Electronic Supplementary Material (ESI) for ChemComm.

Supplementary Information

Solvent-free hydroboration of alkenes and alkynes catalyzed by rhodium-ruthenium nanoparticles on carbon nanotubes

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1. General Remarks

Starting materials were purchased from commercial suppliers and used as received unless otherwise stated. All reactions were performed under an inert atmosphere. Yields refer to isolated compounds estimated to be > 95% pure as determined by ^{1}H-NMR. TLC was performed on Merck TLC Silica Gel 60 $F_{254.}$ Compounds were detected under UV light or by treatment with a solution of potassium permanganate in ethanol and heating. Chromatographic separations were carried out on Merck Geduran SI-60 (0.063–0.200 mm, 70-230 mesh ASTM). Centrifugations were carried out on PowerSpin™ BX C885 Centrifuge for 6 minutes at 5000 rpm. Mass-spectra: EI (Electron impact) mass spectra were recorded on a MAT 95XP spectrometer (70 eV, Thermo ELECTRON CORPORATION). NMR spectra were recorded on a Bruker Avance DRX 400 spectrometer at 400 MHz (^{1}H) and at 100 MHz (^{13}C). Chemical shifts (δ) are provided in ppm, and spectrum refers to residual non-deuterated solvent resonances. Unless otherwise specified, ultrasonic mixing was achieved using a Branson sonifier 550 equipped with a 3 mm tapered microtip (300 ms/s pulses, Output power 40%). Photo-polymerization were carried out using a 40 W low-pressure mercury UV lamp (Heraeus) emitting at a wavelength of 254 nm. For HRSTEM experiments, a Titan-G2 probe corrected STEM was used to study the Ru/Rh repartition on the carbon nanotubes. The emitted X-rays were collected by the four detectors with a 0.7 steradian collecting angle. The probe was smaller than 2 Å and its current was almost 250 pA. ChemDraw Professional 18.1 was used to generate the IUPAC names of the compounds.

2. RhRu particle synthesis

The synthesis of nanoparticles was achieved using a previously reported procedure.[1] A solution of $RhCl_3 \cdot 3H_2O$ (39.5 mg, 0.15 mmol) and $RuCl_3 \cdot 6H_2O$ (47.5 mg, 0.15 mmol) in water (5 mL) was mixed with ethylene glycol (100 mL). An aqueous solution of NaOH 0.5 M (5 mL) was added to the stirred mixture and the reaction was heated to 160 °C. After 3 h, a stable transparent brown homogeneous colloid of RhRuNPs was formed.

3. Assembly of RhRu nanoparticles on carbon nanotubes

The assembly of RhRu nanoparticles at the surface of carbon nanotubes was achieved according to our previously reported procedure.[2]

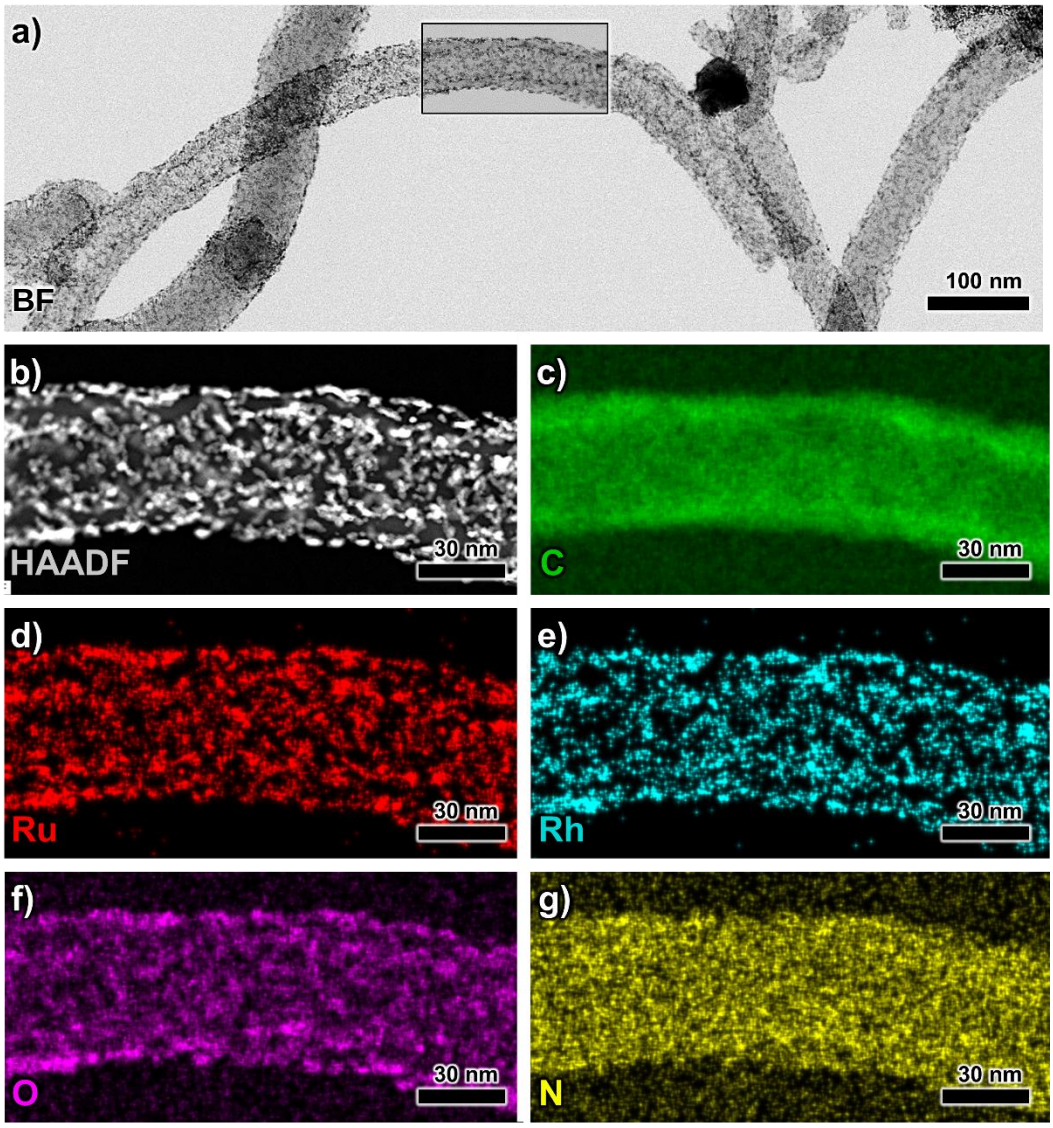


Figure S1. (a) Bright field (BF) TEM image of the RhRuCNT hybrid. (b) High angle annular dark field (HAADF) image of the boxed region and associated EDS mapping for (c) carbon; (d) ruthenium; (e) rhodium; (f) oxygen; (g) nitrogen.

4. General procedure for hydroboration reactions

*The reaction of 1-hexene **1a** with pinacolborane **2** is given as a representative example:*

RhRuCNT (50 µL of an aqueous suspension, [Rh] = 3.8 mM and [Ru] = 4.3 mM, 0.04 mol%) was added to acetone (1 mL) in an Eppendorf tube and the mixture was contrifuged (5 000 ×*g*, 6 min). The supernatant was discarded and the pelleted catalyst was dried by slow purging with nitrogen gas. In the glove-box, 1-hexene **1a** (84 mg, 1 mmol) and pinacolborane **2** (141 mg, 1.1 mmol) were added under nitrogen and the mixture was stirred for 24 h at room temperature. After completion of the reaction,

ethanol (0.5 mL) was added and the mixture was centrifuged (2 000 ×*g*, 5 min). The supernatant was collected and the pelleted catalyst was washed twice with ethanol (2 × 2 mL) by centrifugation/resuspension. The collected organic phases were concentrated under vacuum and product **3a** was obtained as a colourless oil (96%).

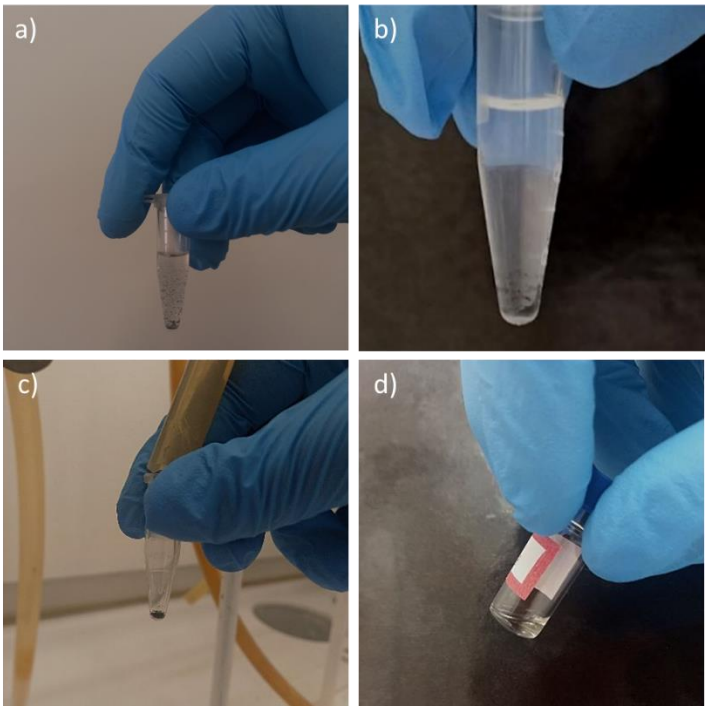


Figure S2. (a) Catalyst before centrifugation; (b) Catalyst after centrifugation; (c) Catalyst dried by slow purging with nitrogen gas; (d) Final product of reaction.

5. Recycling experiments

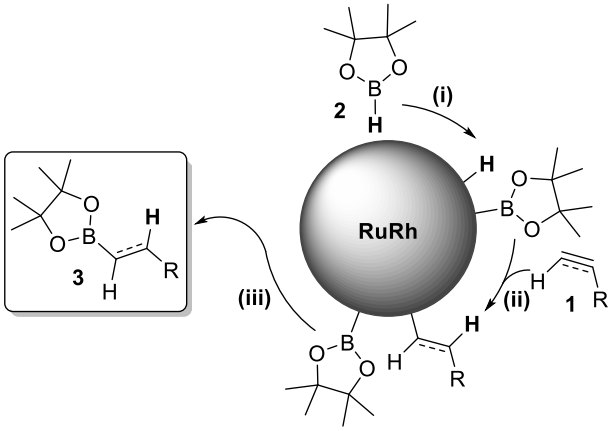
Step 1: RhRuCNT (50 µL of an aqueous suspension, [Rh] = 3.8 mM and [Ru] = 4.3 mM, 0.04 mol%) was added to acetone (1 mL) in an Eppendorf tube and the mixture was contrifuged (2 000 ×*g*, 5 min). The supernatant was discarded and the pelleted catalyst was dried by slow purging with nitrogen gas.

Step 2: 1-Hexene **1a** (84 mg, 1 mmol) and pinacolborane **2** (141 mg, 1.1 mmol) were added under nitrogen and the mixture was stirred for 24 h at room temperature.

Step 3: After completion of the reaction, ethanol (0.5 mL) was added and the mixture was centrifuged (2 000 ×*g*, 5 min). The supernatant was collected and the catalyst was washed twice with ethanol (2 × 2 mL) by centrifugation/resuspension. The collected organic phases were concentrated under vacuum to yield product **3a** as a colourless oil.

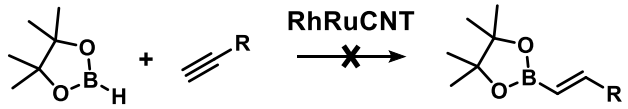
Step 4: The pelleted catalyst was dried under vacuum and used for subsequent cycles by repeating steps 2 and 3.

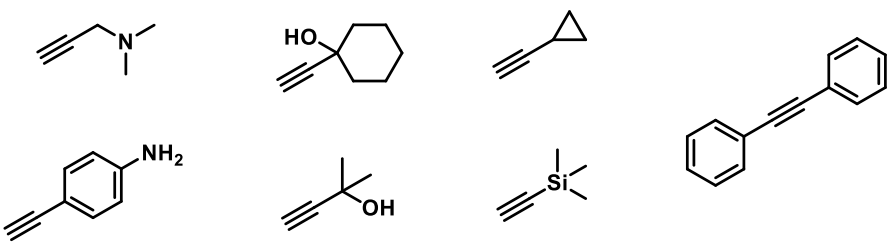
6. Reaction mechanism



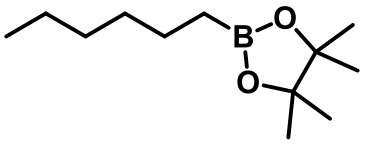
Scheme S1. Postulated mechanism for the RhRuCNT-catalyzed hydroboration reaction.

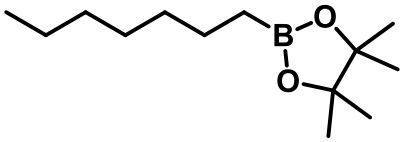
Table S1. Examples of alkynes that proved unreactive under RhRuCNT-catalyzed hydroboration conditions.

