Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting information

Enantioselective Aza-Henry Reaction of Trifluoromethyl Ketimines Catalyzed by Phase-Transfer Catalysts

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1. General information

Unless otherwise stated, all reagents were purchased from commercial suppliers and used without purification. All solvents were obtained from commercial sources and were purified according to standard procedures. TLC was carried out on silica gel plates (HSGF 254), which were visualized with UV light and/or staining with phosphomolybdic acids solution. Purification of reaction products was carried out by column chromatography using silica gel (200-300 mesh). ^{1}H, ^{13}C NMR, and ^{19}F NMR spectra were recorded on a Varian Mercury-300BB (300 MHz), a Bruker NMR Spectrometer (400 MHz), or a Bruker NMR Spectrometer (500 MHz). All chemical shifts (δ) were given in ppm. Chemical shifts are relative to the resonance of the deuterated solvent as the internal standard (CDCl3, δ 7.26 ppm for proton NMR, δ77.16 ppm for carbon NMR; DMSO-d6, δ2.50 ppm for proton NMR, δ 39.52 ppm for carbon NMR). Date are presented as follows: chemical shift, integration, multiplicity (br = broad, s = singlet, d = double, t = triplet, q = quartet, m = multiplet), and coupling constant in hertz. Mass spectra were recorded on a Bruker Agilent 1290 MicrOTOF-Q Ⅱ instrument. Melting points were measured on a melting points apparatus and were uncorrected. The enantioselectivity value determination was carried out using chiral HPLC (Waters) instrumentation with a Chiracel AD-H column and IA-3 column. Optical rotations were measured on a Shanghai ShenGuang SGW-2 polarimeter at λ = 589 nm. Optical rotations are reported as follows: $[\alpha]_D^{25}$(*c* = g/100 mL, solvent).

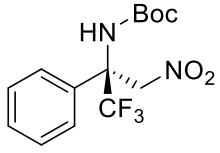
2. Starting Materials.

2,2,2-trifluroacetophenone were prepared according to literature procedures.[1] All phase-transfer catalysts (**1a-1g**) were synthesized according to procedures reported previously[2-4].

3. General Procedure for the Aza-Henry Reaction of Ketimines with Nitroalkanes and Characterization of Products 3a-3m.

Ketimines **2** (0.1 mmol) and catalyst **1d** (0.005 mmol, 5 mmol %) were dissolved in touluene（1 mL), and nitromethane (2.5mmol, 25 equiv) was added. Until the mixture was cooled to -20 ℃, the base $LiOH·H_2O$ was added freshly in one portion. The mixture was stirred sharply at -20 temperature until the reaction was judged to be completed by TLC. Then 5 mL sat. aq. NH_4Cl was added, and the mixture was stirred at room temperature. The aqueous layer was back-extracted (3×10 mL) with CH_2Cl_2. The combined organic layers were dried with Na_2SO_4, filtered and the solvent was removed in vacuo via evaporation. The crude product was purified by chromatography (PE/EA = 10:1).

tert-butyl (S)-(1,1,1-trifluoro-3-nitro-2-phenylpropan-2-yl) carbamate (**3a**)



3a

White solid, 38.8 mg, 95% yield, **m. p.** = 84-85 ℃, $\mathbf{[\alpha]_D^{25}}$ = -16.8 (*c* = 0.1, $CHCl_3$).

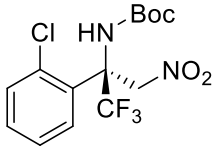
The ee value was 95% (Chiralpak AD-H, hexane/*i*-PrOH = 95:5, 214 nm, 1 mL/min, t_{major} = 9.76min, t_{minor} = 11.55 min).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.44 (s, 5H), 5.59 – 5.39 (m, 3H), 1.46 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 153.6, 132.6, 129.7, 129.0, 126.1, 123.9 (q, *J* = 286.0 Hz), 81.8, 73.1, 63.9 (q, *J* = 28.1 Hz), 28.1.

^{19}F NMR (376 MHz, $CDCl_3$) δ -74.97

HRMS (ESI) calculated for $C_{14}H_{17}F_3N_2O_4$ [M + Na]$^+$: 357.1038, found 357.1033.



3b

tert-butyl (S)-(2-(2-chlorophenyl)-1,1,1-trifluoro-3-nitropropan-2-yl)carbamate (**3b**)

White solid, 33.1 mg, 90% yield, **m. p.** = 72-74℃, **[α]$_D^{25}$** = -14 (*c*=0.1, $CHCl_3$).

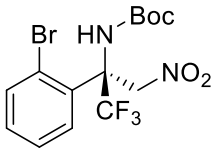
The ee value was 95% (Chiralpak IA-3, hexane/*i*-PrOH = 95:5, 214 nm, 1 mL/min, t_{major} = 12.56 min, t_{minor} = 11.39 min).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.57 – 7.51 (m, 1H), 7.49 – 7.42 (m, 1H), 7.40 – 7.31 (m, 2H), 6.01 (s, 1H), 5.58 – 5.21 (m, 2H), 1.39 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 152.8, 132.8, 132.5, 130.7, 129.2, 129.1, 127.5, 124.4 (q, *J* = 288.5 Hz), 81.4, 74.7, 64.3 (q, *J* = 29.1 Hz), 28.0.

^{19}F NMR (376 MHz, $CDCl_3$) δ -71.69.

HRMS (ESI) calculated for $C_{14}H_{16}ClF_3N_2O_4$ [M + Na]$^+$: 391.0632, found 391.0643.



3c

tert-butyl (S)-(2-(2-bromophenyl)-1,1,1-trifluoro-3-nitropropan-2-yl)carbamate (**3c)**

White solid, 37.1 mg, 91% yield, **m. p.** = 69-71℃,**[α]$_D^{25}$** = -14.8 (*c* =0.1, $CHCl_3$).

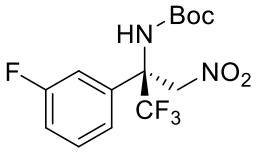
The ee value was 95% (Chiralpak AD-H, hexane/*i*-PrOH = 95:5, 214 nm, 1 mL/min, t_{major} = 19.29 min, t_{minor} = 14.57 min).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.70 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.53 (d, *J* = 8.2 Hz, 1H), 7.43 – 7.36 (m, 1H), 7.28 – 7.23 (m, 1H), 6.13 (s, 1H), 5.63 – 4.94 (m, 2H), 1.41 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 152.5, 136.6, 130.7, 129.3, 129.2, 128.1, 124.4 (q, *J* = 289.4 Hz), 121.1, 81.3, 74.9, , 63.7 (q, *J* = 24.6 Hz), 28.0.

^{19}F NMR (376 MHz, $CDCl_3$) δ -70.94

HRMS (ESI) calculated for $C_{14}H_{16}BrF_3N_2O_4$ [M + Na]$^+$: 435.0129, found 435.0138.



3d

tert-butyl (S)-(1,1,1-trifluoro-2-(3-fluorophenyl)-3-nitropropan-2-yl)carbamate (**3d**)

Pale yellow solid, 32.3 mg, 92% yield, **m. p.** = 56-57℃,**[α]$_D^{25}$** = -7.6 (*c* =0.1, $CHCl_3$).

The ee value was 93% (Chiralpak AD-H, hexane/*i*-PrOH = 95:5, 214 nm, 1 mL/min, t_{major} = 11.13 min, t_{minor} = 12.42 min).

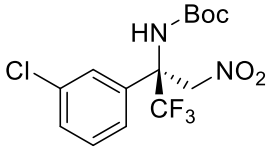
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.46 – 7.38 (m, 1H), 7.22 – 7.11 (m, 3H), 5.44 (dd, *J* = 19.3, 7.1 Hz, 3H), 1.46 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$) δ162.9 (d, *J* = 247.4 Hz), 153.5, 135.1, 130.6 (d, *J* = 8.3 Hz), 123.6 (q, *J* = 286.0 Hz), 121.8, 116.9 (d, *J* = 21.1 Hz), 114.1 (d, *J* = 25.3 Hz), 82.2, 73.0, 63.7 (q, *J* = 29.6 Hz),

28.1.

^{19}F NMR (376 MHz, $CDCl_3$) δ -74.95, -110.87.

HRMS (ESI) calculated for $C_{14}H_{16}F_4N_2O_4$ [M + Na]$^+$: 375.0912, found 375.0938.



3e

tert-butyl (S)-(2-(3-chlorophenyl)-1,1,1-trifluoro-3-nitropropan-2-yl)carbamate (**3e**)

White solid, 34.9 mg, 95% yield, **m. p.** = 65-67℃, **[α]$_D^{25}$** = -15.6 (*c* =0.1, $CHCl_3$).

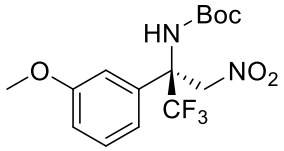
The ee value was 92% (Chiralpak AD-H, hexane/*i*-PrOH = 95:5, 214 nm, 1 mL/min, t_{major} = 12.82 min, t_{minor} = 11.43 min).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.44 – 7.36 (m, 3H), 7.31 (d, *J* = 7.6 Hz, 1H), 5.50 – 5.39 (m, 3H), 1.46 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 153.5, 135.2, 134.6, 130.2, 130.0, 125.7(q, *J* = 238.6 Hz, 123.2, 123.0, 82.2, 73.0,63.7 (q, *J* = 28.0 Hz), 28.1.

^{19}F NMR (376 MHz, $CDCl_3$) δ -74.92.

HRMS (ESI) calculated for $C_{14}H_{16}ClF_3N_2O_4$ [M + Na]$^+$: 391.0629, found 391.0643.



3f

tert-butyl (S)-(1,1,1-trifluoro-2-(3-methoxyphenyl)-3-nitropropan-2-yl)carbamate (**3f**)

White solid, 32.7 mg, 90% yield, **m. p.** = 50-52℃, **[α]$_D^{25}$** = -5.2 (*c* =0.1, $CHCl_3$).

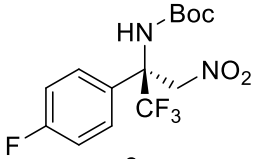
The ee value was 94% (Chiralpak AD-H, hexane/*i*-PrOH = 95:5, 214 nm, 1 mL/min, t_{major} = 12.12 min, t_{minor} = 13.75 min).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.35 (t, *J* = 8.4 Hz, 1H), 7.02 – 6.92 (m, 3H), 5.56 – 5.37 (m, 3H), 3.81 (s, 3H), 1.46 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 159.9, 153.6, 134.1, 130.0, 122.4,116.4 (q, *J* = 367.6 Hz) , 112.9, 81.8, 73.1, 63.8 (q, *J* = 28.2 Hz), 55.4, 28.1.

^{19}F NMR (376 MHz, $CDCl_3$) δ -74.85.

HRMS (ESI) calculated for $C_{15}H_{19}F_3N_2O_5$[M + Na]$^+$: 387.1139, found 387.1138.



3g

tert-butyl (S)-(1,1,1-trifluoro-2-(4-fluorophenyl)-3-nitropropan-2-yl)carbamate (**3g**)

White solid, 34.1 mg, 97% yield, **m. p.** = 63-65℃, **[α]$_D^{25}$** = -12 (*c* =0.1, $CHCl_3$).

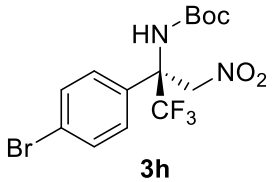
The ee value was 93% (Chiralpak AD-H, hexane/*i*-PrOH = 95:5, 214 nm, 1 mL/min, t_{major} = 9.09 min, t_{minor} = 11.73 min).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.42 (dd, *J* = 8.7, 4.9 Hz, 2H), 7.13 (t, *J* = 8.6 Hz, 2H), 5.47 (s, 2H), 5.40 (s, 1H), 1.46 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$) δ163.24 (d, *J* = 250.5 Hz), 153.6,128.4, 128.3(d, *J* = 7.9 Hz), 123.7 (q, *J* = 286.4 Hz), 116.1 (d, *J* = 22.0 Hz), 82.1, 73.1, 63.7 (q, *J* = 28.3 Hz)., 28.1.

^{19}F NMR (376 MHz, $CDCl_3$) δ -75.26, -111.37.

HRMS (ESI) calculated for $C_{14}H_{16}F_4N_2O_4$ [M + Na]$^+$: 375.0932, found 375.0938.



3h

tert-butyl (S)-(2-(4-bromophenyl)-1,1,1-trifluoro-3-nitropropan-2-yl)carbamate (**3h**)

White solid, 38.8 mg, 94% yield, **m. p.** = 70-72℃, **[α]$_D^{25}$** = -7.6 (*c* =0.1, $CHCl_3$).

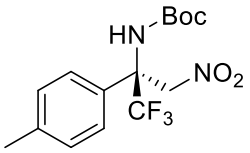
The ee value was 92% (Chiralpak AD-H, hexane/*i*-PrOH = 95:5, 214 nm, 1 mL/min, t_{major} = 9.41 min, t_{minor} = 11.83 min).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.58 (d, *J* = 8.8 Hz, 2H), 7.30 (d, *J* = 8.5 Hz, 2H), 5.42 (d, *J* = 23.5 Hz, 3H), 1.46 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 153.5, 132.2, 131.7, 129.9, 127.9, 124.3,123.6 (d, *J* = 285.7 Hz), 82.2, 72.9, 63.8 (q, *J* = 28.1 Hz)., 28.1.

^{19}F NMR (376 MHz, $CDCl_3$) δ -75.13.

HRMS (ESI) calculated for $C_{14}H_{16}BrF_3N_2O_4$ [M + Na]$^+$: 435.0123, found 435.0138.



3i

tert-butyl (S)-(1,1,1-trifluoro-3-nitro-2-(p-tolyl)propan-2-yl)carbamate (**3i**)

Pale yellow solid, 32.7 mg, 94% yield, **m. p.** = 75-77℃, **[α]$_D^{25}$** = -10.8 (*c* =0.1, $CHCl_3$)

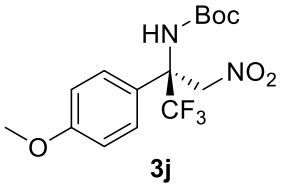
The ee value was 95% (Chiralpak AD-H, hexane/*i*-PrOH = 95:5, 214 nm, 1 mL/min, t_{major} = 7.71 min, t_{minor} = 8.72min).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.31 (d, *J* = 8.3 Hz, 2H), 7.24 (d, *J* = 8.3 Hz, 2H), 5.58 – 5.29 (m, 3H), 2.36 (s, 3H), 1.46 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 153.6, 139.8, 129.8, 129.5, 126.0, 125.6, 123.9 (q, *J* = 285.8 Hz) 81.7, 73.0, 63.8 (d, *J* = 28.3 Hz) 28.1, 21.0.

^{19}F NMR (376 MHz, $CDCl_3$) δ -75.18.

HRMS (ESI) calculated for $C_{15}H_{19}F_3N_2O_4$ [M + Na]$^+$: 371.1180, found 371.1189.



3j

tert-butyl (S)-(1,1,1-trifluoro-2-(4-methoxyphenyl)-3-nitropropan-2-yl)carbamate (**3j**)

Pale yellow solid, 32.7 mg, 98% yield, **m. p.** = 71-72℃, **[α]$_D^{25}$** = -17.2 (*c* =0.1, $CHCl_3$).

The ee value was 96% (Chiralpak AD-H, hexane/*i*-PrOH = 95:5, 214 nm, 1 mL/min, t_{major} = 11.27 min, t_{minor} = 13.04 min).

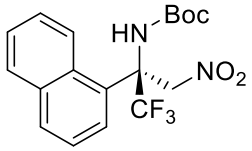
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.34 (d, *J* = 8.8 Hz, 2H), 6.94 (d, *J* = 9.0 Hz, 2H), 5.56 – 5.34 (m,

3H), 3.82 (s, 3H), 1.46 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 160.4, 153.4, 127.5, 124.2, 123.9(q, *J* = 285.8 Hz), 114.4, 81.7, 73.02, 63.6 (q, *J* = 28.3 Hz), 55.3, 28.2.

^{19}F NMR (376 MHz, $CDCl_3$) δ -75.35.

HRMS (ESI) calculated for $C_{15}H_{19}F_3N_2O_5$ $[M + Na]^+$: 387.1135, found 387.1138.



3k

tert-butyl (S)-(1,1,1-trifluoro-2-(naphthalen-1-yl)-3-nitropropan-2-yl)carbamate (**3k**)

White solid, 34.5 mg, 90% yield, **m. p.** = 56-57℃, **$[\alpha]_D^{25}$** = -6.4 (*c* =0.1, $CHCl_3$).

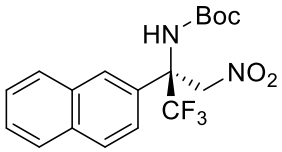
The ee value was 95% (Chiralpak AD-H, hexane/*i*-PrOH = 95:5, 214 nm, 1 mL/min, t_{major} =28.38 min, t_{minor} = 21.45 min).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.68 (d, *J* = 8.7 Hz, 1H), 7.91 (d, *J* = 8.2 Hz, 2H), 7.60 – 7.45 (m, 4H), 6.20 (s, 1H), 5.66 – 5.05 (m, 2H), 1.26 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 152.8, 135.7, 134.7, 131.3, 130.6, 130.1, 127.1, 126.2,125.3 (q, *J* = 113.9 Hz), 123.7, 123.4, 81.3, 73.5,63.5 (q, *J* = 24.4 Hz),27.8.

^{19}F NMR (376 MHz, $CDCl_3$) δ -71.28.

HRMS (ESI) calculated for $C_{18}H_{19}F_3N_2O_4$ $[M + Na]^+$: 407.1182, found 407.1189.



3l

tert-butyl (S)-(1,1,1-trifluoro-2-(naphthalen-2-yl)-3-nitropropan-2-yl)carbamate (**3l**)

White solid, 34.5 mg, 90% yield, **m. p.** = 60-62℃, $[\alpha]_D^{25}$ = -7.6 (*c* =0.1, $CHCl_3$).

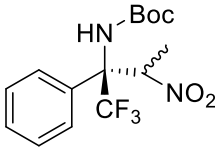
The ee value was 93% (Chiralpak AD-H, hexane/*i*-PrOH = 95:5, 214 nm, 1 mL/min, t_{major} = 16.50 min, t_{minor} = 12.14 min).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.95 – 7.82 (m, 4H), 7.59 – 7.51 (m, 3H), 5.71 – 5.50 (m, 3H), 1.46 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 153.7, 133.4, 132.9, 132.5, 130.0, 128.9, 128.5, 127.6, 127.4, 126.9, 124.6 (q, *J* = 325.0 Hz), 123.0, 122.6, 81.9, 73.3, 64.1 (q, *J* = 28.2 Hz)., 28.1.

^{19}F NMR (376 MHz, $CDCl_3$) δ -74.64.

HRMS (ESI) calculated for $C_{18}H_{19}F_3N_2O_4$ $[M + Na]^+$: 407.1190, found 407.1189.



3m

tert-butyl (1,1,1-trifluoro-3-nitro-2-phenylbutan-2-yl)carbamate (**3m**)

White solid, 27.1 mg, 78% yield, **m. p.** = 67-69℃, **$[\alpha]_D^{25}$** = -16.4 (*c* =0.1, $CHCl_3$).

The ee value was 91% (ChiralpakIA-3, hexane/*i*-PrOH= 98:2, 214nm, 1mL/min, Major-anti (t_{major}= 8.04 min, t_{minor} = 0.76 min), Major-syn (t_{major} = 5.84 min, t_{minor} = 6.87 min).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.40 (s, 5H), 6.10 (s, 1H), 5.28 (s, 1H), 1.71 (d, *J* = 6.8 Hz, 3H), 1.44 (s, 9H).

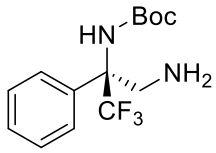
^{13}C NMR (101 MHz, $CDCl_3$) δ 153.3, 129.4, 128.9, 127.7 (d, *J* = 164.5 Hz), 125.9, 123.1, 81.4, 73.7, 67.2 (q, *J* = 27.8 Hz), 28.1, 15.1.

^{19}F NMR (376 MHz, $CDCl_3$) δ -63.48, -74.94, -81.83

HRMS (ESI) calculated for $C_{15}H_{19}F_3N_2O_4$ $[M + Na]^+$: 371.1192, found 371.1189.

4. General procedure of nitroamines 3a to diamines 3n

$NiCl_2$ 6H2O (0.024 g, 0.1 mmol) and $NaBH_4$ (0.019 g, 0.5 mmol) were added to a cooled (0℃) solution of **3a** (0.033 g, 0.1 mmol) in methanol (1 mL), and the resulting mixture was stirred for 40 min. The solvent was evaporated at room temperature. Ammonia (15% aqueous solution; 1 mL) was added to the residue, and the mixture was extracted with CH_2Cl_2 (3×5 mL). The combined organic layer was washed with ammonia (15% aqueous solution; 1 mL), water (2 mL), dried with Na_2SO_4, and evaporated. The residue was purified by column chromatography (CH_2Cl_2/CH_3OH = 20/1) to afford product **3n** as a yellow solid .



3n

tert-butyl (S)-(3-amino-1,1,1-trifluoro-2-phenylpropan-2-yl)carbamate (**3n**)

Yellow solid, 27.3 mg, 90% yield, **m. p.** = 77-78℃, **$[\alpha]_D^{25}$** = -18.4 (*c* =0.1, $CHCl_3$).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.36 (dq, *J* = 14.9, 7.7 Hz, 6H), 2.95 (d, *J* = 13.8 Hz, 1H), 1.65 (s, 2H), 1.32 (s, 9H).

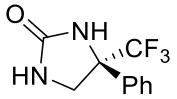
^{13}C NMR (101 MHz, DMSO) δ 154.3, 137.2, 128.0, 126.5, 123.3 (q, *J* = 378.8 Hz), 79.2, 65.6 (q, *J* = 24.6 Hz), 48.5, 28.4.

^{19}F NMR (376 MHz, DMSO) δ -67.46.

HRMS (ESI) calculated for $C_{14}H_{19}F_3N_2O_2$ $[M + H]^+$: 305.1469, found 305.1471.

5. General procedure for the preparation of imidazoline-2-ones 3o

DBU (0.0003 mmmol) was added to a solution of **3o** (0.030 g, 0.01mmol) in toluene (1 mL). The mixture was heated for 12 h, the it was cooled to room temperature and concentrated under reduced pressure. The residue was purified by column chromatography (EA/CH_3OH = 20/1) to afford product **3o** as a white solid.



3o

(S)-4-phenyl-4-(trifluoromethyl)imidazolidin-2-one (**3o**)

White solid, 20.7 mg, 90% yield, **m. p.** = 220℃, **$[\alpha]_D^{25}$** = -37.2 (*c* =0.1, $CHCl_3$)

The ee value was 93% (Chiralpak AD-H, hexane/*i*-PrOH = 95:5, 214 nm, 1 mL/min, t_{major} = 6.73 min, t_{minor} = 19.15 min).

^{1}H NMR (400 MHz, DMSO-d_6) δ 8.30 (s, 1H), 7.53 – 7.35 (m, 5H), 6.78 (s, 1H), 4.01 (d, *J* = 10.4 Hz, 1H), 3.66 (d, *J* = 10.4 Hz, 1H).

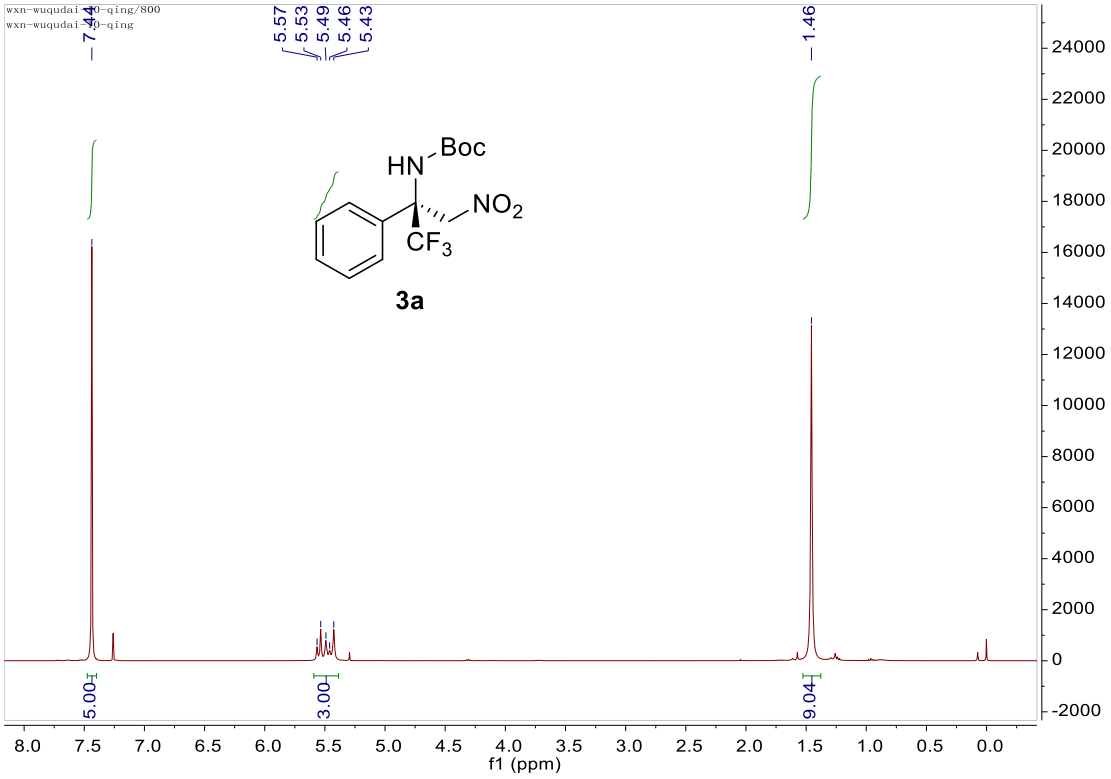
^{13}C NMR (101 MHz, DMSO) δ 161.7, 137.6, 129.2, 128.9, 127.2, 126.3 (q, *J* = 286.7 Hz),126.0 ,64.8

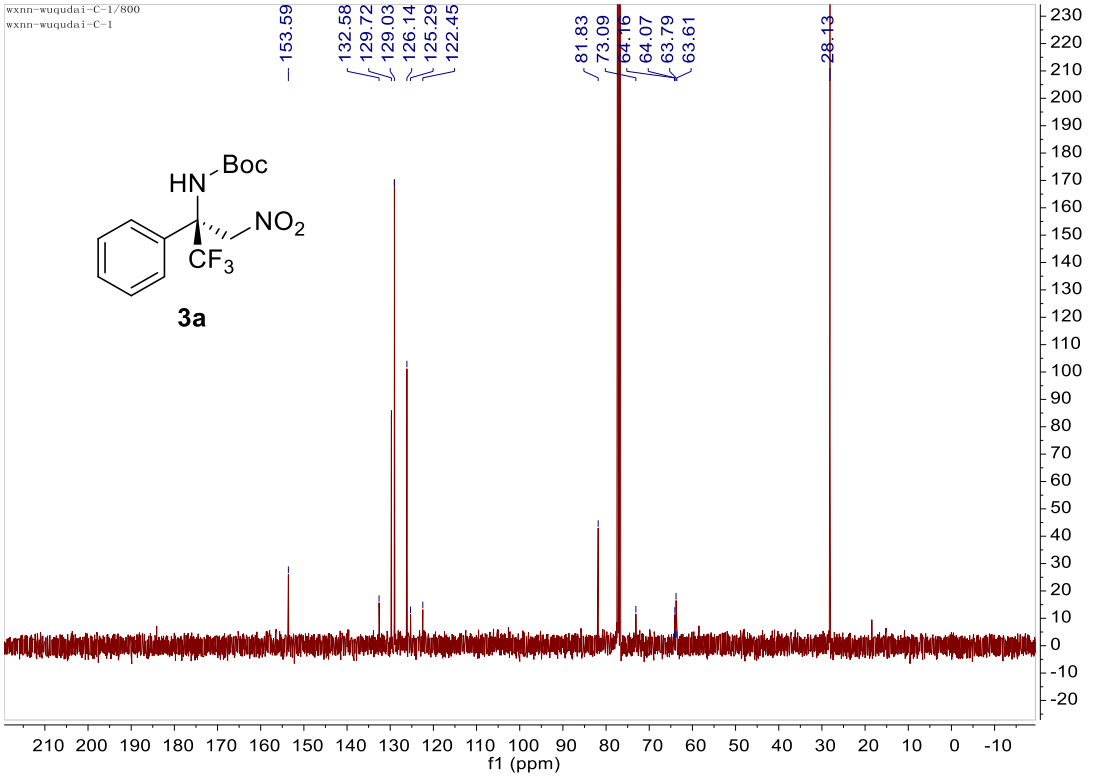
(q, *J* = 28.2 Hz), 48.1.

