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Supporting Information for

Metal-Free Oxidative Cross-Coupling of Diazirines with

Arylboronic Acids

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CONTENTS

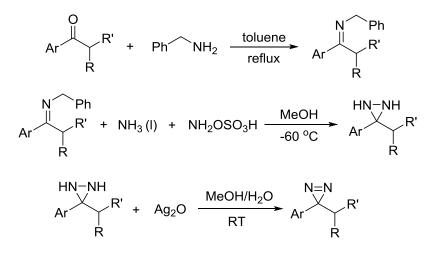
| 1) | General | S2 |
|----|--|--------------|
| 2) | Preparation and spectral data of diazirines | S 2 |
| 3) | Experimental procedure and characterizations | · \$3 |
| 4) | References | S 10 |
| 5) | ¹ H NMR and ¹³ C NMR spectra | S 11 |

1) General

All solvents were distilled prior to use. The solvents for the reaction were distilled to remove water over Na or CaH₂. Column chromatograph was performed on 200-300 mesh silica gal. ¹H NMR and ¹³C NMR spectra were recorded at 300 MHz (or 400 MHz) and 75 MHz (or 100 MHz) with Varian Mercury 300 spectrometer (or Bruker ARX 400 spectrometer) using CDCl₃ (DMSO- d_6) as solvent and tetramethylsilane as the internal standard. IR spectra were recorded with a Thermo Electron Corporation Nicolet AVATAR 300 FT-IR spectrometer. Mass spectra were obtained on Bruker Apex IV FTMS spectrometer.

2) Preparation and spectra data of diazirines

Diazirines used in this investigation were prepared by literature procedure.¹



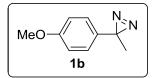
Benzyl amine (100 mmol) and ketone (50 mmol) were mixed in toluene (150 mL). The mixture was refluxed for about 12 hours. Solvent and impurities were removed under vacuum and the benzyl imine was obtained. The benzyl imine was then dissolved in methanol (100 mL). The methanol solution was added to the liquid ammonia (50 mL) and the mixture was stirred at -60 $^{\circ}$ C for 3 hours. A solution of hydroxylamine-*O*-sulfonic acid (75 mmol) in methanol (50 mL) was subsequently added and the mixture was stirred at -60 $^{\circ}$ C for another 2 hours. The reaction mixture was allowed to warm to room temperature to evaporate the excess ammonia. Water was added to the crude residue and the mixture was extracted with diethyl ether (3 x 50 mL). The combined organic phase was concentrated to give a crude oil, which was oxidized with freshly prepared silver oxide in MeOH/H₂O (1:1, 150 mL) at room temperature for 2 hours. The silver salts were filtered and filtrate was extracted with diethyl ether. The combined organic phase was dried and concentrated under reduced pressure to give the crude product, which was purified by flash chromatograph on silica gal. The yields for three steps range from 20% to 30%.

3-Methyl-3-phenyl-3*H***-diazirine 1a**.²



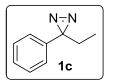
Colorless oil; ¹H NMR (300 MHz, d_6 -DMSO) δ 7.41-7.33 (m, 3H), 6.96-6.93 (m, 2H), 1.50 (s, 3H).

3-(4-Methoxyphenyl)-3-methyl-3*H*-diazirine 1b.²



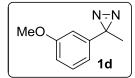
Pale yellow solid; ¹H NMR (300 MHz, CDCl₃) δ 6.85-6.81 (m, 4H), 3.79 (s, 3H), 1.49 (s, 3H).

3-Ethyl-3-phenyl-3*H*-diazirine 1c.²



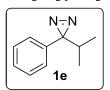
Colorless oil; ¹H NMR (300 MHz, CDCl₃) 7.33-7.25 (m, 3H), 6.96-6.92 (m, 2H), 2.03 (q, J = 7.6 Hz, 2H), 0.84 (t, J = 7.6 Hz, 3H).

3-(3-Methoxyphenyl)-3-methyl-3*H*-diazirine 1d.²



Colorless oil; ¹H NMR (300 MHz, CDCl₃) 7.25-7.21 (m, 1H), 6.84-6.81 (m, 1H), 6.54-6.51 (m, 1H), 6.41-6.40 (m, 1H), 3.78 (s, 3H), 1.50 (s, 3H).