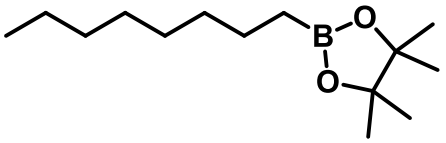


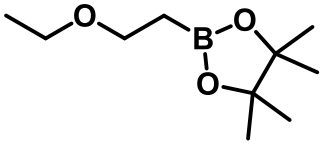


7. Spectroscopic data

2-hexyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3a): 
The general procedure was followed by using 1-hexene (**1a**). Purification by column chromatography on silica gel (DCM) yielded **3a** (96%) as a colorless oil. **^{1}H-NMR** (400 MHz, $CDCl_3$): δ 1.36−1.43 (m, 2H), 1.26 (m, 6H), 1.24 (s, 12H), 0.87 (m, 3H), 0.73 ppm (m, 2H). **^{13}C-NMR** (100 MHz, $CDCl_3$, ppm): δ 83.0, 32.1, 31.6, 24.8, 24.0, 22.6, 14.1 ppm. **HRMS (ESI$^+$, *m/z*):** 213.2026 $[M + H]^+$ (calcd for $C_{12}H_{23}BO_2$, 213.2028).
Data are consistent with the literature.[3]

2-heptyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3b): 
The general procedure was followed by using 1-heptene (**1b**). Purification by column chromatography on silica gel (DCM) yielded **3b** (77%) as a colorless oil. **^{1}H-NMR** (400 MHz, $CDCl_3$): δ 1.44−1.36 (m, 2H), 1.26 (m, 8H), 1.24 (s, 12H), 0.88−0.85 (m, 3H), 0.76 ppm (t, J = 7.7 Hz, 2H). **^{13}C-NMR** (100 MHz, $CDCl_3$): δ 83.0, 32.5, 32.0, 29.2, 24.9, 24.2, 22.8, 14.2 ppm. **GC/MS:** *m*/z (%) 226 (M^+).
Data are consistent with the literature.[4]

4,4,5,5-tetramethyl-2-octyl-1,3,2-dioxaborolane (3c): 
The general procedure was followed by using 1-octene (**1c**). Purification by column chromatography on silica gel (DCM) yielded **3c** (72%) as a colorless oil. **^{1}H-NMR** (400 MHz, $CDCl_3$): δ 1.43−1.35 (m, 2H), 1.30-1.24 (m, 10H), 1.23 (s, 12H), 0.86 (t, J = 6.7 Hz, 3H), 0.76 ppm (t, J = 7.7 Hz, 2H). **^{13}C-NMR** (100 MHz, $CDCl_3$): δ 83.0, 32.6, 32.0, 29.5, 29.4, 25.0, 24.1, 22.8, 14.3 ppm. **GC/MS:** *m*/z (%) 240 (M^+).
Data are consistent with the literature.[5]

2-(2-ethoxyethyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3d): 
The general procedure was followed by using ethyl vinyl ether (**1d**). Purification by column chromatography on silica gel (DCM) yielded **3d** (74%) as a colorless oil. **^{1}H-**

NMR (400 MHz, $CDCl_3$): δ 3.54 (t, J = 8.0 Hz, 2H), 3.45 (q, J = 7.0 Hz, 2H), 1.23 (s, 12H), 1.19– 1.14 ppm (m, 5H). **^{13}C-NMR** (100 MHz, $CDCl_3$): δ 83.2, 67.2, 65.8, 24.9, 15.4 ppm. **HRMS (ESI$^+$, *m*/*z*):** 223.1480 $[M+Na]^+$ (Cald. for $[C_{10}H_{21}BO_3Na]^+$: 223.1481).

Data are consistent with the literature.[6]

4,4,5,5-tetramethyl-2-(2-propoxyethyl)-1,3,2-dioxaborolane (3e): The general procedure was followed by using propyl vinyl ether (**1e**). Purification by column chromatography on silica gel (DCM) yielded **3e** (73%) as a colorless oil. **^{1}H-MR** (400 MHz, $CDCl_3$): δ 3.54 (t, J = 7.9 Hz, 2H), 3.35 (t, J = 6.8 Hz, 2H), 1.56 (sext, J = 7.2 Hz, 2H), 1.23 (s, 12H), 1.15 (t, J = 7.9 Hz, 2H), 0.89 ppm (t, J = 7.4 Hz, 3H). **^{13}C-NMR** (100 MHz, $CDCl_3$): δ 83.2, 72.3, 67.3, 24.9, 23.0, 10.7 ppm. **HRMS (ESI$^+$, *m*/*z*):** 237.1628 $[M+Na]^+$ (Cald. for $[C_{11}H_{23}BO_3Na]^+$: 237.1638).

2-(2-isobutoxyethyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3f): The general procedure was followed by using isobutyl vinyl ether (**1f**). Purification by column chromatography on silica gel (DCM) yielded **3f** (61%) as a colorless oil. **^{1}H-NMR** (400 MHz, $CDCl_3$): δ 3.54 (t, J = 7.8 Hz, 2H), 3.16 (d, J = 6.8 Hz, 2H), 1.84 (m, 1H), 1.24 (s, 12H), 1.15 (t, J = 7.8 Hz, 2H), 0.88 ppm (d, J = 6.7 Hz, 6H). **^{13}C-NMR** (100 MHz, $CDCl_3$): δ 83.2, 77.7, 67.5, 28.5, 24.9, 19.6 ppm. **HRMS (ESI$^+$, *m*/*z*):** 251.1801 $[M+Na]^+$ (Cald. for $[C_{12}H_{25}BO_3Na]^+$: 251.1794).

4,4,5,5-tetramethyl-2-(3-methylpentyl)-1,3,2-dioxaborolane (3g): The general procedure was followed by using 3-methyl-1-pentene (**1g**). Purification by column chromatography on silica gel (DCM) yielded **3g** (69%) as a colorless oil. **^{1}H-NMR** (400 MHz, $CDCl_3$): δ 1.45–1.27 (m, 4H), 1.24 (s, 12H), 1.14–1.05 (m, 1H), 0.87−0.83 (m, 6H), 0.78–0.67 ppm (m, 2H). **^{13}C-NMR** (100 MHz, $CDCl_3$): δ 83.0, 36.8, 30.7, 29.2, 25.0, 18.9, 11.6 ppm. **GC/MS:** *m*/z (%) 212 (M^+).

Data are consistent with the literature.[7]

2-(3-bromopropyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3h): The general procedure was followed by using allyl bromide (**1h**). Purification by column chromatography on silica gel (DCM) yielded **3h** (74%) as a colorless oil. **^{1}H-NMR** (400 MHz, $CDCl_3$): δ 3.44 (t, J = 6.9 Hz, 2H), 2.02−1.94 (m, 2H), 1.23 (s, 12H), 0.94 (t, J = 7.7 Hz, 2H). **^{13}C-NMR** (100 MHz, $CDCl_3$): δ 83.3, 36.4, 27.7, 24.9. **GC/MS:** *m*/z (%) 248 (M^+).

Data are consistent with the literature.[8]

4,4,5,5-tetramethyl-2-(4-(oxiran-2-yl)butyl)-1,3,2-dioxaborolane (3i): The general procedure was followed by using 2-(but-3-en-1-yl)oxirane (**1i**). Purification by column chromatography on silica gel (DCM) yielded **3i** (93%) as a colorless oil. **^{1}H-NMR** (400 MHz, $CDCl_3$): δ 2.88−2.92 (m, 1H), 2.72−2.74 (m, 1H), 2.44−2.46 (m, 1H), 1.43−1.54 (m, 6H), 1.24 (s, 12H), 0.79 ppm (m, 2H). **^{13}C-NMR** (100 MHz, $CDCl_3$): δ 83.0, 52.4, 47.2, 32.3, 28.6, 24.8, 23.9 ppm. **HRMS (ESI$^+$, *m*/*z*):** 227.1819 $[M + H]^+$ (calcd for $C_{12}H_{24}BO_3$, 227.1821).

Data are consistent with the literature.[9]

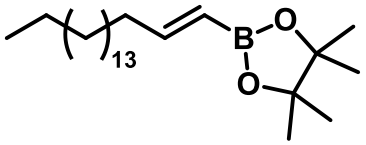
Methyl 11-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)undecanoate (3j): The general procedure was followed by using methyl 10-undecenoate (**1j**). Purification by column chromatography on silica gel (DCM) yielded **3j** (60%) as a colorless oil. **^{1}H-NMR** (400 MHz, $CDCl_3$): δ 3.65 (s, 3H), 2.28 (t, J = 7.6 Hz, 2H), 1.64−1.55 (m, 2H), 1.43−1.35 (m, 2H), 1.31−1.24 (m, 12H), 1.23 (s, 12H), 0.75 ppm (t, J = 7.6 Hz, 2H). **^{13}C-NMR** (100 MHz, $CDCl_3$): δ 174.5, 83.0, 51.6, 34.3, 32.6, 29.6 (2C), 29.5, 29.4, 29.3, 25.1, 24.9, 24.1 ppm. **HRMS (ESI$^+$, *m*/*z*):** 349.2532 $[M+Na]^+$ (Cald. for $[C_{18}H_{35}BO_4Na]^+$: 349.2526).

Data are consistent with the literature.[10]

(*E*)-2-(hex-1-en-1-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3m): The general procedure was followed by using 1-hexyne (**1m**). Purification by column

chromatography on silica gel (DCM) yielded **3m** (97%) as a colorless oil. **^{1}H-NMR** (400 MHz, $CDCl_3$): δ 6.67−6.60 (dt, J = 18 Hz, J = 6.5 Hz, 1H), 5.45−5.40 (d, J = 18 Hz, 1H), 2.18−2.12 (m, 4H), 1.43−1.31 (m, 4H), 1.26 (s, 12H), 0.89 ppm (m, 3H). **^{13}C-NMR** (100 MHz, $CDCl_3$): δ 155.0, 83.1, 30.6, 30.5, 25.0, 22.4, 14.0 ppm. **HRMS (ESI$^+$, *m*/*z*):** 211.1873 $[M + H]^+$ (calcd for $C_{12}H_{24}BO_2$, 211.1872).
Data consistent with the literature.[11]

(*E*)-4,4,5,5-tetramethyl-2-(octadec-1-en-1-yl)-1,3,2-dioxaborolane (3n): The general procedure was followed by using 1-octadecyne (**1n**). Purification by column chromatography on silica gel (DCM) yielded **3n** (93%) as a colorless oil. **^{1}H-NMR** (400 MHz, $CDCl_3$): δ 6.67−6.59 (dt, J = 18 Hz, J = 6.5 Hz, 1H), 5.45−5.40 (d, J = 18 Hz, 1H), 2.16−2.11 (m, 2H), 1.42−1.38 (m, 2H), 1.26−1.25 (m, 38H), 0.88 ppm (m, 3H). **^{13}C-NMR** (100 MHz, $CDCl_3$): δ 155.0, 83.1, 30.6, 30.5, 25.0, 22.4, 14.1 ppm. **HRMS (ESI$^+$, *m*/*z*):** 379.3745 $[M + H]^+$ (calcd for $C_{24}H_{48}BO_2$, 379.3748).



8. Copies of NMR spectra

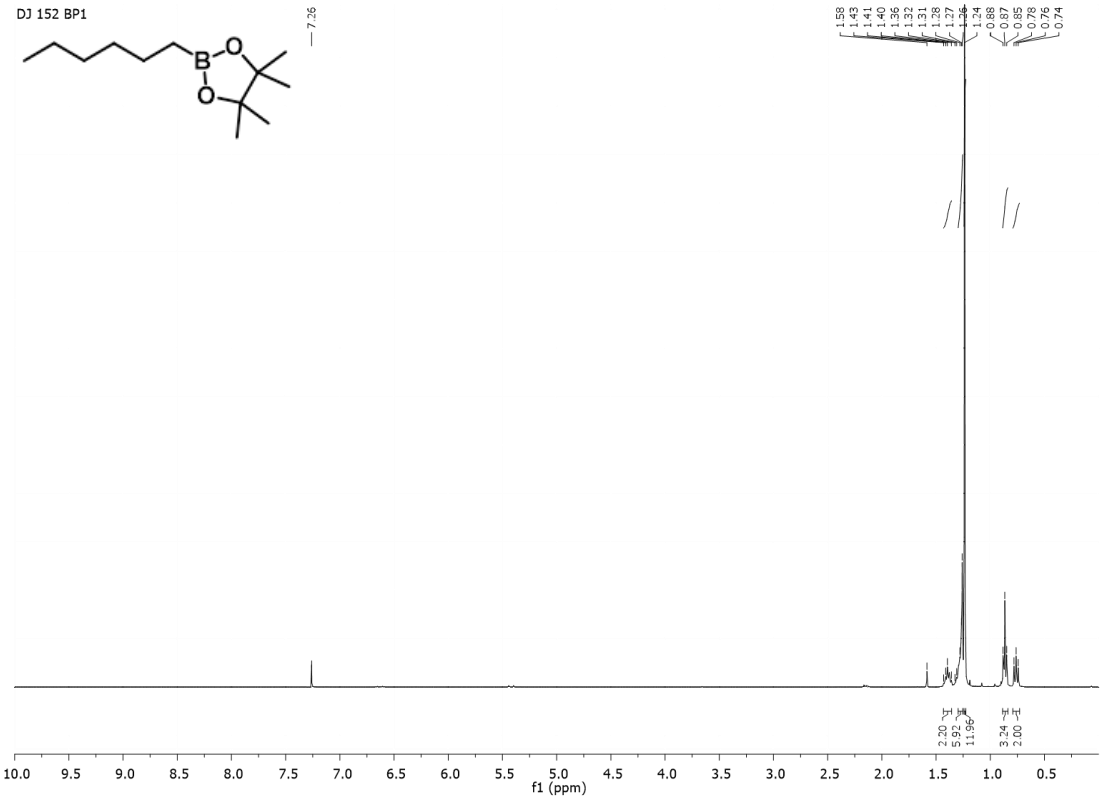


Figure S3. ^{1}H-NMR spectrum of compound **3a** at 400 MHz in $CDCl_3$.

