Supporting Information

Rhodium(I)-catalyzed Carboxylation of Aryl- and Alkenylboronic Esters with CO₂

*Kazutoshi Ukai, Masao Aoki, Jun Takaya, and Nobuharu Iwasawa**

Department of Chemistry, Tokyo Institute of Technology, O-okayama, Meguro-ku,

Tokyo 152-8551, Japan

General. ¹H and ¹³C-NMR spectra were recorded on a JEOL Lambda-400 (400 MHz for ¹H and 100 MHz for ¹³C) or a JEOL AL-400 (400 MHz for ¹H and 100 MHz for ¹³C) spectrometer using CHCl₃ (¹H, δ = 7.26) and CDCl₃ (¹³C, δ = 77.0) as an internal standard. IR spectra were recorded on an FT/IR-460 plus (JASCO Co., Ltd.). Flash column chromatography was conducted on silica gel 60N (Kanto Chemical Co., Inc.). Commercially available anhydrous dioxane was employed for the reaction (Dehydrated, Wako Pure Chemical Industries, Ltd.). Tetrahydrofuran was freshly distilled from benzophenone ketyl, and all other solvents were distilled according to the usual procedures and stored over Molecular Sieves. Cesium fluoride (Wako Pure Chemical Industries, Ltd.) was dried by heating at 150 °C for 2 days in vacuo.

General procedure for preparation of arylboronic esters:

Arylboronic esters **1b**, **1c**, **1e**, **1f**, **1p**, **1r**, **1s**, **1t** were prepared from the corresponding bromobenzene derivatives according to the literature procedure, using 2,2-dimethylpropan-1,3-diol instead of ethylene glycol.¹

1t² was already reported in the literatures. **1f** is commercially available (Wako Pure Chemical Industries, Ltd.).

5,5-Dimethyl-2-(4-*t*-butylphenyl)-1,3,2-dioxaborinane (1b**)**

IR (KBr) 2956, 2896, 1607, 1478, 1419, 1322, 1249, 1140, 1019, 835, 651 cm⁻¹;

¹H NMR (CDCl₃) (400 MHz): δ = 1.02 (6H, s), 1.32 (9H, s), 3.76 (4H, s), 7.39 (2H, d, *J* = 8.3 Hz), 7.74 (2H, d, *J* = 8.3 Hz);

¹³C NMR (CDCl₃) (100 MHz): δ = 22.0, 31.3, 31.9, 34.8, 72.3, 124.5, 133.7, 153.6;

Anal. Calcd for C₁₅H₂₃BO₂: C 73.19, H 9.42. Found: C 73.22, H 9.36.

5,5-Dimethyl-2-[4-(2-methylprop-1-en-1-yl)phenyl]-1,3,2-dioxaborinane (1c**)**

IR (KBr) 2968, 2902, 1604, 1478, 1418, 1379, 1308, 1250, 1178, 1132, 873, 713, 642 cm^{-1} ;

^1H NMR (CDCl_3) (400 MHz): δ = 1.03 (6H, s), 1.88 (3H, s), 1.91 (3H, s), 3.77 (4H, s), 6.29 (1H, s), 7.23 (2H, d, J = 8.0 Hz), 7.76 (2H, d, J = 8.0 Hz);

^{13}C NMR (CDCl_3) (100 MHz): δ = 19.6, 22.0, 27.0, 31.9, 72.3, 125.3, 127.9, 133.5, 136.0, 140.9;

Anal. Calcd for $\text{C}_{15}\text{H}_{21}\text{BO}_2$: C 73.79, H 8.67. Found: C 74.08, H 8.60.

5,5-Dimethyl-2-(4-trifluoromethylphenyl)-1,3,2-dioxaborinane (1e)

IR (KBr) 2964, 2909, 2876, 1519, 1483, 1425, 1313, 1253, 1161, 1119, 1065, 1018, 841, 715, 645 cm^{-1} ;

^1H NMR (CDCl_3) (400 MHz): δ = 1.03 (6H, s), 3.79 (4H, s), 7.60 (2H, d, J = 8.0 Hz), 7.91 (2H, d, J = 8.0 Hz);

^{13}C NMR (CDCl_3) (100 MHz): δ = 21.9, 32.0, 72.4, 124.1 (J_{CF} = 3.9 Hz), 124.2 (J_{CF} = 271 Hz), 132.2 (J_{CF} = 32 Hz), 134.0;

Anal. Calcd for $\text{C}_{12}\text{H}_{14}\text{BF}_3\text{O}_2$: C 55.85, H 5.47. Found: C 55.83, H 5.51.