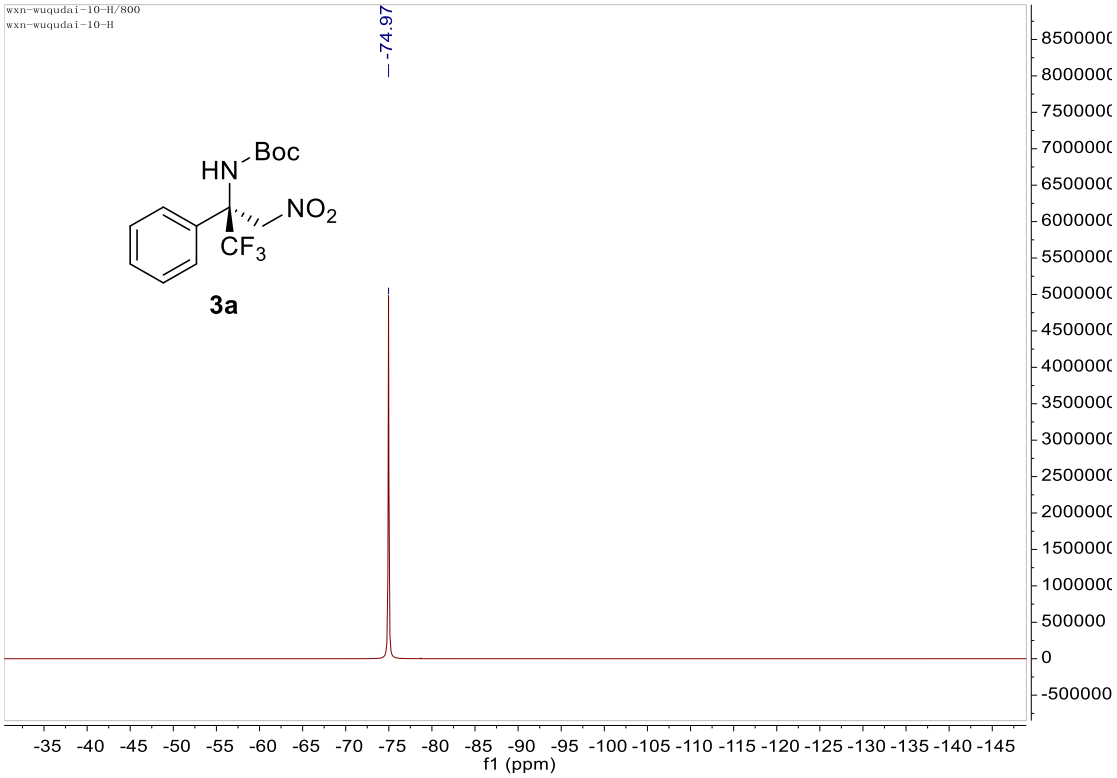
^{19}F NMR (376 MHz, DMSO) δ -78.70.

HRMS (ESI) calculated for $C_{10}H_9F_3N_2O[M + H]^+$: 231.0741, found 231.0740.