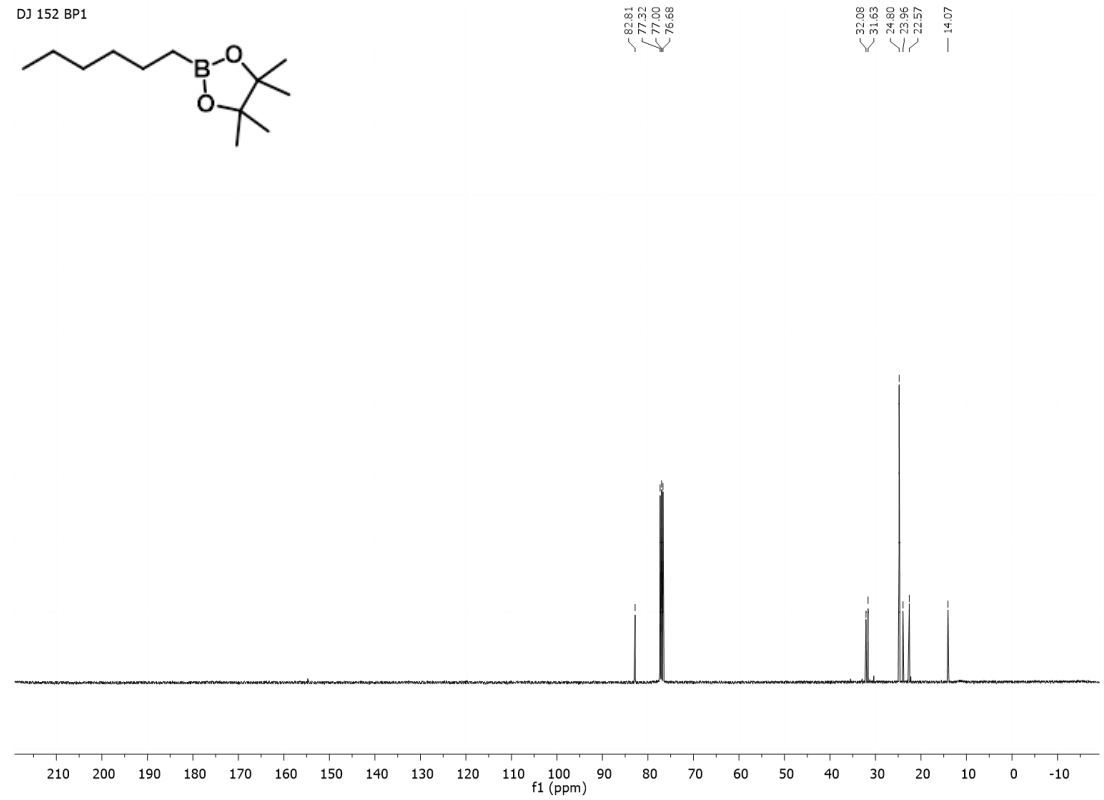


Figure S4. ^{13}C-NMR spectrum of compound **3a** at 100 MHz in $CDCl_3$.

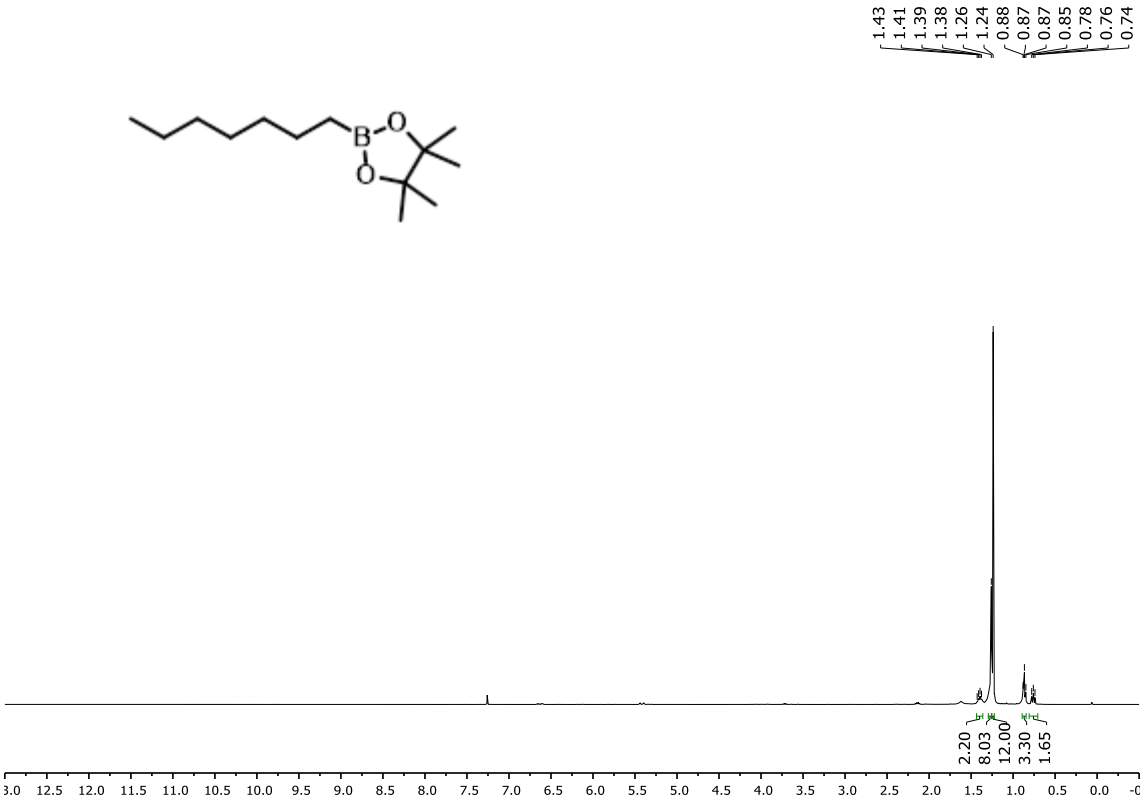


Figure S5. ^{1}H-NMR spectrum of compound **3b** at 400 MHz in $CDCl_3$.

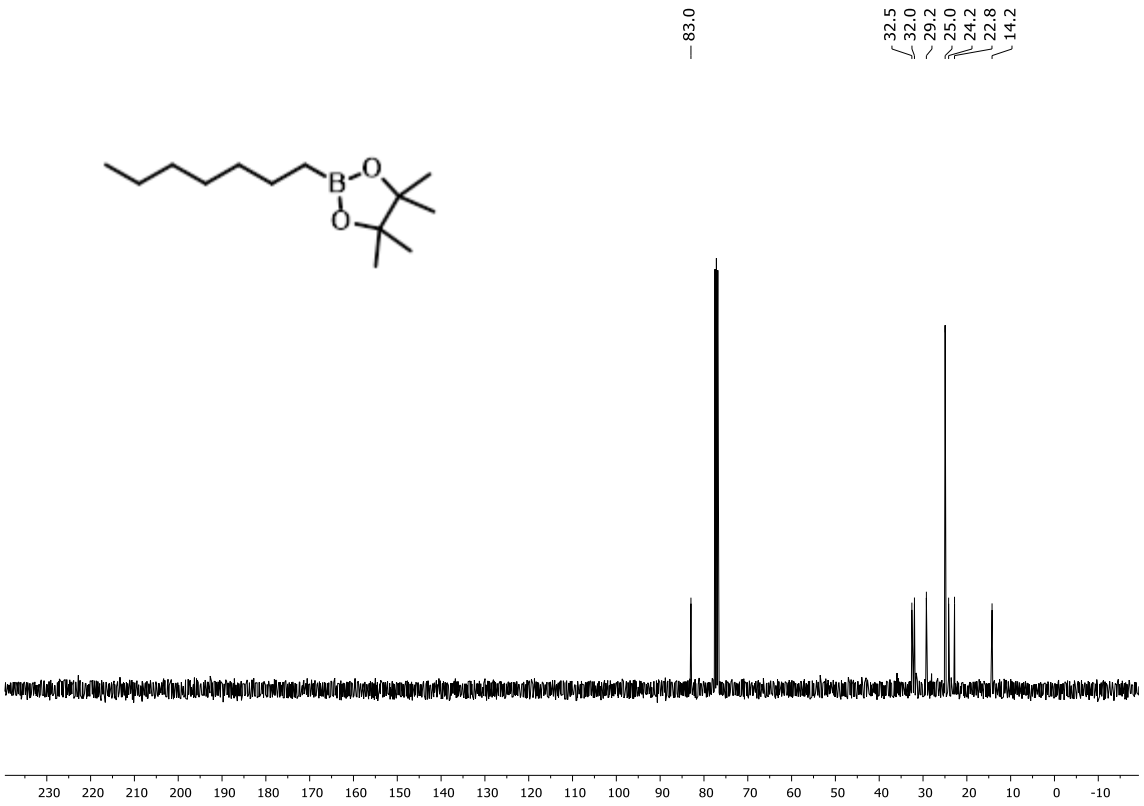


Figure S6. ^{13}C-NMR spectrum of compound **3b** at 100 MHz in $CDCl_3$.

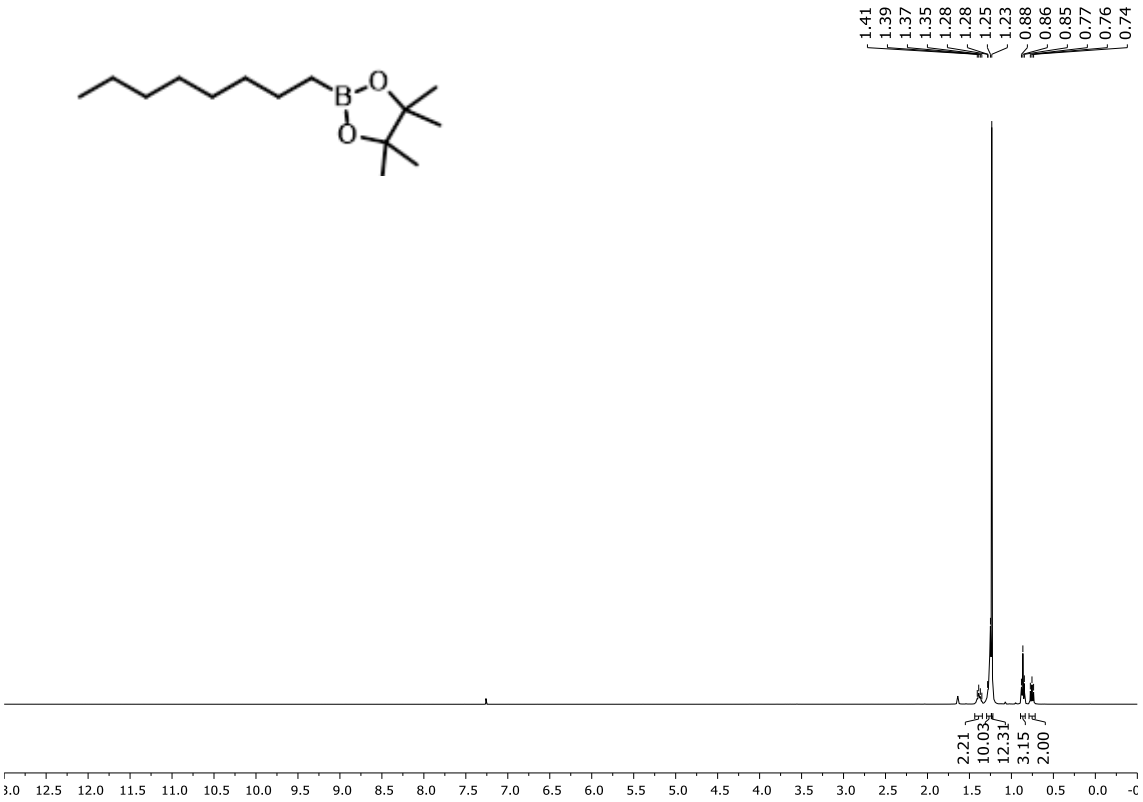


Figure S7. ^{1}H-NMR spectrum of compound **3c** at 400 MHz in $CDCl_3$.

