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Data Article

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

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ABSTRACT

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 (^{1}H and ^{13}C 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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Specifications Table

Subject	Chemistry
Specific subject area	Organic chemistry. Hydroboration of alkynes under microwave irradiation
Type of data	Figure ^{1}H and ^{13}C NMR spectra ESI-MS spectra
How data were acquired	NMR (Bruker DRX400 Spectrometer, 400 MHz for ^{1}H NMR and 100 MHz for ^{13}C NMR), NMR data processing (MestReNova software, version 11.0.2−18153, Mestrelab Research S.L. 2016), Electrospray Ionization High Resolution Mass (Bruker MicroTOF Q Spectrometer)
Data format	Raw and Analyzed
Parameters for data collection	All reagents and solvents were commercially available and used as received. The alkenylboronic acid pinacol esters were synthesized by the hydroboration of alkynes under microwave irradiation and all final compounds were purified by column chromatography.
Description of data collection	The isolated compounds were all characterized by NMR spectroscopy, and for the most part by HRMS.
Data source location	Université Claude Bernard Lyon 1, Lyon, France
Data accessibility	Data are available with the article
Related research article	Bruna Gioia, Alexandre Arnaud, Sylvie Radix, Nadia Walchshofer, Anne Doléans-Jordheim, Luc Rocheblave, Solvent- and metal-free hydroboration of alkynes under microwave irradiation, submitted to Tetrahedron Letters, reference number TETL-D-19-01698.

Value of the Data

- (E)-alkenylboronic acid pinacol esters are versatile building blocks extensively used to create carbon-carbon and carbon-heteroatom bonds, therefore a spectral data compilation of boronates of varied chemical structure, in a single data article, is a valuable asset.
- The provided information on the spectroscopic data of (E)-alkenylboronic acid pinacol esters could be useful for further organic chemists if they focus on the synthesis of alkenyl boronates. In particular, all the ^{13}C NMR signals are identified.
- The data could be helpful for organic chemists if they are interested in cross-coupling reactions using alkenyl boronates.

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 synthetizer. 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

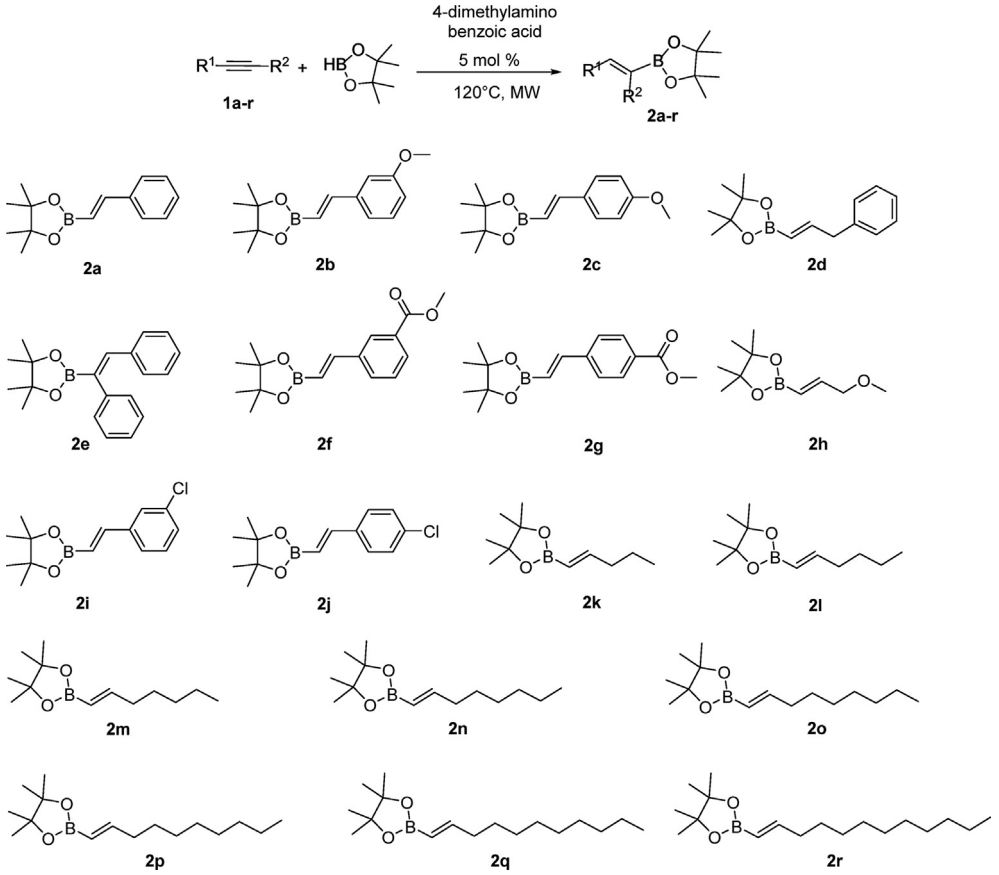

Fig. 1. Synthesis and chemical structures of derivatives **2a-r**.

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 pression on a rotary evaporator.

The samples were dissolved $CDCl_3$, DMSO-*d*6 or CD_3OD to acquire the NMR spectra using a Bruker DRX400 Fourier transform NMR spectrometer. ^1H and ^{13}C 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 ^{13}C 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 CH_3CN 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

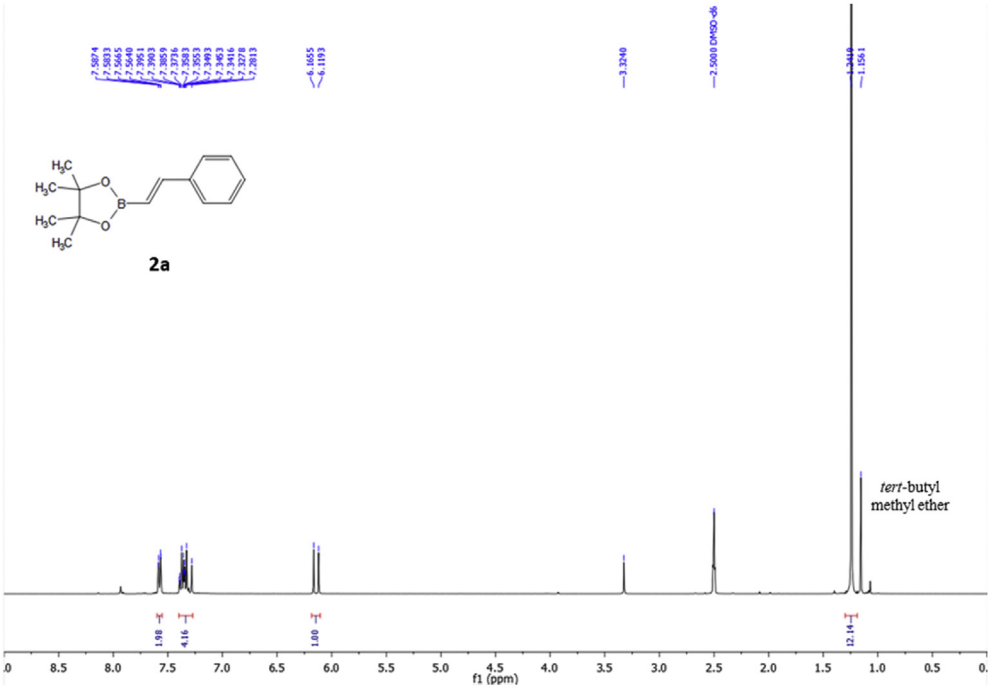

Fig. 2. ^{1}H NMR spectra of compound **2a** (400 MHz) in DMSO-*d*6.

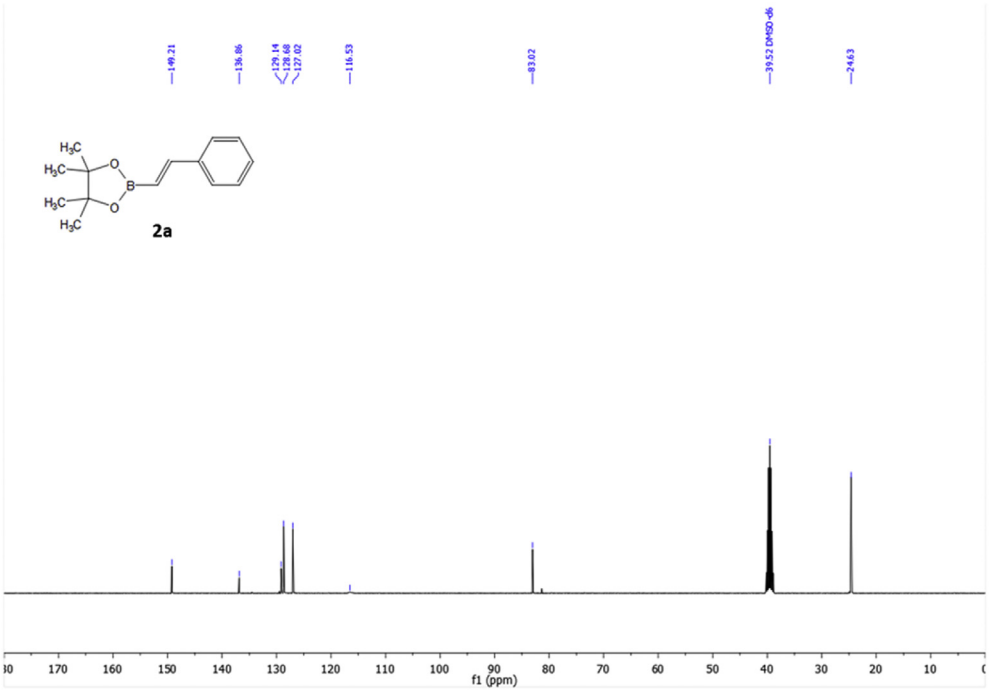

Fig. 3. ^{13}C NMR spectra of compound **2a** (100 MHz) in DMSO-*d*6.

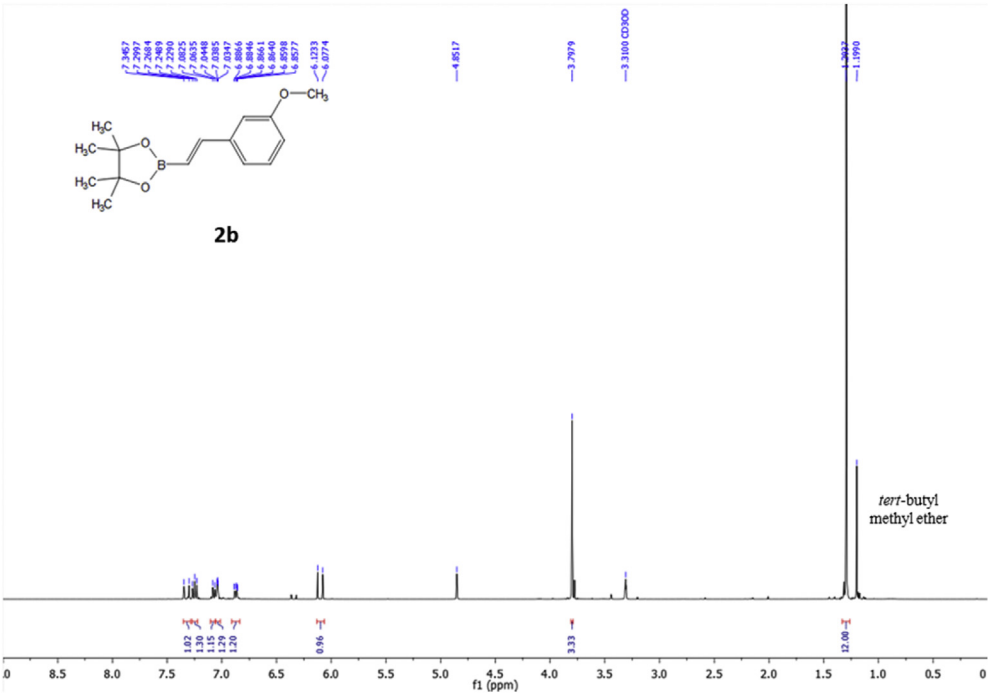

Fig. 4. ^{1}H NMR spectra of compound **2b** (400 MHz) in CD_3OD.

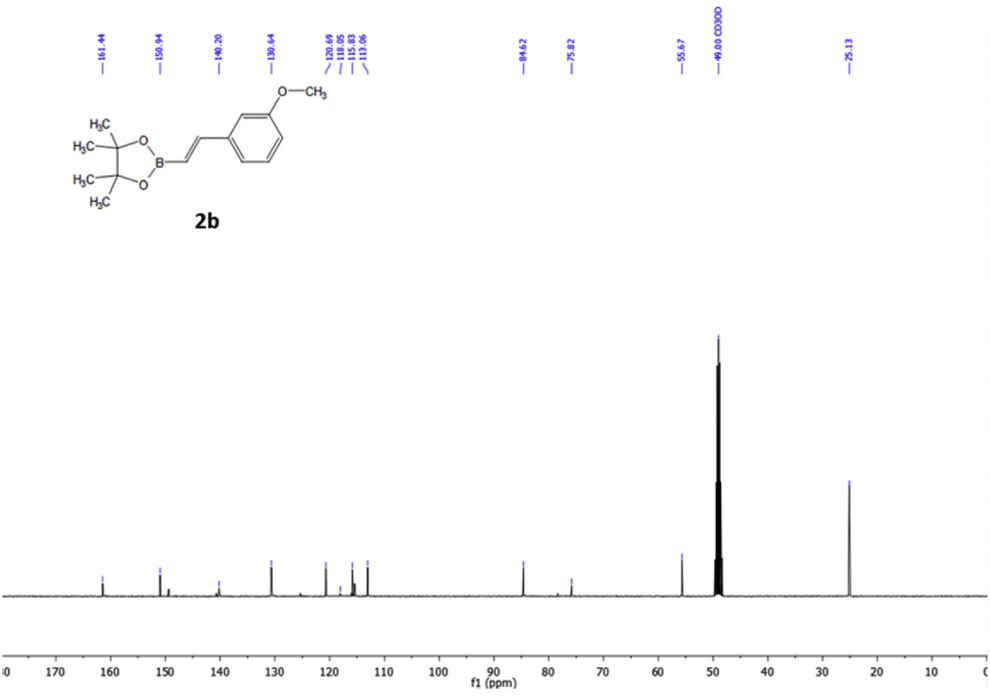

Fig. 5. ^{13}C NMR spectra of compound **2b** (100 MHz) in CD_3OD.

