Supplementary Information

Enantioselective amination of nitroolefins under base-free and water-rich conditions by chiral bifunctional phase-transfer catalysts

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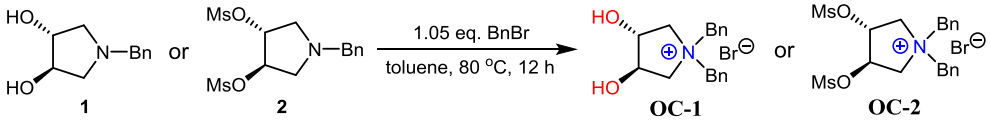
General

^{1}H NMR, ^{13}C NMR, ^{19}F and ^{31}P spectra were recorded at room temperature using 400 MHz *Bruker spectrometer*. The data are reported as follows: chemical shift δ in ppm (from internal tetramethylsilane on the δ scale in case of ^{1}H and $CDCl_3$ triplet in case of ^{13}C), multiplicity (br = broad, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants (Hz) and integration. High resolution mass spectra were obtained by peak matching on *BrukermaXis Spectrometer*. Melting points are reported uncorrected and measured on Fukai-X-6 melting point apparatus. HPLC data were recorded on *Agilent 1260* with UV detector. Analytical thin layer chromatography was performed on 0.25 mm silica gel plates with UV-254 fluorescent indicator. Flash column chromatography was performed using indicated solvent system on 200~300 mesh silica gel (SiO_2). All air- and water-sensitive reactions were carried out under an inert atmosphere in glassware, which had been oven-dried as per standard procedure. Unless otherwise noted, all reagents were commercially obtained and used without further purification. Benzyl chloride and benzyl bromide were freshly distilled and used in following steps.

The synthesis of phase-transfer catalysts

1. Synthesis of chiral phase-transfer catalysts **OC-1**, **OC-2** and **OC-3**

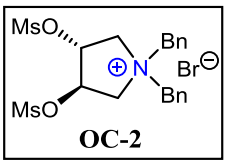


1 and **2** were prepared from D-tartaric acid according to published literature[1].

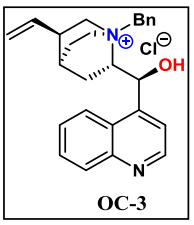
Typical procedure:

To 1.0 mmol of **1** (193 mg) in dry toluene (4.0 mL) was added by 1.05 mmol of BnBr (180 mg) in dry toluene (1.0 mL) at room temperature (r.t.). The mixture was heated to 80 °C by an oil-bath and stirred for 12 h under inert atmosphere. The reaction mixture was cooled to r.t. and the precipitate was collected and washed by dry Et_2O for three times. This precipitate was dried under a reduced pressure to give **OC-1** as a pale powder which was used directly without further purification. **OC-2** was obtained by using the same procedure as **OC-1**.

OC-1 is an known compound[2] and prepared according to the reported procedure.



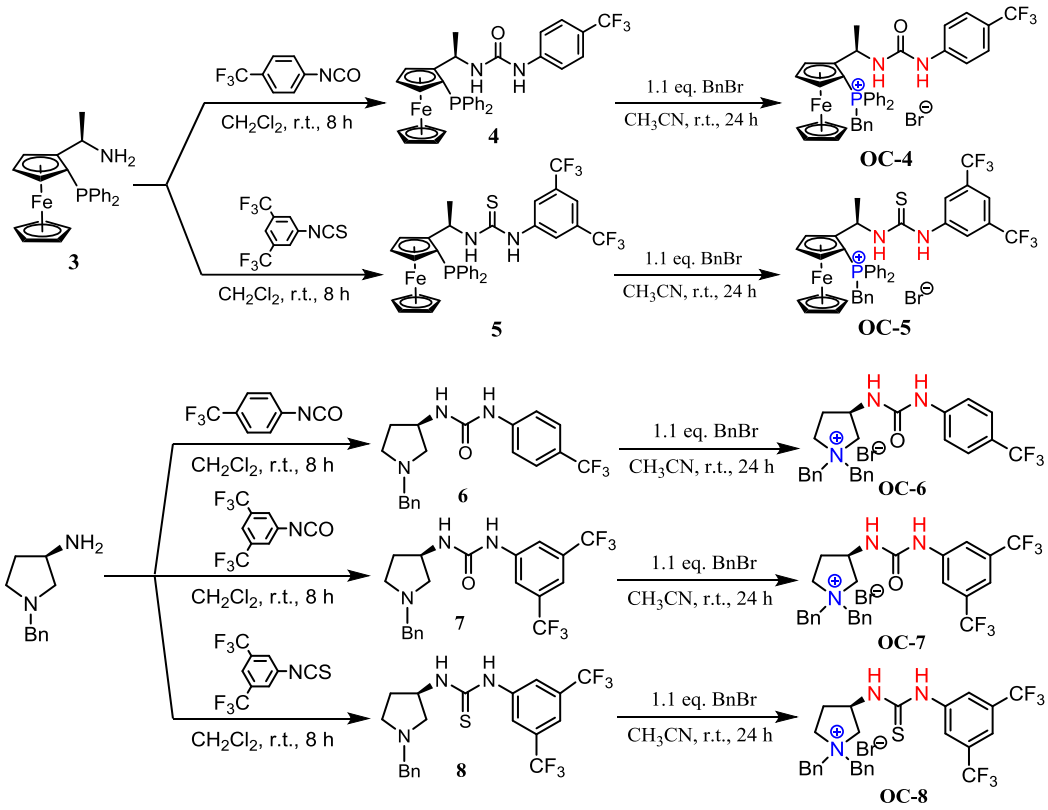
OC-2: pale powder, 93% yield; m.p. 34.8-35.5 °C; $[\alpha]^{20}_{D}$ 0° (c = 1, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.69 (bs, 4 H), 7.49 (bs, 5H), 7.41-7.29 (m, 1H), 5.83 (bs, 2H), 5.69 (d, *J* = 8.4 Hz, 2H), 4.88 (d, *J* = 11.2 Hz, 2H), 4.52 (br, 2H), 4.03 (d, *J* = 12.08 Hz, 2H), 3.49 (bs, 1H), 3.34 (s, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 133.74, 131.37, 129.74, 129.02,128.79, 126.65, 79.51, 66.36, 60.12, 39.05; HRMS (ESI) *m/z* calcd. for $C_{20}H_{26}NO_6S_2^+$ $[M-Br]^+$: 440.1196, found 440.1210.



OC-3 was prepared in 92% yield according to reported process, and the ^{1}H and ^{13}C NMR was in agreement with the published literature[3].

2. Synthesis of chiral phase-transfer catalysts **OC-4**~**OC-8**

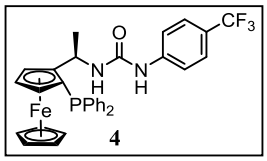
Amine **3** was prepared from (*R*)-Ugi's amine according to our published literatures[4]. (*R*)-*N*-Benzyl-3-amino-pyrrolidine was purchased and used directly.



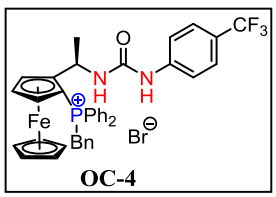
Typical procedure for preparation of chiral bifunctional phase-transfer catalysts with a (thio)urea moiety

Step 1: To 0.5 mmol of amine in dry DCM (4.0 mL) was added by 0.5 mmol of iso(thio)cyanate in dry DCM (1.0 mL) at room temperature (r.t.). The mixture was stirred for 8 h under inert atmosphere (checked by TLC). The solvent was evaporated to afford crude product as an orange glue which purified by a flash column chromatography (petroether/EtOAc, 10/1 to 5/1, v/v) to provide pure product.

Step 2: To 0.4 mmol of (thio)urea in dry CH_3CN (4.0 mL) was added by 0.5 mmol of BnBr in dry CH_3CN (1.0 mL) at room temperature (r.t.). The mixture was stirred for 24 h under inert atmosphere (checked by TLC). The solvent was evaporated to afford crude phase-transfer catalyst which purified by a flash column chromatography ($CHCl_3$/CH_3OH, 20/1~5/1, v/v) to provide pure product.



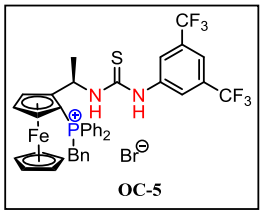
4: orange powder, 90% yield; m.p. 117.0-118.1 °C; $[\alpha]^{20}_D$ +305° (c = 0.26, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.50-7.47 (m, 4H), 7.40 (bs, 3H), 7.26-7.23 (m, 7H), 5.49 (s, 1H), 5.15 (t, *J* = 6.52 Hz, 1H), 4.76 (d, *J* = 6.92 Hz, 1H), 4.51 (s, 1H), 4.33 (s, 1H), 4.10 (s, 1H), 4.06 (s, 4H), 3.80 (s, 1H), 1.53 (d, *J* = 5.64 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 152.86, 142.18, 140.27, 136.42, 135.01, 134.80, 132.77, 132.58, 129.28, 128.36, 128.30, 128.25, 128.17, 126.05, 126.01, 118.39, 95.06, 94.82, 75.06, 74.98, 72.14, 70.07, 69.94, 69.10, 45.77, 21.43; ^{19}F NMR (376 MHz, $CDCl_3$) δ -61.76; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 24.90; HRMS (ESI) *m/z* calcd. for $C_{32}H_{29}F_3FeN_2OP^+$ $[M+H]^+$: 601.1314, found 601.1318.



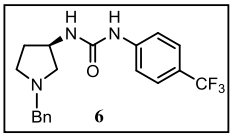
OC-4: golden powder, 72% yield; m.p. 174.6-175.3 °C; $[\alpha]^{20}_D$ +220° (c = 0.24, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.03 (bs, 1H), 7.82 (br, 1H), 7.74-7.69 (m, 4H), 7.61 (d, *J* = 9.08 Hz, 1H), 7.44-7.30 (m, 7H), 7.19 (d, *J* = 3.84 Hz, 4H), 6.98 (dd, *J* = 7.64, 4.2 Hz, 2H), 5.34 (t, *J* = 15.0 Hz, 1H), 5.13 (bs, 1H), 5.10 (br, 1H), 4.75 (br, 1H), 4.67 (d, *J* = 14.72 Hz, 1H), 4.40 (bs, 5H), 3.70 (bs, 1H), 1.71 (br, 1H), 1.69 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 154.42, 143.23, 135.01, 134.26 (d, *J* = 2.87 Hz), 133.97 (q, *J* = 8.96 Hz), 131.38 (d, *J* = 5.35 Hz), 129.70 (t, *J* = 14.18 Hz), 129.02 (d, *J* = 2.74 Hz), 128.83 (*d*, *J* = 3.57 Hz), 127.54 (d, *J* = 8.43

Hz), 125.99, 125.43, 122.83, 122.51, 120.29, 119.42, 117.45, 94.78 (d, *J* = 11.83 Hz), 75.79 (d, *J* = 12.79 Hz), 74.41 (d, *J* = 9.81 Hz), 73.29 (d, *J* = 10.55 Hz), 71.38, 60.14, 59.18, 43.29, 31.25, 30.75, 21.27; ^{19}F NMR (376 MHz, $CDCl_3$) δ: -61.44; ^{31}P NMR (162 MHz, CDCl3) δ: 27.74; HRMS (ESI) *m/z* calcd. for $C_{39}H_{35}F_3N_2OP^+$ $[M\text{-}Br]^+$: 691.1783, found 691.1789.

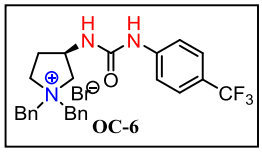
Ferrocene-based thiourea **5** is known compound, and prepared according to our published method[5].



OC-5: golden powder, 80% yield; m.p. 164.5-165.7 °C; $[\alpha]^{20}_D$ +120° (c = 0.22, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 10.15 (s, 1H), 9.38 (d, *J* = 12.56 Hz, 1H), 7.83 (bs, 3H), 7.70 (br, 4H), 7.51 (bs, 3H), 7.44 (d, *J* = 6.44 Hz, 1H), 7.34 (br, 1H), 7.25 (br, 4H), 7.10 (dd, *J* = 7.96, 3.24 Hz, 2H), 5.72 (br, 1H), 5.43 (t, *J* = 14.62 Hz, 1H), 5.19 (br, 1H), 4.81 (t, *J* = 13.92 Hz, 2H), 4.43 (s, 5H), 3.75 (bs, 1H), 1.74 (d, *J* = 5.84 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 178.74, 140.77, 135.16 (d, *J* = 2.77 Hz), 134.42 (d, *J* = 9.68 Hz), 133.89 (d, *J* = 9.78 Hz), 131.37 (d, *J* = 5.53 Hz), 131.10, 130.77, 129.86 (dd, *J* = 8.35, 3.94 Hz), 129.15 (d, *J* = 2.65 Hz), 128.98 (d, *J* = 3.16 Hz), 127.43 (dd, J = 3.17, 1.65 Hz), 124.85, 123.47, 121.94, 119.85, 118.97, 117.72, 117.18, 116.86, 92.84, 76.17 (d, *J* = 12.5 Hz), 74.87 (d, *J* = 9.49 Hz), 73.74 (*d*, J = 10.73 Hz), 71.50, 60.50, 59.54, 47.82, 31.86, 31.37, 20.14; ^{19}F NMR (376 MHz, CDCl3) δ: -62.71; ^{31}P NMR (162 MHz, CDCl3) δ: 27.11; HRMS (ESI) *m/z* calcd. for $C_{40}H_{34}F_6FeN_2PS^+$ $[M\text{-}Br]^+$: 775.1428, found 775.1437.

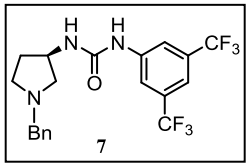


6: white powder, 93% yield; m.p. 109.3-110.9 °C; $[\alpha]^{20}_D$ -48.8° (c = 0.25, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.47 (d, *J* = 7.68 Hz, 2H), 7.38 (d, *J* = 7.48 Hz, 2H), 7.29 (br, 5H), 6.12 (d, *J* = 6.72 Hz, 1H), 4.07 (bs, 1H), 3.64 (s, 2H), 3.01 (bs, 1H), 2.82 (bs, 1H), 2.47 (t, *J* = 7.12 Hz, 1H), 2.31-2.20 (m, 2H), 2.12 (bs, 1H), 1.74 (bs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 155.19, 142.51, 129.03, 128.48, 127.48, 126.14, 126.10, 118.91, 60.97, 60.02, 52.98, 49.71, 32.42; ^{19}F NMR (376 MHz, CDCl3) δ: -61.83; HRMS (ESI) *m/z* calcd. for $C_{19}H_{21}F_3N_3O^+$ $[M+H]^+$: 364.1631, found 364.1643.

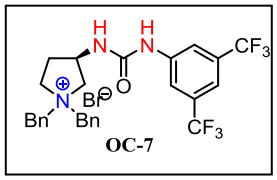


OC-6: pale powder, 81% yield; m.p. 150.6-152.5 °C; $[\alpha]^{20}_D$ -21.7° (c = 0.24, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.79 (s, 1H), 8.20 (s, 1H), 7.63 (dd, *J* = 8.68, 2.60 Hz, 4H), 7.56-7.44 (m, 6H), 7.41 (t, *J* = 7.52 Hz, 1H), 7.33 (t, *J* = 7.64 Hz, 2H), 5.41 (d, *J* = 12.72 Hz, 1H), 5.06 (d, *J* = 12.32 Hz, 1H), 4.94 (d, *J* = 12.84 Hz, 1H), 4.45 (br, 1H), 4.36 (d, *J* = 12.88 Hz, 1H), 4.25-4.19 (m, 1H), 3.91 (d, *J* = 12.84 Hz, 1H), 3.70-3.62 (m, 2H), 2.42-2.34 (m, 1H), 2.10 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 155.27, 142.58, 133.14 (d, $J_{C\text{-}F}$ = 11.73 Hz), 131.05 (d, $J_{C\text{-}F}$ = 17.25 Hz), 129.57 (d, $J_{C\text{-}F}$ = 14.56 Hz), 127.30, 126.88, 125.97(m), 125.74, 123.97, 123.65, 123.04, 117.96, 66.80, 64.11, 62.66, 58.58, 43.13, 28.99; ^{19}F NMR (376 MHz, $CDCl_3$) δ: -61.70; HRMS (ESI) *m/z* calcd. for $C_{26}H_{27}F_3N_3O^+$ $[M\text{-}Br]^+$: 452.2101, found 454.2108.

Compounds **7**[6] and **8**[7] are known, and prepared according to these literatures.

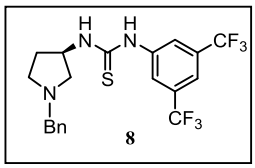


7: white crystal, 92% yield; m.p. 126.9-128.3 °C; $[\alpha]^{20}_D$ -65.6° (c = 0.25, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.79 (bs, 2H), 7.46(bs, 1H), 7.35-7.28 (m, 5H), 6.50 (s, 1H), 3.67 (s, 2H), 3.09 (bs, 1H), 2.89 (bs, 1H), 2.52 (bs, 1H), 2.37-2.26 (m, 2H), 1.79 (br, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 179.09, 155.09, 140.88, 136.83, 132.00 (q, $J_{C\text{-}F}$ = 33 Hz), 129.12, 128. 60, 127.75, 127.27, 124.56, 121.85, 118.81, 115.67, 60.64, 59.97, 52.96, 49.62, 32.18; ^{19}F NMR (376 MHz, $CDCl_3$) δ: -63.05; HRMS (ESI) *m/z* calcd. for $C_{20}H_{20}F_6N_3O^+$ $[M+H]^+$: 432.1505, found 432.1520.



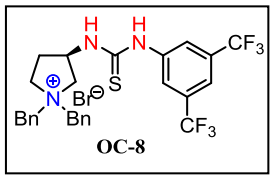
OC-7: white powder, 89% yield; m.p. 115.6-117.1 °C; $[\alpha]^{20}_D$ -20.6° (c = 0.28, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.04 (s, 1H), 8.18 (d, *J* = 5.88 Hz, 1H), 7.99 (s, 2H), 7.61(d, *J* = 7.12 Hz, 2H), 7.54-7.41 (m, 7H), 7.36 (t, *J* = 7.64 Hz, 2H), 5.34 (d, *J* = 12.8 Hz, 1H), 5.04 (d, *J* = 12.88 Hz, 1H), 4.95 (d, *J* = 12.88 Hz, 1H), 4.46 (br, 1H), 4.43 (bs, 1H), 4.23-4.17 (m, 1H), 3.89 (dd, *J* = 9.64, 3.28 Hz, 1H), 3.76-3.69 (m, 1H), 3.68-3.62

(m, 1H), 2.40-2.32 (m, 1H), 2.23-2.14 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 155.10, 140.95, 133.15, 131.91 (q, J_{C-F} = 33 Hz), 131.21, 131.03, 129.70, 129.51, 123.17, 126.71, 124.66, 118.05, 115.25, 66.72, 64.16, 62.28, 58.57, 48.15, 29.00; ^{19}F NMR (376 MHz, $CDCl_3$) δ: -62.91; HRMS (ESI) *m/z* calcd. for $C_{27}H_{26}F_6N_3O^+$ $[M-Br]^+$: 522.1975, found 522.1983.



8

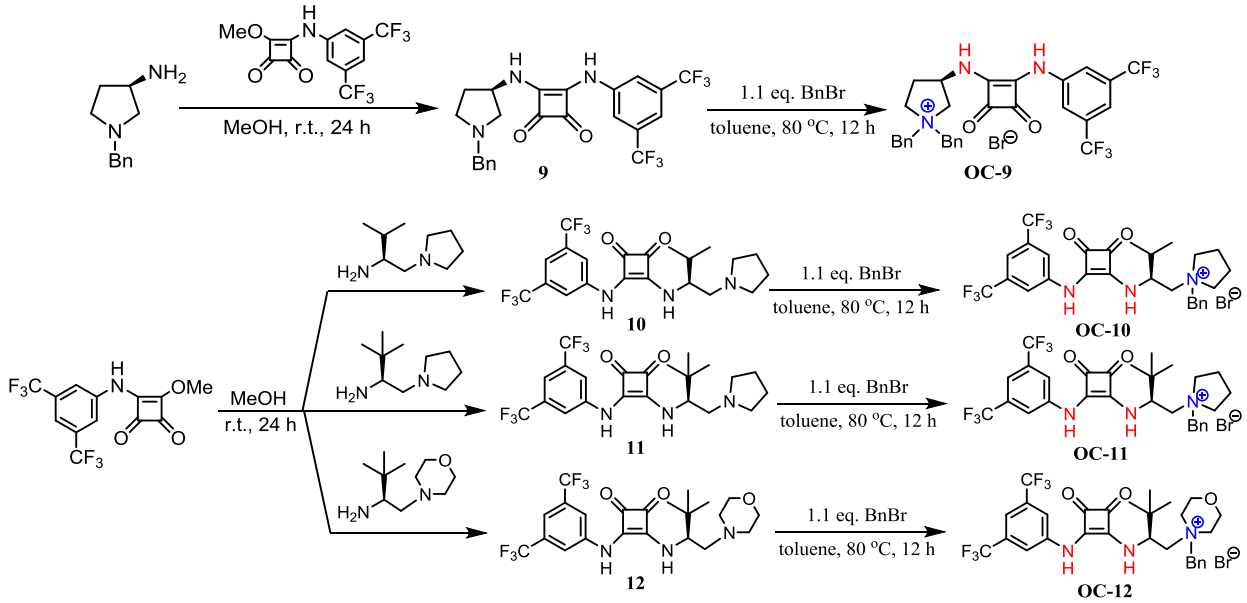
8: white crystal, 92% yield; m.p. 129.0-130.1 °C; $[\alpha]^{20}_D$ -89.1° (c = 0.24, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 12.07 (bs, 1H), 7.89 (bs, 2H), 7.72 (s, 1H), 7.36 (s, 1H), 7.27-7.23 (m, 2H), 7.11 (br, 2H), 3.96 (br, 1H), 3.65 (m, 2H), 3.30 (br, 1H), 3.19 (br, 1H), 2.47-2.45 (m, 1H), 2.43-2.37 (m, 1H), 2.25 (q, *J* = 8.4 Hz, 1H), 2.00 (br, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 179.64, 141.28, 136.03, 131.73, 129.07, 128.61, 128.05, 127.15, 126.13, 124.46, 121.74, 119.03, 59.30, 58.73, 53.73, 53.29, 30.36; ^{19}F NMR (376 MHz, $CDCl_3$) δ: -62.73; HRMS (ESI) *m/z* calcd. for $C_{20}H_{20}F_6N_3S^+$ $[M+H]^+$: 448.1277, found 448.1288.



OC-8

OC-8: pale powder, 90% yield; m.p. 102.0-103.4 °C; $[\alpha]^{20}_D$ +5.2° (c = 1, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 10.15 (s, 1H), 9.86 (d, *J* = 4.92 Hz, 1H), 8.29 (s, 2H), 7.61-7.24 (m, 11H), 5.38 (d, *J* = 12.76 Hz, 1H), 5.08 (d, *J* = 12.76 Hz, 1H), 4.95 (d, *J* = 12.68 Hz, 1H), 4.32-4.27 (m, 2H), 4.09 (d, J = 13.2 Hz, 1H), 3.75-3.63 (m, 2H), 2.60-2.55 (m, 1H), 2.20-2.15 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 181.59, 140.68, 133.22, 131.75, 131.42, 131.28, 131.10, 129.77, 129.60, 124.55, 122.81, 121.84, 117.82, 67.18, 64.06, 61.98, 58.88, 51.84, 29.71, 28.29; ^{19}F NMR (376 MHz, $CDCl_3$) δ: -62.80; HRMS (ESI) *m/z* calcd. for $C_{27}H_{26}F_6N_3S^+$ $[M-Br]^+$: 538.1746, found 538.1754.

4. Synthesis of chiral phase-transfer catalysts **OC-9~12**



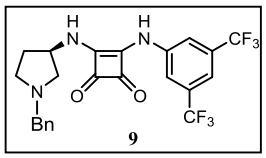
(*R*)-*N*-Benzyl-3-aminopyrrolidine and quinuclidin-3-amine were purchased and used directly. Squaramides **9~12** were prepared according to reported procedures[8]. Squaramides **9**[9] and **11**[10] are known compounds.

Typical procedure for preparation of chiral bifunctional phase-transfer catalysts with a squaramide moiety

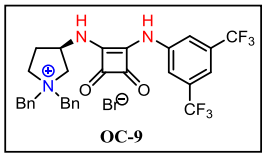
Step 1: To a solution of 3-((3,5-bis(trifluoromethyl)phenyl)amino)-4-methoxycyclobut-3-ene-1,2-dione (1.0 mmol) in MeOH (3 mL) was added a solution of amine (1.0 mmol) in MeOH (2 mL) at r.t.. The mixture was stirred for 24 h. The reaction mixture was filtered, and the precipitate was washed with cold MeOH (2×1.0 mL) to afford pure squaramide.

Step 2: To 0.5 mmol of squaramide in anhydrous toluene (2.0 mL) was added by 1.0 mmol of BnBr in anhydrous toluene (1.0 mL) at room temperature (r.t.). The mixture was stirred for 12 h under inert atmosphere

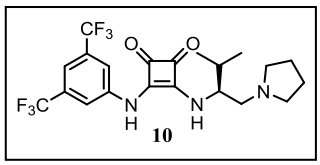
(checked by TLC) at 80 °C. The precipitate was collected and washed by anhydrous Et_2O (3×1.0 mL) to afford pure chiral phase-transfer catalysts.



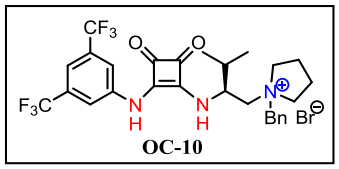
9: light yellow powder, 85% yield; m.p. 169.8-171.0 °C; $[\alpha]^{20}_D$ -64.5° (c = 0.24, CH_2Cl_2); ^{1}H NMR (400 MHz, MeOD) δ: 8.05 (bs, 2H), 7.55 (bs, 1H), 7.34-7.26 (m, 5H), 4.78 (s, 1H), 3.70 (s, 1H), 3.33 (s, 1H), 2.94 (br, 1H), 2.77 (bs, 2H), 2.45 (bs, 2H), 1.84 (bs, 1H), 4.73 (q, *J* = 6.9 Hz, 1H), 4.38 – 4.26 (m, 2H), 3.82 (dd, *J* = 12.3, 6.8 Hz, 1H), 2.86 (t, *J* = 7.0 Hz, 1H), 2.71 (s, 3H), 1.95 (bs, 1H), 1.52 (s, 3H), 1.35 (s, 3H); ^{13}C NMR (100 MHz, MeOD) δ: 168.97, 162.61, 140.90, 132.69, 128.75, 128.06, 127.06, 124.54, 121.84, 117.91, 115.23, 60.53, 59.51, 53.78, 52.21, 32.76; ^{19}F NMR (376 MHz, MeOD) δ: -64.51.



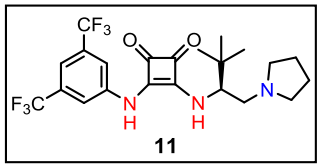
OC-9: pale powder, 90% yield; m.p. 204.6-205.1 °C; $[\alpha]^{20}_D$ +16.5° (c = 0.25, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 10.79 (bs, 1H), 9.78 (d, *J* = 5.84 Hz, 1H), 8.25 (s, 2H), 7.76 (d, *J* = 5.96 Hz, 2H), 7.53-7.46 (m, 9H), 5.17 (d, *J* = 12.92 Hz, 1H), 5.04 (d, *J* = 12.92 Hz, 1H), 4.88-4.85 (m, 2H), 4.58 (d, *J* = 12.08 Hz, 1H), 4.27-4.22 (m, 1H), 4.17 (d, *J* = 4.4 Hz, 2H), 3.67-3.60 (m, 1H), 2.61 (br, 1H), 2.34-2.25 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 183.86, 180.76, 168.60, 165.15, 140.54, 133.17-132.40 (dd, $J_{C\text{-}F}$ = 33.04, 23.4 Hz), 131.42, 129.88, 127.05, 126.63, 124.47, 121.76, 118.32, 116.12, 68.23, 66.06, 65.11, 57.77, 52.36, 30.26; ^{19}F NMR (376 MHz, $CDCl_3$) δ: -62.98; HRMS (ESI) *m/z* calcd. for $C_{30}H_{26}F_6N_3O_2^+$ $[M\text{-}Br]^+$: 574.1924, found 574.1930.



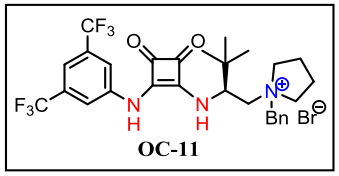
10: white powder, 92% yield; m.p. 178.5-179.2 °C; $[\alpha]^{20}_D$ -48.9° (c = 0.28, CH_2Cl_2); ^{1}H NMR (400 MHz, MeOD) δ: 8.13 (bs, 2H), 7.58 (s, 1H), 4.31 (br, 1H), 2.94 (t, *J* = 11.44 Hz, 1H), 2.79 (br, 2H), 2.63 (br, 3H), 1.99-1.91 (m, 1H), 1.85 (br, 4H), 1.02 (dd, J = 6.72, 5.76 Hz, 6H); ^{13}C NMR (100 MHz, MeOD) δ: 184.20, 180.83, 170.60, 162.78, 141.23, 132.68-132.35 (d, $J_{C\text{-}F}$ = 33.36 Hz), 124.60, 121.89, 117.90, 115.05, 58.68; 53.98, 31.71, 22.84, 18.29, 15.78; ^{19}F NMR (376 MHz, MeOD) δ -63.67; HRMS (ESI) *m/z* calcd. for $C_{21}H_{24}F_6N_3O_2^+$ $[M+H]^+$: 464.1767, found 464.1779.



OC-10: pale powder, 84% yield; m.p. 204.9-206.6 °C; $[[\alpha]^{20}_D$ +78.5° (c = 0.25, CH_2Cl_2); ^{1}H NMR (400 MHz, MeOD) δ: 8.24 (bs, 2H), 7.69-7.60 (m, 6H), 7.55-7.47 (m, 1H), 5.02-5.01 (m, 1H), 4.70 (bs, 2H), 3.90-3.76 (m, 2H), 3.72-3.50 (m, 4H), 2.39-2.34 (m, 1H), 2.28-2.18 (m, 3H), 2.05-1.97 (m, 1H), 1.05 (t, *J* = 6.4 Hz, 6H); ^{13}C NMR (100 MHz, MeOD) δ: 184.39, 180.38, 168.60, 165.29, 140.61, 132.58, 132.28, 131.39, 129.90, 129.82, 129.55, 128.34, 126.84, 124.54, 121.83, 118.50, 115.98, 63.02, 62.82, 62.39, 61.56, 54.67, 33.60, 21.90, 20.26, 19.39, 16.73; ^{19}F NMR (376 MHz, MeOD) δ -62.95; HRMS (ESI) *m/z* calcd. for $C_{28}H_{30}F_6N_3O_2^+$ $[M\text{-}Br]^+$: 554.2237, found 554.2243.

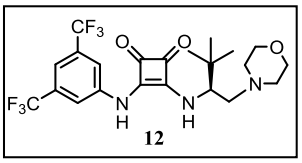


11: white powder, 80% yield; discom. 174.5 °C; $[\alpha]^{20}_D$ +6.49° (c = 0.23, MeOH); ^{1}H NMR (400 MHz, MeOD) δ: 8.13 (s, 2H), 7.59 (s, 1H), 4.20 (d, *J* = 10.6 Hz, 1H), 2.85-2.72 (m, 3H), 2.66 (d, *J* = 11.96 Hz, 1H), 2.53 (br, 2H), 1.83 (bs, 4H), 1.04 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$ + MeOD) δ: 184.17, 180.67, 170.85, 162.46, 141.01, 133.24-132.56 (m), 124.51, 121.80, 117.81, 115.29, 62.27, 56.71, 54.01, 34.30, 25.41, 22.98; ^{19}F NMR (376 MHz, $CDCl_3$ + MeOD) δ: -59.89; HRMS (ESI) *m/z* calcd. for $C_{22}H_{26}F_6N_3O_2^+$ $[M+H]^+$: 478.1924, found 478.1935.



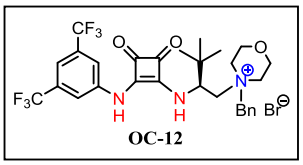
OC-11: white powder, 82% yield; m.p. 234.3-235.9 °C; $[\alpha]^{20}_D$ +19.6° (c = 0.21, MeOH); ^{1}H NMR (400 MHz, $CDCl_3$ + MeOD) δ: 8.26 (s, 2H), 7.82 (s, 1H), 7.68-7.66 (m, 2H), 7.60-7.55 (m, 3H), 4.87 (s, 1H), 4.71 (d, *J* = 13.36 Hz, 1H), 4.59 (d, *J* = 13.44 Hz, 1H), 3.90-3.78 (m, 2H), 3.75 (s, 1H), 3.71-3.60 (m, 3H),

2.39-2.31 (m, 1H), 2.30-2.19 (m, 1H), 2.18-2.06 (m, 2H), 1.09 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$ + MeOD) δ: 184.47, 180.43, 168.61, 164.67, 140.62, 132.70-132.35 (m), 130.79, 129.32, 127.48, 121.78, 118.19, 62.79-61.56 (dd, $J_{C\text{-}F}$ = 60.78, 32.67 Hz), 58.22, 36.68, 25.07, 21.64, 20.39; ^{19}F NMR (376 MHz, $CDCl_3$ + MeOD) δ: -62.96; HRMS (ESI) *m/z* calcd. for $C_{29}H_{32}F_6N_3O_2^+$ $[M\text{-}Br]^+$: 568.2393, found 568.2405.



12

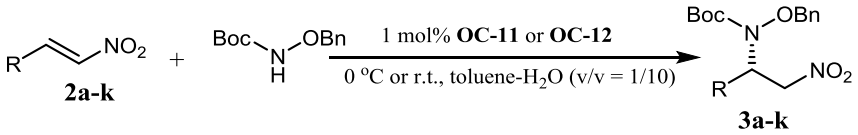
12: white powder, 80% yield; m.p. > 230 °C; $[\alpha]^{20}_D$ +5.3° (c = 0.22, CH_2Cl_2); ^{1}H NMR (400 MHz, MeOD) δ: 8.14 (s, 2H), 7.59 (s, 1H), 4.18 (d, J = 10.28 Hz, 1H), 3.62 (br, 4H), 2.70-2.65 (m, 3H), 2.48 (t, *J* = 12.0 Hz, 1H), 2.37 (br, 2H), 1.05 (s, 9H); ^{13}C NMR (100 MHz, MeOD) δ: 184.29, 180.54, 170.73, 161.93, 140.82, 132.80 (d, $J_{C\text{-}F}$ = 33 Hz), 124.46, 121.75, 117.78, 115.46, 66.83, 60.43, 59.49, 53.89. 33.70, 25.74; ^{19}F NMR (376 MHz, MeOD) δ: -63.99; HRMS (ESI) *m/z* calcd. for $C_{22}H_{26}F_6N_3O_3^+$ $[M+H]^+$: 494.1873, found 494.1886.



OC-12

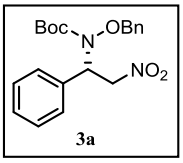
OC-12: white powder, 86% yield; m.p. 227.6-228.5 °C; $[\alpha]^{20}_D$ +23.1° (c = 0.26, MeOH); ^{1}H NMR (400 MHz, $CDCl_3$ + MeOD) δ: 8.22 (s, 2H), 7.63-7.53 (m, 5H), 7.50 (bs, 1H), 4.96-4.93 (d, *J* = 12.0 Hz, 1H), 4.81-4.79 (d, *J* = 8.0 Hz, 1H), 4.63-4.60 (d, *J* = 12.0 Hz, 1H), 4.33-4.18 (m, 3H), 4.07-3.98 (m, 2H), 3.77-3.74 (d, *J* = 12.0 Hz 1H), 3.65-3.50 (m, 3H), 3.40-3.36 (m, 1H), 1.13 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$ + MeOD) δ: 183.97, 180.15, 168.05, 165.02, 140.19, 133.53, 132.76-132.43 (d, $J_{C\text{-}F}$ = 33 Hz), 131.41-131.10 (d, $J_{C\text{-}F}$ = 31 Hz), 129.62, 125.53, 124.39, 121.69, 66.38-60.35 (m), 60.12, 59.92, 57.51, 57.12, 55.50, 37.23, 30.41, 25.57, 19.04, 13.34; ^{19}F NMR (376 MHz, $CDCl_3$ + MeOD) δ: -63.68; HRMS (ESI) *m/z* calcd. for $C_{29}H_{32}F_6N_3O_3^+$ $[M\text{-}Br]^+$: 584.2342, found 584.2355.

The direct enantioselective amination of nitroolefins by phase-transfer catalysts



General procedure for the neutral aminations

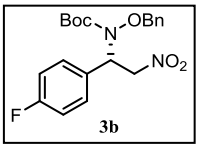
Bifunctional phase-transfer catalyst (**OC-11** or **OC-12**, 1 mol%), nitroolefins (0.1 mmol) and amination reagent (0.3 mmol) were added to a 10-mL vial equipped with a stirring bar, the mixed solvent of toluene (0.2 mL) and H_2O (2.0 mL) was added subsequently. The mixture was stirred at 0 °C or room temperature for the indicated time and then the mixture was extracted by EtOAc (3×5.0 mL), the combined organic layers were dried over anhydrous Na_2SO_4 and evaporated under reduced pressure to yield a light yellow glue which was purified by flash column chromatography on silica gel, eluting with mixtures of petroether/EtOAc (50/1 to 10/1, v/v). The *ee* of products **3a-k** were determined by HPLC using a chiral column (Daicel Chiralpak AS-H), and the absolute configurations of products were assigned according to literature report[11].



3a

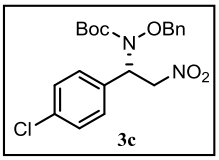
3a: light yellow oil, $[\alpha]^{20}_D$ -11.9° (c = 1.15, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.41 (br, 10H), 5.91 (br, 1H), 5.15-5.10 (m, 1H), 4.82-4.70 (m, 3H), 1.50 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 156.31, 135.13, 135.04, 129.56, 128.89, 128.78, 128.58, 128.08, 82.86, 78.59, 75.19, 61.29, 28.16; HRMS (ESI) *m/z* calcd. for $C_{20}H_{24}N_2NaO_5^+$ $[M+Na]^+$: 395.1577, found 395.1584. HPLC: Chiralpak AS-H (^{i}PrOH/*n*-Hexane) = 3.2:96.8, flow rate = 0.5 mL/min, 210 nm; *rac*-form: t_r = 19.161 and 23.377 min.

Entry	Cat.	T	Yield	t_r [minor]	t_r [major]	*ee*[%]	Entry	Cat.	T	Yield	t_r [minor]	t_r [major]	*ee*[%]
1	**OC-11**	0 °C	91%	15.124	18.089	87	3	**OC-12**	0 °C	92%	14.781	17.610	89
2	**OC-11**	r.t.	96%	15.302	18.318	84	4	**OC-12**	r.t.	96%	15.285	18.227	83



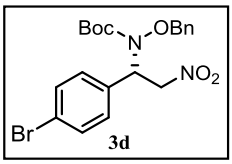
3b: light yellow oil, $[\alpha]^{20}_D$ -9.3° (c = 0.30, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.39 (br, 7H), 7.06 (br, 2H), 5.85 (d, *J* = 4.4 Hz, 1H), 5.06 (m, 1H), 4.79 (m, 1H), 4.75-4.70 (m, 2H), 1.50 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 164.12, 161.65, 156.31, 134.91, 130.85, 130.03, 129.95, 129.50, 128.82, 128.59, 115.90, 115.69, 83.08, 78.64, 75.25, 60.60, 28.14; ^{19}F NMR (376 MHz, $CDCl_3$) δ: -112.41; HRMS (ESI) *m/z* calcd. for $C_{20}H_{23}FN_2O_5Na^+$ $[M+Na]^+$: 413.1483, found 413.1481.
HPLC: Chiralpak AS-H (^{i}PrOH/*n*-Hexane) = 3.2:96.8, flow rate = 0.5 mL/min, 210 nm; *rac*-form: t_r = 19.179 and 25.168 min.

Entry	Cat.	T	Yield	t_r[minor]	t_r[major]	*ee*[%]	Entry	Cat.	T	Yield	t_r[minor]	t_r[major]	*ee*[%]
5	**OC-11**	0 °C	94%	17.895	23.435	86	7	**OC-12**	0 °C	92%	17.623	22.652	86
6	**OC-11**	r.t.	95%	18.371	24.099	85	8	**OC-12**	r.t.	96%	19.153	24.795	84



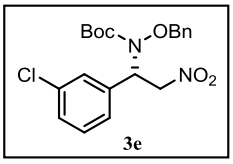
3c: light yellow oil, $[\alpha]^{20}_D$ -22.7° (c = 0.22, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.39 (br, 6H), 7.34 (br, 3H), 5.83 (br, 1H), 5.05 (dd, *J* = 10.12, 2.44 Hz, 1H), 4.79-4.70 (m, 3H), 1.50 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 158.28, 134.90, 134.49, 129.48, 129.12, 129.04, 128.83, 128.59, 83.17, 78.67, 75.10, 60.67, 28.15; HRMS (ESI) *m/z* calcd. for $C_{20}H_{23}ClN_2O_5Na$ $[M+Na]^+$: 429.1188, found 429.1195.
HPLC: Chiralpak AS-H (^{i}PrOH/*n*-Hexane) = 3.2:96.8, flow rate = 0.5 mL/min, 210 nm; *rac*-form: t_r = 18.050 and 23.326 min.

Entry	Cat.	T	Yield	t_r[minor]	t_r[major]	*ee*[%]	Entry	Cat.	T	Yield	t_r[minor]	t_r[major]	*ee*[%]
9	**OC-11**	0 °C	92%	19.680	26.305	91	11	**OC-12**	0 °C	92%	18.365	23.803	89
10	**OC-11**	r.t.	96%	18.434	24.307	85	12	**OC-12**	r.t.	95%	18.081	23.446	84



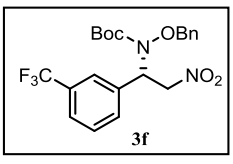
3d: light yellow oil, $[\alpha]^{20}_D$ -0.144° (c = 0.045, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.51 (s, 1H), 7.49 (s, 1H), 7.39 (br, 4H), 7.28 (s, 1H), 7.26 (s, 1H), 5.83-5.79 (dd, *J* = 3.28, 5.84 Hz, 1H), 4.79-4.69 (m, 3H), 1.49 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 156.29, 134.82, 133.93, 132.02, 129.77, 129.51, 128.86, 128.61, 123.10, 83.22, 78.68, 75.01, 60.69, 28.15; HRMS (ESI) *m/z* calcd. for $C_{20}H_{23}BrN_2NaO_5^+$ $[M+Na]^+$: 473.0683, found 473.0688.
HPLC: Chiralpak AS-H (^{i}PrOH/*n*-Hexane) = 3.2:96.8, flow rate = 0.5 mL/min, 210 nm; *rac*-form: t_r = 19.619 and 26.223 min.

Entry	Cat.	T	Yield	t_r[minor]	t_r[major]	*ee*[%]	Entry	Cat.	T	Yield	t_r[minor]	t_r[major]	*ee*[%]
13	**OC-11**	0 °C	93%	20.971	28.186	90	15	**OC-12**	0 °C	96%	18.933	24.623	88
14	**OC-11**	r.t.	96%	19.802	26.237	88	16	**OC-12**	r.t.	96%	19.380	25.142	85



3e: light yellow oil, $[\alpha]^{20}_D$ -7.0° (c = 0.30, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.39-7.31 (m, 9H), 5.83 (d, *J* = 3.92 Hz, 1H), 5.08-5.02 (m, 1H), 4.80 (br, 1H), 4.76-4.67 (m, 2H), 1.52-1.51 (d, *J* = 2.28 Hz, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 158.20, 136.97, 134.79, 134.71, 130.19, 129.56, 129.12, 128.86, 128.61, 128.33, 126.25, 83.25, 78.71, 74.90, 60.75, 28.14; HRMS (ESI) *m/z* calcd. for $C_{20}H_{23}ClN_2O_5Na$ $[M+Na]^+$: 429.1188, found 429.1197.
HPLC: Chiralpak AS-H (^{i}PrOH/*n*-Hexane) = 3.2:96.8, flow rate = 0.5 mL/min, 210 nm; *rac*-form: t_r = 16.241 and 20.425 min.

Entry	Cat.	T	Yield	t_r[minor]	t_r[major]	*ee*[%]	Entry	Cat.	T	Yield	t_r[minor]	t_r[major]	*ee*[%]
17	**OC-11**	0 °C	82%	15.904	19.928	89	19	**OC-12**	0 °C	81%	16.839	21.252	87
18	**OC-11**	r.t.	90%	16.024	20.031	87	20	**OC-12**	r.t.	91%	17.131	21.706	82

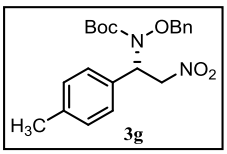


3f: light yellow oil, $[\alpha]^{20}_D$ -5.06 (c = 1.01, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.65-7.60 (m, 3H), 7.52-7.50 (m, 1H), 7.40 (br, 5H), 5.90 (d, *J* = 4.8 Hz, 1H), 5.12-5.05 (m, 1H), 4.80-4.75 (m, 3H), 1.29 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 156.28, 136.03, 134.77, 131.42, 129.44, 128.87, 128.61, 125.77, 125.07, 83.41, 78.72, 74.91, 60.94, 29.73;

^{19}F NMR (376 MHz, $CDCl_3$) δ -62.72; HRMS (ESI) *m/z* calcd. for $C_{21}H_{23}F_3N_2O_5Na$ $[M+Na]^+$: 463.1451, found 463.1466.

HPLC: Chiralpak AS-H (^{i}PrOH/*n*-Hexane) = 3.2:96.8, flow rate = 0.5 mL/min, 210 nm; *rac*-form: t_r = 13.459 and 17.146 min.

Entry	Cat.	T	Yield	t_r[minor]	t_r[major]	*ee*[%]	Entry	Cat.	T	Yield	t_r[minor]	t_r[major]	*ee*[%]
21	**OC-11**	0 °C	82%	13.560	17.489	91	23	**OC-12**	0 °C	84%	13.513	17.182	87
22	**OC-11**	r.t.	86%	13.314	17.048	88	24	**OC-12**	r.t.	85%	13.567	17.316	85

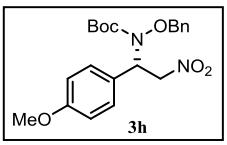


3g

3g: light yellow oil, $[\alpha]^{20}_D$ -0.87° (c = 0.90, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.41 (br, 5H), 7.31 (br, 2H), 7.20 (br, 2H), 5.89-5.87 (m, 1H), 5.11 (br, 1H), 4.83-4.68 (m, 3H), 2.38 (s, 3H), 1.53 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 156.31, 138.73, 135.12, 132.01, 129.52, 128.71, 128.54, 128.01, 82.76, 78.52, 75.27, 60.98, 28.18, 21.16; HRMS (ESI) *m/z* calcd. for $C_{21}H_{26}N_2NaO_5^+$ $[M+Na]^+$: 409.1734, found 409.1743.

HPLC: Chiralpak AS-H (^{i}PrOH/*n*-Hexane) = 3.2:96.8, flow rate = 0.5 mL/min, 210 nm; *rac*-form: t_r = 16.585 and 19.123 min.

Entry	Cat.	T	Yield	t_r[minor]	t_r[major]	*ee*[%]	Entry	Cat.	T	Yield	t_r[minor]	t_r[major]	*ee*[%]
25	**OC-11**	0 °C	90%	15.862	17.913	92	27	**OC-12**	0 °C	91%	16.318	18.616	90
26	**OC-11**	r.t.	91%	15.830	17.875	87	28	**OC-12**	r.t.	90%	16.452	18.899	82

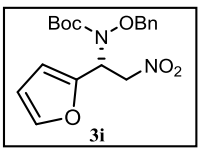


3h

3h: colorless oil, $[\alpha]^{20}_D$ -8.16° (c = 2.5, CH_2Cl_2); the product **3h** with BnONHBoc as 1:1 mixture, ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.38 (br, 9H); 7.32 (br, 2H), 7.29 (m, 1H), 7.12 (bs, 1H), 6.89 (br, 1H), 5.81 (d, *J* = 5.08 Hz, 1H), 5.10-5.03 (m, 1H), 4.89 (br, 2H), 4.80-4.70 (m, 2H), 4.66 (br, 1H), 3.82 (s, 3H), 1.50 (s, 18H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 159.92, 156.72, 156.31, 135.75, 135.04, 129.48, 129.43, 128.70, 128.51, 126.92, 114.15, 82.81, 81.76, 78.50, 75.34, 60.65, 55.28, 28.21, 28.17; HRMS (ESI) *m/z* calcd. for $C_{21}H_{26}N_2O_6Na^+$ $[M+H]^+$: 425.1683, found 425.1696.

HPLC: Chiralpak AS-H (^{i}PrOH/*n*-Hexane) = 3.2:96.8, flow rate = 0.5 mL/min, 210 nm; *rac*-form: t_r = 24.326 and 33.528 min. [a]The pure products **3h** couldn't be separated through a flash column chromatography, but the TLC results indicated full conversion of substrate **2h**.

Entry	Cat.	T	Yield	t_r[minor]	t_r[major]	*ee*[%]	Entry	Cat.	T	Yield	t_r[minor]	t_r[major]	*ee*[%]
29	**OC-11**	0 °C	-[a]	23.851	32.951	89	31	**OC-12**	0 °C	-[a]	23.484	32.007	91
30	**OC-11**	r.t.	-[a]	23.589	32.578	75	32	**OC-12**	r.t.	-[a]	25.017	34.344	80

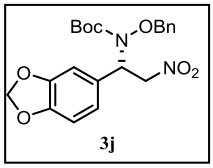


3i

3i: light yellow oil, $[\alpha]^{20}_D$ -1.3° (c = 1.2, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.44-7.37 (m, 8H), 7.15 (s, 1H), 6.39 (s, 1H), 4.98-4.78 (m, 3H), 1.50 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 156.74, 148.18, 142.80, 135.74, 134.92, 129.47, 129.15, 128.72, 128.52, 110.77, 109.15, 83.16, 81.76, 78.47, 73.52, 55.19, 28.22; HRMS (ESI) *m/z* calcd. for $C_{18}H_{22}N_2NaO_6^+$ $[M+Na]^+$: 385.1370, found 385.1384.

HPLC: Chiralpak AS-H (^{i}PrOH/*n*-Hexane) = 3.2:96.8, flow rate = 0.5 mL/min, 210 nm; *rac*-form: t_r = 15.769 and 20.060 min.

Entry	Cat.	T	Yield	t_r[minor]	t_r[major]	*ee*[%]	Entry	Cat.	T	Yield	t_r[minor]	t_r[major]	*ee*[%]
33	**OC-11**	0 °C	93%	15.181	19.348	91	35	**OC-12**	0 °C	92%	16.283	20.703	93
34	**OC-11**	r.t.	92%	15.490	19.930	82	36	**OC-12**	r.t.	95%	16.455	21.307	74



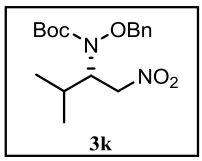
3j

3j: light yellow oil, $[\alpha]^{20}_D$ +15.6° (c = 0.25, CH_2Cl_2); the product **3j** with BnONHBoc as 1:1 mixture, ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.40 (br, 10H), 7.16 (br, 1H), 6.89 (br, 1H), 6.86 (br, 1H), 6.78 (m, 1H), 5.97 (br, 2H), 5.76 (d, *J* = 4.96 Hz, 1H), 5.05-5.01 (m, 1H), 4.88 (br, 2H), 4.81-4.65 (m, 3H), 1.51 (s, 18H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 156.74, 156.26, 148.02,

147.97, 135.76, 134.94, 129.55, 129.14, 128.77, 128.57, 128.51, 121.84, 108.57, 108.39, 101.32, 82.94, 81.74, 78.60, 78.46, 75.37, 60.90, 28.21; HRMS (ESI) *m/z* calcd. for $C_{21}H_{24}N_2O_7Na$ $[M+Na]^+$: 439.1476, found 439.1495.

HPLC: Chiralpak AS-H (^{i}PrOH/*n*-Hexane) = 3.2:96.8, flow rate = 0.5 mL/min, 210 nm; *rac*-form: t_r = 31.435 and 36.199 min. [a]The pure products **3j** couldn't be separated through a flash column chromatography, but the TLC results indicated full conversion of substrate **2j**.

Entry	Cat.	T	Yield	t_r [minor]	t_r [major]	*ee*[%]	Entry	Cat.	T	Yield	t_r [minor]	t_r [major]	*ee*[%]
37	**OC-11**	0 °C	-[a]	31.791	36.713	89	39	**OC-12**	0 °C	-[a]	31.003	35.892	89
38	**OC-11**	r.t.	-[a]	31.478	36.327	84	40	**OC-12**	r.t.	-[a]	31.430	35.927	83



3k

3k: light yellow oil, $[\alpha]^{20}_D$ +1.88° (c = 0.40, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.41 (m, 5H), 5.00 (d, *J* = 9.8 Hz, 1H), 4.83 (d, *J* = 9.8 Hz, 1H), 4.71 (dd, *J* = 1.56, 10.92 Hz, 1H), 4.57-4.45 (m, 2H), 2.08-1.99 (m, 1H), 1.54 (s, 9H), 1.02 (t, *J* = 6.40 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 156.88, 135.44, 129.06, 128.55, 128.49, 82.29, 78.10, 75.29, 64.85, 29.06, 28.21, 19.83, 19.66; HRMS (ESI) *m/z* calcd. for $C_{17}H_{26}N_2NaO_5^+$ $[M+Na]^+$: 361.1734, found 361.1749.

HPLC: Chiralpak AS-H (^{i}PrOH/*n*-Hexane) = 1.5:98.5, flow rate = 0.5 mL/min, 210 nm; *rac*-form: t_r = 13.353 and 14.337 min.

