

Vanadium-Catalyzed C(sp³)-H Fluorination Reactions

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Electronic Supplementary Information

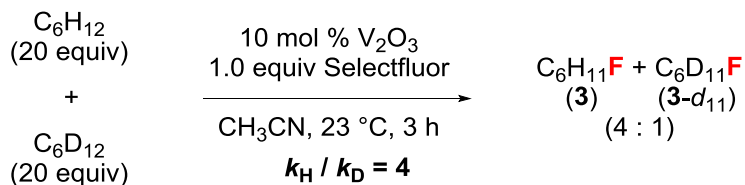
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Materials and Methods

General Information. All reactions were performed in glassware under argon. Organic solutions were concentrated by rotary evaporator at ca. 30 mmHg unless otherwise noted. Flash column chromatography was performed as described by Still¹, employing EMD silica gel 60 (230–400 mesh ASTM). TLC analyses were performed on EMD 250 μm Silica Gel 60 F₂₅₄ plates and visualized by quenching of UV fluorescence ($\lambda_{\text{max}} = 254 \text{ nm}$), or by staining ceric ammonium molybdate. ¹H and ¹³C NMR spectra were recorded on a Varian Inova-500 or Inova-400. Chemical shifts for ¹H and ¹³C NMR spectra are reported in ppm (δ) relative to the ¹H and ¹³C signals in the solvent (CDCl₃: 7.26, 77.00 ppm; C₆D₆: δ 7.16, 128.06 ppm; CD₃CN: δ 1.94, 1.32 ppm) and the multiplicities are presented as follows: s = singlet, d = doublet, t = triplet, q = quartet, hept = heptet, m = multiplet. Crude ¹H and ¹⁹F NMR spectra were recorded on an Inova-400 in CD₃CN using fluorobenzene (¹⁹F NMR δ -114.930) as an external standard. Mass spectra were acquired on an Agilent 6120 Single Quadrupole LC/MS or Agilent 7820A GC/5975 MSD.

Materials. Acetonitrile (anhydrous, 99.8%) was purchased from Sigma-Aldrich. Selectfluor (98+%) was purchased from Alfa Aesar. Vanadium(III) Oxide (95%) was purchased from Strem Chemicals Inc. Fluorobenzene ($\geq 99.5\%$) was purchased from Fluka. Acetonitrile-*d*₃ (D, 99.8%) was purchased from Cambridge Isotope Laboratories, Inc.

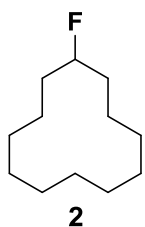
General procedure for the V₂O₃-catalyzed C(sp³)-H fluorination reaction. To a 4 mL clear vial charged with vanadium(III) oxide (3.0 mg, 0.02 mmol, 10 mol%), Selectfluor (106.3 mg, 0.3 mmol, 1.5 equiv) was added anhydrous acetonitrile (2.0 mL), and the reaction substrate (0.2 mmol, 1.0 equiv). The reaction mixture was degassed three times by *Freeze-Pump-Thaw* cycles and stirred at room temperature for 6–48 h. The crude yield was determined by NMR using fluorobenzene as an external standard. Upon completion, the reaction mixture was poured into diethyl ether (20 mL), filtrated, concentrated and purified by silica gel flash column chromatography using diethyl ether/pentane as the eluent.



The intermolecular kinetic isotope effect study. To a 4 mL clear vial charged with vanadium(III) oxide (1.5 mg, 0.01 mmol) and Selectfluor (35.4 mg, 0.1 mmol, 1.0 equiv) was added acetonitrile (1.0 mL), cyclohexane (168.4 mg, 20.0 mmol, 20.0 equiv), and cyclohexane-*d*₁₂ (192.5 mg, 1.0 mmol, 20.0 equiv). The reaction mixture was then degassed three times by *freeze-pump-thaw* cycles and stirred at room temperature for 3 h. The ratio of **3:3-*d*₁₁** was determined by ¹⁹F NMR on a Varian Inova-400 NMR instrument to be 81:19.

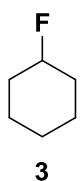
¹ Still, W. C.; Kahn, M.; Mitra, A. *J. Org. Chem.* **1978**, *43*, 2923.

Characterization Data

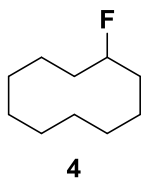


Hz, 1F).

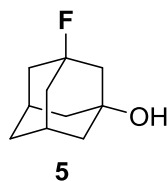
Fluorocyclododecane (2).² Prepared from cyclododecane (33.6 mg, 0.2 mmol, 1.0 equiv) according to the general procedure (24 h), purified by flash column chromatography (pentane), and concentrated by rotary evaporator at ca. 100 mm Hg to give **2** (24.0 mg, 65% yield) as a colorless oil: $R_f = 0.5$ (pentane). $^1\text{H NMR}$ (400 MHz, C_6D_6) δ 4.59 (dddd, $J = 47.3, 11.5, 7.2, 4.6$ Hz, 1H), 1.47–1.73 (m, 4H), 1.33–1.38 (m, 2H), 1.10–1.27 (m, 16H); $^{13}\text{C NMR}$ (100 MHz, C_6D_6) δ 91.8 (d, $J = 167.0$ Hz), 30.3 (d, $J = 21.2$ Hz), 24.4, 24.1, 23.5, 23.4, 20.8 (d, $J = 6.7$ Hz); $^{19}\text{F NMR}$ (376 MHz, C_6D_6) δ -176.12 (dtt, $J = 47.4, 23.7, 15.5$



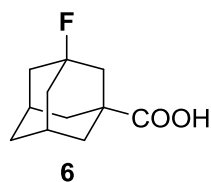
Fluorocyclohexane (3).³ Prepared from cyclohexane (84.2 mg, 1.0 mmol, 5.0 equiv) according to the general procedure (20 h) to give **3** (70% NMR yield). $^{19}\text{F NMR}$ (376 MHz, CD_3CN) δ -172.30 (brs, 1F).



Fluorocyclodecane (4).⁴ Prepared from cyclodecane (140.3 mg, 1.0 mmol, 5.0 equiv) according to the general procedure (20 h) to give **4** (19.2 mg, 61% yield). $^1\text{H NMR}$ (400 MHz, C_6D_6) δ 4.66 (dtt, $J = 46.4, 7.7, 4.1$ Hz, 1H), 1.63–1.87 (m, 4H), 1.41–1.51 (m, 2H), 1.26–1.37 (m, 12H); $^{13}\text{C NMR}$ (100 MHz, C_6D_6) δ 93.5 (d, $J = 165.7$ Hz), 31.1 (d, $J = 21.9$ Hz), 25.3, 25.1, 24.0, 21.8 (d, $J = 7.7$ Hz); $^{19}\text{F NMR}$ (376 MHz, C_6D_6) δ -165.56 (dtt, $J = 46.6, 26.6, 13.7$ Hz, 1F).



3-Fluoroadamantan-1-ol (5).² Prepared from 1-adamantanol (30.4 mg, 0.2 mmol, 1.0 equiv) according to the general procedure (20 h) and purified by flash column chromatography (15% ethyl acetate/hexanes) to give **5** (25.1 mg, 74% yield) as a colorless solid: $R_f = 0.3$ (30% ethyl acetate/hexanes). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 2.36–2.38 (m, 2H), 1.89–1.91 (m, 2H), 1.80–1.83 (m, 4H), 1.60–1.69 (m, 5H), 1.49–1.50 (m, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 93.3 (d, $J = 185.7$ Hz), 71.0 (d, $J = 11.8$ Hz), 50.4 (d, $J = 17.1$ Hz), 43.8 (d, $J = 1.6$ Hz), 41.3 (d, $J = 17.5$ Hz), 34.4 (d, $J = 2.1$ Hz), 31.4 (d, $J = 10.3$ Hz); $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ -133.24 (m, 1F).

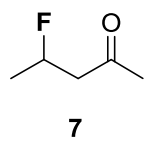


3-Fluoroadamantan-1-carboxylic acid (6).² Prepared from 1-Adamantanecarboxylic acid (36.0 mg, 0.2 mmol, 1.0 equiv) according to the general procedure (20 h) and purified by flash column chromatography (15% ethyl acetate/hexanes) to give **6** (27.9 mg, 70% yield) as a colorless solid: $R_f = 0.2$ (30% ethyl acetate/hexanes). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 11.7 (brs, 1H), 2.36–2.37 (m, 2H), 2.03–2.05 (m, 2H), 1.79–1.89 (m, 8H), 1.60–1.62 (m, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 181.5, 91.0 (d, $J = 184.2$ Hz), 43.7 (d, $J = 10.4$ Hz), 42.3 (d, $J = 20.3$ Hz), 40.7 (d, $J = 17.5$ Hz), 36.3 (d, $J = 1.9$ Hz), 33.7 (d, $J = 1.9$ Hz), 29.7 (d, $J = 10.0$ Hz); $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ -132.83 (s, 1F).

² Amaoka, Y.; Nagatomo, M.; Inoue, M. *Org. Lett.* **2013**, *15*, 2160.

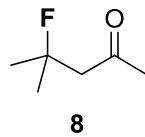
³ Chambers, R. D.; Kenwright, A. M.; Parsons, M.; Sandford, G.; Moilliet, J. S. *J. Chem. Soc. Perkin Trans. 1*, 2002, 2190.

⁴ Bloom, S.; Pitts, C. R.; Miller, D. C.; Haselton, N.; Holl, M. G.; Urheim, E.; Lectka, T. *Angew. Chem. Int. Ed.* **2012**, *51*, 10580.



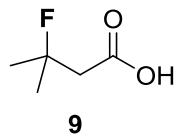
7

4-Fluoropentan-2-one (7). Prepared from 2-pentanone (17.2 mg, 0.2 mmol, 1.0 equiv) according to the general procedure (20 h) to give **7** (47% NMR yield). ^1H NMR (400 MHz, CD_3CN) δ 4.95-5.14 (m, 1H), 2.81 (ddd, $J = 16.8, 15.2, 7.9$ Hz, 1H), 2.62 (ddd, $J = 30.4, 16.8, 4.3$ Hz, 1H), 2.09 (s, 3H), 1.29 (dd, $J = 24.2, 6.2$ Hz, 3H); ^{19}F NMR (376 MHz, CD_3CN) δ -173.37 (ddqd, $J = 48.3, 30.4, 24.2, 15.2$ Hz, 1F).



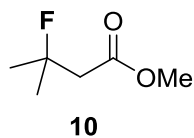
8

4-Fluoro-4-methylpentan-2-one (8).⁵ Prepared from 4-methylpentan-2-one (20.0 mg, 0.2 mmol, 1.0 equiv) according to the general procedure (20 h) to give **8** (78% NMR yield). ^1H NMR (400 MHz, CD_3CN) δ 2.75 (d, $J = 17.5$ Hz, 2H), 2.10 (s, 3H), 1.37 (d, $J = 22.0$ Hz, 6H); ^{19}F NMR (376 MHz, CD_3CN) δ -133.28 (heptt, $J = 22.0, 17.5$ Hz, 1F).



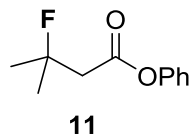
9

3-Fluoro-3-methylbutanoic acid (9). Prepared from methyl 3-methylbutanoic acid (20.4 mg, 0.2 mmol, 1.0 equiv) according to the general procedure (20 h), purified by flash column chromatography (20% ethyl acetate/hexanes), and concentrated by rotary evaporator at ca. 100 mm Hg to give **9** (15.2 mg, 63% yield) as a colorless oil: $R_f = 0.2$ (30% ethyl acetate/hexanes). ^1H NMR (400 MHz, C_6D_6) δ 2.33 (d, $J = 15.6$ Hz, 2H), 1.21 (d, $J = 21.3$ Hz, 6H); ^{13}C NMR (100 MHz, C_6D_6) δ 176.0 (d, $J = 10.3$ Hz), 92.4 (d, $J = 170.2$ Hz), 45.6 (d, $J = 26.3$ Hz), 26.6 (d, $J = 24.0$ Hz); ^{19}F NMR (376 MHz, C_6D_6) δ -134.40 (heptt, $J = 21.7, 16.2$ Hz, 1F); MS(ESI) calcd for $\text{C}_5\text{H}_9\text{FO}_2\text{Na}$ ($\text{M}+\text{Na}$)⁺ 143.1, found 143.1.



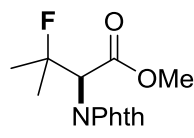
10

Methyl 3-fluoro-3-methylbutanoate (10). Prepared from methyl 3-methylbutanoate (23.2 mg, 0.2 mmol, 1.0 equiv) according to the general procedure (20 h) to give **10** (85% NMR yield). ^1H NMR (400 MHz, CD_3CN) δ 3.61 (s, 3H), 2.63 (d, $J = 17.7$ Hz, 2H), 1.42 (d, $J = 21.7$ Hz, 6H); ^{19}F NMR (376 MHz, CD_3CN) δ -134.91 (heptt, $J = 21.7, 17.7$ Hz, 1F).



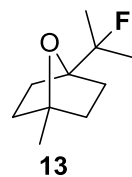
11

Phenyl 3-fluoro-3-methylbutanoate (11). Prepared from phenyl 3-methylbutanoate (35.3 mg, 0.2 mmol, 1.0 equiv) according to the general procedure (20 h) to give **11** (12% NMR yield). ^{19}F NMR (376 MHz, CD_3CN) δ -135.27 (heptt, $J = 21.6, 17.7$ Hz, 1F).



12

(2R)-N-Phthaloyl-3-fluorovaline methyl ester (12). Prepared from (*S*)-*N*-phthaloylvaline methyl ester (52.2 mg, 0.2 mmol, 1.0 equiv) according to the general procedure (18 h) and purified by flash column chromatography (10% ethyl acetate/hexanes) to give **12** (25.5 mg, 46% yield) as a colorless oil: $R_f = 0.2$ (20% ethyl acetate/hexanes). ^1H NMR (400 MHz, CDCl_3) δ 7.89 (dd, $J = 5.5, 3.0$ Hz, 2H), 7.76 (dd, $J = 5.5, 3.1$ Hz, 2H), 4.99 (d, $J = 10.2$ Hz, 1H), 3.72 (s, 3H), 1.69 (d, $J = 22.8$ Hz, 3H), 1.55 (d, $J = 22.5$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 167.3, 166.7 (d, $J = 10.5$ Hz), 134.3, 131.7, 123.7, 95.6 (d, $J = 173.0$ Hz), 57.7 (d, $J = 25.9$ Hz), 52.5, 26.3 (d, $J = 22.8$ Hz), 23.8 (d, $J = 23.7$ Hz); ^{19}F NMR (376 MHz, CDCl_3) δ -136.52 (dq, $J = 10.2, 22.5, 22.8$ Hz, 1F); MS(ESI)⁺ calcd for $\text{C}_{14}\text{H}_{14}\text{FNO}_4\text{Na}$ ($\text{M}+\text{Na}$)⁺ 302.1, found 302.1.

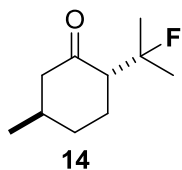


13

1-(2-Fluoropropan-2-yl)-4-methyl-7-oxabicyclo[2.2.1]heptane (13). Prepared from 1,4-cineole (30.9 mg, 0.2 mmol, 1.0 equiv) according to the general procedure (24 h), purified by flash column chromatography (2% diethyl ether/pentane), and concentrated by rotary evaporator at ca. 100 mm Hg to give **13** (18.1 mg, 53% yield) as a colorless oil: $R_f = 0.4$

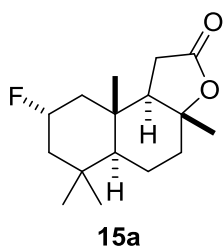
⁵ Shellhamer, D. F.; Anstine, D. T.; Gallego, K. M.; Ganesh, B. R.; Hanson, A. A.; Hanson, K. A.; Henderson, R. D.; Prince, J. M.; Heasley, V. L. *J. Chem. Soc., Perkin Trans. 2*, **1995**, 861.

