SUPPORTING INFORMATION

Evaluation of the Role of Graphene-based Cu(I) Catalysts in Borylation Reactions

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1. General methods

NMR spectra were acquired using $CDCl_3$ as solvent, running at 300 and 75 MHz for ^1H and ^{13}C, respectively. Chemical shifts (δ) are reported in ppm relative to residual solvent signals ($CDCl_3$, 7.26 ppm for ^1H NMR, and 77.0 ppm for ^{13}C NMR). In all ^1H NMR spectra, multiplicity is indicated as follows: bs (broad singlet), s (singlet), d (doublet), t (triplet), q (quartet) or m (multiplet). Coupling constant values (in Hertz) and number of protons for each signal are also indicated.

For thin layer chromatography (TLC) was performed using pre-coated aluminium backed plates (Merck Kieselgel 60 F254) and visualized by ultraviolet irradiation (254 nm) by treatment with a solution of $KMnO_4$ (1.5 g), K_2CO_3 (10 g), and 10% NaOH (1.25 mL) in H_2O (200 mL) or a solution of phosphomolybdic acid (12 g), in EtOH (250 mL) followed by heating. Flash column chromatography (FCC) was performed using Merck pore 60 Å, 40-63 μm silica gel and compressed air.

FTIR analysis was carried out in a Bruker IFS66v spectrometer (KBr, 99%). ICP-MS analysis was performed in a NexION 300XX Perkin-Elmer. TXRF analysis were carried out in a Bruker TXRF S2 PicoFox spectrometer. SEM images were acquired in a Hitachi Tabletop Microscope TM-1000-151. For XPS analysis aXPS Spectrometer Kratos AXIS Supra apparatus was used. The copper amount added to each experiment was calculated based on the %wt Cu determined through TXRF for each material. A 200 KV JEOL 2100 transmission electron microscope was employed for the TEM analysis. The samples were prepared by adding a drop of each material dispersion in ethanol on a lacey carbon-coated copper grid 200 Mesh and then let to dry.

Cyclohexane and EtOAc were supplied by *Carlo Erba* and were used without previous purification. THF, DMF and DMA were purchased dry and with no stabilizers in *Acros Organics*. All reagents were acquired from commercial sources and were used without further purification. For all described synthesized products, spectroscopic data are consistent with the references indicated next to the name of each compound. For separation of materials, we used a mini centrifuge LBX MC7000 working at 7000 rpm.

Graphenit-OX, graphene oxide (GO, synthesized using a modified Hummers' method),[1] reduced graphene oxide (rGO, prepared from GO and reduced using hydrazine in basic media), and Carbon Black were supplied by NanoInnova Technologies SL (https://www.nanoinnova.com/). For the synthesis of the microwave irradiated materials, we used an *Orbegozo MI-2014 conventional microwave at approximately 560 W* (measured by heating 1 L of D.I. water at full power and measuring the temperature difference before and after the radiation).

2. Preparation of materials F-I.

Materials **F-I** have been previously prepared by us.[2] To facilitate the reading, conditions are repeated herein.

Preparation of material F

Material F was prepared following the standard method described for graphenit-Cu(I) (procedure A) but using GO (500 mg) as starting material, $CuCl_2$ (109 mg, 0.8 mmol) and $NaBH_4$ (650 mg, 17.5 mmol) as reducing agent.

Preparation of material G

Material G was prepared following the standard method described for graphenit-Cu(I) (procedure A) but using rGO (500 mg) as starting material, $CuCl_2$ (109 mg, 0.8 mmol) and $NaBH_4$ (650 mg, 17.5 mmol) as reducing agent.

Preparation of materials H

Material H was prepared following the standard method described for graphenit-Cu(I) (procedure A) but using carbon black (500 mg) as starting material, $CuCl_2$ (109 mg, 0.8 mmol) and $NaBH_4$ (650 mg, 17.5 mmol) as reducing agent.

Preparation of material I

Material I was prepared following the standard method described for graphenit-Cu(I) (procedure A), but using $CuCl_2$ (109 mg, 0.8 mmol) and $NaBH_4$ (65 mg, 1.75 mmol) as reducing agent.

3. Characterization of materials A-E_{MW}

3.1. XRD Analysis

XRD patterns of GNPs and materials A, B_{MW}, C_{MW}, D_{MW} and E_{MW} are shown in Figure S1. Characteristic peaks for Cu_2O (36.6º and 42.6º), Cu (43.5º, 50.4º) and GNPs (26.2°, 43.8°, 54.6°, 77.6°) are observed in different proportions in the materials studied. CuO peaks at 35.7º and 39º are absent in all materials.

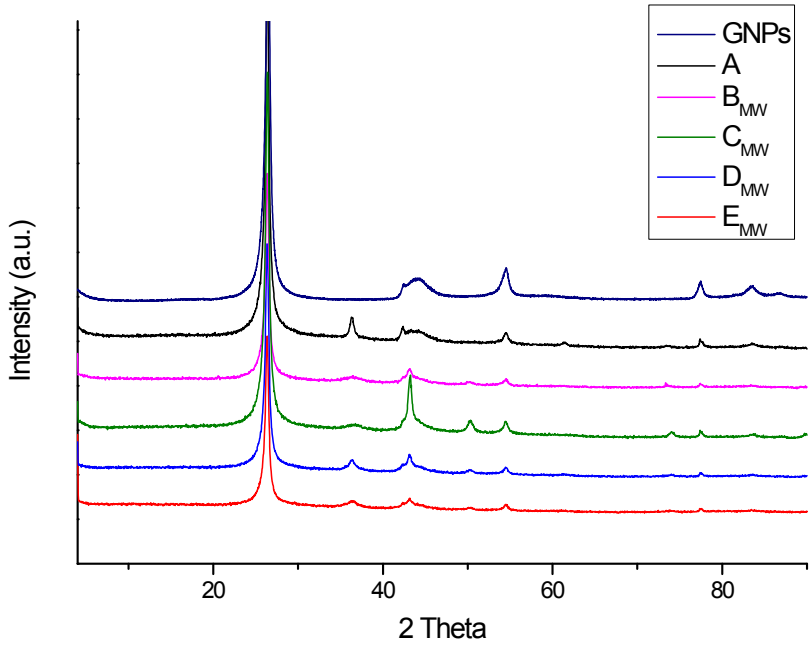


Figure S1. *XRD pattern comparison of the prepared materials.*

The ratio between Cu(I) and Cu(0) for each material has been approximated by fitting the XRD peaks at 36.6 and 50.4 degrees (Figure S2). Table S1 shows for each material the areas under the Cu(I) peak at 36.6 and the Cu (0) peak at 50º, and also the ratio between both areas. Material A only contains Cu_2O as no peak is observed at 50º. For microwave materials E is the one with higher ratio of Cu(I)/Cu(0) and material C is the one with lower ratio.

Table S1. *Calculated Cu(I) and Cu(0) areas form XRD patterns of the materials.*

Material	Cu(I) area	Cu(0) area	Area ratio (I)/(0)
Material **A**	976.58434	--	--
Material **B_{MW}**	943.84039	178.95673	5.27
Material **C_{MW}**	891.03331	750.07325	1.19
Material **D_{MW}**	798.19892	230.8028	3.46
Material **E_{MW}**	806.86873	122.75404	6.57

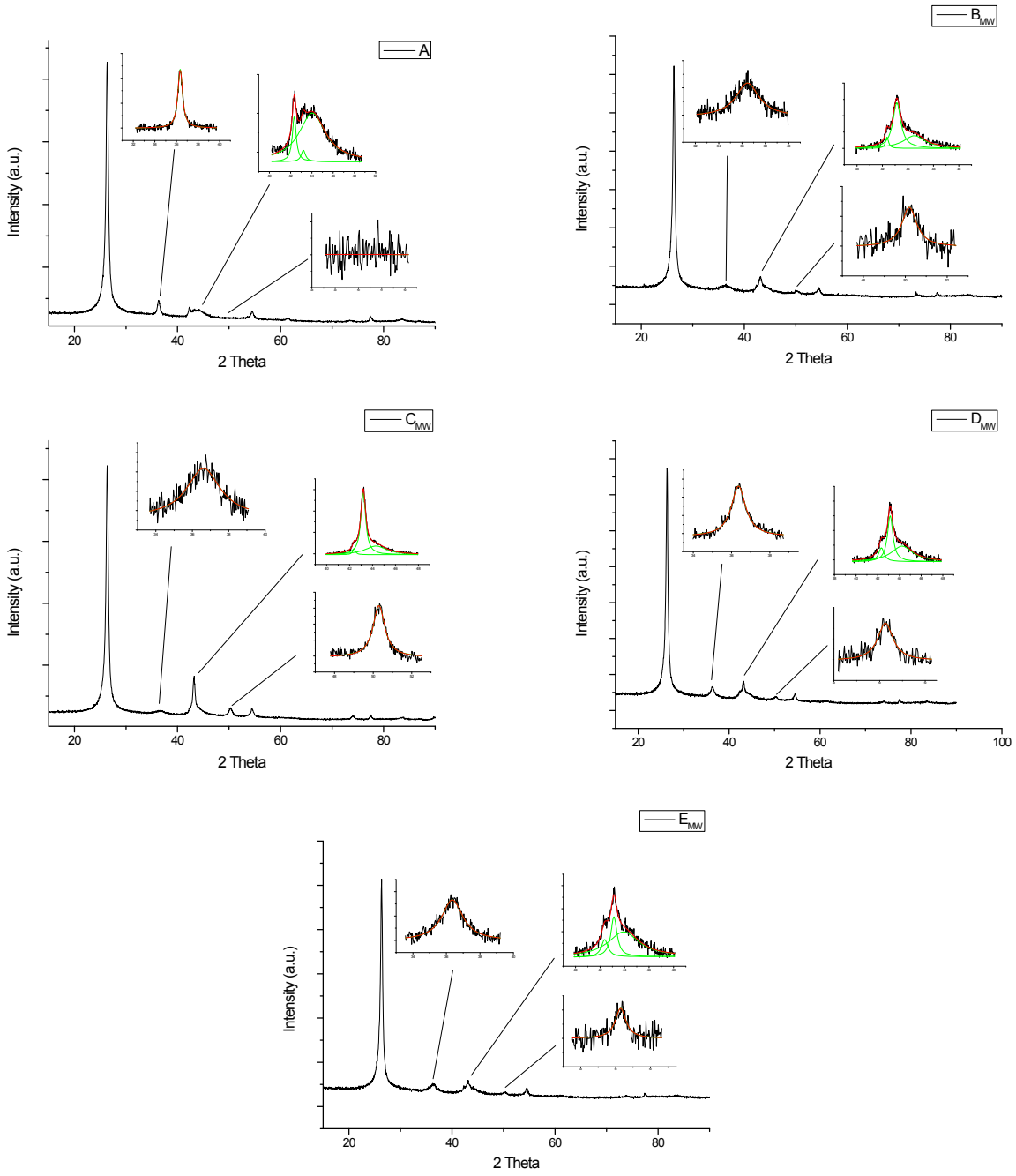


Figure S2. *Zoomed peak areas of the XRD patterns.*

Comparison between initial and used materials are shown in figures S3 (A and Ar) and S4 (E and Er). In both cases the oxidation state of Cu does not change after using the catalysts, but the amount of Cu seems to be lower in used materials and especially for material A (Table S2).

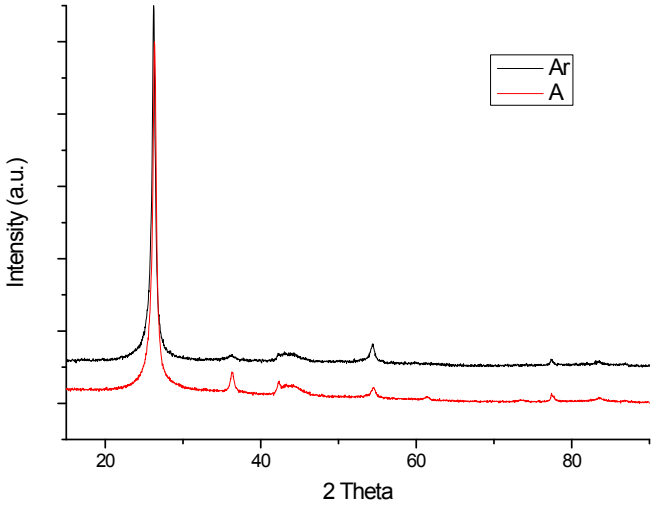


***Figure S3**. XRD pattern of the initial and recycled material A.*

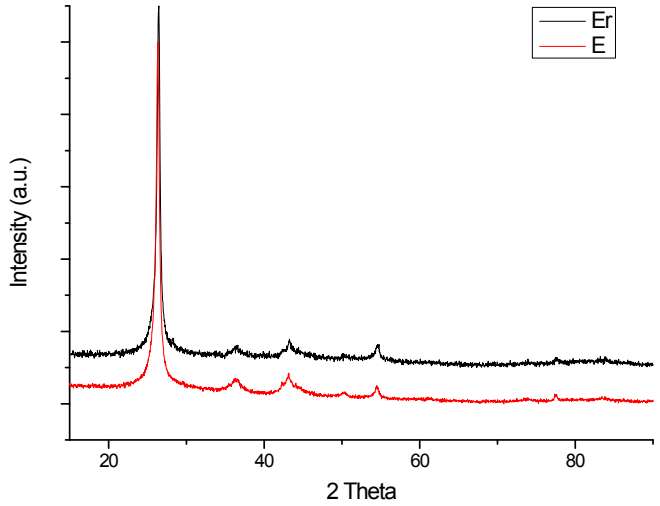


***Figure S4**. XRD pattern of the initial and recycled material E.*

***Table S2.** Calculated Cu(I) and Cu(0) areas form XRD patterns of the materials.*

Material	Cu(I) area 36.4°	Cu(0) area 50°	Area ratio (I)/(0)
Material **A**	976.58	0	--
Material **Ar**	236.34	0	--
Material $\mathbf{E_{MW}}$	806.87	122.75	6.57
Material $\mathbf{E_{MW}r}$	233.43	37.83	6.17

3.2. XPS Analysis

Figure S5 shows the XPS survey spectra of GNPs and materials **A**, **Ar**, **E_{MW}**, **E_{MW}r**.

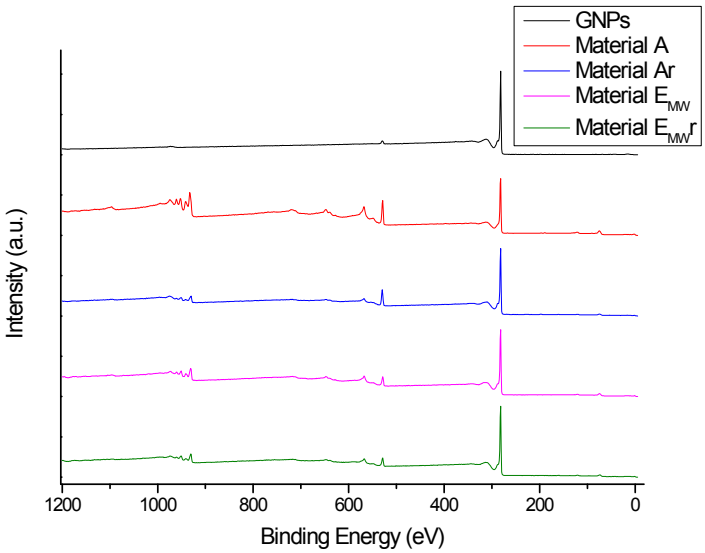


***Figure S5**. XPS spectra of the support and prepared materials.*

Table S3 shows the atomic % for C, O and Cu obtained from the survey spectra of the different materials.

***Table S3.** Atomic percentage of C, O and Cu in the materials.*

Sample	C 1s %	O 1s %	Cu 2p %
GNPs	98.2	1.8	--
Material **A**	81.0	12.8	6.3
Material **Ar**	90.8	8.0	6.3
Material **E_{MW}**	92.9	4.9	2.2
Material **E_{MW}r**	93.9	4.6	1.5

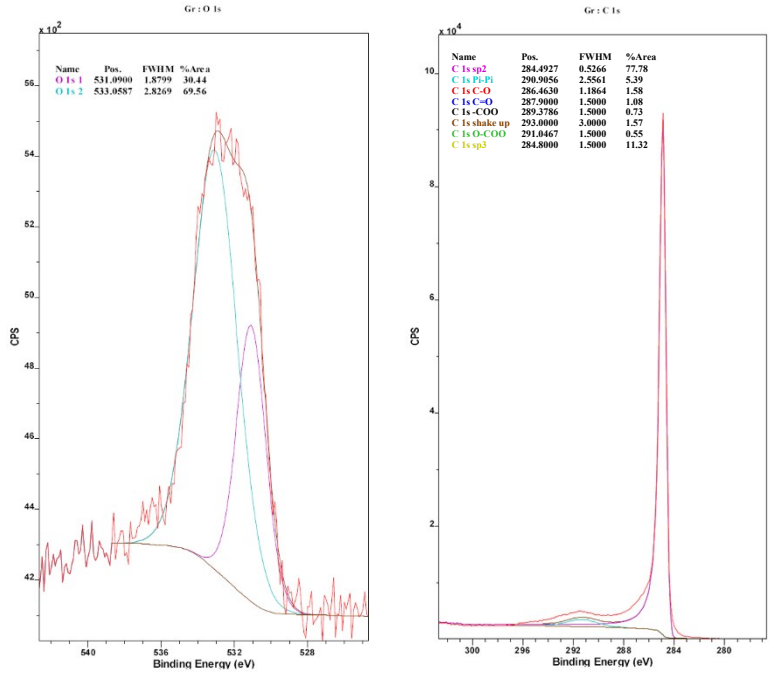


Figure S6. *De-convoluted high resolution XPS C1s and O1s region spectra of GNPs.*

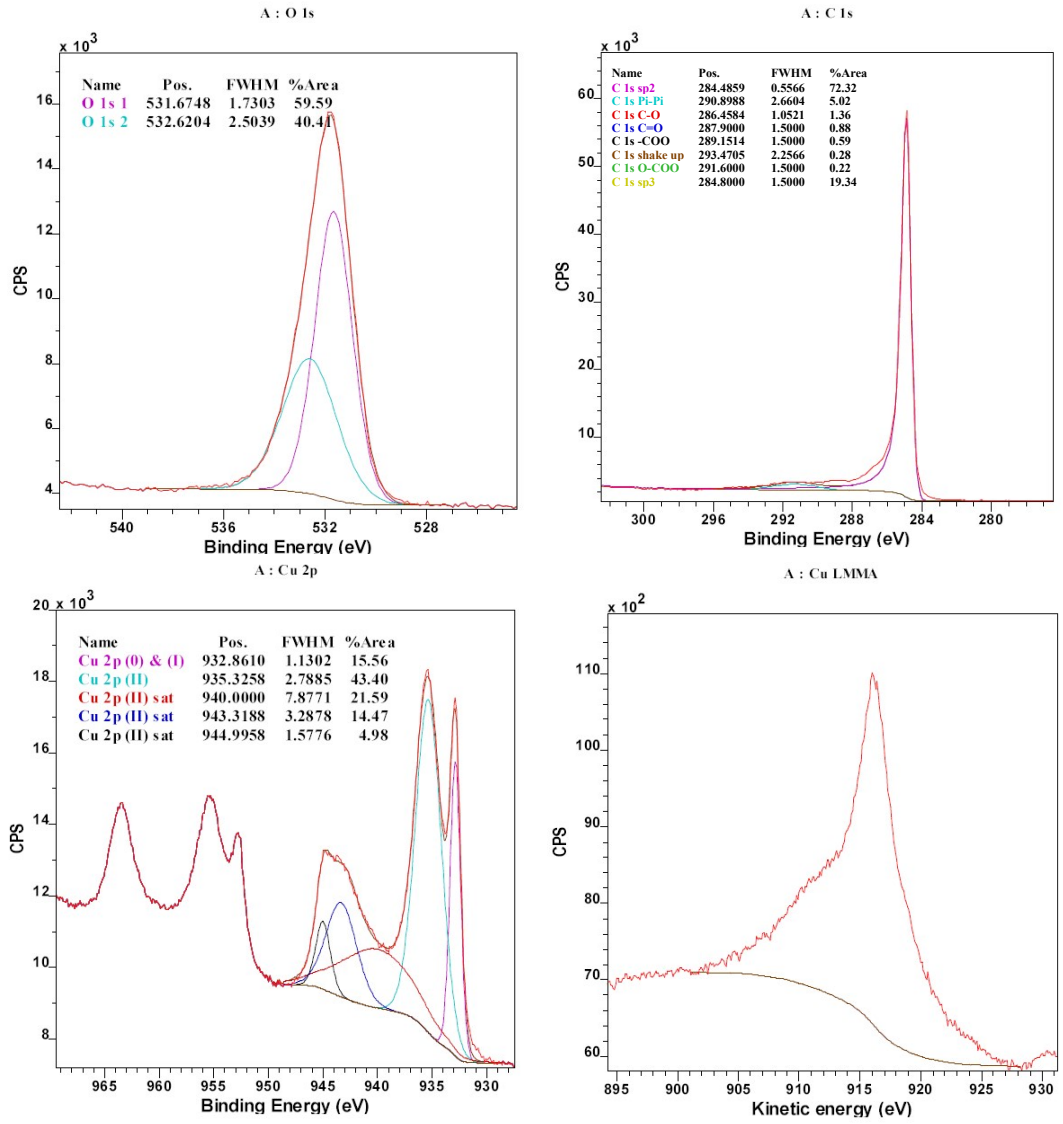


Figure S7. *De-convoluted high resolution XPS C1s, O1s, Cu2p region spectra and Cu Auger LMM kinetic energy spectra of Material A.*

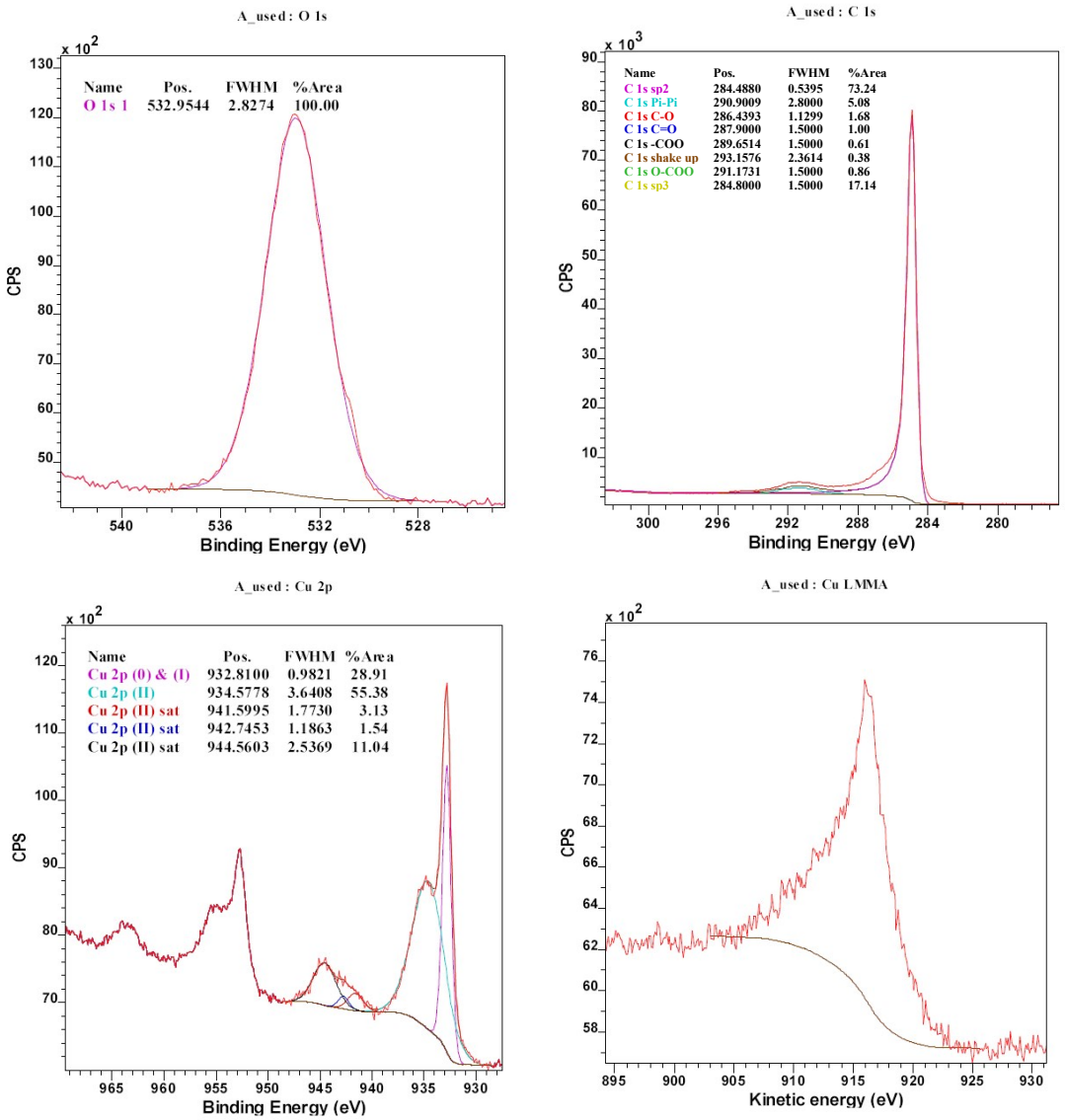


Figure S8. *De-convoluted high resolution XPS C1s, O1s, Cu2p region spectra and Cu Auger LMM kinetic energy spectra of Material Ar.*

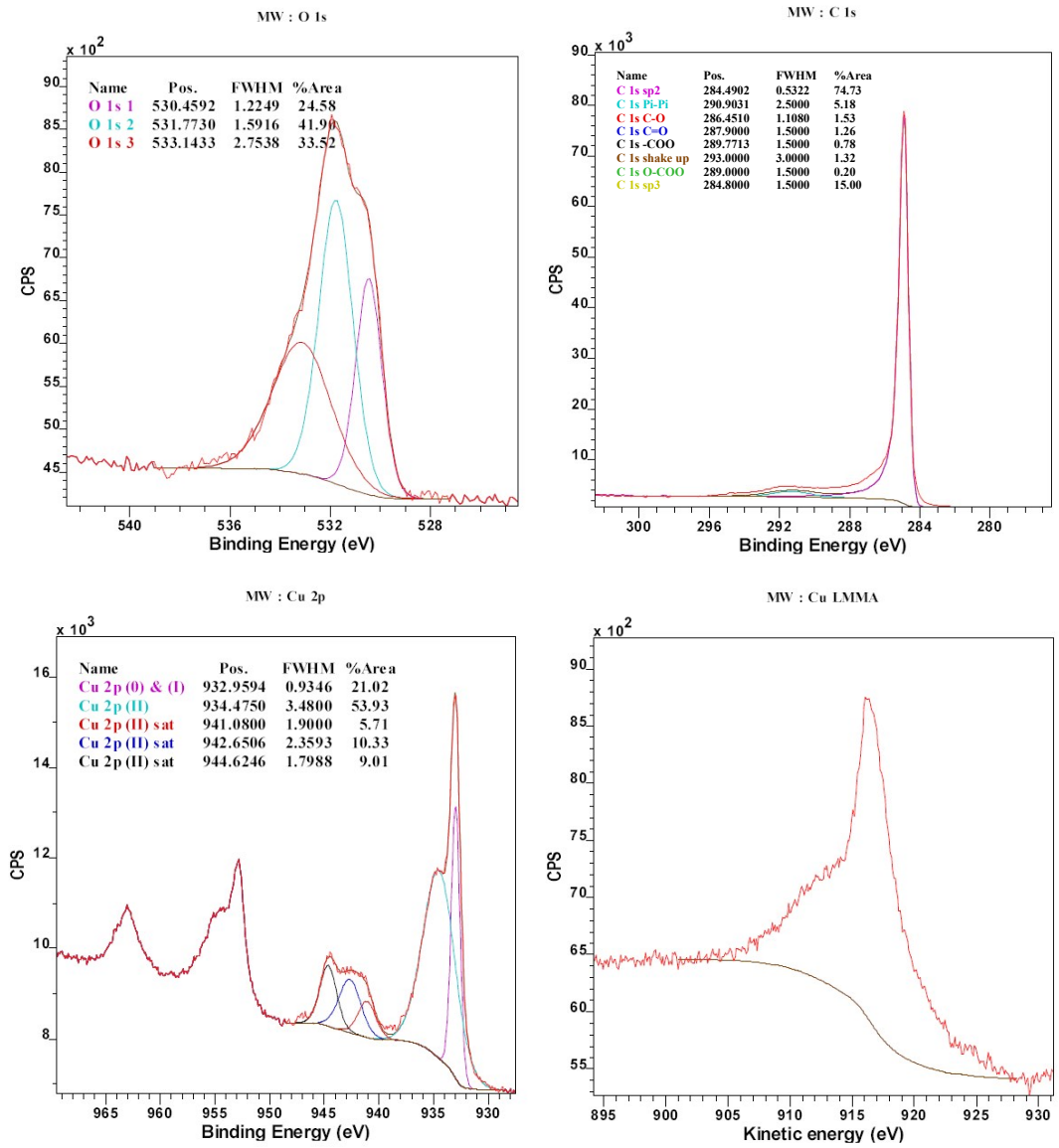


Figure S9. *De-convoluted high resolution XPS C1s, O1s, Cu2p region spectra and Cu Auger LMM kinetic energy spectra of Material* E_{MW}.

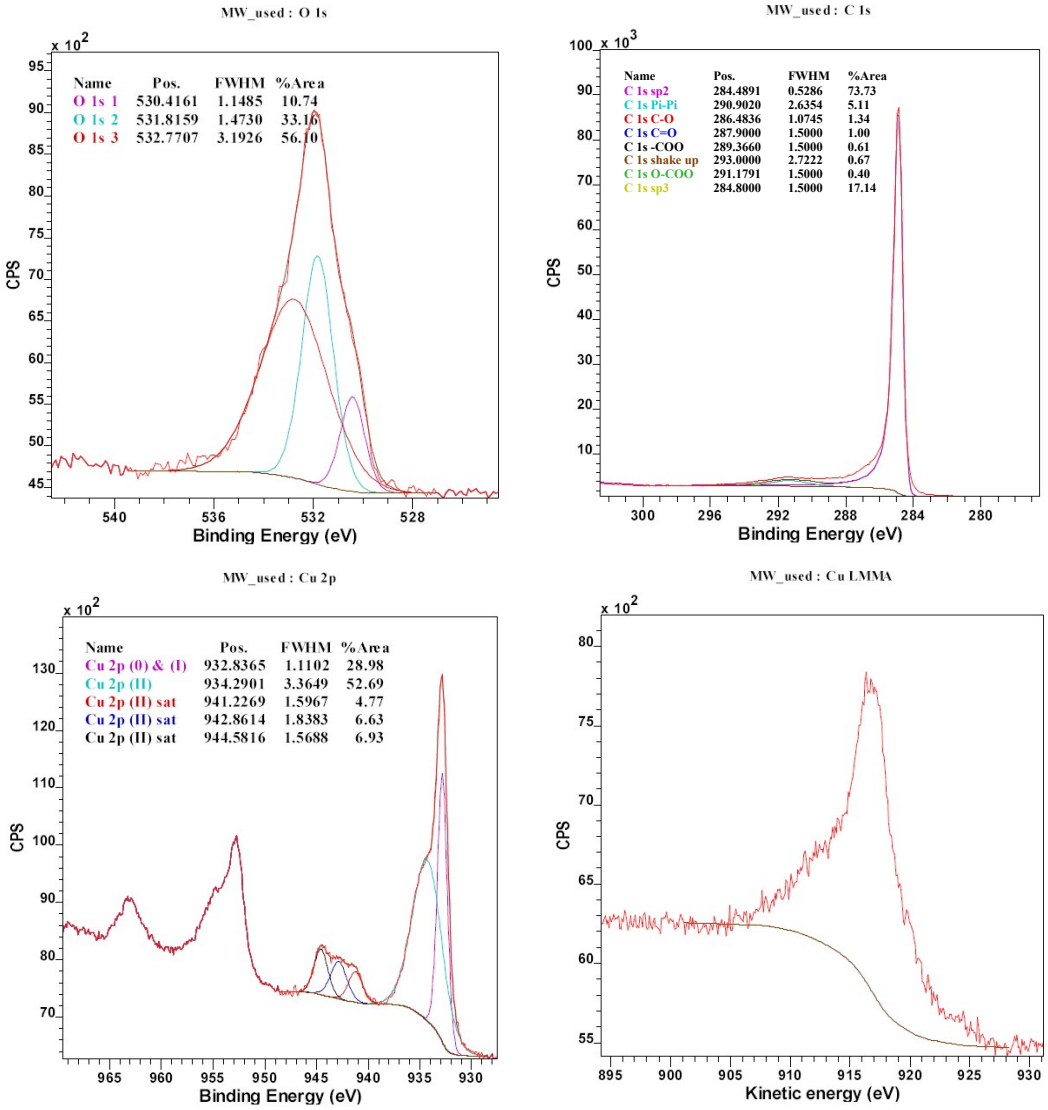


Figure S10. *De-convoluted high resolution XPS C1s, O1s, Cu2p region spectra and Cu Auger LMM kinetic energy spectra of Material* $\boldsymbol{E_{MW}r}$.

