Supporting Information

Autocatalytic Friedel-Crafts Reactions of Tertiary Aliphatic Fluorides Initiated by $B(C_6F_5)_3 \cdot H_2O$

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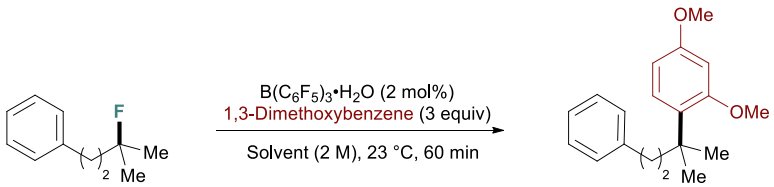
General Information. All defluorination reactions were performed in 2 mL disposable vials under an air atmosphere. All reactions to prepare starting materials were performed in air-dried flasks under nitrogen atmosphere, unless otherwise noted. Purification of reaction products was carried out by flash column chromatography using Merck silica gel (40-63 μm) or by Kugelrohr distillation using standard technique. Analytical thin layer chromatography (TLC) was performed on aluminum sheets pre-coated with silica gel 60 F254 (E. Merck), cut to size. Visualization was accomplished with UV light followed by dipping in a potassium permanganate, *p*-anisaldehyde and/or Seebach's staining solutions and heating.
^{1}H NMR spectra were recorded on a Bruker Avance400 (400 MHz) spectrometer at ambient temperature unless otherwise noted and are reported in ppm using solvent as the internal standard ($CDCl_3$ at 7.26 ppm, C_6D_6 at 7.16 ppm, CD_3NO_2 at 4.33 ppm). Data are reported as: multiplicity (ap = apparent, br = broad, s = singlet, d = doublet, t = triplet, q = quartet, qn = quintet, sx = sextet, sp = septet, oct = octet, non = nonet, m = multiplet), integration and coupling constant(s) in Hz. ^{13}C NMR spectra were recorded on a Bruker Avance400 (100 MHz) spectrometer. Chemical shifts are reported in ppm from tetramethylsilane, with the residual solvent resonance employed as the internal standard ($CDCl_3$ at 77.0 ppm or C_6D_6 at 128.06 ppm). ^{19}F NMR spectra were recorded on a Bruker Avance400 (376 MHz) spectrometer and reported in ppm referred to CF_3COOH (-76.55 ppm) used as the external standard.
Melting points were measured using a *BÜCHI* Melting Point B-540 instrument. GCMS analysis was conducted on a GC System 7820A (G4320) connected to a MSD block 5977E (G7036A) using *Agilent* High Resolution Gas Chromatography Column: PN 19091S – 433UI, HP – 5MS UI, 30m×0.250mm, 0.25 Micron, SN USD 489634H. ESI-MS measurements were conducted on Bruker Micro-TOF instruments.

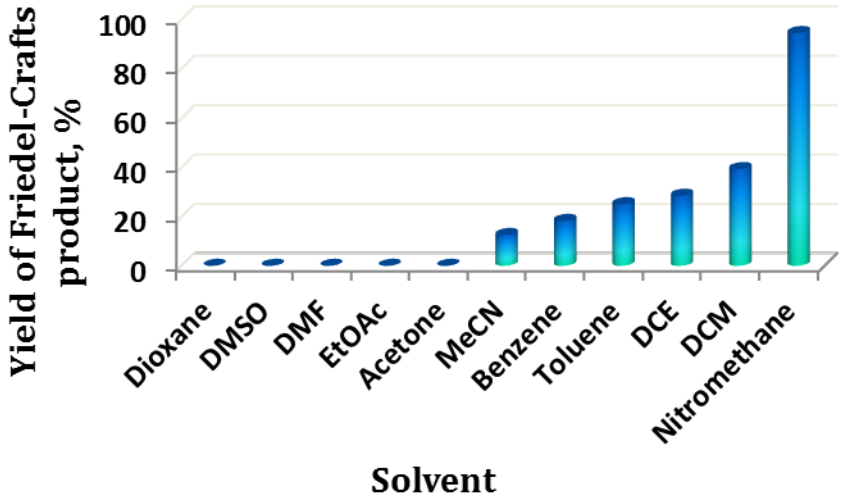
Materials. Unless otherwise noted, all commercial materials were purchased from *Sigma-Aldrich* and used without further purification. The substrates 3-bromo-3-methyl-1-phenylbutane,[1] 3-chloro-3-methyl-1-phenylbutane,[1] 3-methoxy-3-methyl-1-phenylbutane,[2] benzyl 1,1-dimethyl-3-phenylpropyl ether,[3] 1,1-dimethyl-3-phenylpropyl acetate[4] were prepared following a literature procedure. Tris(pentafluorophenyl)borane $B(C_6F_5)_3$ was purchased from *Alfa Aesar* and used under air, without any precaution to exclude moisture or air. $B(C_6F_5)_3$ is known to rapidly hydrate to $B(C_6F_5)_3{\cdot}H_2O$ under these conditions.[5]

Solvent screen. Common organic solvents ware evaluated for the Friedel-Crafts alkylation of 1,3-dimethoxybenzene with tertiary alyphatic fluoride **1a**.

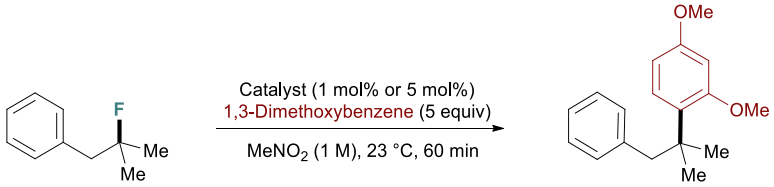


1a **2a**

To a 2 mL disposable vial containing 3-fluoro-3-methyl-1-phenylbutane **1a** (21.0 mg, 0.125 mmol, 1.00 equiv) in nitromethane (62 μL, 2.0 M) was added 1,3-dimethoxybenzene (49.0 μL, 52.0 mg, 0.375 mmol, 3.00 equiv) and *n*-dodecane (8.5 μL, 6.4 mg, 38 μmol, 0.30 equiv) as an internal standard. Finally, tris(pentafluorophenyl)borane hydrate (1.3 mg, 2.5 μmol, 0.020 equiv) was added under stirring. An aliquot was taken after 60 min of reaction time, diluted to 10^{-4} M concentration range with isopropanol and analysed by GCMS. The results are shown below.

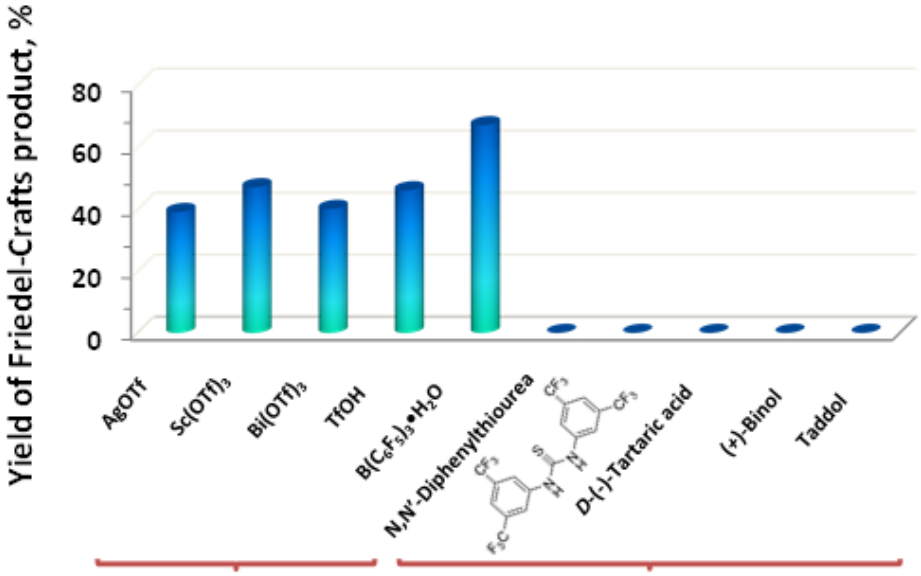


Catalyst Screen. Survey of Lewis acids (1 mol%), Brønsted acids (1 mol%) and H-bond donating catalysts (5 mol%) for Friedel-Crafts alkylation of 1,3-dimethoxybenzene with tertiary aliphatic fluoride **1d**.



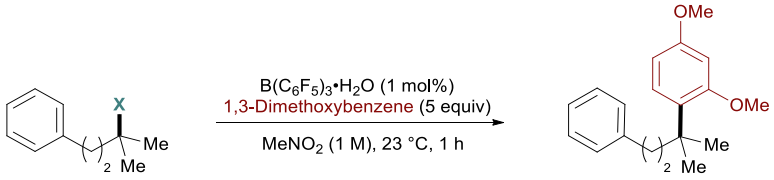
1d **3d**

To a 2 mL disposable vial containing 2-fluoro-2-methyl-1-phenylpropane **1d** (38 mg, 0.25 mmol, 1.0 equiv) in nitromethane (0.25 mL, 1.0 M) was added 1,3-dimethoxybenzene (163 μL, 173 mg, 1.25 mmol, 5.00 equiv). Finally, the catalyst (2.5 μmol, 1.0 mol% for Lewis and Brønsted acids or 12.5 μmol, 5.00 mol% for H-bond donating catalysts; see the table below) was added under stirring. The resulting solution was stirred for 60 min at room temperature. After that time dimethyl sulfoxide (17.8 μL, 19.5 mg, 0.250 mmol, 1.00 equiv) was added to the solution, an aliquot of 50 μL was taken into the NMR tube, filled with $CDCl_3$ and the sample was analyzed by NMR directly by means of the relative integration of the signal of benzylic protons of the product (3.13 ppm) against the internal standard peak (2.66 ppm). The results are shown below.



Catalyst	*Quantity, mg*	*Mol, %*
AgOTf	0.6	1.0
$Sc(OTf)_3$	1.2	1.0
$Bi(OTf)_3$	1.6	1.0
$B(C_6F_5)_3•H_2O$	1.3	1.0
TfOH	0.4	1.0
N,N□-Diphenylthiourea	2.9	5.0
1,3-Bis[3,5-bis(trifluoromethyl)phenyl]thiourea	6.3	5.0
D-(-)-Tartaric acid	1.9	5.0
R-(+)-Binol	3.6	5.0
Taddol	5.8	5.0

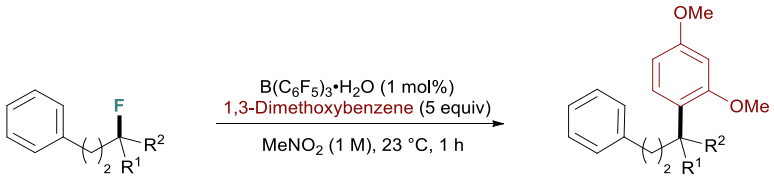
Selectivity with respect to leaving group.



To a 2 mL disposable vial containing a tertiary aliphatic substrate (0.25 mmol, 1.0 equiv) in nitromethane (0.25 mL, 1.0 M) was added 1,3-dimethoxybenzene (163 μL, 173 mg, 1.25 mmol, 5.00 equiv). Finally, tris(pentafluorophenyl)borane hydrate was added (1.3 mg, 2.5 μmol, 1 mol%) under stirring. The resulting solution was stirred for 1 h at room temperature. After that time dimethyl sulfoxide (17.8 μL, 19.5 mg, 0.250 mmol, 1.00 equiv) was added to the solution. An aliquot of 50 μL was taken into an NMR tube, filled with $CDCl_3$ and the sample was analyzed by NMR directly by means of the relative integration of the methylene signal of the starting material against the internal standard peak (2.66 ppm). The results are presented below.

Substrate, leaving group	*Quantity, mg*	*Consumption, %*
F	42	>99
Br	57	<1
Cl	46	<1
OH	41	<1
OMe	45	<1
OBn	64	<1
OAc	52	<1

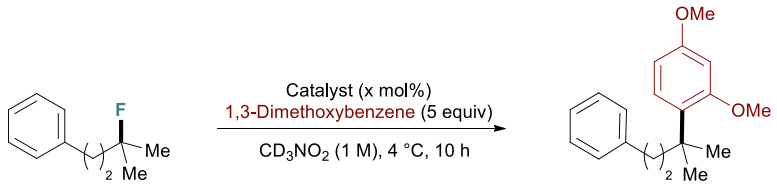
Selectivity with respect to substitution pattern.



To a 2 mL disposable vial containing an aliphatic fluoride (0.25 mmol, 1.0 equiv) in nitromethane (0.25 mL, 1.0 M) was added 1,3-dimethoxybenzene (163 μL, 173 mg, 1.25 mmol, 5.00 equiv). Finally, tris(pentafluorophenyl)borane hydrate was added (1.3 mg, 2.5 μmol, 1 mol%) under stirring. The resulting solution was stirred for 1 h at room temperature. After that time 1,3,5-trimethoxybenzene (42.0 mg, 0.250 mmol, 1.00 equiv) was added to the solution. An aliquot of 50 μL was taken into an NMR tube, filled with $CDCl_3$ and the sample was analyzed by ^{1}H NMR directly by means of the relative integration of the methylene signal of the starting material against the internal standard peak (2.66 ppm). The results are presented below.

R^1	*R^2*	*Quantity, mg*	*Consumption, %*
Me	Me	42	>99
Me	H	38	<1
H	H	35	<1
F	H	39	<1
F	Me	43	<1

Time-dependent NMR experiments:

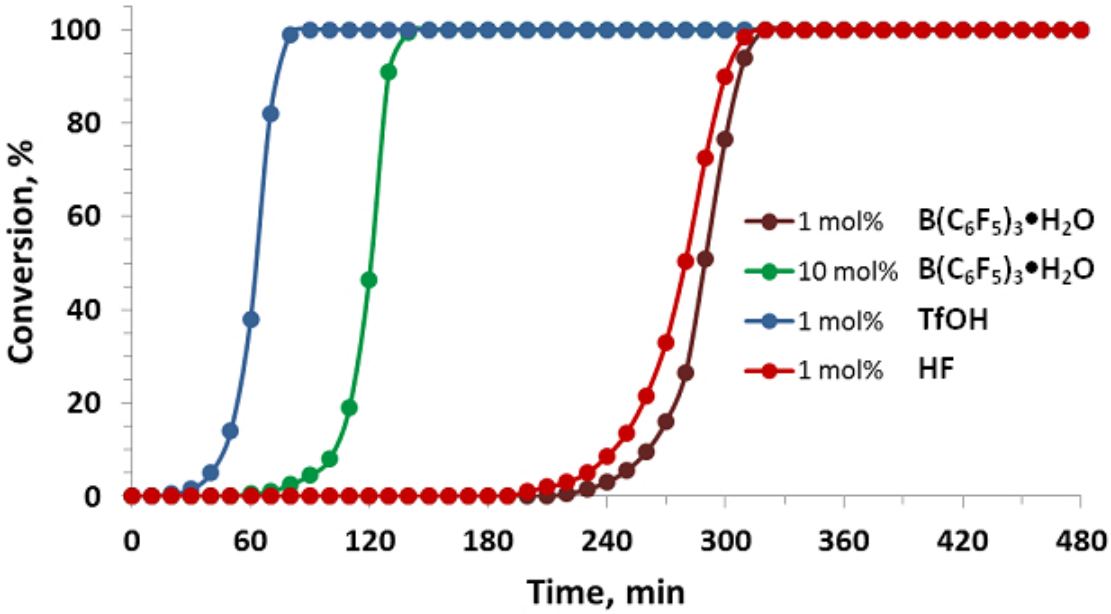


1a **2a**

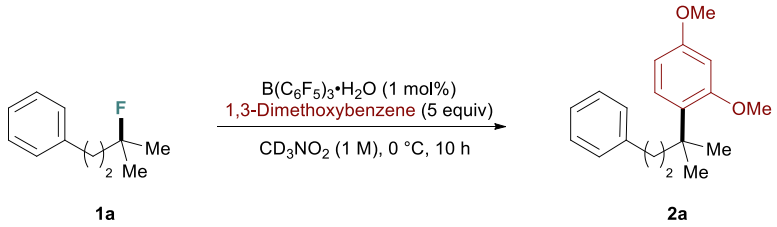
An oven dried NMR tube was charged with 3-fluoro-3-methyl-1-phenylbutane **1a** (42 mg, 0.25 mmol, 1 equiv), nitromethane-d^3 (0.25 mL, 1.0 M) and 1,3-dimethoxybenzene (163 μL, 173 mg, 1.25 mmol, 5.00 equiv). The tube was precooled to 0 °C and the catalyst was added at this temperature. The sample was introduced into the NMR spectrometer at 4 °C and the reaction was monitored at that temperature.

Spectra were recorded every 10 min for 10 hours. Each spectrum was then analysed to calculate the conversion of the starting material into the Friedel-Crafts products. The disappearance of a multiplet of the starting material (2.90-2.76 ppm; 2H) was monitored, along with the appearance of a multiplet for the desired compound (2.40-2.33 ppm; 2H). Integrating both signals, a conversion percentage was calculated for each spectrum and plotted against time. The results are presented below.

Entry	*Catalyst*	*Loading, mg*	*Mol %*
1	$B(C_6F_5)_3 \cdot H_2O$	1.3	1.0
2	$B(C_6F_5)_3 \cdot H_2O$	13.3	10.0
3	TfOH	0.4	1.0
4	HF (48% in H_2O)	0.1	1.0



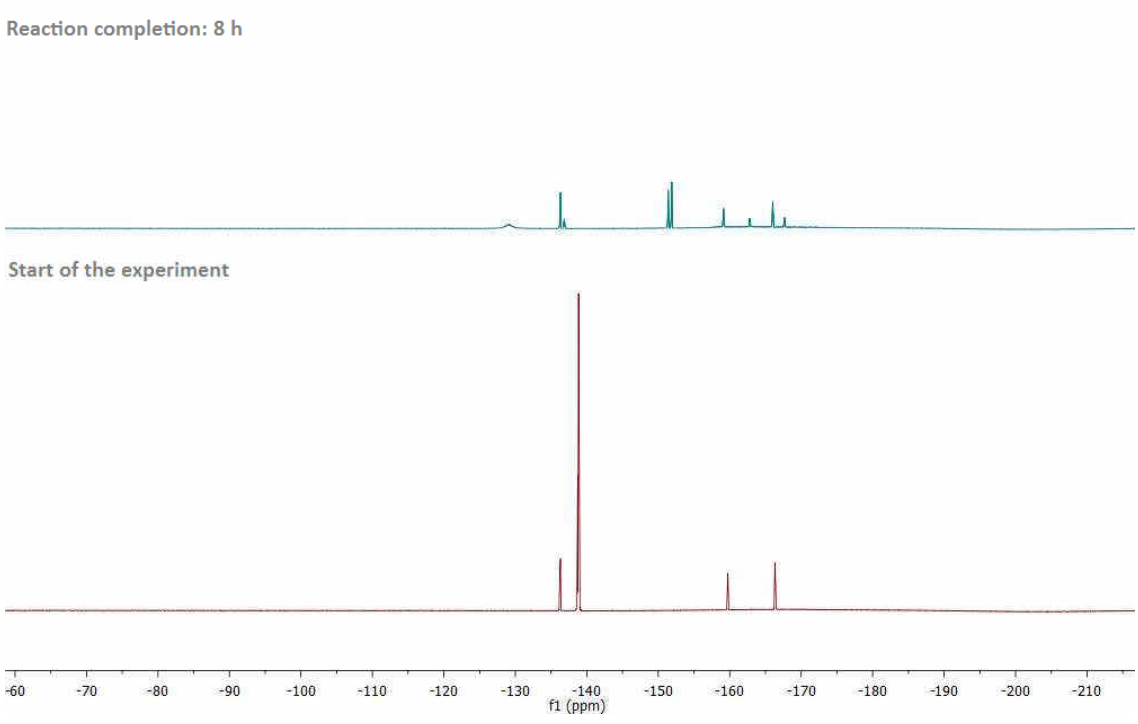
Study on catalyst activity:



1a **2a**

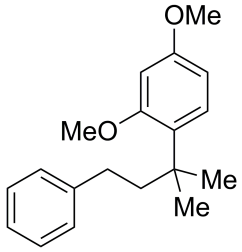
An oven dried NMR tube was charged with 3-fluoro-3-methyl-1-phenylbutane **1a** (42 mg, 0.25 mmol, 1 equiv), nitromethane-d^3 (0.25 mL, 1.0 M) and 1,3-dimethoxybenzene (163 μL, 173 mg, 1.25 mmol, 5.00 equiv). The tube was precooled to 0 °C and tris(pentafluorophenyl)borane hydrate (1.3 mg, 2.5 μmol, 1 mol%) was added at this temperature. The sample was introduced into the NMR spectrometer at 0 °C and the reaction was monitored by ^{19}F NMR. Throughout the reaction, no peaks corresponding to $[FB(C_6F_5)_3]^-$ were detected in the region -185 to -192 ppm.[6]

Chemical shift, ppm	***Resonance***
-136.3	*ortho*-F ($B(C_6F_5)_3•H_2O$)
-138.9	*F* (**1a**)
-159.7	*para*-F ($B(C_6F_5)_3•H_2O$)
-166.3	*meta*-F ($B(C_6F_5)_3•H_2O$)



Isolation of Friedel-Crafts product 2a (eq 1).

To a 2 mL disposable vial containing 3-fluoro-3-methyl-1-phenylbutane **1a** (42 mg, 0.25 mmol, 1 equiv) in nitromethane (0.25 mL, 1.0 M) was added 1,3-dimethoxybenzene (163 μL, 173 mg, 1.25 mmol, 5 equiv), followed by $B(C_6F_5)_3 \bullet H_2O$ (1.3 mg, 2.5 μmol, 1.0 mol%). The vial was capped and the mixture allowed to stir for 1 h at room temperature. After that time, the reaction mixture was filtered through a short bed of Celite and the solvent was removed under reduced pressure. The residue was purified by Kugelrohr distillation to deliver 64 mg (90%) of the desired product as a colorless oil (>20:1 ratio of regioisomers).



3-(2,4-Dimethoxyphenyl)-3-methyl-1-phenylbutane **2a**

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30-7.25 (m, 2H), 7.23-7.10 (m, 4H), 6.53 (d, J = 2.5 Hz, 1H), 6.50 (dd, J = 8.4 Hz, 2.5 Hz, 1H), 3.87 (s, 3H), 3.86 (s, 3H), 2.34-2.26 (m, 2H), 2.19-2.12 (m, 2H), 1.43 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.5, 159.2, 144.0, 128.8, 128.4, 128.3, 128.2, 125.4, 103.4, 99.6, 55.4, 55.1, 43.3, 37.9, 32.2, 28.8. **HRMS** (ESMS) for $C_{19}H_{24}O_2$ + H: calcd. 285.1855; found 285.1849.

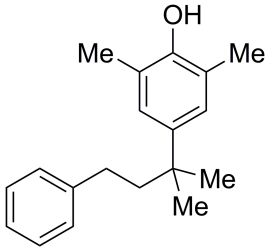
Defluorinative alkylation of arenes with 3-fluoro-3-methyl-1-phenylbutane 1a.

General procedure:

Condition A. To a 2 mL disposable vial containing 3-fluoro-3-methyl-1-phenylbutane **1a** (42 mg, 0.25 mmol, 1 equiv) in nitromethane (0.25 mL, 1.0 M) was added the arene nucleophile (0.75 mmol, 3 equiv), followed by $B(C_6F_5)_3 \bullet H_2O$ (1.3 mg, 2.5 μmol, 1.0 mol%). The vial was capped and the mixture allowed to stir for 1 h at 23 ^{o}C. After completion, the reaction mixture was directly purified by flash chromatography on SiO_2.

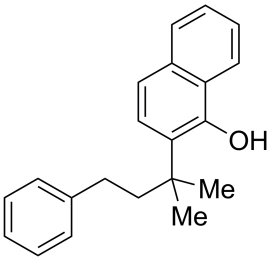
Condition B. To a 2 mL disposable vial containing 3-fluoro-3-methyl-1-phenylbutane **1a** (84 mg, 0.50 mmol, 1 equiv) in nitromethane (0.50 mL, 1.0 M) was added the arene nucleophile (1.50 mmol, 3 equiv), followed by $B(C_6F_5)_3 \bullet H_2O$ (2.6 mg, 5.0 μmol, 1.0 mol%). The vial was capped and the mixture allowed to stir for 1 h at the indicated temperature. After completion, the reaction mixture was filtered through a short bed of Celite and the solvent was removed under reduced pressure. The residue was purified by Kugelrohr distillation.

Spectral data for all alkylation products (Nucleophile Scope):



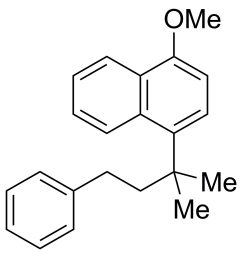
3-(3,5-Dimethyl-4-hydroxyphenyl)-3-methyl-1-phenylbutane **2b**

Synthesized according to *general procedure B* at room temperature using 2,6-dimethylphenol (183 mg, 1.50 mmol). Isolated (127 mg, 95%) as a colorless oil after Kugelrohr distillation (> 20:1 ratio of regioisomers).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26-7.21 (m, 2H), 7.17-7.07 (m, 3H), 6.98 (s, 2H), 4.49-4.41 (br, 1H), 2.40-2.34 (m, 2H), 2.26 (s, 6H), 1.91-1.84 (m, 2H), 1.33 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 150.1, 143.5, 141.0, 128.4, 126.4, 125.6, 122.5, 46.8, 37.3, 31.5, 29.3, 16.4. **HRMS** (ESMS) for $C_{19}H_{24}O$: calcd. 268.1827; found 268.1822.



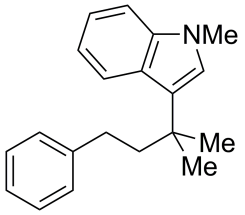
3-(2-Hydroxynaphthyl)-3-methyl-1-phenylbutane **2c**

Synthesized according to *general procedure A* using 1-naphthol (108 mg, 0.750 mmol). Isolated (63 mg, 87%) as a yellowish oil after column chromatography (15% Et_2O in Petroleum ether). R_f = 0.62 (20% Et_2O in Petroleum ether). The product degraded after several days at -20 °C. The presence of minor impurities prevented definitive descrimination between *ortho* and *para* regioisomeric structures by 2D NMR analysis, as well as appropriate determination of the ratio of regioisomers. However, the *ortho* substitution pattern was assigned as a mojor regioisomer by analogy with literature ^{13}C data for 1-methyl-naphth-4-ol[7] and 2-methyl-naphthalen-1-ol.[8]
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (d, *J* = 8.0 Hz, 1H), 7.91 (d, *J* = 8.0 Hz, 1H), 7.61-7.52 (m, 4H), 7.31 (t, *J* = 7.2 Hz, 2H), 7.25-7.17 (m, 3H), 5.64-5.52 (br, 1H), 2.50-2.43 (m, 2H), 2.39-2.31 (m, 2H), 1.67 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 149.0, 143.3, 133.4, 128.4, 128.3, 128.0, 127.8, 126.5, 125.6, 125.5, 125.0, 120.3, 119.7, 44.2, 38.3, 32.1, 29.1. **HRMS** (ESMS) for $C_{21}H_{22}O$ - H: calcd. 289.1592; found 289.1587.



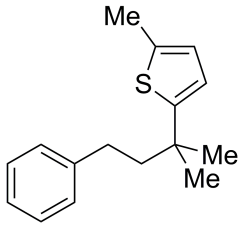
3-(4-Methoxynaphthyl)-3-methyl-1-phenylbutane **2d**

Synthesized according to *general procedure A* using 1-methoxynaphthalene (109 μL, 119 mg, 0.750 mmol). Isolated (33 mg, 43%) of a yellow oil (major regioisomer present in quantities greater than 95%) after column chromatography (Petroleum ether). R_f = 0.15 (Petroleum ether). The presence of minor impurities prevented definitive descrimination between *ortho* and *para* regioisomeric structures by 2D NMR analysis. The *para* substitution pattern was assigned by contrasting with **2c** and in accordance with literature ^{13}C data for 1-methoxy-4-methyl-naphthalene.[9]
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.46 (dd, *J* = 8.1 Hz, 1.5 Hz, 1H), 8.42 (dd, *J* = 8.1 Hz, 1.8 Hz, 1H), 7.56-7.47 (m, 2H), 7.42 (d, *J* = 8.3 Hz, 1H), 7.23-7.18 (m, 2H), 7.12 (t, *J* = 7.2 Hz, 1H), 7.03-6.97 (m, 2H), 6.78 (d, *J* = 8.3 Hz, 1H), 4.02 (s, 3H), 2.41-2.36 (m, 2H), 2.29-2.22 (m, 2H), 1.65 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 154.3, 143.3, 135.8, 132.8, 128.4, 128.3, 127.0, 126.1, 125.6, 125.5, 124.8, 124.4, 123.2, 103.0, 55.5, 45.2, 39.3, 32.0, 30.8. **HRMS** (ES) for $C_{22}H_{24}O$: calcd. 304.1827; found 304.1822.



3-(1-Methyl-1*H*-indol-3-yl)-3-methyl-1-phenylbutane **2e**

Synthesized according to *general procedure B* at 90 °C using 1-methylindole (187 μL, 197 mg, 1.50 mmol). Isolated (115 mg, 83%) as a colorless oil after Kugelrohr distillation (Major regioisomer present in quantities greater than 95%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (d, *J* = 8.1 Hz, 1H), 7.33 (d, *J* = 8.1 Hz, 1H), 7.26-7.19 (m, 3H), 7.16-7.06 (m, 4H), 6.83 (s, 1H), 3.77 (s, 3H), 2.44-2.38 (m, 2H), 2.18-2.12 (m, 2H), 1.49 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 143.6, 138.0, 128.5, 128.3, 126.5, 125.7, 125.5, 123.2, 121.3, 121.2, 118.5, 109.5, 45.2, 35.2, 32.7, 31.9, 29.1. **HRMS** (ESMS) for $C_{20}H_{23}N$: calcd. 277.1830; found 277.1825.

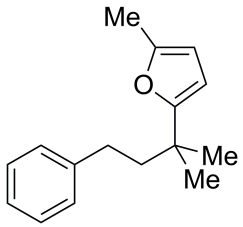


3-Methyl-3-(5-methylthienyl)-1-phenylbutane **2f**

Synthesized according to *general procedure A* using 2-methylthiophene (73 μL, 74 mg, 0.75 mmol). Isolated (51 mg, 83%) as a colorless oil (85:15 mixture of

regioisomers) after column chromatography (Petroleum ether). R_f = 0.50 (Petroleum ether).

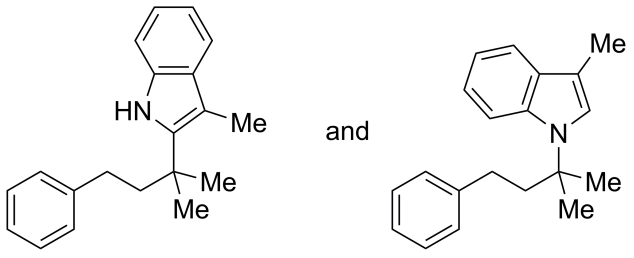
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34-7.27 (m, 2H), 7.24-7.16 (m, 3H), 7.00 (s, 0.15H), 6.77 (d, *J* = 6.1, 0.14H), 6.67 (d, *J* = 3.4, 0.85H), 6.64-6.60 (m, 0.85H), 2.57-2.45 (m, 5H), 2.00-1.90 (m, 2H), 1.45 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) major regioisomer δ 153.2, 143.1, 137.2, 128.5, 128.4, 125.7, 124.5, 122.1, 47.9, 37.8, 31.5, 30.2, 15.5. **HRMS** (ESMS) for $C_{16}H_{20}S$+H: calcd. 245.1364; found 245.1358.



3-Methyl-3-(5-methylfuryl)-1-phenylbutane **2g**

Synthesized according to *general procedure A* using 2-methylfuran (68 μL, 62 mg, 0.75 mmol) and 250 μL of a nitromethane/1,1,1,3,3,3-hexafluoro-2-propanol solution as solvent (1 to 1 v/v). Isolated 18 mg (32%) as a colorless oil (>20:1 ratio of regioisomers) after column chromatography (Petroleum ether). R_f = 0.42 (Petroleum ether).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29-7.24 (m, 2H), 7.19-7.13 (m, 3H), 5.90 (d, *J* = 3.0 Hz, 1H), 5.88-5.84 (dt, *J* = 3.0 Hz, 1.0 Hz, 1H), 2.48-2.42 (m, 2H), 2.28 (d, *J* = 1.0 Hz, 3H), 1.92-1.86 (m, 2H), 1.31 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 161.0, 150.3, 143.3, 128.5, 128.4, 125.7, 105.6, 104.1, 44.3, 36.0, 31.5, 27.1, 13.7. **HRMS** (ESMS) for $C_{16}H_{20}O$: calcd. 228.1514; found 228.1509.



2h$_a$ **2h$_b$**

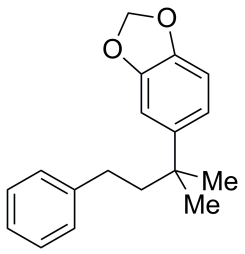
3-(3-Methyl-1*H*-indol-2-yl)-3-methyl-1-phenylbutane **2h$_a$**
3-(3-Methyl-1*H*-indol-1-yl)-3-methyl-1-phenylbutane **2h$_b$**

Synthesized according to *general procedure B* at 90 °C using 3-methylindole (197 mg, 1.50 mmol). By Kugelrohr distillation, the fraction containing the nucleophile was distilled off and the following fraction containing the desired product was collected. The obtained colorless oil was additionally subjected to column chromatography (4% Et_2O in Petroleum ether) to give 9 mg of *N*-alkylated product (R_f = 0.30, 4% Et_2O in Petroleum ether) and 97 mg of *C*-alkylated product (3

regioisomers were detected by GCMS; major regioisomer present in quantities greater than 90%) (R_f = 0.15, 4% Et_2O in Petroleum ether), both as colorless oils. Total yield 108 mg, 76%.

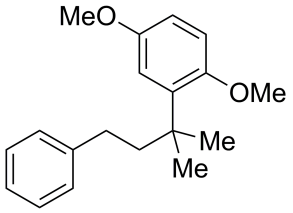
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15-6.87 (m, 10H), 2.62-2.40 (m, 5H), 2.27-2.11 (m, 2H), 1.69-1.56 (m, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) major regioisomer δ 142.8, 139.9, 134.1, 130.6, 128.5, 128.4, 125.8, 121.1, 119.1, 118.0, 110.3, 106.0, 45.2, 36.5, 31.7, 28.1, 10.4. **HRMS** (ESMS) for $C_{20}H_{23}N$: calcd. 277.1830; found 277.1825.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65-7.58 (m, 2H), 7.23-7.11 (m, 5H), 7.08 (s, 1H), 7.00 (d, *J* = 7.4 Hz, 2H), 2.41-2.33 (m, 5H), 2.29-2.22 (m, 2H), 1.76 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 142.1, 135.4, 130.5, 128.5, 128.4, 125.9, 124.0, 121.0, 119.3, 118.4, 113.0, 109.2, 58.2, 43.3, 30.5, 28.6, 9.8. **HRMS** (ESMS) for $C_{20}H_{23}N$: calcd. 277.1830; found 277.1825.



5-(2-Methyl-4-phenylbutan-2-yl)benzo[d][1,3]dioxole **2i**

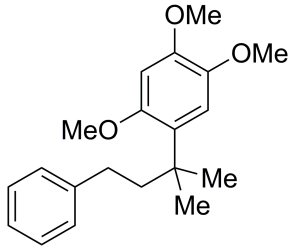
Synthesized according to *general procedure A* using 1,3-benzodioxole (86 μL, 92 mg, 0.75 mmol) and 250 μL of the nitromethane/1,1,1,3,3,3-hexafluoro-2-propanol solution as a solvent (1 to 1 v/v). Isolated (40 mg, 60%) as a colorless oil (92% of the desired regioisomer together with the minor regioisomer and bisalkylation product) after column chromatography; major regioisomer present in quantities greater than 95%). (Petroleum ether). R_f = 0.12 (Petroleum ether).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30-7.23 (m, 2.18H, mixture), 7.20-7.09 (m, 3.14H, mixture); 6.93 (d, *J* = 1.9 Hz, 0.92H, major); 6.86 (dd, *J* = 8.2 Hz, 1.9 Hz, 0.92H, major), 6.84-6.75 (m, 1.15H, mixture), 5.96 (s, 1.85H, major), 5.93 (s, 0.15, mixture), 2.46-2.31 (m, 2.20H, mixture), 2.11-2.03 (m, 0.22H, mixture), 1.95-1.86 (m, 2.20H, mixture), 1.43 (s, 0.41H, minor), 1.36 (s, 5.70, mixture), 1.29 (s, 0.41, minor); **^{13}C NMR** (100 MHz, $CDCl_3$) major regioisomer δ 147.8, 145.4, 143.4, 143.2, 128.4, 128.3, 125.7, 118.8, 107.9, 106.9, 100.9, 47.0, 38.0, 31.5, 29.4. **HRMS** (ESMS) for $C_{18}H_{20}O_2$: calcd. 268.1463; found 268.1458.