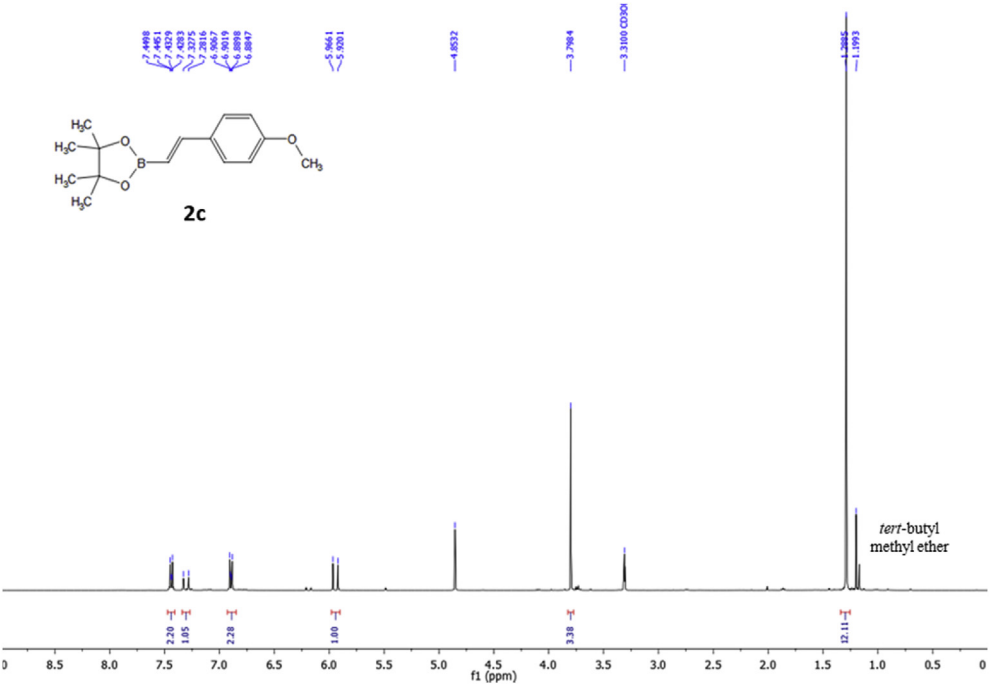

Fig. 6. ^{1}H NMR spectra of compound **2c** (400 MHz) in CD_3OD.

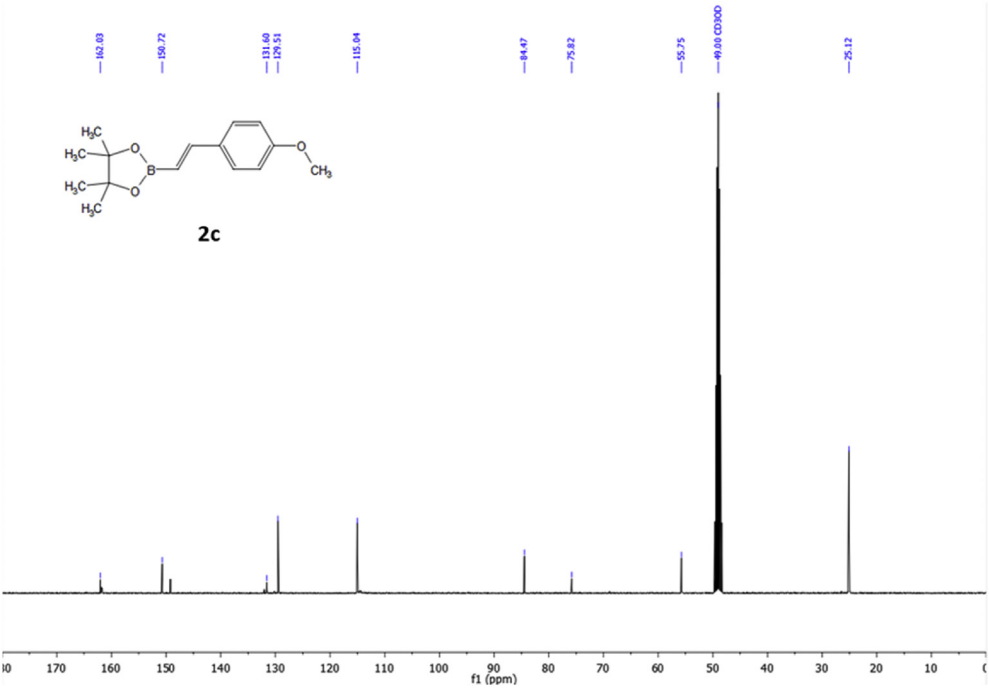

Fig. 7. ^{13}C NMR spectra of compound **2c** (100 MHz) in CD_3OD.

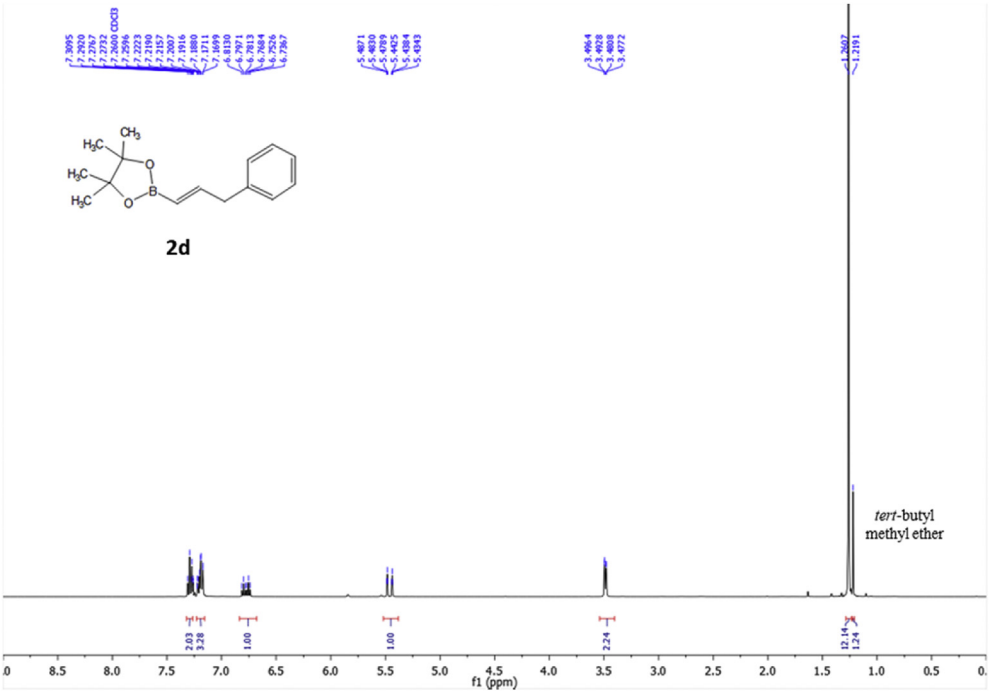

Fig. 8. ^{1}H NMR spectra of compound **2d** (400 MHz) in $CDCl_3$.

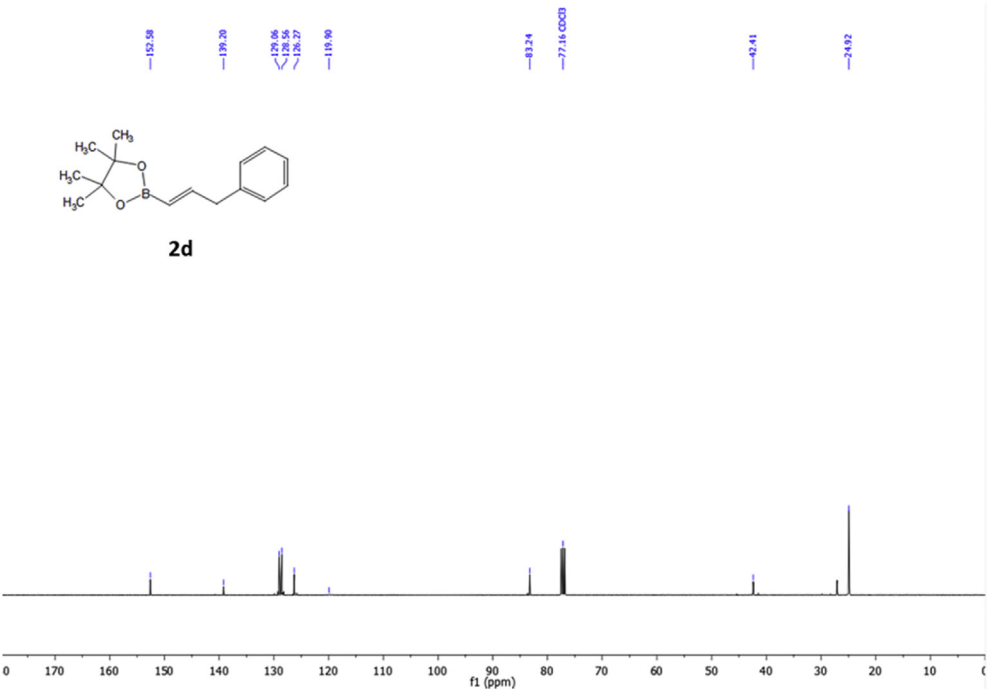

Fig. 9. ^{13}C NMR spectra of compound **2d** (100 MHz) in $CDCl_3$.

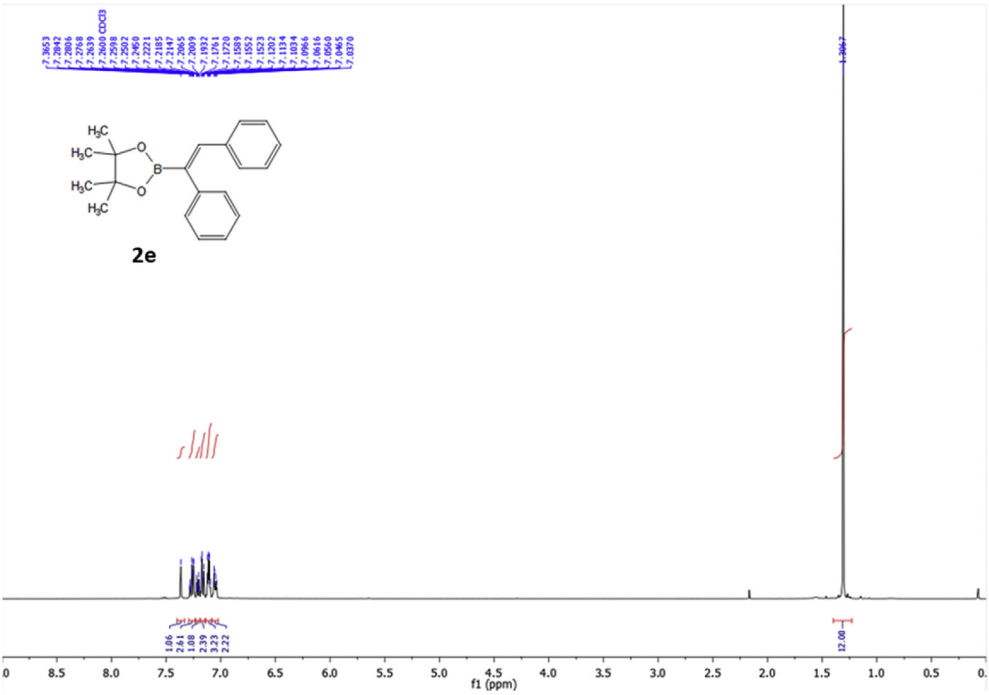

Fig. 10. ^{1}H NMR spectra of compound **2e** (400 MHz) in $CDCl_3$.

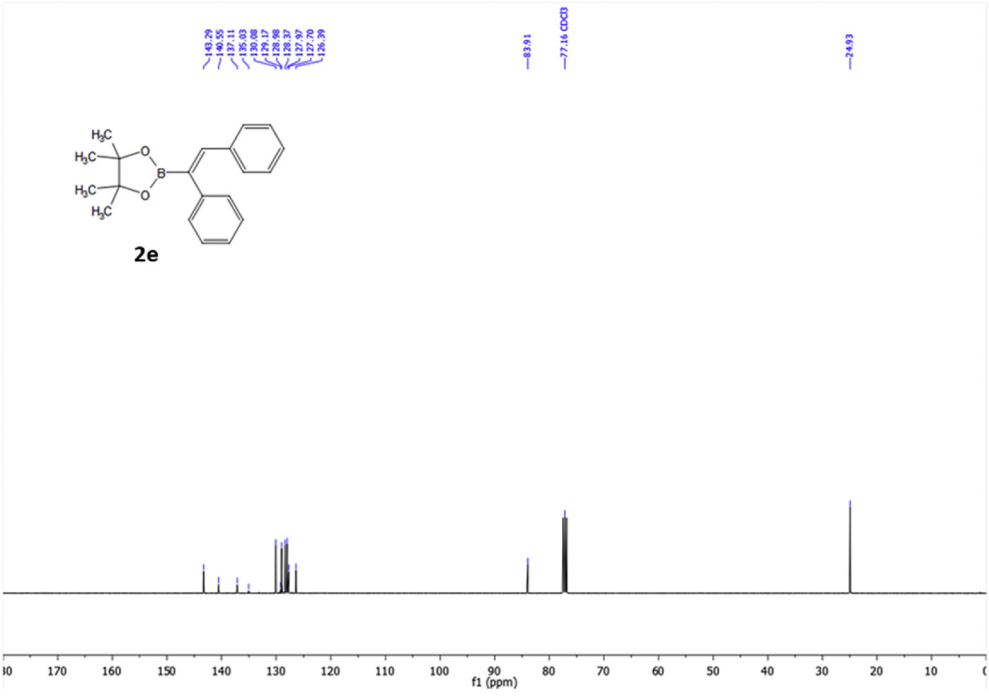

Fig. 11. ^{13}C NMR spectra of compound **2e** (100 MHz) in $CDCl_3$.

