Data Article

Spectral data for the synthesis of (E)-alkenylboronic acid pinacol esters via hydroboration of alkynes

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A B S T R A C T

This data article is related to a research paper entitled “Solvent-and metal-free hydroboration of alkynes under microwave irradiation” (Gioia et al. TETL-D-19-01698) [1]. Herein we present the spectral data acquired from the synthesis of (E)-alkenyl boronic acid pinacol esters. The data include the general information and the synthetic procedure affording the target derivatives, which were fully characterized by Nuclear Magnetic Resonance (1H and 13C NMR) and, for the most part, by Electrospray Ionization High Resolution Mass (ESI-MS). Proton and carbon NMR spectra and ESI-MS spectra were provided which will be useful for further organic chemists if they are interested in the synthesis of these building blocks.

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1. Data description

A series of eighteen (E)-alkenylboronic acid pinacol esters (2a-r) were synthesized from the hydroboration of aromatic or aliphatic alkynes in presence of pinacol borane according to a solvent- and metal-free procedure [1]. The synthetic scheme and the chemical structures of the target derivatives were described in Fig. 1. The final compounds 2a-r were all characterized by \(^1\)H and \(^{13}\)C NMR spectroscopy and HR mass spectra were recorded for the most part of synthesized boranes. All the spectra were provided in this data article (Figs. 2–46). It is noteworthy that the \(^{13}\)C NMR signal for the alkenyl carbon next to the boron atom is identified in all carbon-13 spectra except for compound 2e.

2. Experimental design, materials, and methods

2.1. General information

All reactions were performed under microwave irradiation using an Anton Paar Monowave 300 synthesizer. Pinacol borane (4,4,5,5-tetramethyl-1,3,2-dioxaborolane) was purchased from Sigma Aldrich. Alkynes and carboxylic acids were purchased from Sigma-Aldrich, Fisher Scientific or Fluorochem. Optima LC/MS grade acetonitrile was purchased from Fisher Scientific. 1,4-Dioxane was purchased from Carlo Erba, acetonitrile and dimethylformamide were purchased from Fisher Scientific and...
octane was purchased from Sigma-Aldrich. All purchased compounds or solvents were used as received. All reactions were monitored through thin-layer chromatography on GF254 plates purchased from Merck and spots were detected under a UV lamp (254 nm and 356 nm) or by spraying plates with 0.5% w/v aqueous KMnO4, followed by drying with heat gun. Chromatographic separations were performed on silica gel columns (Kieselgel 300–400 mesh) with eluent indicated for each compound. Organic solutions were concentrated under reduced pressure on a rotary evaporator.

The samples were dissolved CDCl3, DMSO-d6 or CD3OD to acquire the NMR spectra using a Bruker DRX400 Fourier transform NMR spectrometer. 1H and 13C NMR spectra were recorded respectively at 400 MHz and 100 MHz, using an internal deuterium lock. The chemical shift of the solvent residual signal was used as the reference. Data for 1H NMR are reported as follows: chemical shift (δ, ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, sext = sextuplet, dd = doublet of doublets, dt = doublet of triplets, m = multiplet), coupling constant (J, Hz) and integration. Data for 13C NMR are reported as a list of chemical shifts.

High-resolution mass spectroscopy (HRMS) measurements were performed by electrospray ionization (ESI-MS) using 2 mg/ml sample solutions in HPLC grade CH3CN or MeOH.

2.2. General procedure

Pinacol borane (1.4 mL, 9.44 mmol, 4 eq.), alkyne 1a–r (2.36 mmol, 1 eq) and 4-(dimethylamino) benzoic acid (5 mol%) were introduced in a 10 mL microwave sealed flask. Reactions conditions

![Fig. 1. Synthesis and chemical structures of derivatives 2a–r.](image-url)
Fig. 2. $^1$H NMR spectra of compound 2a (400 MHz) in DMSO-d6.

Fig. 3. $^{13}$C NMR spectra of compound 2a (100 MHz) in DMSO-d6.
Fig. 4. $^1$H NMR spectra of compound 2b (400 MHz) in CD$_3$OD.

Fig. 5. $^{13}$C NMR spectra of compound 2b (100 MHz) in CD$_3$OD.
Fig. 6. $^1$H NMR spectra of compound 2c (400 MHz) in CD$_3$OD.

Fig. 7. $^{13}$C NMR spectra of compound 2c (100 MHz) in CD$_3$OD.
Fig. 8. $^1$H NMR spectra of compound 2d (400 MHz) in CDCl$_3$.