Entry	Cat.	T	Yield	t_r [minor]	t_r [major]	*ee*[%]	Entry	Cat.	T	Yield	t_r [minor]	t_r [major]	*ee*[%]
41	**OC-11**	0 °C	92%	12.816	13.708	93	43	**OC-12**	0 °C	92%	12.619	13.456	92
42	**OC-11**	r.t.	95%	11.955	12.708	92	44	**OC-12**	r.t.	93%	14.484	16.478	90

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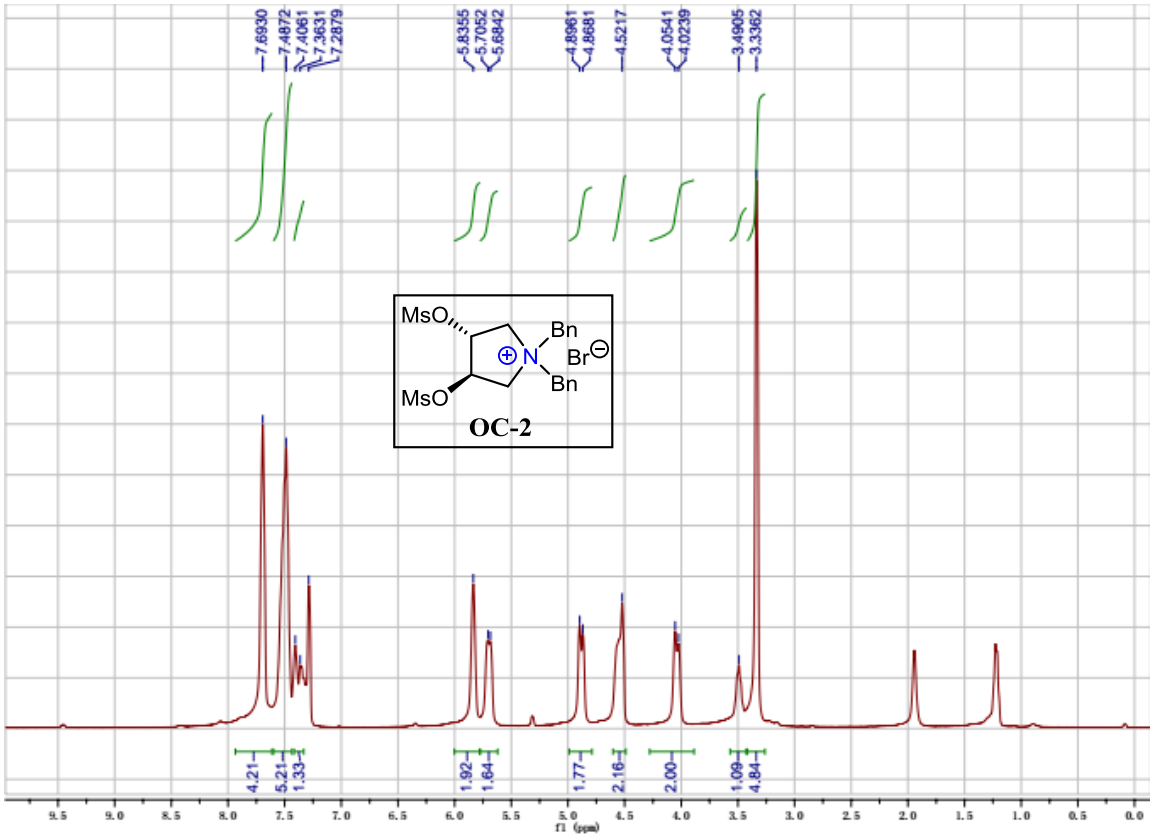
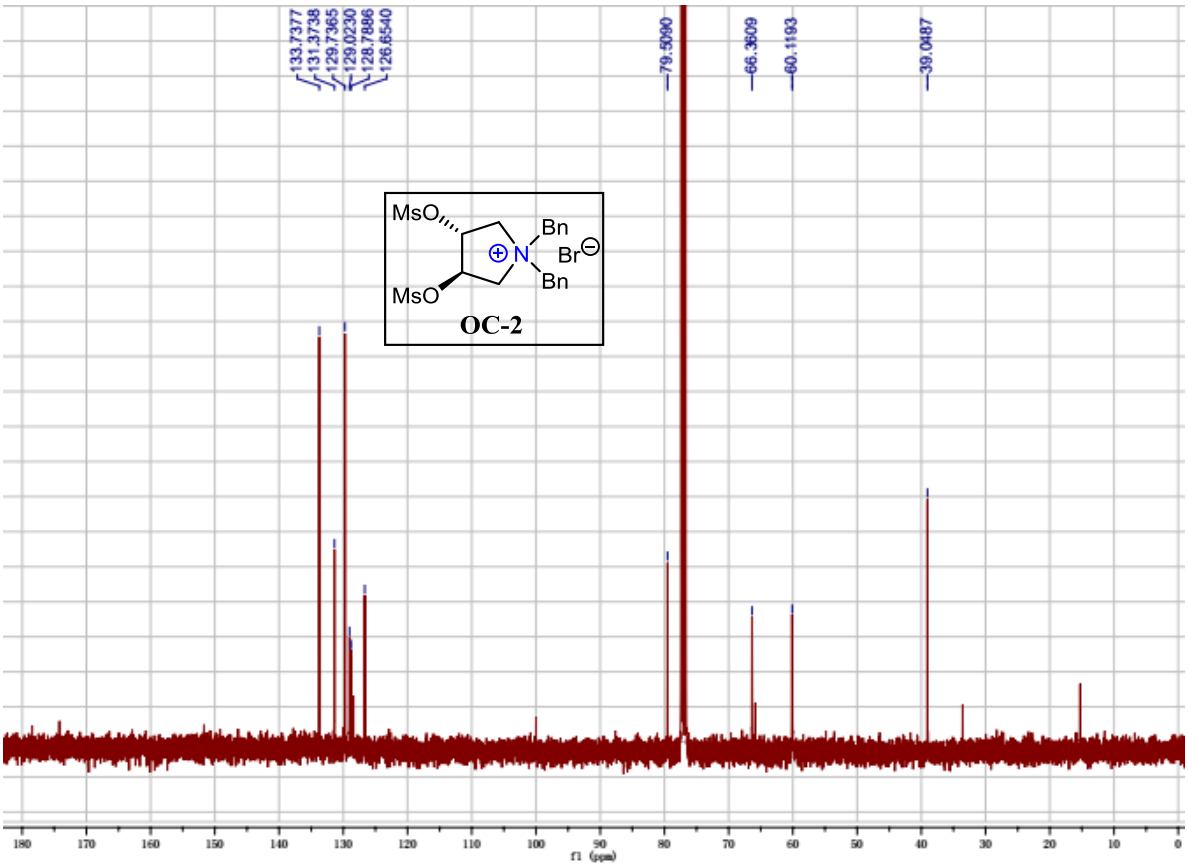
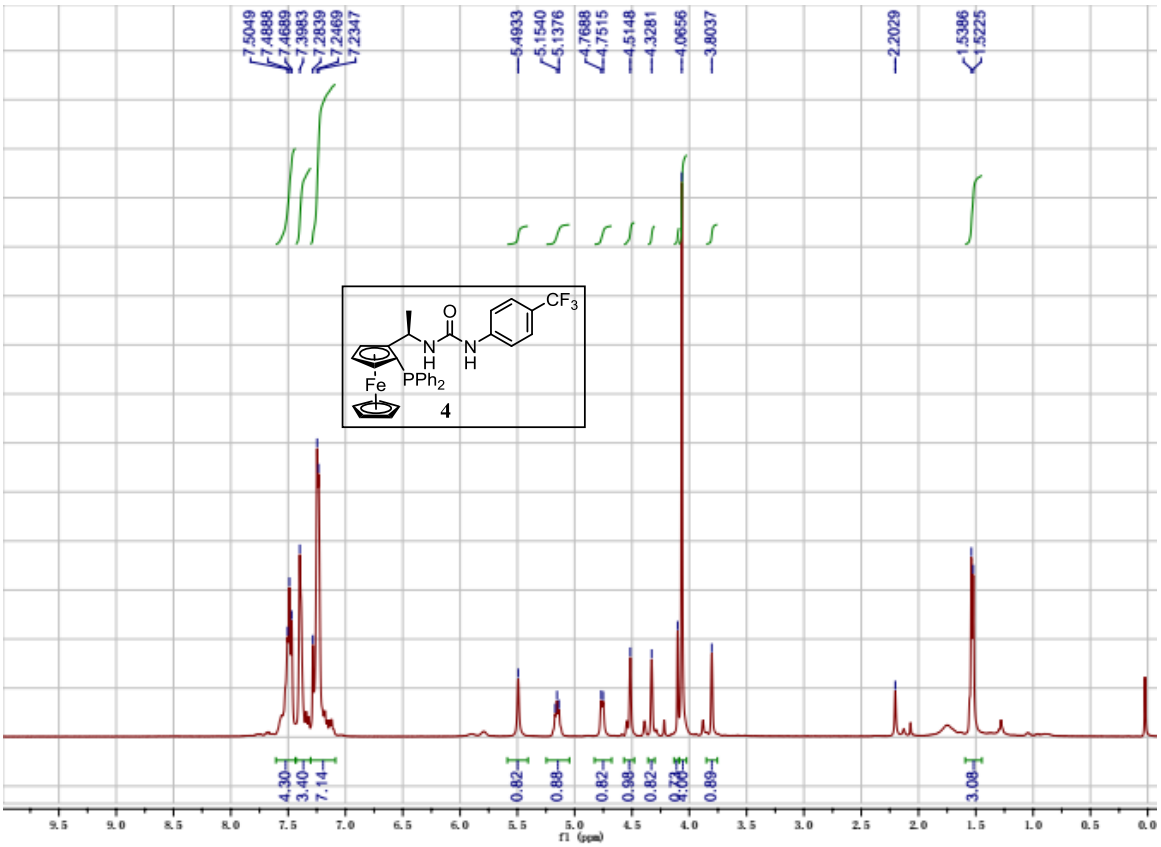
NMR spectra copies of all new compounds



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4.7688
4.7515
4.5148
4.3281
4.0656
3.8037
2.2029
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1.5225
CF3
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H
O
N
H
Fe
PPh2
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0.82
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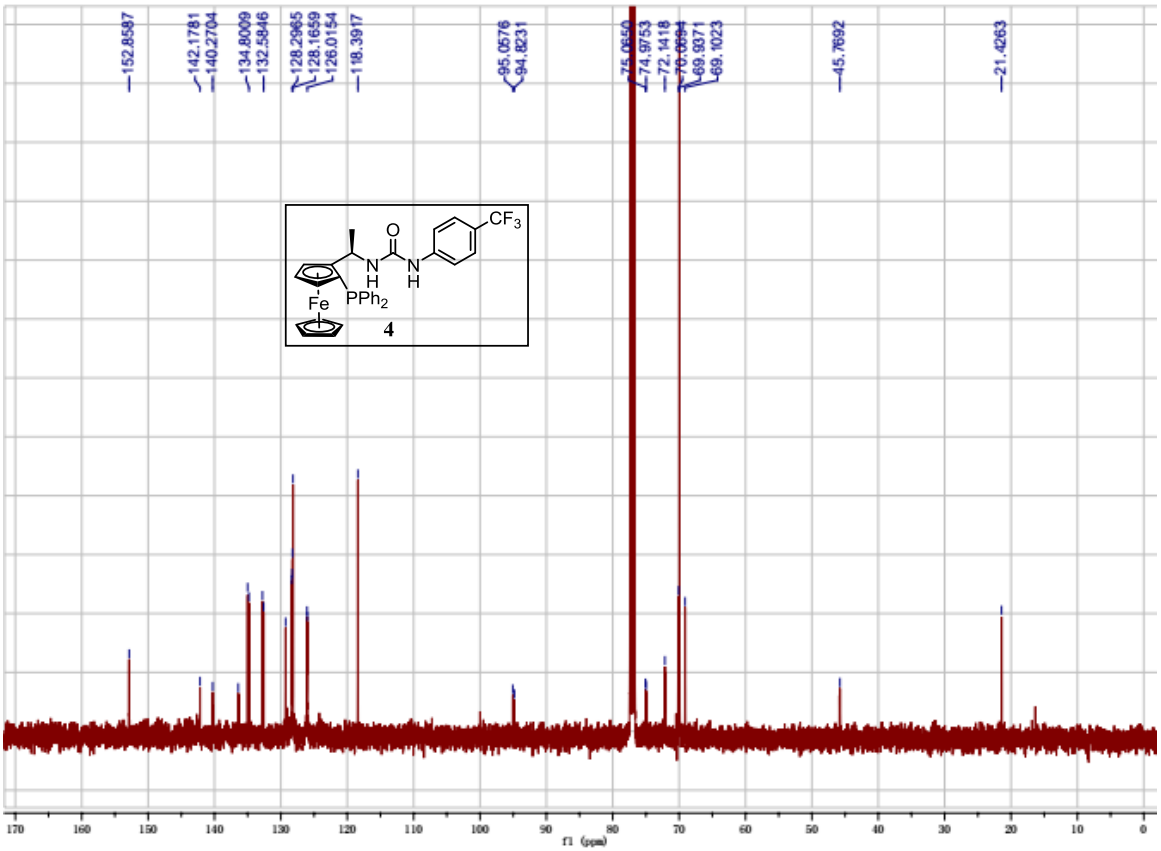
3.08
f1 (ppm)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Fe
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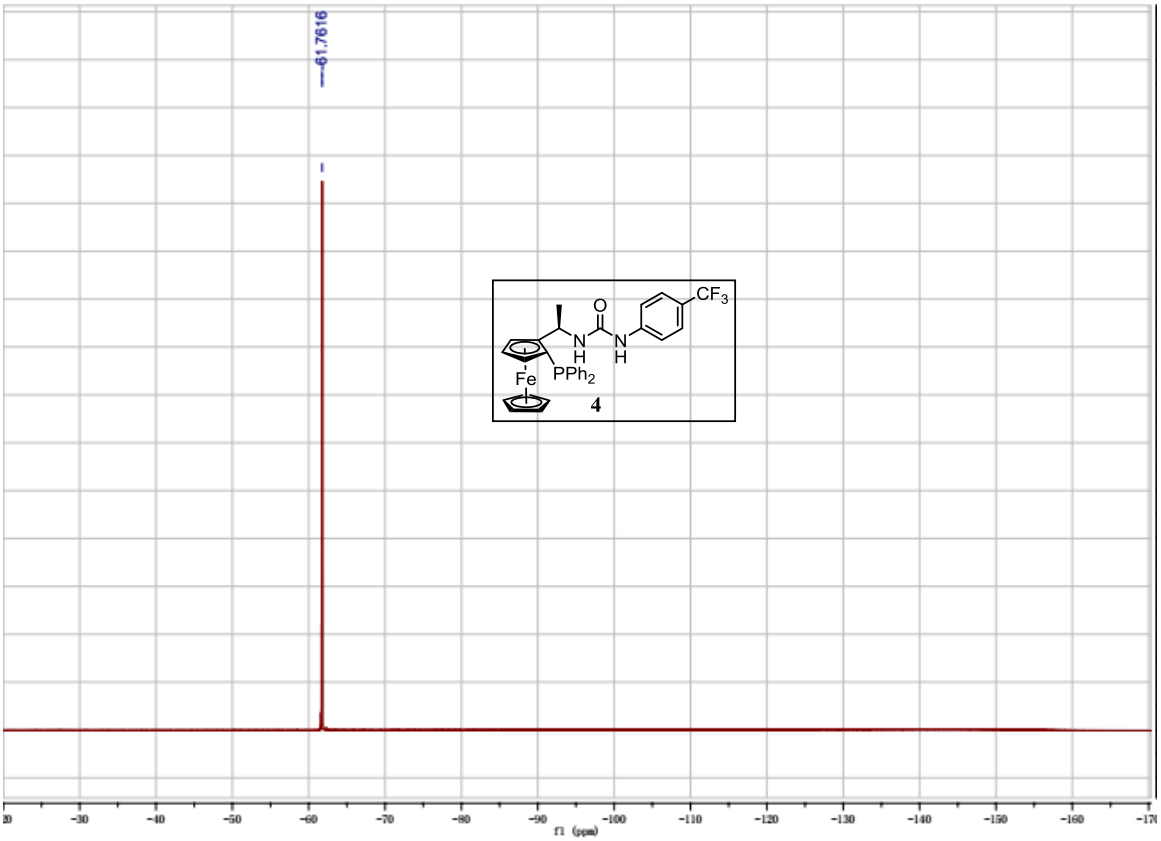
f1 (ppm)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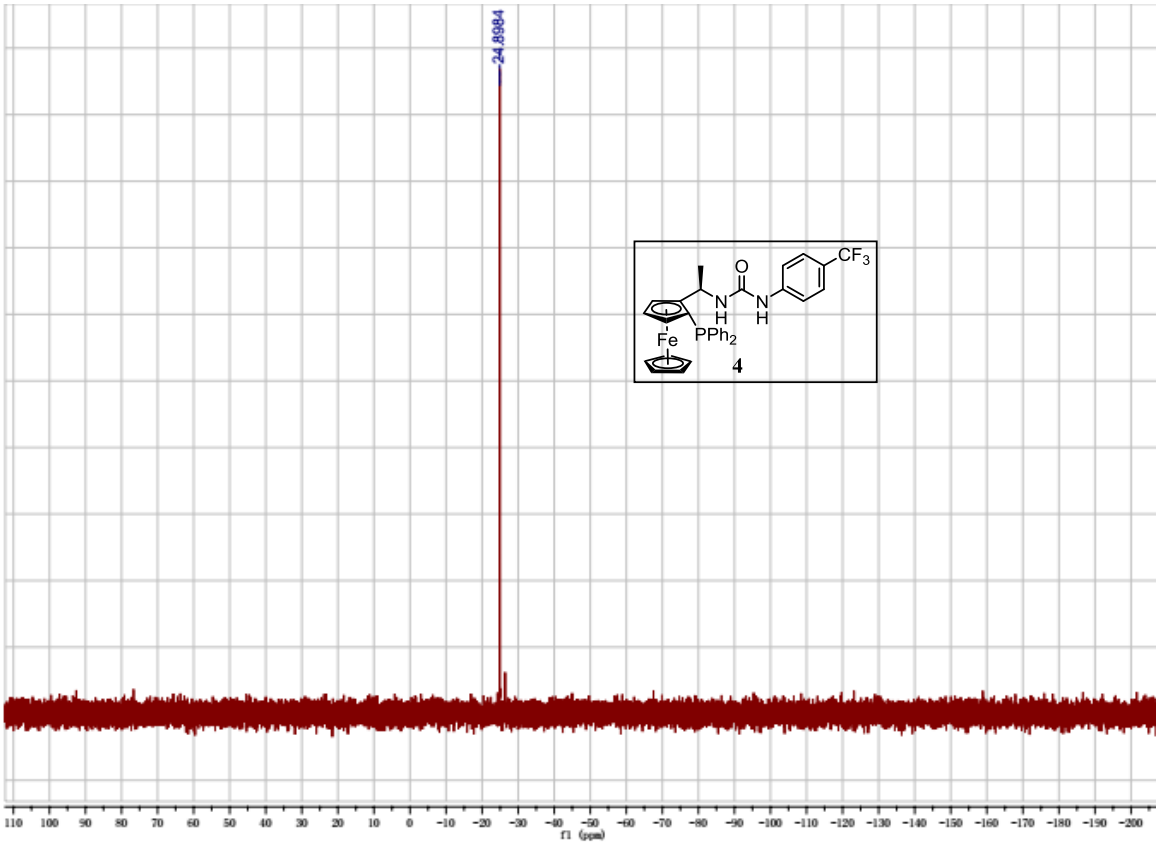
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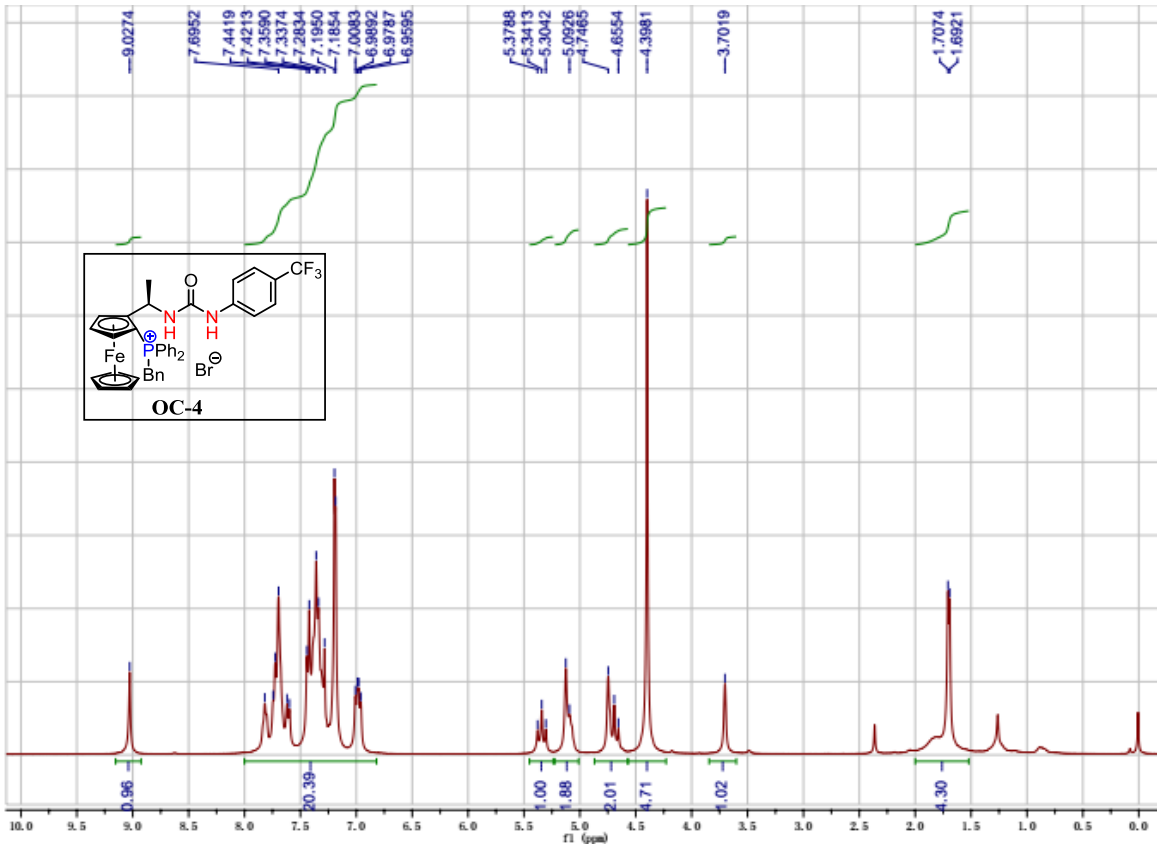
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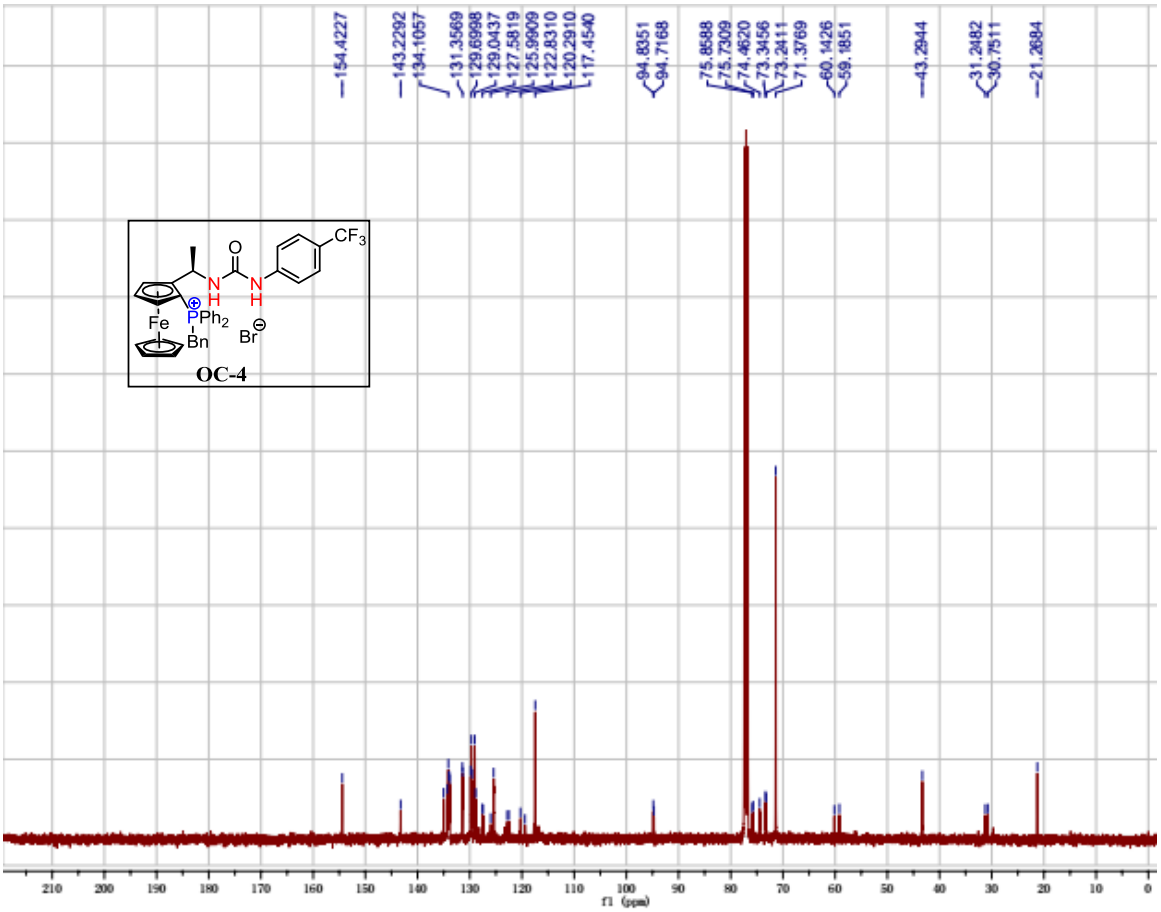
f1 (ppm)
OC-4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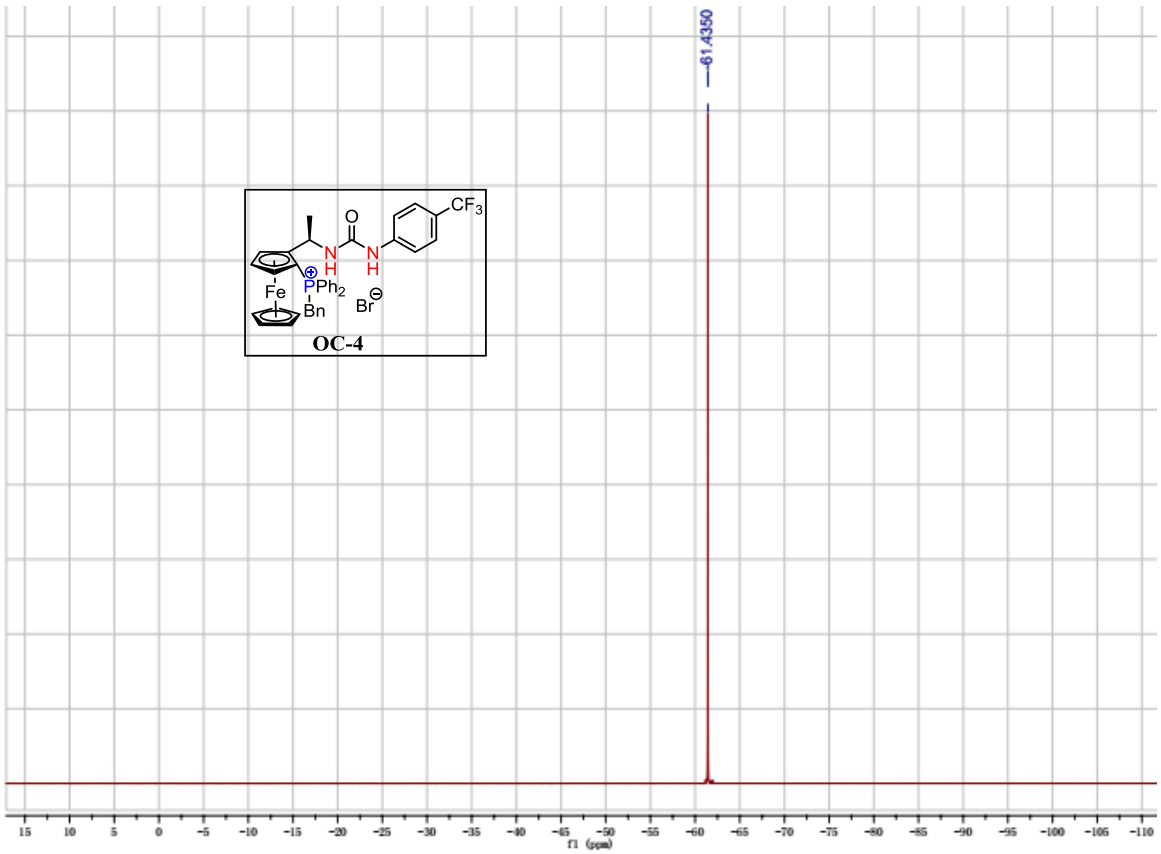
f1 (ppm)
OC-4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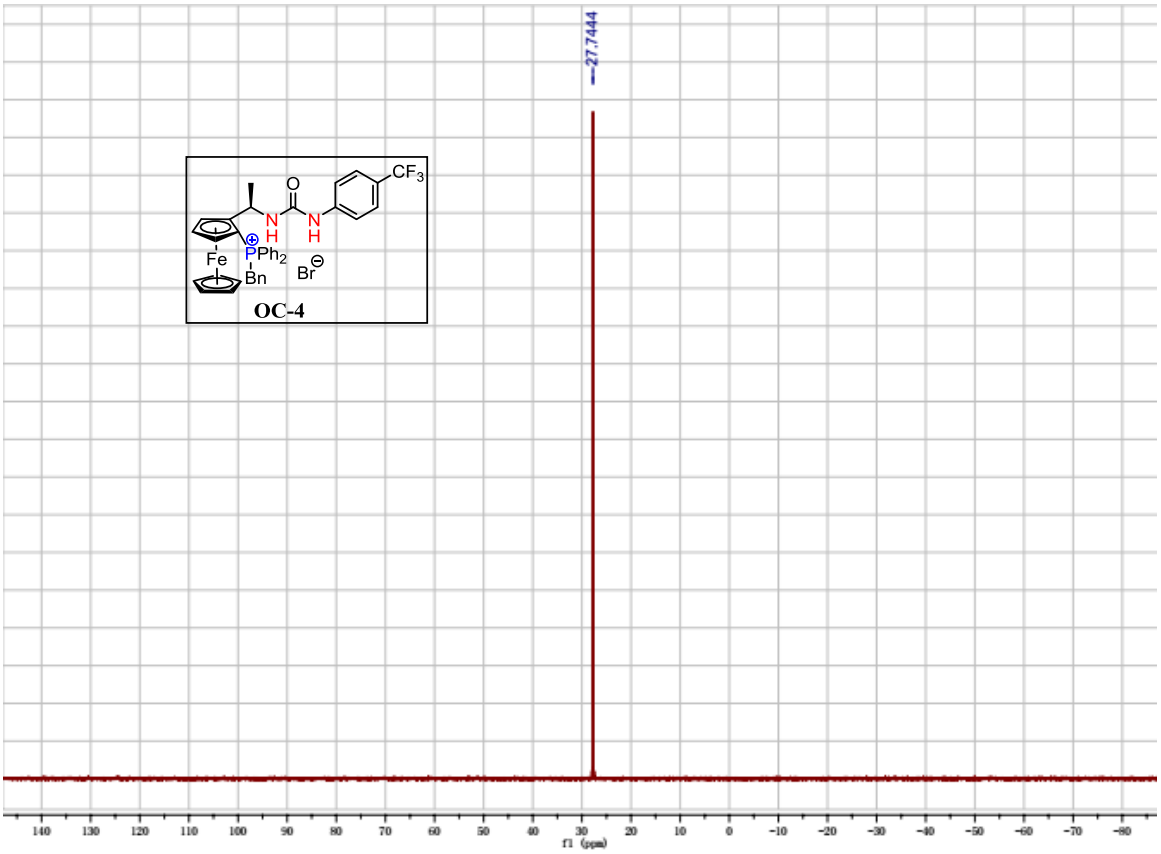
f1 (ppm)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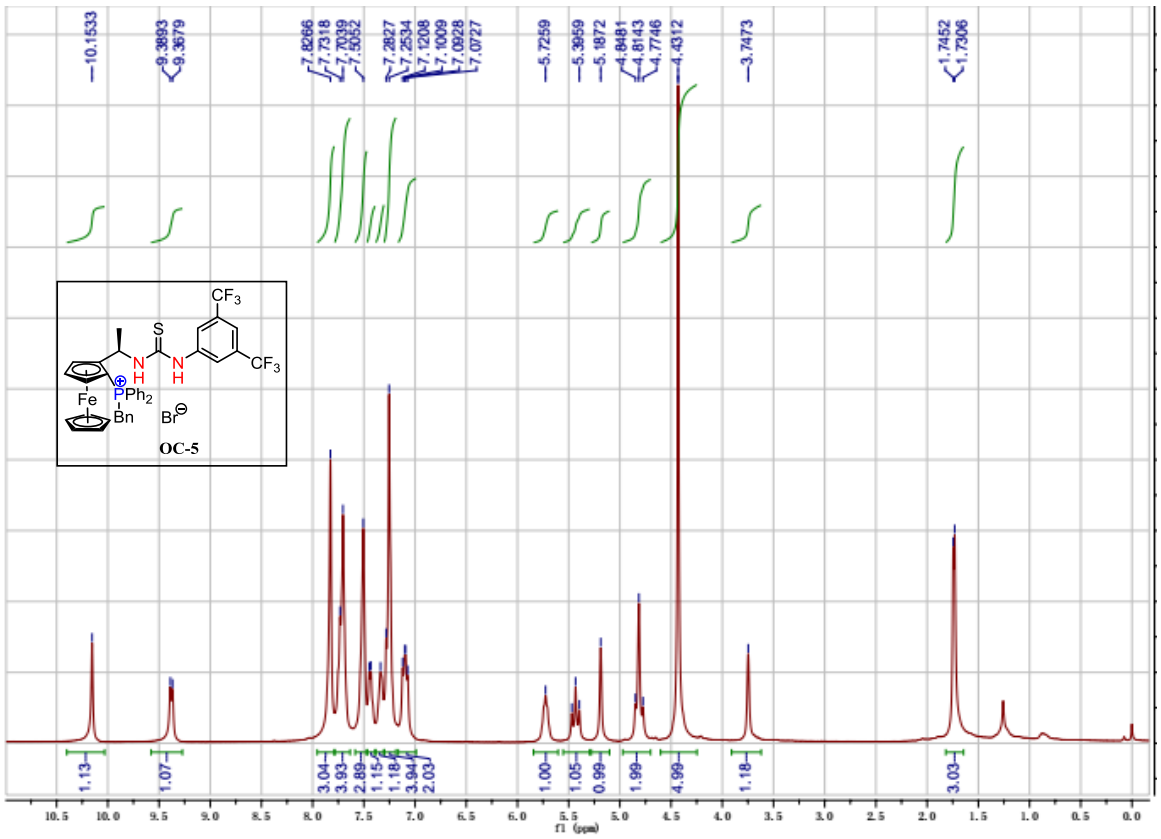
f1 (ppm)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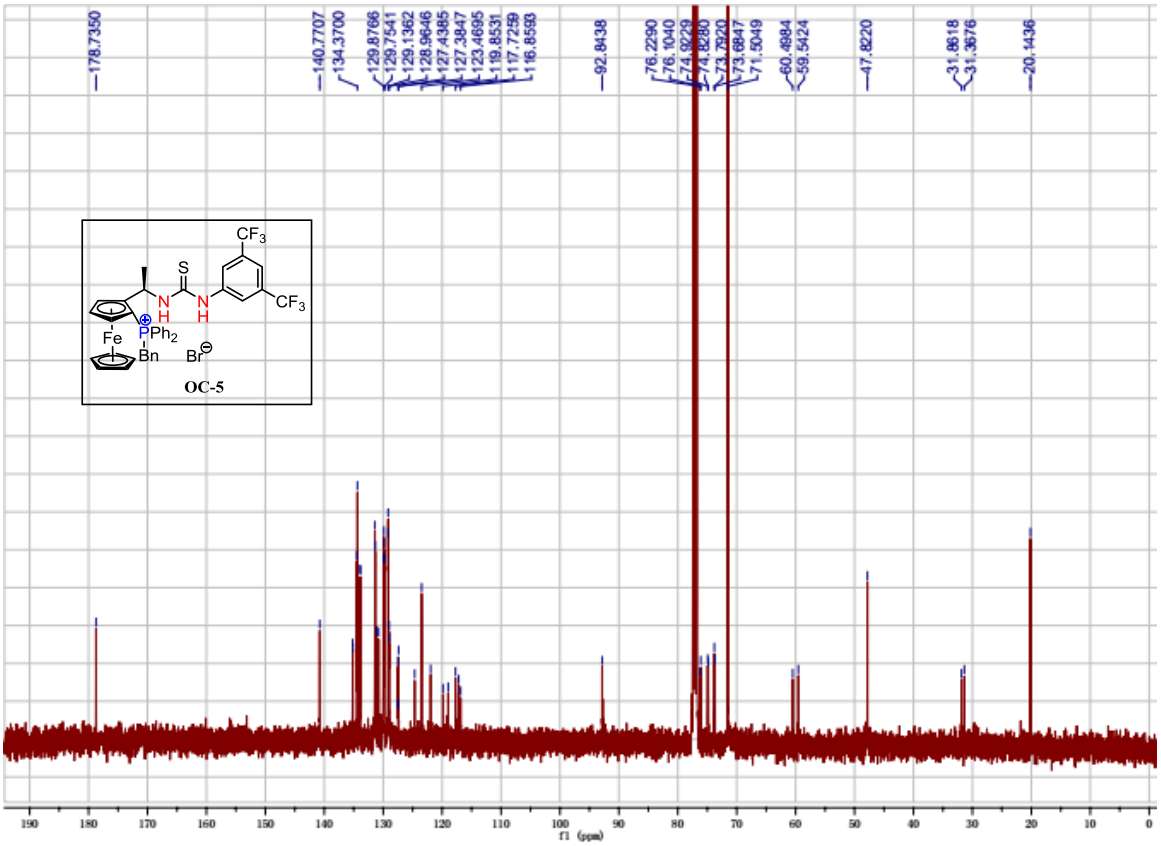
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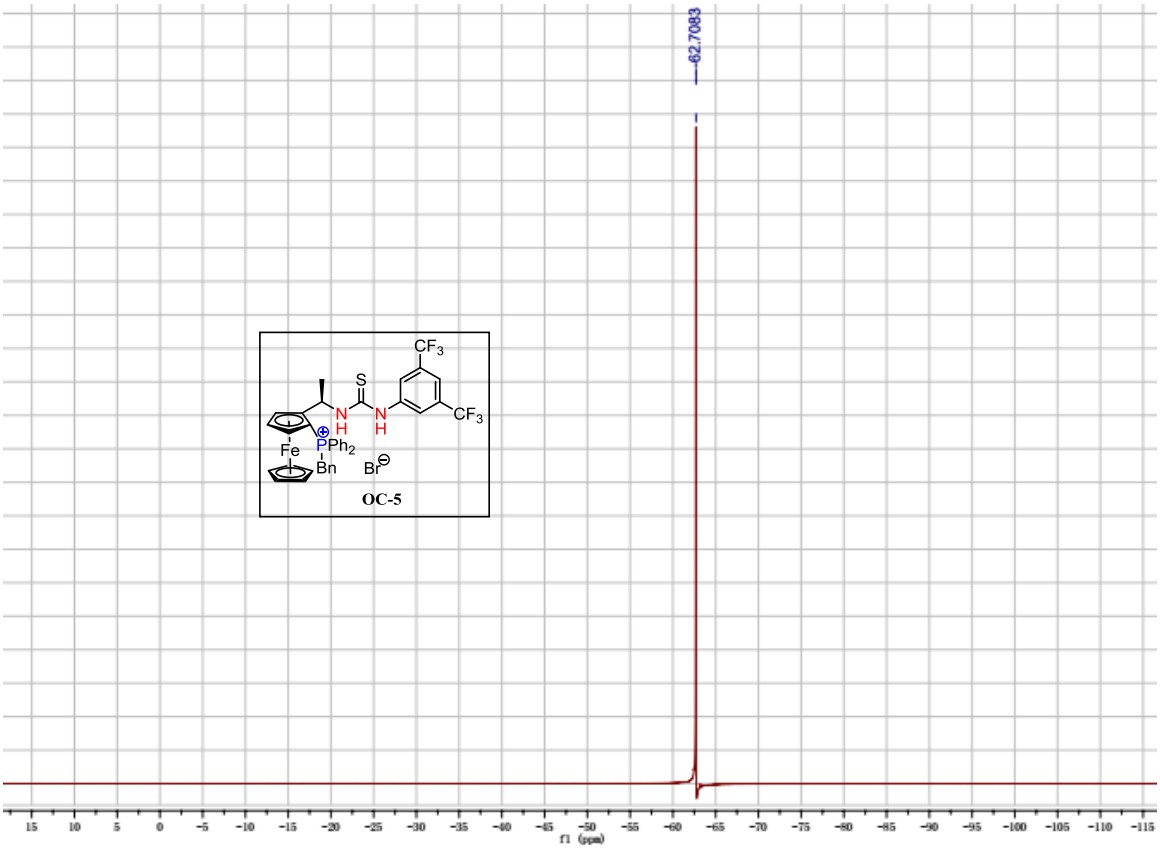
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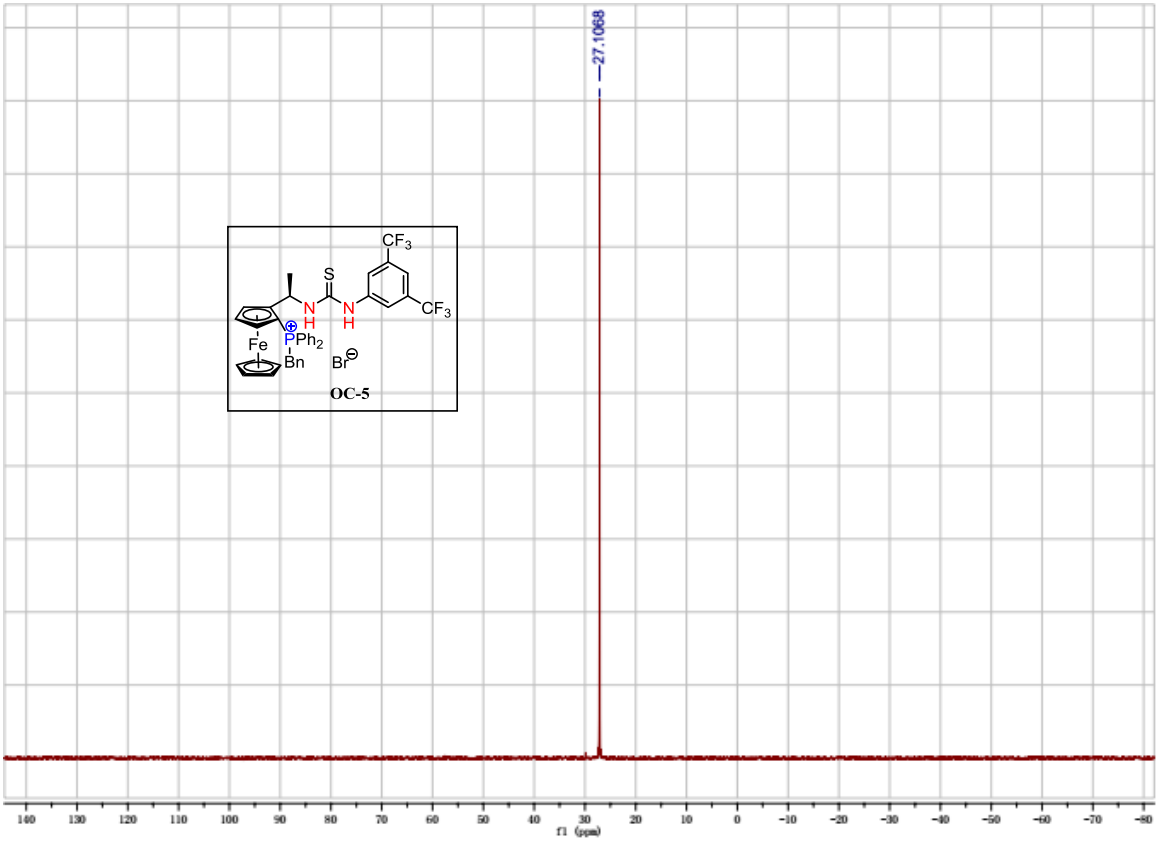
f1 (ppm)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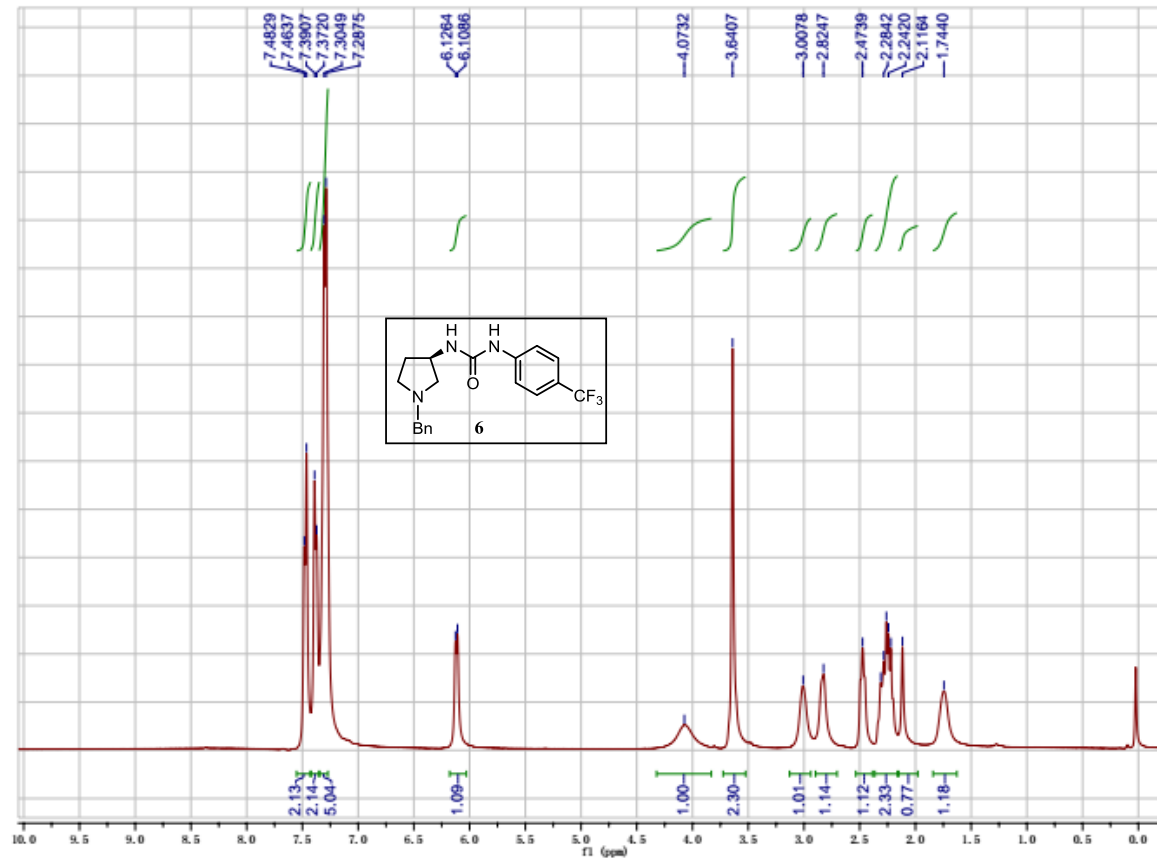
f1 (ppm)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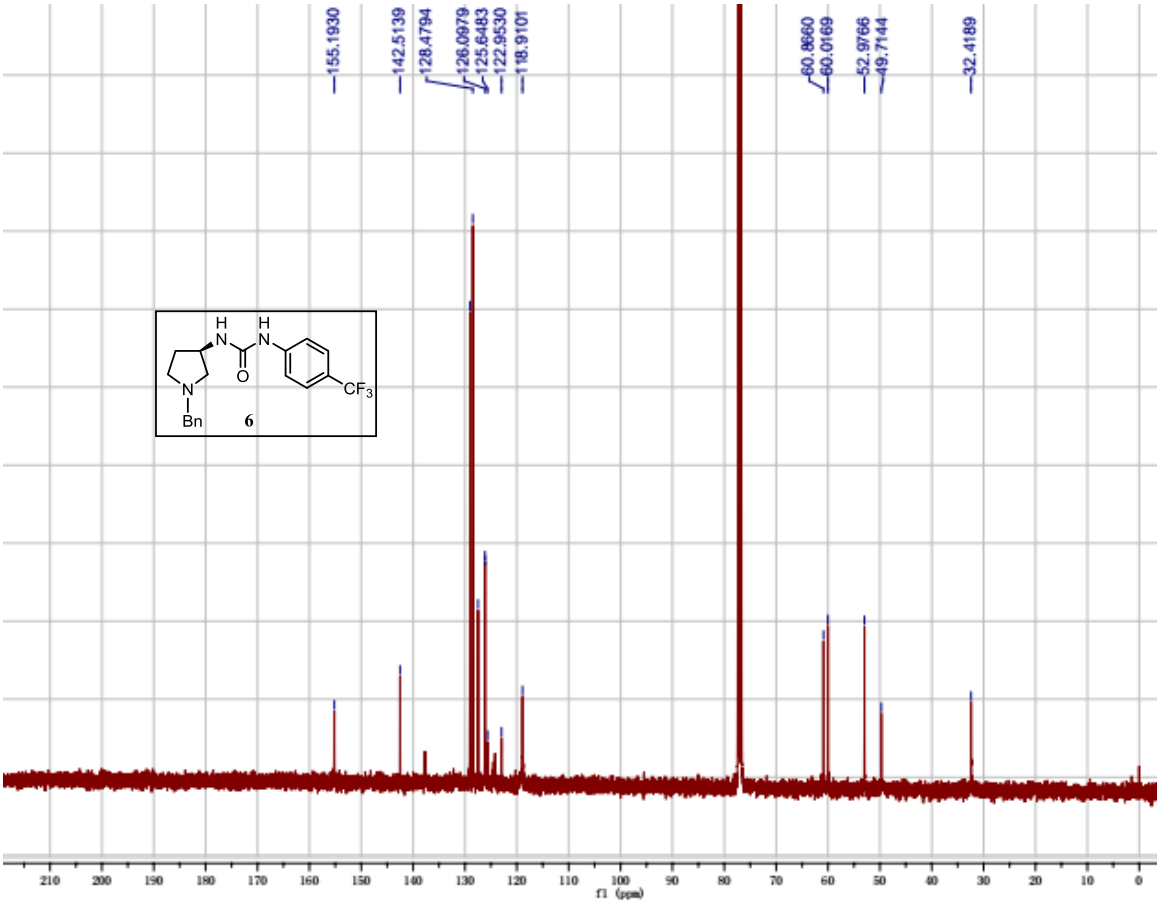
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CF3
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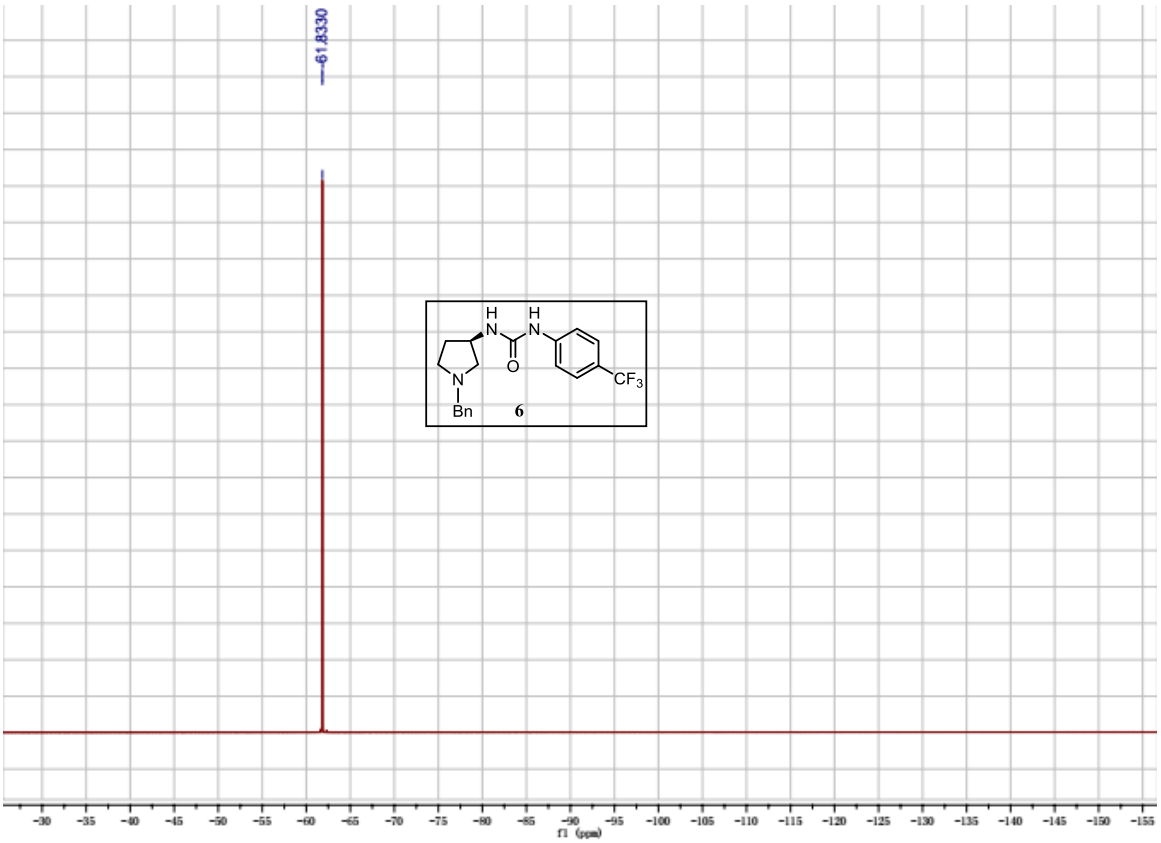
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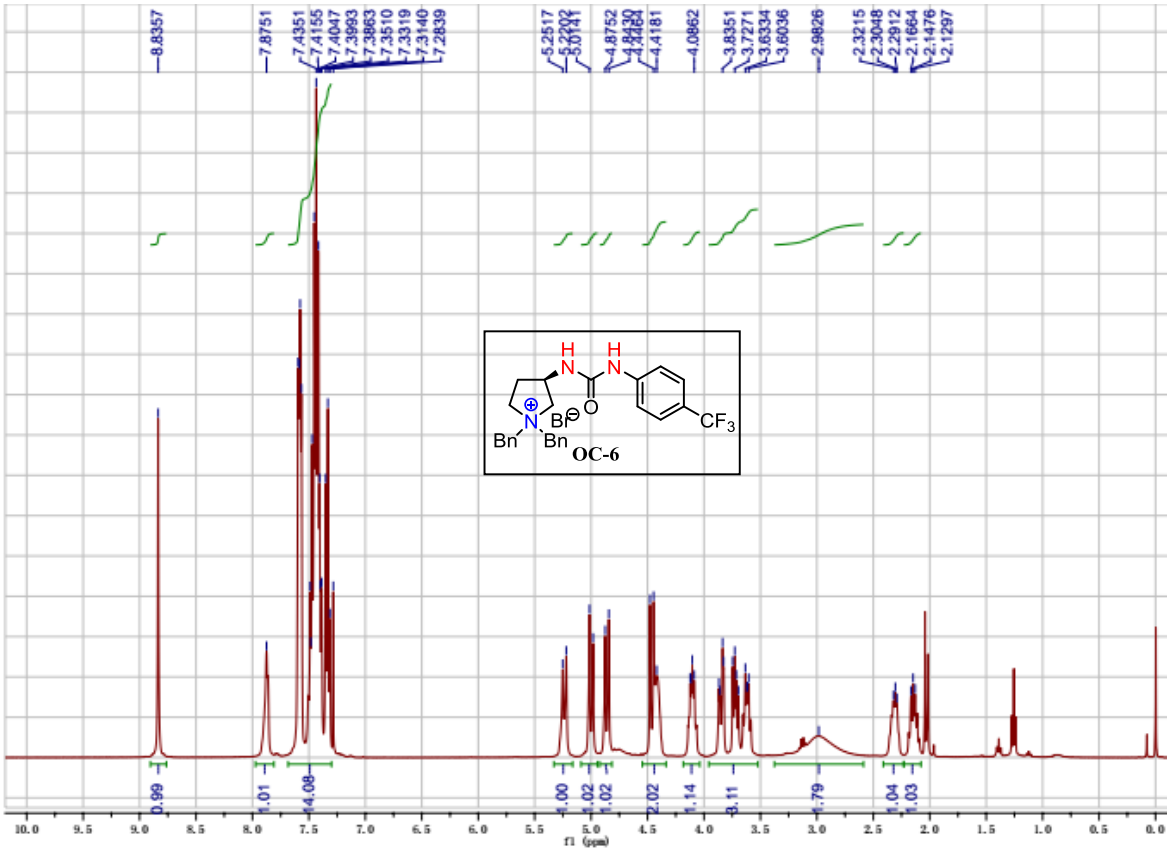
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7.2839
5.2517
5.2202
5.0141
4.8752
4.8430
4.4181
4.0862
3.8351
3.7271
3.6334
3.6036
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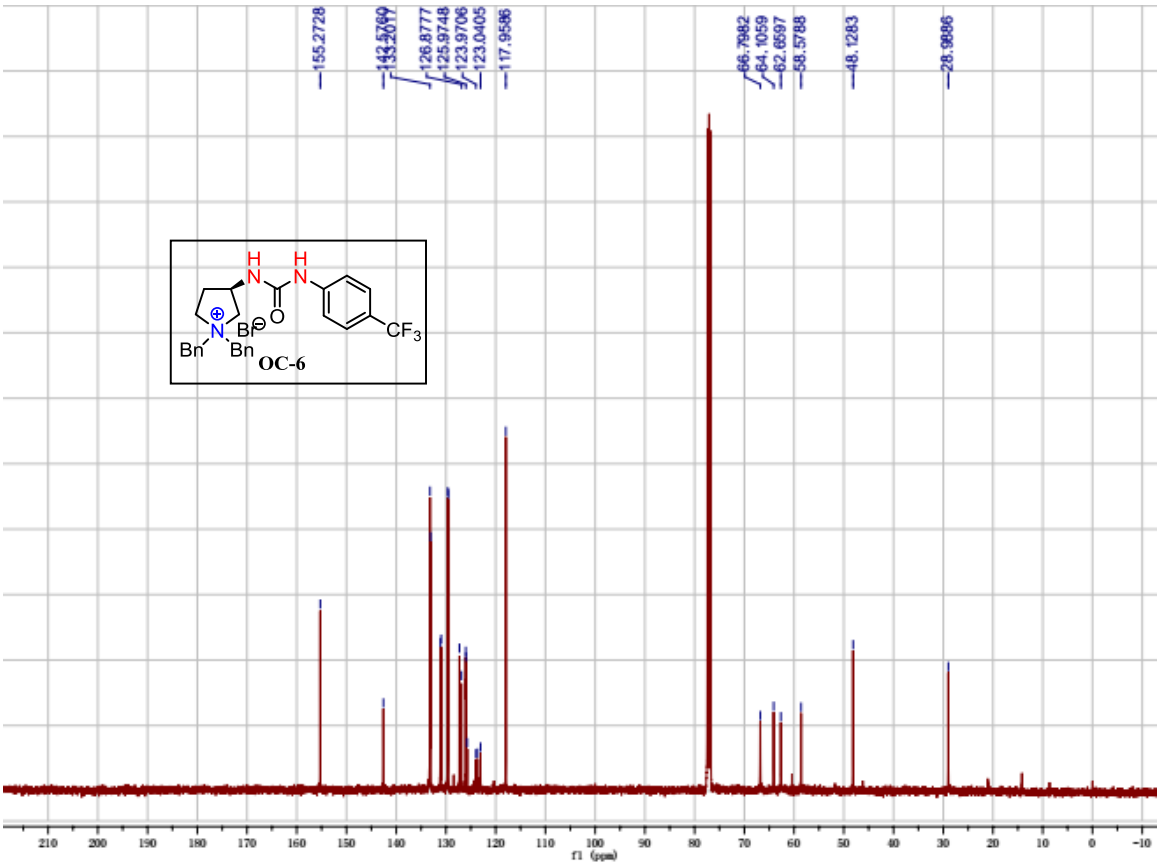
2.2912
2.1664
2.1476
2.1297
OC-6
Bn
Bn
CF3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62.6597
58.5788
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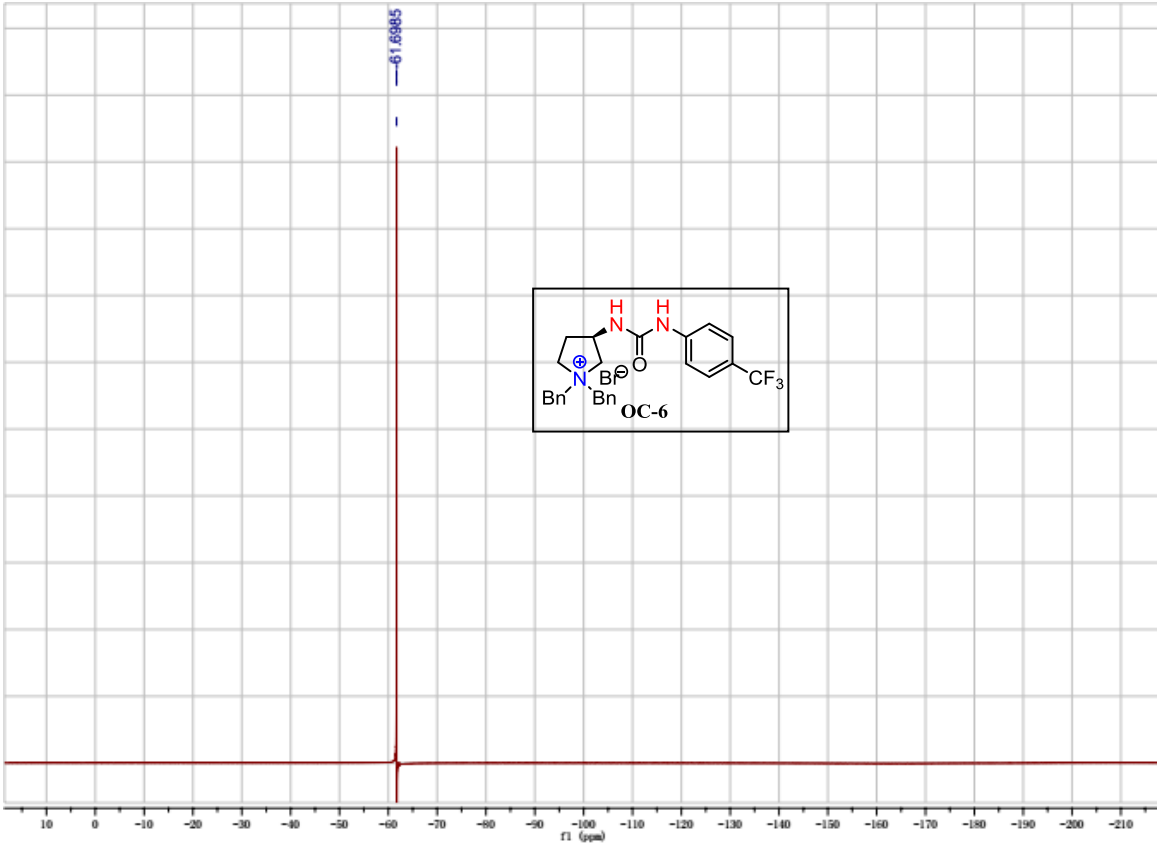
28.9886
OC-6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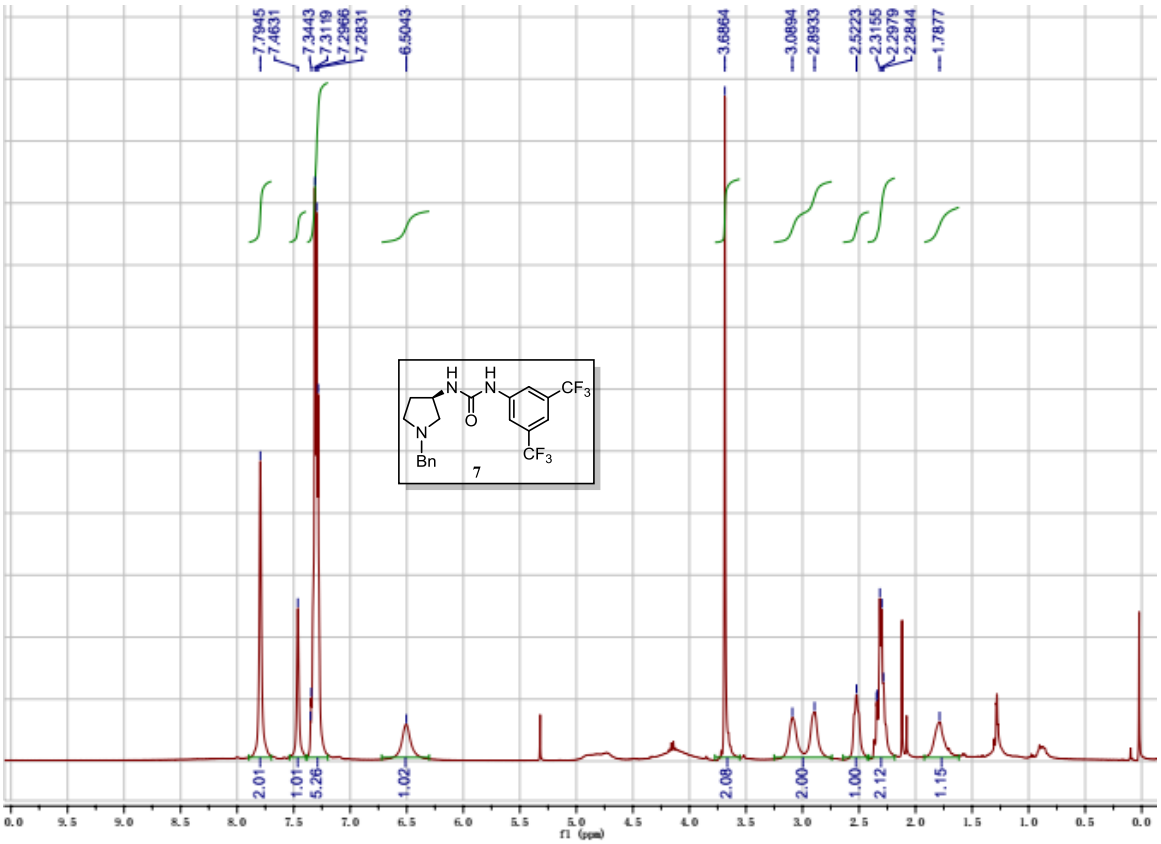
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7.2966
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6.5043
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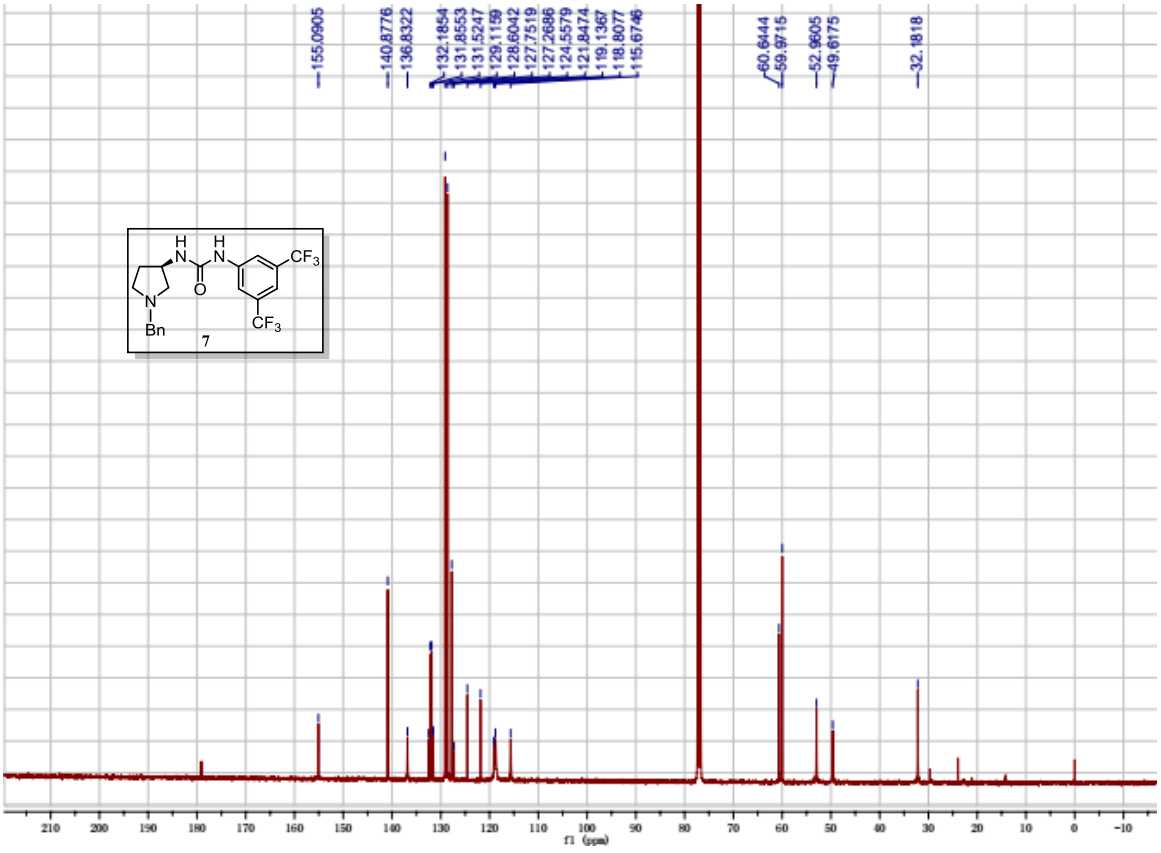
2.5223
2.3155
2.2979
2.2844
1.7877
2.01
1.01
5.26
1.02
2.08
2.00
1.00
2.12
1.15
f1 (ppm)
Bn
7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129.1159
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121.8474
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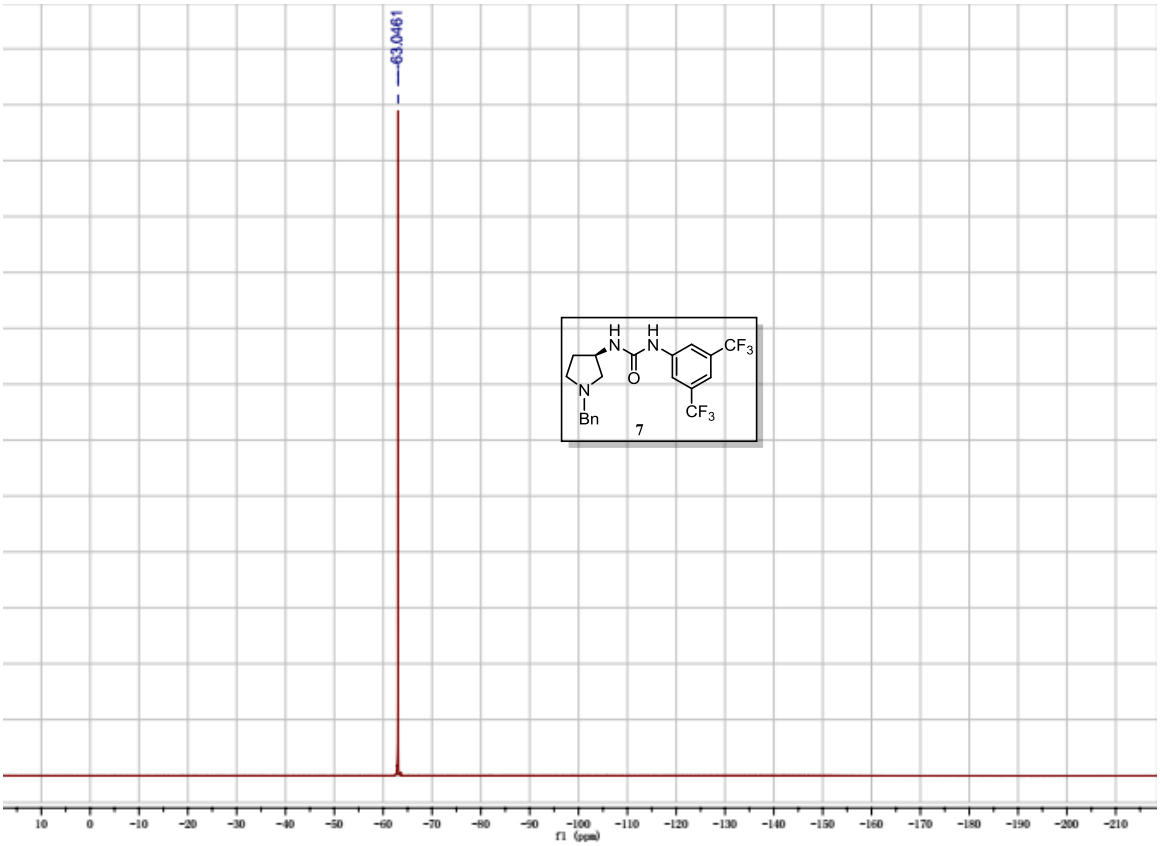
115.6746
60.6444
59.9715
52.9605
49.6175
32.1818
f1 (ppm)
Bn
7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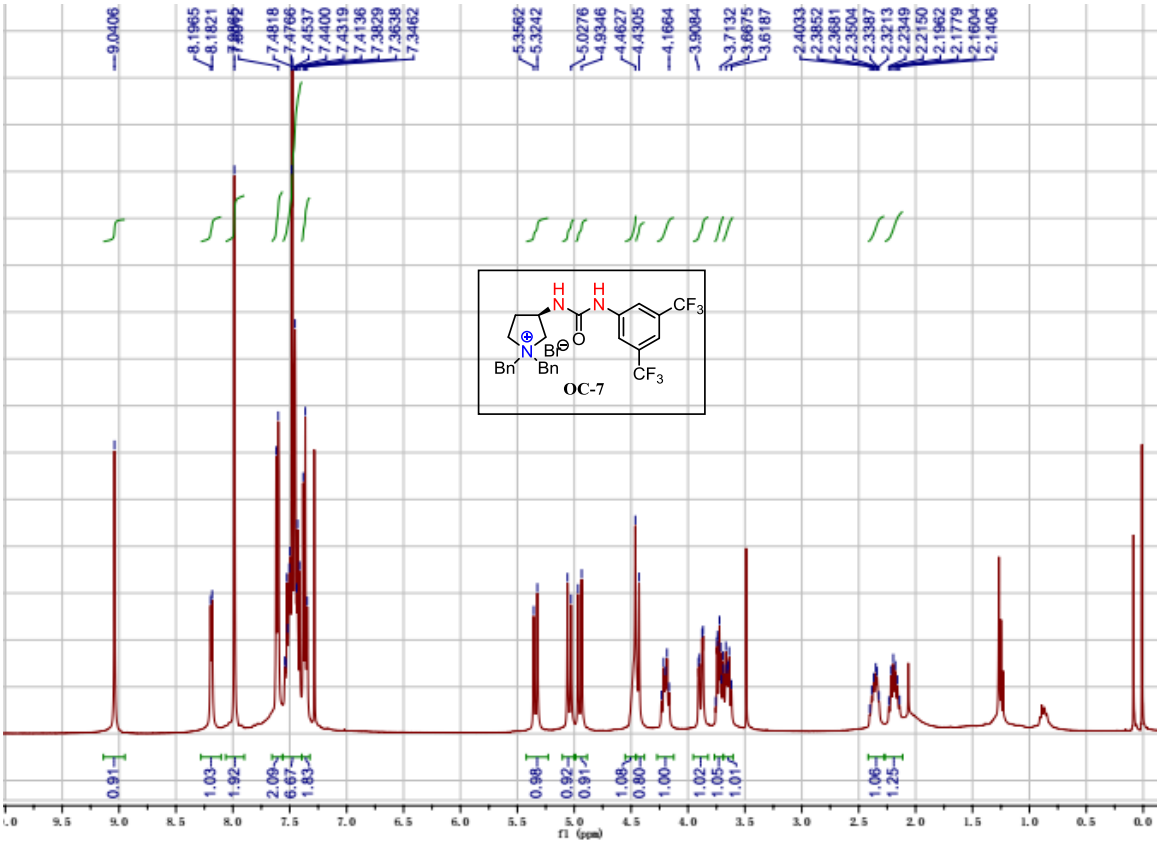
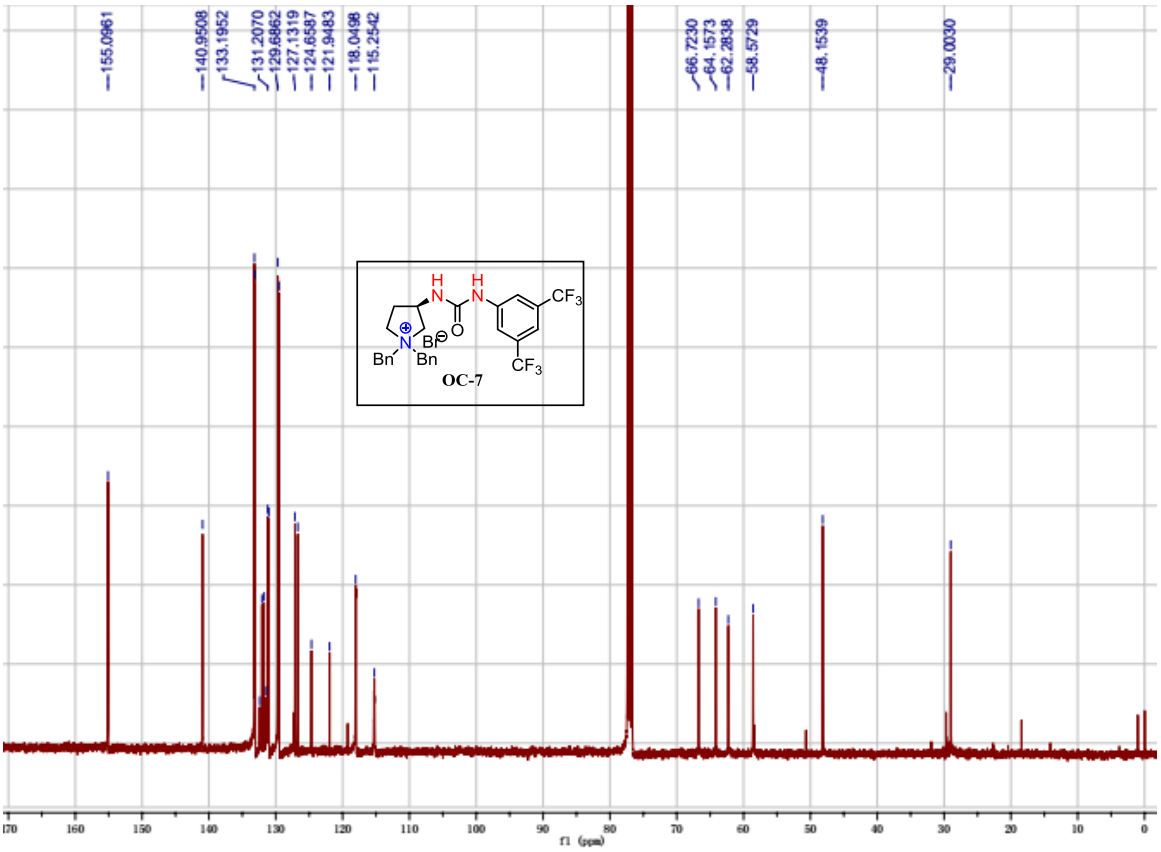

