Supporting Information

Aminoborate-Catalyzed Reductive Counterreactions for Oxidative Electrosynthetic Transformations

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Table of Contents

General Remarks

Unless otherwise noted, all experiments were conducted under a dry atmosphere of nitrogen, and reaction cells were assembled in a nitrogen-filled glove box. Anhydrous, degassed acetonitrile (MeCN) was obtained by sparging with nitrogen for 1 hour and storing over 3 Å molecular sieves for at least 24 hours prior to use. Aryl boronic acids were purchased from chemical suppliers and were converted to the aryl trifluoroborate salt following procedures reported in the literature unless otherwise stated.[1] All chloride salts and electrolytes were dried in a vacuum oven for at least 24 hours and stored in a nitrogen filled glove box before use. Protic solvents (1,1,1,3,3,3-hexafluoroisopropanol (HFIP), 2,2,2-trifluoroethanol (TFE), Methanol (MeOH), and glacial acetic acid (AcOH) were stored over 3 Å molecular sieves in sealed bottles under ambient atmosphere.

^{1}H NMR spectra were obtained at 400 or 600 MHz and chemical shifts were recorded relative to $CHCl_3$ in $CDCl_3$ (δ7.26 ppm). ^{13}C NMR were obtained at 101 MHz and chemical shifts were recorded relative to $CDCl_3$ (δ 77.16 ppm). ^{19}F NMR were obtained at 376 MHz. Quantitative ^{19}F NMR used for reaction development analysis utilized 1,3,5-trifluorobenzene as the internal standard to which chemical shifts were recorded relative to (δ -108.00 ppm). Proof of purity is demonstrated by copies of NMR spectra and high-resolution mass spectrometry. NMR multiplicities are reported as follows: singlet (s), doublet (d), triplet (t), quartet (q), multiplet (m), broad signal (br). GC analysis was performed on an Agilent 7890B GC equipped with an HP-5 column (30 m x 0.32 mm x 0.25 µm film) and an FID detector. Quantitative GC analysis was performed by adding dodecane as an internal standard to the reaction mixture upon completion of the reaction. Response factors for the products relative to the internal standard were measured for reaction development. HRMS was obtained using Bruker Elute SP HPLC with Bruker Impact II QqTOF Mass Spectrometer or Thermo Orbitrap Esploris MX LC-MS.

All electrochemical analyses were carried out in a nitrogen-filled glovebox. Supporting electrolytes were dried in a vacuum oven at 120 °C for 24 hours prior to use. Cyclic voltammetry was performed with a Biologic VSP multichannel potentiostat/galvanostat. Cyclic voltammetry was carried out in a three-electrode electrochemical cell, consisting of either a glassy carbon disk working electrode (3.0 mm diameter, 0.07 cm^2, BASi) or a platinum disk working electrode (1.6 mm diameter, 0.02 cm^2, BASi), a Ag/Ag^+ quasi-reference electrode (BASi) with 0.01 M $AgBF_4$ (Sigma) and 0.10 M supporting electrolyte (see experimental details) in MeCN, and a platinum wire counter electrode (23 cm, ALS). The glassy carbon disk electrode and the platinum disk electrode were polished in a nitrogen-filled glovebox using diamond polish (15 µm, BASi) and anhydrous MeCN. All experiments were performed at a scan rate of 100 mV/s in a MeCN solution containing 0.1 M supporting electrolyte unless otherwise noted. Reference electrodes were calibrated against an internal voltage reference of ferrocene (1-10 mM). Reactions were conducted as two-electrode cells with a LANHE LAND battery testing system using platinum mesh (30 mm x 5 mm x 0.5 mm, purchased from Jiangsu Plaza Premium Electric Instrument Co.,Ltd [Alibaba]) and reticulated vitreous carbon (RVC) (60 ppi, 35 mm x 7.5 mm x 4 mm, purchased from ERG Aerospace Corp.) electrodes. Reactions were conducted in Fisherband disposable borosilicate glass tubes with a threaded end (16 mm x 100 mm).

Safety Note: The electrochemical chlorodeborylation reactions are performed at 80 °C using acetonitrile (boiling point 82 °C) as solvent in a sealed reaction vessel. Heating solvents close to or past their boiling point in a sealed vessel poses a risk of pressure build up and potential explosion.

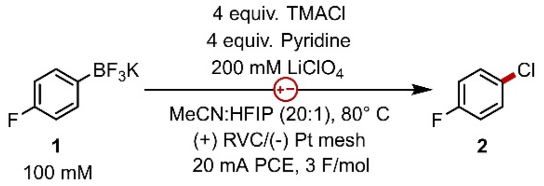
Electrochemical Cell Setup

Figure S1: Guide for electrochemical cell setup

1. Materials Required: 12 mL threaded reaction test tube, threaded test tube cap, PTFE septa (2), 20 D Chromel A wire, wire glue, 60 ppi RVC, Platinum Mesh, PTFE Tubing (3/16” ID, 1/4” OD, 1/32” WT), needle nose pliers with wire cutters. *Note: Copper wire cannot be used due to the oxidizing conditions and the generation of Cl^+/Cl_2*
2. Cut and straighten a 10 cm segment of chromel wire.
3. Using the pliers, bend a hook at one end of the wire.
4. Place that wire through one of the mesh holes right below the top bar and gently crimp. Place the PTFE tubing over the connection.
5. Cut a 3.5 cm x 0.75 cm x 0.4 cm sheet of RVC and another 10 cm segment of chromel wire.
6. Dip the RVC sheet into the wire glue to a depth of ~ 0.5 cm. Dip the chromel wire into the glue to a depth of ~ 0.5 cm.
7. Pierce the chromel wire into the RVC, making sure to not penetrate past the wire glue coverage. Cure overnight at room temperature or in the oven for a few hours.
8. Cut one PTFE septa into a 1.0 cm x 0.3 cm rectangle
9. Pierce the RVC electrode and the platinum mesh electrode through the rectangular piece of PTFE septa followed by the circular septa, with a wire separation distance of roughly 0.7 cm.

10. Place this assembly into the reaction vial and screw on the screw-cap to seal.

Additional Reaction Parameters Tested During Development

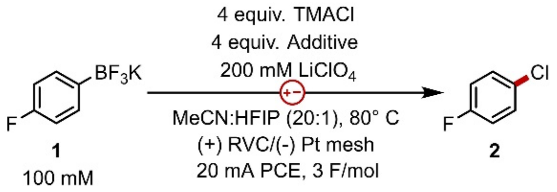


Entry	Reaction Variable	% yield Ar-Cl	E_{cell}
1	-	>95	1.8 V
2	40° C	>95	2.6 V
3	25° C	>95	2.7 V
4	LiCl instead of TMACl	>95	3.3 V
5	NaCl instead of TMACl	9	>5.0 V
6	KCl instead of TMACl	30	1.8 V
7	(+)RVC/(-)RVC	6	>5.0 V
8	(+) Pt Mesh/(-) Pt Mesh	>95	1.8 V
9	(+)Graphite/(-)Pt Plate	>95	2.6 V

Table S1: Control reactions for the chlorodeborylation of potassium 4-fluorophenyltrifluorborate (**1**).

All reactions were carried out on a 0.30 mmol scale following General Procedure A unless otherwise noted. Reactions were analyzed with ^{19}F-NMR using 1,3,5-trifluorobenzene as internal standard. HFIP= 1,1,1,3,3,3-hexafluoroisopropanol, PCE= Pulsed current electrolysis (60s on 30 s off), TMACl= tetramethylammonium chloride, RVC= Reticulated Vitreous Carbon.

Effect of Different Additives on Chlorodeborylation Yield



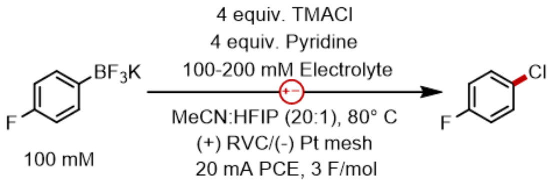
Entry	Additive	% yield Ar-Cl	E_{cell}
1	-	11	4.0 V
2	Triethylamine	0	2.3 V
3	DBU	0	>5.0 V
4	Pipiridine	0	2.8 V
5	Pyrrole	0	2.1 V
6	Pyridine	95	1.8 V
7	4-Methoxypyridine	77	2.8 V
8	2,4,6-collidine	83	2.5 V
9	4-trifluoromethylpyridine	91	2.3 V
10	Methyl isonicotinate	>95	2.1 V

Table S2: Reactions analyzing the effect of additives on the chlorodeborylation of **1**.

All reactions were carried out on a 0.30 mmol scale following General Procedure A unless otherwise noted. Reactions were analyzed with ^{19}F-NMR using 1,3,5-trifluorobenzene as internal standard. HFIP= 1,1,1,3,3,3-

hexafluoroisopropanol, PCE= Pulsed current electrolysis (60s on 30 s off), TMACl= tetramethylammonium chloride, RVC= Reticulated Vitreous Carbon. DBU= 1,8-Diazabicyclo[5.4.0]undec-7-ene.

Effect of Electrolyte on Chlorodeborylation Reaction

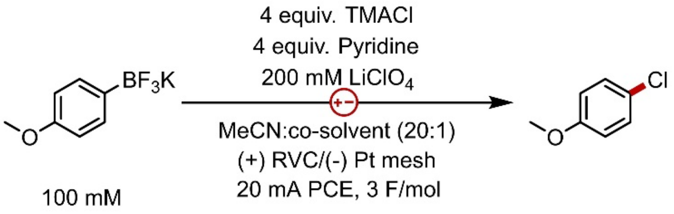


Entry	Electrolyte	% yield Ar-Cl	E_{cell}
1	200 mM $LiClO_4$	>95	1.8 V
2	200 mM $LiBF_4$	>95	2.2 V
3	200 mM $LiPF_6$	92	2.9 V
4	200 mM LiOTf	84	2.1 V
5	100 mM $TBAClO_4$	23	3.6 V
6	100 mM $TBABF_4$	18	3.7 V
7	100 mM $TBAPF_6$	7	4.0 V
8	200 mM KPF_6	20	3.9 V

Table S3: Reactions with a variety of different supporting electrolytes.

All reactions were carried out on a 0.30 mmol scale following General Procedure A unless otherwise noted. Reactions were analyzed with ^{19}F-NMR using 1,3,5-trifluorobenzene as internal standard. HFIP= 1,1,1,3,3,3-hexafluoroisopropanol, PCE= Pulsed current electrolysis (60s on 30 s off), TMACl= tetramethylammonium chloride, RVC= Reticulated Vitreous Carbon.

Reaction Development for electron rich arenes

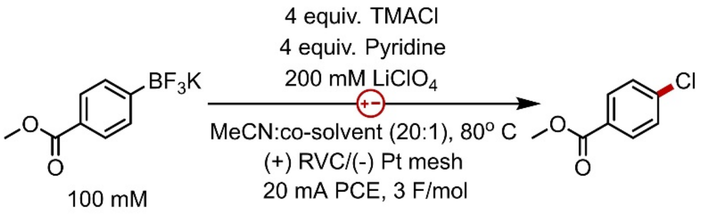


Entry	Additive/Co-solvent	% yield Ar-Cl	E_{cell}
1	HFIP, 25° C	25	5.5 V
2	HFIP, 60° C	37	3.8 V
3	HFIP, 80° C	62	3.6 V
4	HFIP, 3.5 V PPE, 80° C	67	*3.5 V
5	TFE, 80° C	83	2.8 V
6	TFE, 2.5 V PPE, 80° C	90	*2.5 V
7	TFE, 2.5 V PPE, 40° C	76	*2.5 V

Table S4: Varying co-solvent, temperature, and electrolysis conditions for the optimization of electron rich arenes. Asterisk (*) indicates Pulsed Potential Electrolysis (PPE) at the indicated cell potential.

All reactions were carried out on a 0.30 mmol scale following General Procedure A unless otherwise noted. Reactions were analyzed with Gas Column Chromatography using dodecane as internal standard. PCE= Pulsed current electrolysis (60s on 30 s off), TMACl= tetramethylammonium chloride, RVC= Reticulated Vitreous Carbon. HFIP= 1,1,1,3,3,3-hexafluoroisopropanol, TFE= 2,2,2-trifluoroethanol, PPE= Pulsed Potential Electrolysis (60 s on 30 s off)

Reaction Development for electron poor arenes



Entry	Additive/Co-solvent	% yield Ar-Cl	E_{cell}
1	HFIP	45	2.0 V
2	TFE	33	2.1 V
3	HFIP, 2.5 V PPE	45	*2.5 V
4	H_2O	4	2.5 V
5	PyHCl	36	2.3 V
6	AcOH	68	2.5 V
7	20:1:1 MeCN:AcOH:TFE	52	2.0 V
8	20:1:1 MeCN:AcOH:TFE, 1 eq. pyridine	84	2.2 V

Table S5: Varying co-solvent, protic additives, pyridine concentration, and electrolysis conditions for the optimization of electron poor arenes. Asterisk (*) indicates Pulsed Potential Electrolysis (PPE) at the indicated potential.

All reactions were carried out on a 0.30 mmol scale following General Procedure A unless otherwise noted. Reactions were analyzed with Gas Column Chromatography using dodecane as internal standard. PCE= Pulsed current electrolysis (60s on 30 s off), TMACl= tetramethylammonium chloride, RVC= Reticulated Vitreous Carbon. HFIP= 1,1,1,3,3,3-hexafluoroisopropanol, TFE= 2,2,2-trifluoroethanol, AcOH= acetic acid, PyHCl= pyridinium hydrochloride, PPE= Pulsed Potential Electrolysis (60 s on 30 s off)

Reaction Scope utilizing LiCl and 25 °C

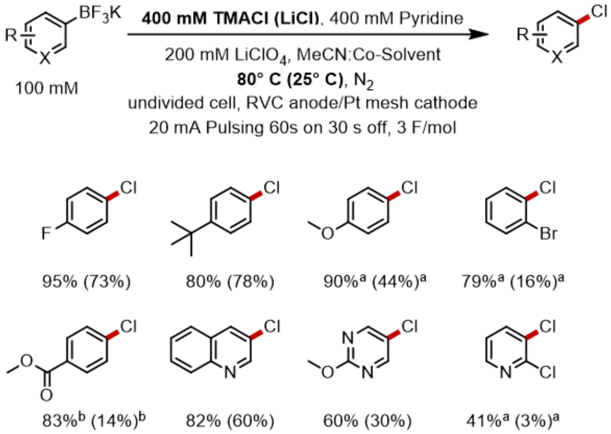


Chart S1: Chlorinated yields of aryl trifluoroborate salts. Yields were determined by GC analysis using dodecane as an internal standard. **Yields in parentheses are from reactions that utilized lithium chloride instead of TMACl and 25 °C instead of 80 °C.** Reactions were conducted utilizing General Procedure A (see below) with the above-mentioned changes when indicated. (a) Reactions were conducted utilizing General Procedure B (see below) with the above-mentioned changes when indicated. (b) Reactions were conducted utilizing General Procedure C (see below) with the above-mentioned changes when indicated.

Low yielding substrate scope

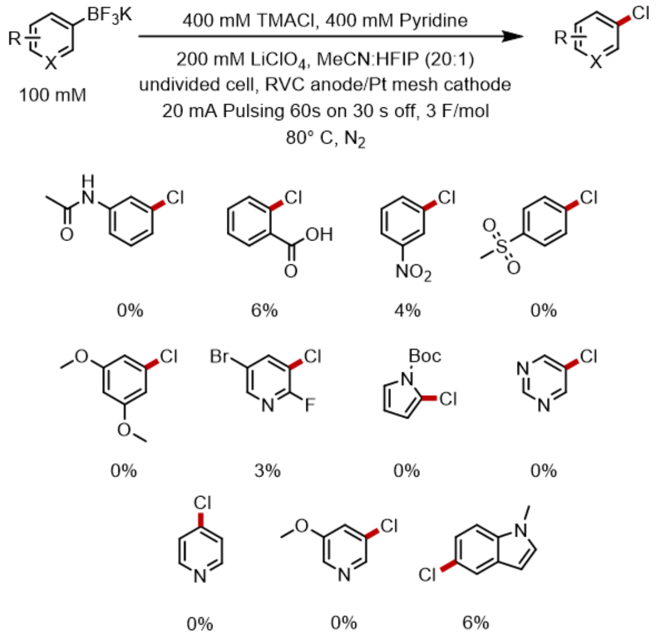
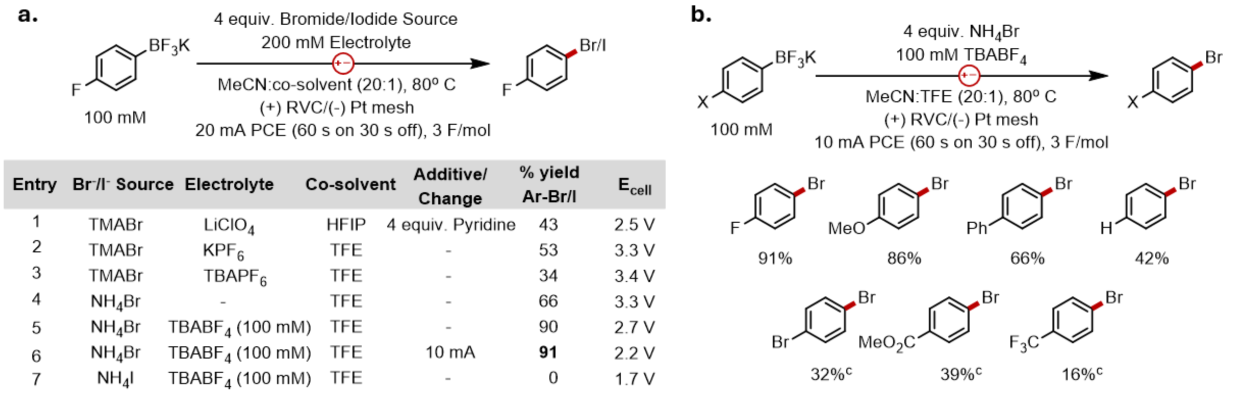


Chart S2: Chlorinated yields for aryl-trifluoroborate substrates that do not perform under the methodology.

All reactions were carried out on a 0.30 mmol scale following General Procedure A. Yields were determined by Gas Column Chromatography analysis using dodecane as an internal standard.

Electrochemical Bromination Conditions for some Aryl-BF₃K Substrates



Entry	Br⁻/I⁻ Source	Electrolyte	Co-solvent	Additive/ Change	% yield Ar-Br/I	E_{cell}
1	TMABr	$LiClO_4$	HFIP	4 equiv. Pyridine	43	2.5 V
2	TMABr	KPF_6	TFE	-	53	3.3 V
3	TMABr	$TBAPF_6$	TFE	-	34	3.4 V
4	NH_4Br	-	TFE	-	66	3.3 V
5	NH_4Br	$TBABF_4$ (100 mM)	TFE	-	90	2.7 V
6	NH_4Br	$TBABF_4$ (100 mM)	TFE	10 mA	**91**	2.2 V
7	NH_4I	$TBABF_4$ (100 mM)	TFE	-	0	1.7 V

Table S6: (**a**) Electrochemical bromination optimization of potassium 4-fluorophenyltrifluoroborate. (**b**) Scope of bromination reaction. (c) Reactions performed without TBABF₄ and with AcOH as co-solvent instead of TFE.

All reactions were carried out on a 0.30 mmol scale following General Procedure D unless otherwise noted. Yields were determined by Gas Column Chromatography analysis using dodecane as an internal standard.

Current Profile With and Without Pulsing

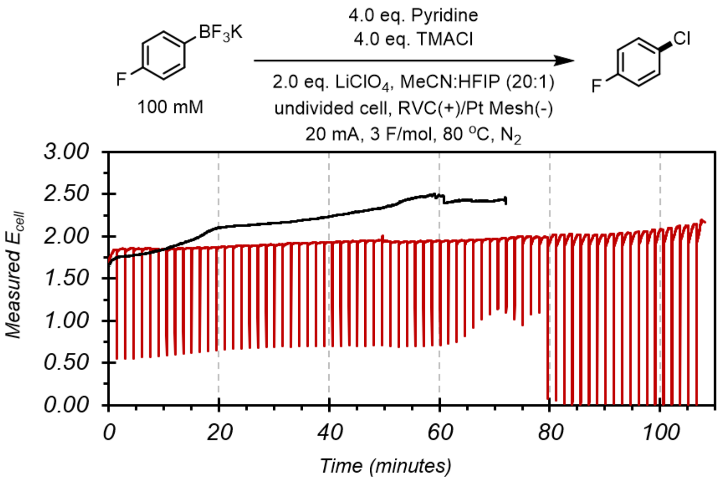


Figure S2: Voltage profile of the chlorodeborylation of potassium 4-fluorophenyltrifluoroborate under 20 mA constant current electrolysis (black trace) or under 20 mA pulsing current electrolysis electrolyzing for 60 seconds with a 30 second rest period (red trace). Electrolysis was performed until 3 F/mol capacity was achieved.

All reactions were carried out on a 0.30 mmol scale following General Procedure A unless otherwise noted. Reaction conditions: 100 mM potassium 4-fluorophenyltrifluoroborate, 200 mM $LiClO_4$, 400 mM Pyridine, 400 mM tetramethylammonium chloride, MeCN-d3:HFIP (20:1), Pt Mesh cathode, RVC anode, 80° C, 20mA (CCE [Black Trace] or pulsing 60 s on 30 s off [Red Trace]), 3 F/mol.

Divided Cell Reaction

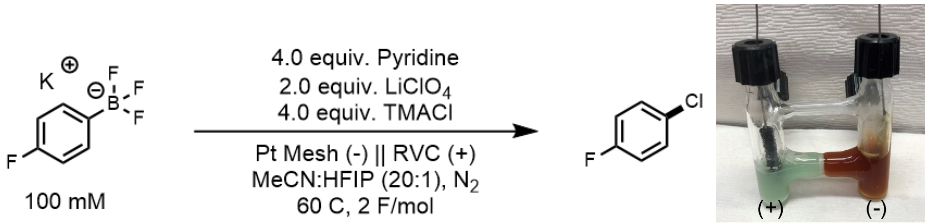


Figure S3: Divided cell reaction for the electrochemical chlorodeborylation of potassium 4-fluorophenyltrifluoroborate. Picture shows divided cell post electrolysis.

This reaction was performed following General Procedure A on a 0.40 mmol scale in a divided cell (glass frit partition).

Anodic Cell: Potassium 4-fluorophenyltrifluoroborate (81 mg, 0.40 mmol, 1.0 equiv.), pyridine (127 mg, 1.60 mmol, 4.0 equiv.), lithium perchlorate (85 mg, 0.80 mmol, 2.0 equiv.), tetramethylammonium chloride (176 mg, 1.60 mmol, 4.0 equiv.), acetonitrile (4 mL), 1,3,5-trifluorobenzene internal standard, and HFIP (0.20 mL).
Cathodic Cell: Potassium 4-fluorophenyltrifluoroborate (81 mg, 0.40 mmol, 1.0 equiv.), pyridine (127 mg, 1.60 mmol, 4.0 equiv.), lithium perchlorate (85 mg, 0.80 mmol, 2.0 equiv.), tetramethylammonium chloride (176 mg, 1.60 mmol, 4.0 equiv.), acetonitrile (4 mL), 1,3,5-trifluorobenzene internal standard, and HFIP (0.20 mL).

The reaction cell was set up in a nitrogen filled glove box. Once removed, it was placed in an oil bath at 60 °C and allowed to stir for 5 minutes. 30 V was applied until 2 F/mol capacity was achieved. Following electrolysis, an aliquot was removed from each chamber and analyzed via ^{19}F NMR.

Yield of 1-chloro-4-fluorobenzene: Anodic Cell: 91%, Cathodic Cell 7% (due to crossover).

Time-point study of aryl chloride yield vs capacity

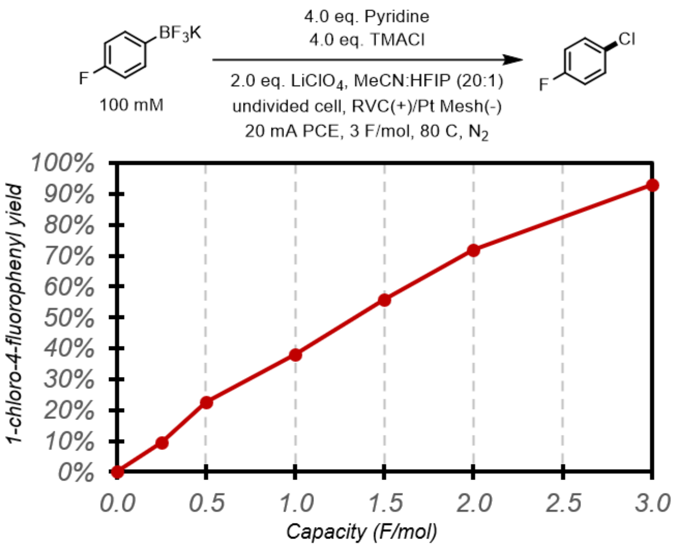


Figure S4: Aryl chloride product yield vs capacity (e^- equiv.).

This reaction was performed following General Procedure A using 1,3,5-trifluorobenzene as ^{19}F NMR internal standard. Aliquots were removed periodically (0 F/mol, 0.5 F/mol, 1.0 F/mol, 1.5 F/mol, 2.0 F/mol, and 3.0 F/mol) and analyzed by ^{19}F NMR.

NMR Analysis of Cathode Fouling Layer

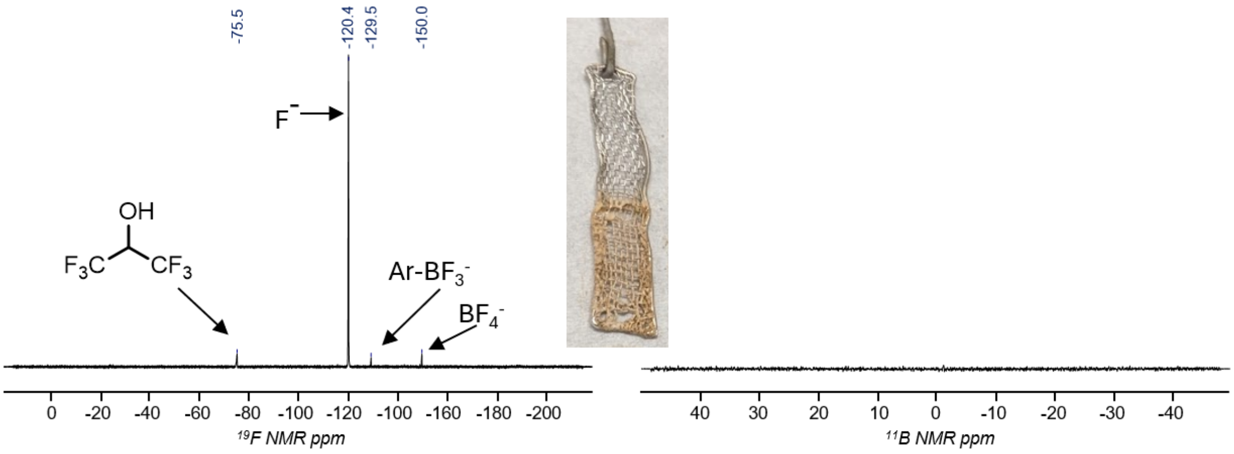


Figure S5: ^{19}F and ^{11}B NMR analysis of the material found on the cathode post electrolysis in reactions excluding pyridine. NMR analysis performed in H_2O solvent.

Cyclic Voltammograms found in Manuscript Figure 2

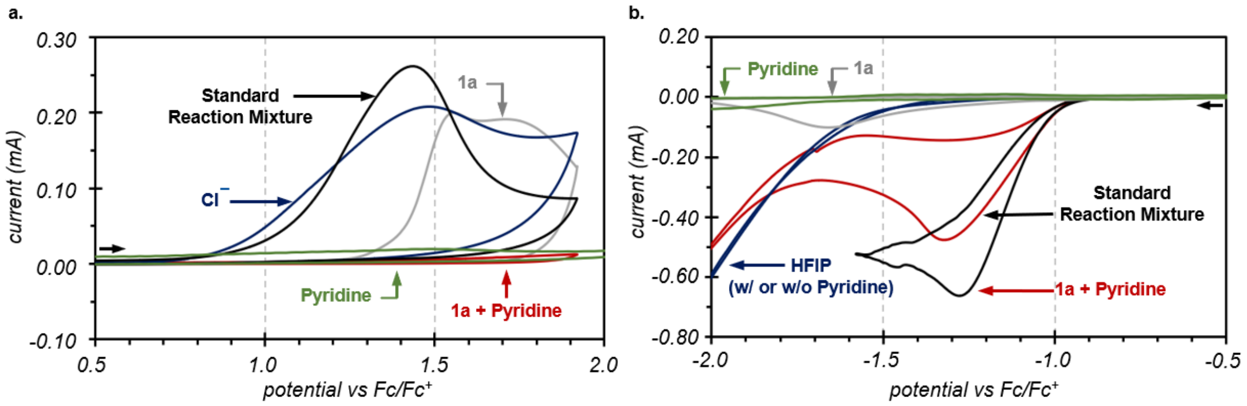


Figure S6: (a) CVs of standard reaction conditions (black trace), TMACl (blue trace), **1a** (grey trace), **1a** and pyridine (red trace), pyridine (green trace). (b) CVs of standard reaction conditions (black trace), HFIP and pyridine (blue trace), **1a** (grey trace), **1a** and pyridine (red trace), pyridine (green trace). CVs are reported in IUPAC convention.

CV conditions: 25 °C, acetonitrile solvent sparged with N_2, 200 mM lithium perchlorate, Glassy Carbon WE (ϕ 3.0 mm) [a] -or-Pt disk WE (ϕ 1.6 mm) [b], Pt wire CE and Ag/Ag$^+$ quasi reference electrode. The electrochemical potentials are normalized to an internal ferrocene reference.

a. Scan starts at 0.0 V vs Fc/Fc$^+$ in the anodic direction and reverses at 1.9 V vs Fc/Fc$^+$.
Black trace: 50 mM 4-fluorophenyl-BF_3K, 200 mM pyridine, 200 mM tetramethylammonium chloride, 250 mM HFIP.
Blue trace: 200 mM tetramethylammonium chloride
Grey trace: 50 mM 4-fluorophenyl-BF_3K.
Red trace: 50 mM 4-fluorophenyl-BF_3K, 200 mM pyridine.
Green trace: 100 mM pyridine

b. Scan starts at 0.0 V vs Fc/Fc$^+$ in the cathodic direction and reverses at -2.0 V vs Fc/Fc$^+$ (red trace reverses at -1.6 V vs Fc/Fc$^+$).
Black trace: 50 mM 4-fluorophenyl-BF_3K, 200 mM pyridine, 200 mM tetramethylammonium chloride, 250 mM HFIP.
Blue trace: 250 mM HFIP
Grey trace: 50 mM 4-fluorophenyl-BF_3K.
Red trace: 50 mM 4-fluorophenyl-BF_3K, 200 mM pyridine.
Green trace: 100 mM pyridine

Cyclic Voltammograms found in Manuscript Figure 3b

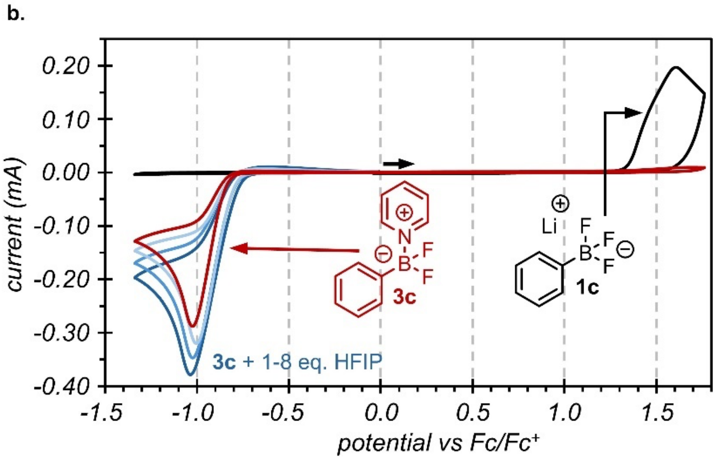


Figure S7: CVs of **1c** (black trace), **1c** and pyridine (red trace), **1c** and pyridine and HFIP (increasing concentrations)(blue traces). CVs are reported in IUPAC convention.

CV conditions: 25 °C, acetonitrile solvent sparged with N_2, 100 mM supporting electrolyte (see below for more detail), Pt disk WE (ϕ 1.6 mm), Pt wire CE and Ag/Ag^+ quasi reference electrode. The electrochemical potentials are normalized to an internal ferrocene reference.

Red trace: Scan starts at 0.0 V vs Fc/Fc^+ in the anodic direction and reverses at 1.8 V vs Fc/Fc^+. 100 mM $TBAClO_4$ supporting electrolyte, 25 mM potassium phenyltrifluoroborate, 50 mM Pyridine, 50 mM $LiClO_4$.

Blue traces: Scan starts at 0.0 V vs Fc/Fc^+ in the anodic direction and reverses at 1.8 V vs Fc/Fc^+. 100 mM $TBAClO_4$ supporting electrolyte, 25 mM potassium phenyltrifluoroborate, 50 mM Pyridine, 50 mM $LiClO_4$, and 1,1,1,3,3,3-hexafluoroisopropanol (25 mM [light blue], 50 mM [blue], 200 mM [dark blue]).

Black trace: Scan starts at 0.0 V vs Fc/Fc^+ in the anodic direction and reverses at 1.8 V vs Fc/Fc^+. 100 mM $TBAClO_4$ supporting electrolyte, 25 mM potassium 4-fluorophenyltrifluoroborate.

Cyclic Voltammograms of Aryl-BF$_3$ + pyridine complexes

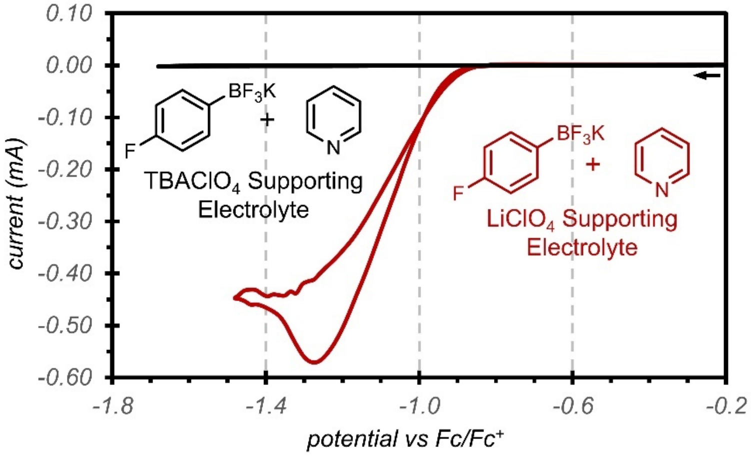


Figure S8: Cyclic voltammograms (IUPAC convention) of potassium 4-fluorophenyltrifluoroborate and pyridine in the presence of a tetrabutylammonium electrolyte (black trace) or a lithium electrolyte (red trace).

CV conditions: 25 °C, acetonitrile solvent sparged with N_2, 200 mM supporting electrolyte (see below for more detail), Pt disk WE (ϕ 1.6 mm), Pt wire CE and Ag/Ag$^+$ quasi reference electrode. The electrochemical potentials are normalized to an internal ferrocene reference.

Black trace: Scan starts at 0.0 V vs Fc/Fc$^+$ in the cathodic direction and reverses at -1.7 V vs Fc/Fc$^+$. 200 mM TBAClO$_4$ supporting electrolyte, 25 mM potassium 4-fluorophenyltrifluoroborate, 100 mM Pyridine.

Red trace: Scan starts at 0.0 V vs Fc/Fc$^+$ in the cathodic direction and reverses at -1.5 V vs Fc/Fc$^+$. 100 mM LiClO$_4$ supporting electrolyte, 25 mM potassium 4-fluorophenyltrifluoroborate, 100 mM Pyridine.

Cyclic Voltammograms of very electron rich and very electron poor aryl trifluoroborate salts

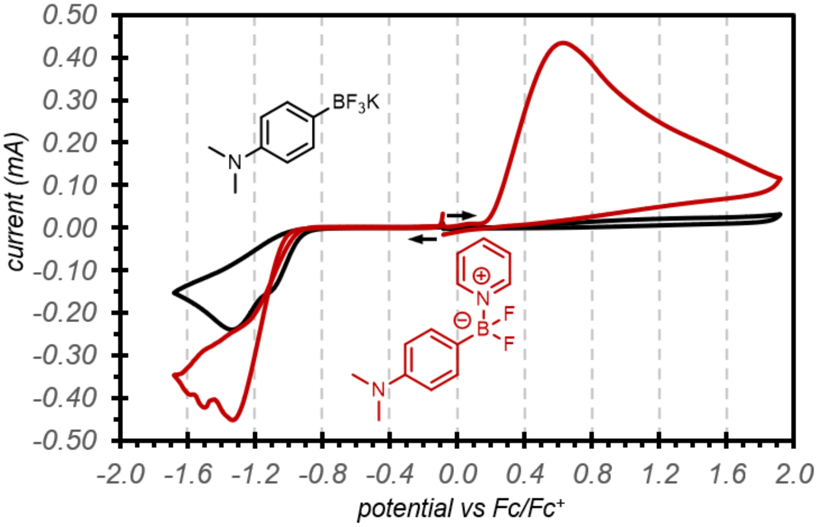


Figure S9: Cyclic voltammograms of potassium 4-dimethylaminopheny trifluoroborate salt without pyridine (black trace) and with pyridine (red trace).

CV conditions: 25 °C, acetonitrile solvent sparged with N_2, 200 mM lithium perchlorate supporting electrolyte, Pt disk WE (ϕ 1.6 mm) or GC WE (ϕ 3.0 mm), Pt wire CE and Ag/Ag$^+$ quasi reference electrode. The electrochemical potentials are normalized to an internal ferrocene reference.

Black trace (cathodic scan): Pt WE, Scan starts at 0.0 V vs Fc/Fc$^+$ in the cathodic direction and reverses at -1.6 V vs Fc/Fc$^+$. 25 mM potassium 4-dimethylaminophenyltrifluoroborate.
Black trace (anodic scan): GC WE, Scan starts at 0.0 V vs Fc/Fc$^+$ in the anodic direction and reverses at 1.9 V vs Fc/Fc$^+$. 25 mM potassium 4-dimethylaminophenyltrifluoroborate.
Red trace (cathodic scan): Pt WE, Scan starts at 0.0 V vs Fc/Fc$^+$ in the cathodic direction and reverses at -1.6 V vs Fc/Fc$^+$. 25 mM potassium 4-dimethylaminophenyltrifluoroborate, 50 mM pyridine
Red trace (cathodic scan): GC WE, Scan starts at 0.0 V vs Fc/Fc$^+$ in the anodic direction and reverses at 1.9 V vs Fc/Fc$^+$. 25 mM potassium 4-dimethylaminophenyltrifluoroborate, 50 mM pyridine.

NMR Analysis of Aryl-BF_2-Py

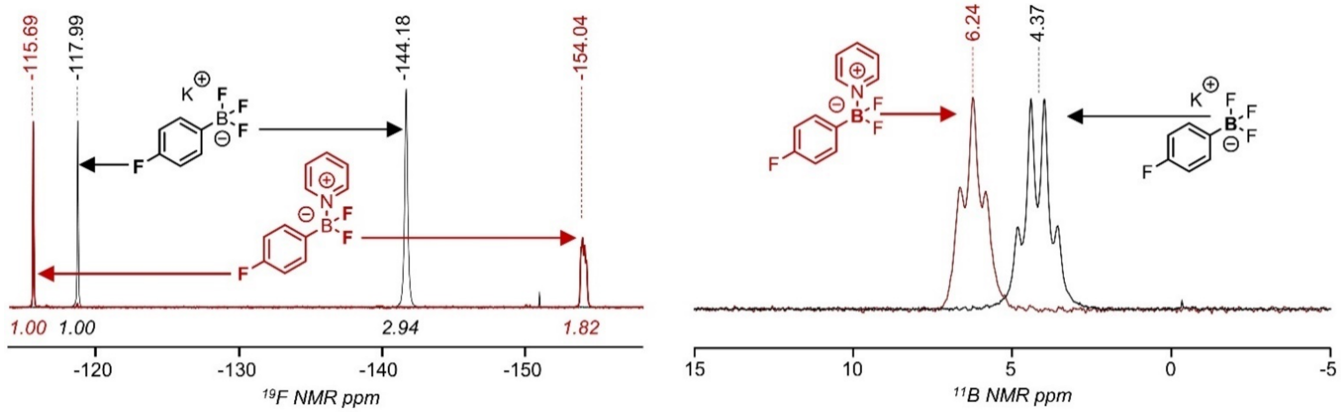


Figure S10: ^{19}F-NMR and ^{11}B-NMR of potassium 4-fluorophenyltrifluoroborate (black spectrum) and 4-fluorophenyltrifluoroborate, pyridine, and lithium perchlorate (red spectrum) in MeCN.

Black Spectrum: 100 mM potassium 4-fluorophenyltrifluoroborate in acetonitrile
Red Spectrum: 100 mM potassium 4-fluorophenyltrifluoroborate, 400 mM pyridine, 400 mM $LiClO_4$ in acetonitrile
^{19}F NMR- Chemical shifts are referenced vs 1,3,5-trifluorobenzene which is normalized to -108.00 ppm
^{11}B-NMR- Chemical Shifts are referenced vs sodium tetraphenylborate which is normalized to -5.74 ppm

^{1}H-NMR Analysis of HER Post-Electrolysis

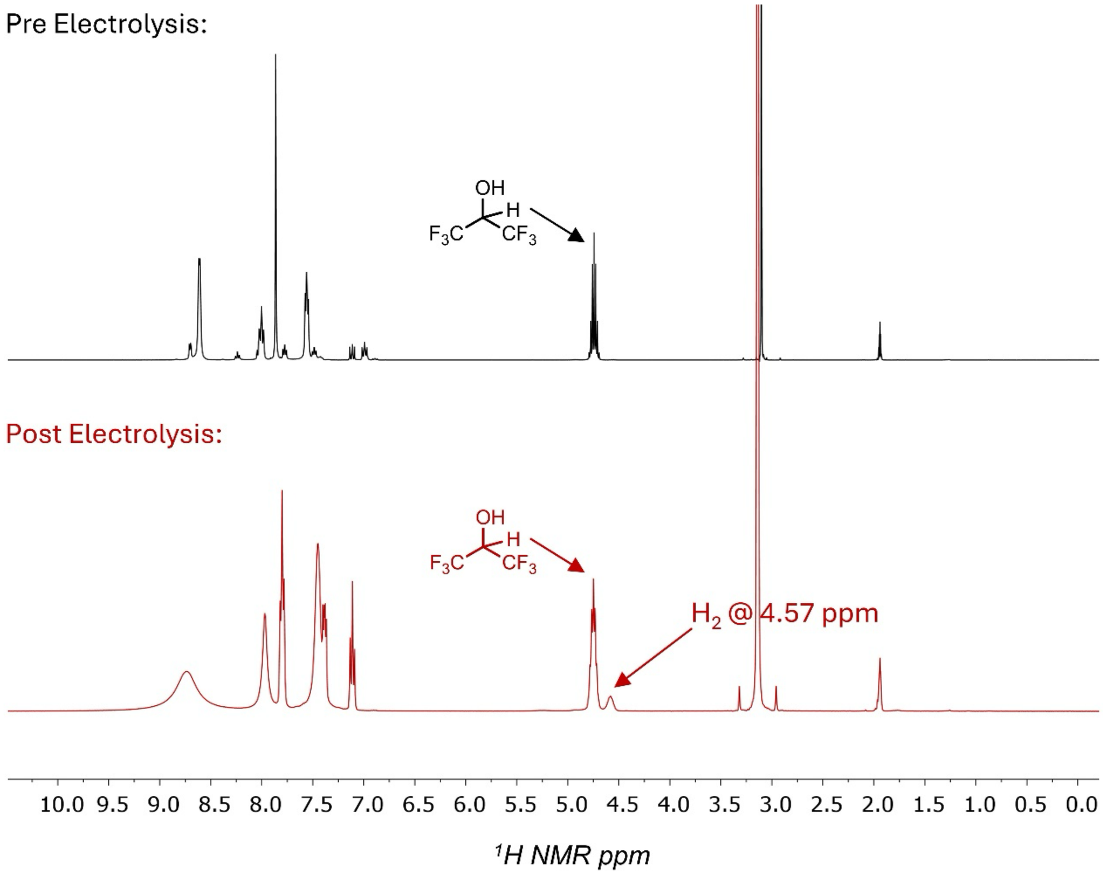


Figure S11: ^{1}H-NMR of a reaction with potassium 4-fluorophenyltrifluoroborate following General Procedure A pre-electrolysis (black spectrum) and post-electrolysis (red spectrum).

Reaction conditions: 100 mM potassium 4-fluorophenyltrifluoroborate, 200 mM $LiClO_4$, 400 mM Pyridine, 400 mM tetramethylammonium chloride, MeCN-d3:HFIP (20:1), Pt Mesh cathode, RVC anode, 80° C, 20mA pulsing 60 s on 30 s off, 3 F/mol. H_2 observed in ^{1}H-NMR at 4.57 ppm post electrolysis.[2]

^{2}H-NMR Analysis of Deuterated Co-solvent Post-Electrolysis

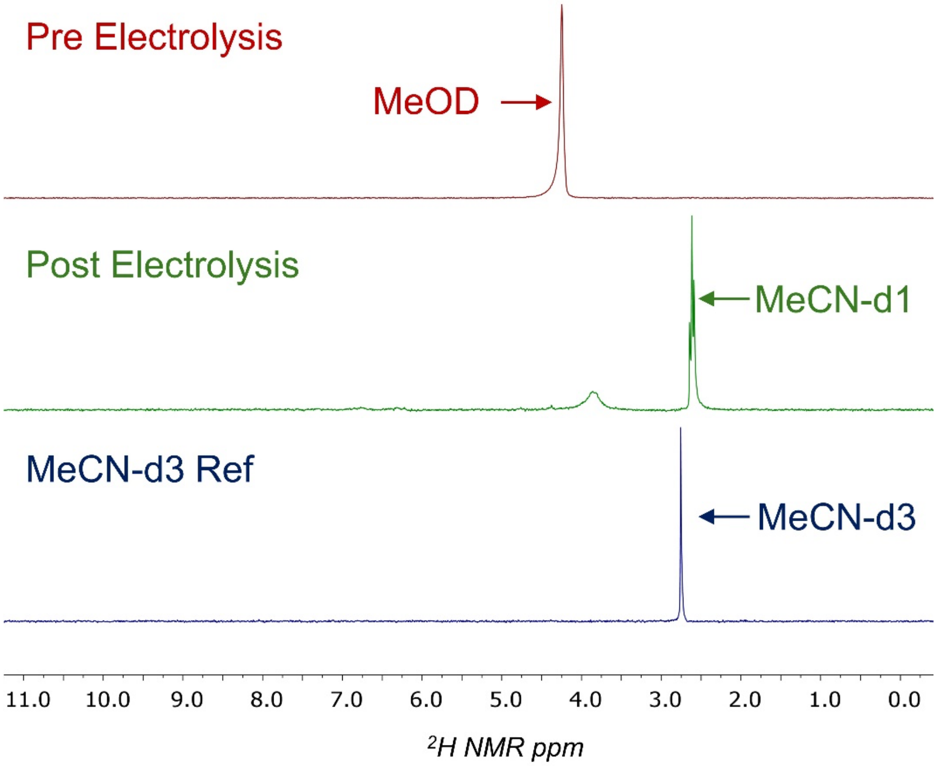


Figure S12: ^{2}H-NMR of a reaction with potassium 4-fluorophenyltrifluoroborate following General Procedure A pre-electrolysis (red spectrum) and post-electrolysis (green spectrum).

Reaction conditions: 100 mM potassium 4-fluorophenyltrifluoroborate, 200 mM $LiClO_4$, 400 mM Pyridine, 400 mM tetramethylammonium chloride, MeCN:MeOD (20:1), Pt Mesh cathode, RVC anode, 80° C, 20mA pulsing 60 s on 30 s off, 3 F/mol. The reaction was analyzed by ^{2}H NMR spectroscopy before and after electrolysis. Post-electrolysis (Green Spectrum), the MeOD resonance has diminished, and a new multiplet resonance appears around 2.6 ppm. We attribute the multiplet to a mixture of deuteria on MeCN. A true sample of MeCN-d3 (Blue Spectrum) shows the methyl deuterium resonance to be around 2.7 ppm. None of these spectra are normalized to a known chemical shift.

NMR analysis and CV Analysis of Aryl-Boronic acid-amine Lewis Acid-Base Adduct

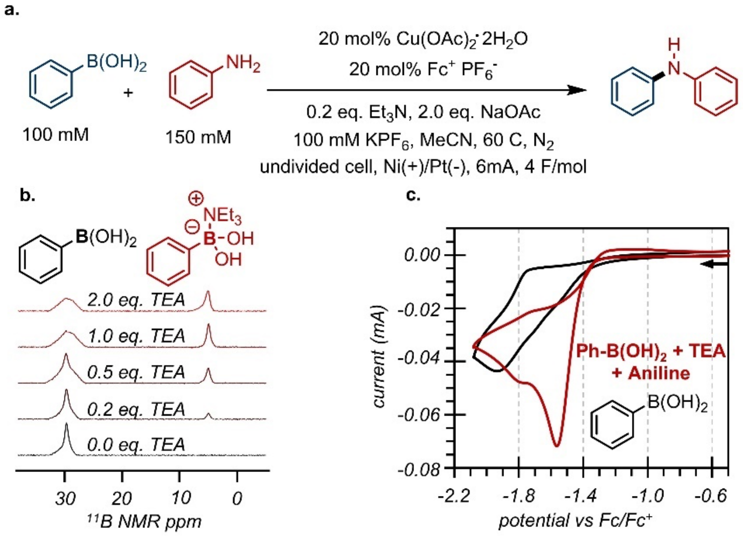


Figure S13: (a) Electrochemical Chan-Lam reaction scheme. (b) ^{11}B-NMR analysis of phenylboronic acid with increasing amounts of triethylamine (c) CV analysis (IUPAC convention) of the Lewis acid-base interaction of phenylboronic acid and amines.

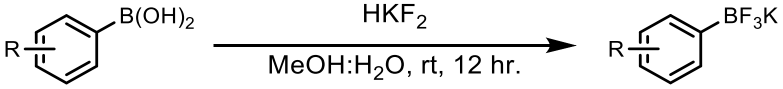
(a) Reaction scheme for the electrochemical Chan-Lam coupling reaction.[3]

(b) Five 4 mL vials were charged with phenylboronic acid (2 mg, 0.02 mmol) each. A stock solution of triethyl amine (15 mg, 0.15 mmol) in acetonitrile (2.8 mL, 53 mM) was added accordingly: Vial 1. 0.00 mL (0.0 eq.), Vial 2. 0.07 mL (0.2 eq.), Vial 3. 0.15 mL (0.5 eq.), Vial 4. 0.35 mL (1.0 eq.), and Vial 5. 0.7 mL (2.0 eq.). Acetonitrile was added to each vial until the total volume reached 0.7 mL. The solutions were then transferred to NMR tubes and analyzed via ^{11}B-NMR with $NaB(Ph)_4$ as internal standard. Increasing concentrations of triethylamine shows a new peak growing at 4.9 ppm accompanied by the disappearing of the boronic acid peak at 29.7 ppm.

(c) **CV conditions:** 25 °C, acetonitrile solvent sparged with N_2, 100 mM KPF_6 supporting electrolyte, Pt disk WE (ϕ 1.6 mm), Pt wire CE and Ag/Ag^+ quasi reference electrode. The electrochemical potentials are normalized to an internal ferrocene reference.
Black trace: 25 mM phenylboronic acid.
Red trace: 25 mM phenylboronic acid, 15 mM triethylamine, 15 mM aniline.
All scans started at 0.0 V vs Fc/Fc^+ and started scanning in the cathodic direction. Scan was reversed at -2.1 V vs Fc/Fc^+.

Experimental Section

General Procedure for Aryl-BF3K Synthesis



A 40 mL vial was charged with a stir bar, aryl boronic acid (3.73 mmol), HKF_2 (11.2 mmol), methanol (19 mL) and water (1 ml). The reaction was allowed to stir overnight at room temperature. The solvent was removed via rotary evaporation and the resulting solid was dissolved in 150 mL of boiling acetone. The boiling solution was filtered over a pad of diatomaceous earth, and the filtrate was concentrated to the cloud point via rotary evaporation. The solution was placed in the freezer to crystalize overnight. The resulting crystals were filtered and washed with diethyl ether to afford the target aryl trifluoroborate potassium salt as a white solid.

General Procedure A for Electrochemical Chloro-deborylation

In a nitrogen filled glovebox, aryl-BF_3K (0.30 mmol) was added to a 10 ml reaction vial along with a stir bar, pyridine (95 mg, 1.2 mmol, 4.0 equiv.), and acetonitrile (2.8 mL). The solution was allowed to stir for 1 minute and then lithium perchlorate (64 mg, 0.60 mmol, 2.0 equiv.) was added along with tetramethylammonium chloride (134 mg, 1.20 mmol, 4.00 equiv.). A platinum mesh cathode and RVC anode are placed in the reaction chamber, pierced through the septa, and sealed with a cap. The reaction was removed from the glove box and 1,1,1,2,2,2-hexafluoroisopropanol (HFIP) (0.15 mL, 5% /v) was added via syringe through the septa. The reaction vial was heated to 80 °C in an aluminum block and allowed to stir for 10 minutes to aid in the dissolution of the salts. Then, a 20 mA current was passed for 60 seconds with a rest period of 30 seconds in a pulsing fashion until 24.1 mAh (3 F/mol) capacity was achieved.

Safety Note: The electrochemical chlorodeborylation reactions are performed at 80 °C using acetonitrile (boiling point 82 °C) as solvent in a sealed reaction vessel. Heating solvents close to or past their boiling point in a sealed vessel poses a risk of pressure build up and potential explosion.

Following electrolysis, the reaction mixture was transferred to a separatory funnel and diluted with 150 mL of water. The solution was extracted with diethyl ether or pentane (3 x 25 ml), and the organic layers were combined and washed with 2 M HCl (only performed on substrates lacking acid sensitive functional groups) followed by a saturated brine solution (100 mL). The organic layer was collected and dried over anhydrous sodium sulfate. The organic solvents were removed via rotary evaporation and purified via flash column chromatography.

General Procedure B for Electrochemical Chloro-deborylation

In a nitrogen filled glovebox, aryl-BF_3K (0.30 mmol) was added to a 10 ml reaction vial along with a stir bar, pyridine (95 mg, 1.2 mmol, 4.0 equiv.), and acetonitrile (2.8 mL). The solution was allowed to stir for 1 minute and then lithium perchlorate (64 mg, 0.60 mmol, 2.0 equiv.) was added along with tetramethylammonium chloride (134 mg, 1.20 mmol, 4.00 equiv.). A platinum mesh cathode and RVC anode are placed in the reaction chamber, pierced through the septa, and sealed with a cap. The reaction was removed from the glove box and 1,1,1-trifluoroethanol (TFE) (0.15 mL, 5% /v) was added via syringe through the septa. The reaction vial was heated to 80 °C in an aluminum block and allowed to stir for 10 minutes to aid in the dissolution of the salts. Then, a 2.5 V of cell potential was held for 60 seconds with a rest period of 30 seconds in a pulsing fashion until 24.1 mAh (3F/mol) capacity was achieved.

Safety Note: The electrochemical chlorodeborylation reactions are performed at 80 °C using acetonitrile (boiling point 82 °C) as solvent in a sealed reaction vessel. Heating solvents close to or past their boiling point in a sealed vessel poses a risk of pressure build up and potential explosion.

Following electrolysis, the reaction mixture was transferred to a separatory funnel and diluted with 150 mL of water. The solution was extracted with diethyl ether or pentane (3 x 25 ml) and the organic layers were combined and washed with 2 M HCl (only performed on substrates lacking acid sensitive functional groups) followed by a saturated brine solution (100 mL). The organic layer was collected and dried over anhydrous sodium sulfate. The organic solvents were removed via rotary evaporation and purified via flash column chromatography.

General Procedure C for Electrochemical Chloro-deborylation

In a nitrogen filled glovebox, aryl-BF_3K (0.30 mmol) was added to a 10 ml reaction vial along with a stir bar, pyridine (24 mg, 0.30 mmol, 1.0 equiv.), and acetonitrile (2.8 mL). The solution was allowed to stir for 1 minute and then lithium perchlorate (64 mg, 0.60 mmol, 2.0 equiv.) was added along with tetramethylammonium chloride (134 mg, 1.20 mmol, 4.00 equiv.). A platinum mesh cathode and RVC anode are placed in the reaction chamber, pierced through the septa, and sealed with a cap. The reaction was removed from the glove box and 1,1,1-trifluoroethanol (TFE) (0.15 mL, 5% /v) and acetic acid (0.15 mL, 5% /v) was added via syringe through the septa. The reaction vial was heated to 80 °C in an aluminum block and allowed to stir for 10 minutes to aid in the dissolution of the salts. Then, a 20 mA current was passed for 60 seconds with a rest period of 30 seconds in a pulsing fashion until 24.1 mAh (3F/mol) capacity was achieved.

Safety Note: The electrochemical chlorodeborylation reactions are performed at 80 °C using acetonitrile (boiling point 82 °C) as solvent in a sealed reaction vessel. Heating solvents close to or past their boiling point in a sealed vessel poses a risk of pressure build up and potential explosion.

Following electrolysis, the reaction mixture was transferred to a separatory funnel and diluted with 150 mL of water. The solution was extracted with diethyl ether or pentane (3 x 25 ml) and the organic layers were combined and washed with 2 M HCl (only performed on substrates lacking acid sensitive functional groups) followed by a saturated brine solution (100 mL). The organic layer was collected and dried over anhydrous sodium sulfate. The organic solvents were removed via rotary evaporation and purified via flash column chromatography.

General Procedure D for Electrochemical Bromo-deborylation

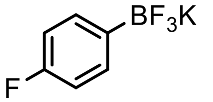
In a nitrogen filled glovebox, aryl-BF_3K (0.30 mmol) was added to a 10 ml reaction vial along with a stir bar, acetonitrile (2.8 mL), tetrabutylammonium tetrafluoroborate (98 mg, 0.30 mmol, 1.0 equiv.), ammonium bromide (118 mg, 1.20 mmol, 4.00 equiv.), and dodecane as internal standard. A platinum mesh cathode and RVC anode are placed in the reaction chamber, pierced through the septa, and sealed with a cap. The reaction was removed from the glove box and 1,1,1-trifluoroethanol (TFE) (0.15 mL, 5% /v) was added via syringe through the septa. The reaction vial was heated to 80 °C in an aluminum block and allowed to stir for 10 minutes to aid in the dissolution of the salts. Then, a 10 mA current was passed for 60 seconds with a rest period of 30 seconds in a pulsing fashion until 24.1 mAh (3F/mol) capacity was achieved.

Safety Note: The electrochemical bromodeborylation reactions are performed at 80 °C using acetonitrile (boiling point 82 °C) as solvent in a sealed reaction vessel. Heating solvents close to or past their boiling point in a sealed vessel poses a risk of pressure build up and potential explosion.

Following electrolysis, an aliquot of the solution was transferred to a test tube. The alliquot was diluted with ethyl acetate (2 mL) and washed with a saturated brine solution (2 mL). The organic phase was then transferred to a GC vial and analyzed via Gas Column Chromatography.

Characterization of Starting Materials

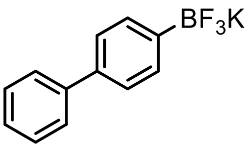
potassium trifluoro(4-fluorophenyl)borate (1a)



Following General Procedure for Aryl-BF3K Synthesis, 4-fluorophenylboronic acid (10.0 g, 71.5 mmol) was used as the aryl-B(OH)$_2$ reagent and the procedure was scaled accordingly. The title compound was isolated via crystallization in acetone. The title compound was obtained in 98% yield (14.1 g, 69.8 mmol) as a white solid. Characterization data match those of previously reported literature.[4]

^{1}H NMR (400 MHz, (CD$_3$)$_2$SO) δ 7.39 – 7.29 (m, 2H), 6.91 – 6.84 (m, 2H).
^{13}C NMR (101 MHz, (CD$_3$)$_2$SO) δ 162.2, 159.8, 132.8 (dq, *J* = 6.6, 1.7 Hz), 112.7 (d, *J* = 18.4 Hz).
^{19}F NMR (376 MHz, (CD$_3$)$_2$SO) δ -117.88 – -119.31 (m), -138.39 – -139.33 (m).
^{11}B NMR (128 MHz, DMSO-*d*$_6$) δ 3.57 (q, *J* = 56.4 Hz).

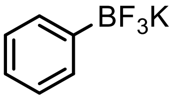
potassium [1,1'-biphenyl]-4-yltrifluoroborate (1b)



Following General Procedure for Aryl-BF3K Synthesis, 4-biphenylboronic acid (3.00 g, 15.2 mmol) was used as the aryl-B(OH)$_2$ reagent and the procedure was scaled accordingly. The title compound was isolated via crystallization in acetone. The title compound was obtained in 41% yield (1.24 mg, 6.14 mmol) as a white solid. Characterization data match those of previously reported literature.[5]

^{1}H NMR (400 MHz, (CD$_3$)$_2$SO) δ 7.63 – 7.55 (m, 2H), 7.45 – 7.36 (m, 6H), 7.33 – 7.25 (m, 1H).
^{13}C NMR (101 MHz, (CD$_3$)$_2$SO) δ 141.5, 136.8, 131.9 (q, *J* = 1.7 Hz), 128.7, 126.5, 126.3, 124.6.
^{19}F NMR (376 MHz, (CD$_3$)$_2$SO) δ -139.21.
^{11}B NMR (128 MHz, (CD$_3$)$_2$SO) δ 3.43.

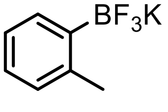
potassium trifluoro(phenyl)borate (1c)



Following General Procedure for Aryl-BF3K Synthesis, phenylboronic acid (455 mg, 3.73 mmol) was used as the aryl-B(OH)$_2$ reagent. The title compound was isolated via crystallization in acetone. The title compound was obtained in 91% yield (624 mg, 3.39 mmol) as a white solid. Characterization data match those of previously reported literature.[6]

^{1}H NMR (400 MHz, (CD$_3$)$_2$SO) δ 7.42 – 7.27 (m, 2H), 7.10 (t, *J* = 7.4 Hz, 2H), 7.06 – 7.00 (m, 1H).
^{13}C NMR (101 MHz, (CD$_3$)$_2$SO) δ 131.3, 126.2, 125.0.
^{19}F NMR (376 MHz, (CD$_3$)$_2$SO) δ -139.08.
^{11}B NMR (128 MHz, (CD$_3$)$_2$SO) δ 3.22.

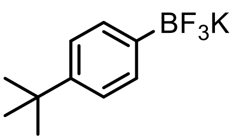
potassium trifluoro(o-tolyl)borate (1d)



Following General Procedure for Aryl-BF3K Synthesis, o-tolylboronic acid (880 mg, 6.47 mmol) was used as the aryl-B(OH)$_2$ reagent and the procedure was scaled accordingly. The title compound was isolated via crystallization in acetone. The title compound was obtained in 88% yield (1.13 g, 5.72 mmol) as a white solid. Characterization data match those of previously reported literature.[7]

^{1}H NMR (400 MHz, (CD$_3$)$_2$SO) δ 7.34 (dd, *J* = 7.5, 1.9 Hz, 1H), 6.96 – 6.90 (m, 1H), 6.89 (m, 1H), 6.88 – 6.85 (m, 1H), 2.31 (s, 3H).
^{13}C NMR (101 MHz, (CD$_3$)$_2$SO) δ 140.9, 132.1 (q, *J* = 3.1 Hz), 128.6, 125.5, 123.78, 22.1.
^{19}F NMR (376 MHz, (CD$_3$)$_2$SO) δ -136.44 – -139.02 (m).
^{11}B NMR (128 MHz, (CD$_3$)$_2$SO) δ 3.30.

potassium (4-(tert-butyl)phenyl)trifluoroborate (1e)



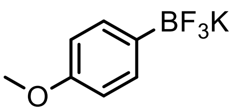
Following General Procedure for Aryl-BF_3K Synthesis, 4-tertbutylphenylboronic acid (2.67 g, 15.0 mmol) was used as the aryl-$B(OH)_2$ reagent and the procedure was scaled accordingly. The title compound was isolated via crystallization in acetone. The title compound was obtained in 71% yield (2.55 g, 10.6 mmol) as a white solid. Characterization data match those of previously reported literature.[8]

^{1}H NMR (400 MHz, $(CD_3)_2SO$) δ 7.28 – 7.21 (m, 2H), 7.13 – 7.08 (m, 2H), 1.25 (s, 9H).

^{13}C NMR (101 MHz, $(CD_3)_2SO$) δ 146.7, 131.1 (q, *J* = 1.7 Hz), 122.8, 33.9, 31.4.

^{19}F NMR (376 MHz, $(CD_3)_2SO$) δ -138.71 (bs).

potassium trifluoro(4-methoxyphenyl)borate (1f)



Following General Procedure for Aryl-BF_3K Synthesis, 4-methoxyphenylboronic acid (1.14 g, 7.50 mmol) was used as the aryl-$B(OH)_2$ reagent and the procedure was scaled accordingly. The title compound was isolated via crystallization in acetone. The title compound was obtained in 78% yield (1.25 g, 5.84 mmol) as a white solid. Characterization data match those of previously reported literature.[6]

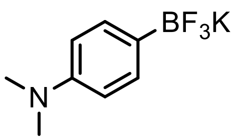
^{1}H NMR (400 MHz, $(CD_3)_2SO$) δ 7.23 (d, *J* = 8.3 Hz, 2H), 6.67 (d, *J* = 7.9 Hz, 2H), 3.68 (s, 3H).

^{13}C NMR (101 MHz, $(CD_3)_2SO$) δ 157.2, 132.2 (q, *J* = 1.7 Hz), 111.8, 54.5.

^{19}F NMR (376 MHz, $(CD_3)_2SO$) δ -138.21 (bs).

^{11}B NMR (128 MHz, DMSO-d_6) δ 3.56 (bs).

potassium (4-(dimethylamino)phenyl)trifluoroborate (1g)



Following General Procedure for Aryl-BF_3K Synthesis, 4-dimethylaminophenylboronic acid (825 mg, 5.00 mmol) was used as the aryl-$B(OH)_2$ reagent and the procedure was scaled accordingly. The title compound was isolated via crystallization in acetone. The title compound was obtained in 42% yield (472 mg, 2.08 mmol) as a white solid.

^{1}H NMR (400 MHz, CD_3CN) δ 7.26 (d, *J* = 8.1 Hz, 2H), 6.71 – 6.56 (m, 2H), 2.83 (s, 6H).

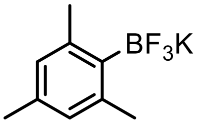
^{13}C NMR (101 MHz, CD_3CN) δ 132.7, 112.8, 40.9.

^{19}F NMR (376 MHz, CD_3CN) δ -141.16.

^{11}B NMR (128 MHz, CD_3CN) δ 5.12 – 1.75 (m).

HRMS (ESI-ion trap) (m/z): $[M]^-$ for $C_8H_{10}BF_3N^-$: calcd for 188.0864, found 181.0864.

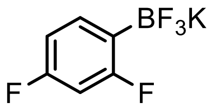
potassium trifluoro(mesityl)borate (1h)



Following General Procedure for Aryl-BF_3K Synthesis, mesitylboronic acid (670 mg, 4.09 mmol) was used as the aryl-$B(OH)_2$ reagent and the procedure was scaled accordingly. The title compound was isolated via crystallization in acetone. The title compound was obtained in 82% yield (759 mg, 3.36 mmol) as a white solid. Characterization data match those of previously reported literature.[9]

^{1}H NMR (400 MHz, CD_3CN) δ 6.60 (s, 2H), 2.32 (s, 6H), 2.15 (s, 3H).

potassium (2,4-difluorophenyl)trifluoroborate (1i)



Following General Procedure for Aryl-BF_3K Synthesis, 2,4-difluorophenylboronic acid (1.70 g, 10.8 mmol) was used as the aryl-$B(OH)_2$ reagent and the procedure was scaled accordingly. The title compound was isolated via crystallization in acetone. The title compound was obtained in 88% yield (2.07 g, 9.43 mmol) as a white solid.

^{1}H NMR (400 MHz, $(CD_3)_2SO$) δ 7.33 (q, *J* = 7.8 Hz, 1H), 6.73 (dtd, *J* = 18.6, 9.6, 9.1, 2.4 Hz, 2H).

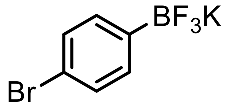
^{13}C NMR (101 MHz, $(CD_3)_2SO$) δ 165.4 (dd, *J* = 241.8, 10.8 Hz), 161.1 (dd, *J* = 240.8, 12.3 Hz), 135.4 – 134.3 (m), 109.1 (d, *J* = 18.1 Hz), 101.8 (dd, *J* = 30.7, 23.3 Hz).

^{19}F NMR (376 MHz, $(CD_3)_2SO$) δ -103.48, -115.85, -137.04.

^{11}B NMR (128 MHz, $(CD_3)_2SO$) δ 2.26 (d, *J* = 50.0 Hz).