6. NMR spectra of addition products 3a-3m and derivatives 3n,3o.



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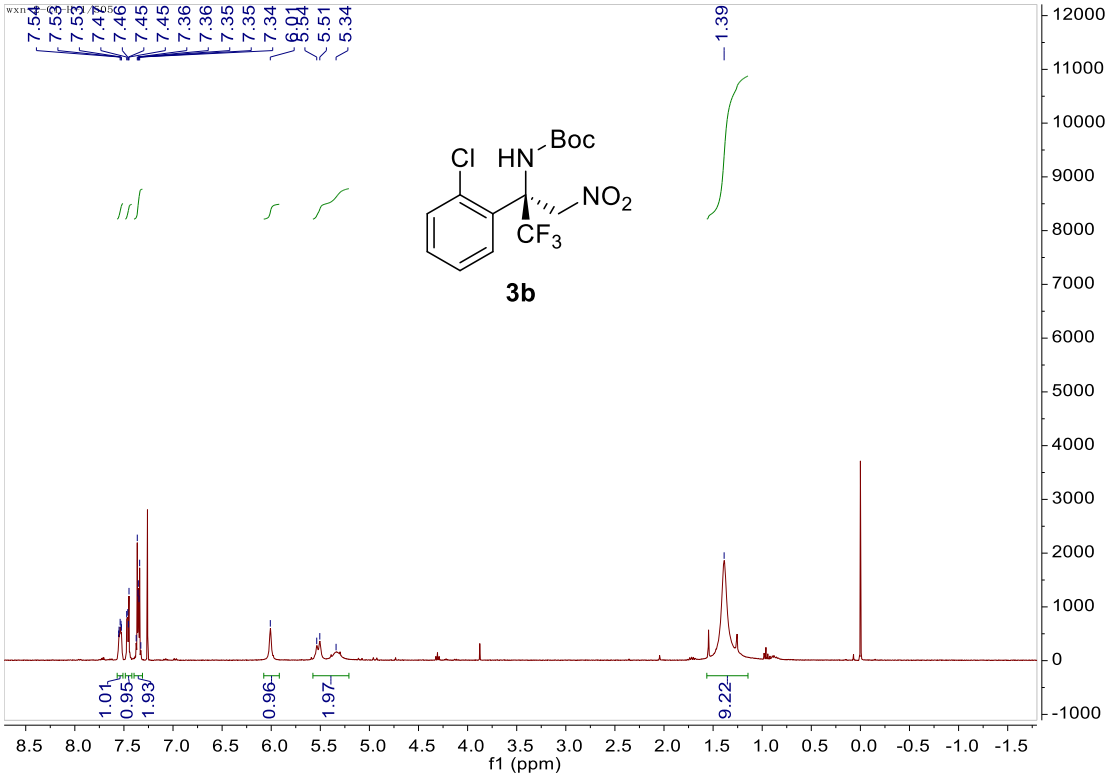
HN
Boc
NO2
CF3
3a
f1 (ppm)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6.01
5.54
5.51
5.34
1.39
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HN
Boc
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CF3
3b
1.01
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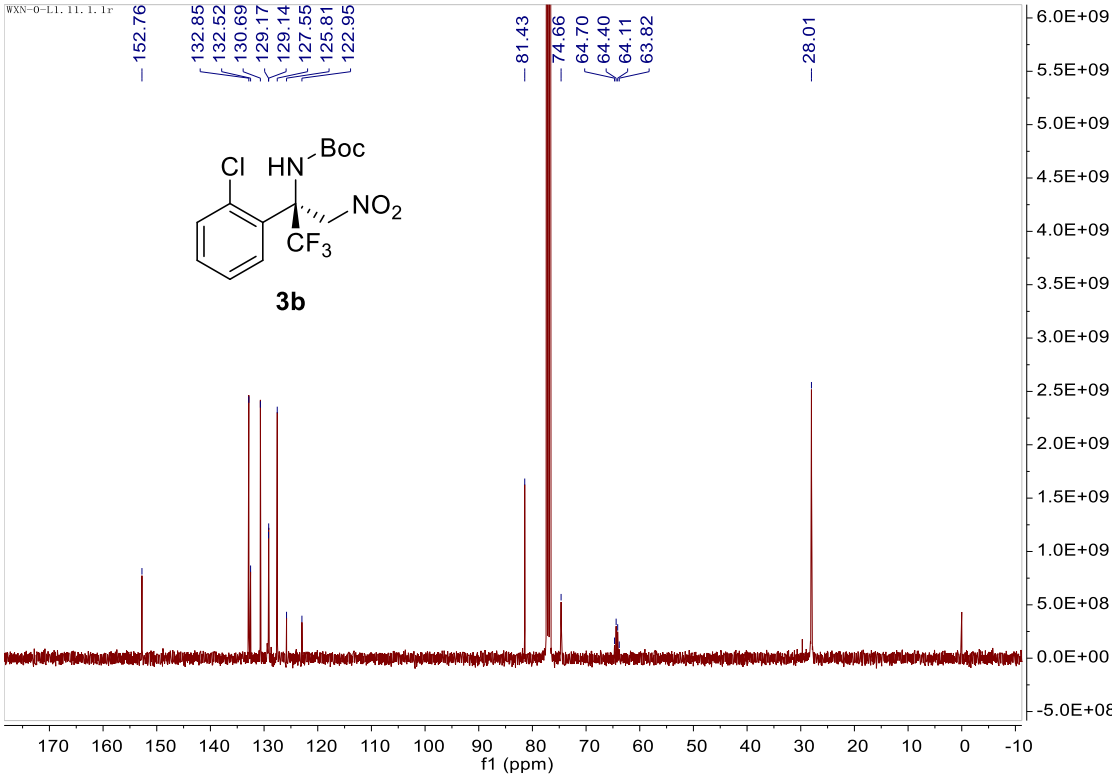
0.96
1.97
9.22
f1 (ppm)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64.40
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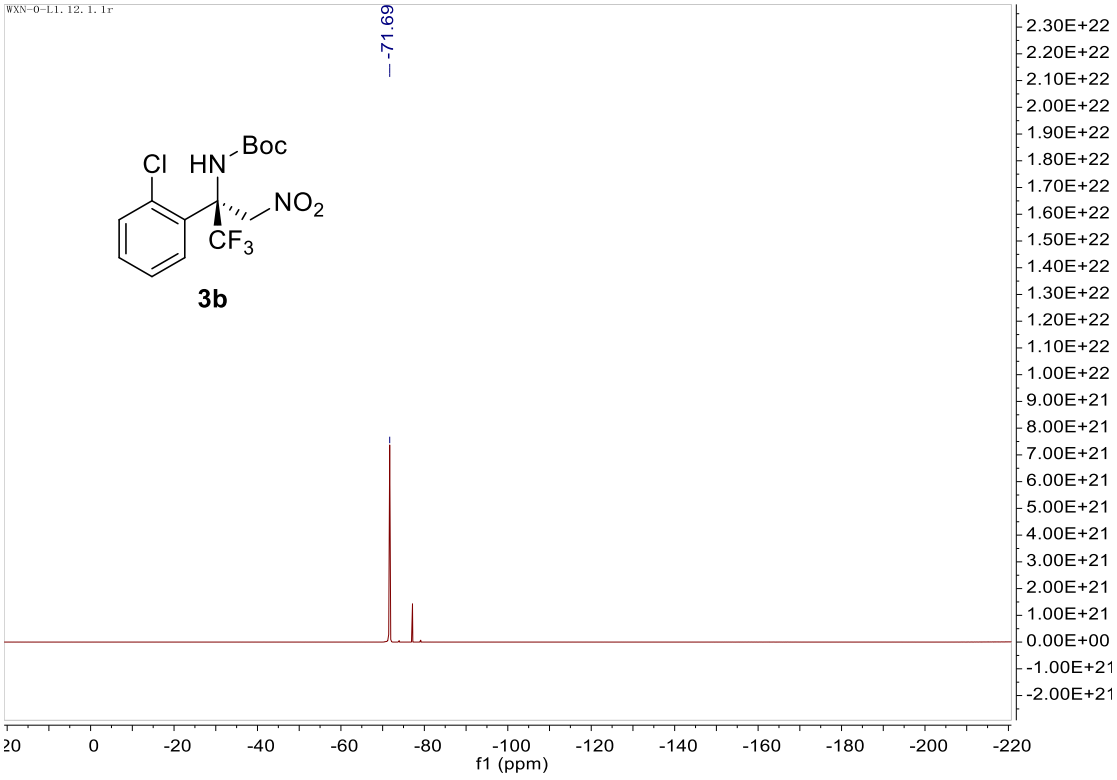
28.01
Cl
HN
Boc
NO2
CF3
3b
f1 (ppm)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Boc
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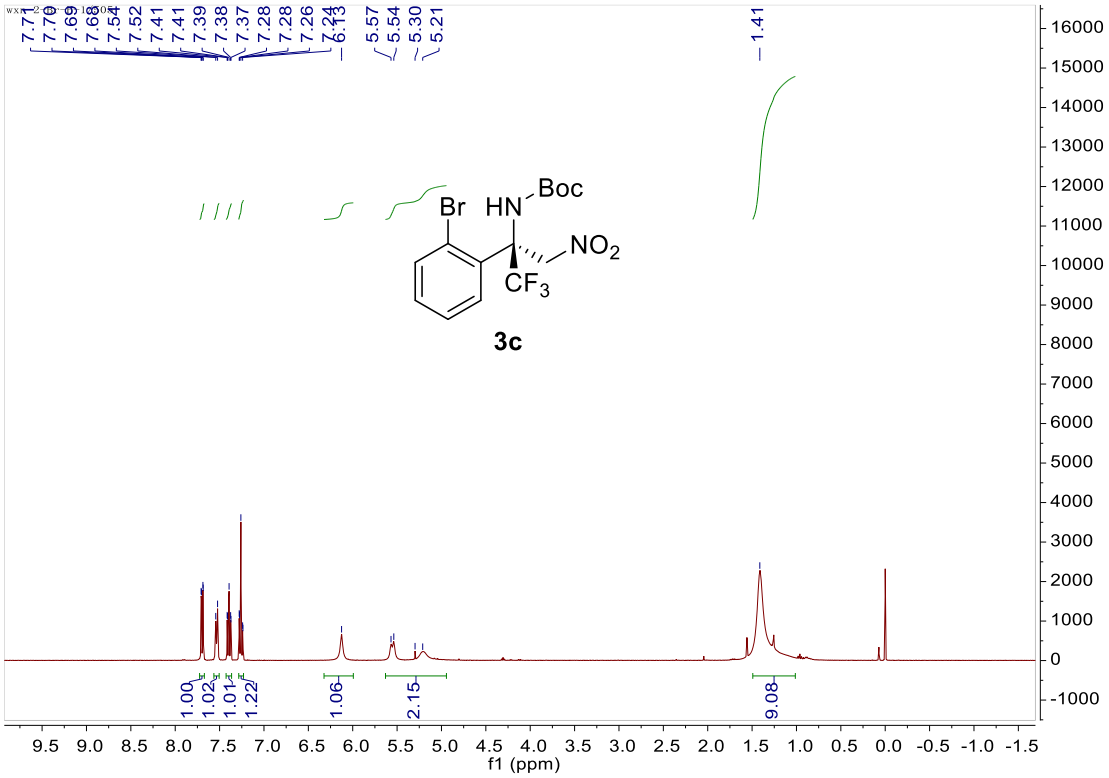
CF3
3b
f1 (ppm)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7.26
7.24
6.13
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5.30
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Br
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Boc
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CF3
3c
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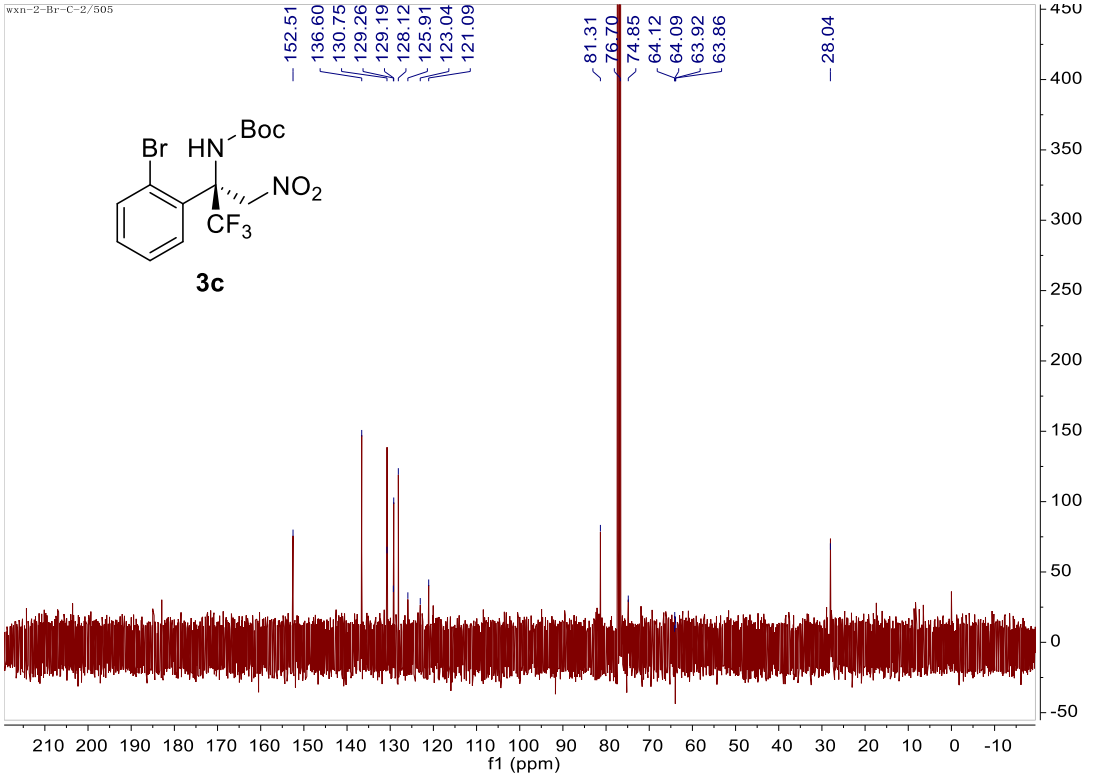
1.06
2.15
9.08
f1 (ppm)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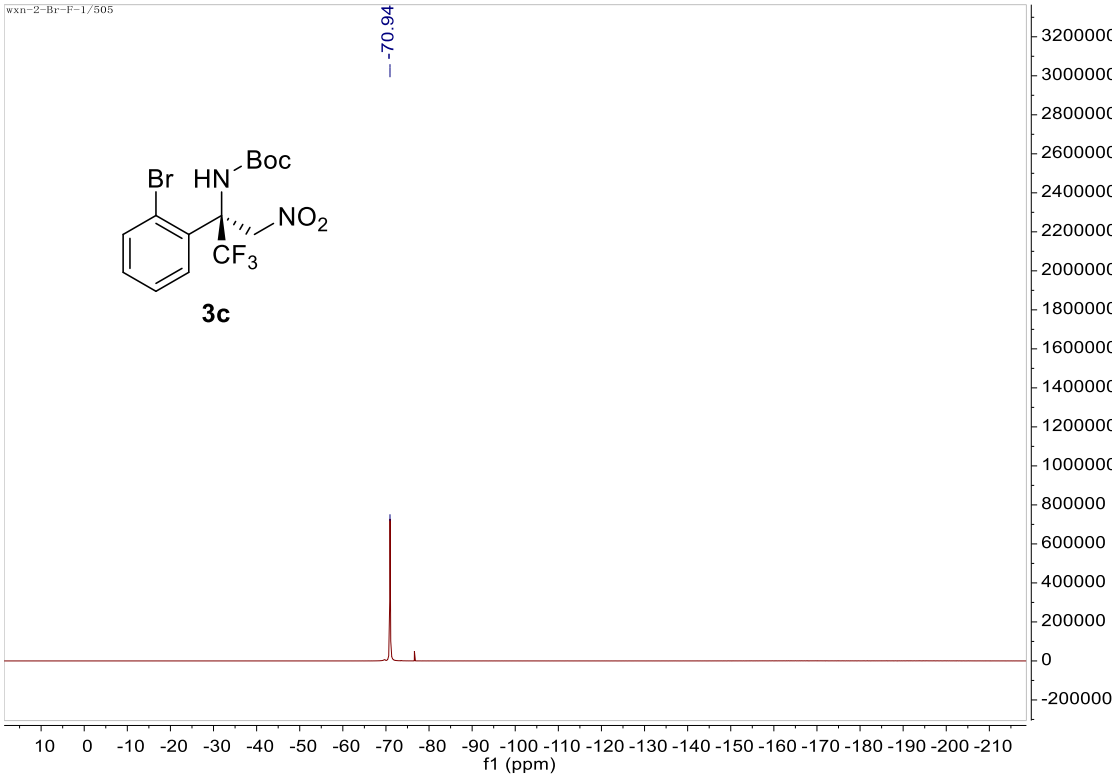
Br
HN
Boc
NO2
CF3
3c
f1 (ppm)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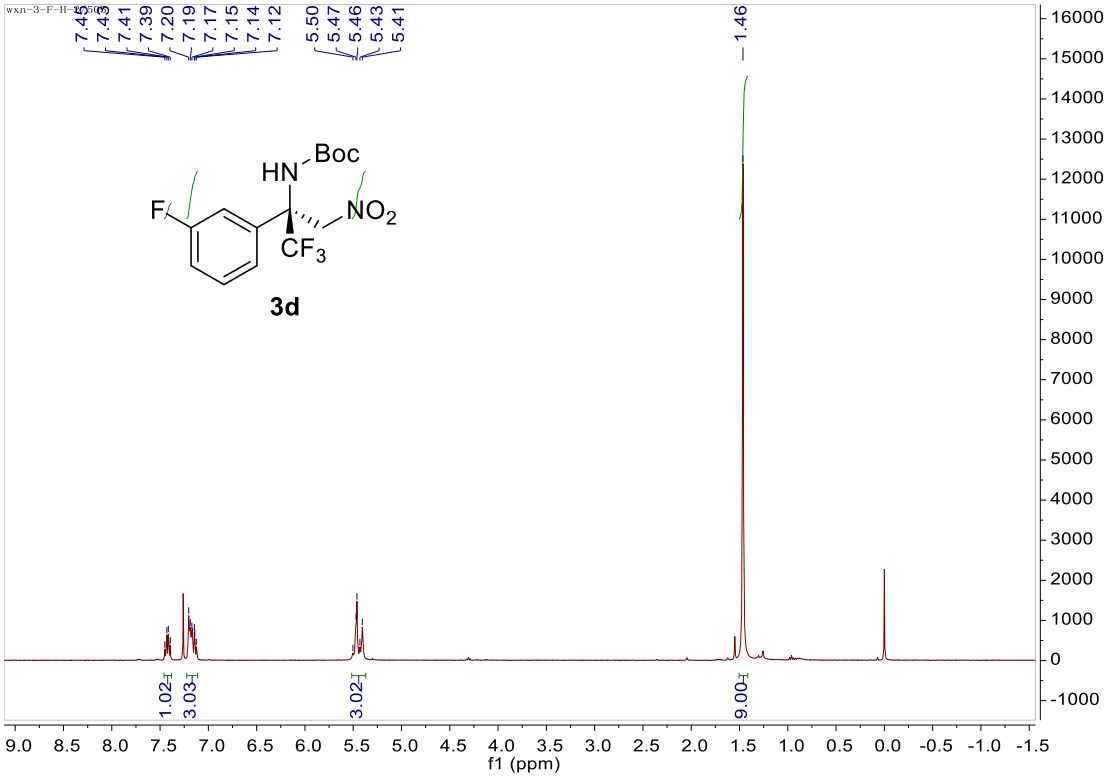
3c
f1 (ppm)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5.46
5.43
5.41
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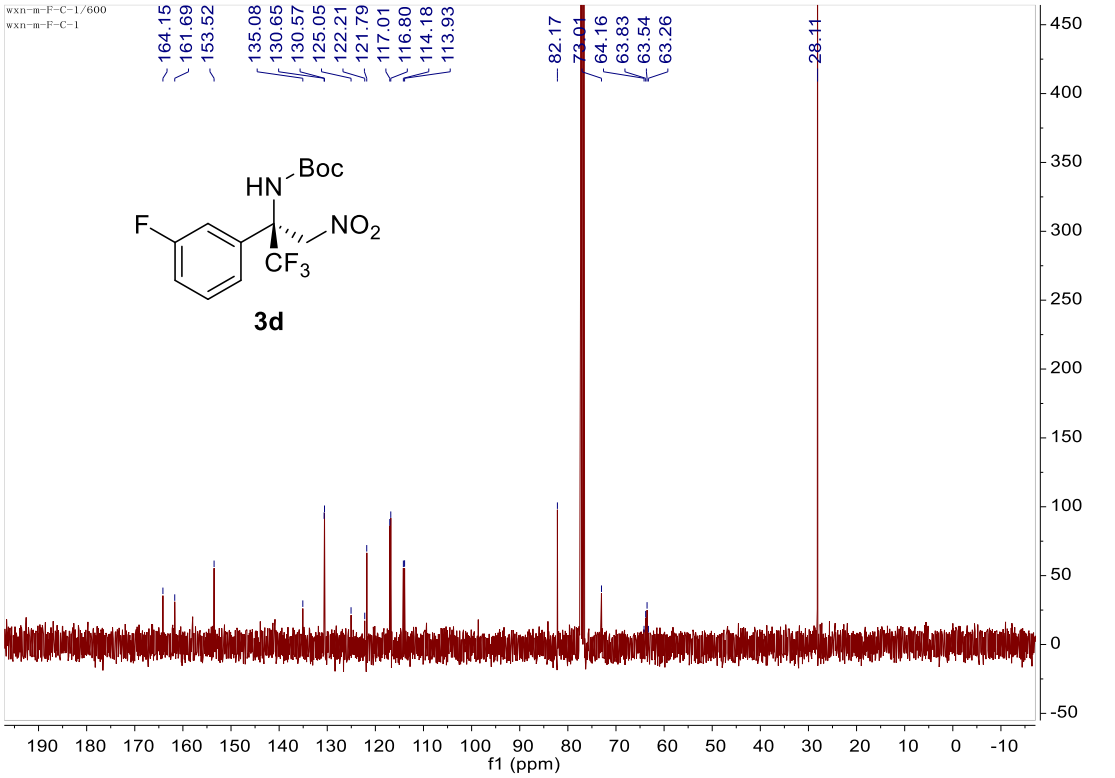
Boc
F
NO2
CF3
3d
1.02
3.03
3.02
9.00
f1 (ppm)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117.01
116.80
114.18
113.93
82.17
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63.54
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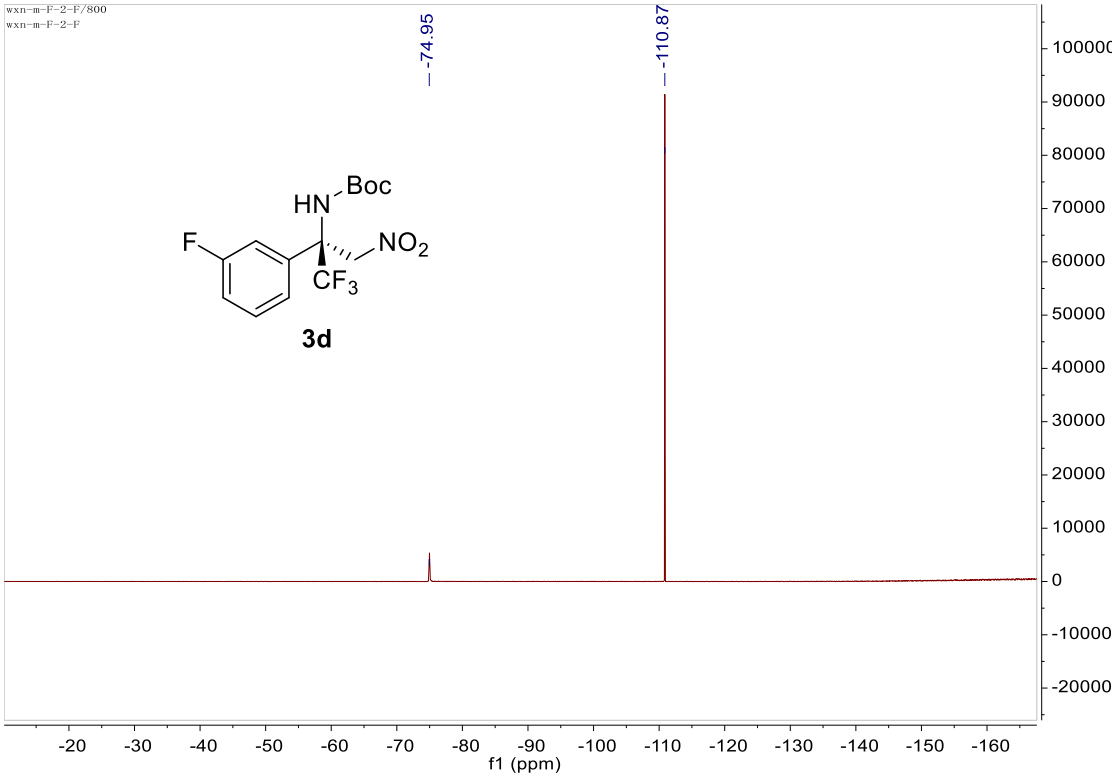
HN
F
NO2
CF3
3d
f1 (ppm)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HN
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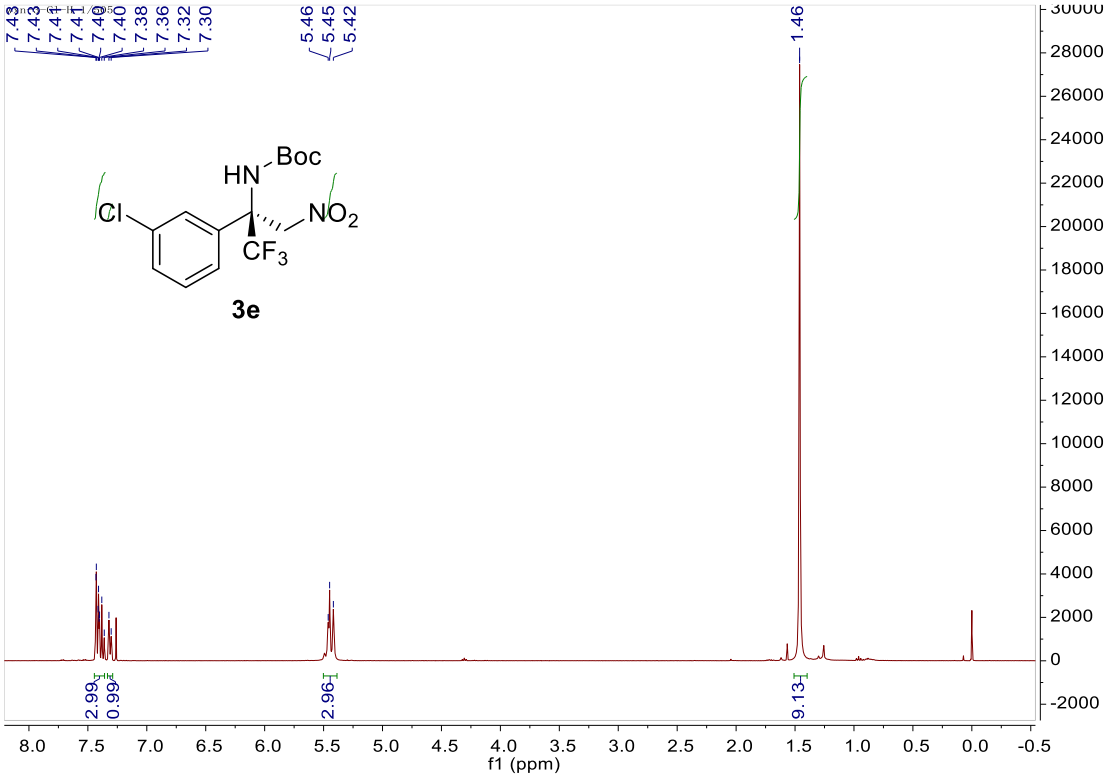
f1 (ppm)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Cl
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CF3
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0.99
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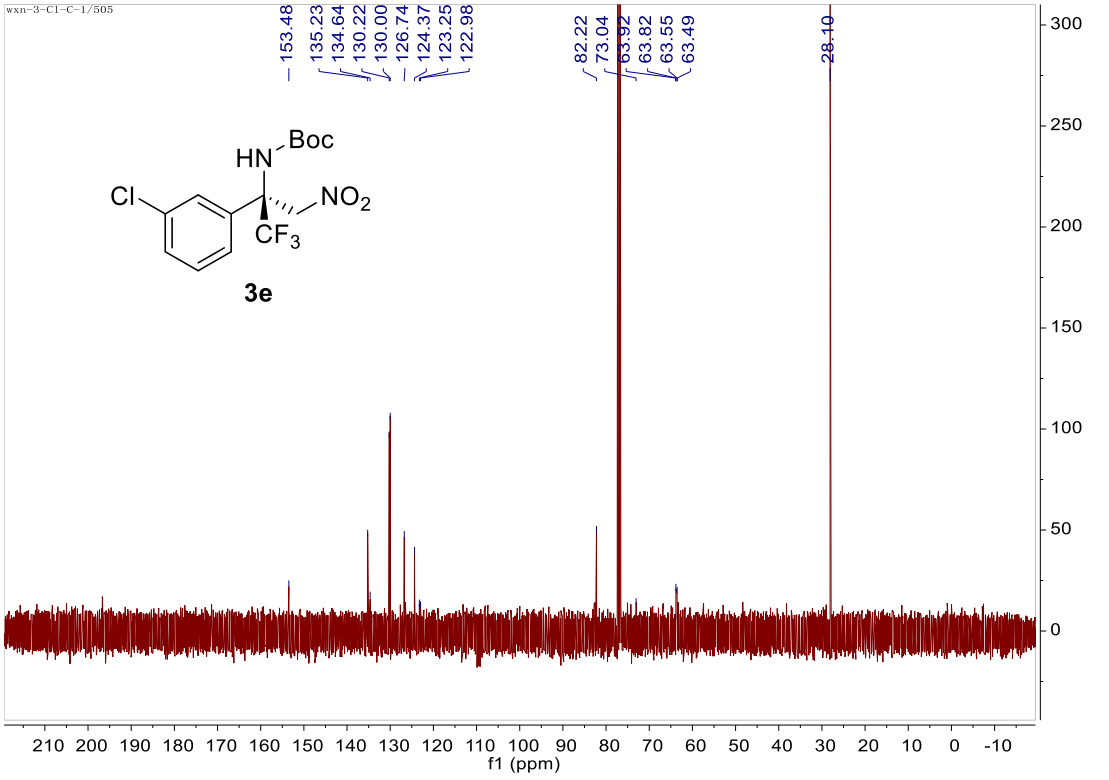
f1 (ppm)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Boc
Cl
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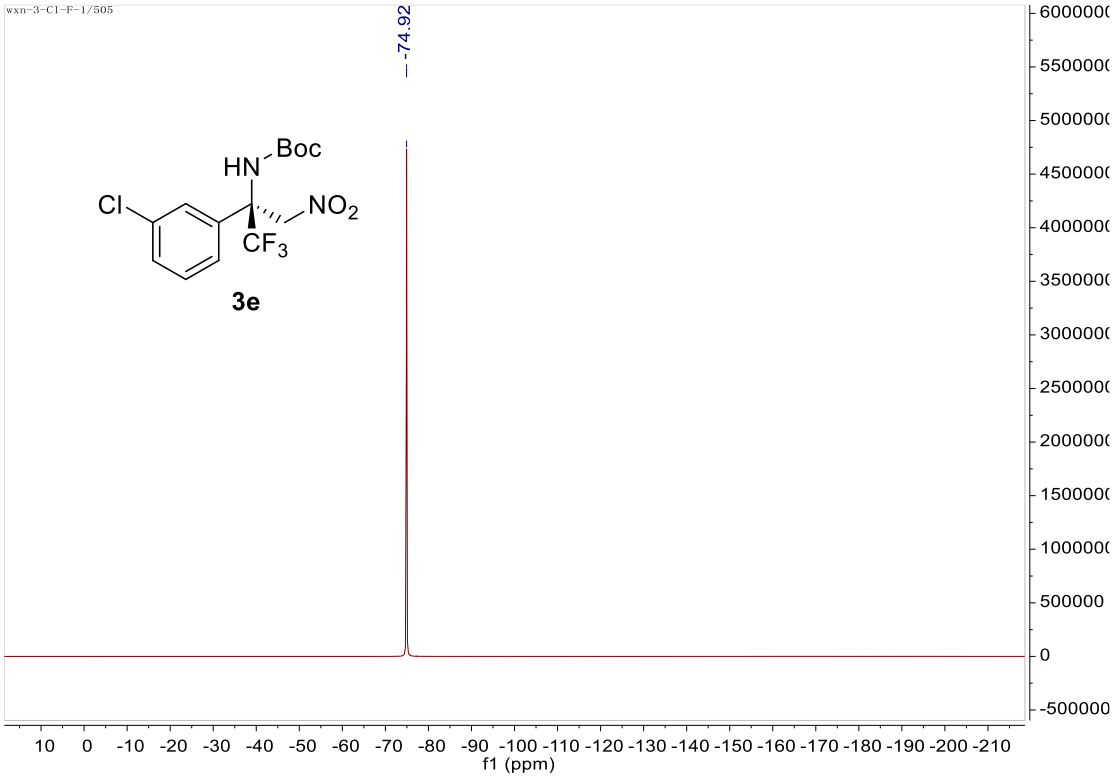
CF3
3e
f1 (ppm)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Boc
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CF3
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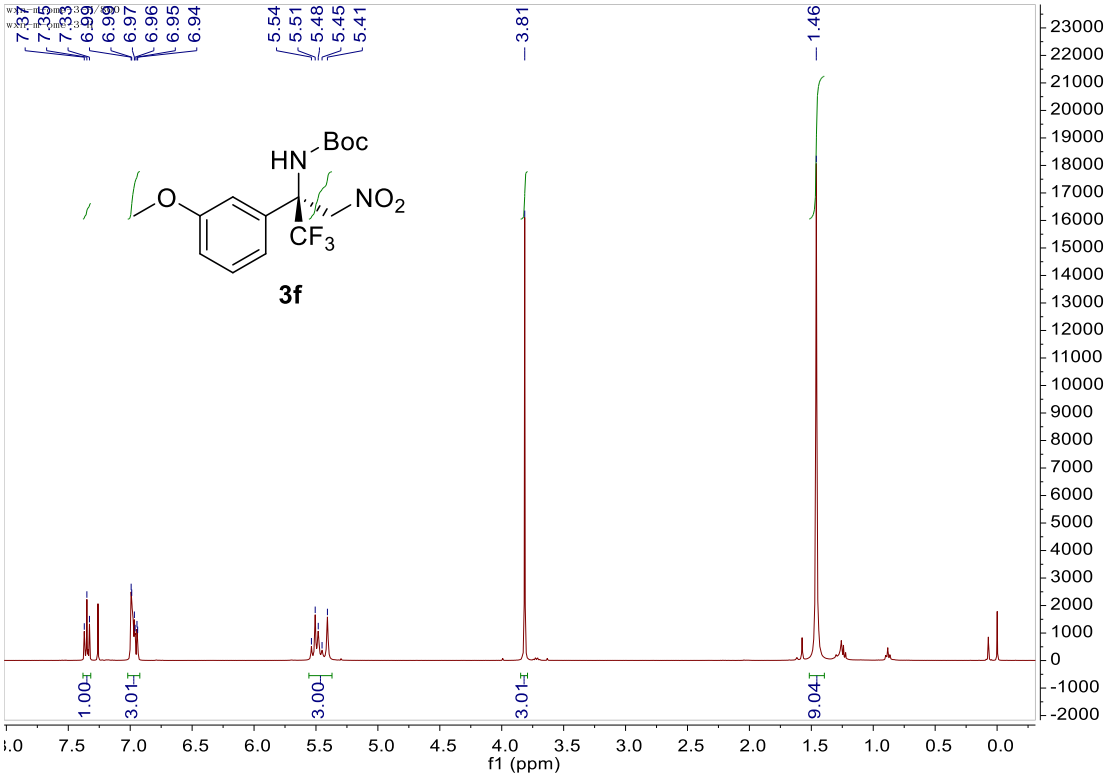
f1 (ppm)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6.96
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6.94
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5.51
5.48
5.45
5.41
3.81
1.46
HN
Boc
O
NO2
CF3
3f
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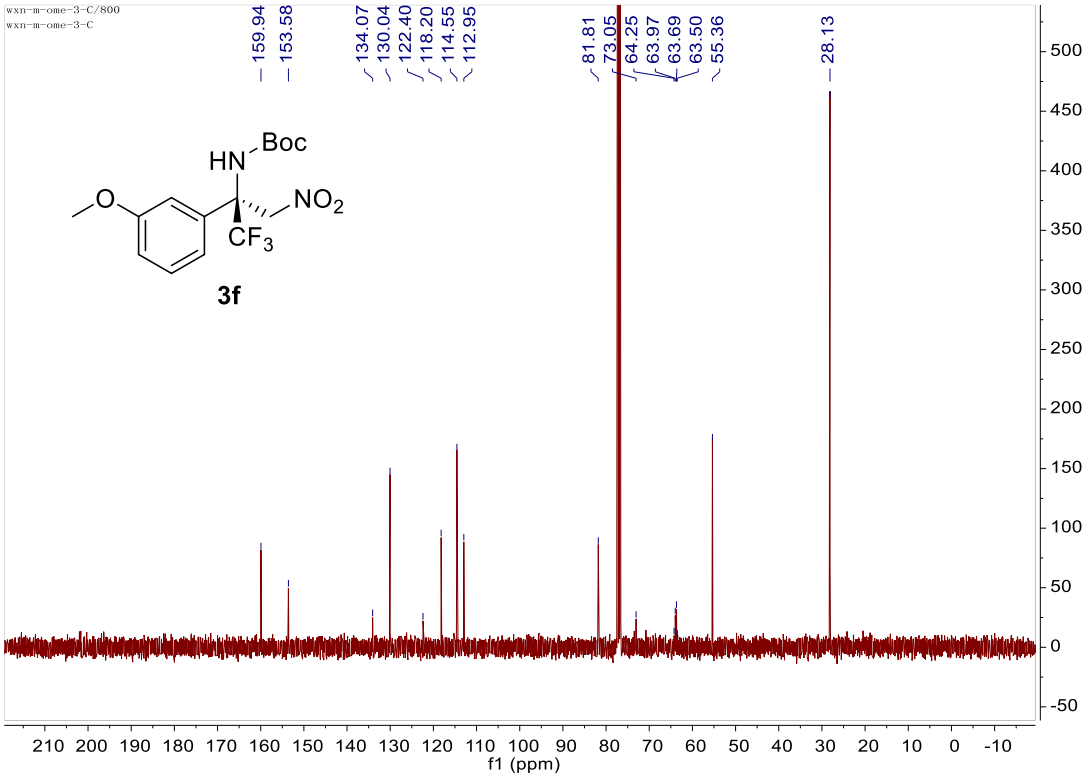
3.01
3.00
3.01
9.04
f1 (ppm)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wxn-m-ome-3-C
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118.20
114.55
112.95
81.81
73.05
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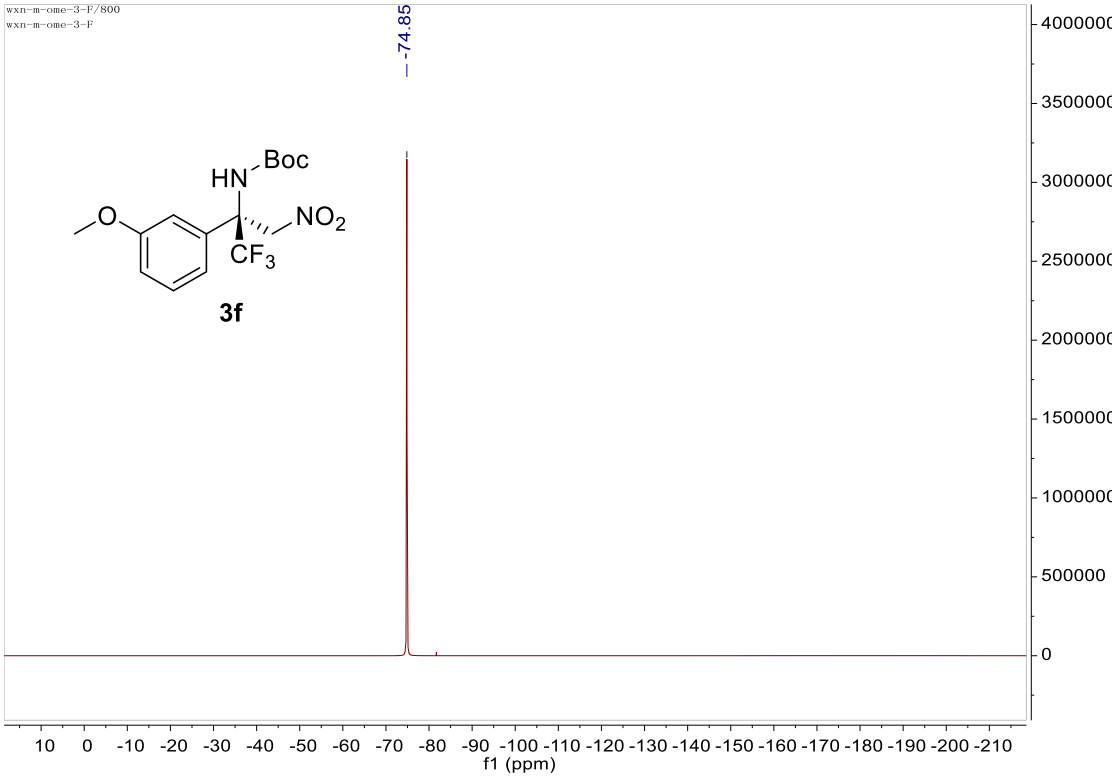
Boc
HN
O
NO2
CF3
3f
f1 (ppm)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wxn-m-ome-3-F
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CF3
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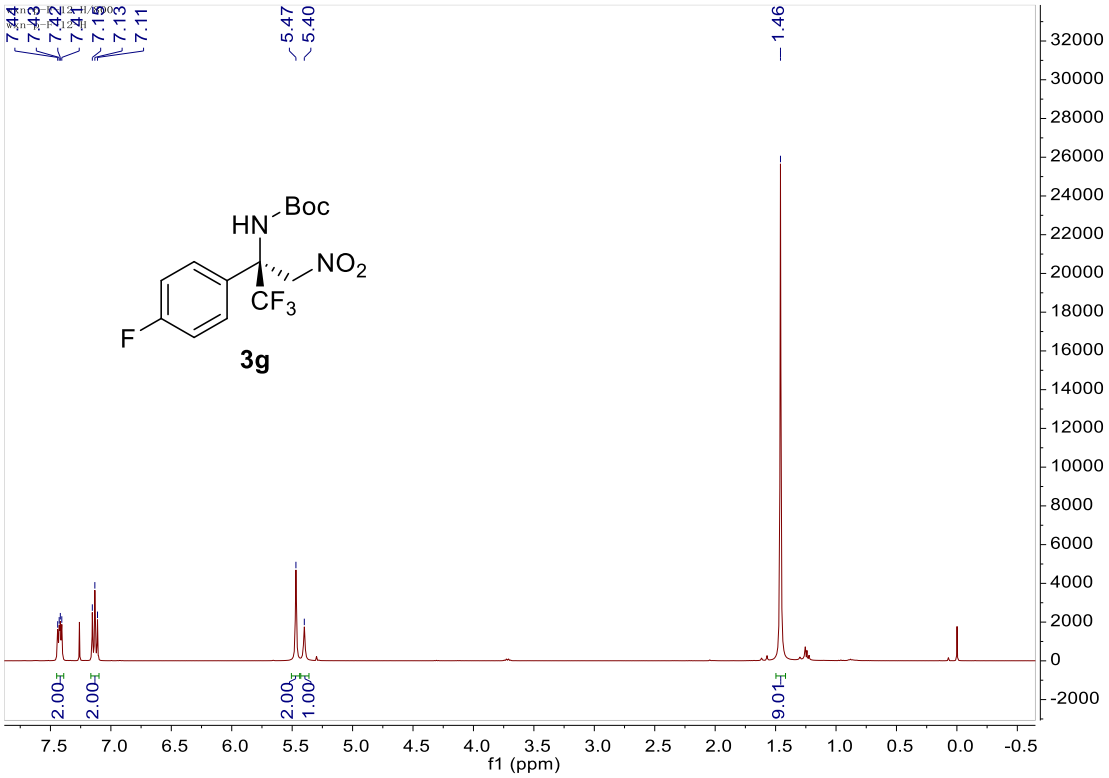
f1 (ppm)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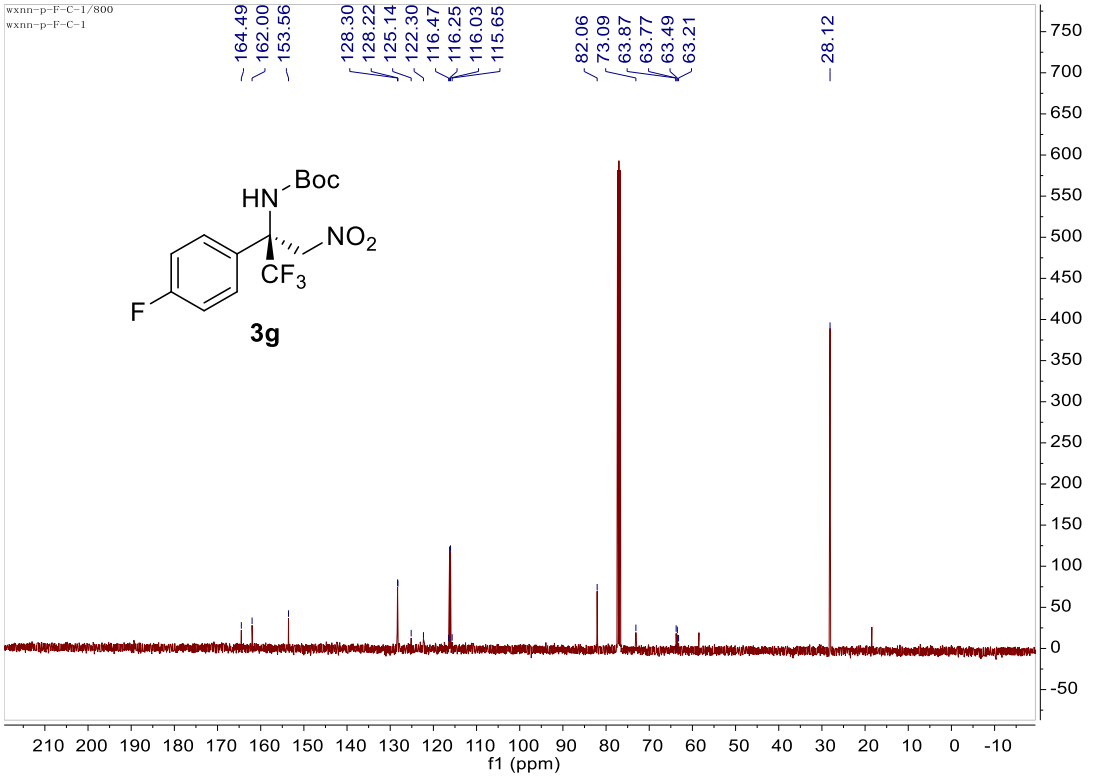
Boc
NO2
CF3
F
3g
2.00
2.00
2.00
1.00
9.01
f1 (ppm)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63.21
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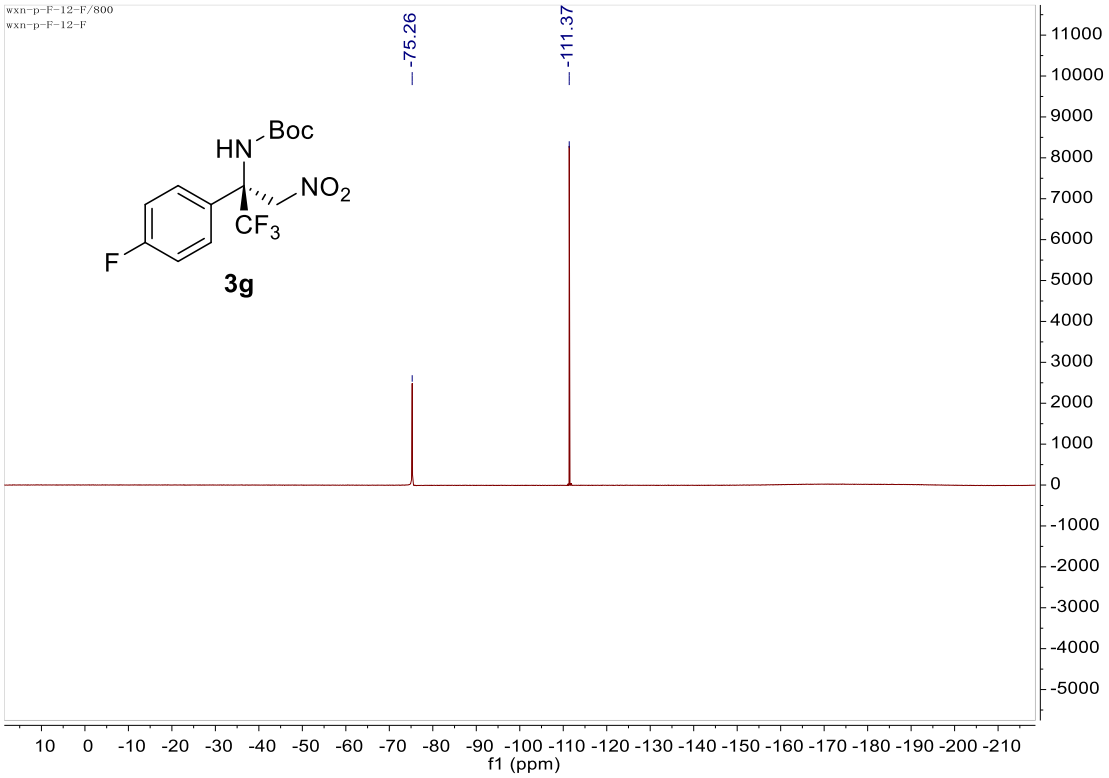
Boc
NO2
CF3
F
3g
f1 (ppm)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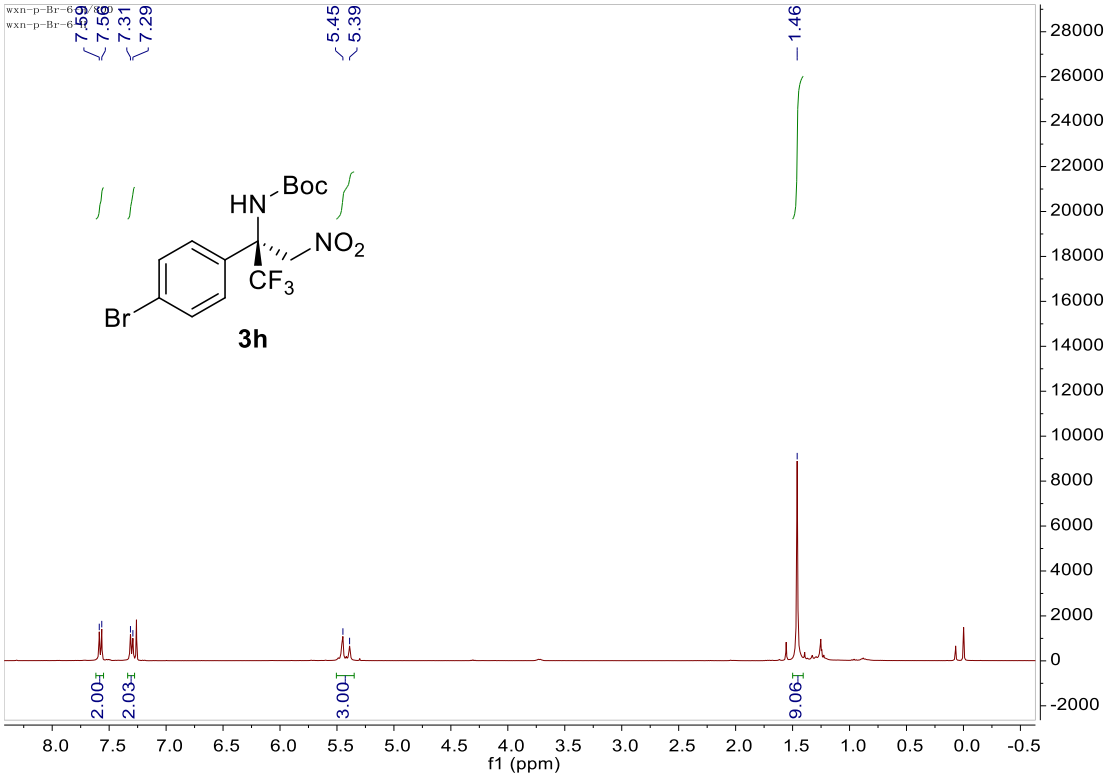
Boc
NO2
CF3
F
3g
f1 (ppm)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CF3
Br
3h
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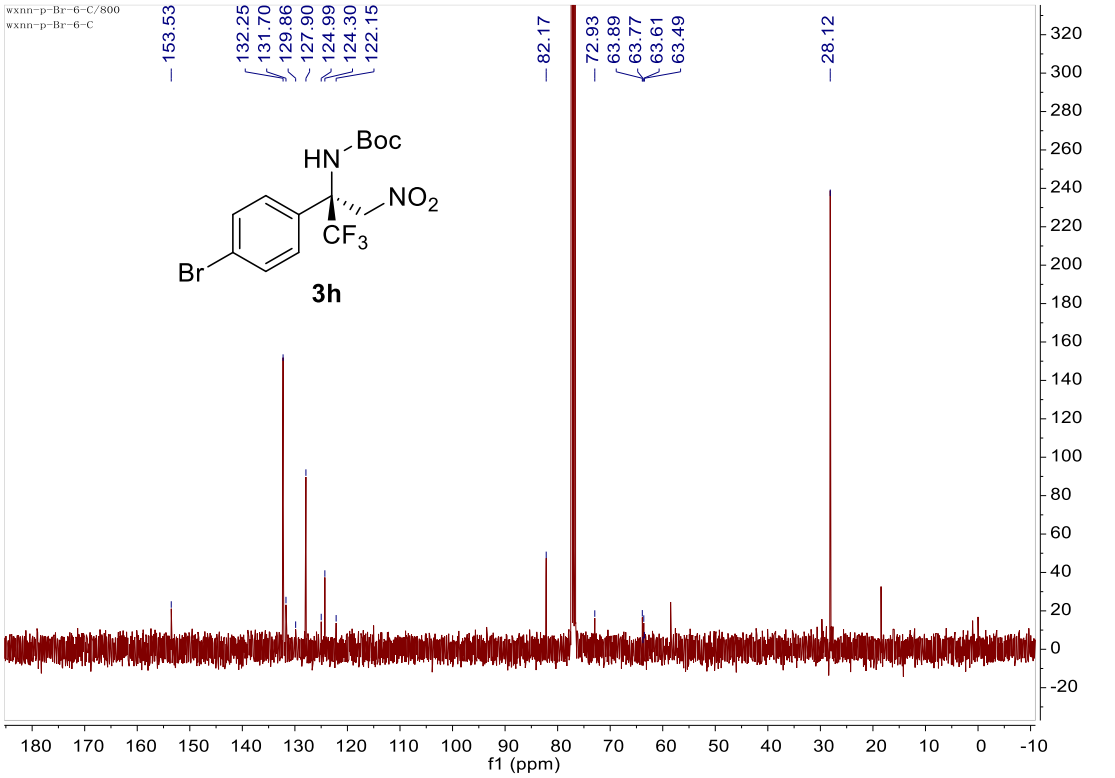
2.03
3.00
9.06
f1 (ppm)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wxnn-p-Br-6-C
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82.17
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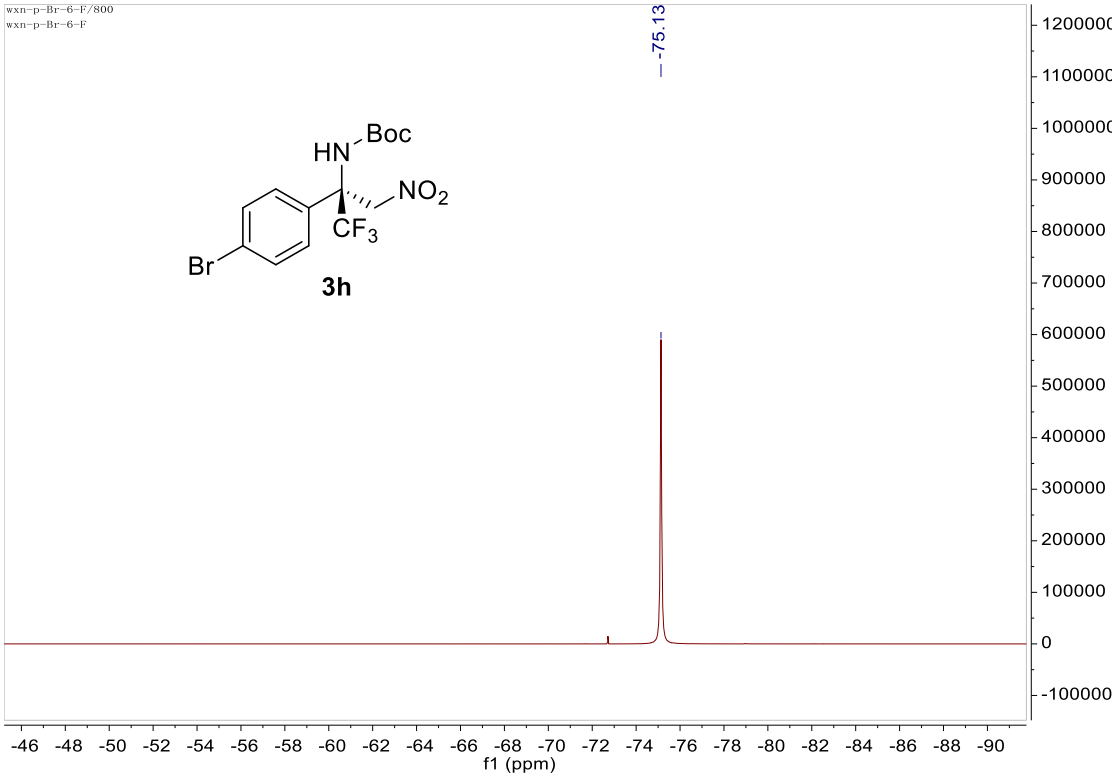
28.12
HN
Boc
NO2
CF3
Br
3h
f1 (ppm)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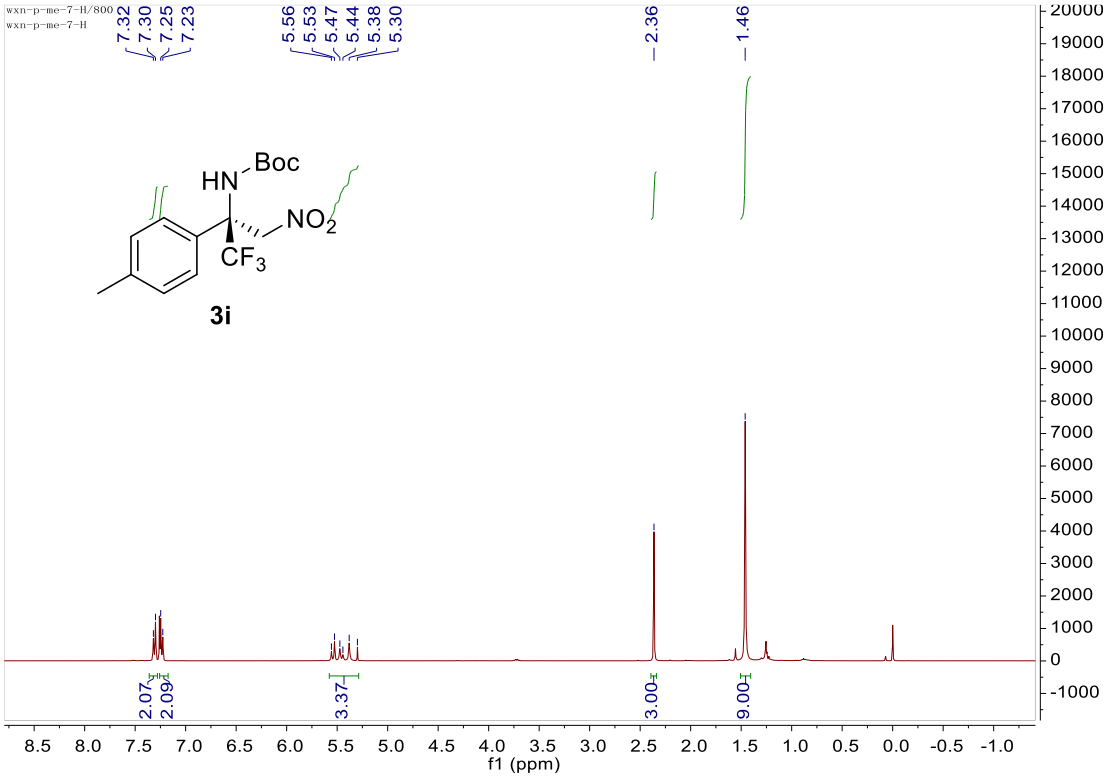
Br
3h
f1 (ppm)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5.44
5.38
5.30
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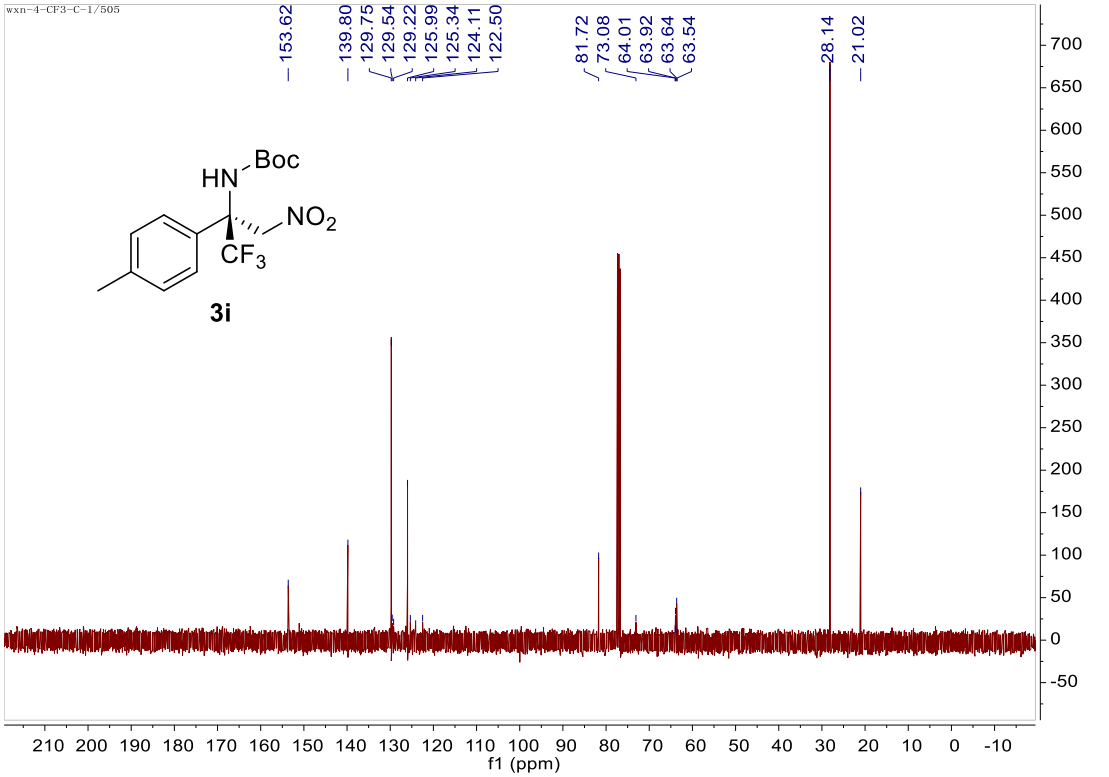
Boc
NO2
CF3
3i
2.07
2.09
3.37
3.00
9.00
f1 (ppm)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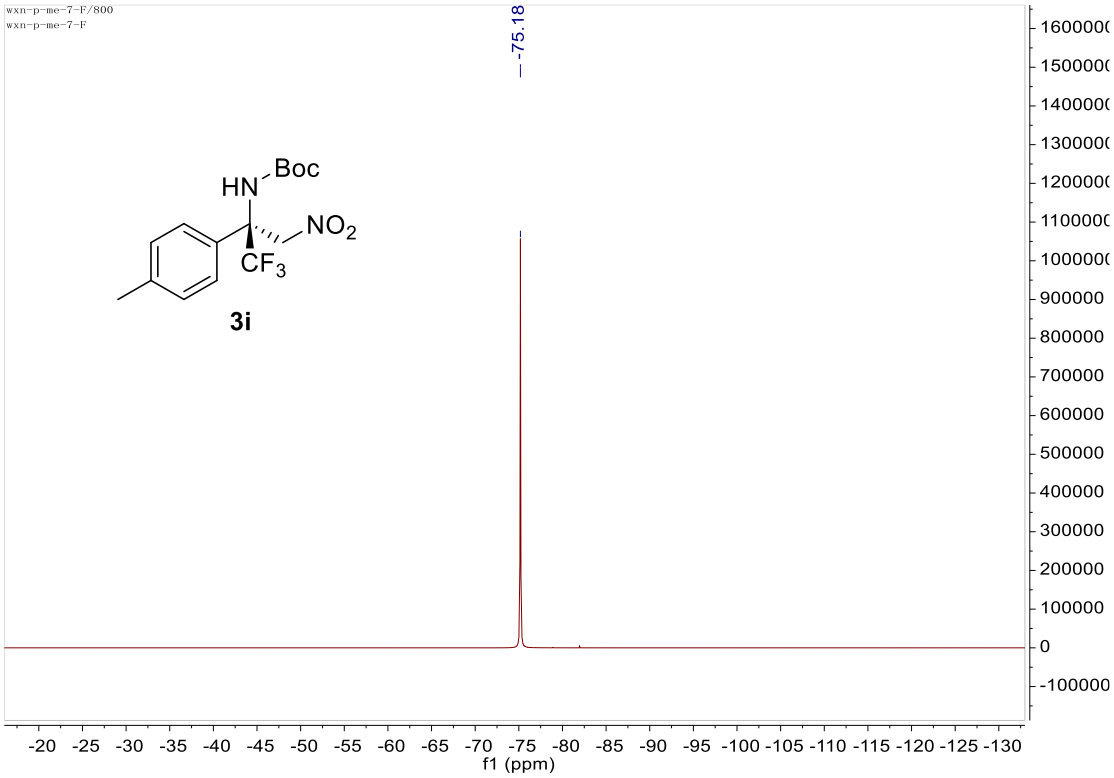
63.54
28.14
21.02
HN
Boc
NO2
CF3
3i
f1 (ppm)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Boc
NO2
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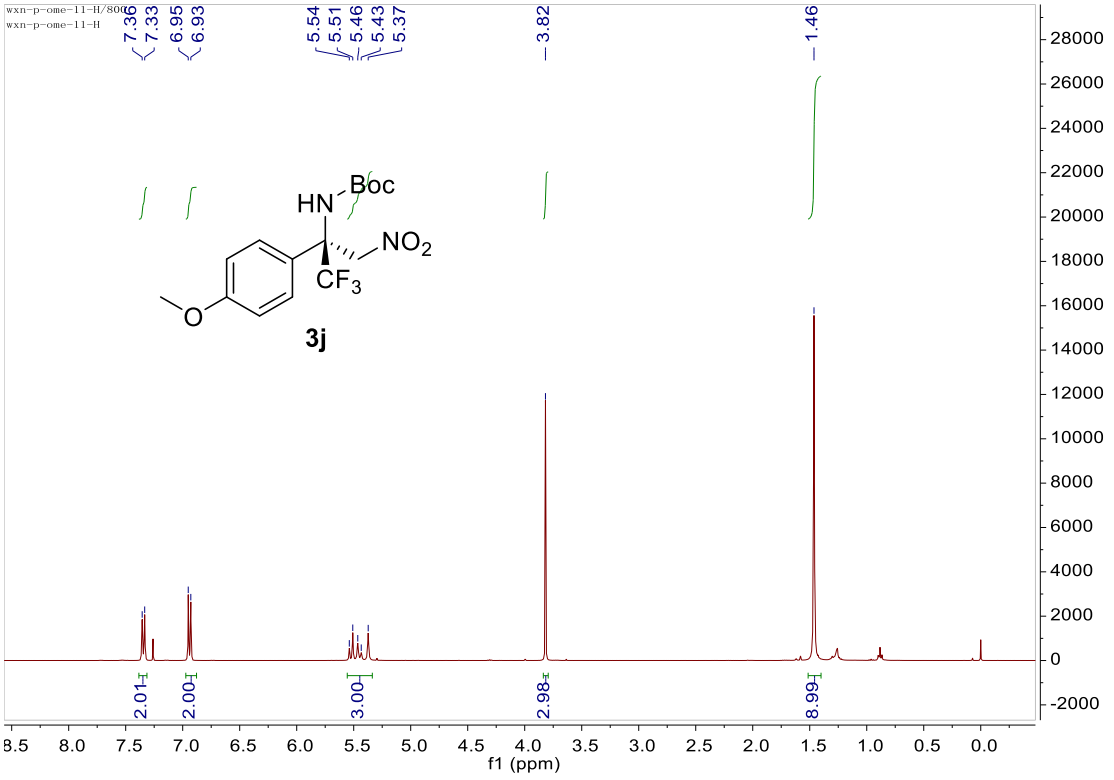
3i
f1 (ppm)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5.51
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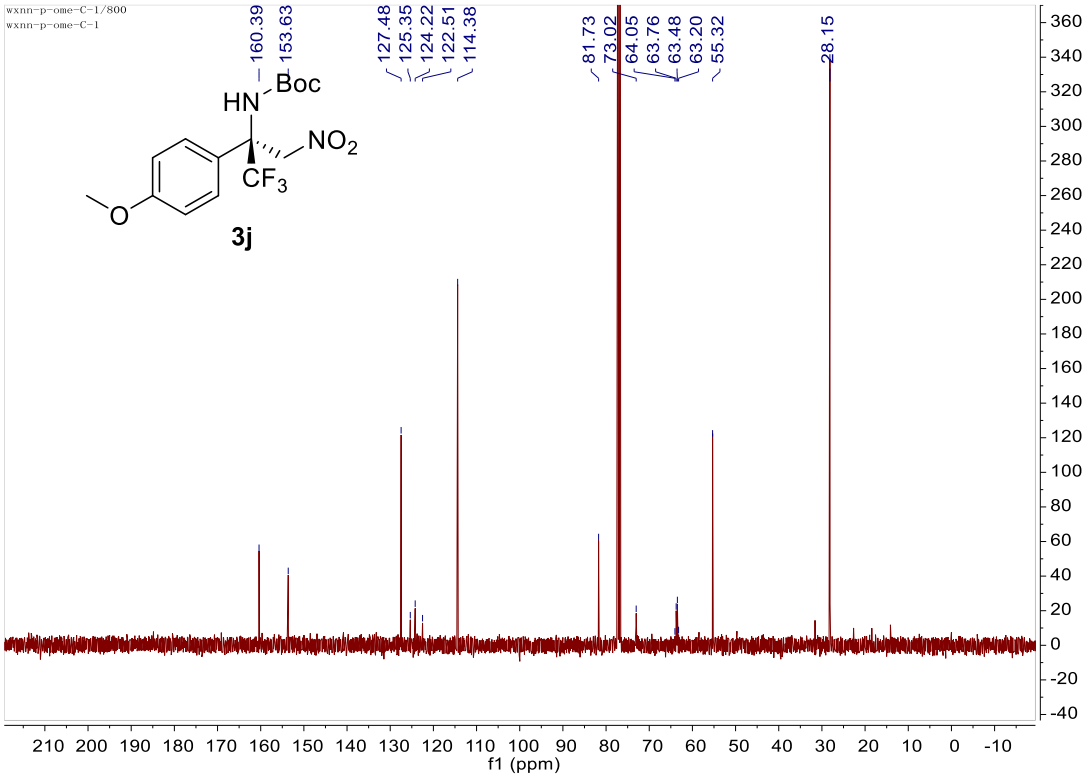
5.43
5.37
3.82
1.46
HN
Boc
NO2
CF3
O
3j
2.01
2.00
3.00
2.98
8.99
f1 (ppm)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wxnn-p-ome-C-1
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153.63
127.48
125.35
124.22
122.51
114.38
81.73
73.02
64.05
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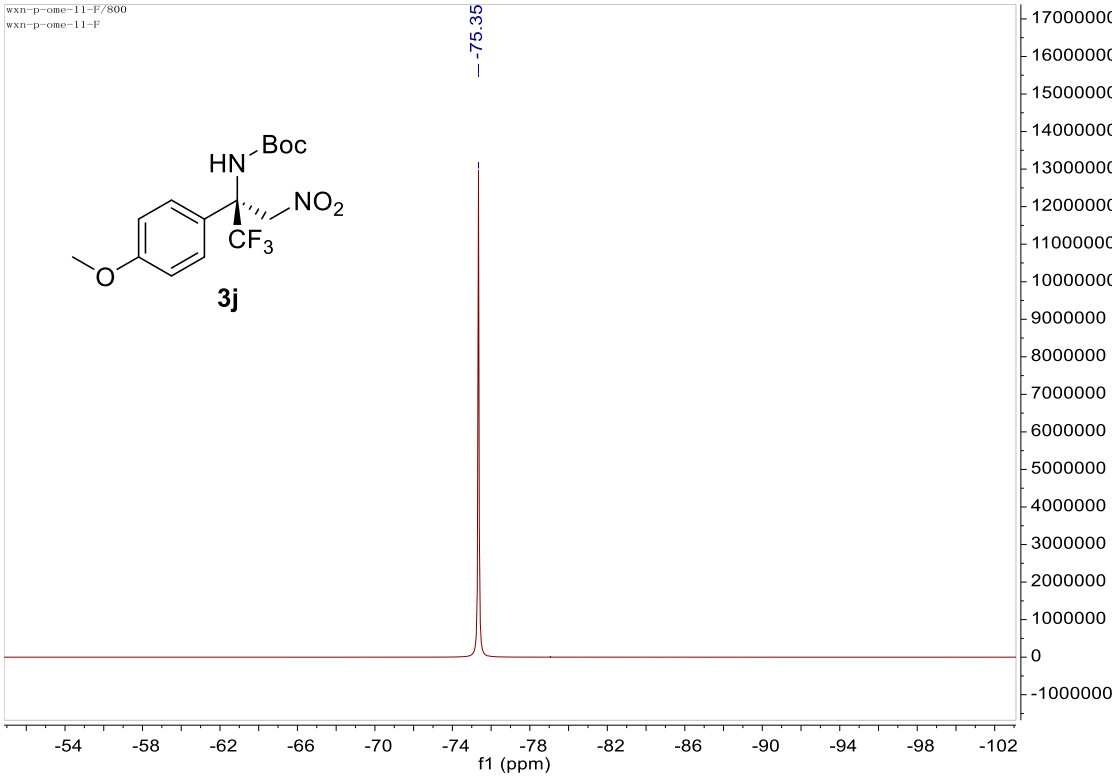
63.48
63.20
55.32
28.15
Boc
HN
NO2
CF3
O
3j
f1 (ppm)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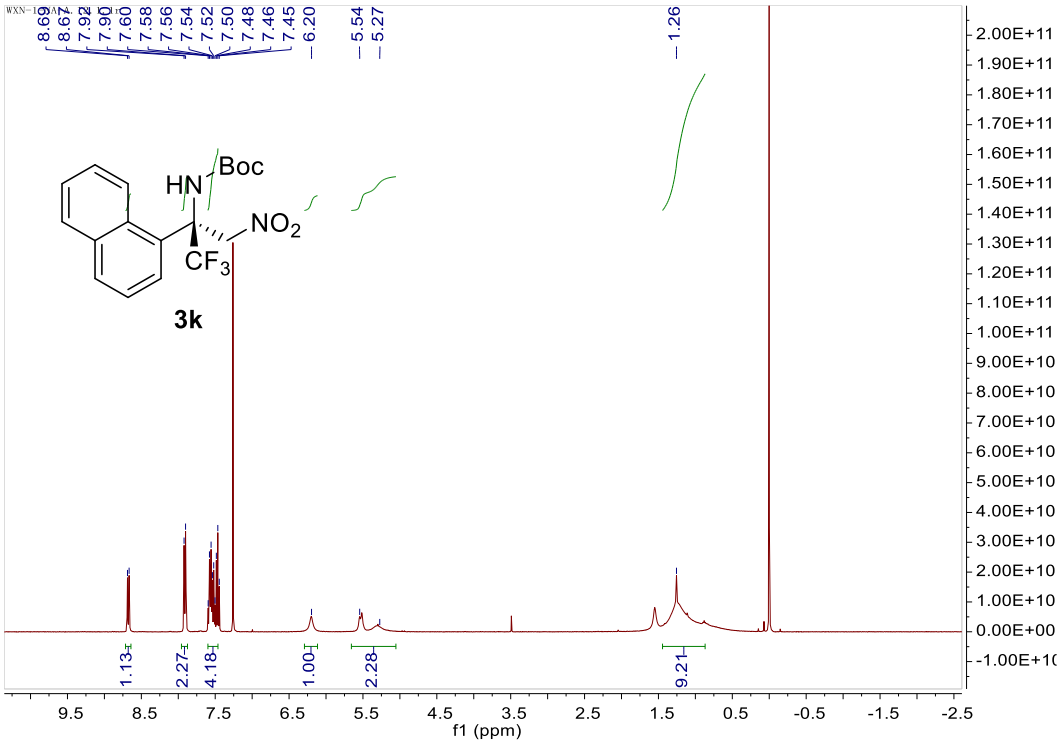
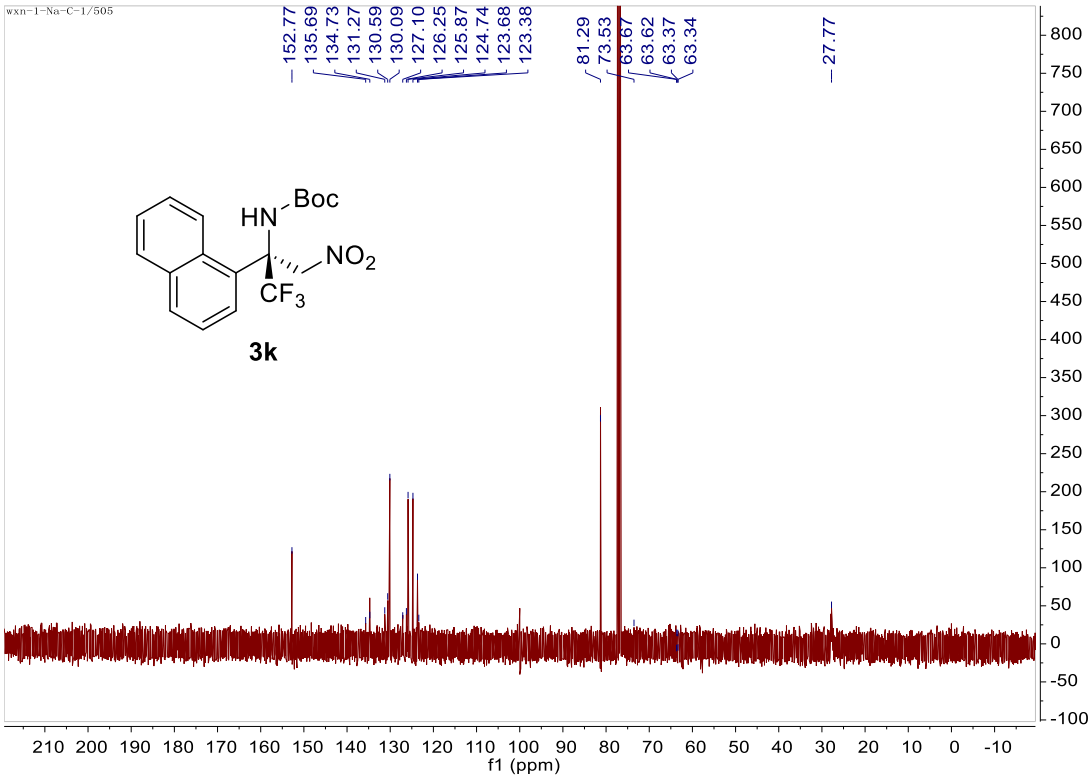
Boc
HN
NO2
CF3
O
3j
f1 (ppm)