OC-7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124.6587
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62.2838
58.5729
48.1539
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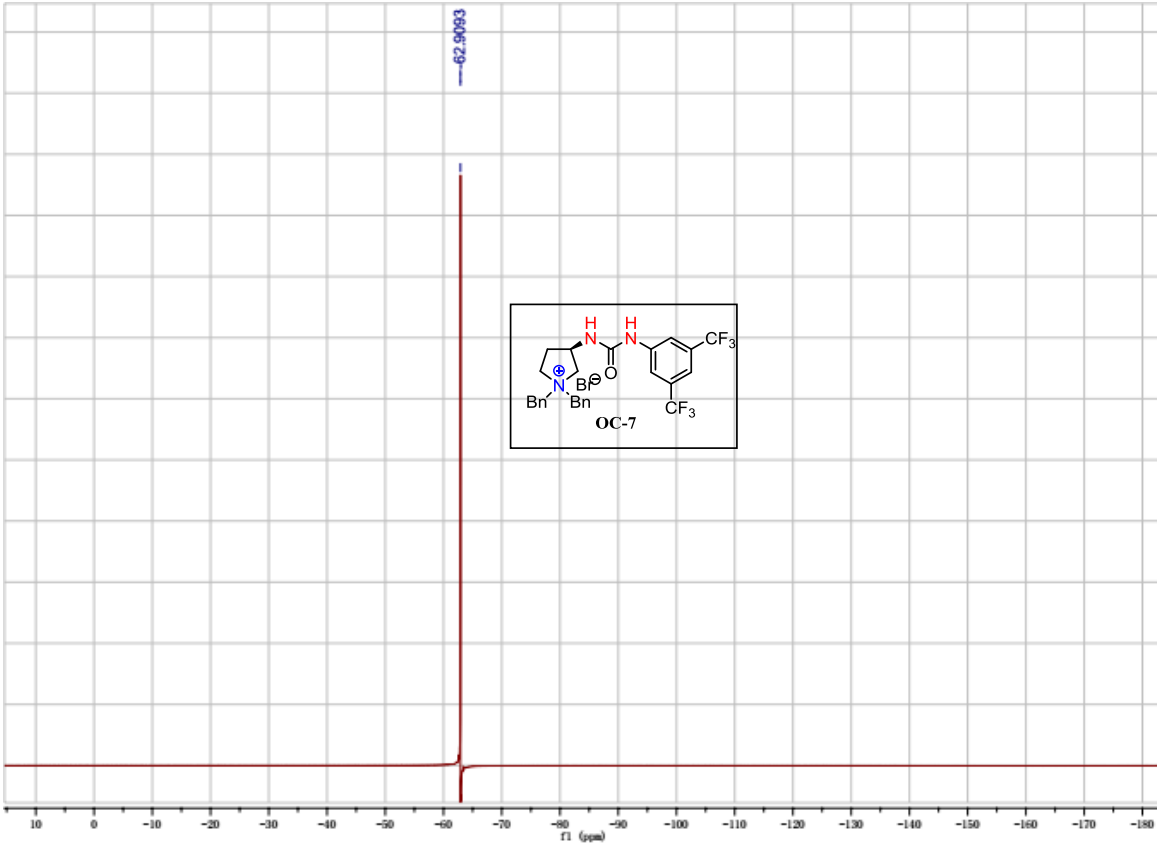
OC-7
f1 (ppm)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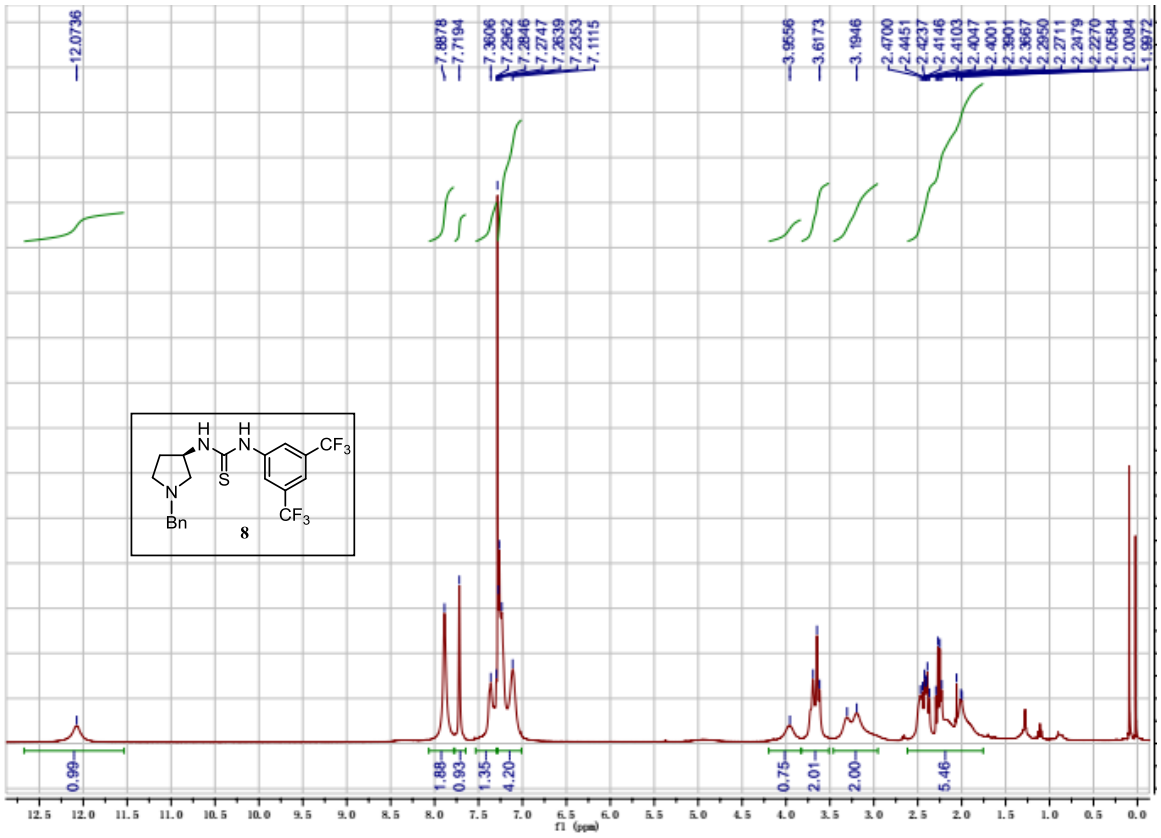
f1 (ppm)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7.3606
7.2962
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7.2747
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3.9556
3.6173
3.1946
2.4700
2.4451
2.4237
2.4146
2.4103
2.4047
2.4001
2.3901
2.3667
2.2950
2.2711
2.2479
2.2270
2.0584
2.0094
1.9872
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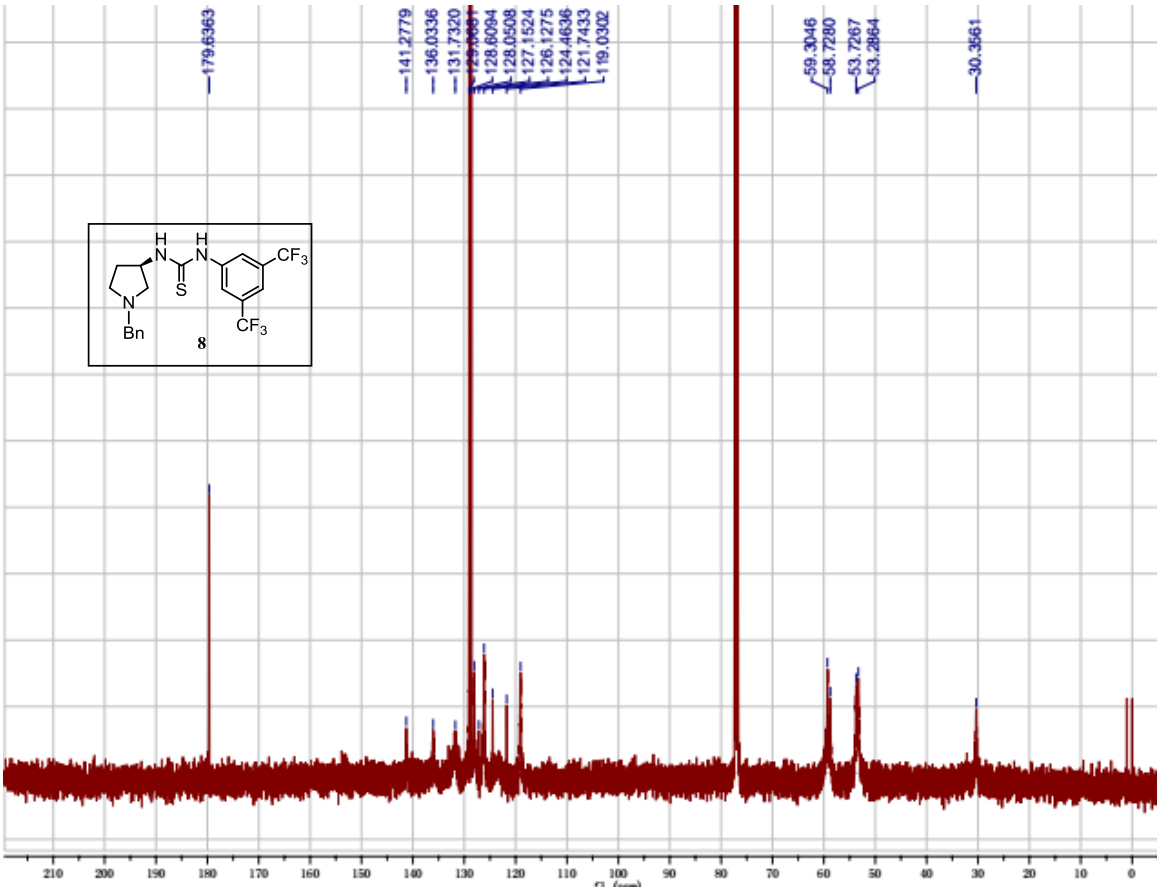
1.88
0.93
1.35
4.20
0.75
2.01
2.00
5.46
f1 (ppm)
8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141.2779
136.0336
131.7320
128.6094
128.0508
127.1524
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121.7433
119.0302
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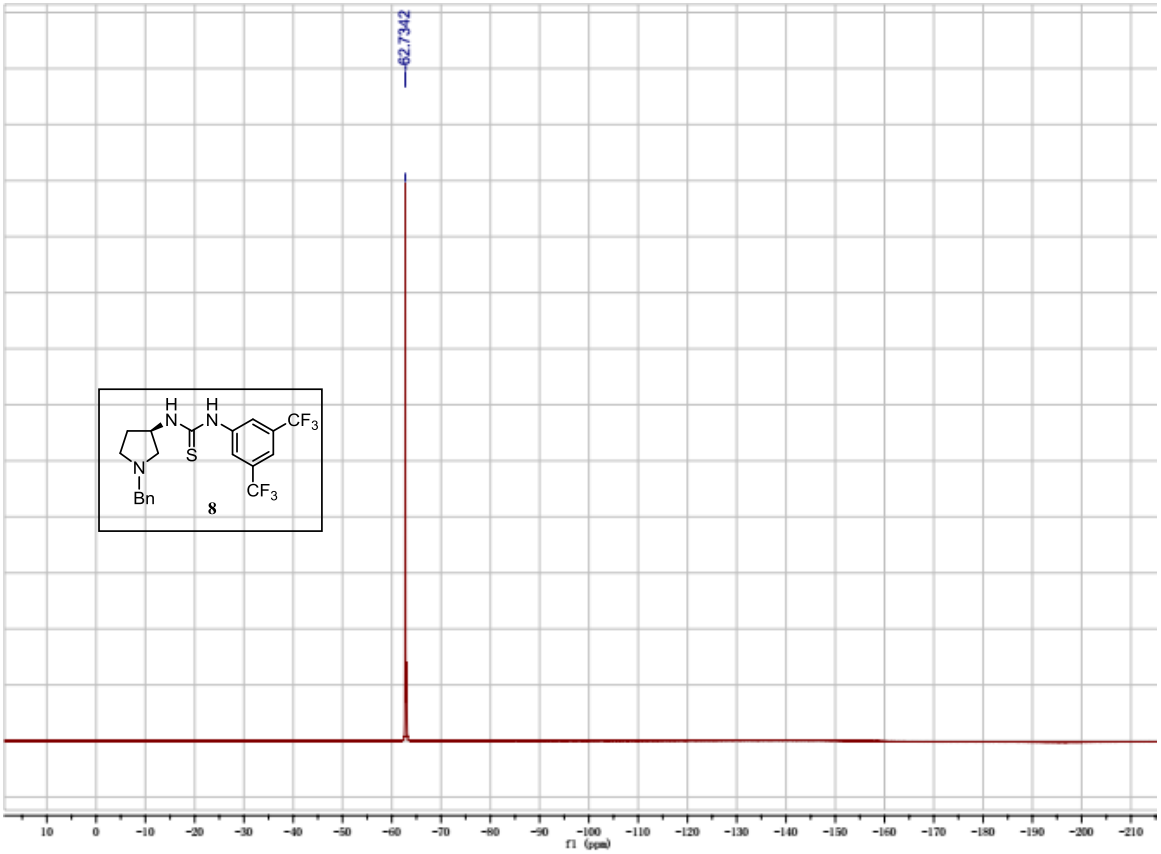
58.7280
53.7267
53.2864
30.3661
8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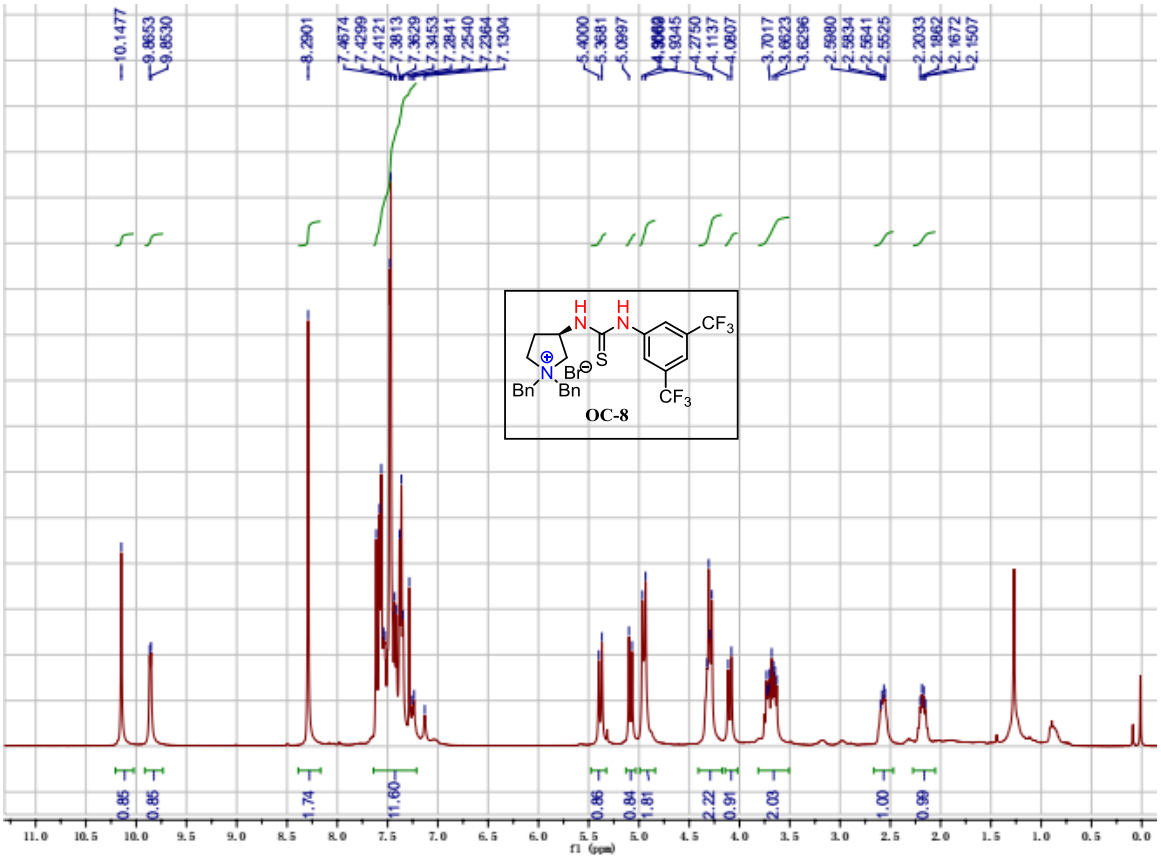
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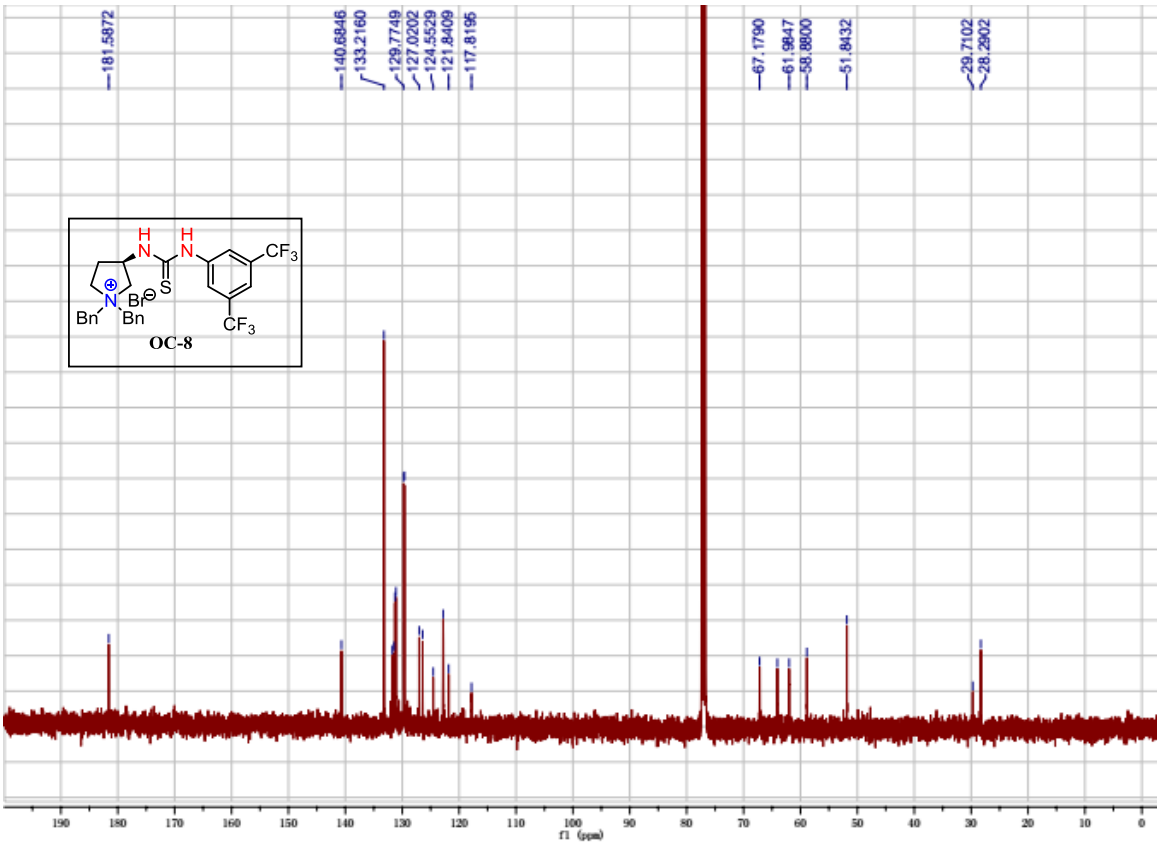
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127.0202
124.5529
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61.9847
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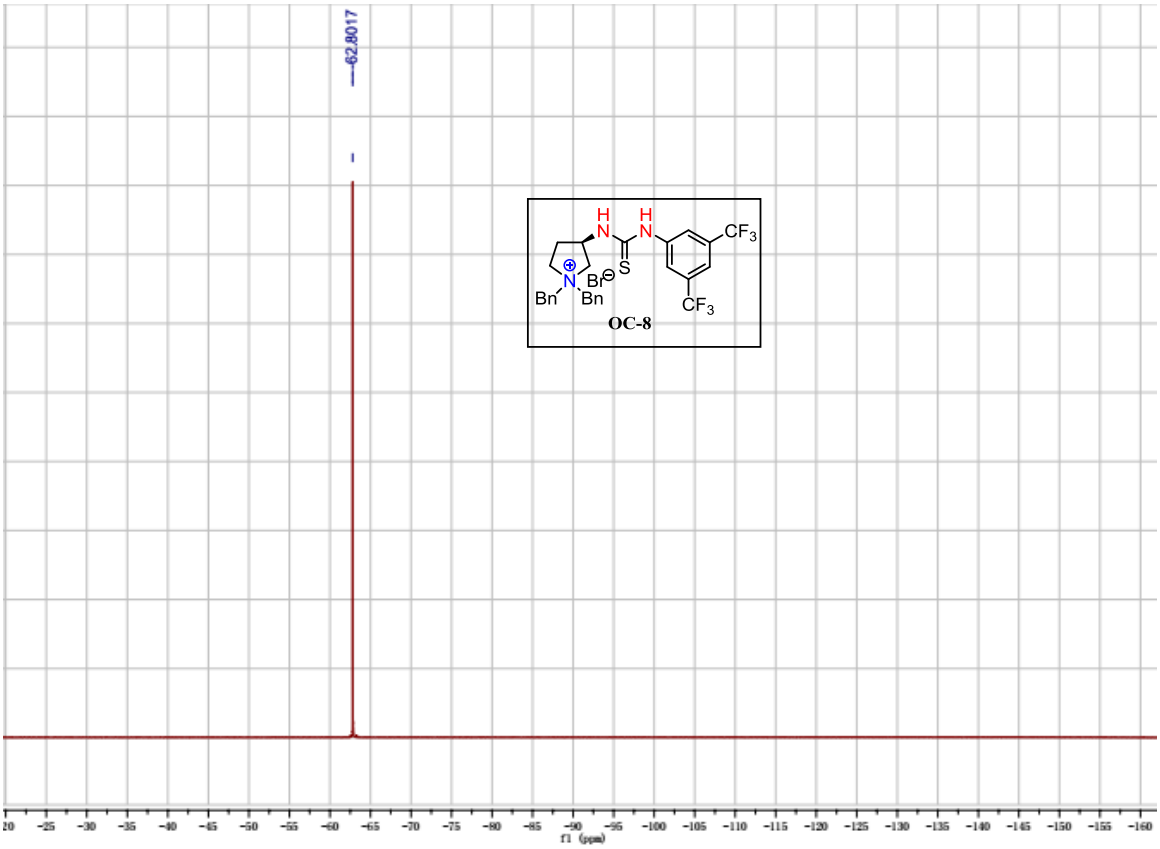
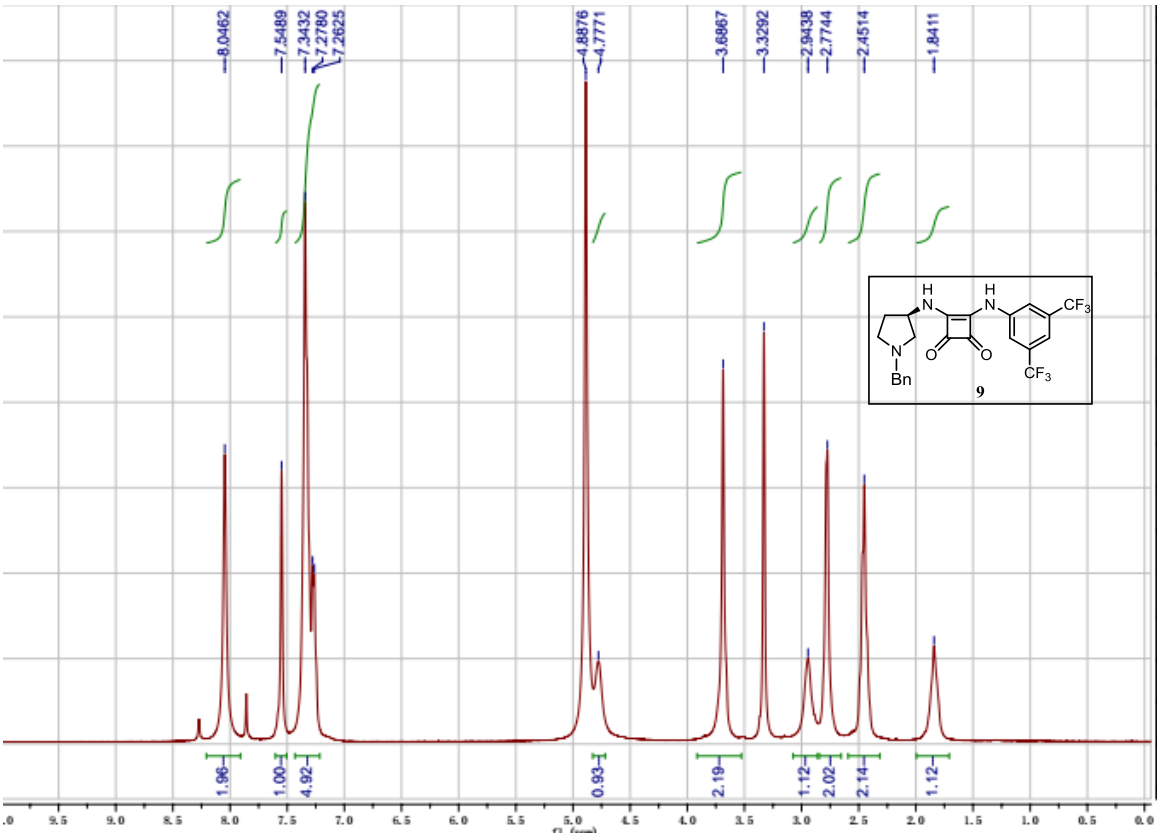
51.8432
29.7102
28.2902
OC-8
f1 (ppm)