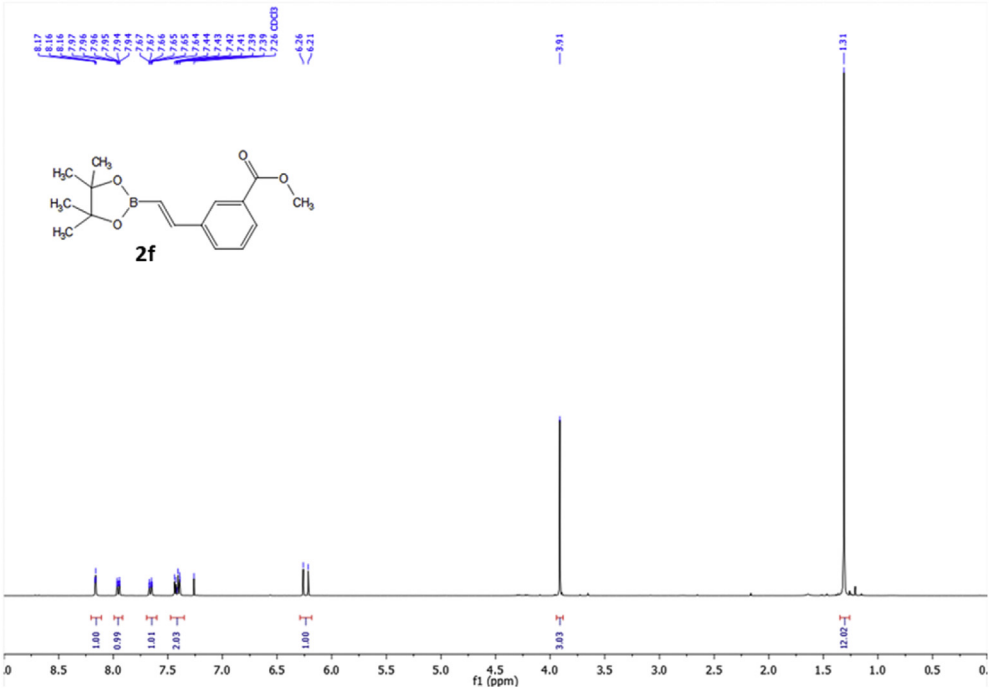

Fig. 12. ^{1}H NMR spectra of compound **2f** (400 MHz) in $CDCl_3$.

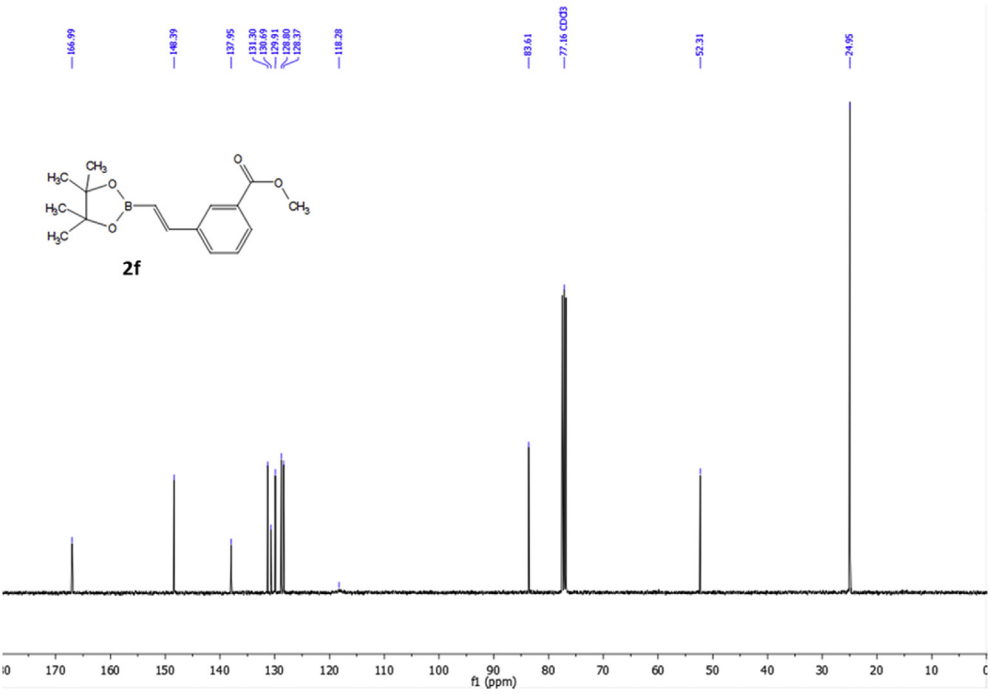

Fig. 13. ^{13}C NMR spectra of compound **2f** (100 MHz) in $CDCl_3$.

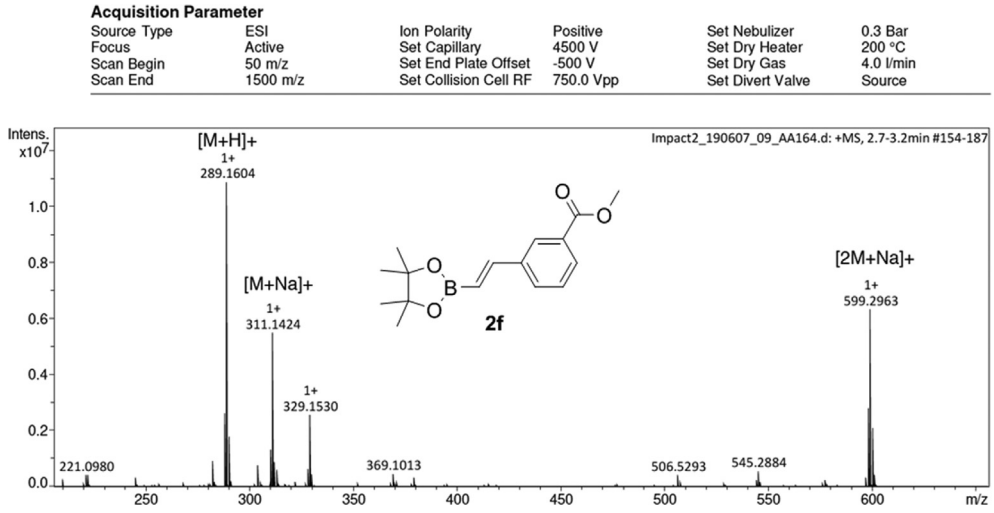

Fig. 14. ESI-MS spectra of compound **2f**.

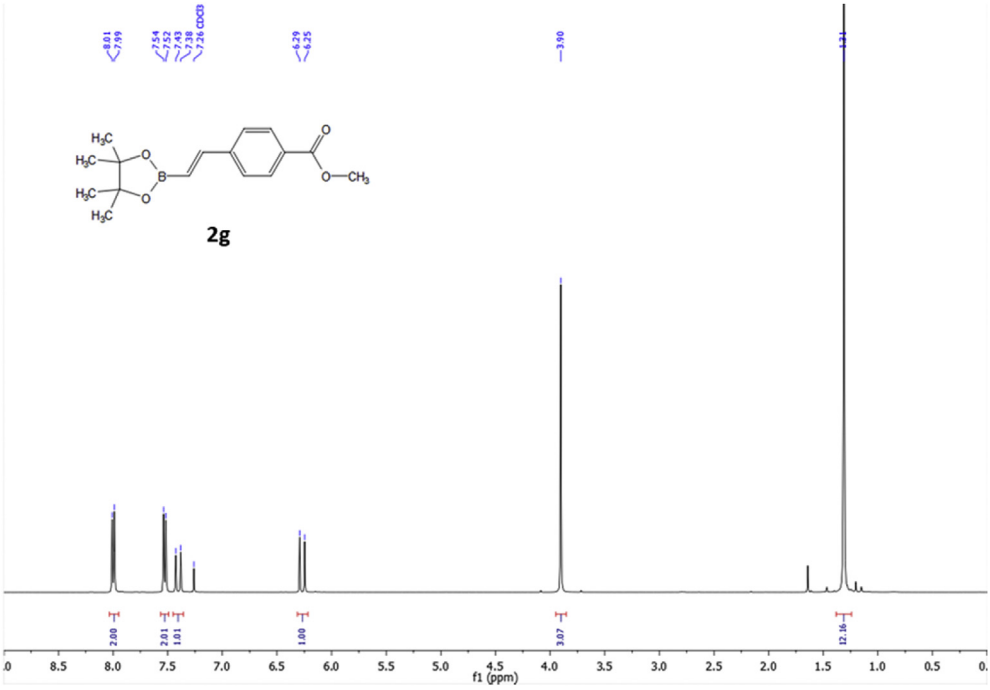

Fig. 15. ^{1}H NMR spectra of compound **2g** (400 MHz) in $CDCl_3$.

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.

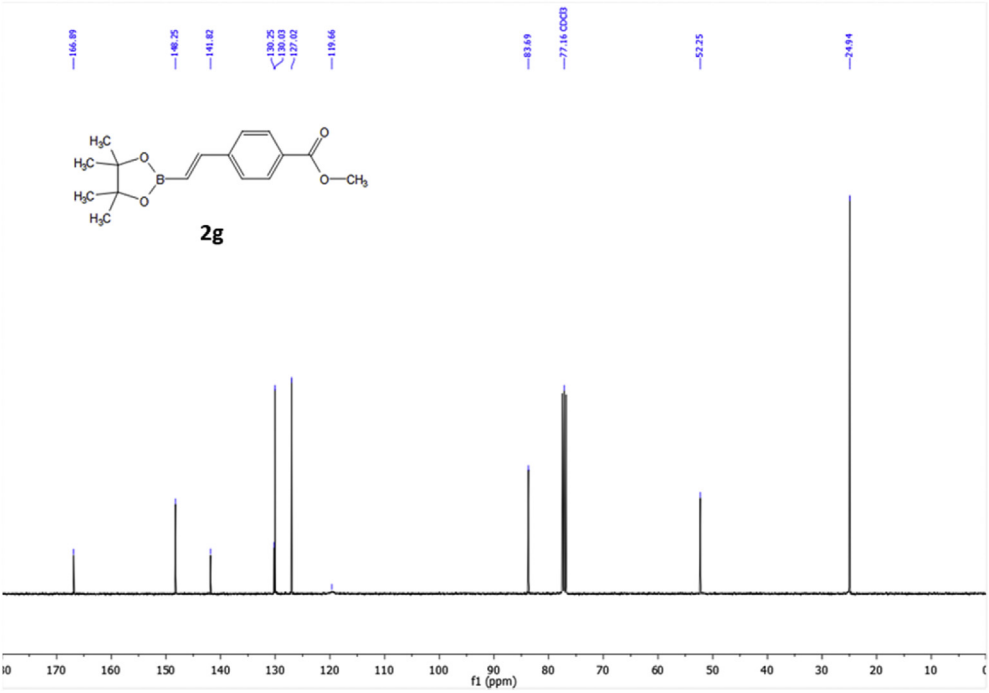

Fig. 16. ^{13}C NMR spectra of compound **2g** (100 MHz) in $CDCl_3$.

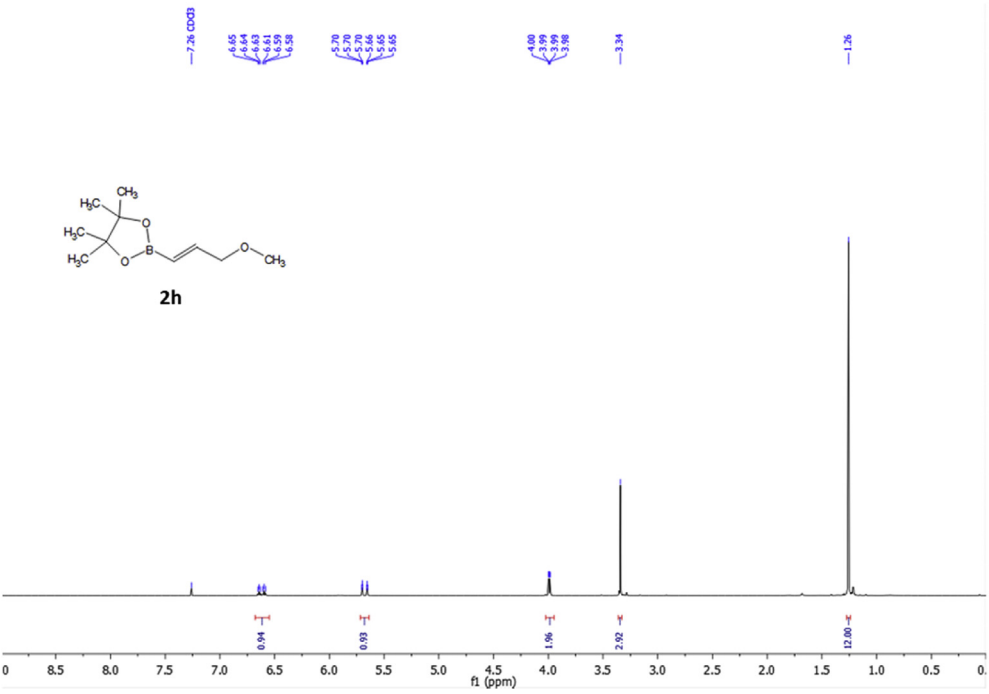

Fig. 17. ^{1}H NMR spectra of compound **2h** (400 MHz) in $CDCl_3$.

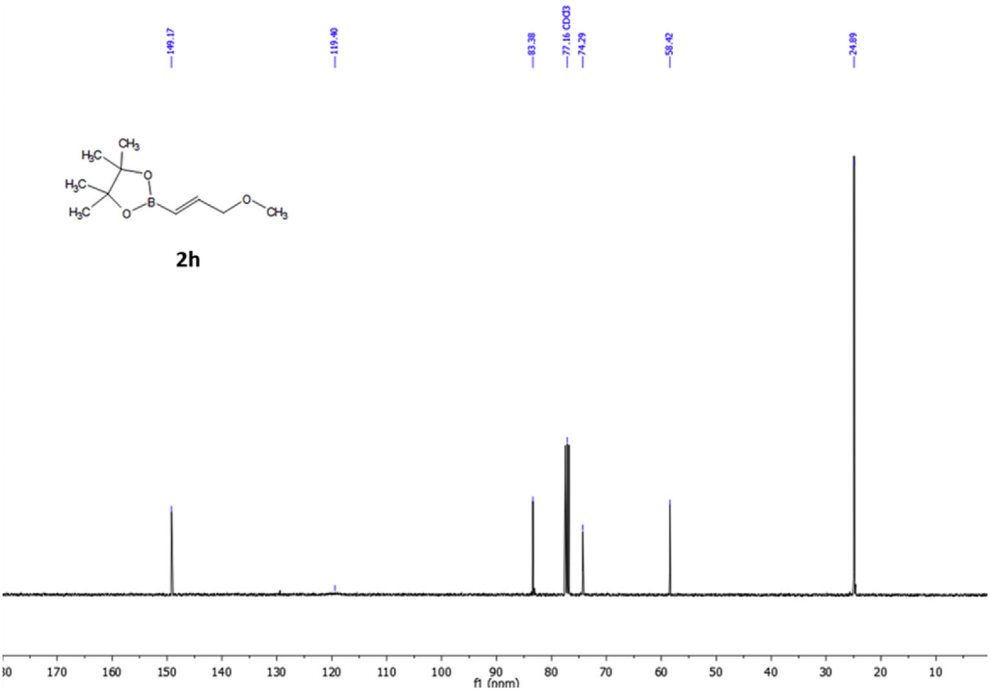

Fig. 18. ^{13}C NMR spectra of compound **2h** (100 MHz) in $CDCl_3$.

