Experimental Procedures and Physical Data:

4,4’-Dimethoxybiphenyl (2a). To a mixture of 4-iodoanisole (0.2269 g, 0.9695 mmol), hydroquinone (59.1 mg, 0.537 mmol), and Cs_2CO_3 (0.3275 g, 1.005 mmol) was added a homogenous pre-stirred DMA solution (2.50 mL) of $Pd(OAc)_2$ (4.3 mg, 0.019 mmol) and tri-*o*-tolylarsine[1’] (6.9 mg, 0.020 mmol). The reaction mixture darkened immediately upon addition of the catalyst solution to the solid reagents. The mixture was degassed, using N_2 and house vacuum, and heated under N_2 at 75°C for 2 hours at which time HPLC analysis shows complete consumption of 4-iodoanisole. The reaction mixture was cooled to room temperature, quenched with 2N HCl (20 mL), diluted with water (20 mL), and extracted with EtOAc (3 x 30 mL). The combined organics were washed with 10% NaOH (4 x 40mL), brine (40 mL), dried over $MgSO_4$, concentrated under reduced pressure, and purified using flash column chromatography on silica gel (elution with 20% ether in hexanes) to afford 98.5 mg (95%) of biphenyl **2a** as a white crystalline solid: mp 175-176°C, Lit[2’] = 175°C; ^1H NMR (300 MHz, $CDCl_3$) δ 3.84 (s, 6H), 6.96 (d, J = 8.7 Hz, 4H), 7.47 (d, J = 8.7 Hz, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 55.3, 114.1, 127.7, 133.4, 158.6; HRMS (*m*/*z*) calcd for $C_{14}H_{14}O_2$ $[M^+]$: 214.0994, found 214.0994.

3,3’-Dimethoxybiphenyl (2b). To a mixture of 3-iodoanisole (0.2155 g, 0.9116 mmol), hydroquinone (55.0 mg, 0.500 mmol), and Cs_2CO_3 (0.2996 g, 0.9195 mmol) was added a homogenous pre-stirred DMA solution (2.3 mL) of $Pd(OAc)_2$ (4.0 mg, 0.018 mmol) and tri-*o*-tolylarsine (6.9 mg, 0.020 mmol). The mixture was degassed, using N_2 and house vacuum, and heated under N_2 at 75°C for 5.5 hours at which time HPLC analysis shows complete consumption of 3-iodoanisole. The reaction mixture was cooled to room temperature, quenched with 2N HCl (20 mL), diluted with water (20 mL), and extracted with EtOAc (3 x 30 mL). The combined organics were washed with 10% NaOH (4 x 40mL), brine (40 mL), dried over $MgSO_4$, concentrated under reduced pressure, and purified using flash column chromatography on silica gel (elution with 11% ether in hexanes) to afford 93.6 mg (96%) of biphenyl **2b** as a white crystalline solid: mp 36-37°C, Lit[3’] = 36°C; ^1H NMR (300 MHz, $CDCl_3$) δ 3.86 (s, 6H), 6.90 (dd = 8.1, 2.2 Hz, 2H), 7.12 (d, J = 1.7 Hz, 2H), 7.18 (d, J = 8.1 Hz, 2H), 7.35 (t, J = 7.9 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 55.3, 112.8, 112.9, 119.7, 129.7, 142.6, 159.8; HRMS (*m*/*z*) calcd for $C_{14}H_{14}O_2$ $[M^+]$: 214.0994, found 214.0990.

2,2’-Dimethoxybiphenyl (2c). To a mixture of 2-iodoanisole (0.3590 g, 1.534 mmol), hydroquinone (87.4 mg, 0.794 mmol), and Cs_2CO_3 (0.5208 g, 1.598 mmol) was added a homogenous pre-stirred DMA solution (1.50 mL) of $Pd(OAc)_2$ (13.4 mg, 0.0598 mmol) and tri-*o*-tolylarsine (21.0 mg, 0.0603 mmol). The mixture was degassed, using N_2 and house vacuum, and heated under N_2 at 100°C for 48 hours at which time HPLC analysis shows almost complete consumption of 2-iodoanisole. The reaction mixture was cooled to room temperature, quenched with 2N HCl (20 mL), diluted with water (20 mL), and extracted with EtOAc (3 x 30 mL). The combined organics were washed with 10% NaOH (4 x 40mL), brine (40 mL), dried over $MgSO_4$, concentrated under reduced pressure, and purified using flash column chromatography on silica gel (elution with 14% ether in hexanes) followed by recrystallization with ether/hexanes to afford 0.1314 g (82%) of biphenyl **2c** as colorless prisms: mp 155.5-156°C, Lit[4’] = 154-155°C; ^1H NMR (300 MHz, $CDCl_3$) δ 3.78 (s, 6H), 6.99 (m, 4H), 7.28 (d, J = 7.0 Hz, 2H), 7.32 (t, J = 7.4 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 55.6, 111.0, 120.3, 127.7, 128.6, 131.4, 157.0; HRMS (*m*/*z*) calcd for $C_{14}H_{14}O_2$ $[M^+]$: 214.0994, found 214.0995.