62.8017
OC-8
f1 (ppm)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3.3292
2.9438
2.7744
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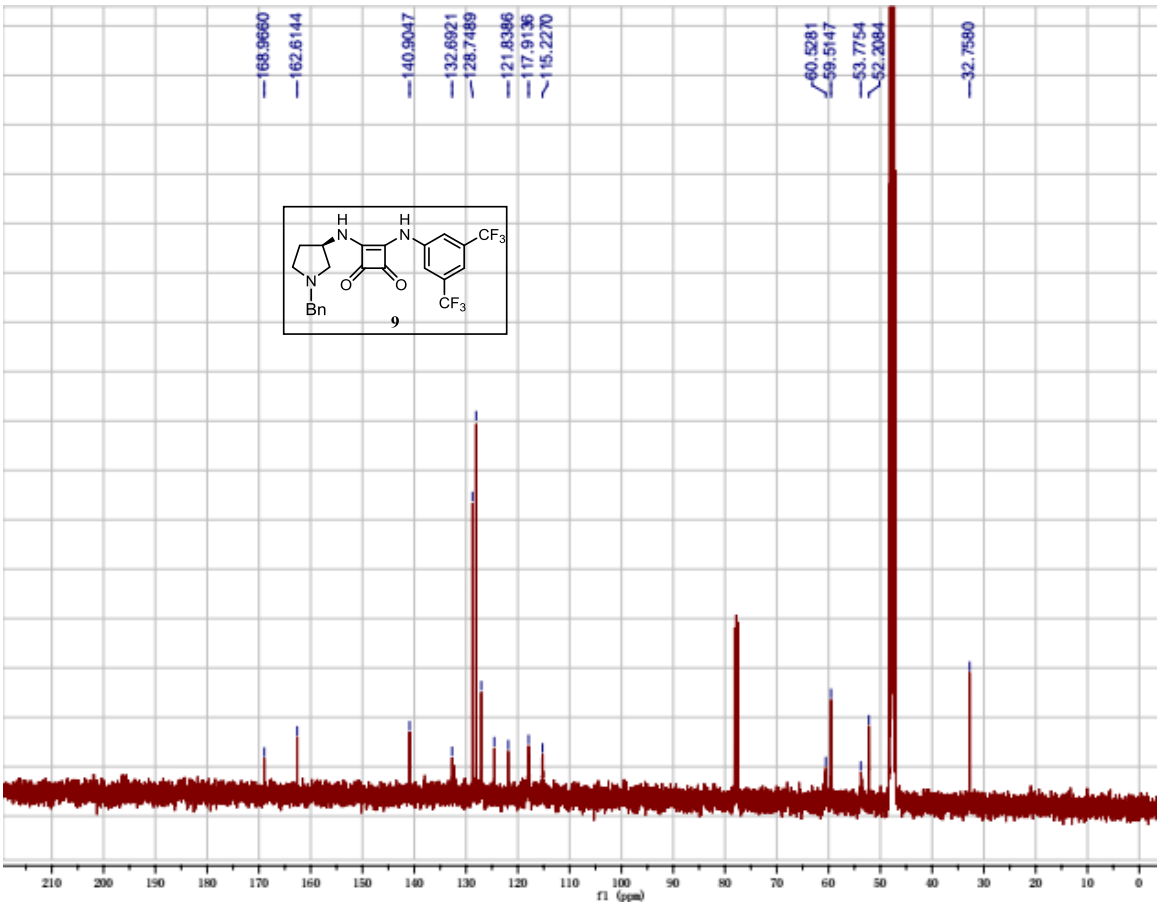
1.96
1.00
4.92
0.93
2.19
1.12
2.02
2.14
1.12
9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140.9047
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115.2270
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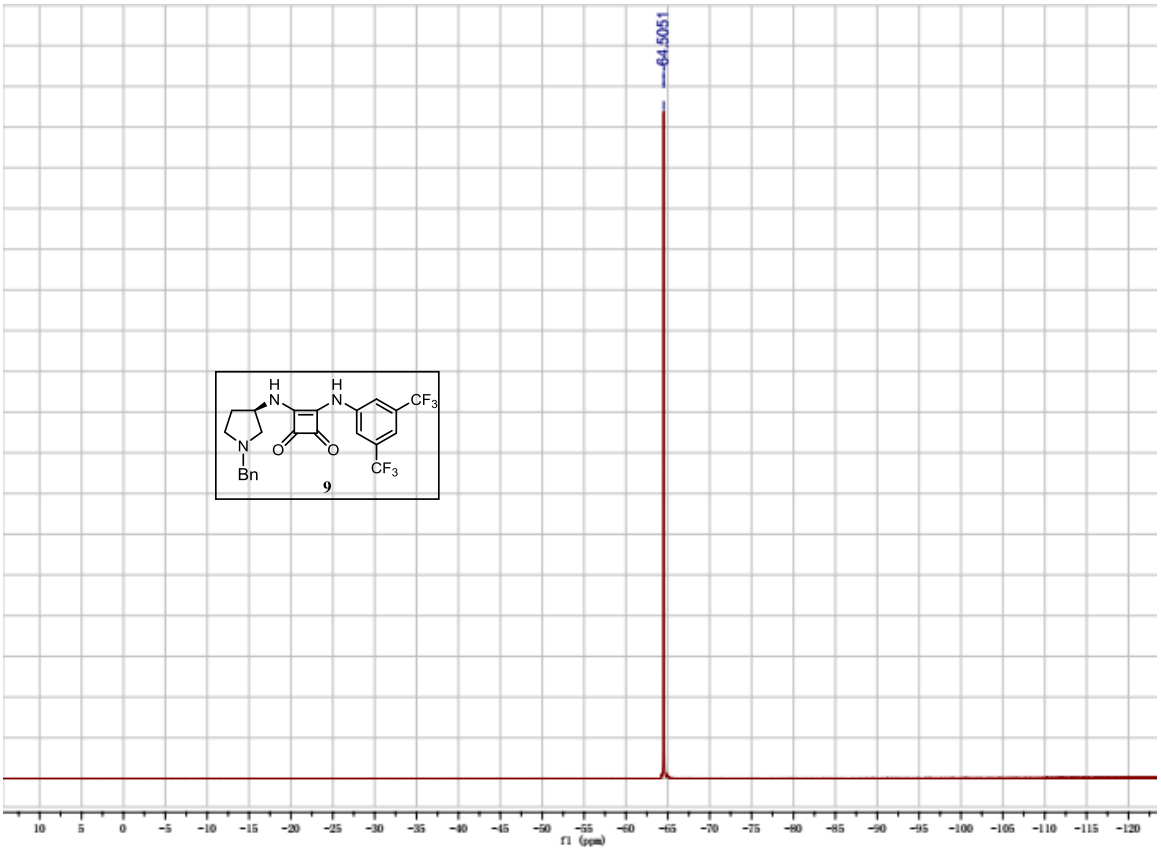
52.2084
32.7580
9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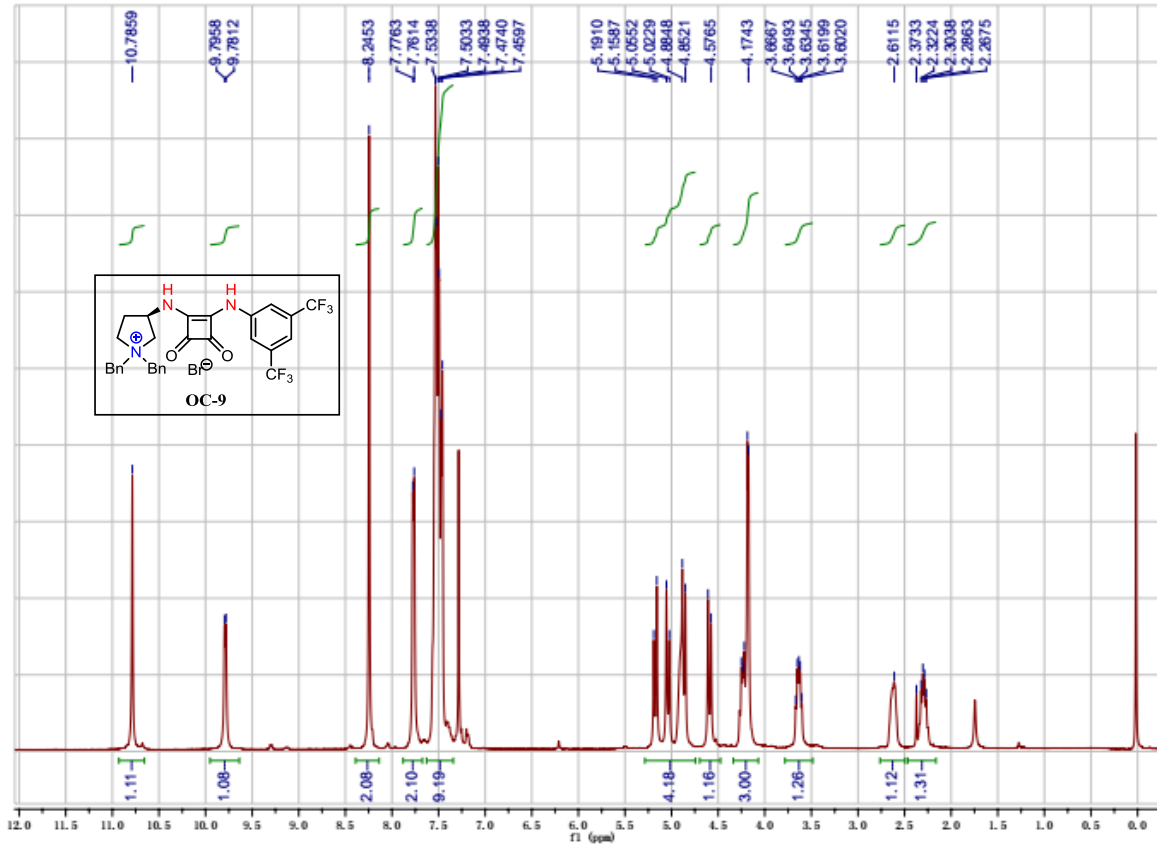
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7.4938
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7.4597
5.1910
5.1587
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3.6667
3.6493
3.6345
3.6199
3.6020
2.6115
2.3733
2.3224
2.3038
2.2863
2.2675
1.11
1.08
2.08
2.10
9.19
4.18
1.16
3.00
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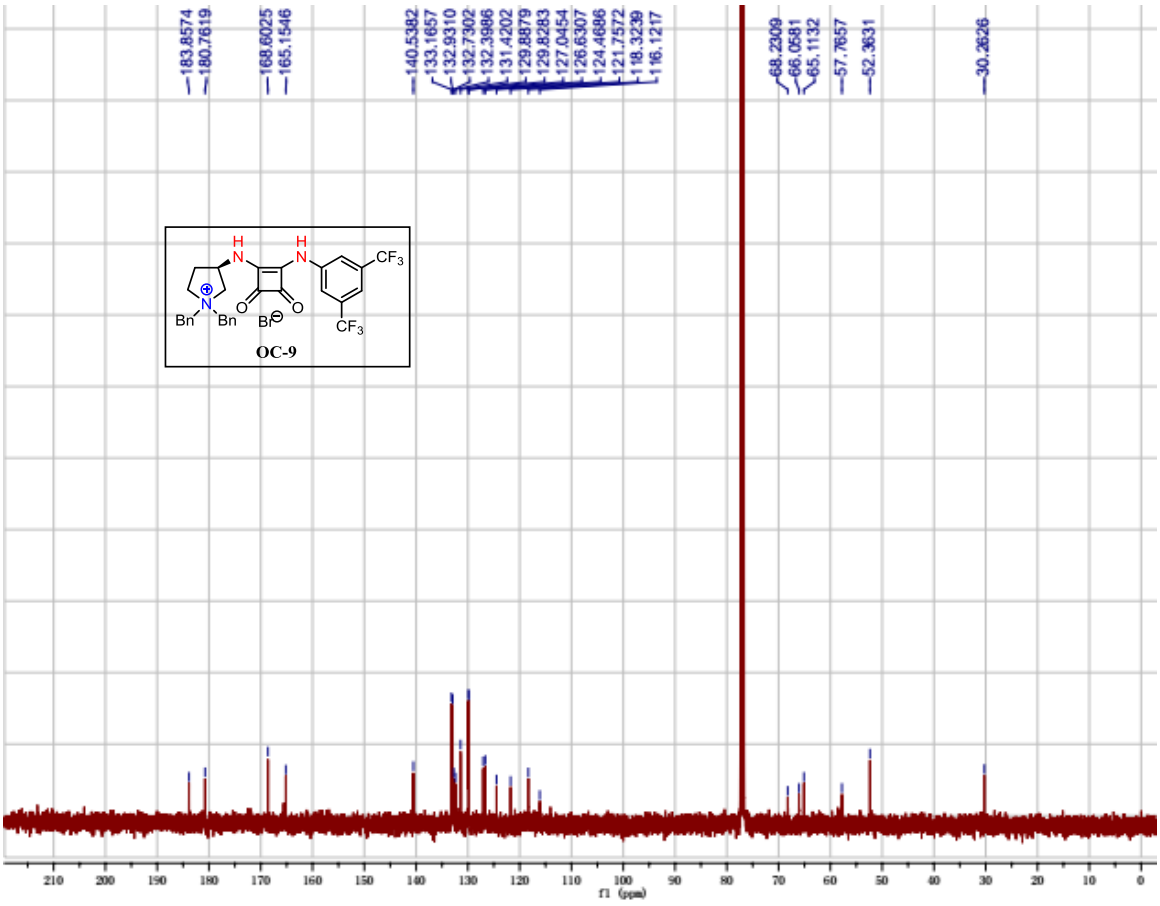
1.12
1.31
OC-9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132.9310
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129.8879
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127.0454
126.6307
124.4686
121.7572
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52.3631
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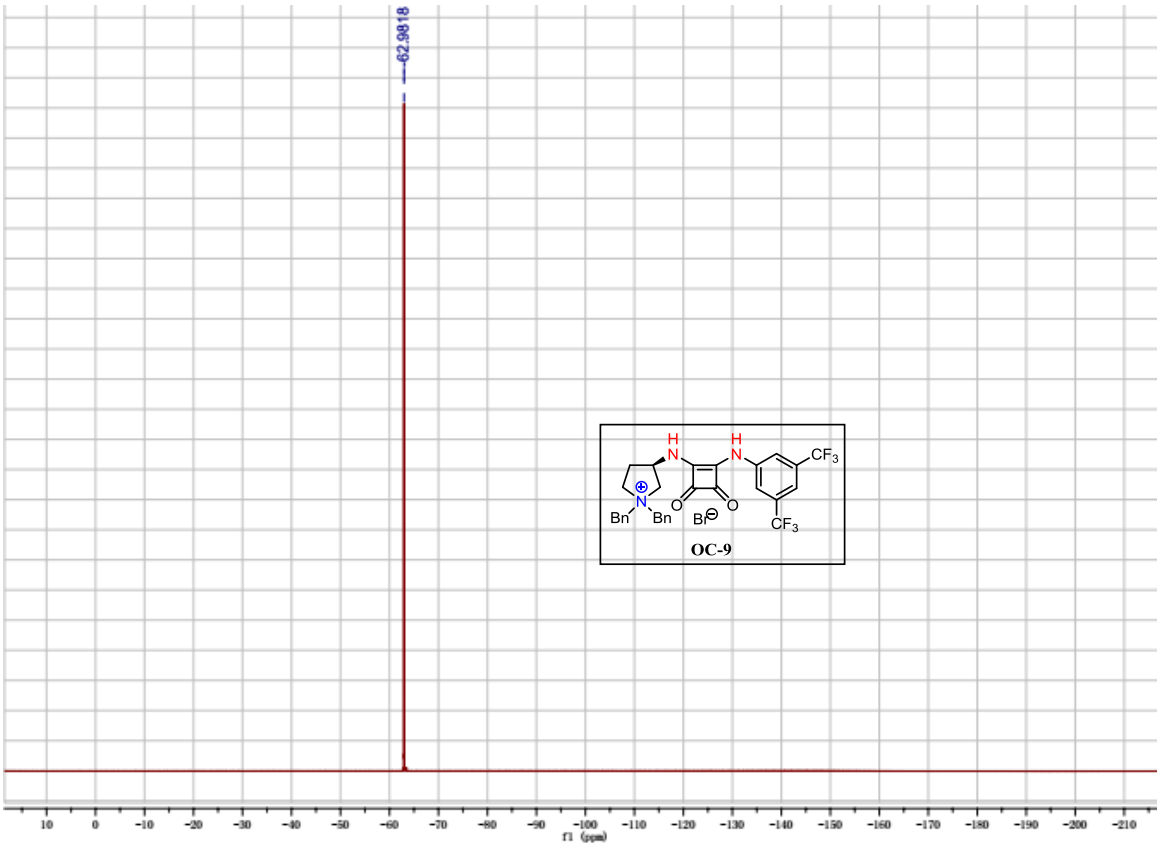
OC-9
f1 (ppm)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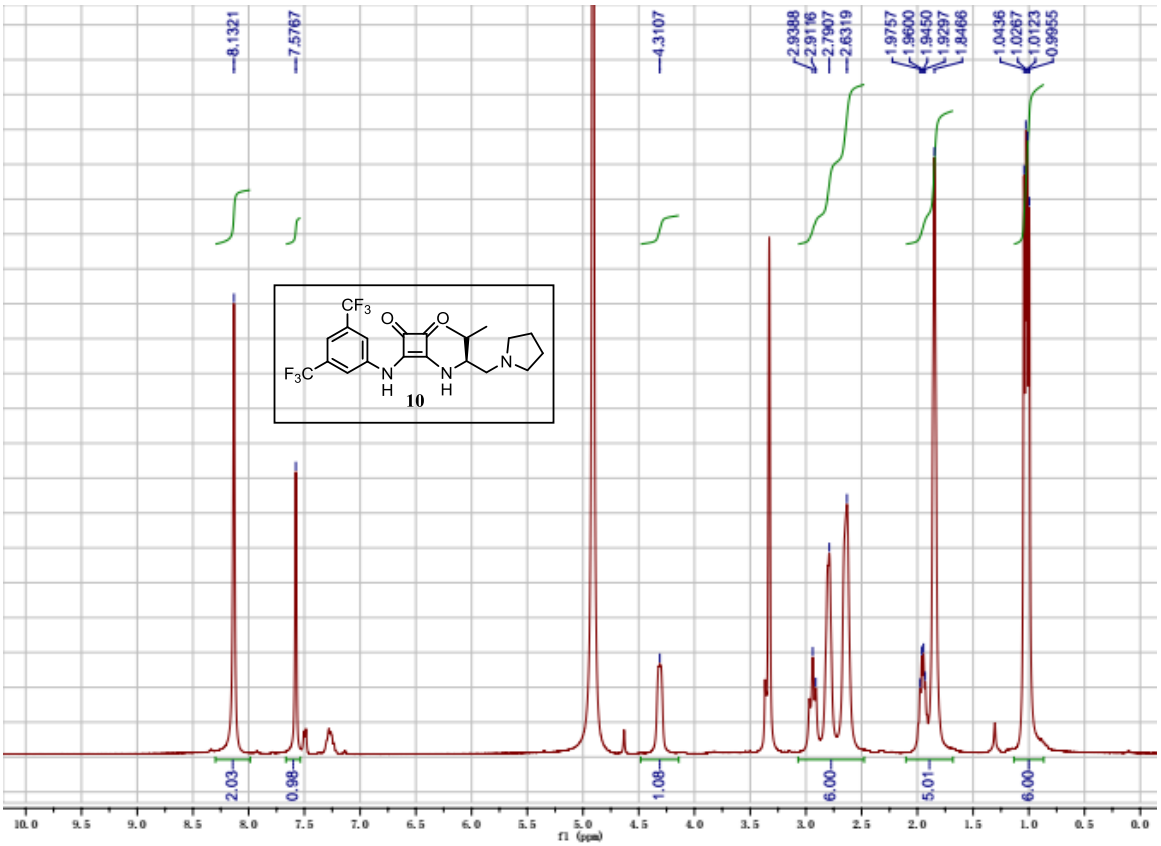
OC-9
f1 (ppm)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4.3107
2.9388
2.9116
2.7907
2.6319
1.9757
1.9600
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1.9297
1.8466
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0.9955
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0.98
1.08
6.00
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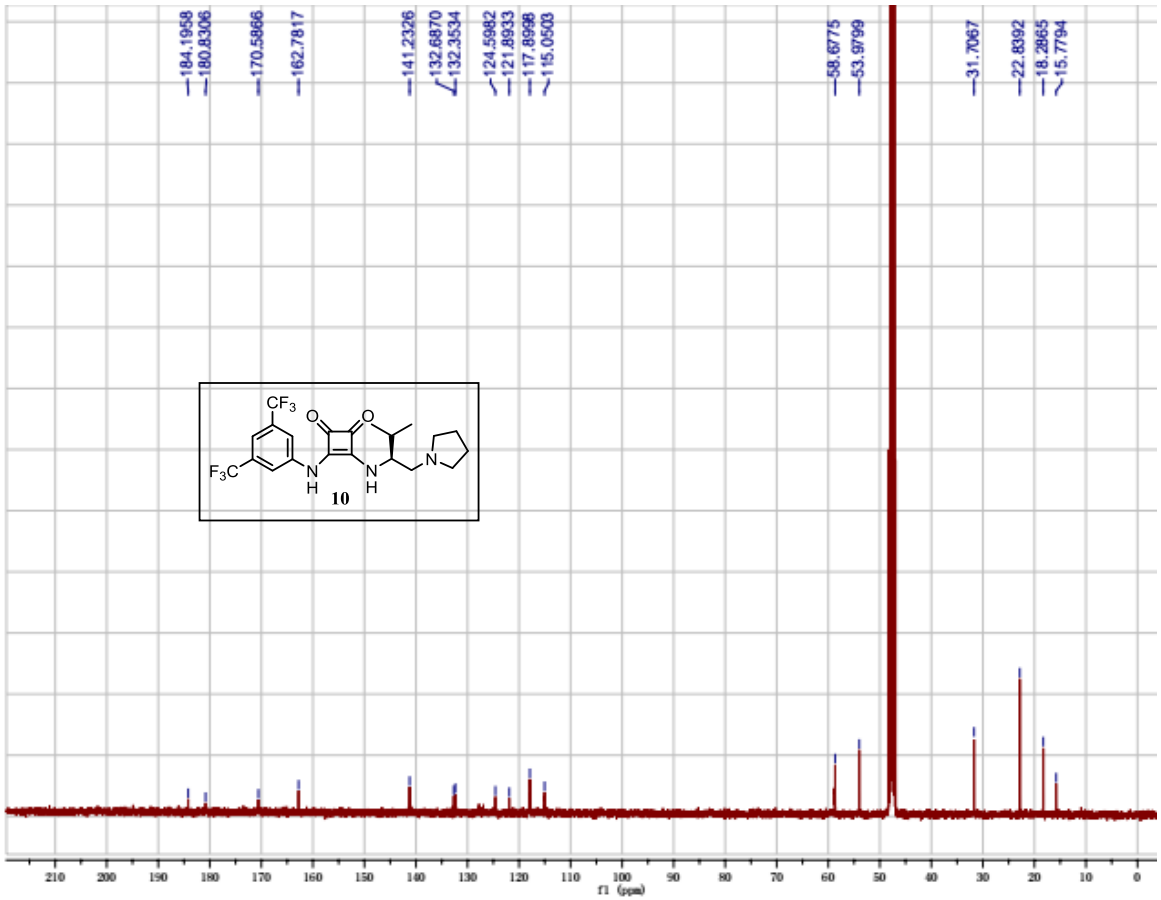
6.00
f1 (ppm)
10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132.3534
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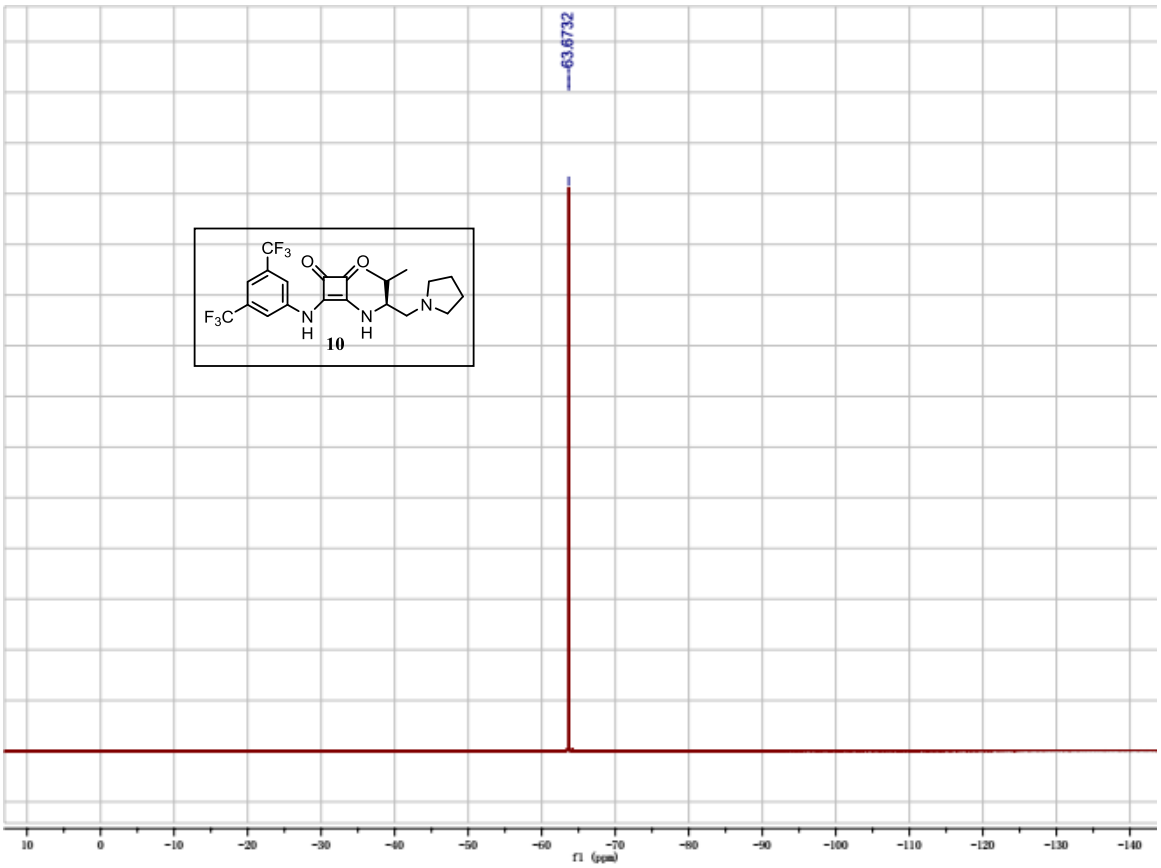
31.7067
22.8392
18.2865
15.7794
f1 (ppm)
10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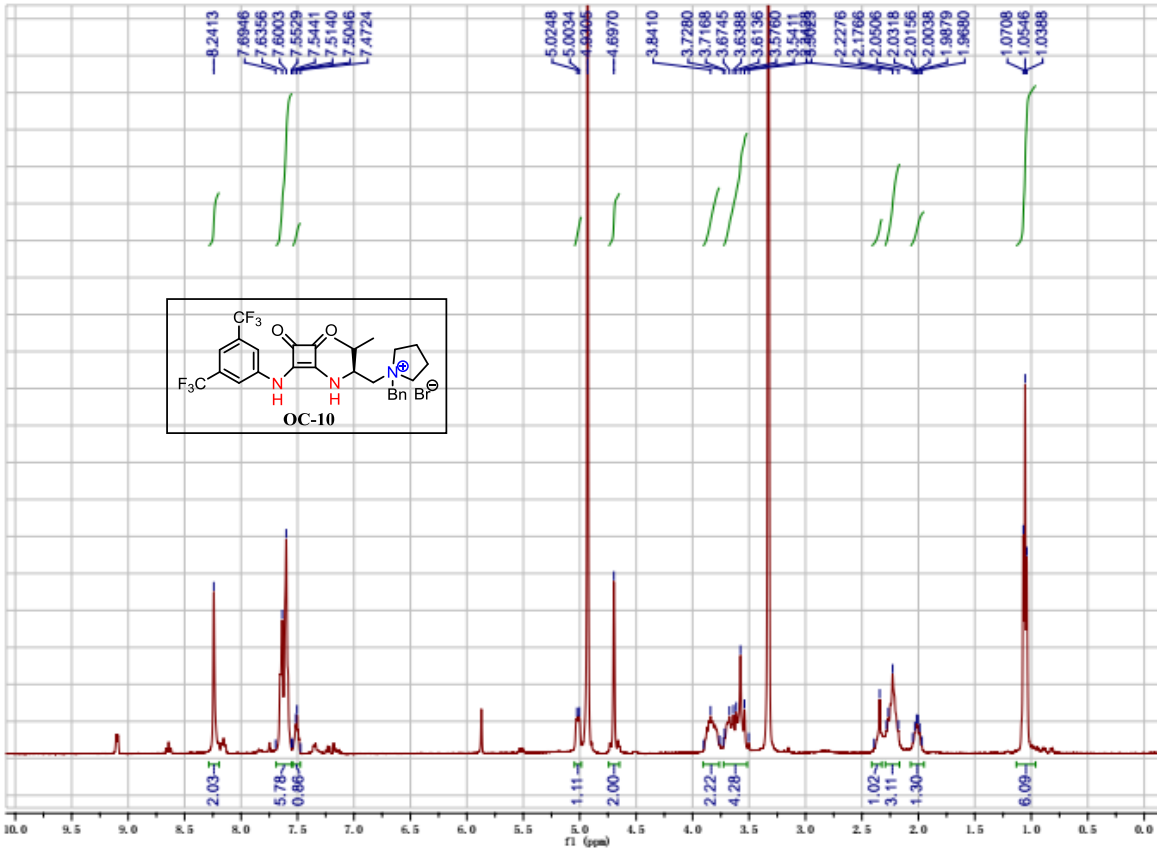
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7.5140
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7.4724
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5.0034
4.6970
3.8410
3.7280
3.7168
3.6745
3.6388
3.6136
3.5760
2.2276
2.1766
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2.0318
2.0156
2.0038
1.9879
1.9680
1.0708
1.0546
1.0388
OC-10
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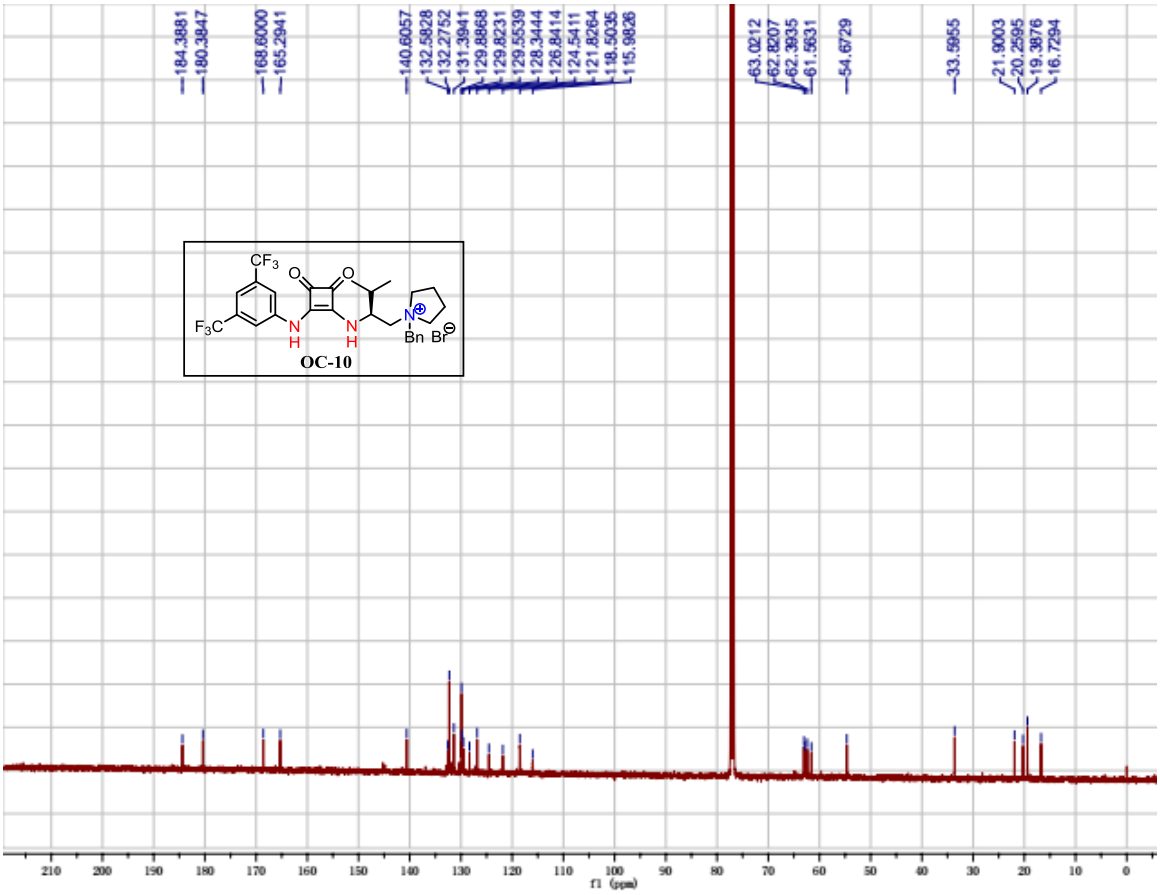
4.28
1.02
3.11
1.30
6.09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124.5411
121.8264
118.5035
115.9826
63.0212
62.8207
62.3935
61.5631
54.6729
33.5955
21.9003
20.2595
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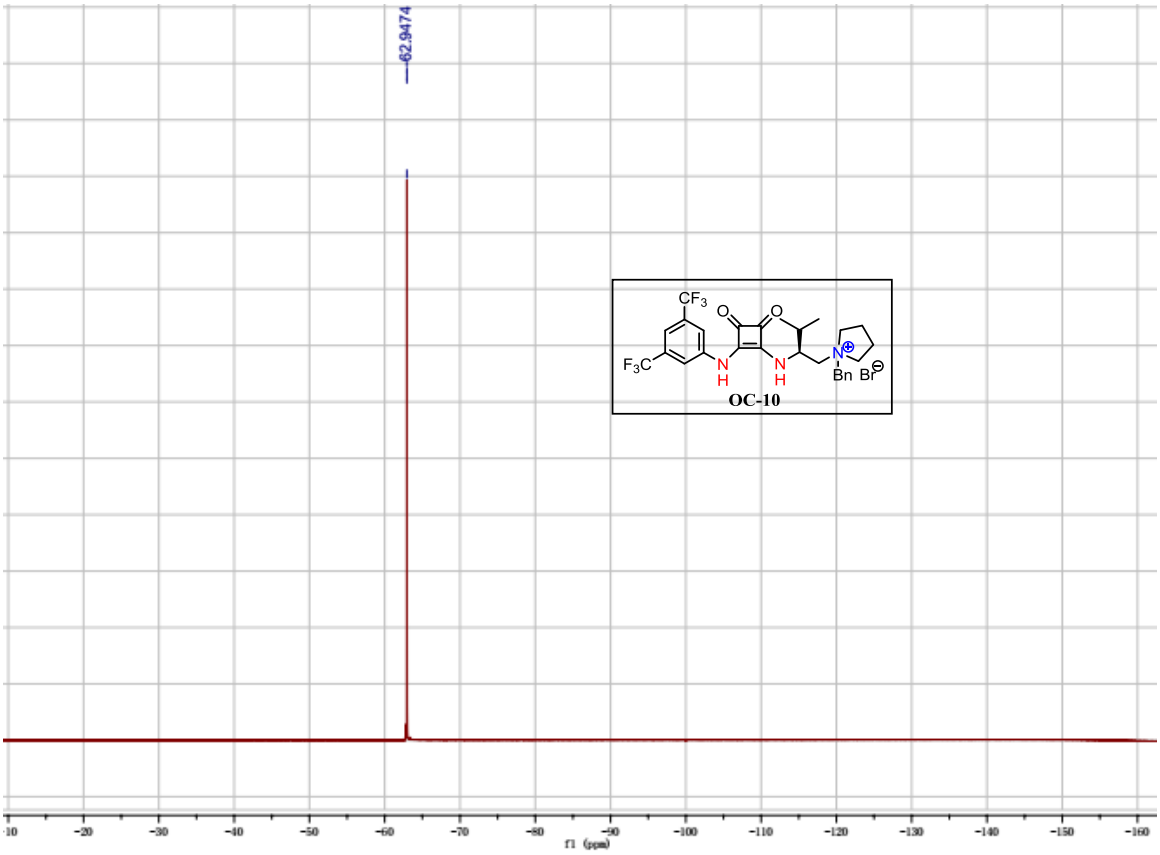
CF3
F3C
O
O
N
H
N
H
N
Bn Br
OC-10
f1 (ppm)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O
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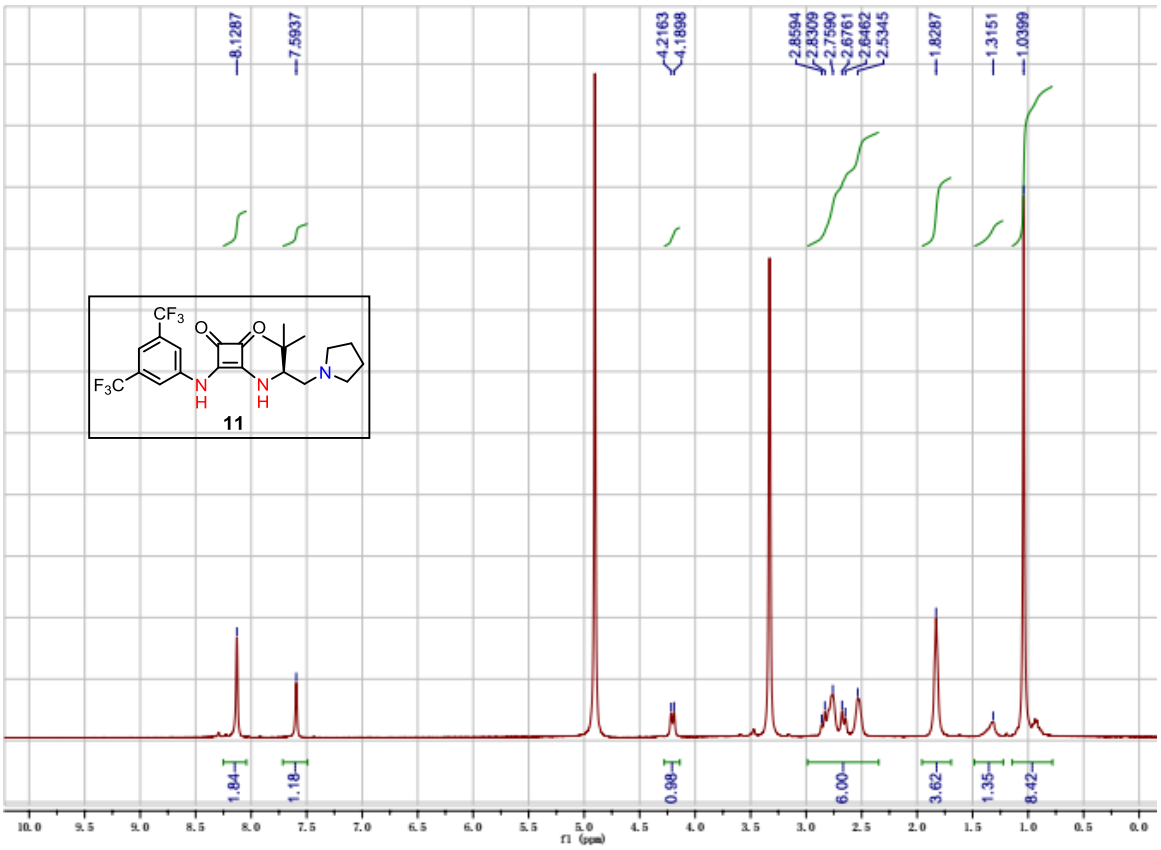
H
N
H
N
Bn Br
OC-10
f1 (ppm)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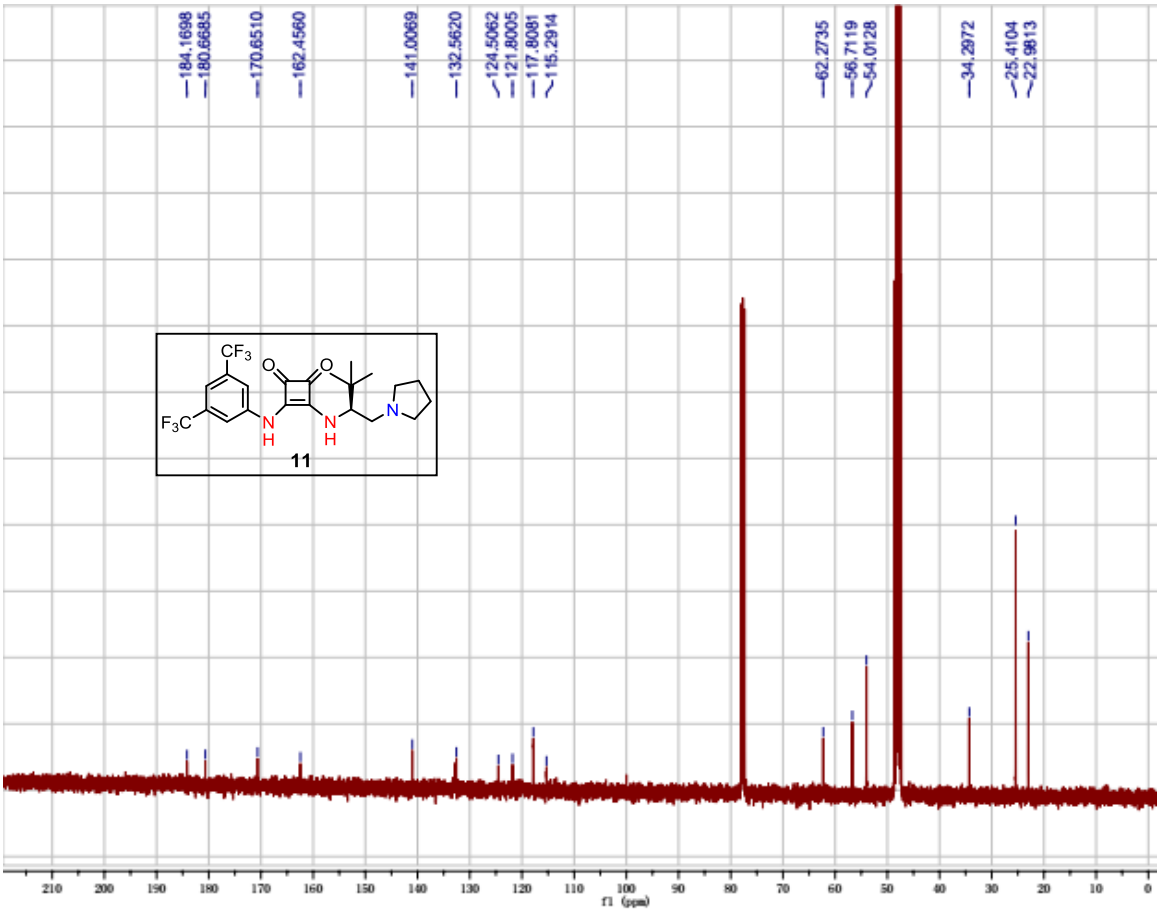
1.35
8.42
11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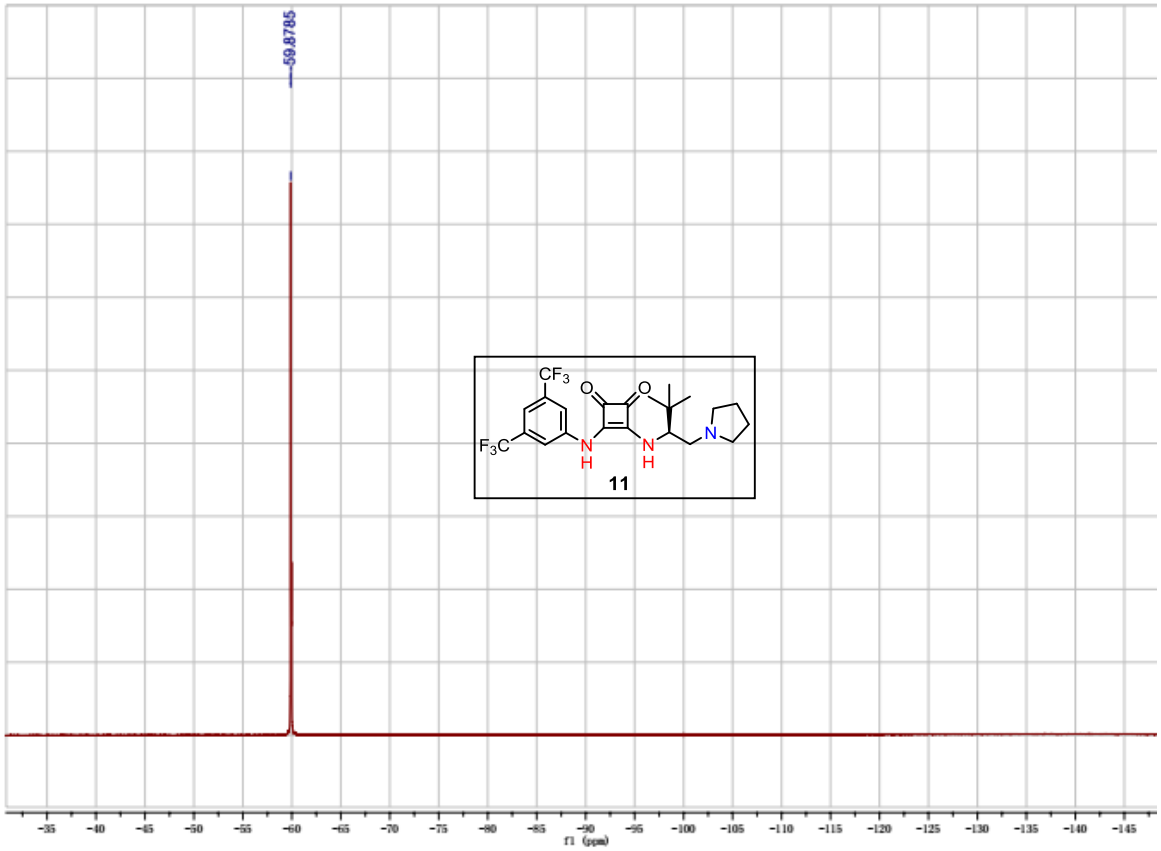
34.2972
25.4104
22.9813
11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F3C
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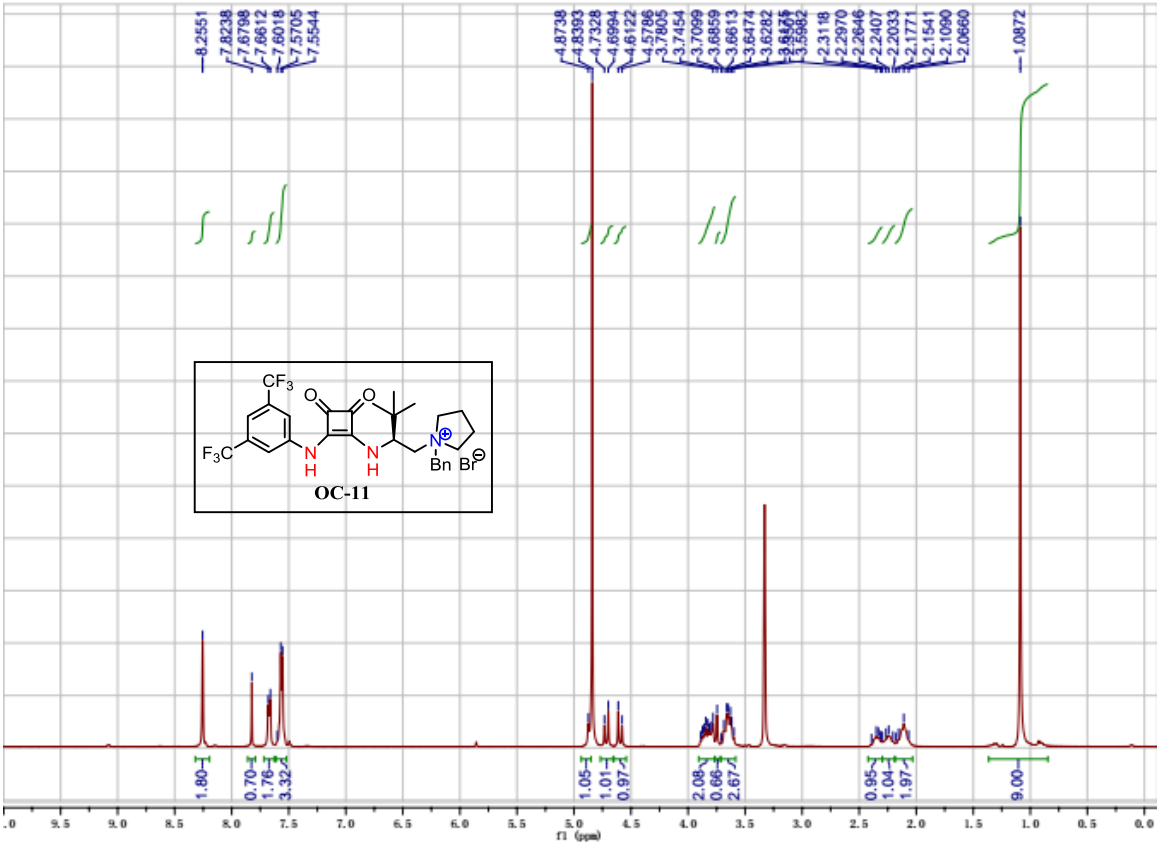
N
H
N
H
N
11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3.7099
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3.6613
3.6474
3.6282
2.3118
2.2970
2.2646
2.2407
2.2033
2.1771
2.1541
2.1090
2.0660
1.0872
CF3
F3C
O
O
N
H
N
H
N
Bn Br
OC-11
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1.01
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2.67
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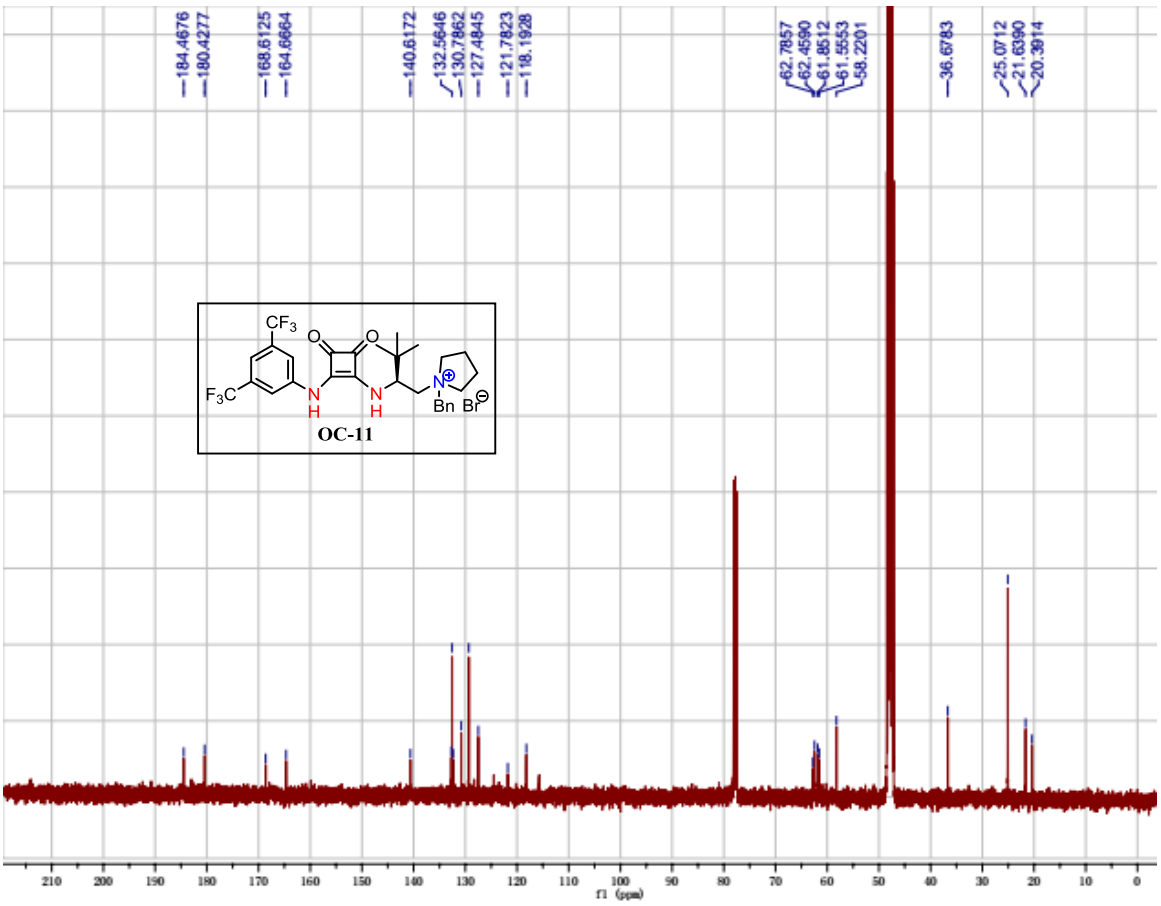
1.04
1.97
9.00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21.6390
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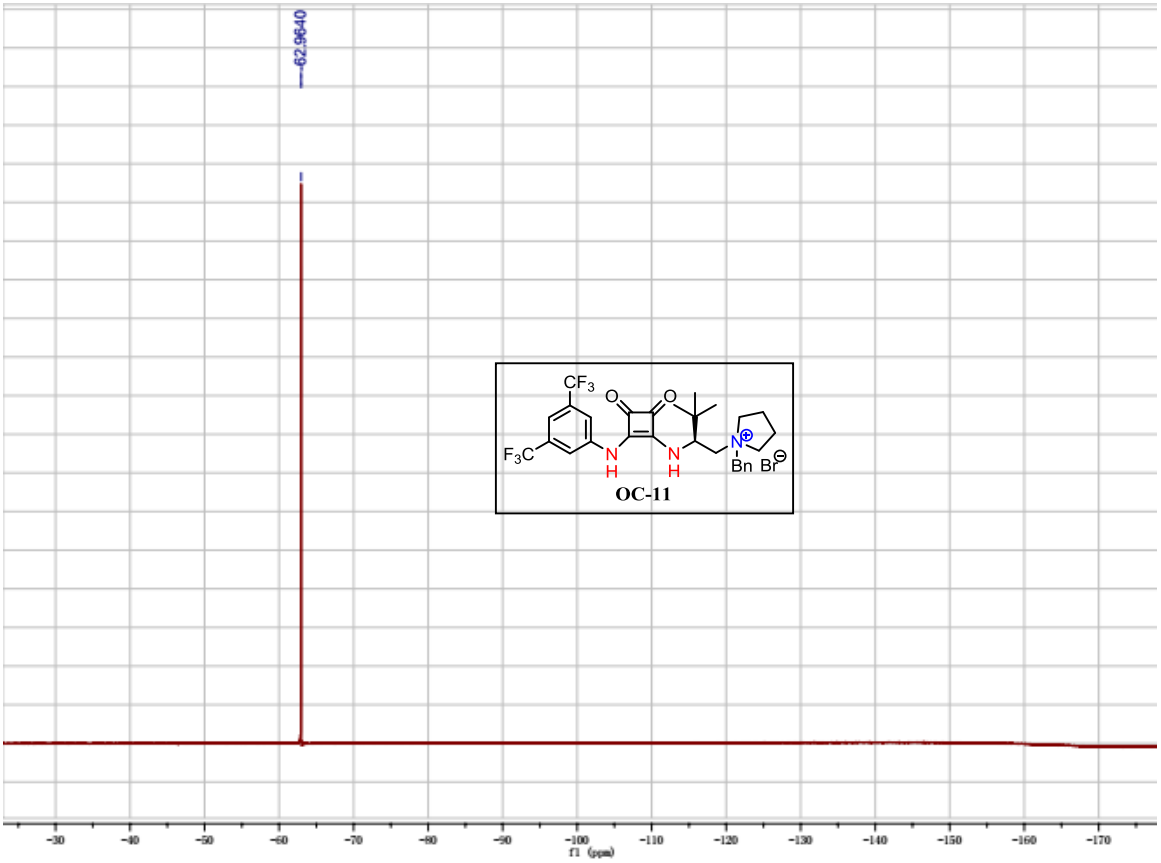
OC-11
f1 (ppm)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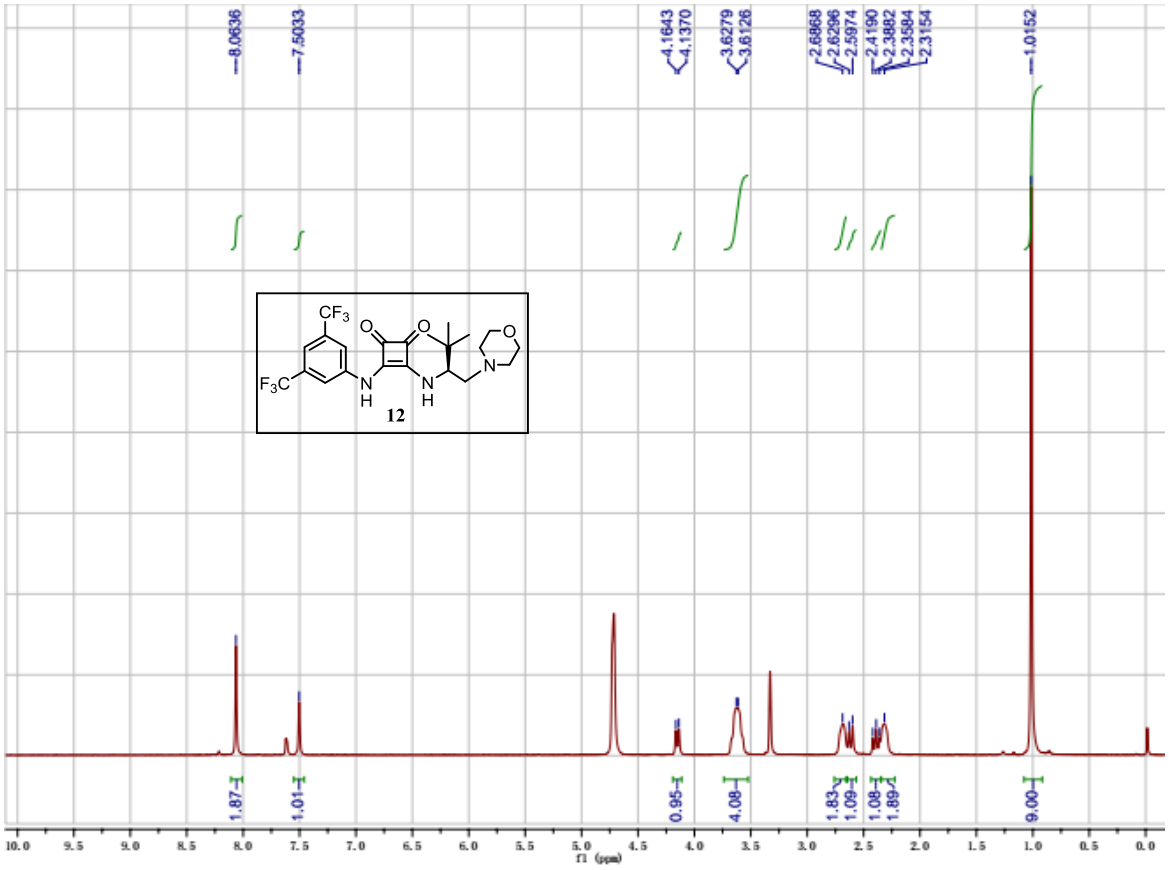
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2.3584
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4.08
1.83
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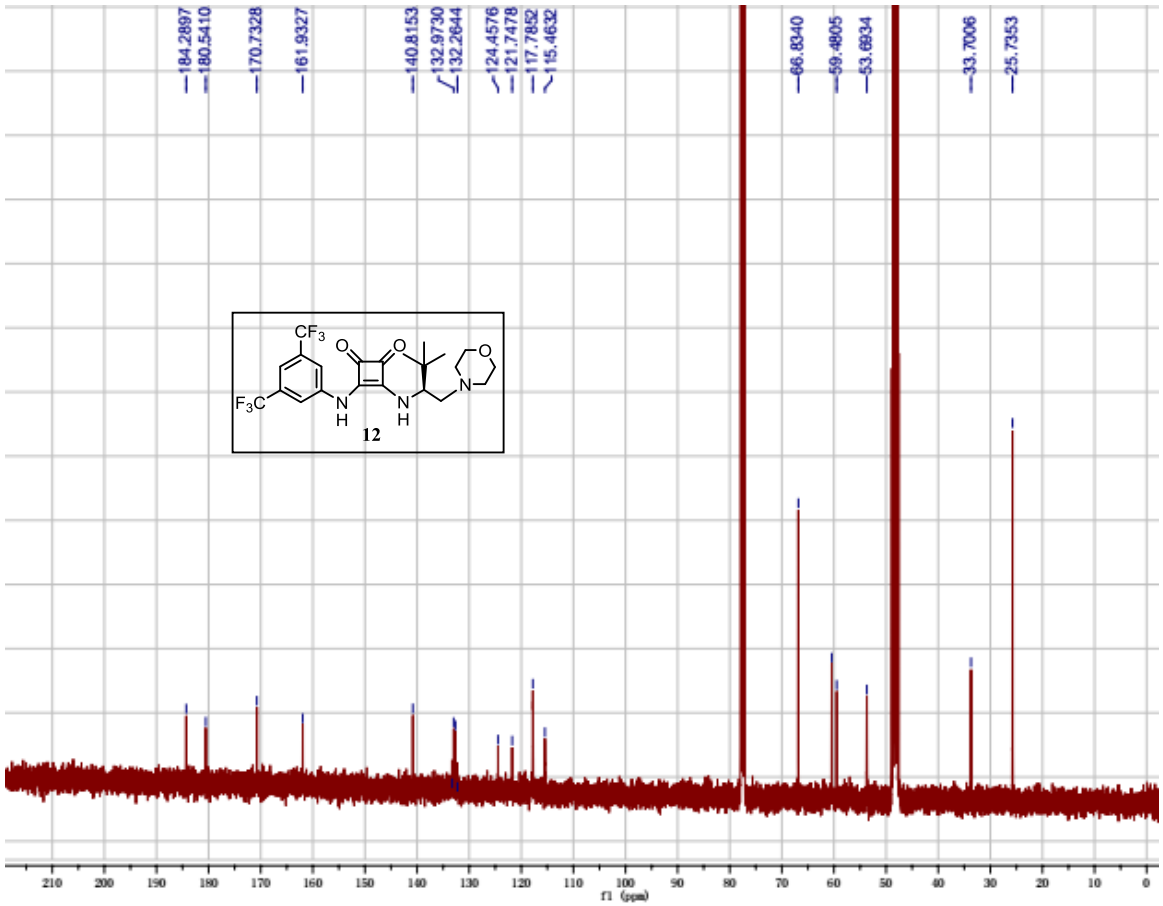
1.08
1.89
9.00
f1 (ppm)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170.7328
161.9327
140.8153
132.9730
132.2644
124.4576
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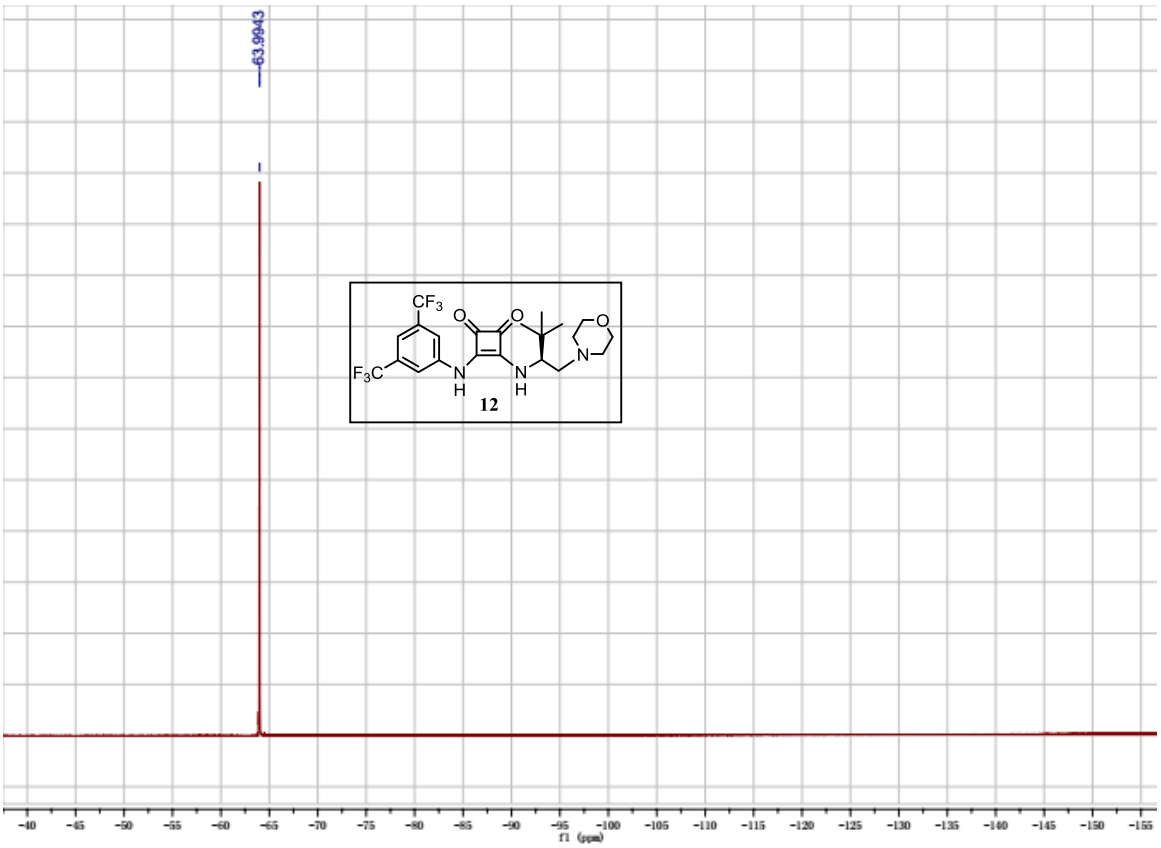
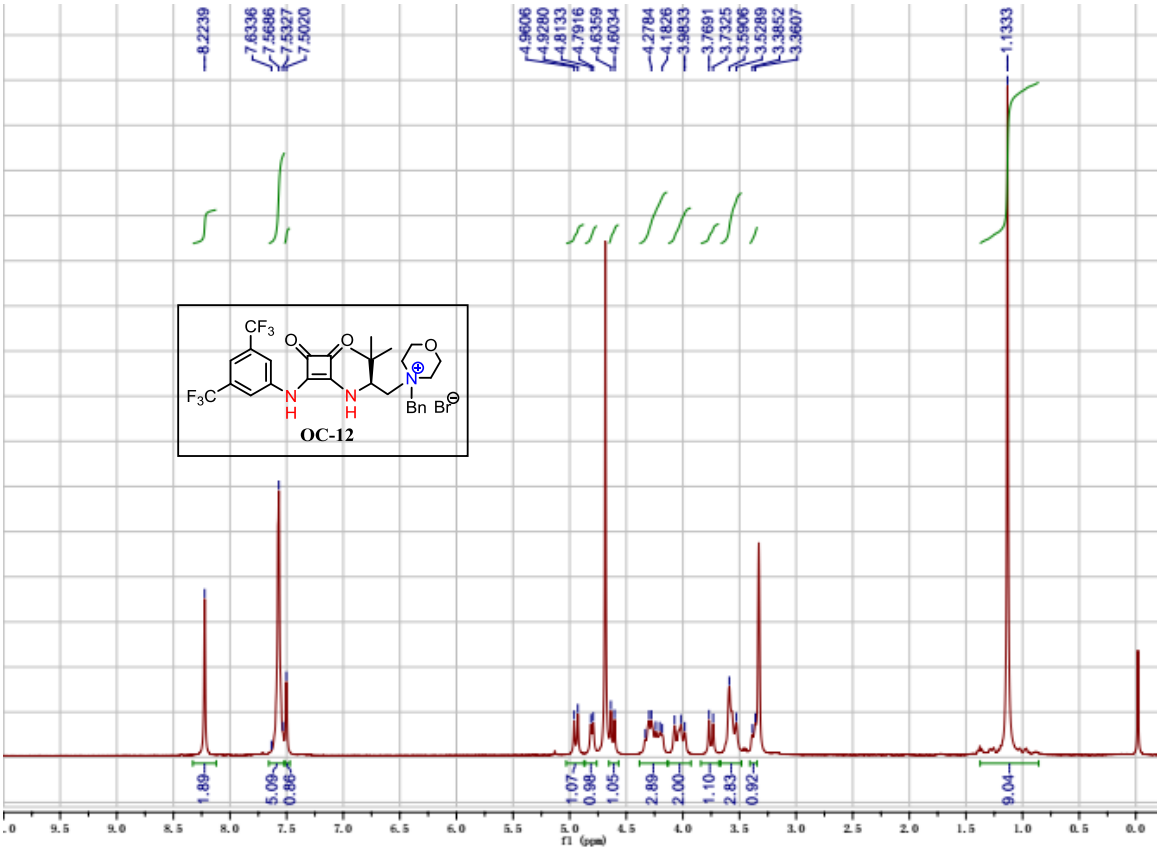
59.4805
53.6934
33.7006
25.7353
12
f1 (ppm)