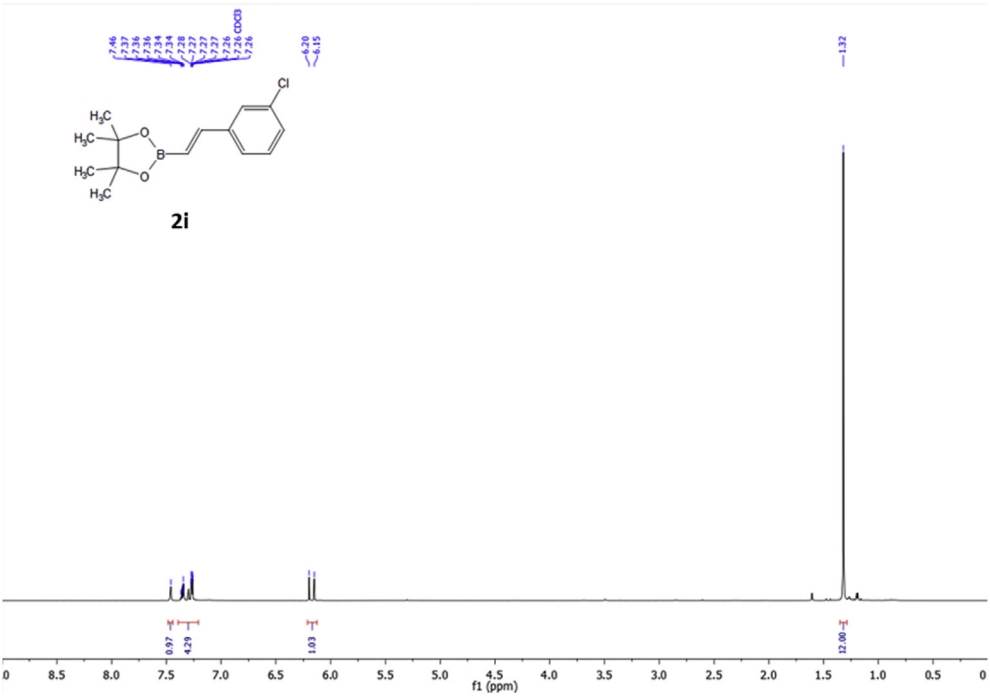

Fig. 19. ^{1}H NMR spectra of compound **2i** (400 MHz) in $CDCl_3$.

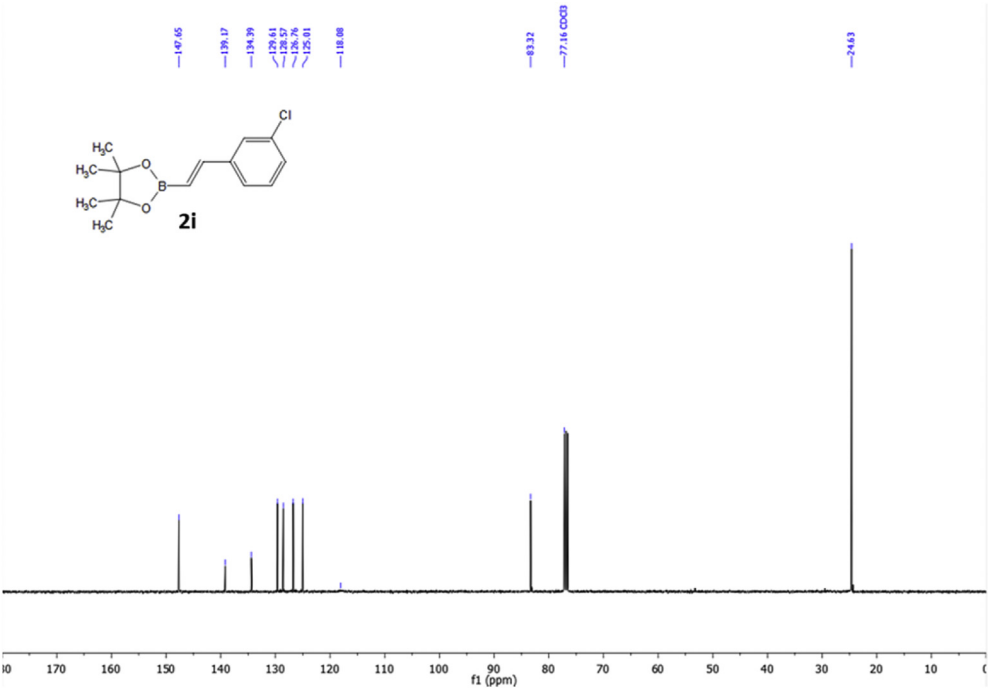

Fig. 20. ^{13}C NMR spectra of compound **2i** (100 MHz) in $CDCl_3$.

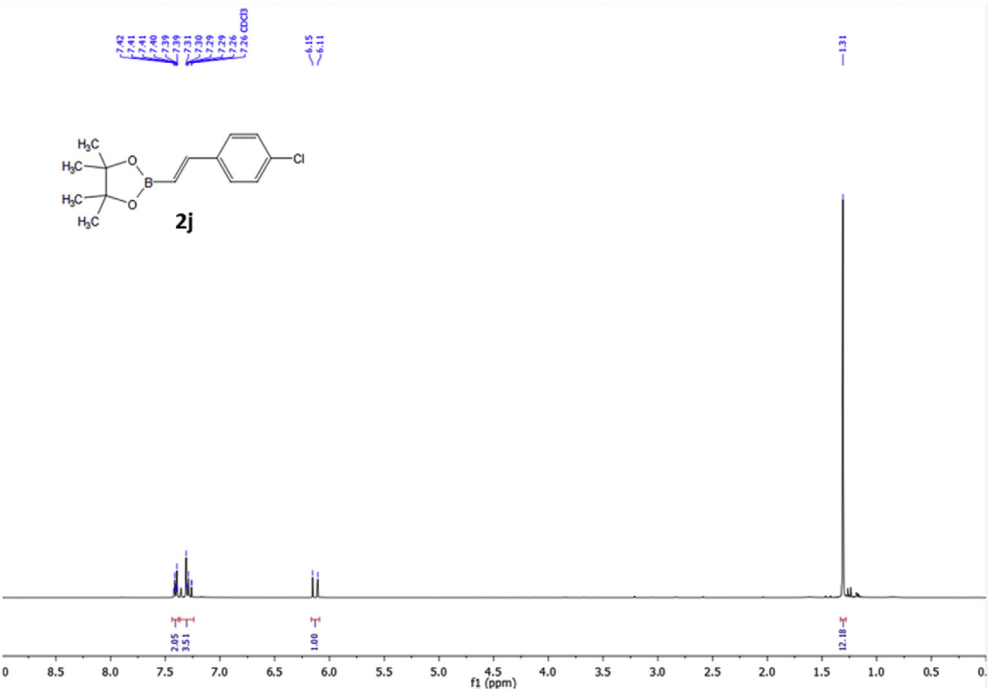

Fig. 21. ^{1}H NMR spectra of compound **2j** (400 MHz) in $CDCl_3$.

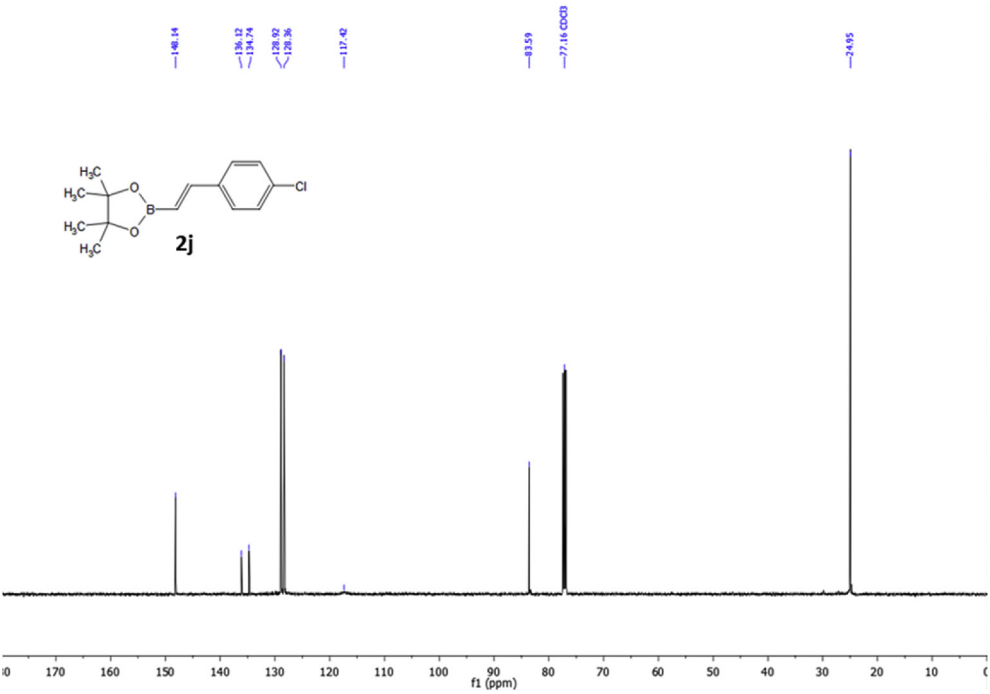

Fig. 22. ^{13}C NMR spectra of compound **2j** (100 MHz) in $CDCl_3$.

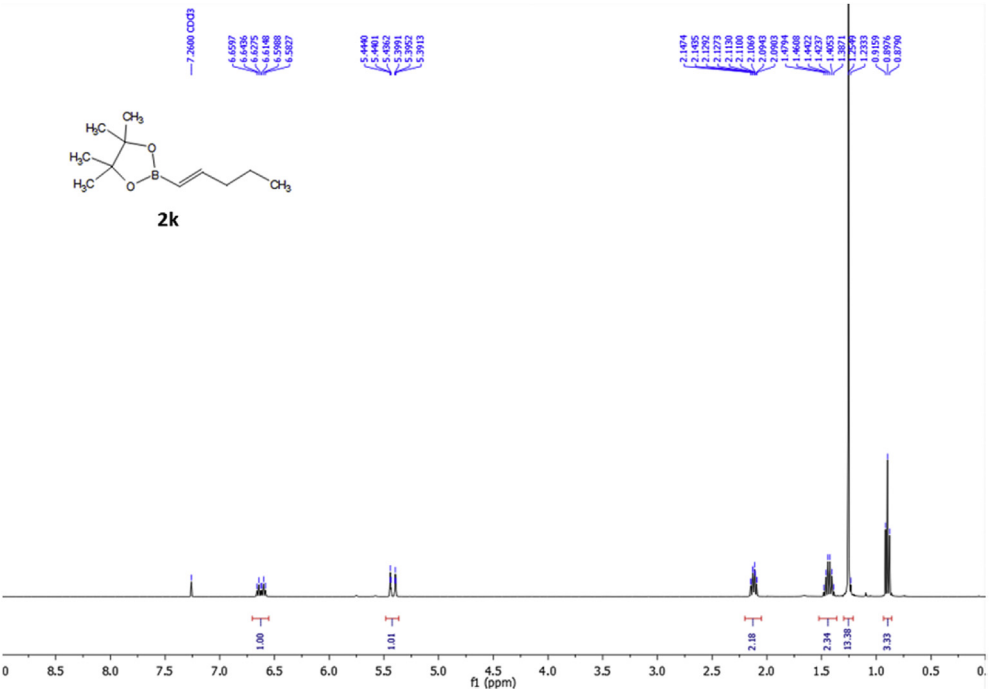

Fig. 23. ^{1}H NMR spectra of compound **2k** (400 MHz) in $CDCl_3$.

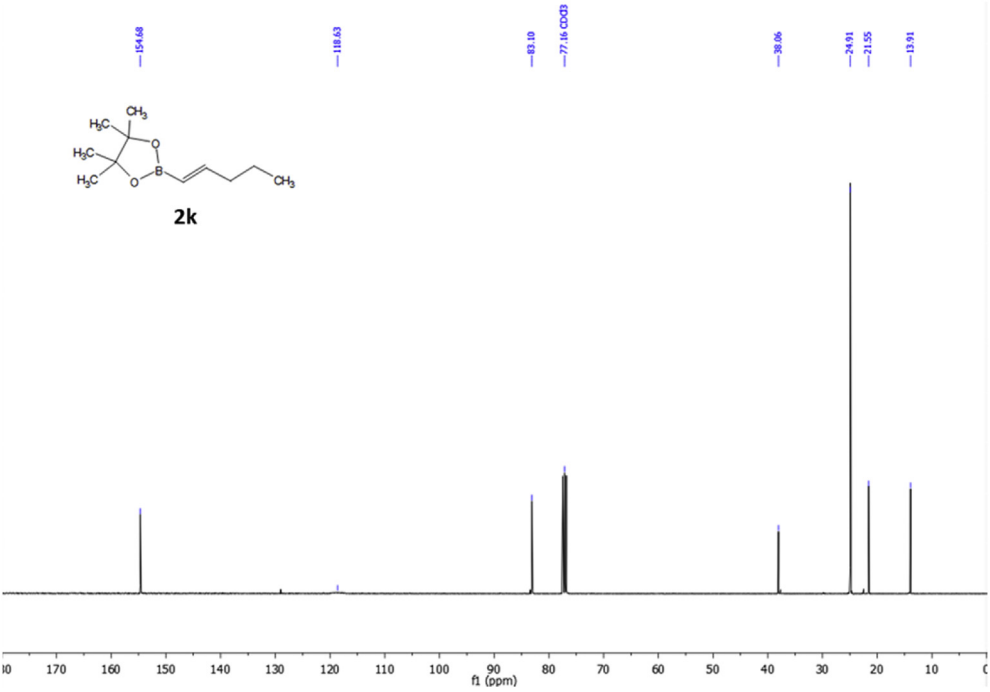

Fig. 24. ^{13}C NMR spectra of compound **2k** (100 MHz) in $CDCl_3$.

