# Pd-Catalyzed arylation of linear and angular spirodiamine salts under aerobic conditions

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Supporting Information

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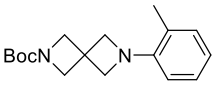
## I. General Details

All chemical compounds were purchased and used without further purification. NMR spectra were taken on a Bruker DMX 500 MHz. Mass spectroscopy data were acquired using ESI technique on 2695 Alliance LCMS. All other commercial reagents were purchased and used without further purification. Purification of organic compounds were carried out on a Biotage Isolera One with a dual-wavelength UV-VIS detector. Chemical shifts (δ) in the NMR spectra ($^{1}$H and $^{13}$C) were referenced by assigning the residual solvent peaks.

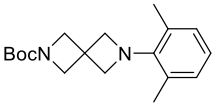
**General procedure for all reactions:** On a bench top, a 40 mL reaction vial was charged with substrate (0.50 mmol), spirodiamine alkane (0.55 mmol), base (3.0 mmol), $Pd_2(dba)_3$ (0.005 mmol), RuPhos (0.01 mmol), and dioxane (1.50 mL). The vial was then closed and sonicated for approximately 1 minute. The vial was then placed in a preheated 100 °C oilbath and stirred vigorously for 20 min. After the allotted reaction time, the reaction vessel was taken out of the oil bath, and solvent was removed under reduced pressure resulting in a crude oily residue. The crude product was purified by flash chromatography on silica gel eluding with a 10-60% gradient of hexanes and EtOAc. Products from Scheme 2, **4a-i**, were eluded with a 20-80% gradient of hexanes and EtOAc. It should be noted, optimal yields of the catalytic products were obtained when using a fresh bottle of NaO-*t*Bu.

## II. Characterization Data of Piperazine Compounds



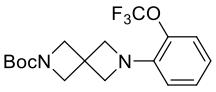
**2a**

Yield: 0.254 g (90%); white solid[1]
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 7.12 (t, $J$ = 7.23, 1H), 7.23 (d, $J$ = 7.23, 1H), 7.46 (t, $J$ = 7.46 1H), 7.95 (d, $J$ = 7.95, 1H), 4.09 (s, 4H), 4.00 (s, 4H), 2.21 (s, 3H), 1.45 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 156.1, 149.5, 131.5, 126.5, 125.1, 119.9, 113.0, 79.6, 63.4, 59.4 (br), 33.2, 28.4, 19.5; **ESI-MS** (m/z): 289.26 [M+H]



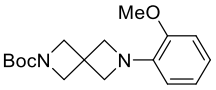
**2b**

Yield: 0.117 g (74%); white solid[1]
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 6.89 (d, $J$ = 7.41, 2H), 6.70 (t, $J$ = 7.69, 2H), 4.22 (s, 4H), 4.06 (s, 4H), 2.28 (s, 6H), 1.45 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 156.1, 149.1, 130.3, 126.1, 120.4, 66.4, 59.7 (br), 33.4, 28.5, 21.1; **ESI-MS** (m/z): 303.41 [M+H]



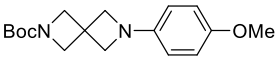
**2c**

Yield: 0.074 g (44%); white solid
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 7.12 (m, 2H), 6.75 (dt, $J$ = 1.20, 7.22, 1H), 6.48 (dd, $J$ = 1.03, 7.01, 1H), 4.08 (s, 4H), 4.07 (s, 4H), 1.45 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 156.1, 143.0, 137.4, 127.4, 121.9 (d, $J_{C,F}$ = 7.18), 120.0 (q, $J_{C,F}$ = 146.25), 114.6, 79.8, 63.5, 59.6 (bs), 33.9, 28.5; **ESI-MS** (m/z): 359.19 [M+H]



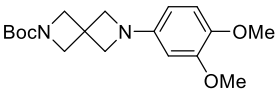
**2d**

Yield: 0.119 g (70%); white solid[2]
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 6.87 (m, 1H), 6.80 (m, 2H), 6.44 (d, $J$ = 7.65, 1H), 4.06 (s, 4H), 4.00 (s, 4H), 3.78 (s, 3H), 1.45 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 156.1, 149.5, 140.7, 121.0, 119.9, 113.4, 111.3, 79.6, 63.6, 59.5 (bs), 55.4, 33.9, 28.5; **ESI-MS** (m/z): 305.28 [M+H]



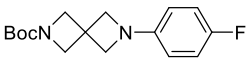
**2e**

Yield: 0.202 g (66%); white solid[1]
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 6.82 (d, $J$ = 8.79, 2H), 6.43 (d, $J$ = 8.79, 2H), 4.07 (s, 4H), 3.90 (s, 4H), 3.75 (s, 3H), 1.45 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 156.1, 152.7, 146.0, 114.8, 113.1, 79.7, 62.8, 59.5 (br), 55.8, 33.5, 28.5; **ESI-MS** (m/z): 305.28 [M+H]



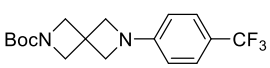
**2f**

Yield: 0.105 g (64%); white solid
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 5.92 (t, $J$ = 1.80, 1H), 5.60 (t, $J$ = 1.94, 2H), 4.07 (s, 4H), 3.93 (s, 4H), 3.75 (s, 6H), 1.44 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 161.7, 156.1, 153.2, 90.7, 90.5, 79.8, 62.2, 59.6 (br), 55.3, 33.3; 28.46; **ESI-MS** (m/z): 335.30 [M+H]



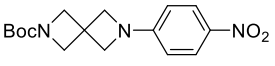
**2g**

Yield: 0.281 g (93%); white solid[3]
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 6.92 (t, $J$ = 8.63, 2H), 6.40 (m, 2H), 4.08 (s, 4H), 3.92 (s, 4H), 1.45 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 157.3 (d, $J_{C,F}$ = 234.78), 156.1, 115.7 (d, $J_{C,F}$ = 1.56), 115.7 (d, $J_{C,F}$ = 22.79), 112.8 (d, $J_{C,F}$ = 7.36), 79.8, 62.6, 59.5 (br), 33.4, 28.4; **ESI-MS** (m/z): 293.27 [M+H]

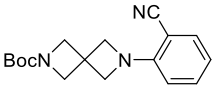


**2h**

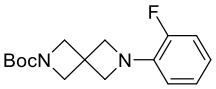
Yield: 0.157 g (91%); white solid[1]
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 7.44 (d, $J$ = 8.58, 2H), 6.44 (d, $J$ = 8.53, 2H), 4.01 (s, 4H), 4.02 (s, 4H), 1.45 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 156.1, 153.0, 126.5 (q, $J_{C,F}$ = 3.73), 124.0 (q, $J_{C,F}$ = 273.25), 119.7 (q, $J_{C,F}$ = 34.11), 111.0, 80.0, 62.0, 59.7 (br), 33.6, 28.5; **ESI-MS** (m/z): 343.17 [M+H]



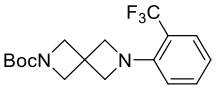
**2i** Yield: 0.141 g (82%); bright-orange solid[4]
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 8.05 (m, 2H), 6.30 (d, $J$ = 9.12, 2H), 4.13 (s, 4H), 4.12 (s, 4H), 1.44 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 156.0, 154.1, 138.3, 126.1, 109.7, 80.1, 61.5, 59.5 (br), 33.4, 28.4; **ESI-MS** (m/z): 320.22 [M+H]



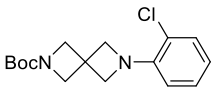
**2j** Yield: 0.062 g (39%); white solid[1]
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 7.40 (dd, $J$ = 1.34, 6.46, 1H), 7.36 (dt, $J$ = 1.43, 7.13, 2H), 6.73 (t, $J$ = 7.23, 1H), 6.42 (d, $J$ = 8.36, 1H), 4.27 (s, 4H), 4.10 (s, 4H), 1.44 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 156.1, 152.6, 134.5, 133.5, 119.3, 117.9, 113.0, 94.9, 80.0, 63.1, 59.4 (br), 33.4, 28.5; **ESI-MS** (m/z): 300.34 [M+H]



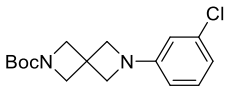
**2k** Yield: 0.123 g (74%); white solid[5]
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 6.96 (m, 2H), 6.71 (m, 1H), 6.45 (t, $J$ = 8.25, 1H), 4.08 (s, 4H), 4.04 (s, 4H), 1.45 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 156.1, 153.6 (d, $J_{C,F}$ =240.79), 139.1 (d, $J_{C,F}$ =10.92), 124.4 (d, $J_{C,F}$ =3.39), 119.3 (d, $J_{C,F}$ =6.78), 115.9 (d, $J_{C,F}$ =18.92), 114.5 (d, $J_{C,F}$ =3.86), 79.8, 63.5 (d, $J_{C,F}$ =1.68), 59.4 (br), 33.3 (d, $J_{C,F}$ =2.38), 28.5; **ESI-MS** (m/z): 293.27 [M+H]



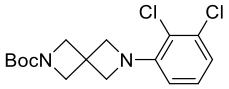
**2l** Yield: 0.129 g (76%); white solid[6]
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 7.49 (d, $J$ = 7.33, 1H), 7.35 (t, $J$ = 8.04, 1H), 6.78 (t, $J$ = 7.45, 1H), 6.47 (d, $J$ = 8.27, 1H), 4.12 (s, 4H), 4.09 (s, 4H), 1.45 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 156.1, 147.4, 127.5 (q, $J_{C,F}$ =5.56), 123.7 (q, $J_{C,F}$ =271.53), 117.3, 114.0 (q, $J_{C,F}$ =30.94), 79.8, 63.4 (d, $J_{C,F}$ =2.47), 60.9 (br), 59.6 (br), 32.6, 28.5; **ESI-MS** (m/z): 343.31 [M+H]



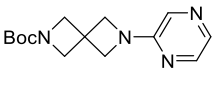
**2m** Yield: 0.084 g (46%); white solid
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 7.20 (d, $J$ = 7.89, 1H), 7.11 (t, $J$ = 7.52, 1H), 6.74 (t, $J$ = 7.52, 1H), 6.49 (d, $J$ = 7.89, 1H), 4.12 (s, 4H), 4.08 (s, 4H), 1.45 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 156.1, 147.1, 130.8, 127.3, 120.6, 120.3, 114.7, 79.8, 63.7, 59.4 (br), 33.3, 28.5; **ESI-MS** (m/z): 309.15 [M+H]



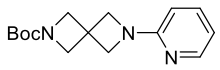
**2n** Yield: 0.114 g (72%); white solid
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 7.12 (t, $J$ = 8.03, 1H), 6.72 (d, $J$ = 7.86, 1H), 6.39 (s, 1H), 6.30 (dd, $J$ = 0.75, 7.41, 1H), 4.08 (s, 4H), 3.95 (s, 4H), 1.44 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 156.1, 152.2, 135.0, 130.1, 118.0, 111.8, 109.9, 79.9, 62.2, 59.7 (br), 33.6, 28.5; **ESI-MS** (m/z): 309.41 [M+H]



**2o** Yield: 0.037 g (19%); white solid
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 7.05 (t, $J$ = 8.01, 1H), 6.92 (d, $J$ = 7.81, 1H), 6.39 (d, $J$ = 8.31, 1H), 4.15 (s, 4H), 4.08 (s, 4H), 1.44 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 156.1, 148.8, 134.3, 127.4, 121.0, 119.0, 112.7, 79.9, 64.0, 59.6 (br), 33.1, 28.5; **ESI-MS** (m/z): 343.17 [M+H]

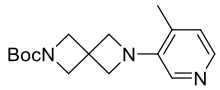


**4a** Yield: 0.085 g (62%); off-white solid
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 7.99 (m, 1H), 7.85 (d, $J$ = 2.55, 1H), 7.76 (s, 1H), 4.17 (s, 4H), 4.09 (s, 4H), 1.41 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 156.0, 115.6, 142.1, 133.6, 130.5, 79.9, 60.8, 59.6 (br), 34.0, 28.4; **ESI-MS** (m/z): 277.26 [M+H]



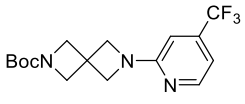
**4b**

Yield: 0.129 g (75%); white solid[1]
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 8.13 (t, *J* = 3.60, 1H), 7.43 (dt, *J* = 1.83, 7.30, 1H), 6.61 (t, *J* = 5.53, 1H), 6.60 (d, *J* = 8.29, 1H), 4.09 (s, 4H), 4.07 (s, 4H), 1.42 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 160.4, 156.1, 148.3, 137.2, 113.5, 106.2, 79.8, 60.9, 59.7 (br), 33.5, 28.5; **ESI-MS** (m/z): 276.32 [M+H]



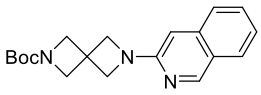
**4c**

Yield: 0.100 g (65%); light yellow solid
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 7.97 (d, *J* = 4.41, 1H), 7.73 (s, 1H), 6.88 (d, *J* = 4.60, 1H), 4.07 (s, 4H), 4.02 (s, 4H), 2.16 (s, 3H), 1.41 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 156.1, 154.1, 141.6, 135.2, 133.2, 125.8, 79.8, 63.5, 59.4 (br), 33.9, 28.4, 18.8; **ESI-MS** (m/z): 290.33 [M+H]



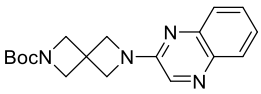
**4d**

Yield: 0.132 g (75%); light-yellow solid
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 8.25 (d, *J* = 5.11, 1H), 6.77 (d, *J* = 5.12, 1H), 6.41 (s, 1H), 4.15 (s, 4H), 4.10 (s, 4H), 1.43 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 160.1, 156.1, 149.7, 139.7 (q, $J_{C,F}$ = 34.32), 124.2 (q, $J_{C,F}$ = 274.56), 108.5 (q, $J_{C,F}$ = 3.23), 101.7 (q, $J_{C,F}$ = 3.23), 80.0, 60.8, 59.6 (br), 33.5, 28.4; **ESI-MS** (m/z): 344.24 [M+H]



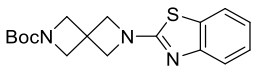
**4e**

Yield: 0.157 g (83%); off-white solid
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 7.84 (d, *J* = 8.86, 1H), 7.73 (d, *J* = 8.42, 1H), 7.58 (dd, *J* = 1.01, 6.95, 1H), 7.52 (dt, *J* = 1.14, 5.48, 1H), 7.23 (dt, *J* = 0.96, 6.95, 1H), 6.54 (d, *J* = 8.85, 1H), 4.24 (s, 4H), 4.11 (s, 4H), 1.45 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 158.4, 156.1, 148.0, 137.5, 129.8, 127.6, 126.5, 123.4, 122.5, 108.7, 79.8, 60.7, 59.7 (bs), 33.3, 28.5; **ESI-MS** (m/z): 326.23 [M+H]



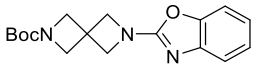
**4f**

Yield: 0.130 g (81%); yellow solid
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 8.62 (d, *J* = 1.68, 1H), 8.50 (d, *J* = 1.72, 1H), 7.86 (d, *J* = 8.90, 1H), 6.98 (dd, *J* = 2.61, 6.50, 1H), 6.79 (d, *J* = 2.38, 1H), 4.12 (s, 4H), 4.11 (s, 4H), 1.42 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 156.0, 151.3, 145.3, 144.7, 141.0, 137.9, 130.4, 118.4, 105.2, 79.9, 62.0, 59.5 (bs), 33.4, 28.4; **ESI-MS** (m/z): 327.29 [M+H]



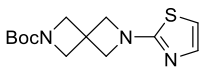
**4g**

Yield: 0.125 g (65%); light-yellow solid
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 7.58 (d, *J* = 8.61, 1H), 7.29 (t, *J* = 8.04, 1H), 7.09 (t, *J* = 7.80, 1H), 4.27 (s, 4H), 4.11 (s, 4H), 1.44 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 168.1, 156.0, 152.8, 131.7, 126.2, 121.9, 121.1, 119.6, 80.1, 63.0, 59.6 (bs), 33.4, 28.4; **ESI-MS** (m/z): 332.23 [M+H]



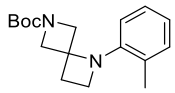
**4h**

Yield: 0.119 g (67%); off-white solid
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 7.43 (d, *J* = 7.76, 1H), 7.31 (d, *J* = 7.57, 1H), 7.22 (dt, *J* = 0.79, 7.73, 1H), 7.11 (dt, *J* = 0.96, 7.69, 1H), 4.41 (s, 4H), 4.17 (s, 4H), 1.49 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 162.4, 156.0, 149.4, 142.9, 124.2, 121.4, 116.9, 109.1, 80.1, 61.5, 59.6 (bs), 34.3, 28.5; **ESI-MS** (m/z): 316.22 [M+H]

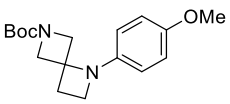


**4i**

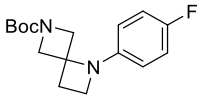
Yield: 0.078 g (45%); tan solid
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 7.14 (d, *J* = 3.54, 1H), 6.56 (d, *J* = 3.65, 1H), 4.15 (s, 4H), 4.08 (s, 4H), 1.41 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 171.7, 156.0, 139.8, 108.9,79.9, 63.5, 59.5 (bs), 34.4, 28.4; **ESI-MS** (m/z): 282.19 [M+H]



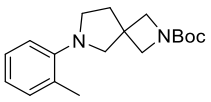
**6a** Yield: 0.266 g (87%); beige white solid
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 7.10 (dt, *J* = 1.36, 8.53, 1H), 7.05 (d, *J* = 7.42, 1H), 6.81 (d, *J* = 8.00, 1H), 6.76 (t, *J* = 7.37, 1H), 4.60 (d, *J* = 10.19, 2H), 4.05 (t, *J* = 7.00, 2H), 3.96 (d, *J* = 10.32, 2H), 2.45 (t, *J* = 6.94, 2H), 2.32 (s, 3H), 1.48 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 156.5, 145.7, 132.7, 126.8, 125.0, 119.4, 114.3, 79.8, 66.2, 59.4 (bs), 50.4, 31.1, 28.5, 20.3; **ESI-MS** (m/z): 289.13 [M+H]



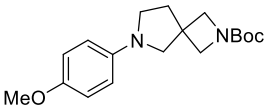
**6b** Yield: 0.101 g (32%); beige white solid
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 6.85 (dt, *J* = 3.67, 8.93, 2H), 6.54 (dt, *J* = 3.64, 8.98, 2H), 4.48 (dd, *J* = 0.78, 10.02, 2H), 3.95 (d, *J* = 10.18, 2H), 3.76 (s, 3H), 3.66 (t, *J* = 6.88, 2H), 2.47 (t, *J* = 6.78, 2H), 1.47 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 156.5, 152.7, 141.3, 115.2, 113.5, 79.7, 65.0, 59.3 (bs), 55.8, 46.4, 29.5, 28.5; **ESI-MS** (m/z): 305.28 [M+H]



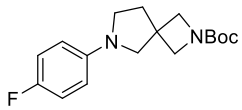
**6c** Yield: 0.217 g (77%); beige white solid
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 6.96 (dt, *J* = 1.53, 8.82, 2H), 6.50 (m, 2H), 4.47 (d, *J* = 10.11, 2H), 3.95 (d, *J* = 10.12, 2H), 3.66 (dt, *J* = 1.34, 6.84, 2H), 2.47 (t, *J* = 7.00, 2H), 1.46 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 157.3 (d, $J_{C,F}$ = 236.61), 156.5, 143.4 (d, $J_{C,F}$ = 1.48), 116.1 (d, $J_{C,F}$ = 22.27), 113.0 (d, $J_{C,F}$ = 7.45), 79.9, 65.0, 59.3 (bs), 46.5, 29.4, 28.5; **ESI-MS** (m/z): 293.13 [M+H]



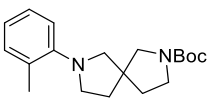
**6d** Yield: 0.283 g (93%); clear viscous oil
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 7.12 (t, *J* = 7.24, 2H), 6.86 (m, 2H), 3.90 (q, *J* = 8.62, 12.24, 4H), 3.34 (s, 2H), 3.25 (t, *J* = 6.82, 2H), 2.31 (s, 3H), 2.15 (t, *J* = 6.92, 2H), 1.47 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 156.4, 148.6, 131.8, 129.1, 126.5, 121.1, 116.0, 61.42, 59.8 (bs), 50.1, 39.7, 36.7, 28.5, 20.3; **ESI-MS** (m/z): 303.27 [M+H]



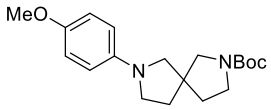
**6e** Yield: 0.164 g (54%); white solid
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 6.86 (dt, *J* = 3.78, 8.93, 2H), 6.51 (dt, *J* = 3.65, 9.04, 2H), 3.91 (d, *J* = 8.67, 2H), 3.85 (d, *J* = 8.67, 2H), 3.75 (s, 3H), 3.40 (s, 2H), 3.31 (t, *J* = 6.86, 2H), 2.18 (t, *J* = 6.85, 2H), 1.45 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 156.3, 151.3, 142.6, 115.1, 112.6, 79.5, 59.3 (bs), 58.3, 56.0, 47.2, 39.9, 36.3, 28.4; **ESI-MS** (m/z): 319.29 [M+H]



**6f** Yield: 0.273 g (86%); white solid
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 6.94 (dt, *J* = 3.73, 8.83, 2H), 6.44 (m, 2H), 3.92 (d, *J* = 8.63, 2H), 3.86 (d, *J* = 8.63, 2H), 3.40 (s, 2H), 3.32 (t, *J* = 6.79, 2H), 2.19 (t, *J* = 6.82, 2H), 1.45 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 156.3 (d, $J_{C,F}$ = 261.87), 156.1, 144.3 (d, $J_{C,F}$ = 1.30), 115.7 (d, $J_{C,F}$ = 22.02), 112.2 (d, $J_{C,F}$ = 7.24), 79.6, 59.3 (bs), 58.1, 47.1, 39.9, 36.3, 28.4; **ESI-MS** (m/z): 307.28 [M+H]

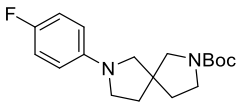


**6g** Yield: 0.146 g (48%); clear oil
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 7.13 (m, 2H), 6.87 (m, 2H), 3.35 (m, 6H), 3.25 (m, 2H), 2.33 (s, 3H), 1.95 (m, 2H), 1.89 (m, 2H), 1.48 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 154.9, 148.8, 131.9, 129.2, 129.1, 126.6, 121.0, 116.1, 79.4, 79.3, 60.8, 60.6, 56.5, 55.9, 53.6, 50.4, 48.6, 47.8, 45.5, 36.5, 35.8, 35.5, 35.4, 28.7, 20.5; **ESI-MS** (m/z): 317.28 [M+H]



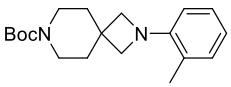
**6h**

Yield: 0.073 g (22%); clear oil
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 6.85 (m, 2H), 6.51 (m, 2H), 3.75 (s, 3H), 3.49 (m, 2H), 3.37 (m, 3H), 3.31 (m, 1H), 3.20 (m, 2H), 1.96 (m, 3H), 1.88 (m, 1H), 1.47 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 154.7, 151.1, 142.8, 115.1, 112.4, 79.3, 57.7, 56.0, 55.2, 53.4, 48.8, 48.0, 47.6, 45.3, 45.0, 36.0, 35.2, 35.1, 28.5; **ESI-MS** (m/z): 333.30 [M+H]



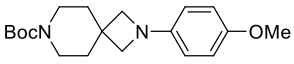
**6i**

Yield: 0.192 g (58%); white solid
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 6.94 (m, 2H), 6.44 (m, 2H), 3.47 (m, 2H), 3.42 (m, 3H), 3.28 (m, 1H), 3.18 (m, 2H), 2.02 (m, 1H), 1.94 (m, 2H), 1.82 (m, 1H), 1.47 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ156.0 (d, $J_{C,F}$ = 234.25), 154.6, 144.5, 115.7 (d, $J_{C,F}$ = 22.18), 112.1 (d, $J_{C,F}$ = 7.21), 79.4, 79.3, 57.5, 55.8, 55.1, 48.9, 48.0, 47.5, 45.3, 45.0. 35.8, 35.1, 34.9, 28.6; **ESI-MS** (m/z): 321.29 [M+H]



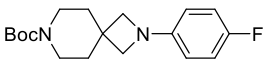
**6j**

Yield: 0.262 g (79%); white solid
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 7.10 (dt, $J$ = 1.09, 8.47, 1H), 7.03 (d, $J$ = 7.47, 1H), 6.75 (dt, $J$ = 0.85, 7.39, 1H), 6.48 (dd, $J$ = 0.44, 7.89, 1H), 3.68 (s, 4H), 3.41 (t, $J$ = 5.65, 4H), 2.23 (s, 3H), 1.78 (t, $J$ = 5.65, 4H), 1.47 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 154.9, 152.5, 142.3, 115.0, 114.7, 79.8, 68.5, 67.2, 55.8,45.5, 41.1 (bs), 34.8, 28.6, 27.7; **ESI-MS** (m/z): 261.25 [M-$CCH_3$+H], 317.15 [M+H]



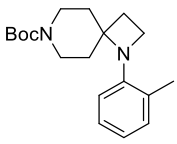
**6k**

Yield: 0.262 g (79%); white solid
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 6.83 (dt, $J$ = 3.47, 8.87, 2H), 6.43 (dt, $J$ = 3.37, 8.91, 2H), 3.91 (d, $J$ = 8.67, 2H), 3.75 (s, 3H), 3.40 (s, 2H), 3.39 (m, 4H), 1.76 (m, $J$ = 6.85, 2H), 1.46 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 154.9, 152.1, 146.5, 114.8, 112.5, 79.5, 62.1, 55.8, 41.2 (bs), 35.7, 34.6, 28.5; **ESI-MS** (m/z): 277.26 [M-$CCH_3$+H], 333.30 [M+H]



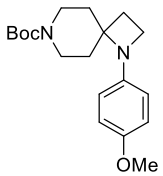
**6l**

Yield: 0.130 g (40%); white solid
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 6.91 (dt, $J$ = 2.21, 8.75, 2H), 6.36 (m, 2H), 3.58 (s, 4H), 3.39 (t, $J$ = 5.66, 4H), 1.77 (t, $J$ = 5.70, 4H), 1.46 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 156.9 (d, $J_{C,F}$ = 233.97), 154.9, 148.4 (d, $J_{C,F}$ = 1.73), 115.6 (d, $J_{C,F}$ = 22.22), 112.2 (d, $J_{C,F}$ = 7.73), 79.6, 62.0, 41.0 (bs), 35.7, 34.7, 28.5; **ESI-MS** (m/z): 265.25 [M-$CCH_3$+H]



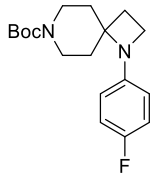
**6m**

Yield: 0.064 g (22%); yellow oil
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 7.06 (t, $J$ = 7.86, 2H), 6.87 (d, $J$ = 7.84, 1H), 6.78 (t, $J$ = 7.35, 1H), 4.09 (bs, 2H), 3.99 (t, $J$ = 6.87, 2H), 2.72 (bs, 2H), 2.33 (s, 3H), 2.10 (t, $J$ = 7.32, 2H), 1.98 (bs, 2H), 1.80 (d, $J$ = 12.19, 2H), 1.46 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 154.8, 146.3, 132.0, 128.6, 126.0, 120.3, 117.9, 79.6, 70.5, 47.5, 40.8 (bs), 34.7, 28.5, 27.9, 20.7; **ESI-MS** (m/z): 317.28 [M+H]

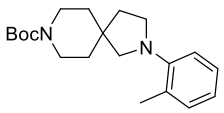


**6n**

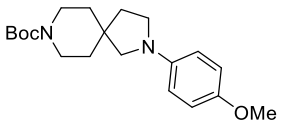
Yield: 0.030 g (<10%); yellow oil
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 6.78 (dt, $J$ = 3.53, 8.97, 2H), 6.46 (dt, $J$ = 3.47, 8.90, 2H), 4.11 (bs, 2H), 3.73 (s, 3H), 3.66 (t, $J$ = 7.10, 2H), 2.71 (m, 2H), 2.11 (t, $J$ = 6.94, 2H), 2.04 (m, 2H), 1.70 (d, $J$ = 12.47, 2H), 1.47 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 154.9, 150.1, 131.5, 126.5, 124.8, 119.2, 112.7, 79.6, 63.0, 40.9 (bs), 35.7, 34.2, 28.5, 19.7; **ESI-MS** (m/z): 333.30 [M+H]



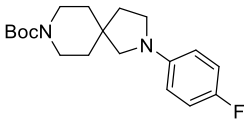
**6o** Yield: 0.071 g (21%); yellow solid
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 6.89 (dt, $J$ = 2.30, 8.84 2H), 6.42 (m, 2H), 4.14 (bs, 2H), 3.68 (m, 2H), 2.71 (bs, 2H), 2.12 (t, $J$ = 7.16, 2H), 2.04 (m, 2H), 1.72 (d, $J$ = 12.72, 2H), 1.47 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 157.1 (d, $J_{C,F}$ = 236.01), 154.9, 144.6 (d, $J_{C,F}$ = 2.12), 115.5 (d, $J_{C,F}$ = 22.16), 114.4 (d, $J_{C,F}$ = 7.57), 79.8, 68.7, 45.6, 41.1 (bs), 34.7, 28.6, 27.7; **ESI-MS** (m/z): 321.15 [M+H]



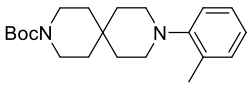
**6p** Yield: 0.286 g (87%); beige solid
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 7.12 (m, 2H), 6.86 (m, 2H), 3.43 (m, 4H), 3.27 (t, $J$ = 6.98, 2H), 3.04 (s, 2H), 1.81 (s, 3H), 1.80 (t, $J$ = 7.04, 2H), 1.62 (m, 4H), 1.48 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 154.9, 148.9, 131.6, 129.1, 126.4, 120.8, 116.0, 79.4, 61.6, 49.7, 41.4 (bs), 40.3, 36.1, 28.5, 20.3; **ESI-MS** (m/z): 331.30 [M+H]



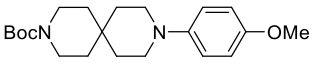
**6q** Yield: 0.229 g (91%); tan solid
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 6.85 (dt, $J$ = 3.25, 8.99, 2H), 6.51 (dt, $J$ = 3.37, 8.82, 2H), 3.75 (s, 3H), 3.51 (m, 2H), 3.35 (m, 2H), 3.31 (t, $J$ = 6.74, 2H), 3.11 (s, 2H), 1.88 (t, $J$ = 6.77, 2H), 1.57 (m, 4H), 1.47 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 154.9, 151.0, 143.0, 115.1, 112.4, 79.5, 58.4, 56.0, 46.8, 41.3 (bs), 40.7, 36.1, 35.4, 28.5; **ESI-MS** (m/z): 347.31 [M+H]



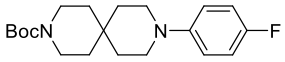
**6r** Yield: 0.244 g (67%); yellow solid
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 6.93 (dt, $J$ = 3.72, 8.85, 2H), 6.44 (m, 2H), 3.48 (m, 2H), 3.38 (m, 2H), 3.33 (t, $J$ = 6.90, 2H), 3.11 (s, 2H), 1.88 (t, $J$ = 6.95, 2H), 1.59 (m, 4H), 1.47 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 155.9 (d, $J_{C,F}$ = 233.79), 154.9, 144.7, 115.7 (d, $J_{C,F}$ = 22.11), 112.0 (d, $J_{C,F}$ = 7.26), 79.5, 58.3, 46.7, 41.4 (bs), 40.8, 36.0, 35.2, 28.5; **ESI-MS** (m/z): 335.17 [M+H]



**6s** Yield: 0.160 g (59%); tan solid
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 7.16 (m, 2H), 7.02 (d, $J$ = 7.50, 1H), 6.96 (dt, $J$ = 0.94, 7.38, 1H), 3.42 (t, $J$ = 5.83, 4H), 2.86 (t, $J$ = 5.39, 4H), 2.30 (s, 3H), 1.65 (t, $J$ = 5.48, 4H), 1.52 (m, 4H), 1.47 (s, 9H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 155.1, 152.4, 132.7, 131.1, 126.5, 122.9, 118.9, 79.3, 47.8, 39.5 (bs), 36.3, 29.7, 28.6, 17.9; **ESI-MS** (m/z): 345.31 [M+H]



**6t** Yield: 0.3410 g (85%); tan solid
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 6.90 (dt, $J$ = 2.25, 9.00, 2H), 6.82 (dt, $J$ = 3.50, 9.00, 2H), 3.74 (s, 3H), 3.39 (t, $J$ = 5.73, 4H), 3.03 (t, $J$ = 5.36, 4H), 1.64 (t, $J$ = 5.66, 4H), 1.45 (s, 13H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 155.0, 153.6, 146.3, 118.4, 114.4, 79.3, 55.6, 46.6, 39.4 (bs),35.6, 29.5, 28.5; **ESI-MS** (m/z): 361.33 [M+H]



**6u** Yield: 0.274 g (59%); white solid[7]
**$^1$H NMR** (500 MHz, $CDCl_3$) δ 6.96 (dt, $J$ = 2.26, 9.03, 2H), 6.87 (m, 2H), 3.42 (t, $J$ = 5.71, 4H), 3.08 (t, $J$ = 5.68, 4H), 1.66 (t, $J$ = 5.70, 4H), 1.49 (s, 13H). **$^{13}$C NMR** (125 MHz, $CDCl_3$) δ 157.9 (d, $J_{C,F}$ = 239.67), 155.0, 148.5 (d, $J_{C,F}$ = 2.20), 117.9 (d, $J_{C,F}$ = 7.58), 115.5 (d, $J_{C,F}$ = 22.11), 79.3, 46.1, 39.5 (bs), 35.4, 29.4, 28.5; **ESI-MS** (m/z): 349.31 [M+H]

## III. References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3. K. Yamamoto, S. Aratake, K. Hemmi, M. Mizutani and Y. Seno, *"Chemokine receptor activity regulator*," EP2781216A1, 2014.
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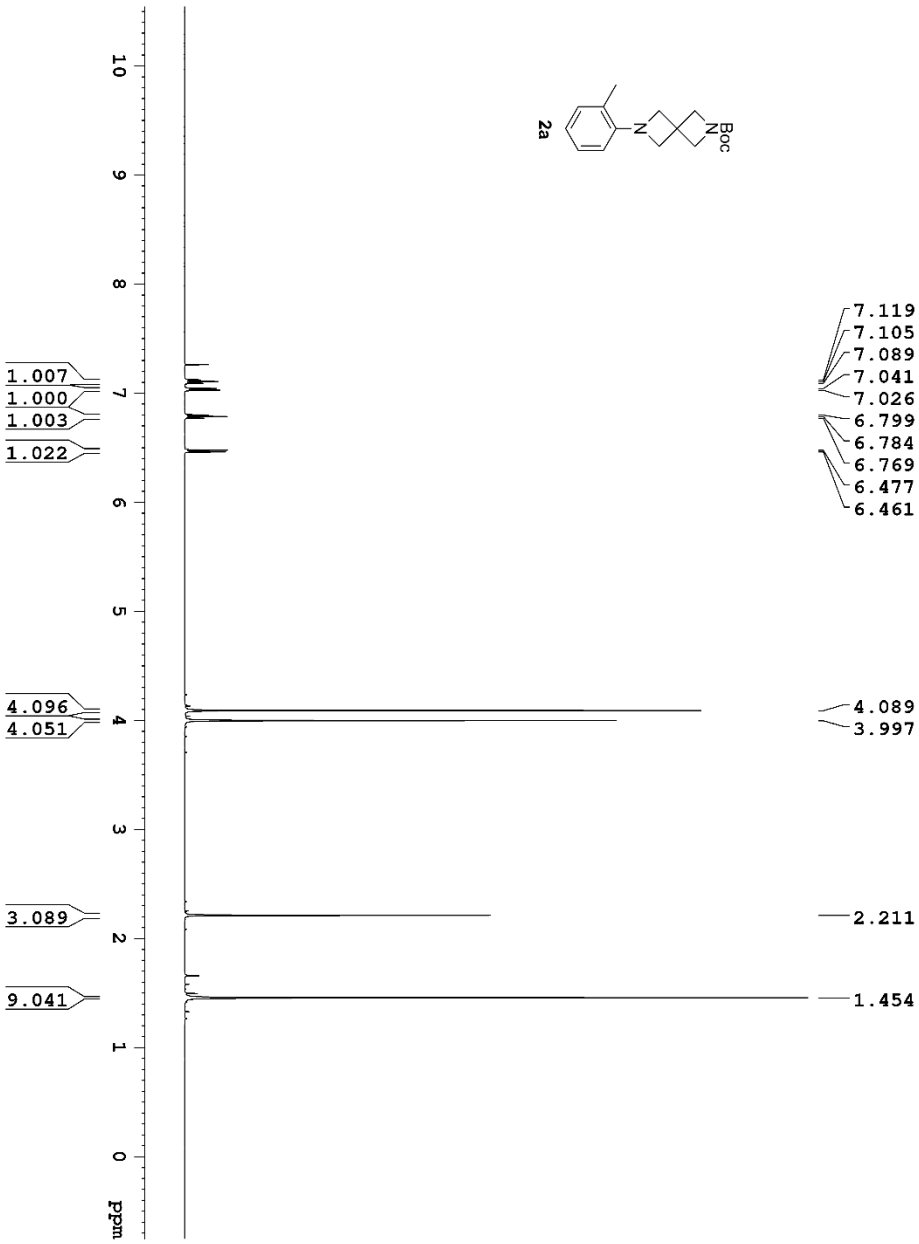
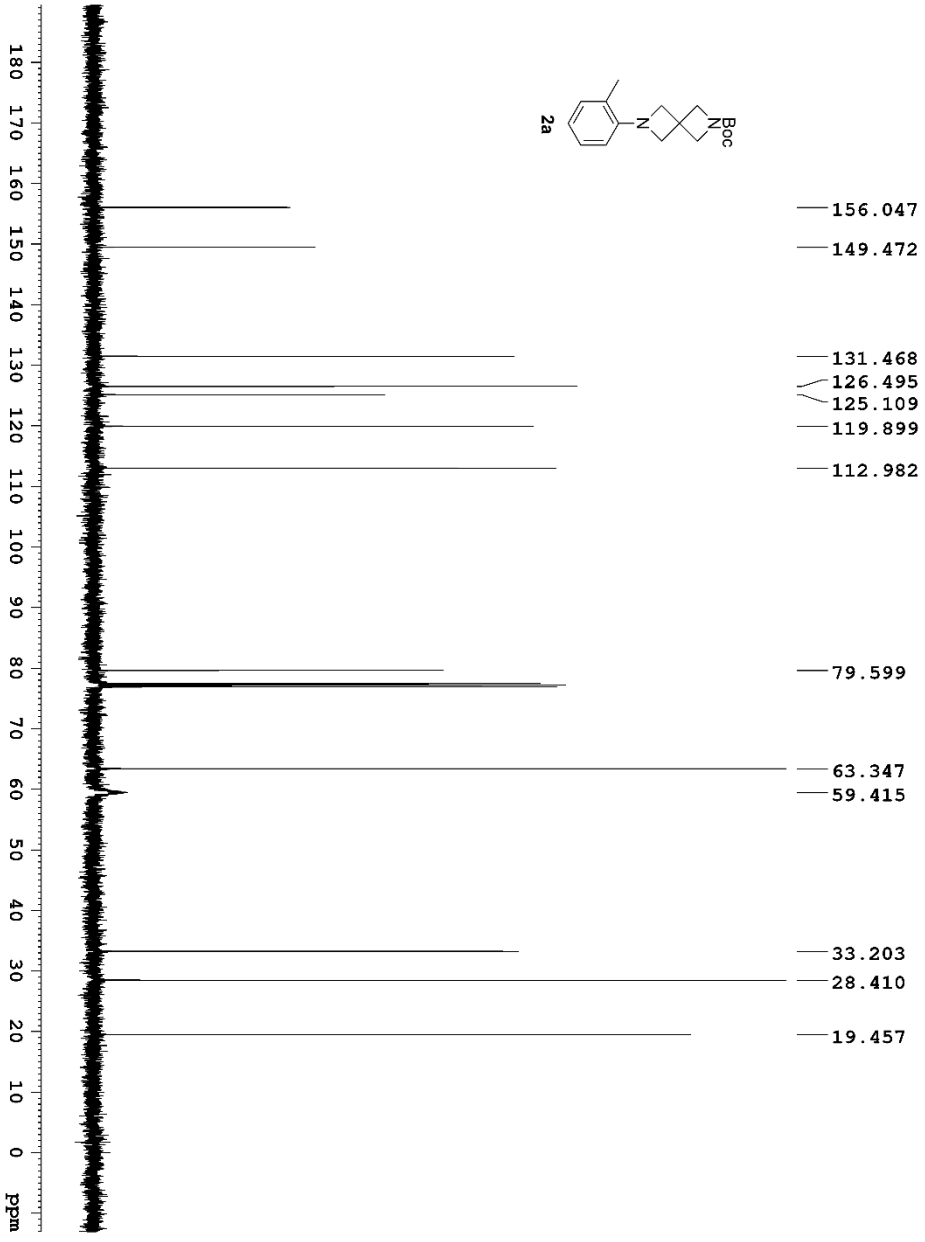
5. T. Endo, R. Takahashi, H. Tanaka and T. Kunigami, *"Diazaspiroalkane derivative*," WO2010123018A1, 2010.
6. C. L. Cioffi, N. Dobri, E. E. Freeman, M. P. Conlon, P. Chen, D. G. Stafford, D. M. C. Schwarz, K. C. Golden, et al., *J. Med. Chem.*, 2014, **57**, 7731.
7. S. Schunk, M. Reich, S. Oberborsch, M. Engels, T. Germann, R. Jostock and C. Kneip, *"Substituted spiro-amide compounds*," WO2010108651A1, 2010.

