Supplementary Materials for

Room-Temperature Enantioselective C–H Iodination via Kinetic Resolution

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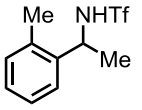
General Information:

Unless otherwise noted all commercial materials were used without further purification. Solvents were obtained from Acros or Sigma-Aldrich and used directly without further purification. Nuclear magnetic resonance (NMR) spectra were recorded with Varian Inova-400, Bruker DRX-600. ^{1}H and ^{13}C chemical shifts are reported in ppm downfield of tetramethylsilane and referenced to residual solvent peak ($CHCl_3$ = 7.26) unless otherwise noted. Multiplicities are reported using the following abbreviations: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad resonance. High resolution mass spectra for new compounds were recorded on an Agilent LC/MSD TOF mass spectrometer or an Agilent 6230 APCI-TOF mass spectrometer. Enantiomeric excesses (ee) were determined on a Hitachi LaChrow Elite HPLC system using commercially available chiral columns. All amino acids were purchased from Bachem or Sigma-Aldrich or synthesized according to literature procedures. All the substrates were synthesized according to the literature procedures (*31*).

Substrates:



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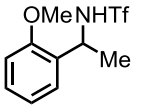

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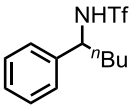

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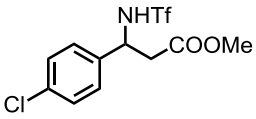

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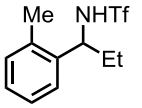

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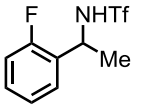

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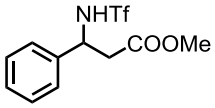

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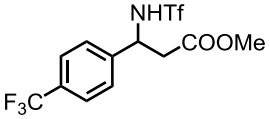

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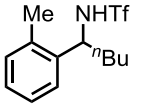

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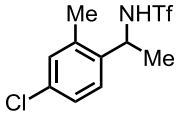

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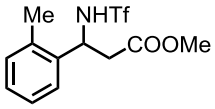

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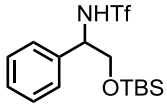

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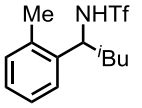

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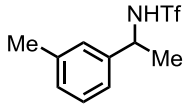

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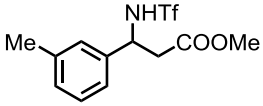

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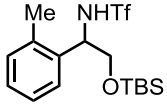

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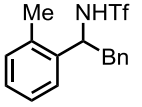

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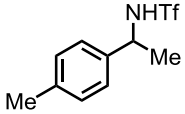

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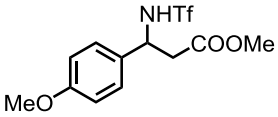

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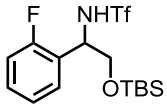

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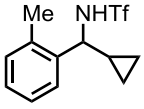

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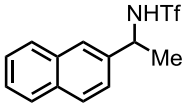

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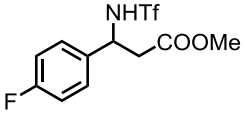

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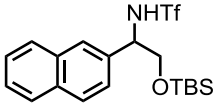


7d

Experimental Procedures and Characterization of Compounds:

Procedures for the Preparation of Substrates

General procedure for preparation of substrates 1b, 1h, 1k:

To a stirred solution of the corresponding amine (5 mmol, 1.0 equiv.) in dichloromethane (20 mL) was added triethylamine (5 mmol, 1.0 equiv.) at -78 °C under nitrogen. After stirring for 10 min at -78 °C, trifluoromethanesulfonic anhydride (5.3 mmol, 1.05 equiv.) was added dropwise and the mixture was stirred for 1 h at that temperature before being quenched by water (20 mL). The organic layer was separated and the aqueous layer was extracted with dichloromethane (10 mL × 2). The combined organic phase was washed with brine (20 mL), and then dried over $MgSO_4$. Evaporation and column chromatography on silica gel (EtOAc/hexane = 1:20 as eluent) afforded corresponding trifluoromethanesulfonamide **1b, 1h, 1k**.

General procedure for preparation of substrates 1a, 1c, 1d, 1e, 1f, 1g, 1i,1j, 1l (*31*)

To a 100 mL round bottle was added the corresponding ketone, $Ti(O^iPr)_4$ (2 equiv.) and a 2 M solution of NH_3 in EtOH (5 equiv.) under nitrogen. The resulting solution was heated to 50 ^oC and stirred for 24 h. Then the mixture was cooled to room temperature and $NaBH_4$ was added and the reaction mixture was stirred at room temperature for 6 h. The reaction was quenched with NH_4Cl solution and filtered through a pad of Celite. The resulting liquid was then extracted with EtOAc and the aqueous layer was washed twice with EtOAc. The organic layers were combined and extracted with 1 M HCl. The aqueous layer from this extraction was washed once with Et_2O, and then treated with 2 M NaOH until a pH of 12 was attained, and was then extracted thrice with EtOAc. The combined organic layer was washed with sat. aq. NaCl and concentrated under reduced pressure to give the crude amine, which could be used directly into the next step without purification. And the corresponding trifluoromethanesulfonamides **1a, 1c, 1d, 1e, 1f, 1g, 1i, 1j, 1l** could be synthesized using the same protocol shown above.

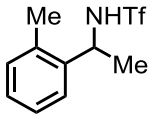
General procedure for preparation of substrates 4a-4g:

To a 100 mL round bottle was added the corresponding β-amino acid and methanol. The mixture was cooled to 0 ^oC and $SOCl_2$ (1.5 equiv.) was added dropwise. The solution was then stirred at room temperature for 12 h. After completion, the solution was concentrated under reduced pressure to give the crude ester. The corresponding trifluoromethanesulfonamides **4a-4g** could be synthesized using the same protocol shown above.

General procedure for preparation of substrates 7a-7d:

The amino alcohol was converted to the corresponding triflate amide according to the protocol shown above.The alcohol was protected as the TBS ether according to the protocol shown below.

To a 100 mL round bottle was added the corresponding alcohol, TBS-Cl (1.1 equiv.), DMAP (0.1 equiv.) and DCM. The mixture was cooled to 0 ^{o}C and Et_3N (1.1 equiv.) was added dropwise. The solution was warmed to room temperature and stirred for 12 h. Then the reaction was quenched with water. The organic layer was separated and the aqueous layer was washed twice with DCM. The combined organic layer was washed with sat. aq. NaCl and concentrated under reduced pressure to give the crude product, which was purified by column chromatography on silica gel (ethyl acetate/hexane= 1:30 as eluent) to afford **7a-7d**.

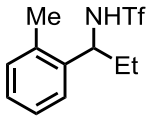


1a 1,1,1-Trifluoro-*N*-(1-(*o*-tolyl)ethyl)methanesulfonamide

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.32 – 7.13 (m, 4H), 5.65 (br s, 1H), 5.03 (q, *J* = 6.8 Hz, 1H), 2.37 (s, 3H), 1.55 (d, *J* = 6.8 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 139.63, 134.28, 130.86, 127.98, 126.77, 124.57, 119.41 (q, *J* = 321.1 Hz) 51.56, 23.42, 18.88.

HRMS (ESI-TOF) *m/z* Calcd for $C_{10}H_{11}F_3NO_2S^-$ $[M-H]^-$ 266.0468, found 266.0469.

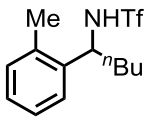


1b 1,1,1-trifluoro-*N*-(1-(o-tolyl)propyl)methanesulfonamide

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.27 – 7.13 (m, 4H), 5.73 (s, 1H), 4.78 (m, 1H), 2.36 (s, 3H), 1.95 – 1.77 (m, 2H), 0.93 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 138.79, 134.65, 130.71, 127.76, 126.69, 124.78, 119.38 (q, *J* = 321.04 Hz), 57.14, 30.75, 19.04, 10.39.

HRMS (ESI-TOF) *m/z* Calcd for $C_{11}H_{13}F_3NO_2S^-$ $[M-H]^-$ 280.0625, found 280.0622.



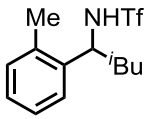
1c 1,1,1-trifluoro-*N*-(1-(o-tolyl)pentyl)methanesulfonamide

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.73 – 6.88 (m, 4H), 4.84 (t, *J* = 7.3 Hz, 1H), 2.36 (s, 3H), 1.90 – 1.72 (m, 2H), 1.47

– 1.18 (m, 4H), 0.88 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 139.16, 134.48, 130.70, 127.71, 126.70, 124.79, 119.36 (q, *J* = 321.0 Hz), 55.72, 37.46, 27.93, 22.17, 19.02, 13.75.

HRMS (ESI-TOF) *m/z* Calcd for $C_{13}H_{18}F_3NO_2SNa^+$ $[MNa]^+$ 332.0902, found 332.0899.

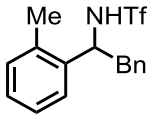


1d 1,1,1-trifluoro-*N*-(3-methyl-1-(o-tolyl)butyl)methanesulfonamide

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.27 – 7.13 (m, 4H), 5.46 (d, *J* = 7.6 Hz, 1H), 4.98 – 4.90 (m, 1H), 2.37 (s, 3H), 1.75 – 1.63 (m, 2H), 1.60 – 1.50 (m, 1H), 0.98 (d, *J* = 6.4 Hz, 3H), 0.94 (d, *J* = 6.5 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 139.31, 134.28, 130.83, 127.76, 126.73, 124.71, 119.30 (q, *J* = 320.9 Hz), 54.06, 47.04, 24.78, 22.80, 21.72, 18.94.

HRMS (ESI-TOF) *m/z* Calcd for $C_{13}H_{18}F_3NO_2SNa^+$ $[MNa]^+$ 322.0902, found 322.0899.

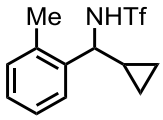


1e 1,1,1-trifluoro-*N*-(2-phenyl-1-(*o*-tolyl)ethyl)methanesulfonamide

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.28 – 7.17 (m, 5H), 7.13 (d, *J* = 7.6 Hz, 1H), 7.10 (d, *J* = 7.4 Hz, 1H), 7.01 – 6.94 (m, 2H), 5.36 (s, 1H), 5.11 (m, 1H), 3.16 – 3.07 (m, 2H), 2.16 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 137.96, 135.28, 134.77, 130.69, 129.49, 128.58, 127.99, 127.25, 126.55, 125.17, 119.28 (q, *J* = 321.2 Hz), 56.53, 43.94, 18.87.

HRMS (ESI-TOF) *m/z* Calcd for $C_{16}H_{16}F_3NO_2SNa^+$ $[MNa]^+$ 366.0746, found 366.0745.

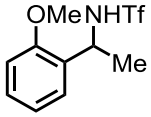


1f *N*-(cyclopropyl(*o*-tolyl)methyl)-1,1,1-trifluoromethanesulfonamide

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.29 – 7.18 (m, 3H), 7.19 – 7.14 (m, 1H), 5.56 (s, 1H), 4.52 (dd, *J* = 7.9 Hz, 7.9 Hz, 1H), 2.35 (s, 3H), 1.41 – 1.30 (m, 1H), 0.73 – 0.64 (m, 1H), 0.58 – 0.50 (m, 1H), 0.37 – 0.26 (m, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 137.26, 134.89, 130.69, 127.96, 126.41, 125.95, 119.41 (q, *J* = 321.0 Hz), 59.19, 19.25, 17.20, 4.48, 2.70.

HRMS (ESI-TOF) *m/z* Calcd for $C_{12}H_{14}F_3NO_2SNa^+$ $[MNa]^+$ 316.0589, found 316.0587.

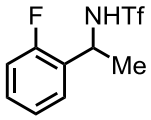


1g 1,1,1-trifluoro-*N*-(1-(2-methoxyphenyl)ethyl)methanesulfonamide

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.30 (t, *J* = 7.6 Hz, 1H), 7.13 (d, *J* = 7.6 Hz, 1H), 6.94 (m, 2H), 6.13 (d, *J* = 8.8 Hz, 1H), 4.74 (m, 1H), 3.90 (s, 3H), 1.61 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 156.56, 129.41, 128.70, 128.08, 119.47 (q, *J* = 321.1 Hz), 116.28, 111.28, 55.40, 55.14, 22.95.

HRMS (ESI-TOF) *m/z* Calcd for $C_{10}H_{12}F_3NO_3SNa^+$ $[MNa]^+$ 306.0382, found 306.0384.

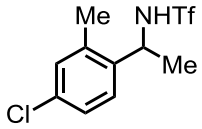


1h 1,1,1-trifluoro-*N*-(1-(2-fluorophenyl)ethyl)methanesulfonamide

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.35 – 7.28 (m, 1H), 7.26 (dd, *J* = 7.1, 6.9 Hz, 1H), 7.14 (d, *J* = 7.1, 7.1 Hz, 1H), 7.12 – 7.00 (m, 1H), 5.78 (br s, 1H), 4.92 (q, *J* = 7.1 Hz, 1H), 1.63 (d, *J* = 7.1 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 160.09 (d, *J* = 246.1 Hz), 129.97 (d, *J* = 8.5 Hz), 128.37 (d, *J* = 12.8 Hz), 127.68 (d, *J* = 4.1 Hz), 124.66 (d, *J* = 3.1 Hz), 119.39 (q, *J* = 320.8 Hz), 116.09 (d, *J* = 21.5 Hz), 51.83 (d, *J* = 1.8 Hz), 23.04 (d, *J* = 2.0 Hz).

HRMS (ESI-TOF) *m/z* Calcd for $C_9H_8F_4NO_2SNa^+$ $[MNa]^+$ 293.0104, found 293.0104.

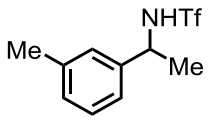


1i *N*-(1-(4-chloro-2-methylphenyl)ethyl)-1,1,1-trifluoromethanesulfonamide

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.20 (s, 2H), 7.17 (s, 1H), 5.75 (s, 1H), 5.02 – 4.95 (m, 1H), 2.35 (s, 3H), 1.53 (d, *J* = 6.9 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 138.31, 136.22, 133.47, 130.64, 126.85, 126.11, 119.35 (q, *J* = 321.1 Hz), 51.11, 23.32, 18.73.

HRMS (ESI-TOF) *m/z* Calcd for $C_{10}H_{10}ClF_3NO_2S^-$ $[M-H]^-$ 300.0078, found 300.0080.

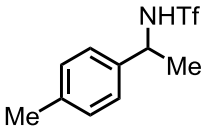


1j 1,1,1-trifluoro-*N*-(1-(*m*-tolyl)ethyl)methanesulfonamide

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.27 (dd, *J* = 7.5, 7.5 Hz, 1H), 7.18 – 7.07 (m, 3H), 5.63 – 5.37 (br s, 1H), 4.80 – 4.71 (m, 1H), 2.37 (s, 3H), 1.61 (d, *J* = 6.9 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 140.91, 138.76, 129.03, 128.88, 126.54, 122.81, 119.49 (q, *J* = 321.0 Hz), 55.32, 23.47, 21.37.

HRMS (ESI-TOF) *m/z* Calcd for $C_{10}H_{12}F_3NO_2SNa^+$ $[MNa]^+$ 290.0433, found 290.0431.

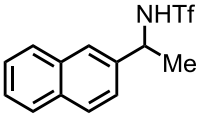


1k 1,1,1-trifluoro-*N*-(1-(*p*-tolyl)ethyl)methanesulfonamide

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.22 – 7.13 (m, 4H), 5.35 (s, 1H), 4.74 (m, 1H), 2.34 (s, 3H), 1.59 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 138.13, 137.99, 129.62, 125.75, 119.49 (q, *J* = 320.8 Hz), 55.14, 23.42, 21.01.

HRMS (ESI-TOF) *m/z* Calcd for $C_{10}H_{12}F_3NO_2SNa^+$ $[MNa]^+$ 290.0433, found 290.0435.

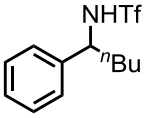


1l 1,1,1-trifluoro-*N*-(1-(naphthalen-2-yl)ethyl)methanesulfonamide

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.87 – 7.78 (m, 3H), 7.73 (s, 1H), 7.54 – 7.46 (m, 2H), 7.38 (d, *J* = 8.5 Hz, 1H), 5.43 (d, *J* = 7.5 Hz, 1H), 4.99 – 4.89 (m, 1H), 1.68 (d, *J* = 6.9 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 138.18, 133.14, 132.97, 129.05, 128.01, 127.68, 126.62, 126.49, 124.74, 123.61, 119.50 (q, *J* = 321.0 Hz), 55.42, 23.37.

HRMS (ESI-TOF) *m/z* Calcd for $C_{13}H_{12}F_3NO_2SNa^+$ $[MNa]^+$ 326.0433, found 326.0434.

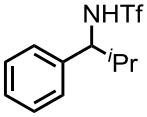


1m 1,1,1-trifluoro-*N*-(1-phenylpentyl)methanesulfonamide

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.41 – 7.35 (m, 2H), 7.35 – 7.30 (m, 1H), 7.27 – 7.20 (m, 2H), 5.30 (br s, 1H), 4.55 (ddd, *J* = 7.7, 7.7, 7.7 Hz, 1H), 2.00 – 1.81 (m, 2H), 1.39 – 1.27 (m, 3H), 1.27 – 1.15 (m, 1H), 0.87 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 140.26 , 128.97 , 128.24 , 126.09 , 119.39 (q, *J* = 321.1 Hz), 60.02 , 37.46 , 27.97 , 22.12 , 13.76 .

HRMS (ESI-TOF) *m/z* Calcd for $C_{12}H_{15}F_3NO_2S^-$ $[M-H]^-$ 294.0781, found 294.0783.

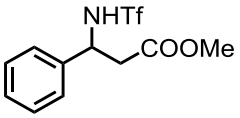


1n 1,1,1-trifluoro-N-(2-methyl-1-phenylpropyl)methanesulfonamide

1H NMR (600 MHz, CDCl3) δ 7.42 – 7.27 (m, 3H), 7.18 (d, J = 7.7 Hz, 2H), 5.54 (br, J = 37.2 Hz, 1H), 4.30 (d, J = 7.6 Hz, 1H), 2.10 – 1.96 (m, 1H), 1.02 (d, J = 6.7 Hz, 3H), 0.86 (d, J = 6.7 Hz, 3H).

13C NMR (151 MHz, CDCl3) δ 139.55, 128.68, 127.97, 126.38, 119.33 (q, J = 320.9 Hz), 77.21, 77.00, 76.79, 65.76, 34.63, 19.45, 18.49.

HRMS (ESI-TOF) *m/z* Calcd for $C_{11}H_{13}F_3NO_2S^-$ $[M-H]^-$ 280.0625, found 280.0626.

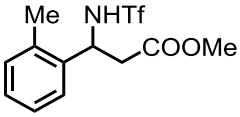


4a methyl 3-phenyl-3-((trifluoromethyl)sulfonamido)propanoate

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.40 – 7.34 (m, 2H), 7.34 – 7.26 (m, 3H), 6.85 (s, 1H), 5.03 (m, 1H), 3.65 (s, 3H), 2.96 (d, *J* = 5.5 Hz, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 171.19, 138.70, 128.93, 128.38, 125.84, 119.36 (q, *J* = 320.9 Hz), 55.52, 52.25, 41.22.

HRMS (ESI-TOF) *m/z* Calcd for $C_{11}H_{12}F_3NO_4SNa^+$ $[MNa]^+$ 334.0331, found 334.0326.

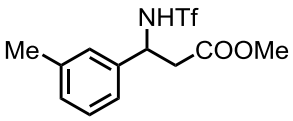


4b methyl 3-(*o*-tolyl)-3-((trifluoromethyl)sulfonamido)propanoate

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.32 (d, *J* = 7.2 Hz, 1H), 7.29 – 7.20 (m, 2H), 7.17 (d, *J* = 7.0 Hz, 1H), 6.81 (s, 1H), 5.27 (dd, *J* = 6.7 Hz, 5.7 Hz, 1H), 3.67 (s, 3H), 2.91 (dd, *J* = 15.8, 5.7 Hz, 1H), 2.84 (dd, *J* = 15.8, 6.7 Hz, 1H), 2.39 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 171.08, 137.23, 134.25, 130.91, 128.28, 126.73, 125.16, 119.32 (q, *J* = 320.8 Hz), 52.28, 52.04, 40.74, 18.88.

HRMS (ESI-TOF) *m/z* Calcd for $C_{12}H_{14}F_3NO_4SNa^+$ $[MNa]^+$ 348.0488, found 348.0488.

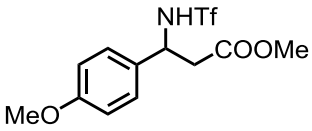


4c methyl 3-(*m*-tolyl)-3-((trifluoromethyl)sulfonamido)propanoate

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.25 (t, *J* = 7.5 Hz, 1H), 7.16 – 7.06 (m, 3H), 6.69 (br s, 1H), 4.98 (m, 1H), 3.66 (s, 3H), 3.01 – 2.91 (m, 2H), 2.35 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 171.21, 138.73, 129.15, 128.83, 126.57, 122.80, 119.41 (q, *J* = 320.7 Hz), 55.45, 52.22, 41.11, 21.41.

HRMS (ESI-TOF) *m/z* Calcd for $C_{12}H_{14}F_3NO_4SNa^+$ $[MNa]^+$ 348.0488, found 348.0490.

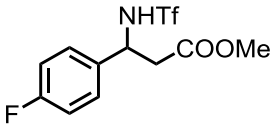


4d methyl 3-(4-methoxyphenyl)-3-((trifluoromethyl)sulfonamido)propanoate

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.23 (d, *J* = 7.6 Hz, 2H), 6.88 (d, *J* = 7.6 Hz, 2H), 6.67 (s, 1H), 4.98 (m, 1H), 3.79 (s, 3H), 3.66 (s, 3H), 2.95 (s, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 171.20, 159.45, 130.74, 127.18, 119.39 (q, *J* = 320.8, 320.2 Hz), 114.26, 55.23, 55.05, 52.22, 41.15.

HRMS (ESI-TOF) *m/z* Calcd for $C_{12}H_{14}F_3NO_5SNa^+$ $[MNa]^+$ 364.0437, found 364.0434.

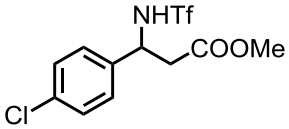


4e methyl 3-(4-fluorophenyl)-3-((trifluoromethyl)sulfonamido)propanoate

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.40 – 7.24 (m, 2H), 7.16 – 6.99 (m, 2H), 6.79 (s, 1H), 5.16 – 4.94 (m, 1H), 3.67 (s, 3H), 3.04 – 2.87 (m, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 171.17 , 162.46 (d, *J* = 247.8 Hz), 134.58 (d, *J* = 3.3 Hz), 127.74 (d, *J* = 8.4 Hz), 119.41 (q, *J* = 320.8 Hz), 115.93 (d, *J* = 21.8 Hz), 54.79 , 52.35 , 40.97 .

HRMS (ESI-TOF) *m/z* Calcd for $C_{11}H_{11}F_4NO_4SNa^+$ $[MNa]^+$ 352.0237, found 352.0237.

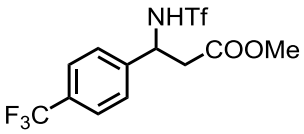


4f methyl 3-(4-chlorophenyl)-3-((trifluoromethyl)sulfonamido)propanoate

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.35 (d, *J* = 8.2 Hz, 2H), 7.26 (d, *J* = 8.2 Hz, 2H), 6.82 (br s, 1H), 4.99 (t, *J* = 4.8 Hz, 1H), 3.67 (s, 3H), 3.02 – 2.86 (m, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 171.12, 137.22, 134.34, 129.16, 127.31, 119.34 (q, *J* = 320.6 Hz), 54.76, 52.38, 40.78.

HRMS (ESI-TOF) *m/z* Calcd for $C_{11}H_{11}ClF_3NO_4SNa^+$ $[MNa]^+$ 367.9942, found 367.9943.



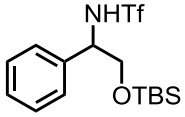
4g methyl 3-(4-trifluoromethylphenyl)-3-((trifluoromethyl)sulfonamide)propanoate

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.65 (d, *J* = 8.4 Hz, 2H), 7.46 (d, *J* = 8.4 Hz, 2H), 6.83 (s, 1H), 5.06 (dd, *J* = 5.3, 4.8 Hz, 1H), 3.67 (s, 3H), 3.05 (dd, *J* = 16.7, 5.3 Hz, 1H), 2.97 (dd, *J* = 16.7, 4.8 Hz, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 171.12 , 142.55 , 130.69 (q, *J* = 32.8 Hz), 126.35 , 126.01 (q, *J* = 3.6 Hz), 123.75 (q,

J = 272.2 Hz), 119.37 (q, *J* = 320.5 Hz), 54.72 , 52.46 , 40.33 .

HRMS (ESI-TOF) *m/z* Calcd for $C_{12}H_{11}F_6NO_4SNa^+$ $[MNa]^+$ 402.0205, found 402.0206.

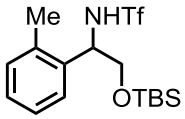


7a *N*-(2-((*tert*-butyldimethylsilyl)oxy)-1-phenylethyl)-1,1,1-trifluoromethanesulfonamide

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.40 – 7.35 (m, 2H), 7.35 – 7.29 (m, 3H), 5.96 (br s, 1H), 4.73 (m, 1H), 3.97 (dd, *J* = 10.4, 4.1 Hz, 1H), 3.80 (dd, *J* = 10.4, 4.7 Hz, 1H), 0.90 – 0.79 (m, 9H), -0.01 – -0.10 (m, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 138.01, 128.56, 128.19, 126.56, 119.50 (q, *J* = 320.9 Hz), 66.71, 60.12, 25.66, 18.20, -5.78, -5.87.

HRMS (ESI-TOF) *m/z* Calcd for $C_{15}H_{25}F_3NO_3SSi^+$ $[MH]^+$ 384.1271, found 384.1270.

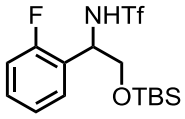


7b *N*-(2-((tert-butyldimethylsilyl)oxy)-1-(o-tolyl)ethyl)-1,1,1-trifluoromethanesulfonamide

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.32 – 7.24 (m, 1H), 7.24 – 7.18 (m, 2H), 7.18 – 7.13 (m, 1H), 5.80 (s, 1H), 5.02 – 4.90 (m, 1H), 3.88 (dd, *J* = 10.5, 4.4 Hz, 1H), 3.67 (dd, *J* = 10.5, 5.5 Hz, 1H), 2.35 (s, 3H), 0.85 (s, 10H), -0.03 – -0.09 (m, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 135.95, 134.44, 130.61, 128.02, 126.33, 126.21, 119.46 (q, *J* = 321.2 Hz), 65.61, 56.40, 25.70, 19.07, 18.23, -5.70, -5.85.

HRMS (ESI-TOF) *m/z* Calcd for $C_{16}H_{27}F_3NO_3SSi^+$ $[MH]^+$ 398.1427, found 398.1428.

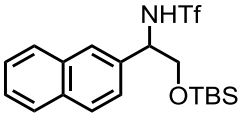


7c *N*-(2-((*tert*-butyldimethylsilyl)oxy)-1-(2-fluorophenyl)ethyl)-1,1,1-trifluoromethanesulfonamide

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.42 – 7.30 (m, 2H), 7.18 (t, *J* = 7.5 Hz, 1H), 7.13 – 7.06 (m, 1H), 6.08 (d, *J* = 8.3 Hz, 1H), 5.04 (ddd, *J* = 8.3, 4.7 Hz, 4.4 Hz, 1H), 3.99 (dd, *J* = 10.4, 4.4 Hz, 1H), 3.86 (dd, *J* = 10.4, 4.7 Hz, 1H), 0.86 (s, 9H), -0.03 (s, 3H), -0.04 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 159.77 (d, *J* = 245.7 Hz), 129.87 (d, *J* = 8.5 Hz), 128.64 (d, *J* = 3.6 Hz), 125.19 (d, *J* = 13.0 Hz), 124.23 (d, *J* = 3.3 Hz), 119.47 (q, *J* = 321.0 Hz), 115.58 (d, *J* = 21.8 Hz), 65.60 , 55.35 , 25.60 , 18.15 , -5.89 , -5.97.

HRMS (ESI-TOF) *m/z* Calcd for $C_{15}H_{24}F_4NO_3SSi^+$ $[MH]^+$ 402.1177, found 402.1179.



7d *N*-(2-((*tert*-butyldimethylsilyl)oxy)-1-(naphthalen-2-yl)ethyl)-1,1,1-trifluoromethanesulfonamide

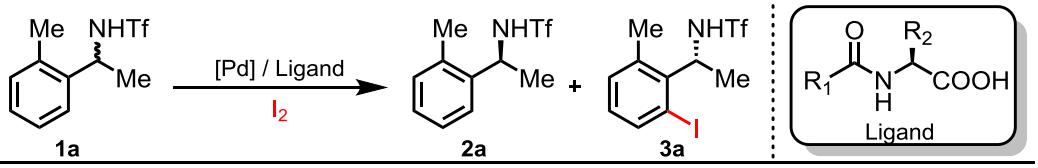
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.87 – 7.79 (m, 3H), 7.77 (s, 1H), 7.54 – 7.46 (m, 2H), 7.40 (d, *J* = 8.5 Hz, 1H), 5.86 (d, *J* = 7.4 Hz, 1H), 4.93 – 4.83 (m, 1H), 4.03 (dd, *J* = 10.4, 3.8 Hz, 1H), 3.87 (dd, *J* = 10.4, 4.3 Hz, 1H), 0.85 (s, 9H), -0.04 (s, 3H), -0.07 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 135.38, 133.05, 133.03, 128.52, 127.96, 127.67, 126.48, 126.35, 125.89, 124.20, 119.52 (q, *J* = 321.2 Hz), 66.59, 60.12, 25.69, 18.21, -5.68, -5.78.

HRMS (ESI-TOF) *m/z* Calcd for $C_{19}H_{27}F_3NO_3SSi^+$ $[MH]^+$ 434.1427, found 434.1424.

Optimization of the Reaction Conditions

Table S1. Optimization of enantioselective C–H iodination

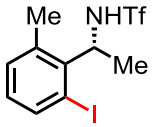


entry	ligand		conv. (%)*	ee†		s‡
	R_1	R_2		2a	3a	
1	C_6H_5	^{n}Pr	38	50	83	17.6
2	C_6H_5	^{n}Bu	48	77	83	24.8
3	**C_6H_5**	**^{i}Bu**	**49**	**86**	**88**	**50**
4	C_6H_5	neopentyl	33	31	63	5.9
5	C_6H_5	^{i}Pr	48	79	87	30.8
6	C_6H_5	^{t}Bu	25	23	70	6.8
7	2-$CF_3C_6H_4$	^{i}Bu	48	80	88	34.2
8	3,5-$(CF_3)_2C_6H_3$	^{i}Bu	45	39	47	4.1
9	4-$OMeC_6H_4$	^{i}Bu	57	90	73	15.5
10	4-FC_6H_4	^{i}Bu	46	70	83	21.2
11	1-naphthyl	^{i}Bu	39	50	81	13.3
12	2-naphthyl	^{i}Bu	47	77	88	32.9
13	Me	^{i}Bu	24	15	48	3.2
14	CF_3	^{i}Bu	5	1	21	1.5
15	^{t}BuO	^{i}Bu	38	24	39	2.9
16§	C_6H_5	^{i}Bu	50	89	90	62.0
17‖	**C_6H_5**	**^{i}Bu**	**50 (50¶)**	**92 (89¶)**	**92 (89¶)**	**78.8 (51.2¶)**
18#	C_6H_5	^{i}Bu	54	94.5	80	33.0

Reaction conditions: 10 mol% $Pd(OAc)_2$, 40 mol% Ligand, 3 eq. Na_2CO_3, 3 eq. CsOAc, 3 eq. I_2,15eq. DMSO, 1mL ^{t}amyl-OH, air, 20°C, 24h *Calculated conversion, c = ee_{2a} / (ee_{2a} + ee_{3a}).†Determined by chiral HPLC analysis. ‡Selectivity(s) = (rate of fast-reacting enantiomer) / (rate of slow-reacting enantiomer). §0.5 mL ^{t}amyl-OH. ‖0.5 mL ^{t}amyl-OH/ DMSO (5 : 2.2). ¶Reaction conditions: 2 mol% $Pd(OAc)_2$, 10 mol% Bz-Leu-OH, 3 equiv. CsOAc, 3 equiv. Na_2CO_3, 3 equiv. I_2, 0.5 mL ^{t}amyl-OH/DMSO (5 : 2.2), air, 20°C, 24h, then 3 equiv. I_2, 20°C, 24h. #20 mol% Ligand.

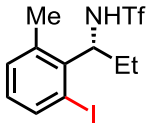
General Procedure for $Pd(OAc)_2$-Catalyzed C–H Iodination

To a 10 mL sealed tube was added substrates (1 equiv., 0.2 mmol), $Pd(OAc)_2$ (10 mol%, 0.02 mmol), Bz-Leu-OH (40 mol%, 0.08 mmol), CsOAc (3 equiv., 0.6 mmol), I_2 (3 equiv., 0.6 mmol), Na_2CO_3 (3 equiv., 0.6 mmol) and ^{t}amyl-OH / DMSO (1 mL, v / v = 5 : 2.2). The mixture was stirred at 20 ^{o}C for 24-48 h. The resulting mixture was diluted by EtOAc, filtered through a pad of celite and washed with $Na_2S_2O_3$. The organic layer was dried over $MgSO_4$, evaporated and purified by prep-TLC (EtOAc: hexanes= 1:5-1:15) to give the product and ee was determined on a Hitachi LaChrow HPLC system using commercially available chiral columns as described below.



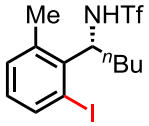
3a 1,1,1-trifluoro-*N*-(1-(2-iodo-6-methylphenyl)ethyl)methanesulfonamide

The compound **3a** was prepared according to the general procedure and was purified by prep-TLC to give a colorless oil in 48 % yield (37 mg). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.76 (d, *J* = 7.8 Hz, 1H), 7.18 (d, *J* = 7.3 Hz, 1H), 6.87 (t, *J* = 7.7 Hz, 1H), 6.52 (d, *J* = 8.7 Hz, 0.8H), 5.53 (s, 0.2H), 5.35 – 5.21 (m, 1H), 2.51 (s, 0.7H), 2.42 (s, 2.3H), 1.73 (d, *J* = 7.0 Hz, 2.3H), 1.62 (s, 0.7H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 140.11, 139.37, 138.10, 131.28, 129.50, 119.32 (q, *J* = 321.2 Hz), 51.86, 20.76, 20.51 . HPLC chiralcel AS-H column (5 % isopropanol in hexanes, 0.5 mL/min) t_r= 12.340 min (major), 16.660 min (minor): 91 % ee. HRMS (ESI-TOF) *m/z* Calcd for $C_{10}H_{10}F_3INO_2S^-$ $[M-H]^-$ 391.9435, found 391.9436.



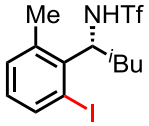
3b 1,1,1-trifluoro-*N*-(1-(2-iodo-6-methylphenyl)propyl)methanesulfonamide

The compound **3b** was prepared according to the general procedure and was purified by prep-TLC to give a colorless oil in 49% yield (40 mg). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.76 (d, *J* = 7.9 Hz, 1H), 7.18 (d, *J* = 7.5 Hz, 0.8H), 7.14 (d, *J* = 7.5 Hz, 0.2H), 6.91 – 6.83 (m, 1H), 6.44 (d, *J* = 9.7 Hz, 0.8H), 5.36 (m, 0.2H), 5.29 (m, 0.2H), 5.01 (m, 0.8H), 2.47 (s, 0.6H), 2.40 (s, 2.4H), 2.25 – 2.16 (m, 0.8H), 2.16 – 2.06 (m, 0.8H), 1.97 – 1.91 (m, 0.4H), 1.09 (t, *J* = 7.4 Hz, 0.6H), 0.99 (t, *J* = 7.4 Hz, 2.4H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 140.15, 139.45, 138.91, 138.54, 138.52, 135.76, 132.98, 131.23, 129.48, 129.36, 119.29 (q, *J* = 321.1 Hz), 101.96, 93.59, 66.79, 57.48, 27.84, 27.28, 21.08, 20.90, 11.02, 10.85. HPLC chiralcel AS-H column (5 % isopropanol in hexanes, 0.5 mL/min) t_r= 11.153 min (major), 13.567 min (minor): 89 % ee. HRMS (ESI-TOF) *m/z* Calcd for $C_{11}H_{13}F_3INaNO_2S^+$ $[MNa]^+$ 429.9556, found 429.9553.



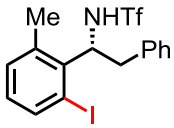
3c 1,1,1-trifluoro-*N*-(1-(2-iodo-6-methylphenyl)pentyl)methanesulfonamide

The compound **3c** was prepared according to the general procedure and was purified by prep-TLC to give a colorless oil in 45 % yield (39 mg). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.76 (d, *J* = 7.9 Hz, 1H), 7.18 (d, *J* = 7.5 Hz, 0.8H), 7.14 (d, *J* = 7.6 Hz, 0.2H), 6.90 – 6.82 (m, 1H), 6.43 (s, 0.8H), 5.38 – 5.32 (m, 0.2H), 5.08 (t, *J* = 7.4 Hz, 0.8H), 2.48 (s, 0.6H), 2.40 (s, 2.4H), 2.26 – 2.16 (m, 0.8H), 2.06 – 1.98 (m, 0.8H), 1.94 – 1.84 (m, 0.4H), 1.51 – 1.30 (m, 3H), 1.30 – 1.19 (m, 1H), 0.96 – 0.88 (m, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 140.14, 139.70, 138.86, 138.74, 138.56, 135.75, 132.99, 131.24, 129.44, 129.33, 119.26 (q, *J* = 321.1 Hz), 101.71, 93.66, 65.46, 56.03, 34.32, 33.78, 28.35, 28.25, 22.23, 22.20, 21.14, 20.87, 13.85, 13.84. HPLC chiralcel AS-H column (5 % isopropanol in hexanes, 0.3 mL/min) t_r= 17.227 min (major), 19.080 min (minor): 87 % ee. HRMS (ESI-TOF) *m/z* Calcd for $C_{13}H_{17}F_3INaNO_2S^+$ $[MNa]^+$ 457.9869, found 457.9868.



3d 1,1,1-trifluoro-*N*-(1-(2-iodo-6-methylphenyl)-3-methylbutyl)methanesulfonamide

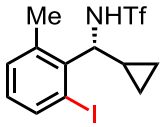
The compound **3d** was prepared according to the general procedure and was purified by prep-TLC to give a colorless oil in 42 % yield (37 mg). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.75 (d, *J* = 6.0 Hz, 1H), 7.20 – 7.10 (m, 1H), 6.86 (dd, *J* = 6.9, 6.9 Hz, 1H), 6.42 (d, *J* = 8.4 Hz, 0.8H), 5.42 (s, 0.2H), 5.38 (s, 0.2H), 5.17 (s, 0.8H), 2.48 (s, 0.6H), 2.40 (s, 2.4H), 1.98 – 1.67 (m, 2H), 1.57 – 1.49 (m, 1H), 1.01 (d, *J* = 5.4 Hz, 6H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 140.14, 139.89, 139.25, 138.61, 138.33, 135.91, 133.01, 131.27, 129.38, 129.30, 119.20 (q, *J* = 321.0 Hz), 101.07, 93.76, 63.95, 54.36, 43.23, 42.90, 25.01, 24.63, 23.52, 23.31, 21.37, 21.22, 20.74. HPLC chiralcel AS-H column (5 % isopropanol in hexanes, 0.5 mL/min) t_r= 8.427 min (major), 9.567 min (minor): 81 % ee. HRMS (ESI-TOF) *m/z* Calcd for $C_{13}H_{17}F_3INaNO_2S^+$ $[MNa]^+$ 457.9869, found 457.9870.



3e 1,1,1-trifluoro-*N*-(1-(2-iodo-6-methylphenyl)-2-phenylethyl)methanesulfonamide

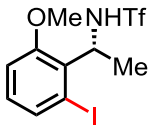
The compound **3e** was prepared according to the general procedure and was purified by prep-TLC to give a colorless oil in 44 % yield (41 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.81 (d, *J* = 7.8 Hz, 0.8H), 7.76 (d, *J* = 7.4 Hz, 0.2H), 7.39 – 7.14 (m, 4H), 7.04 (d, *J* = 8.1 Hz, 2H), 6.92 – 6.80 (m, 1H), 6.53 (d, *J* = 10.0 Hz, 0.8H), 5.65 – 5.57 (m, 0.2H), 5.32 – 5.24 (m, 0.8H), 5.22 (s, 0.2H), 3.58 (dd, *J* = 13.5, 9.1 Hz, 0.8H), 3.33 (dd, *J* = 13.5, 7.1 Hz, 0.8H), 3.25 (dd, *J* = 14.2, 5.5 Hz, 0.2H), 3.03 (dd, *J* = 14.2, 10.3 Hz, 0.2H), 2.50 (s, 0.4H), 2.01 (s, 2.6H). ^{13}C NMR

(151 MHz, $CDCl_3$) δ 140.06, 139.58, 138.66, 138.54, 137.49, 136.21, 135.86, 135.27, 133.12, 131.07, 129.71, 129.54, 129.33, 129.23, 128.99, 128.54, 127.65, 127.20, 119.20 (q, *J* = 321.2 Hz), 101.52, 94.12, 66.47, 57.22, 40.45, 40.24, 21.45, 20.48. HPLC chiralcel AS-H column (5% isopropanol in hexanes, 0.5 mL/min) t_r= 14.480 min (major), 18.213 min (minor): 75 % ee. HRMS (ESI-TOF) *m/z* Calcd for $C_{16}H_{15}F_3INaNO_2S^+$ $[MNa]^+$ 491.9713, found 491.9708.



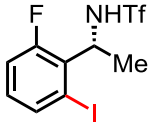
3f *N*-(cyclopropyl(2-iodo-6-methylphenyl)methyl)-1,1,1-trifluoromethanesulfonamide

The compound **3f** was prepared according to the general procedure and was purified by prep-TLC to give a colorless oil in 42 % yield (35 mg). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.80 (d, *J* = 7.8 Hz, 0.7H), 7.74 (d, *J* = 7.9 Hz, 0.3H), 7.18 (d, *J* = 7.6 Hz, 0.7H), 7.16 (d, *J* = 7.5 Hz, 0.3H), 6.88 (dd, *J* = 7.8, 7.7 Hz, 0.7H), 6.84 (dd, *J* = 7.8, 7.7 Hz, 0.3H), 4.64 (d, *J* = 9.3 Hz, 0.3H), 4.29 (d, *J* = 9.7 Hz, 0.7H), 2.56 (s, 1H), 2.35 (s, 2H), 2.05 – 1.96 (m, 0.7H), 1.46 – 1.39 (m, 0.3H), 0.85 – 0.41 (m, 4H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 140.13, 139.41, 138.32, 138.21, 136.75, 132.87, 131.30, 129.40, 129.19, 119.39 (q, *J* = 321.2 Hz), 102.12, 94.49, 68.88, 60.76, 21.02, 20.98, 16.58, 15.69, 5.90, 5.26, 5.14, 4.79. HPLC chiralcel AS-H column (5 % isopropanol in hexanes, 0.5 mL/min) t_r= 13.733 min (major), 19.760 min (minor): 95 % ee. HRMS (ESI-TOF) *m/z* Calcd for $C_{12}H_{13}F_3INaNO_2S^+$ $[MNa]^+$ 441.9556, found 441.9556.



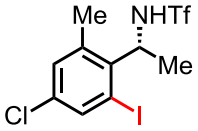
3g 1,1,1-trifluoro-*N*-(1-(2-iodo-6-methoxyphenyl)ethyl)methanesulfonamide

The compound **3g** was prepared according to the general procedure and was purified by prep-TLC to give a colorless oil in 46 % yield (38 mg). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.49 (d, *J* = 7.6 Hz, 1H), 7.00 – 6.90 (m, 2H), 6.51 (d, *J* = 9.7 Hz, 1H), 5.27 – 5.18 (m, 1H), 3.92 (s, 3H), 1.58 – 1.53 (m, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 156.69, 132.69, 131.31, 130.28, 119.38 (q, *J* = 321.1 Hz), 111.82, 99.22, 60.27, 55.94, 21.36. HPLC chiralcel AS-H column (5 % isopropanol in hexanes, 0.8 mL/min) t_r= 9.387 min (major), 21.127 min (minor): 97 % ee. HRMS (ESI-TOF) *m/z* Calcd for $C_{10}H_{11}F_3INaNO_3S^+$ $[MNa]^+$ 431.9349, found 431.9351.



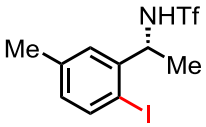
3h 1,1,1-trifluoro-*N*-(1-(2-fluoro-6-iodophenyl)ethyl)methanesulfonamide

The compound **3h** was prepared according to the general procedure and was purified by prep-TLC to give a colorless oil in 47 % yield (37 mg). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.67 (d, *J* = 7.9 Hz, 1H), 7.21 – 7.08 (m, 1H), 7.08 – 6.98 (m, 1H), 5.77 (s, 1H), 5.37 – 5.17 (m, 1H), 1.61 (d, *J* = 7.0 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 160.13 (d, *J* = 250.5 Hz), 136.00 (d, J = 2.2 Hz), 131.45 (d, *J* = 15.3 Hz), 130.97 (d, *J* = 9.8 Hz), 119.30 (q, *J* = 320.7 Hz), 116.77 (d, *J* = 23.0 Hz), 99.57 , 58.93 , 21.80 . HPLC chiralcel AS-H column (5 % isopropanol in hexanes, 0.8 mL/min) t_r= 8.447 min (major), 12.593 min (minor): 96 % ee. HRMS (ESI-TOF) *m/z* Calcd for $C_9H_7F_4INO_2S^-$ $[M-H]^-$ 395.9184, found 395.9189.



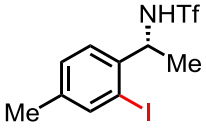
3i *N*-(1-(4-chloro-2-iodo-6-methylphenyl)ethyl)-1,1,1-trifluoromethanesulfonamide

The compound **3i** was prepared according to the general procedure and was purified by prep-TLC to give a colorless oil in 49 % yield (42 mg). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.78 (s, 1H), 7.19 (s, 1H), 6.39 (d, *J* = 8.2 Hz, 1H), 5.33 – 5.19 (m, 1H), 2.59 – 2.27 (m, 3H), 1.82 – 1.57 (m, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 139.18, 138.49, 138.07, 136.35, 133.99, 133.49, 131.14, 130.70, 126.84, 126.20, 119.43 (q, *J* = 321.1 Hz), 99.57, 93.16, 51.46, 51.00, 40.72, 23.43, 20.70, 20.40, 18.84. HPLC chiralcel AS-H column (5 % isopropanol in hexanes, 0.5 mL/min) t_r= 12.047 min (major), 15.640 min (minor): 93 % ee. HRMS (ESI-TOF) *m/z* Calcd for $C_{10}H_9ClF_3INO_2S^-$ $[M-H]^-$ 425.9045, found 425.9047.



3j 1,1,1-trifluoro-*N*-(1-(2-iodo-5-methylphenyl)ethyl)methanesulfonamide

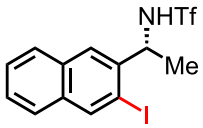
The compound **3j** was prepared according to the general procedure and was purified by prep-TLC to give a colorless oil in 34 % yield (27 mg). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.69 (d, *J* = 8.0 Hz, 1H), 7.08 (s, 1H), 6.83 (d, *J* = 9.4 Hz, 1H), 5.47 (s, 1H), 5.03 (m, 1H), 2.31 (s, 3H), 1.55 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 143.20, 139.91, 139.19, 130.84, 127.14, 119.36 (q, *J* = 321.3 Hz), 92.83, 59.07, 23.46, 21.03. HPLC chiralcel AS-H column (5 % isopropanol in hexanes, 0.8 mL/min) t_r= 7.967 min (major), 15.753 min (minor): 92 % ee. HRMS (ESI-TOF) *m/z* Calcd for $C_{10}H_{11}F_3INaNO_2S^+$ $[MNa]^+$ 415.9400, found 415.9392.



3k 1,1,1-trifluoro-*N*-(1-(2-iodo-4-methylphenyl)ethyl)methanesulfonamide

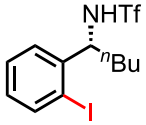
The compound **3k** was prepared according to the general procedure and was purified by prep-TLC to give

a colorless oil in 35 % yield (28 mg). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.69 (s, 1H), 7.20 (d, *J* = 7.9 Hz, 1H), 7.16 (d, *J* = 7.9 Hz, 1H), 5.05 (q, *J* = 6.8 Hz, 1H), 2.30 (s, 3H), 1.55 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 140.61, 140.56, 140.01, 129.83, 125.95, 119.36 (q, *J* = 321.2 Hz), 96.92, 58.93, 23.55, 20.42. HPLC chiralcel AS-H column (10 % isopropanol in hexanes, 0.3 mL/min) t_r= 17.080 min (major), 41.727 min (minor): 95 % ee. HRMS (ESI-TOF) *m/z* Calcd for $C_{10}H_{11}F_3INaNO_2S^+$ $[MNa]^+$ 415.9400, found 415.9392.



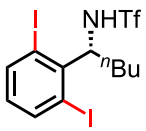
3l 1,1,1-trifluoro-*N*-(1-(3-iodonaphthalen-2-yl)ethyl)methanesulfonamide

The compound **3l** was prepared according to the general procedure and was purified by prep-TLC to give a colorless oil in 37 % yield (32 mg). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.42 (s, 1H), 7.83 – 7.70 (m, 3H), 7.57 – 7.49 (m, 2H), 5.57 (d, *J* = 6.9 Hz, 1H), 5.26 – 5.18 (m, 1H), 1.68 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 139.90, 139.86, 134.30, 132.72, 127.78, 127.29, 127.26, 126.51, 125.45, 119.38 (q, *J* = 321.1 Hz), 93.64, 58.97, 23.86. HPLC chiralcel AS-H column (5 % isopropanol in hexanes, 0.5 mL/min) t_r= 17.880 min (major), 36.760 min (minor): 95 % ee. HRMS (ESI-TOF) *m/z* Calcd for $C_{13}H_{11}F_3INaNO_2S^+$ $[MNa]^+$ 451.9400, found 451.9403.



3m-mono 1,1,1-trifluoro-*N*-(1-(2-iodophenyl)pentyl)methanesulfonamide

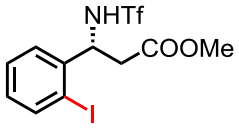
The compound **3m-mono** was prepared according to the general procedure and was purified by prep-TLC to give a colorless oil in 17 % yield (14 mg). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.85 (dd, *J* = 7.9, 1.2 Hz, 1H), 7.38 (td, *J* = 7.5, 1.2 Hz, 1H), 7.21 (dd, *J* = 7.8, 1.6 Hz, 1H), 7.05 – 6.95 (m, 1H), 5.45 (s, 1H), 4.91 (ddd, *J* = 8.5 Hz, 1H), 1.93 – 1.82 (m, 1H), 1.78 – 1.67 (m, 1H), 1.51 – 1.25 (m, 4H), 0.92 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 142.98, 140.21, 129.66, 128.85, 126.71, 119.30 (q, *J* = 321.2 Hz), 63.15, 37.09, 27.92, 22.12, 13.82. HPLC chiralcel AS-H column (10 % isopropanol in hexanes, 0.5 mL/min) t_r= 10.893 min (major), 11.773 min (minor): 87 % ee. HRMS (ESI-TOF) *m/z* Calcd for $C_{12}H_{15}F_3INaNO_2S^+$ $[MNa]^+$ 443.9712, found 443.9711.



3m-di *N*-(1-(2,6-diiodophenyl)pentyl)-1,1,1-trifluoromethanesulfonamide

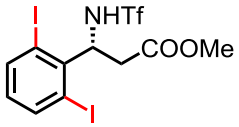
The compound **3m-di** was prepared according to the general procedure and was purified by prep-TLC to

give a colorless oil in 24 % yield (26 mg). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.06 – 7.84 (m, 2H), 6.64 (t, *J* = 7.9 Hz, 1H), 6.32 (br s, 1H), 5.33 (dd, *J* = 9.6, 6.2 Hz, 1H), 2.17 – 2.06 (m, 1H), 2.00 – 1.87 (m, 1H), 1.59 – 1.31 (m, 4H), 0.94 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 142.52, 141.25, 140.80, 130.87, 119.21 (q, *J* = 321.1 Hz), 102.69, 92.22, 65.79, 33.44, 28.05, 22.18, 13.84. HPLC chiralcel AS-H column (10 % isopropanol in hexanes, 0.5 mL/min) t_r= 10.400 min (major), 12.860 min (minor): 99 % ee. HRMS (ESI-TOF) *m/z* Calcd for $C_{12}H_{15}F_3INaNO_2S^+$ $[MNa]^+$ 569.8679, found 569.8680.



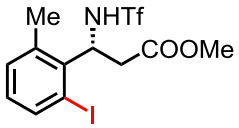
6a-mono methyl 3-(2-iodophenyl)-3-((trifluoromethyl)sulfonamido)propanoate

The compound **6a-mono** was prepared according to the general procedure and was purified by prep-TLC to give a colorless oil in 31 % yield (27 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (dd, *J* = 7.9, 1.1 Hz, 1H), 7.47 – 7.33 (m, 2H), 7.13 (d, *J* = 8.7 Hz, 1H), 7.03 (ddd, *J* = 7.9, 7.0, 2.1 Hz, 1H), 5.25 (dt, *J* = 9.5, 5.0 Hz, 1H), 3.66 (s, 3H), 2.98 (d, *J* = 5.0 Hz, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 171.22, 140.77, 140.08, 130.04, 128.78, 127.30, 119.32 (q, *J* = 322.1, 321.4 Hz), 97.19, 58.89, 52.32, 39.04. HPLC chiralcel AD-H column (10 % isopropanol in hexanes, 0.2 mL/min) t_r= 29.827 min (minor), 32.413 min (major): 96 % ee. HRMS (ESI-TOF) *m/z* Calcd for $C_{11}H_{12}F_3INO_4S^+$ $[MH]^+$ 437.9478, found 437.9477.



6a-di methyl 3-(2,6-diiodophenyl)-3-((trifluoromethyl)sulfonamido)propanoate

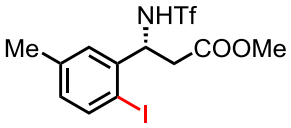
The compound **6a-di** was prepared according to the general procedure and was purified by prep-TLC to give a colorless oil in 16 % yield (18 mg). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.95 (m, 2H), 6.67 (t, *J* = 7.9 Hz, 1H), 6.52 (d, *J* = 9.1 Hz, 1H), 5.88 (ddd, *J* = 10.3, 9.1, 5.0 Hz, 1H), 3.78 (s, 3H), 3.27 (dd, *J* = 15.4, 10.3 Hz, 1H), 2.85 (dd, *J* = 15.4, 5.0 Hz, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 168.79, 142.54, 140.99, 139.67, 131.43, 119.13 (q, *J* = 321.4 Hz), 102.15, 92.52, 62.73, 52.45, 38.23. HRMS (ESI-TOF) *m/z* Calcd for $C_{11}H_9F_3I_2NO_4S^-$ $[M-H]^-$ 561.8299, found 561.8301.



6b methyl 3-(2-iodo-6-methylphenyl)-3-((trifluoromethyl)sulfonamido)propanoate

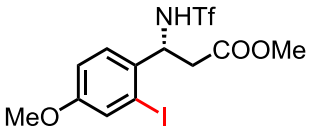
The compound **6b** was prepared according to the general procedure and was purified by prep-TLC to give

a colorless oil in 49 % yield (44 mg). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.76 (d, *J* = 7.6 Hz, 1H), 7.23 – 7.11 (m, 1H), 6.90 (dd, *J* = 7.6, 7.4 Hz, 1H), 5.75 (s, 0.2H), 5.69 – 5.62 (m, 0.8H), 3.77 (s, 0.6H), 3.71 (s, 2.4H), 3.33 (dd, *J* = 15.5, 8.8 Hz, 0.8H), 3.06 (dd, *J* = 15.5, 5.6 Hz, 0.8H), 2.91 (s, 0.2H), 2.80 (s, 0.2H), 2.51 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 176.61, 169.61, 140.04, 139.36, 138.49, 137.55, 133.14, 131.51, 130.05, 129.66, 119.20 (q, *J* = 321.2 Hz), 99.56, 93.97, 52.55, 52.26, 39.27, 38.79, 21.25, 20.70. HPLC chiralcel AS-H column (10 % isopropanol in hexanes, 0.3 mL/min) t_r= 29.640 min (minor), 31.907 min (major): 96 % ee. HRMS (ESI-TOF) *m/z* Calcd for $C_{12}H_{14}F_3INO_4S^+$ $[MH]^+$ 451.9635, found 451.9633.



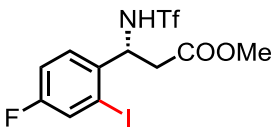
6c methyl 3-(2-iodo-5-methylphenyl)-3-((trifluoromethyl)sulfonamido)propanoate

The compound **6c** was prepared according to the general procedure and was purified by prep-TLC to give a colorless oil in 44 % yield (40 mg). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.70 (d, *J* = 8.0 Hz, 1H), 7.21 (s, 1H), 7.05 (d, *J* = 9.1 Hz, 1H), 6.85 (d, *J* = 8.0 Hz, 1H), 5.21 (dt, *J* = 9.1, 5.1 Hz, 1H), 3.67 (s, 3H), 2.95 (d, *J* = 5.1 Hz, 2H), 2.32 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 171.18, 140.46, 139.76, 138.96, 131.06, 128.11, 119.35 (q, *J* = 320.7 Hz) 93.03, 58.77, 52.27, 39.14, 21.10. HPLC chiralcel AD-H column (5 % isopropanol in hexanes, 0.5 mL/min) t_r= 11.980 min (minor), 14.693 min (major): 94 % ee. HRMS (ESI-TOF) *m/z* Calcd for $C_{12}H_{14}F_3INO_4S^+$ $[MH]^+$ 451.9635, found 451.9629.



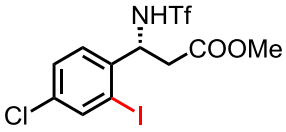
6d methyl 3-(2-iodo-4-methoxyphenyl)-3-((trifluoromethyl)sulfonamido)propanoate

The compound **6d** was prepared according to the general procedure and was purified by prep-TLC to give a colorless oil in 33 % yield (30 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37 (d, *J* = 2.6 Hz, 1H), 7.30 (d, *J* = 8.7 Hz, 1H), 6.92 (dd, *J* = 8.7, 2.6 Hz, 1H), 5.20 (t, *J* = 5.1 Hz, 1H), 3.79 (s, 3H), 3.66 (s, 3H), 2.94 (d, *J* = 5.1 Hz, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 171.39, 159.49, 132.92, 127.62, 124.99, 119.35 (q, *J* = 321.1 Hz), 114.64, 97.27, 58.41, 55.52, 52.29, 39.53. HPLC chiralcel OJ column (10 % isopropanol in hexanes, 0.5 mL/min) t_r= 31.120 min (major), 39.807 min (minor): 98 % ee. HRMS (ESI-TOF) *m/z* Calcd for $C_{12}H_{13}F_3INaNO_5S^+$ $[MNa]^+$ 489.9403, found 489.9337.



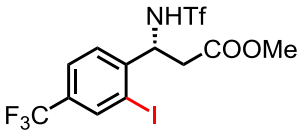
6e methyl 3-(4-fluoro-2-iodophenyl)-3-((trifluoromethyl)sulfonamido)propanoate

The compound **6e** was prepared according to the general procedure and was purified by prep-TLC to give a colorless oil in 41 % yield (39 mg). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.58 (dd, *J* = 7.8, 2.6 Hz, 1H), 7.41 (dd, *J* = 8.8, 5.6 Hz, 1H), 7.12 (m, 1H), 5.21 (t, *J* = 4.9 Hz, 1H), 3.66 (s, 3H), 2.98 – 2.91 (m, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 171.26 , 161.54 (d, *J* = 253.6 Hz), 136.84 (d, *J* = 3.5 Hz), 128.27 (d, *J* = 8.2 Hz), 126.93 (d, *J* = 23.7 Hz), 119.31 (q, *J* = 320.7 Hz), 115.93 (d, *J* = 21.2 Hz), 96.49 (d, *J* = 8.2 Hz), 58.15 , 52.39 , 38.86 . HPLC chiralcel OJ column (10 % isopropanol in hexanes, 0.3 mL/min) t_r= 37.327 min (major), 44.560 min (minor): 98 % ee. HRMS (ESI-TOF) *m/z* Calcd for $C_{11}H_{10}F_4INaNO_4S^+$ $[MNa]^+$ 477.9204, found 477.9201.



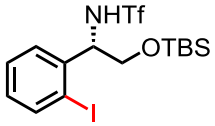
6f methyl 3-(4-chloro-2-iodophenyl)-3-((trifluoromethyl)sulfonamido)propanoate

The compound **6f** was prepared according to the general procedure and was purified by prep-TLC to give a colorless oil in 40 % yield (37 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.93 (d, *J* = 1.4 Hz, 1H), 7.53 – 7.43 (m, 2H), 7.24 (d, *J* = 9.1 Hz, 1H), 5.28 (dt, *J* = 9.1, 4.8 Hz, 1H), 3.75 (s, 3H), 3.11 – 3.00 (m, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 171.21, 139.46, 139.26, 134.90, 129.00, 127.99, 119.29 (q, *J* = 320.7 Hz), 97.06, 58.34, 52.44, 38.79. HPLC chiralcel OJ column (10 % isopropanol in hexanes, 0.5 mL/min) t_r= 19.533 min (major), 28.627 min (minor): 98 % ee. HRMS (ESI-TOF) *m/z* Calcd for $C_{11}H_{10}ClF_3INaNO_4S^+$ $[MH]^+$ 493.8908, found 493.8906.



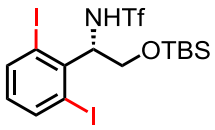
6g methyl 3-(4-trifluoromethyl-2-iodophenyl)-3-((trifluoromethyl)sulfonamido)propanoate

The compound **6g** was prepared according to the general procedure and was purified by prep-TLC to give a colorless oil in 38 % yield (40 mg). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.14 – 8.08 (m, 1H), 7.66 (dd, *J* = 8.3, 1.7 Hz, 1H), 7.58 (d, *J* = 8.3 Hz, 1H), 5.30 – 5.23 (m, 1H), 3.68 (s, 3H), 3.04 – 2.95 (m, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 171.10 , 144.80 , 136.89 (q, *J* = 3.6 Hz), 132.00 (q, *J* = 33.3 Hz), 127.72 , 125.66 (q, *J* = 3.4 Hz), 122.5 (q, *J* = 272.9 Hz), 119.31 (q, *J* = 320.9 Hz), 96.79 , 58.62 , 52.51 , 38.38 . HPLC chiralcel OJ column (10 % isopropanol in hexanes, 0.2 mL/min) t_r= 31.760 min (major), 40.273 min (minor): 99 % ee. HRMS (ESI-TOF) *m/z* Calcd for $C_{11}H_{10}ClF_3INaNO_4S^+$ $[MH]^+$ 505.9352, found 505.9355.



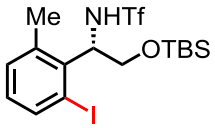
9a-mono *N*-(2-((*tert*-butyldimethylsilyl)oxy)-1-(2-iodophenyl)ethyl)-1,1,1-trifluoromethanesulfonamide

The compound **9a-mono** was prepared according to the general procedure and was purified by prep-TLC to give a colorless oil in 20 % yield (20 mg). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.84 (d, *J* = 7.0 Hz, 1H), 7.38 (t, *J* = 7.5 Hz, 1H), 7.33 (d, *J* = 7.7 Hz, 1H), 7.02 (t, *J* = 7.6 Hz, 1H), 5.05 (dd, *J* = 4.5, 3.8 Hz, 1H), 3.95 (dd, *J* = 10.6, 3.8 Hz, 1H), 3.73 (dd, *J* = 10.6, 4.5 Hz, 1H), 0.84 (s, 9H), -0.06 (s, 3H), -0.09 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 139.77, 139.66, 129.89, 128.25, 128.14, 119.45 (q, *J* = 321.3 Hz), 97.39, 65.07, 63.28, 25.72, 18.24, -5.68, -5.79. The compound was deiodinated and then ee was determined. HPLC chiralcel AD-H column (5 % isopropanol in hexanes, 0.1 mL/min) t_r= 37.320 min (minor), 40.260 min (major): 87 % ee. HRMS (ESI-TOF) *m/z* Calcd for $C_{15}H_{22}F_3INO_3SSi^-$ $[M-H]^-$ 508.0092, found 508.0091.



9a-di *N*-(2-((*tert*-butyldimethylsilyl)oxy)-1-(2,6-diiodophenyl)ethyl)-1,1,1-trifluoromethanesulfonamide

The compound **9a-di** was prepared according to the general procedure and was purified by prep-TLC to give a colorless oil in 20 % yield (25 mg). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.93 (m, 2H), 6.65 (t, *J* = 7.8 Hz, 1H), 6.24 (br, 1H), 5.49 (t, *J* = 7.6 Hz, 1H), 4.09 – 4.01 (m, 1H), 3.94 – 3.87 (m, 1H), 0.89 (s, 9H), 0.12 (s, 3H), 0.06(s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 142.51, 140.72, 138.24, 131.27, 119.33 (q, *J* = 321.7 Hz), 103.48, 92.85, 66.79, 62.04, 25.75, 18.22, -5.28, -5.31. HPLC chiralcel AD-H column (5 % isopropanol in hexanes, 0.1 mL/min) t_r= 32.867 min (minor), 39.473 min (major): 99 % ee. HRMS (ESI-TOF) *m/z* Calcd for $C_{15}H_{21}F_3I_2NO_3SSi^-$ $[M-H]^-$ 633.9058, found 633.9055.

