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Electronic Supplementary Information

Copper(I)/TF-Biphamphos Catalyzed Asymmetric Nitroso Diels-Alders Reaction

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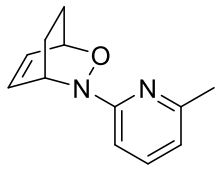
I. General Remarks

^{1}H NMR spectra were recorded on a Bruker 400 MHz spectrometer in $CDCl_3$. Chemical shifts are reported in ppm with the internal TMS signal at 0.0 ppm as a standard. The data are reported as (s = single, d = double, t = triple, q = quarte, m = multiple or unresolved, brs = broad single, coupling constant(s) in Hz, integration). ^{13}C NMR spectra were recorded on a Bruker 100 MHz spectrometer in $CDCl_3$. Chemical shifts are reported in ppm with the internal chloroform signal at 77.0 ppm as a standard. Enantiomeric ratios were determined by HPLC, using a chiralpak OD-H column with hexane and *i*-PrOH as solvents. **1a**, **1h**, **1i** were purchased from J&K Scientific and Aldrich. 1,3-Cyclopentadiene **1f** was cracked and distilled at 200°C. Various substituted 1,3-cyclohexandienes **1b-1e, 1g, 1j** and nitroso compounds **2a-2e** were prepared according to the literature procedure[1,2].

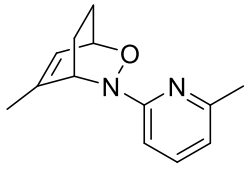
II. General Procedure for the Asymmetric Nitroso Diels-Alder Reaction of 1,3-dienes with nitroso compounds

Under argon atmosphere, TF-Biphamphos (17.6 mg, 0.022 mmol) and $CuBF_4$ (6.3 mg, 0.020 mmol) were dissolved in 4 mL DCM, and stirred at room temperature for about 30 min. The mixture was then cooled into -80°C and nitroso compound (0.20 mmol) dissolved in 0.5 mL of DCM was added. After stirred for 10 min, the 1,3-diene (0.24 mmol) dissolved in another 0.5 mL of DCM was added dropwise. The reaction mixture was gradually warmed to -40°C and kept at this temperature until the reaction complete. Then the organic solvent was removed and the residue was purified by column chromatography to give the product, which was then directly analyzed by HPLC to determine the enantiomeric excess. All the racemic samples were prepared by mixing the nitroso compounds (0.20 mmol) with the dienes (0.24 mmol) in DCM at 0°C



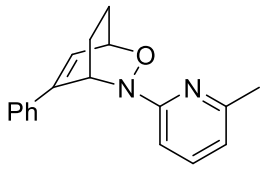
3a (known compound, see ref. 3)

(1*R*,4*S*)-3-(6-methylpyridin-2-yl)-2-oxa-3-azabicyclo[2.2.2]oct-5-ene: Yield (98%); $[\alpha]^{25}_{D}$ = -149 (*c* 0.90, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 400 MHz) δ 7.39 (t, *J* = 8.0 Hz, 1H), 6.71 (d, *J* = 8.0 Hz, 1H), 6.63 (d, *J* = 8.0 Hz, 1H), 6.49-6.45 (m, 1H), 6.30-6.25 (m, 1H), 5.32-5.28 (m, 1H), 4.72-4.69 (m, 1H), 2.42 (s, 3H), 2.30-2.20 (m, 2H), 1.62-1.55 (m, 1H), 1.43-1.34 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 100 MHz) δ 163.7, 156.1, 137.5, 131.6, 130.7, 116.0, 108.0, 69.5, 52.4, 24.2, 24.1, 20.5. The product was analyzed by HPLC to determine the enantiomeric excess: 96% ee (chiralpak OD-H, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min, λ = 254 nm); t_r = 9.25 and 10.43 min.



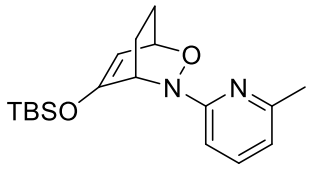
3b (known compound, see ref. 3)

(1*R*,4*S*)-5-methyl-3-(6-methylpyridin-2-yl)-2-oxa-3-azabicyclo[2.2.2]oct-5-ene : Yield (95%); $[\alpha]^{25}_{D}$ = -110 (*c* 0.68, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 400 MHz) \δ 7.39 (t, *J* = 8.0 Hz, 1H), 6.72 (d, *J* = 8.0 Hz, 1H), 6.62 (d, *J* = 8.0 Hz, 1H), 6.03-6.01 (m, 1H), 5.12-5.10 (m, 1H), 4.70-4.66 (m, 1H), 2.42 (s, 3H), 2.26-2.15 (m, 2H), 1.68 (d, *J* = 1.2 Hz, 3H), 1.62-1.51 (m, 1H), 1.38-1.30 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 100 MHz) δ 164.1, 155.9, 141.4, 137.5, 122.6, 115.9, 108.2, 70.6, 56.7, 25.3, 24.2, 20.5, 20.1. The product was analyzed by HPLC to determine the enantiomeric excess: 94% ee (chiralpak OD-H, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min, λ = 254 nm); t_r = 8.33 and 11.03 min.



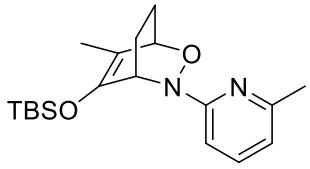
3c (known compound, see ref. 3)

(1*R*,4*S*)-3-(6-methylpyridin-2-yl)-5-phenyl-2-oxa-3-azabicyclo[2.2.2]oct-5-ene: Yield (98%); $[\alpha]^{25}_{D}$ = +111 (*c* 1.10, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 400 MHz) δ 7.55 (d, *J* = 8.0 Hz, 2H), 7.35-7.20 (m, 4H), 6.73 (d, *J* = 8.0 Hz, 1H), 6.65 (dd, *J* = 2.0, 6.0 Hz, 1H), 6.55 (d, *J* = 8.0 Hz, 1H), 5.79-5.78 (m, 1H), 4.90-4.88 (m, 1H), 2.41 (s, 3H), 2.36-2.29 (m, 2H), 1.71-1.64 (m, 1H), 1.47-1.41 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 100 MHz) δ 163.3, 155.8, 142.7, 137.6, 136.0, 128.2, 127.8, 125.5, 122.4, 116.1, 107.9, 70.0, 54.3, 24.6, 24.0, 20.9. The product was analyzed by HPLC to determine the enantiomeric excess: 96% ee (chiralpak OD-H, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min, λ = 254 nm); t_r = 12.90 and 19.07 min.



3d (known compound, see ref. 3)

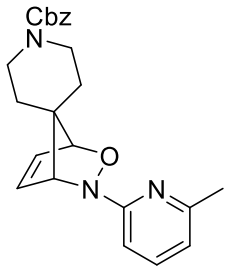
(1*R*,4*S*)-5-((tert-butyldimethylsilyl)oxy)-3-(6-methylpyridin-2-yl)-2-oxa-3-azabicyclo[2.2.2]oct-5-ene: Yield (95%); $[\alpha]^{25}_{D}$ = -64 (*c* 0.91, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 400 MHz) δ 7.38 (t, *J* = 8.0 Hz, 1H), 6.76 (d, *J* = 8.0 Hz, 1H), 6.63 (d, *J* = 8.0 Hz, 1H), 5.14 (dd, *J* = 2.8, 6.4 Hz, 1H), 5.06 (dd, *J* = 2.8, 6.4 Hz, 1H), 4.82-4.79 (m, 1H), 2.39 (s, 3H), 2.25-2.13 (m, 2H), 1.80-1.73 (m, 2H), 0.78 (s, 9H), 0.02 (s, 3H), -0.28 (s, 3H); ^{13}C NMR ($CDCl_3$, TMS, 100 MHz) δ 164.0, 156.3, 153.3, 137.6, 116.4, 108.1, 100.3, 72.0, 58.6, 26.3, 25.3, 24.3, 21.1, 17.8, -4.6, -5.8. The product was analyzed by HPLC to determine the enantiomeric excess: 95% ee (chiralpak OD-H, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min, λ = 254 nm); t_r = 6.68 and 9.45 min.



3e

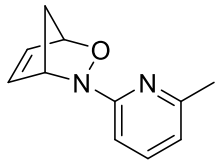
(1*R*,4*S*)-5-((tert-butyldimethylsilyl)oxy)-6-methyl-3-(6-methylpyridin-2-yl)-2-oxa-3-azabicyclo[2.2.2]oct-5-ene: Yield (92%); $[\alpha]^{25}_{D}$ = -82 (*c* 0.73, $CHCl_3$); ^{1}H NMR

($CDCl_3$, TMS, 400 MHz) δ 7.39 (t, *J* = 8.0 Hz, 1H), 6.73 (d, *J* = 8.0 Hz, 1H), 6.63 (d, *J* = 8.0 Hz, 1H), 5.01-4.99 (m, 1H), 4.65-4.63 (m, 1H), 2.41 (s, 3H), 2.24-2.12 (m, 2H), 1.69 (s, 3H), 1.44-1.38 (m, 1H), 0.86 (s, 9H), 0.05 (s, 3H), -0.02 (s, 3H); ^{13}C NMR ($CDCl_3$, TMS, 100 MHz) δ 163.7, 156.2, 145.9, 137.7, 116.3, 112.8, 107.9, 76.8, 58.3, 26.1, 25.5, 24.2, 22.1, 18.0, 11.9, -4.3, -4.5. IR (KBr) ν 2955, 2928, 2856, 2341, 1681, 1589, 1576, 1450, 1259, 1213, 1200, 931, 839, 783, 681, 668 cm^{-1}. HRMS Calcd. For $C_{19}H_{31}O_2N_2Si^+$: 347.2149, found: 347.2149. The product was analyzed by HPLC to determine the enantiomeric excess: 97% ee (chiralpak OD-H, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min, λ = 254 nm); t_r = 5.51 and 8.33 min.



3f

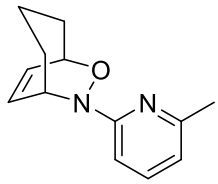
benzyl (1*S*,4*R*)-3-(6-methylpyridin-2-yl)-2-oxa-3-azaspiro[bicyclo[2.2.1]heptane -7,4'-piperidin]-5-ene-1'-carboxylate: Yield (96%); $[\alpha]^{25}_D$ = -89 (*c* 0.83, $CHCl_3$); ^1H NMR ($CDCl_3$, TMS, 400 MHz) δ 7.38-7.30 (m, 6H), 6.63 (d, *J* = 8.0 Hz, 1H), 6.57 (d, *J* = 8.0 Hz, 1H), 6.23-6.21 (m, 1H), 6.01-5.98 (m, 1H), 5.15-5.13 (m, 3H), 4.74-4.72 (m, 1H), 3.68-3.52 (m, 2H), 3.50-3.37 (m, 2H), 2.42 (s, 3H), 2.05-1.93 (m, 2H), 1.60-1.54 (m, 2H); ^{13}C NMR ($CDCl_3$, TMS, 100 MHz) δ 162.9, 156.4, 137.6, 136.7, 134.2, 130.1, 128.4, 127.9, 127.8, 116.4, 108.6, 86.1, 70.4, 67.0, 60.0, 42.1, 41.6, 29.1, 29.0, 24.3. IR (KBr) ν 2960, 2924, 2852, 1589, 1579, 1450, 1330, 1260, 1230, 1021, 926, 853, 799, 736 cm^{-1}. HRMS Calcd. For $C_{23}H_{26}O_3N_3^+$: 392.1969, found: 392.1971. The product was analyzed by HPLC to determine the enantiomeric excess: 90% ee (chiralpak OD-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min, λ = 254 nm); t_r = 10.92 and 20.60 min.



3g (known compound, see ref. 3)

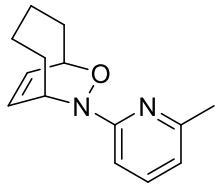
(1*R*,4*S*)-3-(6-methylpyridin-2-yl)-2-oxa-3-azabicyclo[2.2.1]hept-5-ene:

Yield (90%); $[\alpha]^{25}_{D}$ = -114 (*c* 0.95, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 400 MHz) δ 7.39 (t, *J* = 8.0 Hz, 1H), 6.65 (t, *J* = 8.0 Hz, 2H), 6.32-6.30 (m, 1H), 6.12-6.09 (m, 1H), 5.52-5.50 (m, 1H), 5.21-5.19 (m, 1H), 2.44 (s, 3H), 2.12 (dt, *J* = 2.0, 8.4 Hz, 1H), 1.80 (d, *J* = 8.4 Hz, 1H); ^{13}C NMR ($CDCl_3$, TMS, 100 MHz) δ 163.2, 156.4, 137.6, 134.9, 132.3, 116.5, 108.9, 82.7, 66.8, 47.9, 24.2. The product was analyzed by HPLC to determine the enantiomeric excess: 87% ee (chiralpak OD-H, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min, λ = 254 nm); t_r = 9.27 and 12.71 min.



3h (known compound, see ref. 3)

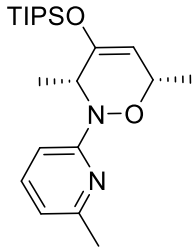
(1*S*,5*R*)-7-(6-methylpyridin-2-yl)-6-oxa-7-azabicyclo[3.2.2]non-8-ene: Yield (90%); $[\alpha]^{25}_{D}$ = -167 (*c* 0.57, CH_2Cl_2); ^{1}H NMR ($CDCl_3$, TMS, 400 MHz) δ 7.41 (t, *J* = 8.0 Hz, 1H), 6.80 (d, *J* = 8.0 Hz, 1H), 6.60 (d, *J* = 8.0 Hz, 1H), 6.19-6.14 (m, 1H), 6.06-6.01 (m, 1H), 5.38-5.33 (m, 1H), 4.81-4.77 (m, 1H), 2.40 (s, 3H), 2.06-1.89 (m, 3H), 1.76-1.70 (m, 1H), 1.64-1.56 (m, 1H), 1.48-1.36 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 100 MHz) δ 163.5, 156.3, 137.7, 130.4, 125.6, 115.5, 107.6, 73.4, 57.0, 31.6, 27.1, 24.3, 18.7. The product was analyzed by HPLC to determine the enantiomeric excess: 71% ee (chiralpak OD-H, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min, λ = 254 nm); t_r = 6.78 and 7.79 min.



3i

(1*S*,6*R*)-8-(6-methylpyridin-2-yl)-7-oxa-8-azabicyclo[4.2.2]dec-9-ene: Yield (80%);

$[\alpha]^{25}_{D}$ =-77 (*c* 0.61 CH_2Cl_2); ^{1}H NMR ($CDCl_3$, TMS, 400 MHz) δ 7.44 (t, *J* = 8.0 Hz, 1H), 6.88 (d, *J* = 8.0 Hz, 1H), 6.61 (d, *J* = 8.0 Hz, 1H), 6.28 (d, *J* = 10.0 Hz, 1H), 6.26 (d, *J* = 10.0 Hz, 1H), 5.71 (d, *J* = 10.0 Hz, 1H),5.70 (d, *J* = 10.0 Hz, 1H), 5.26-5.22 (m, 1H), 4.95-4.92 (m, 1H), 2.40 (s, 3H), 2.32-2.25 (m, 1H), 2.20-2.07 (m, 2H), 1.91-1.60 (m, 6H); ^{13}C NMR ($CDCl_3$, TMS, 100 MHz) δ 163.2, 156.5, 137.9, 131.9, 125.5, 115.4, 106.8, 73.0, 54.6, 34.8, 32.0, 26.2, 24.4, 22.3. IR (KBr) ν 2917, 2854, 1589, 1576, 1447, 1283, 1231, 1178, 972, 830. 783, 637 cm^{-1}. HRMS Calcd. For $C_{14}H_{19}ON_2^+$: 231.1492, found: 231.1488. The product was analyzed by HPLC to determine the enantiomeric excess: 80% ee (chiralpak AD-H, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min, λ = 254 nm); t_r = 5.20 and 7.29 min.



3j (known compound, see ref. 3)

(3*R*,6*S*)-3,6-dimethyl-2-(6-methylpyridin-2-yl)-4-((triisopropylsilyl)oxy)-3,6-dihydro-2H-1,2-oxazine: Yield (93%); $[\alpha]^{25}_{D}$ = -134 (*c* 1.09, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 400 MHz) δ 7.44 (t, *J* = 8.0 Hz, 1H), 6.91 (d, *J* = 8.0 Hz, 1H), 6.59 (d, *J* = 8.0Hz, 1H), 4.79-4.66 (m, 3H), 2.41 (s, 3H), 1.29-1.19 (m, 9H), 1.12 (s, 12H), 1.10 (s, 6H); ^{13}C NMR ($CDCl_3$, TMS, 100 MHz) δ 159.3, 156.6, 152.5, 137.7, 114.7, 106.2, 102.9, 71.9, 54.2, 24.4, 20.0, 18.0, 14.2, 12.6. The product was analyzed by HPLC to determine the enantiomeric excess: 96% ee (chiralpak OD-H, *i*-propanol/hexane = 0/100, flow rate 1.0 mL/min, λ = 254 nm); t_r = 8.75 and 15.06 min.

