4,5,6,7-Tetrachlorobenzo[*d*][1,3,2]dioxaborol-2-ol as an Effective Catalyst for the Amide Condensation of Sterically Demanding Carboxylic Acids

Toshikatsu Maki,[†] Kazuaki Ishihara,^{*,†} and Hisashi Yamamoto^{*,‡}

[†]*Graduate School of Engineering, Nagoya University, Furo-cho, Chikusa, Nagoya 464-8603, Japan*

[‡]*Department of Chemistry, The University of Chicago, 5735 S. Ellis Avenue, Chicago, Illinois 60637*

ishihara@cc.nagoya-u.ac.jp; yamamoto@uchicago.edu

General Methods. Infrared (IR) spectra were recorded on a JASCO FT/IR 460 plus spectrometer. ¹H NMR spectra were measured on a Varian Gemini-2000 spectrometer (300 MHz) at ambient temperature. Data were recorded as follows: chemical shift in ppm from internal tetramethylsilane on the δ scale, multiplicity (s = singlet; d = doublet; t = triplet; m = multiplet), coupling constant (Hz), integration, and assignment. ¹³C NMR spectra were measured on Varian Gemini-2000 (75 MHz) spectrometer. Chemical shifts were recorded in ppm from the solvent resonance employed as the internal standard (deuteriochloroform at 77.00 ppm). All experiments were carried out under an atmosphere of dry nitrogen. For thin-layer chromatography (TLC) analysis throughout this work, Merck precoated TLC plates (silica gel 60 GF₂₅₄ 0.25 mm) were used. The products were purified by column chromatography on silica gel (E. Merck Art. 9385). High resolution mass spectral analysis (HRMS) was performed at Chemical Instrument Center, Nagoya University. In experiments that required dry solvent, ether and tetrahydrofuran (THF) were purchased from Aldrich or Wako as the “anhydrous” and stored over 4A molecular sieves. Hexane and toluene were freshly distilled from calcium hydride. Other simple chemicals were analytical-grade and obtained commercially.

***In situ* Preparation of 4,5,6,7-Tetrachlorobenzo[*d*][1,3,2]dioxaborol-2-ol (4b) (Tables 1 and 2).** A dry, 20-mL round-bottom flask equipped with a Teflon-coated magnetic stirring bar and a Dean-Stark apparatus surmounted by a reflux condenser was charged with B(OH)₃ (6.1 mg, 0.10 mmol) and tetrachlorocatechol (25 mg, 0.10 mmol) in toluene (10 mL). The mixture was

brought to heat at azeotropic reflux with the removal of water for 3 hours to prepare a solution of **4b**. After 3 hours, B(OH)₃ [¹¹B NMR (DMSO-*d*₆, 96 MHz) δ 20] was dissolved completely, and the resulting solution was used as catalyst directly in the amide condensation without further purification. ¹¹B NMR (DMSO-*d*₆, 96 MHz) δ 8.7.

Preparation of 4,5,6,7-Tetrachlorobenzo[*d*][1,3,2]dioxaborole (4a) (Tables 2 and 3).¹

Tetrachlorocatechol (2.5 g, 10 mmol) in Et₂O (10 mL) was added slowly to a stirred solution of BH₃•SMe₂ (1.2 mL, 12 mmol, 10 M) in ether (10 mL) at ambient temperature. Gas evolution began immediately. After a few minutes, all volatile components were removed *in vacuo* to yield almost pure light yellow solid, which after sublimation at 100 °C/1 torr gave 2.2 g of pure **4a** (83% yield) as clusters of very fine needles. Mp. 95–100 °C; ¹¹B NMR (CDCl₃, 96 MHz) δ 29.1; ¹³C NMR (CDCl₃, 75 MHz) δ 117.0 (2C), 127.8 (2C), 143.7 (2C).

Typical Procedure for the Amide Condensation Reaction of an Equimolar Mixture of Carboxylic Acids and Amines Catalyzed by 4a (Table 3). A dry, 20-mL round-bottom flask equipped with a Teflon-coated magnetic stirring bar and a Dean-Stark apparatus surmounted by a reflux condenser was charged with carboxylic acids (2.0 mmol), amines (2.0 mmol) and **4a** (26 mg, 0.10 mmol, 5.0 mol%) in toluene or *o*-xylene (10 mL). The mixture was brought to heat at azeotropic reflux with the removal of water. After the reaction was completed, the resulting mixture was cooled to ambient temperature and wash with both aqueous solutions of ammonium chloride and sodium hydrogen carbonate, and the product was extracted with ethyl acetate. The combined organic layers were dried over magnesium sulfate. The solvent was evaporated, and the residue was purified by column chromatography on silica gel to give the desired amides.