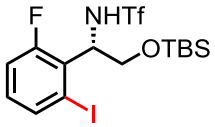


9b

***N*-(2-((*tert*-butyldimethylsilyl)oxy)-1-(2-methyl-6-iodophenyl)ethyl)-1,1,1-trifluoromethanesulfonamide**

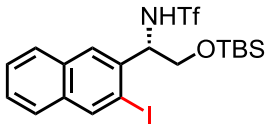
The compound **9b** was prepared according to the general procedure and was purified by prep-TLC to give a colorless oil in 49 % yield (51 mg). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.75 (d, *J* = 7.8 Hz, 1H), 7.19 (d, *J* = 7.4 Hz, 0.7H), 7.14 (d, *J* = 7.4 Hz, 0.3H), 6.94 – 6.82 (m, 1H), 5.48 – 5.40 (m, 0.3H), 5.27 – 5.19 (m, 0.7H), 4.16 – 4.08 (m, 0.7H), 4.06 – 3.98 (m, 0.7H), 3.88 (dd, *J* = 10.9, 5.0 Hz, 0.3H), 3.81 (t, *J* = 9.7 Hz, 0.3H), 2.53 (s, 1H), 2.43 (s, 2H), 0.91 (s, 3H), 0.83 (s, 6H), 0.12 – 0.06 (m, 2H), 0.04 (s, 2H), -0.04 (s, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 140.28,

139.90, 138.40, 137.62, 136.24, 135.98, 133.04, 131.12, 129.87, 129.70, 119.32 (q, *J* = 321.1 Hz), 101.75, 93.97, 66.52, 63.10, 63.04, 56.92, 25.76, 25.67, 21.82, 20.98, 18.27, 18.11, -5.36, -5.43, -5.47, -5.51. The compound was deiodinated and the TBS ether was deprotected and then ee was determined. HPLC chiralcel AD-H column (10 % isopropanol in hexanes, 0.5 mL/min) t_r= 23.200 min (minor), 30.947 min (major): 91 % ee. HRMS (ESI-TOF) *m/z* Calcd for $C_{16}H_{24}F_3INO_3SSi^-$ $[M-H]^-$ 522.0248, found 522.0248.



9c *N*-(2-((*tert*-butyldimethylsilyl)oxy)-1-(2-fluoro-6-iodophenyl)ethyl)-1,1,1-trifluoromethanesulfonamide

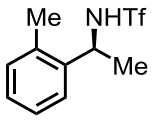
The compound **9c** was prepared according to the general procedure and was purified by prep-TLC to give a colorless oil in 41 % yield (43 mg). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.71 – 7.65 (m, 1H), 7.13 – 7.05 (m, 1H), 7.05 – 6.98 (m, 1H), 5.87 (s, 1H), 5.19 (s, 1H), 3.87 (s, 2H), 0.85 (s, 9H), 0.03 – -0.09 (m, 6H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 160.54 (d, *J* = 250.4 Hz), 136.02, 131.22 (d, *J* = 9.7 Hz), 127.74 (d, *J* = 9.7 Hz), 119.33 (d, *J* = 320.9 Hz), 116.73 (d, *J* = 23.8 Hz), 99.47, 64.27, 63.97, 25.66, 18.18, -5.59, -5.67. HPLC chiralcel AS-H column (5 % isopropanol in hexanes, 0.3 mL/min) t_r= 13.473 min (major), 14.520 min (minor): 99 % ee. HRMS (ESI-TOF) *m/z* Calcd for $C_{15}H_{23}F_4INO_3SSi^+$ $[MH]^+$ 528.0143, found 428.0141.



9d

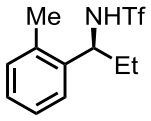
***N*-(2-((*tert*-butyldimethylsilyl)oxy)-1-(3-iodonaphthalen-2-yl)ethyl)-1,1,1-trifluoromethanesulfonamide**

The compound **9d** was prepared according to the general procedure and was purified by prep-TLC to give a colorless oil in 40 % yield (44 mg). ^{1}H NMR (400 MHz, cdcl$_3$) δ 8.40 (s, 1H), 7.83 – 7.75 (m, 2H), 7.72 (d, *J* = 8.9 Hz, 1H), 7.57 – 7.46 (m, 2H), 6.04 (s, 1H), 5.21 (ddd, *J* = 7.3, 4.1, 3.8 Hz, 1H), 4.04 (dd, *J* = 10.7, 3.8 Hz, 1H), 3.79 (dd, *J* = 10.7, 4.1 Hz, 1H), 0.82 (s, 9H), -0.10 (s, 3H), -0.18 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 139.28, 135.74, 134.37, 132.36, 127.86, 127.32, 127.20, 127.18, 126.47, 119.51 (q, *J* = 321.4 Hz), 94.13, 65.13, 63.02, 25.68, 18.20, -5.69, -5.80. HPLC chiralcel AS-H column (5 % isopropanol in hexanes, 0.1 mL/min) t_r= 48.873 min (minor), 53.613 min (major): 96 % ee. HRMS (ESI-TOF) *m/z* Calcd for $C_{19}H_{26}F_3INO_3SSi^+$ $[MH]^+$ 560.0394, found 560.0398.



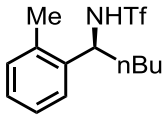
2a

HPLC chiralcel AS-H column (5 % isopropanol in hexanes, 0.5 mL/min) t_r= 11.200 min (minor), 30.613 min (major): 93 % ee.



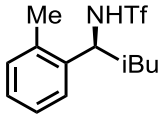
2b

HPLC chiralcel AS-H column (5 % isopropanol in hexanes, 0.5 mL/min) t_r= 10.553 min (minor), 16.260 min (major): 89 % ee.



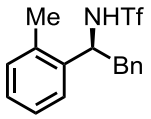
2c

HPLC chiralcel AS-H column (5 % isopropanol in hexanes, 0.5 mL/min) t_r= 9.733 min (minor), 12.880 min (major): 77 % ee.



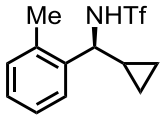
2d

HPLC chiralcel AS-H column (5 % isopropanol in hexanes, 0.5 mL/min) t_r= 8.447 min (minor), 9.600 min (major): 83 % ee.



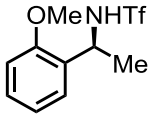
2e

HPLC chiralcel AS-H column (5 % isopropanol in hexanes, 0.5 mL/min) t_r= 14.440 min (minor), 18.167 min (major): 67 % ee.



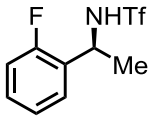
2f

HPLC chiralcel AS-H column (5 % isopropanol in hexanes, 0.8 mL/min) t_r= 7.853 min (minor), 26.180 min (major): 77 % ee.



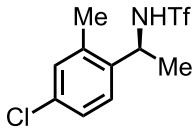
2g

HPLC chiralcel AS-H column (5 % isopropanol in hexanes, 0.8 mL/min) t_r= 9.687 min (minor), 32.247 min (major): 93 % ee.



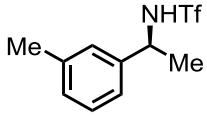
2h

HPLC chiralcel AS-H column (5 % isopropanol in hexanes, 0.8 mL/min) t_r= 7.833 min (minor), 11.200 min (major): 89 % ee.



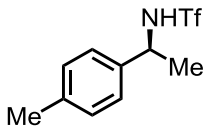
2i

HPLC chiralcel AS-H column (5 % isopropanol in hexanes, 0.5 mL/min) t_r= 11.893 min (minor), 25.320 min (major): 91 % ee.



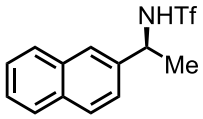
2j

HPLC chiralcel AS-H column (5 % isopropanol in hexanes, 0.8 mL/min) t_r= 7.100 min (minor), 12.013 min (major): 78 % ee.



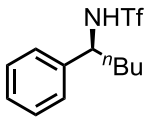
2k

HPLC chiralcel AS-H column (10 % isopropanol in hexanes, 0.3 mL/min) t_r= 15.747 min (minor), 22.540 min (major): 78 % ee.



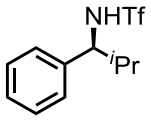
2l

HPLC chiralcel AS-H column (5 % isopropanol in hexanes, 0.5 mL/min) t_r= 17.173 min (minor), 23.427 min (major): 67 % ee.



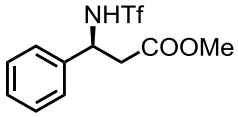
2m

HPLC chiralcel AS-H column (10 % isopropanol in hexanes, 0.5 mL/min) t_r= 9.827 min (minor), 10.853 min (major): 70 % ee.



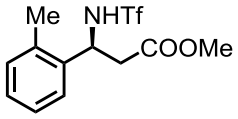
2n

HPLC chiralcel AS-H column (10 % isopropanol in hexanes, 0.5 mL/min) t_r= 7.807 min (minor), 9.287 min (major): 45 % ee.



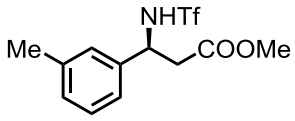
5a

HPLC chiralcel OJ column (10 % isopropanol in hexanes, 0.3 mL/min) t_r= 45.227 min (minor), 50.520 min (major): 85 % ee.



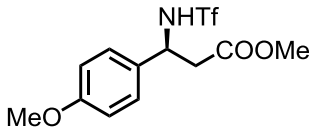
5b

HPLC chiralcel AS-H column (10 % isopropanol in hexanes, 0.5 mL/min) t_r= 17.280 min (minor), 18.913 min (major): 93 % ee.



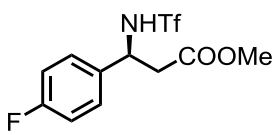
5c

HPLC chiralcel OJ column (10 % isopropanol in hexanes, 0.5 mL/min) t_r= 16.933 min (major), 29.307 min (minor): 93 % ee.



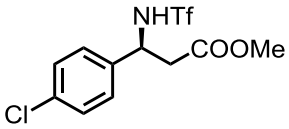
5d

HPLC chiralcel OJ column (10 % isopropanol in hexanes, 0.5 mL/min) t_r= 42.313 min (minor), 53.093 min (major): 82 % ee.



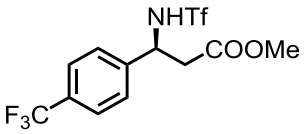
5e

HPLC chiralcel OJ column (10 % isopropanol in hexanes, 0.5 mL/min) t_r= 21.093 min (minor), 26.307 min (major): 90 % ee.



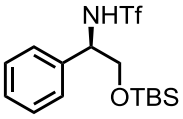
5f

HPLC chiralcel OJ column (10 % isopropanol in hexanes, 0.5 mL/min) t_r= 21.947 min (minor), 27.620 min (major): 92 % ee.



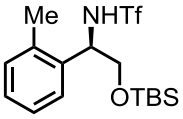
5g

HPLC chiralcel OJ column (10 % isopropanol in hexanes, 0.5 mL/min) t_r= 14.347 min (major), 17.787 min (minor): 65 % ee.



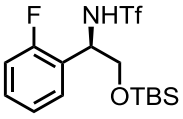
8a

HPLC chiralcel AD-H column (5 % isopropanol in hexanes, 0.1 mL/min) t_r= 37.700 min (major), 40.687 min (minor): 71 % ee.



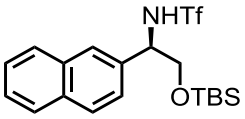
8b

The TBS ether was deprotected and then ee was determined. HPLC chiralcel AS-H column (10 % isopropanol in hexanes, 0.3 mL/min) t_r= 20.167 min (minor), 23.213 min (major): 91 % ee.



8c

The TBS ether was deprotected and then ee was determined. HPLC chiralcel AS-H column (10 % isopropanol in hexanes, 0.2 mL/min) t_r= 39.120 min (major), 44.313 min (minor): 68 % ee.



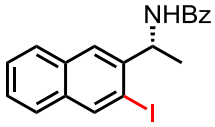
8d

HPLC chiralcel AS-H column (5 % isopropanol in hexanes, 0.2 mL/min) t_r= 21.167 min (major), 24.160 min (minor): 72 % ee.

Deprotection of Trifluoromethanesulfonamides 3l (*32*)

To a 20 mL round bottle was added **3l** (1 equiv. 0.5 mmol), K_2CO_3 (2 equiv. 1.0 mmol), iodoacetonitrile (1.2 equiv. 0.6 mmol) and acetone (9mL). The mixture was stirred at room temperature for 12 h under nitrogen. Upon completion (monitored by TLC), the resulting solution was diluted by EtOAc, filtered and evaporated to afford the alkylated trifluoromethanesulfonamide, which could be used directly into the next step without purification.

To a 20 mL round bottle was added the alkylated trifluoromethanesulfonamide, Cs_2CO_3 (3 equiv.) and anhydrous THF. The mixture was stirred at 40 ^oC overnight under nitrogen. Upon completion, the resulting solution was cooled to 0 ^oC, at which time aq. HCl (3 N) was added. The mixture was stirred for another 2 h at room temperature and cooled to 0 ^oC. 1N NaOH was added to reach pH = 14, and BzCl (1.5 equiv.) was dissolved in Dioxane and added dropwise during 30 min at 0 ^oC. The mixture was warmed to room temperature and stirred for another 12 h. Upon completion, the mixture was cooled to 0 ^oC and 1N HCl was added to reach pH = 1. The mixture was extracted with EtOAc (20 mL × 3) and the organic layer was washed with brine and dried over $MgSO_4$. The solvent was evaporated under reduced pressure and the mixture was purified by column chromatography on silica gel (EtOAc/hexane= 1:10 as eluant) afforded **10** as a white solid (103 mg, 72 % yield). And the ee was determined by converting the product back to the triflate amide (95 % ee).



10 N-(1-((2-iodo)-naphthalen-2-yl)ethyl)benzamide

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.89 – 7.75 (m, 6H), 7.55 – 7.44 (m, 4H), 7.44 – 7.36 (m, 2H), 6.46 (d, *J* = 7.0 Hz, 1H), 5.56 – 5.44 (m, 1H), 1.69 (d, *J* = 6.9 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 166.57, 140.44, 134.53, 133.32, 132.74, 131.47, 128.56, 128.54, 127.89, 127.59, 126.92, 126.23, 125.91, 124.77, 124.61, 49.25, 21.61.

Synthetic Transformations of Iodinated Products

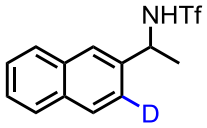
Procedure for the preparation of compound 11

To a 10 mL schlenk tube was added **3l** (1 equiv.), $Pd(PPh_4)_4$ (0.05 equiv.), HCO_2Na (2 equiv.) and DMF. The mixture was stirred under nitrogen at 110 ^oC for 5 h. After completion, the mixture was filtrated through a pad

of celite, concentrated and purified by prep-TLC to obtain a white solid (96 % yield).

Procedure for the preparation of compound 12

To a stirred solution of **3l** in THF was added iPrMgCl-LiCl (1.1 equiv.) dropwise at 0^{o}C. The mixture was stirred at this temperature for 3 h and was quenched with D_2O. The resulting solution was extracted with EtOAc (3 times) and the organic layer was dried over $MgSO_4$, concentrated and purified by prep-TLC to obtain a white solid (99 % yield).



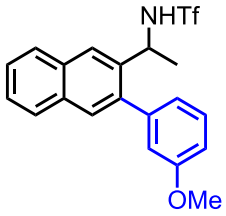
12 1,1,1-trifluoro-*N*-(1-(naphthalen-2-yl-3-d)ethyl)methanesulfonamide

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.91 – 7.80 (m, 3H), 7.75 (s, 1H), 7.56 – 7.47 (m, 2H), 5.28 (d, *J* = 7.9 Hz, 1H), 5.01 – 4.92 (m, 1H), 1.72 (d, *J* = 6.9 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 138.05, 133.17, 133.01, 128.99, 128.02, 127.70, 126.66, 126.54, 124.79, 123.37 (t, *J* = 23.9 Hz), 119.52 (q, *J* = 320.8 Hz), 55.38, 23.37.

Procedure for the preparation of compound 13(*33*)

To a 10 mL schlenk tube was added **3l** (0.1 mmol, 1 equiv.), 3-methoxyphenyl boronic acid (1.5 equiv.), $Pd(OAc)_2$ (0.025 equiv.), S-phos (0.05 equiv.), K_2CO_3 (3 equiv.) and CH_3CN/H_2O. The mixture was stirred under N_2 at 100 ^{o}C for 12 h. After completion, the mixture was filtrated through a pad of celite, concentrated and purified by prep-TLC to obtain the product as a colorless oil (93 % yield).



13 1,1,1-trifluoro-*N*-(1-(3-(3-methoxyphenyl)naphthalen-2-yl)ethyl)methanesulfonamide

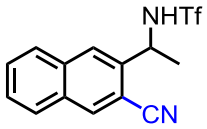
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.89 – 7.83 (m, 2H), 7.83 – 7.79 (m, 1H), 7.71 (s, 1H), 7.54 – 7.48 (m, 2H), 7.39 (t, *J* = 7.9 Hz, 1H), 7.02 – 6.90 (m, 3H), 5.39 (d, *J* = 7.9 Hz, 1H), 5.19 – 5.11 (m, 1H), 3.85 (s, 3H), 1.47 (d, *J* = 6.8 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 159.52, 141.27, 138.29, 138.13, 132.69, 132.42, 129.59, 129.49, 127.63, 127.59, 126.79, 126.57, 124.19, 121.75, 119.40 (q, *J* = 321.2 Hz), 114.70, 113.67, 55.28, 52.50, 24.96.

Procedure for the preparation of compound 14 (*34*)

To a 10 mL schlenk tube was added **3l** (0.1 mmol, 1 equiv.), CuCN (2 equiv.), L-proline (2 equiv.) and DMF

(0.5 mL). The mixture was stirred under N_2 at 120 ^oC for 48 h. After completion, the mixture was filtrated through a pad of celite, concentrated and purified by prep-TLC to obtain the product as a colorless oil (67 % yield).



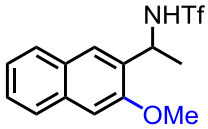
14 N-(1-(3-cyanonaphthalen-2-yl)ethyl)-1,1,1-trifluoromethanesulfonamide

^1H NMR (400 MHz, $CDCl_3$) δ 8.30 (s, 1H), 7.94 – 7.81 (m, 3H), 7.73 – 7.56 (m, 2H), 6.04 (d, *J* = 6.9 Hz, 1H), 5.25 – 5.10 (m, 1H), 1.81 (d, *J* = 7.1 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 138.81, 136.63, 134.58, 131.54, 129.93, 128.18, 126.83, 119.39 (q, *J* = 321.3 Hz), 118.33, 106.82, 54.71, 23.27.

Procedure for the preparation of compound 15 (*35*)

To a 10 mL schlenk tube was added **3l** (0.1 mmol, 1 equiv.), CuI (0.2 equiv.), 1,10-phen (0.4 equiv.) Cs_2CO_3 and MeOH (0.5 mL). The mixture was stirred under air at 110 ^oC for 24 h. After completion, the mixture was filtrated through a pad of celite, concentrated and purified by prep-TLC to obtain the product as a colorless oil (77 % yield).



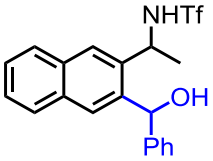
15 1,1,1-trifluoro-*N*-(1-(3-methoxynaphthalen-2-yl)ethyl)methanesulfonamide

^1H NMR (600 MHz, $CDCl_3$) δ 7.75 (t, *J* = 8.5 Hz, 2H), 7.61 (s, 1H), 7.47 (ddd, *J* = 8.1, 7.0, 1.2 Hz, 1H), 7.38 (ddd, *J* = 8.1, 7.0, 1.1 Hz, 1H), 7.26 (s, 1H), 6.14 (d, *J* = 9.4 Hz, 1H), 4.98 – 4.87 (m, 1H), 4.02 (s, 3H), 1.69 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 154.90, 133.97, 129.82, 128.51, 127.71, 127.44, 126.93, 126.45, 124.53, 119.48 (q, *J* = 321.0 Hz), 106.69, 55.53, 23.21.

Procedure for the preparation of compound 16

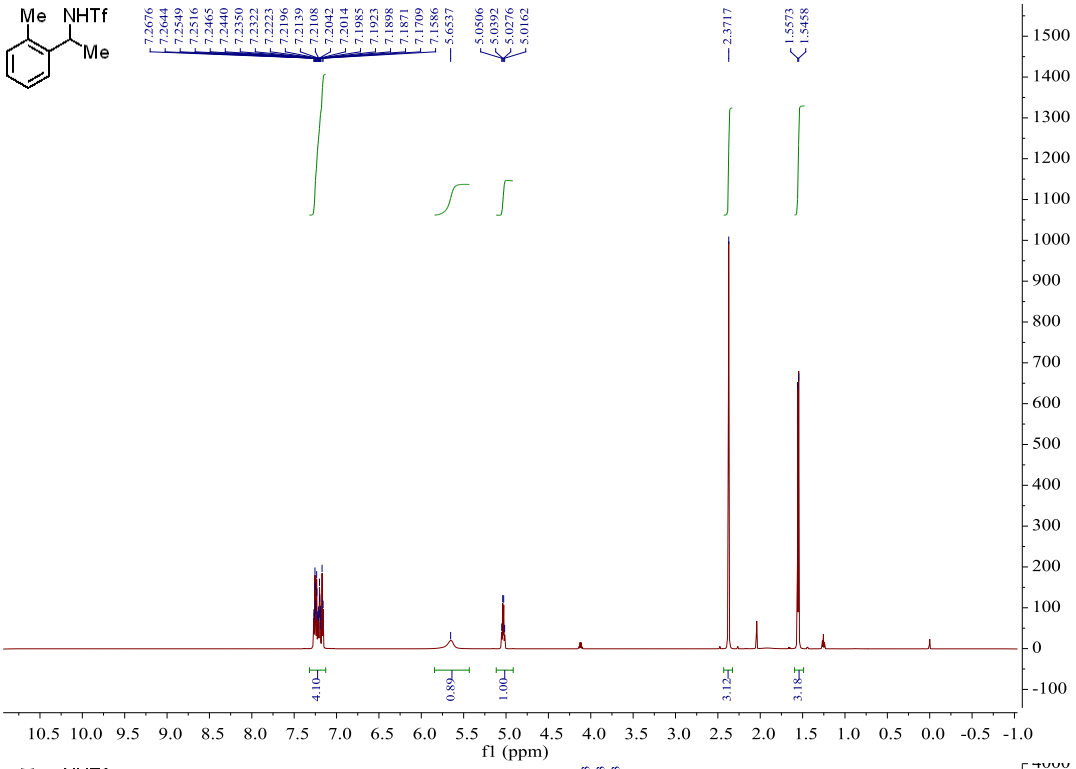
To a stirred solution of **3l** in THF was added iPrMgCl-LiCl (1.1 equiv.) dropwise at 0 ^oC. The mixture was stirred at this temperature for 3 h and PhCHO was added. The resulting solution was warmed to room temperature slowly and stirred for another 2 h. After completion, the mixture was quenched with aq. NH_4Cl, extracted with EtOAc (3 times) and the organic layer was dried over $MgSO_4$, concentrated and purified by prep-TLC to obtain a white solid (98 % yield).



16 1,1,1-trifluoro-*N*-(1-(3-(hydroxy(phenyl)methyl)naphthalen-2-yl)ethyl)methanesulfonamide

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.85 – 7.78 (m, 2H), 7.74 (d, *J* = 7.8 Hz, 1H), 7.58 (s, 1H), 7.53 – 7.45 (m, 2H), 7.43 – 7.36 (m, 4H), 7.37 – 7.30 (m, 1H), 6.27 (s, 1H), 5.96 (s, 1H), 5.42 – 5.32 (m, 1H), 2.64 (s, 1H), 1.49 (d, *J* = 6.8 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 142.04, 138.92, 137.25, 132.97, 132.40, 128.86, 128.65, 127.94, 127.86, 127.40, 126.93, 126.76, 126.56, 126.53, 119.45 (q, *J* = 321.3 Hz), 74.45, 52.30, 24.08.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NHTf
Me
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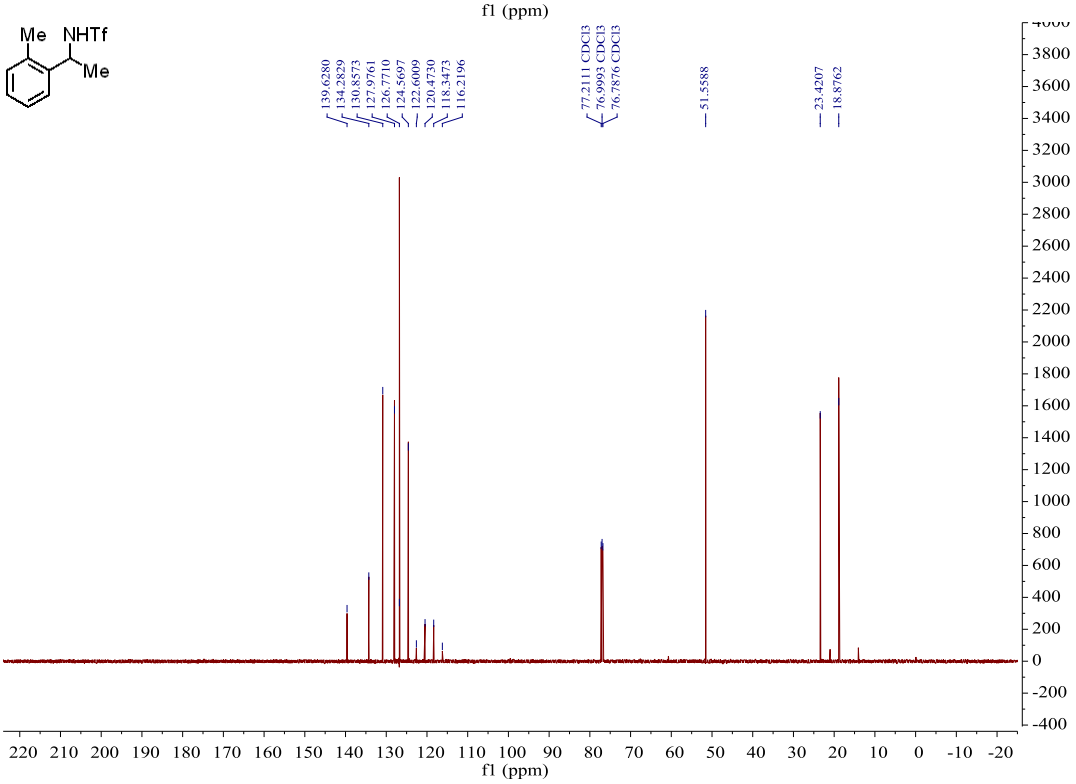
7.2549
7.2516
7.2465
7.2440
7.2350
7.2322
7.2223
7.2196
7.2139
7.2108
7.2042
7.2014
7.1985
7.1923
7.1898
7.1871
7.1709
7.1586
5.6537
5.0506
5.0392
5.0276
5.0162
2.3717
1.5573
1.5458
4.10
0.89
1.00
3.12
3.18
f1 (ppm)


Me
NHTf
Me
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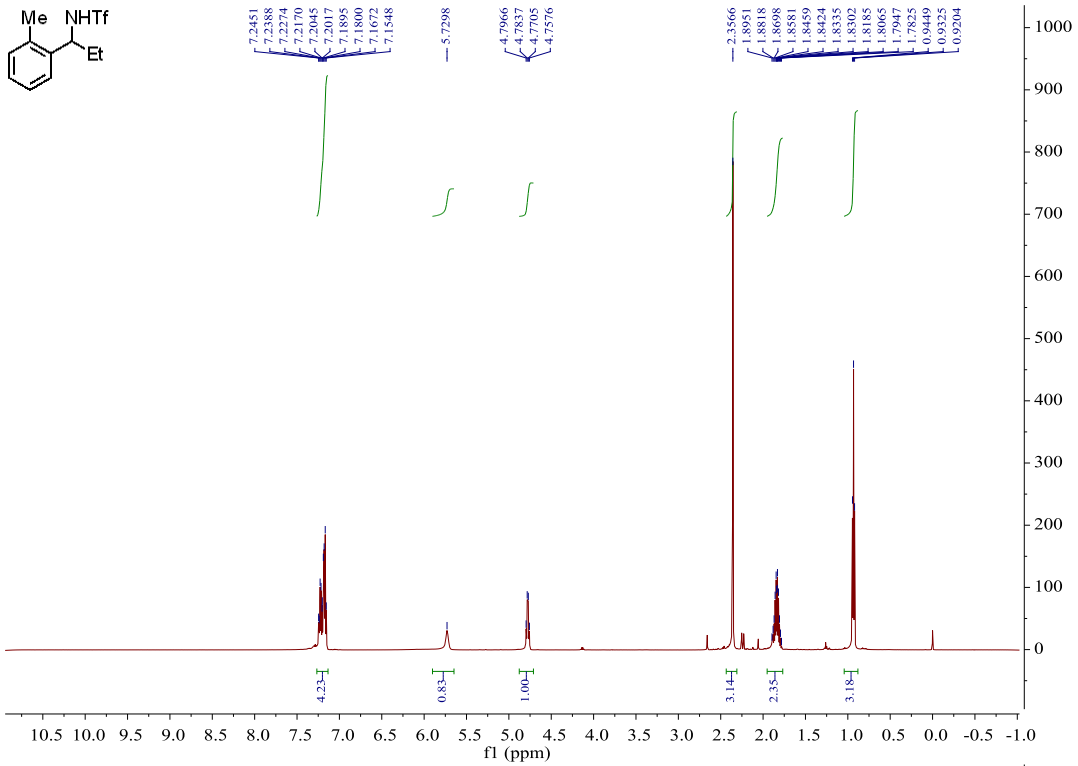
134.2829
130.8573
127.9761
126.7710
124.5697
122.6009
120.4730
118.3473
116.2196
77.2111 CDCl3
76.9993 CDCl3
76.7876 CDCl3
51.5588
23.4207
18.8762
f1 (ppm)


Me
NHTf
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7.2274
7.2170
7.2045
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7.1800
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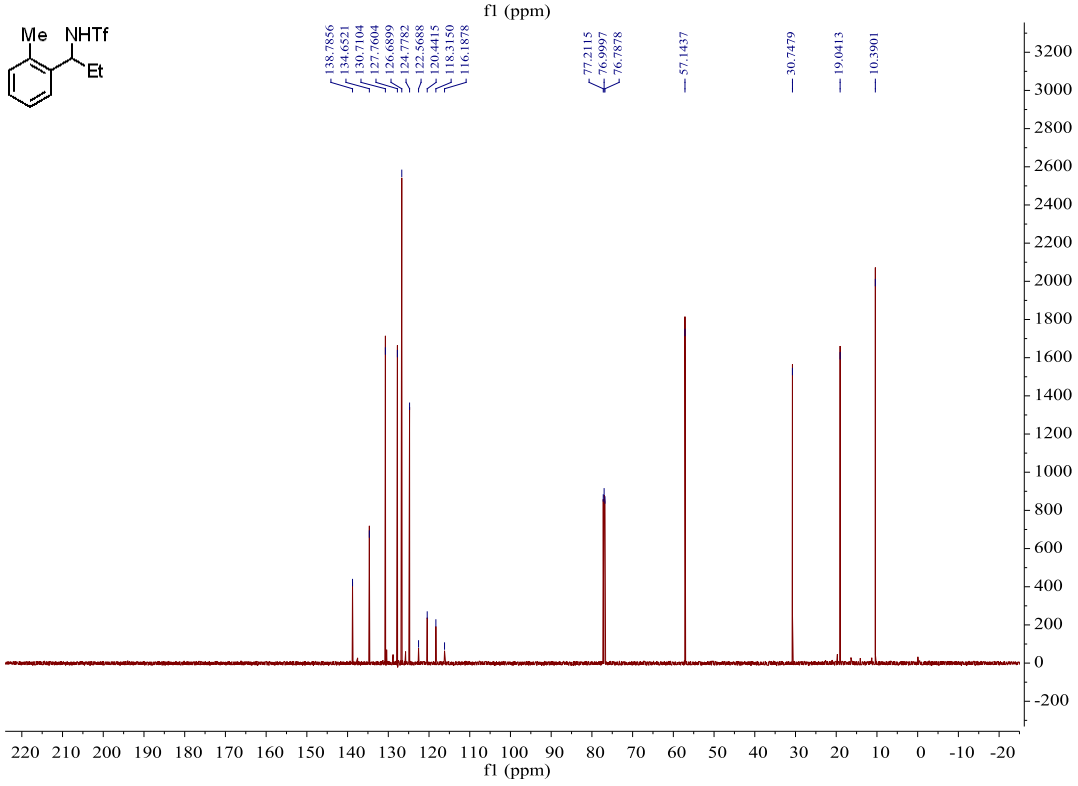
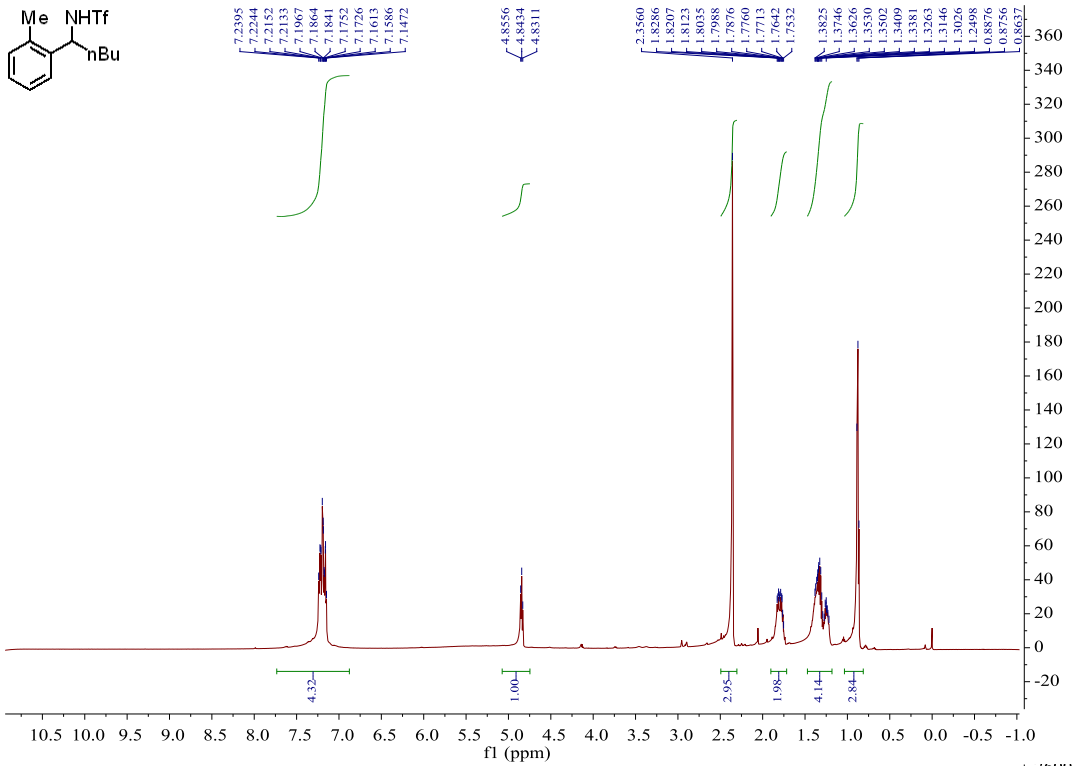
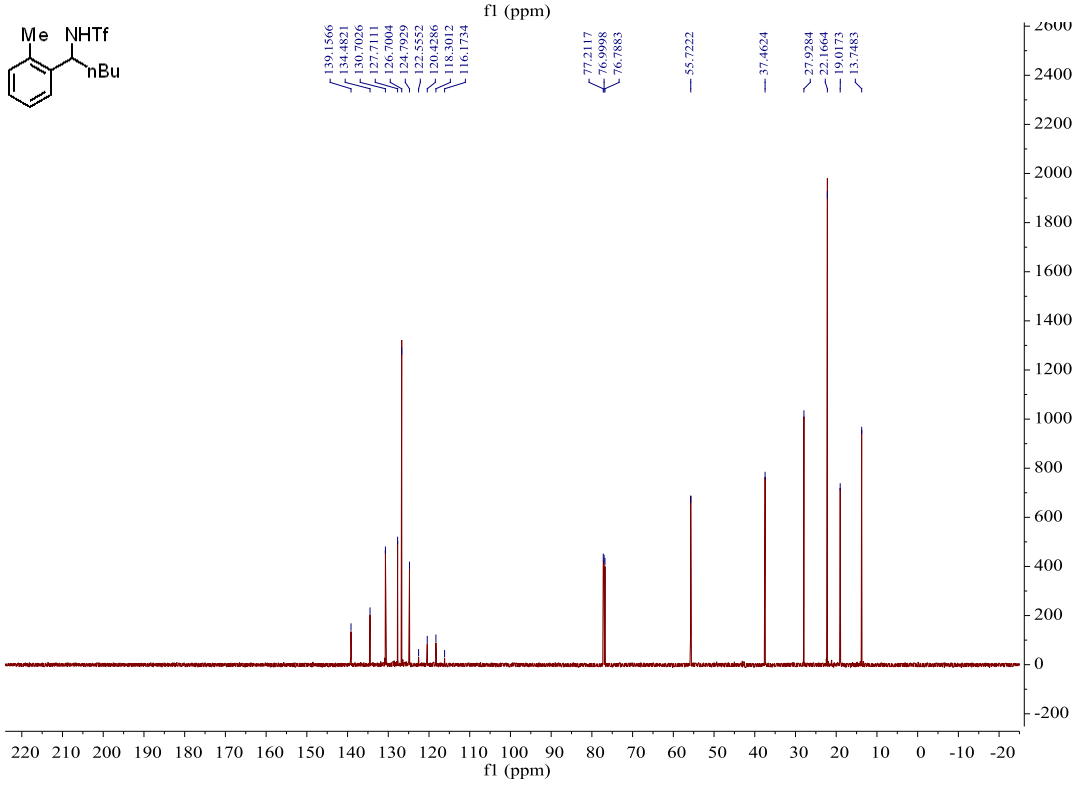
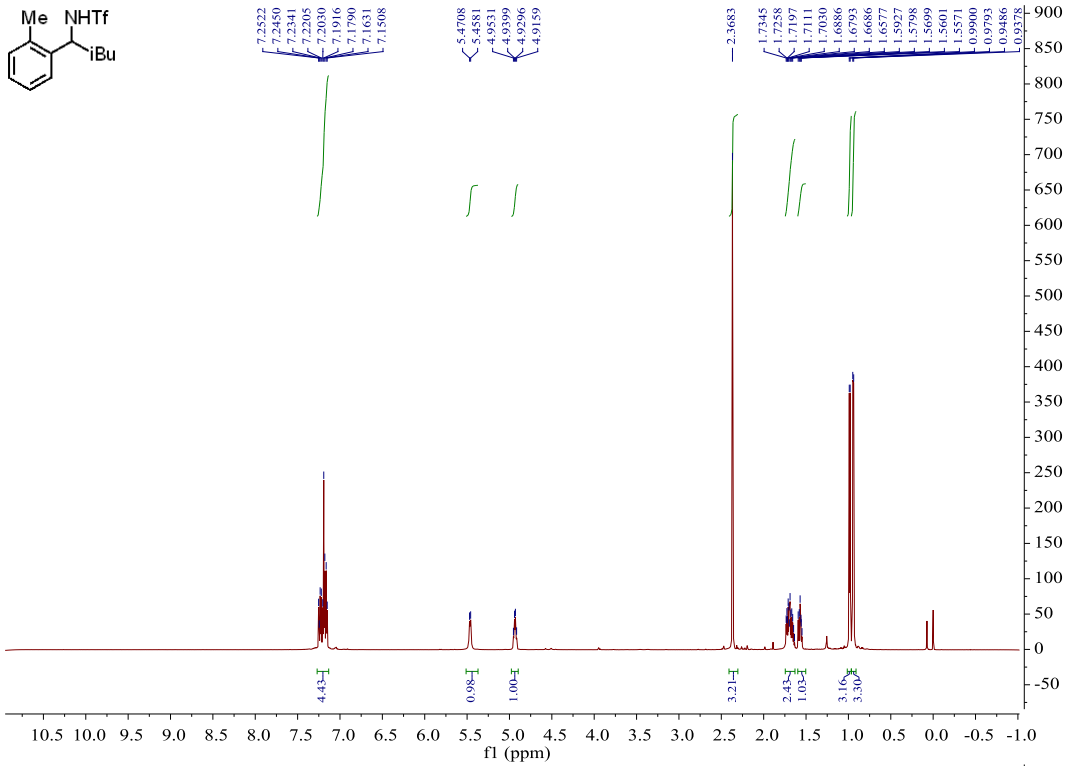
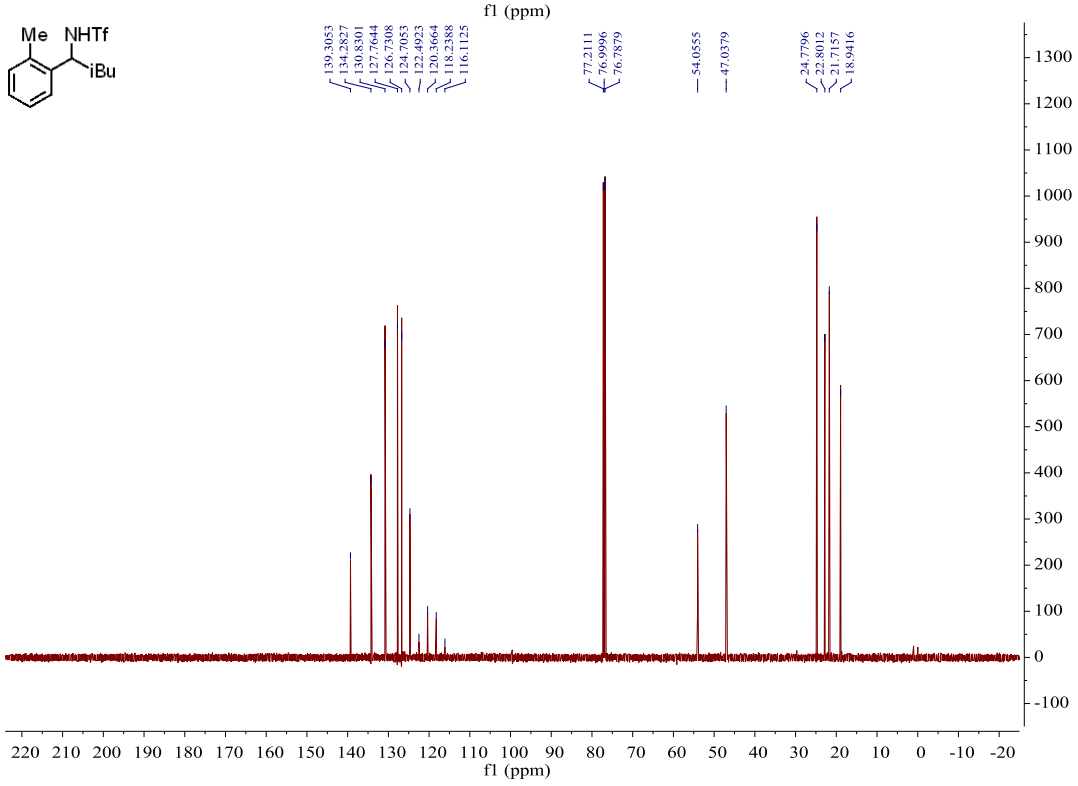
1.8581
1.8459
1.8424
1.8335
1.8302
1.8185
1.8065
1.7947
1.7825
0.9449
0.9325
0.9204
4.23
0.83
1.00
3.14
2.35
3.18
f1 (ppm)

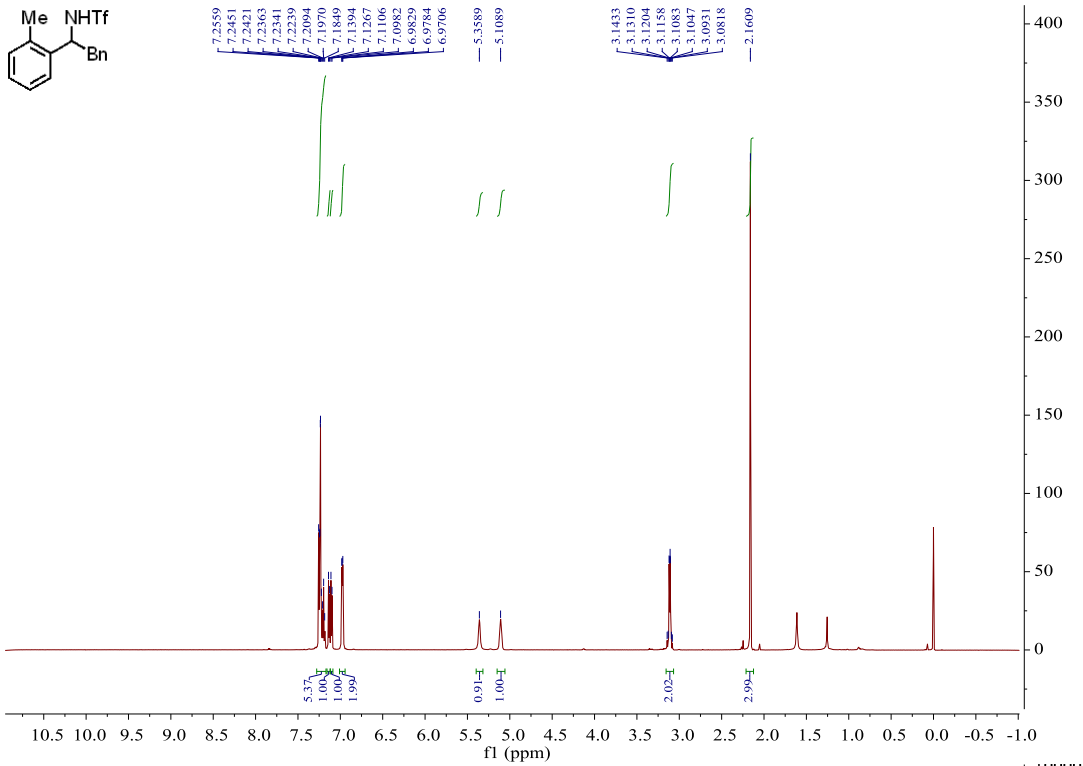

Me
NHTf
Et
138.7856
134.6521
130.7104
127.7604
126.6899
124.7782
122.5688
120.4415
118.3150
116.1878
77.2115
76.9997
76.7878
57.1437
30.7479
19.0413
10.3901
f1 (ppm)

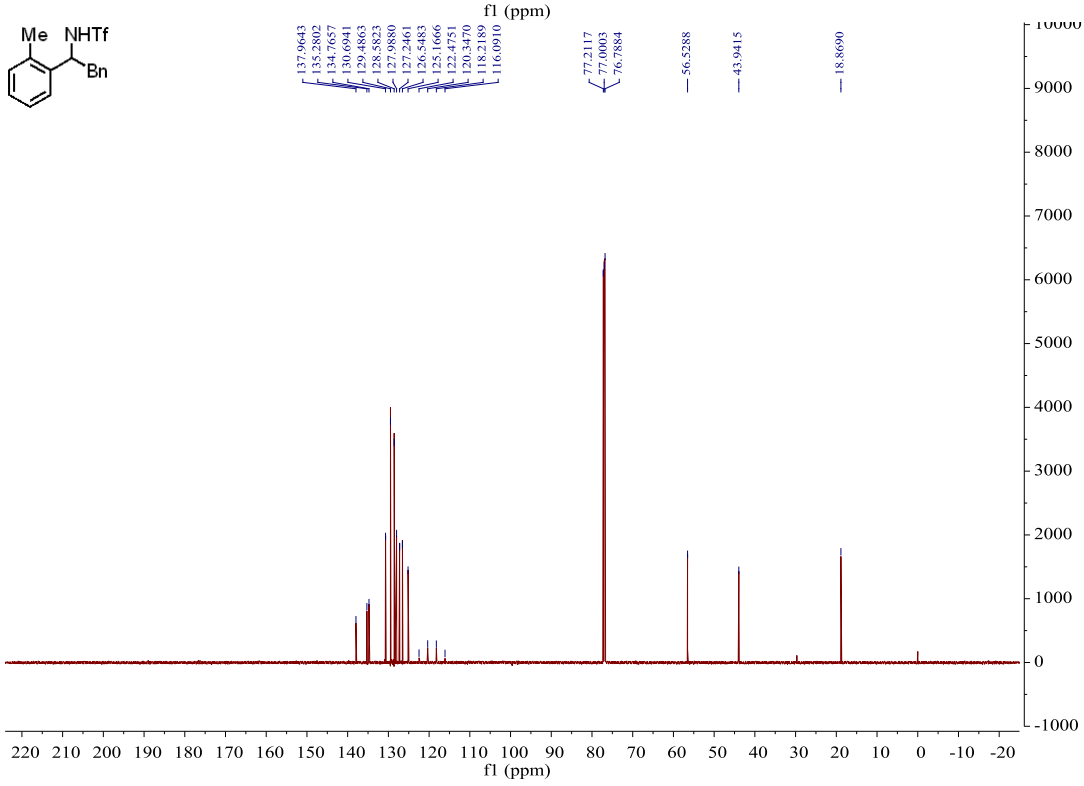

Me NHTf nBu
7.2395 7.2244 7.2152 7.2133 7.1967 7.1864 7.1841 7.1752 7.1726 7.1613 7.1586 7.1472
4.8556 4.8434 4.8311
2.3560 1.8286 1.8207 1.8123 1.8035 1.7988 1.7876 1.7760 1.7713 1.7642 1.7532
1.3825 1.3746 1.3626 1.3530 1.3502 1.3409 1.3381 1.3263 1.3146 1.3026 1.2498 0.8876 0.8756 0.8637
4.32 1.00 2.95 1.98 4.14 2.84
f1 (ppm)

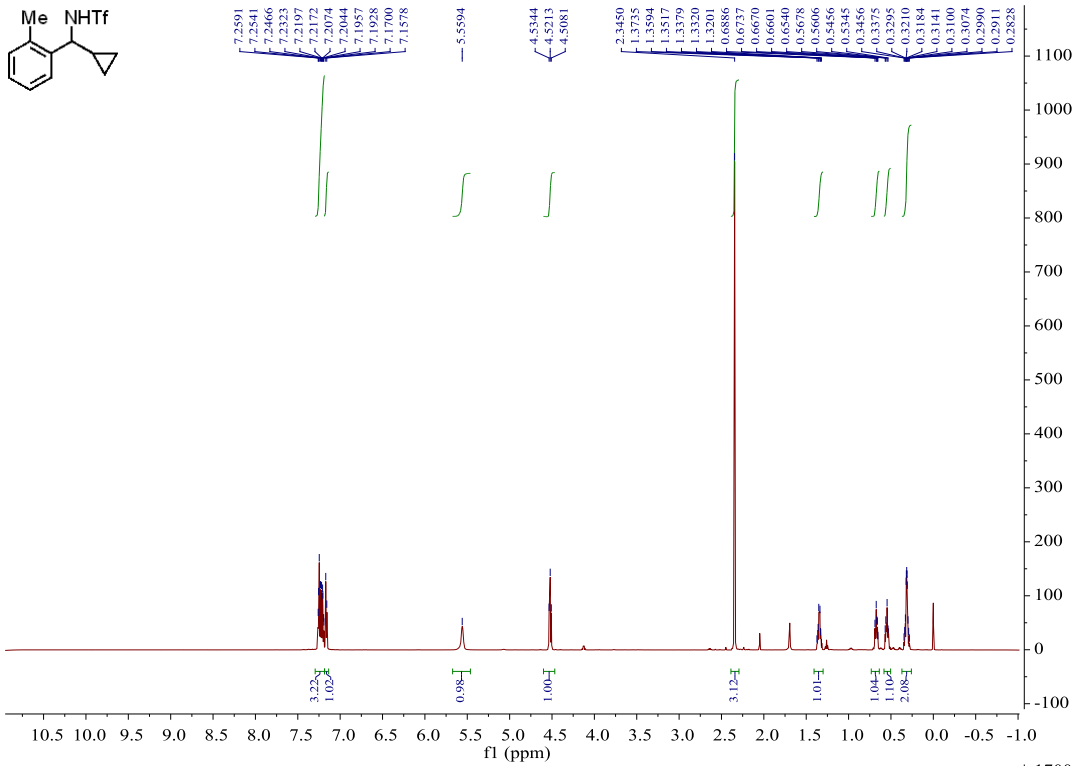

Me NHTf nBu
139.1566 134.4821 130.7026 127.7111 126.7004 124.7929 122.5552 120.4286 118.3012 116.1734
77.2117 76.9998 76.7883
55.7222
37.4624
27.9284 22.1664 19.0173 13.7483
f1 (ppm)

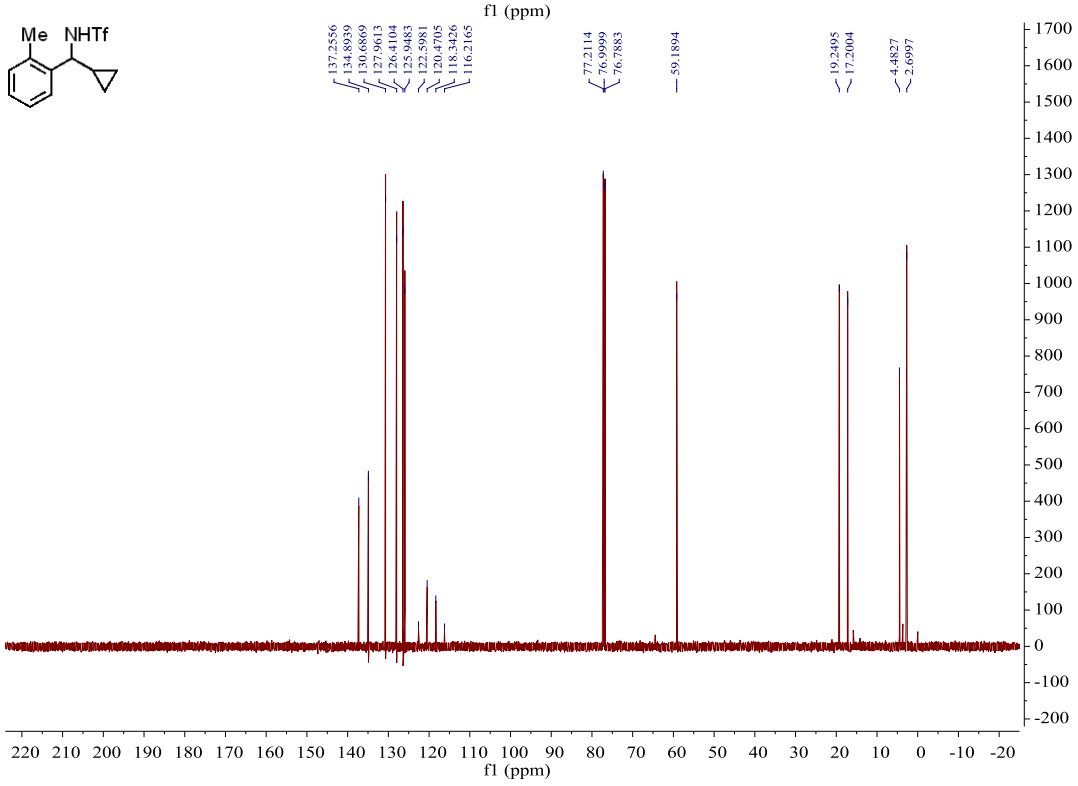

Me
NHTf
iBu
7.2522
7.2450
7.2341
7.2205
7.2030
7.1916
7.1790
7.1631
7.1508
5.4708
5.4581
4.9531
4.9399
4.9296
4.9159
2.3683
1.7345
1.7258
1.7197
1.7111
1.7030
1.6886
1.6793
1.6686
1.6577
1.5927
1.5798
1.5699
1.5601
1.5571
0.9900
0.9793
0.9486
0.9372
4.43
0.98
1.00
3.21
2.43
1.03
3.16
3.30
f1 (ppm)

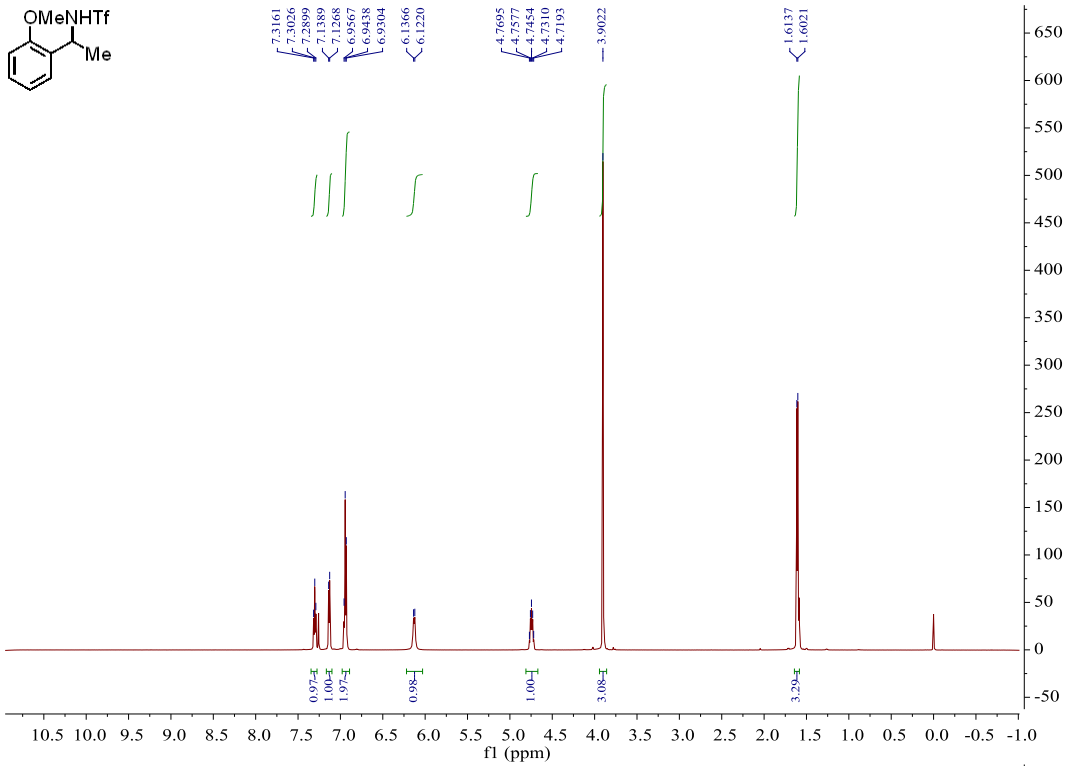

Me
NHTf
iBu
139.3053
134.2827
130.8301
127.7644
126.7308
124.7053
122.4923
120.3664
118.2388
116.1125
77.2111
76.9996
76.7879
54.0555
47.0379
24.7796
22.8012
21.7157
18.9416
f1 (ppm)

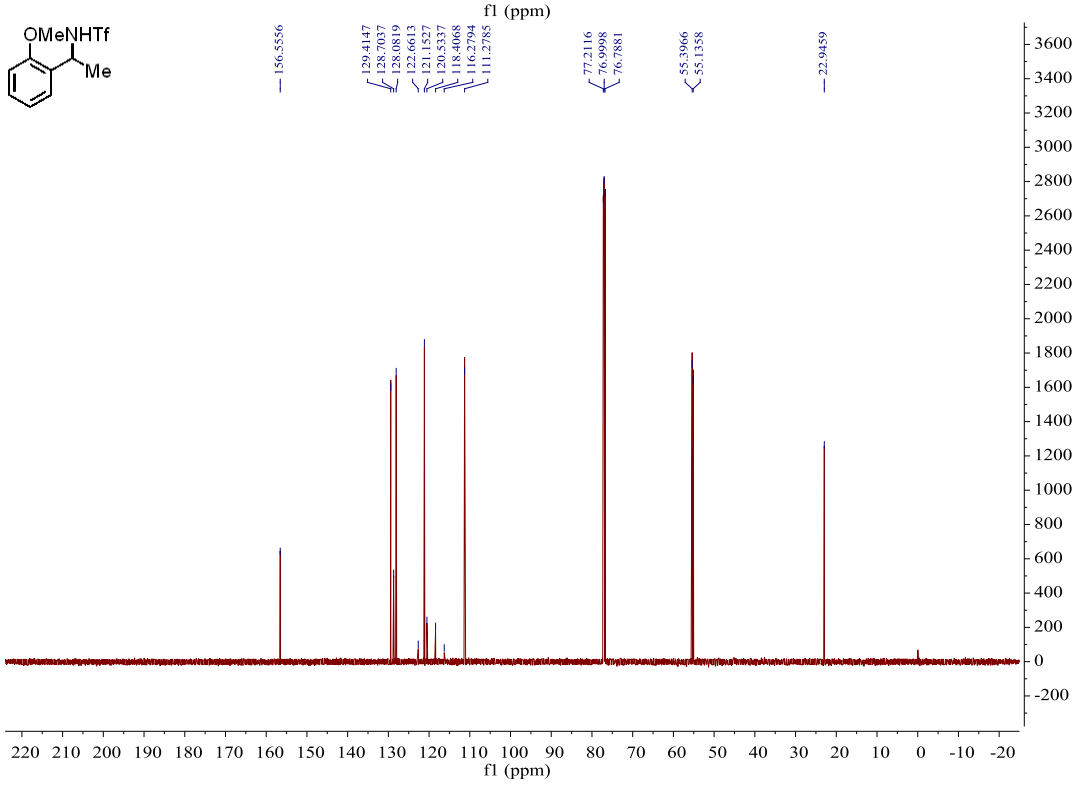

Me
NHTf
Bn
7.2559
7.2451
7.2421
7.2363
7.2341
7.2239
7.2094
7.1970
7.1849
7.1394
7.1267
7.1106
7.0982
6.9829
6.9784
6.9706
5.3589
5.1089
3.1433
3.1310
3.1204
3.1158
3.1083
3.1047
3.0931
3.0818
2.1609
5.37
1.00
1.00
1.99
0.91
1.00
2.02
2.99
f1 (ppm)

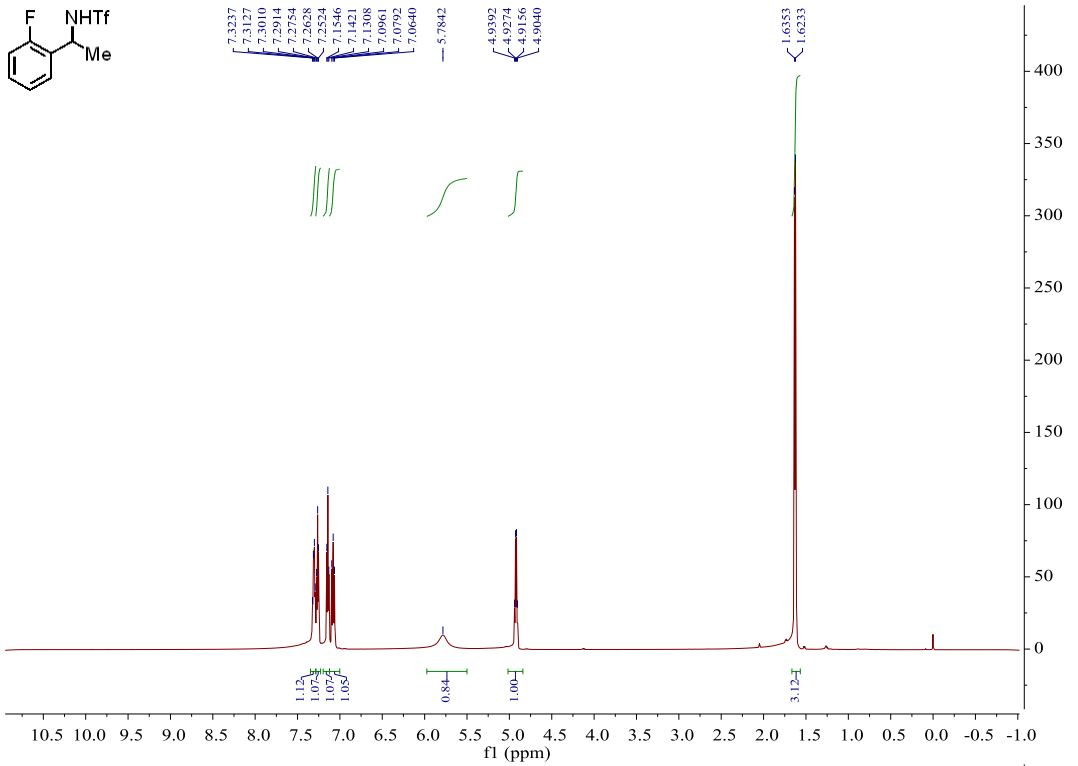

Me
NHTf
Bn
137.9643
135.2802
134.7657
130.6941
129.4863
128.5823
127.9880
127.2461
126.5483
125.1666
122.4751
120.3470
118.2189
116.0910
77.2117
77.0003
76.7884
56.5288
43.9415
18.8690
f1 (ppm)

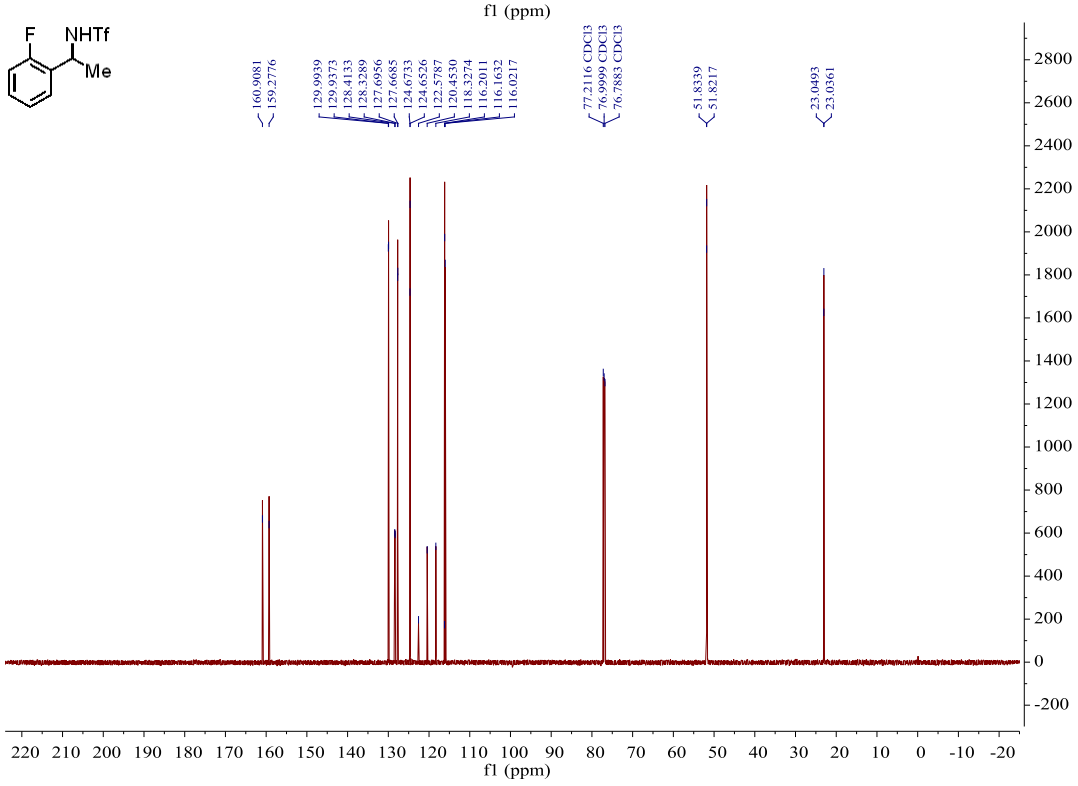

Me NHTf
7.2591
7.2541
7.2466
7.2323
7.2197
7.2172
7.2074
7.2044
7.1957
7.1928
7.1700
7.1578
5.5594
4.5344
4.5213
4.5081
2.3450
1.3735
1.3594
1.3517
1.3379
1.3320
1.3201
0.6886
0.6737
0.6670
0.6601
0.6540
0.5678
0.5606
0.5456
0.5345
0.3456
0.3375
0.3295
0.3210
0.3184
0.3141
0.3100
0.3074
0.2990
0.2911
0.2828
3.22
1.02
0.98
1.00
3.12
1.01
1.04
1.10
2.08
f1 (ppm)