III. References

1. a) A. S. E. Karlstrom, M. Ronn, A. Thorarensen and J. E. Backvall, *J. Org. Chem*. 1998, **63**, 2517-2522; (b) M. E. Jung and M. A. Guzaev, *Org. Lett.*, 2012**, 14**, 5169-5171; (c) W. Lin, A. Gupta, K. H. Kim, D. Mendel and M. J. Miller, *Org. Lett.* 2009**, 11**, 449-452; (d) M. Arisawa, Y. Torisawa, M. Kawahara and M. Nakagawa, *J. Org. Chem.* 1997, **62**, 4327-4329; (e) A. G. Dossetter, T. F. Jamison and E. N. Jacobsen, *Angew. Chem. Int. Ed.* 1999, **43**, 2398-2400.

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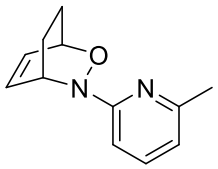
3. (a) B. Maji and H. Yamamoto, *J. Am. Chem. Soc.*, 2015, **137**, 15957-15963; (b) Y. Yamamoto and H. Yamamoto, *Angew. Chem., Int. Ed.,* 2005, **44**, 7082-7085; (c). Y. Yamamoto and H. Yamamoto, *J. Am. Chem. Soc.,* 2004, **126**, 4128-4129.

IV. ^{1}H NMR and ^{13}C NMR Spectra



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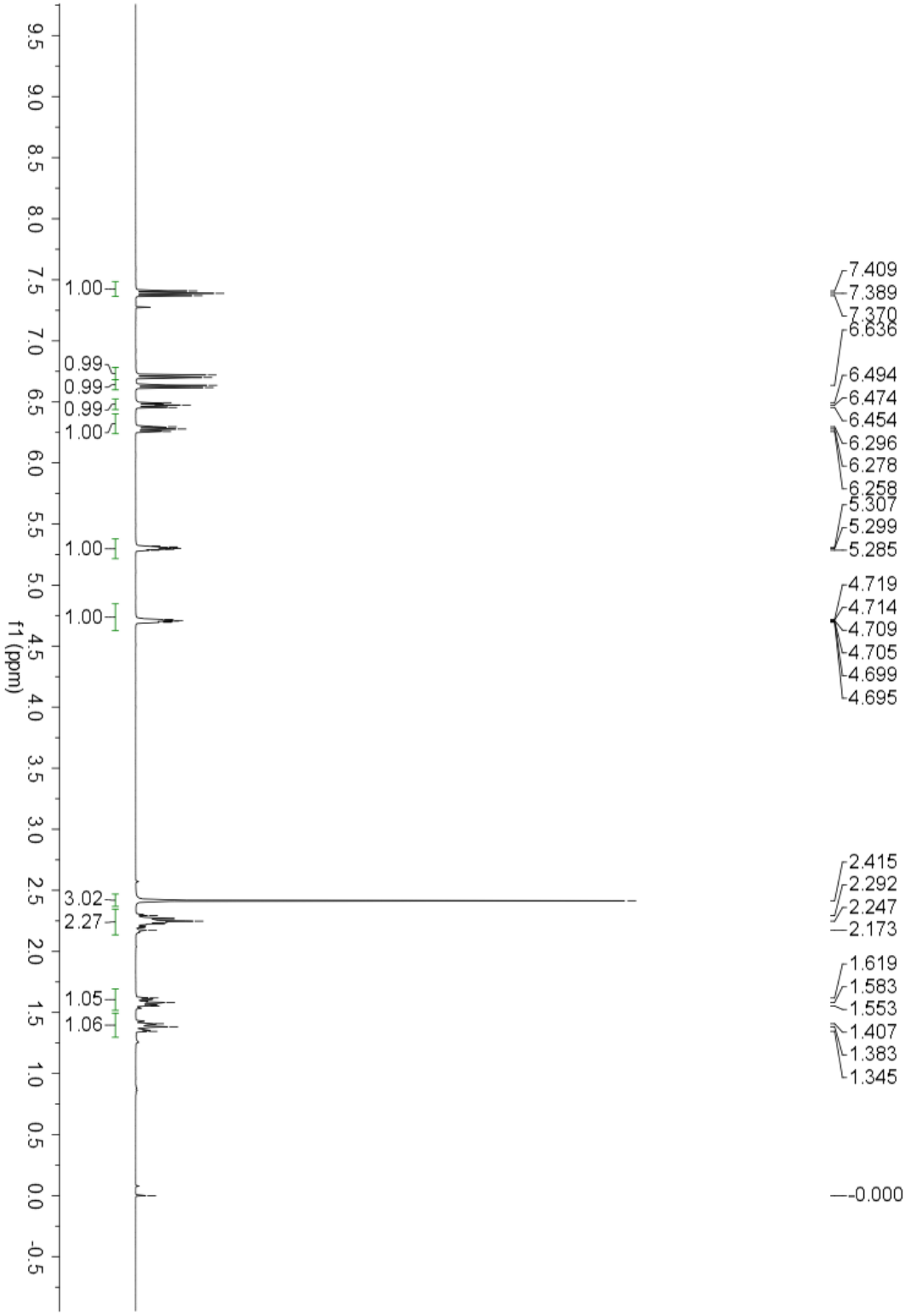

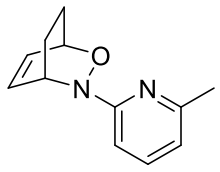

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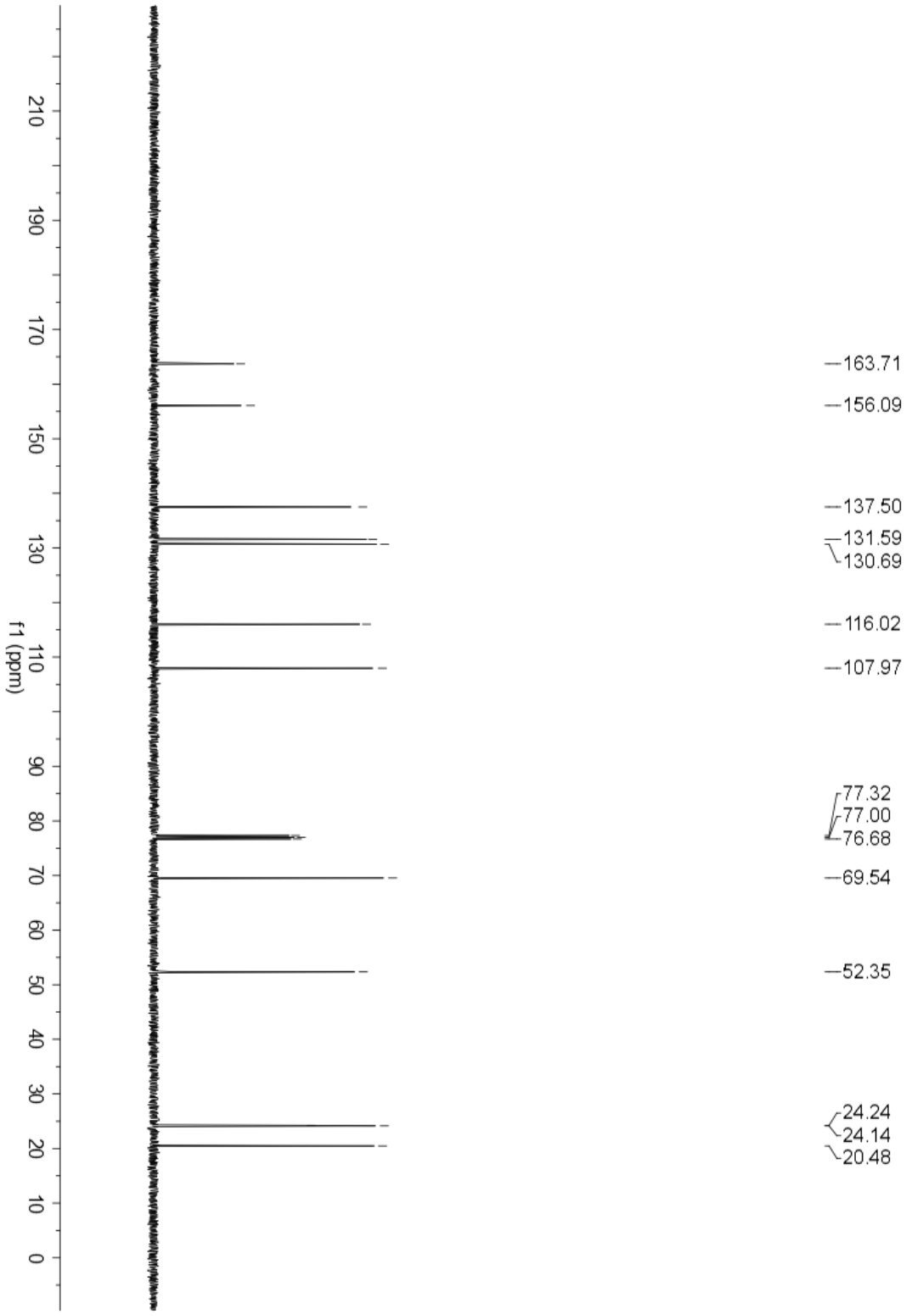
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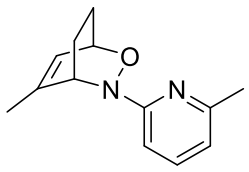

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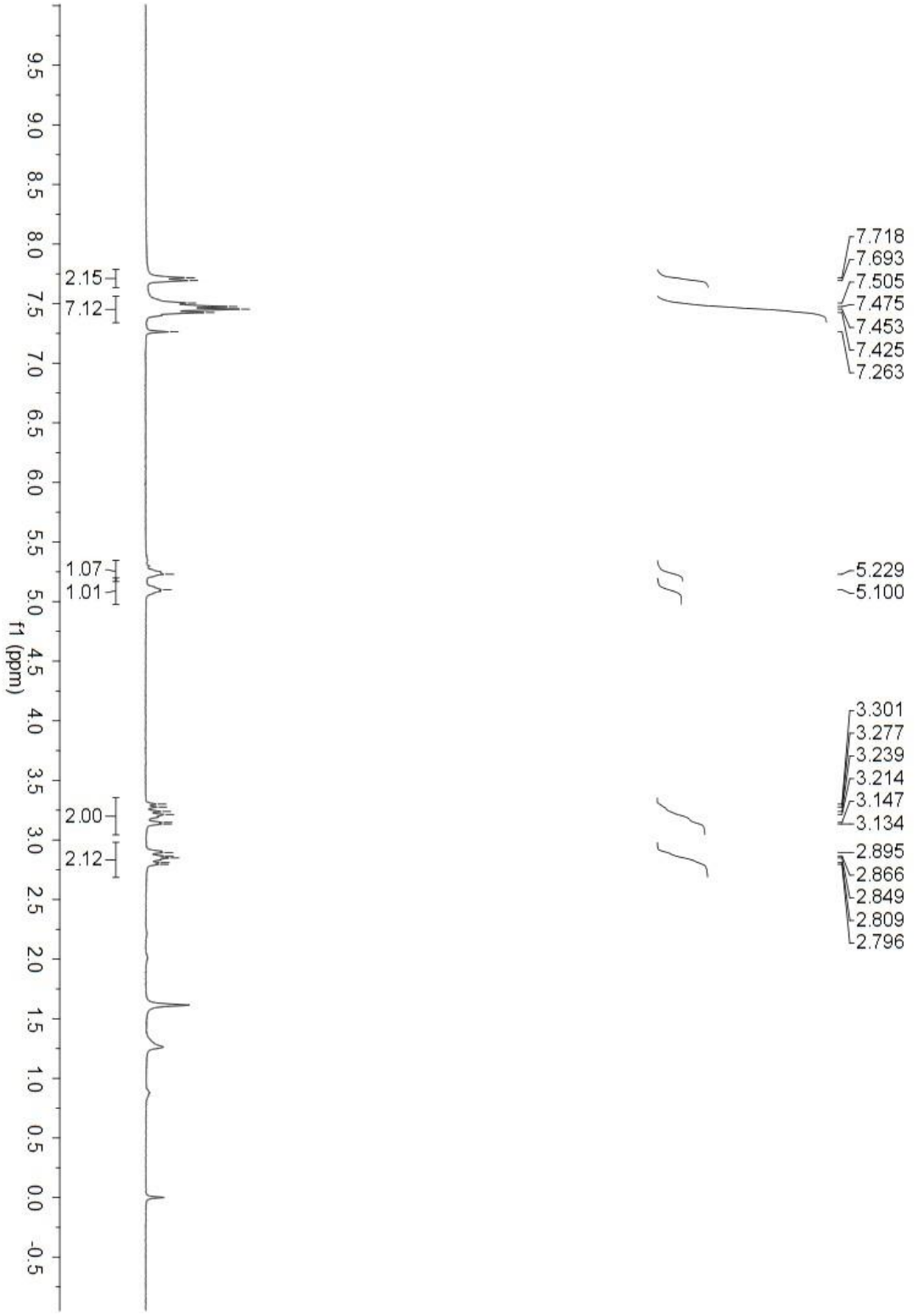
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3.147
3.134
2.895
2.866
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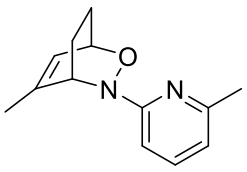
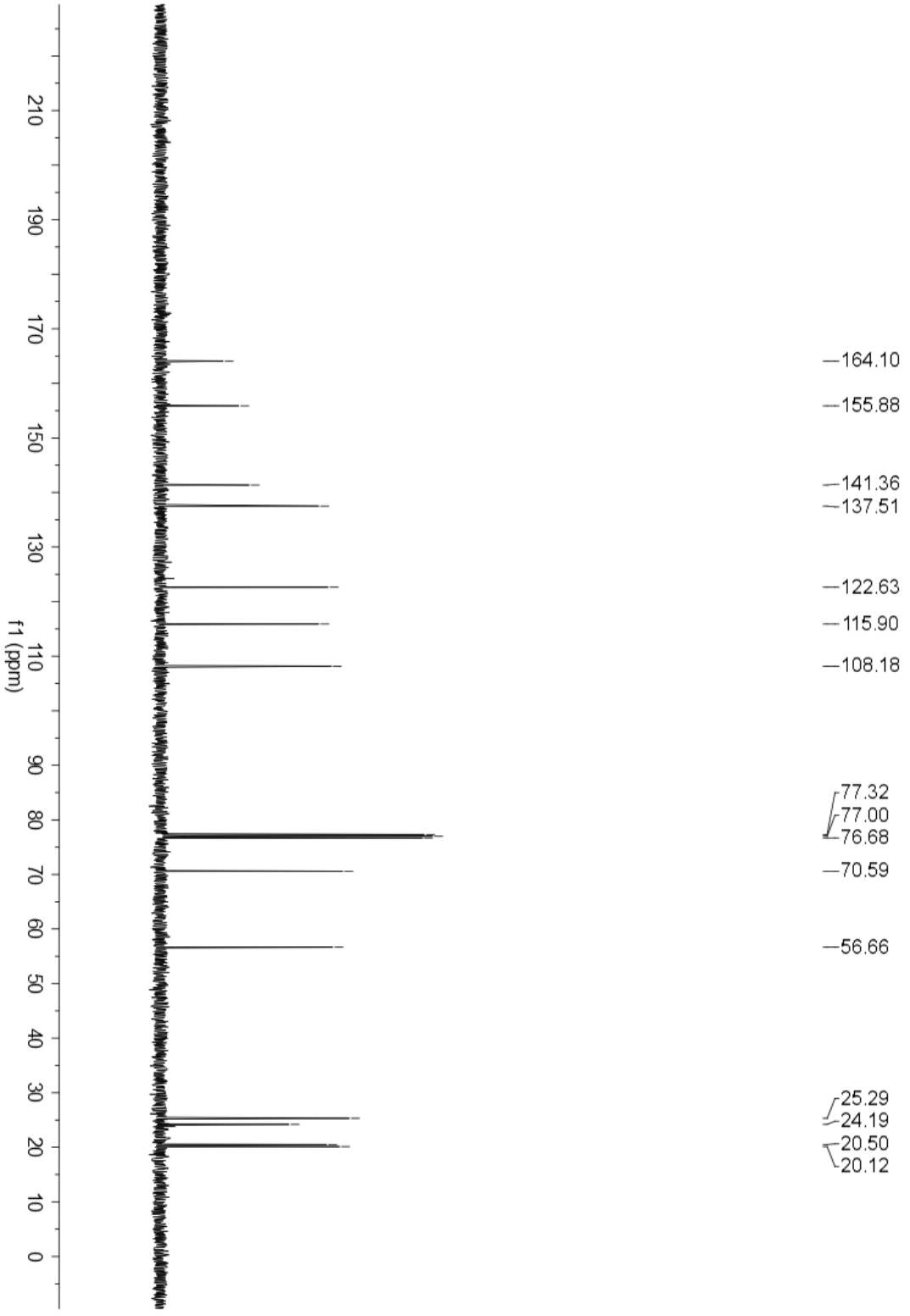
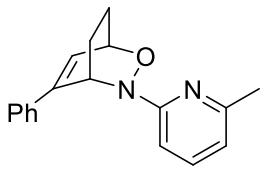
2.15
7.12
1.07
1.01
2.00
2.12
f1 (ppm)