Biphenyl (2d). The coupling protocol used for **2a** was followed using iodobenzene (0.3026 g, 1.483 mmol), hydroquinone (84.9 mg, 0.771 mmol), and Cs_2CO_3 (0.4828 g, 1.482

mmol) and a catalyst solution of DMA (3.7 mL) of $Pd(OAc)_2$ (6.8 mg, 0.030 mmol) and tri-*o*-tolylarsine (10.8 mg, 0.0310 mmol). After 3 hours at 75˚C, standard work-up and purification (elution with 11% ether in hexanes) afforded 0.1103 g (96%) of biphenyl **2d** as a white crystalline solid: mp 67-69˚C, Lit$^{5'}$ = 68˚C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.33 (t, J = 8.4 Hz, 2H), 7.44 (t, J = 7.9 Hz, 4H), 7.58 (d, J = 8.3 Hz, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 127.1, 127.2, 128.7, 141.2; HRMS (*m*/*z*) calcd for $C_{12}H_{10}O_2$ [M^+]: 154.0782, found 154.0779.

4,4'-Dicarbomethoxybiphenyl (2e). The coupling protocol used for **2a** was followed using methyl-4-iodo-benzoate (0.2116 g, 0.8074 mmol), hydroquinone (53.0 mg, 0.481 mmol), and Cs_2CO_3 (0.2671 g, 0.8198 mmol) with a catalyst solution of $Pd(OAc)_2$ (3.9 mg, 0.017 mmol) and tri-*o*-tolylarsine (5.9 mg, 0.017 mmol) in DMA (2.0 mL). After 2 hours at 75˚C, standard work-up (elution with 20% ether in hexanes) afforded 0.1077 g (99%) of biphenyl **2e** as a white crystalline solid: mp 212-213˚C, Lit$^{6'}$ = 214˚C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.95 (s, 6H), 7.68 (d, J = 8.4 Hz, 4H), 8.12 (d, J = 8.5 Hz, 4H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 52.2, 127.2, 129.7, 130.2, 144.3, 166.8; HRMS (*m*/*z*) calcd for $C_{16}H_{14}O_4$ [M^+]: 270.0892, found 270.0892.

2,2'-Dicarbomethoxybiphenyl (2f). The coupling protocol used for **2a** was followed using methyl-2-iodo-benzoate (0.6706 g, 2.559 mmol), hydroquinone (0.1495 g, 1.358 mmol), and Cs_2CO_3 (0.8474 g, 2.600 mmol) with a catalyst solution of $Pd(OAc)_2$ (24.3 mg, 0.108 mmol) and tri-*o*-tolylarsine (35.9 mg, 0.103 mmol) in DMA (2.6 mL). After 18 hours at 100˚C, standard work-up (elution with 20% ether in hexanes) afforded 0.2850 g (82%) of biphenyl **2f** as a pinkish crystalline solid: mp 73-74˚C, Lit$^{7'}$ = 73-74˚C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.60 (s, 6H), 7.19 (d, J = 7.5 Hz, 2H), 7.41 (dt, J = 7.6, 1.0 Hz, 2H), 7.52 (dt, J = 7.5, 1.1 Hz, 2H), 8.00 (dd, J = 7.8, 1.2 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 51.6, 127.0, 129.1, 129.7, 130.0, 131.3, 143.1, 167.2; HRMS (*m*/*z*) calcd for $C_{16}H_{14}O_4$ [M^+]: 270.0892, found 270.0899.