5,5-Dimethyl-2-(2-methylphenyl)-1,3,2-dioxaborinane (1p)

IR (KBr) 2961, 2931, 2888, 1599, 1570, 1477, 1442, 1410, 1377, 1306, 1277, 1244, 1137, 1107, 814, 761, 730, 708, 649 cm^{-1} ;

^1H NMR (CDCl_3) (400 MHz): δ = 1.04 (6H, s), 2.53 (3H, s), 3.78 (4H, s), 7.16 (2H, t, J = 7.6 Hz), 7.29 (1H, t, J = 7.6 Hz), 7.74 (1H, d, J = 7.6 Hz);

^{13}C NMR (CDCl_3) (100 MHz): δ = 21.9, 22.4, 31.7, 72.3, 124.6, 129.85, 129.94, 134.7, 143.8;

Anal. Calcd for $\text{C}_{12}\text{H}_{17}\text{BO}_2$: C 70.63, H 8.40. Found: C 70.40, H 8.34.

5,5-Dimethyl-2-(3,5-dimethylphenyl)-1,3,2-dioxaborinane (1r)

IR (KBr) 2959, 2913, 1599, 1480, 1385, 1330, 1241, 1137, 863, 711, 689 cm^{-1} ;

^1H NMR (CDCl_3) (400 MHz): δ = 1.03 (6H, s), 2.32 (6H, s), 3.77 (4H, s), 7.08 (1H, s), 7.43 (2H, s);

^{13}C NMR (CDCl_3) (100 MHz): δ = 21.3, 22.0, 31.9, 72.3, 131.4, 132.3, 136.8;

Anal. Calcd for $\text{C}_{13}\text{H}_{19}\text{BO}_2$: C 71.59, H 8.78. Found: C 71.82, H 8.92.

5,5-Dimethyl-2-(2-naphthyl)-1,3,2-dioxaborinane (1s)

IR (KBr) 2956, 2933, 2899, 2872, 1481, 1416, 1312, 1251, 1238, 1195, 1136, 1119, 829, 743, 700, 680, 481 cm^{-1} ;

^1H NMR (CDCl_3) (400 MHz): δ = 1.06 (6H, s), 3.84 (4H, s), 7.43-7.53 (2H, m), 7.79-7.91 (4H, m), 8.36 (1H, s);

^{13}C NMR (CDCl_3) (100 MHz): δ = 21.9, 32.0, 72.4, 125.6, 126.6, 126.8, 127.6, 128.7, 129.9, 132.9, 134.8, 135.0;

Anal. Calcd for $\text{C}_{15}\text{H}_{17}\text{BO}_2$: C 75.03, H 7.14. Found: C 75.26, H 7.28.

Arylboronic esters **1a**, **1d**, **1h**, **1i**, **1n**, **1o**, **1q** and **1u** were prepared by esterification of the corresponding commercially available arylboronic acids: To arylboronic acid (3.0 mmol) and 2,2-dimethylpropan-1,3-diol (3.3 mmol) in CH₂Cl₂ (6.0 ml) was added MgSO₄ (4.0 g) and the mixture was stirred at room temperature overnight. After filtration, the solvent was evaporated and the crude product was purified by silica gel chromatography (5~10% ethyl acetate in hexanes) or recrystallization from hexane and ethyl acetate.

1a², **1d**², **1i**², **1n**², and **1q**³ were already reported in the literatures.