3-(2,5-Dimethoxyphenyl)-3-methyl-1-phenylbutane **2j**

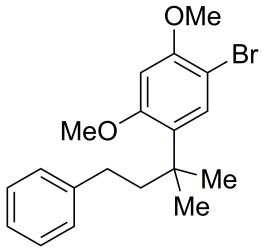
Synthesized according to *general procedure B* at room temperature using 2,6-dimethylphenol (346 mg, 2.50 mmol) and 500 μL of the nitromethane/1,1,1,3,3,3-

hexafluoro-2-propanol solution as a solvent (1 to 1 volume ratio). Isolated (80 mg, 56%) as a colorless oil after Kugelrohr distillation.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27-7.21 (m, 2H), 7.17-7.09 (m, 3H), 6.91 (d, *J* = 3.1 Hz, 1H), 6.83 (d, *J* = 8.8 Hz, 1H), 6.73 (dd, *J* = 8.8 Hz, 3.1 Hz, 1H), 3.81 (s, 3H), 3.80 (s, 3H), 2.33-2.27 (m, 2H), 2.19-2.14 (m, 2H), 1.41 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 153.4, 153.0, 143.8, 137.8, 128.4, 128.3, 125.4, 115.7, 112.1, 110.0, 55.7, 55.6, 43.1, 38.6, 32.1, 28.5. **HRMS** (ES) for $C_{19}H_{24}O_2$: calcd. 284.1776; found 284.1771.



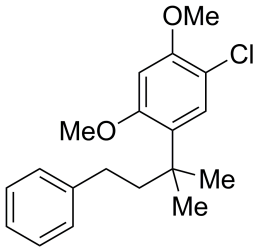
3-(2,4,5-Trimethoxyphenyl)-3-methyl-1-phenylbutane **2k**

Synthesized according to *general procedure B* at room temperature using 1,2,4-trimethoxybenzene (252 mg, 1.50 mmol). Isolated (132 mg, 84%) as a colorless oil after Kugelrohr distillation (>20:1 ratio of regioisomers).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28-7.22 (m, 2H), 7.18-7.09 (m, 3H), 6.89 (s, 1H), 6.57 (s, 1H), 3.92 (s, 3H), 3.89 (s, 3H), 3.84 (s, 3H), 2.35-2.29 (m, 2H), 2.17-2.11 (m, 2H), 1.43 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 152.8, 147.8, 143.8, 142.3, 128.4, 128.2, 128.0, 125.4, 113.4, 98.6, 57.1, 56.2, 56.0, 43.3, 38.1, 32.1, 28.7. **HRMS** (ES) for $C_{20}H_{26}O_3$: calcd. 314.1882; found 314.1876.



3-(5-Bromo-2,4-dimethoxyphenyl)-3-methyl-1-phenylbutane **2l**

Synthesized according to *general procedure B* at room temperature using 1-bromo-2,4-dimethoxybenzene (326 mg, 1.50 mmol). Isolated (137 mg, 75%) as a colorless oil that turns into a waxy solid after Kugelrohr distillation (>20:1 ratio of regioisomers).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (s, 1H), 7.28-7.21 (m, 2H), 7.15 (t, *J* = 7.4 Hz, 1H), 7.11-7.07 (m, 2H), 6.49 (s, 1H), 3.92 (s, 3H), 3.86 (s, 3H), 2.30-2.25 (m, 2H), 2.14-2.08 (m, 2H), 1.39 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.7, 154.9, 143.6, 132.0, 130.2, 128.4, 128.3, 125.5, 101.5, 97.5, 56.5, 55.5, 43.0, 38.0, 32.1, 28.6. **HRMS** (ES) for $C_{19}H_{23}BrO_2$+H: calcd. 363.0960; found 363.0954.
m.p. = 71-73 °C.



3-(5-Chloro-2,4-dimethoxyphenyl)-3-methyl-1-phenylbutane **2m**

Synthesized according to *general procedure B* at room temperature using 1-chloro-2,4-dimethoxybenzene (259 mg, 1.50 mmol). Isolated (124 mg, 78%) as a colorless oil after Kugelrohr distillation (>20:1 ratio of regioisomers).

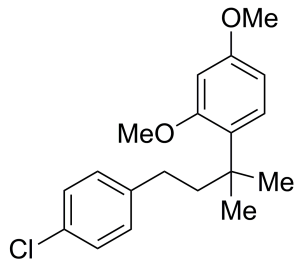
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26-7.20 (m, 3H), 7.13 (t, *J* = 7.3 Hz, 1H), 7.09-7.05 (m, 2H), 6.50 (s, 3H), 3.92 (s, 3H), 3.85 (s, 3H), 2.29-2.24 (m, 2H), 2.13-2.07 (m, 2H), 1.37 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.0, 153.9, 143.6, 129.7, 129.2, 128.4, 128.3, 125.5, 113.0, 97.7, 56.4, 55.6, 43.0, 38.0, 32.1, 28.6. **HRMS** (ES) for $C_{19}H_{23}ClO_2$: calcd. 318.1387; found 318.1381.

Defluorinative alkylation of 1,3-dimethoxybenzene

General procedure A:

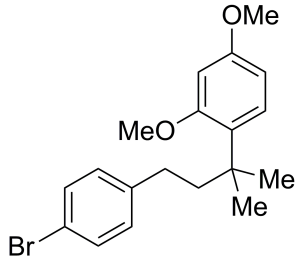
To a 2 mL disposable vial containing tertiary aliphatic fluoride (0.50 mmol, 1 equiv) and 1,3-dimethoxybenzene (196 μL, 207 mg, 1.50 mmol, 3 equiv) in nitromethane (0.50 mL, 1.0 M) was added $B(C_6F_5)_3 \bullet H_2O$ (2.6 mg, 5.0 μmol, 1.0 mol%) under stirring. The mixture was allowed to stir for 1 h at room temperature. After completion, the reaction mixture was filtered through a short bed of Celite and the solvent was removed under reduced pressure. The residue was purified by Kugelrohr distillation.

Spectral data for all alkylation products (Substrate Scope)



1-(4-Chlorophenyl)-3-(2,4-dimethoxyphenyl)-3-methylbutane **3b**

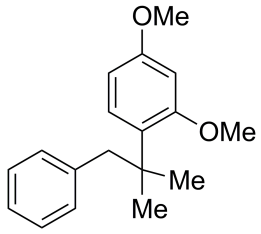
Synthesized according to *general procedure A* using 1-(4-chlorophenyl)-3-fluoro-3-methylbutane (100 mg, 0.500 mmol). Isolated (121 mg, 76%) as a yellow oil after Kugelrohr distillation (>20:1 ratio of regioisomers).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26-7.18 (m, 3H), 7.04 (d, *J* = 8.3 Hz, 2H), 6.54 (d, *J* = 2.4 Hz, 1H), 6.50 (dd, 8.4 Hz, 2.4 Hz, 1H), 3.86 (s, 6H), 2.32-2.25 (m, 2H), 2.19-2.11 (m, 2H), 1.44 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.4, 159.3, 142.3, 131.0, 129.7, 128.4, 128.3, 128.2, 103.5, 99.5, 55.3, 55.0, 43.1, 37.9, 31.5, 28.8. **HRMS** (ESMS) for $C_{19}H_{23}ClO_2$: calcd. 318.1387; found 318.1381.



1-(4-Bromophenyl)-3-(2,4-dimethoxyphenyl)-3-methylbutane **3c**

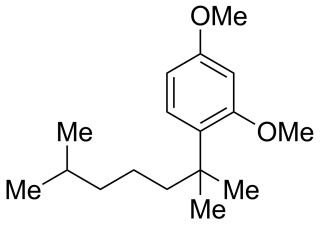
Synthesized according to *general procedure A* using 1-(4-bromophenyl)-2-fluoro-2-methylbutane (123 mg, 0.500 mmol). Isolated (148 mg, 81%) as a colorless oil after Kugelrohr distillation (>20:1 ratio of regioisomers).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (d, *J* = 8.4 Hz, 2H), 7.19 (d, *J* = 8.5 Hz, 1H), 6.98 (d, *J* = 8.4 Hz, 2H), 6.52 (d, *J* = 2.5 Hz, 1H), 6.49 (dd, *J* = 8.5 Hz, 2.5 Hz, 1H), 3.84 (s, 6H), 2.28-2.12 (m, 2H), 2.16-2.09 (m, 2H), 1.42 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.4, 159.3, 142.8, 131.2, 130.2, 128.4, 128.2, 119.0, 103.5, 99.5, 55.3,

55.0, 43.1, 37.9, 31.6, 28.8. **HRMS** (ESMS) for $C_{19}H_{23}BrO_2$: calcd. 362.0881; found 362.0876.



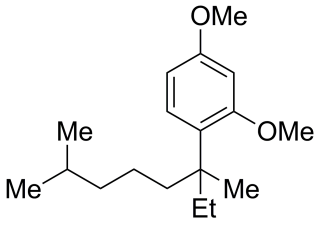
2-(2,4-Dimethoxyphenyl)-2-methyl-1-phenylpropane **3d**

Synthesized according to *general procedure A* using 2-fluoro-2-methyl-1-phenylpropane (76 mg, 0.50 mmol). Isolated (103 mg, 76%) as a yellow oil after Kugelrohr distillation (>20:1 ratio of regioisomers).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.19-7.14 (m, 3H), 6.95-6.89 (m, 3H), 6.60 (d, *J* = 2.5 Hz, 1H), 6.39 (dd, *J* = 8.5 Hz, 2.5 Hz, 1H), 3.96 (s, 3H), 3.84 (s, 3H), 3.18 (s, 2H), 1.40 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.3, 159.2, 140.3, 130.4, 128.7, 128.3, 127.4, 125.6, 103.4, 99.6, 55.3, 55.1, 46.4, 38.7, 28.4. **HRMS** (ESMS) for $C_{18}H_{22}O_2$+H: calcd. 271.1698; found 271.1693.



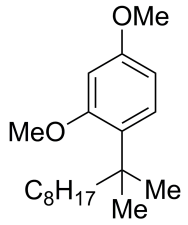
2-(2,4-Dimethoxyphenyl)-2,6-dimethylheptane **3e**

Synthesized according to *general procedure A* using 2-fluoro-2,6-dimethylheptane (73.2 mg, 0.500 mmol). Isolated (95 mg, 72%) as a yellow oil after Kugelrohr distillation (>20:1 ratio of regioisomers).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.14 (d, *J* = 8.5 Hz, 1H), 6.49 (d, *J* = 2.6 Hz, 1H), 6.45 (dd, *J* = 8.5 Hz, 2.6 Hz, 1H), 3.83 (s, 3H), 3.82 (s, 3H), 1.81-1.74 (m, 2H), 1.51 (hep, *J* = 6.6 Hz, 1H), 1.35 (s, 6H), 1.15-1.09 (m, 2H), 1.06-0.98 (m, 2H), 0.83 (d, *J* = 6.6 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.5, 159.0, 129.6, 128.1, 103.3, 99.6, 55.3, 55.0, 41.3, 39.9, 37.7, 28.7, 27.8, 23.0, 22.8. **HRMS** (ESMS) for $C_{17}H_{28}O_2$+H: calcd. 265.2168; found 265.2162.



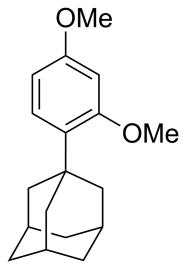
3-(2,4-Dimethoxyphenyl)-3,7-dimethyloctane **3f**

Synthesized according to *general procedure A* using 3-fluoro-3,7-dimethyloctane (80.2 mg, 0.500 mmol). Isolated (111 mg, 80%) of a yellow oil as a mixture of regioisomers (87:13) after Kugelrohr distillation.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.13 (d, *J* = 8.5 Hz, 0.13H), 7.06 (d, *J* = 8.5 Hz, 0.86H), 6.53-6.39 (m, 2H), 3.83-3.77 (m, 6H), 2.16-1.99 (m, 1.78H), 1.87-1.66 (m, 0.22H), 1.54-0.81 (m, 16.47H), 0.64 (t, *J* = 7.6 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) major regioisomer δ 159.5, 158.9, 129.4, 127.7, 103.2, 99.4, 55.2, 55.1, 41.5, 40.6, 40.0, 33.0, 27.8, 24.8, 22.9, 22.7, 9.2. **HRMS** (ESMS) for $C_{18}H_{30}O_2$+H: calcd. 279.2324; found 279.2319.



2-(2,4-Dimethoxyphenyl)-2-methyldecane **3g**

Synthesized according to *general procedure A* using 2-fluoro-2-methyldecane (87 mg, 0.50 mmol). Isolated (119 mg, 81%) as a yellow oil after Kugelrohr distillation (>20:1 ratio of regioisomers).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.14 (d, *J* = 8.5 Hz, 1H), 6.50 (d, *J* = 2.5 Hz, 1H), 6.45 (dd, *J* = 8.5 Hz, 2.5 Hz, 1H), 3.83 (s, 6H), 1.84-1.76 (m, 2H), 1.36 (s, 6H), 1.32-1.19 (m, 10H), 1.06-0.98 (m, 2H), 0.92 (t, *J* = 7.0 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.5, 159.0, 129.6, 128.1, 103.3, 99.6, 55.3, 55.0, 41.1, 37.7, 32.1, 30.6, 29.7, 29.5, 28.7, 25.3, 22.8, 14.3. **HRMS** (ESMS) for $C_{19}H_{32}O_2$+H: calcd. 293.2481; found 293.2475.

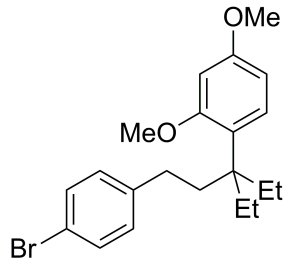


1-(2,4-Dimethoxy)-adamantane **3i**

Synthesized according to *general procedure A* using 1-adamantylfluoride (77.1 mg, 0.500 mmol). The nucleophile fraction was distilled off by Kugelrohr distillation and the residue was purified by column chromatography (2% Et_2O in Petroleum ether) to

give a white solid (95 mg, 70%) (>20:1 ratio of regioisomers). R_f = 0.38 (4% Et_2O in Petroleum ether).

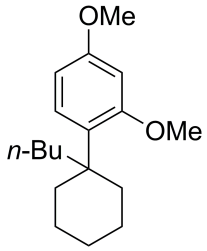
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.15 (d, *J* = 8.6 Hz, 1H), 6.50 (d, *J* = 2.6 Hz, 1H), 6.47 (dd, *J* = 8.6 Hz, 2.6 Hz, 1H), 3.84 (s, 3H), 3.82 (s, 3H), 2.12-2.07 (m, 9H), 1.80 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.8, 158.8, 131.4, 126.9, 103.5, 99.8, 55.3, 55.0, 41.0, 37.3, 36.5, 29.3. The analytical data are in accordance with those reported in the literature.[10] m.p. = 102-103 ºC.



1-(4-Bromophenyl)-3-ethyl-3-(2,4-dimethoxyphenyl)-pentane **3h**

Synthesized according to *general procedure A* using 1-(4-bromophenyl)-3-ethyl-3-fluoropentane (137 mg, 0.500 mmol). Isolated (148 mg, 76%) as a colorless oil after Kugelrohr distillation (>20:1 ratio of regioisomers).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 (d, *J* = 8.3 Hz, 2H), 7.11 (d, *J* = 8.6 Hz, 1H), 6.96 (d, *J* = 8.3 Hz, 2H), 6.48 (d, *J* = 2.6 Hz, 1H), 6.45 (dd, *J* = 8.6 Hz, 2.6 Hz, 1H), 3.81 (s, 3H), 3.79 (s, 3H), 2.23-2.17 (m, 2H), 2.07-2.01 (m, 2H), 1.95-1.85 (m, 2H), 1.82-1.72 (m, 2H), 0.70 (t, *J* = 7.4 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.5, 159.1, 143.1, 131.3, 130.2, 129.7, 126.6, 119.1, 103.4, 99.6, 55.3, 55.1, 43.9, 36.9, 30.6, 26.6, 8.5. **HRMS** (ESMS) for $C_{21}H_{27}BrO_2$: calcd. 390.1194; found 390.1189.

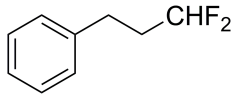


1-Butyl-(2,4-dimethoxyphenyl)-cyclohexane **3j**

Synthesized according to *general procedure A* using 1-butyl-fluorocyclohexane (79.2 mg, 0.500 mmol). Isolated (88 mg, 64%) as a colorless oil after Kugelrohr distillation (>20:1 ratio of regioisomers).

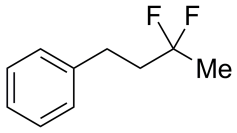
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.11 (d, *J* = 8.4 Hz, 1H), 6.49-6.42 (m, 2H), 3.81 (s, 3H), 3.77 (s, 3H), 2.18-2.07 (m, 2H), 1.77-1.62 (m, 4H), 1.59-1.51 (m, 2H), 1.47-1.37 (m, 4H), 1.16 (ap. sx, *J* = 7.4 Hz, 2H), 0.90-0.82 (m, 2H), 0.79 (t, *J* = 7.4 Hz, 3H);**^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.9, 158.7, 129.9, 127.7, 103.4, 99.8, 55.3, 55.2, 41.4, 37.8, 36.1, 27.1, 26.5, 23.6, 22.8, 14.2. **HRMS** (ESMS) for $C_{18}H_{28}O_2$+H: calcd. 277.2168; found 277.2162.

Procedures for preparation of substrates for leaving group selectivity study



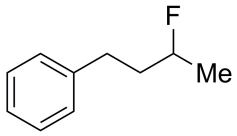
1,1-Difluoro-3-phenylpropane

Synthesized in analogy with the procedure reported by Daub and co-workers.[11] Hydrocinnamaldehyde (658 μL, 671 mg, 5.00 mmol) was dissolved in anhydrous dichloromethane (5 mL) in an oven-dried flask under dry nitrogen. This solution was cooled to 0 ^{o}C and (diethylamino)sulfur trifluoride (991 μL, 1.21 g, 7.50 mmol, 1.50 equiv) was added via syringe over a 1 min period. The reaction mixture was allowed to warm to room temperature and stirred overnight. The reaction was diluted with CH_2Cl_2, washed with saturated aqueous sodium chloride solution, dried over sodium sulfate, filtered and evaporated. The residue was purified by silica gel chromatography (Petroleum ether) to afford product (197 mg, 25% yield) as a colorless oil. R_f = 0.32 (Petroleum ether).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41-7.19 (m, 5H), 5.83 (tt, *J* = 56.7 Hz, 4.4 Hz, 1H), 2.82 (t, *J* = 7.9 Hz, 2H), 2.26-2.09 (m, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 140.1, 128.8, 128.4, 126.5, 116.8 (t, *J* = 239.2 Hz), 35.8 (t, *J* = 21.5 Hz), 28.5 (t, *J* = 6.1 Hz); **^{19}F NMR** (376 MHz, CF_3COOH) δ -115.9 (dt, *J* =56.7 Hz, 17.2 Hz). The analytical data are in accordance with those reported in the literature.[12]



3,3-Difluoro-1-phenylbutane

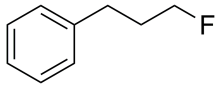
Synthesized in analogy with the procedure for preparation of 1,1-difluoro-3-phenylpropane starting with 4-phenyl-2-butanone (748 μL, 741 mg, 5.00 mmol). Crude product was purified by silica gel chromatography (Petroleum ether) to afford a colorless oil (221 mg, 26% yield). R_f = 0.30 (Petroleum ether).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37-7.19 (m, 5H), 2.89-2.81 (m, 2H), 2.27-2.11 (m, 2H), 1.66 (t, *J* = 18.4 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 140.8, 128.7, 128.4, 126.4, 123.9 (t, *J* = 238.3 Hz), 40.0 (t, *J* = 25.4 Hz), 29.0 (t, *J* = 4.9 Hz), 23.6 (t, *J* = 27.9 Hz); **^{19}F NMR** (376 MHz, CF_3COOH) δ -90.1 (qt, *J* =18.4 Hz, 16.2 Hz). **HRMS** (EI) for $C_{10}H_{12}F_2$: calcd. 170.0907; found 170.0902.



3-Fluoro-1-phenylbutane

To a solution of 4-phenyl-2-butanol (774 μL, 751 mg, 5.00 mmol) in dry THF (50 mL) at -78 °C, was added (diethylamino)sulfur trifluoride (991 μL, 1.21 g, 7.50 mmol, 1.50 equiv). The mixture was slowly warmed to room temperature and stirred for 2 h. The reaction was diluted with EtOAc, washed with saturated aqueous sodium chloride solution, dried over sodium sulfate, filtered and evaporated. The residue was

purified by silica gel chromatography (Petroleum ether) to afford product (406 mg, 53% yield) as a colorless oil. R_f = 0.39 (Petroleum ether).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39-7.19 (m, 5H), 4.82-4.60 (m, 1H), 2.91-2.69 (m, 2H), 2.11-1.78 (m, 2H), 1.40 (dd, *J* = 24.1 Hz, 6.2 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 141.6, 128.6, 126.1, 90.1 (d, *J* = 165.1 Hz), 38.8 (d, *J* = 20.9 Hz), 31.5 (d, *J* = 4.8 Hz), 21.1 (d, *J* = 22.6 Hz); **^{19}F NMR** (376 MHz, CF_3COOH) δ -173.9 – -174.4 (m). The analytical data are in accordance with those reported in the literature.[13]



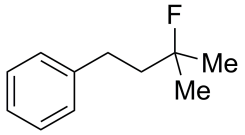
1-Fluoro-3-phenylpropane

Synthesized in analogy with the procedure for preparation of 2-fluoro-4-phenylbutane starting with 3-phenyl-1-propanol (681 μL, 681 mg, 5.00 mmol). The crude product was purified by silica gel chromatography (Petroleum ether) to afford a colorless oil (319 mg, 46% yield). R_f = 0.40 (Petroleum ether).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47-7.23 (m, 5H), 4.65-4.47 (dt, *J* = 47.3 Hz, 6.0 Hz, 2H), 2.91-2.87 (t, *J* = 7.4 Hz, 2H), 2.20-2.04 (m, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 141.2, 128.6, 128.5, 126.1, 83.1 (d, *J* = 164.9 Hz), 32.2 (d, *J* = 19.8 Hz), 31.4 (d, *J* = 5.4 Hz); **^{19}F NMR** (376 MHz, CF_3COOH) δ -218.8 (tt, *J* = 48.0 Hz, 23.1 Hz). The analytical data are in accordance with those reported in the literature.[13]

Procedures for preparation of tertiary aliphatic fluorides

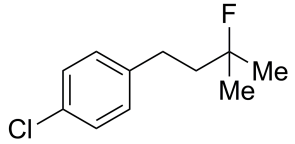
Condition A To a solution of alcohol (5.0 mmol) in dry THF (30 mL) at -78 °C, was added (diethylamino)sulfur trifluoride (1.32 mL, 1.61 g, 10.0 mmol, 2.00 equiv). The mixture was slowly warmed to room temperature and stirred for 2 h. The reaction was diluted with Et_2O, washed with pre-cooled saturated aqueous sodium chloride solution, dried over sodium sulfate, filtered and evaporated on the rotary evaporator (< 20 °C). The residue was purified by silica gel chromatography (Petroleum ether) to afford the product.
Condition B To a solution of alcohol (9.75 mmol) in dry CH_2Cl_2 (30 mL) at -78 °C, was added (diethylamino)sulfur trifluoride (2.60 mL, 3.14 g, 19.5 mmol, 2.00 equiv). The mixture was slowly warmed to room temperature and stirred for 2 h. The reaction was diluted with CH_2Cl_2, washed with saturated aqueous sodium chloride solution, dried over sodium sulfate, filtered and evaporated on the rotary evaporator (< 20 °C). The residue was purified by silica gel chromatography (Petroleum ether) to afford the product.

Spectral data for all tertiary aliphatic fluorides



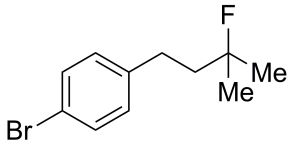
3-Fluoro-3-methyl-1-phenylbutane **1a**

Synthesized according to *general procedure B* starting with α,α-dimethylbenzenepropanol (1.65 mL, 1.60 g, 9.75 mmol). Isolated (1.34 g, 83%) as a colorless oil after column chromatography (Petroleum ether). R_f = 0.26 (Petroleum ether).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33-7.16 (m, 5H), 2.77-2.69 (m, 2H), 2.00-1.87 (m, 2H), 1.42 (d, *J* = 21.4 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 142.2, 128.6, 128.4, 126.0, 95.4 (d, *J* = 165.8 Hz), 43.5 (d, *J* = 23.0 Hz), 30.4 (d, *J* = 5.3 Hz), 26.8 (d, *J* = 25.1 Hz); **^{19}F NMR** (376 MHz, $CDCl_3$; CF_3COOH – ext. std.) δ -137.6 (ap. non, *J* = 20.9 Hz). The analytical data are in accordance with those reported in the literature.[14]



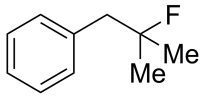
1-(4-Chlorophenyl)-3-fluoro-3-methylbutane **1b**

Synthesized according to *general procedure B* starting with 4-(4-chlorophenyl)-2-methyl-2-butanol[15] (1.29 g, 6.50 mmol). Isolated (970 mg, 74%) as a colorless oil after column chromatography (Petroleum ether). R_f = 0.20 (Petroleum ether).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 (d, *J* = 8.4 Hz, 2H), 7.16 (d, *J* = 8.4 Hz, 2H), 2.77- 2.69 (m, 2H), 1.98-1.86 (m, 2H), 1.44 (d, *J* = 21.3 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 140.6, 131.7, 129.8, 128.7, 95.2 (d, *J* = 166.5 Hz), 43.3 (d, *J* = 23.0 Hz), 29.7 (d, *J* = 5.3 Hz), 26.8 (d, *J* = 24.8 Hz); **^{19}F NMR** (376 MHz, $CDCl_3$; CF_3COOH – ext. std.) δ -138.0 (ap. non, *J* = 20.9 Hz). **HRMS** (EI) for $C_{11}H_{14}ClF$: calcd. 200.0768; found 200.0763.



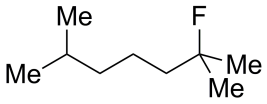
1-(4-Bromophenyl)-3-fluoro-3-methylbutane **1c**

Synthesized according to *general procedure B* starting with 4-(4-bromophenyl)-2-methyl-2-butanol[16] (544 mg, 2.23 mmol). Isolated (494 mg, 90%) as a yellow oil after column chromatography (Petroleum ether). R_f = 0.32 (Petroleum ether).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (d, *J* = 8.3 Hz, 2H), 7.08 (d, *J* = 8.3 Hz, 2H), 2.72- 2.65 (m, 2H), 1.95-1.84 (m, 2H), 1.41 (d, *J* = 21.4 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 141.3, 131.6, 130.2, 119.7, 95.2 (d, *J* = 165.4 Hz), 43.2 (d, *J* = 23.0 Hz), 29.8 (d, *J* = 5.0 Hz), 26.8 (d, *J* = 25.1 Hz);**^{19}F NMR** (376 MHz, $CDCl_3$; CF_3COOH – ext. std.) δ -138.0 (ap. non, *J* = 20.5 Hz). **HRMS** (EI) for $C_{11}H_{14}BrF$: calcd. 244.0263; found 244.0259.



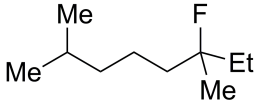
2-Fluoro-2-methyl-1-phenylpropane **1d**

Synthesized according to *general procedure B* starting with 2-methyl-1-phenyl-2-propanol (976 mg, 6.50 mmol). Isolated (625 mg, 63%) as a colorless oil after column chromatography (Petroleum ether). R_f = 0.32 (Petroleum ether).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35-7.21 (m, 5H), 2.93 (d, *J* = 20.5 Hz, 2H), 1.35 (t, *J* = 21.4 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 137.1 (d, *J* = 4.1 Hz), 130.5, 128.2, 126.7, 95.3 (d, *J* = 168.3 Hz), 47.8 (d, *J* = 23.3 Hz), 26.8 (d, *J* = 24.5 Hz); **^{19}F NMR** (376 MHz, $CDCl_3$; CF_3COOH – ext. std.) δ -136.9 (m). The analytical data are in accordance with those reported in the literature.[17]



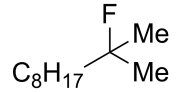
2-Fluoro-2,6-dimethylheptane **1e**

Synthesized according to *general procedure B* starting with 2,6-dimethyl-2-heptanol (937 mg, 6.50 mmol). Isolated (253 mg, 36%) as a colorless oil after column chromatography (Petroleum ether). R_f = 0.63 (Petroleum ether).
^{1}H NMR (400 MHz, $CDCl_3$) δ 1.63-1.50 (m, 3H), 1.42-1.27 (m, 8H), 1.22-1.14 (m, 2H), 0.88 (d, *J* = 6.7 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 95.9, 41.8 (d, *J* = 22.3 Hz), 39.4, 28.1, 26.8 (d, *J* = 24.3 Hz), 22.7, 21.9 (d, *J* = 5.3 Hz); **^{19}F NMR** (376 MHz, $CDCl_3$; CF_3COOH – ext. std.) δ -144.3 (ap. non, *J* = 20.7 Hz). **HRMS** (EI) for $C_9H_{19}F$ – HF: calcd. 126.1409; found 126.1404.



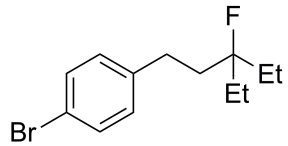
3-Fluoro-3,7-dimethyloctane **1f**

Synthesized according to *general procedure B* starting with 3,7-dimethyl-3-octanol (1.86 mL, 1.54 g, 9.75 mmol). Isolated (565 mg, 36%) as a colorless oil after column chromatography (Petroleum ether). R_f = 0.58 (Petroleum ether).
^{1}H NMR (400 MHz, $CDCl_3$) δ 1.68-1.50 (m, 5H), 1.39-1.32 (m, 2H), 1.28 (d, *J* = 21.9 Hz, 3H), 1.21-1.14 (m, 2H), 0.92 (t, *J* = 7.6 Hz, 3H), 0.88 (d, *J* = 6.7 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 97.9, 39.5, 39.4 (d, *J* = 23.0 Hz), 32.4 (d, *J* = 23.2 Hz), 28.1, 23.9 (d, *J* = 25.0 Hz), 22.7, 21.5 (d, *J* = 5.9 Hz), 8.1 (d, *J* = 6.8 Hz); **^{19}F NMR** (376 MHz, $CDCl_3$; CF_3COOH – ext. std.) δ -144.3 (ap. oct, *J* = 20.9 Hz). **HRMS** (EI) for $C_{10}H_{21}F$ – HF: calcd. 140.1565; found 140.1559.



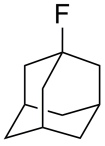
2-Fluoro-2-methyldecane **1g**

Synthesized according to *general procedure B* starting with 2-methyldecan-2-ol.[3] (424 mg, 2.45 mmol). Isolated (353 mg, 83%) as a colorless oil after column chromatography (Petroleum ether). R_f = 0.28 (Petroleum ether).
^{1}H NMR (400 MHz, $CDCl_3$) δ 1.64-1.53 (m, 2H), 1.41-1.21 (m, 18H), 0.88 (t, *J* = 7.0 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 96.0 (d, *J* = 164.5 Hz), 41.7 (d, *J* = 22.6 Hz), 32.0, 30.2, 29.7, 29.4, 26.8 (d, *J* = 25.3 Hz), 24.1 (d, *J* = 5.4 Hz), 22.8, 14.2; **^{19}F NMR** (376 MHz, $CDCl_3$; CF_3COOH – ext. std.) δ -135.8 (ap. nont, *J* = 21.6 Hz, 2.9 Hz). **HRMS** (EI) for $C_{11}H_{23}F$ – HF: calcd. 154.1722; found 154.1716.



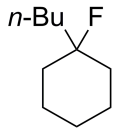
1-(4-Bromophenyl)-3-ethyl-3-fluoropentane **1h**

Synthesized according to *general procedure B* starting with 1-(4-bromophenyl)-3-ethylpentan-3-ol[18] (1.09 g, 4.00 mmol). Isolated (918 mg, 84%) as a yellow oil after column chromatography (Petroleum ether). R_f = 0.28 (Petroleum ether).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (d, *J* = 8.4 Hz, 2H), 7.08 (d, *J* = 8.4 Hz, 2H), 2.66- 2.60 (m, 2H), 1.91-1.81 (m, 2H), 1.76-1.64 (m, 4H), 0.94 (t, *J* = 7.6 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 141.4, 131.6, 130.2, 119.7, 99.2 (d, *J* = 170.5 Hz), 38.3 (d, *J* = 23.1 Hz), 29.2 (d, *J* = 5.8 Hz), 23.9 (d, *J* = 29.0 Hz), 7.8 (d, *J* = 7.3 Hz);**^{19}F NMR** (376 MHz, $CDCl_3$; CF_3COOH – ext. std.) δ -153.4 (ap. sp, *J* = 19.3 Hz). **HRMS** (EI) for $C_{13}H_{18}BrF$: calcd. 272.0576; found 272.0571.



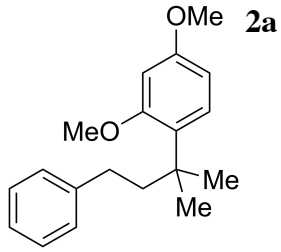
1-Fluoroadamantane **1i**

Synthesized according to the *general procedure B* starting with 1-adamantanol (761 mg, 5.00 mmol). Isolated (635 mg, 82%) as a white solid after column chromatography (Petroleum ether). R_f = 0.33 (Petroleum ether).
^{1}H NMR (400 MHz, $CDCl_3$) δ 2.23 (s, 3H), 1.93-1.84 (m, 6H), 1.68-1.57 (m, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 92.5 (d, *J* = 183.5 Hz), 42.9 (d, *J* = 16.9 Hz), 36.0 (d, *J* = 2.2 Hz), 31.6 (d, *J* = 9.8 Hz); **^{19}F NMR** (376 MHz, $CDCl_3$; CF_3COOH – ext. std.) δ -128.3 to -128.5 (m). mp = 205-207 ºC. The analytical data are in accordance with those reported in the literature.[19]



1-Butyl-fluorocyclohexane **1j**

Synthesized according to the *general procedure B* starting with 1-butylcyclohexanol (938 mg, 6.00 mmol). Isolated (470 mg, 50%) as a colorless oil after column chromatography (Petroleum ether). R_f = 0.66 (Petroleum ether).
^{1}H NMR (400 MHz, $CDCl_3$) δ 1.84-1.74 (m, 2H), 1.65-1.20 (m, 14H), 0.90 (t, *J* = 7.0 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 96.2 (d, *J* = 169.7 Hz), 40.2 (d, *J* = 23.2 Hz), 35.2 (d, *J* = 22.9 Hz), 25.6, 25.2 (d, *J* = 4.6 Hz), 23.3, 22.2 (d, *J* = 3.2 Hz), 14.2; **^{19}F NMR** (376 MHz, $CDCl_3$; CF_3COOH – ext. std.) δ -153.9 (br). **HRMS** (EI) for $C_{10}H_{19}F$ – HF: calcd. 138.1409; found 138.1404.