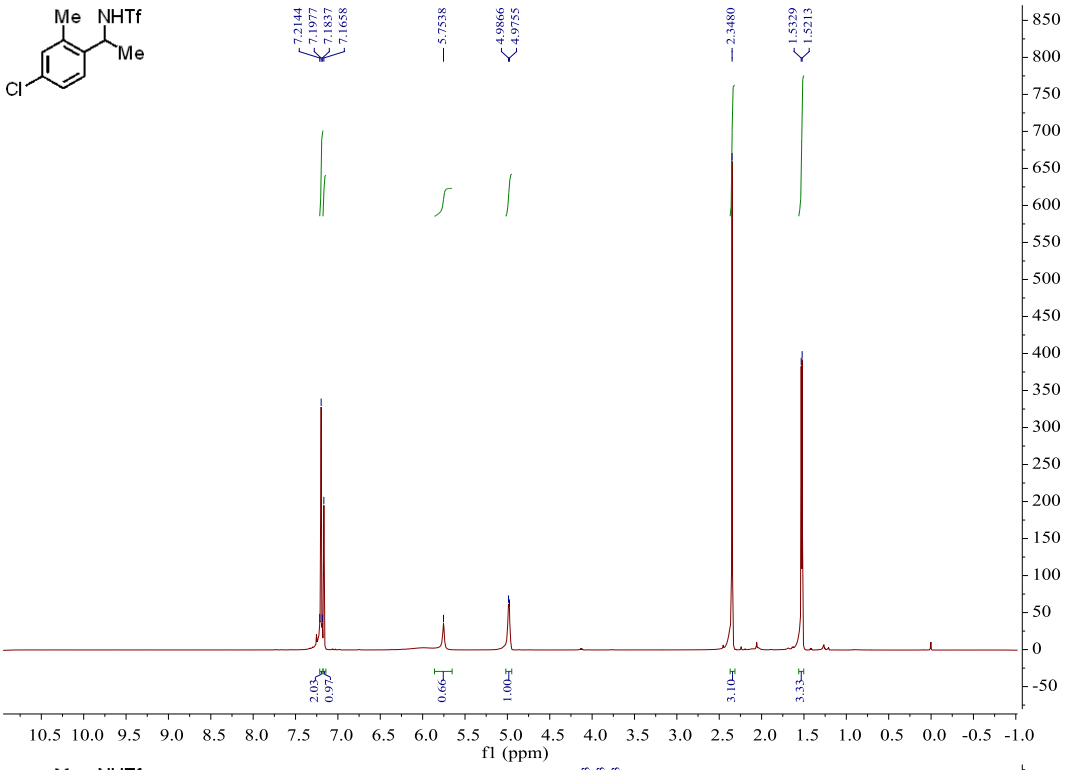

Me NHTf
137.2556
134.8939
130.6869
127.9613
126.4104
125.9483
122.5981
120.4705
118.3426
116.2165
77.2114
76.9999
76.7883
59.1894
19.2495
17.2004
4.4827
2.6997
f1 (ppm)

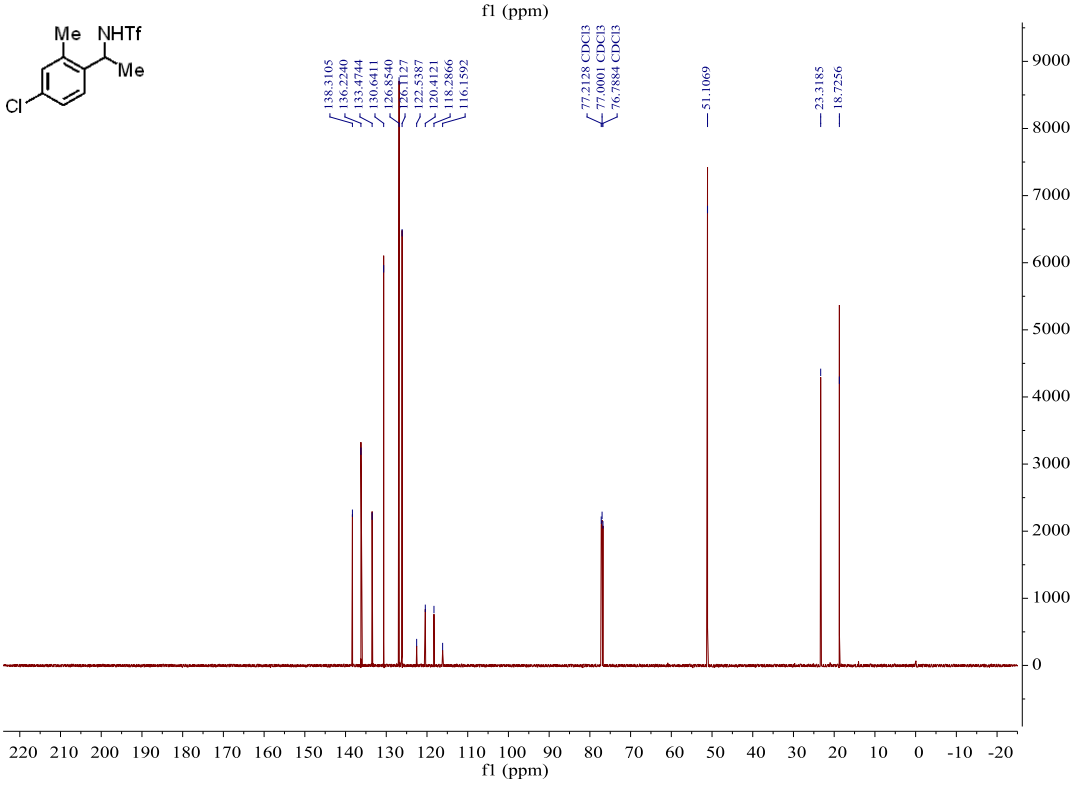

OMeNHTf
Me
7.3161
7.3026
7.2899
7.1389
7.1268
6.9567
6.9438
6.9304
6.1366
6.1220
4.7695
4.7577
4.7454
4.7310
4.7193
3.9022
1.6137
1.6021
0.97
1.00
1.97
0.98
1.00
3.08
3.29
f1 (ppm)

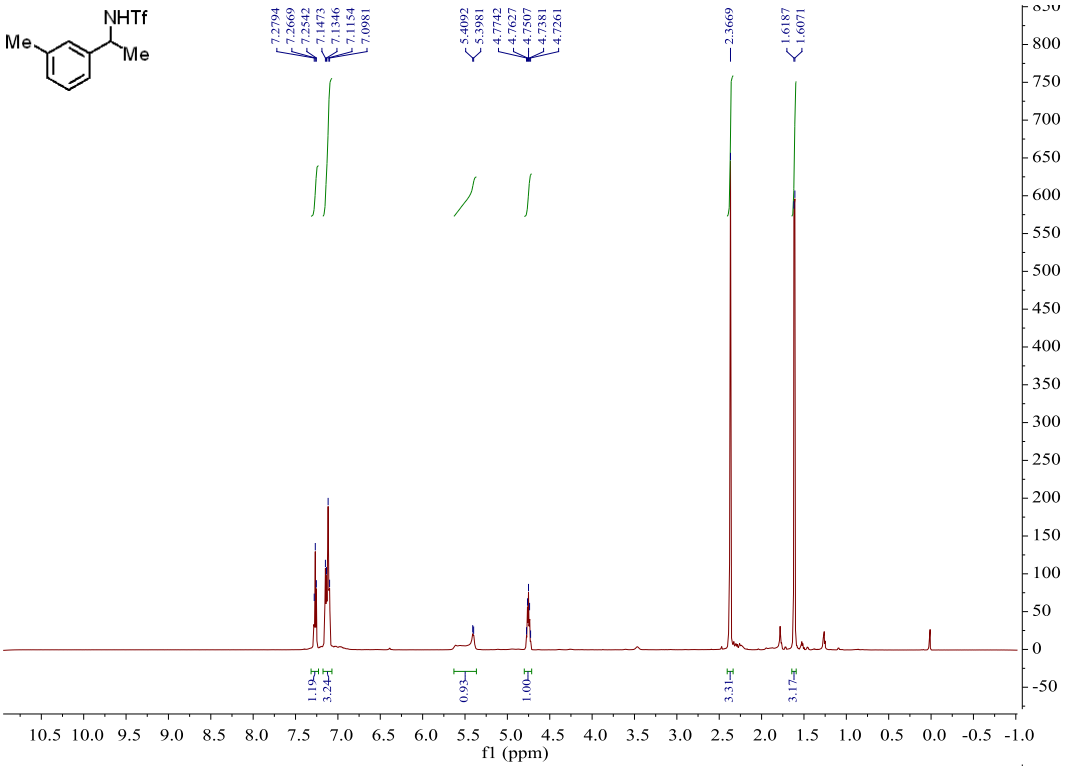

OMeNHTf
Me
156.5556
129.4147
128.7037
128.0819
122.6613
121.1527
120.5337
118.4068
116.2794
111.2785
77.2116
76.9998
76.7881
55.3966
55.1358
22.9459
f1 (ppm)

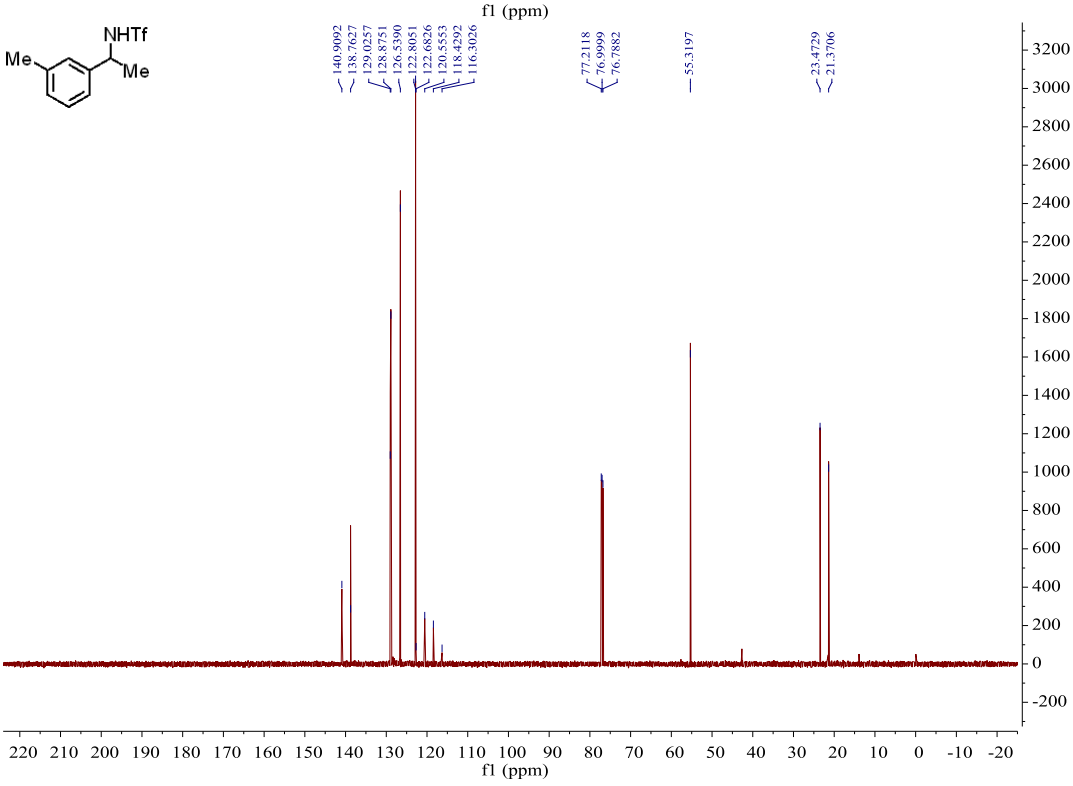

F
NHTf
Me
7.3237
7.3127
7.3010
7.2914
7.2754
7.2628
7.2524
7.1546
7.1421
7.1308
7.0961
7.0792
7.0640
5.7842
4.9392
4.9274
4.9156
4.9040
1.6353
1.6233
1.12
1.07
1.07
1.05
0.84
1.00
3.12
f1 (ppm)


F
NHTf
Me
160.9081
159.2776
129.9939
129.9373
128.4133
128.3289
127.6956
127.6685
124.6733
124.6526
122.5787
120.4530
118.3274
116.2011
116.1632
116.0217
77.2116 CDCl3
76.9999 CDCl3
76.7883 CDCl3
51.8339
51.8217
23.0493
23.0361
f1 (ppm)


Me
NHTf
Me
Cl
7.2144
7.1977
7.1837
7.1658
5.7538
4.9866
4.9755
2.3480
1.5329
1.5213
2.03
0.97
0.66
1.00
3.10
3.33
f1 (ppm)


Me
NHTf
Me
Cl
138.3105
136.2240
133.4744
130.6411
126.8540
126.1127
122.5387
120.4121
118.2866
116.1592
77.2128 CDCl3
77.0001 CDCl3
76.7884 CDCl3
51.1069
23.3185
18.7256
f1 (ppm)


NHTf
Me
Me
7.2794
7.2669
7.2542
7.1473
7.1346
7.1154
7.0981
5.4092
5.3981
4.7742
4.7627
4.7507
4.7381
4.7261
2.3669
1.6187
1.6071
1.19
3.24
0.93
1.00
3.31
3.17
f1 (ppm)


NHTf
Me
Me
140.9092
138.7627
129.0257
128.8751
126.5390
122.8051
122.6826
120.5553
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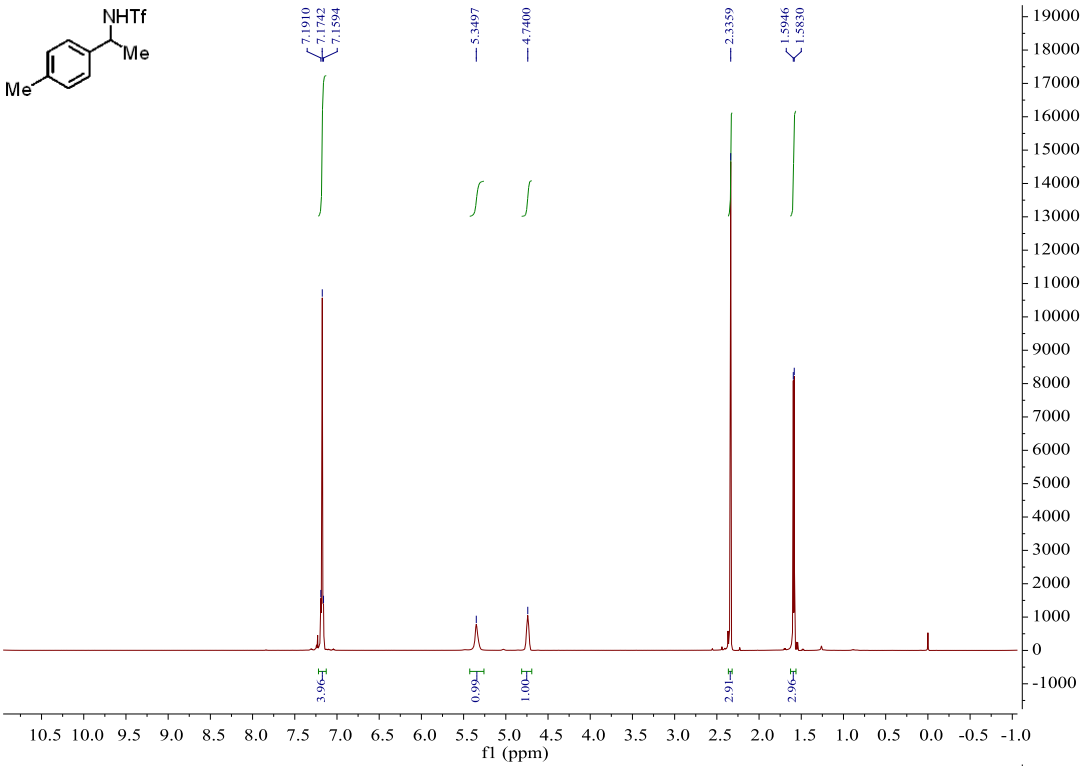
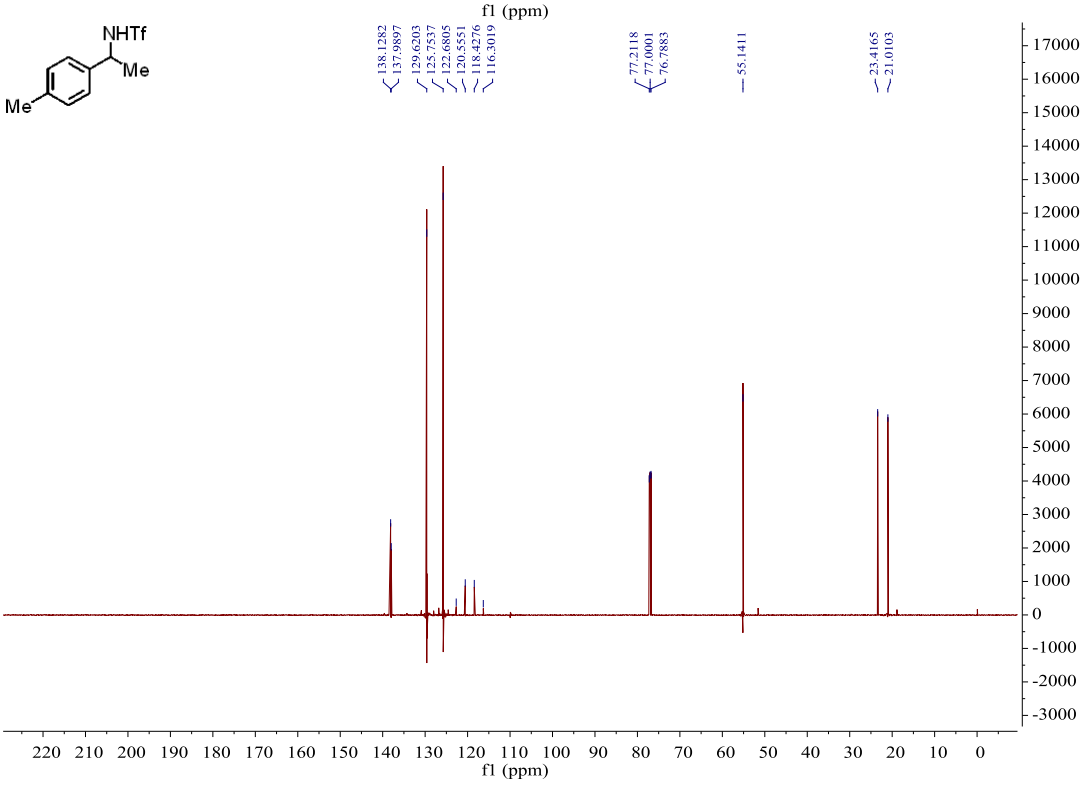
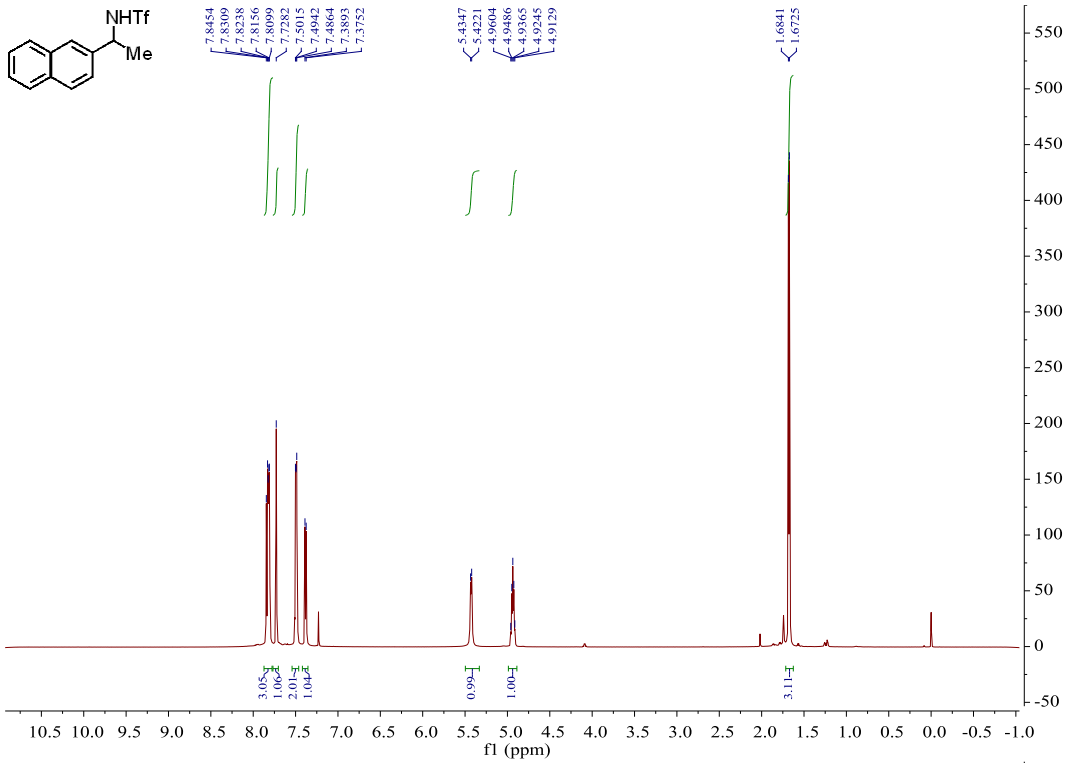
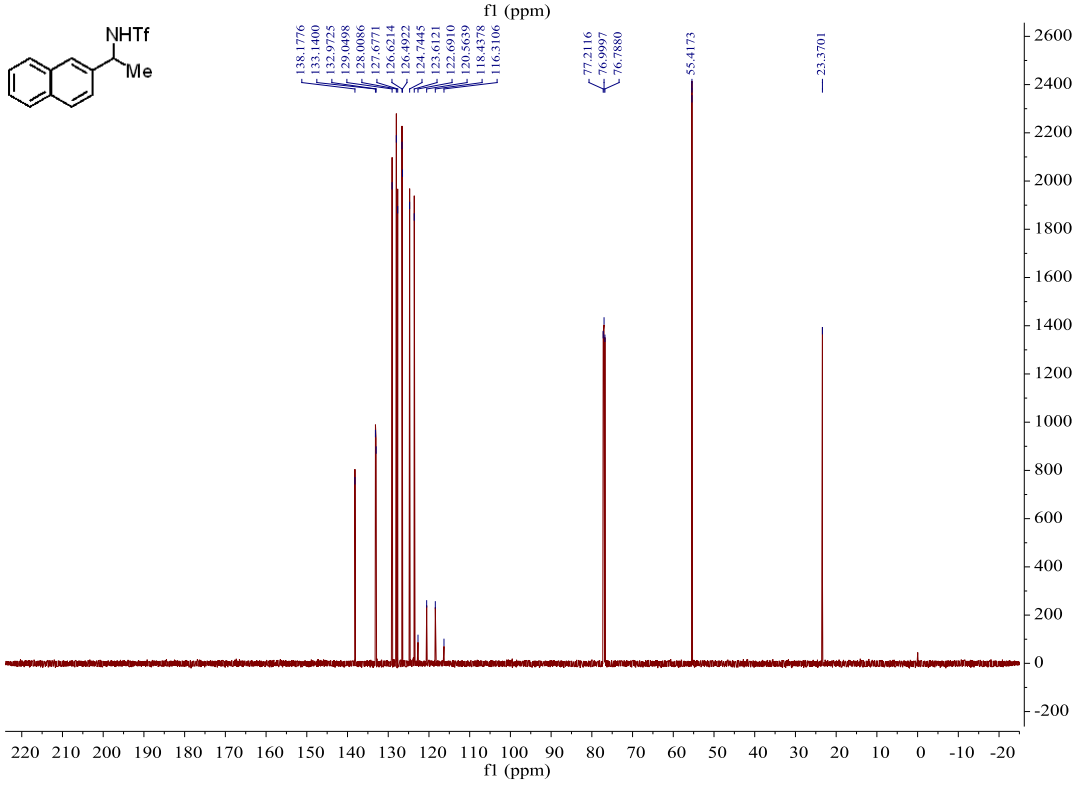
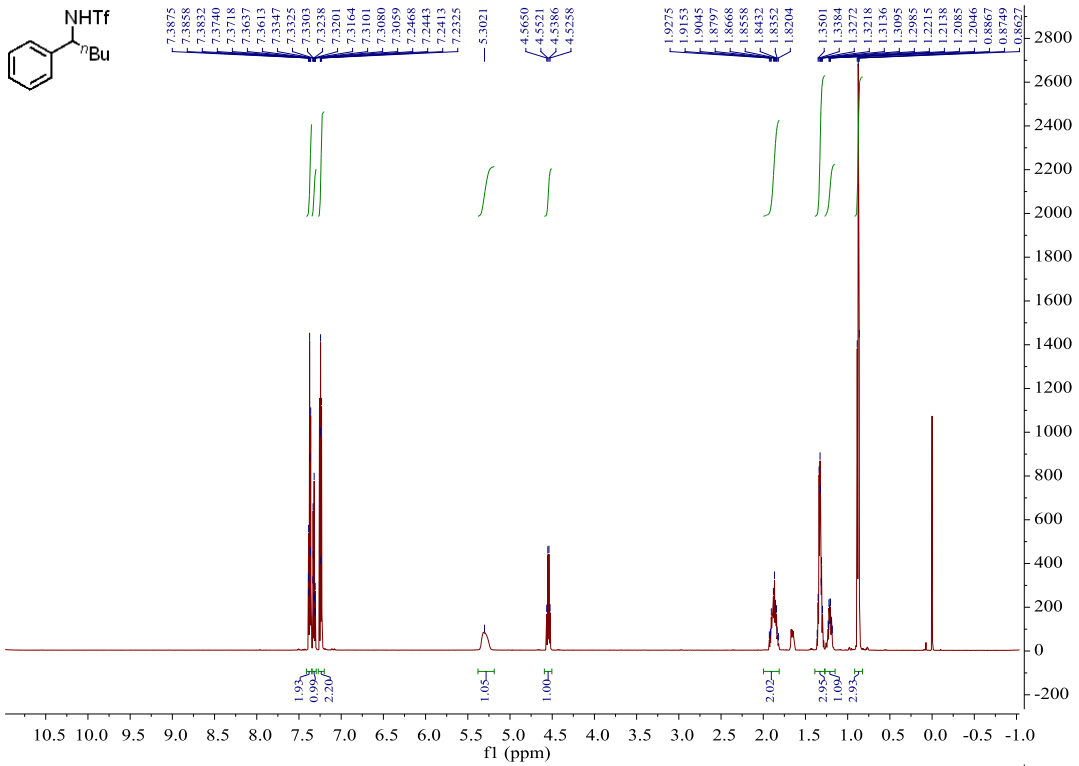
116.3026
77.2118
76.9999
76.7882
55.3197
23.4729
21.3706
f1 (ppm)


NHTf
Me
Me
7.1910
7.1742
7.1594
5.3497
4.7400
2.3359
1.5946
1.5830
3.96
0.99
1.00
2.91
2.96
f1 (ppm)


NHTf
Me
Me
138.1282
137.9897
129.6203
125.7537
122.6805
120.5551
118.4276
116.3019
77.2118
77.0001
76.7883
55.1411
23.4165
21.0103
f1 (ppm)


NHTf
Me
7.8454
7.8309
7.8238
7.8156
7.8099
7.7282
7.5015
7.4942
7.4864
7.3893
7.3752
5.4347
5.4221
4.9604
4.9486
4.9365
4.9245
4.9129
1.6841
1.6725
3.05
1.06
2.01
1.04
0.99
1.00
3.11
f1 (ppm)


NHTf
Me
138.1776
133.1400
132.9725
129.0498
128.0086
127.6771
126.6214
126.4922
124.7445
123.6121
122.6910
120.5639
118.4378
116.3106
77.2116
76.9997
76.7880
55.4173
23.3701
f1 (ppm)


NHTf
nBu
7.3875
7.3858
7.3832
7.3740
7.3718
7.3637
7.3613
7.3347
7.3325
7.3303
7.3238
7.3201
7.3164
7.3101
7.3080
7.3059
7.2468
7.2443
7.2413
7.2325
5.3021
4.5650
4.5521
4.5386
4.5258
1.9275
1.9153
1.9045
1.8797
1.8668
1.8558
1.8432
1.8352
1.8204
1.3501
1.3384
1.3272
1.3218
1.3136
1.3095
1.2985
1.2215
1.2138
1.2085
1.2046
0.8867
0.8749
0.8627
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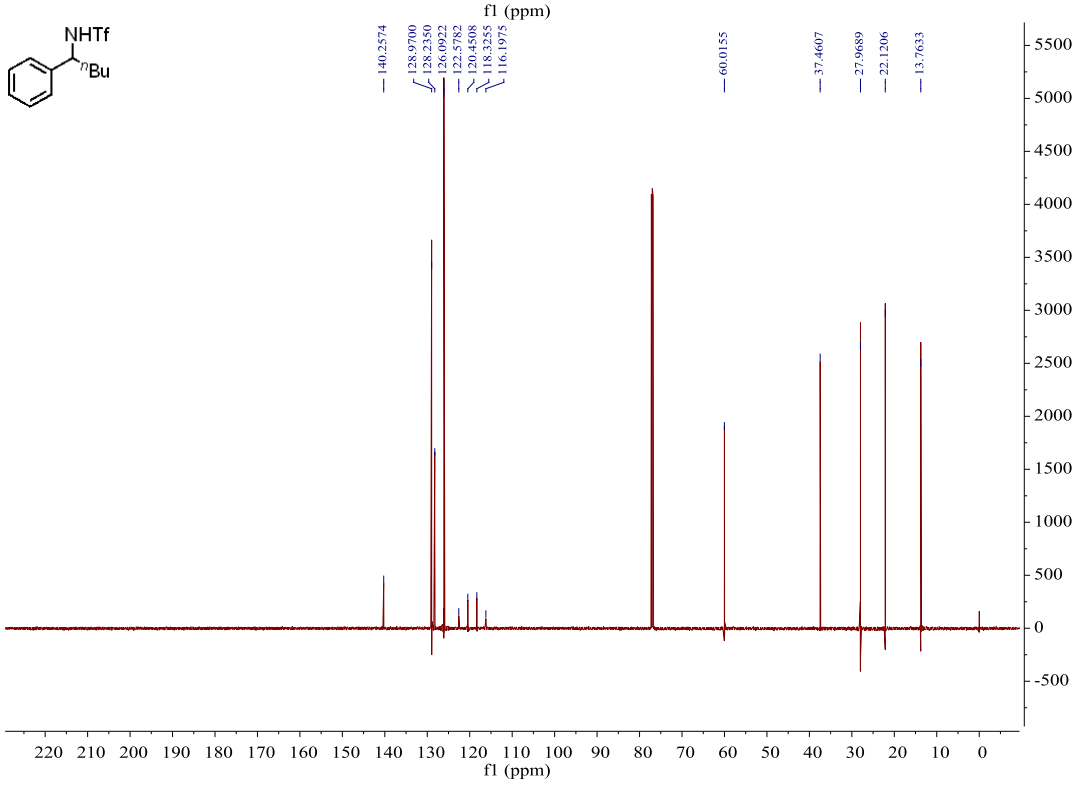
0.99
2.20
1.05
1.00
2.02
2.95
1.09
2.93
f1 (ppm)


NHTf
nBu
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128.2350
126.0922
122.5782
120.4508
118.3255
116.1975
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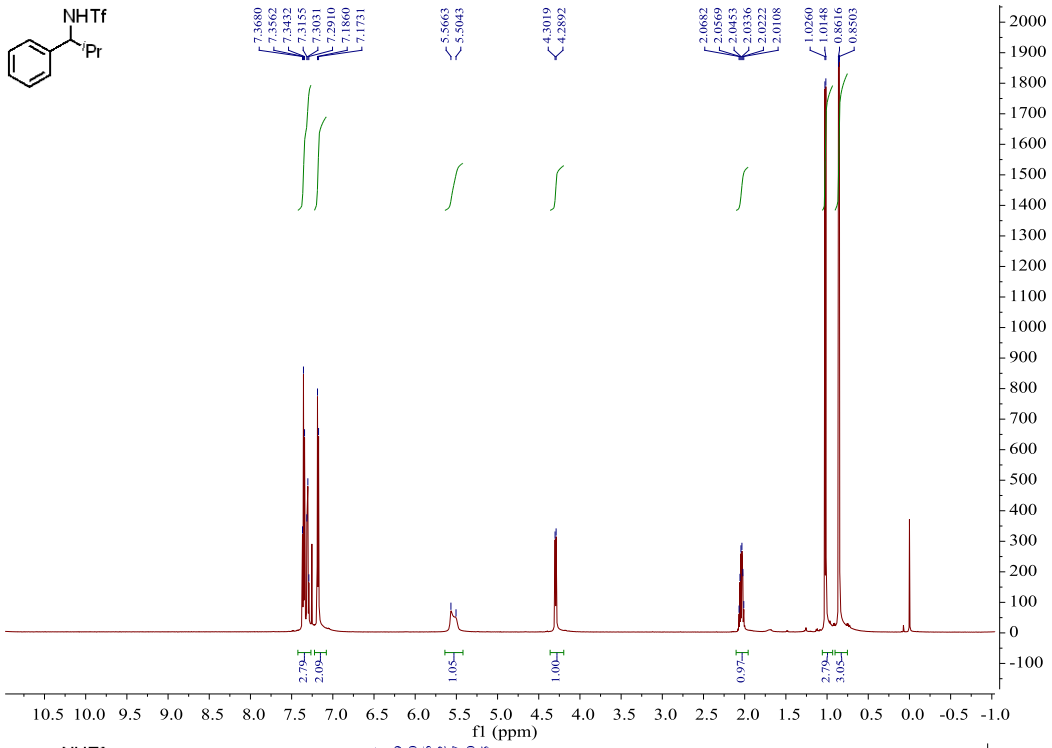
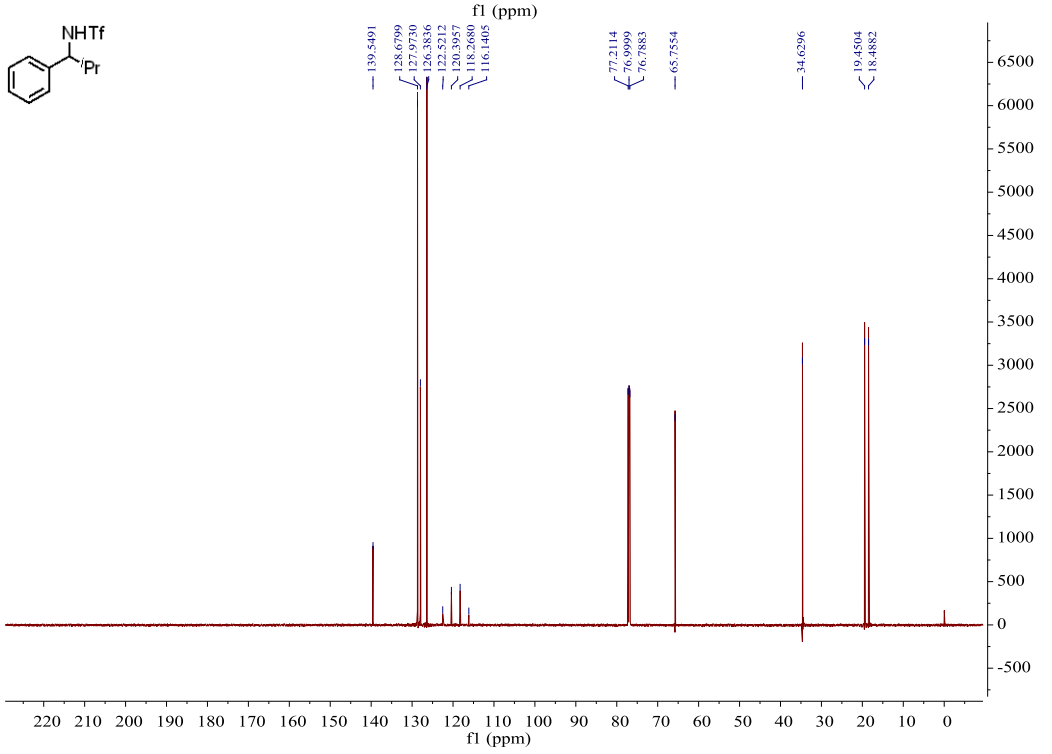
27.9689
22.1206
13.7633
f1 (ppm)


NHTf
iPr
7.3680
7.3562
7.3432
7.3155
7.3031
7.2910
7.1860
7.1731
5.5663
5.5043
4.3019
4.2892
2.0682
2.0569
2.0453
2.0336
2.0222
2.0108
1.0260
1.0148
0.8616
0.8503
2.79
2.09
1.05
1.00
0.97
2.79
3.03
f1 (ppm)


NHTf
iPr
139.5491
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127.9730
126.3836
122.5212
120.3957
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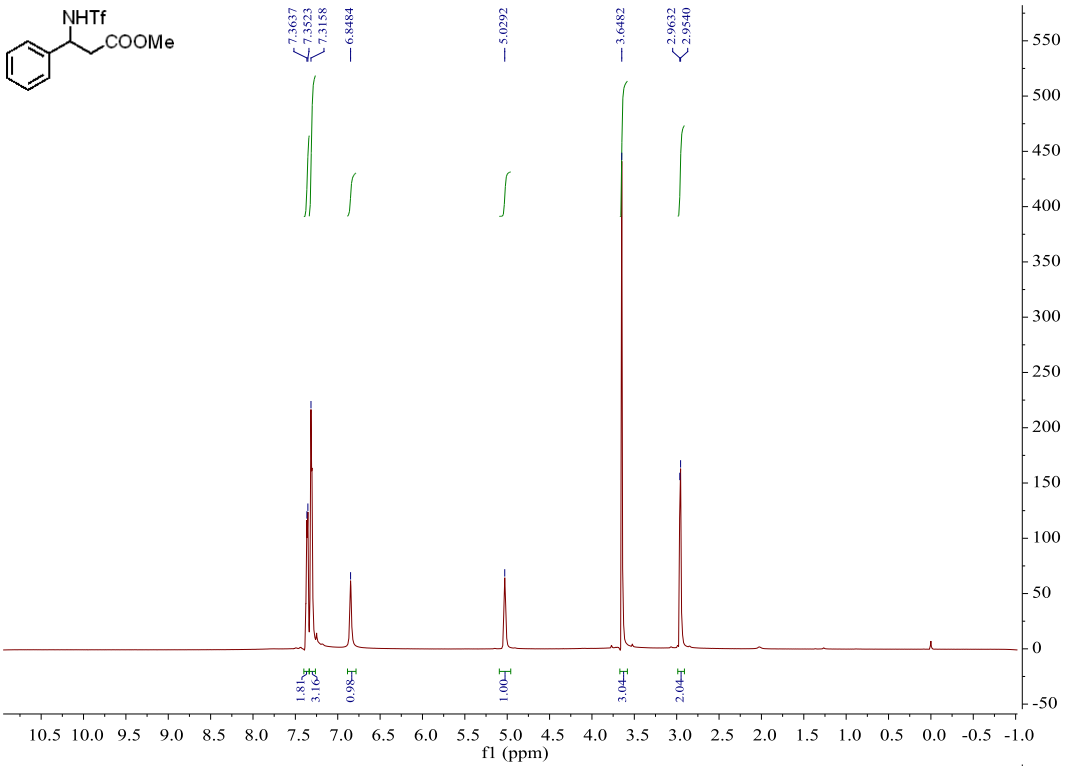
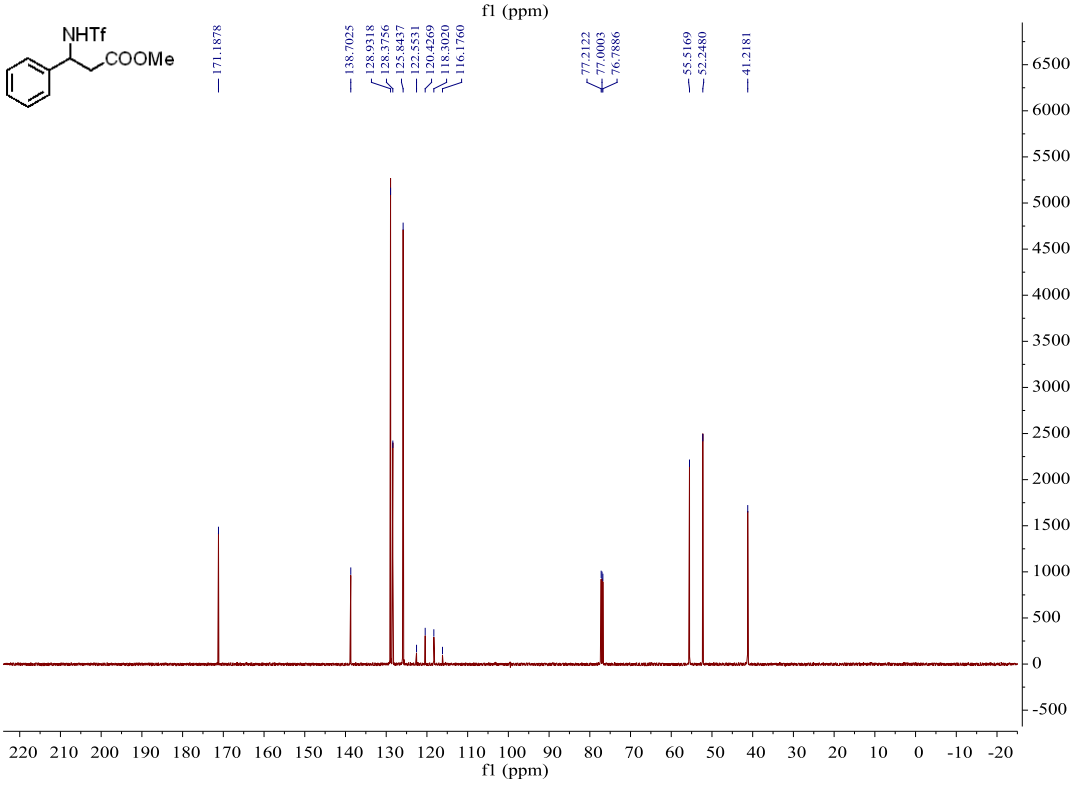
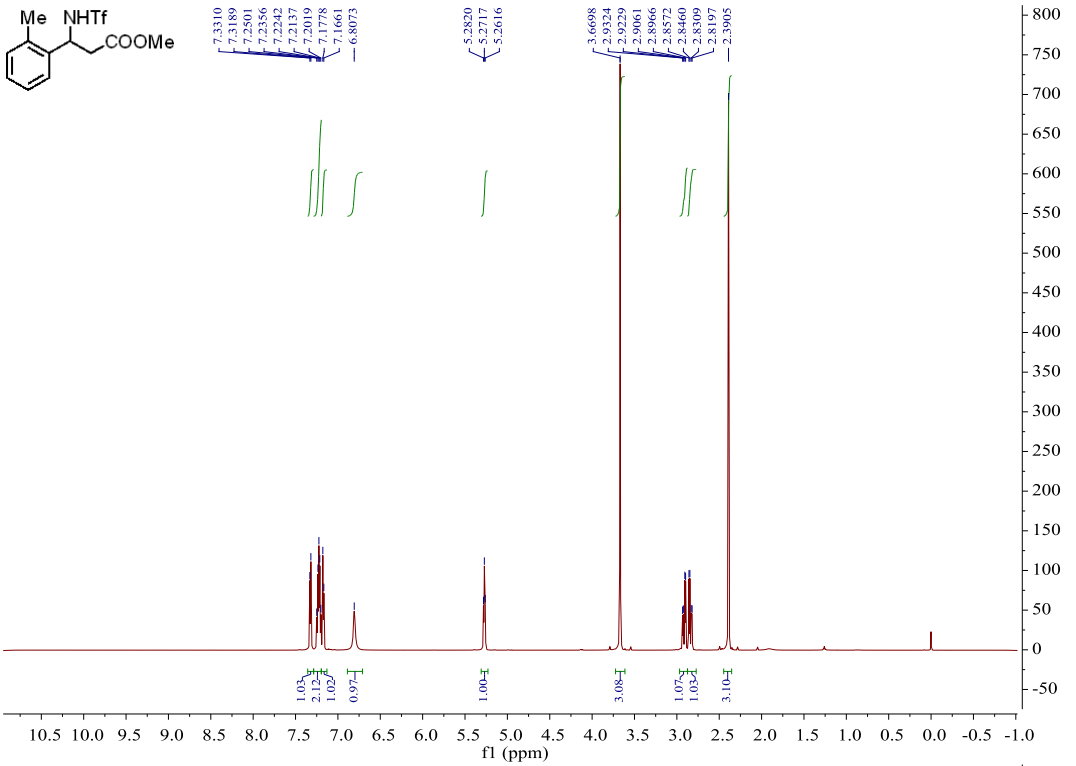
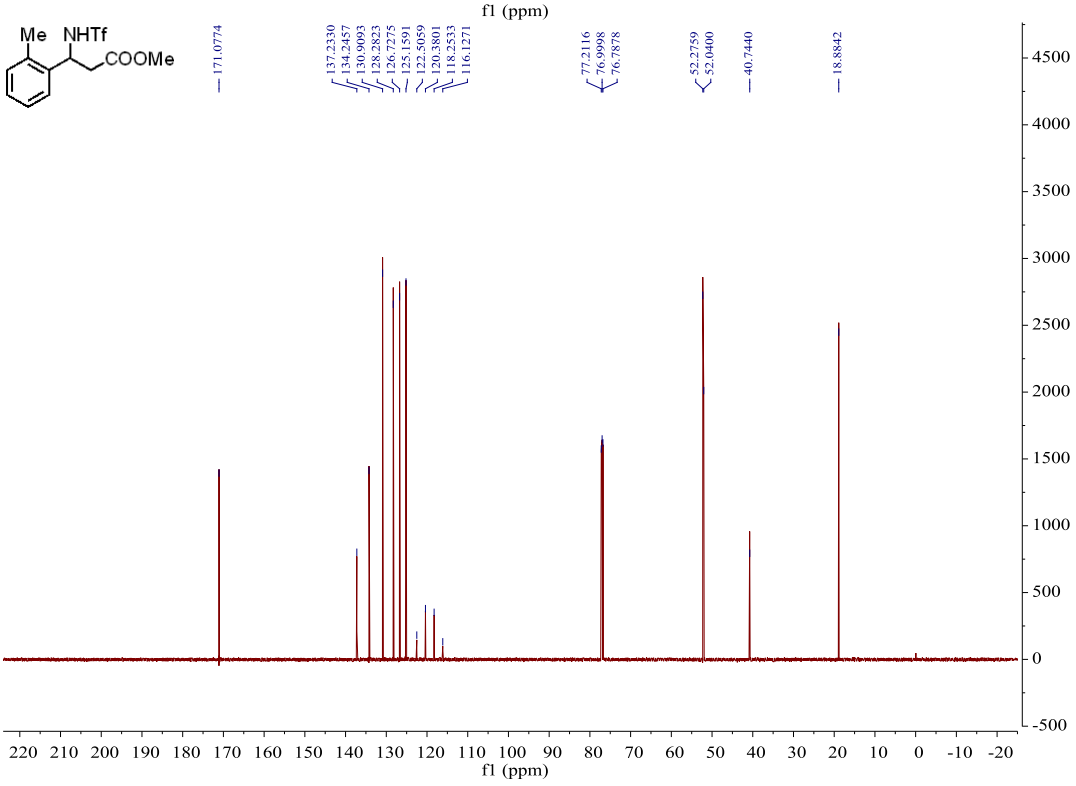
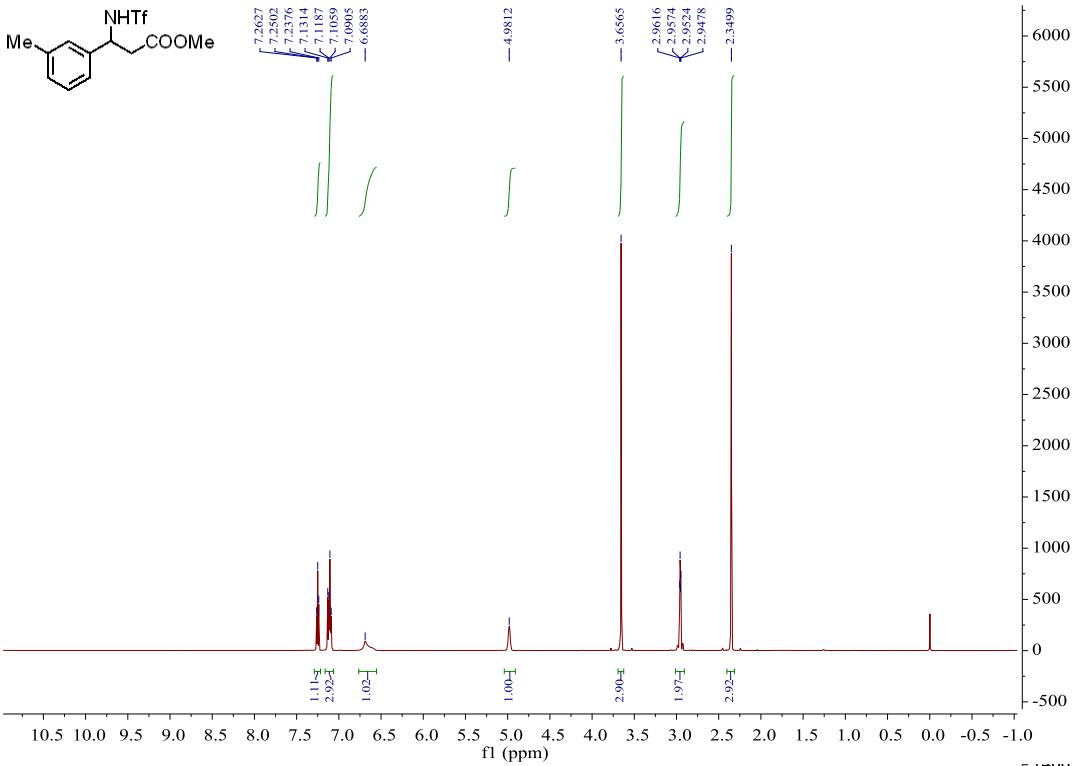
77.2114
76.9999
76.7883
65.7554
34.6296
19.4504
18.4882
f1 (ppm)

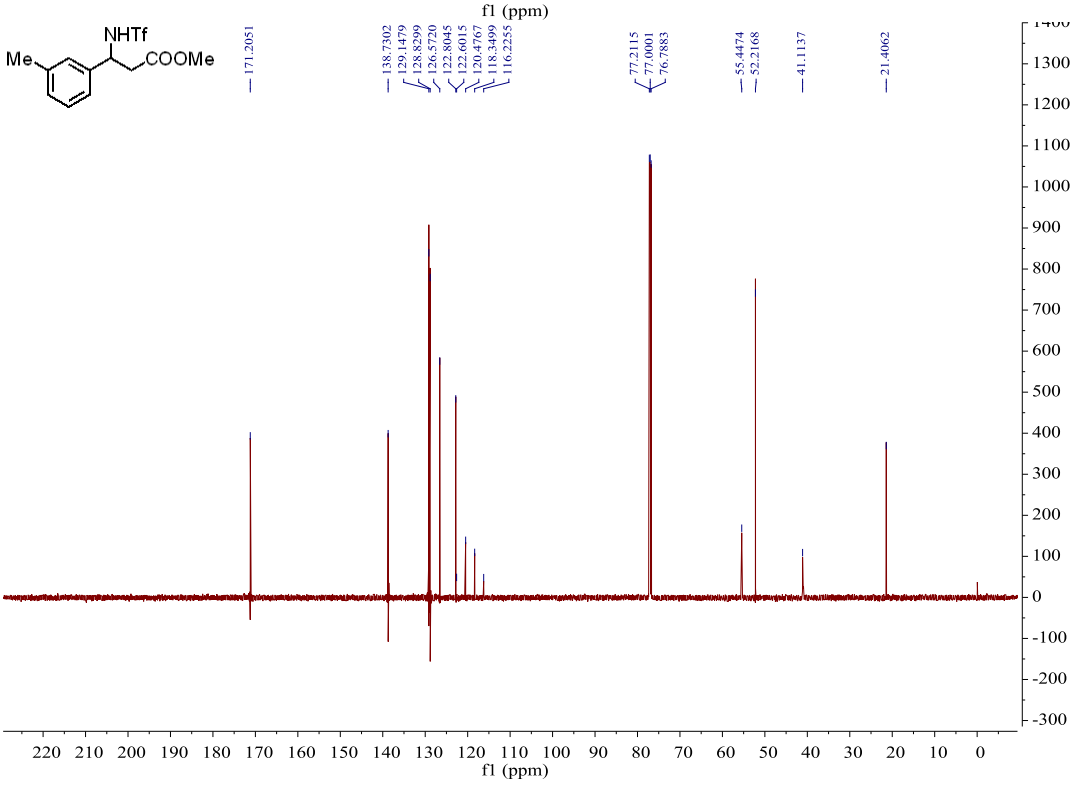

NHTf
COOMe
7.3637
7.3523
7.3158
6.8484
5.0292
3.6482
2.9632
2.9540
1.81
3.16
0.98
1.00
3.04
2.04
f1 (ppm)


NHTf
COOMe
171.1878
138.7025
128.9318
128.3756
125.8437
122.5531
120.4269
118.3020
116.1760
77.2122
77.0003
76.7886
55.5169
52.2480
41.2181
f1 (ppm)


Me NHTf COOMe
7.3310
7.3189
7.2501
7.2356
7.2242
7.2137
7.2019
7.1778
7.1661
6.8073
5.2820
5.2717
5.2616
3.6698
2.9324
2.9229
2.9061
2.8966
2.8572
2.8460
2.8309
2.8197
2.3905
1.03
2.12
1.02
0.97
1.00
3.08
1.07
1.03
3.10
f1 (ppm)


Me NHTf COOMe
171.0774
137.2330
134.2457
130.9093
128.2823
126.7275
125.1591
122.5059
120.3801
118.2533
116.1271
77.2116
76.9998
76.7878
52.2759
52.0400
40.7440
18.8842
f1 (ppm)


NHTf
Me
COOMe
7.2627
7.2502
7.2376
7.1314
7.1187
7.1059
7.0905
6.6883
4.9812
3.6565
2.9616
2.9574
2.9524
2.9478
2.3499
1.11
2.92
1.02
1.00
2.90
1.97
2.92
f1 (ppm)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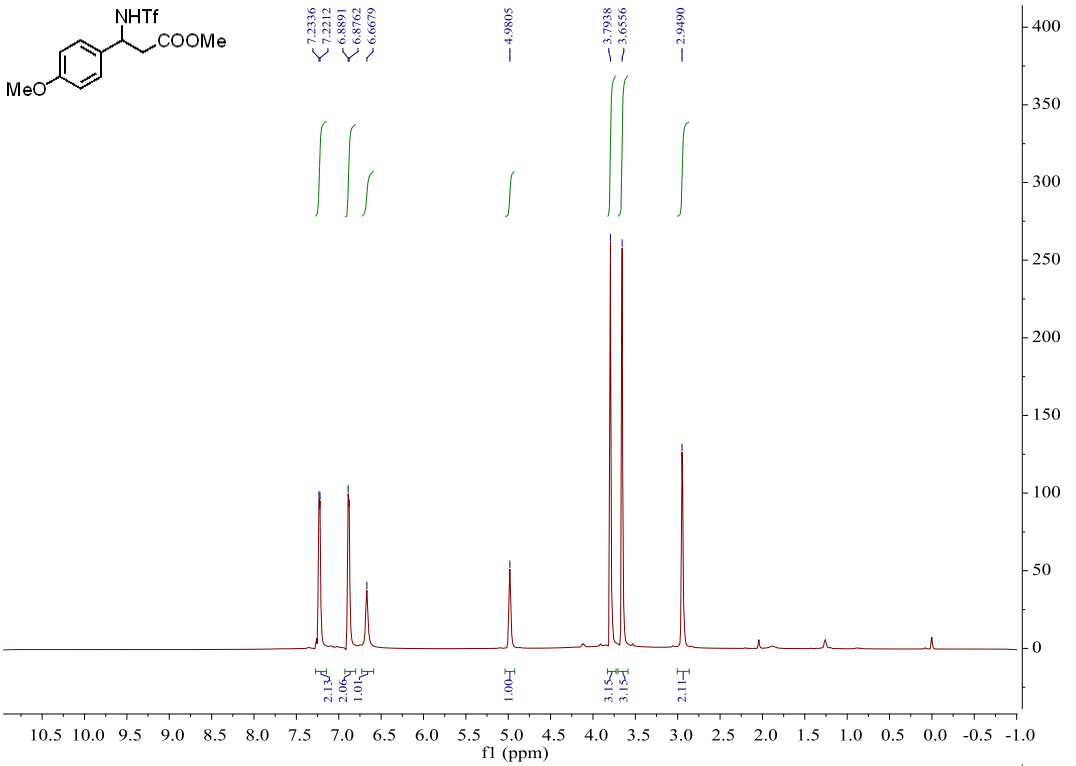
Me
COOMe
171.2051
138.7302
129.1479
128.8299
126.5720
122.8045
122.6015
120.4767
118.3499
116.2255
77.2115
77.0001
76.7883
55.4474
52.2168
41.1137
21.4062
f1 (ppm)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COOMe
MeO
7.2336
7.2212
6.8891
6.8762
6.6679
4.9805
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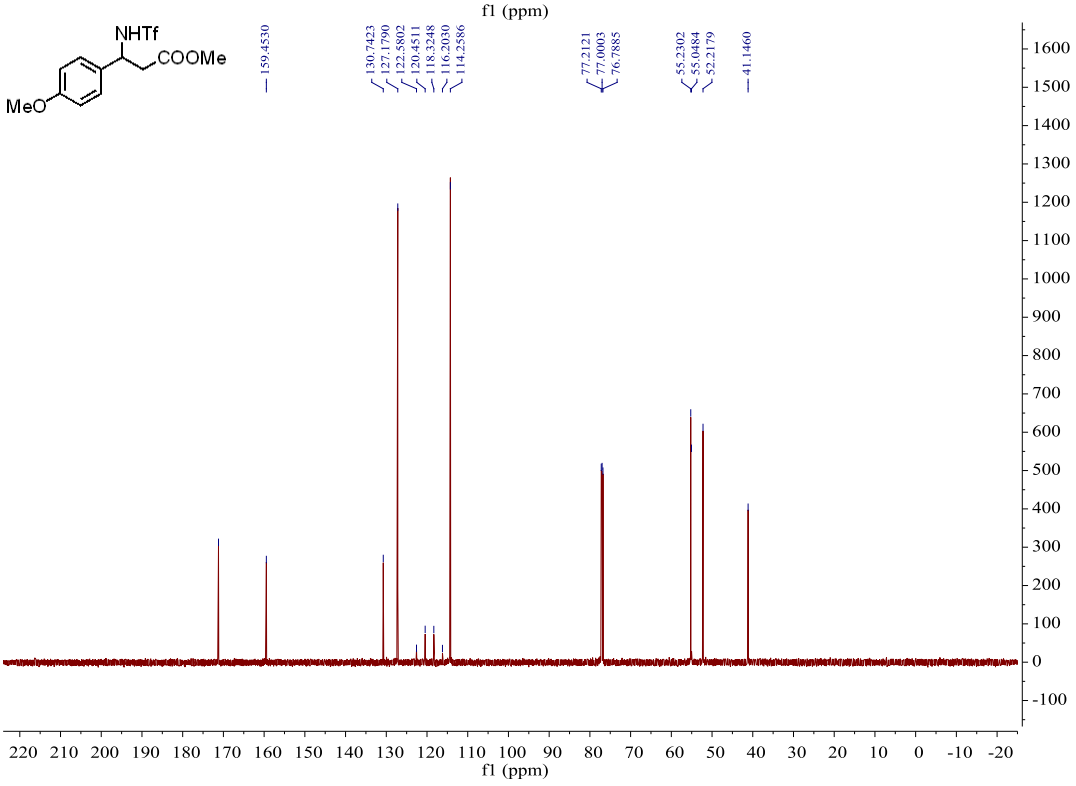
1.00
3.15
3.15
2.11
f1 (ppm)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COOMe
MeO
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127.1790
122.5802
120.4511
118.3248
116.2030
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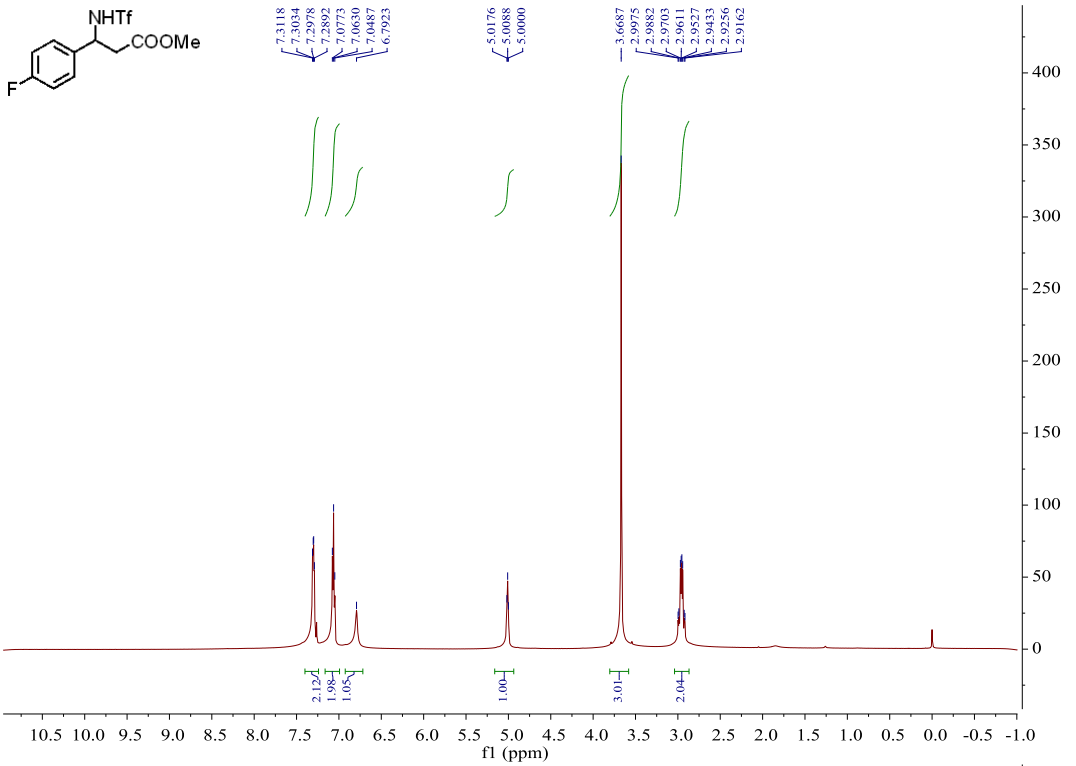
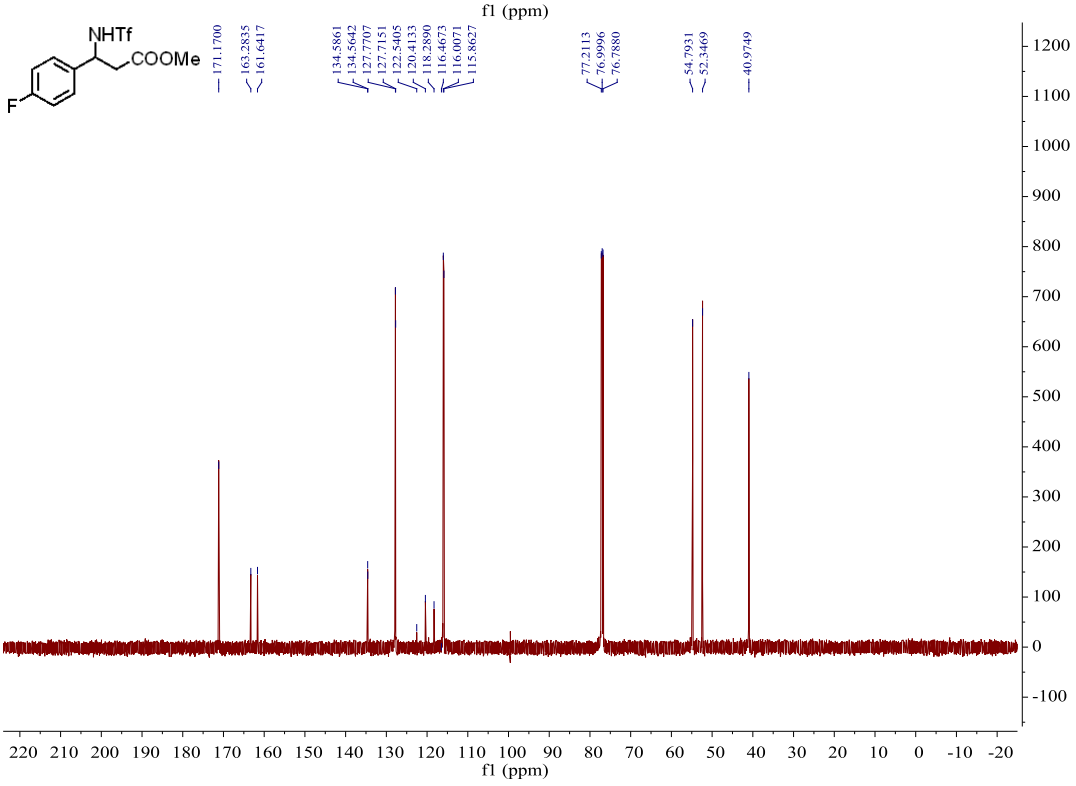
77.2121
77.0003
76.7885
55.2302
55.0484
52.2179
41.1460
f1 (ppm)