***N*-Benzyl-4-phenylbutanamide (5) (entry 1):²** Mp. 83 °C; IR (KBr) 3288, 2921, 1643, 1547, 1215, 696, 494 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz) δ 1.92–2.02 (m, 2H), 2.19 (t, *J* = 6.9 Hz, 2H), 2.64 (t, *J* = 7.5 Hz, 2H), 4.40 (d, *J* = 5.7 Hz, 2H), 5.87 (br, 1H), 7.13–7.35 (m, 10 H); ¹³C NMR (CDCl₃, 75 MHz) δ 27.0, 35.0, 35.5, 43.1, 125.7, 127.1, 127.4 (2C), 128.1 (2C), 128.2 (2C), 128.3 (2C), 138.3, 141.3, 172.7.

***N*-Benzyl-*N*-methyl-4-phenylbutanamide (6) (entry 2):³** Mp. 40 °C; IR (KBr) 3056, 2925,

1619, 1406, 1267, 1068, 731 cm^{-1} ; ^1H NMR ($\text{DMSO-}d_6$, 300 MHz) δ 2.87 (s, 3H), 4.60 (s, 2H), 7.26–7.45 (m, 10H); ^{13}C NMR ($\text{DMSO-}d_6$, 75 MHz, 90 $^\circ\text{C}$) δ 34.6, 52.2, 126.4 (2C), 126.9, 127.0 (2C), 128.0 (2C), 128.3 (2C), 129.0, 136.4, 137.0, 170.4.

***N*-Benzylbenzamide (7) (entry 3):**⁴ Mp. 104 $^\circ\text{C}$; IR (KBr) 3319, 3060, 1638, 1541, 1418, 1314, 1259, 989, 727, 692 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz) δ 4.65 (d, J = 5.4 Hz, 2H), 6.48 (br, 1H), 7.28–7.82 (m, 10H); ^{13}C NMR (CDCl_3 , 75 MHz) δ 44.0, 126.9 (2C), 127.5, 127.8 (2C), 128.5 (2C), 128.7 (2C), 131.5, 134.3, 138.1, 167.3.

***N*-Benzyl-*N*-methylbenzamide (8) (entry 4):**⁵ Liquid; IR (film) 3060, 3026, 2925, 1646, 1495, 1453, 1403, 1356, 1117, 745, 700 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz) δ 2.03 (m, 2H), 2.38 (t, J = 7.8 Hz, 2H), 2.66 (t, J = 7.8 Hz, 0.8H), 2.71 (t, J = 7.8 Hz, 1.2 H), 2.85 (s, 1.8 H), 2.95 (s, 1.2 H), 4.47 (s, 0.8H), 4.59 (s, 1.2 H), 7.10–7.28 (m, 10H); ^{13}C NMR (CDCl_3 , 75 MHz) δ 26.4, 26.6, 32.1, 32.5, 33.8, 34.6, 35.1, 35.2, 50.6, 53.1, 125.7, 126.1, 127.2, 127.4, 127.9, 128.2, 128.3, 128.4, 128.8, 136.6, 137.4, 141.6, 172.7, 173.0. HRMS-FAB (m/z): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{18}\text{H}_{21}\text{NO}$, 268.1701; Found, 268.1692.

***N*-Benzylcyclohexanecarboxamide (9) (entry 5):**² Mp. 113 $^\circ\text{C}$; IR (KBr) 3284, 2926, 1641, 1551, 1219, 696 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz) δ 1.18–1.91 (m, 10H), 2.11 (tt, J = 3.6, 11.4 Hz, 1H), 4.44 (d, J = 5.7 Hz, 2H), 5.75 (br, 1H), 7.25–7.37 (m, 5H); ^{13}C NMR (CDCl_3 , 75 MHz) δ 25.7 (3C), 29.7 (2C), 43.3, 45.5, 127.4, 127.6 (2C), 128.6 (2C), 138.5, 175.9.

***N*-Benzyl-2-ethylbutanamide (10) (entry 6):**⁶ Mp. 82 $^\circ\text{C}$; IR (KBr) 3281, 2961, 2873, 1639, 1546, 1455, 1225, 740, 689 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz) δ 0.91 (t, J = 7.5 Hz, 6H), 1.43–1.73 (m, 4H), 1.83–1.92 (m, 1H), 4.48 (d, J = 6.0 Hz, 2H), 5.74 (br, 1H), 7.26–7.37 (m, 5H); ^{13}C NMR (CDCl_3 , 75 MHz) δ 12.1 (2C), 25.8 (2C), 43.4, 51.5, 127.4, 127.8 (2C), 128.6 (2C), 138.6, 175.6. HRMS-FAB (m/z): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{13}\text{H}_{19}\text{NO}$, 206.1545; Found, 206.1549.