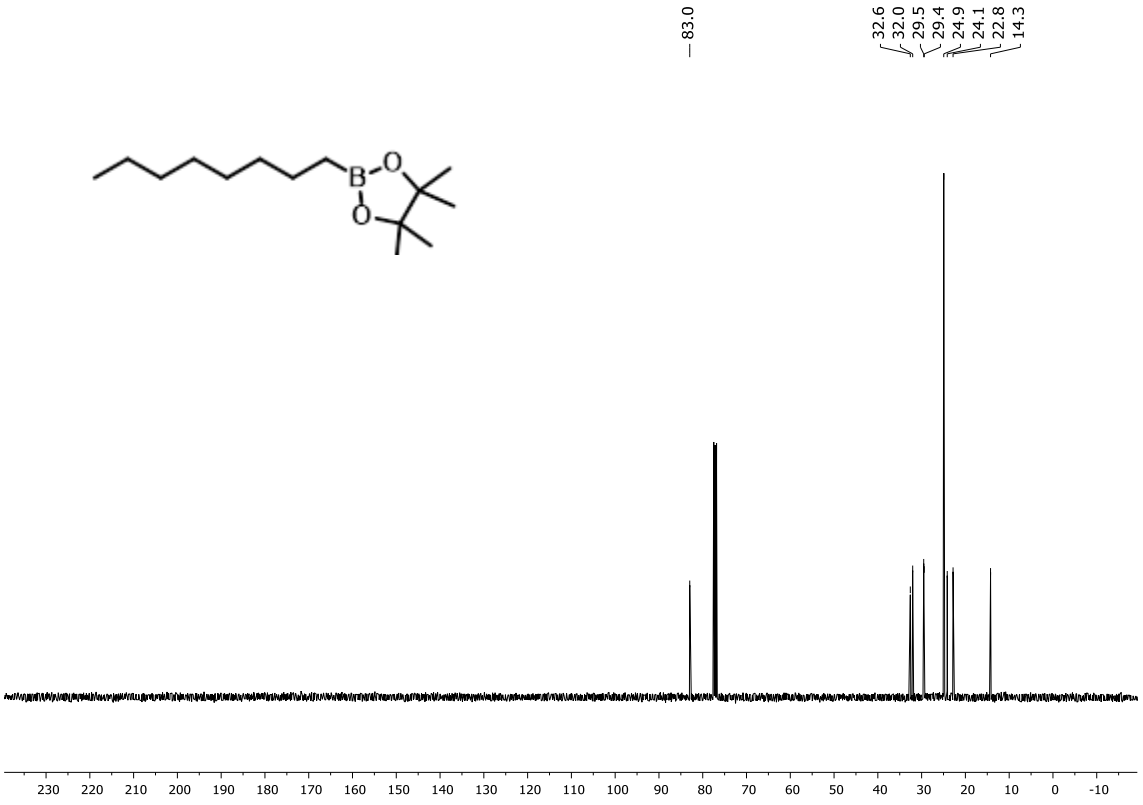


Figure S8. ^{13}C-NMR spectrum of compound **3c** at 100 MHz in $CDCl_3$.

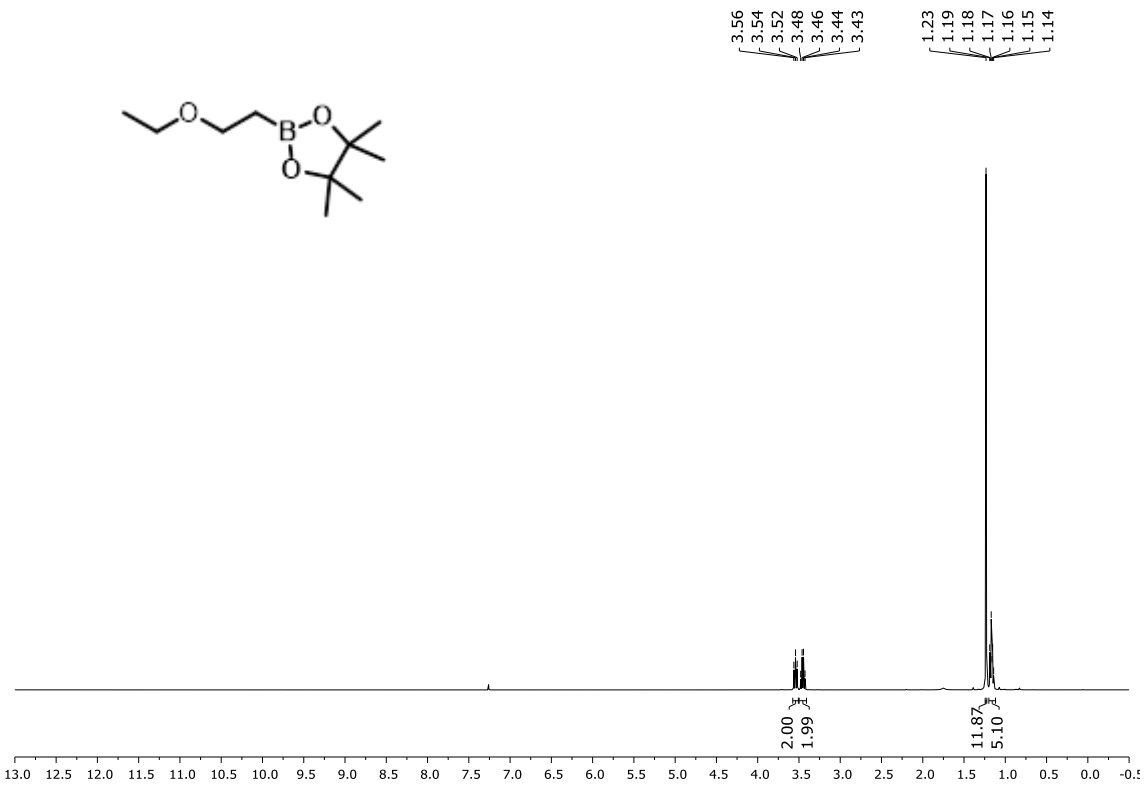


Figure S9. ^{1}H-NMR spectrum of compound **3d** at 400 MHz in $CDCl_3$.

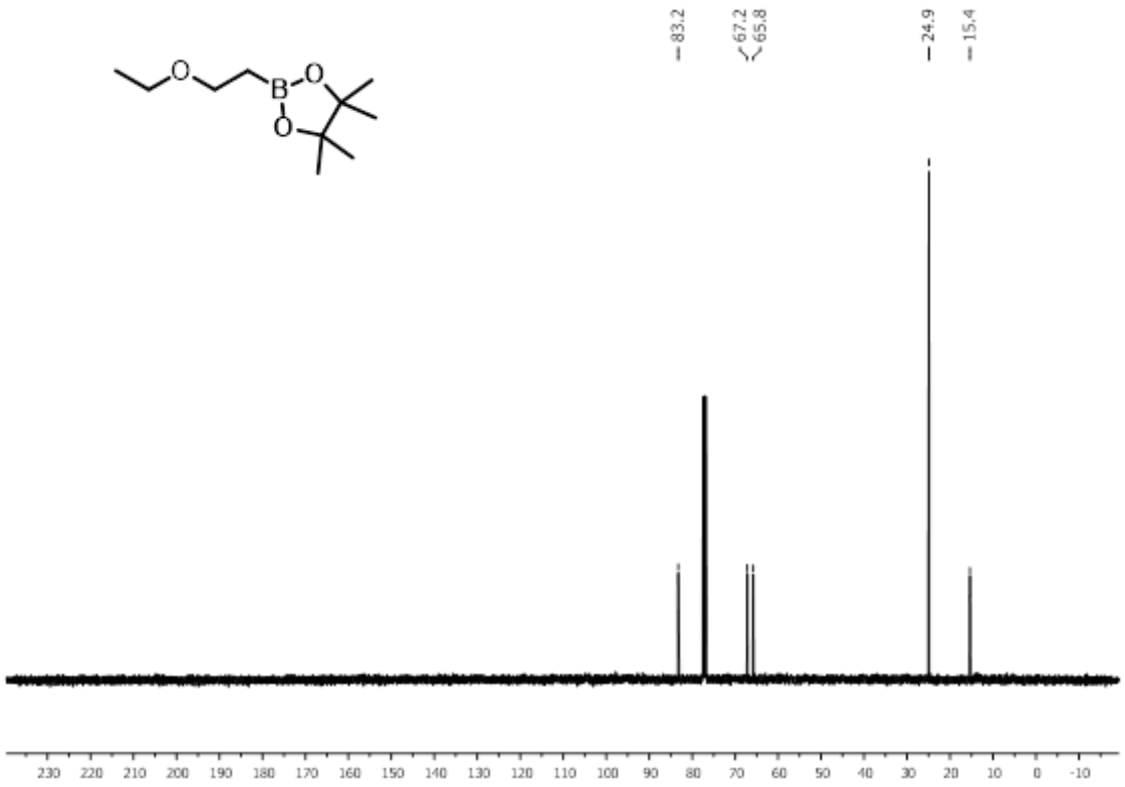


Figure S10. ^{13}C-NMR spectrum of compound **3d** at 100 MHz in $CDCl_3$.

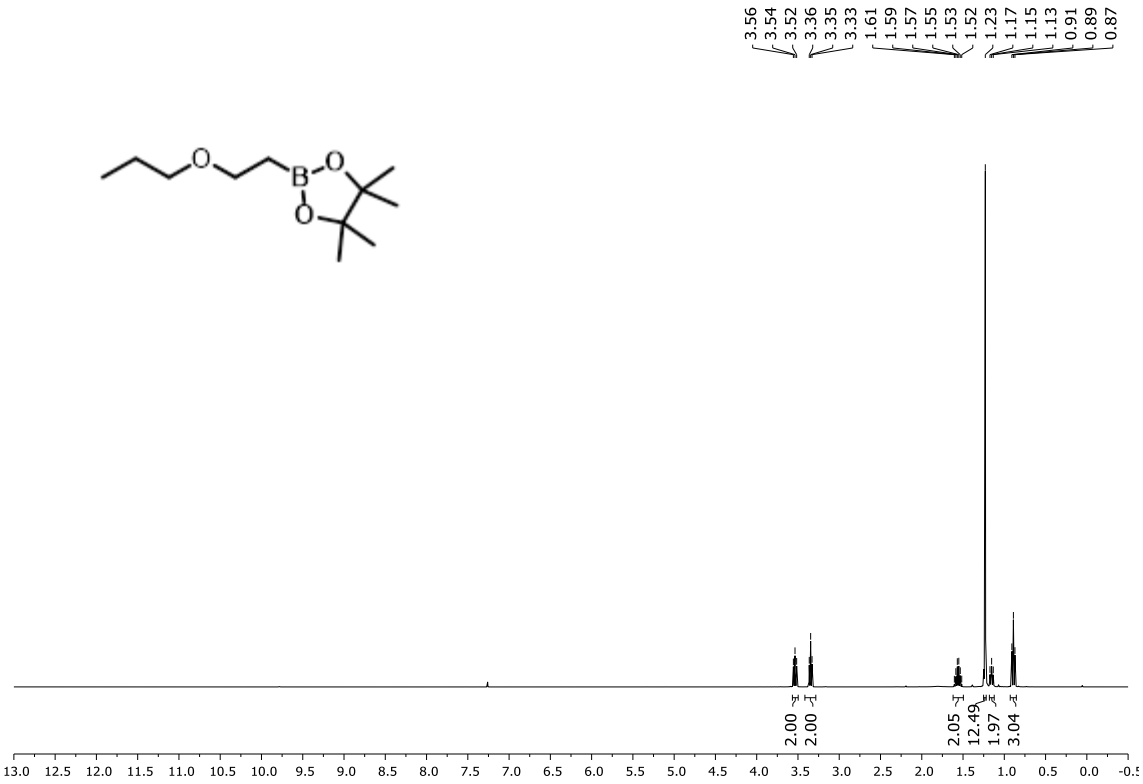


Figure S11. ^{1}H-NMR spectrum of compound **3e** at 400 MHz in $CDCl_3$.