HRMS (ESI-ion trap) (m/z): $[M]^-$ for $C_6H_3BF_5^-$: calcd for 181.0253, found 181.0254.

potassium (4-bromophenyl)trifluoroborate (1j)



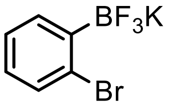
Following General Procedure for Aryl-BF3K Synthesis, 4-bromophenylboronic acid (3.01 g, 15.0 mmol) was used as the aryl-B(OH)2 reagent and the procedure was scaled accordingly. The title compound was isolated via crystallization in acetone. The title compound was obtained in 79% yield (3.10 g, 11.8 mmol) as a white solid. Characterization data match those of previously reported literature.[10]

^{1}H NMR (400 MHz, $(CD_3)_2SO$) δ 7.26 (s, 4H).

^{13}C NMR (101 MHz, $(CD_3)_2SO$) δ 133.6 (q, *J* = 1.7 Hz), 129.0, 118.47, 30.7.

^{19}F NMR (376 MHz, $(CD_3)_2SO$) δ -134.96 – -143.68 (m).

potassium (2-bromophenyl)trifluoroborate (1k)



Following General Procedure for Aryl-BF3K Synthesis, 2-bromophenylboronic acid (1.74 g, 8.66 mmol) was used as the aryl-B(OH)2 reagent and the procedure was scaled accordingly. The title compound was isolated via crystallization in acetone. The title compound was obtained in 63% yield (1.44 g, 5.46 mmol) as a white solid. Characterization data match those of previously reported literature.[11]

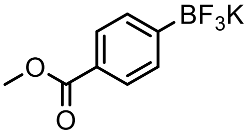
^{1}H NMR (400 MHz, $(CD_3)_2SO$) δ 7.43 (dd, *J* = 7.3, 2.0 Hz, 1H), 7.34 – 7.25 (m, 1H), 7.10 (tdd, *J* = 7.2, 1.1, 0.6 Hz, 1H), 6.96 (td, *J* = 7.5, 2.0 Hz, 1H).

^{13}C NMR (101 MHz, $(CD_3)_2SO$) δ 134.1 (q, *J* = 2.9 Hz), 131.4, 127.7, 127.4, 125.4.

^{19}F NMR (376 MHz, $(CD_3)_2SO$) δ -137.84 – -141.53 (m).

^{11}B NMR (128 MHz, $(CD_3)_2SO$) δ 2.35 (d, *J* = 52.3 Hz).

potassium trifluoro(4-(methoxycarbonyl)phenyl)borate (1l)



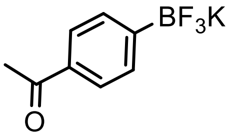
Following General Procedure for Aryl-BF3K Synthesis, 4-methoxycarbonylphenylboronic acid (900 mg, 5.00 mmol) was used as the aryl-B(OH)2 reagent and the procedure was scaled accordingly. The title compound was isolated via crystallization in acetone. The title compound was obtained in 60% yield (732 mg, 3.02 mmol) as a white solid. Characterization data match those of previously reported literature.[6]

^{1}H NMR (400 MHz, $(CD_3)_2SO$) δ 7.73 (dd, *J* = 8.2, 0.8 Hz, 2H), 7.47 (d, *J* = 8.1 Hz, 2H), 3.81 (s, 3H).

^{19}F NMR (376 MHz, $(CD_3)_2SO$) δ -139.92.

^{11}B NMR (128 MHz, $(CD_3)_2SO$) δ 2.92.

potassium (4-acetylphenyl)trifluoroborate (1m)



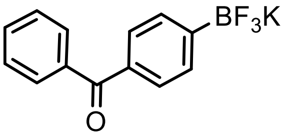
Following General Procedure for Aryl-BF3K Synthesis, 4-acetylphenylboronic acid (405 mg, 2.47 mmol) was used as the aryl-B(OH)2 reagent and the procedure was scaled accordingly. The title compound was isolated via crystallization in acetone. The title compound was obtained in 46% yield (258 mg, 1.14 mmol) as a white solid. Characterization data match those of previously reported literature.[10]

^{1}H NMR (400 MHz, $(CD_3)_2SO$) δ 7.74 – 7.68 (m, 2H), 7.46 (d, *J* = 8.1 Hz, 2H), 2.51 (s, 3H).

^{19}F NMR (376 MHz, $(CD_3)_2SO$) δ -139.78.

^{11}B NMR (128 MHz, $(CD_3)_2SO$) δ 2.76.

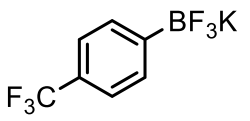
potassium (4-benzoylphenyl)trifluoroborate (1n)



Following General Procedure for Aryl-BF3K Synthesis, 4-benzoylphenylboronic acid (1.18 mg, 5.22 mmol) was used as the aryl-B(OH)2 reagent and the procedure was scaled accordingly. The title compound was isolated via crystallization in acetone. The title compound was obtained in 49% yield (733 mg, 2.54 mmol) as a white solid. Characterization data match those of previously reported literature.[6]

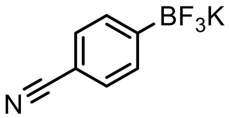
^{1}H NMR (400 MHz, $(CD_3)_2SO$) δ 7.73 – 7.67 (m, 2H), 7.67 – 7.61 (tt, 1H), 7.58 – 7.53 (m, 2H), 7.52 (d, *J* = 0.7 Hz, 4H).
^{13}C NMR (101 MHz, $(CD_3)_2SO$) δ 196.4, 138.0, 134.0, 132.0, 131.3, 129.4, 128.3, 128.1.
^{19}F NMR (376 MHz, $(CD_3)_2SO$) δ -139.85.
^{11}B NMR (128 MHz, $(CD_3)_2SO$) δ 2.84.

potassium trifluoro(4-(trifluoromethyl)phenyl)borate (1o)



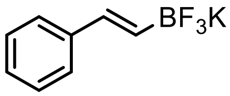
Following General Procedure for Aryl-BF_3K Synthesis, 4-trifluoromethylphenylboronic acid (1.42 g, 7.50 mmol) was used as the aryl-$B(OH)_2$ reagent and the procedure was scaled accordingly. The title compound was isolated via crystallization in acetone. The title compound was obtained in 92% yield (1.73 g, 6.86 mmol) as an off white solid. Characterization data match those of previously reported literature.[6]
^{1}H NMR (400 MHz, $(CD_3)_2SO$) δ 7.55 (d, *J* = 7.7 Hz, 2H), 7.43 (d, *J* = 8.0 Hz, 2H).
^{19}F NMR (376 MHz, $(CD_3)_2SO$) δ -60.48, -140.01.

potassium (4-cyanophenyl)trifluoroborate (1p)



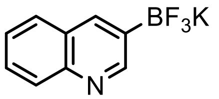
Following General Procedure for Aryl-BF_3K Synthesis, 4-cyanophenylboronic acid (667 mg, 4.00 mmol) was used as the aryl-$B(OH)_2$ reagent and the procedure was scaled accordingly. The title compound was isolated via crystallization in acetone. The title compound was obtained in 73% yield (614 mg, 2.94 mmol) as a white solid. Characterization data match those of previously reported literature.[7]
^{1}H NMR (400 MHz, $(CD_3)_2SO$) δ 7.57 – 7.43 (m, 4H).
^{13}C NMR (101 MHz, $(CD_3)_2SO$) δ 132.0, 132.0, 130.0, 120.1, 107.7.
^{19}F NMR (376 MHz, $(CD_3)_2SO$) δ -140.41 (bs).
^{11}B NMR (128 MHz, $(CD_3)_2SO$) δ 4.80 – -0.51 (m).

potassium (E)-trifluoro(styryl)borate (1q)



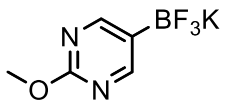
Following General Procedure for Aryl-BF_3K Synthesis, (E)-styrylboronic acid (1100 mg, 7.50 mmol) was used as the aryl-$B(OH)_2$ reagent and the procedure was scaled accordingly. The title compound was isolated via crystallization in acetone. The title compound was obtained in 33% yield (520 mg, 2.48 mmol) as a white solid. Characterization data match those of previously reported literature.[12]
^{1}H NMR (400 MHz, $(CD_3)_2SO$) δ 7.31 (dd, *J* = 8.2, 1.5 Hz, 2H), 7.25 (dd, *J* = 8.4, 6.9 Hz, 2H), 7.14 – 7.07 (m, 1H), 6.47 (d, *J* = 18.8 Hz, 1H), 6.18 (dq, *J* = 18.2, 3.5 Hz, 1H).

potassium trifluoro(quinolin-3-yl)borate (1r)



Following General Procedure for Aryl-BF_3K Synthesis, quinolin-3-ylboronic acid (1.30 g, 7.50 mmol) was used as the aryl-$B(OH)_2$ reagent and the procedure was scaled accordingly. The title compound was isolated via crystallization in acetone. The title compound was obtained in 82% yield (1.45 g, 6.17 mmol) as a white solid. Characterization data match those of previously reported literature.[13]
^{1}H NMR (400 MHz, $(CD_3)_2SO$) δ 8.87 (s, 1H), 8.12 (s, 1H), 7.90 (d, *J* = 8.3 Hz, 1H), 7.83 (d, *J* = 7.9 Hz, 1H), 7.63 – 7.52 (m, 1H), 7.53 – 7.39 (m, 1H).
^{13}C NMR (101 MHz, $(CD_3)_2SO$) δ 155.2 (q, *J* = 1.4 Hz), 146.7, 137.5 (q, *J* = 2.1 Hz), 128.4, 128.2, 127.6, 127.4, 125.1.
^{19}F NMR (376 MHz, $(CD_3)_2SO$) δ -138.80 (bs).
^{11}B NMR (128 MHz, $(CD_3)_2SO$) δ 3.20 (bs).

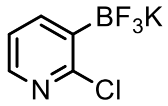
potassium trifluoro(2-methoxypyrimidin-5-yl)borate (1s)



Following General Procedure for Aryl-BF$_3$K Synthesis, 2-methoxypyrimidin-5-ylboronic acid (615 mg, 4.00 mmol) was used as the aryl-B(OH)$_2$ reagent and the procedure was scaled accordingly. The title compound was isolated via crystallization in acetone. The title compound was obtained in 53% yield (460 mg, 2.13 mmol) as a white solid. Characterization data match those of previously reported literature.[14]

^{1}H NMR (400 MHz, (CD$_3$)$_2$SO) δ 8.34 (s, 2H), 3.83 (s, 3H).
^{13}C NMR (101 MHz, (CD$_3$)$_2$SO) δ 164.1, 161.7 (q, *J* = 1.8 Hz), 53.6.
^{19}F NMR (376 MHz, (CD$_3$)$_2$SO) δ -138.36 (bs).
^{11}B NMR (128 MHz, (CD$_3$)$_2$SO) δ 3.66 – 2.00 (m).

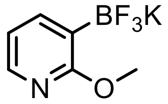
potassium (2-chloropyridin-3-yl)trifluoroborate (1t)



Following General Procedure for Aryl-BF$_3$K Synthesis, 2-chloropyridin-3-ylboronic acid (1.17 g, 7.44 mmol) was used as the aryl-B(OH)$_2$ reagent and the procedure was scaled accordingly. The title compound was isolated via crystallization in acetone. The title compound was obtained in 91% yield (1.49 g, 6.79 mmol) as a white solid.

^{1}H NMR (400 MHz, (CD$_3$)$_2$SO) δ 8.08 (dd, *J* = 4.7, 2.3 Hz, 1H), 7.75 (dd, *J* = 7.2, 2.3 Hz, 1H), 7.17 – 7.08 (m, 1H).
^{13}C NMR (101 MHz, (CD$_3$)$_2$SO) δ 155.3, 147.2, 143.2, 122.1.
^{19}F NMR (376 MHz, (CD$_3$)$_2$SO) δ -138.53 – -142.93 (m).
^{11}B NMR (128 MHz, (CD$_3$)$_2$SO) δ 2.02 (q, *J* = 47.9 Hz).
HRMS (ESI-ion trap) (m/z): [M]$^-$ for $C_5H_3BClF_3N^-$: calcd for 179.9999, found 180.0005.

potassium trifluoro(2-methoxypyridin-3-yl)borate (1u)

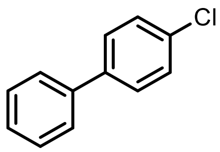


Following General Procedure for Aryl-BF$_3$K Synthesis, 2-methoxypyridin-3-ylboronic acid (1.15 g, 7.50 mmol) was used as the aryl-B(OH)$_2$ reagent and the procedure was scaled accordingly. The title compound was isolated via crystallization in acetone. The title compound was obtained in 78% yield (1.26 g, 5.86 mmol) as a white solid.

^{1}H NMR (400 MHz, (CD$_3$)$_2$SO) δ 7.87 (dd, *J* = 5.0, 2.2 Hz, 1H), 7.56 (dd, *J* = 6.8, 2.2 Hz, 1H), 6.77 – 6.61 (m, 1H).
^{13}C NMR (101 MHz, (CD$_3$)$_2$SO) δ 166.8, 144.0, 142.0, 116.4, 52.6.
^{19}F NMR (376 MHz, (CD$_3$)$_2$SO) δ -138.40.
^{11}B NMR (128 MHz, (CD$_3$)$_2$SO) δ 2.48 (d, *J* = 50.9 Hz).
HRMS (ESI-ion trap) (m/z): [M]$^-$ for $C_6H_6BF_3NO^-$: calcd for 176.0495, found 176.0500.

Characterization of Isolated Products

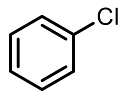
4-chloro-1,1'-biphenyl (2b):



General Procedure A was used but was scaled up to include potassium [1,1'-biphenyl]-4-yltrifluoroborate (260 mg, 1.00 mmol), pyridine (316 mg, 4.00 mM, 4.00 eq.), lithium perchlorate (212 mg, 2.00 mmol, 2.00 eq.), tetramethylammonium chloride (438 mg, 4.00 mmo, 4.00 eq.) in 9.5 mL acetonitrile and 0.5 mL HFIP (5% /v). The title compound was extracted with pentane (50 ml x 3) from 2M HCl (100 mL). The organic phase was washed with saturated brine solution (100 mL) and dried over anhydrous sodium sulfate. The solvent was removed under reduced pressure. The title compound was obtained in 84% yield (158 mg, 0.838 mmol) as a white solid. Characterization data match those of previously reported literature.[16]

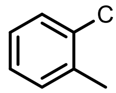
^{1}H NMR (400 MHz, CDCl$_3$) δ 7.60 – 7.49 (m, 4H), 7.49 – 7.40 (m, 4H), 7.40 – 7.34 (m, 1H).
^{13}C{^{1}H} NMR (101 MHz, CDCl$_3$) δ 140.1, 139.8, 133.5, 129.0, 129.0, 128.5, 127.7, 127.1.

chlorobenzene (2c):



Following General Procedure A, potassium trifluoro(phenyl)borate (55 mg, 0.30 mmol) was used as the aryl-BF_3K reagent. The product yield was determined by gas chromatography using dodecane as the internal standard (97%) due to the low boiling point of the product. GC analysis of the title compound reaction mixture agreed with the Gas Chromatography spectra of a commercially available sample.

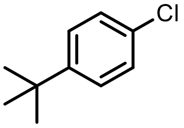
1-chloro-2-methylbenzene (2d):



Following General Procedure B, potassium trifluoro(o-tolyl)borate (59 mg, 0.30 mmol) was used as the aryl-BF_3K reagent. The title compound was extracted with pentane and isolated via flash column chromatography (100% hexane). The title compound was obtained in 90% yield (34 mg, 0.28 mmol) as a clear liquid. Characterization data match those of previously reported literature.[15]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.31 (m, 1H), 7.24 – 7.20 (m, 1H), 7.18 – 7.09 (m, 2H), 2.38 (s, 3H).
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 136.2, 134.5, 131.1, 129.2, 127.2, 126.7, 20.2.

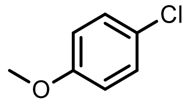
1-(tert-butyl)-4-chlorobenzene (2e):



Following General Procedure B, potassium (4-(tert-butyl)phenyl)trifluoroborate (72 mg, 0.30 mmol) was used as the aryl-BF_3K reagent. The title compound was extracted with pentane and isolated via flash column chromatography (100% hexane). The title compound was obtained in 89% yield (45 mg, 0.27 mmol) as a yellow liquid. Characterization data match those of previously reported literature.[16]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27 – 7.20 (m, 2H), 7.20 – 7.14 (m, 2H), 1.22 (s, 9H).
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 149.7, 131.3, 128.2, 126.9, 34.6, 31.4.

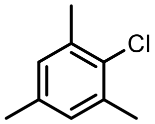
4-chloroanisole (2f):



Following General Procedure B, potassium 4-methoxyphenyltrifluoroborate (64 mg, 0.30 mmol) was used as the aryl-BF_3K reagent. The solution was electrolyzed until 12 mAh (1.5 F/mol) of capacity was achieved. The title compound was extracted with pentane and isolated via flash column chromatography (100% hexane). The title compound was obtained in 63% yield (27 mg, 0.19 mmol) as a yellow liquid. Characterization data match those of previously reported literature.[15]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 – 7.21 (m, 2H), 6.86 – 6.80 (m, 2H), 3.79 (s, 3H).
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 158.3, 129.4, 125.7, 115.3, 55.6.

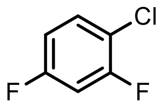
2-chloro-1,3,5-trimethylbenzene (2h):



Following General Procedure A, potassium trifluoro(mesityl)borate (68 mg, 0.30 mmol) was used as the aryl-BF_3K reagent. The title compound was extracted with pentane and isolated via flash column chromatography (100% hexane). The title compound was obtained in 54% yield (25 mg, 0.16 mmol) as a clear liquid. Characterization data match those of previously reported literature.[17]

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.90 (s, 2H), 2.35 (s, 6H), 2.27 (s, 3H).
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 136.0, 135.7, 129.3, 77.2, 20.8, 20.7.

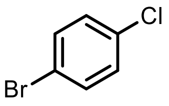
1-chloro-2,4-difluorobenzene (2i):



Following General Procedure A, potassium trifluoro(phenyl)borate (66 mg, 0.30 mmol) was used as the aryl-BF_3K reagent. The product yield was determined by gas chromatography using dodecane as the internal standard (92%) due to the low boiling point of the product. ^{19}F NMR was used to confirm the identity of **2i** with previously reported literature.[18]

^{19}F NMR (376 MHz, $CDCl_3$) δ -110.54 , -111.31.

1-bromo-4-chlorobenzene (2j):

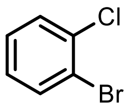


Following General Procedure A, potassium (4-bromophenyl)trifluoroborate (79 mg, 0.30 mmol) was used as the aryl-BF_3K reagent. The title compound was extracted with pentane and isolated via flash column chromatography (100% hexane). The title compound was obtained in 73% yield (42 mg, 0.22 mmol) as a yellow liquid. Characterization data match those of previously reported literature.[19]

^1H NMR (400 MHz, $CDCl_3$) δ 7.46 – 7.38 (m, 2H), 7.25 – 7.17 (m, 2H).

$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 133.4, 132.9, 130.3, 120.4.

1-bromo-2-chlorobenzene (2k):

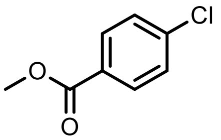


Following General Procedure B, potassium (2-bromophenyl)trifluoroborate (79 mg, 0.30 mmol) was used as the aryl-BF_3K reagent. The title compound was extracted with pentane and isolated via flash column chromatography (100% hexane). The title compound was obtained in 89% yield (51 mg, 0.27 mmol) as a yellow liquid. Characterization data match those of previously reported literature.[19]

^1H NMR (400 MHz, $CDCl_3$) δ 7.62 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.45 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.25 (ddd, *J* = 8.0, 7.4, 1.5 Hz, 1H), 7.12 (ddd, *J* = 8.0, 7.4, 1.6 Hz, 1H).

$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 134.6, 133.9, 130.6, 128.6, 128.0, 122.7.

methyl 4-chlorobenzoate (2l):



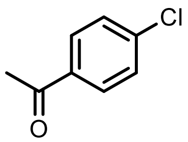
Following General Procedure C, potassium trifluoro(4-(methoxycarbonyl)phenyl)borate (73 mg, 0.30 mmol) was used as the aryl-BF_3K reagent. The title compound was extracted with pentane:diethyl ether (3:1) and isolated via flash column chromatography (30% EtOAc in Hexane). The title compound was obtained in 49% yield (25 mg, 0.15 mmol) as a white solid.

1H NMR (400 MHz, $CDCl_3$) δ 8.28 (d, *J* = 7.9 Hz, 2H), 8.16 (d, *J* = 8.0 Hz, 2H), 3.97 (s, 3H).

$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 167.1, 133.9, 133.7, 129.1, 52.5.

HRMS (ESI-TOF) (m/z): $[M+H]^+$ for $C_8H_8ClO_2$: calcd for 171.0213, found 171.0207.

1-(4-chlorophenyl)ethan-1-one (2m):



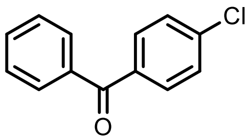
Following General Procedure B, potassium (4-acetylphenyl)trifluoroborate (68 mg, 0.30 mmol) was used as the aryl-BF_3K reagent. The title compound was extracted with pentane and isolated via flash column chromatography (30% EtOAc in Hexane). The title compound was obtained in 39% yield (18 mg, 0.12 mmol) as a clear liquid.

1H NMR (400 MHz, $CDCl_3$) δ 7.94 – 7.85 (dt, 2H), 7.48 – 7.40 (dt, 2H), 2.59 (s, 3H).

$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 197.0, 139.7, 135.6, 129.9, 129.1, 26.7.

HRMS (ESI-TOF) (m/z): $[M+H]^+$ for C_8H_8ClO: calcd for 155.0263, found 155.0257.

(4-chlorophenyl)(phenyl)methanone (2n):



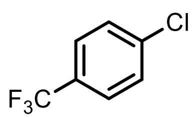
Following General Procedure A, potassium (4-benzoylphenyl)trifluoroborate (86 mg, 0.30 mmol) was used as the aryl-BF_3K reagent. The title compound was extracted with diethyl ether and isolated via flash column chromatography (20% Acetone in Pentane). The title compound was obtained in 63% yield (41 mg, 0.19 mmol) as a brown solid.

1H NMR (400 MHz, $CDCl_3$) δ 7.86 – 7.69 (m, 4H), 7.64 – 7.55 (tt, 1H), 7.55 – 7.41 (m, 4H).

$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 195.6, 139.0, 137.4, 136.0, 132.8, 131.6, 130.1, 128.8, 128.5.

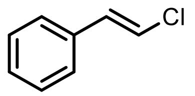
HRMS (ESI-TOF) (m/z): $[M+H]^+$ $C_{13}H_{10}ClO$: calcd for 217.0420, found 217.0412.

1-chloro-4-(trifluoromethyl)benzene (2o):



Following General Procedure C, potassium trifluoro(4-(trifluoromethyl)phenyl)borate (76 mg, 0.30 mmol) was used as the aryl-BF_3K reagent. The product yield was determined by gas chromatography using dodecane as the internal standard (59%) due to the low boiling point of the product.

(E)-(2-chlorovinyl)benzene (2q):

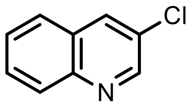


Following General Procedure A, potassium (E)-trifluoro(styryl)borate (63 mg, 0.30 mmol) was used as the aryl-BF_3K reagent. The title compound was extracted with pentane and isolated via flash column chromatography (100% hexane). The title compound was obtained in 60% yield (25 mg, 0.18 mmol) as a yellow liquid. Characterization data match those of previously reported literature.[20]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 – 7.18 (m, 5H), 6.80 (d, *J* = 13.7 Hz, 1H), 6.60 (d, *J* = 13.7 Hz, 1H)

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 135.0, 133.4, 128.9, 128.3, 126.3, 118.8.

3-chloroquinoline (2r):



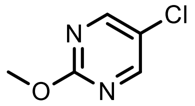
Following General Procedure A, potassium trifluoro(quinolin-3-yl)borate (71 mg, 0.30 mmol) was used as the aryl-BF_3K reagent. The title compound was extracted with 1:1 pentane:diethyl ether and isolated via flash column chromatography (50% diethyl ether in pentane). The title compound was obtained in 53% yield (26 mg, 0.16 mmol) as a clear liquid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.82 (d, *J* = 2.4 Hz, 1H), 8.13 (dd, *J* = 2.5, 0.8 Hz, 1H), 8.10 (dq, *J* = 8.4, 1.0 Hz, 1H), 7.75 (dd, *J* = 8.1, 1.4 Hz, 1H), 7.71 (ddd, *J* = 8.4, 6.9, 1.5 Hz, 1H), 7.58 (ddd, *J* = 8.1, 6.9, 1.2 Hz, 1H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 149.8, 146.4, 134.1, 129.8, 129.6, 128.6, 127.8, 127.1.

HRMS (ESI-TOF) (m/z): $[M+H]^+$ C_9H_7ClN calcd for 164.0267, found 164.0263.

5-chloro-2-methoxypyrimidine (2s):



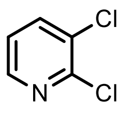
Following General Procedure A, potassium trifluoro(2-methoxypyrimidin-5-yl)borate (65 mg, 0.30 mmol) was used as the aryl-BF_3K reagent. The title compound was extracted with 5:1 pentane:diethyl ether and isolated via flash column chromatography (20% diethyl ether in pentane). The title compound was obtained in 41% yield (18 mg, 0.12 mmol) as an off-white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.45 (s, 2H), 4.00 (s, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 164.1, 157.6, 124.2, 55.6.

HRMS (ESI-TOF) (m/z): $[M+H]^+$ for $C_5H_6ClN_2O$ calcd for 145.0168, found 145.0164.

2,3-dichloropyridine (2t):



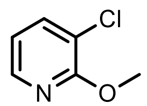
Following General Procedure B, potassium (2-chloropyridin-3-yl)trifluoroborate (66 mg, 0.30 mmol) was used as the aryl-BF_3K reagent. The title compound was extracted with pentane and isolated via flash column chromatography (100% pentane). The title compound was obtained in 17% yield (8 mg, 0.05 mmol) as a yellow liquid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.31 (dd, *J* = 4.7, 1.7 Hz, 1H), 7.78 (dd, *J* = 7.9, 1.7 Hz, 1H), 7.21 (dd, *J* = 7.9, 4.7 Hz, 1H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 149.5, 147.5, 138.9, 130.9, 123.4.

HRMS (ESI-TOF) (m/z): $[M+H]^+$ $C_5H_4Cl_2N$ calcd for 147.9721, found 147.9716.

3-chloro-2-methoxypyridine (2u):



Following General Procedure B, potassium trifluoro(2-methoxypyridin-3-yl)borate (65 mg, 0.30 mmol) was used as the aryl-BF$_3$K reagent. The title compound was extracted with 1:1 pentane:diethyl ether and isolated via flash column chromatography (100% pentane). The title compound was obtained in 30% yield (13 mg, 0.09 mmol) as a yellow liquid.

^{1}H NMR (400 MHz, CDCl$_3$) δ 8.06 (dd, *J* = 4.9, 1.7 Hz, 1H), 7.62 (dd, *J* = 7.6, 1.7 Hz, 1H), 6.84 (dd, *J* = 7.6, 4.9 Hz, 1H), 4.02 (s, 3H).

^{13}C{^{1}H} NMR (101 MHz, CDCl$_3$) δ 159.4, 144.7, 138.2, 118.3, 117.4, 54.2.

HRMS (ESI-TOF) (m/z): [M+H]$^+$ C$_6$H$_7$ClNO calcd for 144.0216, found 144.0210.

Sample Preparation for 4-fluorophenyl-BF$_2$-Py complex (3a)

In a nitrogen filled glove box, a 4 mL vial was charged with a stir bar, potassium 4-fluorophenyl trifluoroborate (20 mg, 0.10 mmol, 1.0 equiv.), pyridine (9.0 mg, 0.11 mmol, 1.1 equiv.), and 1.0 mL of deuterated acetonitrile (MeCN-d3). The solution was allowed to stir until all solids dissolved. Lithium perchlorate (85 mg, 0.80 mmol, 8.0 equiv.) was added and the solution allowed to stir for five minutes. The solution was then filtered through a plug of celite into an NMR tube and analyzed via ^{1}H, ^{19}F, and ^{11}B NMR.

^{1}H NMR (400 MHz, CD$_3$CN) δ 8.76 – 8.68 (m, 2H), 8.32 – 8.17 (m, 1H), 7.83 – 7.74 (m, 2H), 7.52 (dd, *J* = 8.2, 6.2 Hz, 2H), 7.07 – 6.96 (m, 2H).

^{19}F NMR (376 MHz, CH$_3$CN) δ -115.85, -154.97.

^{11}B NMR (128 MHz, CH$_3$CN) δ 6.24 (t, *J* = 54.5 Hz).

Sample Preparation for 4-biphenyl-BF$_2$-Py Crystal (3b)

In a nitrogen filled glove box, a 40 mL vial was charged with a stir bar, 312 mg of potassium 4-biphenyl-trifluoroborate (312 mg, 1.20 mmol, 1.00 equiv.), and 12 mL of dichloromethane. Boron trifluoride diethyl etherate (170 mg, 1.20 mmol, 1.00 equiv.) was added dropwise with stirring followed by pyridine (95 mg, 1.20 mmol, 1.00 equiv.). The solution was allowed to stir overnight at room temperature. 16 mL of a 1:1:1 mixture of dichloromethane:acetone:pentane was added to crash out the potassium tetrafluoroborate salt. The solution was then filtered through a pad of celite. The filtrate was collected and placed in the freezer to crystallize.

^{1}H NMR (400 MHz, CDCl$_3$) δ 8.82 (dd, *J* = 6.5, 1.4 Hz, 2H), 8.13 (t, *J* = 7.7 Hz, 1H), 7.70 (t, *J* = 7.0 Hz, 2H), 7.64 (d, *J* = 8.0 Hz, 2H), 7.59 (dd, *J* = 8.3, 1.2 Hz, 2H), 7.54 (d, *J* = 8.0 Hz, 2H), 7.42 (t, *J* = 7.6 Hz, 2H), 7.32 (t, *J* = 7.4 Hz, 1H).

^{13}C NMR (101 MHz, CDCl$_3$) δ 143.9, 142.1, 141.7, 140.6, 132.1, 128.8, 127.2, 127.1, 126.6, 126.0.

^{19}F NMR (376 MHz, CDCl$_3$) δ -155.20.

^{11}B NMR (128 MHz, CDCl$_3$) δ 5.97.

X-Ray Crystallography of 4-biphenyldifluoropyridiniumborate (3b)

Experimental Summary

The single crystal X-ray diffraction studies were carried out on a Bruker Kappa Photon III CPAD diffractometer equipped with Mo K_α radiation (λ = 0.71073 Å). A 0.168 x 0.111 x 0.106 mm piece of a colorless block was mounted on a MiTeGen MicroMount with Paratone 24EX oil. Data were collected in a nitrogen gas stream at 100(2) K using ϕ and ϖ scans. Crystal-to-detector distance was 50 mm using variable exposure time (3s-10s) depending on θ with a scan width of 1.0°. Data collection was 100% complete to 25.00° in θ (0.83Å). A total of 42338 reflections were collected covering the indices, -15<=h<=15, -17<=k<=17, -10<=l<=10. 2890 reflections were found to be symmetry independent, with a R_{int} of 0.0608. Indexing and unit cell refinement indicated a primitive, monoclinic lattice. The space group was found to be $P2_1/c$. The data were integrated using the Bruker SAINT software program and scaled using the SADABS software program. Solution by dual-space method (SHELXT) produced a complete phasing model for refinement.

All nonhydrogen atoms were refined anisotropically by full-matrix least-squares (SHELXL-2014). All hydrogen atoms were placed using a riding model. Their positions were constrained relative to their parent atom using the appropriate HFIX command in SHELXL-2014. Crystallographic data are summarized in Table S5.

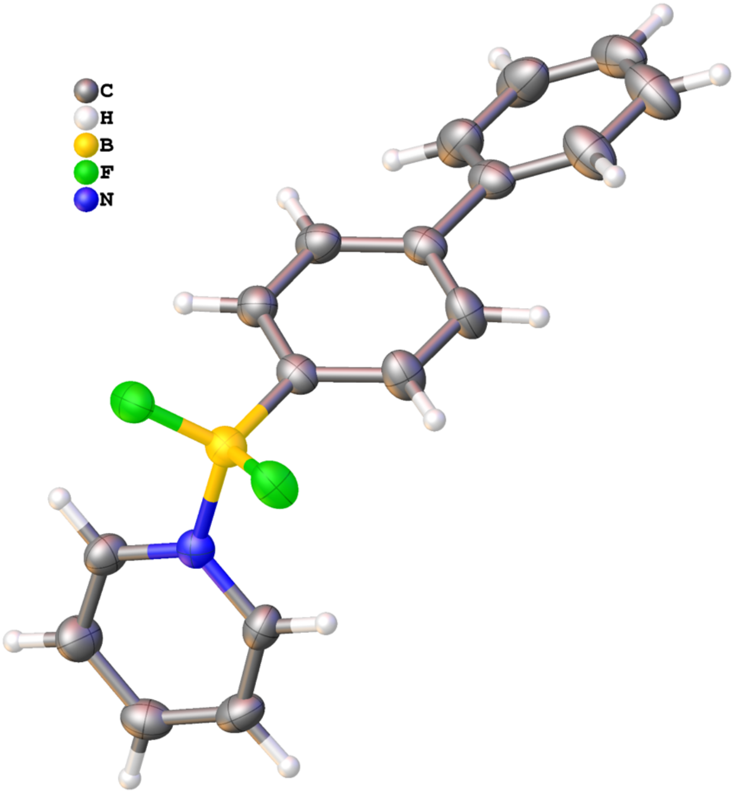


Figure S14: X-ray crystal structure of 4-biphenyl-BF_2-Pyridine complex. Ellipsoid contour 50% probability.

Table S5. Crystal data and structure refinement for **3b**.

Report date	2024-08-05	
Identification code	RES-0343	
Empirical formula	C17.50 H15 B Cl F2 N	
Molecular formula	C17 H14 B F2 N, 0.5(C H2 Cl2)	
Formula weight	323.56	
Temperature	100.0 K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 1 21/c 1	
Unit cell dimensions	a = 12.5824(5) Å	α= 90°.
	b = 14.5804(6) Å	β= 107.6260(10)°.
	c = 9.0368(4) Å	γ = 90°.
Volume	1580.03(11) Å^3	
Z	4	
Density (calculated)	1.360 Mg/m^3	
Absorption coefficient	0.258 mm^{-1}	
F(000)	668	
Crystal size	0.168 x 0.111 x 0.106 mm^3	
Crystal color, habit	colorless block	
Theta range for data collection	2.199 to 25.349°.	
Index ranges	-15<=h<=15, -17<=k<=17, -10<=l<=10	
Reflections collected	42338	
Independent reflections	2890 [R(int) = 0.0608, R(sigma) = 0.0216]	
Completeness to theta = 25.000°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.0916 and 0.0696	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2890 / 26 / 232	
Goodness-of-fit on F^2	1.036	
Final R indices [I>2sigma(I)]	R1 = 0.0392, wR2 = 0.0958	
R indices (all data)	R1 = 0.0555, wR2 = 0.1077	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.235 and -0.267 e.Å^{-3}	

Table S6. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for **3b**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
F(1)	-317(1)	7512(1)	8221(1)	34(1)
F(2)	0(1)	6239(1)	9789(1)	36(1)
N(1)	-558(1)	6022(1)	7016(2)	26(1)
C(1)	1454(1)	6645(1)	8512(2)	29(1)
C(2)	1846(2)	7234(1)	7576(2)	33(1)
C(3)	2953(2)	7233(1)	7593(2)	35(1)
C(4)	3726(2)	6637(1)	8546(2)	32(1)
C(5)	3345(2)	6044(2)	9482(2)	38(1)
C(6)	2235(2)	6055(1)	9464(2)	37(1)
C(7)	4916(2)	6621(1)	8563(2)	36(1)
C(8)	5229(2)	6915(1)	7297(3)	45(1)
C(9)	6336(2)	6891(1)	7307(3)	52(1)
C(10)	7148(2)	6575(2)	8585(3)	49(1)
C(11)	6862(2)	6277(2)	9848(3)	54(1)
C(12)	5755(2)	6300(2)	9841(2)	48(1)
C(13)	-1132(2)	6418(1)	5675(2)	33(1)
C(14)	-1743(2)	5912(1)	4422(2)	40(1)
C(15)	-1755(2)	4967(1)	4538(2)	38(1)
C(16)	-1150(2)	4559(1)	5916(2)	39(1)
C(17)	-569(2)	5101(1)	7135(2)	34(1)
B(1)	176(2)	6645(1)	8477(2)	29(1)
Cl(1S)	5245(8)	5646(5)	13775(8)	58(1)
Cl(2S)	5027(8)	4475(4)	16301(7)	58(1)
C(1S)	4310(8)	5218(11)	14762(16)	50(2)
Cl(1B)	5230(18)	5837(7)	13837(13)	58(1)
Cl(2B)	5546(12)	4479(7)	16323(8)	58(1)
C(1SB)	4536(17)	5130(30)	14940(40)	50(2)
Cl(1C)	4280(20)	5731(13)	13980(30)	58(1)
Cl(2C)	6004(16)	4537(13)	15970(20)	58(1)
C(1SC)	4540(20)	4810(30)	15390(60)	50(2)

Table S7. Bond lengths [Å] and angles [°] for **3b**.

F(1)-B(1)	1.396(2)	C(16)-H(16)	0.9500
F(2)-B(1)	1.401(2)	C(16)-C(17)	1.373(3)
N(1)-C(13)	1.339(2)	C(17)-H(17)	0.9500
N(1)-C(17)	1.348(2)	Cl(1S)-C(1S)	1.791(8)
N(1)-B(1)	1.638(2)	Cl(2S)-C(1S)	1.778(9)
C(1)-C(2)	1.397(3)	C(1S)-H(1SA)	0.9900
C(1)-C(6)	1.391(3)	C(1S)-H(1SB)	0.9900
C(1)-B(1)	1.598(3)	Cl(1B)-C(1SB)	1.826(16)
C(2)-H(2)	0.9500	Cl(2B)-C(1SB)	1.767(17)
C(2)-C(3)	1.389(3)	C(1SB)-H(1SE)	0.9900
C(3)-H(3)	0.9500	C(1SB)-H(1SF)	0.9900
C(3)-C(4)	1.391(3)	Cl(1C)-C(1SC)	1.82(2)
C(4)-C(5)	1.392(3)	Cl(2C)-C(1SC)	1.794(19)
C(4)-C(7)	1.493(2)	C(1SC)-H(1SC)	0.9900
C(5)-H(5)	0.9500	C(1SC)-H(1SD)	0.9900
C(5)-C(6)	1.392(3)		
C(6)-H(6)	0.9500	C(13)-N(1)-C(17)	119.08(16)
C(7)-C(8)	1.385(3)	C(13)-N(1)-B(1)	120.56(14)
C(7)-C(12)	1.388(3)	C(17)-N(1)-B(1)	120.33(15)
C(8)-H(8)	0.9500	C(2)-C(1)-B(1)	121.72(16)
C(8)-C(9)	1.391(3)	C(6)-C(1)-C(2)	116.21(16)
C(9)-H(9)	0.9500	C(6)-C(1)-B(1)	122.07(16)
C(9)-C(10)	1.369(3)	C(1)-C(2)-H(2)	119.0
C(10)-H(10)	0.9500	C(3)-C(2)-C(1)	121.99(17)
C(10)-C(11)	1.367(3)	C(3)-C(2)-H(2)	119.0
C(11)-H(11)	0.9500	C(2)-C(3)-H(3)	119.4
C(11)-C(12)	1.392(3)	C(2)-C(3)-C(4)	121.26(17)
C(12)-H(12)	0.9500	C(4)-C(3)-H(3)	119.4
C(13)-H(13)	0.9500	C(3)-C(4)-C(5)	117.34(17)
C(13)-C(14)	1.375(3)	C(3)-C(4)-C(7)	121.82(17)
C(14)-H(14)	0.9500	C(5)-C(4)-C(7)	120.84(17)
C(14)-C(15)	1.384(3)	C(4)-C(5)-H(5)	119.5
C(15)-H(15)	0.9500	C(6)-C(5)-C(4)	120.96(18)
C(15)-C(16)	1.382(3)	C(6)-C(5)-H(5)	119.5

C(1)-C(6)-C(5)	122.25(18)
C(1)-C(6)-H(6)	118.9
C(5)-C(6)-H(6)	118.9
C(8)-C(7)-C(4)	121.43(18)
C(8)-C(7)-C(12)	117.16(18)
C(12)-C(7)-C(4)	121.41(18)
C(7)-C(8)-H(8)	119.3
C(7)-C(8)-C(9)	121.4(2)
C(9)-C(8)-H(8)	119.3
C(8)-C(9)-H(9)	119.9
C(10)-C(9)-C(8)	120.2(2)
C(10)-C(9)-H(9)	119.9
C(9)-C(10)-H(10)	120.2
C(11)-C(10)-C(9)	119.6(2)
C(11)-C(10)-H(10)	120.2
C(10)-C(11)-H(11)	119.9
C(10)-C(11)-C(12)	120.3(2)
C(12)-C(11)-H(11)	119.9
C(7)-C(12)-C(11)	121.3(2)
C(7)-C(12)-H(12)	119.3
C(11)-C(12)-H(12)	119.3
N(1)-C(13)-H(13)	119.1
N(1)-C(13)-C(14)	121.88(17)
C(14)-C(13)-H(13)	119.1
C(13)-C(14)-H(14)	120.4
C(13)-C(14)-C(15)	119.23(18)
C(15)-C(14)-H(14)	120.4
C(14)-C(15)-H(15)	120.6
C(16)-C(15)-C(14)	118.79(18)
C(16)-C(15)-H(15)	120.6
C(15)-C(16)-H(16)	120.4
C(17)-C(16)-C(15)	119.29(17)
C(17)-C(16)-H(16)	120.4
N(1)-C(17)-C(16)	121.73(17)
N(1)-C(17)-H(17)	119.1
C(16)-C(17)-H(17)	119.1
F(1)-B(1)-F(2)	110.28(14)
F(1)-B(1)-N(1)	104.72(14)
F(1)-B(1)-C(1)	113.37(15)
F(2)-B(1)-N(1)	104.64(14)
F(2)-B(1)-C(1)	113.53(15)
C(1)-B(1)-N(1)	109.56(13)
Cl(1S)-C(1S)-H(1SA)	109.6
Cl(1S)-C(1S)-H(1SB)	109.6
Cl(2S)-C(1S)-Cl(1S)	110.3(6)
Cl(2S)-C(1S)-H(1SA)	109.6
Cl(2S)-C(1S)-H(1SB)	109.6
H(1SA)-C(1S)-H(1SB)	108.1
Cl(1B)-C(1SB)-H(1SE)	109.8
Cl(1B)-C(1SB)-H(1SF)	109.8
Cl(2B)-C(1SB)-Cl(1B)	109.2(11)
Cl(2B)-C(1SB)-H(1SE)	109.8
Cl(2B)-C(1SB)-H(1SF)	109.8
H(1SE)-C(1SB)-H(1SF)	108.3
Cl(1C)-C(1SC)-H(1SC)	109.9
Cl(1C)-C(1SC)-H(1SD)	109.9
Cl(2C)-C(1SC)-Cl(1C)	108.7(13)
Cl(2C)-C(1SC)-H(1SC)	109.9
Cl(2C)-C(1SC)-H(1SD)	109.9
H(1SC)-C(1SC)-H(1SD)	108.3

Table S8. Anisotropic displacement parameters (Å^{2}x 10^3) for **3b**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
F(1)	29(1)	33(1)	40(1)	-9(1)	12(1)	2(1)
F(2)	32(1)	51(1)	27(1)	0(1)	12(1)	-1(1)
N(1)	24(1)	29(1)	27(1)	0(1)	11(1)	1(1)
C(1)	28(1)	34(1)	26(1)	-7(1)	9(1)	0(1)
C(2)	31(1)	31(1)	37(1)	-1(1)	11(1)	1(1)
C(3)	33(1)	31(1)	44(1)	-3(1)	17(1)	-3(1)
C(4)	29(1)	35(1)	32(1)	-9(1)	10(1)	-3(1)
C(5)	29(1)	54(1)	30(1)	3(1)	7(1)	6(1)
C(6)	32(1)	52(1)	30(1)	5(1)	12(1)	3(1)
C(7)	32(1)	33(1)	44(1)	-11(1)	13(1)	-3(1)
C(8)	37(1)	40(1)	65(1)	7(1)	25(1)	4(1)
C(9)	45(1)	40(1)	83(2)	6(1)	36(1)	2(1)
C(10)	32(1)	44(1)	76(2)	-18(1)	23(1)	-3(1)
C(11)	32(1)	75(2)	51(1)	-24(1)	6(1)	3(1)
C(12)	33(1)	70(2)	41(1)	-14(1)	11(1)	2(1)
C(13)	41(1)	30(1)	29(1)	2(1)	12(1)	-2(1)
C(14)	48(1)	41(1)	28(1)	1(1)	7(1)	-3(1)
C(15)	39(1)	42(1)	35(1)	-10(1)	14(1)	-9(1)
C(16)	44(1)	27(1)	49(1)	-4(1)	18(1)	-4(1)
C(17)	38(1)	29(1)	36(1)	5(1)	13(1)	5(1)
B(1)	30(1)	33(1)	26(1)	-4(1)	10(1)	1(1)
Cl(1S)	73(2)	60(2)	43(1)	6(1)	21(1)	-13(2)
Cl(2S)	73(2)	60(2)	43(1)	6(1)	21(1)	-13(2)
C(1S)	56(4)	58(3)	33(3)	10(3)	10(4)	-7(3)
Cl(1B)	73(2)	60(2)	43(1)	6(1)	21(1)	-13(2)
Cl(2B)	73(2)	60(2)	43(1)	6(1)	21(1)	-13(2)
C(1SB)	56(4)	58(3)	33(3)	10(3)	10(4)	-7(3)
Cl(1C)	73(2)	60(2)	43(1)	6(1)	21(1)	-13(2)
Cl(2C)	73(2)	60(2)	43(1)	6(1)	21(1)	-13(2)
C(1SC)	56(4)	58(3)	33(3)	10(3)	10(4)	-7(3)

Table S9. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **3b**.

	x	y	z	U(eq)
H(2)	1340	7649	6908	39
H(3)	3187	7646	6941	42
H(5)	3850	5625	10142	46
H(6)	2004	5645	10124	45
H(8)	4675	7138	6404	55
H(9)	6530	7095	6424	63
H(10)	7904	6563	8594	59
H(11)	7422	6053	10734	65
H(12)	5569	6092	10728	57
H(13)	-1116	7067	5588	40
H(14)	-2151	6209	3487	48
H(15)	-2173	4604	3687	45
H(16)	-1135	3910	6018	46
H(17)	-164	4819	8086	41
H(1SA)	3985	5738	15184	60
H(1SB)	3692	4881	14020	60
H(1SE)	4127	5529	15469	60
H(1SF)	3992	4718	14225	60
H(1SC)	4321	4997	16305	60
H(1SD)	4103	4259	14927	60

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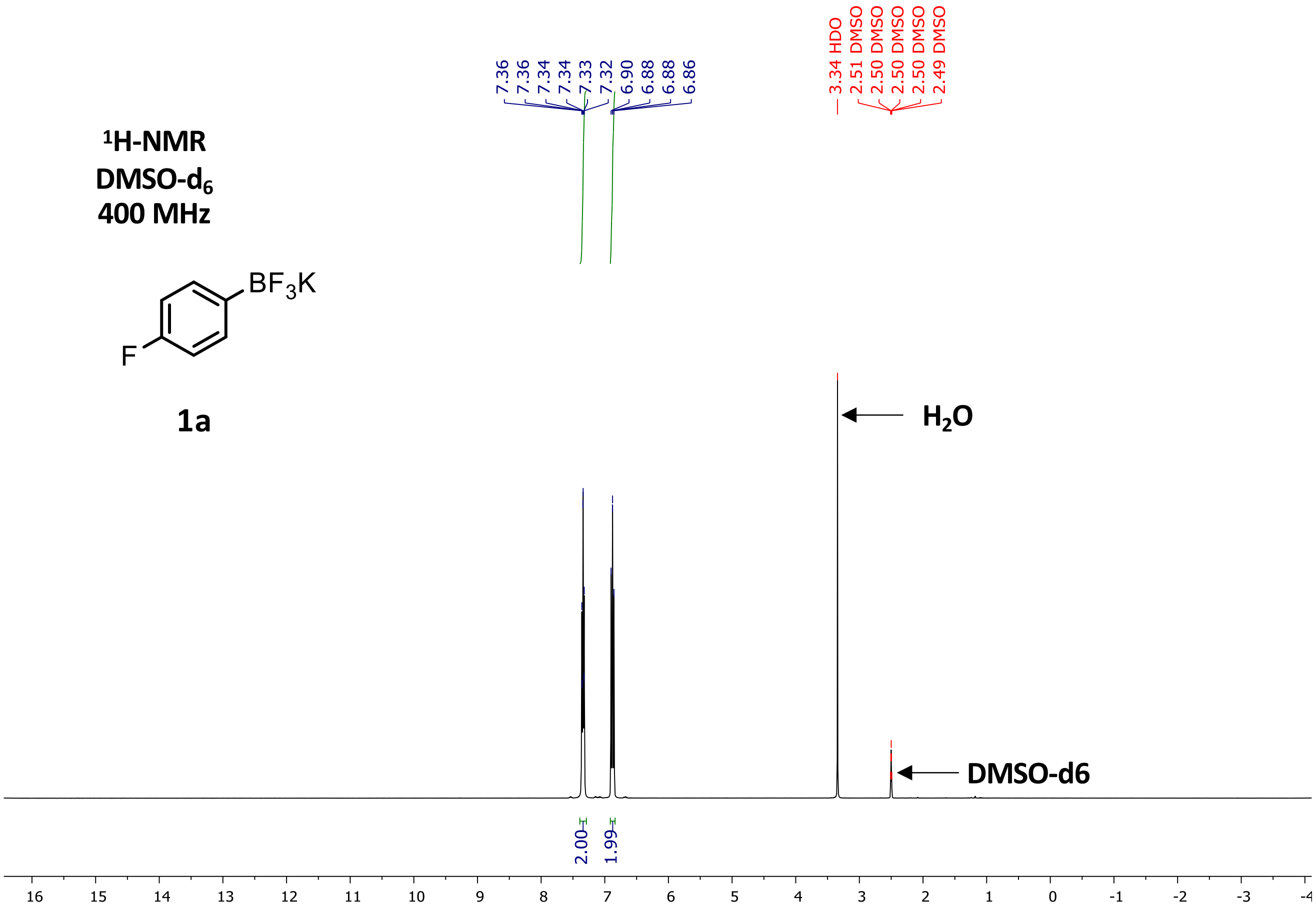
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6.88
6.88
6.86
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2.50 DMSO
2.50 DMSO
2.50 DMSO
2.49 DMSO
^{1}H-NMR
DMSO-d$_6$
400 MHz
BF$_3$K
F
H$_2$O
DMSO-d6
2.00
1.99
16
15
14
13
12
11
10
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8
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6
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2
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-2
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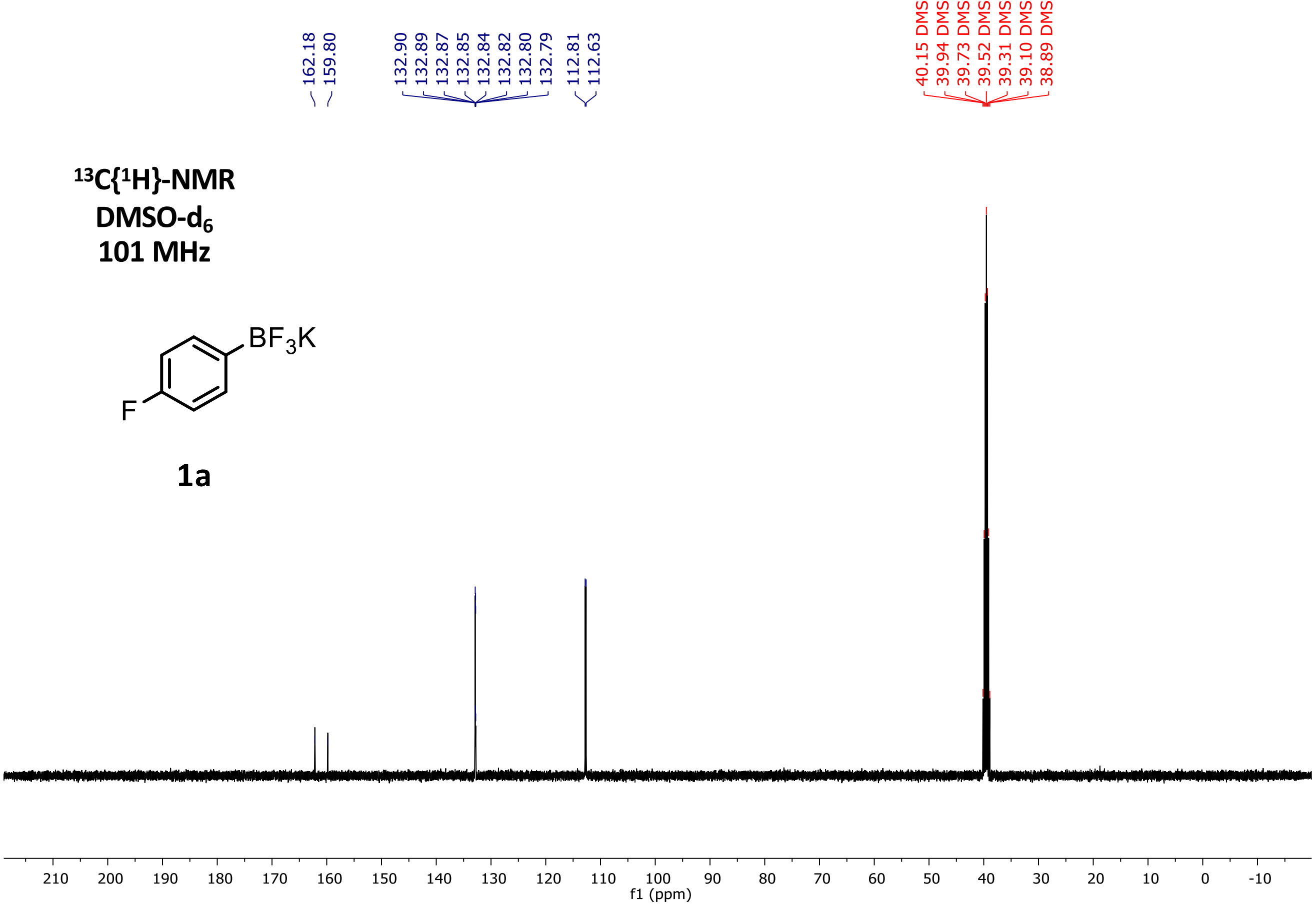
f1 (ppm)

1a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40.15 DMSO
39.94 DMSO
39.73 DMSO
39.52 DMSO
39.31 DMSO
39.10 DMSO
38.89 DMSO
^{13}C{^{1}H}-NMR
DMSO-d$_6$
101 MHz
BF$_3$K
F
210
200
190
180
170
160
150
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60
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20
10
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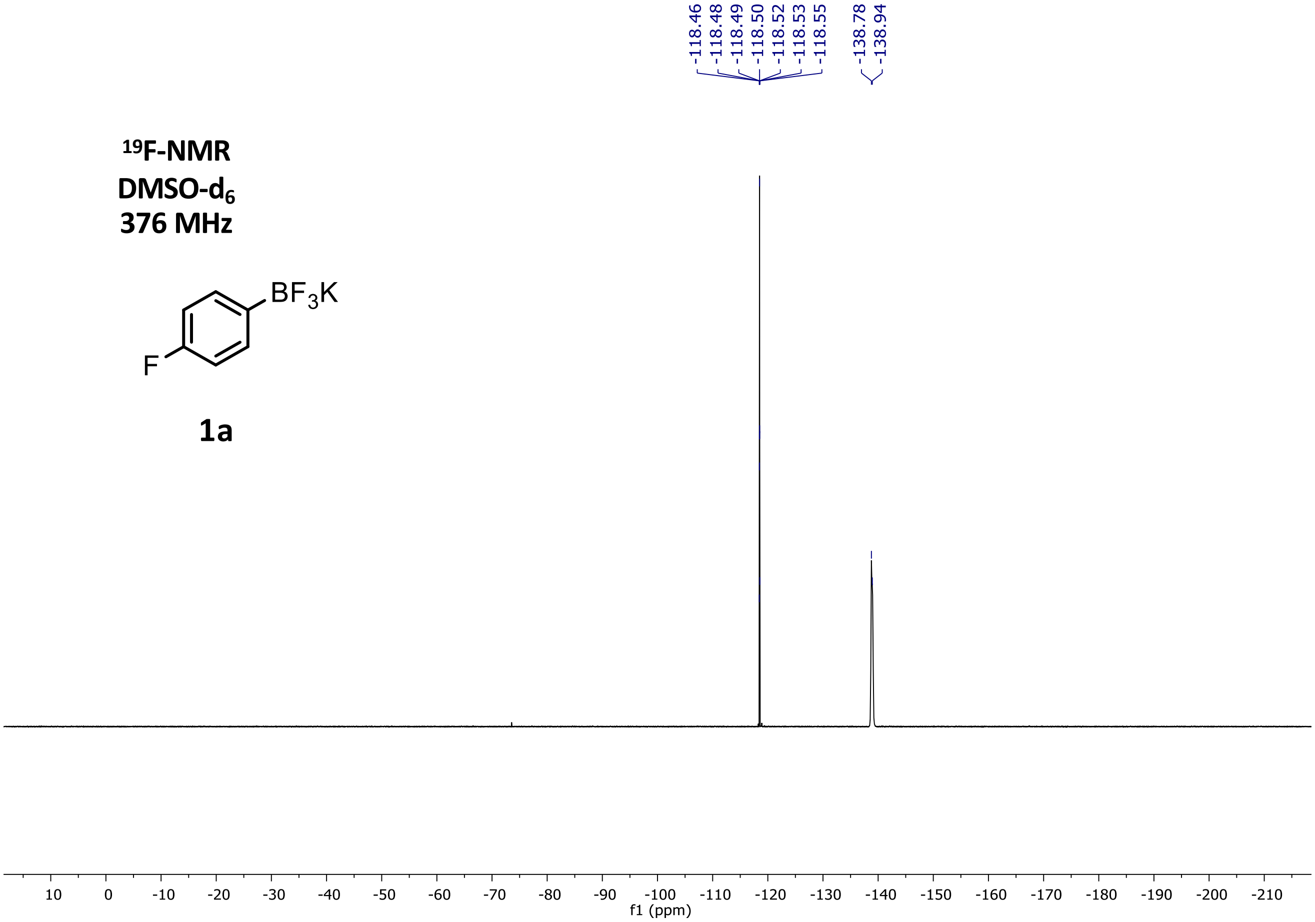
-10
f1 (ppm)