OMe
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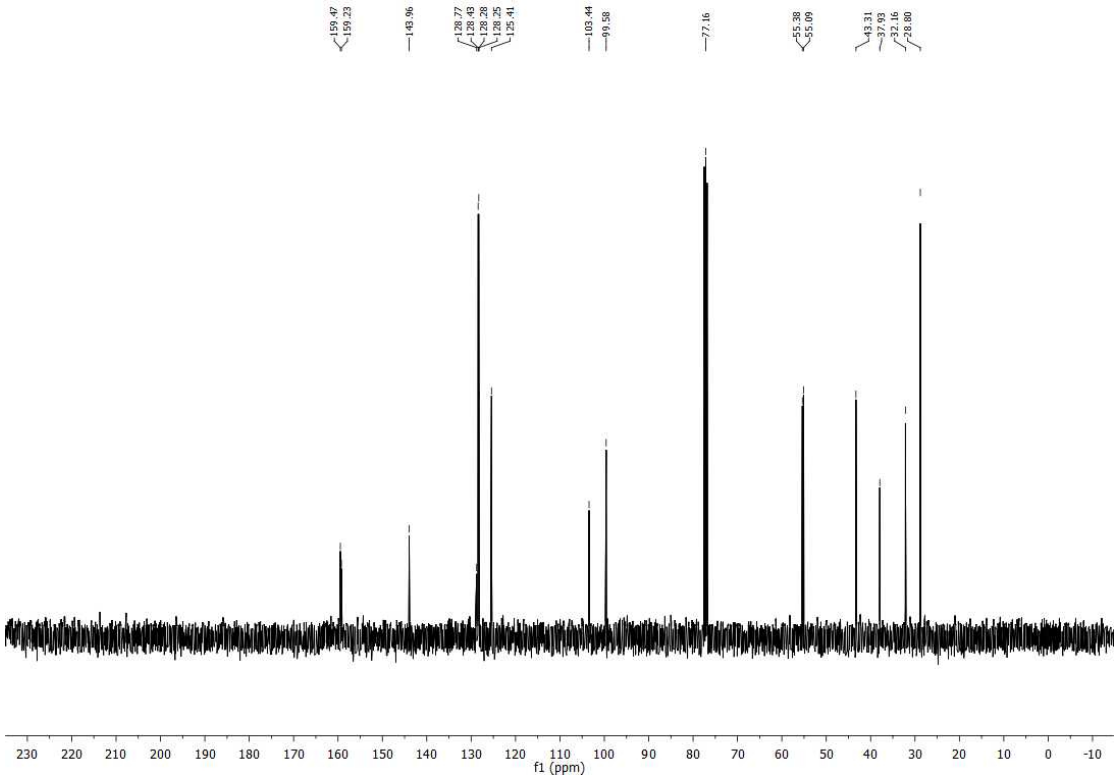
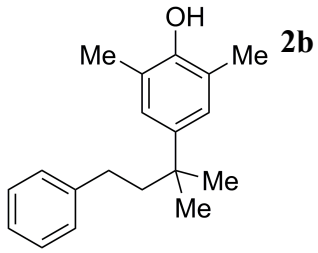
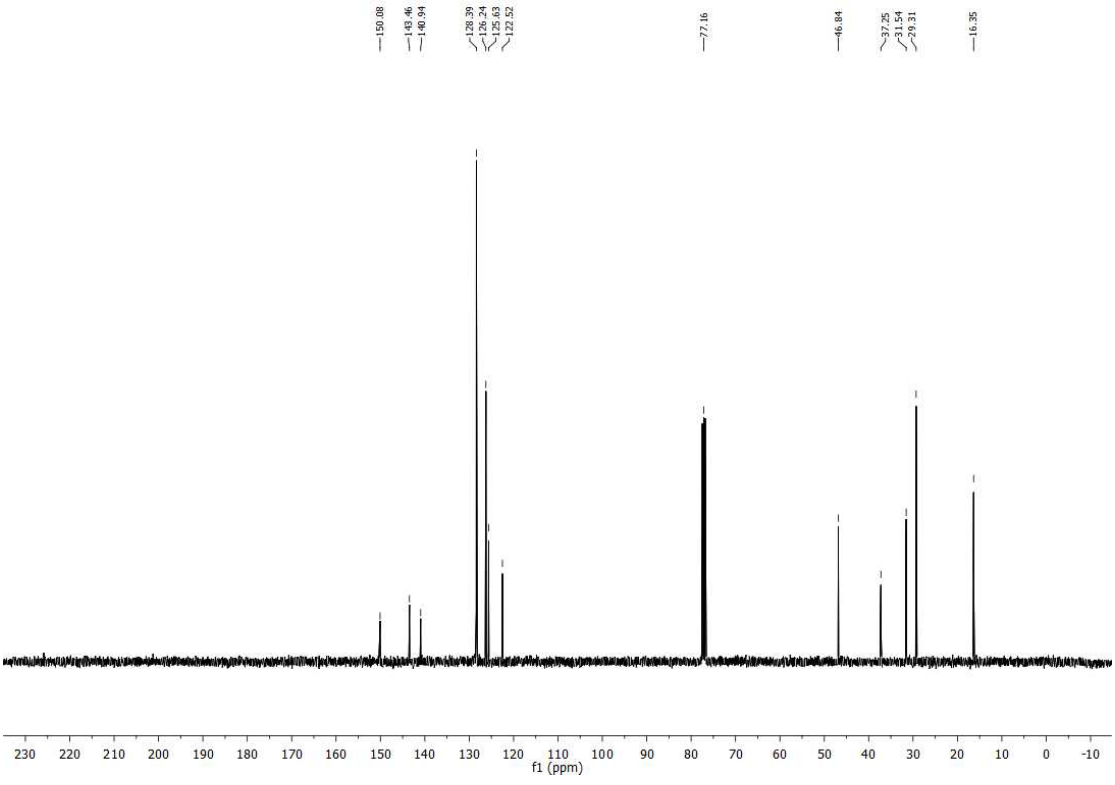
1.00
1.01
6.03
2.03
2.02
6.03
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


159.47
159.23
143.96
128.77
128.43
128.28
126.25
125.41
103.44
99.58
77.16
55.38
55.09
43.31
37.93
32.16
28.80
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

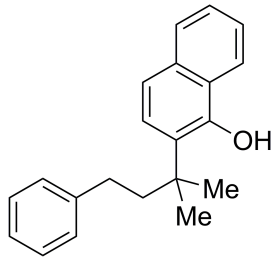

OH
Me
Me
Me
Me

2b

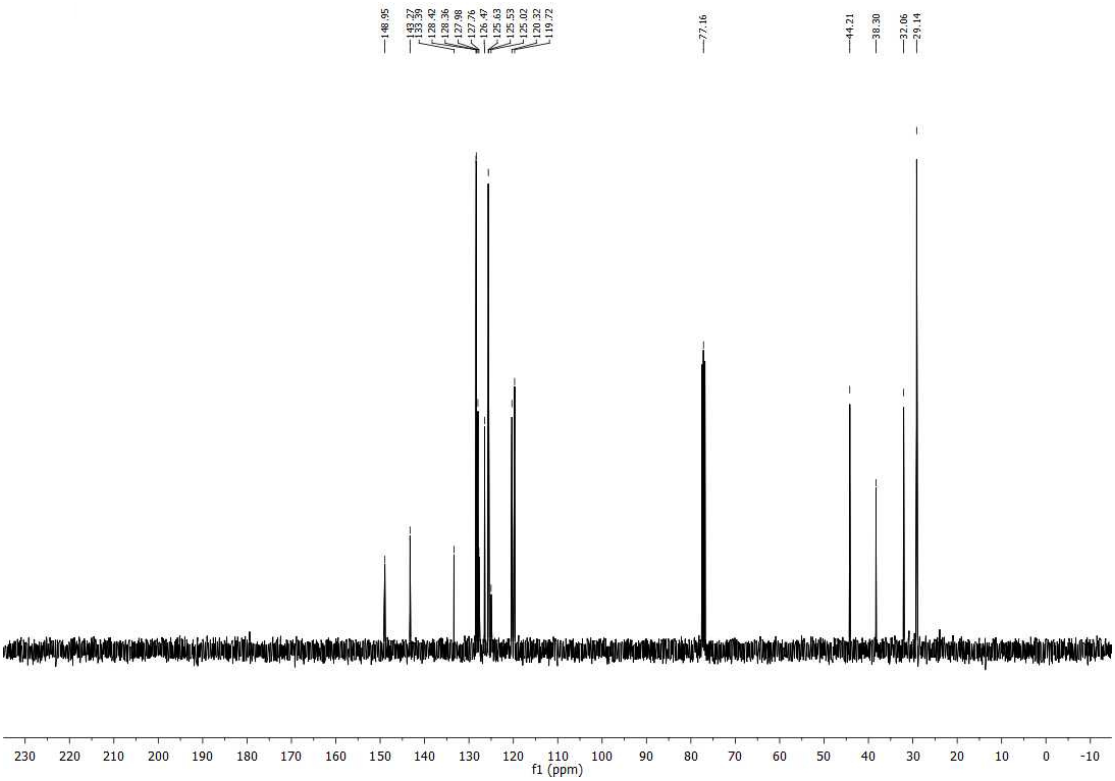
2.03
3.03
2.00
0.88
2.00
6.01
2.01
6.04
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

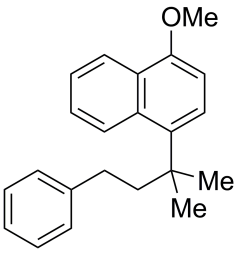

150.08
143.46
140.94
128.39
126.24
125.63
122.52
77.16
46.84
37.25
31.54
29.31
16.35
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

2c

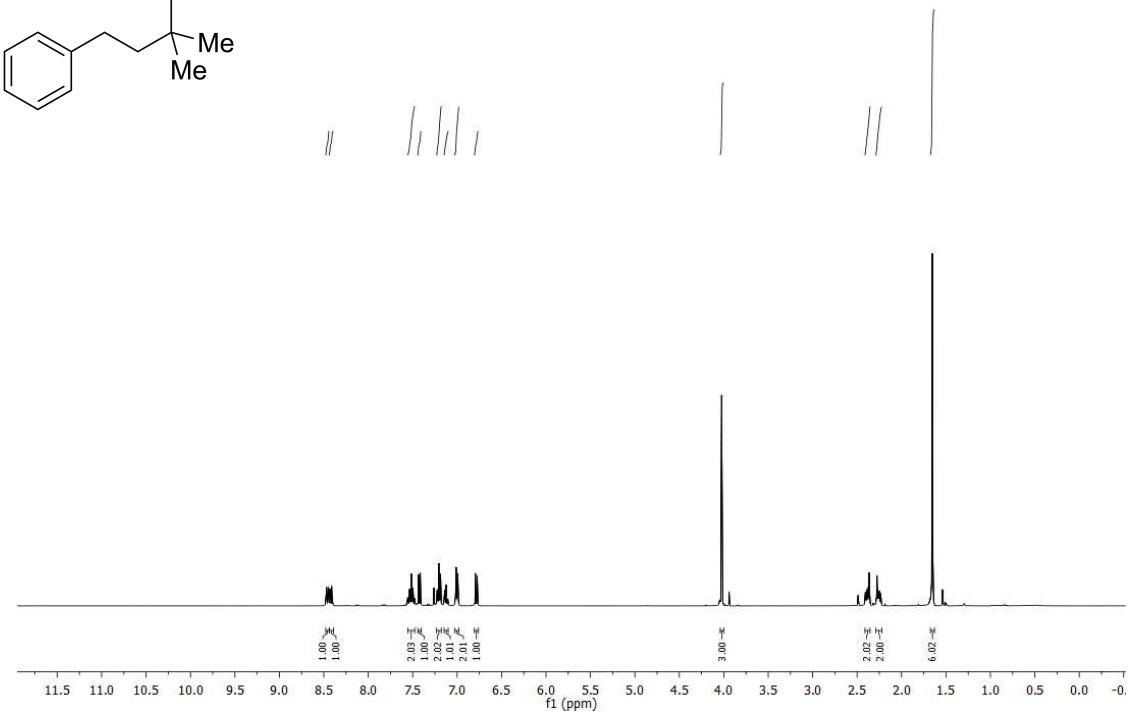

OH
Me
Me

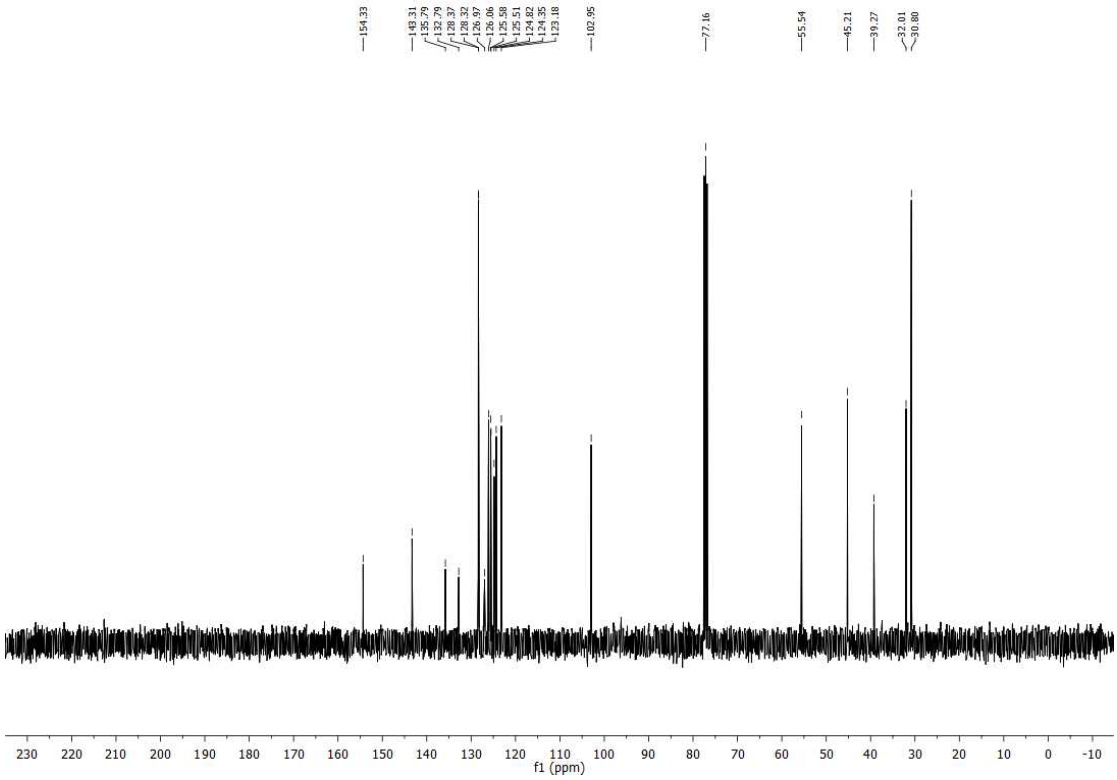
1.00
1.00
4.02
2.00
3.00
1.01
2.02
2.02
6.04
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

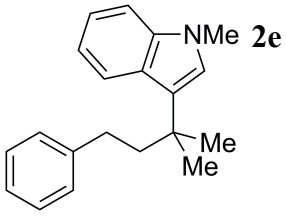

148.95
137.77
133.56
128.42
128.36
127.98
127.76
126.47
125.63
125.53
125.02
120.32
119.72
77.16
44.21
38.30
32.06
29.14
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


OMe
Me
Me

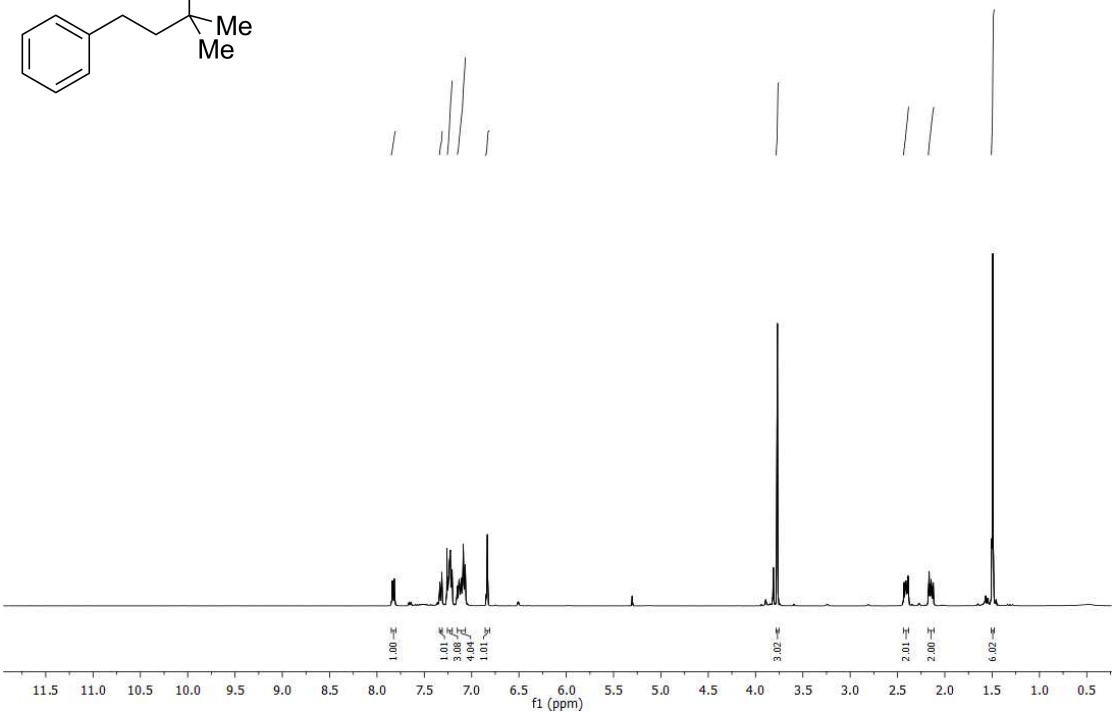
2d

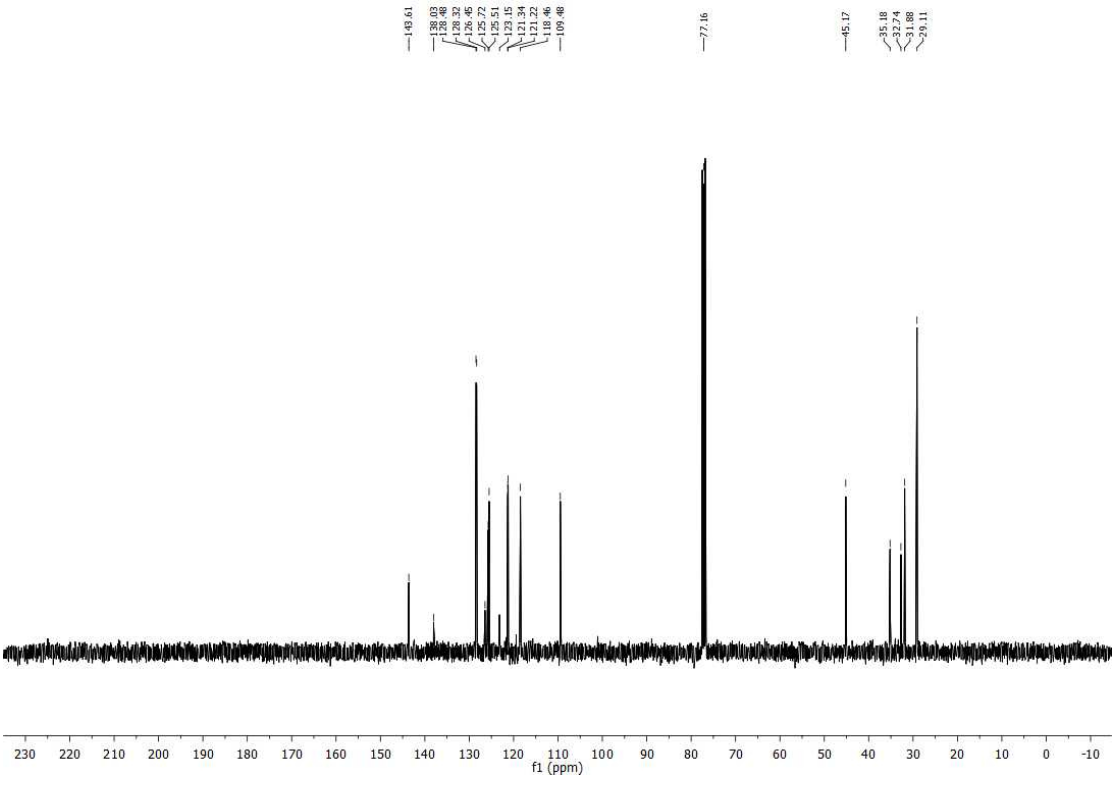

1.00
1.00
2.03
1.00
2.02
1.01
2.01
1.00
3.00
2.02
2.00
6.02
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

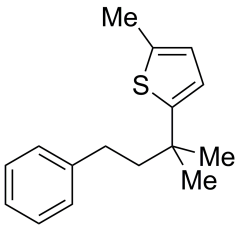

154.33
148.31
135.79
132.79
128.37
128.32
126.97
126.06
125.58
125.51
124.82
124.35
123.18
102.95
77.16
55.54
45.21
39.27
32.01
30.80
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


NMe
Me
Me

2e

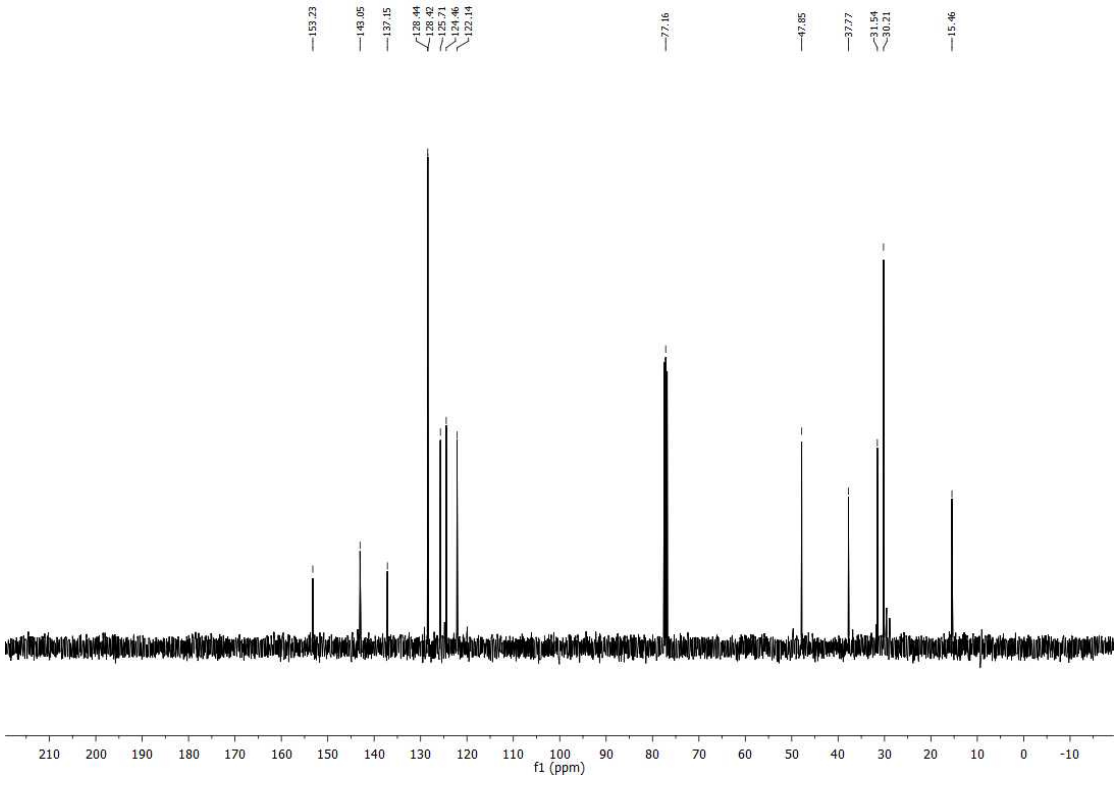

1.00
1.01
3.08
4.04
1.01
3.02
2.01
2.00
6.02
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

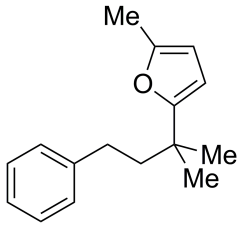

143.61
138.03
128.48
128.32
126.46
125.72
125.51
123.15
121.34
121.22
118.46
109.48
77.16
45.17
35.18
32.74
31.88
29.11
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


Me
S
Me
Me

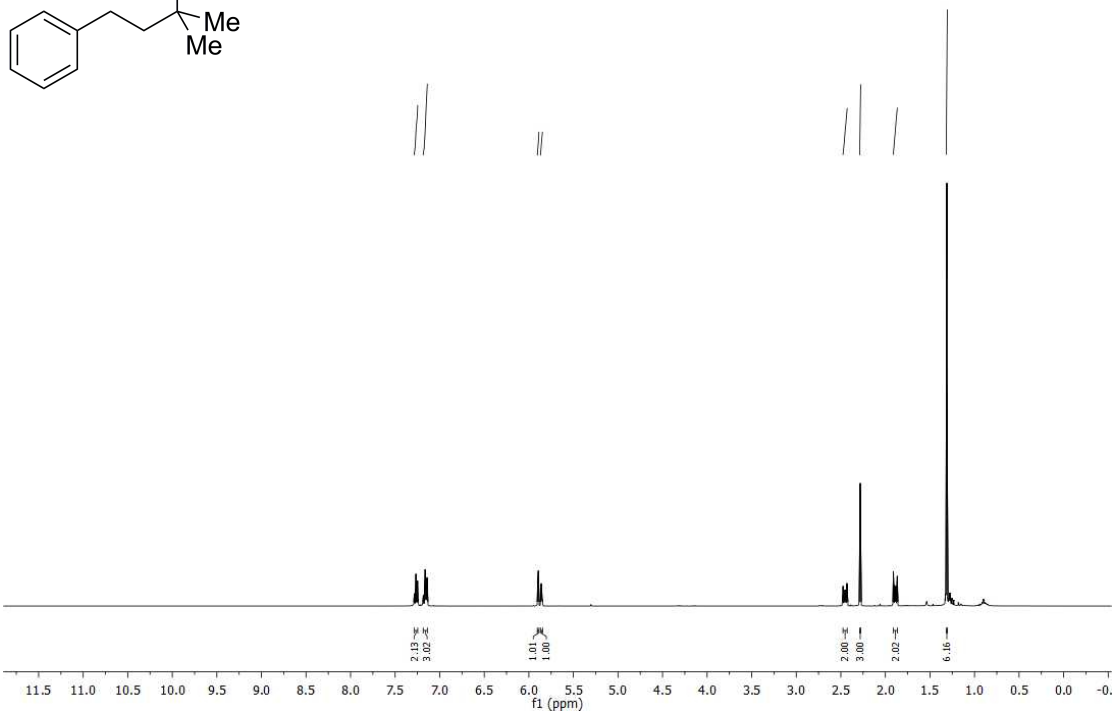
2f

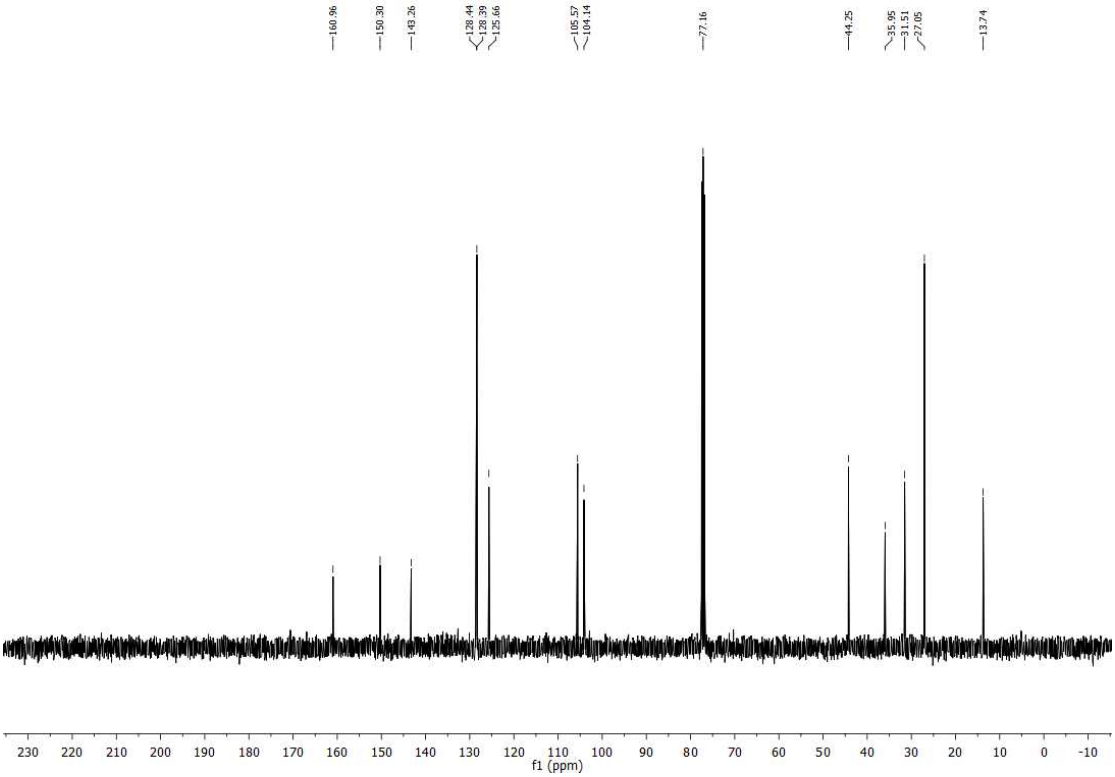
2.00
3.03
0.15
0.14
0.85
0.85
5.01
2.00
6.00
f1 (ppm)

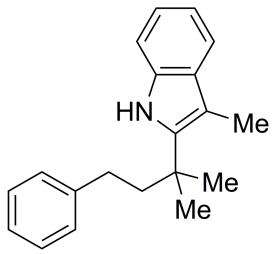

153.23
143.05
137.15
128.44
128.42
125.71
124.46
122.14
77.16
47.85
37.77
31.54
30.21
15.46
f1 (ppm)


Me
O
Me
Me

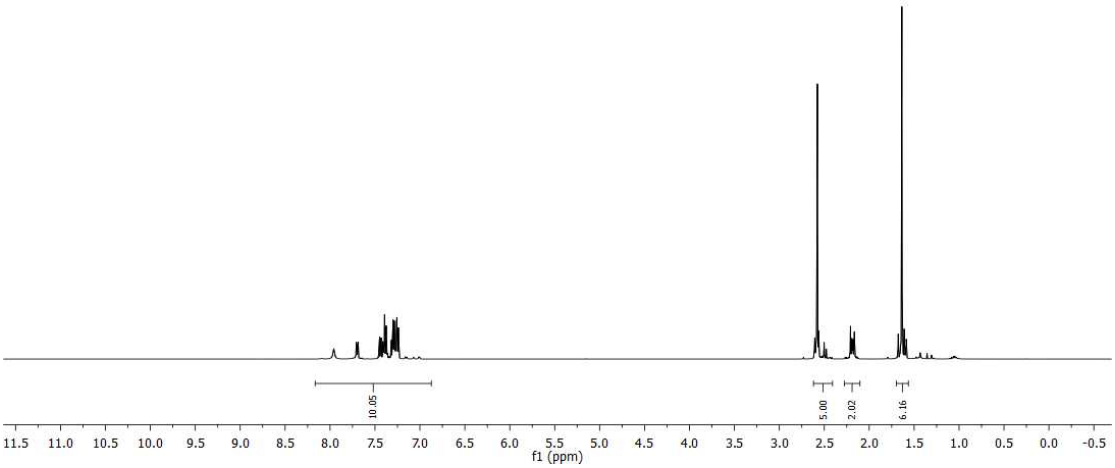
2g

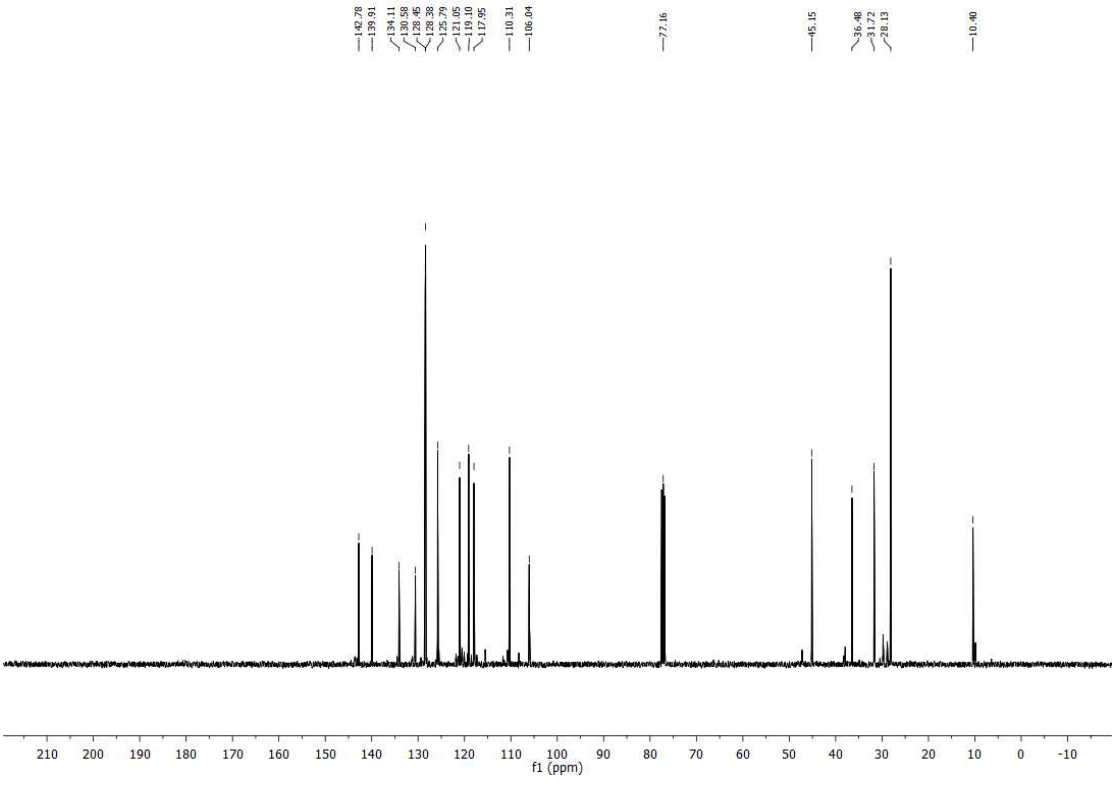

2.13
3.02
1.01
1.00
2.00
3.00
2.02
6.16
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.
f1 (ppm)

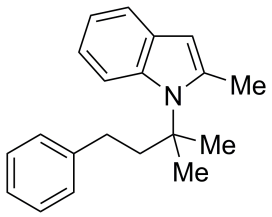

160.96
150.30
143.26
128.44
128.39
125.66
105.57
104.14
77.16
44.25
35.95
31.51
27.05
13.74
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


HN
Me
Me
Me

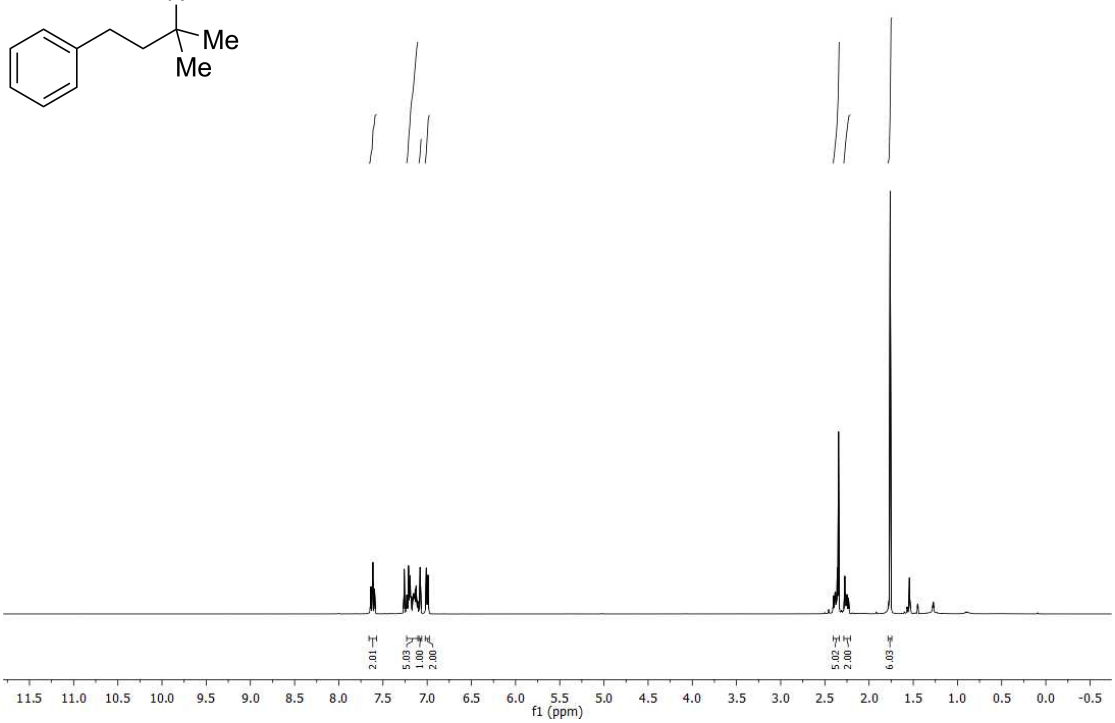
2h-major

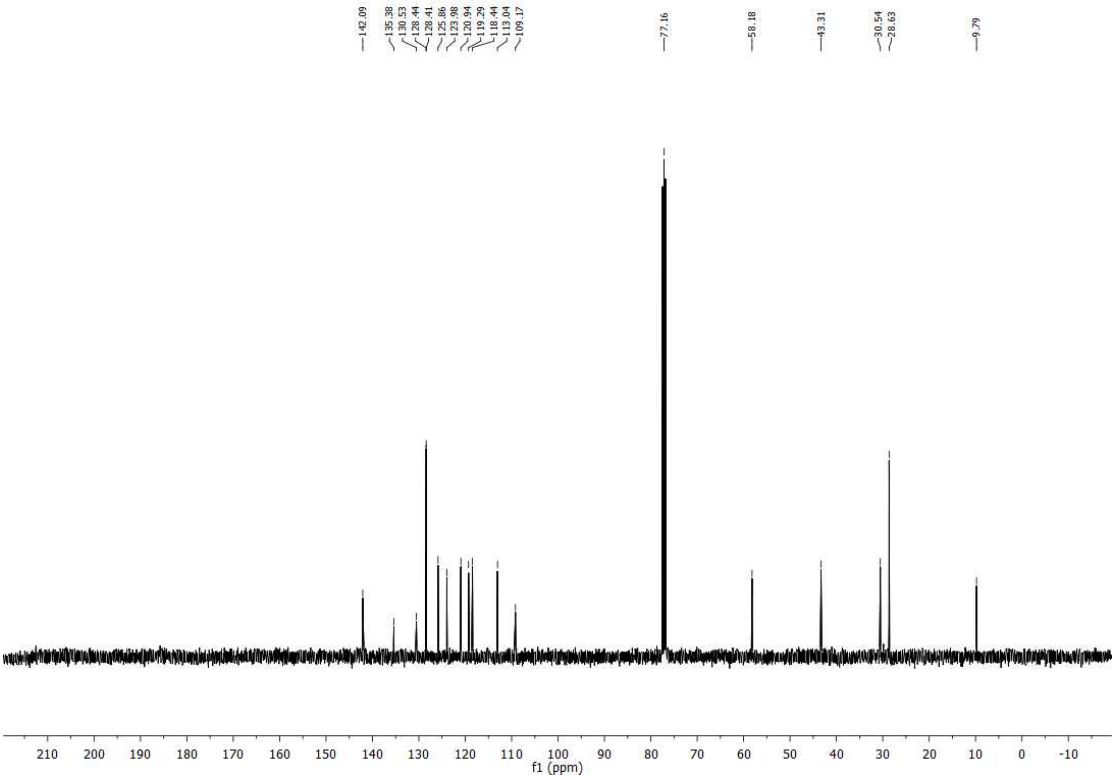

10.05
5.00
2.02
6.16
f1 (ppm)