NHTf
COOMe
F
7.3118
7.3034
7.2978
7.2892
7.0773
7.0630
7.0487
6.7923
5.0176
5.0088
5.0000
3.6687
2.9975
2.9882
2.9703
2.9611
2.9527
2.9433
2.9256
2.9162
2.12
1.98
1.05
1.00
3.01
2.04
f1 (ppm)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COOMe
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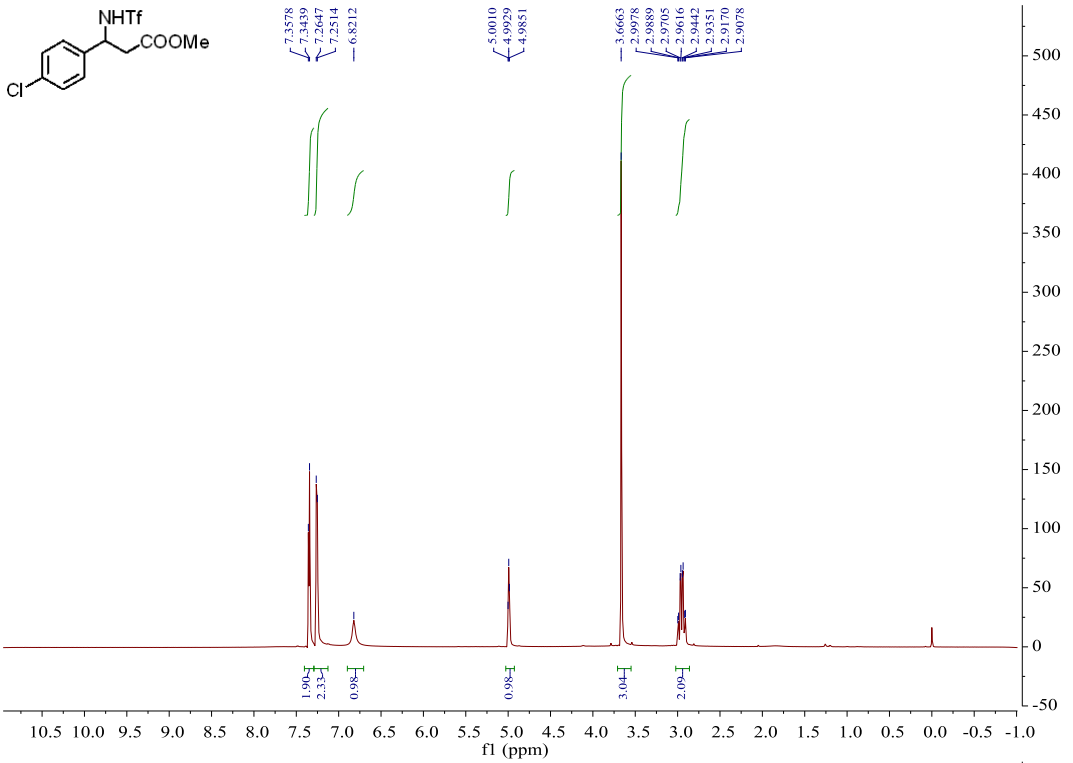
161.6417
134.5861
134.5642
127.7707
127.7151
122.5405
120.4133
118.2890
116.4673
116.0071
115.8627
77.2113
76.9996
76.7880
54.7931
52.3469
40.9749
f1 (ppm)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COOMe
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7.2514
6.8212
5.0010
4.9929
4.9851
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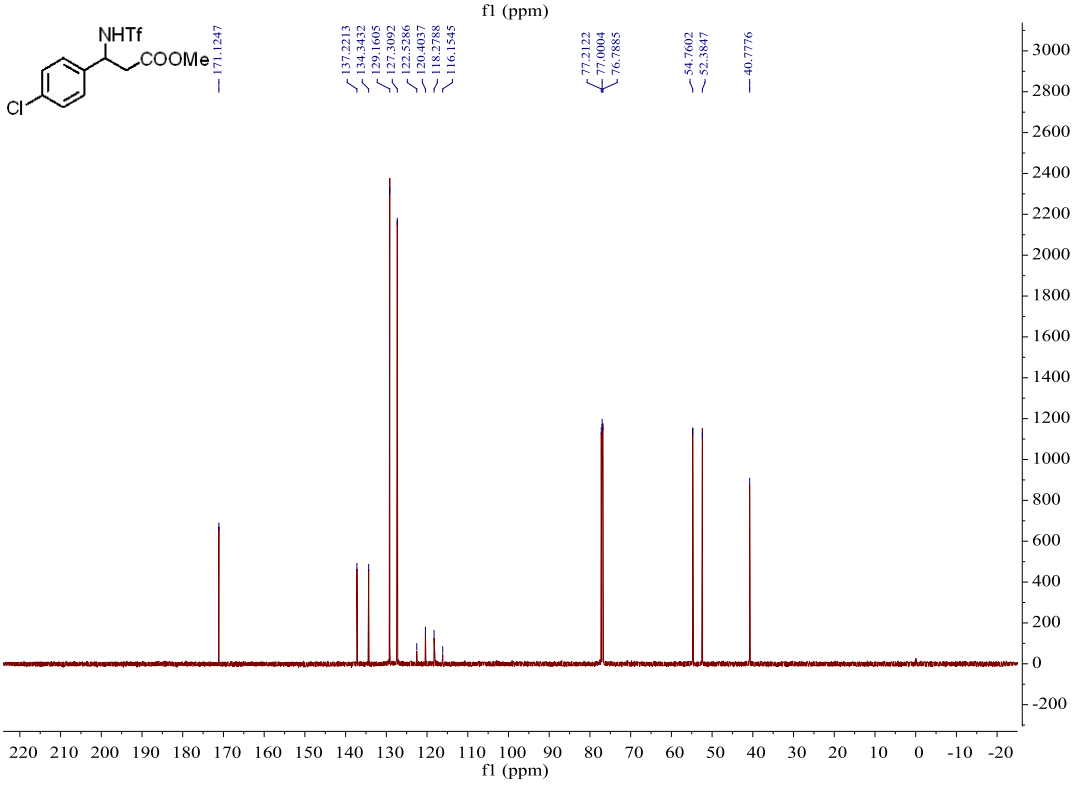
2.9978
2.9889
2.9705
2.9616
2.9442
2.9351
2.9170
2.9078
1.90
2.33
0.98
0.98
3.04
2.09
f1 (ppm)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COOMe
Cl
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134.3432
129.1605
127.3092
122.5286
120.4037
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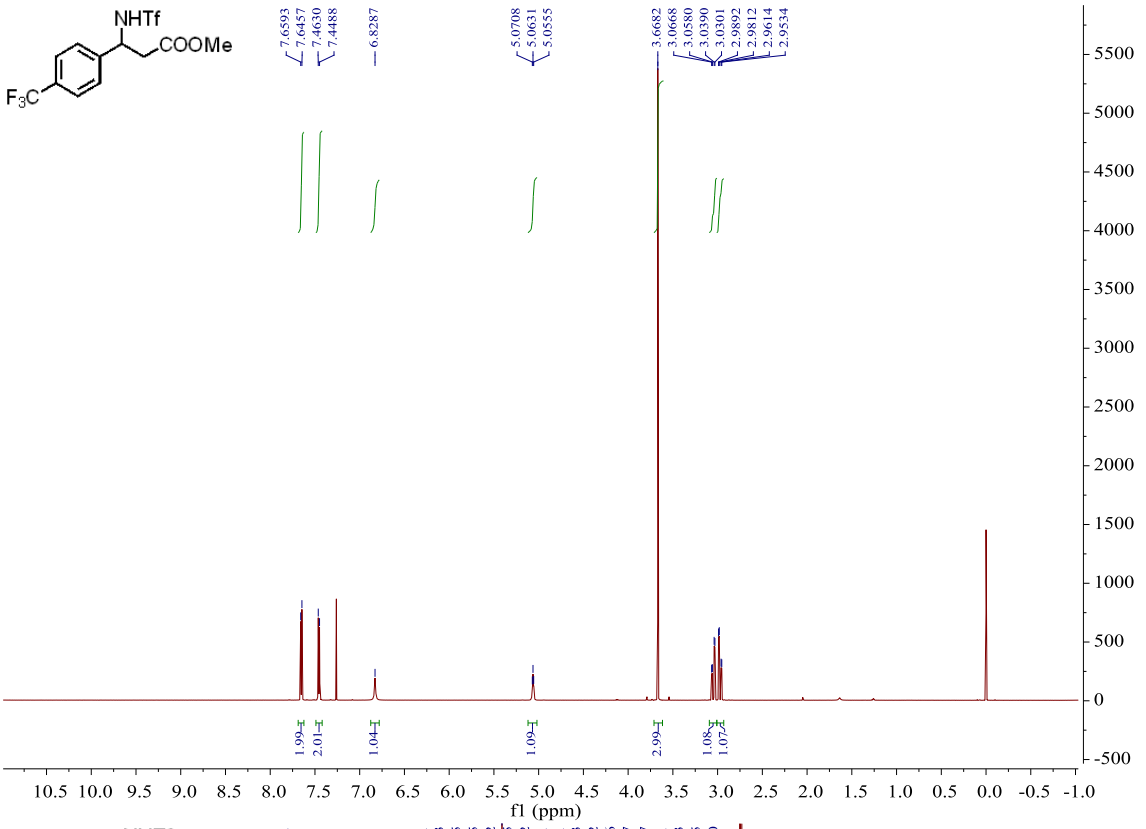
116.1545
77.2122
77.0004
76.7885
54.7602
52.3847
40.7776
f1 (ppm)


NHTf
COOMe
F3C
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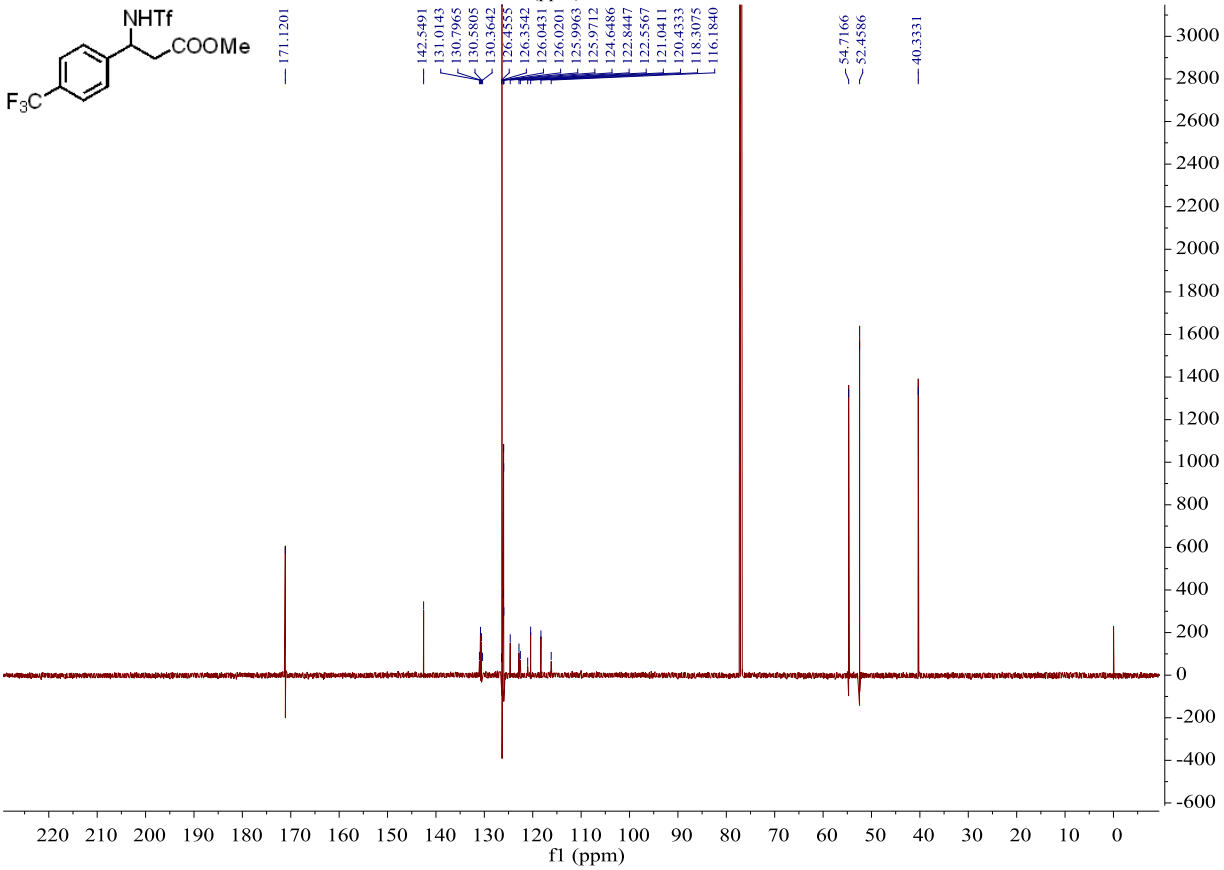
7.6457
7.4630
7.4488
6.8287
5.0708
5.0631
5.0555
3.6682
3.0668
3.0580
3.0390
3.0301
2.9892
2.9812
2.9614
2.9534
1.99
2.01
1.04
1.09
2.99
1.08
1.07
f1 (ppm)


NHTf
COOMe
F3C
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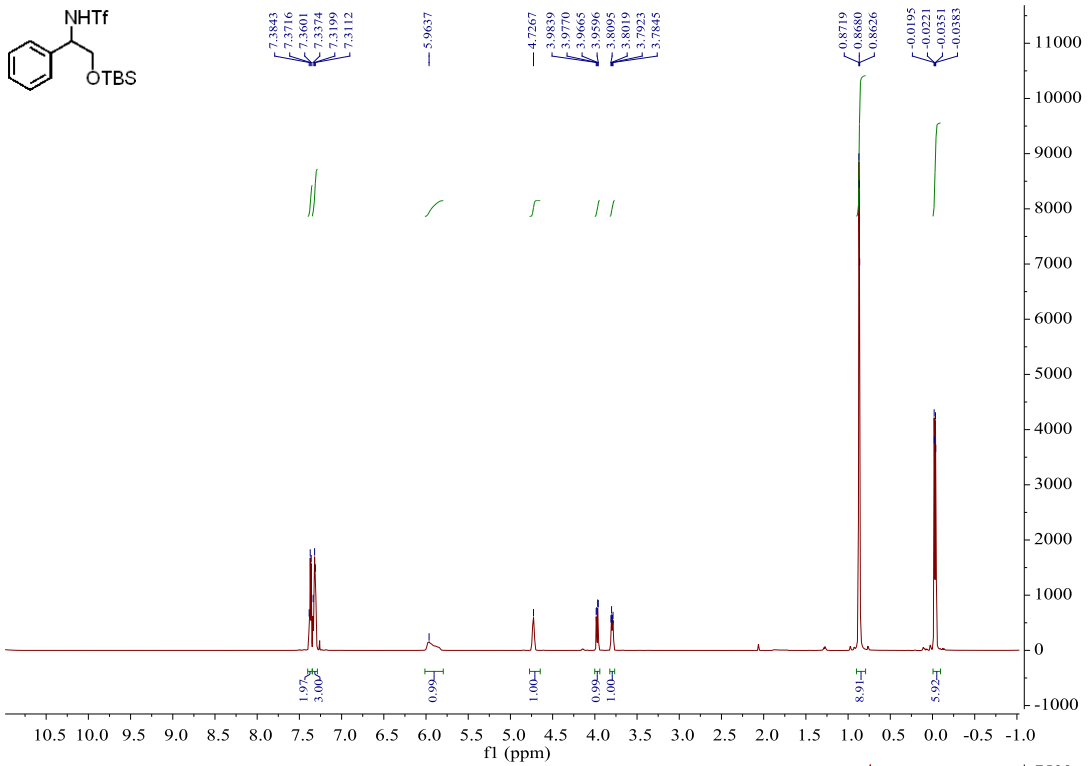
142.5491
131.0143
130.7965
130.5805
130.3642
126.4555
126.3542
126.0431
126.0201
125.9963
125.9712
124.6486
122.8447
122.5567
121.0411
120.4333
118.3075
116.1840
54.7166
52.4586
40.3331
f1 (ppm)


NHTf
OTBS
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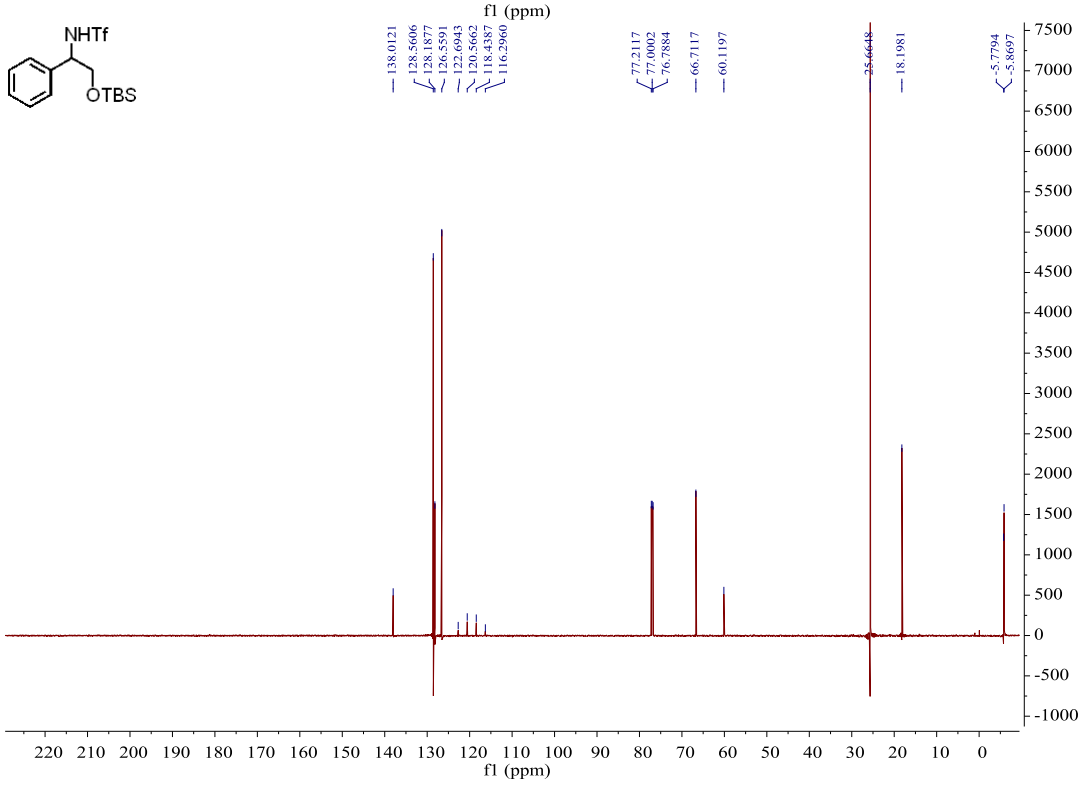
7.3716
7.3601
7.3374
7.3199
7.3112
5.9637
4.7267
3.9839
3.9770
3.9665
3.9596
3.8095
3.8019
3.7923
3.7845
0.8719
0.8680
0.8626
-0.0195
-0.0221
-0.0351
-0.0383
1.97
3.00
0.99
1.00
0.99
1.00
8.91
5.92
f1 (ppm)


NHTf
OTBS
138.0121
128.5606
128.1877
126.5591
122.6943
120.5662
118.4387
116.2960
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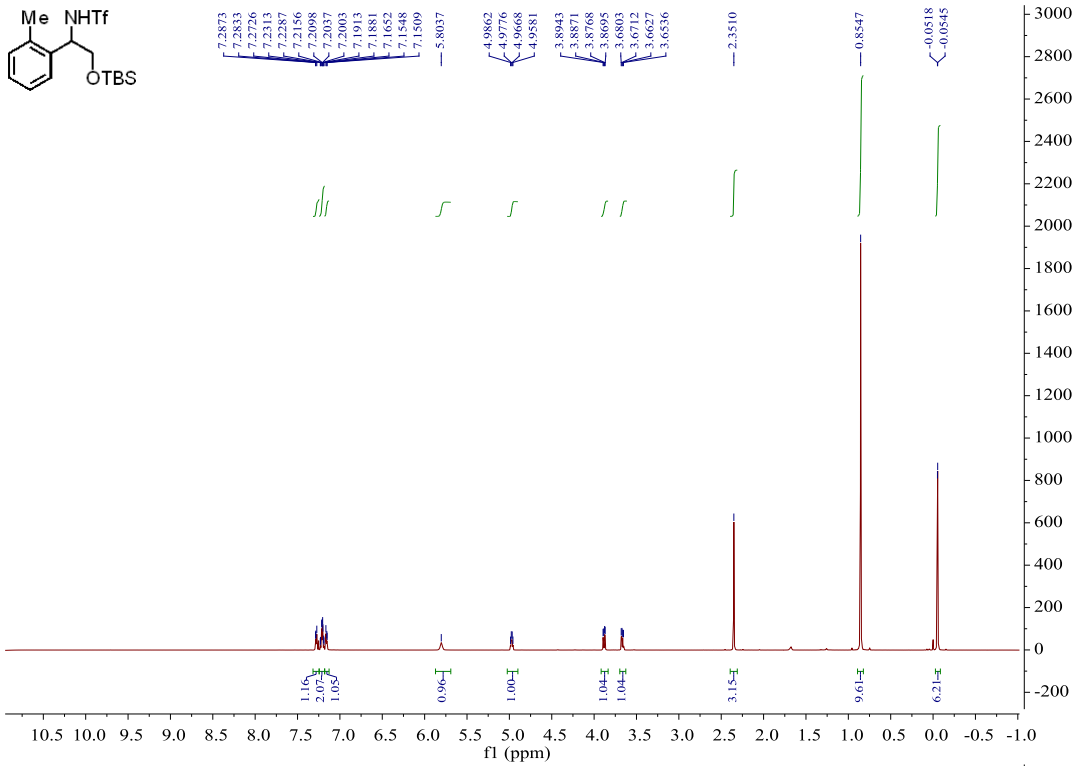
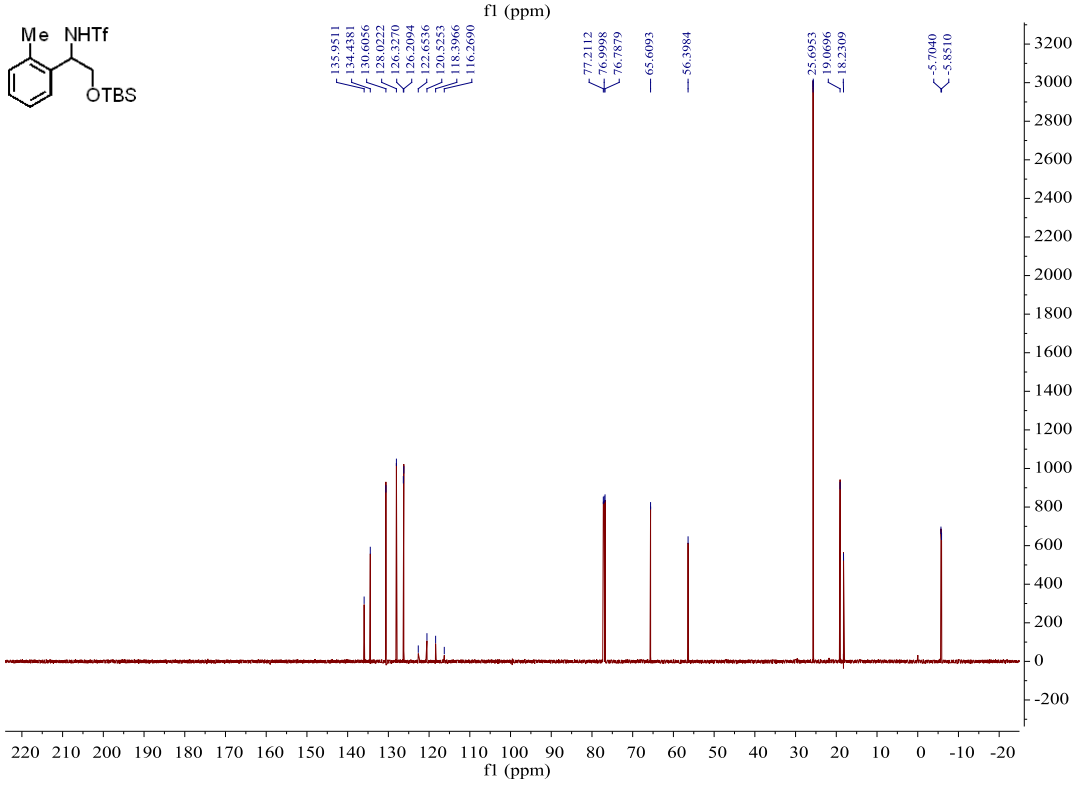
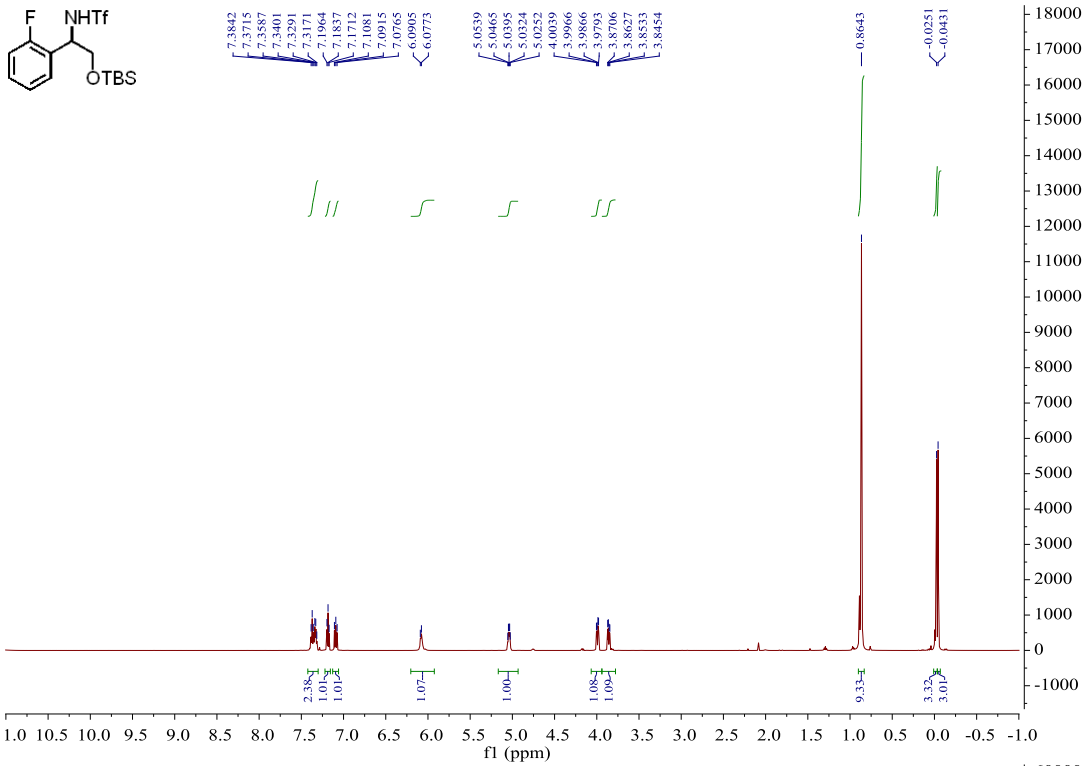
18.1981
-5.7794
-5.8697
f1 (ppm)


Me
NHTf
OTBS
f1 (ppm)


Me
NHTf
OTBS
f1 (ppm)


F
NHTf
OTBS
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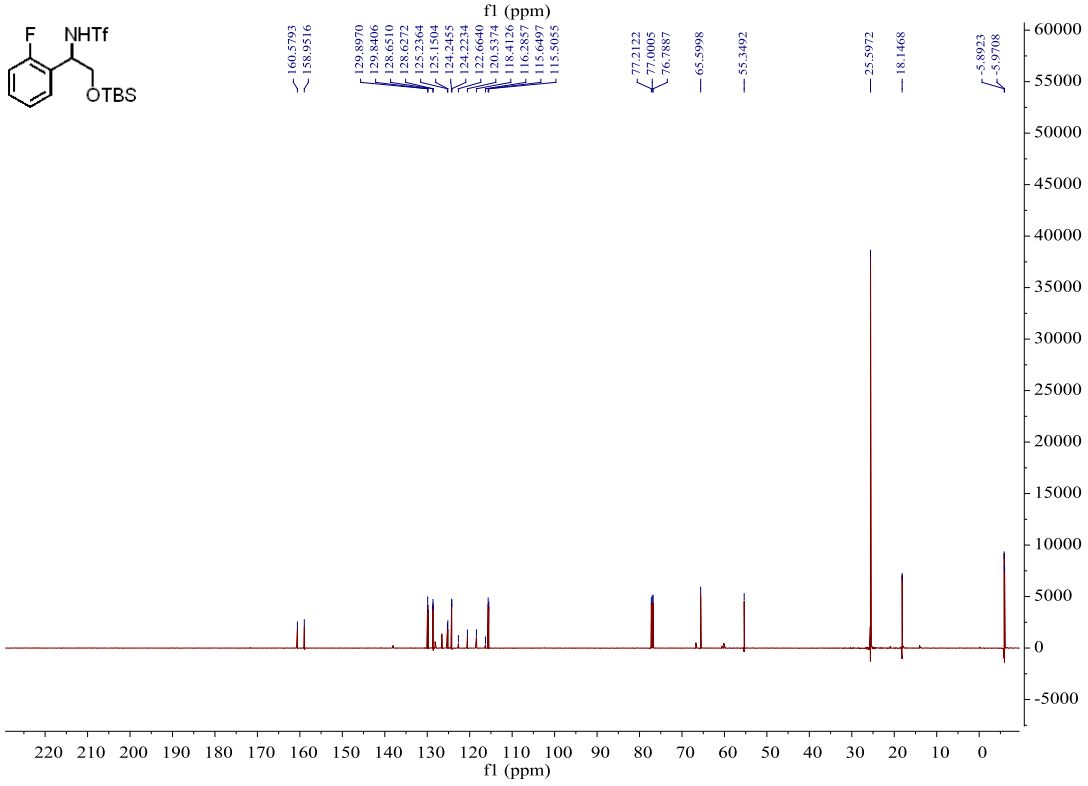
7.3715
7.3587
7.3401
7.3291
7.3171
7.1964
7.1837
7.1712
7.1081
7.0915
7.0765
6.0905
6.0773
5.0539
5.0465
5.0395
5.0324
5.0252
4.0039
3.9966
3.9866
3.9793
3.8706
3.8627
3.8533
3.8454
0.8643
-0.0251
-0.0431
2.38
1.01
1.01
1.07
1.00
1.08
1.09
9.33
3.32
3.01
f1 (ppm)


F
NHTf
OTBS
160.5793
158.9516
129.8970
129.8406
128.6510
128.6272
125.2364
125.1504
124.2455
124.2234
122.6640
120.5374
118.4126
116.2857
116.6497
115.5055
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77.0005
76.7887
65.5998
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25.5972
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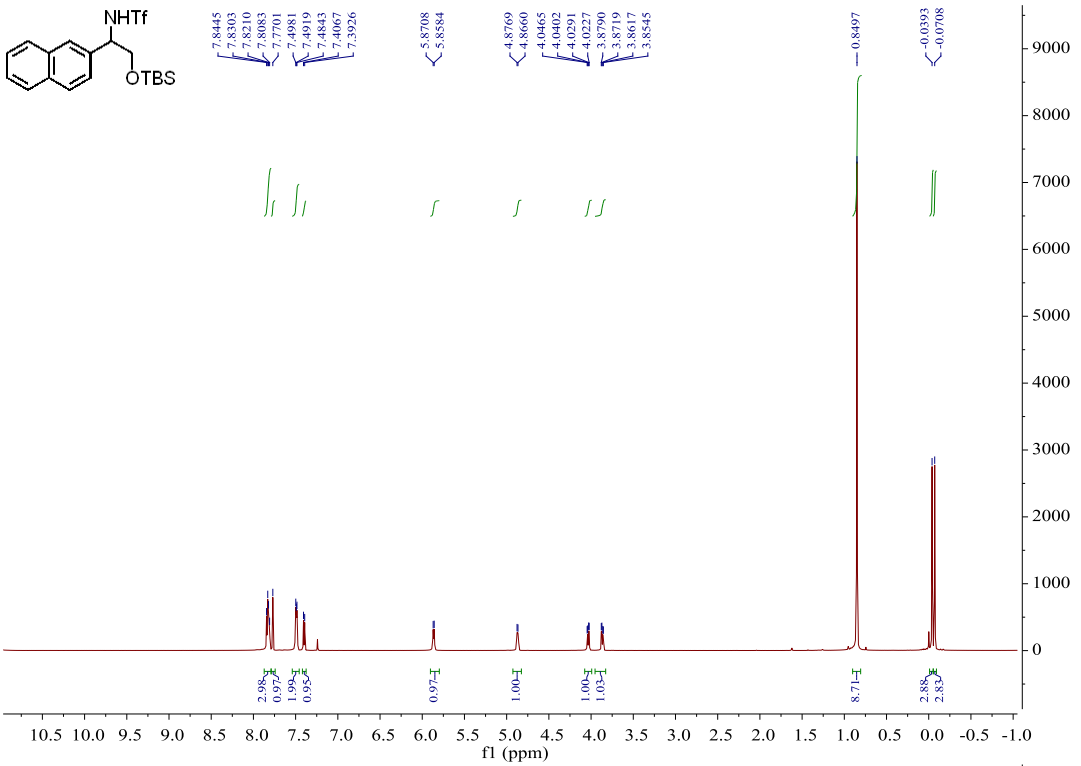
-5.8923
-5.9708
f1 (ppm)


NHTf
OTBS
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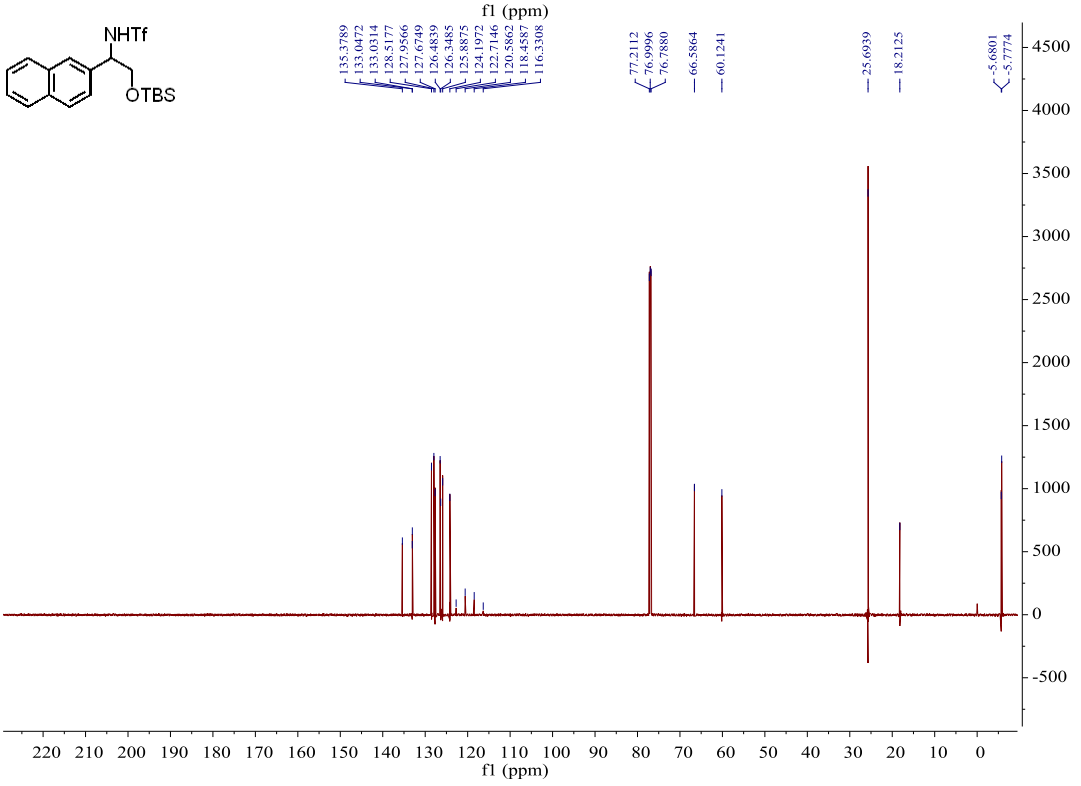
7.8303
7.8210
7.8083
7.7701
7.4981
7.4919
7.4843
7.4067
7.3926
5.8708
5.8584
4.8769
4.8660
4.0465
4.0402
4.0291
4.0227
3.8790
3.8719
3.8617
3.8545
0.8497
-0.0393
-0.0708
2.98
0.97
1.99
0.95
0.97
1.00
1.00
1.03
8.71
2.88
2.83
f1 (ppm)


NHTf
OTBS
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133.0472
133.0314
128.5177
127.9566
127.6749
126.4839
126.3485
125.8875
124.1972
122.7146
120.5862
118.4587
116.3308
77.2112
76.9996
76.7880
66.5864
60.1241
25.6939
18.2125
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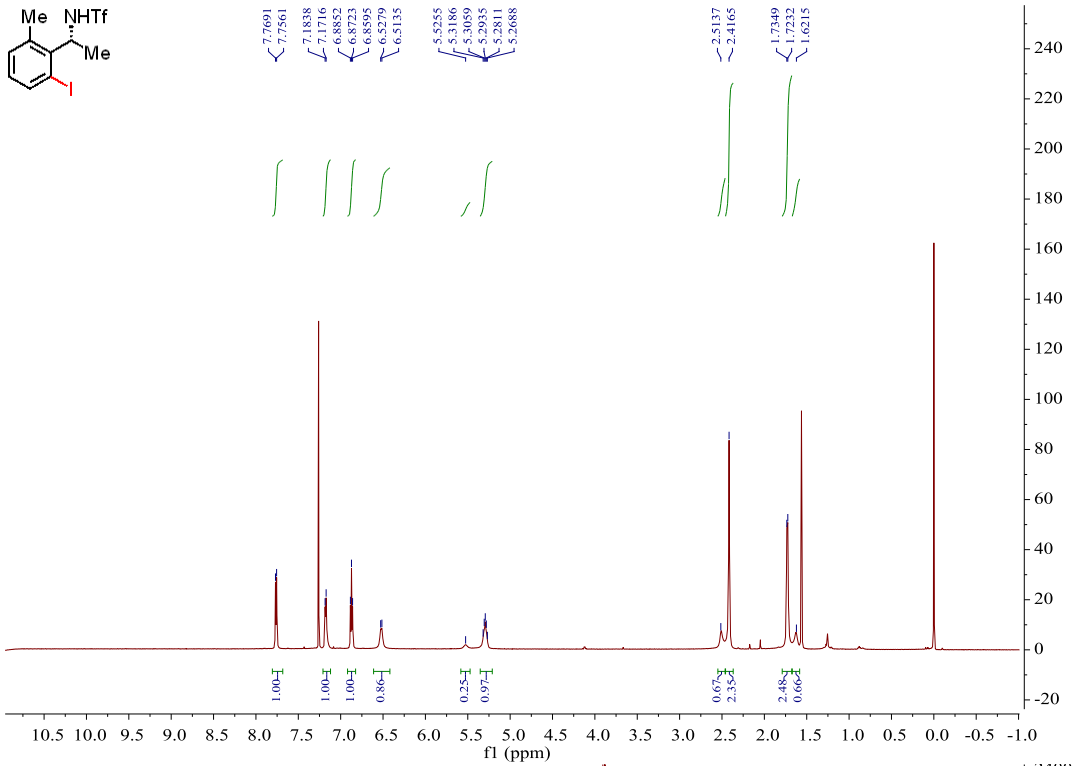
-5.7774
f1 (ppm)


Me
NHTf
Me
I
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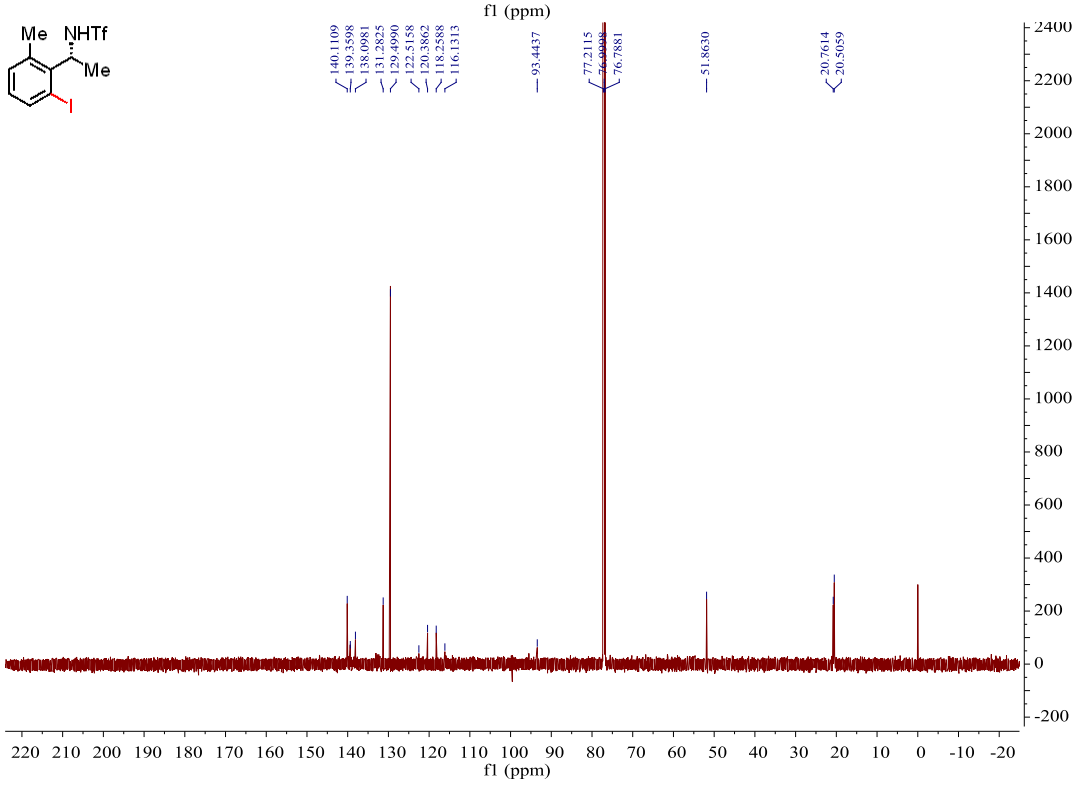
7.1838
7.1716
6.8852
6.8723
6.8595
6.5279
6.5135
5.5255
5.3186
5.3059
5.2935
5.2811
5.2688
2.5137
2.4165
1.7349
1.7232
1.6215
1.00
1.00
1.00
0.86
0.25
0.97
0.67
2.35
2.48
0.66
f1 (ppm)


Me
NHTf
Me
I
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139.3598
138.0981
131.2825
129.4990
122.5158
120.3862
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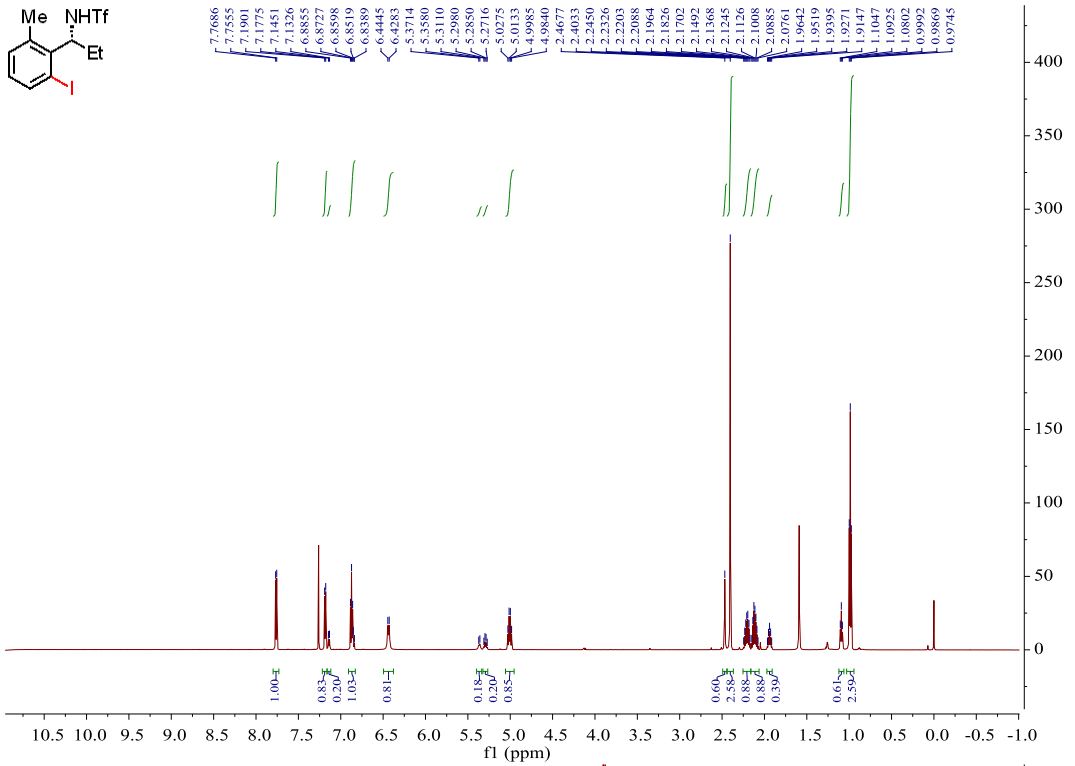
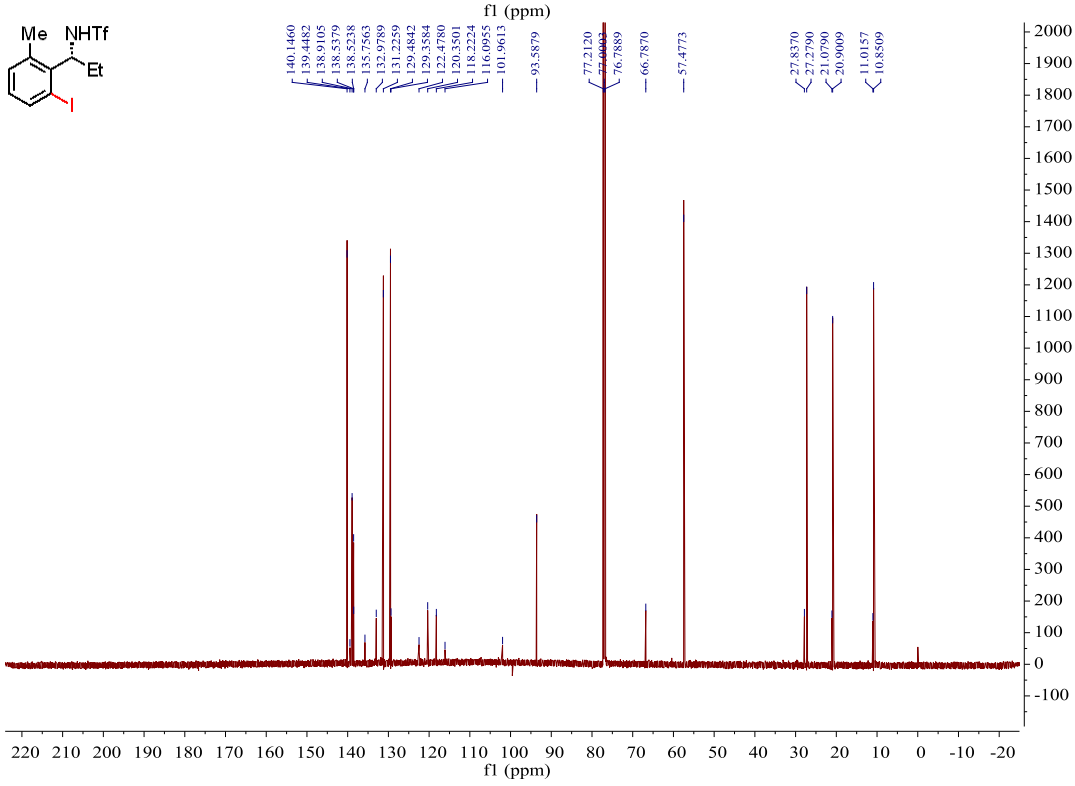
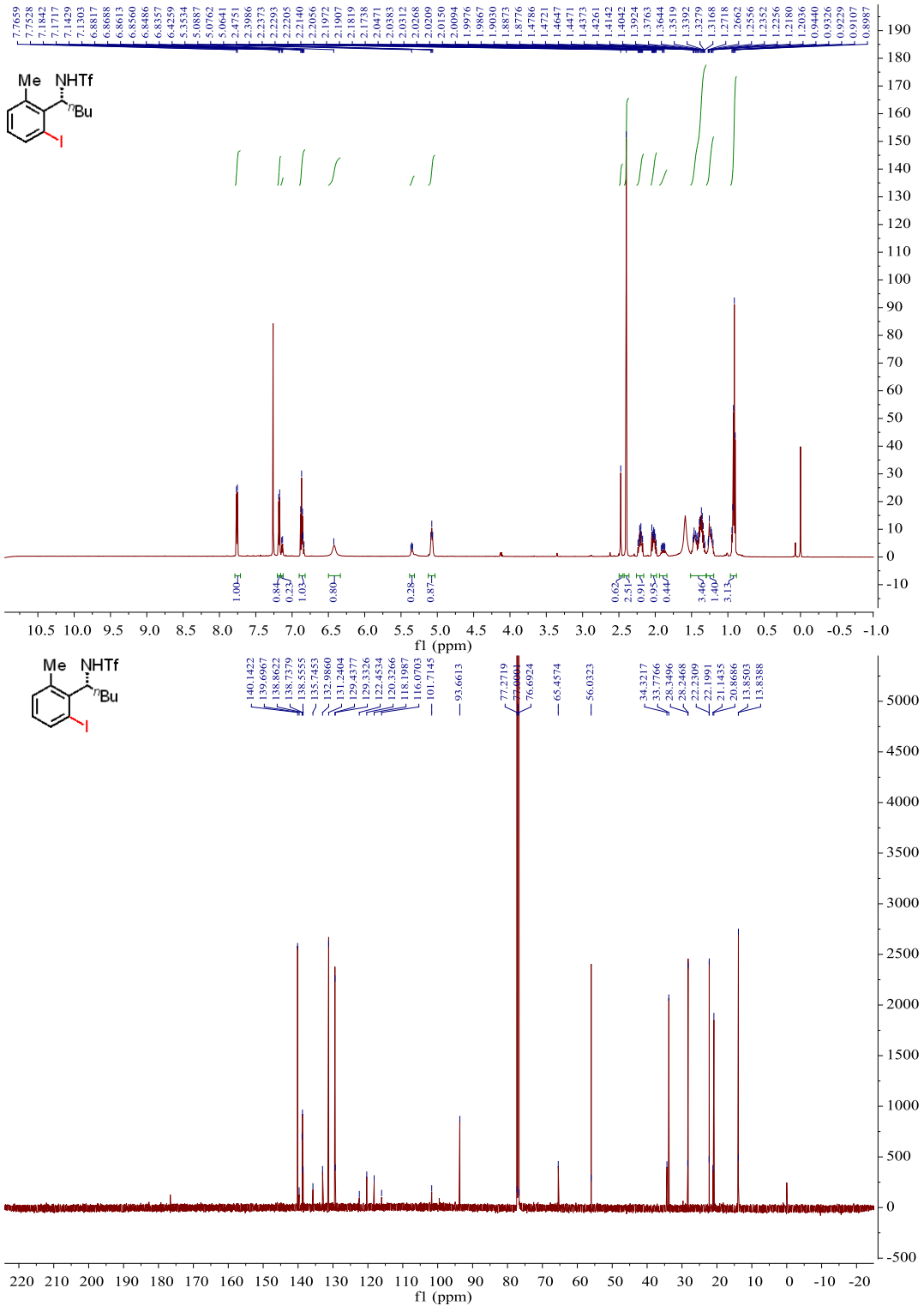
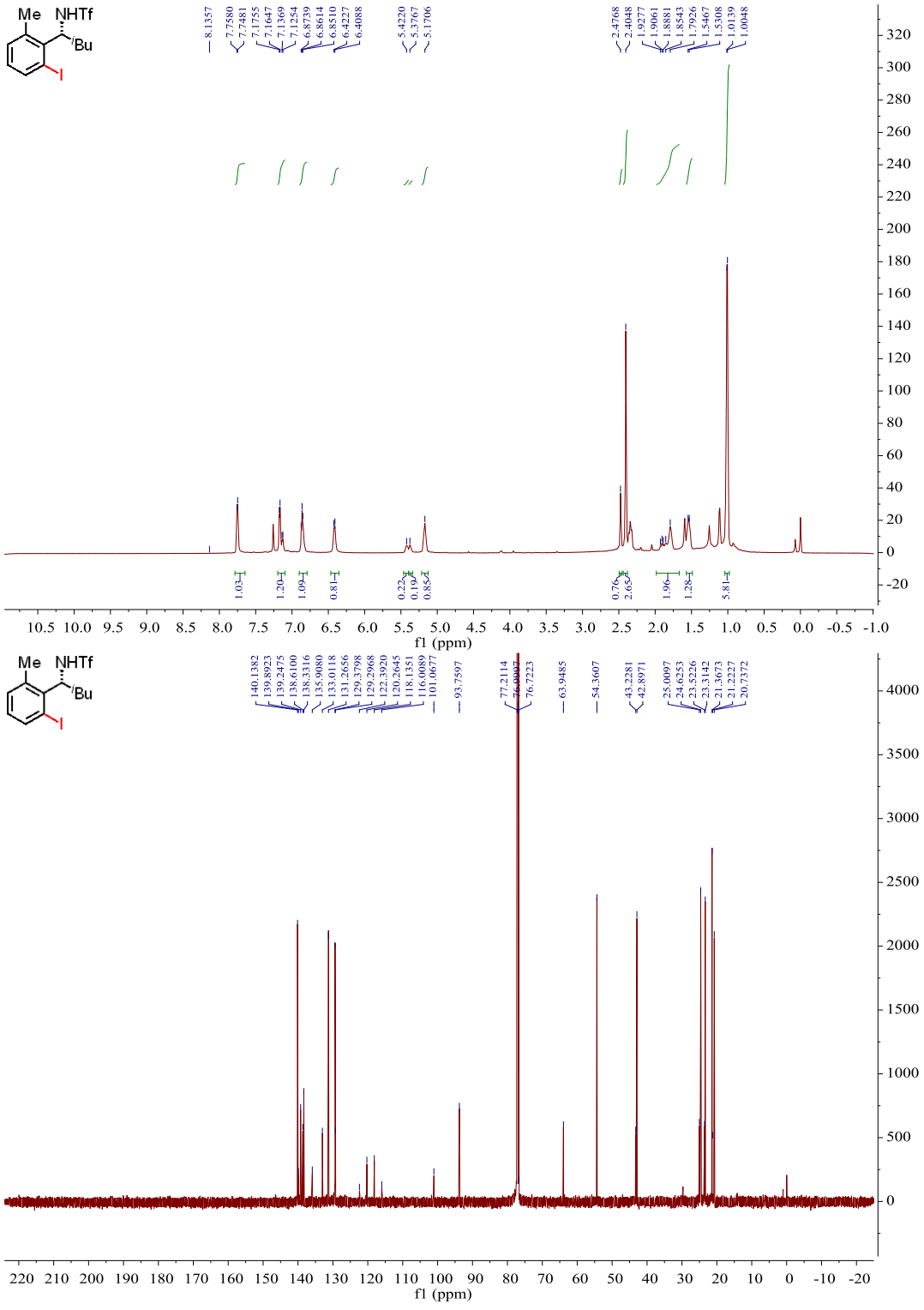
93.4437
77.2115
76.9998
76.7881
51.8630
20.7614
20.5059
f1 (ppm)


Me
NHTf
Et
I
f1 (ppm)


Me
NHTf
Et
I
f1 (ppm)


Me
NHTf
nBu
I
f1 (ppm)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tBu
I
8.1357
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7.7481
7.1755
7.1647
7.1369
7.1254
6.8739
6.8614
6.8510
6.4227
6.4088
5.4220
5.3767
5.1706
2.4768
2.4048
1.9277
1.9061
1.8881
1.8543
1.7926
1.5467
1.5308
1.0139
1.0048
1.03
1.20
1.09
0.81
0.22
0.19
0.85
0.76
2.65
1.96
1.28
5.81
f1 (ppm)
140.1382
139.8923
139.2475
138.6100
138.3316
135.9080
133.0118
131.2656
129.3798
129.2968
122.3920
120.2645
118.1351
116.0089
101.0677
93.7597
77.2114
76.0007
76.7223
63.9485
54.3607
43.2281
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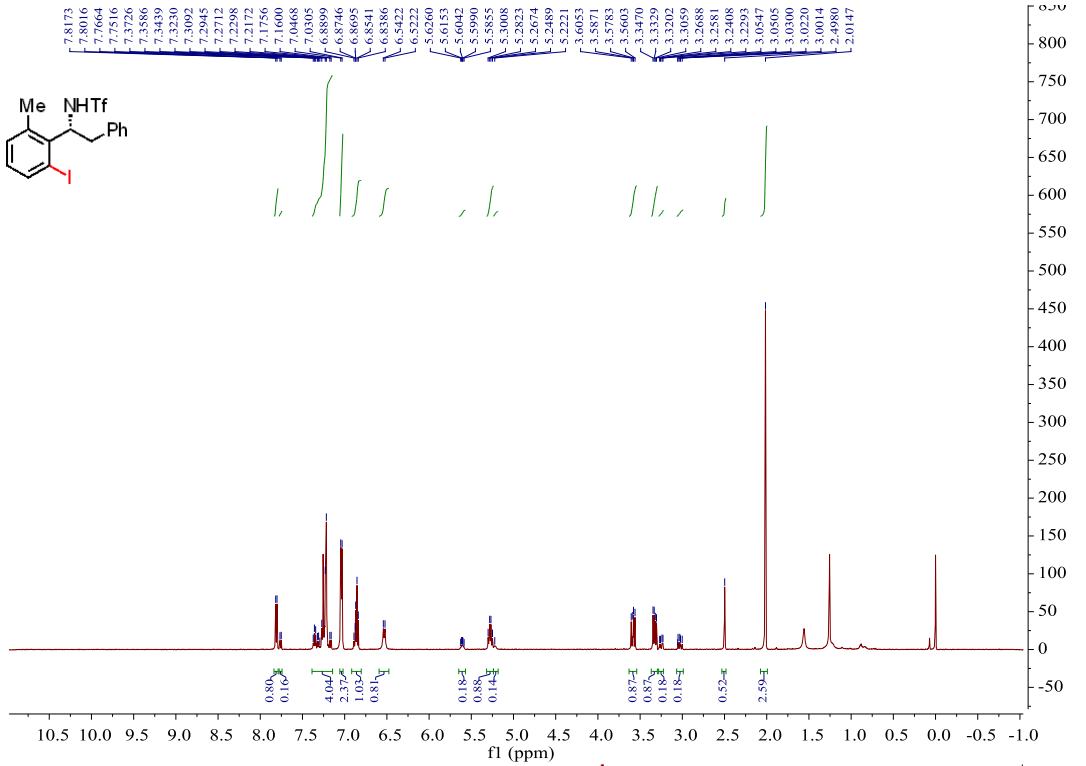
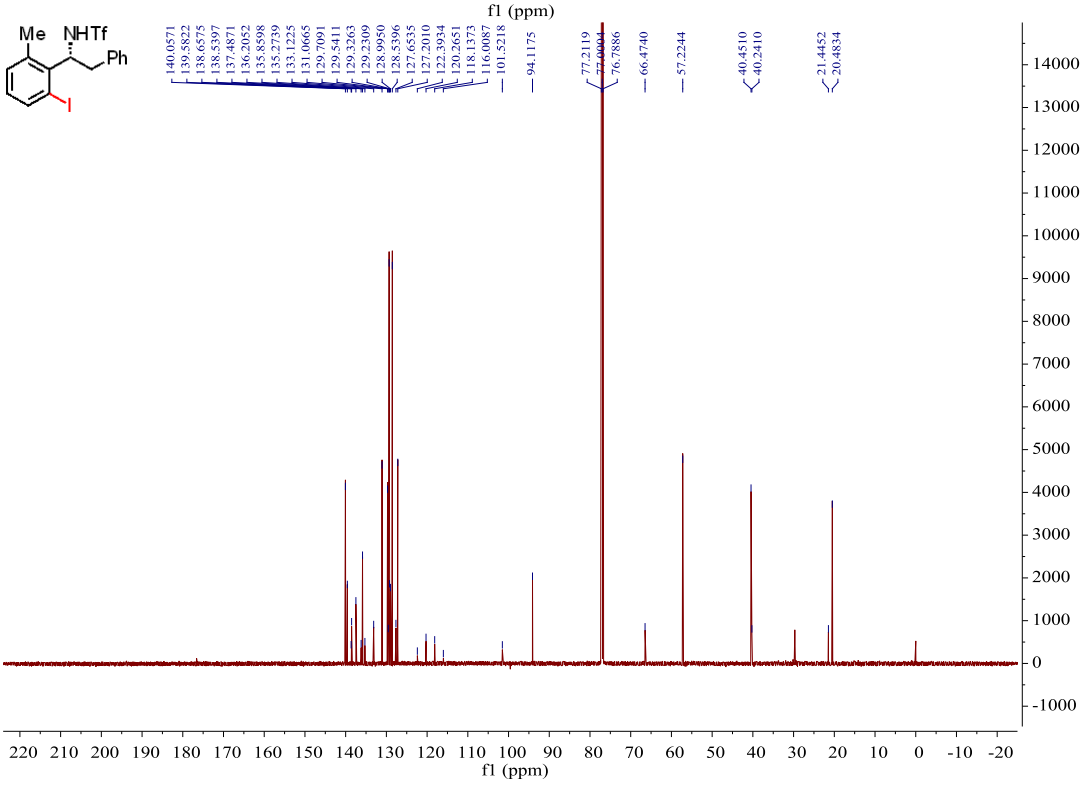
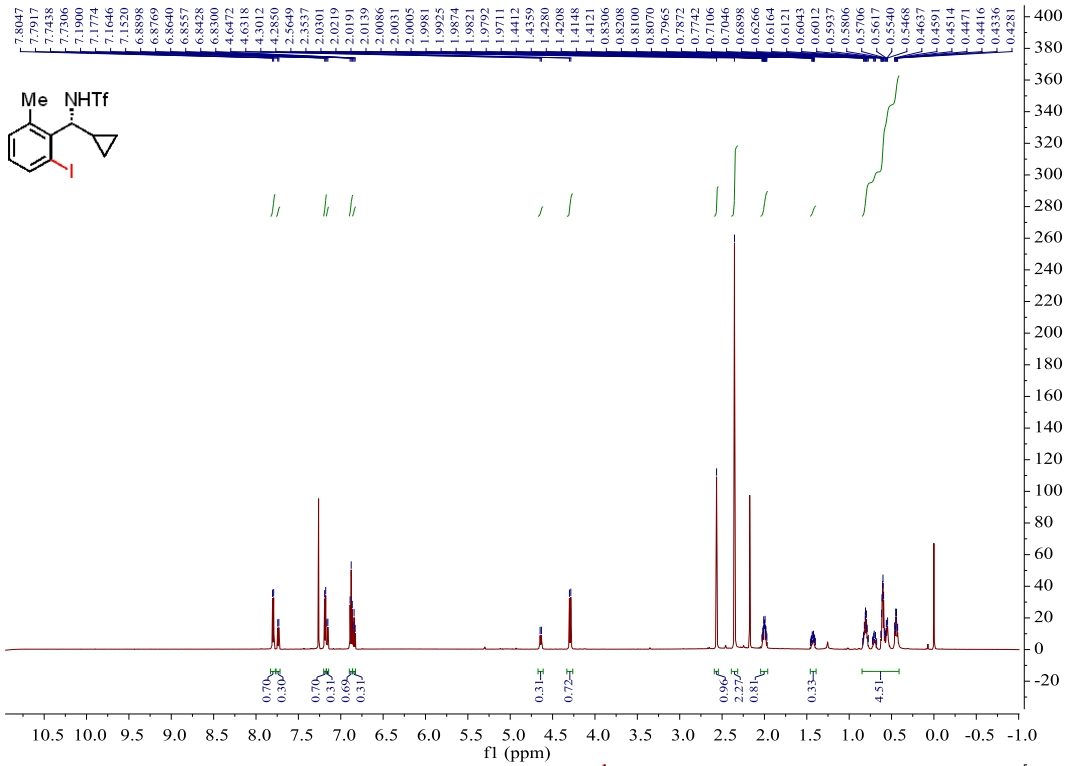
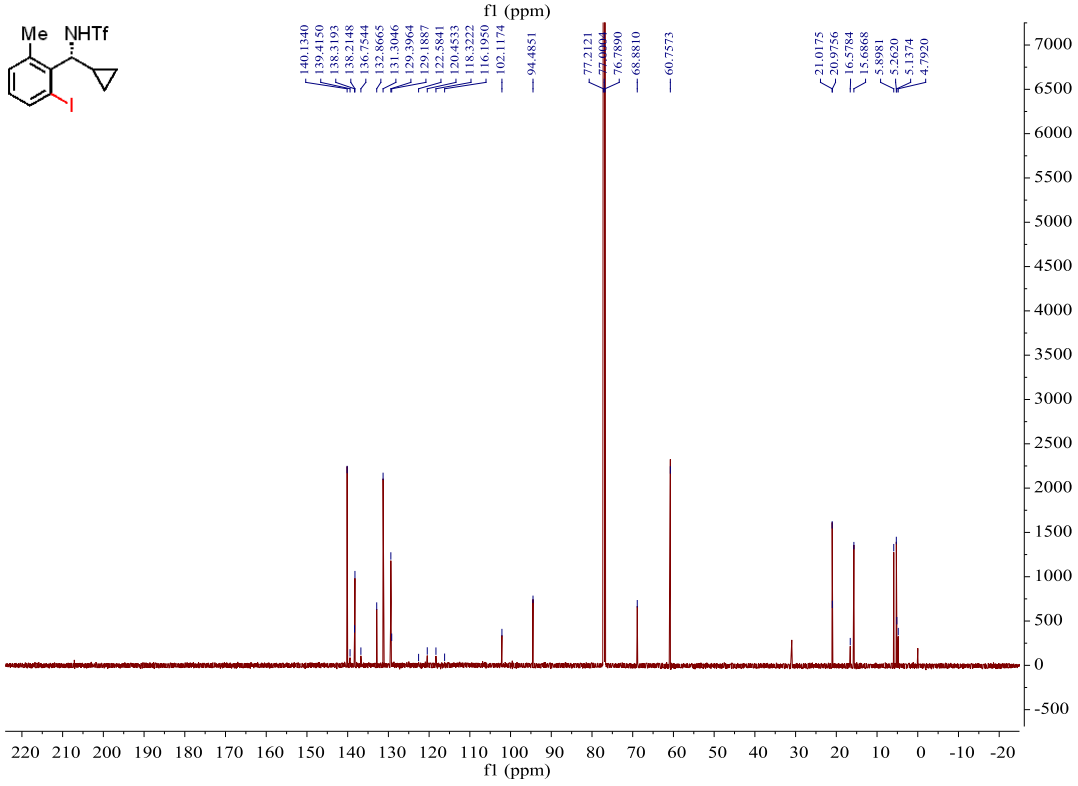
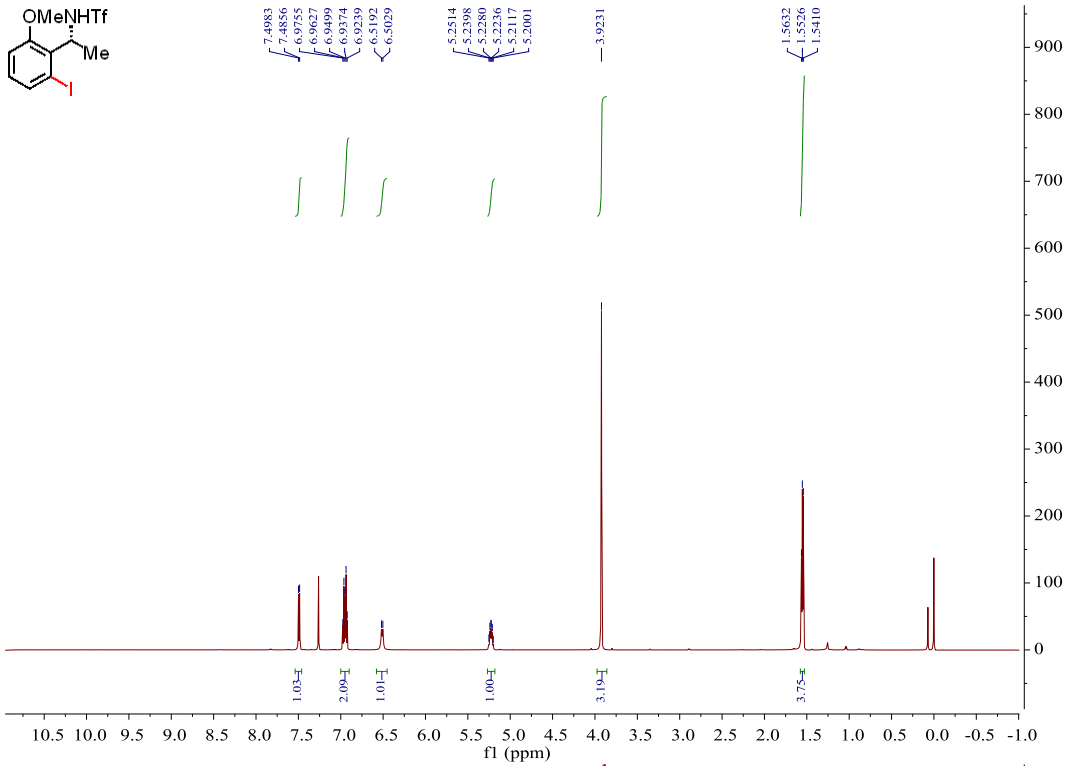
21.3673
21.2227
20.7372
f1 (ppm)