-63.9943
12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4.2784
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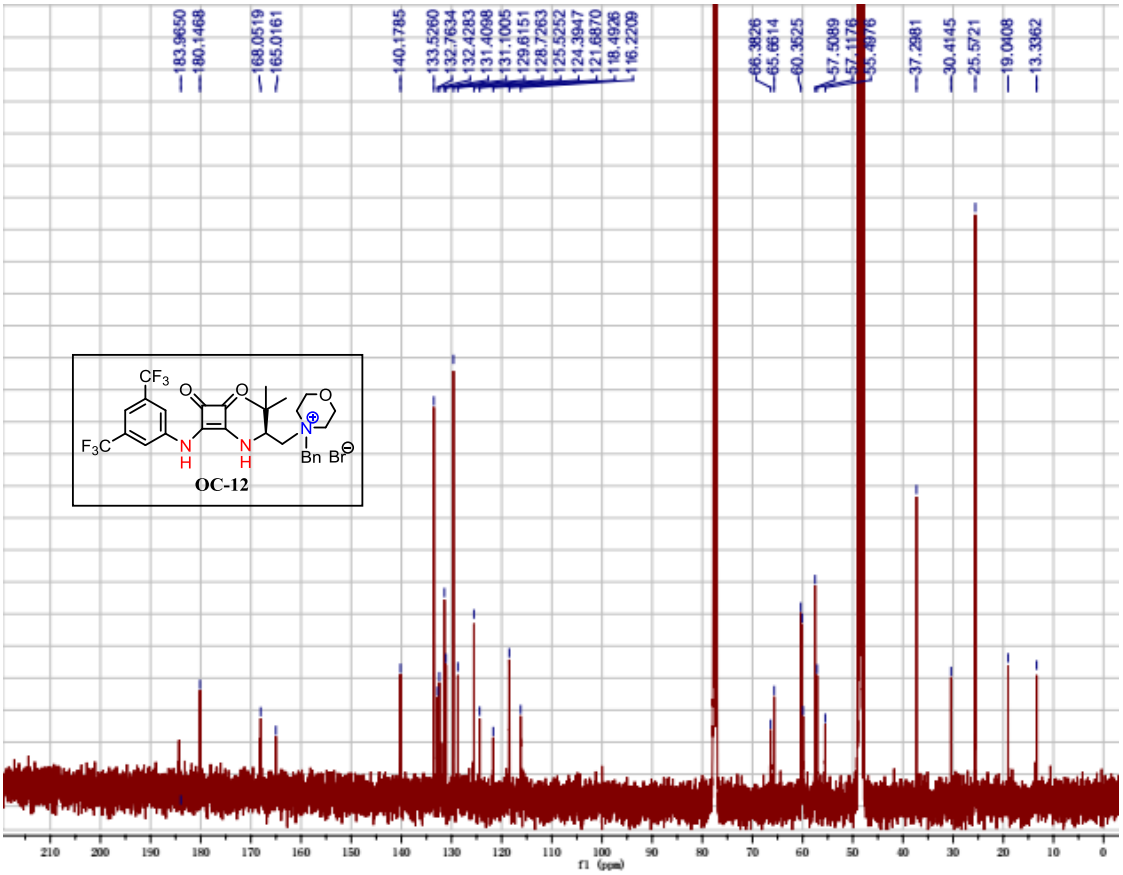
3.7325
3.5906
3.5289
3.3852
3.3607
1.1333
OC-12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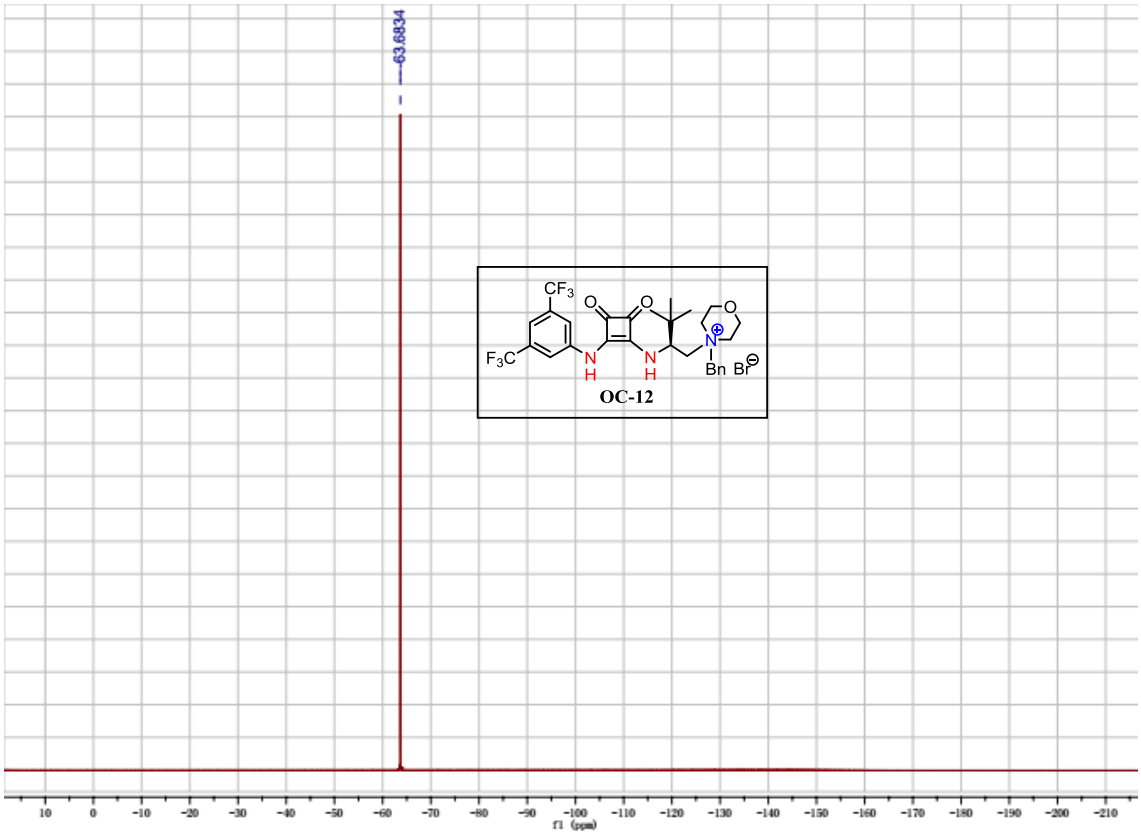
131.1005
129.6151
128.7263
125.5252
124.3947
121.6870
118.4926
116.2209
66.3826
65.6614
60.3525
57.5089
57.1176
55.4876
37.2981
30.4145
25.5721
19.0408
13.3362
CF3
F3C
O
O
O
N
H
N
H
N
Bn Br
OC-12
f1 (ppm)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N
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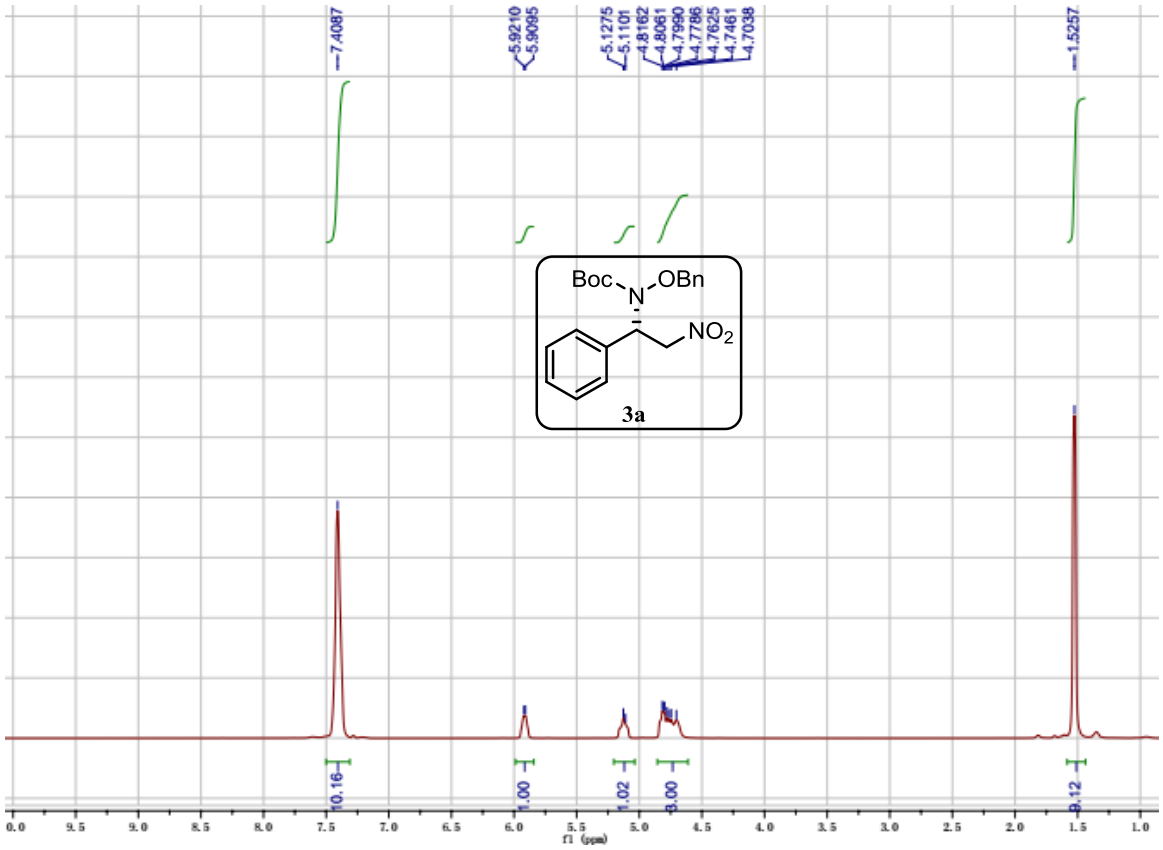
N
Bn Br
OC-12
f1 (ppm)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Boc
N
OBn
NO2
3a
10.16
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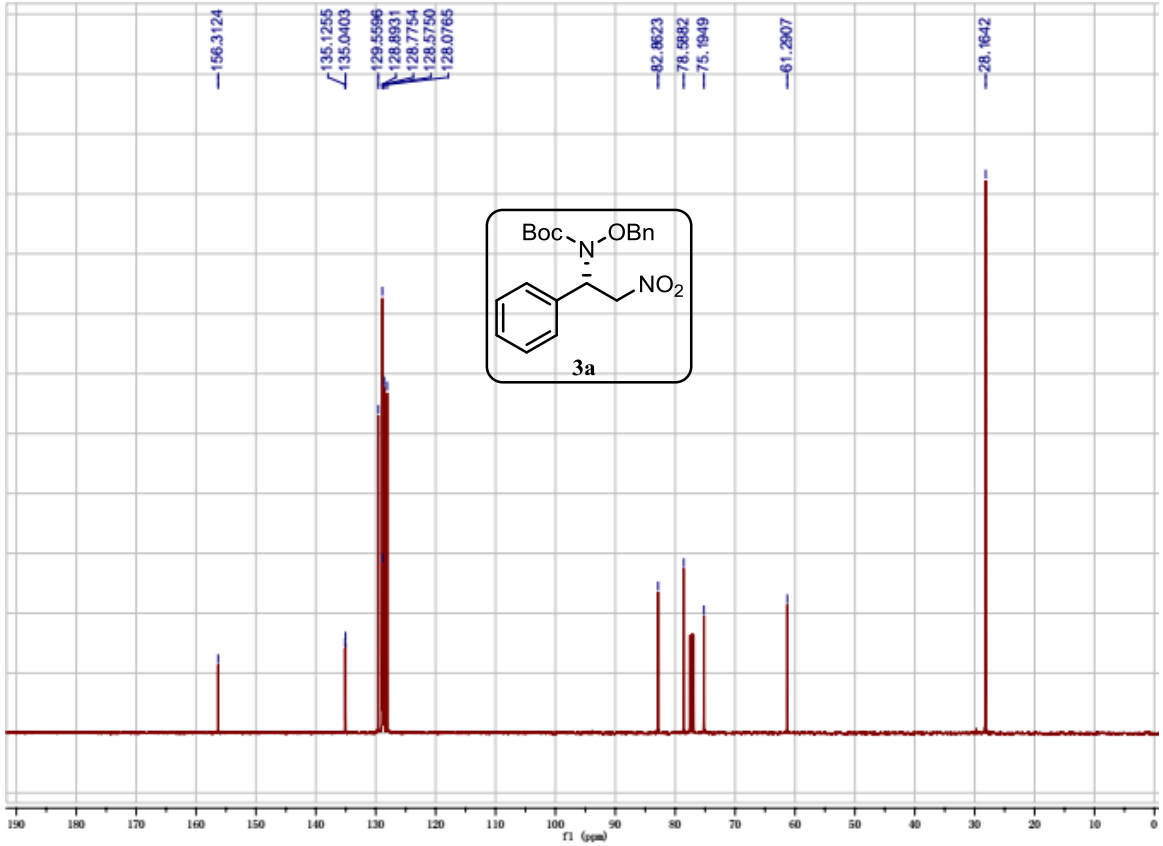
1.02
3.00
9.12
f1 (ppm)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128.8931
128.7754
128.5750
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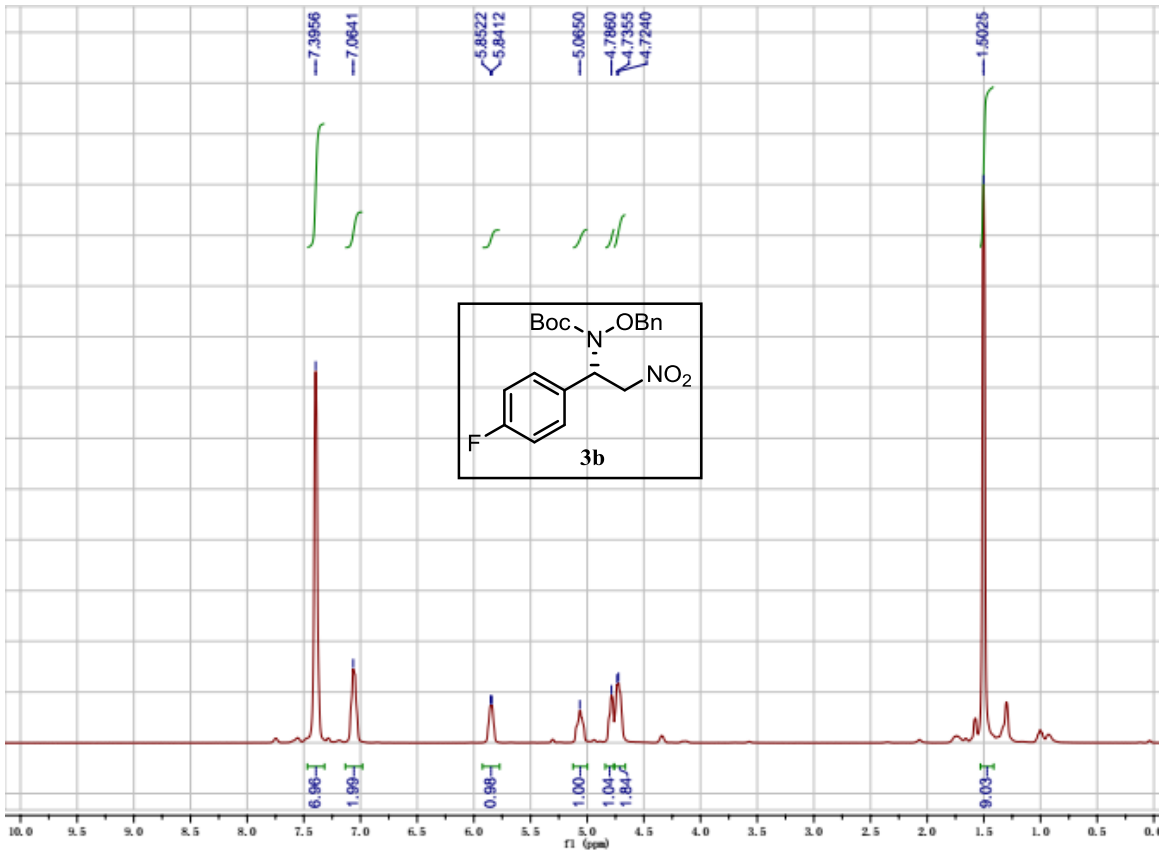
Boc
N
OBn
NO2
3a
f1 (ppm)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5.0650
4.7860
4.7355
4.7240
1.5025
Boc
N
OBn
NO2
F
3b
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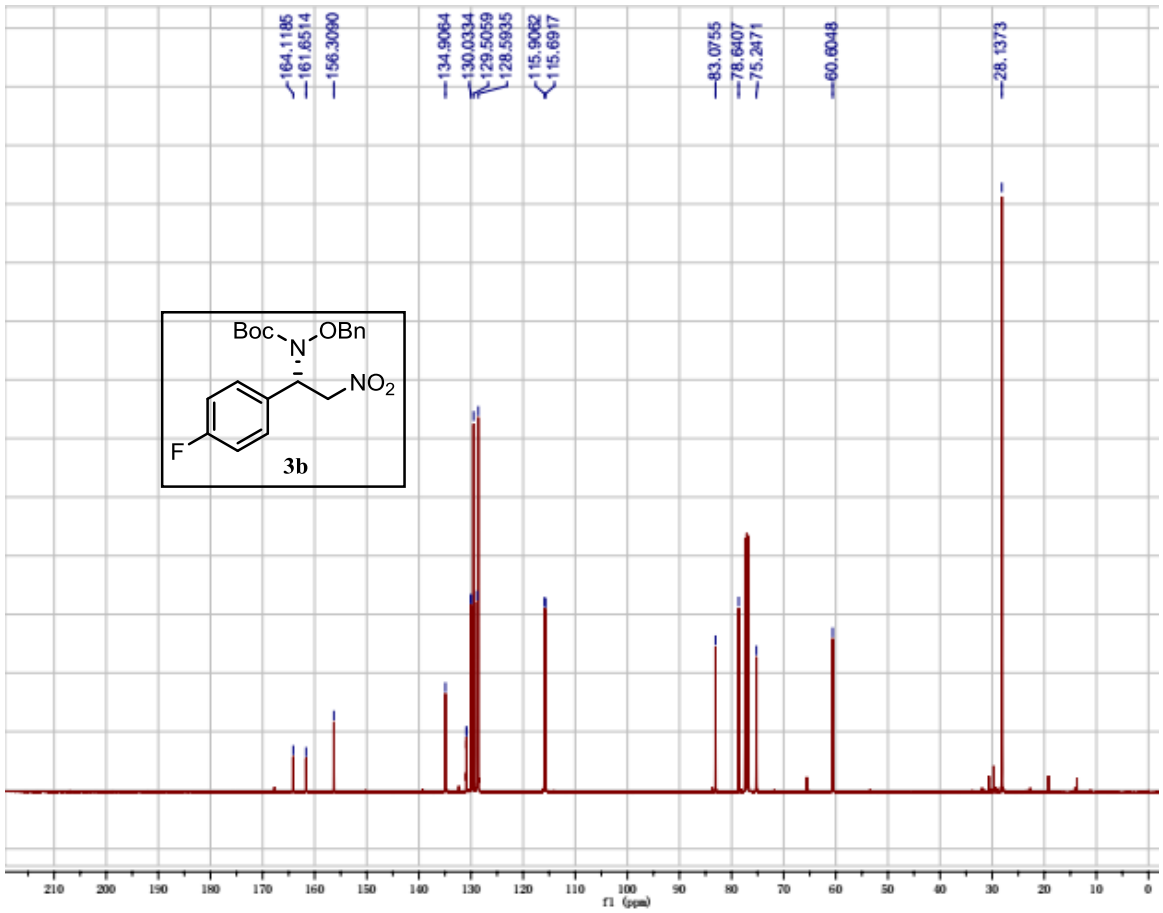
1.99
0.98
1.00
1.04
1.84
9.03
f1 (ppm)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129.5059
128.5935
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115.6917
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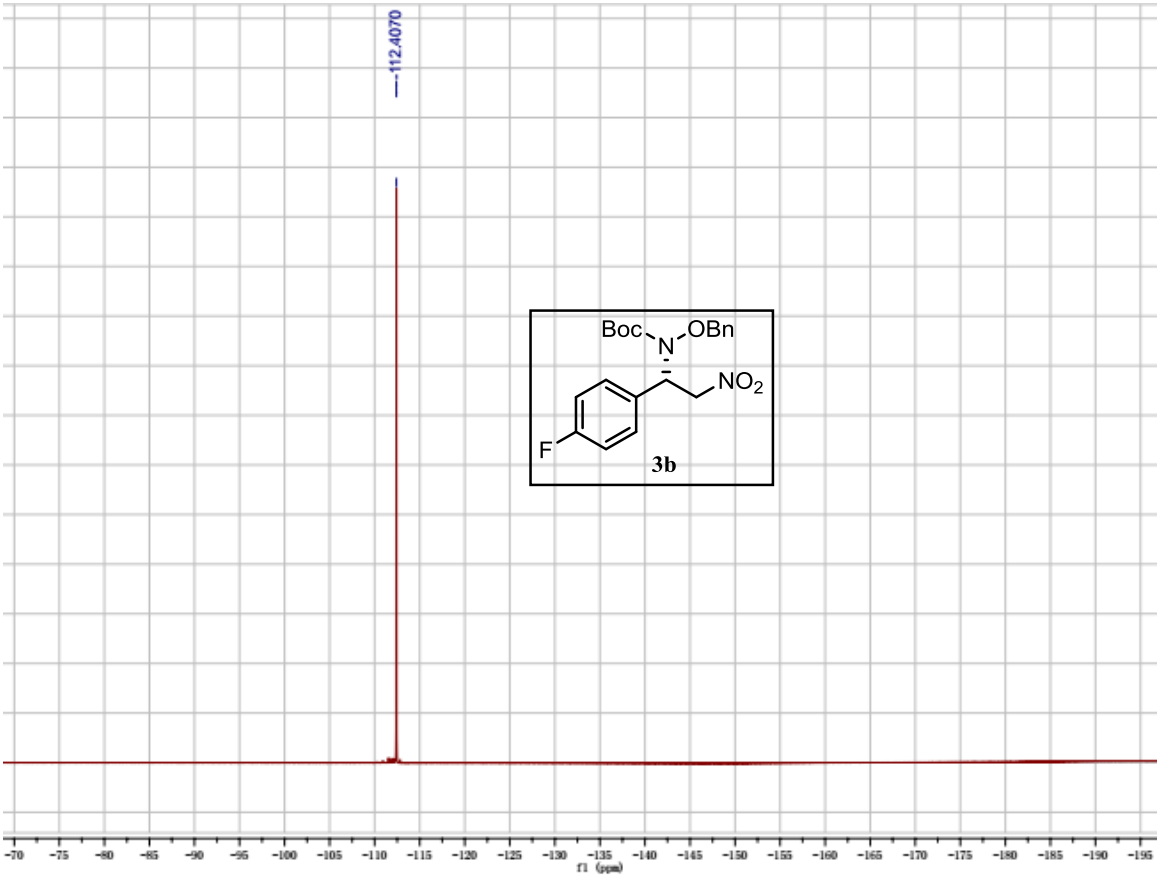
Boc
N
OBn
NO2
F
3b
f1 (ppm)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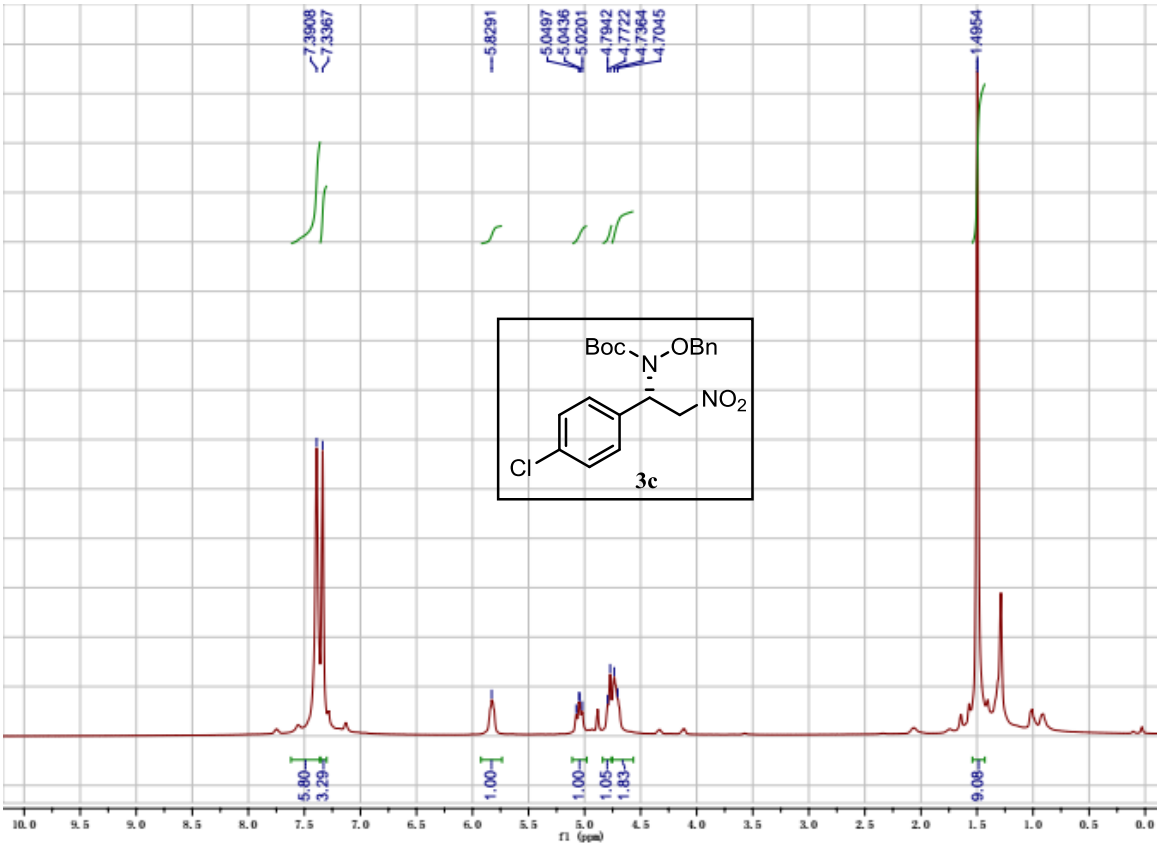
OBn
NO2
F
3b
f1 (ppm)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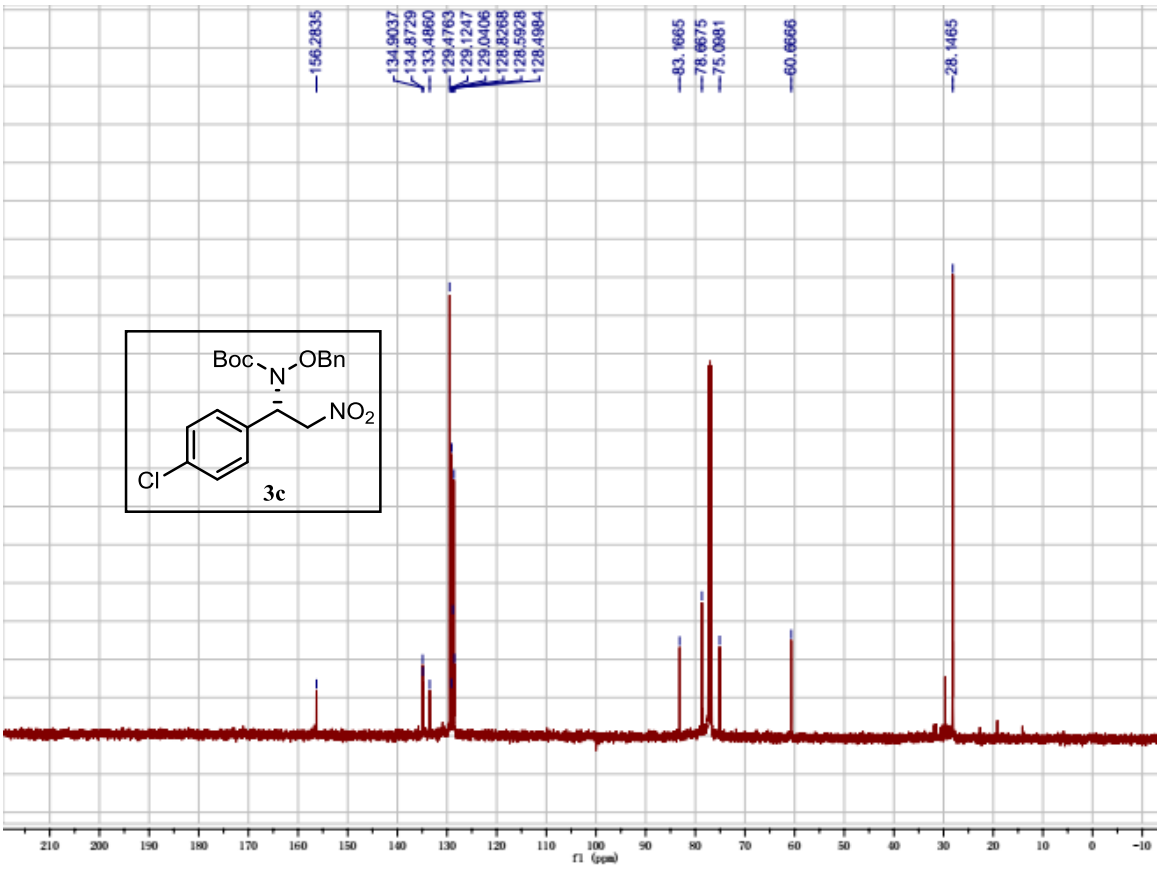
5.0497
5.0436
5.0201
4.7942
4.7722
4.7364
4.7045
1.4954
Boc
N
OBn
NO2
Cl
3c
5.80
3.29
1.00
1.00
1.05
1.83
9.08
f1 (ppm)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129.4763
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129.0406
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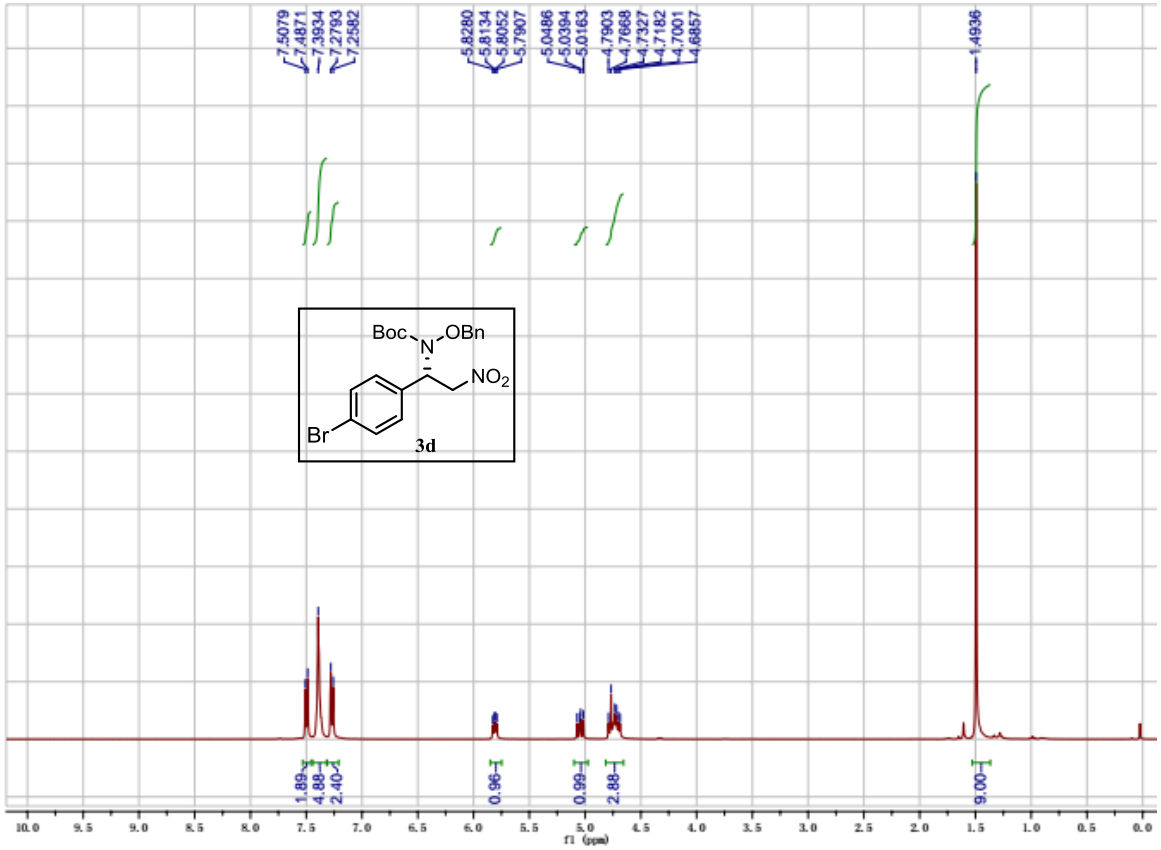
Boc
N
OBn
NO2
Cl
3c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5.8280
5.8134
5.8052
5.7907
5.0486
5.0394
5.0163
4.7903
4.7668
4.7327
4.7182
4.7001
4.6857
1.4936
Boc
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OBn
NO2
Br
3d
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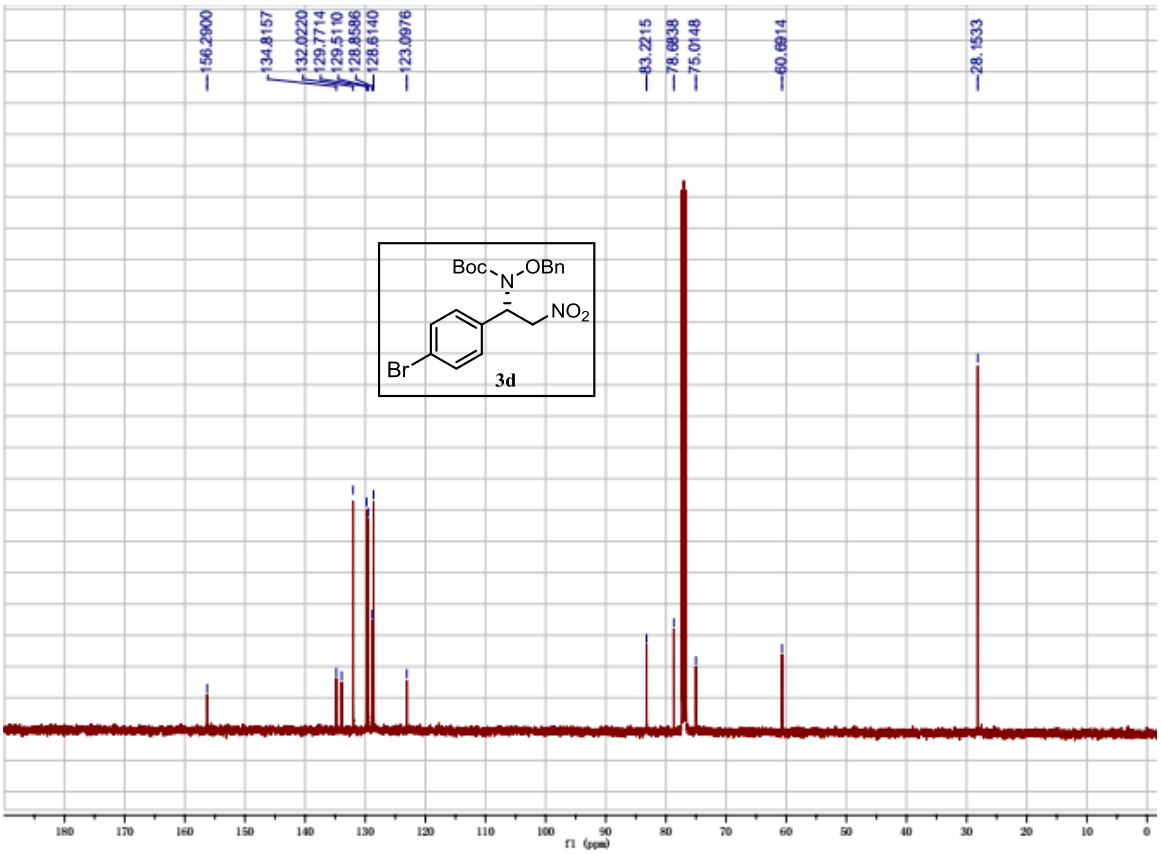
4.88
2.40
0.96
0.99
2.88
9.00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128.8586
128.6140
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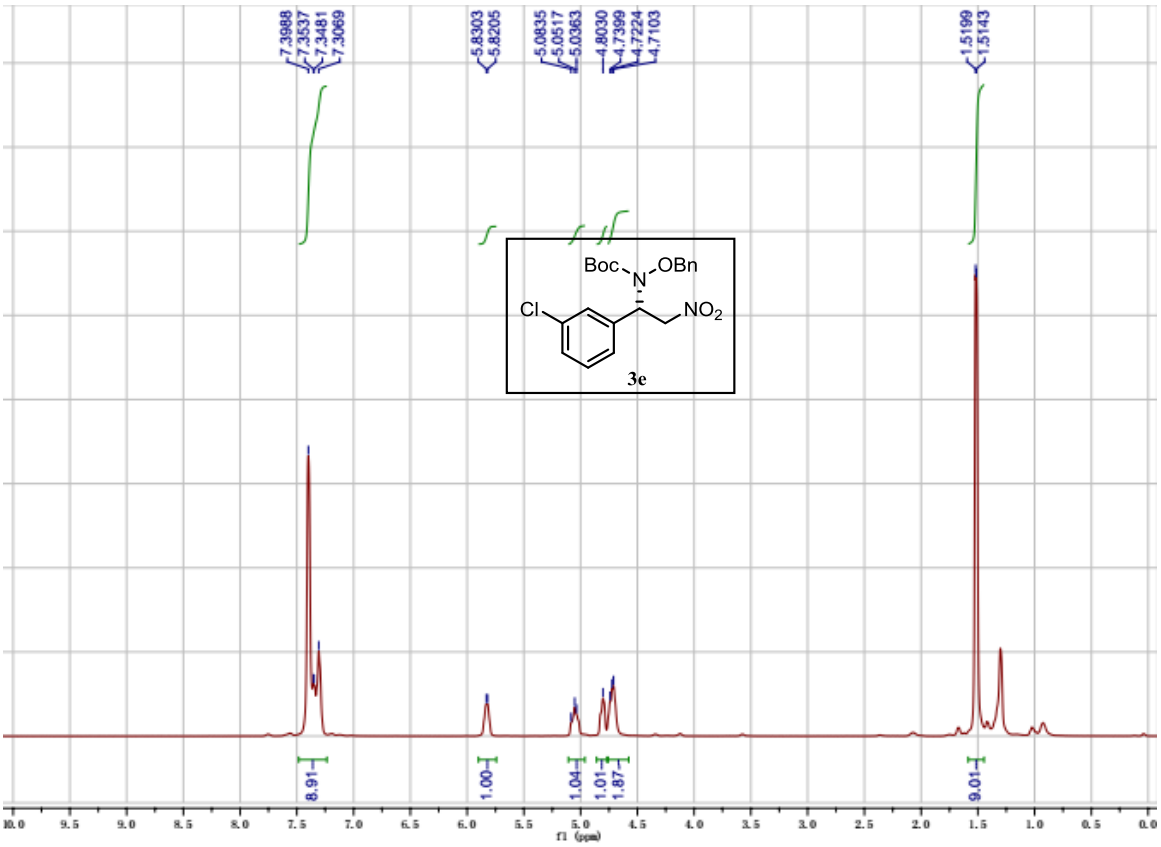
78.6838
75.0148
60.6914
28.1533
Boc
N
OBn
NO2
Br
3d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5.8205
5.0835
5.0517
5.0363
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4.7399
4.7224
4.7103
1.5199
1.5143
Boc
N
OBn
Cl
NO2
3e
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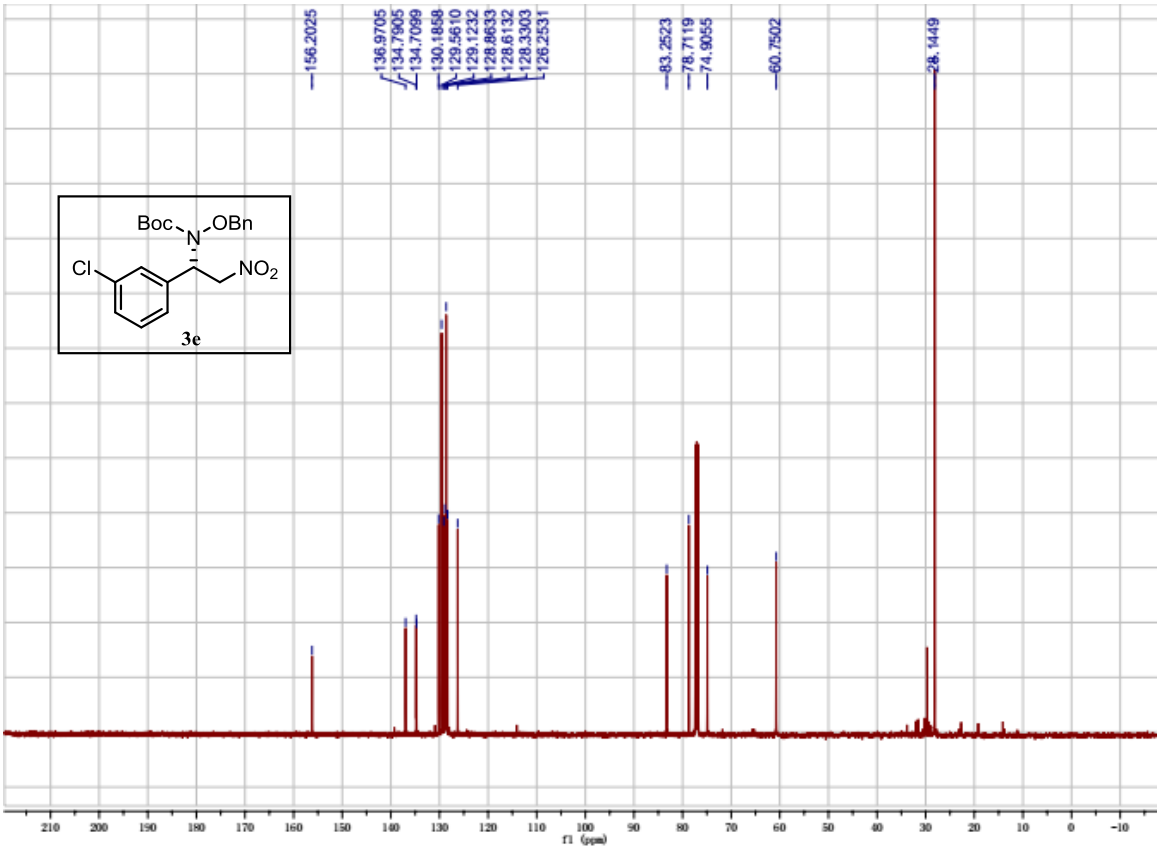
1.00
1.04
1.01
1.87
9.01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128.6132
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126.2531
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78.7119
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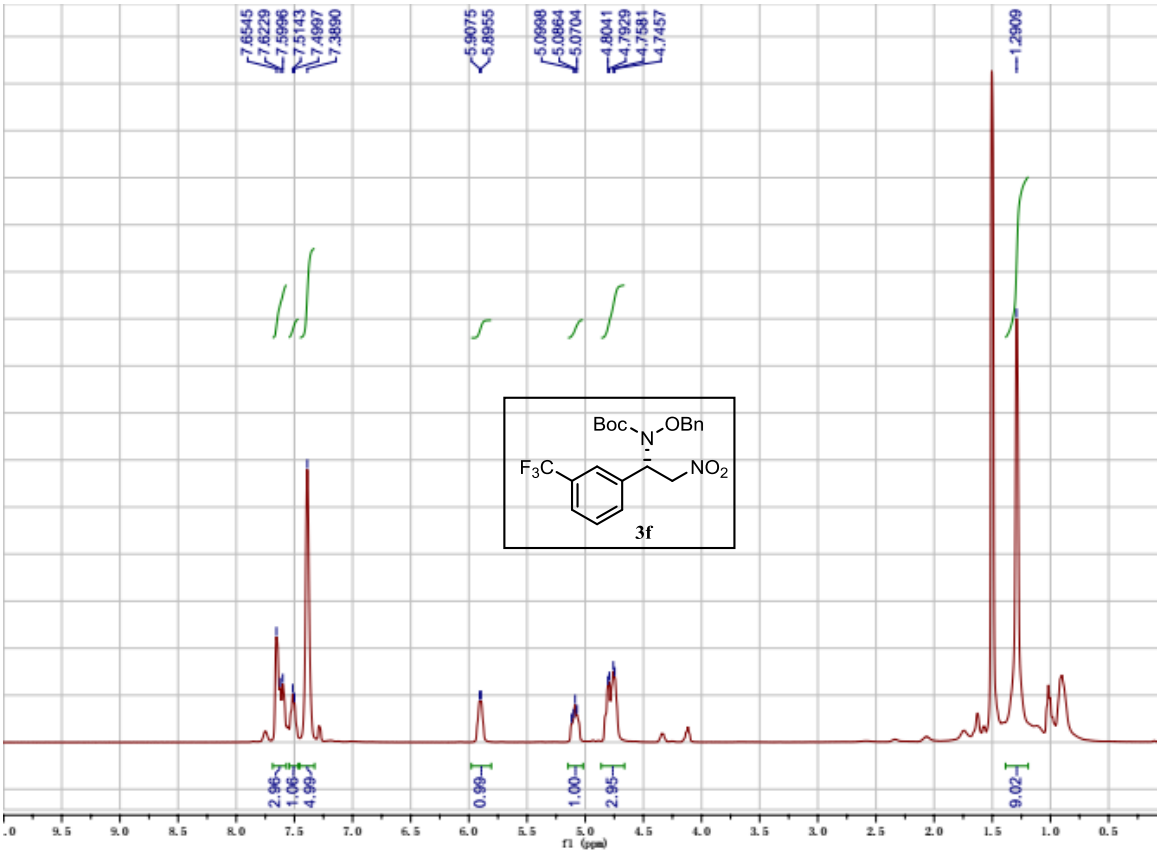
Boc
N
OBn
Cl
NO2
3e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4.7929
4.7581
4.7457
1.2909
Boc
N
OBn
F3C
NO2
3f
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4.99
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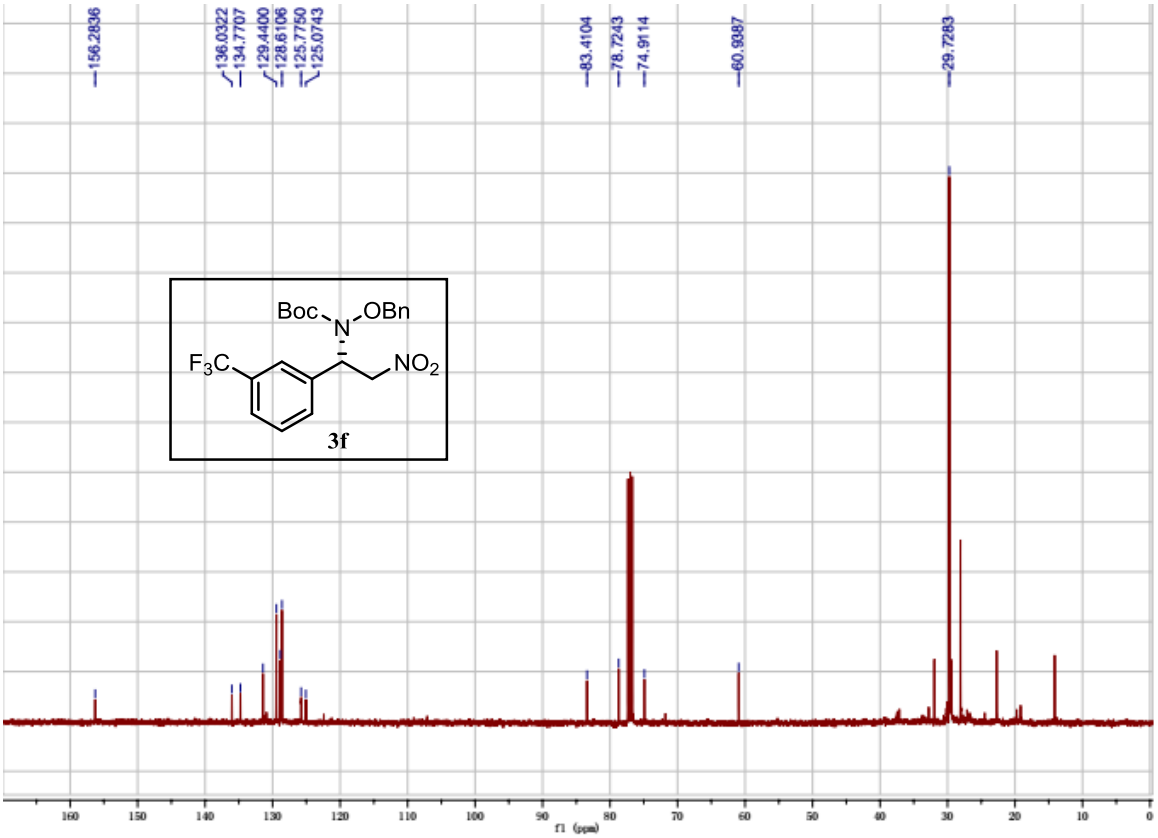
1.00
2.95
9.02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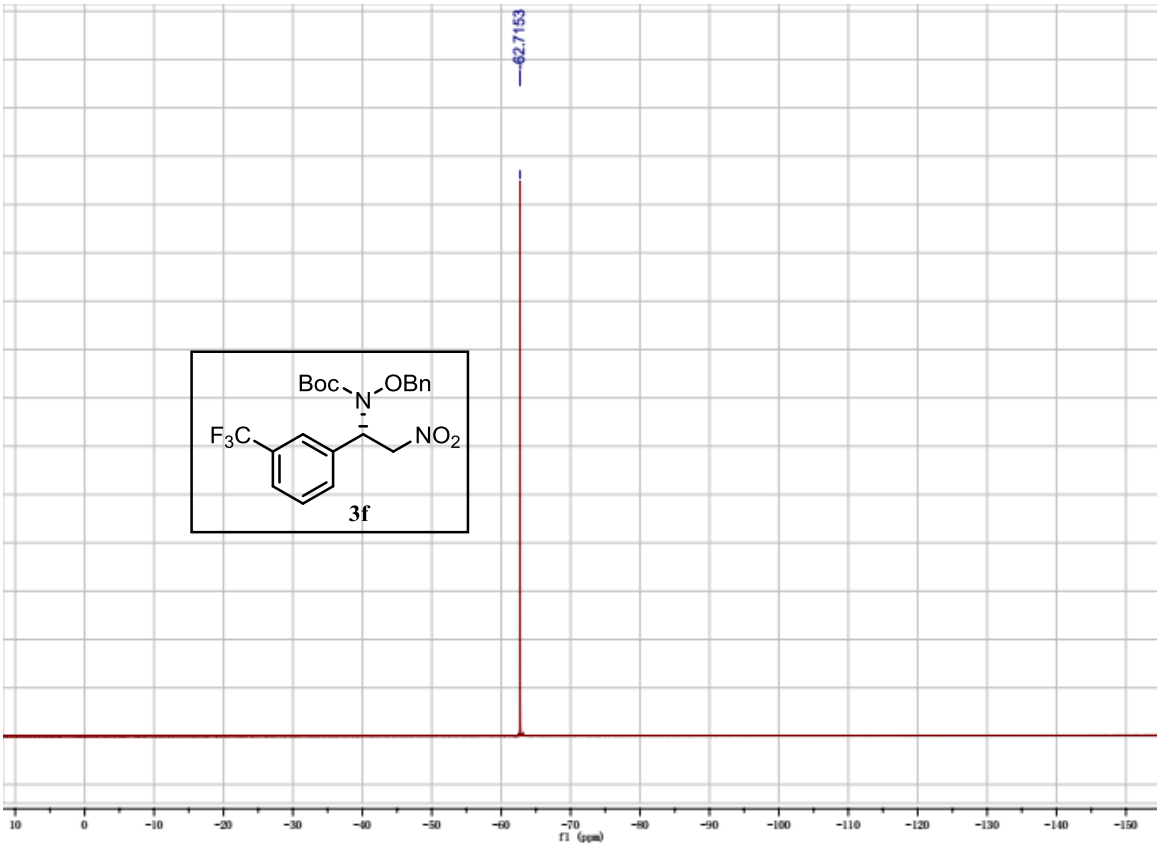
N
OBn
F3C
NO2
3f
f1 (ppm)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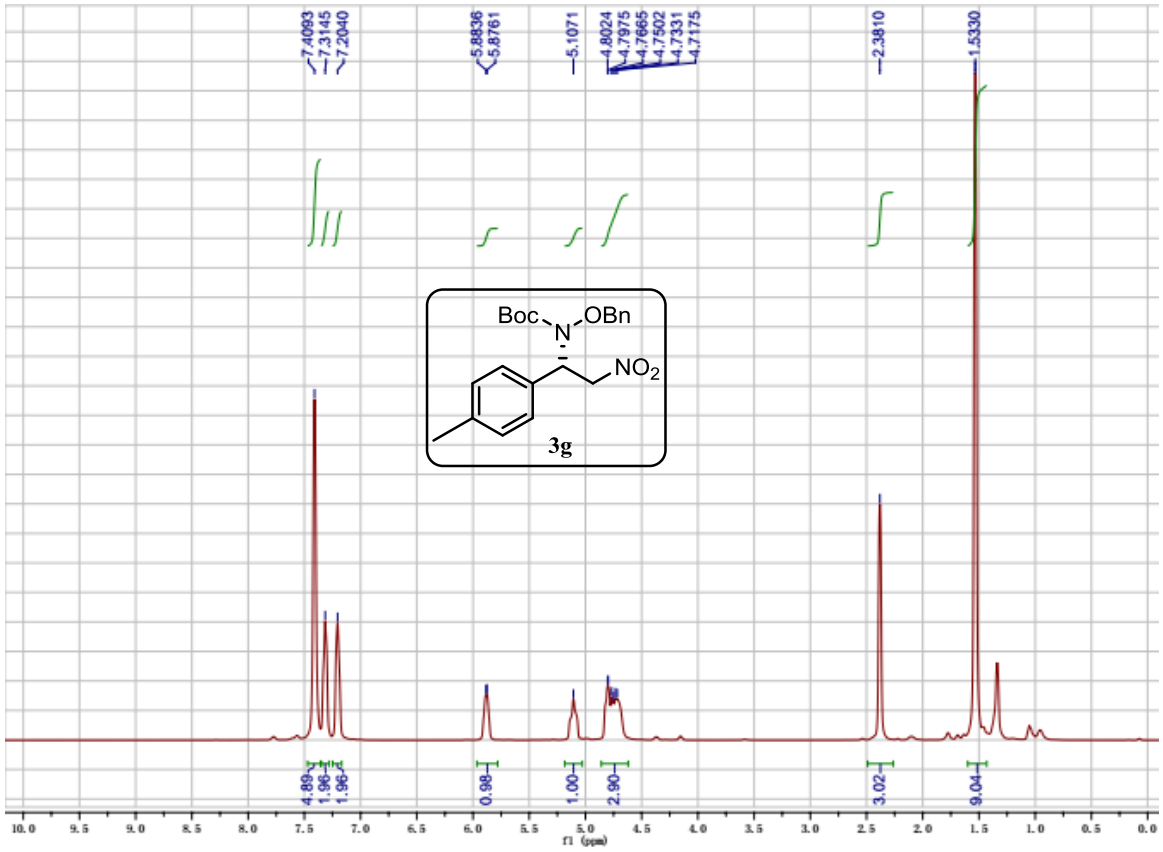
OBn
F3C
NO2
3f
f1 (ppm)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4.8024
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4.7665
4.7502
4.7331
4.7175
2.3810
1.5330
Boc
N
OBn
NO2
3g
4.89
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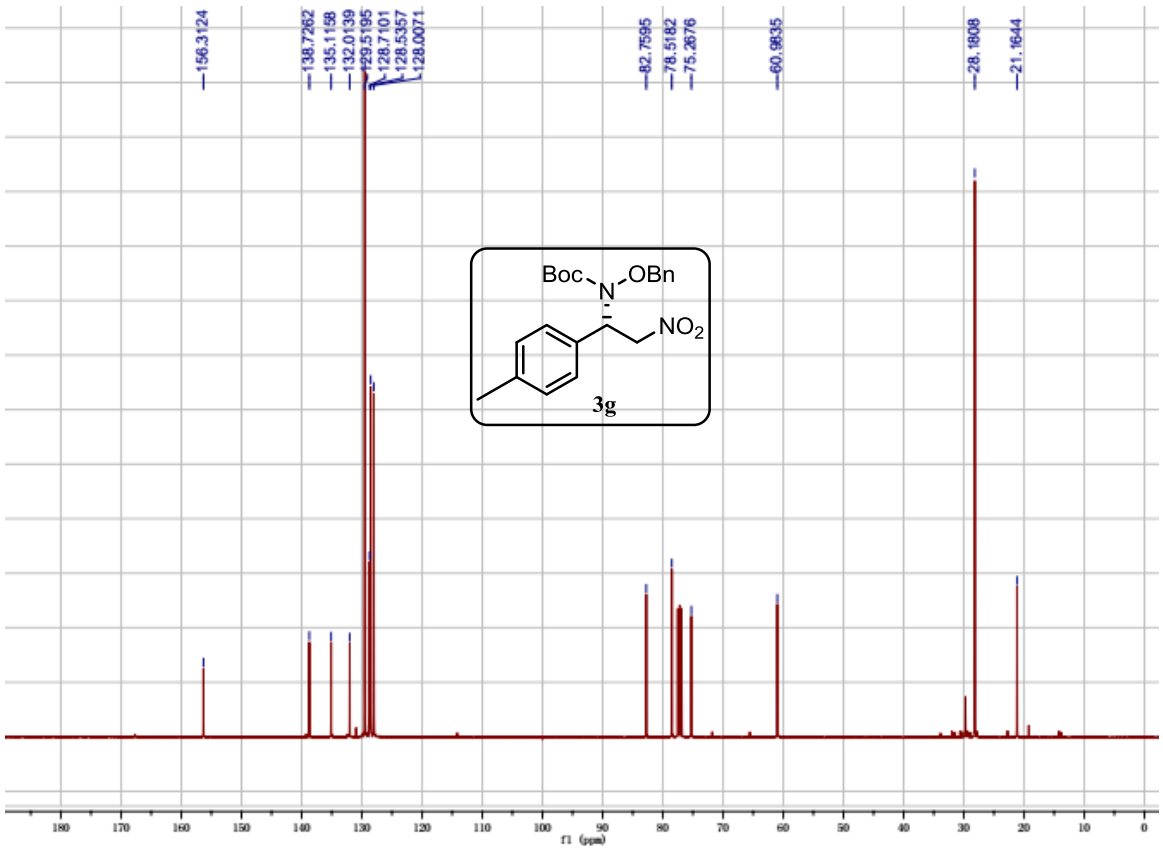
1.96
0.98
1.00
2.90
3.02
9.04
f1 (ppm)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135.1158
132.0139
129.5195
128.7101
128.5357
128.0071
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75.2676
60.9835
28.1808
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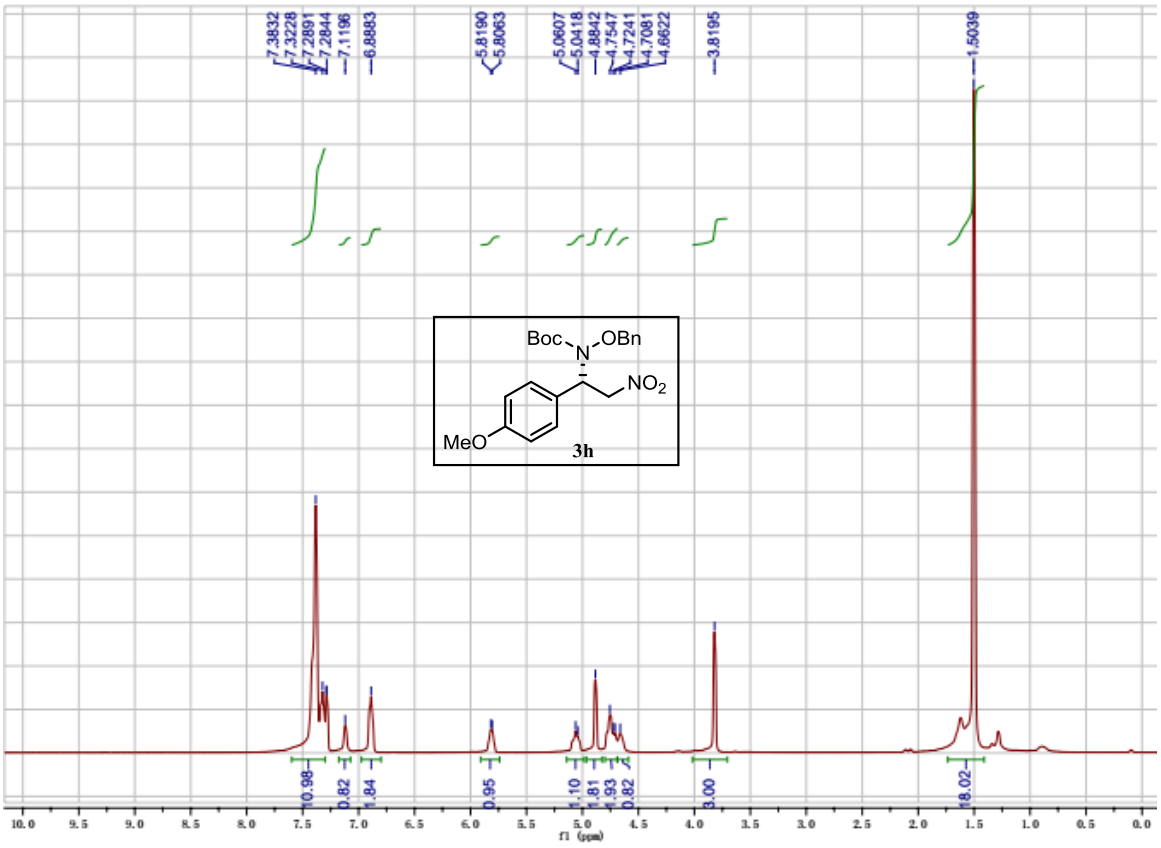
Boc
N
OBn
NO2
3g
f1 (ppm)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4.7547
4.7241
4.7081
4.6622
3.8195
1.5039
Boc
N
OBn
NO2
MeO
3h
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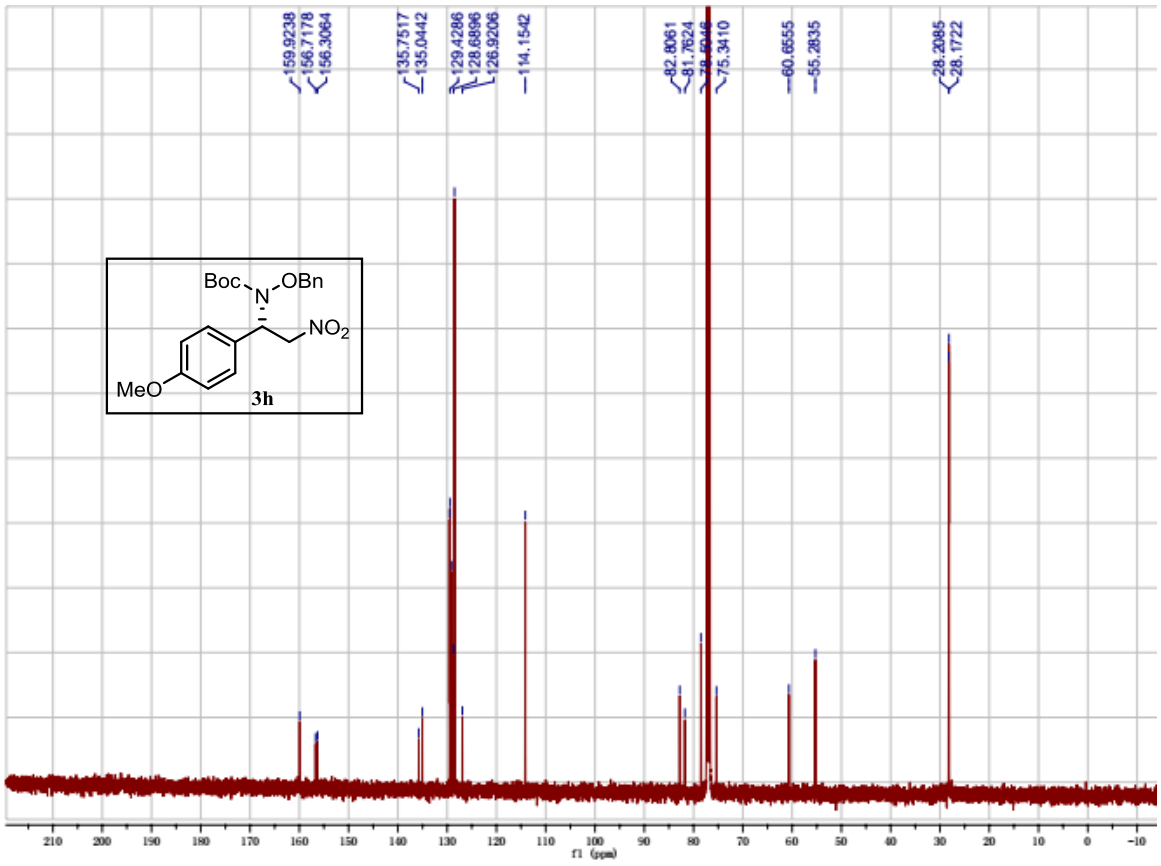
1.84
0.95
1.10
1.81
1.93
0.82
3.00
18.02
f1 (ppm)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81.7624
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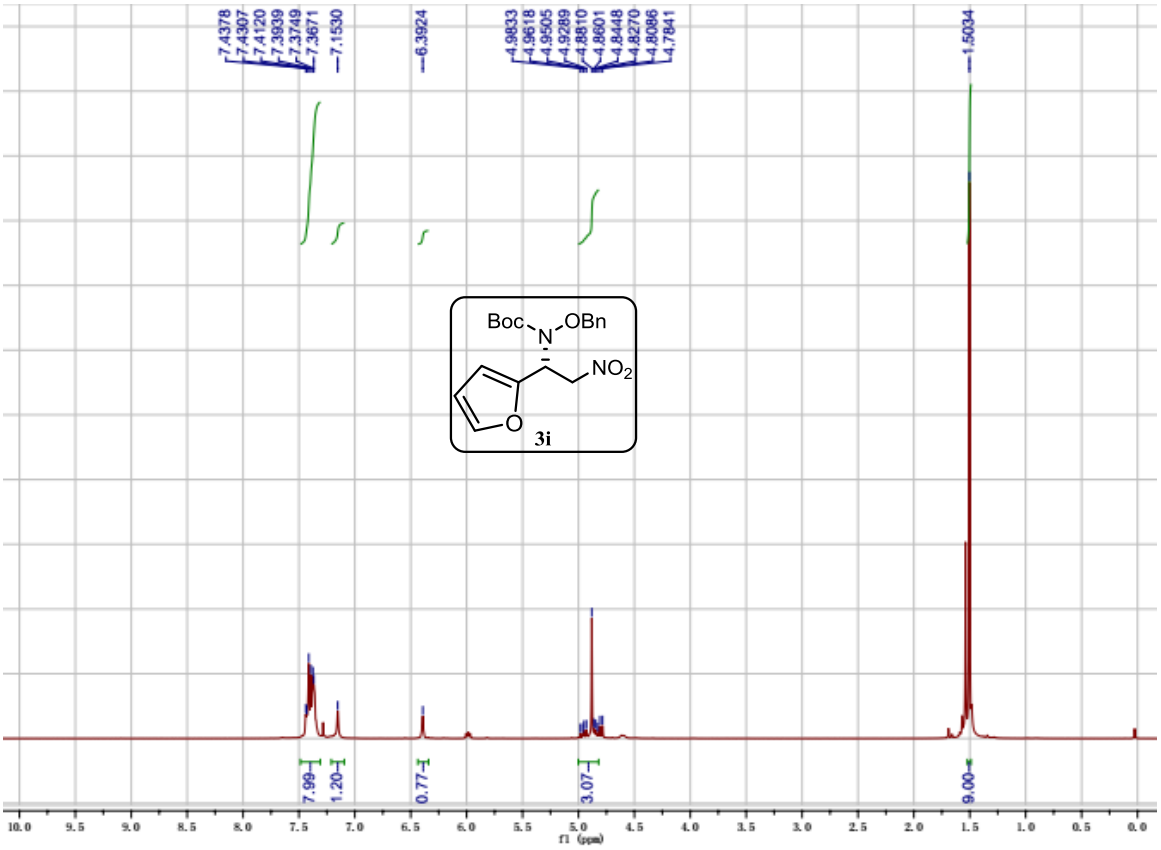
OBn
NO2
MeO
3h
f1 (ppm)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4.8601
4.8448
4.8270
4.8086
4.7841
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Boc
N
OBn
NO2
O
3i
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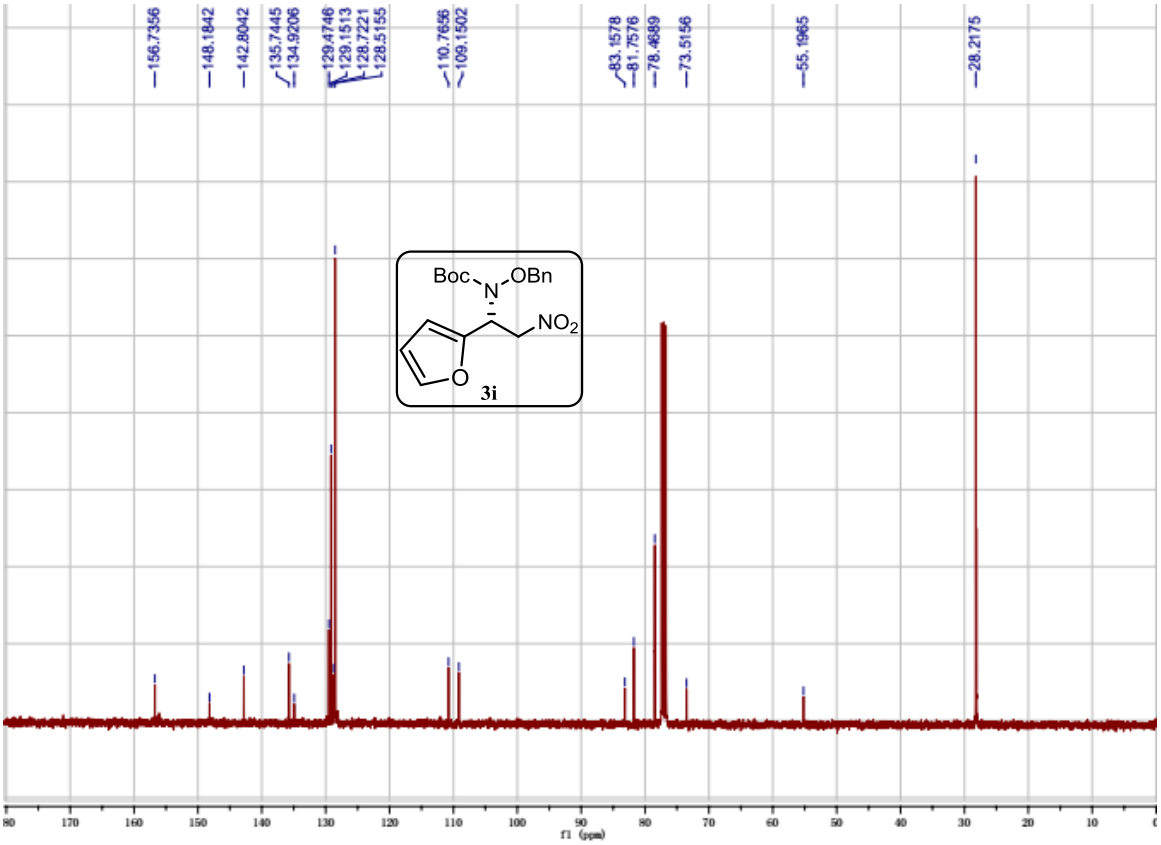
3.07
9.00
f1 (ppm)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83.1578
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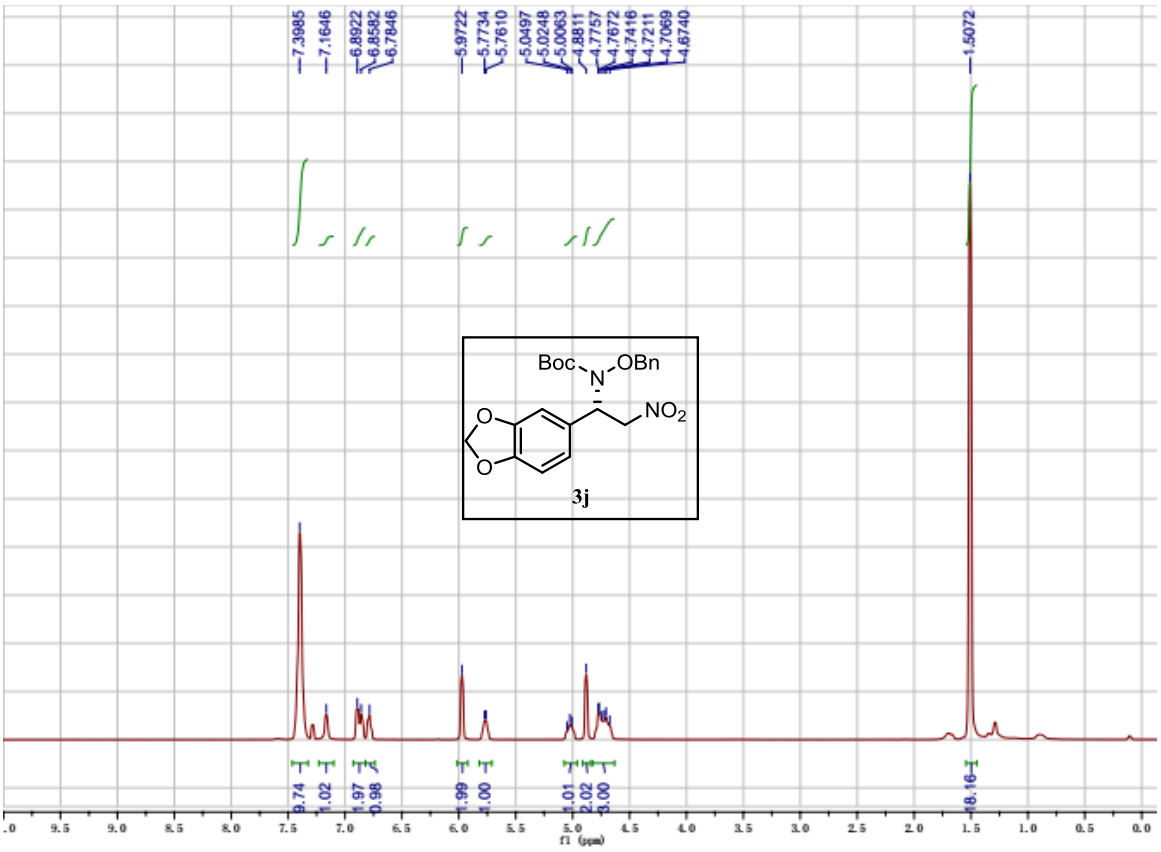
Boc
N
OBn
NO2
O
3i
f1 (ppm)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4.7069
4.6740
1.5072
Boc
N
OBn
NO2
O
O
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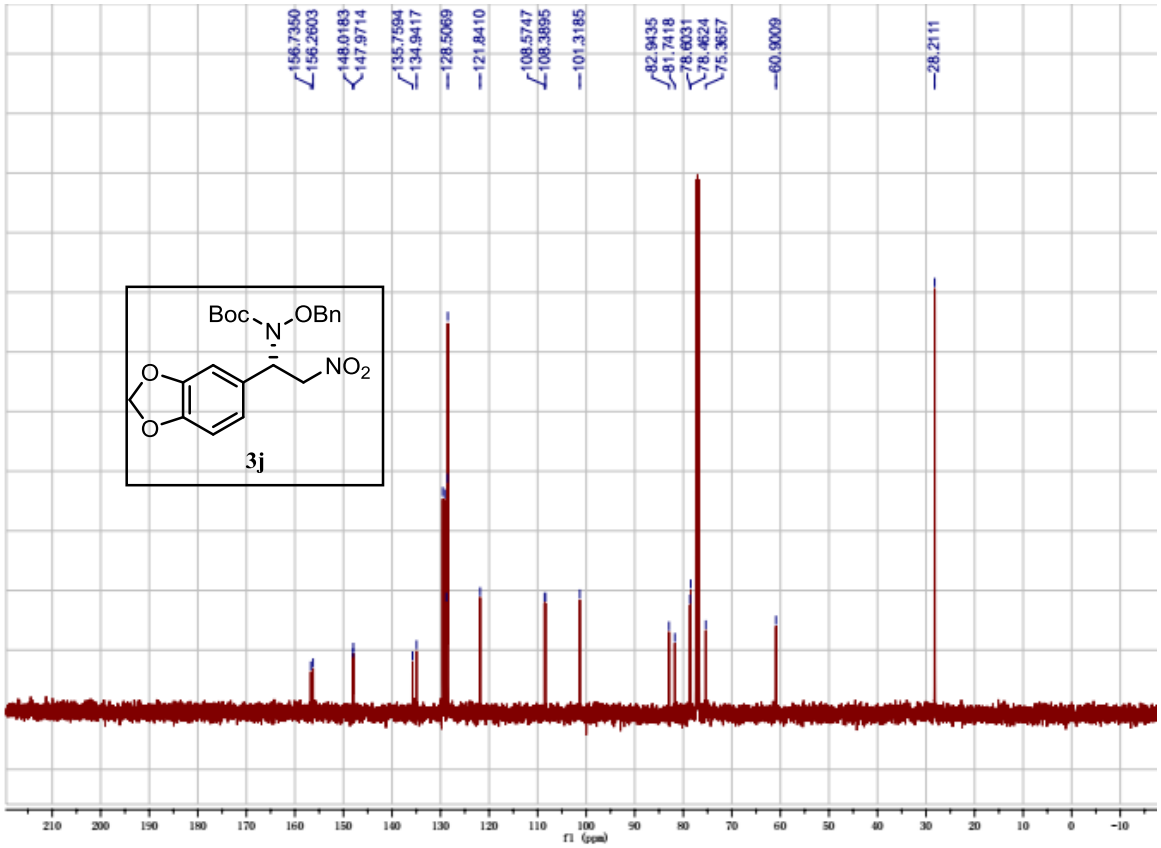
1.01
2.02
3.00
18.16
f1 (ppm)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82.9435
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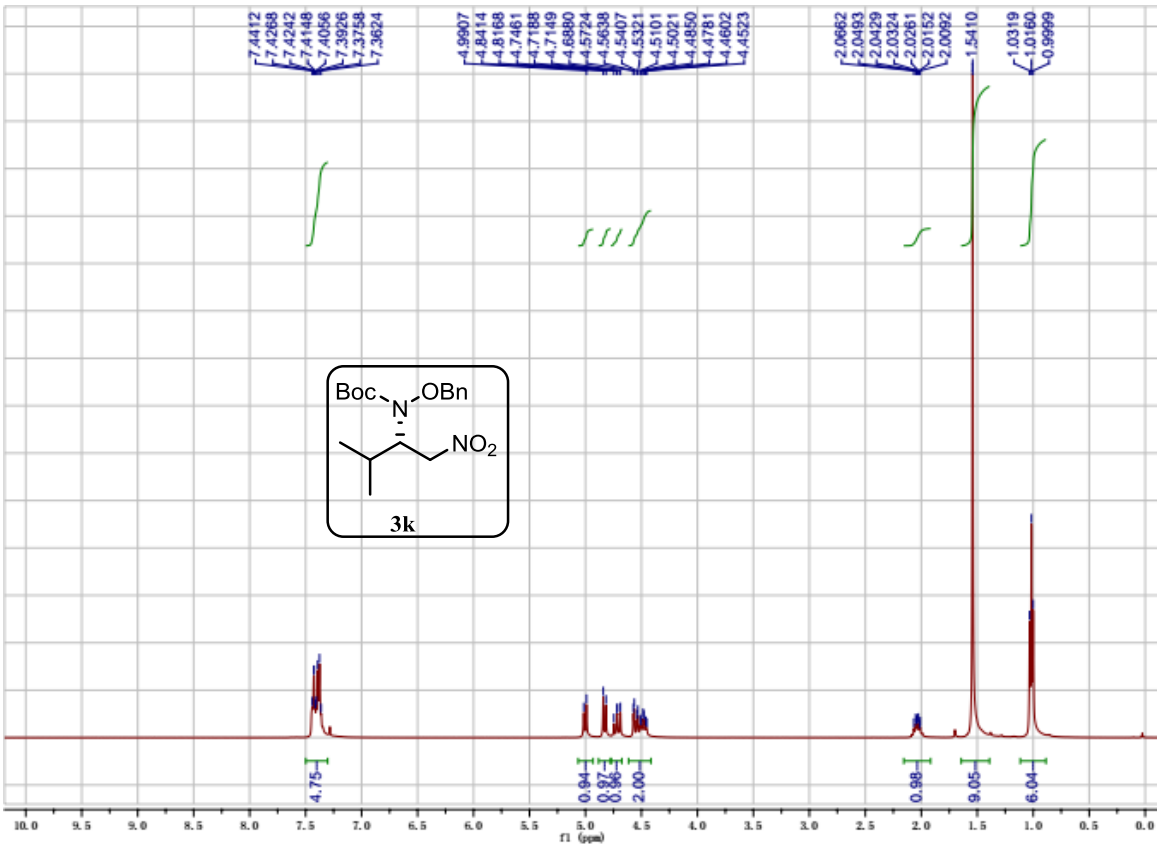
N
OBn
NO2
O
O
3j
f1 (ppm)