## IV. Spectroscopic Data

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59.415
33.203
28.410
19.457
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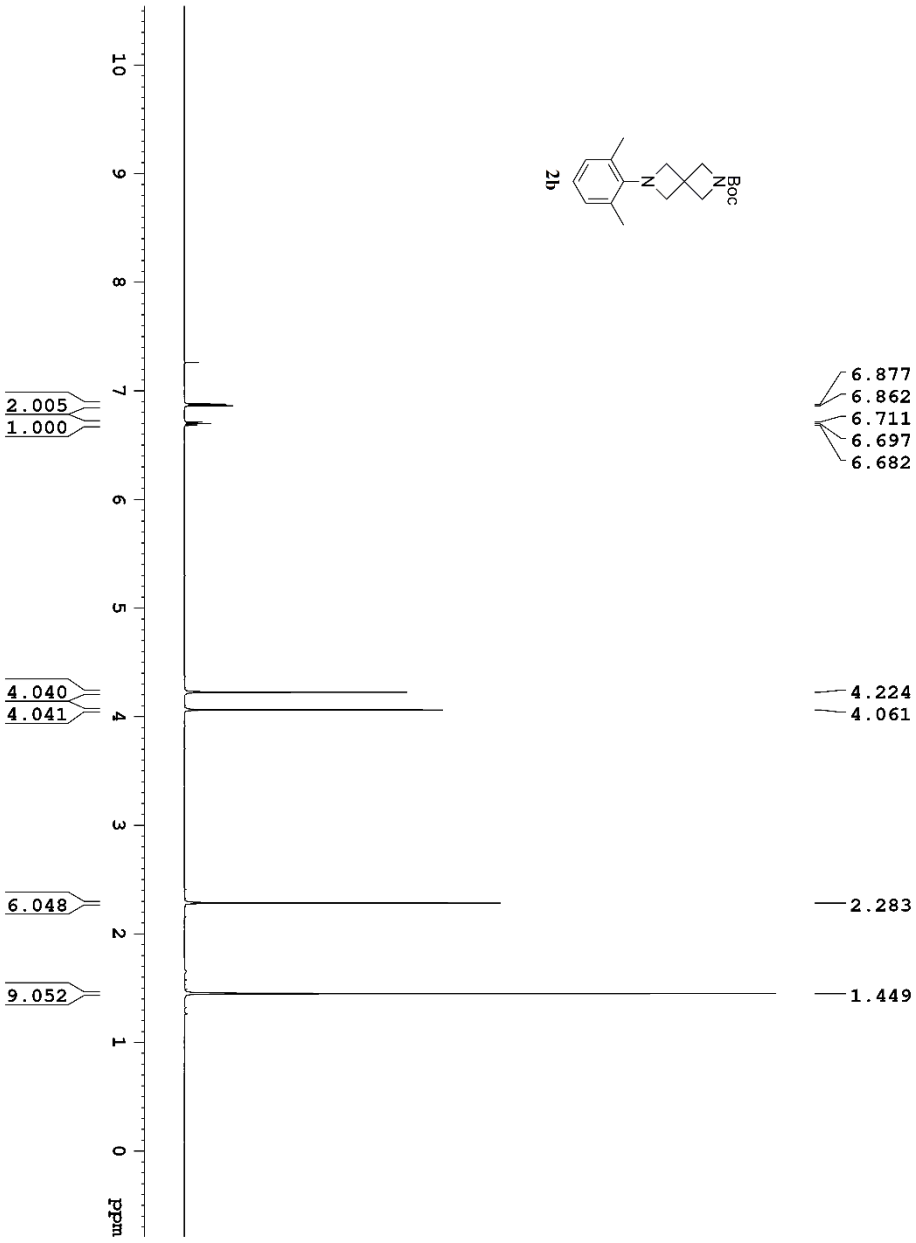
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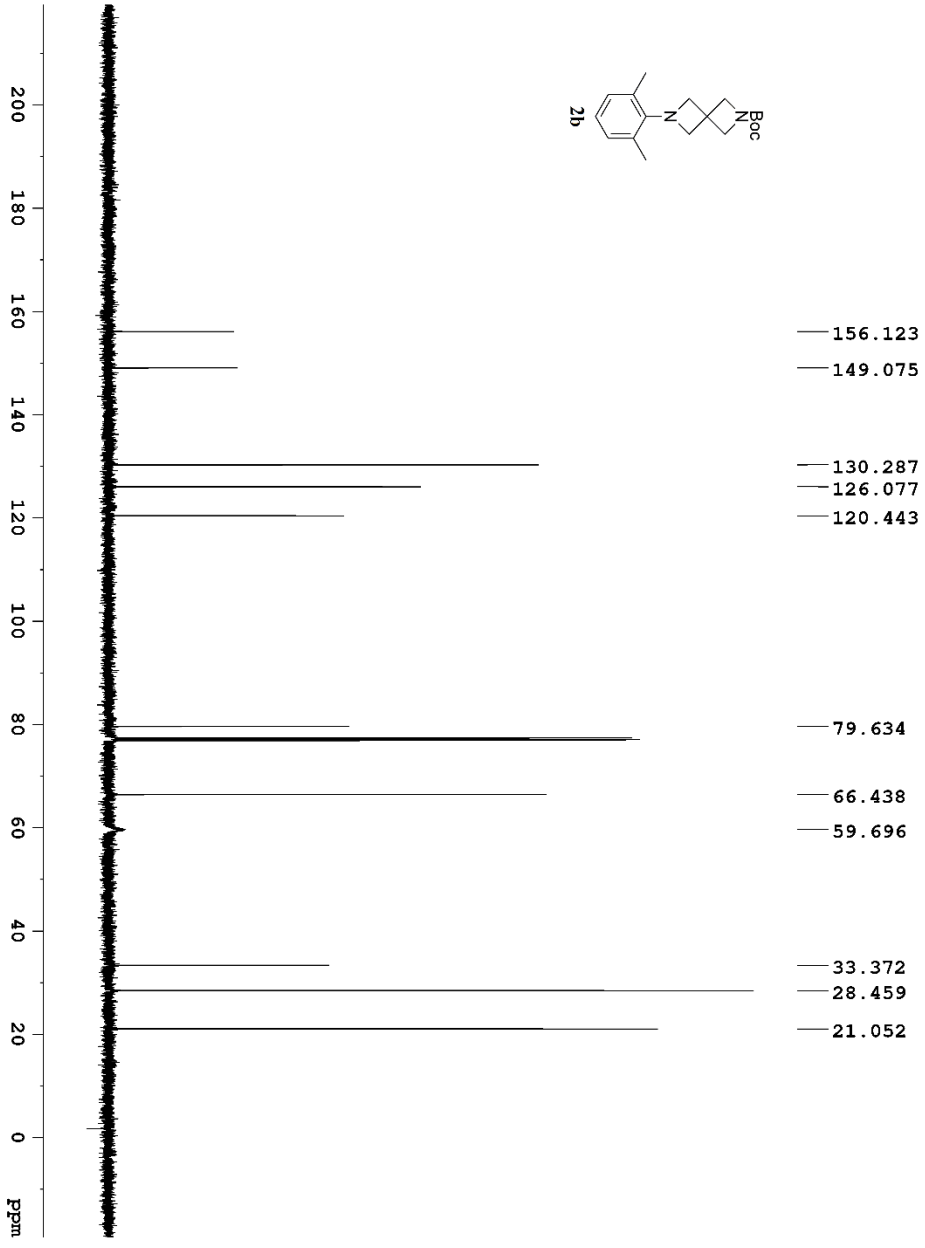
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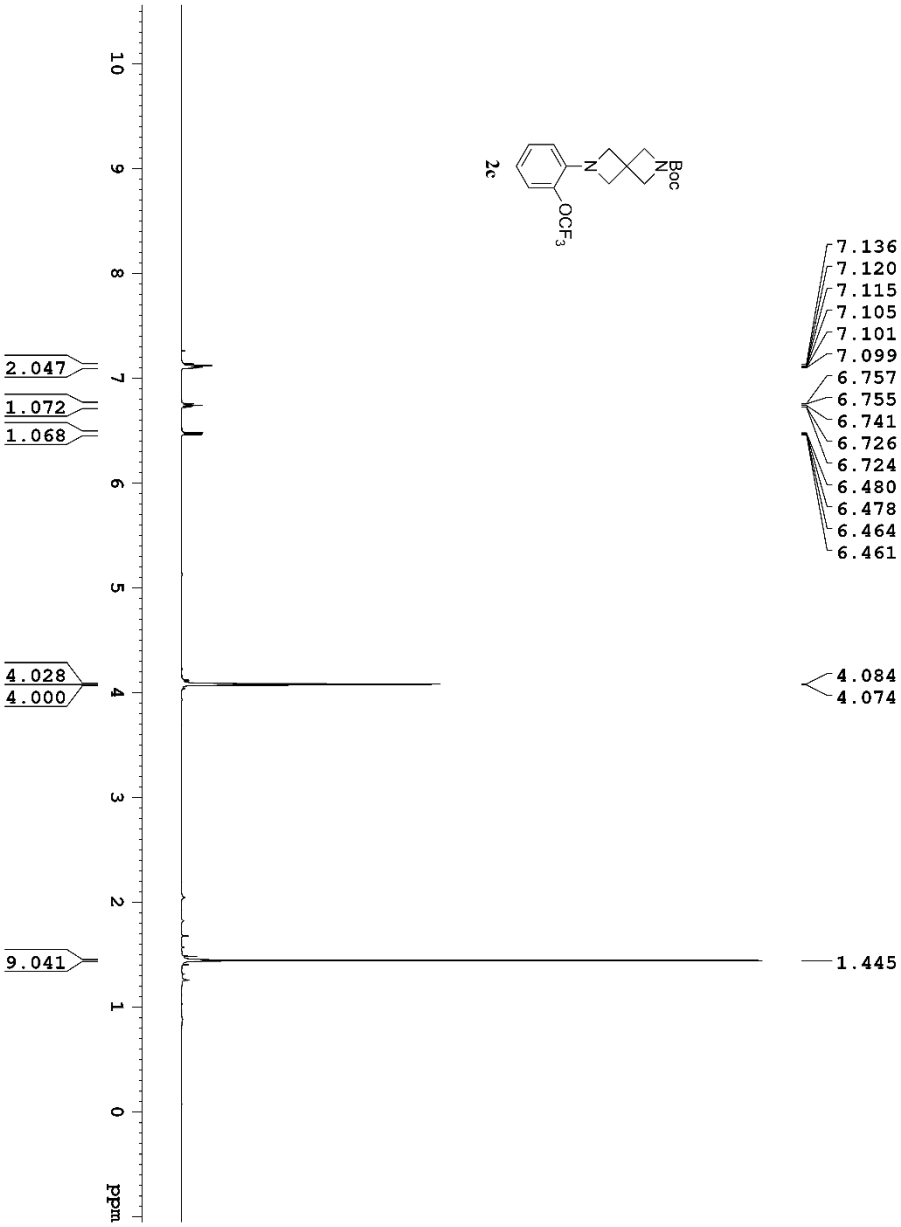
20
0
ppm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6.724
6.480
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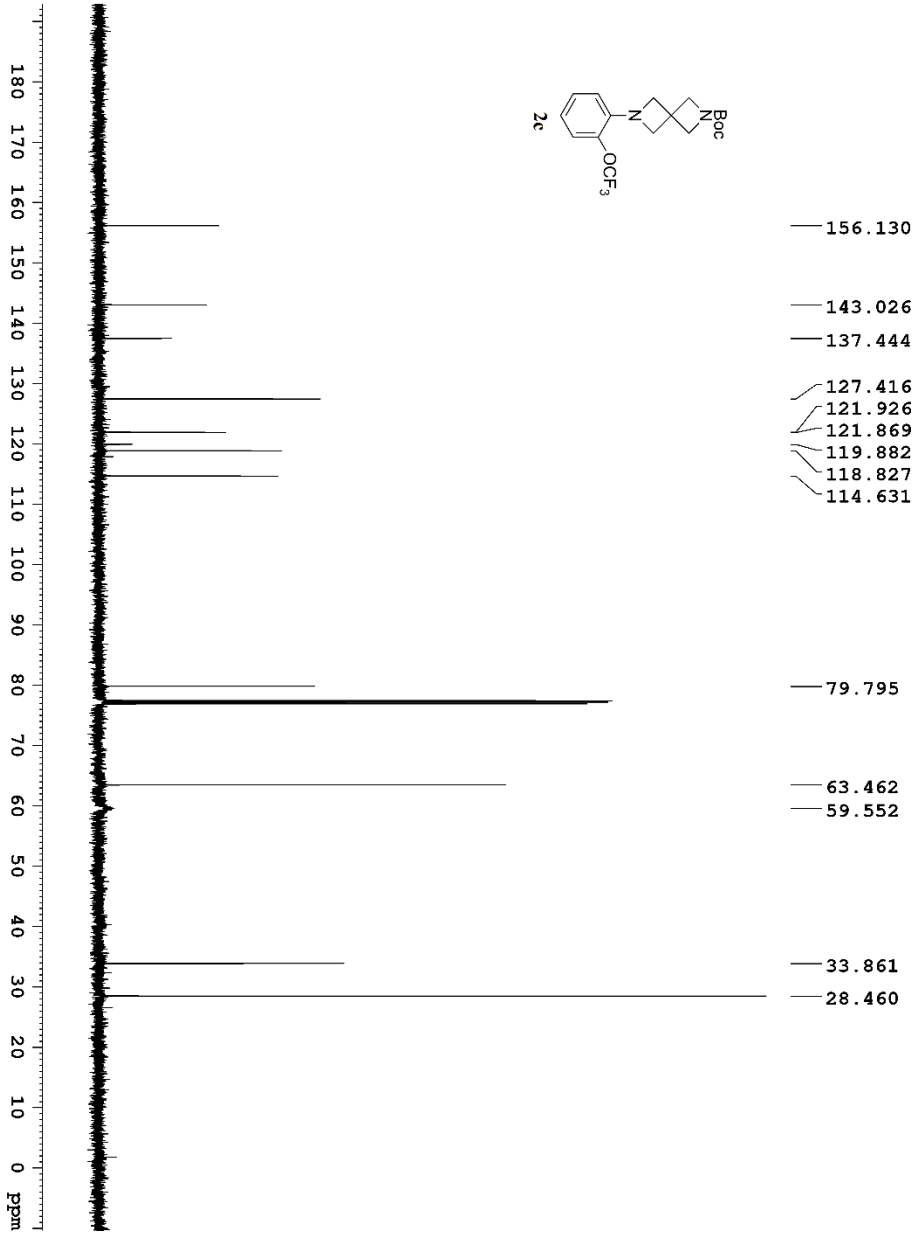
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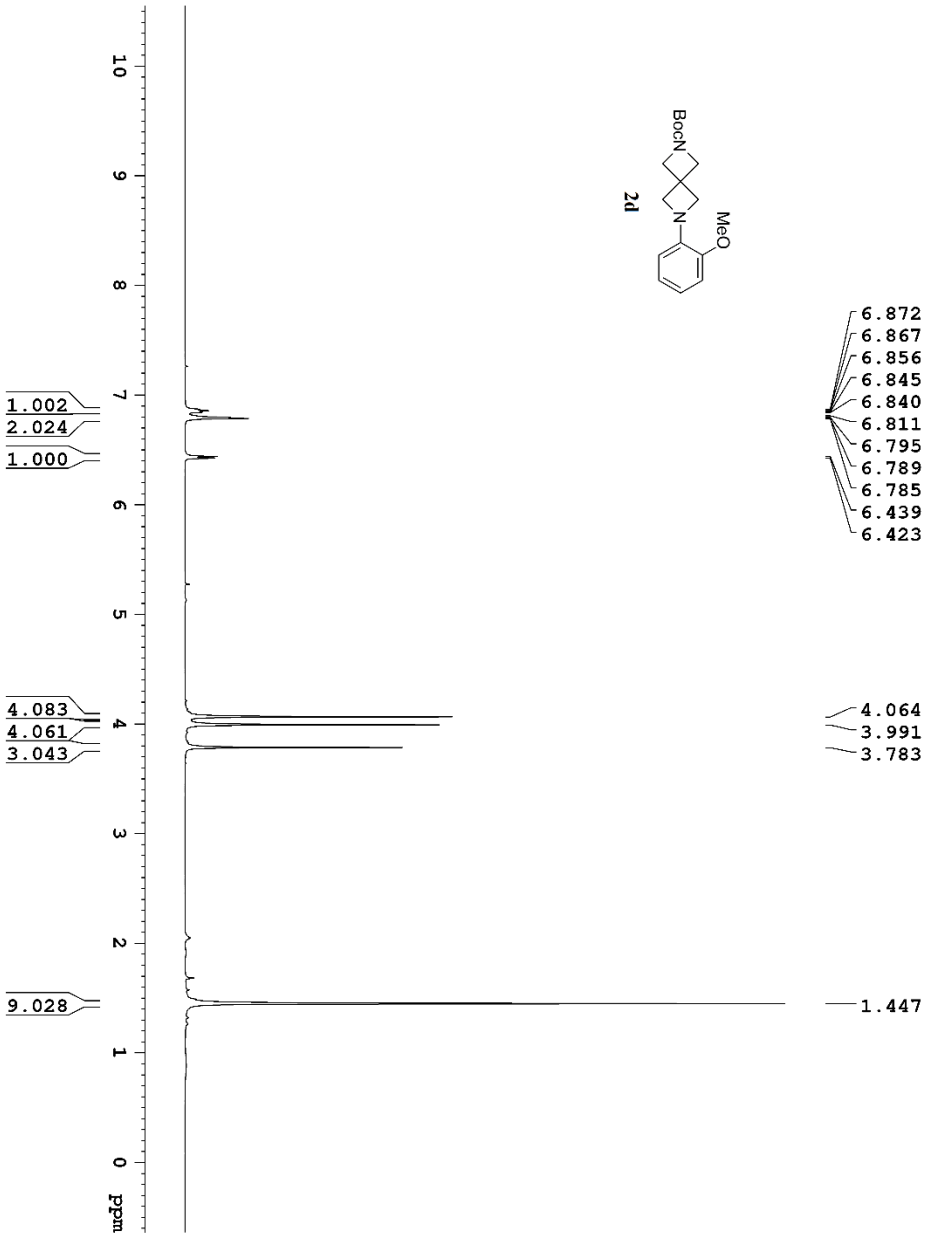
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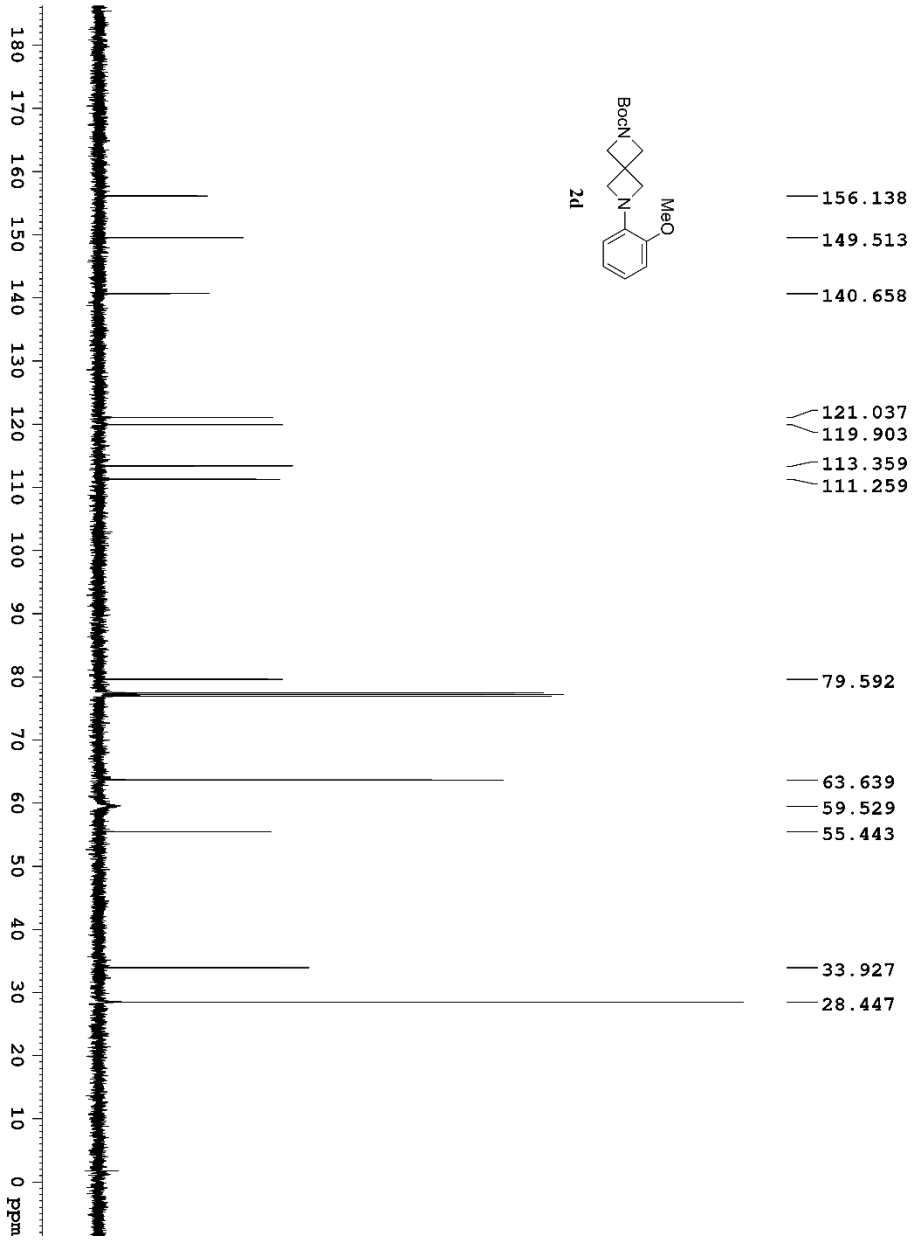
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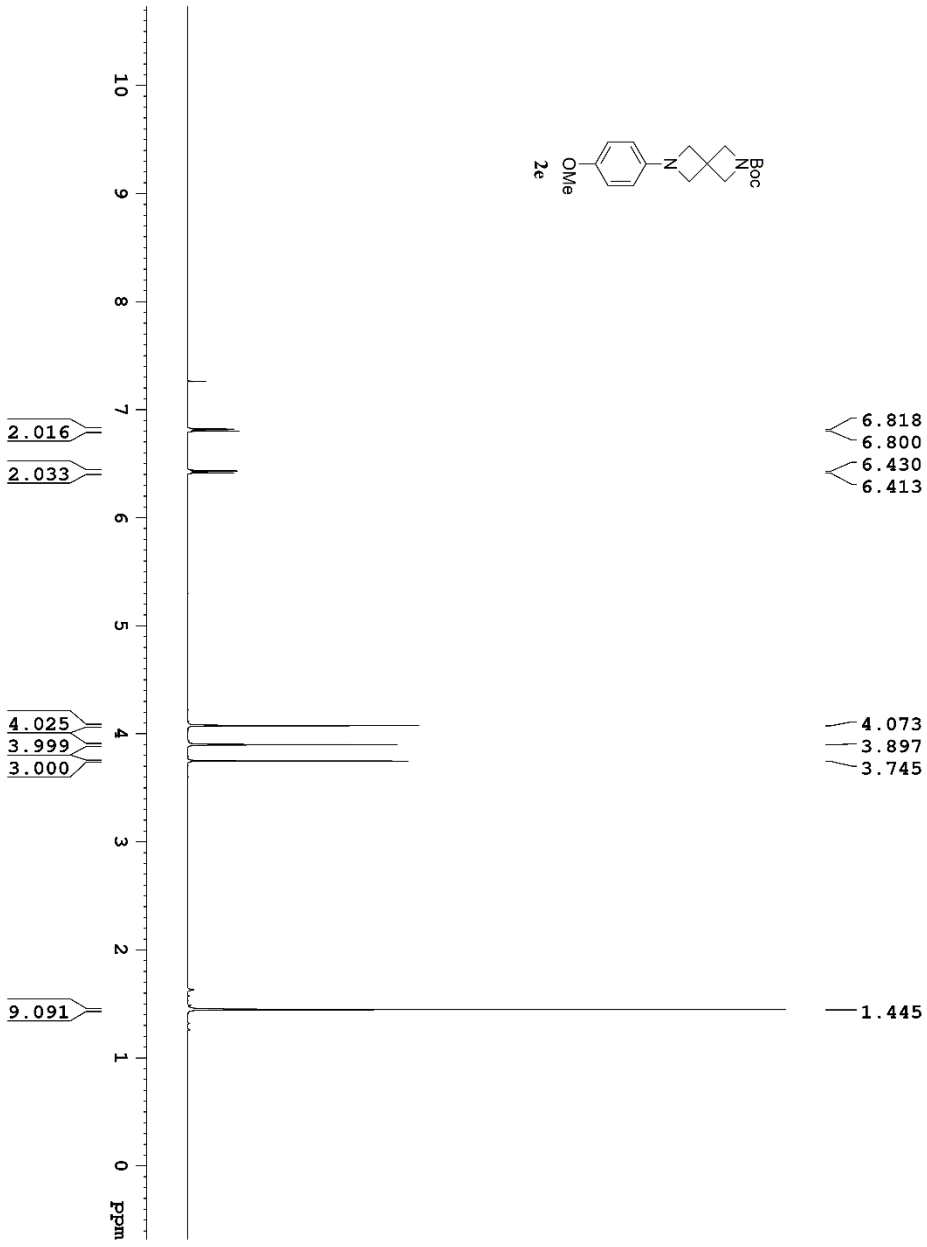
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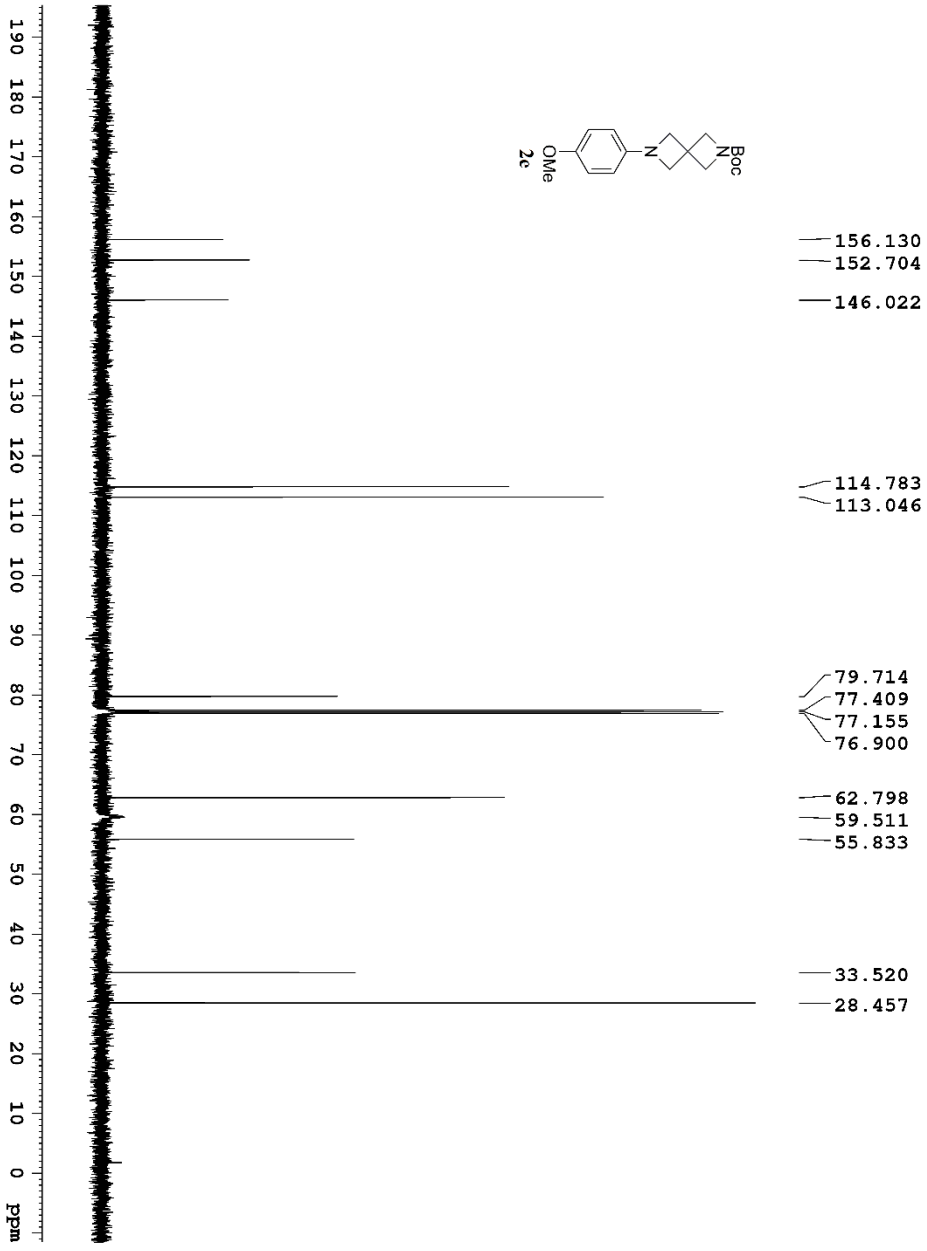
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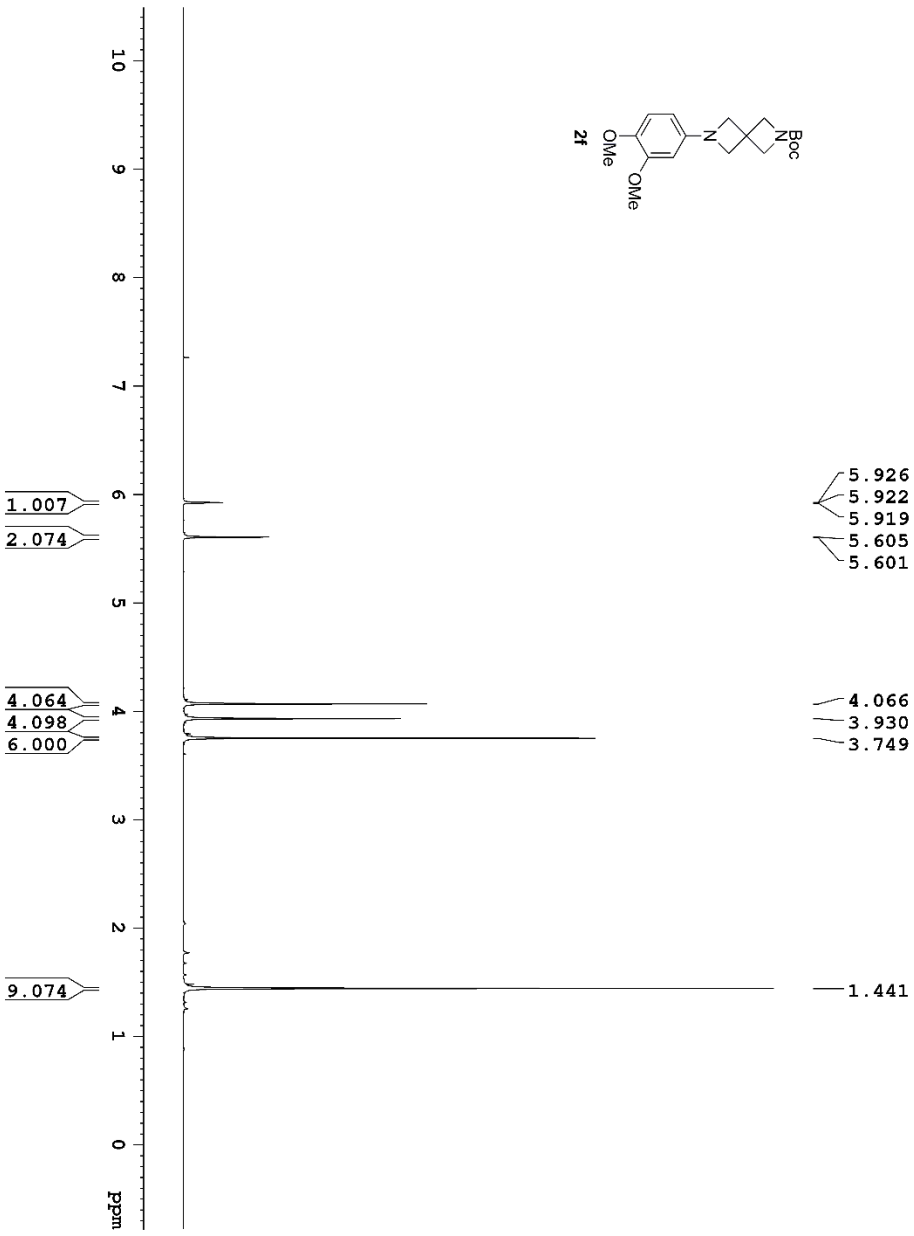
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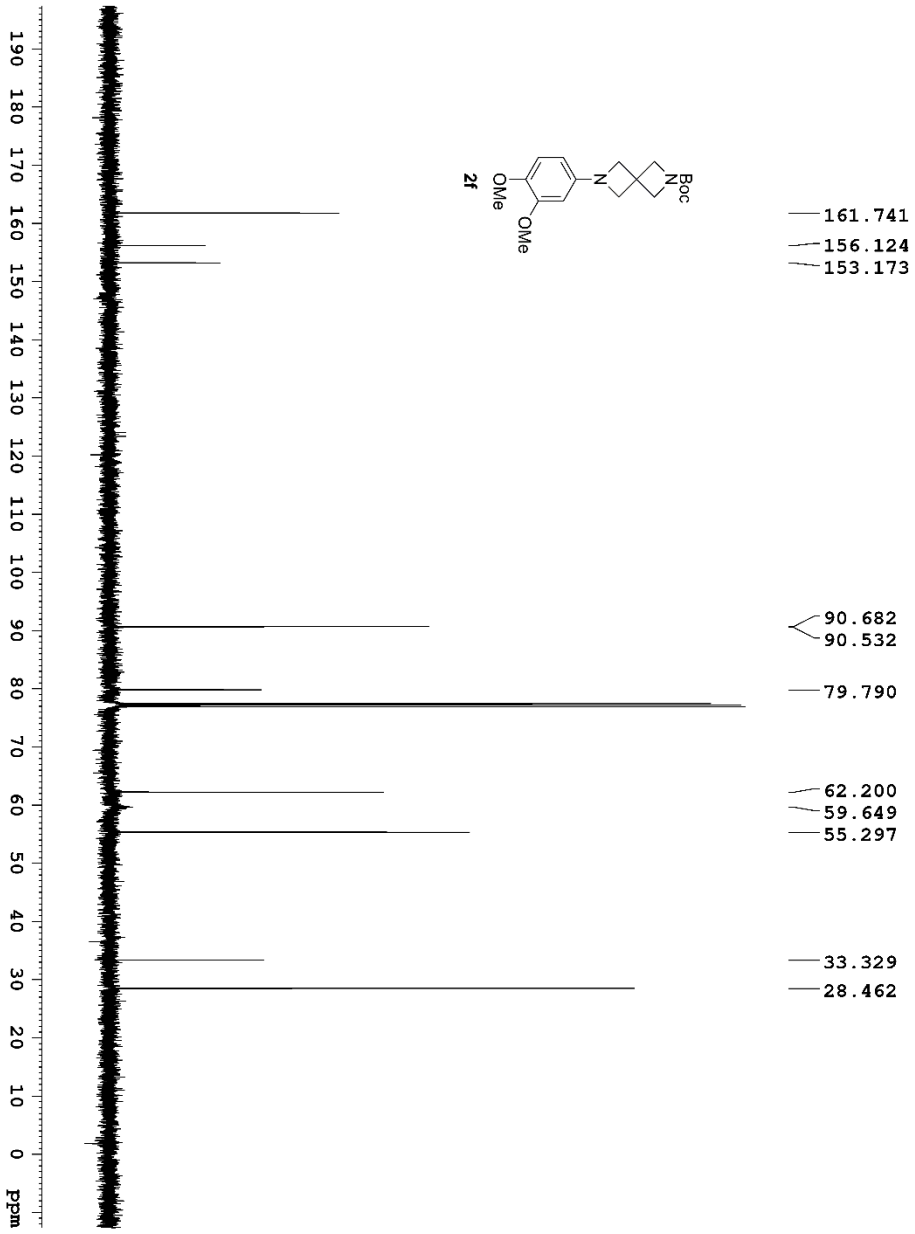
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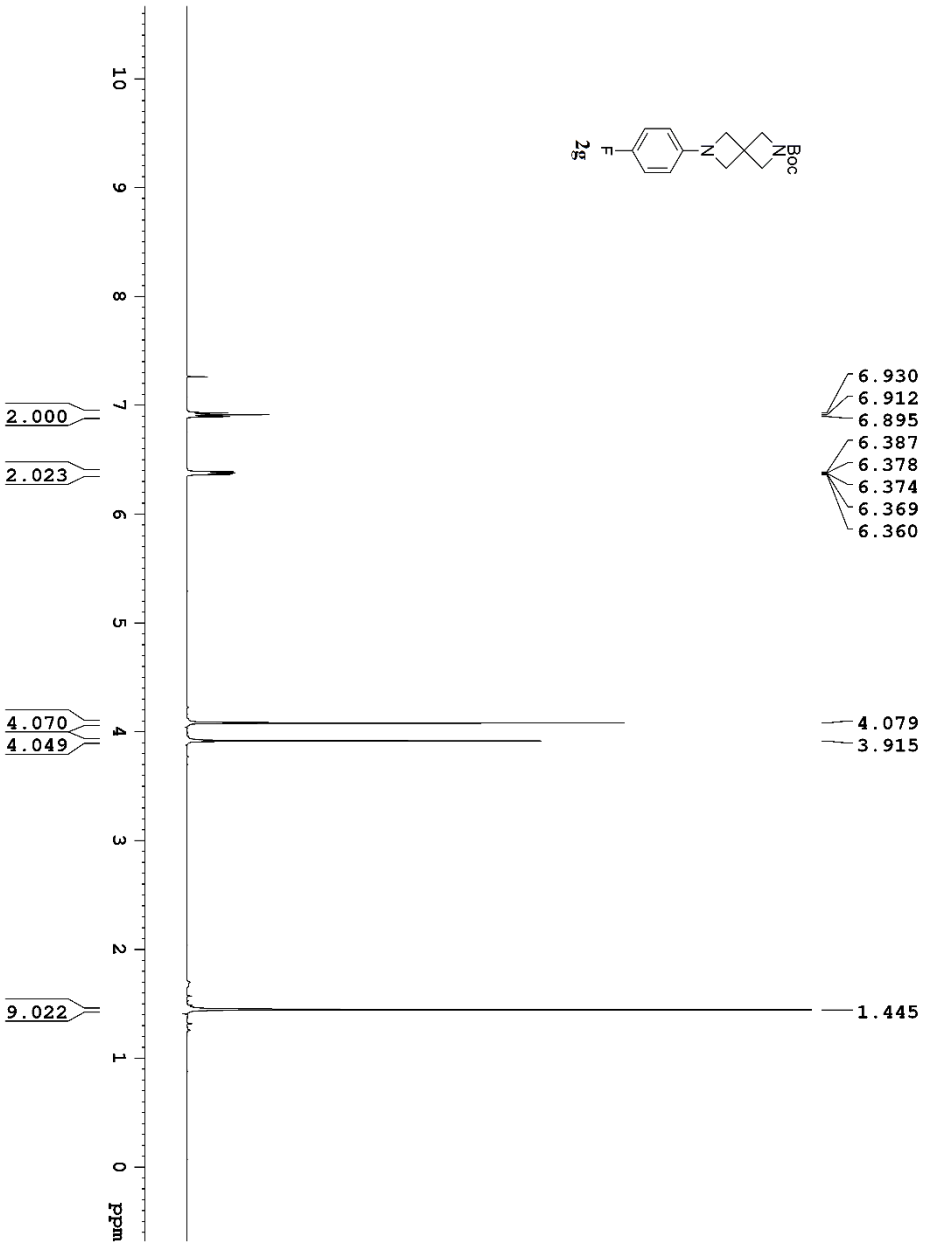
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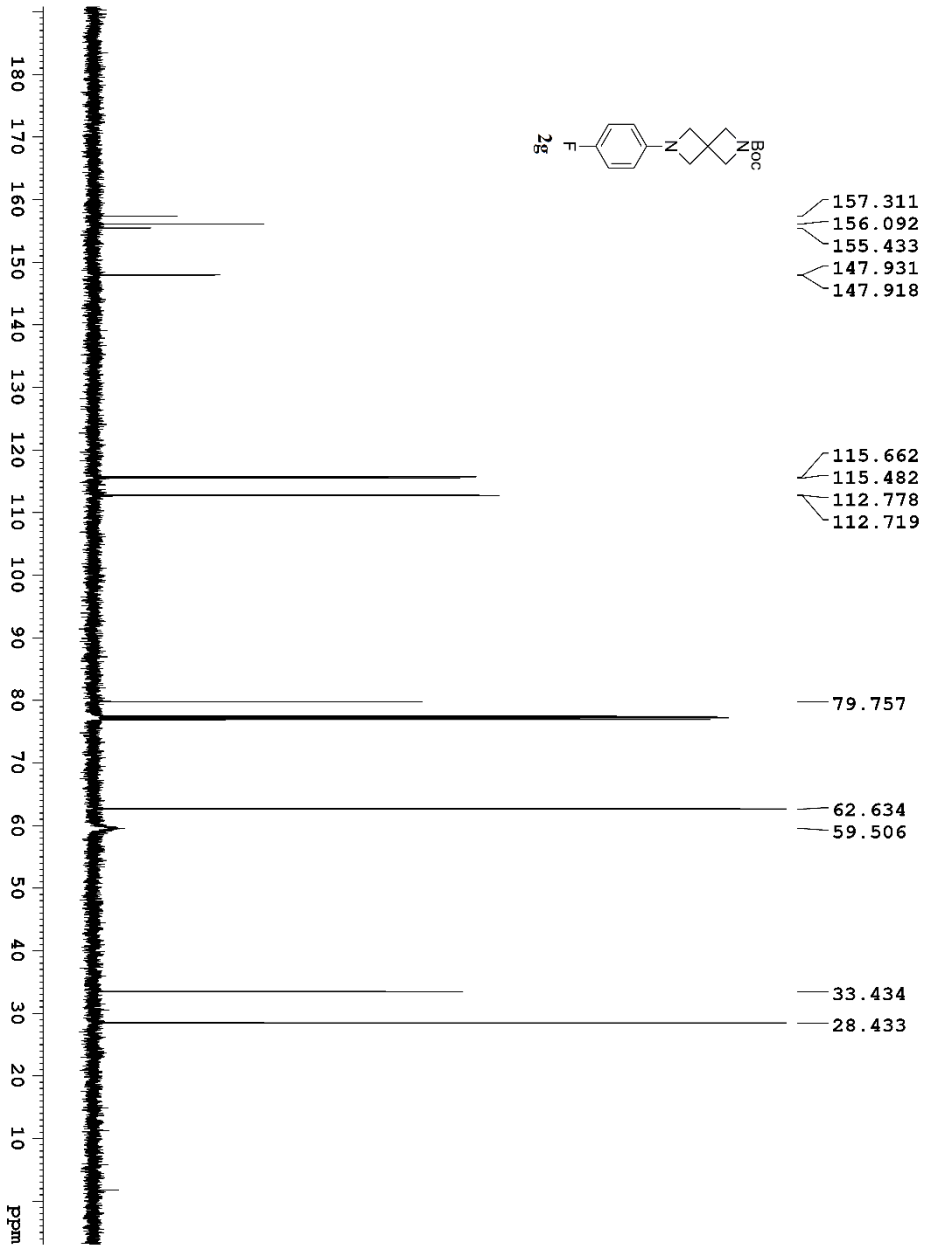
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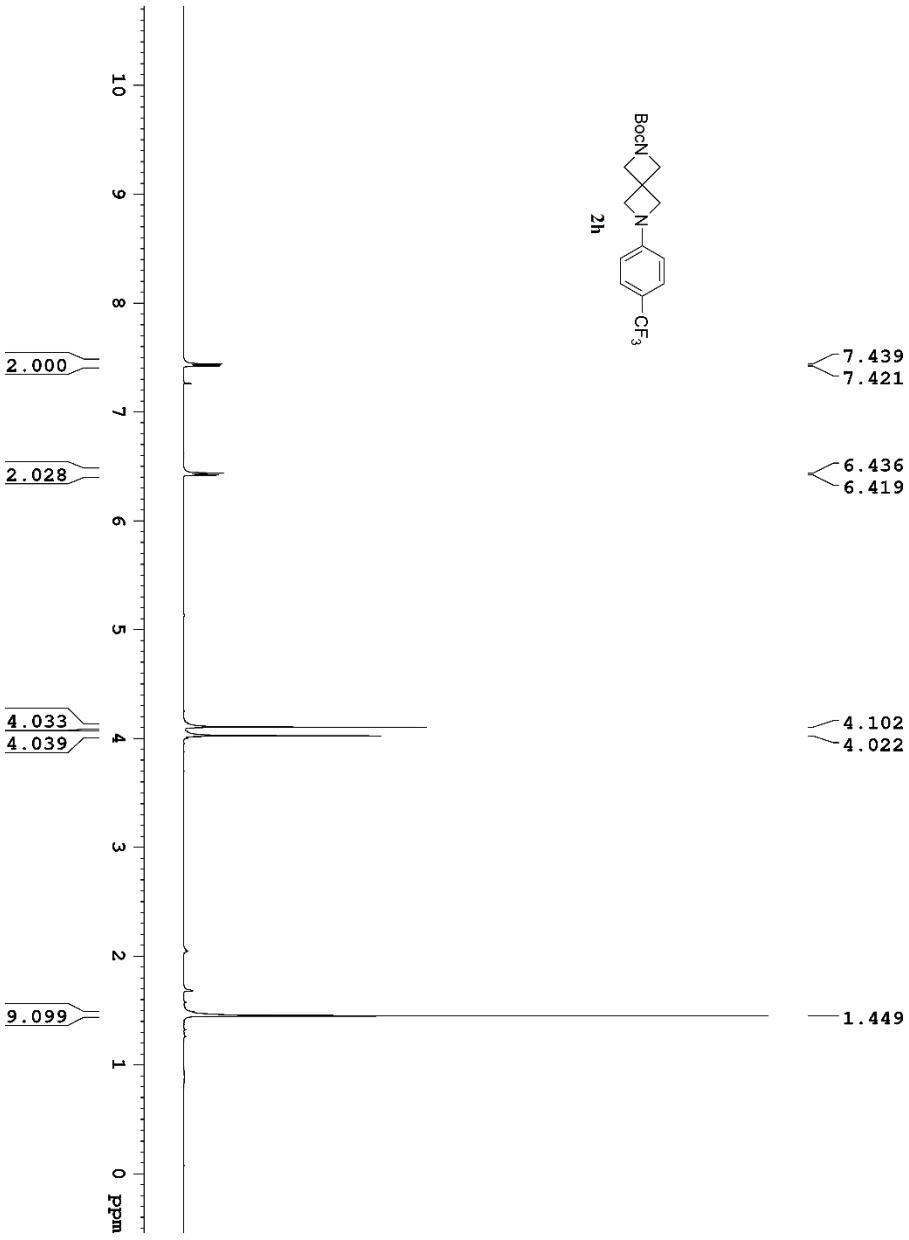