(5% diethyl ether/pentane). ^1H NMR (400 MHz, C_6D_6) δ 1.76–1.84 (m, 2H), 1.24–1.46 (m, 15H); ^{13}C NMR (100 MHz, C_6D_6) δ 94.9 (d, $J = 172.0$ Hz), 89.6 (d, $J = 21.1$ Hz), 83.7, 37.2, 32.5 (d, $J = 5.3$ Hz), 23.5 (d, $J = 25.6$ Hz), 21.3; ^{19}F NMR (376 MHz, C_6D_6) δ -146.24 (hept, $J = 21.5$ Hz, 1F); MS(EI) calcd for $\text{C}_{12}\text{H}_{17}\text{FO}$ (M) $^+$ 172.1, found 172.2.



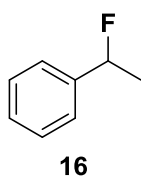
(2*R*,5*R*)-2-(2-Fluoropropan-2-yl)-5-methylcyclohexanone (14). Prepared from (-)-menthone (30.9 mg, 0.2 mmol, 1.0 equiv) according to the general procedure (30 h), purified by preparative TLC [(the plate was pretreated with ammonium (1 mL, 7M in methanol) in hexane (50 mL)] (5% ethyl acetate/hexanes), and concentrated by rotary evaporator at ca. 100 mm Hg to give **14** (26.0 mg, 75% yield) as a colorless oil: $R_f = 0.4$

(5% ethyl acetate/hexanes). ^1H NMR (400 MHz, C_6D_6) δ 2.27–2.33 (m, 1H), 2.09–2.19 (m, 2H), 1.55 (d, $J = 23.2$ Hz, 3H), 1.44 (d, $J = 22.8$ Hz, 3H), 1.31–1.45 (m, 3H), 1.18–1.29 (m, 1H), 0.78–0.88 (m, 1H), 0.62 (d, $J = 6.0$ Hz, 3H); ^{13}C NMR (100 MHz, C_6D_6) δ 207.9 (d, $J = 11.0$ Hz), 95.3 (d, $J = 165.5$ Hz), 58.8 (d, $J = 22.5$ Hz), 51.2 (d, $J = 4.2$ Hz), 35.7, 33.6, 27.7 (d, $J = 4.8$ Hz), 27.5 (d, $J = 23.3$ Hz), 22.3 (d, $J = 23.3$ Hz), 22.1; ^{19}F NMR (376 MHz, C_6D_6) δ -133.75 (heptd, $J = 23.0, 7.5$ Hz, 1F); MS(ESI) calcd for $\text{C}_{10}\text{H}_{17}\text{FONa}$ ($\text{M}+\text{Na}$) $^+$ 195.1, found 195.1.

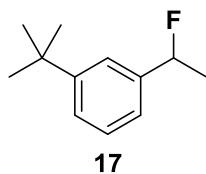


2 α -Fluorosclareolide (15a).⁶ Prepared from sclareolide (50.1 mg, 0.2 mmol, 1.5 equiv) according to the general procedure (48 h) to give 2 α -fluorosclareolide **15a** (56% NMR yield), 2 β -fluorosclareolide **15b** (6% NMR yield) and 3 α -fluorosclareolide **15c** (16% NMR yield). Purified by flash column chromatography (10% ethyl acetate/hexanes) to give a mixture of all three isomers (40.6 mg, 76% yield) as a colorless solid: $R_f = 0.3$ (20% ethyl acetate/hexanes). **15a**: ^{19}F NMR (376 MHz, CD_3CN) δ -179.22 (dddd, $J = 48.0, 11.1, 11.1, 5.5, 5.5$ Hz, 1F); **15b**: ^{19}F

NMR (376 MHz, CD_3CN) δ -172.05 (m, 1F); **15c**: ^{19}F NMR (376 MHz, CD_3CN) δ -187.44 (ddd, $J = 47.8, 47.7, 11.9$ Hz, 1F).



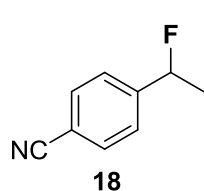
(1-Fluoroethyl)benzene (16).⁷ Prepared from ethylbenzene (21.2 mg, 0.2 mmol, 1.0 equiv) according to the general procedure (24 h) to give **2** (67% NMR yield). ^{19}F NMR (376 MHz, CD_3CN) δ -166.38 (dq, $J = 47.8, 23.9$ Hz, 1F).



1-(*tert*-Butyl)-3-(1-fluoroethyl)benzene (17).⁷ Prepared from 1-(*tert*-butyl)-3-ethylbenzene (32.5 mg, 0.2 mmol, 1.0 equiv) according to the general procedure (48 h) and purified by flash column chromatography (1% diethyl ether/pentane) to give **3** (17.0 mg, 47% yield) as a colorless oil: $R_f = 0.3$ (1% diethyl ether/pentane). ^1H NMR (400 MHz, CDCl_3) δ 7.31–7.39 (m, 2H), 7.29–7.34 (m, 1H), 7.17–7.20 (m, 1H), 5.64 (dq, $J = 47.7, 6.4$ Hz, 1H), 1.67 (dd, $J = 23.9, 6.4$ Hz, 3H), 1.33 (s, 9H); ^{19}F NMR (376 MHz, CDCl_3) δ -165.97 (dq, $J = 47.8, 23.9$ Hz, 1F).

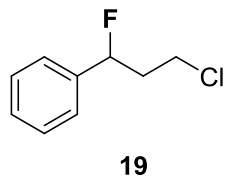
⁶ Liu, W.; Huang, X.; Cheng, M.-J.; Nielsen, R. J.; Goddard III, W. A.; Groves, J. T. *Science* **2012**, 337, 1322.

⁷ Xia, J.-B.; Zhu, C.; Chen, C. *J. Am. Chem. Soc.*, 2013, **135**, 17494.



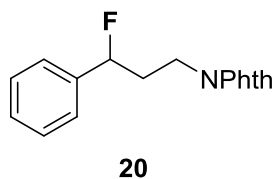
4-(1-Fluoroethyl)benzonitrile (18).⁷ Prepared from 4-ethylbenzonitrile (26.2 mg, 0.2 mmol, 1.0 equiv) according to the general procedure (24 h) and purified by flash column chromatography (3% diethyl ether/pentane) to give **18** (7.2 mg, 24% yield) as a colorless oil: $R_f = 0.3$ (3% diethyl ether/pentane). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.68 (d, $J = 8.0$ Hz, 2H), 7.45 (d, $J = 8.0$ Hz, 2H), 5.67 (dq, $J = 47.4, 6.5$ Hz, 1H), 1.64 (dd, $J = 24.0, 6.5$ Hz, 3H); $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ -172.73 (dq, $J = 47.9, 24.0$

Hz, 1F).



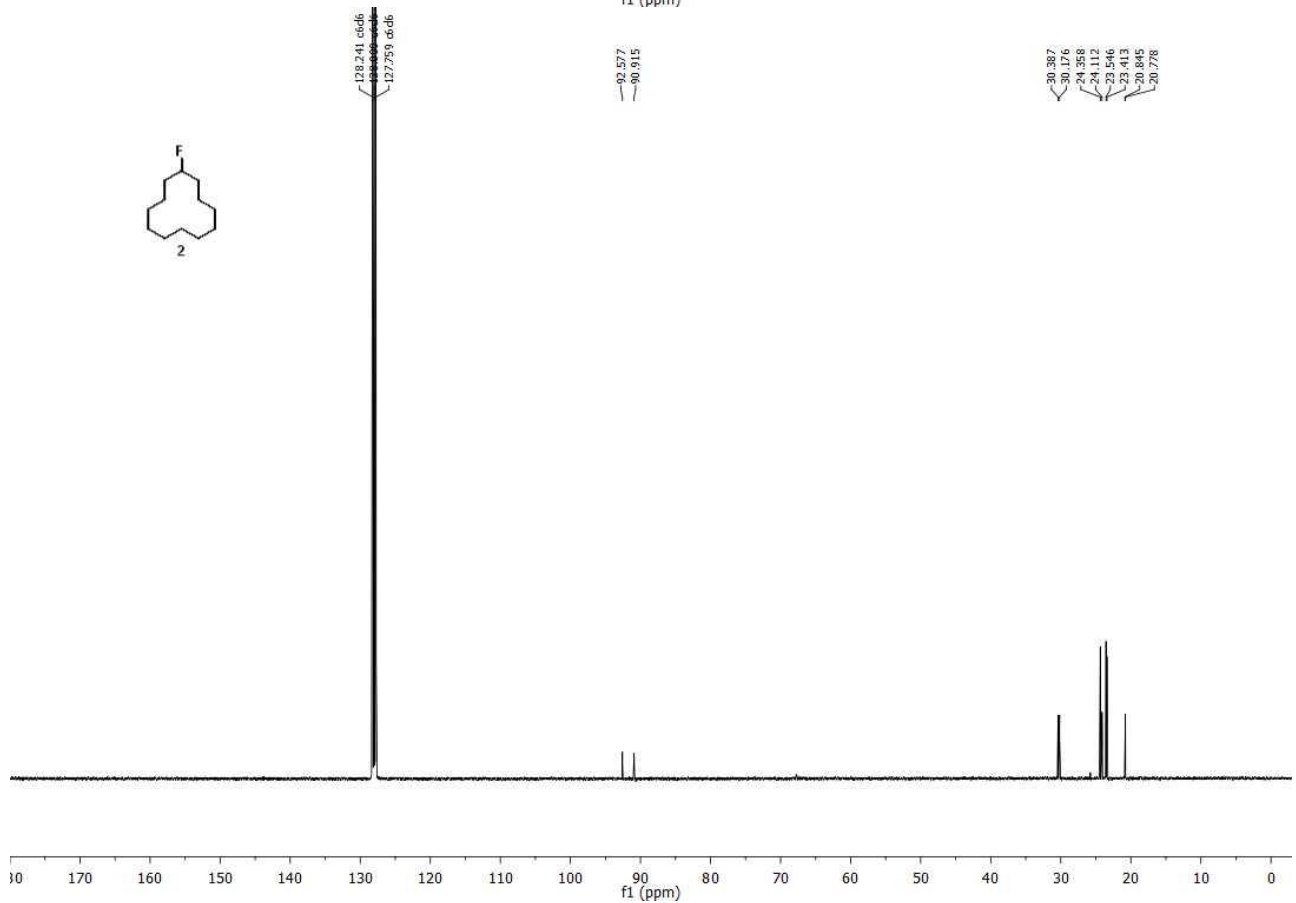
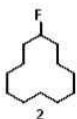
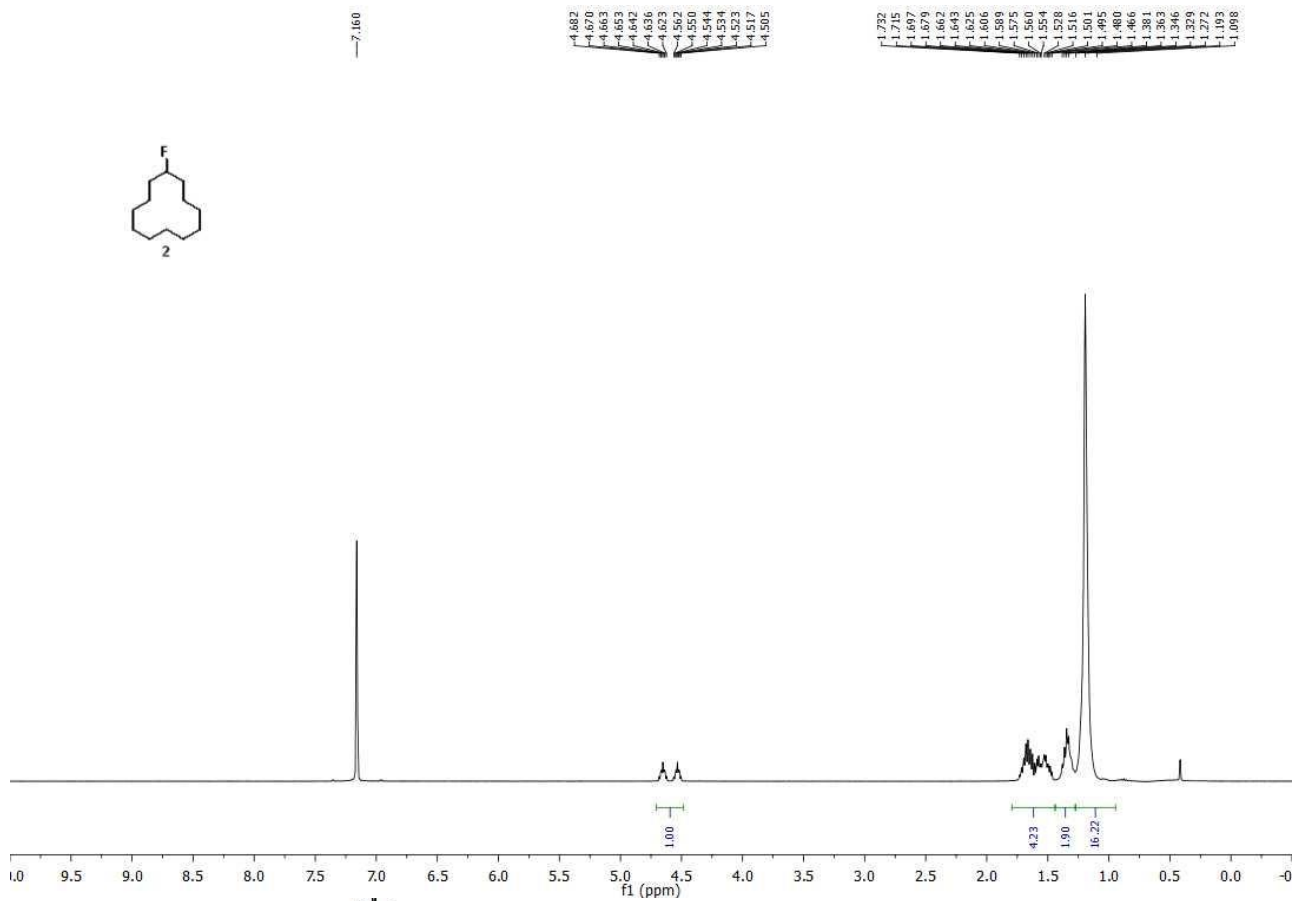
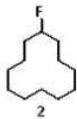
(3-Chloro-1-fluoropropyl)benzene (19).⁷ Prepared from (3-chloropropyl)benzene (30.9 mg, 0.2 mmol, 1.0 equiv) according to the general procedure (24 h) and purified by preparative TLC (1% ethyl acetate/hexanes) to give **19** (12.0 mg, 35% yield) as a colorless oil: $R_f = 0.4$ (1% ethyl acetate/hexanes). $^1\text{H NMR}$ (400 MHz, C_6D_6) δ 7.03–7.07 (m, 5H), 5.42 (ddd, $J = 48.0, 9.0, 3.9$ Hz, 1H), 3.26–3.33 (m, 1H),