Boc
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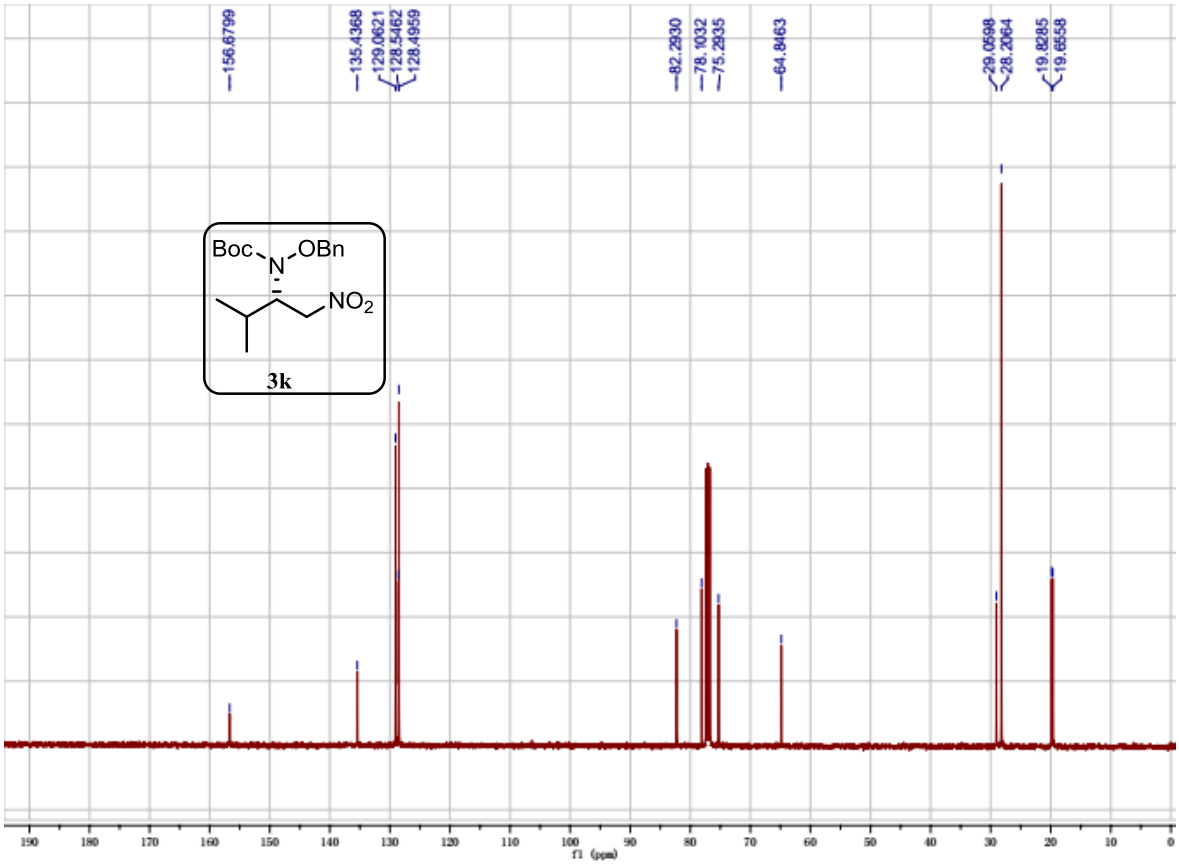

Boc
N
OBn
NO2
3k

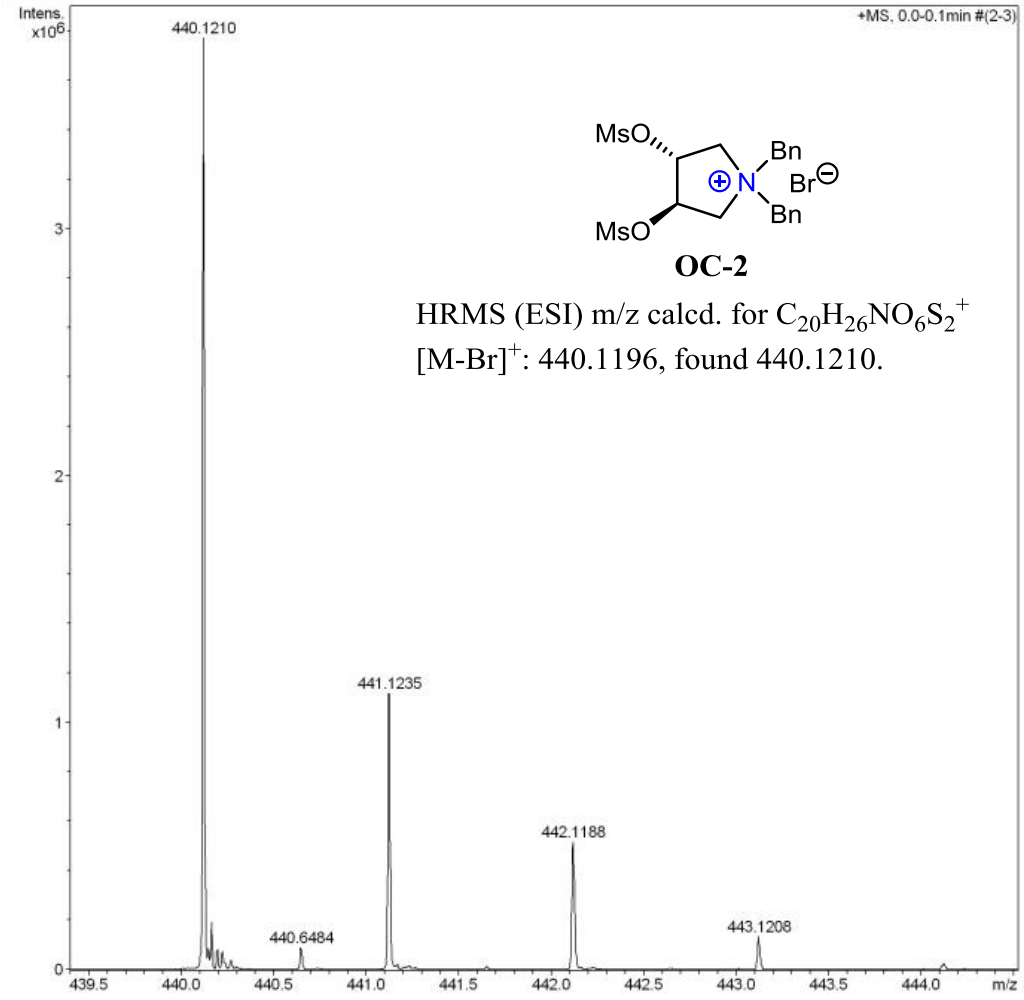
Display Report

Analysis Info

Analysis Name	D:\Data\FAN\data\2017\0911\lay-1.d	Acquisition Date	9/11/2017 10:07:53 AM
Method	pos_low-20151116.m	Operator	Fan
Sample Name	liuaiyun	Instrument	maXis 10103
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	180 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Waste



OC-2

HRMS (ESI) m/z calcd. for $C_{20}H_{26}NO_6S_2^+$ $[M\text{-}Br]^+$: 440.1196, found 440.1210.

Bruker Compass DataAnalysis 4.0 printed: 9/11/2017 10:08:51 AM Page 1 of 1

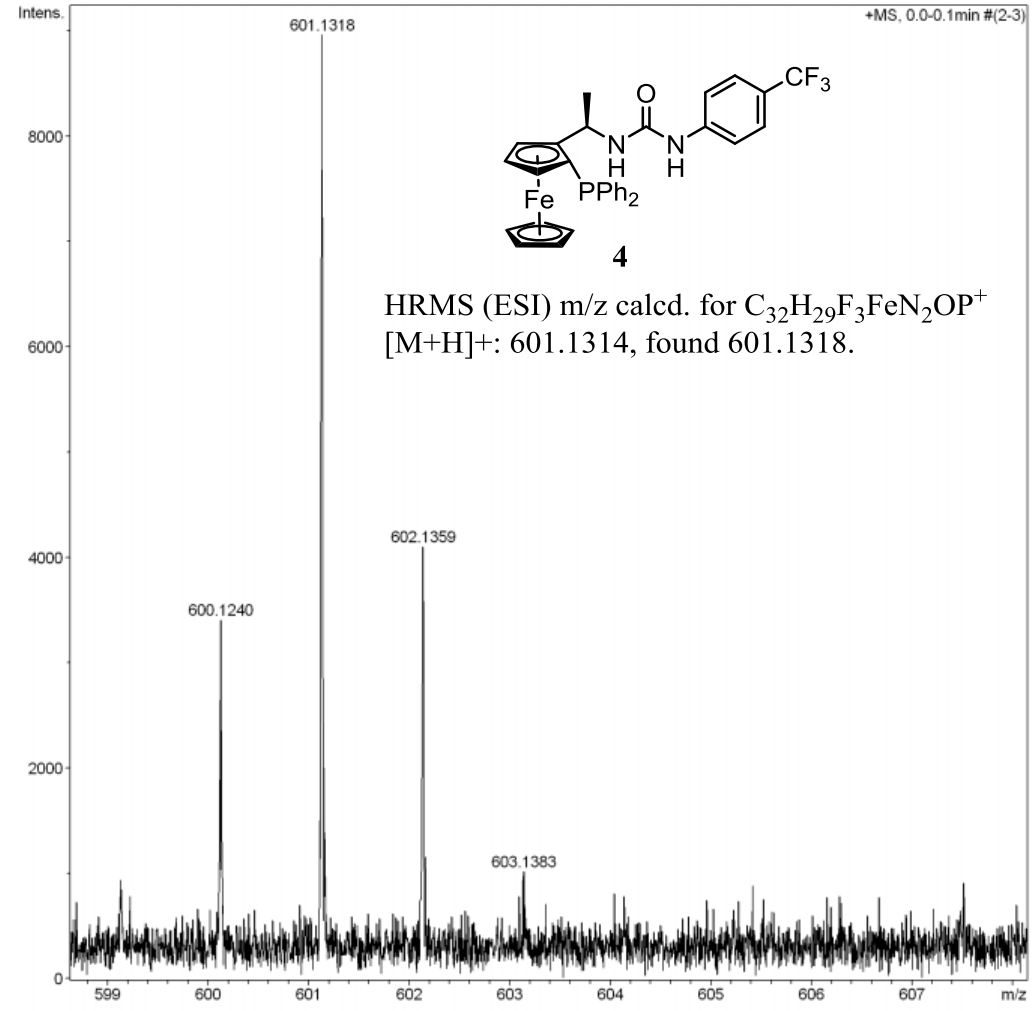
Display Report

Analysis Info

Analysis Name	D:\Data\FAN\data\2017\0911\lay-2.d	Acquisition Date	9/11/2017 10:10:46 AM
Method	pos_low-20151116.m	Operator	Fan
Sample Name	liuaiyun	Instrument	maXis 10103
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	180 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Waste



4

HRMS (ESI) m/z calcd. for $C_{32}H_{29}F_3FeN_2OP^+$ [M+H]+: 601.1314, found 601.1318.

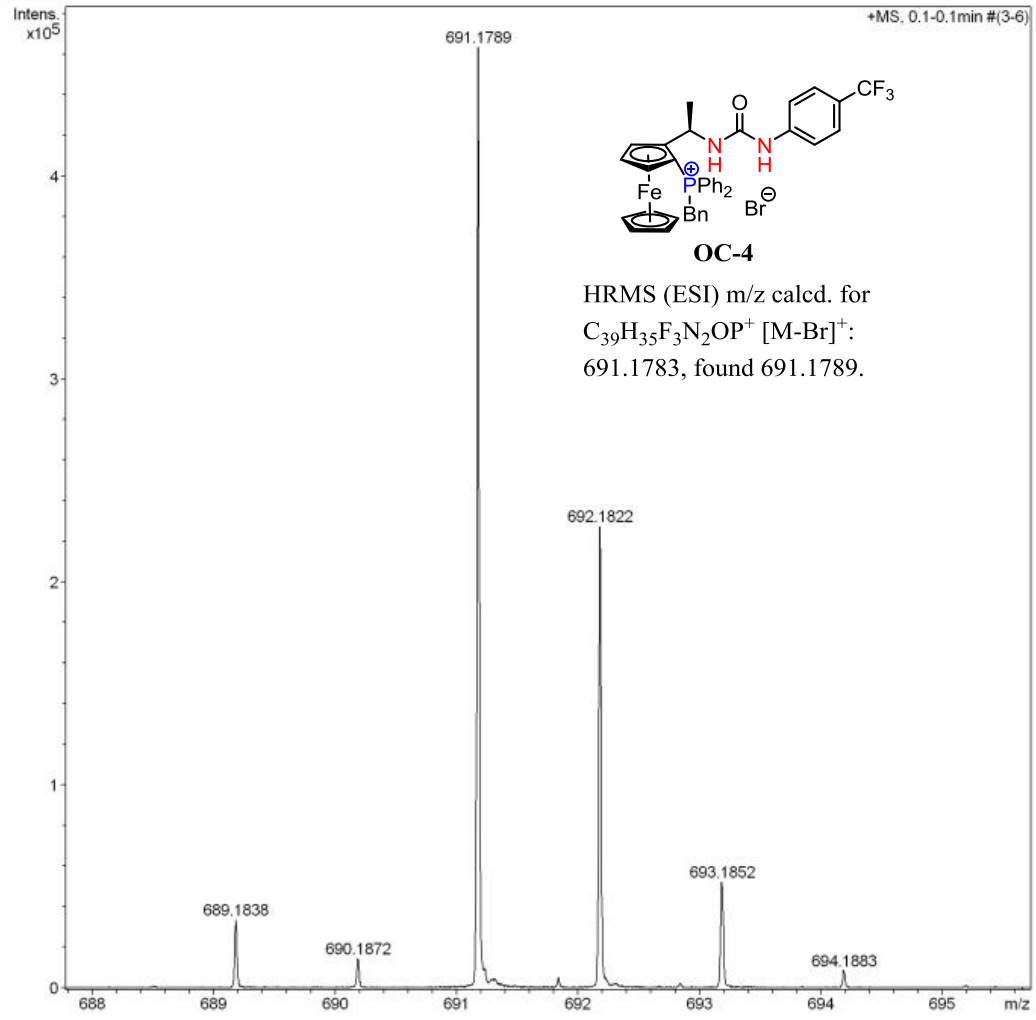
Display Report

Analysis Info

Analysis Name	D:\Data\FAN\data\2017\0707\ckh-2.d	Acquisition Date	7/7/2017 9:28:30 AM
Method	pos_low-20151116.m	Operator	Fan
Sample Name	caikaihua	Instrument	maXis 10103
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1200 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Waste



OC-4

HRMS (ESI) m/z calcd. for $C_{39}H_{35}F_3N_2OP^+$ $[M\text{-}Br]^+$: 691.1783, found 691.1789.

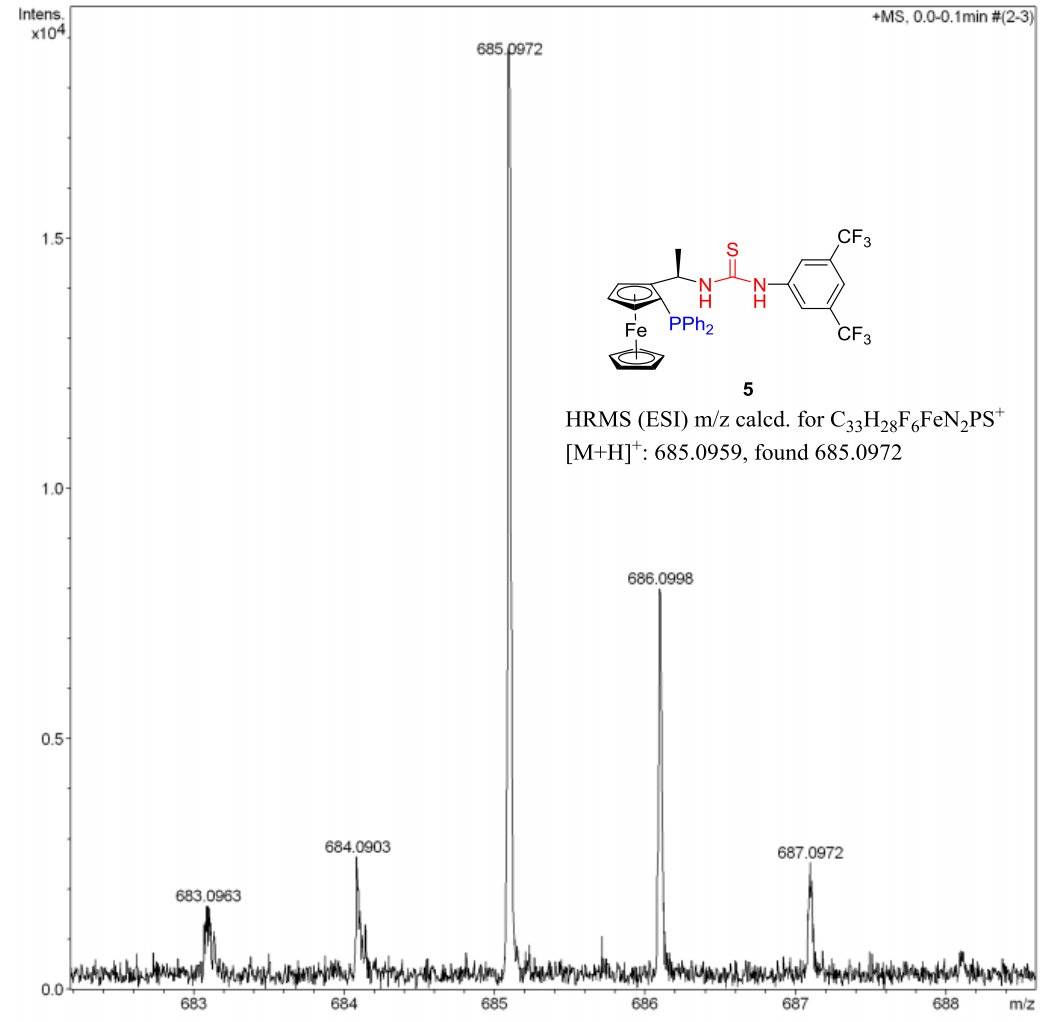
Display Report

Analysis Info

Analysis Name	D:\Data\FAN\data\2017\0911\lay-3a.d	Acquisition Date	9/11/2017 10:15:12 AM
Method	pos_low-20151116.m	Operator	Fan
Sample Name	liuaiyun	Instrument	maXis 10103
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	180 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Waste



5

HRMS (ESI) m/z calcd. for $C_{33}H_{28}F_6FeN_2PS^+$ $[M+H]^+$: 685.0959, found 685.0972

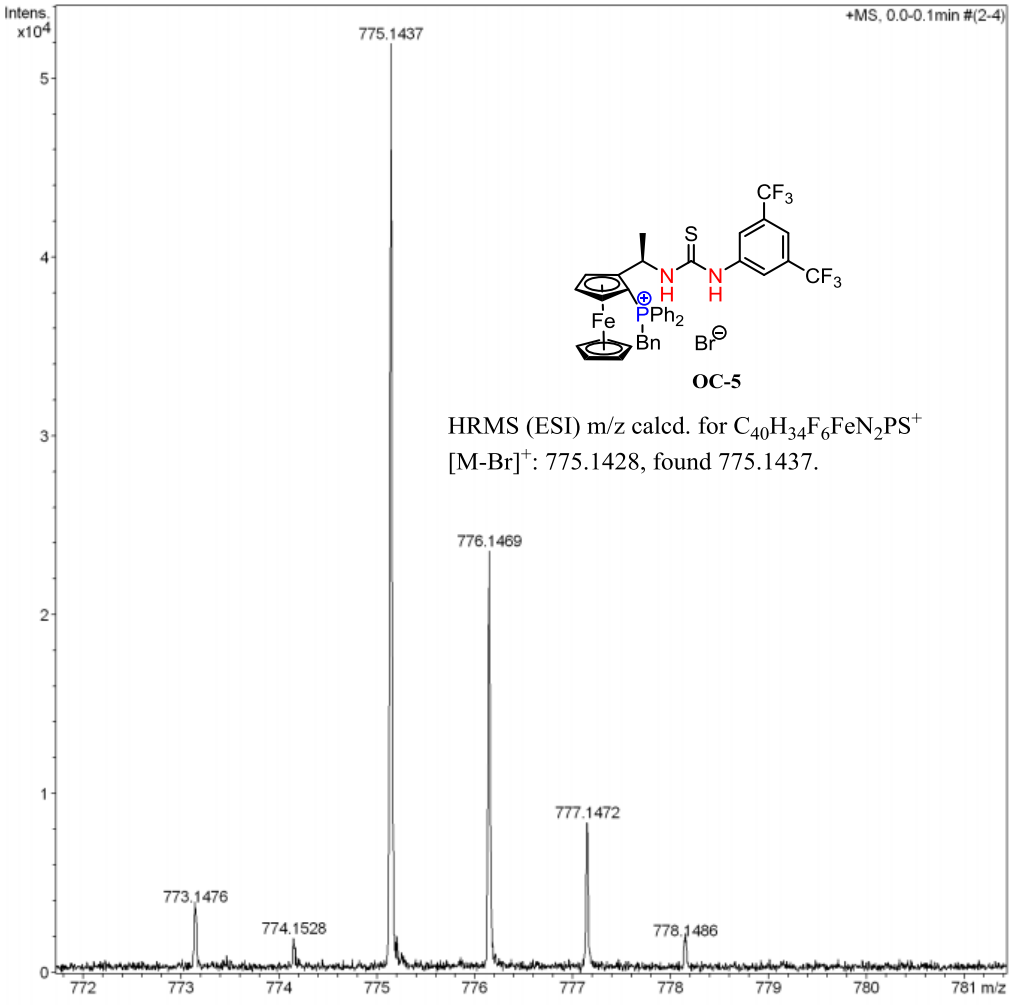
Display Report

Analysis Info

Analysis Name	D:\Data\FAN\data\2017\0707\ckh-3a.d	Acquisition Date	7/7/2017 9:34:49 AM
Method	pos_low-20151116.m	Operator	Fan
Sample Name	caikaihua	Instrument	maXis 10103
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1200 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Waste



OC-5

HRMS (ESI) m/z calcd. for $C_{40}H_{34}F_6FeN_2PS^+$ $[M\text{-}Br]^+$: 775.1428, found 775.1437.

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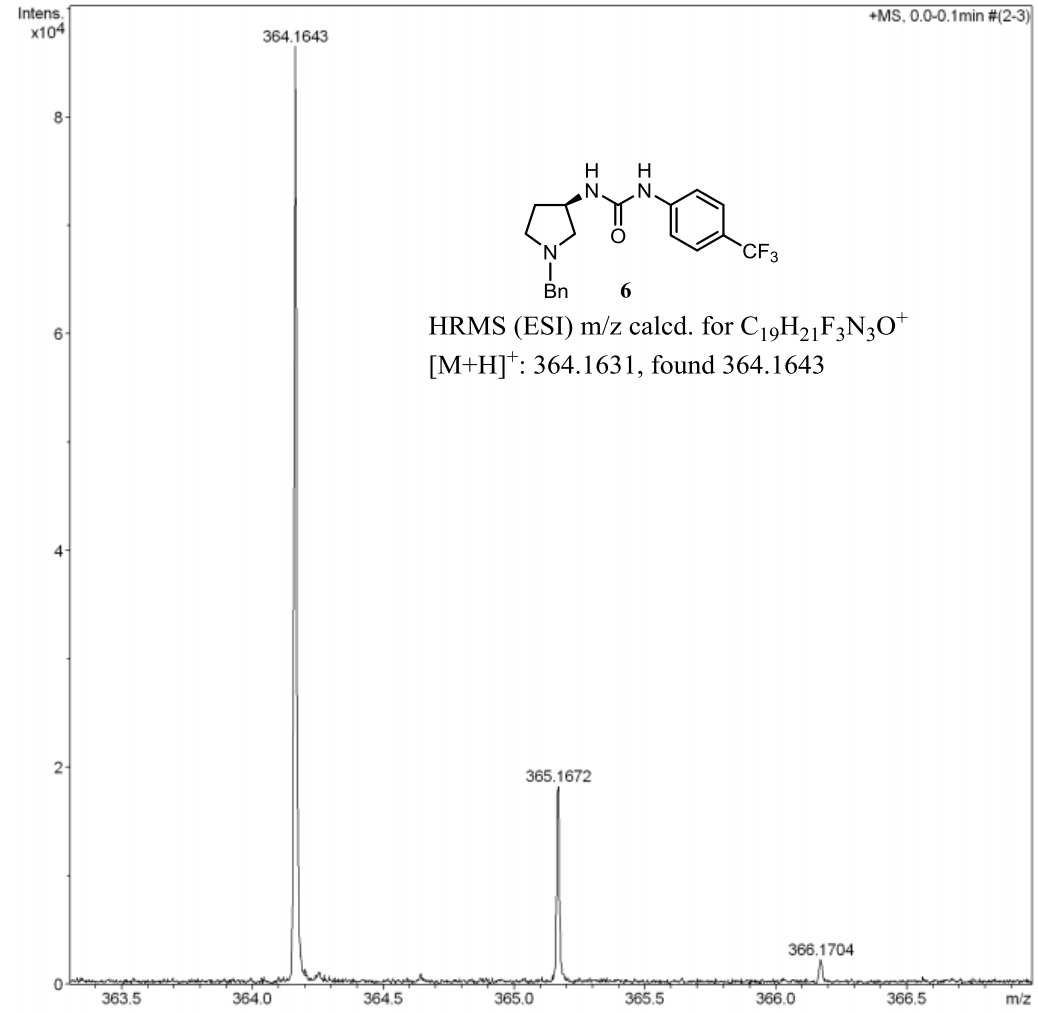
Display Report

Analysis Info

Analysis Name	D:\Data\FAN\data\2017\0911\lay-4a.d	Acquisition Date	9/11/2017 10:19:23 AM
Method	pos_low-20151116.m	Operator	Fan
Sample Name	liuaiyun	Instrument	maXis 10103
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	180 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Waste



6

HRMS (ESI) m/z calcd. for $C_{19}H_{21}F_3N_3O^+$ $[M+H]^+$: 364.1631, found 364.1643

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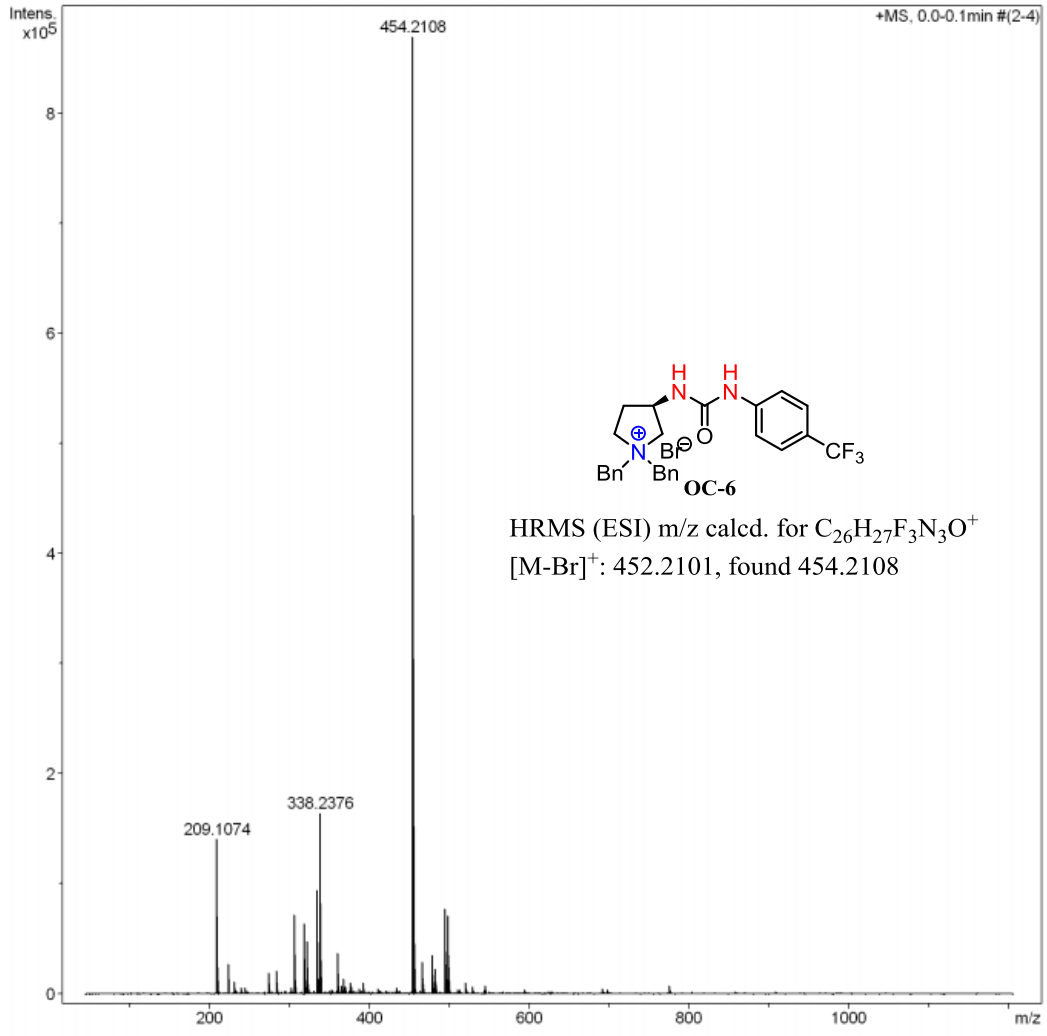
Display Report

Analysis Info

Analysis Name	D:\Data\FAN\data\2017\0707\ckh-4.d	Acquisition Date	7/7/2017 9:42:07 AM
Method	pos_low-20151116.m	Operator	Fan
Sample Name	caikaihua	Instrument	maXis 10103
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1200 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Waste



OC-6

HRMS (ESI) m/z calcd. for $C_{26}H_{27}F_3N_3O^+$ $[M\text{-}Br]^+$: 452.2101, found 454.2108

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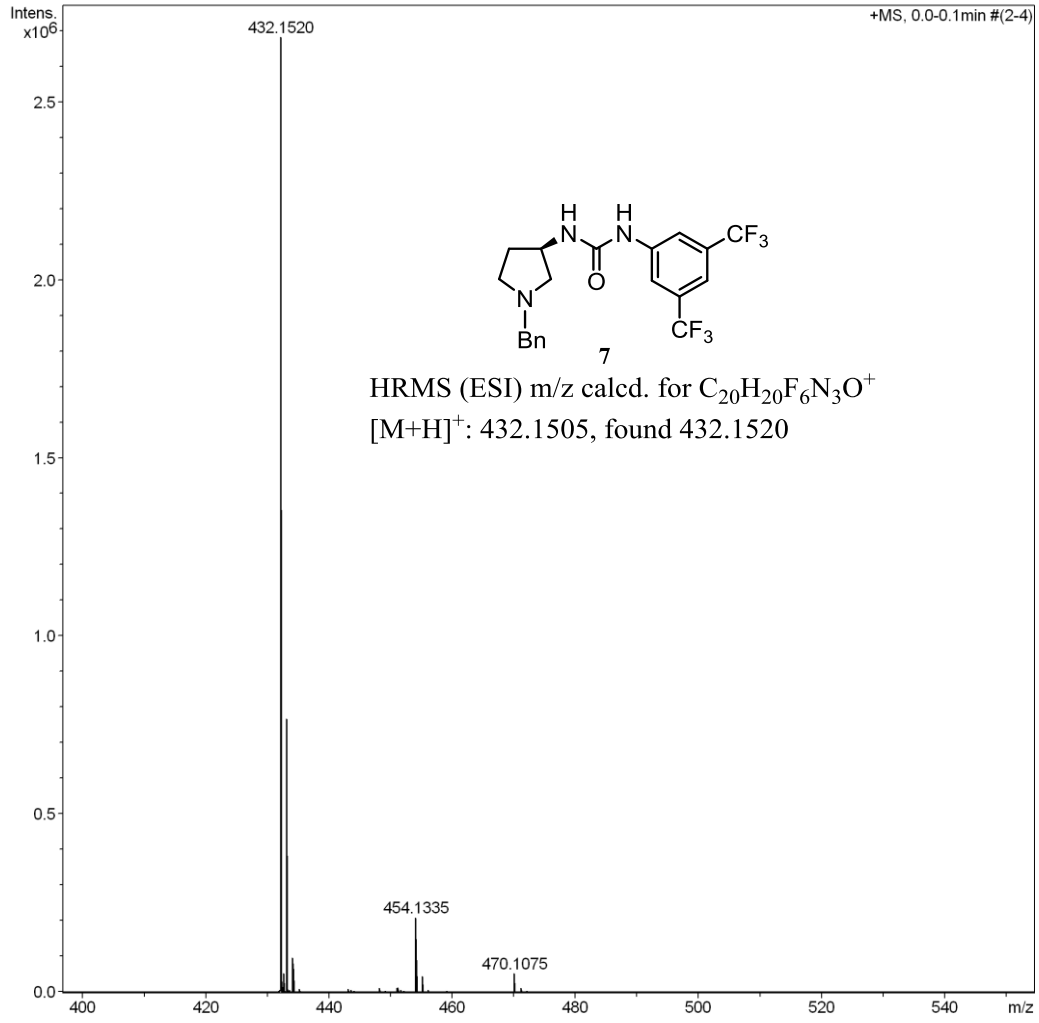
Display Report

Analysis Info

Analysis Name	D:\Data\FAN\data\2017\0911\lay-5.d	Acquisition Date	9/11/2017 10:20:59 AM
Method	pos_low-20151116.m	Operator	Fan
Sample Name	liuaiyun	Instrument	maXis 10103
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	180 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Waste



7

HRMS (ESI) m/z calcd. for $C_{20}H_{20}F_6N_3O^+$ $[M+H]^+$: 432.1505, found 432.1520

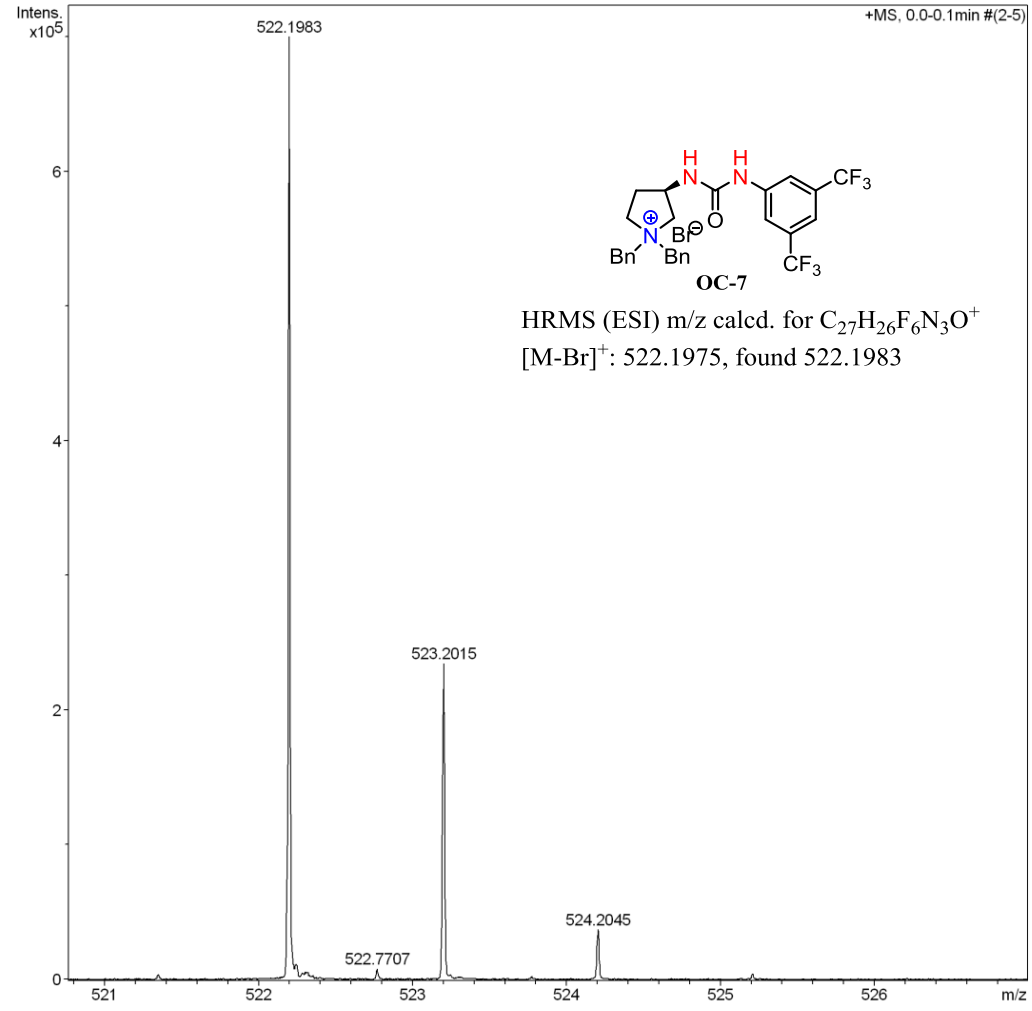
Display Report

Analysis Info

Analysis Name	D:\Data\FAN\data\2017\0707\ckh-5a.d	Acquisition Date	7/7/2017 9:46:12 AM
Method	pos_low-20151116.m	Operator	Fan
Sample Name	caikaihua	Instrument	maXis 10103
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1200 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Waste



OC-7

HRMS (ESI) m/z calcd. for $C_{27}H_{26}F_6N_3O^+$ $[M\text{-}Br]^+$: 522.1975, found 522.1983

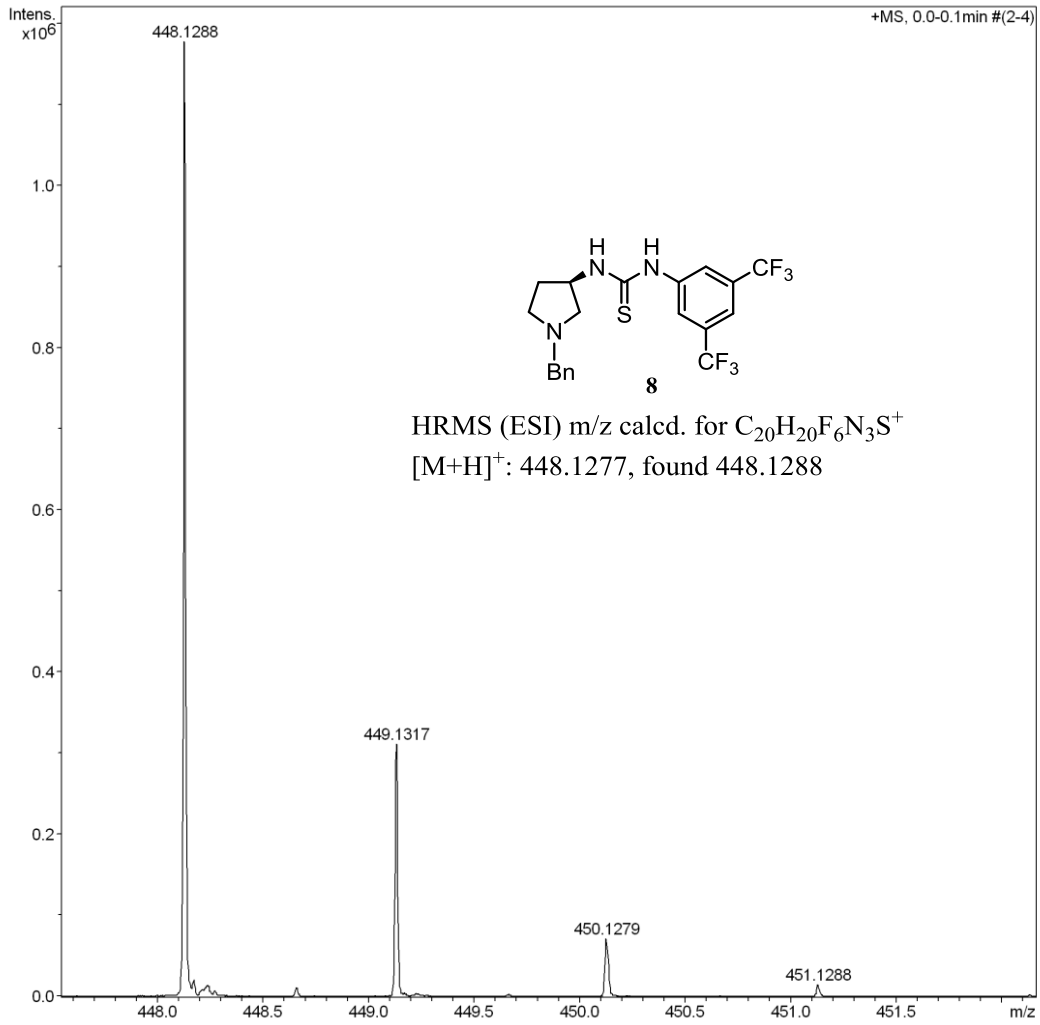
Display Report

Analysis Info

Analysis Name	D:\Data\FAN\data\2017\0911\lay-6.d	Acquisition Date	9/11/2017 10:24:26 AM
Method	pos_low-20151116.m	Operator	Fan
Sample Name	liuaiyun	Instrument	maXis 10103
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	180 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Waste



8

HRMS (ESI) m/z calcd. for $C_{20}H_{20}F_6N_3S^+$ $[M+H]^+$: 448.1277, found 448.1288

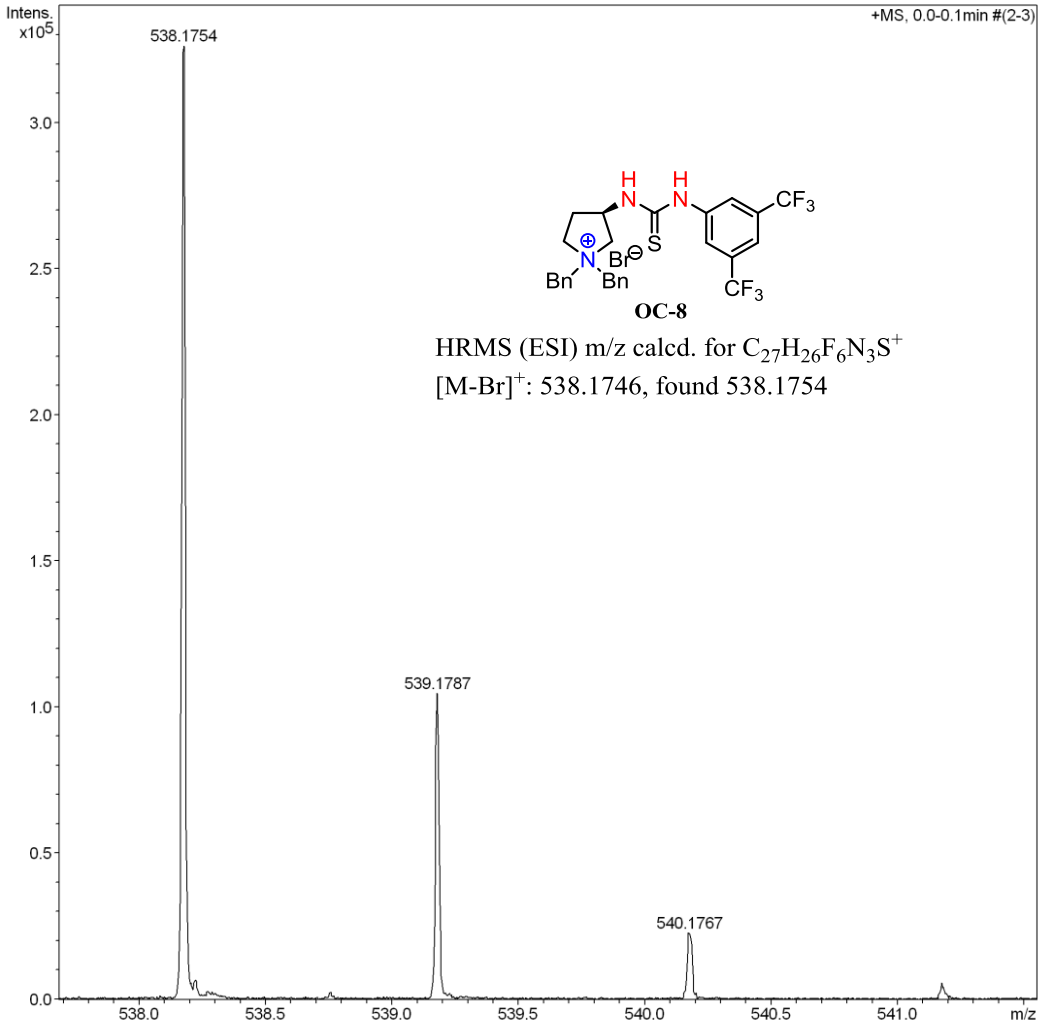
Display Report

Analysis Info

Analysis Name	D:\Data\FAN\data\2017\0707\ckh-6.d	Acquisition Date	7/7/2017 9:50:56 AM	
Method	pos_low-20151116.m	Operator	Fan	
Sample Name	caikaihua	Instrument	maXis	10103
Comment				

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1200 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Waste



OC-8

HRMS (ESI) m/z calcd. for $C_{27}H_{26}F_6N_3S^+$ $[M-Br]^+$: 538.1746, found 538.1754

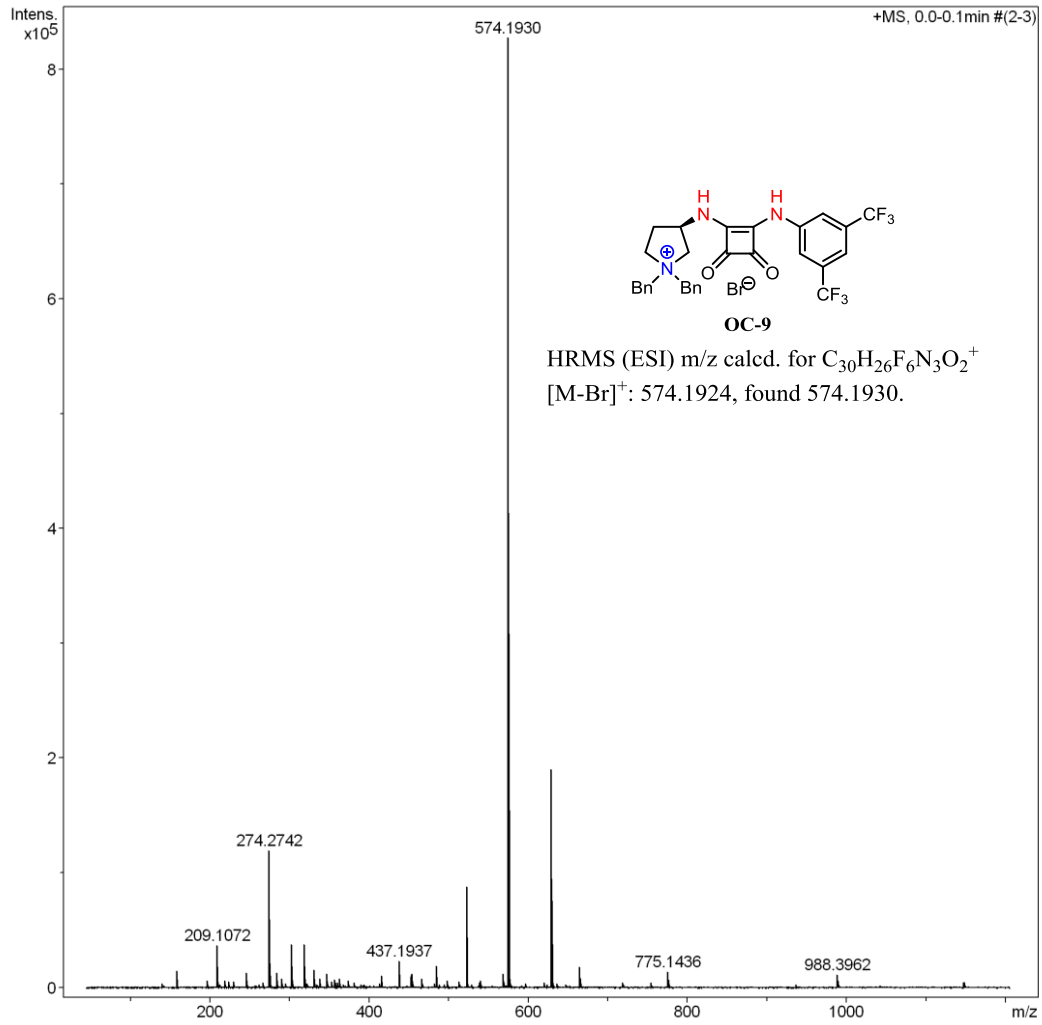
Display Report

Analysis Info

Analysis Name	D:\Data\FAN\data\2017\0707\ckh-7.d	Acquisition Date	7/7/2017 9:53:50 AM
Method	pos_low-20151116.m	Operator	Fan
Sample Name	caikaihua	Instrument	maXis 10103
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1200 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Waste



OC-9

HRMS (ESI) m/z calcd. for $C_{30}H_{26}F_6N_3O_2^+$ $[M\text{-}Br]^+$: 574.1924, found 574.1930.