Fig. 9. $^{13}$C NMR spectra of compound 2d (100 MHz) in CDCl$_3$. 
Fig. 10. $^1$H NMR spectra of compound 2e (400 MHz) in CDCl$_3$.

Fig. 11. $^{13}$C NMR spectra of compound 2e (100 MHz) in CDCl$_3$. 
Fig. 12. $^1$H NMR spectra of compound 2f (400 MHz) in CDCl$_3$.

Fig. 13. $^{13}$C NMR spectra of compound 2f (100 MHz) in CDCl$_3$. 
depended on alkyl or aromatic alkynes, time was set respectively at 30 and 15 min and temperature was set at 120 °C for both alkyne types. The reaction medium was directly purified by flash chromatography to obtain the final product 2a-r. Methyl tert-butyl ether (MTBE) was used for crystallization attempts.

Fig. 14. ESI-MS spectra of compound 2f.

Fig. 15. 1H NMR spectra of compound 2g (400 MHz) in CDCl₃.
Fig. 16. $^{13}$C NMR spectra of compound 2g (100 MHz) in CDCl$_3$.

Fig. 17. $^{1}$H NMR spectra of compound 2h (400 MHz) in CDCl$_3$. 
Fig. 18. $^{13}$C NMR spectra of compound 2h (100 MHz) in CDCl$_3$.

Fig. 19. $^1$H NMR spectra of compound 2i (400 MHz) in CDCl$_3$. 
Fig. 20. $^{13}$C NMR spectra of compound 2i (100 MHz) in CDCl$_3$.

Fig. 21. $^1$H NMR spectra of compound 2j (400 MHz) in CDCl$_3$. 
Fig. 22. $^{13}$C NMR spectra of compound 2j (100 MHz) in CDCl$_3$.

Fig. 23. $^1$H NMR spectra of compound 2k (400 MHz) in CDCl$_3$. 

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Fig. 24. $^{13}$C NMR spectra of compound 2k (100 MHz) in CDCl$_3$.

Fig. 25. ESI-MS spectra of compound 2k.
Fig. 26. $^1$H NMR spectra of compound 2l (400 MHz) in CDCl$_3$.

Fig. 27. $^{13}$C NMR spectra of compound 2l (100 MHz) in CDCl$_3$. 
Fig. 28. ESI-MS spectra of compound 2l.

Fig. 29. $^1$H NMR spectra of compound 2m (400 MHz) in CDCl$_3$. 
**Fig. 30.** $^{13}$C NMR spectra of compound 2m (100 MHz) in CDCl$_3$.

**Fig. 31.** ESI-MS spectra of compound 2m.
Fig. 32. $^1$H NMR spectra of compound 2n (400 MHz) in CDCl$_3$.

Fig. 33. $^{13}$C NMR spectra of compound 2n (100 MHz) in CDCl$_3$. 
Fig. 34. ESI-MS spectra of compound 2n.

Fig. 35. ¹H NMR spectra of compound 2o (400 MHz) in CDCl₃.
**Fig. 36.** $^{13}$C NMR spectra of compound 2o (100 MHz) in CDCl$_3$.

**Fig. 37.** ESI-MS spectra of compound 2o.
Fig. 38. $^1$H NMR spectra of compound 2p (400 MHz) in CDCl$_3$.

Fig. 39. $^{13}$C NMR spectra of compound 2p (100 MHz) in CDCl$_3$. 
Fig. 40. ESI-MS spectra of compound 2p.

Fig. 41. $^1$H NMR spectra of compound 2q (400 MHz) in CDCl$_3$. 
Fig. 42. $^{13}$C NMR spectra of compound 2q (100 MHz) in CDCl$_3$.

Fig. 43. ESI-MS spectra of compound 2q.
Fig. 44. $^1$H NMR spectra of compound $2r$ (400 MHz) in CDCl$_3$.

Fig. 45. $^{13}$C NMR spectra of compound $2r$ (100 MHz) in CDCl$_3$. 

2.3. Characterization data

2.3.1. (E)-2-Styrylboronic acid pinacol ester (2a) [2]

Purification by flash chromatography with cyclohexane: diethyl ether (97:3) to obtain a pale yellow oil (336 mg, 62%). $^{1}$H NMR (400 MHz, DMSO-d$_6$) $\delta$ 7.58 (d, $J$ 8.0 Hz, 2H), 7.40–7.28 (m, 4H), 6.14 (d, $J$ 18.5 Hz, 1H), 1.24 (s, 12H); $^{13}$C NMR (100 MHz, DMSO-d$_6$) $\delta$ 149.21, 136.86, 129.14, 128.68, 127.02, 116.53, 83.02, 24.63.