1a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DMSO-d_6
376 MHz
BF_3K
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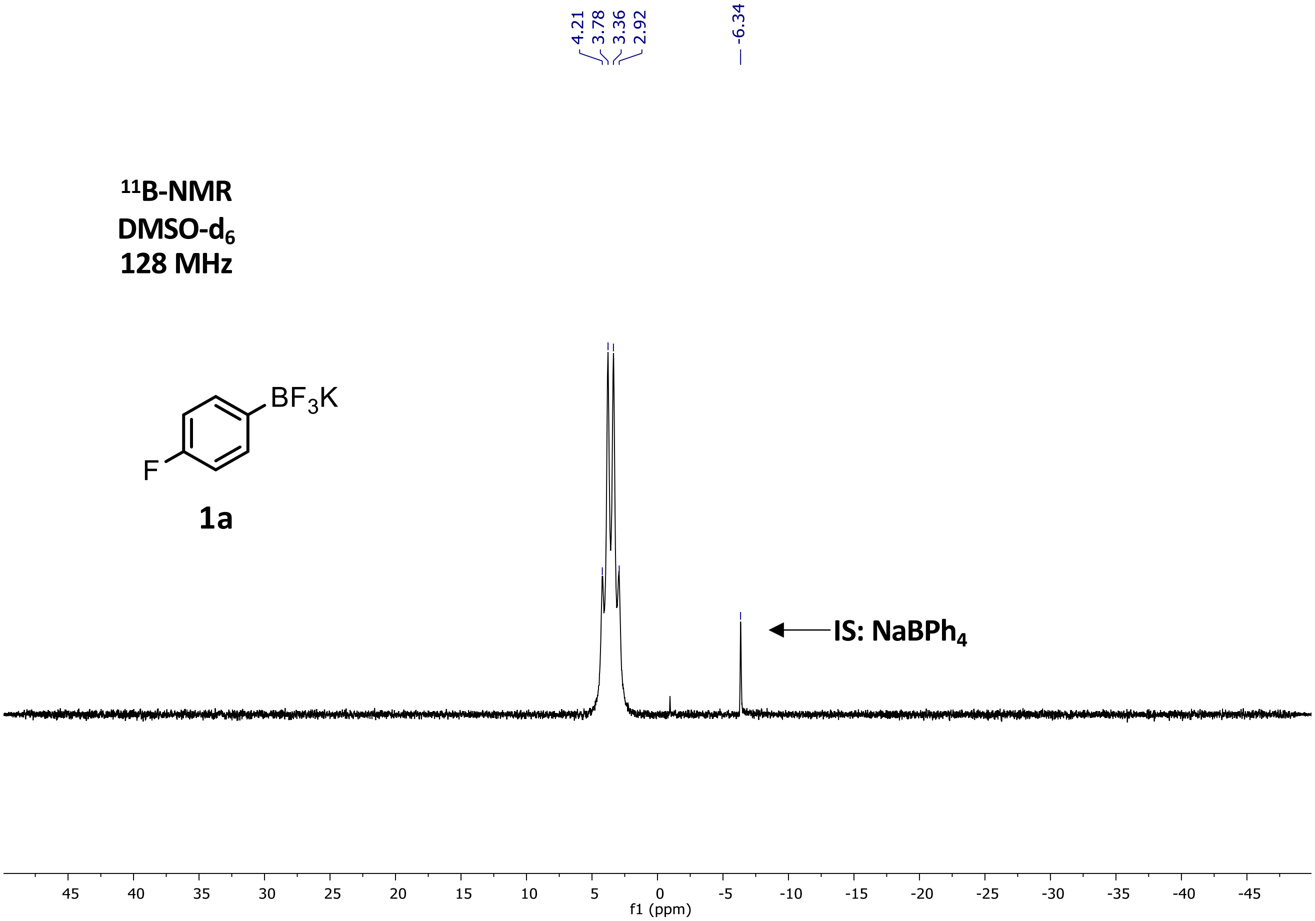
f1 (ppm)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128 MHz
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F
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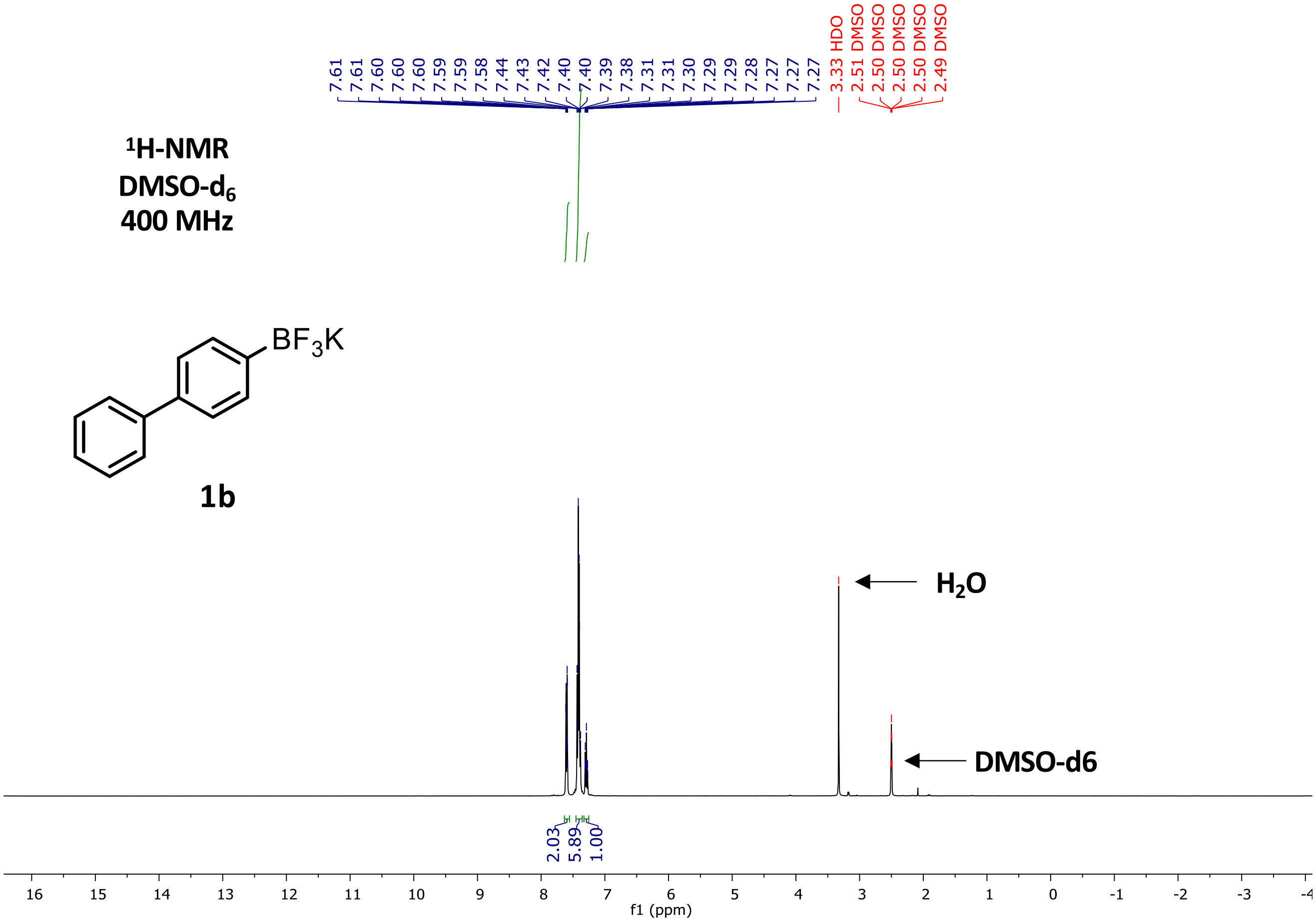
f1 (ppm)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7.29
7.28
7.27
7.27
7.27
3.33 HDO
2.51 DMSO
2.50 DMSO
2.50 DMSO
2.50 DMSO
2.49 DMSO
^{1}H-NMR
DMSO-d_6
400 MHz
BF_3K
1b
H_2O
DMSO-d6
2.03
5.89
1.00
16
15
14
13
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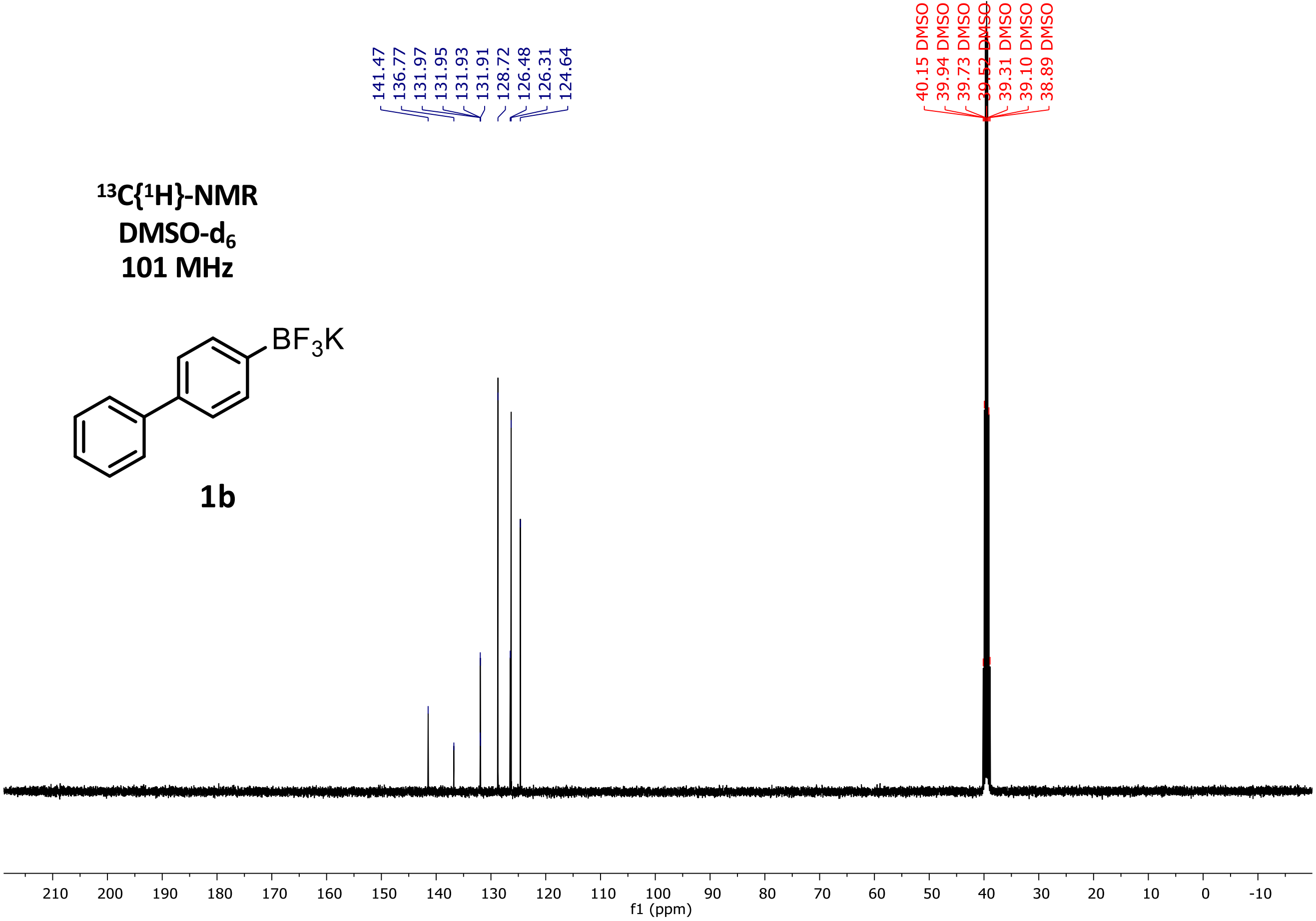
-3
-4
f1 (ppm)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39.73 DMSO
39.52 DMSO
39.31 DMSO
39.10 DMSO
38.89 DMSO
^{13}C{^{1}H}-NMR
DMSO-d_6
101 MHz
BF_3K
1b
210
200
190
180
170
160
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140
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120
110
100
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30
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0
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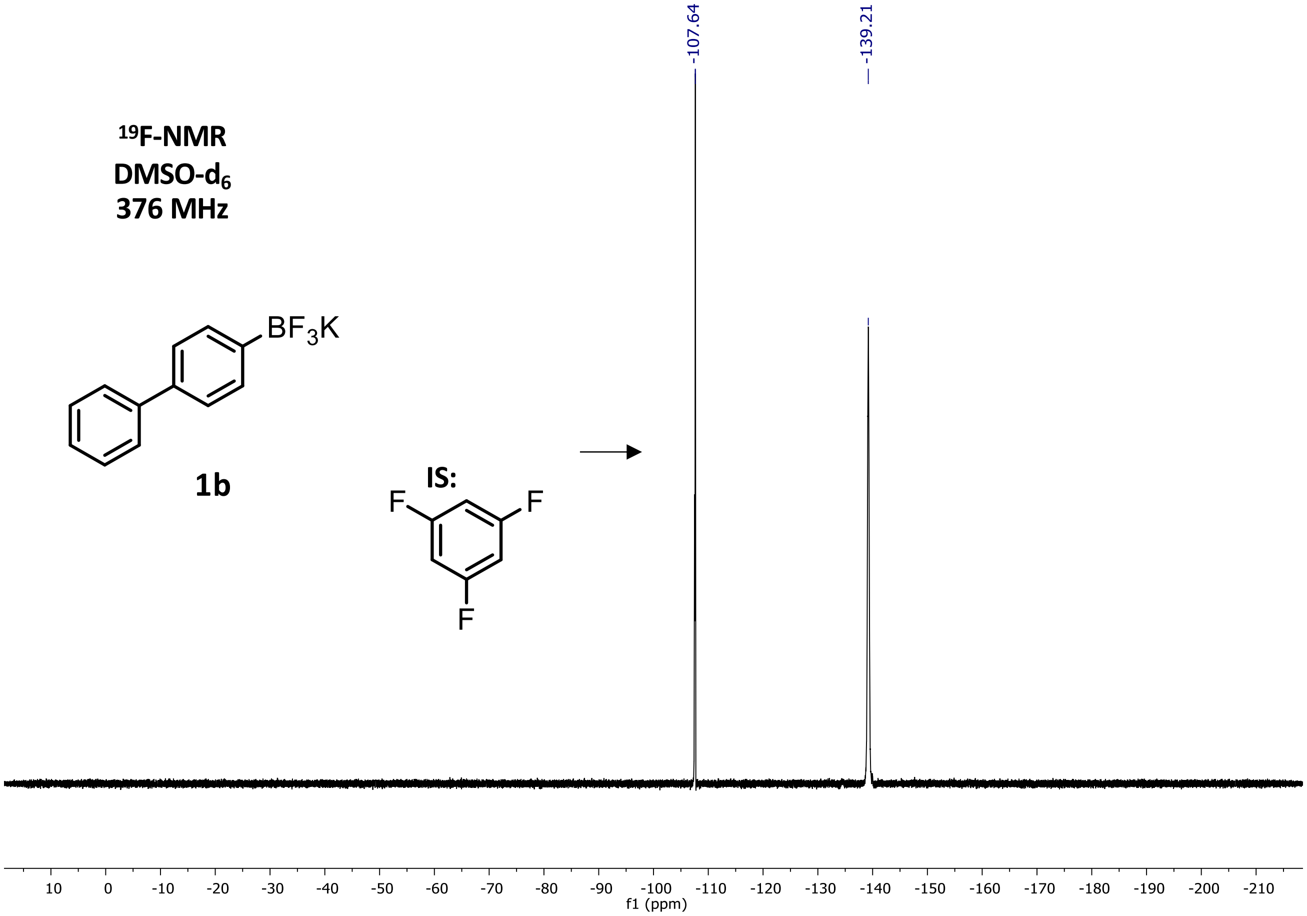
f1 (ppm)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F
F
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-10
-20
-30
-40
-50
-60
-70
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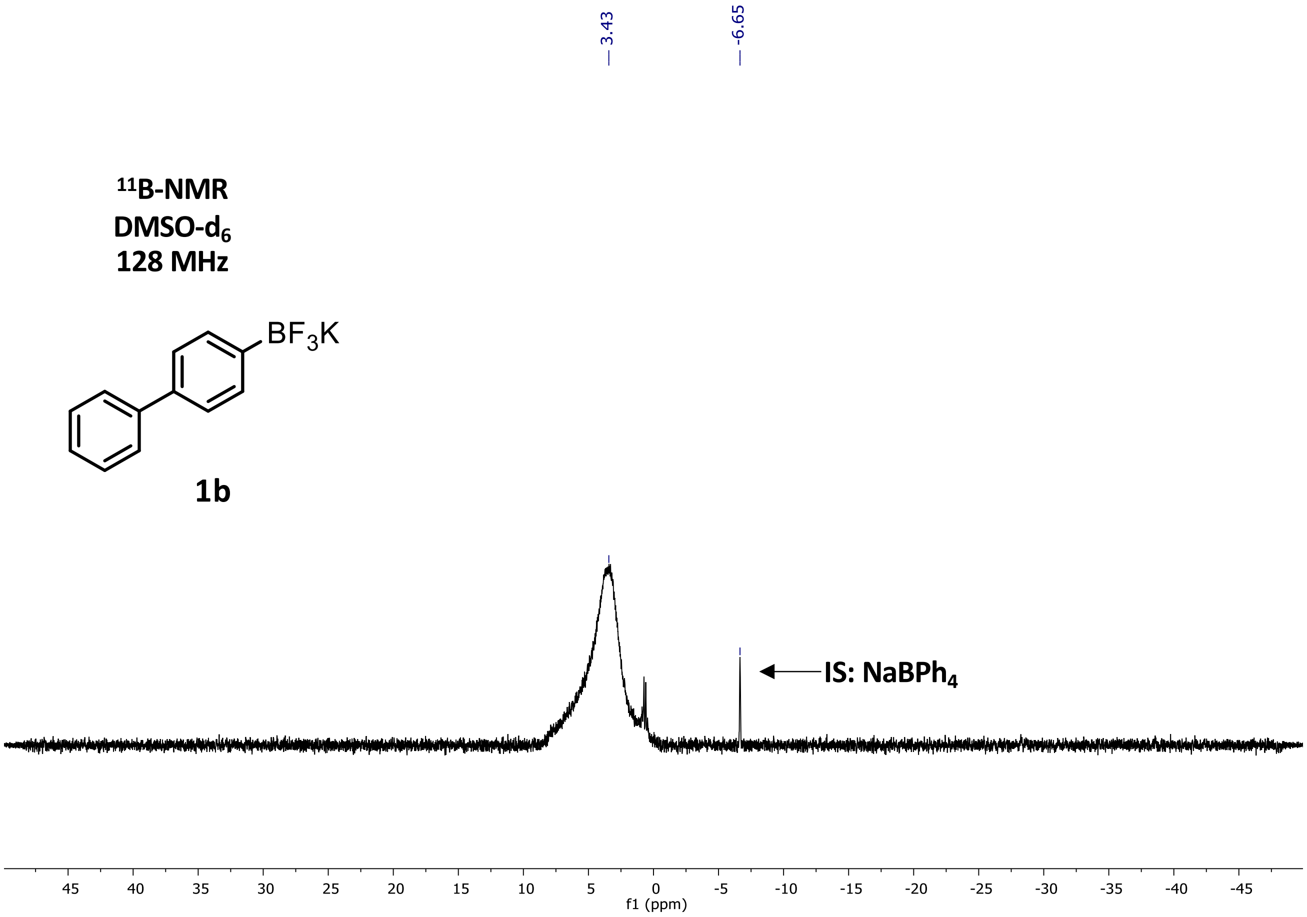
-200
-210
f1 (ppm)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DMSO-d_6
128 MHz
BF_3K
1b
IS: $NaBPh_4$
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35
30
25
20
15
10
5
0
-5
-10
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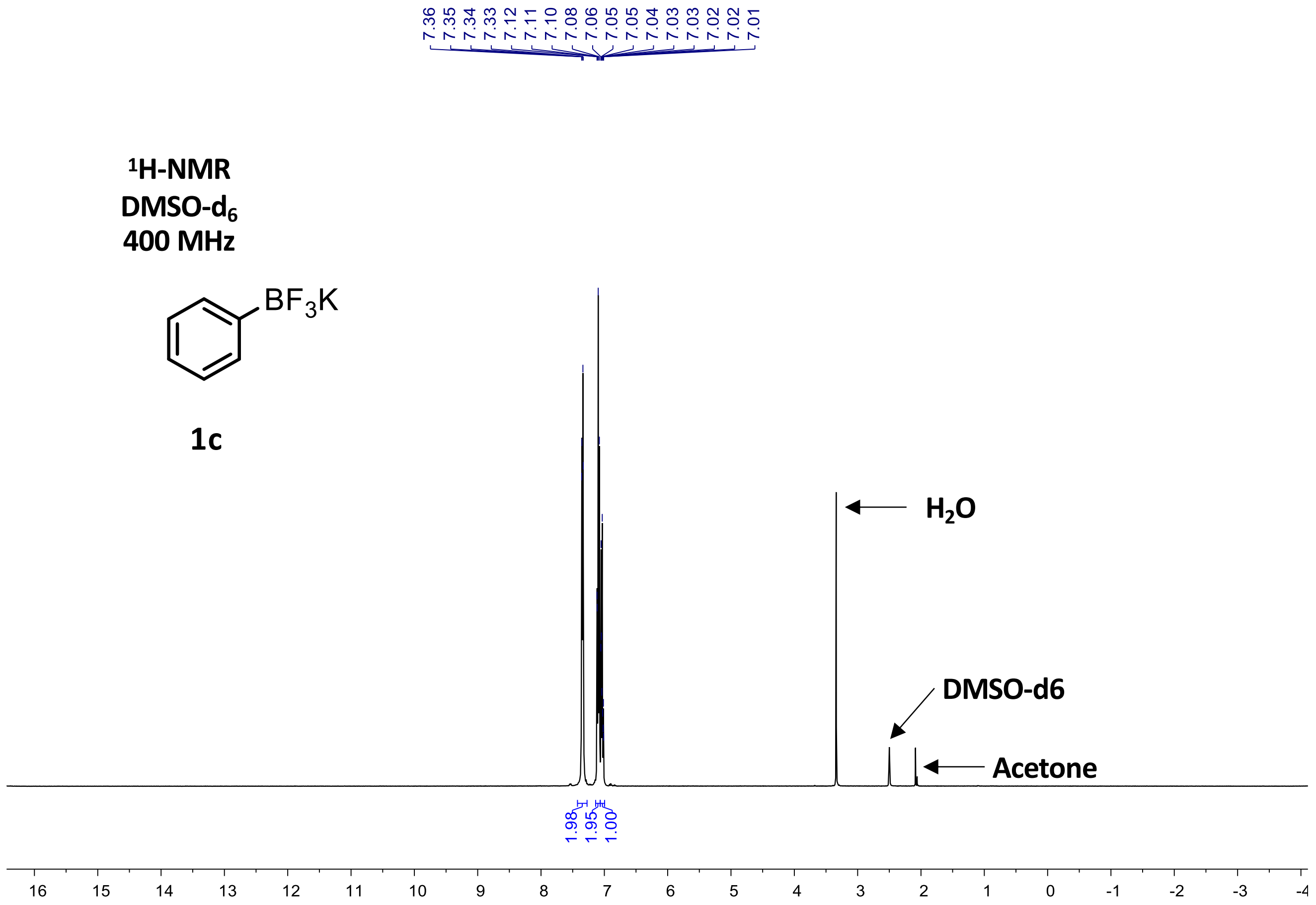
-20
-25
-30
-35
-40
-45
f1 (ppm)

^{1}H-NMR
DMSO-d_6
400 MHz



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DMSO-d_6
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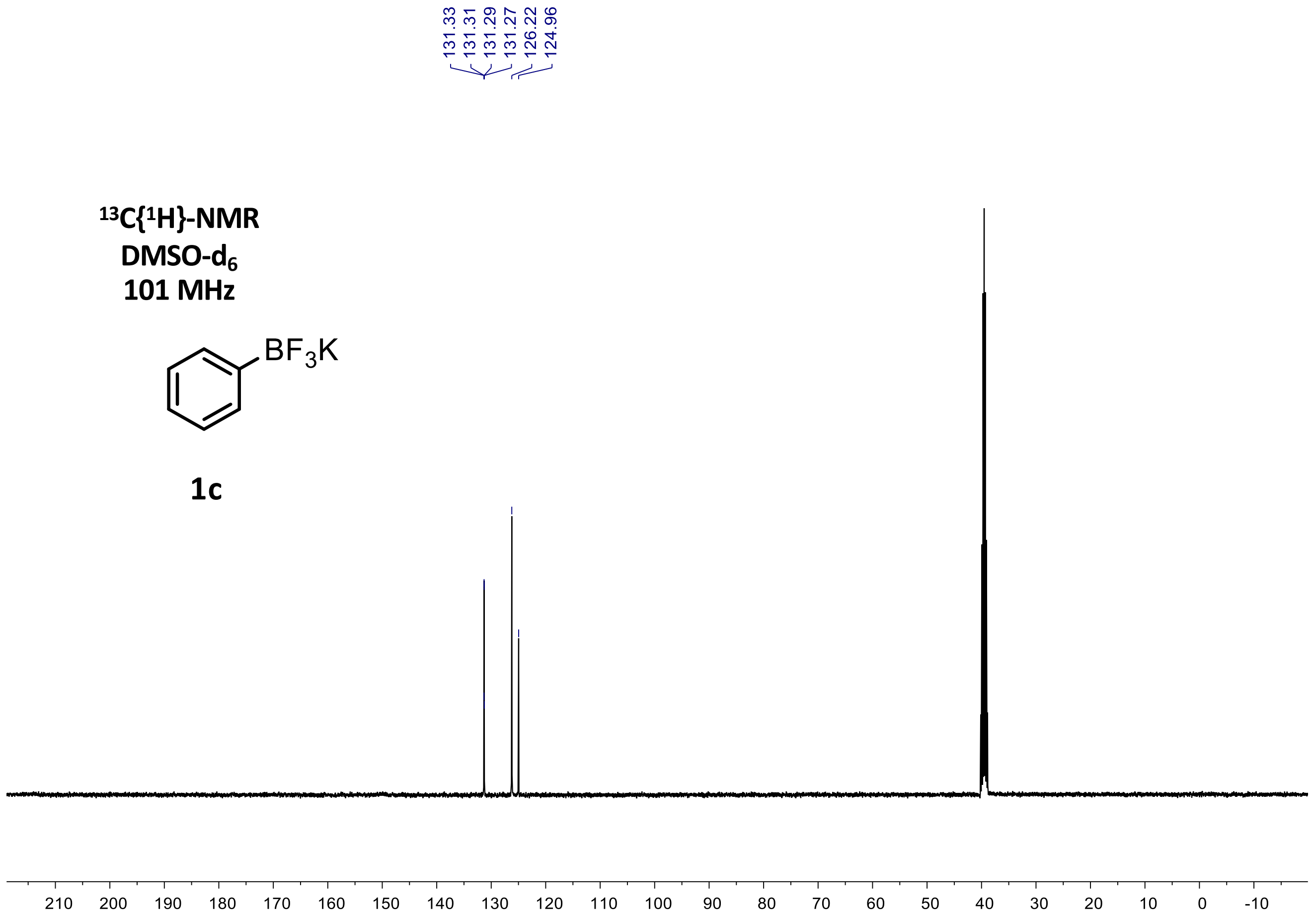

1c


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DMSO-d_6
376 MHz
BF_3K
1c
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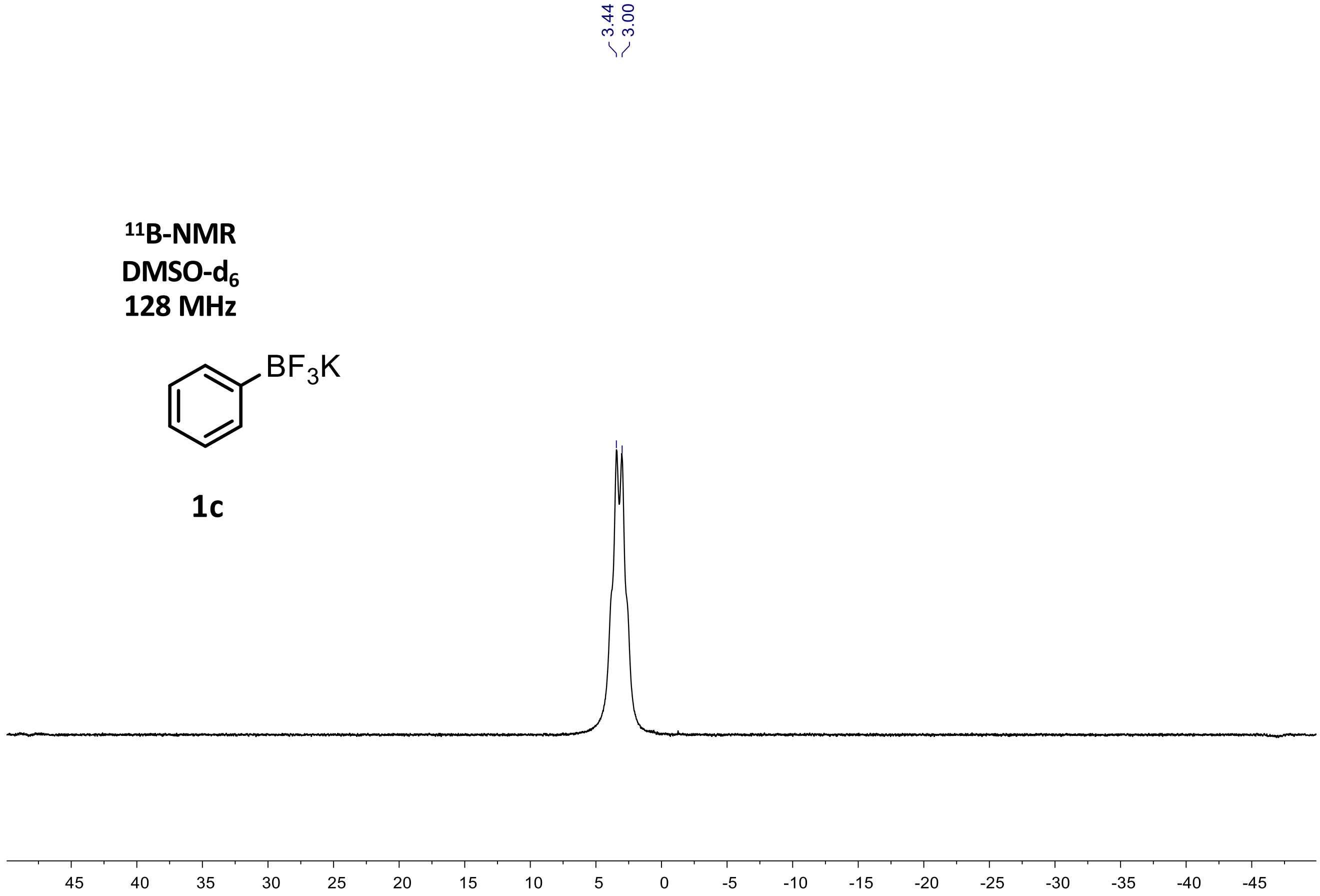
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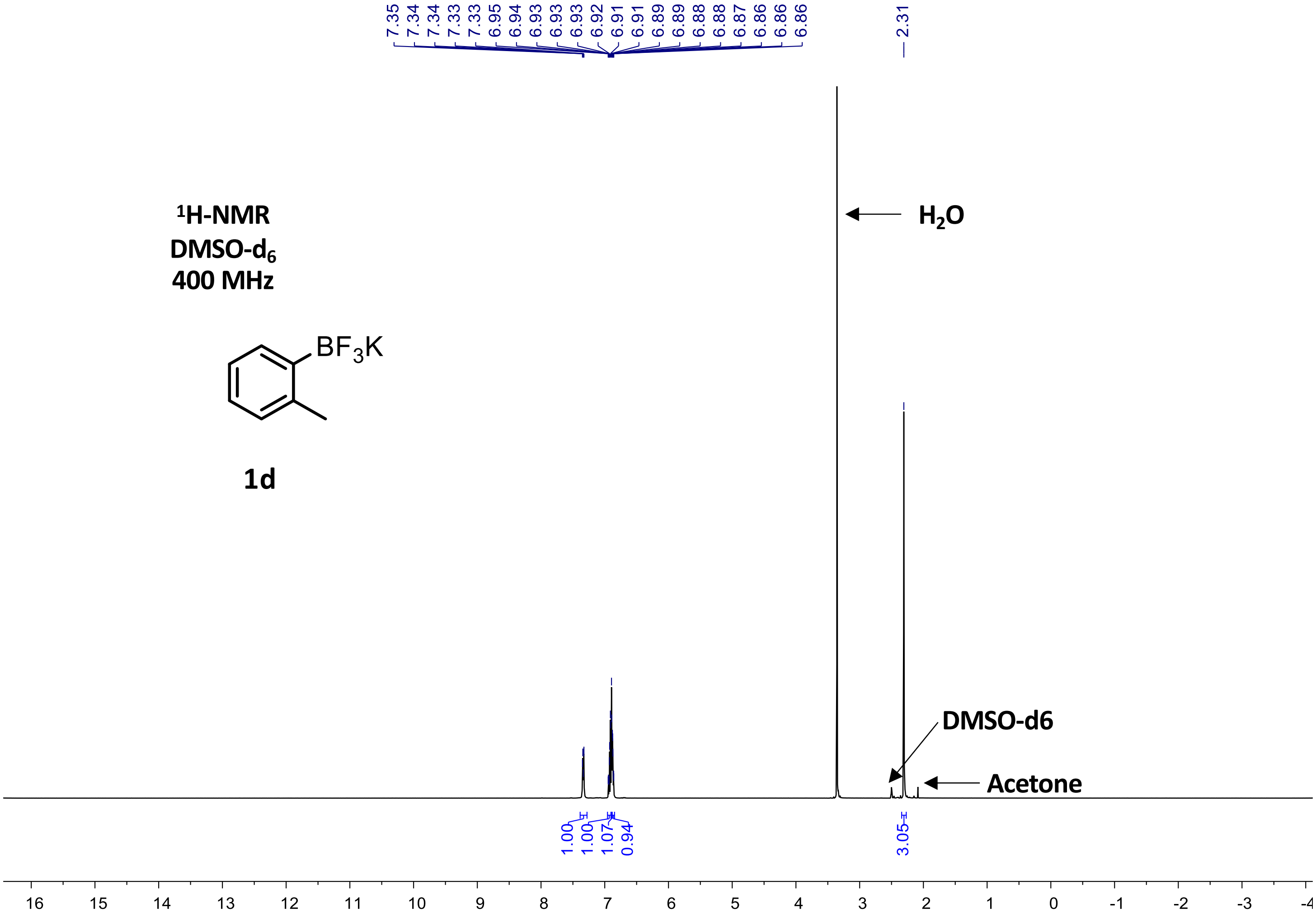

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6.91
6.89
6.89
6.88
6.88
6.87
6.86
6.86
6.86
2.31
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400 MHz
BF$_3$K
1d
H$_2$O
DMSO-d6
Acetone
1.00
1.00
1.07
0.94
3.05
16
15
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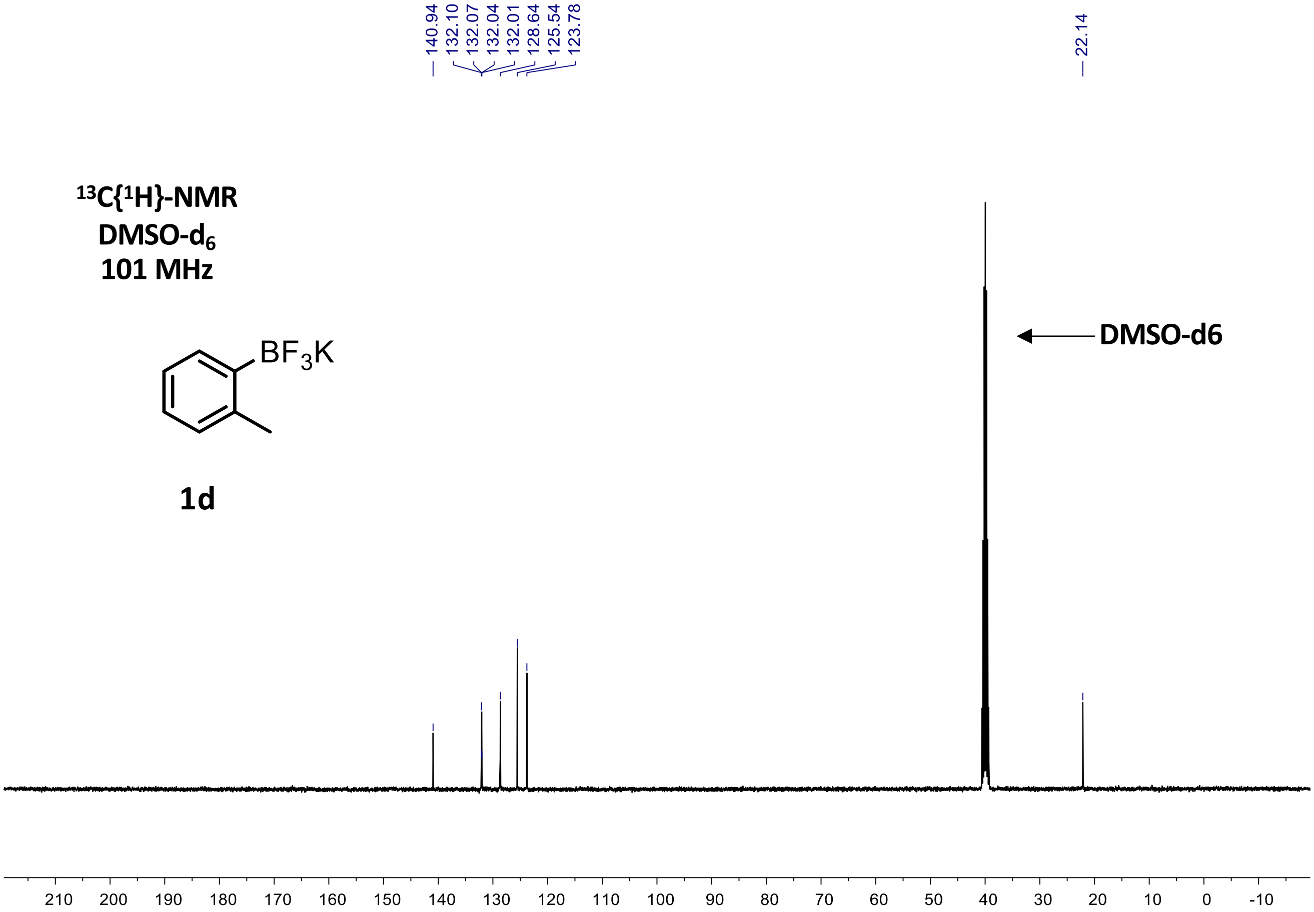
-2
-3
-4



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DMSO-d$_6$
101 MHz
BF$_3$K
1d
DMSO-d6
210
200
190
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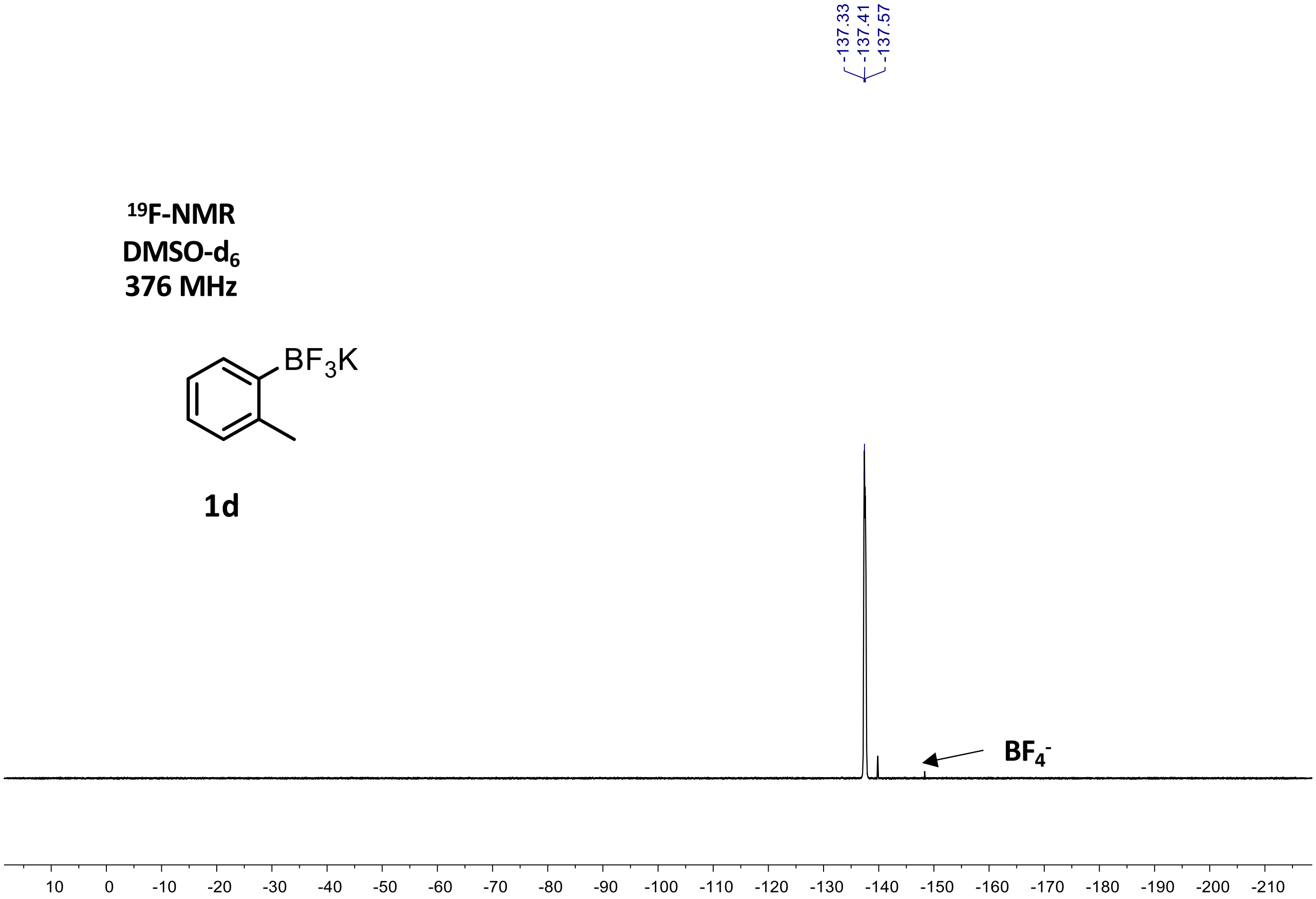

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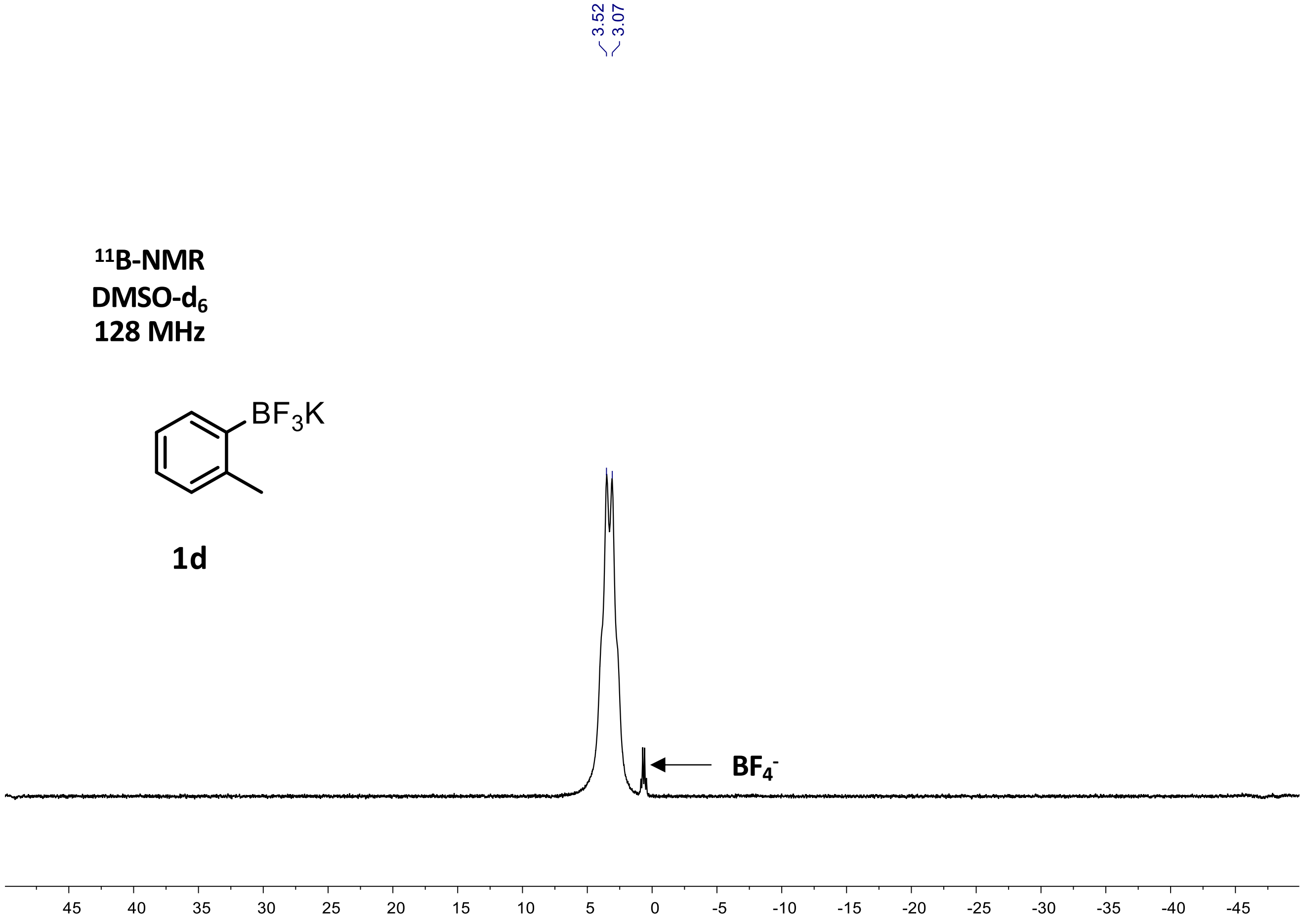
DMSO-d_6
376 MHz

1d



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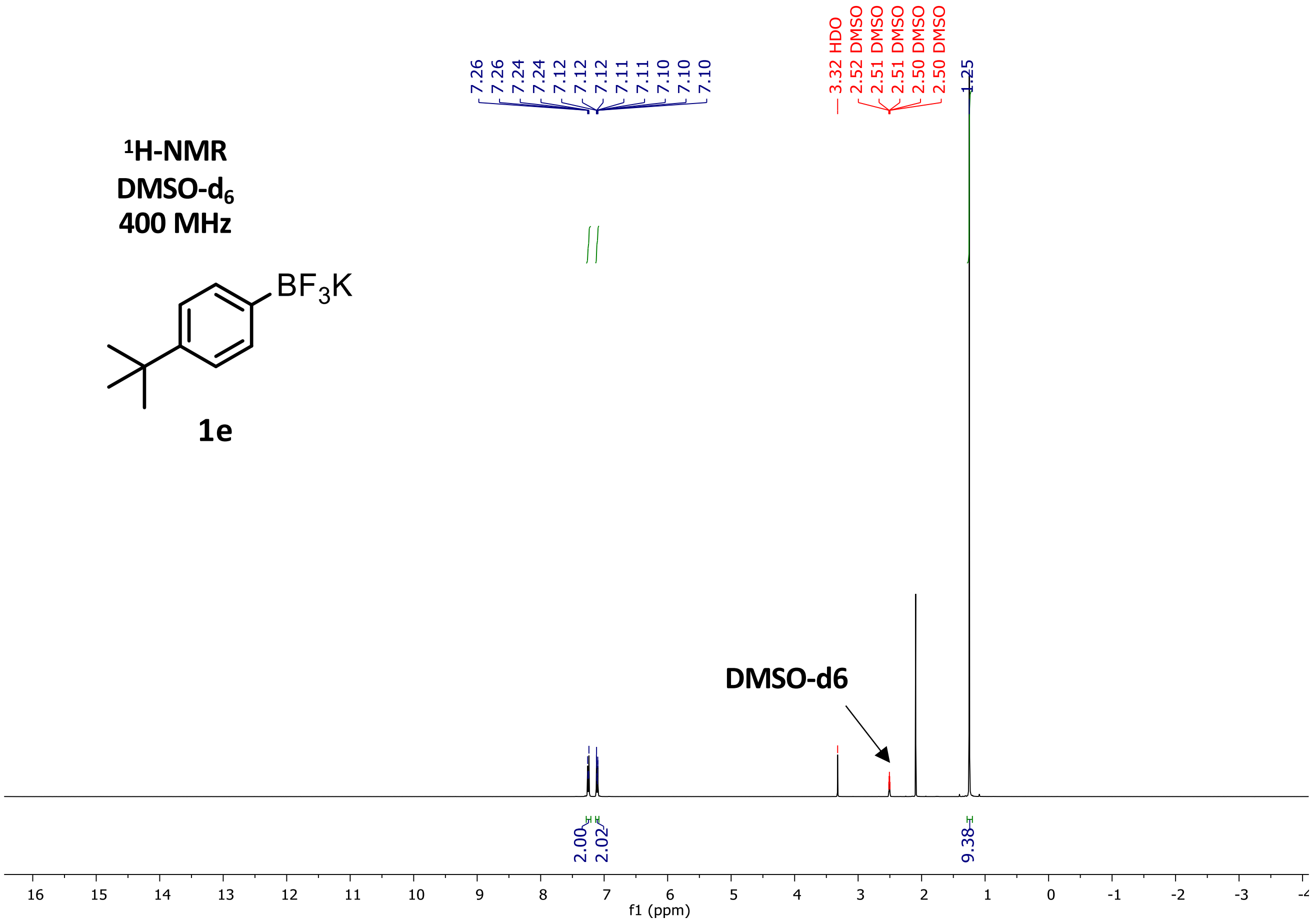
1d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2.52 DMSO
2.51 DMSO
2.51 DMSO
2.50 DMSO
2.50 DMSO
1.25
1H-NMR
DMSO-d6
400 MHz
BF3K
1e
DMSO-d6
2.00
2.02
9.38
16
15
14
13
12
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9
8
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5
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3
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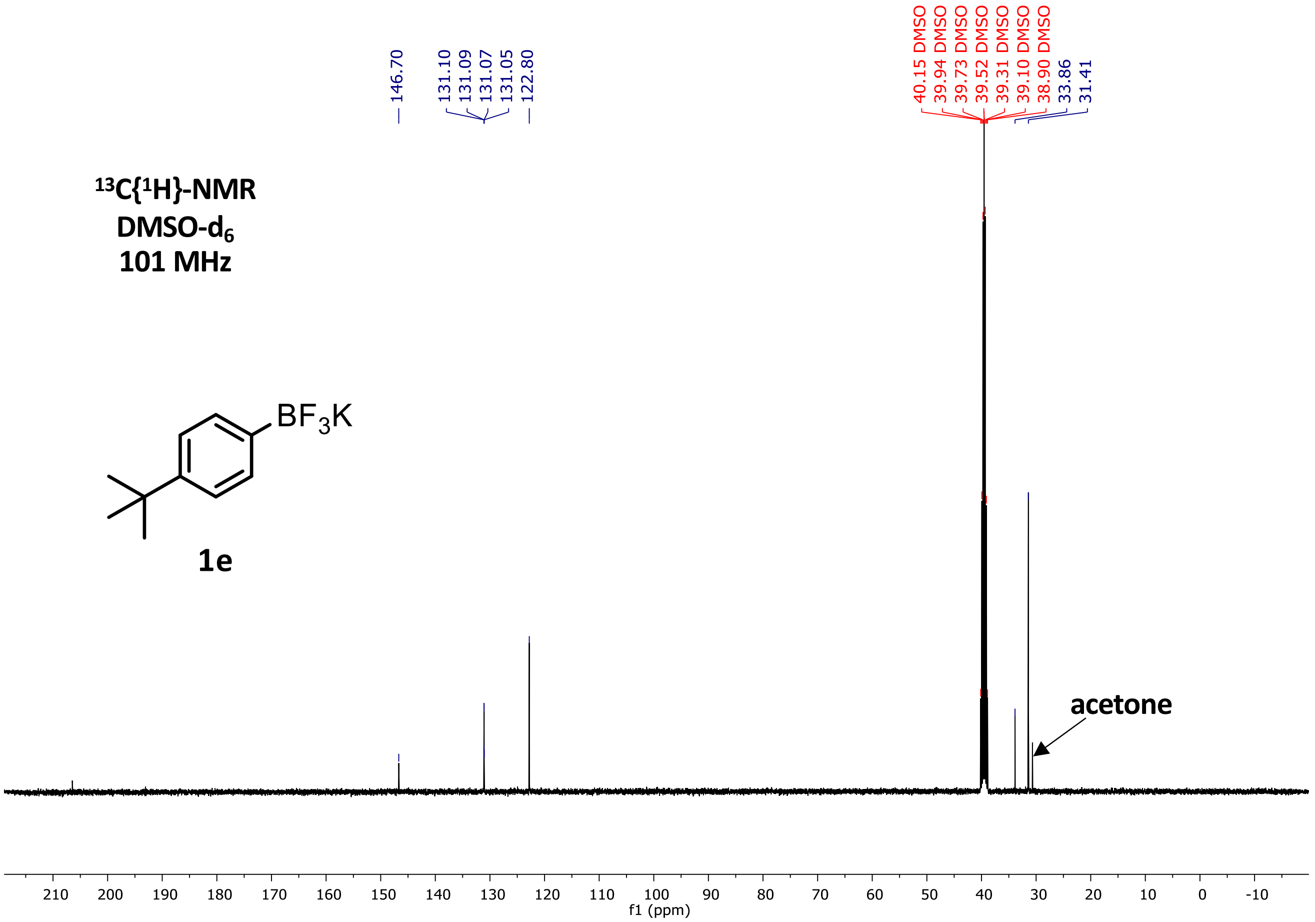
-2
-3
f1 (ppm)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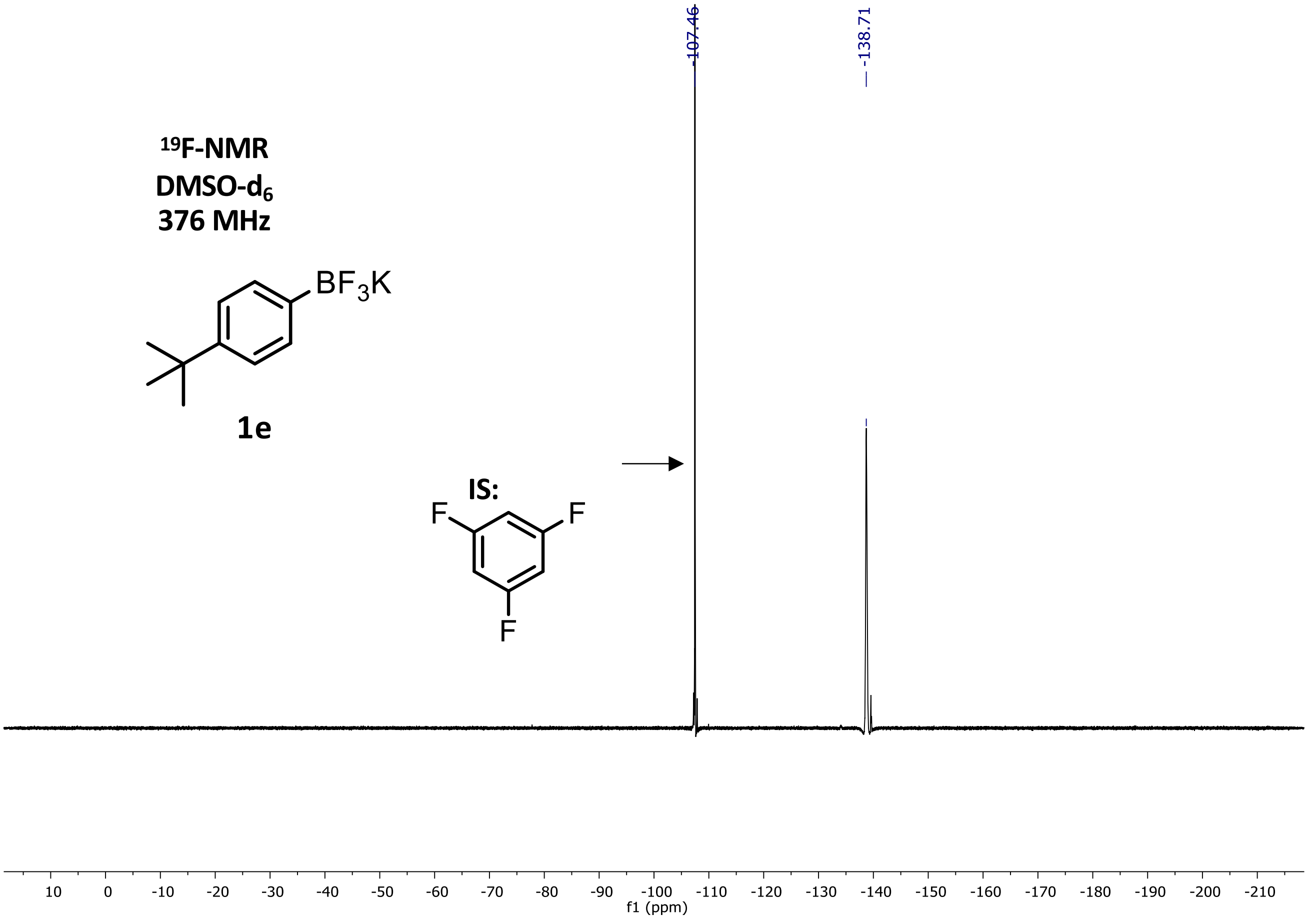
39.10 DMSO
38.90 DMSO
33.86
31.41
13C{1H}-NMR
DMSO-d6
101 MHz
BF3K
1e
acetone
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

^{19}F-NMR
DMSO-d_6
376 MHz



1e

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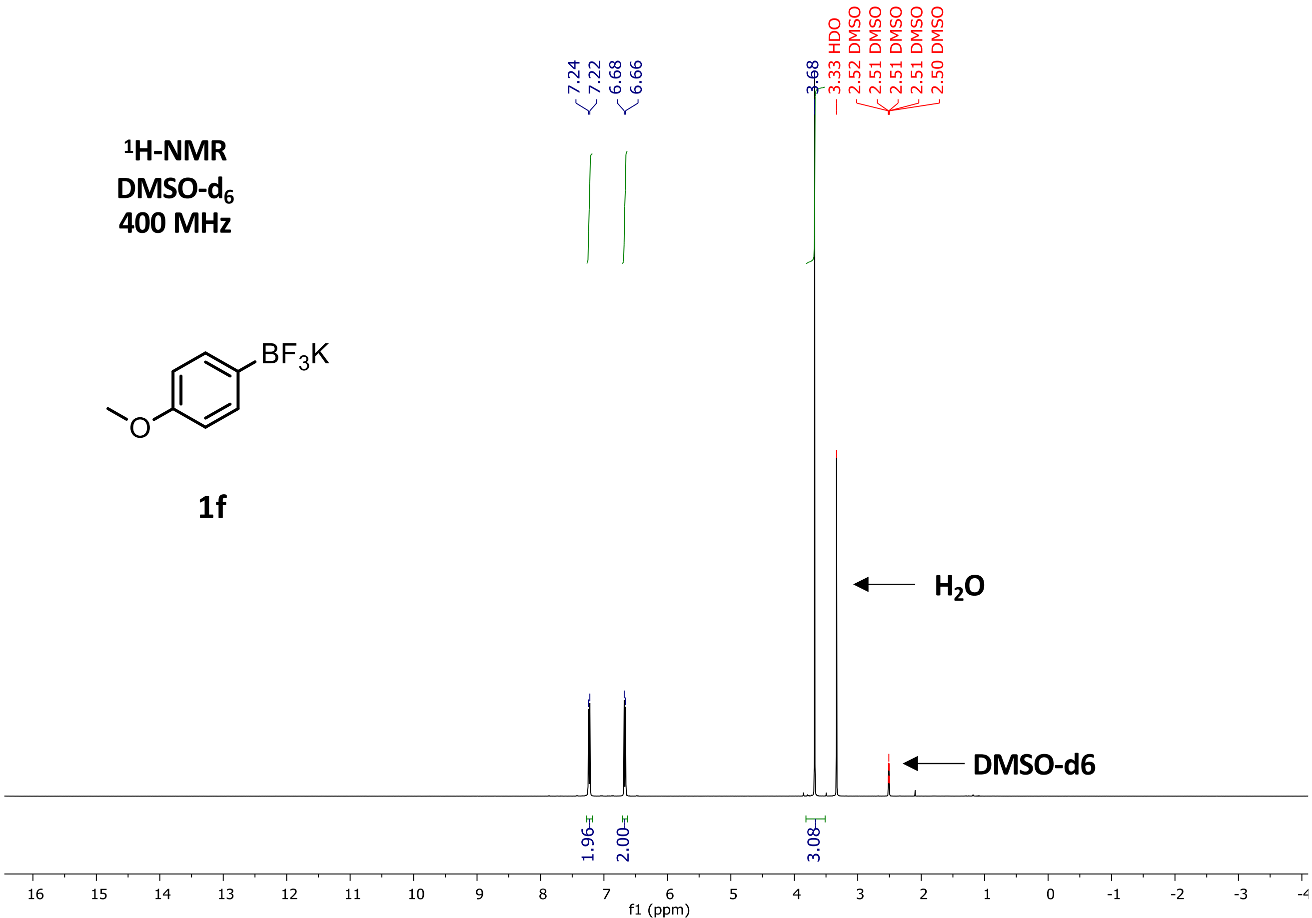
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6.66
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2.51 DMSO
2.51 DMSO
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2.50 DMSO
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1.96
2.00
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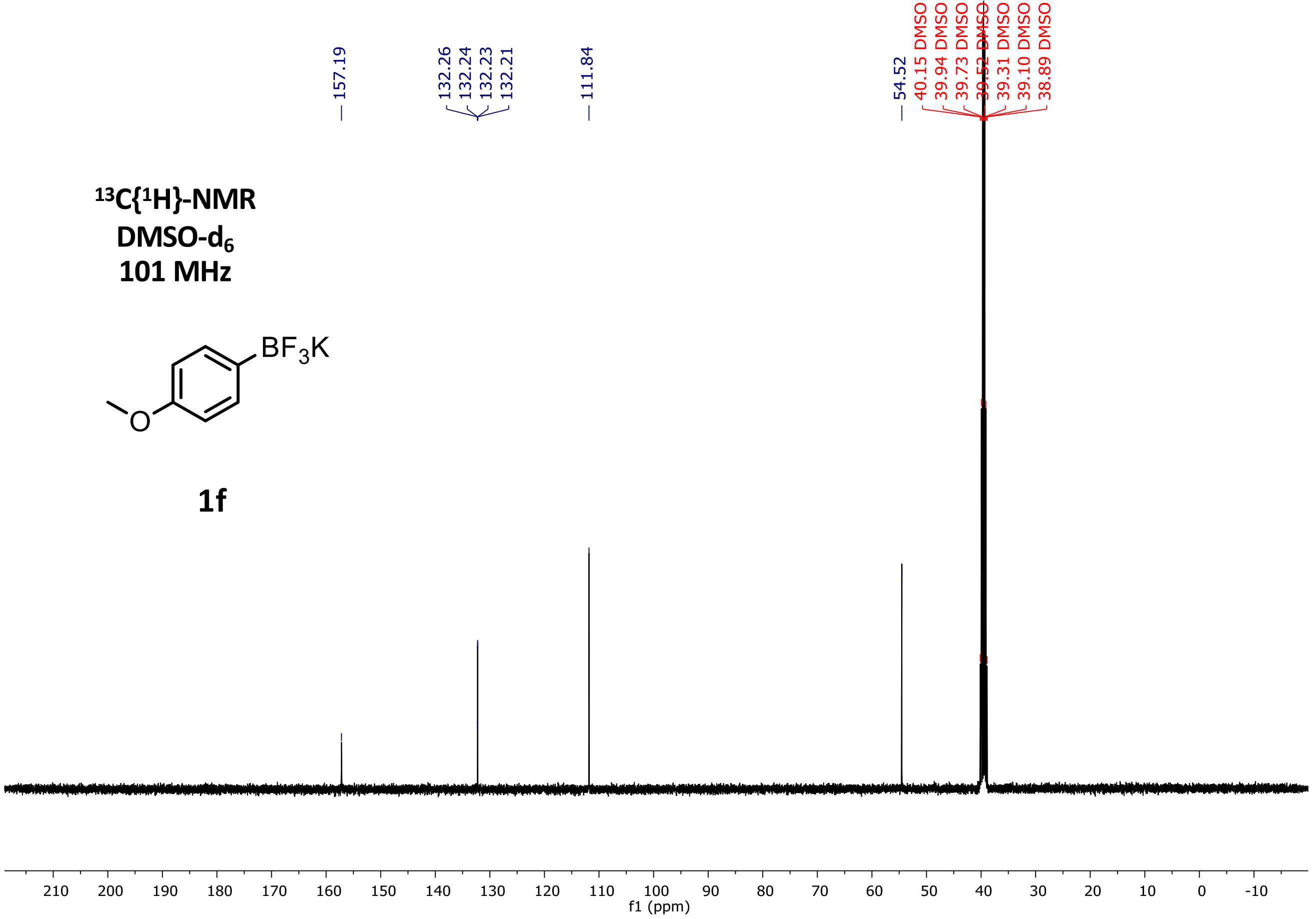
f1 (ppm)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39.94 DMSO
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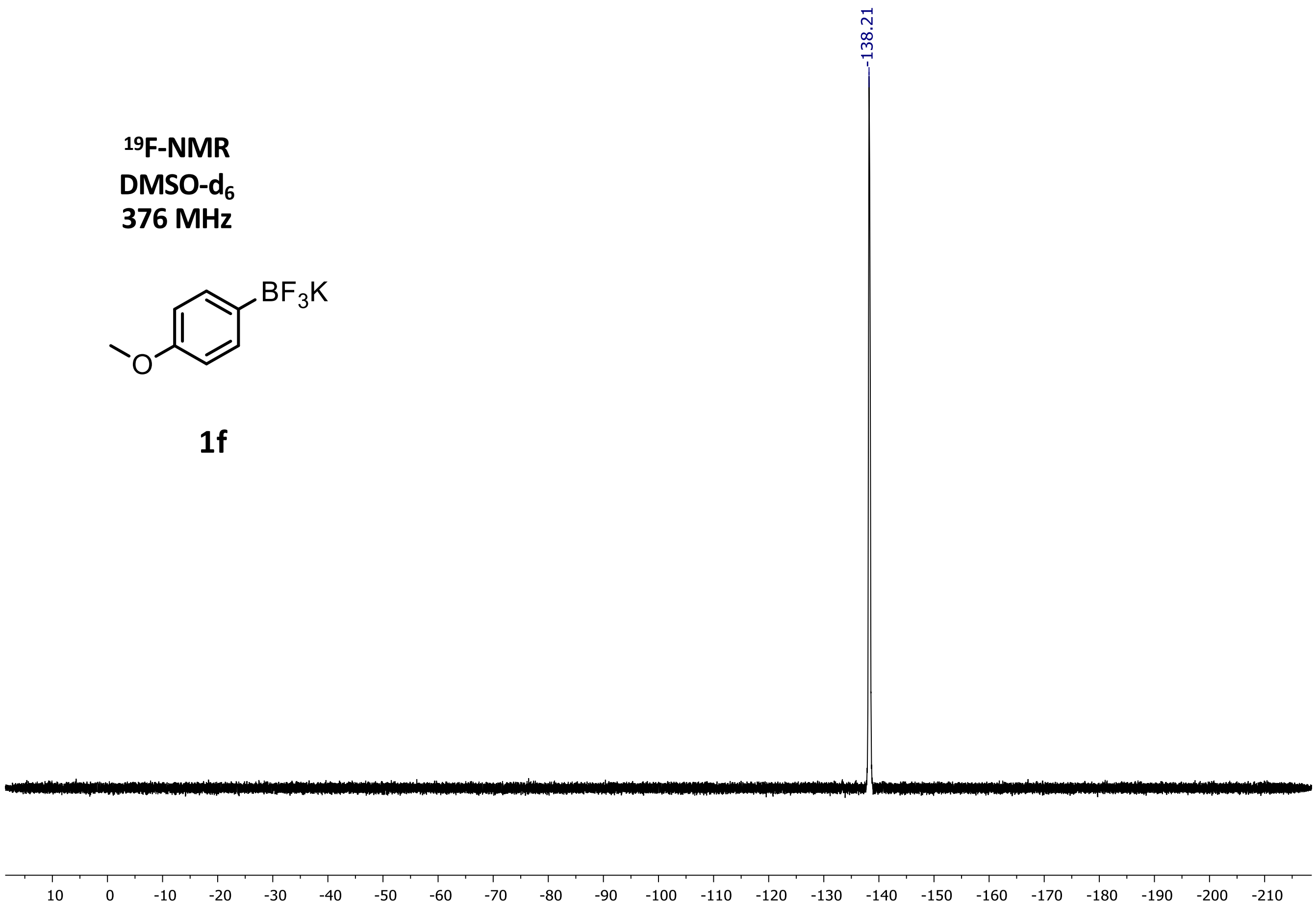
f1 (ppm)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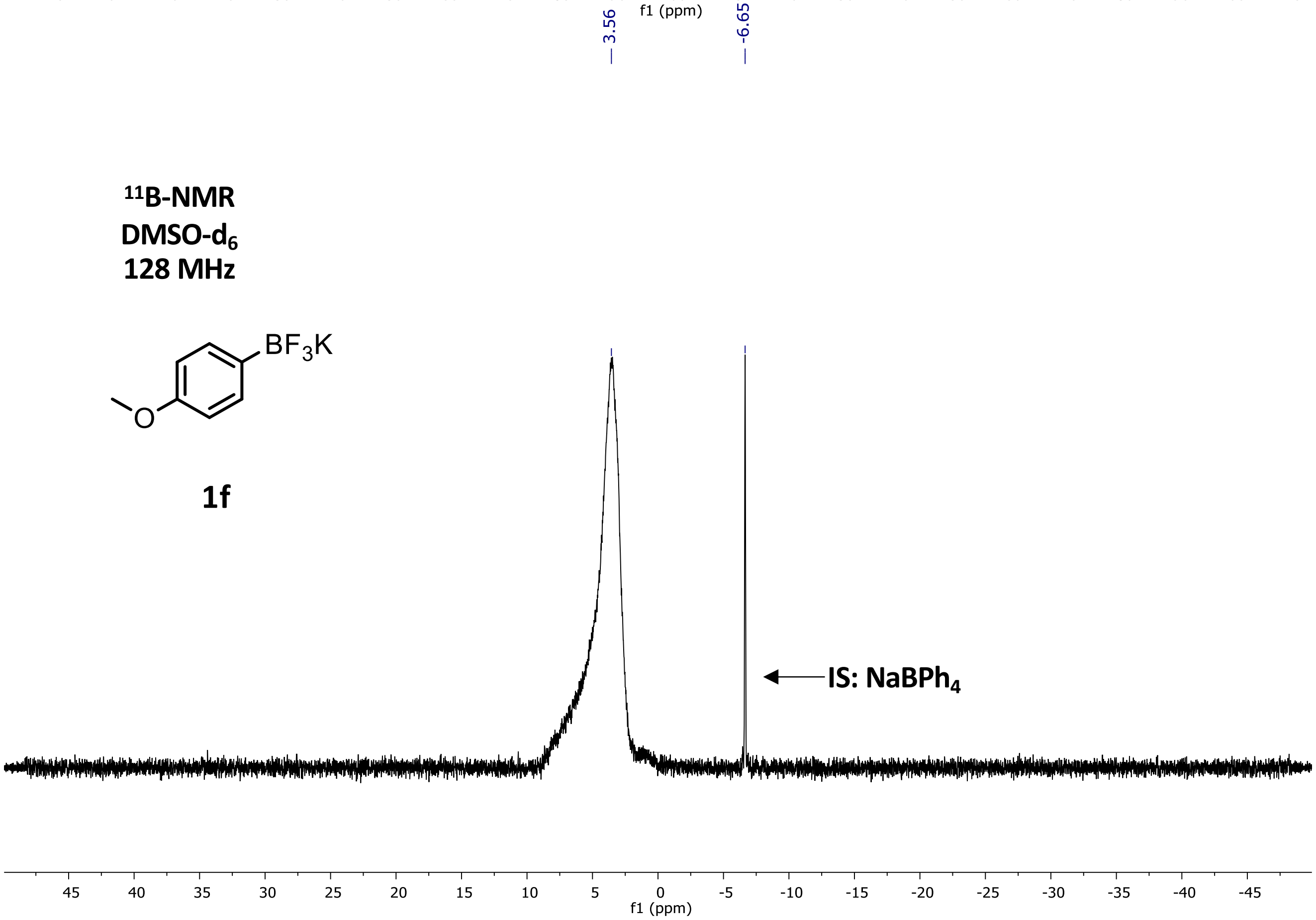
f1 (ppm)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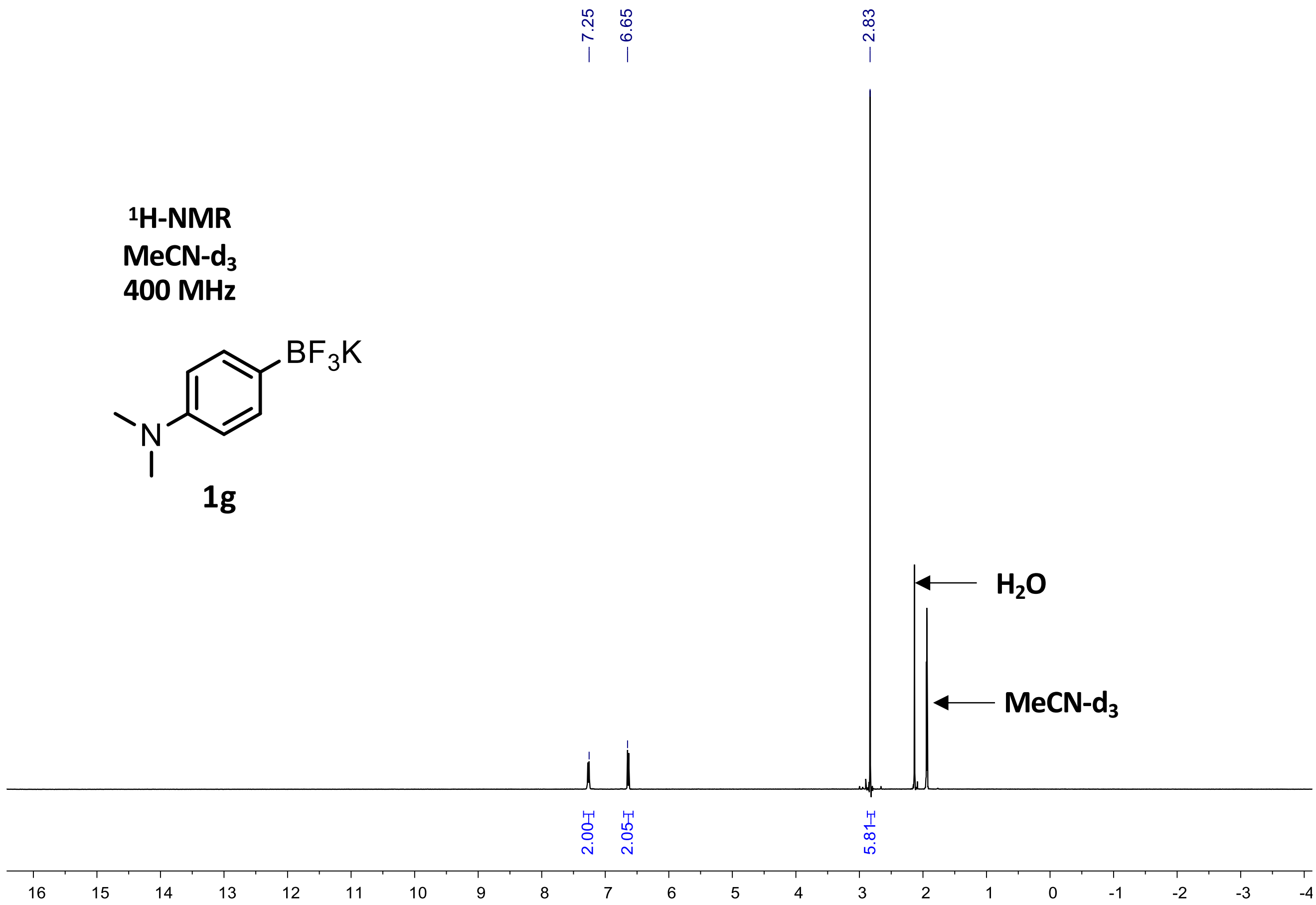
-45
f1 (ppm)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MeCN-d3
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2.05
5.81
16
15
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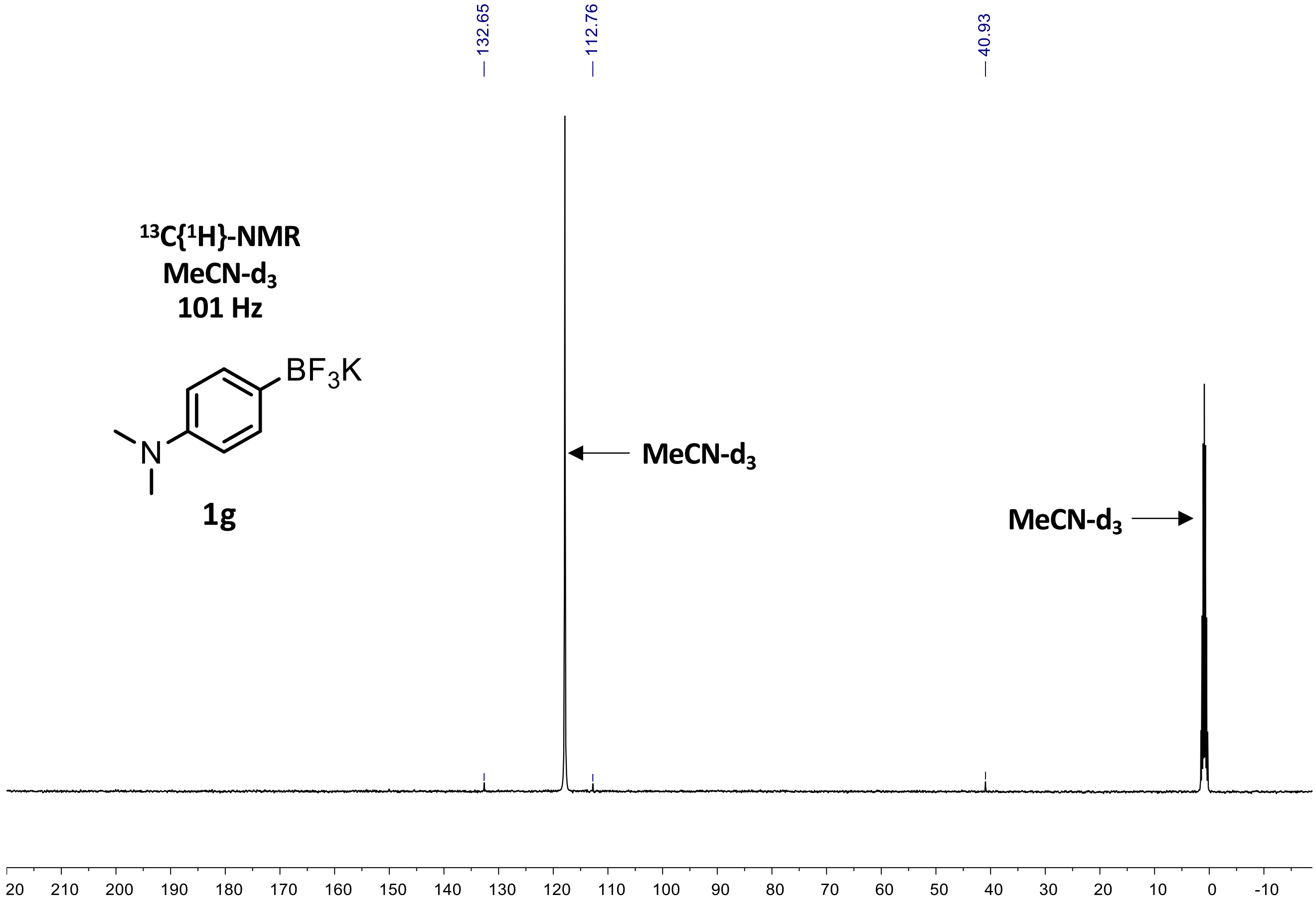
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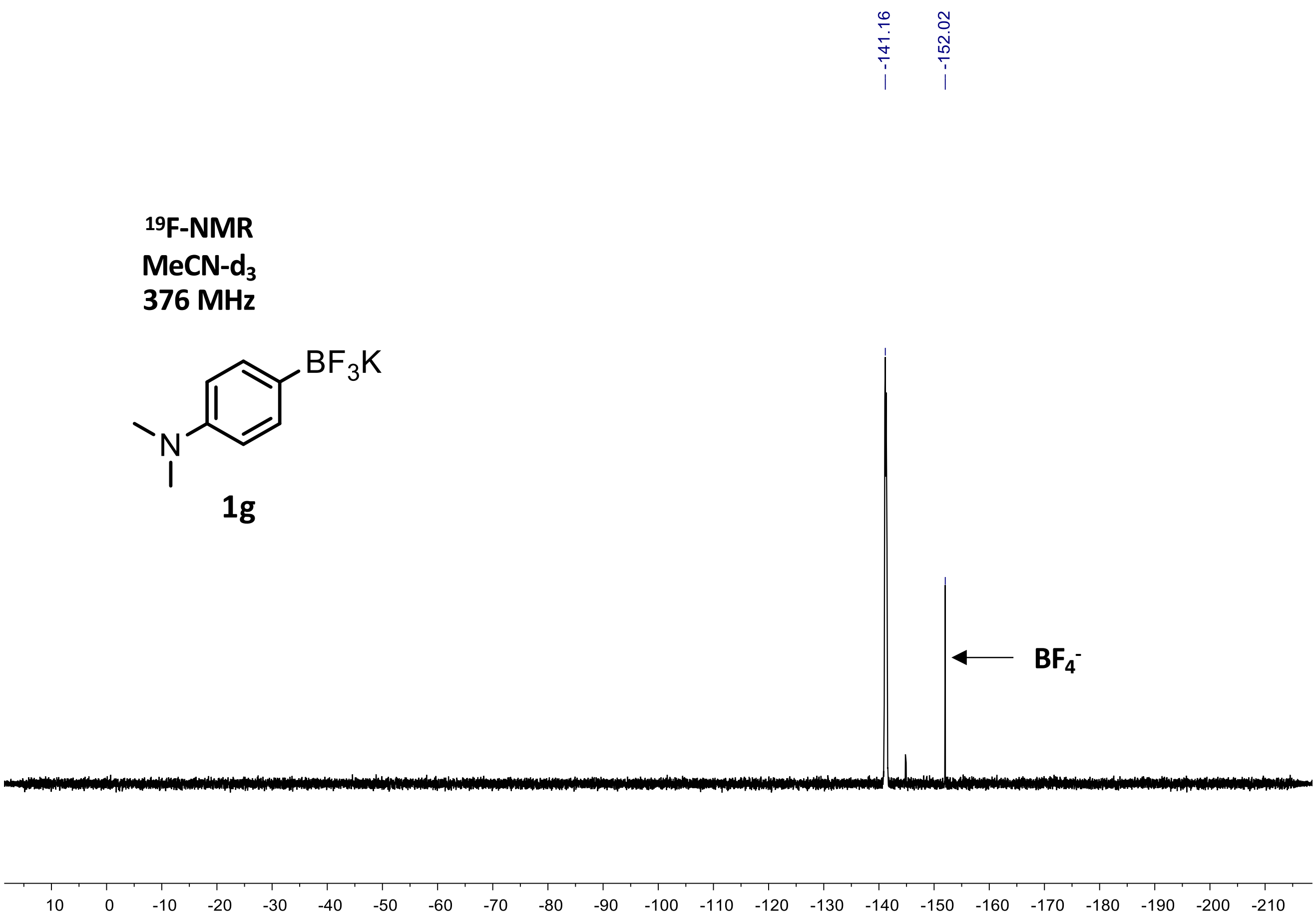

