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An Electrophilic Reagent for the Synthesis of OCHFMe-containing Molecules

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Supporting information

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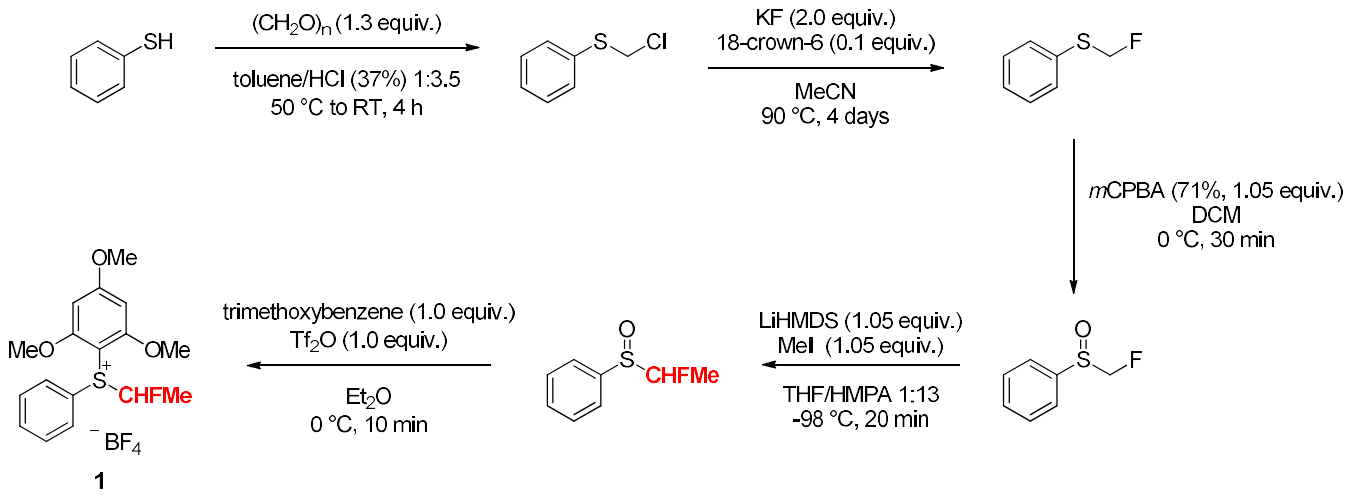
1. General information

All reactions were carried out using oven dried glassware and magnetic stirring under an atmosphere of argon unless otherwise stated. Flash chromatography was performed with silica gel (0.040-0.063 mm). Reverse phase chromatography was performed on a puriFlash® 215 using a puriFlash® C18HP column. Analytical thin layer chromatography was performed on silica gel aluminum plates with F-254 indicator and visualized by UV light (254 nm) and/or chemical staining with a $KMnO_4$ solution.^{1}H NMR spectra were recorded on a Bruker DXP 300 at 300.1 MHz, ^{13}C NMR spectra at 75.5 MHz and ^{19}F NMR spectra at 282.4 MHz. Chemical shifts (δ) are quoted in parts per million (ppm) relative to the residual solvent peak for $CDCl_3$ (δ_H = 7.26 ppm; δ_C = 77.0 ppm or relative to external $CFCl_3$: δ = 0 ppm), CH_3CN (δ_H = 1.94 ppm; δ_C = 118.2 ppm, 1.3 ppm or relative to external $CFCl_3$: δ = 0 ppm). The following abbreviations have been used: δ (chemical shift), *J* (coupling constant), br (broad), s (singlet), d (doublet), t (triplet), q (quadruplet), m (multiplet), dd (doublet of doublet), dq (doublet of quadruplet), td (triplet of doublet). High-resolution mass spectra (HRMS) were recorded on Waters LCT Premier. IR spectra were recorded on a PerkinElmer Spectrum 100, the wave numbers (ν) of recorded IR-signals (ATR) are quoted in cm^{-1}.

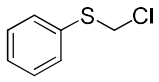
2. Materials

DCM, HMPA and thiophenol were distilled over CaH_2 prior to use. THF was distilled overNa/benzophenone prior to use. 18-Crown-6 (99%) and Cs_2CO_3 (99.9%) were purchased from Alfa Aesar. MeCN (99.9 %, sealed bottle over molecular sieves) and *m*CPBA (70-75%) were purchased from Acros Organics. Trifluoromethanesulfonic anhydride was purchased from Fluorochem. LiHMDS solution (1M in THF), methyl iodide (99%), paraformaldehyde (99%), potassium fluoride (spray dried) and trimethoxybenzene (> 99%) were purchased from Sigma Aldrich.

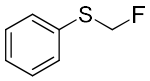
3. Synthesis of reagent 1



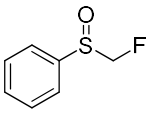
Procedures



Chloromethyl phenyl sulfide is commercially available (CAS number 7205-91-6) or can be synthesized following the slightly modified procedure reported by Cheng and Roush.[1] Paraformaldehyde (2.7 g, 91 mmol, 1.3 equiv.) was added to a mixture of toluene (20 mL) and HCl (37%, 70 mL). The resulting mixture was stirred at 50 °C for 10 min and then a solution of thiophenol (7.2 mL, 70 mmol, 1.0 equiv.) in toluene (20 mL) was added over 45 min at the same temperature. The reaction mixture was stirred at 50 °C for 1h and then at RT for 3h. The reaction mixture was flushed with Ar and the layers were separated. The aqueous layer was then extracted with DCM (3 x 30 mL). The combined organic layers were dried over $MgSO_4$ and concentrated under vacuum to furnish a yellow oil (10.5 g, 95 %) which was used without further purification. The data were consistent with the literature.[1]

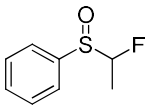


Fluoromethyl phenyl sulfide. Synthesized following the slightly modified procedure reported by Montgomery and Rawal.[2] Potassium fluoride (7.7 g, 132.4 mmol, 2.0 equiv.), 18-crown-6 (1.7 g, 6.6 mmol, 0.1 equiv.) and MeCN (44 mL) were added into a 250 mL flask followed bychloromethyl phenyl sulfide (10.5 g, 66.2 mmol, 1.0 equiv.). The reaction mixture was heated at 90 °C and stirred at this temperature for 4 days. The reaction mixture was cooled down to RT and flushed with Ar. Then, water (20 mL) and DCM (50 mL) were added and the layers were separated. The aqueous layer was extracted with DCM (3 x 50 mL) and then the combined organic layers were dried over $MgSO_4$ and concentrated under vacuum to furnish a yellow oil (9.2 g, 98 %) which was used without further purification. The data were consistent with the literature.[3] Alternatively, fluoromethyl phenyl sulfide can be synthesized from thioanisole in one step with 1-fluoropyridinium triflate (DCM, RT, 8 h).[4]

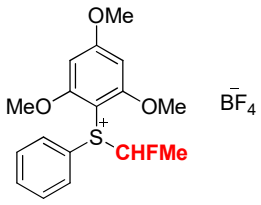


Phenyl fluoromethyl sulfoxide. Synthesized following the modified procedure reported by Olah and co-workers.[5] Fluoromethyl phenyl sulfide (8.0 g, 56.3 mmol, 1.0 equiv.) was dissolved in DCM (115 mL). The resulting solution was cooled down to 0 °C and *m*CPBA (71%, 14.4 g, 59.2 mmol, 1.05 eq.) was added portionwise. The reaction mixture was stirred for 30 min at 0 °C and 1M NaOH aqueous solution (50 mL) was slowly added at 0 °C. The layers were then separated and the aqueous layer was extracted with DCM (3 x 100 mL). The combined organic layers were dried over $MgSO_4$ and concentrated under vacuum. The crude reaction mixture was purified by flash column chromatography on silica gel (height 15 cm, width 4 cm, petroleum ether/diethyl ether, gradient: 60:40 to 0:100) to yield a colorless oil (5.6 g, 63%). $\mathbf{R_f}$ (petroleum ether/diethyl ether = 70:30): 0.11. **^{1}H NMR** ($CDCl_3$, 300.1 MHz) δ 7.70-7.63 (m, 2H), 7.60-7.53 (m, 3H), 5.08 (d, J = 48.0 Hz, 2H). **^{13}C NMR** ($CDCl_3$, 75.5 MHz)

δ 138.5, 132.0, 129.5, 124.6, 98.1 (d, J = 220.5 Hz). **^{19}F NMR** ($CDCl_3$, 282.4 MHz) δ -212.3 (t, J = 48.0 Hz). **IR** (cm^{-1}): 3058, 2929, 1445, 1090, 1054, 1021, 749, 689. **HRMS** (EI) calcd for C_7H_7FOS *m/z* 158.02016 $[M]^+$, found 158.01941. The data were consistent with the literature.[4]



((1-Fluoroethyl)sulfinyl)benzene. Phenyl fluoromethylsulfoxide (7.2 g, 45.5 mmol, 1 equiv.) and methyl iodide (3.1 mL, 50.05 mmol, 1.1 equiv.) were dissolved into a mixture of THF/HMPA (13:1, 91/6.8 mL). The resulting solution was cooled down to –98 °C (Et_2O/dry ice bath) and LiHMDS (C = 1 mol.L^{-1} in THF, 50 mL, 50.05 mmol, 1.1 equiv.) was added dropwise. The reaction mixture was stirred at -98 °C for 20 min and then saturated aqueous NH_4Cl solution (40 mL) and water (45 mL) were added. The mixture was warmed to RT and Et_2O (150 mL) was added. The layers were separated and the aqueous layer was extracted with Et_2O (2 x 150 mL). The combined organic layers were washed with water (1 x 200 mL), dried over $MgSO_4$ and concentrated under vacuum. The crude reaction mixture was purified by flash column chromatography on silica gel (height 17 cm, width 4 cm, petroleum ether/diethyl ether, gradient: 60:40 to 0:100) to yield the two diastereoisomers. Diastereoisomer 1 was obtained as a colorless liquid (2.8 g, 36 %). Diastereoisomer 2 was obtained as a colorless liquid with 16% of diastereoisomer 1 (3.4 g, 43 %) Diastereoisomer 1: $\mathbf{R_f}$ (petroleum ether/diethyl ether = 70:30): 0.36. **^{1}H NMR** ($CDCl_3$, 300.1 MHz) δ 7.68-7.45 (m, 5H), 5.10 (dq, J = 48.3, 6.3 Hz, 1H), 1.56 (dd, J = 23.7, 6.3 Hz, 3H). **^{13}C NMR** ($CDCl_3$, 75.5 MHz) δ 139.6 (d, J = 5.3 Hz), 131.6, 129.2, 124.6 (d, J = 1.5 Hz), 104.9 (d, J = 215.9 Hz), 12.7 (d, J = 20.4 Hz). **^{19}F NMR** ($CDCl_3$, 282.4 MHz) δ -171.8 (dq, J = 48.0, 22.6, 1F). **IR** (cm^{-1}): 3093, 3026, 1741, 1712, 1262, 1242, 1033, 751, 710, 524. **HRMS** (EI) calcd for C_8H_9FOS *m/z* 172.0358 $[M]^+$, found 172.0355 (-1.54 ppm). Diastereoisomer 2: $\mathbf{R_f}$ (petroleum ether/diethyl ether = 70:30): 0.18. **^{1}H NMR** ($CDCl_3$, 300.1 MHz) δ 7.67-7.48 (m, 5H), 5.27 (dq, J = 48.0, 6.3 Hz, 1H), 1.46 (dd, J = 23.7, 6.0 Hz, 3H). **^{13}C NMR** ($CDCl_3$, 75.5 MHz) δ 138.3 (d, J = 3.0 Hz), 131.8, 129.1, 125.2 (d, J = 0.8 Hz), 102.1 (d, J = 222.7 Hz), 13.9 (d, J = 21.1 Hz). **^{19}F NMR** ($CDCl_3$, 282.4 MHz) δ -178.2 (dd, J = 48.0, 22.6 Hz, 1F). **IR** (cm^{-1}): 3063, 2932, 1444, 1114, 1082, 1044, 1022, 882, 748, 690, 542. **HRMS** (EI) calcd for C_8H_9FOS *m/z* 172.0358 $[M]^+$, found 172.0355.

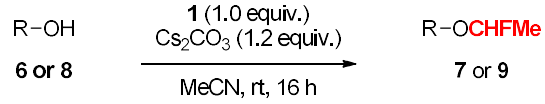


1

(1-fluoroethyl)(phenyl)(2,4,6-trimethoxyphenyl)sulfoniumtetrafluoroborate 1. All the solvents used for this reaction were technical grade solvents. ((1-Fluoroethyl)sulfinyl)benzene (1 g, 5.8 mmol, 1.0 equiv.) and trimethoxybenzene (0.98 g, 5.8 mmol, 1.0 equiv.) were dissolved in Et_2O (58 mL). The resulting solution was cooled down to 0 °C and Tf_2O (0.98 mL, 5.8 mmol, 1.0 equiv.) was added dropwise. The reaction mixture was stirred for 10 min

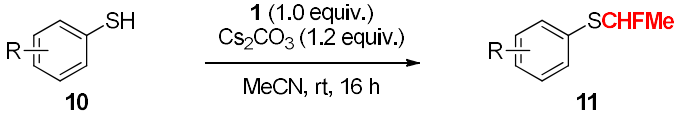
and then Et_2O was removed with a syringe. Then, Et_2O (25 mL) was added and removed with a syringe. This operation was repeated twice. DCM (25 mL) was then added and the solution was washed with aqueous 1M $NaBF_4$ solution (3 x 30 mL). The resulting organic layer was dried over $MgSO_4$ and concentrated under vacuum to furnish **1** as a 3:1 mixture of two diastereoisomers as a dark-purple solid (1.6 g, 67 %) which was used without further purification. **mp**: 119-120 °C. **^{1}H NMR** (CD_3CN, 300.1 MHz) δ 7.90-7.61 (m, 5H, major + minor dia), 7.28-7.02 (m, 1H, major + minor dia), 6.49-6.41 (m, 2H, major + minor dia), 3.98-3.91 (m, 9H, major + minor dia), 1.92-1.77 (m, 3H, major + minor dia). **^{13}C NMR** (CD_3CN, 75.5 MHz) δ 170.5 (major dia), 170.1 (minor dia), 164.2 (minor dia), 163.8 (major dia), 135.3 (minor dia), 134.9 (major dia), 132.1 (minor dia), 132.0 (d, *J* = 1.5 Hz, minor dia), 132.0 (major dia), 130.4 (d, *J* = 1.5 Hz, major dia), 105.2 (d, *J* = 225.7, major dia), 101.8 (d, *J* = 234.8 Hz, minor dia), 94.0 (major dia), 93.9 (minor dia), 58.2 (major dia), 58.0 (minor dia), 57.5 (major dia), 57.4 (minor dia), 19.5 (d, *J* = 19.6 Hz, minor dia), 18.9 (d, *J* = 18.9Hz, major dia). Note that two quaternary carbons are missing. **^{19}F NMR** (CD_3CN, 282.4 MHz) δ - 150.2 (s, BF_4 minor dia), -150.3 (s, BF_4 major dia), -155.4 (dq, *J* = 48.0, 25.4 Hz, major dia, CH**F**Me), -155.7 (dq, *J* = 48.0, 25.4 Hz, minor dia, CH**F**Me). **IR** (cm^{-1}): 2947, 1594, 1576, 1476, 1352, 1233, 1212, 1049, 481. **HRMS** (ESI^+) calcd for $C_{17}H_{20}FO_3S^+$ *m/z* 323.1117 $[M]^+$, found 323.1117.

4. General procedure for the synthesis of derivatives 7 and 9



(1-Fluoroethyl)(phenyl)(2,4,6-trimethoxyphenyl)sulfoniumtetrafluoroborate **1** (3:1 mixture of diastereosiomers, 82 mg, 0.2 mmol, 1.0 equiv.) was dissolved in MeCN (1 mL). The phenol derivative **6** or the alcohol derivative **8** (0.24 mmol, 1.2 equiv.) was added followed by Cs_2CO_3 (85 mg, 0.24 mmol, 1.2 equiv.). The resulting heterogeneous solution was stirred at rt for 16 h. The reaction mixture was filtered over cotton and then the solvent was removed under reduced pressure. The crude product was purified by column chromatography either on normal or reverse phase to furnish the corresponding *O*-monofluoroethyl derivative **7** or **9**.

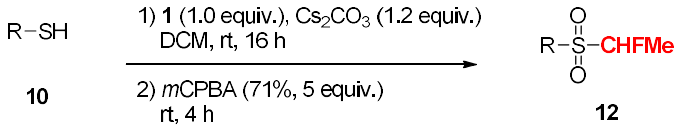
5. General procedure for the synthesis of derivatives 11



(1-Fluoroethyl)(phenyl)(2,4,6-trimethoxyphenyl)sulfoniumtetrafluoroborate **1** (3:1 mixture of diastereosiomers, 82 mg, 0.2 mmol, 1.0 equiv.) was dissolved in MeCN (1 mL). The thiophenol derivative **10** (0.24 mmol, 1.2 equiv.) was added followed by Cs_2CO_3 (85 mg, 0.24

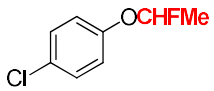
mmol, 1.2 equiv.). The resulting heterogeneous solution was stirred at rt for 16 h. The reaction mixture was filtered over cotton and then the solvent was removed under reduced pressure. The crude product was purified by column chromatography to furnish the corresponding the [(1-fluoroethyl)thio]benzene **11**.

6. General procedure for the synthesis of derivative 12



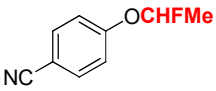
(1-Fluoroethyl)(phenyl)(2,4,6-trimethoxyphenyl)sulfoniumtetrafluoroborate **1** (3:1 mixture of diastereosiomers, 82 mg, 0.2 mmol, 1.0 equiv.) was dissolved in DCM (1 mL). The thiophenol derivative or the thiol derivative **10** (0.24 mmol, 1.2 equiv.) was added followed by Cs_2CO_3 (85 mg, 0.24 mmol, 1.2 equiv.). The resulting heterogeneous solution was stirred at rt for 16 h. Then, *m*CPBA (71%, 243 mg, 1.2 mmol, 5 equiv.) was added and the reaction mixture was stirred at rt for 4h. 5 mL of a 1M NaOH aqueous solution and 5 mL of DCM were added. The layers were separated and the aqueous layer was extracted with DCM (3 x 10 mL). The organic layers were combined, dried over $MgSO_4$ and concentrated under vacuum. The crude product was purified by column chromatography to furnish the corresponding *S*-monofluoroethyl derivative **12**.

7. Purification and characterization of products 7, 9, 11 and 12



7a

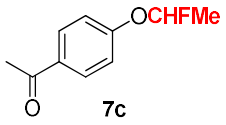
4-Chloro-(1-fluoroethoxy)benzene 7a. The product was purified by flash column chromatography on silica gel (height 12 cm, width 1.2 cm, 100% pentane) to yield **7a** as a yellow oil (29 mg, 83%). $\mathbf{R_f}$ (pentane): 0.63. **^{1}H NMR** ($CDCl_3$, 300.1 MHz): δ 7.20 (d, J = 8.7 Hz, 2H), 6.93 (d, J = 8.7 Hz, 2H), 5.83 (dq, J = 62.7, 5.1 Hz, 1H), 1.58 (dd, J = 19.8, 4.8 Hz, 3H). **^{13}C NMR** ($CDCl_3$, 75.5 MHz): δ 154.9 (d, J = 3.0 Hz), 129.5, 128.3, 118.3 (d, J = 1.5 Hz), 107.9 (d, J = 217.4 Hz), 21.0 (d, J = 24.9 Hz). **^{19}F NMR** ($CDCl_3$, 282.4 MHz): δ -116.5 (dq, J = 62.1, 19.8 Hz, 1F). **IR** (cm^{-1}): 2927, 2856, 1489, 1402, 1229, 1128, 1075, 945, 859, 827, 670. **HRMS** (EI) calcd for C_8H_8ClFO *m/z* 174.0247 $[M]^+$, found 174.0256.



7b

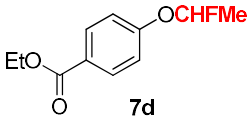
4-Cyano-(1-fluoroethoxy)benzene 7b. The product was purified by flash column chromatography on silica gel (height 12 cm, width 1 cm, 100 % pentane) to yield **7b** a colorless oil (28 mg, 85%). $\mathbf{R_f}$ (pentane): 0.48. **^{1}H NMR** ($CDCl_3$, 300.1 MHz): δ 7.62 (d, J =

8.7 Hz, 2H), 7.12 (d, *J* = 8.7 Hz, 2H), 6.01 (dq, *J* = 61.5, 4.8 Hz, 1H), 1.69 (dd, *J* = 20.1, 4.8 Hz, 3H). **^{13}C NMR** ($CDCl_3$, 75.5 MHz): δ 159.3 (d, *J* = 2.3 Hz), 134.0, 118.7, 117.1 (d, *J* = 2.3 Hz), 106.9 (d, *J* = 220.5 Hz), 106.5, 20.8 (d, *J* = 24.2 Hz). **^{19}F NMR** ($CDCl_3$, 282.4 MHz): δ -118.3 (dq, *J* = 62.1, 19.8 Hz, 1F). **IR** (cm^{-1}): 3004, 2946, 2227, 1606, 1506, 1404, 1239, 1121, 1077, 942, 837, 545. **HRMS** (EI) calcd for C_9H_8FNO *m/z* 165.0589 $[M]^+$, found 165.0594.



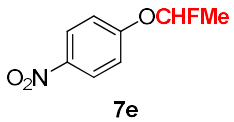
7c

Ethyl 4-acetyl-(1-fluoroethoxy)benzene 7c. The product was purified by flash column chromatography on silica gel (height 12 cm, width 1.2 cm, pentane/acetone, gradient: 100:0 to 92:8) to yield **7c** a colorless oil (31 mg, 85%). **R_f** (pentane/acetone 90:10): 0.31. **^{1}H NMR** ($CDCl_3$, 300.1 MHz): δ 7.95 (d, *J* = 8.7 Hz, 2H), 7.10 (d, *J* = 8.7 Hz, 2H), 6.04 (dq, *J* = 61.8, 4.8 Hz, 1H), 2.57 (s, 3H), 1.69 (dd, *J* = 19.8, 4.8 Hz, 3H). **^{13}C NMR** ($CDCl_3$, 75.5 MHz): δ 196.8, 159.9, 132.2, 130.5, 116.2 (d, *J* = 1.5 Hz), 107.0 (d, *J* = 219.0 Hz), 26.4, 20.9 (d, *J* = 24.9 Hz). **^{19}F NMR** ($CDCl_3$, 282.4 MHz): δ -117.7 (dq, *J* = 62.1, 19.8 Hz, 1F). **IR** (cm^{-1}): 3003, 1678, 1602, 1404, 1359, 1235, 1125, 1075, 942, 837, 589. **HRMS** (EI) calcd for $C_{10}H_{11}FO_2$ *m/z* 182.0738 $[M]^+$, found 182.0743.



7d

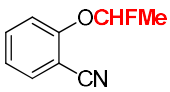
4-Ethoxycarbonyl-(1-fluoroethoxy)benzene 7d. The product was purified by reverse phase chromatography (column 55 g, $MeCN/H_2O$, gradient : 5:95 to 100:0) to yield **7d** as a yellow oil (29 mg, 68%). **^{1}H NMR** ($CDCl_3$, 300.1 MHz): δ 8.02 (d, *J* = 8.7 Hz, 2H), 7.08 (d, *J* = 8.7 Hz, 2H), 6.03 (dq, *J* = 62.1, 4.8 Hz, 1H), 4.35 (q, *J* = 7.2 Hz, 2H), 1.68 (dd, *J* = 19.8, 4.8 Hz, 3H), 1.38 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** ($CDCl_3$, 75.5 MHz): δ 166.1, 159.7 (d, *J* = 2.3 Hz), 131.5, 125.2, 116.1 (d, *J* = 1.5 Hz), 107.0 (d, *J* = 219.0 Hz), 60.8, 20.9 (d, *J* = 24.9 Hz), 14.3. **^{19}F NMR** ($CDCl_3$, 282.4 MHz): δ -117.5 (dq, *J* = 62.1, 19.8 Hz, 1F). **IR** (cm^{-1}): 2984, 1712, 1608, 1404, 1274, 1234, 1171, 1098, 1110, 1076, 944, 854, 769. **HRMS** (EI) calcd for $C_{11}H_{13}FO_3$ *m/z* 212.0848 $[M]^+$, found 212.0857.



7e

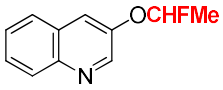
4-Nitro-(1-fluoroethoxy)benzene 7e. The product was purified by flash column chromatography on silica gel (height 12 cm, width 1.2 cm, petroleum ether/ethyl acetate, gradient 90:10 to 80:20) to yield **7e** as a colorless oil (30 mg, 81%). **R_f** (petroleum ether/diethyl ether 80:20): 0.55. **^{1}H NMR** ($CDCl_3$, 300.1 MHz): δ 8.22 (d, *J* = 9.3 Hz, 2H), 7.15 (d, *J* = 9.3 Hz, 2H), 6.06 (dq, *J* = 61.5, 4.8 Hz, 1H), 1.71 (dd, *J* = 20.4, 4.8 Hz, 3H). **^{13}C NMR** ($CDCl_3$, 75.5 MHz): δ 160.9 (d, *J* = 2.3 Hz,), 143.1, 125.8, 116.5, (d, *J* = 1.5 Hz), 106.9 (d, *J* = 220.5 Hz), 20.8 (d, *J* = 24.2 Hz). **^{19}F NMR** ($CDCl_3$, 282.4 MHz): δ -118.7 (dq, *J* = 62.1, 19.8 Hz,

1F). **IR** (cm^{-1}): 2924, 2856, 1594, 1496, 1339, 1242, 1111, 1074, 936, 847, 752, 660. **HRMS** (EI) calcd for $C_8H_8FNO_3$ *m/z* 185.0488 $[M]^+$, found 185.0493.



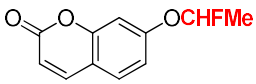
7f

2-Cyano-(1-fluoroethoxy)benzene 7f. The product was purified by flash column chromatography on silica gel (height 12 cm, width 1.2 cm, petroleum ether/ethyl acetate, gradient 90:10 to 80:20) to yield **7f** as a yellow oil (31 mg, 94%). $\mathbf{R_f}$ (petroleum ether/diethyl ether 80:20): 0.63. **^{1}H NMR** ($CDCl_3$, 300.1 MHz): δ 7.57-7.46 (m, 2H), 7.22-7.05 (m, 2H), 5.95 (dq, *J* = 61.5, 4.8 Hz, 1H), 1.69 (dd, *J* = 20.1, 4.8 Hz, 3H). **^{13}C NMR** ($CDCl_3$, 75.5 MHz): δ 157.7 (d, *J* = 2.3 Hz), 134.4, 133.7, 123.3, 115.9 (d, *J* = 2.3 Hz), 115.7, 107.8 (d, *J* = 221.2 Hz), 103.5, 20.8 (d, *J* = 24.2 Hz). **^{19}F NMR** ($CDCl_3$, 282.4 MHz): δ -117.7 (dq, *J* = 62.1, 19.8 Hz), -117.7 (dq, *J* = 62.1, 19.8 Hz). **IR** (cm^{-1}): 3005, 2947, 2231, 1600, 1492, 1452, 1404, 1290, 1244, 1126, 1078, 943, 867, 757, 495. **HRMS** (EI) calcd for C_9H_8FNO *m/z* 165.0589 $[M]^+$, found 165.0593.



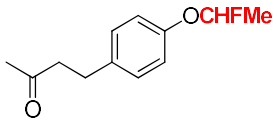
7g

3-(1-Fluoroethoxy)quinoline 7g. The product was purified by reverse phase chromatography (column 55 g, CH_3CN/H_2O, gradient: 5:95 to 100:0) to yield **7g** as a yellow oil (33 mg, 86%). **^{1}H NMR** ($CDCl_3$, 300.1 MHz): δ 8.74 (br. s, 1H), 8.08 (d, *J* = 8.4 Hz, 1H), 7.82-7.72 (m, 2H), 7.67-7.50 (m, 2H), 6.08 (dq, *J* = 62.1, 4.8 Hz, 1H), 1.75 (dd, *J* = 20.1, 4.8 Hz, 3H). **^{13}C NMR** ($CDCl_3$, 75.5 MHz): δ 144.7, 144.3, 129.1, 127.9, 127.3, 127.2, 118.3, 118.3, 108.1 (d, *J* = 219.0 Hz), 21.0 (d, *J* = 24.2 Hz). Note that one carbon overlap. **^{19}F NMR** ($CDCl_3$, 282.4 MHz): δ -117.5 (dq, *J* = 62.1, 19.8 Hz, 1F). **IR** (cm^{-1}): 3005, 2947, 1603, 1400, 1340, 1209, 1185, 1115, 1074, 989, 939, 854, 782, 750. **HRMS** (EI) calcd for $C_{11}H_{10}FNO$ *m/z* 191.0746 $[M]^+$, found 191.0752.



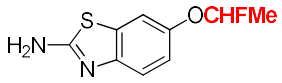
7h

7-(1-Fluoroethoxy)-*2H*-chromen-2-one 7h. The product was purified by reverse phase chromatography (column 55 g, CH_3CN/H_2O, gradient: 5:95 to 100:0) to yield **7h** as a colorless oil (33 mg, 79%). **^{1}H NMR** ($CDCl_3$, 300.1 MHz): δ 7.64 (d, *J* = 9.6 Hz, 1H), 7.41 (d, *J* = 8.4 Hz, 1H), 7.04-6.92 (m, 2H), 6.29 (d, *J* = 9.6, 1H), 6.01 (dq, *J* = 61.8, 4.8 Hz, 1H), 1.69 (dd, *J* = 20.1, 4.8 Hz, 3H). **^{13}C NMR** ($CDCl_3$, 75.5 MHz): δ 160.7, 158.9 (d, *J* = 3.0 Hz), 155.3, 143.1, 128.9, 114.4, 114.3, 113.6 (d, *J* = 1.5 Hz), 107.1 (d, *J* = 219.7 Hz), 104.3 (d, *J* = 1.5 Hz), 20.8 (d, *J* = 24.9 Hz). **^{19}F NMR** ($CDCl_3$, 282.4 MHz): δ -118.2 (dq, *J* = 59.3, 22.6 Hz, 1F). **IR** (cm^{-1}): 3082, 3005, 1725, 1626, 1403, 1237, 1142, 1072, 858, 841, 752, 590. **HRMS** (EI) calcd for $C_{11}H_9FO_3$ *m/z* 208.0535 $[M]^+$, found 208.0538.



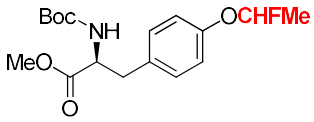
7i

4-(3-Oxobutyl)-(1-fluoroethoxy)benzene 7i. The product was purified by reverse phase chromatography (column 55 g, CH_3CN/H_2O, gradient: 5:95 to 100:0) to yield **7i** as a yellow oil (29 mg, 69%). **^{1}H NMR** ($CDCl_3$, 300.1 MHz): δ 7.12 (d, *J* = 8.7 Hz, 2H), 6.97 (d, *J* = 8.7 Hz, 2H), 5.91 (dq, *J* = 62.7, 4.8 Hz, 1H), 2.90-2.66 (m, 4H), 2.13 (s, 3H), 1.64 (dd, *J* = 19.5, 4.8 Hz, 3H). **^{13}C NMR** ($CDCl_3$, 75.5 MHz): δ 207.9, 154.7 (d, *J* = 3.0 Hz), 135.7, 129.3, 117.0 (d, *J* = 1.5 Hz), 107.9 (d, *J* = 215.9 Hz), 45.2, 30.0, 28.8, 21.1 (d, *J* = 25.7 Hz). **^{19}F NMR** ($CDCl_3$, 282.4 MHz): δ -115.7 (dq, *J* = 62.1, 19.8 Hz, 1F). **IR** (cm^{-1}): 3006, 2939, 1713, 1510, 1402, 1224, 1128, 1071, 945, 853, 822. **HRMS** (EI) calcd for $C_{15}H_{15}FO_2$ *m/z* 210.10561 $[M]^+$, found 210.1062.



