

Supporting Information

Pd-catalyzed Decarboxylative Arylation of Thiazole, Benzoxazole and Polyfluorobenzene with Benzoic Acids †

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Table of Contents

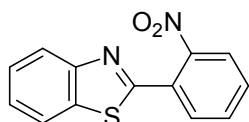
I General Consideration	S 2
II Representative Procedure	S 2
III Characterization Data for Selected Compounds	S 2- S 29

General Consideration:

All solvents and reagents were purchased from the suppliers and used without further purification. ^1H NMR and ^{13}C NMR were recorded in CDCl_3 at room temperature on the Varian INOVA-400 spectrometer (400 MHz ^1H) and Bruker spectrometer (400 MHz ^1H). The chemical-shifts scale is based on internal TMS. Trifluoroacetic acid was employed as an external standard in ^{19}F NMR. All reactions were carried out under dry nitrogen atmosphere.

Representative Procedure and Selected Compounds Data

2-(2-nitrophenyl)benzo[d]thiazole (3a) ^[1]



To a flame dried three neck round bottom flask under nitrogen was added benzothiazole (68 mg, 0.5 mmol), 2-nitrobenzoic acid (126 mg, 0.75 mmol), Ag_2CO_3 (414 mg, 1.5 mmol), PdCl_2 (18 mg, 0.1 mmol), PPh_3 (53 mg, 0.2 mmol) and DMSO (3 mL). The reaction mixture was heated at 130°C for 12 h. After cooling to room temperature, the reaction mixture was diluted with ether and filtered through Celite. The organic phase was washed with saturated NH_4Cl , dried with MgSO_4 , filtered, and concentrated via vacua. The residue was purified by flash column chromatography on silica gel (petroleum ether/ ethyl acetate, 50/1) to afford 77 mg of the desired 2-(2-nitrophenyl)benzo[d]thiazole in 60% yield.

TLC R_f 0.20 (petroleum ether/ ethyl acetate, 12/1); Yield: 60%, yellow crystals; m.p.: $127\text{--}129^\circ\text{C}$;

^1H NMR (CDCl_3 , 400 MHz) δ 8.01 (d, $J = 8.4$ Hz, 1 H), 7.87 (t, $J = 7.2$ Hz, 2 H), 7.73 (d, $J = 6.4$ Hz, 1 H), 7.64 (d, $J = 8.0$ Hz, 1 H), 7.58 (d, $J = 7.6$ Hz, 1 H), 7.47 (t, $J = 8.4$ Hz, 1 H), 7.38 (t, $J = 7.2$ Hz, 1 H);

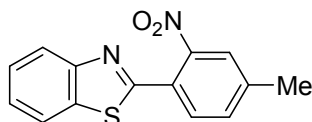
^{13}C NMR (CDCl_3 , 100 MHz) δ 162.33, 153.50, 148.86, 135.74, 132.39, 131.78, 128.93, 128.06, 126.57, 125.85, 124.51, 123.91, 121.57;

LRMS: m/z calcd for $\text{C}_{13}\text{H}_8\text{N}_2\text{O}_2\text{S}$ (M+H): 257, found: 257;

Anal. Calcd for $\text{C}_{13}\text{H}_8\text{N}_2\text{O}_2\text{S}$: Elemental Analysis: C, 60.93; H, 3.15; N, 10.93; Found: C, 61.05; H, 3.30; N, 10.86;

IR (KBr) ν (cm^{-1}): 3064, 2962, 2924, 2854, 1742, 1608, 1574, 1532, 1472, 1456, 1360, 1312, 1260, 1091, 970, 860, 760.

2-(4-methyl-2-nitrophenyl)benzo[d]thiazole (3b)



Flash chromatography (petroleum ether/ ethyl acetate, 50/1); TLC R_f 0.22 (petroleum ether/ ethyl acetate, 12/1); Yield: 61%, yellow crystals, m.p.: $80\text{--}82^\circ\text{C}$;

^1H NMR (CDCl_3 , 400 MHz) δ 8.07 (d, $J = 8.0$ Hz, 1 H), 7.92 (d, $J = 8.0$ Hz, 1 H), 7.72 (s, 1 H), 7.68 (d, $J = 8.0$ Hz, 1 H), 7.54 (d, $J = 8.0$ Hz, 1 H), 7.50 (t, $J = 7.2$ Hz, 1 H), 7.44 (t, $J = 7.2$