3-Isopropyl-3-phenyl-3*H*-diazirine 1e.²



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Colorless oil; ¹H NMR (300 MHz, CDCl₃) 7.35-7.23 (m, 3H), 7.00-6.97 (m, 2H), 2.97-2.83 (m, 1H), 0.81 (d, J = 6.9 Hz, 6H).

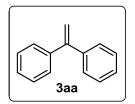
3) Experimental procedure and characterizations

$$Ar \bigvee_{N}^{Me} N + Ar'B(OH)_2 \xrightarrow{BQ, 110 \circ C} Ar \xrightarrow{Ar'} + N_2$$

Arylboronic acid (0.6 mmol), diazirine (1.2 mmol), *p*-benzoquinone (0.72 mmol) and 1, 4-dioxane (0.8 mL) were mixed in a microwave tube. The mixture was stirred at 110 $^{\circ}$ C for 2 hours. Then the crude reaction mixture was filtered through short silica

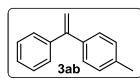
gal column. The solvent was evaporated under reduced pressure to give a crude residue, which was purified by flash chromatograph on silica gal to afford the pure products.

Ethene-1,1-diyldibenzene 3aa.³



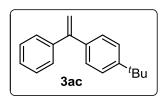
Yield 72%; colorless oil; ¹H NMR (400 MHz, CDCl₃) δ 7.32 -7.29 (m, 10H), 5.44 (s, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 150.1, 141.6, 128.3, 128.2, 127.8, 114.3.

1-Methyl-4-(1-phenylvinyl)benzene 3ab.³



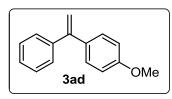
Yield 70%; colorless oil; ¹H NMR (300 MHz, CDCl₃) δ 7.35-7.29 (m, 5H) 7.23 (d, J = 8.1 Hz, 2H), 7.13 (d, J = 7.9 Hz, 2H), 5.41 (dd, J = 8.5, 1.3 Hz, 2H), 2.36 (s, 3H).

1-tert-Butyl-4-(1-phenylvinyl)benzene 3ac.⁴



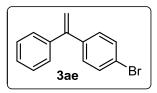
Yield 64%; colorless oil; ¹H NMR (300 MHz, CDCl₃) δ 7.36-7.28 (m, 9H), 5.45 (d, *J* = 1.3 Hz, 1H), 5.40 (d, *J* = 1.3 Hz, 1H), 1.33 (s, 9H).

1-Methoxy-4-(1-phenylvinyl)benzene 3ad.³



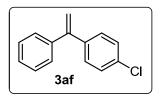
Yield 57%; white Solid; ¹H NMR (300 MHz, CDCl₃) δ 7.34-7.32 (m, 5H), 7.27 (d, *J*=9.2 Hz, 2H), 6.86 (d, *J* = 8.9 Hz, 2H), 5.40 (d, *J* = 1.3 Hz, 1H), 5.35 (d, *J* = 1.3 Hz, 1H), 3.82 (s, 3H).

1-Bromo-4-(1-phenylvinyl)benzene 3ae.⁵



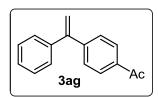
Yield 73%; Colorless oil; ¹H NMR (300 MHz, CDCl₃) δ 7.45 (d, *J* = 8.6 Hz, 2H), 7.36 - 7.29 (m, 5H), 7.20 (d, *J* = 8.5 Hz, 2H), 5.46 (d, *J* = 1.1 Hz, 1H), 5.44 (d, *J* = 1.0 Hz, 1H).

1-Chloro-4-(1-phenylvinyl)benzene 3af.³



Yield 82%; colorless oil; ¹H NMR (300 MHz, CDCl₃) δ 7.32-7.23 (m, 9H), 5.45 (d, J = 1.1 Hz, 1H), 5.43 (d, J = 1.1 Hz, 1H).