8.69
8.67
7.92
7.90
7.60
7.58
7.56
7.54
7.52
7.50
7.48
7.46
7.45
6.20
5.54
5.27
1.26
HN
Boc
NO2
CF3
3k
1.13
2.27
4.18
1.00
2.28
9.21
f1 (ppm)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126.25
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123.68
123.38
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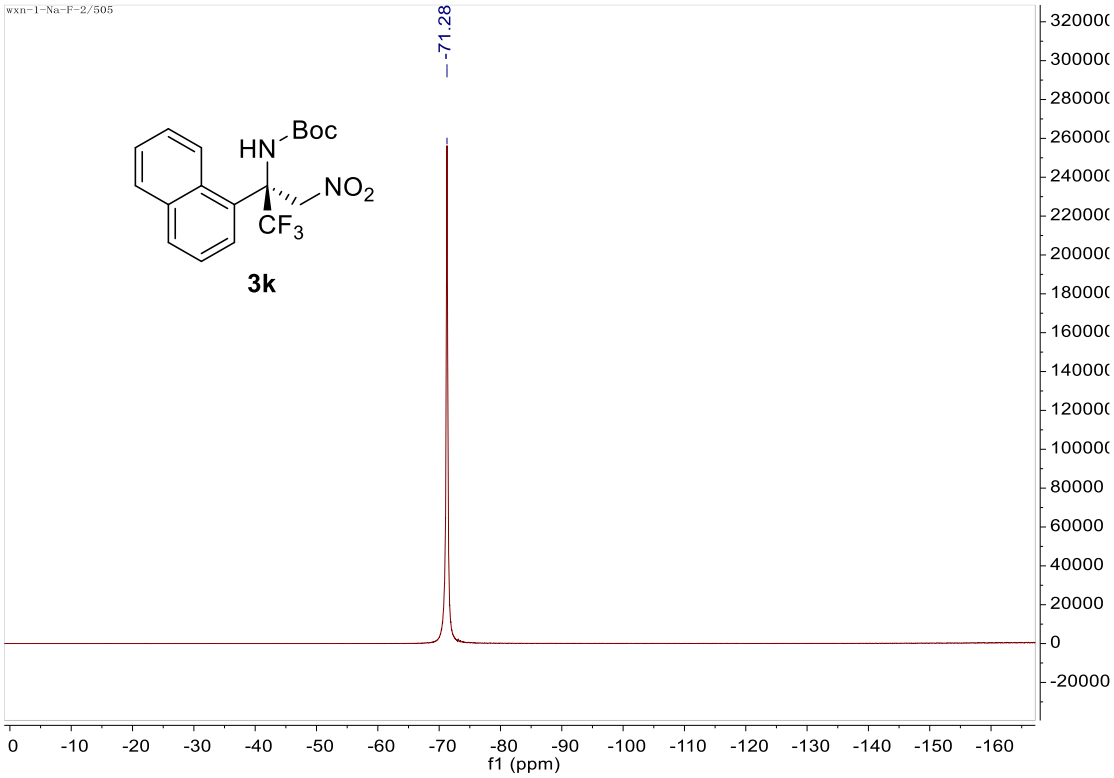
63.62
63.37
63.34
27.77
HN
Boc
NO2
CF3
3k
f1 (ppm)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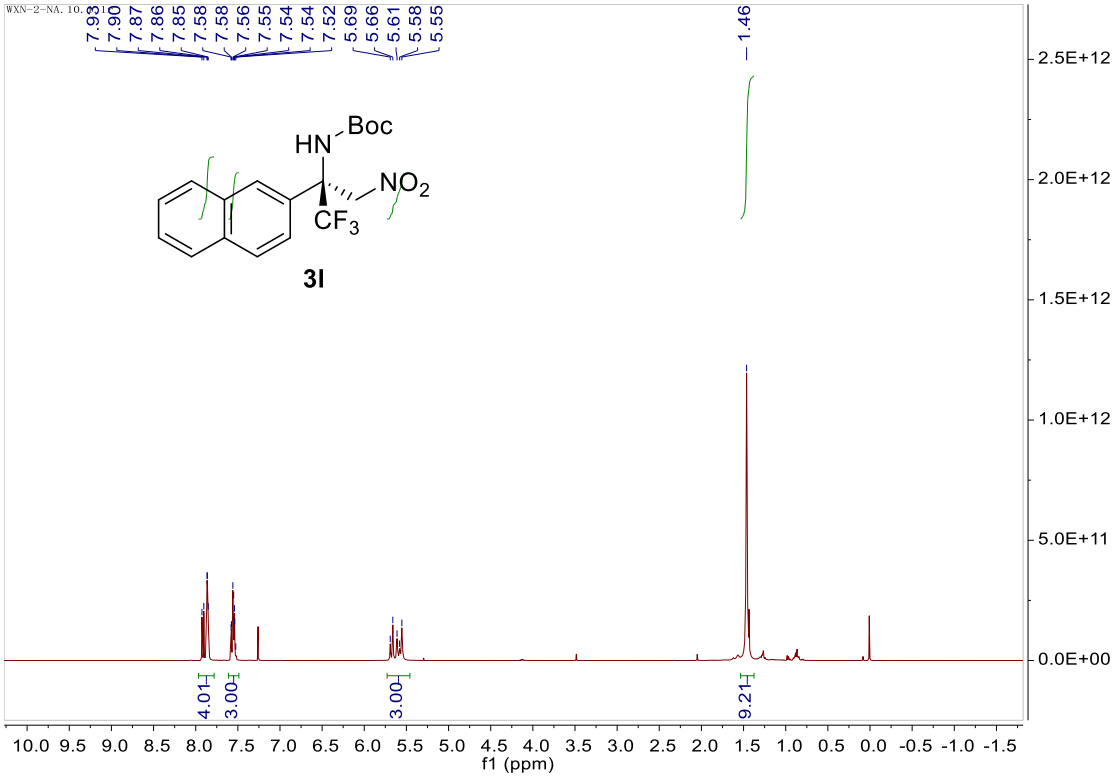
Boc
NO2
CF3
3k
f1 (ppm)