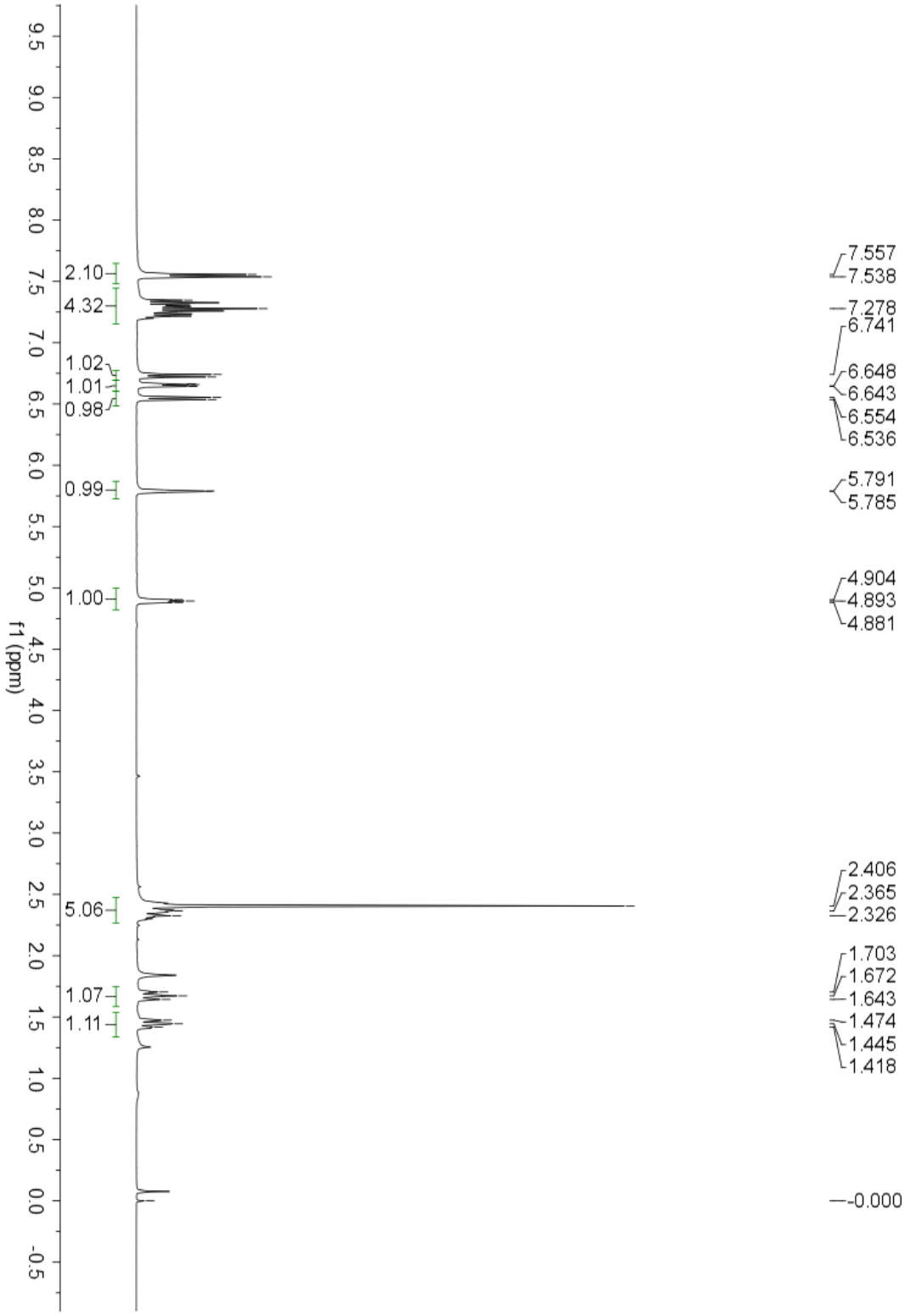


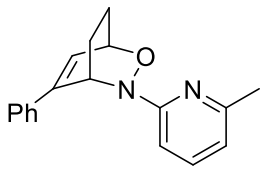
3b


f1 (ppm)
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
164.10
155.88
141.36
137.51
122.63
115.90
108.18
77.32
77.00
76.68
70.59
56.66
25.29
24.19
20.50
20.12

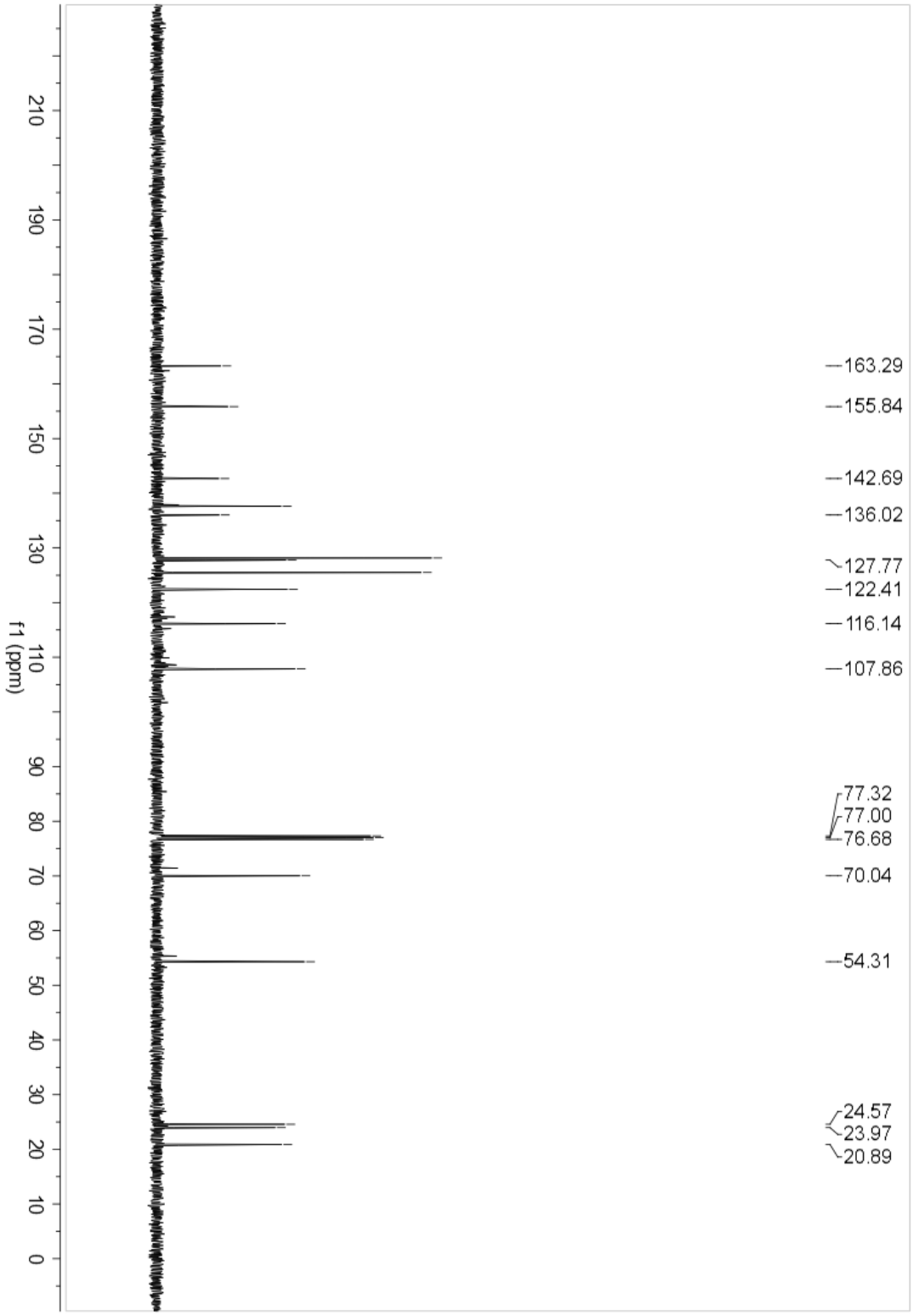

Ph
O
N
N

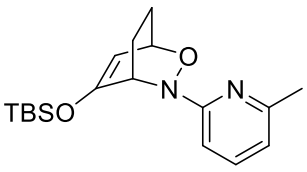
3c


f1 (ppm)
2.10
4.32
1.02
1.01
0.98
0.99
1.00
5.06
1.07
1.11
7.557
7.538
7.278
6.741
6.648
6.643
6.554
6.536
5.791
5.785
4.904
4.893
4.881
2.406
2.365
2.326
1.703
1.672
1.643
1.474
1.445
1.418
0.000

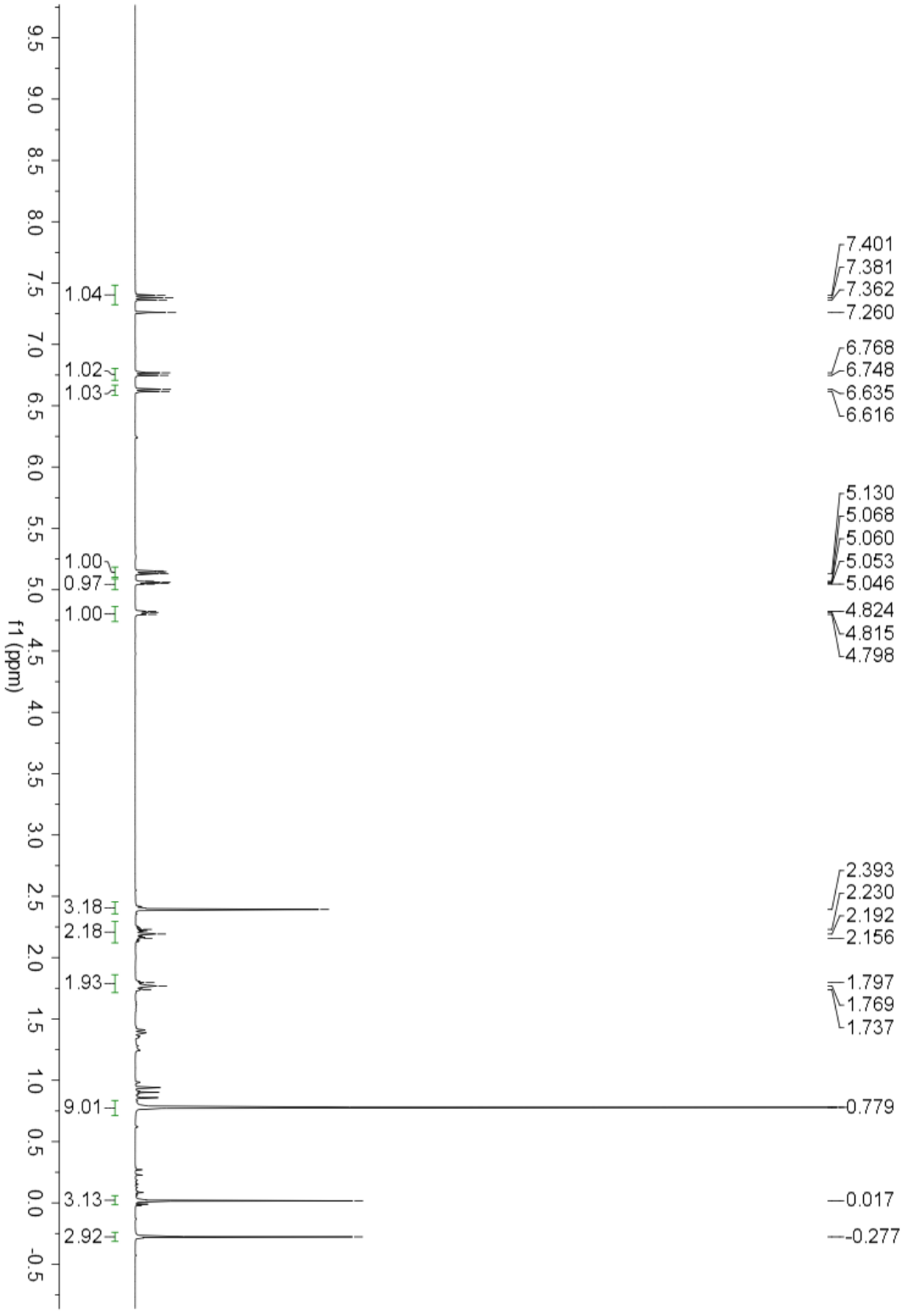

Ph
O
N
N

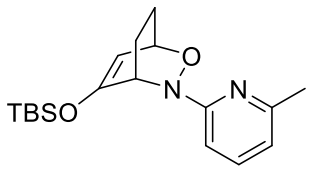
3c


f1 (ppm)
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
163.29
155.84
142.69
136.02
127.77
122.41
116.14
107.86
77.32
77.00
76.68
70.04
54.31
24.57
23.97
20.89

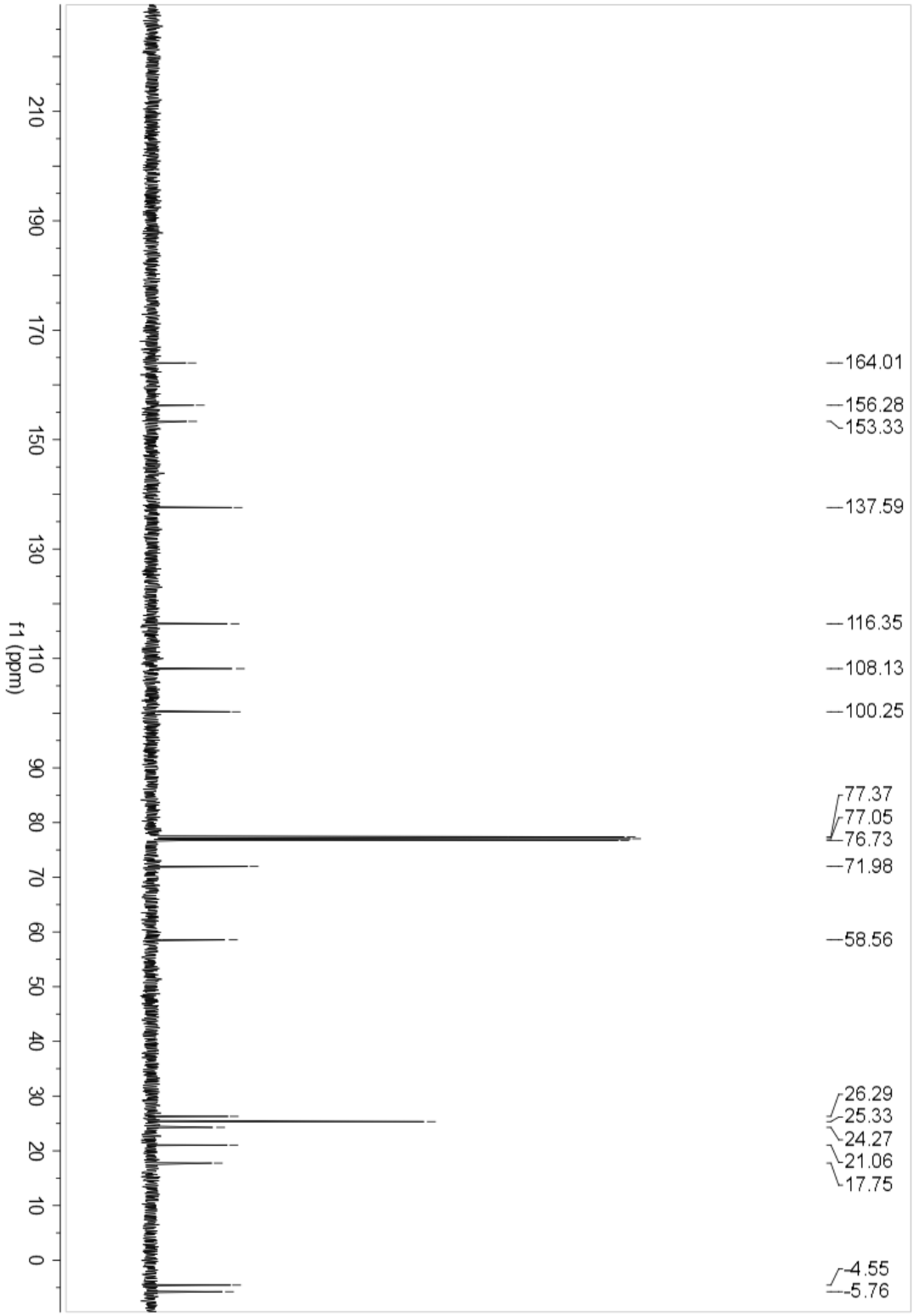

TBSO
O
N
N

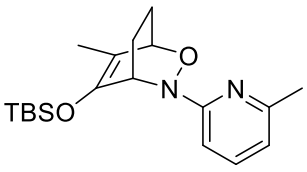
3d


f1 (ppm)
1.04
1.02
1.03
1.00
0.97
1.00
3.18
2.18
1.93
9.01
3.13
2.92
7.401
7.381
7.362
7.260
6.768
6.748
6.635
6.616
5.130
5.068
5.060
5.053
5.046
4.824
4.815
4.798
2.393
2.230
2.192
2.156
1.797
1.769
1.737
0.779
0.017
-0.277