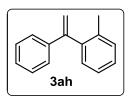
1-(4-(1-Phenylvinyl)phenyl)ethanone 3ag.



Yield 27%; colorless oil; IR (film) 2918, 1683, 1605, 1358, 1267, 959.8, 904.1, 850.1, 778.8, 705.1; ¹H NMR (300 MHz, CDCl₃) δ 7.93 (d, *J* = 8.7 Hz, 2H), 7.43 (d, *J* = 8.7 Hz, 2H), 7.36-7.30 (m, 5H), 5.56 (d, *J* = 1.0 Hz, 1H), 5.55 (d, *J* = 1.0

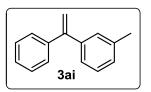
Hz, 1H), 2.61 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 197.7, 149.2, 146.2, 140.7, 136.3, 128.5, 128.3, 116.0, 26.6; HRMS (ESI) *m/e* calcd for C₁₆H₁₅O (M+H)⁺ 223.1117, found 223.1118.

1-Methyl-2-(1-phenylvinyl)benzene 3ah.⁶



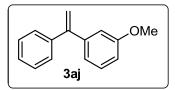
Yield 34%; Colorless oil; ¹H NMR (300 MHz, CDCl₃) δ 7.28-7.19 (m, 9H), 5.77 (d, J = 1.4 Hz, 1H), 5.19 (d, J = 1.4 Hz, 1H), 2.05 (s, 3H).

1-Methyl-3-(1-phenylvinyl)benzene 3ai.⁴



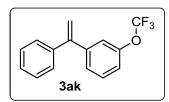
Yield 78%; colorless oil; ¹H NMR (300 MHz, CDCl₃) δ 7.34 - 7.11(m, 9H), 5.43 (s, 2H), 2.33 (s, 3H).

1-Methoxy-3-(1-phenylvinyl)benzene 3aj.³



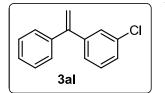
Yield 71%; colorless oil; ¹H NMR (300 MHz, CDCl₃) δ 7.35- 7.26 (m, 5H), 7.24 - 7.21 (m, 1H), 6.94- 6.83 (m, 3H), 5.45 (s, 2H), 3.77 (s, 3H).

1-(1-Phenylvinyl)-3-(trifluoromethoxy)benzene 3ak.⁴



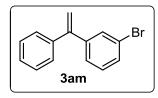
Yield 42%; colorless oil; ¹H NMR (300 MHz, CDCl₃) δ 7.35 -7.19 (m, 9H), 5.48 (d, J = 1.0 Hz, 1H), 5.45 (d, J = 0.9 Hz, 1H).

1-Chloro-3-(1-phenylvinyl)benzene 3al.³



Yield 50%, Colorless oil; ¹H NMR (300 MHz, CDCl₃) δ 7.38-7.25 (m, 9H), 5.52 (d, *J* = 1.0 Hz, 1H), 5.49 (d, *J* = 1.0 Hz, 1H).

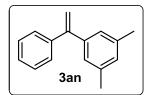
1-Bromo-3-(1-phenylvinyl)benzene 3am.



Yield 17%; colorless oil; IR (film) 3057, 2917, 1554, 1492, 1470, 1074, 904.1, 776.2, 697.2; ¹H NMR (300 MHz, CDCl₃) δ 7.46 -7.39 (m, 2H), 7.31-7.27 (m, 5H), 7.23 -7.13 (m, 2H), 5.45 (d, *J* = 1.0 Hz, 1H), 5.42 (d, *J* = 1.0 Hz, 1H); ¹³C NMR

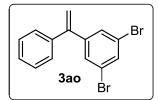
(75 MHz, CDCl₃) δ 148.8, 143.7, 140.7, 131.2, 130.7, 129.7, 128.3, 128.2, 128.0, 126.9, 122.4, 115.3. HRMS (EI) *m/e* calcd for C₁₄H₁₁⁷⁹Br (M) 258.0044, found 258.0048; C₁₄H₁₁⁸¹Br (M) 260.0024, found 260.0028.