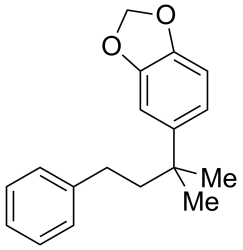

142.78
139.91
134.11
130.58
128.45
128.38
125.79
121.05
119.10
117.95
110.31
106.04
77.16
45.15
36.48
31.72
28.13
10.40
f1 (ppm)


Me
N
Me
Me

2h minor

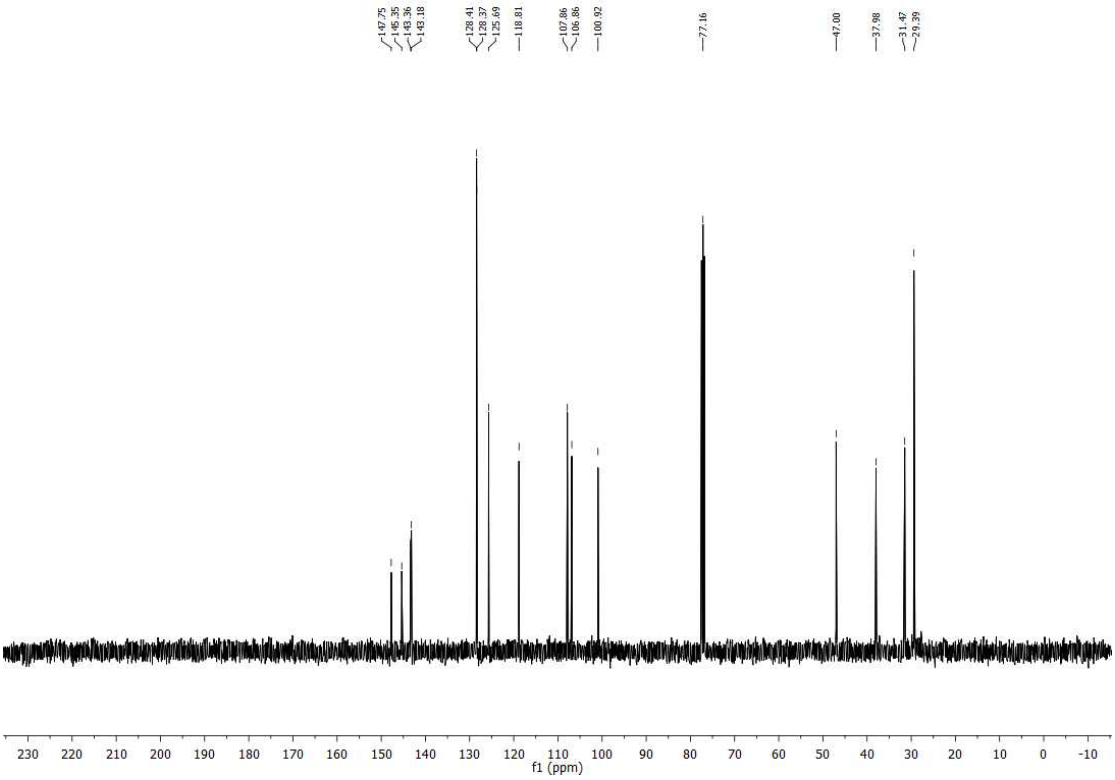

2.01
5.03
1.00
2.00
5.02
2.00
6.03
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

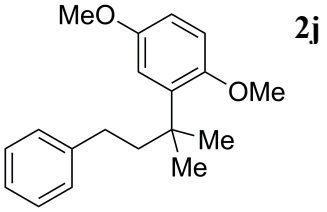

142.09
135.38
130.53
128.44
128.41
125.86
123.98
120.94
119.29
118.44
113.04
109.17
77.16
58.18
43.31
30.54
28.63
9.79
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


O
O
Me
Me

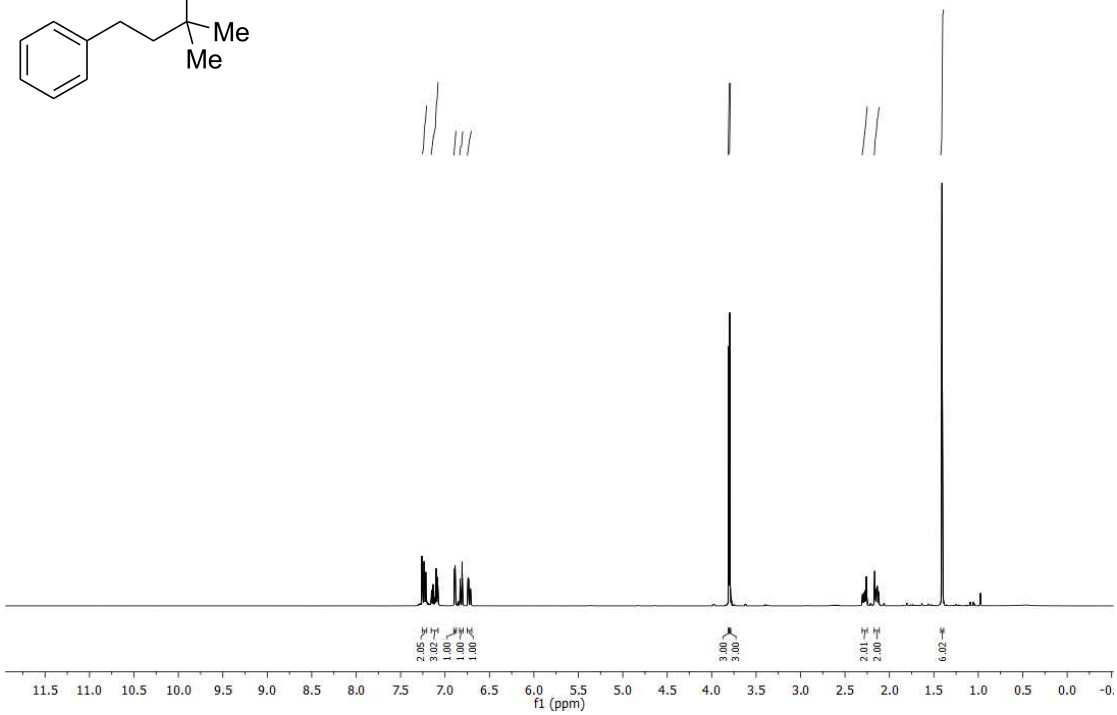
2i

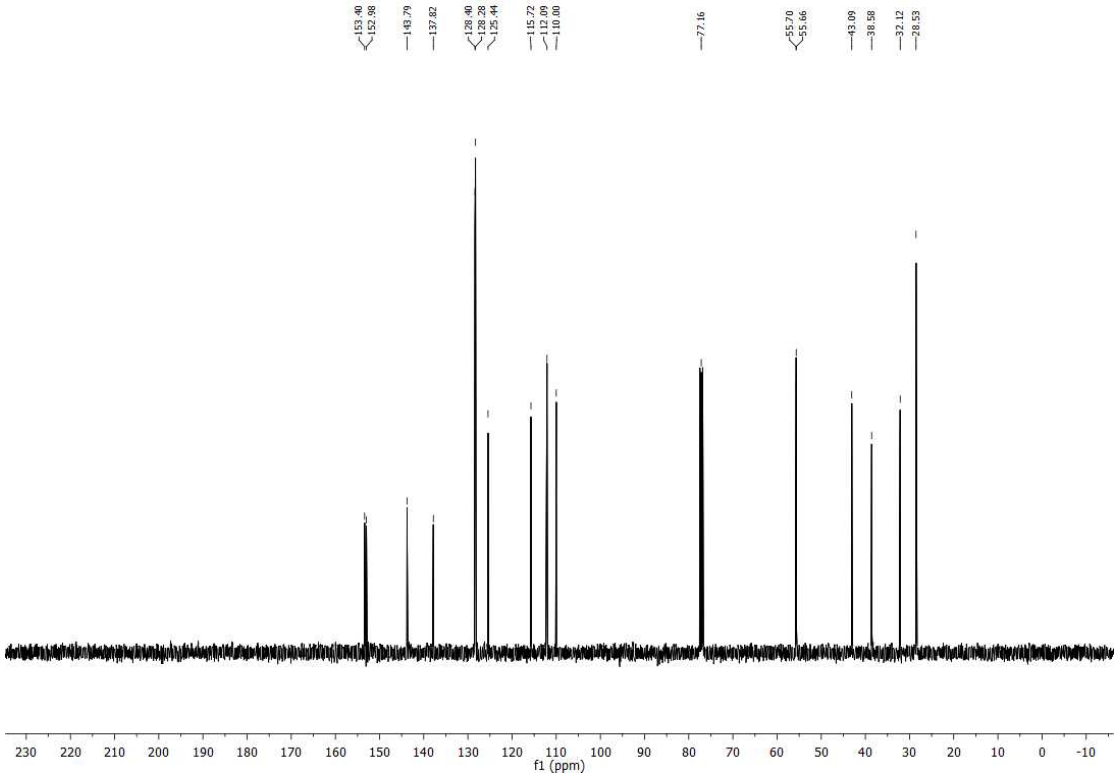
2.18
3.14
0.92
0.92
1.15
1.85
0.15
2.20
0.22
1.98
0.41
5.65
0.41
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0
f1 (ppm)

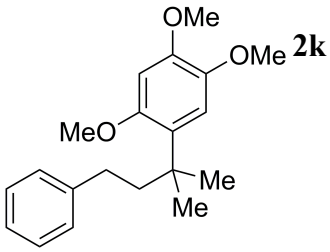

147.75
145.35
143.36
143.18
128.41
128.37
125.69
118.81
107.86
106.86
100.92
77.16
47.00
37.98
31.47
29.39
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


MeO
OMe
Me
Me

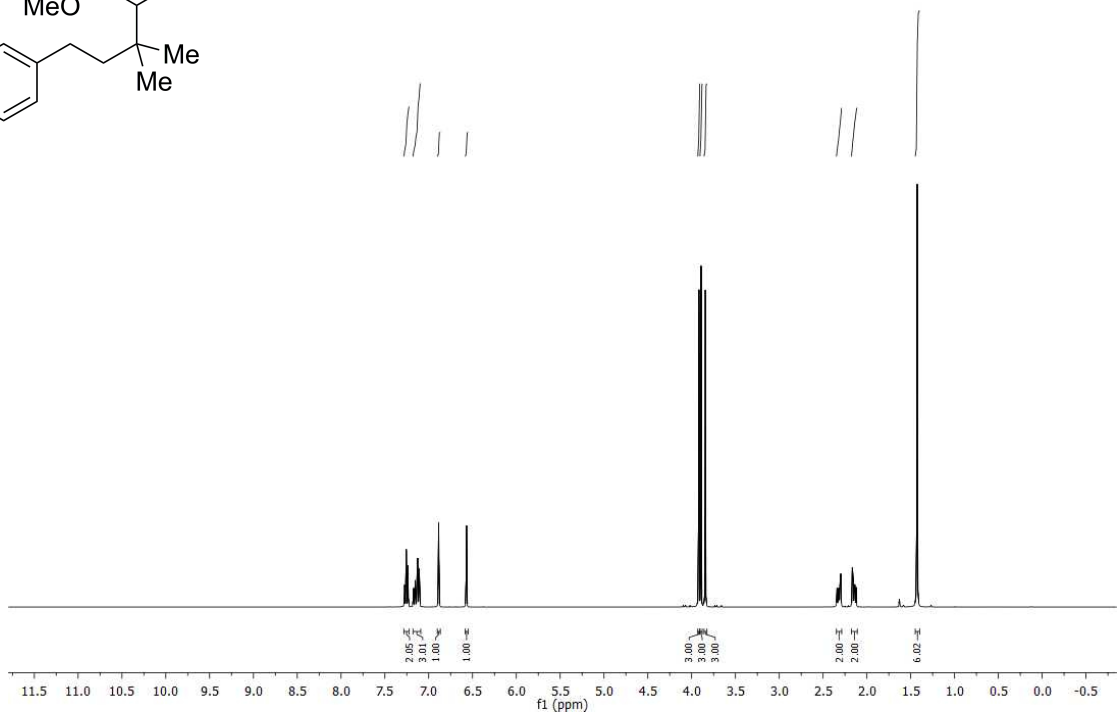
2j

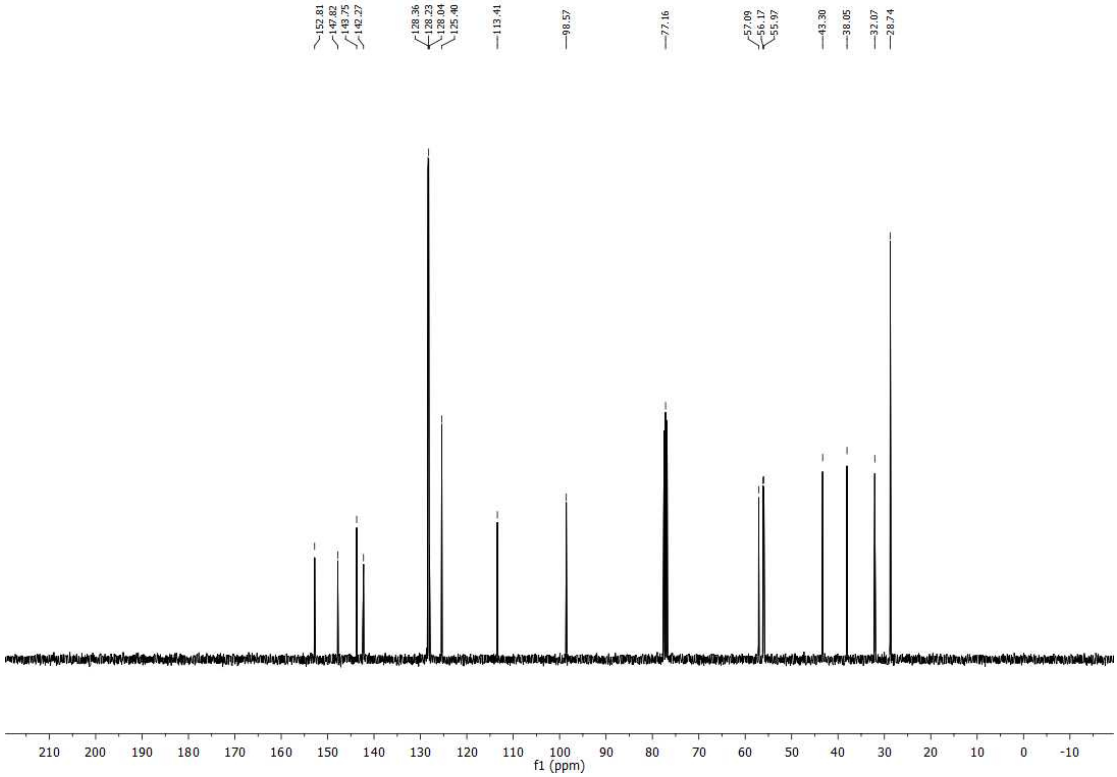

2.05
3.02
1.00
1.00
1.00
3.00
3.00
2.01
2.00
6.02
f1 (ppm)

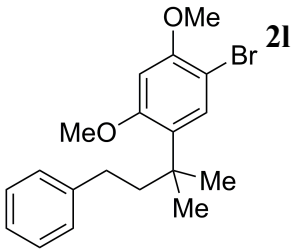

153.40
152.98
143.79
137.82
128.40
128.28
125.44
115.72
112.09
110.00
77.16
55.70
55.66
43.09
38.58
32.12
28.53
f1 (ppm)


OMe
OMe
MeO
Me
Me

2k

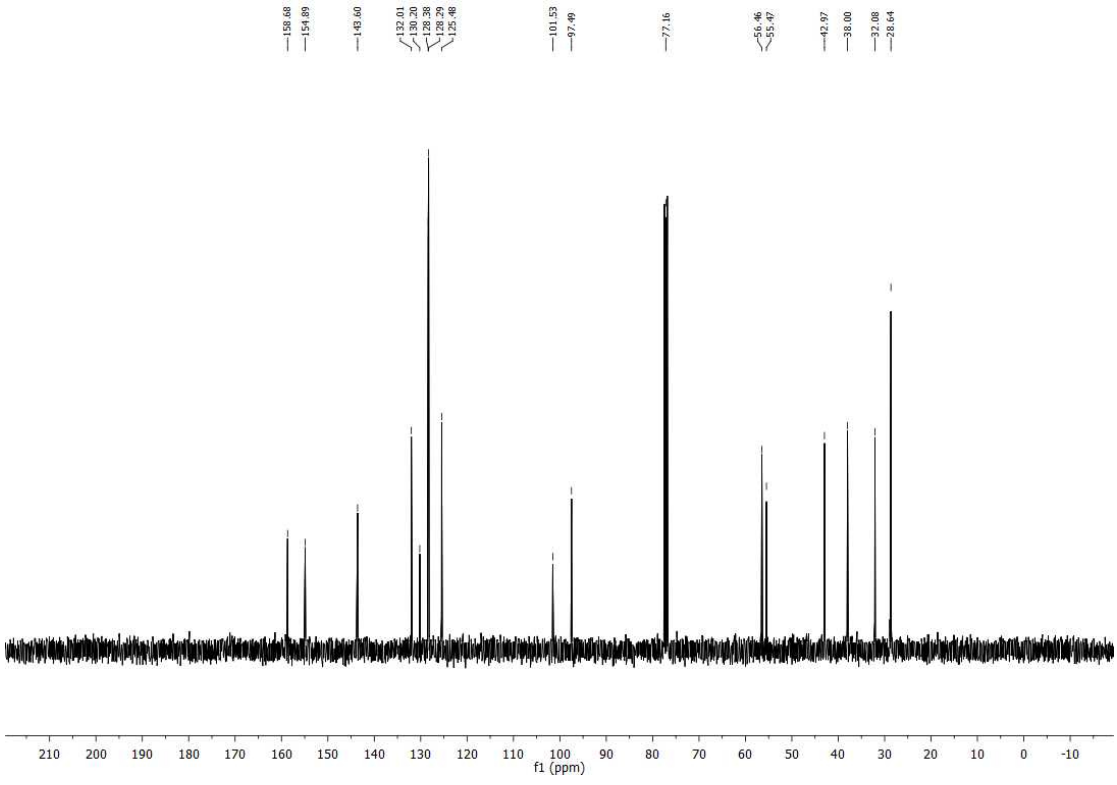

2.05
3.01
1.00
1.00
3.00
3.00
3.00
2.00
2.00
6.02
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

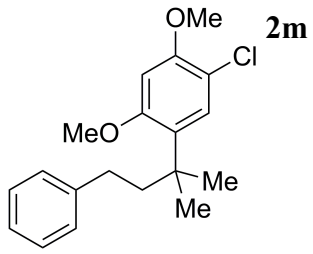

152.81
147.82
143.75
142.27
128.36
128.23
128.04
125.40
113.41
98.57
77.16
57.09
56.17
55.97
43.30
38.05
32.07
28.74
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


OMe
Br
MeO
Me
Me

2l

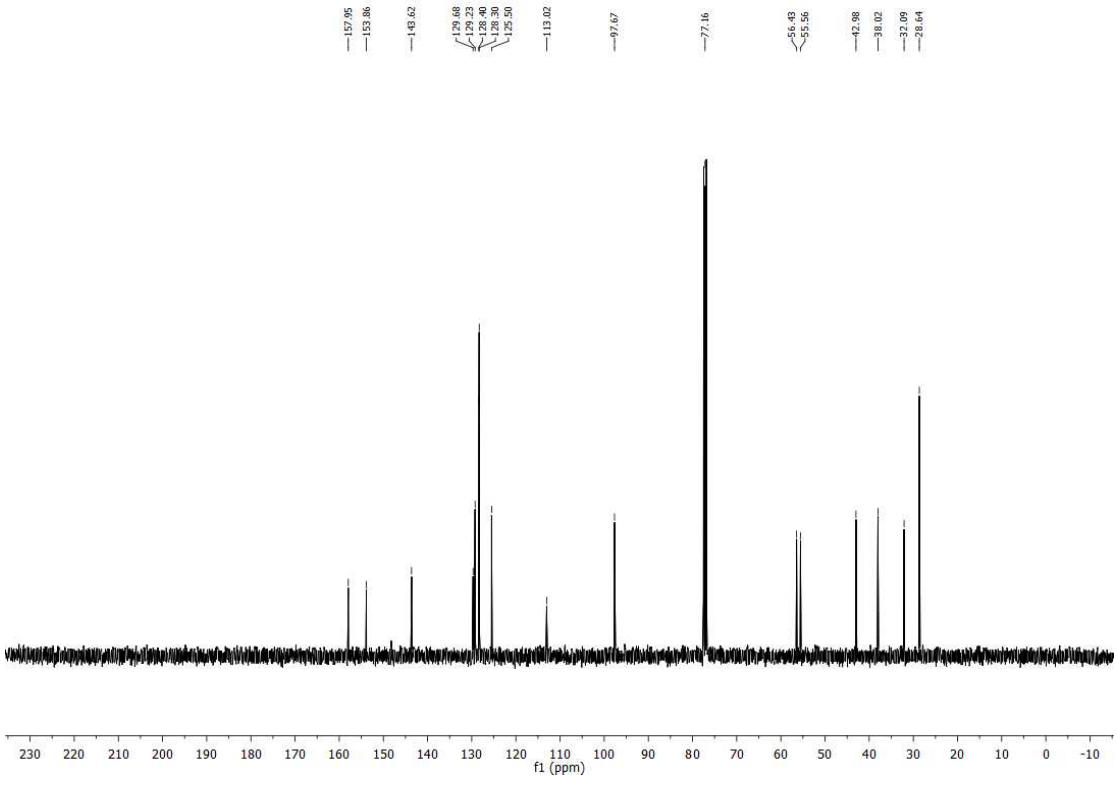
1.00
2.29
1.00
2.00
1.00
3.03
3.01
2.01
2.00
6.09
f1 (ppm)

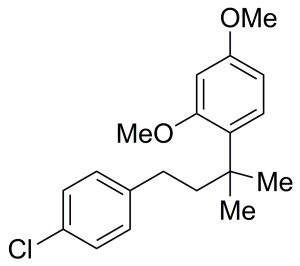

158.68
154.89
148.60
132.01
130.20
128.38
128.29
125.48
101.53
97.49
77.16
56.46
55.47
42.97
38.00
32.08
28.64
f1 (ppm)


OMe
Cl
MeO
Me
Me

2m

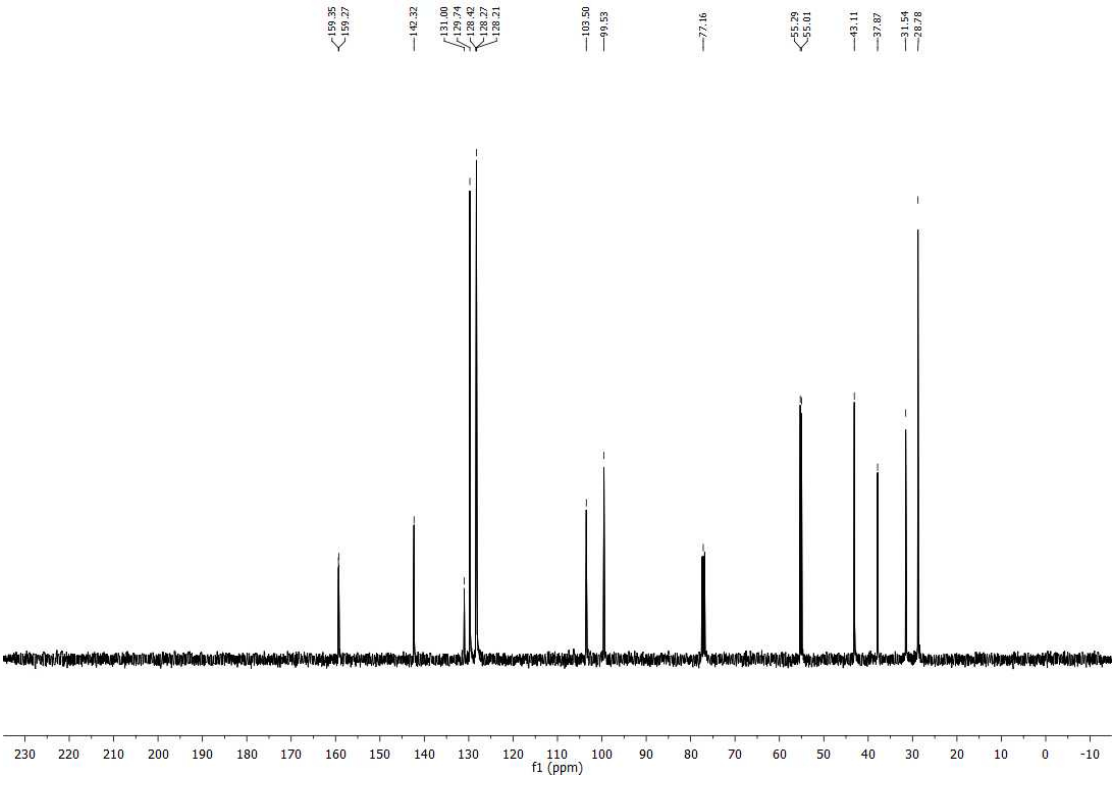
3.32
1.02
2.01
1.00
3.00
3.00
2.01
2.01
6.06
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

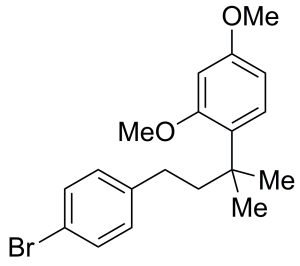

157.95
153.86
148.62
129.68
129.23
128.40
128.30
125.50
113.02
97.67
77.16
56.43
55.56
42.98
38.02
32.09
28.64
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


OMe
MeO
Me
Me
Cl

3b

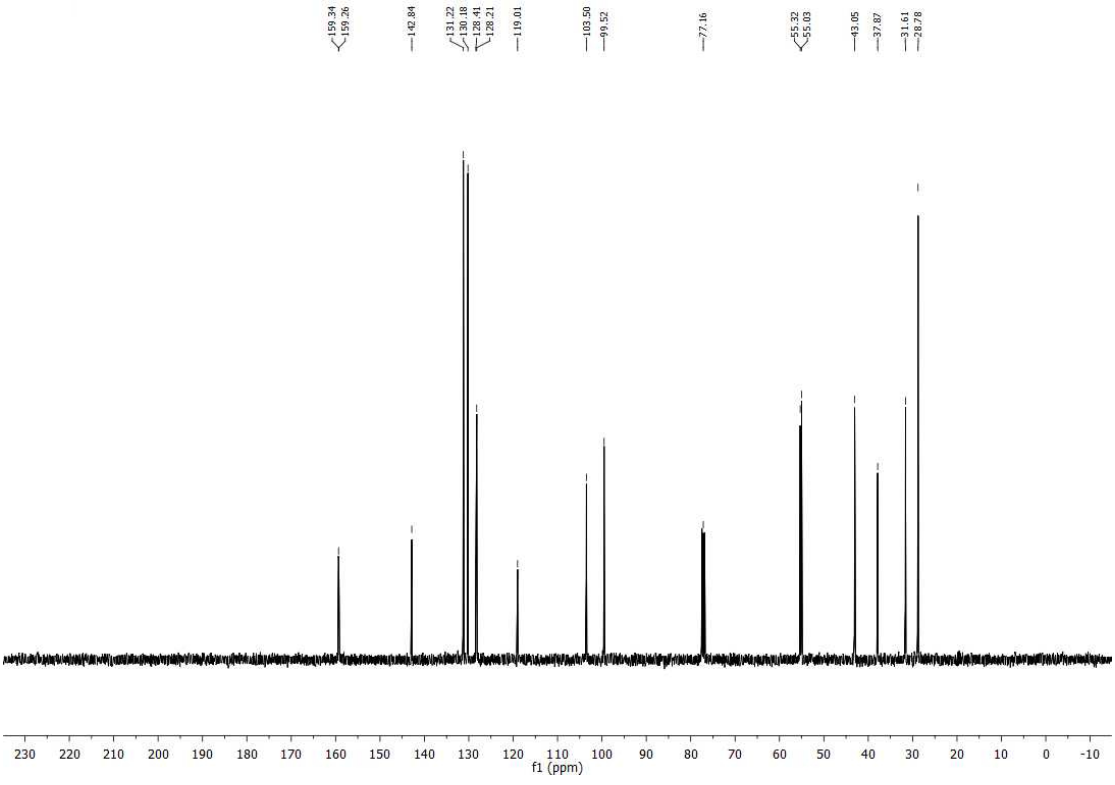
3.01
2.02
1.08
1.00
6.03
2.02
2.00
6.07
f1 (ppm)

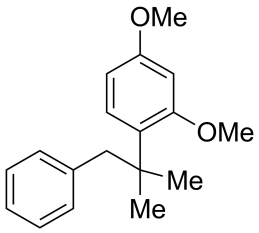

159.35
159.27
142.32
131.00
129.74
128.42
128.27
128.21
103.50
99.53
77.16
55.29
55.01
43.11
37.87
31.54
28.78
f1 (ppm)


OMe
MeO
Me
Me
Br

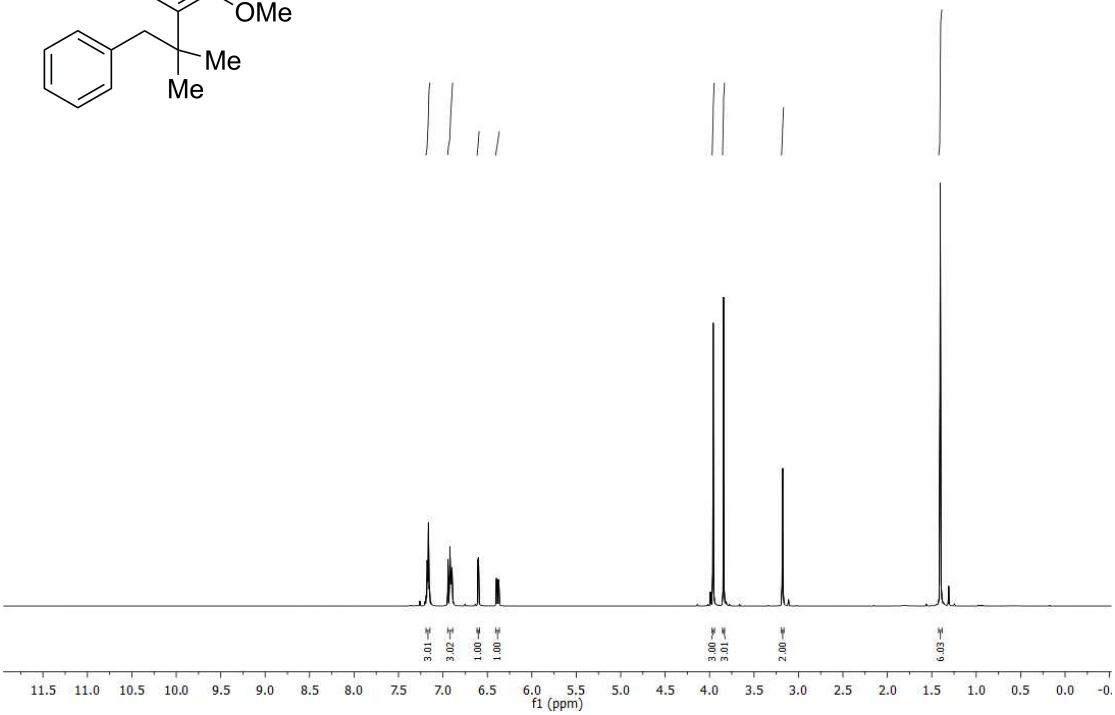
3c

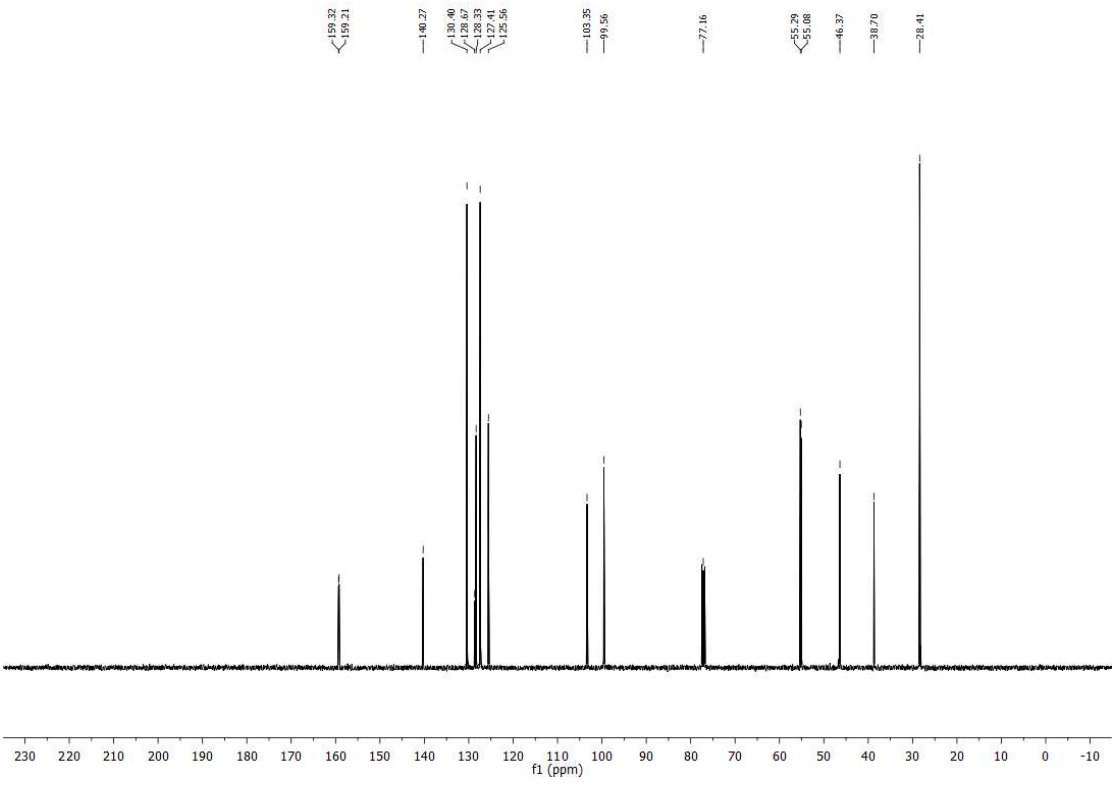
2.00
1.00
2.00
1.01
1.00
6.05
2.02
2.01
6.01
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