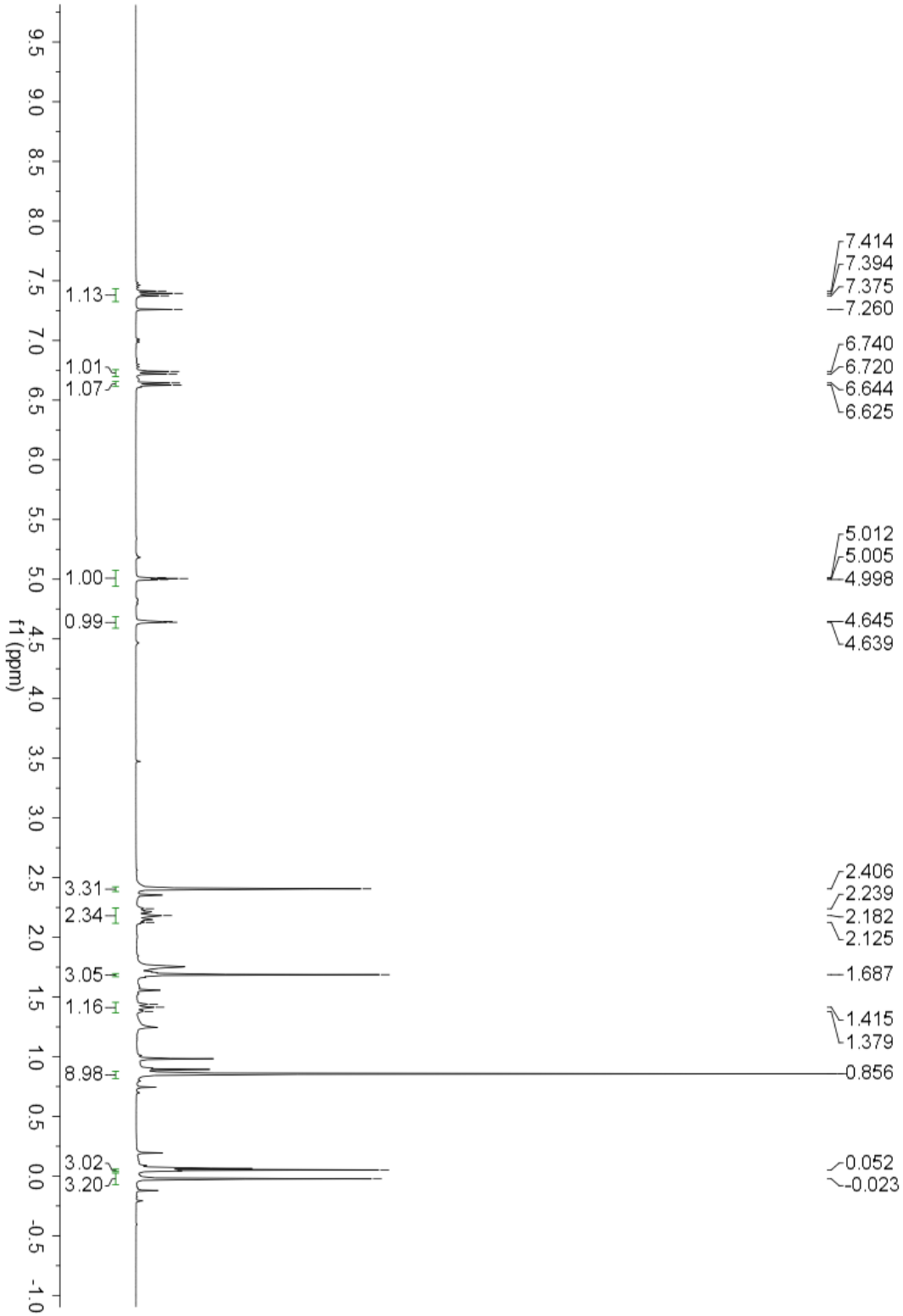

TBSO
O
N
N

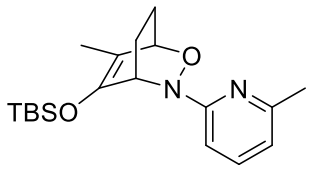
3d


f1 (ppm)
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
164.01
156.28
153.33
137.59
116.35
108.13
100.25
77.37
77.05
76.73
71.98
58.56
26.29
25.33
24.27
21.06
17.75
-4.55
-5.76

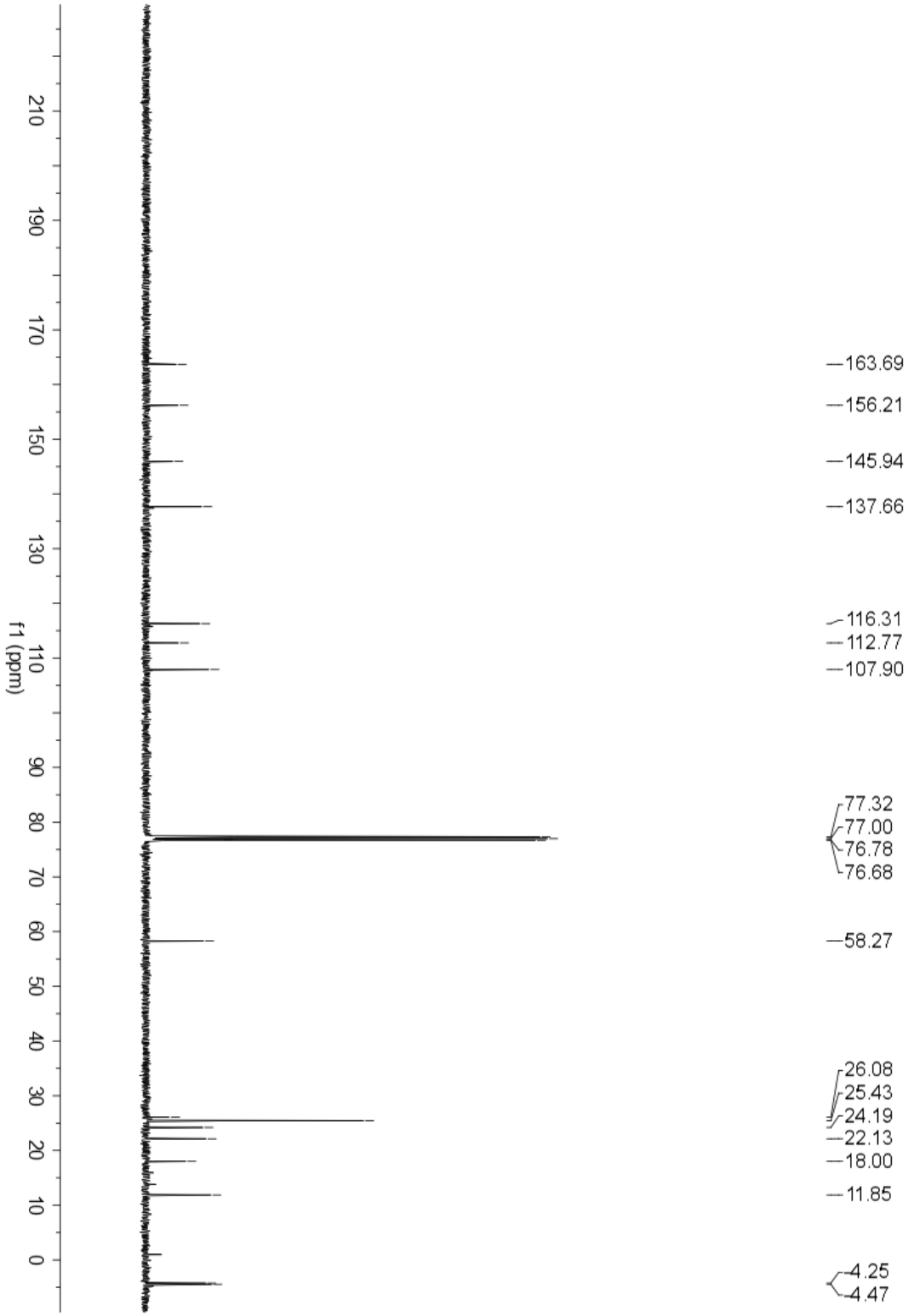

TBSO
O
N
N

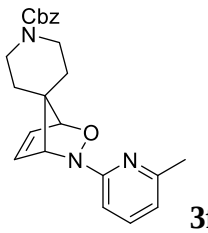
3e


f1 (ppm)
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
1.13
1.01
1.07
1.00
0.99
3.31
2.34
3.05
1.16
8.98
3.02
3.20
7.414
7.394
7.375
7.260
6.740
6.720
6.644
6.625
5.012
5.005
4.998
4.645
4.639
2.406
2.239
2.182
2.125
1.687
1.415
1.379
0.856
0.052
-0.023

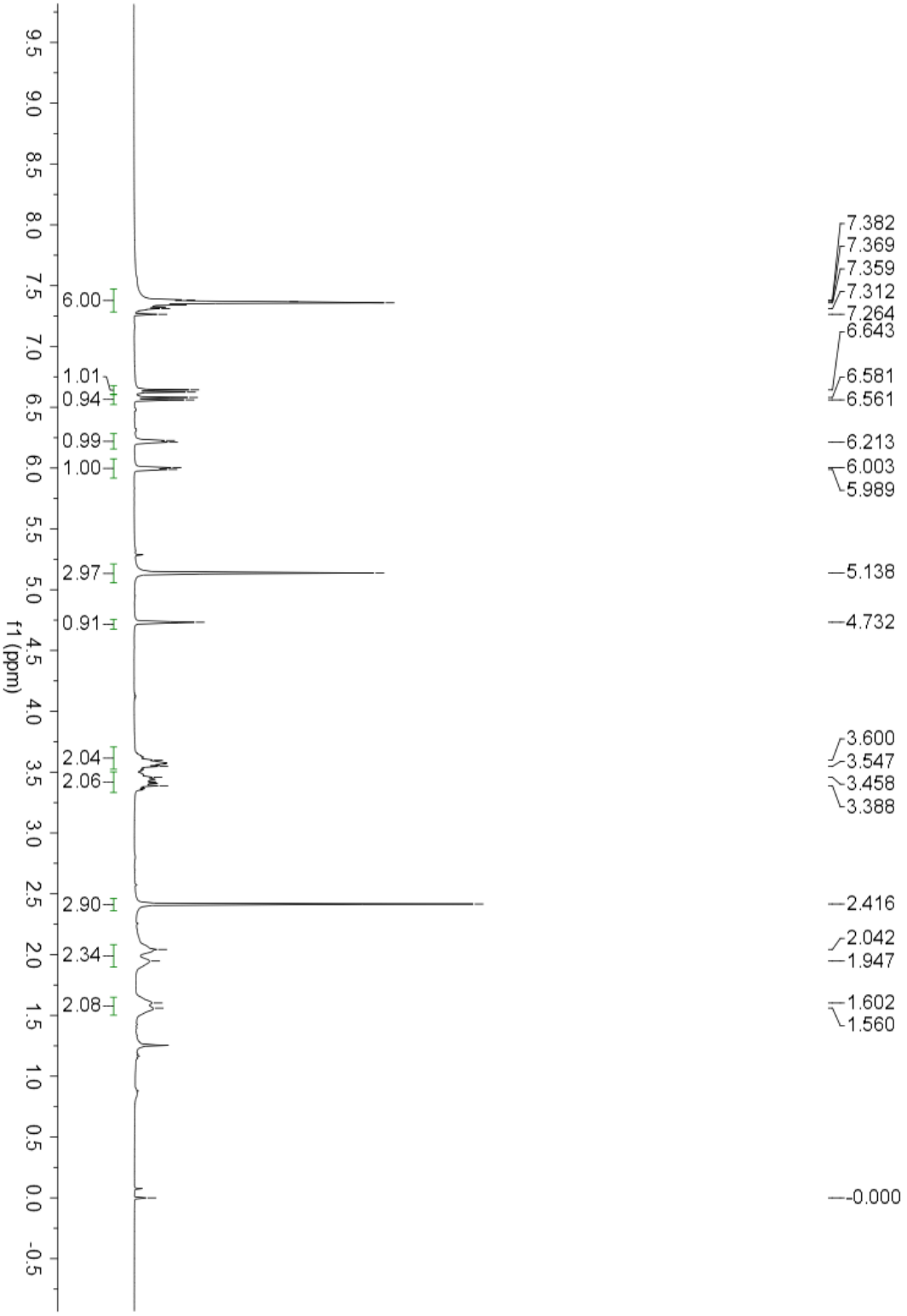

TBSO
O
N
N

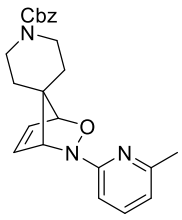
3e


f1 (ppm)
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
163.69
156.21
145.94
137.66
116.31
112.77
107.90
77.32
77.00
76.78
76.68
58.27
26.08
25.43
24.19
22.13
18.00
11.85
-4.25
-4.47

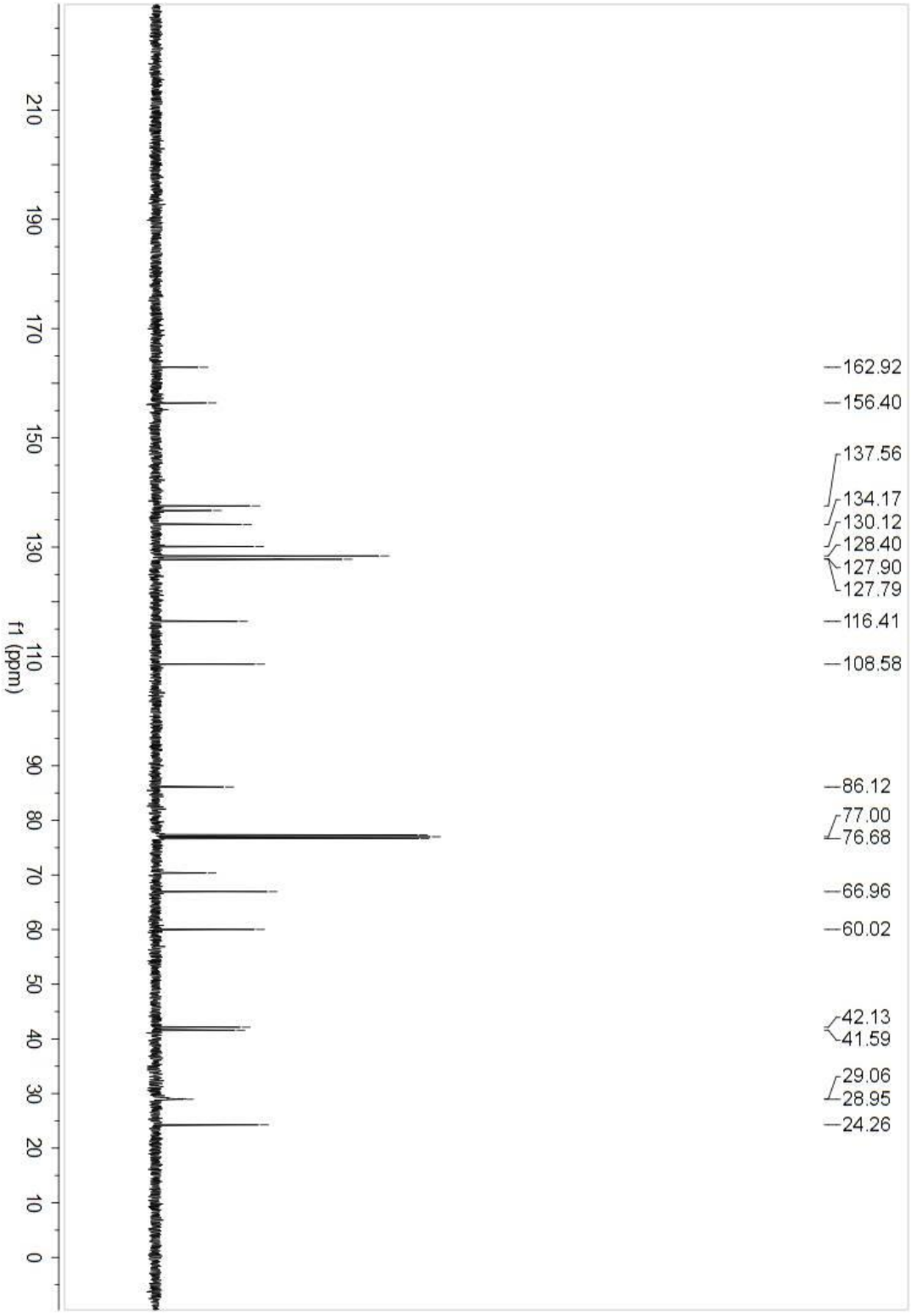

Cbz
N
O
N
N

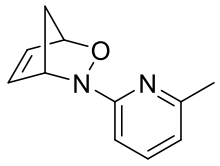
3f


f1 (ppm)
6.00
1.01
0.94
0.99
1.00
2.97
0.91
2.04
2.06
2.90
2.34
2.08
7.382
7.369
7.359
7.312
7.264
6.643
6.581
6.561
6.213
6.003
5.989
5.138
4.732
3.600
3.547
3.458
3.388
2.416
2.042
1.947
1.602
1.560
0.000