Hz, 1 H), 2.51 (s, 3 H);

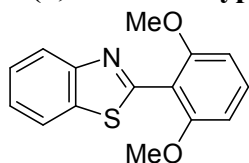
^{13}C NMR (CDCl_3 , 100 MHz) δ 162.62, 153.58, 148.77, 142.14, 135.76, 133.07, 131.63, 126.73, 125.78, 125.23, 124.96, 123.87, 121.61, 21.33;

LRMS: m/z calcd for $\text{C}_{14}\text{H}_{10}\text{N}_2\text{O}_2\text{S}$ ($\text{M}+\text{H}$): 271, found: 271;

Anal. Calcd for $\text{C}_{14}\text{H}_{10}\text{N}_2\text{O}_2\text{S}$: Elemental Analysis: C, 62.21; H, 3.73; N, 10.36; Found: C, 62.41; H, 3.99; N, 10.06;

IR (KBr) ν (cm^{-1}): 2922, 1615, 1537, 1485, 1485, 1434, 1362, 1312, 1276, 1230, 1124, 972, 829, 759.

2-(2,6-dimethoxyphenyl)benzo[d]thiazole (3c) ^[2]



Flash chromatography (petroleum ether/ ethyl acetate, 30/1); TLC R_f 0.15 (petroleum ether/ ethyl acetate, 12/1); Yield: 58%, yellow crystals, m.p.: 119-121°C;

^1H NMR (CDCl_3 , 400 MHz) δ 8.06 (d, J = 8.4 Hz, 1 H), 7.84 (d, J = 7.6 Hz, 1 H), 7.41 (t, J = 8.0 Hz, 1 H), 7.32 (m, 2 H), 6.58 (d, J = 8.0 Hz, 2 H), 3.71 (s, 6 H);

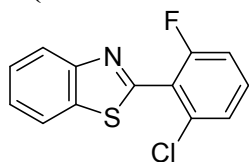
^{13}C NMR (CDCl_3 , 100 MHz) δ 161.93, 158.70 (2 C), 153.21, 136.53, 131.61, 125.50, 124.75, 123.38, 121.26, 114.89, 103.83 (2 C), 56.02 (2 C);

LRMS: m/z calcd for $\text{C}_{15}\text{H}_{13}\text{NO}_2\text{S}$ ($\text{M}+\text{H}$): 272, found: 272;

Anal. Calcd for $\text{C}_{15}\text{H}_{13}\text{NO}_2\text{S}$: Elemental Analysis: C, 66.40; H, 4.83; N, 5.16; Found: C, 66.56; H, 4.93; N, 5.02;

IR (KBr) ν (cm^{-1}): 3339, 2936, 2837, 1601, 1589, 1523, 1474, 1432, 1302, 1256, 1212, 1110, 958, 783.

2-(2-chloro-6-fluorophenyl)benzo[d]thiazole (3d) ^[3]



Flash chromatography (petroleum ether/ ethyl acetate, 50/1); TLC R_f 0.45 (petroleum ether/ ethyl acetate, 12/1); Yield: 55%, yellow oil;

^1H NMR (CDCl_3 , 400 MHz) δ 8.19 (d, J = 8.0 Hz, 1 H), 7.98 (d, J = 8.0 Hz, 1 H), 7.56 (t, J = 8.0 Hz, 1 H), 7.48 (t, J = 8.0 Hz, 1 H), 7.42 (d, J = 8.4 Hz, 1 H), 7.36 (d, J = 8.0 Hz, 1 H), 7.16 (t, J = 8.0 Hz, 1 H);

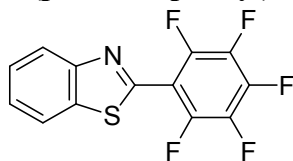
^{13}C NMR (CDCl_3 , 100 MHz) δ 159.80 (d, $J_{\text{C-F}}$ = 252.5 Hz), 157.47, 151.97, 135.27, 133.98, 130.82 (d, $J_{\text{C-F}}$ = 9.2 Hz), 125.27, 124.80, 124.74, 122.88, 121.05 (d, $J_{\text{C-F}}$ = 17.0 Hz), 120.51, 113.57 (d, $J_{\text{C-F}}$ = 22.1 Hz);