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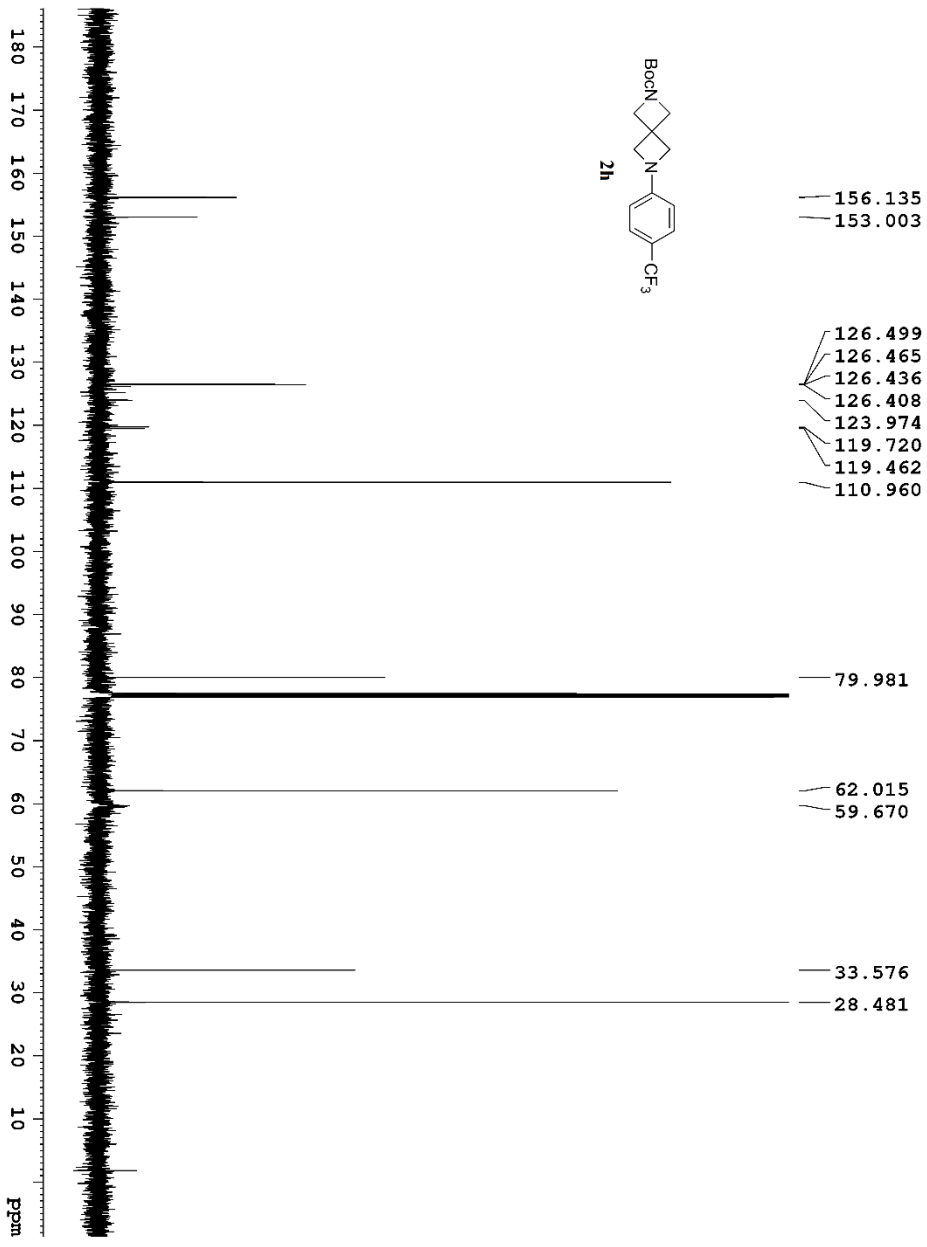
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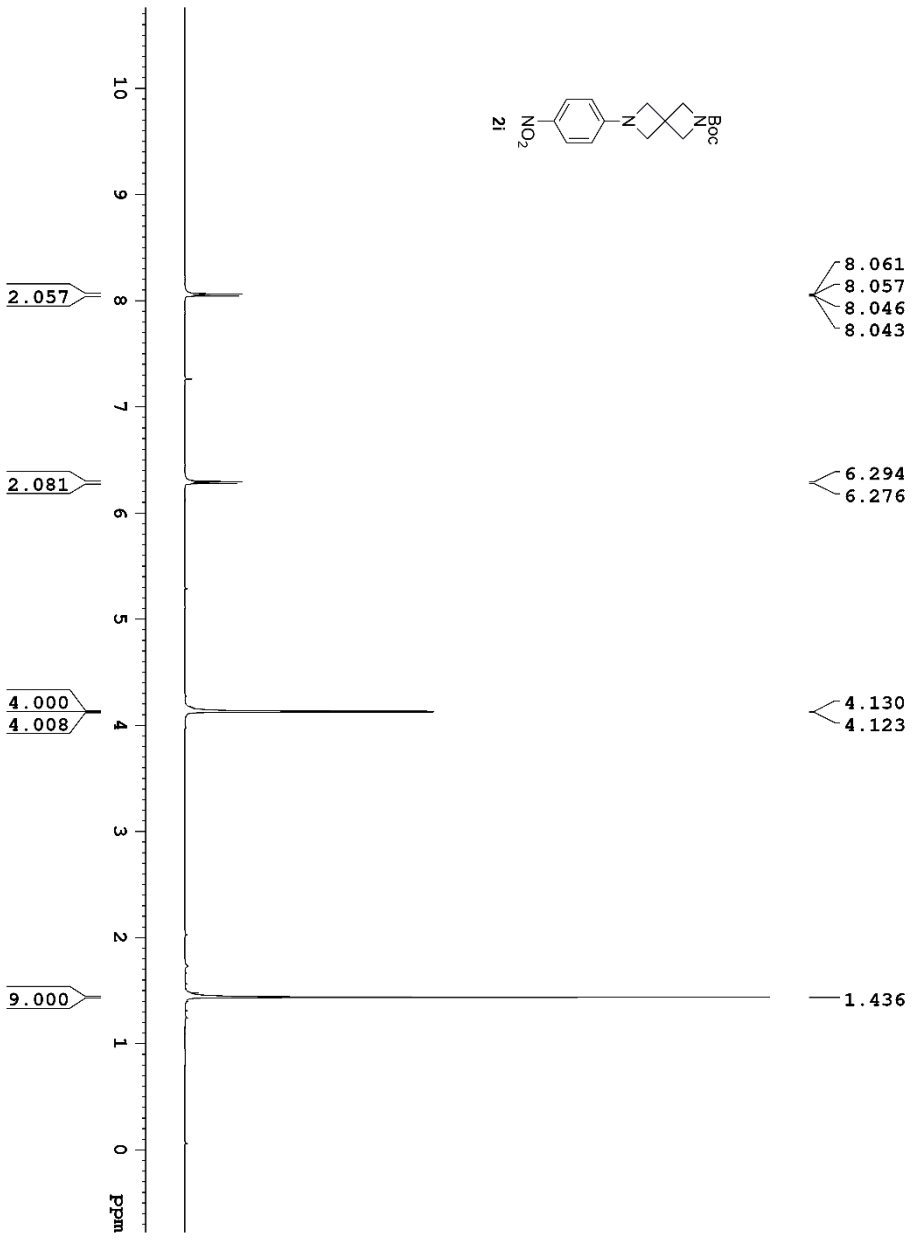
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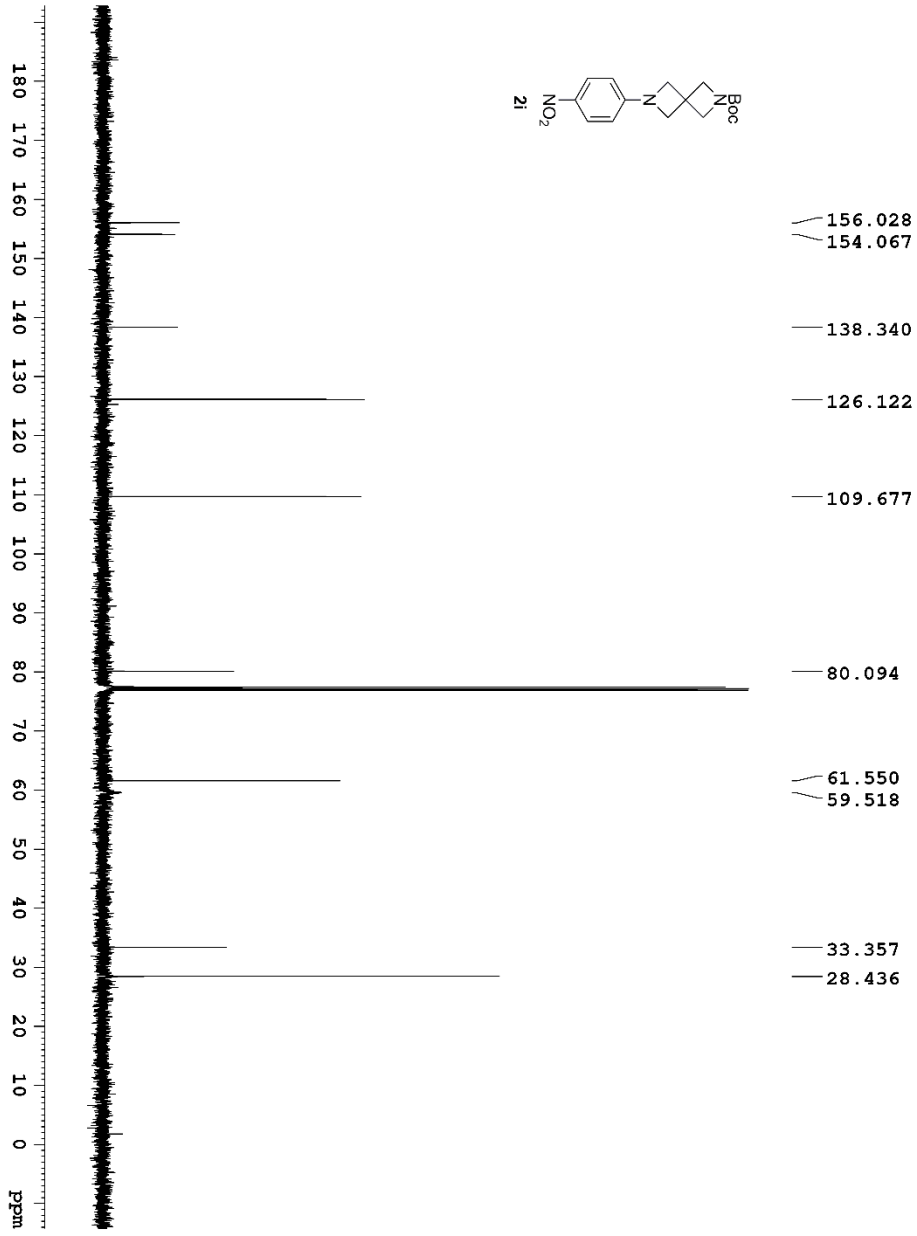
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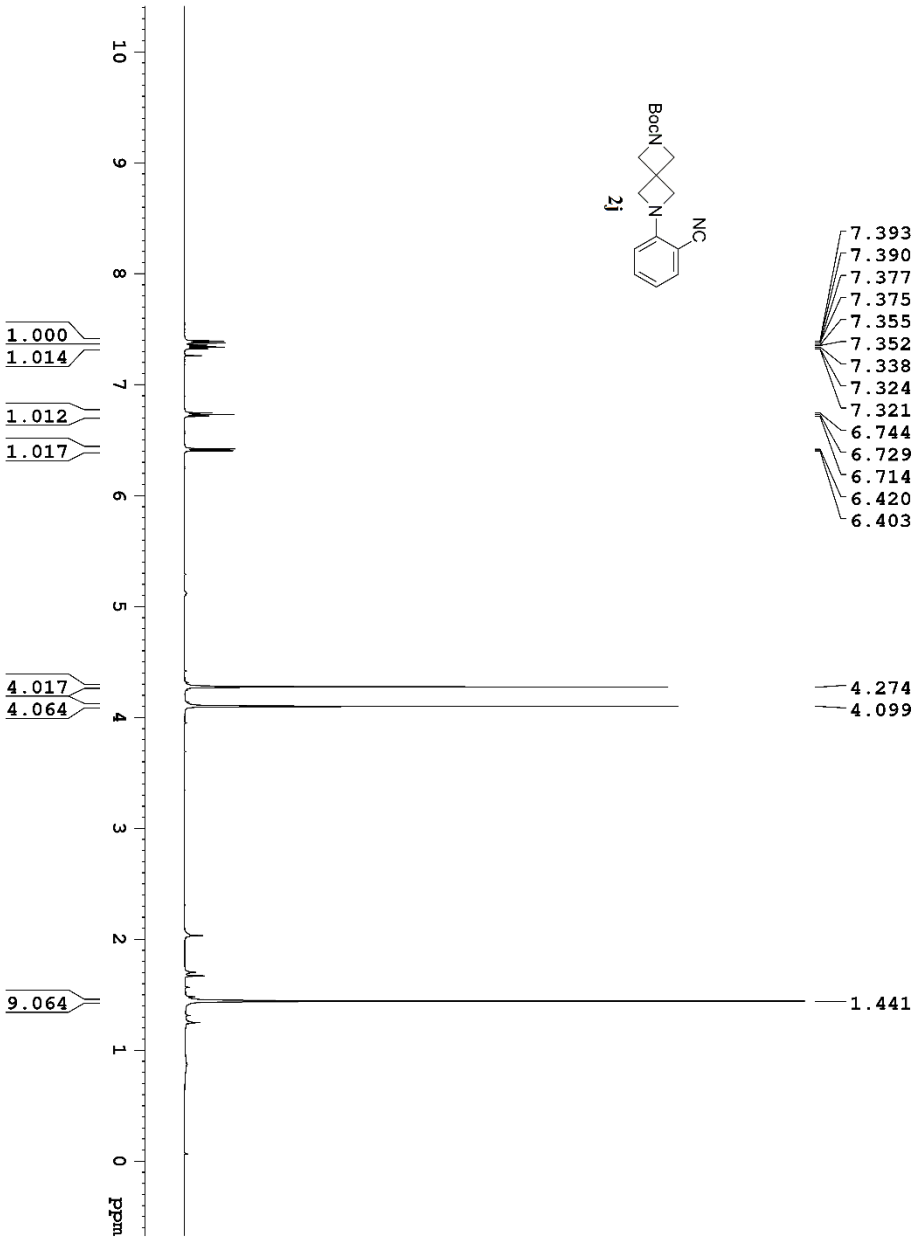
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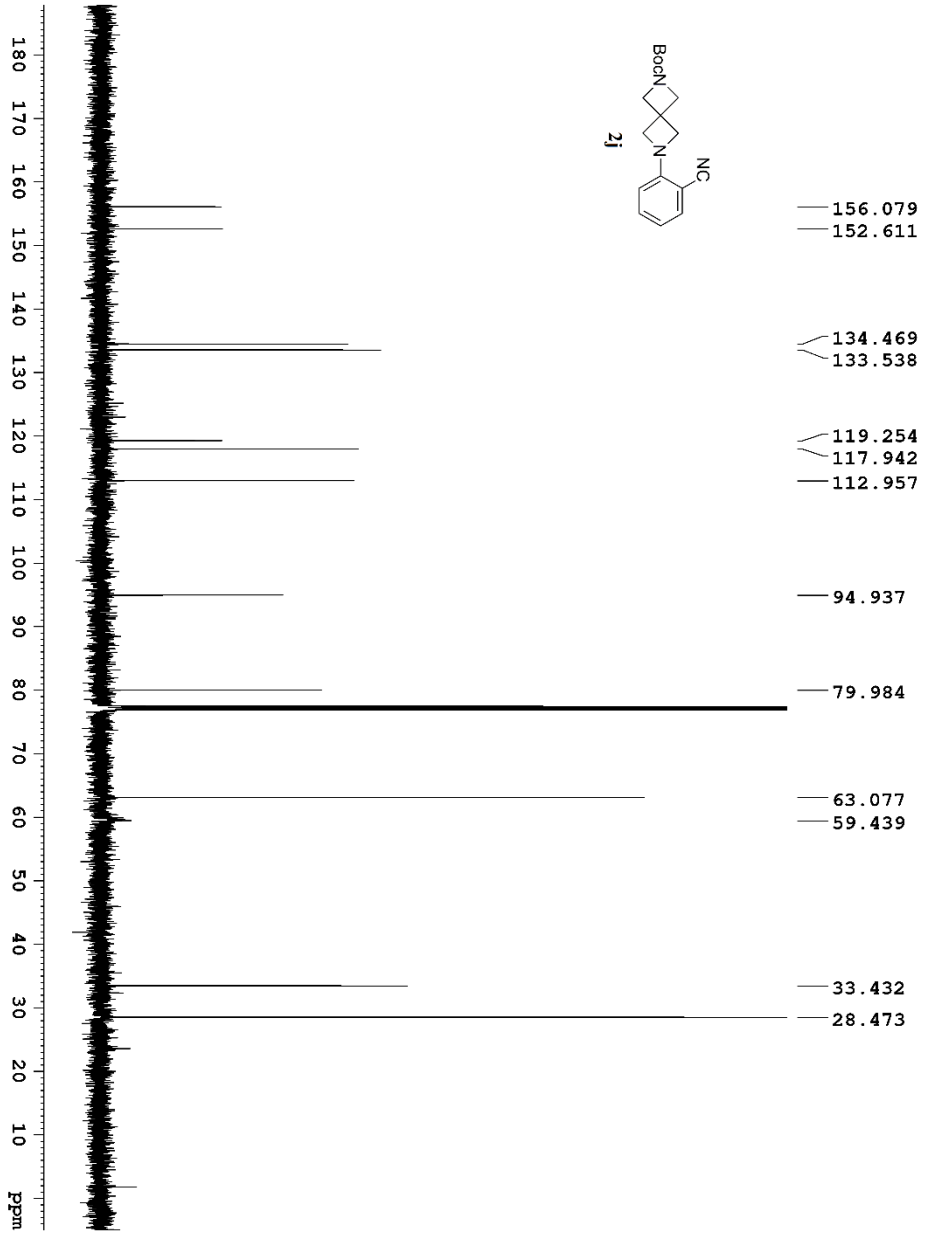
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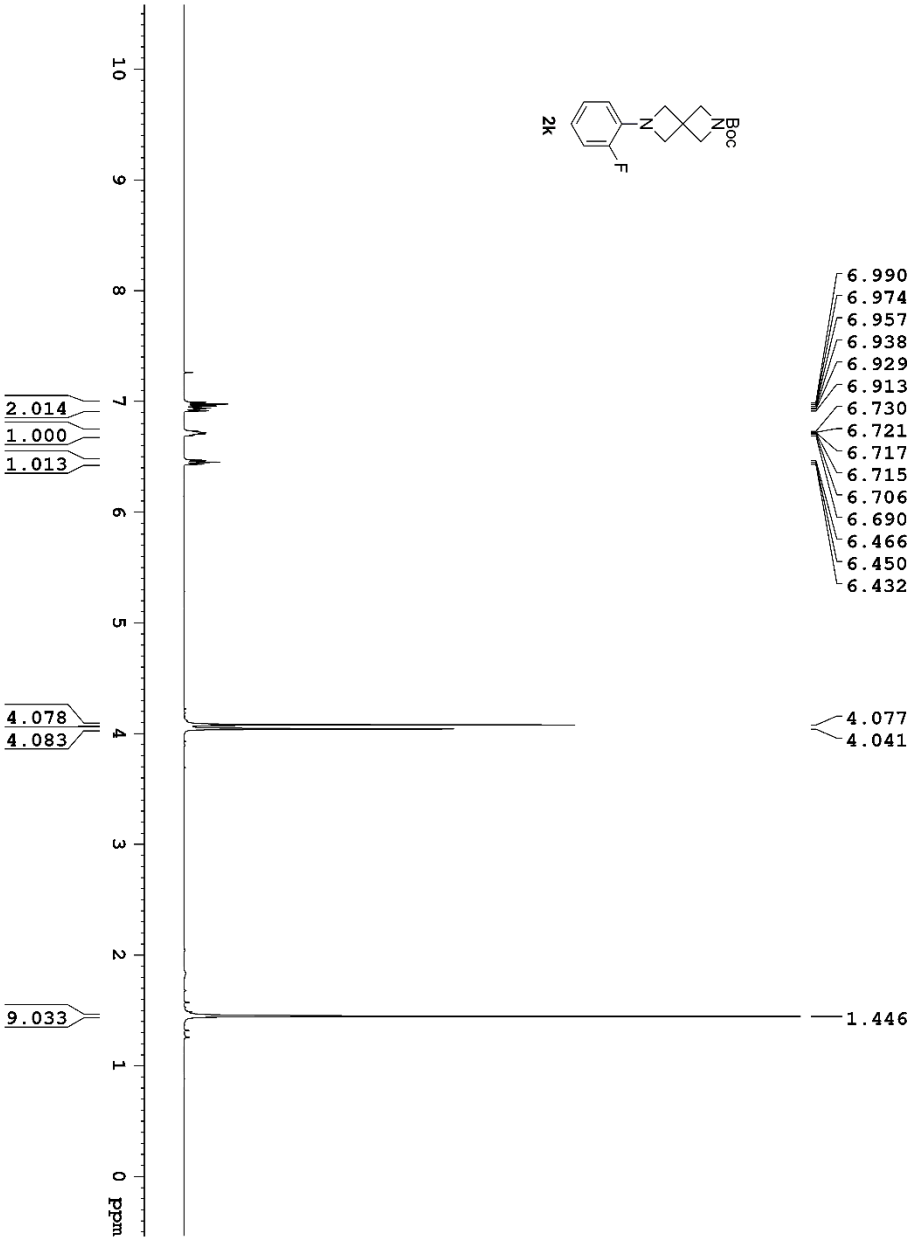
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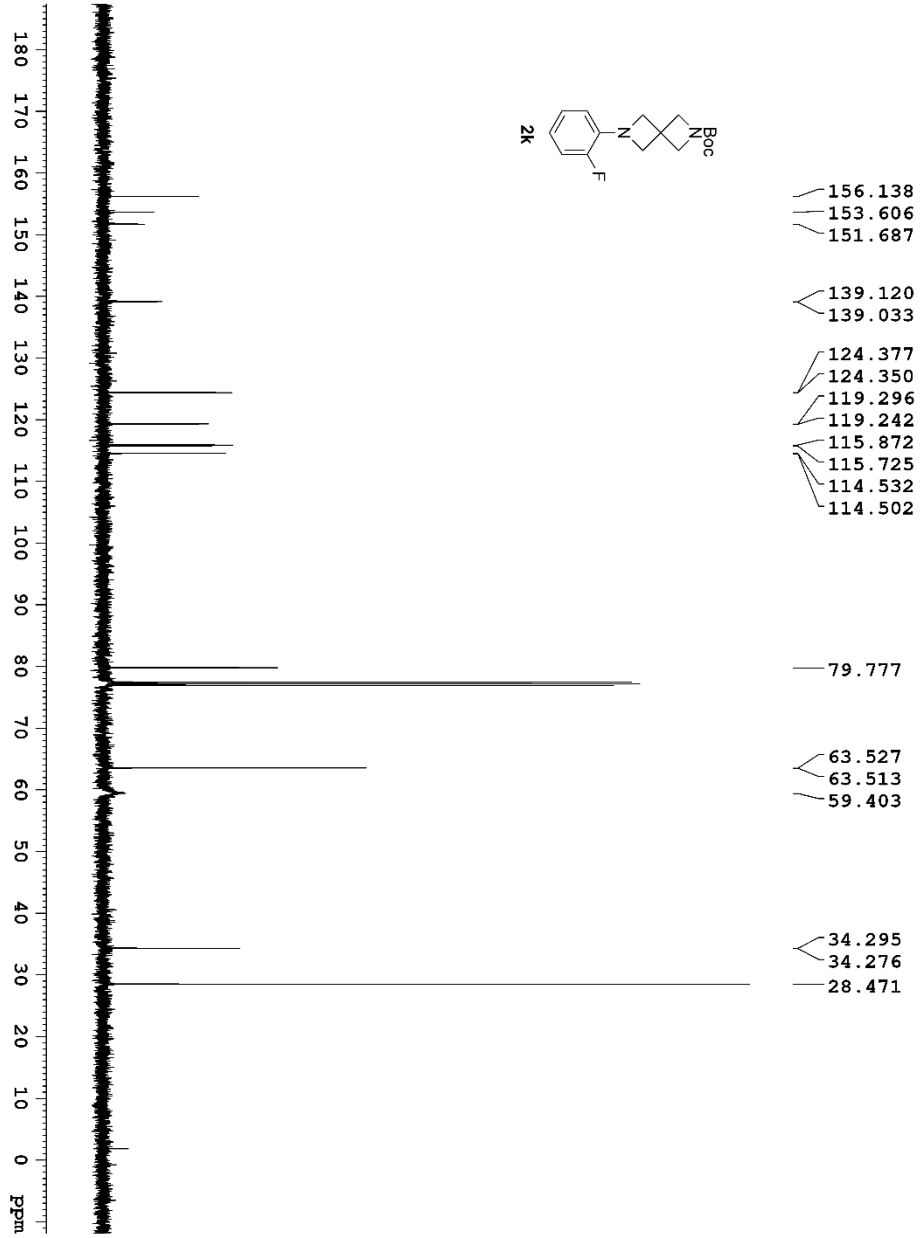
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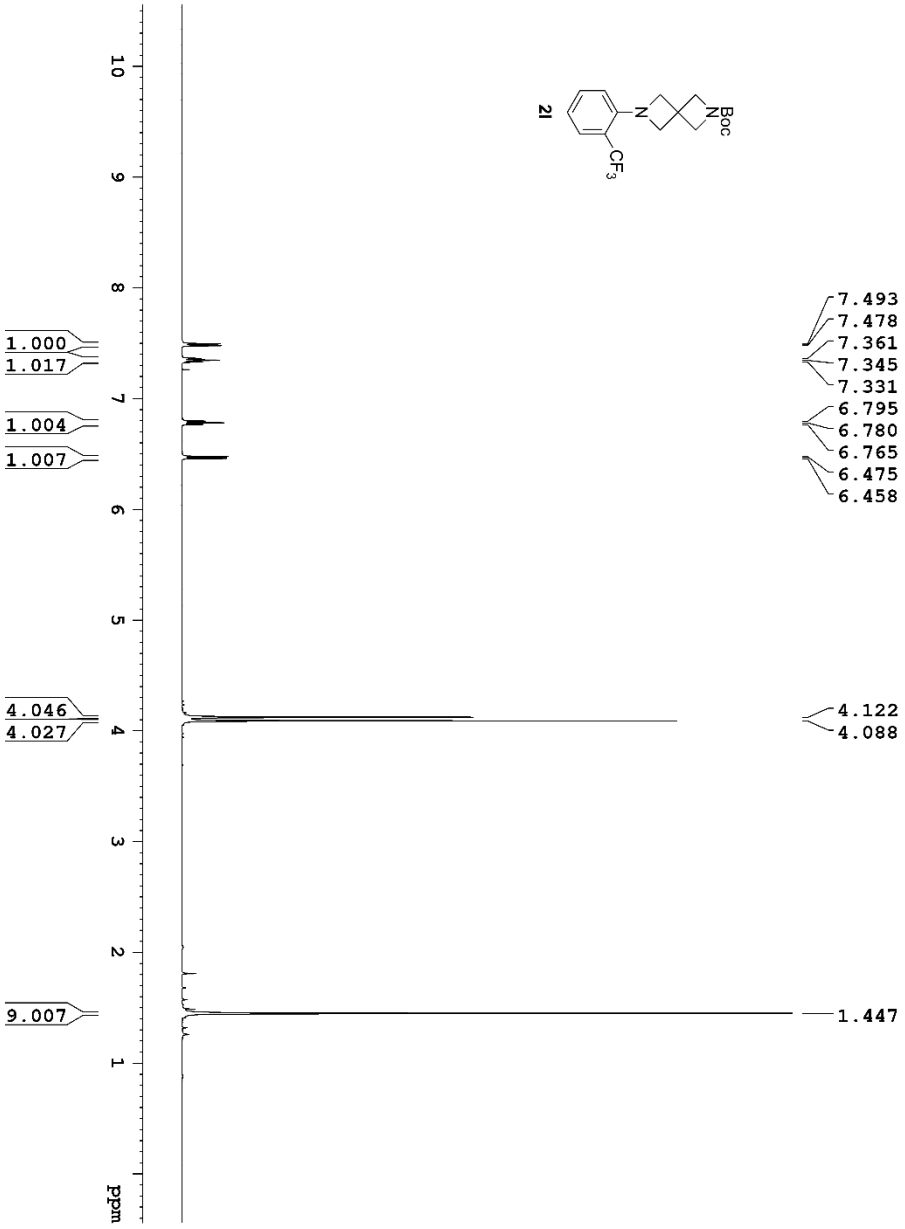
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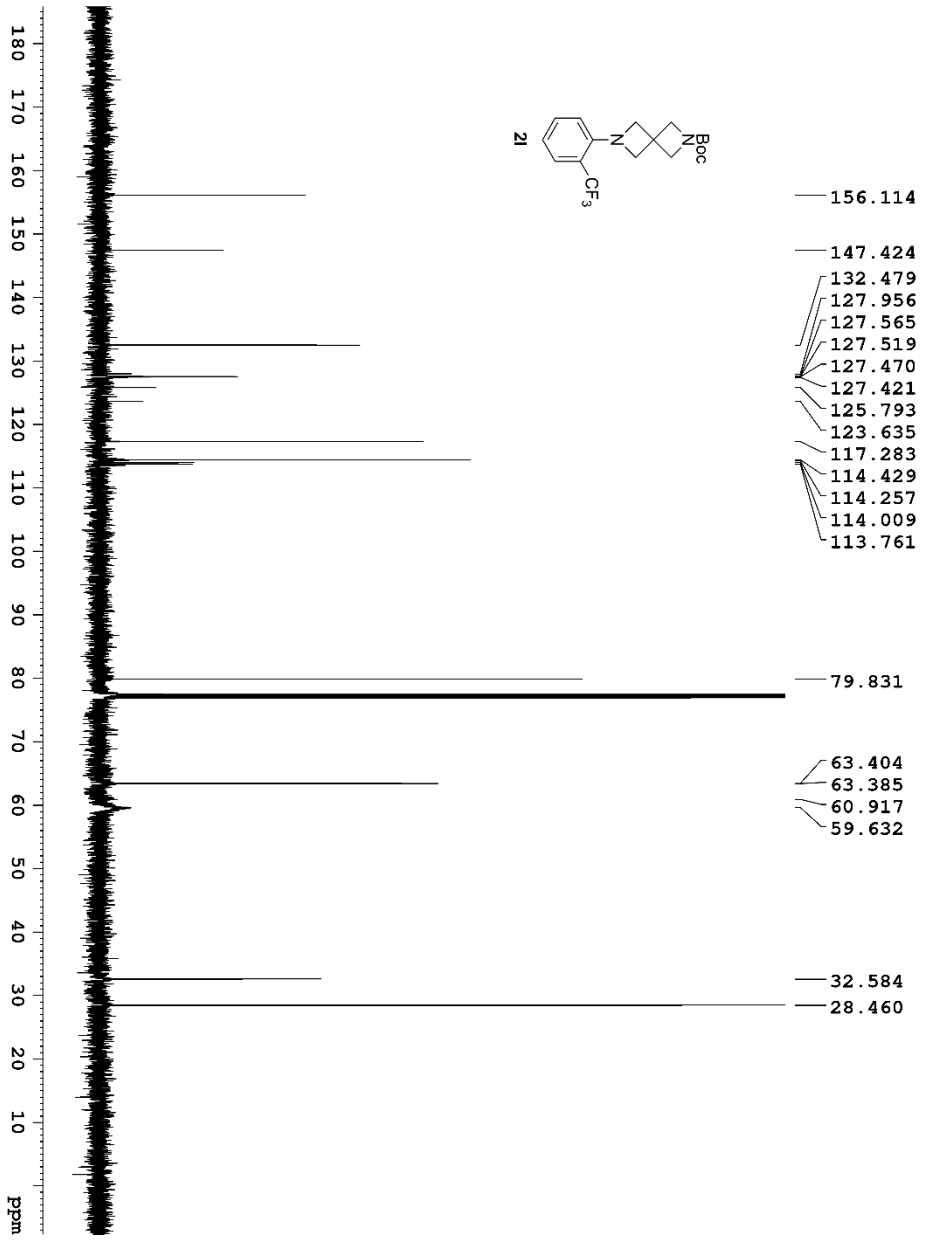
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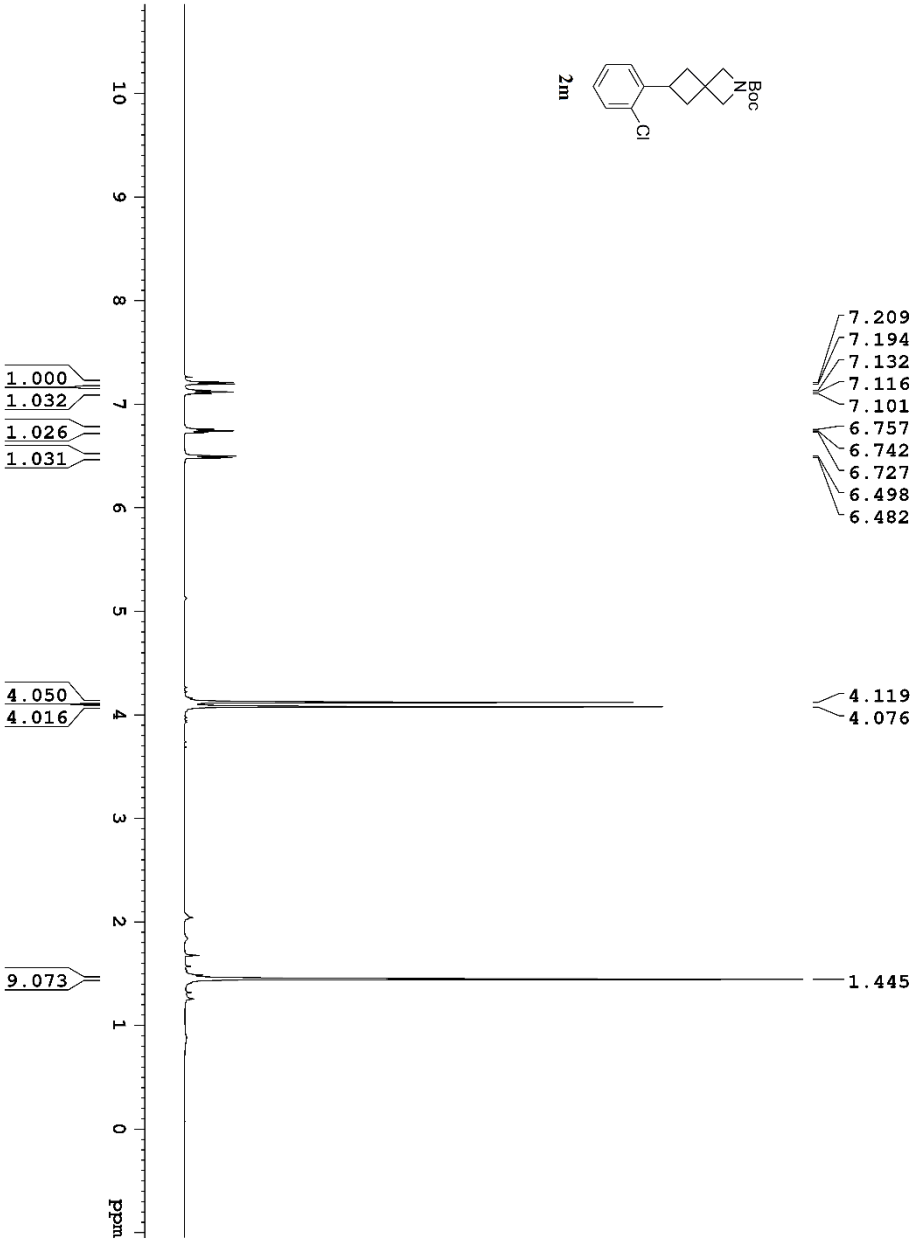
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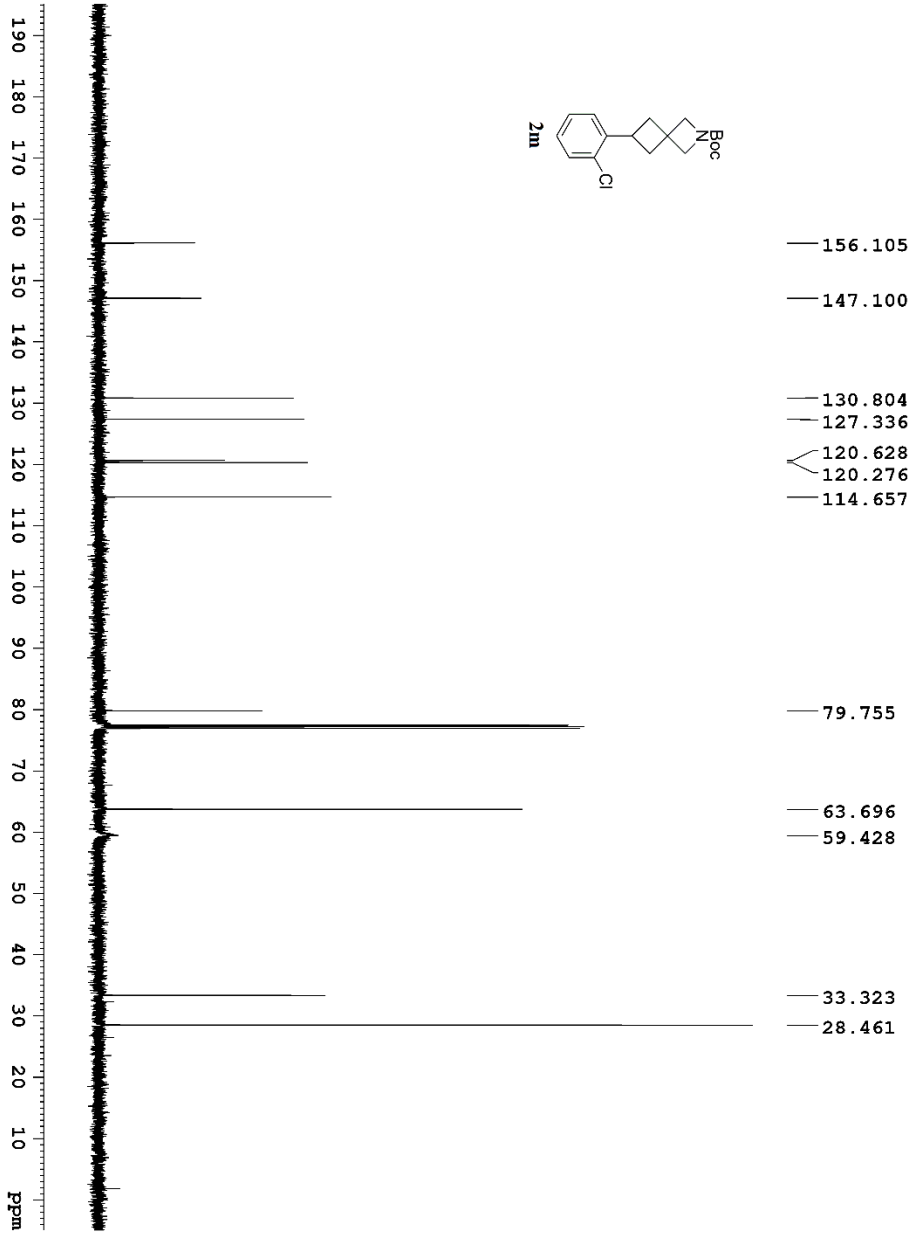
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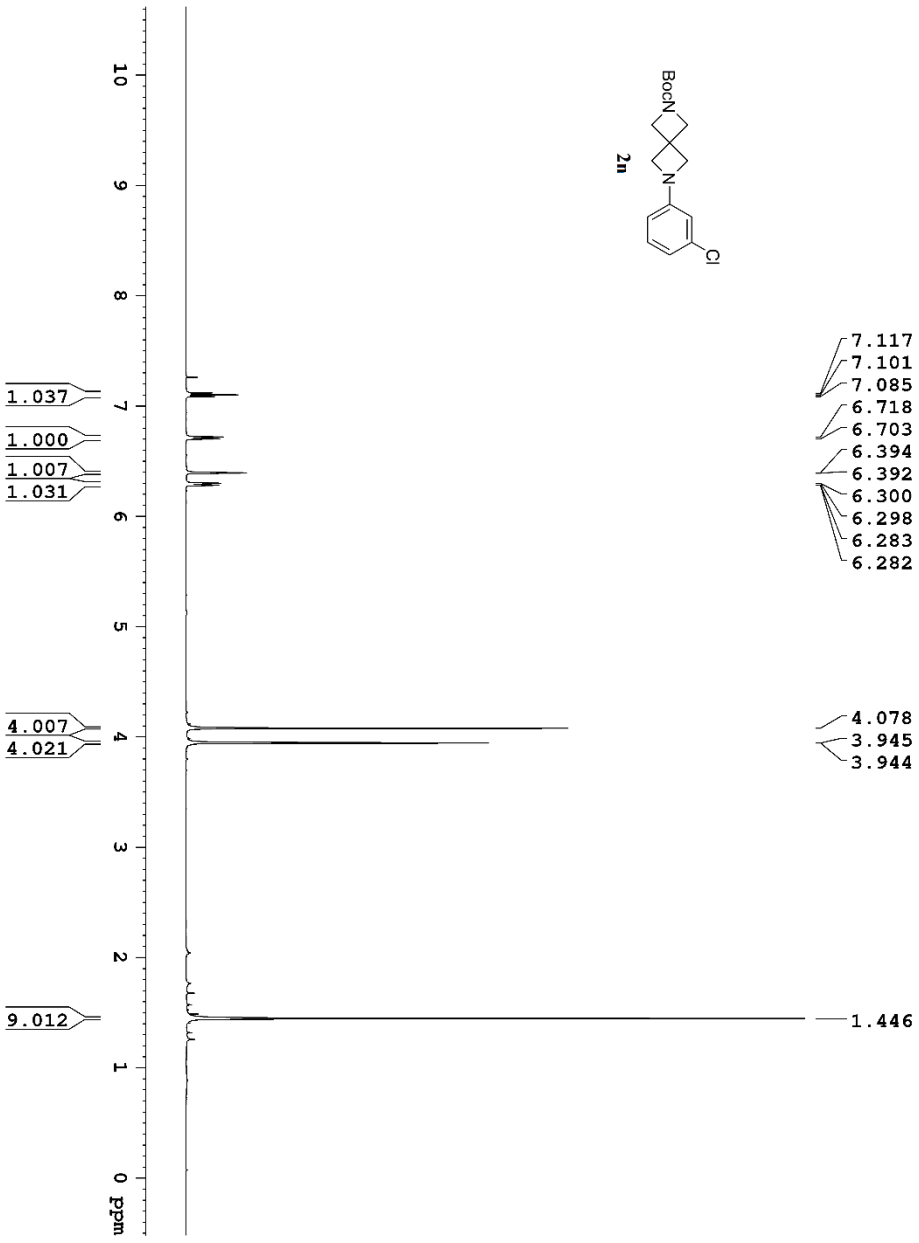
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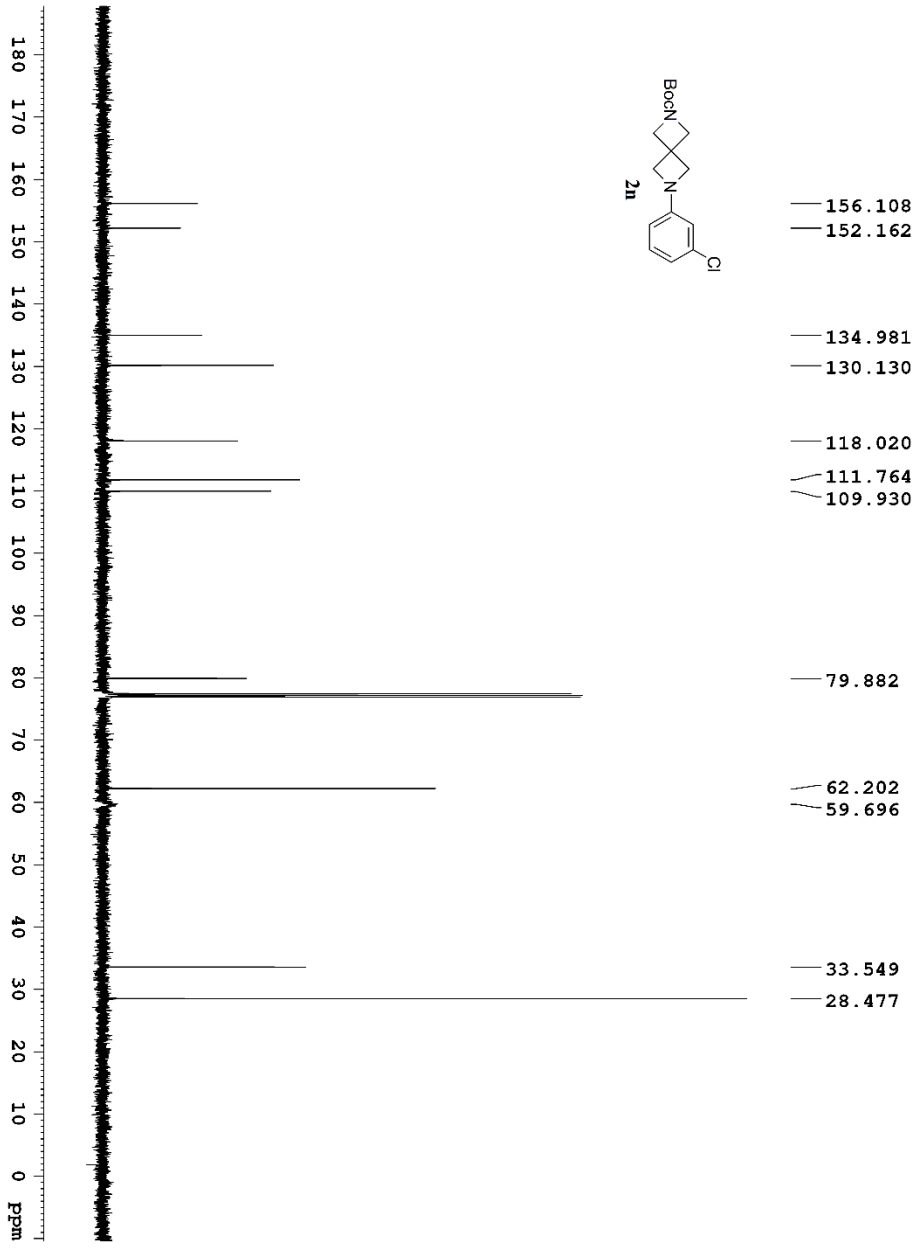
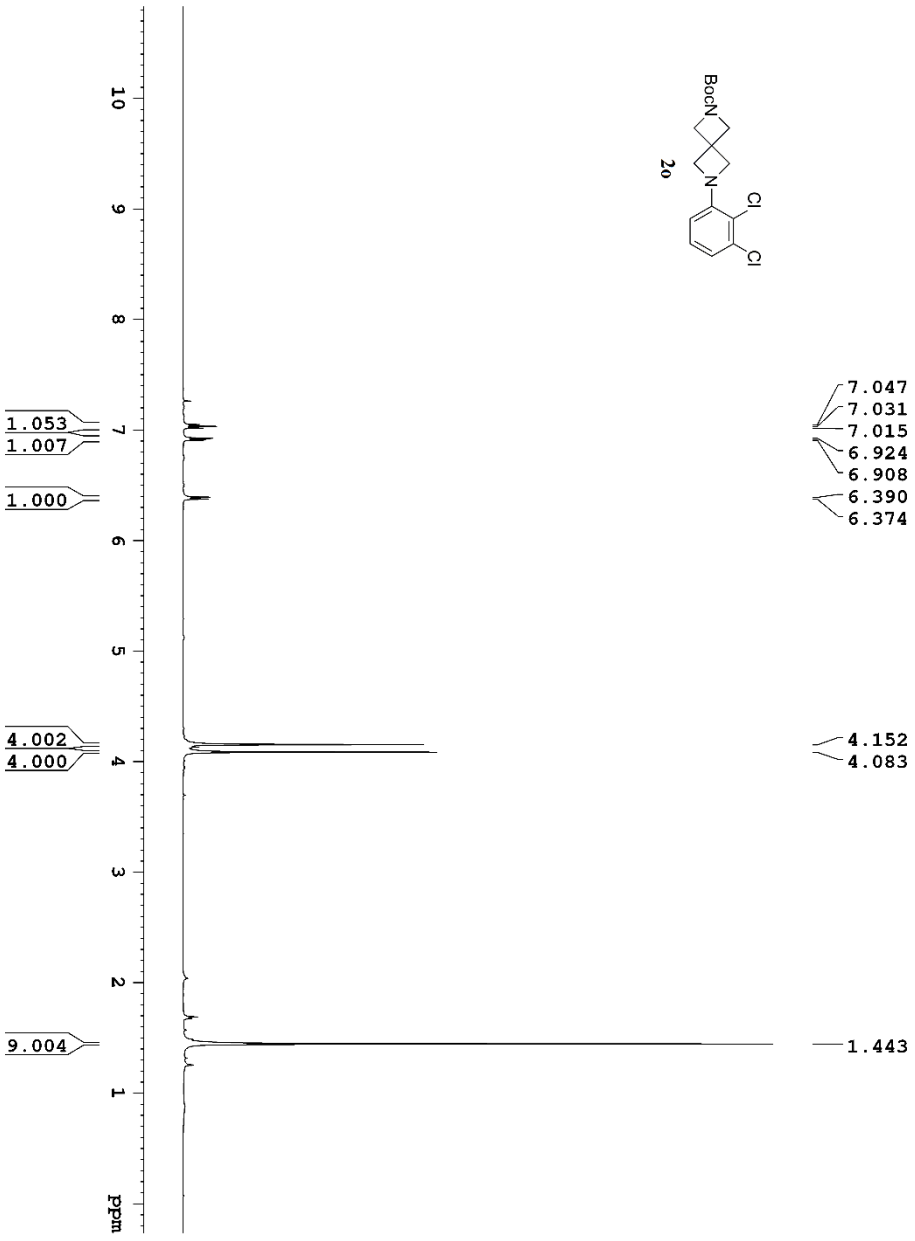
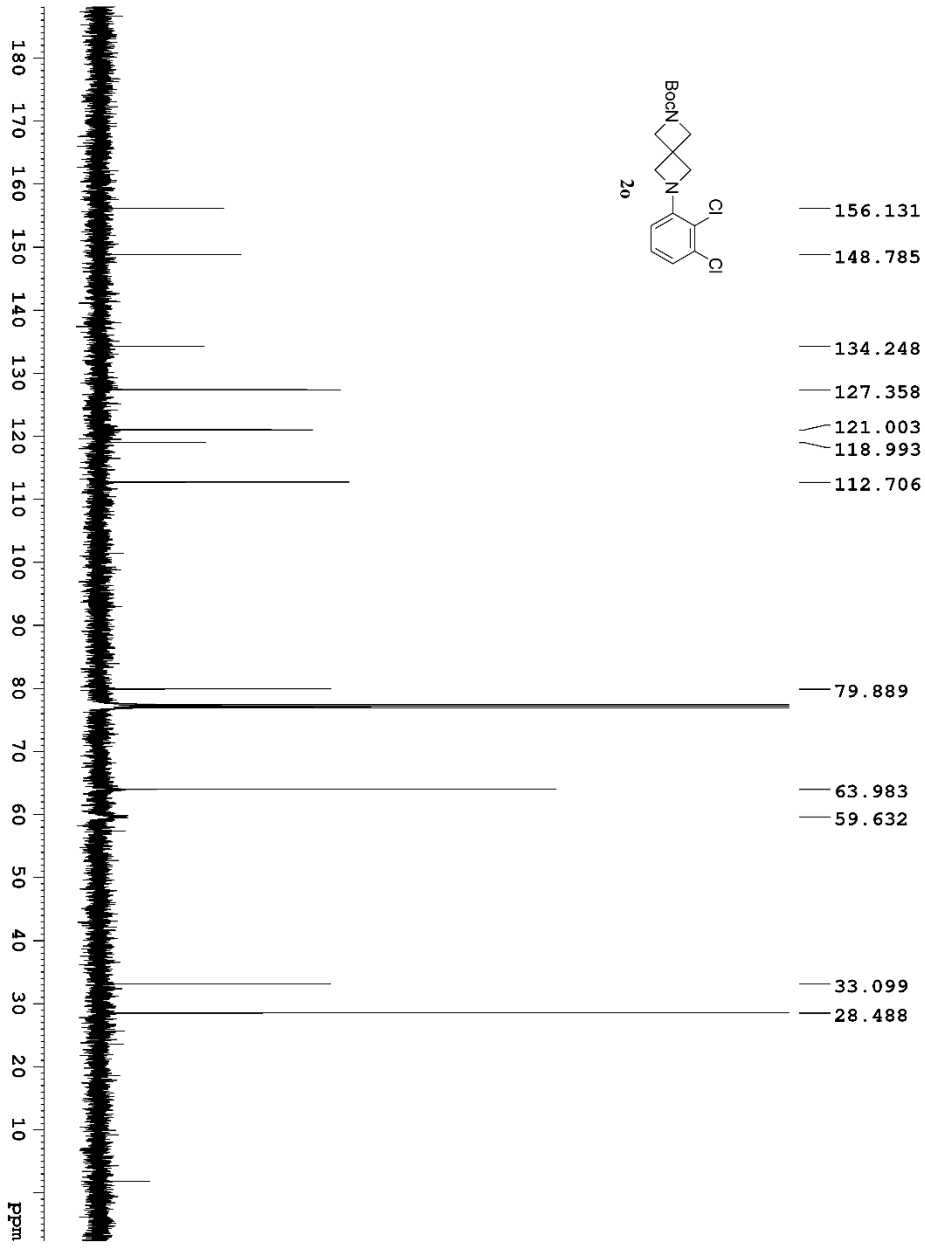
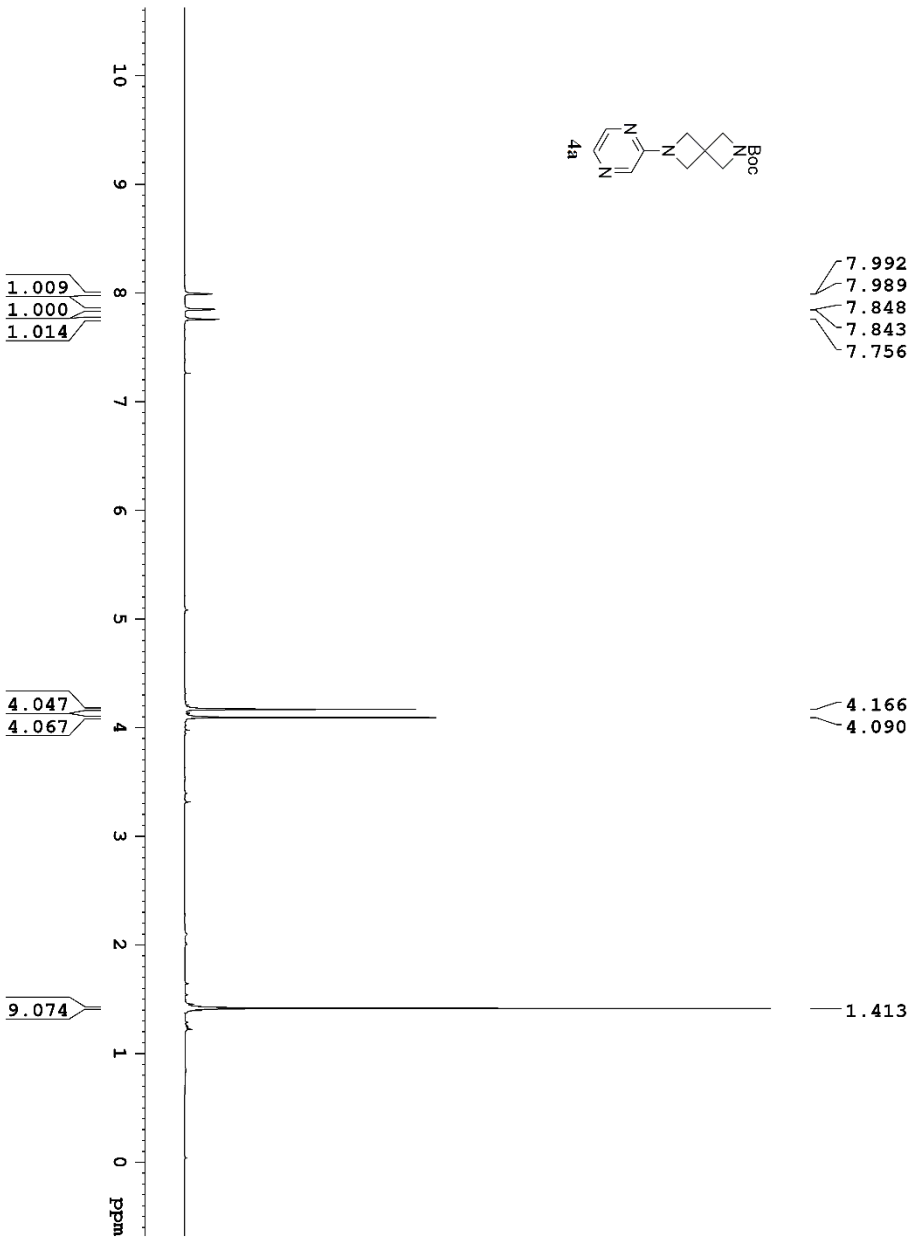
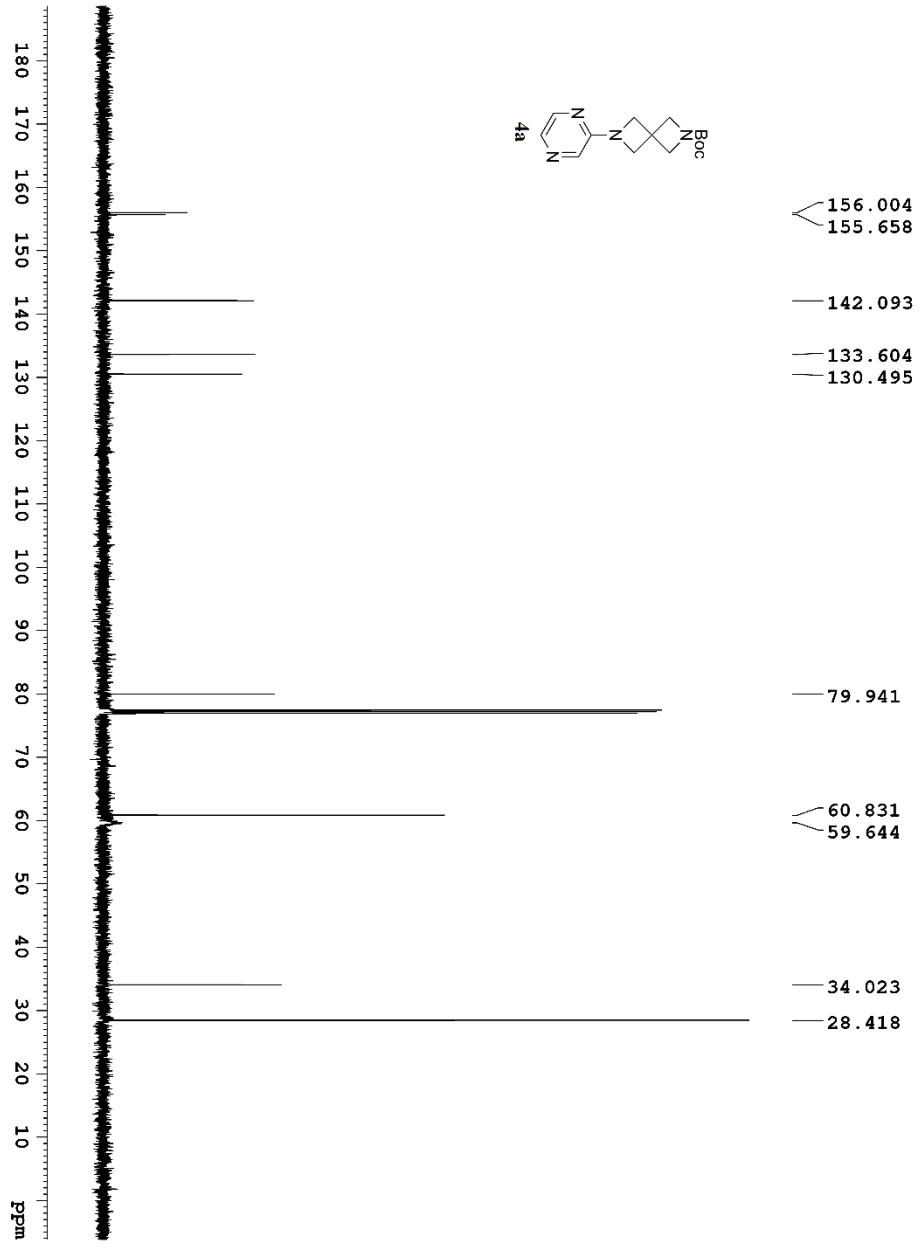
1.000
1.007
1.031
4.007
4.021
9.012
ppm