Acquisition Parameter

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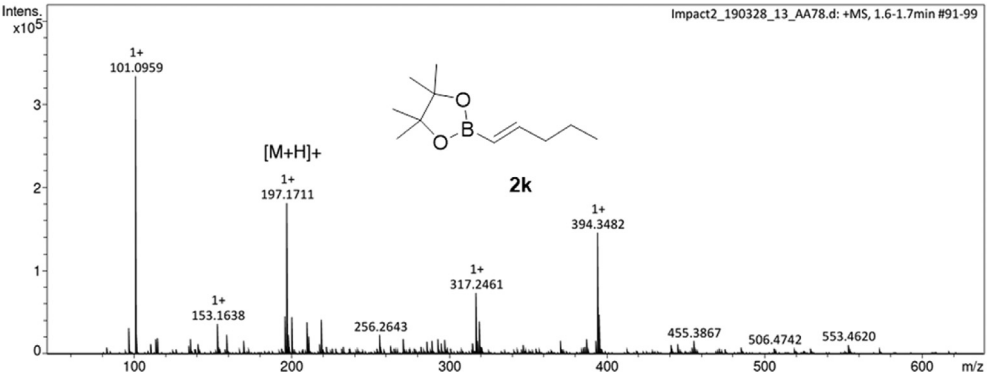

Fig. 25. ESI-MS spectra of compound **2k**.

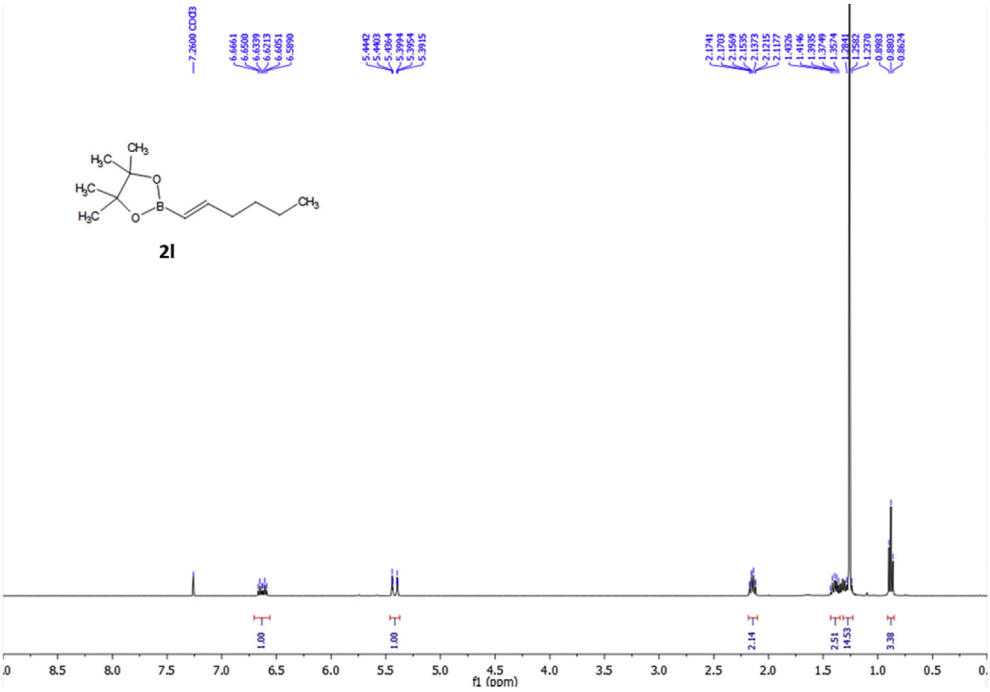

Fig. 26. ^{1}H NMR spectra of compound **2l** (400 MHz) in $CDCl_3$.

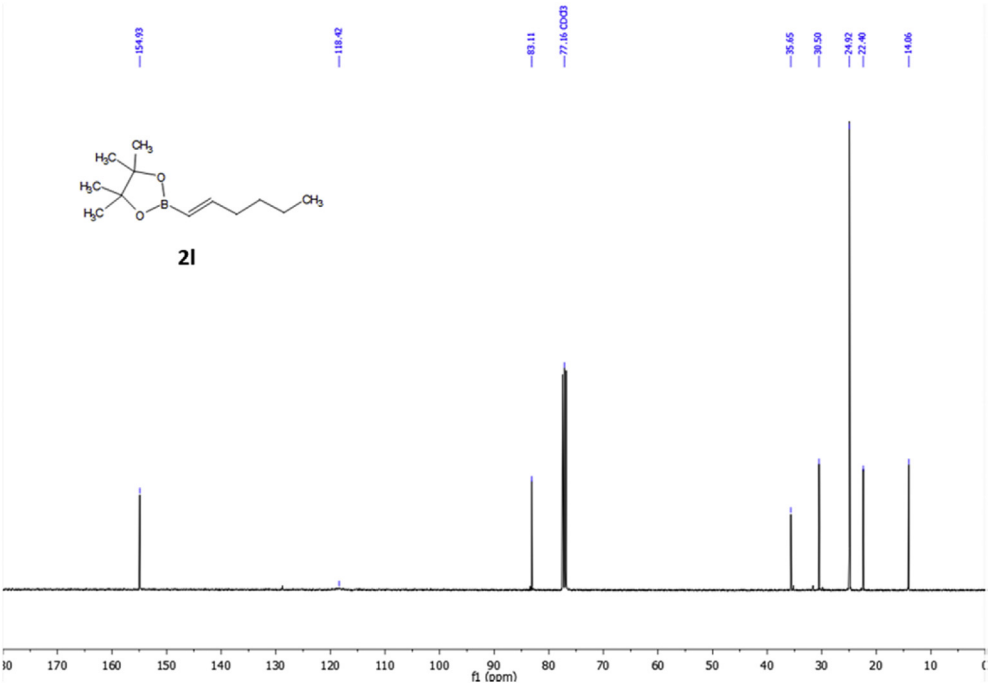

Fig. 27. ^{13}C NMR spectra of compound **2l** (100 MHz) in $CDCl_3$.

Acquisition Parameter

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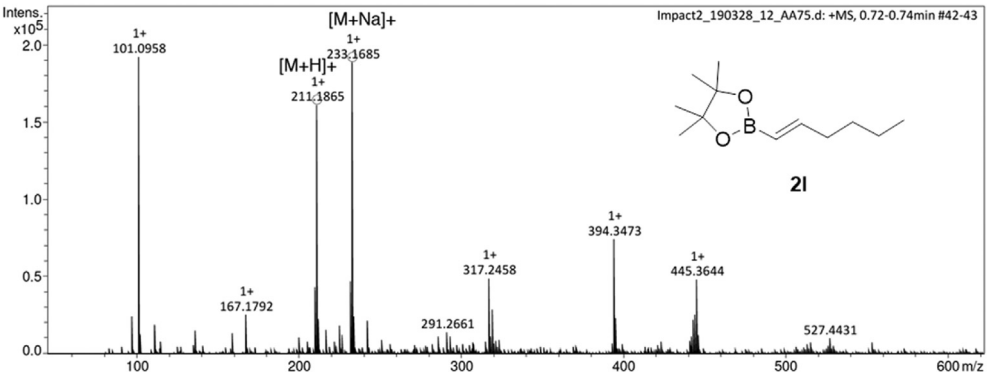

Fig. 28. ESI-MS spectra of compound **2l**.

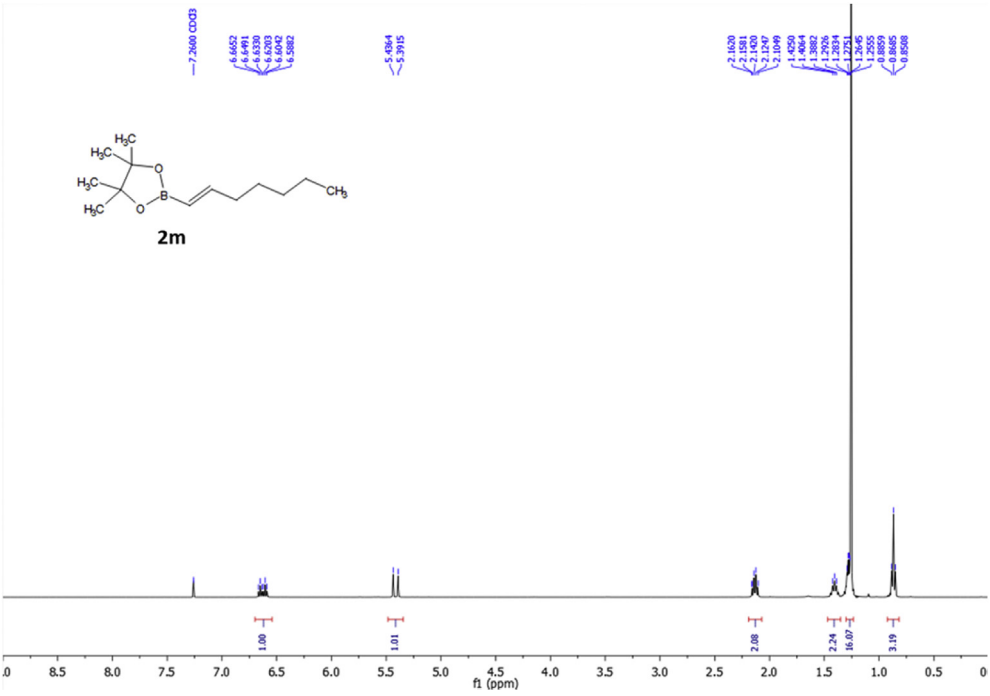

Fig. 29. ^{1}H NMR spectra of compound **2m** (400 MHz) in $CDCl_3$.

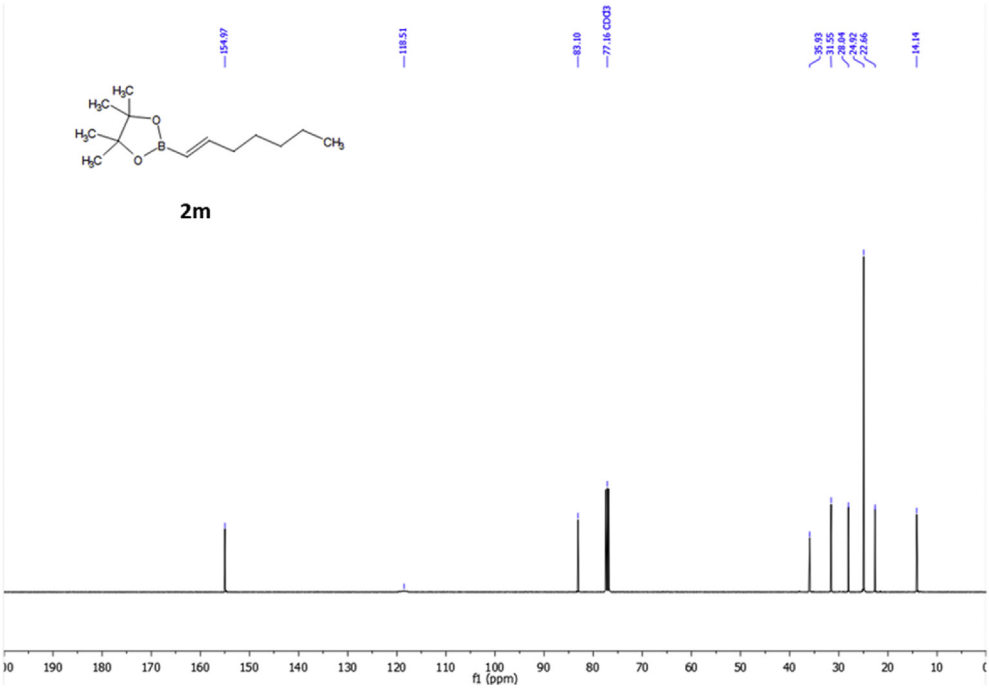

Fig. 30. ^{13}C NMR spectra of compound **2m** (100 MHz) in $CDCl_3$.

Acquisition Parameter

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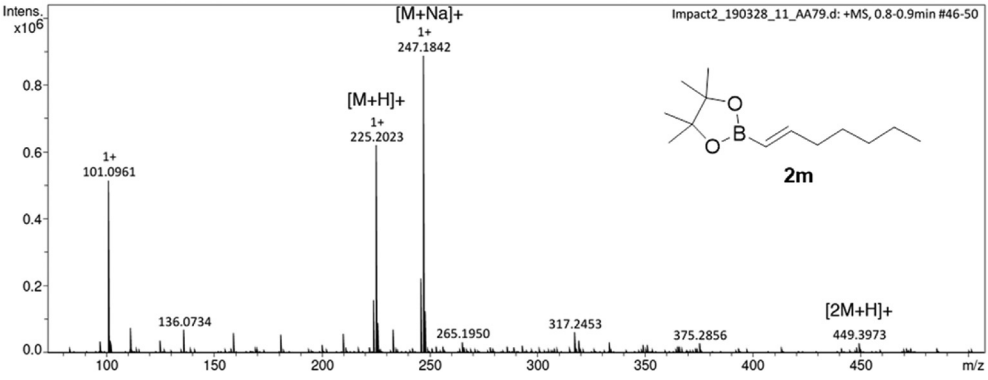

Fig. 31. ESI-MS spectra of compound **2m**.

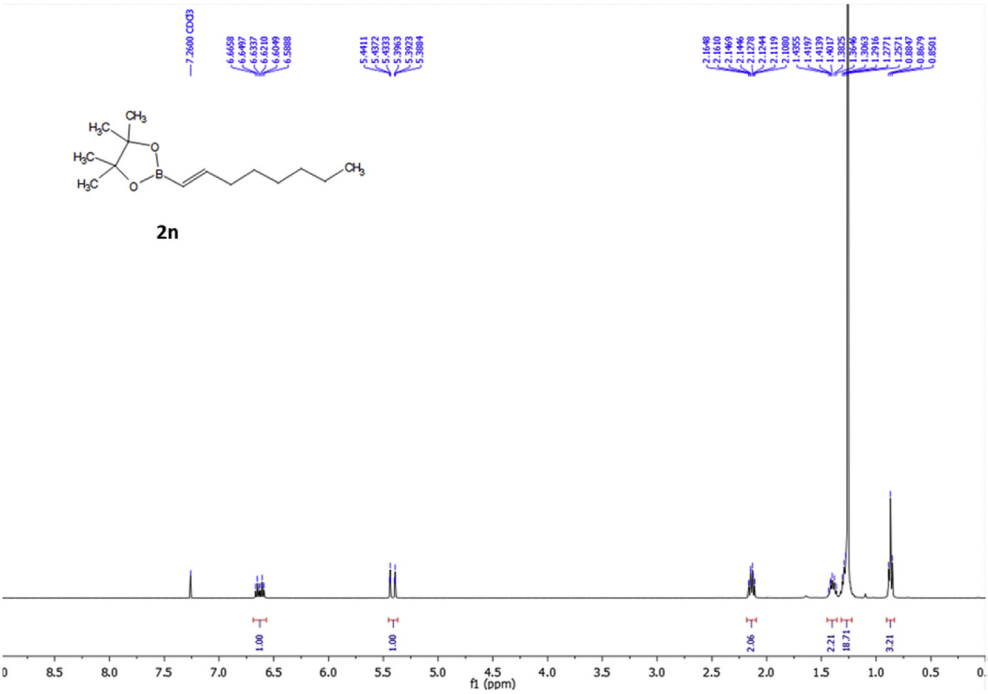

Fig. 32. ^{1}H NMR spectra of compound **2n** (400 MHz) in $CDCl_3$.