7j

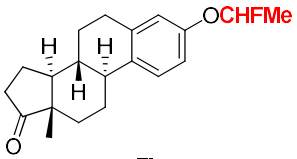
6-(1-Fluoroethoxy)benzo[*d*]thiazol-2-amine 7j. The product was purified by flash column chromatography on silica gel (height 12 cm, width 1.2 cm, petroleum ether/ethyl acetate, gradient 90:10 to 60:40) to yield **7j** as a beige oil (22 mg, 52%). **R_f** (petroleum ether/diethyl ether 80:20): 0.05. **^{1}H NMR** ($CDCl_3$, 300.1 MHz): δ 7.44 (d, *J* = 8.7 Hz, 1H), 7.33 (d, *J* = 2.4 Hz, 1H), 7.03 (dd, *J* = 8.7, 2.4 Hz, 1H), 5.89 (dq, *J* = 63.0, 4.8 Hz, 1H), 5.59 (br. s, 2H), 1.66 (dd, *J* = 19.6, 4.8 Hz, 3H). **^{13}C NMR** ($CDCl_3$, 75.5 MHz): δ 165.4, 152.0 (d, *J* = 2.3 Hz), 148.1, 132.3, 119.4, 116.5 (d, *J* = 1.5 Hz), 109.8 (d, *J* = 1.5 Hz), 108.9 (d, *J* = 216.7 Hz), 21.2 (d, *J* = 25.7 Hz). **^{19}F NMR** ($CDCl_3$, 282.4 MHz): δ -115.1 (dq, *J* = 62.1, 19.8 Hz, 1F). **IR** (cm^{-1}): 3398, 3072, 1645, 1538, 1466, 1407, 1262, 1206, 1131, 1074, 954, 855, 816, 433. **HRMS** (EI) calcd for $C_9H_9FN_2OS$ *m/z* 212.0419 $[M]^+$, found 212.0415.



7k

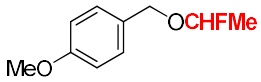
***N*-(*tert*-Butoxycarbonyl)-4-fluoroethoxy-L-tyrosine methyl ester derivative 7k**. The product was purified by flash column chromatography on silica gel (height 12 cm, width 1.2 cm, petroleum ether/ethyl acetate, gradient 90:10 to 60:40) and then by reverse phase chromatography (column 55 g, CH_3CN/H_2O, gradient: 5:95 to 100:0) to yield **7k** as a 1:1.4 mixture of two diastereosiomers as a yellow oil (21 mg, 31%). **^{1}H NMR** ($CDCl_3$, 300.1 MHz): δ 7.10-7.03 (m, 2H, major + minor dia), 7.01-6.95 (m, 2H, major + minor dia), 5.92 (dq, *J* = 63.0, 4.8 Hz, 1H, major + minor dia), 5.03-4.91 (m, 1H, major + minor dia), 4.60-4.49 (m, 1H, major + minor dia), 3.71 (s, 3H, major + minor dia), 3.14-2.97 (m, 2H, major + minor dia), 1.65 (dd, *J* = 19.6, 4.8 Hz, 3H, major + minor dia), 1.41 (s, 9H, major + minor dia). **^{13}C NMR** ($CDCl_3$, 75.5 MHz): δ 172.3 (major + minor dia), 155.5 (major + minor dia), 130.7 (major + minor dia), 130.4 (major + minor dia), 117.0 (major + minor dia), 115.4 (major + minor dia),

107.8 (d, J = 216.7 Hz, major + minor dia), 80.1 (major + minor dia), 54.4 (major + minor dia), 52.2 (major + minor dia), 37.5 (major + minor dia), 28.3 (major + minor dia), 21.1 (d, J = 24.9 Hz, major + minor dia). **^{19}F NMR** ($CDCl_3$, 282.4 MHz): δ -116.0 (dq, J = 65.0, 19.8 Hz, major dia), -116.0 (dq, J = 62.1, 19.8 Hz, minor dia). **IR** (cm^{-1}): 3365, 2932, 1711, 1510, 1366, 1226, 1163, 1129, 1071, 1017, 947, 858, 731. **HRMS** (EI) calcd for $C_{17}H_{24}FNO_5$ *m/z* 341.1638 $[M]^+$, found 341.1628.



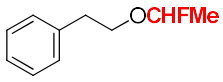
7l

(8*R*,9*S*,13*S*,14*S*)-3-(1-fluoroethoxy)-13-methyl-7,8,9,11,12,13,15,16-octahydro-6*H*-cyclopenta[α]phenanthren-17(14*H*)-one 7l. The product was purified by reverse phase chromatography (column 55 g, CH_3CN/H_2O, gradient: 5:95 to 100:0) to yield **7l** as a 1:1 mixture of two diastereoisomers as a colorless oil (44 mg, 70%). **^{1}H NMR** ($CDCl_3$, 300.1 MHz): δ 7.28-7.21 (m, 1H, major + minor dia), 6.90-6.79 (m, 2H, major + minor dia), 5.93 (dq, J = 63.0, 4.8 Hz, 1H, major + minor dia), 2.96-2.86 (m, 2H, major + minor dia), 2.58-2.45 (m, 1H, major + minor dia), 2.46-1.21 (m, 16H, major + minor dia), 0.89 (s, 3H, major + minor dia). **^{13}C NMR** ($CDCl_3$, 75.5 MHz): δ 220.8, 154.4 (d, J = 2.3 Hz, major + minor dia), 138.0 (d, J = 1.5 Hz, major + minor dia), 134.6 (major + minor dia), 126.4 (major dia), 126.4 (minor dia), 117.1 (d, J = 1.5 Hz, major dia), 117.0 (d, J = 1.5 Hz, minor dia), 114.4 (major + minor dia), 107.9 (d, J = 216.7 Hz, major dia), 107.9 (d, J = 216.7 Hz, minor dia), 50.3 (major + minor dia), 47.9 (major + minor dia), 44.0 (major dia), 43.9 (minor dia), 38.1 (major dia), 38.1 (minor dia), 35.8 (major + minor dia), 31.5 (major + minor dia), 29.5 (major dia), 29.5 (minor dia), 26.4 (major + minor dia), 25.8 (major + minor dia), 21.5 (major + minor dia), 21.1 (d, J = 24.9 Hz, major + minor dia), 13.8 (major + minor dia). **^{19}F NMR** ($CDCl_3$, 282.4 MHz): δ -115.4 (dq, J = 62.1, 19.8 Hz, major dia), -115.4 (dq, J = 62.1, 19.8 Hz, minor dia). **IR** (cm^{-1}): 2929, 2863, 1729, 1498, 1402, 1241, 1127, 1072, 959, 852, 732. **HRMS** (EI) calcd for $C_{20}H_{25}FO_2$ *m/z* 316.1838 $[M]^+$, found 316.1851.



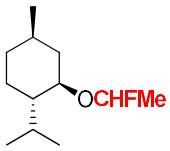
9a

1-((1-fluoroethoxy)methyl)-4-methoxybenzene 9a. The product was purified by flash column chromatography on silica gel (height 15 cm, width 1.2 cm, pentane/diethyl ether, gradient 100:0 to 70:30) to yield **9a** as a yellow oil (11 mg, 30%). **R_f** (pentane/diethyl ether 90:10): 0.44. **^{1}H NMR** ($CDCl_3$, 300.1 MHz): δ 7.34 (d, J = 8.7 Hz, 2H), 6.90 (d, J = 8.7 Hz, 2H), 6.35 (dq, J = 56.4, 5.1 Hz, 1H), 5.15 (s, 2H), 3.81 (s, 3H), 1.55 (dd, J = 20.7, 5.1 Hz, 3H). **^{13}C NMR** ($CDCl_3$, 75.5 MHz): δ 160.1, 130.4, 126.6, 114.0, 104.3 (d, J = 223.5 Hz), 70.2, 55.3, 19.6 (d, J = 24.2 Hz). **^{19}F NMR** ($CDCl_3$, 282.4 MHz): δ -121.6 (dq, J = 56.5, 19.8 Hz, 1F). **IR** (cm^{-1}): 2925, 2858, 1761, 1251, 1092, 1065, 905, 699.



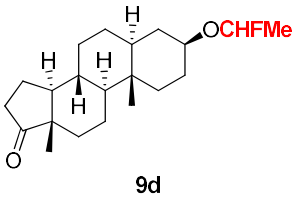
9b

(2-(1-Fluoroethoxy)ethyl)benzene 9b. The product was purified by flash column chromatography on silica gel (height 12 cm, width 1.2 cm, pentane/acetone, gradient: 98:2 to 90:10)to yield **9b** as a yellow oil (20 mg, 59%). $\mathbf{R_f}$ (pentane/acetone 90:10): 0.53. **^{1}H NMR** ($CDCl_3$, 300.1 MHz): δ 7.31-7.11 (m, 5H), 6.26 (dq, *J* = 56.4, 5.1 Hz, 1H), 4.32 (td, *J* = 7.2, 1.8 Hz, 2H), 2.94 (t, *J* = 7.2 Hz, 2H), 1.49 (dd, *J* = 20.7, 5.1 Hz, 3H). **^{13}C NMR** ($CDCl_3$, 75.5 MHz): δ 136.9, 128.9, 128.7, 126.9, 104.3 (d, *J* = 223.5 Hz), 69.0, 35.0, 19.7 (d, *J* = 23.4 Hz). **^{19}F NMR** ($CDCl_3$, 282.4 MHz): δ -121.7 (dq, *J* = 56.5, 19.8 Hz, 1F). **IR** (cm^{-1}): 2923, 2854, 1762, 1255, 1094, 1066, 906, 699.



9c

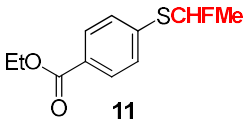
(1*S*,2*R*,4*R*)-2-(1-fluoroethoxy)-1-isopropyl-4-methylcyclohexane 9c. The product was purified by flash column chromatography on silica gel (height 18 cm, width 1.2 cm, pentane/acetone, gradient: 100 to 96:4) to yield **9c** as a 1:1 mixture of two diastereoisomers as a colorless oil (19 mg, 47%). $\mathbf{R_f}$ (pentane/acetone 90:10): 0.79. **^{1}H NMR** ($CDCl_3$, 300.1 MHz): δ 6.35 (dq, *J* = 56.7, 5.1 Hz, major dia), 6.34 (dq, *J* = 56.7, 5.1 Hz, minor dia), 4.58 (td, *J* = 10.8, 4.5 Hz, 0.5 H, major dia), 4.58 (td, *J* = 10.8, 4.5 Hz, 0.5 H, minor dia), 2.21-1.86 (m, 2H, major + minor dia), 1.77-0.73 (m, 18H, major + minor dia). **^{13}C NMR** ($CDCl_3$, 75.5 MHz): δ 104.0 (d, *J* = 222.7 Hz, major dia), 104.0 (d, *J* = 222.7 Hz, minor dia), 79.5 (d, *J* = 2.3 Hz, major + minor dia), 46.9 (major dia), 46.9 (minor dia), 40.5 (major dia), 40.5 (minor dia), 34.0 (major + minor dia), 31.4 (major dia), 31.4 (minor dia), 26.0 (major dia), 26.0 (minor dia), 23.2 (major dia), 23.2 (minor dia), 21.9 (major dia), 21.9 (minor dia), 20.7 (major dia), 20.7 (minor dia), 19.7 (d, *J* = 24.2 Hz, major dia), 19.6 (d, *J* = 23.4 Hz, minor dia). **^{19}FNMR** ($CDCl_3$, 282.4 MHz): δ - 121.2-121.6 (m,1F). **IR** (cm^{-1}): 2956, 2927, 2872, 1759, 1257, 1132, 1094, 954, 906, 789. **HRMS** (EI) calcd for $C_{10}H_{18}$ *m/z* 138.1408 $[M\text{-}C_2H_5FO]^+$, found 138.1403.



9d

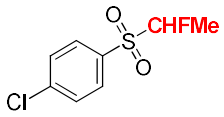
(3*S*,5*S*,8*R*,9*S*,10*S*,13*S*,14*S*)-3-(1-Fluoroethoxy)-10,13-dimethyltetradecahydro-1*H*-cyclopenta[α]phenanthren-17(2*H*)-one 9d. The product was purified by flash column chromatography on silica gel (height 12 cm, width 1.2 cm, pentane/acetone, gradient: 98:2 to 70:30) to yield **9d** as a 1:1 mixture of diastereoisomers as a colorless oil (22 mg, 33%). $\mathbf{R_f}$ (pentane/acetone 90:10): 0.33. **^{1}H NMR** ($CDCl_3$, 300.1 MHz): δ 6.33 (dq, *J* = 56.7, 5.1 Hz, 1H, major + minor dia), 4.70-4.65 (m, 1H, major + minor dia), 2.51-2.37 (m, 1H, major +

minor dia), 2.20-0.61 (m, 30H, major + minor dia). **^{13}C NMR** ($CDCl_3$, 75.5 MHz): δ 152.6 (major + minor dia), 104.0 (d, J = 222.7 Hz, major + minor dia), 78.4 (major + minor dia), 54.2 (major + minor dia), 51.3 (major + minor dia), 47.7 (major + minor dia), 44.6 (major + minor dia), 36.5 (major + minor dia), 35.8 (major + minor dia), 35.5 (major + minor dia), 35.0 (major + minor dia), 33.6 (major + minor dia), 31.5 (major + minor dia), 30.7 (major + minor dia), 28.2 (major dia), 28.2 (minor dia), 27.1 (major dia), 27.1 (minor dia), 21.7 (major + minor dia), 20.4 (major + minor dia), 19.6 (d, J = 24.2 Hz, major + minor dia), 13.8 (major + minor dia), 12.1 (major + minor dia). **^{19}F NMR** ($CDCl_3$, 282.4 MHz): δ -121.5 (dq, J = 56.5, 19.8 Hz, major dia), -121.5 (dq, J = 56.5, 19.8 Hz, minor dia). **IR** (cm^{-1}): 2934, 2856, 1759, 1737, 1265, 1251, 1096, 910, 730. **HRMS** (EI) calcd for $C_{21}H_{33}FO_2$ *m/z* 336.2464 $[M]^+$, found 336.2132.



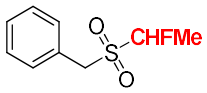
11

5-(Ethoxycarbonyl)-1-((fluoroethyl)thio)benzene 11. The product was purified by flash column chromatography on silica gel (height 13 cm, width 1.2 cm, pentane/acetone, gradient: 98:2 to 96:4) to yield **11** as a yellow oil (41 mg, 90%). **H NMR** ($CDCl_3$, 300.1 MHz): δ 7.98 (d, J = 8.4 Hz, 2H), 7.50 (d, J = 8.4 Hz, 2H), 6.07 (dq, J = 55.8, 6.6 Hz, 1H), 4.36 (q, J = 7.2 Hz, 2H), 1.73 (dd, J = 20.1, 6.3 Hz, 3H), 1.38 (t, J = 6.9 Hz, 3H). **^{13}C NMR** ($CDCl_3$, 75.5 MHz): δ 166.0, 139.6 (d, J = 2.3 Hz), 130.0, 130.0 (d, J = 2.3 Hz), 129.2, 96.8 (d, J = 215.9 Hz), 61.0, 21.5 (d, J = 24.2 Hz), 14.3. **^{19}F NMR** ($CDCl_3$, 282.4 MHz): δ -140.7 (dq, J = 56.5, 19.8 Hz, 1F). **IR** (cm^{-1}): 2963, 1714, 1596, 1261, 1104, 1068, 1017, 852, 786, 760, 734, 688. **HRMS** (EI) calcd for $C_{11}H_{13}FO_2S$ *m/z* 228.0620 $[M]^+$, found 228.0623.



12a

1-Chloro-4-((1-fluoroethyl)sulfonyl)benzene 12a. The product was purified by flash column chromatography on silica gel (height 13 cm, width 1.2 cm, petroleum ether/ethyl acetate, gradient: 90:10 to 60:40) to yield **12a** as a yellow oil (41 mg, 92%). **R_f** (petroleum ether/diethyl ether 70:30): 0.31. **^{1}H NMR** ($CDCl_3$, 300.1 MHz): δ 7.87 (d, J = 8.4 Hz, 2H), 7.58 (d, J = 8.7 Hz, 2H), 5.27 (dq, J = 48.0, 6.3 Hz, 1H), 1.70 (dd, J = 23.4, 6.6 Hz, 3H). **^{13}C NMR** ($CDCl_3$, 75.5 MHz): δ 141.6, 133.2, 131.1 (d, J = 0.8 Hz), 129.7, 99.8 (d, J = 216.7 Hz), 13.6 (d, J = 20.4 Hz). **^{19}F NMR** ($CDCl_3$, 282.4 MHz): δ -172.6 (dq, J = 48.0, 22.6 Hz, 1F). **IR** (cm^{-1}): 3094, 2942, 1326, 1153, 1080, 1013, 763, 719, 613, 585. **HRMS** (EI) calcd for $C_8H_8ClFO_2S$ *m/z* 221.9917 $[M]^+$, found 221.9922.



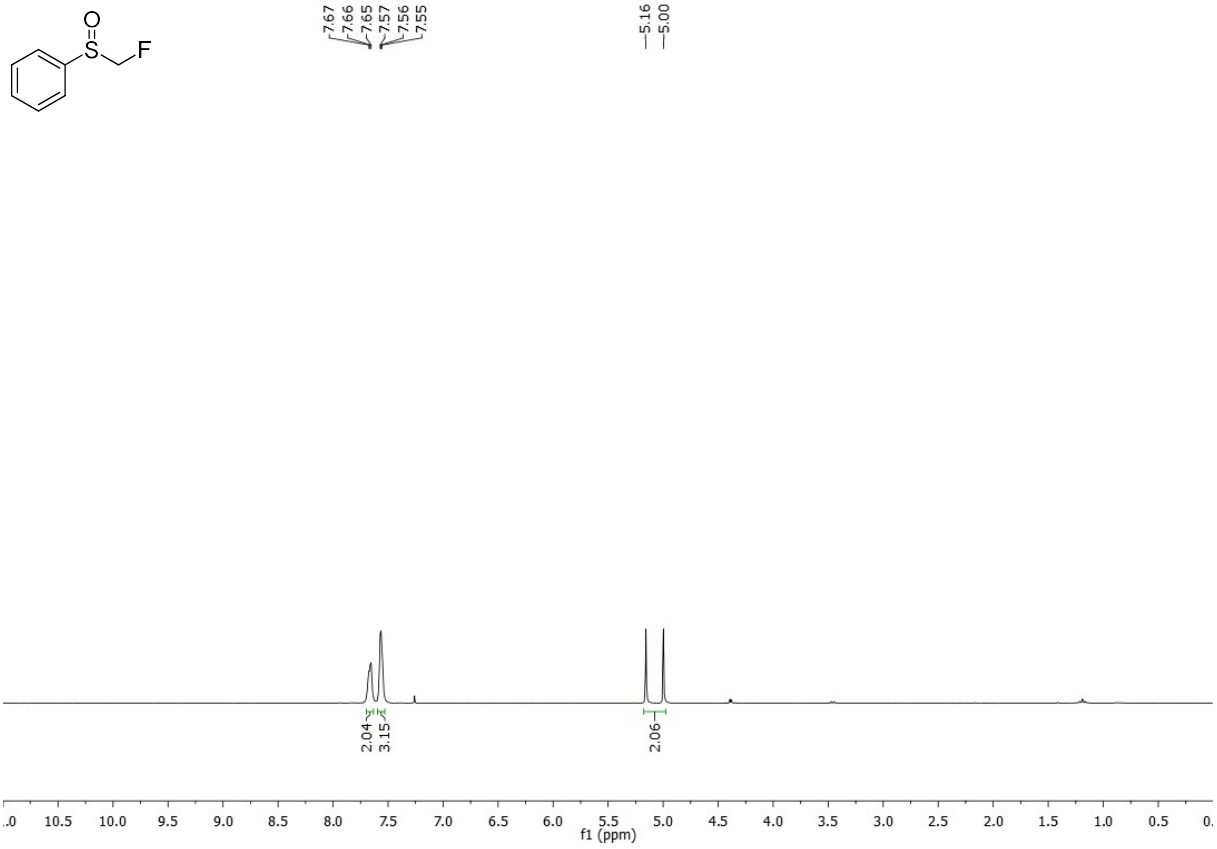
12b

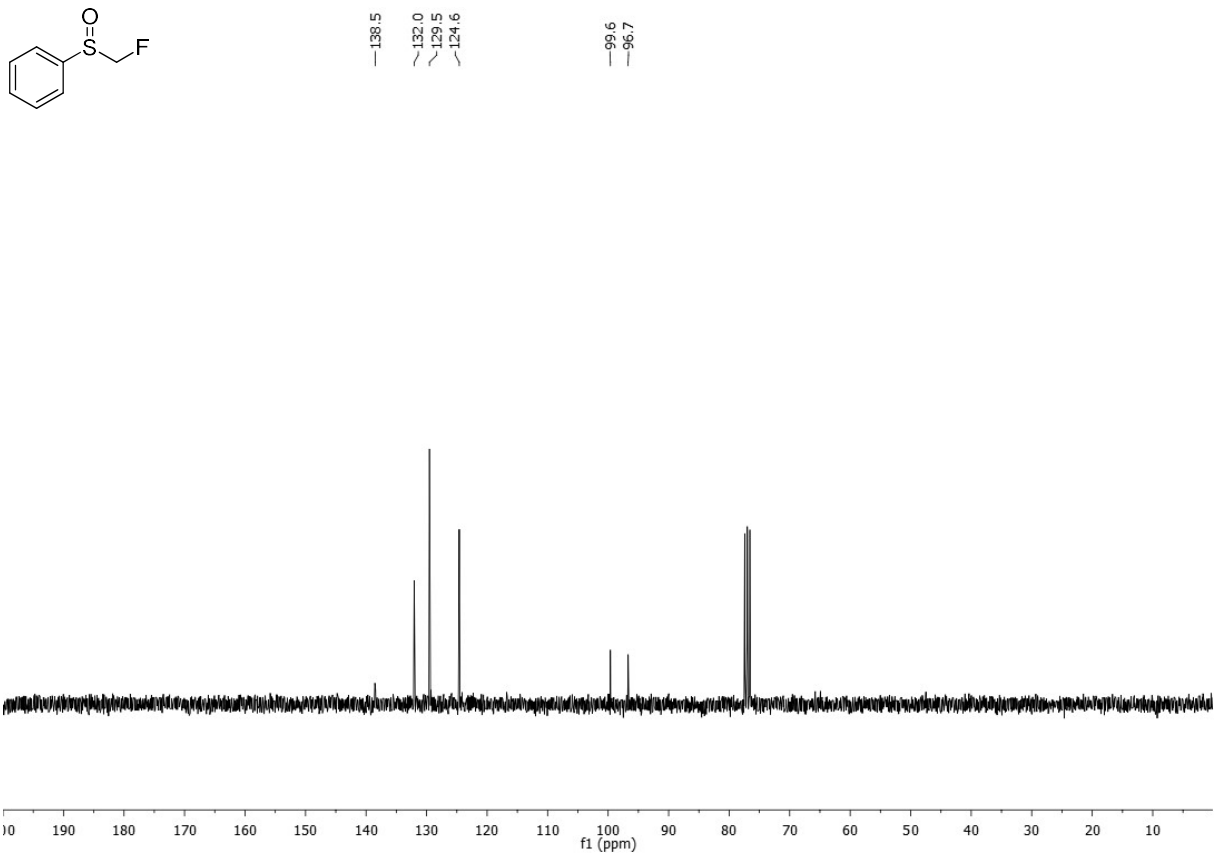
(((1-Fluoroethyl)sulfonyl)methyl)benzene 12b. The product was purified by flash column chromatography on silica gel (height 13 cm, width 1.2 cm, petroleum ether/diethyl ether, gradient: 90:10 to 60:40) to yield **12b** as a colorless oil (29 mg, 72%). $\mathbf{R_f}$ (petroleum ether/diethyl ether 70:30): 0.38. **^{1}H NMR** ($CDCl_3$, 300.1 MHz): δ 7.42 (s, 5H), 5.16 (dq, *J* = 49.2, 6.6 Hz, 1H), 4.50-4.41 (m, 1H), 4.32-4.20 (m, 1H), 1.66 (dd, *J* = 24.0, 6.3 Hz, 3H). **^{13}C NMR** ($CDCl_3$, 75.5 MHz): δ 130.8, 129.2, 129.1, 126.6, 95.5 (d, *J* = 215.2 Hz), 55.8, 11.5 (d, *J* = 20.4 Hz). **^{19}F NMR** ($CDCl_3$, 282.4 MHz): δ -169.6 (m, 1F). **IR** (cm^{-1}): 3621, 3008, 2947, 2294, 2254, 1630, 1143, 1376, 1039, 919, 750. **HRMS** (EI) calcd for $C_9H_{11}FO_2S$ *m/z* 202.0463 $[M]^+$, found 202.0466.

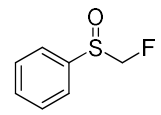
8. References

(1) J. Cheng and W. R. Roush, *Org. Synth.,* 2002, **78**, 169.
(2) T. D. Montgomery and V. H. Rawal, *Org. Synth.,* 2013, **90***,* 130.
(3) W. Zhang, L. Zhu and J. Hu, *Tet. Lett.,* 2007, **63**, 10569.
(4) T. Umemoto and G. Tomizawa, *Bull. Chem. Soc. Jpn.*, 1986, **59**, 3625
(5) G. K. S. Prakash, I. Ledneczki, S. Chacko and G. A. Olah, *Org. Lett.,* 2008, **10**, 557.

9. NMR spectra







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f1 (ppm)


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7.63
7.62
7.61
7.61
7.61
7.60
7.54
7.53
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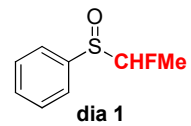
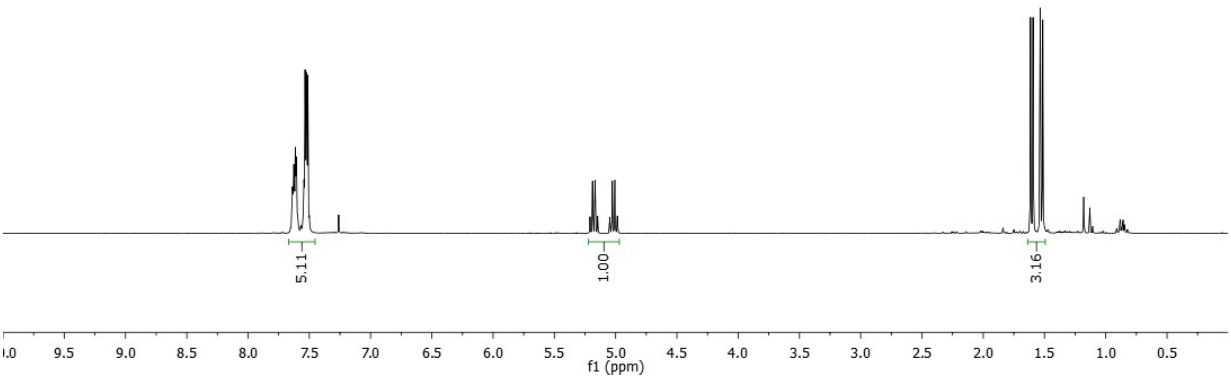
1.51