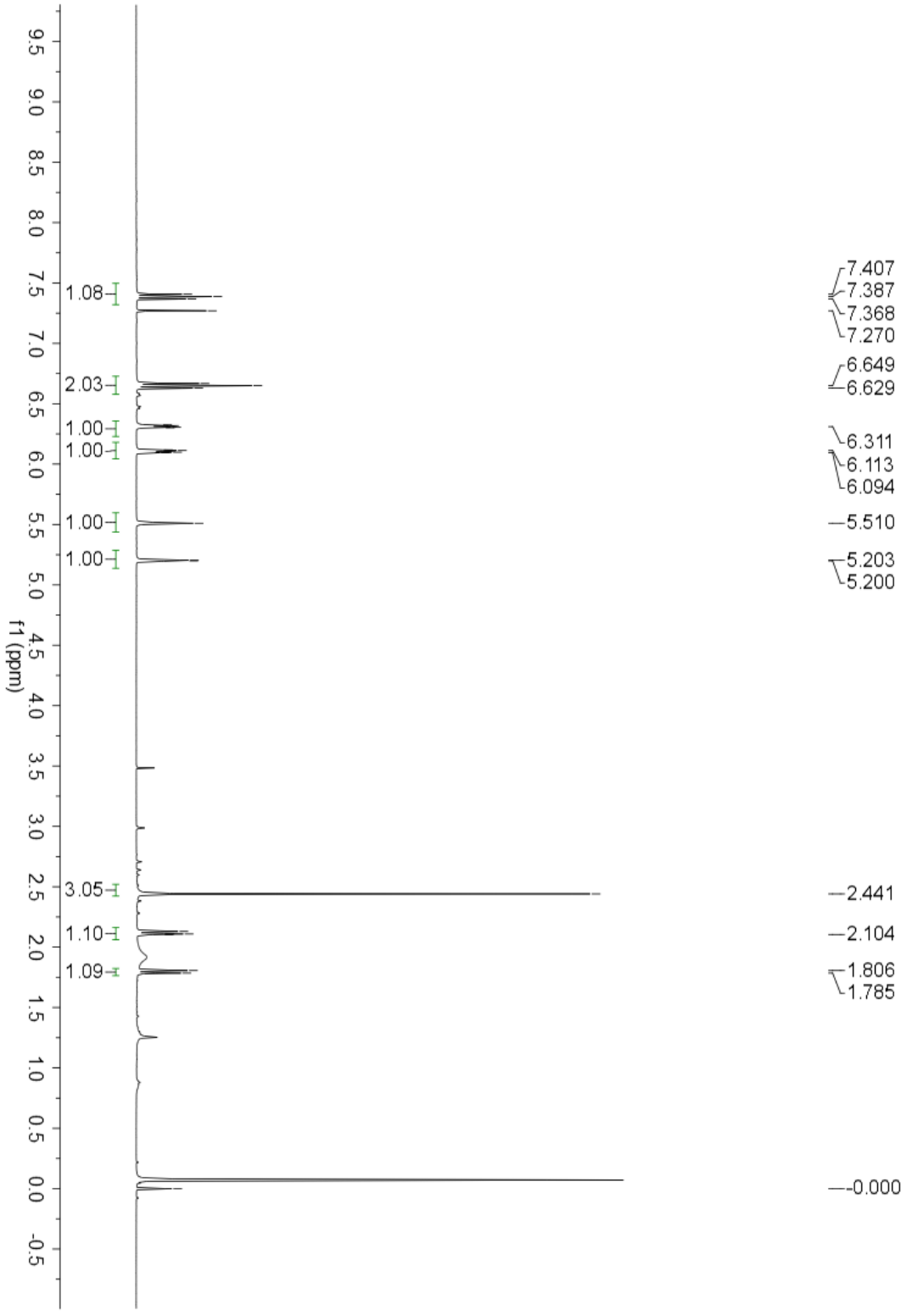

Cbz
N
O
N
N

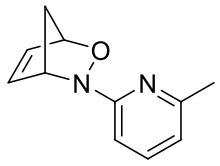
3f


f1 (ppm)
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
162.92
156.40
137.56
134.17
130.12
128.40
127.90
127.79
116.41
108.58
86.12
77.00
76.68
66.96
60.02
42.13
41.59
29.06
28.95
24.26

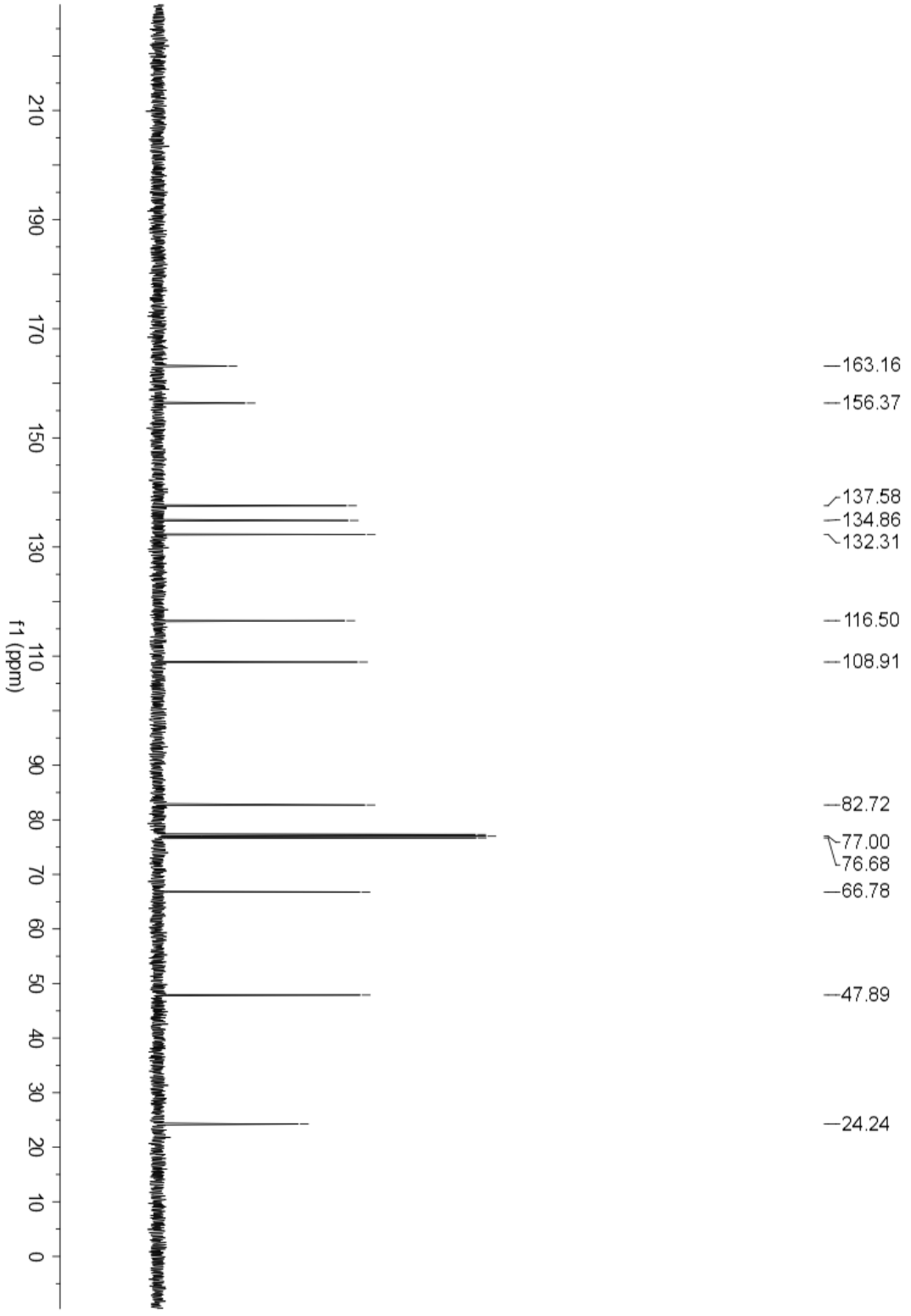


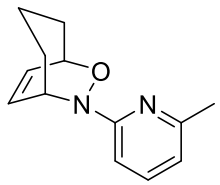
3g


7.407
7.387
7.368
7.270
6.649
6.629
6.311
6.113
6.094
5.510
5.203
5.200
2.441
2.104
1.806
1.785
-0.000
1.08
2.03
1.00
1.00
1.00
1.00
3.05
1.10
1.09
f1 (ppm)

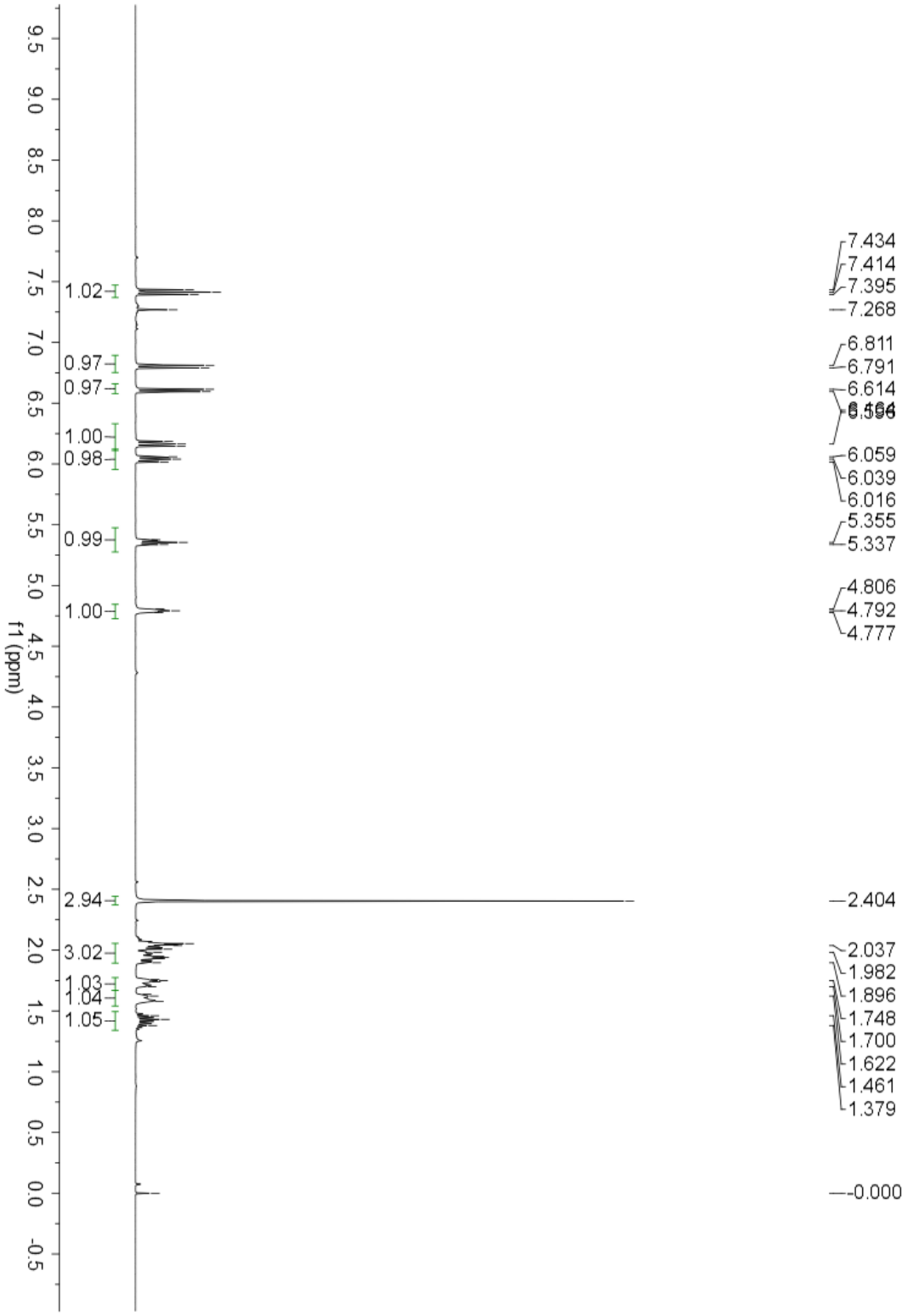


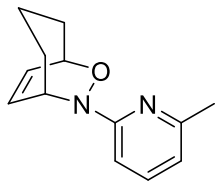
3g


f1 (ppm)
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
163.16
156.37
137.58
134.86
132.31
116.50
108.91
82.72
77.00
76.68
66.78
47.89
24.24

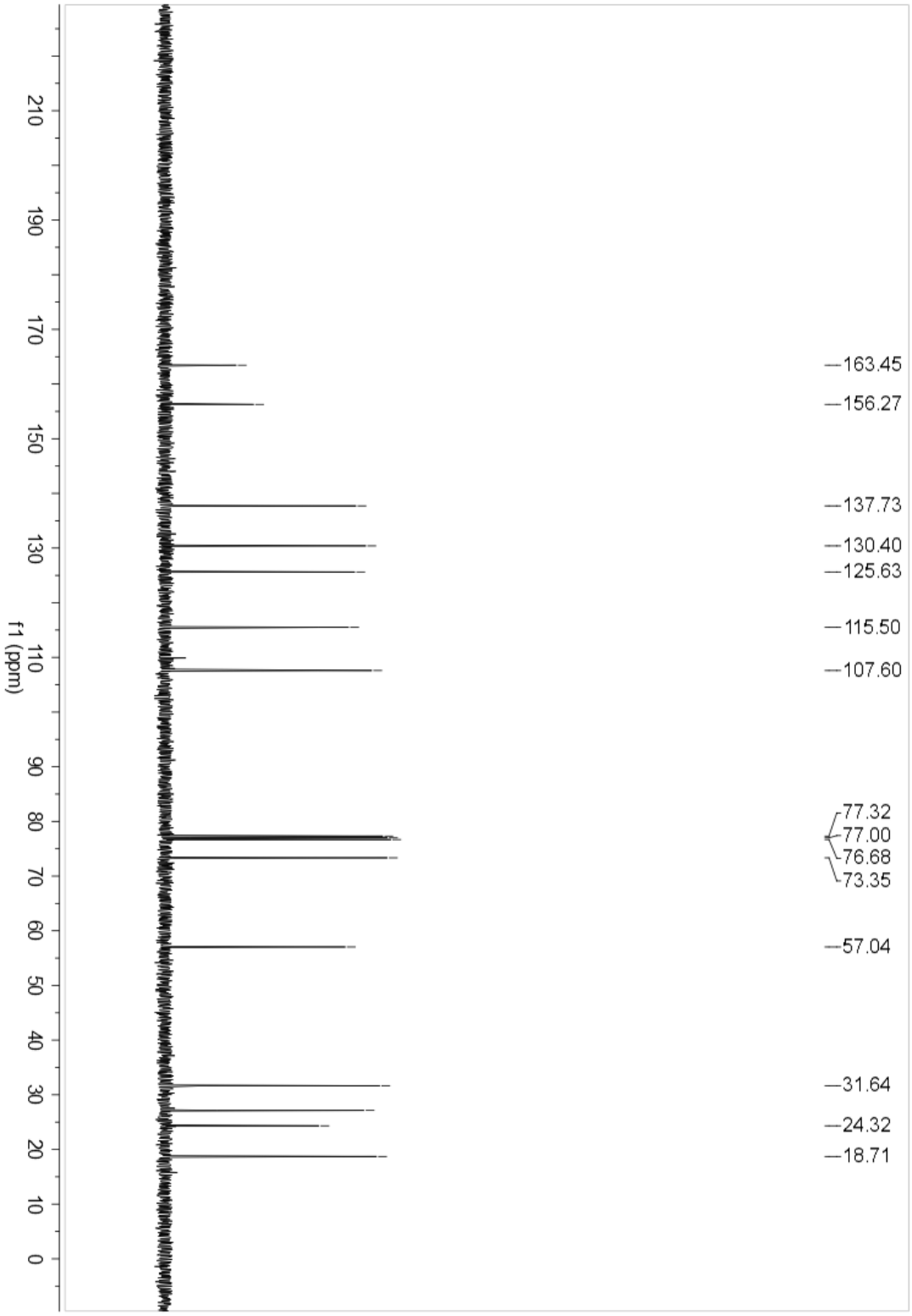


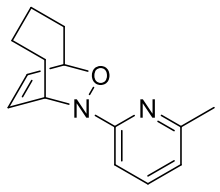
3h


f1 (ppm)
1.02
0.97
0.97
1.00
0.98
0.99
1.00
2.94
3.02
1.03
1.04
1.05
7.434
7.414
7.395
7.268
6.811
6.791
6.614
6.059
6.039
6.016
5.355
5.337
4.806
4.792
4.777
2.404
2.037
1.982
1.896
1.748
1.700
1.622
1.461
1.379
0.000