WXN-2-NA. 10.
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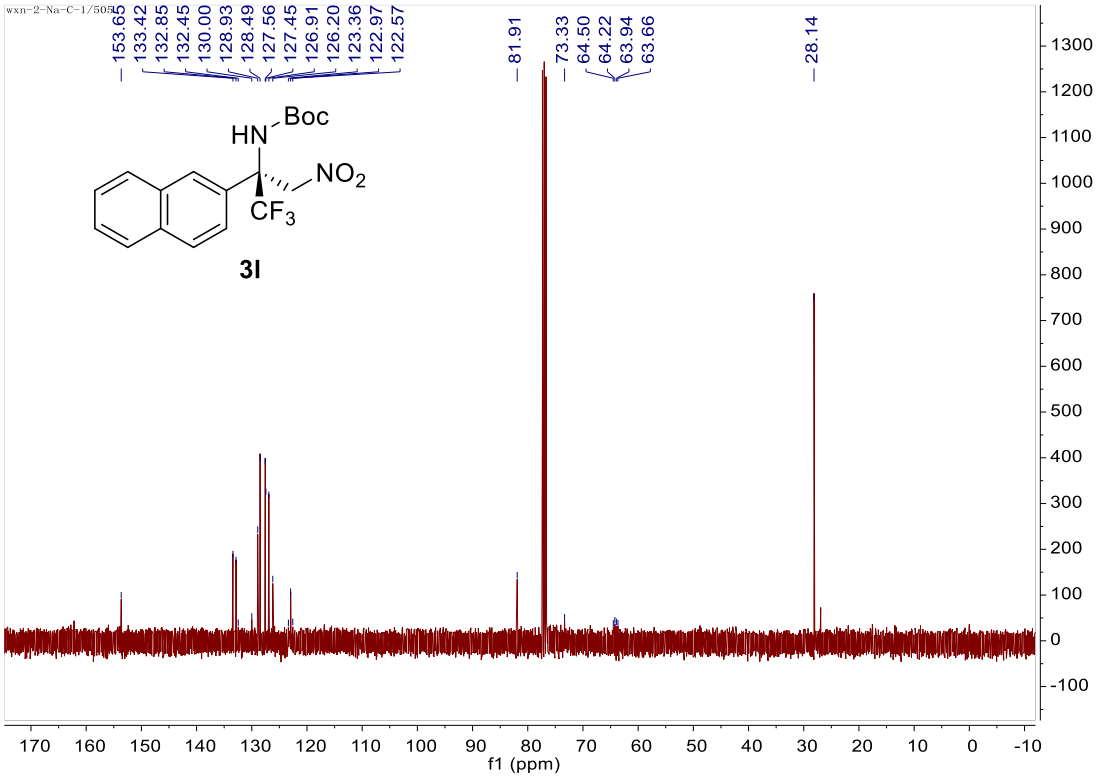
7.87
7.86
7.85
7.58
7.58
7.56
7.55
7.54
7.54
7.52
5.69
5.66
5.61
5.58
5.55
1.46
HN
Boc
NO2
CF3
3l
4.01
3.00
3.00
9.21
f1 (ppm)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128.49
127.56
127.45
126.91
126.20
123.36
122.97
122.57
81.91
73.33
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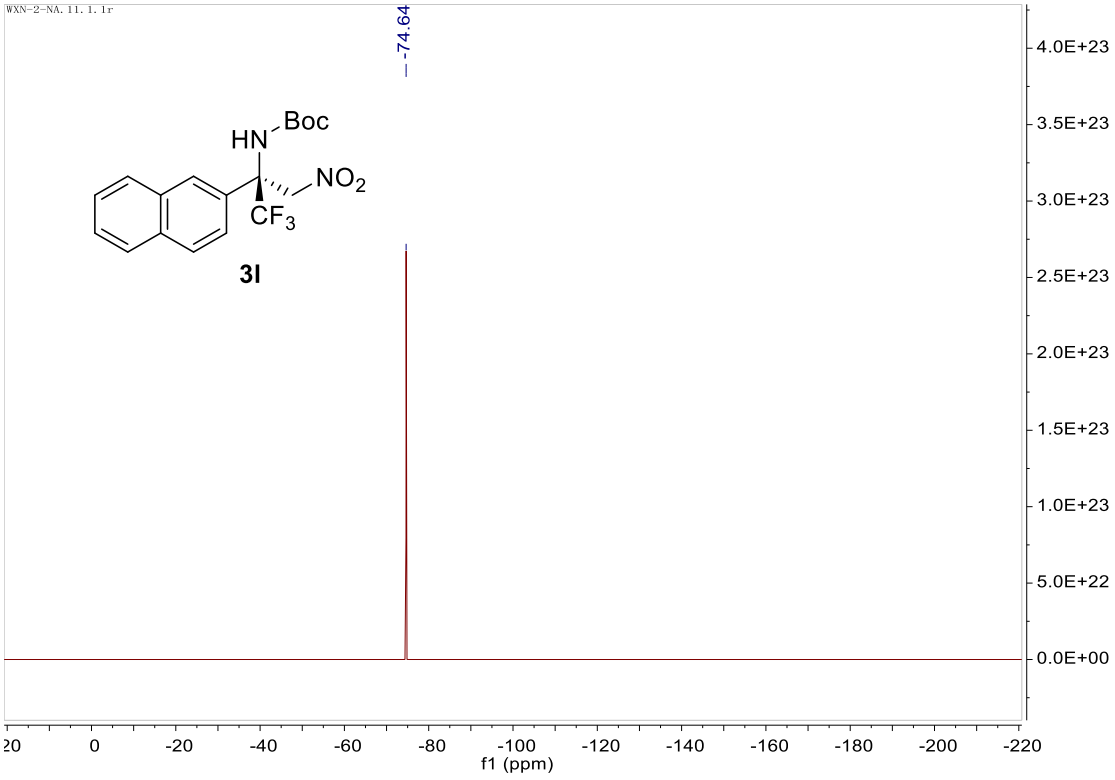
63.94
63.66
28.14
Boc
HN
NO2
CF3
3l
f1 (ppm)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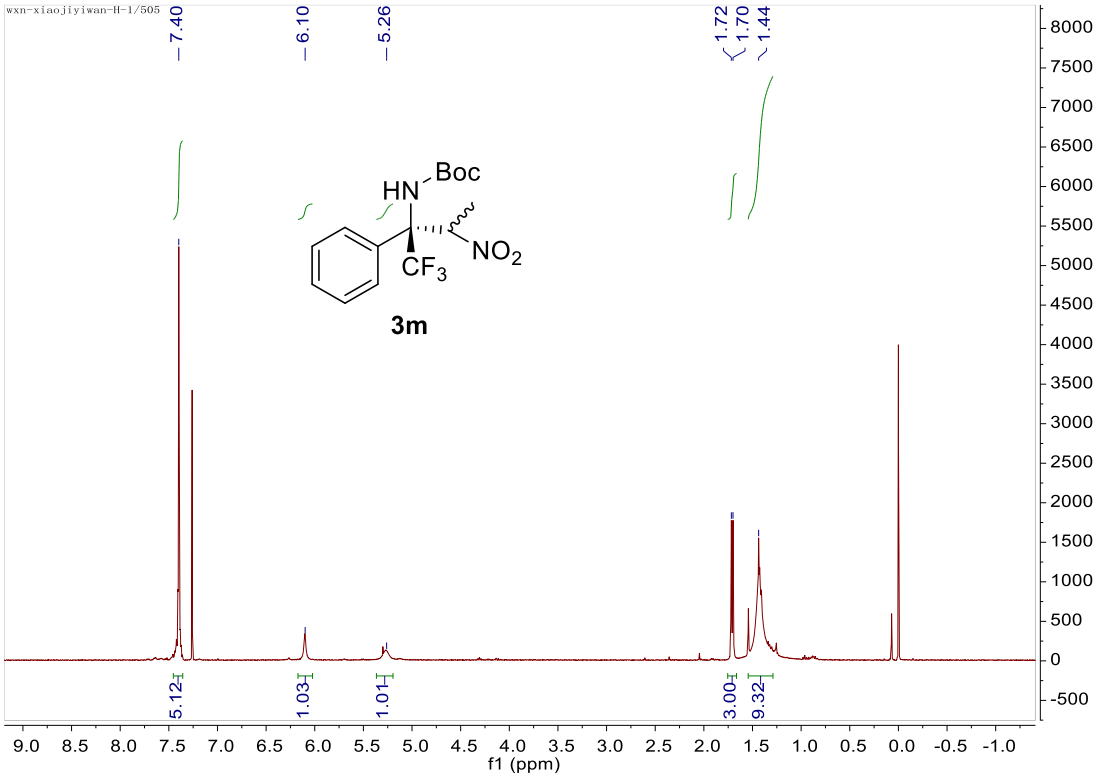
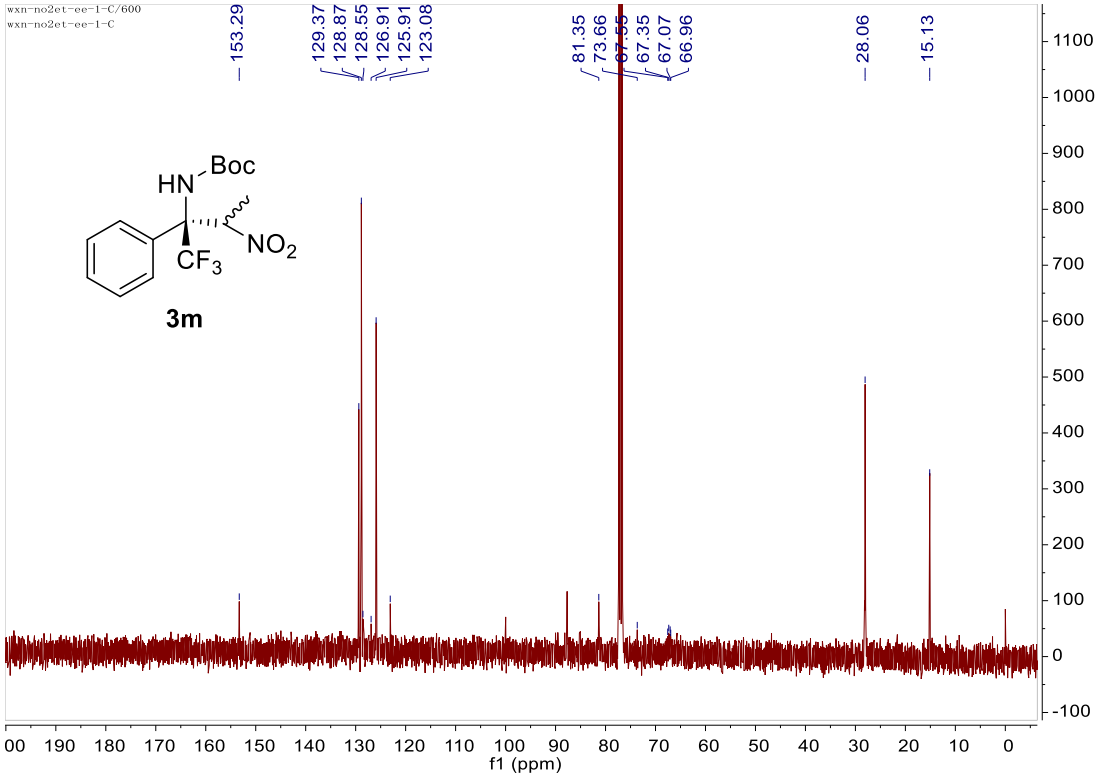

