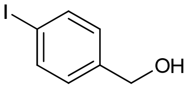
4-Iodo-N,N-dimethylaniline (2i). Flash chromatography afforded the product as white solid (yield: 56%). ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.47 (d, J = 9.1 Hz, 2H), 6.50 (d, J = 9.0 Hz, 2H), 2.92 (s, 6H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 149.9, 137.6, 114.8, 77.2, 40.5. The ^{1}H and ^{13}C data were in agreement with previously published data.[8]



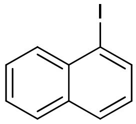
Ethyl 2-iodobenzoate (2j). Flash chromatography afforded the product as yellow liquid (yield: 90%). ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.98 (dd, J = 7.9, 1.0 Hz, 1H), 7.78 (dd, J = 7.8, 1.7 Hz, 1H), 7.40 (td, J = 7.6, 1.1 Hz, 1H), 7.17-7.11 (m, 1H), 4.40 (q, J = 7.1 Hz, 2H), 1.41 (t, J = 7.2 Hz, 3H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 166.6, 141.2, 135.5, 132.5, 130.8, 127.9, 94.0, 61.7, 14.2. The ^{1}H and ^{13}C data were in agreement with previously published data.[9]



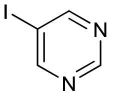
4-Iodophenyl acetonitrile (2k). Flash chromatography afforded the product as white solid (yield: 85%). ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.71 (d, J = 8.3 Hz, 2H), 7.08 (d, J = 8.3 Hz, 2H), 3.70 (s, 2H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 138.3, 129.8, 129.6, 117.3, 93.6, 23.3. The ^{1}H and ^{13}C data were in agreement with previously published data.[10]



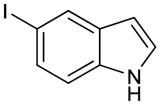
4-Iodobenzyl alcohol (2l). Flash chromatography afforded the product as white solid (yield: 78%). ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.69 (d, J = 8.3 Hz, 2H), 7.12 (d, J = 8.0 Hz, 2H), 4.65 (s, 2H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 140.4, 137.6, 128.8, 93.0, 64.7. The ^{1}H and ^{13}C data were in agreement with previously published data.[11]



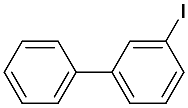
1-Iodo-naphthalene (2m). Flash chromatography afforded the product as deep brown liquid (yield: 82%). ^{1}H NMR (500 MHz, CDCl$_3$) δ 8.11 (d, J = 3.3 Hz, 1H), 8.09 (d, J = 0.9 Hz, 1H), 7.84 (d, J = 8.2 Hz, 1H), 7.78 (d, J = 7.6 Hz, 1H), 7.61-7.56 (m, 1H), 7.54-7.51 (m, 1H), 7.21-7.17 (m, 1H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 137.4, 134.4, 134.2, 132.2, 129.0, 128.6, 127.7, 126.9, 126.7, 99.6. The ^{1}H and ^{13}C data were in agreement with previously published data.[12]



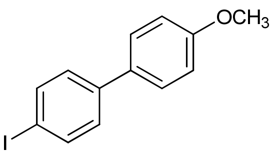
5-Iodopyrimidine (2n). Flash chromatography afforded the product as light yellow solid (yield: 74%). ^{1}H NMR (500 MHz, CDCl$_3$) δ 9.14 (s, 1H), 8.95 (s, 2H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 162.5, 156.6, 93.8. The ^{1}H data were in agreement with previously published data.[13]



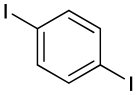
5-Iodoindole (2o). Flash chromatography afforded the product as white solid (yield: 63%). ^{1}H NMR (500 MHz, CDCl$_3$) δ 8.18 (s, br, 1H), 7.99 (s, br, 1H), 7.44 (dd, *J* = 8.5, 1.6 Hz, 1H), 7.24-7.12 (m, 2H), 6.45-6.51 (m, 1H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 134.8, 130.5, 130.3, 129.6, 124.9, 112.9, 102.0, 83.3. The ^{1}H data was in agreement with previously published data.[14] The ^{13}C data was in agreement with previously published data.[15]



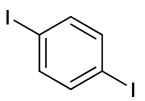
3-Iodobiphenyl (2p). Flash chromatography afforded the product as white solid (yield: 87%). ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.95 (t, *J* = 1.7 Hz, 1H), 7.68 (ddd, *J* = 7.9, 1.5, 1.1 Hz, 1H), 7.58-7.52 (m, 3H), 7.44 (t, *J* = 7.5 Hz, 2H), 7.41-7.34 (m, 1H), 7.17 (t, *J* = 7.8 Hz, 1H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 143.47, 139.64, 136.19, 136.15, 130.40, 128.87, 127.83, 127.11, 126.41, 94.78. The ^{1}H and ^{13}C data were in agreement with previously published data.[16]



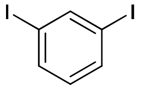
4-Iodo-4’-methoxybiphenyl (2q). Flash chromatography afforded the product as yellow solid (yield: 60%) ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.73 (d, *J* = 8.5 Hz, 2H), 7.49 (d, *J* = 8.8 Hz, 2H), 7.29 (d, *J* = 8.5 Hz, 2H), 6.97 (d, *J* = 8.8 Hz, 2H), 3.85 (s, 3H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 159.5, 140.3, 137.8, 132.5, 128.6, 127.9, 114.3, 92.1, 55.4. The ^{1}H and ^{13}C data were in agreement with previously published data.[17]