3.3. TEM images

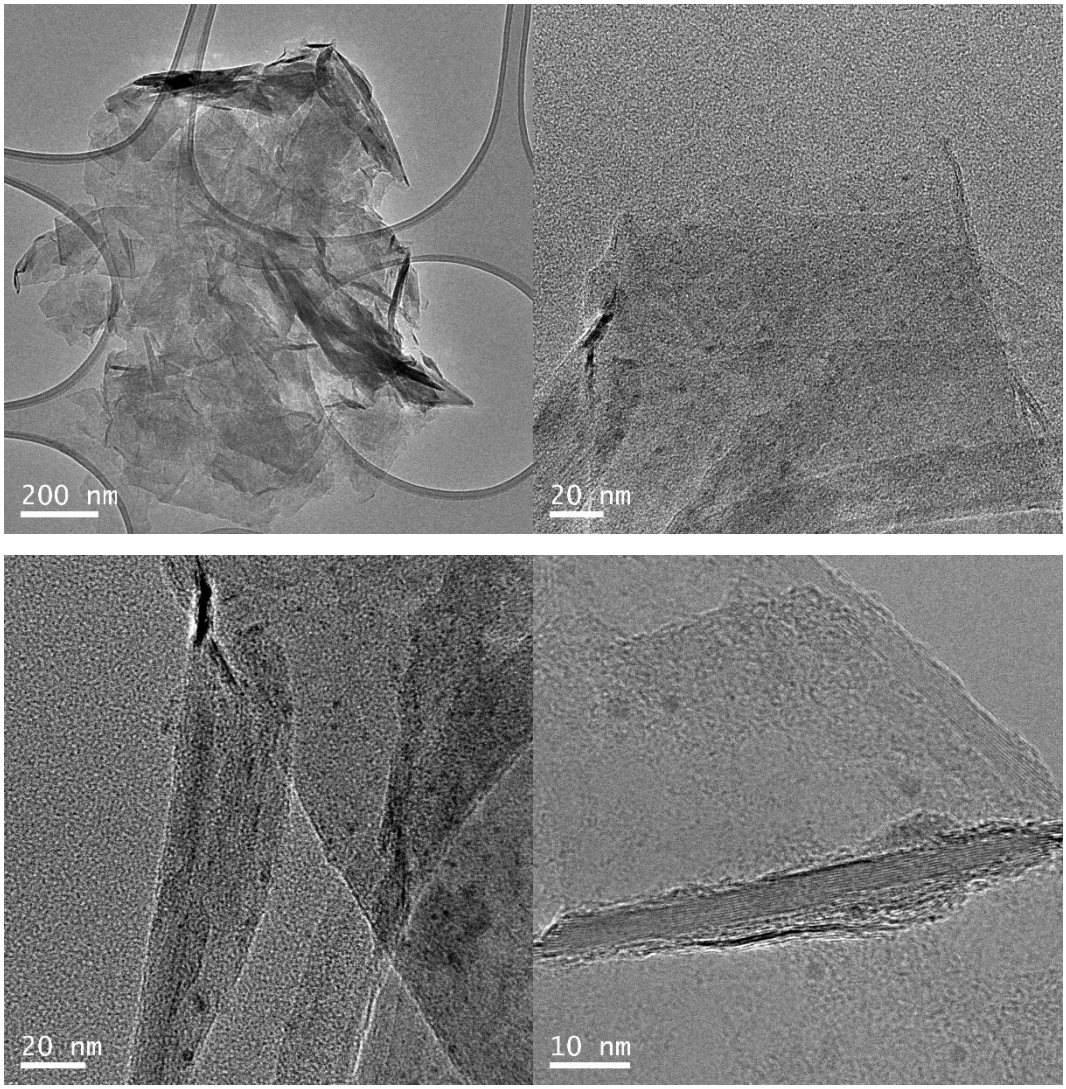


Figure S11. TEM images of material **A**.

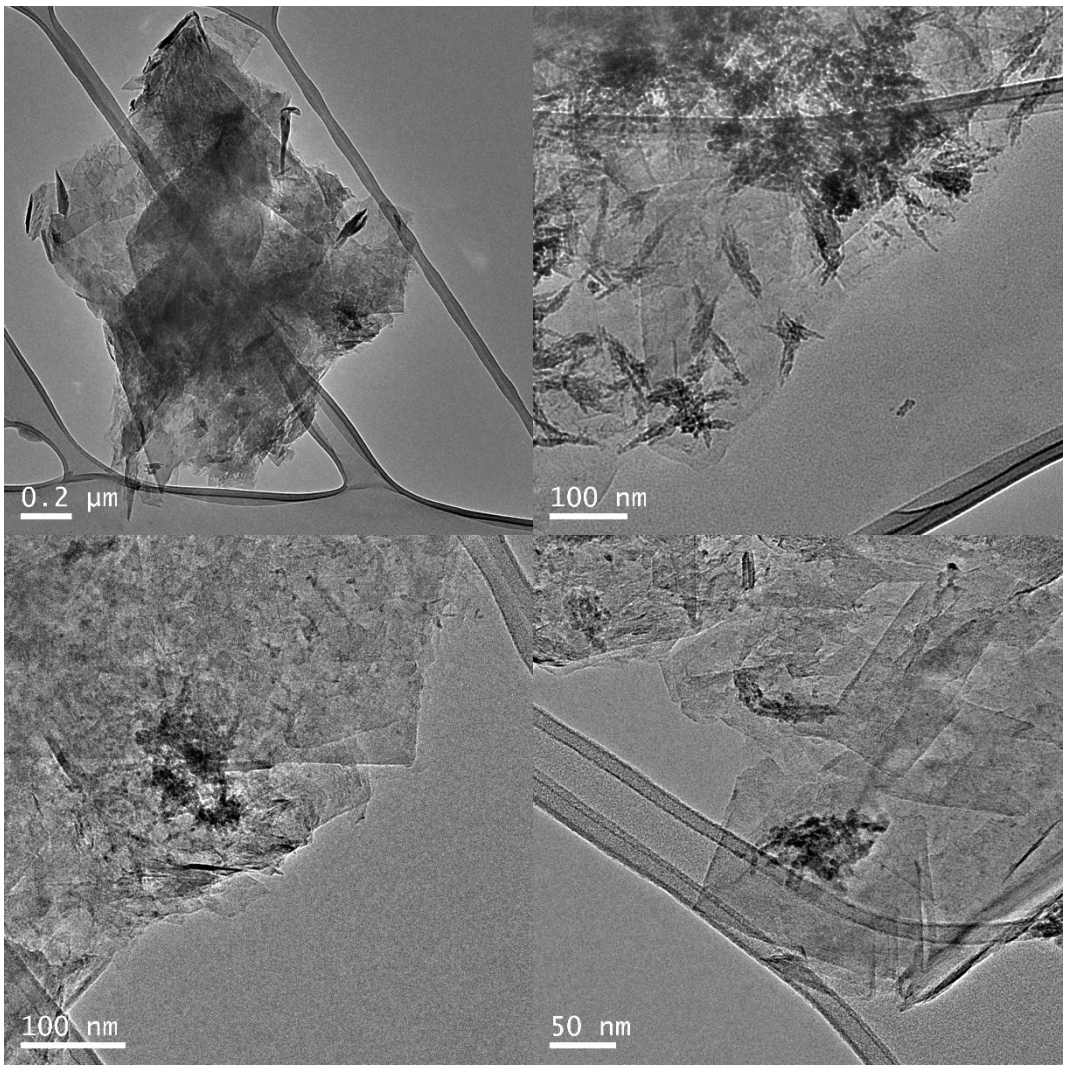


Figure S12. TEM images of Material Ar

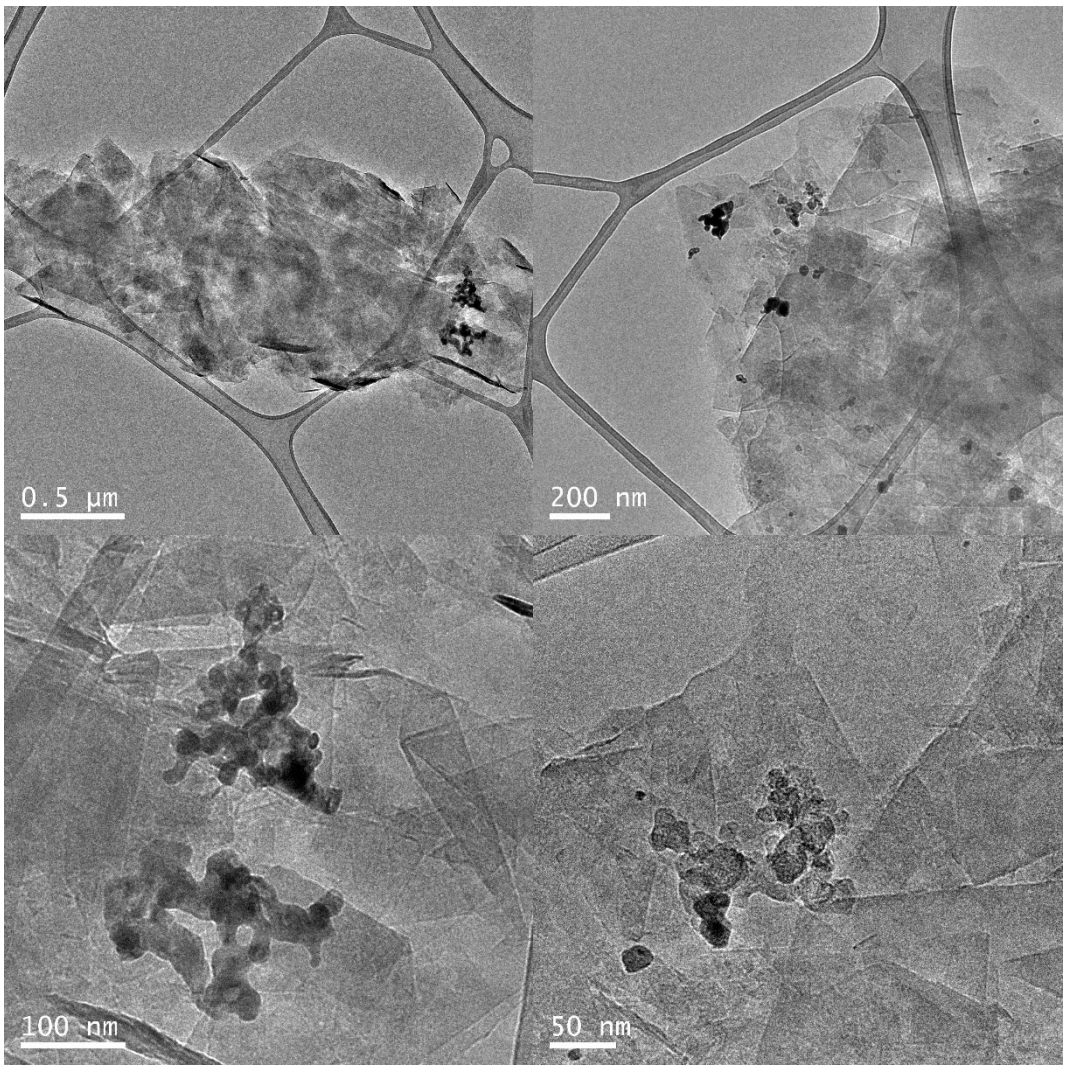


Figure S13. TEM images of material E_{MW}.

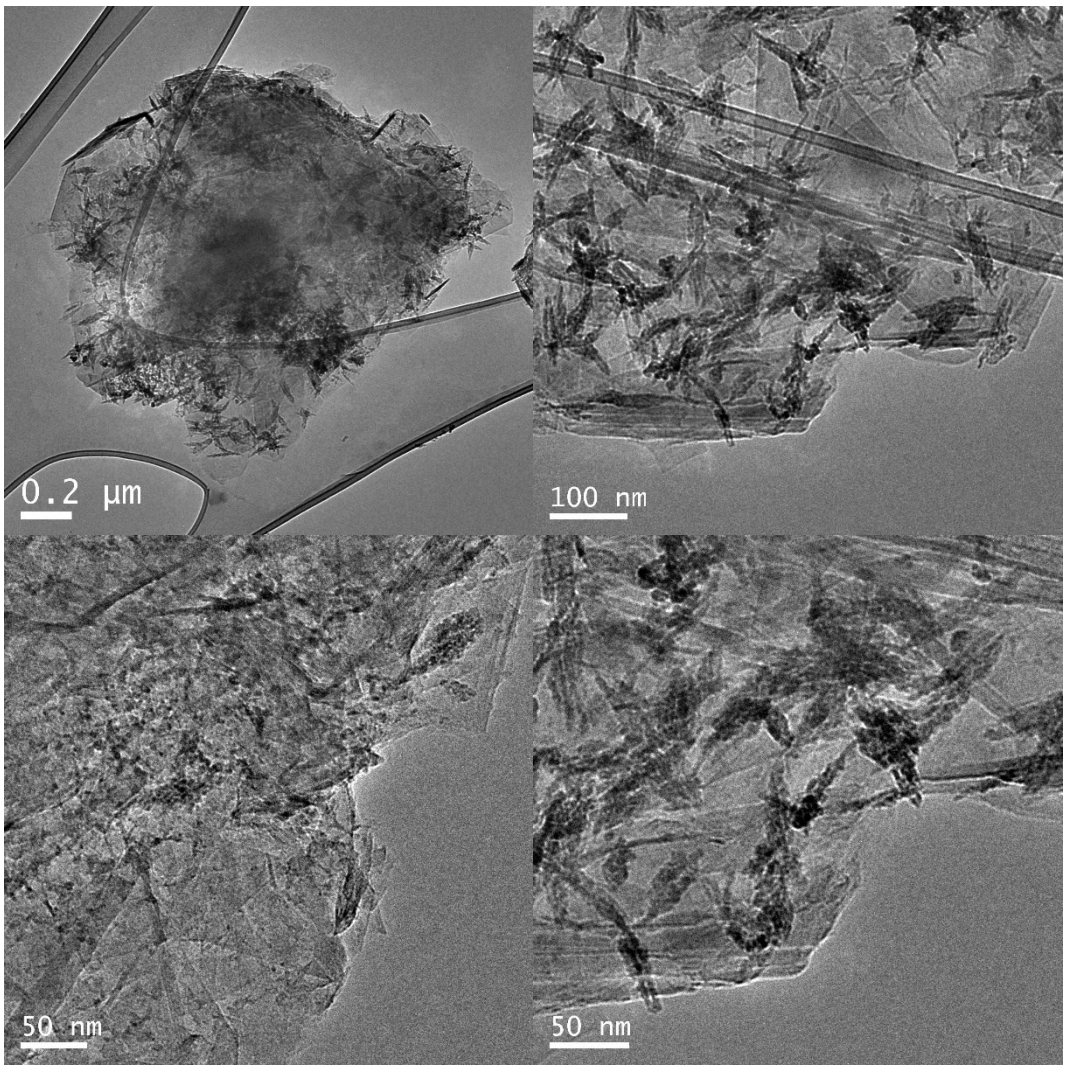
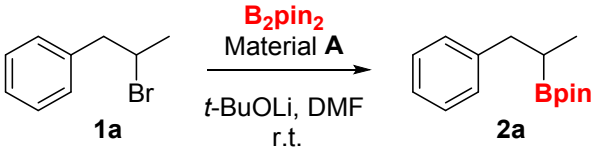


Figure S14. TEM images of material $E_{MW}r$.

4. Borylation Reaction

4.1. Alkyl bromides borylation optimization

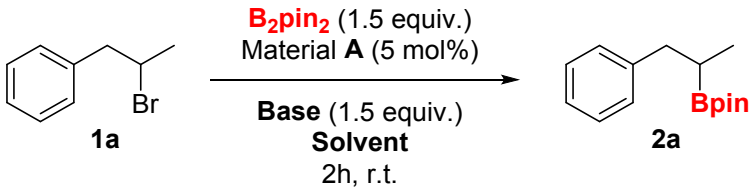
Table S4. Alkyl bromide borylation reaction: reagents ratio and catalyst loading screening



Entry	Catalyst (%)	B_2pin_2 (equiv.)	*t*-BuOLi (equiv.)	t (h)	Conv[a] (%)
1	10	2	2	1	100
2	5	2	2	2	100
3	2	2	2	4	60
4	2	2	2	15	100
5	1	1.5	1.5	24	0
6	10	1.5	2	2	100
7	5	1.5	2	2	100
8	**5**	**1.5**	**1.5**	**2**	**100(90)**
9	5	1.2	1.2	2	50

[a]Conversions determined by ^{1}H-NMR as starting material disappearance. Results in parenthesis correspond to the yields of the isolated products.

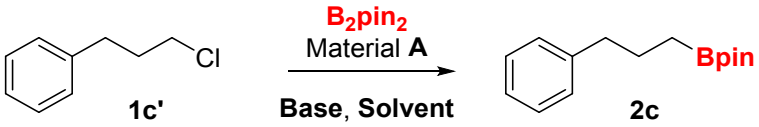
Table S5. Alkyl bromide borylation reaction: solvent and base screening



Entry	**Solvent**	**Base**	Conv[a] (%)
1	**DMF**	***t*-BuOLi**	**100**
2	Toluene	*t*-BuOLi	20
3	THF	*t*-BuOLi	75
4	CH_3CN	*t*-BuOLi	35
5	EtOH	*t*-BuOLi	50
6	DMF	*t*-BuOK	90
7	DMF	*t*-BuONa	50
8	DMF	MeOLi	50
9	DMF	MeOK	0
10	DMF	MeONa	0

[a]Conversions determined by ^{1}H-NMR as starting material disappearance.

Table S6. Alkyl chloride borylation optimization



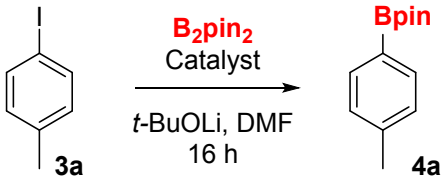
1c' 2c

Entry	Catalyst (%)	B_2pin_2 (equiv.)	**Base** (equiv.)	t (h)	T (°C)	Conv[a] (%)
1	**5**	**1.5**	**tBuOLi (1.5)**	**72**	**t.a.**	**40**
2	5	1.5	tBuOK (1.5)	72	t.a.	25
3	5	1.5	MeOK (1.5)	72	t.a.	12
4	5	1.5	tBuOLi (1.5)	72	60	36
5	10	2	tBuOLi (2)	120	60	10
6	10	2	tBuOLi (2)	72	110	0
7	10	2	Cs_2CO_3 (2)	72	110	0
8	10	2	CsF (2)	72	110	0
9[b]	10	2	tBuOLi (2)	48	t.a.	0
11[c]	5	1.5	tBuOLi (1.5)	72	t.a.	0

[a]Conversions determined by ^{1}H-NMR as starting material disappearance. [b] Reaction performed under white light LED radiation; toluene used as solvent. [c] 20 mol% of *N*-methylaniline added.

4.2. Aromatic halides borylation optimization

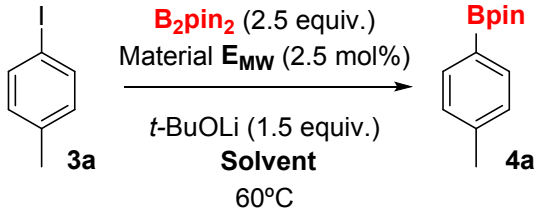
Table S7. Aryl iodide borylation reaction: reagents ratio and catalyst loading screening



Entry	Catalyst (%)	B_2pin_2 (equiv.)	Base (equiv.)	T (°C)	Conv[a]	Yield[b]
1	**A** (15)	2.5	2.5	25	100	20
2	**A** (10)	1.5	1.5	25	100	26
3	**A** (10)	1.5	1.5	60	100	30
4	**A** (5)	2	1.5	60	100	35
5	$\mathbf{E_{MW}}$ (5)	2	1.5	60	100	40
6	$\mathbf{E_{MW}}$ (5)	2	1.2	60	83	34
7	$\mathbf{E_{MW}}$ (2.5)	2	1.5	60	100	44
8	$\mathbf{E_{MW}}$ **(2.5)**	**2.5**	**1.5**	**60**	**100**	**50**
9	$\mathbf{E_{MW}}$ (15)	2.5	2.5	60	100	38
10	$\mathbf{E_{MW}}$ (5)	1.5	1.5	60	100	34
11	$\mathbf{E_{MW}}$ (5)	2	2.0	60	100	34
12	$\mathbf{E_{MW}}$ (5)	2.5	1.5	60	100	44
13	$\mathbf{E_{MW}}$ (5)	-	1.5	60	20	-
14	$\mathbf{E_{MW}}$ (5)	2	-	60	22	-

[a]Conversions determined by ^{1}H-NMR as starting material disappearance. [b]Yields determined by ^{1}H-NMR using nitromethane as internal standard.

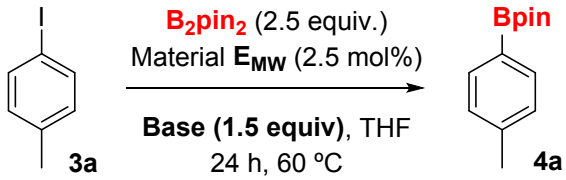
Table S8. Aryl iodide borylation reaction: solvent screening



Entry	**Solvent**	t (h)	Conv (%)	Yield[a] (%)
1	DMF	16	100	50
2	Toluene	16	74	13
3	Dioxane	16	62	trace
4	CH_3CN	16	48	16
5	THF	16	80	50
6	**THF**	**24**	**100**	**75 (68)**
7[b]	**THF**	**24**	**100**	**75 (67)**
8	2-MeTHF	24	14	-
9	DMA	16	100	40
10	DMPU	16	90	28
11	NMP	16	26	-
12	MTBE	16	20	-

[a]Yields determined by ^{1}H-NMR using nitromethane as internal standard. Yields in parenthesis correspond to the isolated products. [b]Material A used as catalyst.

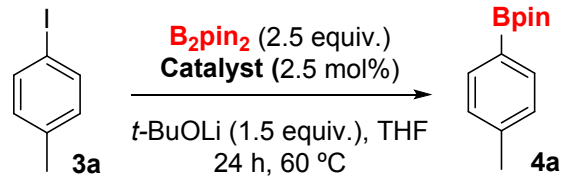
Table S9. *Aryl iodide borylation reaction: base screening*



Entry	**Base**	Conv (%)	Yield (%)[a]
1	***t*-BuOLi**	**100**	**75**
2	*t*-BuOK	35	-
3	*t*-BuONa	38	-
4	MeOLi	36	10
5	MeOK	26	5
6	MeONa	18	-
7	LiHMDS	30	6
8	KHMDS	80	23
9	NaHMDS	68	8
10	CsF	30	-
11	NaH	90	24

[a]Yields determined by ^{1}H-NMR using nitromethane as internal standard.

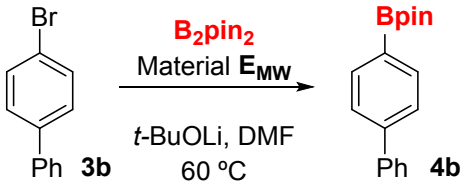
Table S*10*. *Aryl iodide borylation reaction: graphene-based catalysts comparison*



Entry	% catalyst	Conv (%)	Yield (%)
1	**Material A**	**100**	**75**
2	-	70	22
3	GraphenitOx	72	21
4	GO-Cu (**F**)	72	-
5	rGO-Cu (**G**)	100	70
6	CB-Cu (**H**)	100	67
7	Cu-NPs (**I**)	73	-

[a]Yields determined by ^{1}H-NMR using nitromethane as internal standard.

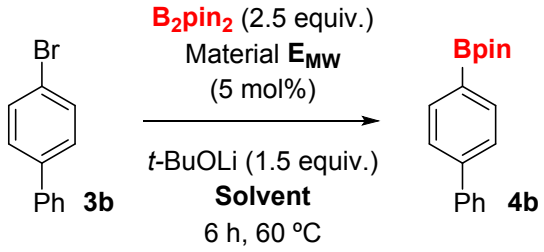
Table S*11*. *Aryl bromide borylation reaction: reagents ratio and catalyst loading screening*



Entry	% catalyst	B_2pin_2 (equiv.)	Base (equiv.)	t (h)	Yield[a] (%)
1[b]	15	2.5	*t*-BuOLi (2.5)	24	0
2	15	2.5	*t*-BuOLi (2.5)	24	35
3	10	2.5	*t*-BuOLi (2.5)	24	34
4	5	2.5	*t*-BuOLi (2.5)	24	32
5	15	2	*t*-BuOLi (2.5)	24	22
6	15	2.5	*t*-BuOLi (1.5)	24	45
7[c]	15	2.5	*t*-BuOLi (1.5)	24	40
8	10	2.5	*t*-BuOLi (1.5)	24	47
9	5	2.5	*t*-BuOLi (1.5)	24	45
10	15	2.5	*t*-BuOLi (1.5)	48	45
11	15	2.5	*t*-BuOLi (1.5)	16	47
12	10	2.5	*t*-BuOLi (1.5)	6	48
13	**5**	**2.5**	***t*-BuOLi (1.5)**	**6**	**44**
14	10	2.5	*t*-BuOLi (1.5)	3	45
15	5	2.5	*t*-BuOLi (1.5)	3	40
16	10	2.5	*t*-BuOLi (1.5)	3	45
17	10	3	*t*-BuOLi (1.5)	3	42

[a]Yields determined by ^{1}H-NMR using nitromethane as internal standard. [b]THF used as solvent. [c] 120 °C.

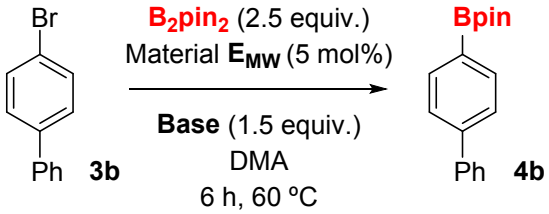
Table S*12*. *Aryl bromide borylation reaction: solvent screening*



Entry	Solvent	Yield[a] (%)
1	**DMA**	**50**
2	CH_3CN	0
3	Dioxane	0
4	MTBE	0
5	CH_2Cl_2	0
6	NMP	0
7	EtOH	Trace

[a]Yields determined by ^{1}H-NMR using nitromethane as internal standard.

Table S13. *Aryl bromide borylation reaction: base screening*

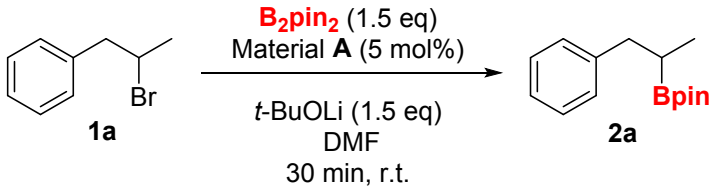


Entry	Base	Yield[a] (%)
1	*t*-BuOK	33
2	*t*-BuONa	41
3	MeOLi	45
4	MeOK	44
5	**MeONa**	**60**
6	LiHMDS	Trace
7	KHMDS	Trace
8	NaHMDS	26
9	CsF	Trace
10	NaH	45

[a]Yields determined by ^{1}H-NMR using nitromethane as internal standard.

4.3. Additional recyclability tests

Table S14. *Catalyst recyclability using vortex instead of US*

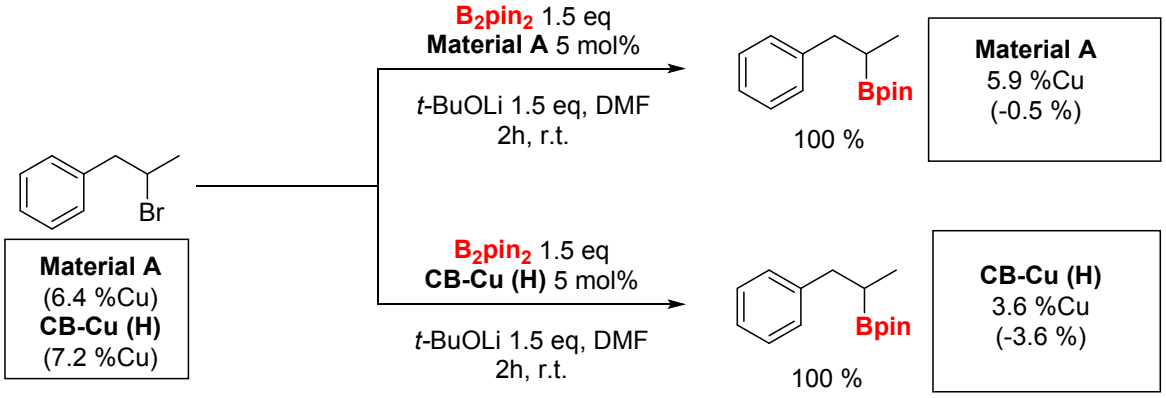


Run	Yield[a] (%)
1	90
2	87
3	87
4	86
5	84
6	22
7	0

[a]Yields showed correspond to the isolated yields.

Table S14 shows the efficiency of material **A** in the alkyl bromide borylation reaction after 7 runs. In this recyclability test, we checked if the selected dispersion method of the material (US, see **Table 5** in the manuscript) was comparable with another method such as vortex stirring. Under the same dispersion time (5 min), we observed a remarkable yield drop in the 5th cycle when using vortex, meaning a beneficial effect of the ultrasound to the catalysis. This behavior could be explained by a better exfoliation of the graphene sheets thanks to the ultrasound, exposing the catalytically active Cu(I) nanoparticles.

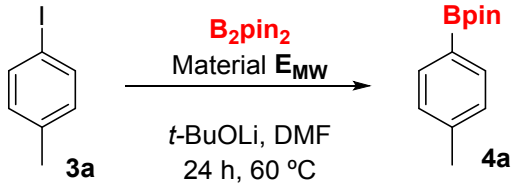
Scheme S1. *Recyclability comparison between Material A and Material H*



Scheme S1 shows the analysis of the copper contain of Graphenit-Cu(I) (**Material A**) and the catalyst prepared from carbon black (**H**) before and after one use. As can be seen, carbon black losses half of the copper content (only a 3.6 % of copper remains in the material) whereas material **A** is recovered with a 5.9 % of Cu.