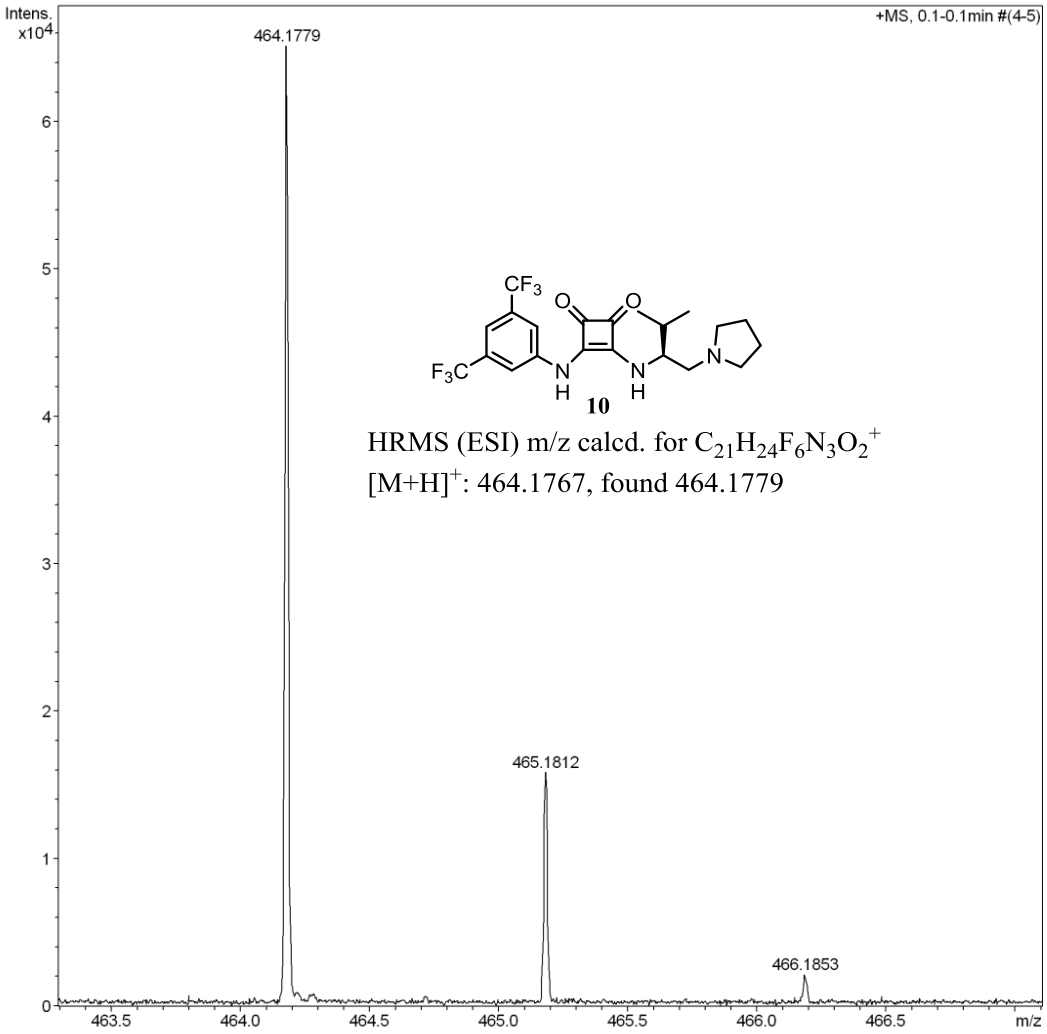
Display Report

Analysis Info

Analysis Name	D:\Data\FAN\data\2017\0911\lay-8.d	Acquisition Date	9/11/2017 10:35:30 AM
Method	pos_low-20151116.m	Operator	Fan
Sample Name	liuaiyun	Instrument	maXis 10103
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	180 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Waste



10

HRMS (ESI) m/z calcd. for $C_{21}H_{24}F_6N_3O_2^+$ $[M+H]^+$: 464.1767, found 464.1779

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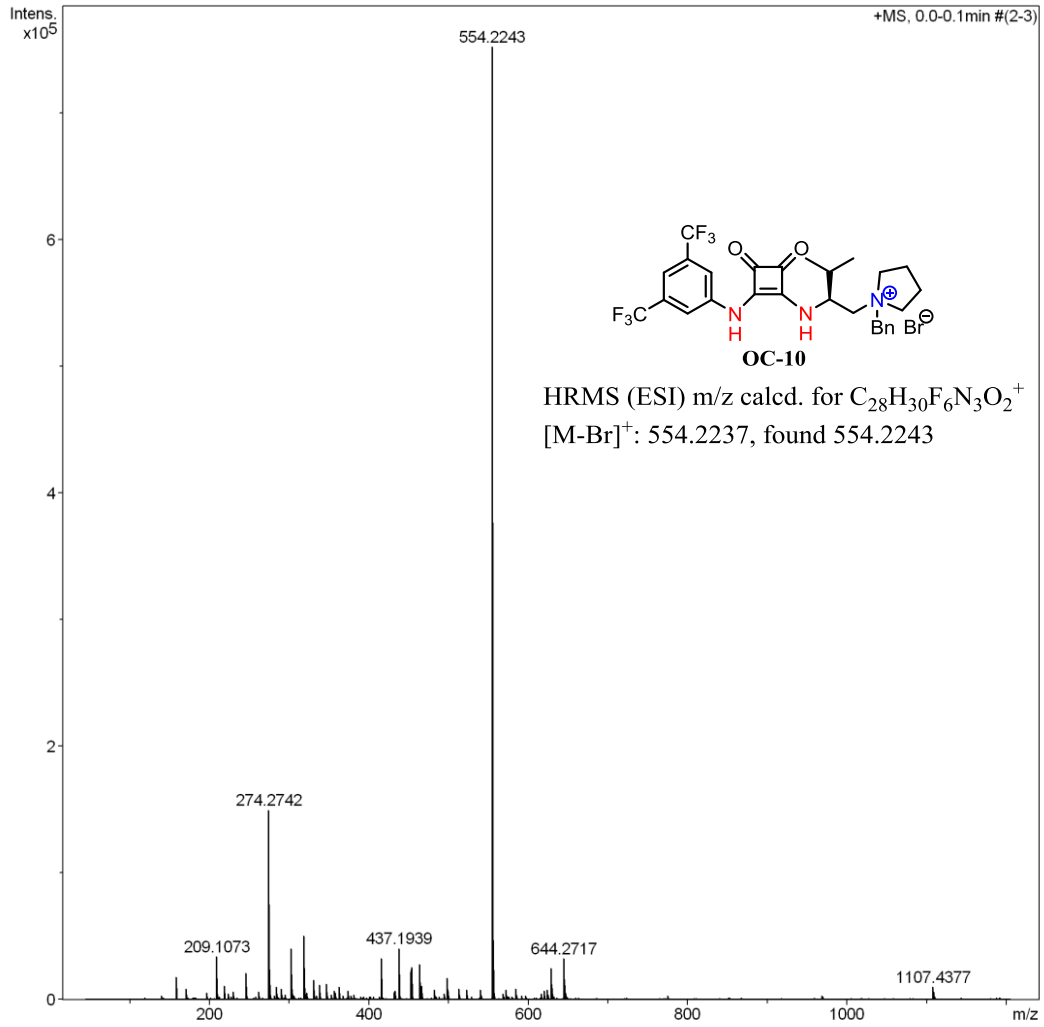
Analysis Info

Analysis Name	D:\Data\FAN\data\2017\0707\ckh-8.d	Acquisition Date	7/7/2017 9:57:02 AM
Method	pos_low-20151116.m	Operator	Fan
Sample Name	caikaihua	Instrument	maXis 10103
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1200 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Waste



OC-10

HRMS (ESI) m/z calcd. for $C_{28}H_{30}F_6N_3O_2^+$ [M-Br]$^+$: 554.2237, found 554.2243

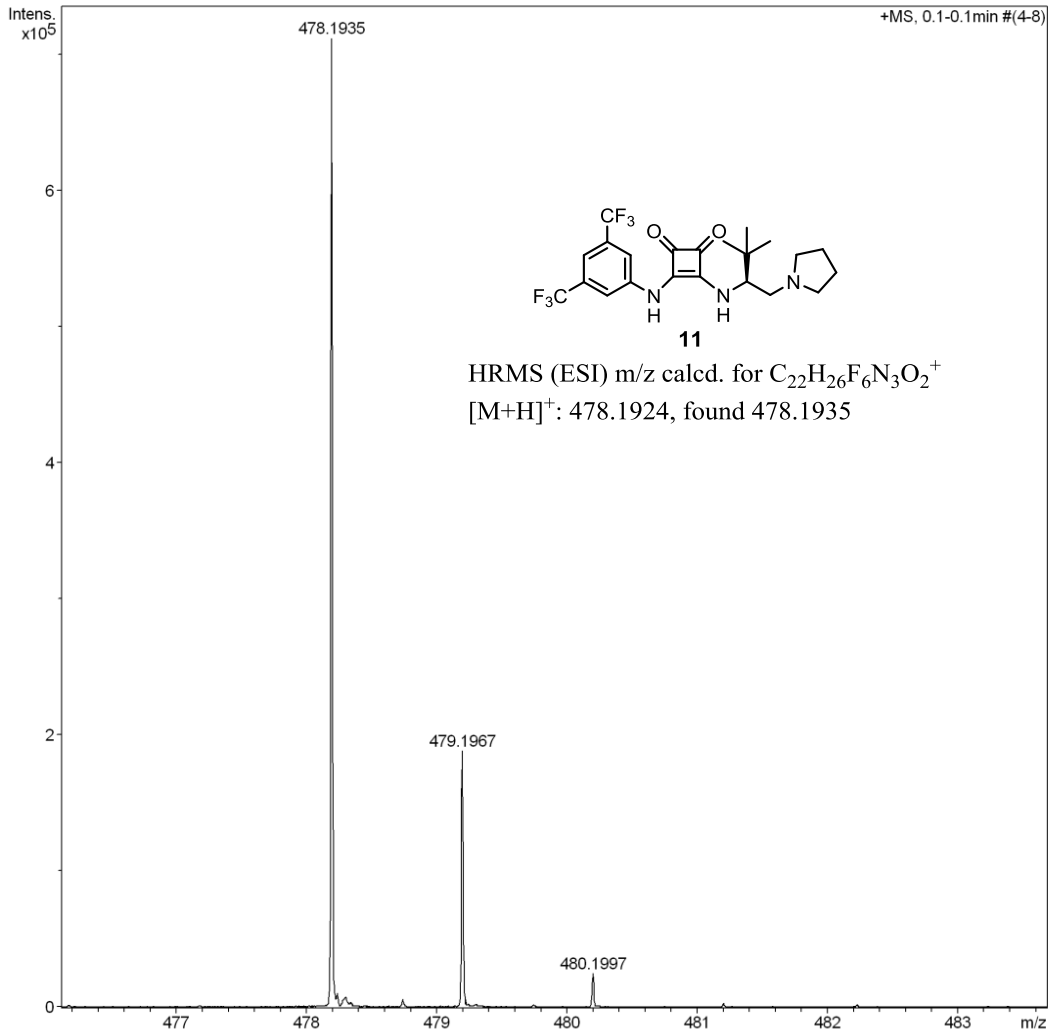
Display Report

Analysis Info

Analysis Name	D:\Data\FAN\data\2017\0911\lay-9.d	Acquisition Date	9/11/2017 10:39:06 AM
Method	pos_low-20151116.m	Operator	Fan
Sample Name	liuaiyun	Instrument	maXis 10103
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	180 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Waste



11

HRMS (ESI) m/z calcd. for $C_{22}H_{26}F_6N_3O_2^+$ $[M+H]^+$: 478.1924, found 478.1935

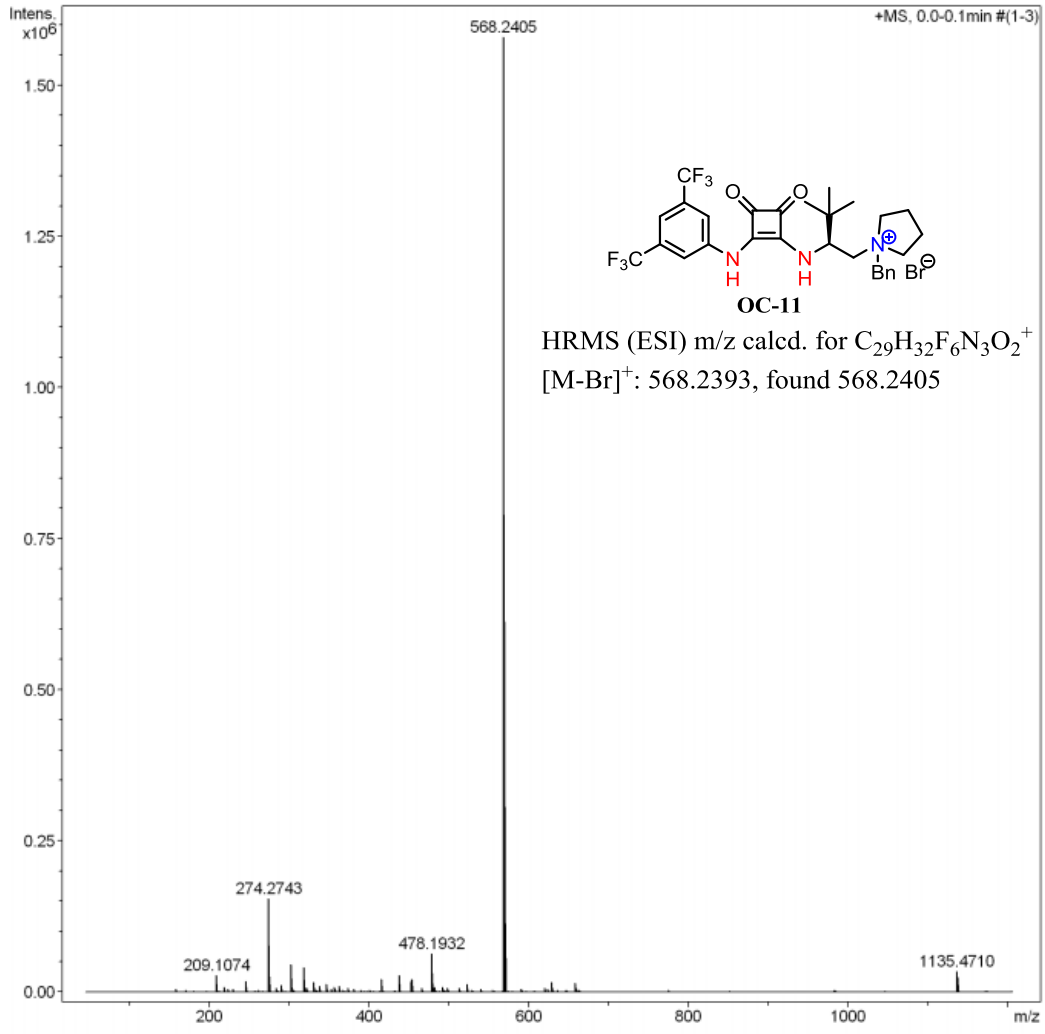
Display Report

Analysis Info

Analysis Name	D:\Data\FAN\data\2017\0707\ckh-9.d	Acquisition Date	7/7/2017 9:58:56 AM
Method	pos_low-20151116.m	Operator	Fan
Sample Name	caikaihua	Instrument	maXis 10103
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1200 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Waste



OC-11

HRMS (ESI) m/z calcd. for $C_{29}H_{32}F_6N_3O_2^+$ $[M-Br]^+$: 568.2393, found 568.2405

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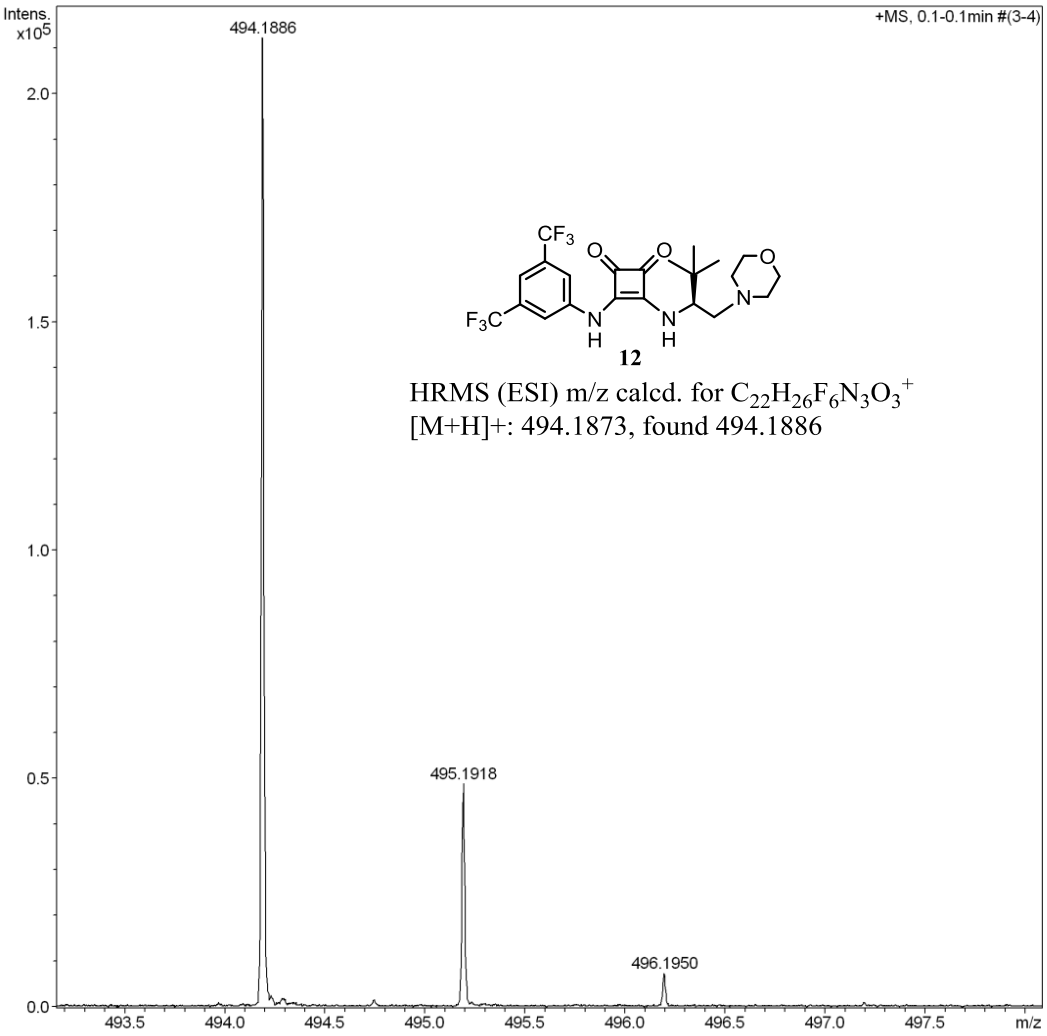
Display Report

Analysis Info

Analysis Name	D:\Data\FAN\data\2017\0911\lay-10.d	Acquisition Date	9/11/2017 10:43:18 AM
Method	pos_low-20151116.m	Operator	Fan
Sample Name	liuaiyun	Instrument	maXis 10103
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	180 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Waste



12

HRMS (ESI) m/z calcd. for $C_{22}H_{26}F_6N_3O_3^+$ [M+H]+: 494.1873, found 494.1886

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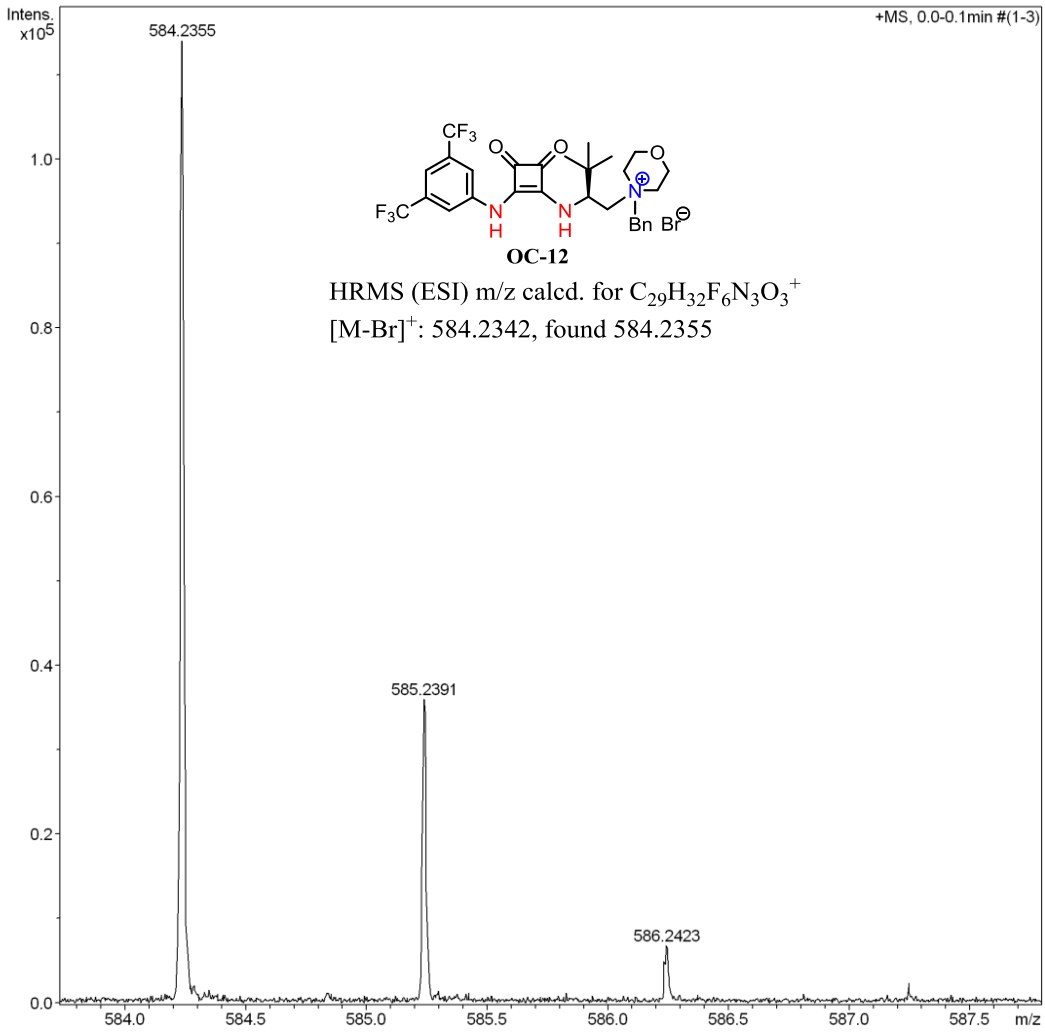
Display Report

Analysis Info

Analysis Name	D:\Data\FAN\data\2017\0707\ckh-10.d	Acquisition Date	7/7/2017 10:01:45 AM
Method	pos_low-20151116.m	Operator	Fan
Sample Name	caikaihua	Instrument	maXis 10103
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1200 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Waste



OC-12

HRMS (ESI) m/z calcd. for $C_{29}H_{32}F_6N_3O_3^+$ $[M\text{-}Br]^+$: 584.2342, found 584.2355

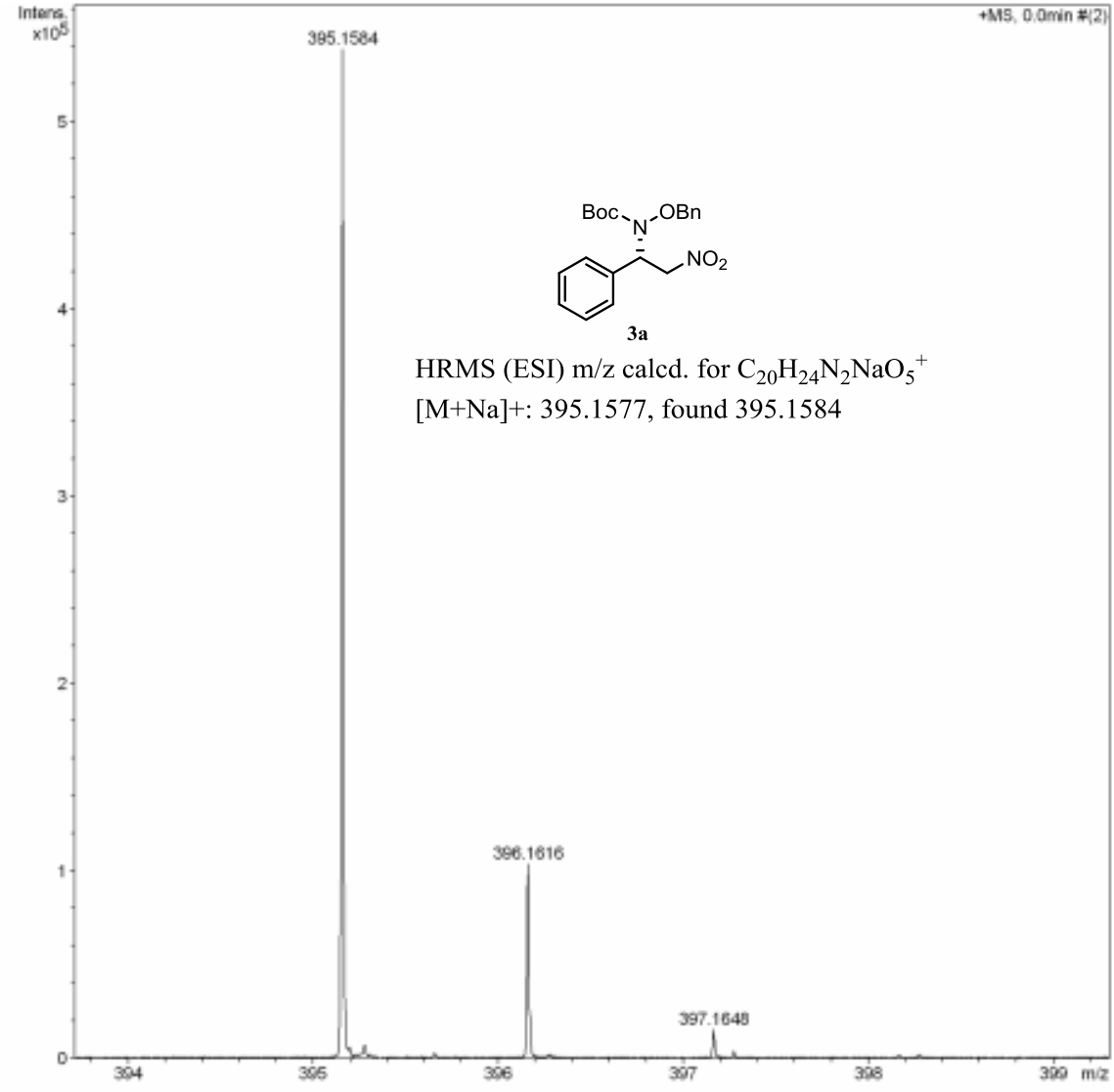
Display Report

Analysis Info

Analysis Name	D:\Data\2018\0112\lay-1.d	Acquisition Date	1/12/2018 9:40:38 AM
Method	pos_low-20151116.m	Operator	Fan
Sample Name	liusaiyun	Instrument	maXis 10103
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	180 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Waste



3a

HRMS (ESI) m/z calcd. for $C_{20}H_{24}N_2NaO_5^+$ [M+Na]+: 395.1577, found 395.1584

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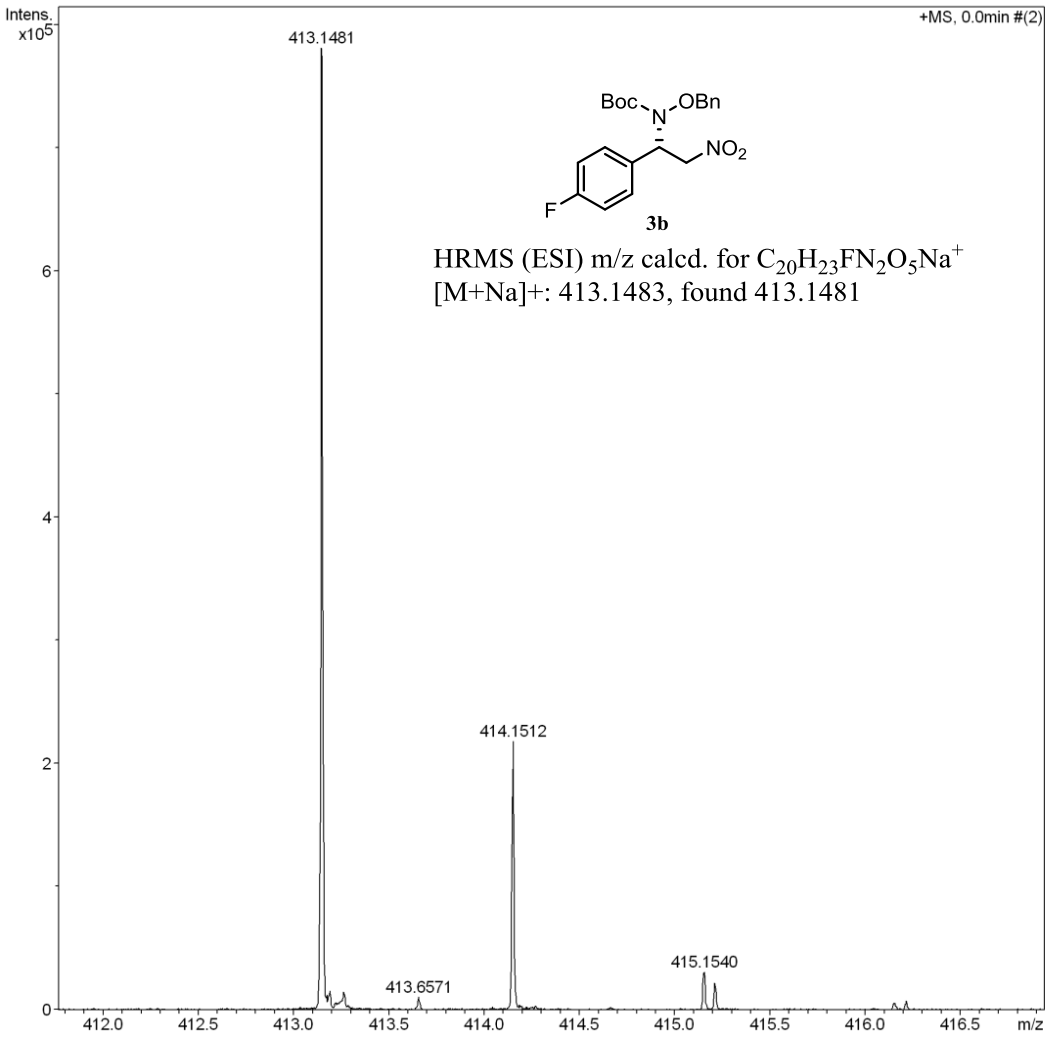
Display Report

Analysis Info

Analysis Name	D:\Data\FAN\data\2017\0707\ckh-16a.d	Acquisition Date	7/7/2017 10:24:17 AM
Method	pos_low-20151116.m	Operator	Fan
Sample Name	caikaihua	Instrument	maXis 10103
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1200 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Waste



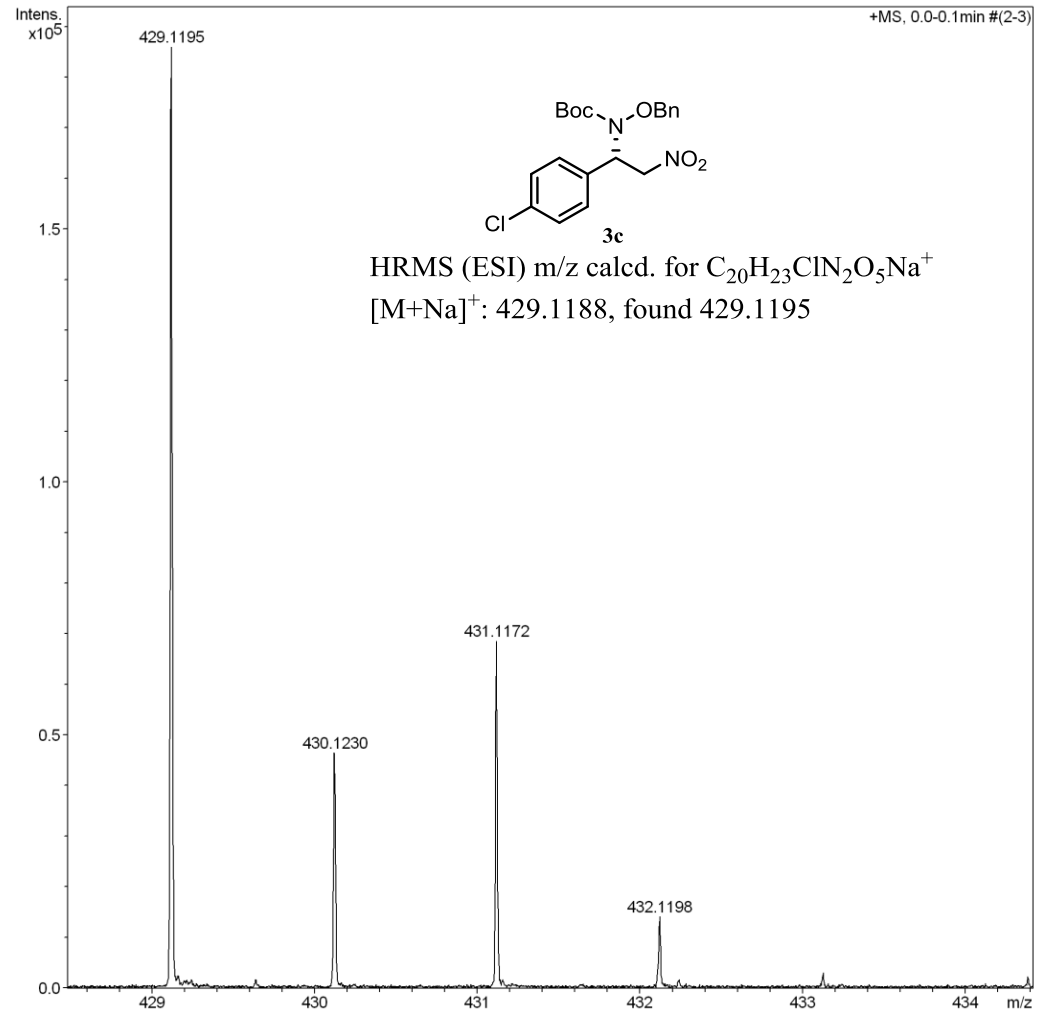
Display Report

Analysis Info

Analysis Name	D:\Data\FAN\data\2017\0707\ckh-11.d	Acquisition Date	7/7/2017 10:03:51 AM
Method	pos_low-20151116.m	Operator	Fan
Sample Name	caikaihua	Instrument	maXis 10103
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1200 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Waste



3c

HRMS (ESI) m/z calcd. for $C_{20}H_{23}ClN_2O_5Na^+$ $[M+Na]^+$: 429.1188, found 429.1195

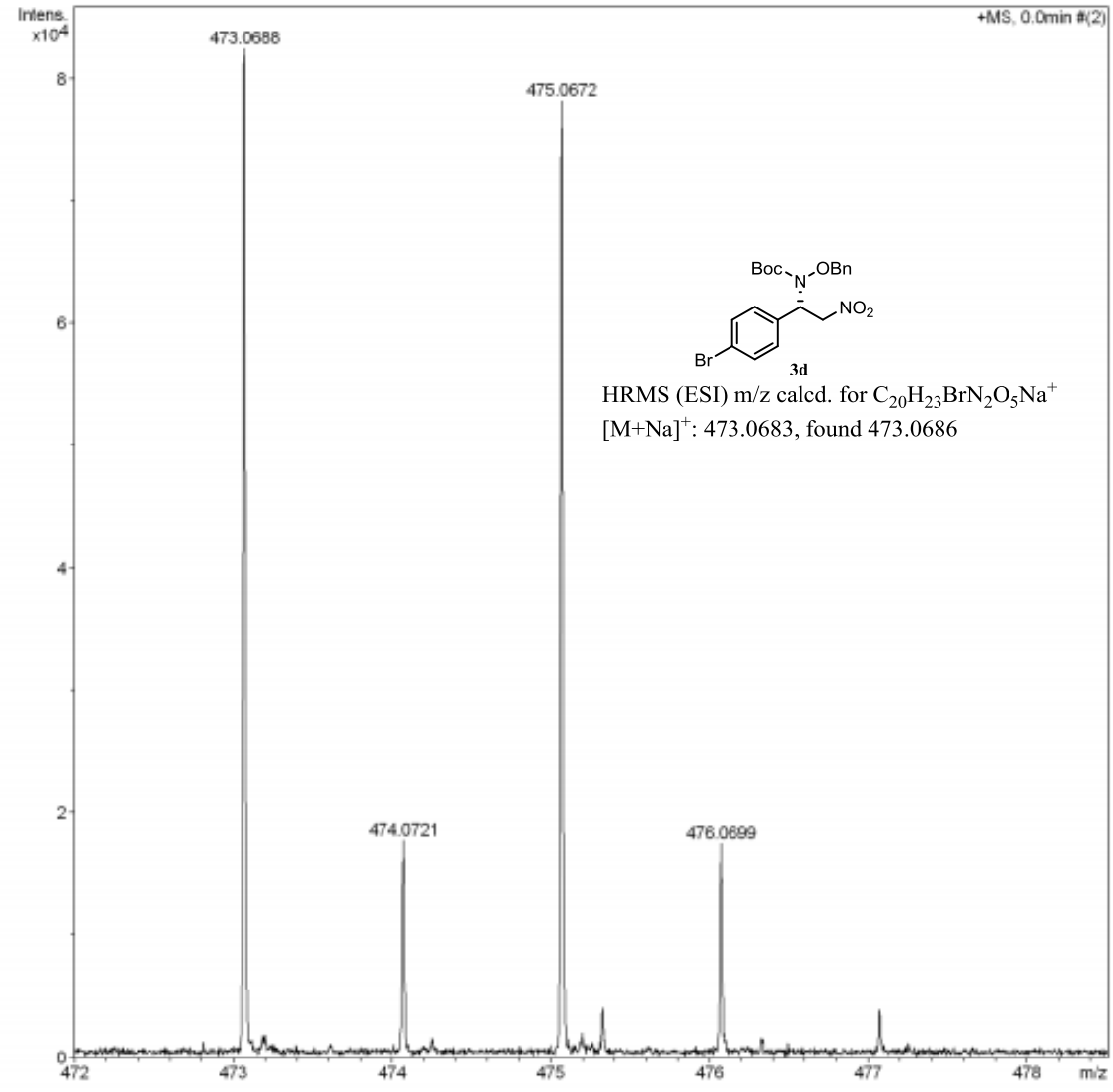
Display Report

Analysis Info

Analysis Name	D:\Data\2018\0112\lay-2.d	Acquisition Date	1/12/2018 9:43:58 AM
Method	pos_low-20151116.m	Operator	Fan
Sample Name	liualyun	Instrument	maXis 10103
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	180 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Waste



3d

HRMS (ESI) m/z calcd. for $C_{20}H_{23}BrN_2O_5Na^+$ $[M+Na]^+$: 473.0683, found 473.0686

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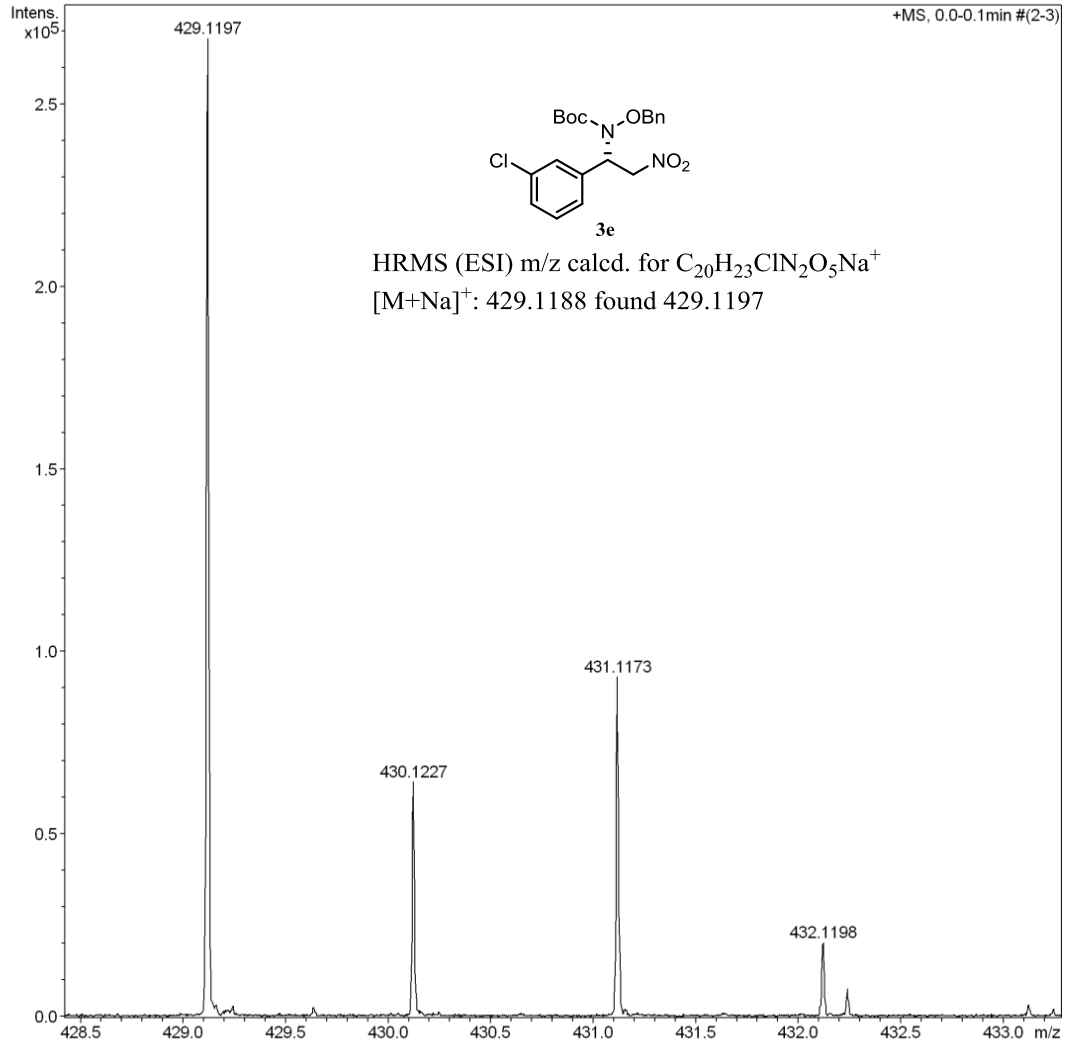
Display Report

Analysis Info

Analysis Name	D:\Data\FAN\data\2017\0707\ckh-12.d	Acquisition Date	7/7/2017 10:06:21 AM
Method	pos_low-20151116.m	Operator	Fan
Sample Name	caikaihua	Instrument	maXis 10103
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1200 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Waste



3e

HRMS (ESI) m/z calcd. for $C_{20}H_{23}ClN_2O_5Na^+$ $[M+Na]^+$: 429.1188 found 429.1197

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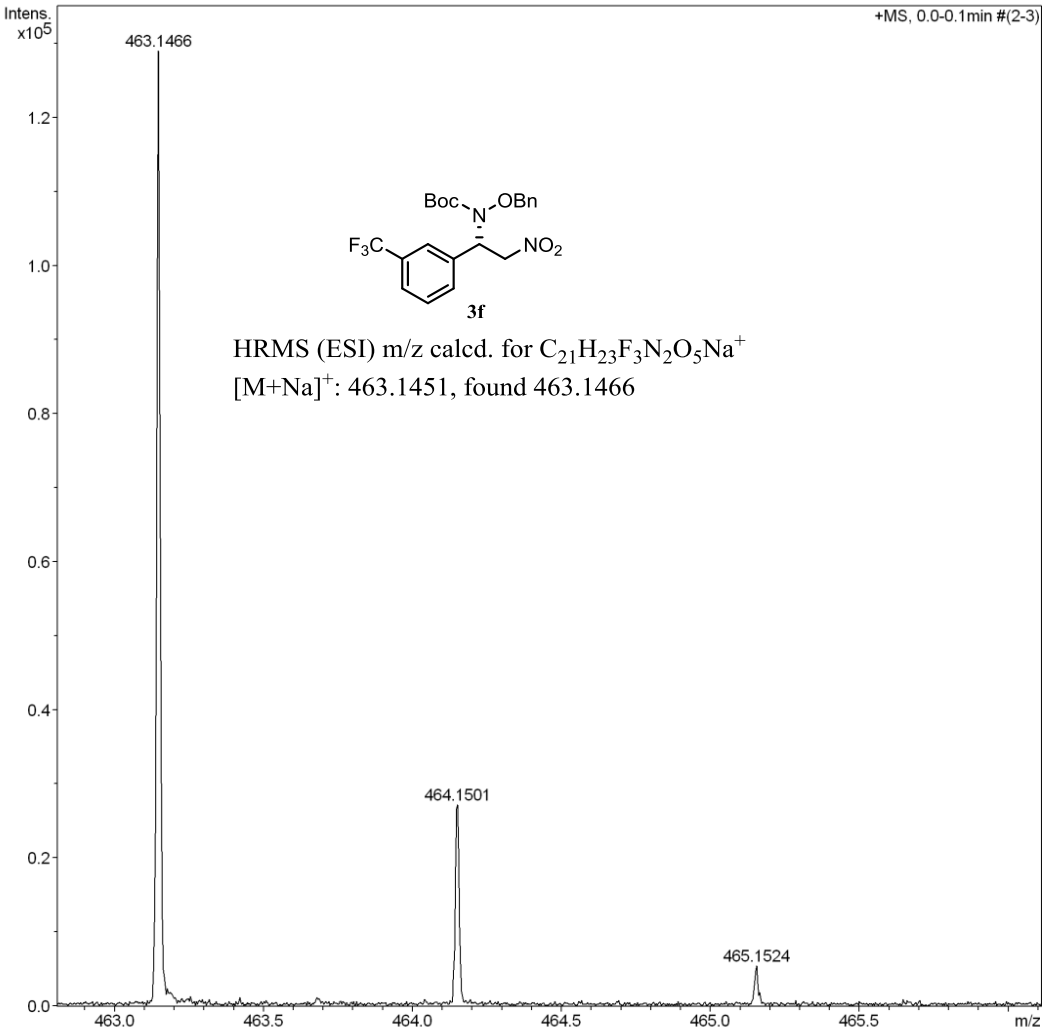
Display Report

Analysis Info

Analysis Name	D:\Data\FAN\data\2017\0707\ckh-14.d	Acquisition Date	7/7/2017 10:11:04 AM
Method	pos_low-20151116.m	Operator	Fan
Sample Name	caikaihua	Instrument	maXis 10103
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1200 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Waste



3f

HRMS (ESI) m/z calcd. for $C_{21}H_{23}F_3N_2O_5Na^+$ $[M+Na]^+$: 463.1451, found 463.1466

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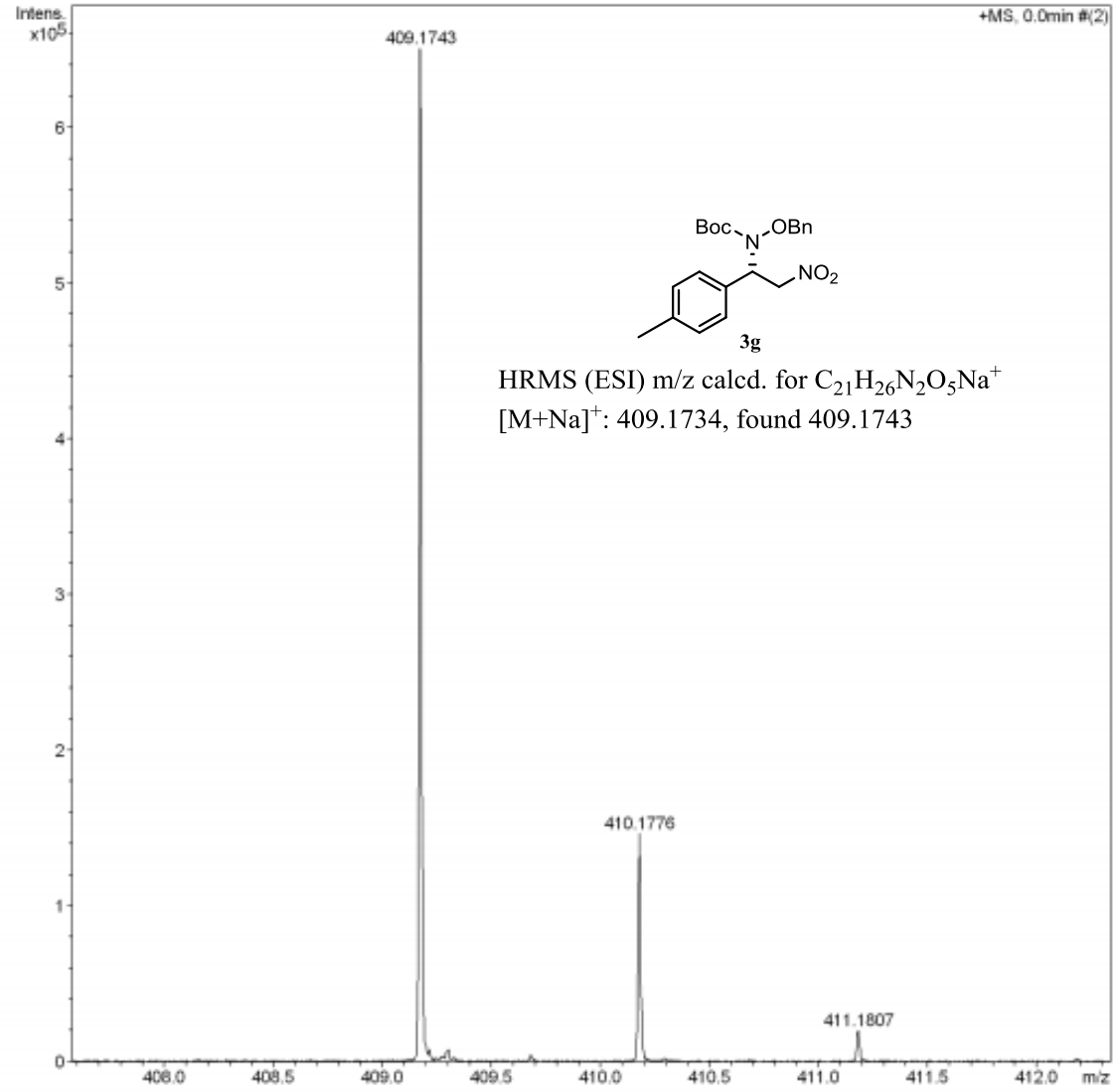
Display Report

Analysis Info

Analysis Name	D:\Data\2018\0112\lay-3.d	Acquisition Date	1/12/2018 9:47:48 AM
Method	pos_low-20151116.m	Operator	Fan
Sample Name	liuaiyun	Instrument	maXis 10103
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	180 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Waste



3g

HRMS (ESI) m/z calcd. for $C_{21}H_{26}N_2O_5Na^+$ $[M+Na]^+$: 409.1734, found 409.1743

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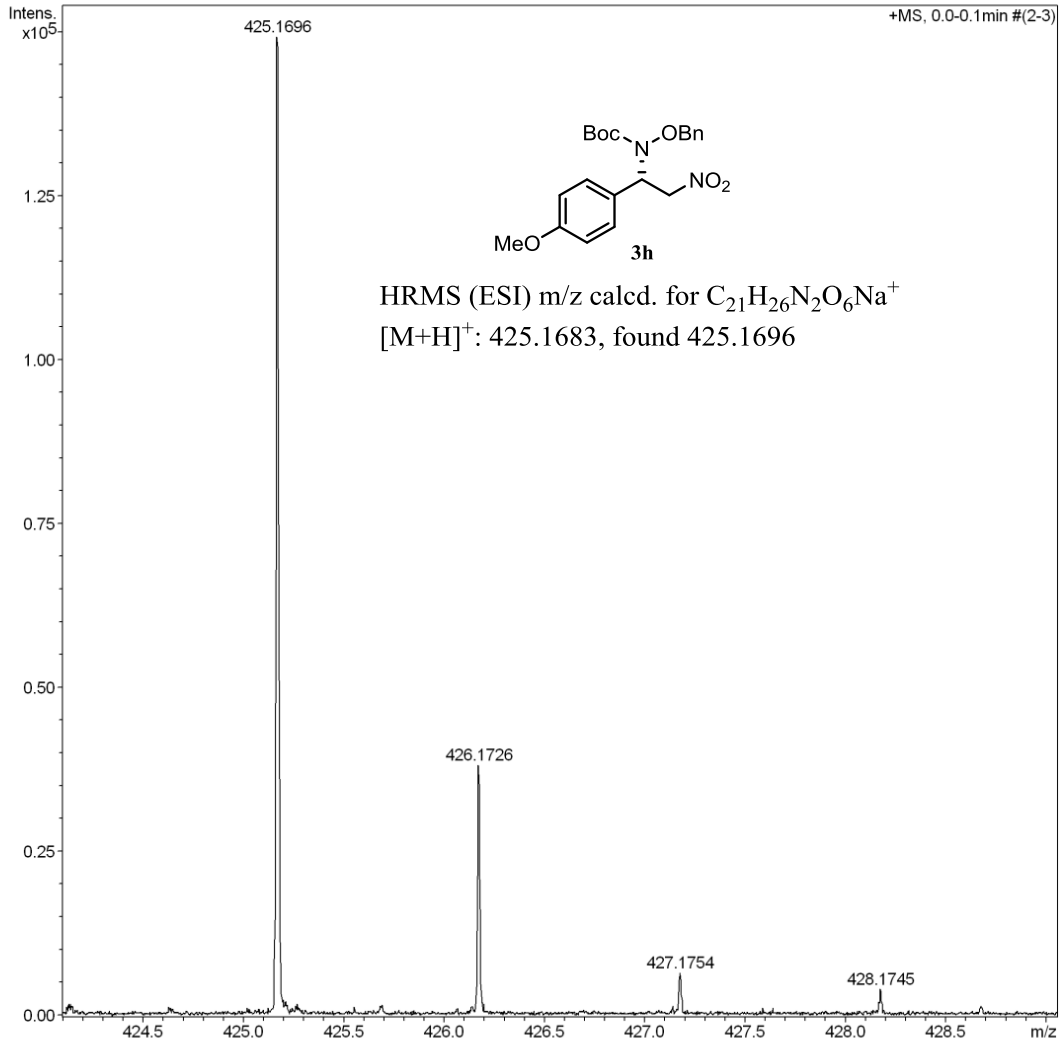
Display Report

Analysis Info

Analysis Name	D:\Data\FAN\data\2017\0707\ckh-13.d	Acquisition Date	7/7/2017 10:08:47 AM
Method	pos_low-20151116.m	Operator	Fan
Sample Name	caikaihua	Instrument	maXis 10103
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1200 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Waste



3h

HRMS (ESI) m/z calcd. for $C_{21}H_{26}N_2O_6Na^+$ $[M+H]^+$: 425.1683, found 425.1696

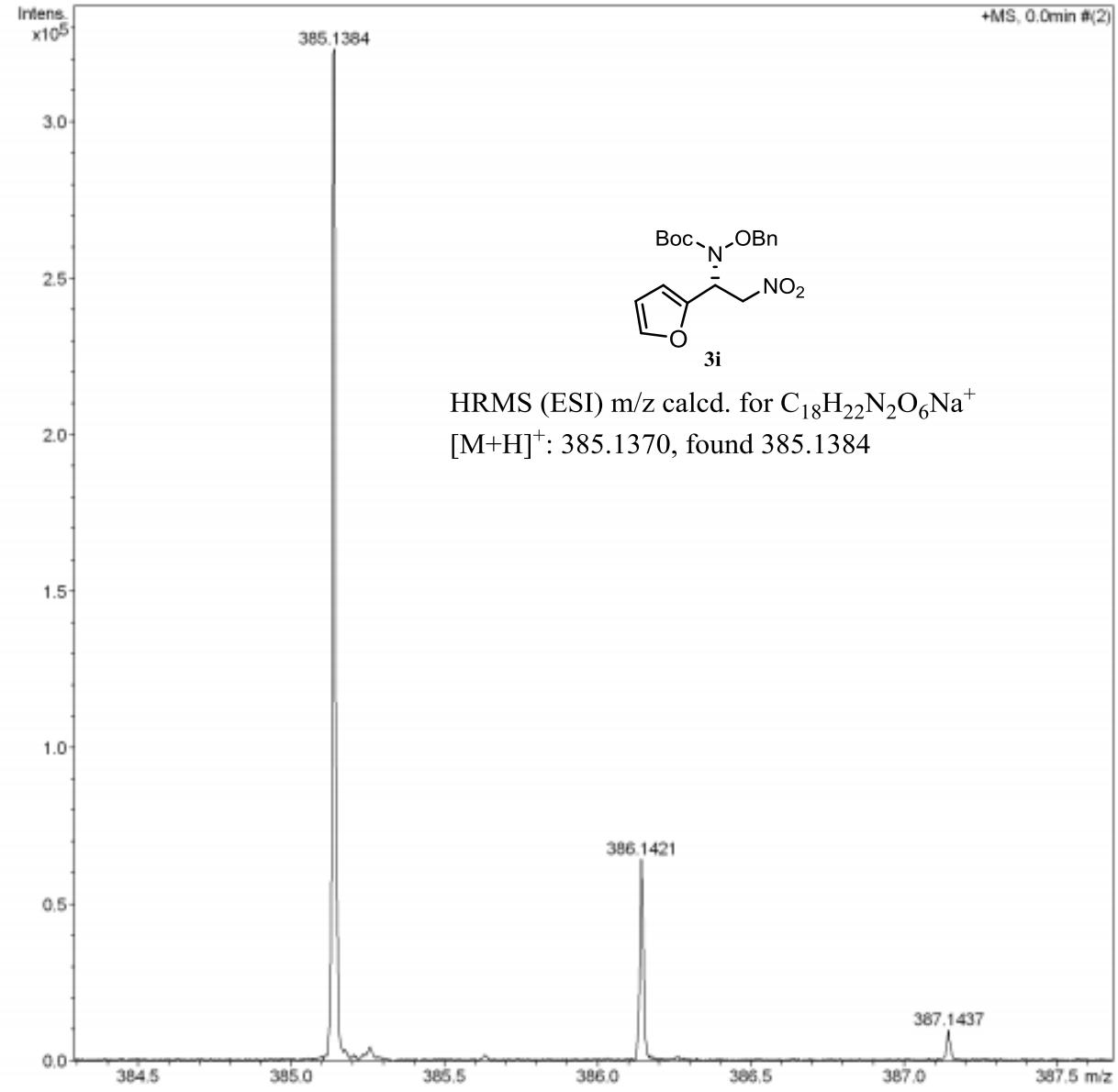
Display Report

Analysis Info

Analysis Name	D:\Data\2018\0112\lay-4.d	Acquisition Date	1/12/2018 9:53:09 AM
Method	pos_low-20151116.m	Operator	Fan
Sample Name	liuaiyun	Instrument	maXis 10103
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	180 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Waste



3i

HRMS (ESI) m/z calcd. for $C_{18}H_{22}N_2O_6Na^+$ $[M+H]^+$: 385.1370, found 385.1384

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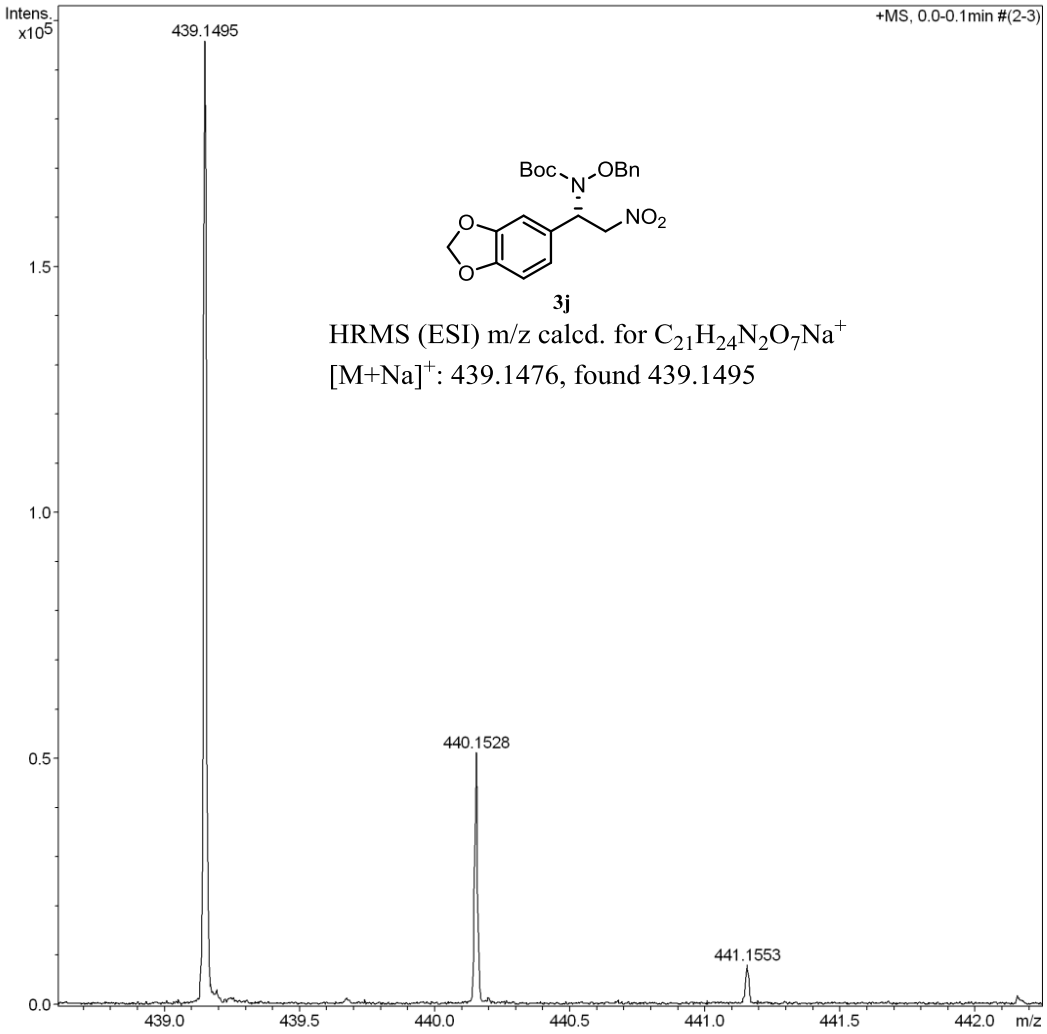
Display Report

Analysis Info

Analysis Name	D:\Data\FAN\data\2017\0707\ckh-15.d	Acquisition Date	7/7/2017 10:13:54 AM
Method	pos_low-20151116.m	Operator	Fan
Sample Name	caikaihua	Instrument	maXis 10103
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1200 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Waste



3j

HRMS (ESI) m/z calcd. for $C_{21}H_{24}N_2O_7Na^+$ $[M+Na]^+$: 439.1476, found 439.1495

Bruker Compass DataAnalysis 4.0 printed: 7/7/2017 10:14:35 AM Page 1 of 1

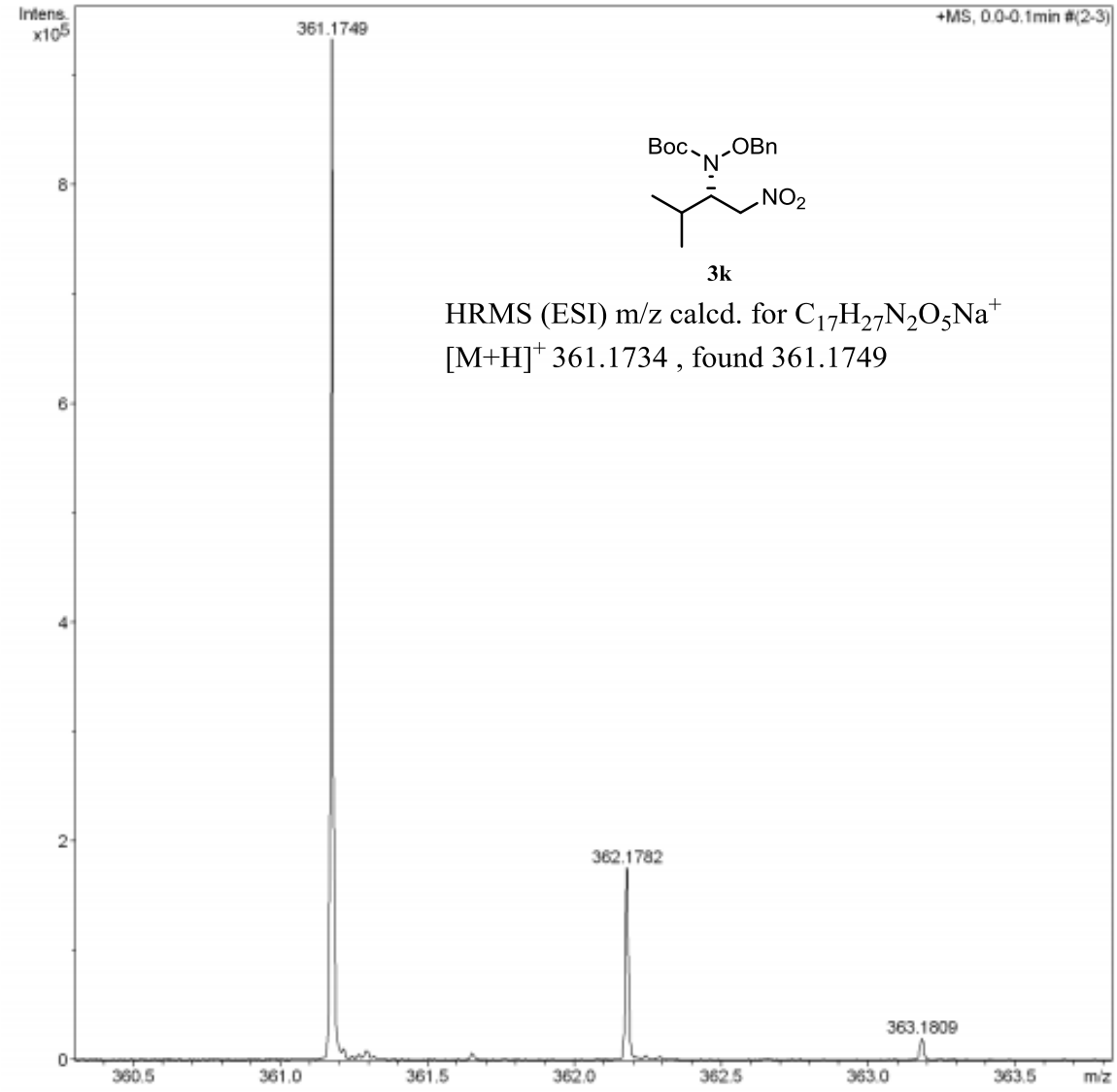
Display Report

Analysis Info

Analysis Name	D:\Data\2018\0112\lay-5.d	Acquisition Date	1/12/2018 9:58:29 AM
Method	pos_low-20151116.m	Operator	Fan
Sample Name	liualiyun	Instrument	maXis 10103
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	180 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Waste



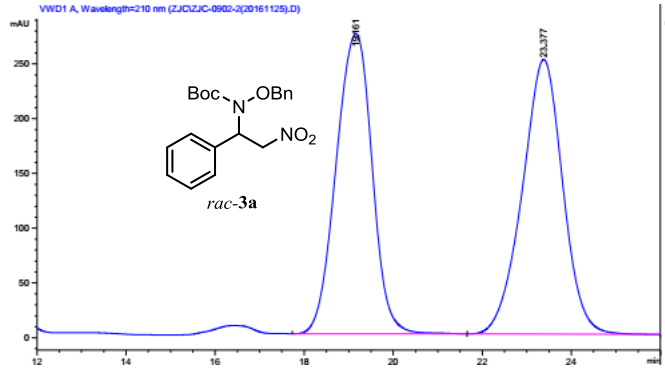
3k

HRMS (ESI) m/z calcd. for $C_{17}H_{27}N_2O_5Na^+$ $[M+H]^+$ 361.1734 , found 361.1749

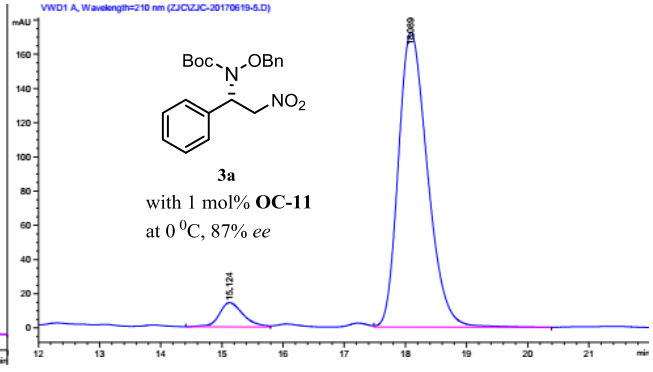
Bruker Compass DataAnalysis 4.0 printed: 1/12/2018 10:00:50 AM Page 1 of 1

HPLC copies of all amination products 3a~3k

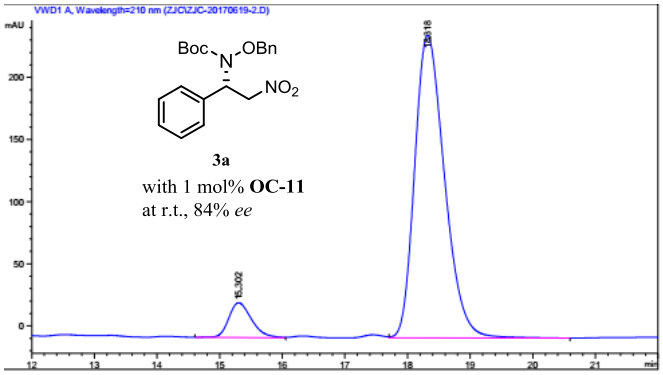
3a: HPLC: Chiralpak AS-H (^{i}PrOH/*n*-Hexane) = 3.2:96.8, flow rate = 0.5 mL/min, 210 nm.