4,4'-Dinitrobiphenyl (2g). The coupling protocol used for **2a** was followed using 4-iodo-nitrobenzene (0.2990 g, 1.201 mmol), hydroquinone (69.8 mg, 0.634 mmol), and Cs_2CO_3 (0.4094 g, 1.257 mmol) with a catalyst solution of $Pd(OAc)_2$ (5.7 mg, 0.025 mmol) and tri-*o*-tolylarsine (9.0 mg, 0.026 mmol) in DMA (3.0 mL). After 1 hour at 75˚C, standard work-up (elution with 14% ether in hexanes) afforded 0.1256 g (86%) of biphenyl **2g** as a tan crystalline solid: mp 235˚C, Lit$^{8'}$ = 240˚C; ^{1}H NMR (300 MHz, DMSO-d_6) δ 8.08 (d, J = 8.8 Hz, 4H), 8.37 (d, J = 8.8 Hz, 4H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 124.3, 128.7, 144.1, 147.6; HRMS (*m*/*z*) calcd for $C_{12}H_8N_2O_4$ [M^+]: 244.0484, found 244.0484.

3,3'-Dinitrobiphenyl (2h). The coupling protocol used for **2a** was followed using 3-iodo-nitrobenzene (0.2302 g, 0.9245 mmol), hydroquinone (54.6 mg, 0.496 mmol), and Cs_2CO_3 (0.3033 g, 1.014 mmol) with a catalyst solution of $Pd(OAc)_2$ (4.3 mg, 0.019 mmol) and tri-*o*-tolylarsine (6.6 mg, 0.019 mmol) in DMA (2.3 mL). After 2.5 hours at 75˚C, the reaction mixture was cooled to room temperature, quenched with 2N HCl (20 mL), diluted with water (20 mL), and extracted with EtOAc (3 x 30 mL). The combined organics were washed with 10% NaOH (4 x 40mL), brine (40 mL), dried over $MgSO_4$, concentrated under reduced pressure, and triturated with 1:1 hexanes/acetone to afford 96.9 mg (86%) of biphenyl **2h** as a tan crystalline solid: mp 200-201˚C, Lit$^{9'}$ = 200˚C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.67 (t, J = 8.0 Hz, 2H), 6.86 (dt, J = 7.8, 1.5 Hz, 2H), 7.75 (dt, J = 8.1, 1.5 Hz, 2H), 7.93 (t, J = 1.9 Hz, 2H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 121.8, 123.3, 130.8, 133.8, 139.5, 148.5; HRMS (*m*/*z*) calcd for $C_{12}H_8N_2O_4$ [M^+]: 244.0484, found 244.0498.

1,1'-Binaphthyl (4). The coupling protocol used for **2a** was followed using 1-bromonaphthalene (1.0105 g, 4.782 mmol), hydroquinone (0.2854 g, 2.592 mmol), and Cs_2CO_3

(1.6243 g, 4.985 mmol) with a catalyst solution of $Pd(OAc)_2$ (41.7 mg, 0.186 mmol) and tri-*o*-tolylarsine (66.9 mg, 0.192 mmol) in DMA (4.8 mL). After 48 hours at 100˚C, standard work-up (elution with 100% hexanes) afforded 0.5481 g (90%) of binaphthalene **4** as a white crystalline solid: mp 155-156˚C, Lit[10'] = 155-156˚C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.28 (t, J = 7.8 Hz, 2H), 7.38 (d, J = 8.5 Hz, 2H), 7.47 (m, 4H), 7.58 (t, J = 8.0 Hz, 2H), 7.93 (d, J = 8.1 Hz, 2H), 7.94 (d, J = 8.2 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 125.4, 125.8, 125.9, 126.5, 127.8, 127.9, 128.1, 132.8, 133.5, 138.4; HRMS (*m*/*z*) calcd for $C_{20}H_{14}$ [M$^+$]: 254.1096, found 254.1092.

2,2'-Bipyridine (8). To a mixture of 2-bromopyridine (0.6737 g, 4.264 mmol), hydroquinone (0.2642 g, 2.399 mmol), and Cs_2CO_3 (1.37 g, 4.20 mmol) was added a homogenous pre-stirred DMA solution (3.8 mL) of $Pd(OAc)_2$ (37.4 mg, 0.167 mmol) and tri-*o*-tolylarsine (59.1 mg, 0.170 mmol). The mixture was degassed, using N_2 and house vacuum, and heated under N_2 at 100˚C for 24 hours at which time GC analysis shows almost complete consumption of 2-bromopyridine. The reaction mixture was cooled to room temperature, diluted with ethyl acetate and washed with 1M NaOH. The combined aqueous phase was washed with ethyl acetate followed by washing the combined organics with brine. The organic phase was dried over $MgSO_4$, concentrated under reduced pressure, and purified using flash column chromatography on silica gel (elution with 9% ether in hexanes followed by 50% ether in hexanes) to afford 0.2385 g (72%) of bipyridyl **8** as a pinkish crystalline solid. Recrystallization from ether/hexanes gave a white crystalline solid: mp 69-72˚C, Lit = 70-73˚C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.17 (m, 2H), 7.69 (td, J = 7.7, 1.8 Hz, 2H), 8.29 (dt, J = 8.1, 1.1 Hz, 2H), 8.57 (d, J = 5.5 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 120.9, 123.5, 136.7, 149.0, 156.0; Mass spectrum (EI) *m/e* (rel. intensity) 156.05 (100). Calcd for $C_{10}H_8N_2$ [M$^+$]: 156.07.