4.4. Mechanistic assays and blank experiments

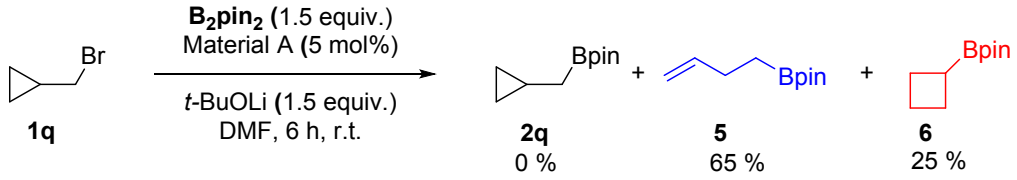
Table S15. *Mechanistic experiments for the aromatic halide borylation*



Entry	Additive	Catalyst (%)	B_2pin_2 (equiv.)	Base (equiv.)	Conv (%)	Yield[a] (%)
1[b]	-	15	2.5	2.5	100	20
2	-	10	1.5	1.5	100	30
3	-	15	-	2.5	70	0
4	-	10	-	1.5	42	0
5	-	15	-	-	60	0
6	Air	2.5	2.5	1.5	76	13
7	H_2O (2 eq)	2.5	2.5	1.5	66	53
8	TEMPO (2 eq)	2.5	2.5	1.5	20	0
9	BHT (2 eq)	2.5	2.5	1.5	40	0

[a]Yields determined by ^{1}H-NMR using nitromethane as internal standard. [b]25 °C.

***Scheme S2**. Radical clock reaction using cyclopropyl bromide as substrate of the borylation reaction*



The reaction was carried out following general procedure B. The isolated **5/6** products mixture was obtained by flash column chromatography purification of the crude using pentane : diethyl ether 20:1 as eluent.

According to the observed spectra of the mixture, we could identify the NMR signals of both open (**5**) and cycled products (**6**).[3] The difficulty to separate the two borylated products by conventional flash column chromatography led us to their isolation as a mixture. Is important to note that in both ^{1}H and ^{13}C spectra appear important signals of different solvents such as pentane or diethyl ether. This is due to the impossibility of completely drying the products after the purification given the low boiling points of the products.

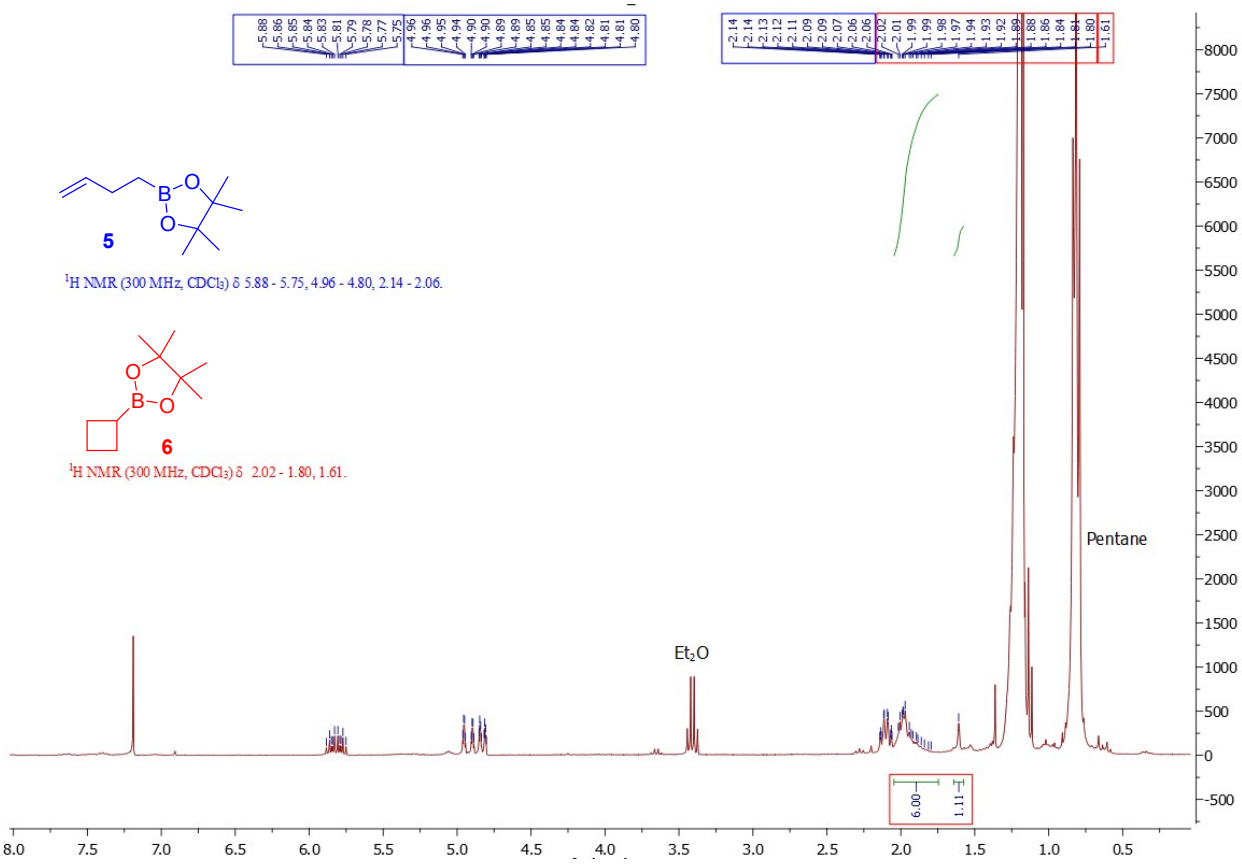


***Figure S15**. ^{1}H-NMR spectra of the pinacol boronic esters **5** and **6** mixture.*

In the ^{1}H-NMR spectra (Figure S15), we could perfectly distinguish the NMR signals of the olefin present in the open product (**5**) at 6 and 5 ppm, as well as the allylic methylene signals at 2.2 ppm. Close to this multiplet, we could find a complex signal at 2 ppm that goes with a singlet at 1.6 ppm in a 6 to 1 ratio. These two multiplets match the described signals for the cyclobutyl boronic acid pinacol ester (**6**).

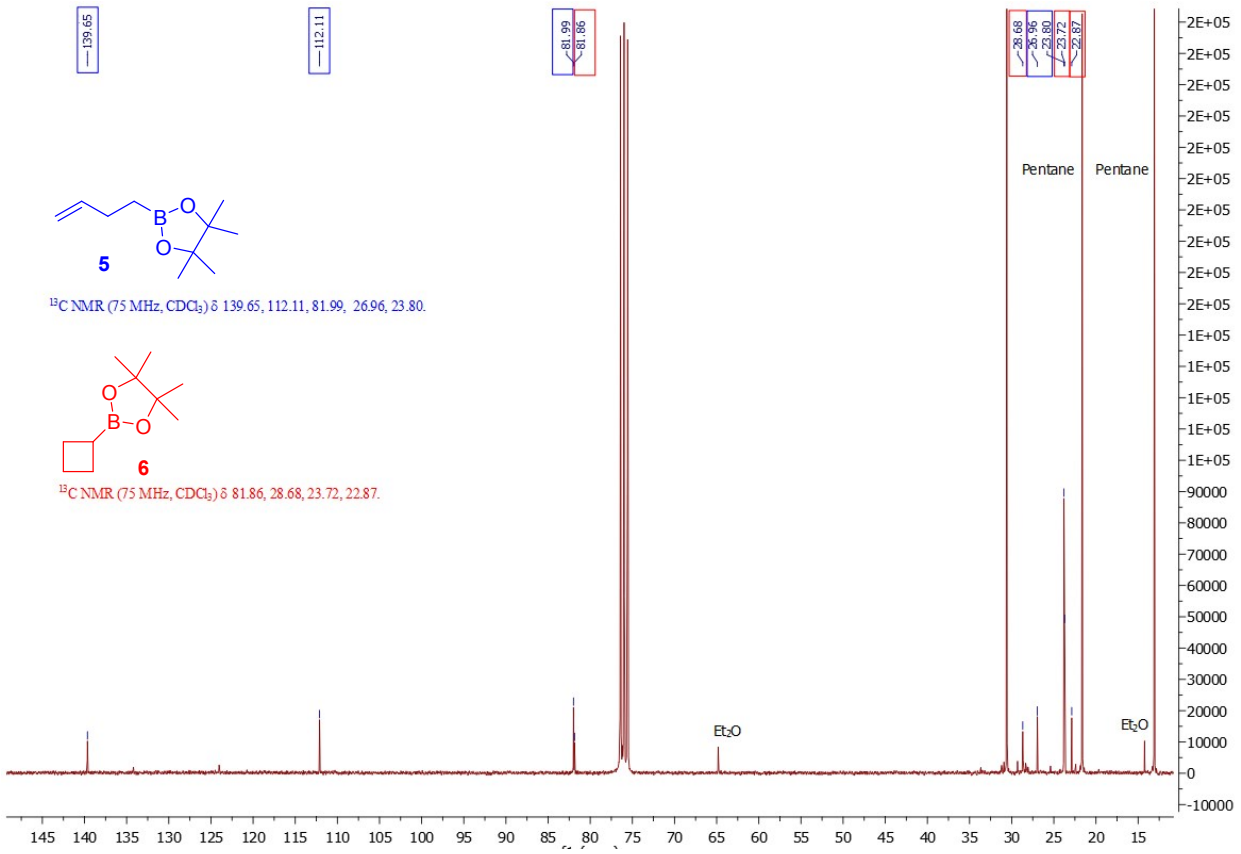
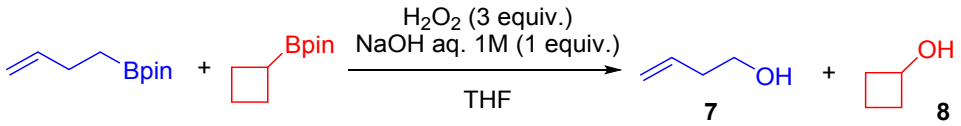


***Figure S16**. ^{13}C-NMR spectra of the pinacol boronic esters **5** and **6** mixture*

The ^{13}C-NMR spectra shows again the signals of both products matching the previously described spectroscopic data (figure S16). The quadrupolar nature of the boron isotopes makes the signal of the carbon directly attached to the boron so broad that remains unseen in the spectra.

***Scheme S3**. Oxidation reaction of the pinacol boronic esters mixture*



In order to ensure the existence of the cyclobutyl product, we carried out the oxidation of the mixture using the H_2O_2/NaOH system (scheme S3). After reaction completion, we could detect in the ^{1}H-NMR spectra of the mixture a quintuplet at 4 ppm that could belong to the CH attached to the alcohol in the oxidized product **8** (figure S17). The comparison between this signal and the described for the cyclobutanol revealed that they have the same multiplicity and chemical shift.

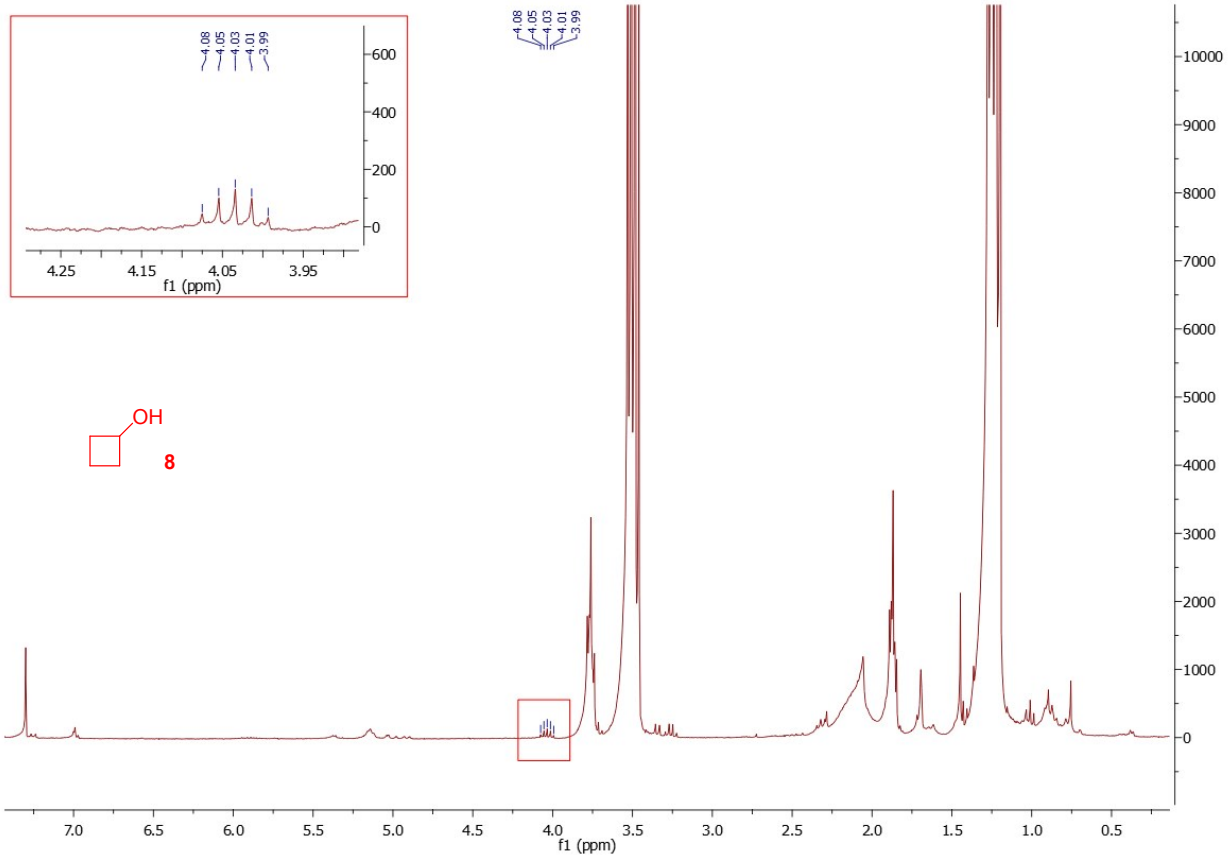
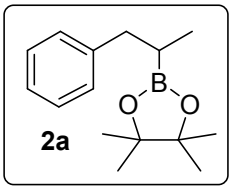


Figure S17. *^{1}H-NMR spectra of the pinacol boronic ester oxidation.*

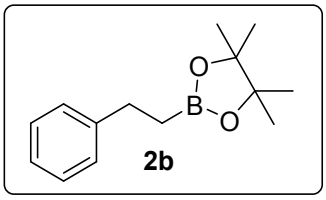
5. Characterization of compounds

4,4,5,5-tetramethyl-2-(1-phenylpropan-2-yl)-1,3,2-dioxaborolane (2a)[4]



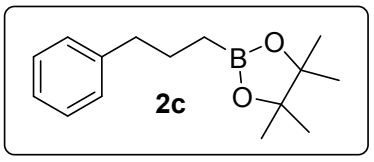
Prepared following general procedure A with no need of Ar atmosphere. The product was purified by column chromatography (cyclohexane:ethyl acetate 20:1) to get the product **2a** (55.4 mg, 90%) as a white solid. Spectroscopical data is in accordance with the literature. **^{1}H NMR ($CDCl_3$, 300 MHz)** δ 7.31 – 7.11 (m, 5H), 2.82 (dd, *J* = 13.6, 7.5 Hz, 1H), 2.56 (dd, *J* = 13.6, 8.3 Hz, 1H), 1.39 (m, 1H), 1.20 (d, *J* = 3.1 Hz, 12H), 0.98 (d, *J* = 7.4 Hz, 3H).

4,4,5,5-tetramethyl-2-phenethyl-1,3,2-dioxaborolane (2b)[5]



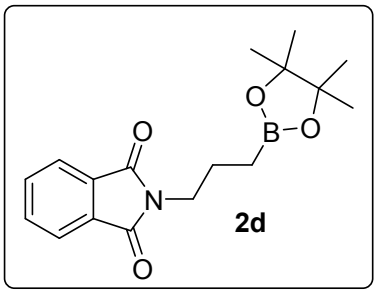
Prepared following general procedure A with no need of Ar atmosphere. The product was purified by column chromatography (cyclohexane:ethyl acetate 20:1) to get the product **2b** (52.3 mg, 90%) as a colorless oil. Spectroscopical data is in accordance with the literature. **^{1}H NMR ($CDCl_3$, 300 MHz)** δ 7.30-7.13 (m, 5H), 2.77 (t, J = 7.9 Hz, 2H), 1.24 (s, 12H), 1.16 (t, J = 7.9 Hz, 2H).

4,4,5,5-tetramethyl-2-(3-phenylpropyl)-1,3,2-dioxaborolane (2c)[6]



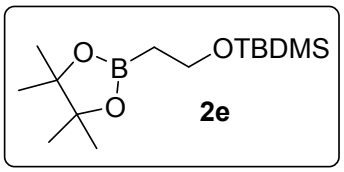
Prepared following general procedure A with no need of Ar atmosphere. The product was purified by column chromatography (cyclohexane:ethyl acetate 20:1) to get the product **2c** (56.6 mg, 92%) as a white solid. Spectroscopical data is in accordance with the literature. **^{1}H NMR ($CDCl_3$, 300 MHz, $CDCl_3$)** 7.32-7.26 (m, 2H), 7.23-7.13 (m, 3H), 2.63 (t, J = 7.8 Hz, 2H), 1.75 (p, J = 7.8 Hz, 2H), 1.26 (s, 12H), δ 0.85 (t, J = 7.9 Hz, 2H).

2-(3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propyl)isoindoline-1,3-dione (2d)



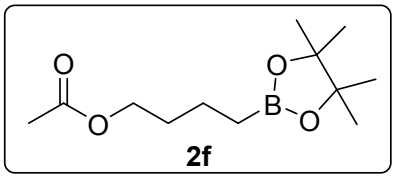
Prepared following general procedure A under Ar atmosphere. The product was purified by column chromatography (cyclohexane:ethyl acetate 4:1) to get the product **2d** (50.4 mg, 64%) as a pale yellow oil. This compound is new. **^{1}H NMR ($CDCl_3$, 300 MHz)** δ 7.78-7.81 (m, 2H), 7.69-7.65 (m, 2H), 3.66 (t, J = 7 Hz, 2H), 1.77 (m, 2H), 1.19 (s, 12H), 0.79 (t, J = 8 Hz, 2H). **^{13}C NMR (75 MHz, $CDCl_3$):** δ 168.8 (2 CO), 134.1 (2 CH), 132.6 (2 CH), 123.4 (2 CH), 83.5 (2 C), 40.2 (CH_2), 25.1 (4 CH_3), 23.5 (CH_2). **MS** (ES): 337 (M^+, 1), 316 (22), 216 (122), 173 (165), 139 (199). **HRMS** (ES): calculated for $C_{17}H_{22}BNO_4$ (M^+): 338.1537; found: 338.1543.

Tert-butyldimethyl(2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)ethoxy)silane (2e)



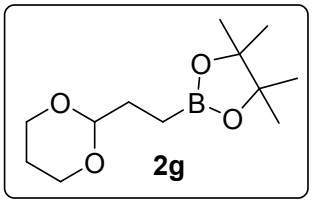
Prepared following general procedure A with no need of Ar atmosphere. The product was purified by column chromatography (cyclohexane:ethyl acetate 10:1) to get the product **2e** (60.8 mg, 85%) as a colorless oil. This compound is new. **^{1}H NMR ($CDCl_3$, 300 MHz)** δ 3.76 (t, J = 7.5 Hz, 2H), 1.23 (s, 12H), 1.10 (t, J = 7.6 Hz, 2H), 0.87 (s, 9H), 0.03 (s, 6H). **^{13}C NMR (75 MHz, $CDCl_3$):** δ 83.4 (2 C), 60.3 (CH_2), 26.3 (3 CH_3), 25.2 (4 CH_3), 18.7 (C), -4.8 (2 CH_3). **MS** (ES): 308 (M^+, 1), 287, (22), 172 (137), 139 (170), 113(196). **HRMS** (ES): calculated for $C_{14}H_{31}BO_3Si$ (M^+): 309.2031; found: 309.2036.

4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)butyl acetate (2f)



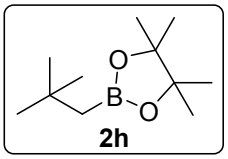
Prepared following general procedure A with no need of Ar atmosphere. The product was purified by column chromatography (cyclohexane:ethyl acetate 4:1) to get the product **2f** (52.7 mg, 87%) as a colorless oil. This compound is new. **^{1}H NMR ($CDCl_3$, 300 MHz)** δ 4.02 (t, J = 6.5 Hz, 2H), 3.00 (s, 3H), 1.60 (m, 2H) 1.45 (m, 2H), 1.22 (s, 12H), 0.77 (t, J = 7.7 Hz, 2H). **^{13}C NMR ($CDCl_3$, 75 MHz)** δ 171.5 (CO), 83.3 (2 C), 64.8 (CH_2), 31.5 (CH_2), 25.1 (4 CH_3), 21.4 (CH_3), 20.8 (CH_2). **MS** (ES): 264 (M^+, 1), 243 (22), 143 (122), 139 (126), 113 (152), 59 (206). **HRMS** (ES): calculated for $C_{12}H_{23}BO_4$ (M^+): 265.1584; found: 265.1579.

2-(2-(1,3-dioxan-2-yl)ethyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (2g)[4]



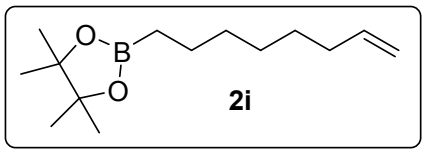
Prepared following general procedure A with no need of Ar atmosphere. The product was purified by column chromatography (cyclohexane:ethyl acetate 3:1) to get the product **2g** (54.5 mg, 90%) as a pale yellow oil. Spectroscopical data is in accordance with the literature. **^{1}H NMR ($CDCl_3$, 300 MHz)** δ 4.48 (t, *J* = 5.1 Hz, 1H), 4.09 (ddd, *J* = 11.7, 5.0, 1.2 Hz, 2H), 3.74 (td, *J* = 12.4, 2.5 Hz, 2H), 2.16 – 1.98 (m, 1H), 1.73 (td, *J* = 7.7, 5.2 Hz, 2H), 1.30 (m, 1H), 1.24 (s, 12H), 0.83 (t, *J* = 7.7 Hz, 2H).

4,4,5,5-tetramethyl-2-neopentyl-1,3,2-dioxaborolane (2h)[7]



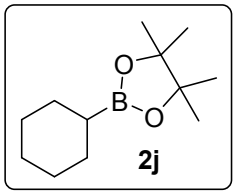
Prepared following general procedure A with no need of Ar atmosphere. The product was purified by column chromatography (pentane:diethyl ether 50:1) to get the product **2h** (37.1 mg, 75%) as a colorless oil. Spectroscopical data is in accordance with the literature. **^{1}H NMR ($CDCl_3$, 300 MHz)** δ 1.25 (s, 12H), 0.99 (s, 9H), 0.81 (s, 2H). **^{13}C NMR (75 MHz, $CDCl_3$):** δ 83.2 (2 C), 27.3 (3 CH_3), 25.2 (4 CH_3), 25.2 (C).

4,4,5,5-tetramethyl-2-(oct-7-en-1-yl)-1,3,2-dioxaborolane (2i)



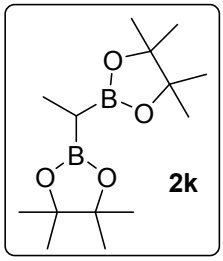
Prepared following general procedure A with no need of Ar atmosphere. The product was purified by column chromatography (cyclohexane:ethyl acetate 50:1) to get the product **2i** (29.8 mg, 50%) as a colorless oil. This compound is new. **^{1}H NMR ($CDCl_3$, 300 MHz)** δ 5.80 (ddt, *J* = 16.9, 10.1, 6.7 Hz, 1H), 5.04 – 4.86 (m, 2H), 2.02 (q, *J* = 6.9 Hz, 2H), 1.41 - 1.28 (m, 8H), 1.25 (s, 12H), 0.76 (t, *J* = 7.6 Hz, 2H). **^{13}C NMR (75 MHz, $CDCl_3$):** δ 139.6 (CH), 114.4 (CH), 83.2 (2 C), 34.2 (CH_2), 32.6 (CH_2), 29.3 (CH_2), 29.2 (CH_2) 25.2 (4 CH_3), 24.3 (CH_2). **MS** (ES): 499 (M^+, dimer), 261 (M^+), 139 (122), 113 (148). **HRMS** (ES): calculated for $C_{14}H_{27}BO_2$ (M^+, dimer): 499.4110; found: 499.4121.

2-cyclohexyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (2j)[4]



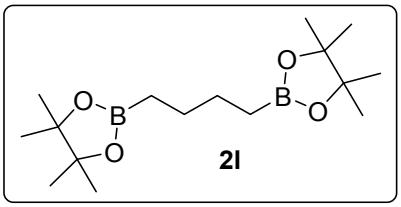
Prepared following general procedure A with no need of Ar atmosphere. The product was purified by column chromatography (pentane:diethyl ether 50:1) to get the product **2j** (31.0 mg, 59%) as a colorless oil. Spectroscopical data is in accordance with the literature. **^{1}H NMR ($CDCl_3$, 300 MHz)** δ 1.70-1.52 (m, 4H), 1.38-1.23 (m, 6H), 1.22 (s, 12H), 1.02-0.90 (m, 1H).

2,2'-(ethane-1,1-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (2k)[4]



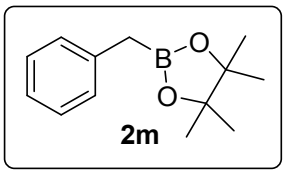
Prepared following general procedure A with no need of Ar atmosphere. The product was purified by column chromatography (cyclohexane:ethyl acetate 10:1) to get the product **2k** (42.3 mg, 60%) as a pale yellow oil. Spectroscopical data is in accordance with the literature. **^{1}H NMR ($CDCl_3$, 300 MHz)** δ 1.21 (s, 24H), 1.03 (d, *J* = 7.1 Hz, 3H), 0.71 (q, *J* = 6.9 Hz, 1H).

1,4-bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)butane (2l)



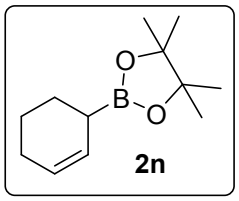
Prepared following general procedure A with no need of Ar atmosphere. The product was purified by column chromatography (cyclohexane:ethyl acetate 50:1) to get the product **2l** (59.7 mg, 77%) as a white solid. This compound is new. **^{1}H NMR ($CDCl_3$, 300 MHz)** δ 1.44-1-35 (m, 4H), 1.22 (s, 24H), 0.78-0.70 (m, 4H). **^{13}C NMR (75 MHz, $CDCl_3$):** δ 83.6 (4 C), 27.7 (2 CH_2), 25.6 (8 CH_3). **MS** (ES): 332 (M^+, 1), 328 (5), 211 (122), 139 (194). **HRMS** (ES): calculated for $C_{16}H_{32}B_2O_4$ (M^+): 333.2385; found: 333.2395.

2-benzyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (2m)[4]



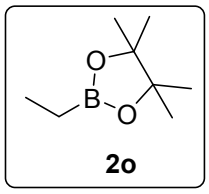
Prepared following general procedure A with no need of Ar atmosphere. The product was purified by column chromatography (cyclohexane:ethyl acetate 20:1) to get the product **2m** (44.1 mg, 81%) as a colorless oil. Spectroscopical data is in accordance with the literature. **^{1}H NMR ($CDCl_3$, 300 MHz)** δ 7.09-7.23 (m, 5H), 2.29 (s,2H), 1.29 (s, 12H).

2-(cyclohex-2-en-1-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (2n)[5]



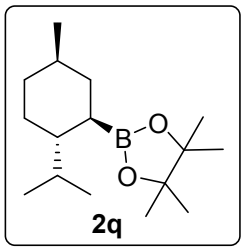
Prepared following general procedure A under Ar atmosphere. The product was purified by column chromatography (cyclohexane:ethyl acetate 50:1) to get the product **2n** (38.0 mg, 73%) as a colorless oil. Spectroscopical data is in accordance with the literature. **^{1}H NMR ($CDCl_3$, 300 MHz)** δ 5.74 – 5.64 (m, 2H), 1.99 (m, 2H), 1.55 – 1.80 (m, 5H), 1.25 (s, 2H).

2-ethyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (2o)[8]



Prepared following general procedure A (starting from 10 mmol of bromoethane) with no need of Ar atmosphere. The product was purified by column chromatography (petroleum ether:diethyl ether 50:1) to get the product **2o** (1.322 g, 85%) as a colorless oil. Spectroscopical data is in accordance with the literature. **^{1}H NMR ($CDCl_3$, 300 MHz)** δ 1.23 (s, 12H), 0.93 (t, J = 7.6 Hz, 3H), 0.74 (m, 2H).

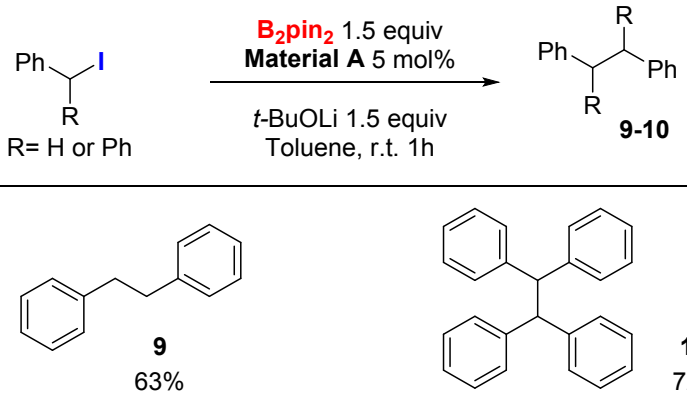
2-((1R,2R,5R)-2-isopropyl-5-methylcyclohexyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (2p)[4]



Prepared following general procedure A with no need of Ar atmosphere. The product was purified by column chromatography (cyclohexane:ethyl acetate 20:1) to get the product **2p** (39.8 mg, 60%) as a white solid. Spectroscopical data is in accordance with the literature. **^{1}H NMR ($CDCl_3$, 300 MHz)** δ 0.77 (d, J = 7 Hz, 2H), 0.84 (d, J = 6.5 Hz, 2H), 0.88-1.02 (m, 7H), 1.20-1.35 (m, 14H), 1.55-1.75 (m, 4H).

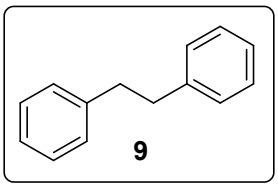
Selective formation of the benzylic homocoupled products

***Scheme S5**. Reaction conditions to obtain the homocoupled benzylic products*



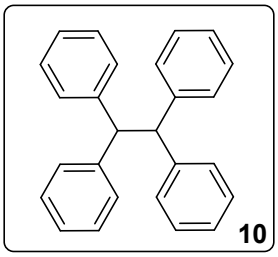
Scheme S5 shows the reaction conditions found for the selective obtention of the homocoupled products using benzylic or bibenzylic bromides as starting materials.

1,2-diphenylethane (9)[9]



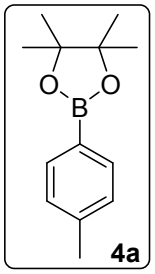
Prepared following general procedure A using toluene as solvent with no need of Ar atmosphere. The product was purified by column chromatography (cyclohexane) to get the product **9** (28.7 mg, 63%) as a colorless oil. Spectroscopical data is in accordance with the literature. **^{1}H NMR ($CDCl_3$, 300 MHz)** δ 2.93 (s, 4H), 7.15-7.30 (m, 10H).

1,1,2,2-tetraphenylethane (10)[10]



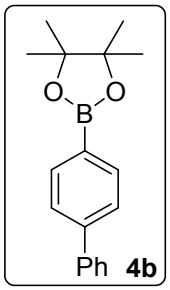
Prepared following general procedure A using toluene as solvent with no need of Ar atmosphere. The product was purified by column chromatography (cyclohexane) to get the product **10** (60.2 mg, 72%) as a colorless oil. Spectroscopical data is in accordance with the literature. **^{1}H NMR ($CDCl_3$, 300 MHz)** δ 4.77 (s, 2H), 6.93-7.02(m, 4H), 7.07-7.11 (m, 8H), 7.14-7.17 (m, 8H).