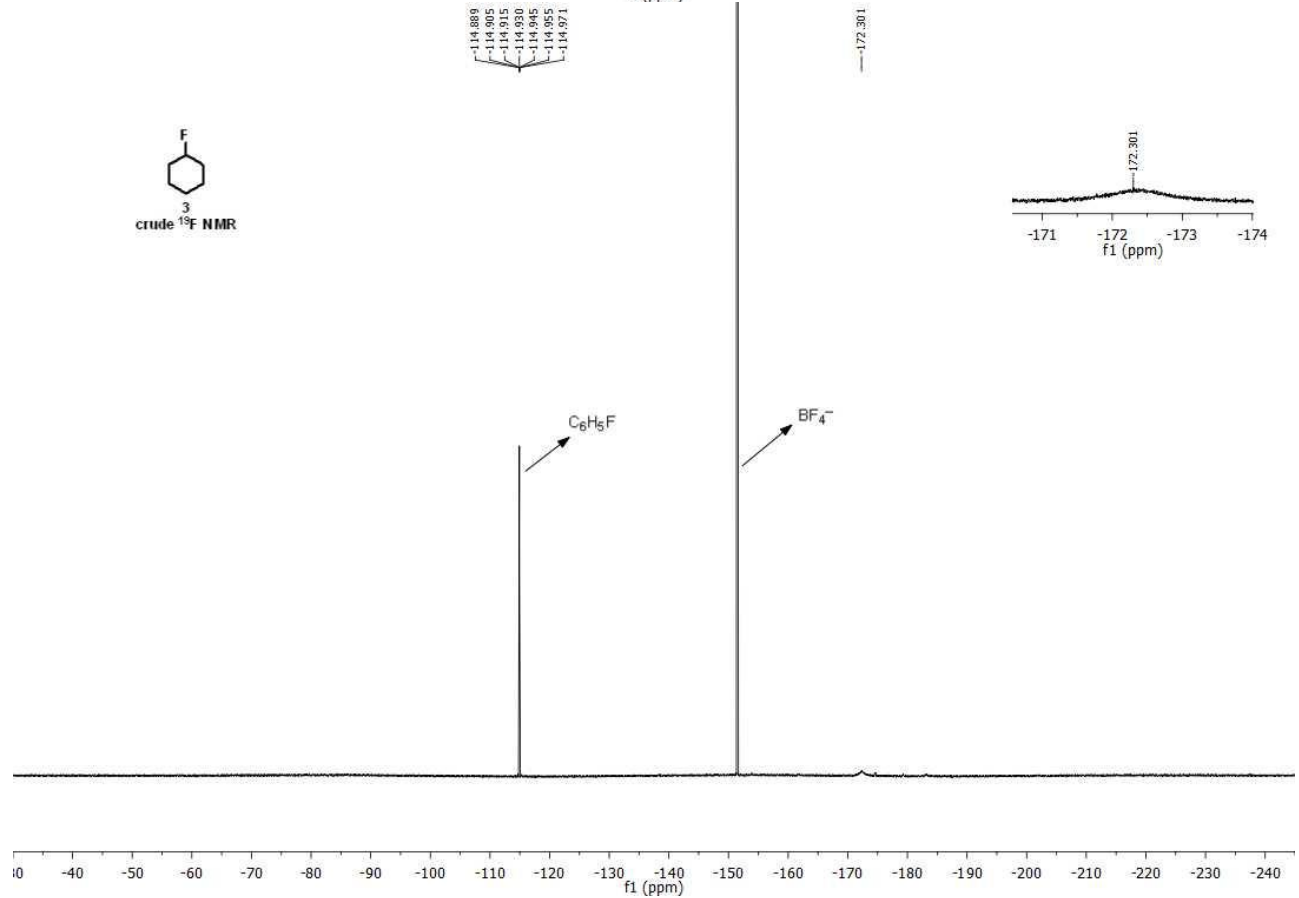
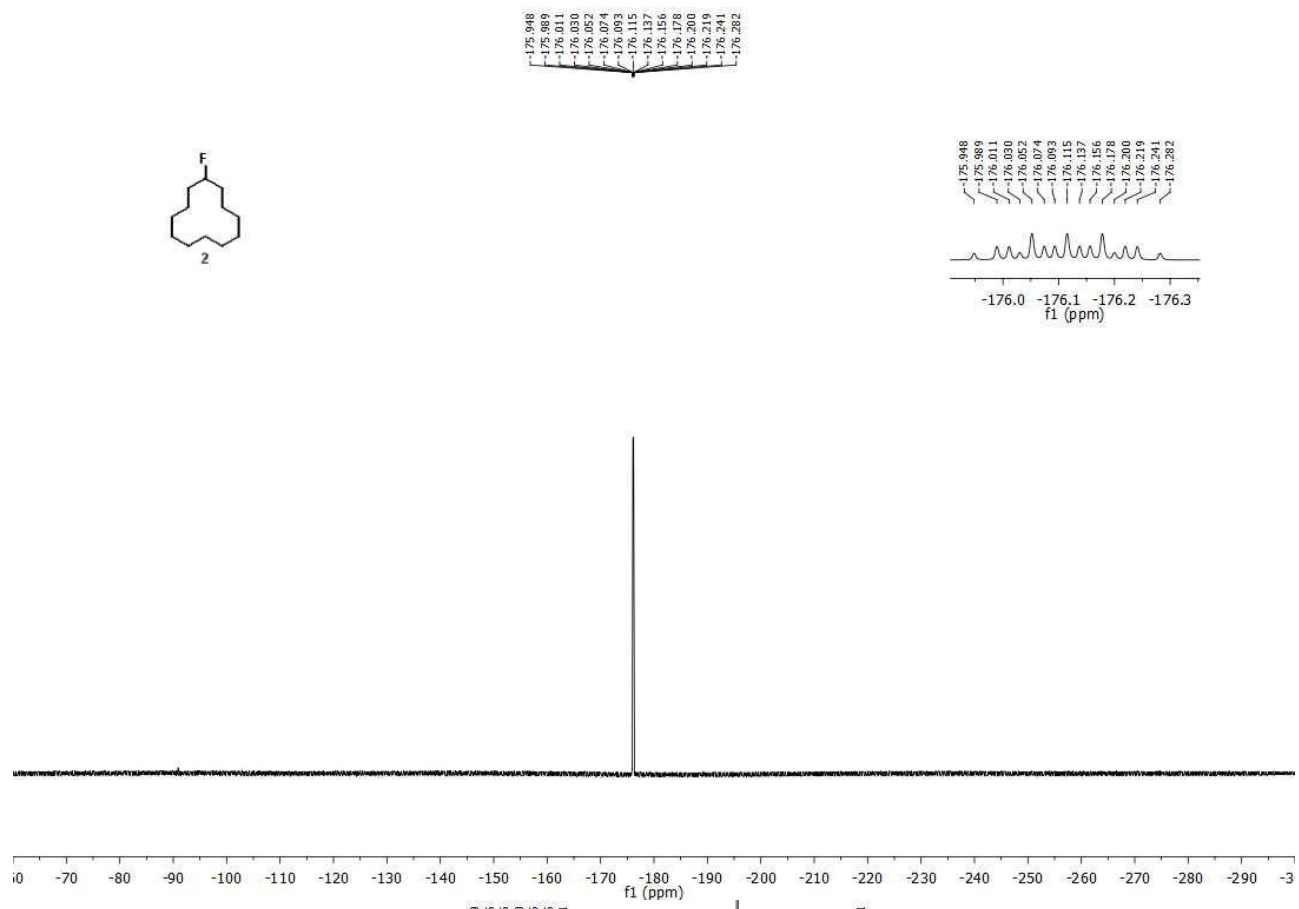
3.04–3.09 (m, 1H), 1.92–2.04 (m, 1H), 1.64–1.80 (m, 1H); $^{19}\text{F NMR}$ (376 MHz, C_6D_6) δ -179.53 (ddd, $J = 48.2, 31.0, 13.5$ Hz, 1F).

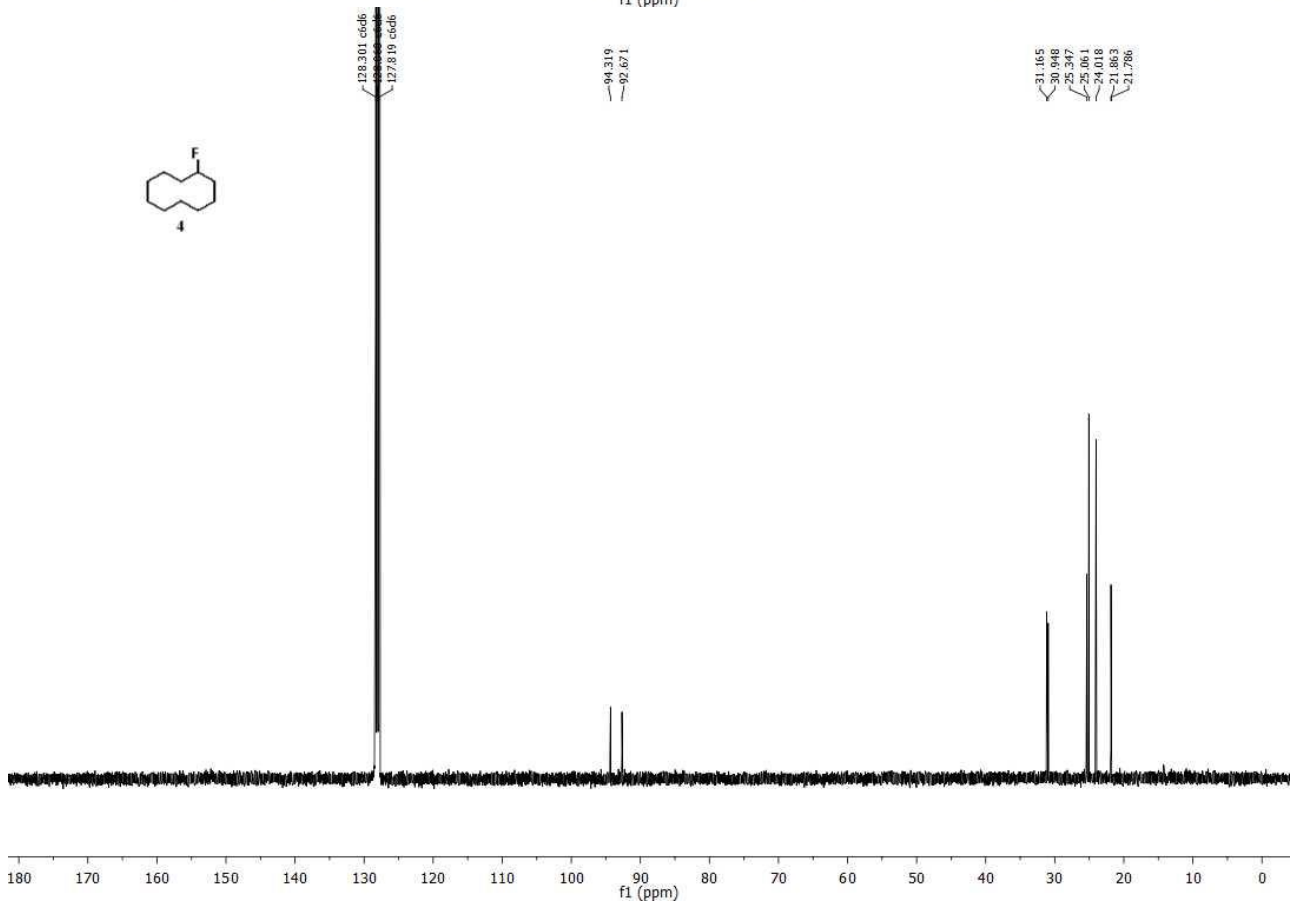
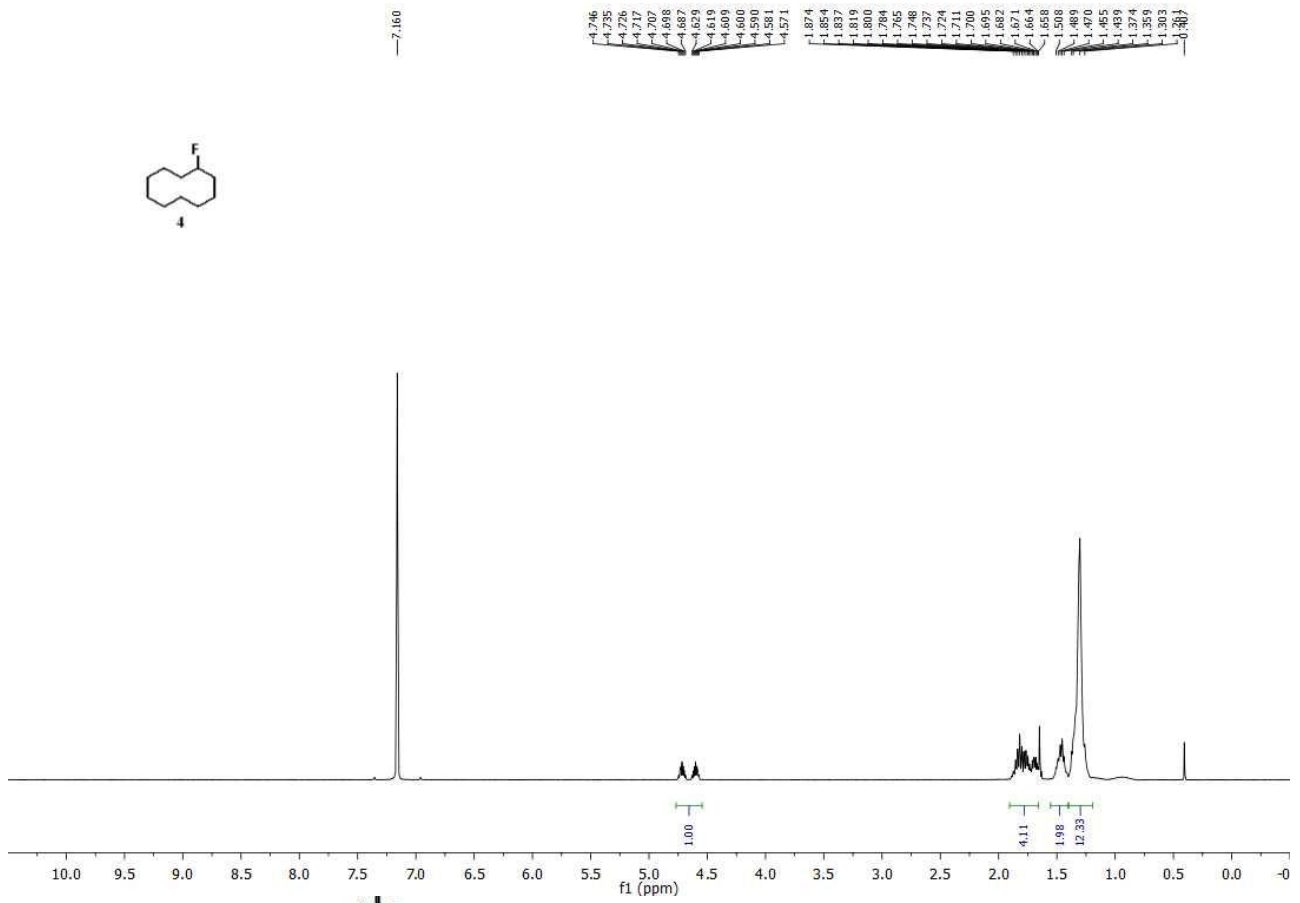


N-Phthaloyl 3-fluoro-3-phenylpropylamine (20).⁷ Prepared from 2-(3-phenylpropyl)isoindoline-1,3-dione (53.0 mg, 0.2 mmol, 1.0 equiv) according to the general procedure (6 h) and purified by preparative TLC (20% ethyl acetate/hexanes) to give **20** (24.5 mg, 43% yield) as a colorless oil: $R_f = 0.4$ (20% ethyl acetate/hexanes). $^1\text{H NMR}$ (400 MHz, C_6D_6) δ 7.43–7.45 (m, 2H),

6.96–7.11 (m, 5H), 6.88–6.90 (m, 2H), 5.28 (ddd, $J = 48.1, 9.2, 4.0$ Hz, 1H), 3.61–3.76 (m, 2H), 2.12–2.25 (m, 1H), 1.87–2.02 (m, 1H); $^{19}\text{F NMR}$ (376 MHz, C_6D_6) δ -176.15 (ddd, $J = 47.4, 30.7, 16.2$ Hz, 1F).