***N*-Benzyl-2-propylpentanamide (11) (entry 7):**⁷ Mp. 90 $^\circ\text{C}$; IR (KBr) 3274, 2958, 2927, 1637, 1554, 1452, 1258, 1222, 736, 689 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz) δ 0.89 (t, J = 7.2 Hz, 6H), 1.20–1.44 (m, 6H), 1.55–1.68 (m, 2H), 2.00–2.10 (m, 1H), 4.43 (d, J = 5.7 Hz, 2H), 5.96 (br, 1H), 7.24–7.35 (m, 5H); ^{13}C NMR (CDCl_3 , 75 MHz) δ 14.1 (2C), 20.8 (2C), 35.3 (2C), 43.4, 47.8, 127.4, 127.8 (2C), 138.5, 175.8. HRMS-FAB (m/z): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{15}\text{H}_{23}\text{NO}$, 234.1858; Found, 234.1856.

***N*-Benzylpivalamide (12) (entry 8):**⁸ Mp. 83 °C; IR (KBr) 3303, 2968, 1636, 1541, 1220, 1000, 735, 695 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz) δ 1.23 (s, 9H), 4.44 (d, *J* = 5.4 Hz, 2H), 5.93 (br, 1H), 7.25–7.37 (m, 5H); ¹³C NMR (CDCl₃, 75 MHz) δ 27.5 (3C), 38.6, 43.4, 127.2, 127.5 (2C), 128.5 (2C), 138.6, 178.2.

***N*-Benzyl-2-phenylpropanamide (13) (entry 9):**⁹ Mp. 76 °C; IR (KBr) 3307, 1647, 1556, 1451, 698 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz) δ 1.56 (d, *J* = 7.2 Hz, 3H), 3.60 (q, *J* = 7.2 Hz, 3H), 4.36 (dd, *J* = 5.7, 14.4 Hz, 1H), 4.43 (dd, *J* = 5.7, 14.6 Hz, 1H), 5.65 (br, 1H), 7.13–7.38 (m, 10H); ¹³C NMR (CDCl₃, 75 MHz) δ 18.4, 43.2, 46.7, 127.0, 127.1, 127.2 (2C), 127.4 (2C), 128.3 (2C), 128.6 (2C), 138.2, 141.2, 174.1

***N*-Benzyl-1-adamantanecarboxamide (14) (entry 10):**³ Mp. 172 °C; IR (KBr) 3343, 2903, 2848, 1633, 1532, 1451, 1284, 1003, 716, 692 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz) δ 1.67–1.89 (m, 12H), 2.05 (br, 3H), 4.44 (d, *J* = 5.7 Hz, 2H), 5.87 (br, 1H), 7.25–7.37 (m, 5H); ¹³C NMR (CDCl₃, 75 MHz) δ 28.0 (3C), 36.4 (3C), 39.2 (3C), 40.5, 43.2, 127.3, 127.5 (2C), 128.6 (2C), 138.6, 177.8. HRMS-FAB (*m/z*): [M+H]⁺ calcd for C₁₈H₂₃NO, 270.1858; Found, 270.1852.

***N*-Benzyl-2,2-diphenylacetamide (15) (entry 11):**¹⁰ Mp. 130 °C; IR (KBr) 3310, 3061, 1641, 1521, 1226, 1029, 751, 696 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz) δ 4.47 (d, *J* = 5.7 Hz, 2H), 4.96 (s, 1H), 5.95 (br, 1H), 7.18–7.35 (m, 15H); ¹³C NMR (CDCl₃, 75 MHz) δ 43.6, 58.9, 127.2 (2C), 127.3, 127.5 (2C), 128.6 (2C), 128.7 (4C), 128.8 (4C), 138.1, 139.3 (2C), 171.8.