4,4,5,5-tetramethyl-2-(p-tolyl)-1,3,2-dioxaborolane (4a)[11]



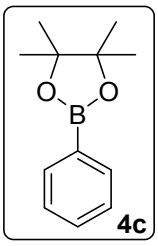
Prepared following general procedures B or C (starting from the aryl iodide or bromide, respectively). The product was purified by column chromatography (cyclohexane:ethyl acetate 20:1) to get the product **4a** (37.0 mg, 68%) as a pale yellow oil. Spectroscopical data in accordance with the literature. **^{1}H NMR ($CDCl_3$, 300 MHz)** δ 7.73 (d, *J* = 7.6 Hz, 2H), 7.21 (d, *J* = 7.5 Hz, 2H), 2.38 (s, 3H), 1.36 (s, 12H).

2-([1,1'-biphenyl]-4-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (4b)[12]



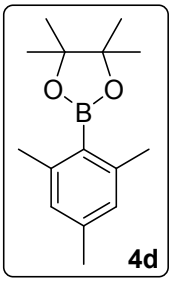
Prepared following general procedure C (starting from the aryl bromide). The product was purified by column chromatography (cyclohexane:ethyl acetate 20:1) to get the product **4b** (39.2 mg, 56%) as a white solid. Spectroscopical data in accordance with the literature. **^{1}H NMR ($CDCl_3$, 300 MHz)** δ 7.92 (d, *J* = 8.2 Hz, 2H), 7.68 – 7.61 (m, 4H), 7.51 – 7.43 (m, 2H), 7.42 – 7.34 (m, 1H), 1.39 (s, 12H).

4,4,5,5-tetramethyl-2-phenyl-1,3,2-dioxaborolane (4c)[13]



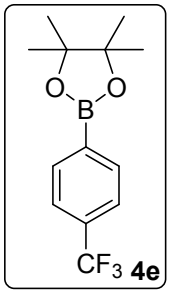
Prepared following general procedures B or C (starting from the aryl iodide or bromide, respectively). The product was purified by column chromatography (cyclohexane:ethyl acetate 30:1) to get the product **4c** (34.1 mg, 67%) as a pale yellow liquid. Spectroscopical data in accordance with the literature. **^{1}H NMR ($CDCl_3$, 300 MHz)** δ 7.84 (d, *J* = 6.4 Hz, 2H), 7.51-7.45 (m, 1H), 7.41-7.36 (m, 2H), 1.37 (s, 12H).

2-mesityl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (4d)[11]



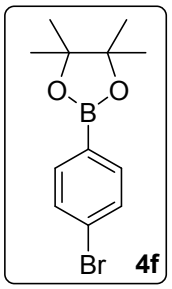
Prepared following general procedure B (starting from the aryl iodide). The product was purified by column chromatography (cyclohexane:ethyl acetate 50:1) to get the product **4d** (30.2 mg, 49%) as a pale yellow oil. Spectroscopical data in accordance with the literature. **^{1}H NMR ($CDCl_3$, 300 MHz)** δ 6.79 (s, 2H), 2.38 (s, 6H), 2.25 (s, 3H), 1.39 (s, 12H).

4,4,5,5-tetramethyl-2-(4-(trifluoromethyl)phenyl)-1,3,2-dioxaborolane (4e)[11]



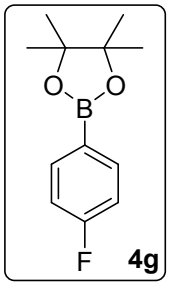
Prepared Following general procedure B or C (starting from the aryl iodide or bromide, respectively).The product was purified by column chromatography (cyclohexane:ethyl acetate 20:1) to get the product **4e** (47.6 mg, 70%) as a white solid. Spectroscopical data in accordance with the literature. **^{1}H NMR ($CDCl_3$, 300 MHz)** δ 7.93 (d, *J* = 7.8 Hz, 2H), 7.63 (d, *J* = 7.8 Hz, 2H), 1.37 (s, 12H).

2-(4-bromophenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (4f)[11]



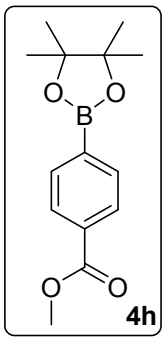
Prepared following general procedure B (starting from the aryl iodide).. The product was purified by column chromatography (cyclohexane:ethyl acetate 20:1) to get the product **4f** (53.0mg, 75%) as a pale yellow oil. Spectroscopical data in accordance with the literature. **^{1}H NMR ($CDCl_3$, 300 MHz)** δ 7.67 (d, *J* = 7.9 Hz, 2H), 7.51 (d, *J* = 7.9 Hz, 2H), 1.34 (s, 12H).

2-(4-fluorophenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (4g)[13]



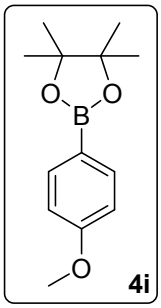
Prepared following general procedure B (starting from the aryl iodide). The product was purified by column chromatography (cyclohexane:ethyl acetate 20:1) to get the product **4g** (41.1 mg, 74%) as a pale yellow oil. Spectroscopical data in accordance with the literature. **^{1}H NMR ($CDCl_3$, 300 MHz)** δ 7.87-7.81 (m, 2H), 7.13-7.05 (m, 2H), 1.38 (s, 12H).

Methyl 4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzoate (4h)[12]



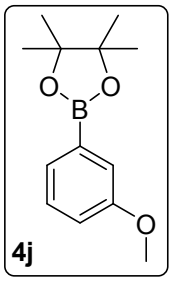
Prepared following general procedure B (starting from the aryl iodide). The product was purified by column chromatography (cyclohexane:ethyl acetate 10:1) to get the product **4h** (40.0 mg, 61%) as a pale yellow oil. Spectroscopical data in accordance with the literature. **^{1}H NMR ($CDCl_3$, 300 MHz)** δ 8.02 (d, *J* = 8.3 Hz, 2H), 7.87 (d, *J* = 8.3 Hz, 2H), 3.92 (s, 3H), 1.36 (s, 12H).

2-(4-methoxyphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (4i)[12]



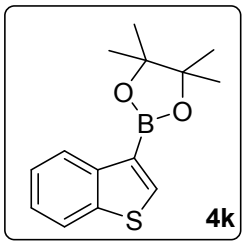
Prepared following general procedure B or C (starting from the aryl iodide or bromide, respectively).The product was purified by column chromatography (cyclohexane:ethyl acetate 20:1) to get the product **4i** (45.1 mg, 77%) as a pale yellow oil. Spectroscopical data in accordance with the literature. **^{1}H NMR ($CDCl_3$, 300 MHz)** δ 7.78-7.74 (m, 2H), 6.92-6.88 (m, 2H), 3.84 (s, 3H), 1.34 (s, 12H).

2-(3-methoxyphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (4j)[13]



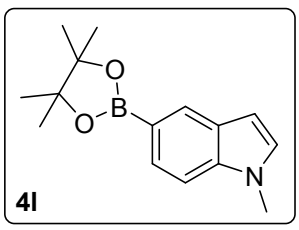
Prepared following general procedure B (starting from the aryl iodide). The product was purified by column chromatography (cyclohexane:ethyl acetate 20:1) to get the product **4j** (38.0 mg, 65%) as a pale yellow oil. Spectroscopical data in accordance with the literature. **^{1}H NMR ($CDCl_3$, 300 MHz)** δ 7.42 (d, J = 7.2 Hz, 1H), 7.38-7.25 (m, 2H), 7.06-6.99 (m, 1H), 3,85 (s, 3H), 1.36 (s, 12H).

2-(benzo[b]thiophen-3-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (4k)[14]



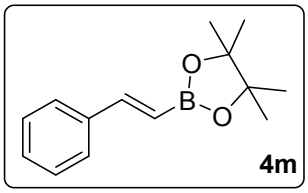
Prepared following general procedure C (starting from the aryl bromide). The product was purified by column chromatography (cyclohexane:ethyl acetate 20:1) to get the product **4k** (19.5 mg, 30%) as a white solid. Spectroscopical data in accordance with the literature. **^{1}H NMR ($CDCl_3$, 300 MHz)** δ 8.42 – 8.36 (m, 1H), 8.09 (s, 1H), 7.92-7.88 (m, 1H), 7.45 – 7.31 (m, 2H), 1.40 (s, 12H).

1-methyl-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1H-indole (4l)[15]



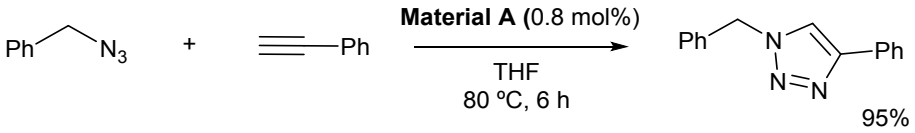
Prepared following general procedure C (starting from the aryl bromide). The product was purified by column chromatography (cyclohexane:ethyl acetate 20:1) to get the product **4l** (27.0 mg, 42%) as a white solid. Spectroscopical data in accordance with the literature. **^{1}H NMR ($CDCl_3$, 300 MHz)** δ 8.17 (s, 1H), 7.68 (dd, J = 8.3, 1.0 Hz, 1H), 7.33 (d, J = 8.3 Hz, 1H), 7.05 (d, J = 3.1 Hz, 1H), 6.51 (dd, J = 3.1, 0.8 Hz, 1H), 3.80 (s, 3H), 1.38 (s, 12H).

(E)-4,4,5,5-tetramethyl-2-styryl-1,3,2-dioxaborolane (4m)[16]



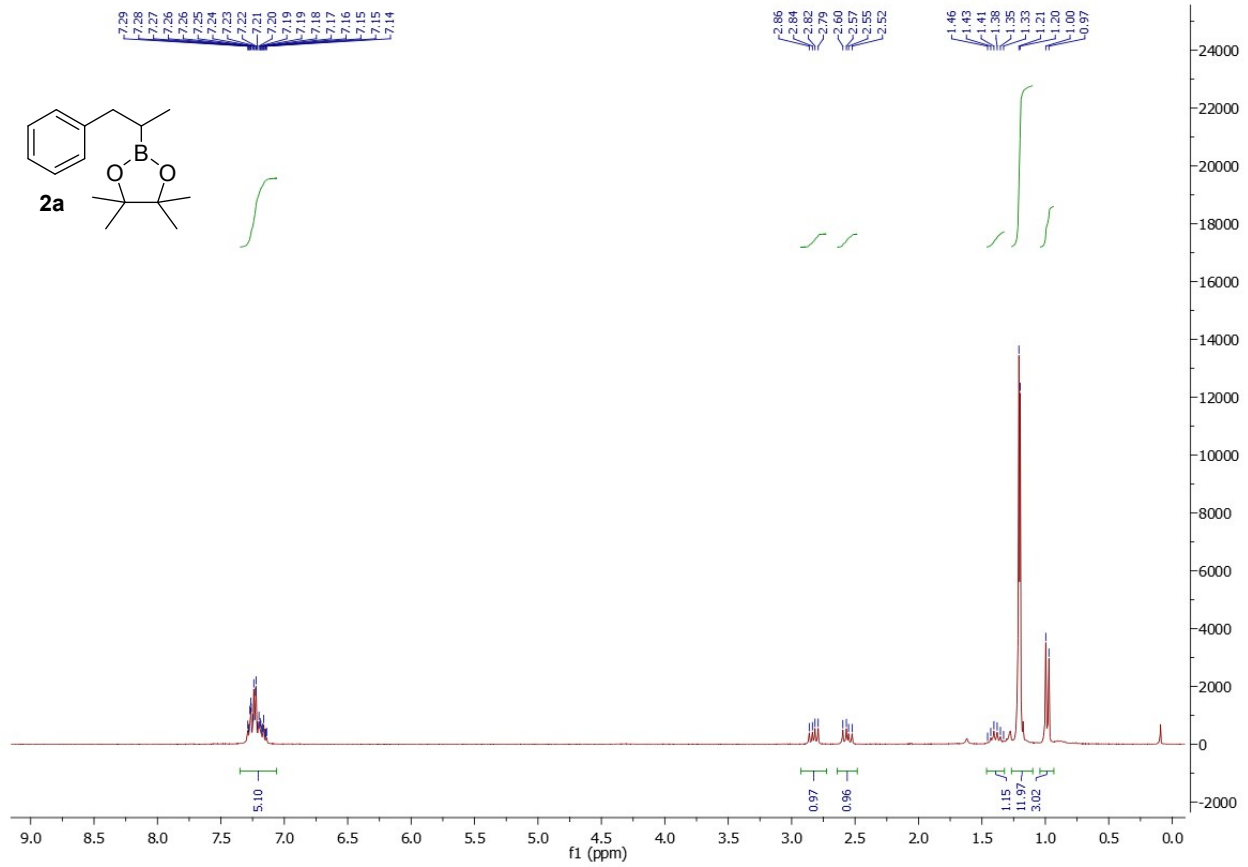
Prepared following general procedure C (starting from the vinyl bromide). The product was purified by column chromatography (cyclohexane:ethyl acetate 50:1) to get the product **4m** (38.0 mg, 66%) as a colorless oil. Spectroscopical data in accordance with the literature. **^{1}H NMR ($CDCl_3$, 300 MHz)** δ 7.57 – 7.51 (m, 2H), 7.45 (d, *J* = 18.6 Hz, 1H), 7.42 – 7.25 (m, 3H), 6.22 (d, *J* = 18.4 Hz, 1H), 1.36 (s, 12H).

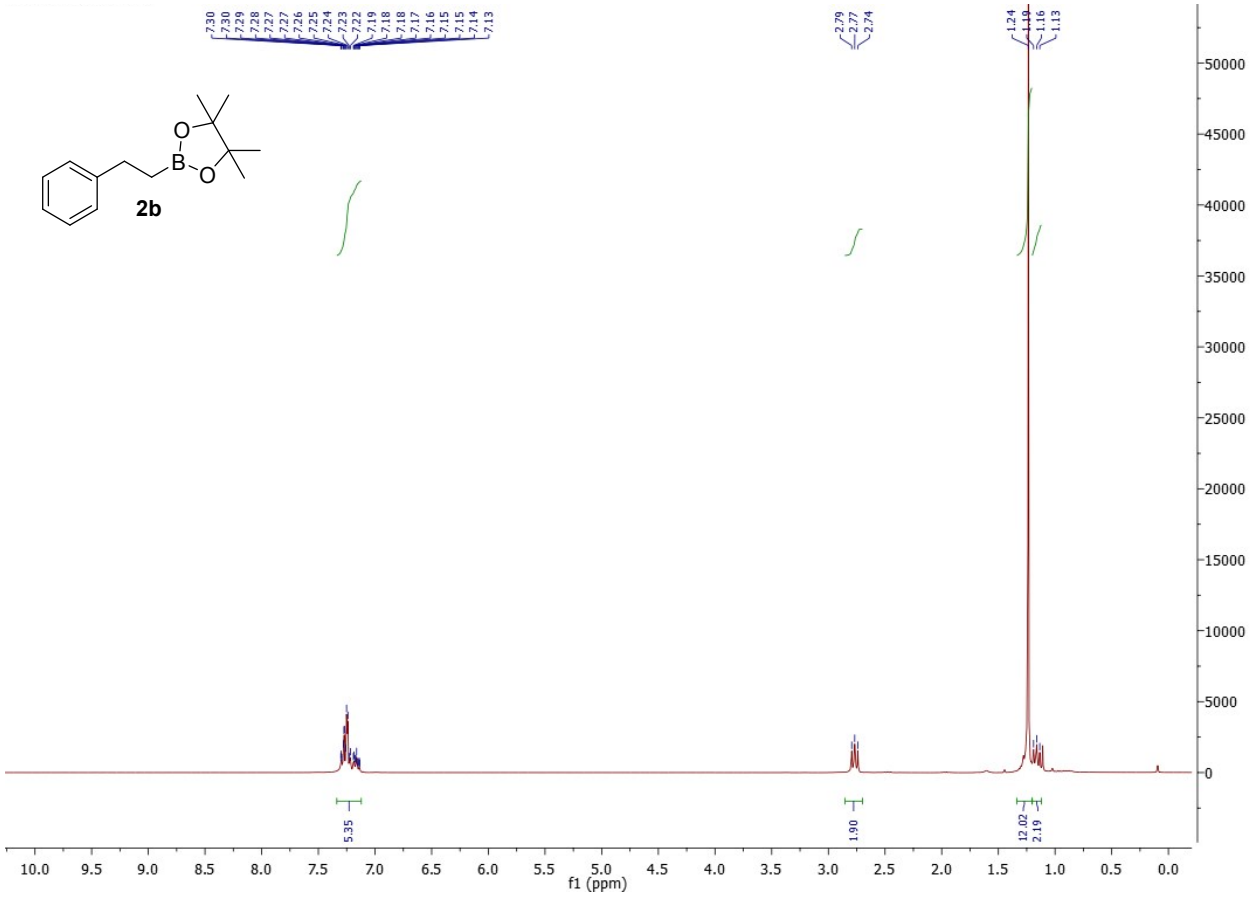
Procedure for 1-benzyl-4-phenyl-1H-1,2,3-triazole synthesis

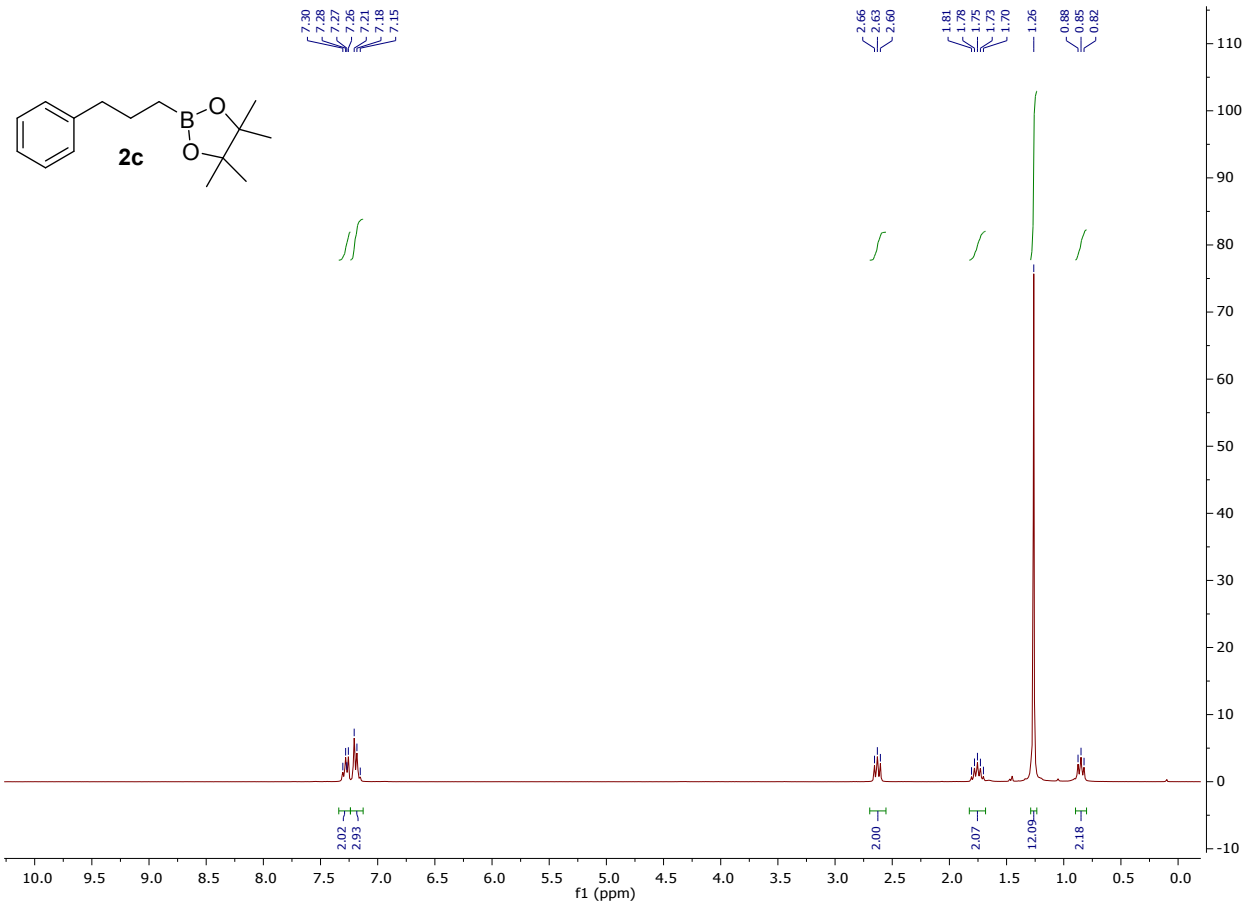


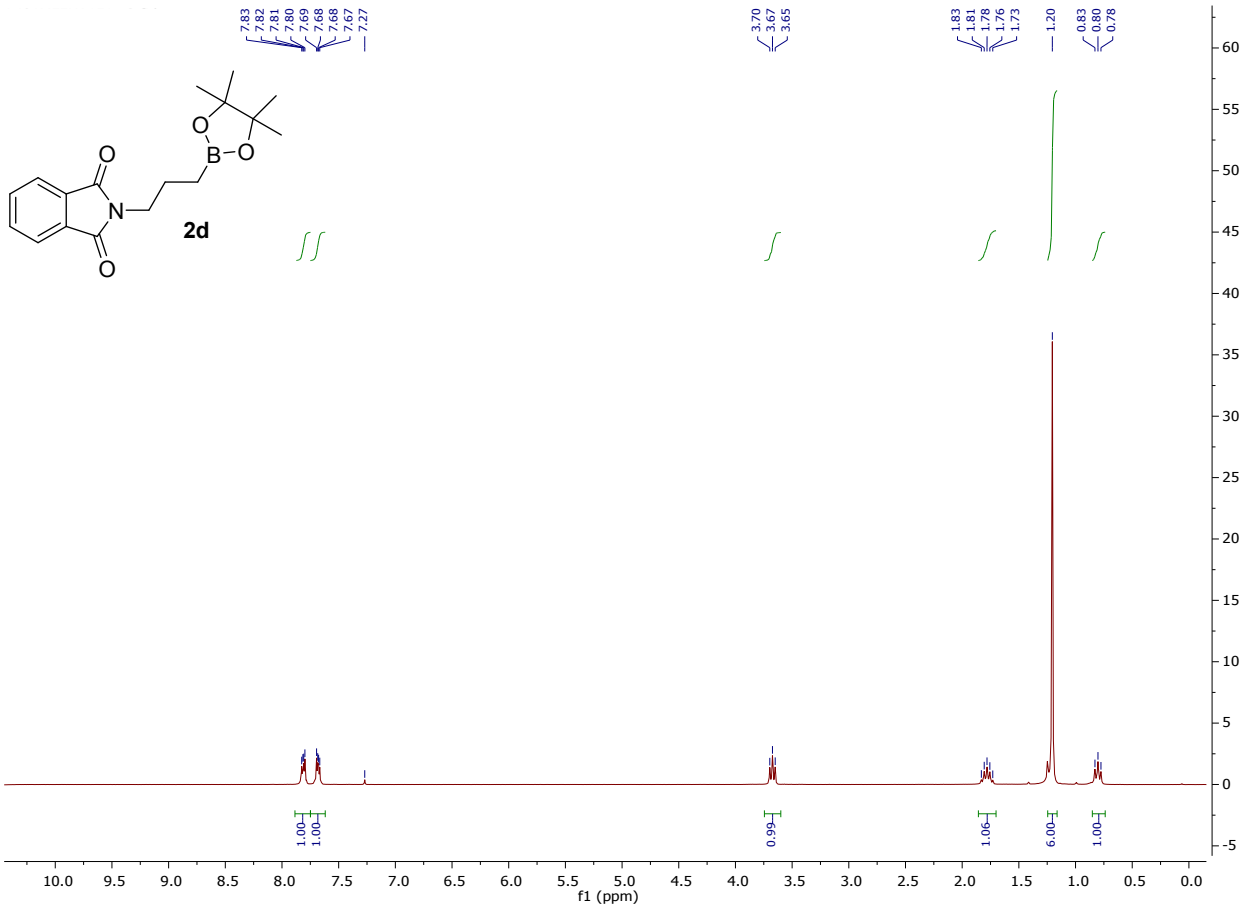
In a capped vial, material **A** (2.4 mg, 0.8 mol%) was suspended in THF (1 mL) and dispersed in an ultrasound bath during 5 min. Then, benzyl azide (0.43 mmol) and phenylacetylene (0.43 mmol) were added to the solution and heated at 80 °C for 6 h. Then, the catalyst is separated from the mixture by centrifugation at 7000 rpm, collecting the supernatant. Once separated, the catalyst is washed and resuspended with EtOAc, and separated by centrifugation collecting the supernatant. This process is repeated one more time. The solution is concentrated in vacuo and the residue was purified by flash chromatography (cyclohexane:ethyl acetate, 6:4) to afford the corresponding 1,2,3-triazole (96.1 mg, 95%) as a white solid. **^{1}H-RMN ($CDCl_3$, 300 MHz):** δ 7.77 (m, 2H), 7.65 (s, 1H), 7.32 (m, 8H), 5.53 (s, 2H). The spectroscopic data match with the described in the literature.[17]

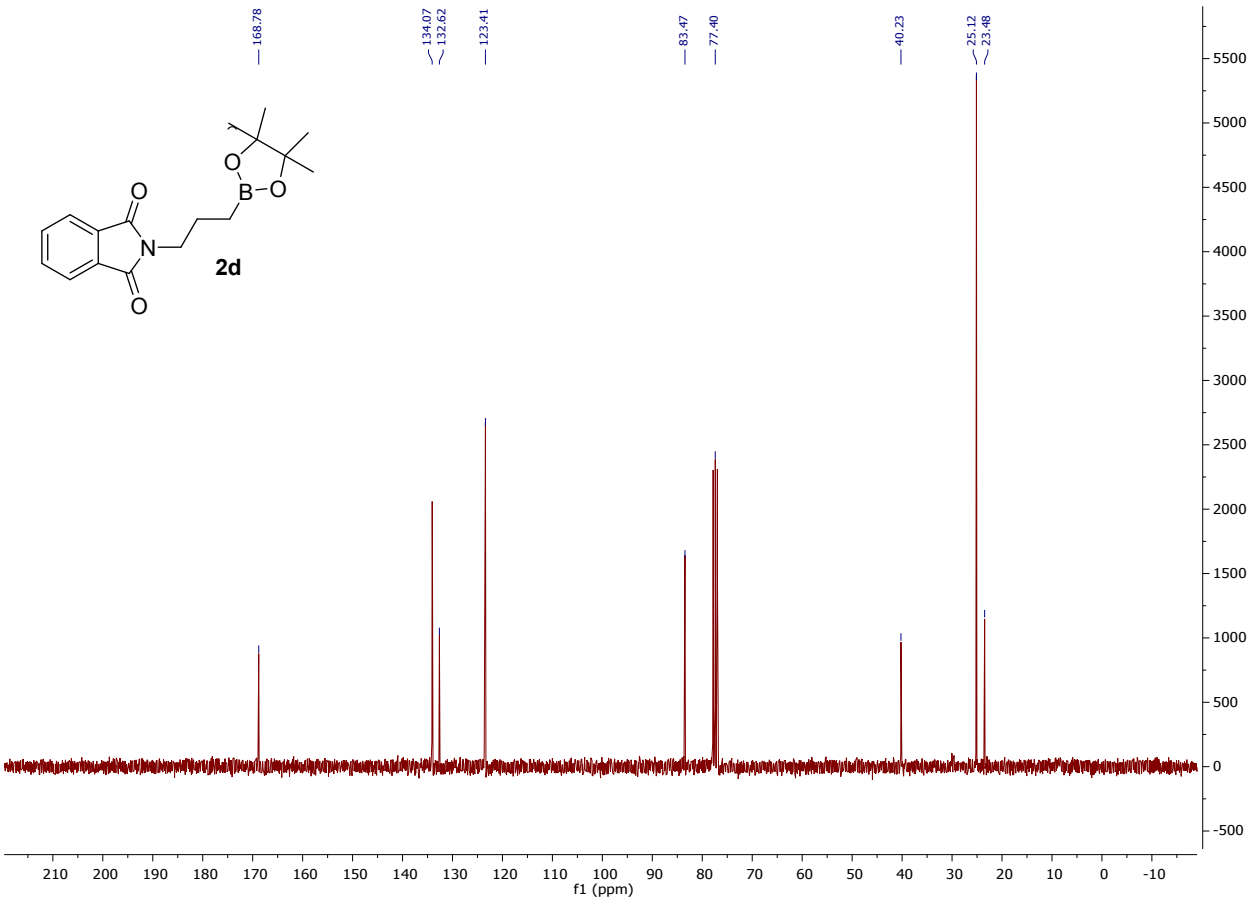
6. ^{1}H NMR and ^{13}C NMR spectra

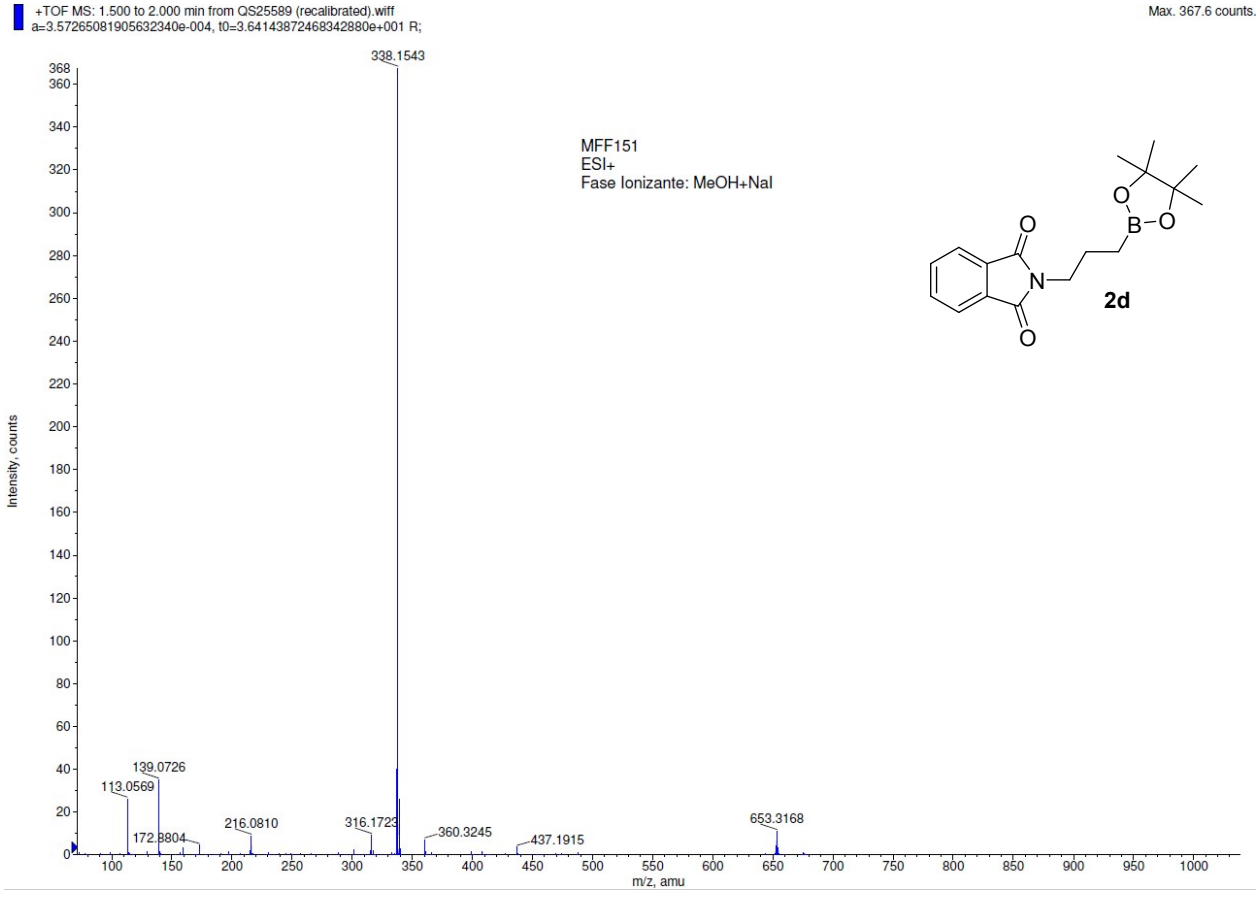





2c
7.30
7.28
7.27
7.26
7.21
7.18
7.15
2.66
2.63
2.60
1.81
1.78
1.75
1.73
1.70
1.26
0.88
0.85
0.82
2.02
2.93
2.00
2.07
12.09
2.18
f1 (ppm)