Me
NHTf
Ph
I
f1 (ppm)


Me
NHTf
Ph
I
f1 (ppm)


Me
NHTf
I
f1 (ppm)


Me
NHTf
I
f1 (ppm)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I
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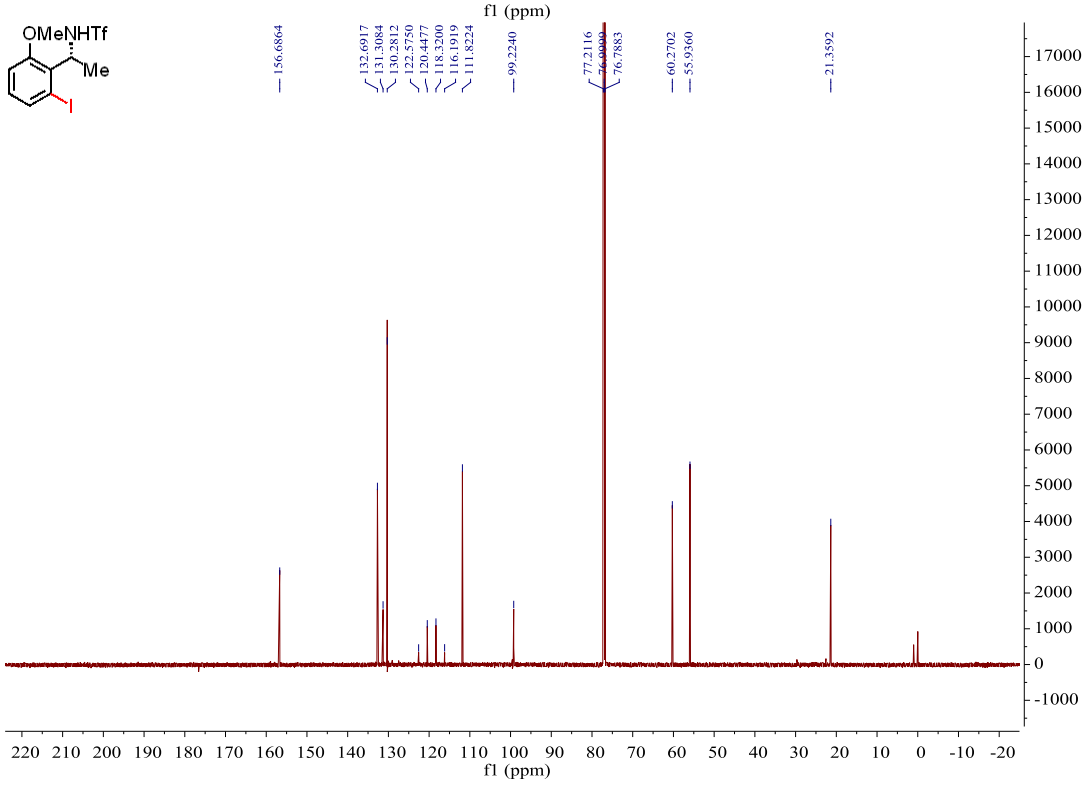
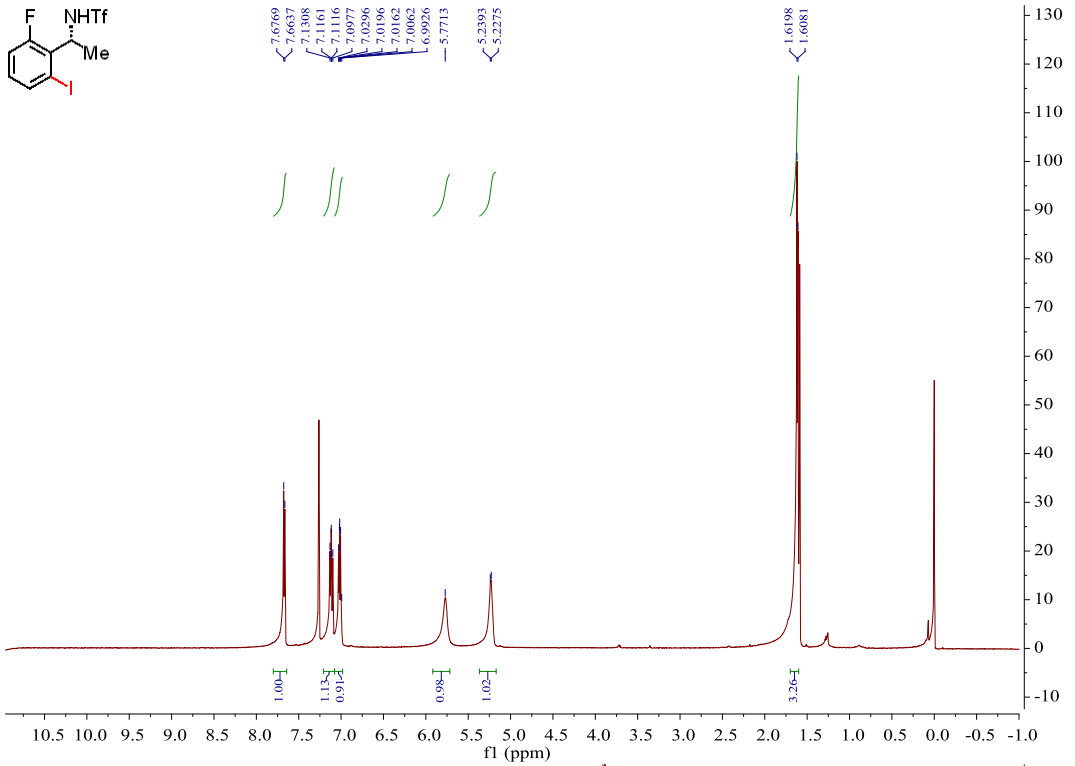
6.9755
6.9627
6.9499
6.9374
6.9239
6.5192
6.5029
5.2514
5.2398
5.2280
5.2236
5.2117
5.2001
3.9231
1.5632
1.5526
1.5410
1.03
2.09
1.01
1.00
3.19
3.75
f1 (ppm)


OMeNHTf
Me
I
156.6864
132.6917
131.3084
130.2812
122.5750
120.4477
118.3200
116.1919
111.8224
99.2240
77.2116
76.9999
76.7883
60.2702
55.9360
21.3592
f1 (ppm)


F
NHTf
Me
I
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7.6637
7.1308
7.1161
7.1116
7.0977
7.0296
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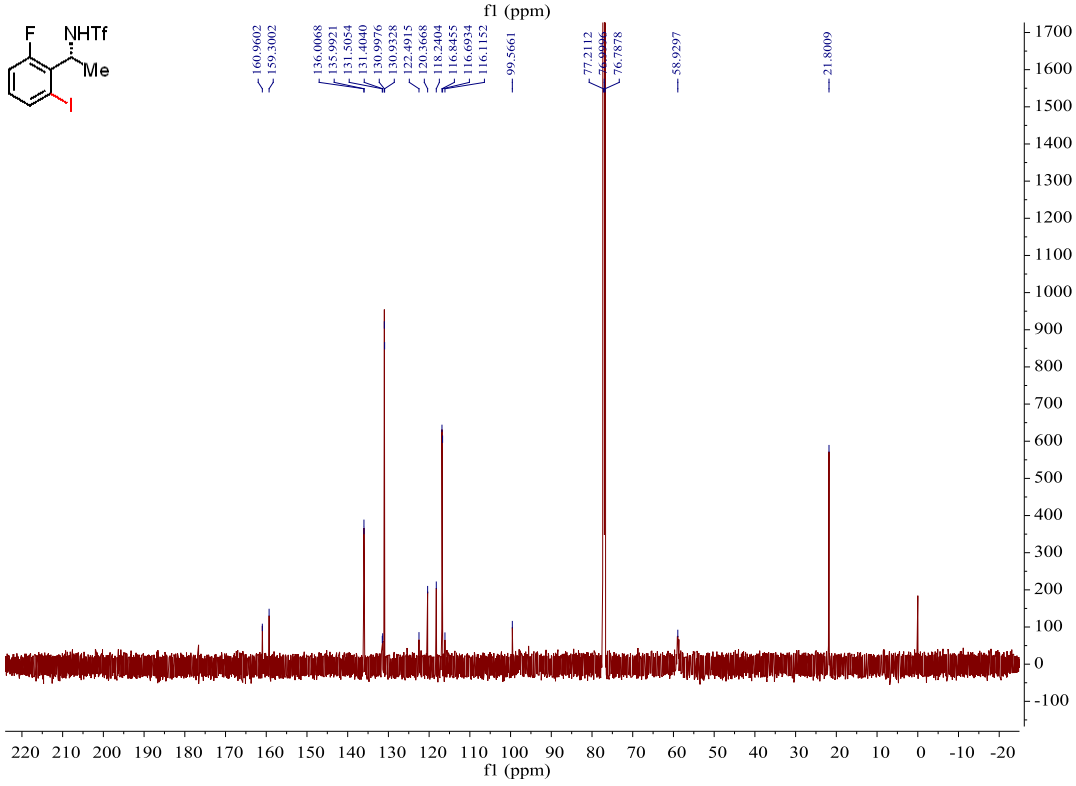
7.0162
7.0062
6.9926
5.7713
5.2393
5.2275
1.6198
1.6081
1.00
1.13
0.91
0.98
1.02
3.26
f1 (ppm)


F
NHTf
Me
I
160.9602
159.3002
136.0068
135.9921
131.5054
131.4040
130.9976
130.9328
122.4915
120.3668
118.2404
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116.1152
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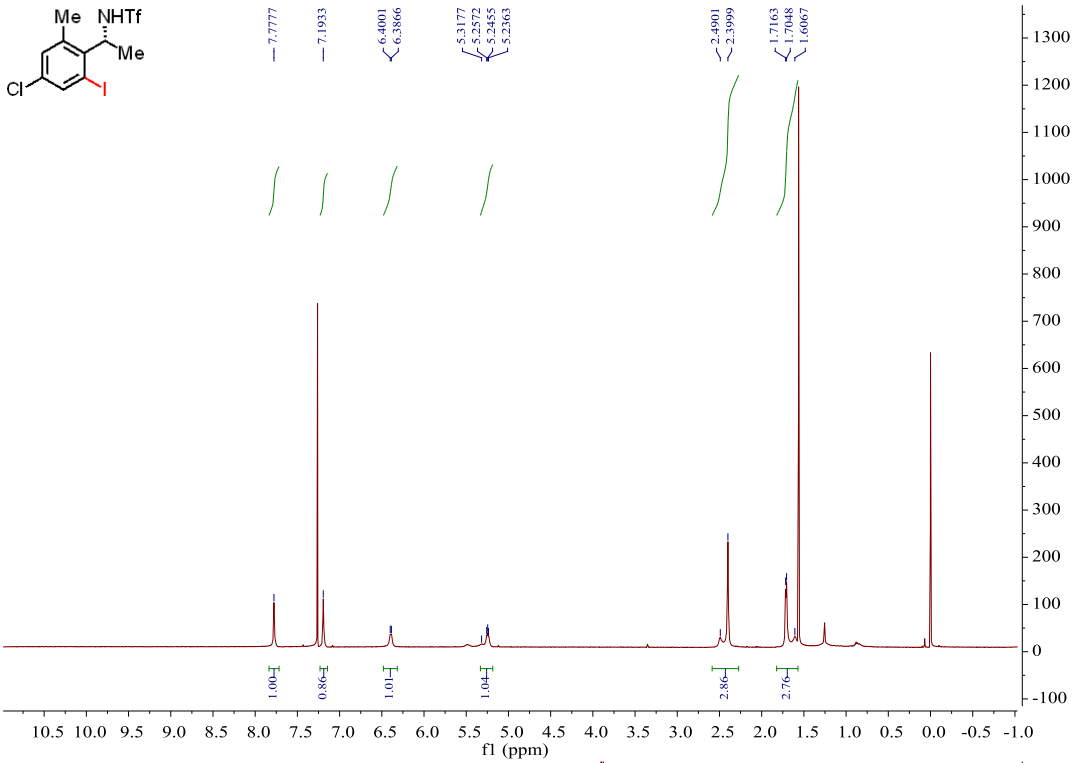
77.2112
76.9996
76.7878
58.9297
21.8009
f1 (ppm)


Me NHTf
Me
Cl
I
7.7777
7.1933
6.4001
6.3866
5.3177
5.2572
5.2455
5.2363
2.4901
2.3999
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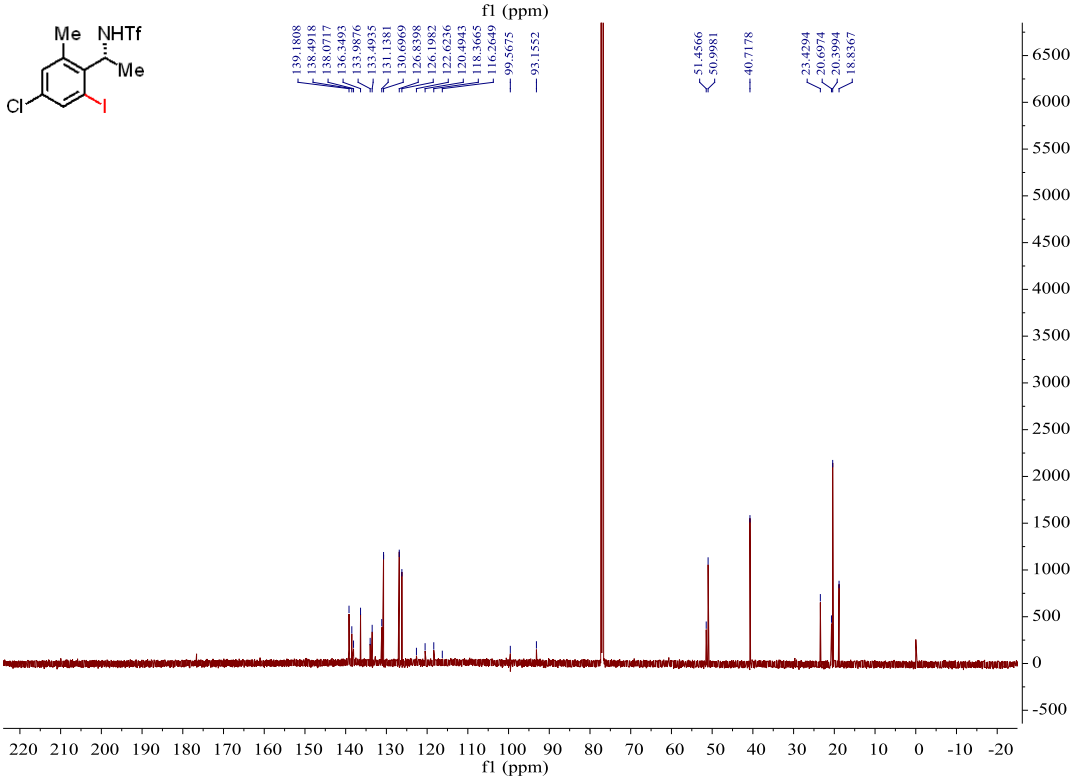
1.7048
1.6067
1.00
0.86
1.01
1.04
2.86
2.76
f1 (ppm)


Me NHTf
Me
Cl
I
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133.9876
133.4935
131.1381
130.6969
126.8398
126.1982
122.6236
120.4943
118.3665
116.2649
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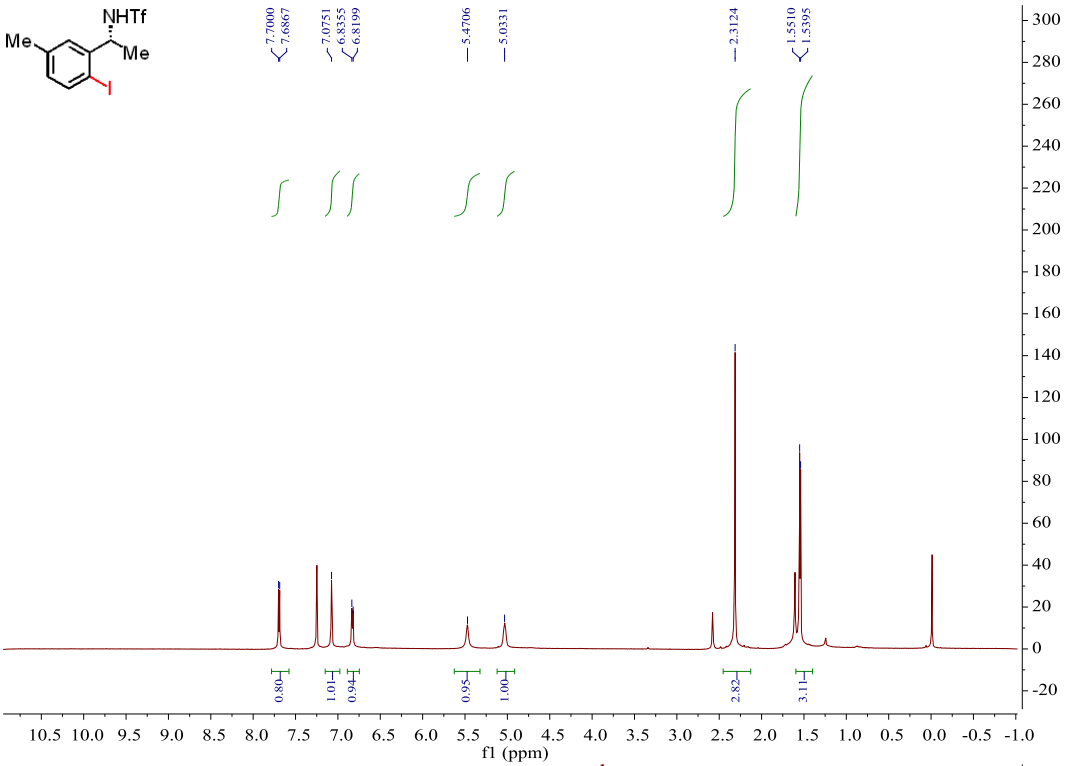
93.1552
51.4566
50.9981
40.7178
23.4294
20.6974
20.3994
18.8367
f1 (ppm)


NHTf
Me
Me
I
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7.0751
6.8355
6.8199
5.4706
5.0331
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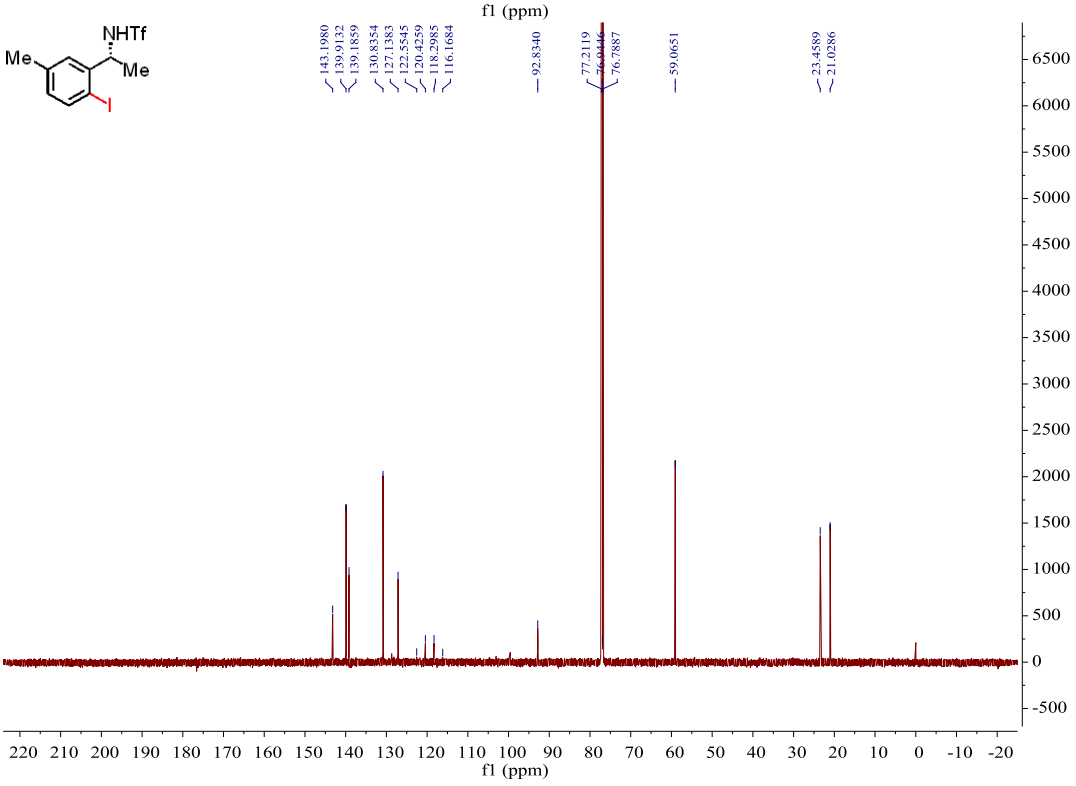
1.5395
0.80
1.01
0.94
0.95
1.00
2.82
3.11
f1 (ppm)


NHTf
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Me
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120.4259
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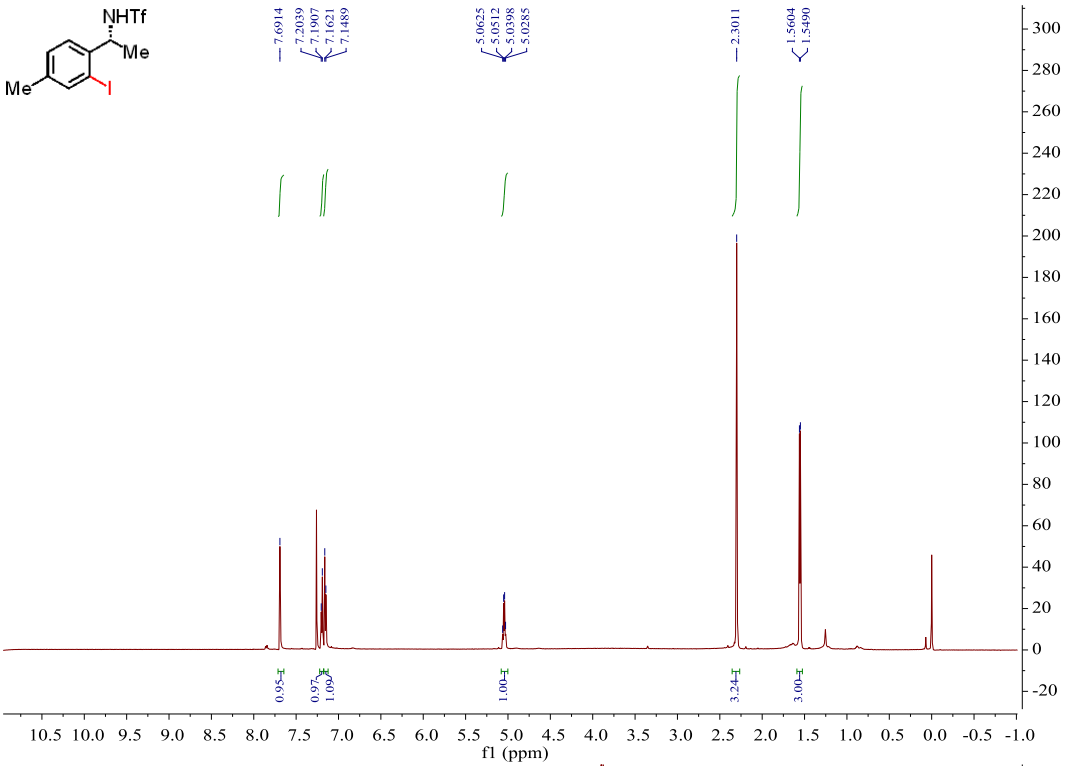
76.7887
59.0651
23.4589
21.0286
f1 (ppm)


NHTf
Me
Me
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7.1907
7.1621
7.1489
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5.0512
5.0398
5.0285
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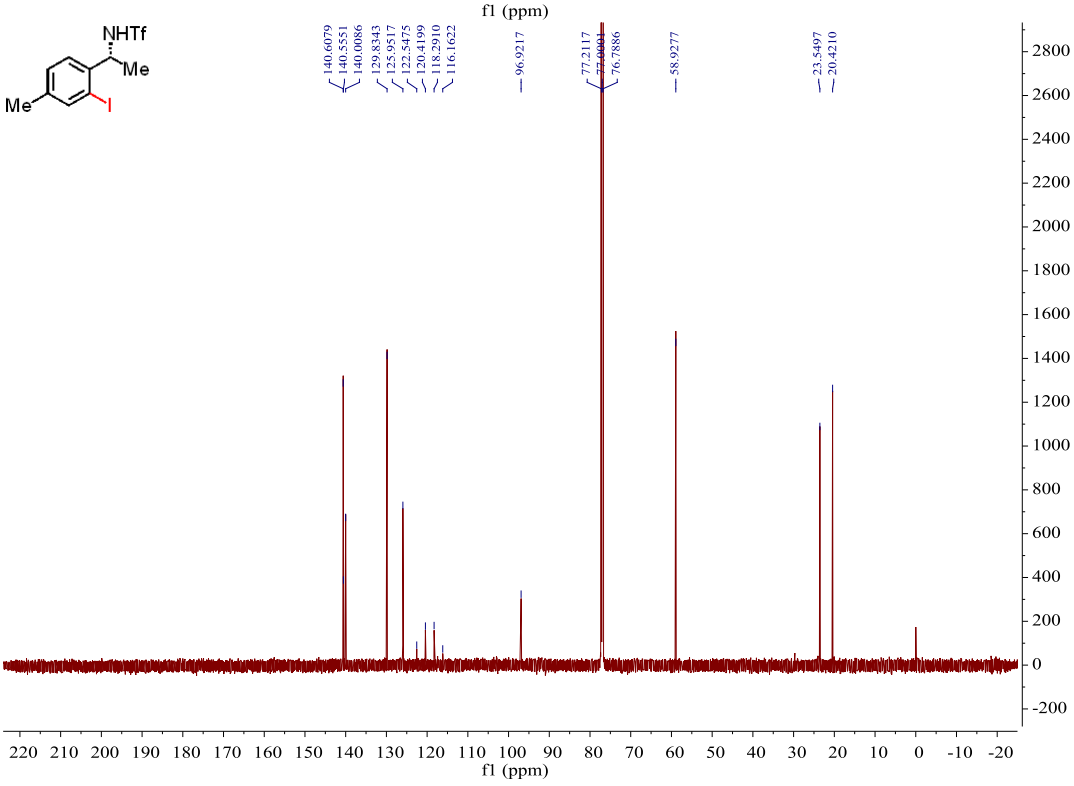
1.5490
0.95
0.97
1.09
1.00
3.24
3.00
f1 (ppm)


NHTf
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120.4199
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77.0994
76.7886
58.9277
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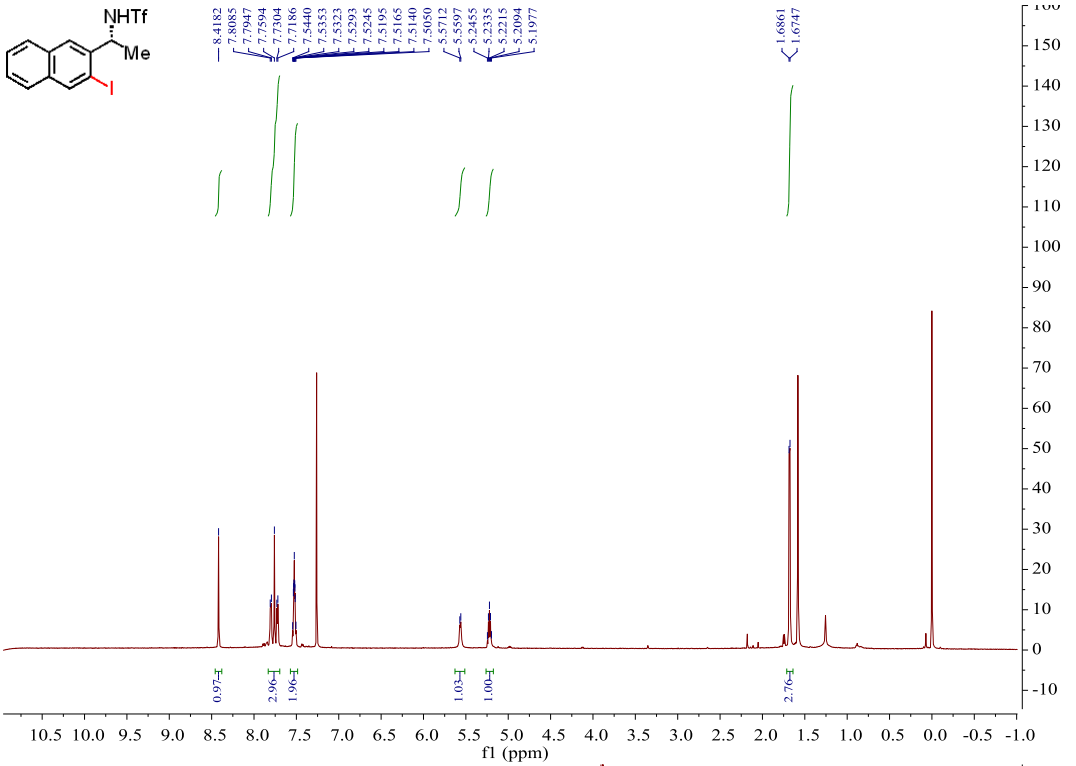
20.4210
f1 (ppm)


NHTf
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I
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7.7947
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7.7304
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7.5440
7.5353
7.5323
7.5293
7.5245
7.5195
7.5165
7.5140
7.5050
5.5712
5.5597
5.2455
5.2335
5.2215
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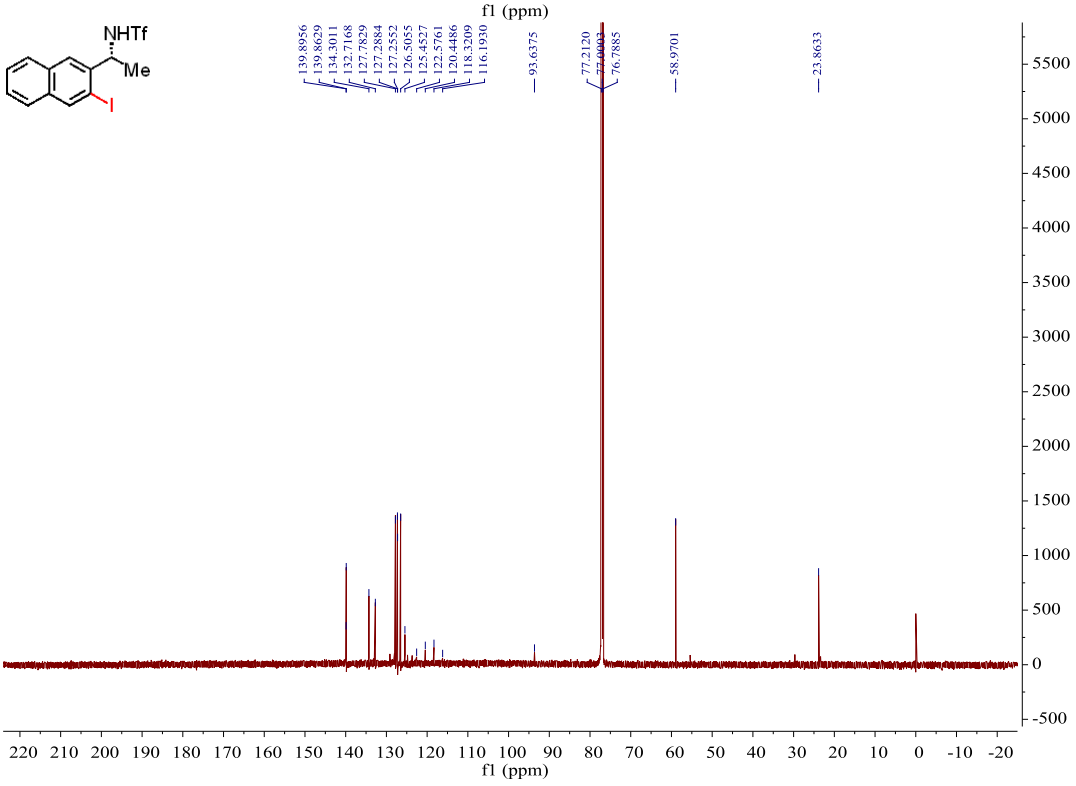
1.6747
0.97
2.96
1.96
1.03
1.00
2.76
f1 (ppm)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127.2552
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125.4527
122.5761
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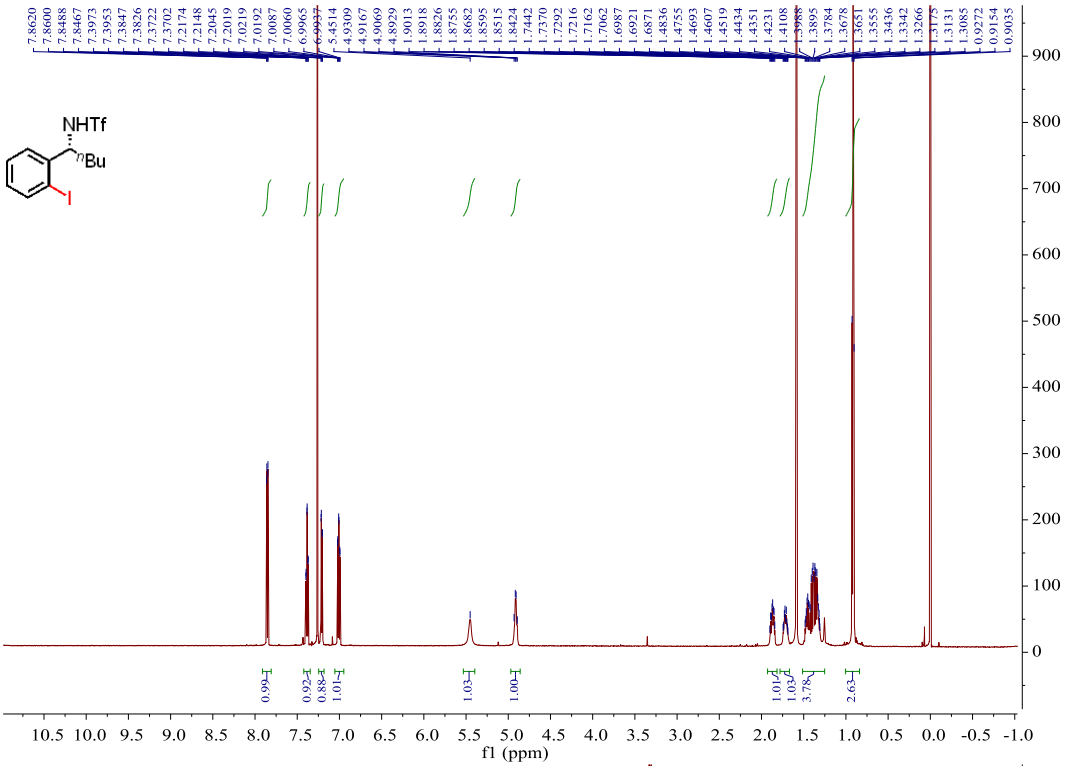
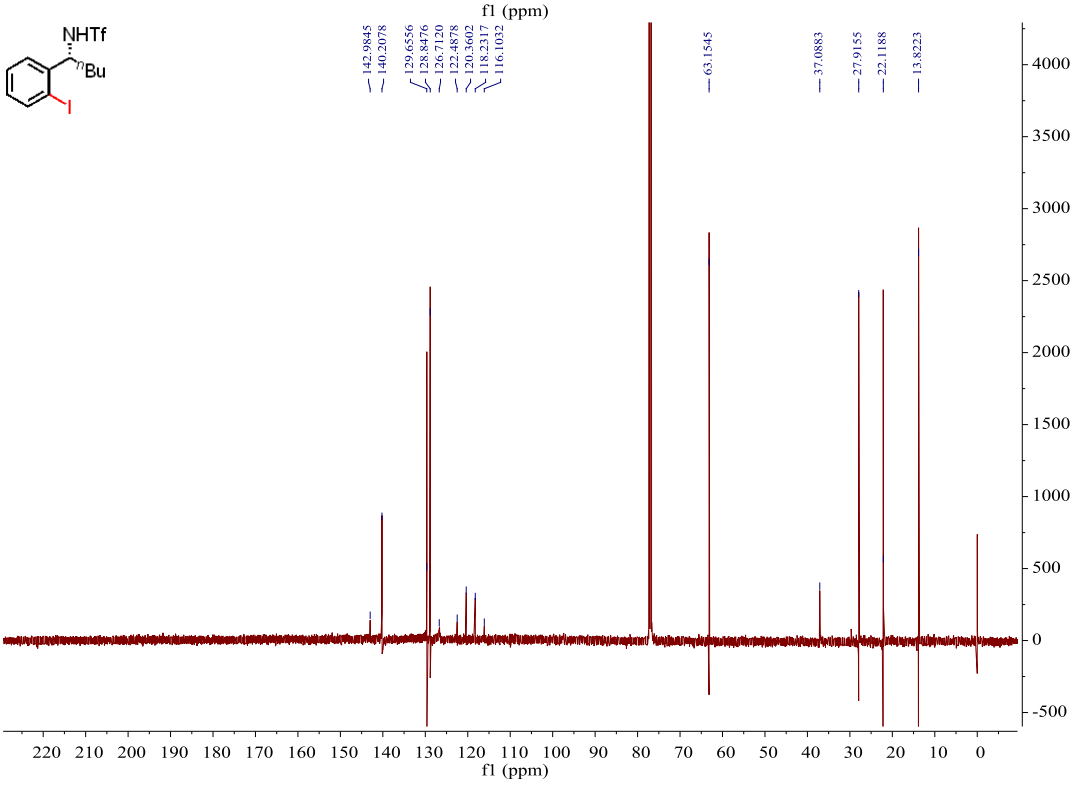
116.1930
93.6375
77.2120
77.0003
76.7885
58.9701
23.8633
f1 (ppm)


NHTf
nBu
I
f1 (ppm)


NHTf
nBu
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126.7120
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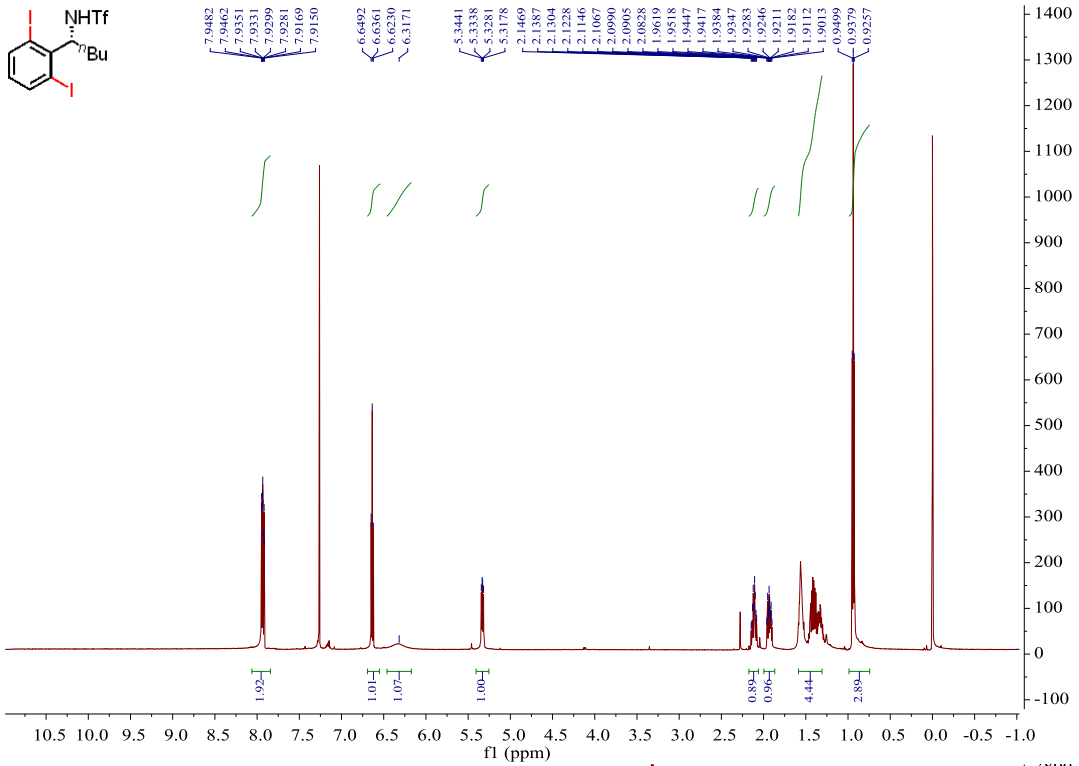
118.2317
116.1032
63.1545
37.0883
27.9155
22.1188
13.8223
f1 (ppm)


NHTf
nBu
I
I
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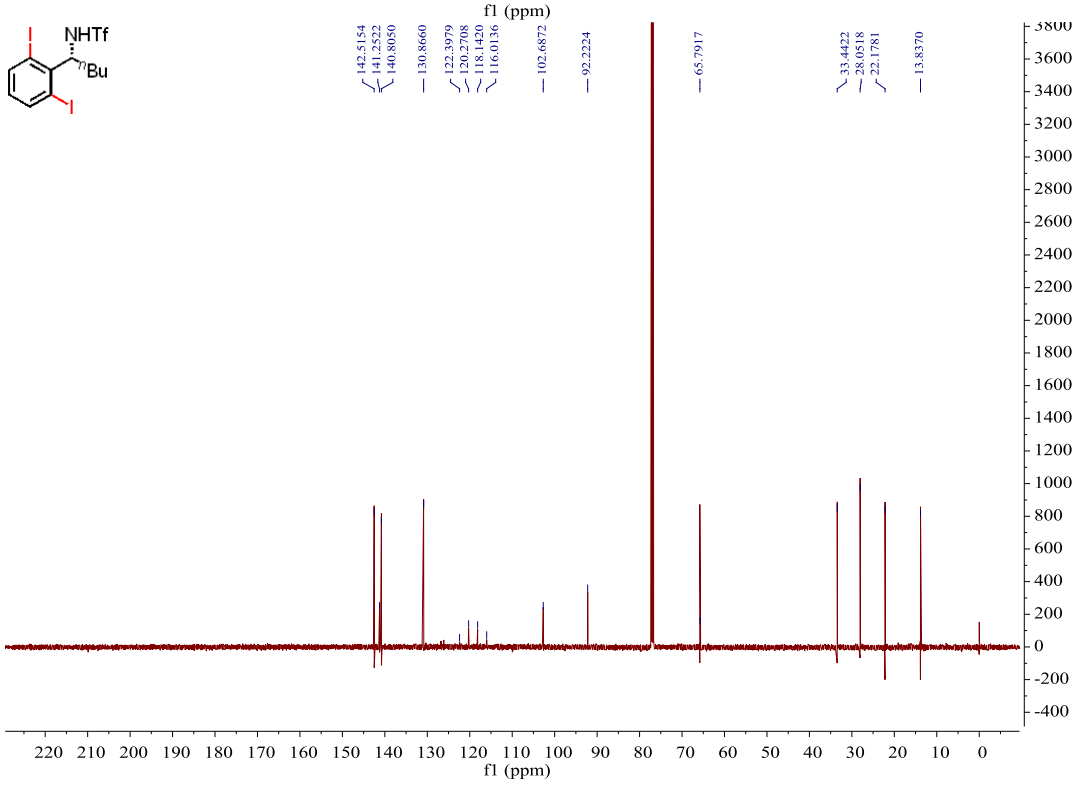
7.9351
7.9331
7.9299
7.9281
7.9169
7.9150
6.6492
6.6361
6.6230
6.3171
5.3441
5.3338
5.3281
5.3178
2.1469
2.1387
2.1304
2.1228
2.1146
2.1067
2.0990
2.0905
2.0828
1.9619
1.9518
1.9447
1.9417
1.9384
1.9347
1.9283
1.9246
1.9211
1.9182
1.9112
1.9013
0.9499
0.9379
0.9257
1.92
1.01
1.07
1.00
0.89
0.96
4.44
2.89
f1 (ppm)


NHTf
nBu
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I
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120.2708
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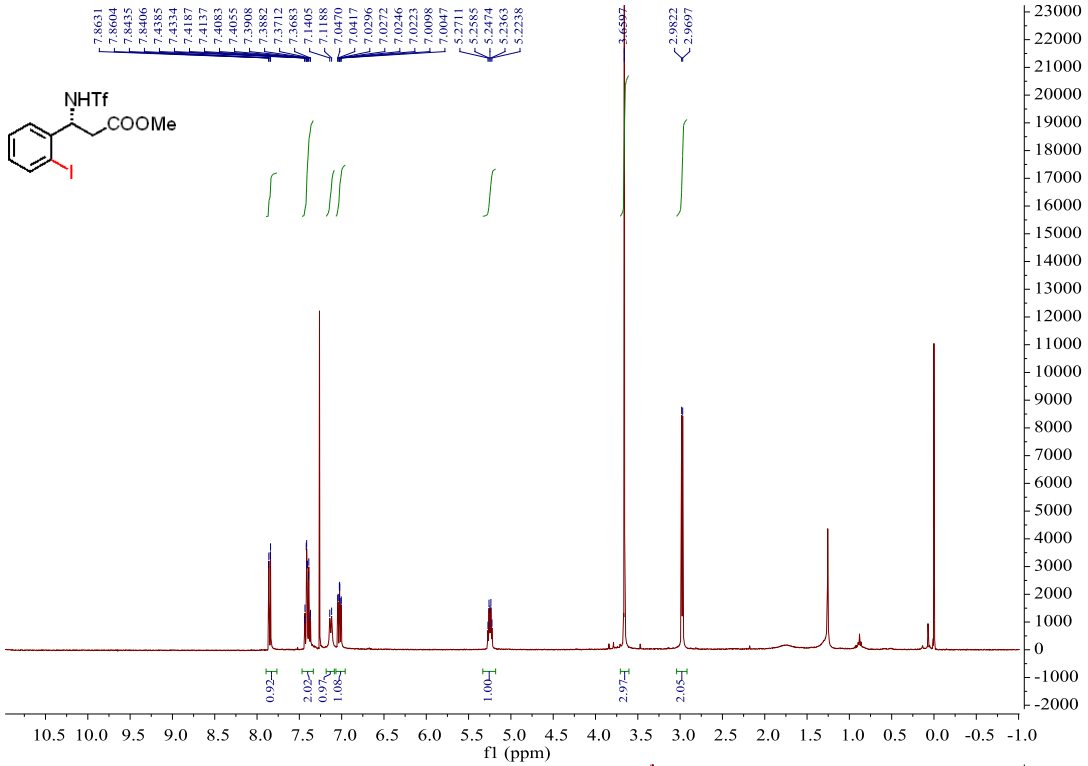
92.2224
65.7917
33.4422
28.0518
22.1781
13.8370
f1 (ppm)


NHTf
COOMe
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7.8435
7.8406
7.4385
7.4334
7.4187
7.4137
7.4083
7.4055
7.3908
7.3882
7.3712
7.3683
7.1405
7.1188
7.0470
7.0417
7.0296
7.0272
7.0246
7.0223
7.0098
7.0047
5.2711
5.2585
5.2474
5.2363
5.2238
3.6897
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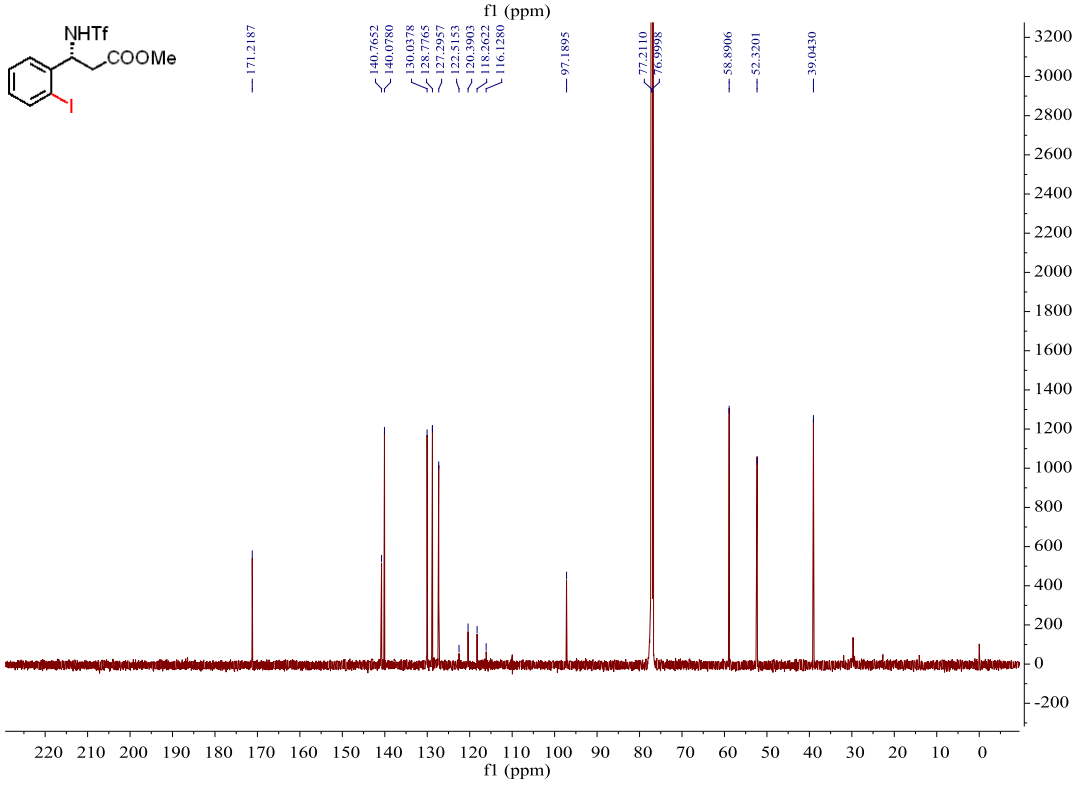
0.92
2.02
0.97
1.08
1.00
2.97
2.05
f1 (ppm)


NHTf
COOMe
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128.7765
127.2957
122.5153
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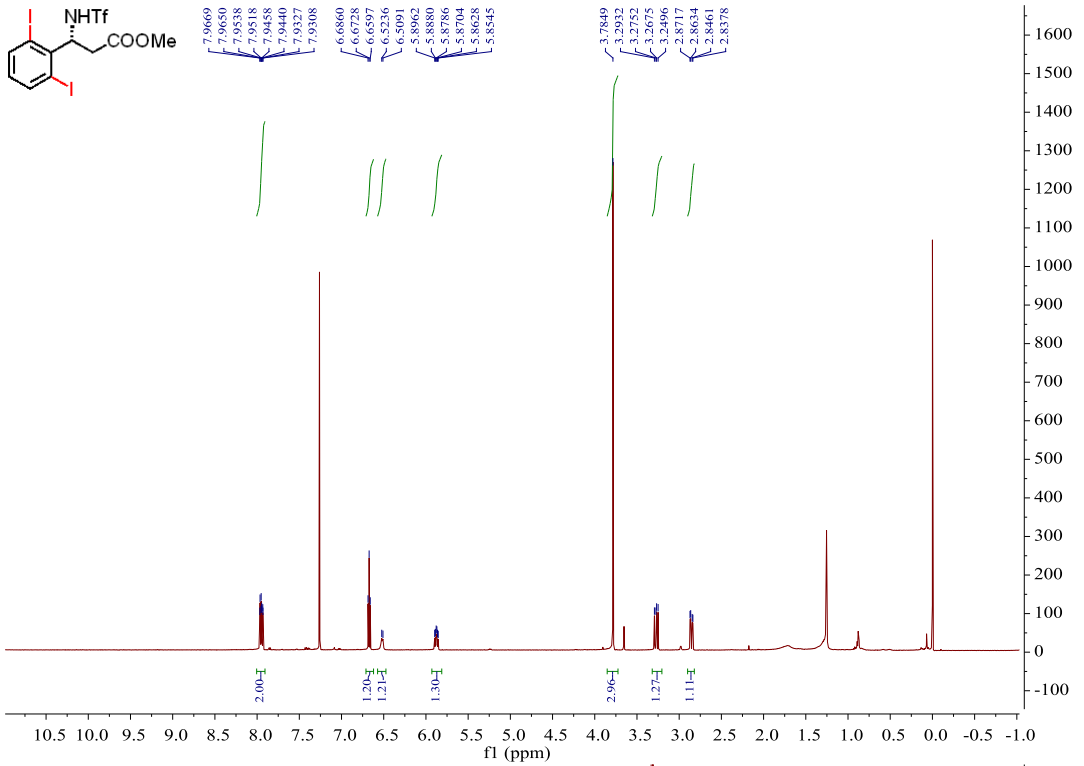
77.2110
76.9998
58.8906
52.3201
39.0430
f1 (ppm)


NHTf
COOMe
I
I
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7.9440
7.9327
7.9308
6.6860
6.6728
6.6597
6.5236
6.5091
5.8962
5.8880
5.8786
5.8704
5.8628
5.8545
3.7849
3.2932
3.2752
3.2675
3.2496
2.8717
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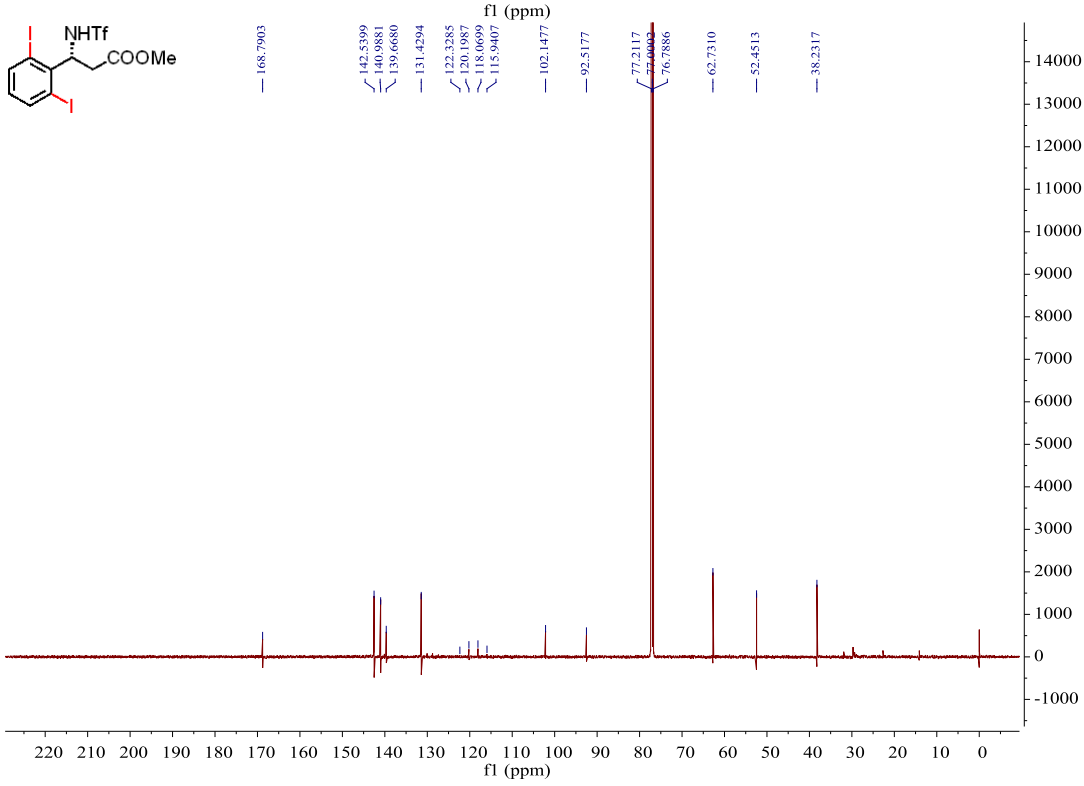
1.20
1.21
1.30
2.96
1.27
1.11
f1 (ppm)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COOMe
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76.7886
62.7310
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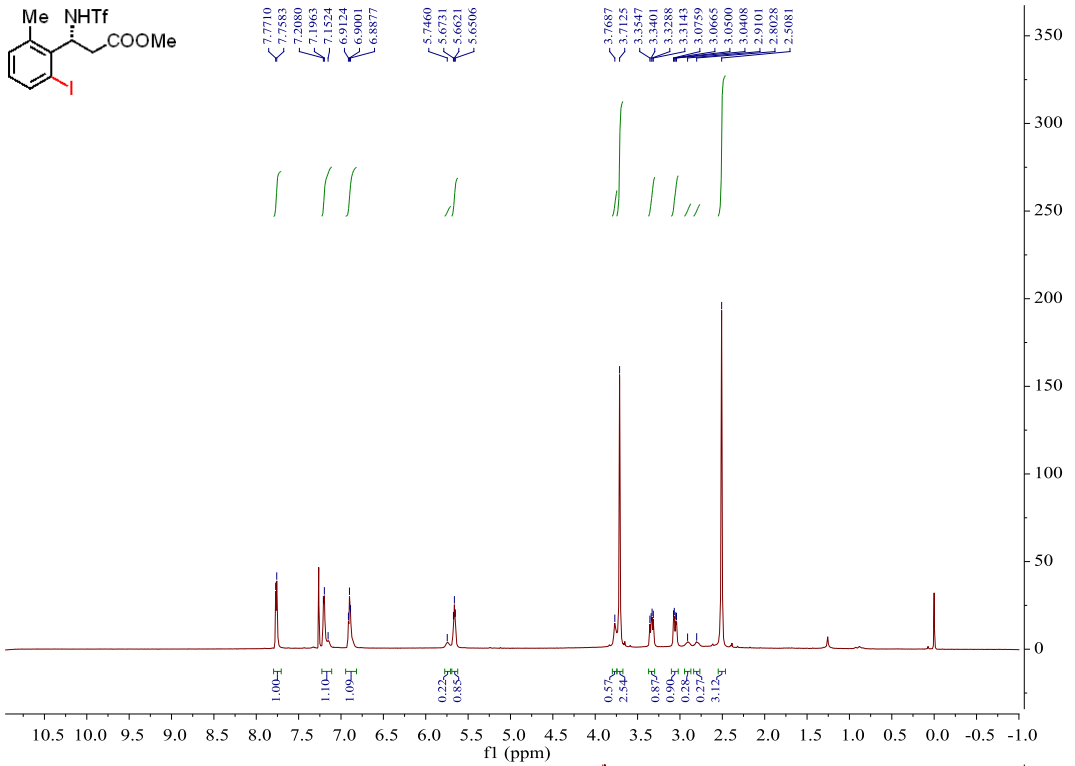
38.2317
f1 (ppm)


Me
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6.9001
6.8877
5.7460
5.6731
5.6621
5.6506
3.7687
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3.5547
3.3401
3.3288
3.3143
3.0759
3.0665
3.0500
3.0408
2.9101
2.8028
2.5081
1.00
1.10
1.09
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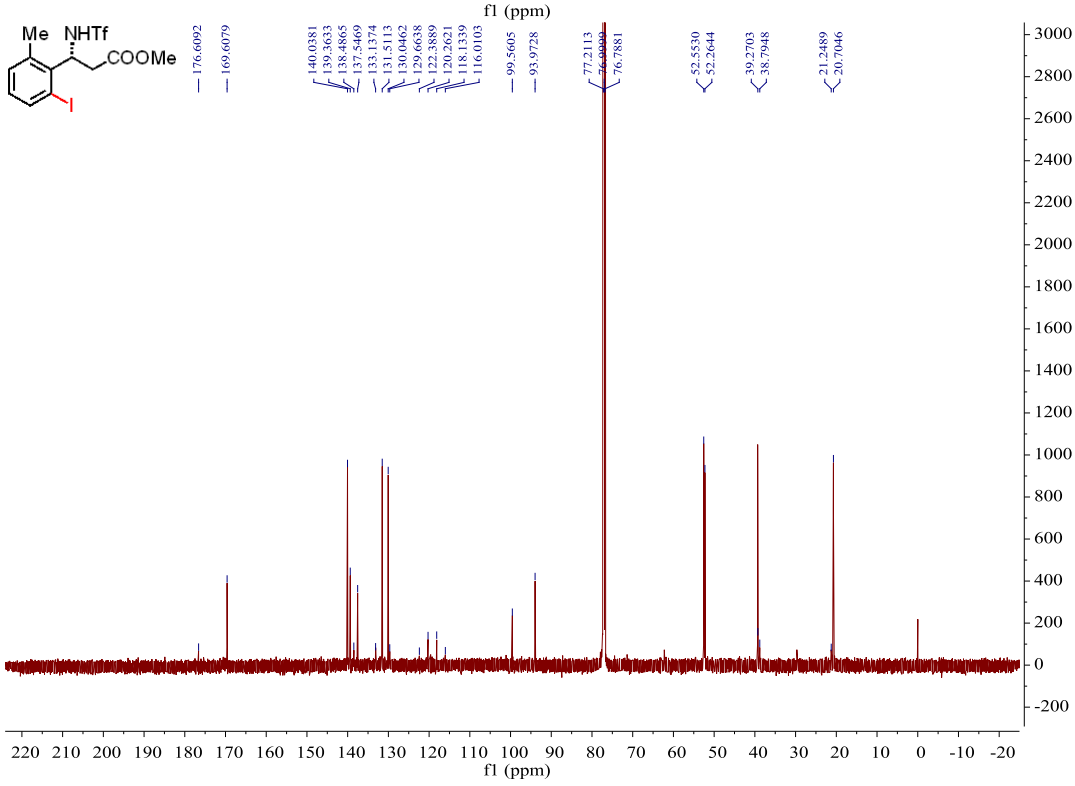
0.85
0.57
2.54
0.87
0.90
0.28
0.27
3.12
f1 (ppm)


Me
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COOMe
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131.5113
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129.6638
122.3889
120.2621
118.1339
116.0103
99.5605
93.9728
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76.7881
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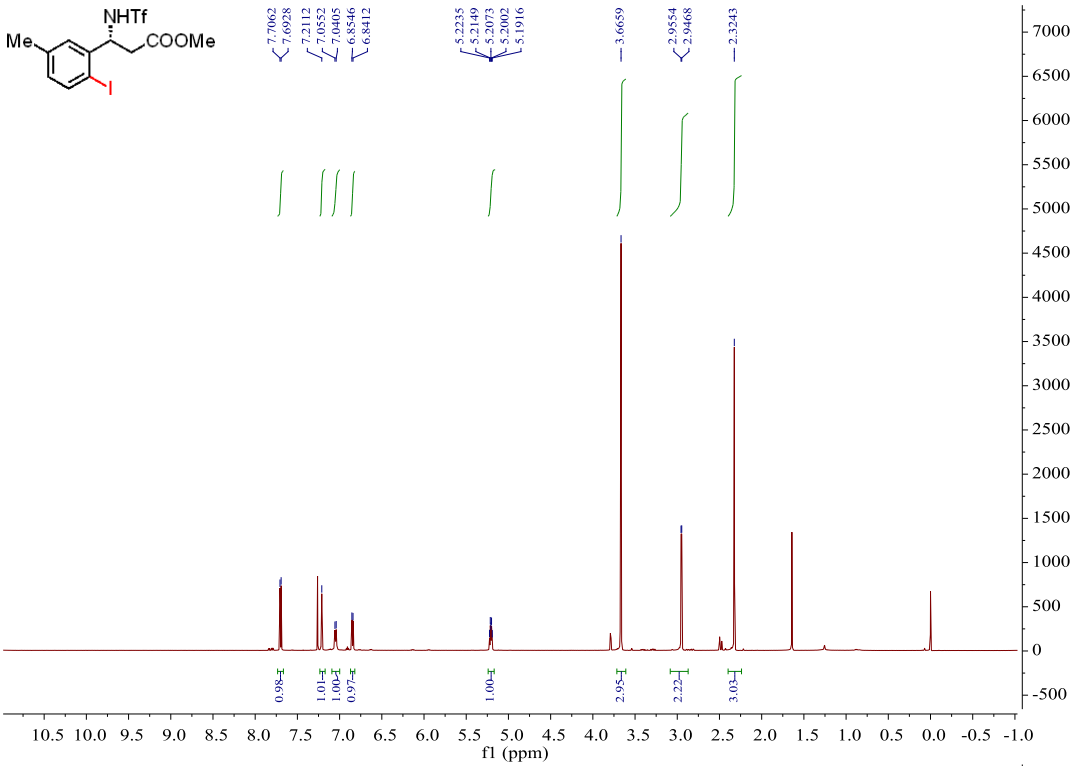
38.7948
21.2489
20.7046
f1 (ppm)