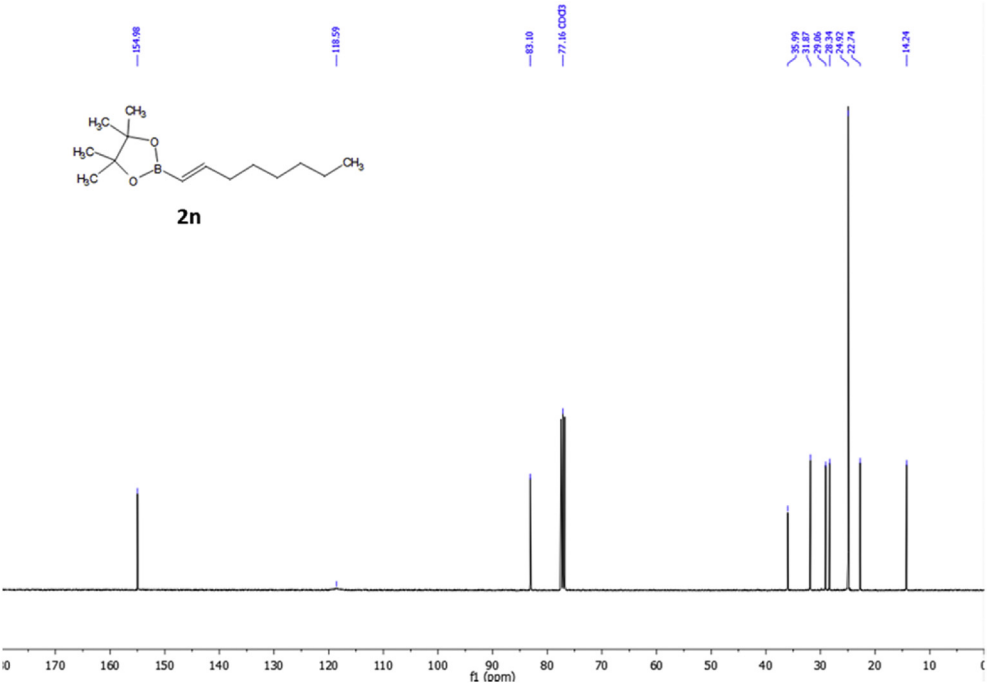

Fig. 33. ^{13}C NMR spectra of compound **2n** (100 MHz) in $CDCl_3$.

Acquisition Parameter

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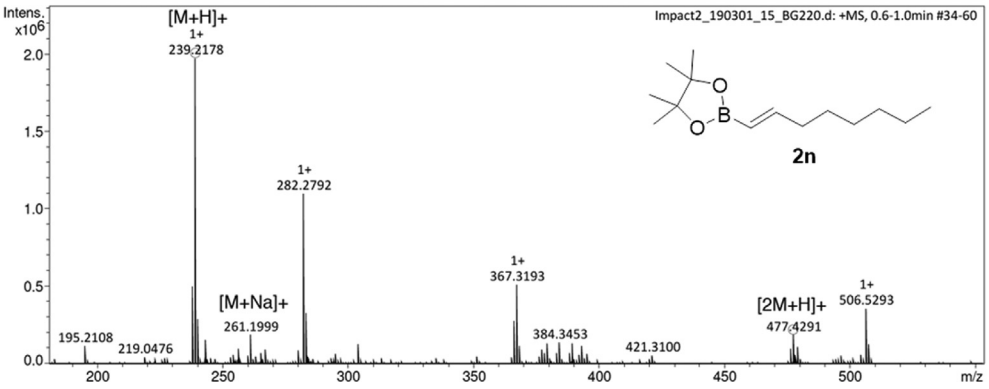

Fig. 34. ESI-MS spectra of compound **2n**.

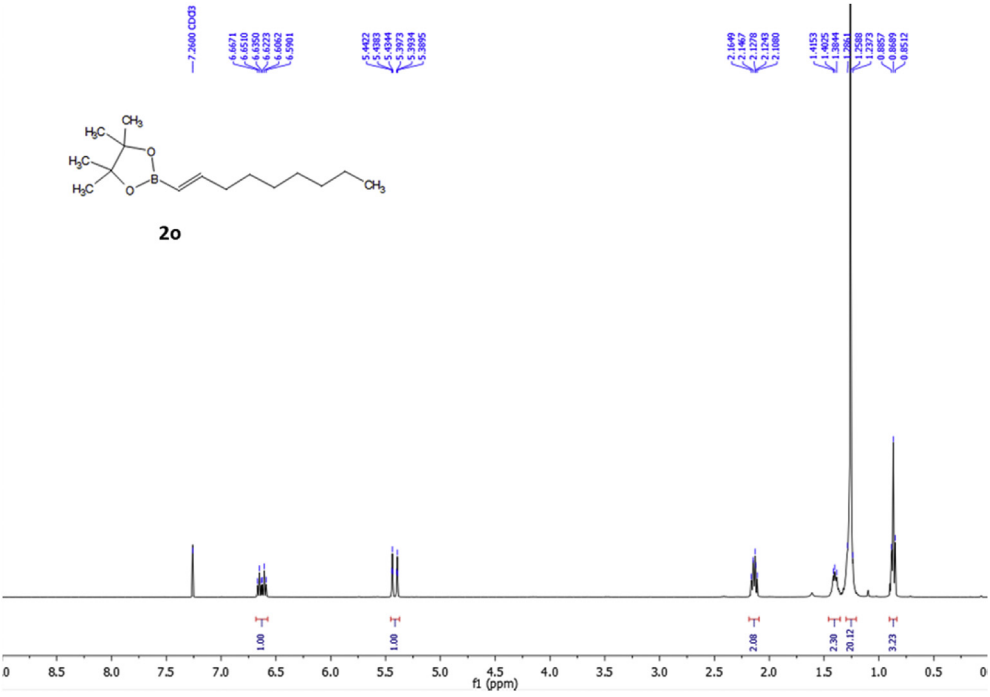

Fig. 35. ^{1}H NMR spectra of compound **2o** (400 MHz) in $CDCl_3$.

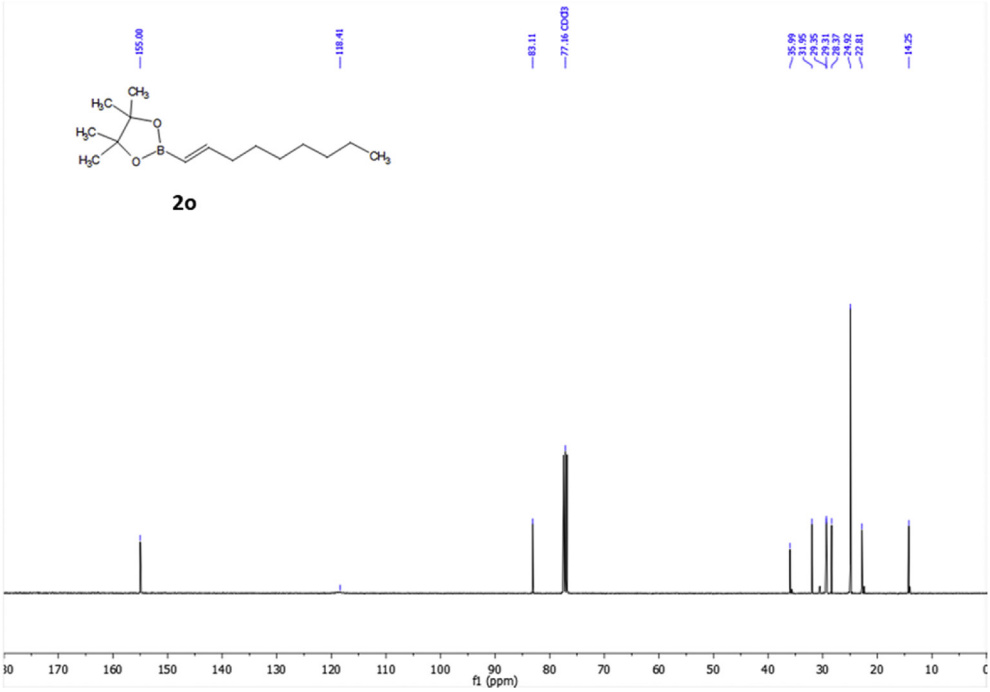

Fig. 36. ^{13}C NMR spectra of compound **2o** (100 MHz) in $CDCl_3$.

Acquisition Parameter

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Scan End	1000 m/z	Set Collision Cell RF	750.0 Vpp	Set Divert Valve	Source

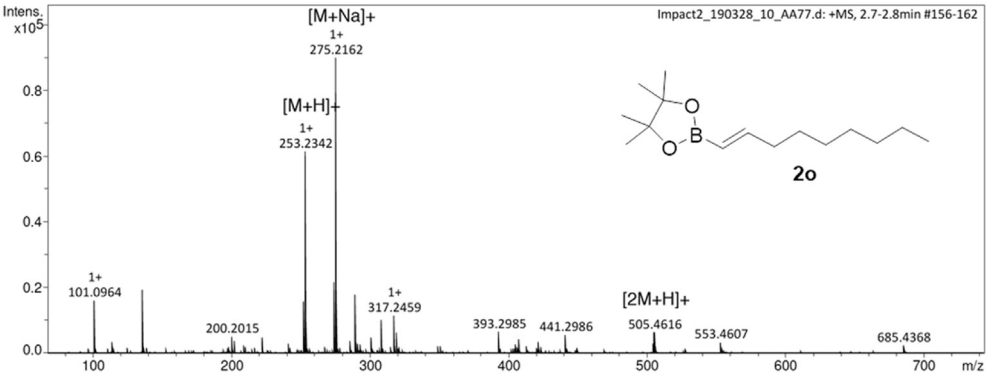


Fig. 37. ESI-MS spectra of compound **2o**.

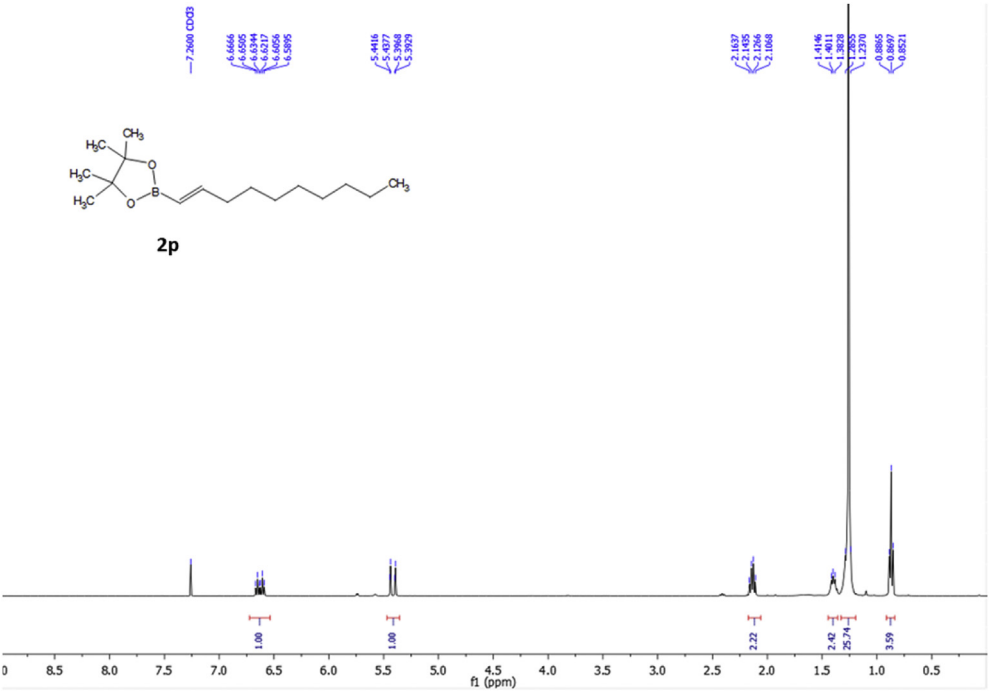


Fig. 38. ^{1}H NMR spectra of compound **2p** (400 MHz) in $CDCl_3$.

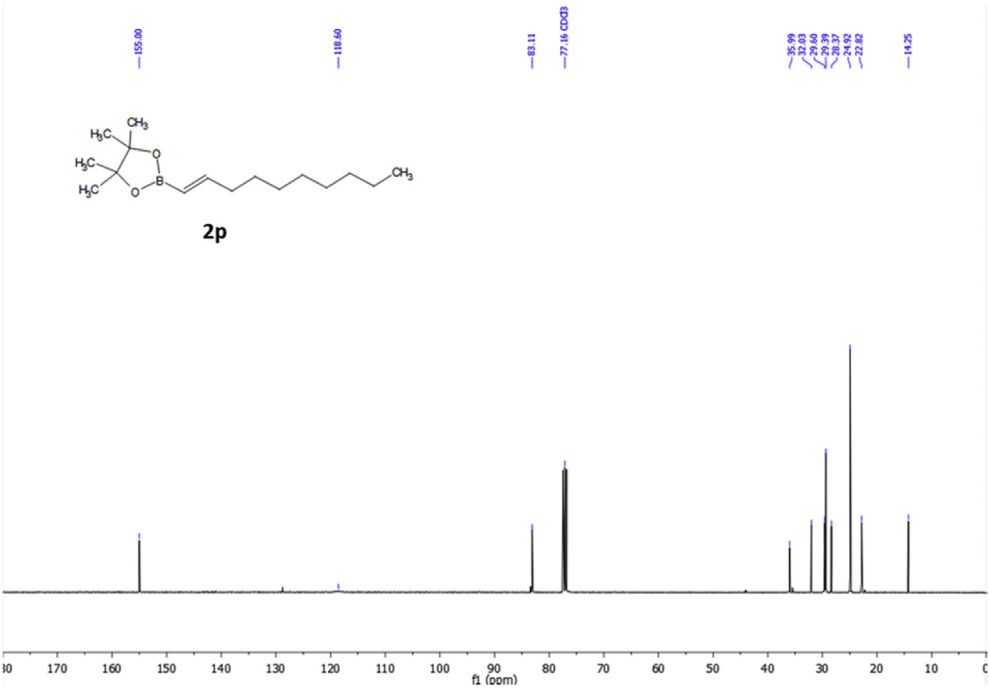


Fig. 39. ^{13}C NMR spectra of compound **2p** (100 MHz) in $CDCl_3$.