2d
7.83
7.82
7.81
7.80
7.69
7.68
7.68
7.67
7.27
3.70
3.67
3.65
1.83
1.81
1.78
1.76
1.73
1.20
0.83
0.80
0.78
1.00
1.00
0.99
1.06
6.00
1.00
f1 (ppm)


168.78
134.07
132.62
123.41
83.47
77.40
40.23
25.12
23.48
2d
f1 (ppm)


+TOF MS: 1.500 to 2.000 min from QS25589 (recalibrated).wiff
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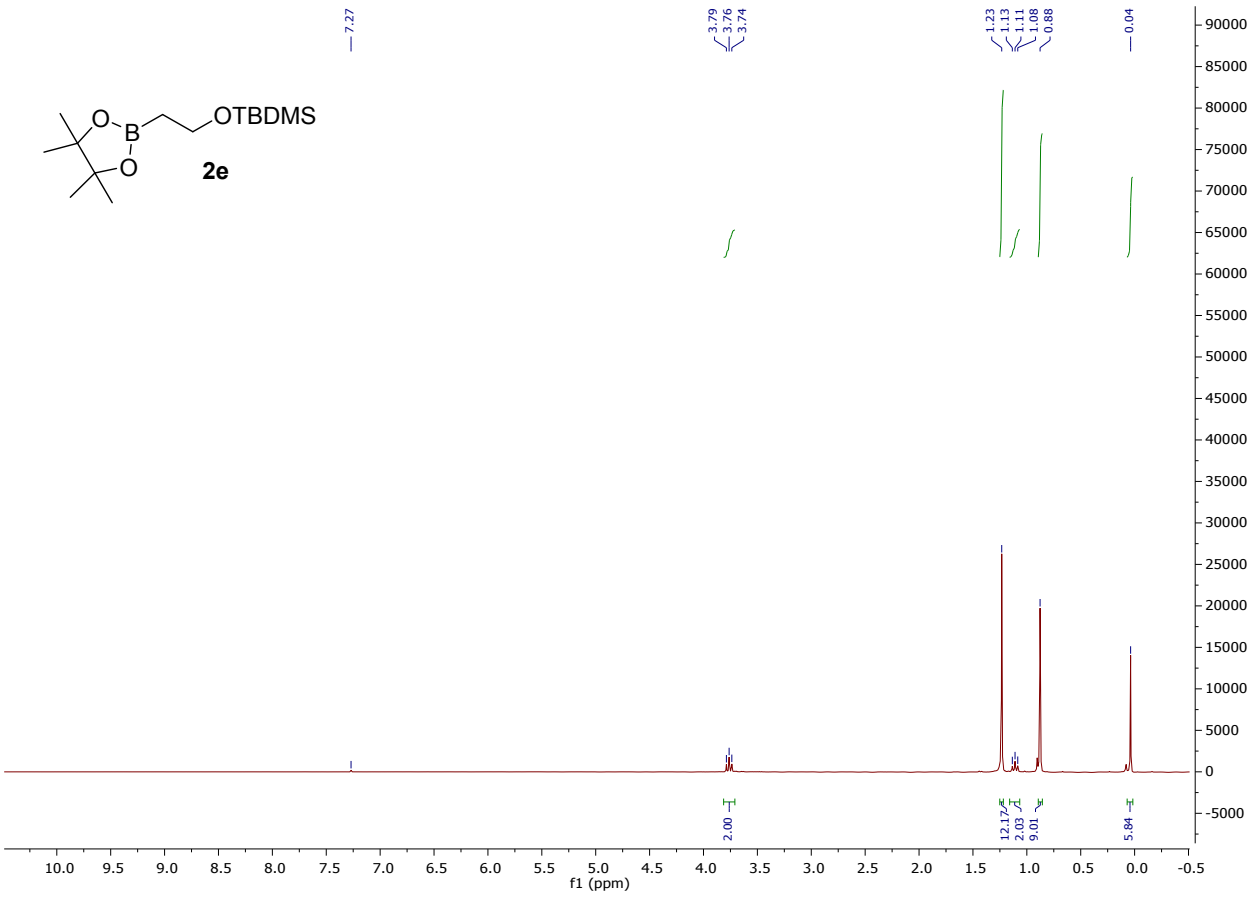
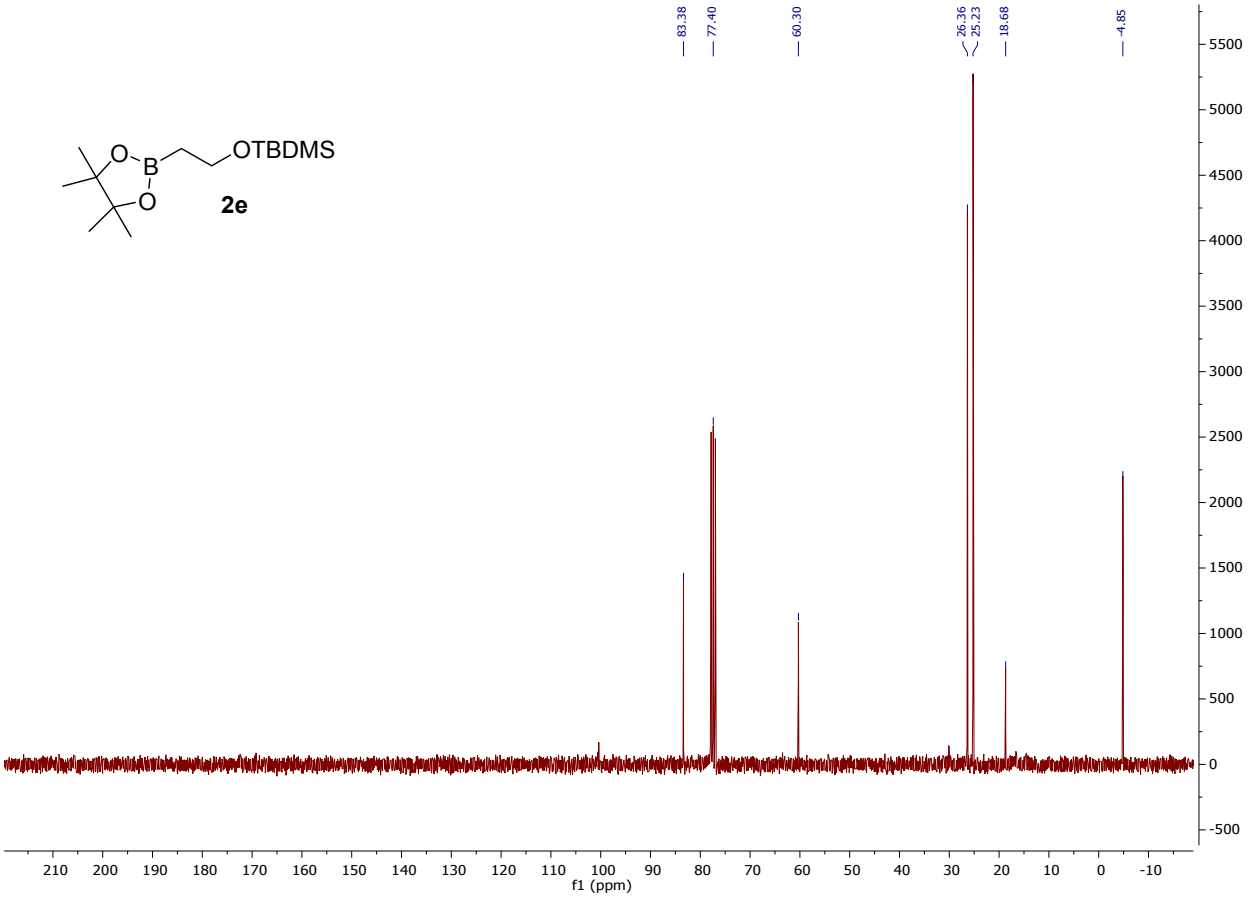
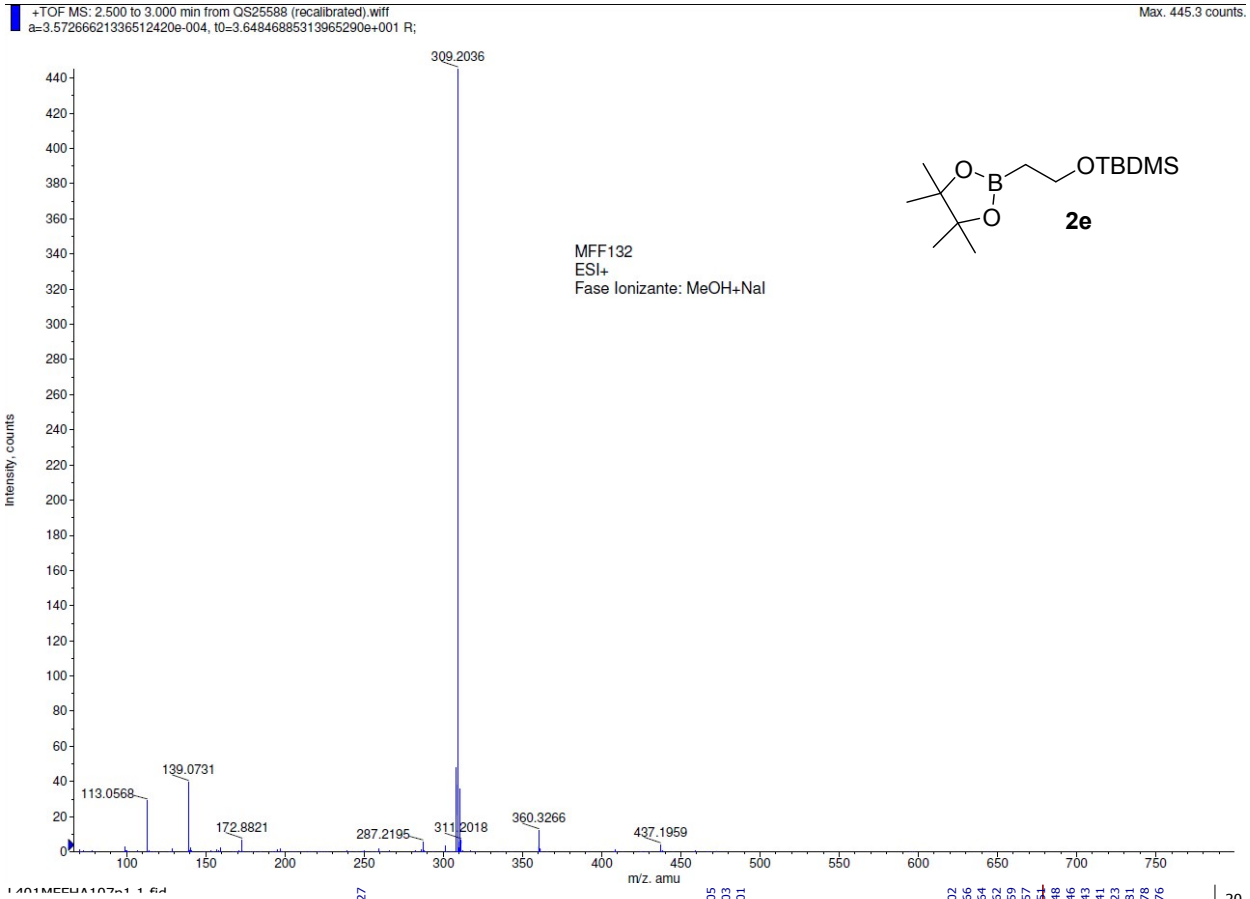
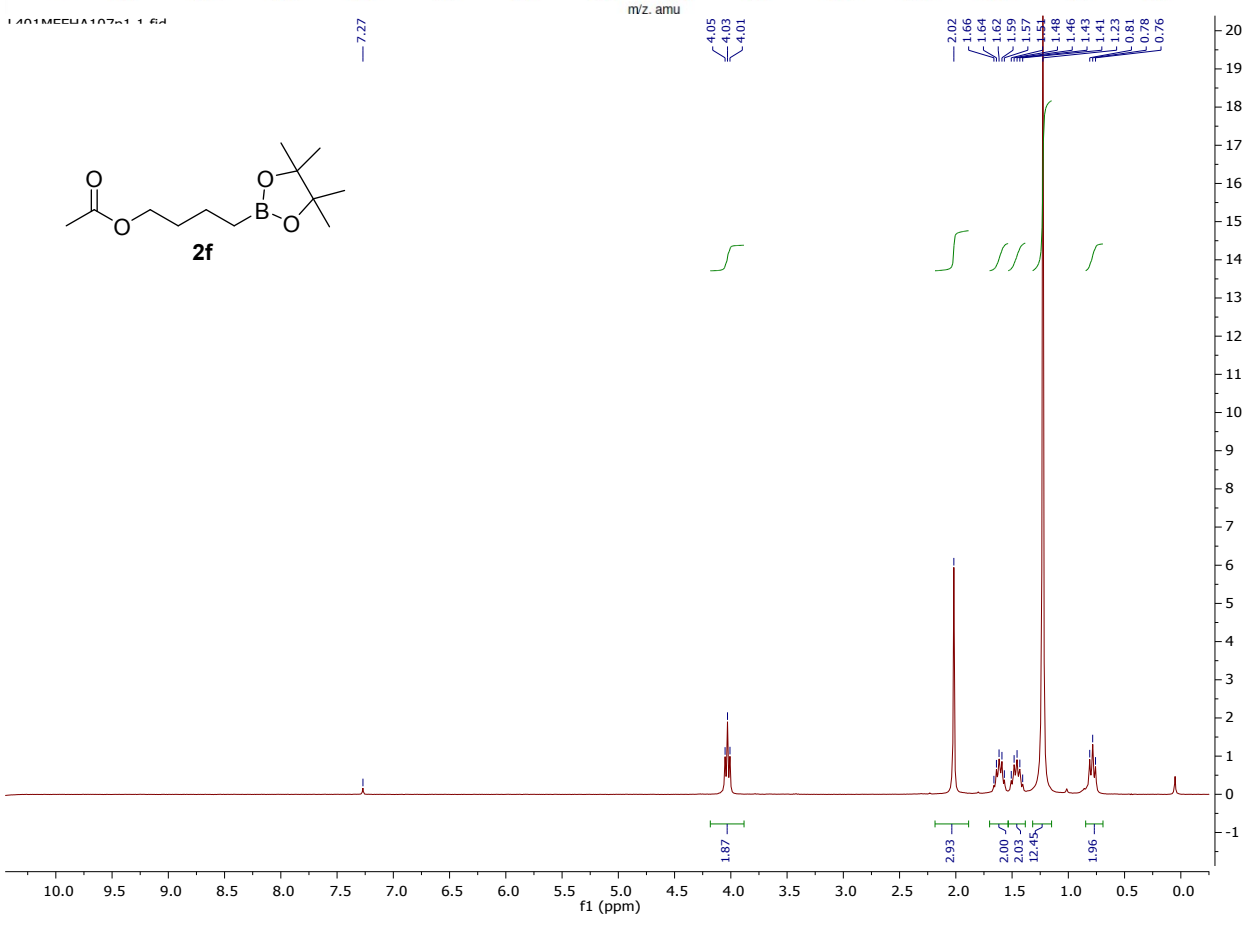
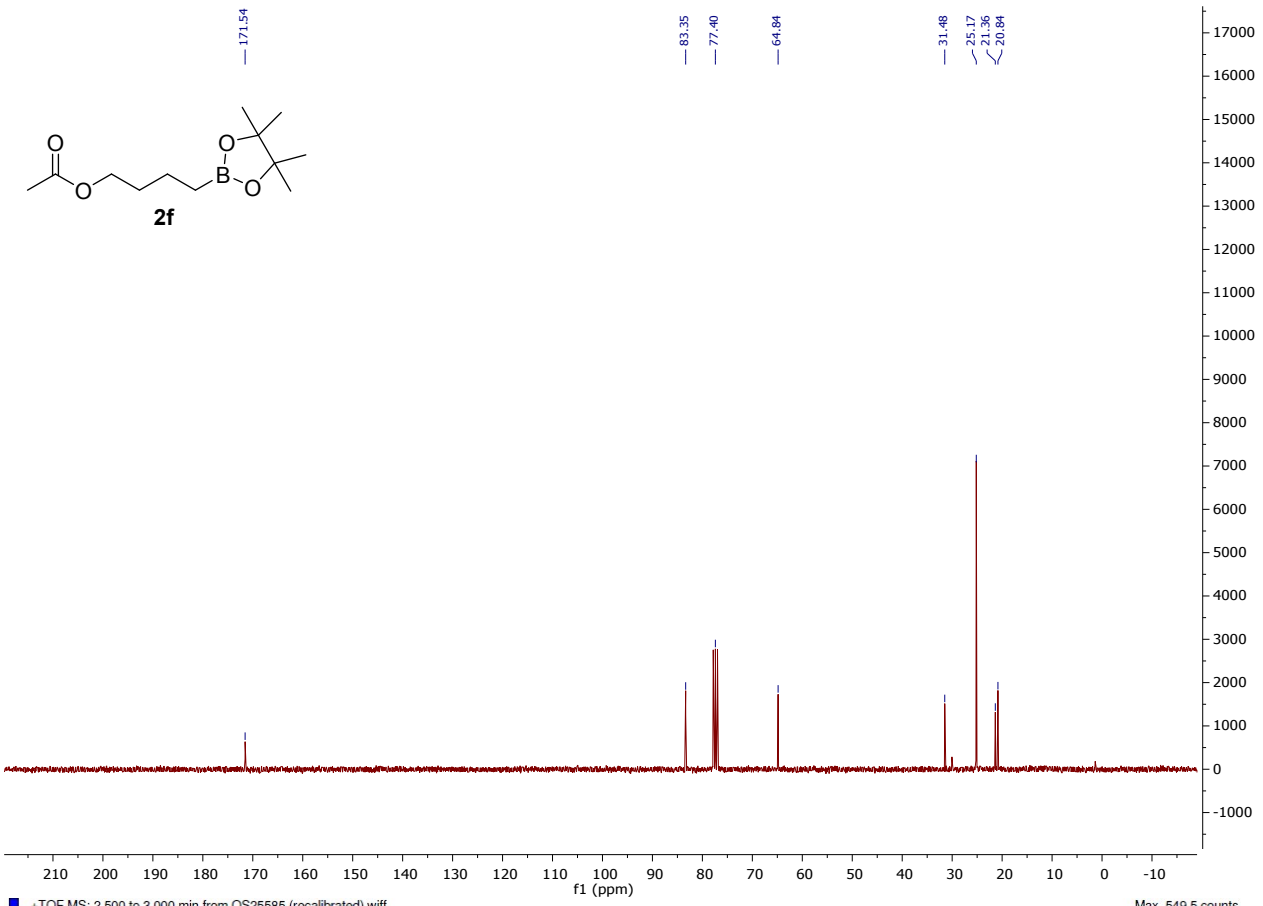
Max. 367.6 counts.
338.1543
MFF151
ESI+
Fase Ionizante: MeOH+NaI
2d
Intensity, counts
m/z, amu
139.0726
113.0569
172.3804
216.0810
316.1723
360.3245
437.1915
653.3168

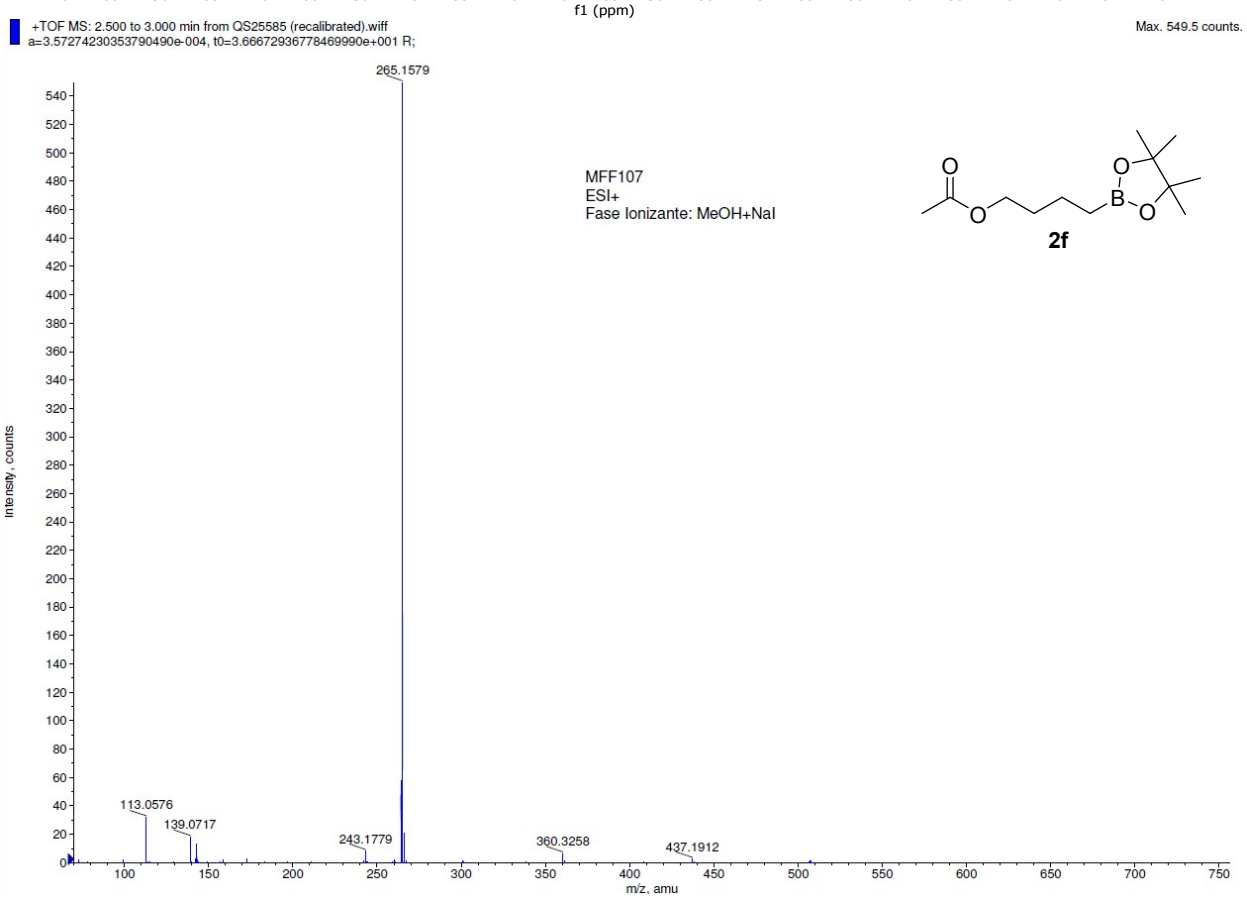

OTBDMS
2e
7.27
3.79
3.76
3.74
1.23
1.13
1.11
1.08
0.88
0.04
2.00
12.17
2.03
9.01
5.84
f1 (ppm)

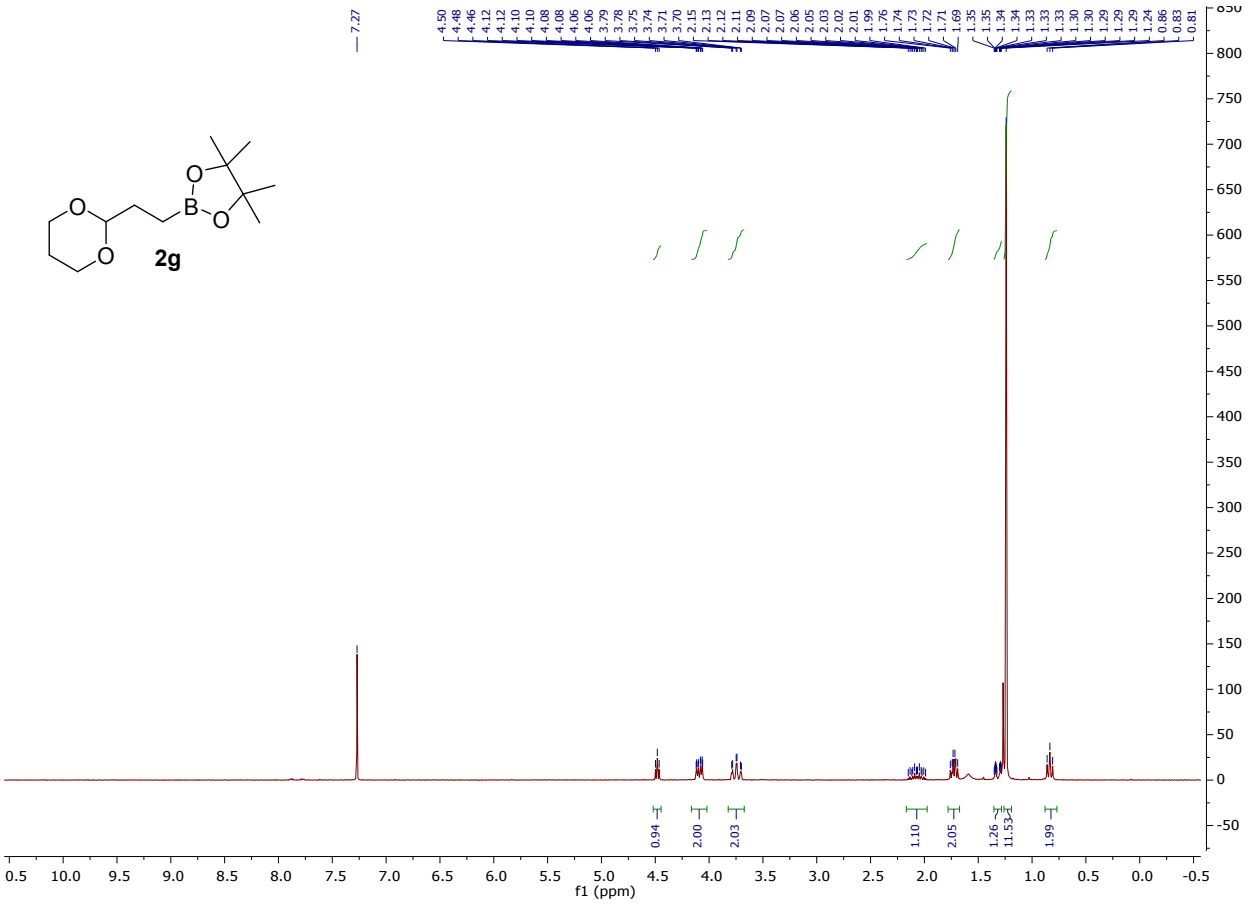

OTBDMS
2e
83.38
77.40
60.30
26.36
25.23
18.68
-4.85
f1 (ppm)

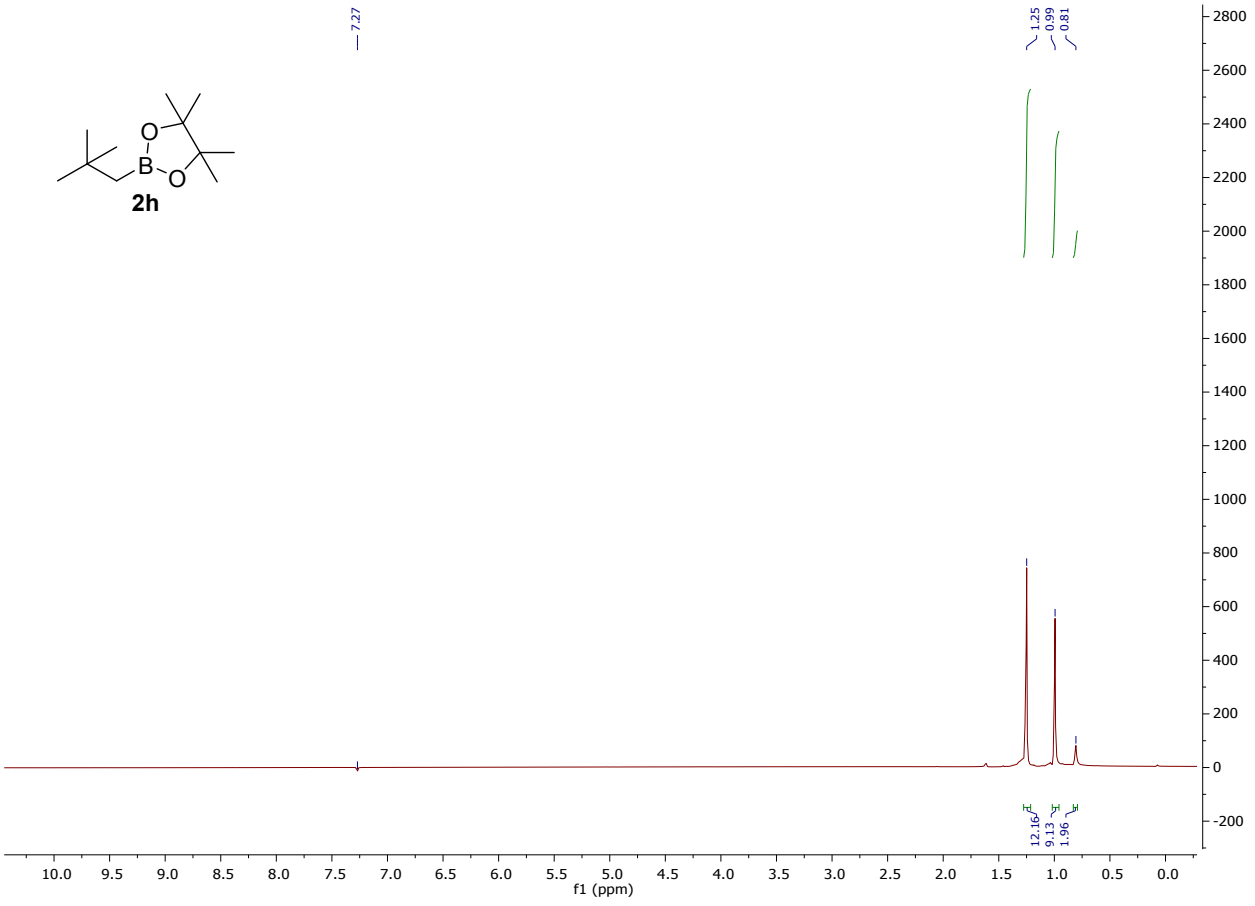

+TOF MS: 2.500 to 3.000 min from QS25588 (recalibrated).wiff
a=3.57266621336512420e-004, t0=3.64846885313965290e+001 R;
Max. 445.3 counts.
309.2036
139.0731
113.0568
172.8821
287.2195
311.2018
360.3266
437.1959
Intensity, counts
m/z, amu
MFF132
ESI+
Fase Ionizante: MeOH+NaI
OTBDMS
2e