NHTf
Me
COOMe
I
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6.8412
5.2235
5.2149
5.2073
5.2002
5.1916
3.6659
2.9554
2.9468
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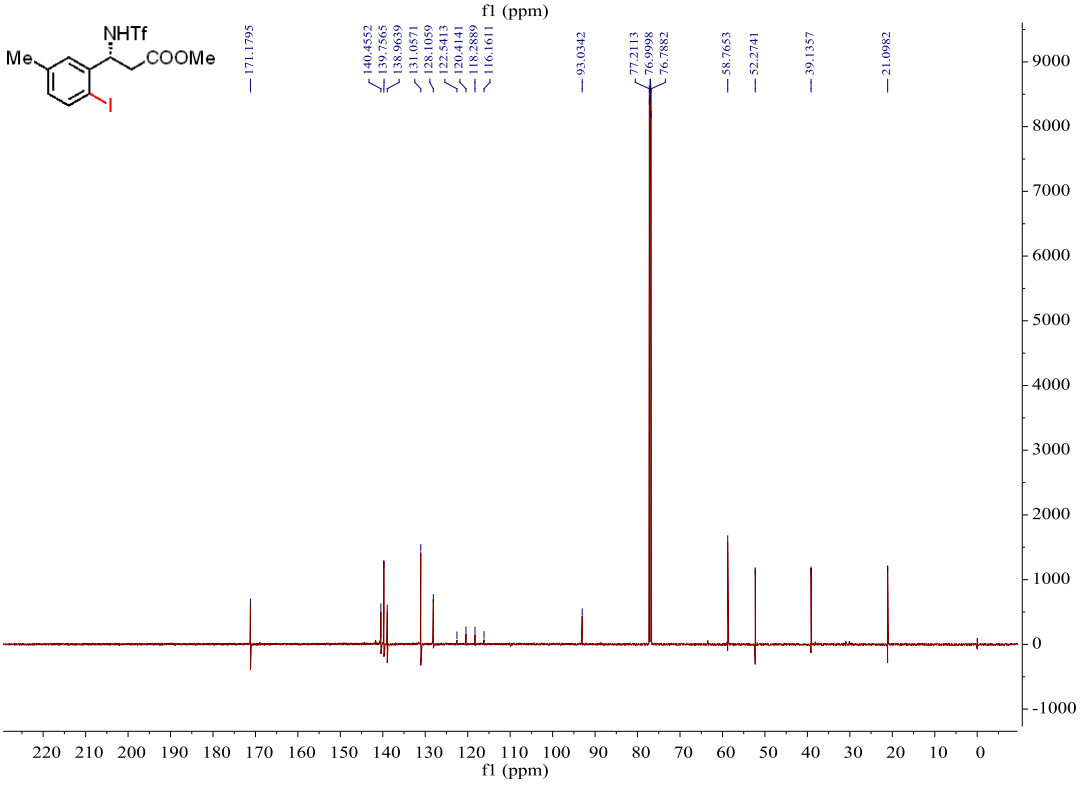
1.01
1.00
0.97
1.00
2.95
2.22
3.03
f1 (ppm)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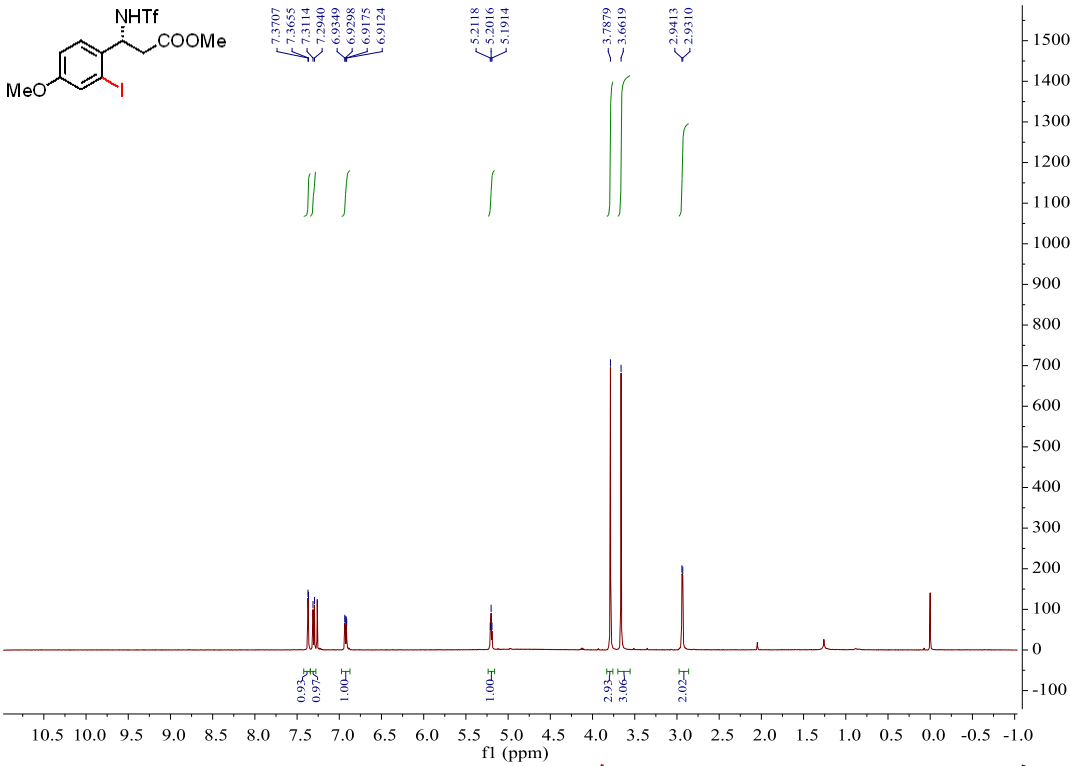
21.0982
f1 (ppm)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COOMe
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6.9175
6.9124
5.2118
5.2016
5.1914
3.7879
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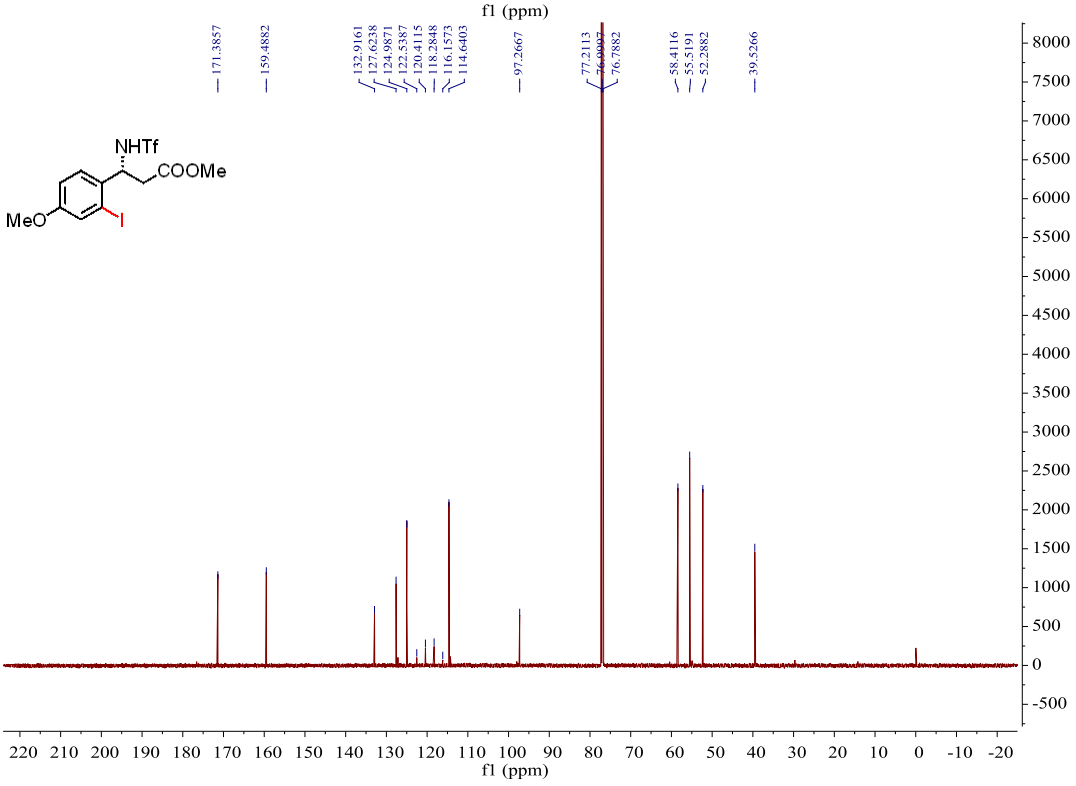
0.93
0.97
1.00
1.00
2.93
3.06
2.02
f1 (ppm)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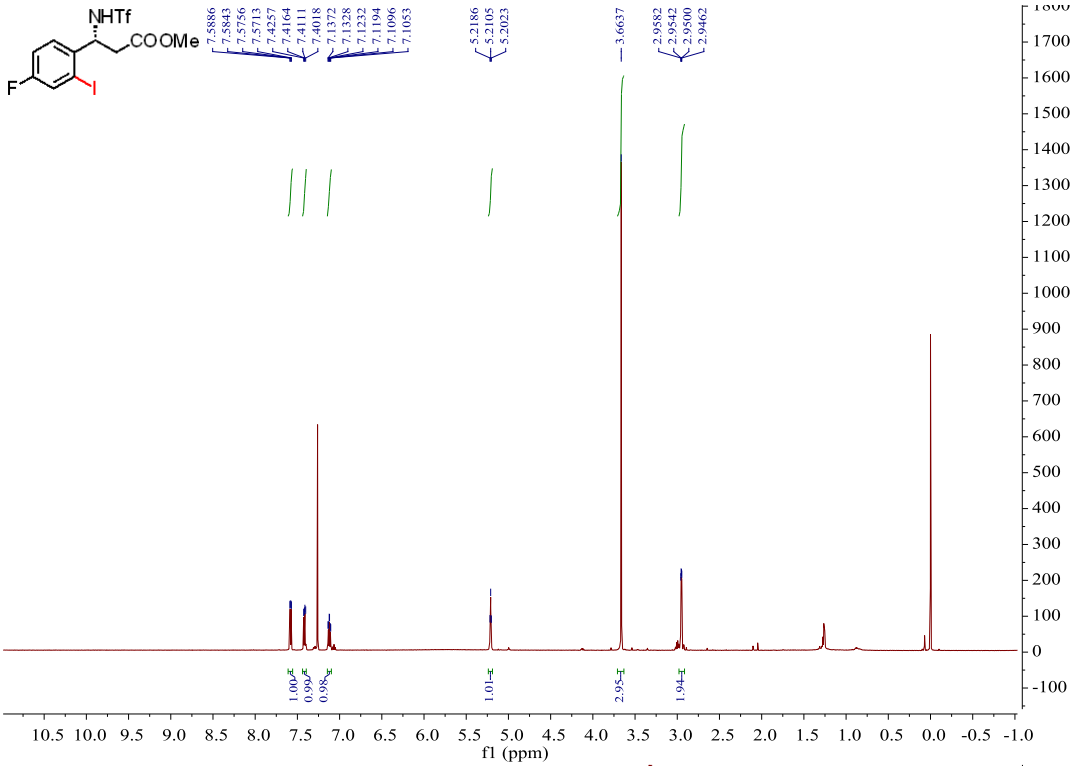
39.5266
f1 (ppm)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7.1328
7.1232
7.1194
7.1096
7.1053
5.2186
5.2105
5.2023
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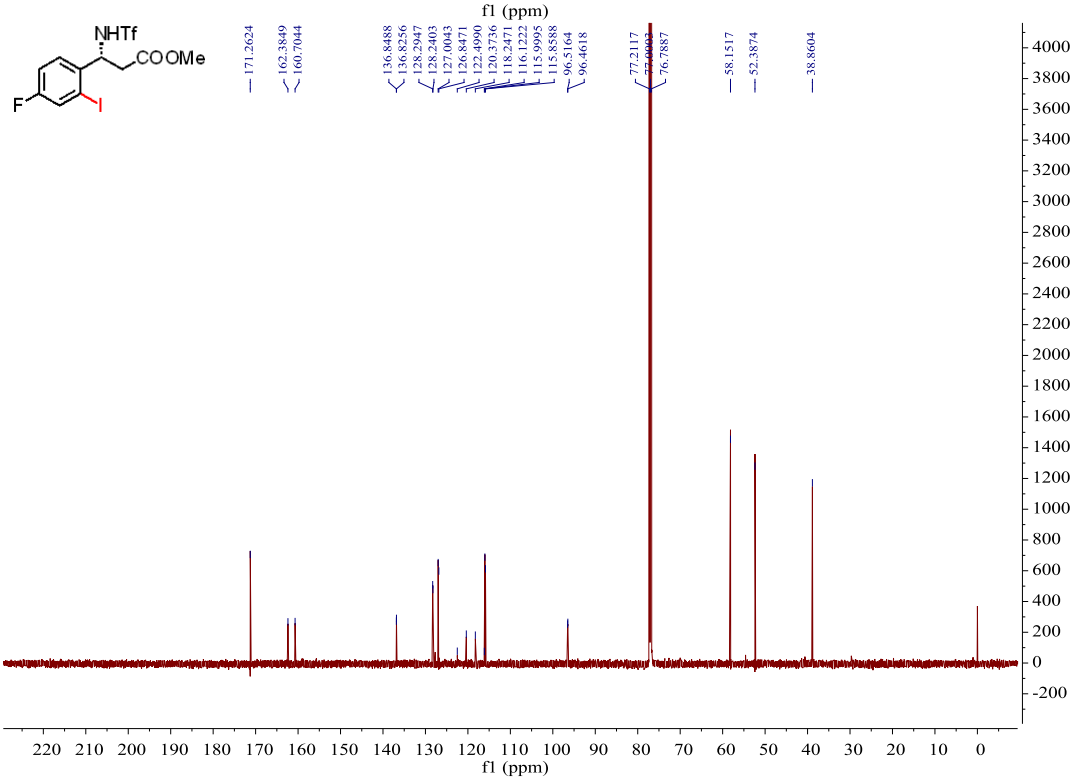
1.01
2.95
1.94
f1 (ppm)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127.0043
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118.2471
116.1222
115.9995
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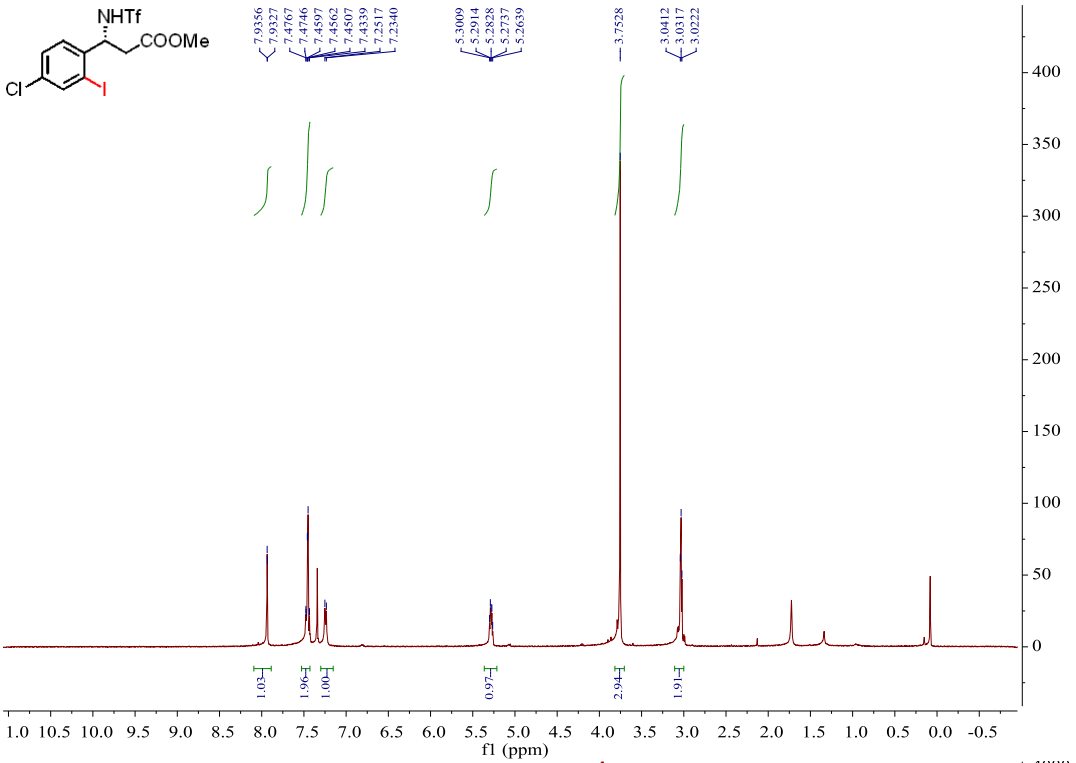
77.0003
76.7887
58.1517
52.3874
38.8604
f1 (ppm)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COOMe
Cl
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5.2828
5.2737
5.2639
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3.0222
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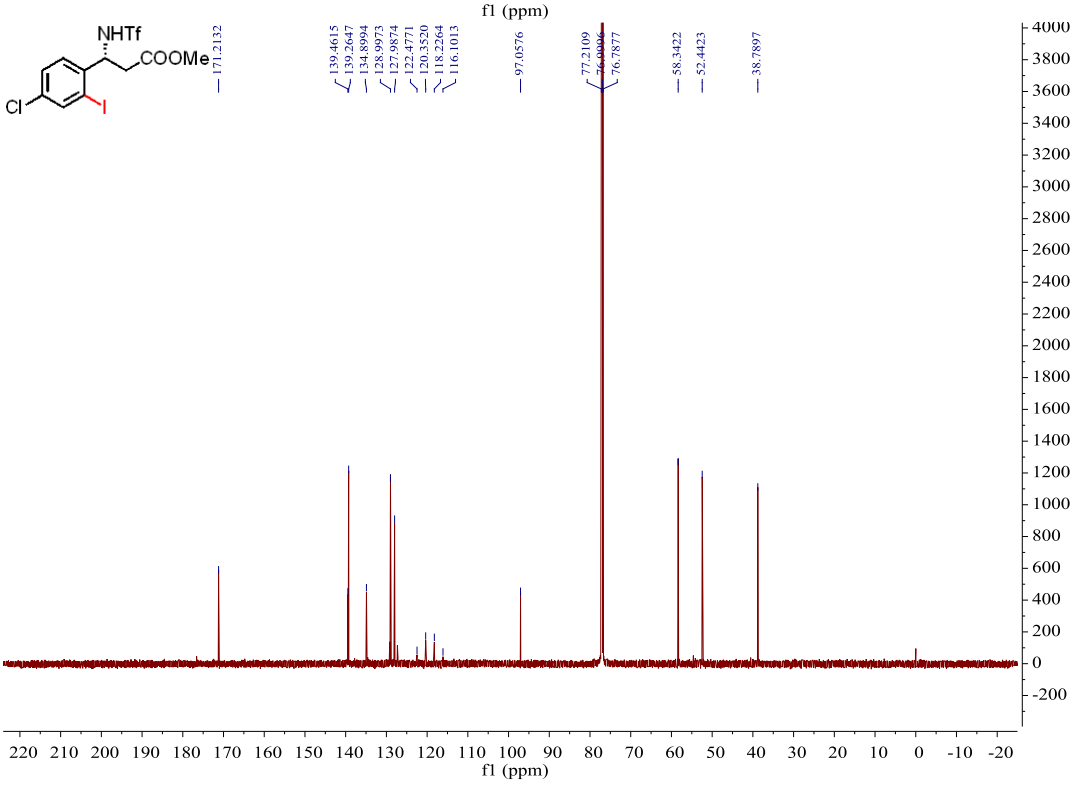
1.91
f1 (ppm)


NHTf
COOMe
Cl
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120.3520
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76.6006
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58.3422
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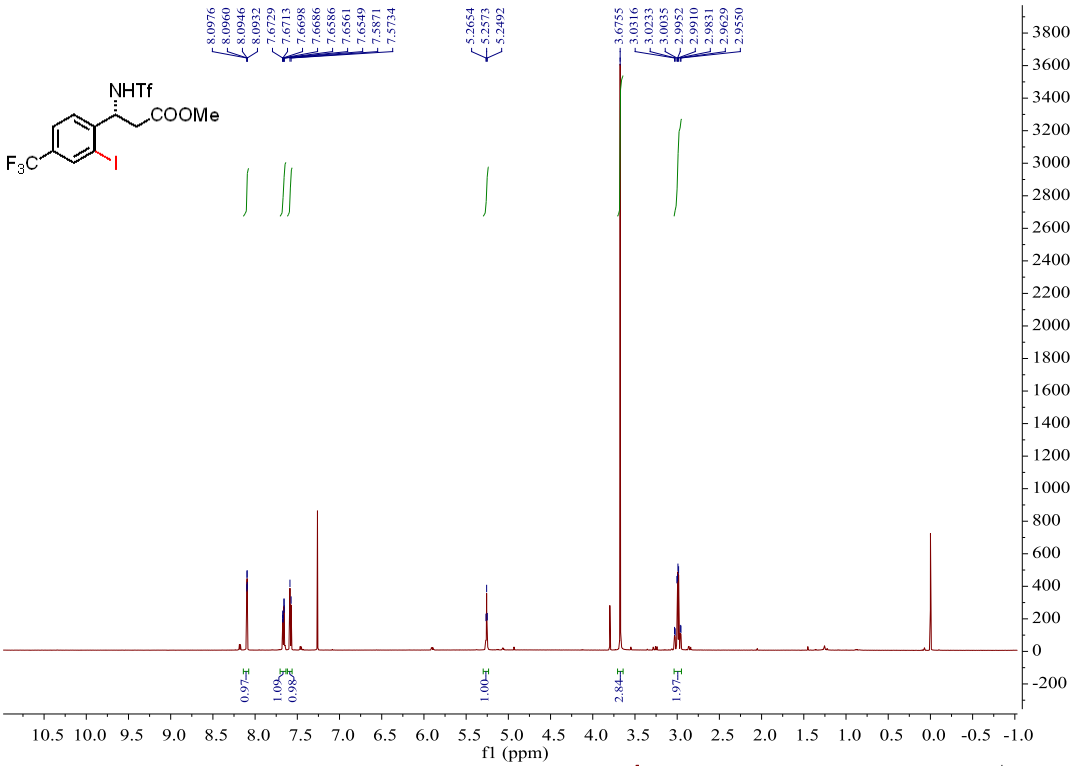
38.7897
f1 (ppm)


NHTf
COOMe
F3C
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7.6561
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7.5871
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5.2492
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3.0233
3.0035
2.9952
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2.9550
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1.00
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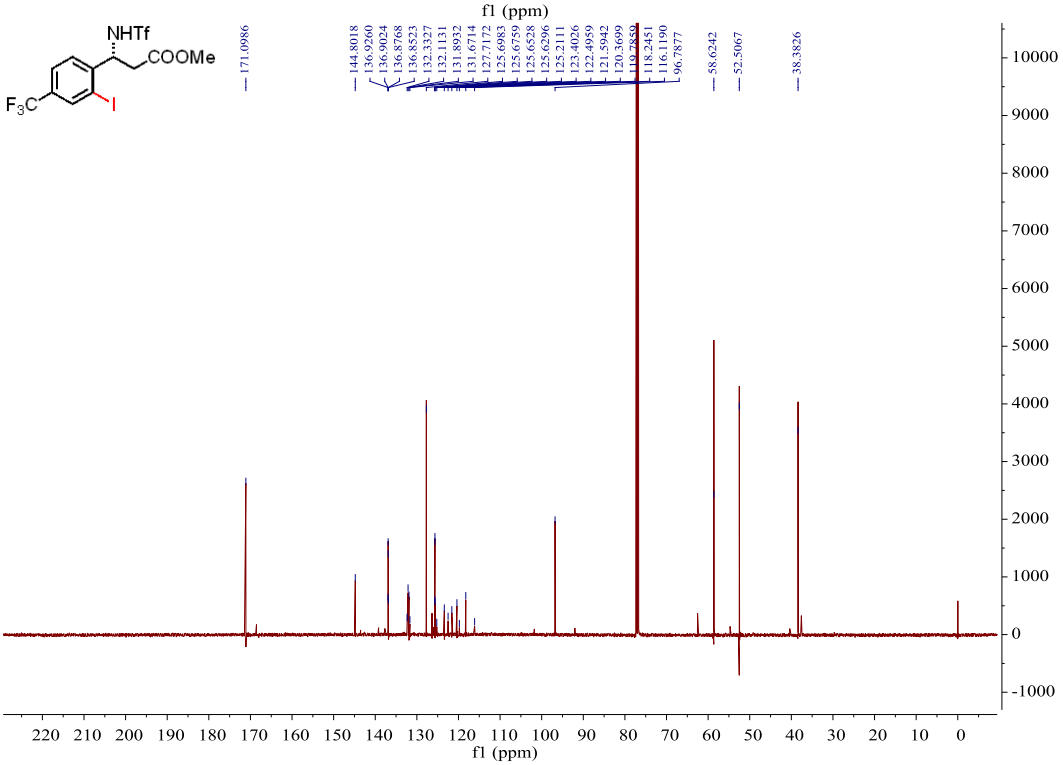
1.97
f1 (ppm)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COOMe
F3C
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125.6296
125.2111
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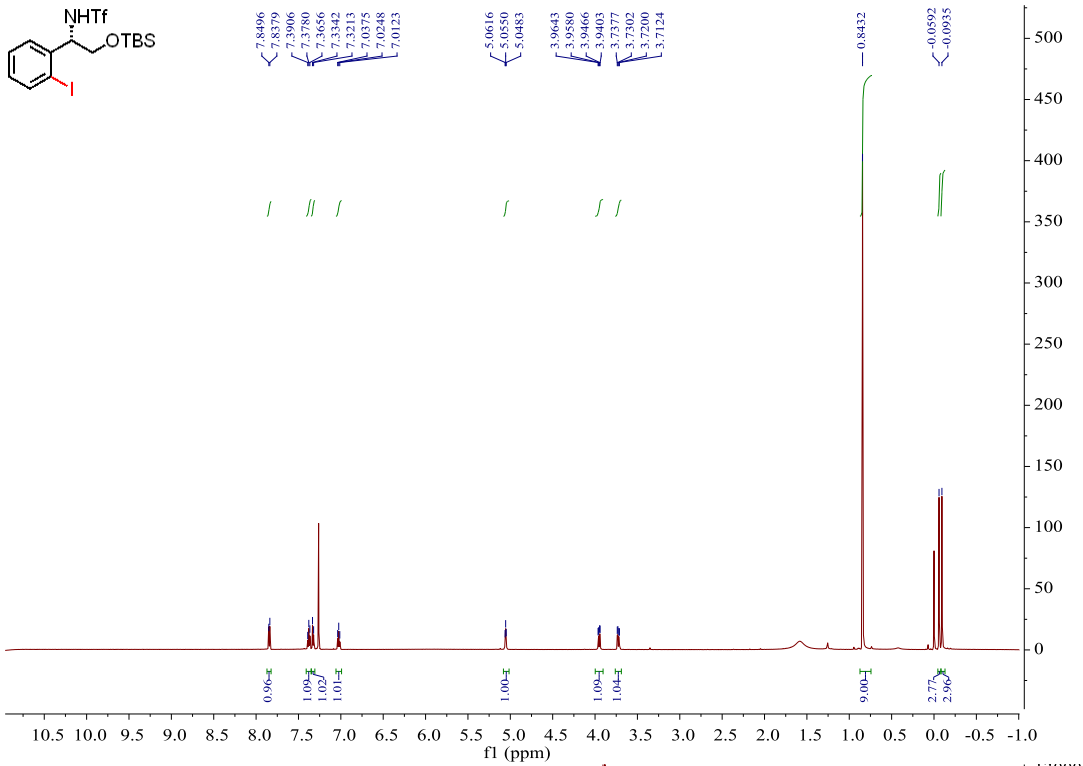
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OTBS
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7.0123
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5.0550
5.0483
3.9643
3.9580
3.9466
3.9403
3.7377
3.7302
3.7200
3.7124
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-0.0935
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1.02
1.01
1.00
1.09
1.04
9.00
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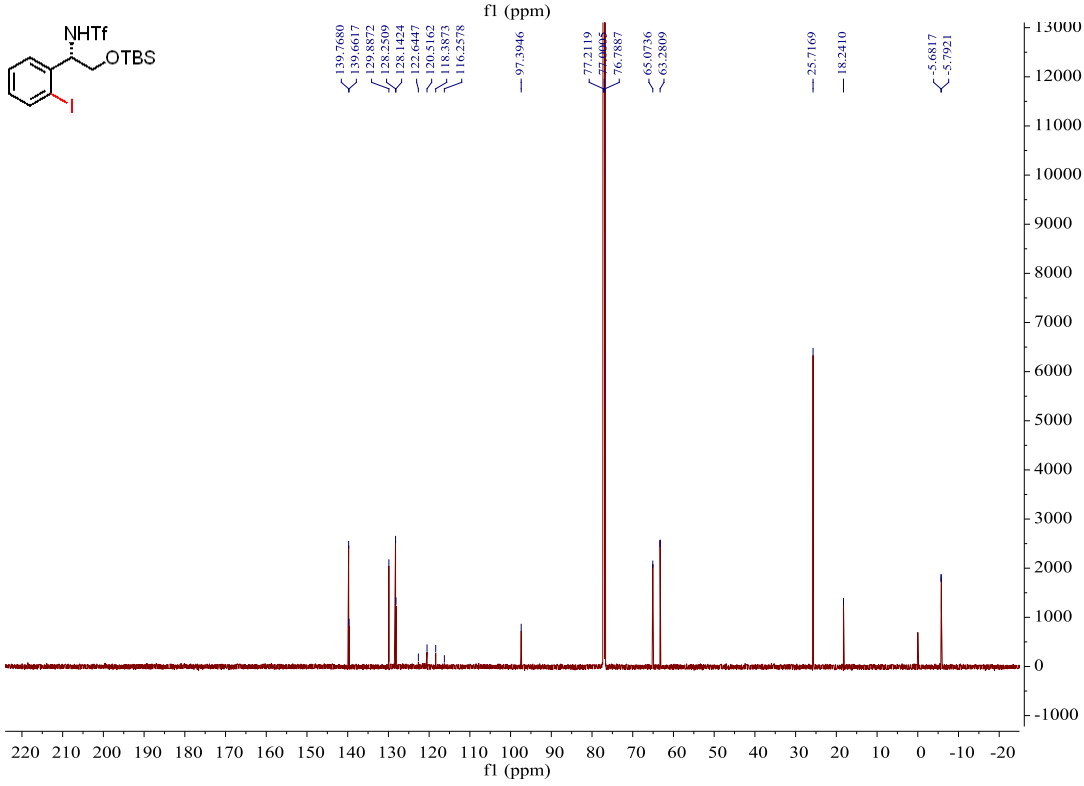
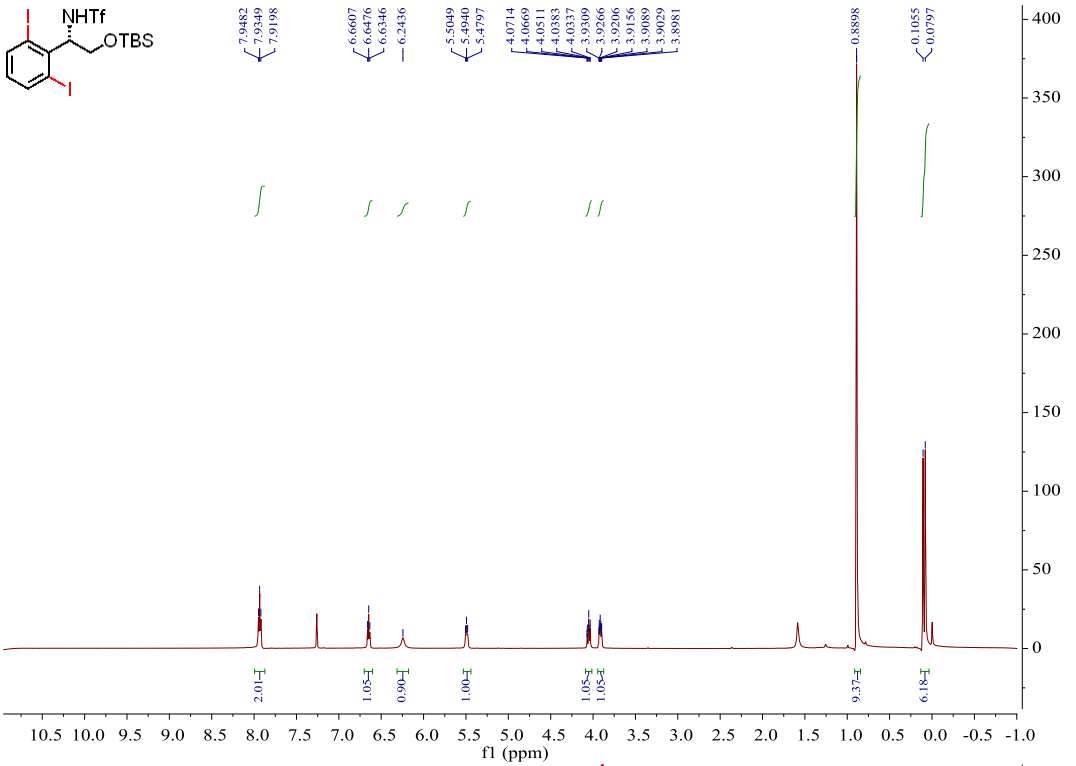
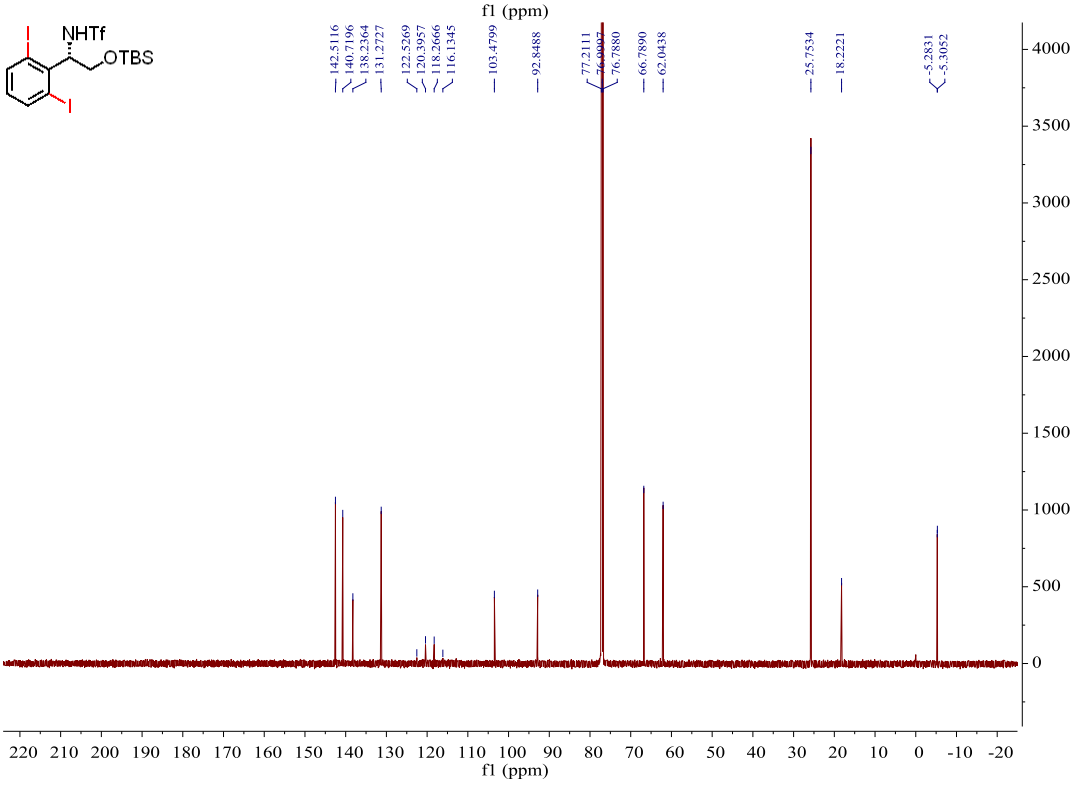
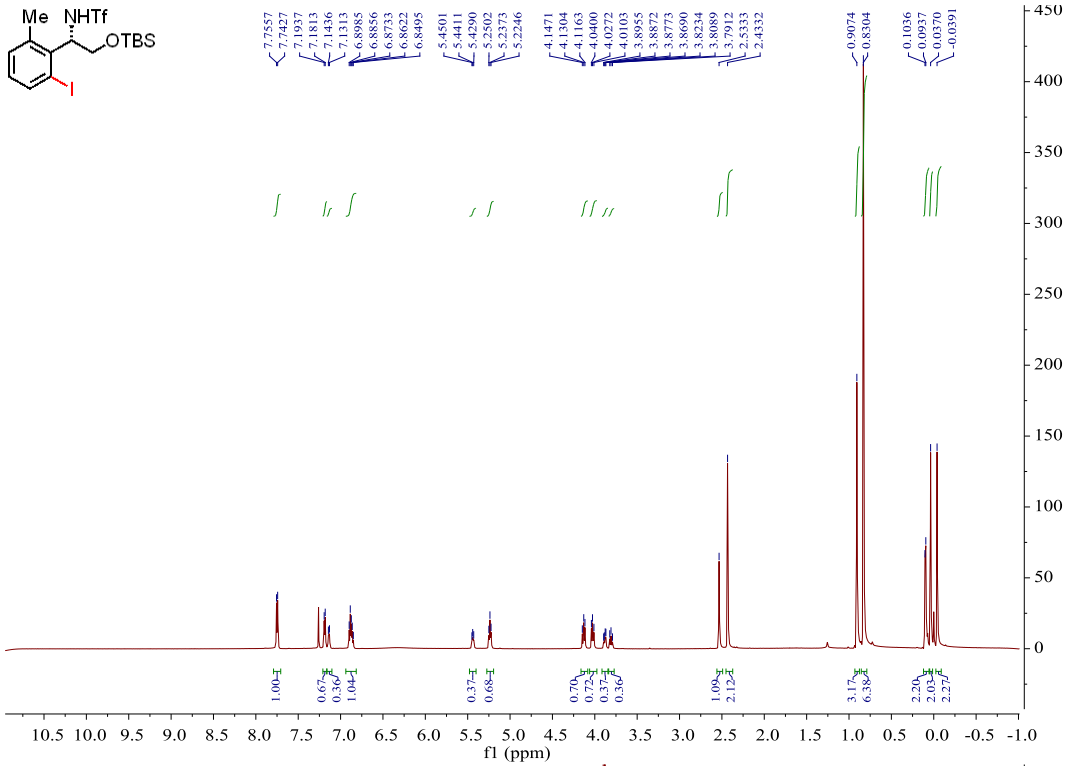
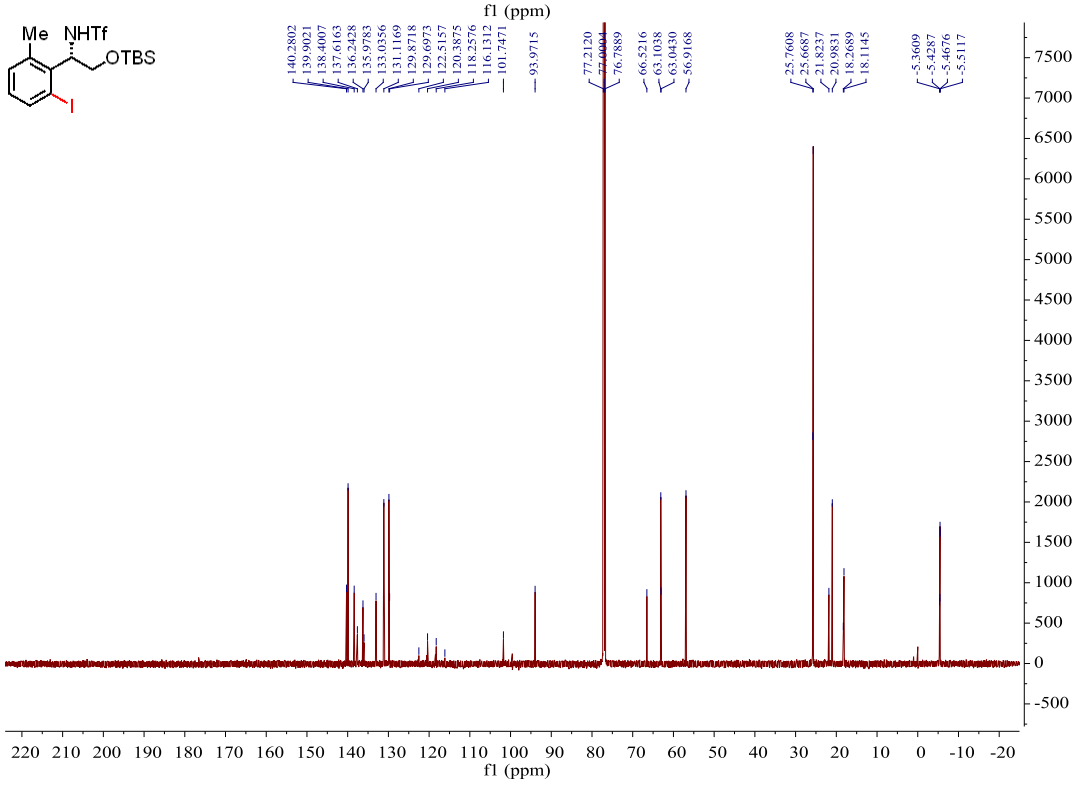
2.96
f1 (ppm)

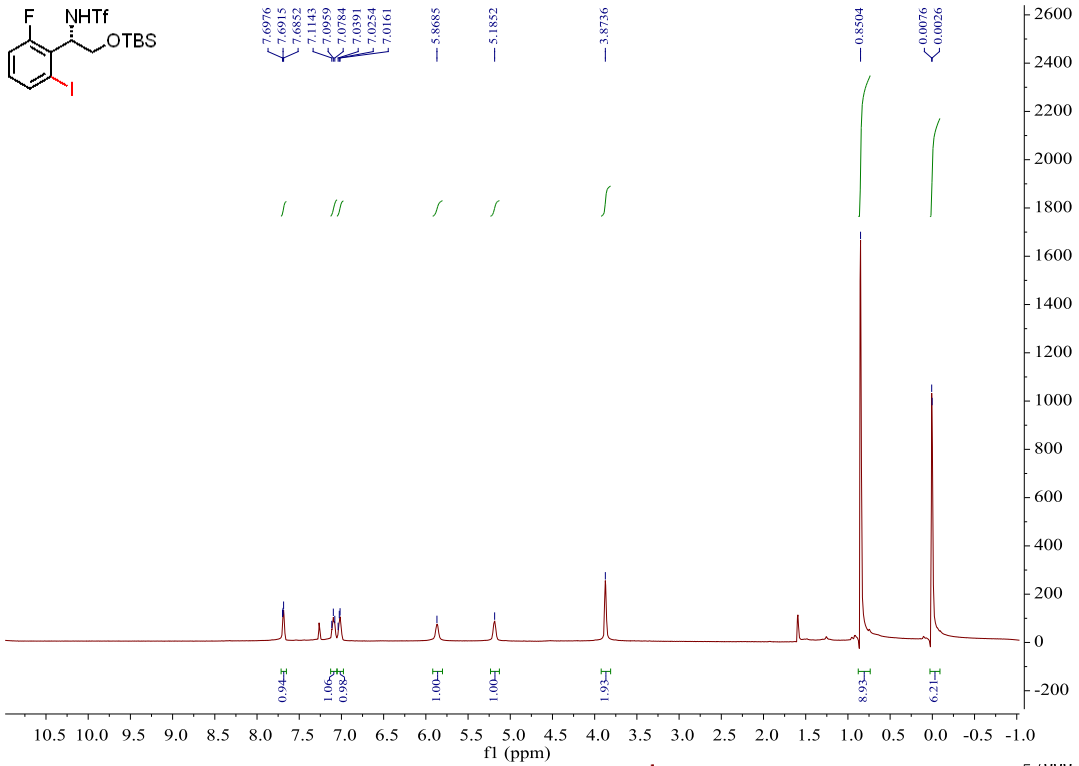

NHTf
OTBS
I
139.7680
139.6617
129.8872
128.2509
128.1424
122.6447
120.5162
118.3873
116.2578
97.3946
77.2119
77.0005
76.7887
65.0736
63.2809
25.7169
18.2410
-5.6817
-5.7921
f1 (ppm)

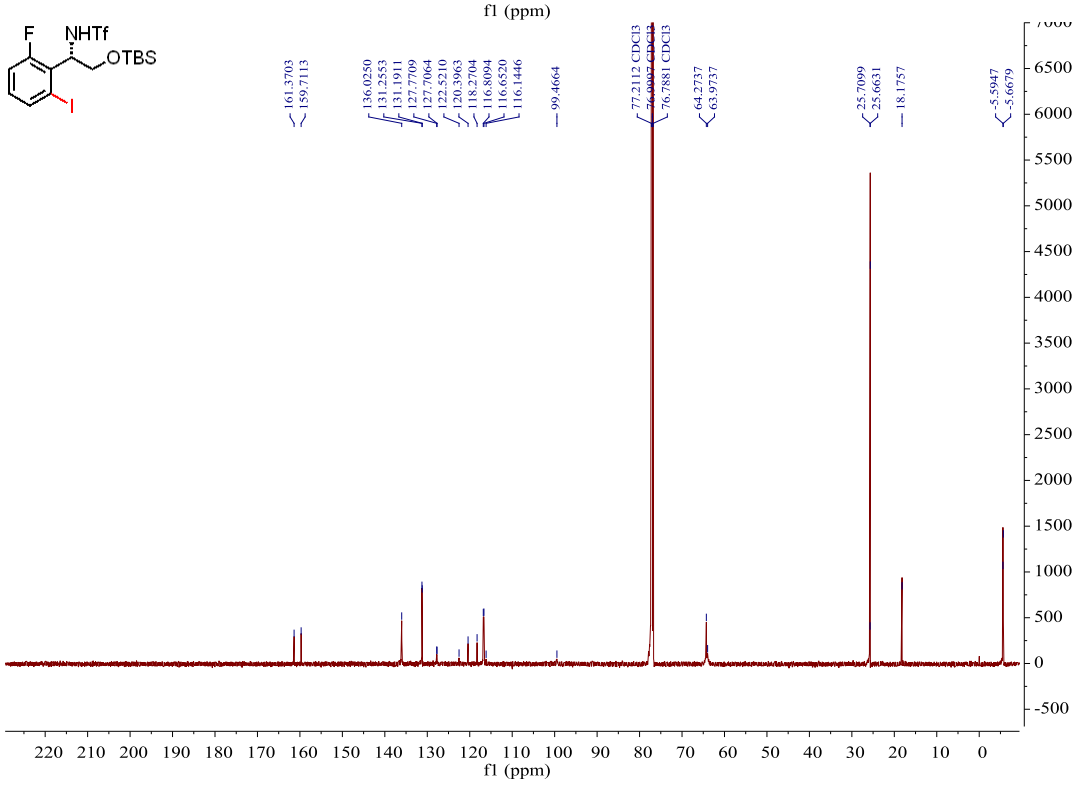

NHTf
OTBS
I
I
7.9482
7.9349
7.9198
6.6607
6.6476
6.6346
6.2436
5.5049
5.4940
5.4797
4.0714
4.0669
4.0511
4.0383
4.0337
3.9309
3.9266
3.9206
3.9156
3.9089
3.9029
3.8981
0.8898
0.1055
0.0797
2.01
1.05
0.90
1.00
1.05
1.05
9.37
6.18
f1 (ppm)

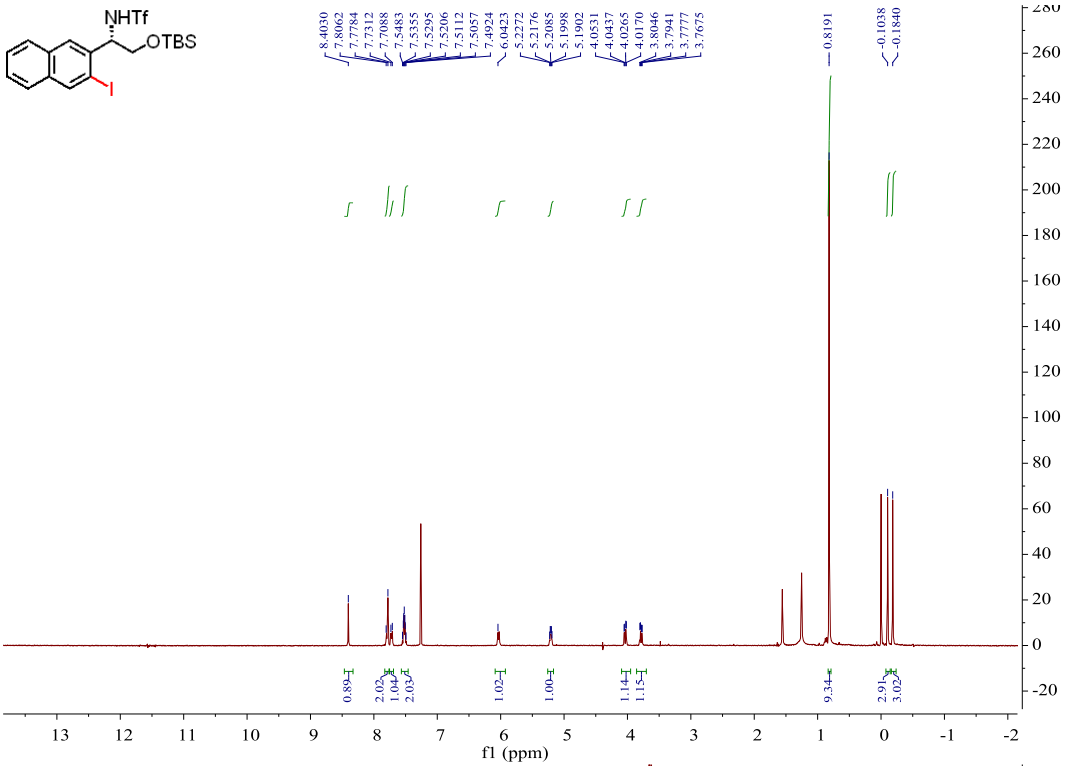

NHTf
OTBS
I
I
142.5116
140.7196
138.2364
131.2727
122.5269
120.3957
118.2666
116.1345
103.4799
92.8488
77.2111
77.0002
76.7880
66.7890
62.0438
25.7534
18.2221
-5.2831
-5.3052
f1 (ppm)

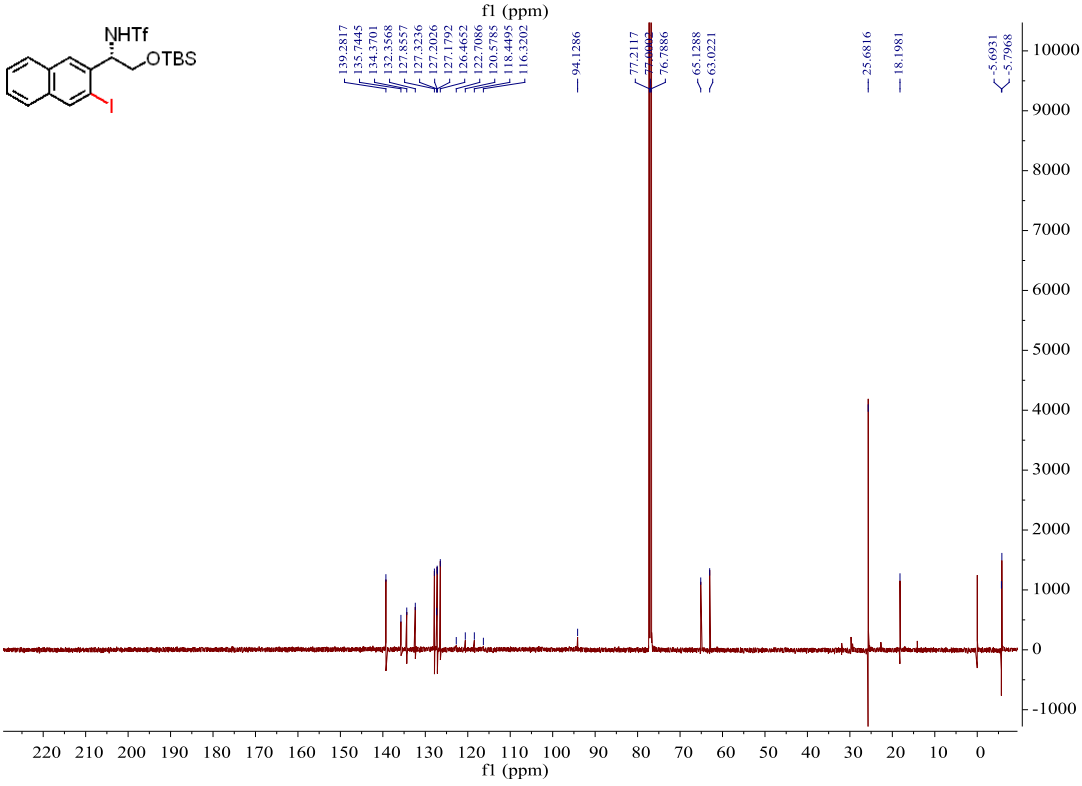

Me
NHTf
OTBS
I
7.7557
7.7427
7.1937
7.1813
7.1436
7.1313
6.8985
6.8856
6.8733
6.8622
6.8495
5.4501
5.4411
5.4290
5.2502
5.2373
5.2246
4.1471
4.1304
4.1163
4.0400
4.0272
4.0103
3.8955
3.8872
3.8773
3.8690
3.8234
3.8089
3.7912
2.5333
2.4332
0.9074
0.8304
0.1036
0.0937
0.0370
-0.0391
1.00
0.67
0.36
1.04
0.37
0.68
0.70
0.72
0.37
0.36
1.09
2.12
3.17
6.38
2.20
2.03
2.27
f1 (ppm)

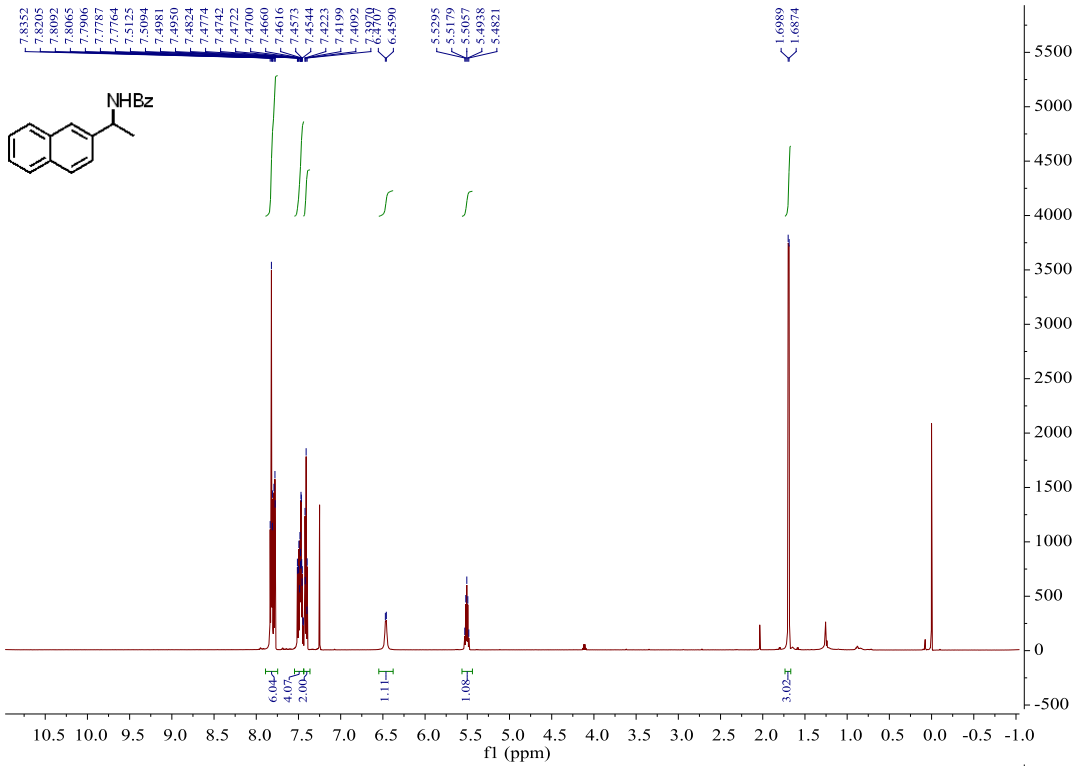

Me
NHTf
OTBS
I
140.2802
139.9021
138.4007
137.6163
136.2428
135.9783
133.0356
131.1169
129.8718
129.6973
122.5157
120.3875
118.2576
116.1312
101.7471
93.9715
77.2120
77.0004
76.7889
66.5216
63.1038
63.0430
56.9168
25.7608
25.6687
21.8237
20.9831
18.2689
18.1145
-5.3609
-5.4287
-5.4676
-5.5117
f1 (ppm)

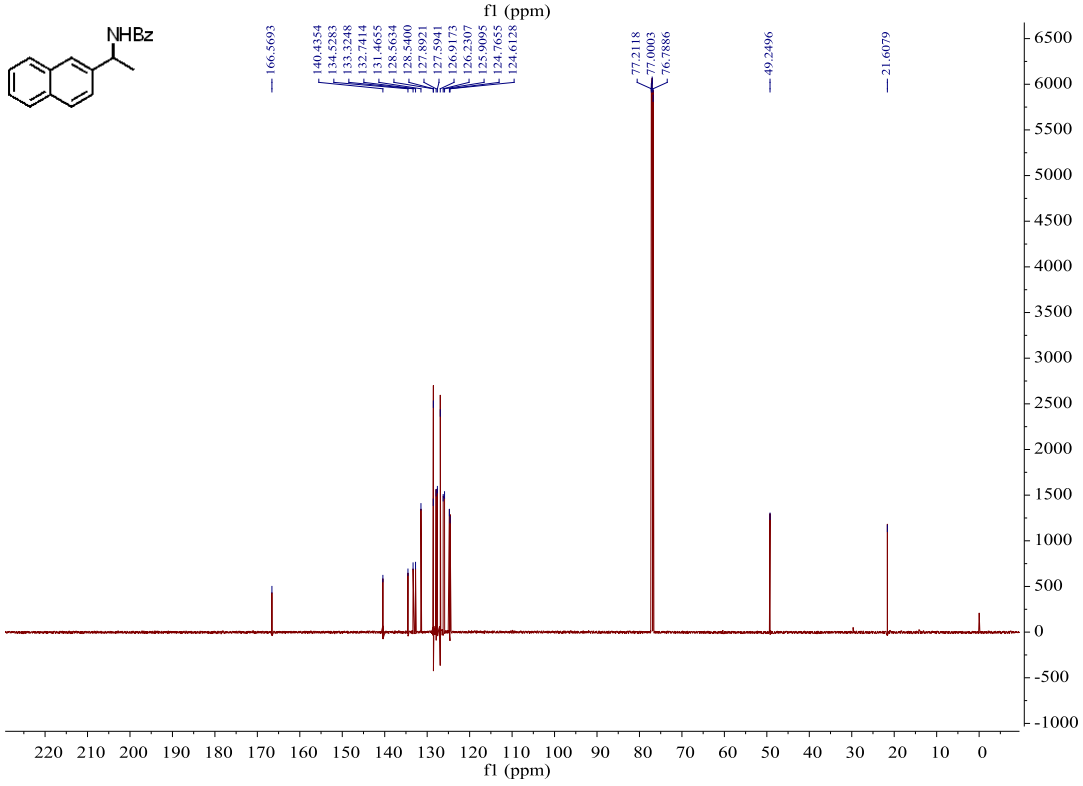

F
NHTf
OTBS
I
7.6976
7.6915
7.6852
7.1143
7.0959
7.0784
7.0391
7.0254
7.0161
5.8685
5.1852
3.8736
0.8504
0.0076
0.0026
0.94
1.06
0.98
1.00
1.00
1.93
8.93
6.21
f1 (ppm)

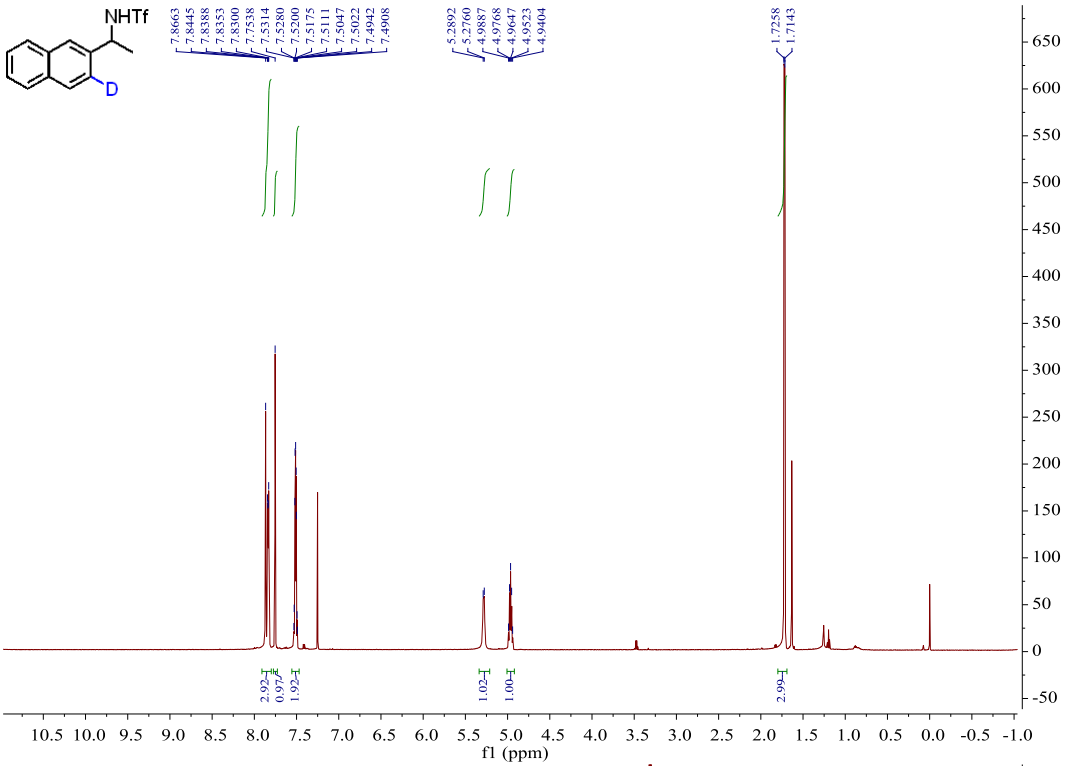

F
NHTf
OTBS
I
161.3703
159.7113
136.0250
131.2553
131.1911
127.7709
127.7064
122.5210
120.3963
118.2704
116.8094
116.6520
116.1446
99.4664
77.2112 CDCl3
76.7881 CDCl3
64.2737
63.9737
25.7099
25.6631
18.1757
-5.5947
-5.6679
f1 (ppm)

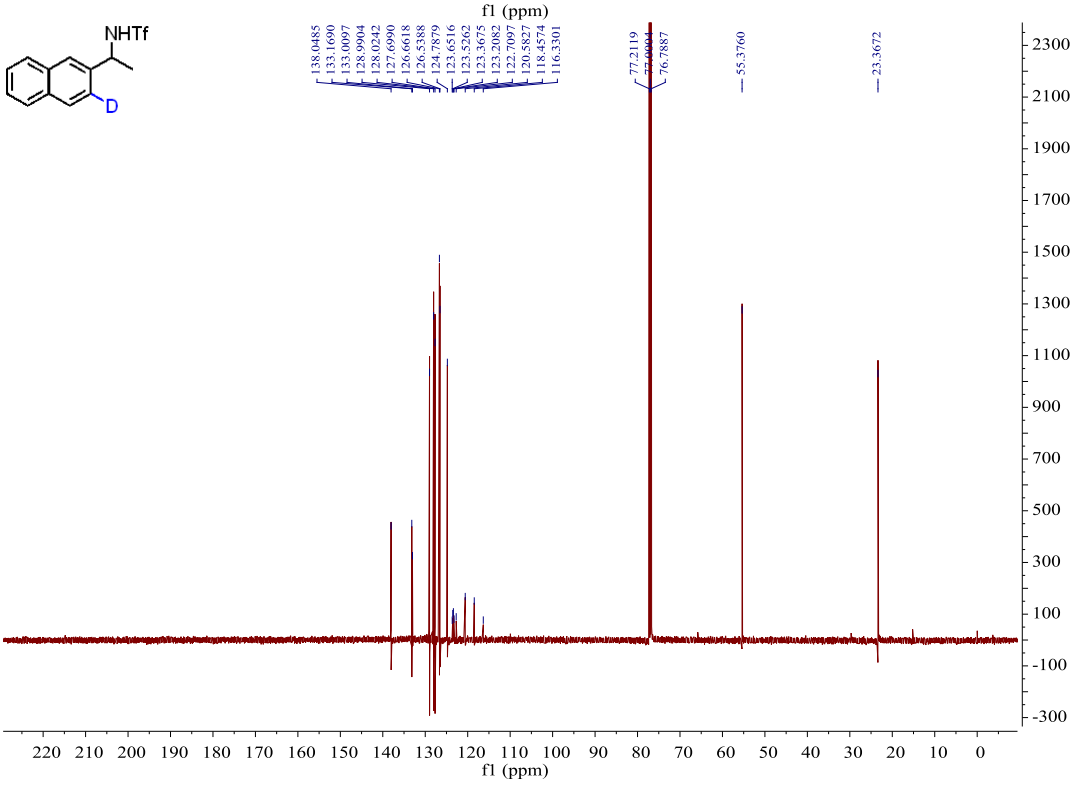

NHTf
OTBS
I
f1 (ppm)