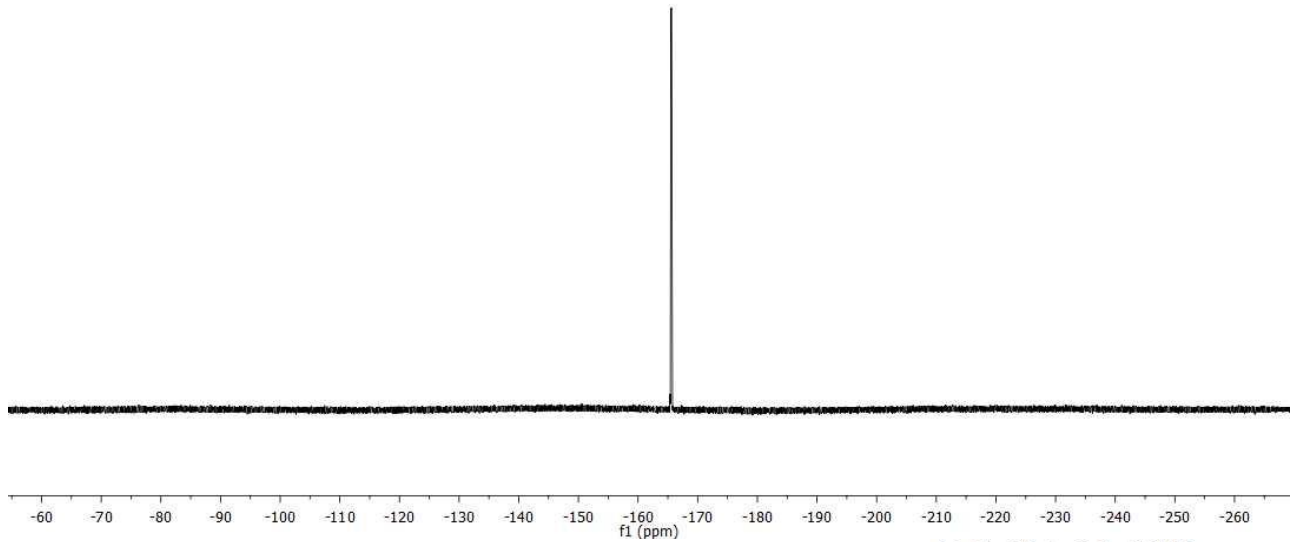
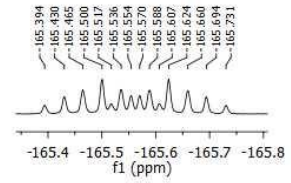






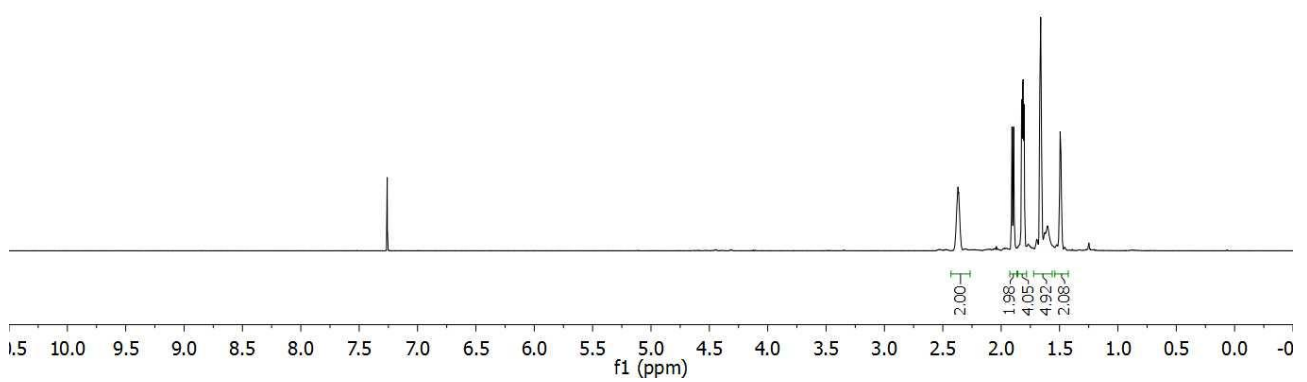
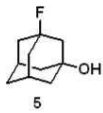


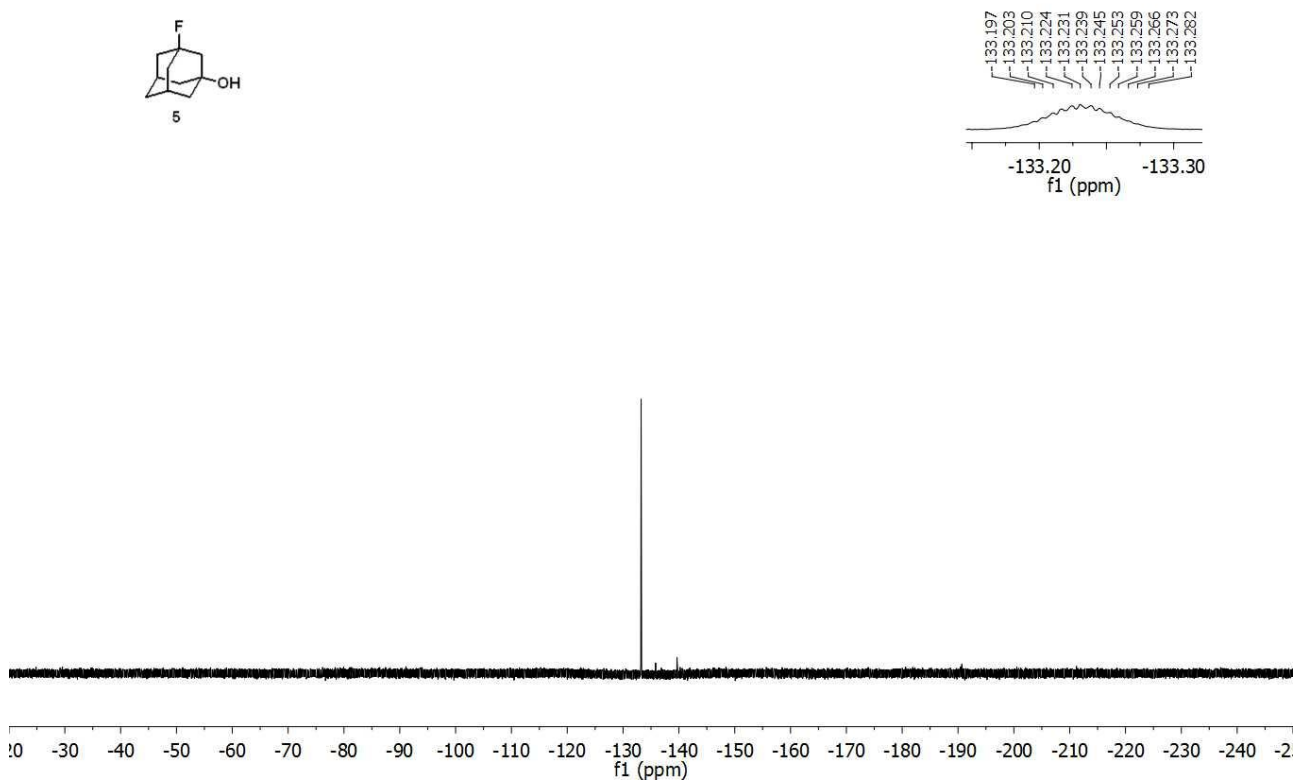
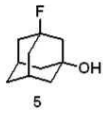
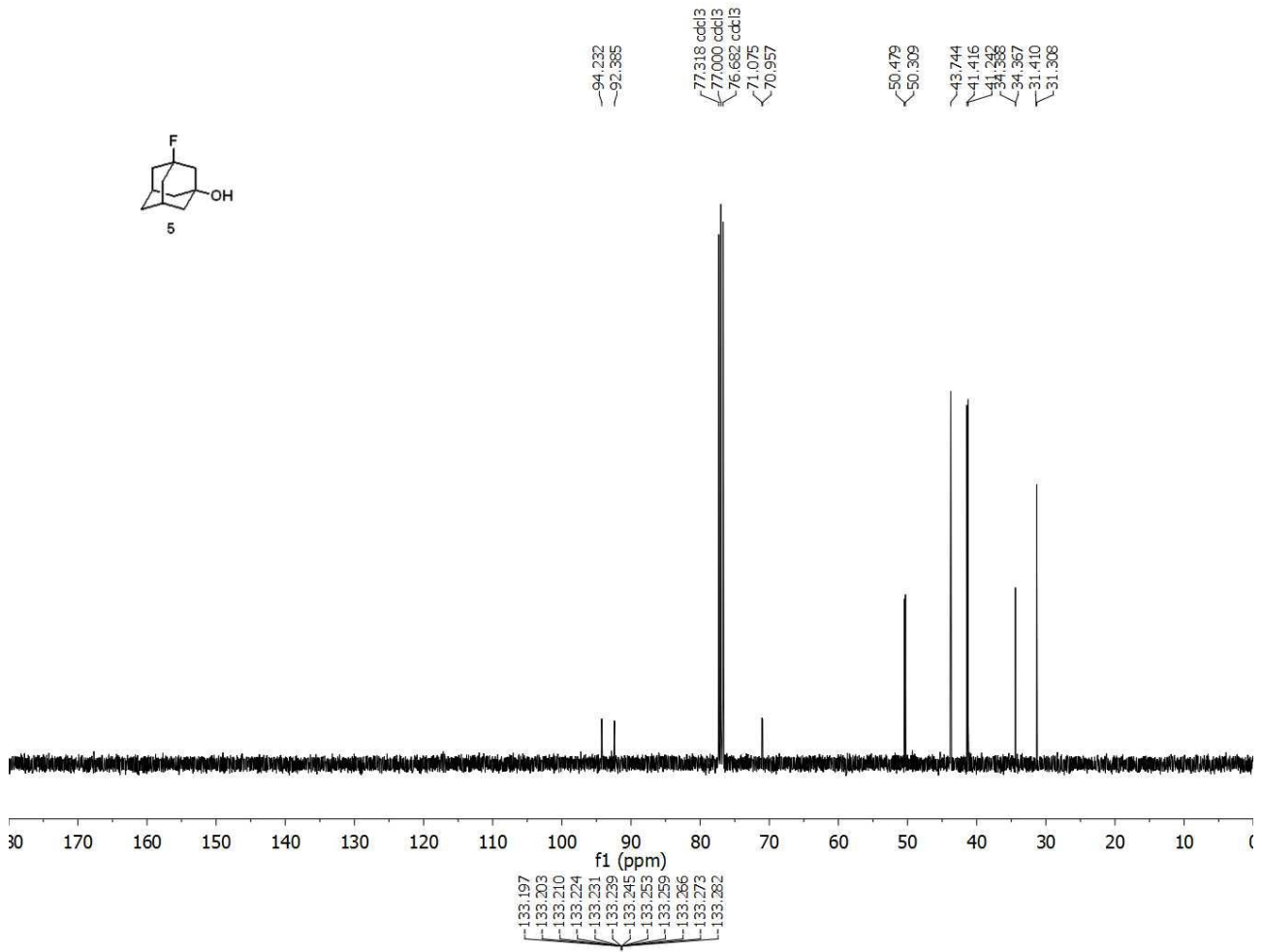
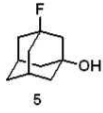
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165.731

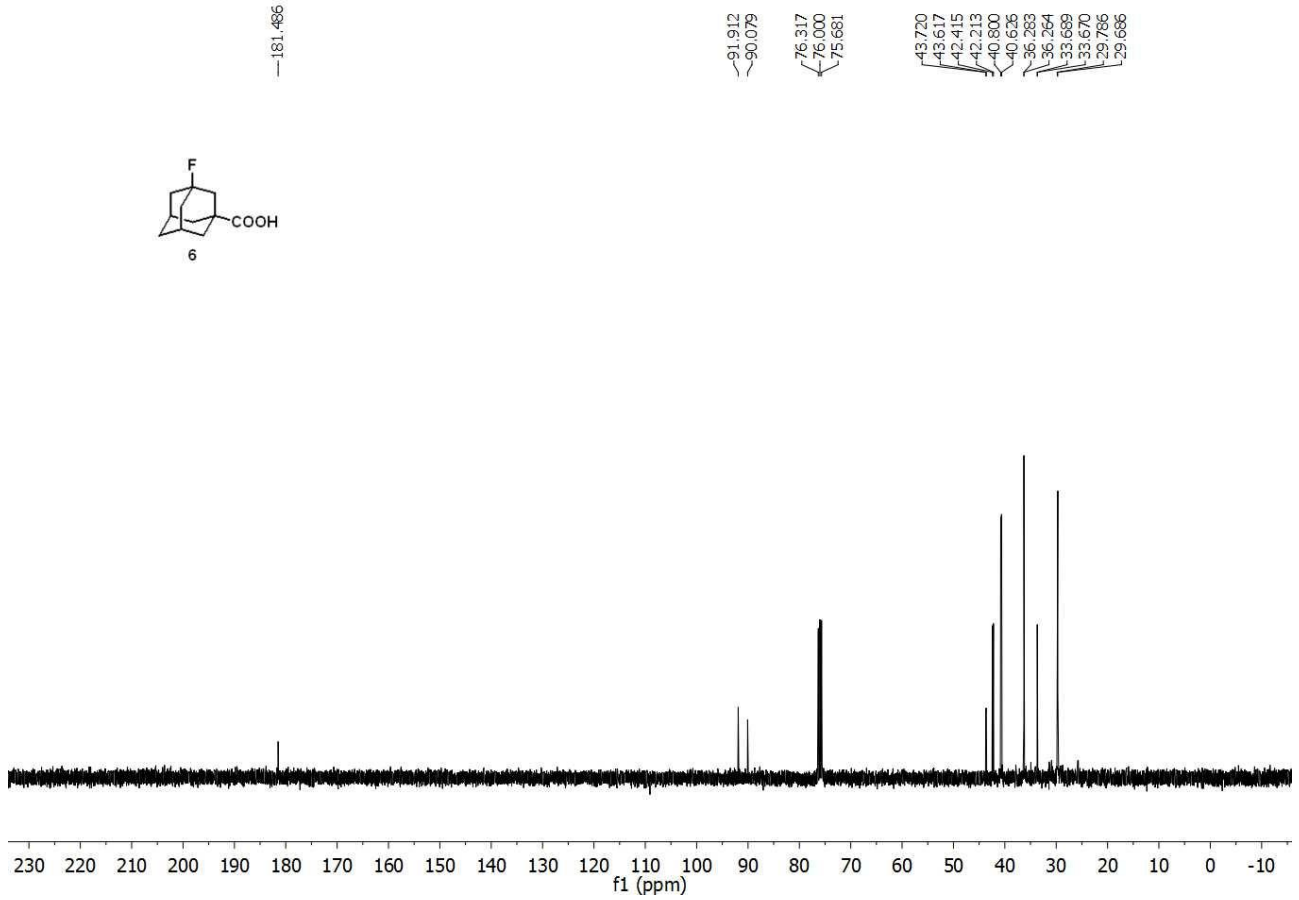
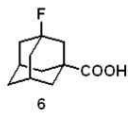
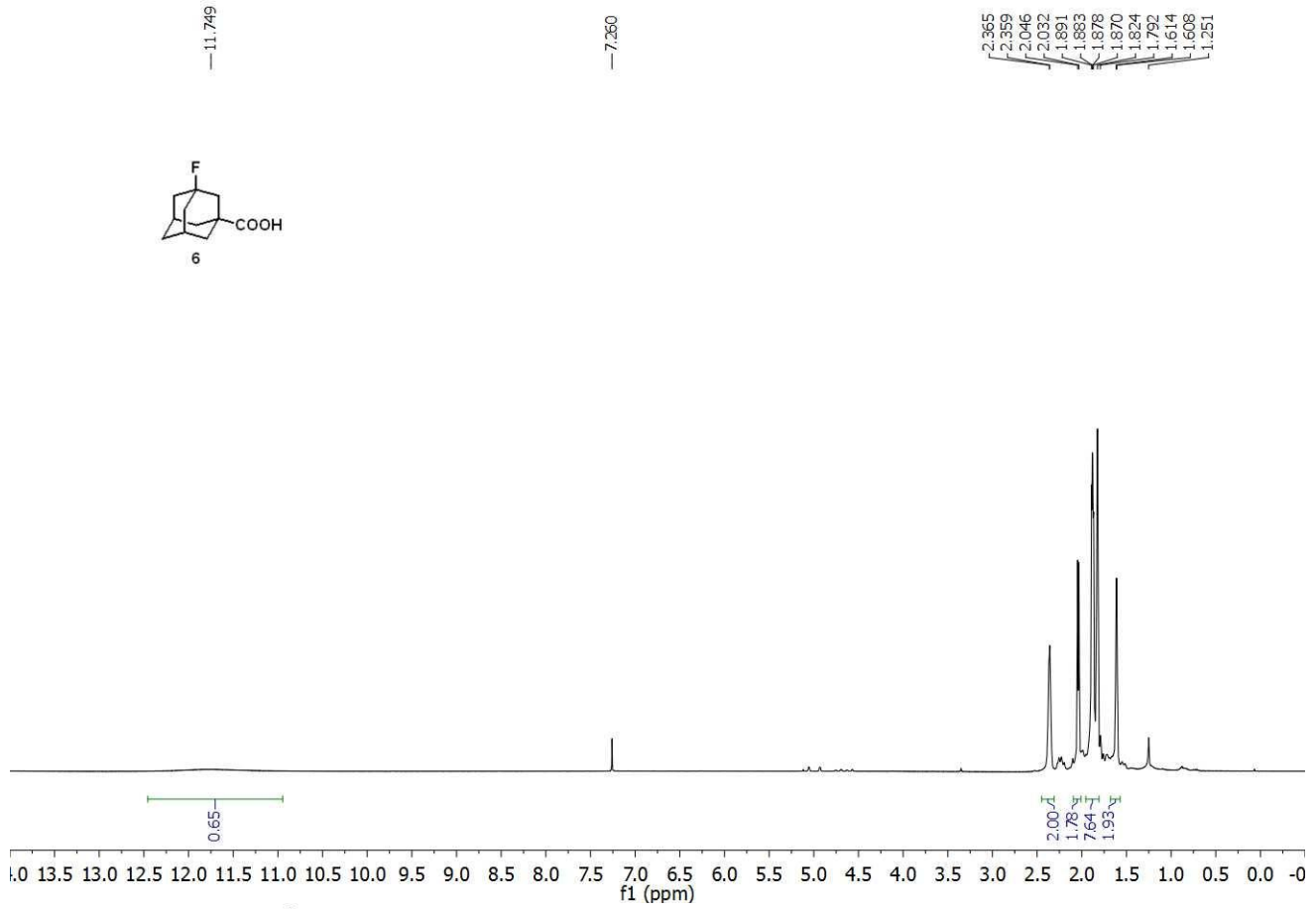