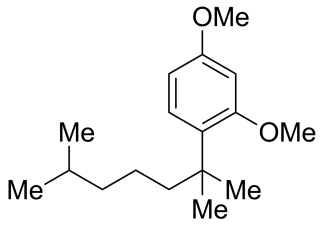

159.34
159.26
142.84
131.22
130.18
128.41
128.21
119.01
103.50
99.52
77.16
55.32
55.03
43.05
37.87
31.61
28.78
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


OMe
OMe
Me
Me

3d

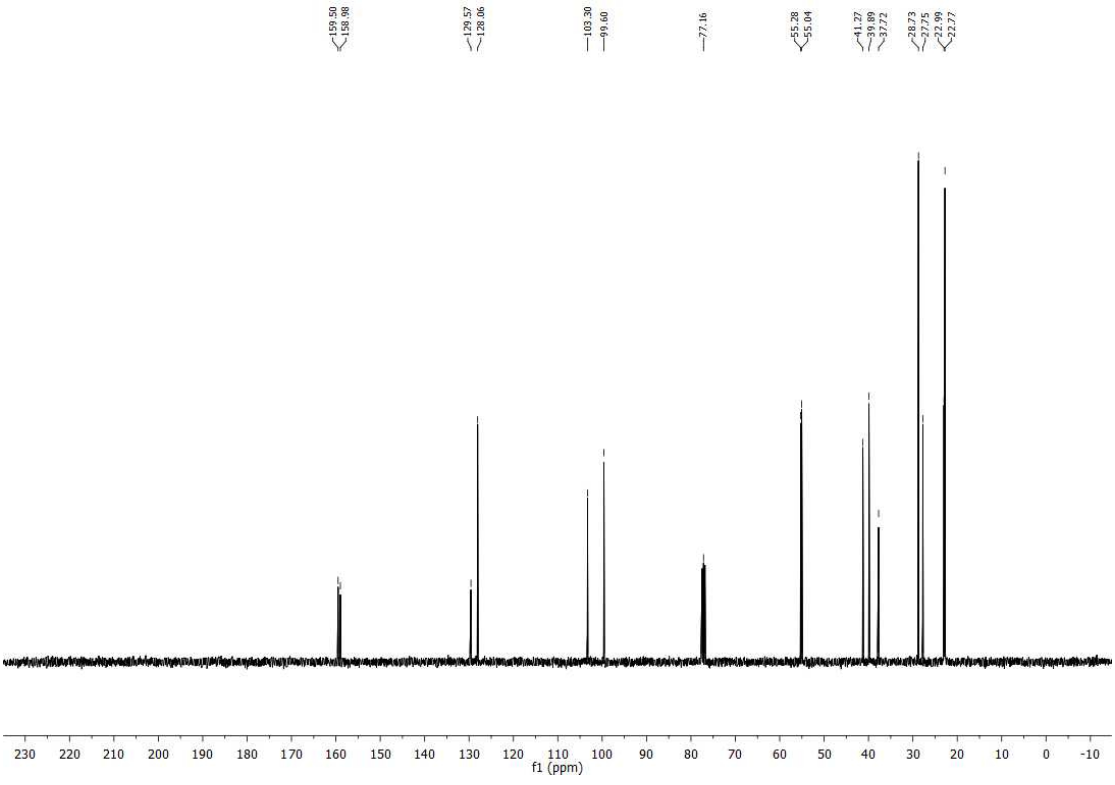

3.01
3.02
1.00
1.00
3.00
3.01
2.00
6.03
f1 (ppm)


159.32
159.21
140.27
130.40
128.67
128.33
127.41
125.56
103.35
99.56
77.16
55.29
55.08
46.37
38.70
28.41
f1 (ppm)

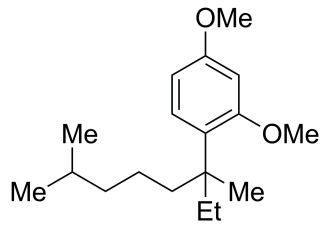

OMe
OMe
Me
Me
Me
Me

3e

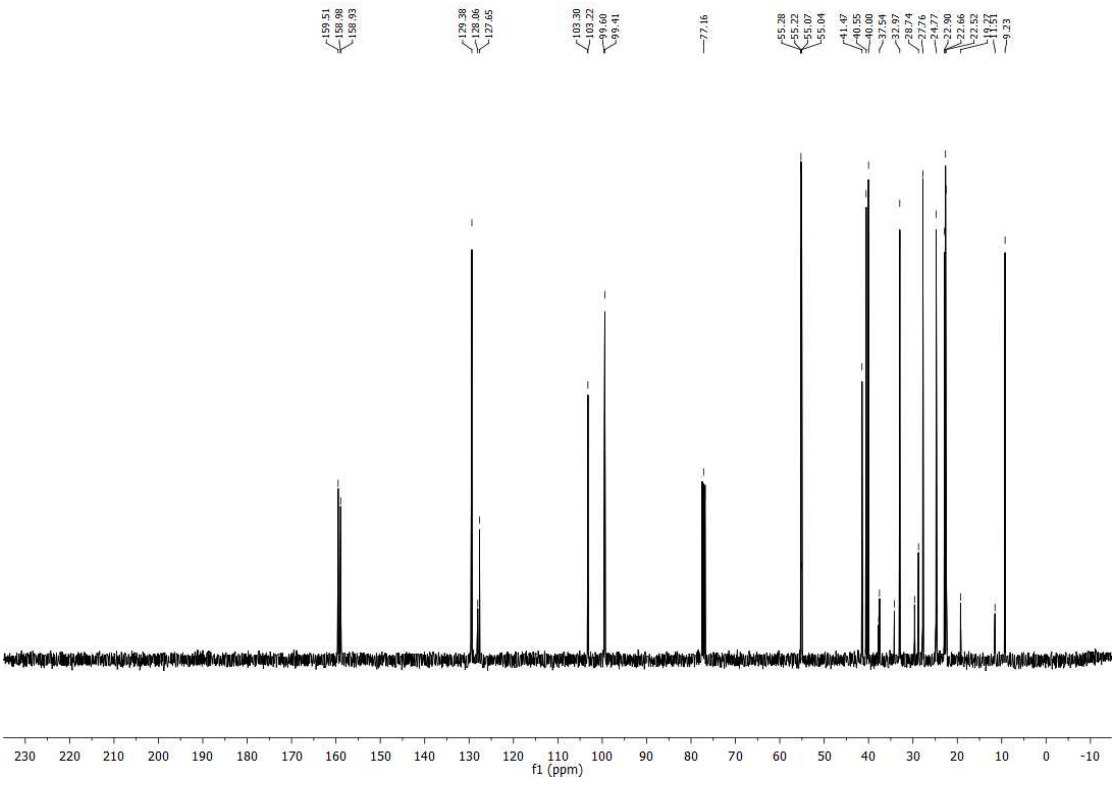
1.01
1.00
1.00
6.01
2.02
1.03
6.01
2.01
2.04
6.07
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

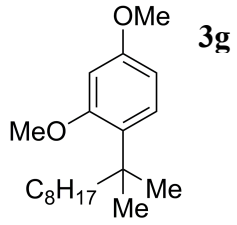

159.50
158.98
129.57
128.06
103.30
99.60
77.16
55.28
55.04
41.27
39.89
37.72
28.73
27.75
22.99
22.77
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

3f

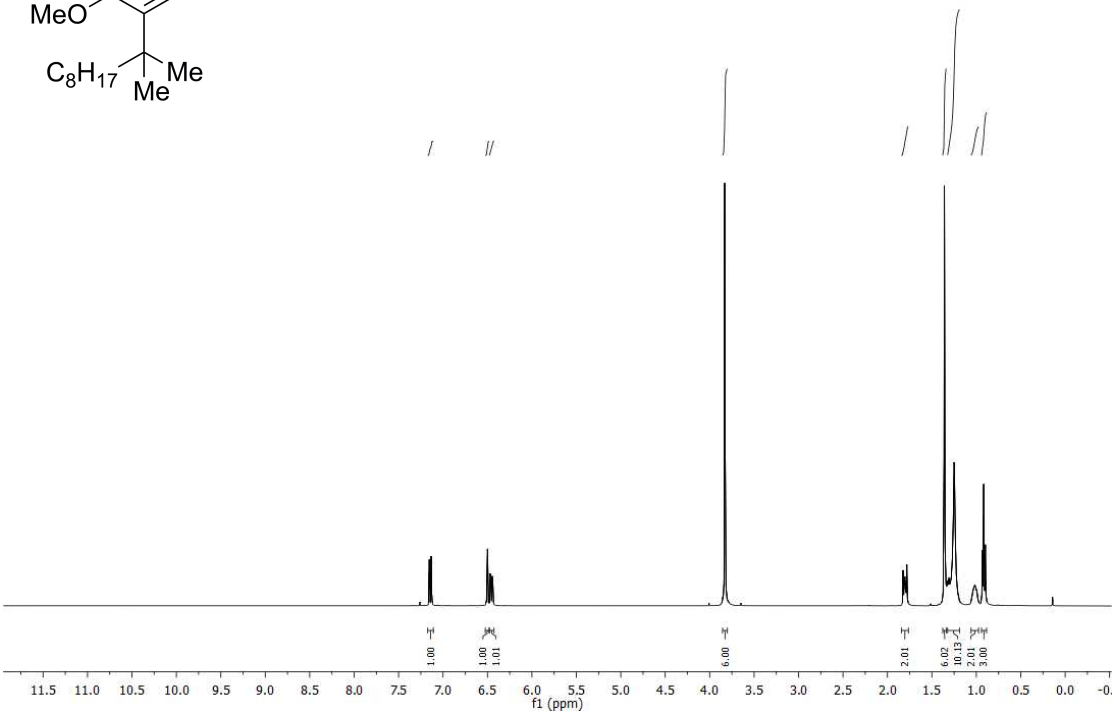

OMe
OMe
Me
Me
Me
Et

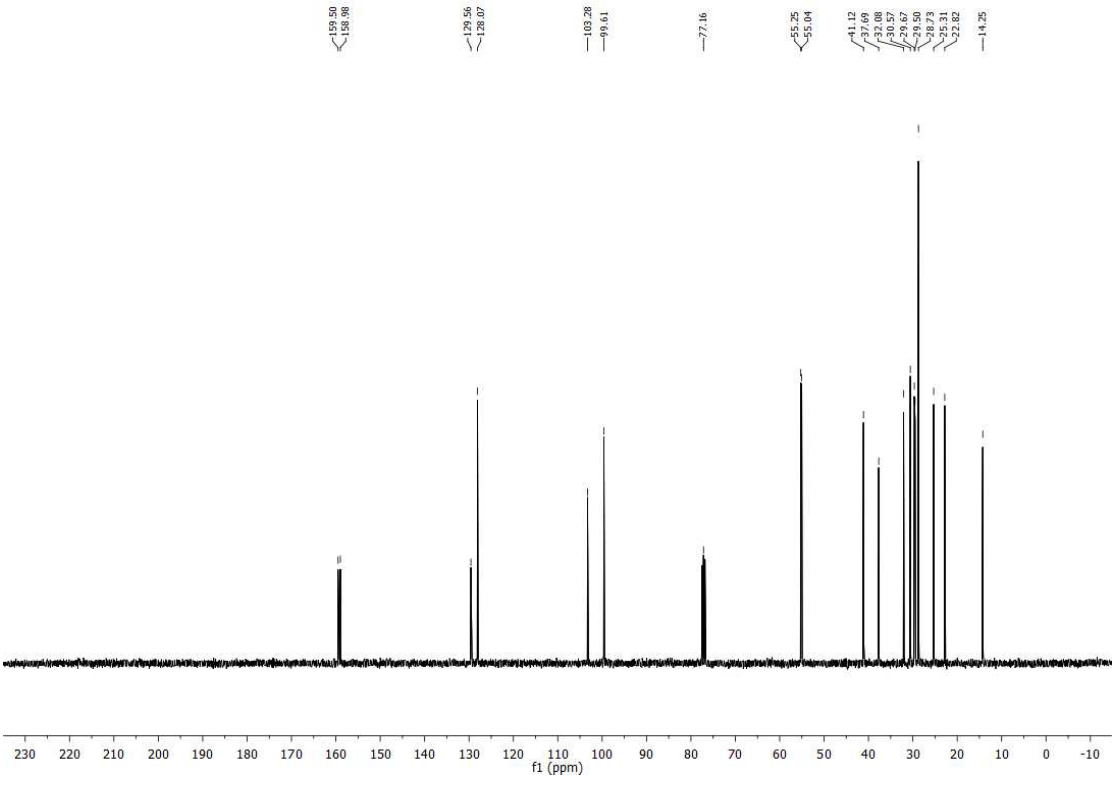
0.13
0.86
2.00
6.05
1.78
0.22
16.47
3.00
f1 (ppm)

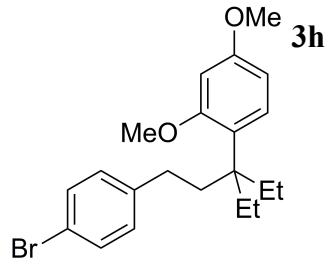

159.51
158.98
158.93
129.38
128.06
127.65
103.30
103.22
99.60
99.41
77.16
55.28
55.22
55.07
55.04
41.47
40.55
40.00
37.54
32.97
28.74
27.76
24.77
22.90
22.66
22.52
11.27
11.03
9.23
f1 (ppm)


OMe
MeO
C_8H_{17}
Me
Me

3g

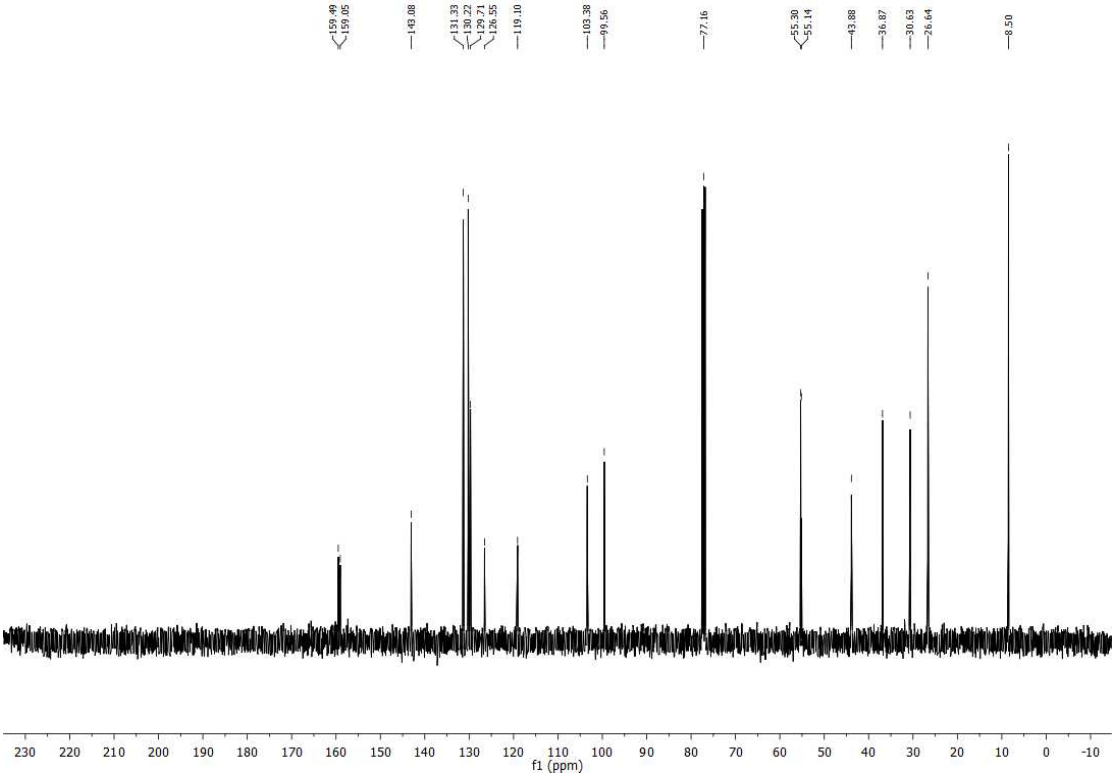

1.00
1.00
1.01
6.00
2.01
6.02
10.13
2.01
3.00
f1 (ppm)

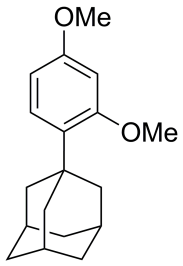

159.50
158.98
129.56
128.07
103.28
99.61
77.16
55.25
55.04
41.12
37.69
32.08
30.57
29.67
29.50
28.73
25.31
22.82
14.25
f1 (ppm)


OMe
MeO
Et
Et
Br

3h

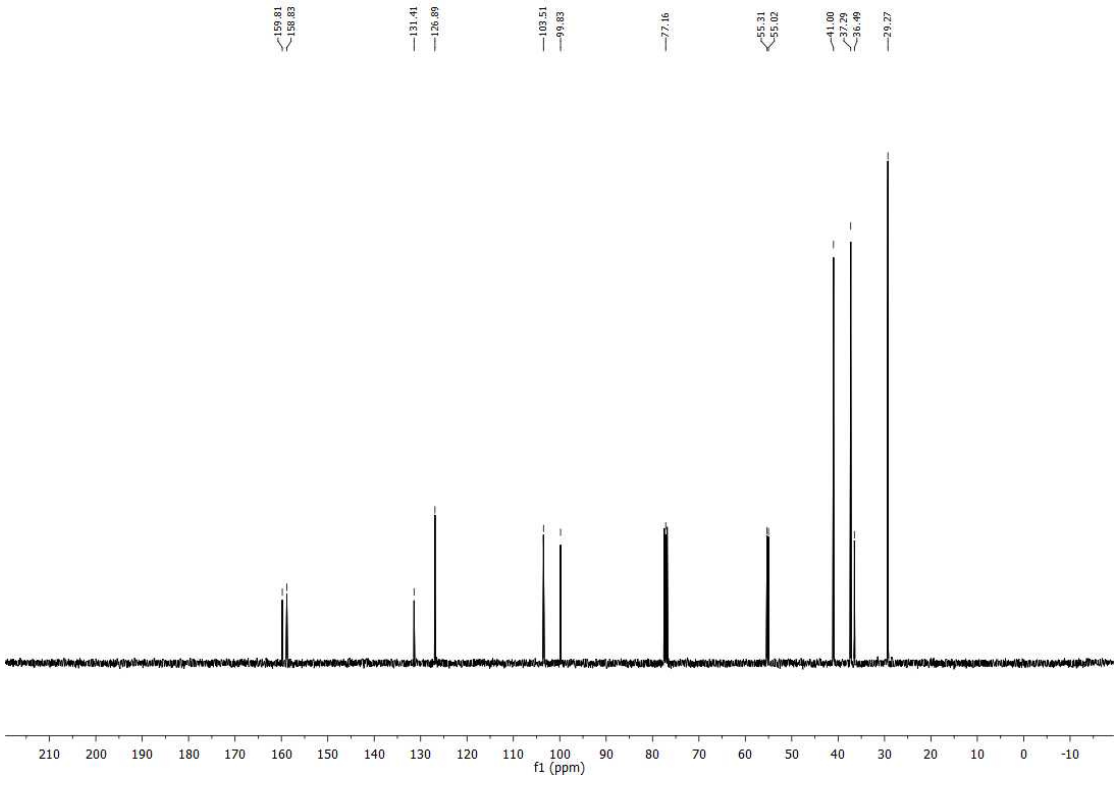
2.01
1.00
2.01
1.00
1.00
3.01
3.00
2.00
2.00
2.01
2.01
6.05
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

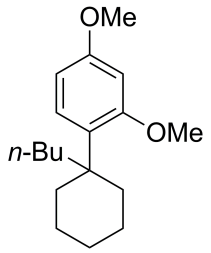

159.49
159.05
143.08
131.33
130.22
129.71
126.55
119.10
103.38
99.56
77.16
55.30
55.14
43.88
36.87
30.63
26.64
8.50
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


OMe
OMe

3i

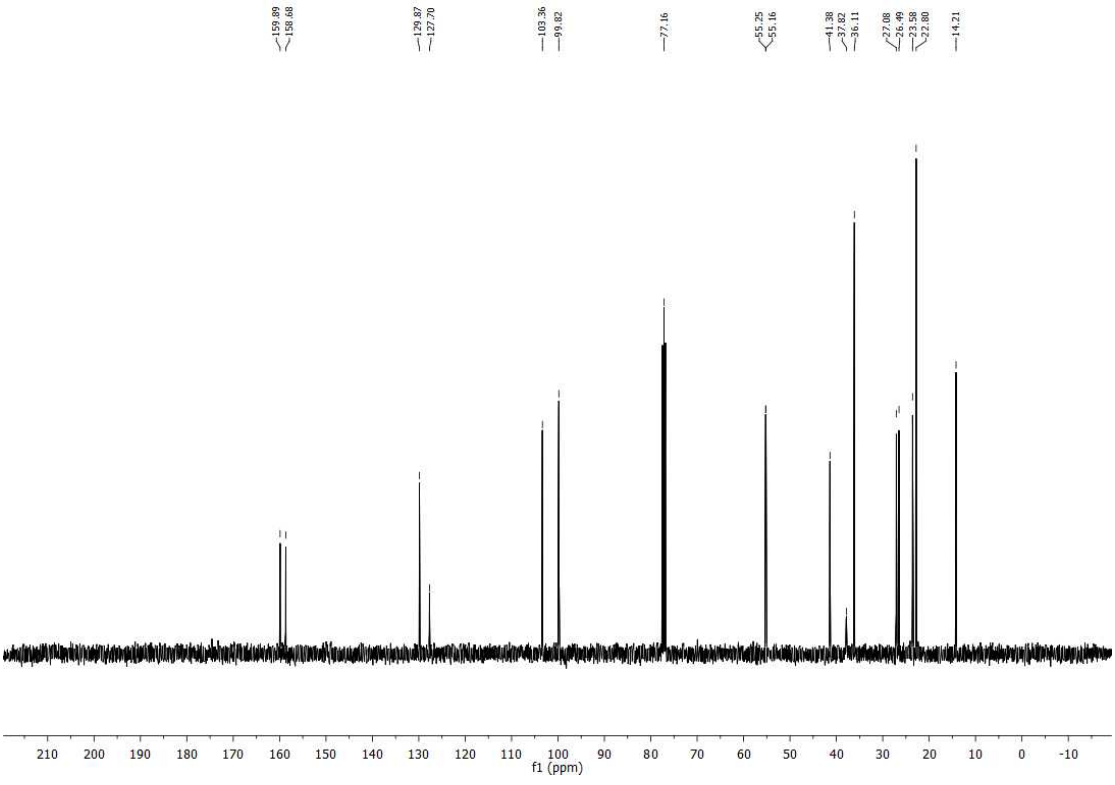
1.00
1.00
1.00
3.00
3.01
9.02
6.00
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

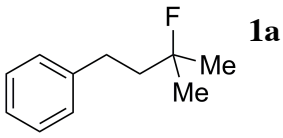

159.81
158.83
131.41
126.89
103.51
99.83
77.16
55.31
55.02
41.00
37.29
36.49
29.27
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


OMe
OMe
n-Bu

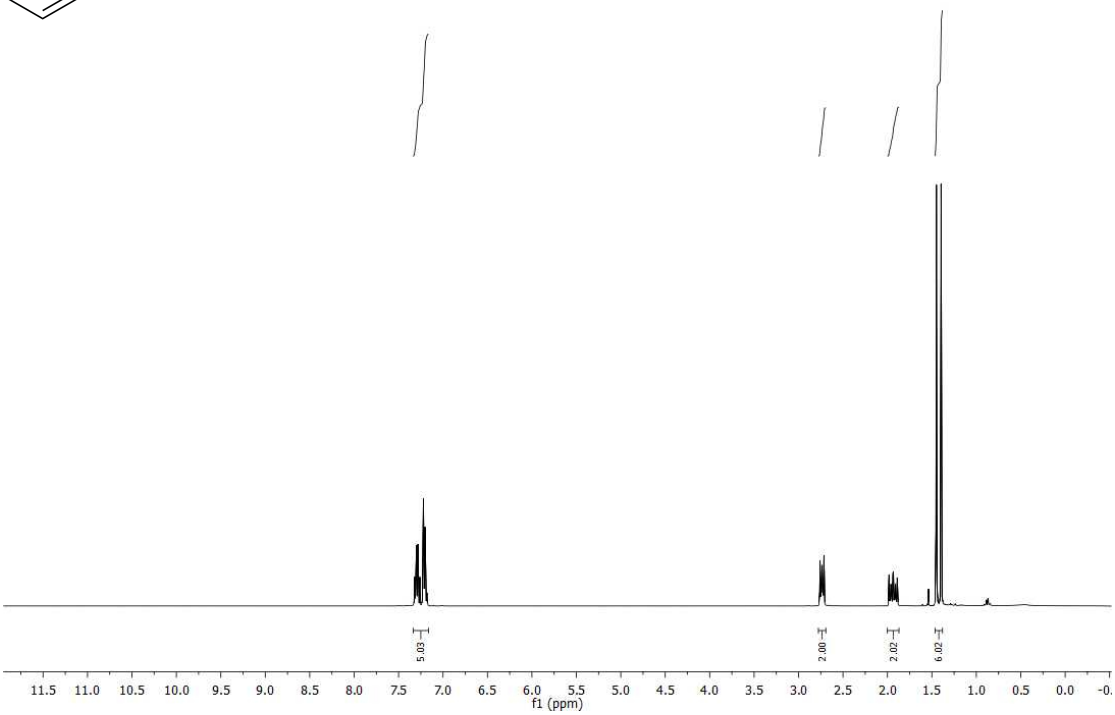
3j

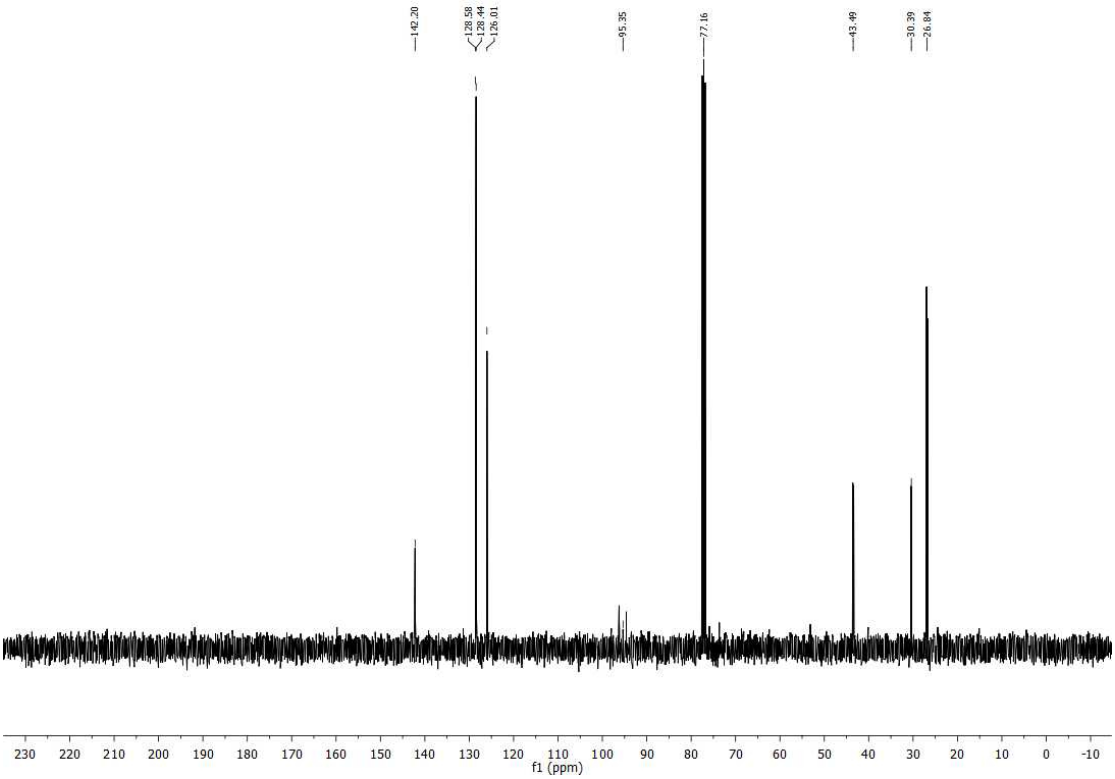
1.00
2.00
3.01
3.02
2.00
4.00
2.11
4.01
2.02
2.01
3.03
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

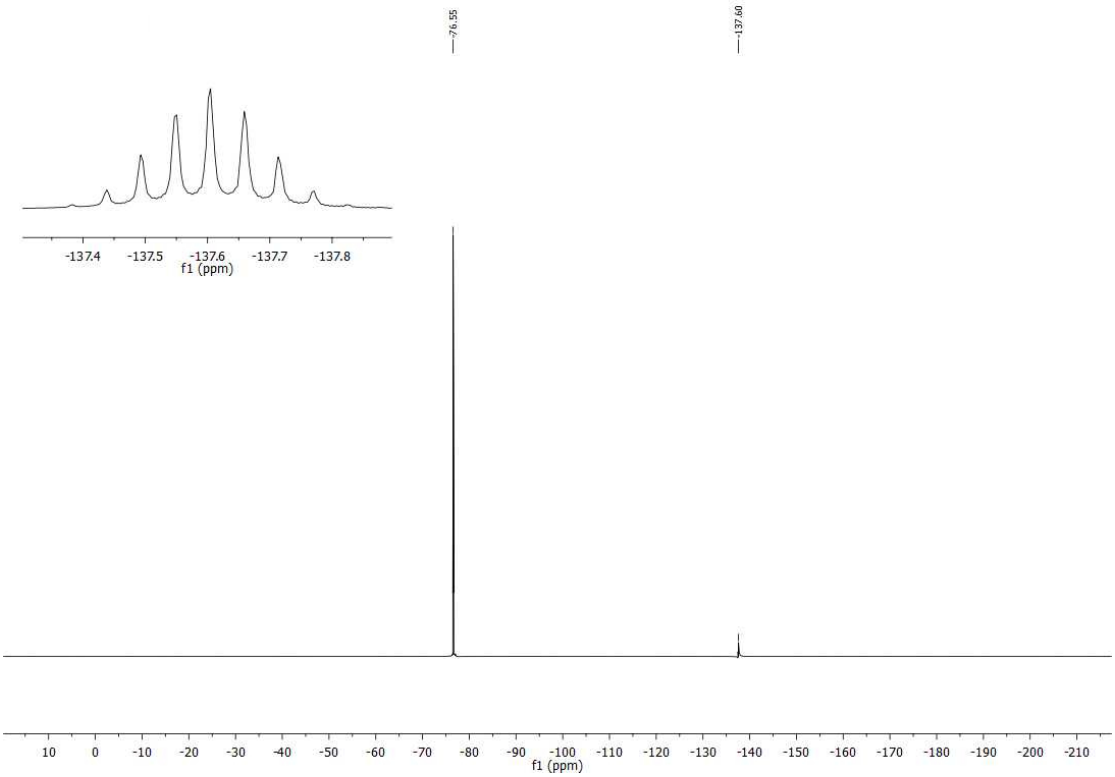

159.89
158.68
129.87
127.70
103.36
99.82
77.16
55.25
55.16
41.38
37.22
36.11
27.08
26.49
23.58
22.80
14.21
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

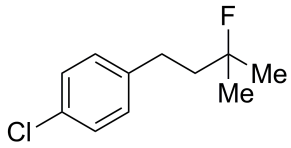

F
Me
Me

1a

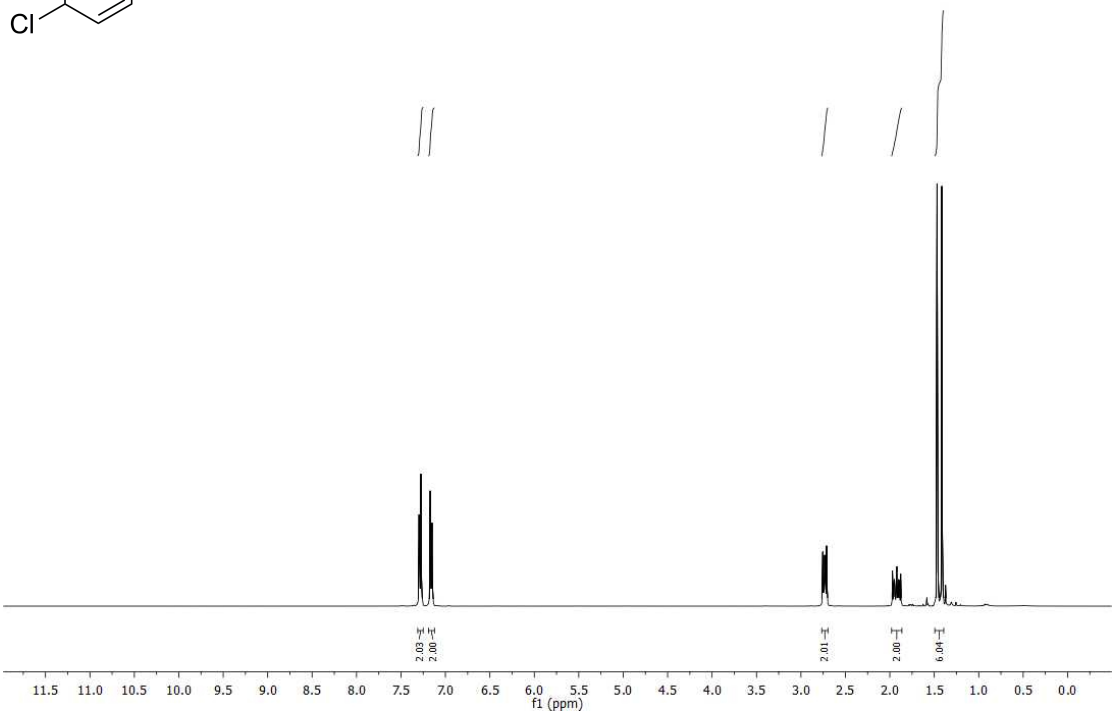

5.03
2.00
2.02
6.02
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

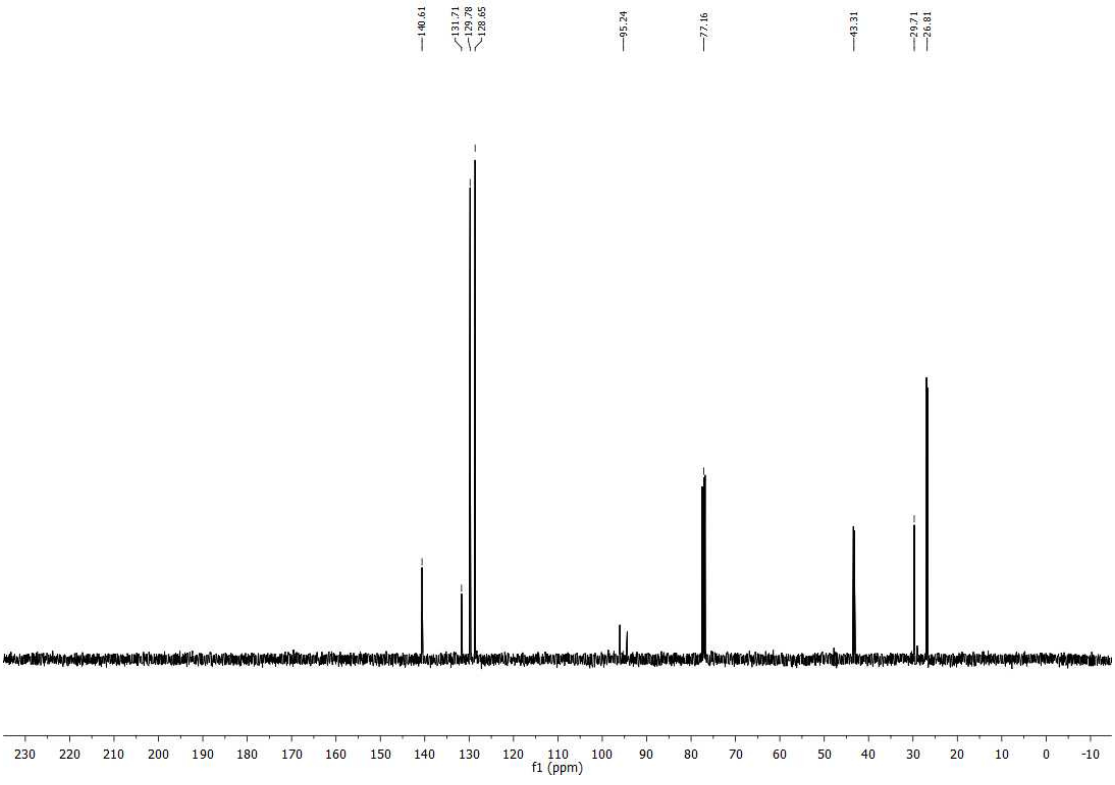

142.20
128.58
128.44
126.01
95.35
77.16
43.49
30.39
26.84
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