wxn-xiaojiyiwan-H-1/505
7.40
6.10
5.26
1.72
1.70
1.44
HN
Boc
NO2
CF3
3m
5.12
1.03
1.01
3.00
9.32
f1 (ppm)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wxn-no2et-ee-1-C
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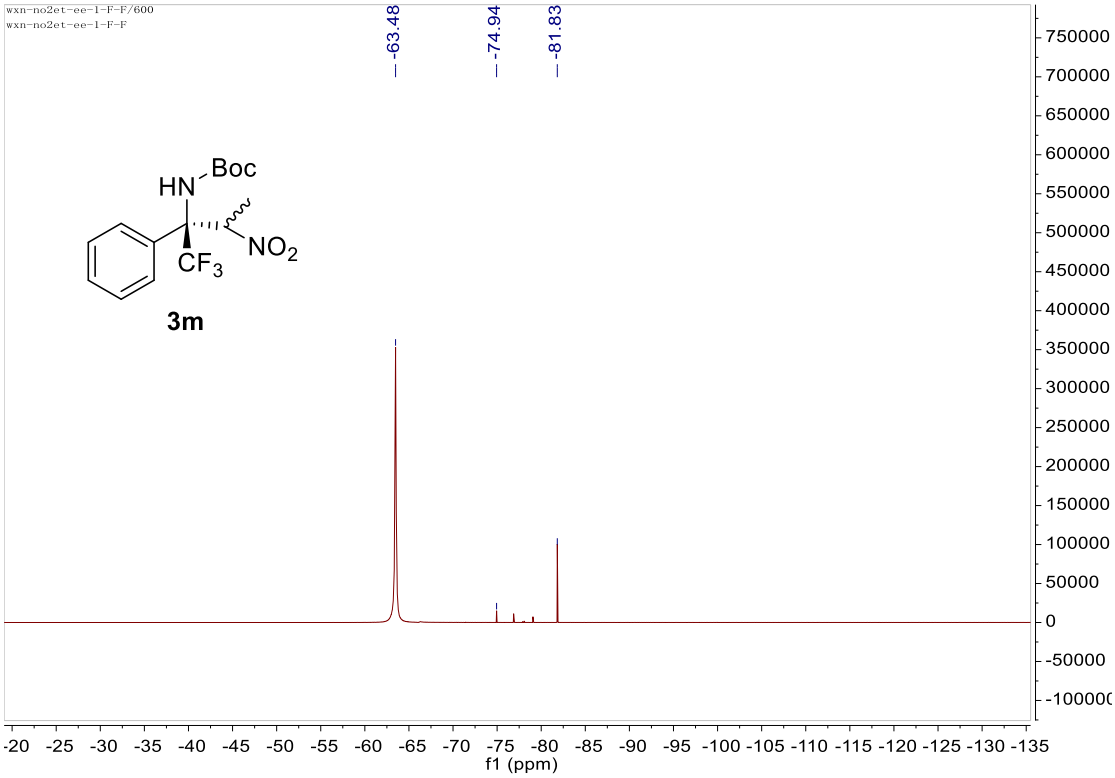
129.37
128.87
128.55
126.91
125.91
123.08
81.35
73.66
67.35
67.07
66.96
28.06
15.13
HN
Boc
NO2
CF3
3m
f1 (ppm)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wxn-no2et-ee-1-F-F
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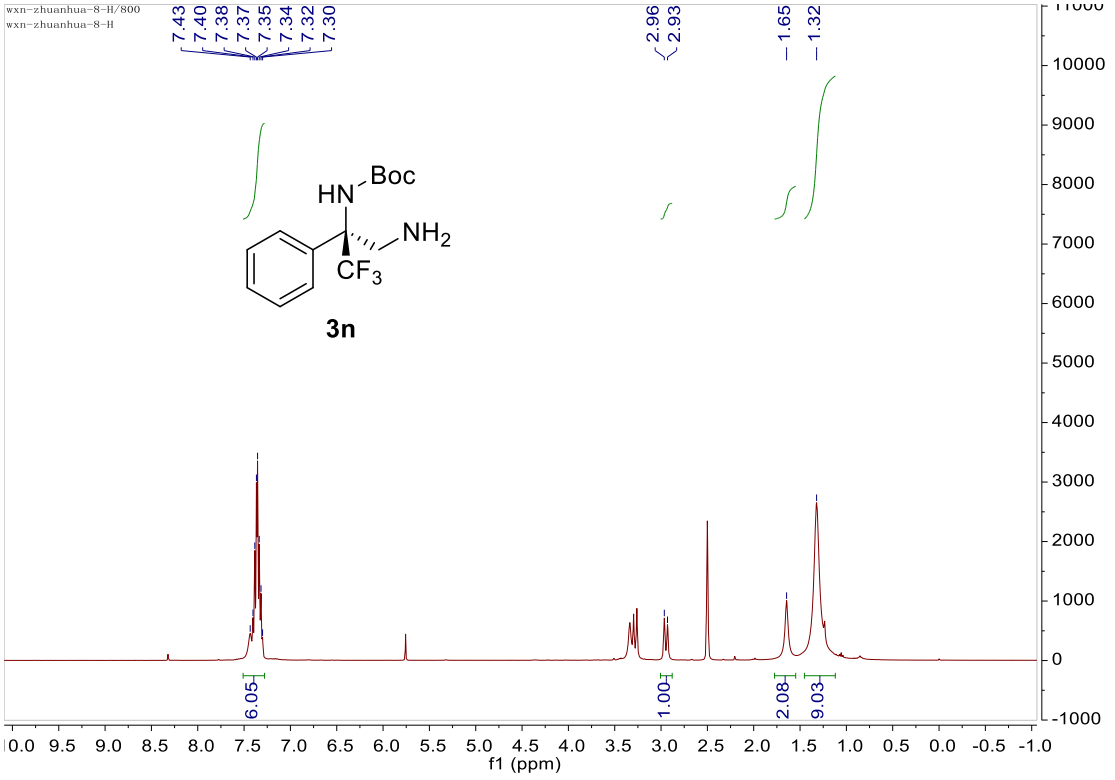
-81.83
HN
Boc
NO2
CF3
3m
f1 (ppm)


wxn-zhuanhua-8-H/800
wxn-zhuanhua-8-H
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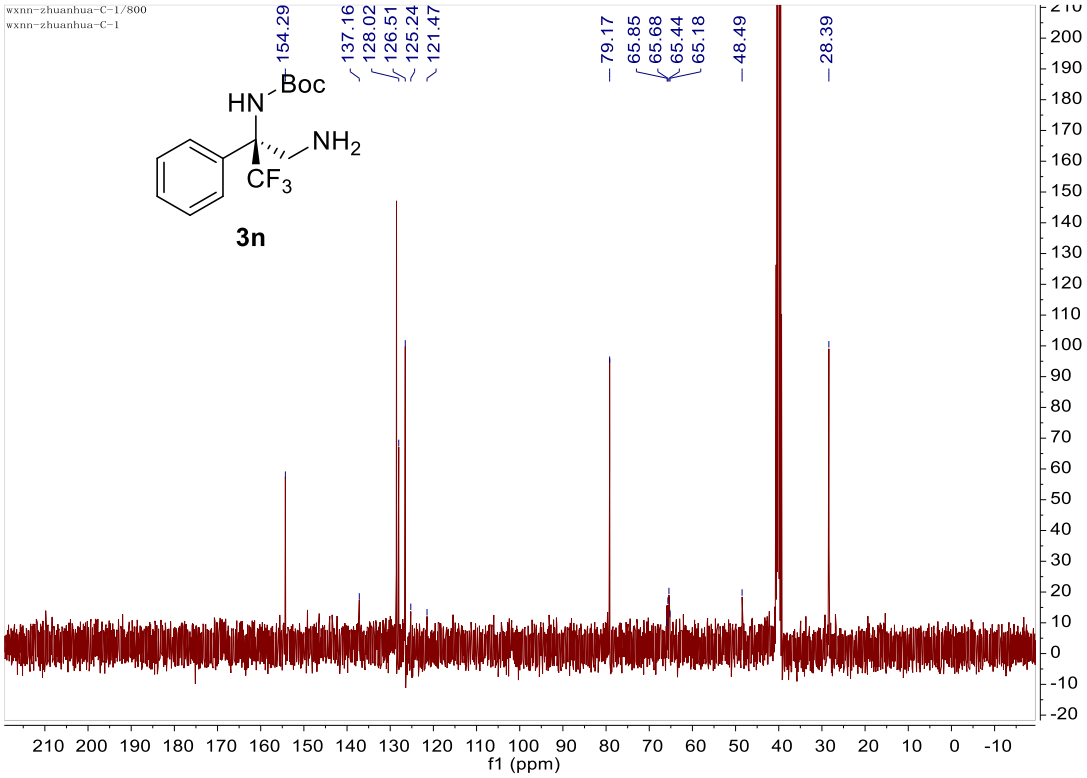
7.40
7.38
7.37
7.35
7.34
7.32
7.30
2.96
2.93
1.65
1.32
HN
Boc
NH2
CF3
3n
6.05
1.00
2.08
9.03
f1 (ppm)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wxnn-zhuanhua-C-1
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128.02
126.51
125.24
121.47
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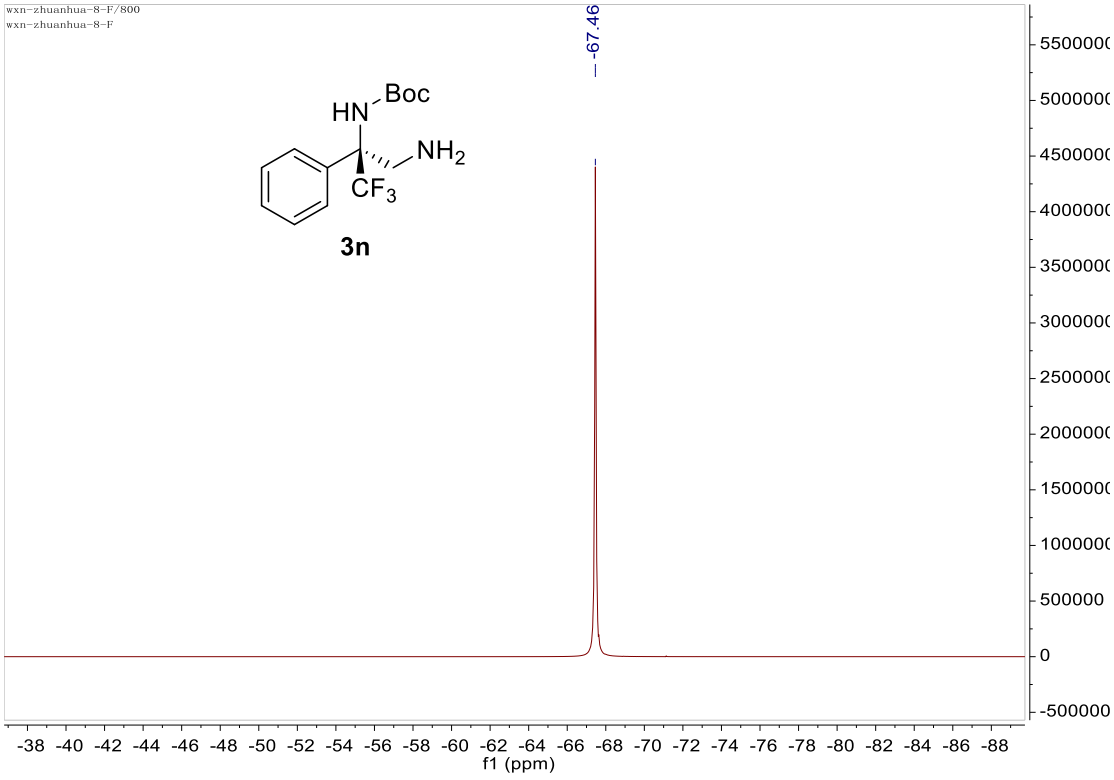
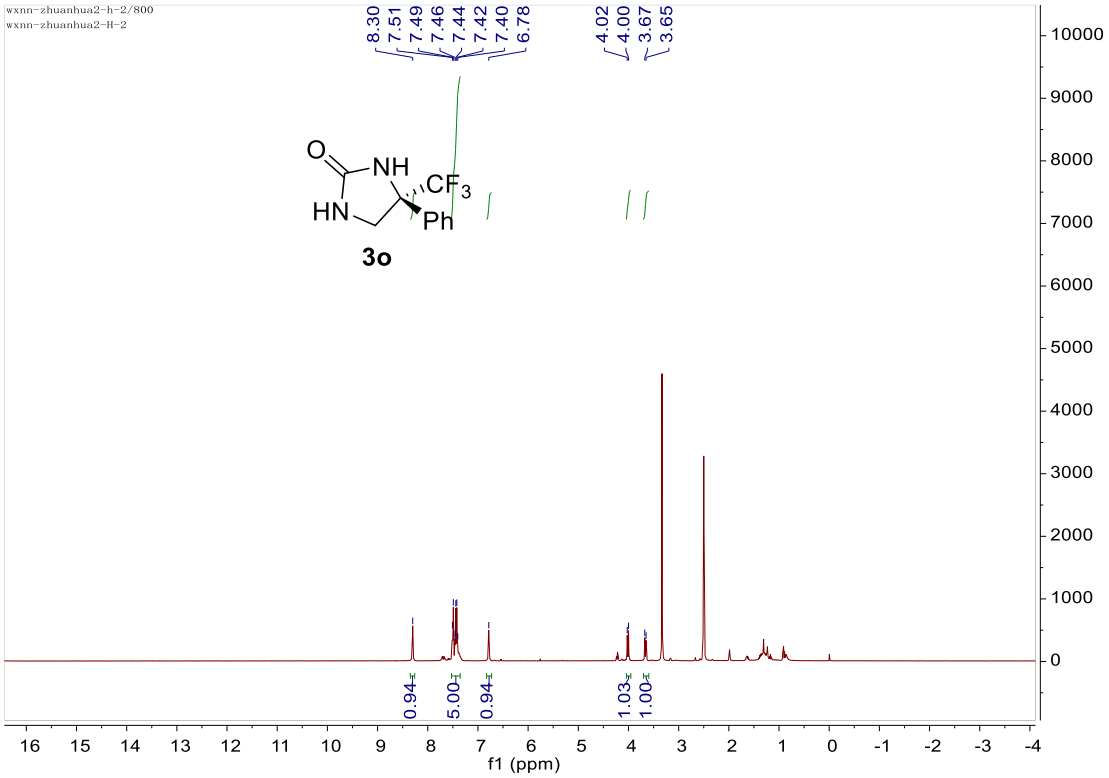
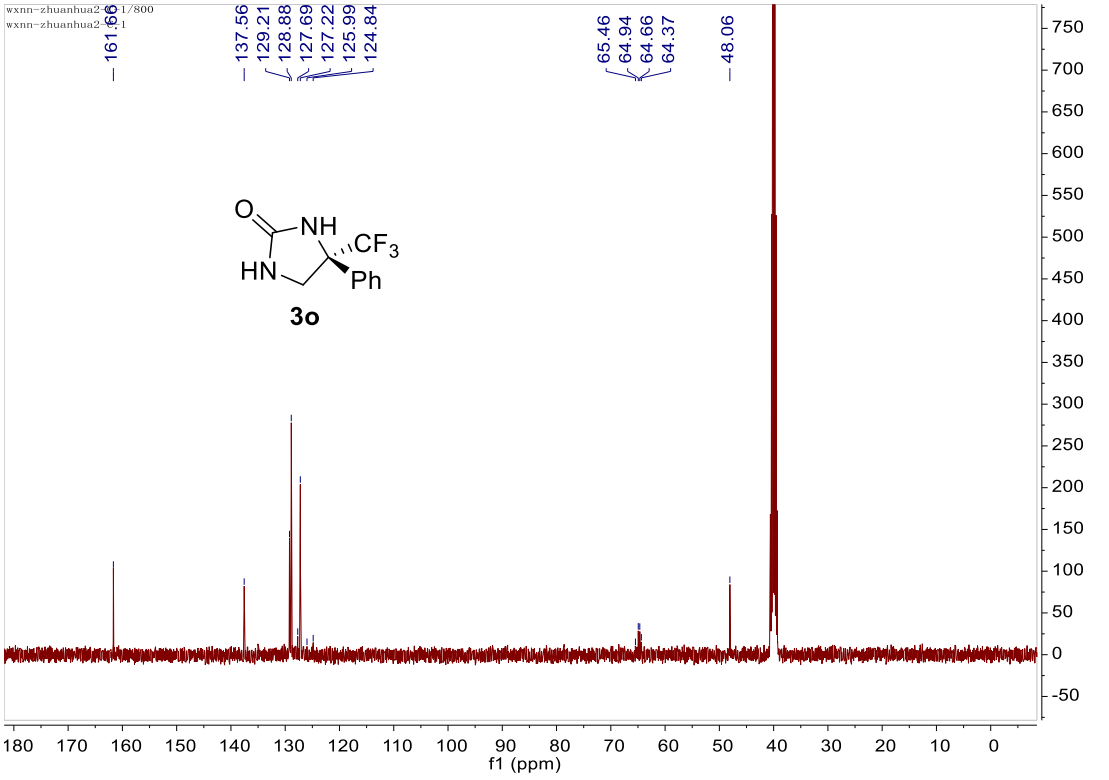
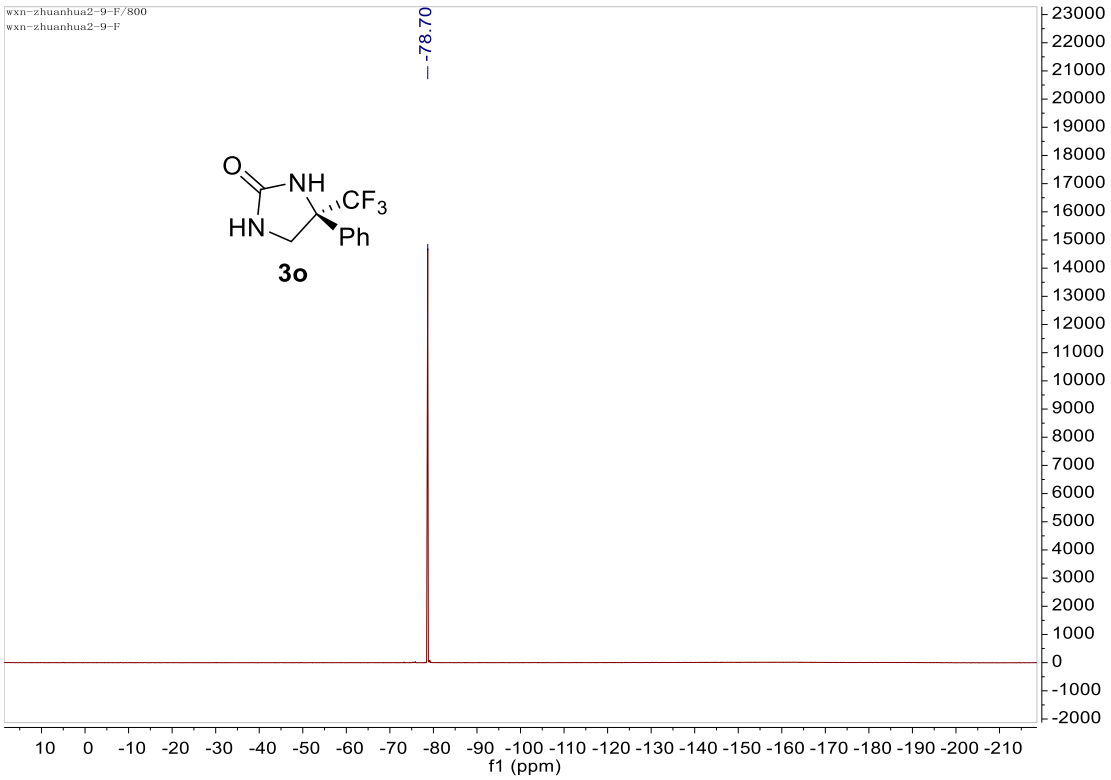
65.85
65.68
65.44
65.18
48.49
28.39
Boc
HN
NH2
CF3
3n
f1 (ppm)

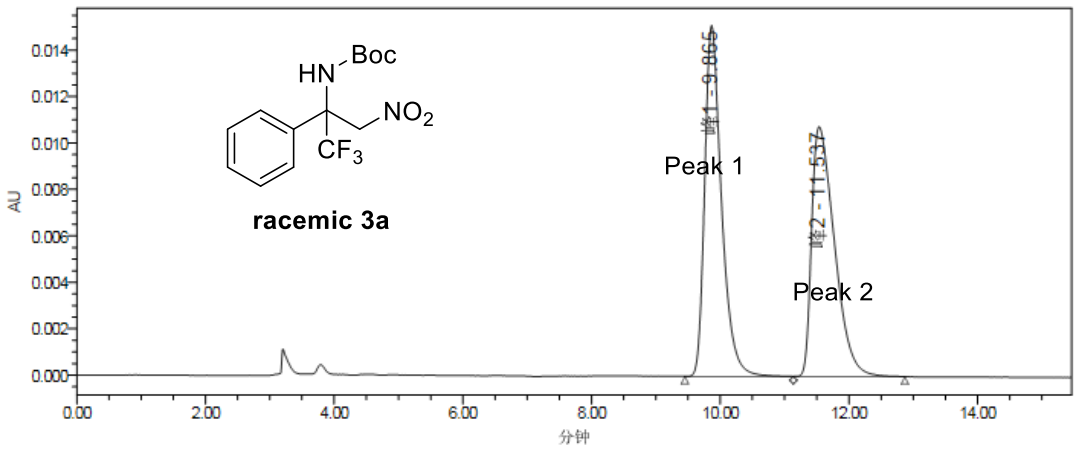

wxn-zhuanhua-8-F/800
wxn-zhuanhua-8-F
-67.46
Boc
HN
NH2
CF3
3n
f1 (ppm)


wxnn-zhuanhua2-h-2/800
wxnn-zhuanhua2-H-2
8.30
7.51
7.49
7.46
7.44
7.42
7.40
6.78
4.02
4.00
3.67
3.65
O
NH
CF3
HN
Ph
3o
0.94
5.00
0.94
1.03
1.00
f1 (ppm)


wxnn-zhuanhua2-C-1/800
wxnn-zhuanhua2-C-1
161.66
137.56
129.21
128.88
127.69
127.22
125.99
124.84
65.46
64.94
64.66
64.37
48.06
O
NH
CF3
HN
Ph
3o
f1 (ppm)

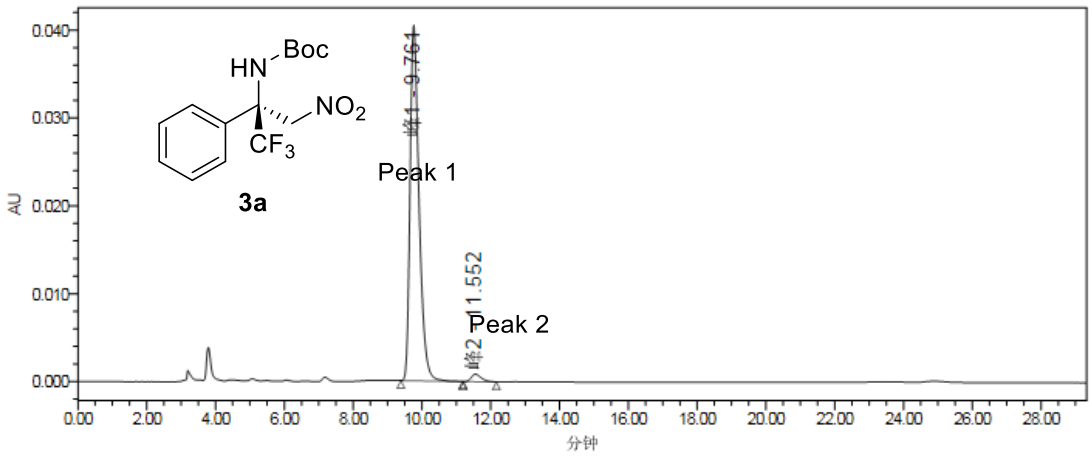

wxn-zhuanhua2-9-F/800
wxn-zhuanhua2-9-F
-78.70
O
NH
CF3
HN
Ph
3o
23000
22000
21000
20000
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-2000
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

7. HPLC traces of compounds 3a-3m and 3o



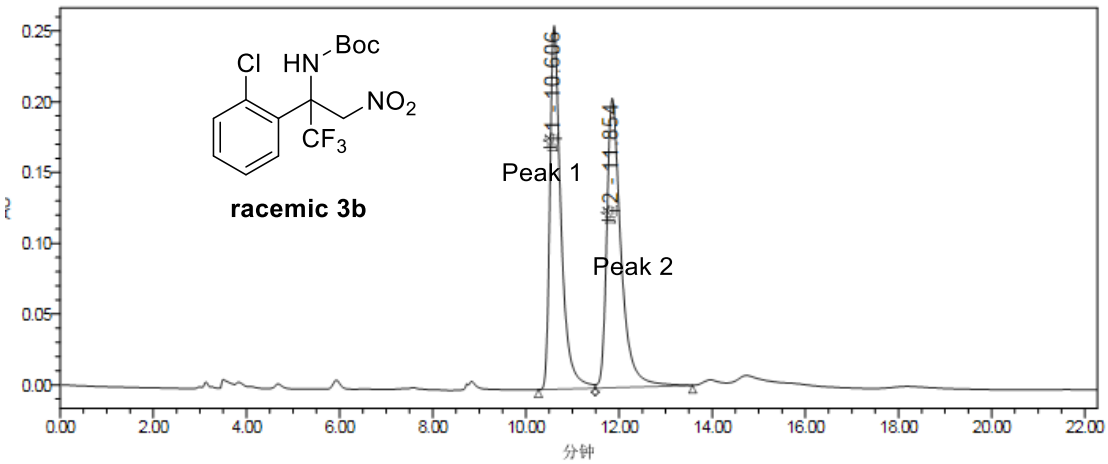
Channel: W2489 ChA; Channel Desc.: W2489 ChA 254nm; Processing Method: 0

	Channel Description	Peak Name	RT (min)	Area (碼*sec) μAU* sec	% Area	Height (碼) μAU
1	W2489 ChA 254nm	峰1 Peak 1	9.865	275028	50.90	15105
2	W2489 ChA 254nm	峰2 Peak 2	11.537	265343	49.10	10757



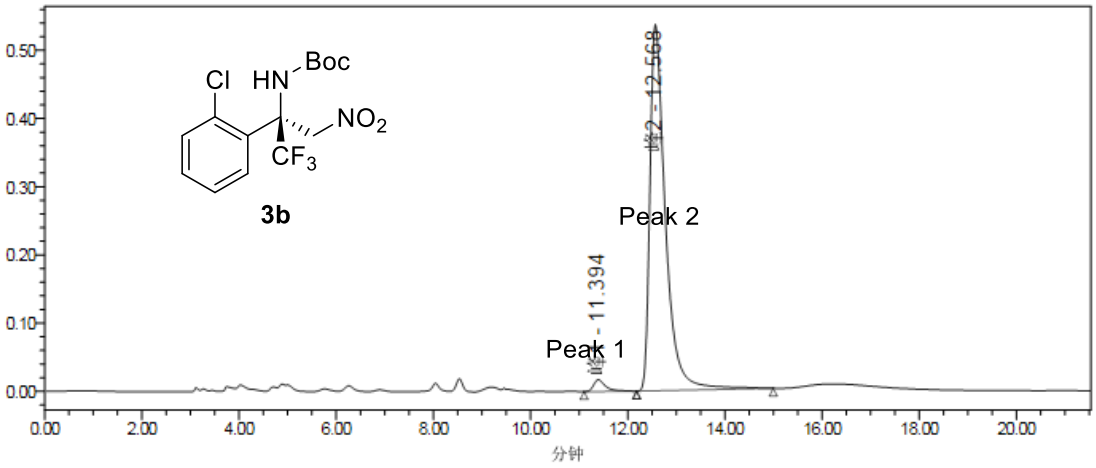
Channel: W2489 ChA; Channel Desc.: W2489 ChA 254nm; Processing Method: 0