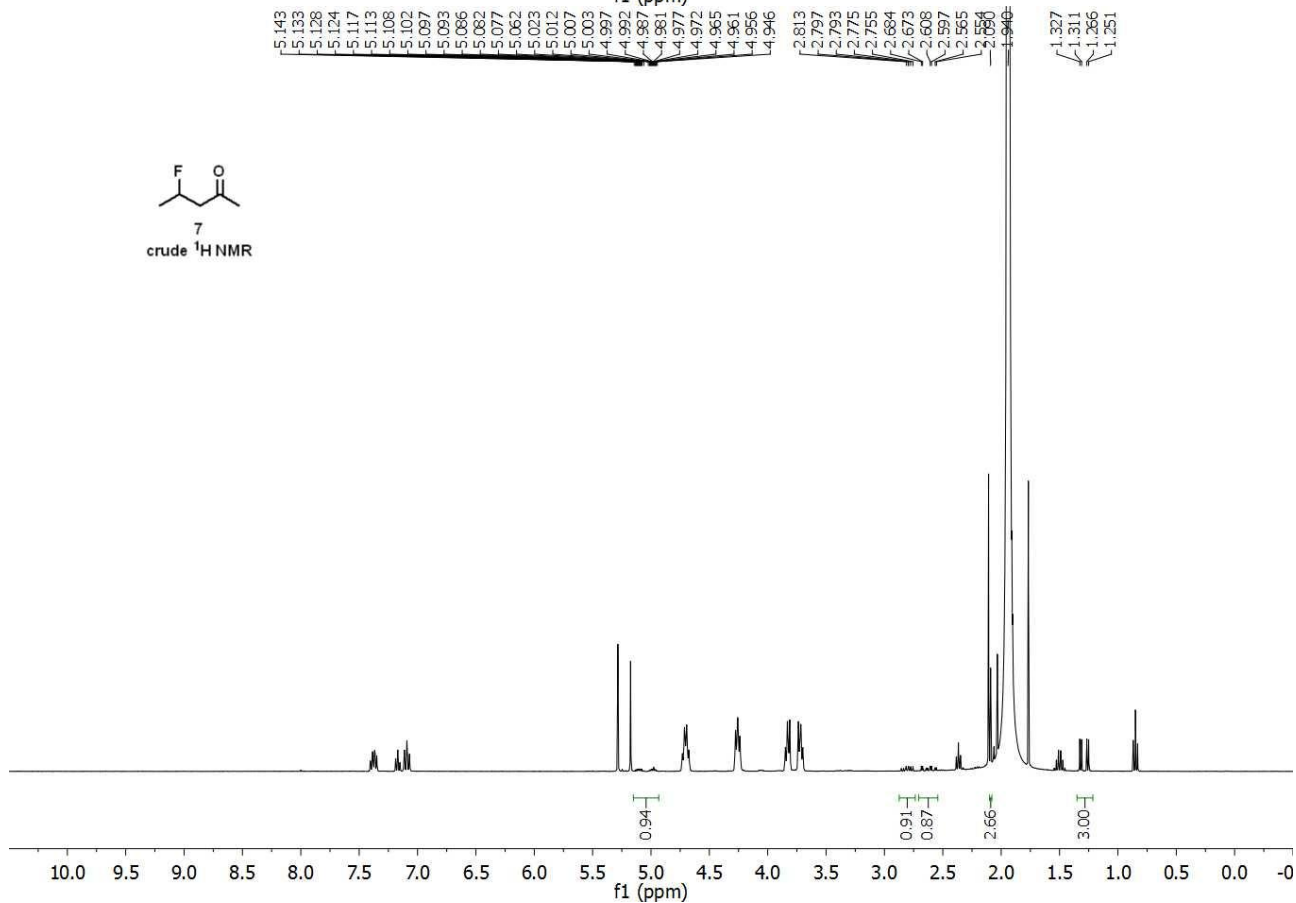
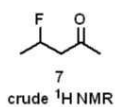
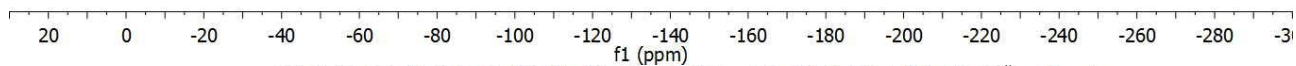
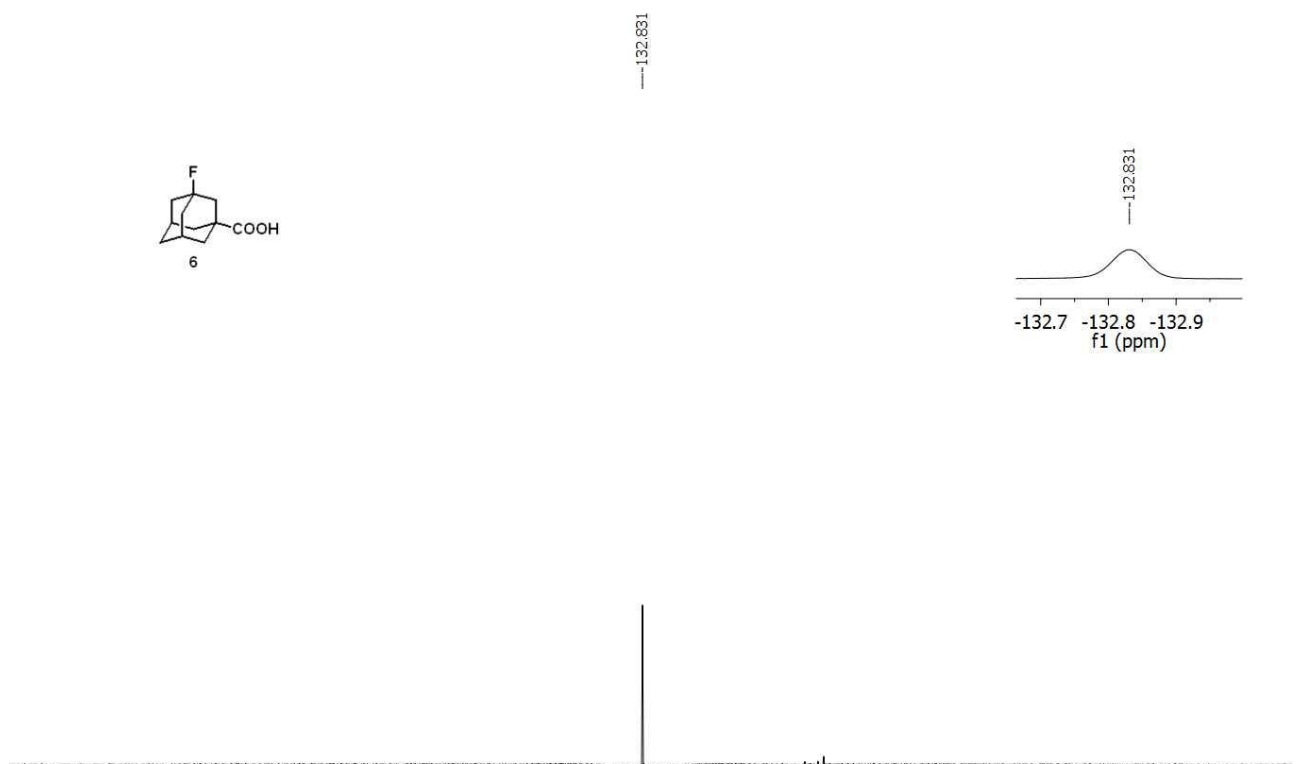
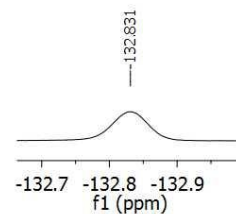
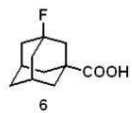
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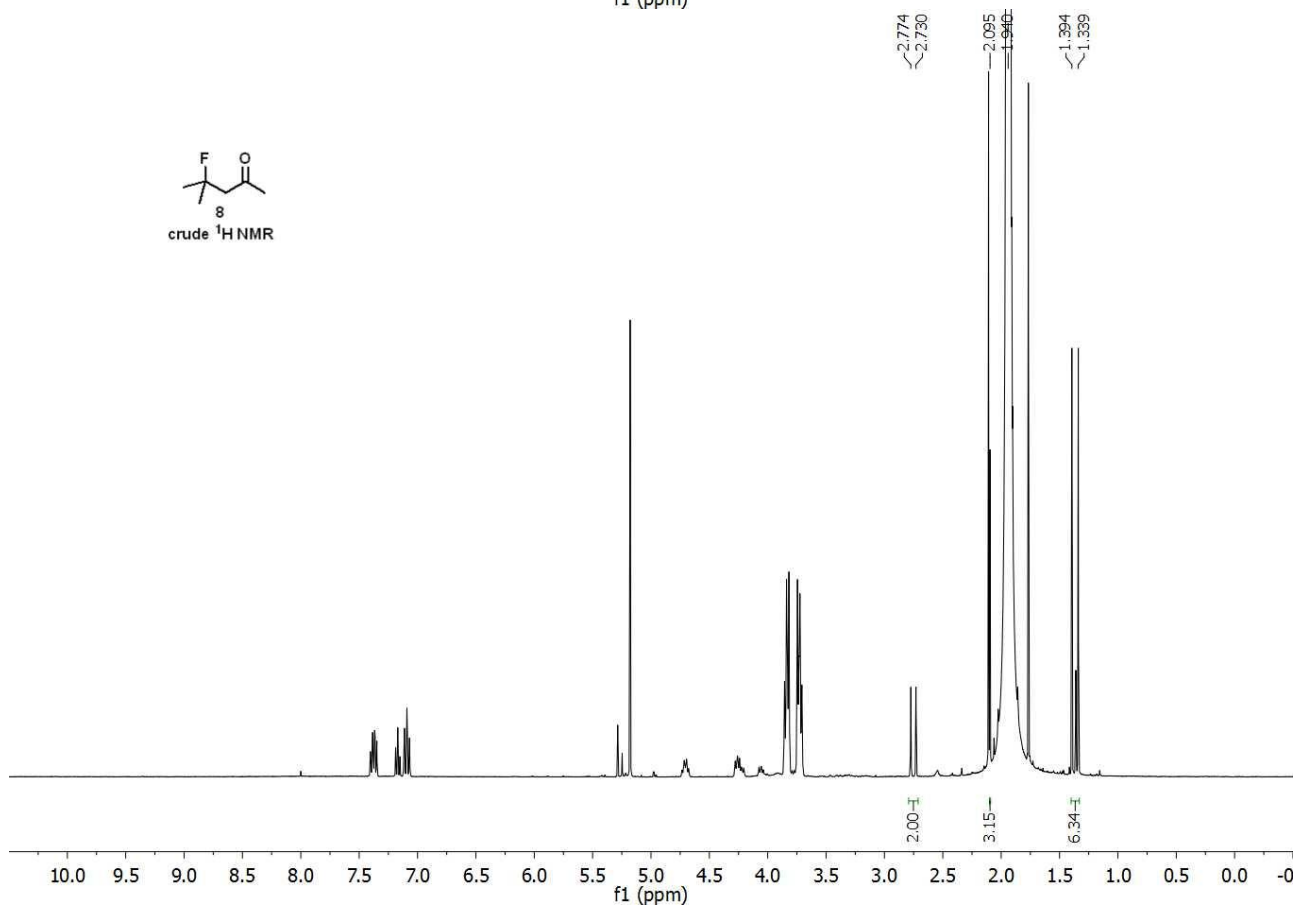
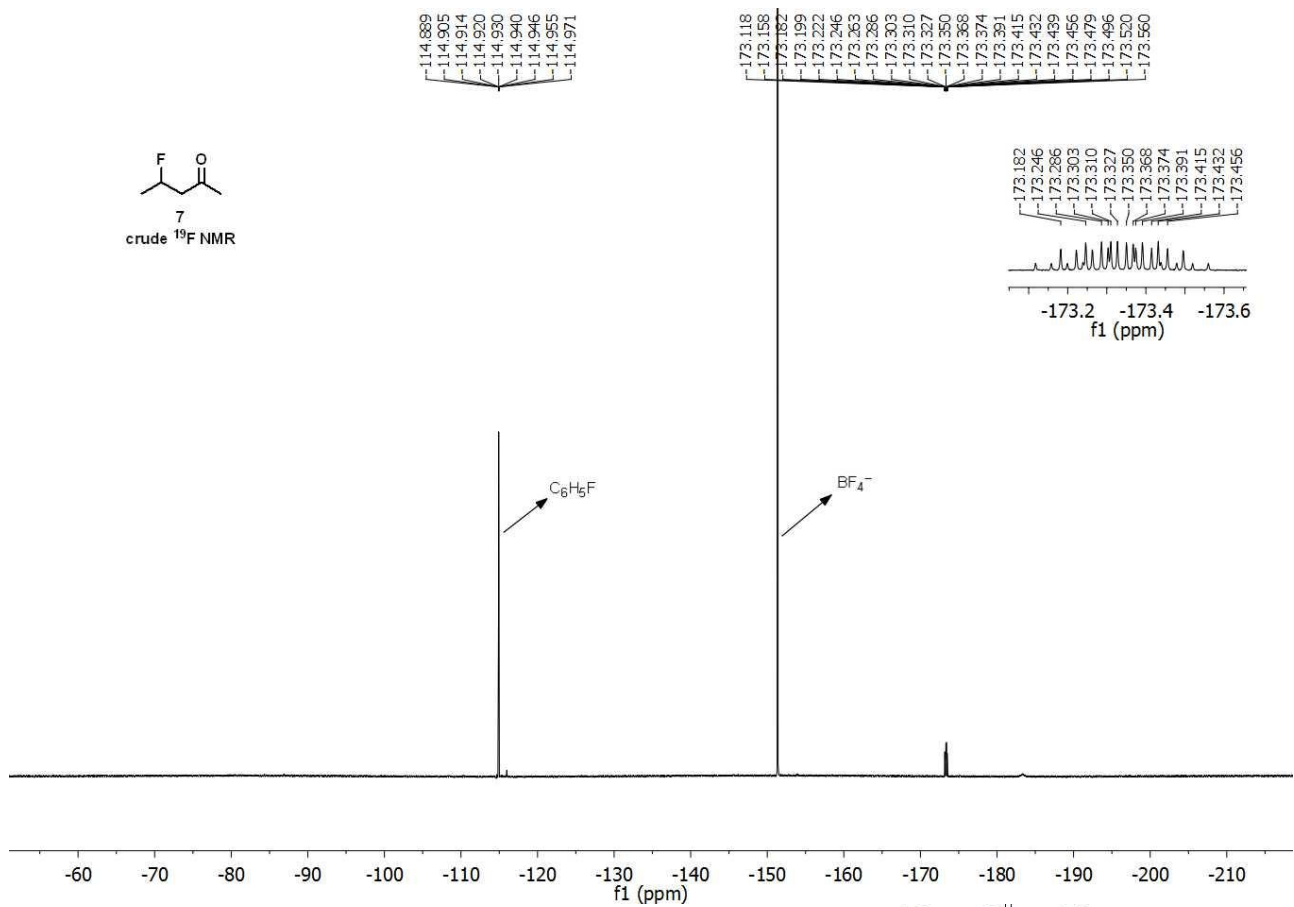
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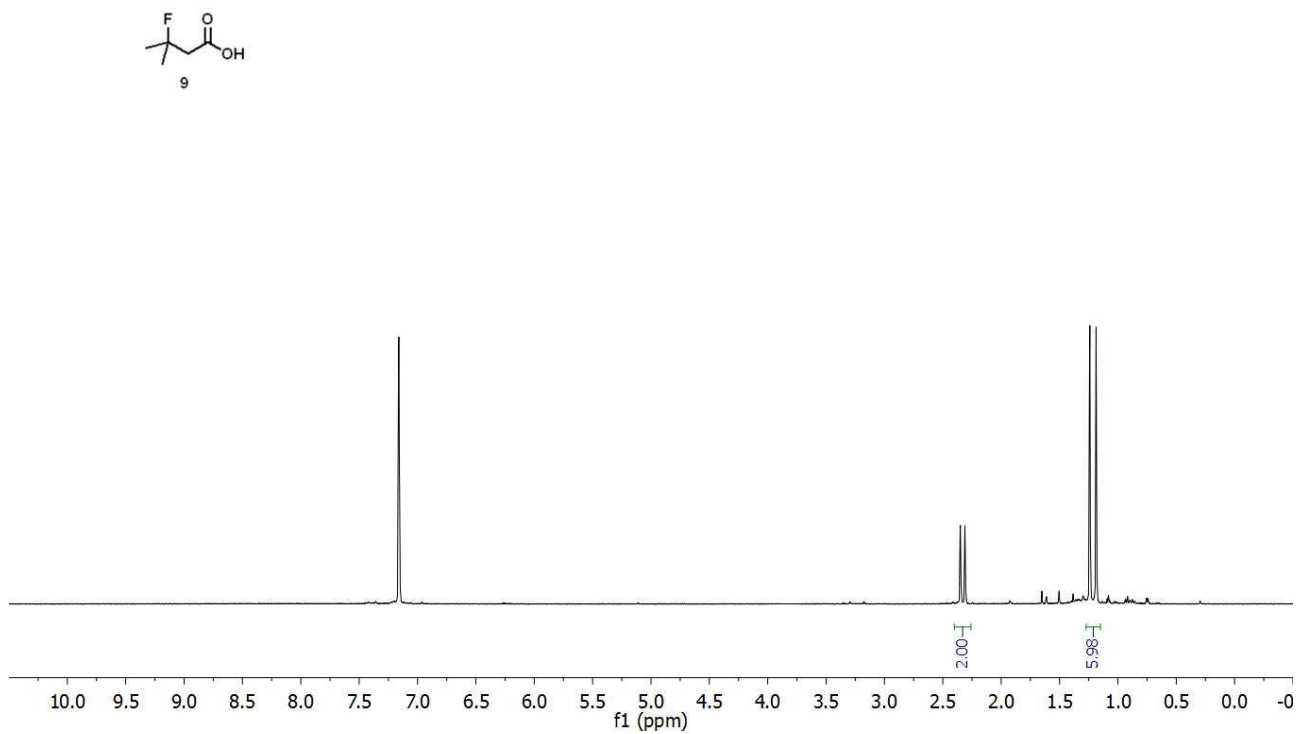
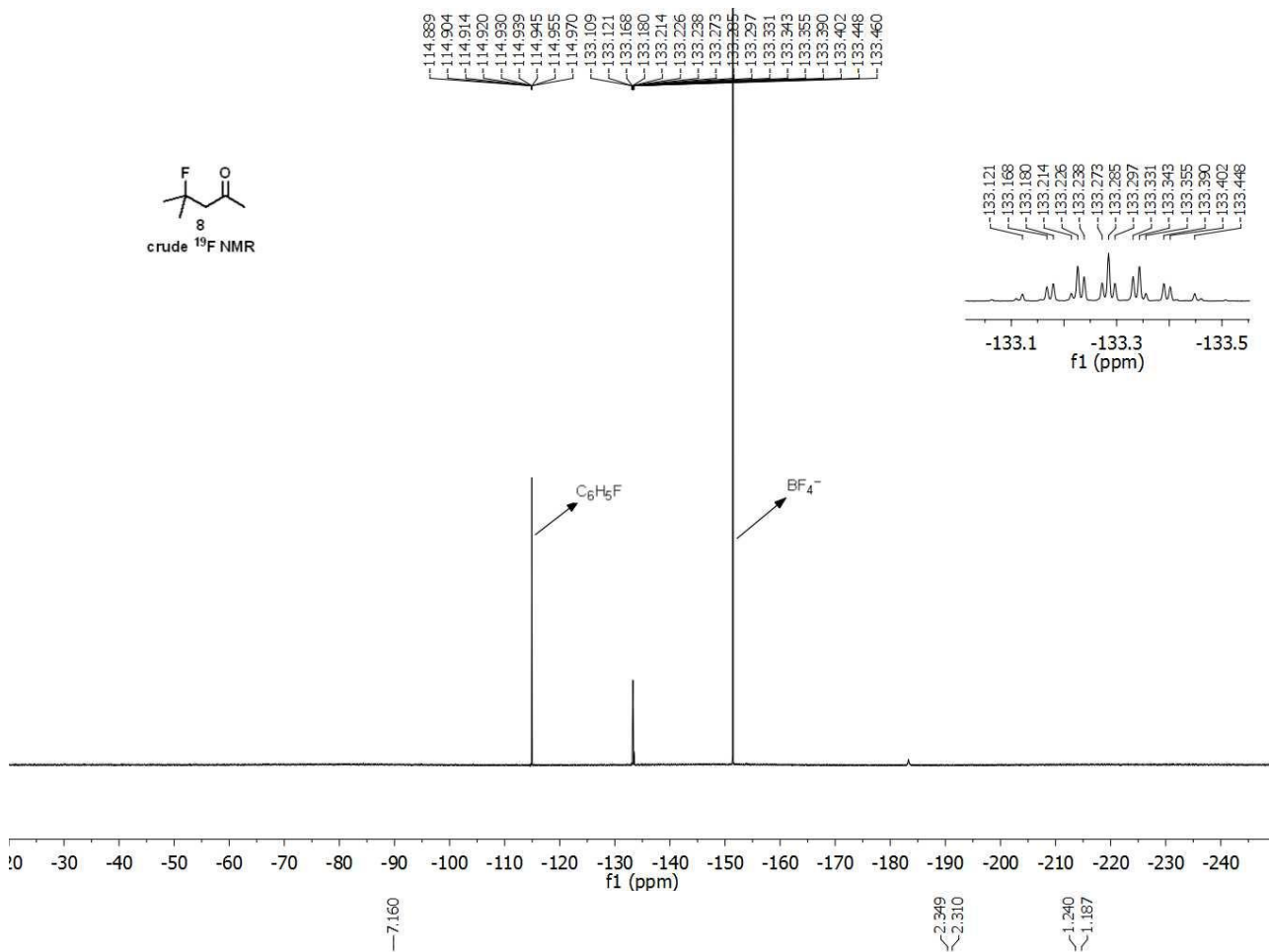