***N*-Benzyl-*N*-methylcyclohexanecarboxamide (16) (entry 12):**¹¹ Liquid; IR (film) 2928, 2853, 1641, 1451, 1409, 1106, 734, 699 cm⁻¹; ¹H NMR (DMSO-*d*₆, 90 °C, 300 MHz) δ 1.17–1.71 (m, 10H), 2.62 (tt, *J* = 3.0, 11.1 Hz, 1H), 2.90 (s, 3H), 4.54 (s, 2 H), 7.18–7.36 (m, 5H); ¹³C NMR (DMSO-*d*₆, 90 °C, 75 MHz) δ 24.9, 25.3 (2C), 28.9 (2C), 39.4, 50.0, 126.6 (3C), 128.1 (2C), 137.8, 174.9. HRMS-FAB (*m/z*): [M+Na]⁺ calcd for C₁₅H₂₁NO, 254.1521; Found, 254.1532.

(*S*)-*tert*-Butyl 1-(Benzylamino)-1-oxopropan-2-ylcarbamate (17) (entry 13):¹² Mp. 104 °C; IR (KBr) 3312, 2981, 1645, 1527, 1319, 1171, 1074, 747, 701, 656 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz) δ 1.36 (d, *J* = 7.2 Hz, 3H), 1.40 (s, 9H), 4.20 (br, 1H), 4.49 (br, 2H), 5.13 (br, 1H), 6.74 (br, 1H), 7.23–7.33 (m, 5H); ¹³C NMR (CDCl₃, 75 MHz) δ 18.5, 28.2 (3C), 43.2, 50.0, 79.9, 127.3, 127.4 (2C), 128.5 (2C), 138.1, 155.5, 172.8.

***N*-Benzyl-2-methylbenzamide (18) (entry 14):**⁴ Mp. 107 °C; IR (KBr) 3278, 3025, 2925,

1623, 1456, 1273, 739, 697 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz) δ 2.48 (s, 3H), 4.64 (d, J = 5.7 Hz, 2H), 6.02 (br, 1H), 7.18–7.39 (m, 9H); ^{13}C NMR (CDCl_3 , 75 MHz) δ 19.7, 43.7, 125.6, 126.6, 127.4, 127.7 (2C), 128.6 (2C), 129.8, 130.9, 136.0, 136.1, 138.2, 169.9.

Typical Procedure for Ritter Reaction Catalyzed by 4c (Table 4). A dry, 10-mL round-bottom flask equipped with a Teflon-coated magnetic stirring bar and a reflux condenser was charged with B(OH)_3 (6.2 mg, 0.10 mmol), tetrachlorocatechol (50 mg, 0.20 mmol) and benzylic alcohols (2.0 mmol) in nitriles (5 mL). The mixture was brought to reflux. After the reaction completed, the resulting mixture was cooled to ambient temperature and wash with an aqueous solution of sodium hydrogen carbonate, and the product was extracted with ethyl acetate. The combined organic layers were dried over magnesium sulfate. The solvent was evaporated, and the residue was purified by column chromatography on silica gel to give the desired amides.

LBA 4c: ^{11}B NMR ($\text{DMSO}-d_6$, 96 MHz) δ 14.7.

***N*-Benzhydrylacetamide (19) (entry 1):**¹³ Mp. 151 °C; IR (KBr) 3254, 3050, 3028, 1646, 1546, 1495, 1289, 744, 700, 553 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz) δ 2.07 (s, 3H), 6.06 (d, J = 7.8 Hz, 1H), 6.25 (d, J = 7.8 Hz, 1H), 7.22–7.37 (m, 10H); ^{13}C NMR (CDCl_3 , 75 MHz) 23.4, 56.9, 127.3 (4C), 127.4 (2C), 128.6 (4C), 141.4 (2C), 169.1.

***N*-Benzhydrylpropionamide (20) (entry 2):**¹⁴ Mp. 144 °C; IR (KBr) 3279, 3027, 1641, 1536, 1495, 1455, 1230, 741, 696 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz) δ 1.20 (t, J = 7.5 Hz, 3H), 2.30 (q, J = 7.5 Hz, 2H), 6.03 (d, J = 7.8 Hz, 1H), 6.26 (d, J = 7.8 Hz, 1H), 7.20–7.36 (m, 10H); ^{13}C NMR (CDCl_3 , 75 MHz) δ 9.8, 29.7, 56.7, 127.3 (4C), 127.4 (2C), 128.6 (4C), 141.6 (2C), 172.7.

***N*-Benzhydrylbenzamide (21) (entry 3):**¹⁴ Mp. 170 °C; IR (KBr) 3311, 1638, 1521, 1487, 706, 692 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz) δ 6.46 (d, J = 7.8 Hz, 1H), 6.68 (d, J = 7.8 Hz, 1H), 7.28–7.84 (m, 15H); ^{13}C NMR (CDCl_3 , 75 MHz) δ 57.4, 127.0 (2C), 127.5 (4C), 127.6 (2C), 128.6 (2C), 128.7 (4C), 131.7, 134.2, 141.4 (2C), 166.4.