5,5-Dimethyl-2-(4-methoxycarbonylphenyl)-1,3,2-dioxaborinane (1h)

IR (KBr) 2957, 1721, 1479, 1425, 1308, 1277, 1254, 1130, 1020, 728, 710, 644 cm⁻¹;

¹H NMR (CDCl₃) (400 MHz): δ = 1.03 (6H, s), 3.78 (4H, s), 3.91 (3H, s), 7.86 (2H, d, *J* = 8.3 Hz), 8.00 (2H, d, *J* = 8.3 Hz);

¹³C NMR (CDCl₃) (100 MHz): δ = 21.9, 31.9, 52.1, 72.4, 128.4, 131.7, 133.7, 167.2;

Anal. Calcd for C₁₃H₁₇BO₄: C 62.94, H 6.91. Found: C 62.71, H 6.85.

5,5-Dimethyl-2-(3-cyanophenyl)-1,3,2-dioxaborinane (1o)

IR (KBr) 2963, 2874, 2227, 1598, 1481, 1431, 1378, 1308, 1256, 1184, 1121, 980, 809, 699, 661, 556, 499 cm⁻¹;

¹H NMR (CDCl₃) (400 MHz): δ = 1.02 (6H, s), 3.77 (4H, s), 7.43 (1H, t, *J* = 7.6 Hz), 7.67 (1H, d, *J* = 7.6 Hz), 7.99 (1H, d, *J* = 7.6 Hz), 8.08 (1H, s);

¹³C NMR (CDCl₃) (100 MHz): δ = 21.8, 31.9, 72.4, 111.7, 119.1, 128.1, 133.7, 137.5, 137.8;

Anal. Calcd for C₁₂H₁₄BNO₂: C 67.02, H 6.56, N 6.51. Found: C 67.16, H 6.68, N 6.36.

5,5-Dimethyl-2-(3-thienyl)-1,3,2-dioxaborinane (1u)

IR (KBr) 2954, 1518, 1479, 1420, 1290, 1253, 1123, 848, 801, 694, 677 cm⁻¹;

¹H NMR (CDCl₃) (400 MHz): δ = 1.02 (6H, s), 3.75 (4H, s), 7.31 (1H, dd, *J* = 2.6 Hz, 4.8 Hz), 7.38 (1H, dd, *J* = 1.0 Hz, 4.8 Hz), 7.84 (1H, dd, *J* = 1.0 Hz, 2.6 Hz);

¹³C NMR (CDCl₃) (100 MHz): δ = 21.9, 31.9, 72.2, 124.9, 131.6, 134.8;

Anal. Calcd for C₉H₁₃BO₂S: C 55.13, H 6.68, S 16.35. Found: C 55.14, H 6.95, S 16.30.

Arylboronic esters **1g**, **1j**, **1k**, **1l**, and **1m** were prepared according to the procedure of cross coupling reaction of arylbromide and diboron reagent⁴ using dioxane as the solvent instead of DMSO to prevent hydrolysis.

5,5-Dimethyl-2-(4-pivaloyloxyphenyl)-1,3,2-dioxaborinane (1g)

IR (KBr) 2967, 2934, 2874, 1746, 1597, 1477, 1422, 1379, 1346, 1320, 1277, 1250, 1199, 1168, 1115, 1014, 900, 814, 642 cm^{-1} ;

^1H NMR (CDCl_3) (400 MHz): δ = 1.02 (6H, s), 1.36 (9H, s), 3.76 (4H, s), 7.04 (2H, d, J = 8.4 Hz), 7.81 (2H, d, J = 8.4 Hz);

^{13}C NMR (CDCl_3) (100 MHz): δ = 22.0, 27.2, 31.9, 39.1, 72.3, 120.5, 135.1, 153.2, 176.7;

Anal. Calcd for $\text{C}_{16}\text{H}_{23}\text{BO}_4$: C 66.23, H 7.99. Found: C 66.17, H 8.19.