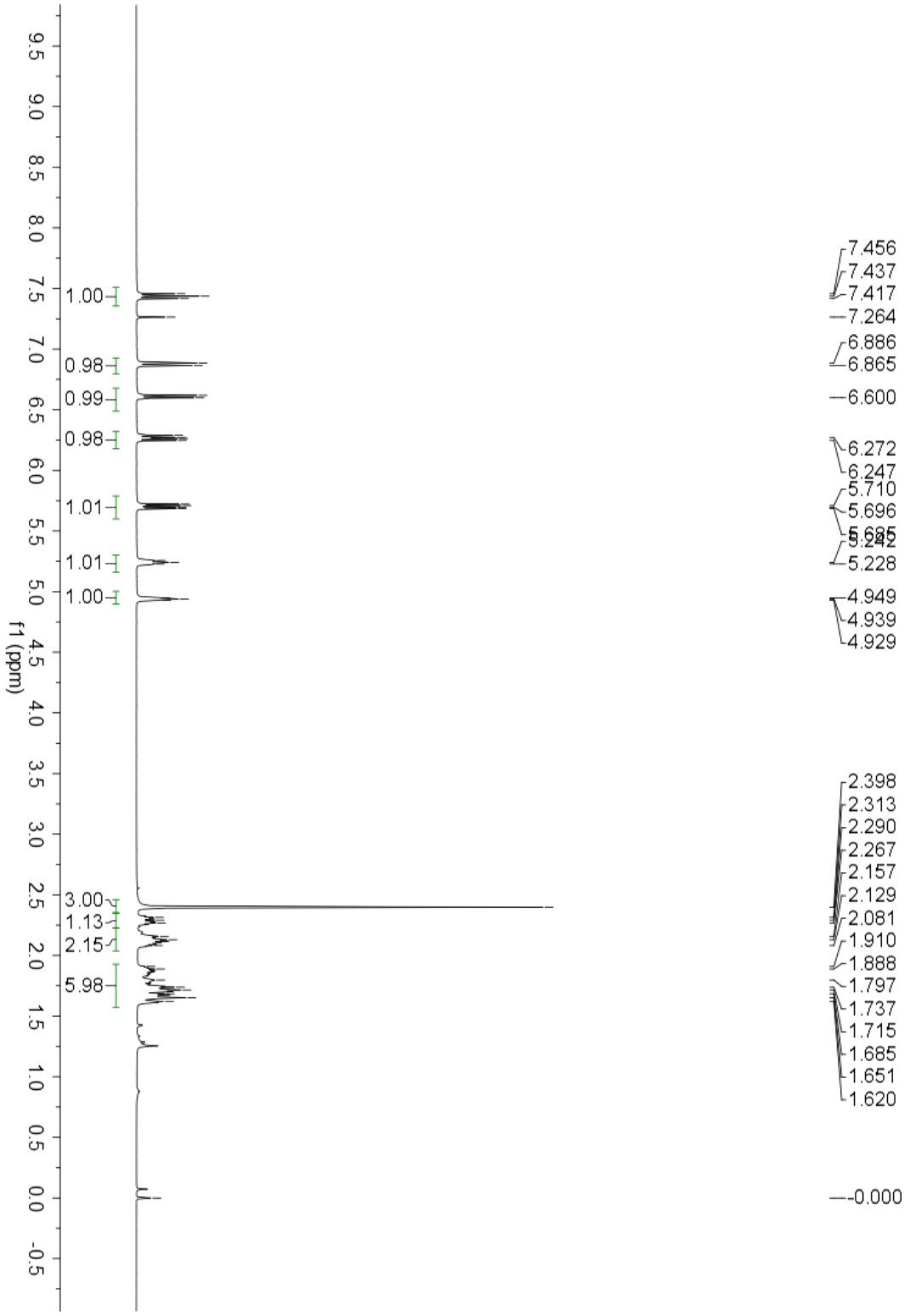


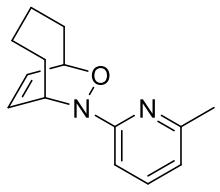
3h


f1 (ppm)
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
163.45
156.27
137.73
130.40
125.63
115.50
107.60
77.32
77.00
76.68
73.35
57.04
31.64
24.32
18.71

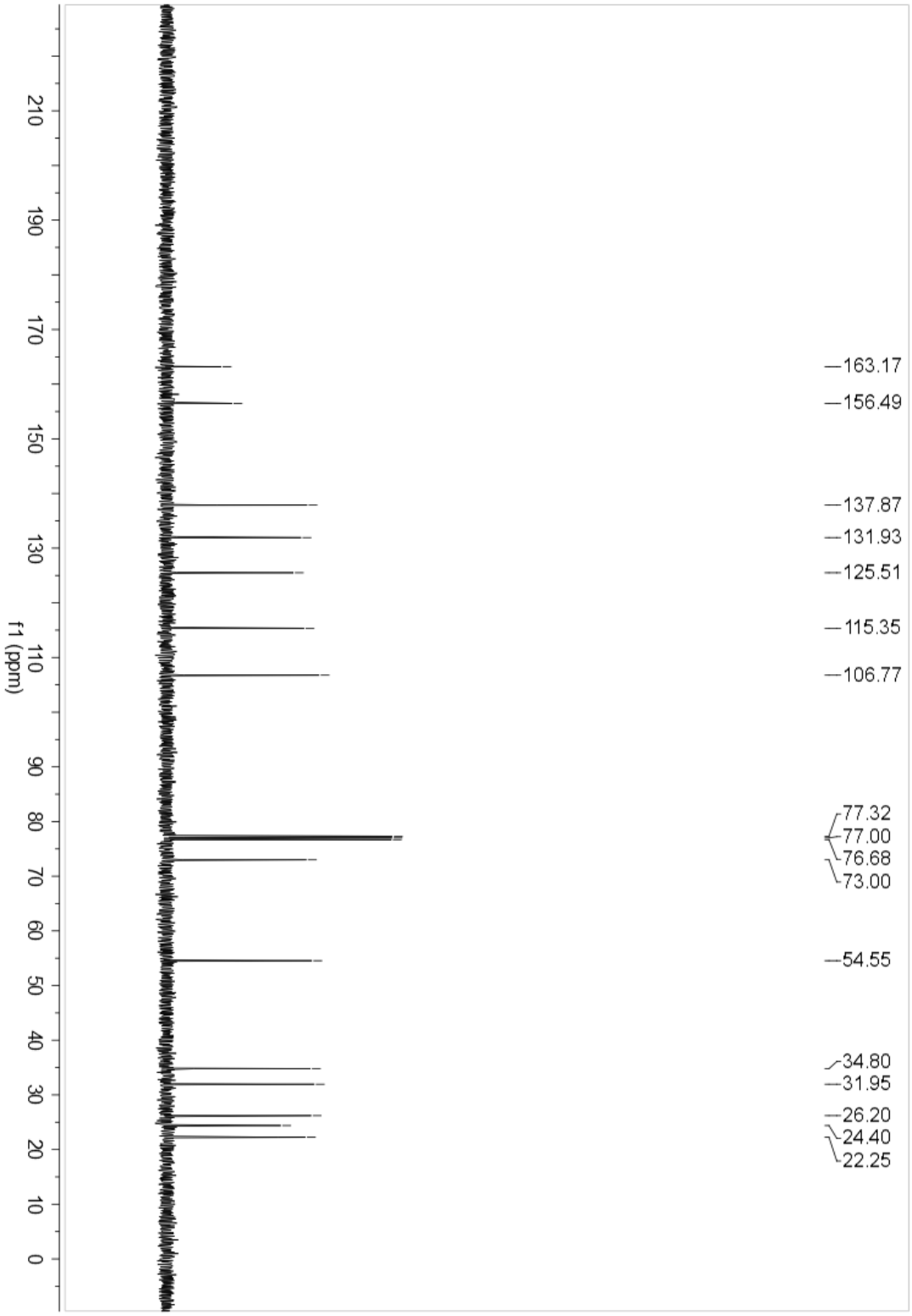


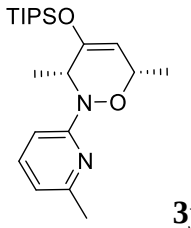
3i


f1 (ppm)
1.00
0.98
0.99
0.98
1.01
1.01
1.00
3.00
1.13
2.15
5.98
7.456
7.437
7.417
7.264
6.886
6.865
6.600
6.272
6.247
5.710
5.696
5.242
5.228
4.949
4.939
4.929
2.398
2.313
2.290
2.267
2.157
2.129
2.081
1.910
1.888
1.797
1.737
1.715
1.685
1.651
1.620
0.000

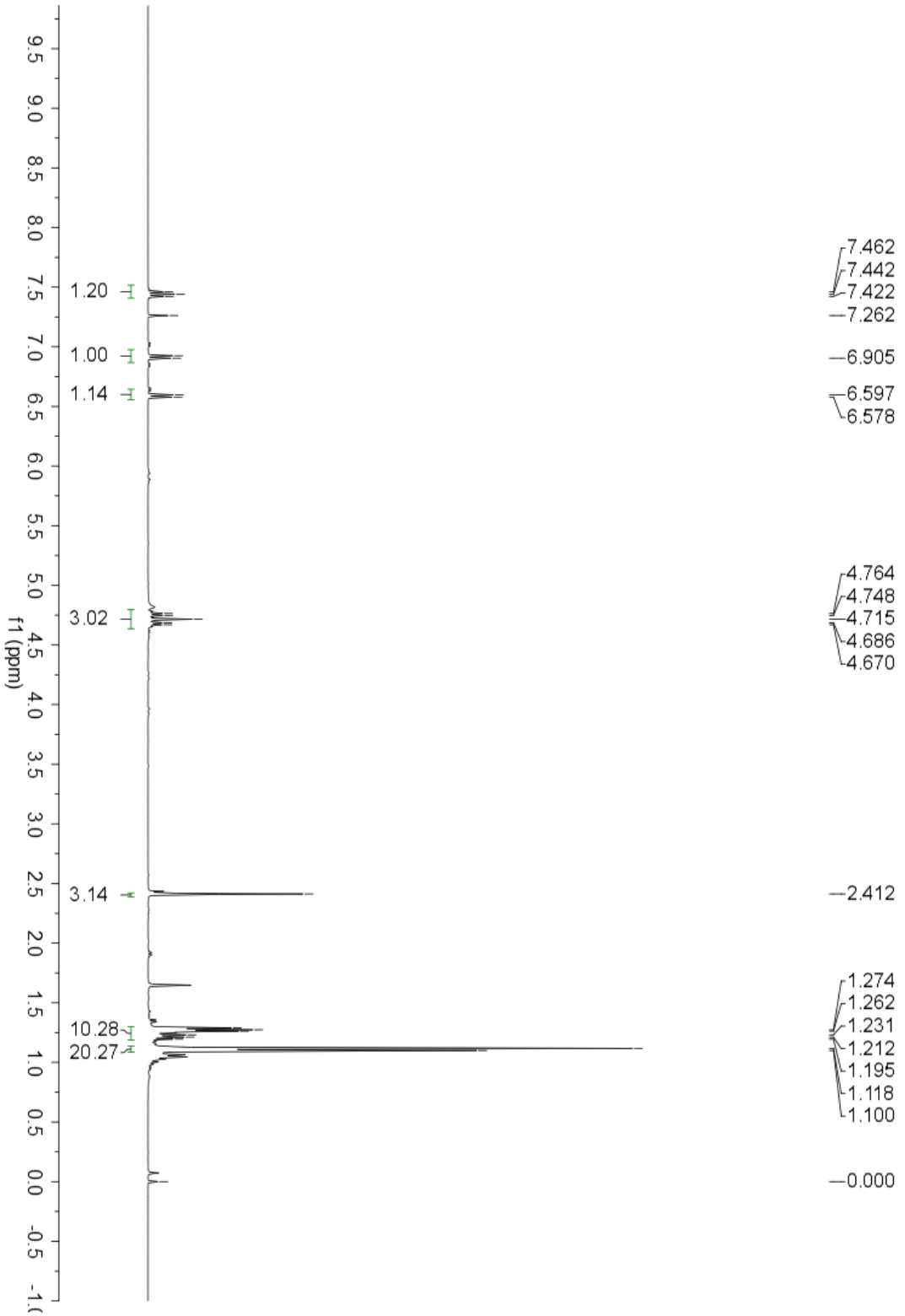


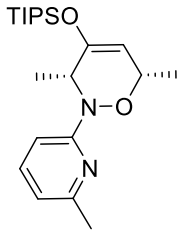
3i


f1 (ppm)
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
163.17
156.49
137.87
131.93
125.51
115.35
106.77
77.32
77.00
76.68
73.00
54.55
34.80
31.95
26.20
24.40
22.25

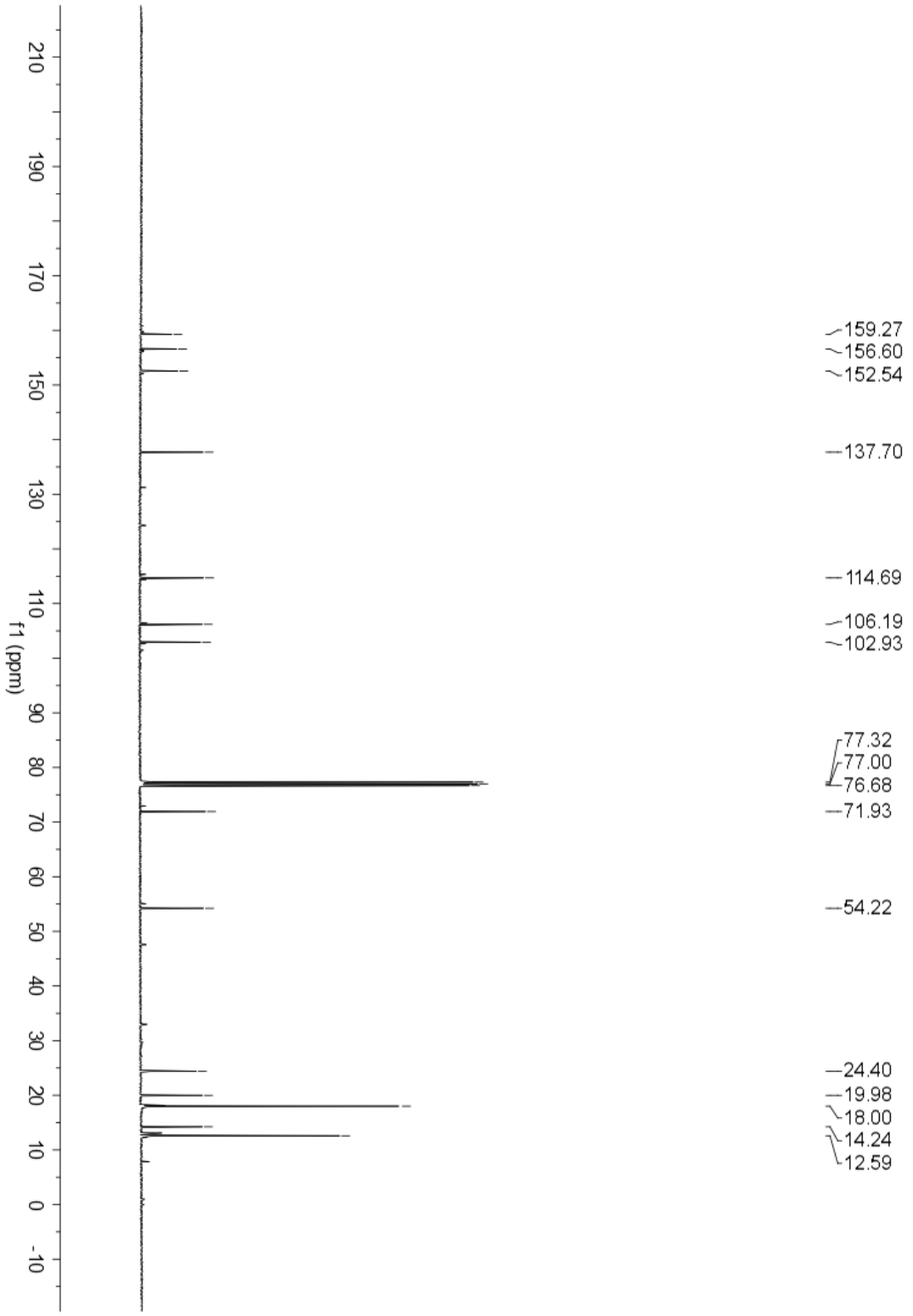

TIPSO
N
O
N

3j

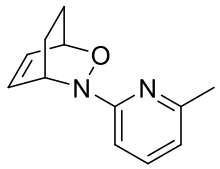

f1 (ppm)
1.20
1.00
1.14
3.02
3.14
10.28
20.27
7.462
7.442
7.422
7.262
6.905
6.597
6.578
4.764
4.748
4.715
4.686
4.670
2.412
1.274
1.262
1.231
1.212
1.195
1.118
1.100
0.000