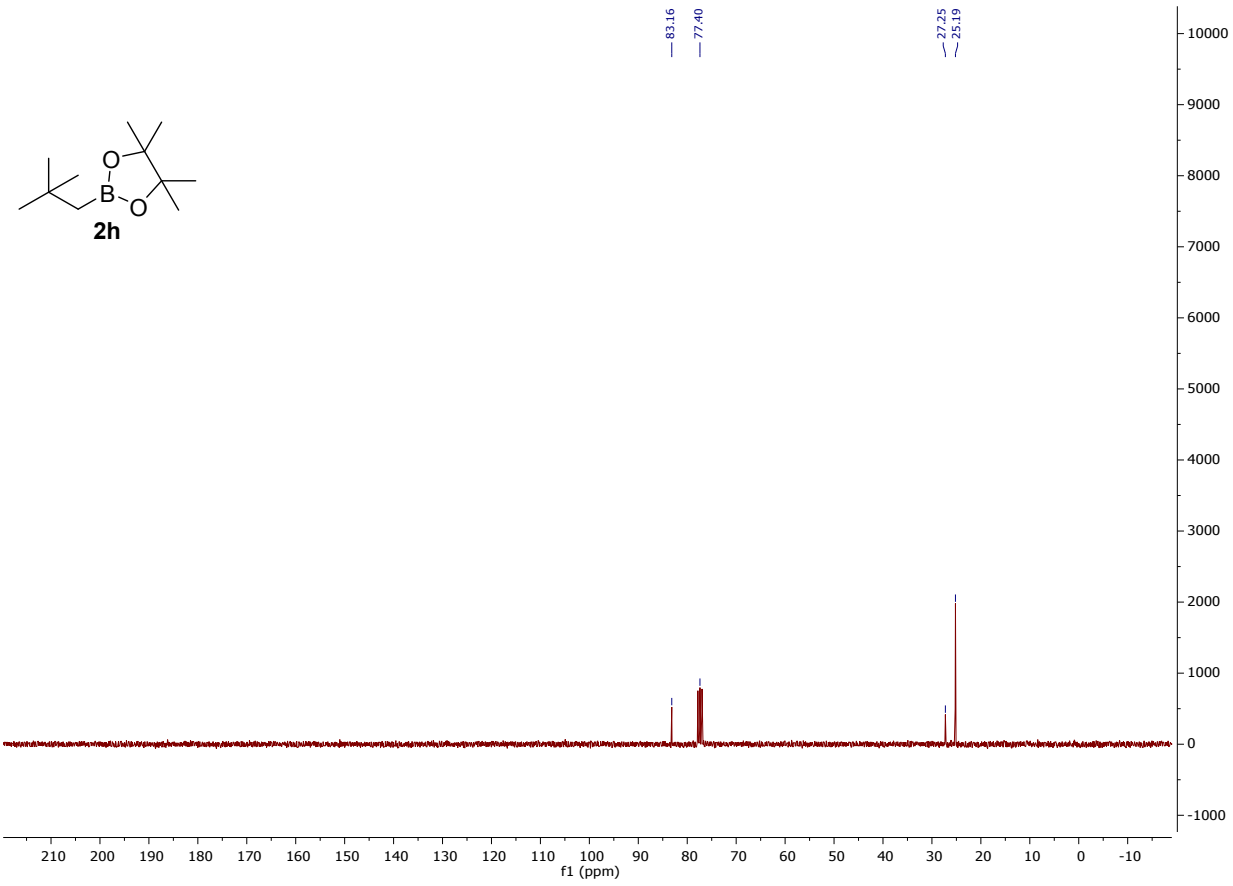

7.27
4.05
4.03
4.01
2.02
1.66
1.64
1.62
1.59
1.57
1.54
1.48
1.46
1.43
1.41
1.23
0.81
0.78
0.76
1.87
2.93
2.00
2.03
12.45
1.96
f1 (ppm)
2f

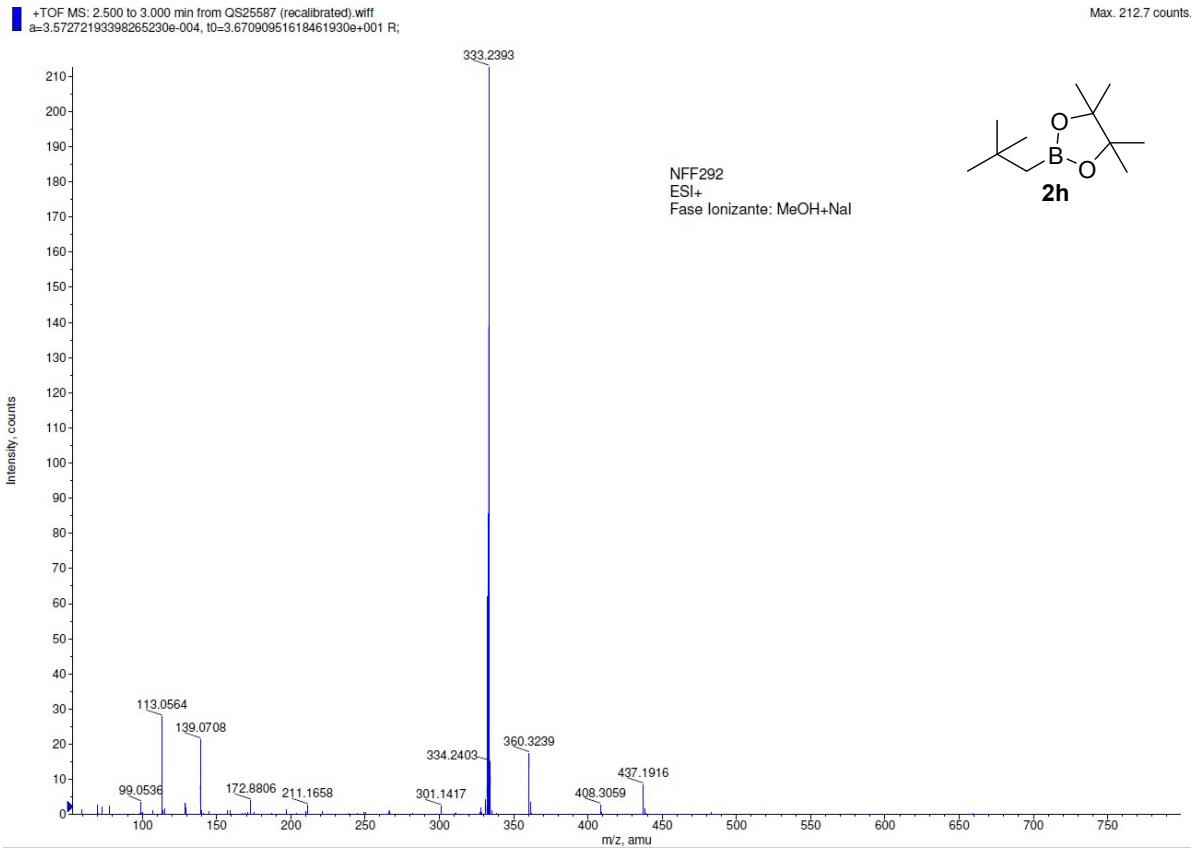

171.54
83.35
77.40
64.84
31.48
25.17
21.36
20.84
2f
f1 (ppm)

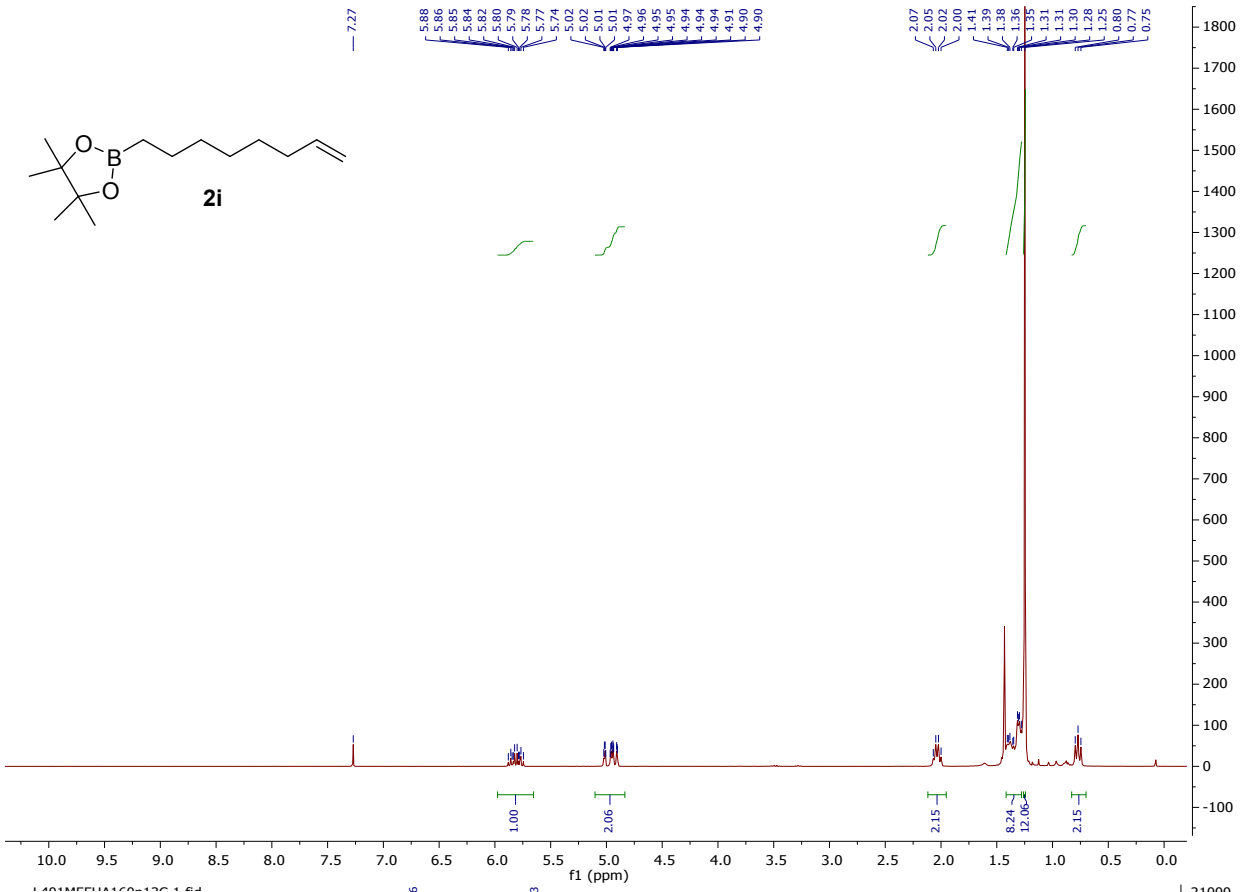

+TOF MS: 2.500 to 3.000 min from QS25585 (recalibrated).wiff
a=3.57274230353790490e-004, t0=3.66672936778469990e+001 R;
Max. 549.5 counts.
265.1579
MFF107
ESI+
Fase Ionizante: MeOH+NaI
2f
Intensity, counts
113.0576
139.0717
243.1779
360.3258
437.1912
m/z, amu

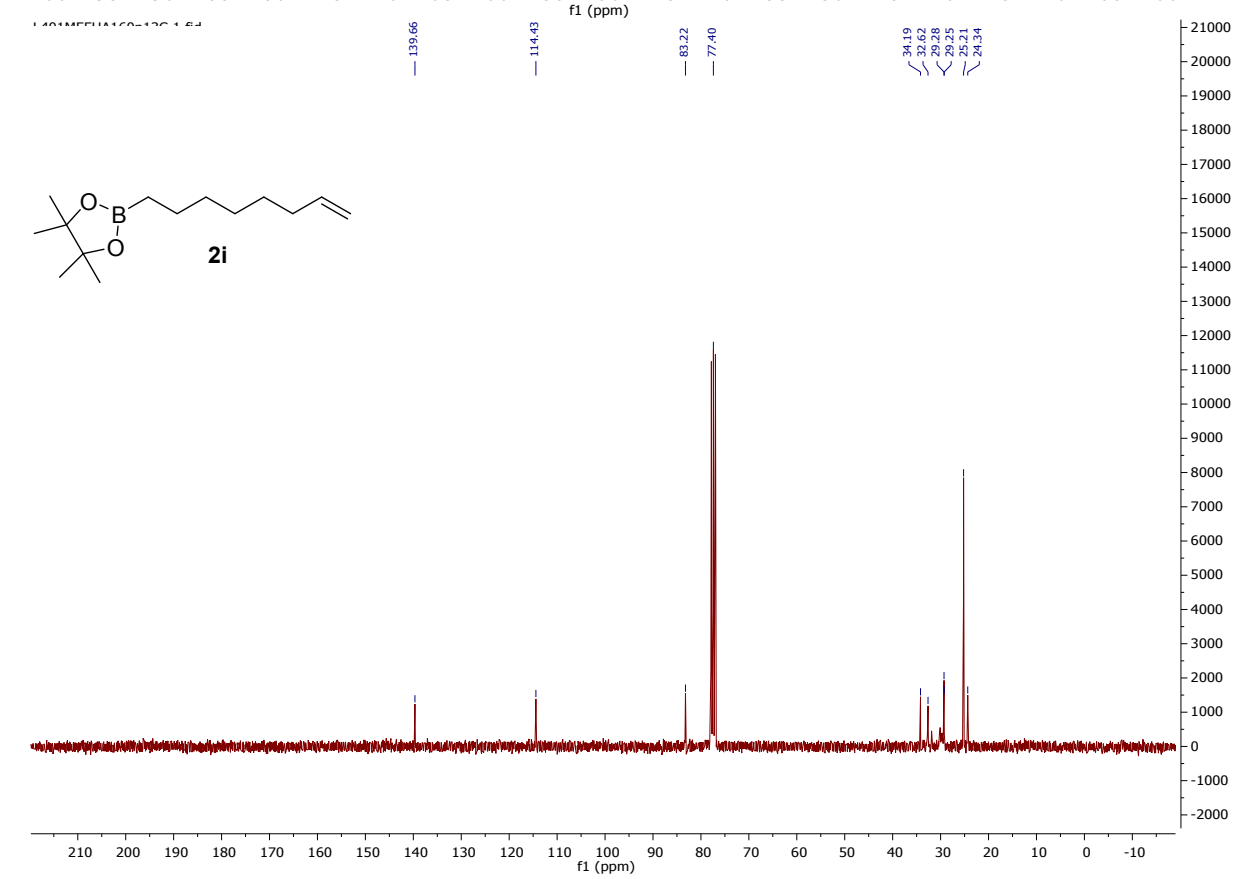

2g
f1 (ppm)

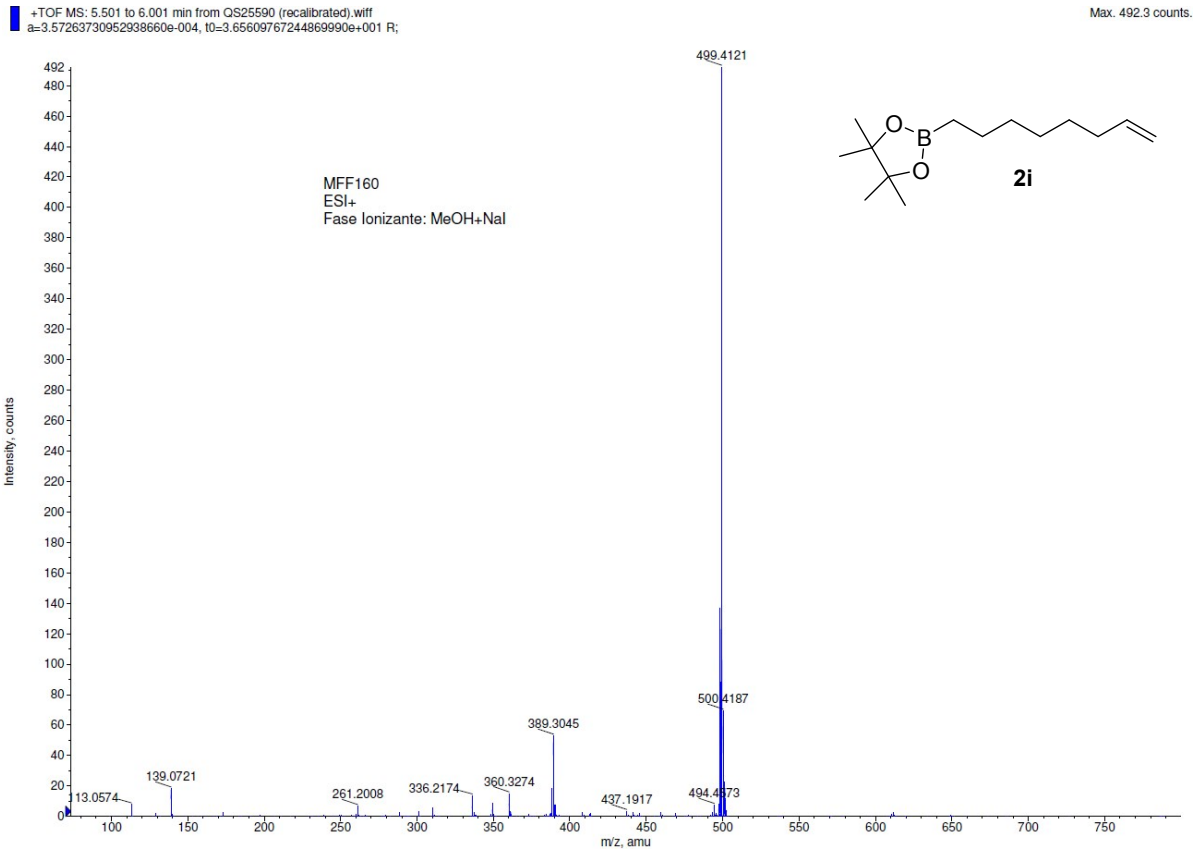

2h
f1 (ppm)


2h
83.16
77.40
27.25
25.19
f1 (ppm)

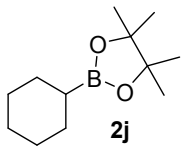

+TOF MS: 2.500 to 3.000 min from QS25587 (recalibrated).wiff
a=3.57272193398265230e-004, t0=3.67090951618461930e+001 R;
Max. 212.7 counts.
333.2393
NFF292
ESI+
Fase Ionizante: MeOH+NaI
2h
Intensity, counts
m/z, amu
113.0564
139.0708
99.0536
172.8806
211.1658
301.1417
334.2403
360.3239
408.3059
437.1916


7.27
5.88
5.86
5.85
5.84
5.82
5.80
5.79
5.78
5.77
5.74
5.02
5.02
5.01
5.01
4.97
4.96
4.95
4.95
4.94
4.94
4.94
4.91
4.90
4.90
2.07
2.05
2.02
2.00
1.41
1.39
1.38
1.36
1.35
1.31
1.31
1.30
1.28
1.25
0.80
0.77
0.75
1.00
2.06
2.15
8.24
12.06
2.15
f1 (ppm)
2i

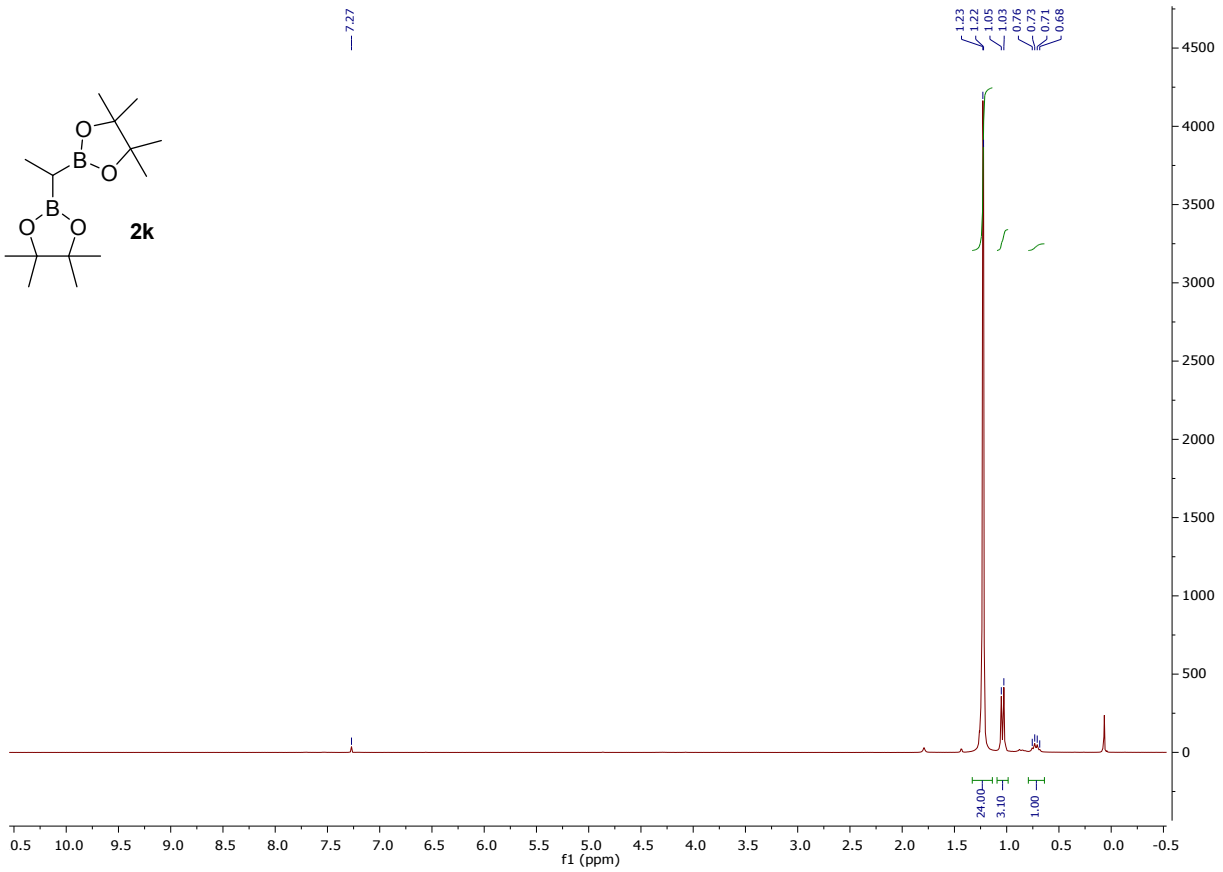

139.66
114.43
83.22
77.40
34.19
32.62
29.28
29.25
25.21
24.34
f1 (ppm)
2i

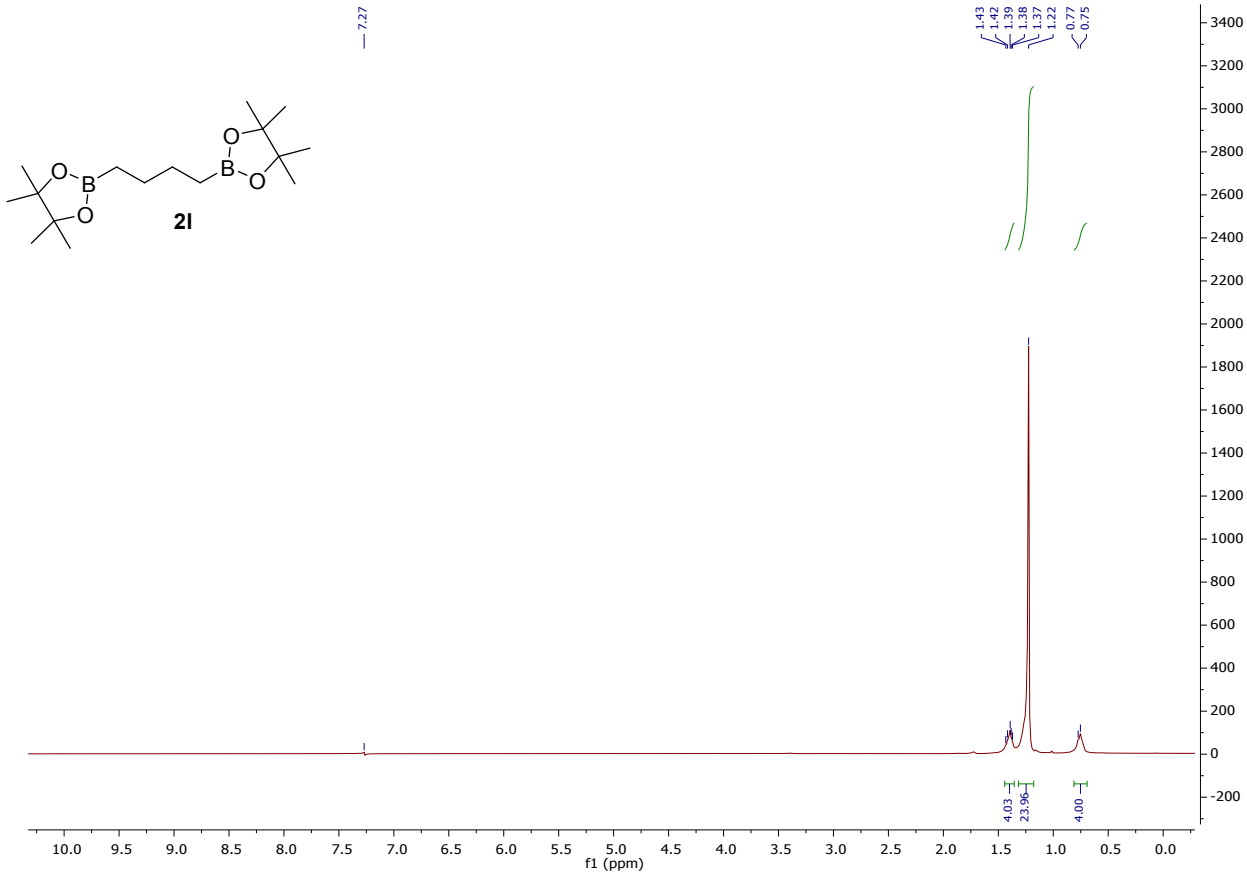

+TOF MS: 5.501 to 6.001 min from QS25590 (recalibrated).wiff
a=3.57263730952938660e-004, t0=3.65609767244869990e+001 R;
Max. 492.3 counts.
MFF160
ESI+
Fase Ionizante: MeOH+NaI
499.4121
500.4187
389.3045
139.0721
113.0574
261.2008
336.2174
360.3274
437.1917
494.4273
Intensity, counts
m/z, amu

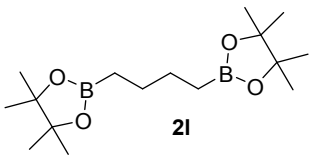
2i


2j

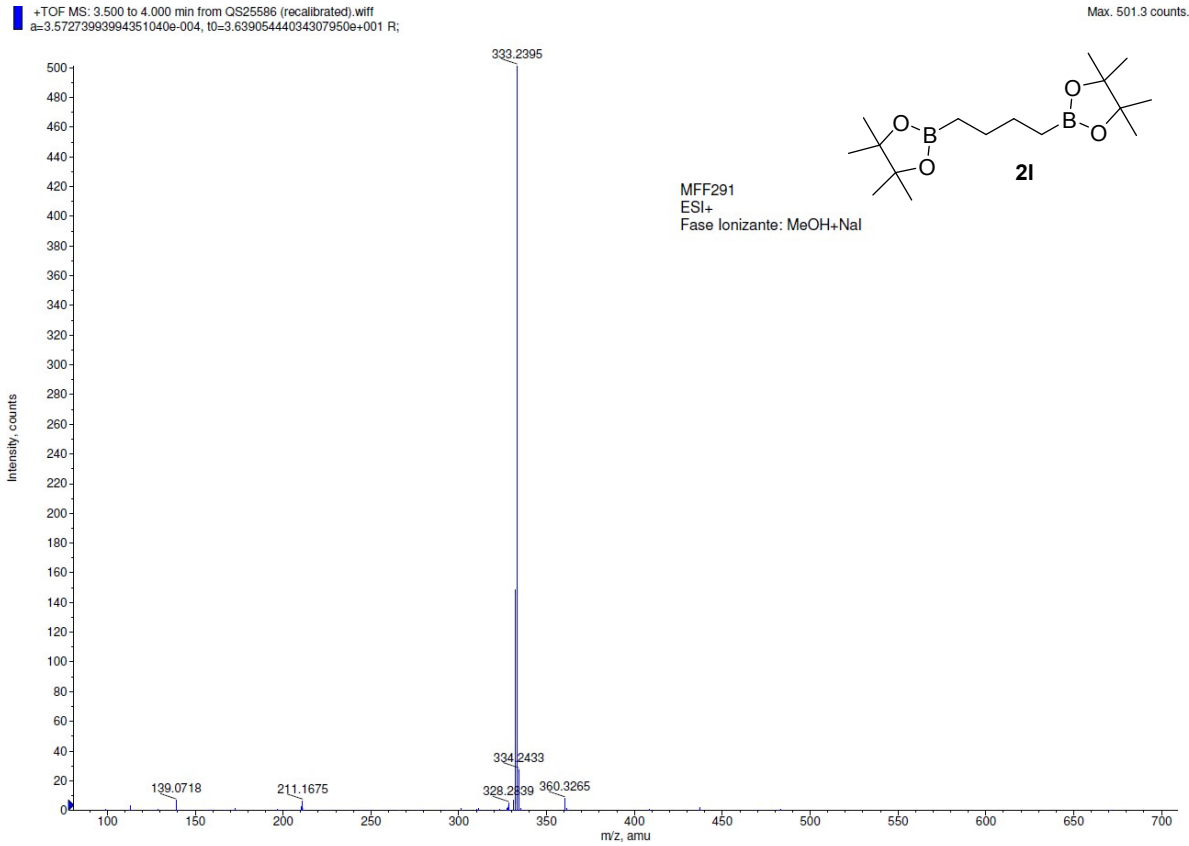
7.27
1.67
1.64
1.61
1.60
1.58
1.39
1.35
1.31
1.28
1.23
0.99
0.97
5.00
4.93
11.98
1.04
f1 (ppm)


7.27
1.23
1.22
1.05
1.03
0.76
0.73
0.71
0.68
2k
24.00
3.10
1.00
f1 (ppm)


7.27
1.43
1.42
1.39
1.38
1.37
1.22
0.77
0.75
2l
4.03
23.96
4.00
f1 (ppm)