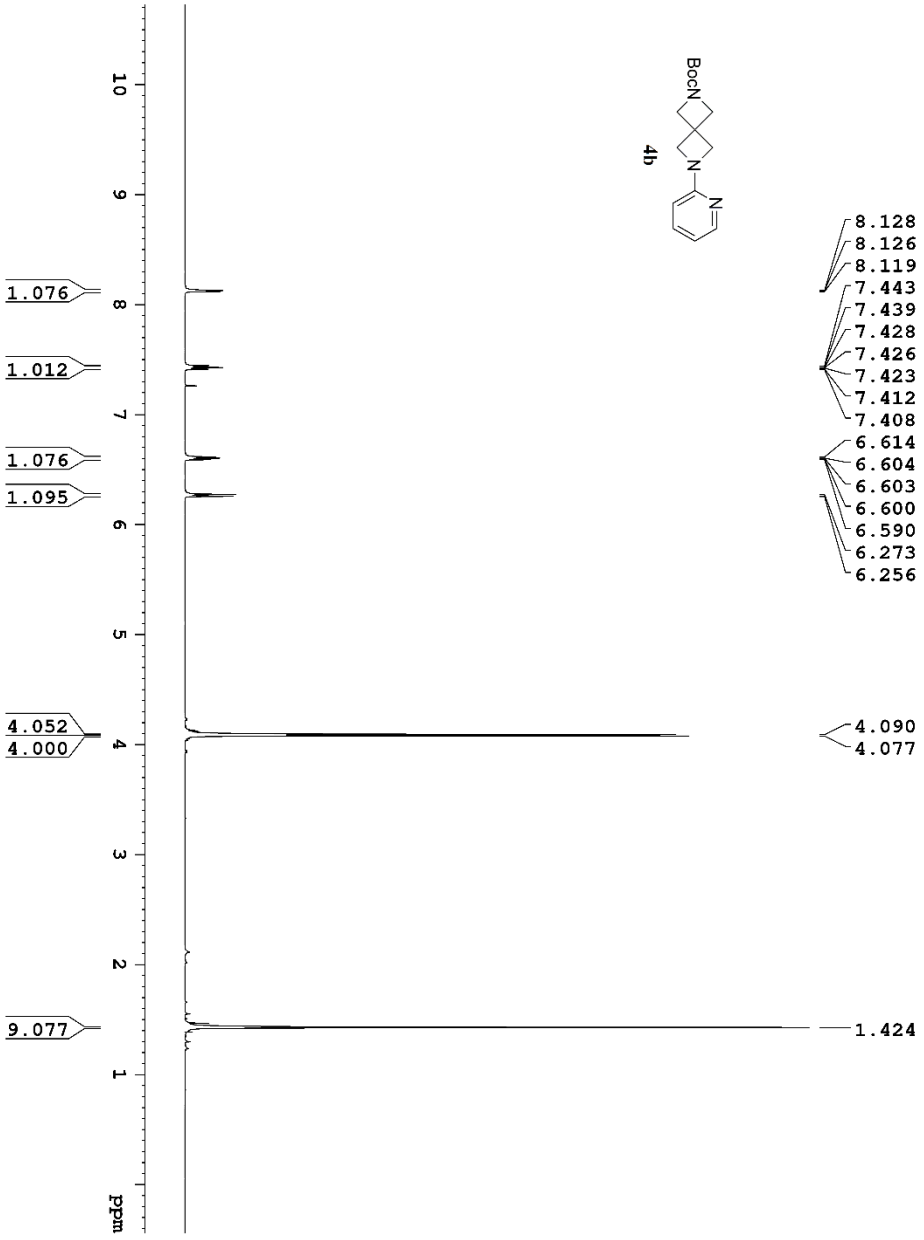

BocN
N
Cl
2n
156.108
152.162
134.981
130.130
118.020
111.764
109.930
79.882
62.202
59.696
33.549
28.477
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

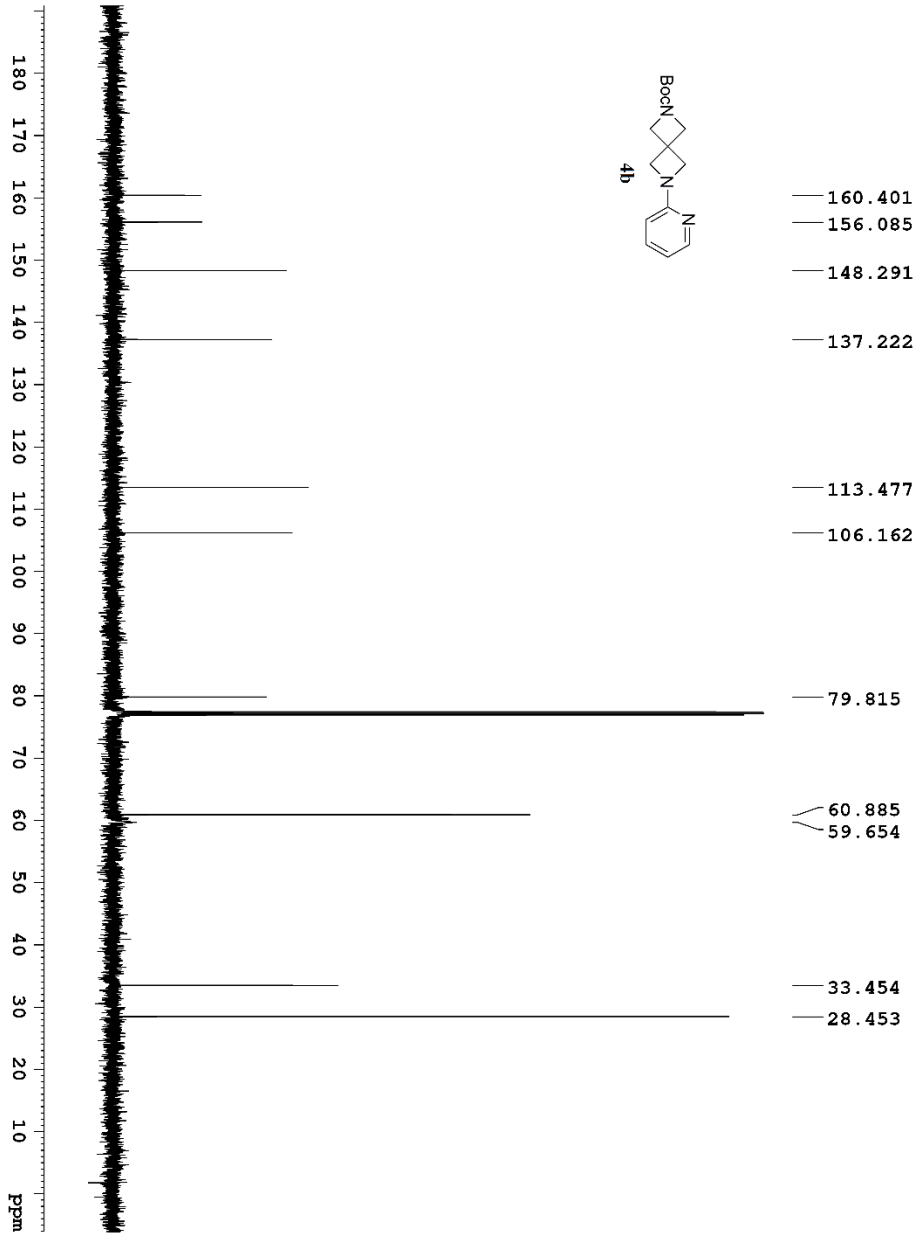

BocN
N
Cl
Cl
2o
7.047
7.031
7.015
6.924
6.908
6.390
6.374
4.152
4.083
1.443
1.053
1.007
1.000
4.002
4.000
9.004
ppm

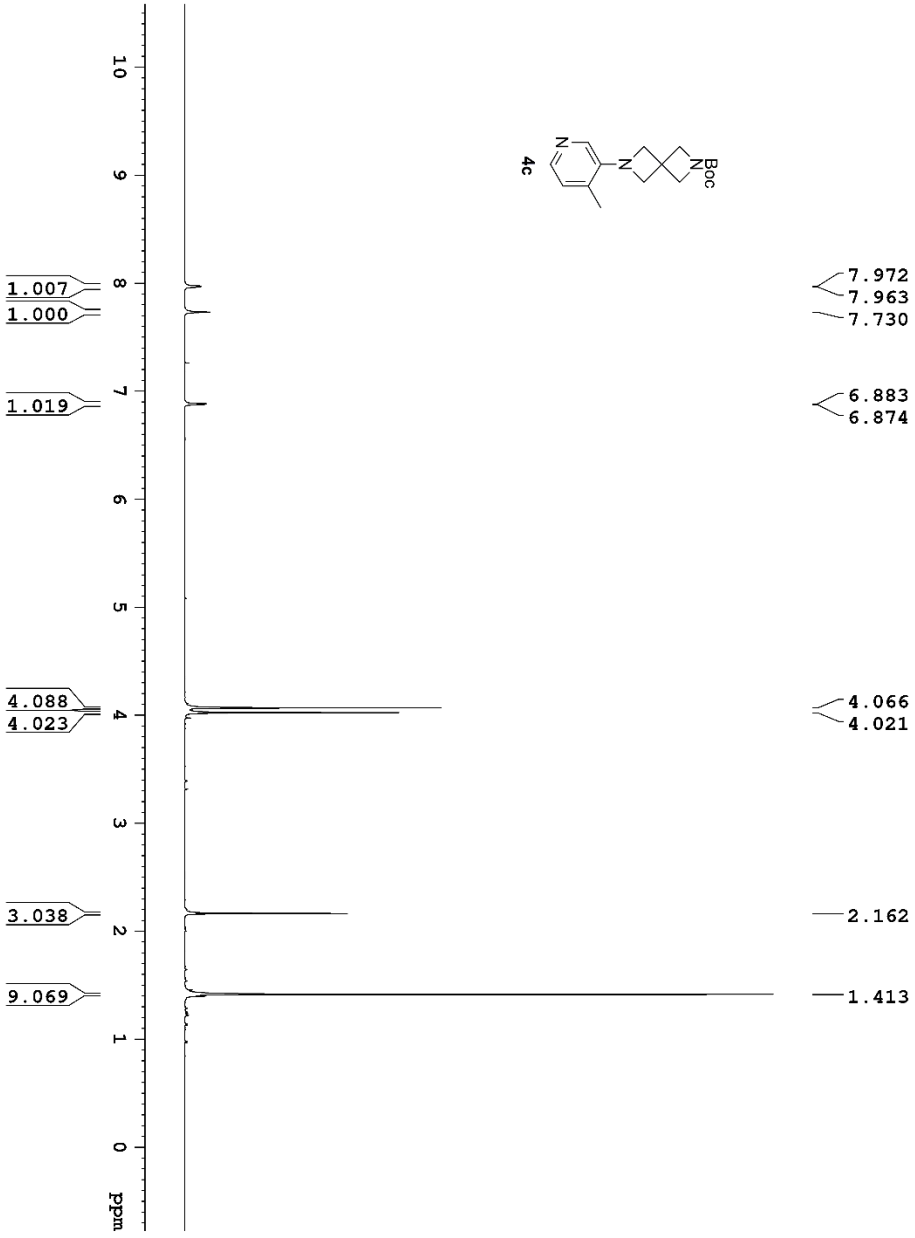

BocN
N
Cl
Cl
2o
156.131
148.785
134.248
127.358
121.003
118.993
112.706
79.889
63.983
59.632
33.099
28.488
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
ppm

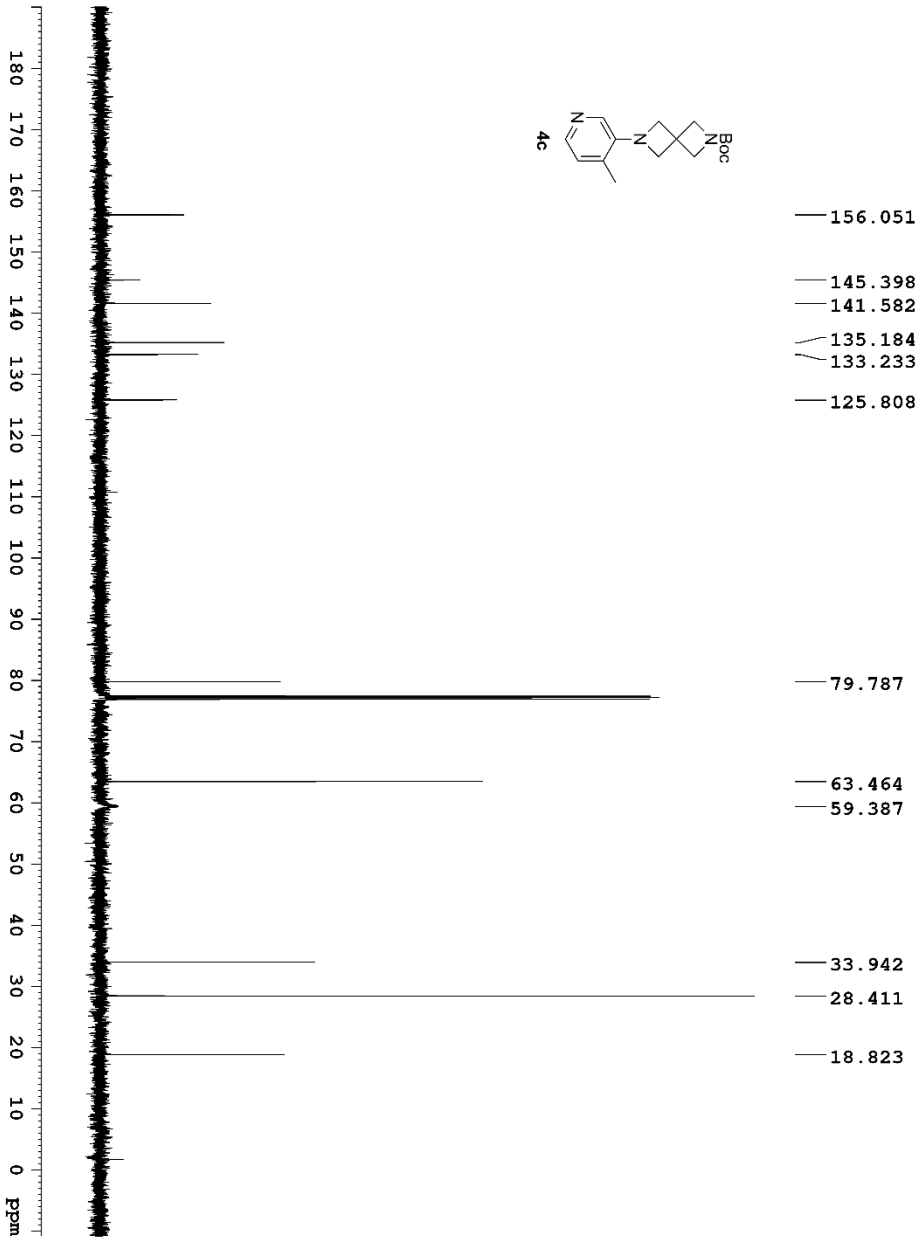

4a
Boc
7.992
7.989
7.848
7.843
7.756
4.166
4.090
1.413
1.009
1.000
1.014
4.047
4.067
9.074
ppm

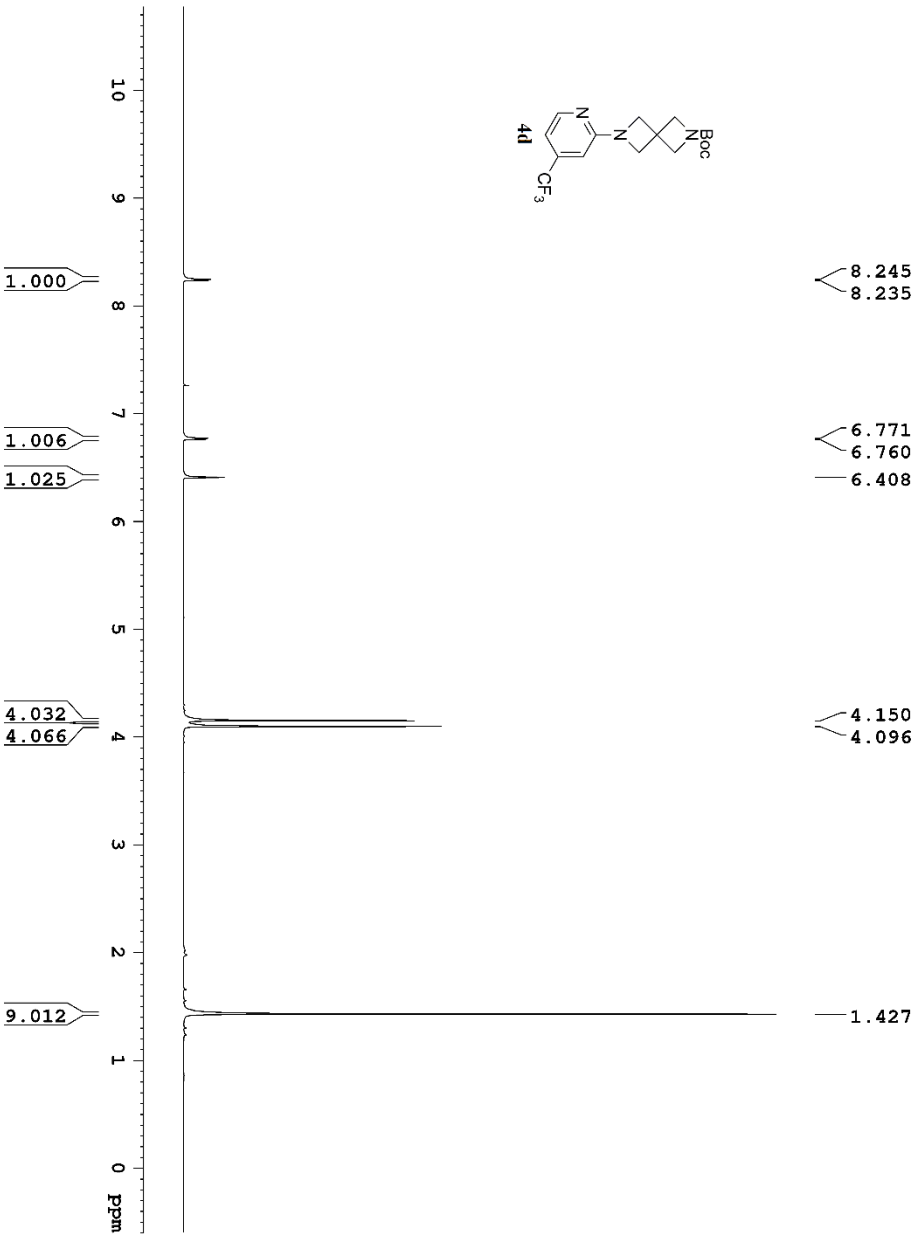

4a
Boc
156.004
155.658
142.093
133.604
130.495
79.941
60.831
59.644
34.023
28.418
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

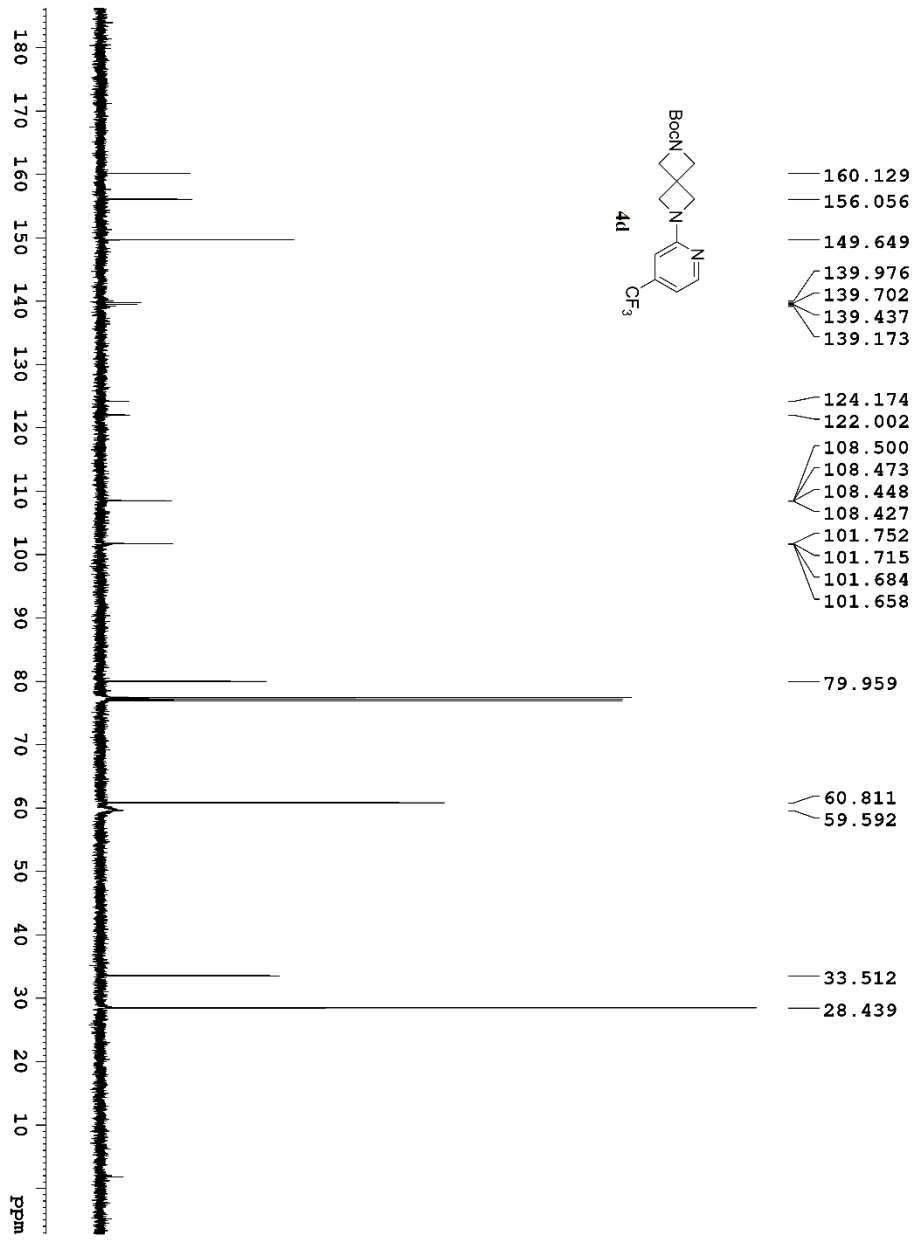

BocN
N
N
4h
8.128
8.126
8.119
7.443
7.439
7.428
7.426
7.423
7.412
7.408
6.614
6.604
6.603
6.600
6.590
6.273
6.256
4.090
4.077
1.424
1.076
1.012
1.076
1.095
4.052
4.000
9.077
10
9
8
7
6
5
4
3
2
1
ppm

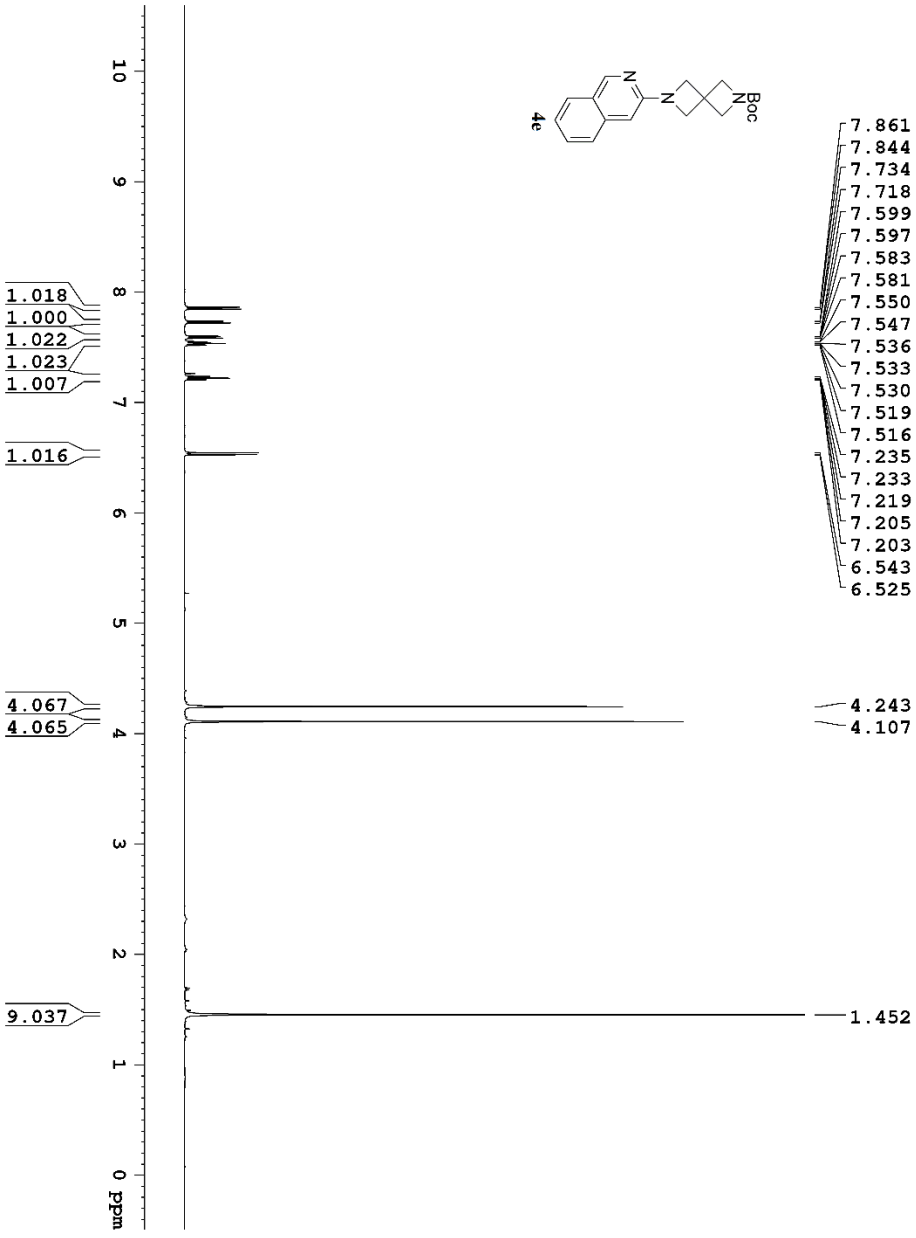

BocN
N
N
4b
160.401
156.085
148.291
137.222
113.477
106.162
79.815
60.885
59.654
33.454
28.453
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

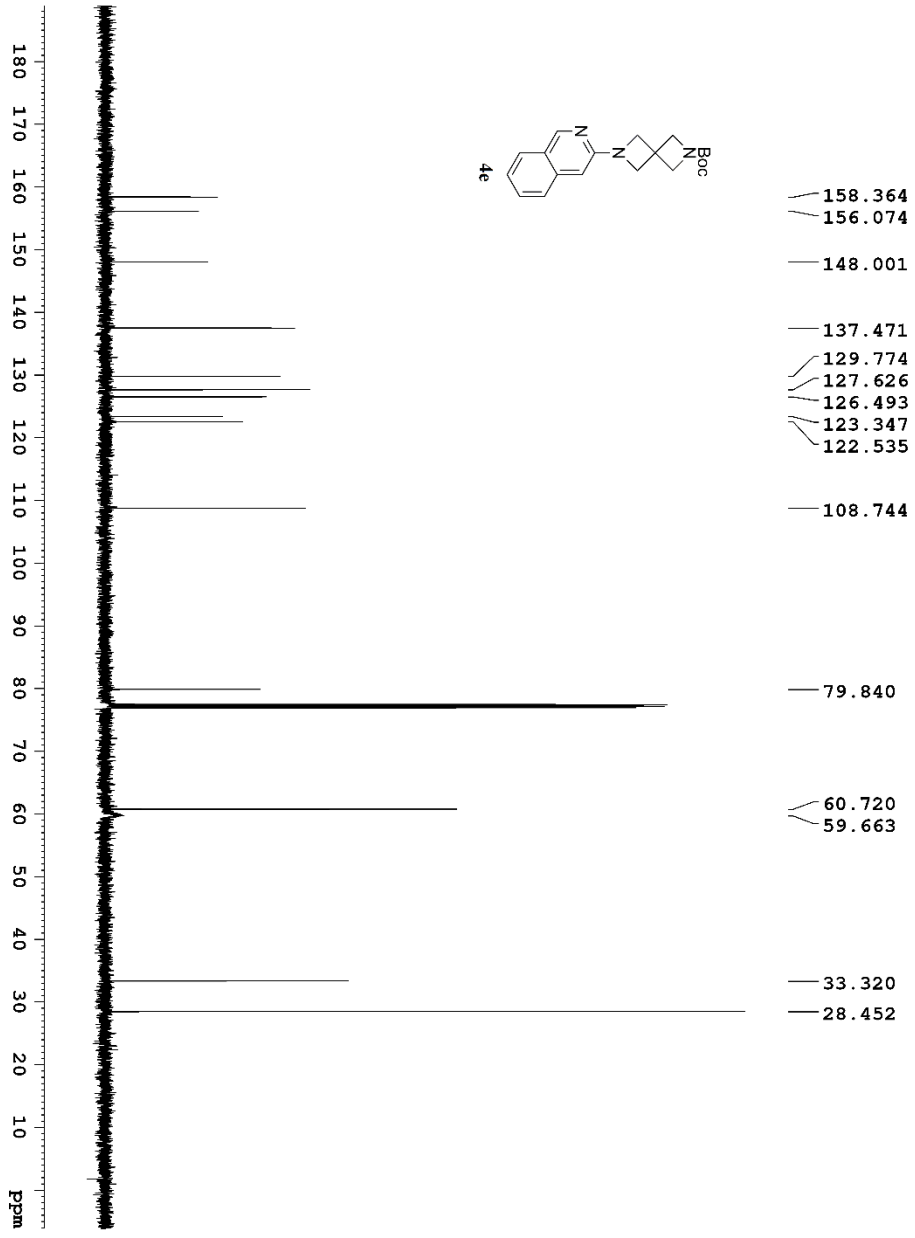

4c
Boc
1.007
1.000
1.019
4.088
4.023
3.038
9.069
7.972
7.963
7.730
6.883
6.874
4.066
4.021
2.162
1.413
ppm

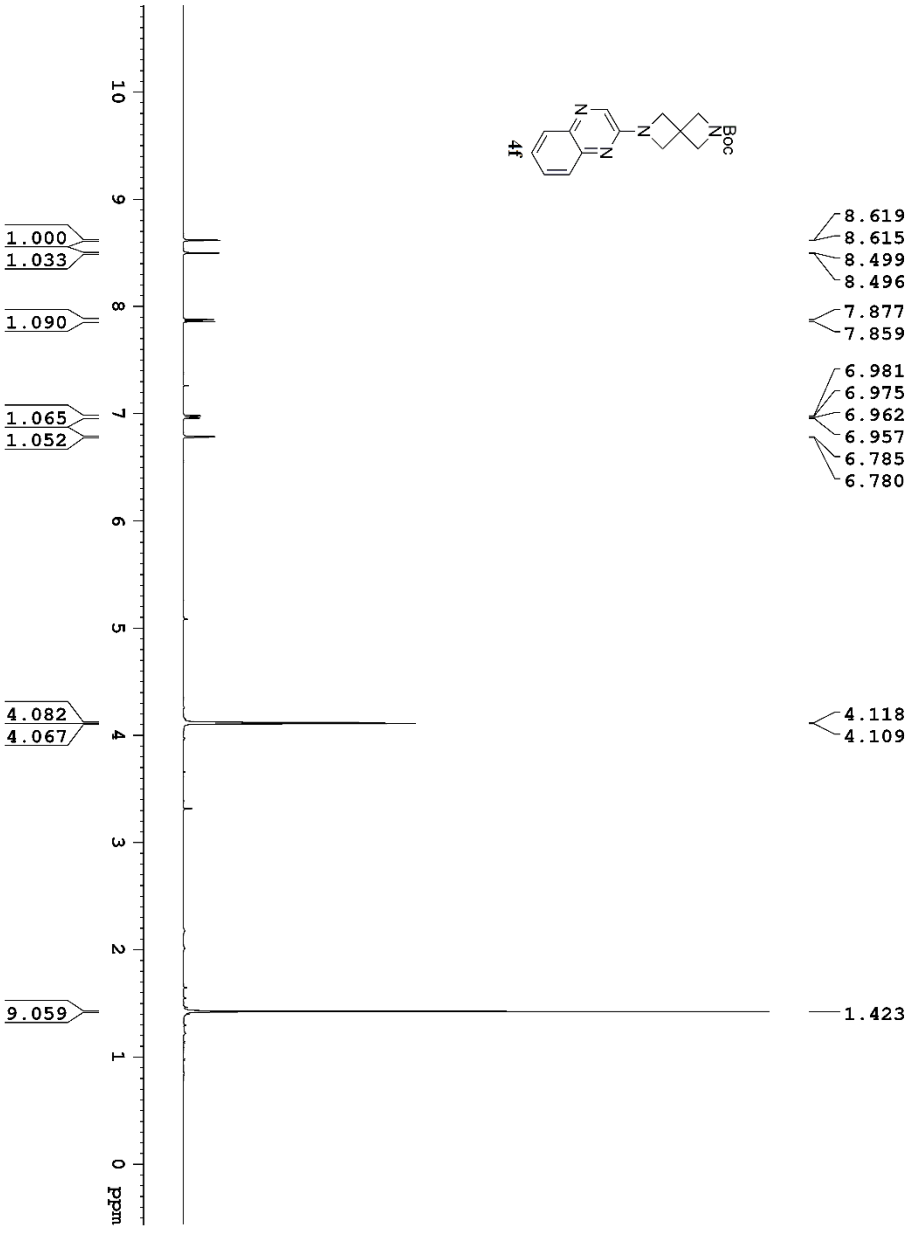

4c
N
N
N
Boc
156.051
145.398
141.582
135.184
133.233
125.808
79.787
63.464
59.387
33.942
28.411
18.823
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

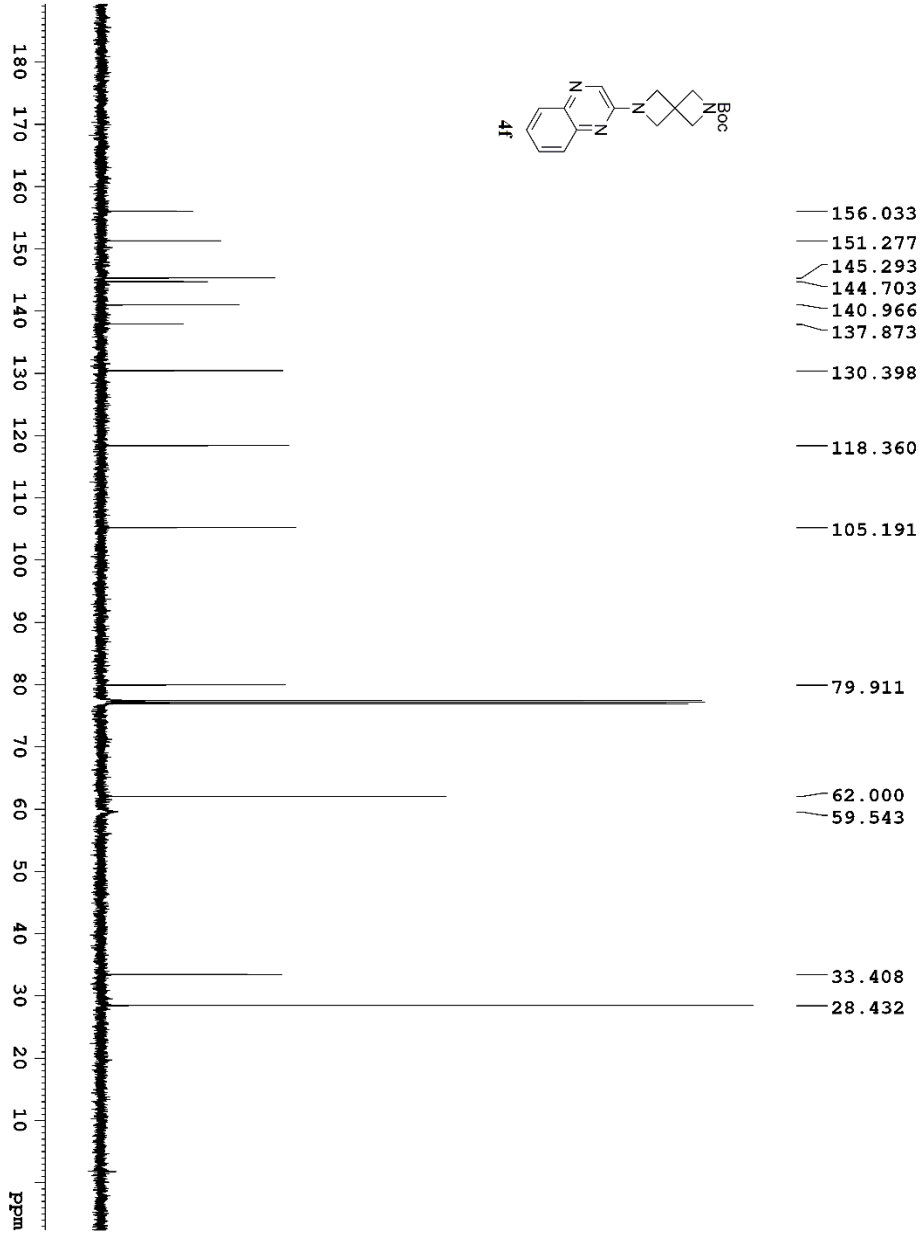

4ul
Boc
CF3
1.000
1.006
1.025
4.032
4.066
9.012
8.245
8.235
6.771
6.760
6.408
4.150
4.096
1.427
ppm