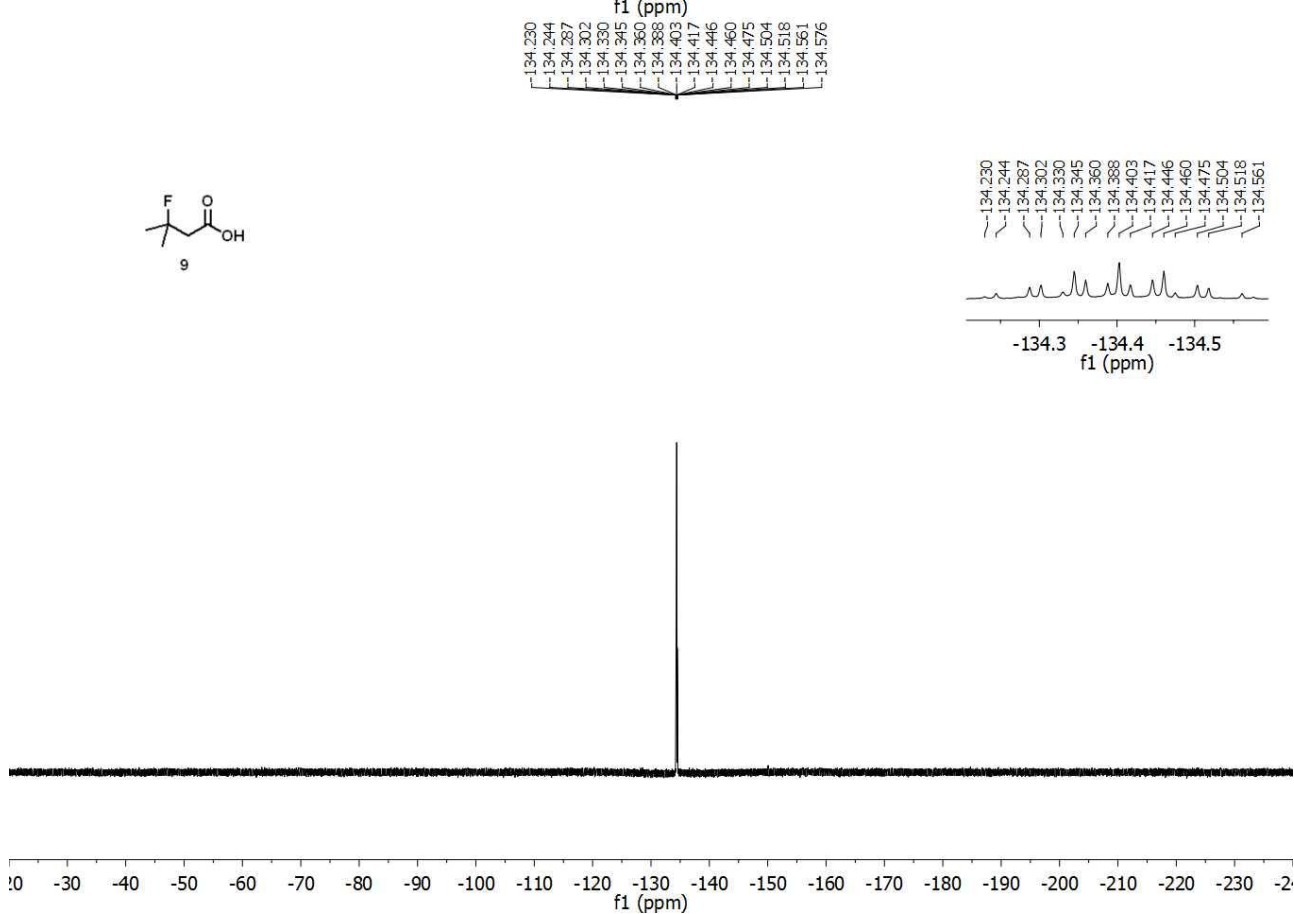
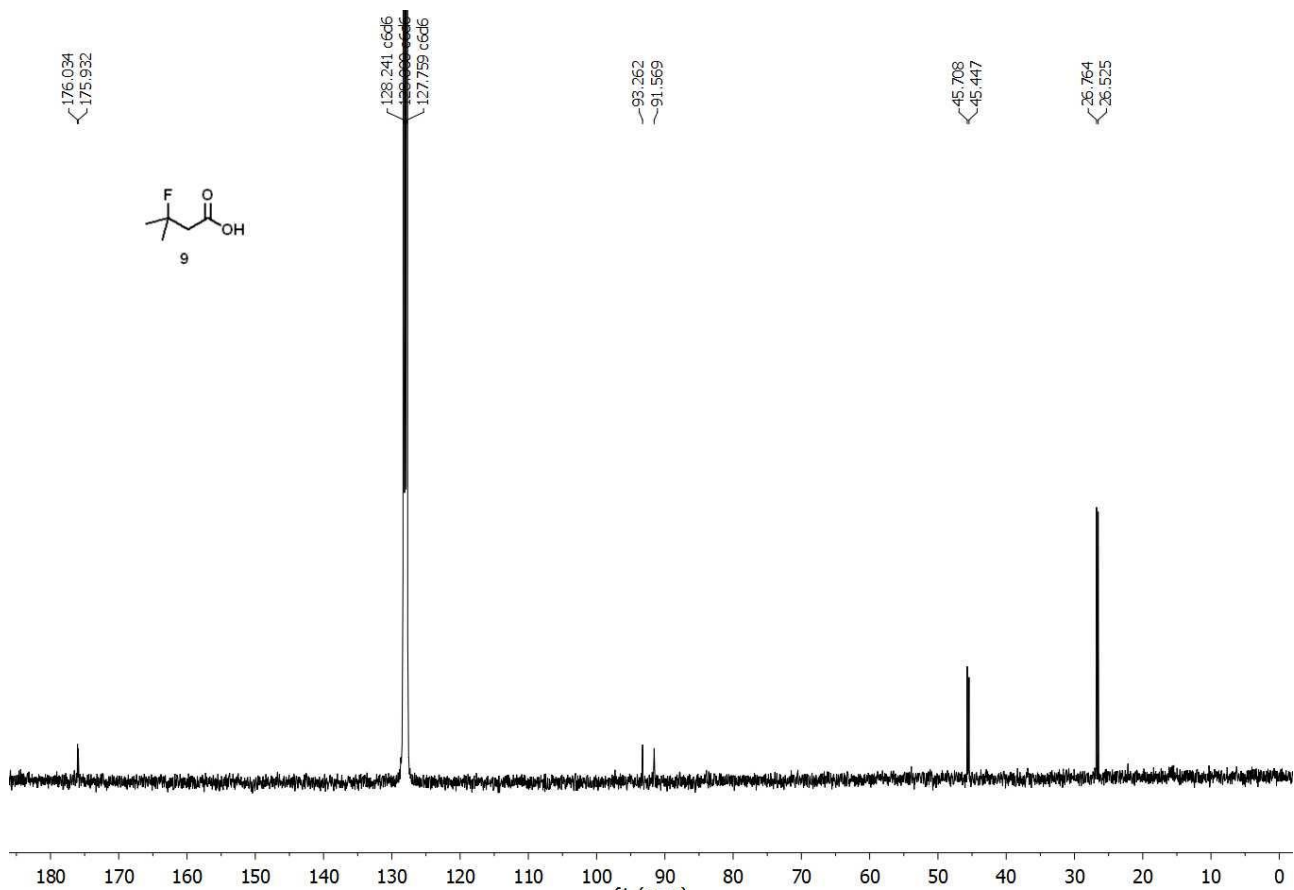