^{19}F-NMR
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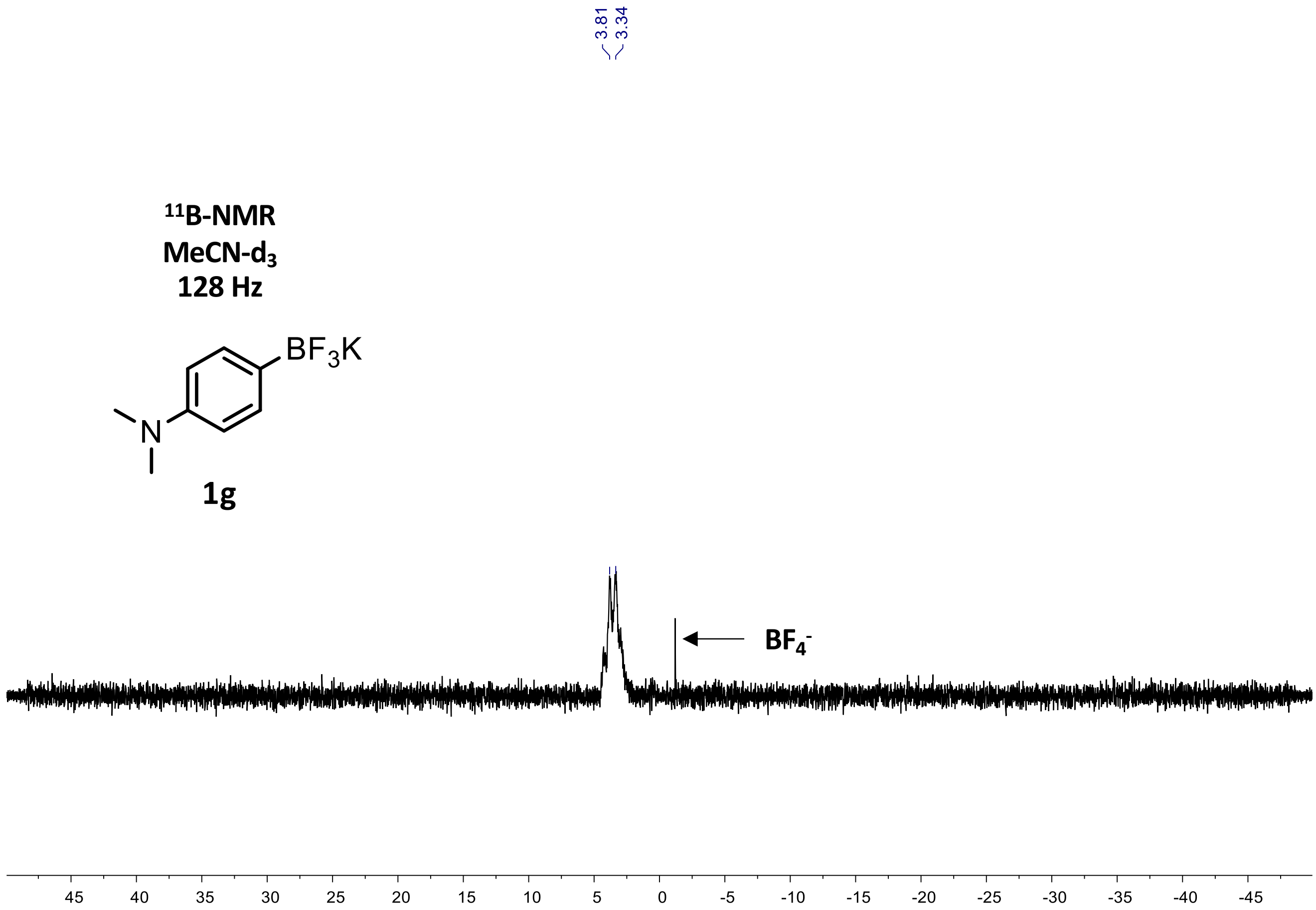
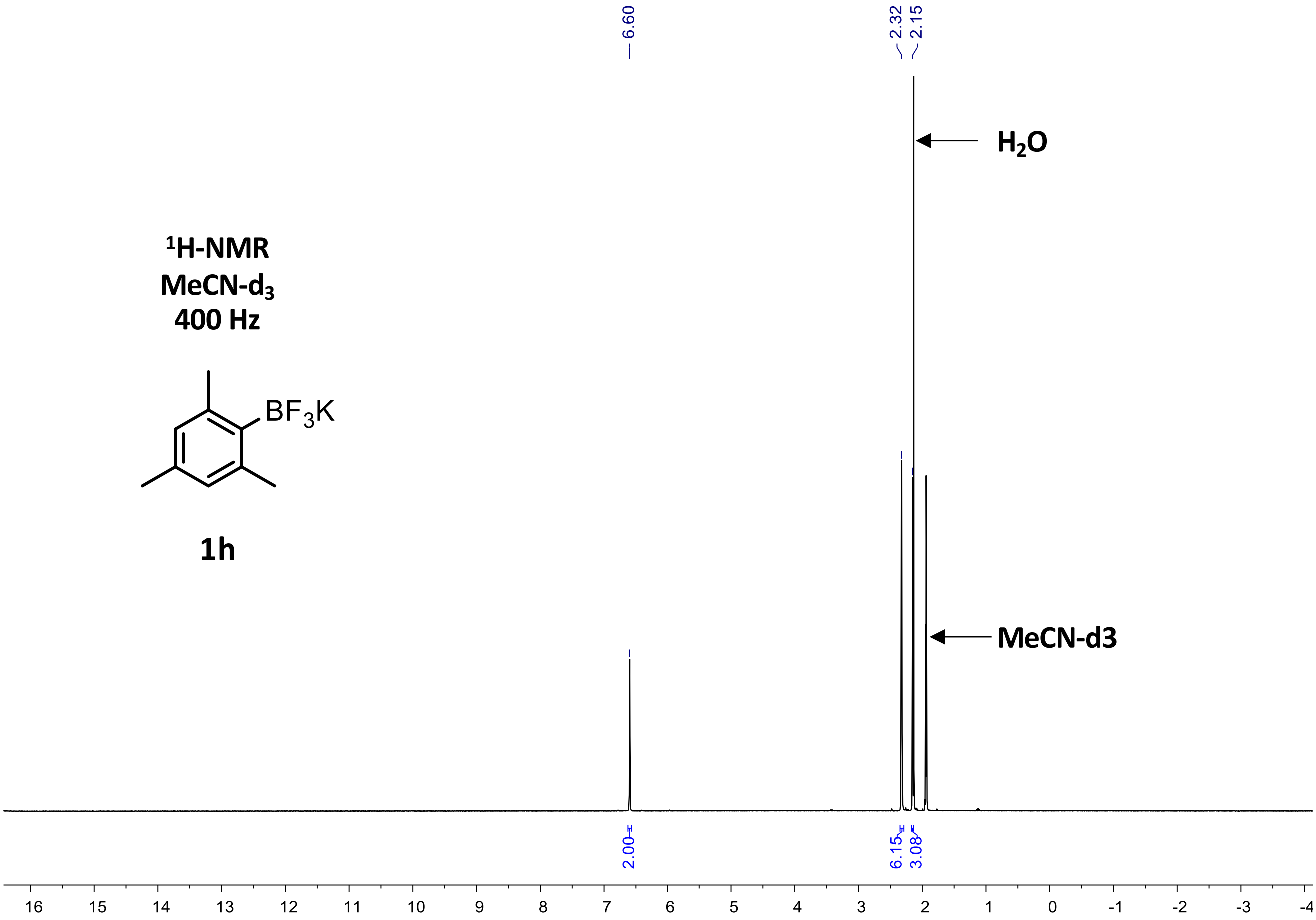
376 MHz

1g



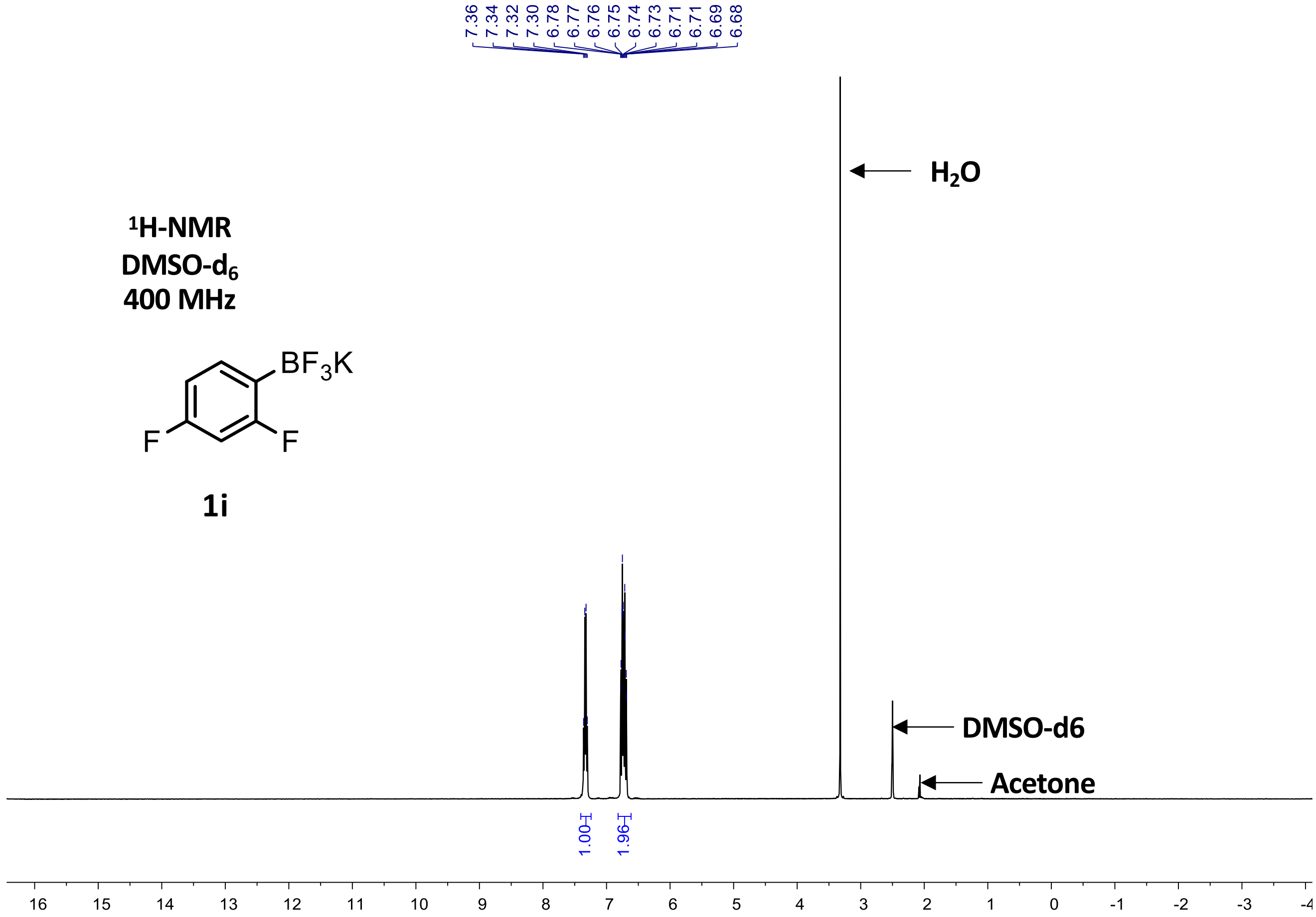
^{11}B-NMR
MeCN-d_3
128 Hz

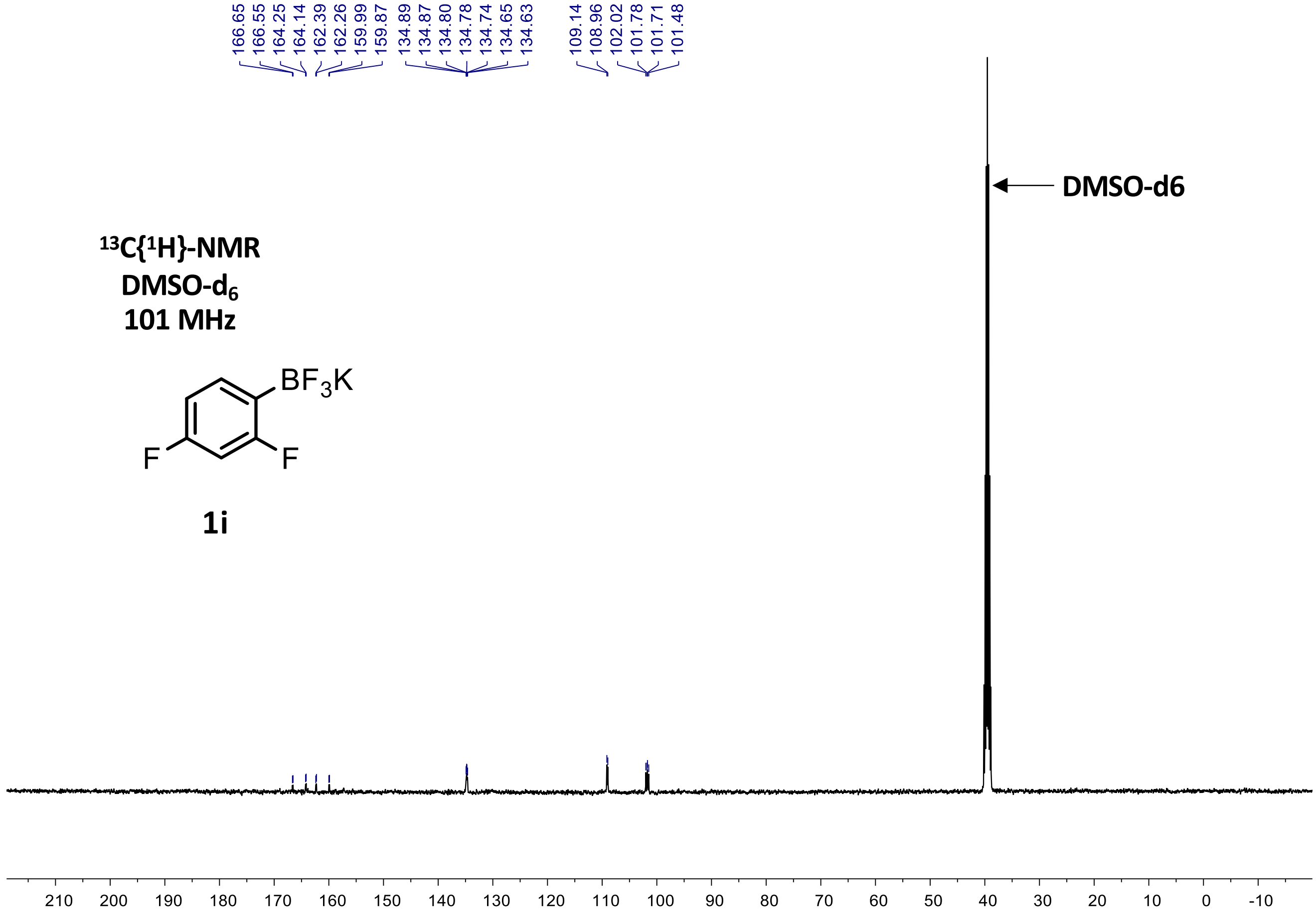
1g

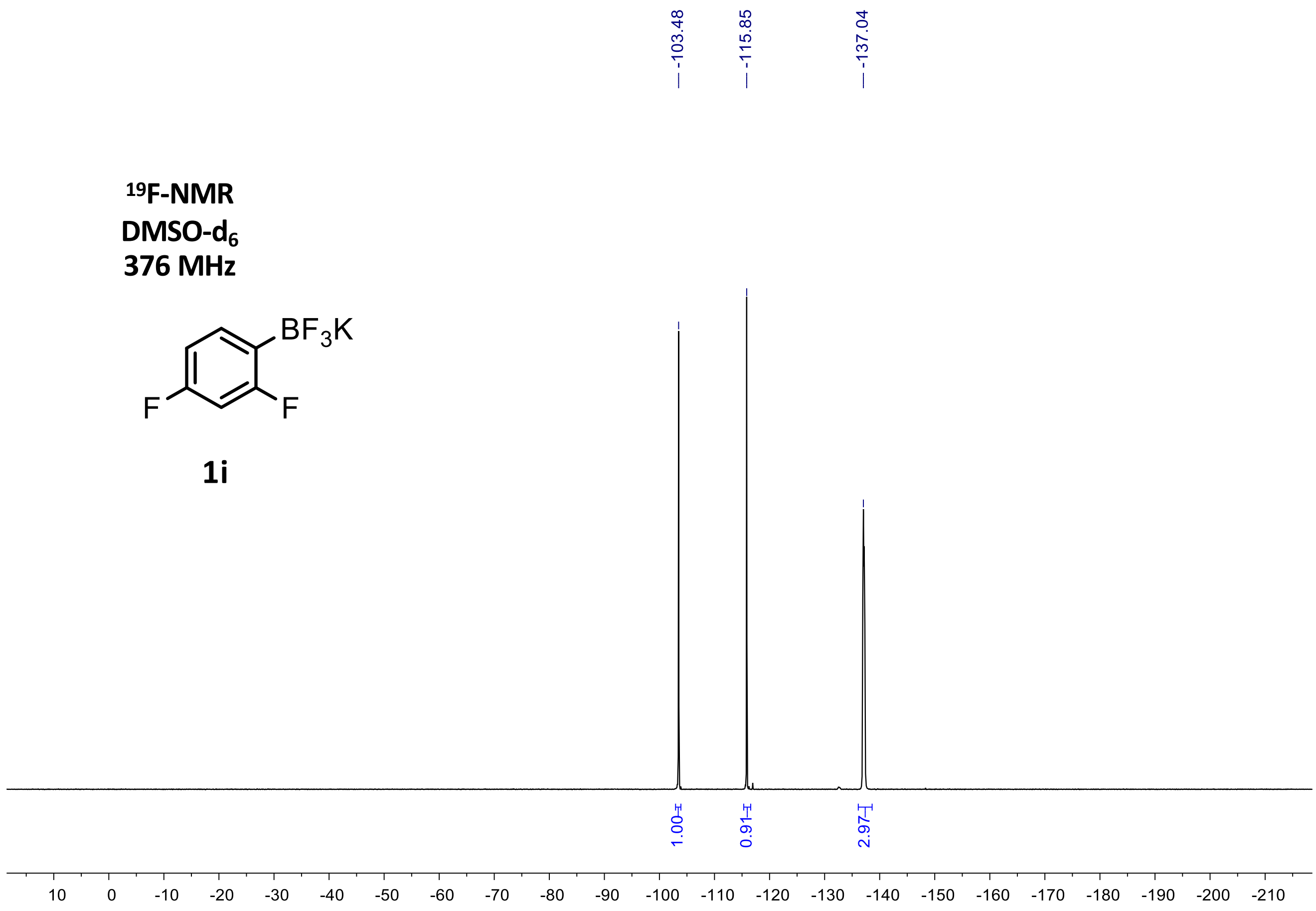


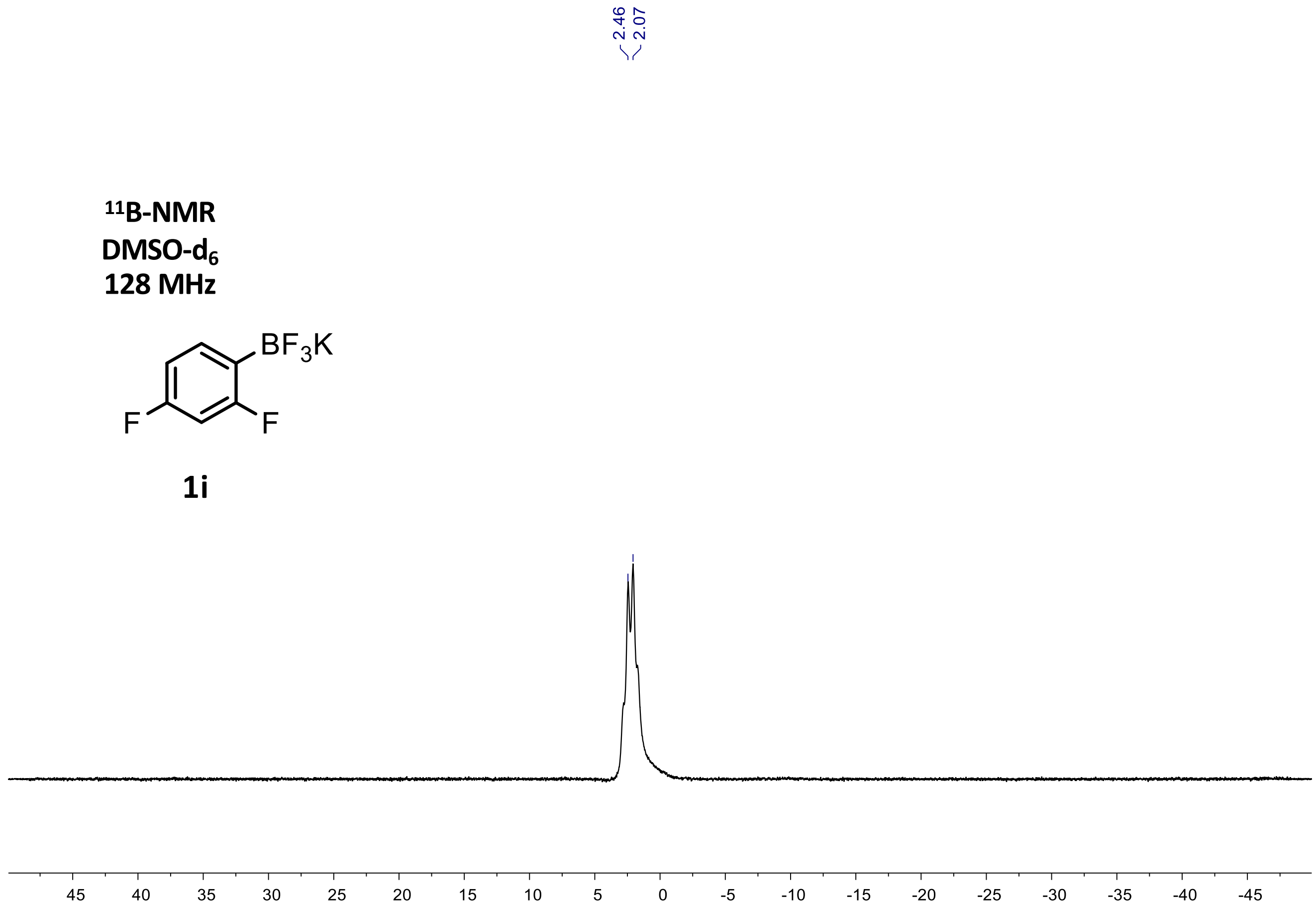
^{1}H-NMR
MeCN-d$_3$
400 Hz
BF$_3$K
6.60
2.32
2.15
H$_2$O
MeCN-d3
2.00
6.15
3.08
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
-4

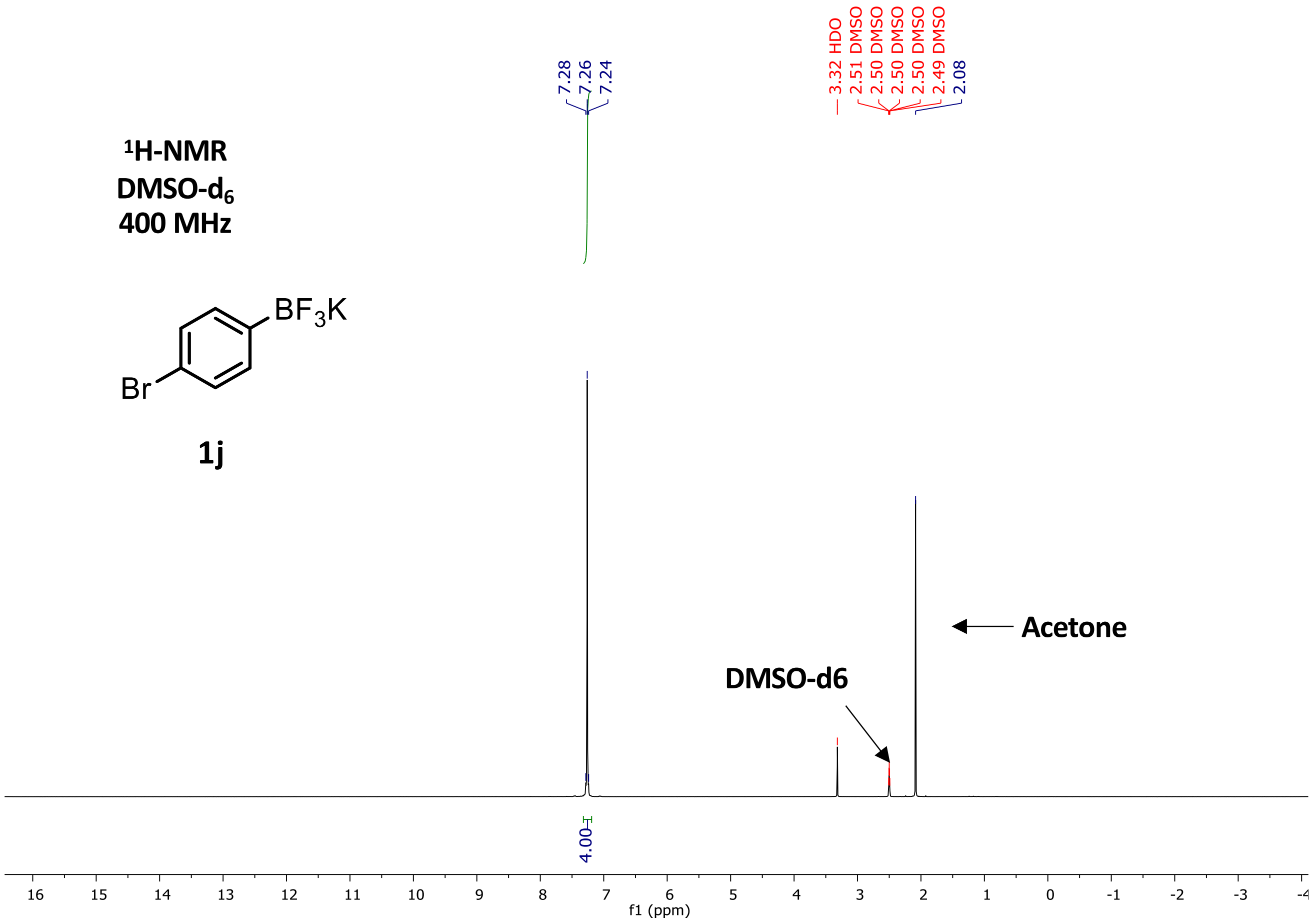
1h

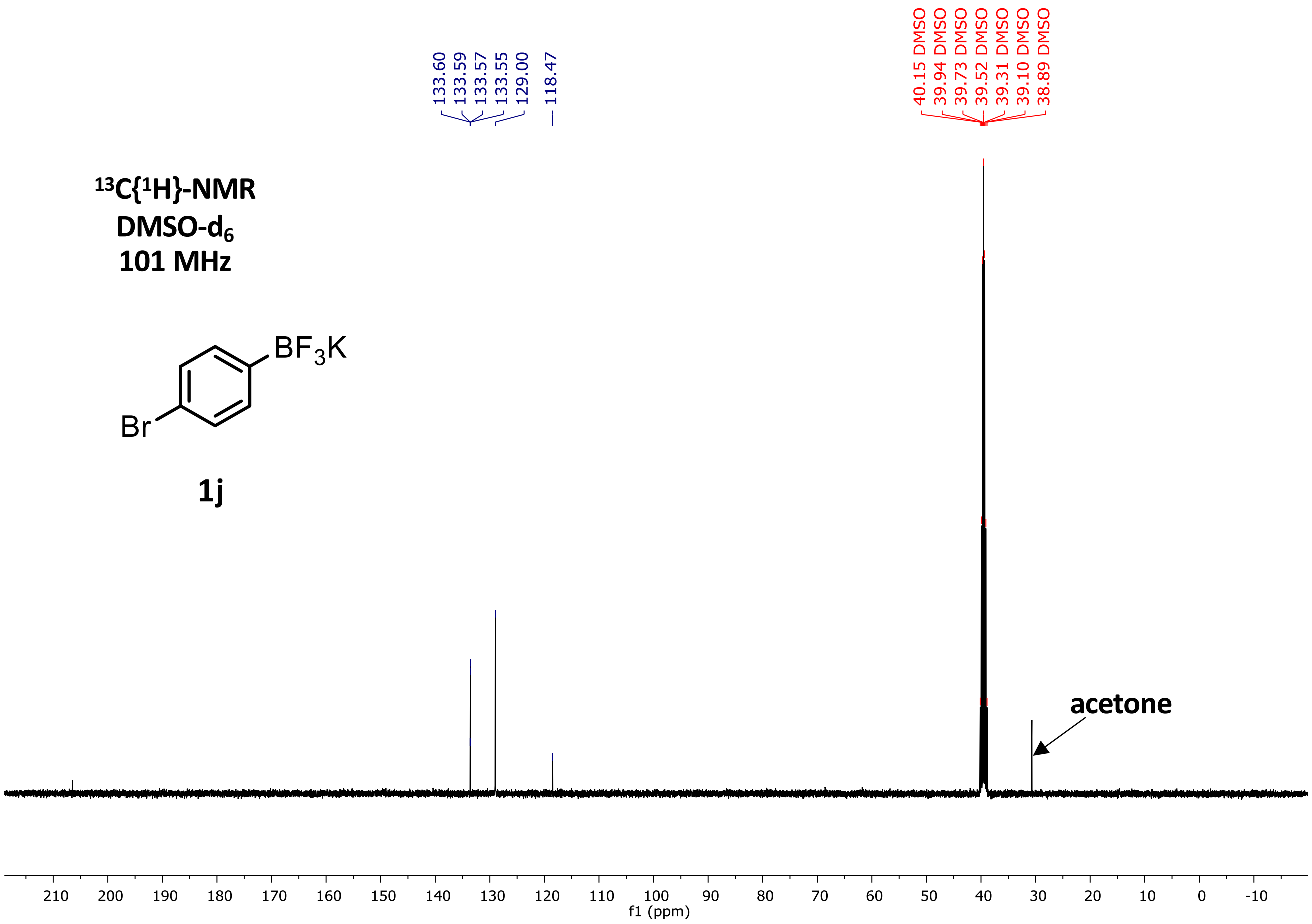

7.36
7.34
7.32
7.30
6.78
6.77
6.76
6.75
6.74
6.73
6.71
6.71
6.69
6.68
^{1}H-NMR
DMSO-d_6
400 MHz
BF$_3$K
F
F
1i
H_2O
DMSO-d6
Acetone
1.00
1.96
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
-4


166.65
166.55
164.25
164.14
162.39
162.26
159.99
159.87
134.89
134.87
134.80
134.78
134.74
134.65
134.63
109.14
108.96
102.02
101.78
101.71
101.48
^{13}C{^{1}H}-NMR
DMSO-d_6
101 MHz
BF$_3$K
F
F
1i
DMSO-d6
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10

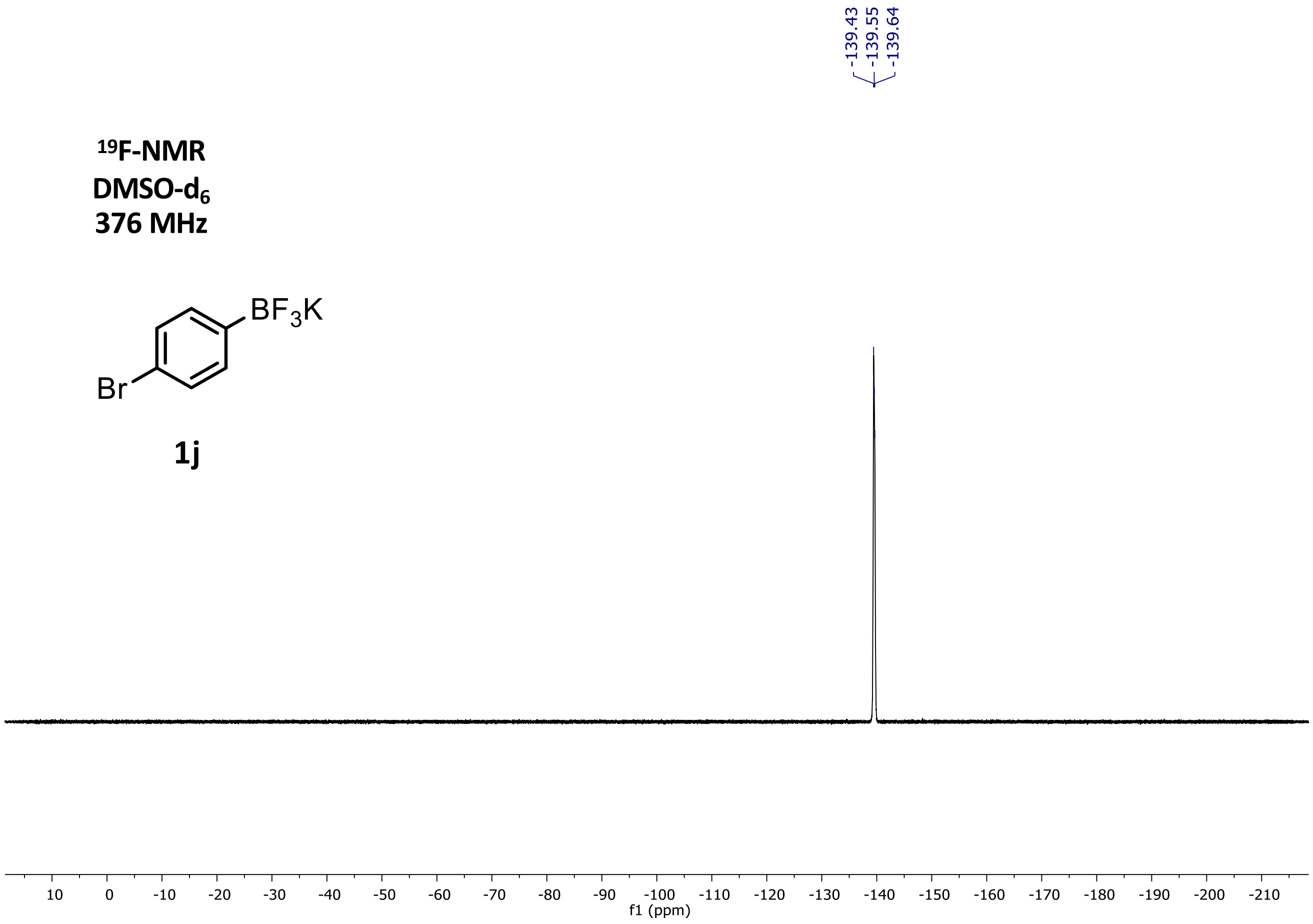

-103.48
-115.85
-137.04
19F-NMR
DMSO-d6
376 MHz
BF3K
F
F
1i
1.00
0.91
2.97
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210


2.46
2.07
11B-NMR
DMSO-d6
128 MHz
BF3K
F
F
1i
45 40 35 30 25 20 15 10 5 0 -5 -10 -15 -20 -25 -30 -35 -40 -45

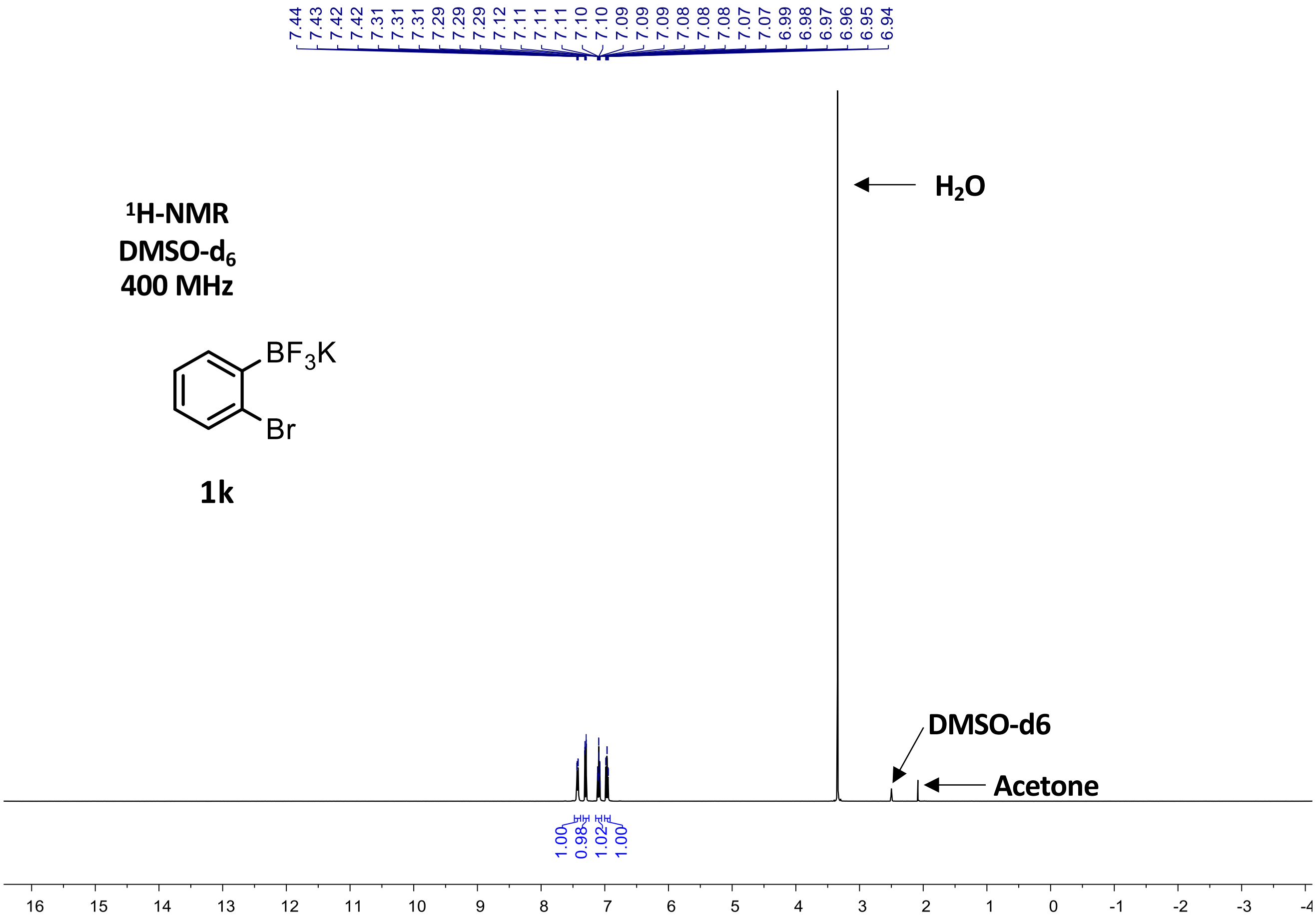

7.28
7.26
7.24
3.32 HDO
2.51 DMSO
2.50 DMSO
2.50 DMSO
2.50 DMSO
2.49 DMSO
2.08
^{1}H-NMR
DMSO-d_6
400 MHz
BF$_3$K
Br
1j
Acetone
DMSO-d6
4.00
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
-4
f1 (ppm)

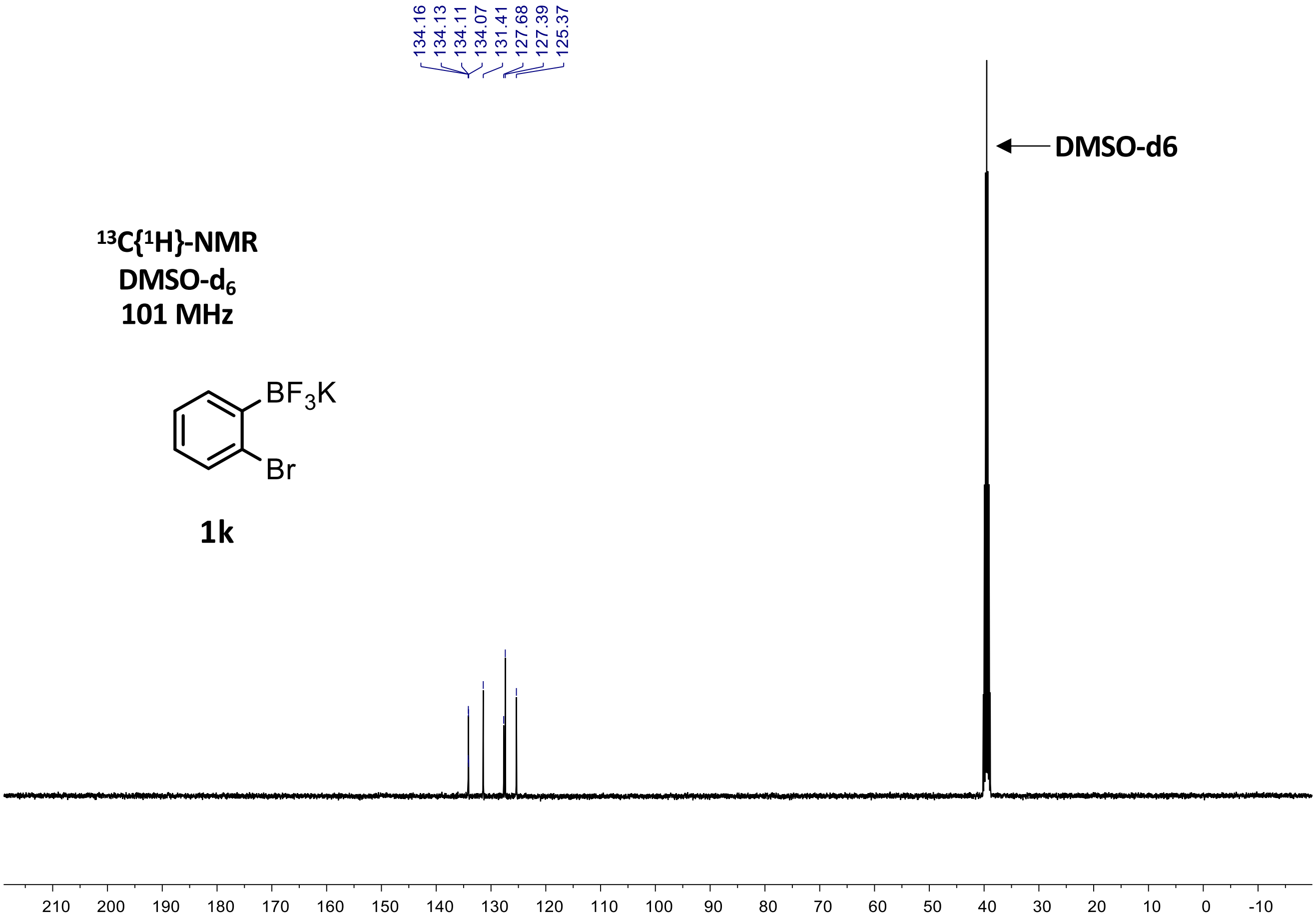

133.60
133.59
133.57
133.55
129.00
118.47
40.15 DMSO
39.94 DMSO
39.73 DMSO
39.52 DMSO
39.31 DMSO
39.10 DMSO
38.89 DMSO
^{13}C{^{1}H}-NMR
DMSO-d_6
101 MHz
BF$_3$K
Br
1j
acetone
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

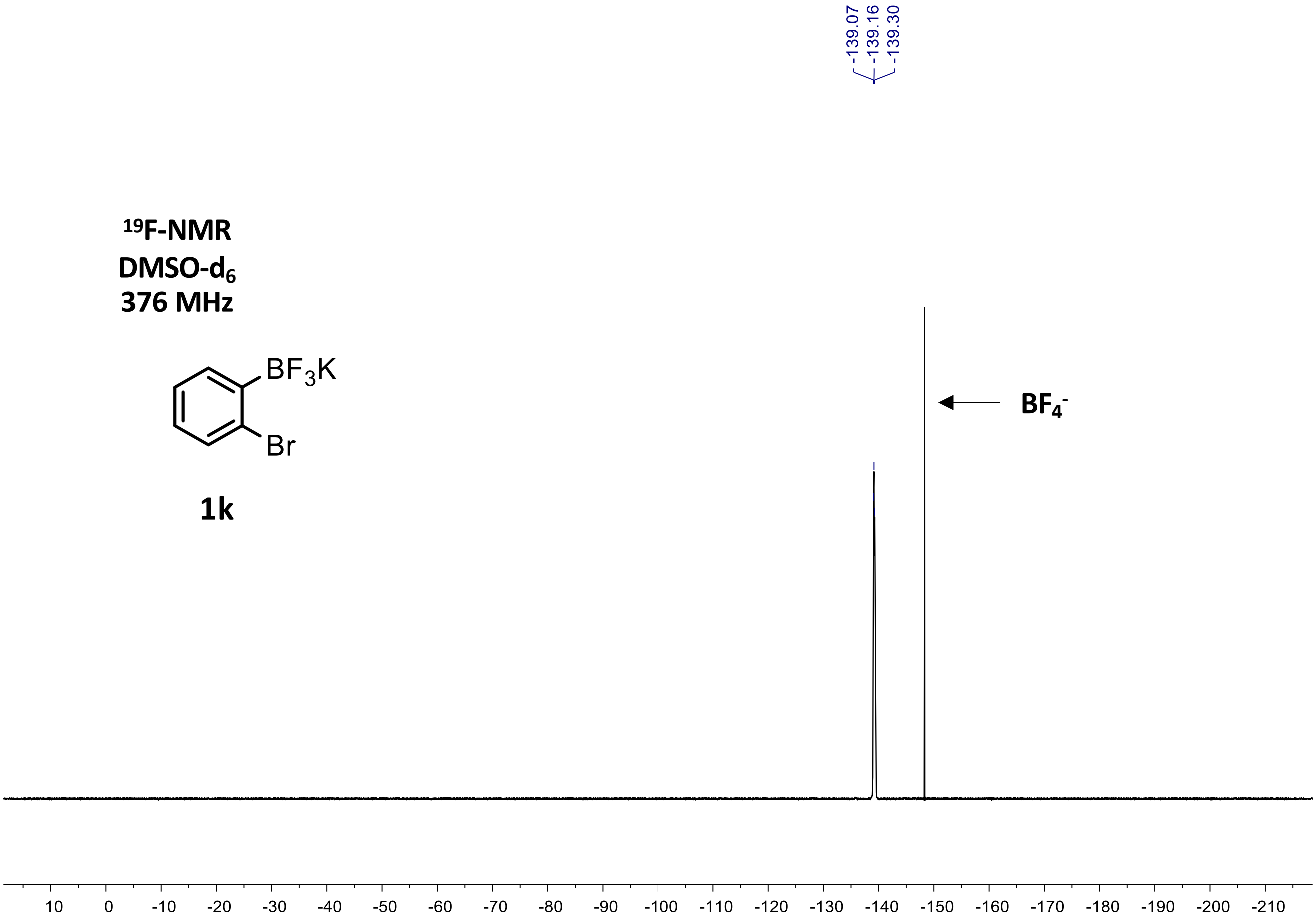
^{19}F-NMR
DMSO-d_6
376 MHz

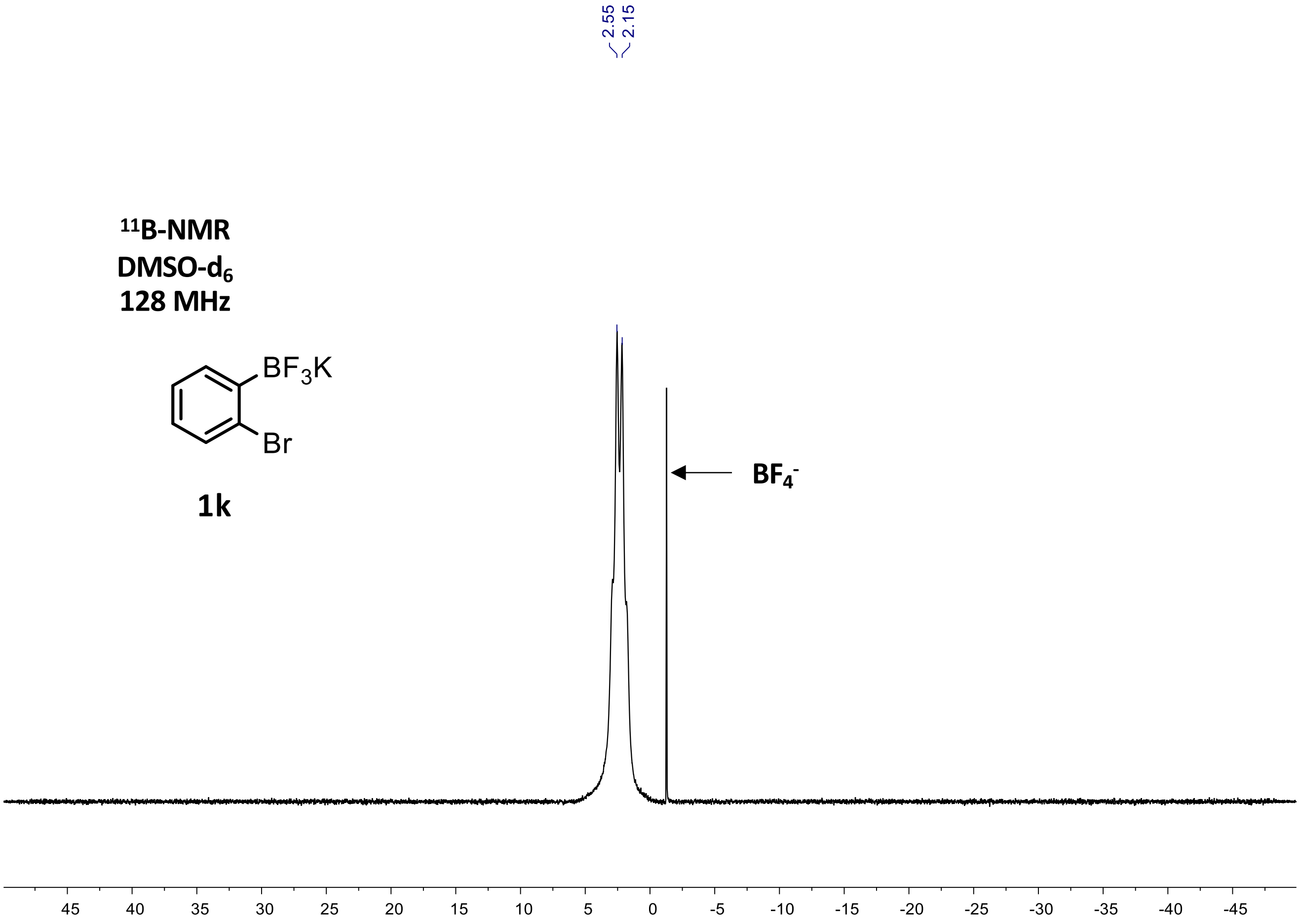


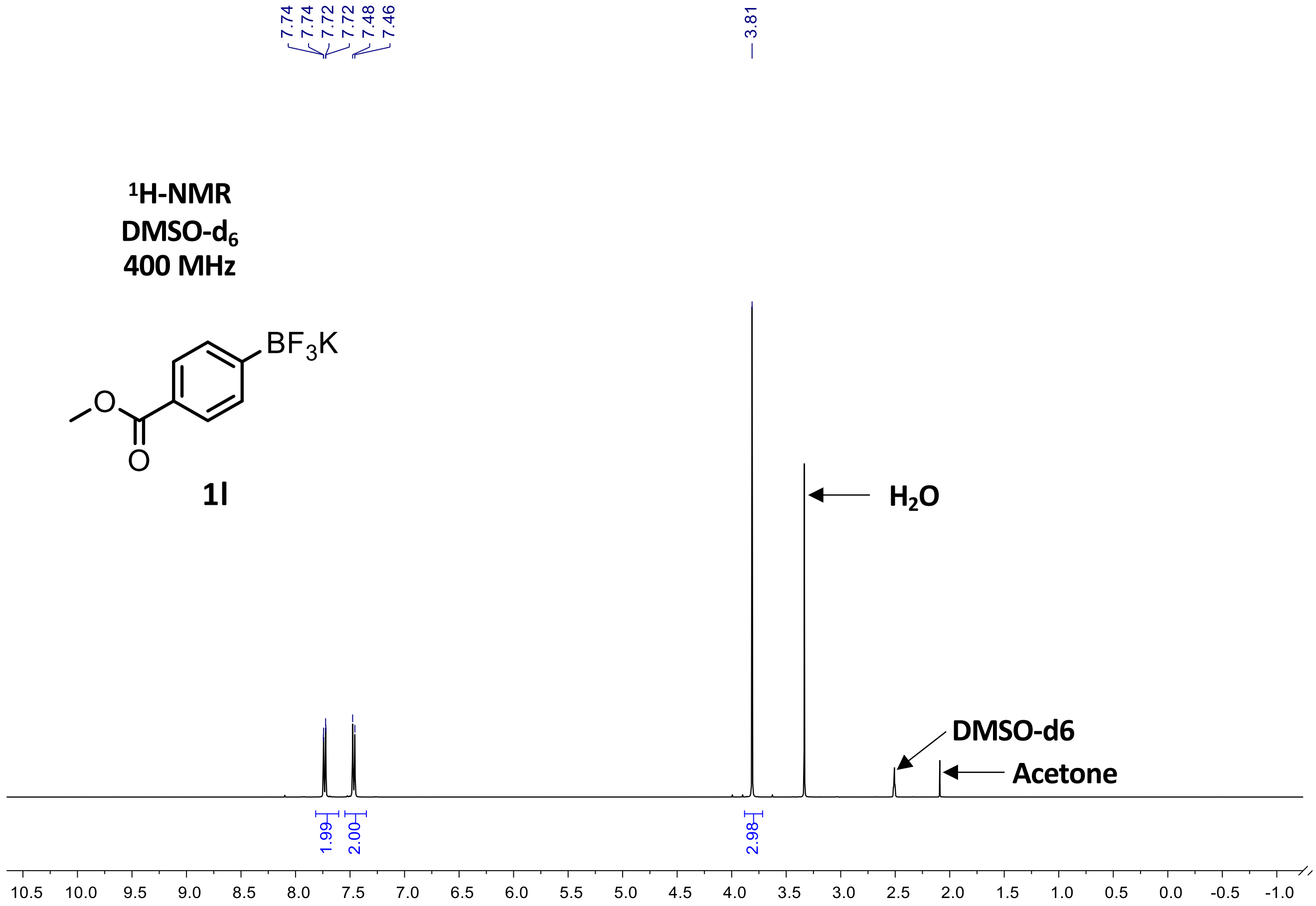
1j

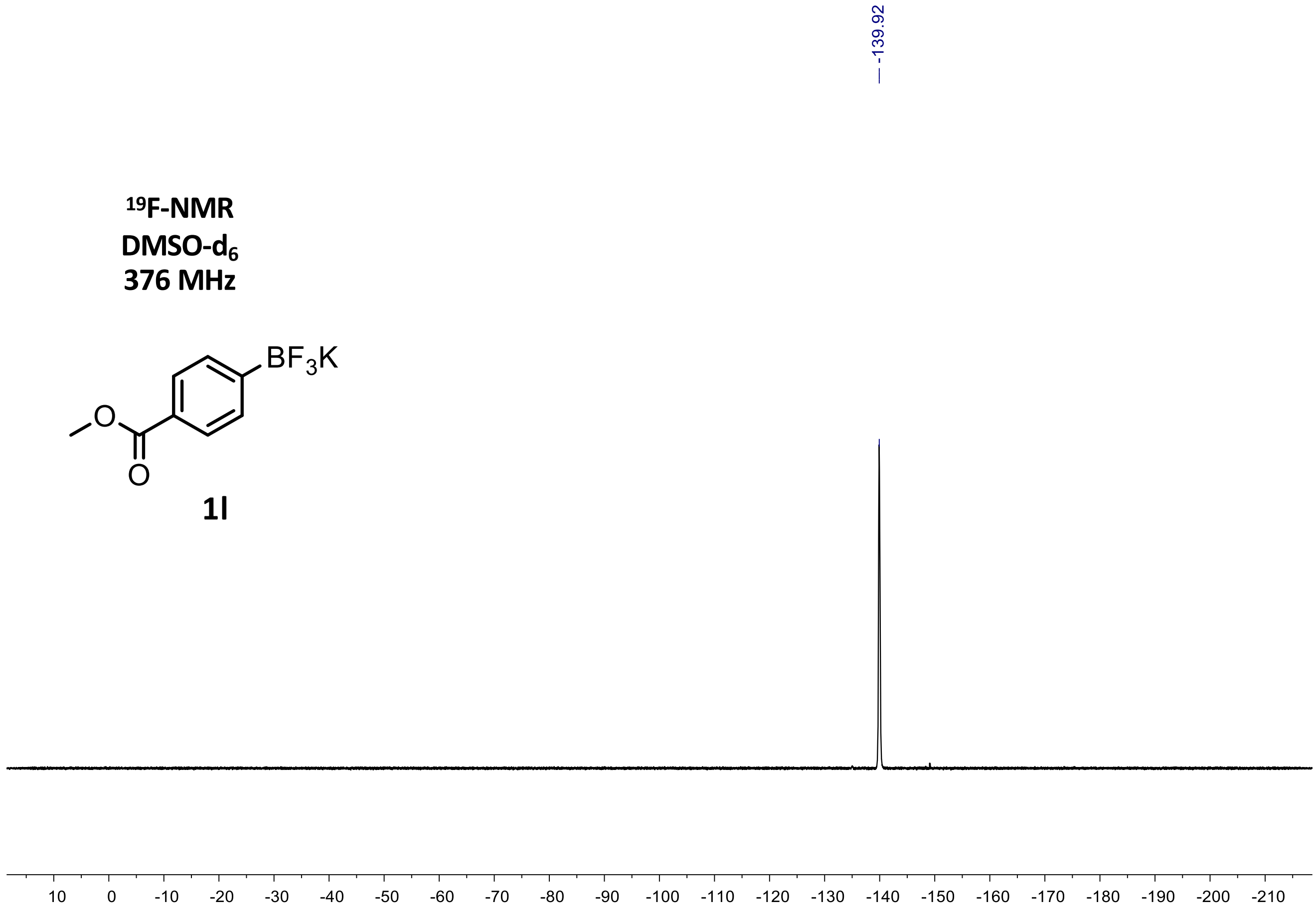

7.44
7.43
7.42
7.42
7.31
7.31
7.31
7.29
7.29
7.29
7.12
7.11
7.11
7.11
7.10
7.10
7.09
7.09
7.09
7.08
7.08
7.08
7.07
7.07
6.99
6.98
6.97
6.96
6.95
6.94
¹H-NMR
DMSO-d6
400 MHz
BF3K
Br
1k
H2O
DMSO-d6
Acetone
1.00
0.98
1.02
1.00
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4

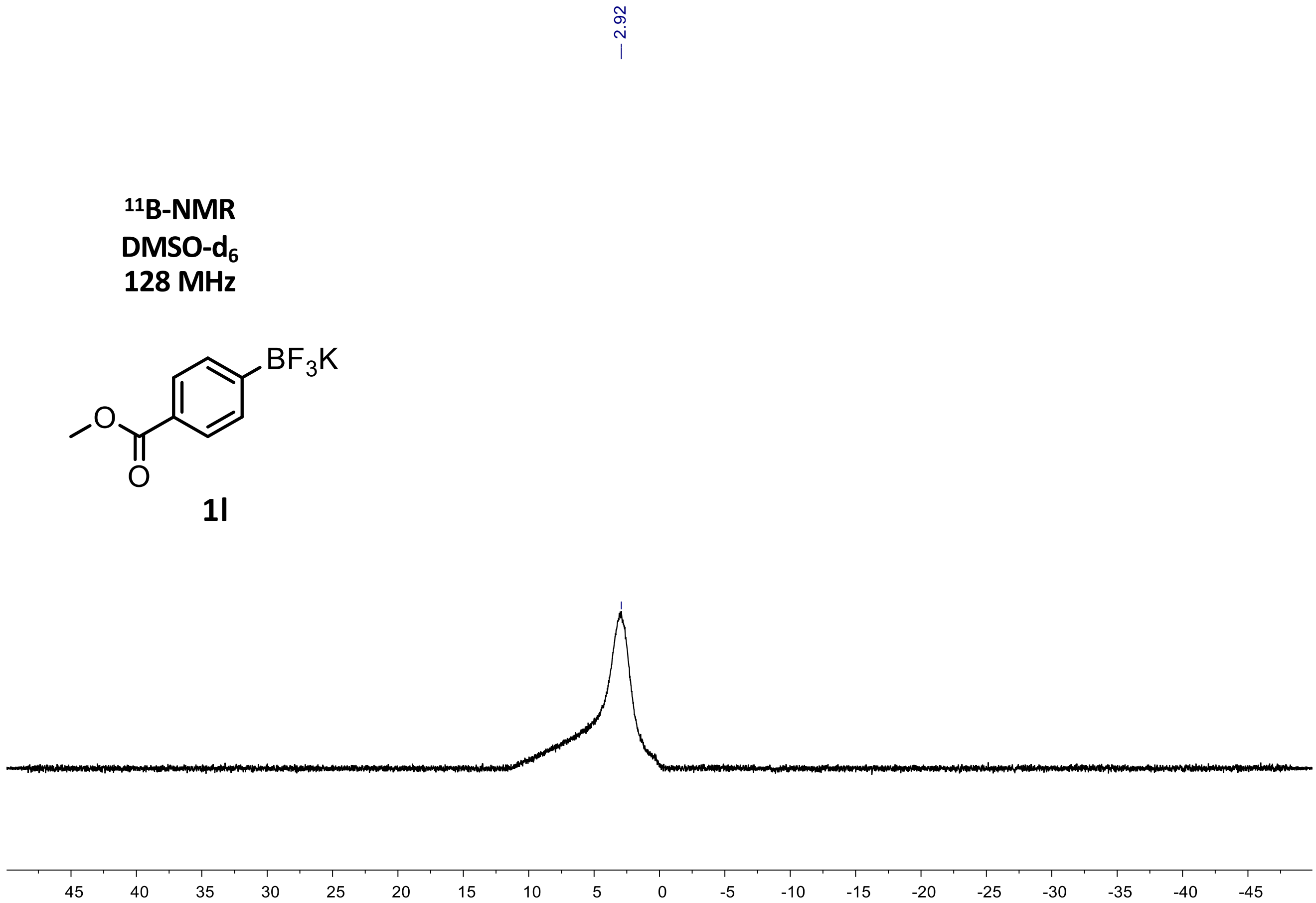

134.16
134.13
134.11
134.07
131.41
127.68
127.39
125.37
¹³C{¹H}-NMR
DMSO-d6
101 MHz
BF3K
Br
1k
DMSO-d6
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

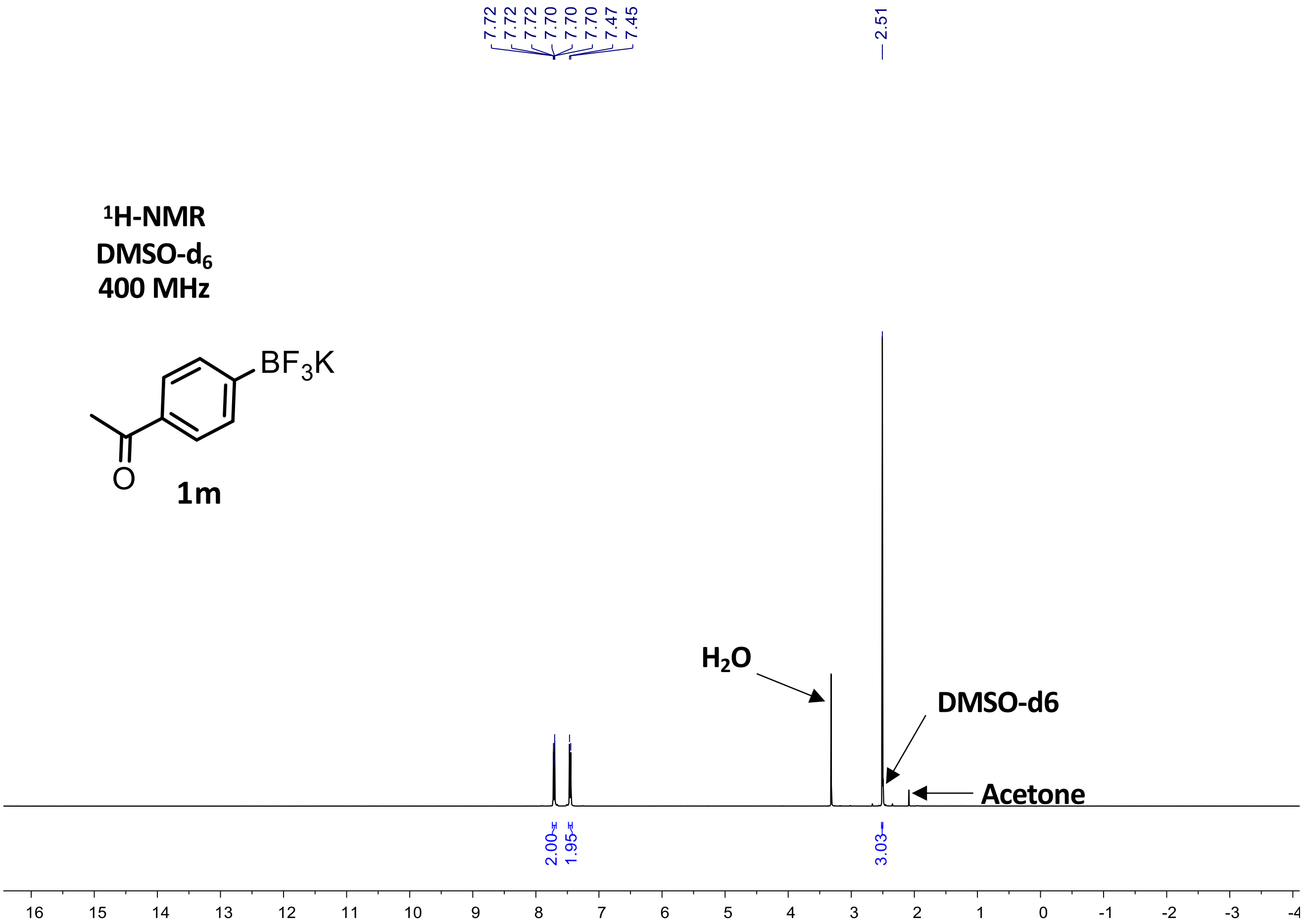

-139.07
-139.16
-139.30
19F-NMR
DMSO-d6
376 MHz
BF3K
Br
1k
BF4-
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210