***N*,*N*-Di-(2-iodobenzyl)-*p*-toluenesulfonamide (9)**. Sulfonamide **9** was prepared as described for **10** using NaH (0.22 g, 5.5 mmol), *p*-toluenesulfonamide (0.43 g, 2.5 mmol), 2-iodobenzyl bromide (1.50 g, 5.07 mmol), and DMF (50 mL) with purification on silica gel (elution with 20% ether in hexanes) to afford 1.3718 g (91%) of bis-benzylated sulfonamide **9** as a white crystalline solid: mp 144-145˚C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.46 (s, 3H), 4.44 (s, 4H), 6.84 (dt, J = 7.8, 1.6 Hz, 2H), 6.86 (dt, J = 7.6, 1.0 Hz, 2H), 7.30 (dd, J = 7.7, 1.4 Hz, 2H), 7.33 (d, J = 8.0 Hz, 2H), 7.64 (dd, J = 7.9, 1.0 Hz, 2H), 7.76 (d, J = 8.3 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.6, 57.0, 98.8, 127.3, 128.2, 129.1, 129.6, 129.8, 136.6, 137.6, 139.3, 143.6; IR (neat, cm^{-1}) 3056, 2920, 1436, 1341, 1159, 1091, 1011, 907; HRMS (*m*/*z*) calcd for $C_{21}H_{19}I_2NO_2S$ [M$^+$]: 602.9226, found 602.9219.

6-*p*-Toluenesulfonyl-6,7-dihydro-5H-dibenz<*c*,*e*>azepine (10). The coupling protocol used for **2a** was followed using bis-iodide **7** (0.3571 g, 0.5919 mmol), hydroquinone (34.5 mg, 0.313 mmol), and Cs_2CO_3 (0.1992 g, 0.6114 mmol) and a catalyst solution of DMA (1.5 mL) of $PdOAc_2$ (6.8 mg, 0.030 mmol) and tri-*o*-tolylarsine (10.6 mg, 0.0304 mmol). After 48 hours at 100˚C, standard work-up (elution with 20% ether in hexanes) afforded 0.1704 g (82%) of biaryl **10** as a white crystalline solid: mp 161˚C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.39 (s, 3H), 4.15 (bs, 4H), 7.23 (d, J = 7.3 Hz, 2H), 7.27 (m, 4H), 7.41 (m, 4H), 7.74 (d, J = 7.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 21.5, 48.9, 127.4, 127.9, 128.1, 128.8, 129.5, 129.8, 131.9, 136.4, 140.4, 143.3; IR (neat, cm^{-1}) 3053, 2923, 2860, 1451, 1335, 1158, 1089, 915; HRMS (*m*/*z*) calcd for $C_{21}H_{19}NO_2S$ [M$^+$]: 349.1137, found 349.1144.

Di-2-iodobenzyl ether (11). Sodium hydride (0.1587g, 3.97 mmol) was washed free of mineral oil (3 hexane washings) and treated with DMF (15 mL) followed by a solution of 2-iodobenzyl alcohol (0.8578 g, 3.67 mmol) in DMF (5mL). After H_2 evolution had ceased, solid 2-iodo-benzyl bromide (1.07 g, 3.62 mmol) was added and the reaction mixture was stirred at room temperature for 16 hours followed by stirring at 70˚C for 2 additional hours. The reaction mixture was cooled to room temperature, poured into 2N HCl (50 mL), and extracted with ether (3 x 30 mL). The combined organics were washed with brine (50 mL), dried over $MgSO_4$, and concentrated under reduced pressure, and purified using flash column chromatography on silica gel (elution with 11% diethyl ether in hexanes) to give 1.3643 g (84%) of benzylic ether **11** as a pinkish-white crystalline solid: mp 65-66˚C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.63 (s, 4H), 6.99 (dt, J = 8.0, 1.5 Hz, 2H), 7.36 (t, J = 7.5 Hz, 2H), 7.53 (d, J = 7.5 Hz, 2H), 7.83 (d, J = 7.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 76.4, 97.7, 128.3, 128.8, 129.2, 139.1, 140.3; IR (neat, cm^{-1}) 2850, 1460, 1432, 1344, 1207, 1136, 1097, 1012; HRMS (*m*/*z*) calcd for $C_{14}H_{12}I_2O$ [M^+]: 449.8978, found 449.8975.