5,5-Dimethyl-2-(4-cyanomethylphenyl)-1,3,2-dioxaborinane (1j)

IR (KBr) 2956, 2909, 2246, 1610, 1479, 1424, 1377, 1347, 1324, 1252, 1136, 1020, 812, 720, 709, 641 cm^{-1} ;

^1H NMR (CDCl_3) (400 MHz): δ = 1.02 (6H, s), 3.73 (2H, s), 3.77 (4H, s), 7.30 (2H, d, J = 7.6 Hz), 7.81 (2H, d, J = 7.6 Hz);

^{13}C NMR (CDCl_3) (100 MHz): δ = 21.8, 23.6, 31.8, 72.2, 117.6, 126.9, 132.0, 134.5;

Anal. Calcd for $\text{C}_{13}\text{H}_{16}\text{BNO}_2$: C 68.16, H 7.04, N 6.11. Found: C 68.39, H 7.13, N 6.36.

5,5-Dimethyl-2-[4-(*t*-butoxycarbonylamino)phenyl]-1,3,2-dioxaborinane (1k)

IR (KBr) 3345, 2965, 1697, 1584, 1524, 1507, 1310, 1248, 1158, 838, 646 cm^{-1} ;

^1H NMR (CDCl_3) (400 MHz): δ = 1.01 (6H, s), 1.51 (9H, s), 3.75 (4H, s), 6.58 (1H, brs), 7.34 (2H, d, J = 8.0 Hz), 7.72 (2H, d, J = 8.0 Hz);

^{13}C NMR (CDCl_3) (100 MHz): δ = 21.9, 28.3, 31.8, 72.2, 80.5, 117.2, 134.8, 140.5, 152.5;

Anal. Calcd for $\text{C}_{16}\text{H}_{24}\text{BNO}_4$: C 62.97, H 7.93, N 4.59. Found: C 62.88, H 7.90, N 4.40.

5,5-Dimethyl-2-[4-(3-oxobutyl)phenyl]-1,3,2-dioxaborinane (1l)

IR (KBr) 2959, 1720, 1609, 1479, 1308, 1131, 815, 644 cm^{-1} ;

^1H NMR (CDCl_3) (400 MHz): δ = 1.02 (6H, s), 2.13 (3H, s), 2.75 (2H, t, J = 7.0 Hz), 2.90 (2H, t, J = 7.0 Hz), 3.76 (4H, s), 7.18 (2H, d, J = 7.2 Hz), 7.72 (2H, d, J = 7.2 Hz);

^{13}C NMR (CDCl_3) (100 MHz): δ = 21.9, 29.9, 30.1, 31.9, 45.1, 72.3, 127.5, 134.0, 143.5, 207.7;

Anal. Calcd for $\text{C}_{15}\text{H}_{21}\text{BO}_3$: C 69.26, H 8.14. Found: C 69.50, H 8.43.

5,5-Dimethyl-2-[4-{2-(methoxycarbonyl)ethyl}phenyl]-1,3,2-dioxaborinane (1m)

IR (KBr) 2950, 1736, 1478, 1318, 1134, 836, 648 cm^{-1} ;

^1H NMR (CDCl_3) (400 MHz): δ = 1.01 (6H, s), 2.63 (2H, t, J = 7.7 Hz), 2.96 (2H, t, J = 7.7 Hz), 3.66 (3H, s), 3.76 (4H, s), 7.19 (2H, d, J = 7.5 Hz), 7.72 (2H, d, J = 7.5 Hz);

^{13}C NMR (CDCl_3) (100 MHz): δ = 21.9, 31.1, 31.9, 35.5, 51.6, 72.3, 127.6, 134.1, 143.1, 173.3;

Anal. Calcd for C₁₅H₂₁BO₄: C 65.24, H 7.67. Found: C 65.48, H 7.78.

Typical procedure for preparation of boronic esters bearing aromatic heterocycles:

1-Methyl-2-(5,5-dimethyl-1,3,2-dioxaborinan-2-yl)-1H-indole (1w)

To a stirred solution of 2-lithio-*N*-methylindole (3.520 mM) in THF⁵ was added triethylborate (0.80 ml, 4.70 mM) dropwise over 5 min at -78 °C. The mixture was stirred for 5 min and allowed to warm to room temperature. After 1.5 h, the mixture was treated with 1M HCl and then extracted with Et₂O three times. The combined organic layer was treated with Na₂SO₄ and 2,2-dimethyl-1,3-propanediol (0.47 g, 5.33 mM) and stirred overnight for the esterification of the resulting *N*-methylindolylboronic acid. Removal of the solvent under reduced pressure afforded white solids, which were recrystallized from CH₂Cl₂-Et₂O-hexane to give the product (505 mg, 2.08 mM) in 59% yield. (Benzofuran-2-yl)-5,5-dimethyl-1,3,2-dioxaborinane (**1v**) was prepared according to the same procedure.