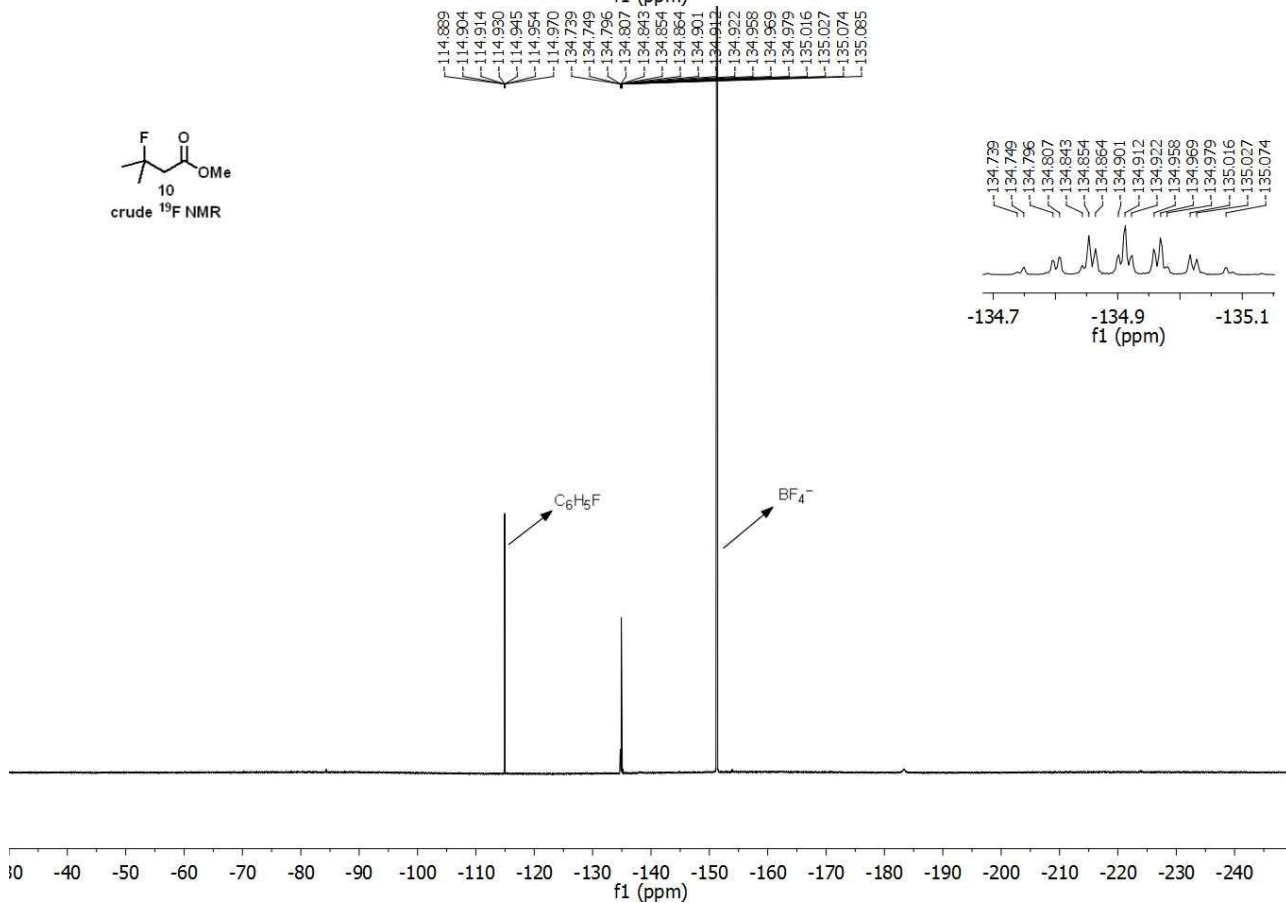
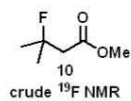
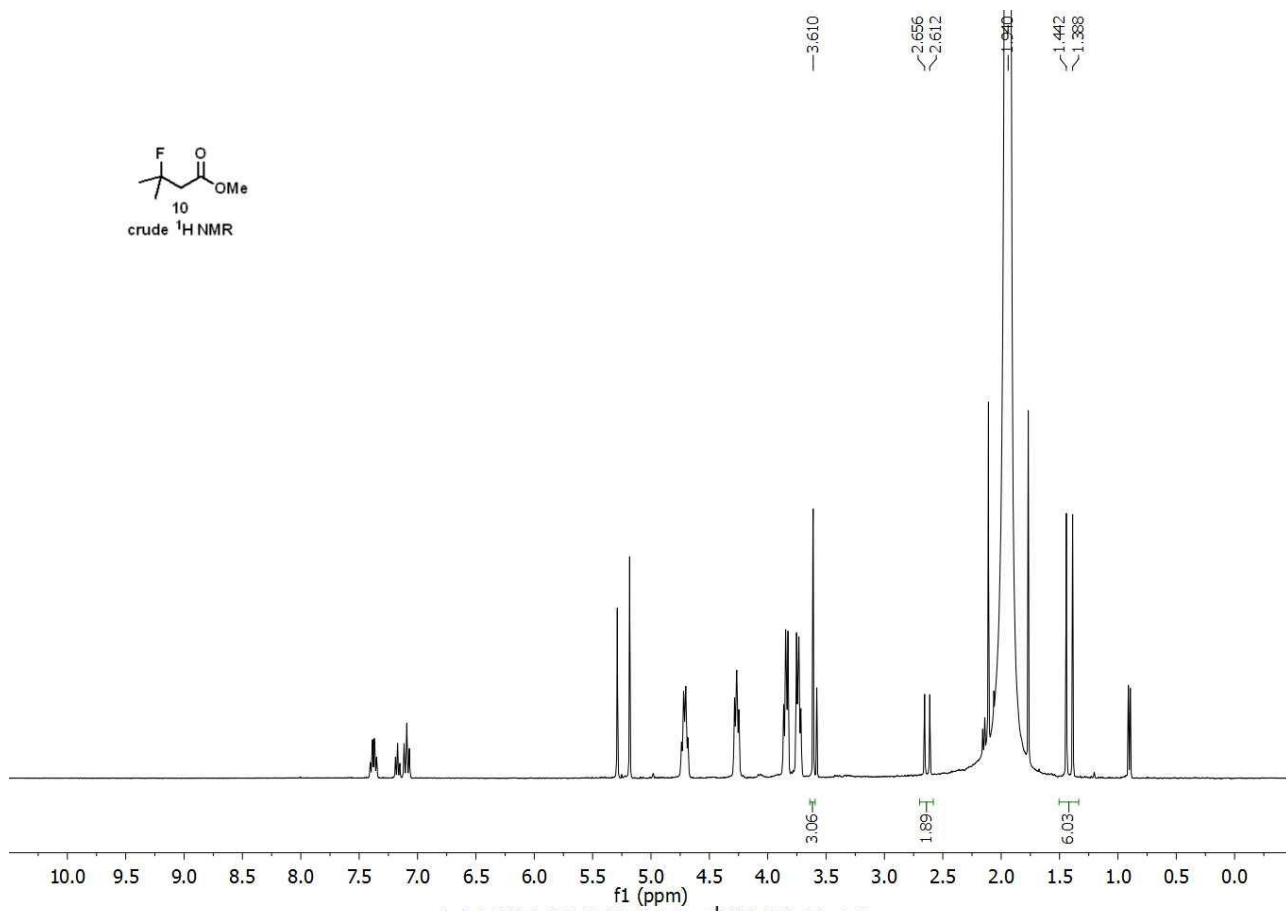
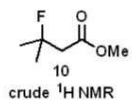


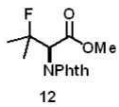




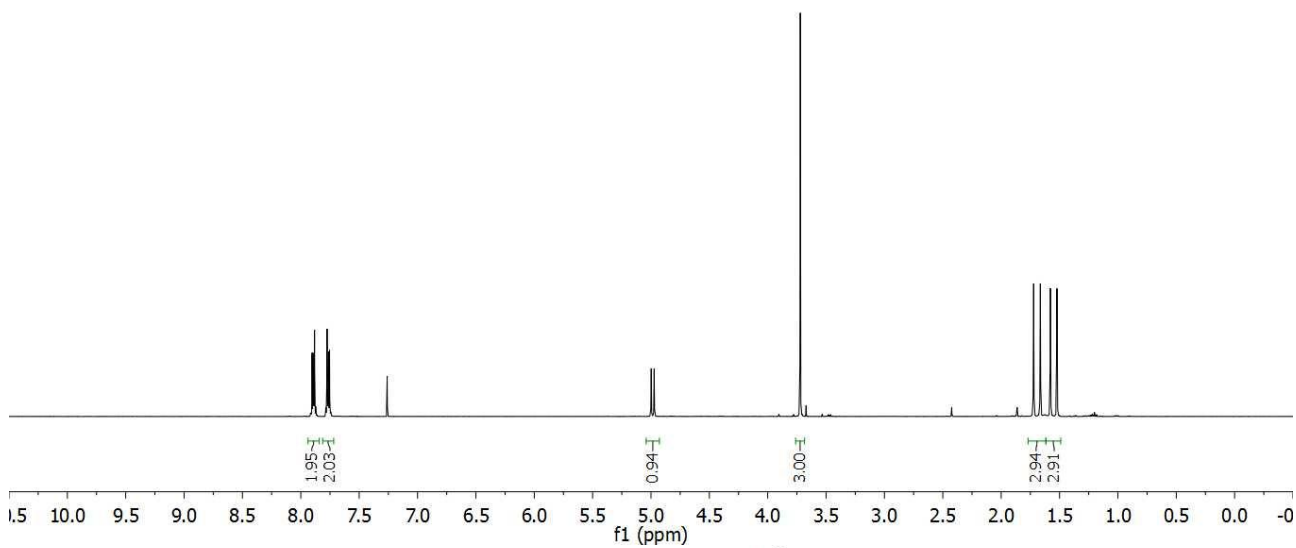




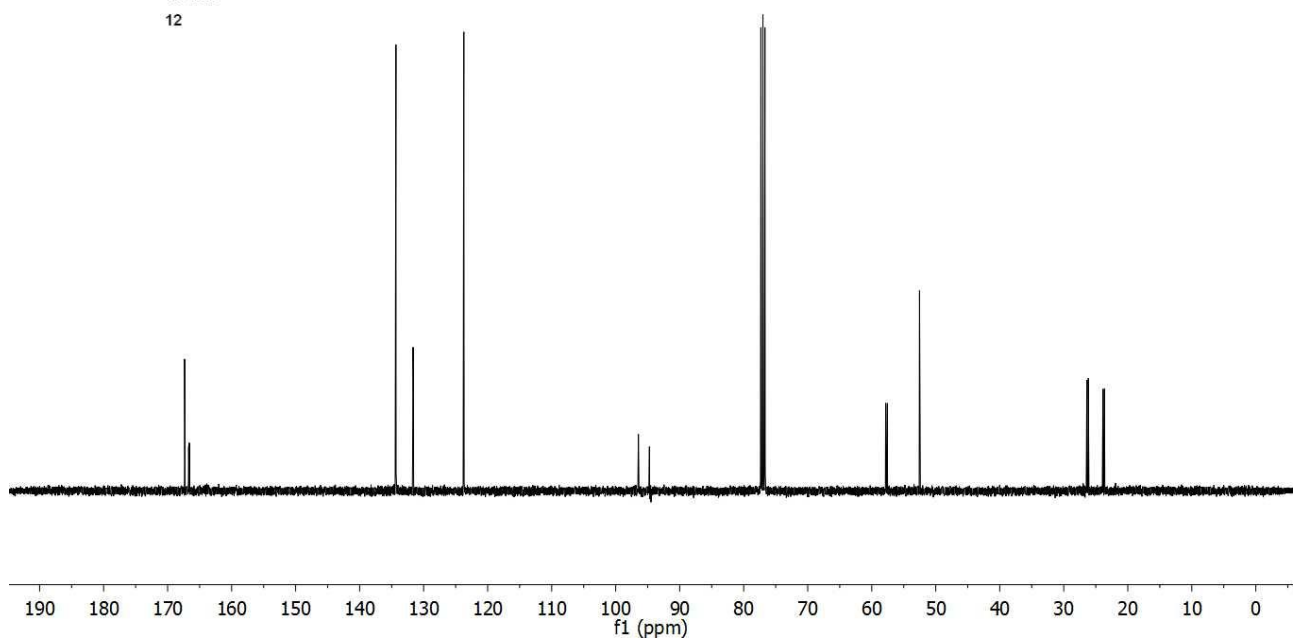
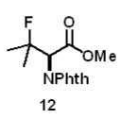


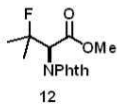


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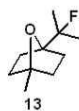
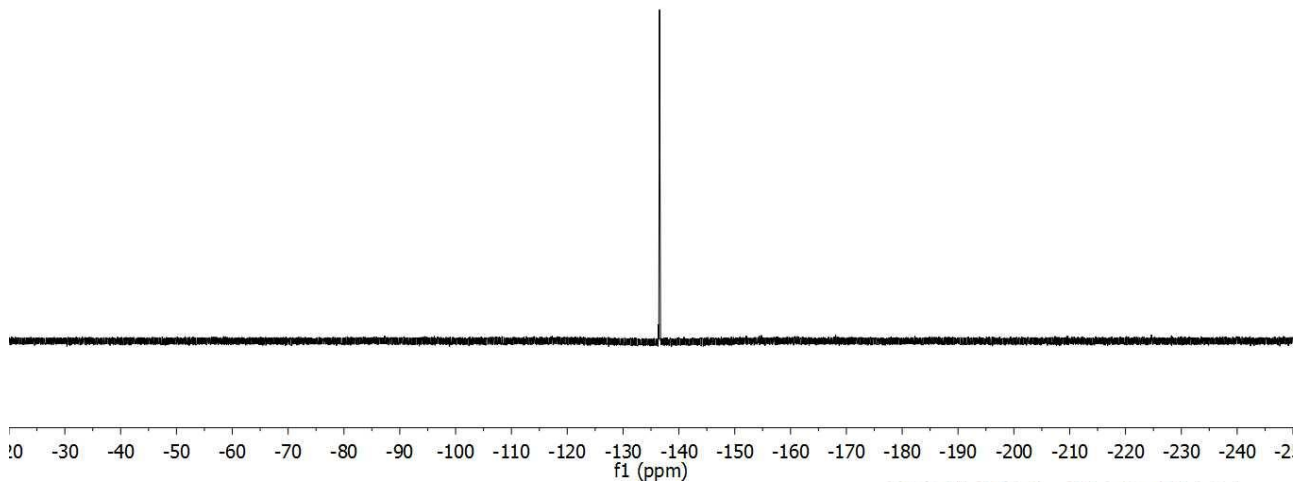
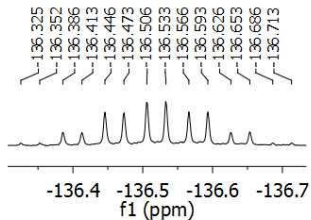


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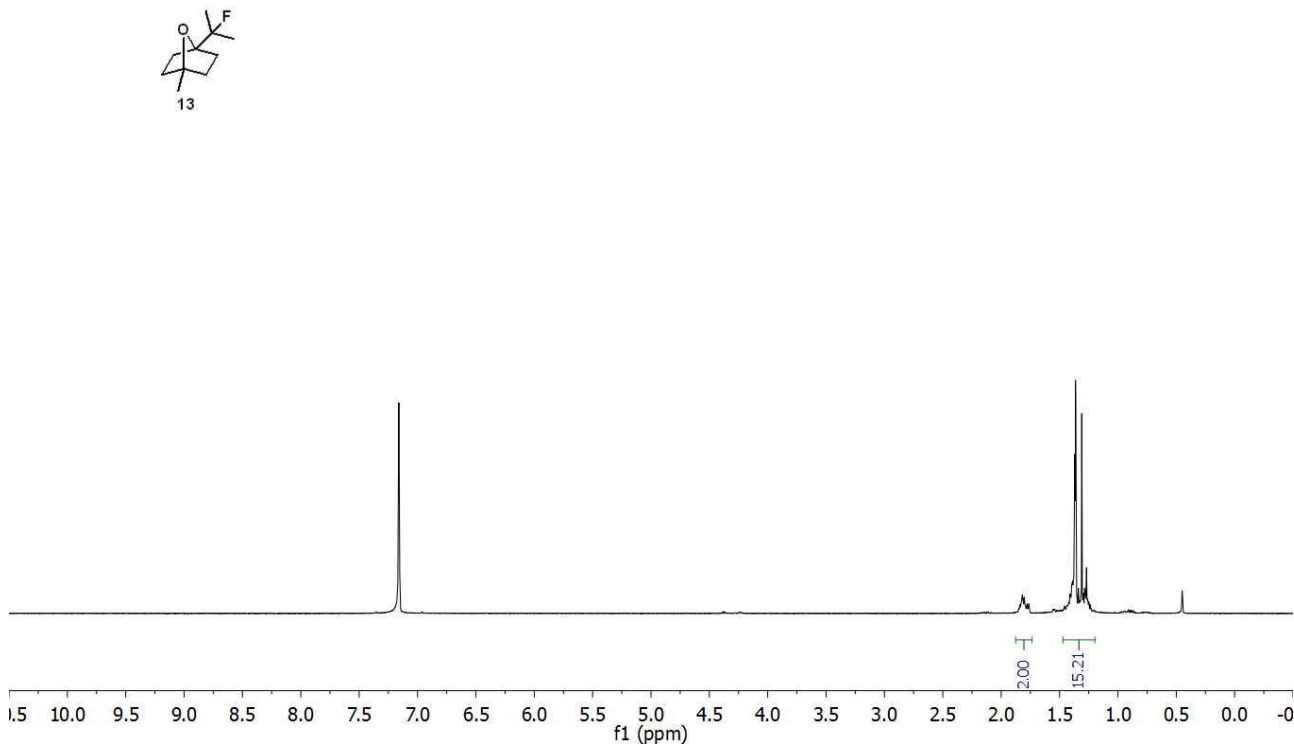