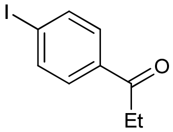
1,4-Diiodobenzene (1-bromo-4-iodobenzene) (2r). Flash chromatography afforded the product as light brown solid (yield: 78%). ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.41 (s, 4H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 139.3, 93.3. The ^{1}H and ^{13}C data were in agreement with previously published data.[18]



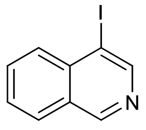
1,4-Diiodobenzene (1,4-dibromobenzene) (2s). Flash chromatography afforded the product as white solid (yield: 62%). ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.41 (s, 4H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 139.3, 93.3. The ^{1}H and ^{13}C data were in agreement with previously published data.[18]



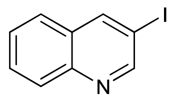
1,3-Diiodobenzene (2t). Flash chromatography afforded the product as white solid (yield: 62%). ^{1}H NMR (500 MHz, CDCl$_3$) δ 8.07 (t, J = 1.6 Hz, 1H), 7.66 (dd, J = 7.9, 1.6 Hz, 2H), 6.83 (t, J = 7.9 Hz, 1H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 145.2, 136.7, 131.6, 94.9. The ^{1}H and ^{13}C data were in agreement with previously published data.[19]



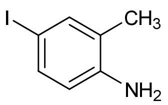
4’-Iodo-propiophenone (2u). Flash chromatography afforded the product as white solid (yield: 78%). ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.82 (d, J = 8.6 Hz, 2H), 7.67 (d, J = 8.6 Hz, 2H), 2.96 (q, J = 7.2 Hz, 2H), 1.22 (t, J = 7.2 Hz, 3H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 200.0, 137.9, 136.2, 129.4, 100.7, 31.7, 8.1. The ^{1}H and ^{13}C data were in agreement with previously published data.[20]



4-Iodoisoquinoline (2v). Flash chromatography afforded the product as light yellow needle solid (yield: 80%). ^{1}H NMR (500 MHz, CDCl$_3$) δ 9.14 (s, 1H), 8.94 (s, 1H), 8.00 (dd, J = 8.5, 0.6 Hz, 1H), 7.90 (d, J = 8.2 Hz, 1H), 7.80 (ddd, J = 8.4, 6.9, 1.2 Hz, 1H), 7.67 (ddd, J = 8.0, 7.0, 1.0 Hz, 1H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 152.6, 151.0, 137.1, 132.0, 130.7, 129.8, 128.3, 128.2, 96.8. The ^{1}H and ^{13}C data were in agreement with previously published data.[21]

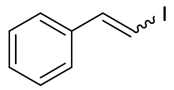


3-Iodoquinoline (2w). Flash chromatography afforded the product as yellow solid (yield: 56%). ^{1}H NMR (500 MHz, CDCl$_3$) δ 9.03 (d, J = 2.0 Hz, 1H), 8.53 (d, J = 1.8 Hz, 1H), 8.06 (d, J = 8.5 Hz, 1H), 7.80-7.66 (m, 2H), 7.60-7.55 (m, H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 155.6, 146.3, 143.7, 130.0, 129.9, 129.5, 127.4, 126.8, 89.8. The ^{1}H and ^{13}C data were in agreement with previously published data.[15]



4-Iodo-2-methylaniline (2x). Flash chromatography afforded the product as light brown solid (yield: 75%). ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.35-7.33 (m, 1H), 7.31-7.27 (m, 1H), 6.45 (d, J = 8.3 Hz, 1H), 3.62 (s, br, 2H), 2.11 (s, 3H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 144.3, 138.7, 135.6, 124.9, 116.9, 79.5, 17.0. The ^{1}H and ^{13}C data were in agreement with previously published data.[22]

1-iodo-2-phenylethene (2y). Flash chromatography afforded the product as yellow liquid (yield: 63%, Z/E = 1.7/1). The E/Z isomer ratio was determined by ^{1}H NMR analysis.



(*E*)-1-iodo-2-phenylethene: ^{1}H NMR (500 MHz; $CDCl_3$): 7.44 (d, J = 15.0 Hz, 1H), 7.38-7.27 (m, 5H), 6.84 (d, J =15.0, 1H). ^{13}C NMR (126 MHz, $CDCl_3$): δ 145.0, 137.7, 128.7, 128.4, 126.0, 76.6. The ^{1}H and ^{13}C data were in agreement with previously published data.[23]

(*Z*)-1-iodo-2-phenylethene: ^{1}H NMR (500 MHz; $CDCl_3$): 7.68-7.60 (m, 2H), 7.39-7.34 (m, 3H), 7.31 (d, J = 7.0 Hz, 1H), 6.58 (d, J = 8.5 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$): δ 138.6, 136.7, 128.4, 128.3, 128.2 (2C), 79.3 ppm. The ^{1}H and ^{13}C data were in agreement with previously published data.[23]

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