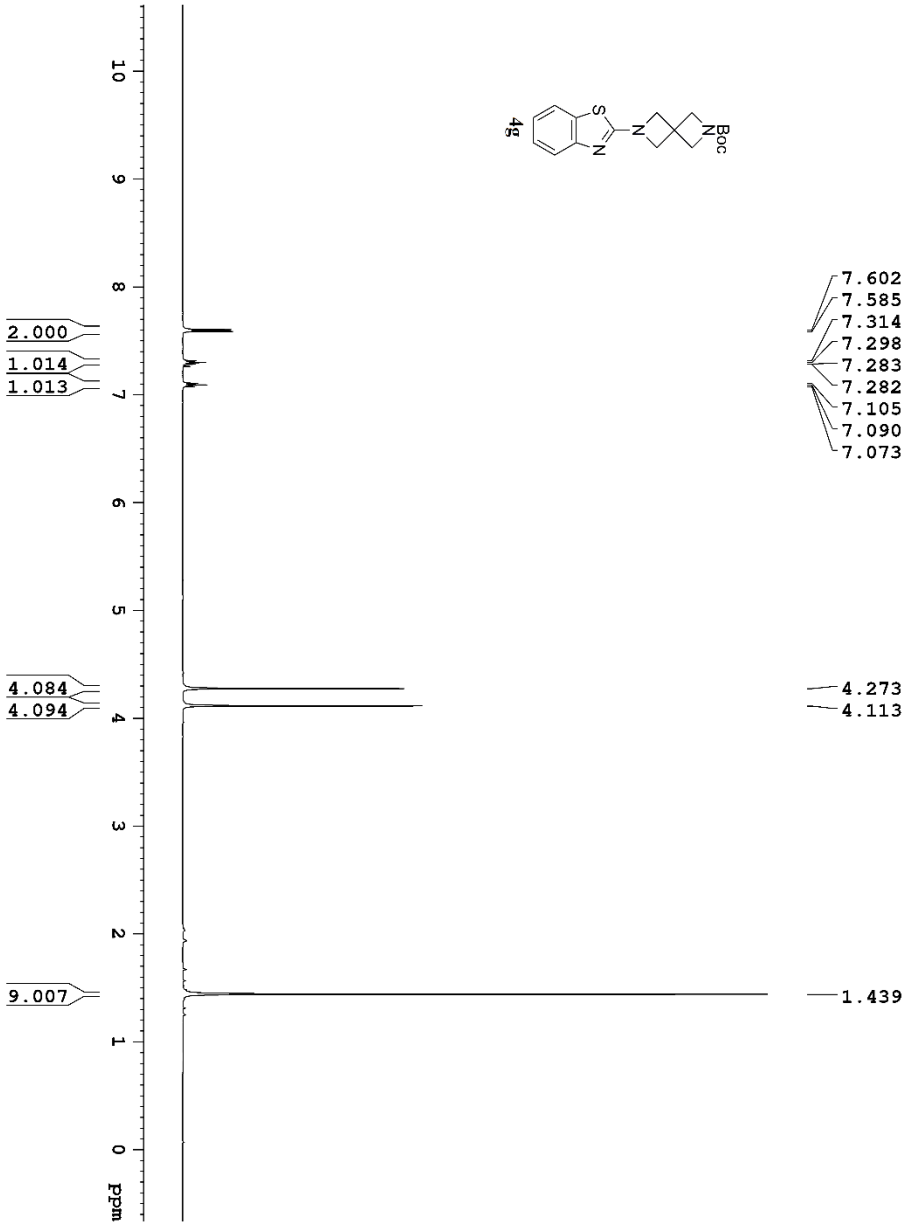

BocN
N
N
CF3
4d
160.129
156.056
149.649
139.976
139.702
139.437
139.173
124.174
122.002
108.500
108.473
108.448
108.427
101.752
101.715
101.684
101.658
79.959
60.811
59.592
33.512
28.439
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

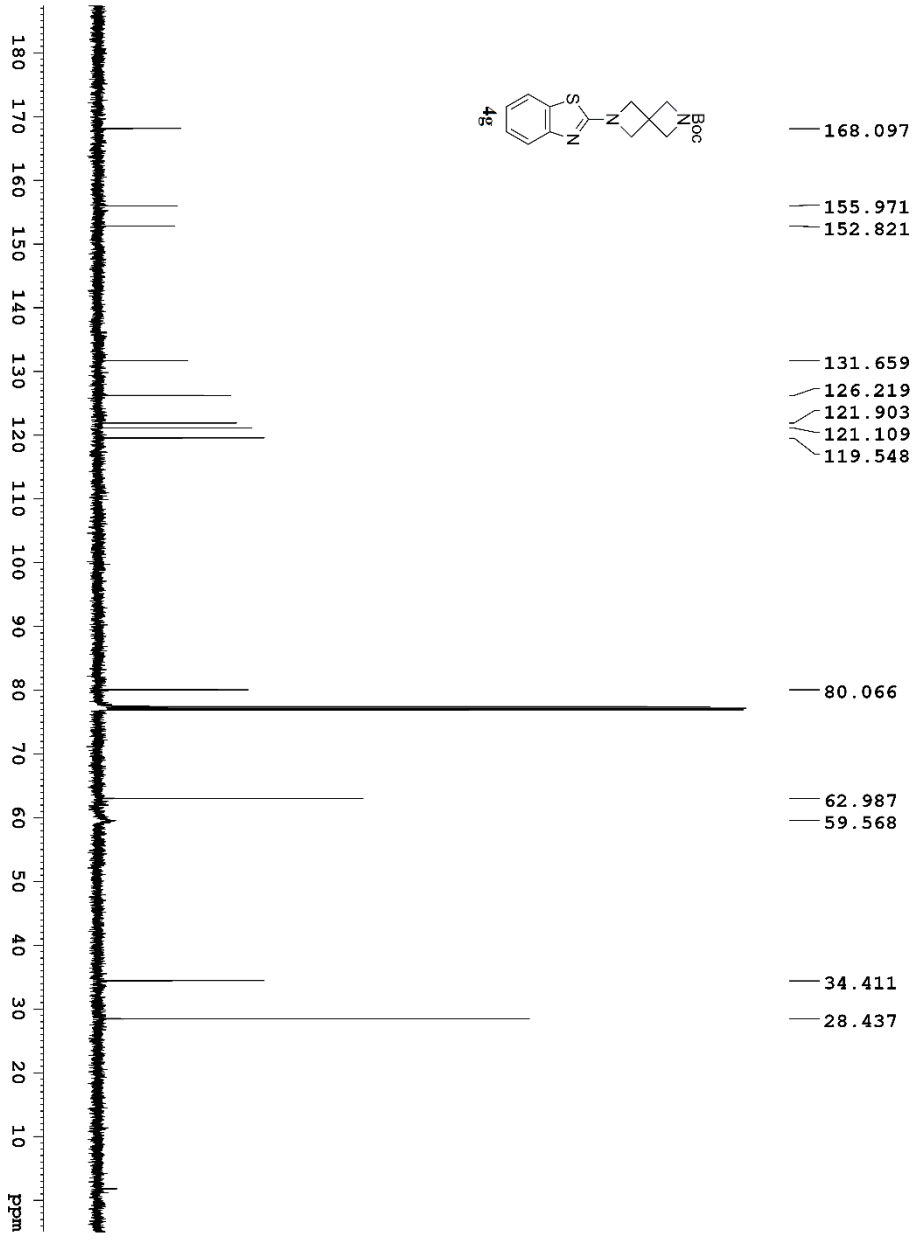

4e
Boc
7.861
7.844
7.734
7.718
7.599
7.597
7.583
7.581
7.550
7.547
7.536
7.533
7.530
7.519
7.516
7.235
7.233
7.219
7.205
7.203
6.543
6.525
4.243
4.107
1.452
1.018
1.000
1.022
1.023
1.007
1.016
4.067
4.065
9.037
ppm

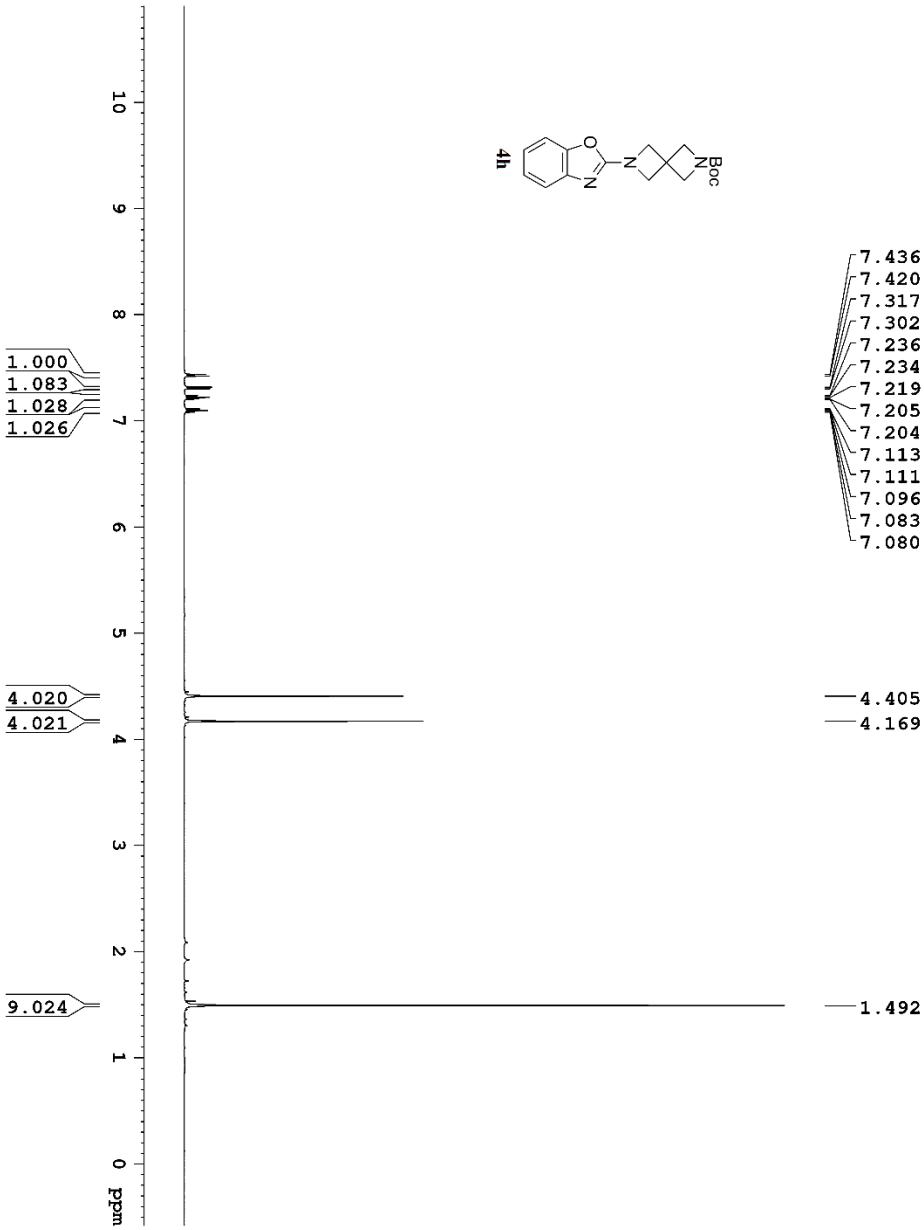

4e
Boc
158.364
156.074
148.001
137.471
129.774
127.626
126.493
123.347
122.535
108.744
79.840
60.720
59.663
33.320
28.452
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

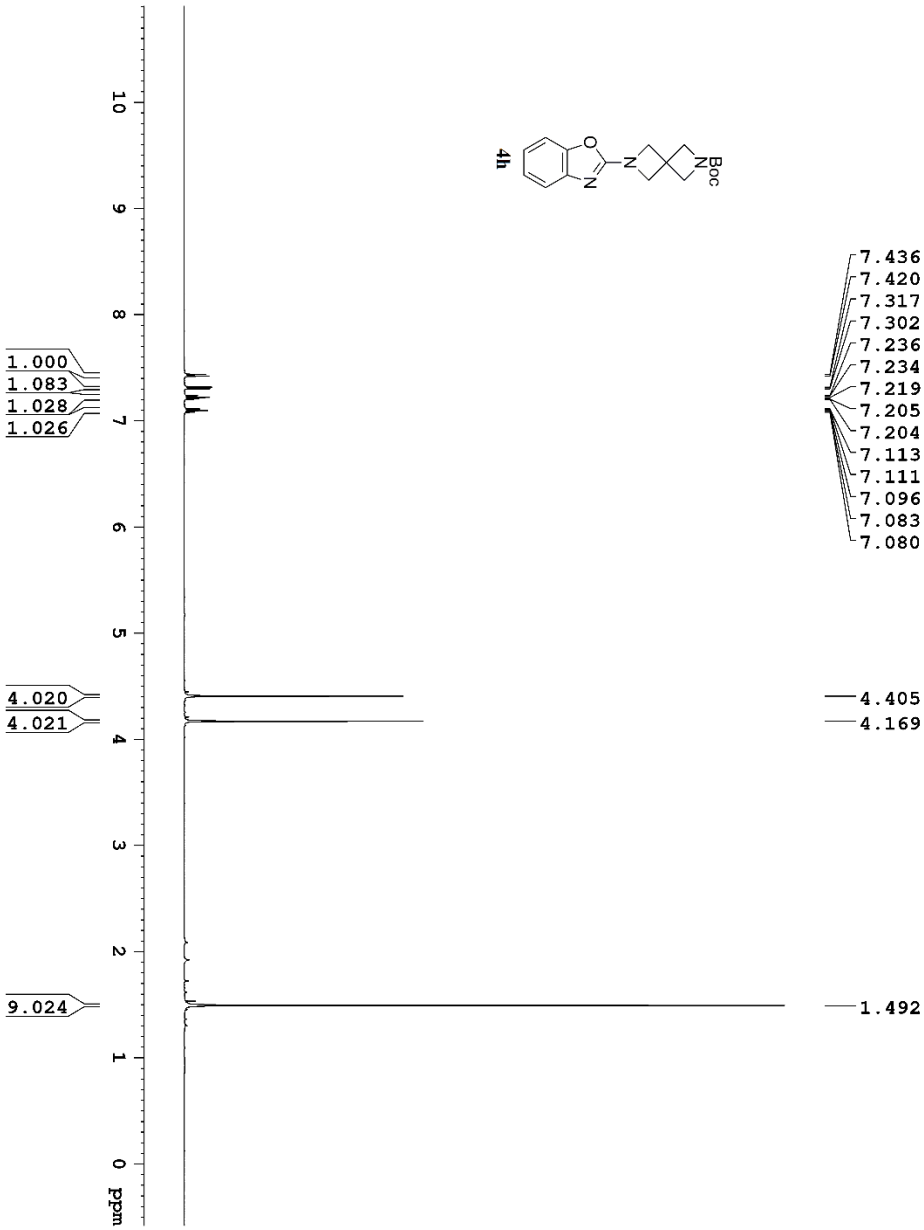

4f
Boc
8.619
8.615
8.499
8.496
7.877
7.859
6.981
6.975
6.962
6.957
6.785
6.780
4.118
4.109
1.423
1.000
1.033
1.090
1.065
1.052
4.082
4.067
9.059
ppm

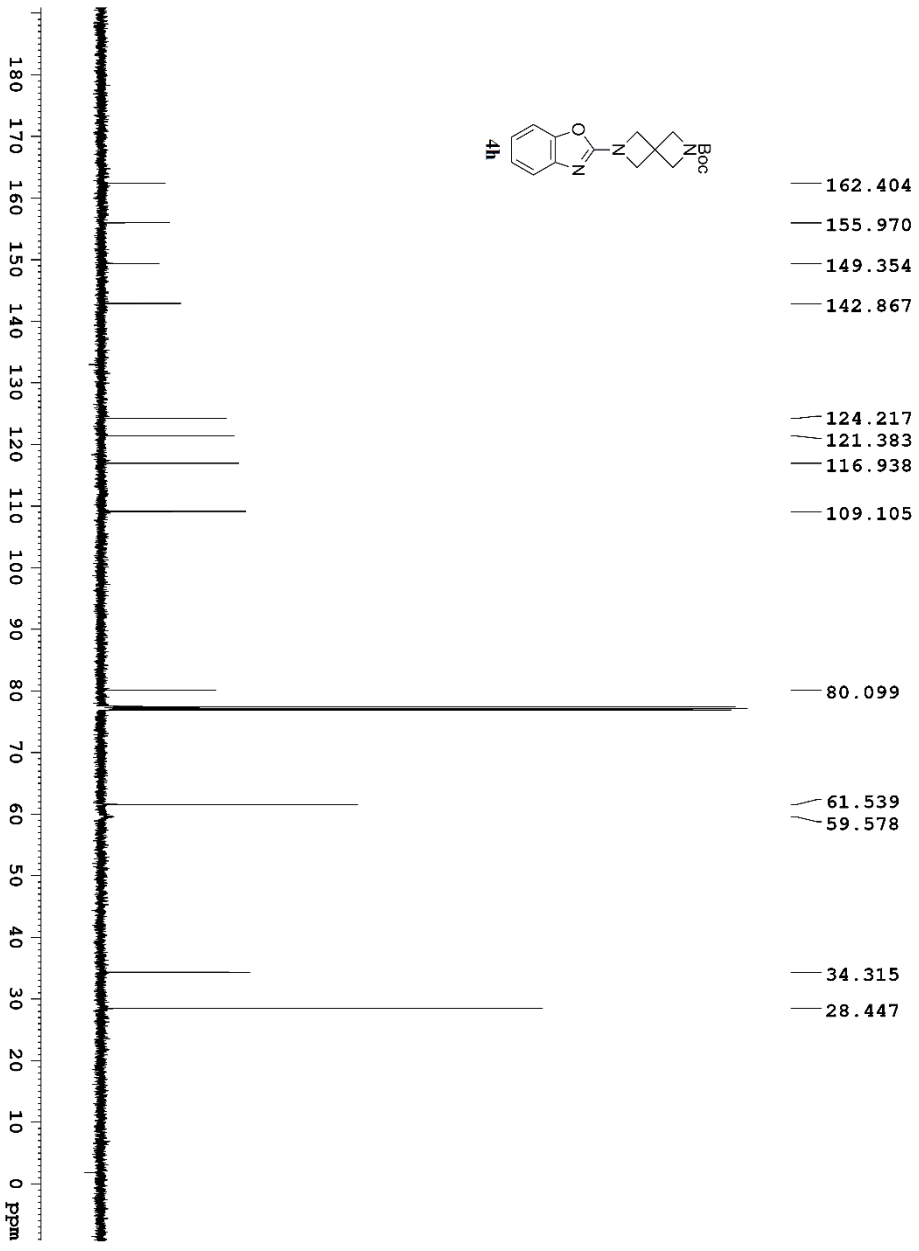

4f
Boc
156.033
151.277
145.293
144.703
140.966
137.873
130.398
118.360
105.191
79.911
62.000
59.543
33.408
28.432
ppm

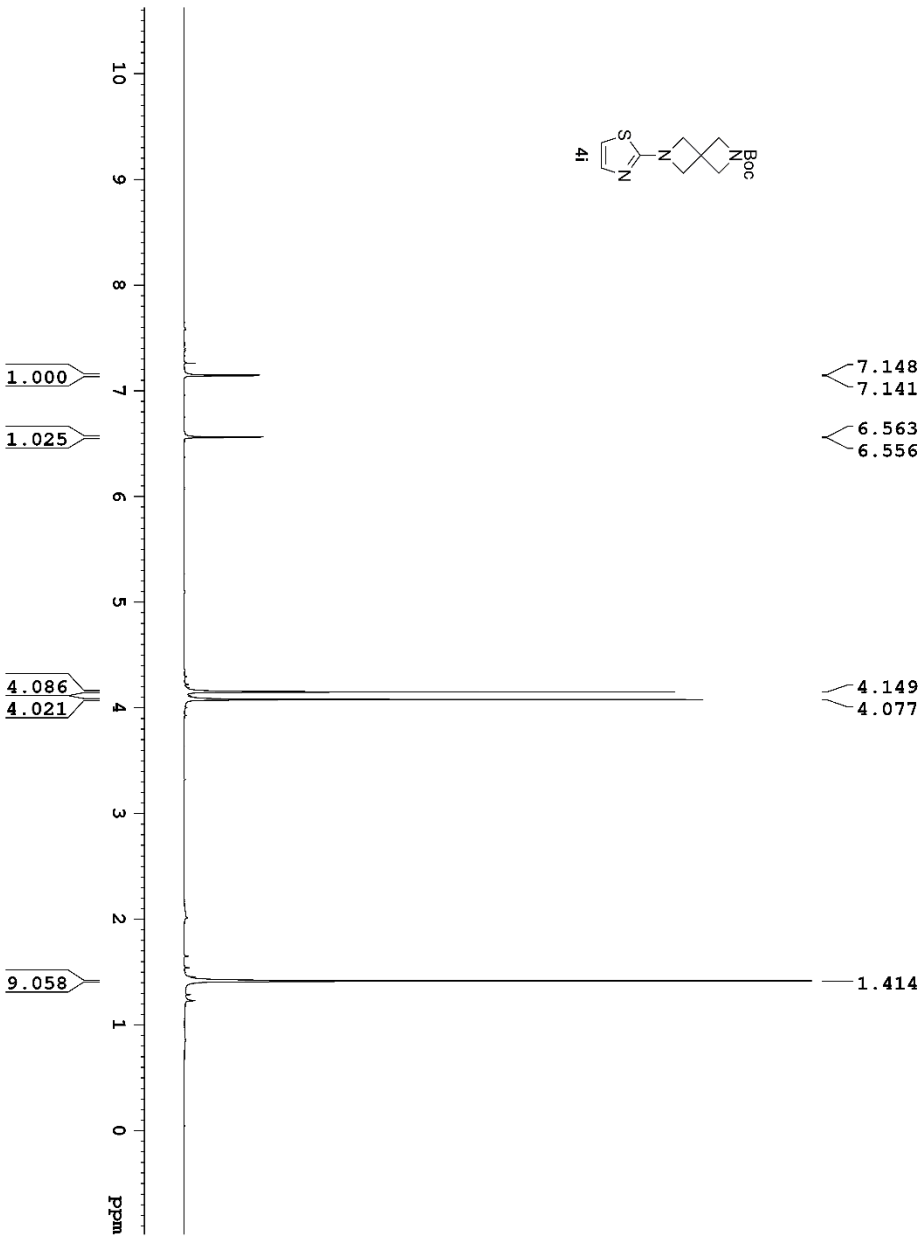

4g
S
N
N
N
Boc
2.000
1.014
1.013
4.084
4.094
9.007
7.602
7.585
7.314
7.298
7.283
7.282
7.105
7.090
7.073
4.273
4.113
1.439
ppm

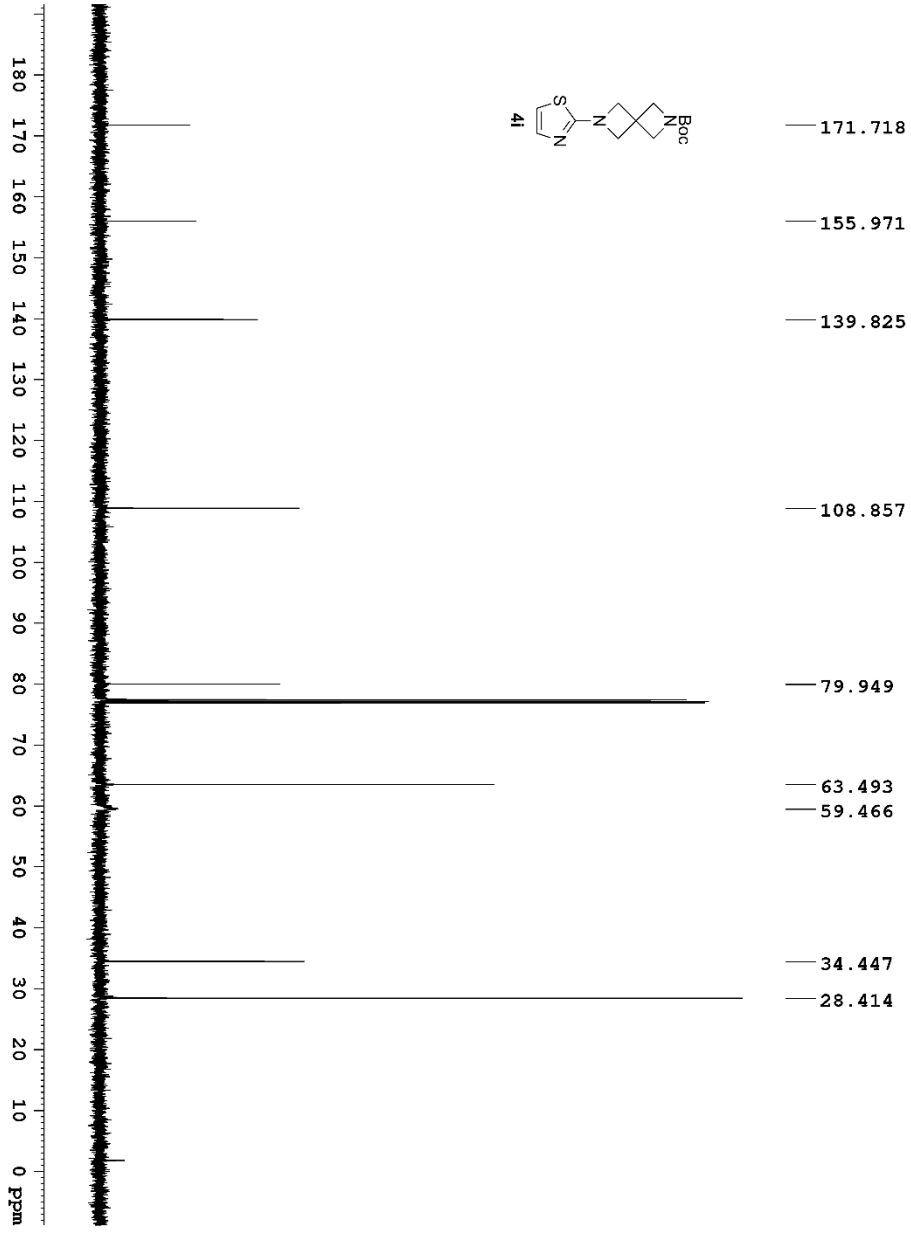

4g
Boc
168.097
155.971
152.821
131.659
126.219
121.903
121.109
119.548
80.066
62.987
59.568
34.411
28.437
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

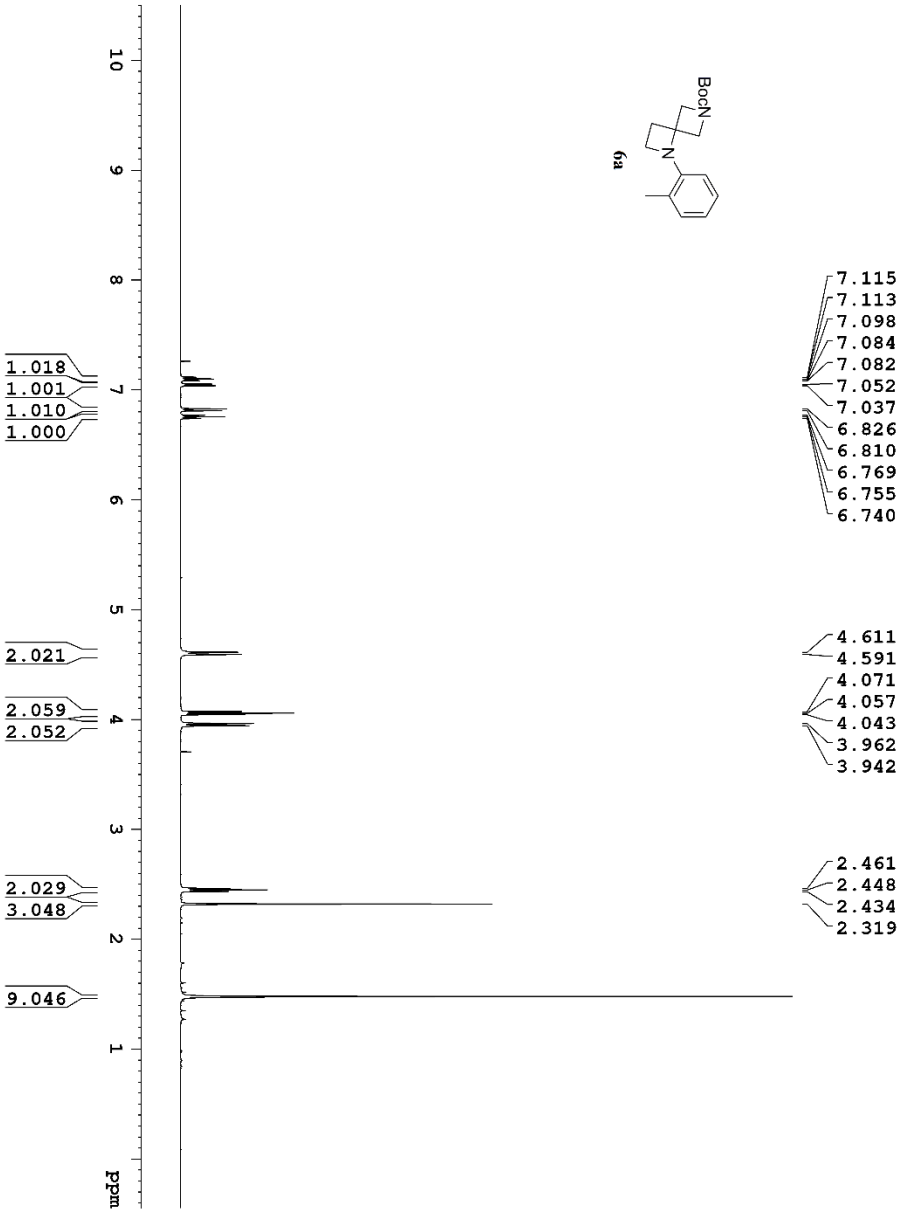

4h
Boc
7.436
7.420
7.317
7.302
7.236
7.234
7.219
7.205
7.204
7.113
7.111
7.096
7.083
7.080
4.405
4.169
1.492
1.000
1.083
1.028
1.026
4.020
4.021
9.024
ppm

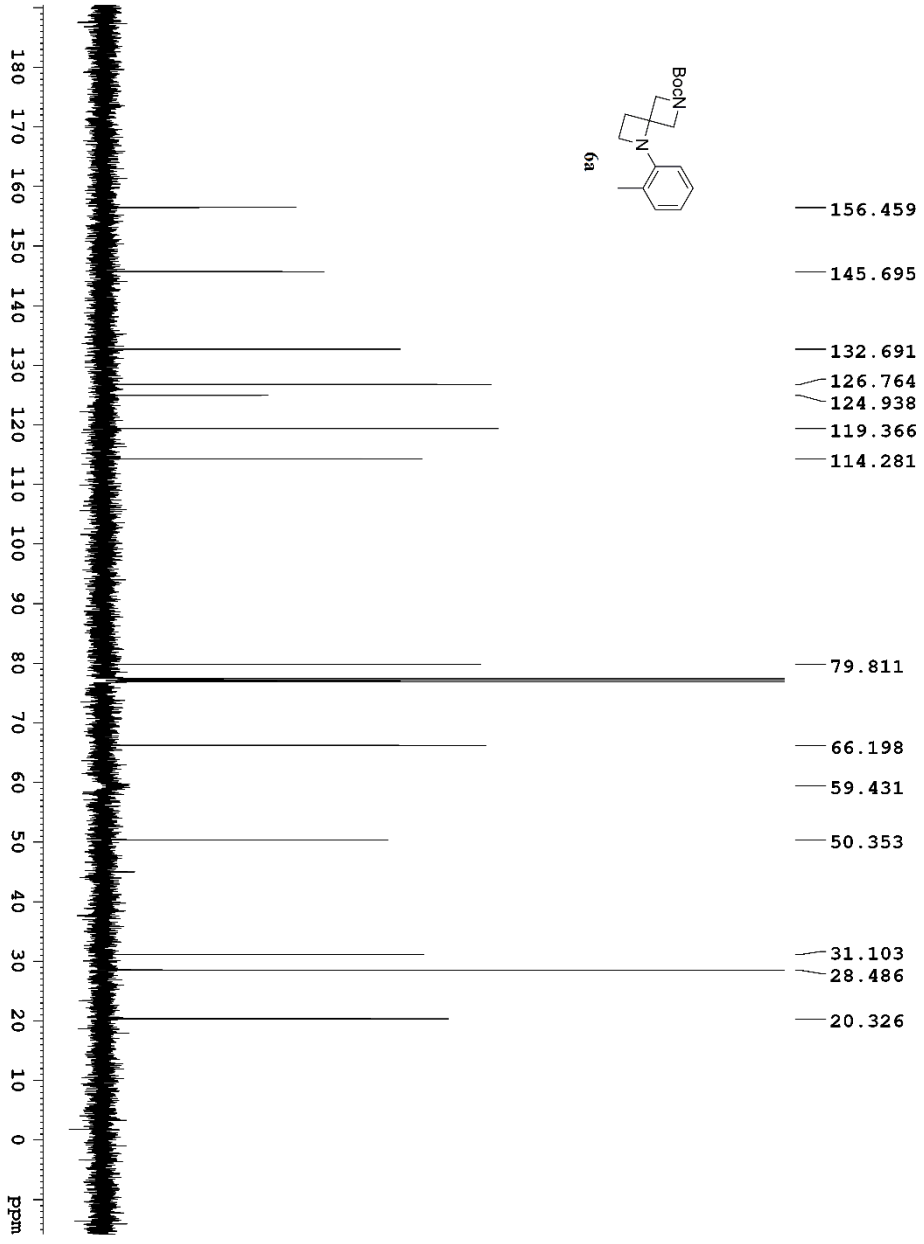

4h
Boc
7.436
7.420
7.317
7.302
7.236
7.234
7.219
7.205
7.204
7.113
7.111
7.096
7.083
7.080
4.405
4.169
1.492
1.000
1.083
1.028
1.026
4.020
4.021
9.024
ppm

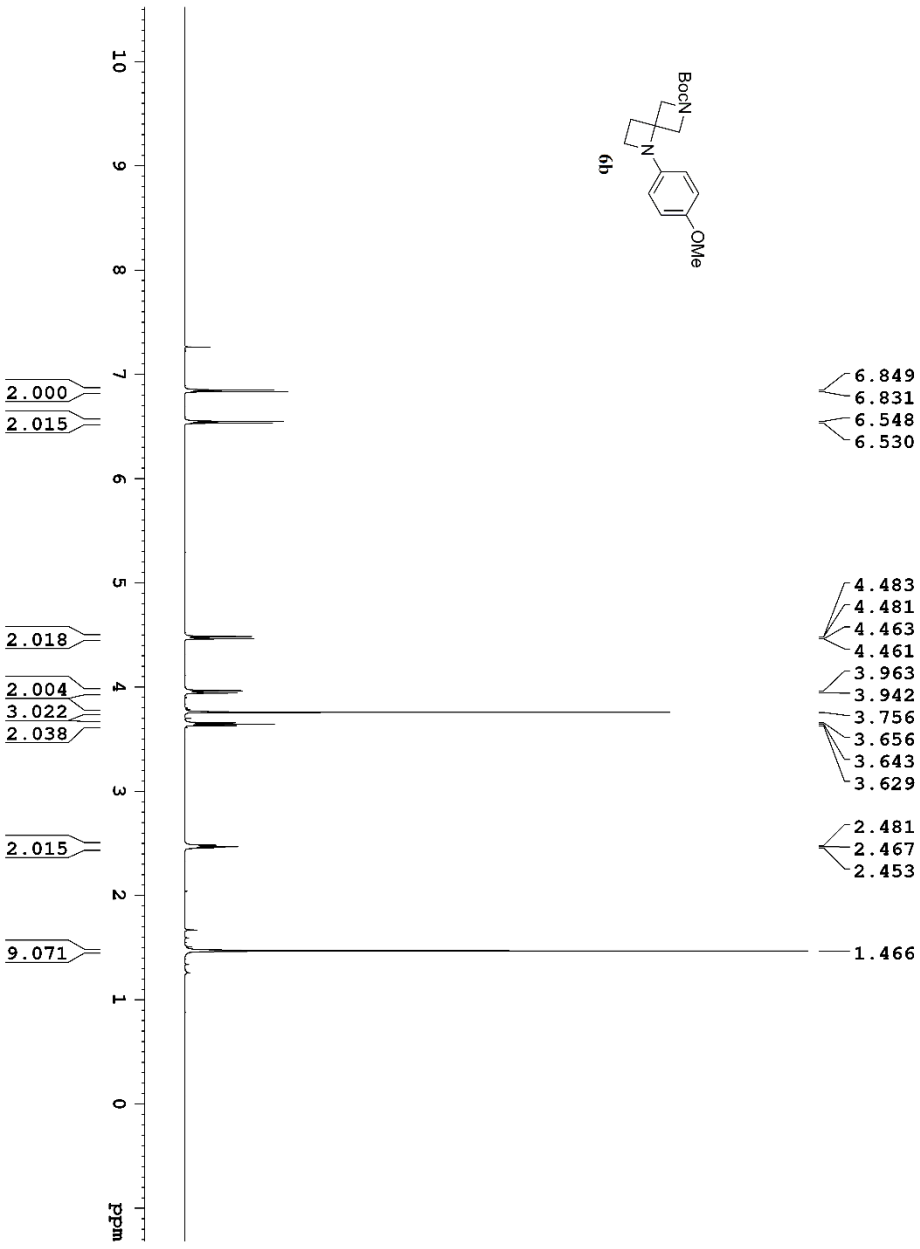

4h
Boc
162.404
155.970
149.354
142.867
124.217
121.383
116.938
109.105
80.099
61.539
59.578
34.315
28.447
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

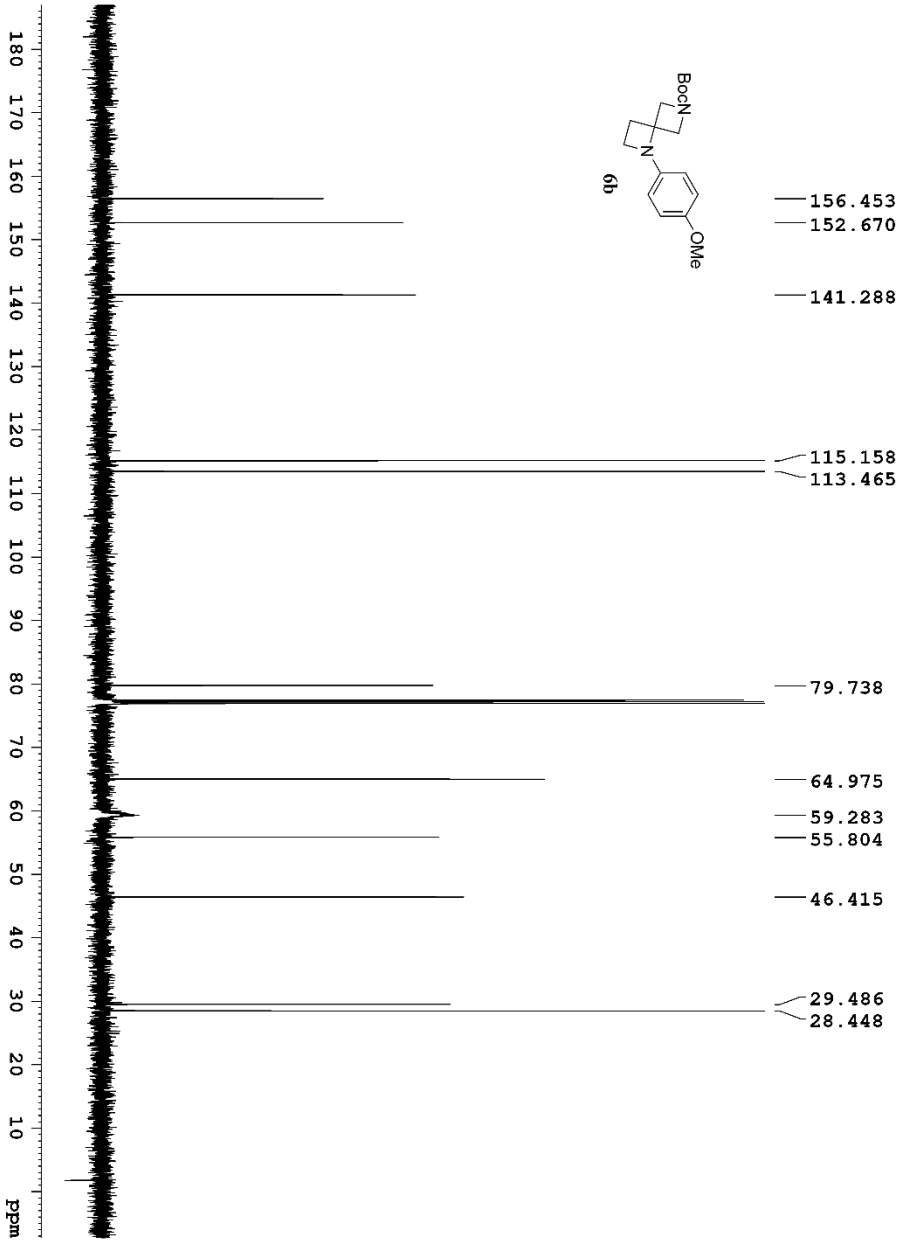

4i
S
N
N
N
Boc
1.000
1.025
4.086
4.021
9.058
7.148
7.141
6.563
6.556
4.149
4.077
1.414
ppm

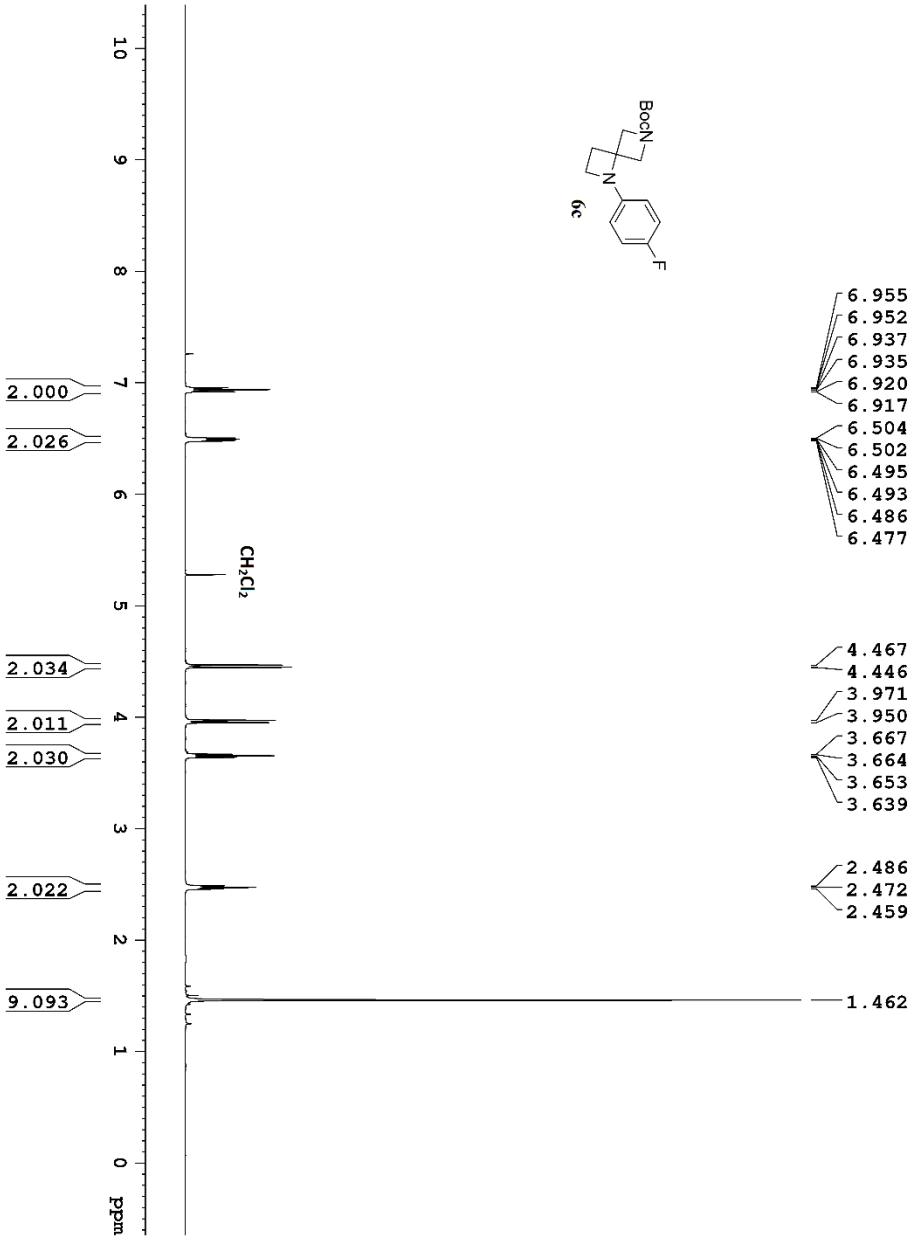

4i
Boc
171.718
155.971
139.825
108.857
79.949
63.493
59.466
34.447
28.414
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


BocN
N
6ja
7.115
7.113
7.098
7.084
7.082
7.052
7.037
6.826
6.810
6.769
6.755
6.740
4.611
4.591
4.071
4.057
4.043
3.962
3.942
2.461
2.448
2.434
2.319
1.018
1.001
1.010
1.000
2.021
2.059
2.052
2.029
3.048
9.046
ppm


BocN
N
6ga
156.459
145.695
132.691
126.764
124.938
119.366
114.281
79.811
66.198
59.431
50.353
31.103
28.486
20.326
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


6b
BocN
N
OMe
2.000
2.015
2.018
2.004
3.022
2.038
2.015
9.071
6.849
6.831
6.548
6.530
4.483
4.481
4.463
4.461
3.963
3.942
3.756
3.656
3.643
3.629
2.481
2.467
2.453
1.466
ppm


BocN
N
OMe
6b
156.453
152.670
141.288
115.158
113.465
79.738
64.975
59.283
55.804
46.415
29.486
28.448
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
ppm