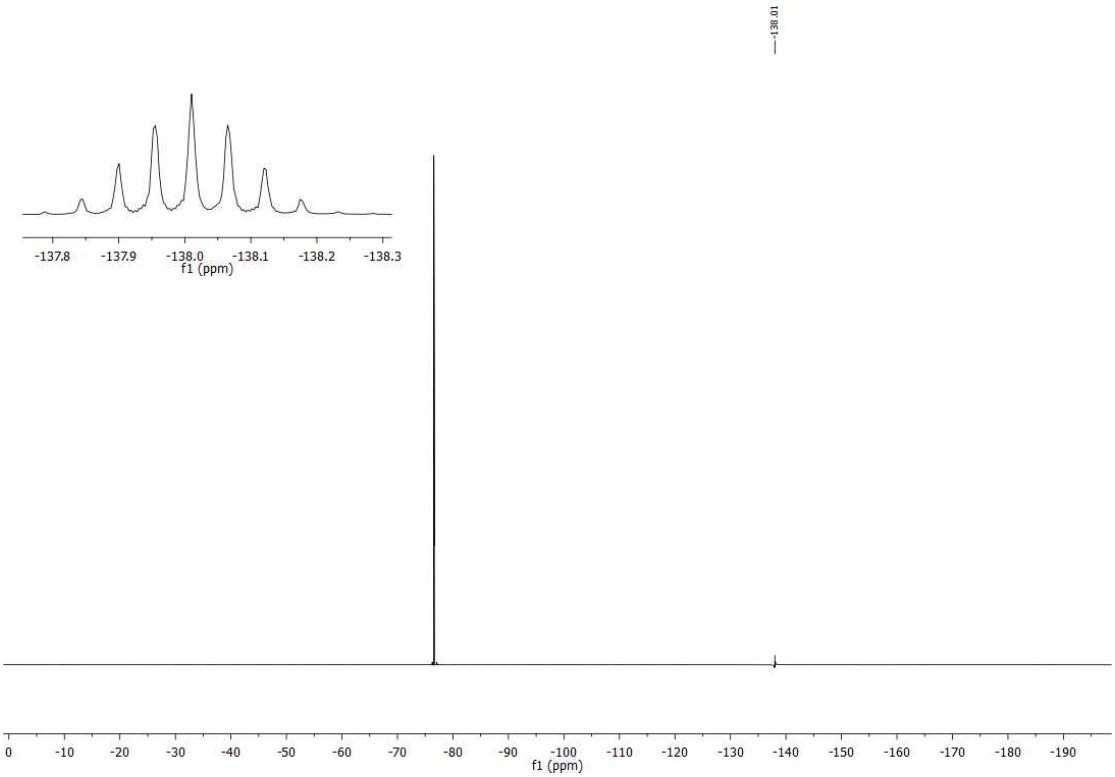

-76.55
-137.60
-137.4
-137.5
-137.6
-137.7
-137.8
f1 (ppm)
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

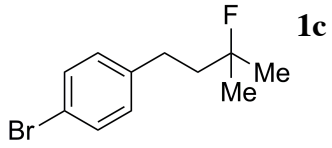

F
Me
Me
Cl

1b

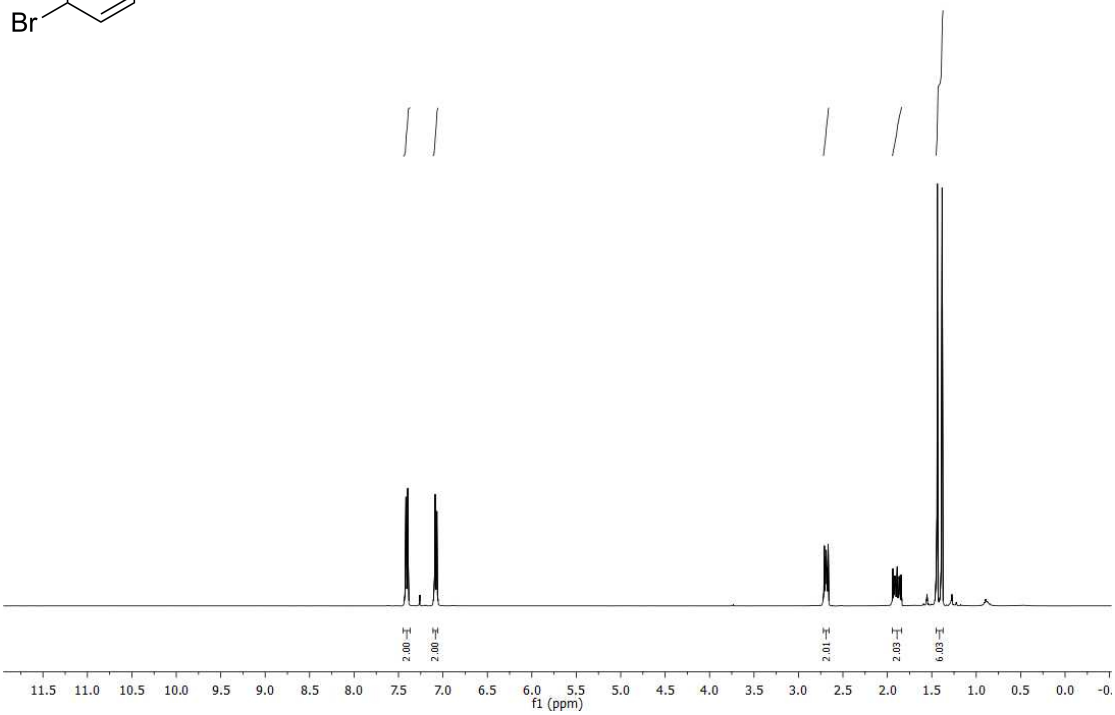

2.03
2.00
2.01
2.00
6.04
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

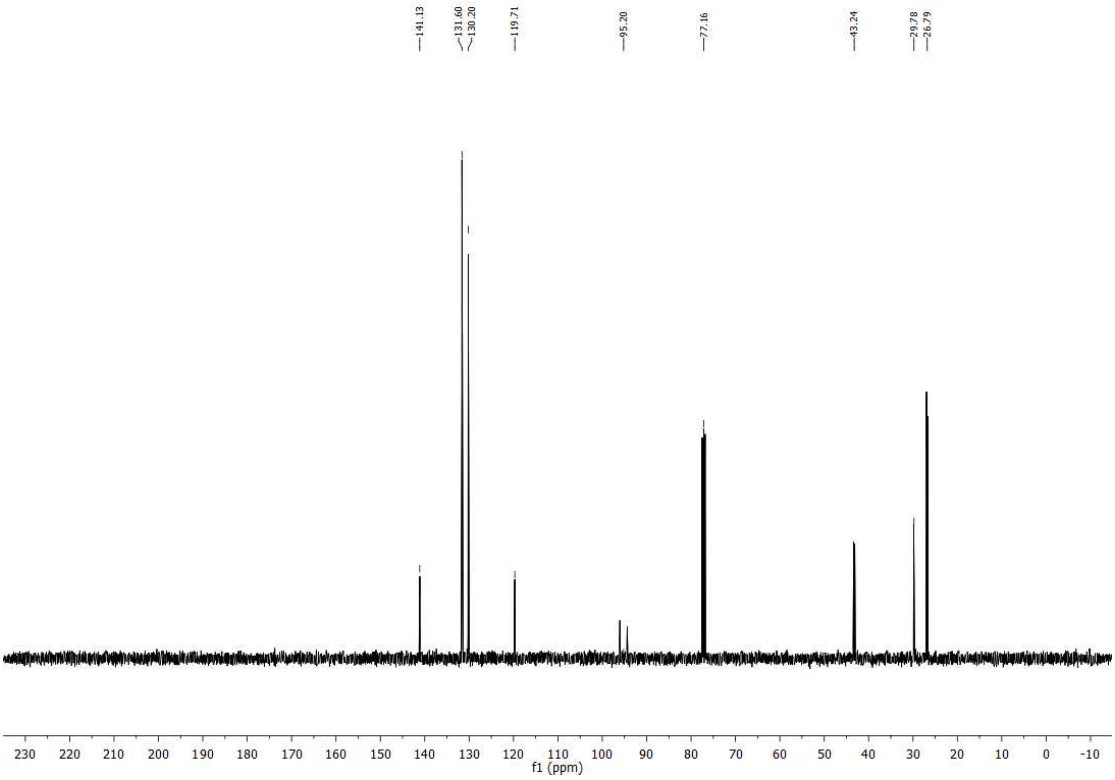

140.61
131.71
129.78
128.65
95.24
77.16
43.31
29.71
26.81
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

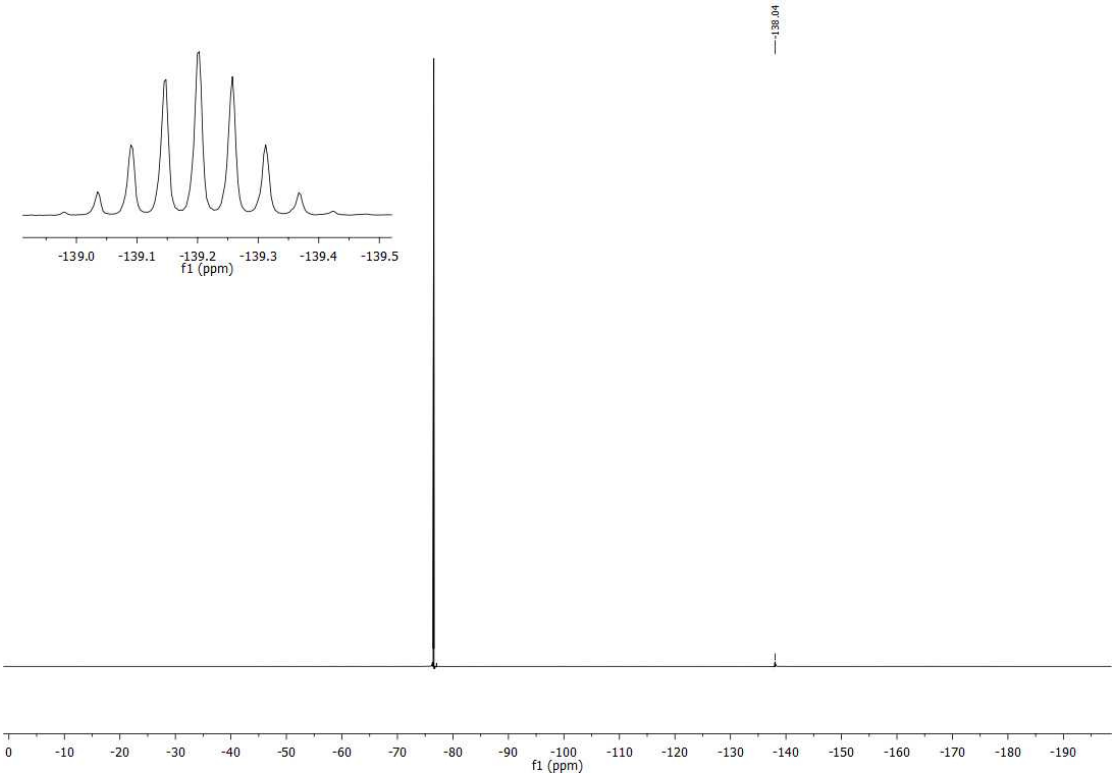

-138.01
-137.8
-137.9
-138.0
-138.1
-138.2
-138.3
f1 (ppm)
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)

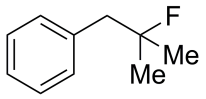

F
Me
Me
Br

1c

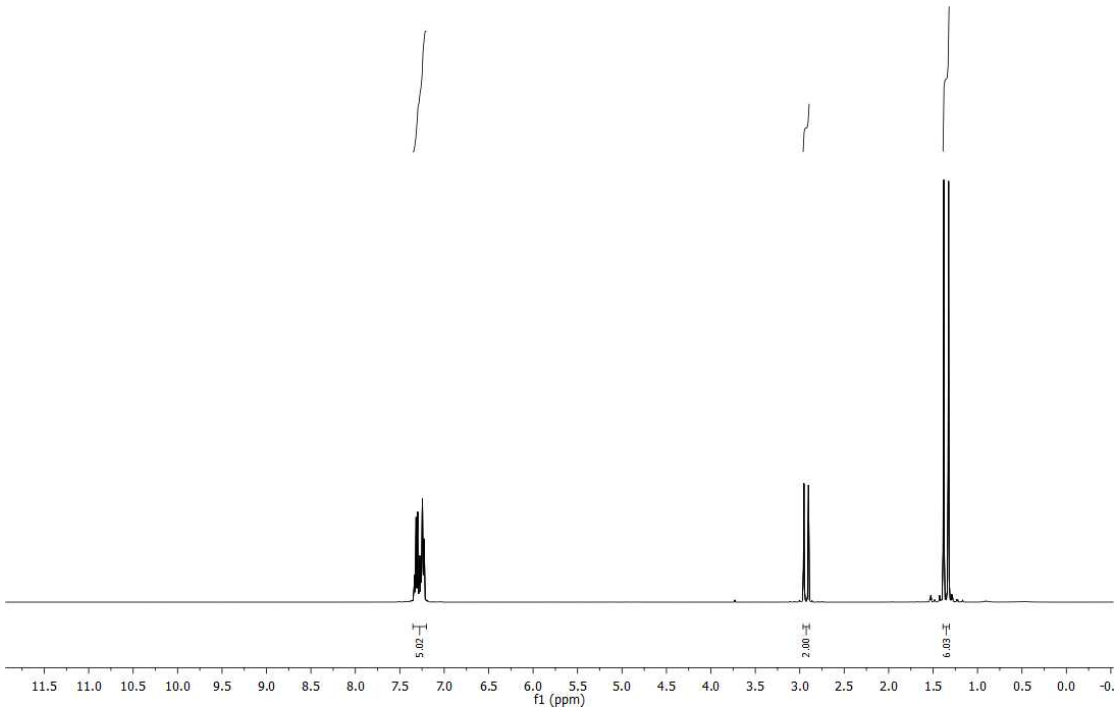

2.00
2.00
2.01
2.03
6.03
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

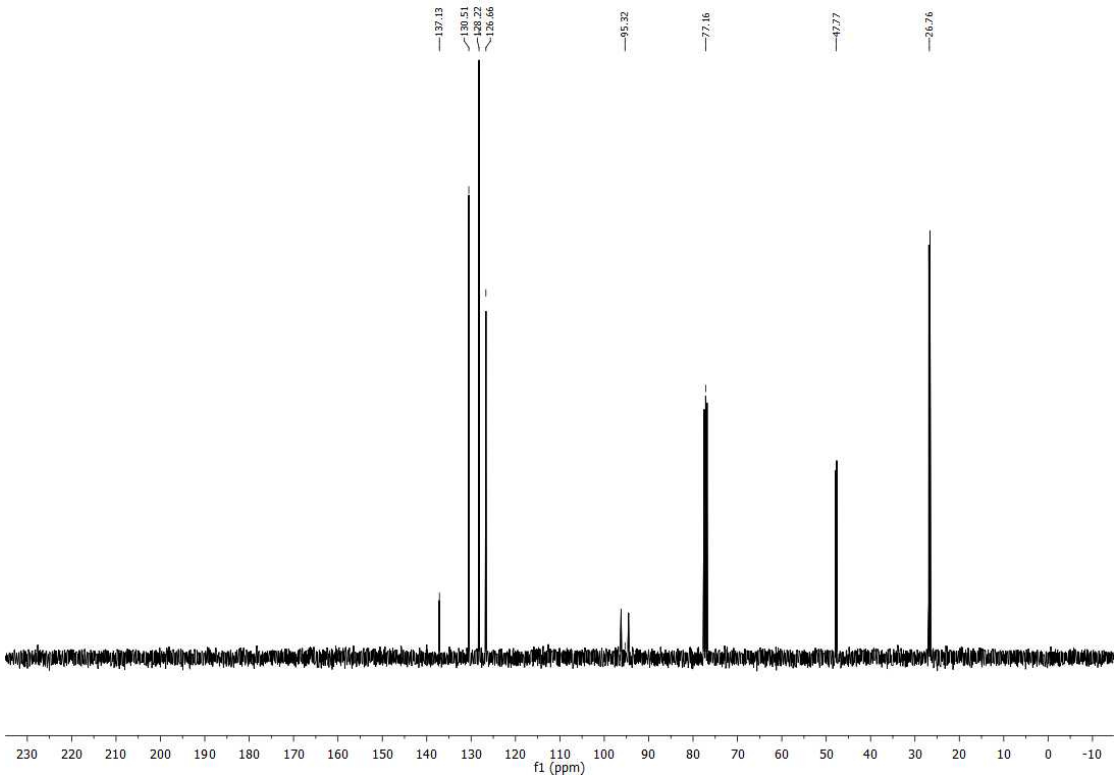

141.13
131.60
130.20
119.71
95.20
77.16
43.24
29.78
26.79
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

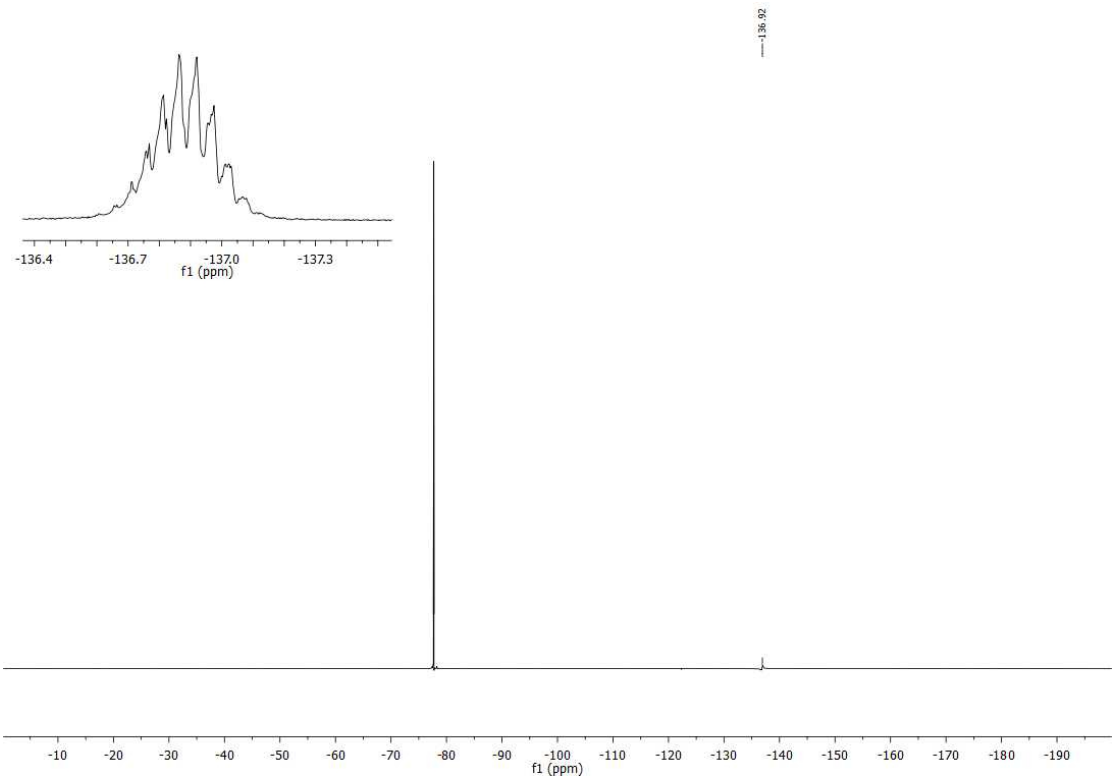

-138.04
-139.0
-139.1
-139.2
-139.3
-139.4
-139.5
f1 (ppm)
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)

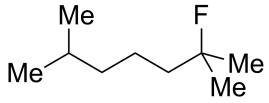

F
Me
Me

1d

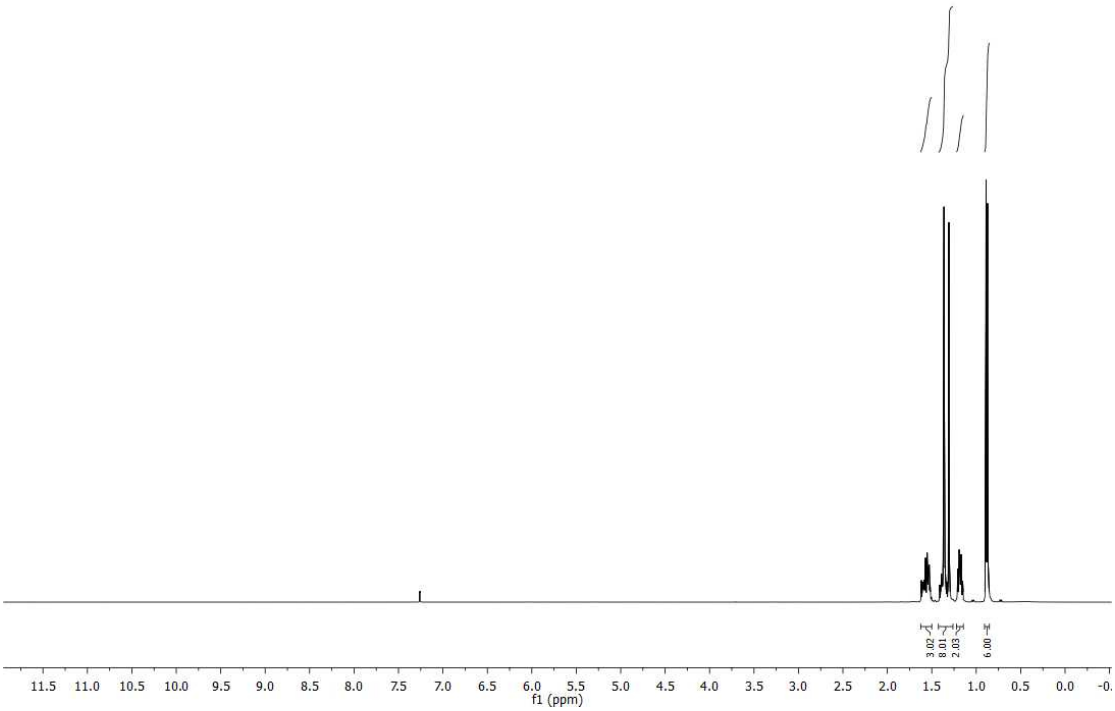

5.02
2.00
6.03
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

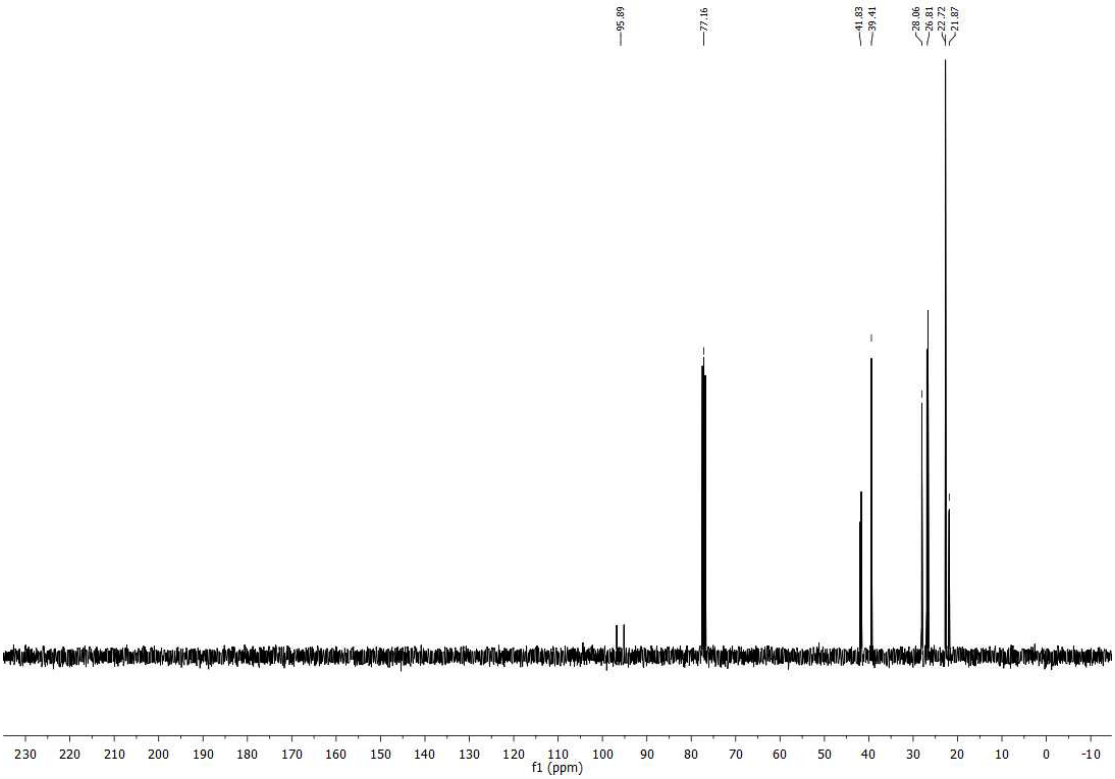

137.13
130.51
128.22
126.66
95.32
77.16
47.77
26.76
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

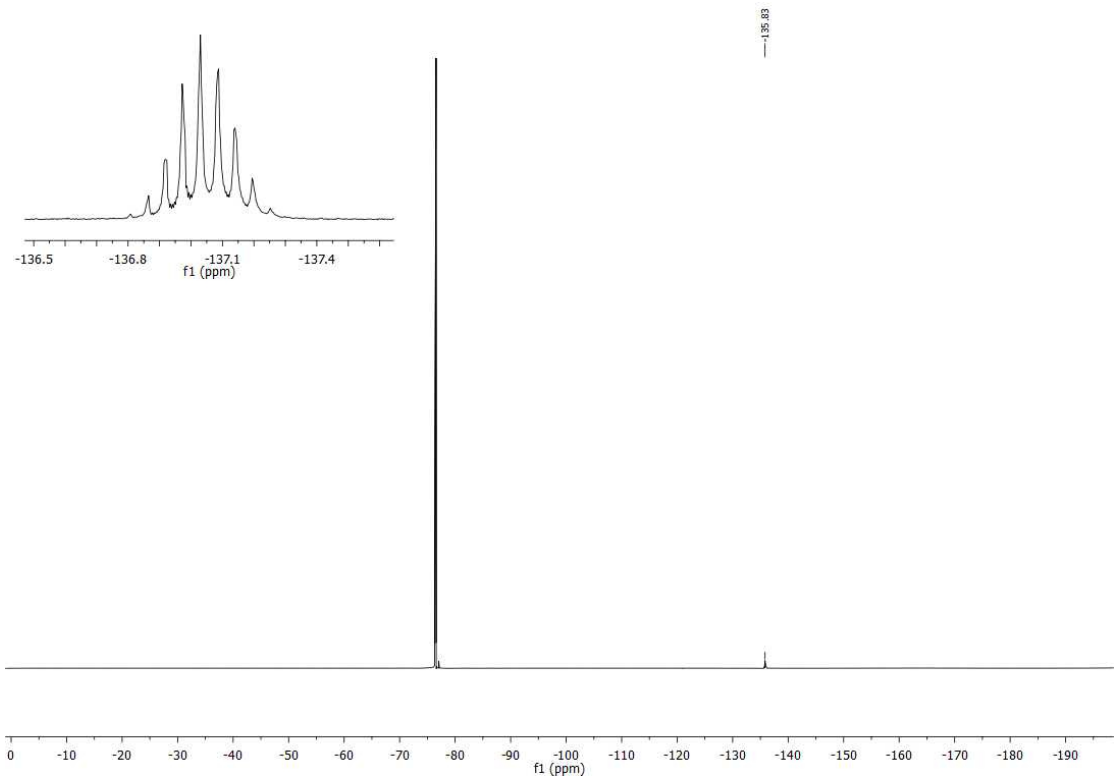

-136.92
-136.4
-136.7
-137.0
-137.3
f1 (ppm)
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)

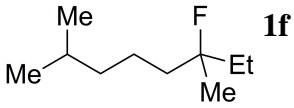

Me
F
Me
Me
Me

1e

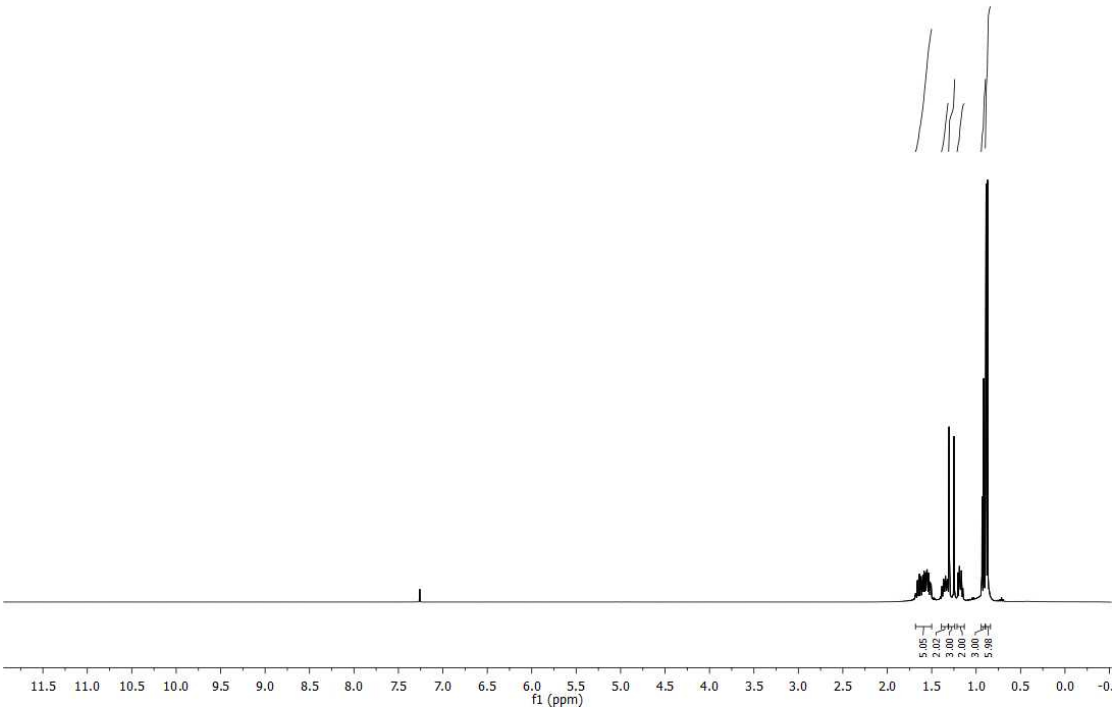

3.02
8.01
2.03
6.00
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

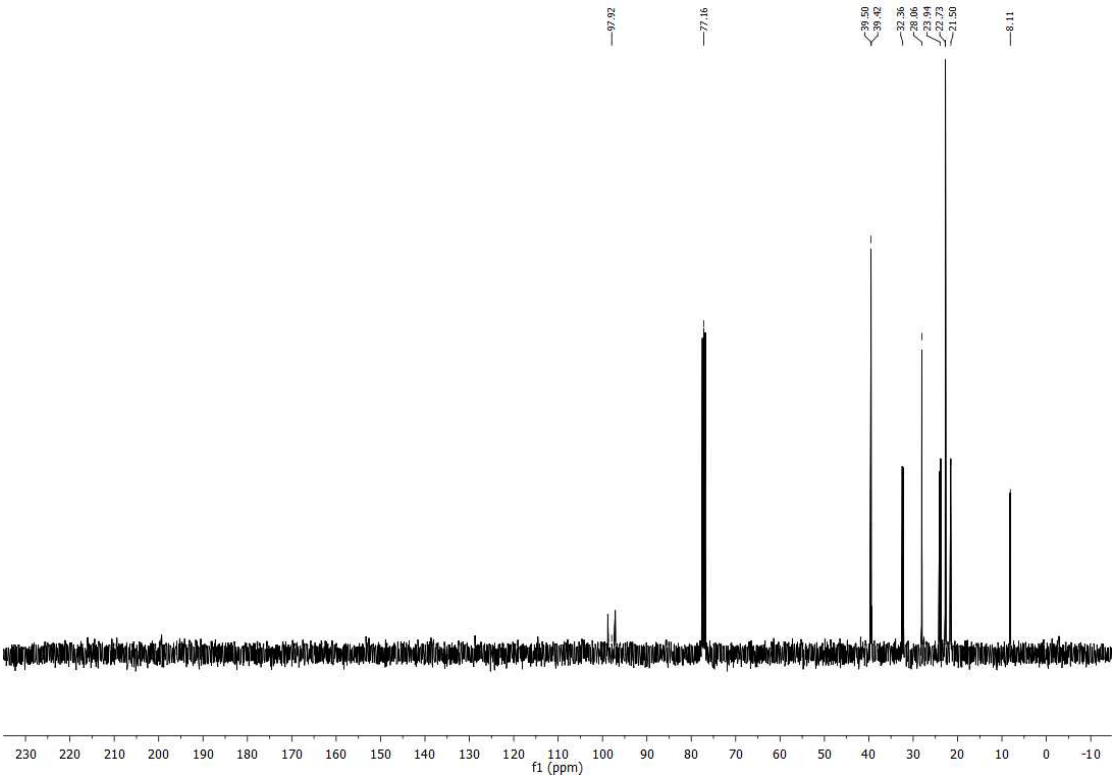

95.89
77.16
41.83
39.41
28.06
26.81
22.72
21.87
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

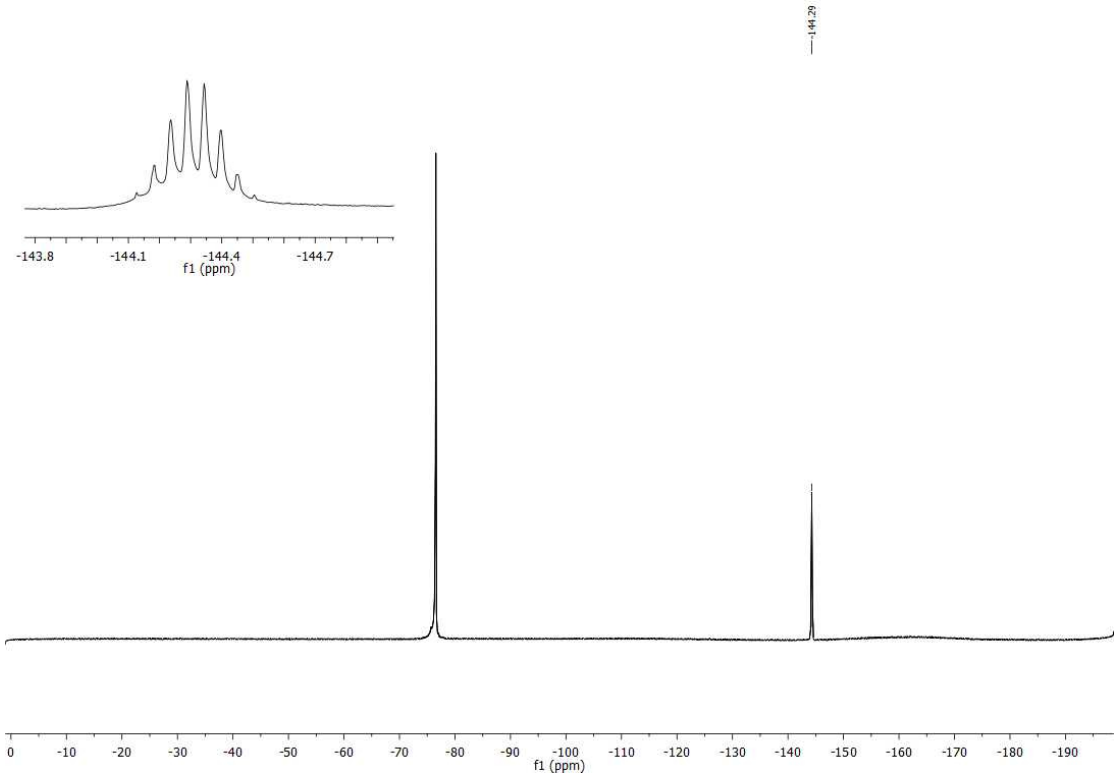

-135.83
-136.5
-136.8
-137.1
-137.4
f1 (ppm)
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)