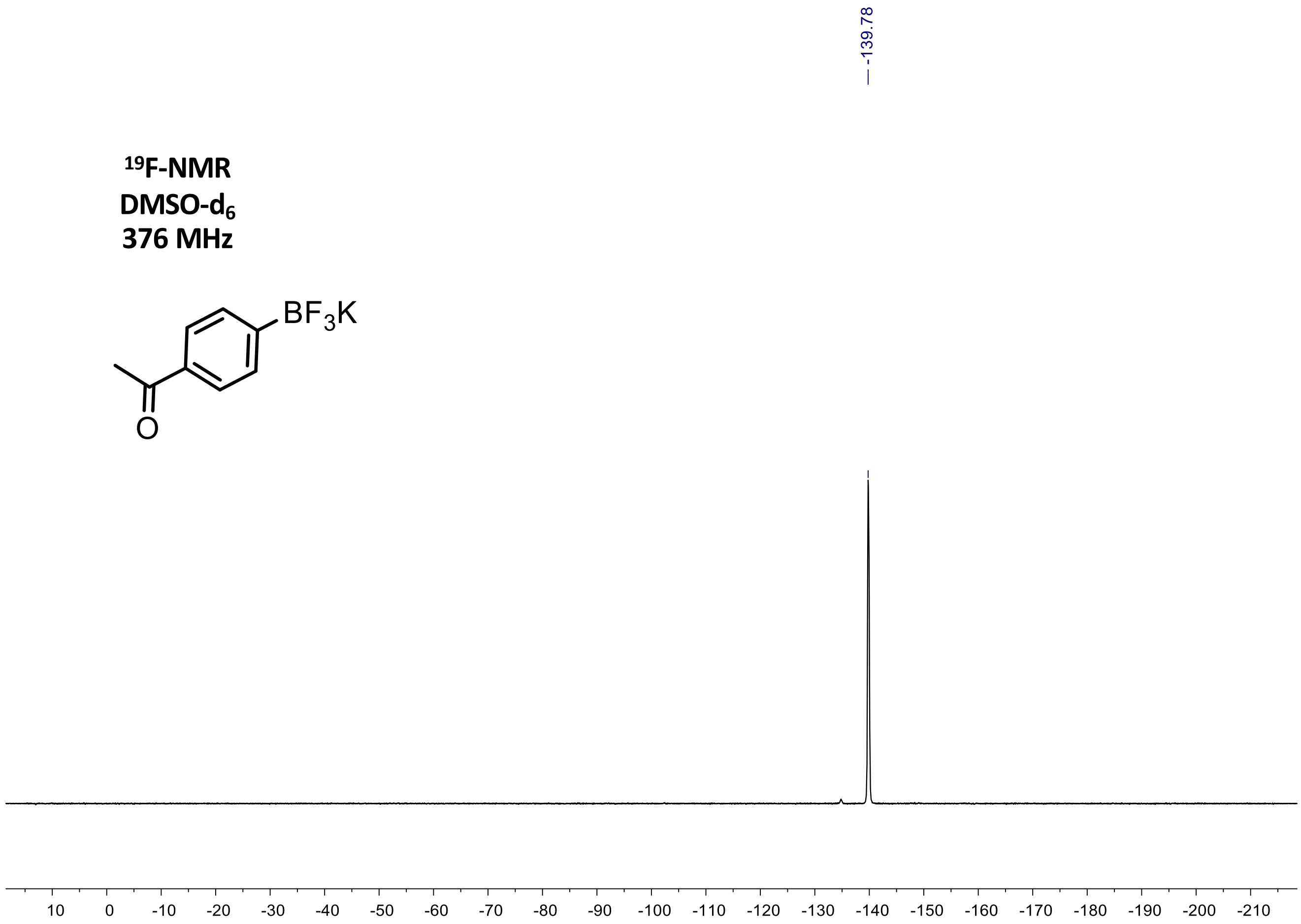

2.55
2.15
11B-NMR
DMSO-d6
128 MHz
BF3K
Br
1k
BF4-
45
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
-45


7.74
7.74
7.72
7.72
7.48
7.46
3.81
¹H-NMR
DMSO-d₆
400 MHz
BF₃K
O
O
1l
H₂O
DMSO-d6
Acetone
1.99
2.00
2.98
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0

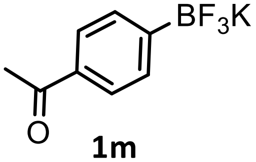

-139.92
^{19}F-NMR
DMSO-d_6
376 MHz
BF_3K
O
O
1l
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210


2.92
^{11}B-NMR
DMSO-d_6
128 MHz
BF_3K
O
O
1l
45
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
-45

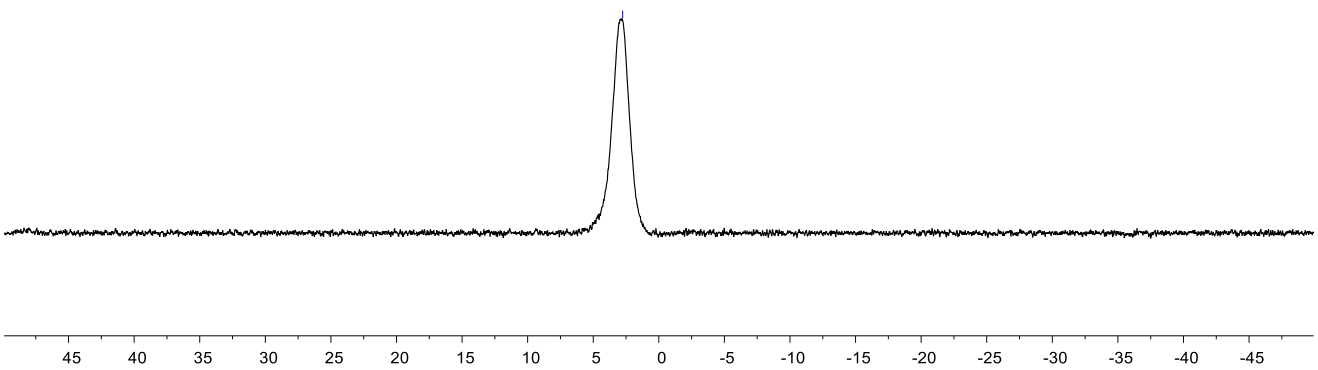

7.72
7.72
7.72
7.70
7.70
7.70
7.47
7.45
2.51
^{1}H-NMR
DMSO-d$_6$
400 MHz
BF$_3$K
O
1m
H$_2$O
DMSO-d6
Acetone
2.00
1.95
3.03
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
-4

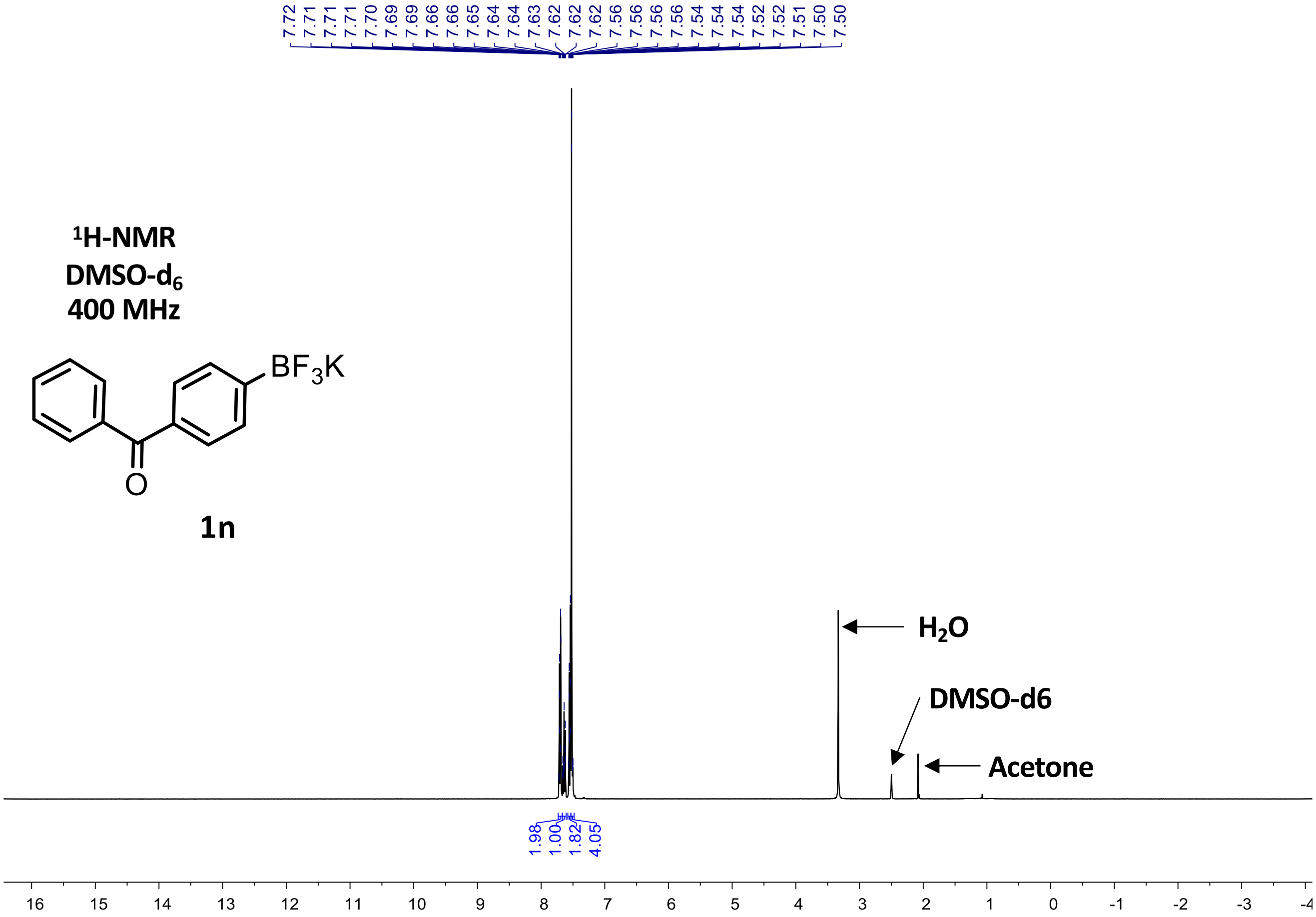

-139.78
^{19}F-NMR
DMSO-d$_6$
376 MHz
BF$_3$K
O
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210

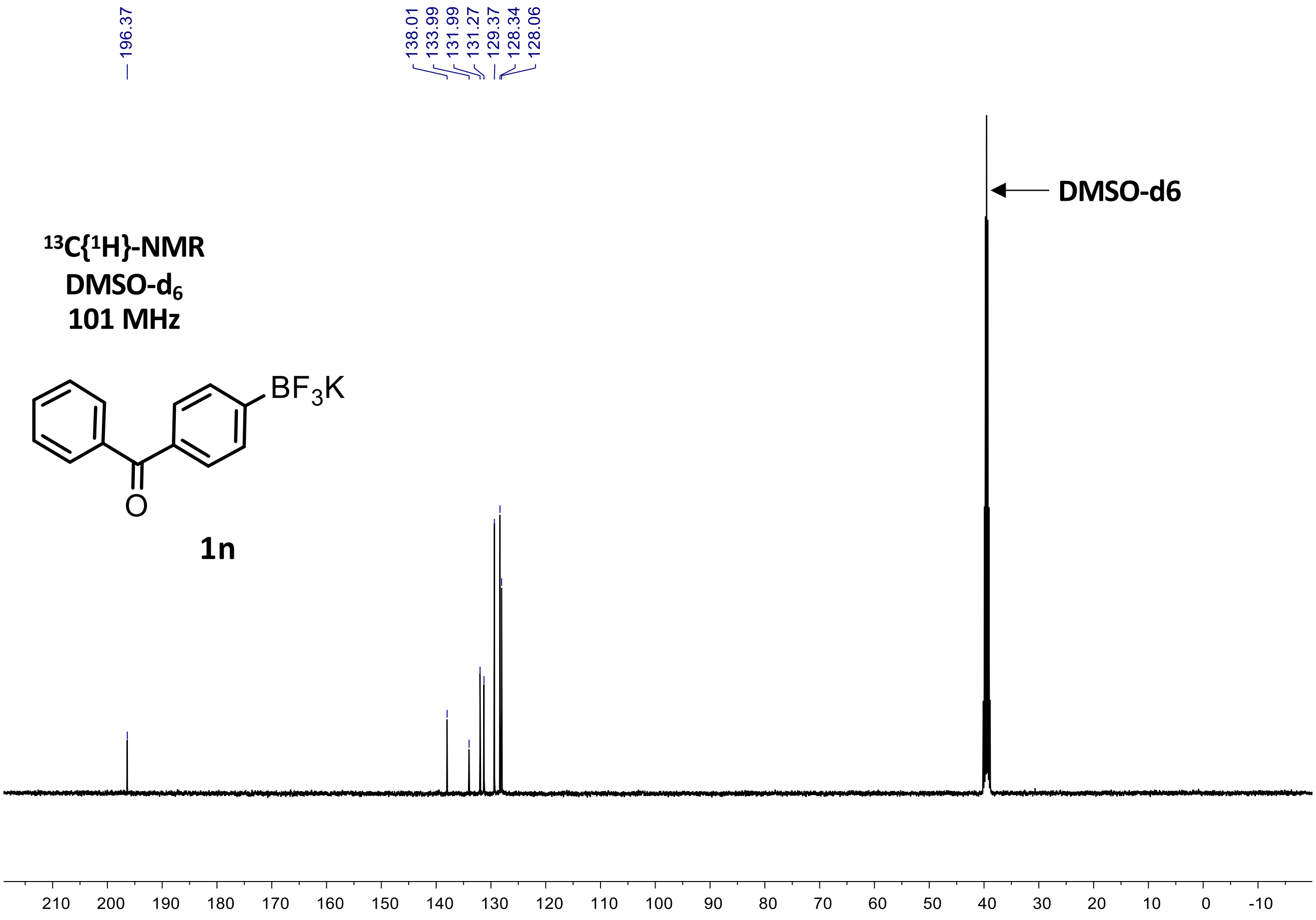
^{11}B-NMR
DMSO-d_6
128 MHz

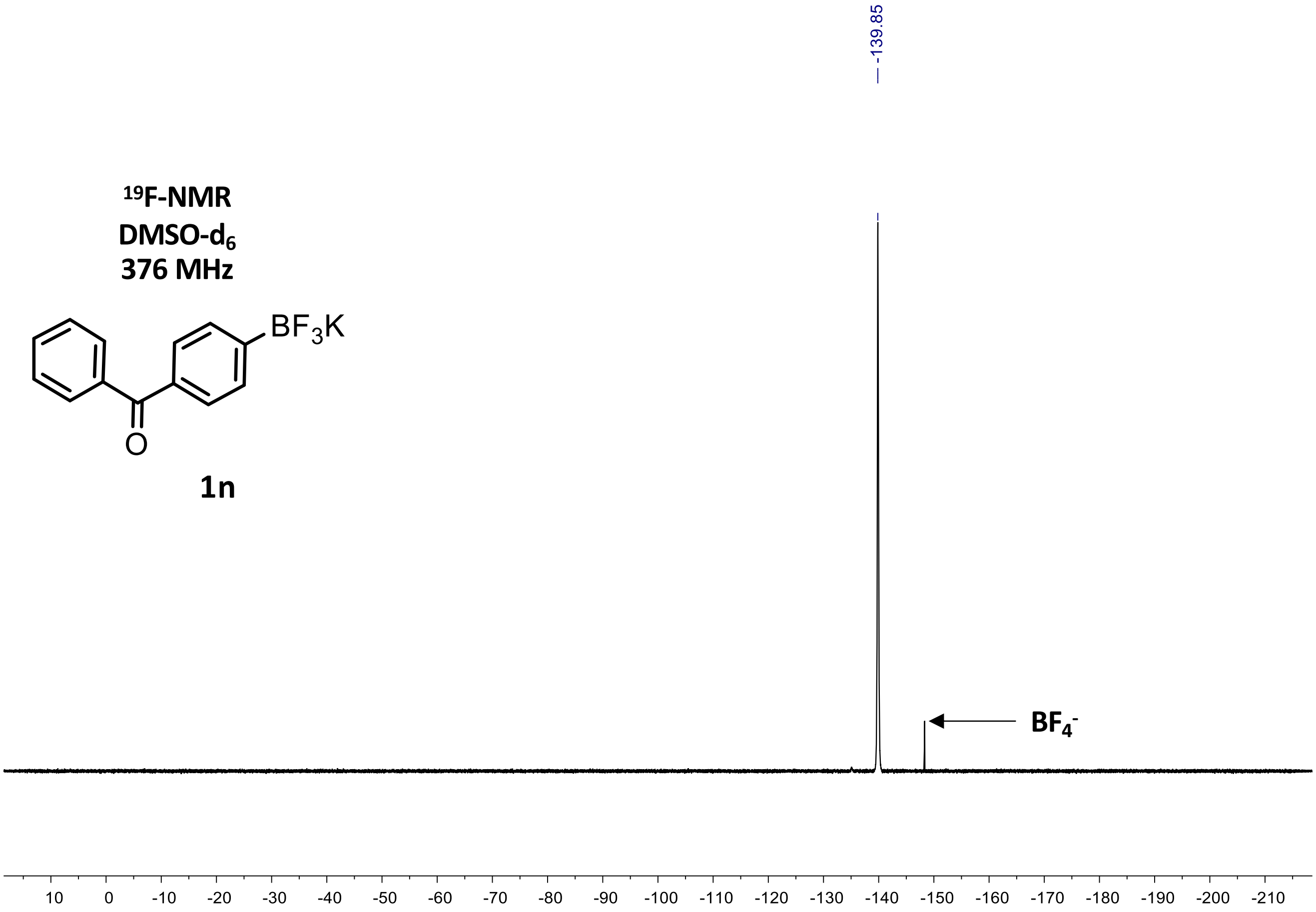


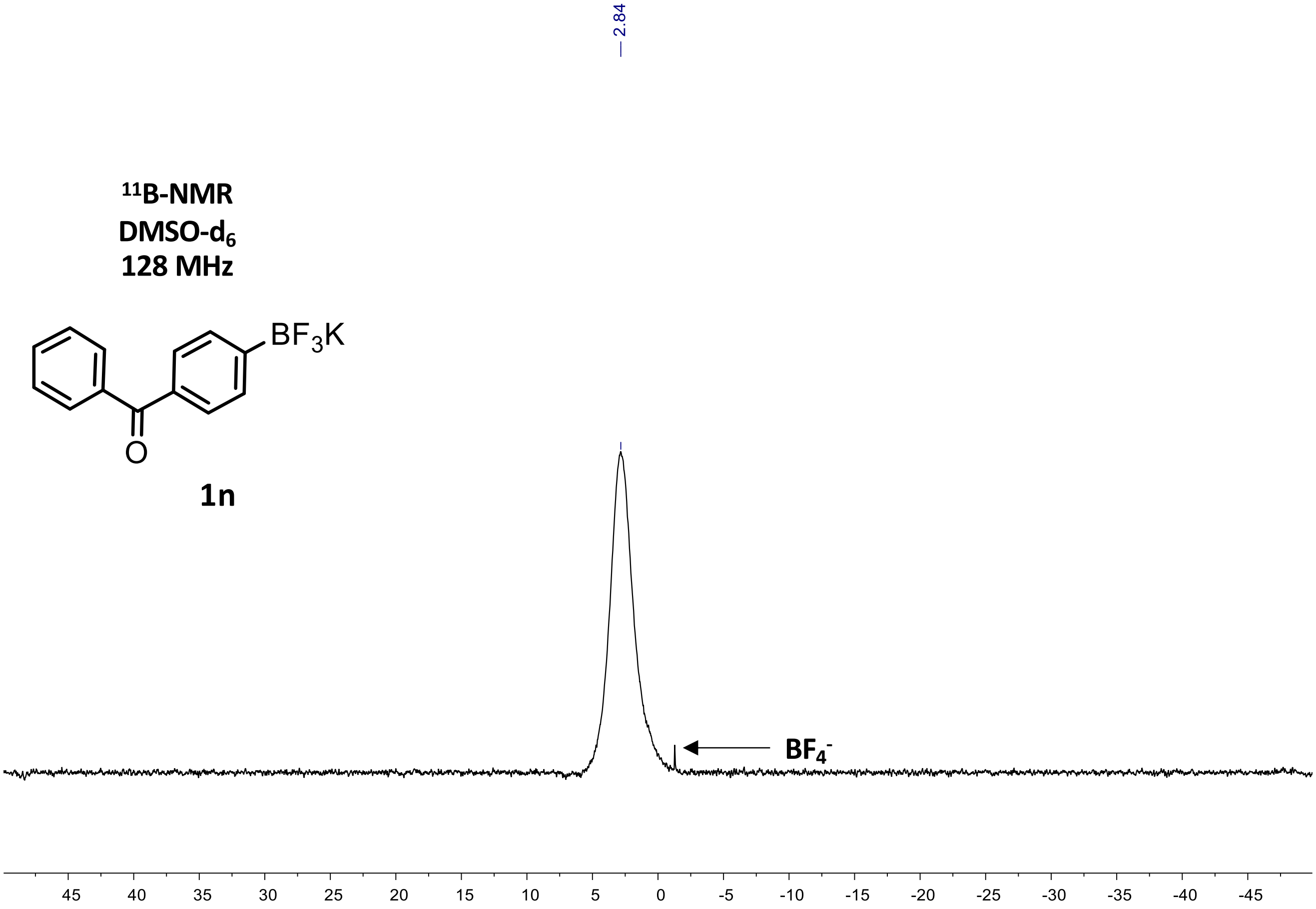
1m

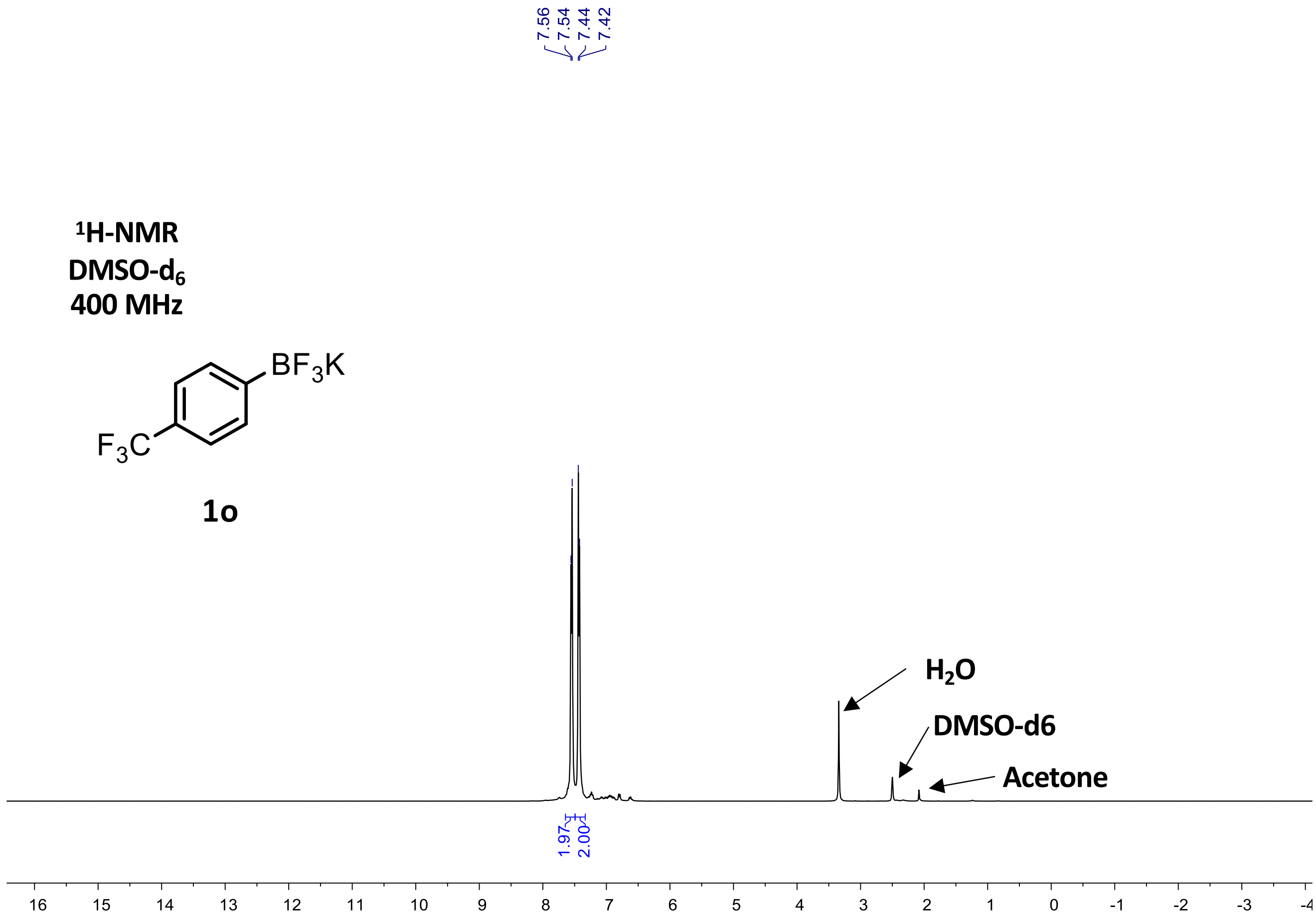


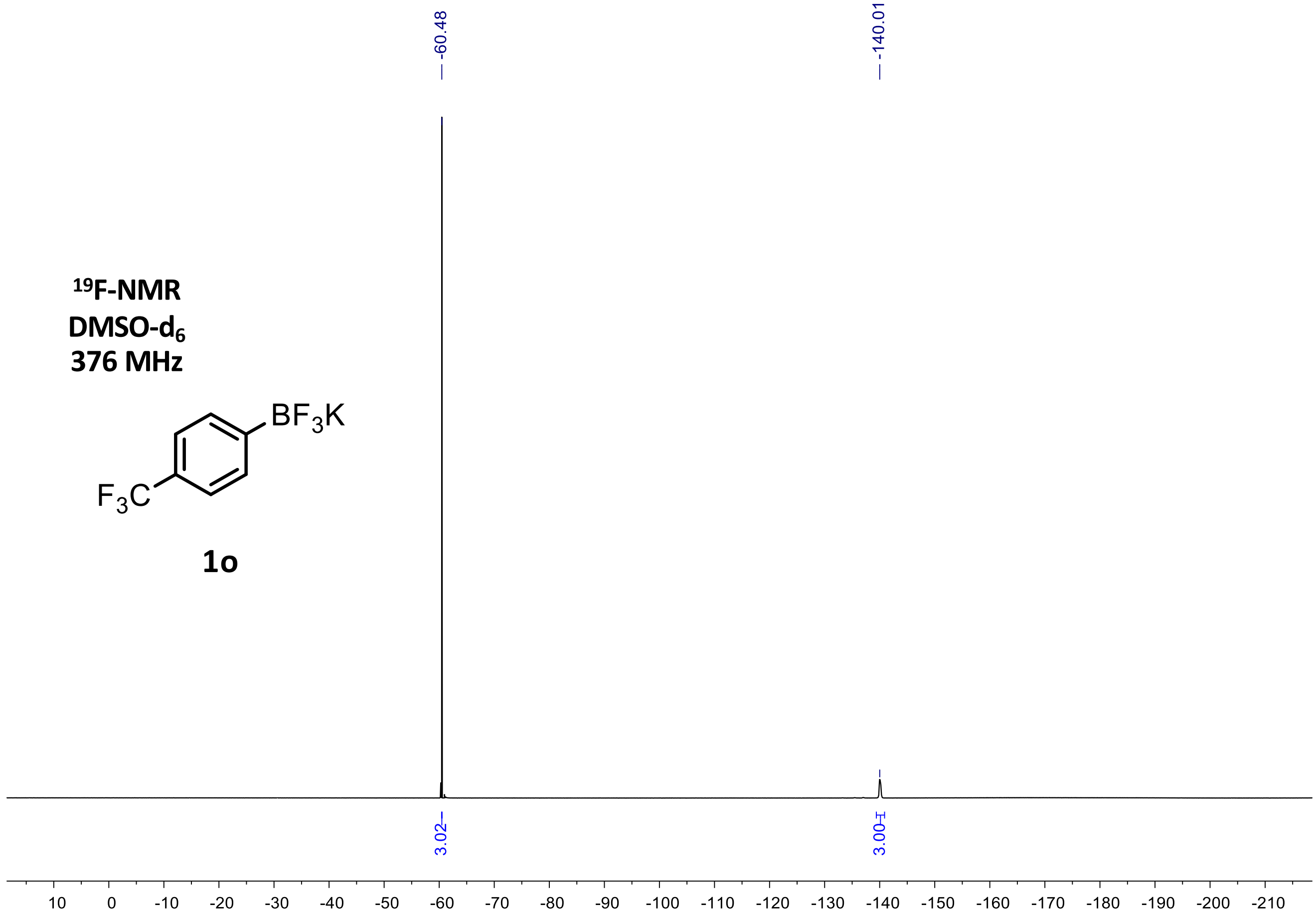

7.72
7.71
7.71
7.70
7.69
7.69
7.66
7.65
7.64
7.63
7.62
7.62
7.62
7.56
7.56
7.56
7.54
7.54
7.54
7.52
7.52
7.51
7.50
7.50
^{1}H-NMR
DMSO-d_6
400 MHz
BF_3K
O
1n
H_2O
DMSO-d6
Acetone
1.98
1.00
1.82
4.05
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
-4

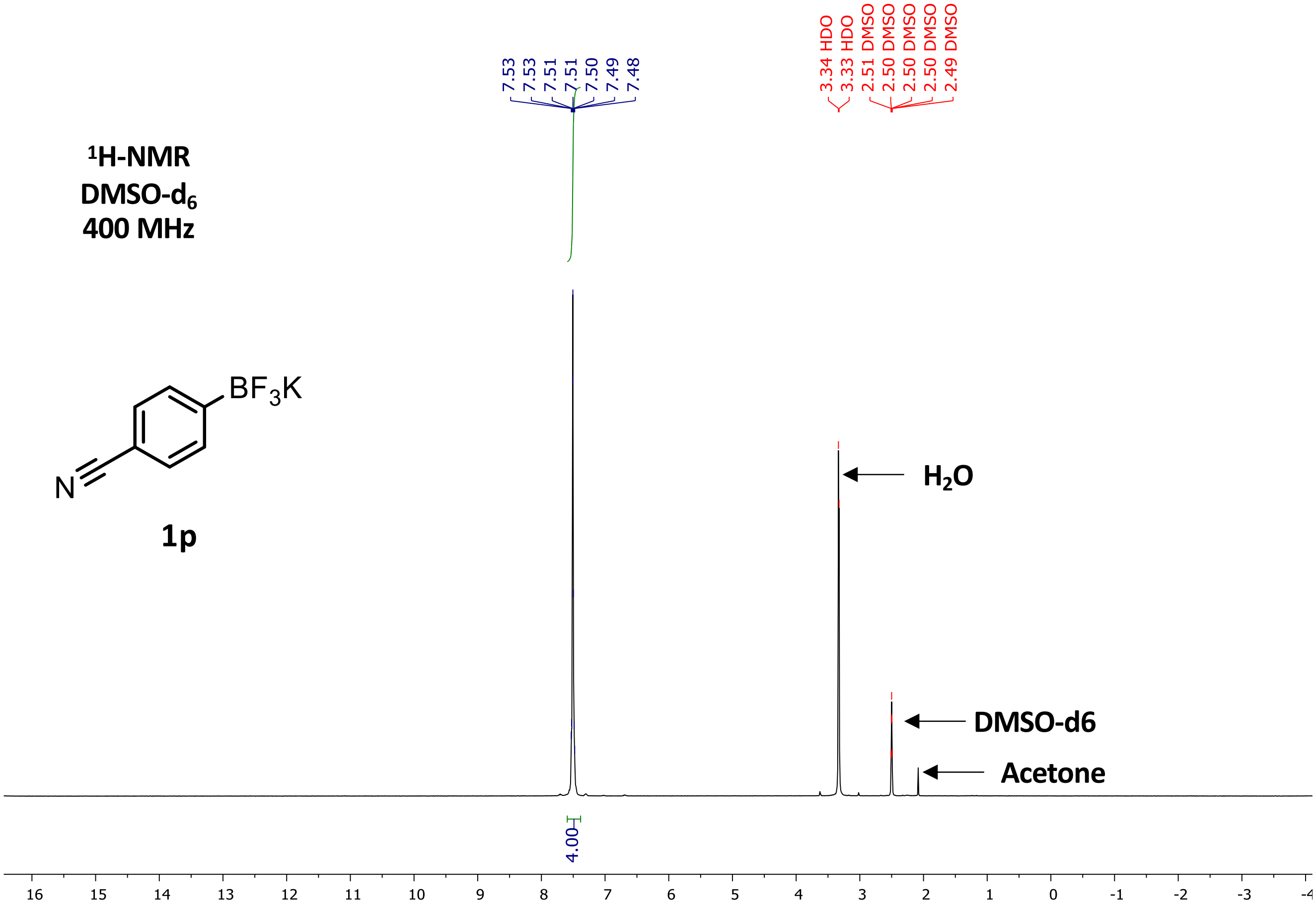

196.37
138.01
133.99
131.99
131.27
129.37
128.34
128.06
^{13}C{^{1}H}-NMR
DMSO-d_6
101 MHz
BF_3K
O
1n
DMSO-d6
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10


-139.85
^{19}F-NMR
DMSO-d$_6$
376 MHz
BF$_3$K
O
1n
BF$_4^-$
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210


2.84
^{11}B-NMR
DMSO-d$_6$
128 MHz
BF$_3$K
O
1n
BF$_4^-$
45 40 35 30 25 20 15 10 5 0 -5 -10 -15 -20 -25 -30 -35 -40 -45

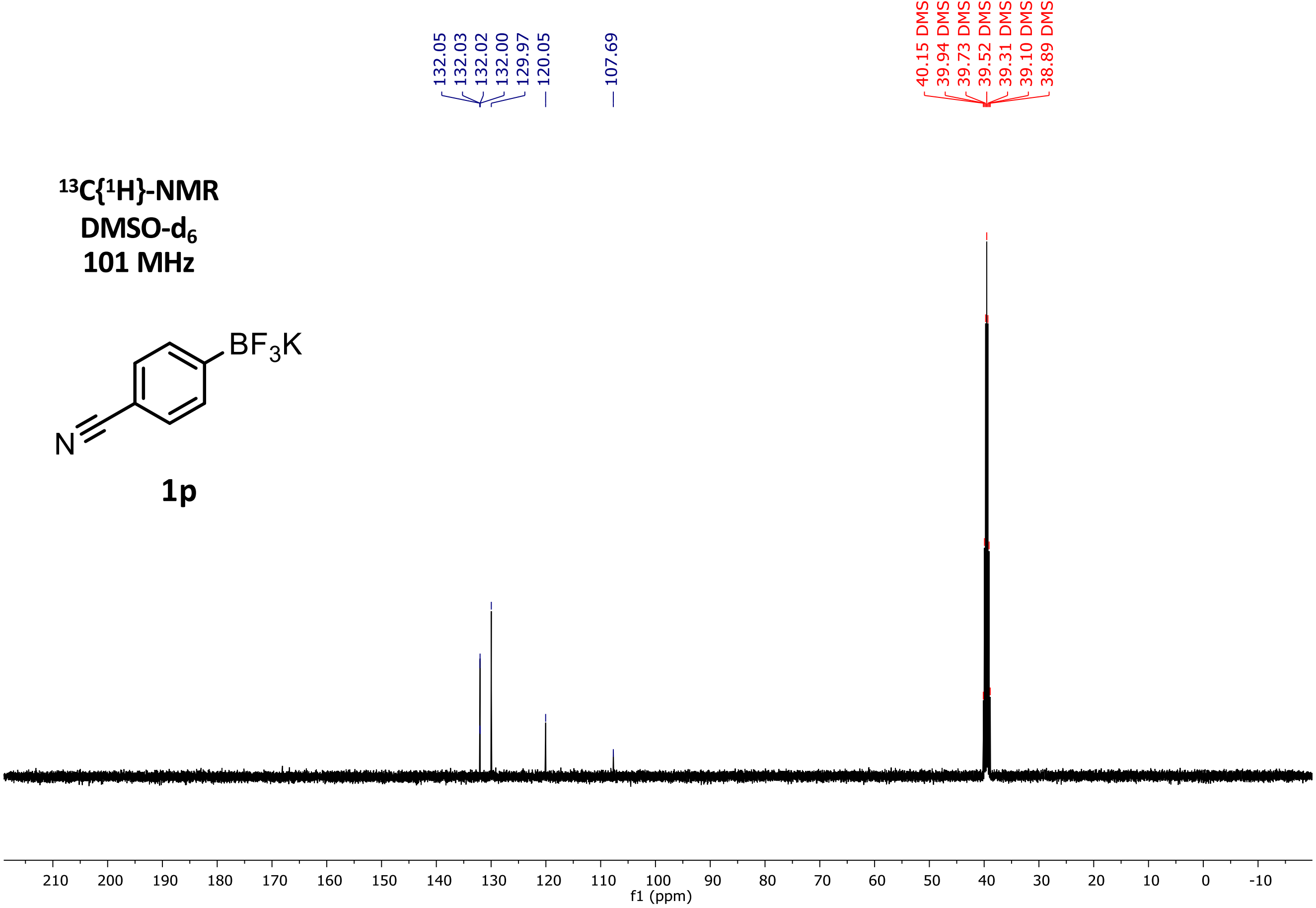

7.56
7.54
7.44
7.42
^{1}H-NMR
DMSO-d_6
400 MHz
BF_3K
F_3C
1o
H_2O
DMSO-d6
Acetone
1.97
2.00
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
-4


-60.48
-140.01
^{19}F-NMR
DMSO-d_6
376 MHz
BF_3K
F_3C
1o
3.02
3.00
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210


^{1}H-NMR
DMSO-d_6
400 MHz

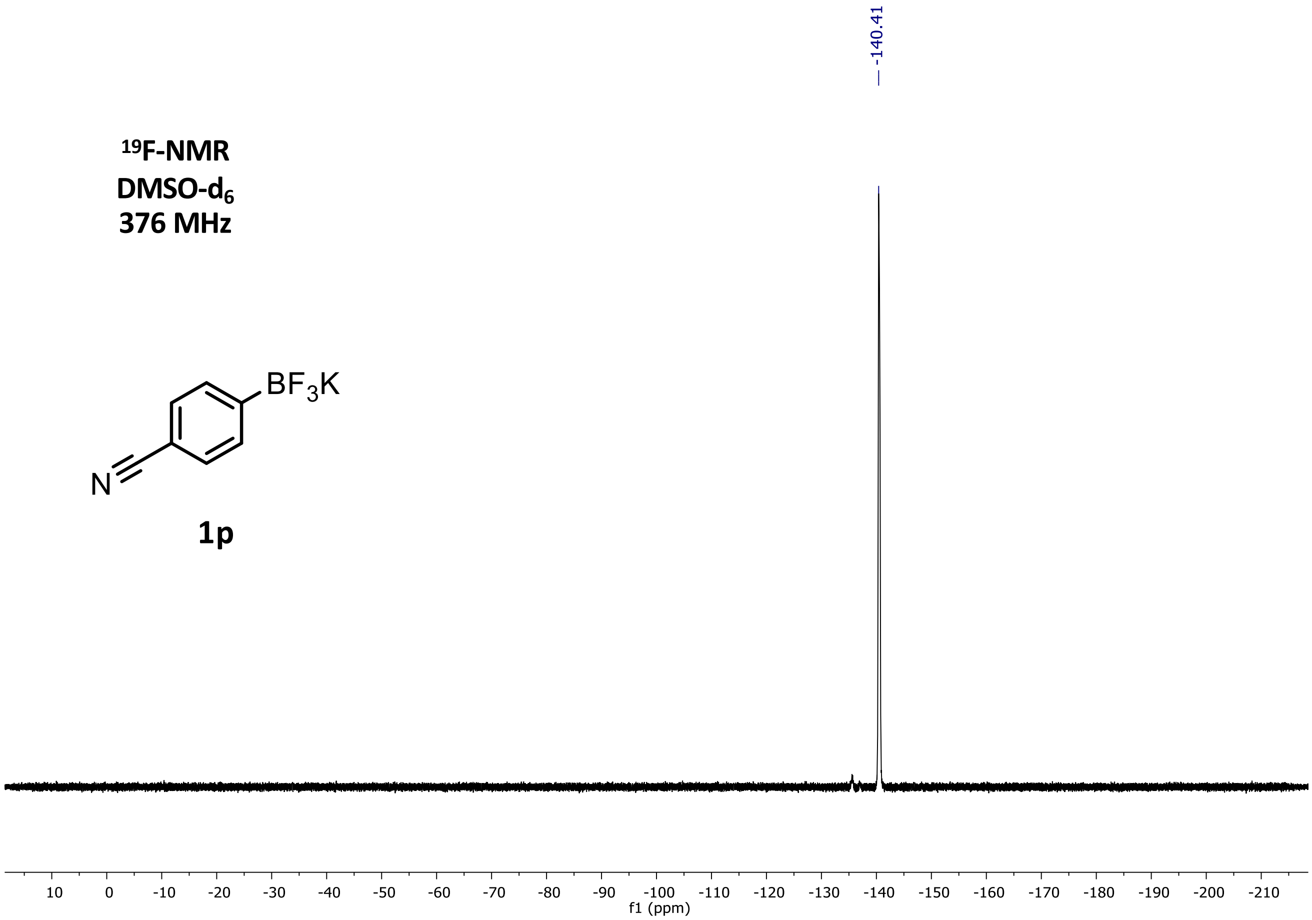
BF_3K

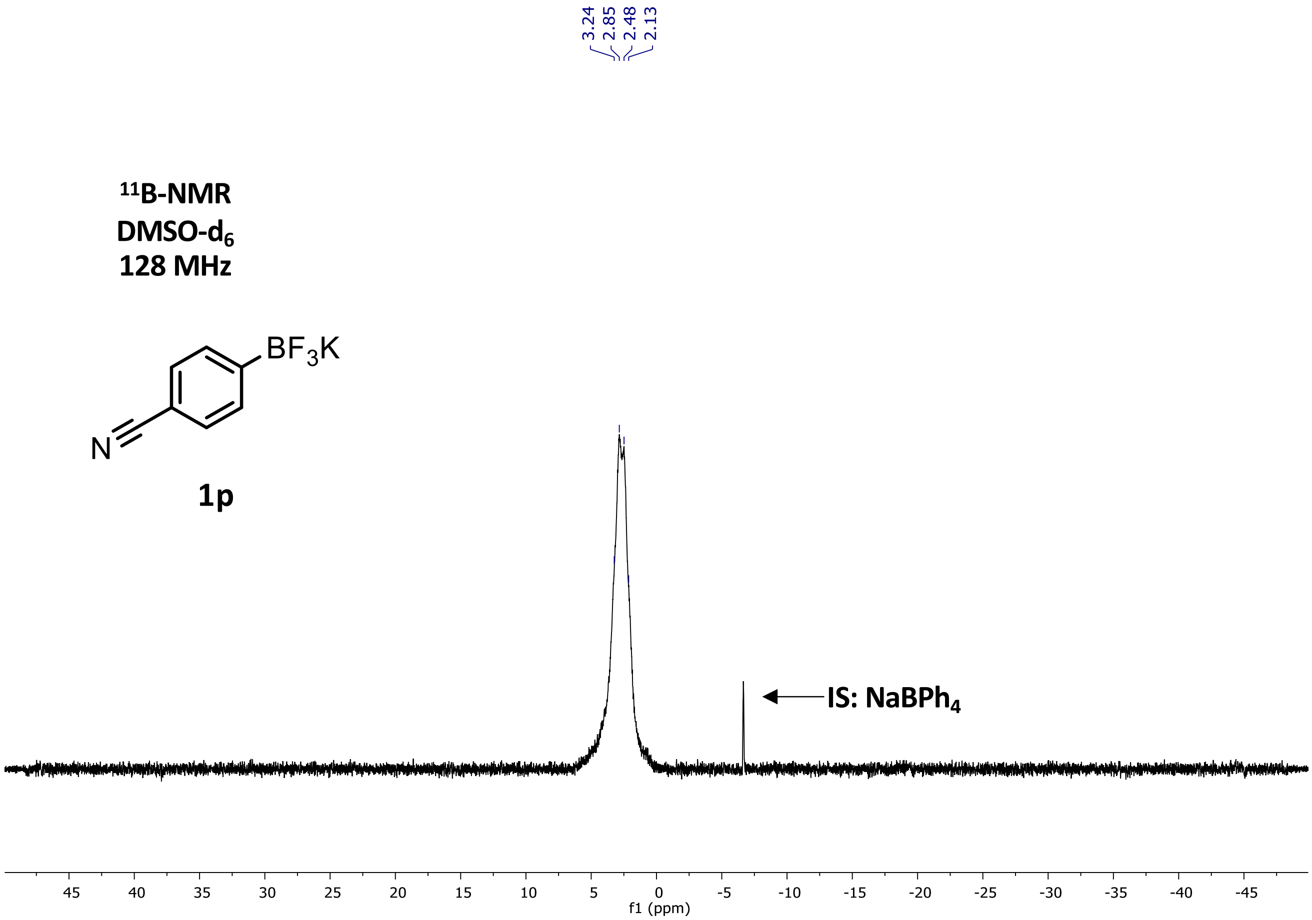
1p

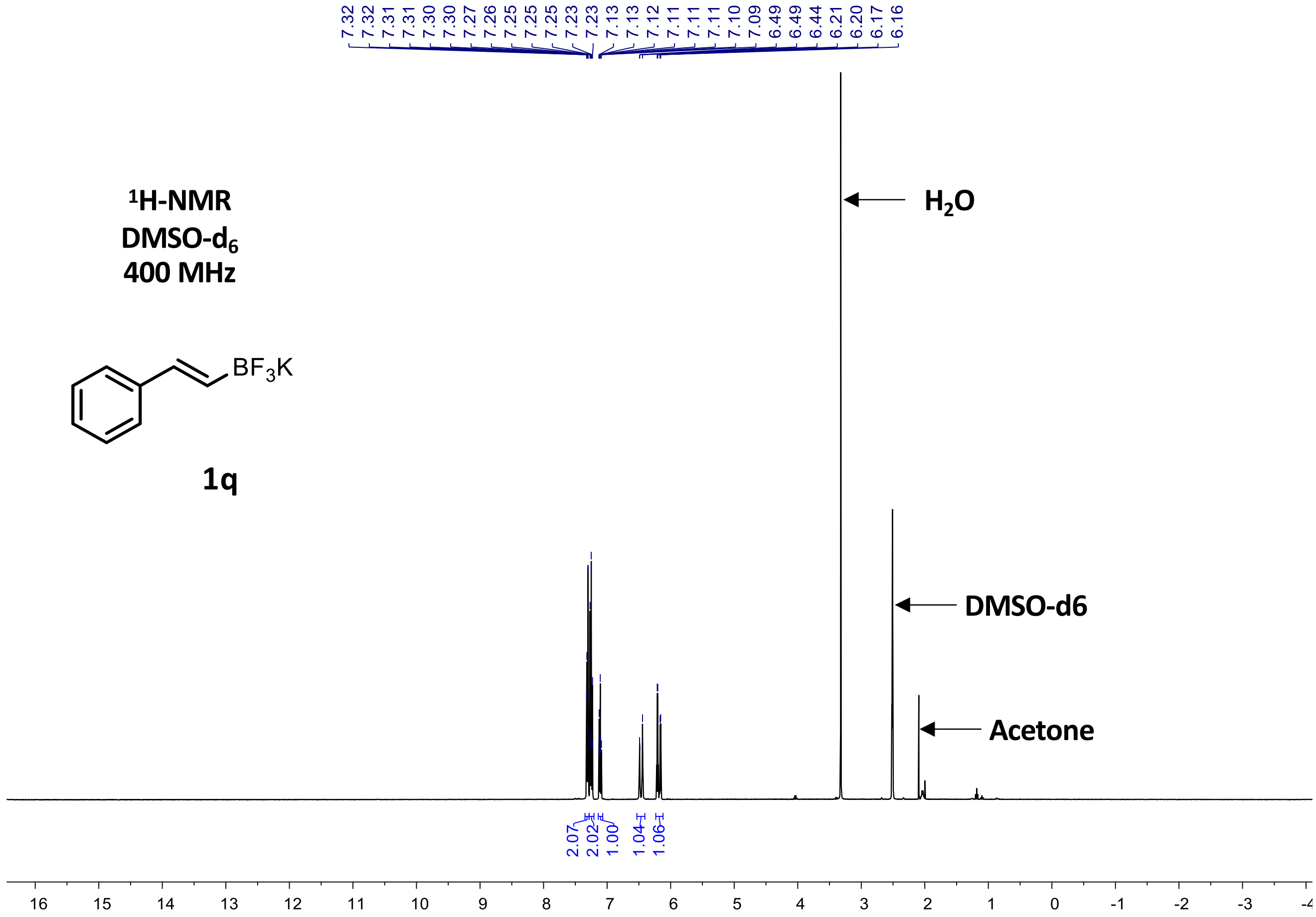

^{13}C{^{1}H}-NMR
DMSO-d_6
101 MHz

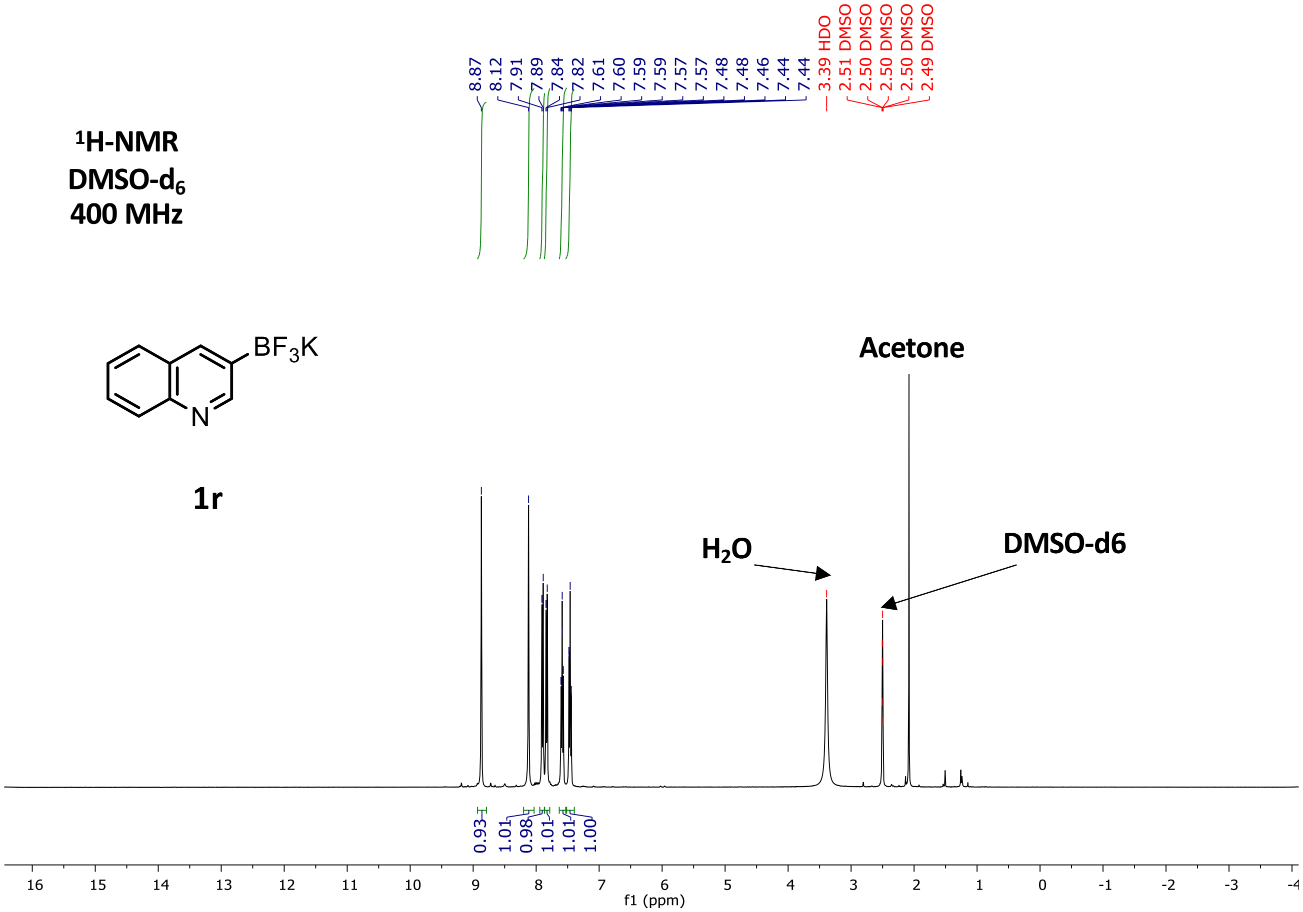
BF_3K

1p


^{19}F-NMR
DMSO-d_6
376 MHz
BF$_3$K
N
1p
-140.41
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

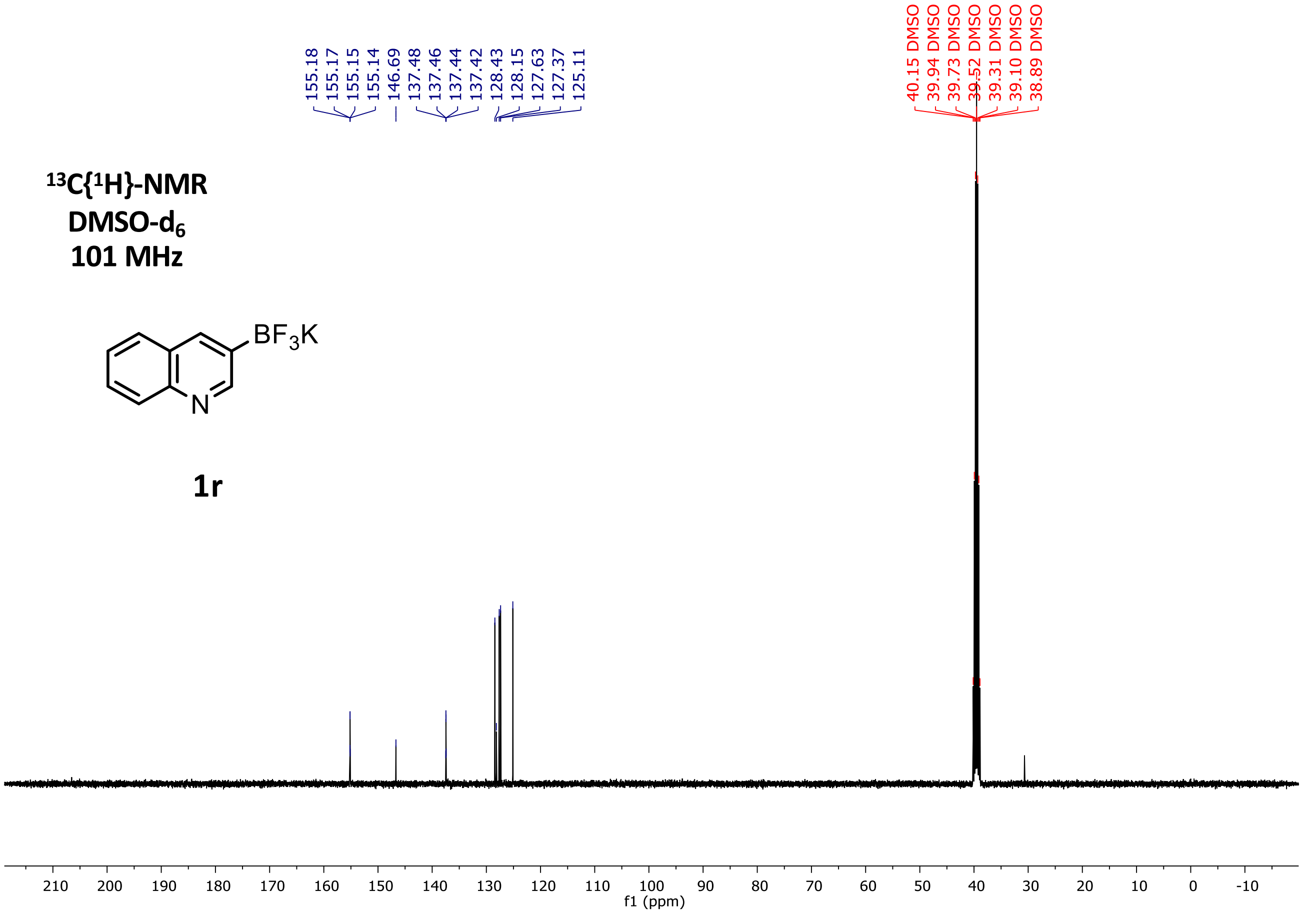

^{11}B-NMR
DMSO-d_6
128 MHz
BF$_3$K
N
1p
3.24
2.85
2.48
2.13
IS: NaBPh$_4$
45
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
-45
f1 (ppm)


7.32
7.32
7.31
7.31
7.30
7.30
7.27
7.26
7.25
7.25
7.25
7.23
7.23
7.13
7.13
7.12
7.11
7.11
7.11
7.10
7.09
6.49
6.49
6.44
6.21
6.20
6.17
6.16
^{1}H-NMR
DMSO-d_6
400 MHz
BF_3K
1q
H_2O
DMSO-d6
Acetone
2.07
2.02
1.00
1.04
1.06
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
-4



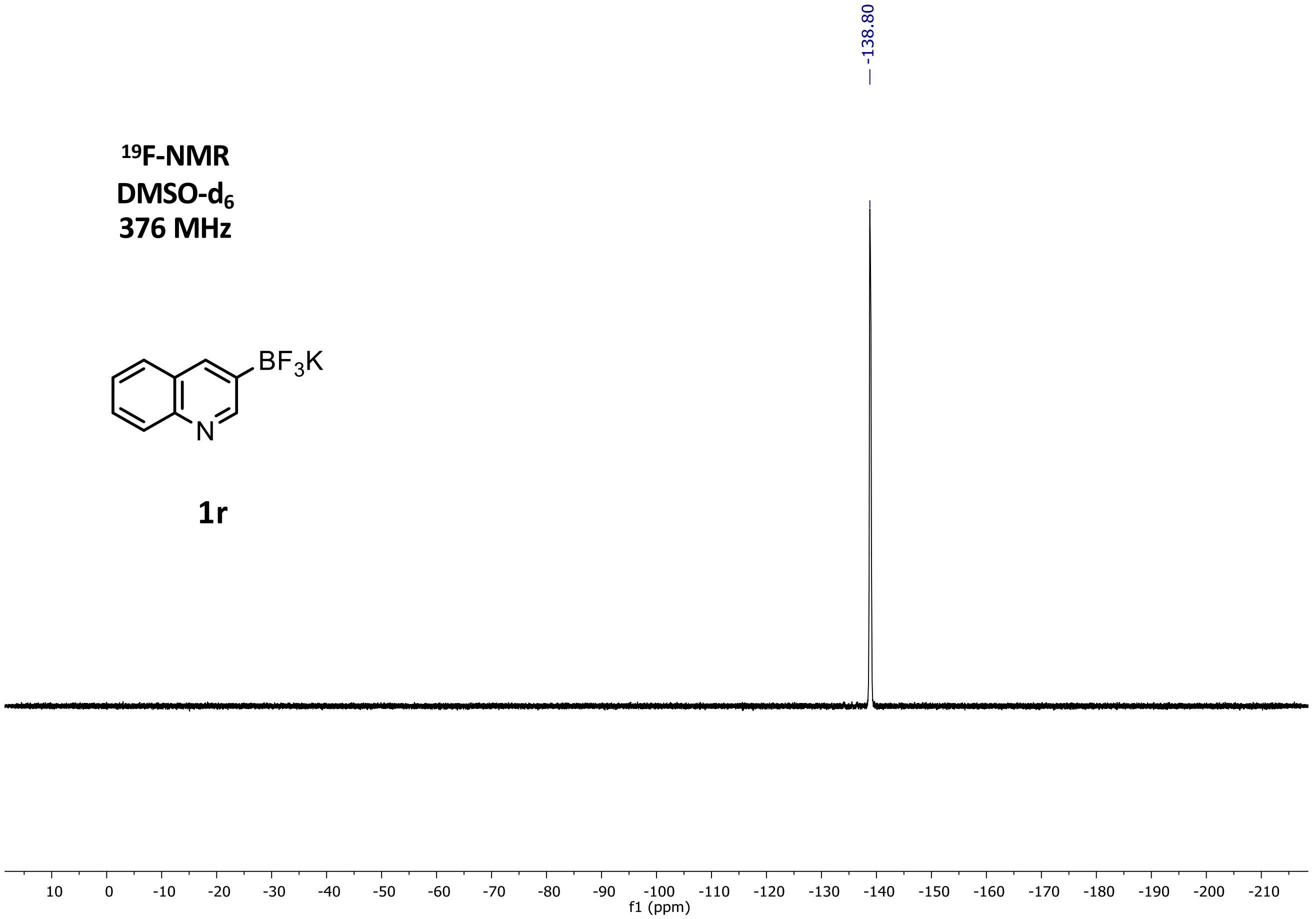
^{1}H-NMR
DMSO-d_6
400 MHz

1r



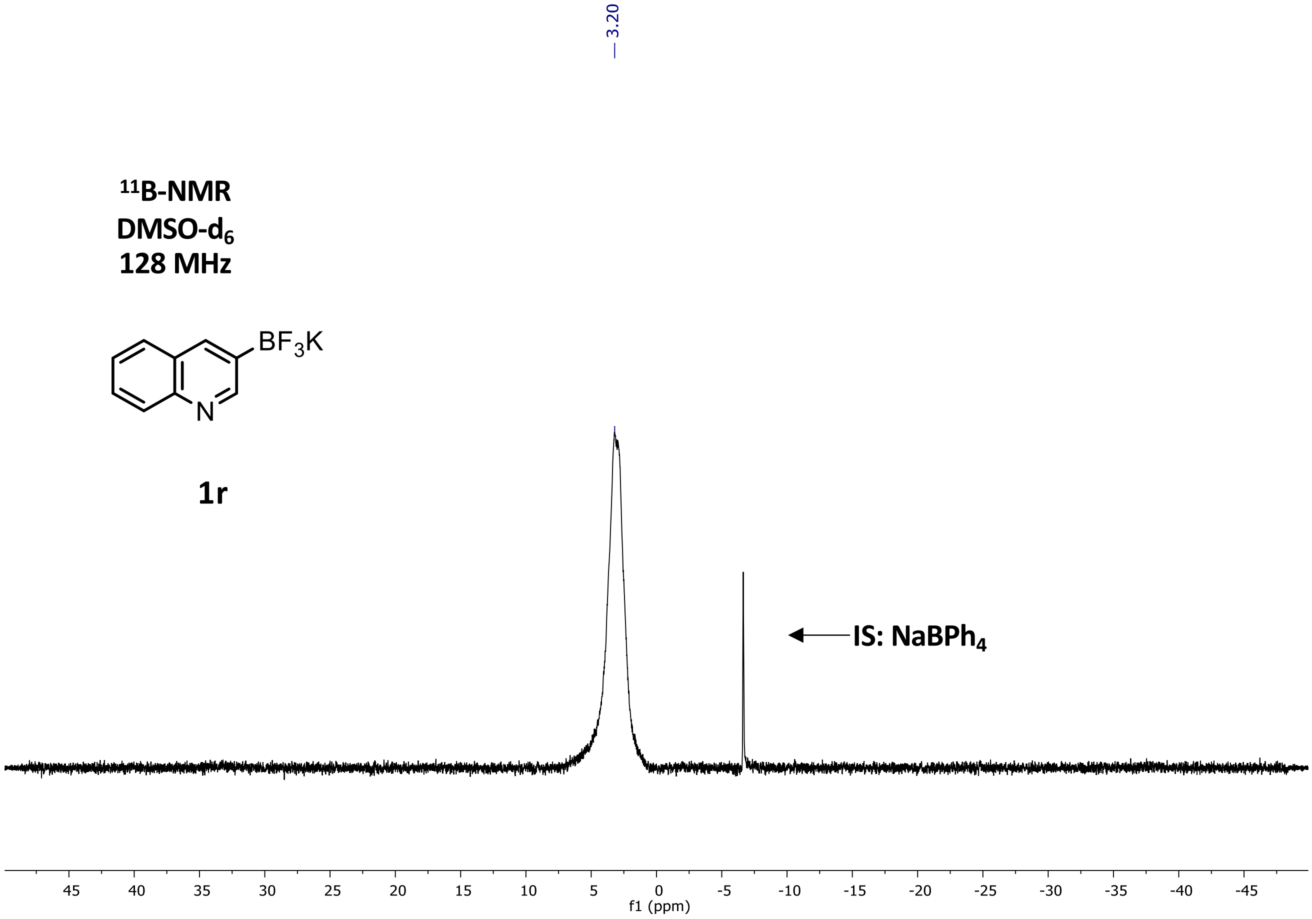
^{13}C{^{1}H}-NMR
DMSO-d_6
101 MHz

1r



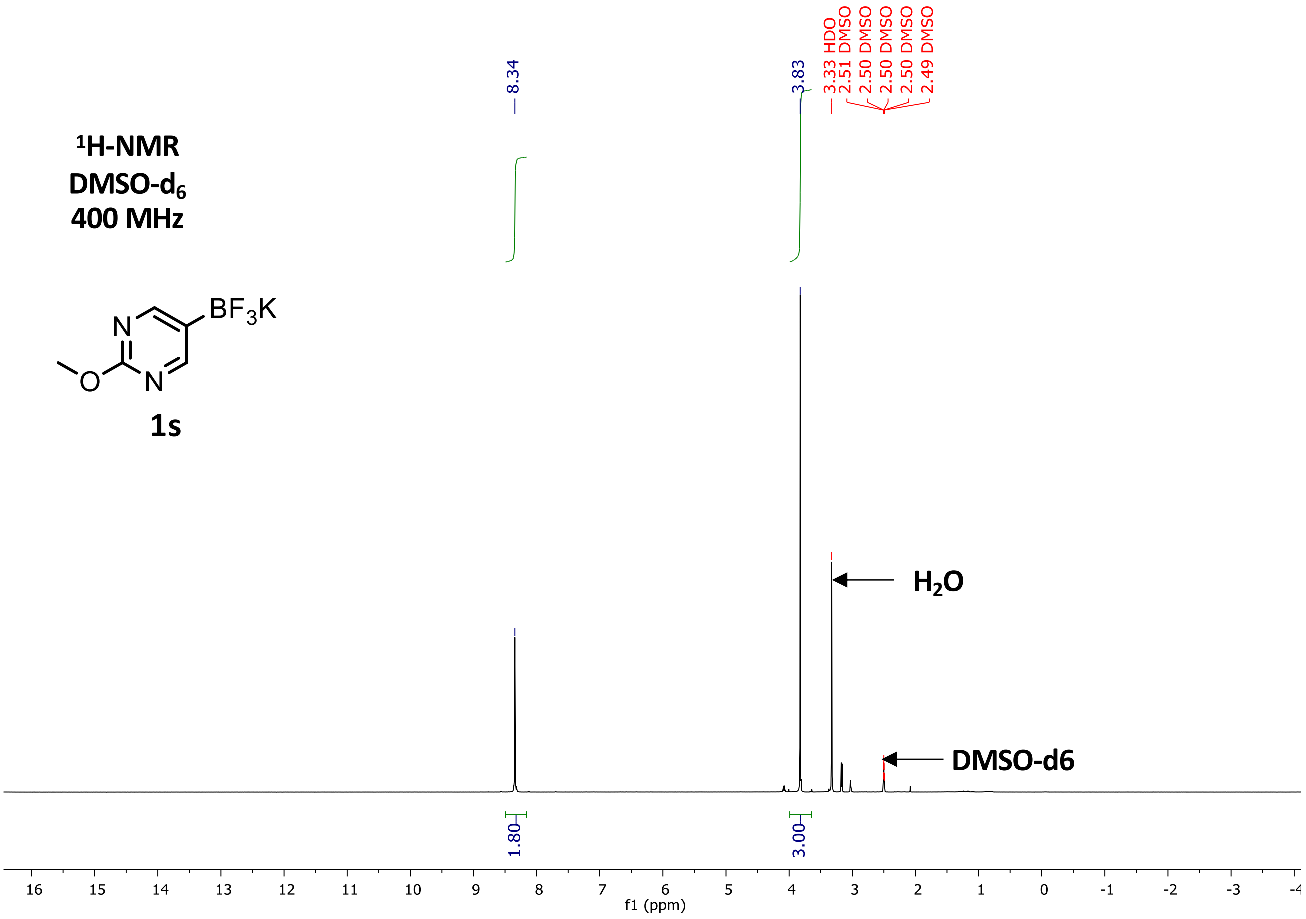
^{19}F-NMR
DMSO-d_6
376 MHz

1r

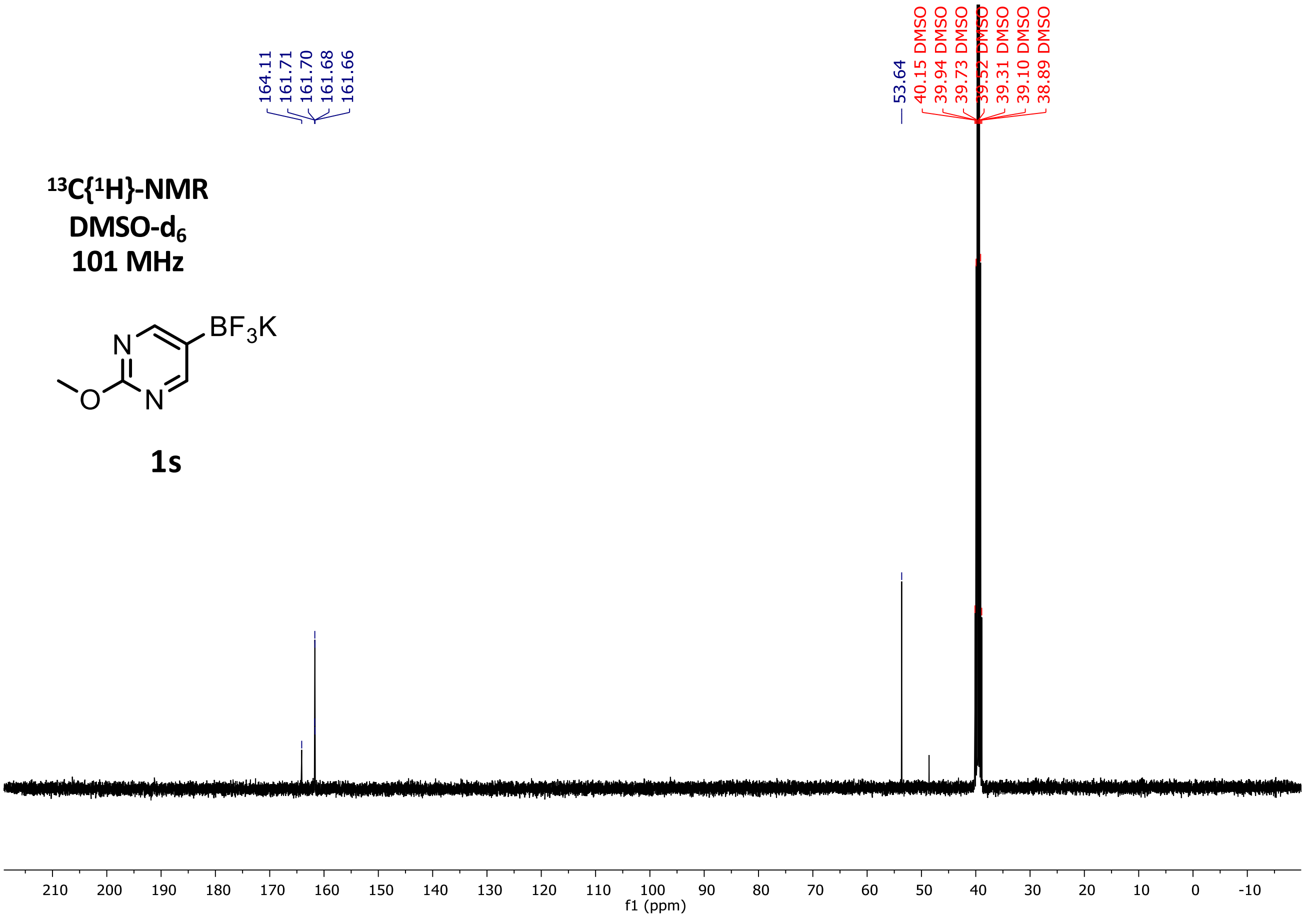


^{11}B-NMR
DMSO-d_6
128 MHz

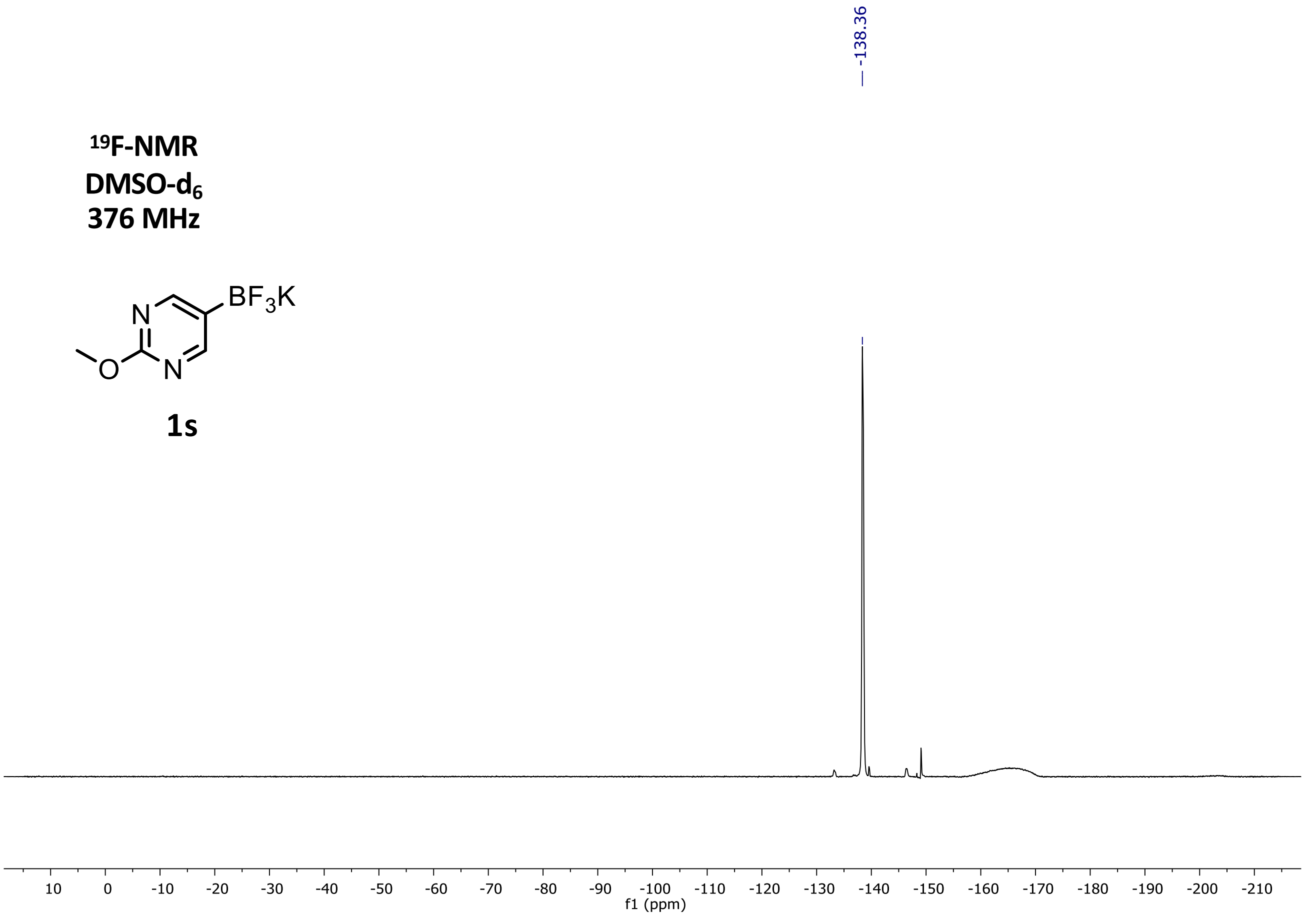
1r



^{1}H-NMR
DMSO-d$_6$
400 MHz
BF$_3$K
N
N
O
1s
8.34
3.83
3.33 HDO
2.51 DMSO
2.50 DMSO
2.50 DMSO
2.50 DMSO
2.49 DMSO
H$_2$O
DMSO-d6
1.80
3.00
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)