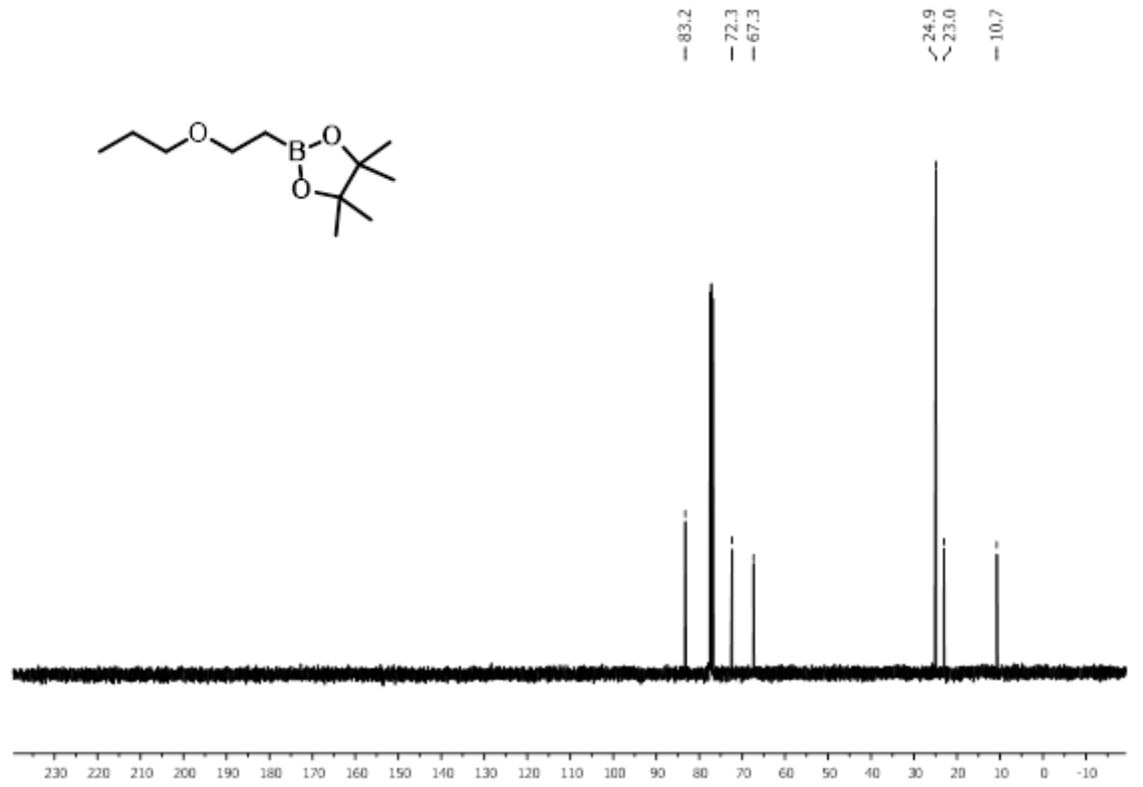


Figure S12. ^{13}C-NMR spectrum of compound **3e** at 100 MHz in $CDCl_3$.

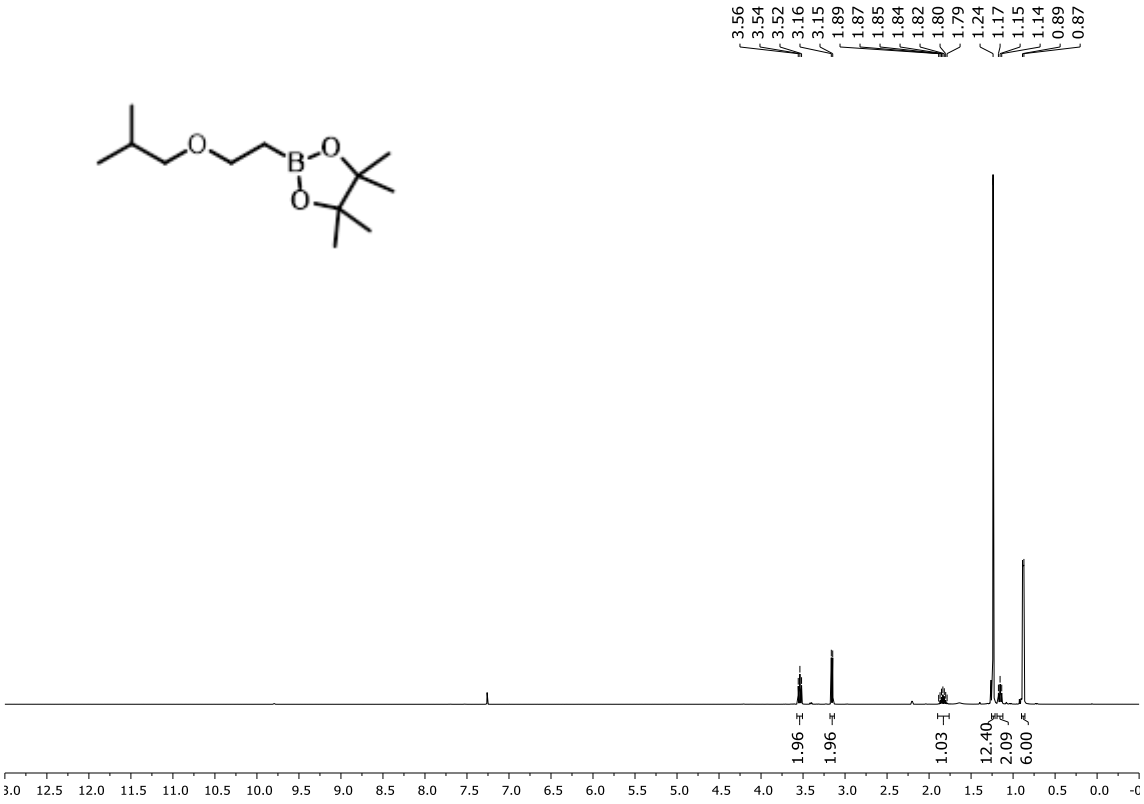


Figure S13. ^{1}H-NMR spectrum of compound **3f** at 400 MHz in $CDCl_3$.

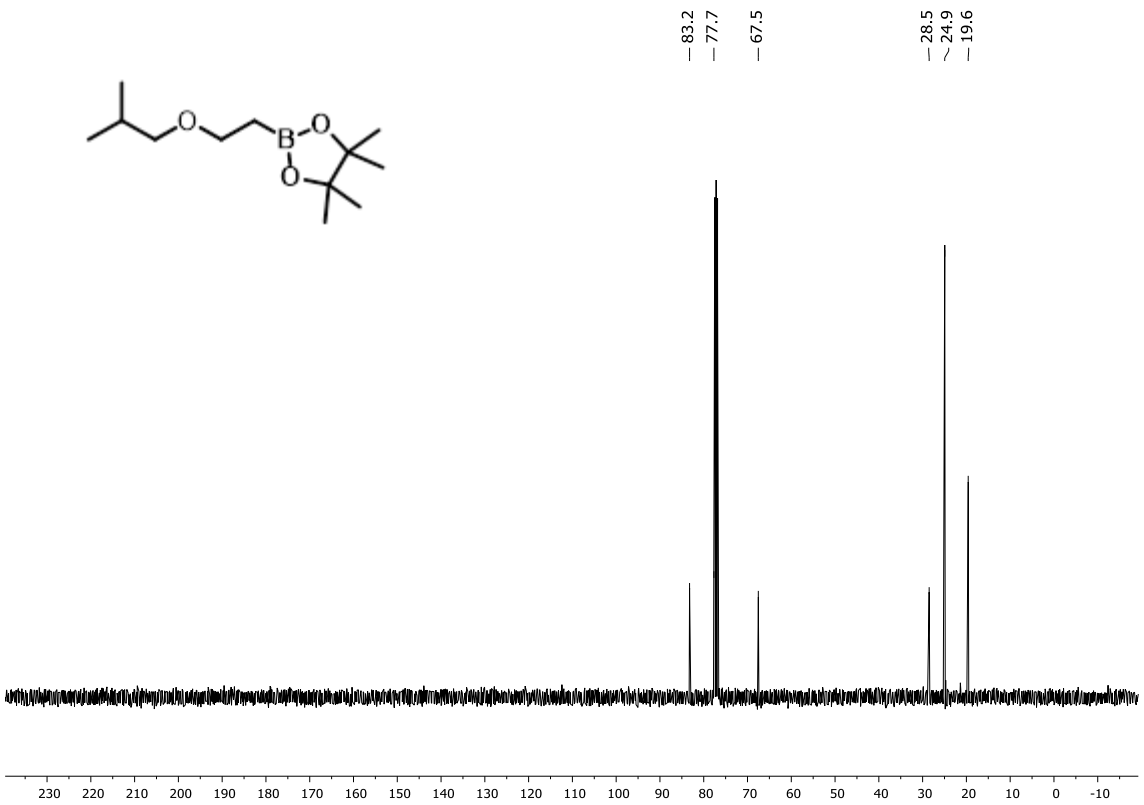


Figure S14. ^{13}C-NMR spectrum of compound **3f** at 100 MHz in $CDCl_3$.

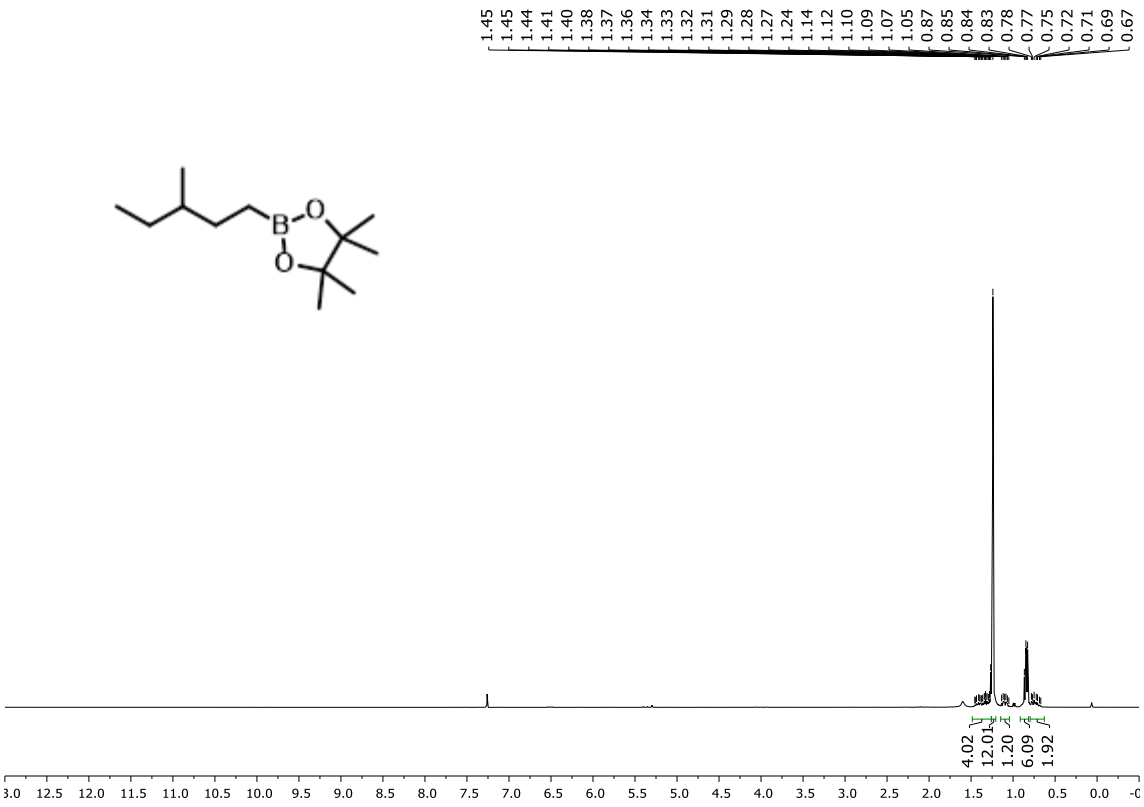


Figure S15. ^{1}H-NMR spectrum of compound **3g** at 400 MHz in $CDCl_3$.

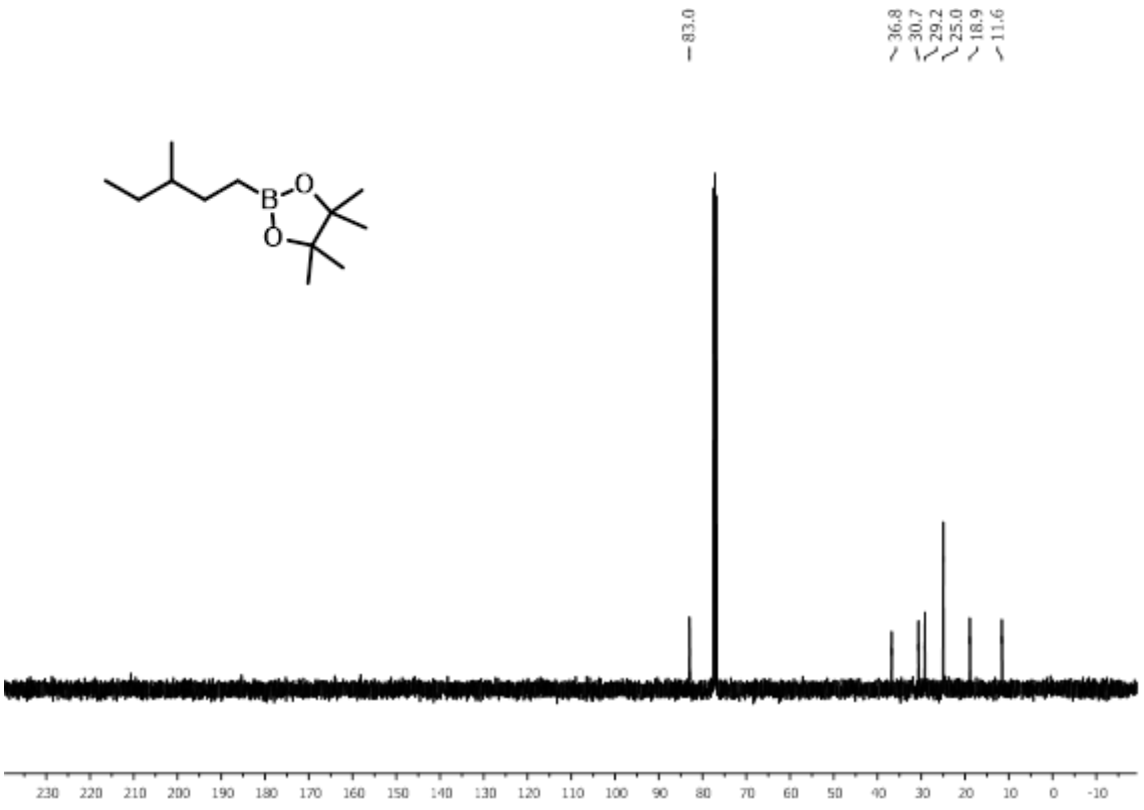


Figure S16. ^{13}C-NMR spectrum of compound **3g** at 100 MHz in $CDCl_3$.