CHFMe

dia 1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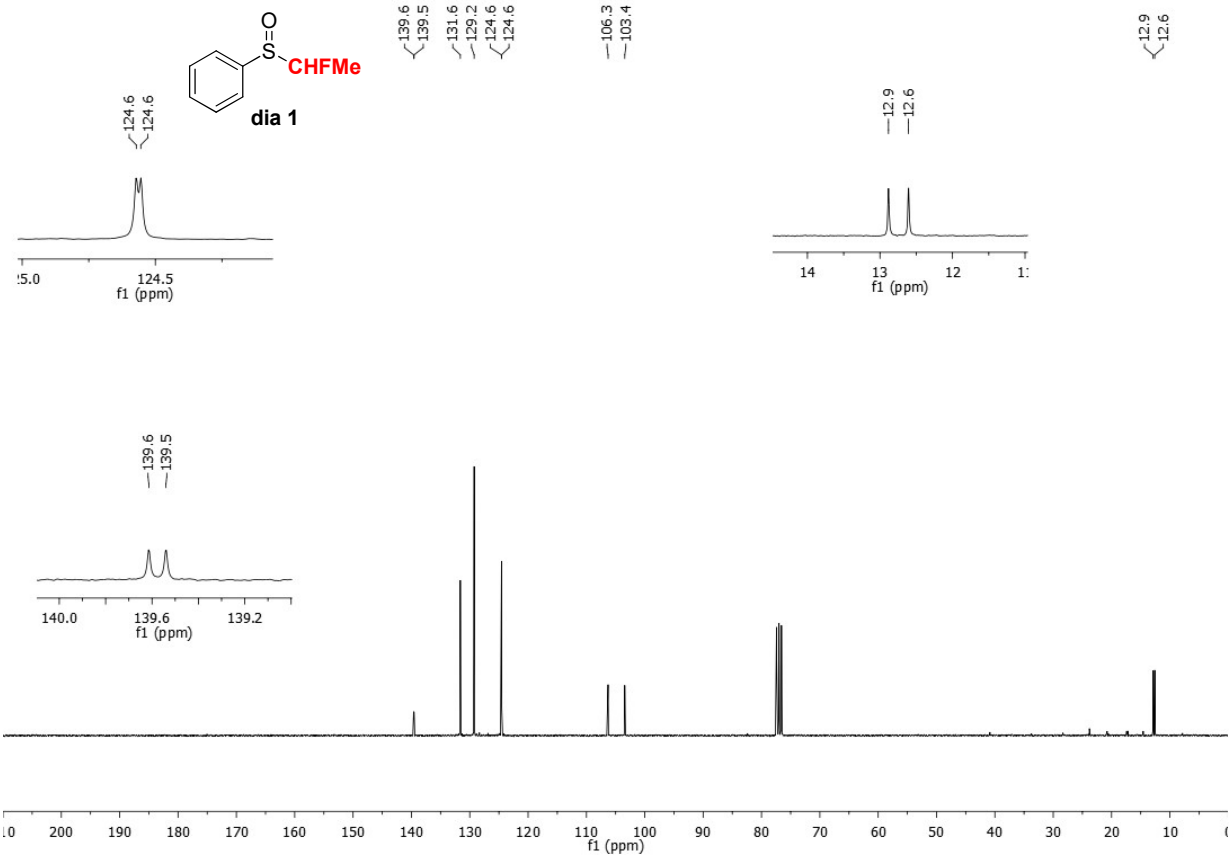
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CHFMe
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124.6
124.6
124.5
f1 (ppm)
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12
f1 (ppm)
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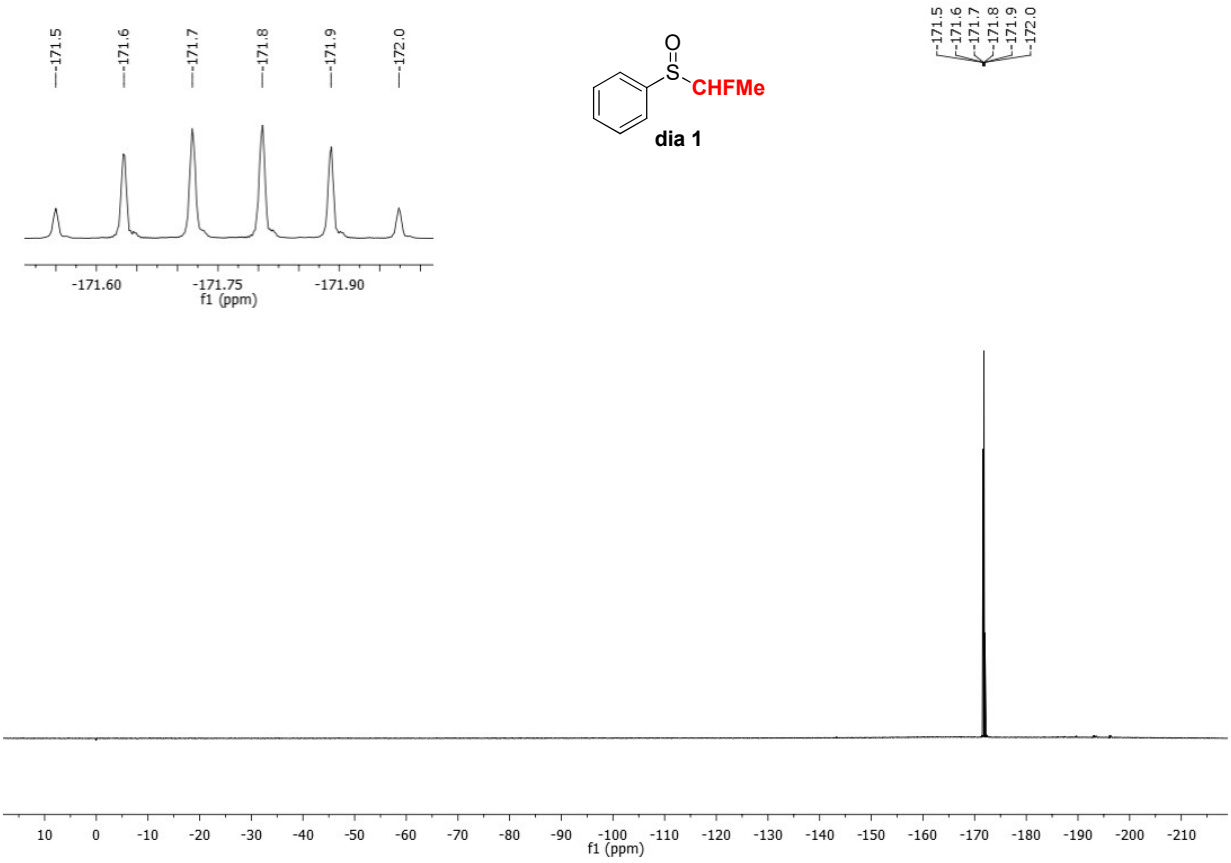
f1 (ppm)
f1 (ppm)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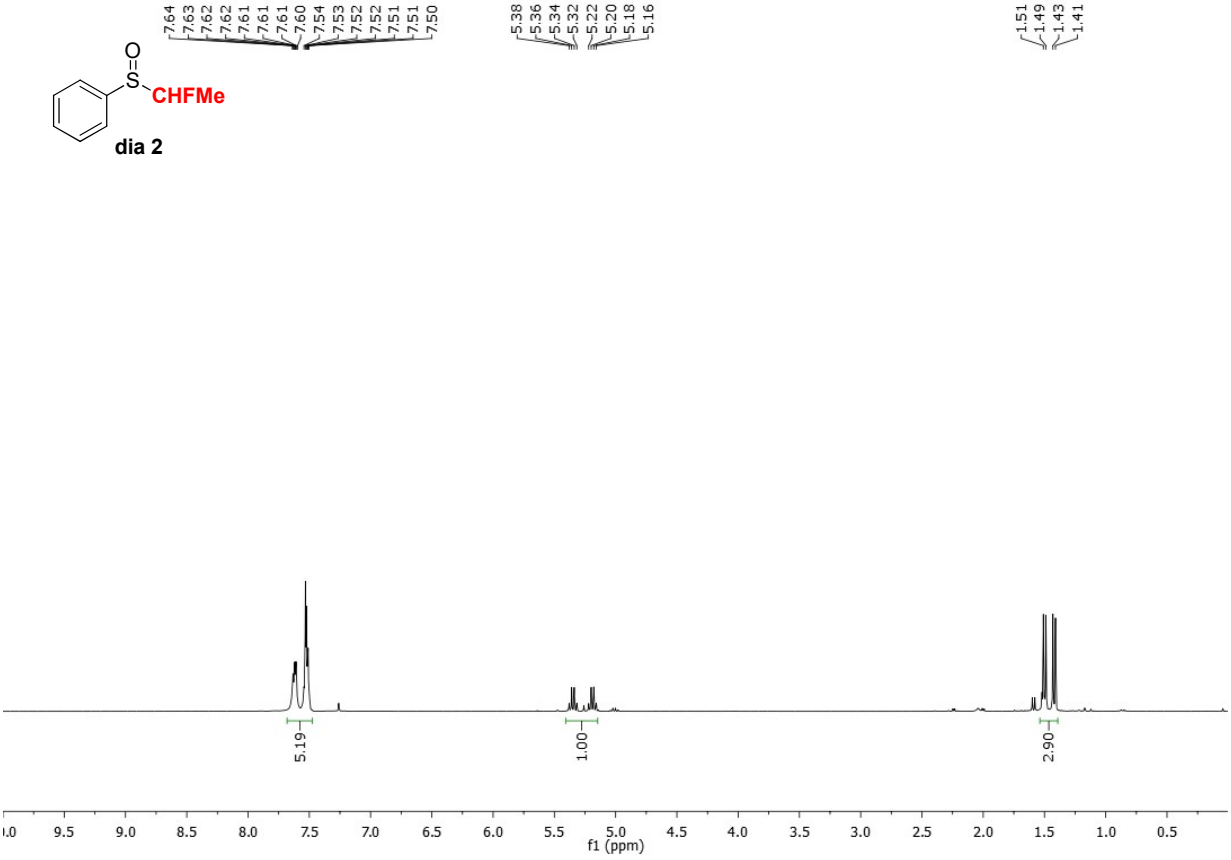
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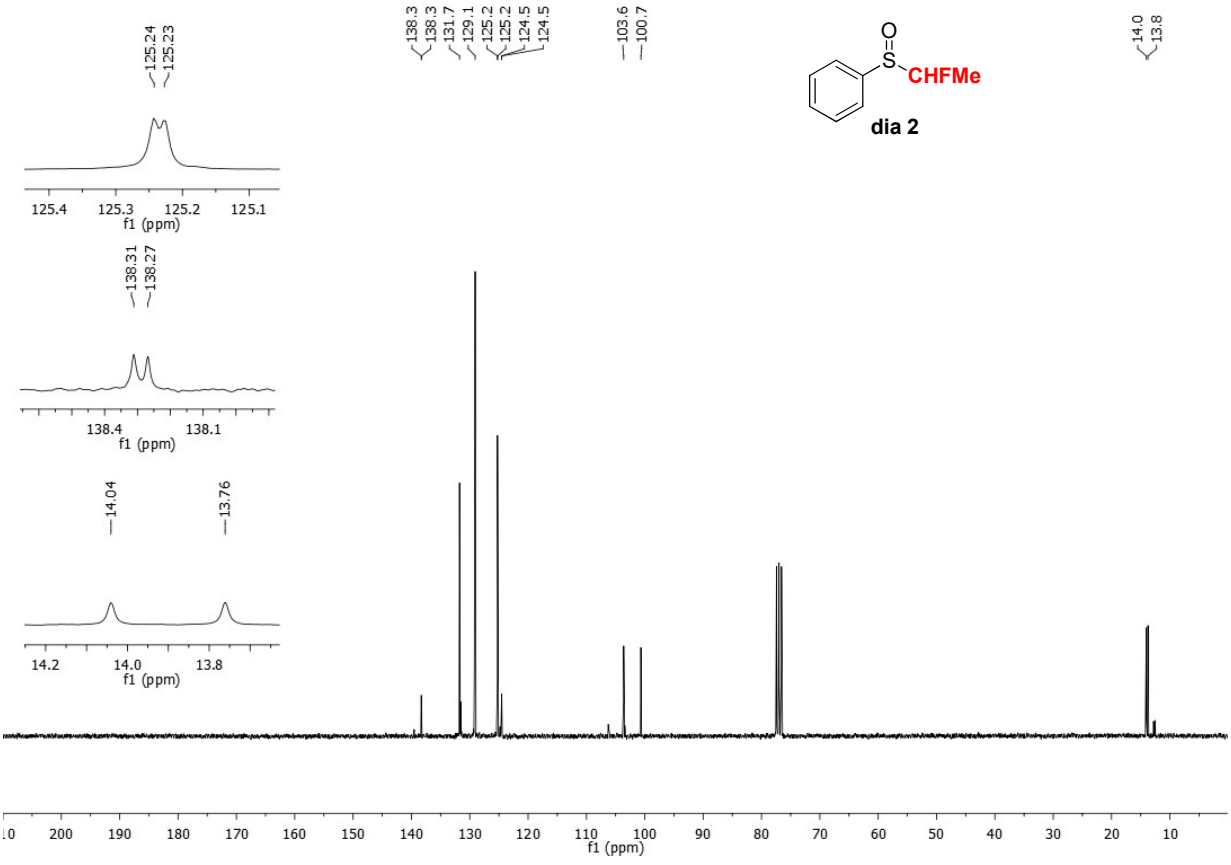
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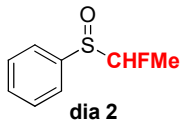
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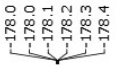
dia 2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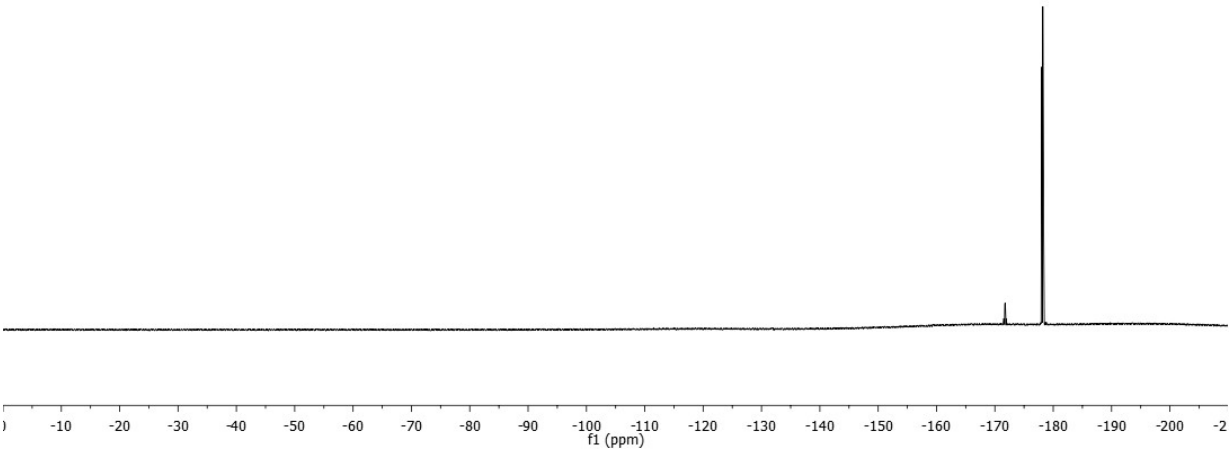
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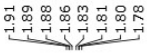
3.93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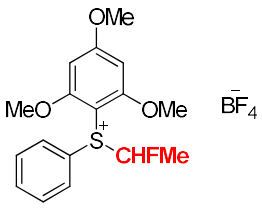
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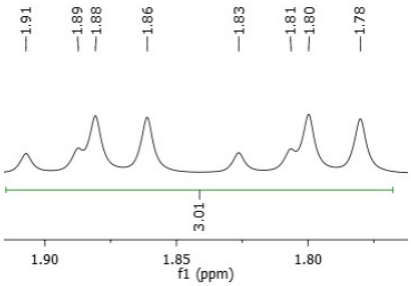
1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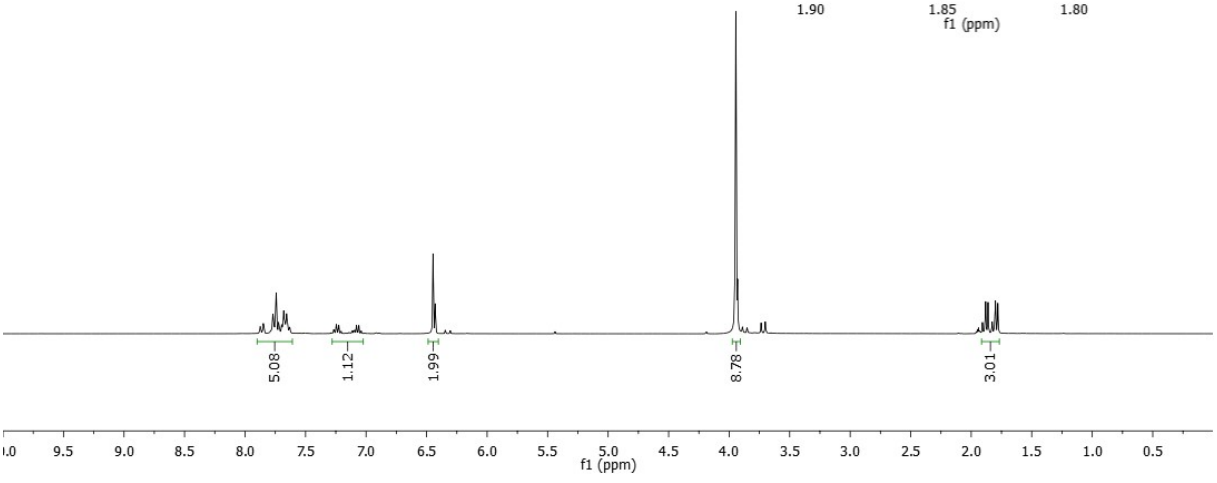
f1 (ppm)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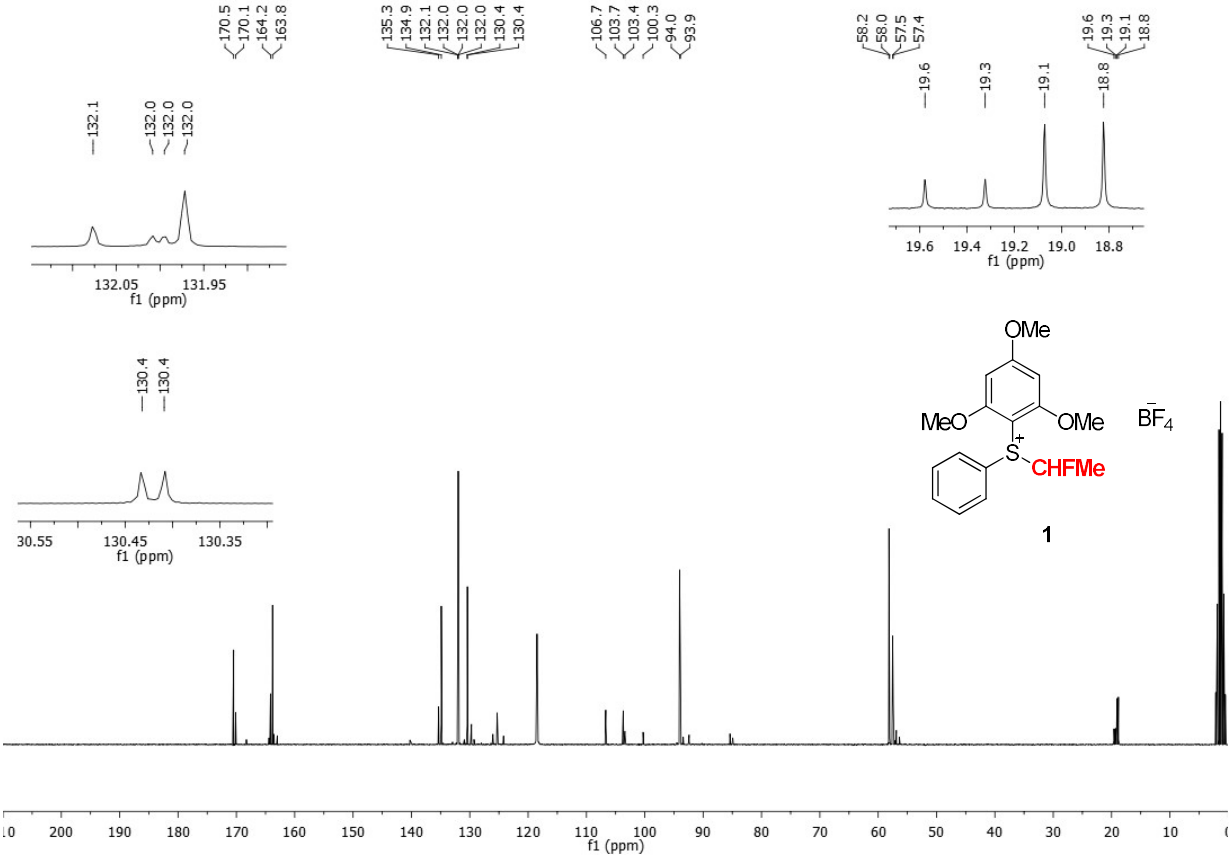
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58.0
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f1 (ppm)
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OMe
S
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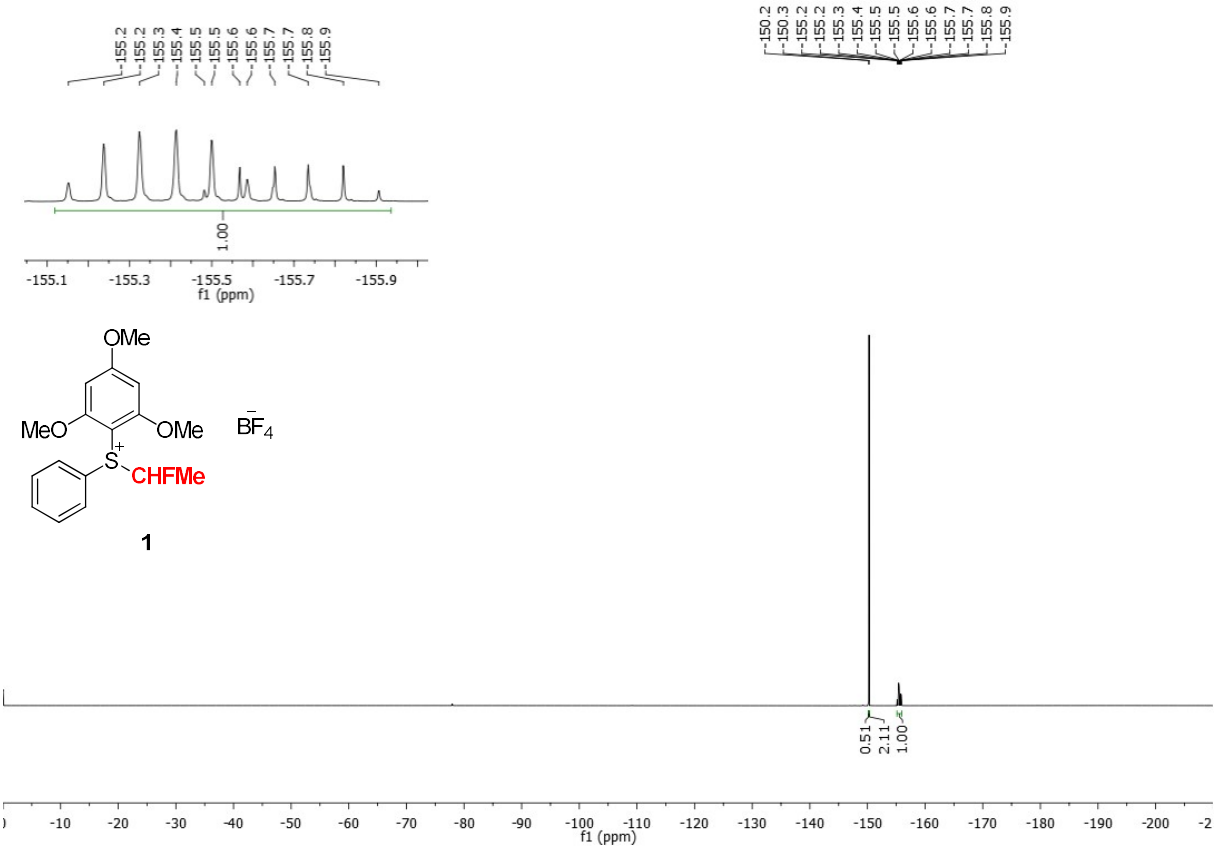
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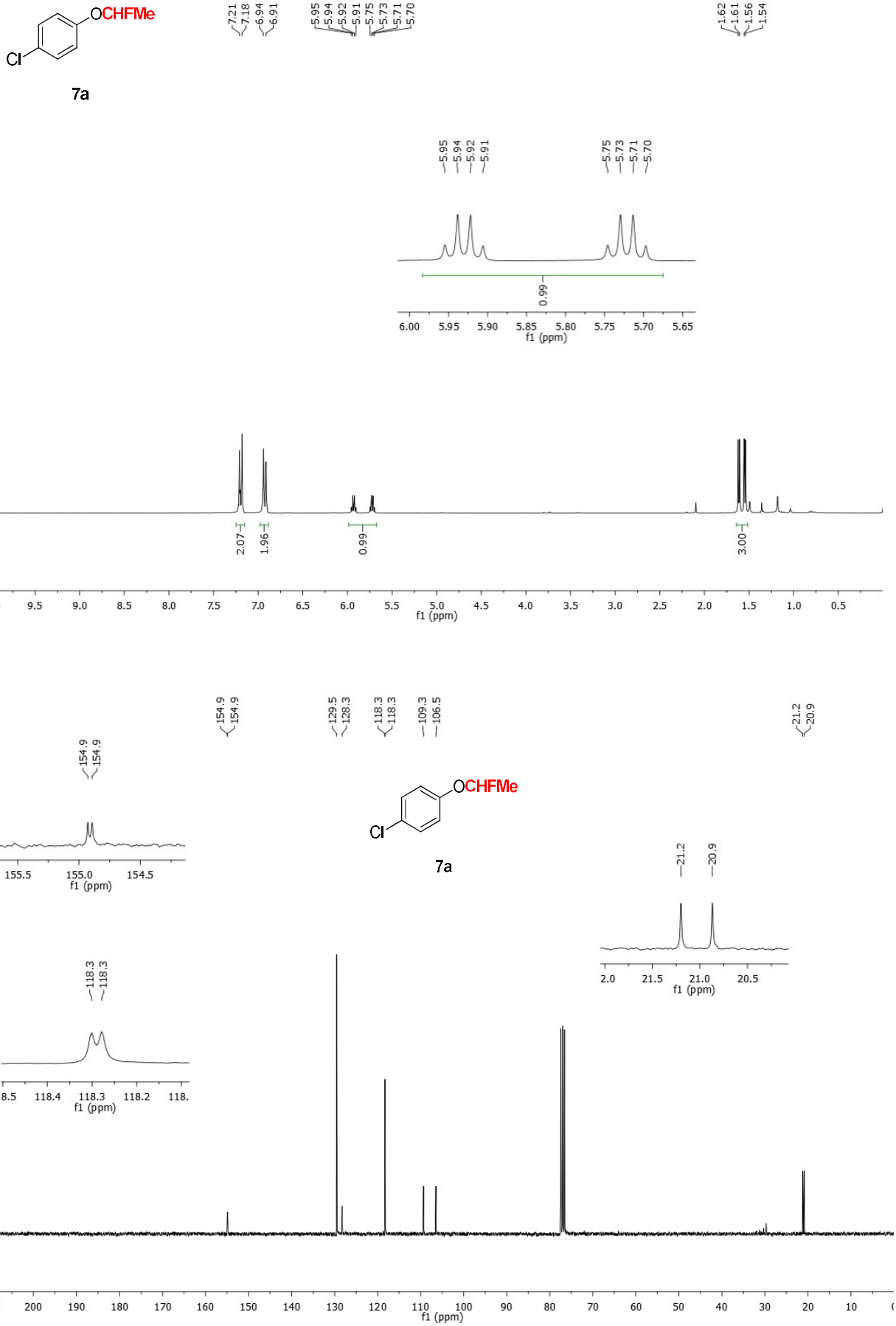
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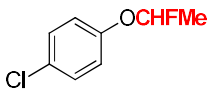
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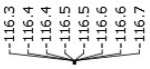
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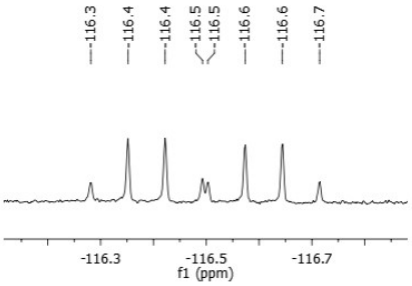
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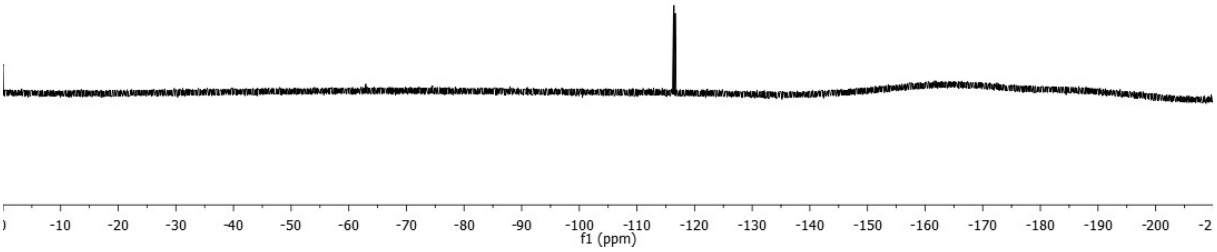
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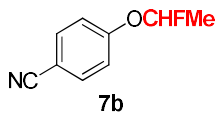
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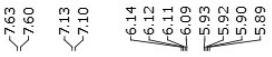
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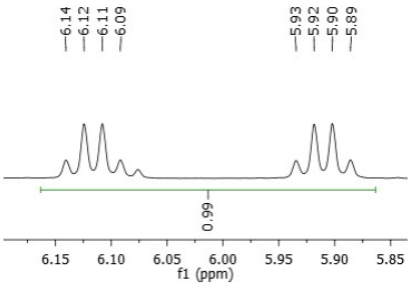
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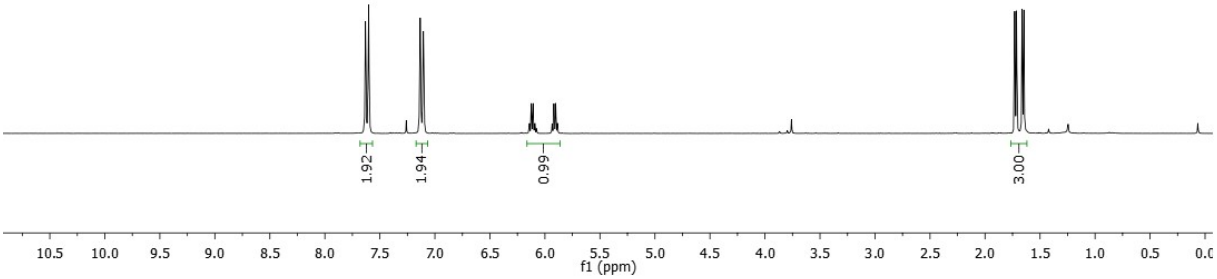
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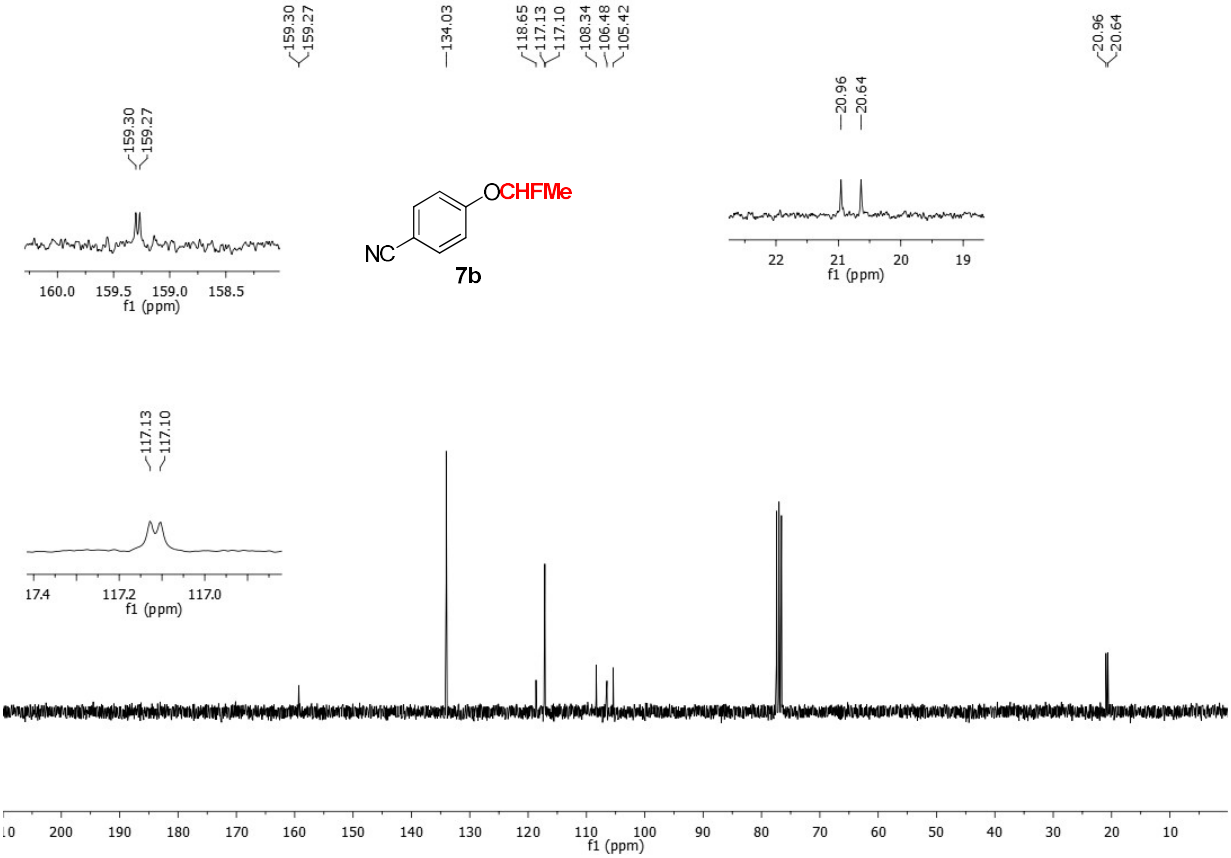
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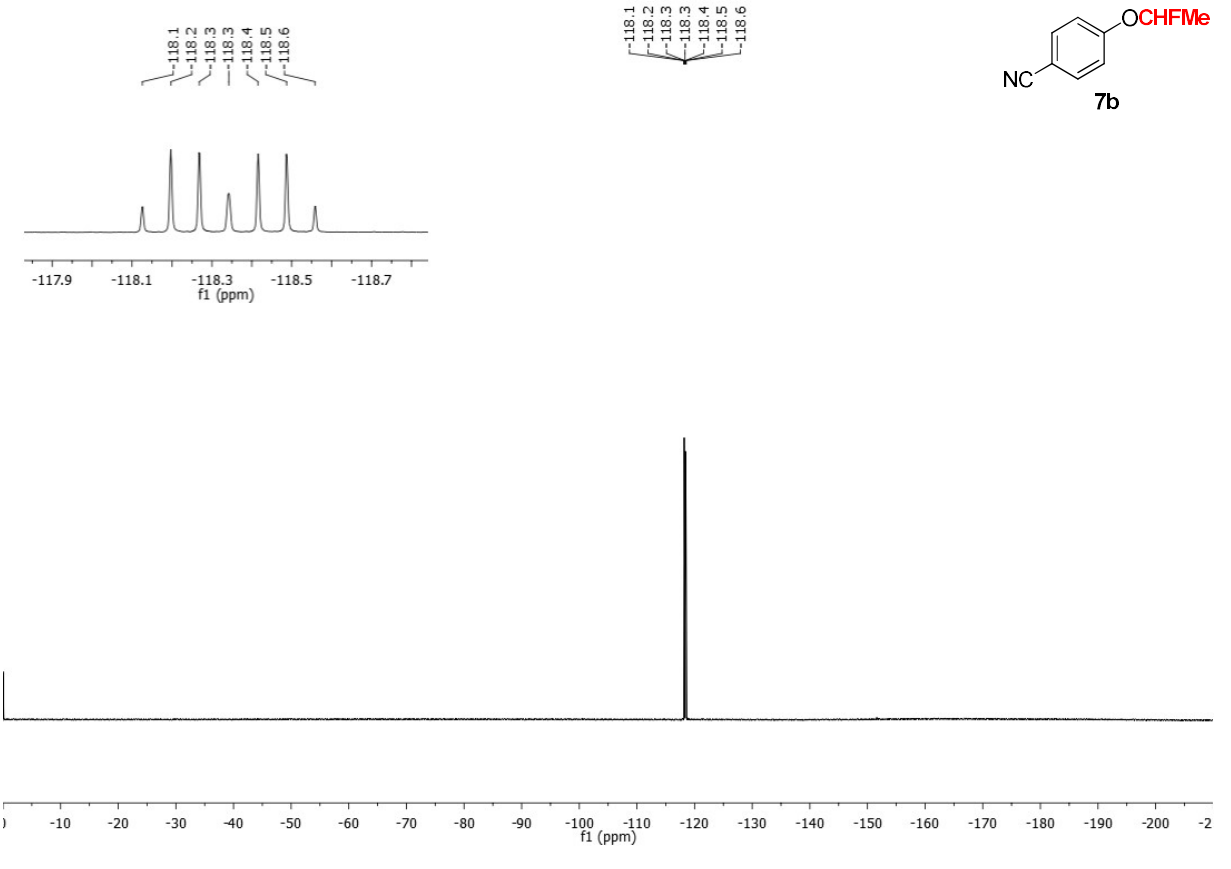
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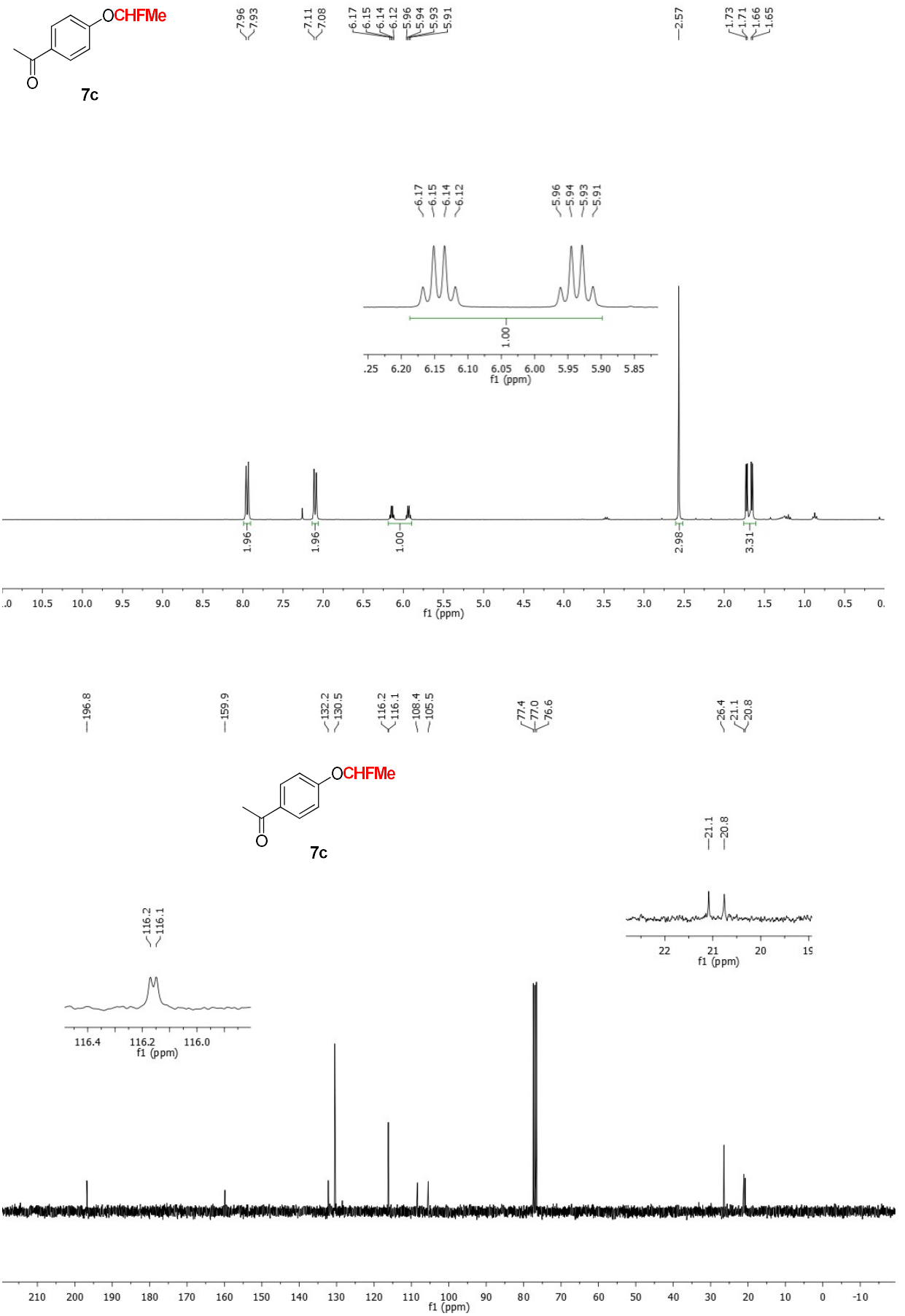