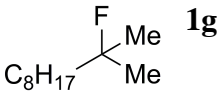

Me
F
Et
Me
Me

1f

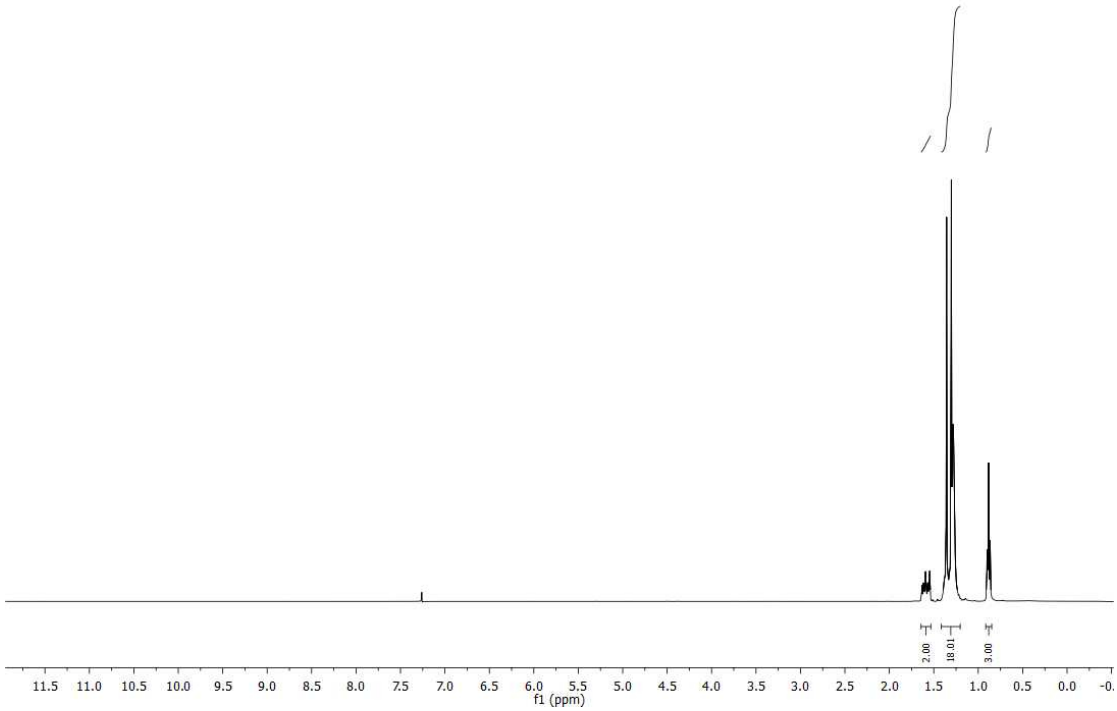

5.05
2.02
3.00
2.00
3.00
5.98
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

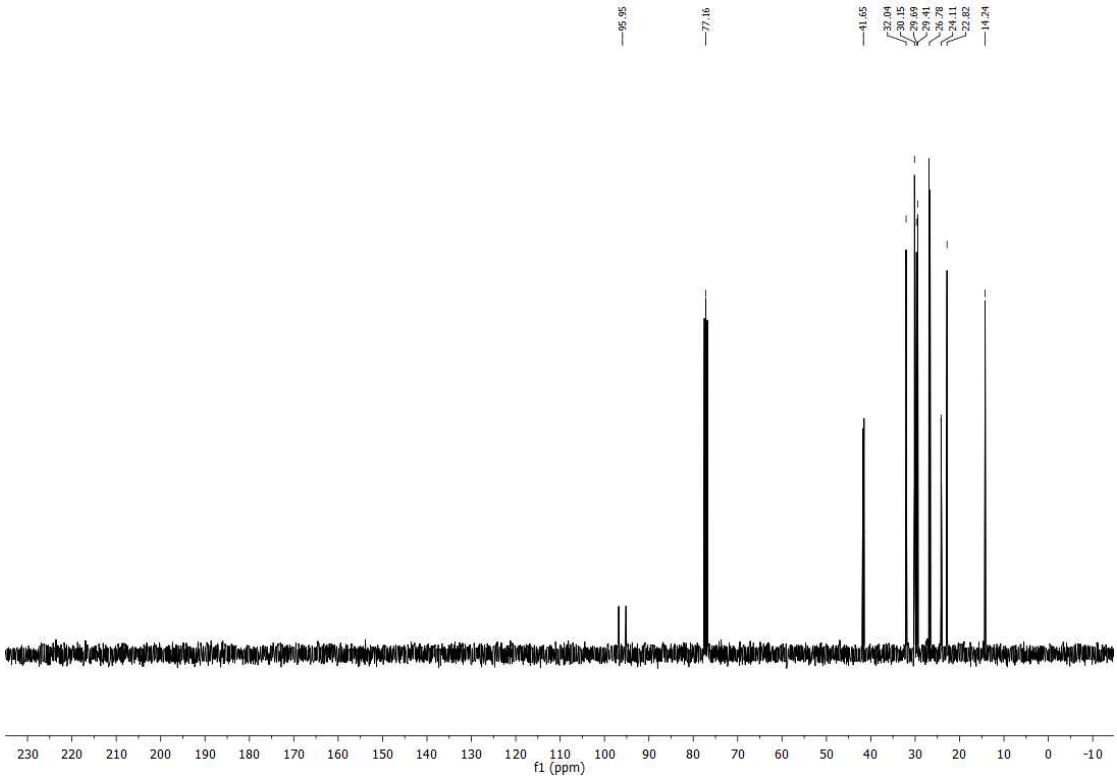

97.92
77.16
39.50
39.42
32.36
28.06
23.94
23.73
21.73
21.50
8.11
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

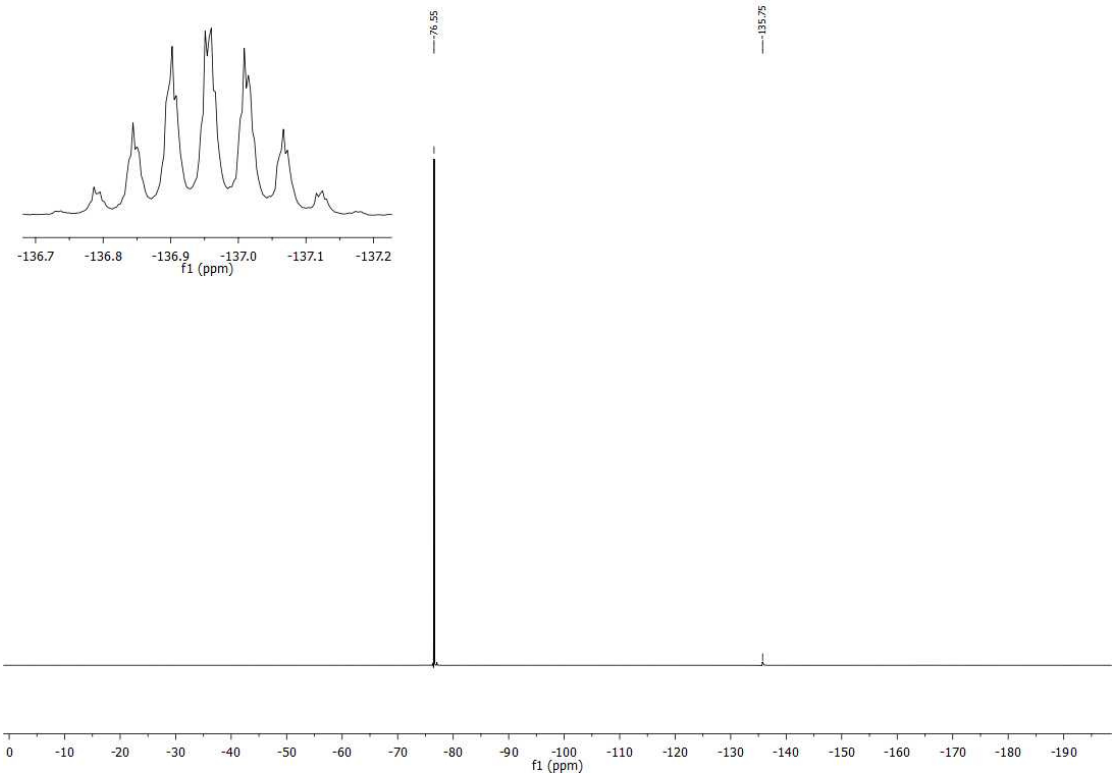

-144.29
-143.8
-144.1
-144.4
-144.7
f1 (ppm)
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)

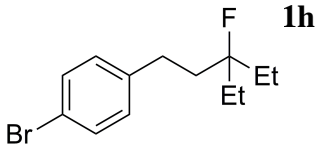

F
Me
Me
C8H17

1g

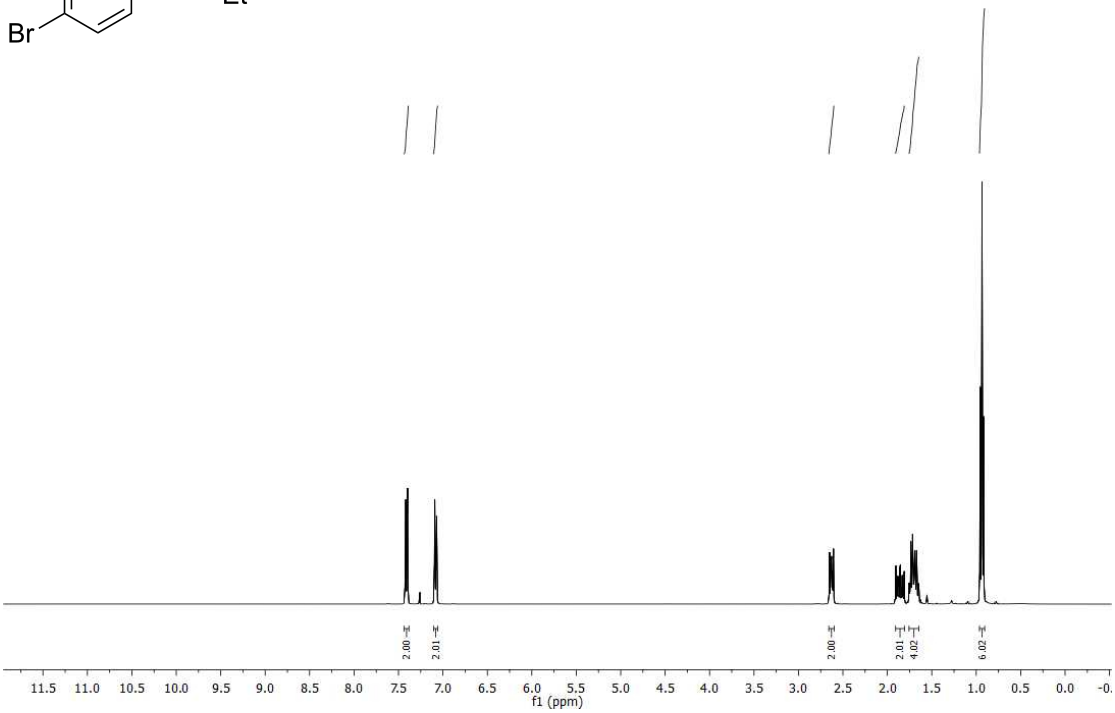

2.00
18.01
3.00
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

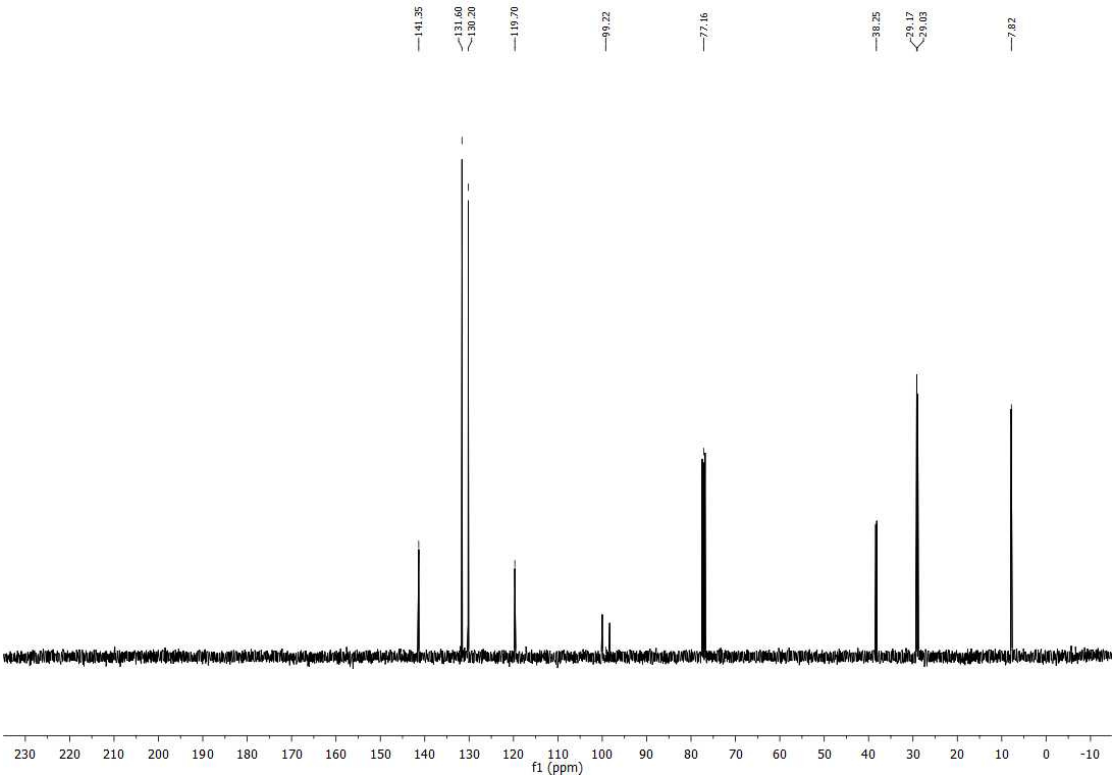

95.95
77.16
41.65
32.04
30.15
29.69
29.41
26.78
24.11
22.82
14.24
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

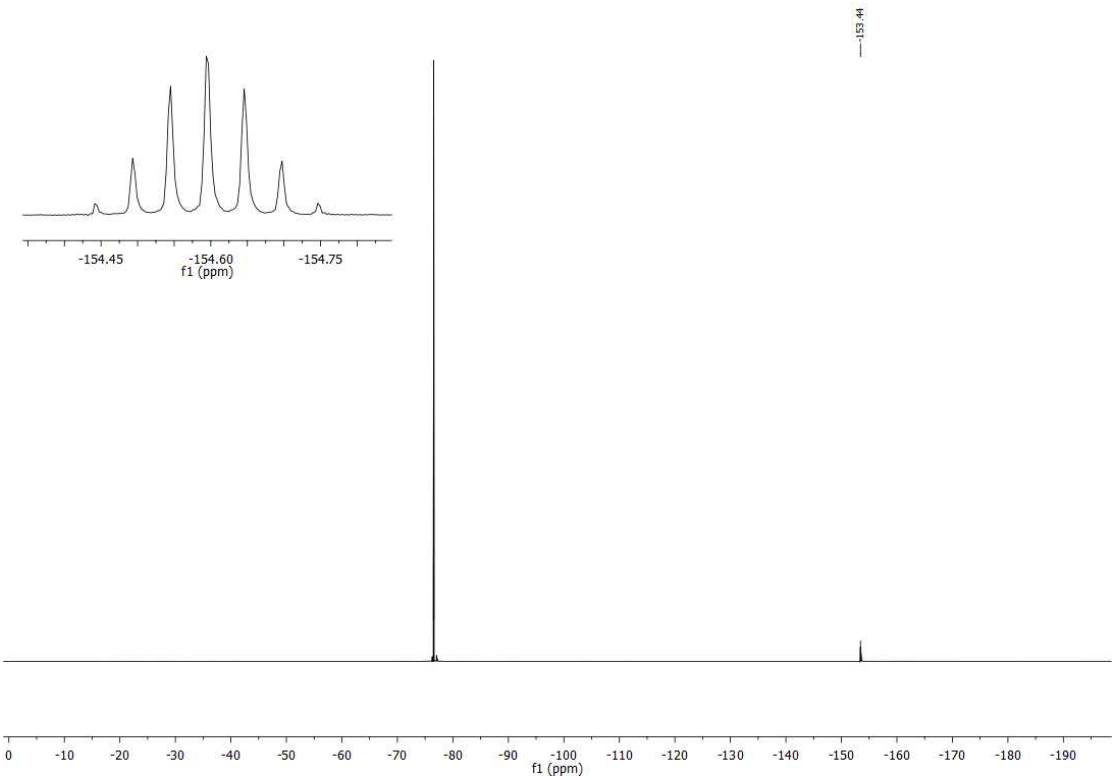

-76.55
-135.75
-136.7
-136.8
-136.9
-137.0
-137.1
-137.2
f1 (ppm)
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)


F
Et
Et
Br

1h


2.00
2.01
2.00
2.01
4.02
6.02
11.5 11.0 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
f1 (ppm)


141.35
131.60
130.20
119.70
99.22
77.16
38.25
29.17
29.03
7.82
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


-153.44
-154.45
-154.60
-154.75
f1 (ppm)
0
-10
-20
-30
-40
-50
-60
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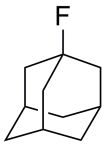
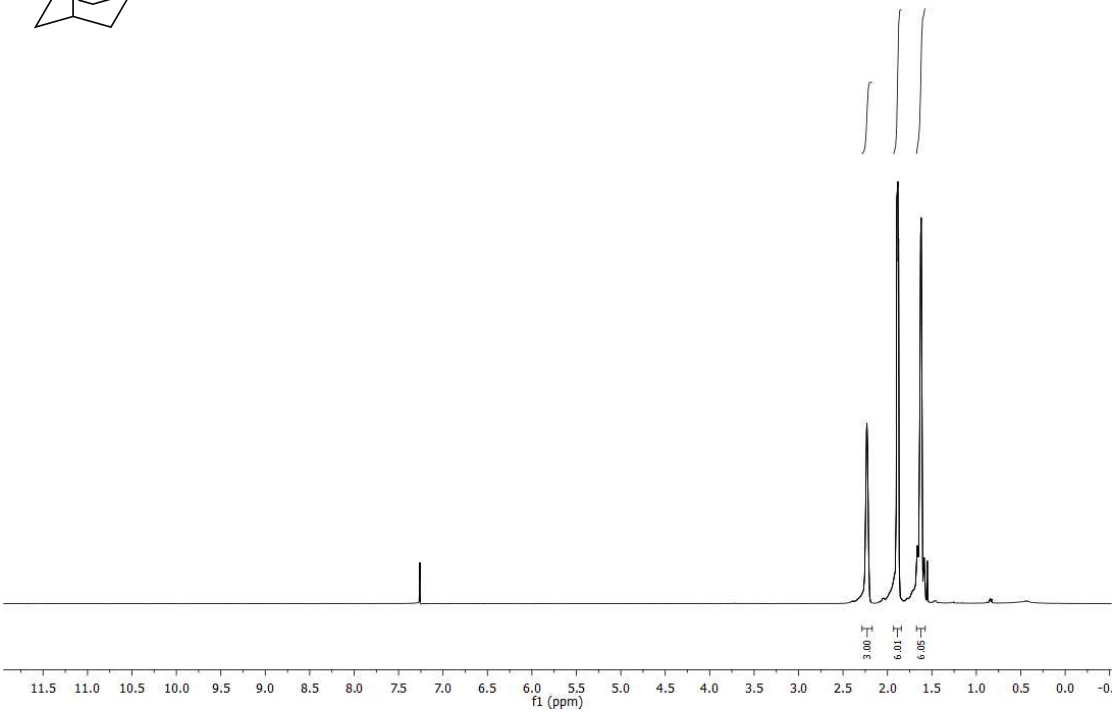
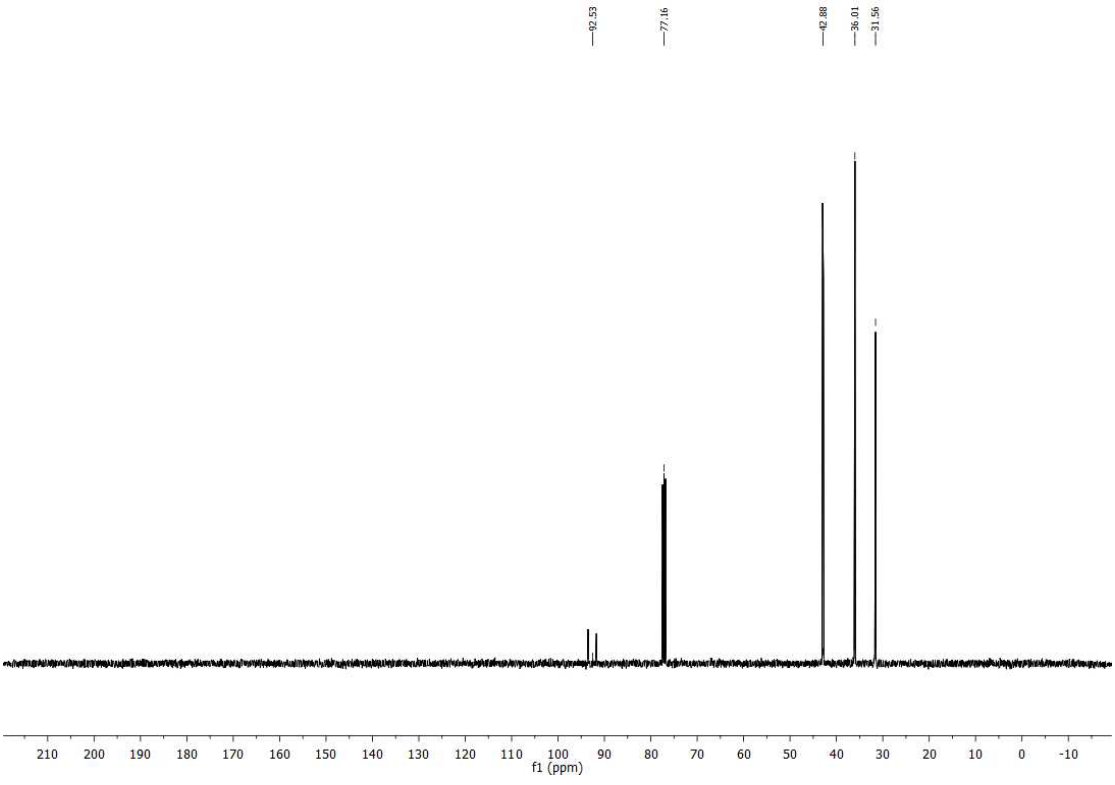
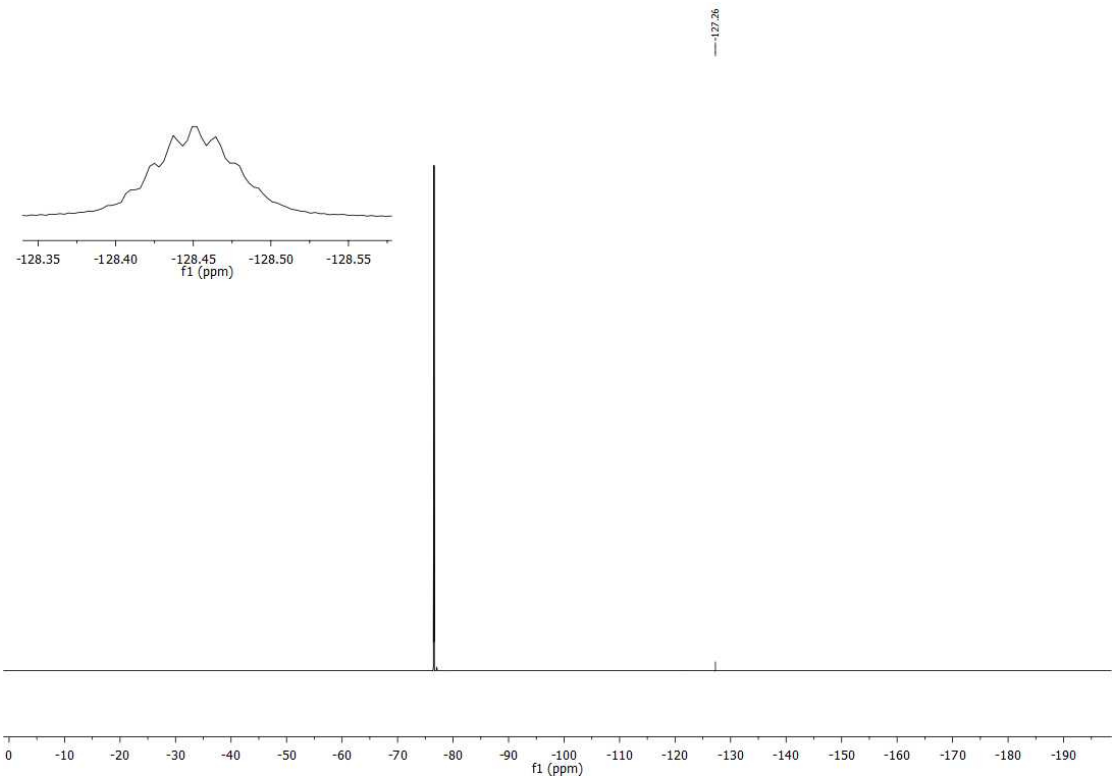
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)

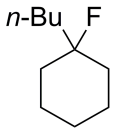

F

1i

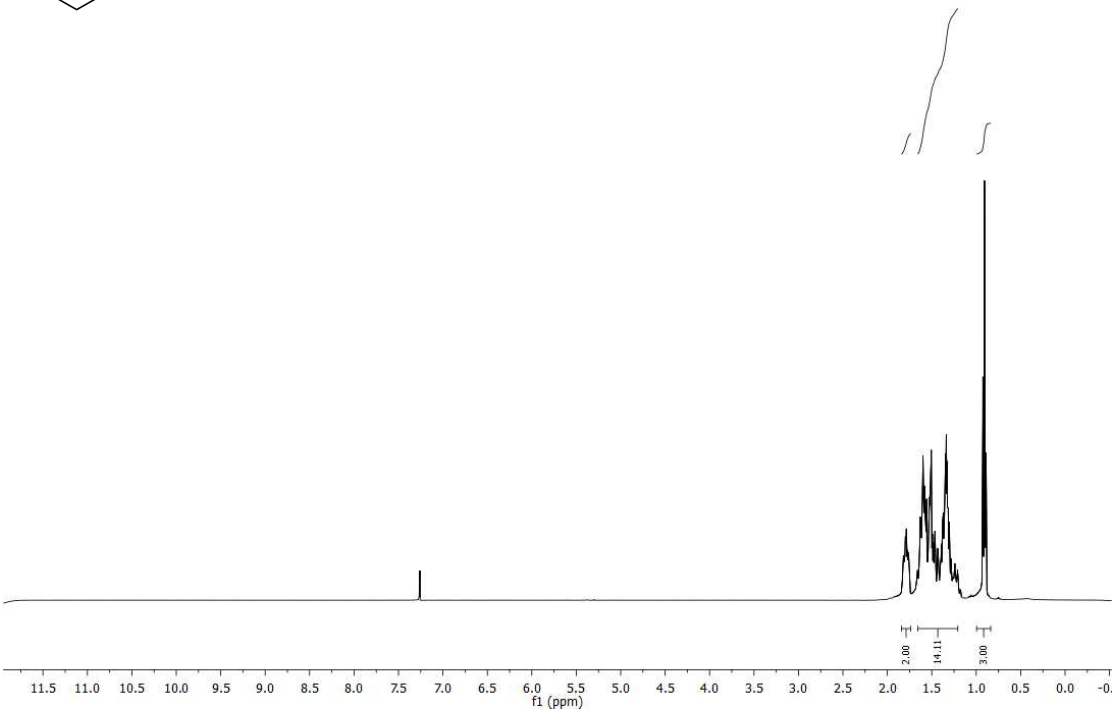

3.00
6.01
6.05
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

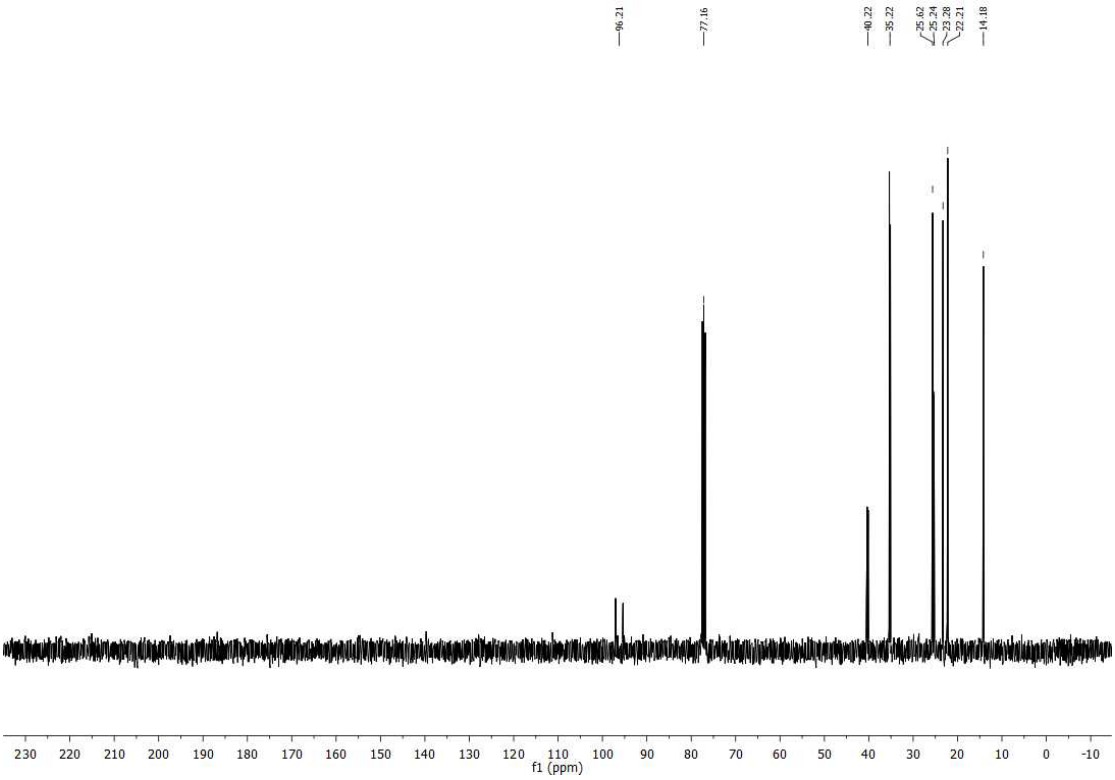

92.53
77.16
42.88
36.01
31.56
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

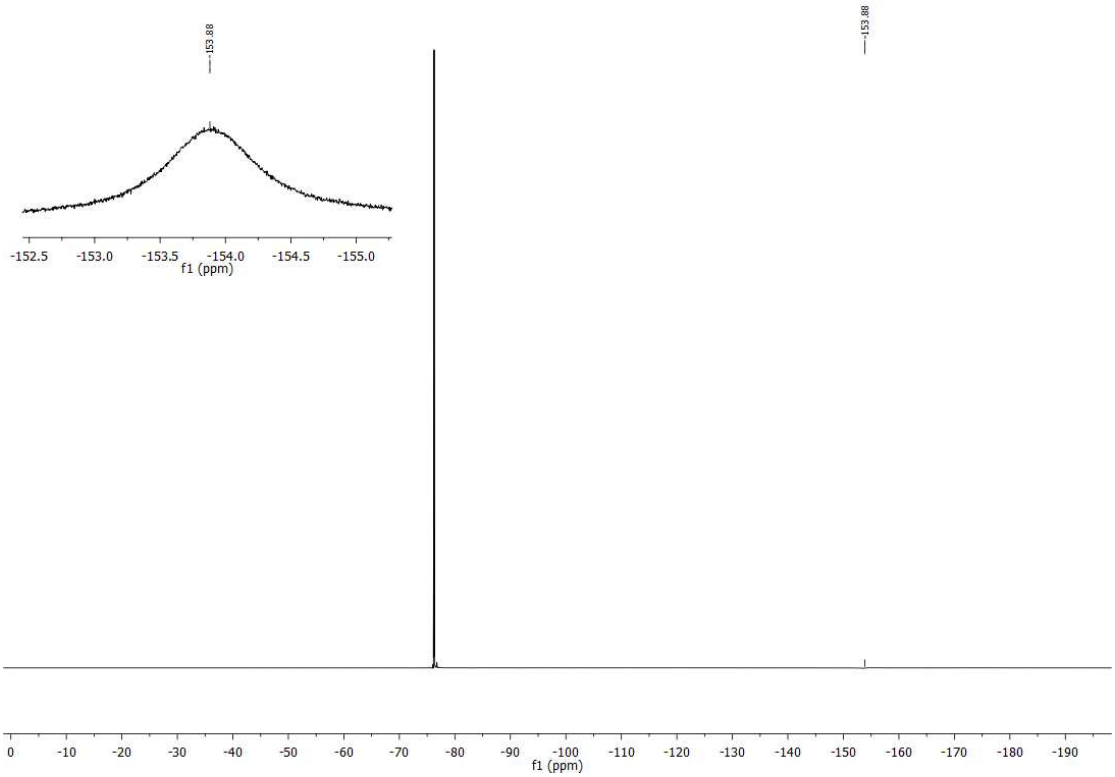

-127.26
-128.35
-128.40
-128.45
-128.50
-128.55
f1 (ppm)
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)

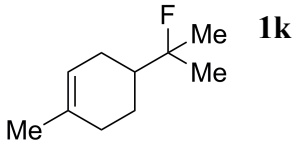

n-Bu
F

1j

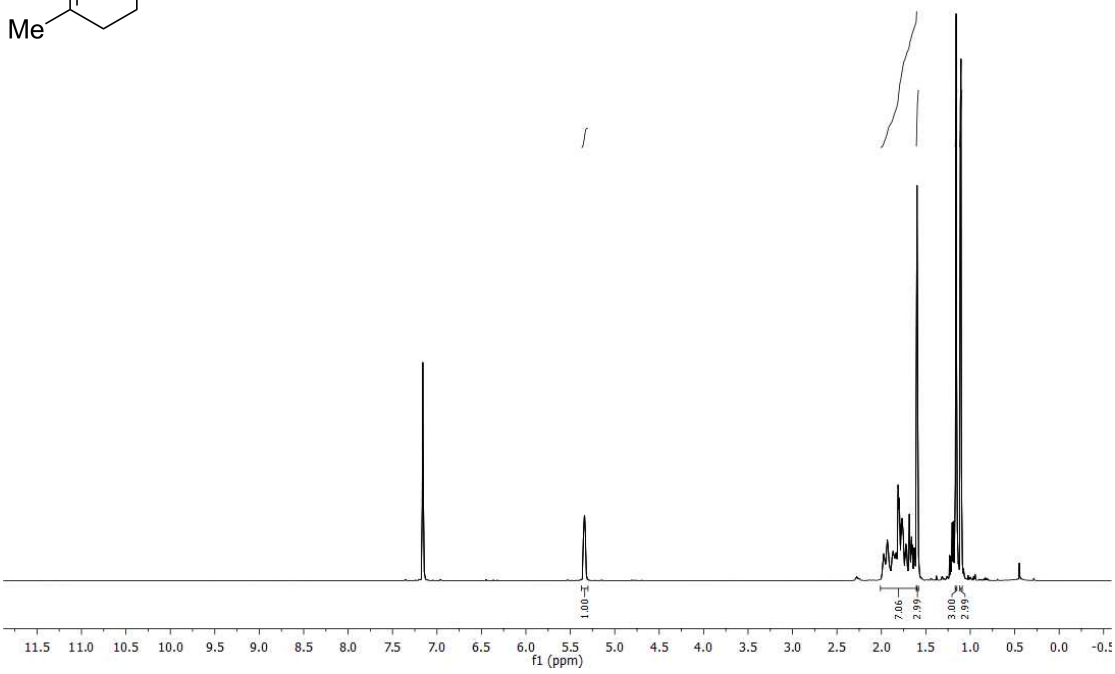

2.00
14.11
3.00
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

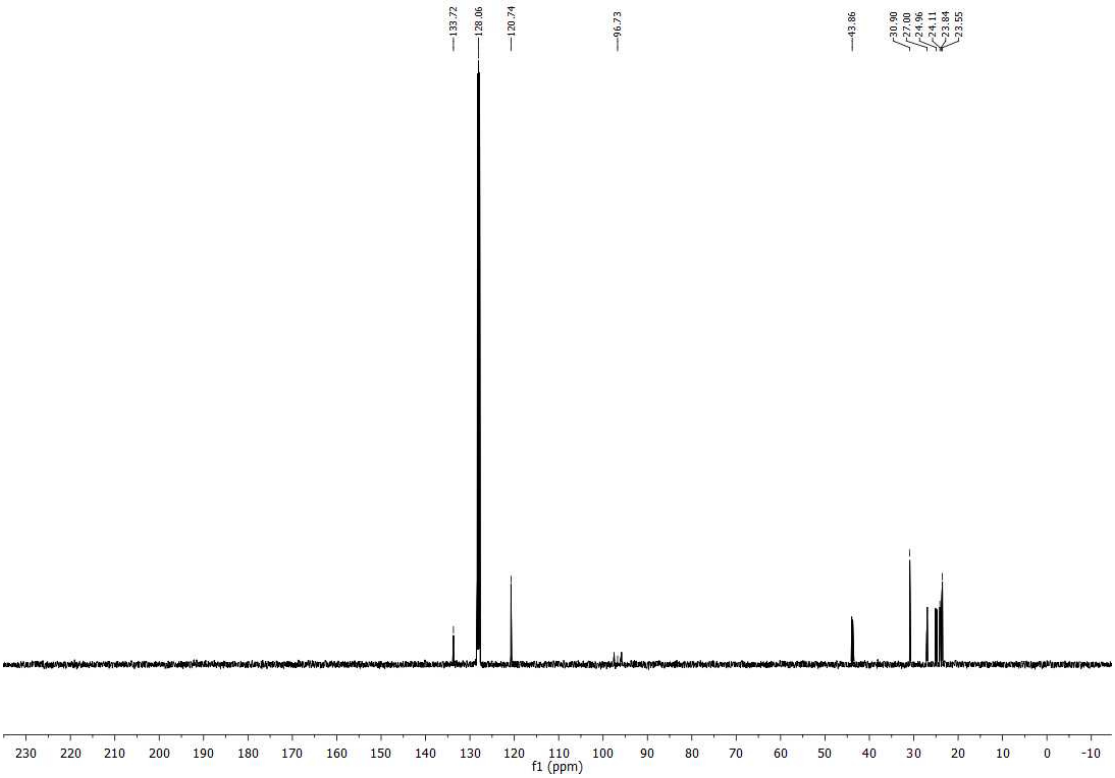

96.21
77.16
40.22
35.22
25.62
25.24
23.28
22.21
14.18
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