	Channel Description	Peak Name	RT (min)	Area (碼*sec) μAU* sec	% Area	Height (碼) μAU
1	W2489 ChA 254nm	峰1 Peak 1	9.761	732912	97.67	40404
2	W2489 ChA 254nm	峰2 Peak 2	11.552	17498	2.33	860



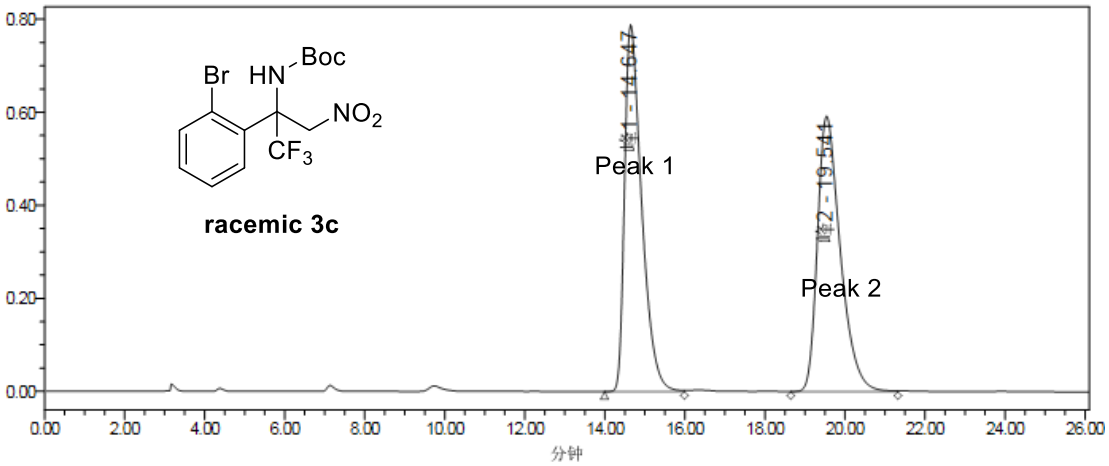
Channel: W2489 ChA; Channel Desc.: W2489 ChA 214nm; Processing Method: 0

	Channel Description	Peak Name	RT (min)	Area (峰*sec) μAU* sec	% Area	Height (峰) μAU
1	W2489 ChA 214nm	峰1 Peak 1	10.606	4231417	49.34	256481
2	W2489 ChA 214nm	峰2 Peak 2	11.854	4344489	50.66	204185



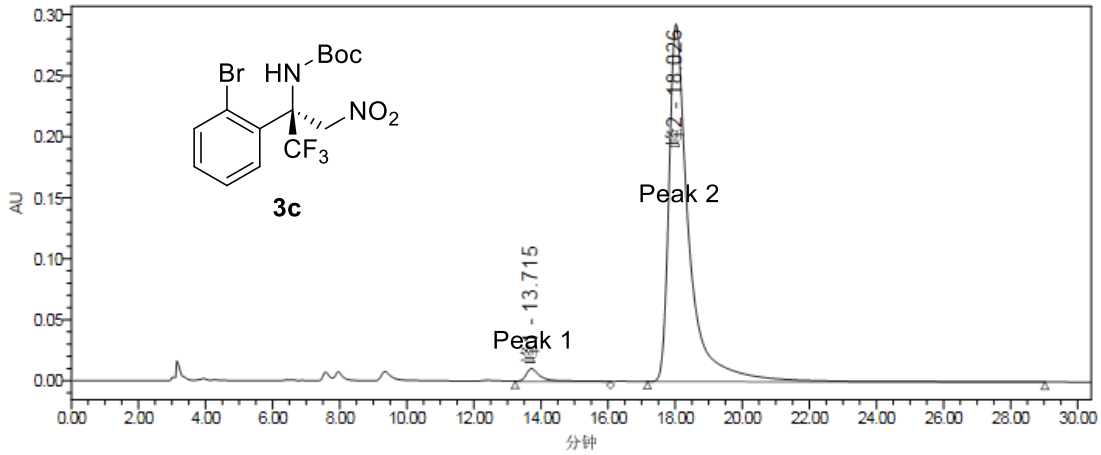
Channel: W2489 ChA; Channel Desc.: W2489 ChA 214nm; Processing Method: 0

	Channel Description	Peak Name	RT (min)	Area (峰*sec) μAU*sec	% Area	Height (峰) μAU
1	W2489 ChA 214nm	峰1 Peak 1	11.394	297452	2.44	17246
2	W2489 ChA 214nm	峰2 Peak 2	12.568	11887034	97.56	536796



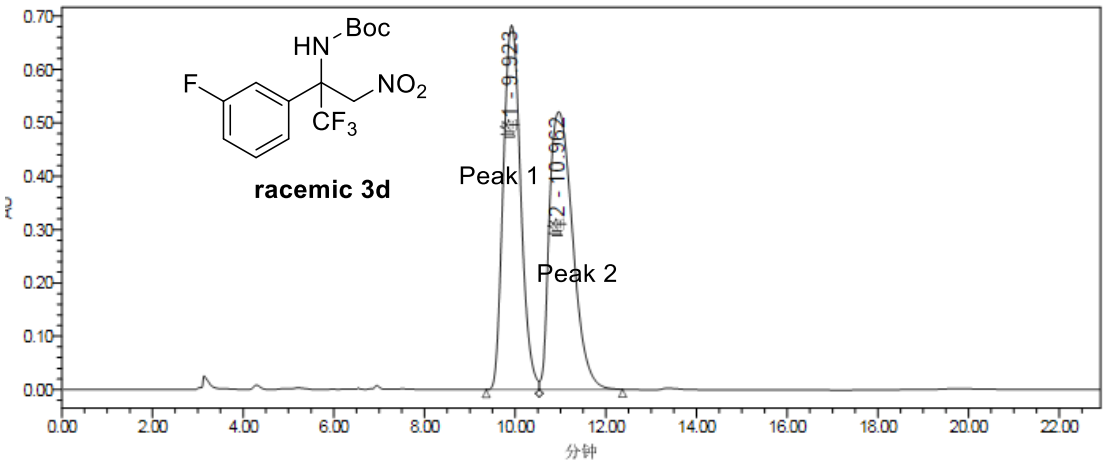
Channel: W2489 ChA; Channel Desc.: W2489 ChA 214nm; Processing Method: 0

	Channel Description	Peak Name	RT (min)	Area (μAU*sec)	% Area	Height μAU
1	W2489 ChA 214nm	Peak 1	14.647	23382864	49.82	787914
2	W2489 ChA 214nm	Peak 2	19.541	23552017	50.18	591905



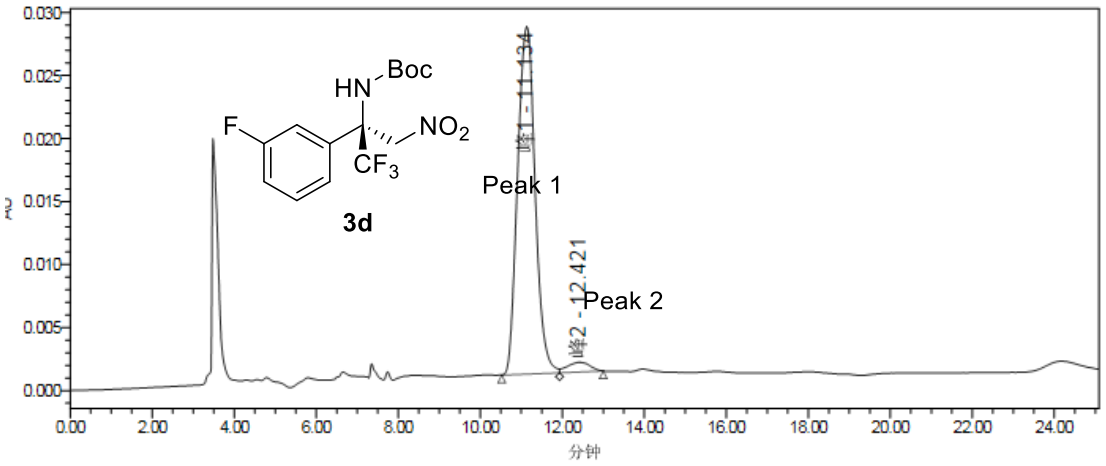
Channel: W2489 ChA; Channel Desc.: W2489 ChA 214nm; Processing Method: wxn

	Channel Description	Peak Name	RT (min)	Area μAU*sec	% Area	Height μAU
1	W2489 ChA 214nm	Peak 1	13.715	302105	2.45	10334
2	W2489 ChA 214nm	Peak 2	18.026	12043656	97.55	292843



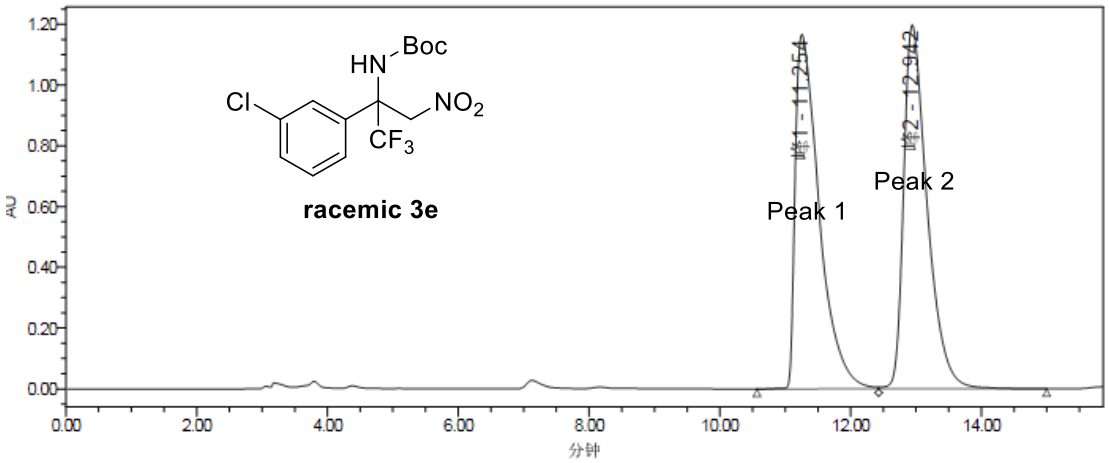
Channel: W2489 ChA; Channel Desc.: W2489 ChA 214nm; Processing Method: 0

	Channel Description	Peak Name	RT (min)	Area μAU*sec	% Area	Height μAU
1	W2489 ChA 214nm	Peak 1	9.923	18288466	49.70	682115
2	W2489 ChA 214nm	Peak 2	10.962	18508609	50.30	520314



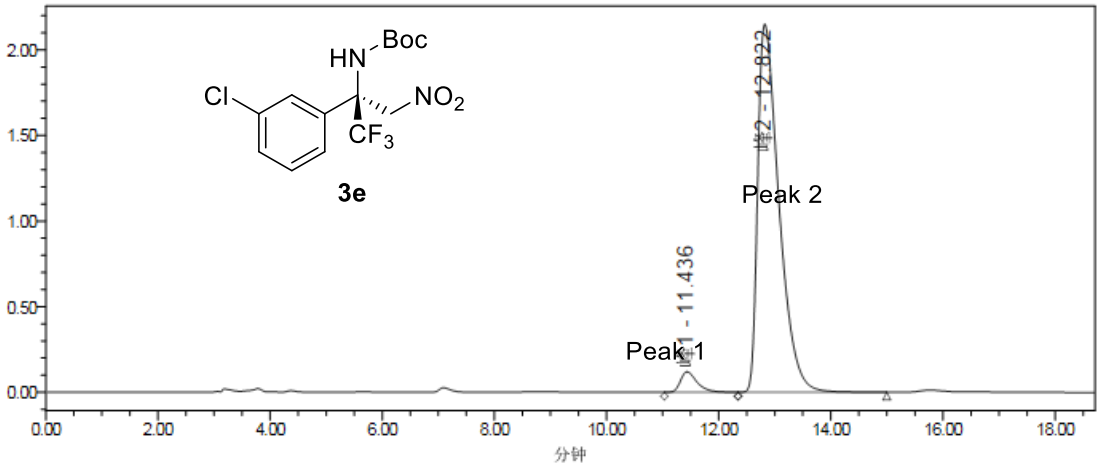
Channel: W2489 ChA; Channel Desc.: W2489 ChA 214nm; Processing Method: 0

	Channel Description	Peak Name	RT (min)	Area μAU*sec	% Area	Height μAU
1	W2489 ChA 214nm	Peak 1	11.134	817132	96.51	27567
2	W2489 ChA 214nm	Peak 2	12.421	29535	3.49	764



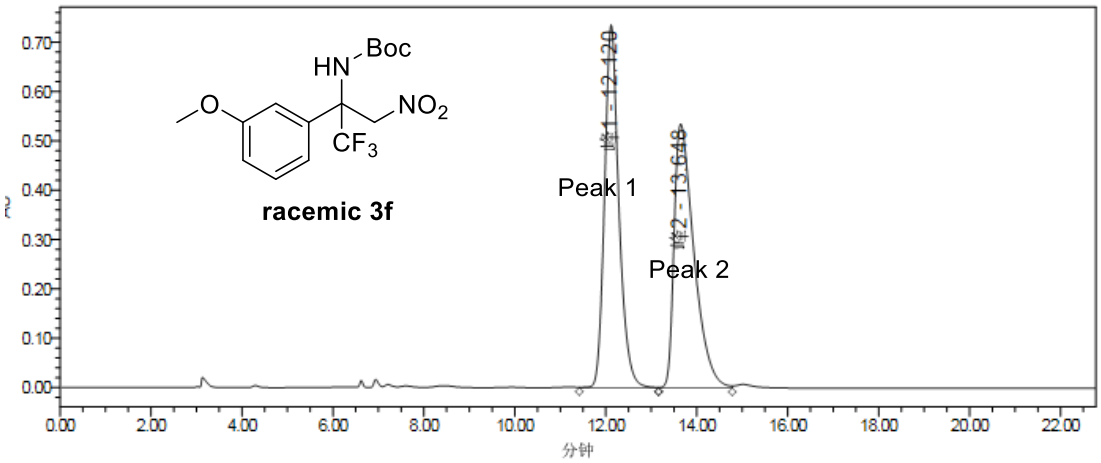
Channel: W2489 ChA; Channel Desc.: W2489 ChA 214nm; Processing Method: 0

	Channel Description	Peak Name	RT (min)	Area (μAU*sec)	% Area	Height (μAU)
1	W2489 ChA 214nm	峰1 Peak 1	11.254	29621772	49.77	1165698
2	W2489 ChA 214nm	峰2 Peak 2	12.942	29896051	50.23	1197123



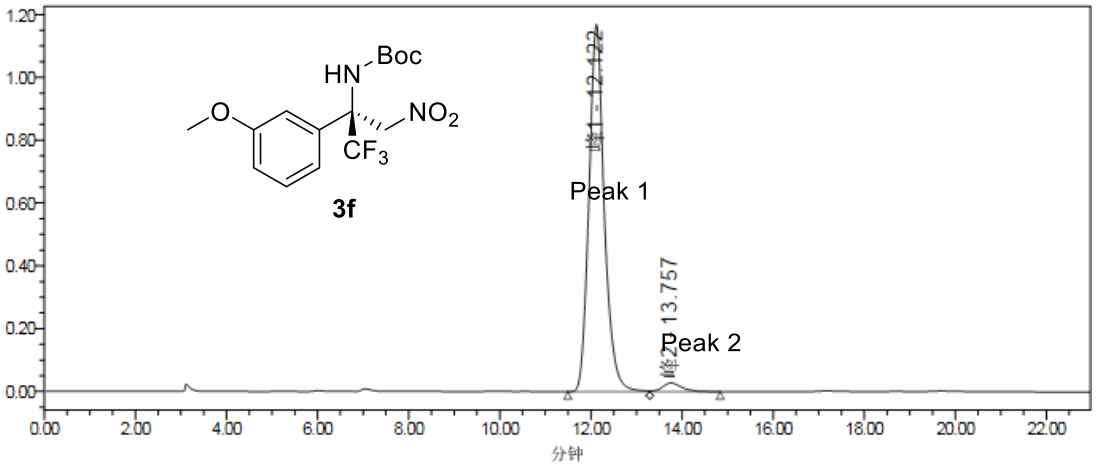
Channel: W2489 ChA; Channel Desc.: W2489 ChA 214nm; Processing Method: 0

	Channel Description	Peak Name	RT (min)	Area (μAU*sec)	% Area	Height (μAU)
1	W2489 ChA 214nm	峰1 Peak 1	11.436	2481974	4.21	119959
2	W2489 ChA 214nm	峰2 Peak 2	12.822	56465135	95.79	2148842



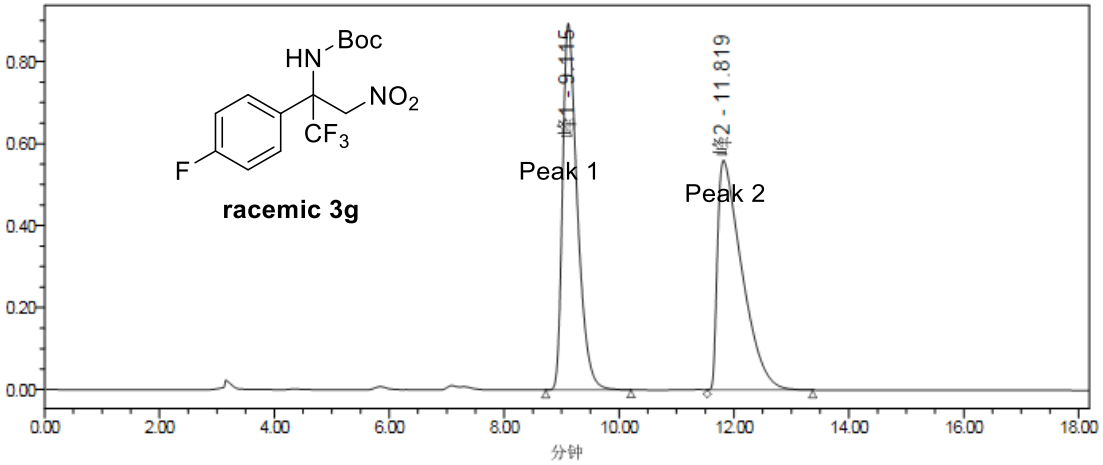
Channel: W2489 ChA; Channel Desc.: W2489 ChA 214nm; Processing Method: 0

	Channel Description	Peak Name	RT (min)	Area μAU*sec	% Area	Height μAU
1	W2489 ChA 214nm	峰1 Peak 1	12.120	16033800	49.77	734749
2	W2489 ChA 214nm	峰2 Peak 2	13.648	16182828	50.23	534472



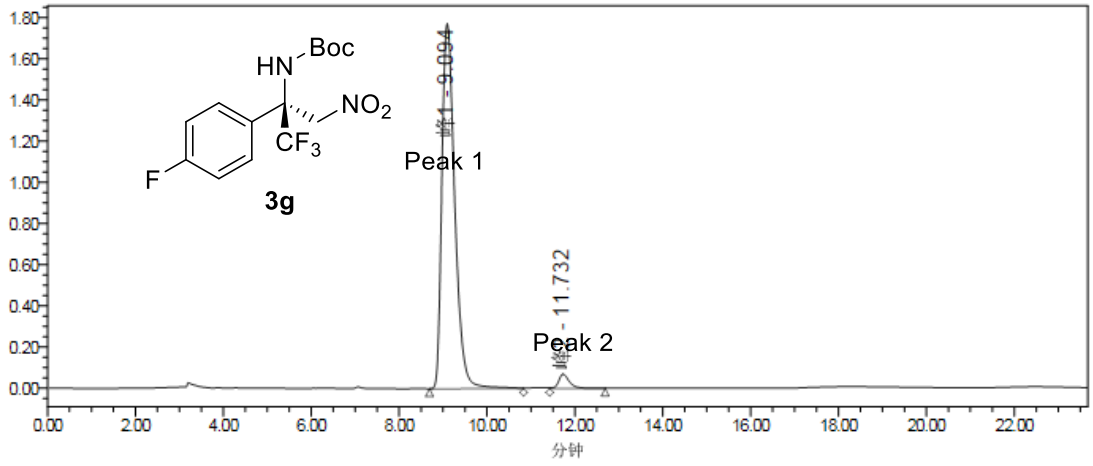
Channel: W2489 ChA; Channel Desc.: W2489 ChA 214nm; Processing Method: 0

	Channel Description	Peak Name	RT (min)	Area μAU*sec	% Area	Height μAU
1	W2489 ChA 214nm	峰1 Peak 1	12.122	27270142	97.11	1168057
2	W2489 ChA 214nm	峰2 Peak 2	13.757	812111	2.89	27788



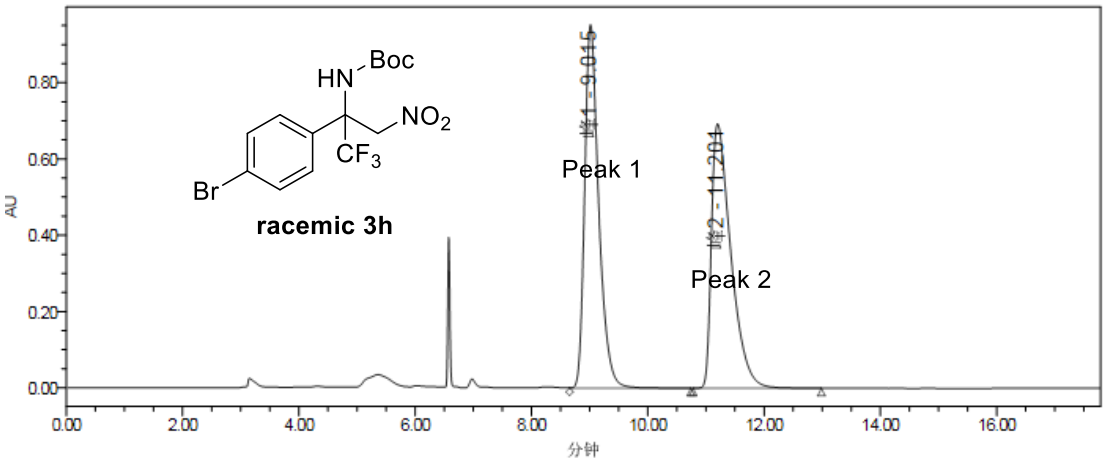
Channel: W2489 ChA; Channel Desc.: W2489 ChA 214nm; Processing Method: 0

	Channel Description	Peak Name	RT (min)	Area μAU*sec	% Area	Height μAU
1	W2489 ChA 214nm	Peak 1	9.115	15943975	49.15	893568
2	W2489 ChA 214nm	Peak 2	11.819	16496193	50.85	559624



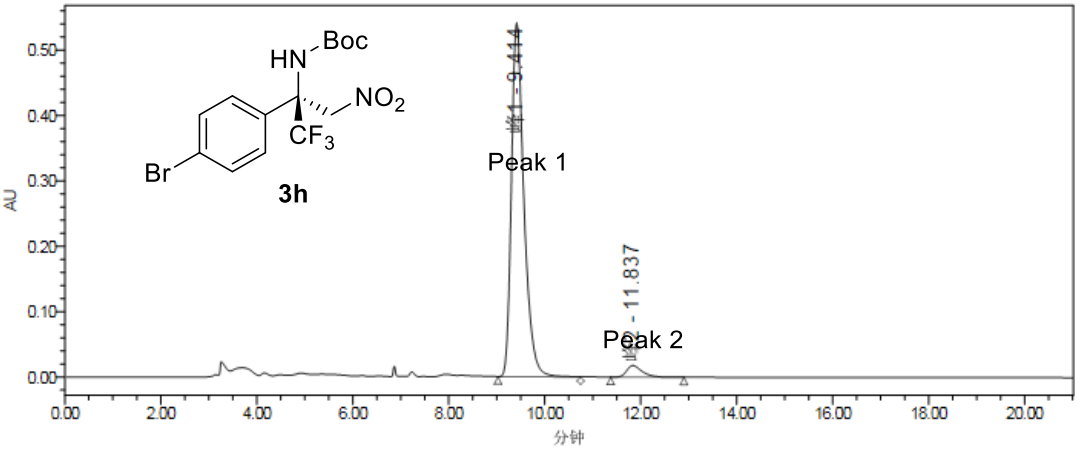
Channel: W2489 ChA; Channel Desc.: W2489 ChA 214nm; Processing Method: 0

	Channel Description	Peak Name	RT (min)	Area μAU*sec	% Area	Height μAU
1	W2489 ChA 214nm	Peak 1	9.094	36072437	96.85	1770972
2	W2489 ChA 214nm	Peak 2	11.732	1171988	3.15	69684



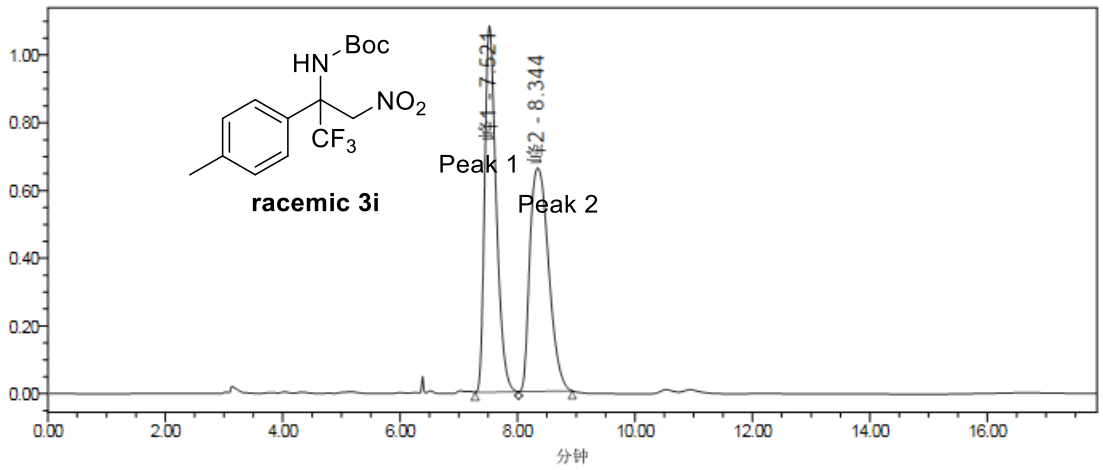
Channel: W2489 ChA; Channel Desc.: W2489 ChA 214nm; Processing Method: 0