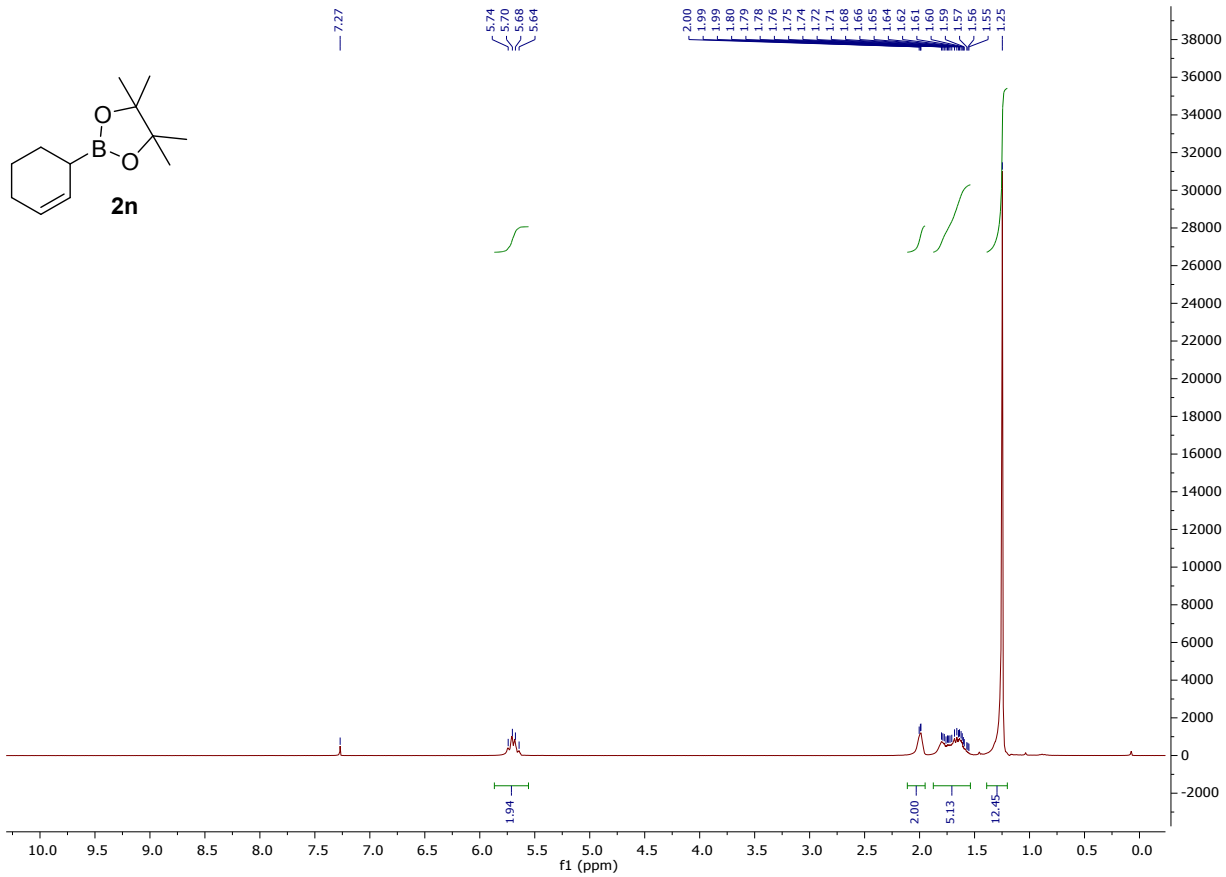


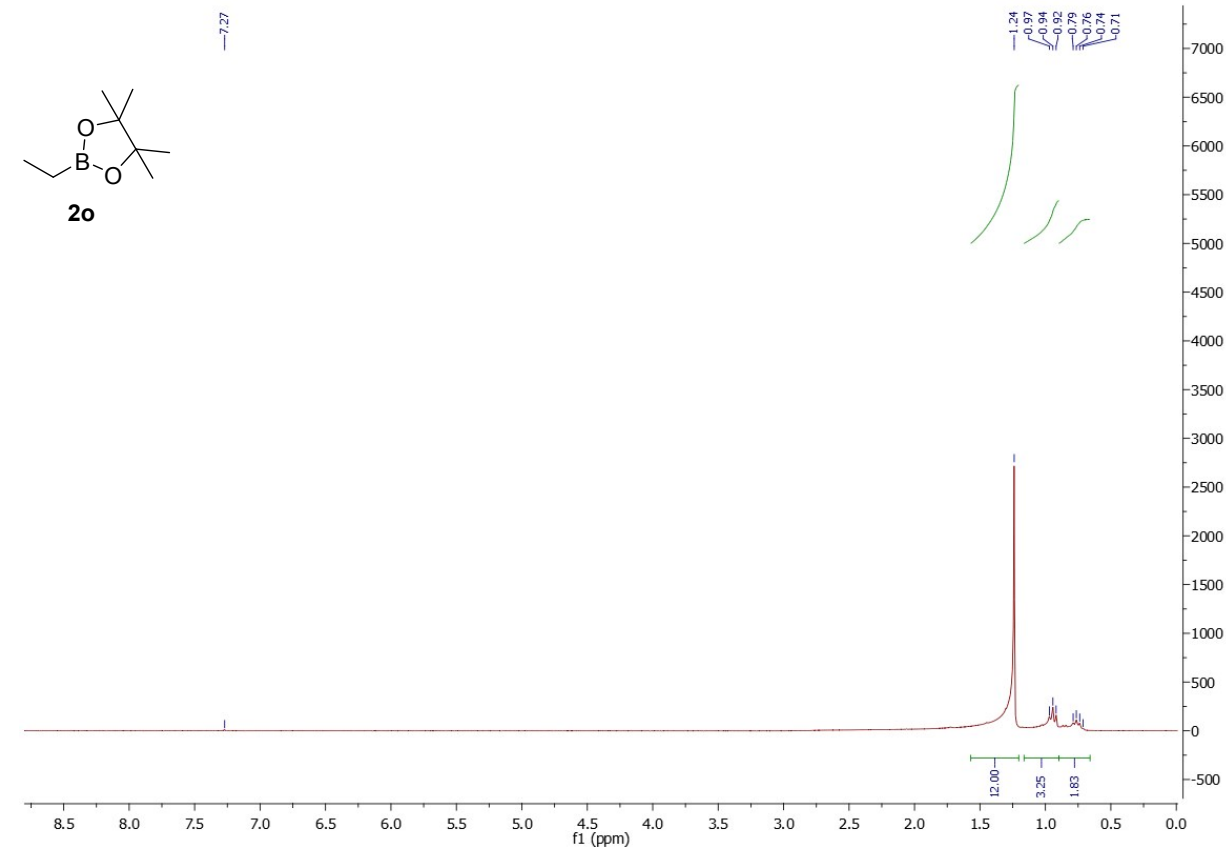
2l

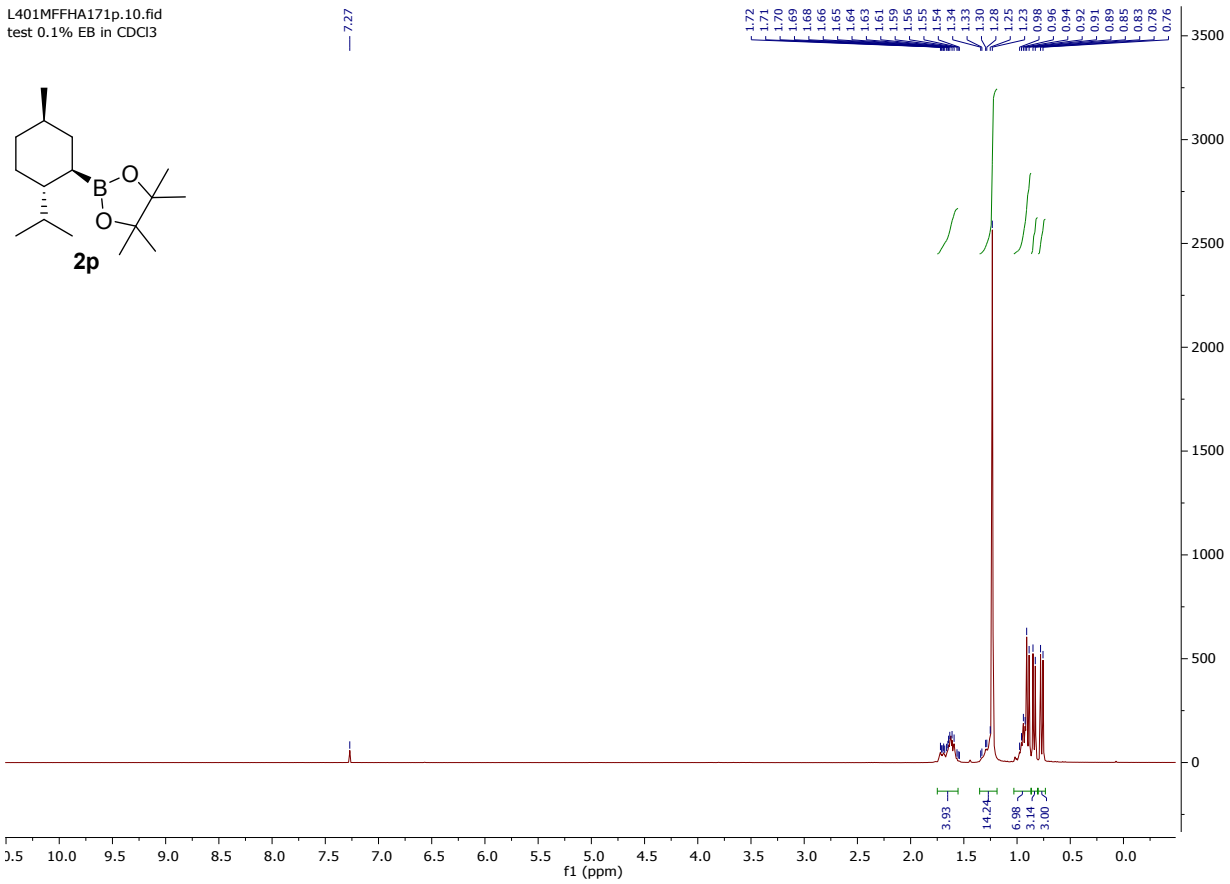


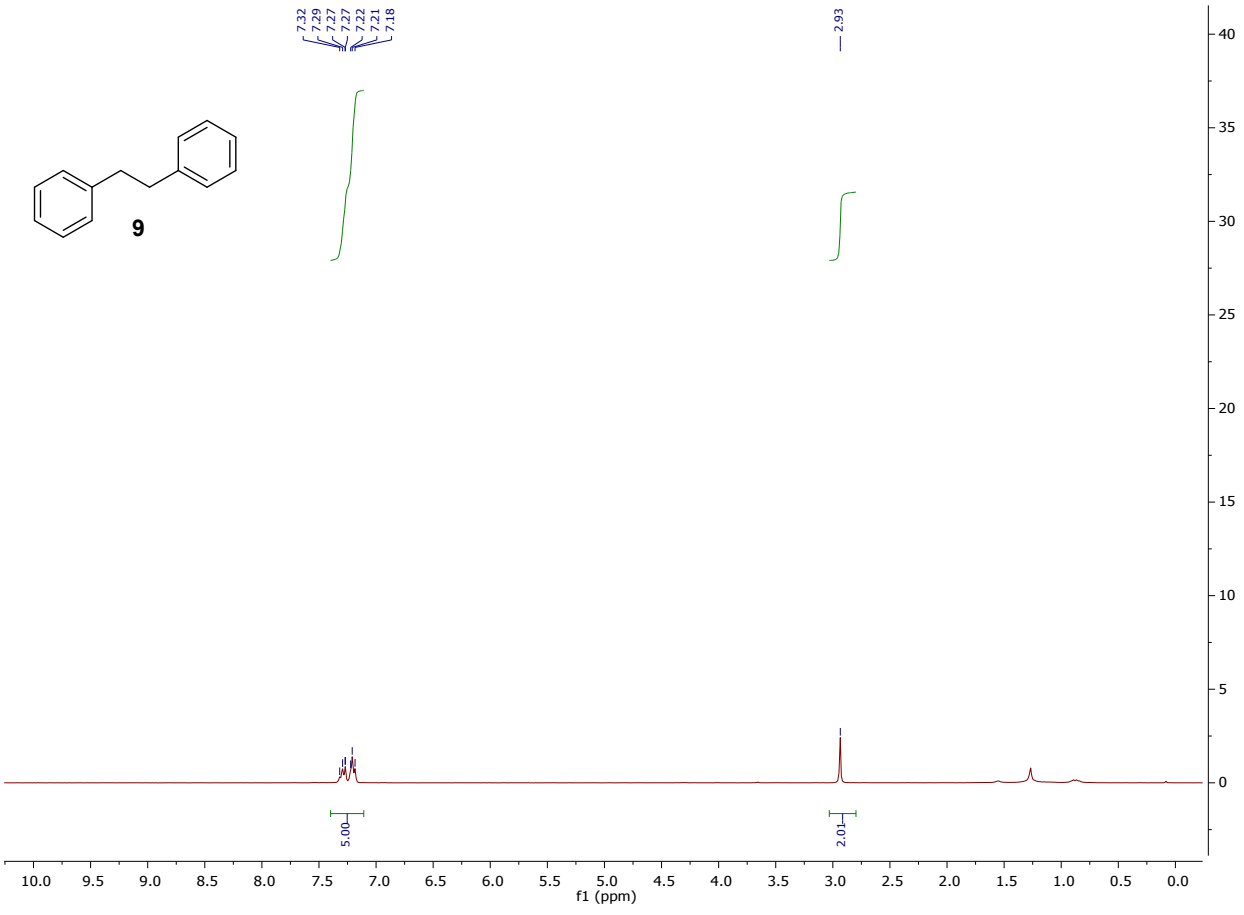
2l

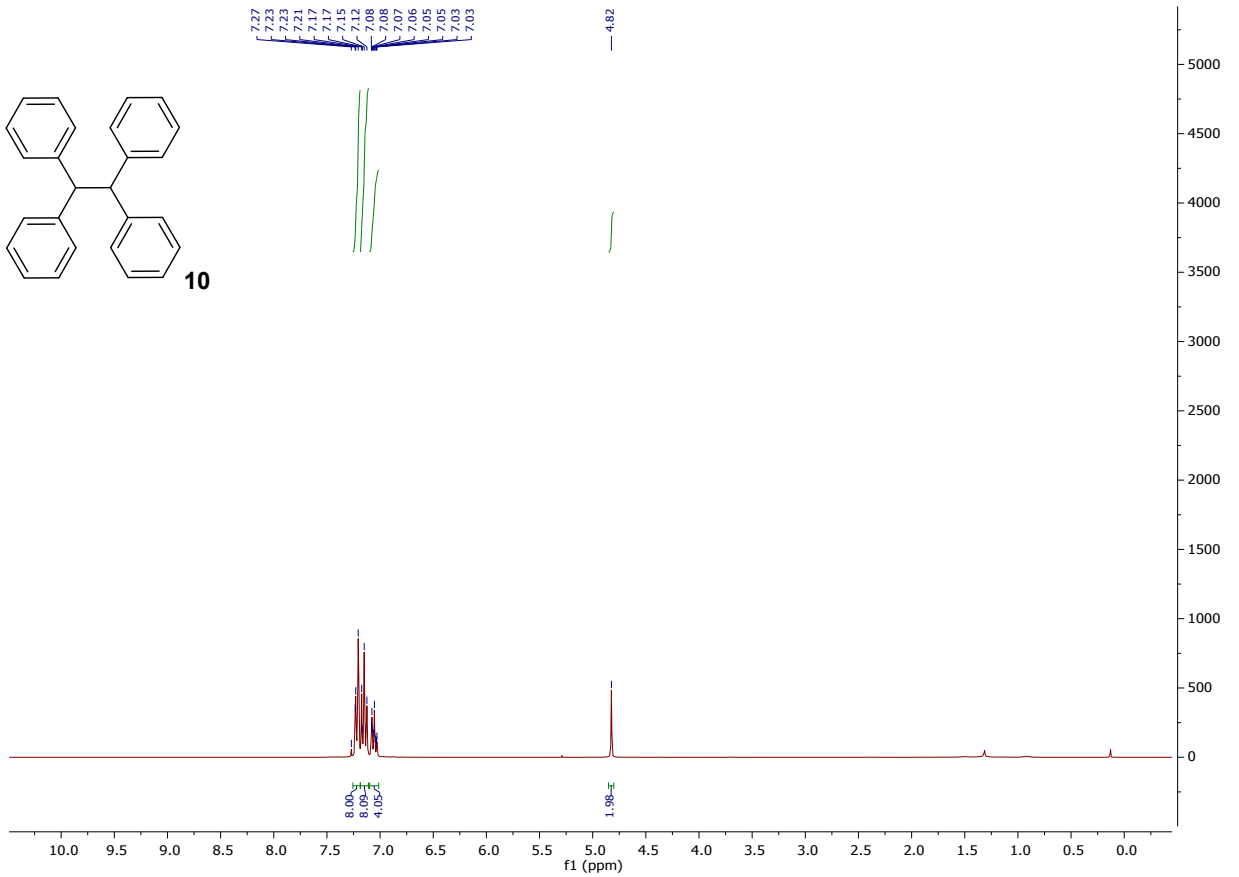
MFF291
ESI+
Fase Ionizante: MeOH+NaI

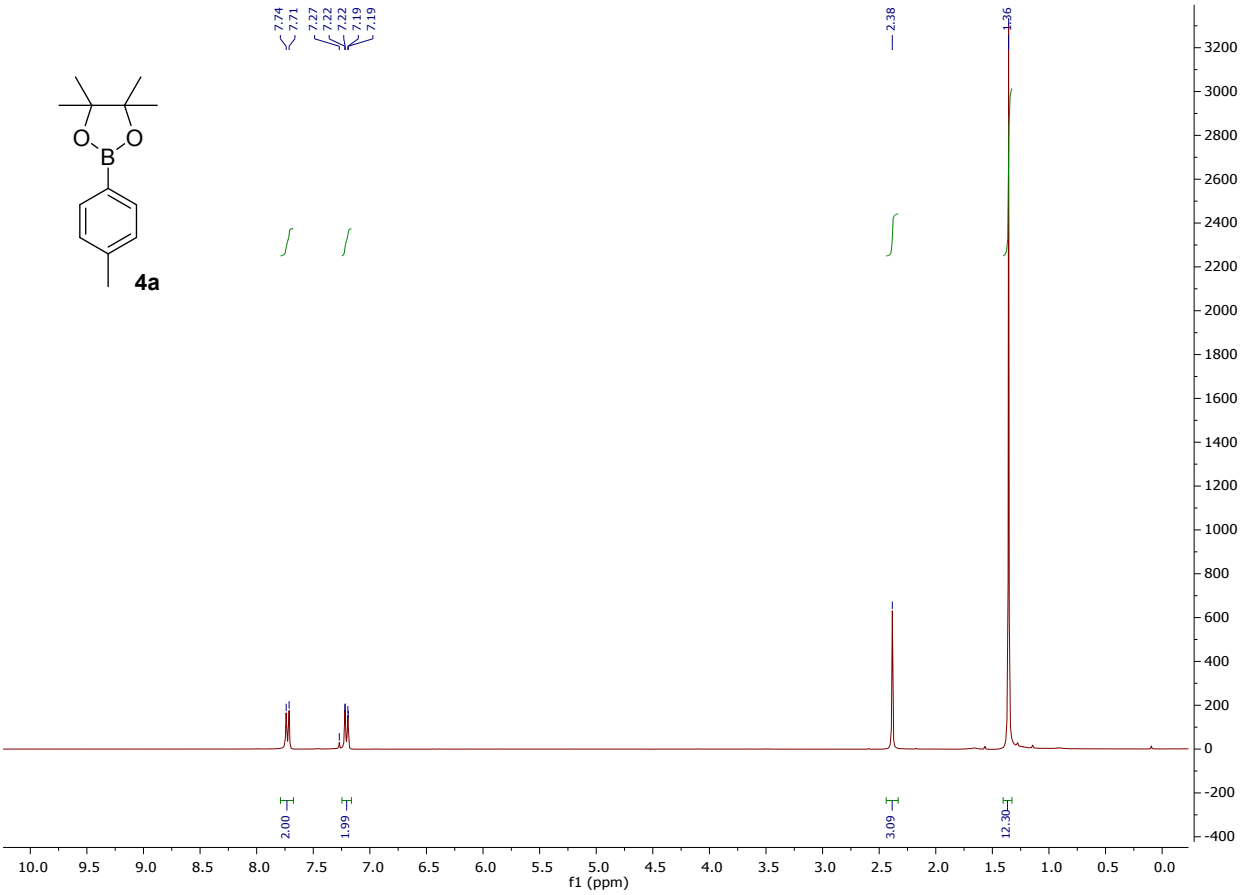

2n
7.27
5.74
5.70
5.68
5.64
2.00
1.99
1.99
1.80
1.79
1.78
1.76
1.75
1.74
1.72
1.71
1.68
1.66
1.65
1.64
1.62
1.61
1.60
1.59
1.57
1.56
1.55
1.25
1.94
2.00
5.13
12.45
f1 (ppm)

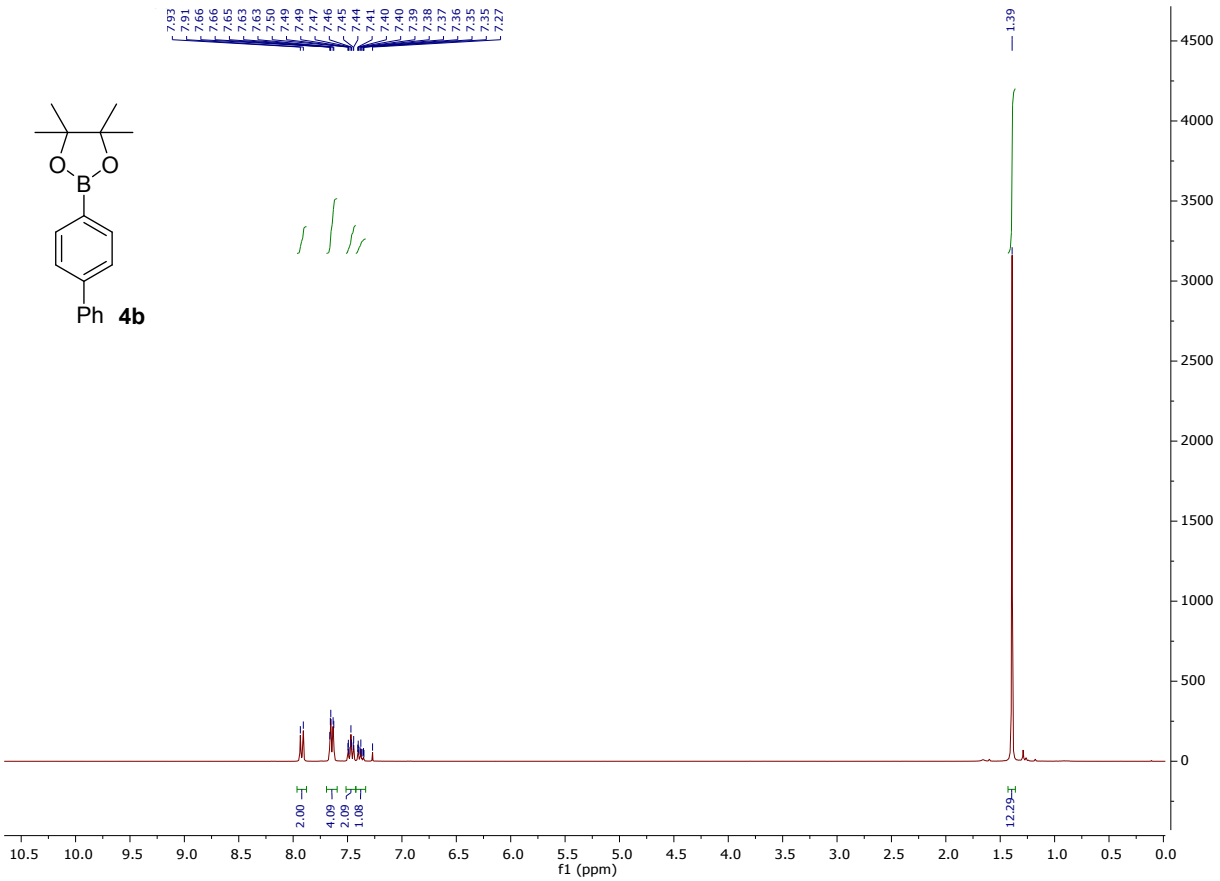

2o
7.27
1.24
0.97
0.94
0.92
0.79
0.76
0.74
0.71
12.00
3.25
1.83
f1 (ppm)

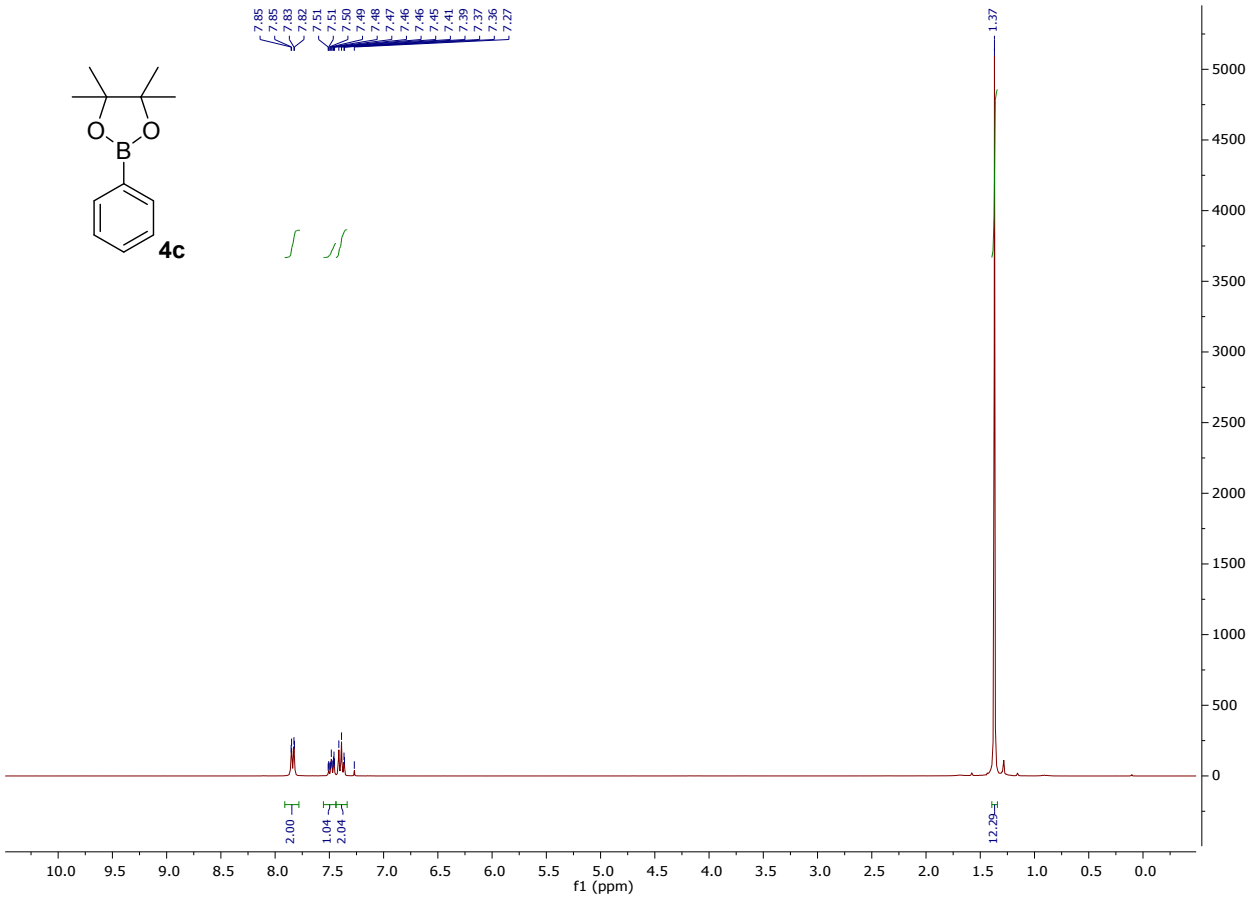

L401MFFHA171p.10.fid
test 0.1% EB in CDCl3
2p
f1 (ppm)

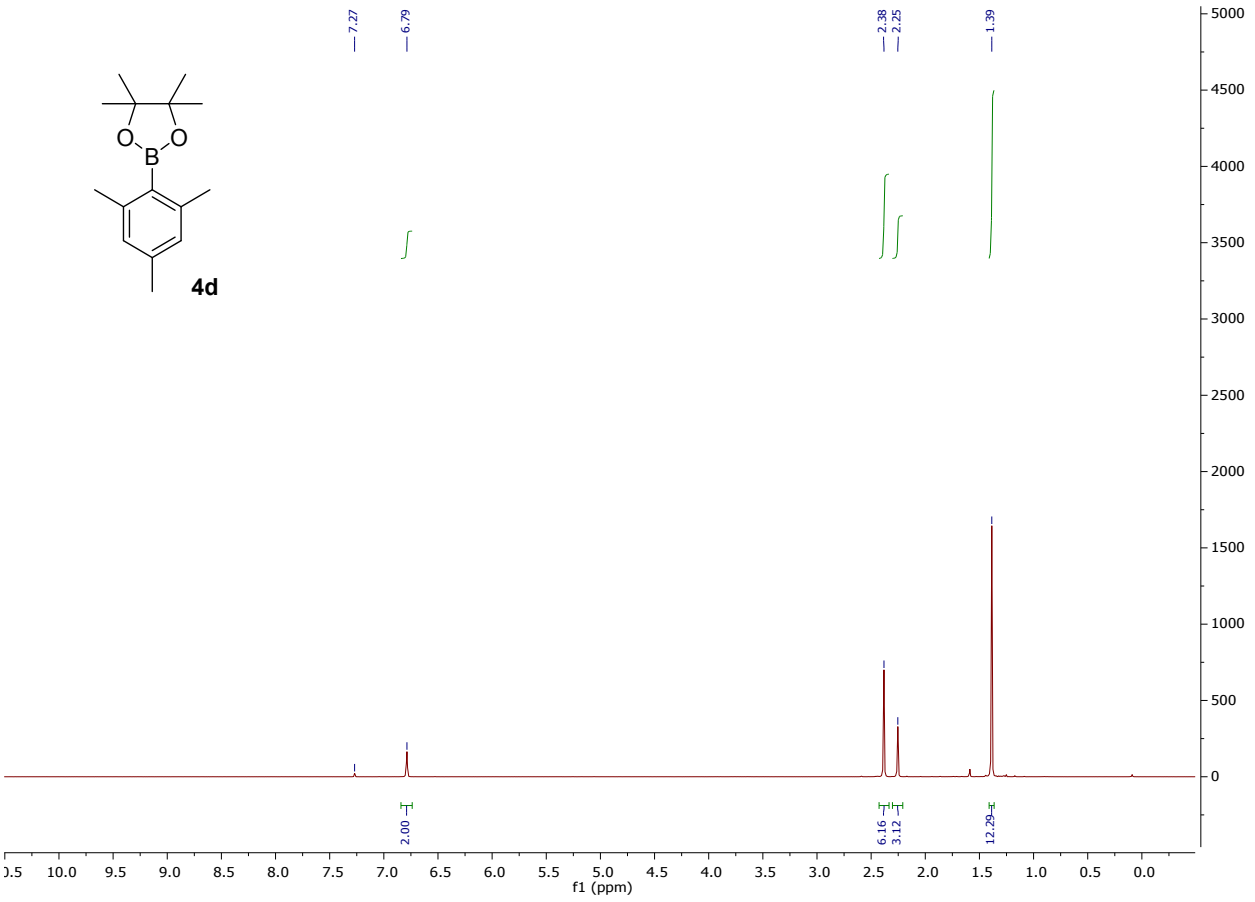

9
f1 (ppm)

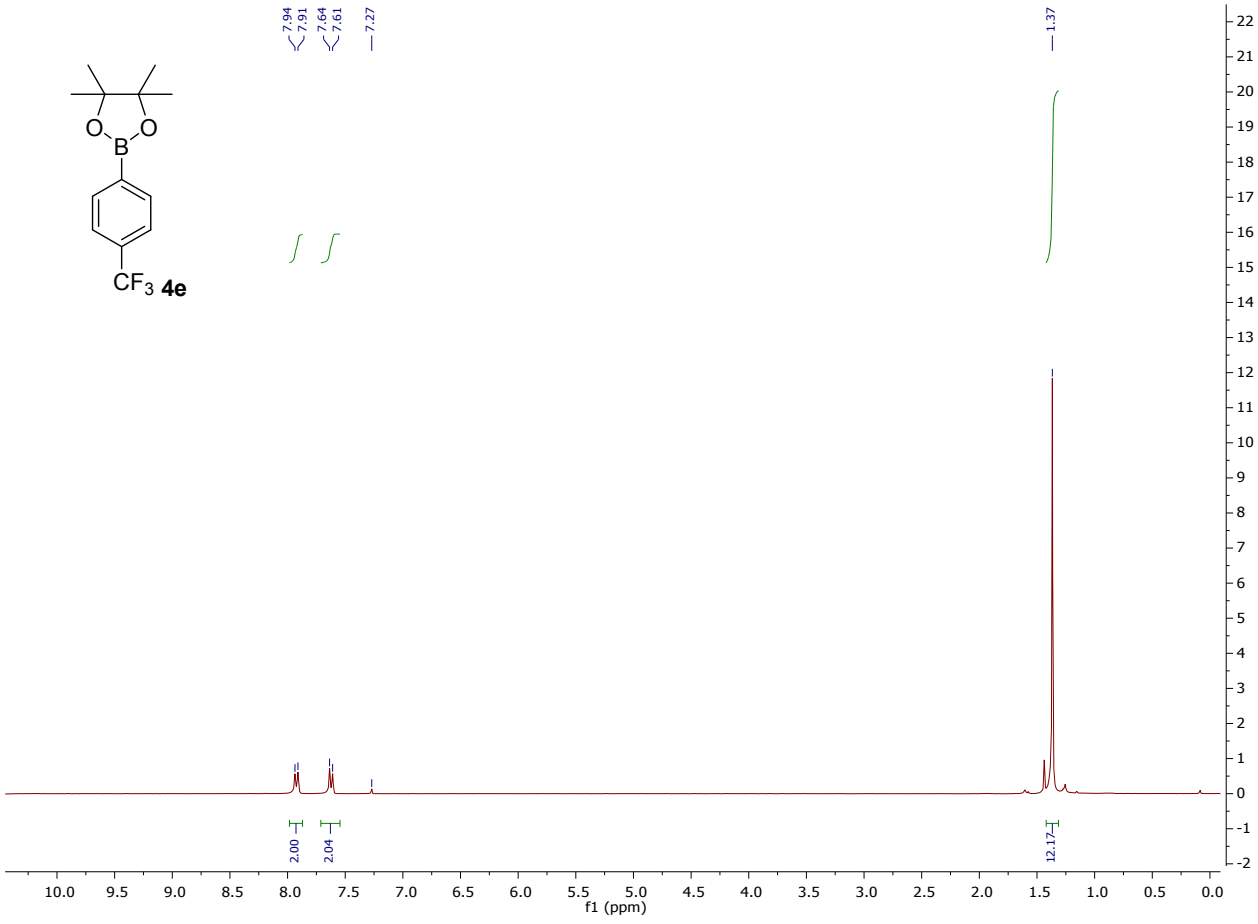

7.27
7.23
7.23
7.21
7.17
7.17
7.15
7.12
7.08
7.08
7.07
7.06
7.05
7.05
7.03
7.03
4.82
10
8.00
8.09
4.05
1.98
f1 (ppm)