IR (KBr) 2956, 1516, 1383, 1145, 1112, 810, 737, 688 cm⁻¹;

¹H NMR (CDCl₃) (400 MHz): δ = 1.06 (6H, s), 3.80 (4H, s), 3.97 (3H, s), 7.05-7.10 (2H, m), 7.22-7.27 (1H, m), 7.33-7.36 (1H, m), 7.63-7.66 (1H, m);

¹³C NMR (CDCl₃) (100 MHz): δ = 22.0, 32.0, 32.1, 72.2, 109.5, 113.0, 119.0, 121.3, 122.6, 127.7, 140.1;

Anal. Calcd for C₁₄H₁₈BNO₂: C 69.17, H 7.46, N 5.76. Found: C 68.97, H 7.66, N 5.51.

(Benzofuran-2-yl)-5,5-dimethyl-1,3,2-dioxaborinane (1v)

IR (KBr) 2956, 1567, 1482, 1428, 1297, 1261, 1163, 1104, 741, 689 cm⁻¹;

¹H NMR (CDCl₃) (400 MHz): δ = 1.06 (6H, s), 3.82 (4H, s), 7.22 (1H, t, *J* = 7.6 Hz), 7.30-7.35 (2H, m), 7.56 (1H, d, *J* = 8.0 Hz), 7.62 (1H, d, *J* = 7.6 Hz);

¹³C NMR (CDCl₃) (100 MHz): δ = 21.9, 32.2, 72.4, 111.8, 117.7, 121.6, 122.5, 125.4, 127.7, 157.2;

Anal. Calcd for C₁₃H₁₅BNO₃: C 67.87, H 6.57. Found: C 67.65, H 6.33.

General procedure for preparation of alkenylboronic esters:

Alkenylboronic esters were prepared by hydroboration of alkynes⁶ followed by ester exchange. A mixture of an alkyne (10 mmol) and catecholborane (10 mmol) was stirred for 1 ~ 4 hours at 80 °C, and then water (4.0 ml) was added. After the mixture was stirred for 1 hour at 80 °C, organic material was extracted with ether three times and the combined ether extract was washed with water three times. After the organic layer was dried over MgSO₄, the solvent was removed under reduced pressure. To the mixture of the crude product and 2,2-dimethylpropan-1,3-diol (22 mmol) was added toluene (30 ml) and the mixture was heated to reflux with the azeotropic removal of water. Organic layer was extracted with ether and the combined ether extract was washed with water three times. After the organic layer was dried over MgSO₄, the solvent was removed under reduced pressure. The residue was purified by silica gel

chromatography (5~10% ethyl acetate in hexanes) and/or recrystallization from hexane to give the corresponding alkenylboronic ester.

5,5-Dimethyl-2-(β -phenylethenyl)-1,3,2-dioxaborinane (3a) was already reported in the literature.⁷

5,5-Dimethyl-2-[β -(4-methoxyphenylethenyl)]-1,3,2-dioxaborinane (3b)

IR (KBr) 2960, 1604, 1574, 1512, 1475, 1415, 1379, 1279, 1109, 1086, 1033, 1000, 814, 744, 632, 566, 515 cm⁻¹;

¹H NMR (CDCl₃) (400 MHz): δ = 1.00 (6H, s), 3.69 (4H, s), 3.79 (3H, s), 5.97 (1H, d, J = 18.4 Hz), 6.86 (2H, d, J = 8.4 Hz), 7.30 (1H, d, J = 18.4 Hz), 7.44 (2H, d, J = 8.4 Hz);

¹³C NMR (CDCl₃) (100 MHz): δ = 21.9, 31.8, 55.2, 72.1, 113.8, 128.2, 130.6, 146.5, 159.8;

Anal. Calcd for C₁₄H₁₉BO₃: C 68.32, H 7.78. Found: C 68.43, H 7.95.