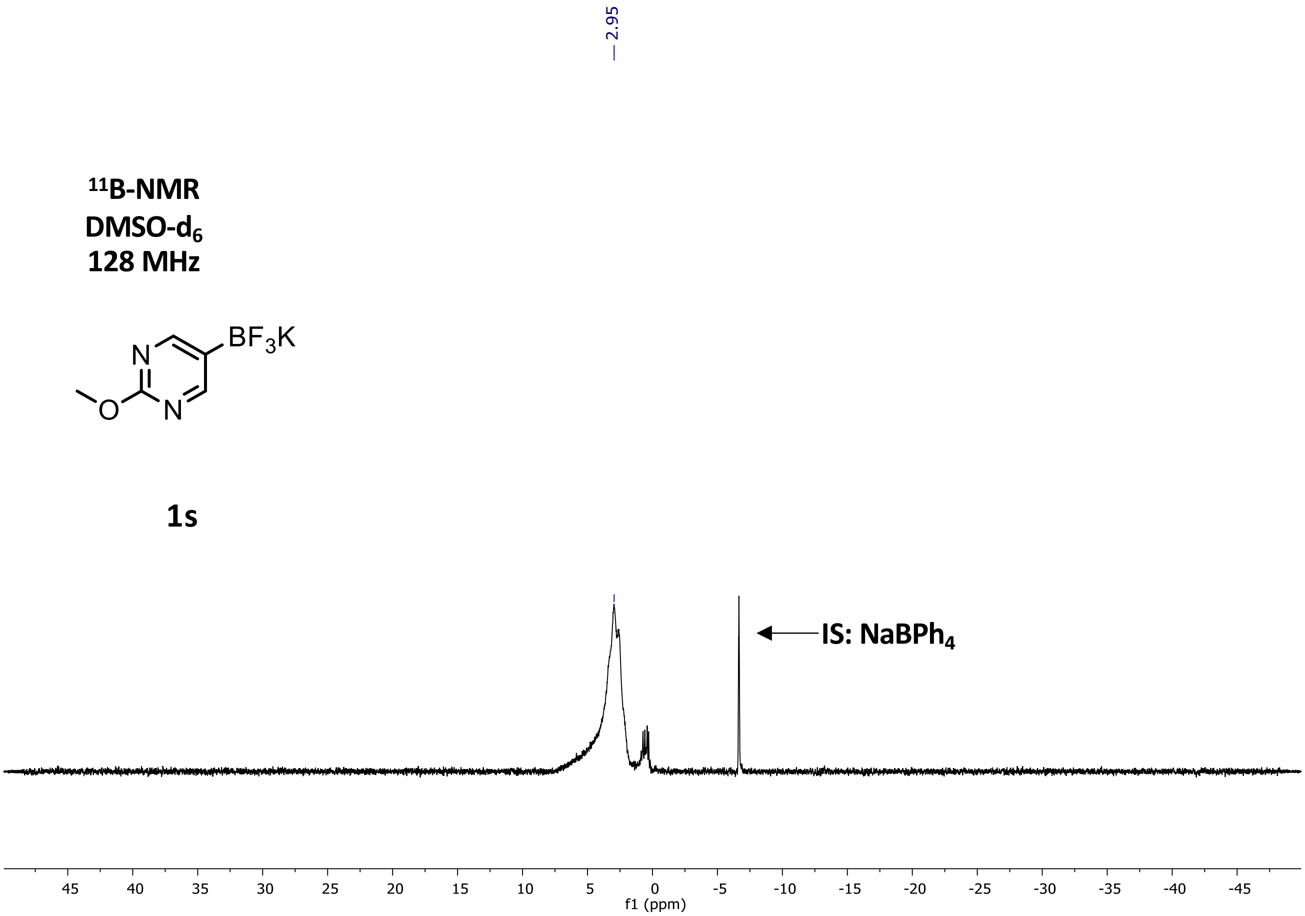


^{13}C{^{1}H}-NMR
DMSO-d$_6$
101 MHz
BF$_3$K
N
N
O
1s
164.11
161.71
161.70
161.68
161.66
53.64
40.15 DMSO
39.94 DMSO
39.73 DMSO
39.52 DMSO
39.31 DMSO
39.10 DMSO
38.89 DMSO
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



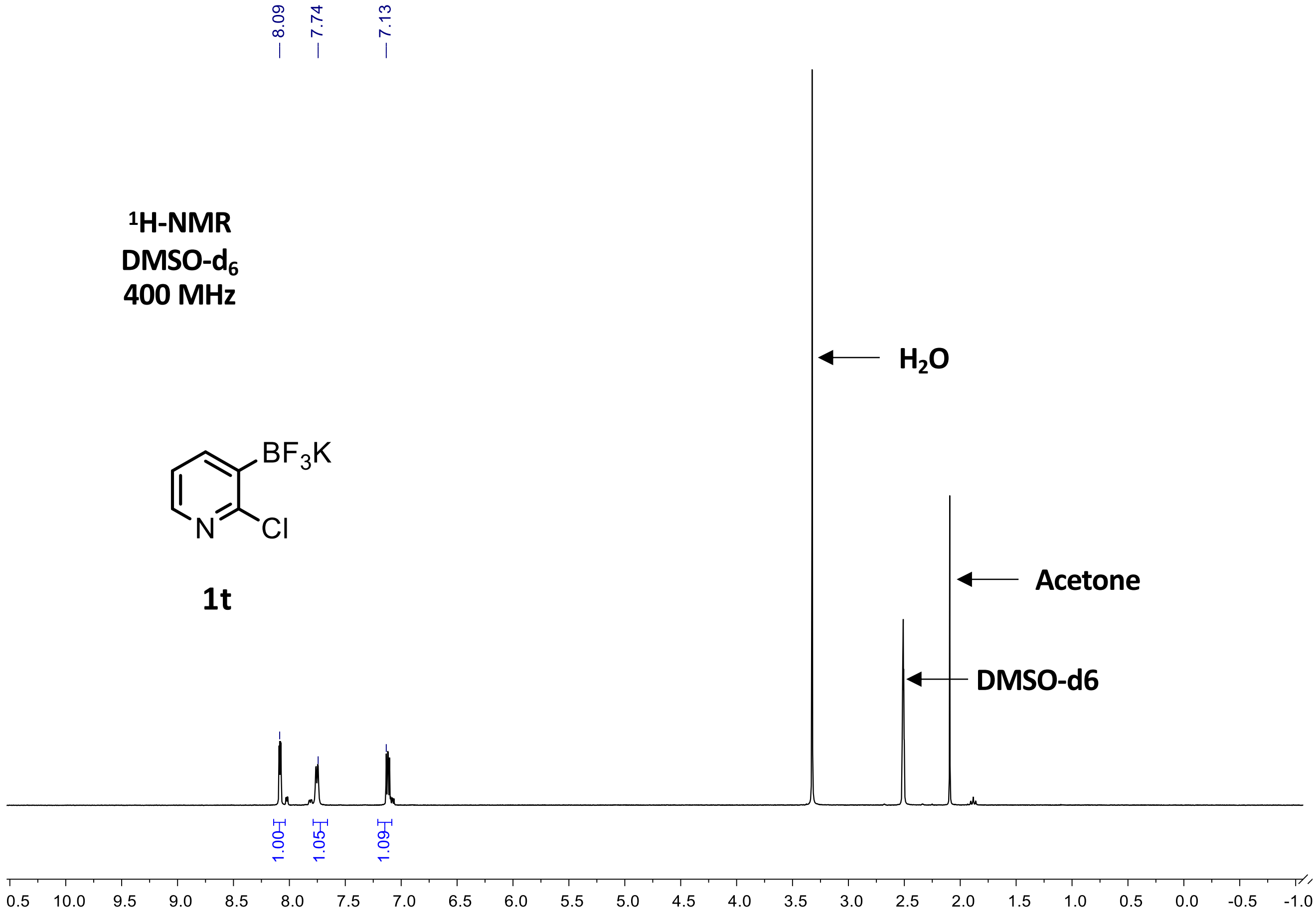
^{19}F-NMR
DMSO-d_6
376 MHz

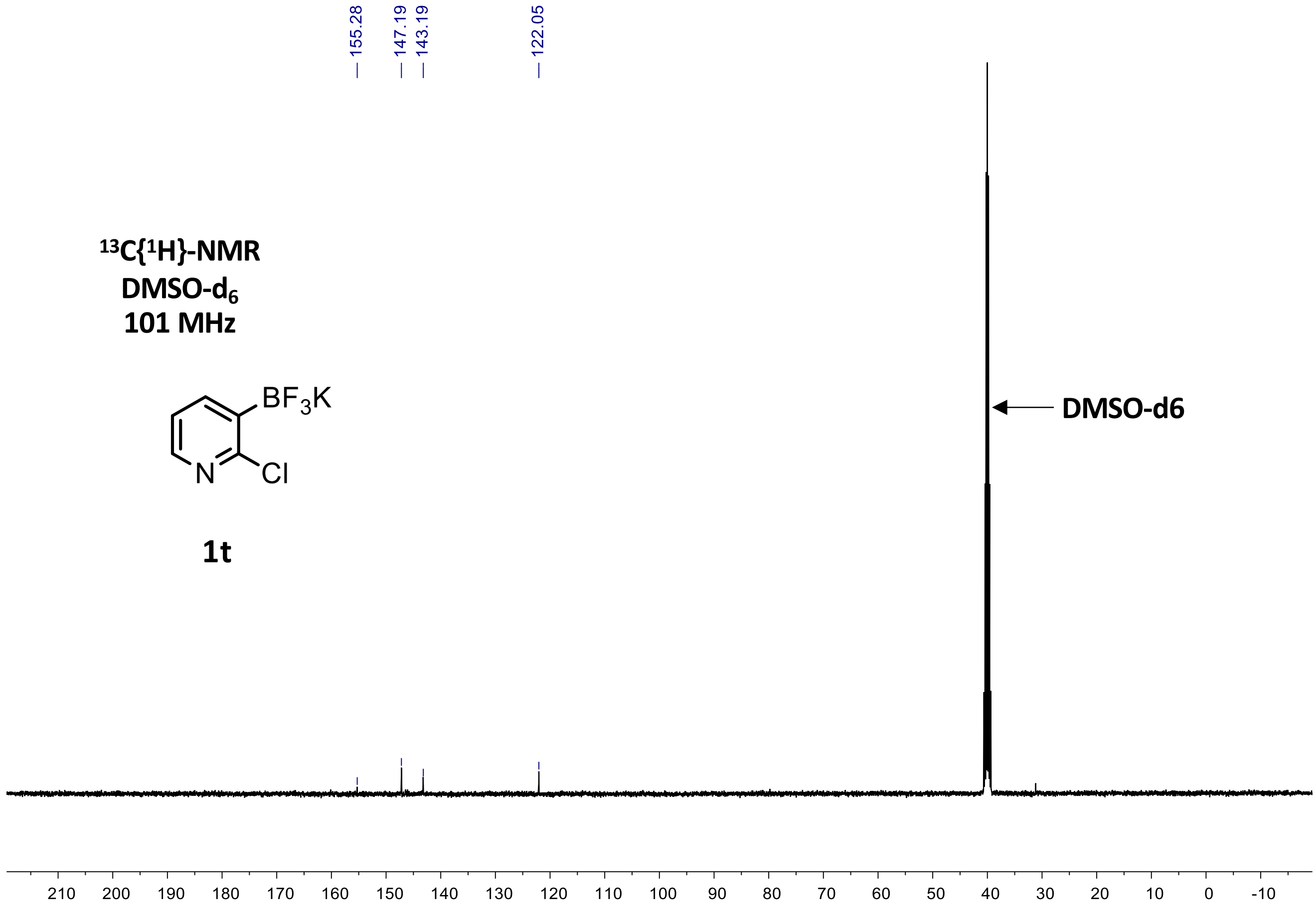
1s

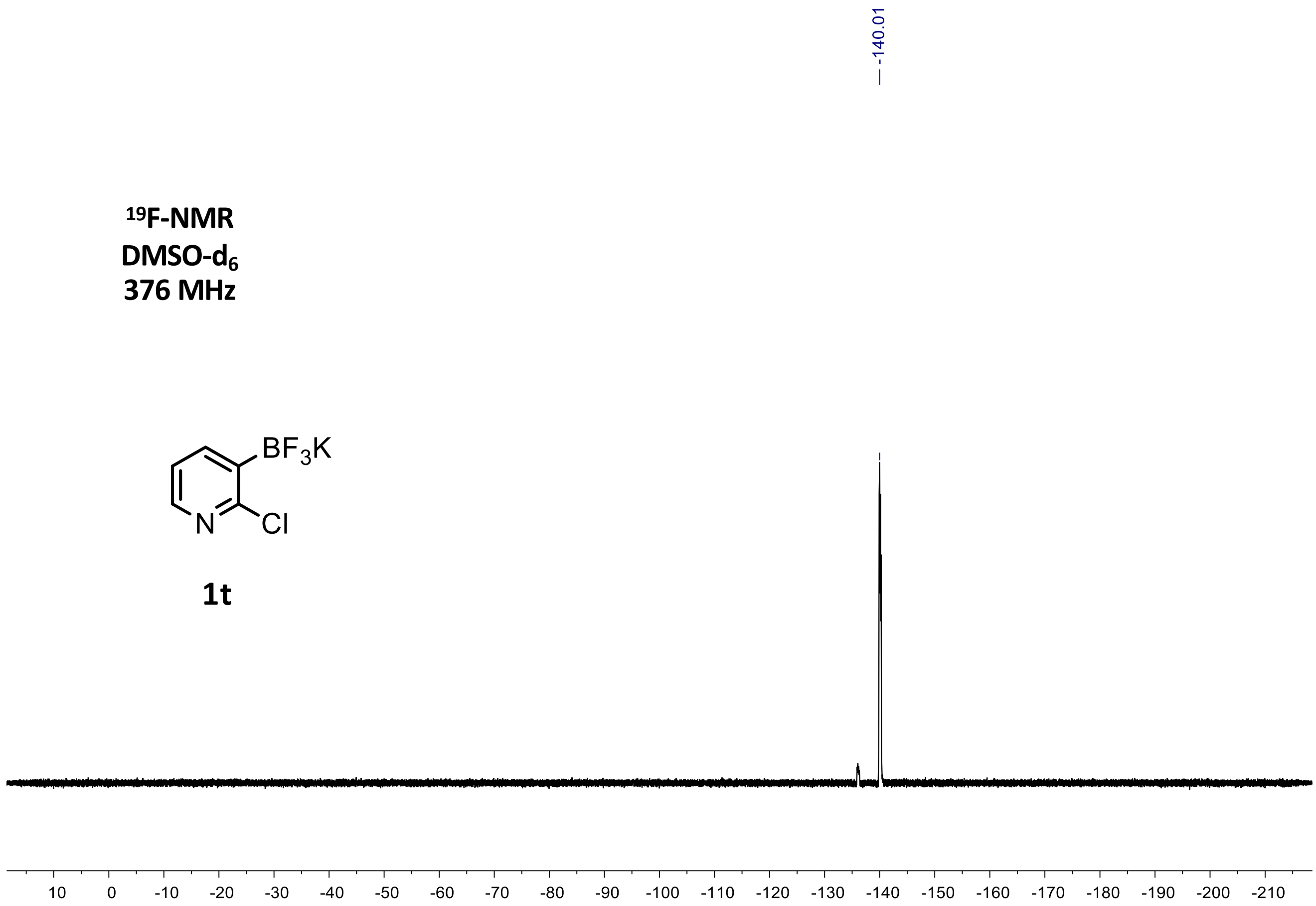


^{11}B-NMR
DMSO-d_6
128 MHz

1s

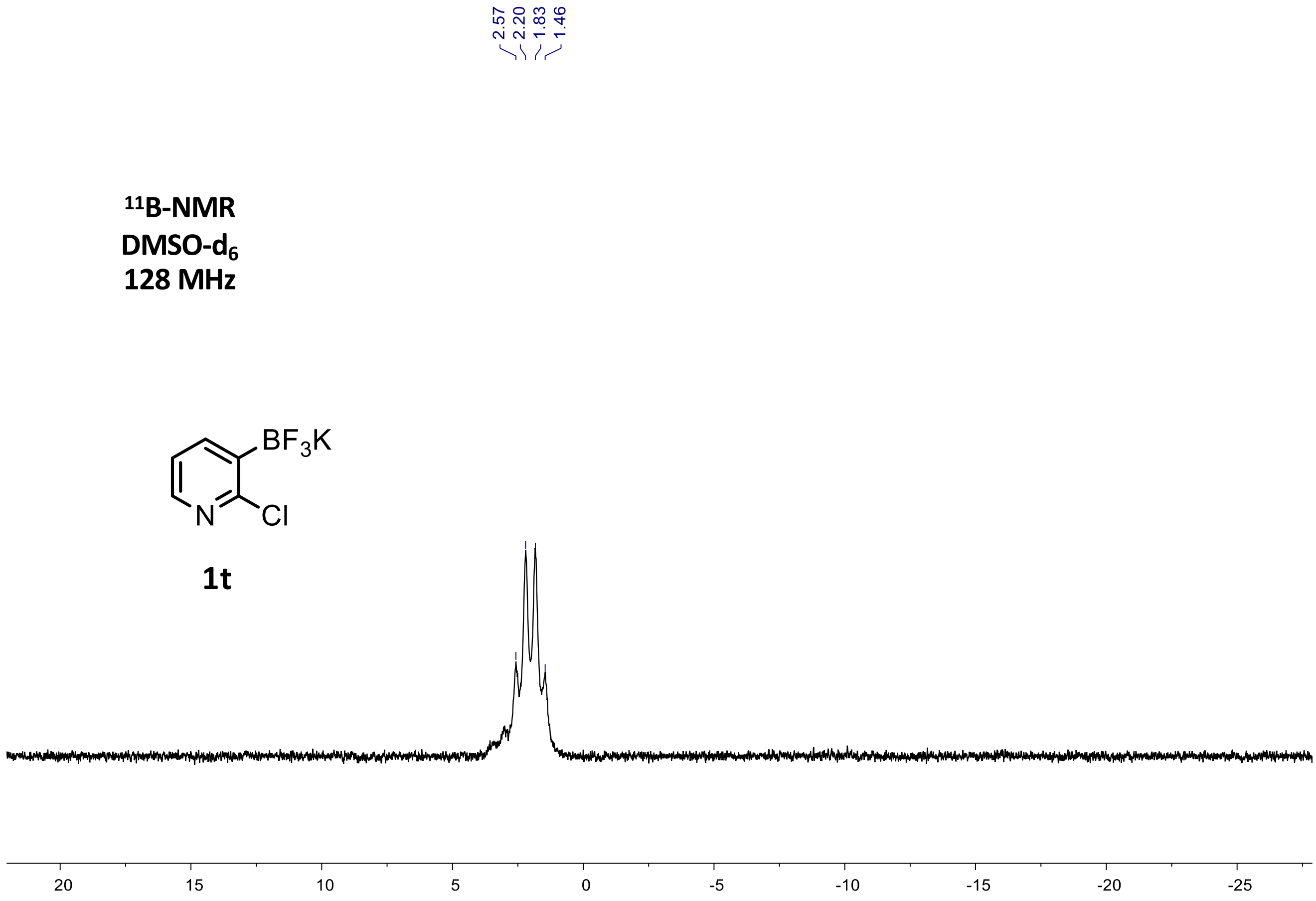

8.09
7.74
7.13
^{1}H-NMR
DMSO-d_6
400 MHz
BF$_3$K
N
Cl
1t
H$_2$O
Acetone
DMSO-d6
1.00
1.05
1.09
0.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0


155.28
147.19
143.19
122.05
^{13}C{^{1}H}-NMR
DMSO-d_6
101 MHz
BF$_3$K
N
Cl
1t
DMSO-d6
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10



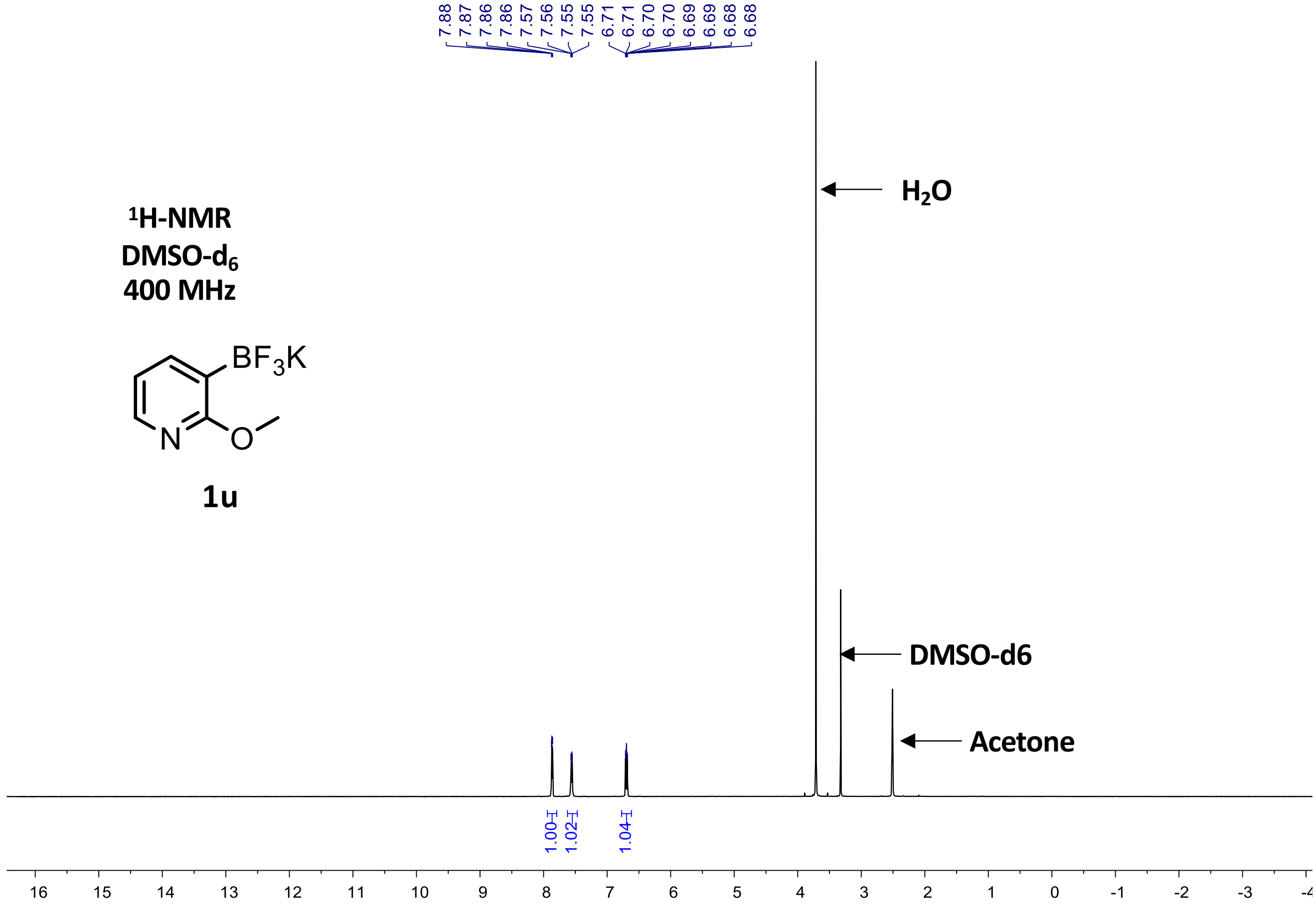
^{19}F-NMR
DMSO-d_6
376 MHz

1t



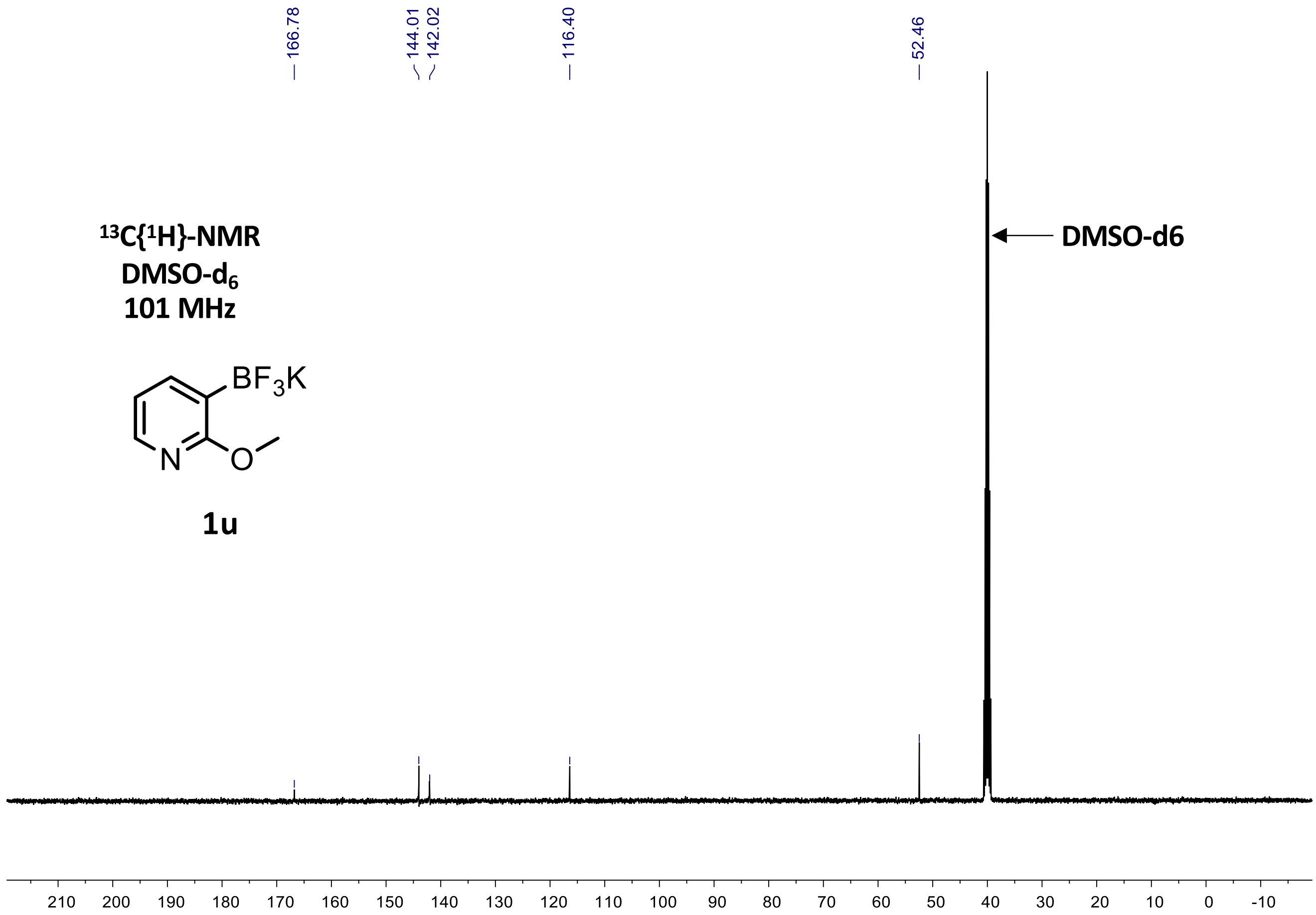
^{11}B-NMR
DMSO-d_6
128 MHz

1t



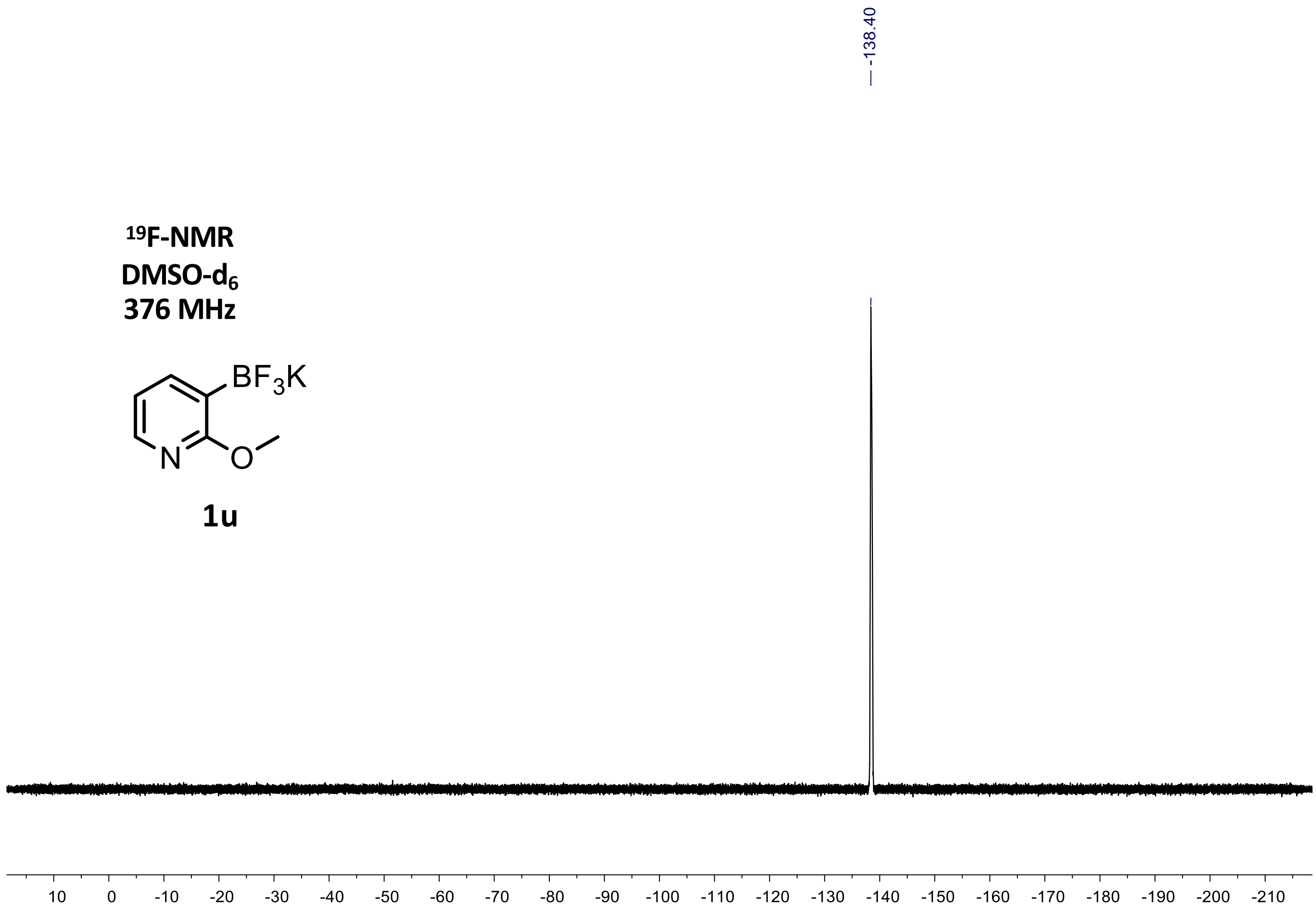
^{1}H-NMR
DMSO-d_6
400 MHz

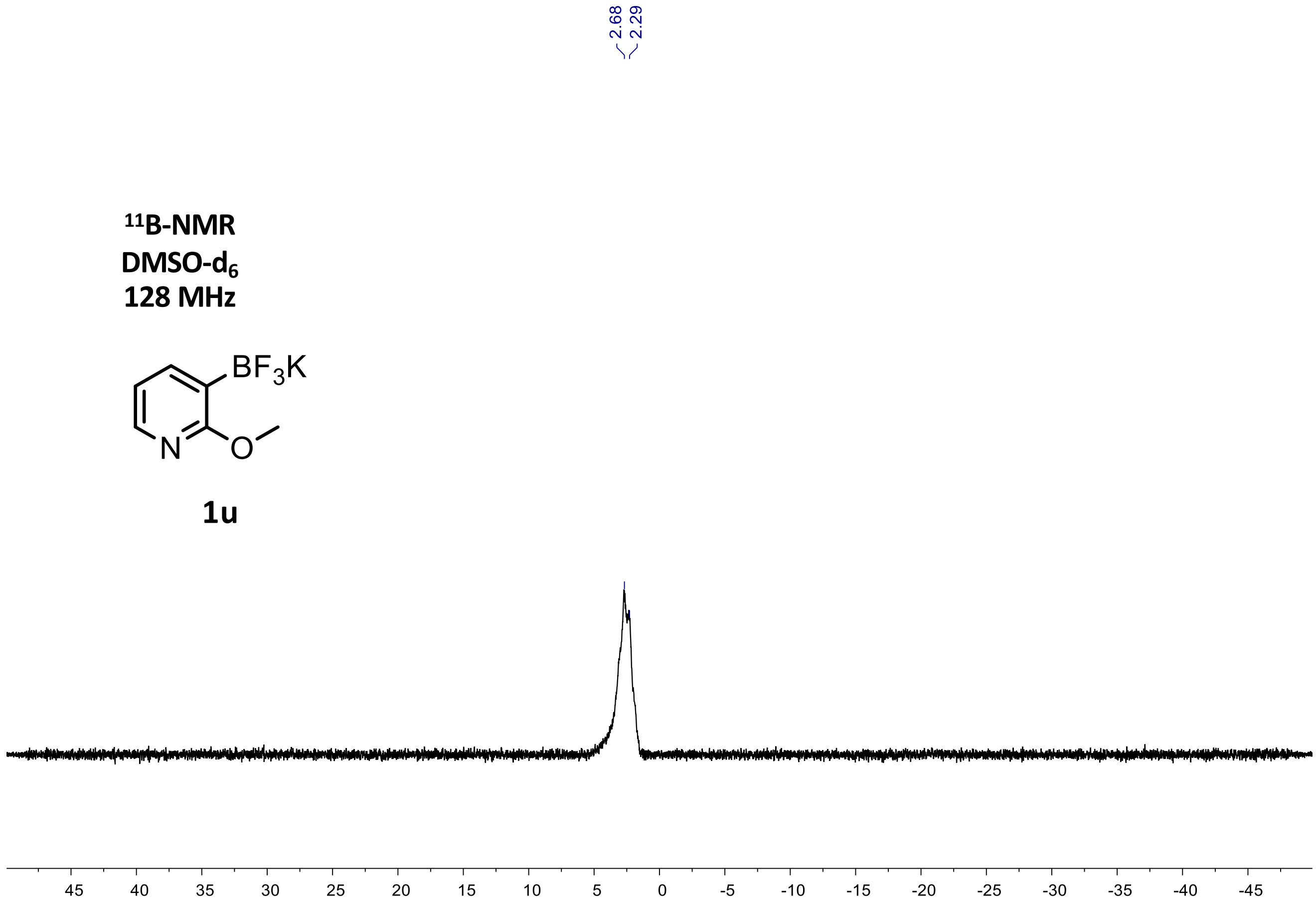
1u

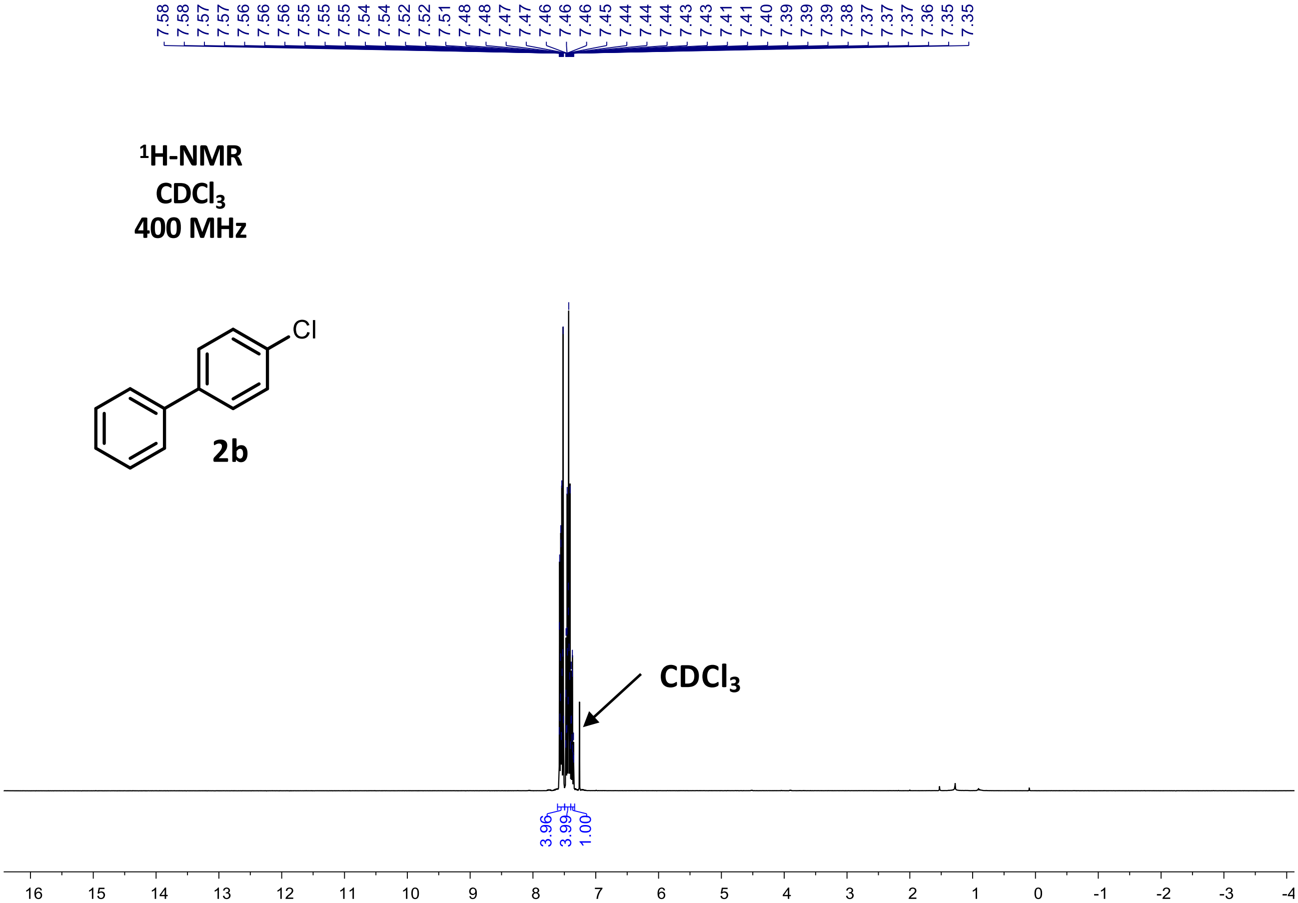


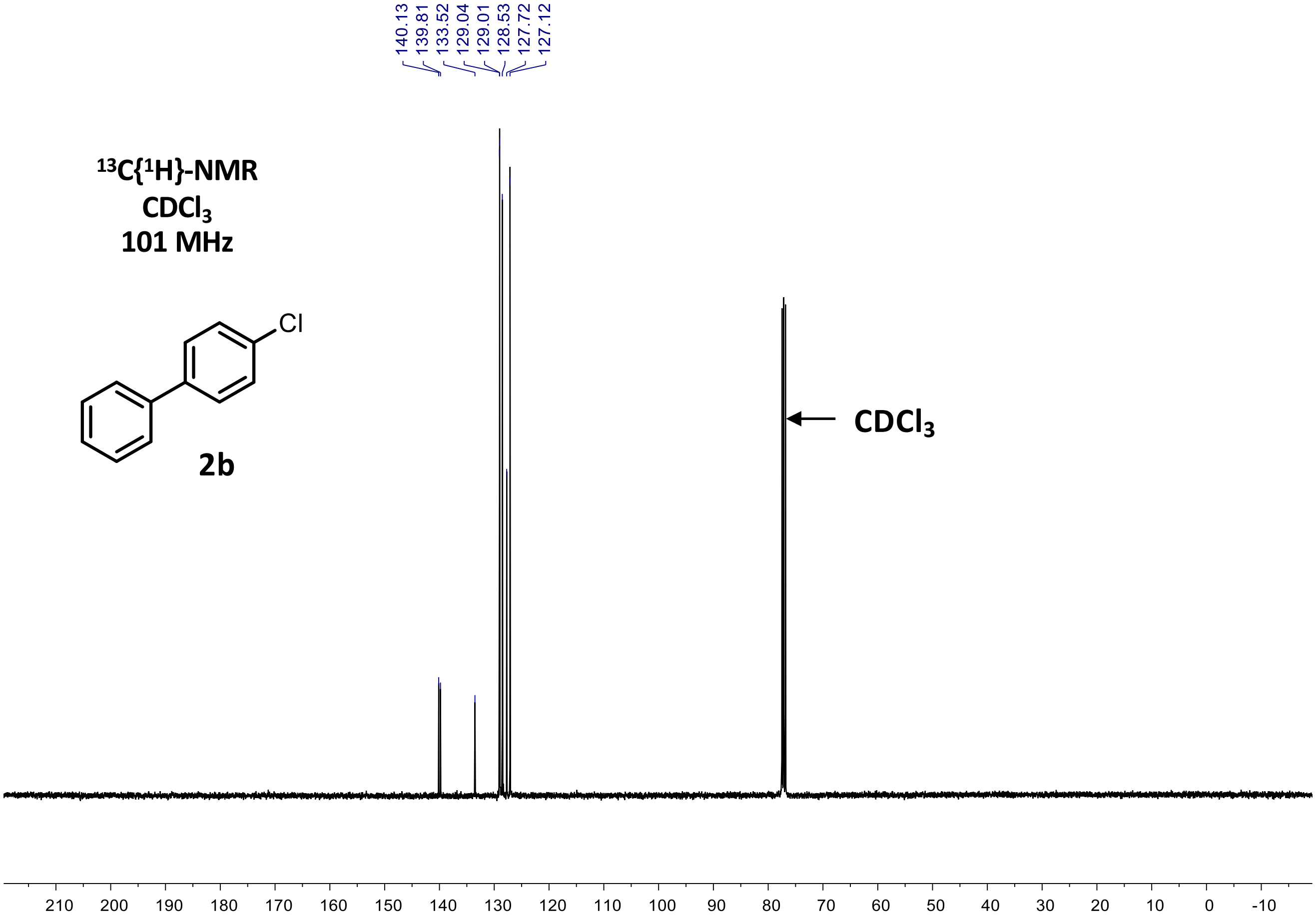
^{13}C{^{1}H}-NMR
DMSO-d_6
101 MHz

1u

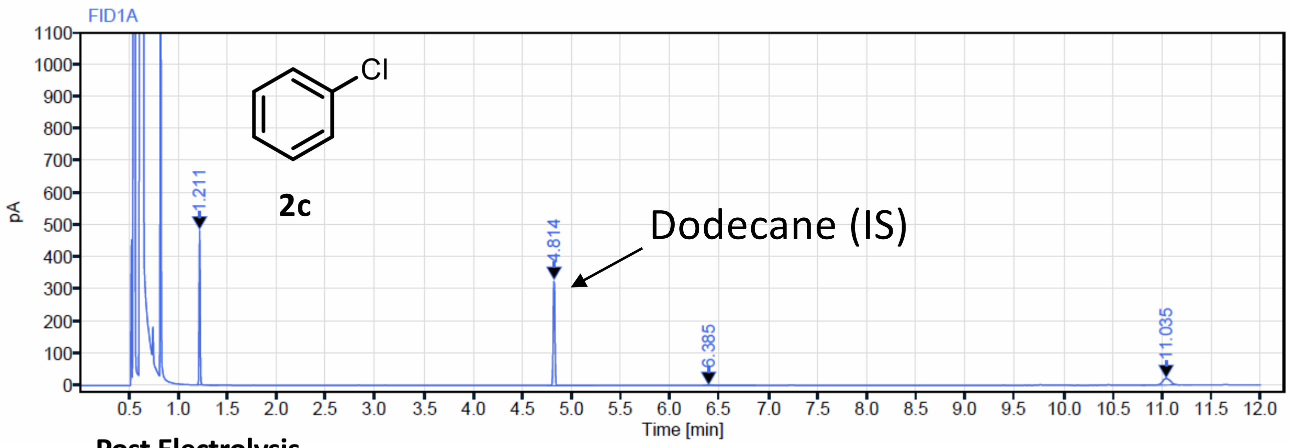

-138.40
^{19}F-NMR
DMSO-d$_6$
376 MHz
BF$_3$K
N
O
1u
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210


2.68
2.29
^{11}B-NMR
DMSO-d$_6$
128 MHz
BF$_3$K
N
O
1u
45 40 35 30 25 20 15 10 5 0 -5 -10 -15 -20 -25 -30 -35 -40 -45


7.58
7.58
7.57
7.57
7.56
7.56
7.55
7.55
7.54
7.54
7.52
7.52
7.51
7.48
7.48
7.47
7.47
7.46
7.46
7.45
7.44
7.44
7.43
7.43
7.41
7.41
7.40
7.39
7.39
7.38
7.37
7.37
7.36
7.35
7.35
^{1}H-NMR
$CDCl_3$
400 MHz
Cl
2b
$CDCl_3$
3.96
3.99
1.00
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
-4


140.13
139.81
133.52
129.04
129.01
128.53
127.72
127.12
$^{13}C\{^1H\}$-NMR
$CDCl_3$
101 MHz
Cl
2b
$CDCl_3$
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10

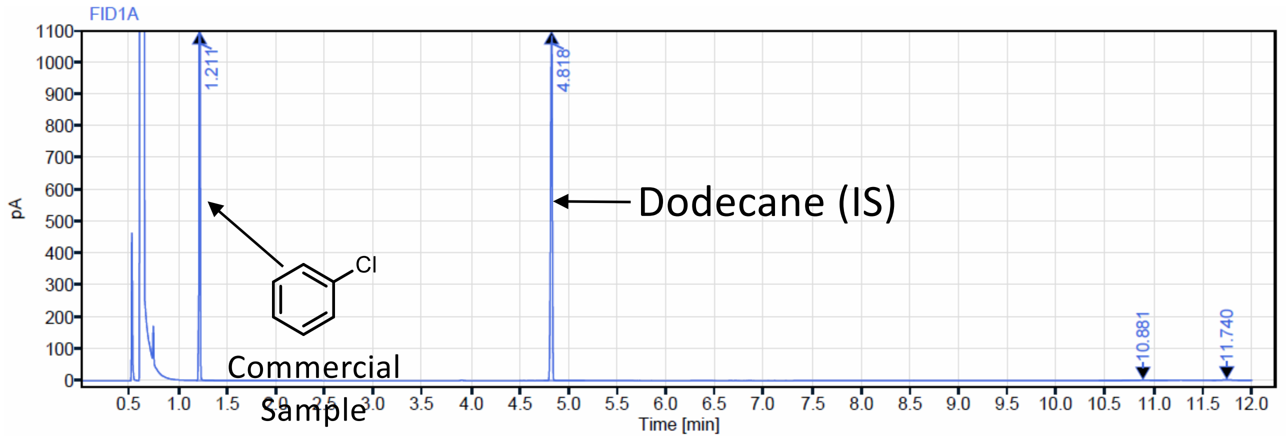
Gas Chromatogram



Post Electrolysis

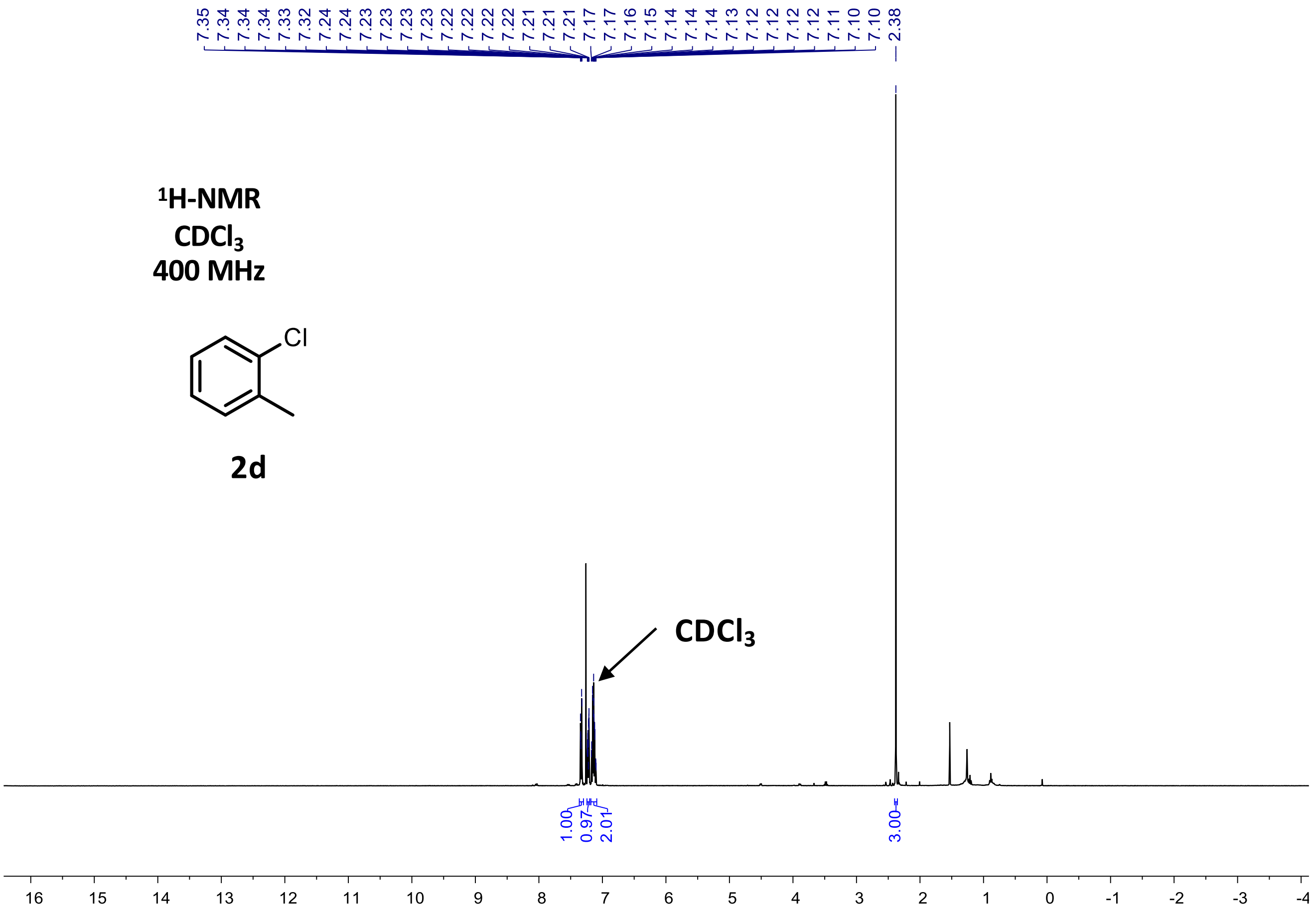
Signal: FID1A

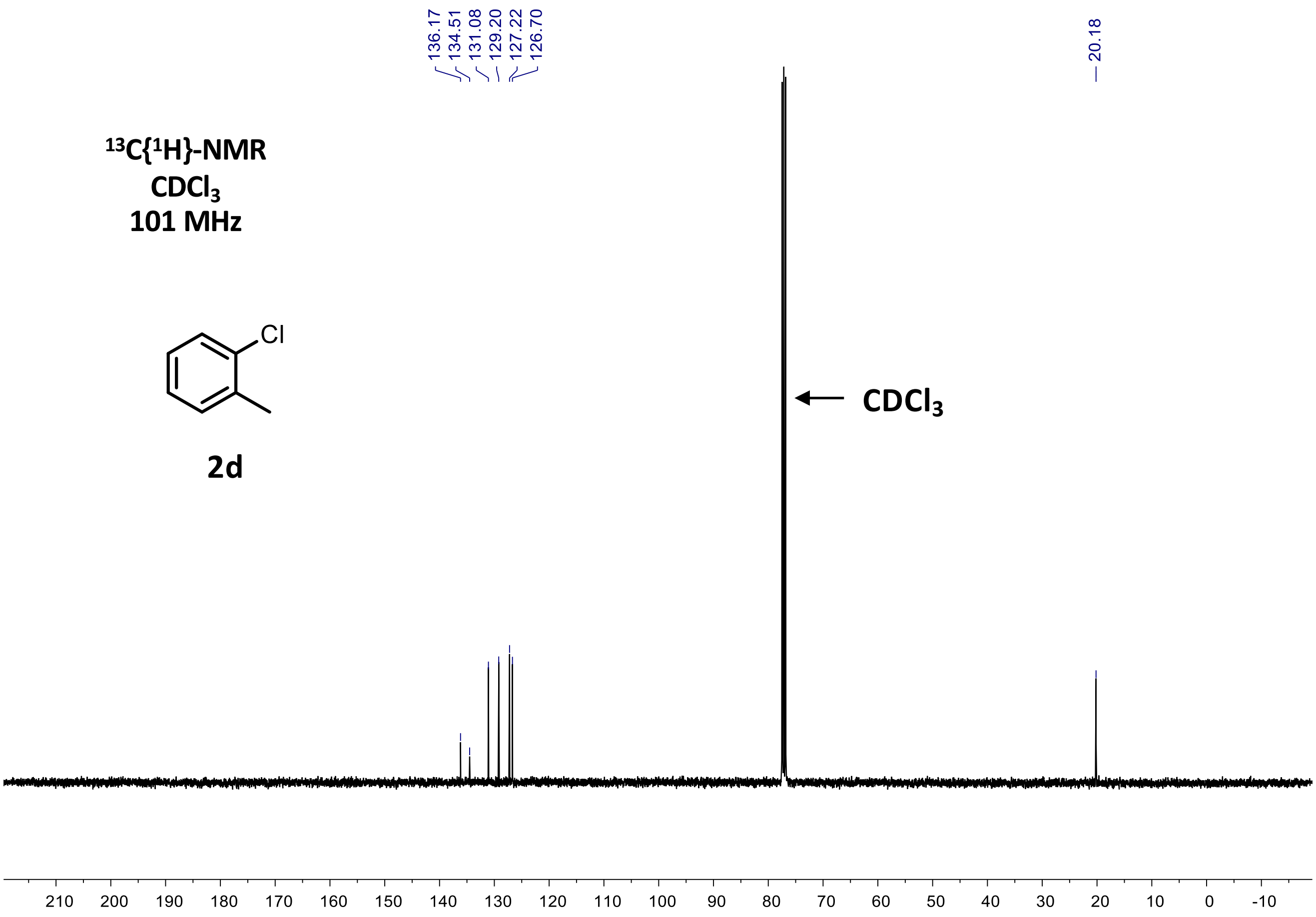
RT [min]	Type	Width [min]	Area	Height	Area%	Name
1.211	BB	0.21	382.30	483.81	44.40	
4.814	BV R	0.46	343.12	328.35	39.85	
6.385	BV	0.53	13.25	0.83	1.54	
11.035	VV R	0.51	122.28	20.84	14.20	
		Sum	**860.95**			

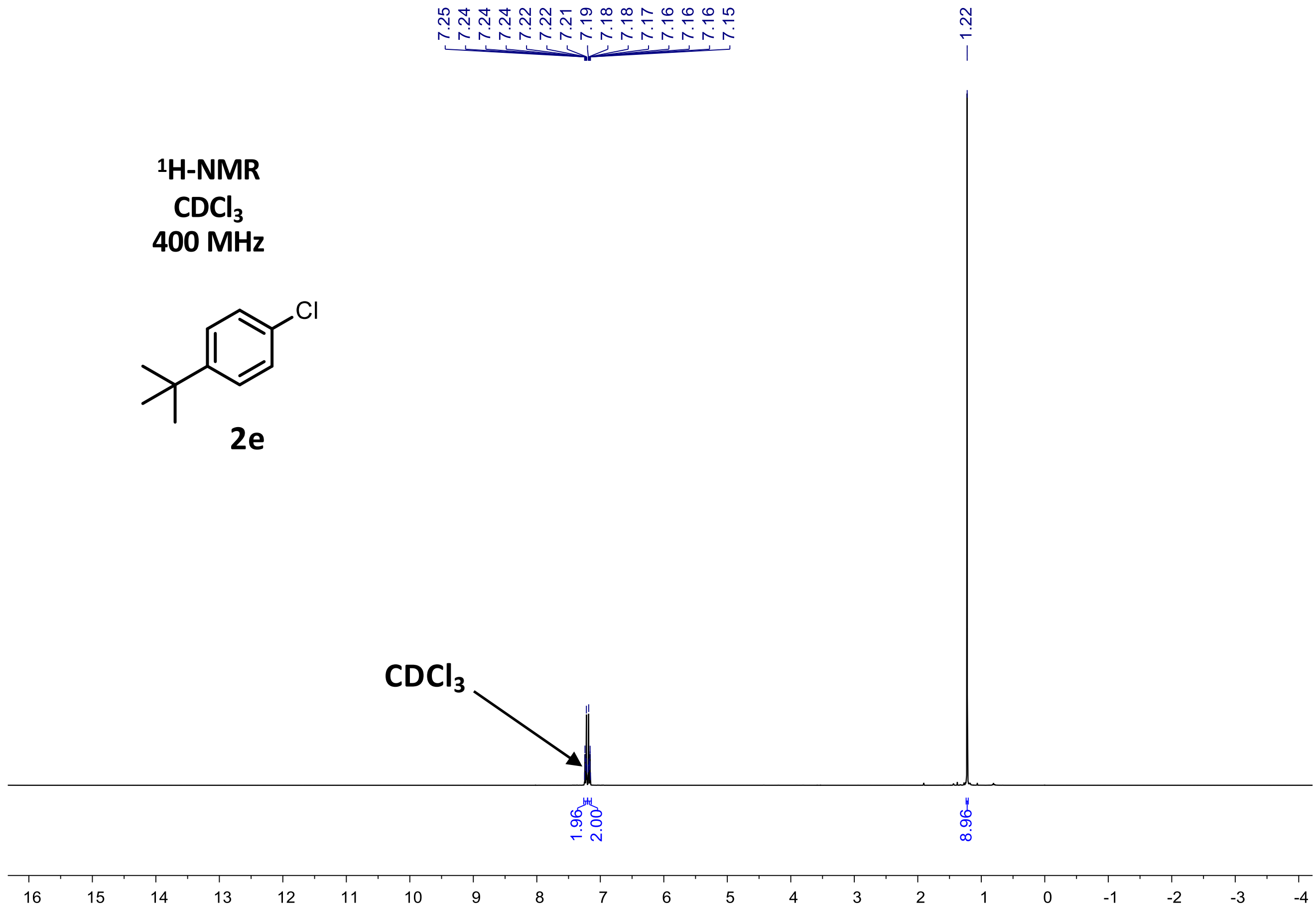


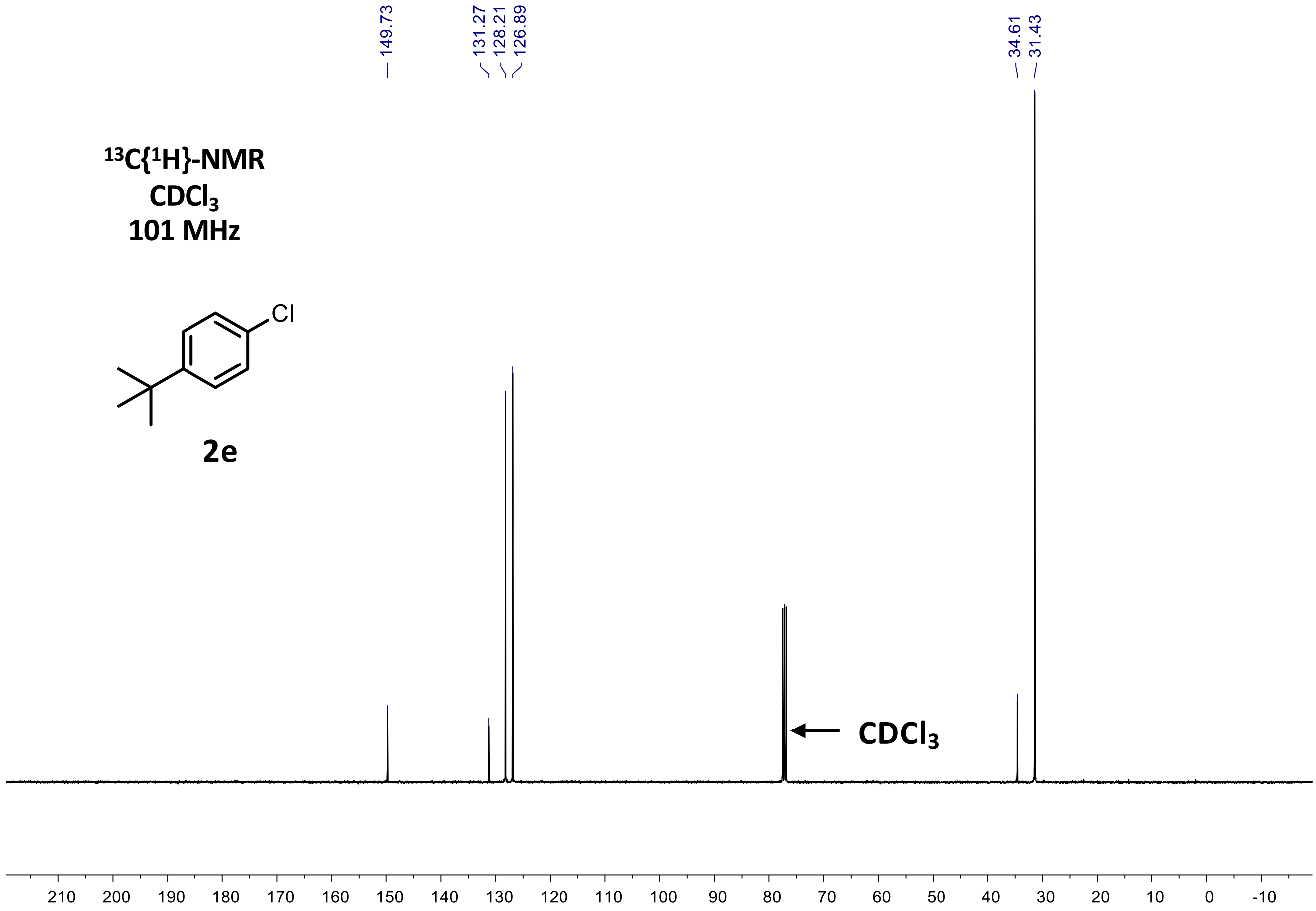
Signal: FID1A

RT [min]	Type	Width [min]	Area	Height	Area%	Name
1.211	VB	0.83	1240.88	1544.28	49.77	
4.818	BV R	0.42	1223.86	1103.92	49.09	
10.881	VV R	0.70	13.92	1.07	0.56	
11.740	BB	0.39	14.50	2.06	0.58	
		Sum	**2493.16**			