TIPSO
N–O
N

3j


f1 (ppm)
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
159.27
156.60
152.54
137.70
114.69
106.19
102.93
77.32
77.00
76.68
71.93
54.22
24.40
19.98
18.00
14.24
12.59

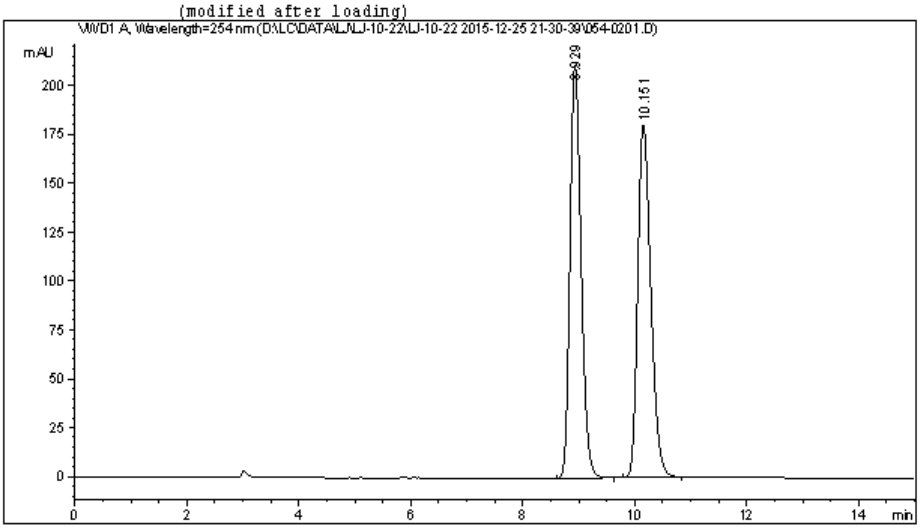
V. HPLC Chromatograms



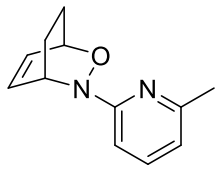
3a

Data File D:\LC\DATA\LJ\LJ-10-22\LJ-10-22 2015-12-25 21-30-39\054-0201.D
Sample Name: LJ-10-22

```
    ==================================================================
    Acq. Operator   : LHC                          Seq. Line :   2
    Acq. Instrument : Instrument 1                  Location : Vial 54
    Injection Date  : 12/25/2015 9:43:08 PM              Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\DATA\LJ\LJ-10-22\LJ-10-22 2015-12-25 21-30-39\ODH-5-95-1ML-254NM-
                      20MIN.M
    Last changed    : 12/16/2015 3:05:20 PM by LHC
    Analysis Method : D:\LC\DATA\LJ\LJ-10-22\LJ-10-22 2015-12-25 21-30-39\054-0201.D\DA.M (ODH-
                      5-95-1ML-254NM-20MIN.M)
    Last changed    : 6/30/2016 4:00:23 PM by LHC
                      (modified after loading)
```



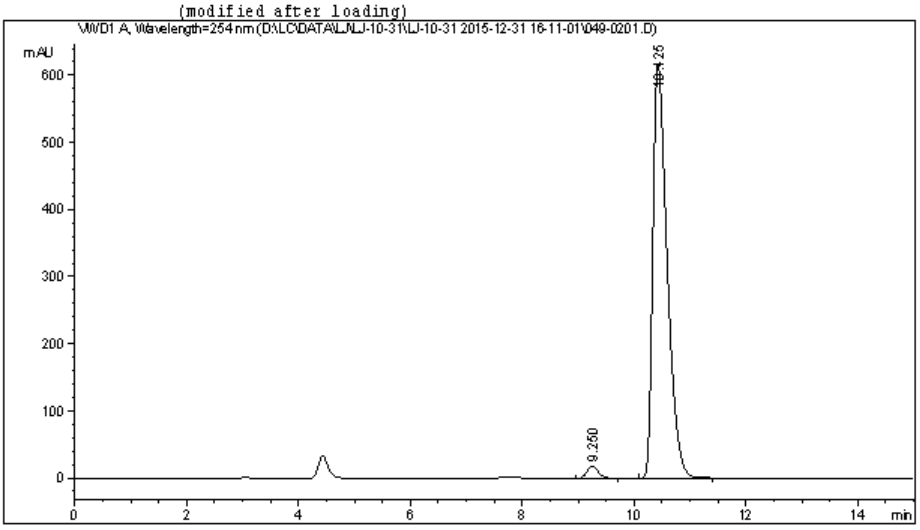

```
    ==================================================================
                            Area Percent Report
    ==================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   8.929 BB    0.2122 2921.22168  211.40381  50.0379
       2  10.151 BB    0.2499 2916.79443  180.24718  49.9621

    Totals :                  5838.01611  391.65099

    ==================================================================
```



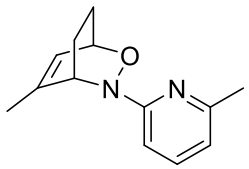
3a

Data File D:\LC\DATA\LJ\LJ-10-31\LJ-10-31 2015-12-31 16-11-01\049-0201.D
Sample Name: C

```
    ==================================================================
    Acq. Operator   : LHC                          Seq. Line :   2
    Acq. Instrument : Instrument 1                  Location : Vial 49
    Injection Date  : 12/31/2015 4:23:50 PM            Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\DATA\LJ\LJ-10-31\LJ-10-31 2015-12-31 16-11-01\ODH-5-95-1ML-254NM-
                      20MIN.M
    Last changed    : 12/16/2015 3:05:20 PM by LHC
    Analysis Method : D:\LC\DATA\LJ\LJ-10-31\LJ-10-31 2015-12-31 16-11-01\049-0201.D\DA.M (ODH-
                      5-95-1ML-254NM-20MIN.M)
    Last changed    : 6/30/2016 4:03:30 PM by LHC
                      (modified after loading)
```



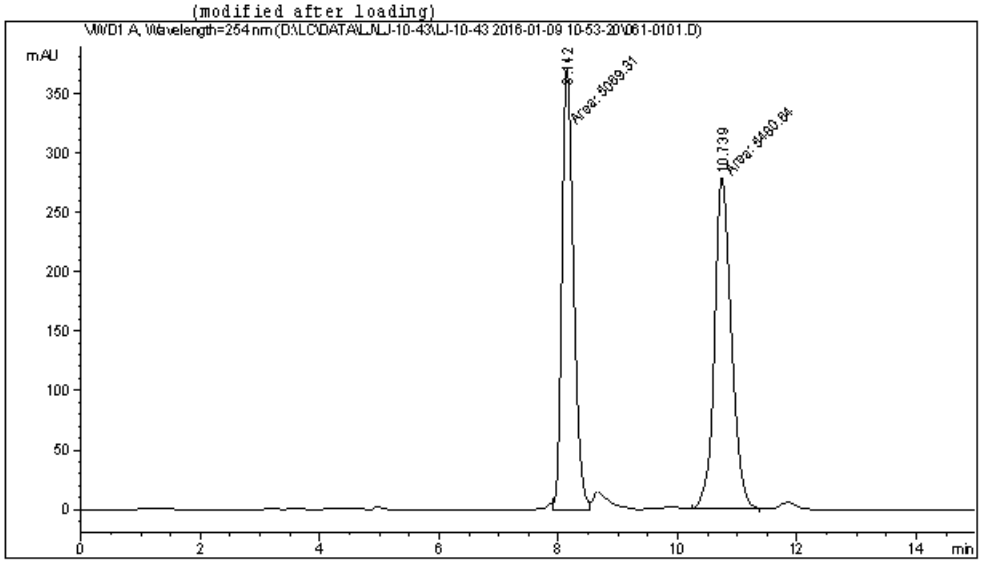

```
    ==================================================================
                            Area Percent Report
    ==================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|------|----|------|----------|----------|-------|
       1   9.250 BB    0.2185  249.83473   17.70325   2.2288
       2  10.425 BB    0.2717 1.09598e4   615.91583  97.7712

    Totals :                  1.12096e4   633.61908

    ==================================================================
```



3b

Data File D:\LC\DATA\LJ\LJ-10-43\LJ-10-43 2016-01-09 10-53-20\061-0101.D
Sample Name: c

Acq. Operator	: LHC	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 61
Injection Date	: 1/9/2016 10:54:45 AM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\DATA\LJ\LJ-10-43\LJ-10-43 2016-01-09 10-53-20\ODH-5-95-1ML-254NM-20MIN.M		
Last changed	: 1/9/2016 10:53:45 AM by LHC (modified after loading)		
Analysis Method	: D:\LC\DATA\LJ\LJ-10-43\LJ-10-43 2016-01-09 10-53-20\061-0101.D\DA.M (ODH-5-95-1ML-254NM-20MIN.M)		
Last changed	: 6/30/2016 7:28:00 PM by LHC (modified after loading)		

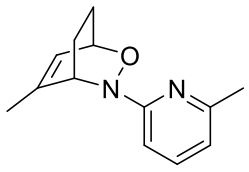


Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

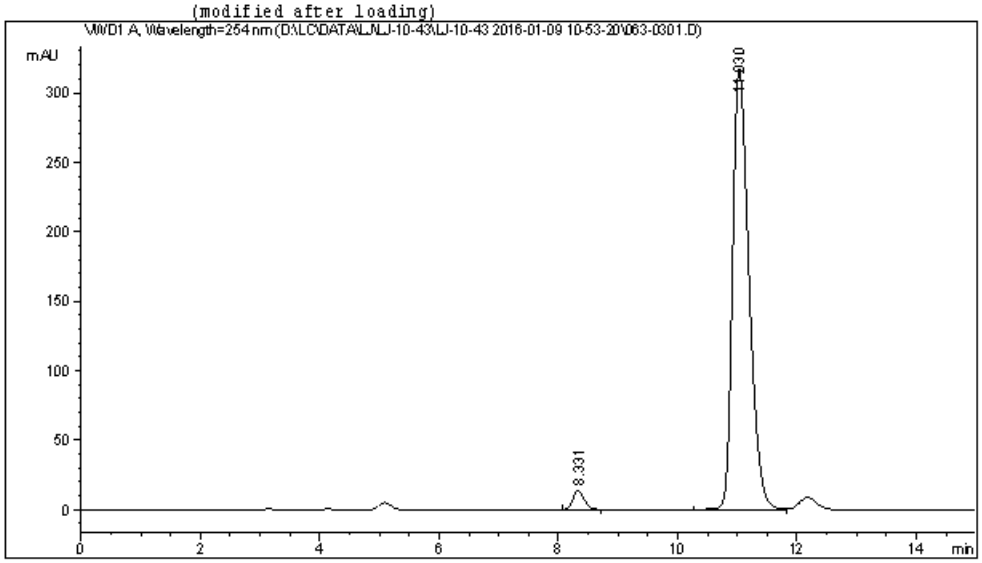
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.142	MF	0.2275	5069.31201	371.41397	48.0506
2	10.739	MM	0.3286	5480.64209	277.96939	51.9494
Totals :				1.05500e4	649.38336	



3b

Data File D:\LC\DATA\LJ\LJ-10-43\LJ-10-43 2016-01-09 10-53-20\063-0301.D
Sample Name: b

Acq. Operator	: LHC	Seq. Line	: 3
Acq. Instrument	: Instrument 1	Location	: Vial 63
Injection Date	: 1/9/2016 11:27:55 AM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\DATA\LJ\LJ-10-43\LJ-10-43 2016-01-09 10-53-20\ODH-5-95-1ML-254NM-20MIN.M		
Last changed	: 1/9/2016 11:26:27 AM by LHC (modified after loading)		
Analysis Method	: D:\LC\DATA\LJ\LJ-10-43\LJ-10-43 2016-01-09 10-53-20\063-0301.D\DA.M (ODH-5-95-1ML-254NM-20MIN.M)		
Last changed	: 6/30/2016 7:24:53 PM by LHC (modified after loading)		

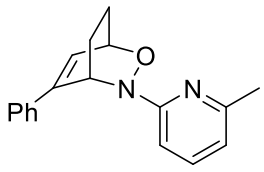


Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

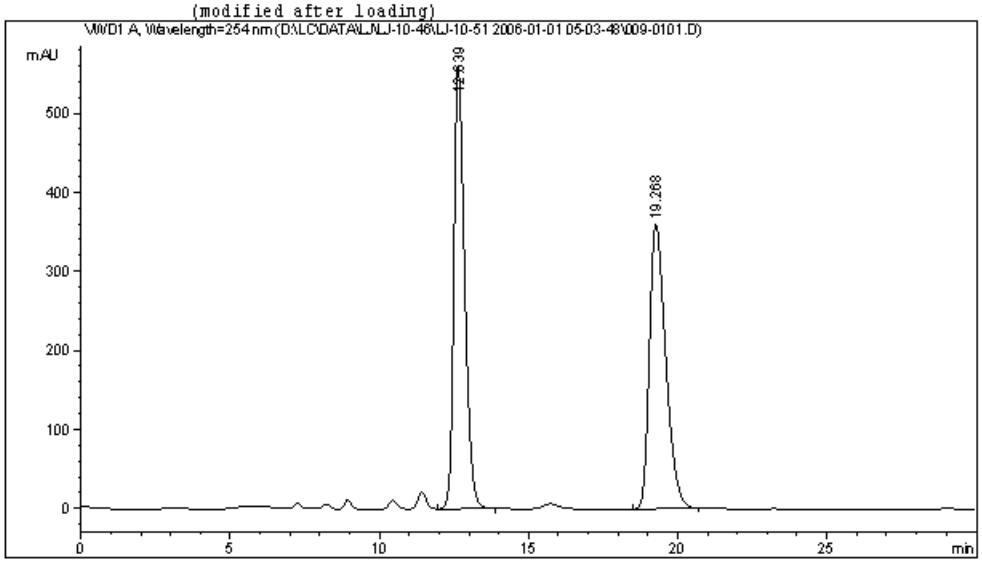
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.331	VB	0.2047	181.26678	13.75265	2.8957
2	11.030	VV	0.2947	6078.59863	317.44678	97.1043
Totals :				6259.86542	331.19943	



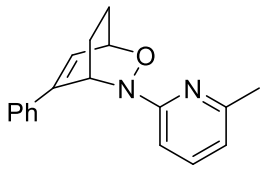
3c

Data File D:\LC\DATA\LJ\LJ-10-46\LJ-10-51 2006-01-01 05-03-48\009-0101.D
Sample Name: LJ-10-51A

```
    ====================================================================
    Acq. Operator   : LHC                          Seq. Line :   1
    Acq. Instrument : Instrument 1                  Location : Vial 9
    Injection Date  : 1/1/2006 5:05:26 AM              Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\DATA\LJ\LJ-10-46\LJ-10-51 2006-01-01 05-03-48\0DH-5-95-1ML-254NM-
                      30MIN.M
    Last changed    : 1/1/2006 5:03:25 AM by LHC
    Analysis Method : D:\LC\DATA\LJ\LJ-10-46\LJ-10-51 2006-01-01 05-03-48\009-0101.D\DA.M (0DH-
                      5-95-1ML-254NM-30MIN.M)
    Last changed    : 10/5/2016 7:22:10 PM by LHC
                      (modified after loading)
```



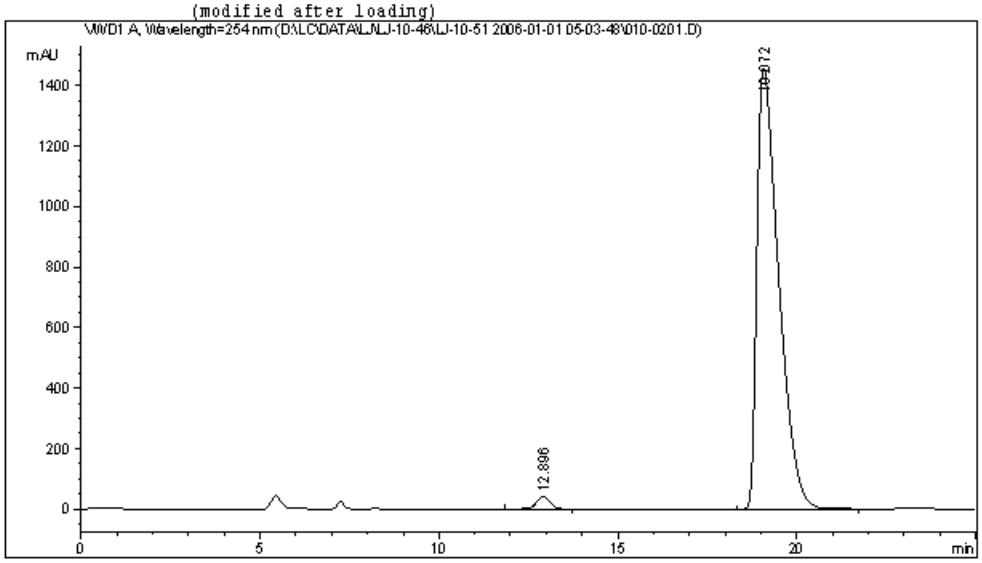

```
    ====================================================================
                            Area Percent Report
    ====================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  12.639 BB    0.3856 1.40430e4   557.47107  50.6786
       2  19.268 BB    0.5787 1.36669e4   359.65460  49.3214

    Totals :                  2.77099e4   917.12567


    ====================================================================
```