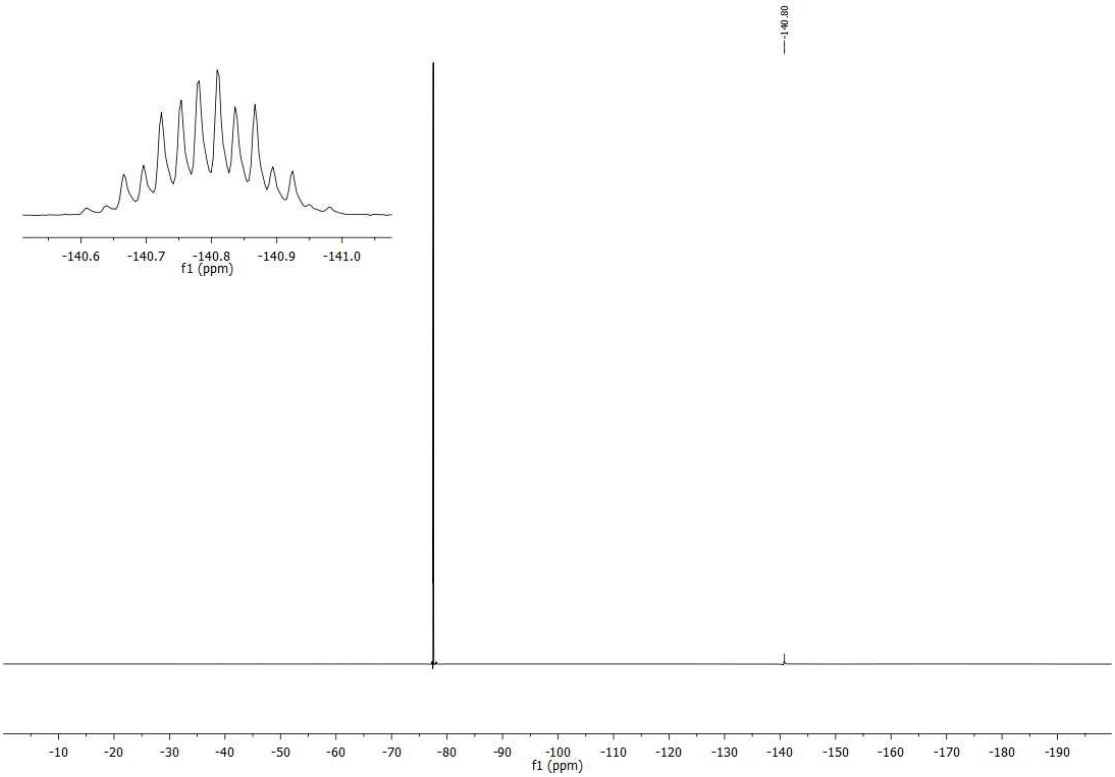

-153.88
-152.5
-153.0
-153.5
-154.0
-154.5
-155.0
f1 (ppm)
-153.88
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)

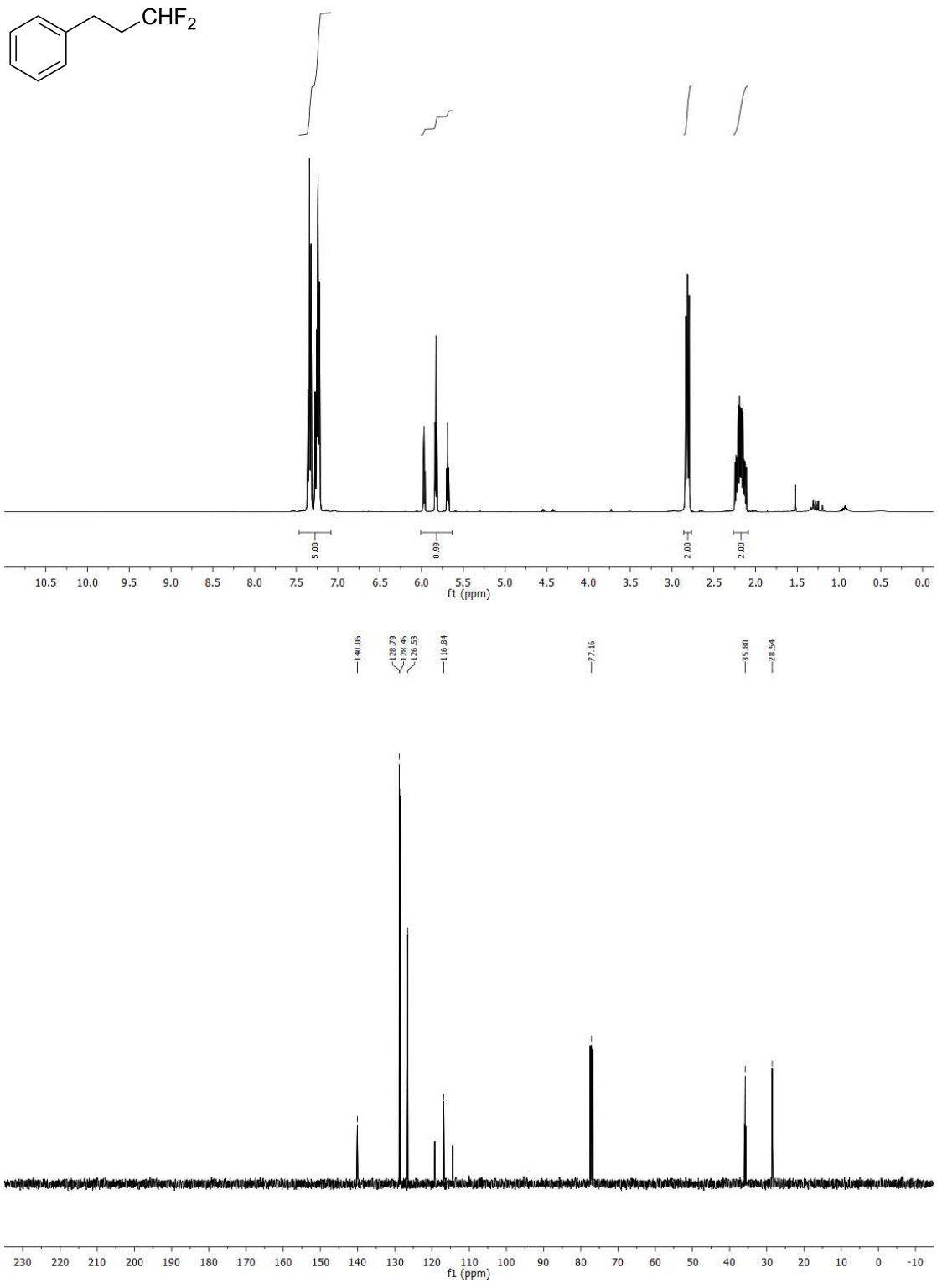

F
Me
Me
Me

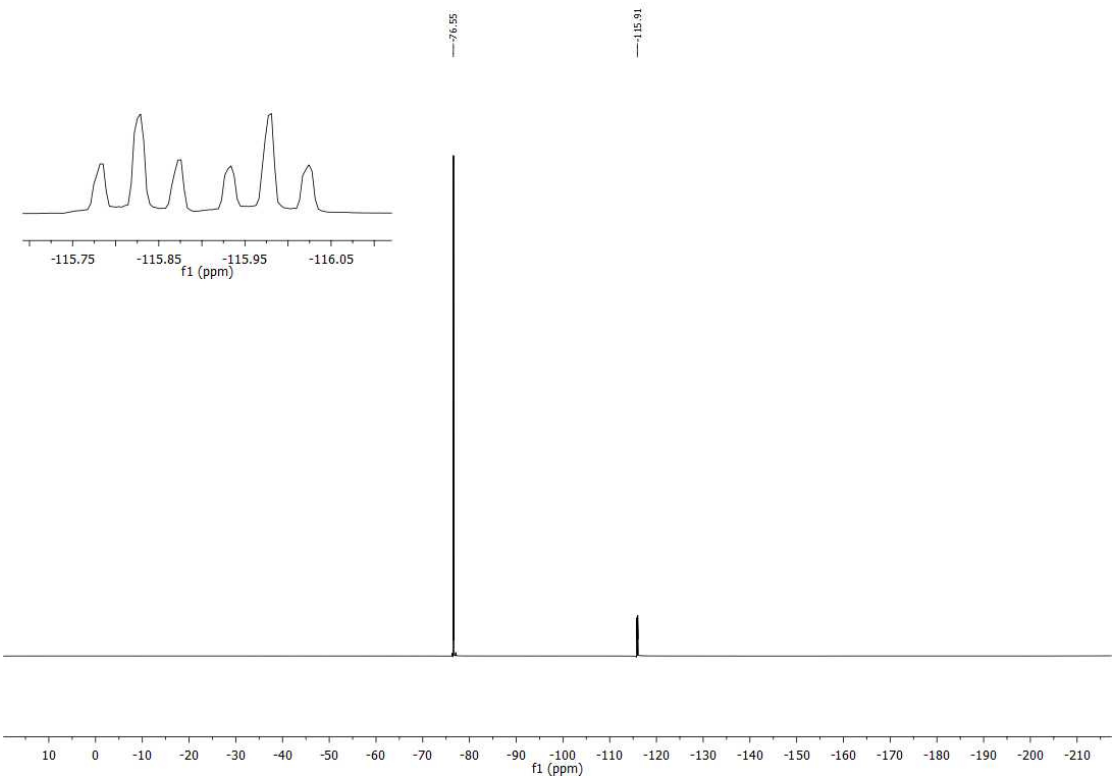
1k

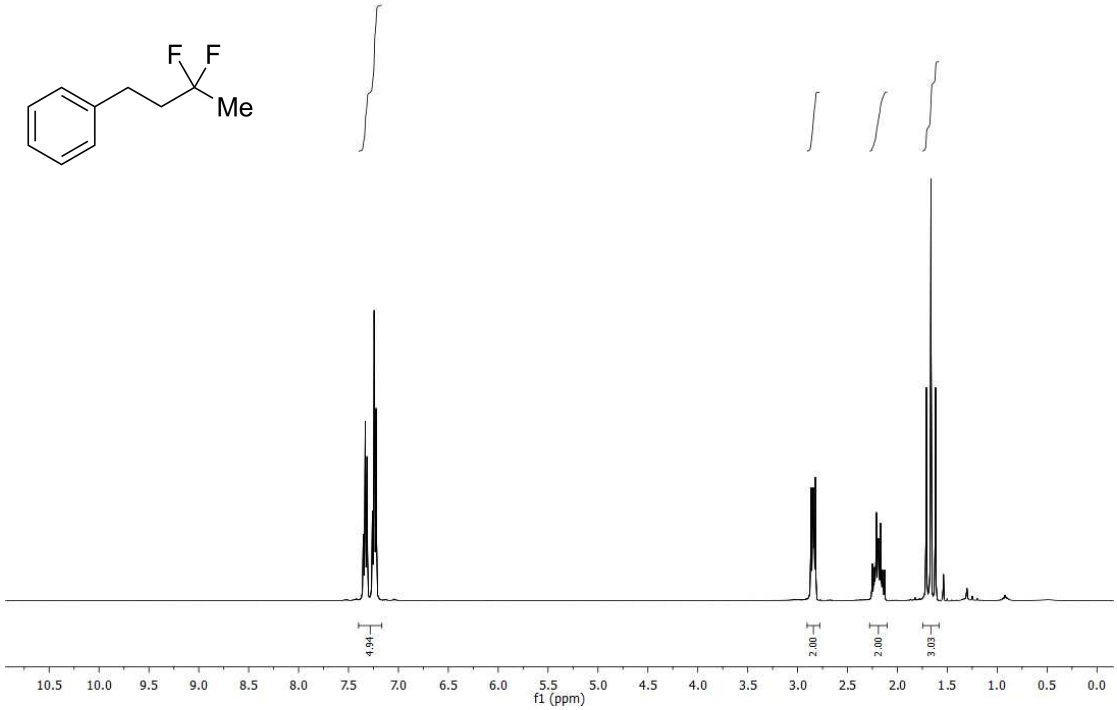

1.00
7.06
2.99
3.00
2.99
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

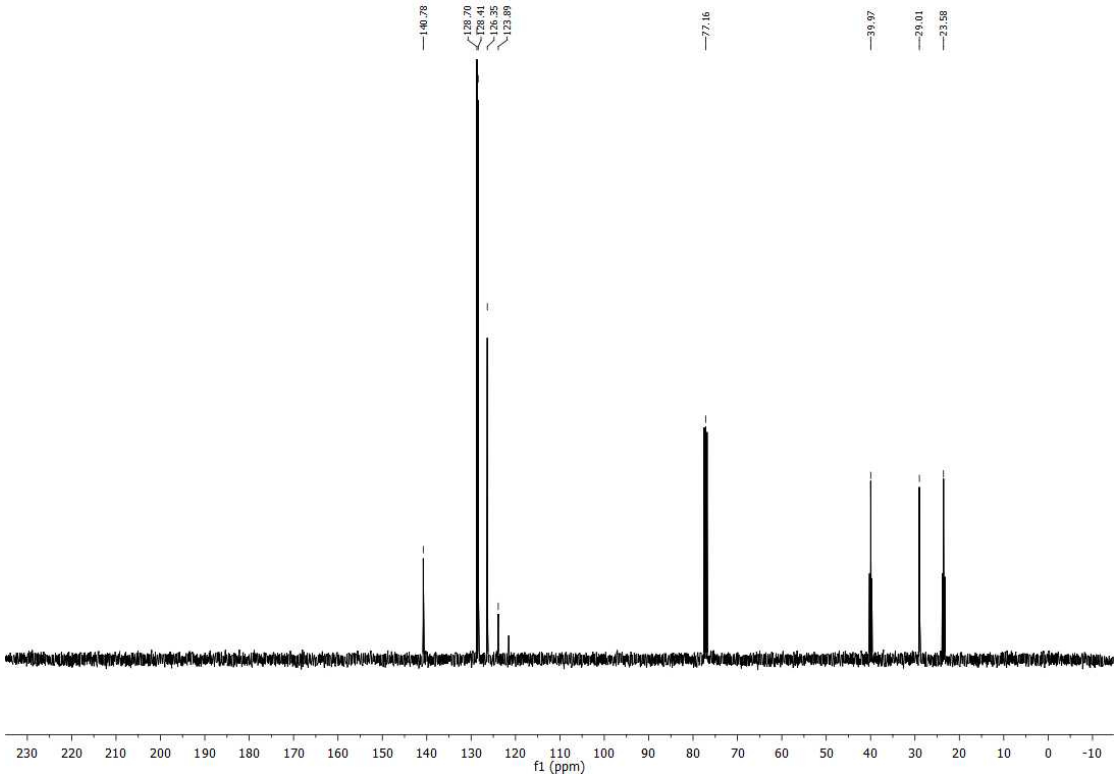

133.72
128.06
120.74
96.73
43.86
30.90
27.00
24.96
24.11
23.84
23.55
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

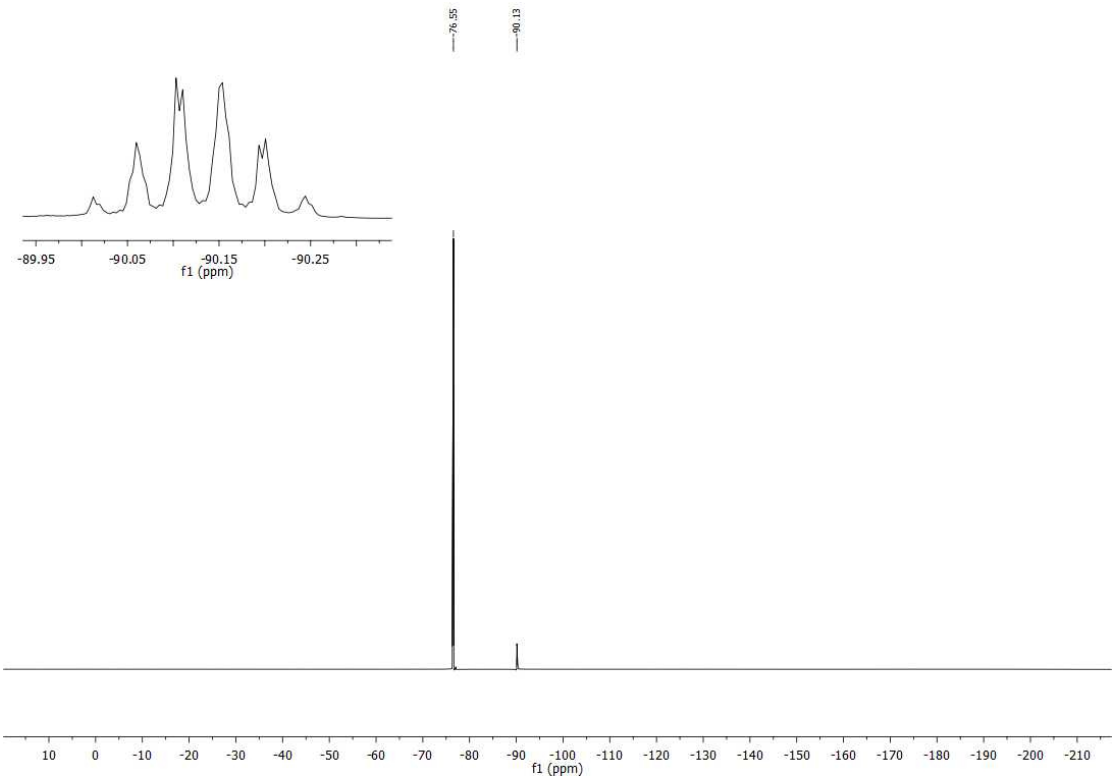

-140.80
-140.6
-140.7
-140.8
-140.9
-141.0
f1 (ppm)
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)

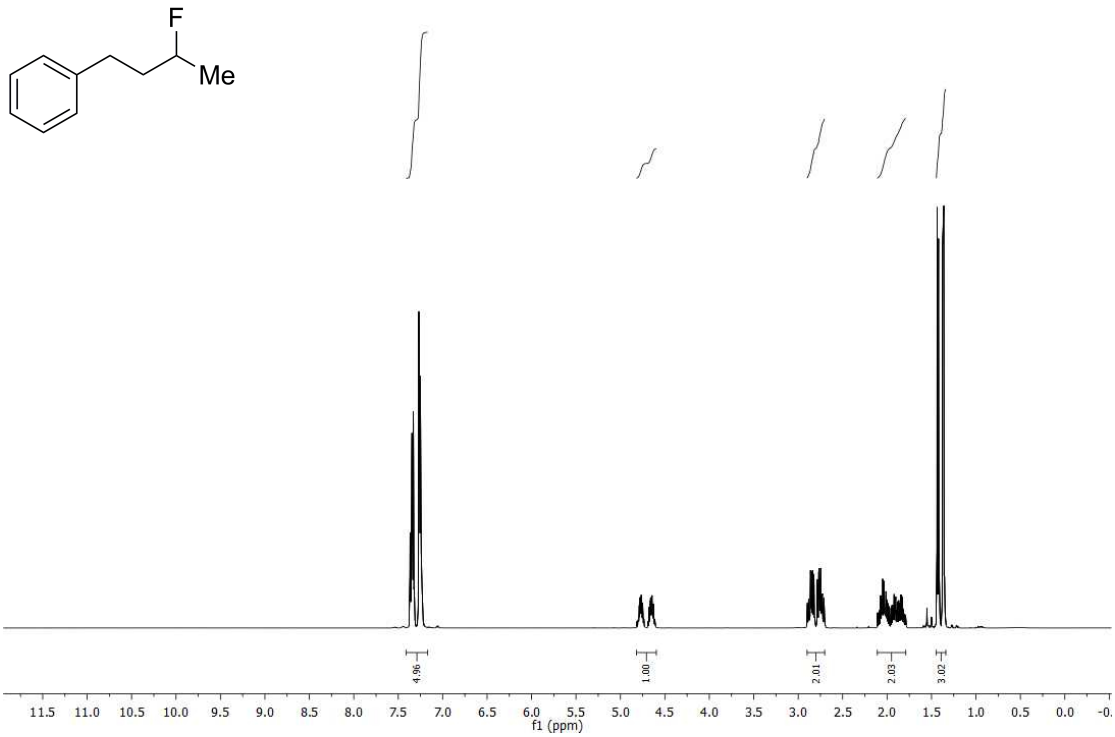

CHF2
5.00
0.99
2.00
2.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
140.06
128.79
128.45
126.53
116.84
77.16
35.80
28.54
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

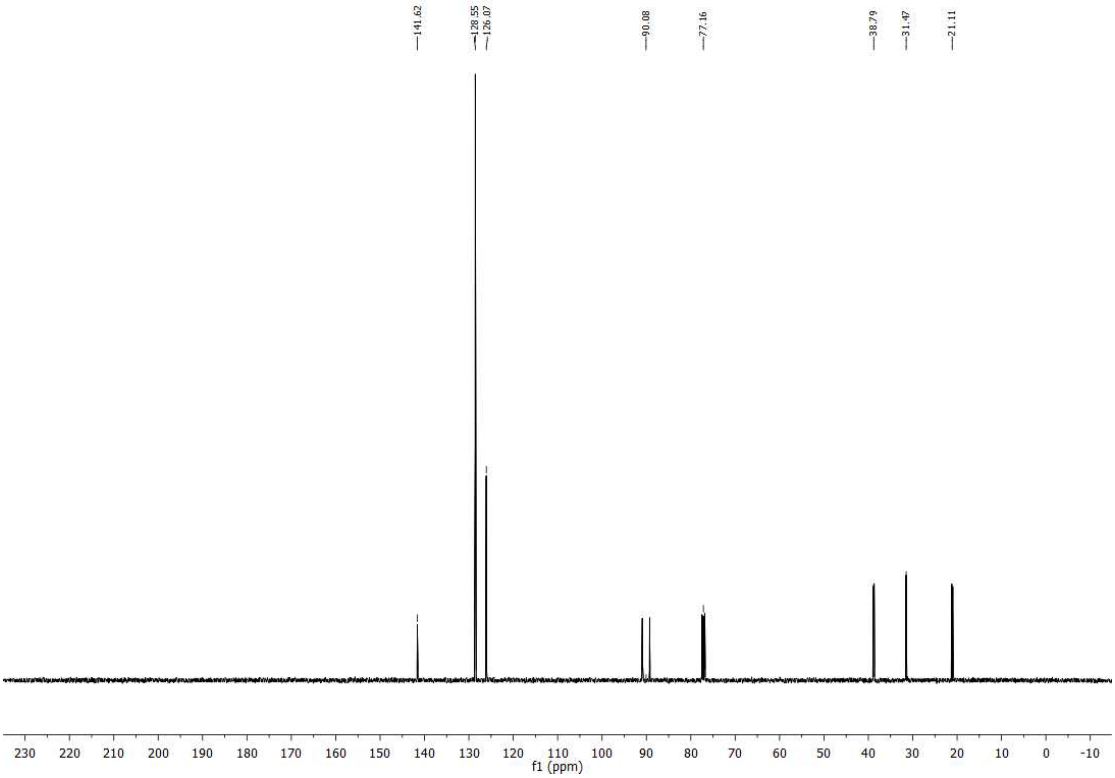

-76.55
-115.91
-115.75
-115.85
-115.95
-116.05
f1 (ppm)
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

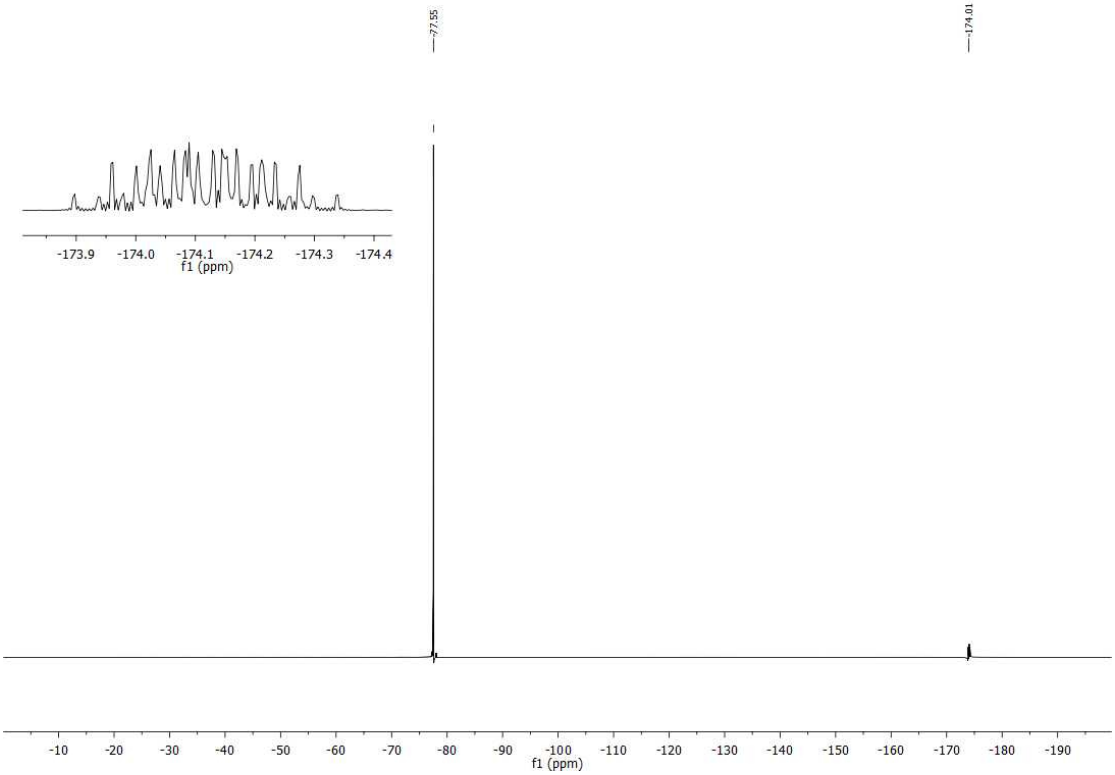

F
F
Me
4.94
2.00
2.00
3.03
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

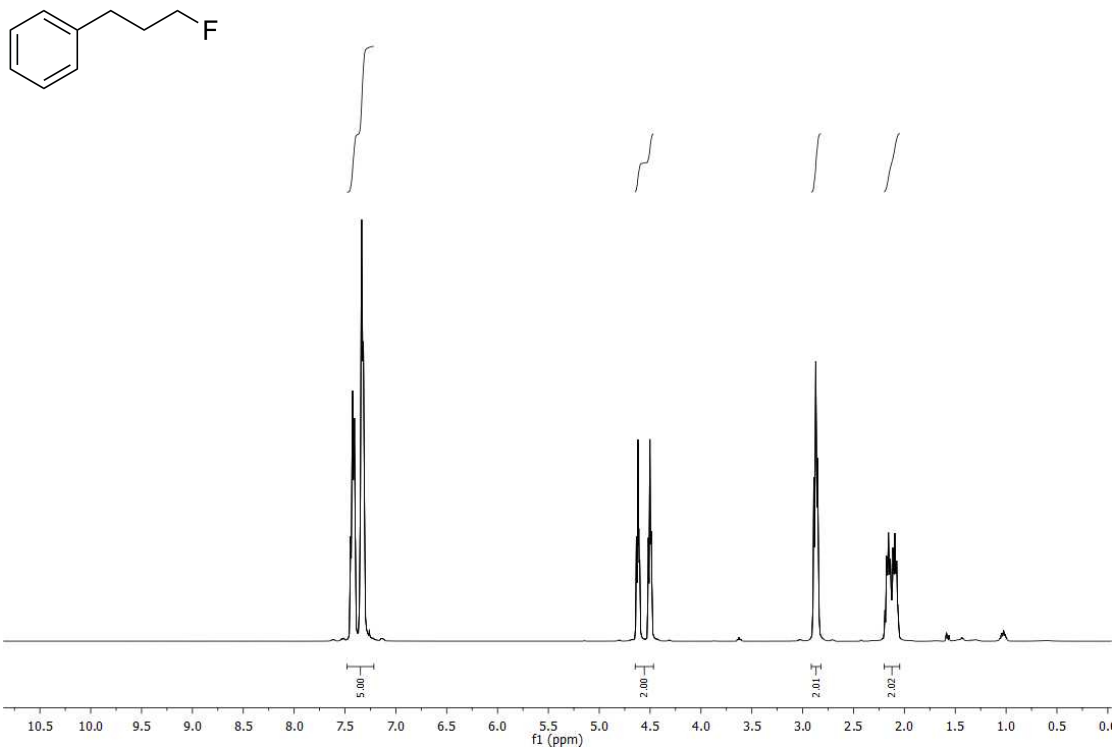

140.78
128.70
128.41
126.35
123.89
77.16
39.97
29.01
23.58
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

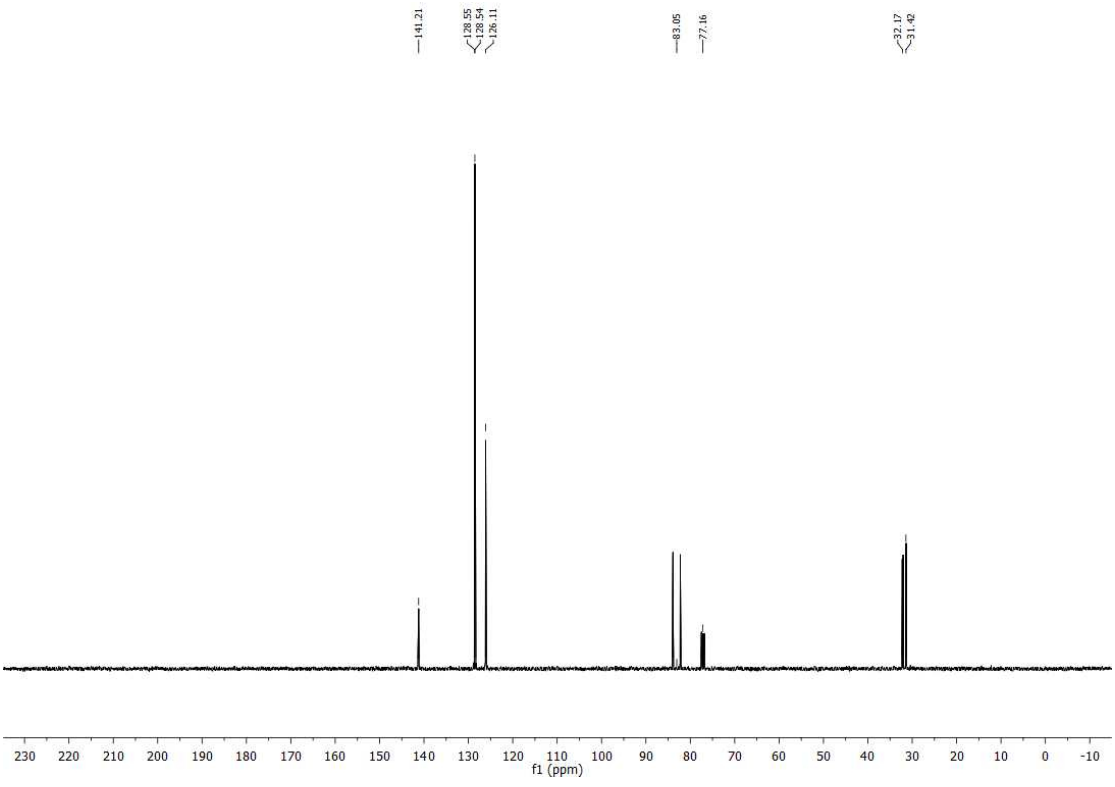

-76.55
-90.13
-89.95
-90.05
-90.15
-90.25
f1 (ppm)
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

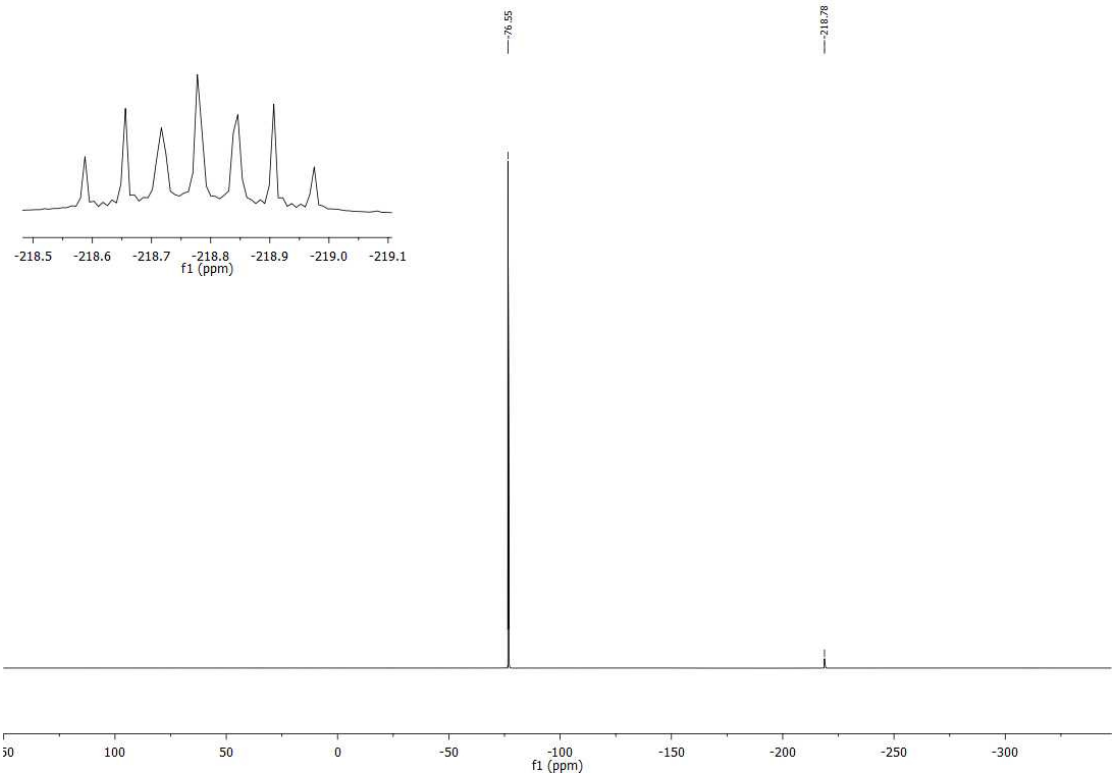

F
Me
4.96
1.00
2.01
2.03
3.02
f1 (ppm)


141.62
128.55
126.07
90.08
77.16
38.79
31.47
21.11
f1 (ppm)


-77.55
-174.01
-173.9
-174.0
-174.1
-174.2
-174.3
-174.4
f1 (ppm)
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)


F
5.00
2.00
2.01
2.02
f1 (ppm)


141.21
128.55
128.54
126.11
83.05
77.16
32.17
31.42
f1 (ppm)


-76.55
-218.78
-218.5
-218.6
-218.7
-218.8
-218.9
-219.0
-219.1
f1 (ppm)
100
50
0
-50
-100
-150
-200
-250
-300
f1 (ppm)

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