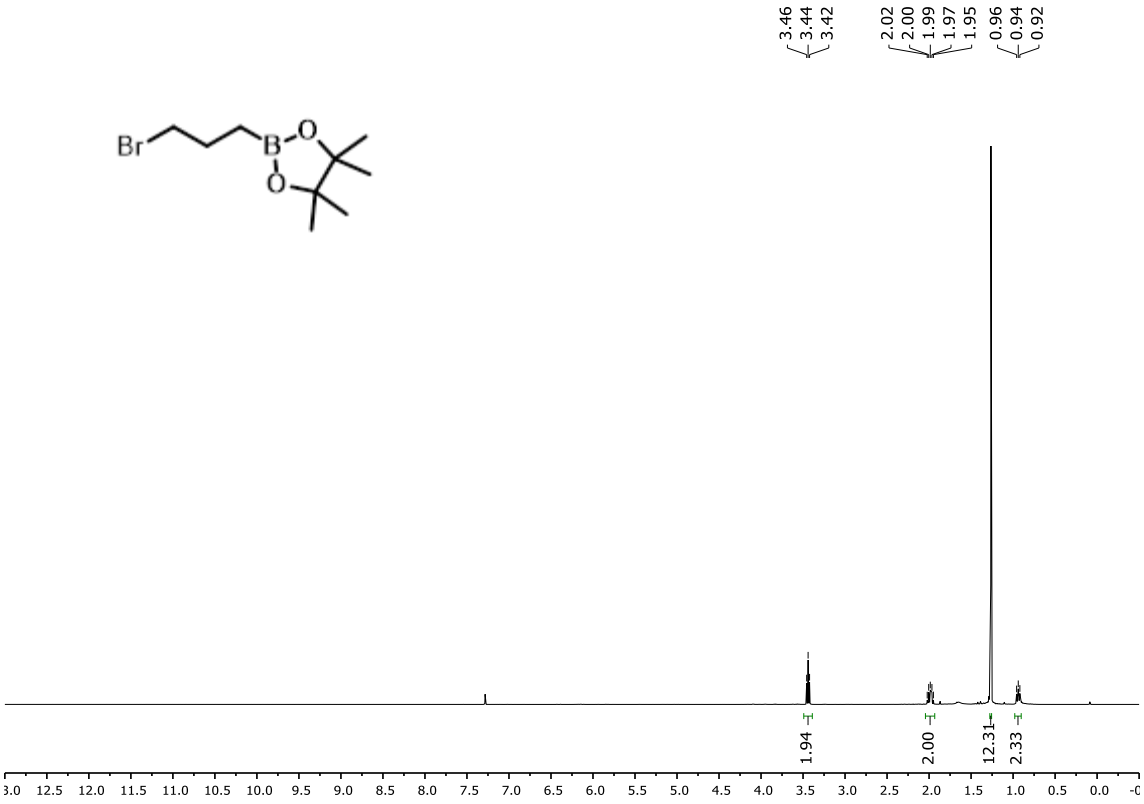


Figure S17. ^{1}H-NMR spectrum of compound **3h** at 400 MHz in $CDCl_3$.

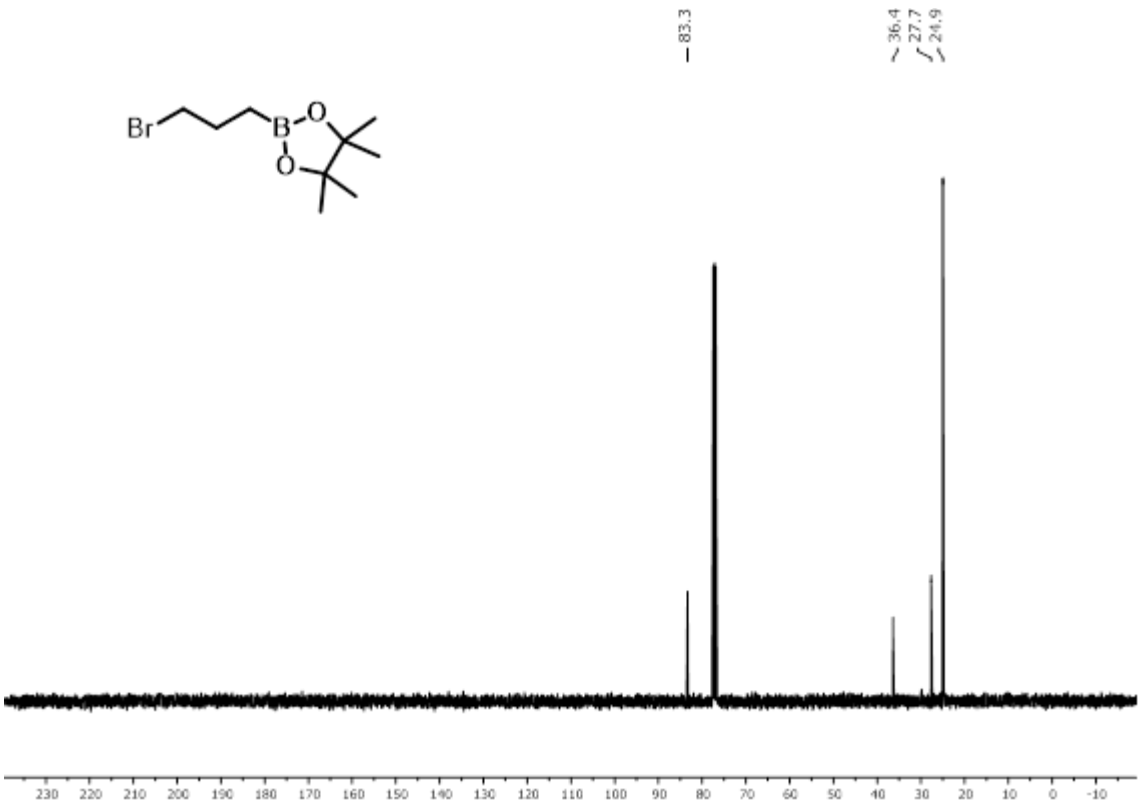


Figure S18. ^{13}C-NMR spectrum of compound **3h** at 100 MHz in $CDCl_3$.

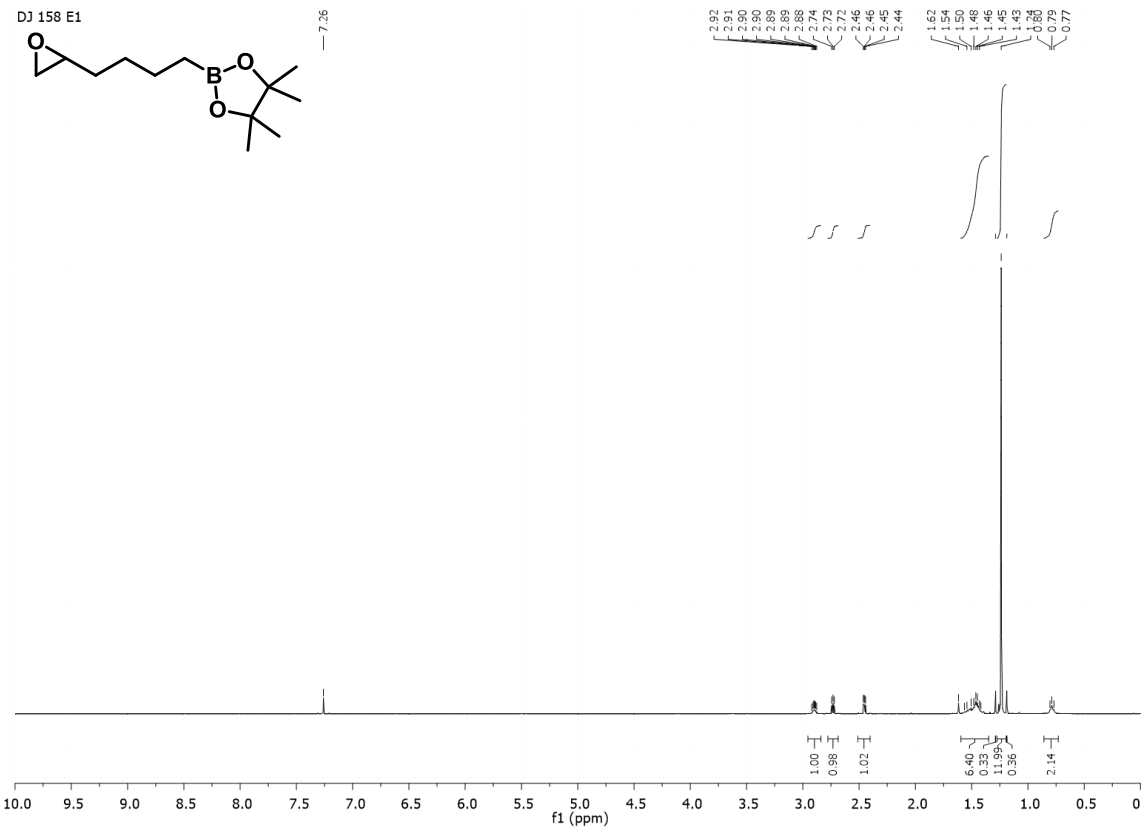


Figure S19. ^{1}H-NMR spectrum of compound **3i** at 400 MHz in $CDCl_3$.

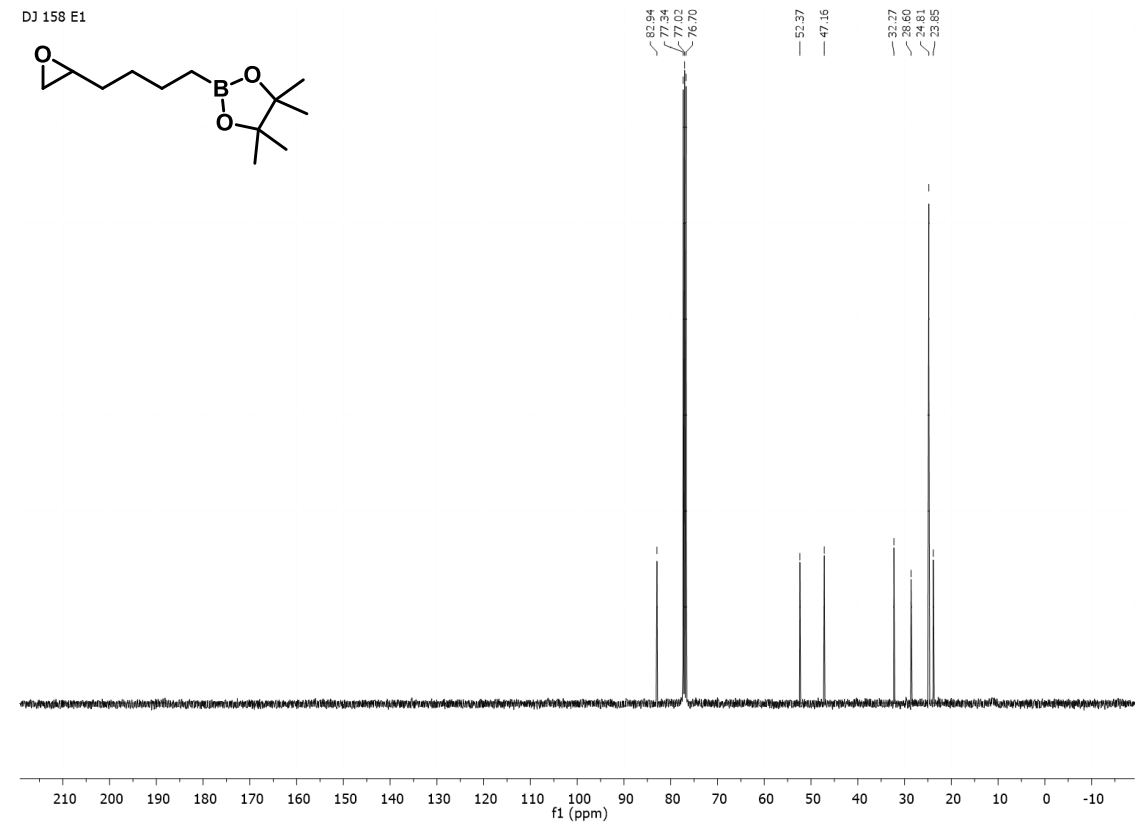


Figure S20. ^{13}C-NMR spectrum of compound **3i** at 100 MHz in $CDCl_3$.