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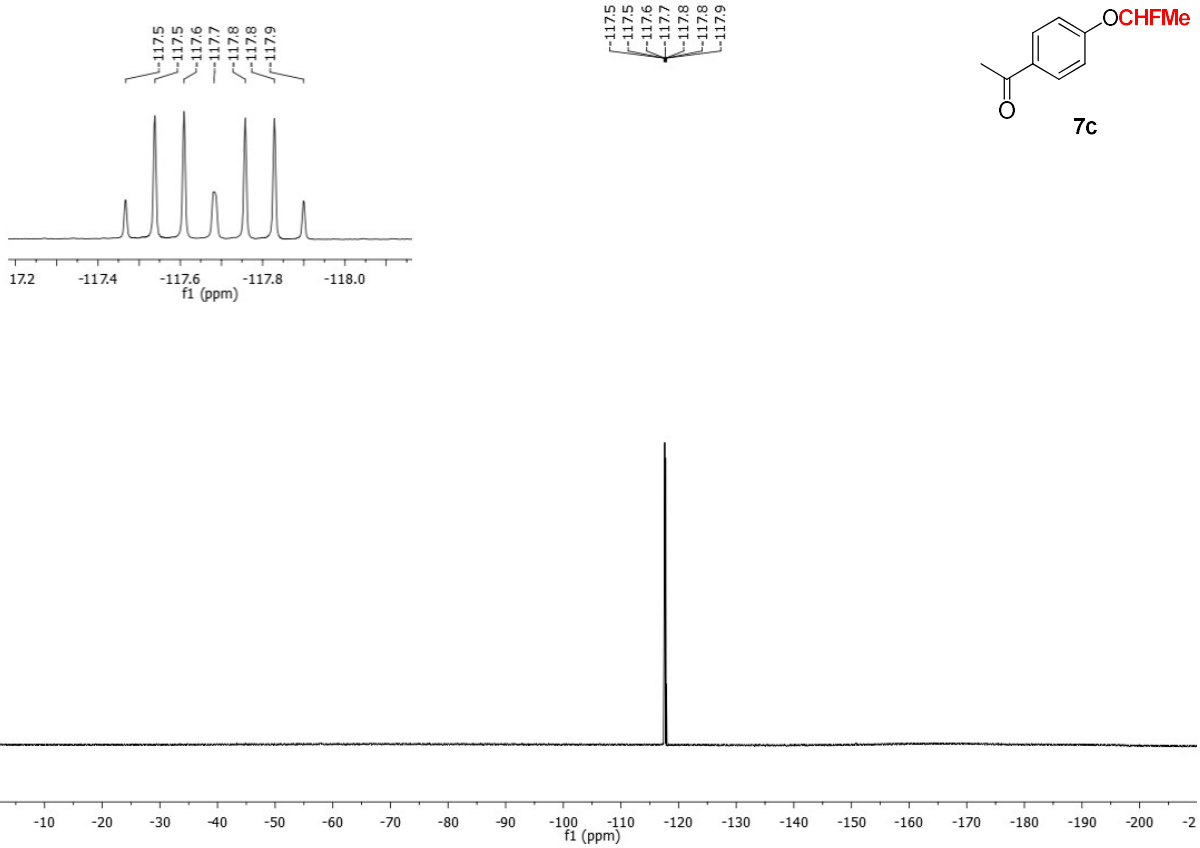
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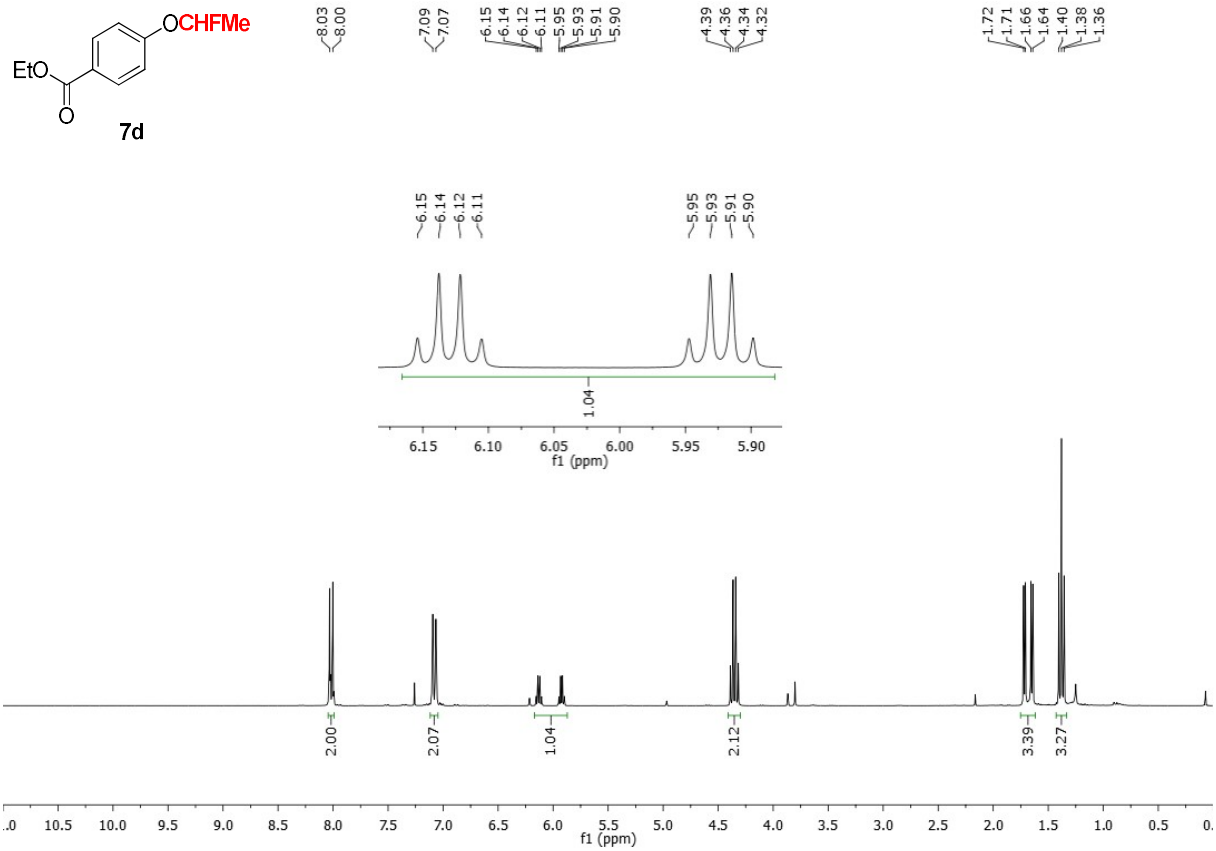
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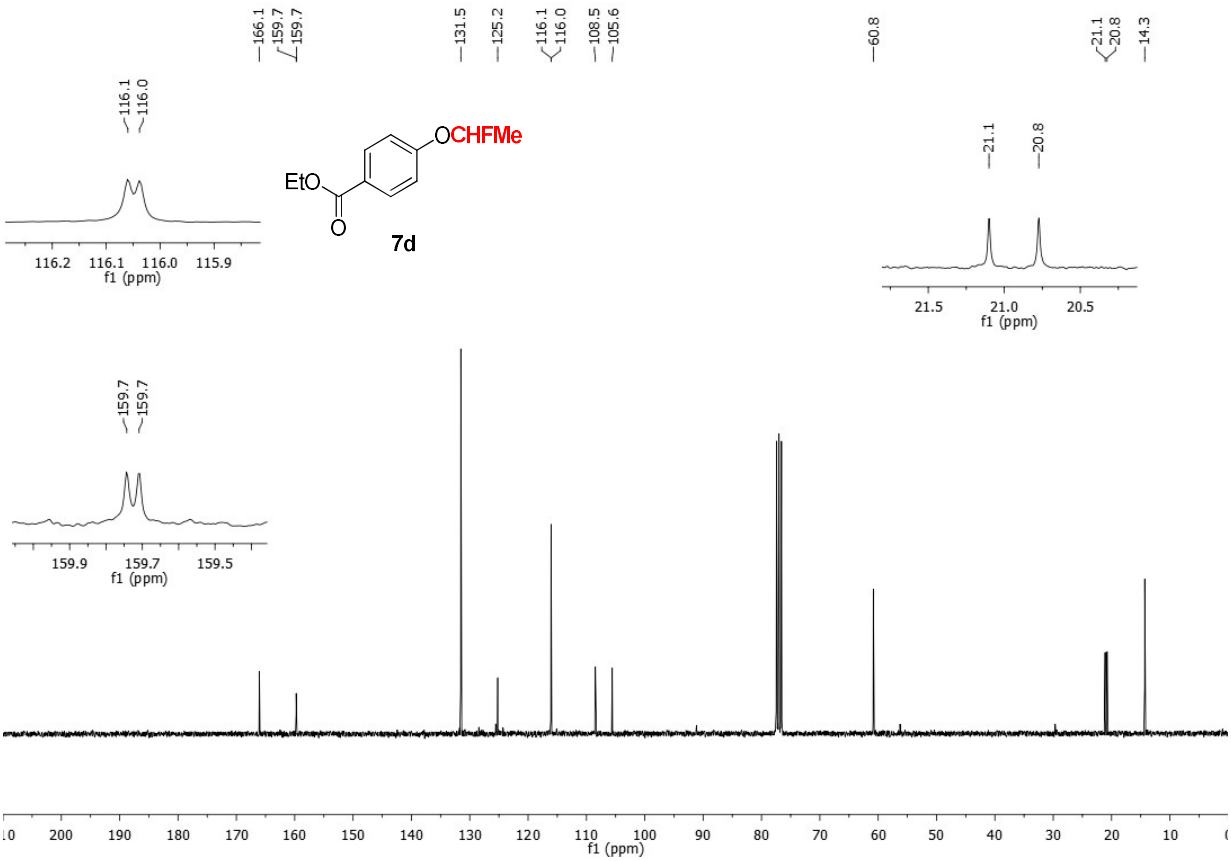
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60.8
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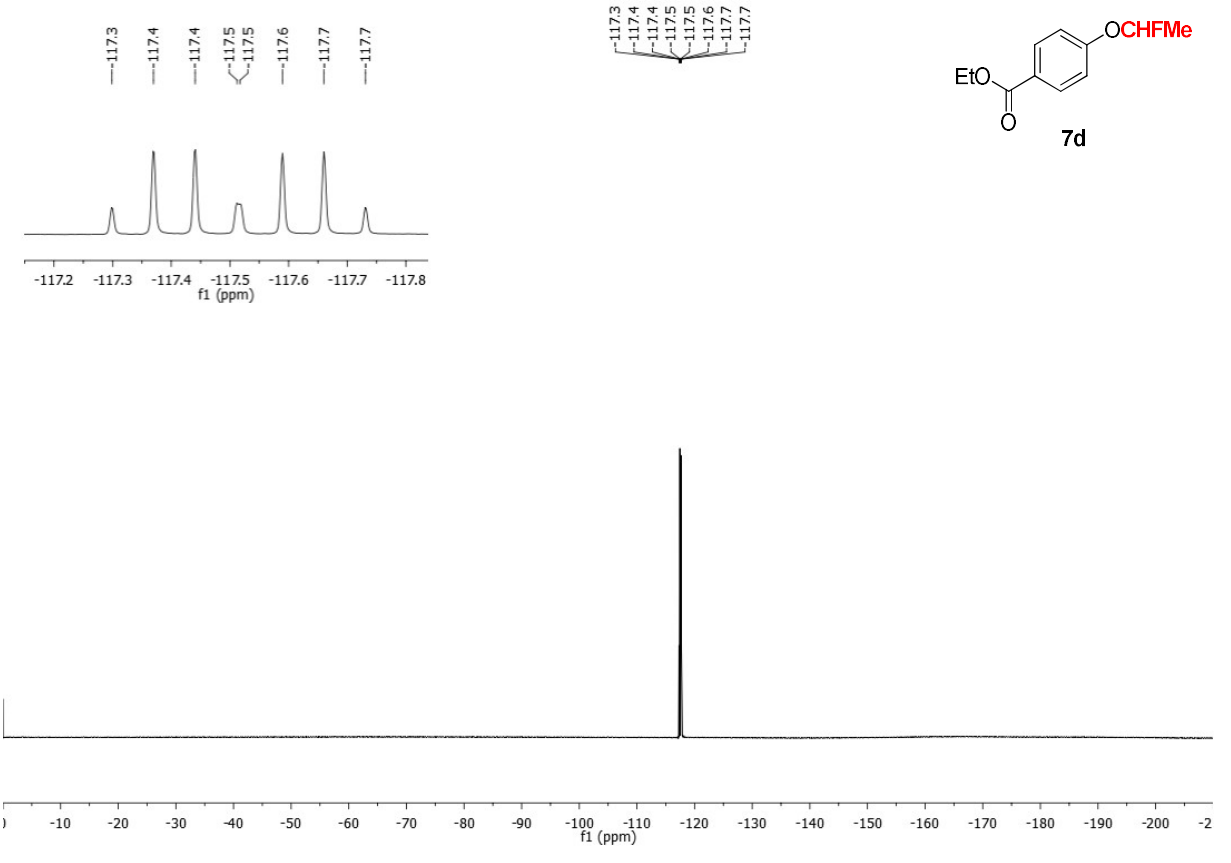
20.8
14.3
116.1
116.0
116.2 116.1 116.0 115.9
f1 (ppm)
OCHFMe
EtO
O
7d
21.1
20.8
21.5 21.0 20.5
f1 (ppm)
159.7
159.7
159.9 159.7 159.5
f1 (ppm)
f1 (ppm)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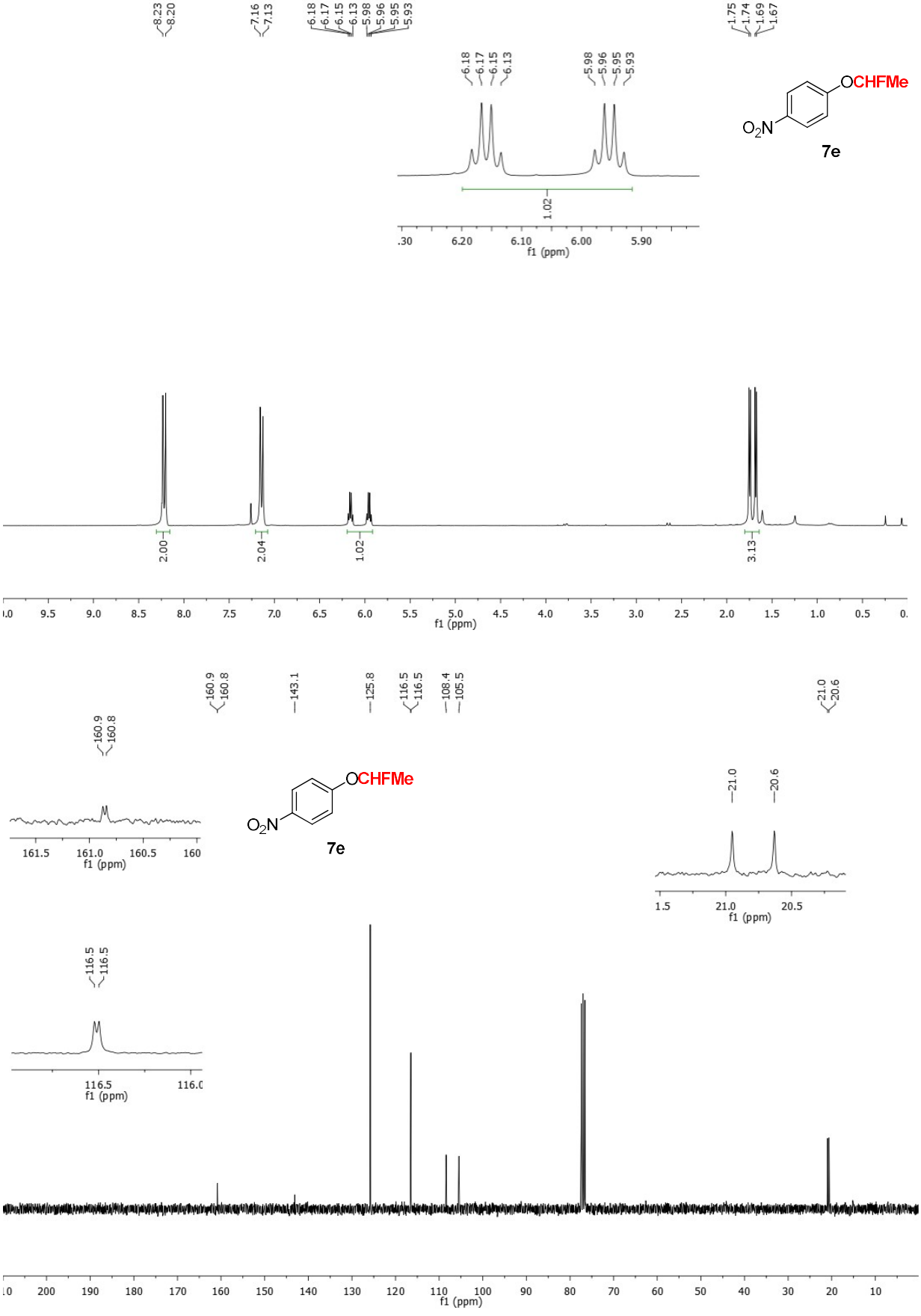
117.4
117.4
117.5
117.5
117.6
117.7
117.7
-117.2 -117.3 -117.4 -117.5 -117.6 -117.7 -117.8
f1 (ppm)
117.3
117.4
117.4
117.5
117.5
117.6
117.7
117.7
OCHFMe
EtO
O
7d
f1 (ppm)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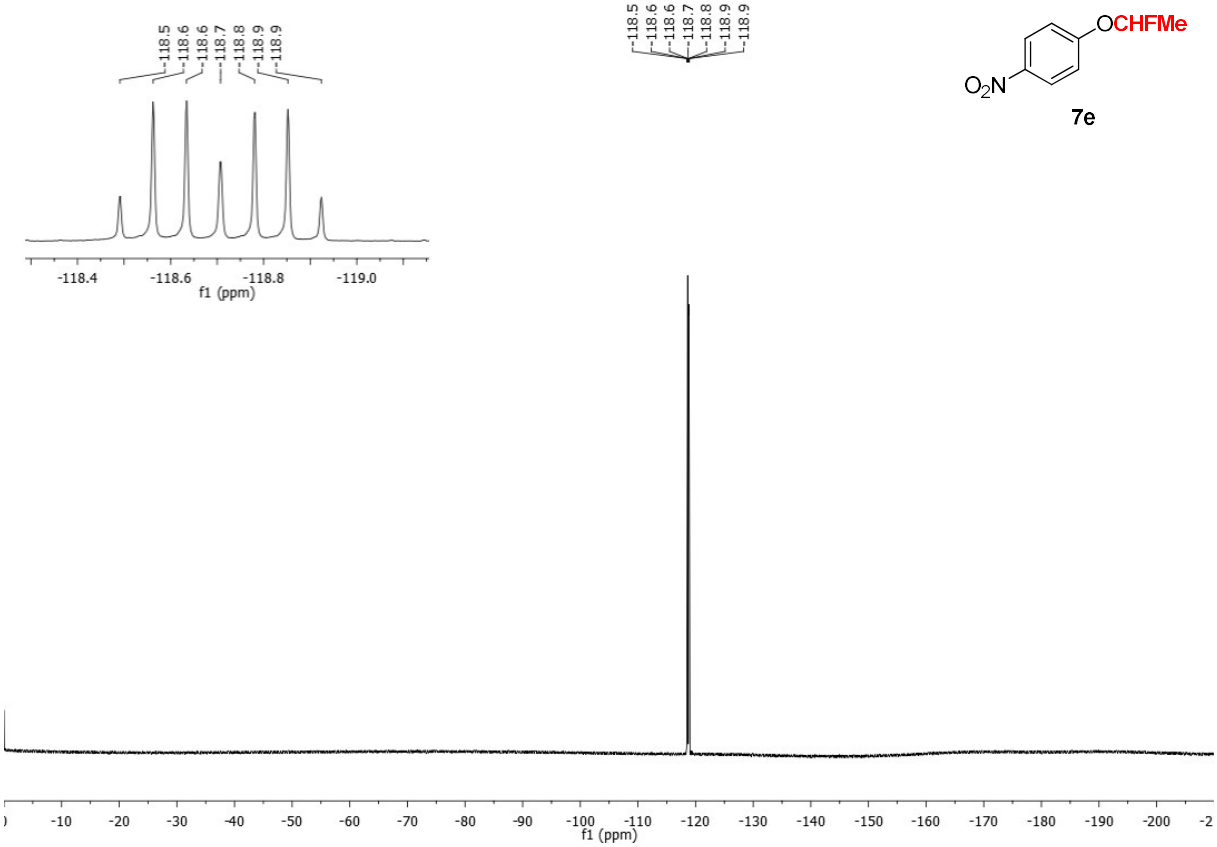
7.13
6.18
6.17
6.15
6.13
5.98
5.96
5.95
5.93
1.75
1.74
1.69
1.67
OCHFMe
O2N
7e
1.02
f1 (ppm)
2.00
2.04
1.02
3.13
160.9
160.8
143.1
125.8
116.5
116.5
108.4
105.5
21.0
20.6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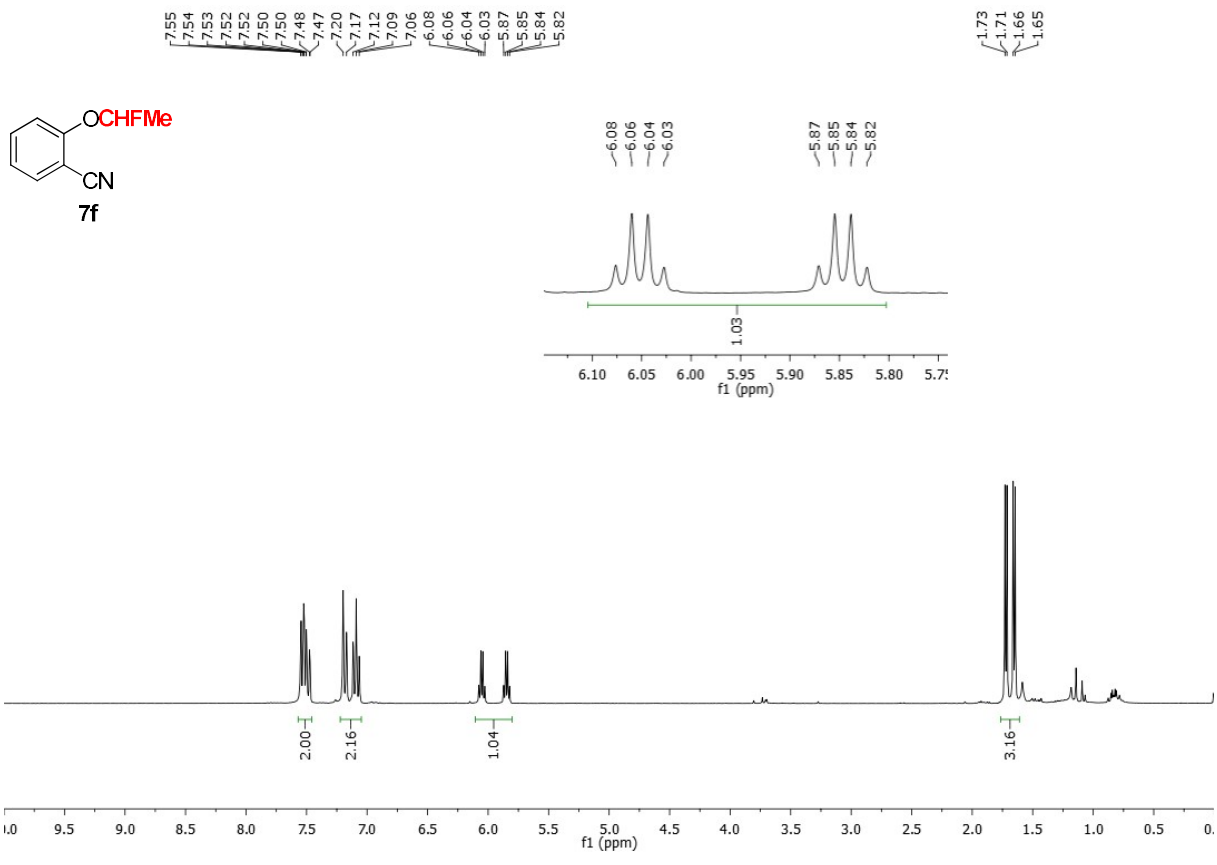
-118.6
-118.6
-118.7
-118.8
-118.9
-118.9
-118.4
-118.6
-118.8
-119.0
f1 (ppm)
OCHFMe
O2N
7e
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
f1 (ppm)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7.53
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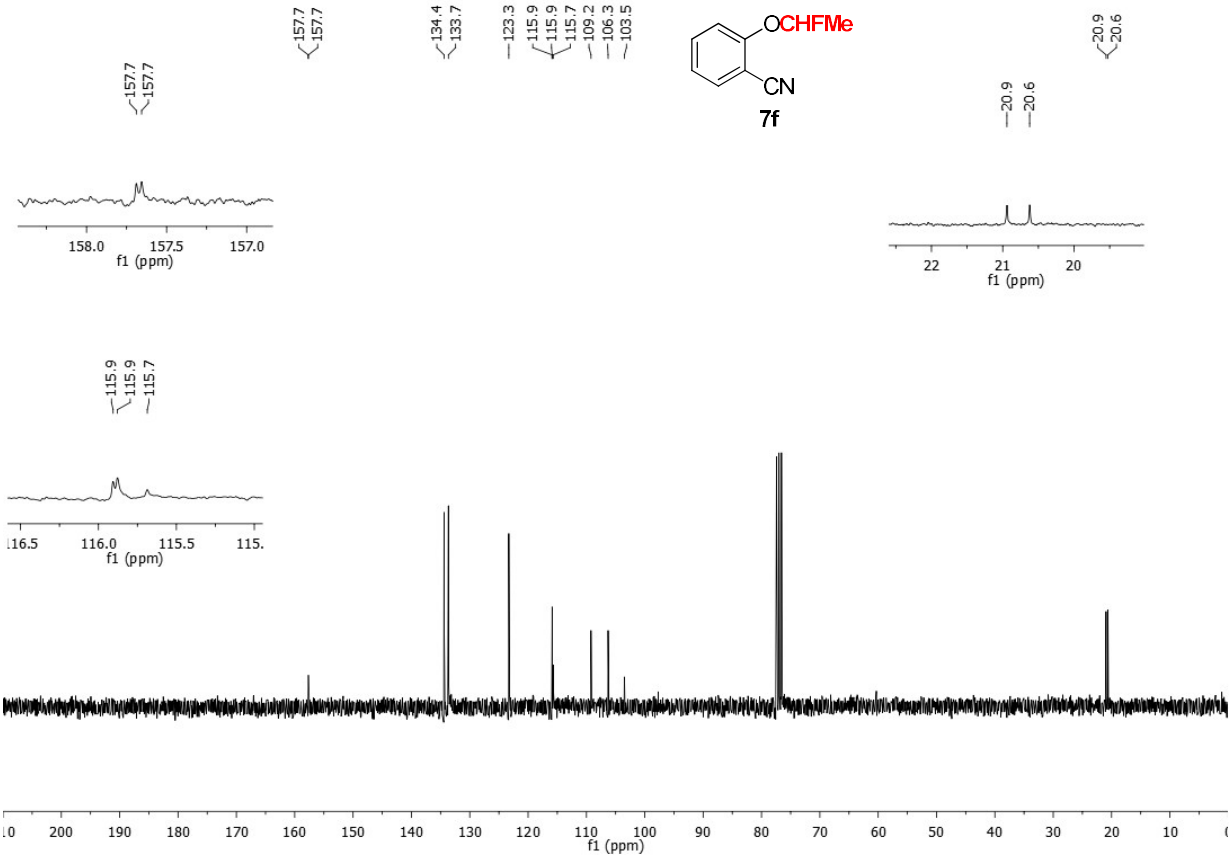
7.48
7.47
7.20
7.17
7.12
7.09
7.06
6.08
6.06
6.04
6.03
5.87
5.85
5.84
5.82
1.73
1.71
1.66
1.65
OCHFMe
CN
7f
1.03
6.10
6.05
6.00
5.95
5.90
5.85
5.80
f1 (ppm)
2.00
2.16
1.04
3.16
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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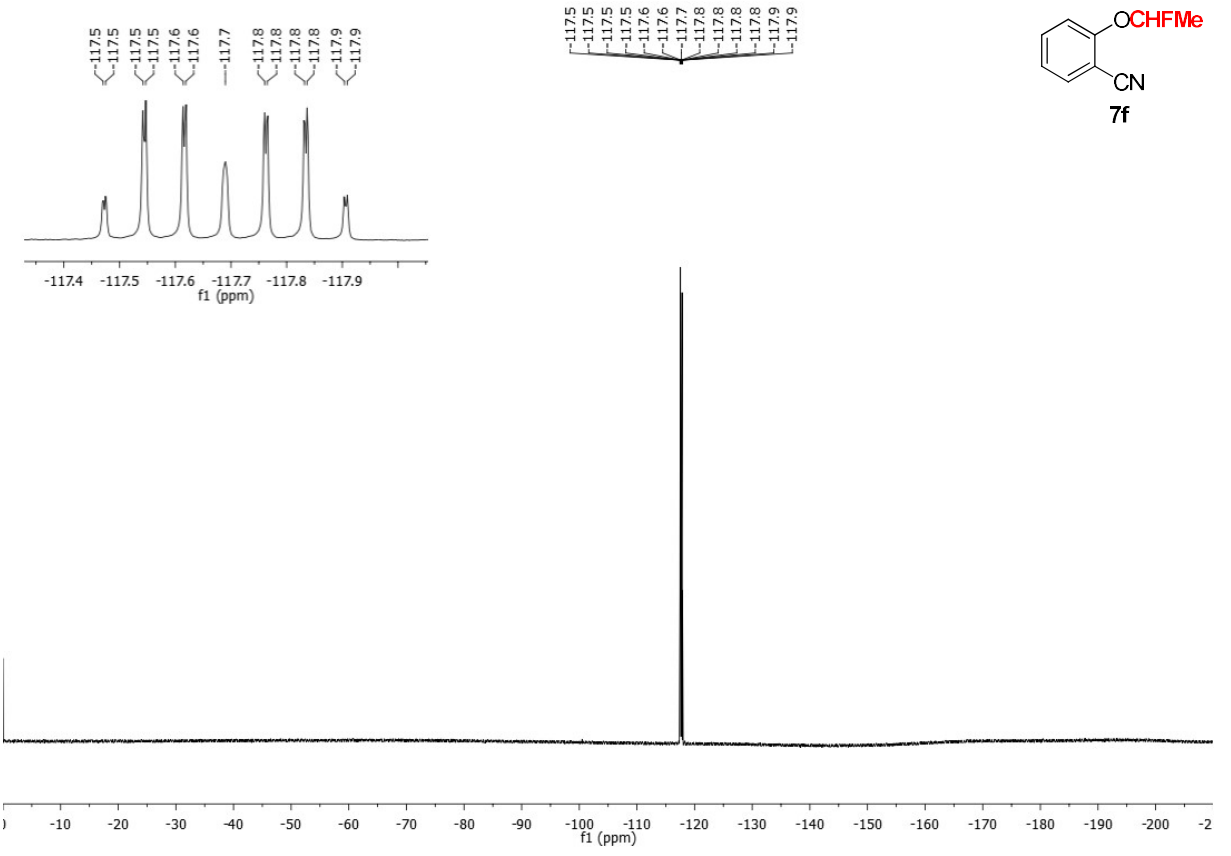
157.7
134.4
133.7
123.3
115.9
115.9
115.7
109.2
106.3
103.5
OCHFMe
CN
7f
20.9
20.6
158.0
157.5
157.0
f1 (ppm)
22
21
20
f1 (ppm)
115.9
115.9
115.7
116.5
116.0
115.5
115.
f1 (ppm)
f1 (ppm)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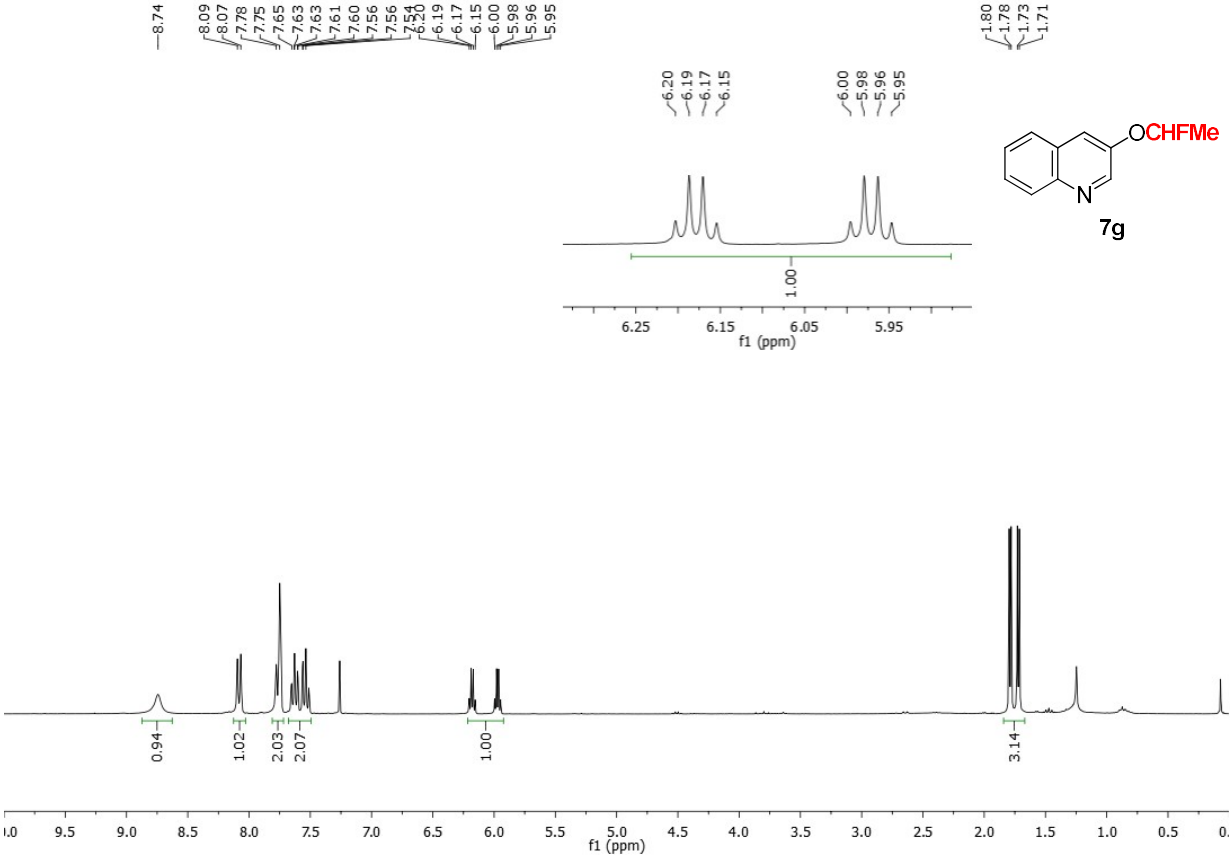
117.5
117.5
117.5
117.6
117.6
117.7
117.8
117.8
117.8
117.9
117.9
-117.4
-117.5
-117.6
-117.7
-117.8
-117.9
f1 (ppm)
OCHFMe
CN
7f
f1 (ppm)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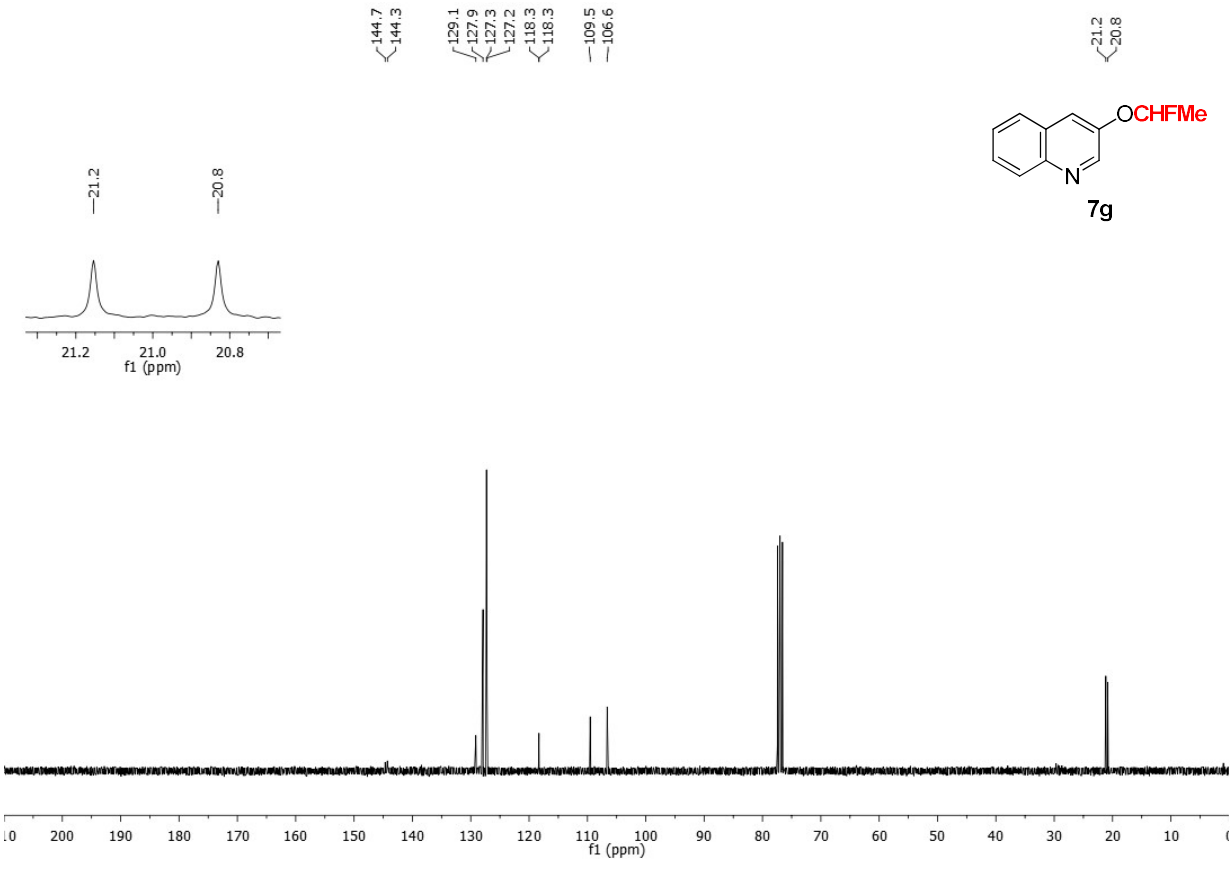
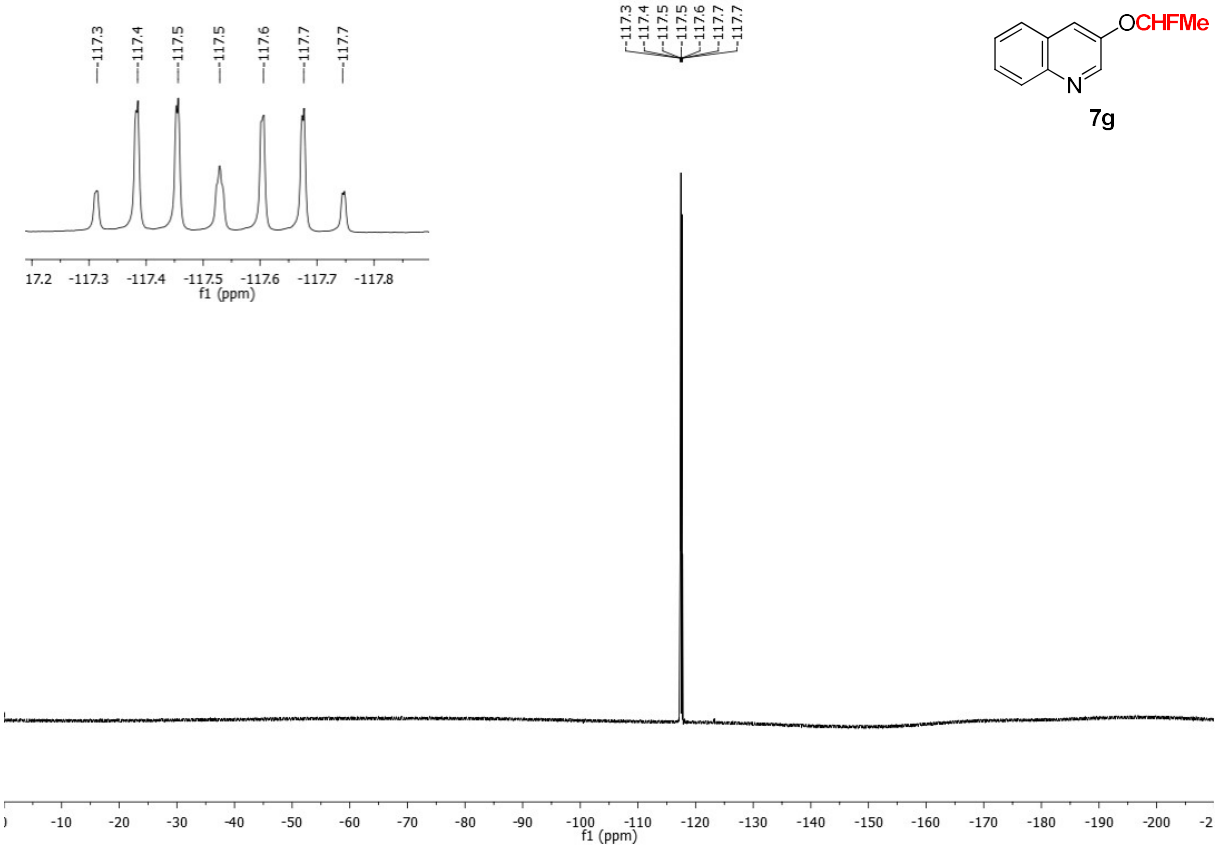
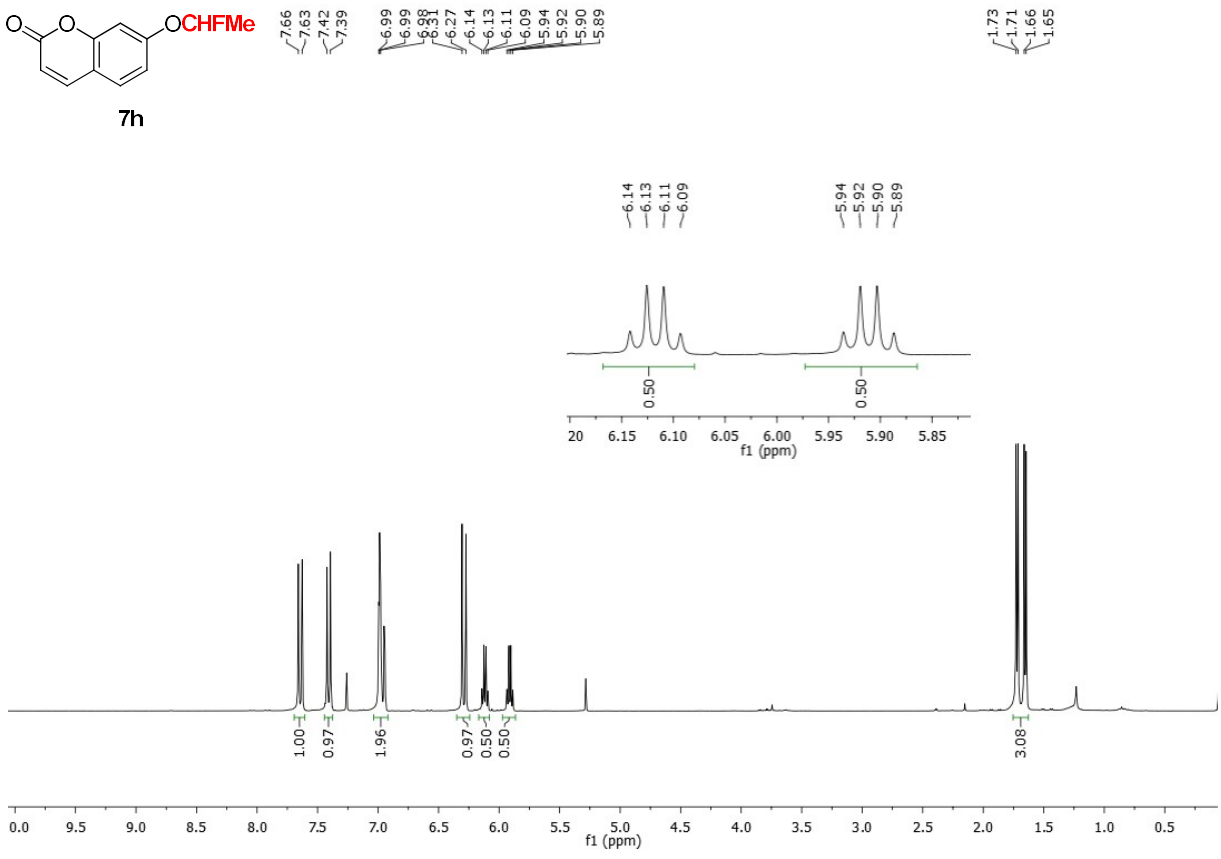
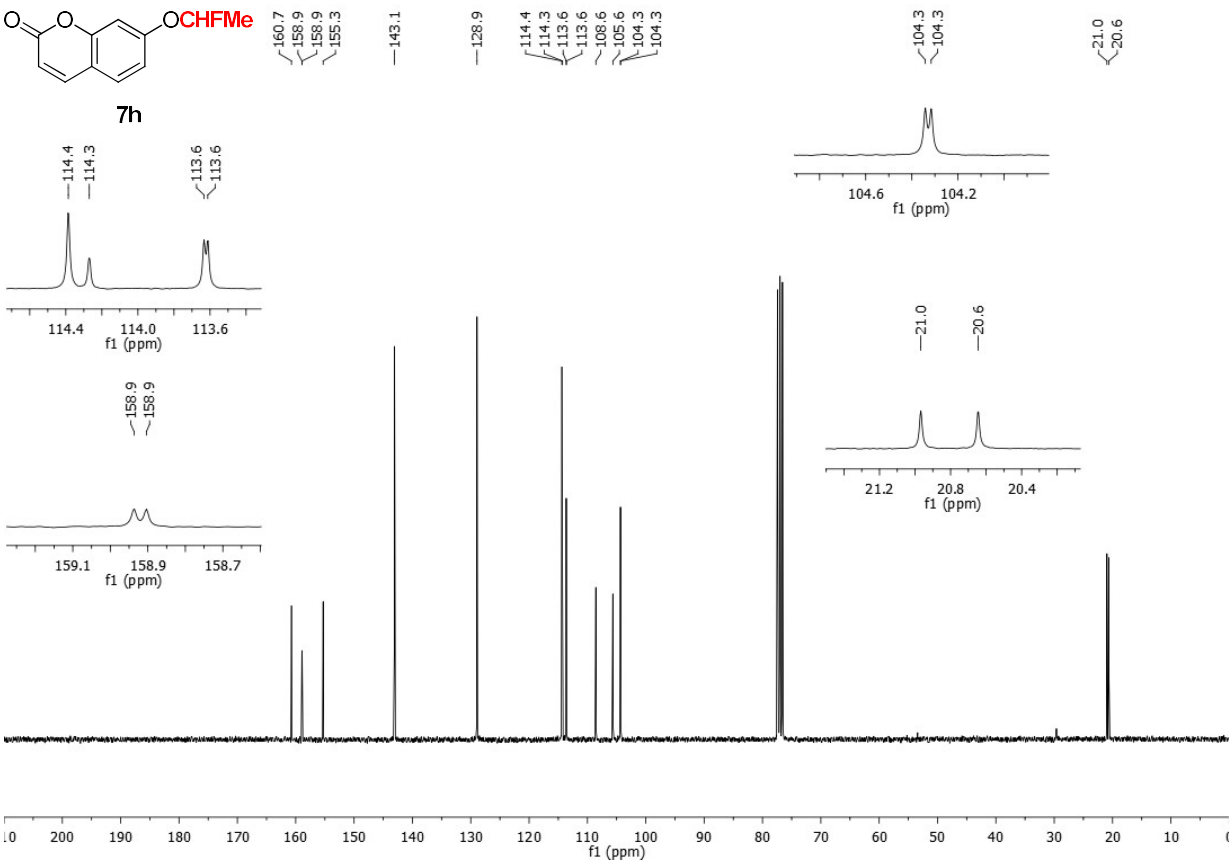
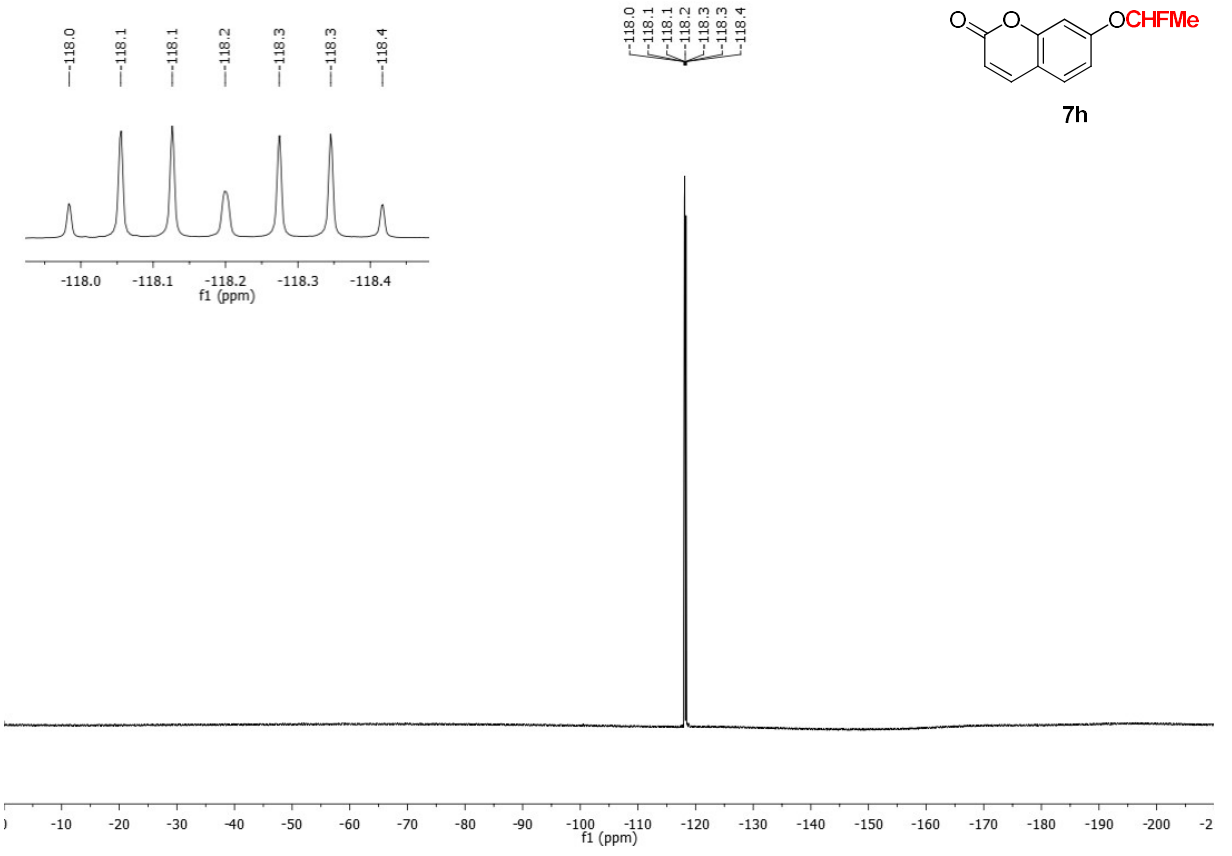
8.09
8.07
7.78
7.75
7.65
7.63
7.63
7.61
7.60
7.56
7.56
7.54
6.20
6.19
6.17
6.15
6.00
5.98
5.96
5.95
1.80
1.78
1.73
1.71
6.20
6.19
6.17
6.15
6.00
5.98
5.96
5.95
1.00
6.25
6.15
6.05
5.95
f1 (ppm)
OCHFMe
N
7g
0.94
1.02
2.03
2.07
1.00
3.14
f1 (ppm)