5,5-Dimethyl-2-(oct-1-en-1-yl)-1,3,2-dioxaborinane (3c)

IR (neat) 2959, 2927, 1638, 1476, 1414, 1378, 1326, 1255, 1092, 814 cm⁻¹;

¹H NMR (CDCl₃) (400 MHz): δ = 0.87 (3H, t, J = 7.2 Hz), 0.96 (6H, s), 1.20-1.34 (6H, m), 1.34-1.45 (2H, m), 2.08-2.16 (2H, m), 3.63 (4H, s), 5.33 (1H, d, J = 17.6 Hz), 6.54 (1H, dt, J = 17.6, 6.4 Hz);

¹³C NMR (CDCl₃) (100 MHz): δ = 14.2, 21.9, 22.7, 28.5, 29.0, 31.79, 31.81, 35.6, 72.0, 151.9;

Anal. Calcd for C₁₃H₂₅BO₂: C 69.66, H 11.24. Found: C 69.45, H 11.06.

General procedure for rhodium-catalyzed carboxylation of aryl- and alkenylboronic esters with carbon dioxide:

A dioxane suspension (1.7 ml) of [Rh(OH)(cod)]₂ (2.3 mg, 0.005 mmol), dppp (4.6 mg, 0.011 mmol), arylboronic ester (0.16 mmol) and CsF (0.5 mmol, 76 mg) was stirred in a glass tube (ϕ = 1.7 cm, 18 cm) under an atmospheric pressure of CO₂ and then the system was closed. The mixture was heated at 60 °C for 10 ~ 30 hours, and then the reaction was quenched with 1 M HCl aq. Organic layer was extracted with CH₂Cl₂ four times and the combined extract was dried over MgSO₄. After removal of the solvent under reduced pressure, the residue was purified by silica gel chromatography (hexane : CH₂Cl₂ = 7:1 – 0:1, followed by CH₂Cl₂ : ethyl acetate = 9:1) to afford the corresponding carboxylic acid.

Carboxylic acids **2a**, **2b**, **2d** ~ **2j**, **2n** ~ **2v**, **3a** ~ **3c** were commercially available. **2k**,⁸ **2l**,⁹ **2m**,¹⁰ and **2w**¹¹ were reported in the literatures.

4-(2-methylprop-1-en-1-yl)benzoic acid (2c)

IR (KBr) 2974, 2549, 1681, 1604, 1425, 1318, 1289, 1181, 949, 881, 761, 549 cm⁻¹;

¹H NMR (CDCl₃) (400 MHz): δ = 1.91 (3H, s), 1.95 (3H, s), 6.31 (1H, s), 7.32 (2H, d, J = 8.4 Hz), 8.06 (2H, d, J = 8.4

Hz);

^{13}C NMR (CDCl_3) (100 MHz): δ = 19.7, 27.2, 124.5, 126.3, 128.6, 130.0, 138.4, 144.3, 171.8;

Anal. Calcd for $\text{C}_{11}\text{H}_{12}\text{O}_2$: C 74.98, H 6.86. Found: C 74.98, H 7.01.

4-(2,2-dimethoxycarbonylpropan-1-yl)benzoic acid (2k)

IR (KBr) 2953, 2668, 2551, 1737, 1682, 1611, 1431, 1321, 1293, 1237, 1210, 1184, 1112, 861, 761, 707, 545 cm^{-1} ;

^1H NMR (CDCl_3) (400 MHz): δ = 1.37 (3H, s), 3.30 (2H, s), 3.74 (6H, s), 7.22 (2H, d, J = 8.1 Hz), 8.01 (2H, d, J = 8.1 Hz);

^{13}C NMR (CDCl_3) (100 MHz): δ = 19.9, 41.3, 52.6, 54.8, 128.1, 130.1, 130.3, 142.5, 171.8, 172.0;

Anal. Calcd for $\text{C}_{14}\text{H}_{16}\text{O}_6$: C 59.99, H 5.75. Found: C 59.78, H 5.76

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