LRMS: m/z calcd for $\text{C}_{13}\text{H}_7\text{ClFNS}$ ($\text{M}+\text{H}$): 264, found: 264;

Anal. Calcd for $\text{C}_{13}\text{H}_7\text{ClFNS}$: Elemental Analysis: C, 59.21; H, 2.68; N, 5.31; Found: C, 59.30; H, 2.75; N, 5.22;

IR (KBr) ν (cm^{-1}): 3063, 2360, 1609, 1569, 1518, 1449, 1429, 1313, 1278, 1224, 1175, 1075, 965, 896, 760.

2-(perfluorophenyl)benzo[d]thiazole (3e) ^[4]



Flash chromatography (petroleum ether/ ethyl acetate, 50/1); TLC R_f 0.40 (petroleum ether/ ethyl acetate, 12/1); Yield: 42%, yellow crystals, m.p.: 124-126°C;

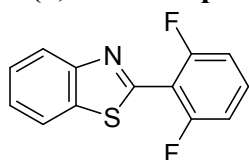
¹H NMR (CDCl₃, 400 MHz) δ 8.13 (d, *J* = 8.4 Hz, 1 H), 7.93 (d, *J* = 8.4 Hz, 1 H), 7.54 (t, *J* = 7.2 Hz, 1 H), 7.44 (t, *J* = 7.2 Hz, 1 H);

¹⁹F NMR (CDCl₃, 376 MHz) δ -138.69- -138.77 (m, 2 F), -150.41 (t, *J*_F = 21.0 Hz, 1 F), -160.81- -160.96 (m, 2 F);

LRMS: *m/z* calcd for C₁₃H₄F₅NS (M+H): 302, found: 302;

Anal. Calcd for C₁₃H₄F₅NS: Elemental Analysis: C, 51.83; H, 1.34; N, 4.65; Found: C, 51.89; H, 1.49; N, 4.52.

2-(2,6-difluorophenyl)benzo[d]thiazole (3f) ^[5]



Flash chromatography (petroleum ether/ ethyl acetate, 50/1); TLC R_f 0.40 (petroleum ether/ ethyl acetate, 12/1); Yield: 55%, yellow oil;

¹H NMR (CDCl₃, 400 MHz) δ 8.20 (d, *J* = 8.0 Hz, 1 H), 7.98 (d, *J* = 8.0 Hz, 1 H), 7.55 (t, *J* = 8.0 Hz, 1 H), 7.49-7.46 (m, 2 H), 7.10 (t, *J* = 8.4 Hz, 2 H);

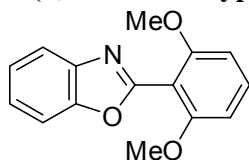
¹³C NMR (CDCl₃, 100 MHz) δ 160.64 (dd, *J*_{C-F} = 254.1 Hz, 5.6 Hz, 2 C), 156.02 (m), 153.11, 135.82, 132.08 (t, *J*_{C-F} = 10.6 Hz), 126.97 (d, *J*_{C-F} = 21.2 Hz), 126.52, 125.94, 124.02, 121.51, 112.47 (m, 2 C);

¹⁹F NMR (CDCl₃, 376 MHz) δ -110.09 (s, 2 F);

LRMS: *m/z* calcd for C₁₃H₇F₂NS (M+H): 248, found: 248;

Anal. Calcd for C₁₃H₇F₂NS: Elemental Analysis: C, 53.15; H, 2.85; N, 5.66; Found: C, 53.23; H, 2.92; N, 5.52.