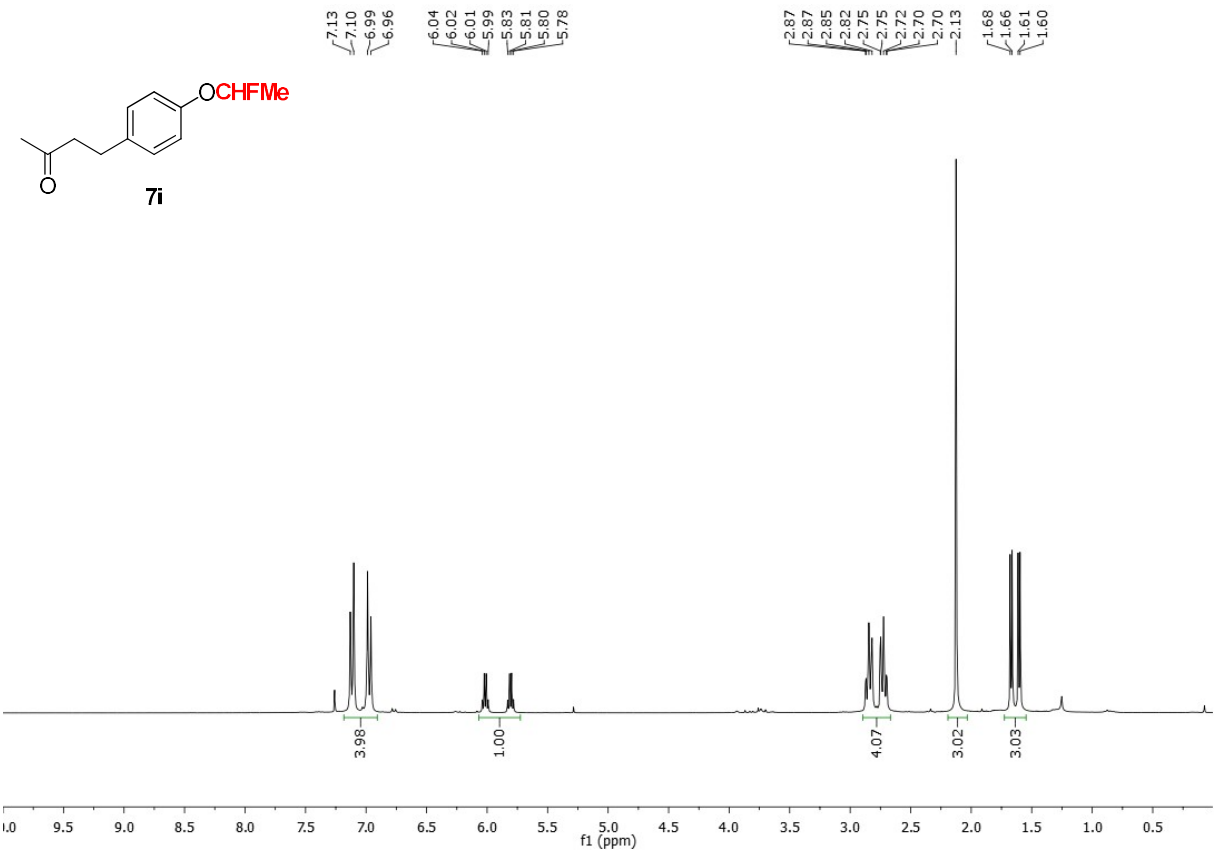

144.7
144.3
129.1
127.9
127.3
127.2
118.3
118.3
109.5
106.6
21.2
20.8
21.2
20.8
21.2
21.0
20.8
f1 (ppm)
OCHFMe
N
7g
f1 (ppm)


-117.3
-117.4
-117.5
-117.5
-117.6
-117.7
-117.7
17.2 -117.3 -117.4 -117.5 -117.6 -117.7 -117.8
f1 (ppm)
OCHFMe
N
7g
f1 (ppm)


OCHFMe
7h
7.66
7.63
7.42
7.39
6.99
6.99
6.98
6.91
6.27
6.14
6.13
6.11
6.09
5.94
5.92
5.90
5.89
1.73
1.71
1.66
1.65
0.50
0.50
20 6.15 6.10 6.05 6.00 5.95 5.90 5.85
f1 (ppm)
1.00
0.97
1.96
0.97
0.50
0.50
3.08
f1 (ppm)


OCHFMe
7h
160.7
158.9
158.9
155.3
143.1
128.9
114.4
114.3
113.6
113.6
108.6
105.6
104.3
104.3
104.3
104.3
21.0
20.6
f1 (ppm)