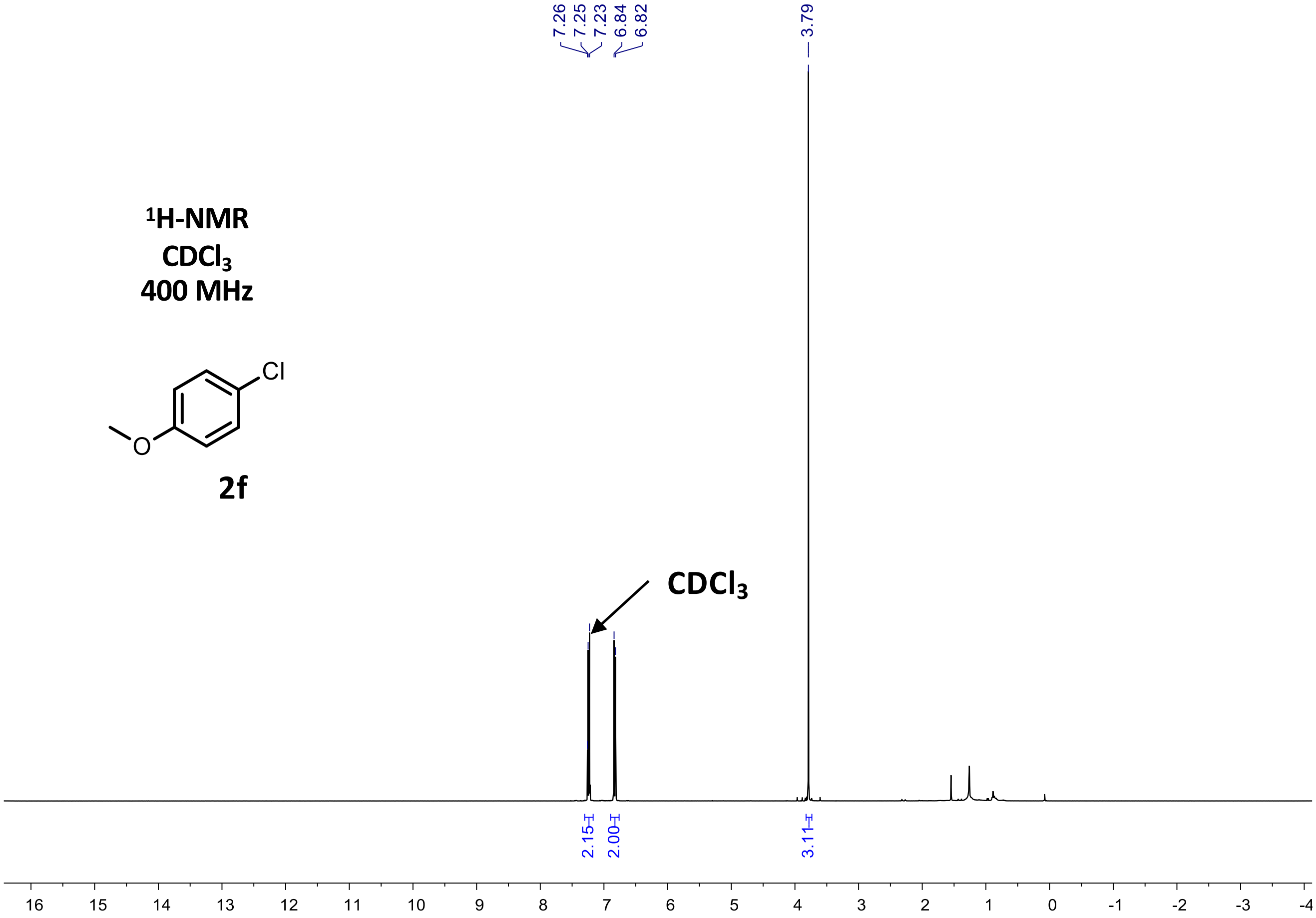

7.35 7.34 7.34 7.34 7.33 7.32 7.24 7.24 7.23 7.23 7.23 7.23 7.22 7.22 7.22 7.22 7.21 7.21 7.21 7.17 7.17 7.16 7.15 7.14 7.14 7.14 7.13 7.12 7.12 7.12 7.12 7.11 7.10 7.10
2.38
¹H-NMR
CDCl₃
400 MHz
Cl
2d
CDCl₃
1.00
0.97
2.01
3.00
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4


136.17
134.51
131.08
129.20
127.22
126.70
20.18
¹³C{¹H}-NMR
CDCl₃
101 MHz
Cl
2d
CDCl₃
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

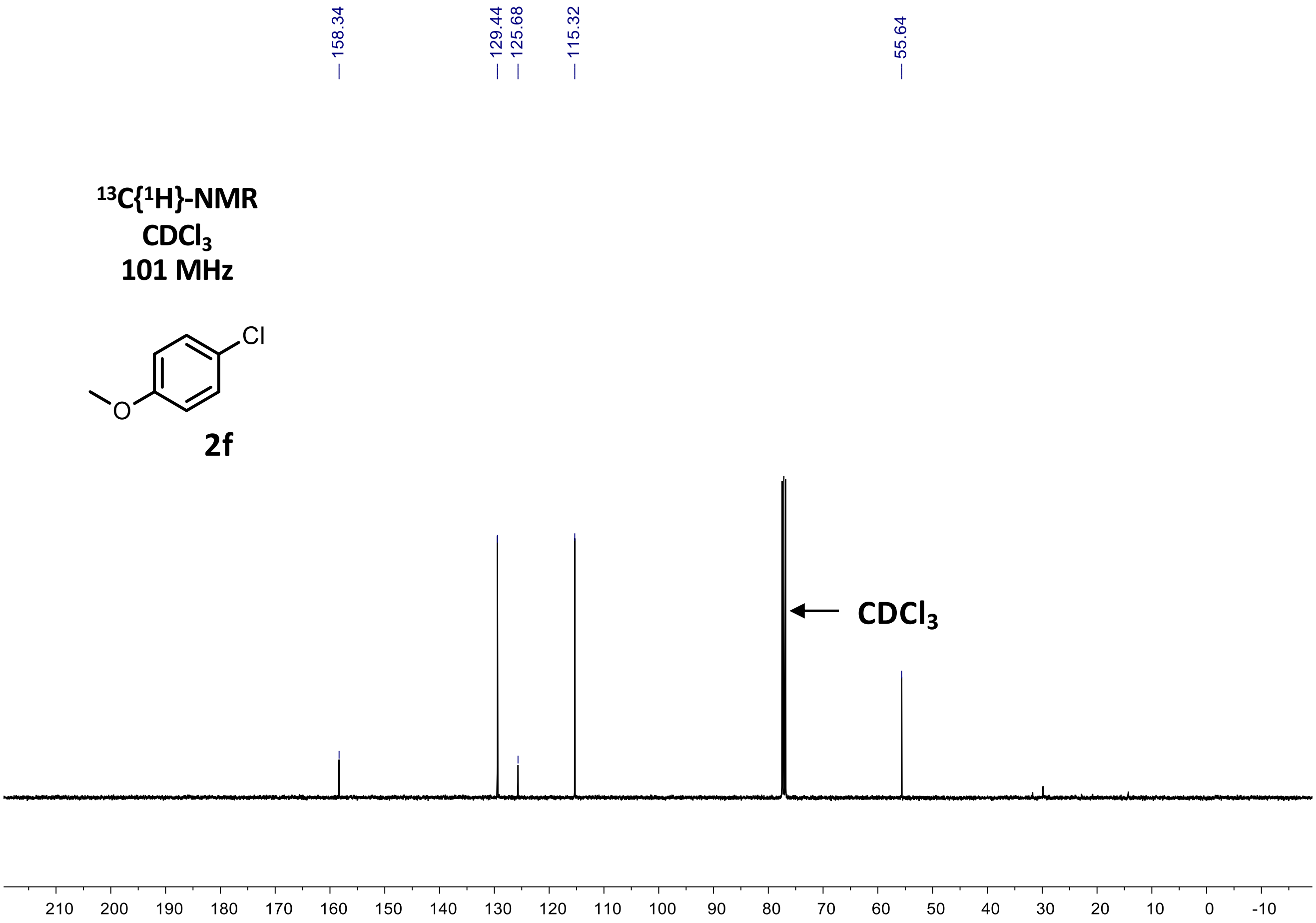

7.25
7.24
7.24
7.24
7.22
7.22
7.21
7.19
7.18
7.18
7.17
7.16
7.16
7.16
7.15
1.22
¹H-NMR
CDCl₃
400 MHz
Cl
2e
CDCl₃
1.96
2.00
8.96
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
-4

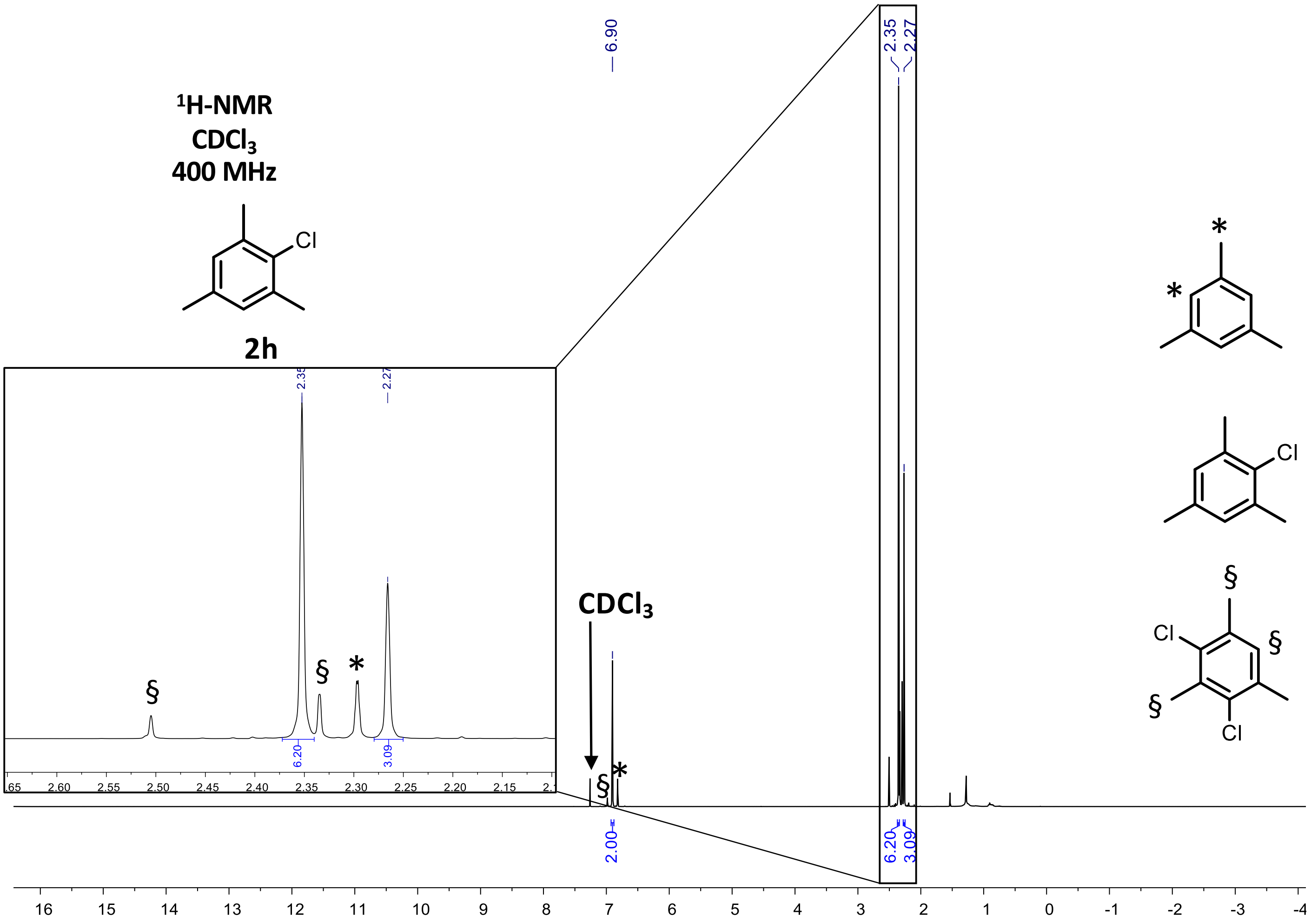

149.73
131.27
128.21
126.89
34.61
31.43
¹³C{¹H}-NMR
CDCl₃
101 MHz
Cl
2e
CDCl₃
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10

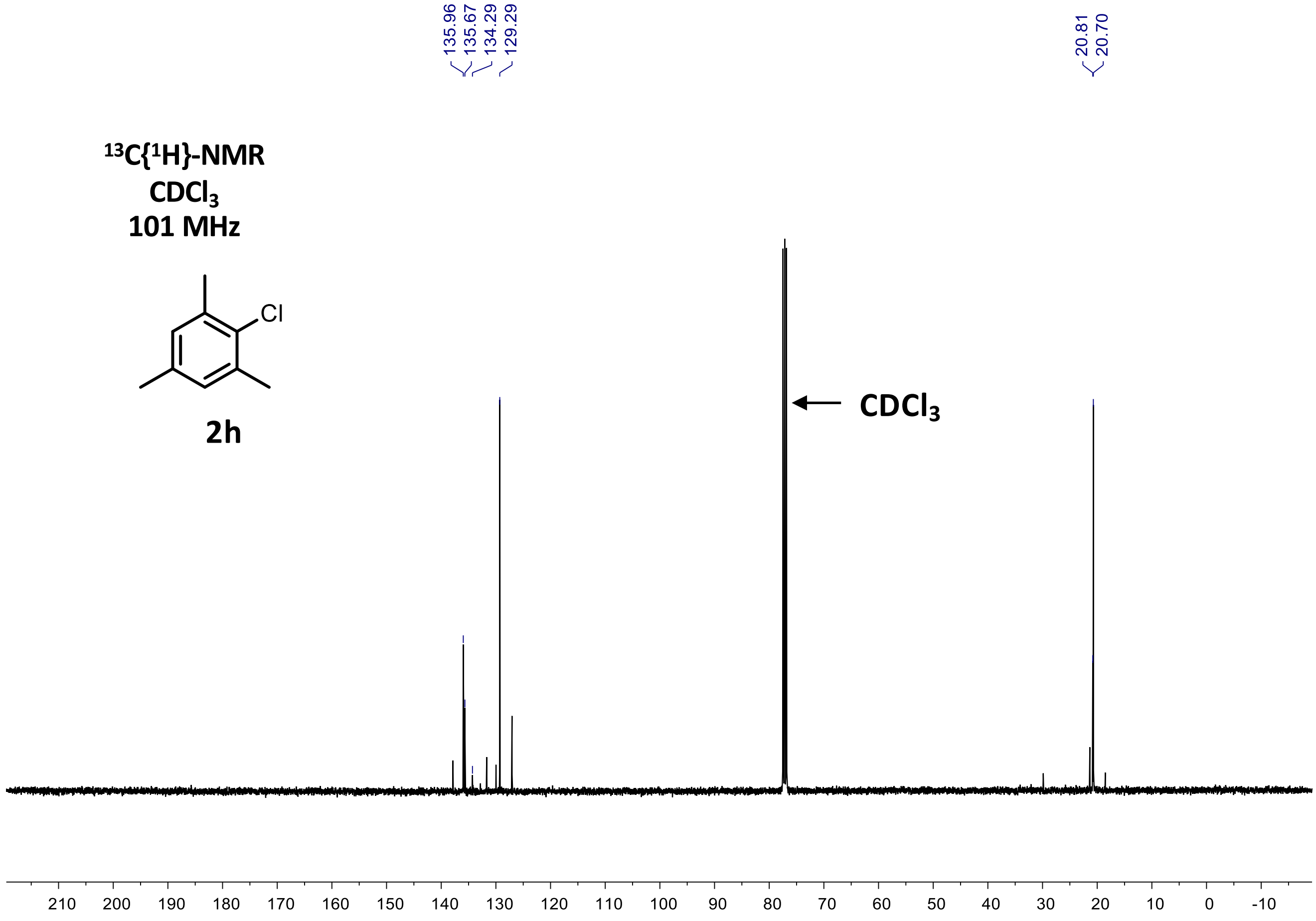
^{1}H-NMR
CDCl$_3$
400 MHz

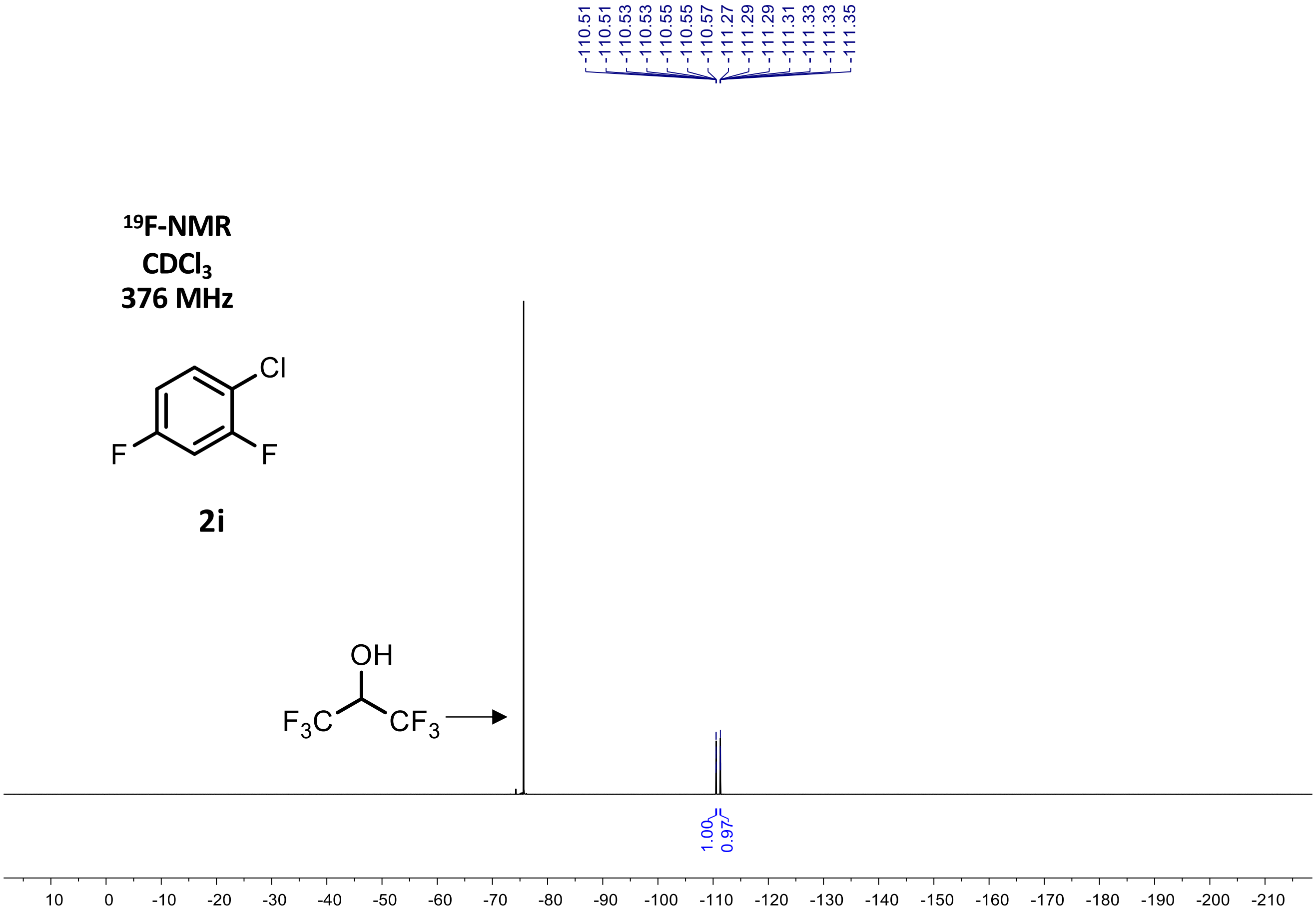


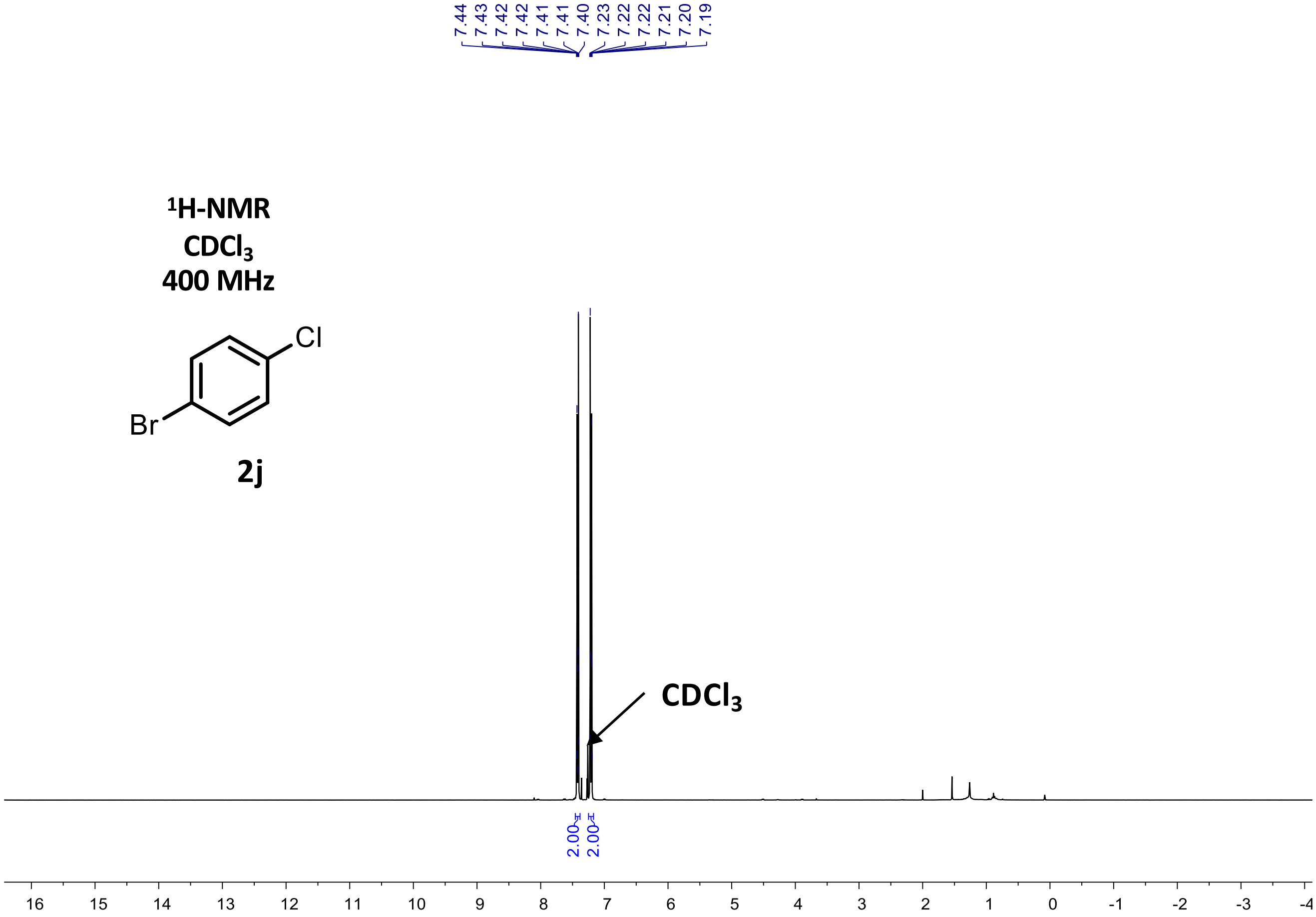
^{13}C{^{1}H}-NMR
CDCl$_3$
101 MHz



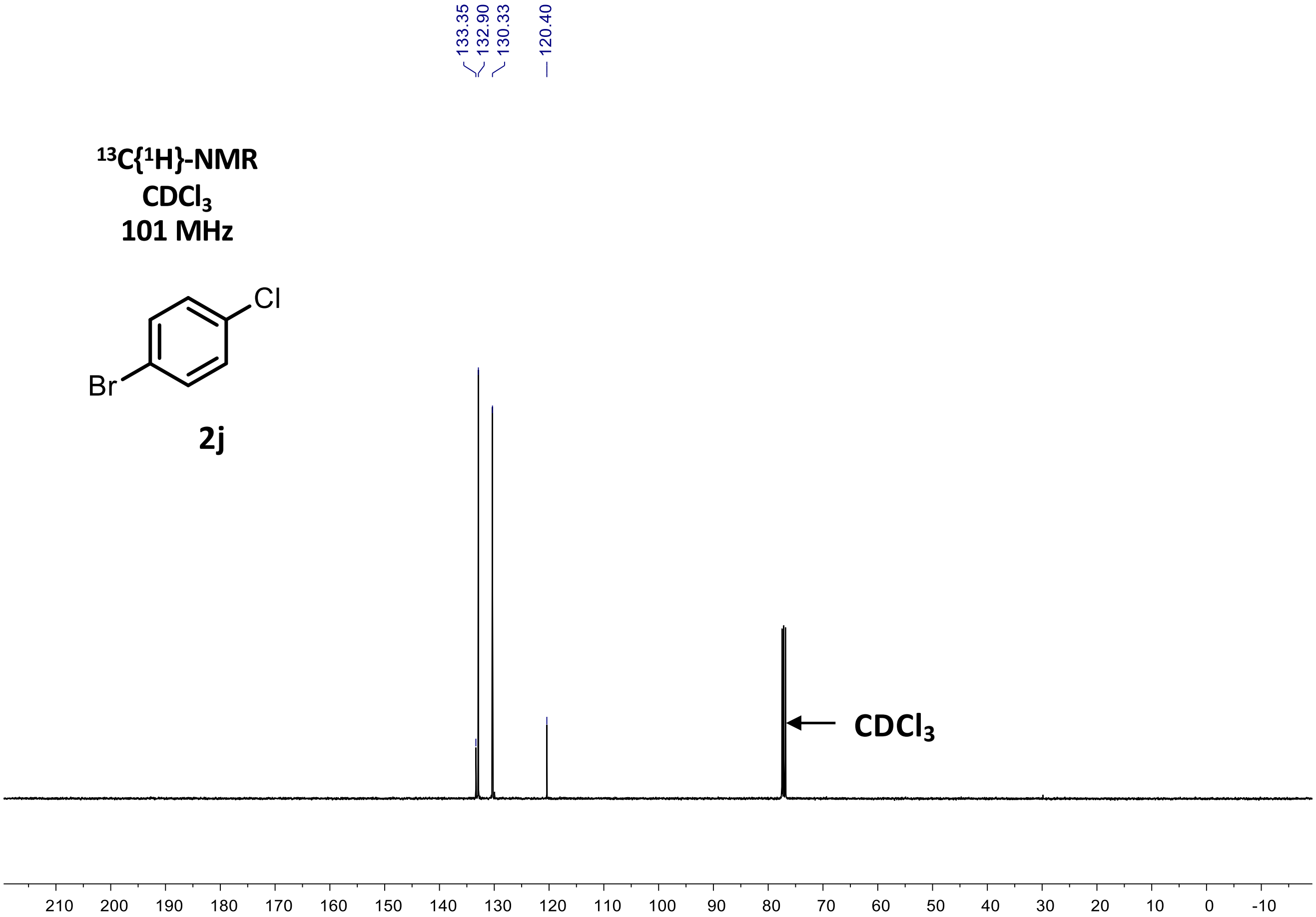

¹H-NMR
CDCl₃
400 MHz
Cl
2h
6.90
2.35
2.27
§
§
*
2.35
2.27
6.20
3.09
2.65 2.60 2.55 2.50 2.45 2.40 2.35 2.30 2.25 2.20 2.15 2.1
CDCl₃
§
*
2.00
6.20
3.09
*
*
Cl
§
Cl
§
§
Cl
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4


135.96
135.67
134.29
129.29
20.81
20.70
¹³C{¹H}-NMR
CDCl₃
101 MHz
Cl
2h
CDCl₃
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

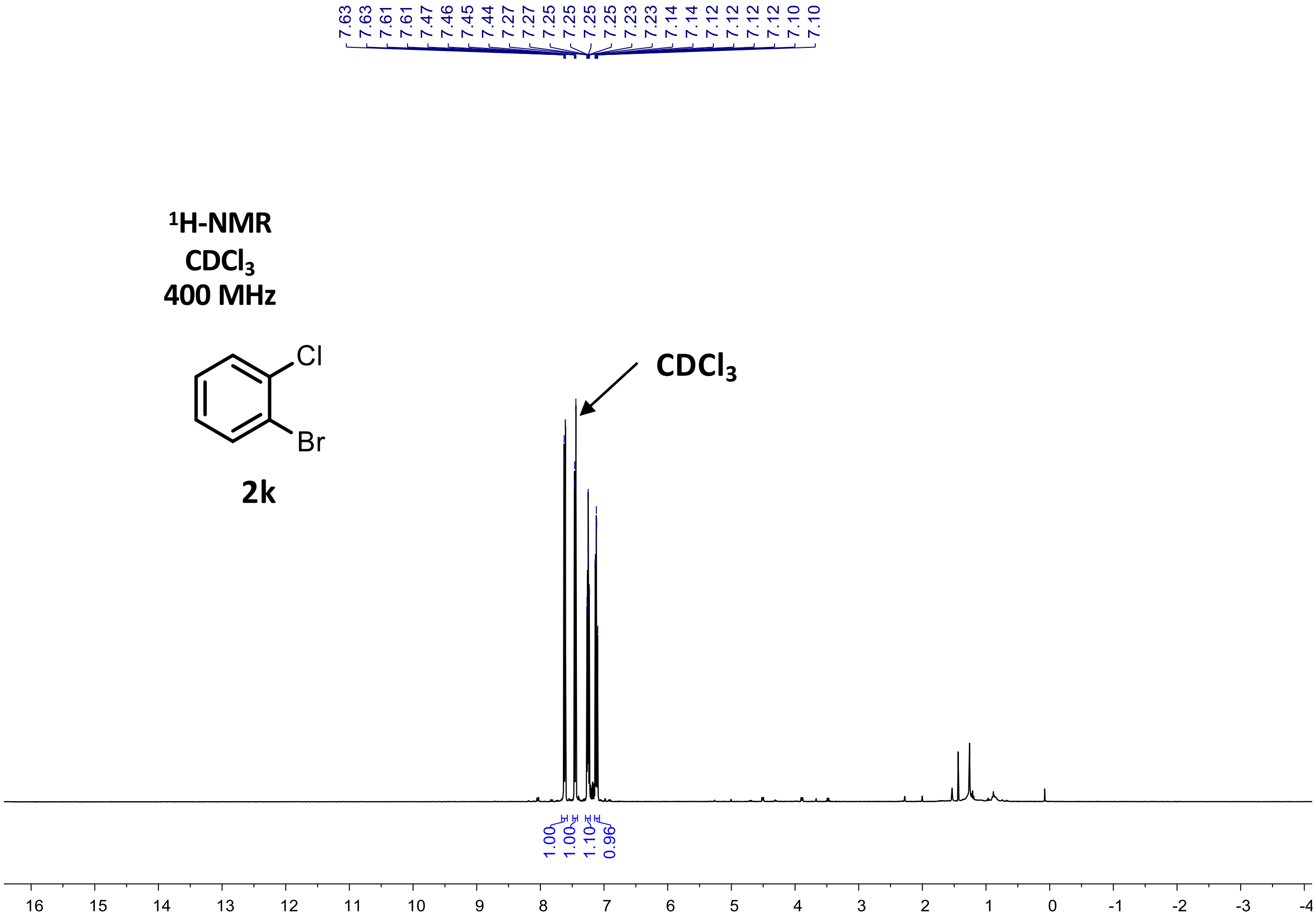

-110.51
-110.51
-110.53
-110.53
-110.55
-110.55
-110.57
-111.27
-111.29
-111.29
-111.31
-111.33
-111.33
-111.35
^{19}F-NMR
CDCl$_3$
376 MHz
Cl
F
F
2i
OH
F$_3$C
CF$_3$
1.00
0.97
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210

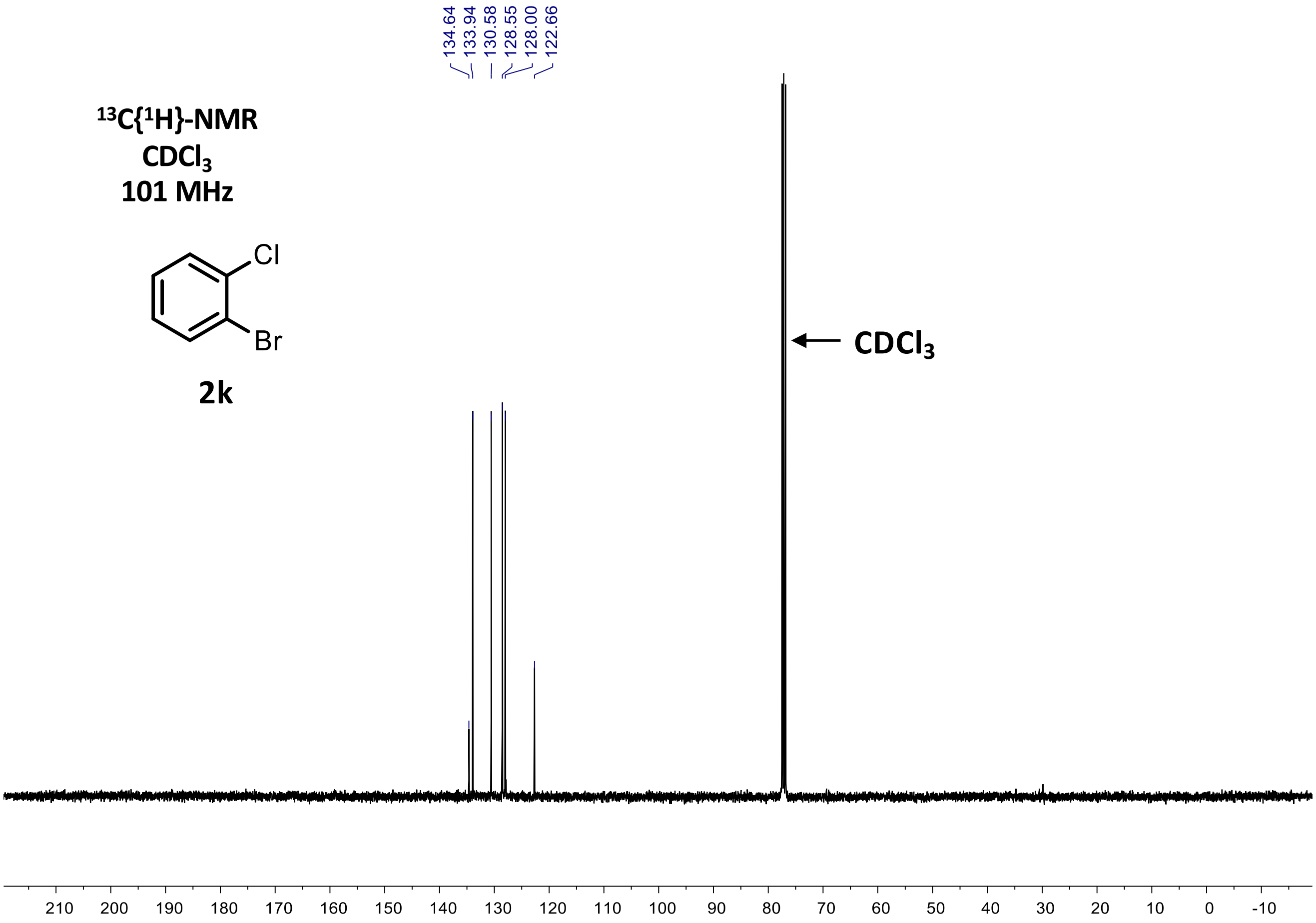

7.44
7.43
7.42
7.42
7.41
7.41
7.40
7.23
7.22
7.22
7.21
7.20
7.19
^{1}H-NMR
$CDCl_3$
400 MHz
Cl
Br
$CDCl_3$
2.00
2.00
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
-4

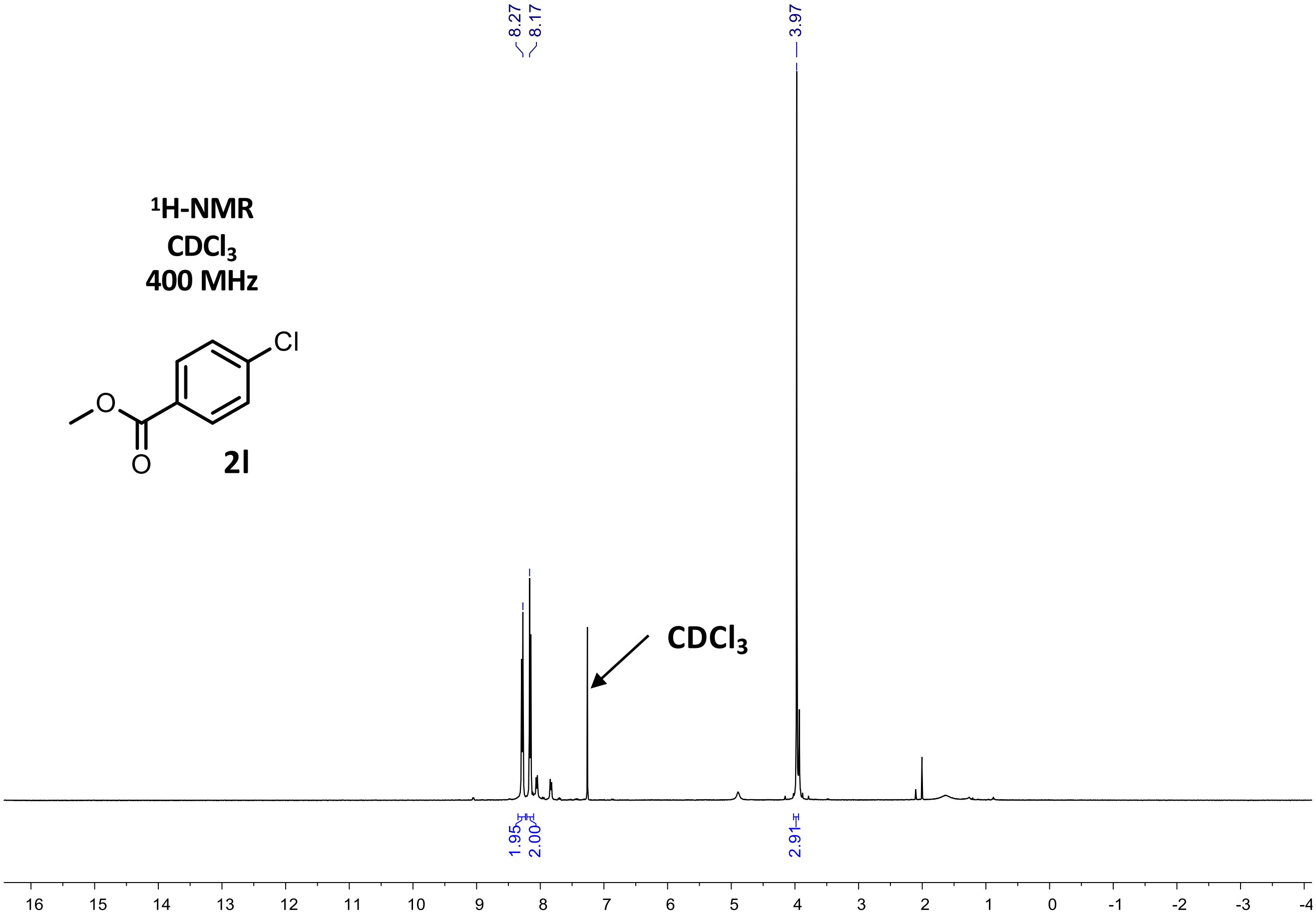
2j

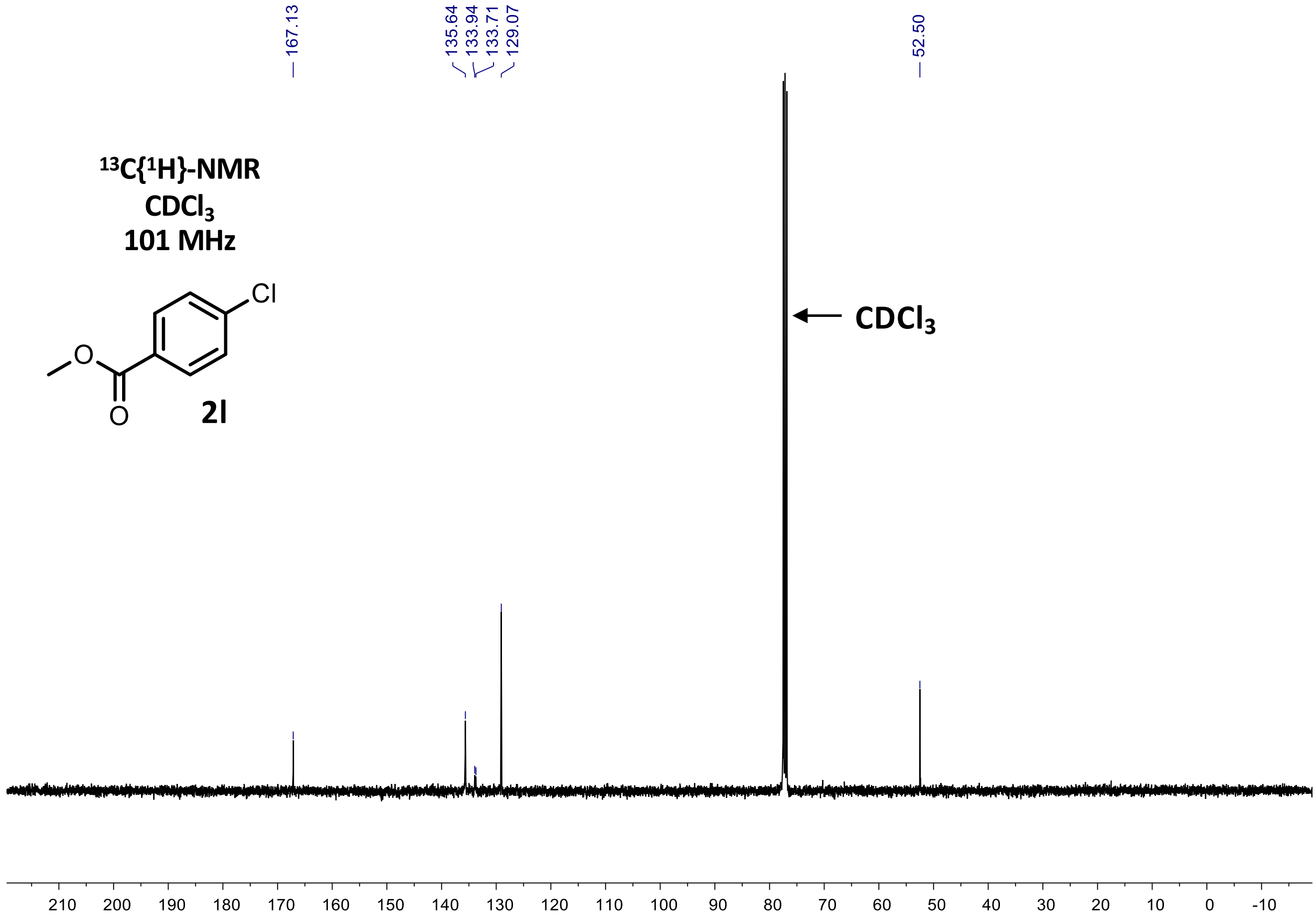

133.35
132.90
130.33
120.40
^{13}C{^{1}H}-NMR
$CDCl_3$
101 MHz
Cl
Br
$CDCl_3$
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10

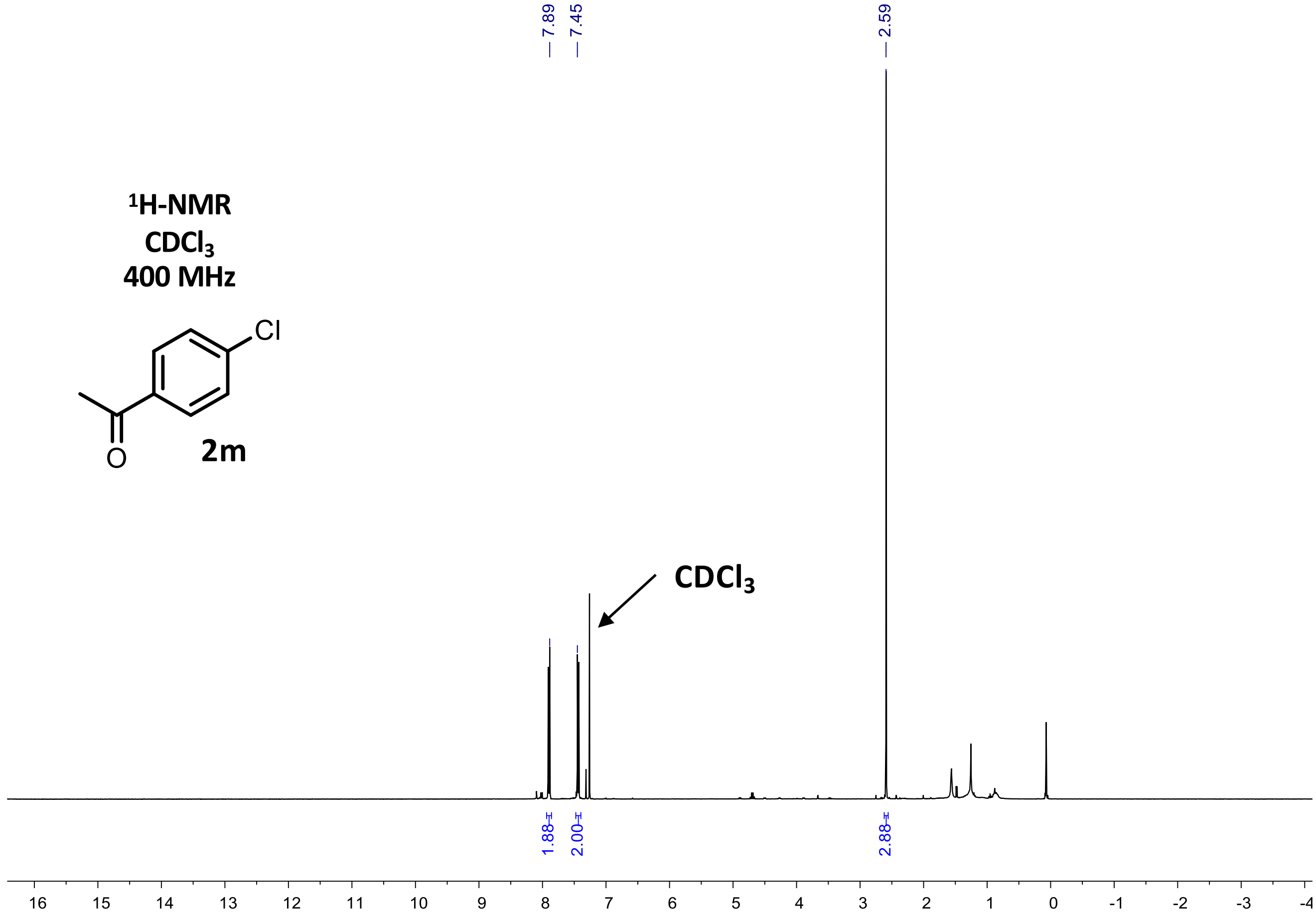
2j


7.63
7.63
7.61
7.61
7.47
7.46
7.45
7.44
7.27
7.27
7.25
7.25
7.25
7.25
7.23
7.23
7.14
7.14
7.12
7.12
7.12
7.12
7.10
7.10
^{1}H-NMR
CDCl$_3$
400 MHz
Cl
Br
2k
CDCl$_3$
1.00
1.00
1.10
0.96
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
-4

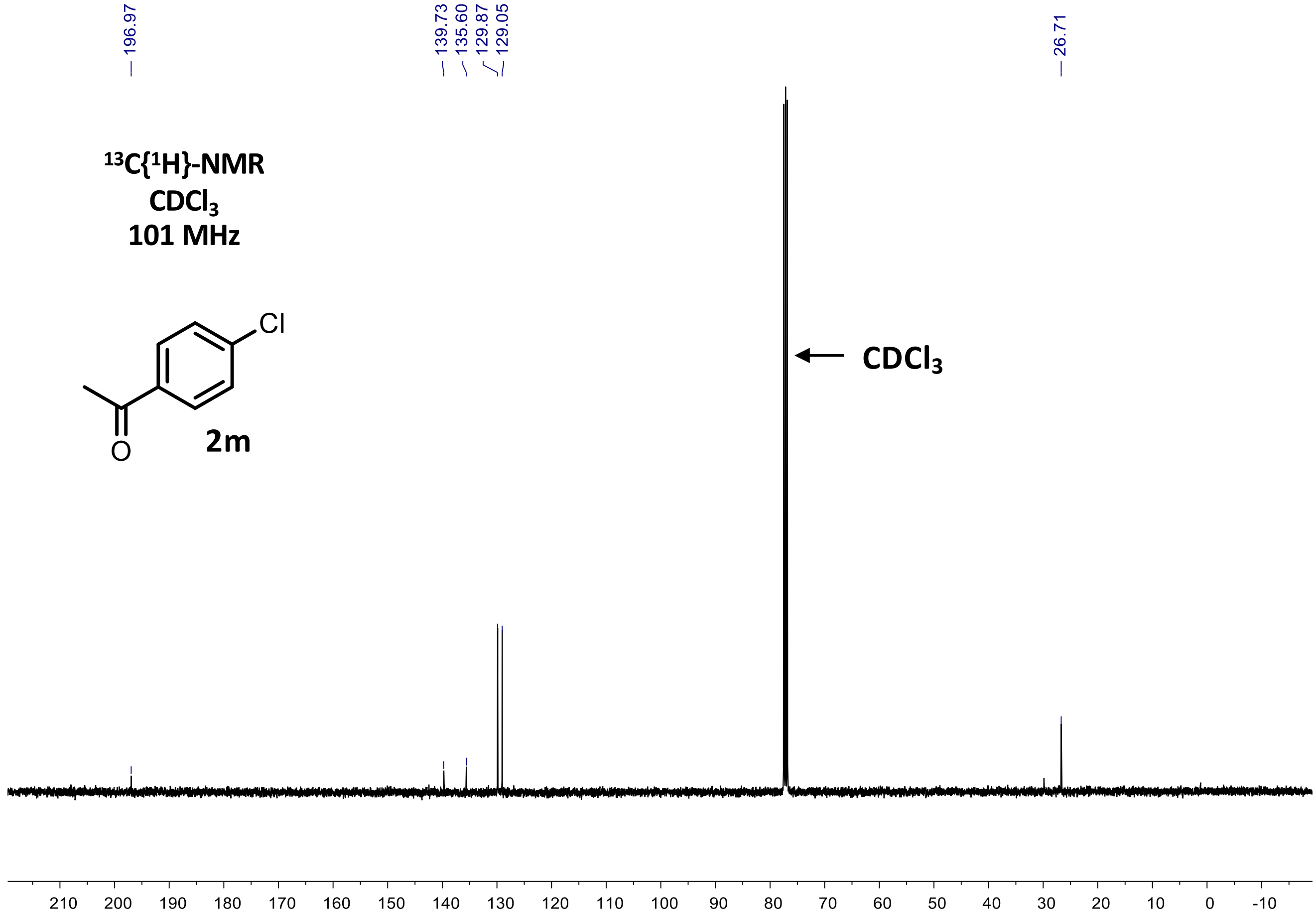

134.64
133.94
130.58
128.55
128.00
122.66
^{13}C{^{1}H}-NMR
CDCl$_3$
101 MHz
Cl
Br
2k
CDCl$_3$
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10


8.27
8.17
3.97
^{1}H-NMR
$CDCl_3$
400 MHz
Cl
O
O
2l
$CDCl_3$
1.95
2.00
2.91
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
-4

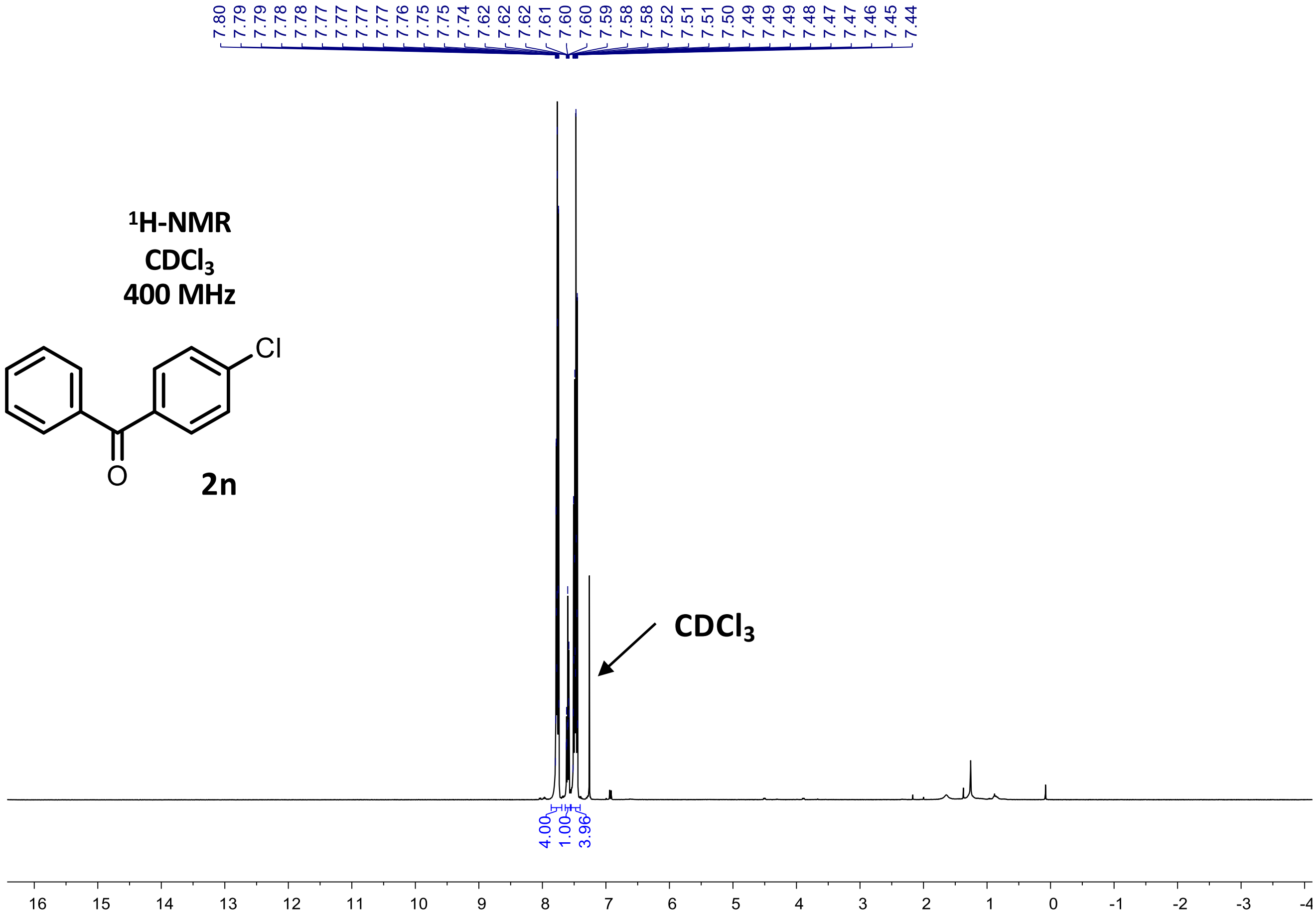

167.13
135.64
133.94
133.71
129.07
52.50
^{13}C{^{1}H}-NMR
$CDCl_3$
101 MHz
Cl
O
O
2l
$CDCl_3$
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10

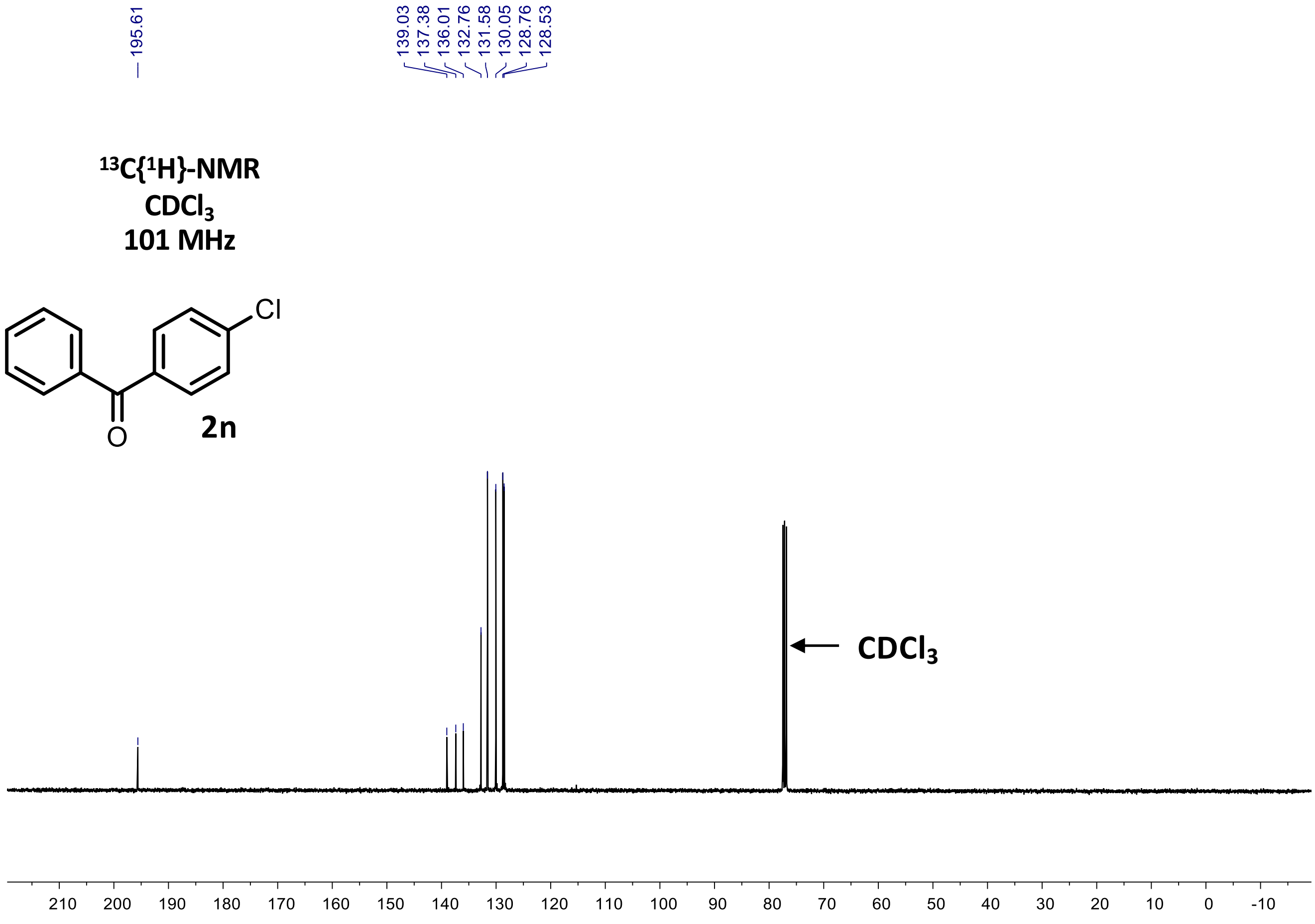


7.89
7.45
2.59
^{1}H-NMR
$CDCl_3$
400 MHz
Cl
O
2m
$CDCl_3$
1.88
2.00
2.88
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
-4

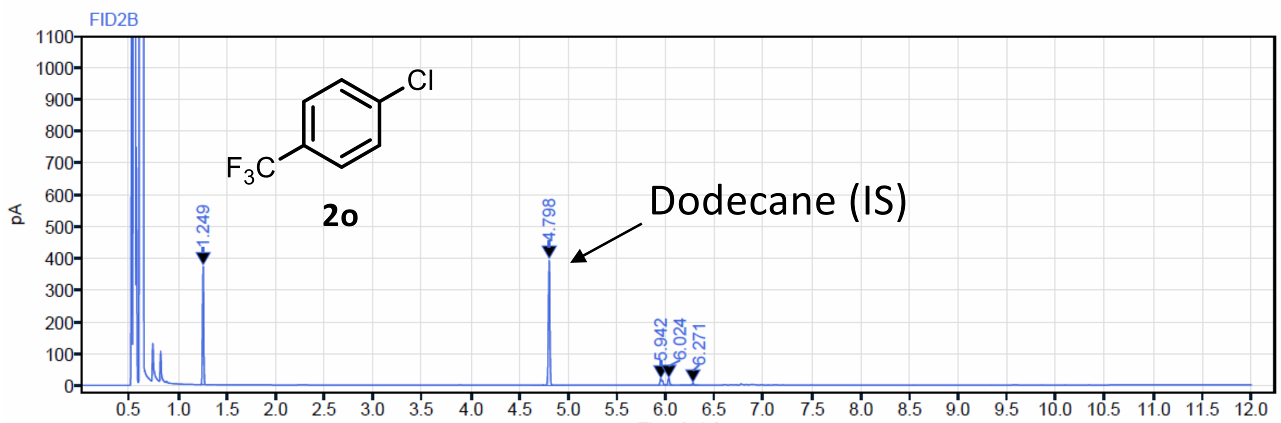


196.97
139.73
135.60
129.87
129.05
26.71
$^{13}C\{^1H\}$-NMR
$CDCl_3$
101 MHz
Cl
O
2m
$CDCl_3$
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10


7.80
7.79
7.79
7.78
7.78
7.77
7.77
7.77
7.77
7.76
7.75
7.75
7.74
7.62
7.62
7.62
7.61
7.60
7.60
7.59
7.58
7.58
7.52
7.51
7.51
7.50
7.49
7.49
7.49
7.48
7.47
7.47
7.46
7.45
7.44
^{1}H-NMR
CDCl$_3$
400 MHz
Cl
O
2n
CDCl$_3$
4.00
1.00
3.96
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
-4


195.61
139.03
137.38
136.01
132.76
131.58
130.05
128.76
128.53
^{13}C{^{1}H}-NMR
CDCl$_3$
101 MHz
Cl
O
2n
CDCl$_3$
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10