1,3-Dimethyl-5-(1-phenylvinyl)benzene 3an.³



Yield 62%; Colorless oil; ¹H NMR (300 MHz, CDCl₃) δ 7.35 -7.30 (m, 5H), 6.95 (s, 3H), 5.41 (s, 2H), 2.29 (s, 6H).

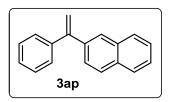
1,3-Dibromo-5-(1-phenylvinyl)benzene 3ao.



Yield 31%; colorless oil; IR (film) 3057, 3023, 1578, 1544, 907.3, 856.7, 777.2, 743.1, 696.0; ¹H NMR (300 MHz, CDCl₃) δ 7.61 (t, *J* = 1.8 Hz, 1H), 7.40 (d, *J* = 1.8 Hz, 2H), 7.36 - 7.25 (m, 5H), 5.51 (d, *J* = 0.8 Hz, 1H), 5.45 (d, *J* = 0.8

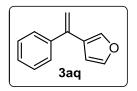
Hz, 1H); ¹³C NMR (75 MHz, CDCl₃) δ 147.7, 145.2, 140.1, 133.1, 130.0, 128.5, 128.3, 128.1, 122.8, 116.3. HRMS (ESI) *m/e* calcd for C₁₄H₁₁Br₂ (M+H)⁺ 336.9222, found 336.9228.

2-(1-Phenylvinyl)naphthalene 3ap.⁷



Yield 66%; white solid; mp: 53-55 °C; ¹H NMR (300 MHz, CDCl₃) δ 7.85-7.78 (m, 4H), 7.50-7.45 (m, 3H), 7.37-7.34 (m, 5H), 5.59 (d, *J* = 1.2 Hz, 1H), 5.55 (d, *J* = 1.2 Hz, 1H).

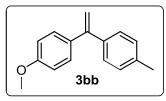
3-(1-Phenylvinyl)furan 3aq.



Yield 58%; colorless oil; IR (film) 3026, 2918, 2843, 1684, 1492, 1161, 1066, 1022, 873.4, 794.8, 775.8, 732.1, 698.6; ¹H NMR (300 MHz, CDCl₃) δ 7.44– 7.40 (m, 3H), 7.38-7.31 (m, 4H), 6.56 -6.52 (m, 1H), 5.44 (d, *J* = 1.2 Hz, 1H), 5.44 (d, *J* = 1.2 Hz,

1H), 5.23 (d, J = 1.2 Hz, 2H), 5.23 (d, J = 1.2 Hz, 1H). ¹³C NMR (75 MHz, CDCl₃) δ 143.2, 143.1, 141.1, 141.0, 140.9, 128.2, 127.9, 126.6, 112.7, 109.4 (d, J = 2.9 Hz, 1C). HRMS (ESI) *m/e* calcd for C₁₂H₁₁O (M+H)⁺ 171.0804, found 171.0805.

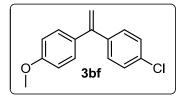
1-Methoxy-4-(1-p-tolylvinyl)benzene 3bb.



Yield 71%; white solid; mp: 72-74 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.29-7.13 (m, 4H), 6.86 (dd, J = 2.2, 6.7 Hz, 2H), 5.34 (dd, J = 1.4, 6.1 Hz, 2H), 5.34 (d, J = 1.3 Hz, 1H), 5.33 (d, J = 1.4 Hz, 1H), 3.83 (s, 3H), 2.37 (s, 3H); ¹³C

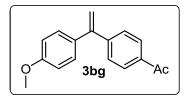
NMR (100 MHz, CDCl₃) δ 159.5, 149.4, 139.0, 137.4, 134.2, 129.4, 128.8, 128.2, 55.3, 21.2.

1-Chloro-4-(1-(4-methoxyphenyl)vinyl)benzene 3bf.³



Yield 88%; white solid; mp: 66-68 °C; ¹H NMR (300 MHz, CDCl₃) δ 7.31-7.24 (m, 6H), 6.88 (dd, J = 2.1, 6.7 Hz, 2H), 5.41 (d, J = 1.1 Hz, 1H), 5.35 (d, J = 1.1 Hz, 1H).