2-(2,6-dimethoxyphenyl)benzo[d]oxazole (3g)



Flash chromatography (petroleum ether/ ethyl acetate, 30/1); TLC R_f 0.15 (petroleum ether/ ethyl acetate, 12/1); Yield: 45%, yellow crystals, m.p.: 132-134°C;

¹H NMR (CDCl₃, 400 MHz) δ 7.75 (m, 1 H), 7.52 (m, 1 H), 7.36 (t, *J* = 8.4 Hz, 1 H), 7.28 (m, 2 H), 6.58 (t, *J* = 8.4 Hz, 2 H), 3.72 (s, 6 H);

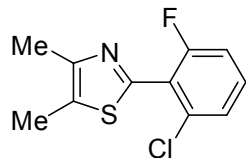
¹³C NMR (CDCl₃, 100 MHz) δ 158.77, 158.03 (2 C), 149.99, 140.74, 131.61, 123.71, 122.93, 119.19, 109.64, 105.65, 102.75 (2 C), 55.08 (2 C);

LRMS: m/z calcd for $C_{15}H_{13}NO_3$ (M+H): 256, found: 256;

Anal. Calcd for $C_{15}H_{13}NO_3$: Elemental Analysis: C, 70.58; H, 5.13; N, 5.49; Found: C, 70.63; H, 5.24; N, 5.40;

IR (KBr) ν (cm^{-1}): 3339, 2937, 2840, 1625, 1582, 1477, 1454, 1433, 1275, 1258, 1111, 1024, 750.

2-(2-chloro-6-fluorophenyl)-4,5-dimethylthiazole (3h)



Flash chromatography (petroleum ether/ ethyl acetate, 50/1); TLC R_f 0.40 (petroleum ether/ ethyl acetate, 12/1); Yield: 63%, yellow oil;

1H NMR ($CDCl_3$, 400 MHz) δ 7.19-7.28 (m, 2 H), 7.00 (t, J = 8.4 Hz, 1 H), 2.38 (s, 6 H);

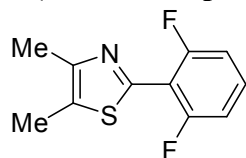
^{13}C NMR ($CDCl_3$, 100 MHz) δ 161.05 (d, J_{C-F} = 250.4 Hz), 152.87, 140.06, 135.24 (d, J_{C-F} = 3.0 Hz), 133.15 (d, J_{C-F} = 9.5 Hz), 129.39, 125.78 (d, J_{C-F} = 3.5 Hz), 121.43 (d, J_{C-F} = 17.6 Hz), 114.61 (d, J_{C-F} = 22.5 Hz), 14.96, 11.55;

^{19}F NMR ($CDCl_3$, 376 MHz) δ -109.96 (s, 1F);

LRMS: m/z calcd for $C_{11}H_9ClFNS$ (M+H): 242, found: 242;

Anal. Calcd for $C_{11}H_9ClFNS$: Elemental Analysis: C, 54.66; H, 3.75; N, 5.79; Found: C, 54.71; H, 3.80; N, 5.72.

2-(2,6-difluorophenyl)-4,5-dimethylthiazole (3i)



Flash chromatography (petroleum ether/ ethyl acetate, 50/1); TLC R_f 0.35 (petroleum ether/ ethyl acetate, 12/1); Yield: 58%, yellow oil;

1H NMR ($CDCl_3$, 400 MHz) δ 7.32 (t, J = 8.0 Hz, 1 H), 7.00 (d, J = 8.4 Hz, 2 H), 2.43 (s, 6 H);

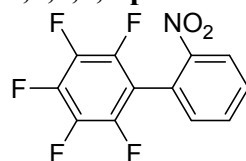
^{13}C NMR ($CDCl_3$, 100 MHz) δ 160.30 (dd, J_{C-F} = 252.2 Hz, 5.9 Hz, 2 C), 150.35 (t, J_{C-F} = 3.3 Hz), 149.23, 130.54 (t, J_{C-F} = 10.5 Hz), 129.00 (m), 112.26 (m, 3 C), 15.01, 11.42;

^{19}F NMR ($CDCl_3$, 376 MHz) δ -111.24 (s, 2 F);

LRMS: m/z calcd for $C_{11}H_9F_2NS$ (M+H): 226, found: 226;

Anal. Calcd for $C_{11}H_9F_2NS$: Elemental Analysis: C, 58.65; H, 4.03; N, 6.22; Found: C, 58.71; H, 4.09; N, 6.14.