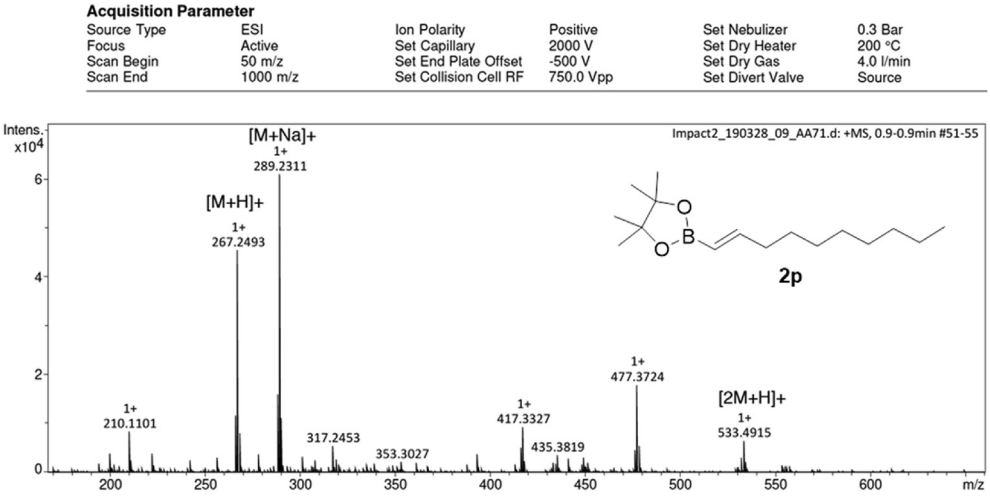


Fig. 40. ESI-MS spectra of compound **2p**.

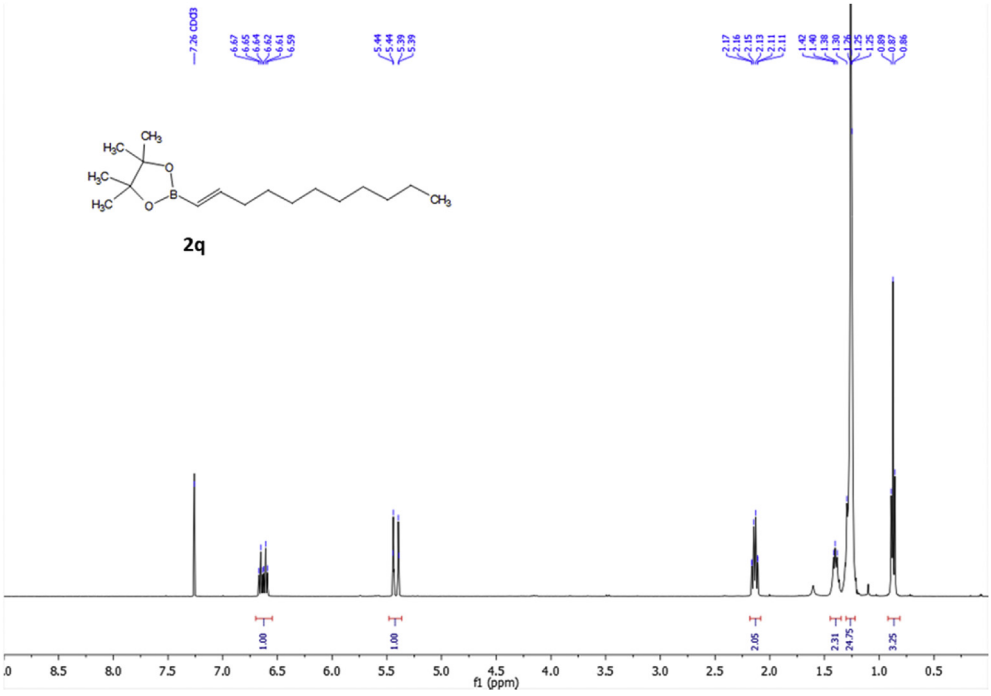


Fig. 41. ^{1}H NMR spectra of compound **2q** (400 MHz) in $CDCl_3$.

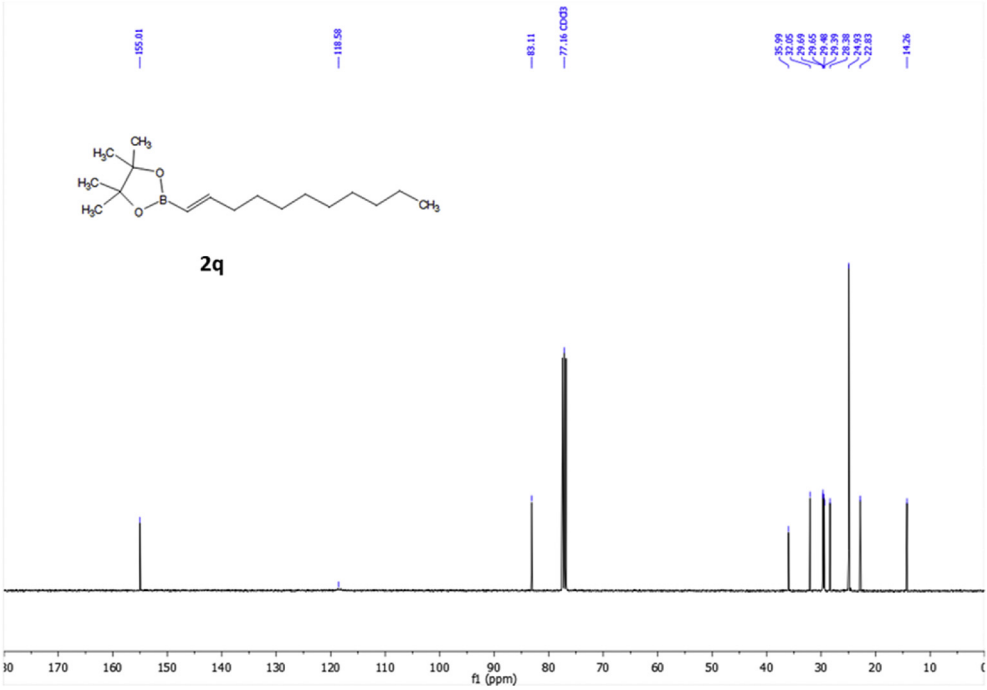


Fig. 42. ^{13}C NMR spectra of compound **2q** (100 MHz) in $CDCl_3$.

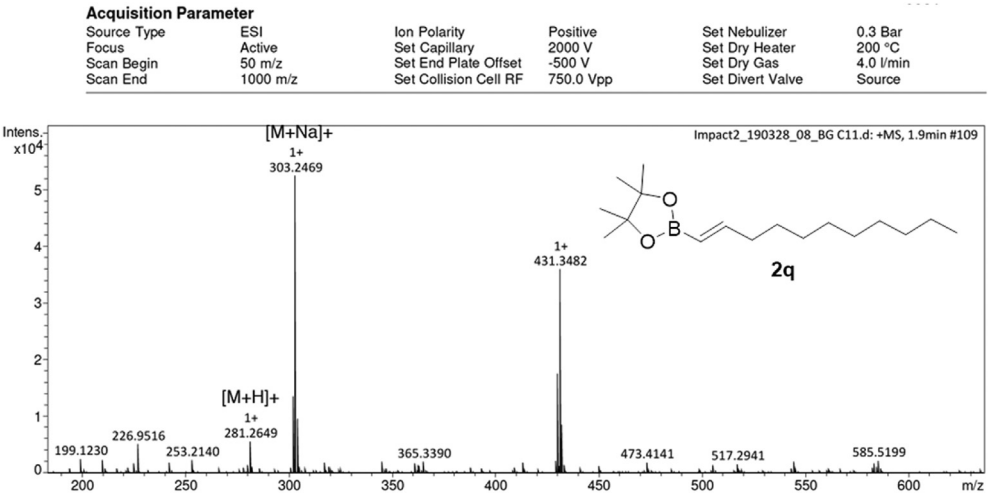


Fig. 43. ESI-MS spectra of compound **2q**.

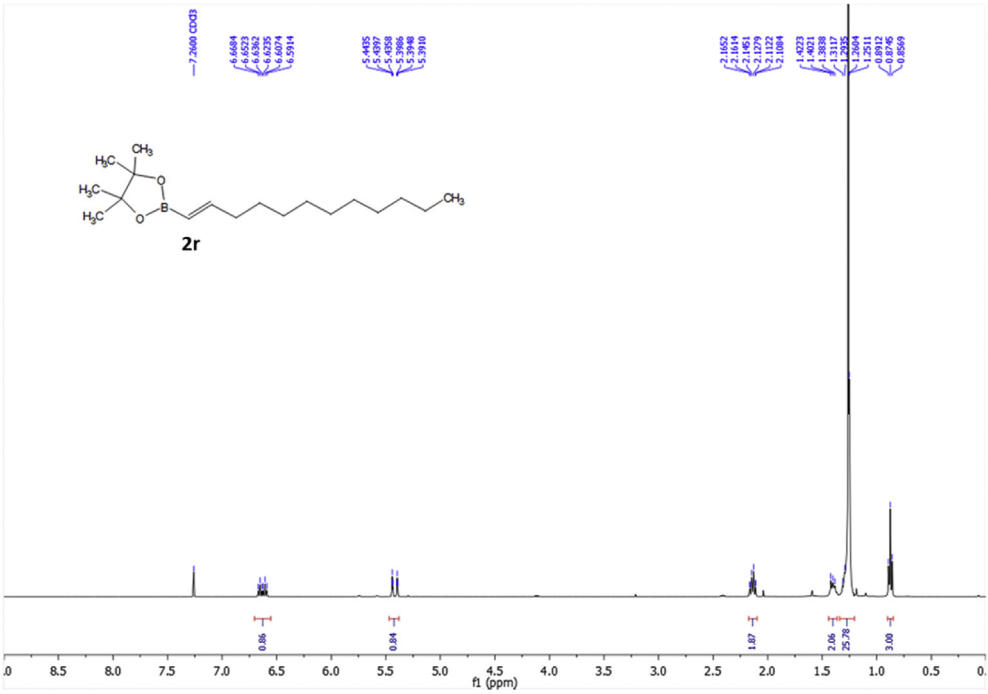


Fig. 44. ^{1}H NMR spectra of compound **2r** (400 MHz) in $CDCl_3$.

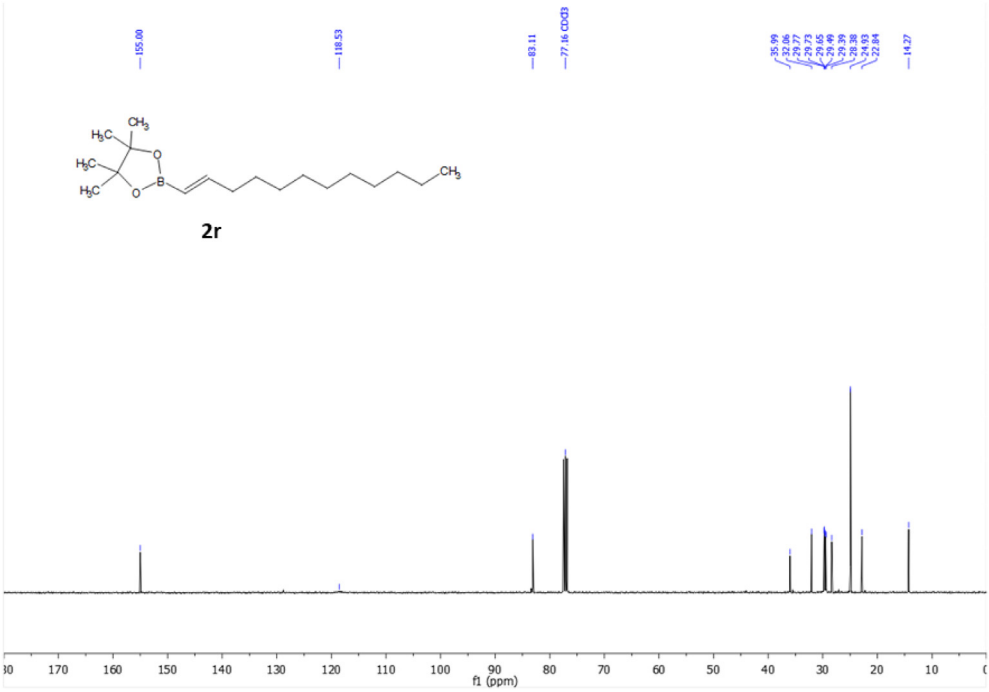


Fig. 45. ^{13}C NMR spectra of compound **2r** (100 MHz) in $CDCl_3$.

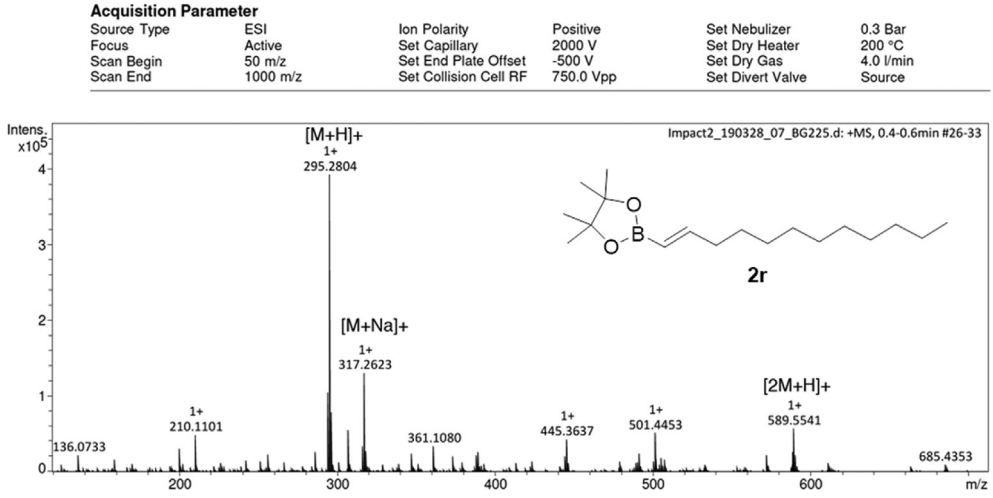
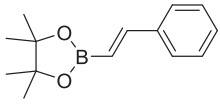


Fig. 46. ESI-MS spectra of compound **2r**.