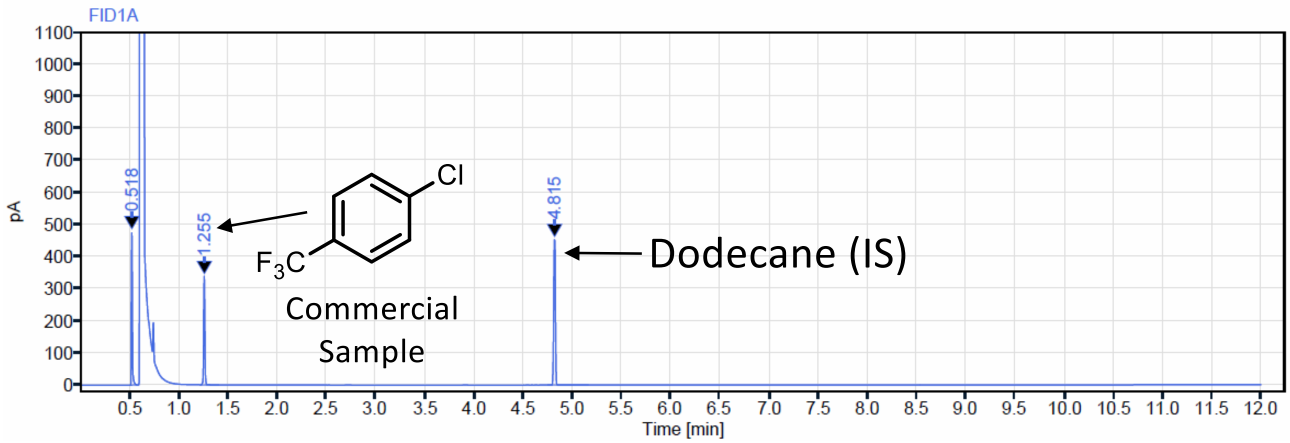
Gas Chromatogram



Post Electrolysis

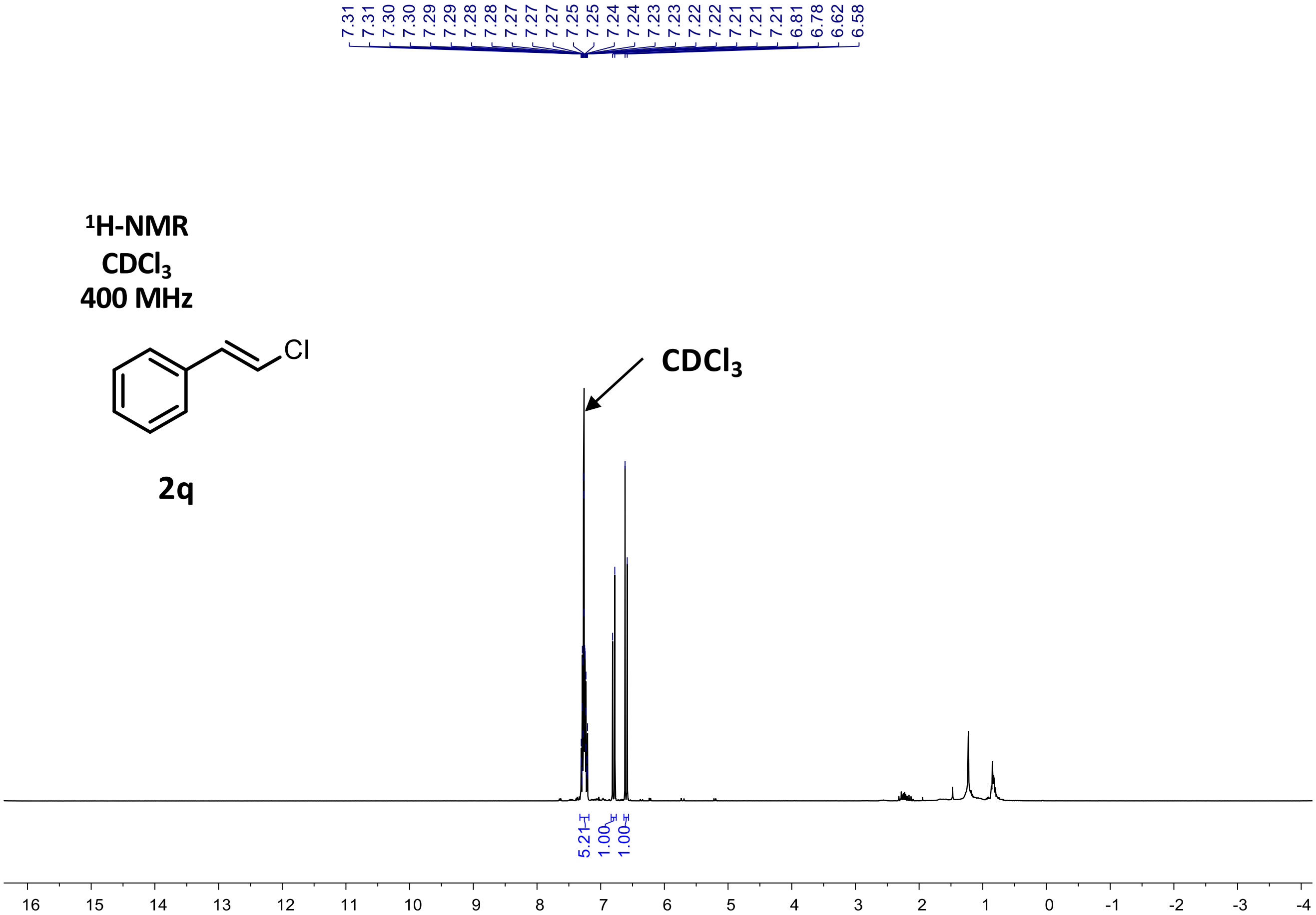
Signal: FID2B

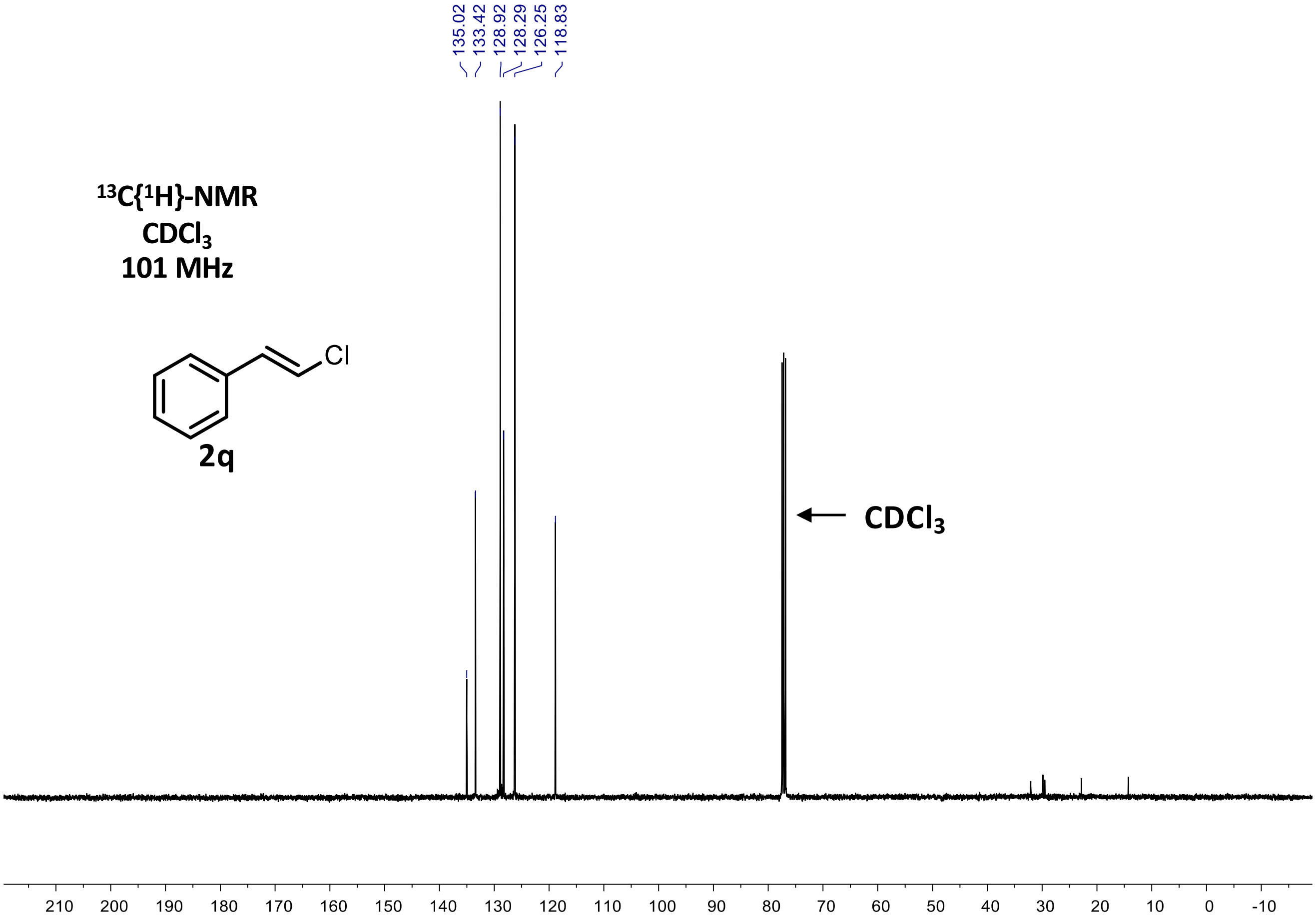
RT [min]	Type	Width [min]	Area	Height	Area%	Name
1.249	BB	0.15	313.36	374.48	39.02	
4.798	VV R	0.34	421.47	396.57	52.48	
5.942	BV R	0.08	34.50	19.94	4.30	
6.024	BB	0.06	26.59	21.49	3.31	
6.271	VB R	0.19	7.15	7.17	0.89	
		Sum	**803.07**			

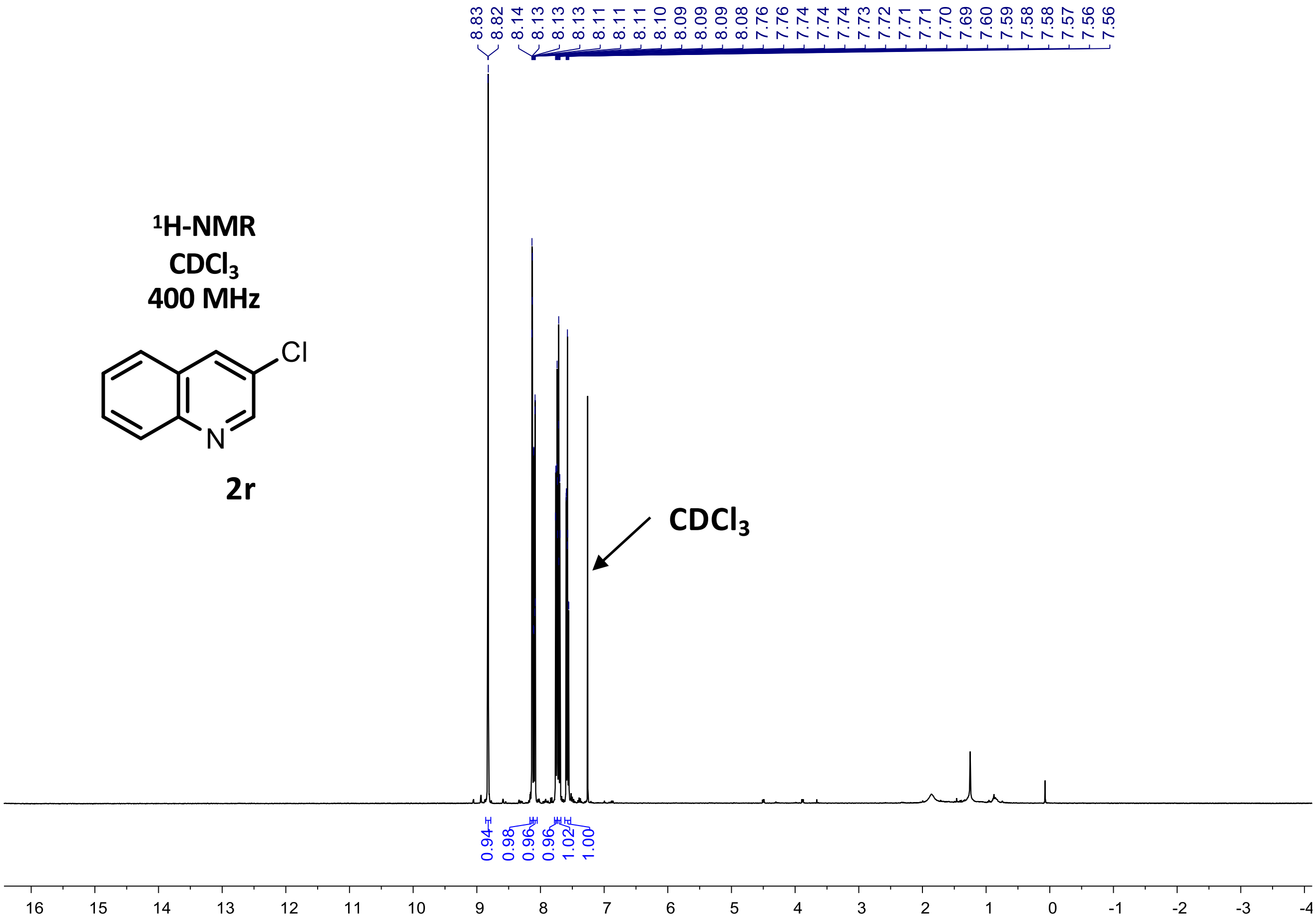


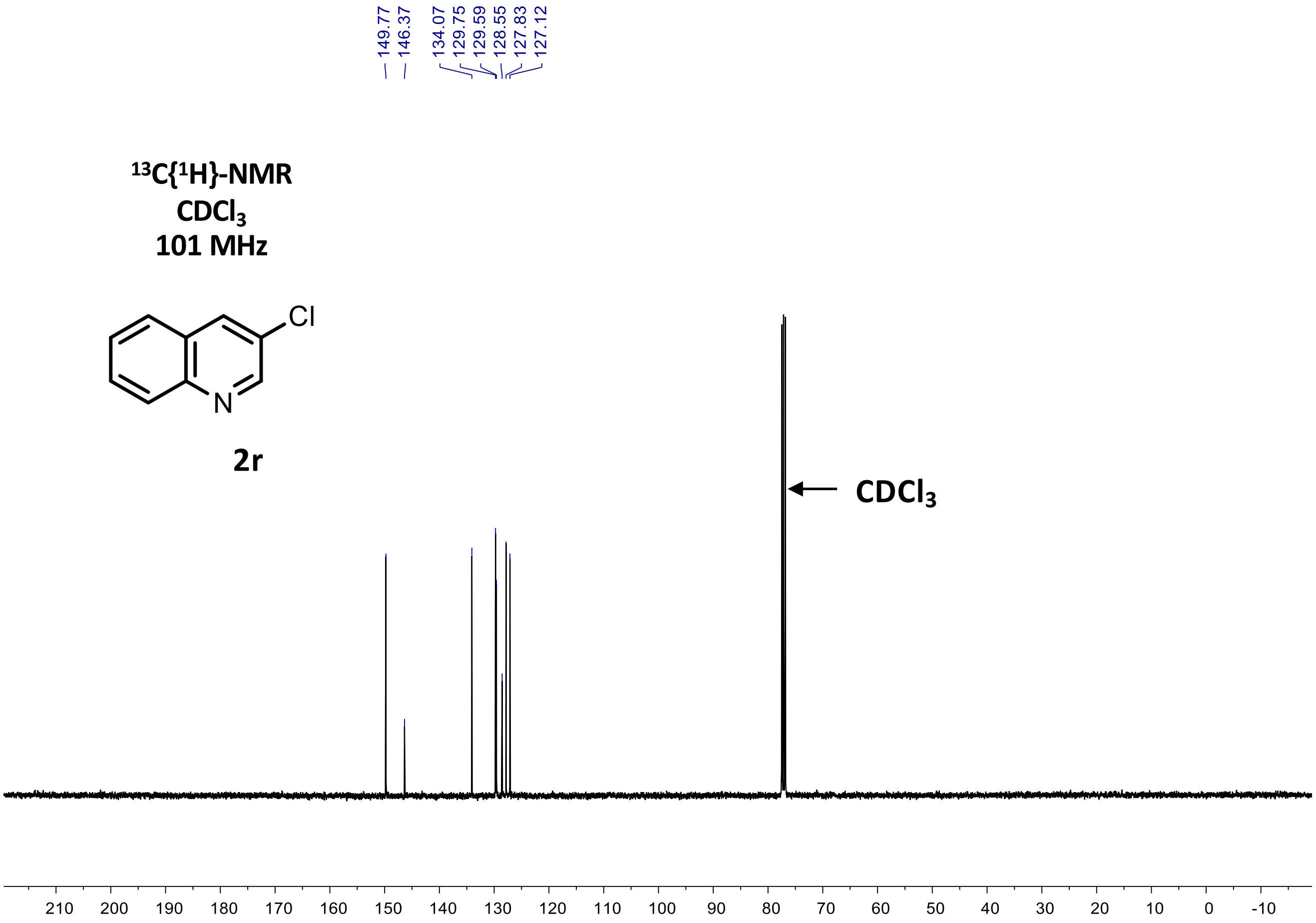
Signal: FID1A

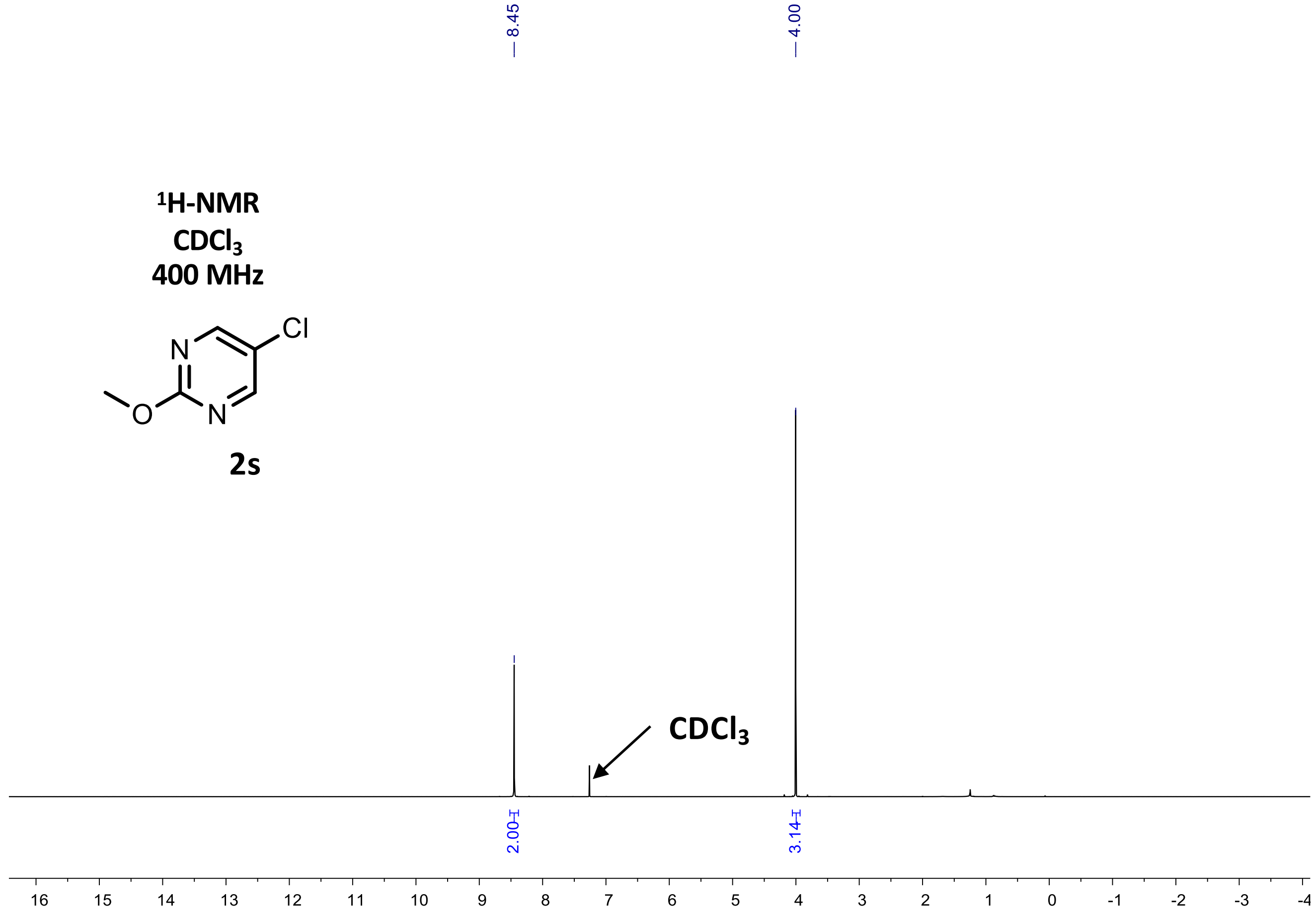
RT [min]	Type	Width [min]	Area	Height	Area%	Name
0.518	BB	0.08	333.32	485.26	30.05	
1.255	BV R	0.19	291.63	344.31	26.29	
4.815	BV R	0.42	484.15	460.61	43.65	
		Sum	**1109.10**			

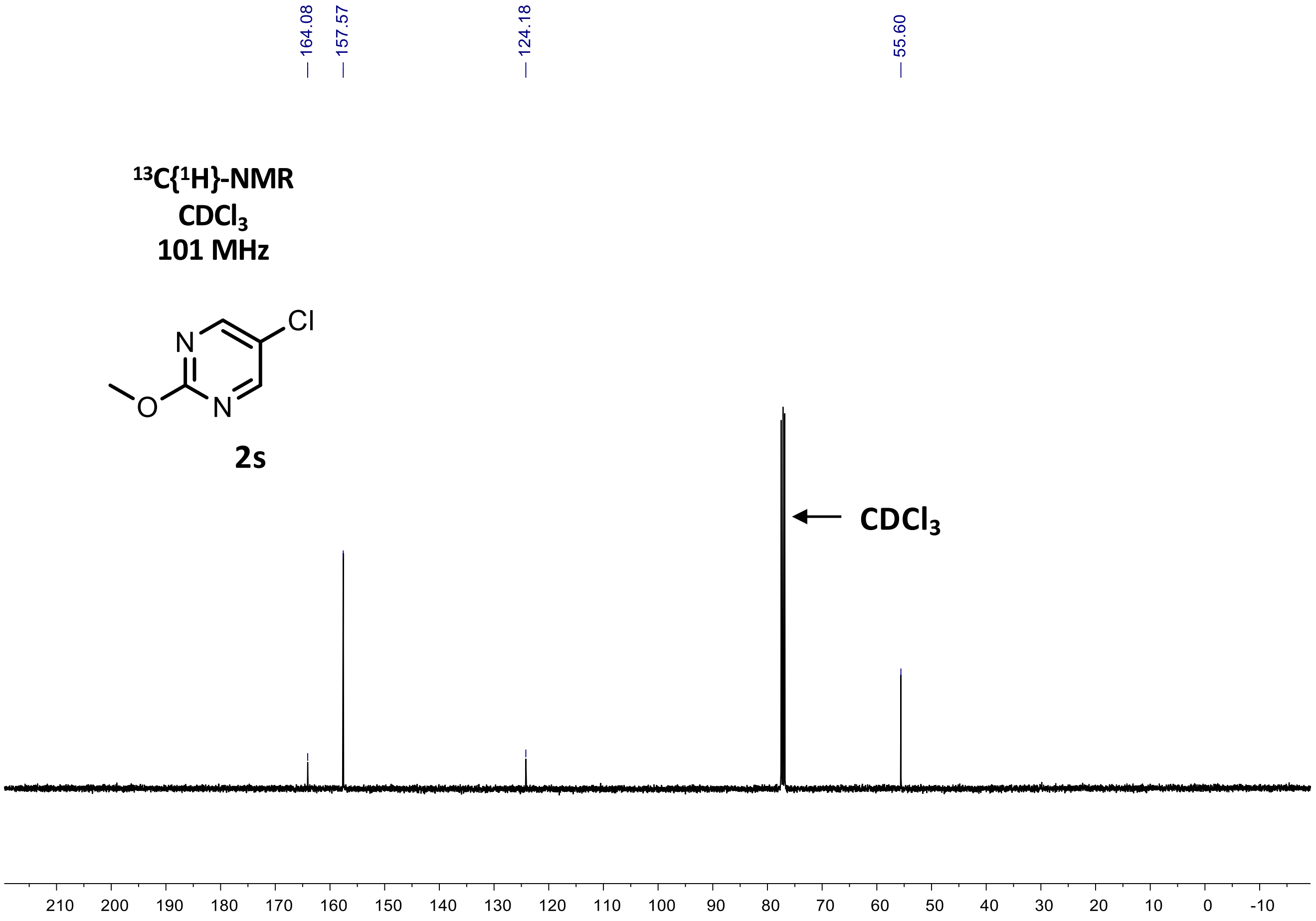

7.31
7.31
7.30
7.30
7.29
7.29
7.28
7.28
7.27
7.27
7.27
7.25
7.25
7.24
7.24
7.23
7.23
7.22
7.22
7.21
7.21
7.21
6.81
6.78
6.62
6.58
^{1}H-NMR
$CDCl_3$
400 MHz
Cl
2q
$CDCl_3$
5.21
1.00
1.00
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
-4

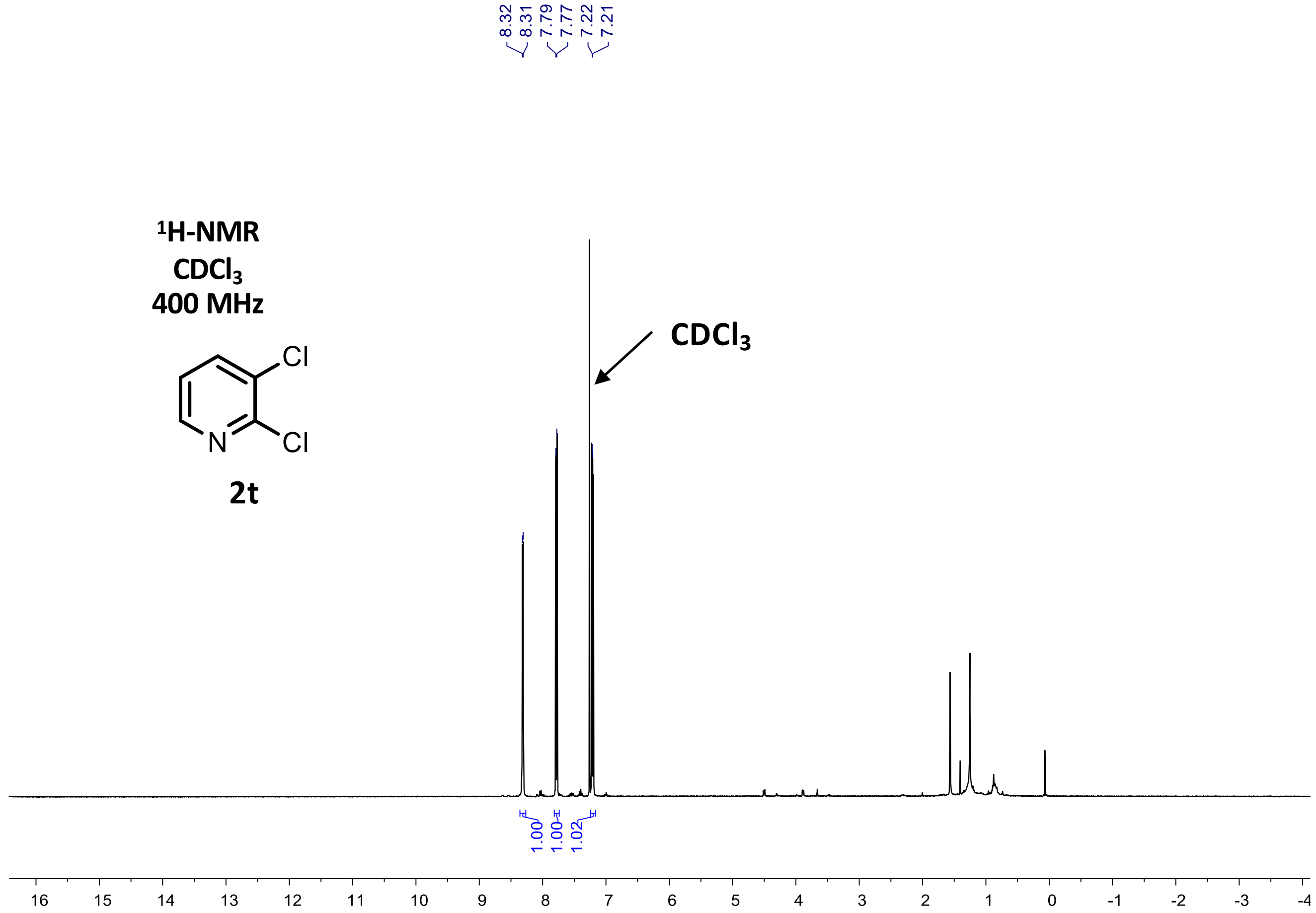

135.02
133.42
128.92
128.29
126.25
118.83
$^{13}C\{^1H\}$-NMR
$CDCl_3$
101 MHz
Cl
2q
$CDCl_3$
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10

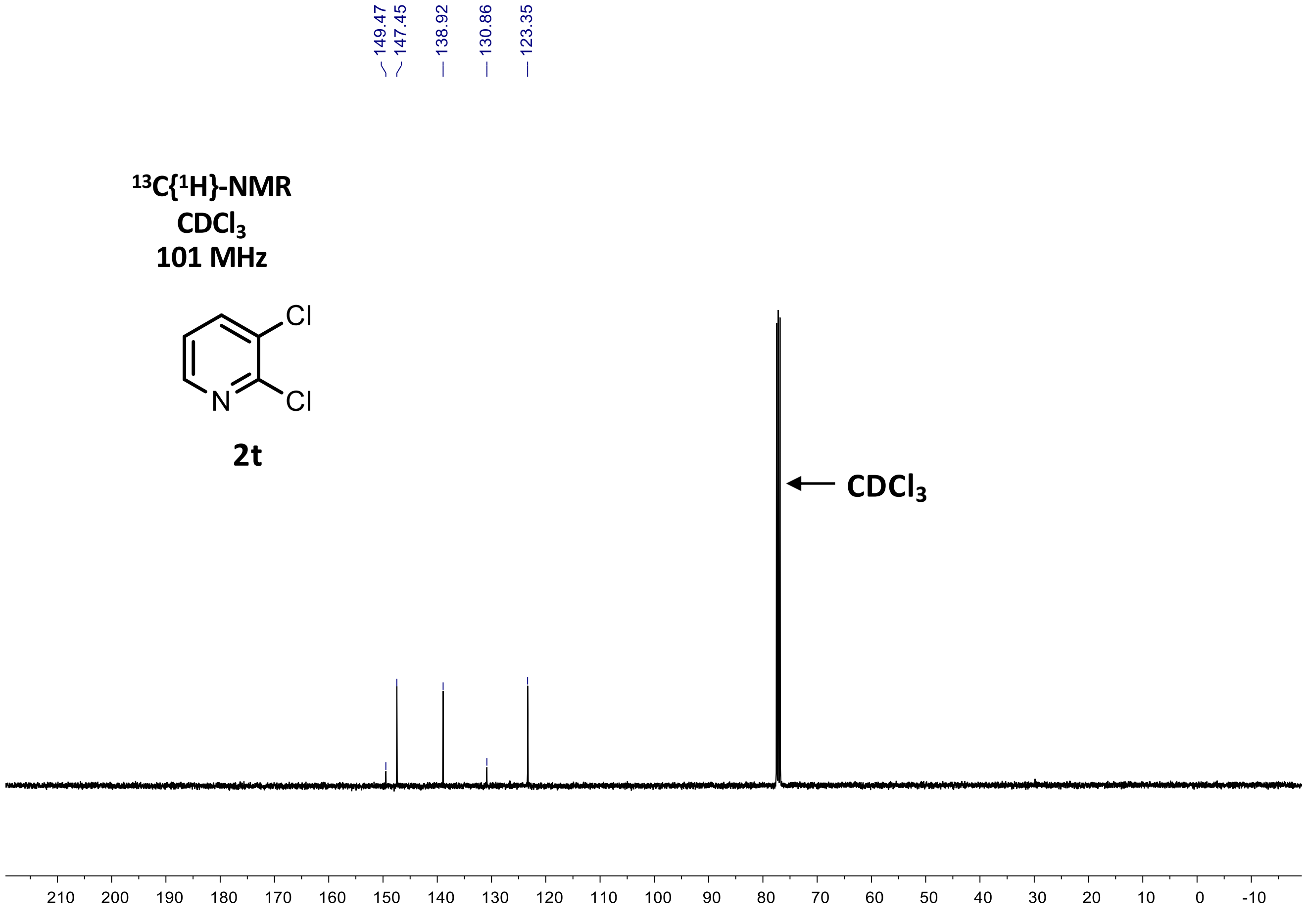

8.83
8.82
8.14
8.13
8.13
8.13
8.11
8.11
8.11
8.10
8.09
8.09
8.09
8.08
7.76
7.74
7.74
7.74
7.73
7.72
7.71
7.71
7.70
7.69
7.60
7.59
7.58
7.58
7.57
7.56
7.56
¹H-NMR
CDCl₃
400 MHz
Cl
N
2r
CDCl₃
0.94
0.98
0.96
0.96
1.02
1.00
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
-4

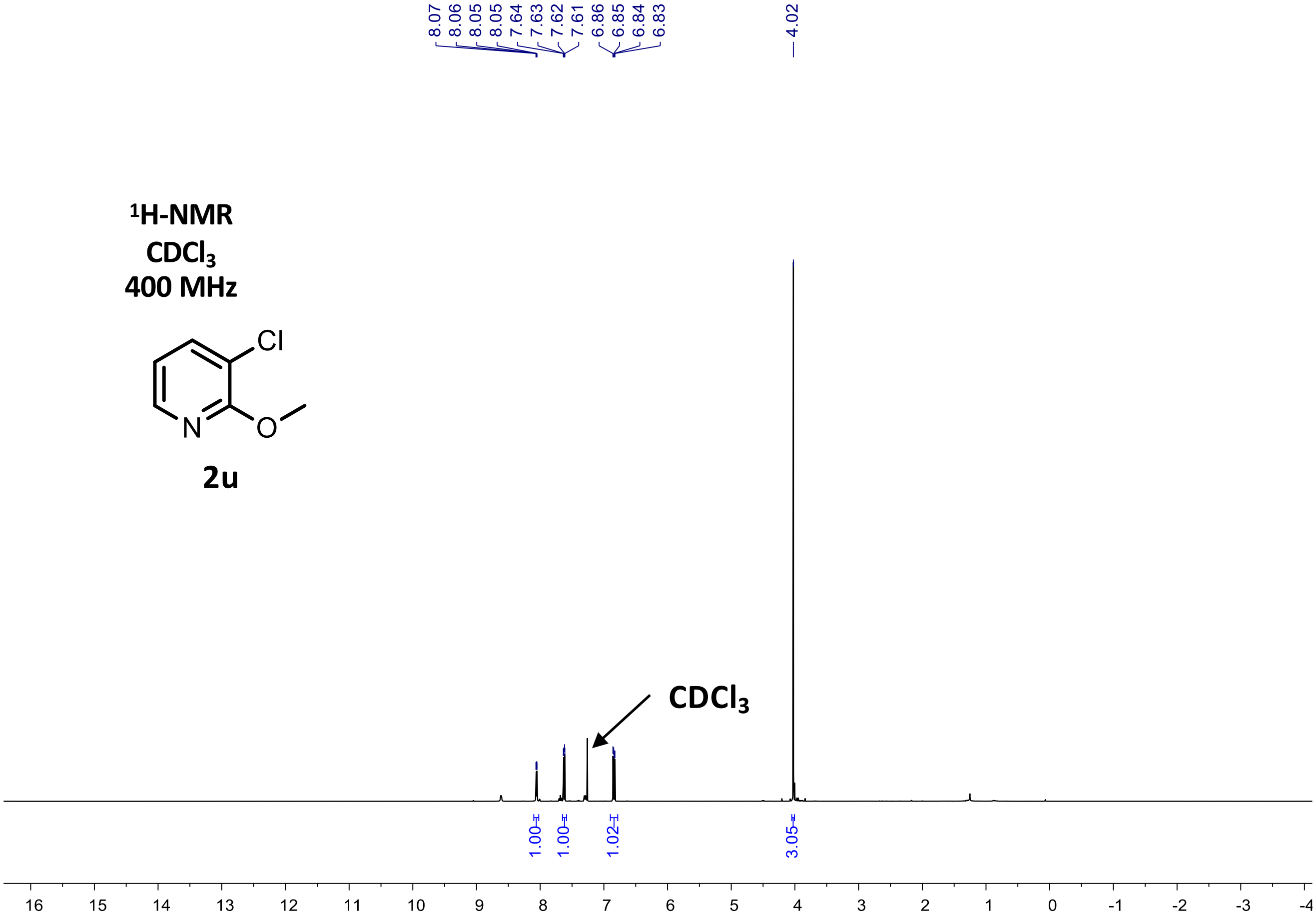

149.77
146.37
134.07
129.75
129.59
128.55
127.83
127.12
¹³C{¹H}-NMR
CDCl₃
101 MHz
Cl
N
2r
CDCl₃
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10

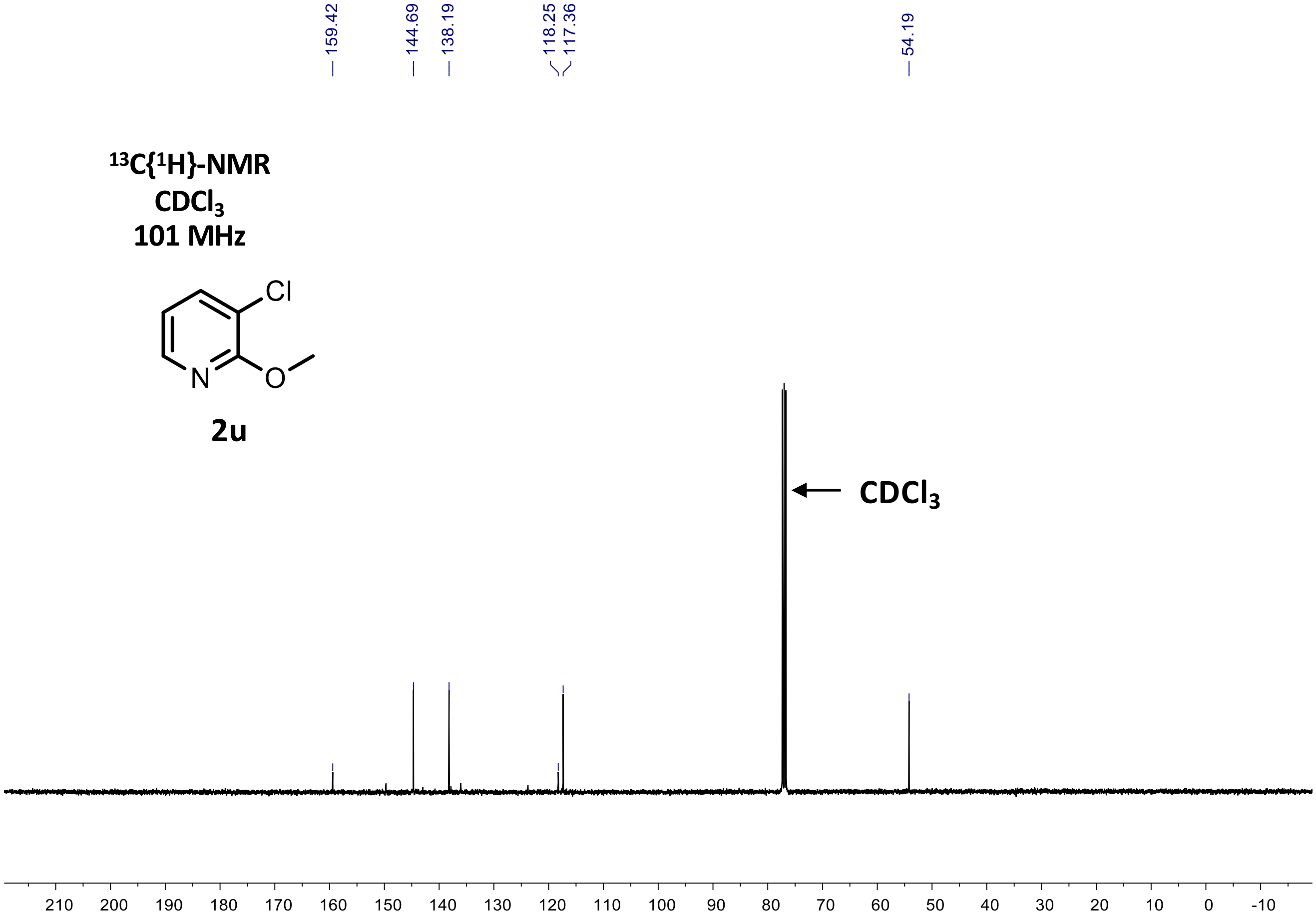

8.45
4.00
¹H-NMR
CDCl₃
400 MHz
Cl
N
N
O
2s
CDCl₃
2.00
3.14
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
-4

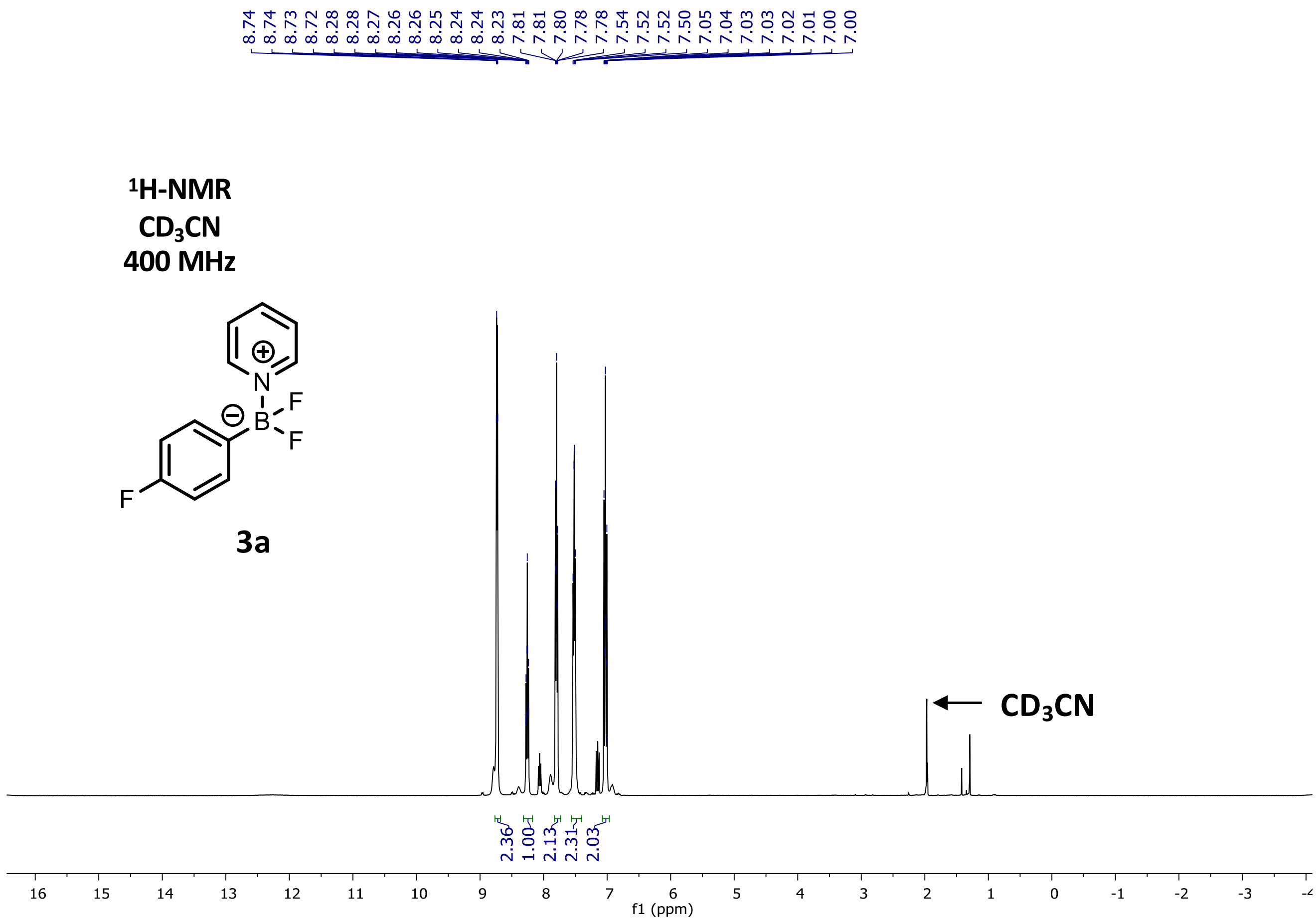

164.08
157.57
124.18
55.60
¹³C{¹H}-NMR
CDCl₃
101 MHz
Cl
N
N
O
2s
CDCl₃
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10

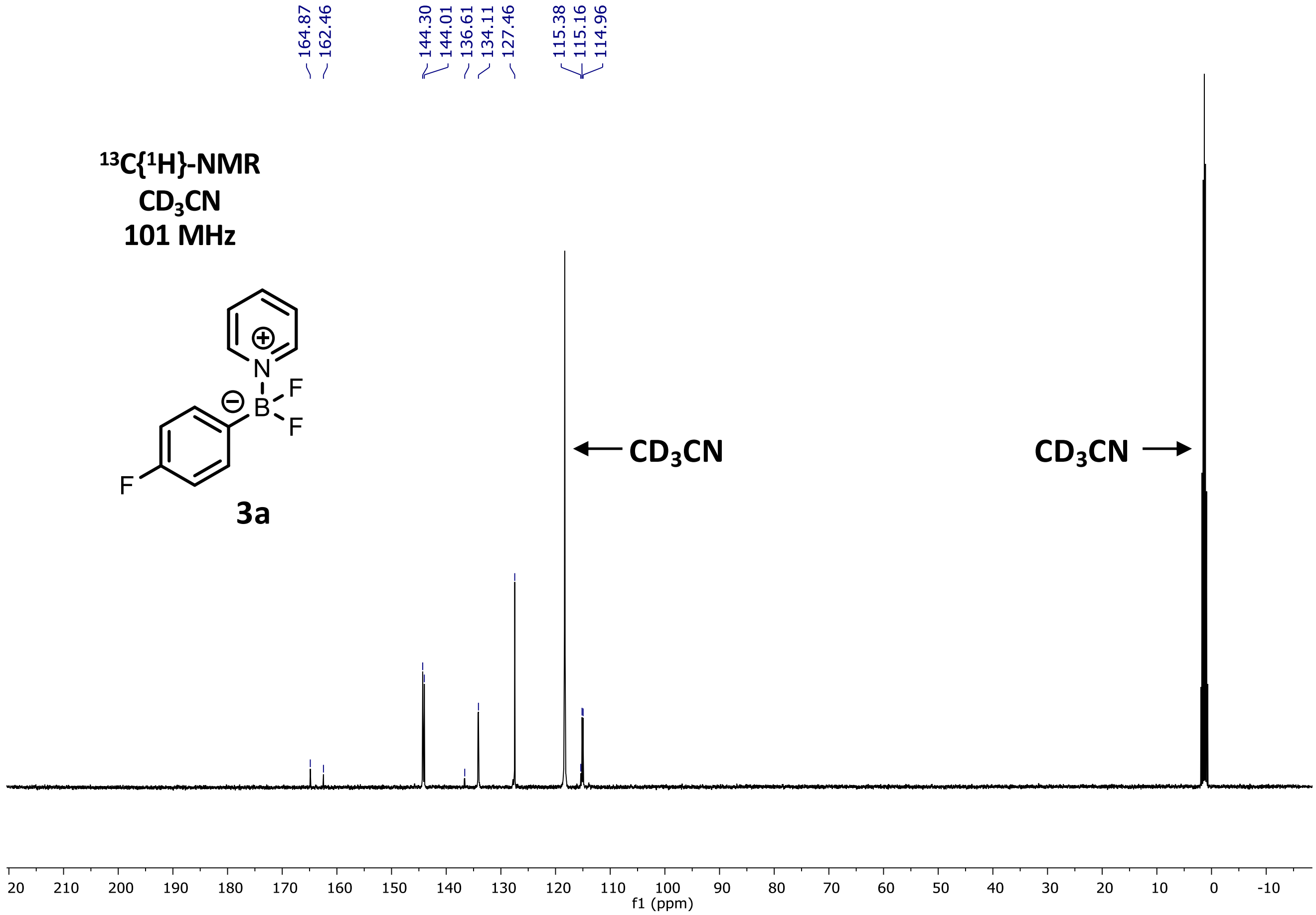

8.32
8.31
7.79
7.77
7.22
7.21
1H-NMR
CDCl3
400 MHz
Cl
N
Cl
2t
CDCl3
1.00
1.00
1.02
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
-4

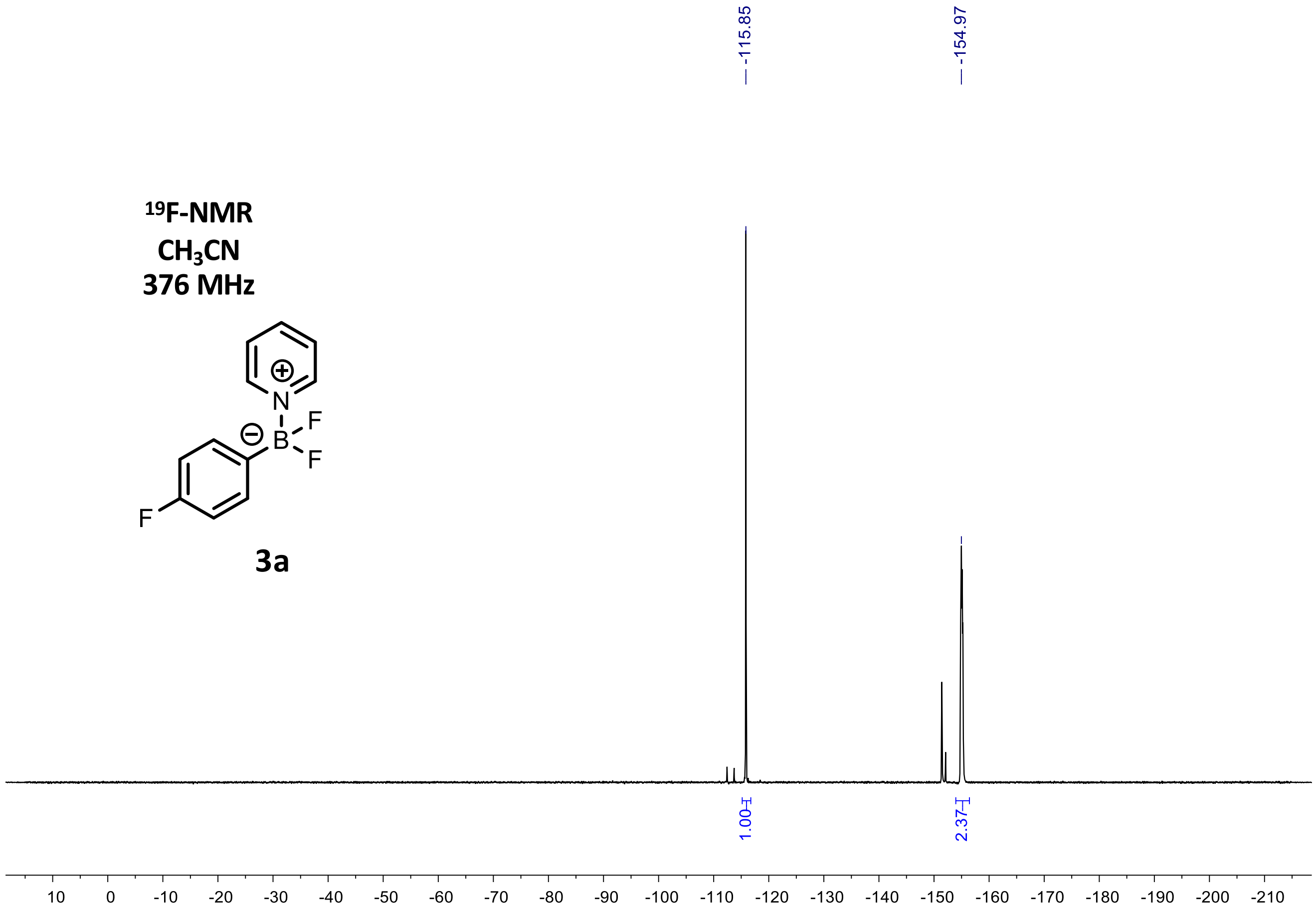

149.47
147.45
138.92
130.86
123.35
13C{1H}-NMR
CDCl3
101 MHz
Cl
N
Cl
2t
CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10

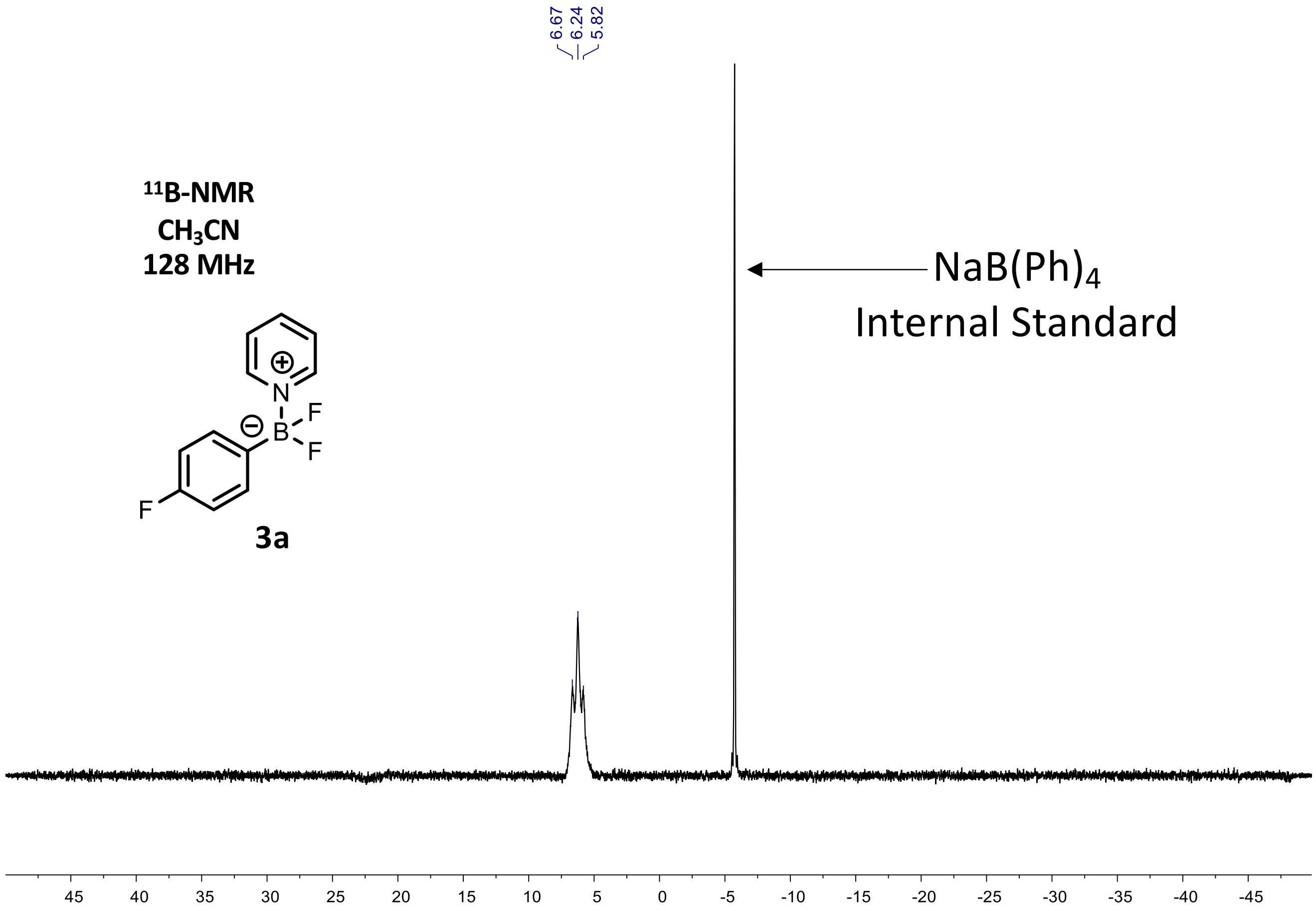

8.07
8.06
8.05
8.05
7.64
7.63
7.62
7.61
6.86
6.85
6.84
6.83
4.02
^{1}H-NMR
$CDCl_3$
400 MHz
Cl
N
O
2u
$CDCl_3$
1.00
1.00
1.02
3.05
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
-4

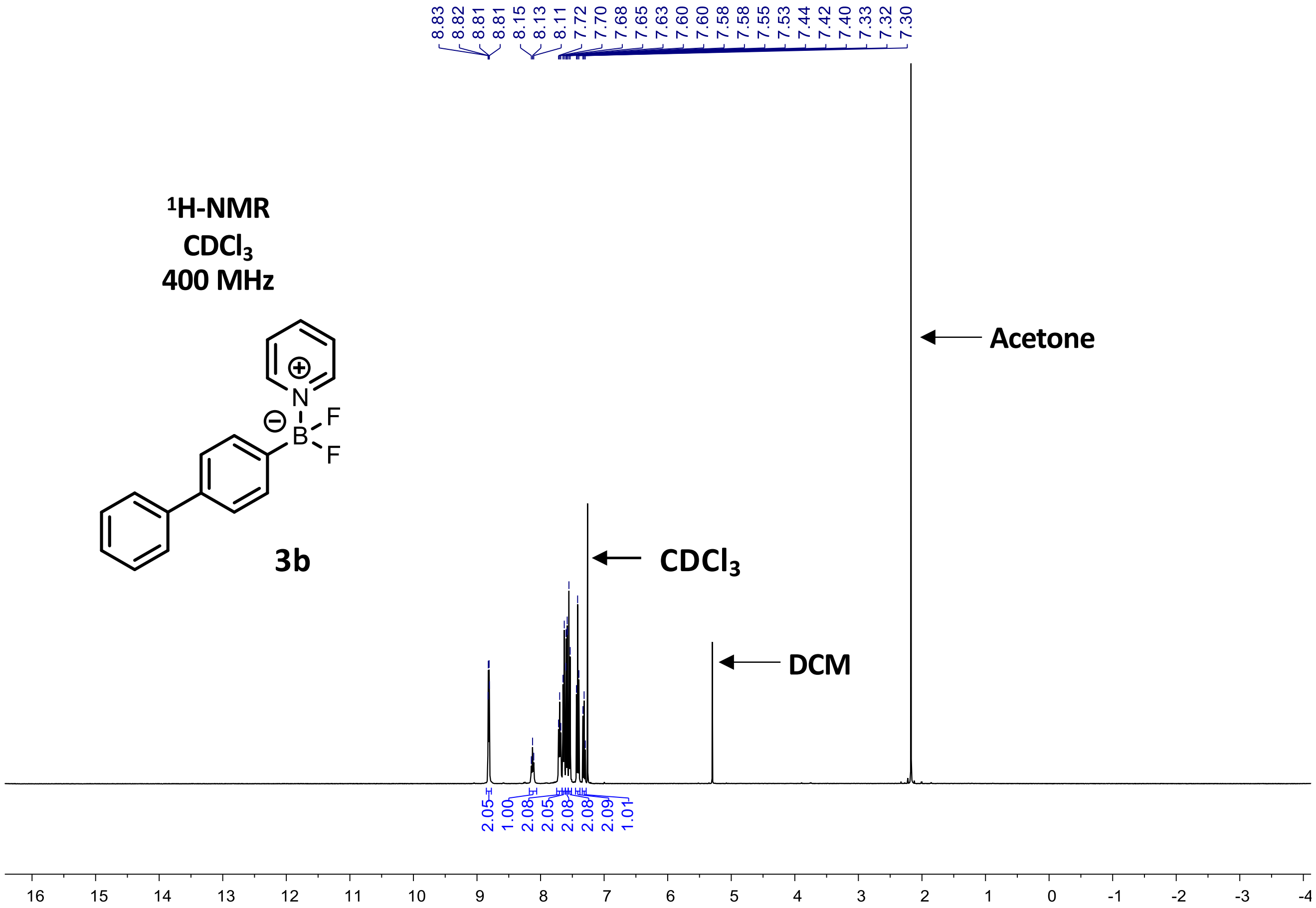

159.42
144.69
138.19
118.25
117.36
54.19
$^{13}C\{^1H\}$-NMR
$CDCl_3$
101 MHz
Cl
N
O
2u
$CDCl_3$
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10

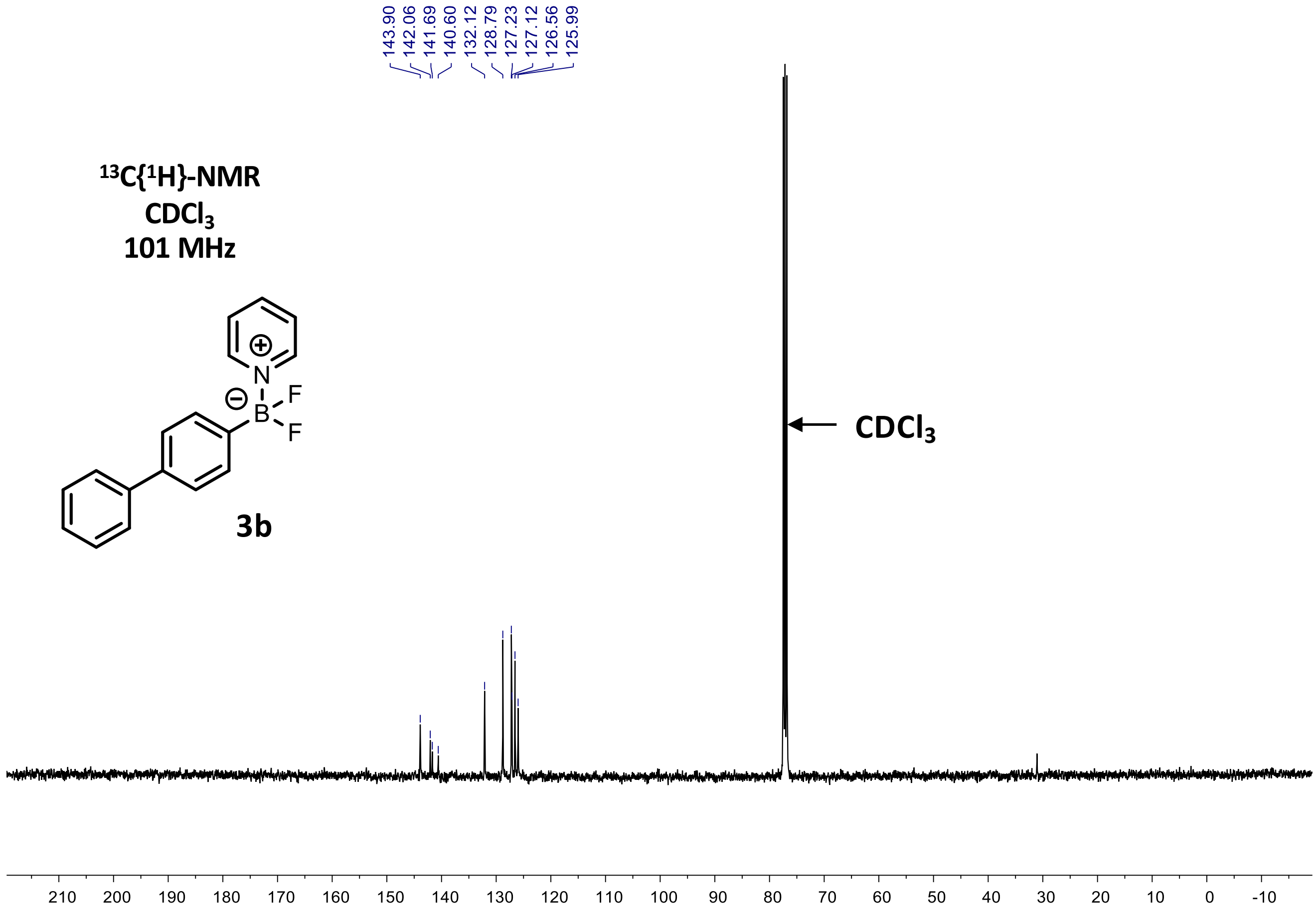

8.74
8.74
8.73
8.72
8.28
8.28
8.27
8.26
8.26
8.25
8.24
8.24
8.23
7.81
7.81
7.80
7.78
7.78
7.54
7.52
7.52
7.50
7.05
7.04
7.03
7.03
7.02
7.01
7.00
7.00
^{1}H-NMR
CD$_3$CN
400 MHz
N
B
F
F
F
3a
CD$_3$CN
2.36
1.00
2.13
2.31
2.03
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)

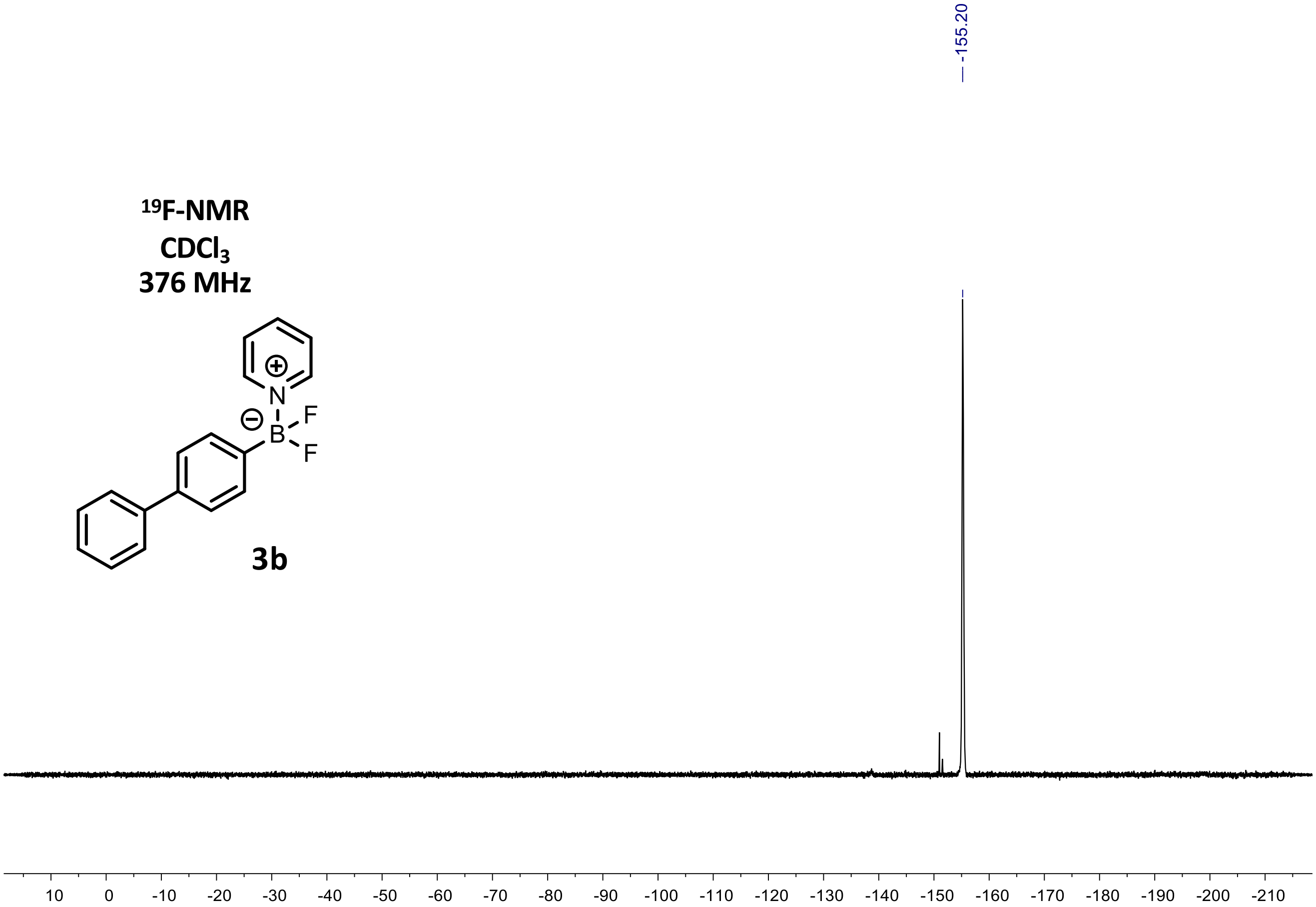

164.87
162.46
144.30
144.01
136.61
134.11
127.46
115.38
115.16
114.96
^{13}C{^{1}H}-NMR
CD$_3$CN
101 MHz
N
B
F
F
F
3a
CD$_3$CN
CD$_3$CN
20
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

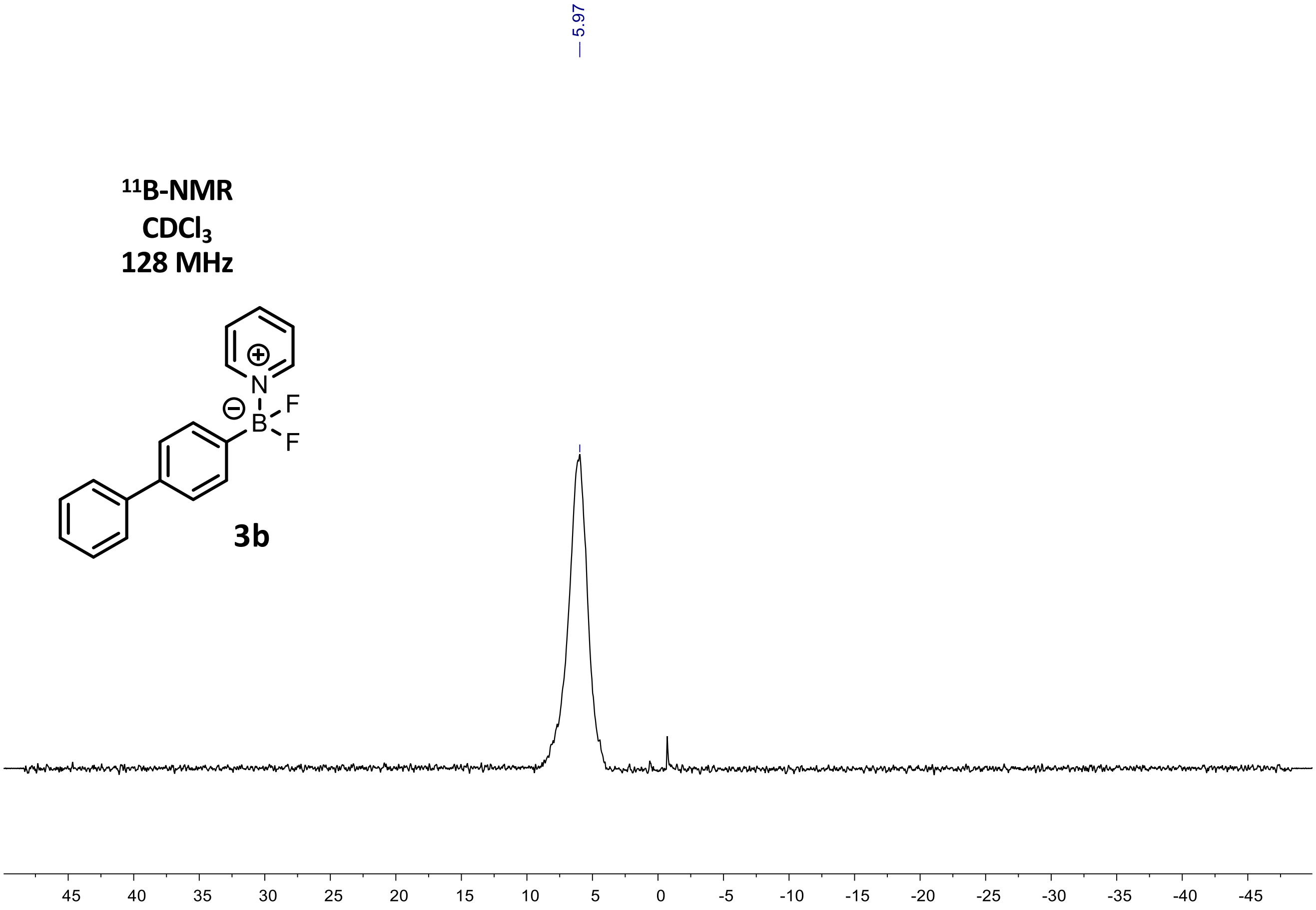

-115.85
-154.97
19F-NMR
CH3CN
376 MHz
N
B
F
F
F
3a
1.00
2.37
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210


6.67
6.24
5.82
11B-NMR
CH3CN
128 MHz
NaB(Ph)4
Internal Standard
N
B
F
F
F
3a
45 40 35 30 25 20 15 10 5 0 -5 -10 -15 -20 -25 -30 -35 -40 -45


8.83
8.82
8.81
8.81
8.15
8.13
8.11
7.72
7.70
7.68
7.65
7.63
7.60
7.60
7.58
7.58
7.55
7.53
7.44
7.42
7.40
7.33
7.32
7.30
^{1}H-NMR
$CDCl_3$
400 MHz
N
B
F
F
3b
Acetone
$CDCl_3$
DCM
2.05
1.00
2.08
2.05
2.08
2.08
2.09
1.01
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
-4


143.90
142.06
141.69
140.60
132.12
128.79
127.23
127.12
126.56
125.99
$^{13}C\{^1H\}$-NMR
$CDCl_3$
101 MHz
N
B
F
F
3b
$CDCl_3$
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10


-155.20
^{19}F-NMR
CDCl$_3$
376 MHz
N
B
F
F
3b
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210


5.97
^{11}B-NMR
CDCl$_3$
128 MHz
N
B
F
F
3b
45
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
-45