2,3,4,5,6-pentafluoro-2'-nitro-1,1'-biphenyl (5a) ^[6]



Flash chromatography (petroleum ether/ ethyl acetate, 50/1); TLC R_f 0.55 (petroleum ether/

ethyl acetate, 12/1); Yield: 58%, yellow crystals, 82-84°C;

^1H NMR (CDCl_3 , 400 MHz) δ 8.18 (d, $J = 8.4$ Hz, 1 H), 7.71 (t, $J = 8.0$ Hz, 1 H), 7.63 (t, $J = 8.0$ Hz, 1 H), 7.38 (d, $J = 7.2$ Hz, 1 H);

^{13}C NMR (CDCl_3 , 100 MHz) δ 148.34, 144.98-142.49 (m, 2 C), 140.02 (m), 139.01-136.55 (m, 2 C), 133.81, 133.09, 130.96, 125.55, 121.51, 112.65 (dt, $J_{\text{C-F}} = 18.3$ Hz, $J_{\text{C-F}} = 4.2$ Hz);

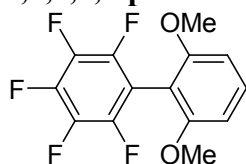
^{19}F NMR (CDCl_3 , 376 MHz) δ -141.64 (dd, $J_{\text{F}} = 22.6$ Hz, 7.5 Hz, 2 F), -153.71 (t, $J_{\text{F}} = 21.0$ Hz, 1 F), -161.73- -161.86 (m, 2 F);

LRMS: m/z calcd for $\text{C}_{12}\text{H}_4\text{F}_5\text{NO}_2$ (M+H): 290, found: 290;

Anal. Calcd for $\text{C}_{12}\text{H}_4\text{F}_5\text{NO}_2$: Elemental Analysis: C, 49.84; H, 1.39; N, 4.84; Found: C, 49.90; H, 1.48; N, 4.73;

IR (KBr) ν (cm^{-1}): 3336, 2924, 1656, 1575, 1524, 1439, 1349, 1094, 1058, 988, 744.

2,3,4,5,6-pentafluoro-2',6'-dimethoxy-1,1'-biphenyl (5b) ^[7]



Flash chromatography (petroleum ether/ ethyl acetate, 100/1); TLC R_f 0.65 (petroleum ether/ ethyl acetate, 12/1); Yield: 51%, yellow crystals, m.p.: 81-83°C;

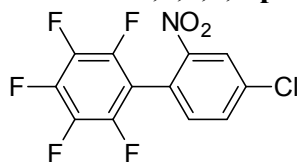
^1H NMR (CDCl_3 , 400 MHz) δ 7.14 (t, $J = 8.4$ Hz, 1 H), 6.60 (d, $J = 10.0$ Hz, 2 H), 3.82 (d, $J = 30.8$ Hz, 6 H);

^{19}F NMR (CDCl_3 , 376 MHz) δ -140.74 (dd, $J_{\text{F}} = 23.7$ Hz, 7.9 Hz, 2 F), -156.98 (t, $J_{\text{F}} = 21.0$ Hz, 1 F), -163.62- -163.76 (m, 2 F);

LRMS: m/z calcd for $\text{C}_{14}\text{H}_9\text{F}_5\text{O}_2$ (M+H): 305, found: 305;

Anal. Calcd for $\text{C}_{14}\text{H}_9\text{F}_5\text{O}_2$: Elemental Analysis: C, 55.27; H, 2.98; Found: C, 55.33; H, 3.06.

4'-chloro-2,3,4,5,6-pentafluoro-2'-nitro-1,1'-biphenyl (5c)



Flash chromatography (petroleum ether/ ethyl acetate, 50/1); TLC R_f 0.60 (petroleum ether/ ethyl acetate, 12/1); Yield: 53%, yellow oil;

^1H NMR (CDCl_3 , 400 MHz) δ 8.19 (s, 1 H), 7.75 (d, $J = 6.4$ Hz, 1 H), 7.38 (d, $J = 8.4$ Hz, 1 H);

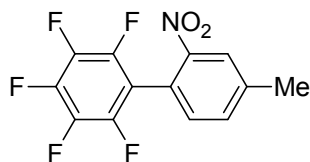
^{13}C NMR (CDCl_3 , 100 MHz) δ 148.86, 145.18-142.89 (m, 2 C), 140.37 (m), 139.24-136.72 (m, 2 C), 137.32, 134.23, 134.09, 126.08, 120.02, 111.79 (dt, $J_{\text{C-F}} = 20.4$ Hz, $J_{\text{C-F}} = 4.2$ Hz);