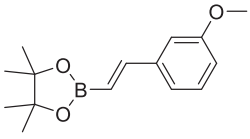
2.3. Characterization data

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



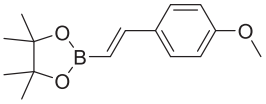
Purification by flash chromatography with cyclohexane: diethyl ether (97:3) to obtain a pale yellow oil (336 mg, 62%). ^{1}H NMR (400MHz, DMSO-*d*6) δ 7.58 (d, *J* 8.0 Hz, 2H), 7.40–7.28 (m, 4H), 6.14 (d, *J* 18.5 Hz, 1H), 1.24 (s, 12H); ^{13}C NMR (100 MHz, DMSO-*d*6) δ 149.21, 136.86, 129.14, 128.68, 127.02, 116.53, 83.02, 24.63.

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



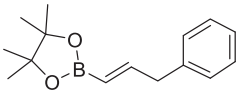
Purification by flash chromatography with cyclohexane: diethyl ether (70:30) to obtain a colorless oil (374 mg, 60%). ^{1}H NMR (400 MHz, CD_3OD) δ 7.32 (d, *J* 18.4 Hz, 1H), 7.25 (m, 1H), 7.10–7.02 (m, 2H), 6.87 (m, 1H), 6.10 (d, *J* 18.4 Hz, 1H), 3.80 (s, 3H), 1.29 (s, 12H); ^{13}C NMR (100 MHz, CD_3OD) δ 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]*



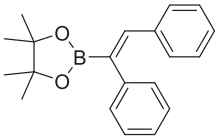
Purification by flash chromatography with cyclohexane: diethyl ether (70:30) to obtain a white solid (361.3 mg, 58%). ^{1}H NMR (400 MHz, CD_3OD) δ 7.44 (m, 2H), 7.30 (d, *J* 18.4 Hz, 1H), 6.89 (m, 2H), 5.94 (d, *J* 18.4 Hz, 1H), 3.80 (s, 3H), 1.29 (s, 12H); ^{13}C NMR (100 MHz, CD_3OD) δ 162.03, 150.72, 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]*



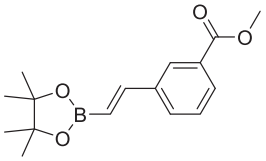
Purification by flash chromatography with cyclohexane: diethyl ether (90:10) to obtain a colorless oil (455.2 mg, 79%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 (m, 2H), 7.23–7.09 (m, 3H), 6.77 (dt, *J* 17.8 Hz, 6.3 Hz, 1H), 5.46 (dt, *J* 17.8 Hz, 1.5 Hz, 1H), 3.49 (dd, *J* 6.3 Hz, 1.5 Hz, 2H), 1.26 (s, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 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]*



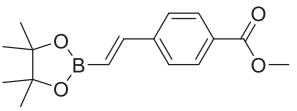
Purification by flash chromatography with pentane: diethyl ether (97:3) to obtain a pale yellow solid (202.2 mg, 28%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (s, 1H), 7.28–7.24 (m, 2H), 7.21–7.18 (m, 1H), 7.17–7.14 (m, 2H), 7.11–7.09 (m, 3H), 7.05–7.03 (m, 2H), 1.20 (s, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 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**)*



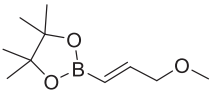
Purification by flash chromatography with petroleum ether (40–60 °C): ethyl acetate (90:10) to obtain a pale yellow solid (446.0 mg, 65%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16 (t, *J* 1.6 Hz, 1H), 7.95 (dt, *J* 7.8 Hz, 1.6 Hz, 1H), 7.66 (dt, *J* 7.8 Hz, 1.6 Hz, 1H), 7.47–7.36 (m, 2H), 6.24 (d, *J* 18.4 Hz, 1H), 3.91 (s, 3H), 1.31 (s, 12H); ^{13}C NMR (100MHz, $CDCl_3$) δ 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]*



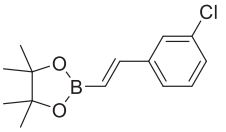
Purification by flash chromatography with petroleum ether (40–60 °C): ethyl acetate (90:10) to obtain a colourless solid (330.9 mg, 48%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (d, *J* 8.3 Hz, 2H), 7.53 (d, *J* 8.3 Hz, 2H), 7.40 (d, *J* 18.4 Hz, 1H), 6.27 (d, *J* 18.4 Hz, 1H), 3.90 (s, 3H), 1.31 (s, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 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]*



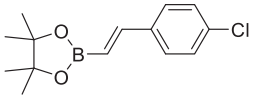
Purification by flash chromatography with petroleum ether (40–60 °C): ethyl acetate (50:50) to obtain a pale yellow oil (164.0 mg, 34%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.62 (dt, *J* 18.2 Hz, 4.8 Hz, 1H), 5.68 (dt, *J* 18.2 Hz, 1.8 Hz, 1H), 3.99 (dd, *J* 4.8 Hz, 1.8 Hz, 2H), 3.34 (s, 3H), 1.26 (s, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 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]*



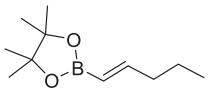
Purification by flash chromatography with pentane: diethyl ether (97:3) to obtain a pale yellow oil (348.48 mg, 55%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 (s, 1H), 7.37–7.25 (m, 4H), 6.17 (d, *J* 18.4 Hz, 1H), 1.32 (s, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.65, 139.17, 134.39, 129.61, 128.57, 126.76, 125.01, 118.08, 83.32, 24.63.

*2.3.10. (*E*)-2-(4-chlorostyryl)boronic acid pinacol ester (**2j**) [2]*



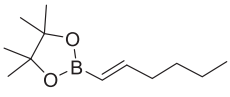
Purification by flash chromatography with cyclohexane: ethyl acetate (60:40) to obtain a pale yellow solid (573.2 mg, 92%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (m, 2H), 7.37–7.24 (m, 3H), 6.13 (d, *J* 18.4 Hz, 1H), 1.31 (s, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.14, 136.12, 134.74, 128.92, 128.36, 117.42, 83.59, 24.95.

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



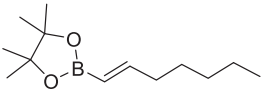
Purification by flash chromatography with pentane: diethyl ether (97:3) to obtain a pale yellow oil (161.9 mg, 35%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.62 (dt, *J* 18.0 Hz, 6.4 Hz, 1H), 5.42 (dt, *J* 18.0 Hz, 1.6 Hz, 1H), 2.11 (m, 2H), 1.43 (sext., *J* 7.4 Hz, 2H), 1.24 (s, 12H), 0.90 (t, *J* 7.4 Hz,3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.68, 118.63, 83.10, 38.06, 24.91, 21.55, 13.91; HRMS (ESI): calcd. for $C_{11}H_{22}BO_2$: 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%). ^{1}H NMR (400 MHz, $CDCl_3$) 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), 1.24–1.28 (m, 14H), 0.88 (t, *J* 7.2 Hz, 3H); ^{13}C 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 $C_{12}H_{23}BNaO_2$: 233.1683; found $[M+Na]^+$: 233.1685.

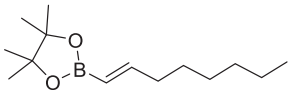
*2.3.13. (*E*)-2-(hept-1-en-1-yl)boronic acid pinacol ester (**2m**) [9]*



Purification by flash chromatography with pentane: diethyl ether (97:3) to obtain a pale yellow oil (339.8 mg, 64%). ^{1}H NMR (400 MHz, $CDCl_3$) 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), 1.26–1.29 (m, 16H), 0.88 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.97, 118.51,

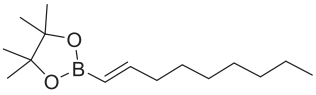
83.10, 35.93, 31.55, 28.04, 24.92, 22.66, 14.14; HRMS (ESI): calcd. for $C_{13}H_{25}BNaO_2$: 247.1840 found $[M+Na]^+$: 247.1842.

*2.3.14. (*E*)-2-(oct-1-en-1-yl)boronic acid pinacol ester (**2n**) [9]*



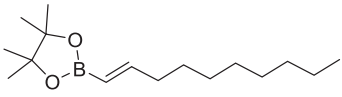
Purification by flash chromatography with pentane: diethyl ether (97:3) to obtain a pale yellow oil (315.1 mg, 58%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.63 (dt, *J* 17.9 Hz, 6.4 Hz, 1H), 5.41 (dt, *J* 17.9 Hz, 1.6 Hz, 1H), 2.14 (m, 2H), 1.40 (m, 2H), 1.26 (s, 18H), 0.86 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.98, 118.59, 83.10, 35.99, 31.87, 29.06, 28.34, 24.92, 22.74, 14.24; HRMS (ESI): calcd. for $C_{14}H_{28}BO_2$: 239.2177; found $[M+H]^+$: 239.2178.

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



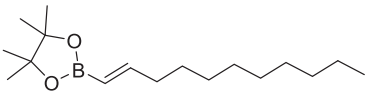
Purification by flash chromatography with pentane: diethyl ether (97:3) to obtain a pale yellow oil (282.4 mg, 50%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.63 (dt, *J* 17.9 Hz, 6.4 Hz, 1H), 5.42 (dt, *J* 17.9 Hz, 1.6 Hz 1H), 2.13 (m, 2H), 1.40 (m, 2H), 1.26 (s, 20H), 0.86 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 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 $C_{15}H_{29}BNaO_2$: 275.2153 found $[M+Na]^+$: 275.2162.

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



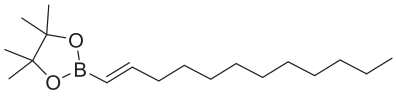
Purification by flash chromatography with pentane: diethyl ether (97:3) to obtain a pale yellow oil (388.4 mg, 62%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.63 (dt, *J* 18.0 Hz, 6.4 Hz, 1H), 5.42 (dd, *J* 18.0 Hz, 1.6 Hz, 1H), 2.14 (m, 2H), 1.40 (m, 2H), 1.26 (s, 22H), 0.86 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 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, $CDCl_3$) δ 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), 1.30–1.25 (m, 24H), 0.86 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 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 $[M+Na]^+$: 303.2469.

*2.3.18. (E)-2-(dodec-1-en-1-yl)boronic acid pinacol ester (**2r**) [12]*



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, $CDCl_3$) δ 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), 1.30–1.25 (m, 26H), 0.87 (t, *J* 6.9 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 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 $[M+H]^+$: 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.

References

[1] B. Gioia, A. Arnaud, S. Radix, N. Walchshofer, A. Doléans-Jordheim, L. Rocheblave, Solvent- and metal-free hydroboration of alkynes under microwave irradiation, Tetrahedron Lett. 61 (2020) 151596.
[2] W. Lu, Z. Shen, Direct synthesis of alkenylboronates from alkenes and pinacol diboron via copper catalysis, Org. Lett. 21 (2019) 142–146, https://doi.org/10.1021/acs.orglett.8b03599.
[3] Z. Liu, W. Wei, L. Xiong, Q. Feng, Y. Shi, N. Wang, L. Yu, Selective and efficient synthesis of *trans*-arylvinylboronates and *trans*-hetarylvinylboronates using palladium catalyzed cross-coupling, New J. Chem. 41 (2017) 3172–3176, https://doi.org/10.1039/c6nj03984g.
[4] S. Tanaka, Y. Saito, T. Yamamoto, T. Hattori, Electrophilic borylation of terminal alkenes with BBr_3/2,6-disubstituted pyridines, Org. Lett. 20 (2018) 1828–1831, https://doi.org/10.1021/acs.orglett.8b00335.
[5] M. Fleige, J. Möbus, T. vom Stein, F. Glorius, D.W. Stephan, Lewis acid catalysis: catalytic hydroboration of alkynes initiated by Piers' borane, Chem. Commun. 52 (2016) 10830–10833, https://doi.org/10.1039/c6cc05360b.
[6] K. Shirakawa, A. Arase, M. Hoshi, Preparation of (E)-1-alkenylboronic acid Pinacol esters via transfer of Alkenyl Group from boron to boron, Synthesis (2004) 1814–1820, https://doi.org/10.1055/s-2004-829165.
[7] J. Zhao, Z. Niu, H. Fu, Y. Li, Ligand-free hydroboration of alkynes catalyzed by heterogeneous copper powder with high efficiency, Chem. Commun. 50 (2014) 2058–2060, https://doi.org/10.1039/c3CC48670b.
[8] E.K. Edelstein, S. Namirembe, J.P. Morken, Enantioselective conjunctive cros-coupling of bis-(alkenyl)borates: a general synthesis of chiral allylboron reagents, J. Am. Chem. Soc. 139 (2017) 5027–5030, https://doi.org/10.1021/jacs.7b01774.
[9] D.P. Ojha, K.R. Prabhu, Pd-catalyzed hydroborylation of alkynes: a ligand controlled regioselectivity switch for the synthesis of α- or β-vinylboronates, Org. Lett. 18 (2016) 432–435, https://doi.org/10.1021/acs.orglett.5b03416.
[10] S. Mandal, P.K. Verma, K. Geetharani, Lewis acid catalysis: regioselective hydroboration of alkynes and alkenes promoted by scandium triflate, Chem. Commun. 54 (2018) 13690–13693, https://doi.org/10.1039/c8cc8361d.
[11] Z.J. Yao, S. Hong, W. Zhang, M. Liu, W. Deng, Copper-catalyzed regioselective hydroboration of terminal alkynes in aqueous medium, Tetrahedron Lett. 57 (2016) 910–913, https://doi.org/10.1016/j.tetlet.2016.01.049.
[12] H.E. Ho, N. Asao, Y. Yamamoto, T. Jin, Carboxylic acid-catalyzed highly efficient and selective hydroboration of alkynes with pinacolborane, Org. Lett. 16 (2014) 4670–4673, https://doi.org/10.1021/ol502285s.