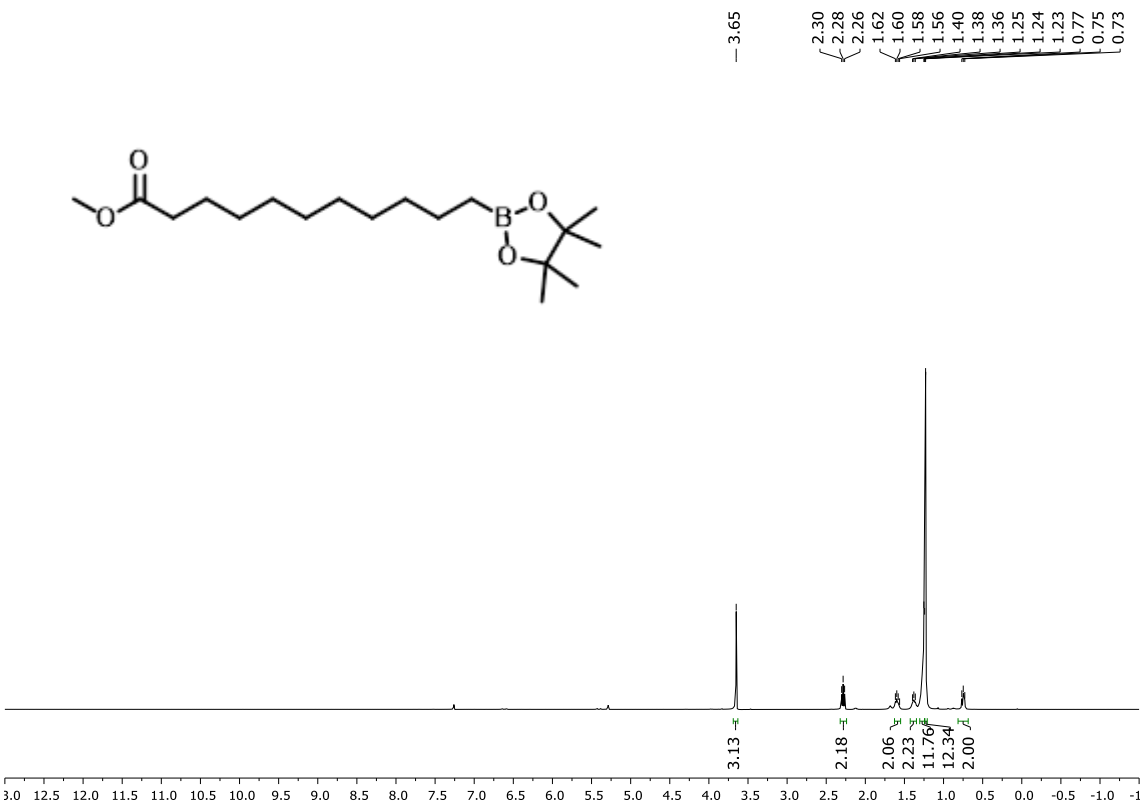


Figure S21. ^{1}H-NMR spectrum of compound **3j** at 400 MHz in $CDCl_3$.

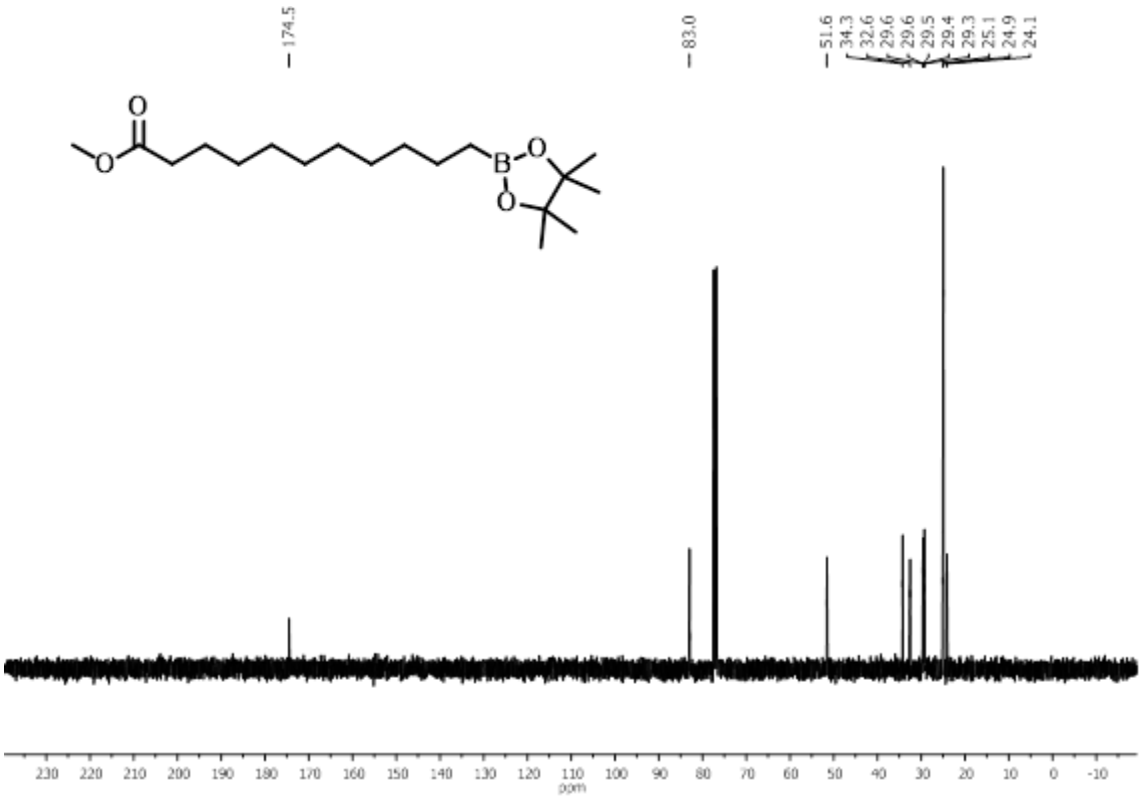


Figure S22. ^{13}C-NMR spectrum of compound **3j** at 100 MHz in $CDCl_3$.

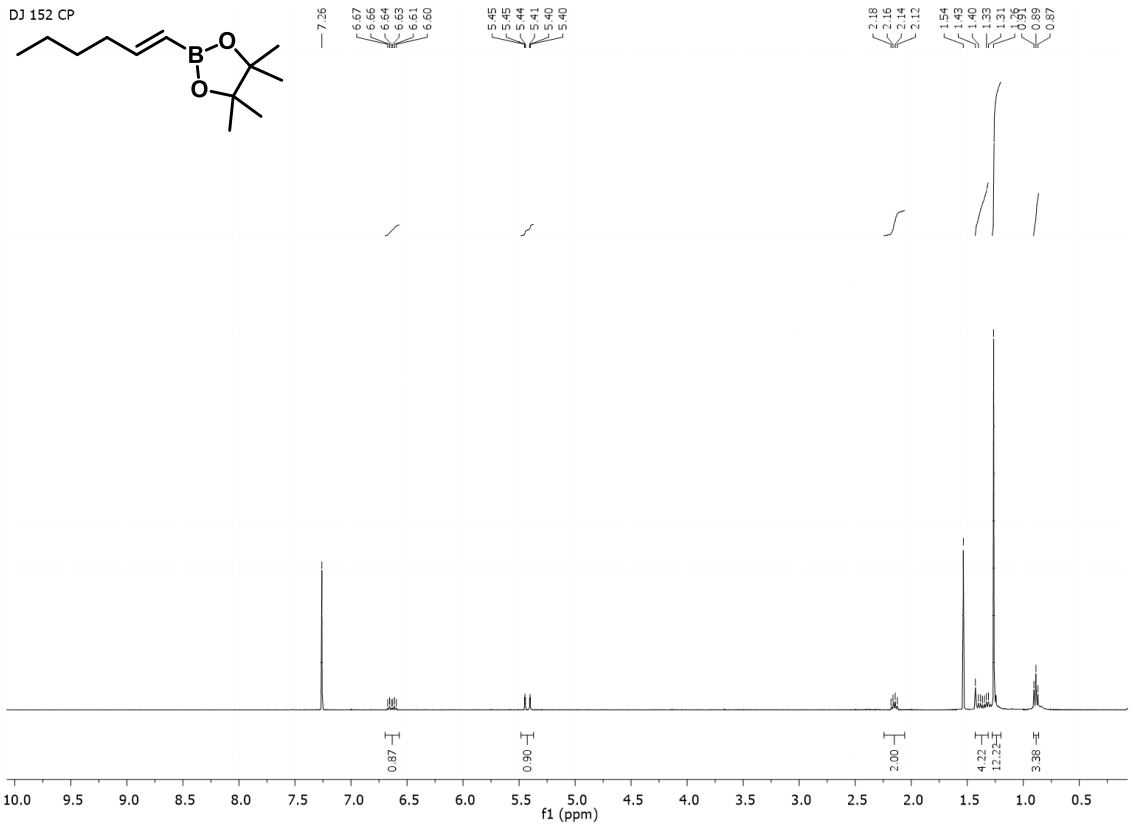


Figure S23. ^{1}H-NMR spectrum of compound **3m** at 400 MHz in $CDCl_3$.

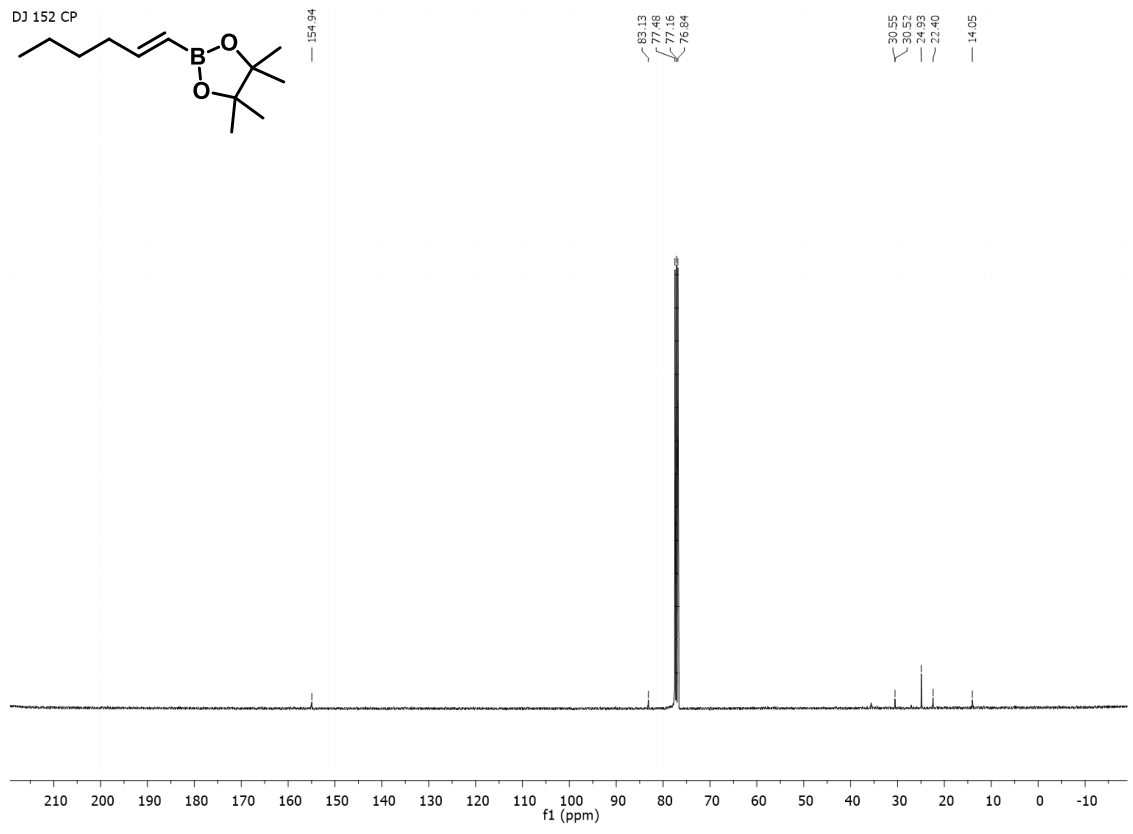


Figure S24. ^{13}C-NMR spectrum of compound **3m** at 100 MHz in $CDCl_3$.