OCHFMe
7h
118.0
118.1
118.1
118.2
118.3
118.3
118.4
f1 (ppm)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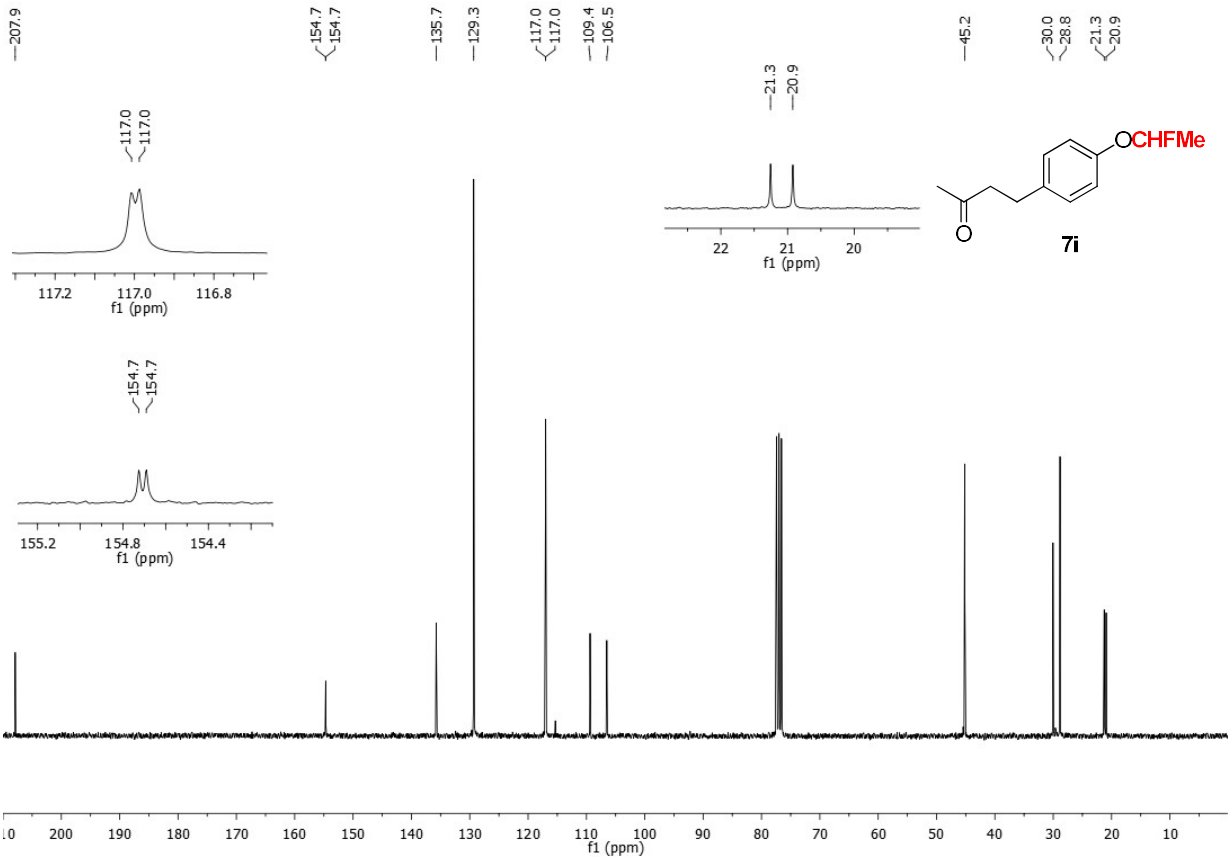
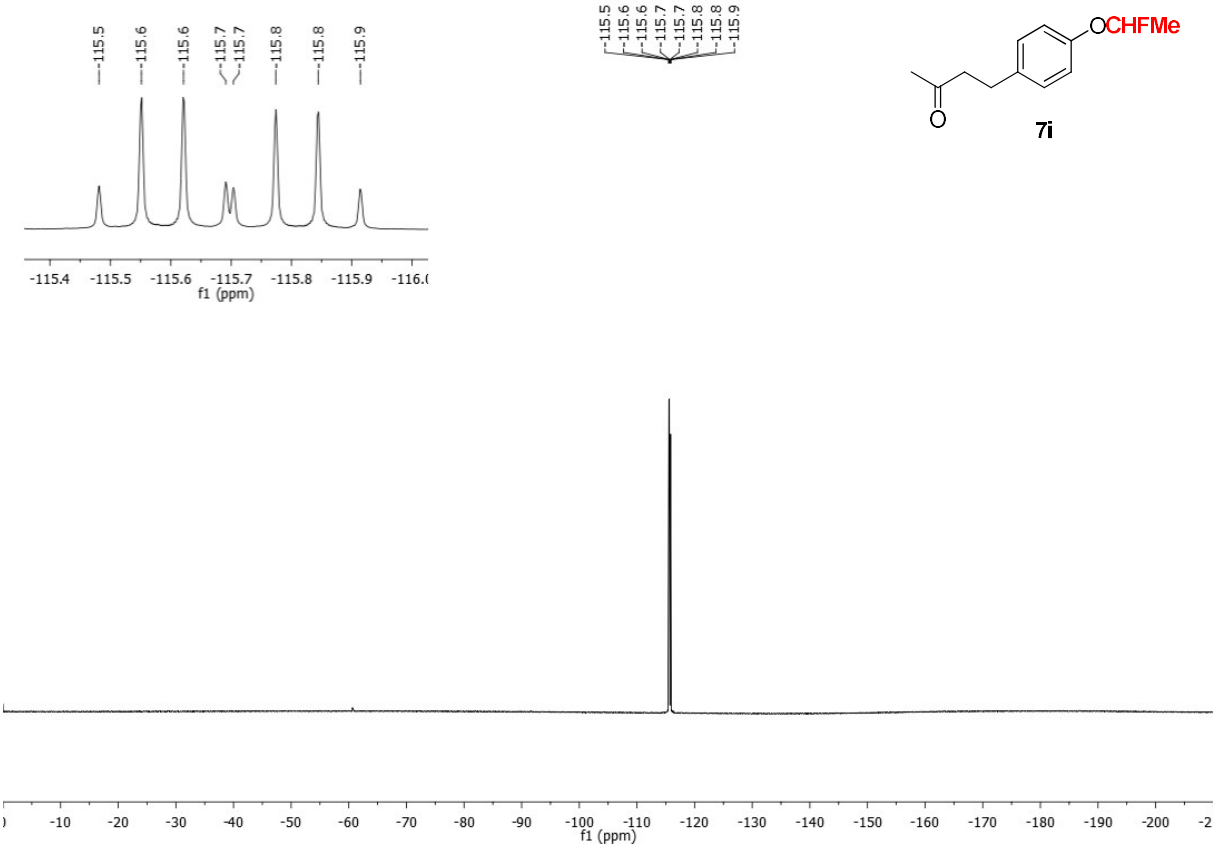
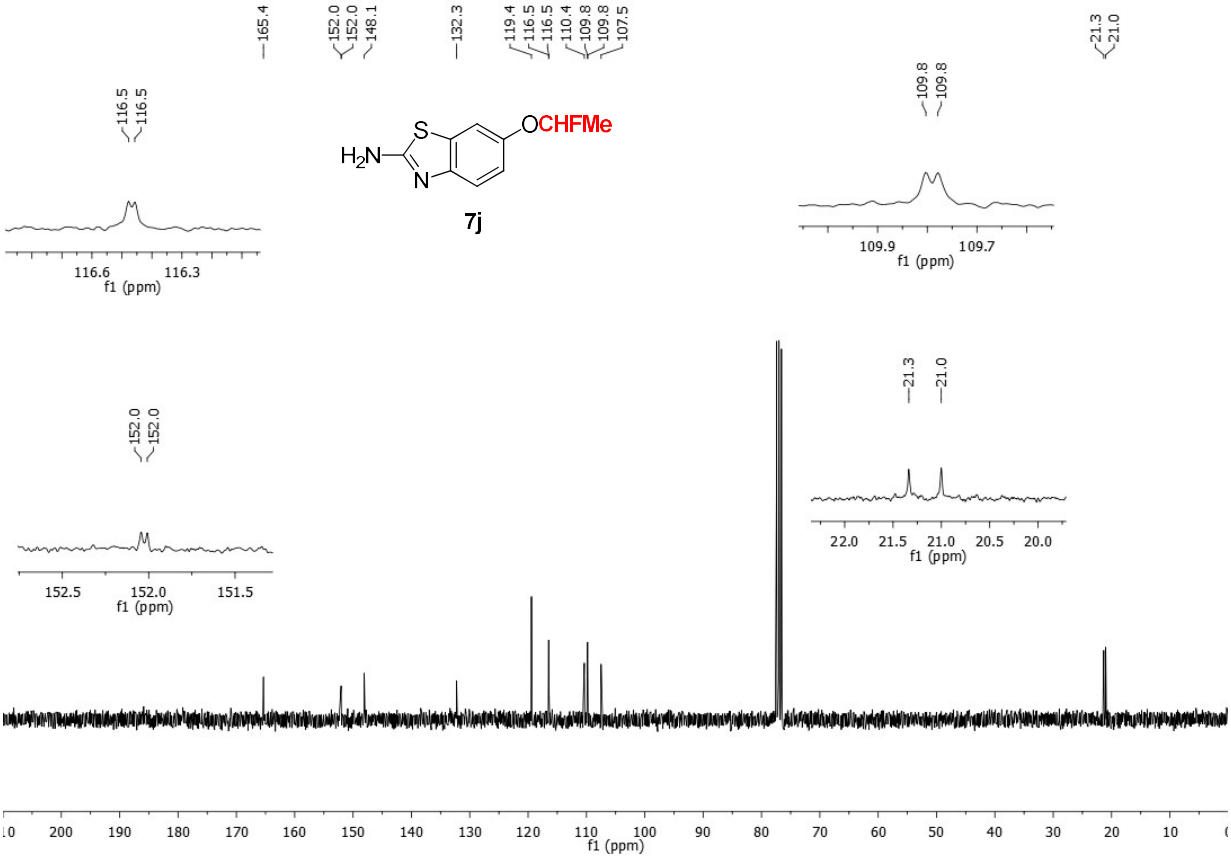
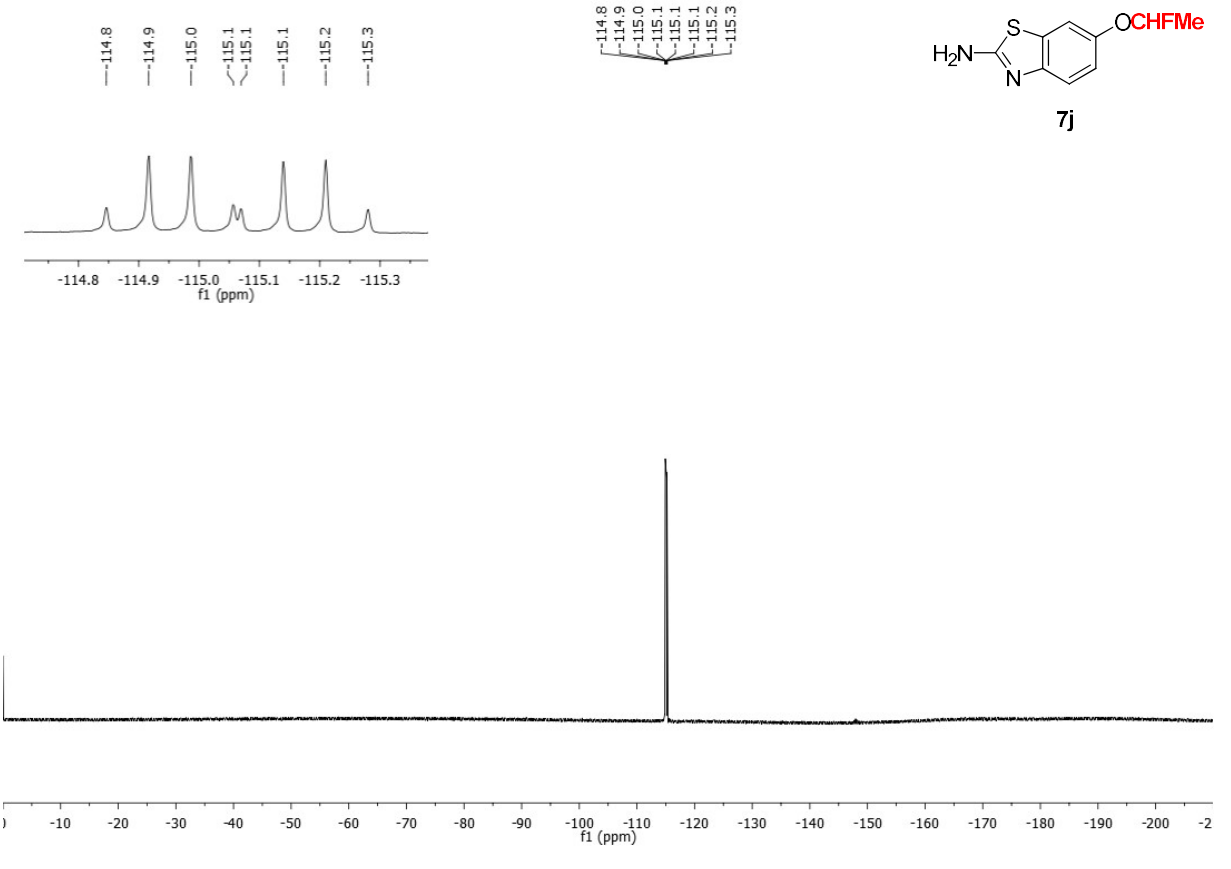
7.10
6.99
6.96
6.04
6.02
6.01
5.99
5.83
5.81
5.80
5.78
2.87
2.87
2.85
2.82
2.75
2.75
2.72
2.70
2.70
2.13
1.68
1.66
1.61
1.60
OCHFMe
O
7i
3.98
1.00
4.07
3.02
3.03
f1 (ppm)


207.9
154.7
154.7
135.7
129.3
117.0
117.0
109.4
106.5
45.2
30.0
28.8
21.3
20.9
117.0
117.0
117.2
117.0
116.8
f1 (ppm)
21.3
20.9
22
21
20
f1 (ppm)
OCHFMe
O
7i
154.7
154.7
155.2
154.8
154.4
f1 (ppm)
f1 (ppm)


-115.5
-115.6
-115.6
-115.7
-115.7
-115.8
-115.8
-115.9
-115.4 -115.5 -115.6 -115.7 -115.8 -115.9
f1 (ppm)
OCHFMe
O
7i
-10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200
f1 (ppm)
7.45
7.42
7.33
7.33
7.05
7.04
7.02
7.01
6.02
6.01
5.99
5.97
5.81
5.80
5.78
5.76
5.58
1.70
1.69
1.64
1.62
H2N
S
N
OCHFMe
7j
1.01
0.96
0.96
1.12
1.60
3.00
7.5 7.4 7.3 7.2 7.1 7.0
f1 (ppm)
1.12
6.00 5.95 5.90 5.85 5.80 5.75
f1 (ppm)
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)


165.4
152.0
152.0
148.1
132.3
119.4
116.5
116.5
110.4
109.8
109.8
107.5
21.3
21.0
116.5
116.5
109.8
109.8
OCHFMe
S
H2N
N
7j
116.6
116.3
f1 (ppm)
109.9
109.7
f1 (ppm)
152.0
152.0
21.3
21.0
152.5
152.0
151.5
f1 (ppm)
22.0
21.5
21.0
20.5
20.0
f1 (ppm)
f1 (ppm)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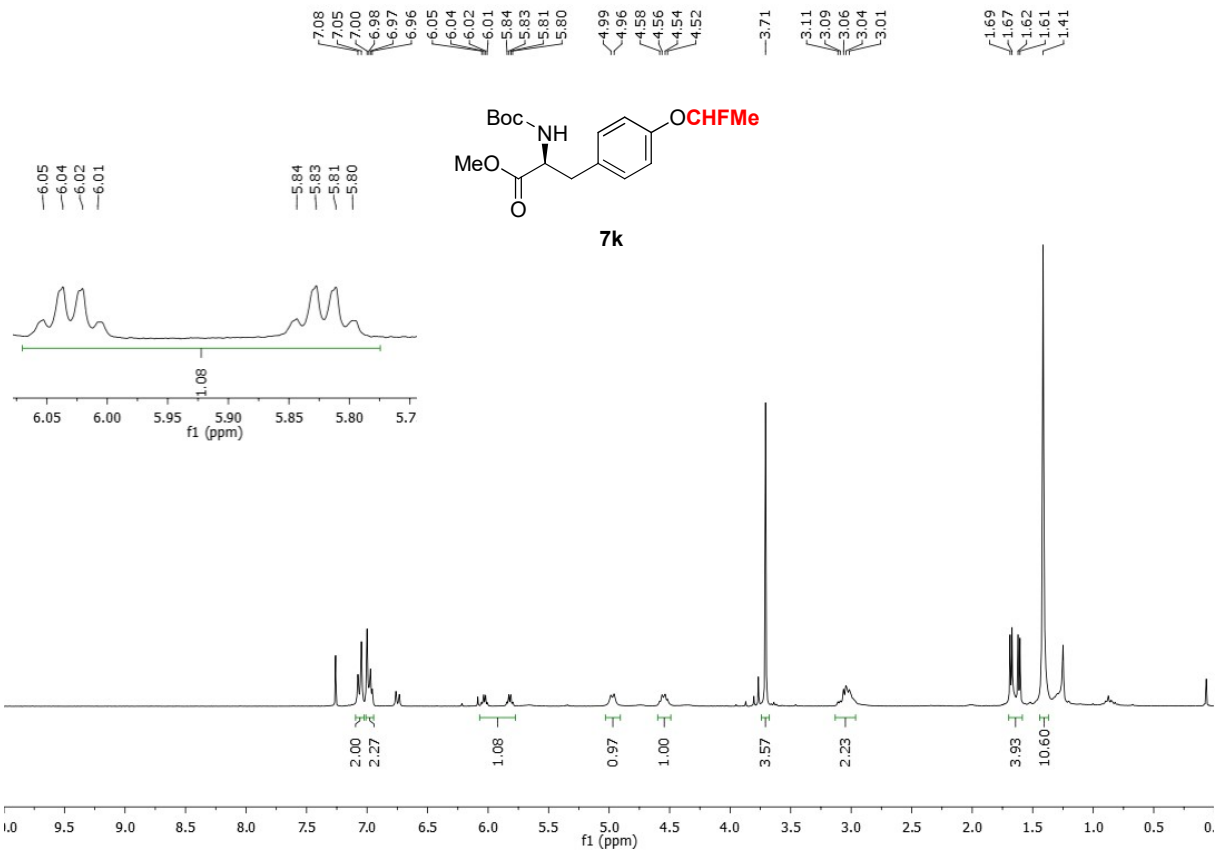
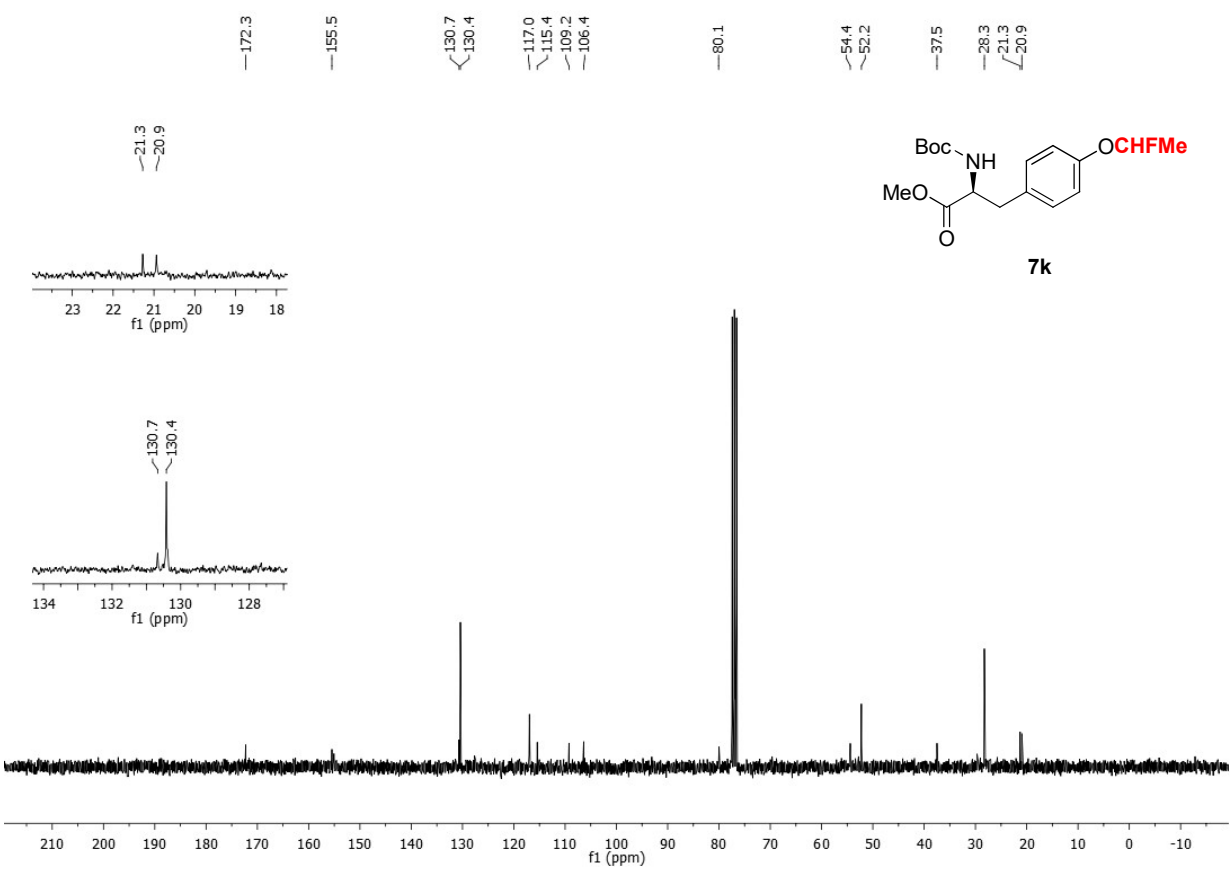
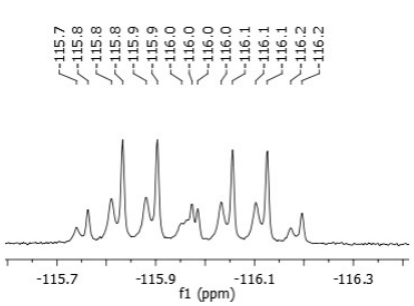
-114.9
-115.0
-115.1
-115.1
-115.1
-115.2
-115.3
-114.8
-114.9
-115.0
-115.1
-115.1
-115.1
-115.2
-115.3
OCHFMe
S
H2N
N
7j
-114.8
-114.9
-115.0
-115.1
-115.2
-115.3
f1 (ppm)
f1 (ppm)


7.08
7.05
7.00
6.98
6.97
6.96
6.05
6.04
6.02
6.01
5.84
5.83
5.81
5.80
4.99
4.96
4.58
4.56
4.54
4.52
3.71
3.11
3.09
3.06
3.04
3.01
1.69
1.67
1.62
1.61
1.41
Boc
NH
MeO
O
OCHFMe
7k
1.08
f1 (ppm)
2.00
2.27
1.08
0.97
1.00
3.57
2.23
3.93
10.60
f1 (ppm)


172.3
155.5
130.7
130.4
117.0
115.4
109.2
106.4
80.1
54.4
52.2
37.5
28.3
21.3
20.9
Boc
NH
MeO
O
OCHFMe
7k
f1 (ppm)
f1 (ppm)
f1 (ppm)


-115.7
-115.8
-115.8
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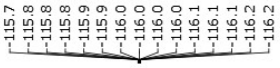
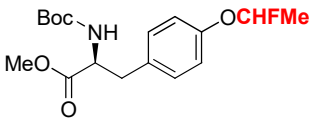
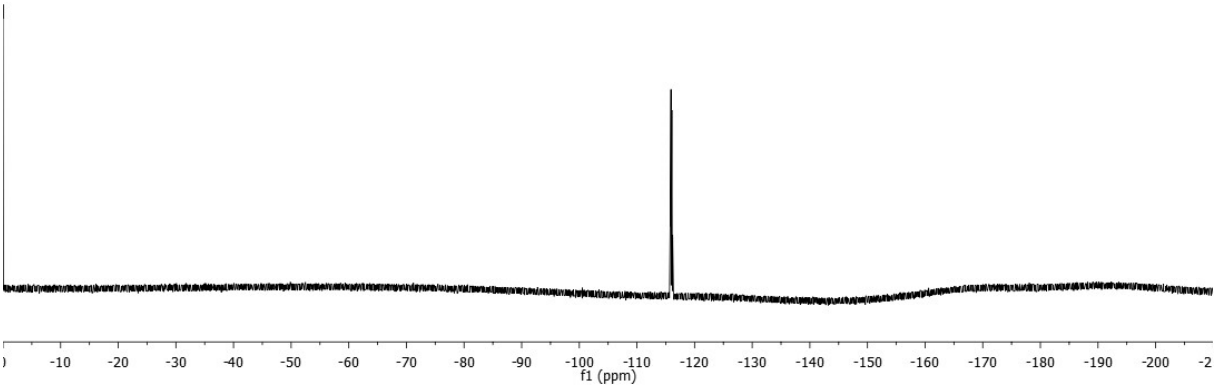
-115.9
-115.9
-116.0
-116.0
-116.0
-116.0
-116.1
-116.1
-116.1
-116.2
-116.2
-115.7
-115.9
-116.1
-116.3
f1 (ppm)

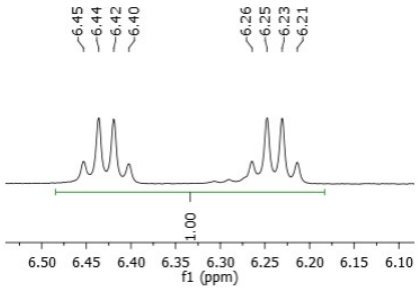

-115.7
-115.8
-115.8
-115.8
-115.9
-115.9
-116.0
-116.0
-116.0
-116.0
-116.1
-116.1
-116.1
-116.2
-116.2


Boc
NH
OCHFMe
MeO
O

7k


-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
f1 (ppm)


6.44
6.42
6.25
6.23
4.62
2.48
2.45
2.42
2.39
2.17
2.09
2.06
2.03
2.02
2.00
1.97
1.95
1.93
1.91
1.88
1.81
1.78
1.74
1.71
1.70
1.70
1.69
1.66
1.62
1.59
1.58
1.53
1.51
1.49
1.48
1.46
1.45
1.45
1.44
1.38
1.37
1.36
1.34
1.33
1.31
1.29
1.27
1.26
1.24
1.24
1.23
1.20
1.19
1.18
1.08
1.05
1.03
1.00
0.99
0.85
0.84
0.82
0.74
0.71
0.70


6.45
6.44
6.42
6.40
6.26
6.25
6.23
6.21
1.00
6.50
6.45
6.40
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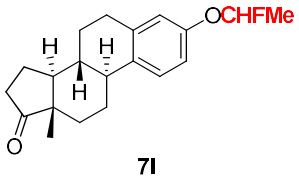
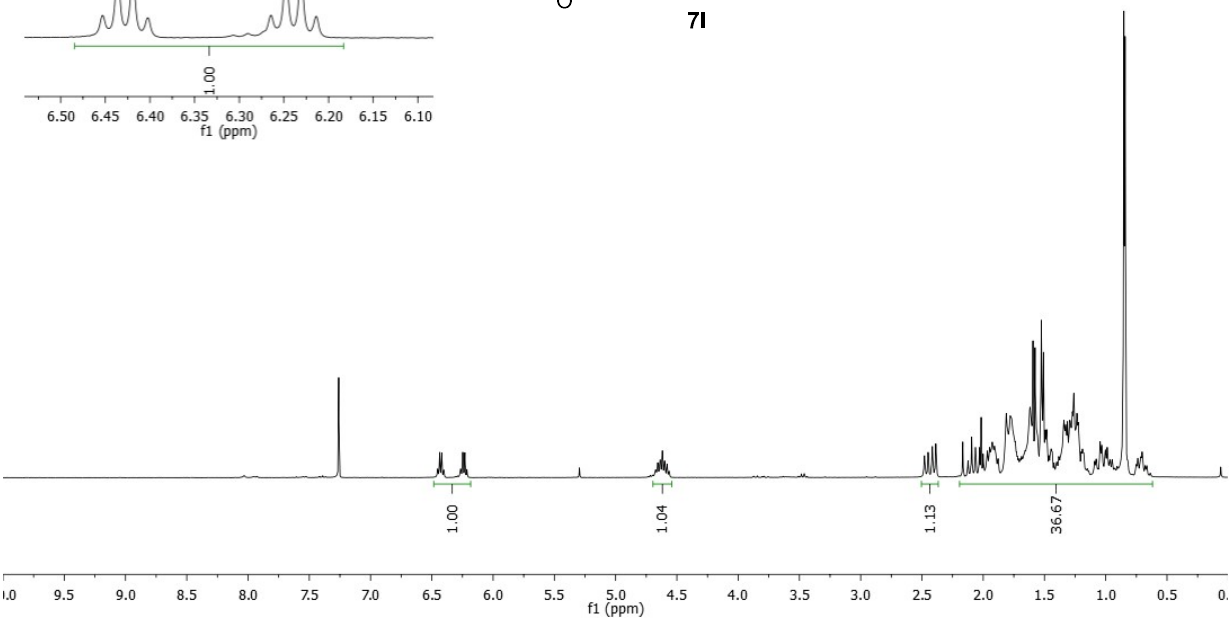
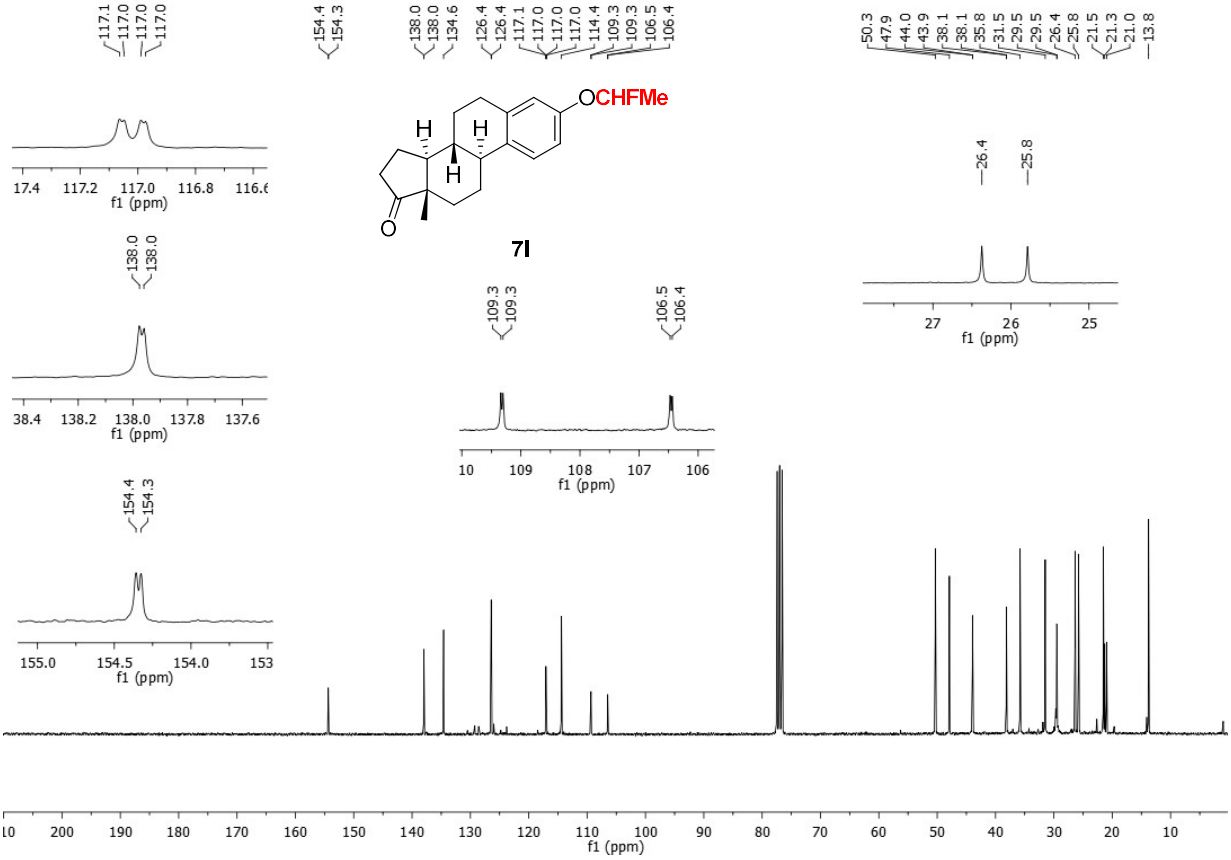
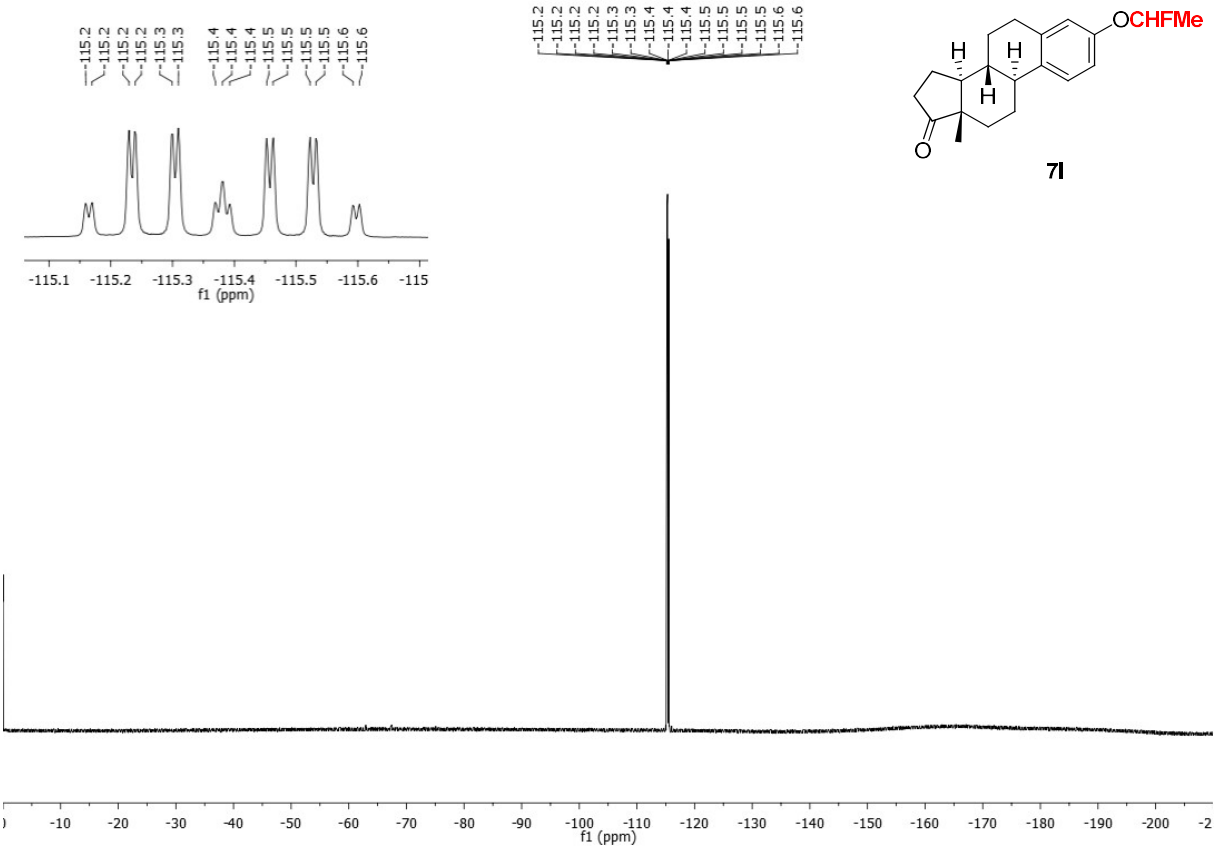
6.30
6.25
6.20
6.15
6.10
f1 (ppm)