1-(4-(1-(4-Methoxyphenyl)vinyl)phenyl)ethanone 3bg.

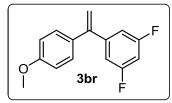


Yield 85%; white solid; mp: 96 °C; IR (film) 2958, 2837, 1686, 1604, 1510, 1250, 1180, 1026, 902.6, 842.0, 732.0; ¹H NMR (300 MHz, CDCl₃) δ 7.92 (dd, *J* = 1.7, 6.5 Hz, 2H), 7.45-7.42 (m, 2H), 7.26-7.23 (m, 2H), 6.88 (dd, *J* =

2.0, 6.6 Hz, 2H), 5.49 (d, J= 1.0 Hz, 1H), 5.44 (d, J = 1.0 Hz, 1H), 3.83 (s, 3H), 2.62

(s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 197.7, 159.5, 148.7, 146.6, 136.3, 133.2, 129.3, 128.5, 128.3, 114.6, 113.7, 55.3, 26.6; HRMS (ESI) *m/e* calcd for C₁₇H₁₇O₂ (M+H)⁺ 253.1223, found 253.1225.

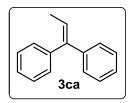
1, 3-Difluoro-5-(1-(4-methoxyphenyl)vinyl)benzene 3br.



Yield 96%; white solid; mp: 46-47 °C; IR (film) 3091, 2958, 2834, 1620, 1586, 1511, 1346, 1247, 1178, 1117, 1034, 987.0, 866.6, 835.5; ¹H NMR (400 MHz, CDCl₃) δ 7.26 -7.22 (m, 2H), 6.90- 6.84 (m, 4H), 6.78- 6.73 (m,

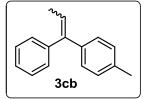
1H), 5.44 (s, 1H), 5.39 (d, J = 0.8 Hz, 1H), 3.83 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 162.8 (dd, J = 12.9, 247.9 Hz, 2C), 159.6, 147.7, 145.2 (t, J = 9.3 Hz, 1C), 132.7, 129.4, 114.5, 113.8, 111.1 (dd, J = 6.7, 18.5, 2C), 102.9 (t, J = 25.5 Hz, 1C), 55.3. HRMS (ESI) *m/e* calcd for C₁₅H₁₃F₂O (M+H)⁺ 247.0929, found 247.0932.

Prop-1-ene-1,1-diyldibenzene 3ca.



Yield 54%; colorless oil; ¹H NMR (400 MHz, CDCl₃) δ 7.39 -7.17 (m, 10H), 6.17 (q, *J* = 7.0 Hz, 1H), 1.76 (d, *J* = 7.0 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 143.0, 142.4, 140.0, 130.0, 128.1, 128.0, 127.2, 126.8, 126.7, 124.1, 15.7.

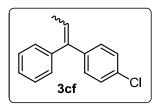
1-Methyl-4-(1-phenylprop-1-enyl)benzene 3cb.



Yield 55%; colorless oil; IR (film) 3026, 2911, 2856, 1511, 1441, 809.8, 759.0, 702.3; ¹H NMR (400 MHz, CDCl₃) δ 7.36-7.06 (m, 9H), 6.13 (q, *J* = 7.0 Hz, 1H), 2.38 (s, 1.25H), 2.32 (s, 1.75H), 1.76 (d, *J* = 7.0 Hz, 1.20H), 1.74 (d, *J* = 7.0

Hz, 1.80H); ¹³C NMR (100 MHz, CDCl₃) δ 143.2, 142.3, 142.2, 140.2, 137.0, 136.4, 130.0, 129.9, 128.8, 128.7, 128.1, 128.0, 127.2, 127.1, 126.7, 126.6, 123.9, 123.2, 21.2, 21.0, 15.7, 15.6. HRMS (ESI) *m/e* calcd for C₁₆H₁₇ (M+H)⁺ 209.1325, found 209.1326.

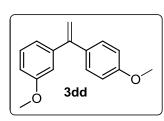
1-Chloro-4-(1-phenylprop-1-enyl)benzene 3cf.