BocN
N
F
6c
6.955
6.952
6.937
6.935
6.920
6.917
6.504
6.502
6.495
6.493
6.486
6.477
4.467
4.446
3.971
3.950
3.667
3.664
3.653
3.639
2.486
2.472
2.459
1.462
CH2Cl2
2.000
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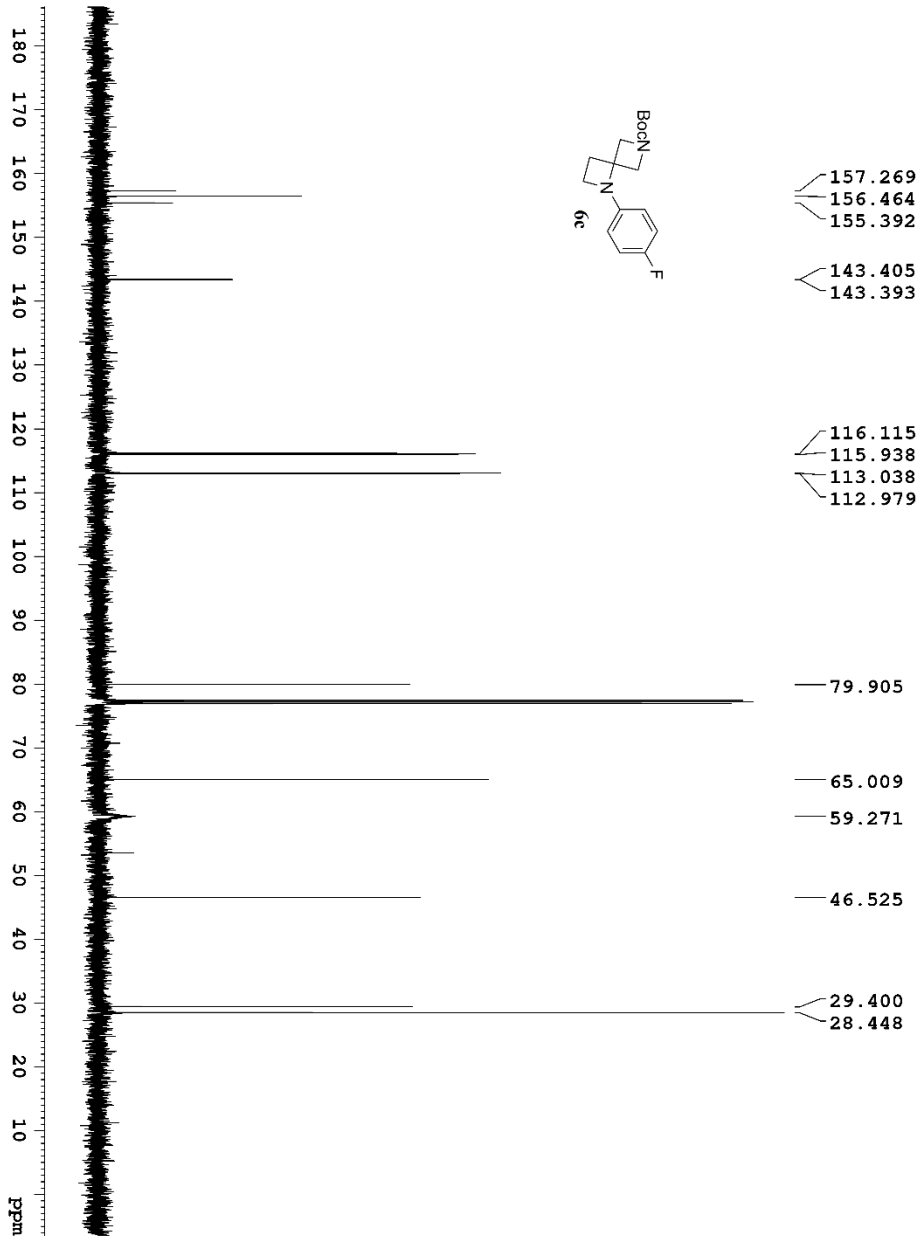
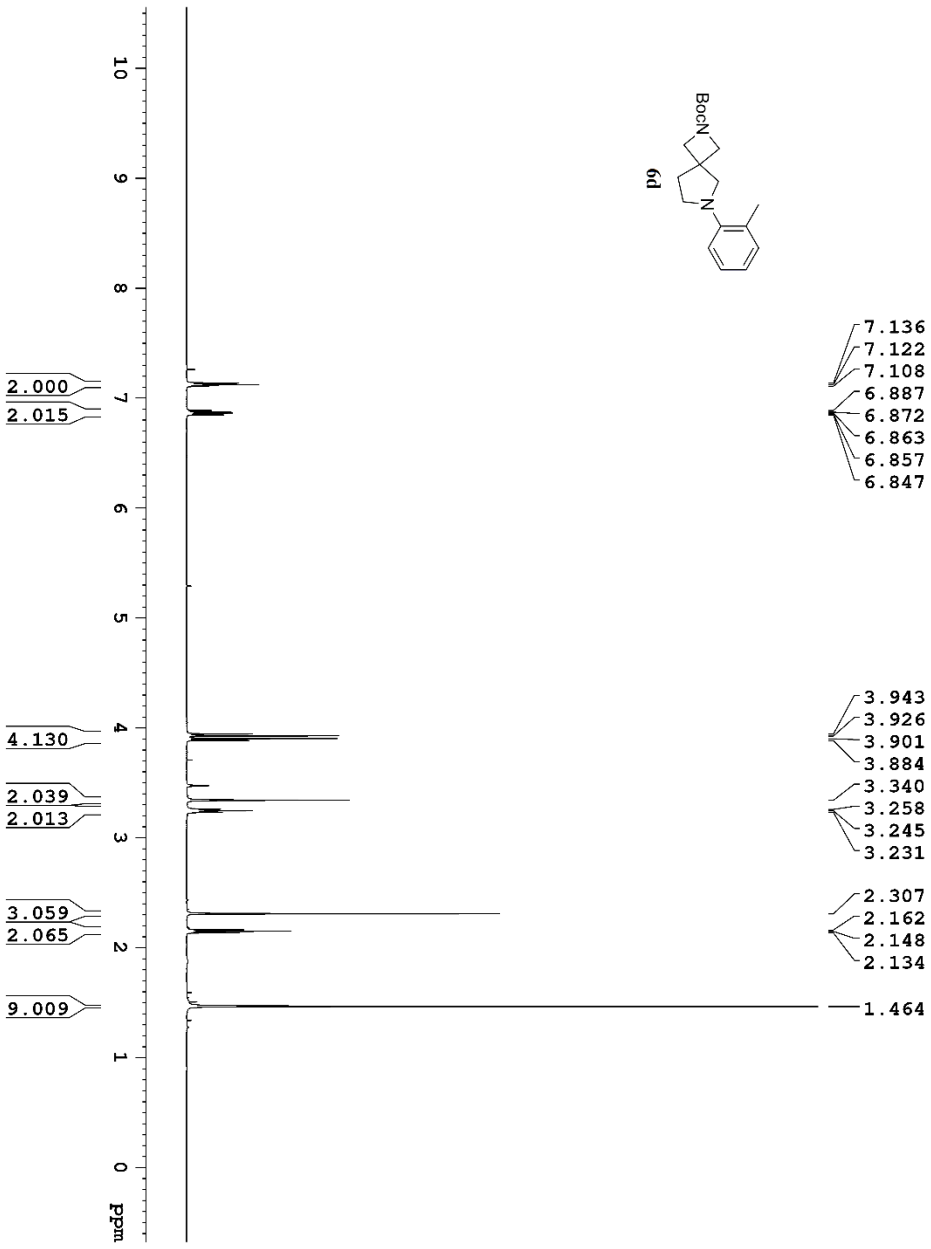
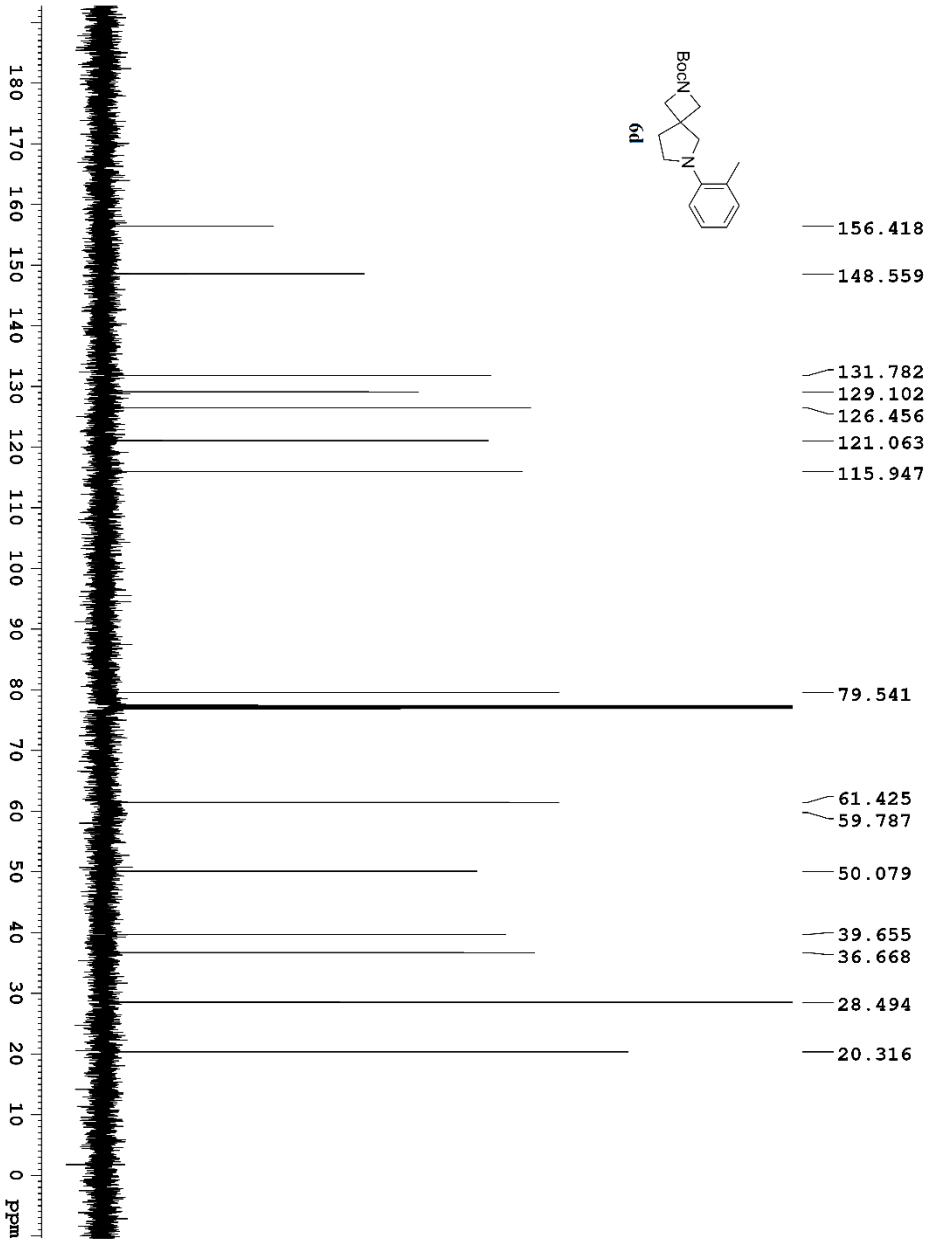
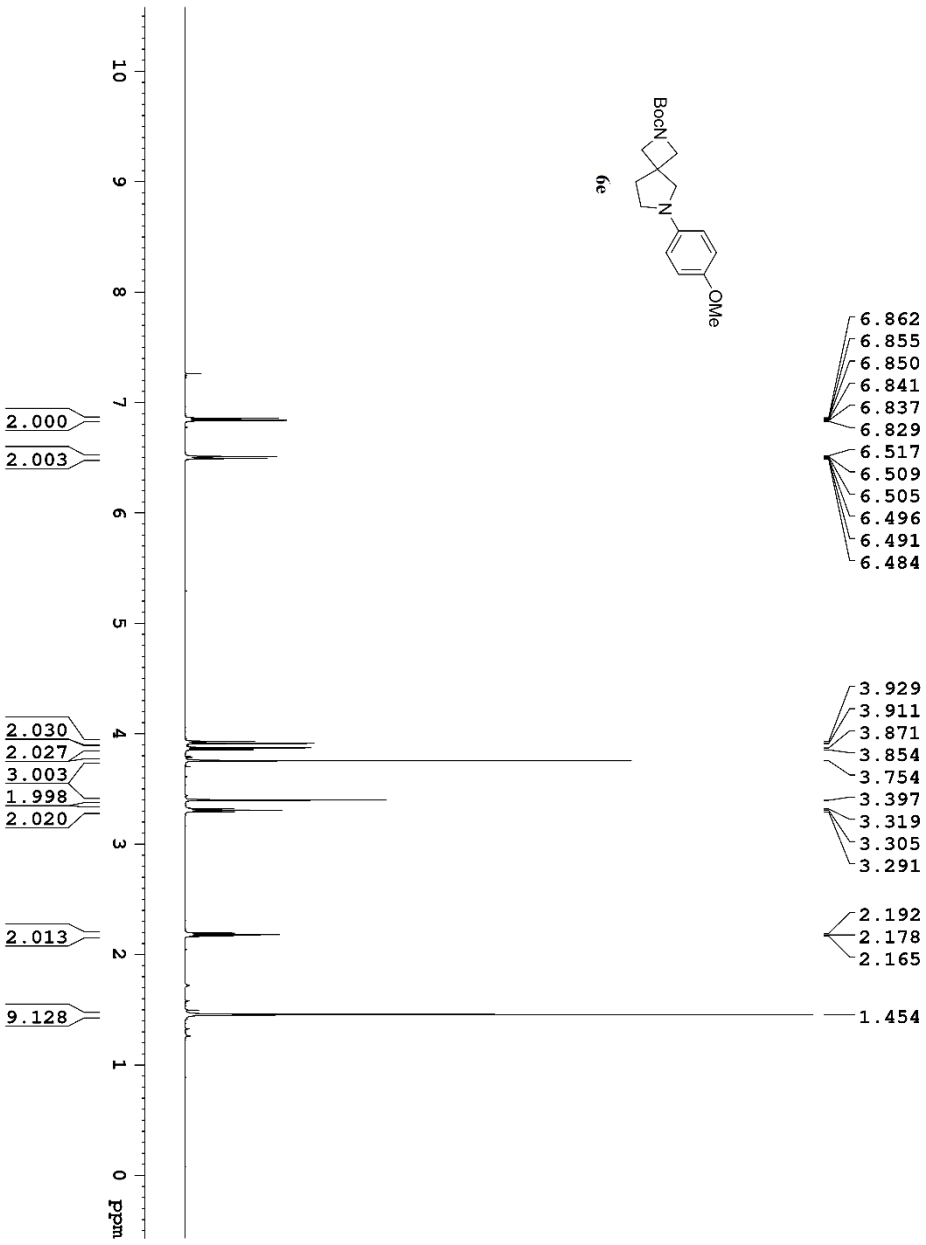
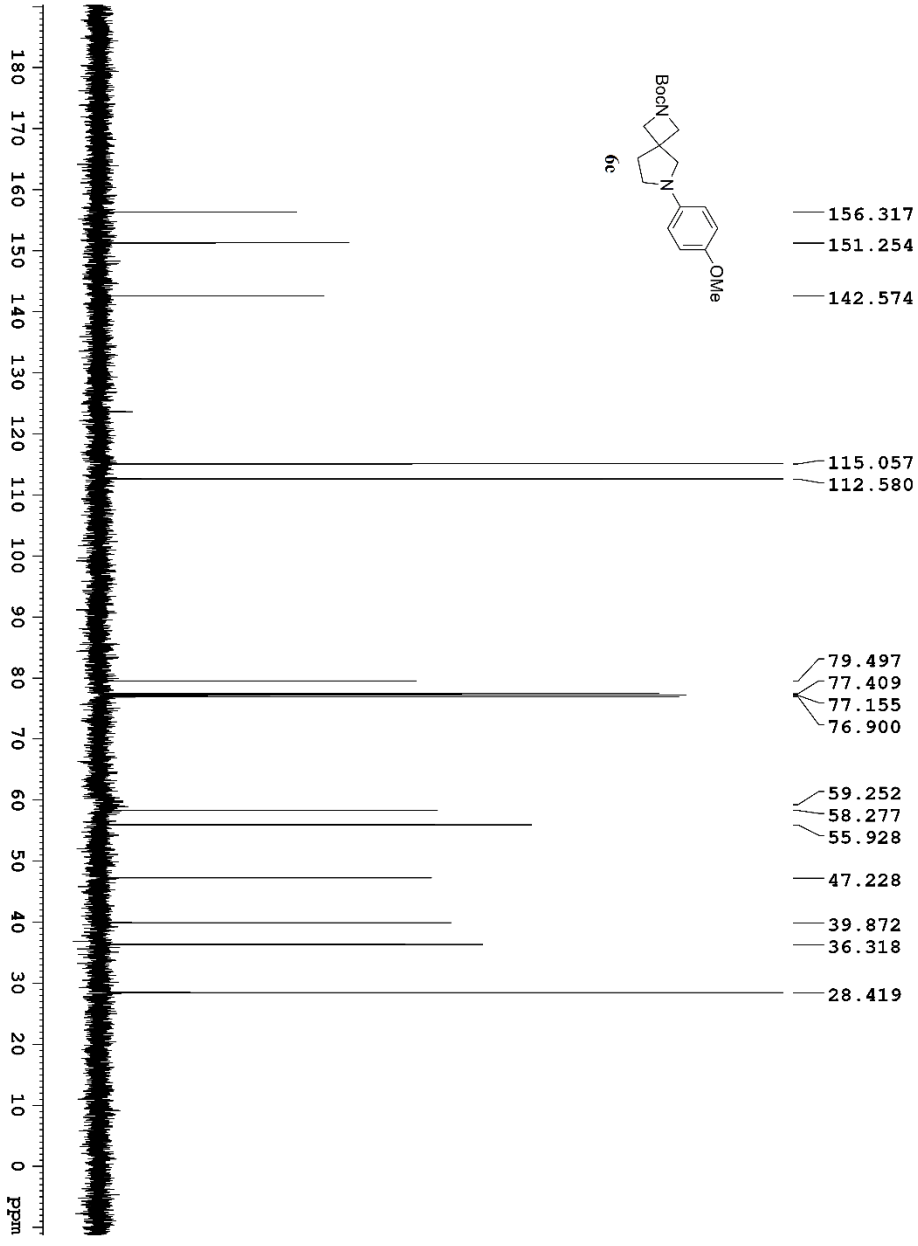
2.034
2.011
2.030
2.022
9.093
ppm

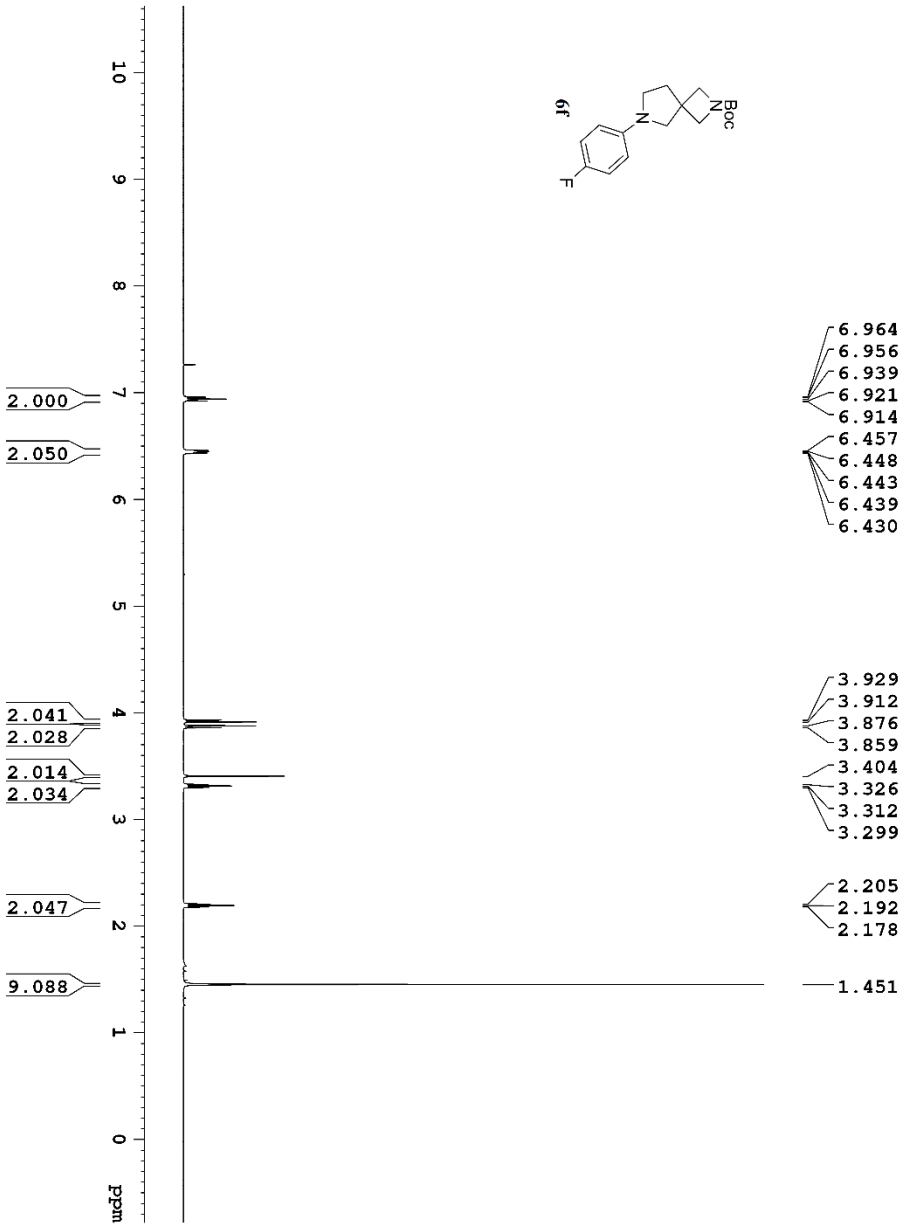

BocN
N
F
6c
157.269
156.464
155.392
143.405
143.393
116.115
115.938
113.038
112.979
79.905
65.009
59.271
46.525
29.400
28.448
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
ppm

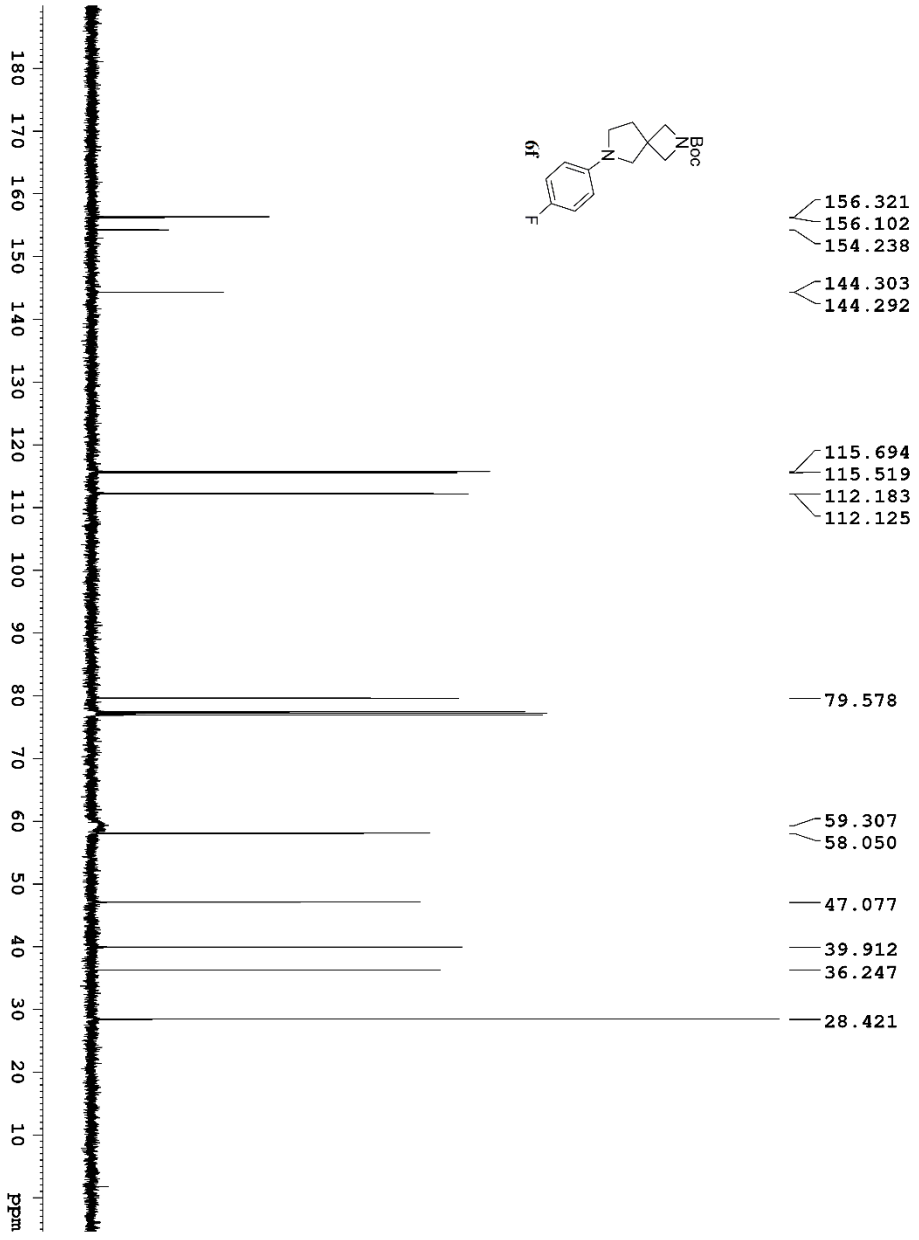

6d
BocN
N
7.136
7.122
7.108
6.887
6.872
6.863
6.857
6.847
3.943
3.926
3.901
3.884
3.340
3.258
3.245
3.231
2.307
2.162
2.148
2.134
1.464
2.000
2.015
4.130
2.039
2.013
3.059
2.065
9.009
ppm

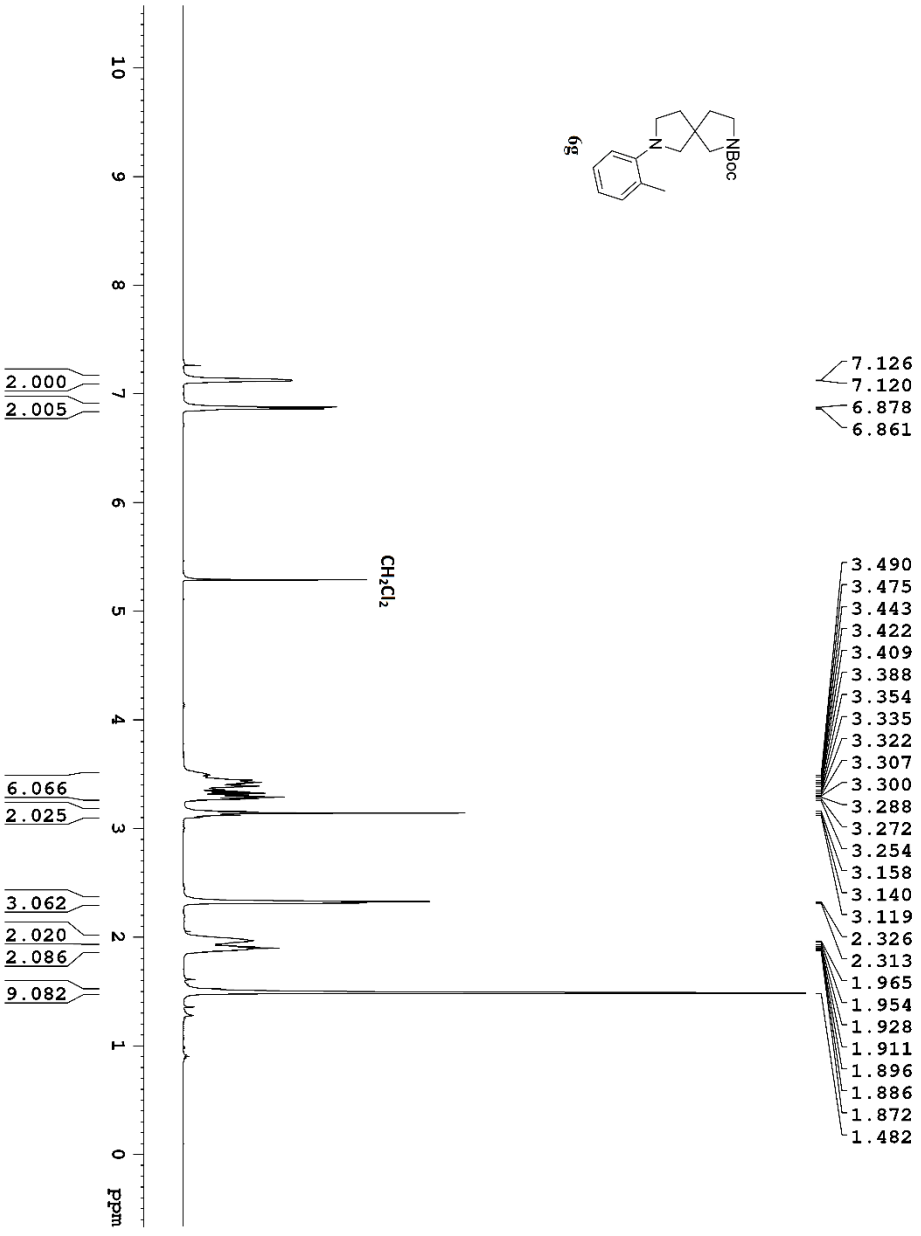

6d
BocN
N
156.418
148.559
131.782
129.102
126.456
121.063
115.947
79.541
61.425
59.787
50.079
39.655
36.668
28.494
20.316
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

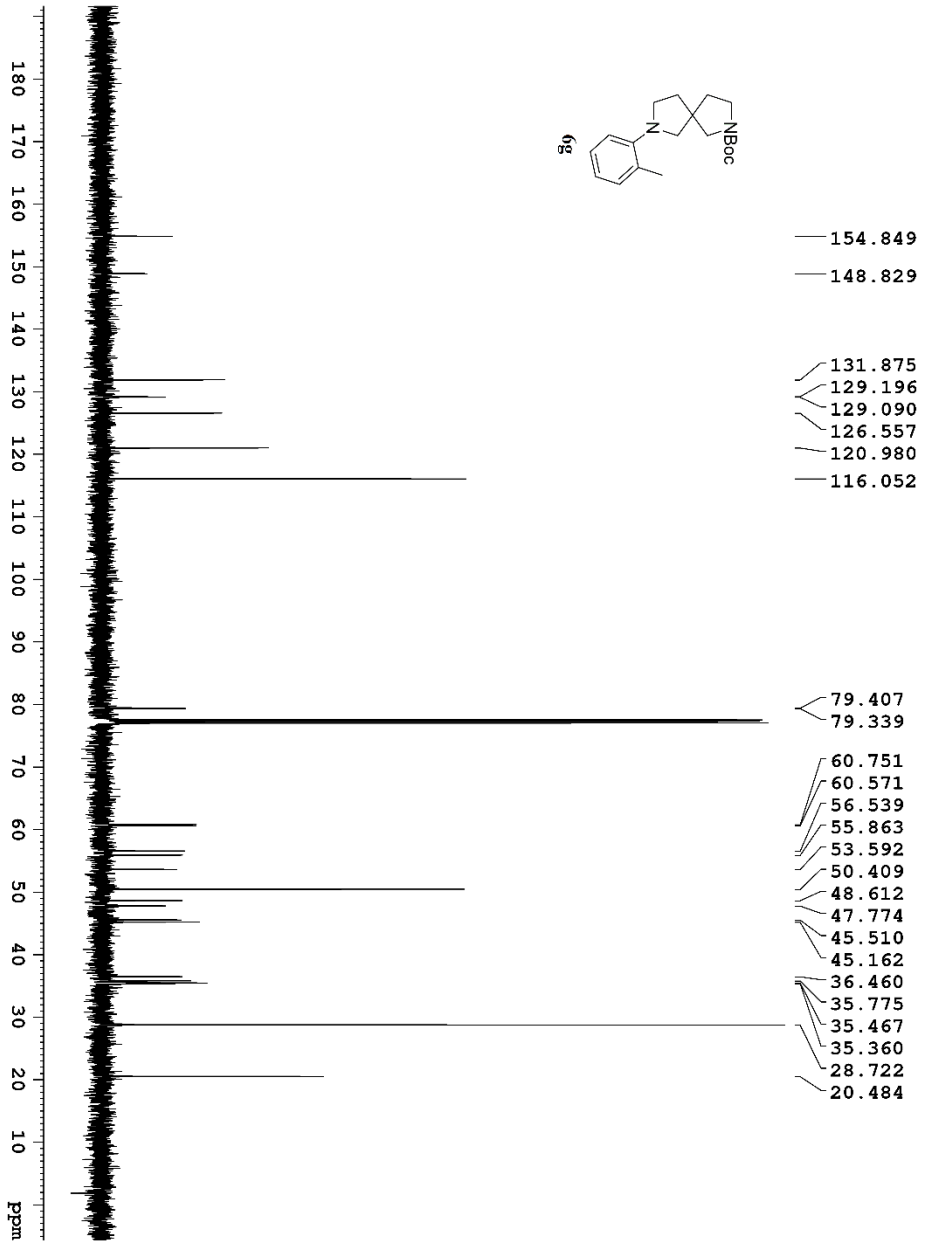

6e
BocN
N
OMe
6.862
6.855
6.850
6.841
6.837
6.829
6.517
6.509
6.505
6.496
6.491
6.484
3.929
3.911
3.871
3.854
3.754
3.397
3.319
3.305
3.291
2.192
2.178
2.165
1.454
2.000
2.003
2.030
2.027
3.003
1.998
2.020
2.013
9.128
ppm

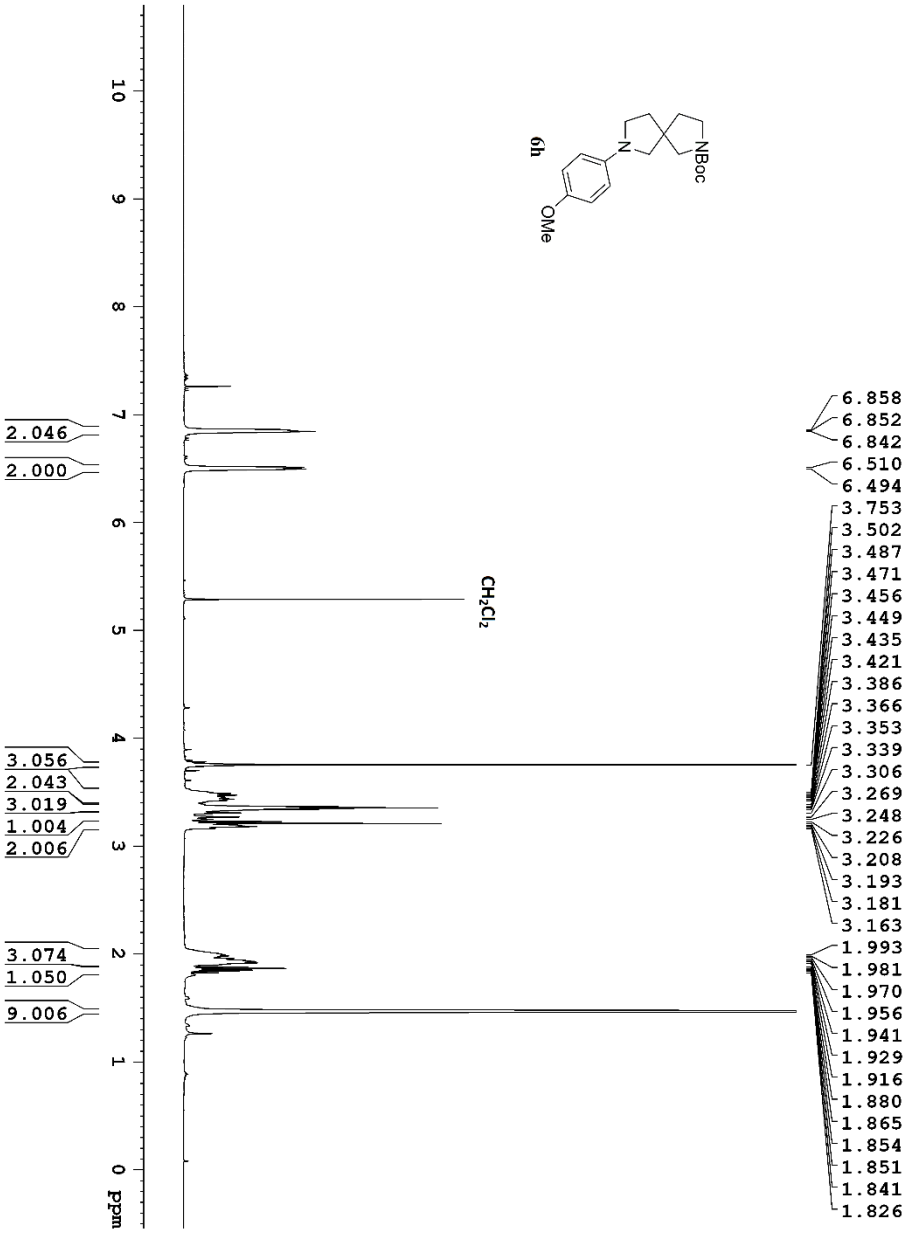

BocN
N
OMe
6g
156.317
151.254
142.574
115.057
112.580
79.497
77.409
77.155
76.900
59.252
58.277
55.928
47.228
39.872
36.318
28.419
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

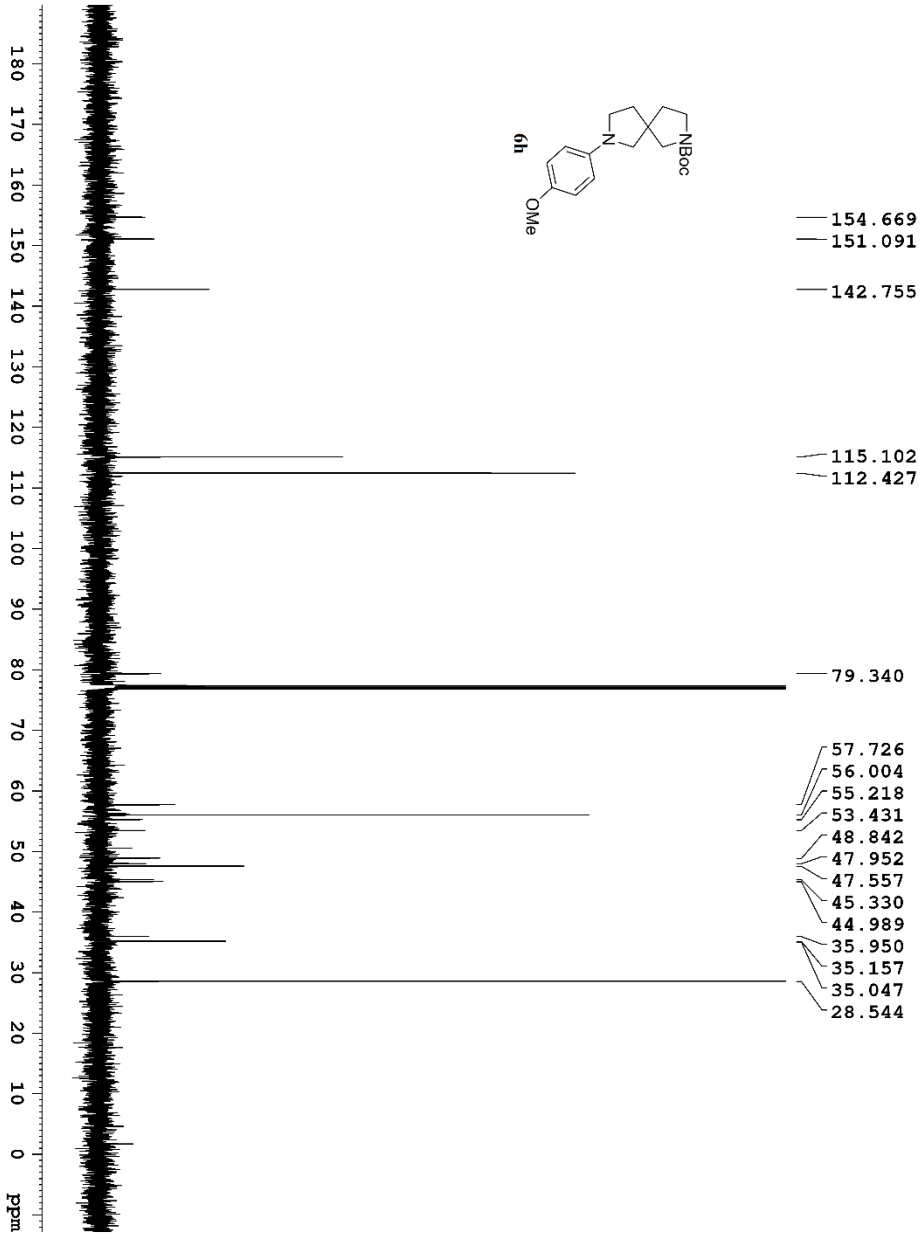

6f
Boc
N
N
F
6.964
6.956
6.939
6.921
6.914
6.457
6.448
6.443
6.439
6.430
3.929
3.912
3.876
3.859
3.404
3.326
3.312
3.299
2.205
2.192
2.178
1.451
2.000
2.050
2.041
2.028
2.014
2.034
2.047
9.088
ppm

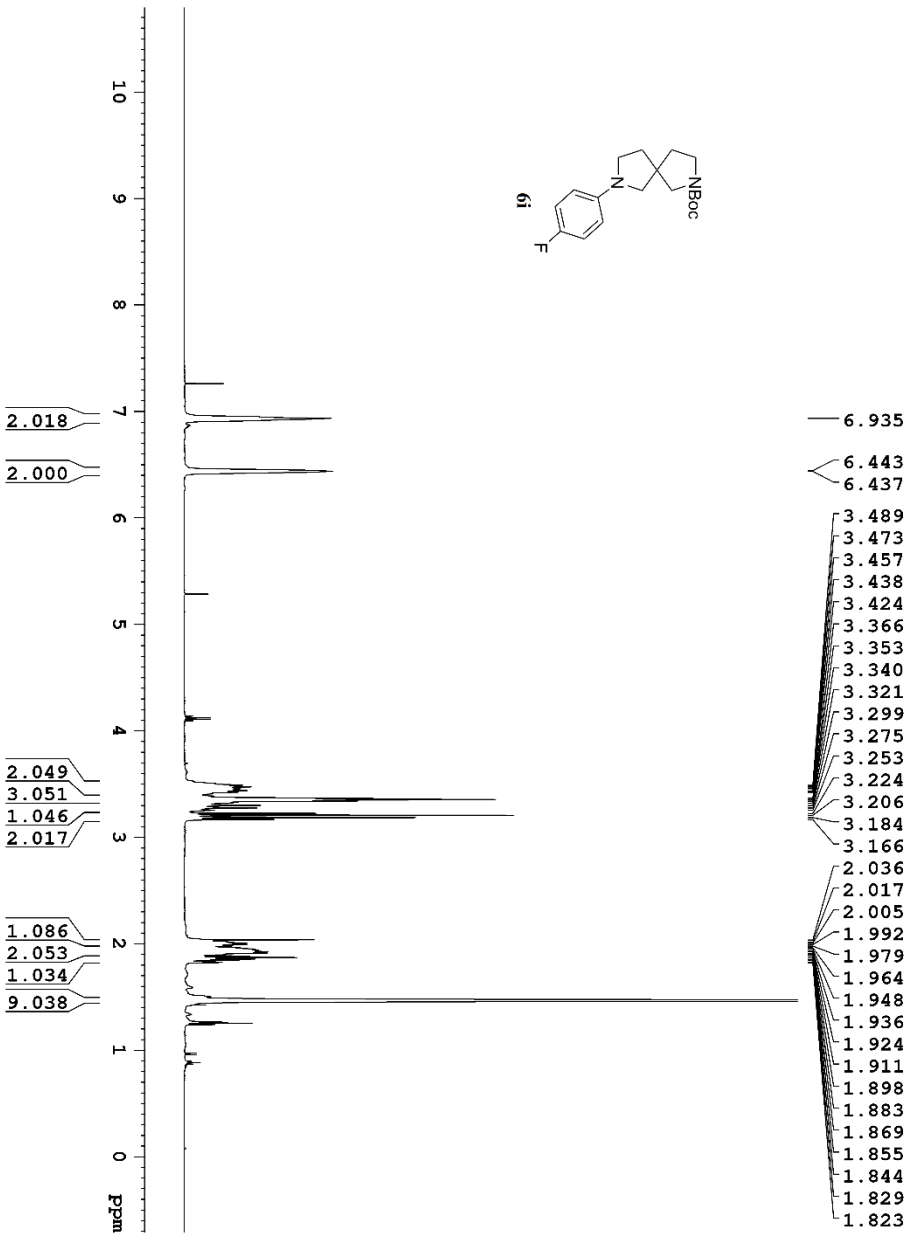

6f
Boc
N
N
F
156.321
156.102
154.238
144.303
144.292
115.694
115.519
112.183
112.125
79.578
59.307
58.050
47.077
39.912
36.247
28.421
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

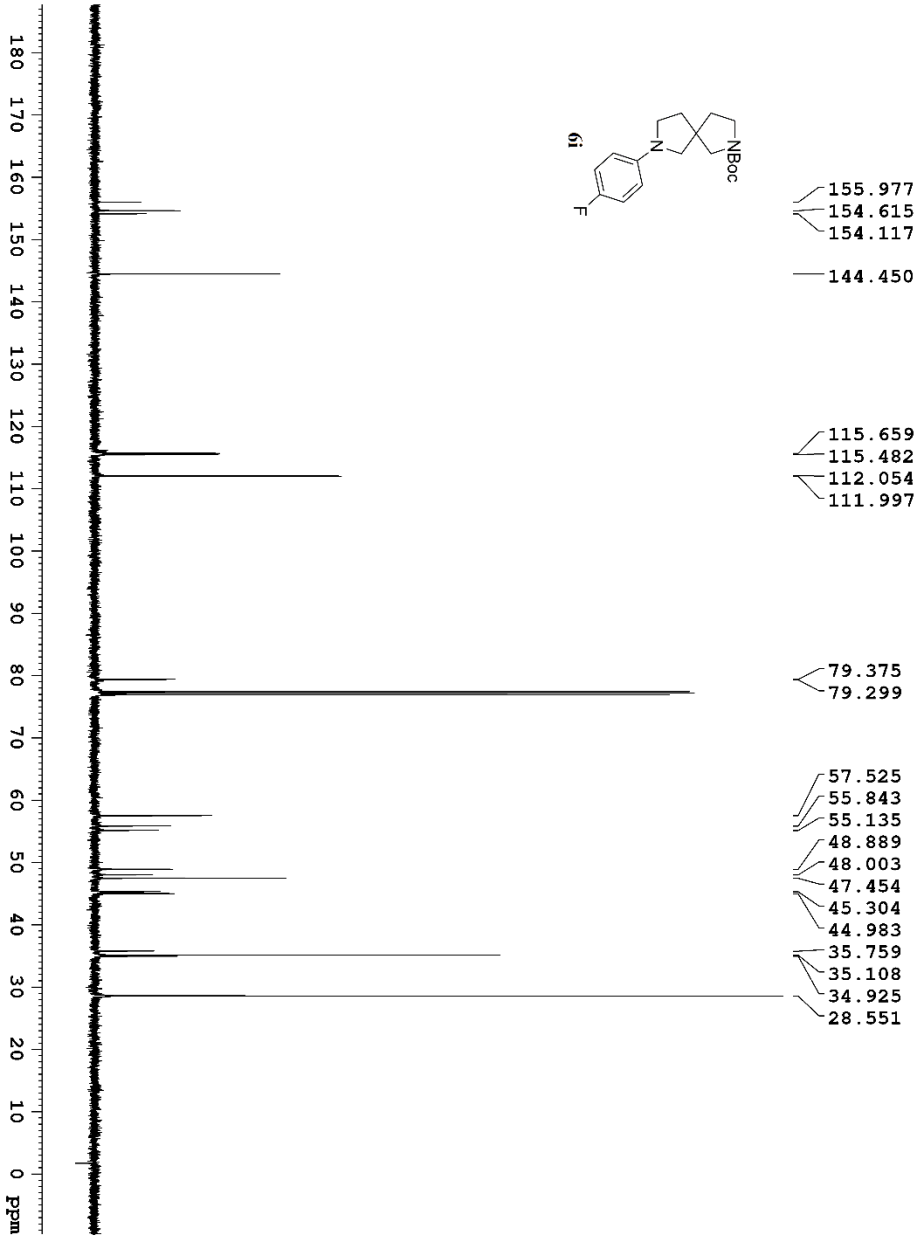

6g
N
NBoc
CH2Cl2
2.000
2.005
6.066
2.025
3.062
2.020
2.086
9.082
7.126
7.120
6.878
6.861
3.490
3.475
3.443
3.422
3.409
3.388
3.354
3.335
3.322
3.307
3.300
3.288
3.272
3.254
3.158
3.140
3.119
2.326
2.313
1.965
1.954
1.928
1.911
1.896
1.886
1.872
1.482
ppm