^{19}F NMR (CDCl_3 , 376 MHz) δ -141.37 (dd, $J_{\text{F}} = 22.2$ Hz, 7.2 Hz, 2 F), -152.80 (t, $J_{\text{F}} = 21.0$ Hz, 1 F), -161.26- -161.40 (m, 2 F);

LRMS: m/z calcd for $\text{C}_{12}\text{H}_3\text{ClF}_5\text{NO}_2$ (M+H): 324, found: 324;

Anal. Calcd for $\text{C}_{12}\text{H}_3\text{ClF}_5\text{NO}_2$: Elemental Analysis: C, 44.94; H, 0.93; N, 4.33; Found: C, 44.99; H, 1.00; N, 4.21.

2,3,4,5,6-pentafluoro-4'-methyl-2'-nitro-1,1'-biphenyl (5d)



Flash chromatography (petroleum ether/ ethyl acetate, 50/1); TLC R_f 0.55 (petroleum ether/ ethyl acetate, 12/1); Yield: 55%, yellow crystals, m.p.: 80-82°C;

^1H NMR (CDCl_3 , 400 MHz) δ 8.05 (s, 1 H), 7.57 (d, $J = 7.6$ Hz, 1 H), 7.32 (d, $J = 8.0$ Hz, 1 H), 2.54 (s, 3 H);

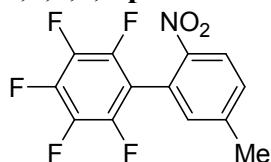
^{13}C NMR (CDCl_3 , 100 MHz) δ 148.29, 145.26-142.76 (m, 2 C), 142.49-140.00 (m), 142.10, 139.11-136.60 (m, 2 C), 134.47, 132.92, 126.08, 118.58, 112.89 (dt, $J_{\text{C-F}} = 18.4$ Hz, $J_{\text{C-F}} = 4.2$ Hz), 21.21;

^{19}F NMR (CDCl_3 , 376 MHz) δ -141.31 (dd, $J_{\text{F}} = 22.6$ Hz, 7.5 Hz, 2 F), -153.83 (t, $J_{\text{F}} = 21.0$ Hz, 1F), -161.61- -161.75 (m, 2 F);

LRMS: m/z calcd for $\text{C}_{13}\text{H}_6\text{F}_5\text{NO}_2$ (M+H): 304, found: 304;

Anal. Calcd for $\text{C}_{13}\text{H}_6\text{F}_5\text{NO}_2$: Elemental Analysis: C, 51.50; H, 1.99; N, 4.62; Found: C, 51.58; H, 2.06; N, 4.52.

2,3,4,5,6-pentafluoro-5'-methyl-2'-nitro-1,1'-biphenyl (5e)



Flash chromatography (petroleum ether/ ethyl acetate, 50/1); TLC R_f 0.55 (petroleum ether/ ethyl acetate, 12/1); Yield: 49%, yellow crystals, m.p.: 80-82°C;

^1H NMR (CDCl_3 , 400 MHz) δ 8.10 (d, $J = 8.4$ Hz, 1 H), 7.40 (d, $J = 8.4$ Hz, 1 H), 7.15 (s, 1 H), 2.44(s, 3 H);

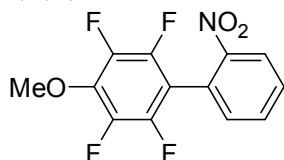
^{13}C NMR (CDCl_3 , 100 MHz) δ 146.16, 145.51, 145.15-142.63 (m, 2 C), 140.02 (m), 139.10-136.59 (m, 2 C), 133.68, 131.55, 125.80, 121.60, 113.16 (dt, $J_{\text{C-F}} = 20.4$ Hz, $J_{\text{C-F}} = 4.2$ Hz), 21.65;

^{19}F NMR (CDCl_3 , 376 MHz) δ -141.66 (dd, $J_{\text{F}} = 22.6$ Hz, 7.5 Hz, 2 F), -154.18 (t, $J_{\text{F}} = 21.0$ Hz, 1F), -162.00- -162.14 (m, 2 F);

LRMS: m/z calcd for $\text{C}_{13}\text{H}_6\text{F}_5\text{NO}_2$ (M+H): 304, found: 304;

Anal. Calcd for $\text{C}_{13}\text{H}_6\text{F}_5\text{NO}_2$: Elemental Analysis: C, 51.50; H, 1.99; N, 4.62; Found: C, 51.56; H, 2.07; N, 4.54.