5,7-Dihydro-dibenzo<*c*,*e*>oxepine (12). The coupling protocol used for **2a** was followed using bis-iodide **11** (0.2426 g, 0.5390 mmol), hydroquinone (29.7 mg, 0.270 mmol), and Cs_2CO_3 (0.1763 g, 0.5410 mmol) and a catalyst solution of DMA (1.5 mL) of $PdOAc_2$ (6.7 mg, 0.030 mmol) and tri-*o*-tolylarsine (9.9 mg, 0.028 mmol). After 24 hours at 100˚C, standard work-up (elution with 14% ether in hexanes) afforded 31.8 mg (13%) of recovered **11** along with 64.1 mg (61%) of biaryl **12** as a white crystalline solid: mp 71-72˚C, Lit[11'] = 72.5-73˚C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.37 (s, 4H), 7.44 (m, 4H), 7.52 (dt, J = 7.6, 2.1 Hz, 2H), 7.56 (d, J = 7.4 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 67.5, 127.5, 128.3, 128.9, 129.7, 135.1, 141.2; IR (neat, cm^{-1}) 3054, 3017, 2953, 2854, 1443, 1197, 1077, 1044; HRMS (*m*/*z*) calcd for $C_{14}H_{12}O$ [M^+]: 196.0888, found 196.0889.

Diethyl bis(2-iodobenzyl)malonate (13). To a suspension of 60% sodium hydride (0.480 g, 12 mmol) was added diethyl malonate (759 µL, 5 mmol) dropwise at room temperature. After 30 min, 2-iodo-benzyl bromide (3.563 g, 12 mmol) was added in several portions at room temperature (caution: exothermic). After 2 h, the reaction mixture was poured into water (100 mL) and extracted with diethyl ether (3 x 80 mL). The combined organics were washed with brine twice), dried over $MgSO_4$, and concentrated under reduced pressure, and purified using flash column chromatography on silica gel (elution with 9% diethyl ether in hexanes) to give 2.762 g (93%) of malonate **13** as a white solid: ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.06 (t, J = 7 Hz, 6H), 3.60. (s, 4H), 4.09 (q, J = 7 Hz, 4H), 6.87 (dt, J = 1.5, 7.5 Hz, 2H), 7.22 (dt, J = 1.5, 7.5 Hz, 2H), 7.29 (dd, J = 1.5, 7.5 Hz, 2H), 7.81 (dd, J = 1, 7.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 13.6, 43.6, 59.8, 61.6, 103.1, 128.0, 128.4, 130.5, 139.7, 140.0, 170.5.

5,7-Dihydro-dibenzo<*a*,*c*>cycloheptene-6,6-dicarboxylic acid diethyl ester (14). To a mixture of **13** (0.592 g, 1 mmol), hydroquinone (55 mg, 0.5 mmol), and Cs_2CO_3 (0.326 g, 1 mmol) was added a homogenous pre-stirred DMA solution (2.50 mL) of $Pd(OAc)_2$ (11.2 mg, 0.05 mmol) and tri-*o*-tolylarsine (17.4 mg, 0.05 mmol). The reaction mixture darkened immediately upon addition of the catalyst solution to the solid reagents. The mixture was degassed, using N_2 and house vacuum, and heated under N_2 at 100 ˚C for . The reaction mixture was cooled to room temperature, quenched with 2N HCl (20 mL), diluted with water (20 mL), and extracted with EtOAc (3 x 25 mL). The combined organics were washed with 10% NaOH (4 x 20mL), brine, dried over $MgSO_4$, concentrated under reduced pressure, and purified using flash column chromatography on silica gel (elution with hexanes : toluene : ether = 30 : 10 : 1) to afford 81 mg (14%) of recovered **13** along with 221 mg (65%) of biaryl **14** as a colorless oil: ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.27 (t, J = 7 Hz, 6H), 2.89 (br s, 2H), 3.22 (br s, 2H), 4.21 (br s, 4H), 7.25-7.30 (m, 4H), 7.32-7.38 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.1, 36.8, 61.5, 64.8, 127.4, 127.5, 128.1, 130.1, 135.3, 140.5, 170.7.

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