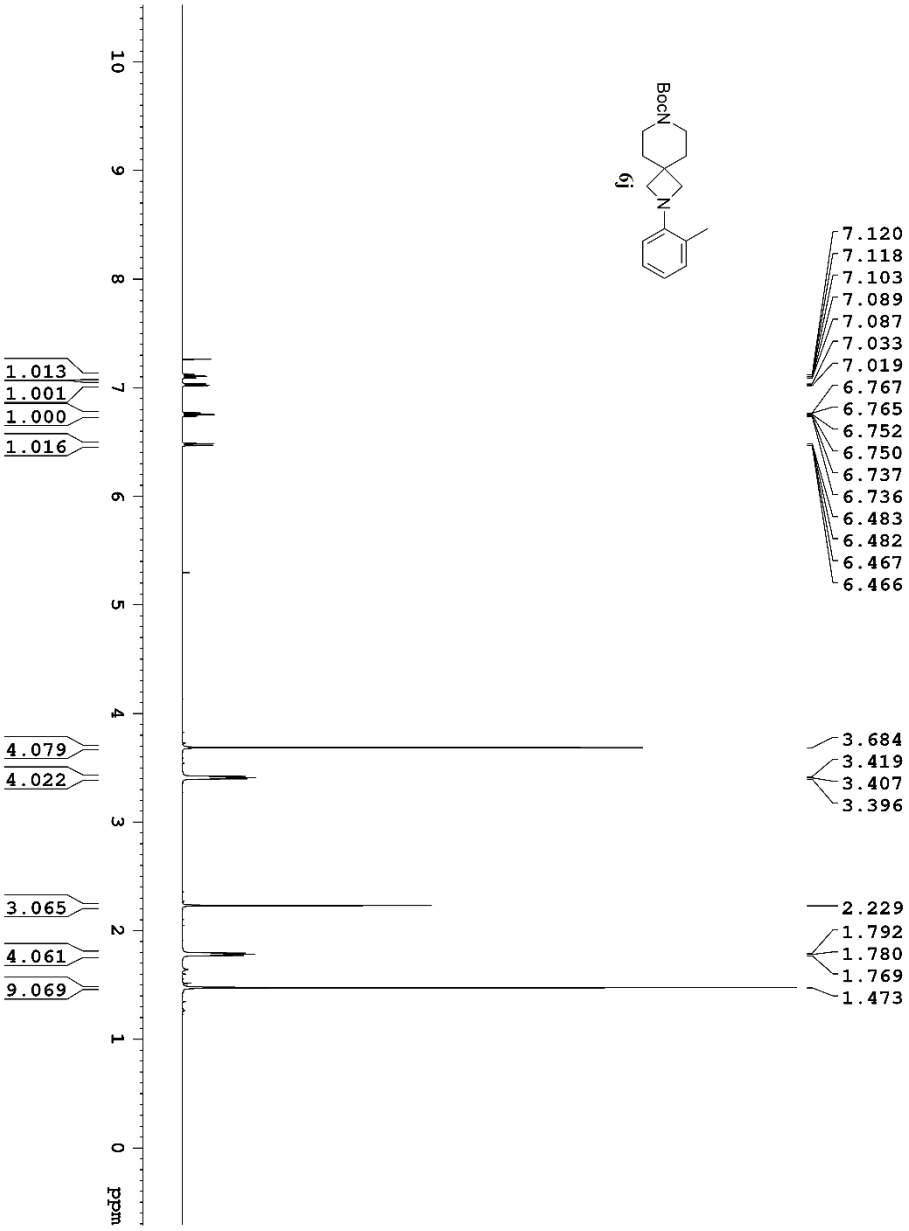

6g
N
NBoc
154.849
148.829
131.875
129.196
129.090
126.557
120.980
116.052
79.407
79.339
60.751
60.571
56.539
55.863
53.592
50.409
48.612
47.774
45.510
45.162
36.460
35.775
35.467
35.360
28.722
20.484
ppm

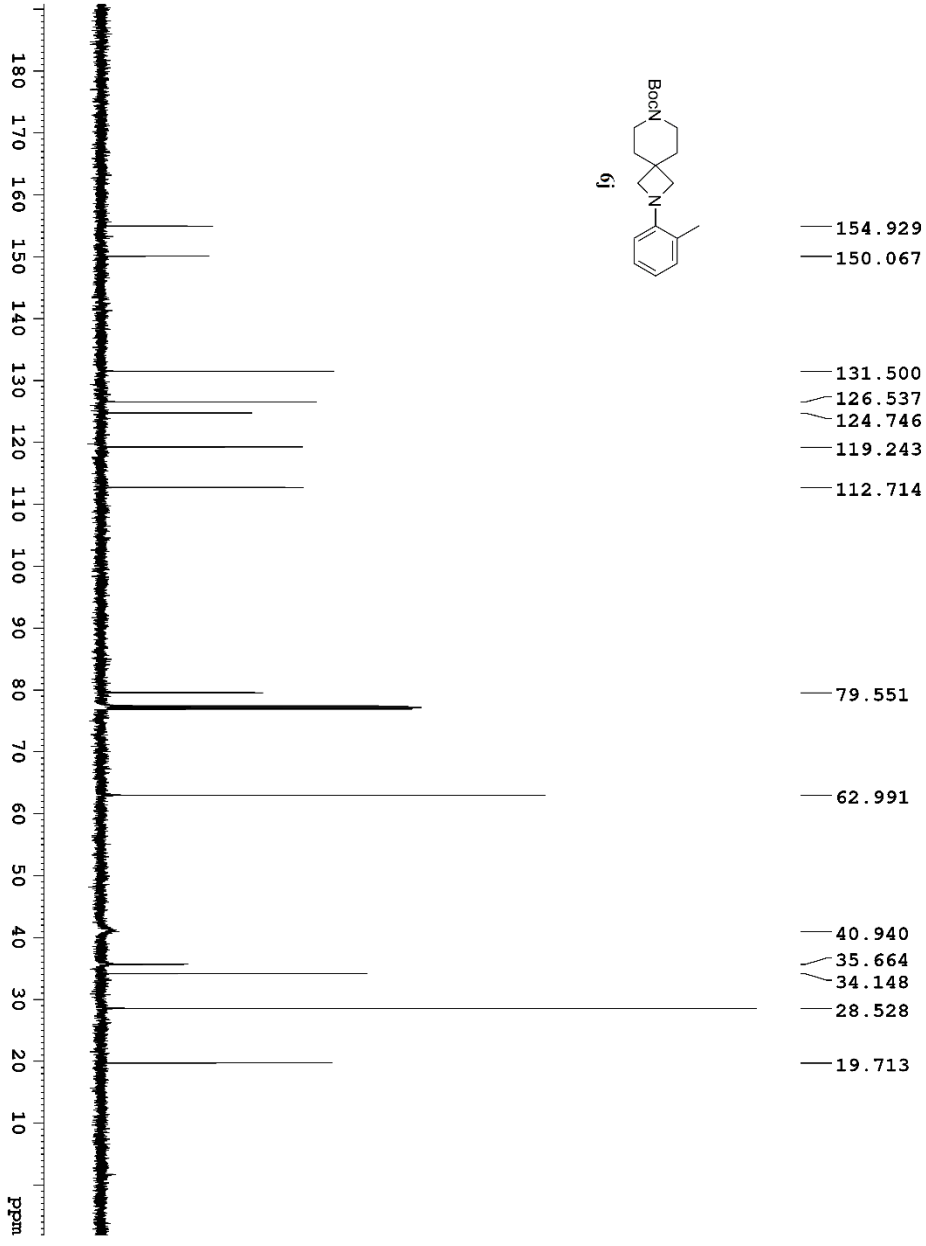

6h
NBoc
N
OMe
$CH_2Cl_2$
2.046
2.000
3.056
2.043
3.019
1.004
2.006
3.074
1.050
9.006
6.858
6.852
6.842
6.510
6.494
3.753
3.502
3.487
3.471
3.456
3.449
3.435
3.421
3.386
3.366
3.353
3.339
3.306
3.269
3.248
3.226
3.208
3.193
3.181
3.163
1.993
1.981
1.970
1.956
1.941
1.929
1.916
1.880
1.865
1.854
1.851
1.841
1.826
ppm

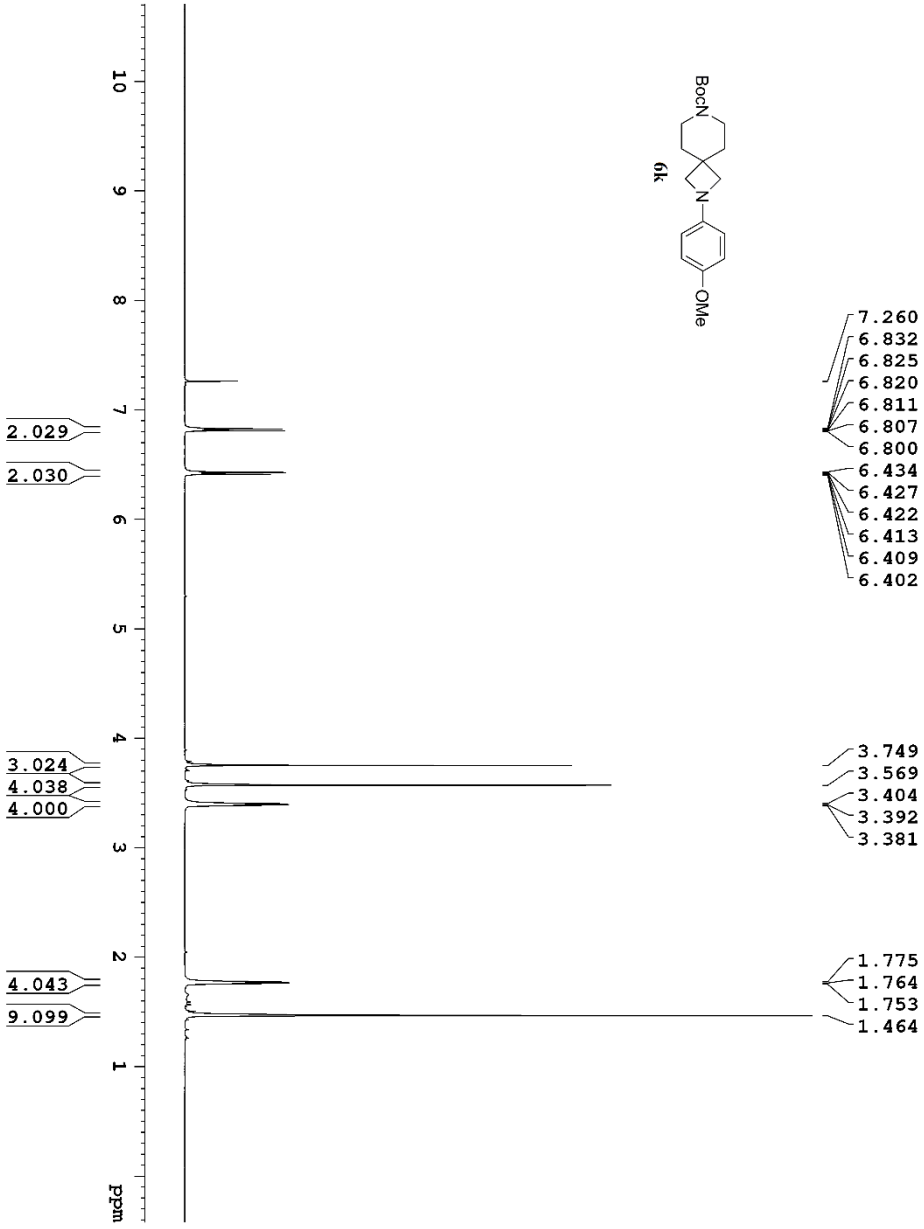

6h
N
NBoc
OMe
154.669
151.091
142.755
115.102
112.427
79.340
57.726
56.004
55.218
53.431
48.842
47.952
47.557
45.330
44.989
35.950
35.157
35.047
28.544
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

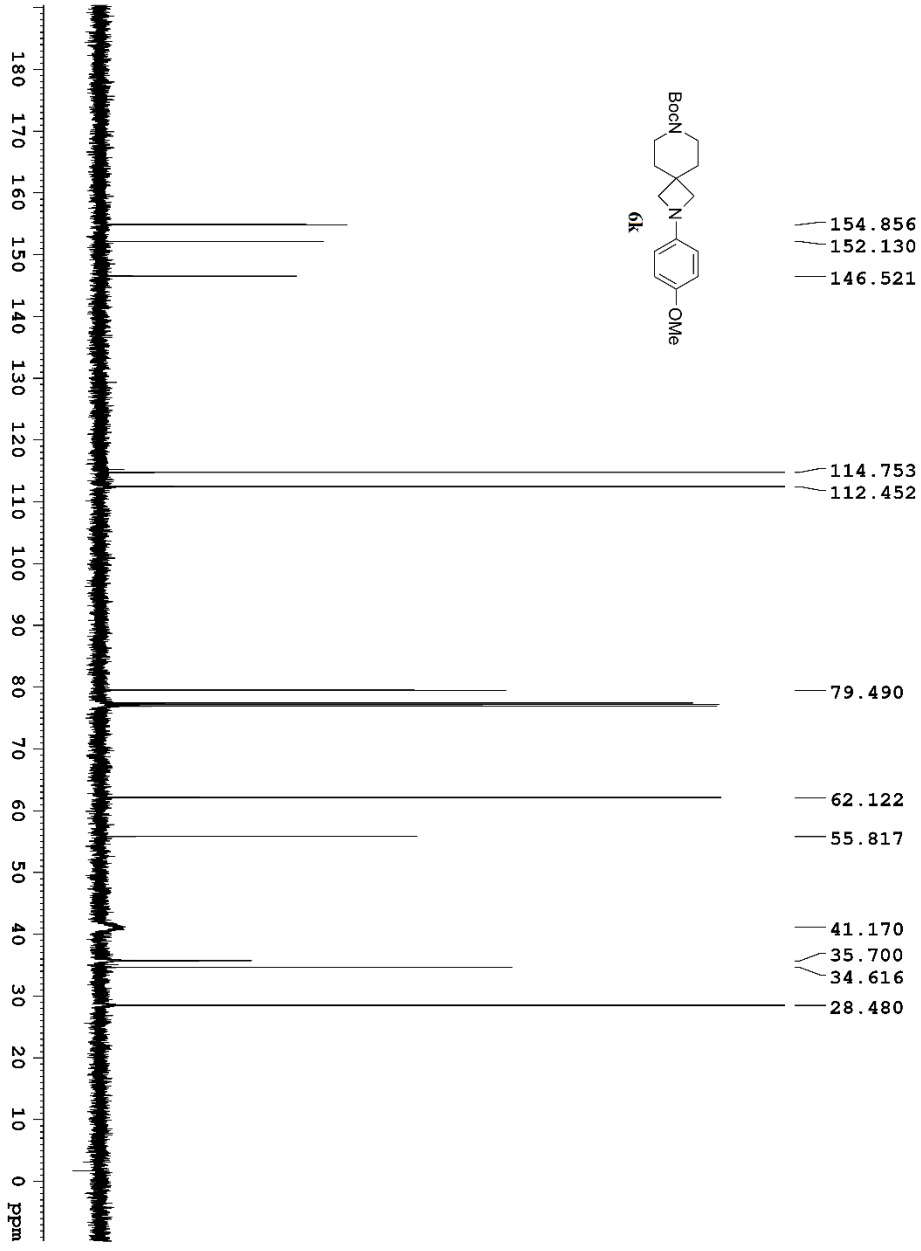

6.935
6.443
6.437
3.489
3.473
3.457
3.438
3.424
3.366
3.353
3.340
3.321
3.299
3.275
3.253
3.224
3.206
3.184
3.166
2.036
2.017
2.005
1.992
1.979
1.964
1.948
1.936
1.924
1.911
1.898
1.883
1.869
1.855
1.844
1.829
1.823
2.018
2.000
2.049
3.051
1.046
2.017
1.086
2.053
1.034
9.038
ppm
NBoc
F
6j

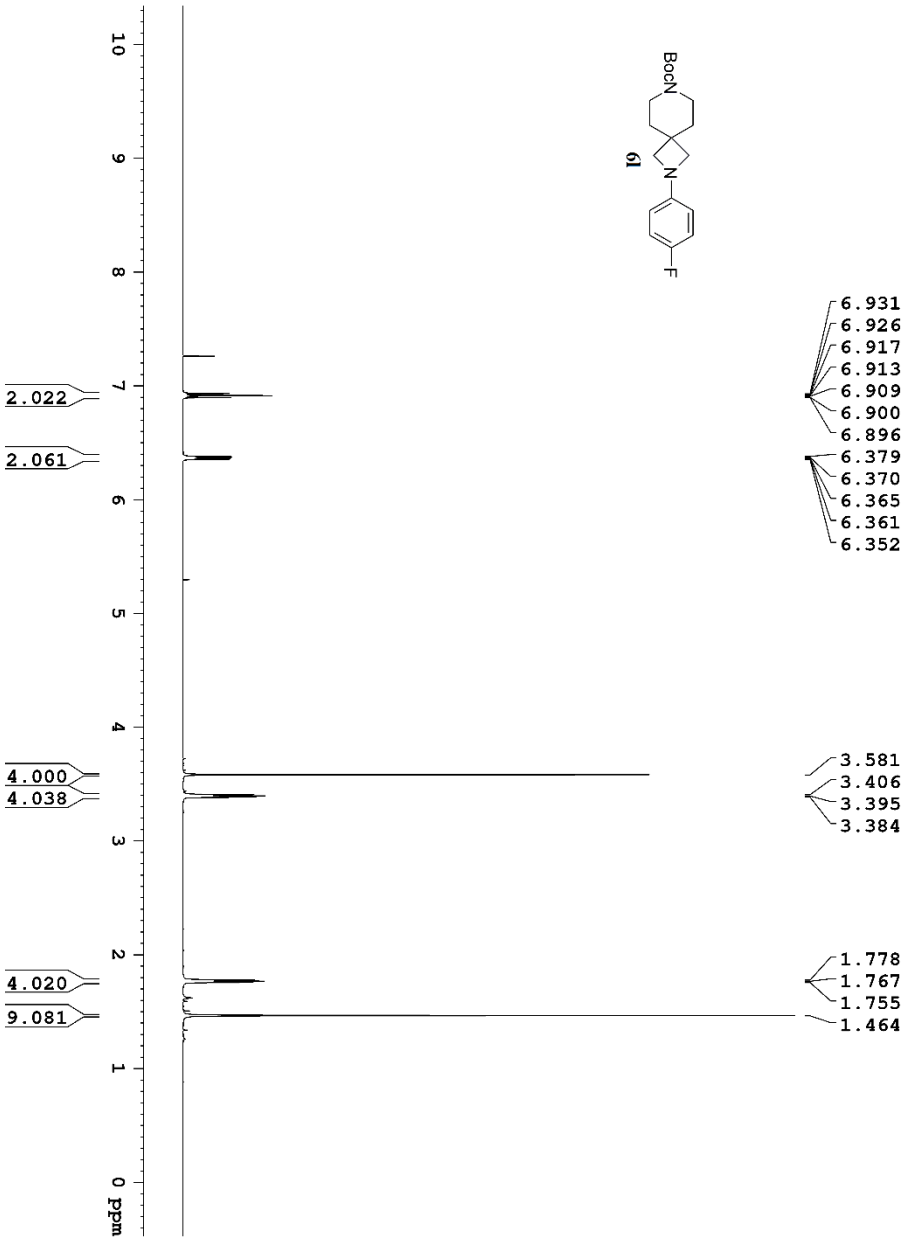

6i
N
NBoc
F
155.977
154.615
154.117
144.450
115.659
115.482
112.054
111.997
79.375
79.299
57.525
55.843
55.135
48.889
48.003
47.454
45.304
44.983
35.759
35.108
34.925
28.551
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

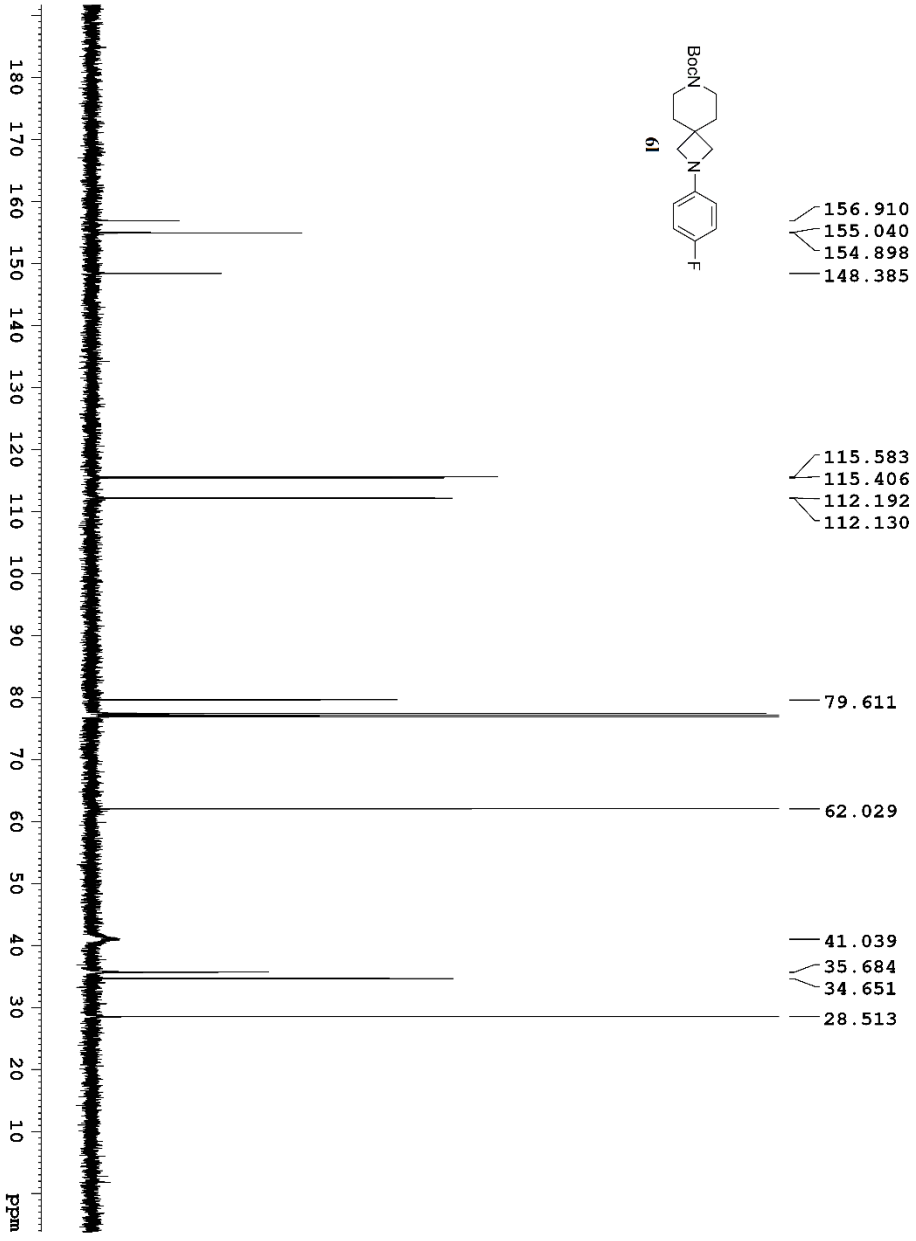

BocN
N
fg
7.120
7.118
7.103
7.089
7.087
7.033
7.019
6.767
6.765
6.752
6.750
6.737
6.736
6.483
6.482
6.467
6.466
3.684
3.419
3.407
3.396
2.229
1.792
1.780
1.769
1.473
1.013
1.001
1.000
1.016
4.079
4.022
3.065
4.061
9.069
ppm

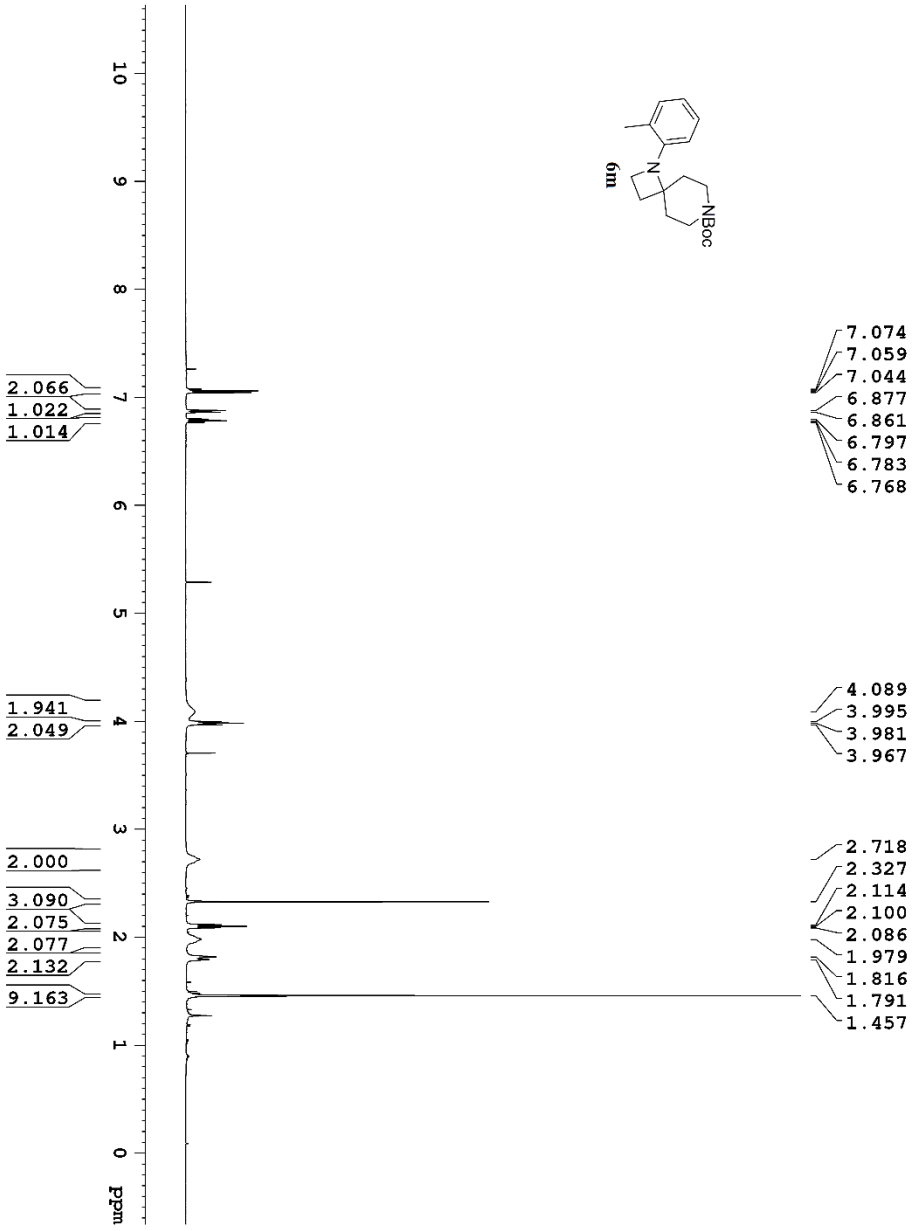

6j
BocN
N
154.929
150.067
131.500
126.537
124.746
119.243
112.714
79.551
62.991
40.940
35.664
34.148
28.528
19.713
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
ppm

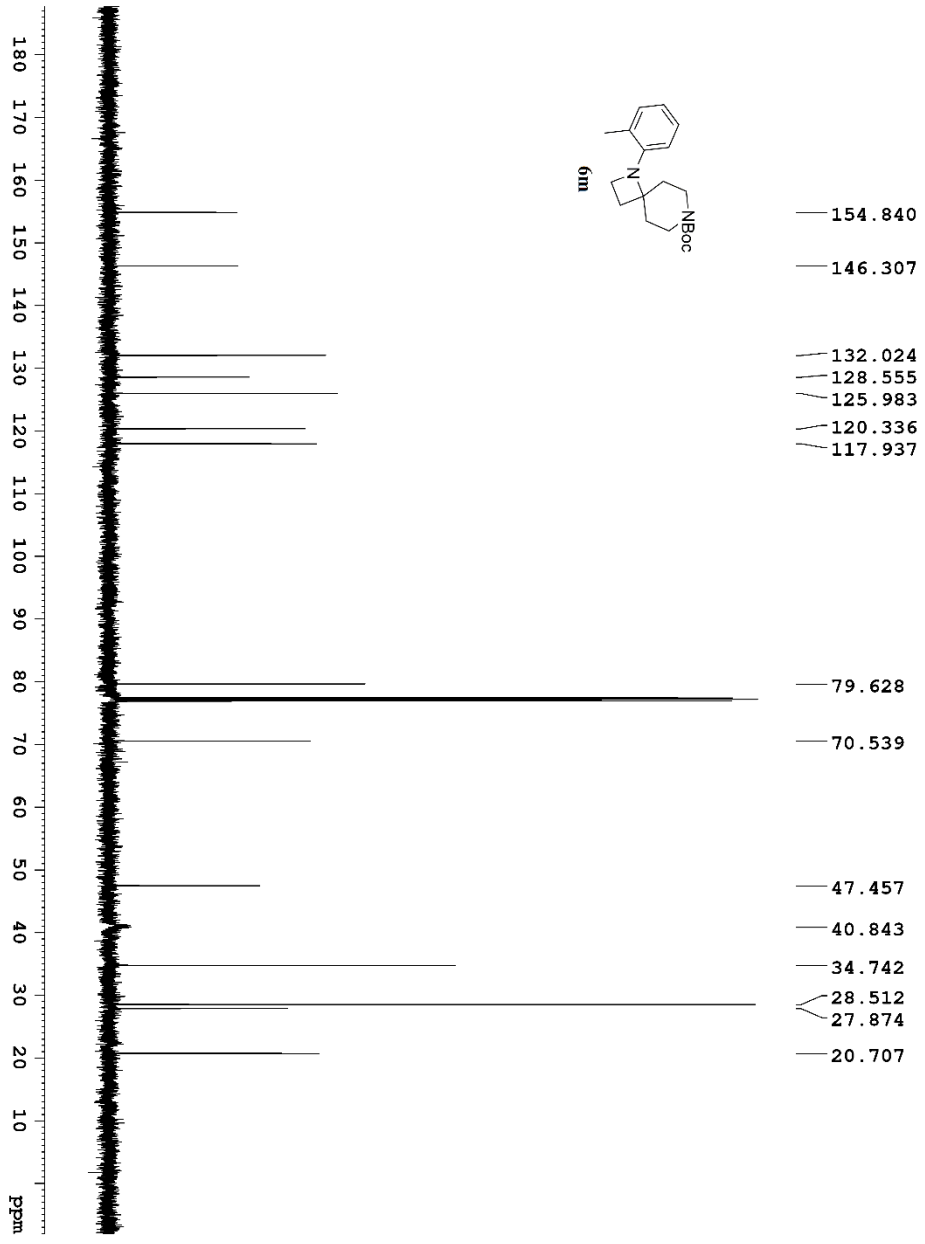

BocN
N
OMe
6k
7.260
6.832
6.825
6.820
6.811
6.807
6.800
6.434
6.427
6.422
6.413
6.409
6.402
3.749
3.569
3.404
3.392
3.381
1.775
1.764
1.753
1.464
2.029
2.030
3.024
4.038
4.000
4.043
9.099
ppm

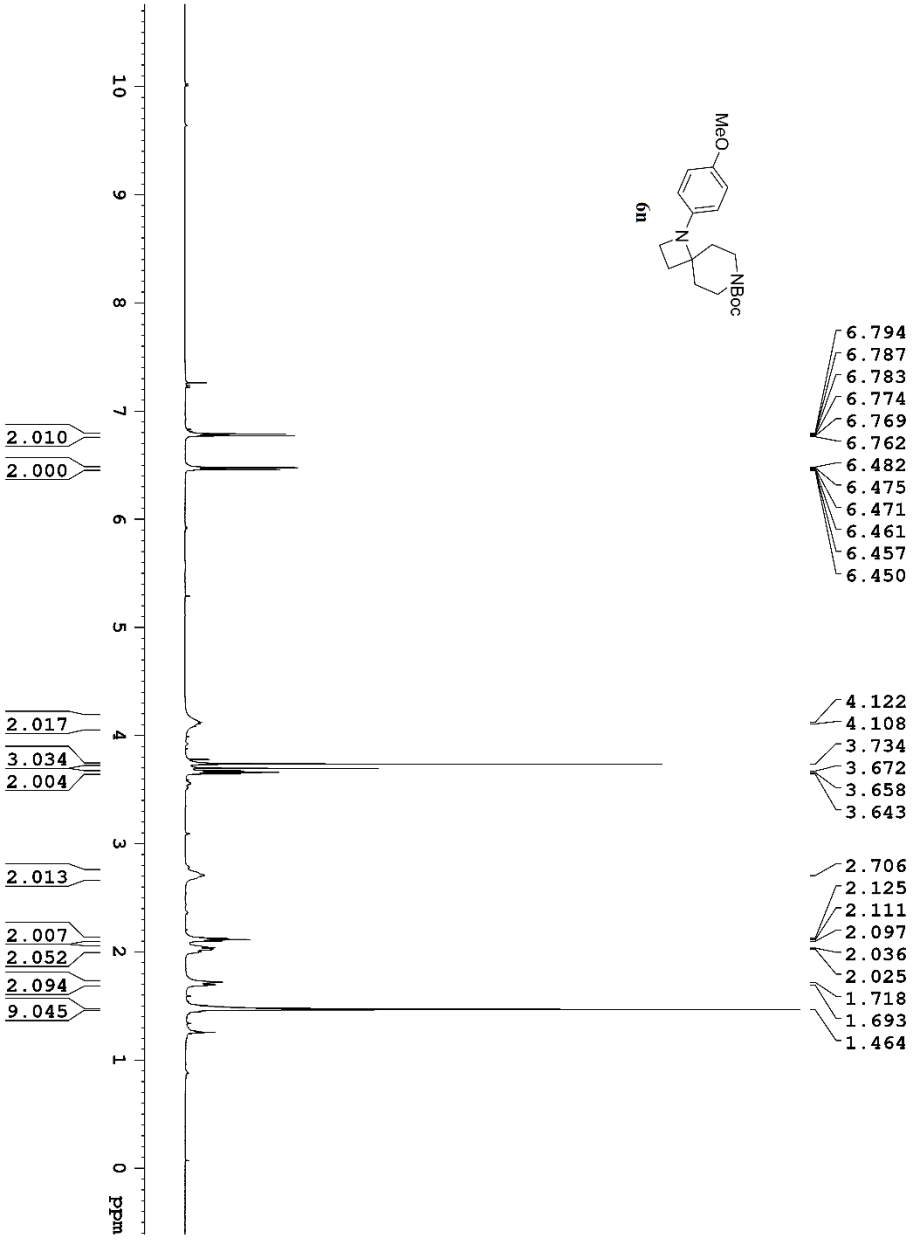

BocN
N
OMe
6k
154.856
152.130
146.521
114.753
112.452
79.490
62.122
55.817
41.170
35.700
34.616
28.480
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

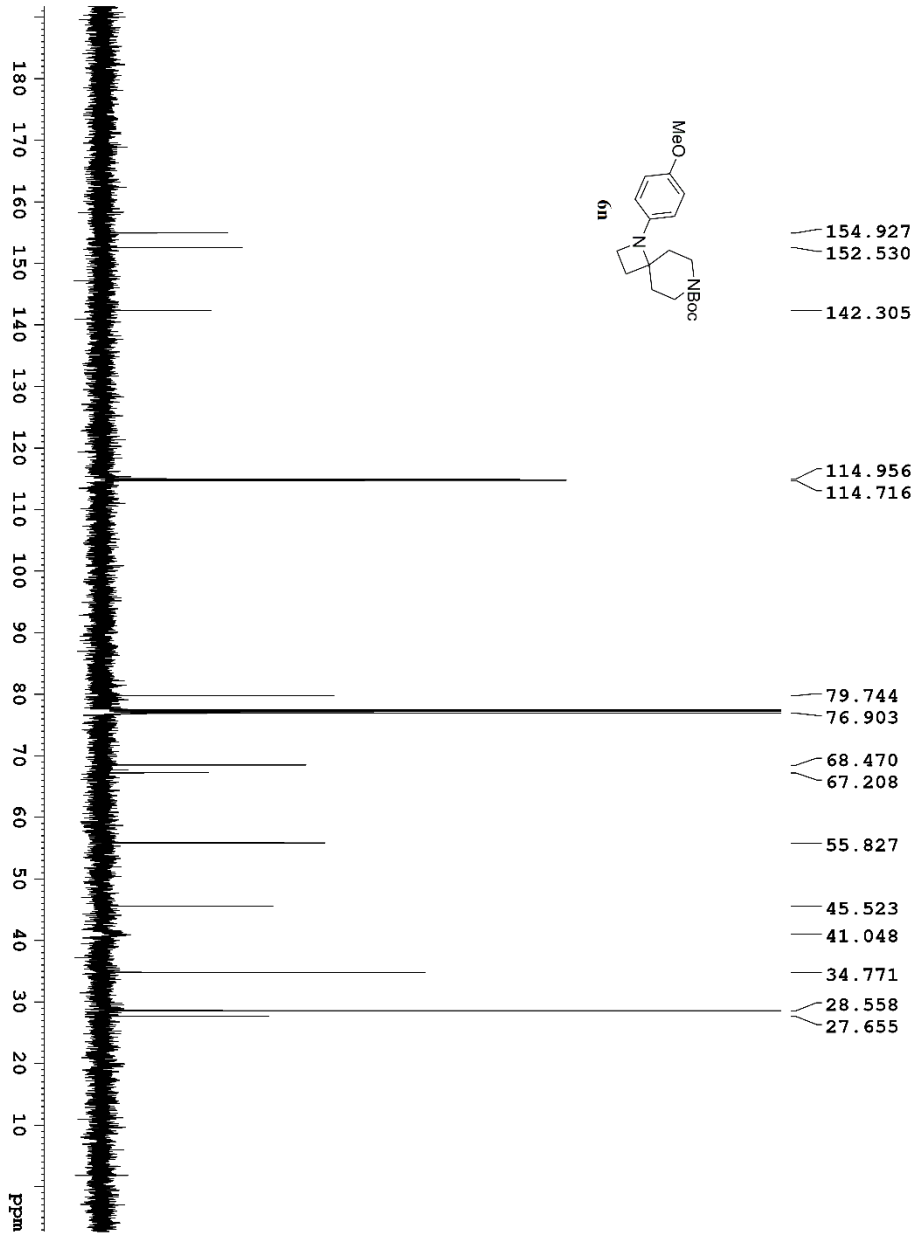

BocN
N
F
61
6.931
6.926
6.917
6.913
6.909
6.900
6.896
6.379
6.370
6.365
6.361
6.352
3.581
3.406
3.395
3.384
1.778
1.767
1.755
1.464
2.022
2.061
4.000
4.038
4.020
9.081
ppm

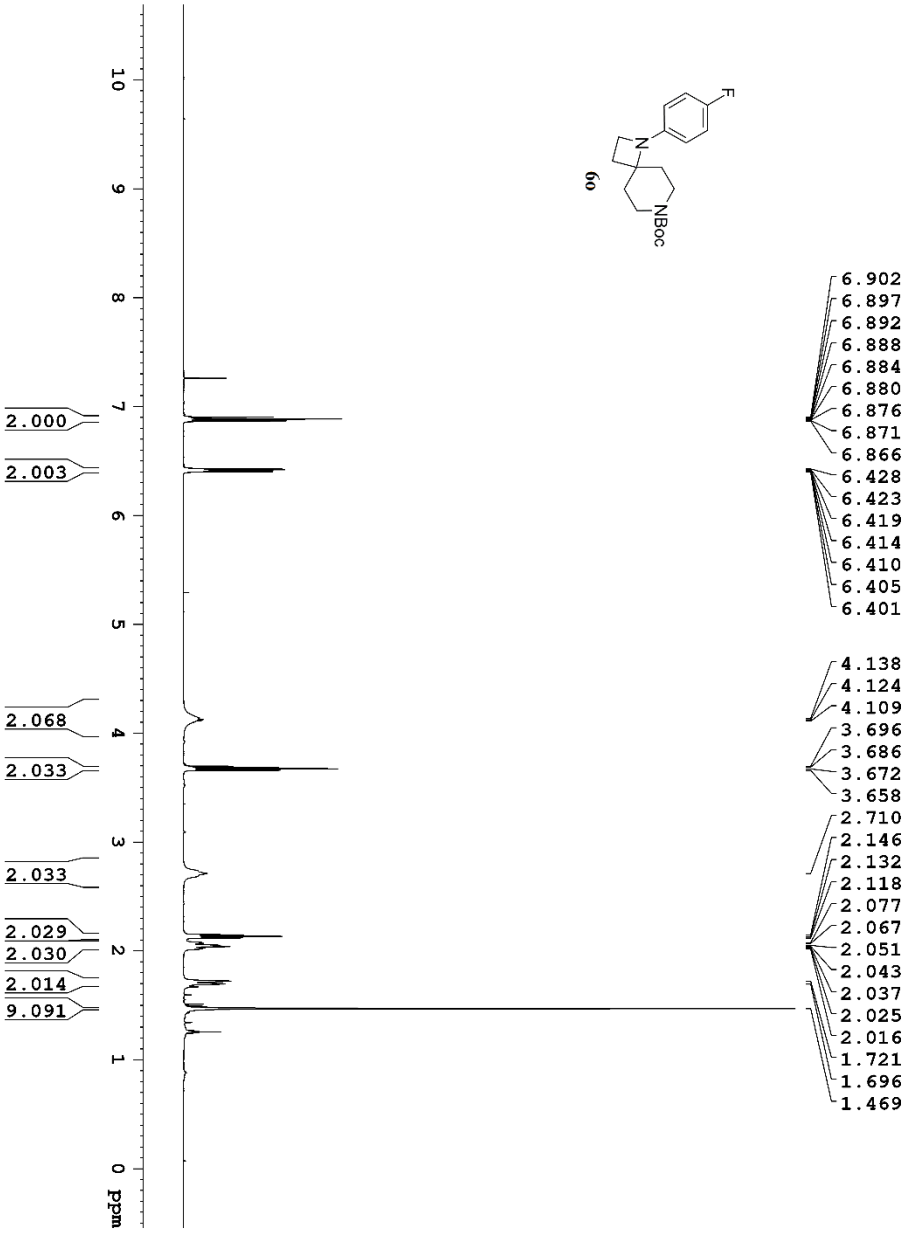

BocN
N
F
61
156.910
155.040
154.898
148.385
115.583
115.406
112.192
112.130
79.611
62.029
41.039
35.684
34.651
28.513
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
ppm

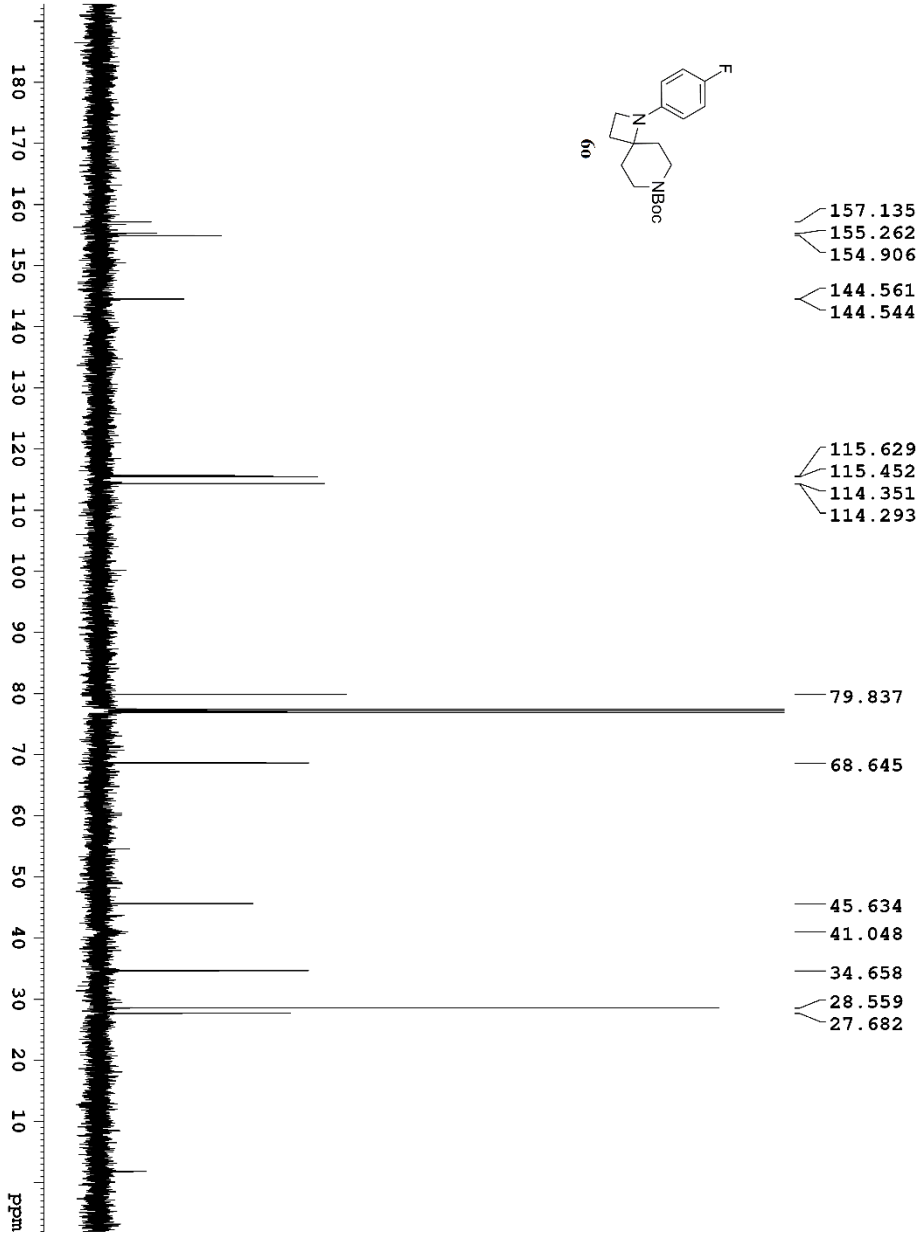

6m
N
NBoc
7.074
7.059
7.044
6.877
6.861
6.797
6.783
6.768
4.089
3.995
3.981
3.967
2.718
2.327
2.114
2.100
2.086
1.979
1.816
1.791
1.457
2.066
1.022
1.014
1.941
2.049
2.000
3.090
2.075
2.077
2.132
9.163
ppm