2.3.2. (E)-2-(3-methoxystyryl)boronic acid pinacol ester (2b) [3]

Purification by flash chromatography with cyclohexane: diethyl ether (70:30) to obtain a colorless oil (374 mg, 60%). $^{1}$H NMR (400 MHz, CD$_3$OD) $\delta$ 7.32 (d, $J$ 18.4 Hz, 1H), 7.25 (m, 1H), 7.10–7.02 (m, 2H), 6.87 (m, 1H), 6.10 (d, $J$ 18.4 Hz, 1H), 3.80 (s, 3H), 1.29 (s, 12H); $^{13}$C NMR (100 MHz, CD$_3$OD) $\delta$ 161.44, 150.94, 149.38, 130.64, 120.69, 118.05, 115.83, 113.06, 84.62, 55.67, 25.13.
2.3.3. (E)-2-(4-methoxystyryl)boronic acid pinacol ester (2c) [2]

Purification by flash chromatography with cyclohexane: diethyl ether (70:30) to obtain a white solid (361.3 mg, 58%). $^1$H NMR (400 MHz, CD$_3$OD) $\delta$ 7.44 (m, 2H), 7.30 (d, $J$ 18.4 Hz, 1H), 6.89 (m, 2H), 5.94 (d, $J$ 18.4 Hz, 1H), 3.80 (s, 3H), 1.29 (s, 12H); $^{13}$C NMR (100 MHz, CD$_3$OD) $\delta$ 162.03, 150.72, 149.21, 131.60, 129.51, 115.04, 84.47, 55.75, 25.12.

2.3.4. (E)-2-(3-phenylprop-1-en-1-yl)boronic acid pinacol ester (2d) [4]

Purification by flash chromatography with cyclohexane: diethyl ether (90:10) to obtain a colorless oil (455.2 mg, 79%). $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.28 (m, 2H), 7.23–7.09 (m, 3H), 6.77 (dt, $J$ 17.8 Hz, 6.3 Hz, 1H), 5.46 (dt, $J$ 17.8 Hz, 1.5 Hz, 1H), 3.49 (dd, $J$ 6.3 Hz, 1.5 Hz, 2H), 1.26 (s, 12H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 152.58, 139.20, 129.05, 128.56, 126.27, 119.90, 83.24, 42.41, 24.92.

2.3.5. (Z)-2-(1,2-diphenylvinyl)boronic acid pinacol ester (2e) [5]

Purification by flash chromatography with pentane: diethyl ether (97:3) to obtain a pale yellow solid (202.2 mg, 28%). $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.36 (s, 1H), 7.28–7.24 (m, 2H), 7.21–7.18 (m, 1H), 7.17–7.14 (m, 2H), 7.11–7.09 (m, 3H), 7.05–7.03 (m, 2H), 1.20 (s, 12H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 143.29, 140.55, 137.11, 130.08, 128.98, 128.37, 127.97, 127.70, 126.39, 83.91, 24.93. Carbon signal next to boron atom was not observed.

2.3.6. Methyl (E)-3-(2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)vinyl)benzoate (2f)
Purification by flash chromatography with petroleum ether (40–60 °C); ethyl acetate (90:10) to obtain a pale yellow solid (446.0 mg, 65%). $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.16 (t, $J$ 1.6 Hz, 1H), 7.95 (dt, $J$ 7.8 Hz, 1.6 Hz, 1H), 7.66 (dt, $J$ 7.8 Hz, 1.6 Hz, 1H), 7.47–7.36 (m, 2H), 6.24 (d, $J$ 18.4 Hz, 1H), 3.91 (s, 3H), 1.31 (s, 12H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 166.99, 148.39, 137.95, 131.30, 130.69, 129.91, 128.80, 128.37, 118.28, 83.61, 52.31, 24.95; HRMS (ESI): calcd. for C$_{16}$H$_{22}$BO$_4$: 289.1606; found [M+H]$^+$: 289.1604.

2.3.7. Methyl (E)-4-(2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)vinyl)benzoate (2g) [2]

Purification by flash chromatography with petroleum ether (40–60 °C); ethyl acetate (90:10) to obtain a colourless solid (330.9 mg, 48%). $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.00 (d, $J$ 8.3 Hz, 2H), 7.53 (d, $J$ 8.3 Hz, 2H), 7.40 (d, $J$ 18.4 Hz, 1H), 6.27 (d, $J$ 18.4 Hz, 1H), 3.90 (s, 3H), 1.31 (s, 12H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 166.98, 148.25, 141.82, 130.25, 130.03, 127.02, 119.66, 83.69, 52.25, 24.94.