Yield 58%; colorless oil; IR (film) 3029, 2911, 2849, 1489, 1442, 1091, 1014, 894.9, 816.5, 759.4, 701.0; ¹H NMR (400 MHz, CDCl₃) δ 7.37-7.11 (m, 9H), 6.20-6.13 (m, 1H), 1.76 (s, 1.5H), 1.74 (s, 1.5H); ¹³C NMR (100 MHz, CDCl₃) δ 142.5, 141.4, 141.4, 141.3, 139.5, 138.4, 132.7, 132.5, 131.4,

129.9, 128.4, 128.4, 128.2, 128.1, 128.1, 127.1, 127.0, 126.9, 124.7, 124.7, 15.7, 15.7. HRMS (ESI) *m/e* calcd for C₁₅H₁₄Cl (M+H)⁺ 229.0779, found 229.0781.

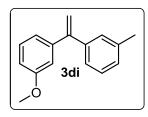
1-Methoxy-3-(1-(4-methoxyphenyl)vinyl)benzene 3dd.



Yield 72%; colorless oil; IR (film) 3001, 2958, 2831, 1606, 1510, 1247, 1178, 1035, 885.6, 836.7, 786.6, 715.5; ¹H NMR (400 MHz, CDCl₃) δ 7.29-7.23 (m, 3H), 6.94-6.85 (m, 5H), 5.39 (d, *J* = 1.2 Hz, 1H), 5.36 (d, *J* = 1.2 Hz, 1H), 3.82 (s, 3H), 3.79 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ

159.4, 159.3, 149.3, 143.3, 133.8, 129.4, 129.0, 120.9, 113.9, 113.4, 113.1, 113.0, 55.3, 55.2. HRMS (ESI) *m/e* calcd for $C_{16}H_{17}O_2$ (M+H)⁺ 241.1123, found 241.1124.

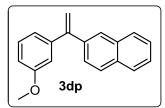
1-Methoxy-3-(1-m-tolylvinyl)benzene 3di.



Yield 71%; colorless oil; IR (film) 2948, 2834, 1598, 1577, 1486, 1247, 1047, 882.6, 791.5, 706.1; ¹H NMR (400 MHz, CDCl₃) δ 7.24-7.20 (m, 2H), 7.16-7.11 (m, 3H), 6.93-6.85 (m, 3H), 5.43 (s, 2H), 3.78 (s, 3H), 2.34 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 159.4, 150.0, 143.1, 141.3, 137.7, 129.0,

128.9, 128.5, 128.0, 125.4, 120.9, 114.2, 113.9, 113.1, 55.2, 21.4. HRMS (ESI) m/e calcd for C₁₆H₁₇O (M+H)⁺ 225.1274, found 225.1276.

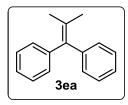
2-(1-(3-Methoxyphenyl)vinyl)naphthalene 3dp.



Yield 56%; white Solid; mp: 79-81 °C; IR (film) 3054, 2914, 2849, 1600, 1577, 1489, 1248, 1046, 896.5, 859.8, 822.1, 785.9, 752.5, 700.0; ¹H NMR (400 MHz, CDCl₃) δ 7.85-7.79 (m, 4H), 7.50-7.46 (m, 3H), 6.99-6.89 (m, 3H), 5.59 (d, *J* = 1.2 Hz, 1H), 5.55 (d, *J* = 1.2 Hz, 1H), 3.79 (s,

3H); ¹³C NMR (100 MHz, CDCl₃) δ 159.5, 149.9, 143.0, 138.7, 133.3, 132.9, 129.2, 128.2, 127.7, 127.6, 127.3, 126.4, 126.1, 126.0, 121.0, 114.9, 114.0, 113.4, 55.2. HRMS (ESI) *m/e* calcd for C₁₉H₁₇O (M+H)⁺ 261.1274, found 261.1276.

(2-Methylprop-1-ene-1,1-diyl)dibenzene 3ea.⁸



Yield 10 %; colorless oil; ¹H NMR (400 MHz, CDCl₃) δ 7.29-7.12 (m, 10H), 1.80 (s, 6H).

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5) ¹H NMR and ¹³C NMR spectra