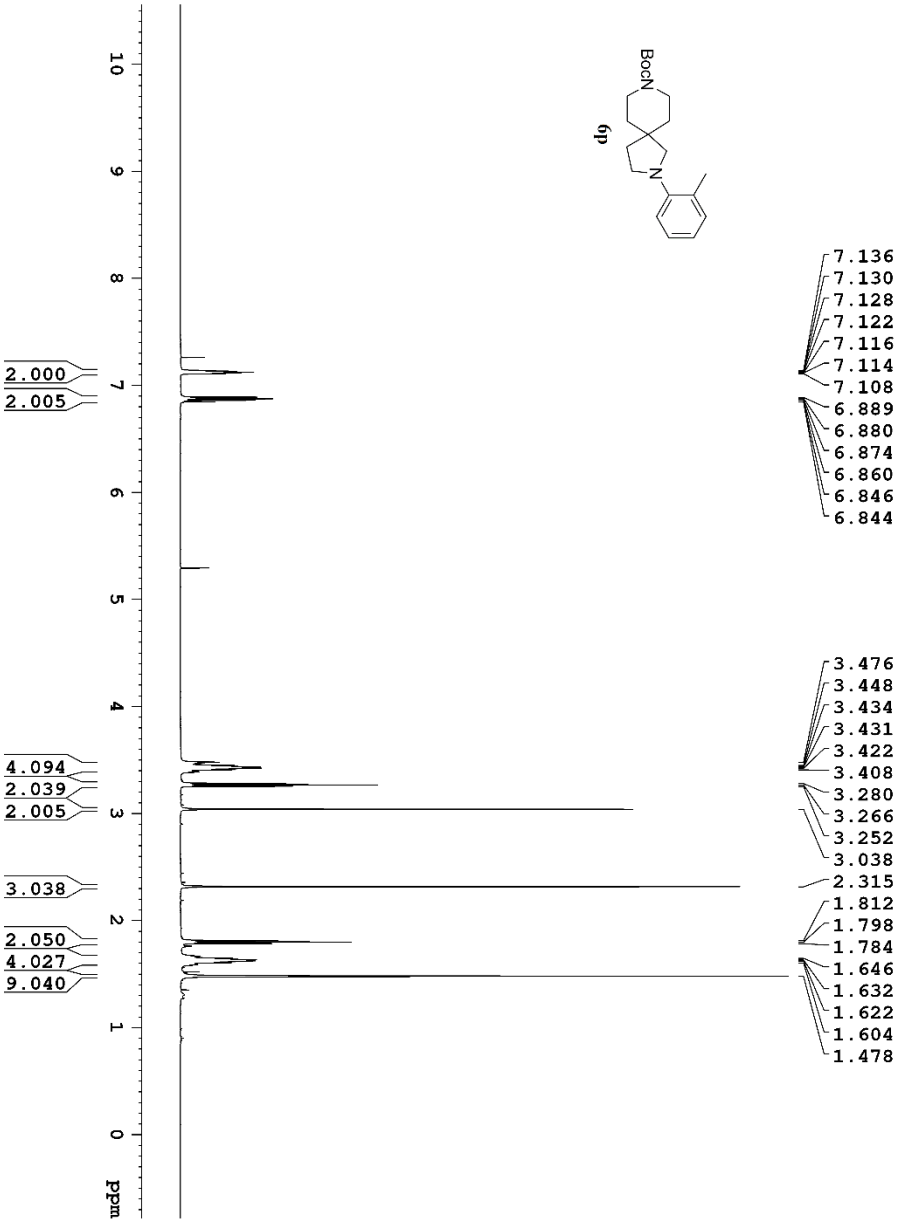

6m
N
NBoc
154.840
146.307
132.024
128.555
125.983
120.336
117.937
79.628
70.539
47.457
40.843
34.742
28.512
27.874
20.707
ppm

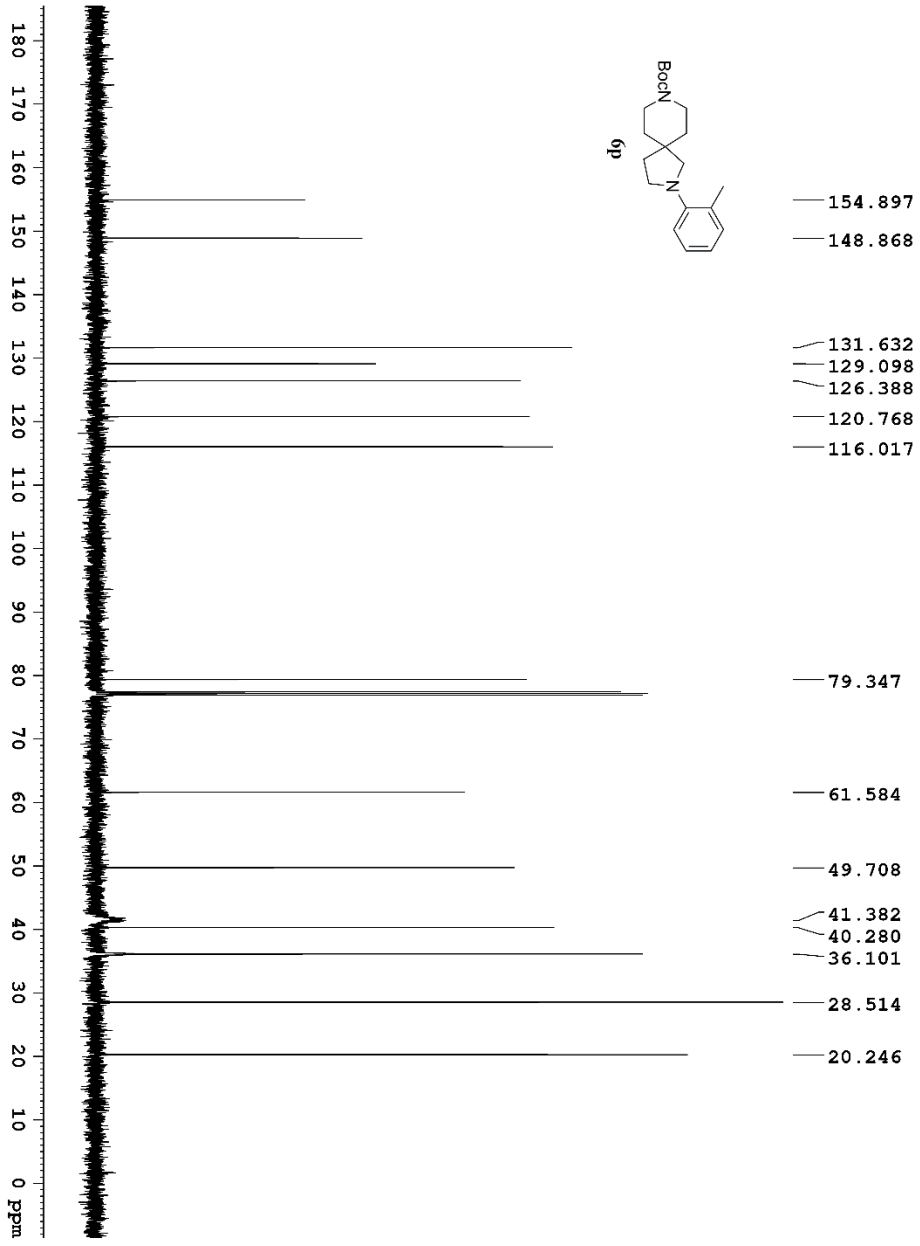

6n
MeO
N
NBoc
6.794
6.787
6.783
6.774
6.769
6.762
6.482
6.475
6.471
6.461
6.457
6.450
4.122
4.108
3.734
3.672
3.658
3.643
2.706
2.125
2.111
2.097
2.036
2.025
1.718
1.693
1.464
2.010
2.000
2.017
3.034
2.004
2.013
2.007
2.052
2.094
9.045
ppm

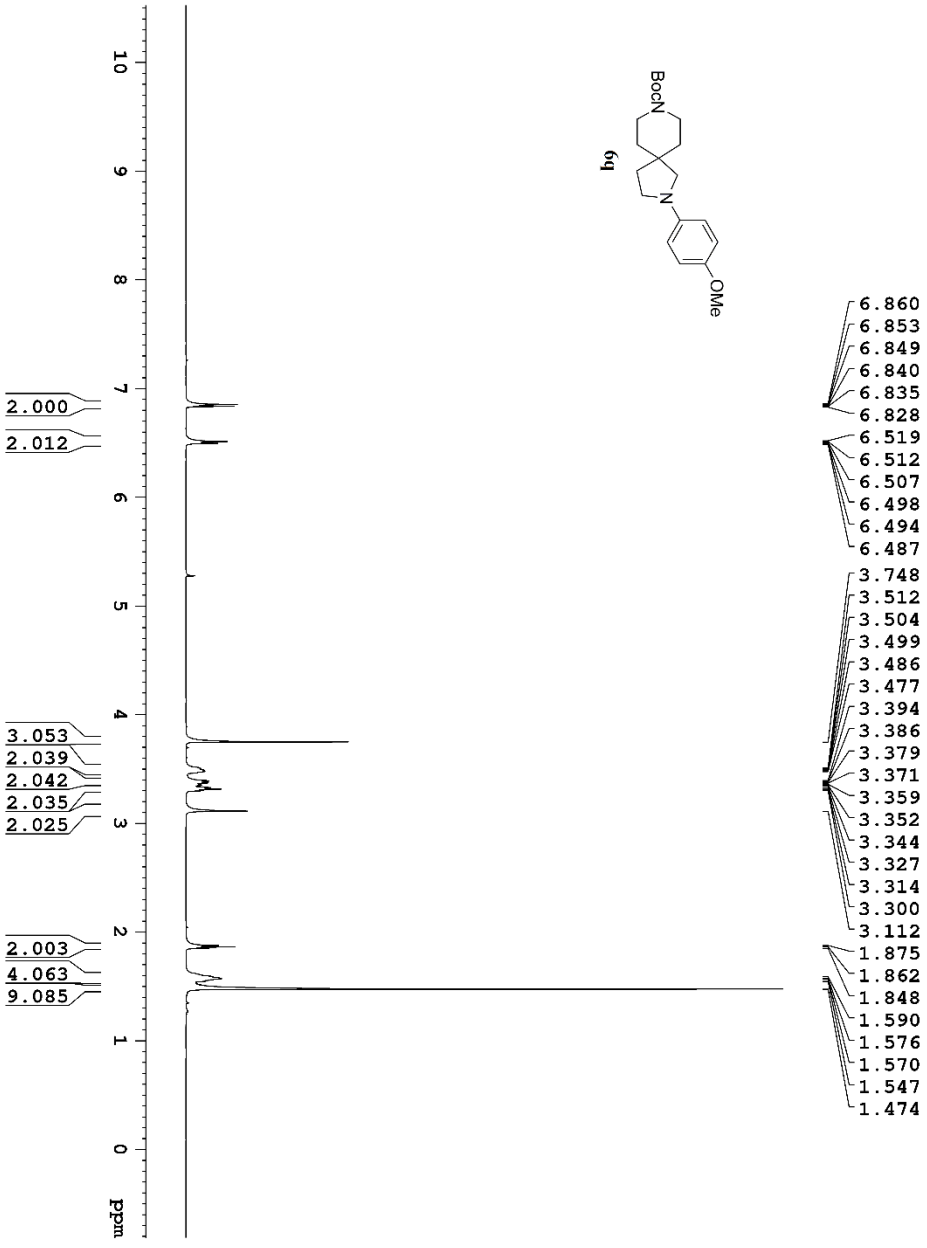

6m
MeO
N
NBoc
154.927
152.530
142.305
114.956
114.716
79.744
76.903
68.470
67.208
55.827
45.523
41.048
34.771
28.558
27.655
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
ppm


6g
F
N
NBoc
6.902
6.897
6.892
6.888
6.884
6.880
6.876
6.871
6.866
6.428
6.423
6.419
6.414
6.410
6.405
6.401
4.138
4.124
4.109
3.696
3.686
3.672
3.658
2.710
2.146
2.132
2.118
2.077
2.067
2.051
2.043
2.037
2.025
2.016
1.721
1.696
1.469
2.000
2.003
2.068
2.033
2.033
2.029
2.030
2.014
9.091
ppm


F
N
NBoc
6g
157.135
155.262
154.906
144.561
144.544
115.629
115.452
114.351
114.293
79.837
68.645
45.634
41.048
34.658
28.559
27.682
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
ppm


6p
BocN
N
7.136
7.130
7.128
7.122
7.116
7.114
7.108
6.889
6.880
6.874
6.860
6.846
6.844
3.476
3.448
3.434
3.431
3.422
3.408
3.280
3.266
3.252
3.038
2.315
1.812
1.798
1.784
1.646
1.632
1.622
1.604
1.478
2.000
2.005
4.094
2.039
2.005
3.038
2.050
4.027
9.040
ppm


6p
BocN
N
154.897
148.868
131.632
129.098
126.388
120.768
116.017
79.347
61.584
49.708
41.382
40.280
36.101
28.514
20.246
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


6q
BocN
N
OMe
6.860
6.853
6.849
6.840
6.835
6.828
6.519
6.512
6.507
6.498
6.494
6.487
3.748
3.512
3.504
3.499
3.486
3.477
3.394
3.386
3.379
3.371
3.359
3.352
3.344
3.327
3.314
3.300
3.112
1.875
1.862
1.848
1.590
1.576
1.570
1.547
1.474
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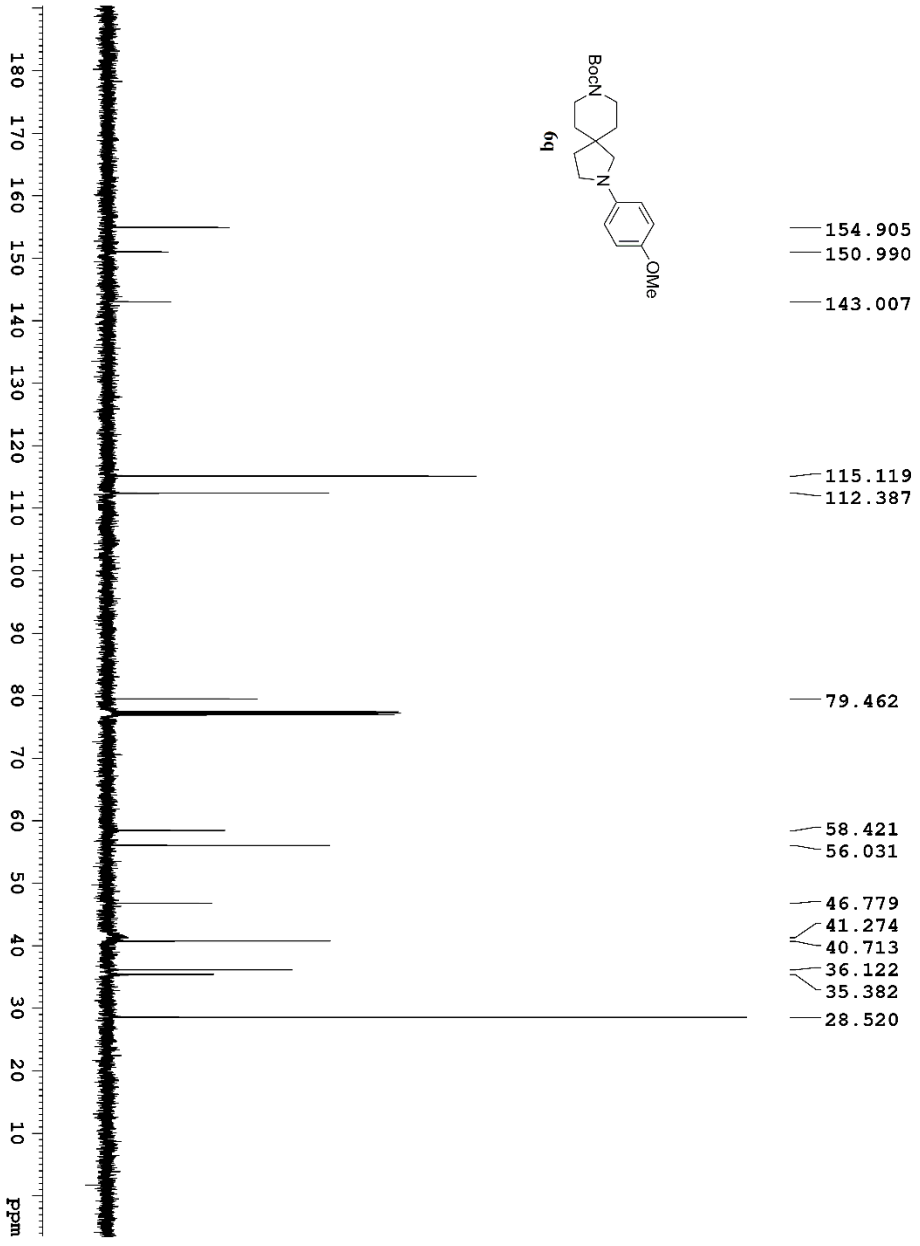
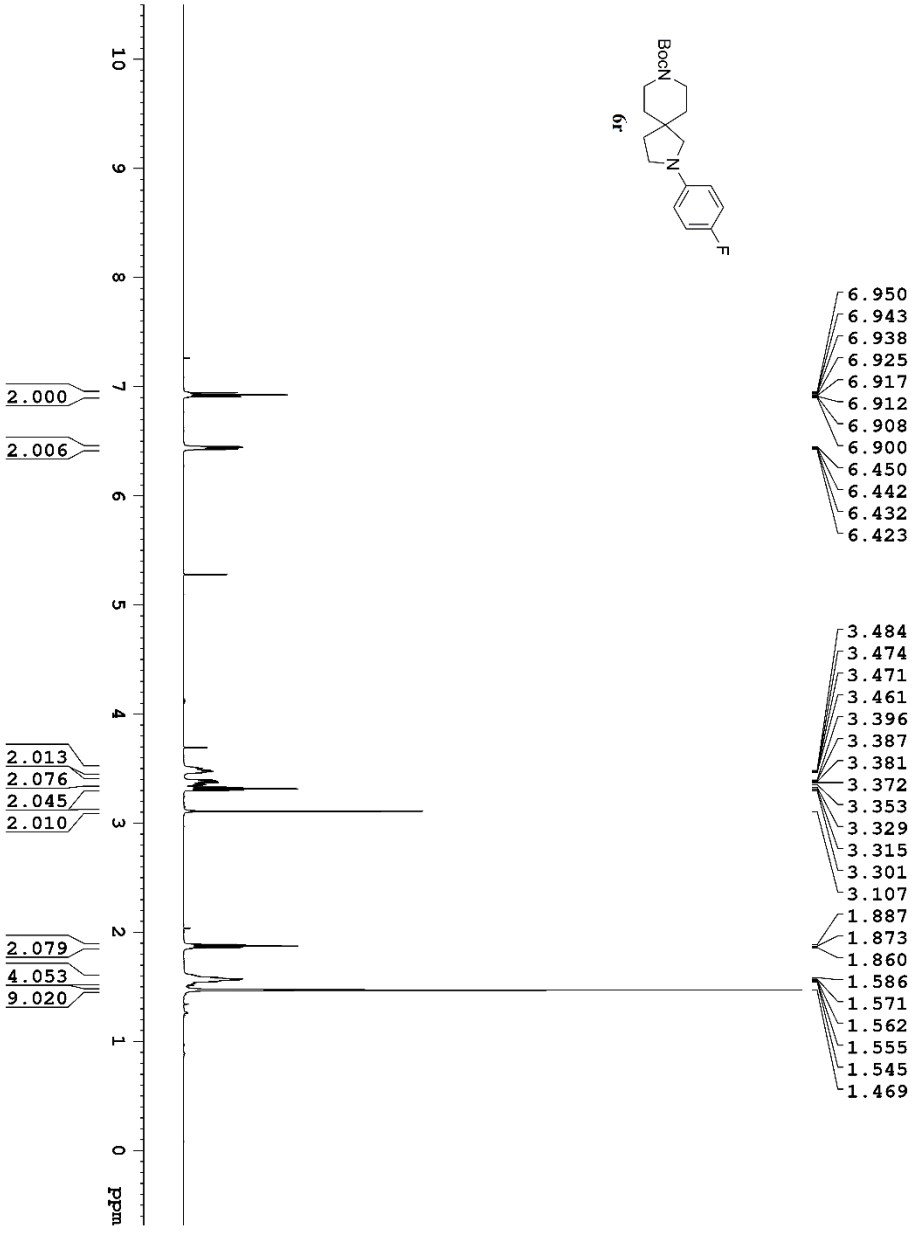
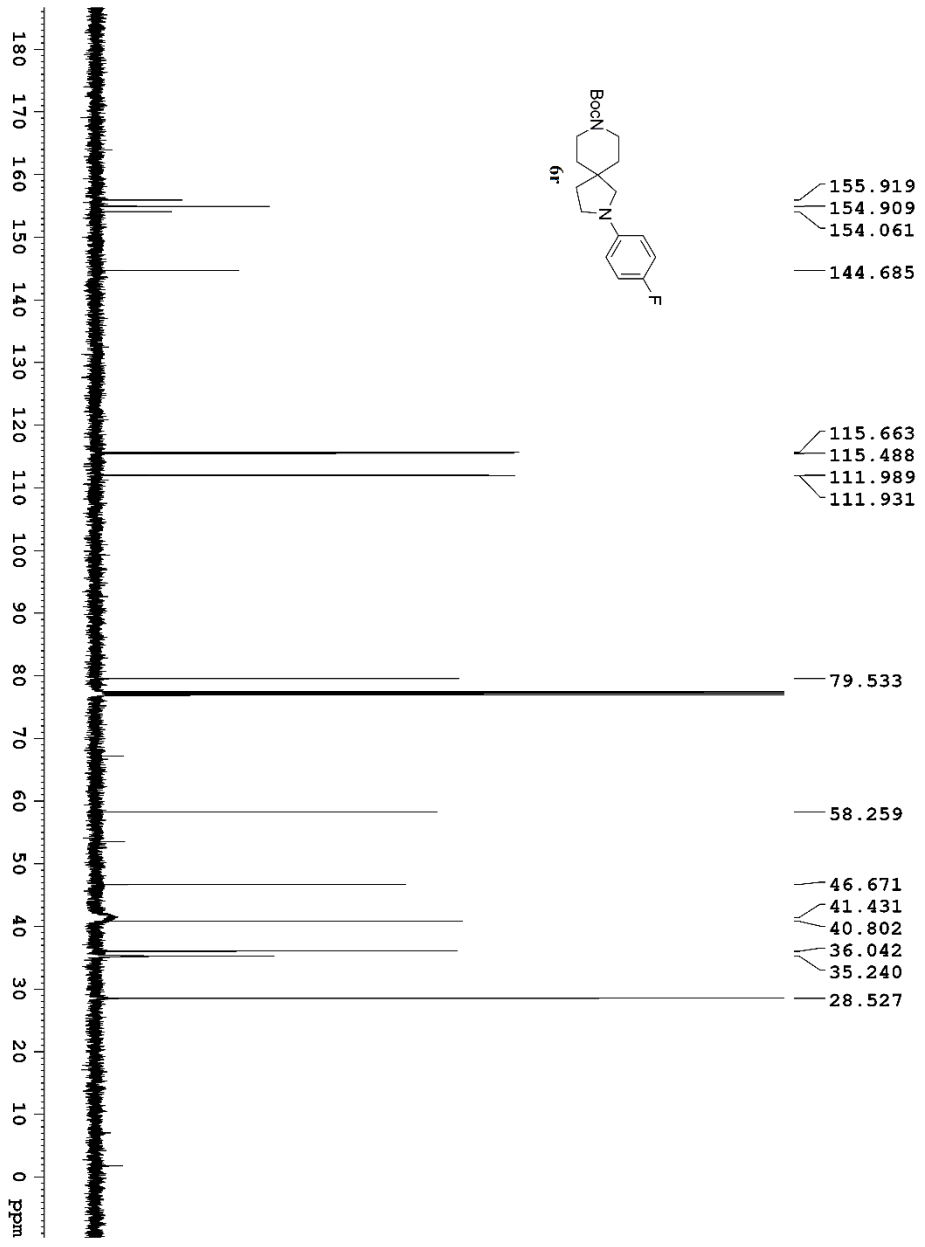
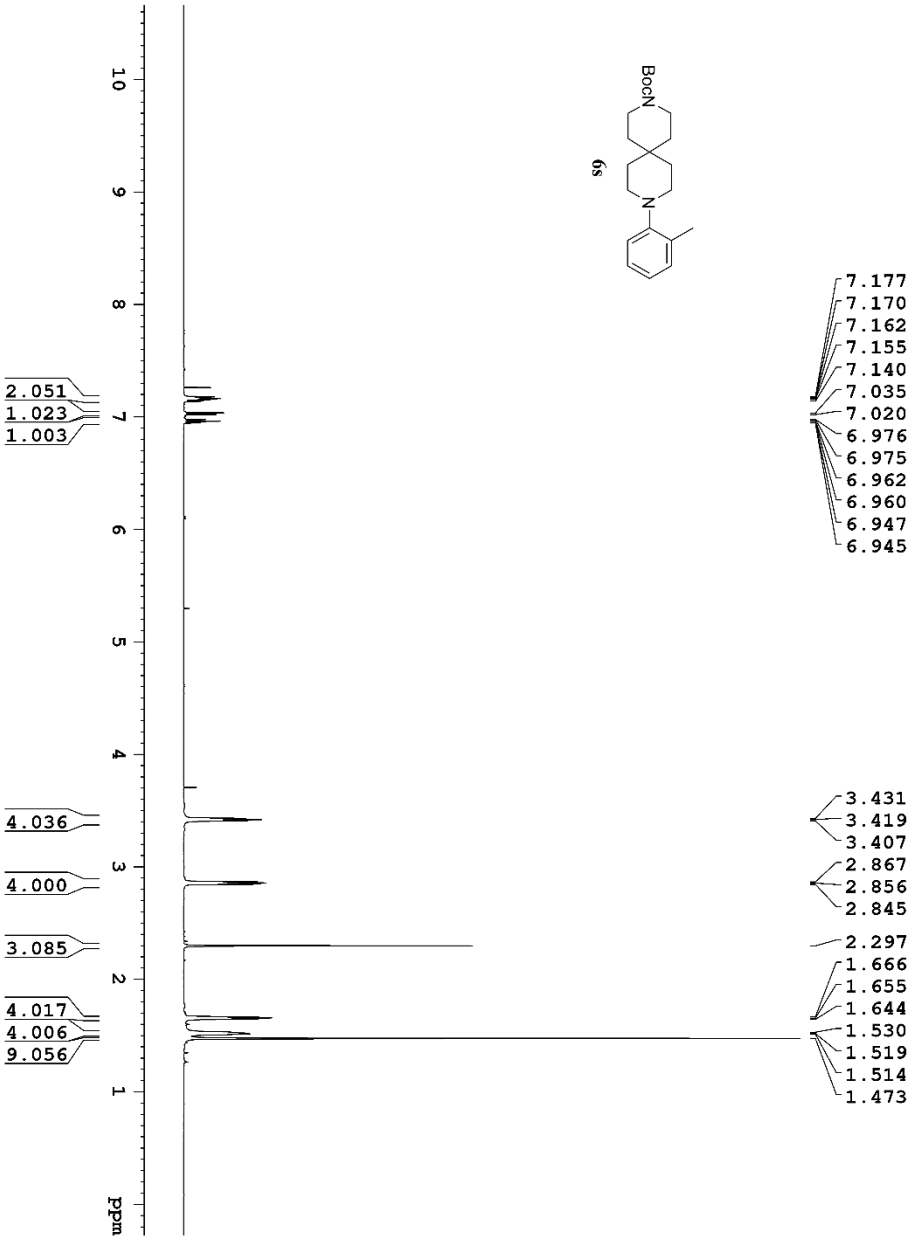
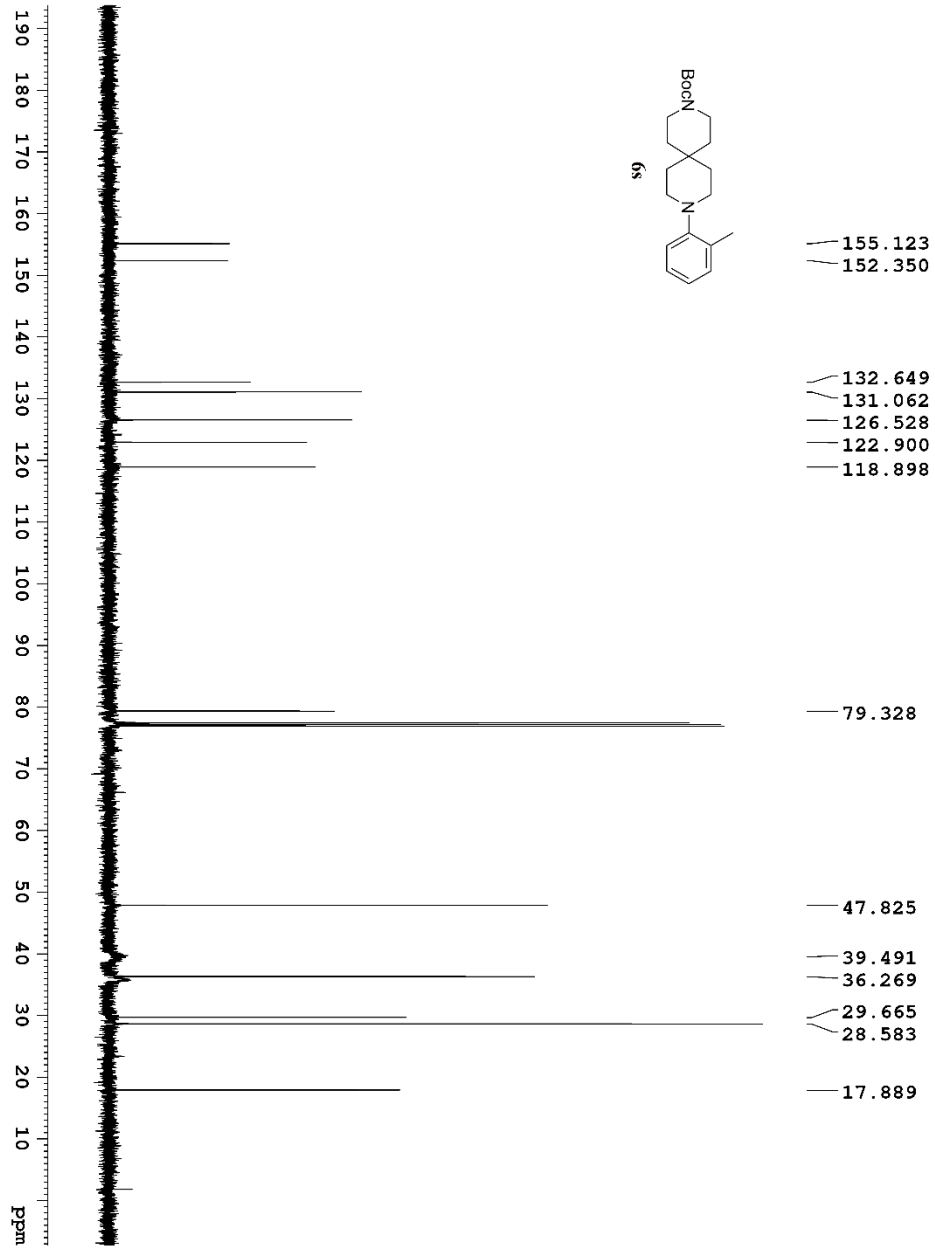
2.012
3.053
2.039
2.042
2.035
2.025
2.003
4.063
9.085
ppm

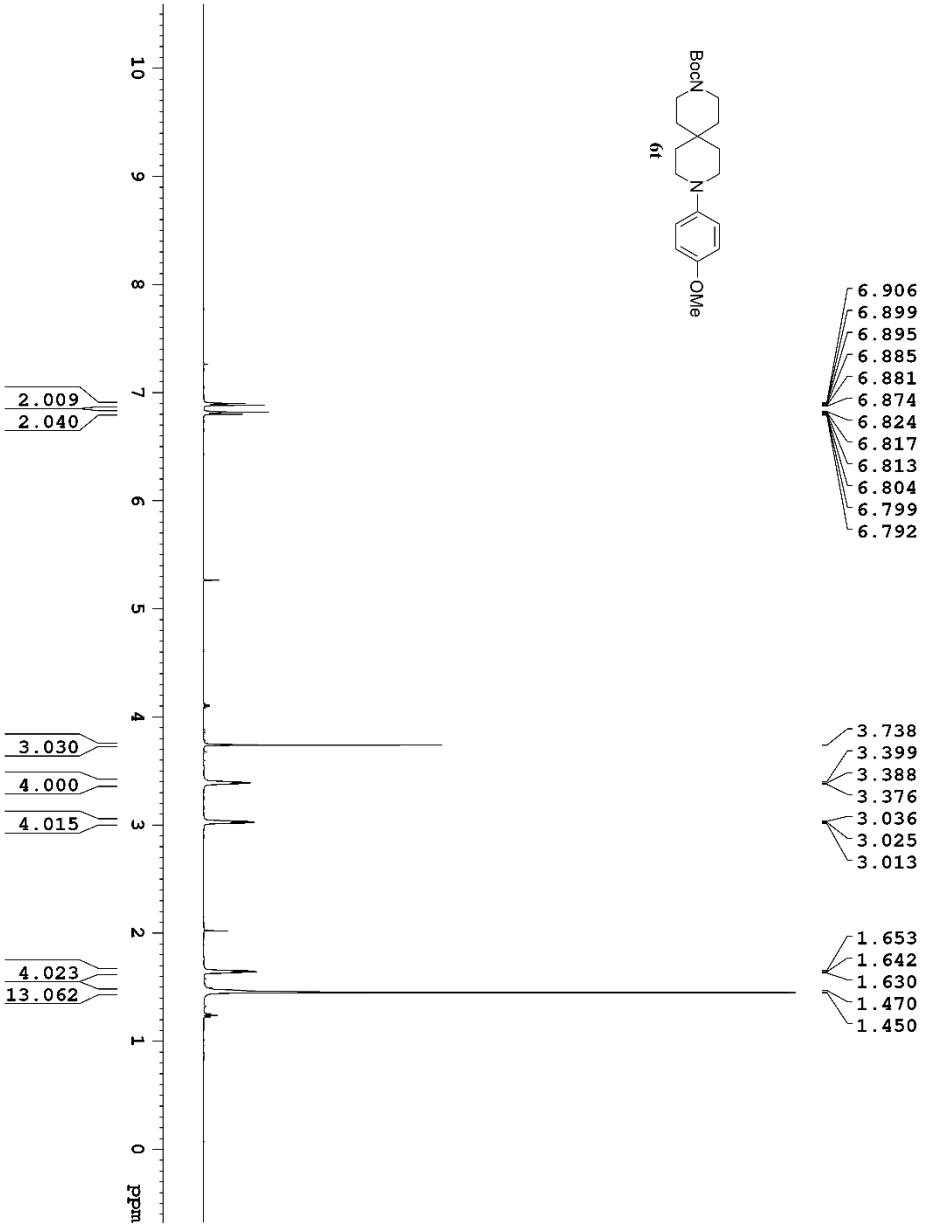

6q
BocN
N
OMe
154.905
150.990
143.007
115.119
112.387
79.462
58.421
56.031
46.779
41.274
40.713
36.122
35.382
28.520
ppm


6r
BocN
N
F
6.950
6.943
6.938
6.925
6.917
6.912
6.908
6.900
6.450
6.442
6.432
6.423
3.484
3.474
3.471
3.461
3.396
3.387
3.381
3.372
3.353
3.329
3.315
3.301
3.107
1.887
1.873
1.860
1.586
1.571
1.562
1.555
1.545
1.469
2.000
2.006
2.013
2.076
2.045
2.010
2.079
4.053
9.020
ppm


6r
BocN
N
F
155.919
154.909
154.061
144.685
115.663
115.488
111.989
111.931
79.533
58.259
46.671
41.431
40.802
36.042
35.240
28.527
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


BocN
N
9s
7.177
7.170
7.162
7.155
7.140
7.035
7.020
6.976
6.975
6.962
6.960
6.947
6.945
3.431
3.419
3.407
2.867
2.856
2.845
2.297
1.666
1.655
1.644
1.530
1.519
1.514
1.473
2.051
1.023
1.003
4.036
4.000
3.085
4.017
4.006
9.056
10
9
8
7
6
5
4
3
2
1
ppm


sg
BocN
N
155.123
152.350
132.649
131.062
126.528
122.900
118.898
79.328
47.825
39.491
36.269
29.665
28.583
17.889
ppm


BocN
N
OMe
6.906
6.899
6.895
6.885
6.881
6.874
6.824
6.817
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6.804
6.799
6.792
3.738
3.399
3.388
3.376
3.036
3.025
3.013
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1.630
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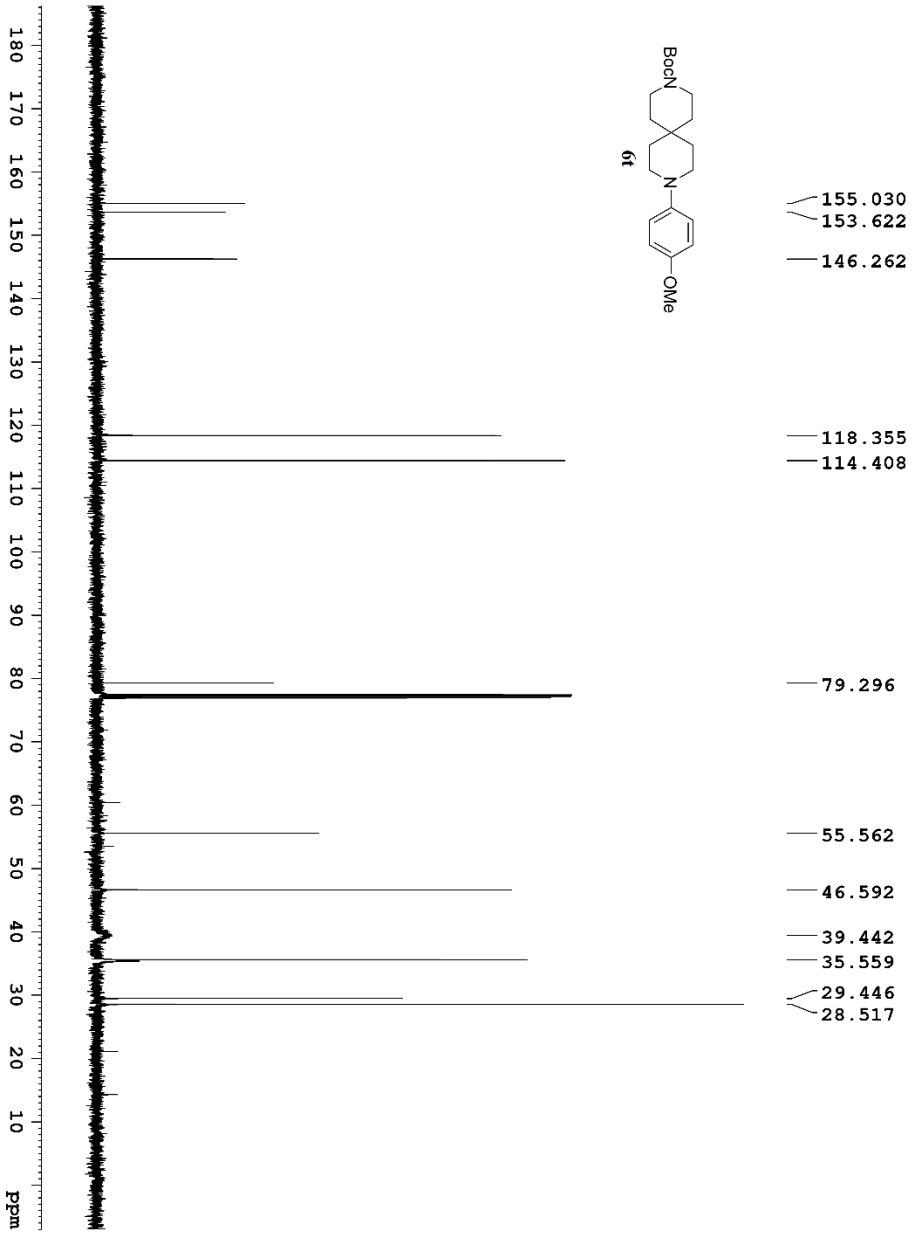
1.450
2.009
2.040
3.030
4.000
4.015
4.023
13.062
ppm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OMe
6g
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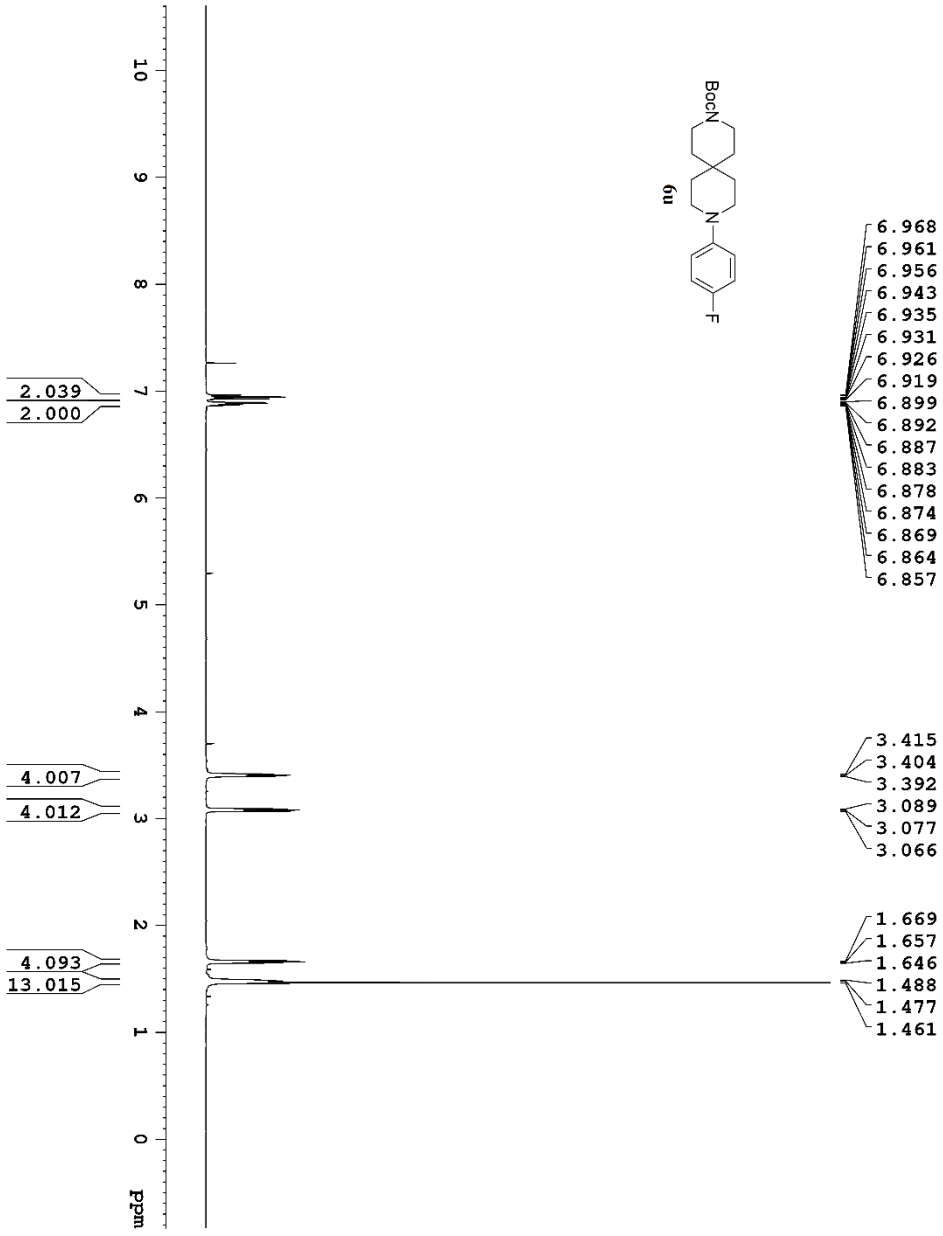
28.517
ppm


6u
BocN
N
F
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6.892
6.887
6.883
6.878
6.874
6.869
6.864
6.857
3.415
3.404
3.392
3.089
3.077
3.066
1.669
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1.477
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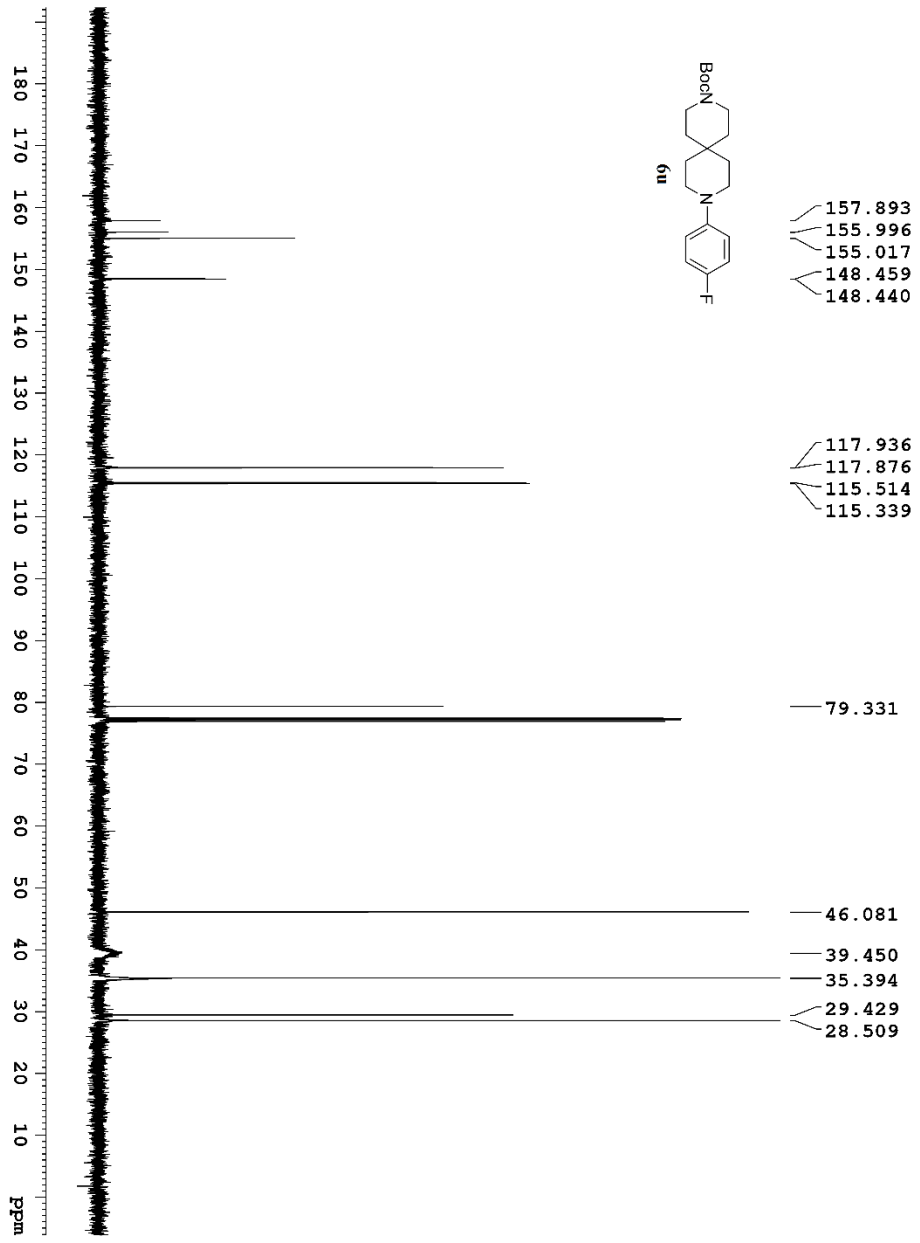
ppm


6u
BocN
N
F
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155.017
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117.936
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46.081
39.450
35.394
29.429
28.509
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
ppm