2.3.8. (E)-2-(3-methoxyprop-1-en-1-yl)boronic acid pinacol ester (2h) [6]

Purification by flash chromatography with petroleum ether (40–60 °C); ethyl acetate (50:50) to obtain a pale yellow oil (164.0 mg, 34%). $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 6.62 (dt, $J$ 18.2 Hz, 4.8 Hz, 1H), 5.68 (dt, $J$ 18.2 Hz, 1.8 Hz, 1H), 3.99 (dd, $J$ 4.8 Hz, 1.8 Hz, 2H), 3.34 (s, 3H), 1.26 (s, 12H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 149.17, 119.40, 83.38, 74.29, 58.42, 24.98.

2.3.9. (E)-2-(3-chlorostyryl)boronic acid pinacol ester (2i) [2]

Purification by flash chromatography with pentane: diethyl ether (97:3) to obtain a pale yellow oil (348.48 mg, 55%). $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.46 (s, 1H), 7.37–7.25 (m, 4H), 6.17 (d, $J$ 18.4 Hz, 1H), 1.32 (s, 12H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 147.65, 139.17, 134.39, 129.61, 128.57, 126.76, 125.01, 118.08, 83.32, 24.63.
2.3.10. (E)-2-(4-chlorostyryl)boronic acid pinacol ester (2j) [2]

Purification by flash chromatography with cyclohexane: ethyl acetate (60:40) to obtain a pale yellow solid (573.2 mg, 92%). ^1H NMR (400 MHz, CDCl₃) δ 7.41 (m, 2H), 7.37–7.24 (m, 3H), 6.13 (d, J 18.4 Hz, 1H), 1.31 (s, 12H); ^13C NMR (100 MHz, CDCl₃) δ 148.14, 136.12, 134.74, 128.92, 128.36, 117.42, 83.59, 24.95.

2.3.11. (E)-2-(pent-1-en-1-yl)boronic acid pinacol ester (2k) [7]

Purification by flash chromatography with pentane: diethyl ether (97:3) to obtain a pale yellow oil (161.9 mg, 35%). ^1H NMR (400 MHz, CDCl₃) δ 6.62 (dt, J 18.0 Hz, 6.4 Hz, 1H), 5.42 (dt, J 18.0 Hz, 1.6 Hz, 1H), 2.11 (m, 2H), 1.43 (sext., J 7.4 Hz, 2H), 1.24 (s, 12H), 0.90 (t, J 7.4 Hz, 3H); ^13C NMR (100 MHz, CDCl₃) δ 154.68, 118.63, 83.10, 38.06, 24.91, 21.55, 13.91; HRMS (ESI): calcd. for C₁₁H₂₂BO₂: 197.1707; found [M+H]^+: 197.1711.

2.3.12. (E)-2-(hex-1-en-1-yl)boronic acid pinacol ester (2l) [8]

Purification by flash chromatography with pentane: diethyl ether (97:3) to obtain a pale yellow oil (267.2 mg, 54%). ^1H NMR (400 MHz, CDCl₃) δ 6.63 (dt, J 17.9 Hz, 6.4 Hz, 1H), 5.42 (dt, J 17.9 Hz, 1.6 Hz, 1H), 2.14 (m, 2H), 1.39 (m, 2H), 1.28–1.28 (m, 14H), 0.88 (t, J 7.2 Hz, 3H); ^13C NMR (100 MHz, CDCl₃) δ 154.93, 118.42, 83.11, 35.65, 30.50, 24.92, 22.40, 14.06; HRMS (ESI): calcd. for C₁₂H₂₃BNaO₂: 233.1683; found [M+Na]^+: 233.1685.

2.3.13. (E)-2-(hept-1-en-1-yl)boronic acid pinacol ester (2m) [9]

Purification by flash chromatography with pentane: diethyl ether (97:3) to obtain a pale yellow oil (339.8 mg, 64%). ^1H NMR (400 MHz, CDCl₃) δ 6.63 (dt, J 18.0 Hz, 6.4 Hz, 1H), 5.41 (d, J 18.0 Hz, 1H), 2.13 (m, 2H), 1.41 (m, 2H), 1.41–1.29 (m, 16H), 0.88 (m, 3H); ^13C NMR (100 MHz, CDCl₃) δ 154.97, 118.51,
2.3.14. (E)-2-(oct-1-en-1-yl)boronic acid pinacol ester (2n) \[9\]