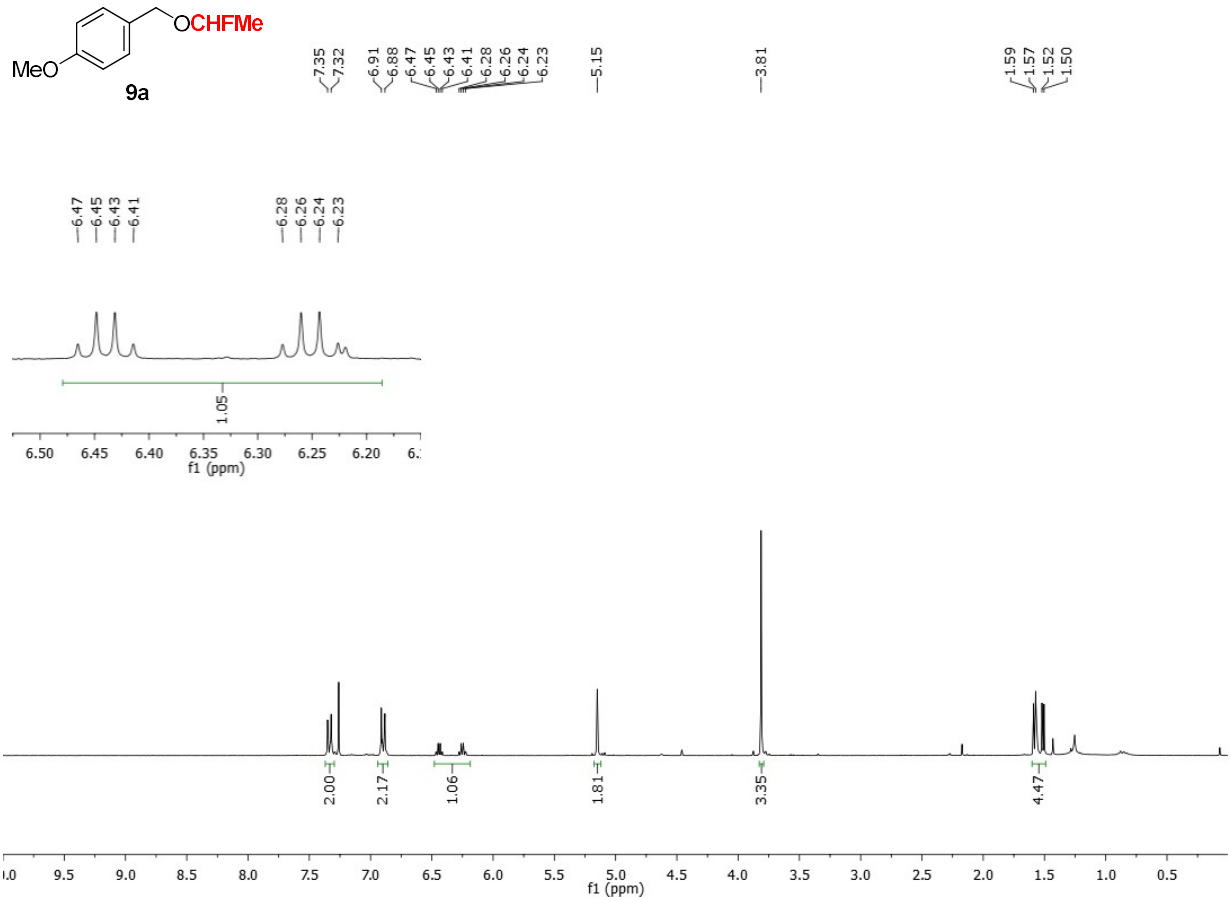

OCHFMe
H
H
H
O

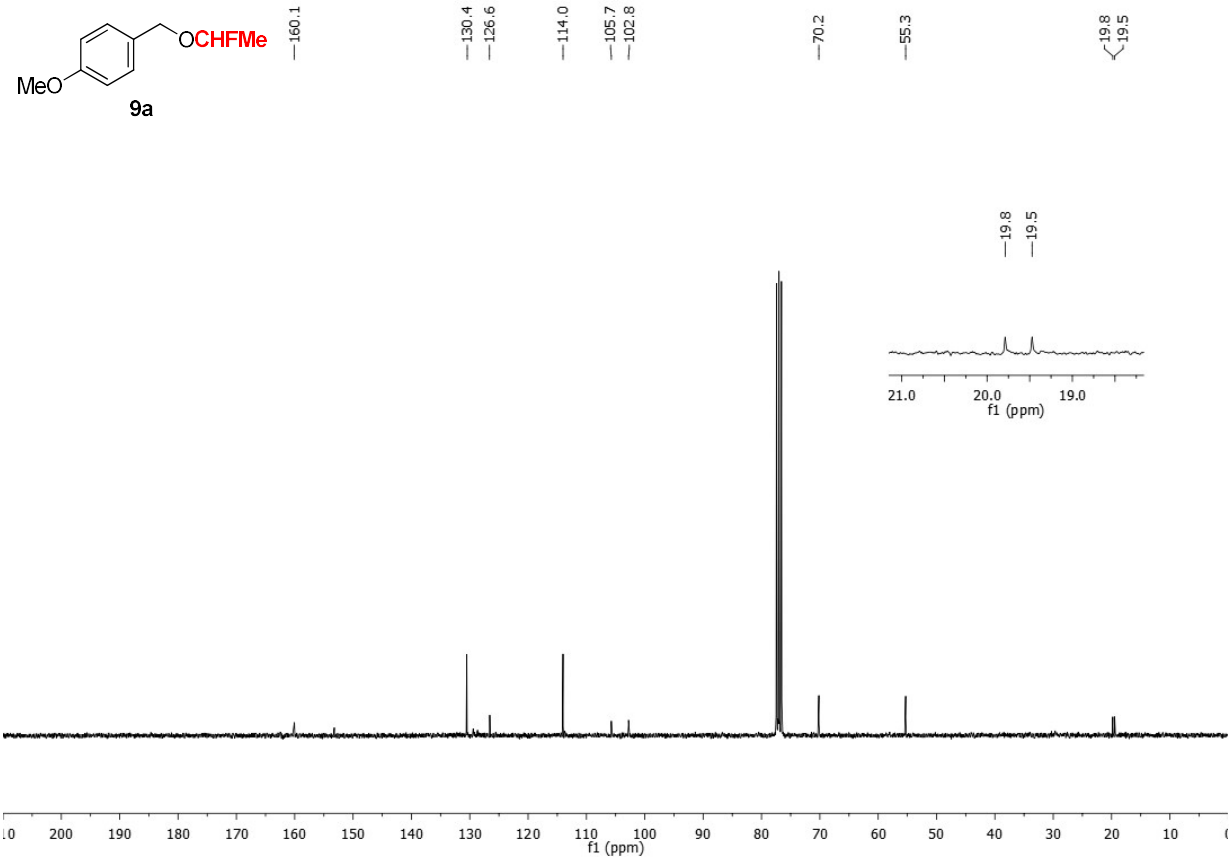
7l

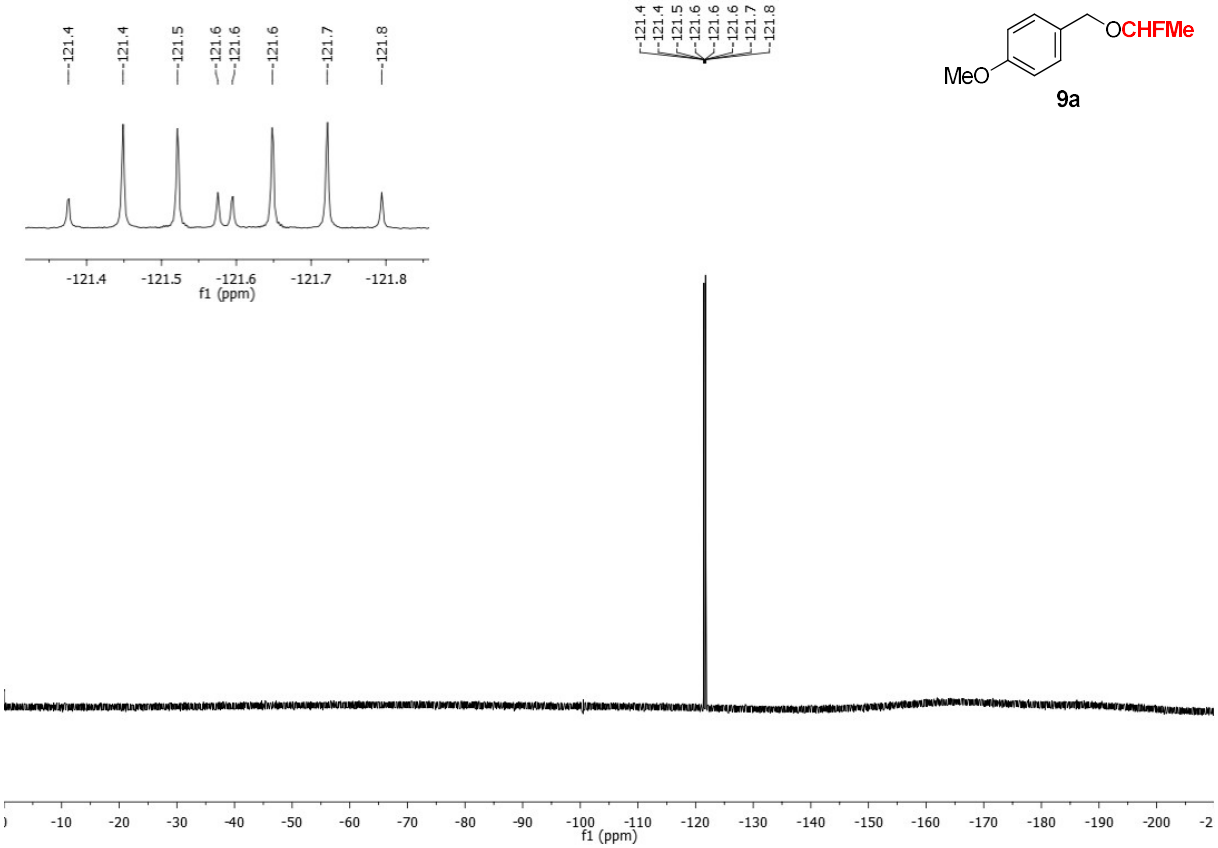

1.00
1.04
1.13
36.67
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

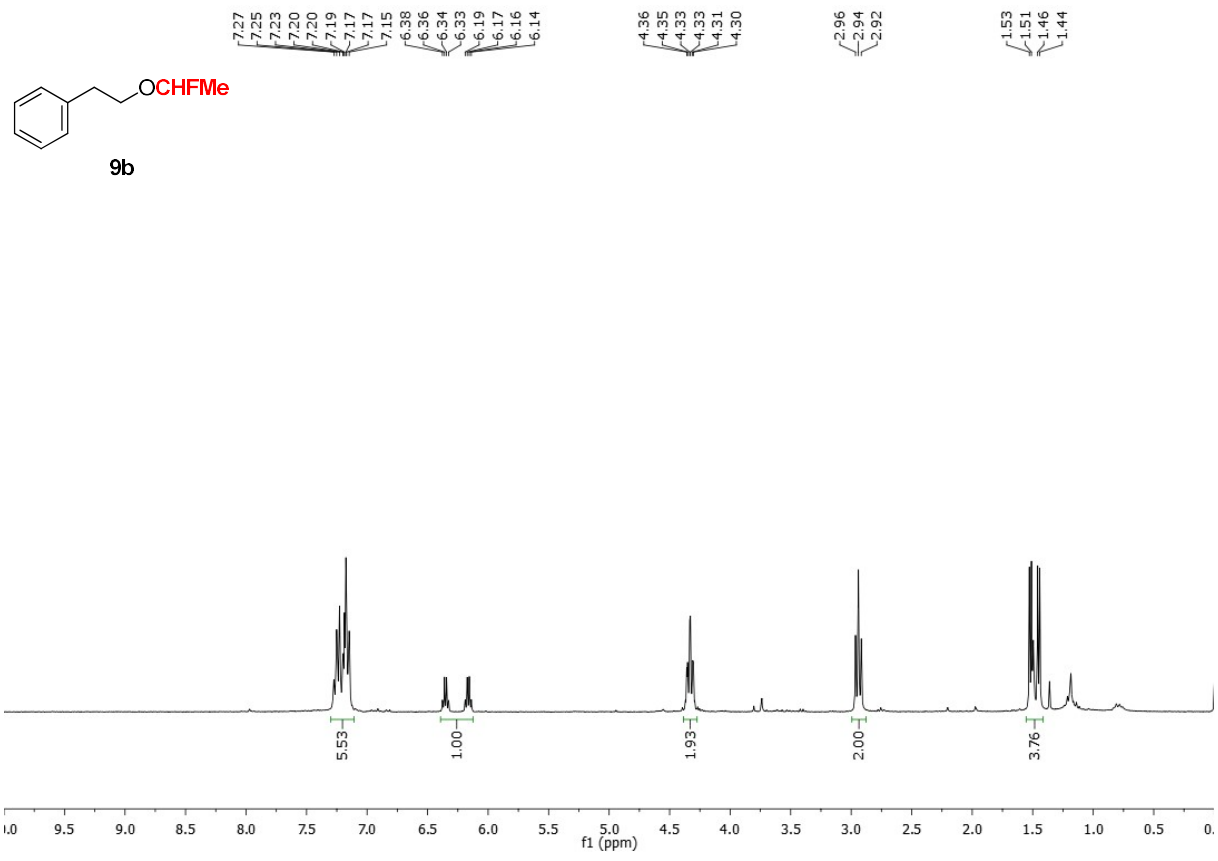

OCHFMe
H
H
H
O
7l
f1 (ppm)

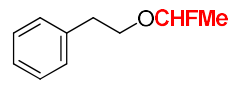

OCHFMe
H
H
H
O
7l
f1 (ppm)


OCHFMe
MeO
9a
7.35
7.32
6.91
6.88
6.47
6.45
6.43
6.41
6.28
6.26
6.24
6.23
5.15
3.81
1.59
1.57
1.52
1.50
1.05
f1 (ppm)
2.00
2.17
1.06
1.81
3.35
4.47
f1 (ppm)


OCHFMe
MeO
9a
160.1
130.4
126.6
114.0
105.7
102.8
70.2
55.3
19.8
19.5
f1 (ppm)
f1 (ppm)

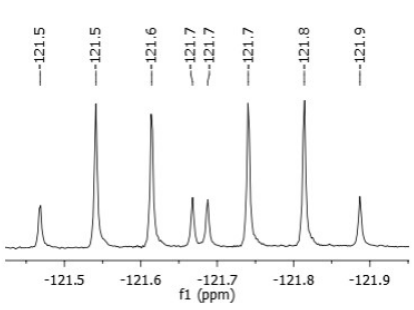

-121.4
-121.4
-121.5
-121.6
-121.6
-121.6
-121.7
-121.8
-121.4
-121.5
-121.6
-121.7
-121.8
f1 (ppm)
OCHFMe
MeO
9a
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
f1 (ppm)

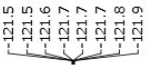

7.27
7.25
7.23
7.20
7.20
7.19
7.17
7.17
7.15
6.38
6.36
6.34
6.33
6.19
6.17
6.16
6.14
4.36
4.35
4.33
4.33
4.31
4.30
2.96
2.94
2.92
1.53
1.51
1.46
1.44
OCHFMe
9b
5.53
1.00
1.93
2.00
3.76
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

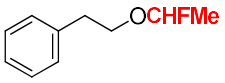

OCHFMe

9b

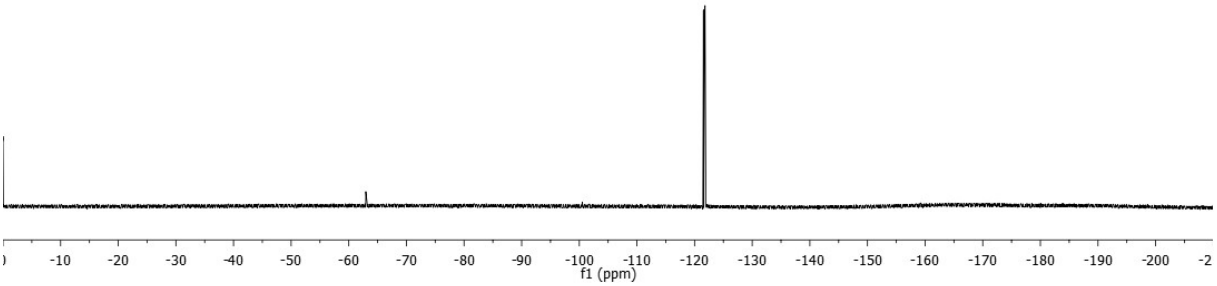
136.9
128.9
128.7
126.8
105.7
102.8
69.0
35.0
19.8
19.5
128.9
128.7
129.5
129.0
128.5
128.0
f1 (ppm)
19.8
19.5
22
21
20
19
18
f1 (ppm)
f1 (ppm)

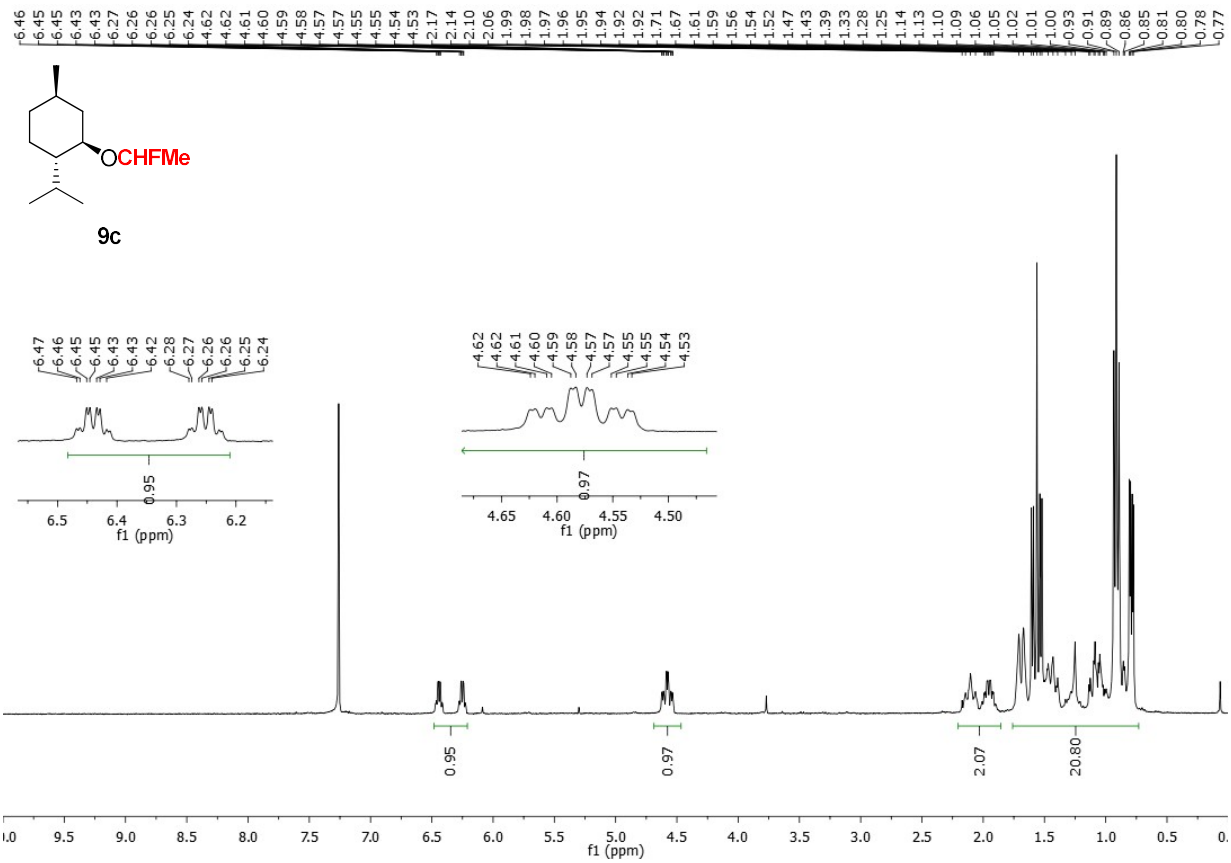

-121.5
-121.5
-121.6
-121.7
-121.7
-121.7
-121.8
-121.9
-121.5
-121.6
-121.7
-121.8
-121.9
f1 (ppm)

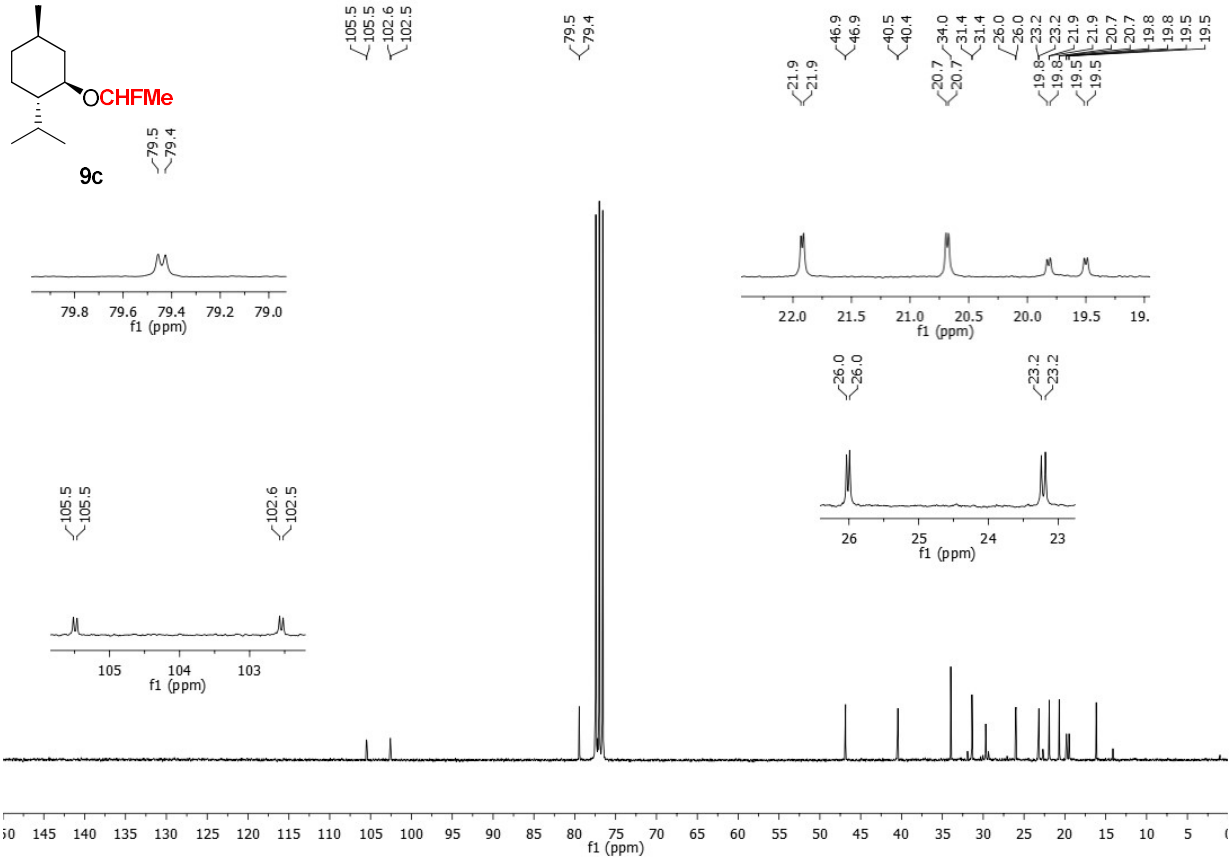

-121.5
-121.5
-121.6
-121.7
-121.7
-121.7
-121.8
-121.9

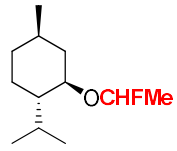

OCHFMe

9b

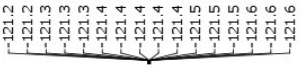

-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
f1 (ppm)

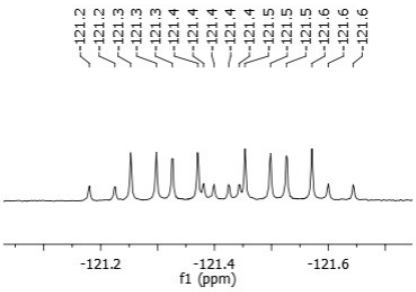

OCHFMe
9c
f1 (ppm)
0.95
0.97
2.07
20.80


OCHFMe
9c
f1 (ppm)

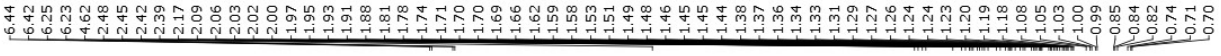

OCHFMe

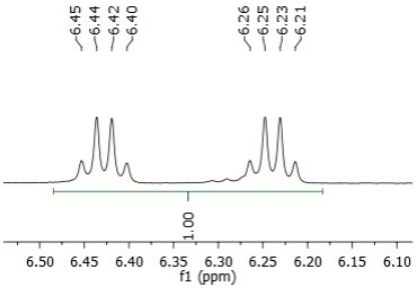
9c

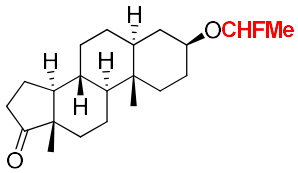

-121.2
-121.2
-121.3
-121.3
-121.3
-121.4
-121.4
-121.4
-121.4
-121.4
-121.5
-121.5
-121.5
-121.6
-121.6
-121.6


-121.2
-121.4
-121.6
f1 (ppm)

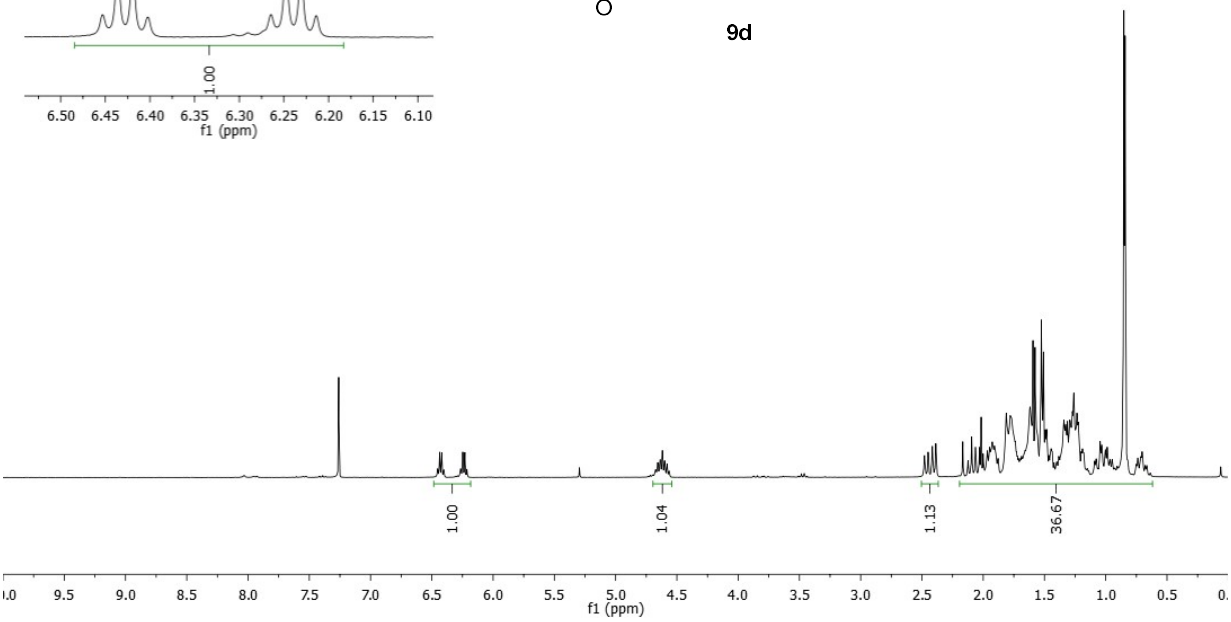
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
f1 (ppm)