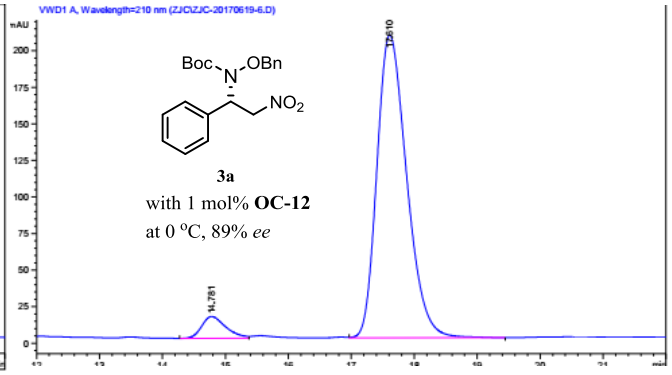
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	19.161	BB	0.8973	1.53921e4	274.02924	49.2622
2	23.377	BB	0.9553	1.58531e4	250.83952	50.7378



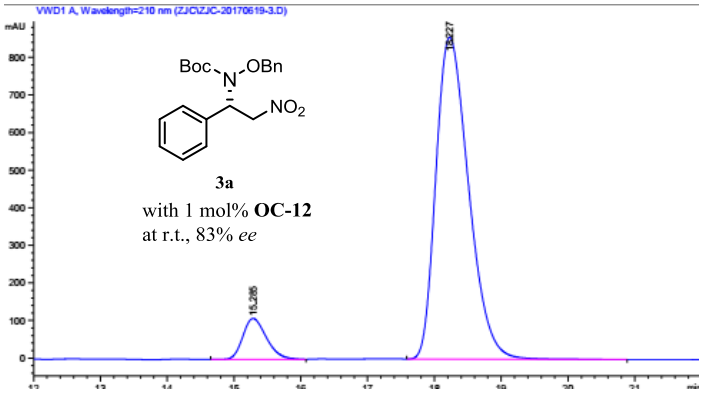
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	15.124	BV	0.4123	383.48282	14.09812	6.3131
2	18.089	VB	0.5130	5690.87549	172.43600	93.6869



峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	15.302	BV	0.3888	704.45618	27.97300	8.0264
2	18.318	VB	0.5155	8072.25146	243.66042	91.9736

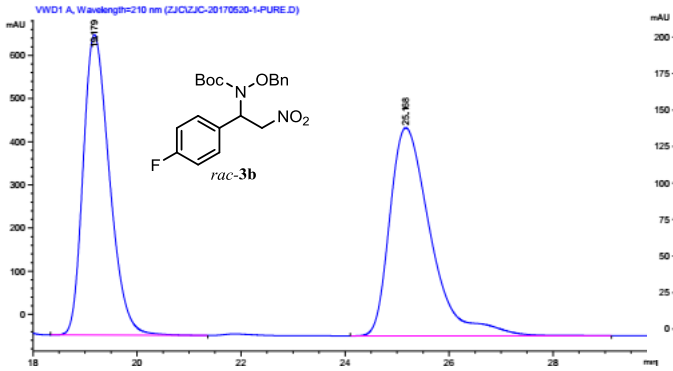


峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	14.781	BV	0.3987	395.55267	14.89901	5.5006
2	17.610	VB	0.5108	6795.50000	207.10387	94.4994



峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	15.285	BV	0.3796	2671.31323	109.10512	8.3946
2	18.227	VB	0.5320	2.91507e4	856.71912	91.6054

3b: HPLC: Chiralpak AS-H (^{i}PrOH/*n*-Hexane) = 3.2:96.8, flow rate = 0.5 mL/min, 210 nm.



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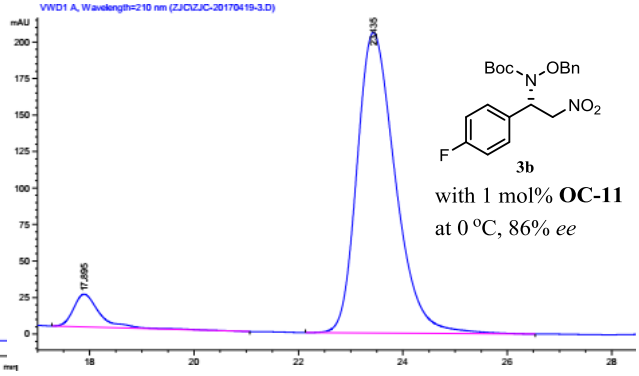
1	19.179	BB	0.5447	2.44357e4	695.83325	48.2281
2	25.168	BB	0.8358	2.62312e4	482.10376	51.7719



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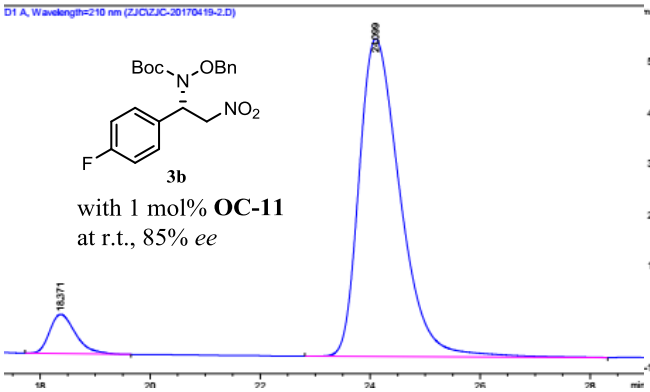
1	17.895	BB	0.5257	790.39105	22.39944	7.0169
2	23.435	BB	0.7856	1.04738e4	205.84108	92.9831



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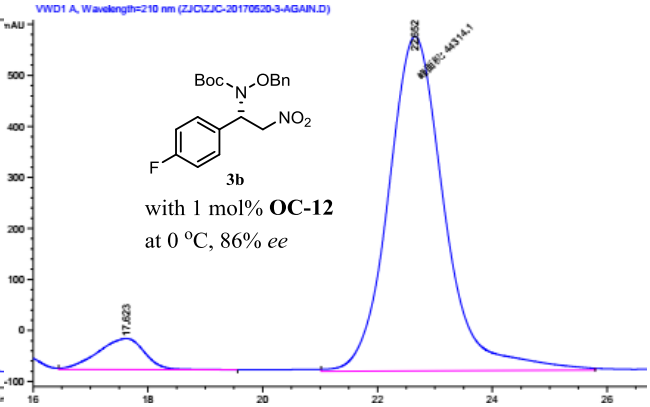
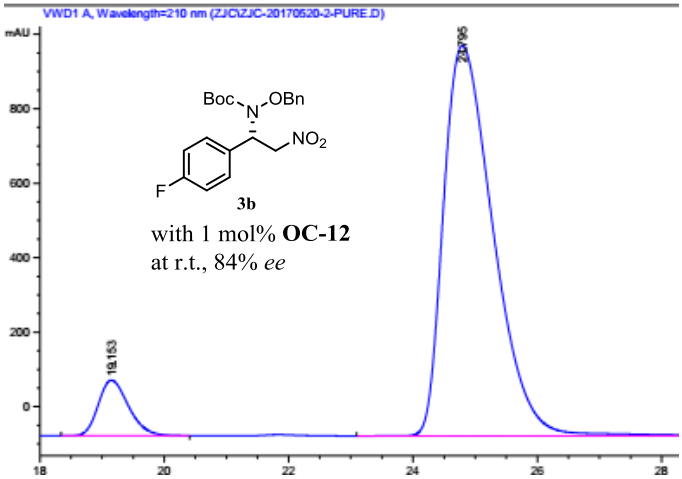
1	18.371	BB	0.5040	1634.31909	50.03896	7.2398
2	24.099	BB	0.7963	2.09397e4	405.60080	92.7602

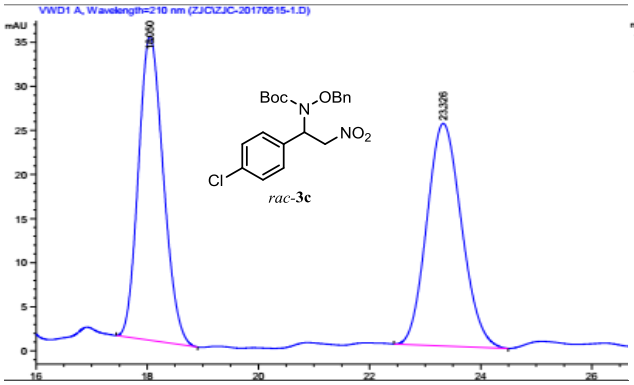


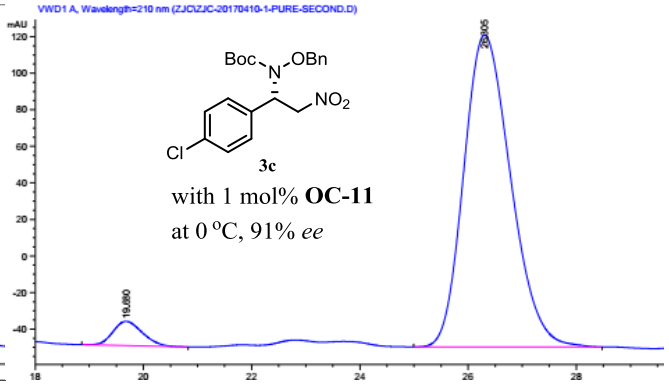
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	17.623	VB	0.8859	3469.14136	60.96505	7.2602
2	22.652	MM	1.1263	4.43141e4	655.77277	92.7398



峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	19.153	BB	0.5218	5043.16895	149.42206	7.9812
2	24.795	BB	0.8675	5.81448e4	1047.62109	92.0188

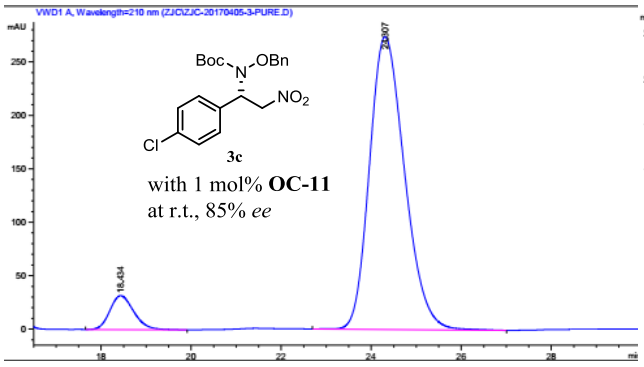
3c: HPLC: Chiralpak AS-H (^{i}PrOH/*n*-Hexane) = 3.2:96.8, flow rate = 0.5 mL/min, 210 nm.

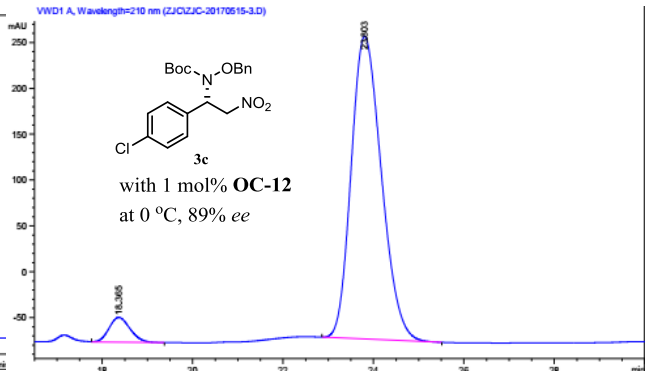




峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	18.050	BB	0.4852	1073.40002	34.48182	49.5602
2	23.326	BB	0.6713	1092.45300	25.22215	50.4398

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	19.680	BB	0.5806	500.28012	13.27684	4.6443
2	26.305	BB	0.9381	1.02716e4	170.67894	95.3557





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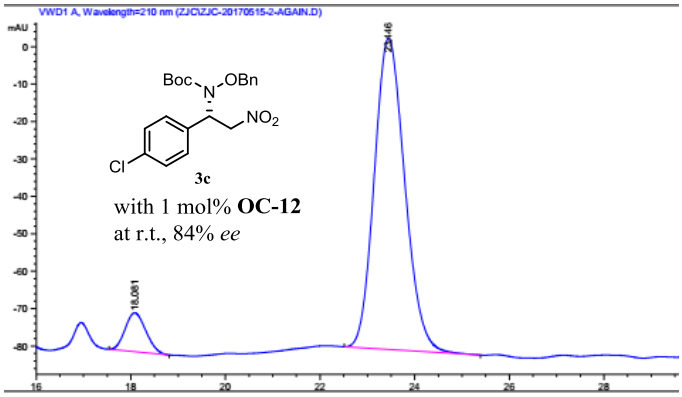
1	18.434	BB	0.5745	1173.34631	31.72623	7.3231
2	24.307	BB	0.8422	1.48491e4	274.02274	92.6769

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	18.365	VB	0.5016	880.43073	27.13395	5.4289
2	23.803	BB	0.7291	1.53369e4	330.08698	94.5711



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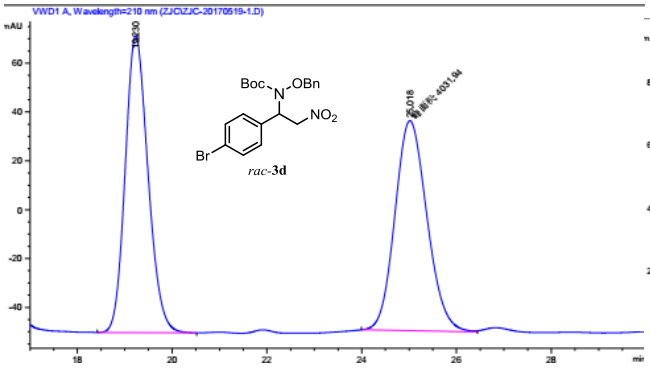
1	18.081	BB	0.4765	315.00906	10.33922	7.8212
2	23.446	BB	0.6962	3712.61938	83.10224	92.1788

3d: HPLC: Chiralpak AS-H (^{i}PrOH/*n*-Hexane) = 3.2:96.8, flow rate = 0.5 mL/min, 210 nm.



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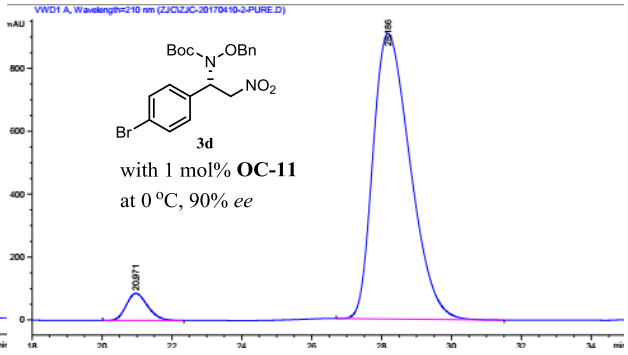
1	19.230	BB	0.5182	4063.01514	121.47465	50.1920
2	25.018	MM	0.7817	4031.93701	85.96800	49.8080



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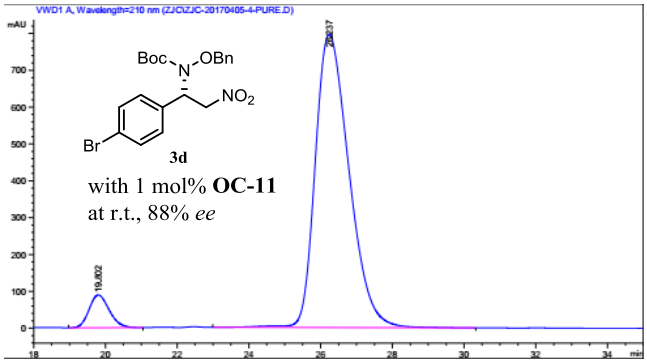
1	20.971	BB	0.6573	3642.74731	86.14391	5.1675
2	28.186	BB	1.1275	6.68507e4	907.40930	94.8325



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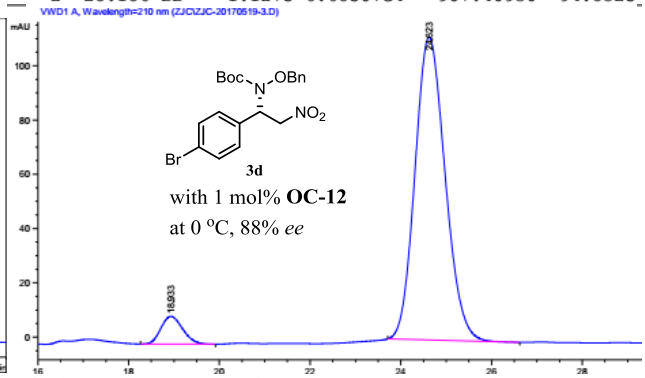
1	19.802	BB	0.6027	3455.75952	88.87244	6.2202
2	26.237	BB	1.0188	5.21014e4	795.85242	93.7798



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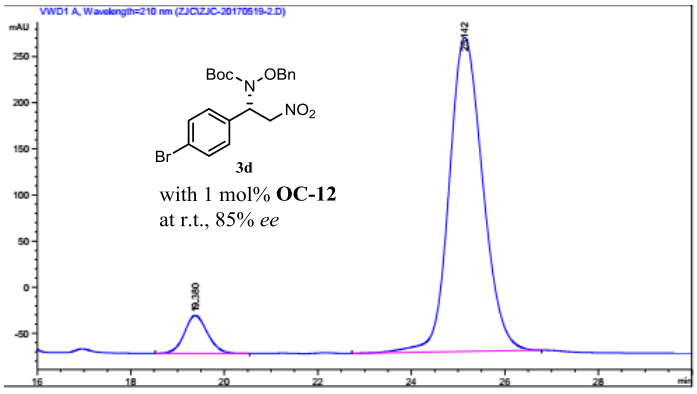
1	18.933	BB	0.5199	339.50070	10.20968	6.1821
2	24.623	BB	0.6834	5152.15869	111.63903	93.8179



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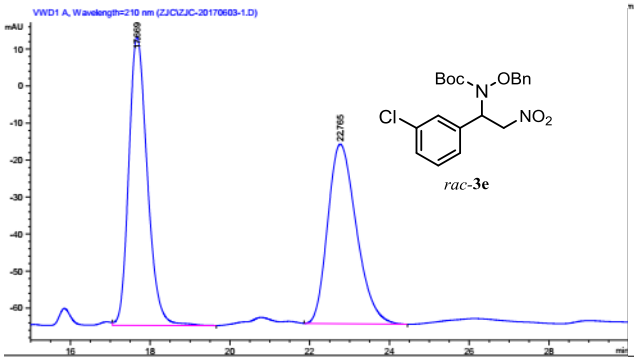
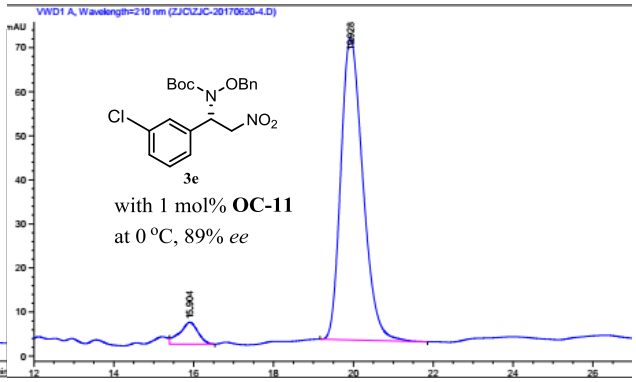
1	19.380	BB	0.5257	1408.71777	41.11862	7.6582
2	25.142	BB	0.7713	1.69862e4	340.35352	92.3418

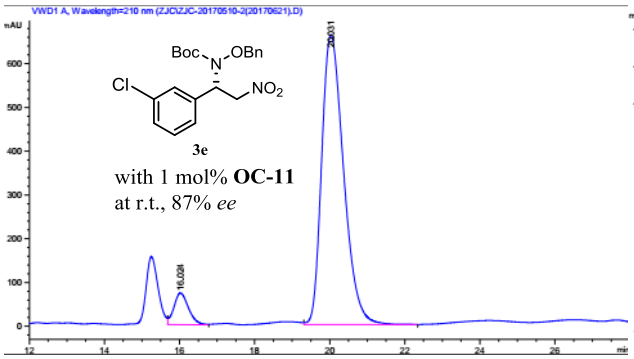
3e: HPLC: Chiralpak AS-H (^{i}PrOH/*n*-Hexane) = 3.2:96.8, flow rate = 0.5 mL/min, 210 nm.



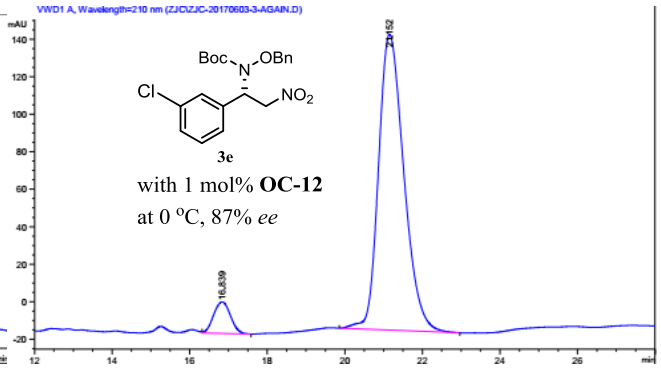
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	16.241	VV	0.4247	2.10995e4	770.07281	50.3327
2	20.425	BB	0.6416	2.08206e4	506.46207	49.6673



峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	15.904	VB	0.4639	156.47989	4.92197	5.6303
2	19.928	BB	0.5926	2622.77979	68.66443	94.3697



峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	16.024	VV	0.4111	1942.08838	72.35827	6.7515
2	20.031	VB	0.6308	2.68231e4	661.82819	93.2485



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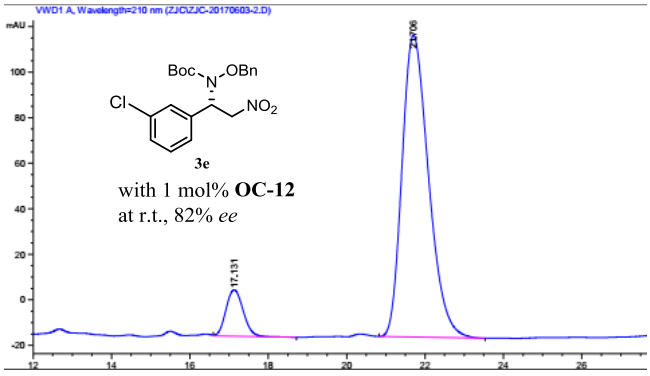
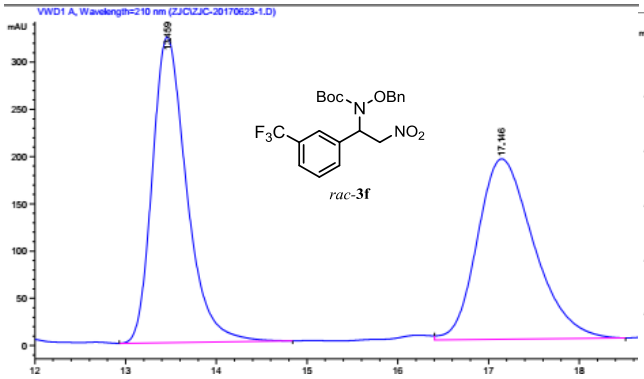
1	16.839	VB	0.4825	505.43359	16.77800	6.5900
2	21.152	BB	0.7033	7164.24316	157.91637	93.4100

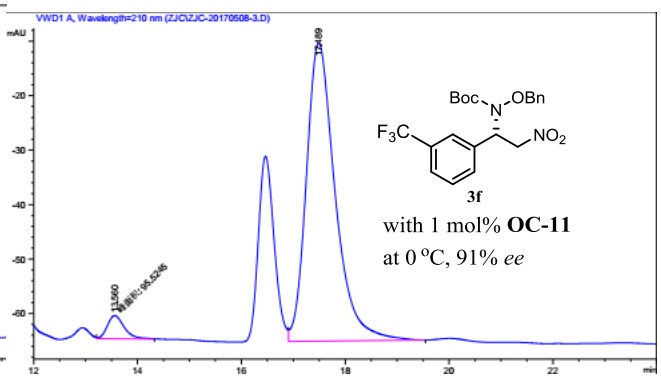


峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	17.131	VB	0.4851	625.55426	20.38276	9.1247
2	21.706	BB	0.7303	6230.07178	132.55443	90.8753

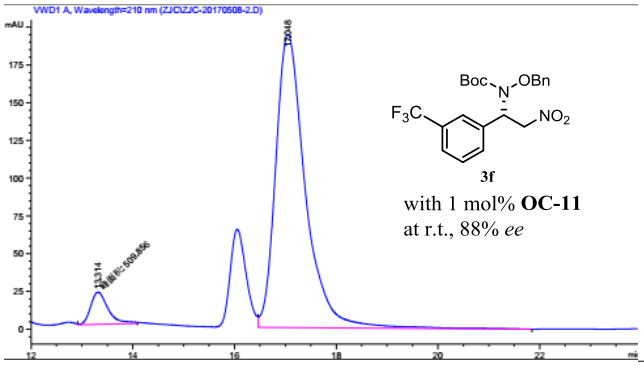
3f: HPLC: Chiralpak AS-H (^{i}PrOH/*n*-Hexane) = 3.2:96.8, flow rate = 0.5 mL/min, 210 nm.



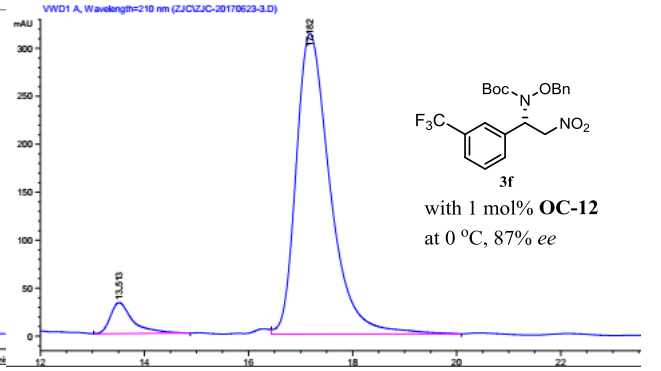
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	13.459	BB	0.4011	8491.17090	323.58392	50.7676
2	17.146	VB	0.6633	8234.40527	190.86440	49.2324



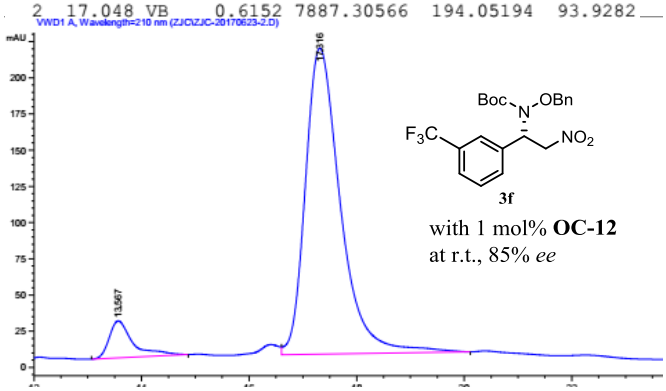
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	13.560	MM	0.3756	95.52453	4.23871	4.3245
2	17.489	VB	0.5833	2113.39478	54.74968	95.6755



峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	13.314	MM	0.3977	509.85583	21.36546	6.0718
2	17.048	VB	0.6152	7887.30566	194.05194	93.9282

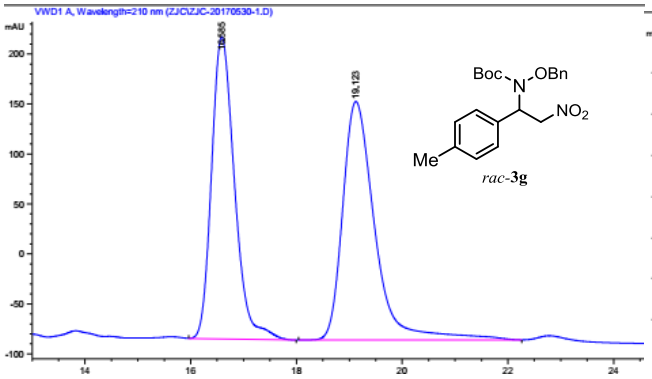


峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	13.513	BB	0.4416	961.11700	32.26261	6.3588
2	17.182	VB	0.6886	1.41535e4	312.97992	93.6412

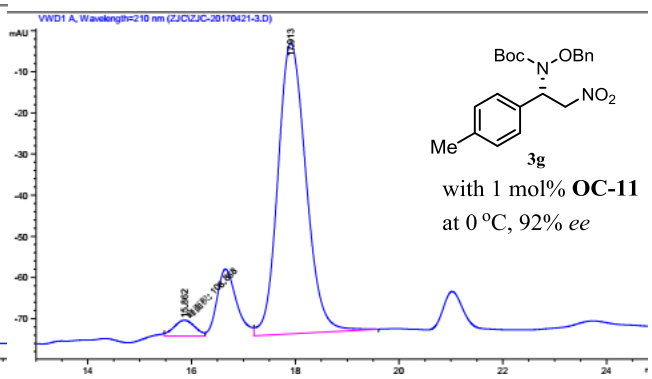


峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	13.567	BB	0.4521	782.23608	25.55308	7.5379
2	17.316	VB	0.6899	9595.10645	211.25783	92.4621

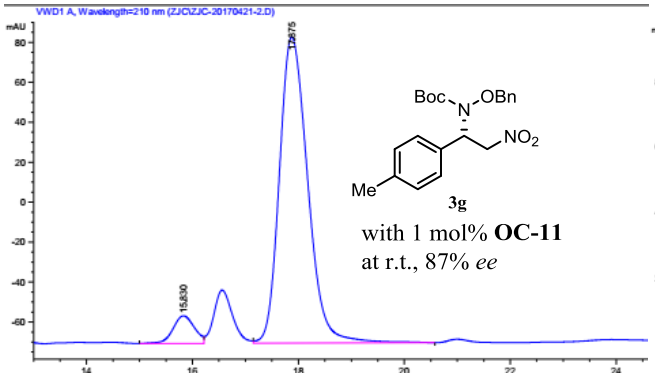
3g: HPLC: Chiralpak AS-H (^{i}PrOH/*n*-Hexane) = 3.2:96.8, flow rate = 0.5 mL/min, 210 nm.



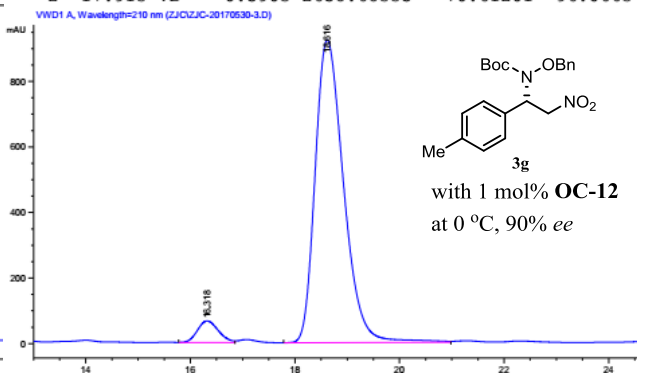
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	16.585	VB	0.4661	9122.59961	302.24399	47.4726
2	19.123	BB	0.6435	1.00939e4	238.64725	52.5274



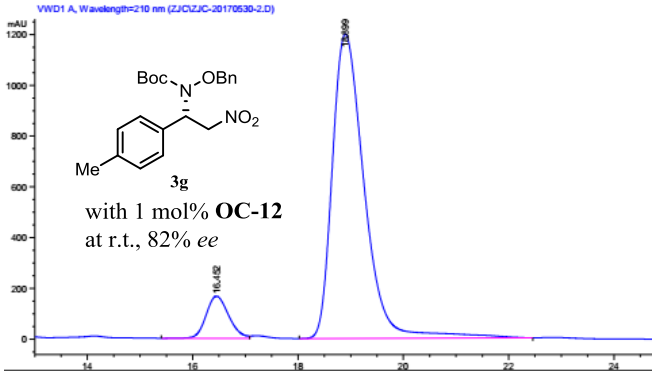
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	15.862	MM	0.4701	108.86800	3.85942	3.9337
2	17.913	VB	0.5903	2658.68335	70.61201	96.0663



峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	15.830	BV	0.4336	388.43280	13.83419	6.3812
2	17.875	VB	0.5849	5698.72070	153.23016	93.6188

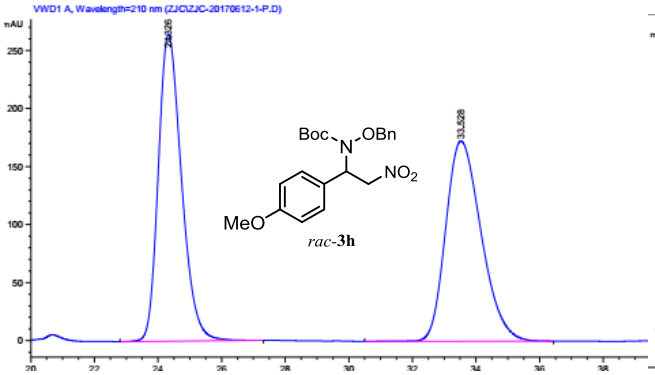


峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	16.318	VV	0.4349	1849.52295	66.20596	5.0366
2	18.616	BV	0.5865	3.48719e4	923.60822	94.9634

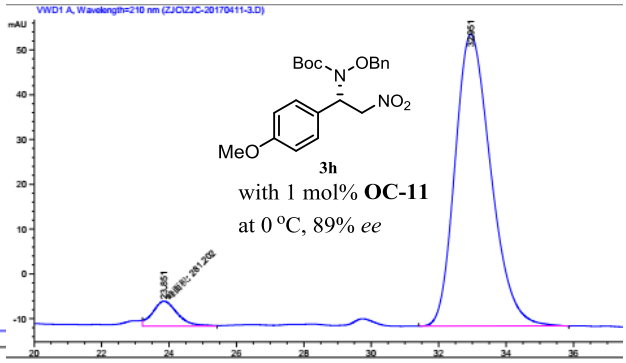


峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	16.452	BV	0.4623	4964.21289	166.26392	8.9308
2	18.899	BB	0.6496	5.06208e4	1199.00464	91.0692

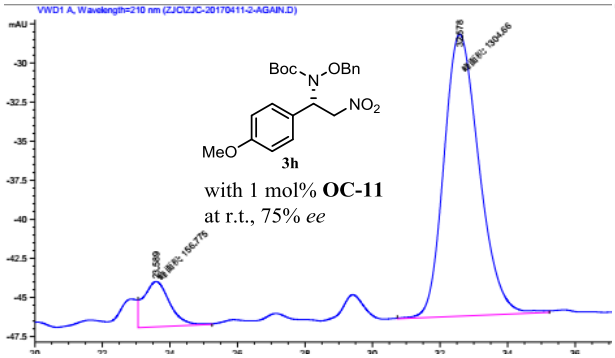
3h: HPLC: Chiralpak AS-H (^{i}PrOH/*n*-Hexane) = 3.2:96.8, flow rate = 0.5 mL/min, 210 nm.



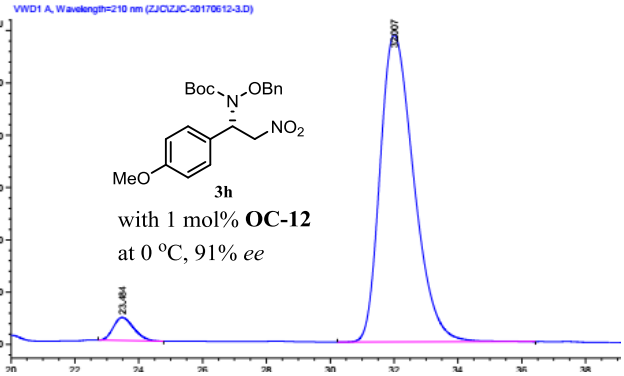
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	24.326	BB	0.7844	1.34022e4	264.40491	50.0759
2	33.528	BB	1.2093	1.33616e4	172.57155	49.9241



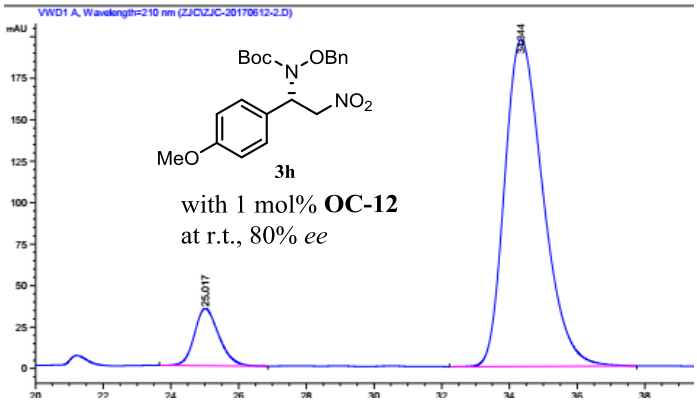
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	23.851	BB	0.8013	294.26517	5.36154	5.7733
2	32.951	BB	1.1330	4802.74902	65.15211	94.2267



峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	23.589	BB	0.9114	180.17036	2.64608	12.3594
2	32.578	BB	1.0952	1277.59412	17.95295	87.6406

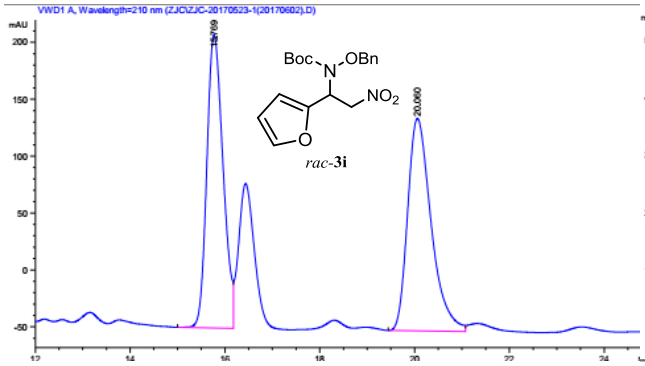


峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	23.484	BB	0.6805	977.93170	22.08734	4.3407
2	32.007	BB	1.1325	2.15514e4	293.90591	95.6593

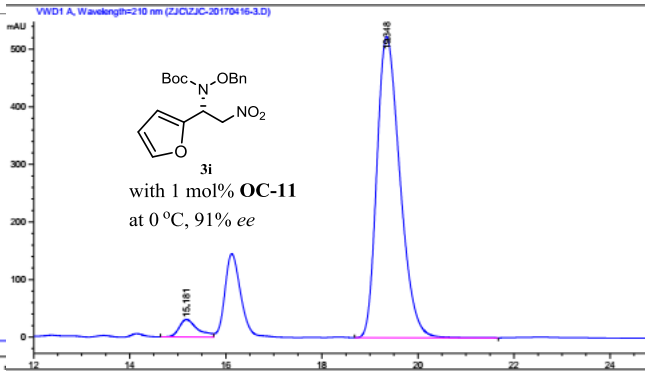


峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	25.017	BB	0.7665	1727.06628	34.47236	10.0310
2	34.344	BB	1.2215	1.54903e4	196.97749	89.9690

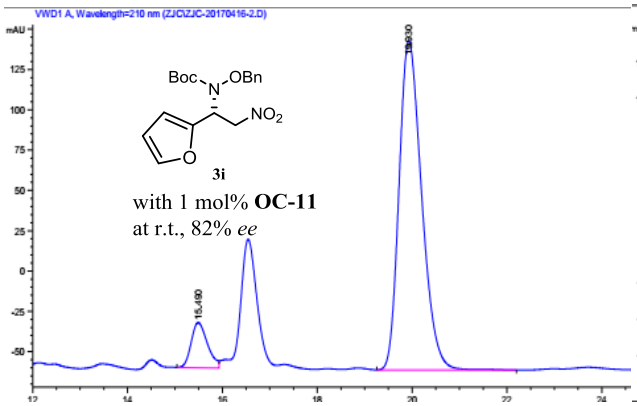
3i: HPLC: Chiralpak AS-H (^{i}PrOH/*n*-Hexane) = 3.2:96.8, flow rate = 0.5 mL/min, 210 nm.



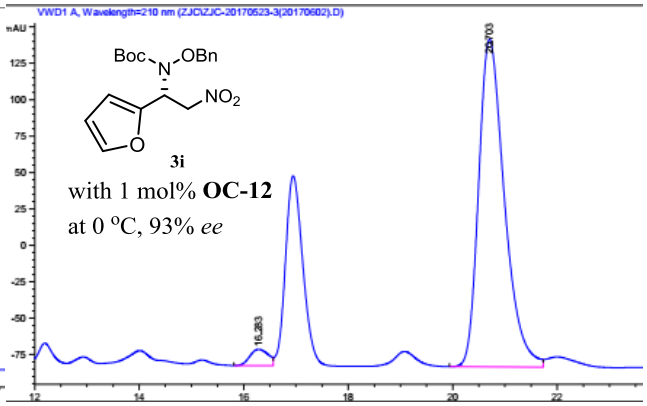
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	15.769	BV	0.3668	6117.26953	257.83551	48.7624
2	20.060	BV	0.5286	6427.77539	186.72919	51.2376



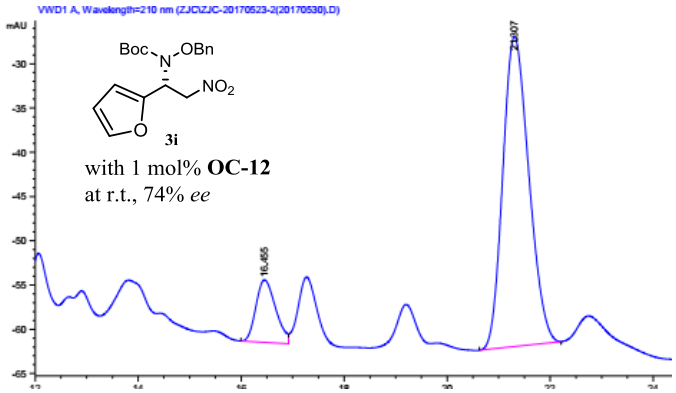
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	15.181	BV	0.3938	807.45898	30.40582	4.4266
2	19.348	BB	0.5156	1.74338e4	523.44342	95.5734



峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	15.490	BV	0.3646	663.65045	27.99099	8.9398
2	19.930	BB	0.5125	6759.88477	204.04015	91.0602

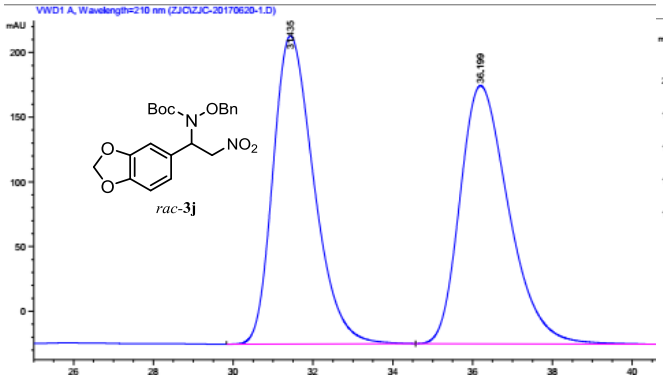


峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	16.283	BV	0.3942	275.86328	11.20533	3.4538
2	20.703	BV	0.5249	7711.48535	224.98076	96.5462

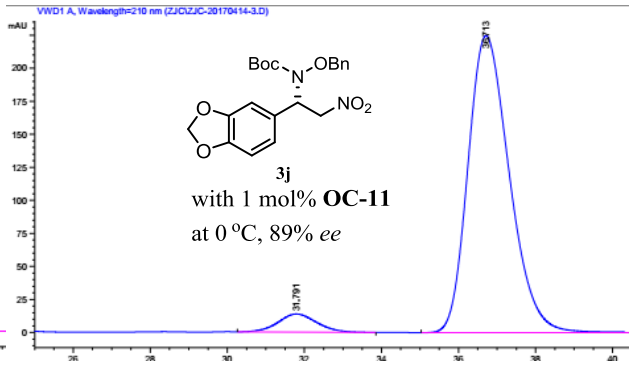


峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	16.455	BV	0.4139	187.55701	7.05988	13.2595
2	21.307	BB	0.5456	1226.95618	35.03143	86.7405

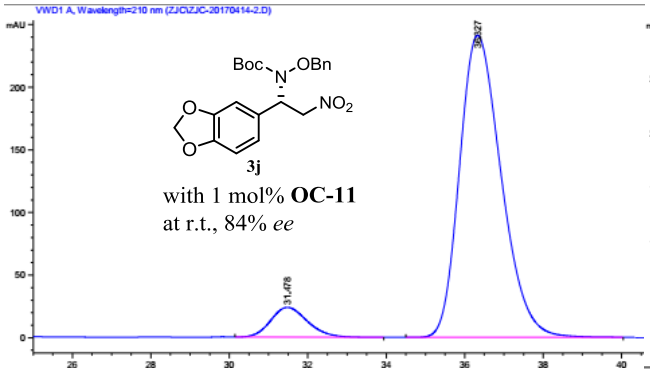
3j: HPLC: Chiralpak AS-H (^{i}PrOH/*n*-Hexane) = 3.2:96.8, flow rate = 0.5 mL/min, 210 nm.



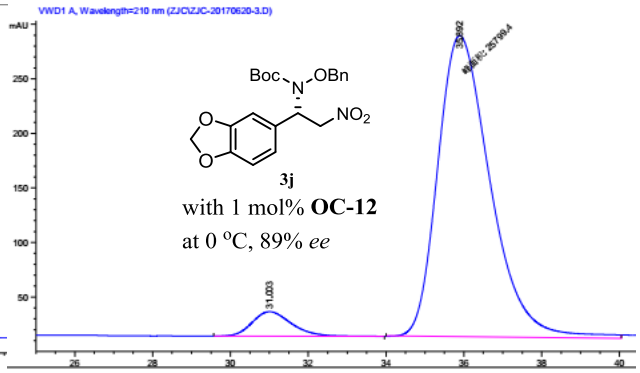
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	31.435	BB	1.1008	1.68917e4	238.06201	49.9809
2	36.199	BB	1.3022	1.69047e4	199.37091	50.0191



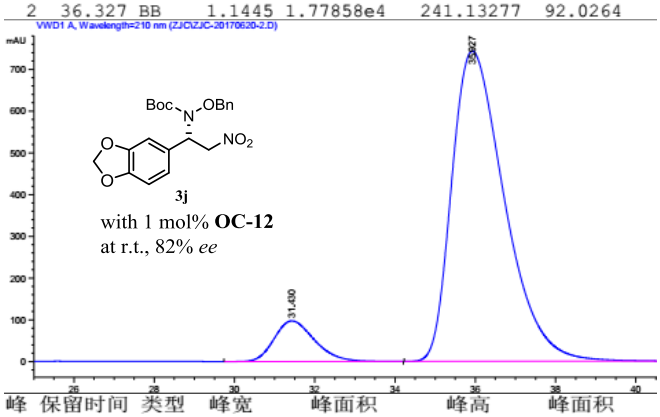
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	31.791	BB	1.0377	958.14807	13.68788	5.3969
2	36.713	BB	1.1555	1.67954e4	224.84811	94.6031



峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	31.478	BB	0.9902	1541.03894	23.71176	7.9736
2	36.327	BB	1.1445	1.77858e4	241.13277	92.0264

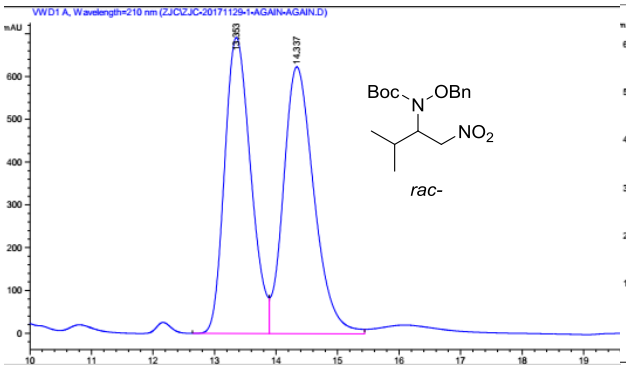


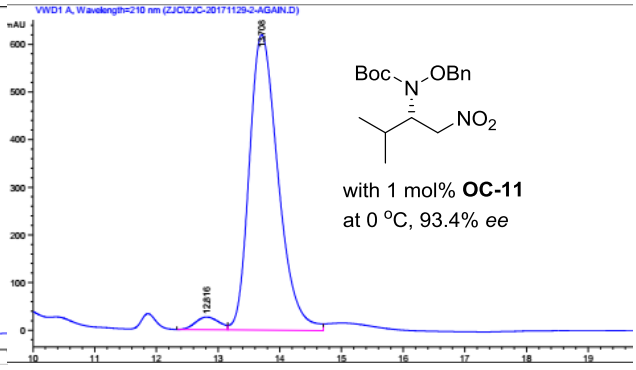
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	31.003	BB	1.0525	1567.39282	22.42312	5.7274
2	35.892	MM	1.5588	2.57994e4	275.84027	94.2726



峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	31.430	BB	1.0834	6809.32813	97.42136	9.1306
2	35.927	BB	1.4145	6.77677e4	743.42853	90.8694

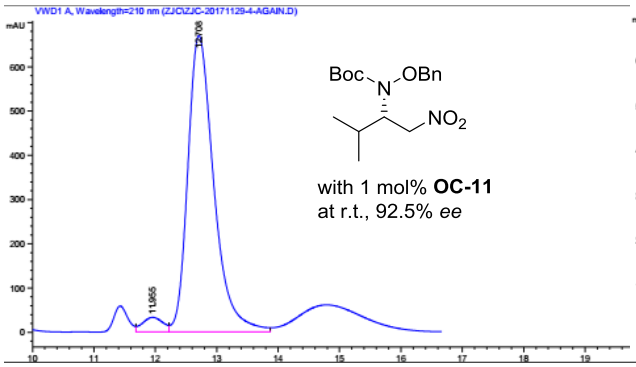
3k: HPLC: Chiralpak AS-H (^{i}PrOH/*n*-Hexane) = 1.5:98.5, flow rate = 0.5 mL/min, 210 nm.

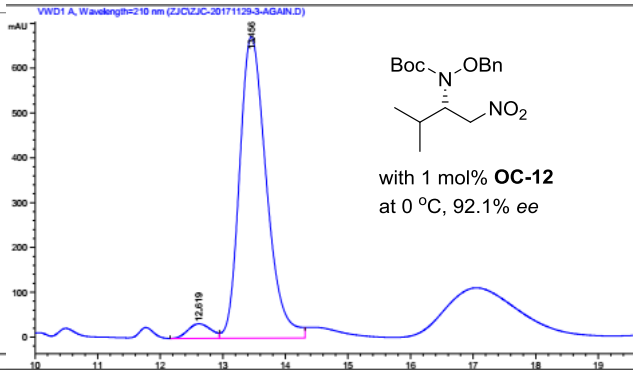




峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	13.353	BV	0.4610	2.04078e4	692.06104	48.6137
2	14.337	VV	0.5313	2.15718e4	624.07593	51.3863

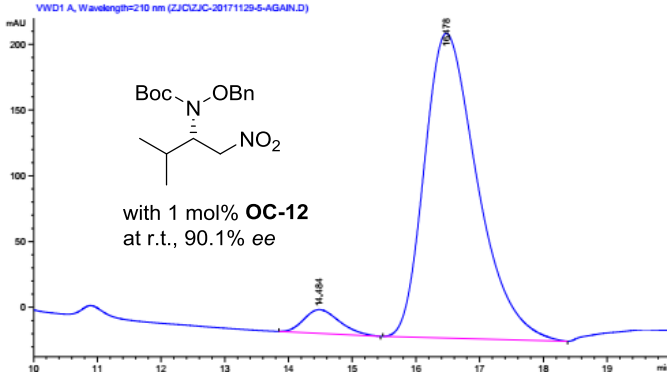
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	12.816	BV	0.4134	703.70599	26.70556	3.3765
2	13.708	VV	0.5029	2.01378e4	620.08466	96.6235





峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	11.955	VV	0.3572	766.23889	33.08878	3.7421
2	12.708	VV	0.4504	1.97100e4	669.71185	96.2579

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	12.619	BV	0.3973	821.14148	32.55119	3.9423
2	13.456	VV	0.4597	2.00081e4	673.22314	96.0577



峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	14.484	BB	0.5986	695.31122	18.00089	4.9596
2	16.478	BB	0.8833	1.33243e4	231.58272	95.0404