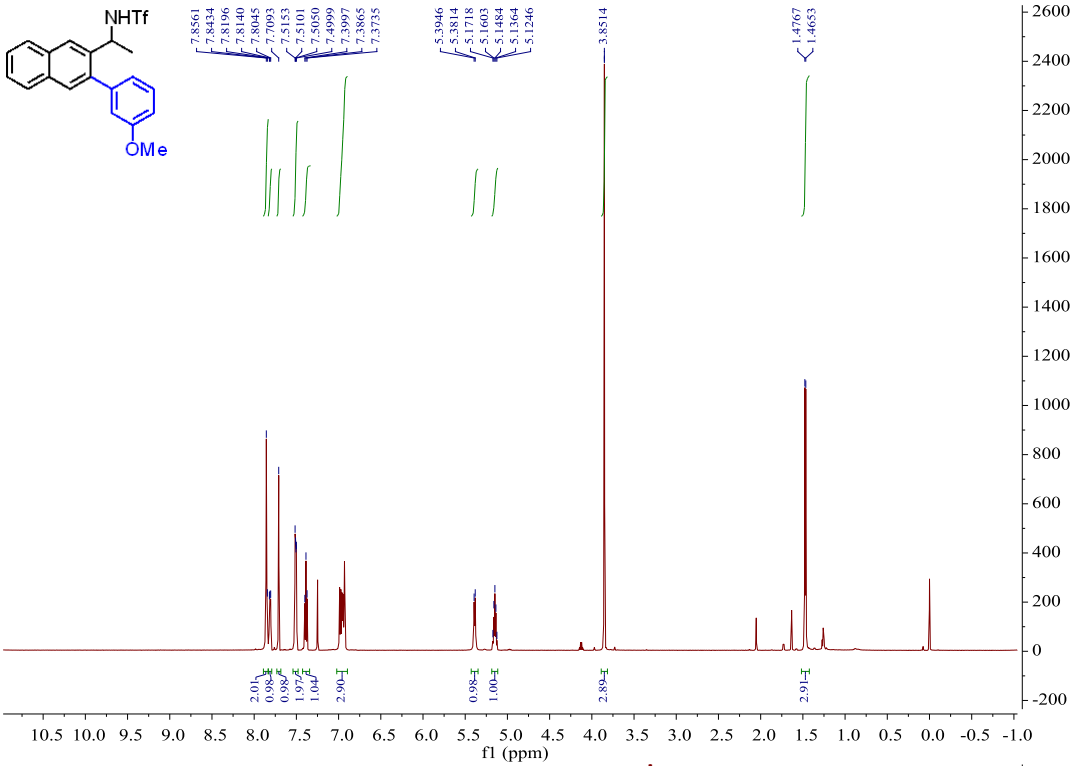

NHTf
OTBS
I
f1 (ppm)

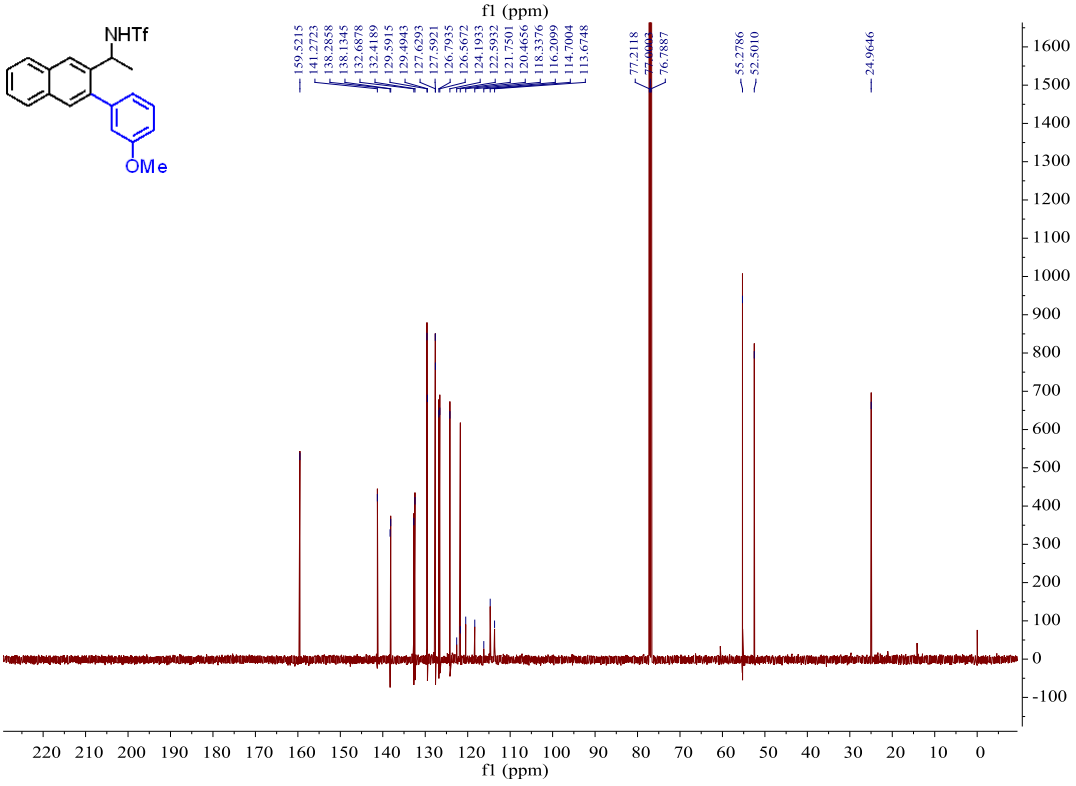

NHBz
7.8352
7.8205
7.8092
7.8065
7.7906
7.7787
7.7764
7.5125
7.5094
7.4981
7.4950
7.4824
7.4774
7.4742
7.4722
7.4700
7.4660
7.4616
7.4573
7.4544
7.4223
7.4199
7.4092
7.3970
6.4707
6.4590
5.5295
5.5179
5.5057
5.4938
5.4821
1.6989
1.6874
6.04
4.07
2.00
1.11
1.08
3.02
f1 (ppm)

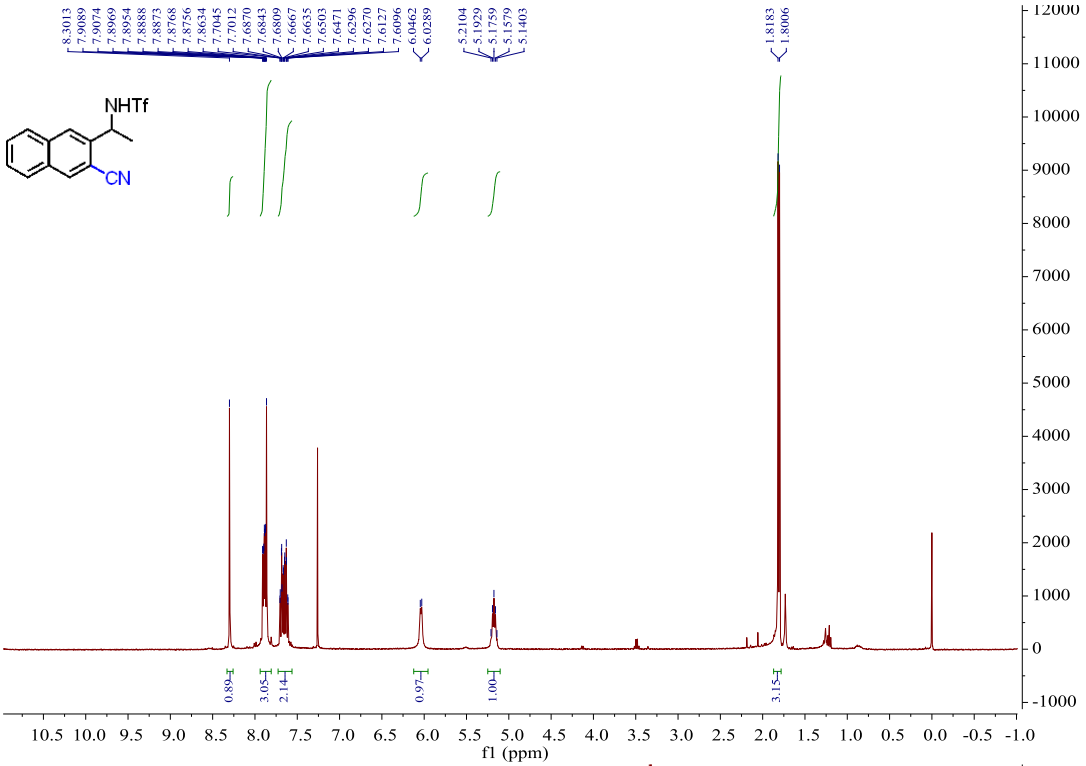

NHBz
166.5693
140.4354
134.5283
133.3248
132.7414
131.4655
128.5634
128.5400
127.8921
127.5941
126.9173
126.2307
125.9095
124.7655
124.6128
77.2118
77.0003
76.7886
49.2496
21.6079
f1 (ppm)

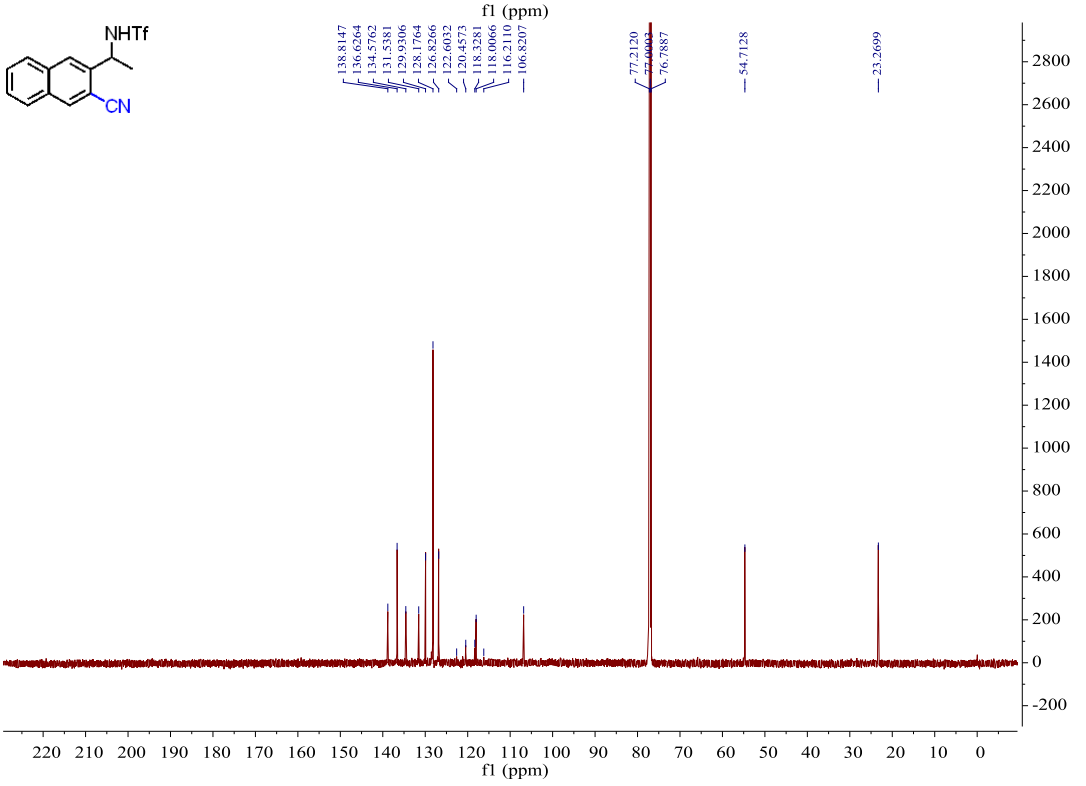

NHTf
D
7.8663
7.8445
7.8388
7.8353
7.8300
7.7538
7.5314
7.5280
7.5200
7.5175
7.5111
7.5047
7.5022
7.4942
7.4908
5.2892
5.2760
4.9887
4.9768
4.9647
4.9523
4.9404
1.7258
1.7143
2.92
0.97
1.92
1.02
1.00
2.99
f1 (ppm)

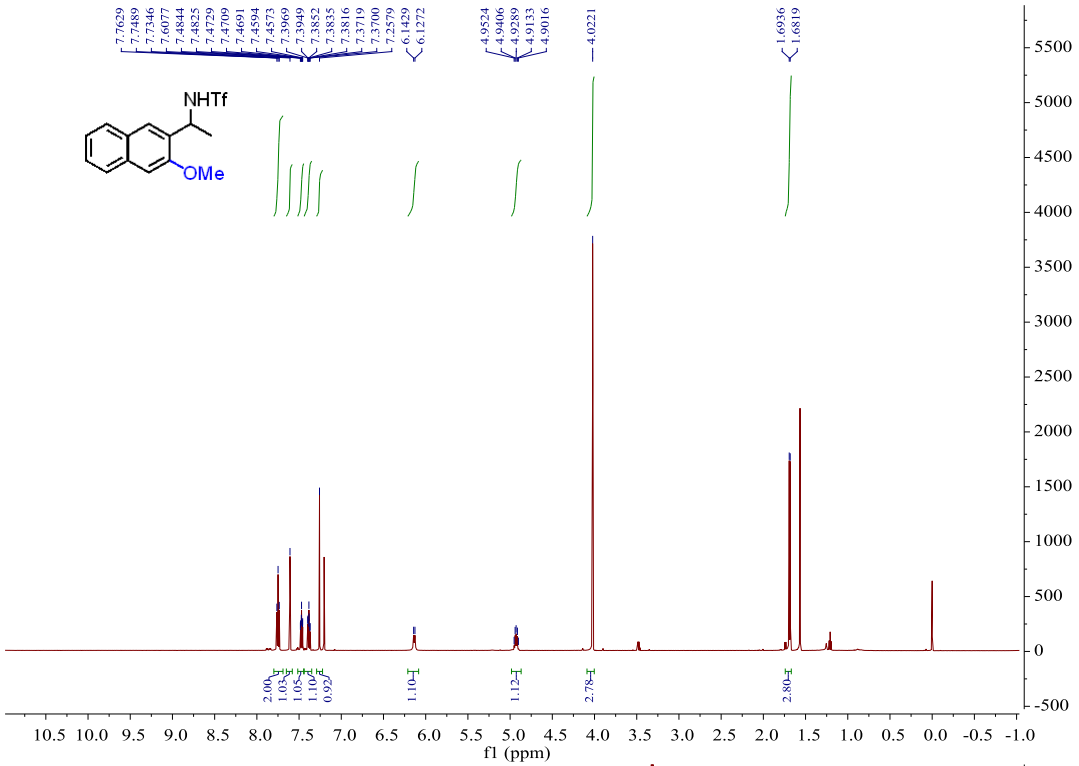

NHTf
D
138.0485
133.1690
133.0097
128.9904
128.0242
127.6990
126.6618
126.5388
124.7879
123.6516
123.5262
123.3675
123.2082
122.7097
120.5827
118.4574
116.3301
77.2119
77.0004
76.7887
55.3760
23.3672
f1 (ppm)

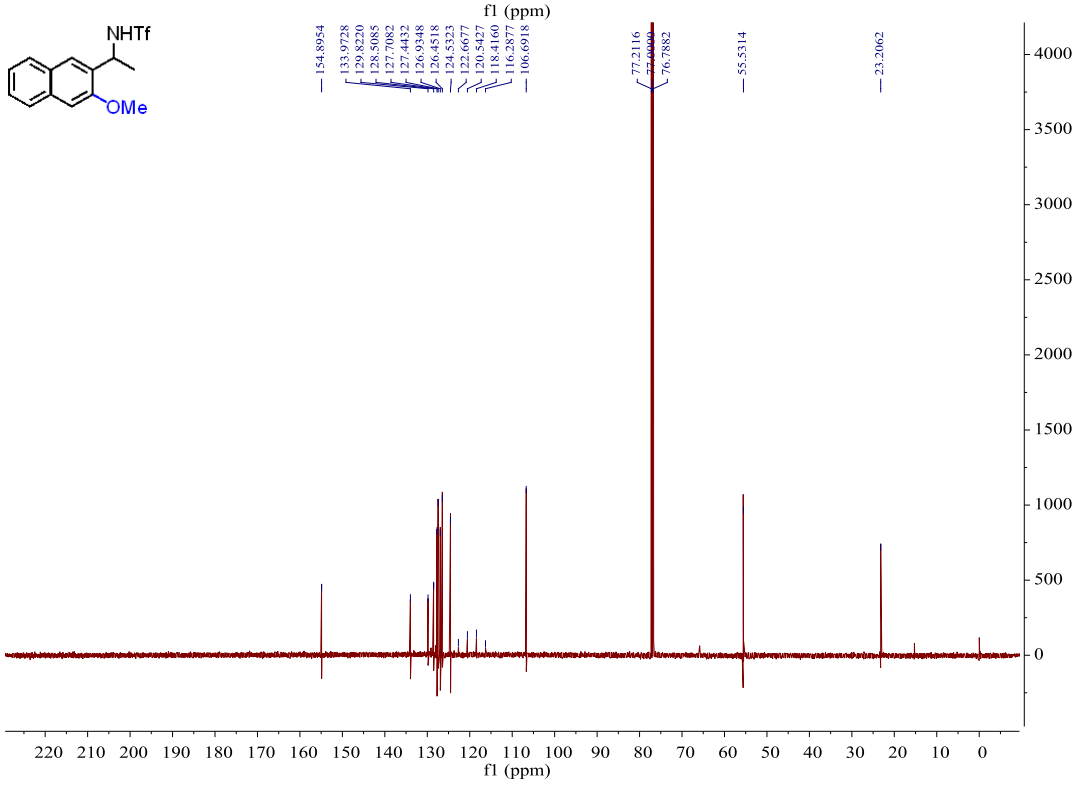

NHTf
OMe
7.8561
7.8434
7.8196
7.8140
7.8045
7.7093
7.5153
7.5101
7.5050
7.4999
7.3997
7.3865
7.3735
5.3946
5.3814
5.1718
5.1603
5.1484
5.1364
5.1246
3.8514
1.4767
1.4653
2.01
0.98
0.98
1.97
1.04
2.90
0.98
1.00
2.89
2.91
f1 (ppm)

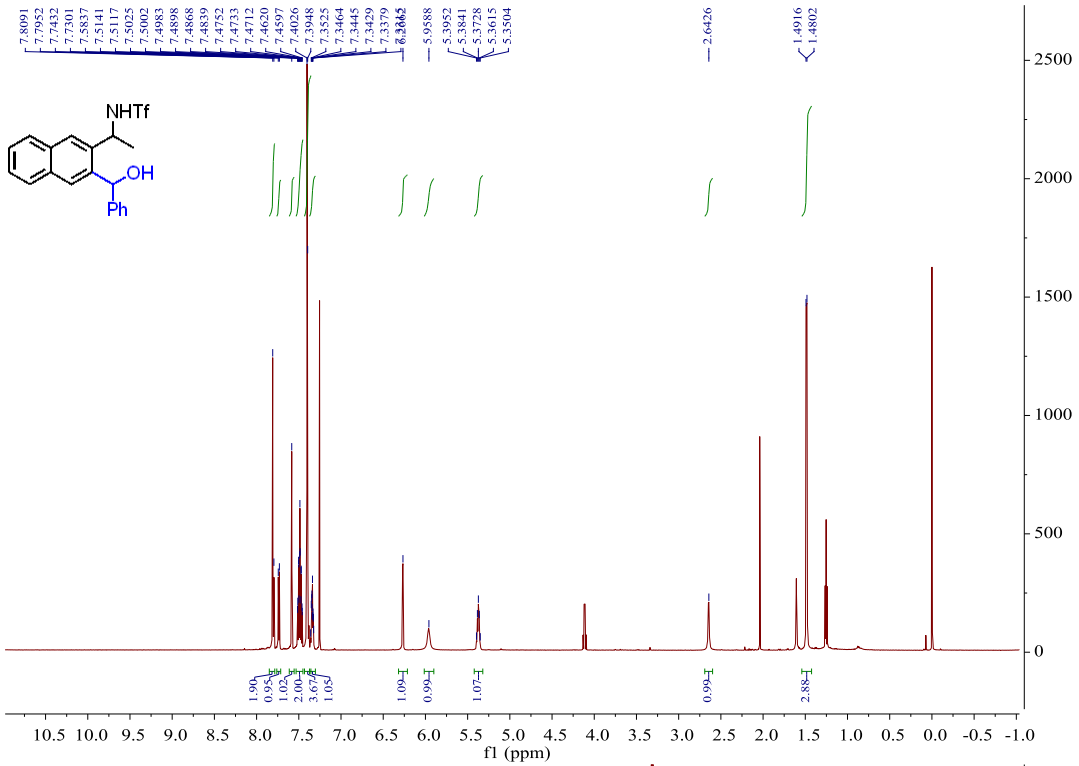

NHTf
OMe
159.5215
141.2723
138.2858
138.1345
132.6878
132.4189
129.5915
129.4943
127.6293
127.5921
126.7935
126.5672
124.1933
122.5932
121.7501
120.4656
118.3376
116.2099
114.7004
113.6748
77.2118
77.0003
76.7887
55.2786
52.5010
24.9646
f1 (ppm)

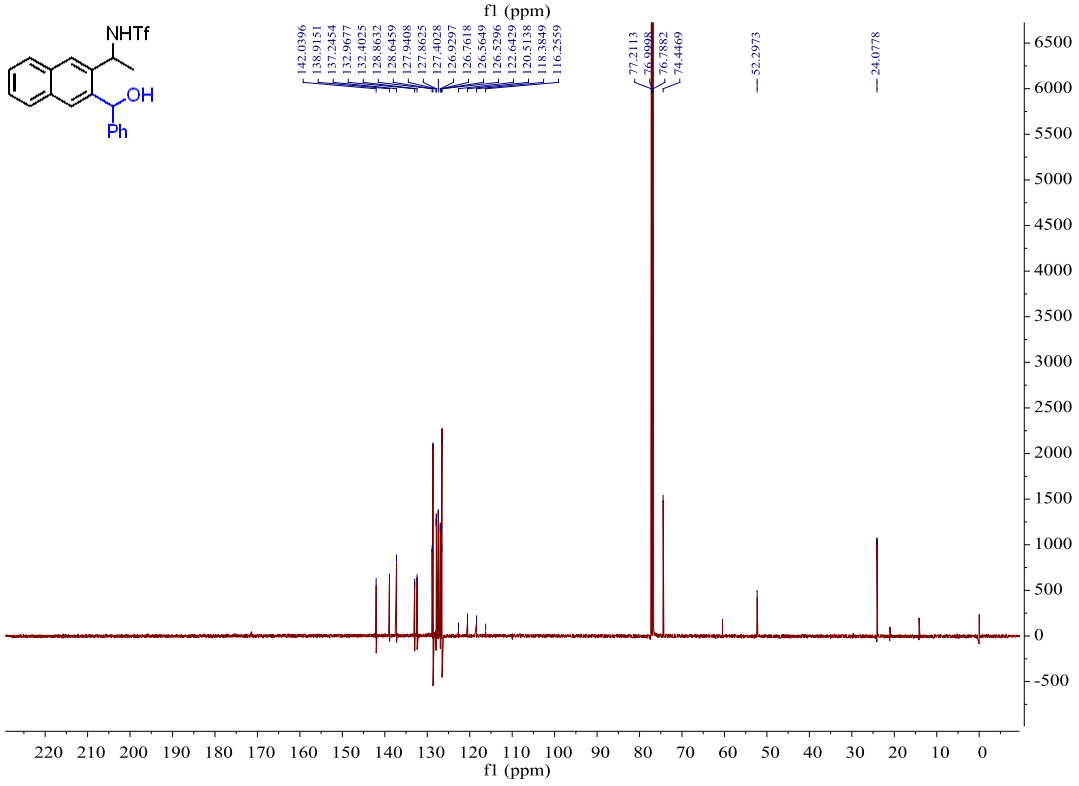

NHTf
CN
f1 (ppm)


NHTf
CN
f1 (ppm)


NHTf
OMe
7.7629
7.7489
7.7346
7.6077
7.4844
7.4825
7.4729
7.4709
7.4691
7.4594
7.4573
7.3969
7.3949
7.3852
7.3835
7.3816
7.3719
7.3700
7.2579
6.1429
6.1272
4.9524
4.9406
4.9289
4.9133
4.9016
4.0221
1.6936
1.6819
2.00
1.03
1.05
1.10
0.92
1.10
1.12
2.78
2.80
f1 (ppm)

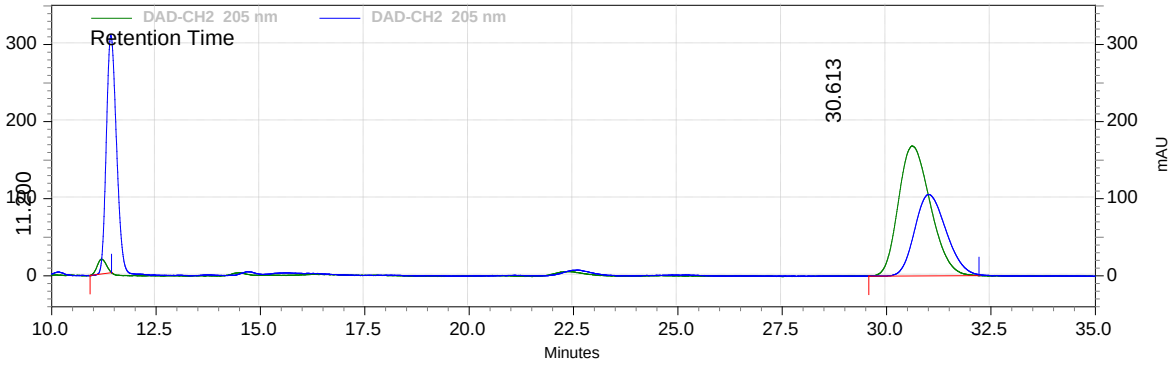

NHTf
OMe
154.8954
133.9728
129.8220
128.5085
127.7082
127.4432
126.9348
126.4518
124.5323
122.6677
120.5427
118.4160
116.2877
106.6918
77.2116
77.0000
76.7882
55.5314
23.2062
f1 (ppm)


NHTf
OH
Ph
f1 (ppm)


NHTf
OH
Ph
f1 (ppm)

2a
Area % Report

Data File: 1a-SM-C:\Documents and Settings\Yu lab hplc\Desktop\lingchu-kinetic resolution\CL07028-SM-ee-5%-0.5mL-40min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 9/2/2013 4:40:02 PM
Printed: 1/9/2014 9:17:59 PM



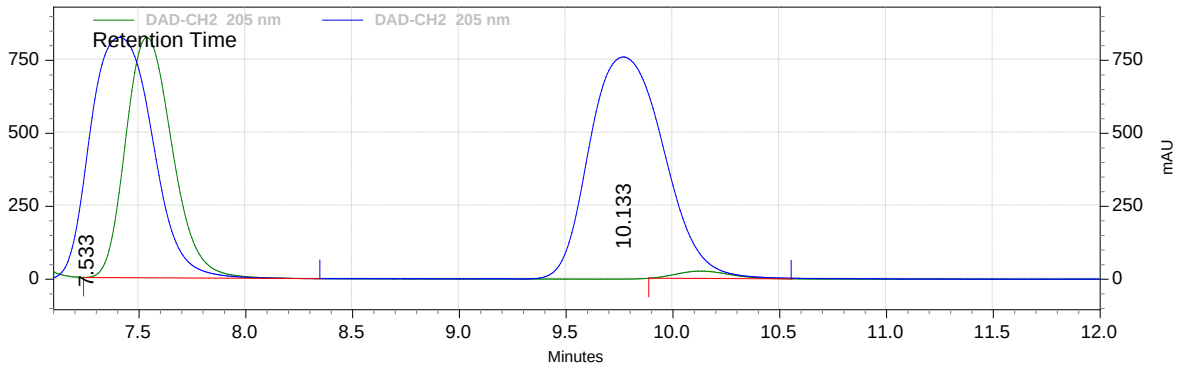
DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
11.200	1145874	3.09	76299	10.17
30.613	35976428	96.91	674125	89.83

Totals	37122302	100.00	750424	100.00

3a
Area % Report

Data File: 1a-PR-C:\Documents and Settings\Yu lab hplc\Desktop\lingchu-kinetic resolution\CL07028-PR-ee-5%-0.5mL-40min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 9/2/2013 5:21:37 PM
Printed: 1/9/2014 9:21:36 PM

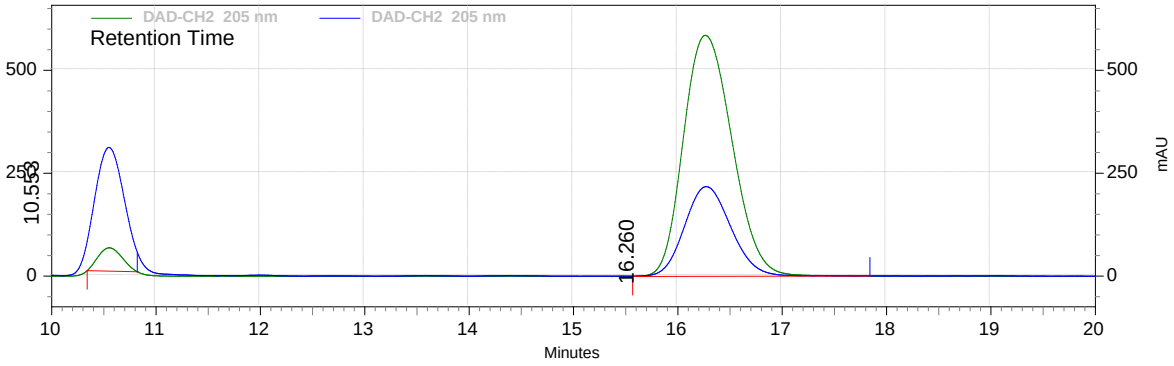


DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
7.533	50036964	96.61	3294342	97.02
10.133	1753692	3.39	101221	2.98
Totals	51790656	100.00	3395563	100.00

2b
Area % Report

Data File: 1b-SM-C:\Documents and Settings\Yu lab hplc\Desktop\lingchu-kinetic resolution\CL07026-SM-ee-5%-0.5mL-20min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 9/2/2013 12:48:50 PM
Printed: 1/9/2014 9:26:15 PM

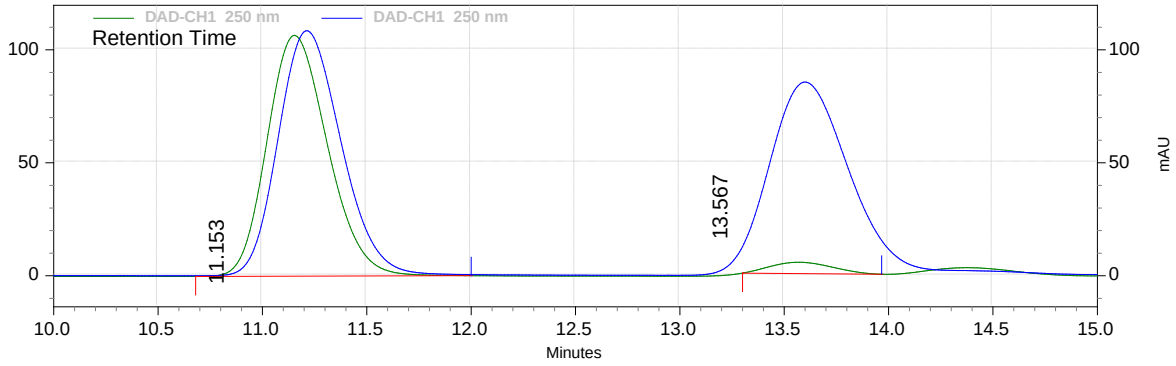


DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
10.553	3593116	4.51	226860	8.83
16.260	76022434	95.49	2341695	91.17
Totals	79615550	100.00	2568555	100.00

3b
Area % Report

Data File: 1b-PR-C:\Documents and Settings\Yu lab hplc\Desktop\lingchu-kinetic resolution\CL07026-PR-ee-5%-0.5mL-20min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 9/2/2013 1:31:36 PM
Printed: 1/9/2014 9:29:42 PM

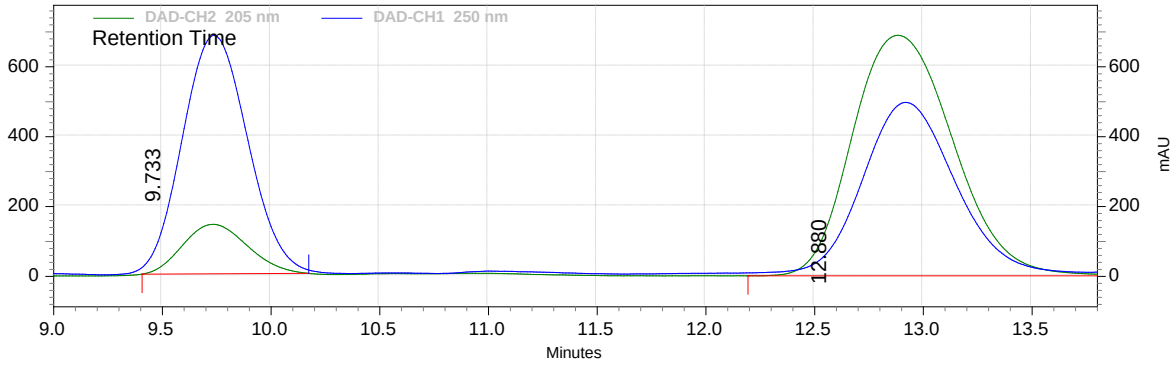


DAD-CH1 250 nm Results

Retention Time	Area	Area %	Height	Height %
11.153	8587300	95.40	426316	95.51
13.567	414327	4.60	20051	4.49
Totals	9001627	100.00	446367	100.00

2c
Area % Report

Data File: 1c-SM-C:\Documents and Settings\Yu lab hplc\Desktop\lingchu-kinetic resolution\CL07024-SM-ee-5%-0.5mL-20min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 9/2/2013 2:14:19 PM
Printed: 1/9/2014 9:32:42 PM

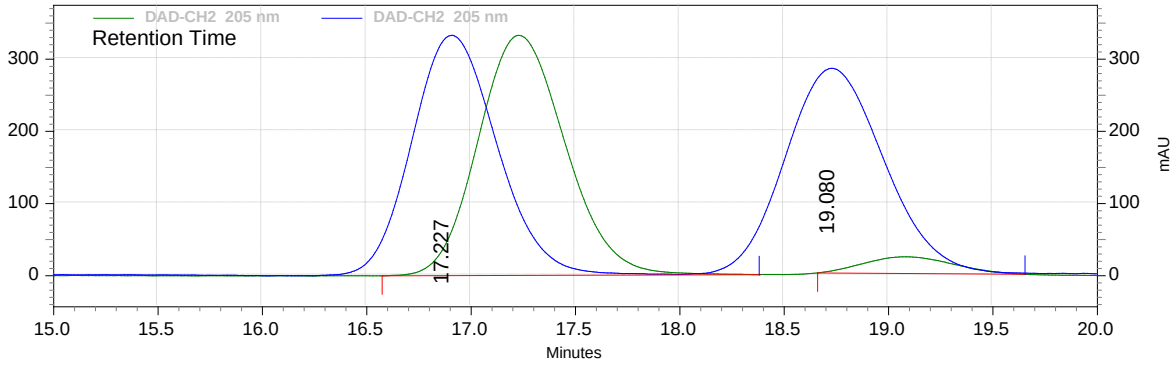


DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
9.733	11711510	11.63	566571	17.06
12.880	89015576	88.37	2753515	82.94
Totals	100727086	100.00	3320086	100.00

3c
Area % Report

Data File: 1c-PR-C:\Documents and Settings\Yu lab hplc\Desktop\lingchu-kinetic resolution\CL07024-PR-ee-5%-0.3mL-40min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 9/2/2013 3:17:06 PM
Printed: 1/9/2014 9:36:01 PM

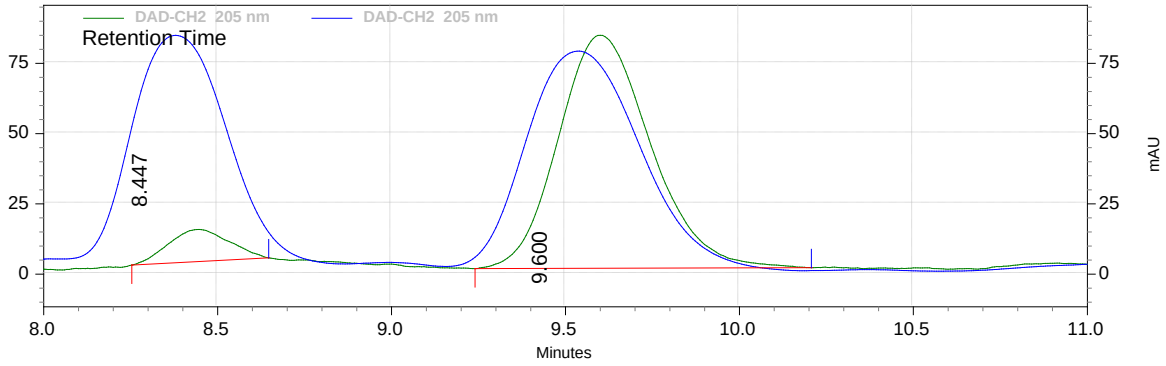


DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
17.227	38692545	93.36	1330248	93.49
19.080	2750934	6.64	92660	6.51
Totals	41443479	100.00	1422908	100.00

2d
Area % Report

Data File: 1f-SM-C:\Documents and Settings\Yu lab hplc\Desktop\lingchu-kinetic resolution\CL07099-2-sm-ee-5%-0.5mL-20min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 10/19/2013 1:18:18 PM
Printed: 1/9/2014 9:57:34 PM

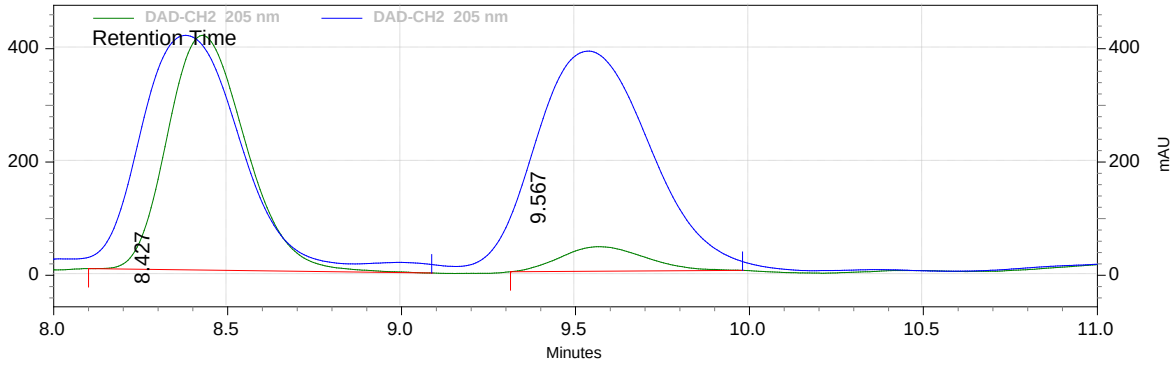


DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
8.447	553170	8.29	45644	12.10
9.600	6121454	91.71	331511	87.90
Totals	6674624	100.00	377155	100.00

3d
Area % Report

Data File: 1f-PR-C:\Documents and Settings\Yu lab hplc\Desktop\lingchu-kinetic resolution\CL07099-pr-ee-5%-0.5mL-20min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 10/19/2013 1:35:39 PM
Printed: 1/9/2014 10:00:46 PM

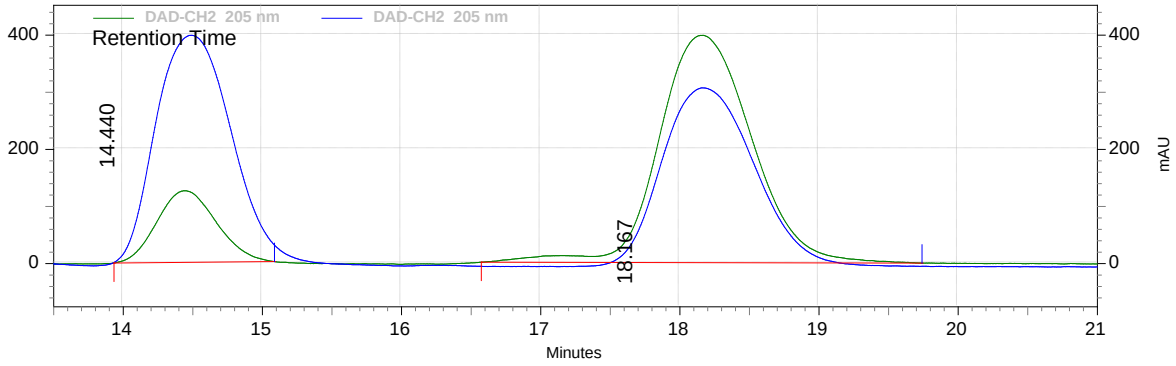


DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
8.427	25936124	89.85	1657243	90.55
9.567	2928957	10.15	172898	9.45
Totals	28865081	100.00	1830141	100.00

2e
Area % Report

Data File: 1e-SM-C:\Documents and Settings\Yu lab hplc\Desktop\lingchu-kinetic resolution\CL07058-sm-ee-5%-0.5mL-25min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 10/9/2013 2:10:40 PM
Printed: 1/9/2014 9:52:36 PM

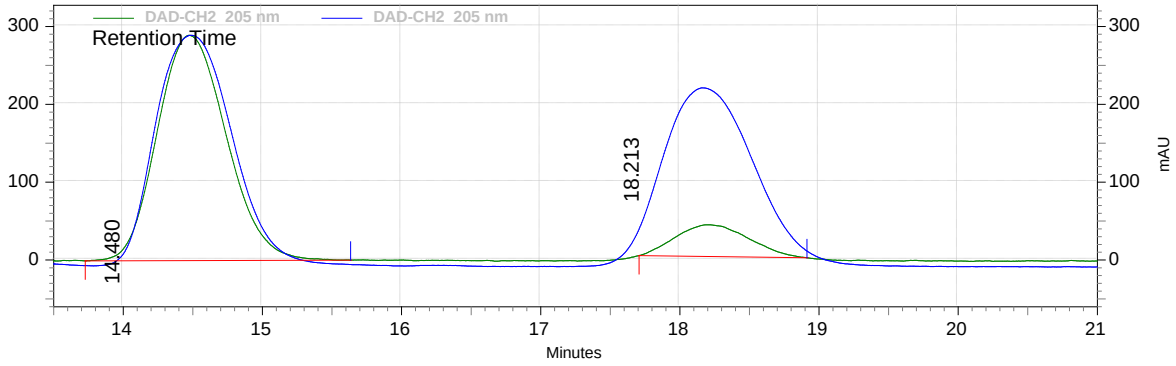


DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
14.440	14849463	16.78	501233	23.95
18.167	73659831	83.22	1591743	76.05
Totals	88509294	100.00	2092976	100.00

3e
Area % Report

Data File: 1e-PR-C:\Documents and Settings\Yu lab hplc\Desktop\lingchu-kinetic resolution\CL07058-pr-re-ee-5%-0.5mL-25min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 10/9/2013 3:03:36 PM
Printed: 1/9/2014 9:54:44 PM

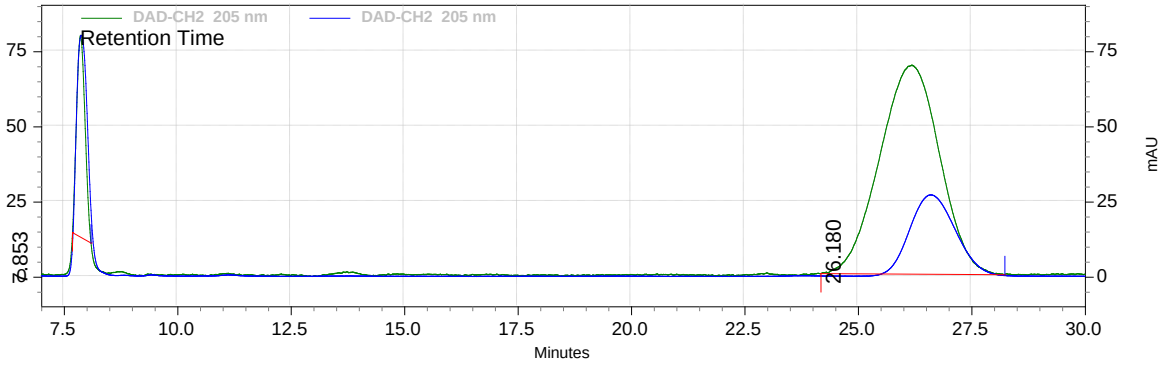


DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
14.480	39925253	87.03	1157407	87.66
18.213	5948378	12.97	162980	12.34
Totals	45873631	100.00	1320387	100.00

2f
Area % Report

Data File: 1d-SM-C:\Documents and Settings\Yu lab hplc\Desktop\lingchu-kinetic resolution\CL06105-SM-ee-5%-0.8mL-30min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 8/23/2013 12:30:53 AM
Printed: 1/9/2014 9:43:35 PM

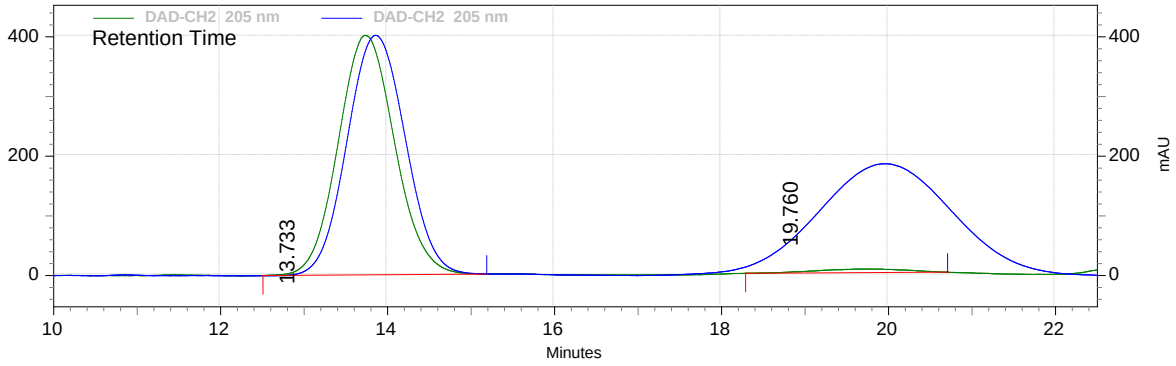


DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
7.853	3447949	11.88	268289	49.09
26.180	25587027	88.12	278199	50.91
Totals	29034976	100.00	546488	100.00

3f
Area % Report

Data File: 1d-PR-C:\Documents and Settings\Yu lab hplc\Desktop\lingchu-kinetic resolution\CL06105-pr-ee-5%-0.5mL-40min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 9/5/2013 11:33:46 PM
Printed: 1/9/2014 9:49:32 PM

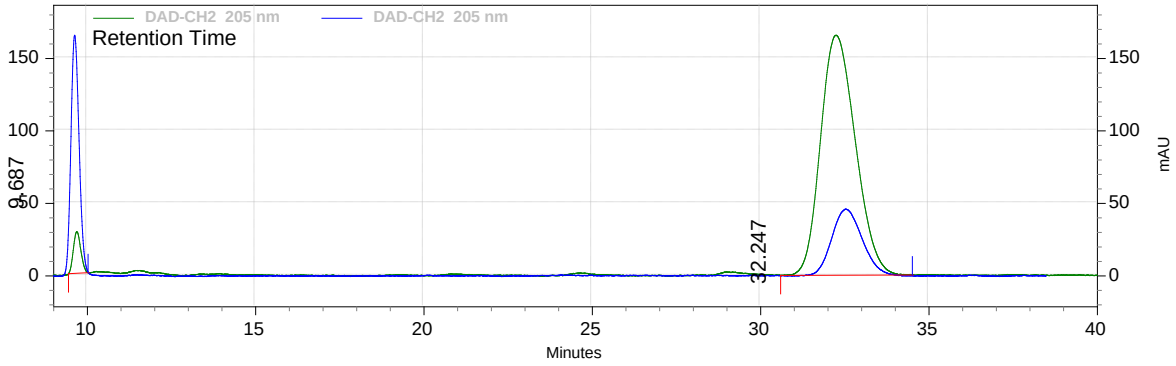


DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
13.733	75586167	97.67	1605480	98.52
19.760	1803880	2.33	24080	1.48
Totals	77390047	100.00	1629560	100.00

2g
Area % Report

Data File: 3a-SM-C:\Documents and Settings\Yu lab hplc\Desktop\lingchu-kinetic resolution\CL06134-SM-ee-5%-0.8mL-40min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 8/22/2013 10:14:50 PM
Printed: 1/10/2014 10:24:15 AM



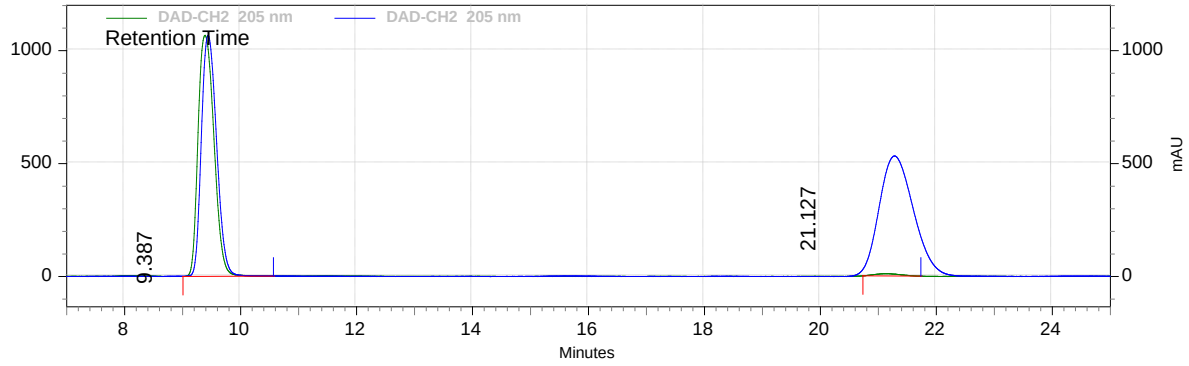
DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
9.687	1759980	3.56	114474	14.75
32.247	47725381	96.44	661858	85.25

Totals	49485361	100.00	776332	100.00

3g
Area % Report

Data File: 3a-PR-C:\Documents and Settings\Yu lab hplc\Desktop\lingchu-kinetic resolution\CL06134-PR-ee-5%-0.8mL-40min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 8/22/2013 11:27:53 PM
Printed: 1/10/2014 10:26:28 AM

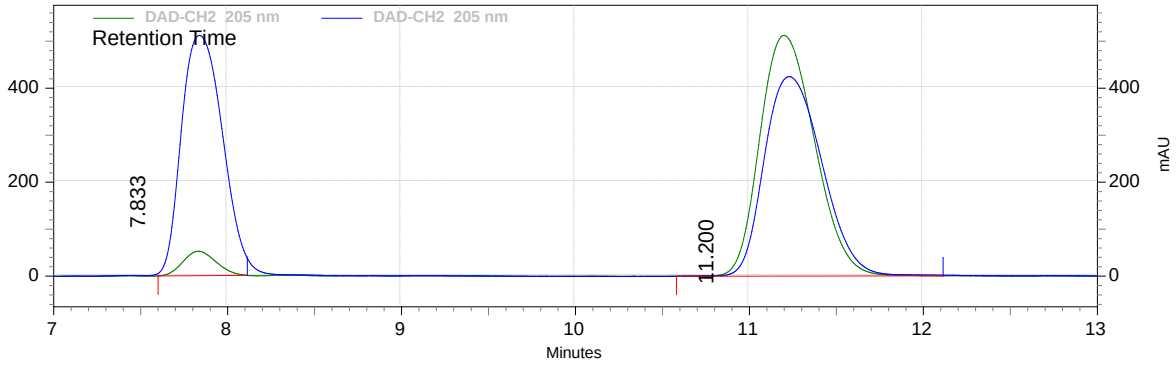


DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
9.387	81172696	98.49	4268645	99.06
21.127	1248480	1.51	40304	0.94
Totals	82421176	100.00	4308949	100.00

2h
Area % Report

Data File: 3b-SM-C:\Documents and Settings\Yu lab hplc\Desktop\lingchu-kinetic resolution\CL06123-SM-ee-5%-0.8mL-13min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 8/23/2013 1:59:28 AM
Printed: 1/10/2014 10:28:16 AM



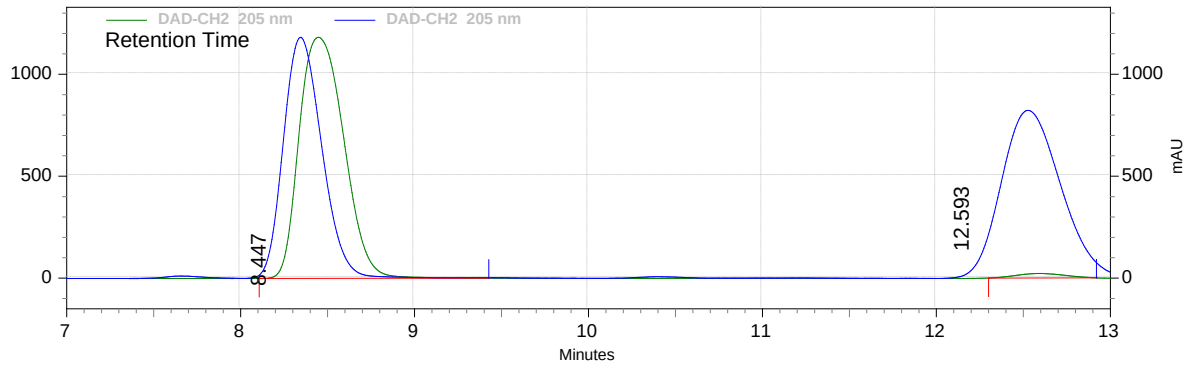
DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
7.833	2750965	5.78	208061	9.22
11.200	44857038	94.22	2049113	90.78

Totals	47608003	100.00	2257174	100.00

3h
Area % Report

Data File: 3b-PR-C:\Documents and Settings\Yu lab hplc\Desktop\lingchu-kinetic resolution\CL06123-PR-ee-5%-0.8mL-13min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 8/23/2013 2:28:09 AM
Printed: 1/10/2014 10:30:04 AM

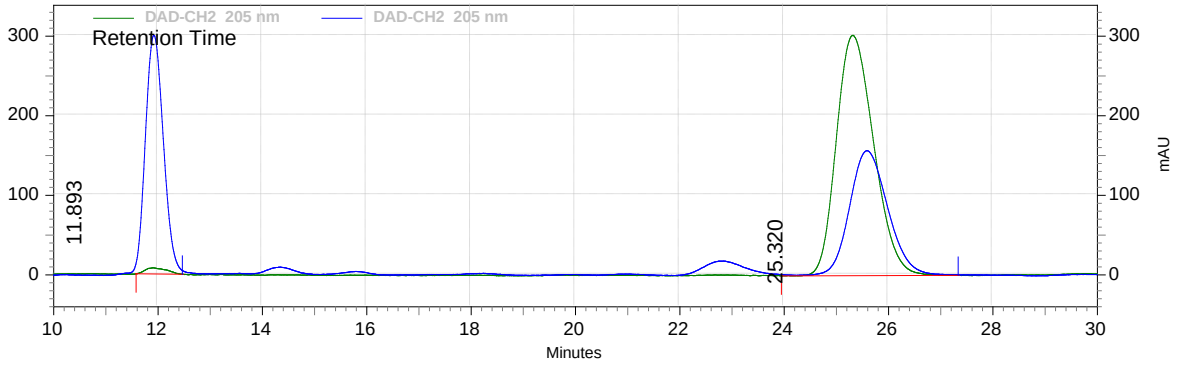


DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
8.447	84402874	98.06	4729336	98.18
12.593	1673606	1.94	87486	1.82
Totals	86076480	100.00	4816822	100.00

2i
Area % Report

Data File: 3c-SM-C:\Documents and Settings\Yu lab hplc\Desktop\lingchu-kinetic resolution\CL07029-sm-ee-5%-0.5mL-40min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 9/6/2013 12:56:41 AM
Printed: 1/10/2014 10:32:03 AM

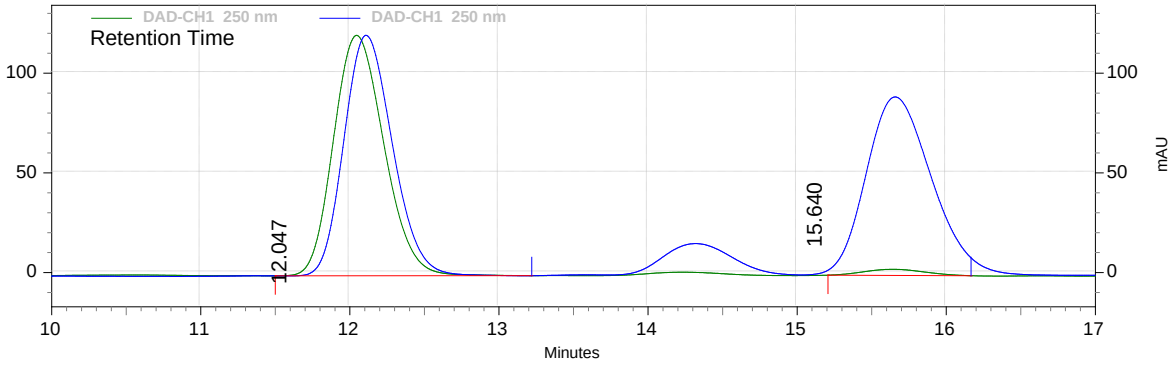


DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
11.893	892586	1.46	30779	2.48
25.320	60213612	98.54	1208100	97.52
Totals	61106198	100.00	1238879	100.00

3i
Area % Report

Data File: 3c-PR-C:\Documents and Settings\Yu lab hplc\Desktop\lingchu-kinetic resolution\CL07029-pr-ee-5%-0.5mL-25min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 9/6/2013 2:04:30 AM
Printed: 1/10/2014 10:35:13 AM

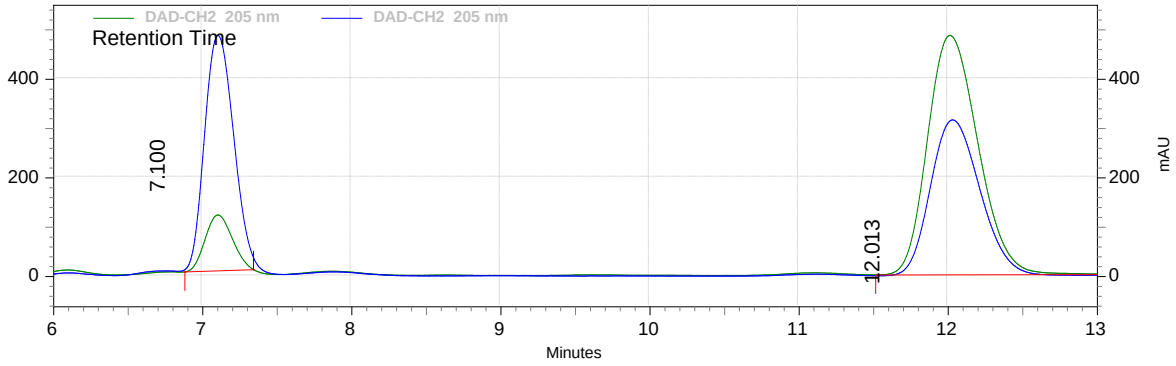


DAD-CH1 250 nm Results

Retention Time	Area	Area %	Height	Height %
12.047	11338520	97.04	482224	97.52
15.640	346014	2.96	12270	2.48
Totals	11684534	100.00	494494	100.00

2j
Area % Report

Data File: 3d-SM-C:\Documents and Settings\Yu lab hplc\Desktop\lingchu-kinetic resolution\CL06120-SM-ee-5%-0.8mL-20min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 8/22/2013 8:38:28 PM
Printed: 1/10/2014 10:37:29 AM

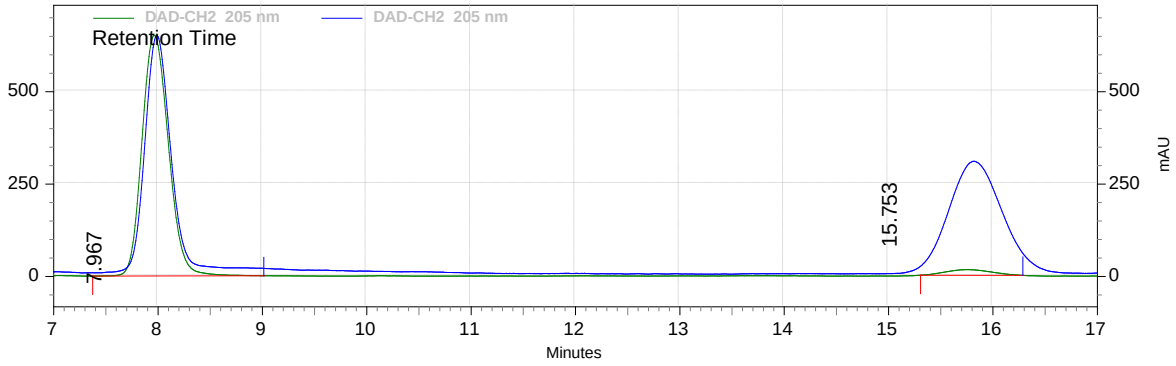


DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
7.100	5722341	11.12	452938	18.91
12.013	45735849	88.88	1941802	81.09
Totals	51458190	100.00	2394740	100.00

3j
Area % Report

Data File: 3d-PR-C:\Documents and Settings\Yu lab hplc\Desktop\lingchu-kinetic resolution\CL06120-PR-ee-5%-0.8mL-20min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 8/22/2013 9:15:44 PM
Printed: 1/10/2014 10:40:00 AM

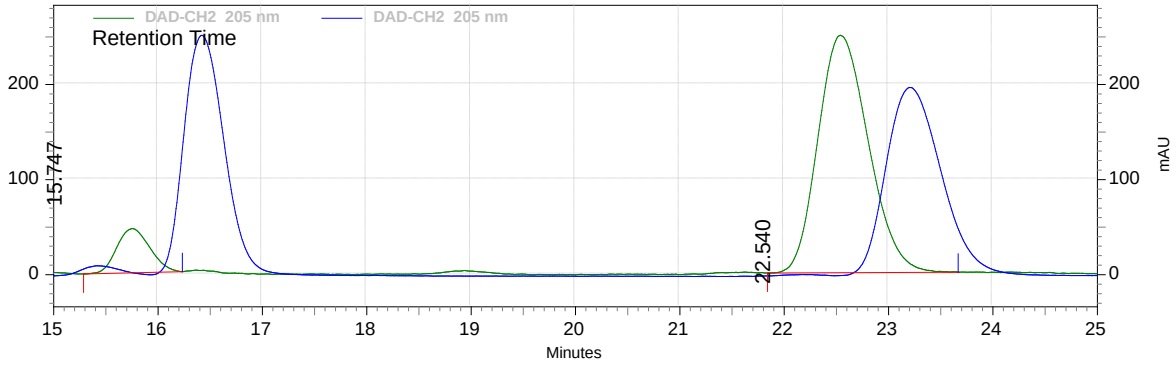


DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
7.967	45919187	96.11	2604305	97.69
15.753	1860675	3.89	61618	2.31
Totals	47779862	100.00	2665923	100.00

2k
Area % Report

Data File: 3e-SM-C:\EZChrom Elite\Enterprise\Projects\Default\Data\LingChu\CL08098-SM-ee-10%-0.3mL-40min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 12/26/2013 10:41:34 AM
Printed: 1/10/2014 10:44:33 AM



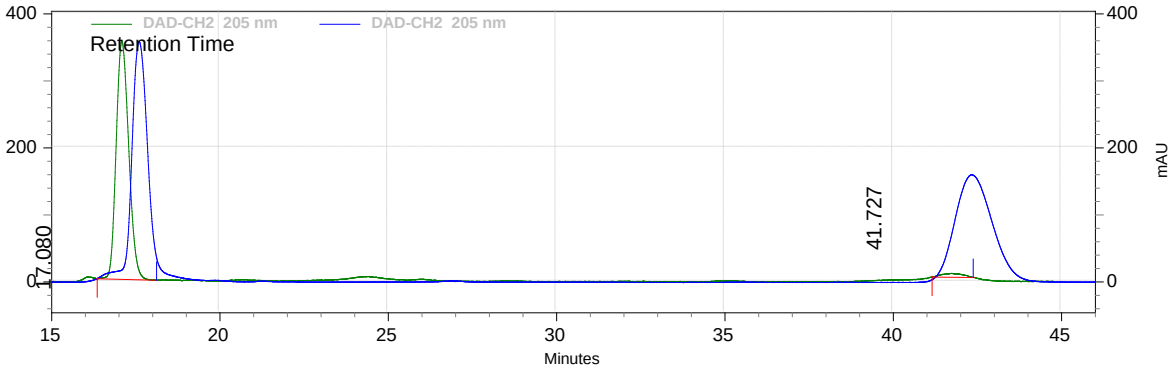
DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
15.747	4094157	10.89	185908	15.70
22.540	33507222	89.11	998394	84.30

Totals	37601379	100.00	1184302	100.00

3k
Area % Report

Data File: 3e-PR-C:\EZChrom Elite\Enterprise\Projects\Default\Data\LingChu\CL08098-PR-ee-10%-0.3mL-60min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 12/26/2013 11:09:20 AM
Printed: 1/10/2014 10:47:38 AM

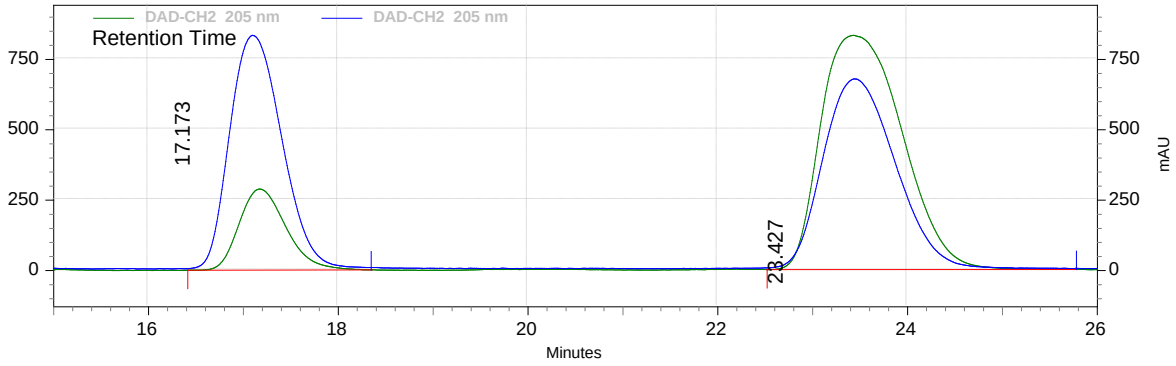


DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
17.080	36972202	97.33	1420820	98.38
41.727	1015904	2.67	23411	1.62
Totals	37988106	100.00	1444231	100.00