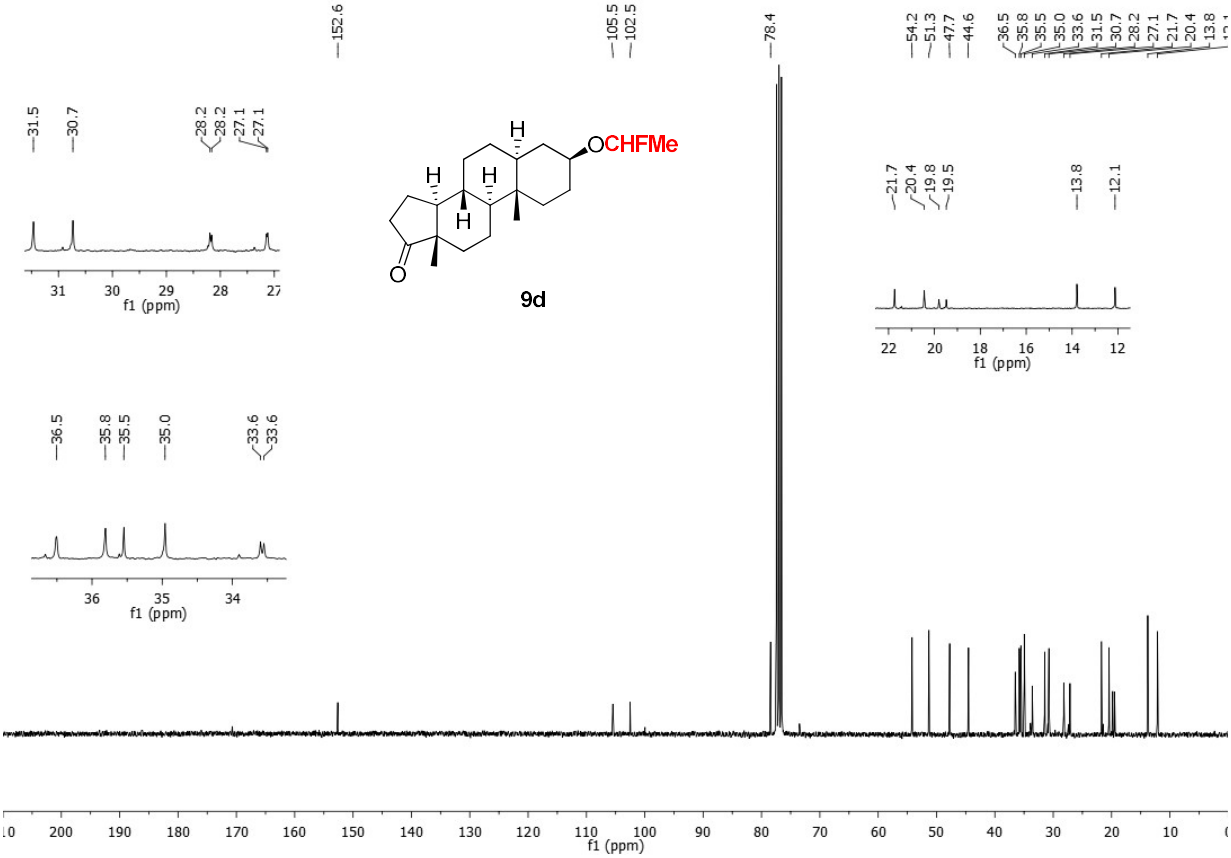

6.44
6.42
6.25
6.23
4.62
2.48
2.45
2.42
2.39
2.17
2.09
2.06
2.03
2.02
2.00
1.97
1.95
1.93
1.91
1.88
1.81
1.78
1.74
1.71
1.70
1.70
1.69
1.66
1.62
1.59
1.58
1.53
1.51
1.49
1.48
1.46
1.45
1.45
1.44
1.38
1.37
1.36
1.34
1.33
1.31
1.29
1.27
1.26
1.24
1.24
1.23
1.20
1.19
1.18
1.08
1.05
1.03
1.00
0.99
0.85
0.84
0.82
0.74
0.71
0.70

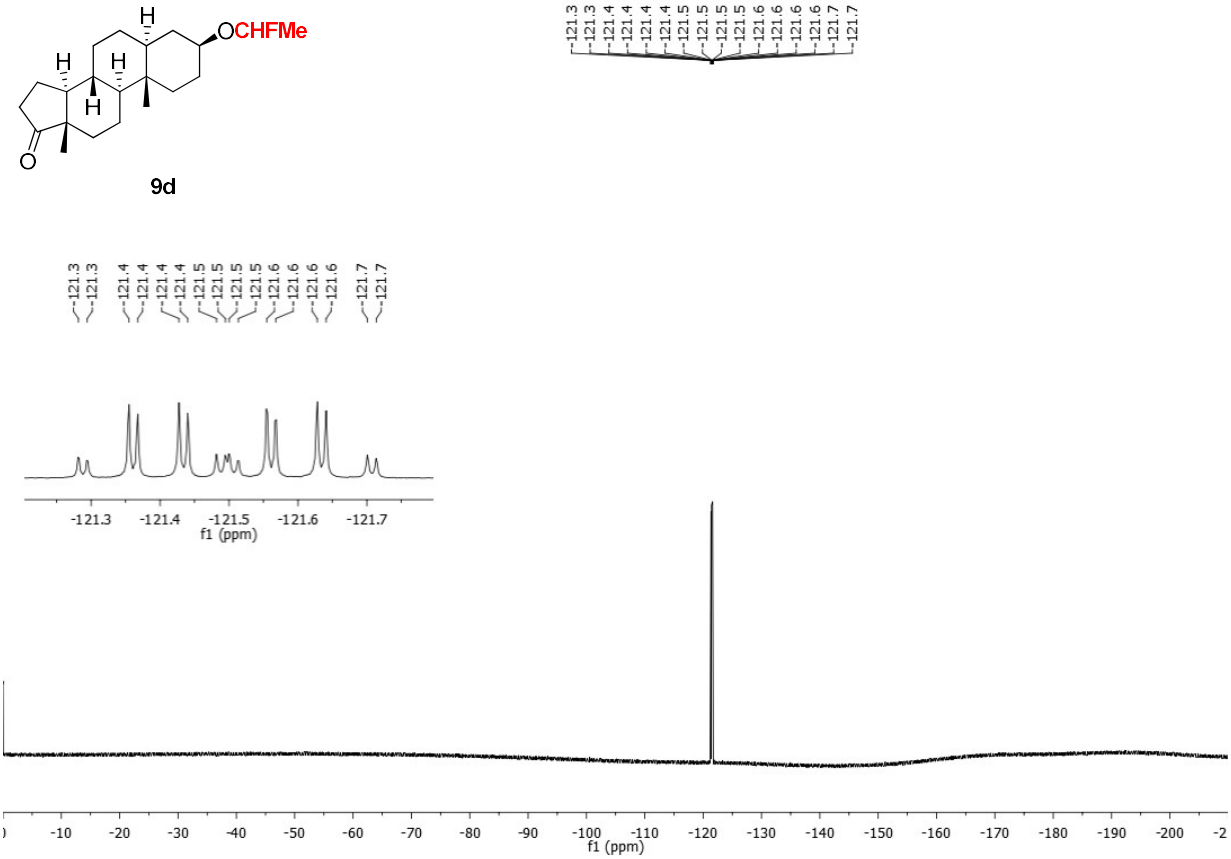

6.45
6.44
6.42
6.40
6.26
6.25
6.23
6.21
1.00
6.50
6.45
6.40
6.35
6.30
6.25
6.20
6.15
6.10
f1 (ppm)

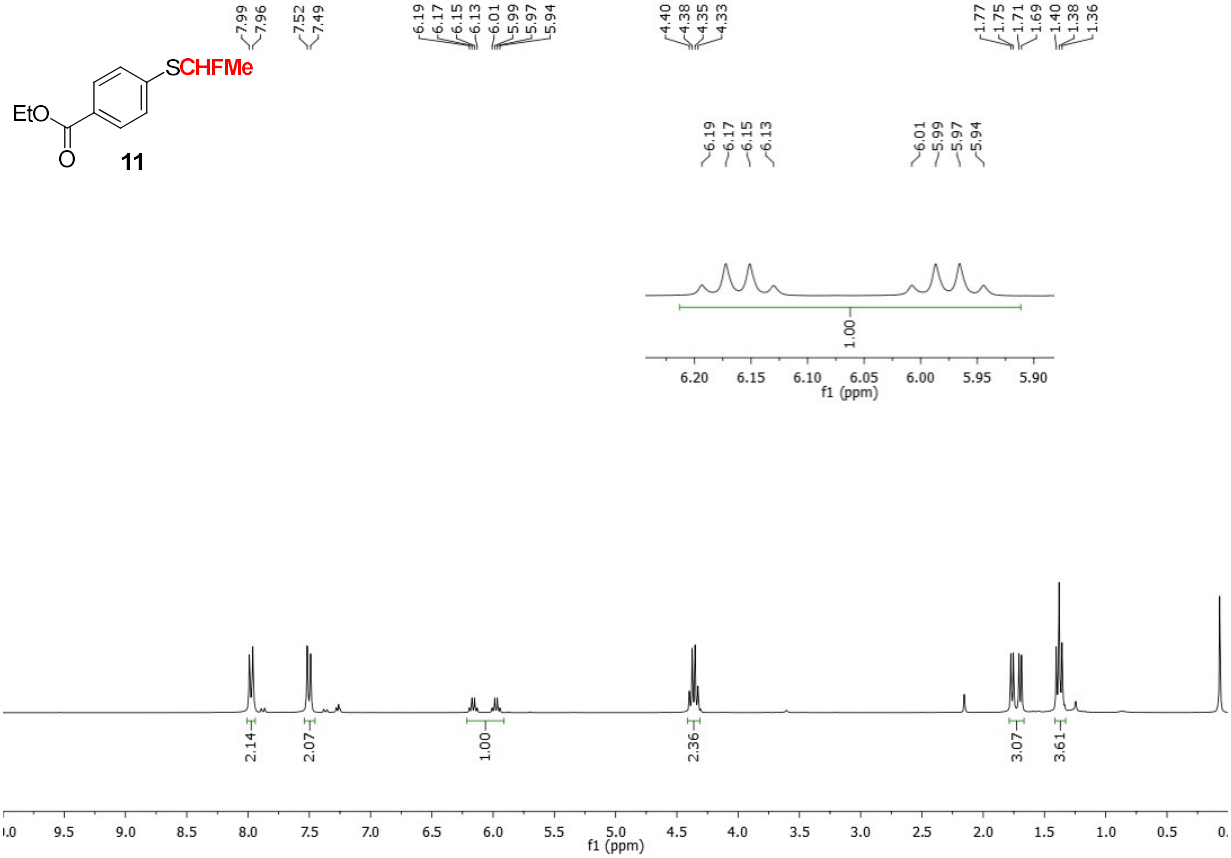

H
OCHFMe
H
H
H
O

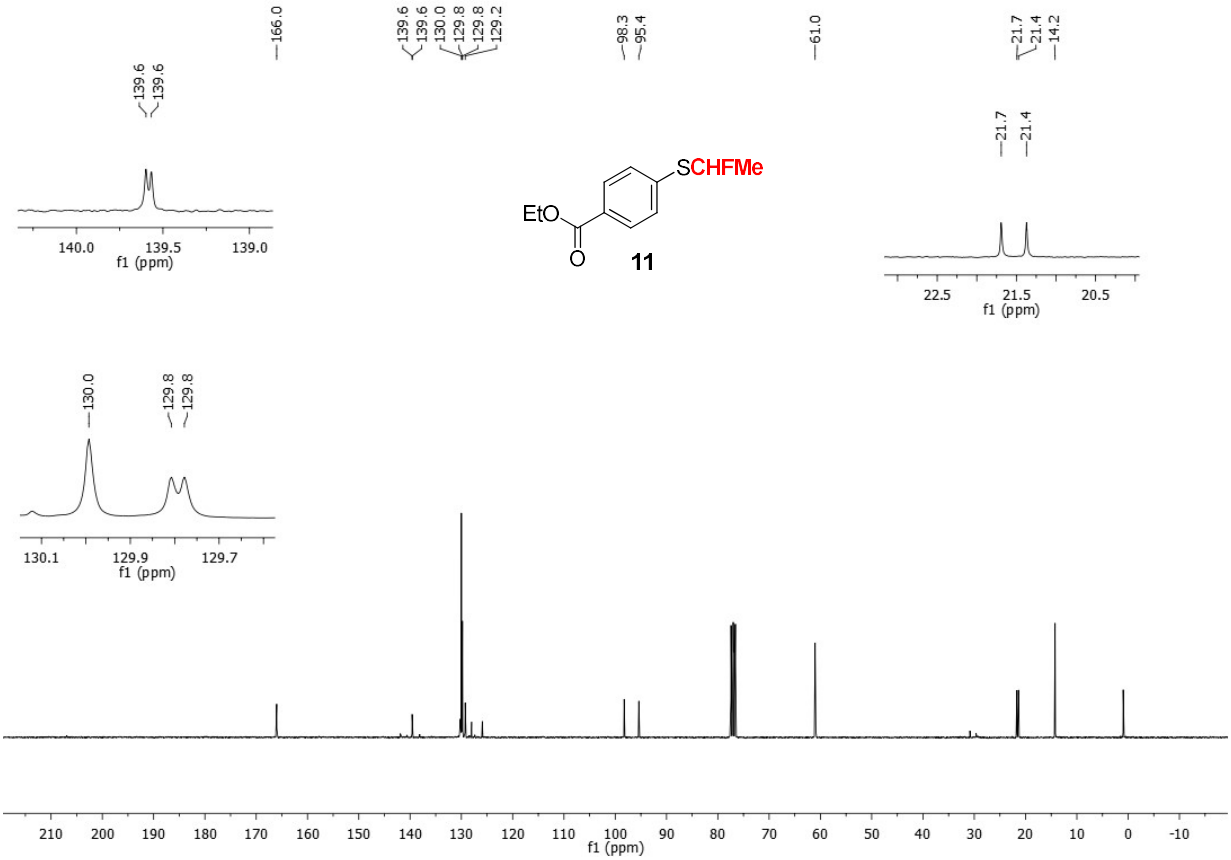
9d

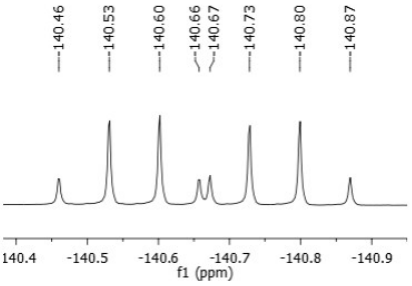

1.00
1.04
1.13
36.67
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


152.6
105.5
102.5
78.4
54.2
51.3
47.7
44.6
36.5
35.8
35.5
35.0
33.6
31.5
30.7
28.2
27.1
21.7
20.4
13.8
31.5
30.7
28.2
28.2
27.1
27.1
f1 (ppm)
OCHFMe
9d
21.7
20.4
19.8
19.5
13.8
12.1
f1 (ppm)
36.5
35.8
35.5
35.0
33.6
33.6
f1 (ppm)
f1 (ppm)

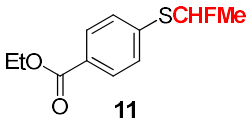

121.3
121.3
121.4
121.4
121.4
121.4
121.5
121.5
121.5
121.5
121.6
121.6
121.6
121.6
121.7
121.7
OCHFMe
9d
121.3
121.3
121.4
121.4
121.4
121.4
121.5
121.5
121.5
121.5
121.6
121.6
121.6
121.6
121.7
121.7
-121.3
-121.4
-121.5
-121.6
-121.7
f1 (ppm)
f1 (ppm)


SCHFMe
EtO
O
11
7.99
7.96
7.52
7.49
6.19
6.17
6.15
6.13
6.01
5.99
5.97
5.94
4.40
4.38
4.35
4.33
1.77
1.75
1.71
1.69
1.40
1.38
1.36
1.00
6.20
6.15
6.10
6.05
6.00
5.95
5.90
f1 (ppm)
2.14
2.07
1.00
2.36
3.07
3.61
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

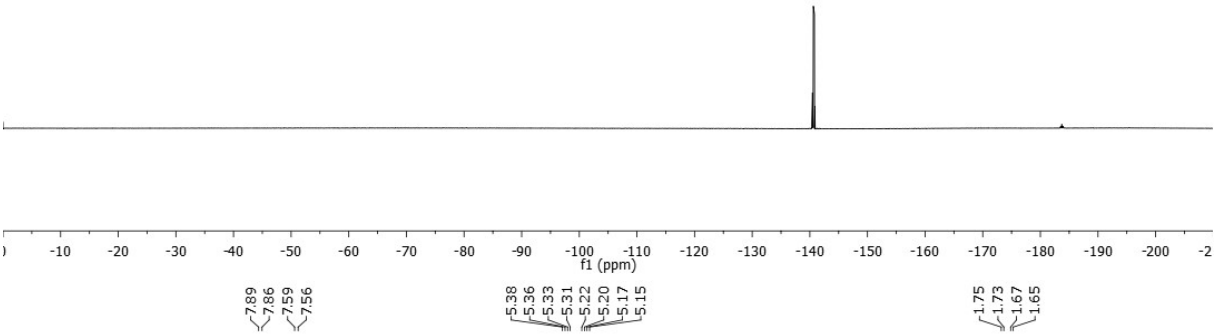

166.0
139.6
139.6
130.0
129.8
129.8
129.2
98.3
95.4
61.0
21.7
21.4
14.2
SCHFMe
EtO
O
11
140.0
139.5
139.0
f1 (ppm)
22.5
21.5
20.5
130.1
129.9
129.7
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10

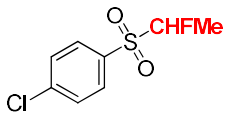

-140.46
-140.53
-140.60
-140.66
-140.67
-140.73
-140.80
-140.87
140.4
-140.5
-140.6
-140.7
-140.8
-140.9
f1 (ppm)


-140.5
-140.5
-140.6
-140.7
-140.7
-140.7
-140.8
-140.9

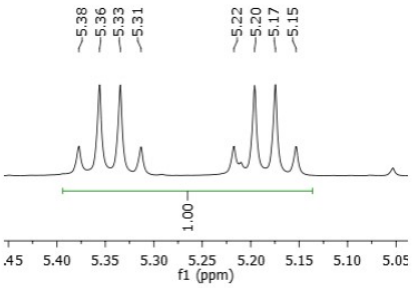

SCHFMe
EtO
O

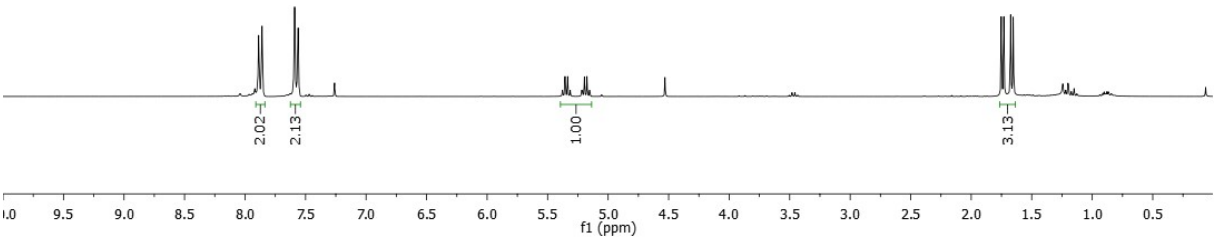
11

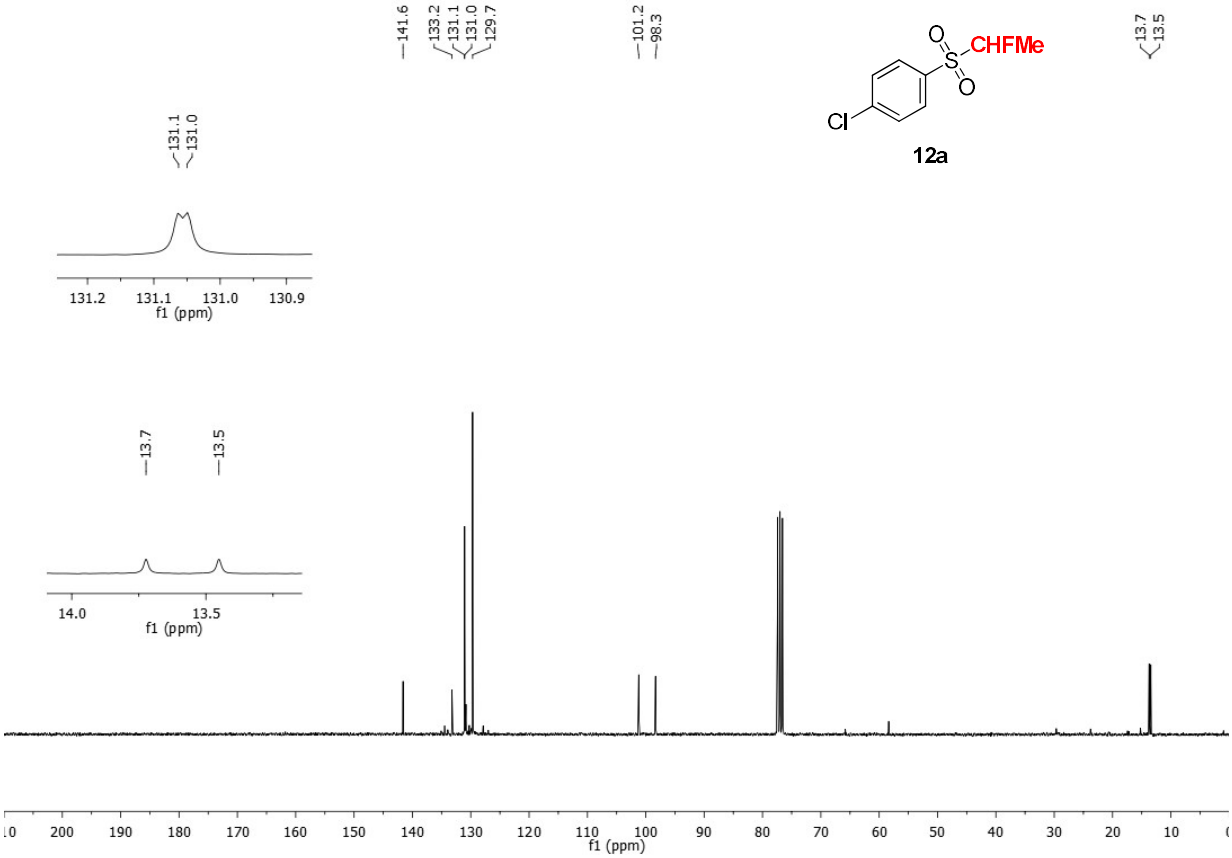

-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
f1 (ppm)
7.89
7.86
7.59
7.56
5.38
5.36
5.33
5.31
5.22
5.20
5.17
5.15
1.75
1.73
1.67
1.65

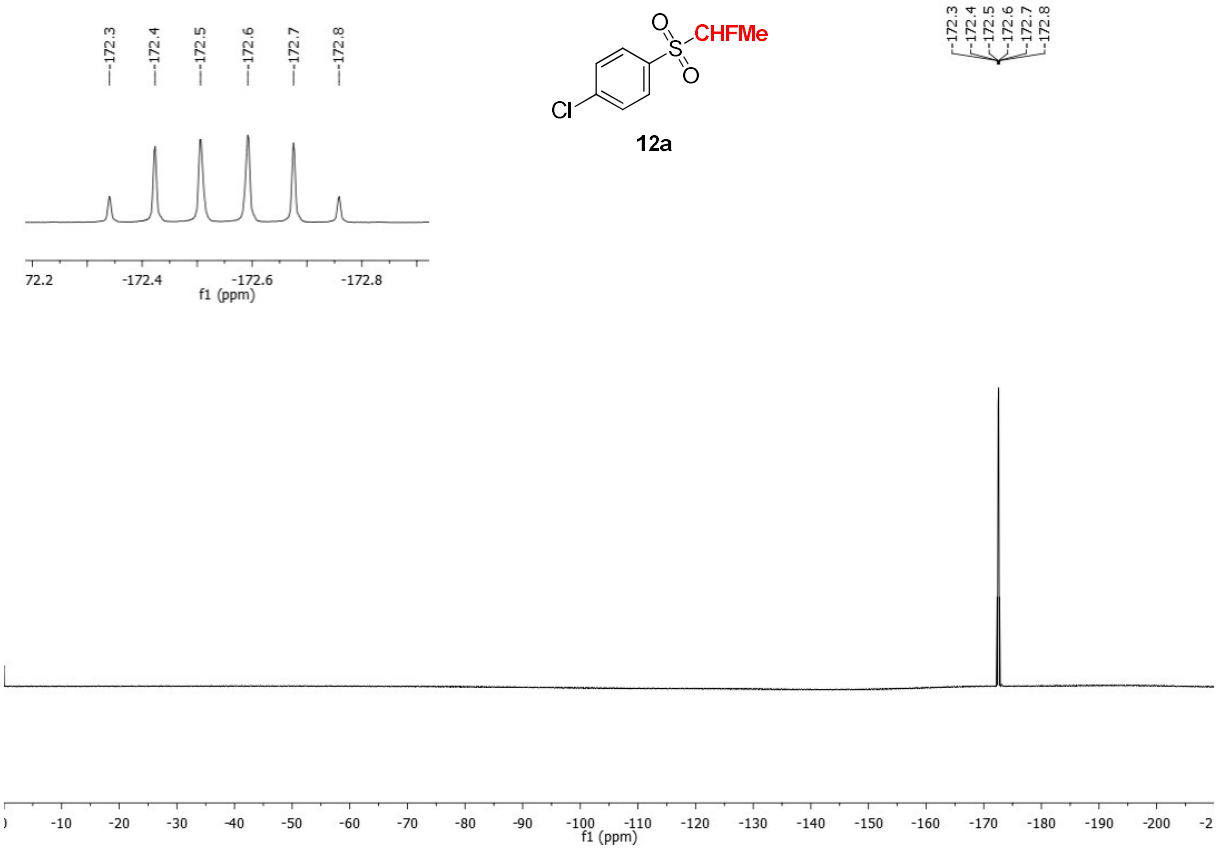

O
S
CHFMe
O
Cl

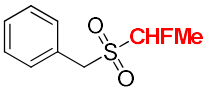
12a


5.38
5.36
5.33
5.31
5.22
5.20
5.17
5.15
1.00
.45
5.40
5.35
5.30
5.25
5.20
5.15
5.10
5.05
f1 (ppm)


2.02
2.13
1.00
3.13
.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)

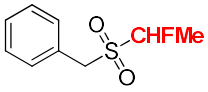

141.6
133.2
131.1
131.0
129.7
101.2
98.3
13.7
13.5
CHFMe
Cl
12a
131.1
131.0
131.2
131.1
131.0
130.9
f1 (ppm)
13.7
13.5
14.0
13.5
f1 (ppm)
f1 (ppm)


172.3
172.4
172.5
172.6
172.7
172.8
CHFMe
Cl
12a
72.2
-172.4
-172.6
-172.8
f1 (ppm)
f1 (ppm)


CHFMe

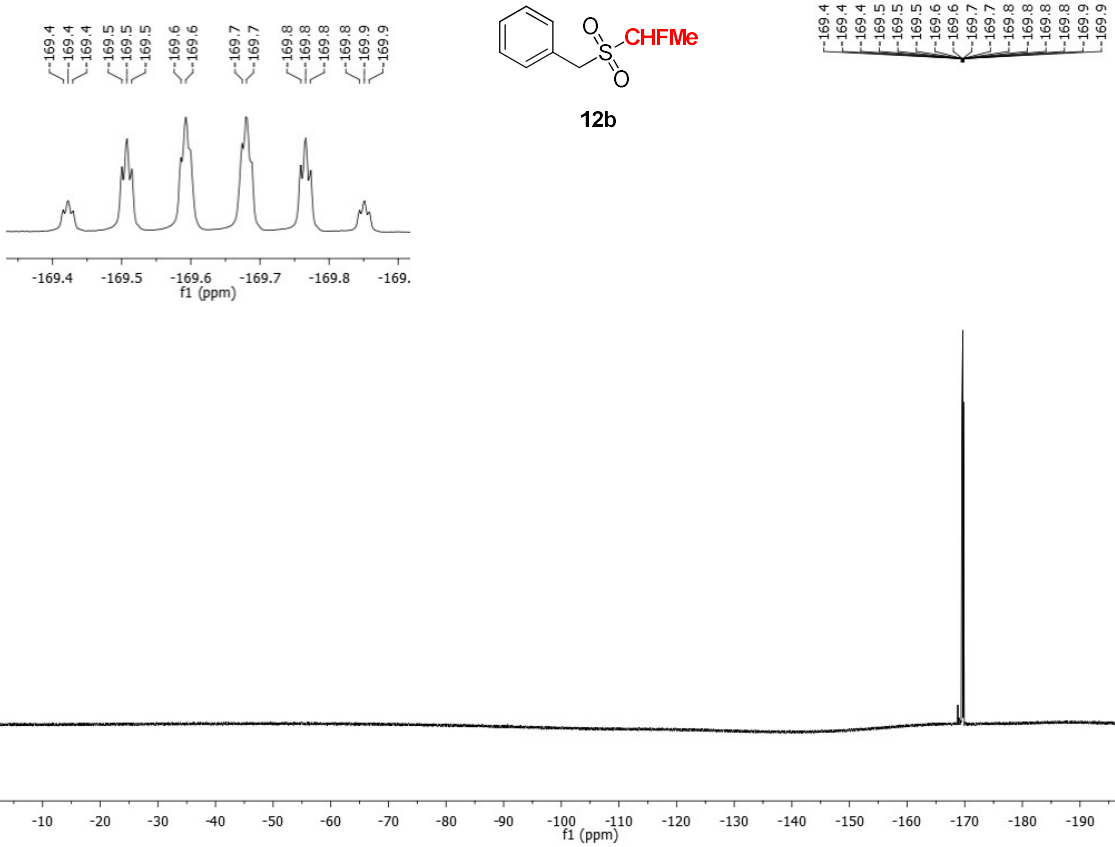
12b

7.42
5.27
5.25
5.23
5.21
5.11
5.09
5.07
5.04
4.49
4.48
4.44
4.43
4.42
4.30
4.29
4.25
4.25
4.23
1.71
1.69
1.63
1.61
4.93
0.94
1.04
1.05
3.00
f1 (ppm)


CHFMe

12b

130.8
129.2
129.1
126.6
97.0
94.1
55.8
11.7
11.4
f1 (ppm)


-169.4
-169.4
-169.4
-169.5
-169.5
-169.5
-169.6
-169.6
-169.7
-169.7
-169.8
-169.8
-169.8
-169.8
-169.9
-169.9
CHFMe
O
S
O
12b
-169.4
-169.5
-169.6
-169.7
-169.8
f1 (ppm)
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
f1 (ppm)