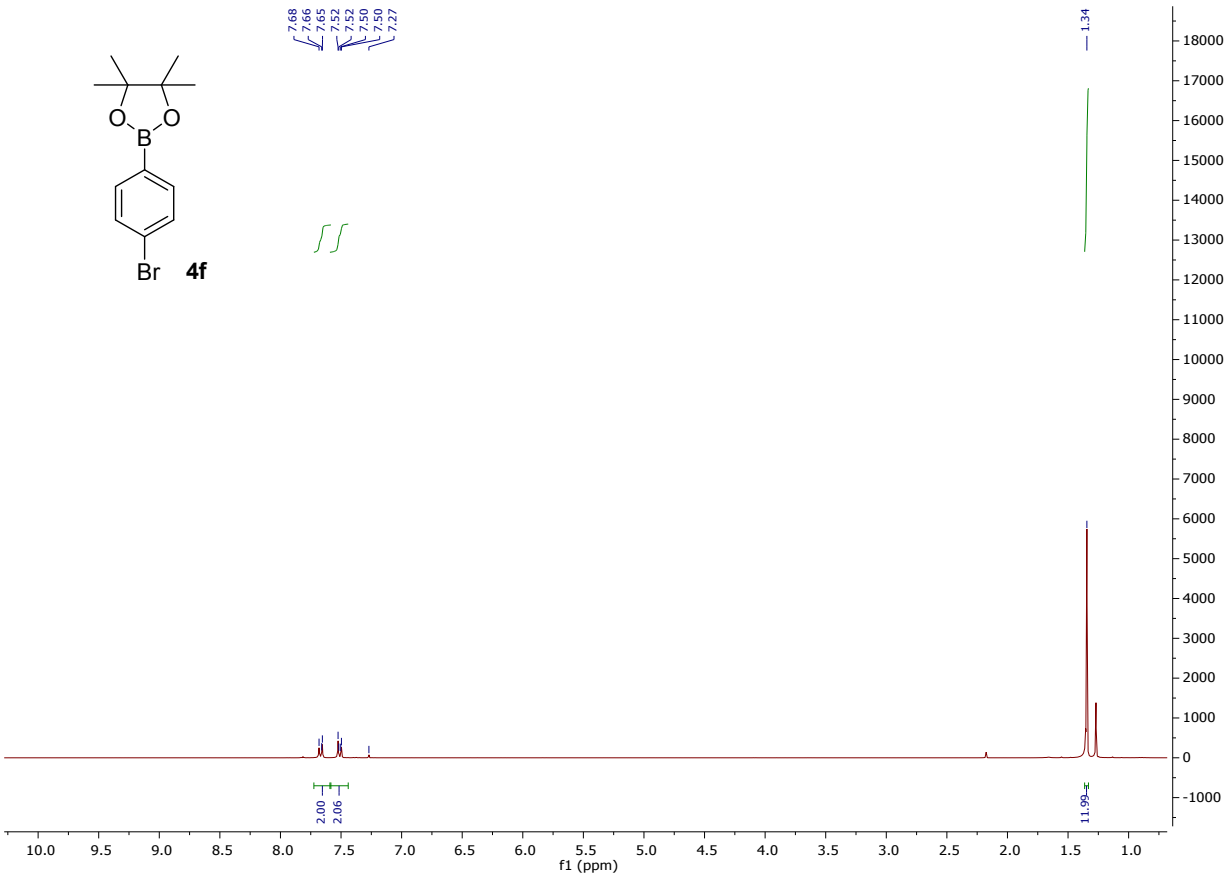

7.74
7.71
7.27
7.22
7.22
7.19
7.19
2.38
1.36
O
B
O
4a
2.00
1.99
3.09
12.30
f1 (ppm)

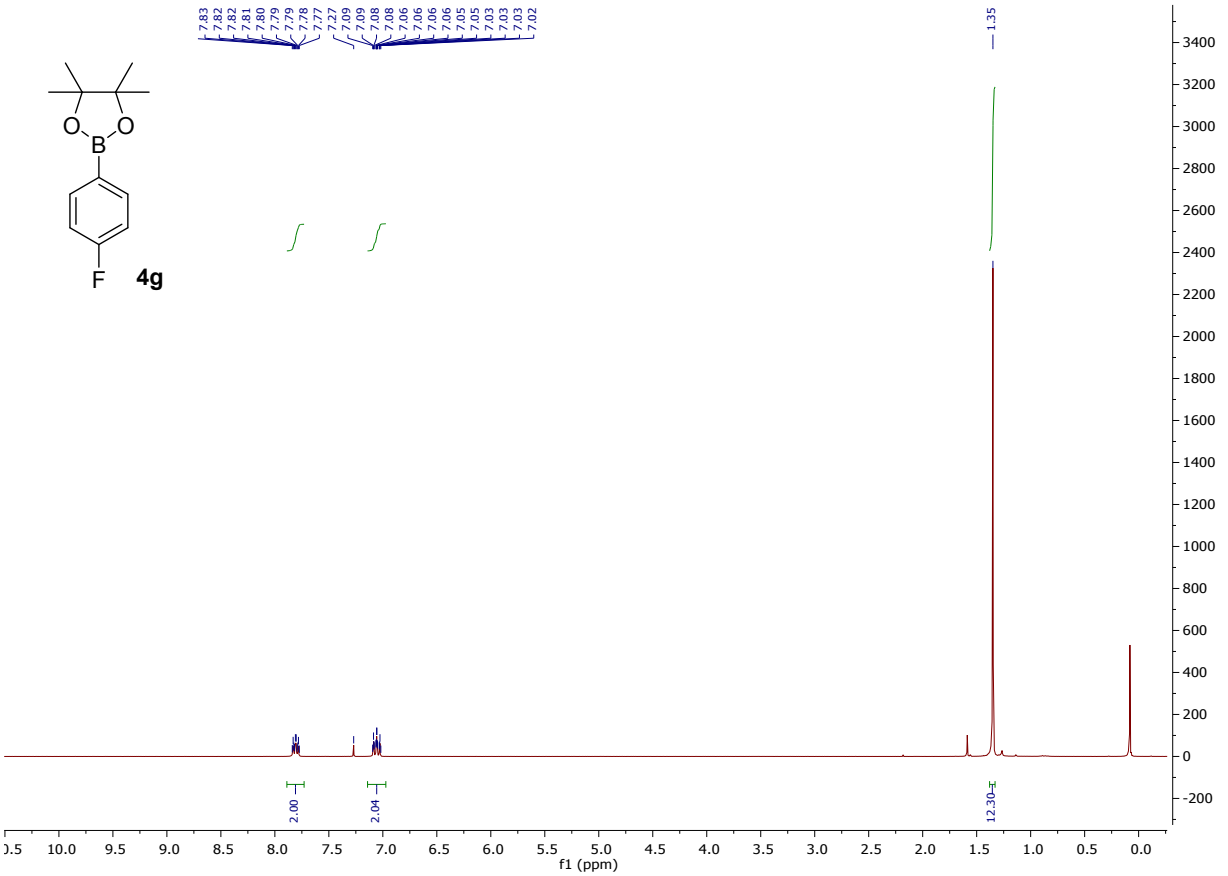

7.93
7.91
7.66
7.66
7.65
7.63
7.63
7.50
7.49
7.49
7.47
7.46
7.45
7.44
7.41
7.40
7.40
7.39
7.38
7.37
7.36
7.35
7.35
7.27
1.39
O
B
O
Ph
4b
2.00
4.09
2.09
1.08
12.29
f1 (ppm)

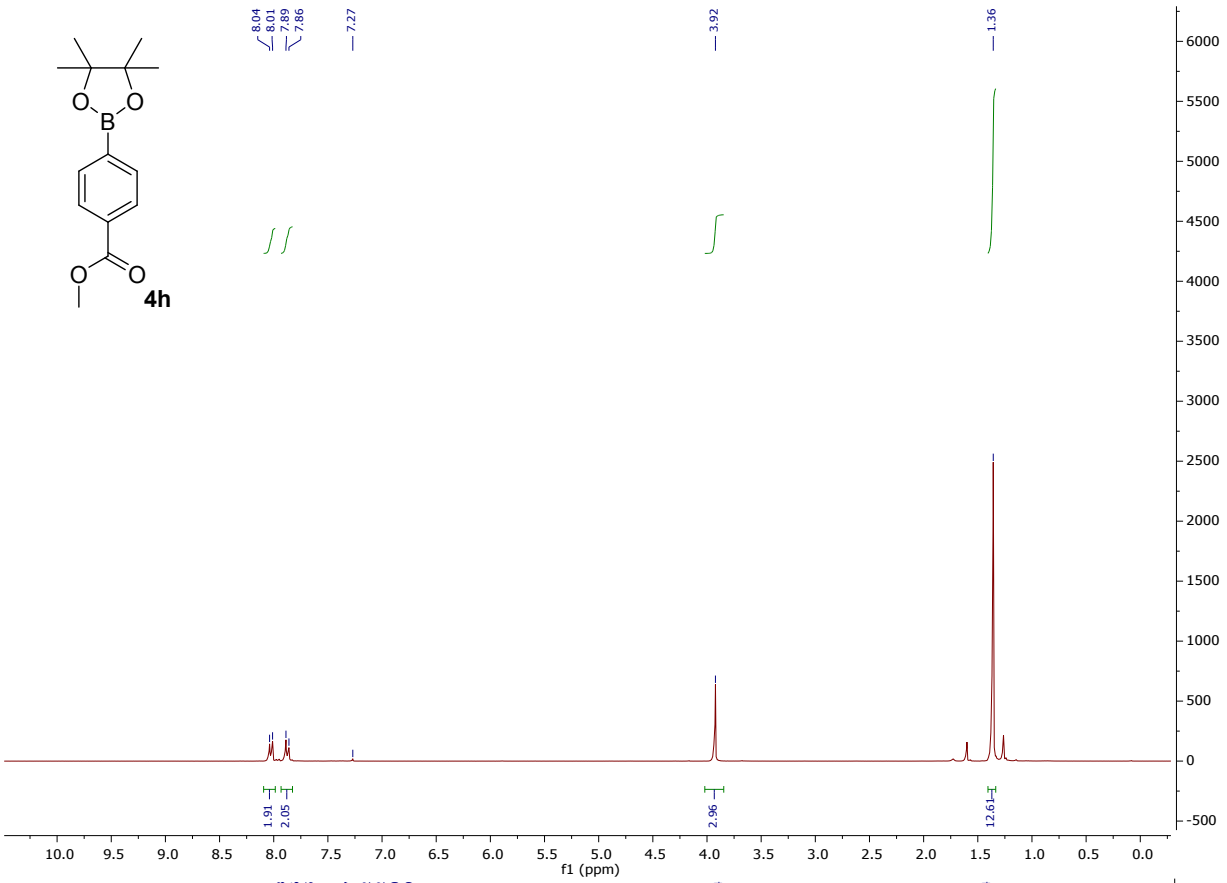

7.85
7.85
7.83
7.82
7.51
7.51
7.50
7.49
7.48
7.47
7.46
7.46
7.45
7.41
7.39
7.37
7.36
7.27
1.37
O
B
O
4c
2.00
1.04
2.04
12.29
f1 (ppm)

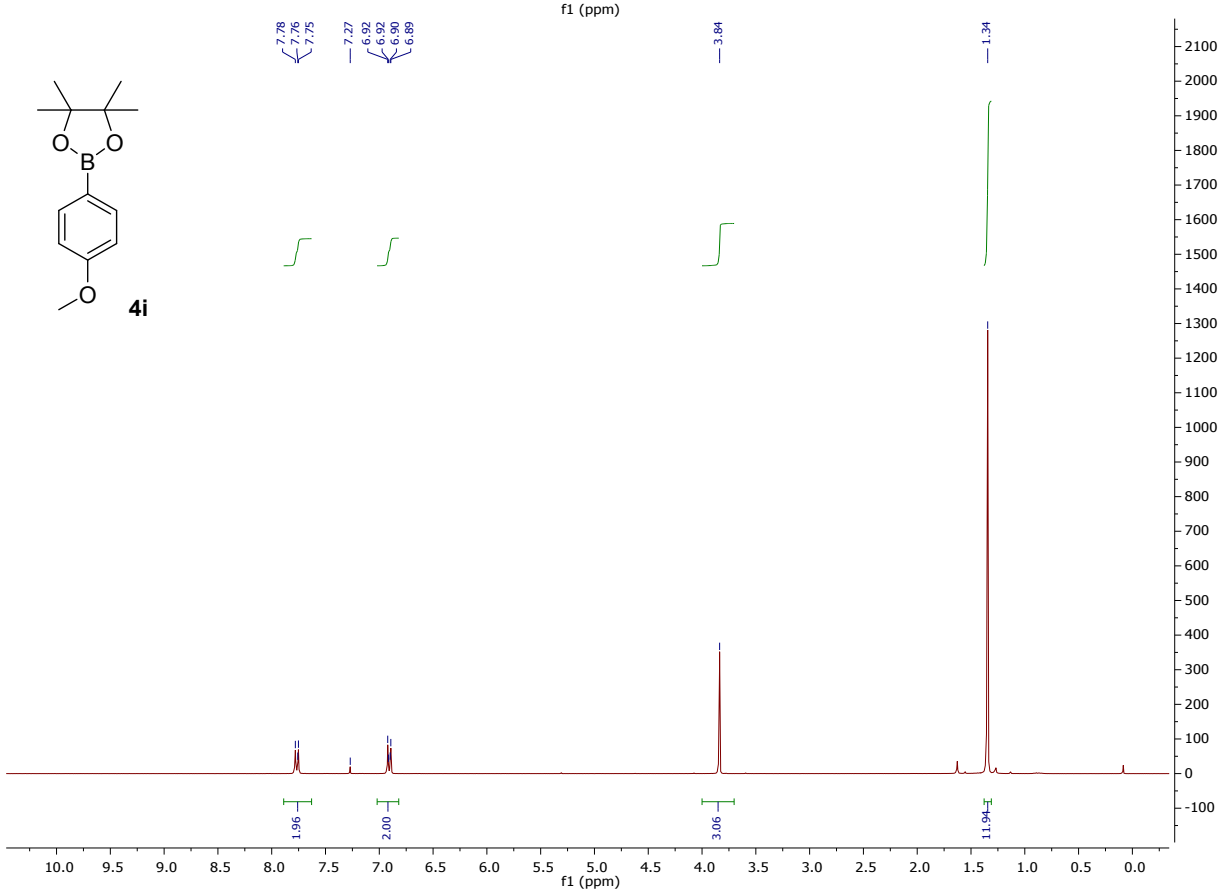

4d
7.27
6.79
2.38
2.25
1.39
2.00
6.16
3.12
12.29
f1 (ppm)

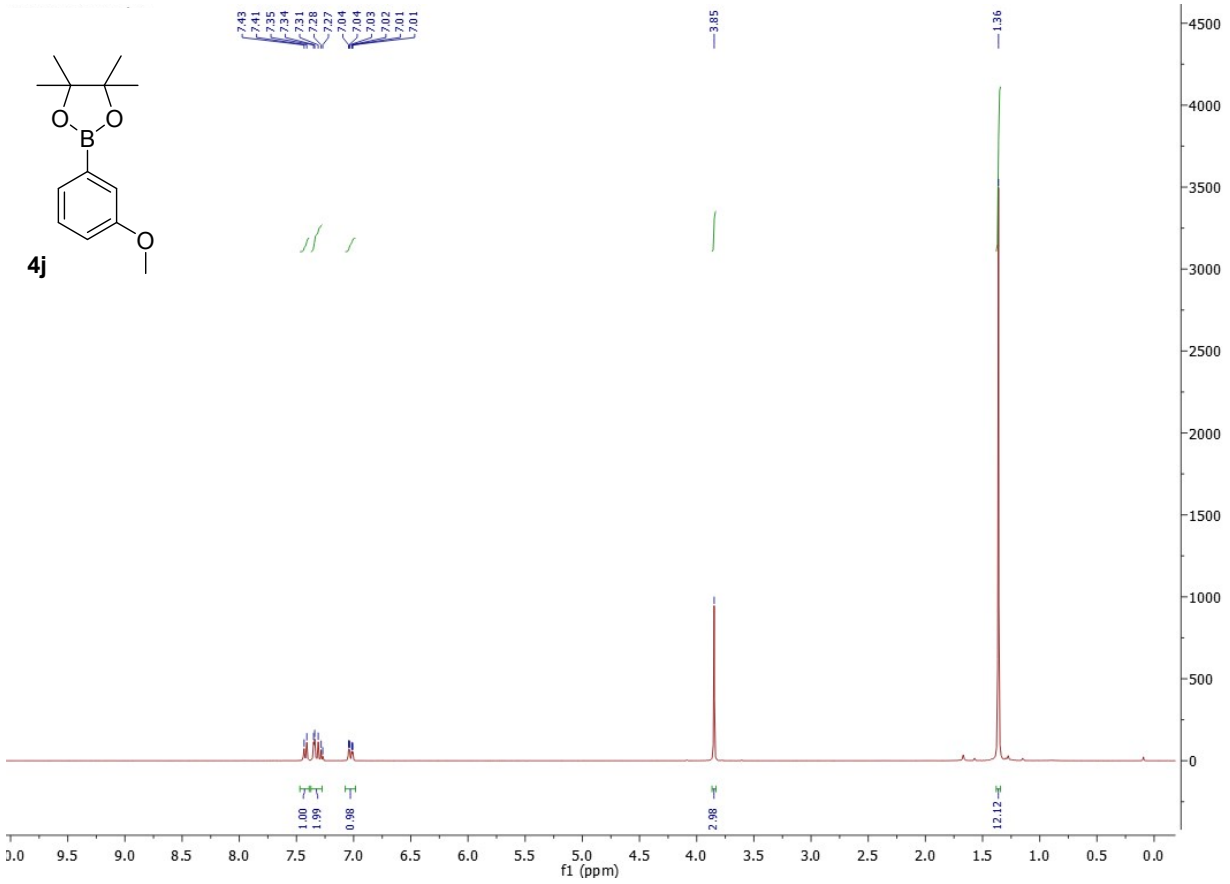

CF3 4e
7.94
7.91
7.64
7.61
7.27
1.37
2.00
2.04
12.17
f1 (ppm)

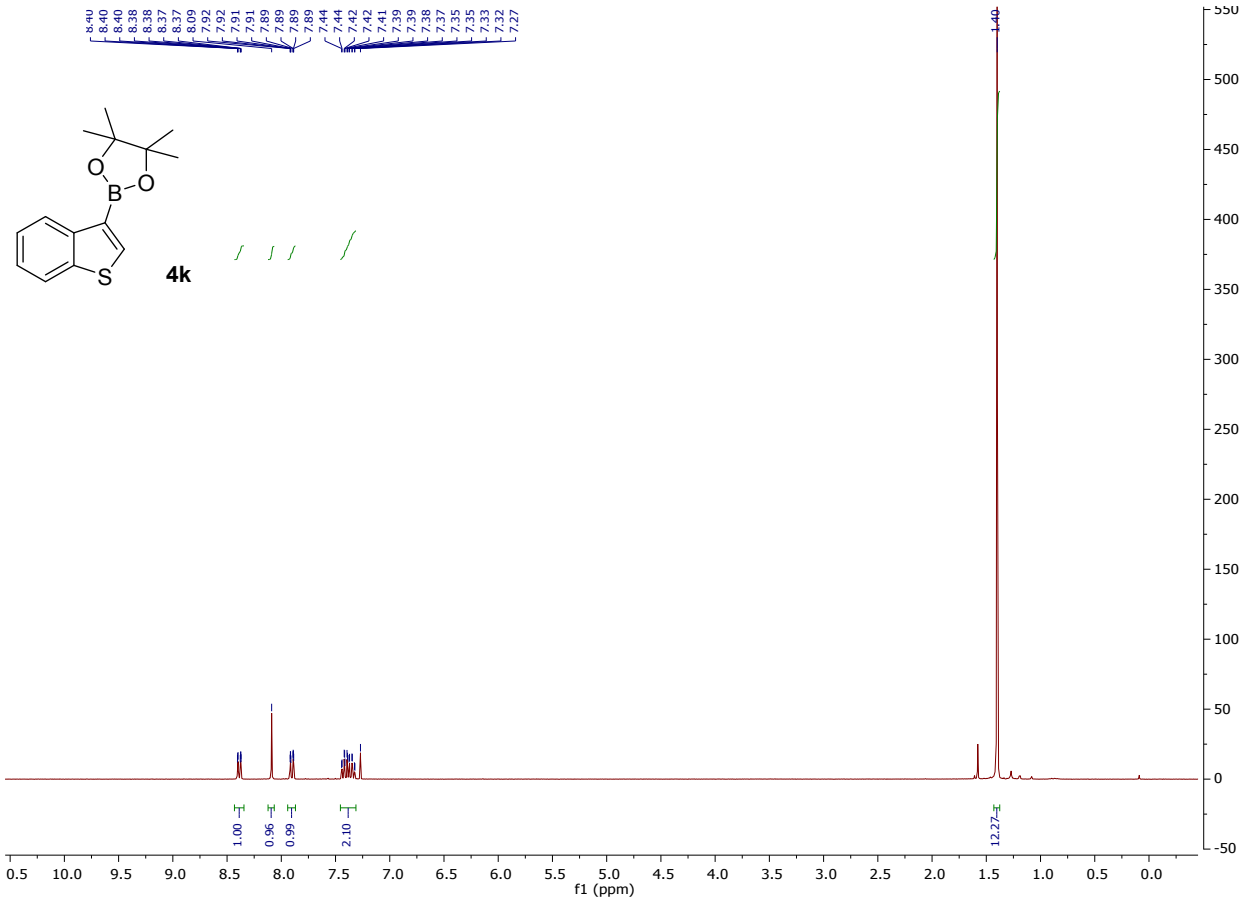

7.68
7.66
7.65
7.52
7.52
7.50
7.50
7.27
1.34
O
B
O
Br
4f
2.00
2.06
11.99
f1 (ppm)

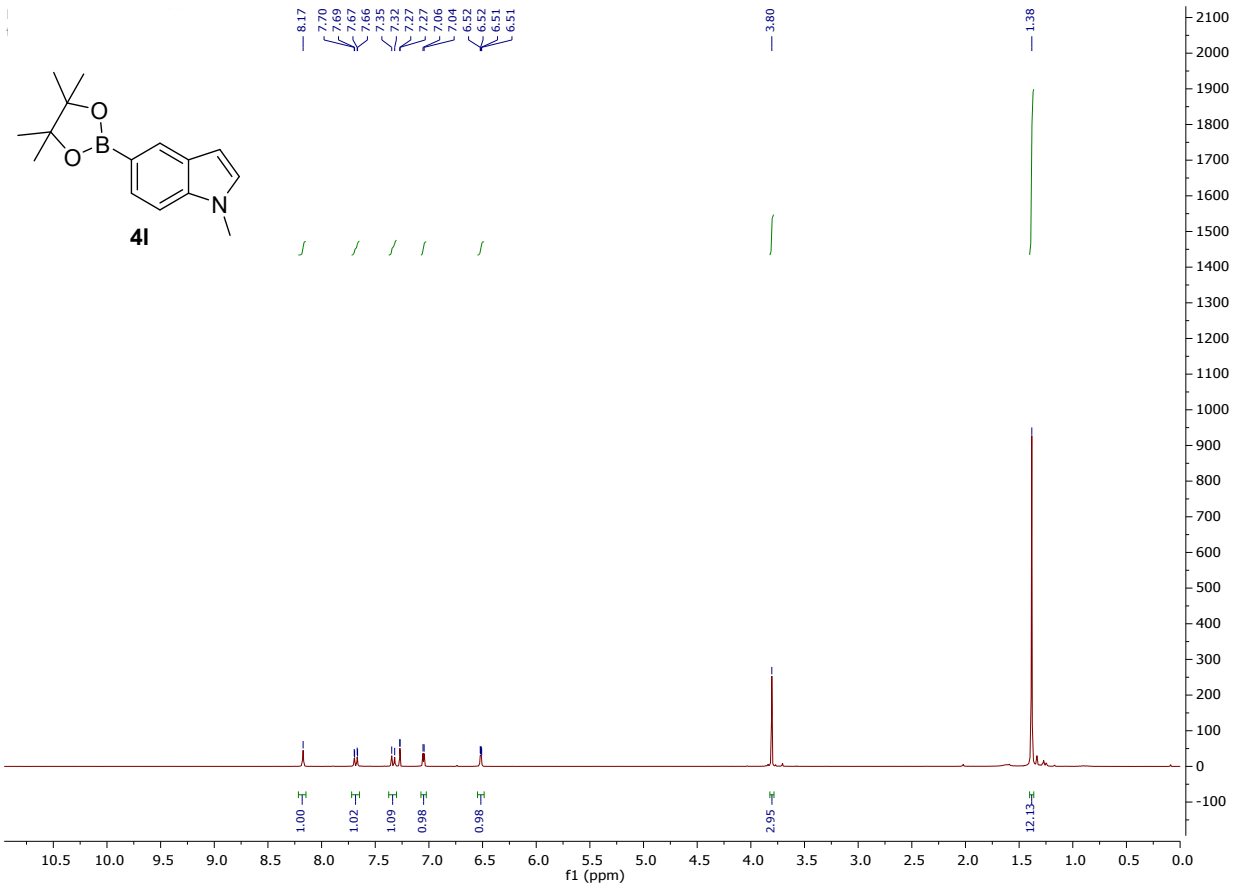

7.83
7.82
7.82
7.81
7.80
7.79
7.79
7.78
7.77
7.27
7.09
7.09
7.08
7.08
7.06
7.06
7.06
7.06
7.05
7.05
7.03
7.03
7.03
7.02
1.35
O
B
O
F
4g
2.00
2.04
12.30
f1 (ppm)


8.04
8.01
7.89
7.86
7.27
3.92
1.36
4h
1.91
2.05
2.96
12.61
f1 (ppm)

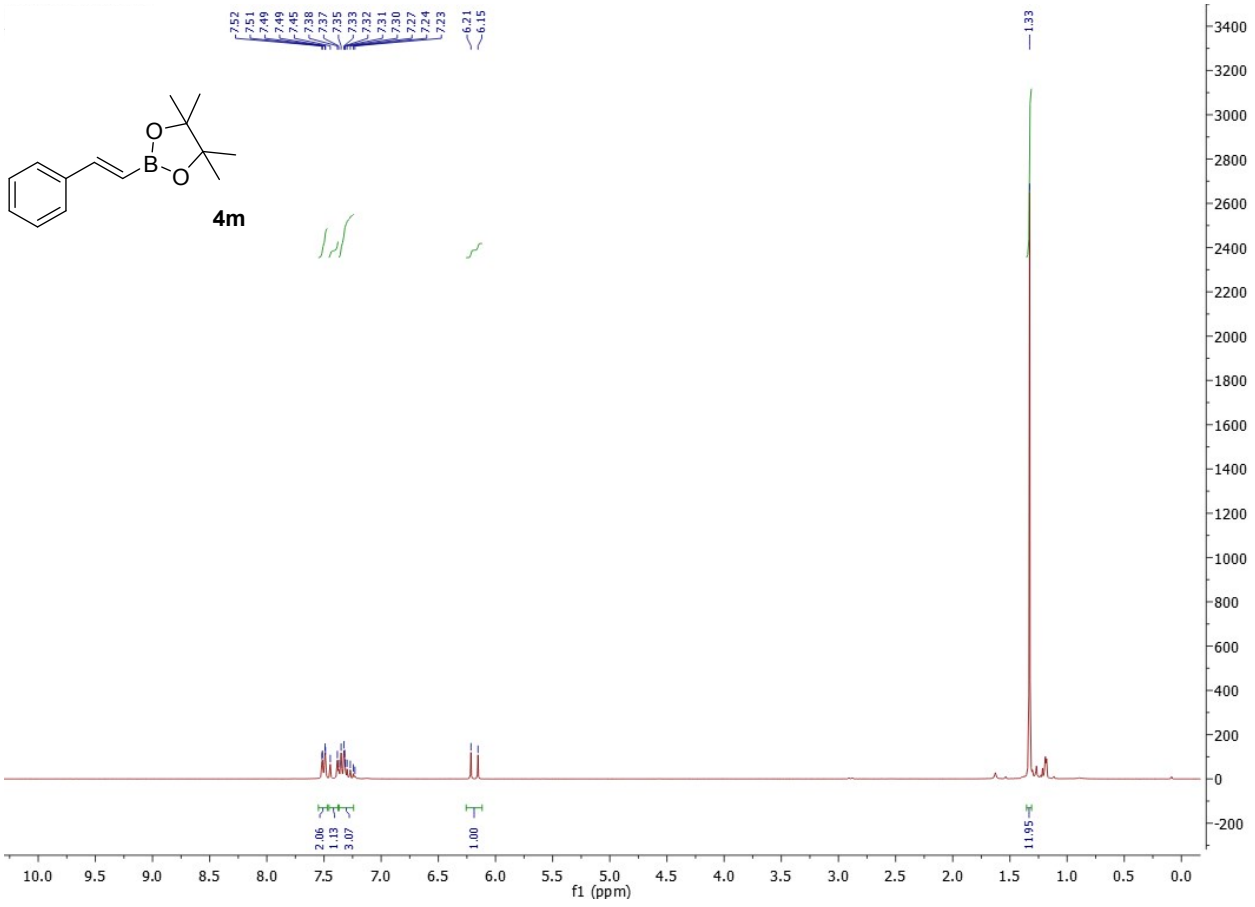

7.78
7.76
7.75
7.27
6.92
6.92
6.90
6.89
3.84
1.34
4i
1.96
2.00
3.06
11.94
f1 (ppm)


4j
7.43
7.41
7.35
7.34
7.31
7.28
7.27
7.04
7.04
7.03
7.02
7.01
7.01
3.85
1.36
1.00
1.99
0.98
2.98
12.12
f1 (ppm)


4k
8.40
8.40
8.40
8.38
8.38
8.37
8.37
8.09
7.92
7.92
7.91
7.91
7.89
7.89
7.89
7.89
7.44
7.44
7.42
7.42
7.41
7.39
7.39
7.38
7.37
7.35
7.35
7.33
7.32
7.27
1.40
1.00
0.96
0.99
2.10
12.27
f1 (ppm)


8.17
7.70
7.69
7.67
7.66
7.35
7.32
7.27
7.27
7.06
7.04
6.52
6.52
6.51
6.51
3.80
1.38
1.00
1.02
1.09
0.98
0.98
2.95
12.13
f1 (ppm)

4l


7.52
7.51
7.49
7.49
7.45
7.38
7.37
7.35
7.33
7.32
7.31
7.30
7.27
7.24
7.23
6.21
6.15
1.33
2.06
1.13
3.07
1.00
11.95
f1 (ppm)

4m

7.-References

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