2l
Area % Report

Data File: 3f-SM-C:\Documents and Settings\Yu lab hplc\Desktop\lingchu-kinetic resolution\CL07084-sm-ee-5%-0.5mL-30min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 10/9/2013 10:49:05 AM
Printed: 1/10/2014 10:50:34 AM

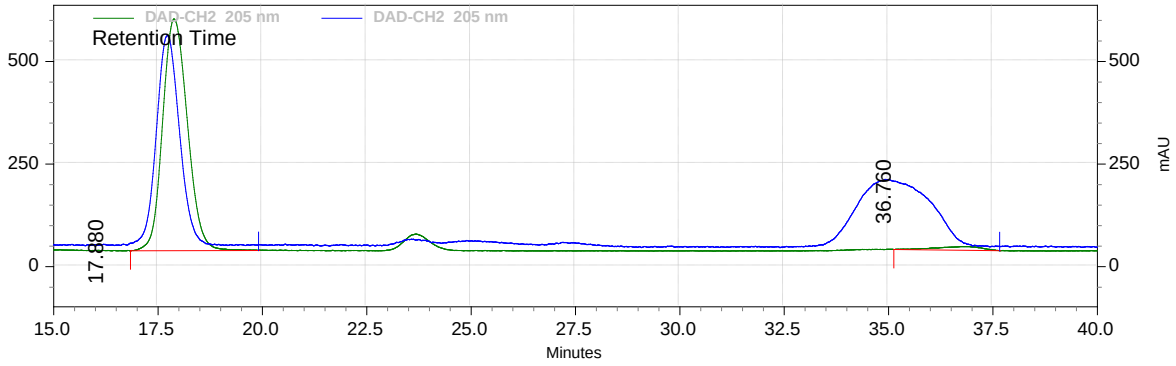


DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
17.173	39431375	16.58	1150810	25.70
23.427	198359465	83.42	3326859	74.30
Totals	237790840	100.00	4477669	100.00

3l
Area % Report

Data File: 3f-PR-C:\Documents and Settings\Yu lab hplc\Desktop\lingchu-kinetic resolution\CL07084-pr-ee-5%-0.5mL-45min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 10/9/2013 12:06:58 PM
Printed: 1/10/2014 10:52:17 AM

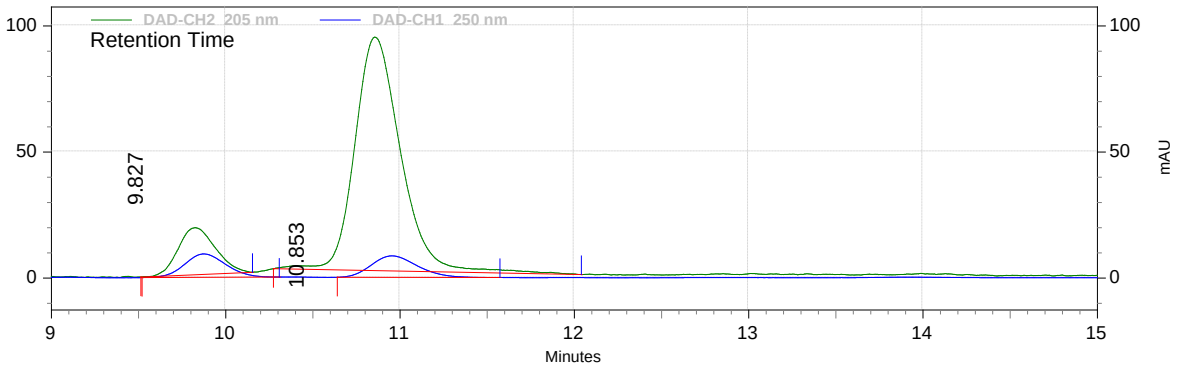


DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
17.880	93406456	97.10	2258366	98.39
36.760	2792665	2.90	36964	1.61
Totals	96199121	100.00	2295330	100.00

2m
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Default\Data\LingChu\CL10022-1-sm-re-ee-10%-0.5mL-25min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 25 min without frc 0.5 ml per min.met
Acquired: 7/3/2014 2:26:48 PM
Printed: 7/6/2014 3:10:04 PM

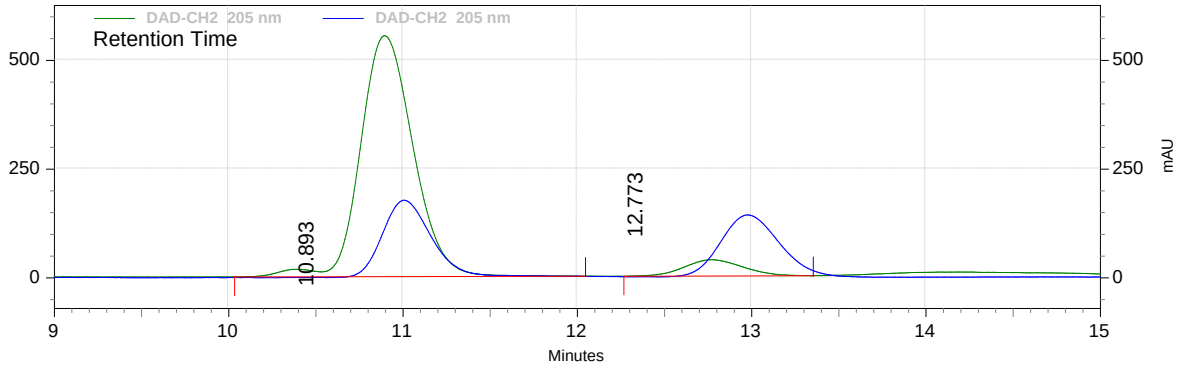


DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
9.827	1111503	14.05	74561	16.76
10.853	6799405	85.95	370221	83.24
Totals	7910908	100.00	444782	100.00

3m-mono
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Default\Data\LingChu\CL10022-1-PR-ee-10%-0.5mL-20min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 20 min without fc 0.5 ml per min.met
Acquired: 7/2/2014 11:11:43 PM
Printed: 7/6/2014 3:15:48 PM

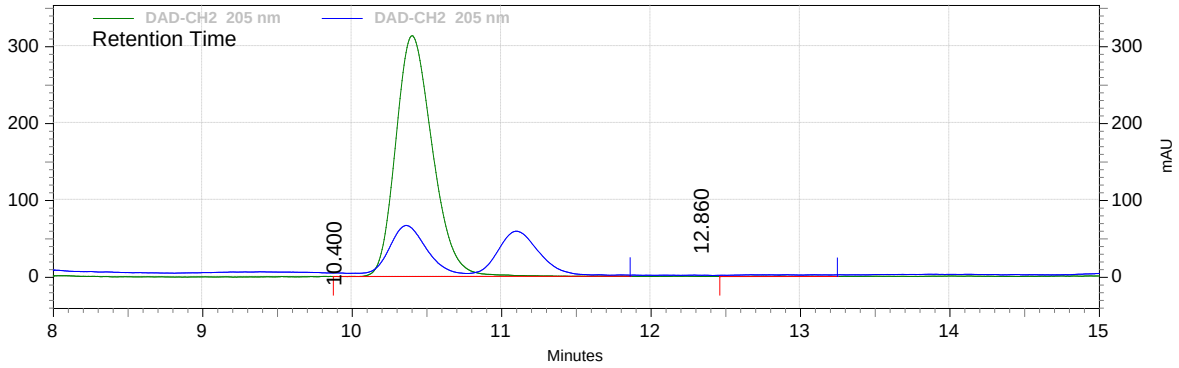


DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
10.893	46737471	93.06	2215271	93.66
12.773	3487817	6.94	149915	6.34
Totals	50225288	100.00	2365186	100.00

3m-di
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Default\Data\LingChu\CL10022-1-di-ee-10%-0.5mL-25min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 25 min without frc 0.5 ml per min.met
Acquired: 7/3/2014 2:00:26 PM
Printed: 7/6/2014 3:19:30 PM



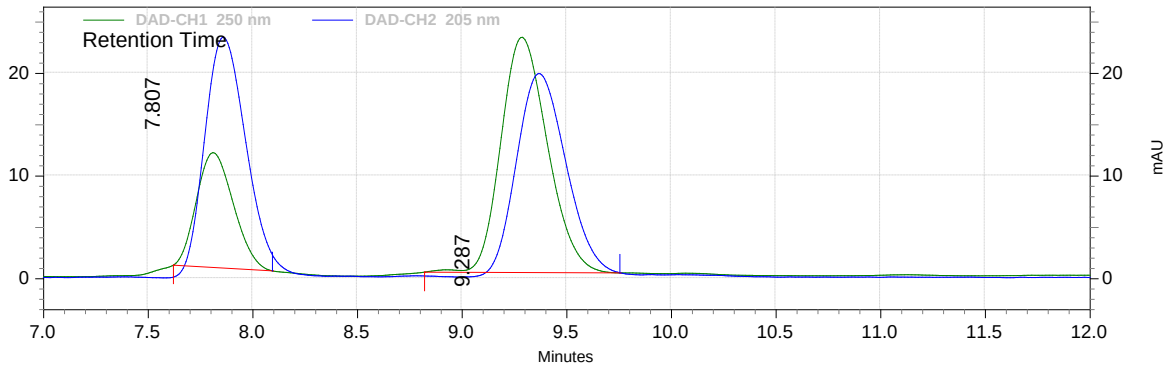
DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
10.400	21413422	99.73	1252953	99.79
12.860	57268	0.27	2630	0.21

Totals	21470690	100.00	1255583	100.00

2n
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Default\Data\LingChu\CL10075-1-sm-ee-10%-0.5mL-25min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 25 min without frc 0.5 ml per min.met
Acquired: 8/19/2014 11:15:48 AM
Printed: 8/24/2014 5:40:09 PM



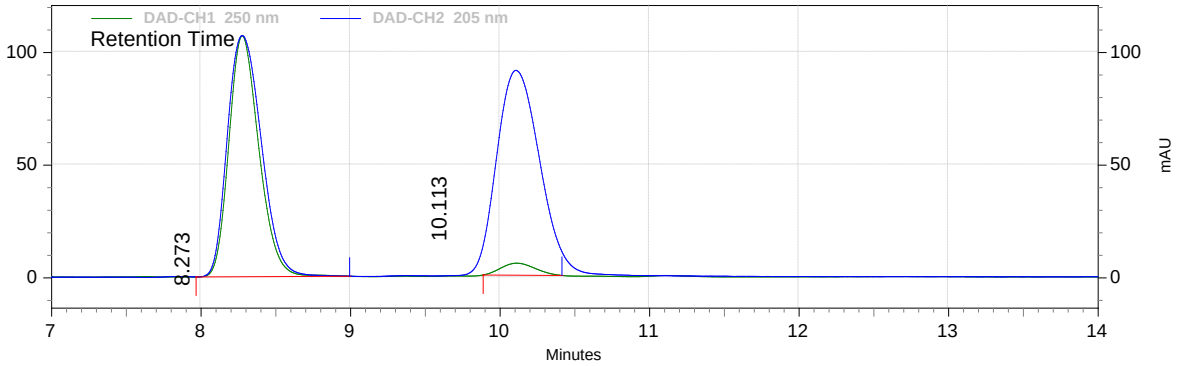
DAD-CH1 250 nm Results

Retention Time	Area	Area %	Height	Height %
7.807	548903	27.89	44676	32.78
9.287	1419192	72.11	91613	67.22
Totals	1968095	100.00	136289	100.00

3n

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Default\Data\LingChu\CL10075-1-re-pr-ee-10%-0.5mL-25min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 25 min without frc 0.5 ml per min.met
Acquired: 8/19/2014 11:42:10 AM
Printed: 8/24/2014 5:42:48 PM

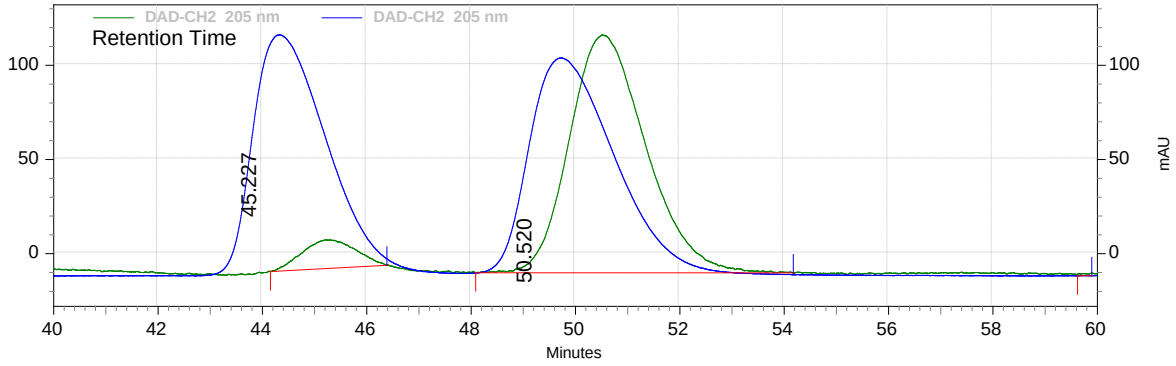


DAD-CH1 250 nm Results

Retention Time	Area	Area %	Height	Height %
8.273	5953359	94.65	427788	95.26
10.113	336756	5.35	21301	4.74
Totals	6290115	100.00	449089	100.00

5a
Area % Report

Data File: 5a-SM-C:\Documents and Settings\Yu lab hplc\Desktop\lingchu-kinetic resolution\CL08040-SM-ee-10%-0.3mL-60min-OJ
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 12/27/2013 12:39:27 PM
Printed: 1/10/2014 11:03:52 AM

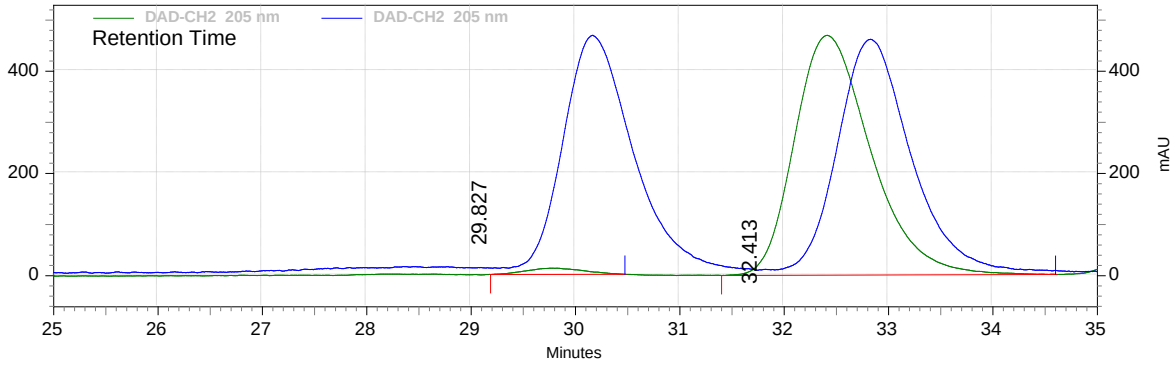


DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
45.227	4338698	7.77	61188	10.81
50.520	51512114	92.23	505050	89.19
Totals	55850812	100.00	566238	100.00

6a
Area % Report

Data File: 5a-PR-C:\EZChrom Elite\Enterprise\Projects\Default\Data\LingChu\CL08040-PR-ee-10%-0.2mL-45min-ADH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 1/11/2014 11:54:45 AM
Printed: 1/14/2014 10:05:51 AM

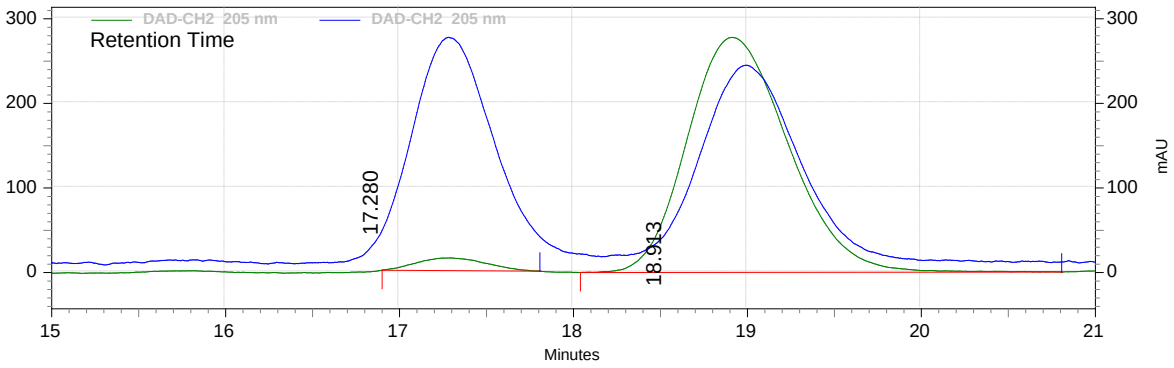


DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
29.827	1888425	1.95	49058	2.55
32.413	95174677	98.05	1876398	97.45
Totals	97063102	100.00	1925456	100.00

5b
Area % Report

Data File: 5b-SM-C:\Documents and Settings\Yu lab hplc\Desktop\lingchu-kinetic resolution\CL07116-sm-ee-10%-0.5mL-30min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 10/17/2013 9:22:01 PM
Printed: 1/10/2014 11:07:20 AM



DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
17.280	1670982	3.59	59640	5.09
18.913	44850513	96.41	1111276	94.91

Totals	46521495	100.00	1170916	100.00

6b
Area % Report

Data File: 5b-PR-C:\Documents and Settings\Yu lab hplc\Desktop\lingchu-kinetic resolution\CL07116-pr-ee-10%-0.3mL-40min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 10/18/2013 5:00:43 PM
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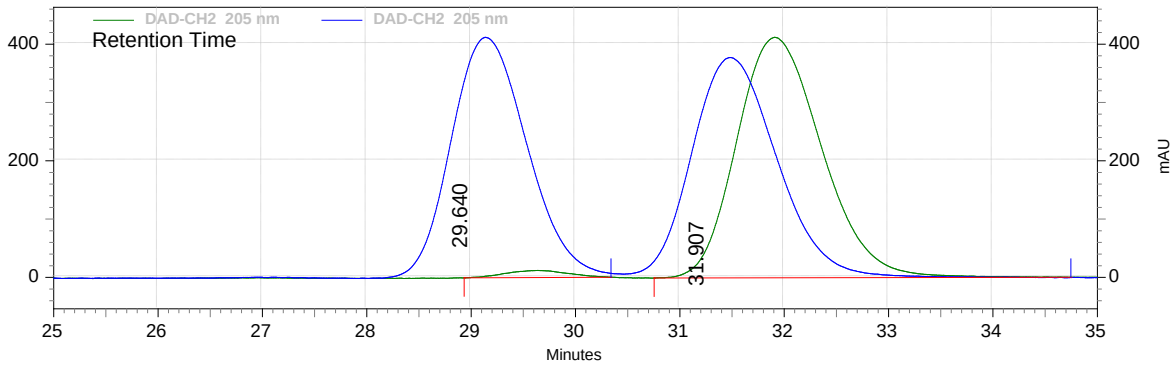
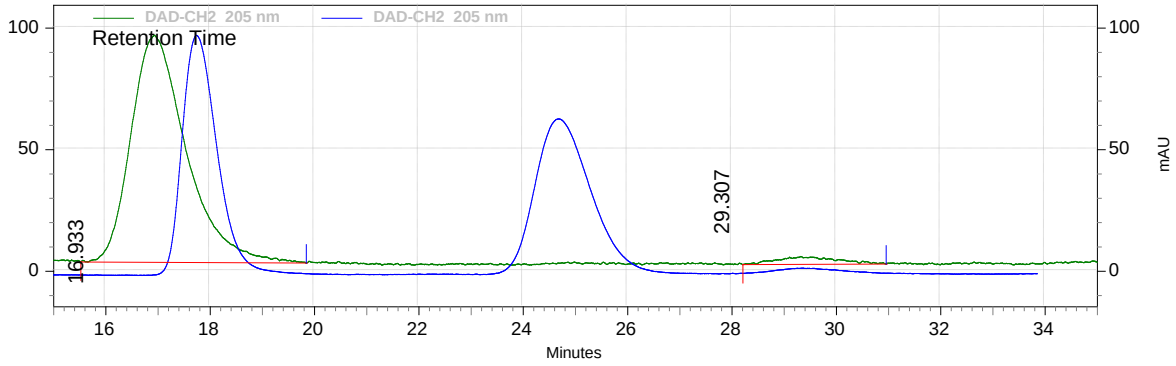


DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
29.640	2037367	2.13	48992	2.88
31.907	93423783	97.87	1649466	97.12
Totals	95461150	100.00	1698458	100.00

5c
Area % Report

Data File: 5c-SM-C:\EZChrom Elite\Enterprise\Projects\Default\Data\LingChu\CL08096-SM-ee-10%-0.5mL-60min-OJ
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 1/5/2014 2:21:19 PM
Printed: 1/10/2014 11:17:15 AM



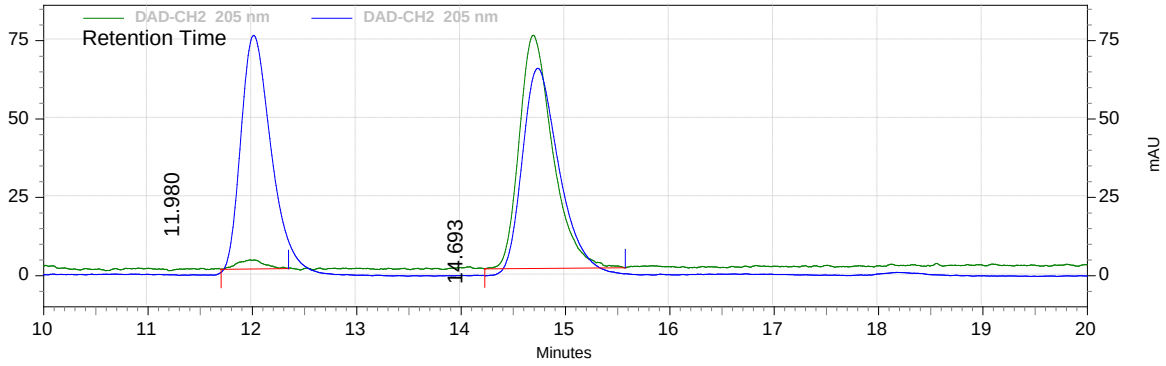
DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
16.933	27482431	96.36	373223	96.65
29.307	1037923	3.64	12939	3.35

Totals	28520354	100.00	386162	100.00

6c
Area % Report

Data File: 5c-PR-C:\EZChrom Elite\Enterprise\Projects\Default\Data\LingChu\CL08096-PR-ee-5%-0.5mL-40min-ADH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 1/6/2014 9:35:51 PM
Printed: 1/10/2014 11:25:25 AM

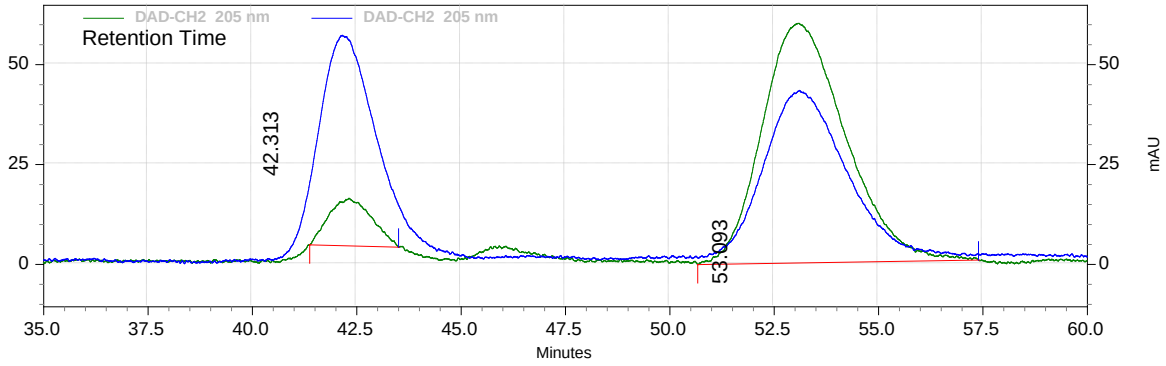


DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
11.980	211866	3.09	11678	3.78
14.693	6650494	96.91	297508	96.22
Totals	6862360	100.00	309186	100.00

5d
Area % Report

Data File: 5d-SM-C:\Documents and Settings\Yu lab hplc\Desktop\lingchu-kinetic resolution\CL08081-SM-ee-10%-0.5mL-60min-OJ
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 12/14/2013 5:25:59 PM
Printed: 1/10/2014 2:25:40 PM

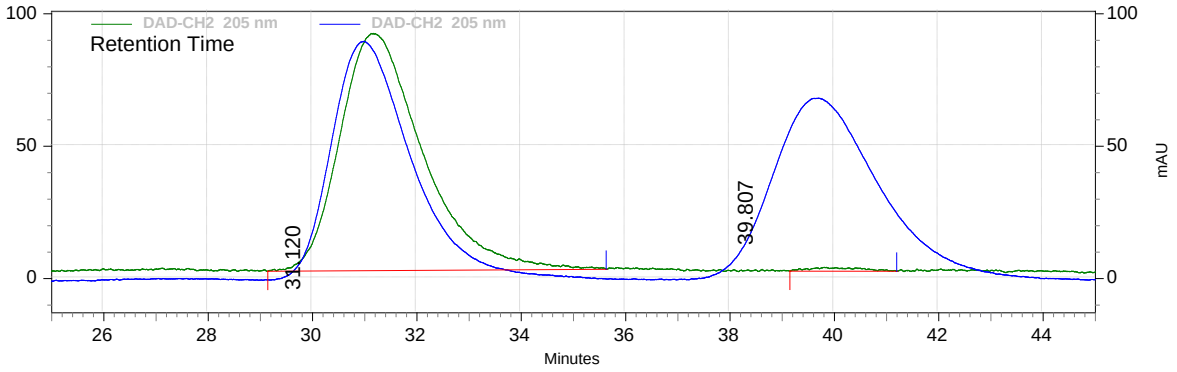


DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
42.313	3405330	9.28	47562	16.55
53.093	33304182	90.72	239824	83.45
Totals	36709512	100.00	287386	100.00

6d
Area % Report

Data File: 5d-PR-C:\Documents and Settings\Yu lab hplc\Desktop\lingchu-kinetic resolution\CL08081-PR-ee-10%-0.5mL-60min-OJ
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 12/14/2013 6:27:30 PM
Printed: 1/10/2014 2:29:20 PM



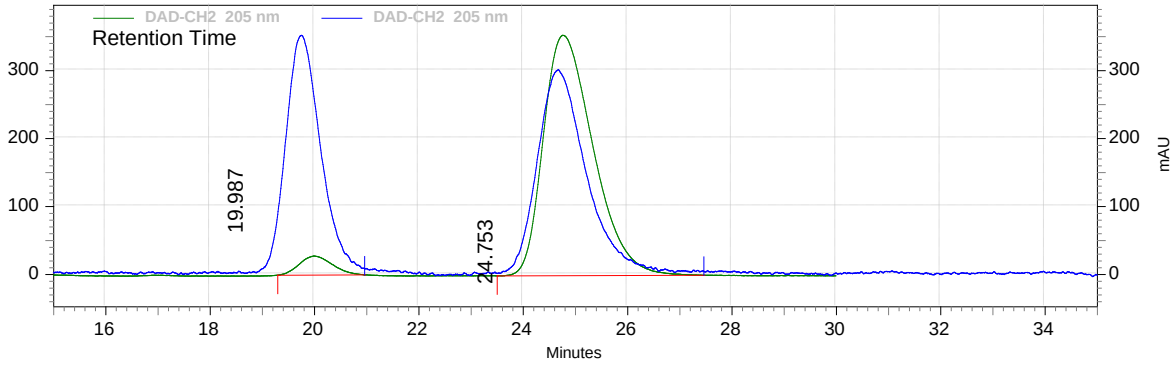
DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
31.120	39645952	99.00	358370	98.41
39.807	402004	1.00	5804	1.59

Totals	40047956	100.00	364174	100.00

5e
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Default\Data\LingChu\CL09065-2-SM-ee-10%-0.5mL-75min-OJ
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 60 min without fc 0.4 ml per min.met
Acquired: 4/2/2014 1:07:23 PM
Printed: 4/4/2014 9:18:36 PM



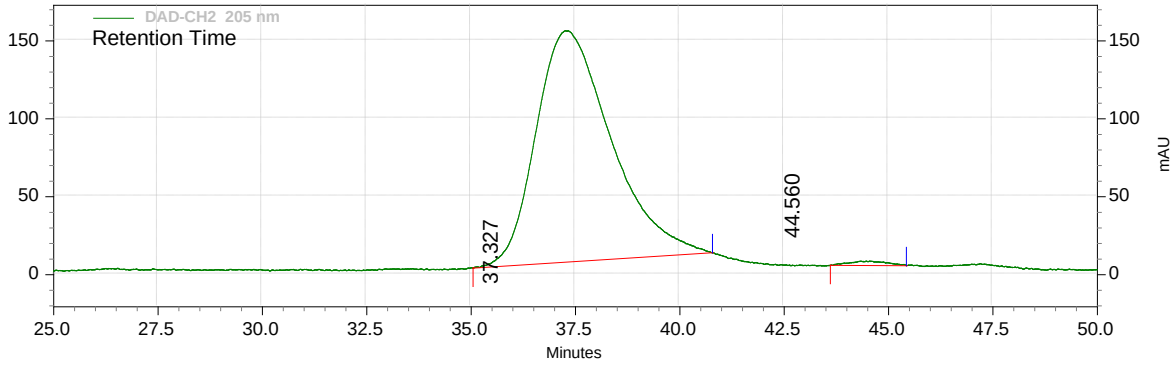
DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
19.987	4926216	4.99	111914	7.33
24.753	93889663	95.01	1414079	92.67
Totals	98815879	100.00	1525993	100.00

6e

Area % Report

Data File: 5f-PR-C:\EZChrom Elite\Enterprise\Projects\Default\Data\LingChu\CL08058-PR-ee-10%-0.3mL-75min-OJ
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 12/13/2013 10:18:54 PM
Printed: 1/10/2014 2:42:08 PM

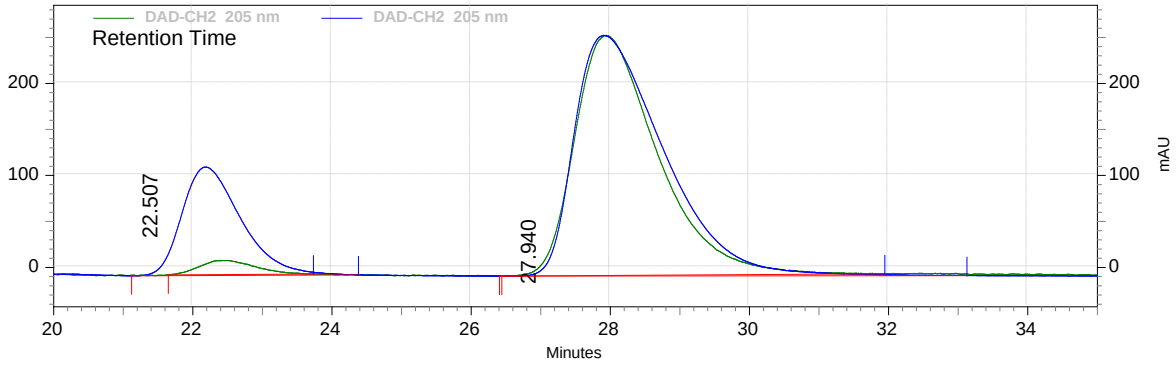


DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
37.327	74377760	99.06	593490	98.15
44.560	707487	0.94	11171	1.85
Totals	75085247	100.00	604661	100.00

5f
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Default\Data\LingChu\CL08127-SM-ee-10%-0.5mL-40min-OJ
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 60 min without fc 0.4 ml per min.met
Acquired: 2/20/2014 10:03:16 AM
Printed: 4/4/2014 9:16:47 PM



DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
22.507	3389678	3.77	61198	5.54
27.940	86516469	96.23	1044197	94.46
Totals	89906147	100.00	1105395	100.00

6f
Area % Report

Data File: 5e-PR-C:\Documents and Settings\Yu lab hplc\Desktop\lingchu-kinetic resolution\CL08027-pr-ee-10%-0.5mL-40min-OJ
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 11/15/2013 11:23:38 AM
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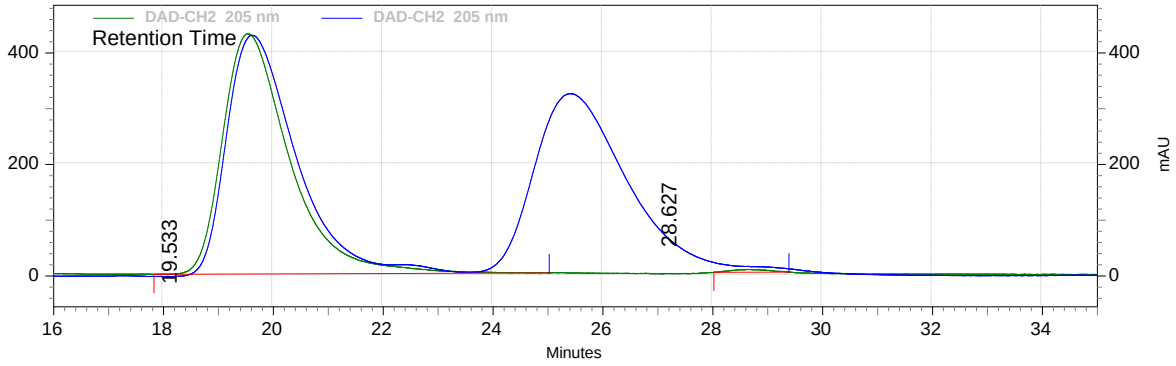
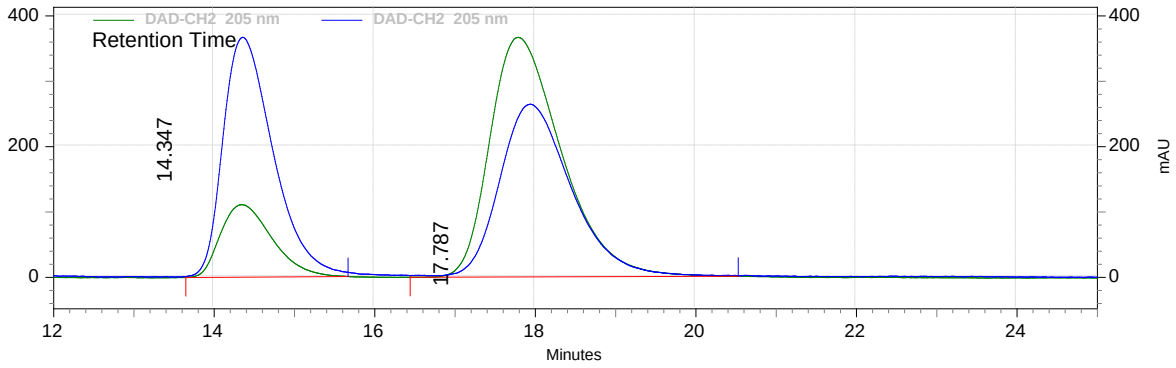


DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
19.533	145655911	99.37	1723908	98.87
28.627	923417	0.63	19697	1.13
Totals	146579328	100.00	1743605	100.00

5g
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Default\Data\LingChu\CL09066-2-SM-ee-10%-0.5mL-75min-OJ
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 60 min without fc 0.4 ml per min.met
Acquired: 4/2/2014 1:38:43 PM
Printed: 4/4/2014 9:22:34 PM

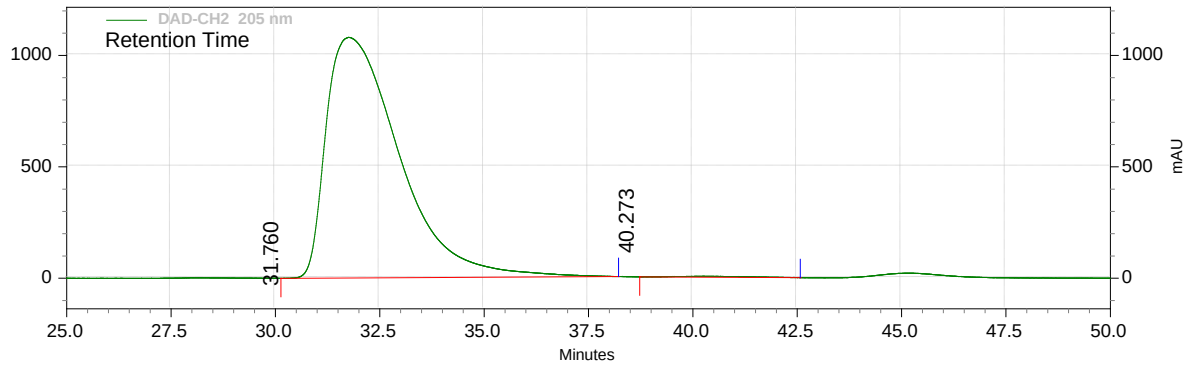


DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
14.347	19794564	17.88	443985	23.24
17.787	90889220	82.12	1466167	76.76
Totals	110683784	100.00	1910152	100.00

6g
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\Default\Data\LingChu\CL09066-1-PR-ee-10%-0.2mL-75min-OJ
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 60 min without fc 0.4 ml per min.met
Acquired: 4/2/2014 3:06:55 PM
Printed: 4/4/2014 9:20:20 PM



DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
31.760	525971055	99.66	4319443	99.64
40.273	1776529	0.34	15579	0.36

Totals	527747584	100.00	4335022	100.00

8a
Area % Report

Data File: 5g-SM-C:\Documents and Settings\Yu lab hplc\Desktop\lingchu-kinetic resolution\CL08032-sm-ee-5%-0.1mL-60min-ADH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 11/16/2013 6:20:03 PM
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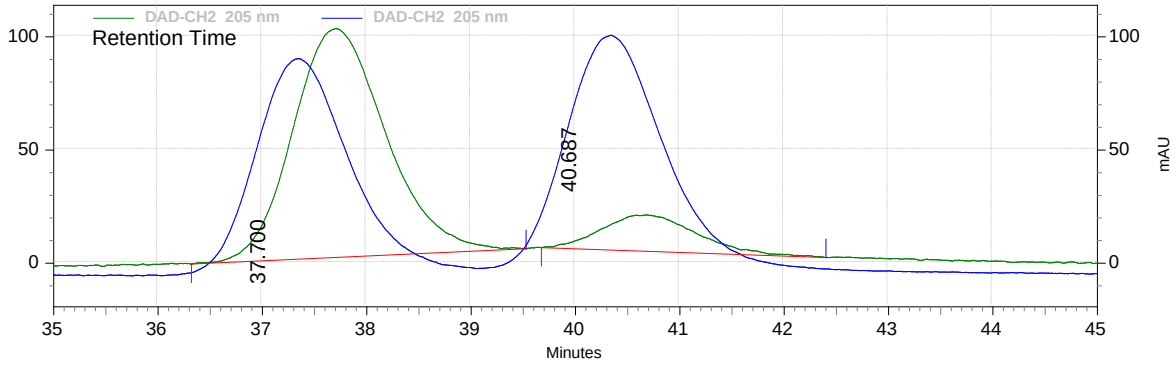
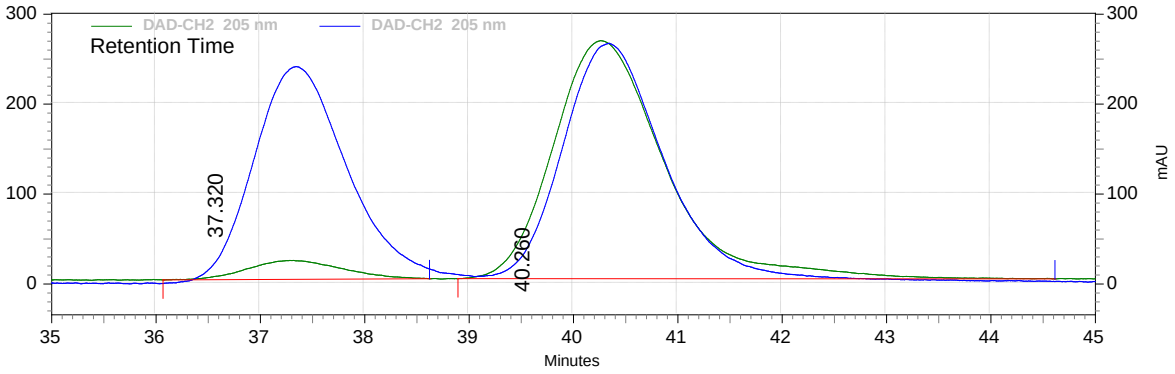


DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
37.700	25074572	85.99	404499	86.26
40.687	4086691	14.01	64419	13.74
Totals	29161263	100.00	468918	100.00

9a-mono
Area % Report

Data File: 5g-mono-C:\Documents and Settings\Yu lab hplc\Desktop\lingchu-kinetic resolution\CL08032-mono-ee-5%-0.1mL-60min-ADH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 11/16/2013 3:15:12 PM
Printed: 1/10/2014 2:49:37 PM



DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
37.320	5302078	6.34	83864	7.34
40.260	78360330	93.66	1058741	92.66
Totals	83662408	100.00	1142605	100.00

9a-di
Area % Report

Data File: 5g-di-C:\Documents and Settings\Yu lab hplc\Desktop\lingchu-kinetic resolution\CL08032-di-ee-5%-0.1mL-60min-ADH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 11/16/2013 5:18:26 PM
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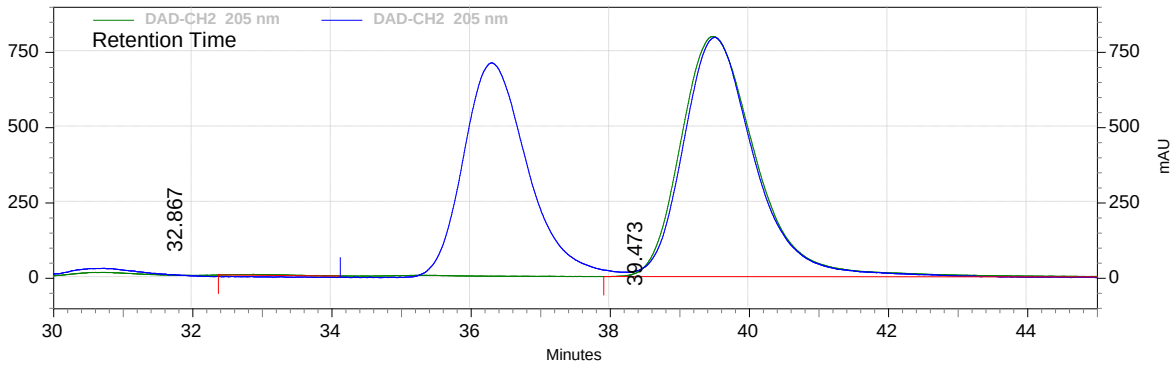


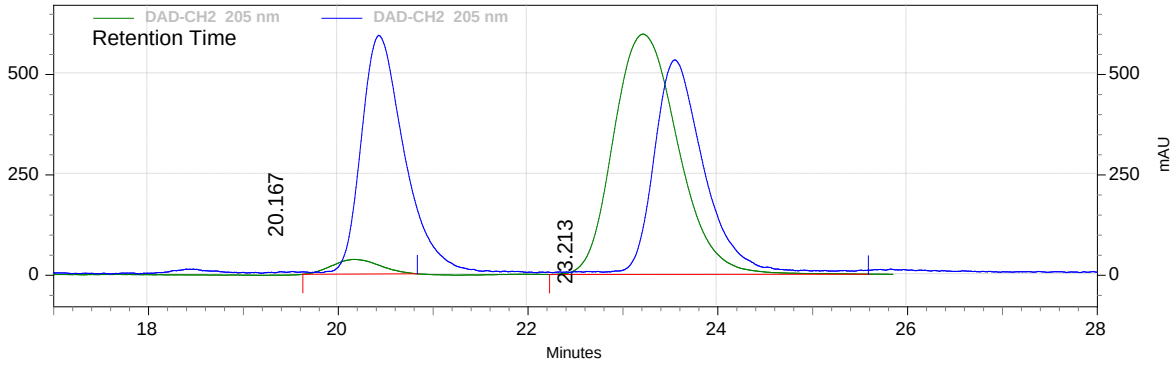
DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
32.867	608676	0.25	11685	0.36
39.473	238492352	99.75	3190222	99.64

Totals	239101028	100.00	3201907	100.00

8b
Area % Report

Data File: 5h-SM-C:\Documents and Settings\Yu lab hplc\Desktop\lingchu-kinetic resolution\CL07119-3-sm-ee-10%-0.3mL-40min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 10/18/2013 2:50:24 PM
Printed: 1/10/2014 2:54:38 PM



DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
20.167	5106688	4.33	146380	5.76
23.213	112728212	95.67	2393278	94.24
Totals	117834900	100.00	2539658	100.00

9b
Area % Report

Data File: 5h-PR-C:\Documents and Settings\Yu lab hplc\Desktop\lingchu-kinetic resolution\CL07119-pr-ee-10%-0.3mL-40min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 11/18/2013 11:40:42 PM
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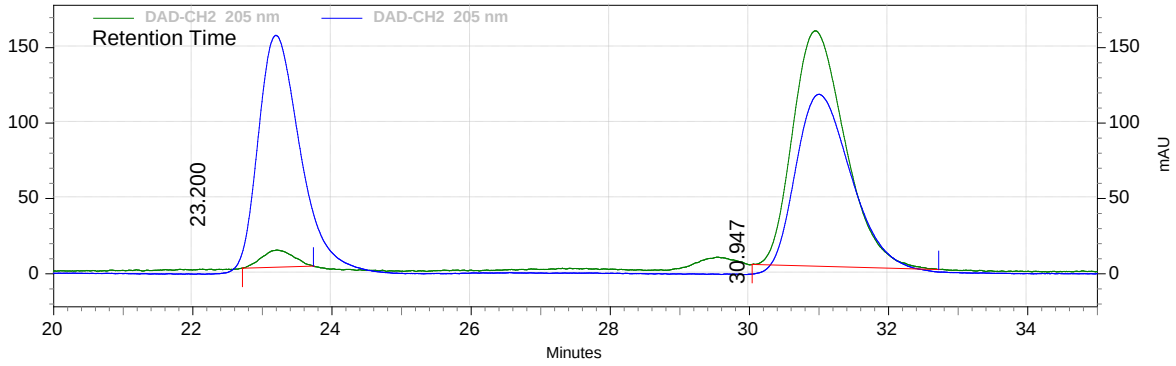
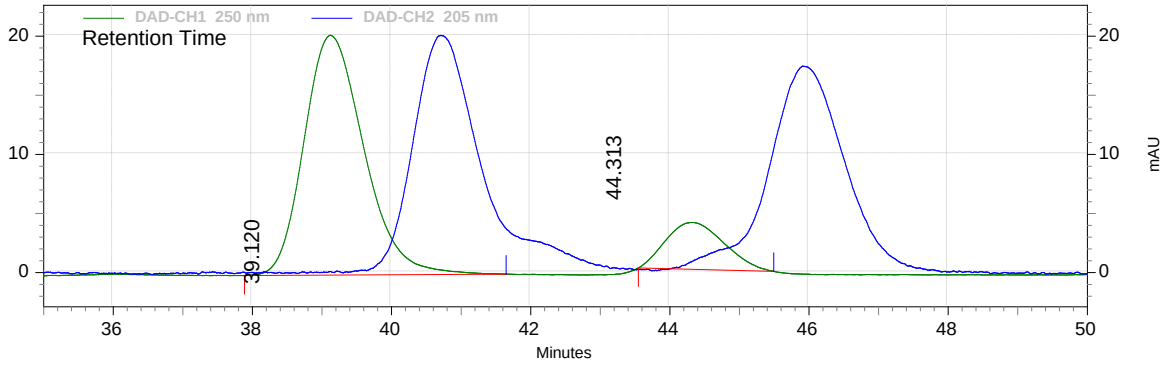


DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
23.200	1435500	4.21	45548	6.80
30.947	32677521	95.79	624377	93.20
Totals	34113021	100.00	669925	100.00

8c
Area % Report

Data File: 5i-SM-C:\EZChrom Elite\Enterprise\Projects\Default\Data\LingChu\CL08079-SM-ee-re-10%-0.2mL-45min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
Acquired: 1/12/2014 10:28:40 PM
Printed: 1/14/2014 10:09:32 AM



DAD-CH1 250 nm Results

Retention Time	Area	Area %	Height	Height %
39.120	4841826	83.98	81094	83.65
44.313	923718	16.02	15851	16.35

Totals	5765544	100.00	96945	100.00

9c
Area % Report

Data File: 5i-PR-C:\EZChrom Elite\Enterprise\Projects\Default\Data\LingChu\CL08079-PR-ee-5%-0.3mL-60min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
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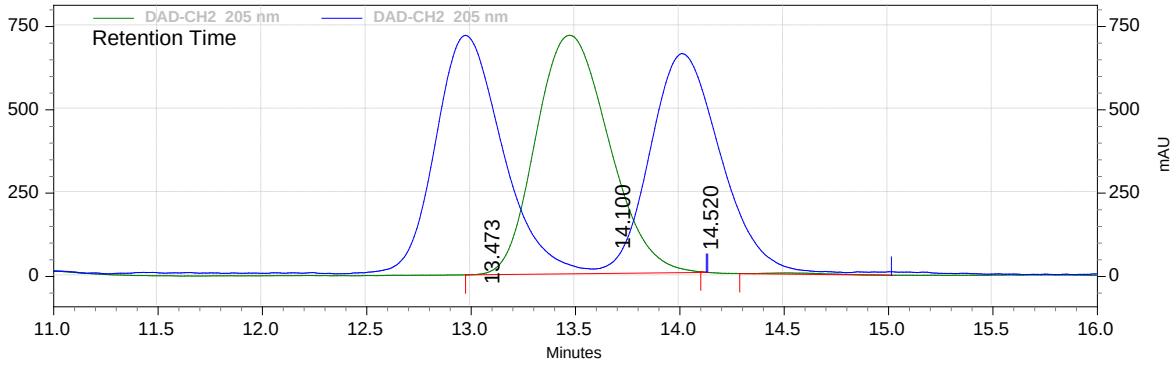

DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
13.473	69496817	99.62	2861660	99.54
14.520	266465	0.38	13167	0.46
Totals	69763476	100.00	2874827	100.00

8d
Area % Report

Data File: 5j-SM-C:\EZChrom Elite\Enterprise\Projects\Default\Data\LingChu\CL08077-SM-ee-5%-0.2mL-45min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
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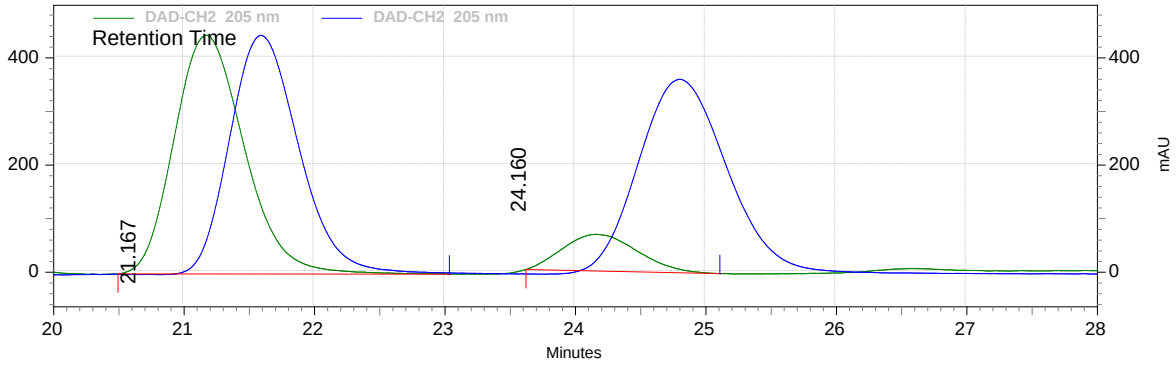

DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
21.167	66528912	85.95	1780549	86.69
24.160	10879612	14.05	273294	13.31

Totals	77408524	100.00	2053843	100.00

9d
Area % Report

Data File: 5j-PR-C:\EZChrom Elite\Enterprise\Projects\Default\Data\LingChu\CL08077-PR-ee-5%-0.1mL-90min-ASH
Method: C:\EZChrom Elite\Enterprise\Projects\Default\Method\A 120 min without fc 0.5 ml per min.met
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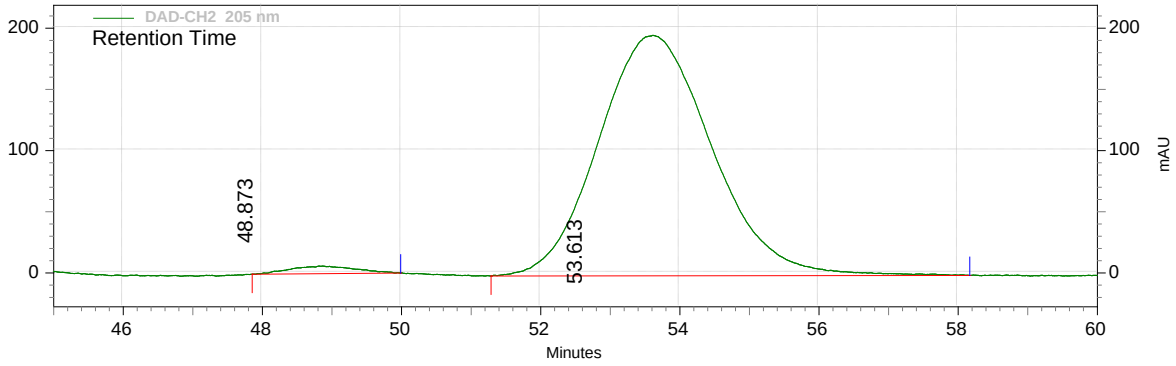
Printed: 1/14/2014 10:22:56 AM



DAD-CH2 205 nm Results

Retention Time	Area	Area %	Height	Height %
48.873	1635782	1.81	24985	3.08
53.613	88890110	98.19	785945	96.92
Totals	90525892	100.00	810930	100.00

X-ray crystallographic data for 3l

The single crystal X-ray diffraction studies were carried out on a Bruker Kappa APEX CCD diffractometer equipped with Mo K_α radiation (λ = 0.71073 Å). A 0.217 x 0.153 x 0.055 mm colorless block was mounted on a Cryoloop with Paratone oil. Data were collected in a nitrogen gas stream at 100(2) K using ϕ and ϖ scans. Crystal-to-detector distance was 40 mm and exposure time was 5 seconds per frame using a scan width of 2.0°. Data collection was 99.6% complete to 25.00° in θ. A total of 8740 reflections were collected covering the indices, -11<=h<=11, -9<=k<=9, -13<=l<=13. 2957 reflections were found to be symmetry independent, with a R_{int} of 0.0307. Indexing and unit cell refinement indicated a primitive, monoclinic lattice. The space group was found to be $P2_1$. The data were integrated using the Bruker SAINT software program and scaled using the SADABS software program. Solution by direct methods (SHELXM) produced a complete phasing model consistent with the proposed structure.

All nonhydrogen atoms were refined anisotropically by full-matrix least-squares (SHELXL-2014). All hydrogen atoms were placed using a riding model. Their positions were constrained relative to their parent atom using the appropriate HFIX command in SHELXL-2014. Crystallographic data are summarized in Table S2.

Figure S1. X-ray Crystallographic Data of **3l**

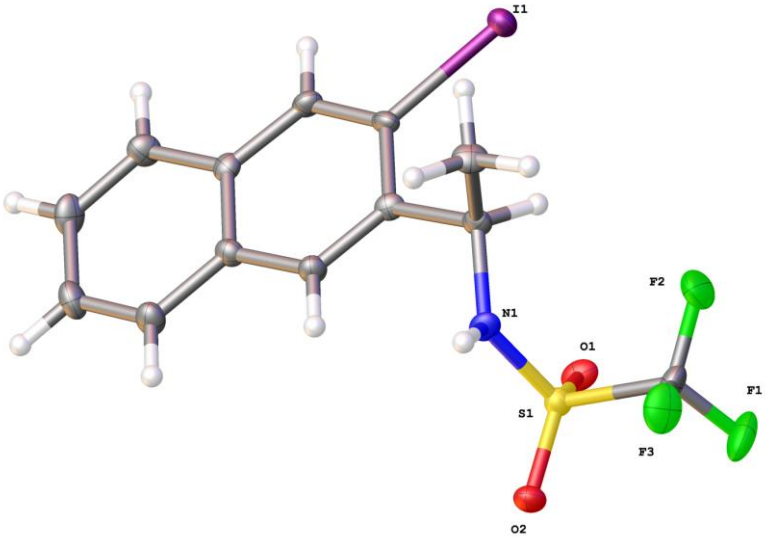


Table S2. Crystal data and structure refinement for **3l**

Report date	2014-03-27	
Identification code	Yu34	
Empirical formula	C13 H11 F3 I N O2 S	
Molecular formula	C13 H11 F3 I N O2 S	
Formula weight	429.19	
Temperature	100 K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 1 21 1	
Unit cell dimensions	a = 9.653(2) Å	α= 90°.
	b = 7.5500(16) Å	β= 106.626(6)°.
	c = 10.651(2) Å	γ = 90°.
Volume	743.7(3) $Å^3$	
Z	2	
Density (calculated)	1.916 Mg/m^3	
Absorption coefficient	2.328 mm^{-1}	
F(000)	416	
Crystal size	0.217 x 0.153 x 0.055 mm^3	
Crystal color, habit	Colorless Block	
Theta range for data collection	3.357 to 26.358°.	
Index ranges	-11<=h<=11, -9<=k<=9, -13<=l<=13	
Reflections collected	8740	
Independent reflections	2957 [R(int) = 0.0307]	
Completeness to theta = 25.000°	99.6 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.0932 and 0.0585	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2957 / 2 / 195	
Goodness-of-fit on F^2	1.043	
Final R indices [I>2sigma(I)]	R1 = 0.0235, wR2 = 0.0449	
R indices (all data)	R1 = 0.0254, wR2 = 0.0460	
Absolute structure parameter	0.007(13)	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.662 and -0.445 e.$Å^{-3}$	

References:

1. R. Giri, B.-F. Shi, K. M. Engle, N. Maugel, J.-Q. Yu, Transition metal-catalyzed C–H activation reactions: diastereoselectivity and enantioselectivity. *Chem. Soc. Rev.* **38**, 3242–3272 (2009).

2. M. P. Doyle, R. Duffy, M. Ratnikov, L. Zhou, Catalytic carbene insertion into C–H bonds. *Chem. Rev.* **110**, 704–724 (2010).

3. R. P. Reddy, H. M. L. Davies, Dirhodium tetracarboxylates derived from adamantylglycine as chiral catalyst for enantioselective C–H aminations. *Org. Lett.* **8**, 5013–5016 (2006).

4. C. Liang, F. Robert-Peillard, C. Fruit, P. Muller, R. H. Dodd, P. Dauban, Efficient diastereoselective intermolecular rhodium-catalyzed C–H amination. *Angew. Chem. Int. Ed.* **45**, 4641–4644 (2006).

5. D. N. Zalatan, J. Du Bois, A chiral rhodium carboxamidate catalyst for enantioselective C–H amination. *J. Am. Chem. Soc.* **130**, 9220–9221 (2008).

6. E. Milczek, N. Boudet, S. Blakey, Enantioselective C–H amination using cationic ruthenium (II)–pybox catalysts. *Angew. Chem. Int. Ed.* **47**, 6825–6828 (2008).

7. T. K. Hyster, L. Knorr, T. R. Ward, T. Rovis, Biotinylated Rh(III) complex in engineered streptavidin for accelerated asymmetric C–H activation. *Science* **338**, 500–503 (2012).

8. B. Ye, N. Cramer, Chiral cyclopentadienyl ligand as stereocontrolling element in asymmetric C–H functionalization. *Science* **338**, 504–506 (2012).

9. F. Kakiuchi, P. L. Gendre, A. Yamada, H. Ohtaki, S. Murai, Atropselective alkylation of biaryl compounds by means of transition metal-catalyzed C–H/olefin coupling. *Tetrahedron: Asymmetry* **11**, 2647–2651 (2000).

10. B.-F. Shi, N. Maugel, Y.-H. Zhang, J.-Q. Yu, Pd^{II}-catalyzed enantioselective activation of $C(sp^2)$–H and $C(sp^3)$–H bonds using monoprotected amino acids as chiral ligands. *Angew. Chem. Int. Ed.* **47**, 4882–4886 (2008).

11. L. Chu, X.-C. Wang, C. E. Moore, A. L. Rheingold, J.-Q. Yu, Pd-catalyzed enantioselective C–H iodination: asymmetric synthesis of chiral diarylmethylamines. *J. Am. Chem. Soc.* **135**, 16344–16347 (2013).

12. M. Nakanishi, D. Katayev, C. Besnard, E. P. K*ü*ndig, Fused indolines by palladium-catalyzed asymmetric C–C coupling involving an unactivated methylene group. *Angew. Chem. Int. Ed.* **50**, 7438–7441 (2011).

13. S. Anas, A. Cordi, H. B. Kagan, Enantioselective synthesis of 2-methyl indolines by palladium catalyzed asymmetric $C(sp^3)$–H activation/cyclisation. *Chem. Comm.* **47**, 11483–11485 (2011).

14. T. Saget, S. Lemouzy, N. Cramer, Chiral monodentate phosphines and bulky carboxylic acids: cooperative effects in palladium-catalyzed enantioselective $C(sp^3)$–H functionalization. *Angew. Chem. Int. Ed.* **51**, 2238–2242 (2012).

15. D. Katayev, M. Nakanishi, T. B*ü*rgi, E. P. K*ü*ndig, Asymmetric $C(sp^3)$-H/C(Ar) coupling reactions. Highly enantio-enriched indolines via regiodivergent reaction of a racemic mixture. *Chem. Sci.* **3**, 1422–1425 (2012).

16. F. Schmidt, R. T. Stemmler, J. Rudolph, C. Bolm, Catalytic asymmetric approaches towards enantiomerically enriched diarylmethanols and diarylmethylamines. *Chem. Soc. Rev.* **35**, 454–470 (2006).

17. M. T. Robak, M. A. Herbage, J. A. Ellman, Synthesis and application of *tert*-butanesulfinamide. *Chem. Rev.* **110**, 3600–3740 (2010).

18. J. Paetzold, J. E. Bäckvall, Chemoenzymatic dynamic resolution of primary amines. *J. Am. Chem. Soc.* **127**, 17620–17621 (2005).

19. M. Breuer, K. Ditrich, T. Habicher, B. Hauer, M. Keβeler, R. Stürmer, T. Zelinski, Industrial methods for the production of optically active intermediates. *Angew. Chem. Int. Ed.* **43**, 788–824 (2004).

20. S. Arai, S. Bellemin-Laponnaz, G. C. Fu, Kinetic resolution of amines by a nonenzymatic acylation catalyst. *Angew. Chem. Int. Ed.* **40**, 234–236 (2001).

21. C. K. De, E. G. Klauber, D. Seidel, Merging nucleophilic and hydrogen bonding catalysis: an anion binding approach to the kinetic resolution of amines. *J. Am. Chem. Soc.* **131**, 17060–17061 (2009).

22. B. S. Fowler, P. J. Mikochik, S. J. Miller, Peptide-catalyzed kinetic resolution of formamides and thioformamides as an entry to nonracemic amines. *J. Am. Chem. Soc.* **132**, 2870–2871 (2011).

23. D. -W. Gao, Q. Gu, S. -L. You, Pd(II)-catalyzed intermolecular direct C–H bond Iodination: an efficient approach toward the synthesis of axially chiral compounds via kinetic resolution. *ACS Catal.* **4**, 2741–2745 (2014).

24. D. R. Jensen, J. S. Pugsley, M. S. Sigman, Palladium-catalyzed enantioselective oxidations of alcohols using molecular oxygen. *J. Am. Chem. Soc.* **123**, 7475–7476 (2001).

25. E. M. Ferreira, B. M. Stoltz, The palladium-catalyzed oxidative kinetic resolution of secondary alcohols with molecular oxygen. *J. Am. Chem. Soc.* **123**, 7725–7726 (2001).

26. M. Tokunaga, J. F. Larrow, F. Kakiuchi, E. N. Jacobsen, Asymmetric catalysis with water: efficient kinetic resolution of terminal epoxides by means of catalytic hydrolysis. *Science* **277**, 936–938 (1997).

27. J. Song, Y. Wang, L. Deng, The mannich reaction of malonates with simple imines catalyzed by bifunctional cinchona alkaloids: enantioselective synthesis of β-amino acids. *J. Am. Chem. Soc.* **128**, 6048–6049 (2006).

28. A. Berkessel, F. Cleemann, S. Mukherjee, Kinetic resolution of oxazinones: an organocatalytic approach to enantiomerically pure β-amino acids. *Angew. Chem. Int. Ed.* **44**, 7466–7469 (2005).

29. V. D. Bumbu, V. B. Birman, Kinetic resolution of *N*-acyl-β-lactams via benzotetramisole-catalyzed enantioselective alcoholysis. *J. Am. Chem. Soc.* **133**, 13902–13905 (2011).

30. S. Zhou, J. Wang, X. Chen, J. L. Acena, V. A. Soloshonok, H. Liu, Chemical kinetic resolution of unprotected β-substituted-β-amino acids using recyclable chiral ligands. *Angew. Chem. Int. Ed.* **53**, 7883–7886 (2014).

31. Y. Zhang, Z. Lu, A. Desai, W. D. Wulff, Mapping the active site in a chemzyme: diversity in the *N*-substituent in the catalytic asymmetric aziridination of imines. *Org. Lett.* **10**, 5429–5432 (2008).

32. K. S. L., Chan, M. Wasa, L. Chu, B. N., Laforteza, M. Miura, J.-Q. Yu, Ligand–enabled cross-coupling of C(sp^3)–H bonds with arylboron reagents via Pd(II)/Pd(0) catalysis. *Nature Chem.* **6**, 146–150 (2014).

33. K. L. Billingsley, K. W., Anderson, S. L. Buchwald, A highly active catalyst for Suzuki–Miyaura cross-coupling reactions of heteroaryl compounds. *Angew. Chem. Int. Ed.* **45**, 3484–3488 (2006).

34. T.-S. Mei, D.-H. Wang, J.-Q. Yu, Expedient drug synthesis and diversification via *ortho*-C−H iodination using recyclable PdI_2 as the precatalyst. *Org. Lett.* **12**, 3140–3143 (2010).

35. M. Wolter, G. Nordmann, G. E. Job, S. L. Buchwald, Copper-catalyzed coupling of aryl iodides with aliphatic alcohols. *Org. Lett.* **4**, 973–976 (2002).