2,3,5,6-tetrafluoro-4-methoxy-2'-nitro-1,1'-biphenyl (5f)



Flash chromatography (petroleum ether/ ethyl acetate, 50/1); TLC R_f 0.50 (petroleum ether/ ethyl acetate, 12/1); Yield: 63%, yellow crystals, m.p.: 83-85°C;

^1H NMR (CDCl_3 , 400 MHz) δ 8.19 (d, $J = 8.0$ Hz, 1 H), 7.74 (t, $J = 7.6$ Hz, 1 H), 7.65 (t, $J = 7.6$ Hz, 1 H), 7.45 (d, $J = 7.6$ Hz, 1 H), 4.14 (s, 3 H);

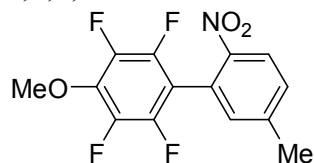
^{13}C NMR (CDCl_3 , 100 MHz) δ 148.74, 145.29-142.84 (m, 2 C), 141.08 (ddt, $J_{\text{C-F}} = 246.2$ Hz, 15.0 Hz, 4.3 Hz, 2 C), 138.74 (m), 133.69, 133.39, 130.62, 125.47, 122.43, 110.61 (t, $J_{\text{C-F}} = 18.4$ Hz), 62.40 (t, $J_{\text{C-F}} = 3.8$ Hz);

^{19}F NMR (CDCl_3 , 376 MHz) δ -143.74 (dd, $J_{\text{F}} = 21.0$ Hz, 7.5 Hz, 2 F), -158.10 (dd, $J_{\text{F}} = 21.4$ Hz, 7.5 Hz, 2 F);

LRMS: m/z calcd for $\text{C}_{13}\text{H}_7\text{F}_4\text{NO}_3$ (M+H): 302, found: 302;

IR (KBr) ν (cm^{-1}): 2960, 2857, 2409, 1988, 1843, 1727, 1656, 1574, 1483, 1399, 1351, 1100, 882, 793.

2,3,5,6-tetrafluoro-4-methoxy-5'-methyl-2'-nitro-1,1'-biphenyl (5g)



Flash chromatography (petroleum ether/ ethyl acetate, 50/1); TLC R_f 0.50 (petroleum ether/ ethyl acetate, 12/1); Yield: 57%, yellow crystals, m.p.: 85-87°C;

^1H NMR (CDCl_3 , 400 MHz) δ 8.12 (d, $J = 8.0$ Hz, 1 H), 7.42 (d, $J = 8.0$ Hz, 1 H), 7.23 (s, 1 H), 4.14 (s, 3 H), 2.49 (s, 3 H);

^{13}C NMR (CDCl_3 , 100 MHz) δ 146.40, 145.26-142.83 (m, 2 C), 145.17, 141.06 (ddt, $J_{\text{C-F}} = 246.0$ Hz, 15.1 Hz, 4.4 Hz, 2 C), 138.50 (m), 133.83, 131.09, 125.58, 122.39, 111.04 (t, $J_{\text{C-F}} = 18.5$ Hz), 62.40 (t, $J_{\text{C-F}} = 3.7$ Hz), 21.59;

^{19}F NMR (CDCl_3 , 376 MHz) δ -143.71 (dd, $J_{\text{F}} = 21.4$ Hz, 7.9 Hz, 2 F), -158.29 (dd, $J_{\text{F}} = 21.4$ Hz, 7.9 Hz, 2 F);

LRMS: m/z calcd for $\text{C}_{14}\text{H}_9\text{F}_4\text{NO}_3$ (M+H): 316, found: 316;

Anal. Calcd for $\text{C}_{14}\text{H}_9\text{F}_4\text{NO}_3$: Elemental Analysis: C, 53.34; H, 2.88; N, 4.44; Found: C, 53.43; H, 2.91; N, 4.36;

IR (KBr) ν (cm^{-1}): 3350, 2960, 1654, 1589, 1526, 1494, 1428, 1345, 1261, 1196, 1109, 989, 843, 780.

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