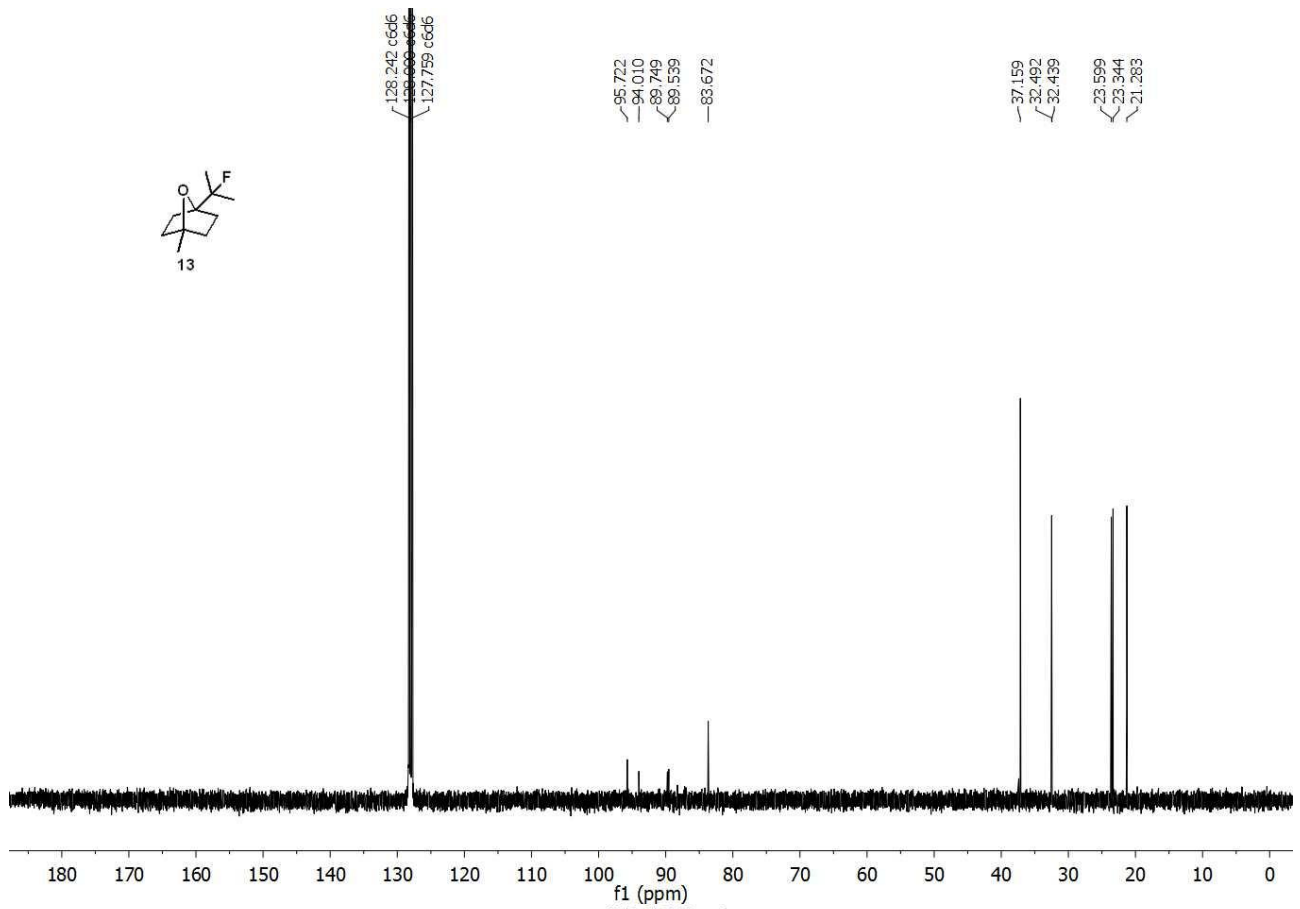


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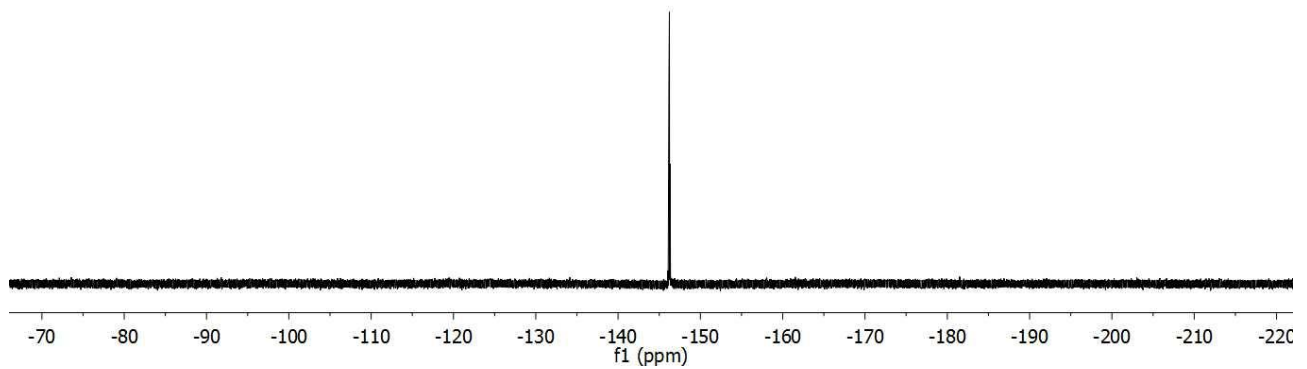
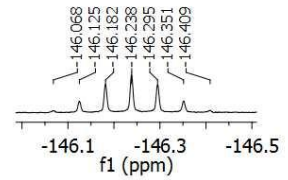
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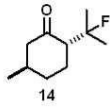
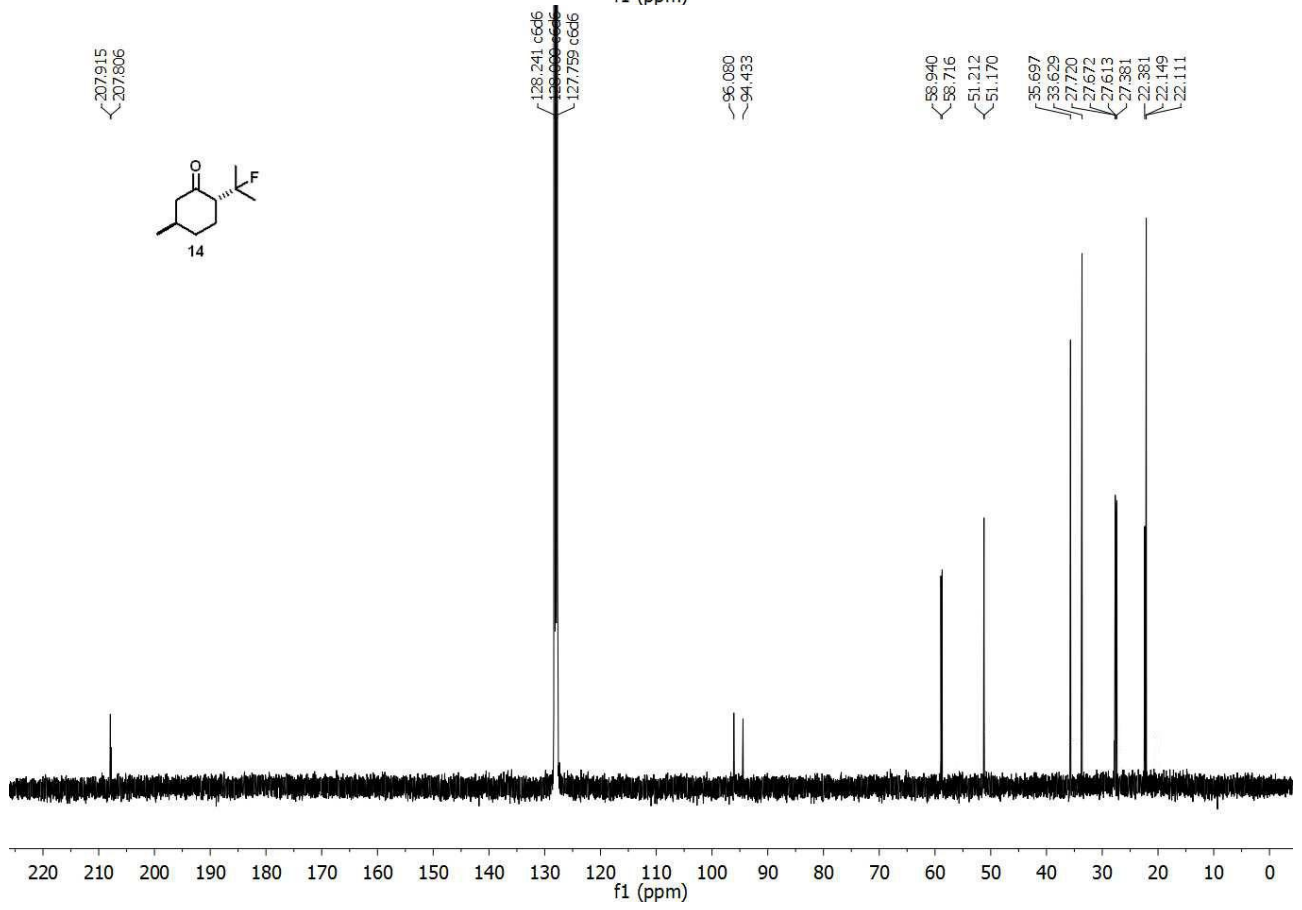
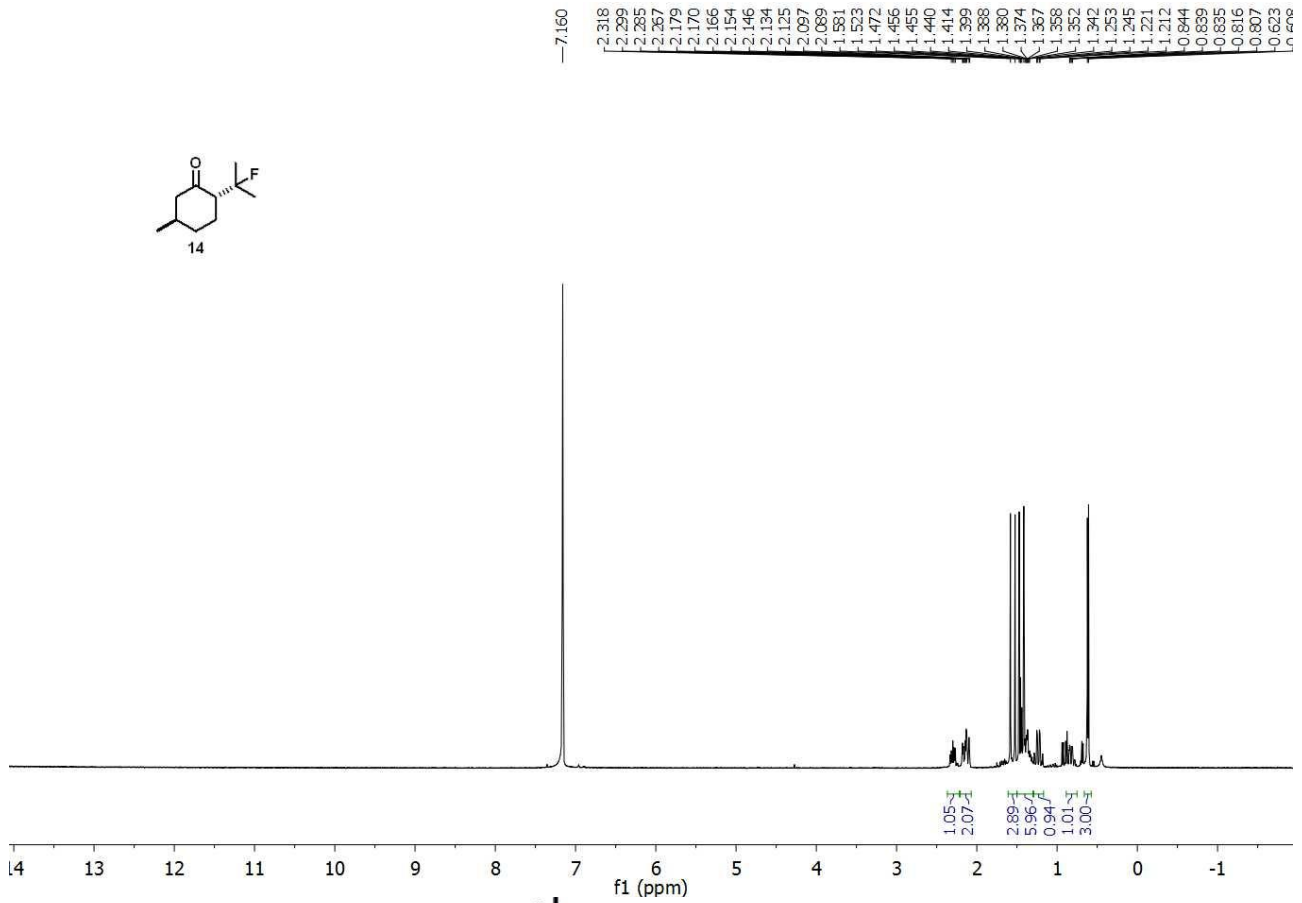
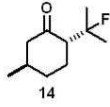


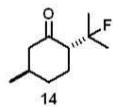


f1 (ppm)

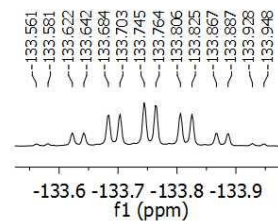
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146.351
146.409



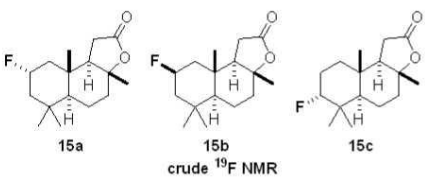




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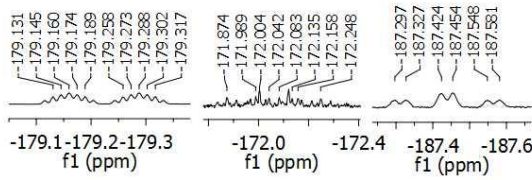


10 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220 -230 -240
f1 (ppm)



crude ¹⁹F NMR

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114.920
114.930
114.945
114.955
114.970
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171.912
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171.989
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172.042
172.083
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172.158
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172.213
172.248
172.268
179.160
179.257
187.424
187.454
187.548
187.581



C₆H₅F

BF₄⁻

-30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220 -230 -240 -250
f1 (ppm)