	Channel Description	Peak Name	RT (min)	Area μAU*sec	% Area	Height μAU
1	W2489 ChA 214nm	Peak 1	9.015	15949090	49.89	951824
2	W2489 ChA 214nm	Peak 2	11.201	16018367	50.11	692874



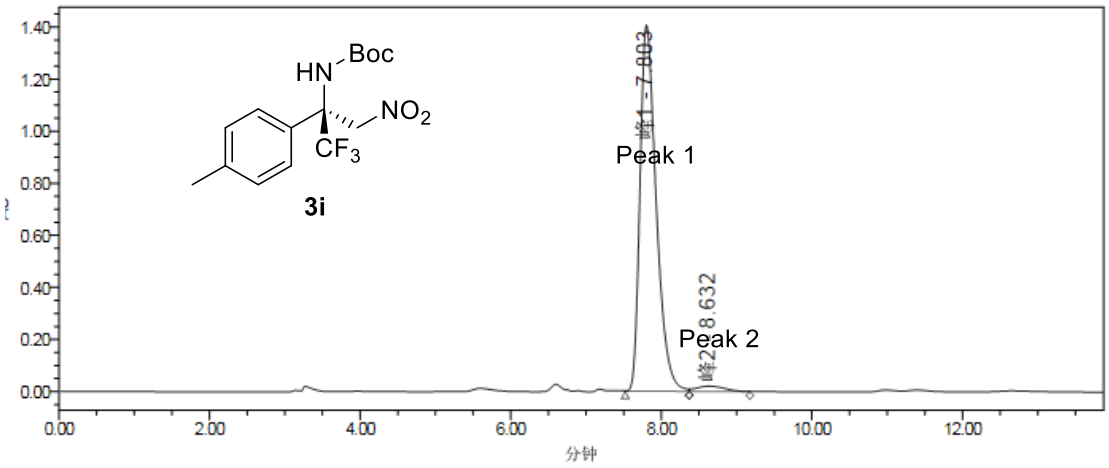
Channel: W2489 ChA; Channel Desc.: W2489 ChA 214nm; Processing Method: 0

	Channel Description	Peak Name	RT (min)	Area μAU*sec	% Area	Height μAU
1	W2489 ChA 214nm	Peak 1	9.414	9777303	96.09	540666
2	W2489 ChA 214nm	Peak 2	11.837	397975	3.91	17794



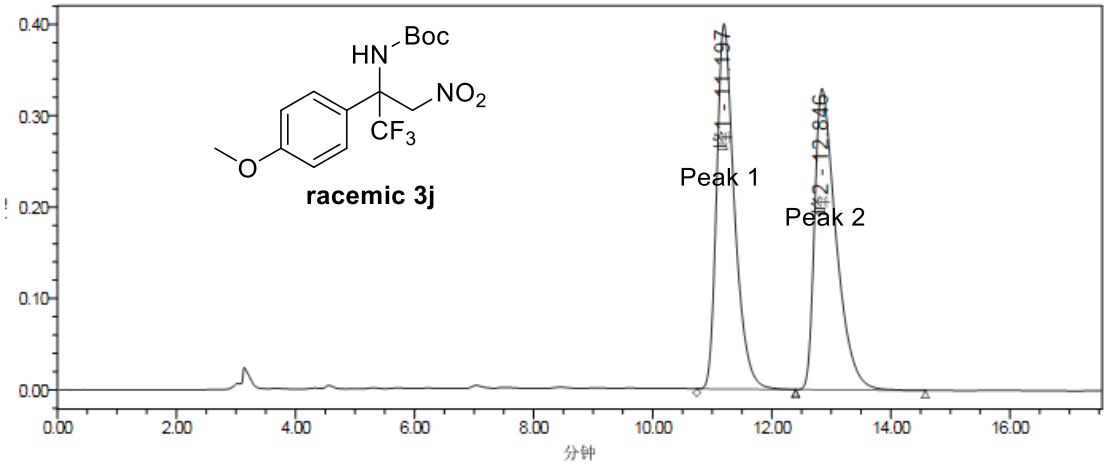
Channel: W2489 ChA; Channel Desc.: W2489 ChA 214nm; Processing Method: 0

	Channel Description	Peak Name	RT (min)	Area μAU*sec	% Area	Height μAU
1	W2489 ChA 214nm	峰1 Peak 1	7.521	14756542	50.19	1080642
2	W2489 ChA 214nm	峰2 Peak 2	8.344	14644187	49.81	659043



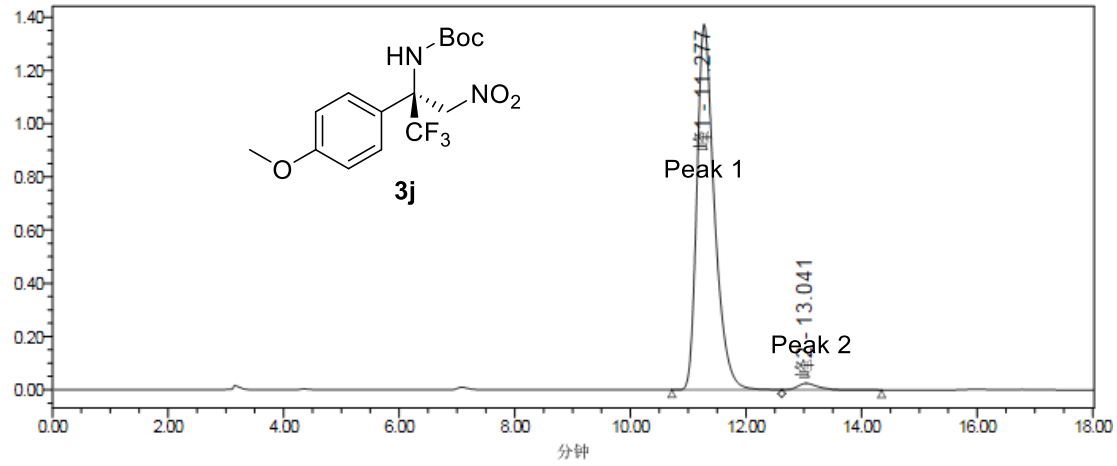
Channel: W2489 ChA; Channel Desc.: W2489 ChA 214nm; Processing Method: 0

	Channel Description	Peak Name	RT (min)	Area μAU*sec	% Area	Height μAU
1	W2489 ChA 214nm	峰1 Peak 1	7.803	20975970	97.54	1403591
2	W2489 ChA 214nm	峰2 Peak 2	8.632	528924	2.46	20663



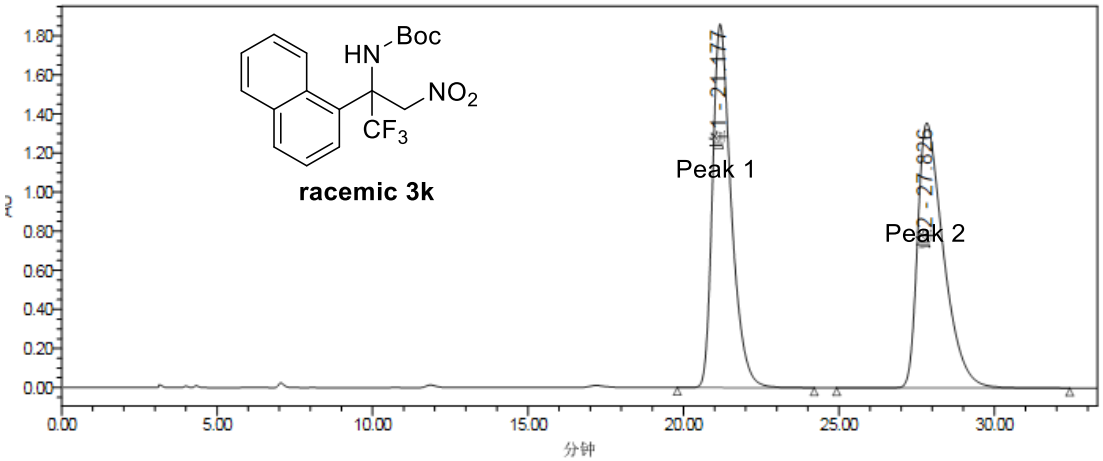
Channel: W2489 ChA; Channel Desc.: W2489 ChA 214nm; Processing Method: 0

	Channel Description	Peak Name	RT (min)	Area μAU*sec	% Area	Height μAU
1	W2489 ChA 214nm	峰1 Peak 1	11.197	8291232	50.08	399502
2	W2489 ChA 214nm	峰2 Peak 2	12.846	8263346	49.92	329242



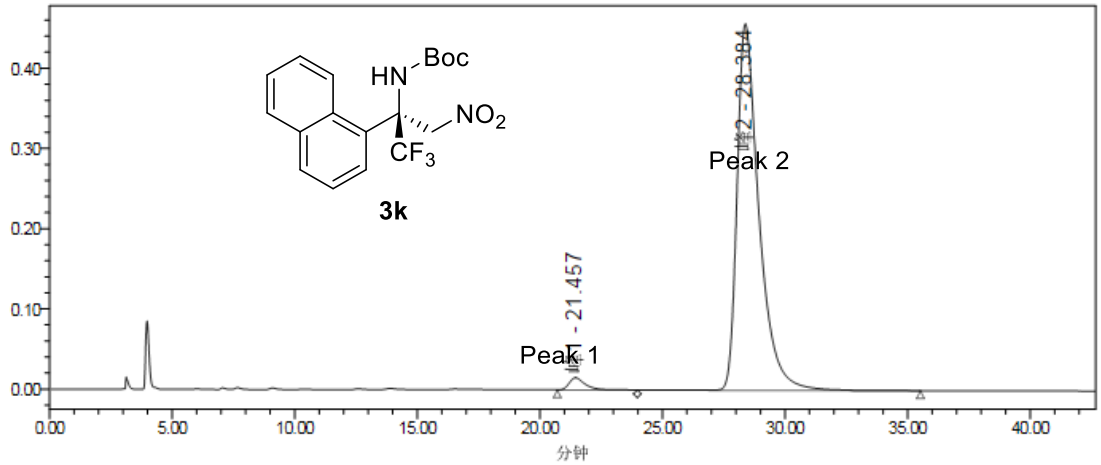
Channel: W2489 ChA; Channel Desc.: W2489 ChA 214nm; Processing Method: 0

	Channel Description	Peak Name	RT (min)	Area μAU*sec	% Area	Height μAU
1	W2489 ChA 214nm	峰1 Peak 1	11.277	28906989	97.97	1373016
2	W2489 ChA 214nm	峰2 Peak 2	13.041	599464	2.03	24352



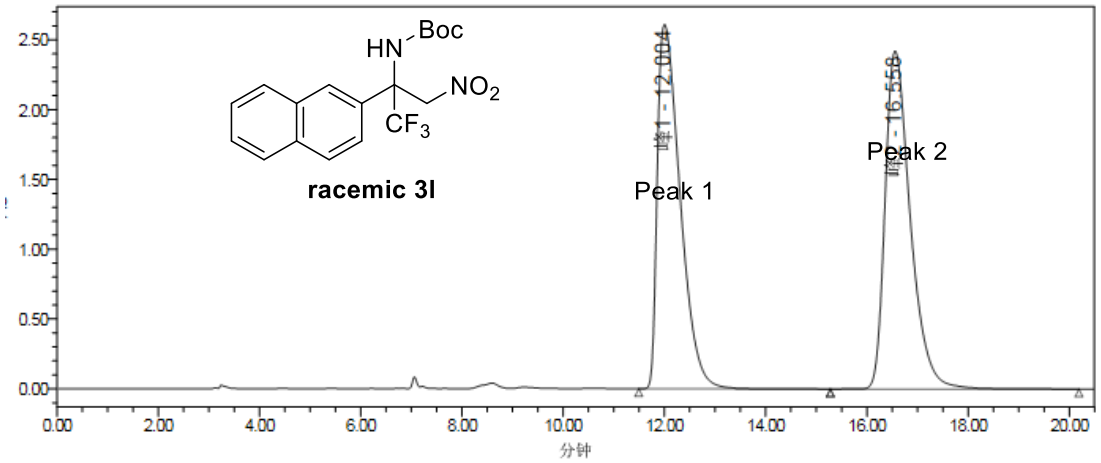
Channel: W2489 ChA; Channel Desc.: W2489 ChA 214nm; Processing Method: 0

	Channel Description	Peak Name	RT (min)	Area μAU* sec	% Area	Height μAU
1	W2489 ChA 214nm	峰1 Peak 1	21.177	77627010	49.73	1858211
2	W2489 ChA 214nm	峰2 Peak 2	27.826	78475011	50.27	1354802



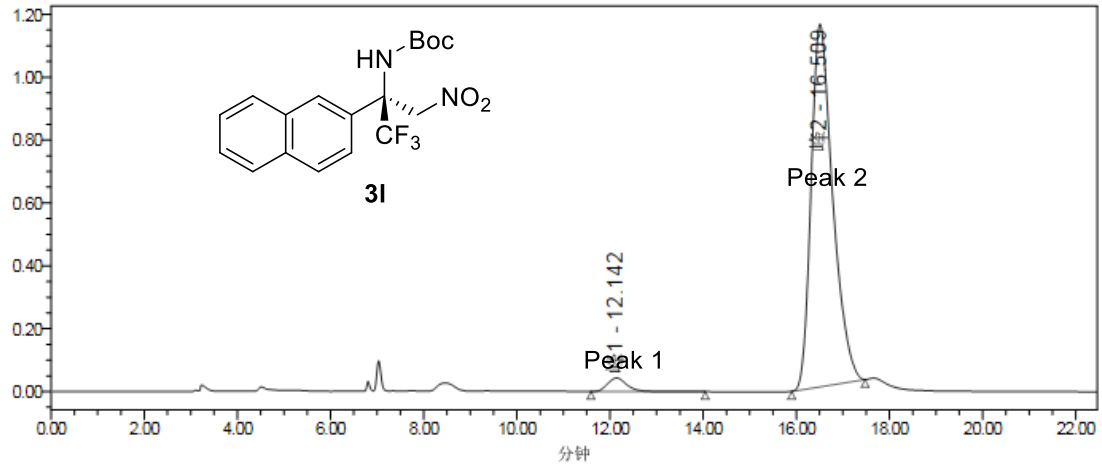
Channel: W2489 ChA; Channel Desc.: W2489 ChA 214nm; Processing Method: 0

	Channel Description	Peak Name	RT (min)	Area μAU* sec	% Area	Height μAU
1	W2489 ChA 214nm	峰1 Peak 1	21.457	694822	2.39	15206
2	W2489 ChA 214nm	峰2 Peak 2	28.384	28341510	97.61	456438



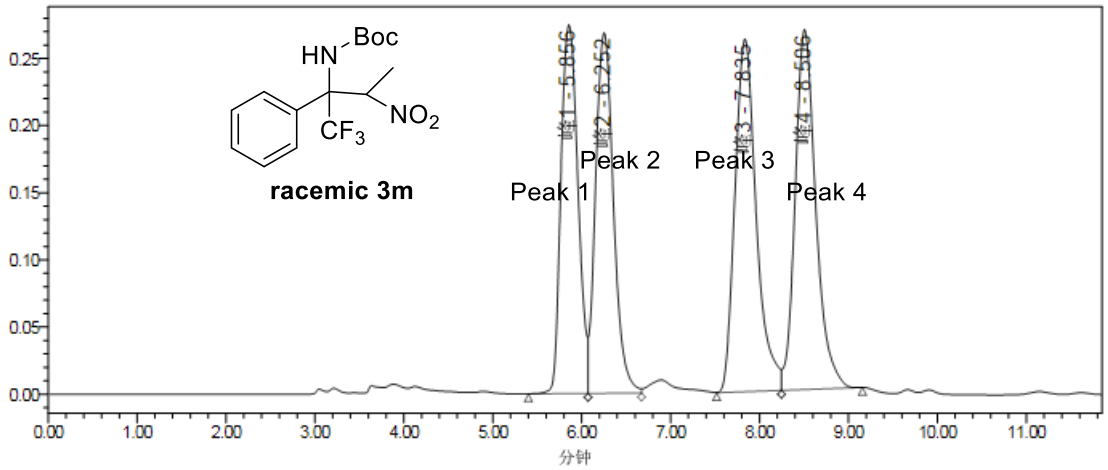
Channel: W2489 ChA; Channel Desc.: W2489 ChA 214nm; Processing Method: 0

	Channel Description	Peak Name	RT (min)	Area μAU*sec	% Area	Height μAU
1	W2489 ChA 214nm	峰1 Peak 1	12.004	81343793	49.36	2608047
2	W2489 ChA 214nm	峰2 Peak 2	16.558	83466500	50.64	2419335



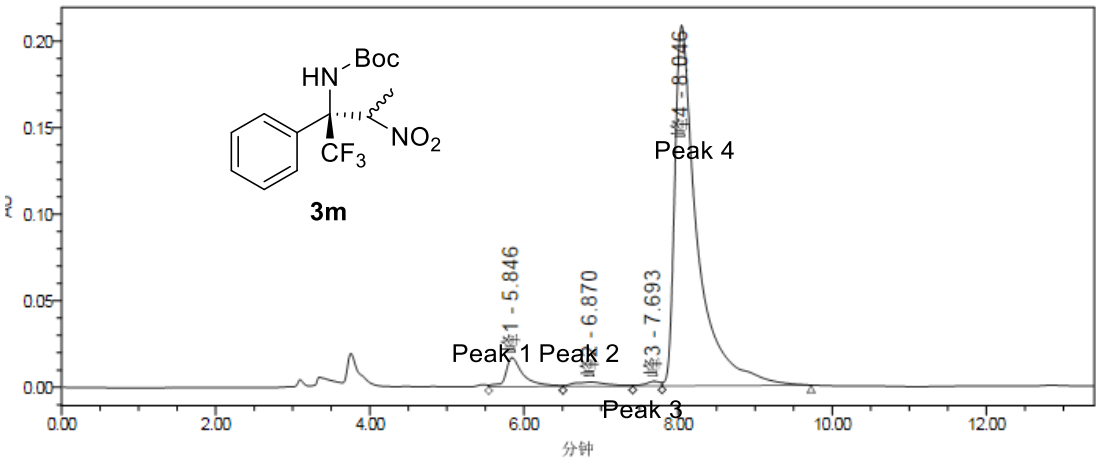
Channel: W2489 ChA; Channel Desc.: W2489 ChA 214nm; Processing Method: 0

	Channel Description	Peak Name	RT (min)	Area μAU*sec	% Area	Height μAU
1	W2489 ChA 214nm	峰1 Peak 1	12.142	1230087	3.20	43041
2	W2489 ChA 214nm	峰2 Peak 2	16.509	37197717	96.80	1153096



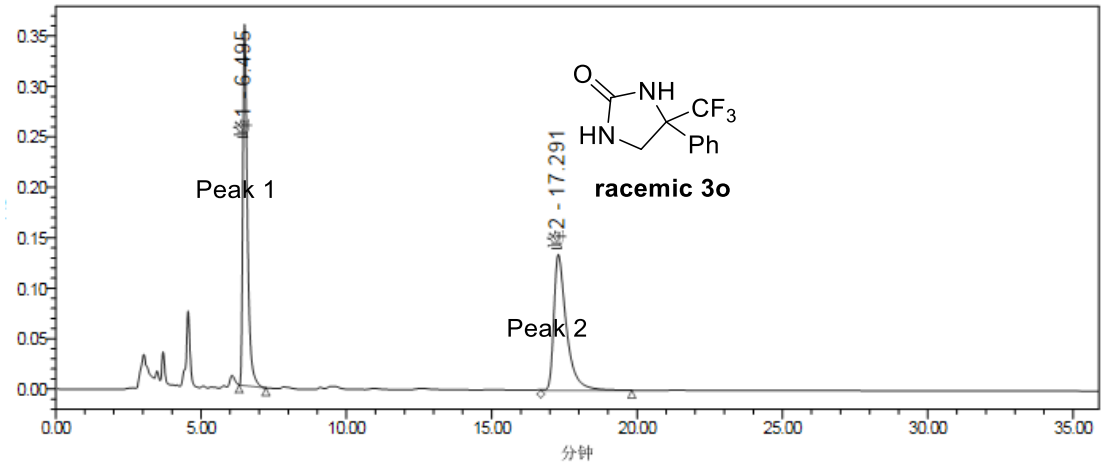
Channel: W2489 ChA; Channel Desc.: W2489 ChA 214nm; Processing Method: 0

	Channel Description	Peak Name	RT (min)	Area (μAU*sec)	% Area	Height (μAU)
1	W2489 ChA 214nm	峰1 Peak 1	5.856	3628583	22.57	274356
2	W2489 ChA 214nm	峰2 Peak 2	6.252	3767654	23.44	268329
3	W2489 ChA 214nm	峰3 Peak 3	7.835	4362593	27.14	262408
4	W2489 ChA 214nm	峰4 Peak 4	8.506	4314997	26.84	267840



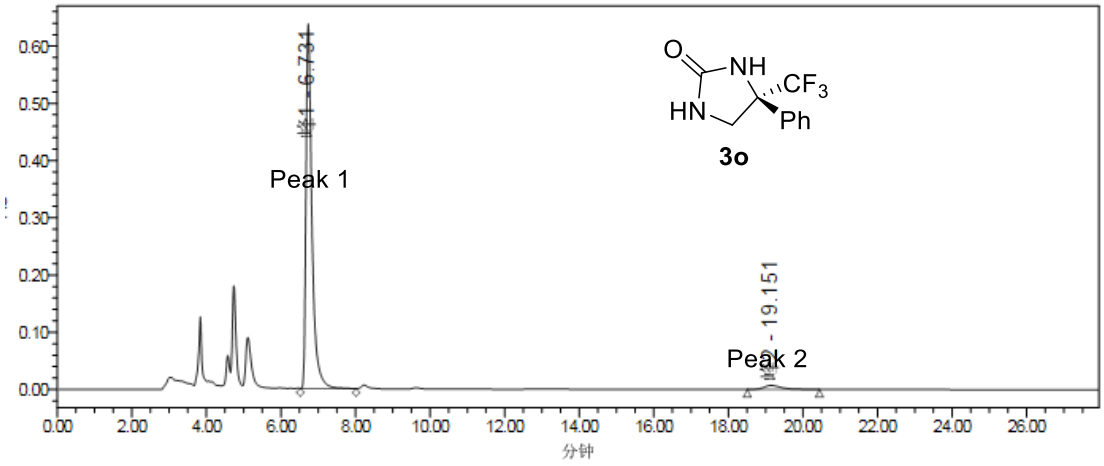
Channel: W2489 ChA; Channel Desc.: W2489 ChA 214nm; Processing Method: 0

	Channel Description	Peak Name	RT (min)	Area (μAU*sec)	% Area	Height (μAU)
1	W2489 ChA 214nm	峰1 Peak 1	5.846	265020	5.74	16683
2	W2489 ChA 214nm	峰2 Peak 2	6.870	75101	1.63	2461
3	W2489 ChA 214nm	峰3 Peak 3	7.693	34917	0.76	2631
4	W2489 ChA 214nm	峰4 Peak 4	8.046	4241448	91.88	208112



Channel: W2489 ChA; Channel Desc.: W2489 ChA 214nm; Processing Method: 0

	Channel Description	Peak Name	RT (min)	Area (μAU*sec)	% Area	Height (μAU)
1	W2489 ChA 214nm	峰1 Peak 1	6.495	3891660	49.07	357908
2	W2489 ChA 214nm	峰2 Peak 2	17.291	4039921	50.93	134408



Channel: W2489 ChA; Channel Desc.: W2489 ChA 214nm; Processing Method: 0

	Channel Description	Peak Name	RT (min)	Area (μAU*sec)	% Area	Height (μAU)
1	W2489 ChA 214nm	峰1 Peak 1	6.731	7118605	96.87	636658
2	W2489 ChA 214nm	峰2 Peak 2	19.151	229846	3.13	7146

8. X-ray Crystallographic Date of compound 3o

Table Crystal data and structure refinement for 3o.

Identification code	1937396
Empirical formula	$C_2H_{1.8}F_{0.6}N_{0.4}O_{0.2}$
Formula weight	46.04
Temperature/K	293(2)
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	7.1385(9)
b/Å	7.5039(10)
c/Å	19.126(3)
α/°	90.00
β/°	90.00
γ/°	90.00
Volume/Å^3	1024.5(2)
Z	20
ρ_{calc}g/cm^3	1.492
μ/mm^{-1}	0.135
F(000)	472.0
Crystal size/mm^3	? × ? × ?
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.26 to 52.84
Index ranges	-8 ≤ h ≤ 8, -9 ≤ k ≤ 8, -14 ≤ l ≤ 23
Reflections collected	5878
Independent reflections	2087 [R_{int} = 0.0518, R_{sigma} = 0.0692]
Data/restraints/parameters	2087/0/145
Goodness-of-fit on F^2	0.933
Final R indexes [I>=2σ (I)]	R_1 = 0.0458, wR_2 = 0.0892
Final R indexes [all data]	R_1 = 0.0883, wR_2 = 0.1034
Largest diff. peak/hole / e Å^{-3}	0.14/-0.17
Flack parameter	0.4(12)

9. References

1. M. Emilie, V. Anna Chiara, M. Michele, M. Andrea, Z. Paolo, B. Luca and F. Mariafrancesca, *Chem. Commun.*, 2015, **46**, 658-660.
2. W. Bin, L. Yuxin, S. Cong, W. Zhonglin, C. Jungang, L. Dapeng, L. Yingjie and D. Haifeng, *Org. Lett.*, 2015, **46**, 6432.
3. K. M. Johnson, M. S. Rattley, F. Sladojevich, D. M. Barber, M. G. Nunez, A. M. Goldys and D. J. Dixon, *Org. Lett.*, 2012, **14**, 2492-2495.
4. B. Wang, T. Xu, L. Zhu, Y. Lan, J. Wang, N. Lu, Z. Wei, Y. Lin and H. Duan, *Org. Chem. Front.* , 2017, **4**, 1266-1271.