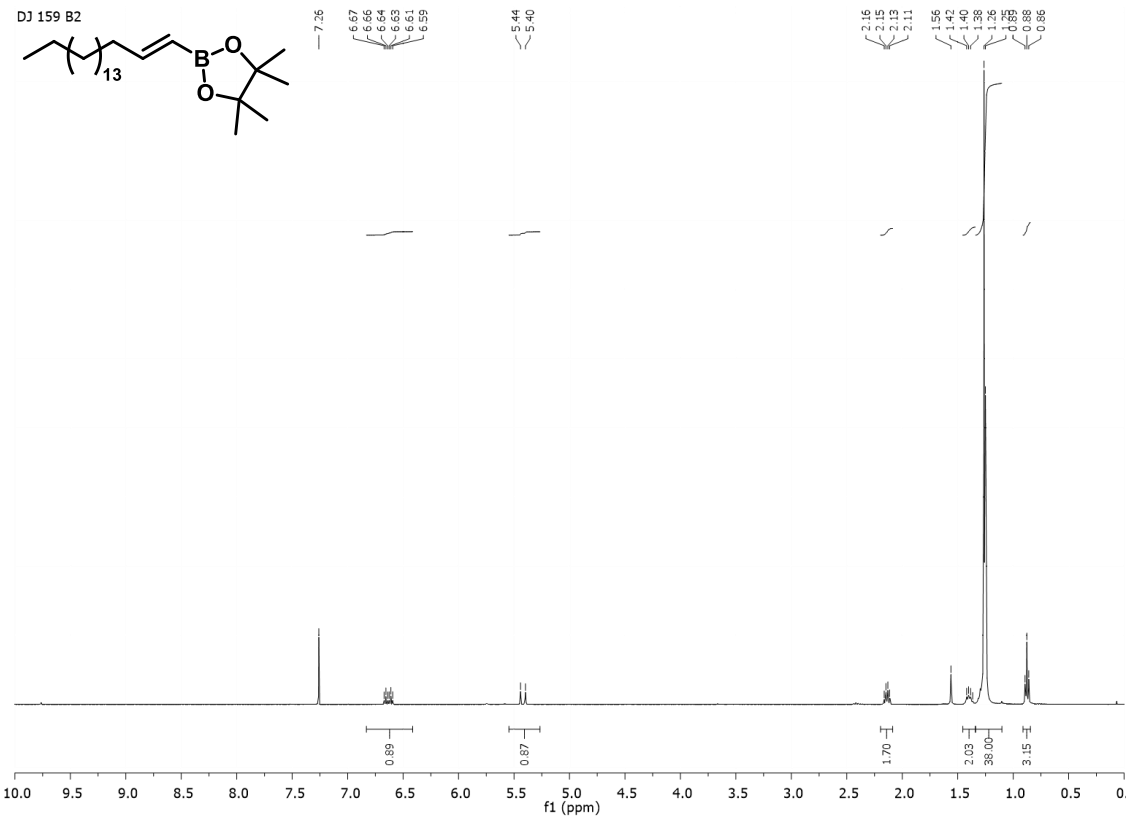


Figure S25. ^{1}H-NMR spectrum of compound **3n** at 400 MHz in $CDCl_3$.

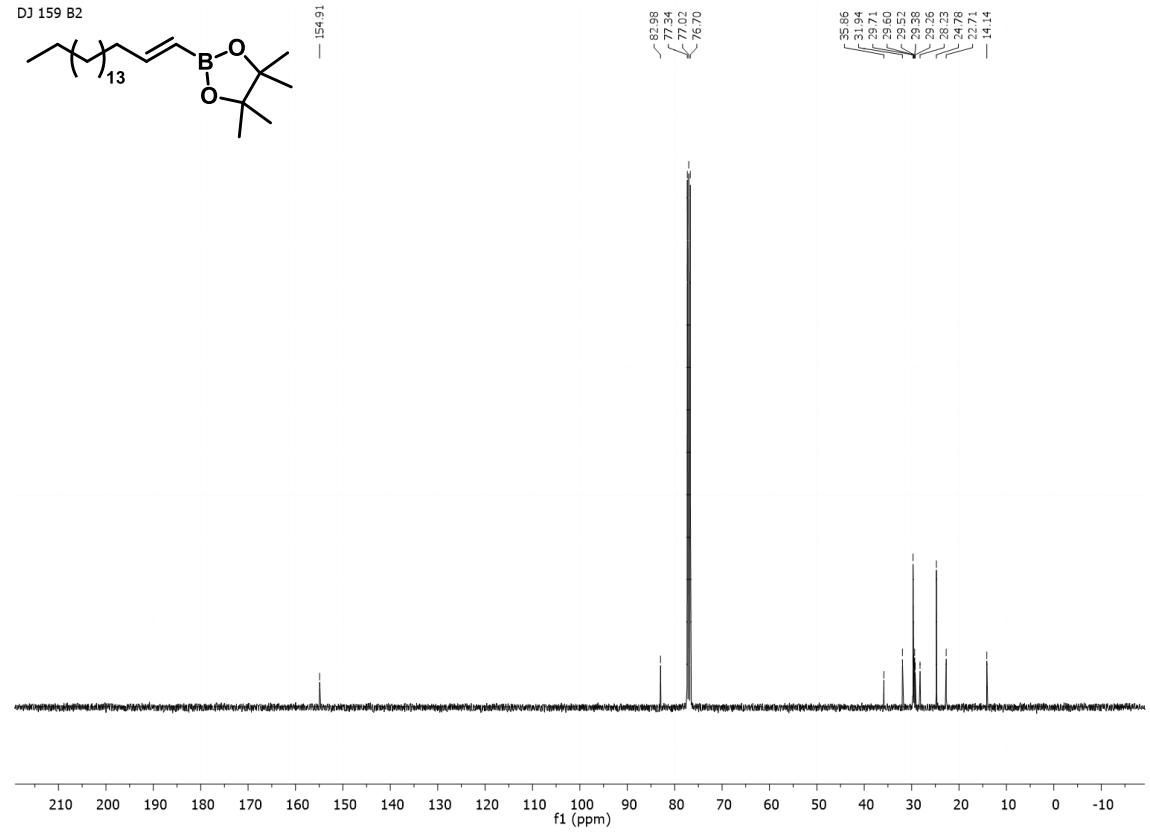


Figure S26. ^{13}C-NMR spectrum of compound **3n** at 100 MHz in $CDCl_3$.

9. Catalyst stability

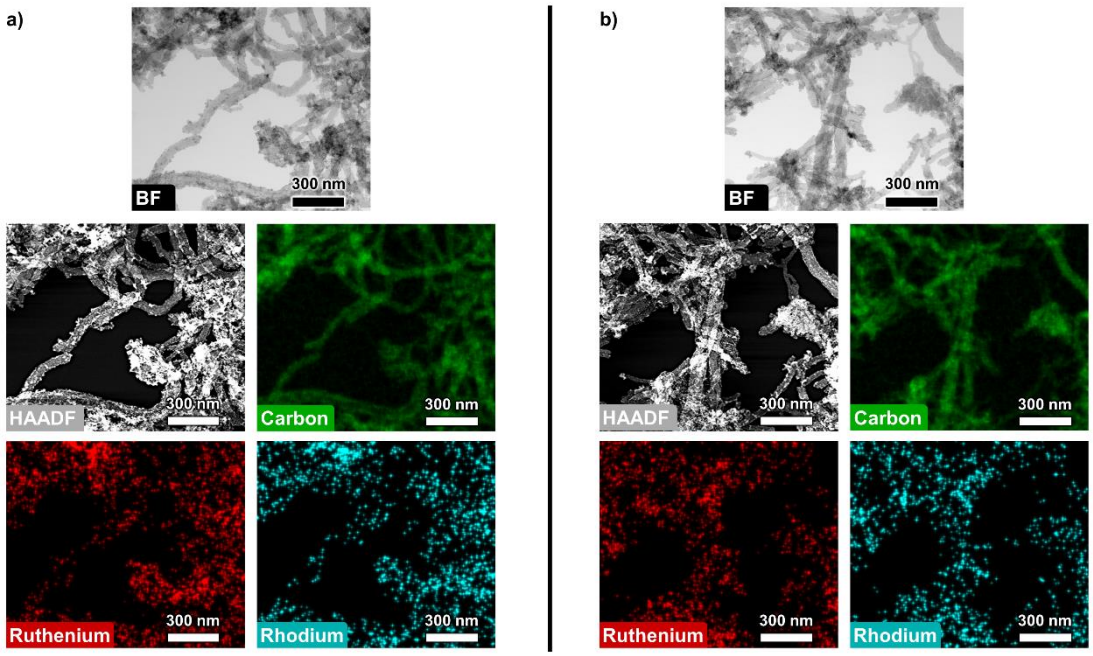


Figure S27. Images of the RhRuCNT catalyst a) before catalysis and b) after catalysis: Bright field (BF) TEM, high angle annular dark field (HAADF) and associated EDS mappings for carbon, ruthenium, and rhodium.

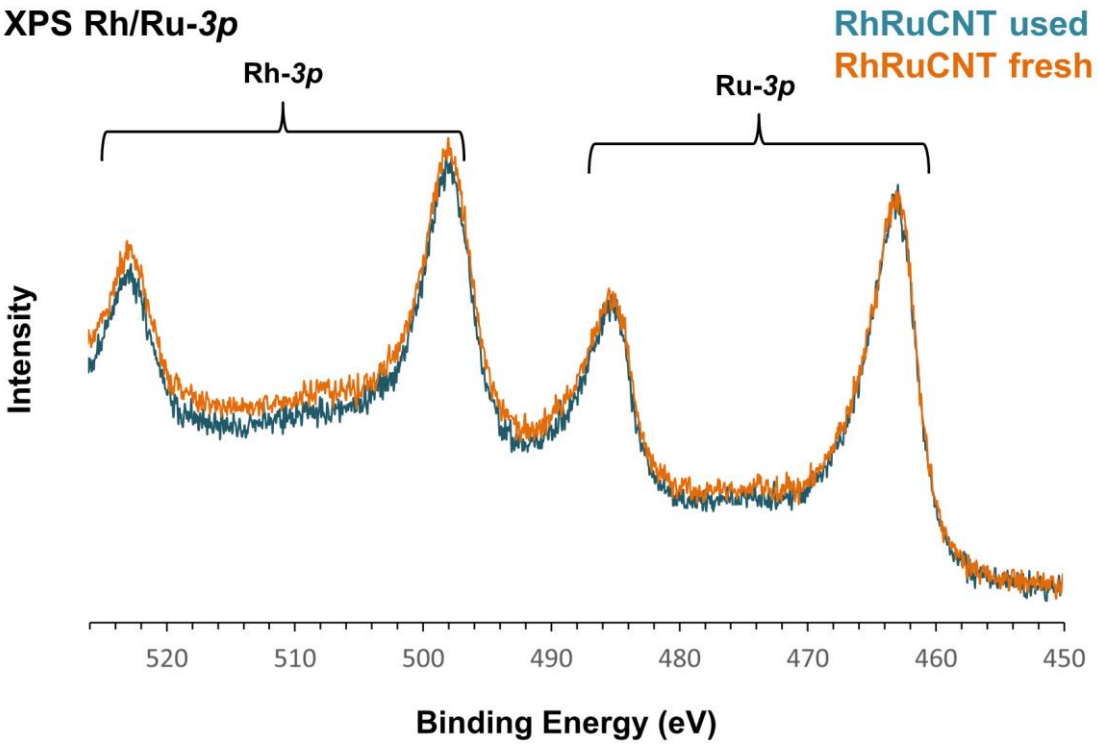


Figure S28. XPS spectra of the Rh-*3p* and Ru-*3p* regions for the RhRuCNT catalyst before (orange line) and after (blue line) catalysis.

10. Comparison with other catalytic systems

Table S2. Comparison of the performances of the RhRuCNT hybrid with other systems recently reported for the hydroboration reaction.

Catalyst	mol%	Additive(s)	Solvent	*T* (° C)	Recycling	Ref.
RhRuCNT	0.04	none	none	25	6 cycles (stable)	This work
Cu-TiC	0.28	none	toluene	80	8 cycles (stable)	Wang *et al.*[12]
Cu-O(I)-CeO_2	0.5	none	EtOH	70	5 cycles (stable)	Li *et al.*[13]
Cu_5Ni/Al_2O_3	6	none	EtOH	25	3 cycles (−10%)	Furukawa *et al.*[14]
PCN-222 (Cu)	1.5	3-methylpyridine (20 mol%)	EtOH/H_2O	80	5 cycles (stable)	Zhang *et al.*[15]
{[Co_5(4,4'-H_2dbpt)$_2$ Br_{10}]·8(MEK)}$_n$	0.5	$NaBHEt_3$ (5 mol%)	MTBE	45	5 cycles (stable)	Huang *et al.*[16]
Cu/N-CNT	13	KOMe (1.2 equiv) MeOH (2 equiv)	DMF	25	5 cycles (stable)	Bose *et al.*[17]
{[$Cu^{II}Cu^{I}_4I_4$(-PMCA)$_2$ (Py)]NH(CH_3)$_{23}$DMF}	0.3	NaOMe (10 mol%)	EtOH	25	5 cycles (stable)	Zhao *et al.*[18]

11. References

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