Purification by flash chromatography with pentane: diethyl ether (97:3) to obtain a pale yellow oil (315.1 mg, 58%). \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 6.63 (dt, \(J\) 17.9 Hz, 6.4 Hz, 1H), 5.41 (dt, \(J\) 17.9 Hz, 1.6 Hz, 1H), 2.14 (m, 2H), 1.40 (m, 2H), 1.26 (s, 18H), 0.86 (m, 3H); \(^1^3\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 154.98, 118.59, 83.10, 35.99, 31.87, 29.06, 28.34, 24.92, 22.74, 14.24; HRMS (ESI): calcd. for C\(_{13}\)H\(_{25}\)BNaO\(_2\): 247.1840 found [M+Na]\(^+\): 247.1842.

2.3.15. (E)-2-(non-1-en-1-yl)boronic acid pinacol ester (2o) \[9\]

Purification by flash chromatography with pentane: diethyl ether (97:3) to obtain a pale yellow oil (282.4 mg, 50%). \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 6.63 (dt, \(J\) 17.9 Hz, 6.4 Hz, 1H), 5.42 (dt, \(J\) 17.9 Hz, 1.6 Hz 1H), 2.13 (m, 2H), 1.40 (m, 2H), 1.26 (s, 20H), 0.86 (m, 3H); \(^1^3\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 155.00, 118.41, 83.11, 35.99, 31.95, 29.35, 29.31, 28.37, 24.92, 22.81, 14.25; HRMS (ESI): calcd. for C\(_{14}\)H\(_{28}\)BO\(_2\): 239.2177; found [M+H]\(^+\): 239.2178.

2.3.16. (E)-2-(dec-1-en-1-yl)boronic acid pinacol ester (2p) \[10\]

Purification by flash chromatography with pentane: diethyl ether (97:3) to obtain a pale yellow oil (388.4 mg, 62%). \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 6.63 (dt, \(J\) 18.0 Hz, 6.4 Hz, 1H), 5.42 (dd, \(J\) 18.0 Hz, 1.6 Hz, 1H), 2.14 (m, 2H), 1.40 (m, 2H), 1.26 (s, 22H), 0.86 (m, 3H); \(^1^3\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 155.00, 118.60, 83.11, 35.99, 32.03, 29.60, 29.39 (2xCH\(_2\)), 28.37, 24.92, 22.82, 14.25; HRMS (ESI): calcd. for C\(_{16}\)H\(_{31}\)BNaO\(_2\): 289.2309 found [M+Na]\(^+\): 289.2311.

2.3.17. (E)-2-(undec-1-en-1-yl)boronic acid pinacol ester (2q) \[11\]
Purification by flash chromatography with pentane: diethyl ether (97:3) to obtain a pale yellow oil (405.4 mg, 61%).\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 6.63 (dt, \(J\) 17.9 Hz, 6.4 Hz; 1H), 5.42 (dt, \(J\) 17.9 Hz, 1.5 Hz; 1H), 2.13 (m, 2H), 1.40 (m, 2H), 1.30 e 1.25 (m, 24H), 0.86 (m, 3H);\(^1\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 155.01, 118.58, 83.11, 35.99, 32.05, 29.69, 29.65, 29.48, 29.39, 28.38, 24.93, 22.83, 14.26; HRMS (ESI): calcd. for C\(_{17}\)H\(_{33}\)BNaO\(_2\): 303.2466; found [M\(\text{+Na}\)]\(^+\): 303.2469.

2.3.18. (E)-2-(dodec-1-en-1-yl)boronic acid pinacol ester (2r)\(^{[12]}\)

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Purification by flash chromatography with pentane: diethyl ether (97:3) to obtain a pale yellow oil (612.9 mg, 88%).\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 6.63 (dt, \(J\) 17.9 Hz, 6.4 Hz, 1H), 5.42 (dt, \(J\) 17.9 Hz, 1.5 Hz, 1H), 2.13 (m, 2H), 1.40 (m, 2H), 1.30 e 1.25 (m, 26H), 0.87 (t, \(J\) 6.9 Hz, 3H);\(^1\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 155.00, 118.53, 83.11, 35.99, 32.05, 29.77, 29.73, 26.65, 29.49, 29.39, 28.38, 24.93, 22.84, 14.27; HRMS (ESI): calcd. for C\(_{18}\)H\(_{36}\)BO\(_2\): 295.2803; found [M\(\text{+H}\)]\(^+\): 295.2804.

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Conflict of Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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