***N*-Benzylbenzamide (7) (entry 4):**⁴ See page S2.

***N*-(4-Methylbenzyl)benzamide (22) (entry 5):**¹⁵ Mp. 137 °C; IR (KBr) 3304, 1638, 1550, 1316, 787, 693 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz) δ 2.35 (s, 3H), 4.62 (d, J = 5.4 Hz, 2H), 6.32 (br, 1H), 7.16–7.81 (m, 9H); ^{13}C NMR (CDCl_3 , 75 MHz) δ 21.1, 43.9, 126.9 (2C), 127.9 (2C), 128.5

(2C), 129.4 (2C), 131.5, 134.4, 135.1, 137.3, 167.2.

***N*-[Di(*p*-tolyl)methyl]propionamide (23) (entry 6):** Mp. 165 °C; IR (KBr) 3303, 1649, 1539, 1513, 811, 759, 476 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz) δ 1.19 (t, *J* = 7.5 Hz, 3H), 2.28 (q, *J* = 7.5 Hz, 2H), 2.32 (s, 6H), 5.94 (d, *J* = 8.1 Hz, 1H), 6.18 (d, *J* = 8.1 Hz, 1H), 7.08–7.15 (m, 8H); ¹³C NMR (CDCl₃, 75 MHz) δ 9.8, 21.0 (2C), 29.7, 56.2, 127.2 (4C), 129.3 (4C), 137.0 (2C), 138.9 (2C), 172.6. HRMS-FAB (*m/z*): [M+H]⁺ calcd for C₁₈H₂₁NO, 268.1701; Found, 268.1695.

***N*-[Naphthalen-2-yl(phenyl)methyl]propionamide (24) (entry 7):** Mp. 162 °C; IR (KBr) 3261, 3050, 1646, 1540, 817, 733, 700 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz) δ 1.22 (t, *J* = 7.5 Hz, 3H), 2.34 (q, *J* = 7.5 Hz, 2H), 6.07 (d, *J* = 7.8 Hz, 1H), 6.44 (d, *J* = 7.8 Hz, 1H), 7.25–7.83 (m, 12H); ¹³C NMR (CDCl₃, 75 MHz) δ 9.8, 29.6, 56.7, 125.6, 125.9, 126.0, 126.2, 127.4, 127.5 (2C), 127.6, 127.9, 128.4, 128.6 (2C), 132.6, 133.1, 138.9, 141.4, 172.9. HRMS-FAB (*m/z*): [M+H]⁺ calcd for C₂₀H₁₉NO, 290.1545; Found, 290.1544.

***N*-[Di(*p*-fluorophenyl)methyl]propionamide (25) (entry 8):** Mp. 145 °C; IR (KBr) 3298, 2987, 1642, 1506, 1229, 1156, 1017, 834, 560 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz) δ 1.19 (t, *J* = 7.5 Hz, 3H), 2.30 (q, *J* = 7.5 Hz, 2H), 5.92 (d, *J* = 7.8 Hz, 1H), 6.22 (d, *J* = 7.8 Hz, 1H), 7.00–7.06 (m, 4H), 7.15–7.20 (m, 4H); ¹³C NMR (CDCl₃, 75 MHz) δ 9.7, 29.6, 55.5, 115.6 (d, *J* = 21.4 Hz, 4C), 129.0 (d, *J* = 8.3 Hz, 4C), 137.1 (2C), 162.0 (d, *J* = 245.0 Hz, 2C), 172.8. HRMS-FAB (*m/z*): [M+H]⁺ calcd for C₁₆H₁₅F₂NO, 276.1200; Found, 276.1211.

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5 (entry 1, Table 3)

6 (entry 2, Table 3)

7 (entry 3, Table 3)

8 (entry 4, Table 3)

9 (entry 5, Table 3)

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11 (entry 7, Table 3)

12 (entry 8, Table 3)

13 (entry 9, Table 3)

14 (entry 10, Table 3)

15 (entry 11, Table 3)

16 (entry 12, Table 3)

17 (entry 13, Table 3)

18 (entry 14, Table 3)

19 (entry 1, Table 4)

20 (entry 2,
Table 4)

21 (entry 3, Table 4)

22 (entry 5, Table 4)

23 (entry 6, Table 4)

24 (entry 7, Table 4)

25 (entry 8, Table 4)