3c

Data File D:\LC\DATA\LJ\LJ-10-46\LJ-10-51 2006-01-01 05-03-48\010-0201.D
Sample Name: LJ-10-51B

Acq. Operator	: LHC	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 10
Injection Date	: 1/1/2006 5:37:03 AM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\DATA\LJ\LJ-10-46\LJ-10-51 2006-01-01 05-03-48\0DH-5-95-1ML-254NM-30MIN.M		
Last changed	: 1/1/2006 5:03:25 AM by LHC		
Analysis Method	: D:\LC\DATA\LJ\LJ-10-46\LJ-10-51 2006-01-01 05-03-48\010-0201.D\DA.M (0DH-5-95-1ML-254NM-30MIN.M)		
Last changed	: 6/30/2016 4:14:29 PM by LHC (modified after loading)		

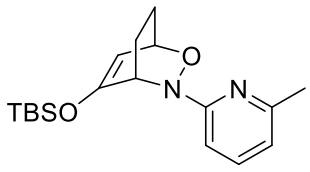


Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

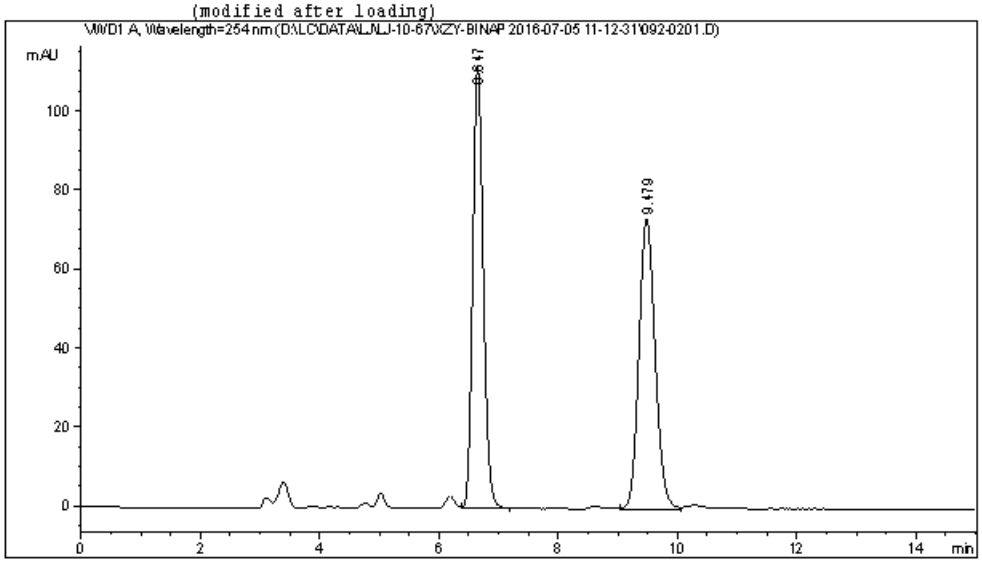
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	12.896	VB	0.4134	1134.27441	41.70160	1.8155
2	19.072	BB	0.6314	6.13435e4	1455.66687	98.1845
Totals :				6.24777e4	1497.36847	



3d

Data File D:\LC\DATA\LJ\LJ-10-67\XZY-BINAP 2016-07-05 11-12-31\092-0201.D
Sample Name: lj-10-67-1

Acq. Operator	: LHC	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 92
Injection Date	: 7/5/2016 11:25:06 AM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\DATA\LJ\LJ-10-67\XZY-BINAP 2016-07-05 11-12-31\ODH-5-95-1ML-254NM-20MIN.M		
Last changed	: 12/16/2015 3:05:20 PM by LHC		
Analysis Method	: D:\LC\DATA\LJ\LJ-10-67\XZY-BINAP 2016-07-05 11-12-31\092-0201.D\DA.M (ODH-5-95-1ML-254NM-20MIN.M)		
Last changed	: 7/6/2016 10:32:56 AM by LHC (modified after loading)		



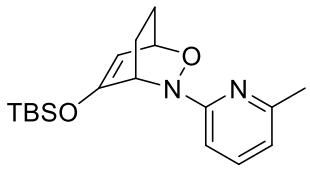
Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

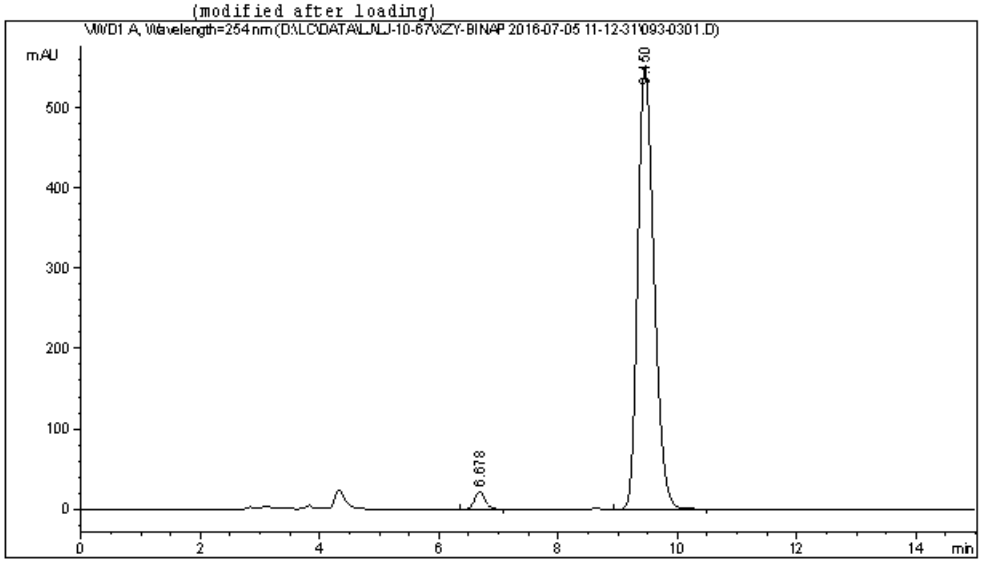
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.647	VB	0.1903	1371.15271	111.32205	49.7800
2	9.479	BV	0.2929	1383.27271	73.32944	50.2200
Totals :				2754.42542	184.65150	



3d

Data File D:\LC\DATA\LJ\LJ-10-67\XZY-BINAP 2016-07-05 11-12-31\093-0301.D
Sample Name: lj-10-67-2

Acq. Operator	: LHC	Seq. Line	: 3
Acq. Instrument	: Instrument 1	Location	: Vial 93
Injection Date	: 7/5/2016 11:46:28 AM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\DATA\LJ\LJ-10-67\XZY-BINAP 2016-07-05 11-12-31\ODH-5-95-1ML-254NM-20MIN.M		
Last changed	: 12/16/2015 3:05:20 PM by LHC		
Analysis Method	: D:\LC\DATA\LJ\LJ-10-67\XZY-BINAP 2016-07-05 11-12-31\093-0301.D\DA.M (ODH-5-95-1ML-254NM-20MIN.M)		
Last changed	: 7/6/2016 10:35:08 AM by LHC (modified after loading)		



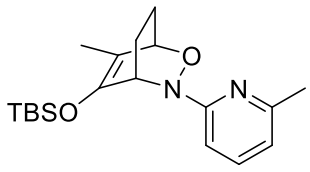
Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.678	VB	0.1906	269.37753	21.81923	2.5131
2	9.450	VB	0.2934	1.04495e4	548.95526	97.4869
Totals :				1.07189e4	570.77449	

Instrument 1 7/6/2016 10:35:14 AM LHC



3e

Data File D:\LC\DATA\LJ\LJ-11-51\LJ-11-51 2016-05-04 16-42-30\012-0201.D
Sample Name: LJ-11-51

```
    ====================================================================
    Acq. Operator   : LHC                          Seq. Line :   2
    Acq. Instrument : Instrument 1                  Location : Vial 12
    Injection Date  : 5/4/2016 4:54:55 PM              Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\DATA\LJ\LJ-11-51\LJ-11-51 2016-05-04 16-42-30\0DH-5-95-1ML-254NM-
                      30MIN.M
    Last changed    : 1/1/2006 5:03:25 AM by LHC
    Analysis Method : D:\LC\DATA\LJ\LJ-11-51\LJ-11-51 2016-05-04 16-42-30\012-0201.D\DA.M (0DH-
                      5-95-1ML-254NM-30MIN.M)
    Last changed    : 10/5/2016 8:35:29 PM by LHC
                      (modified after loading)
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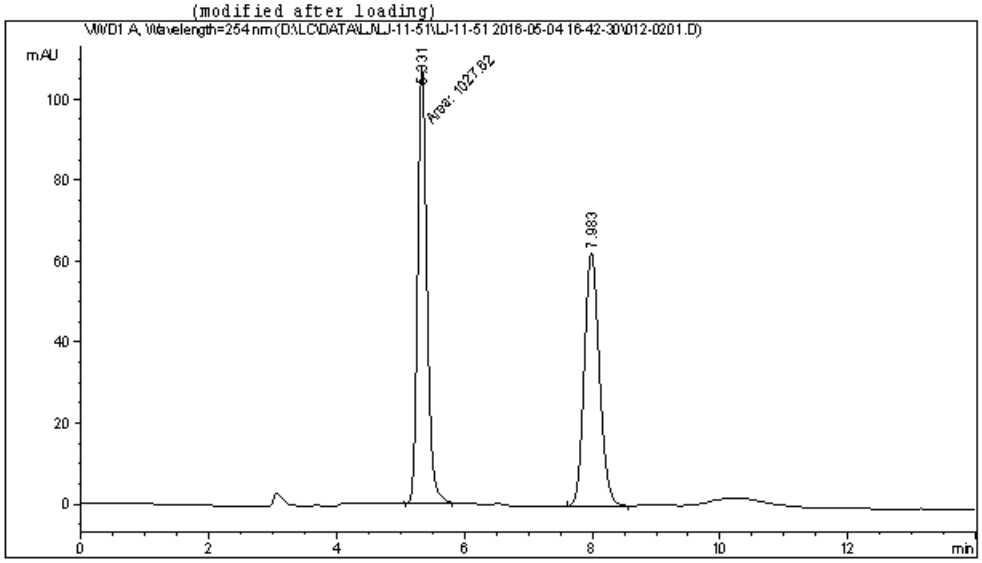



```
    ====================================================================
                            Area Percent Report
    ====================================================================

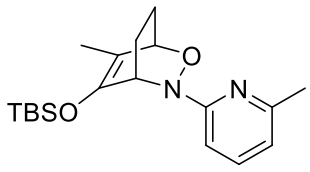
    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   5.331 MM    0.1628 1027.62244  105.21992  49.7920
       2   7.983 BB    0.2543 1036.20618   62.59076  50.2080

    Totals :                   2063.82861  167.81068

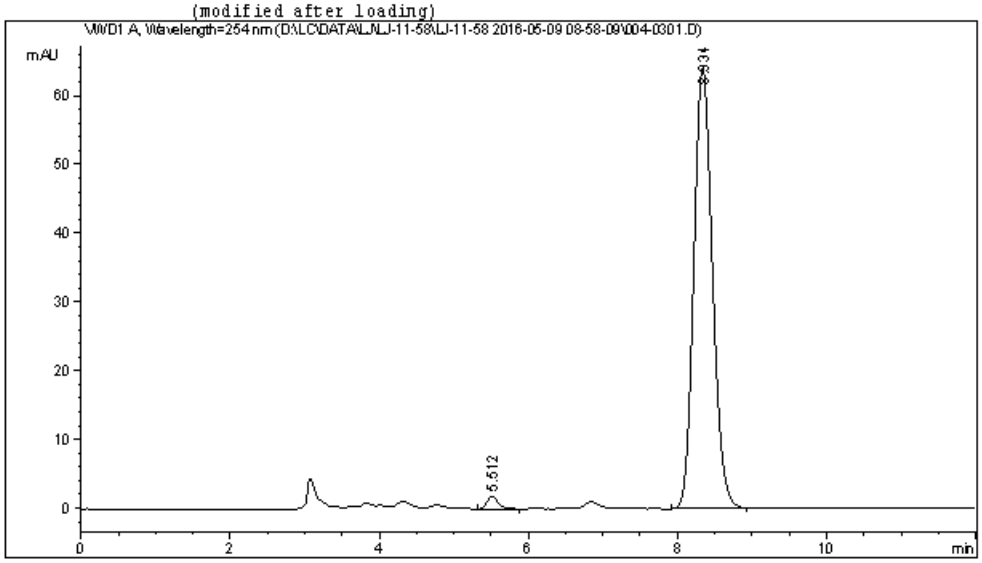

    ====================================================================
```



3e

Data File D:\LC\DATA\LJ\LJ-11-58\LJ-11-58 2016-05-09 08-58-09\004-0301.D
Sample Name: LJ-11-58

```
    ====================================================================
    Acq. Operator   : LHC                          Seq. Line :   3
    Acq. Instrument : Instrument 1                  Location : Vial 4
    Injection Date  : 5/9/2016 9:32:26 AM              Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\DATA\LJ\LJ-11-58\LJ-11-58 2016-05-09 08-58-09\0DH-5-95-1ML-254NM-
                      20MIN.M
    Last changed    : 12/16/2015 3:05:20 PM by LHC
    Analysis Method : D:\LC\DATA\LJ\LJ-11-58\LJ-11-58 2016-05-09 08-58-09\004-0301.D\DA.M (0DH-
                      5-95-1ML-254NM-20MIN.M)
    Last changed    : 10/5/2016 7:35:04 PM by LHC
                      (modified after loading)
```




```
    ====================================================================
                            Area Percent Report
    ====================================================================

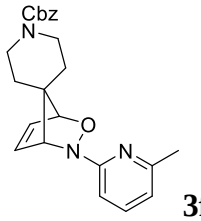
    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   5.512 BB    0.1615   19.27642    1.81834   1.7045
       2   8.334 BB    0.2693 1111.63354   64.09818  98.2955

    Totals :                   1130.90996   65.91652


    ====================================================================
```

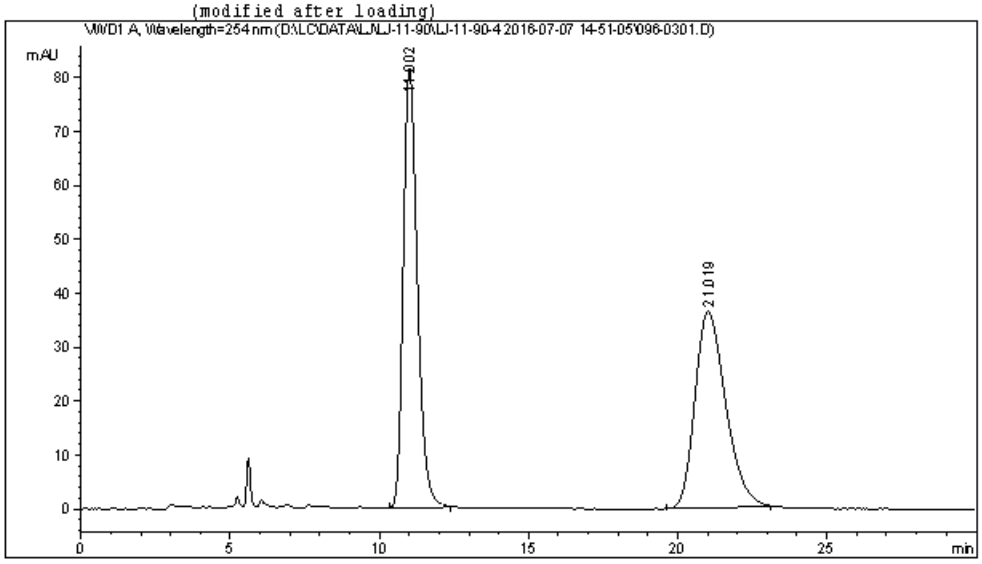


3f

Data File D:\LC\DATA\LJ\LJ-11-90\LJ-11-90-4 2016-07-07 14-51-05\096-0301.D
Sample Name: LJ-11-90-1

==========

Acq. Operator : LHC Seq. Line : 3
Acq. Instrument : Instrument 1 Location : Vial 96
Injection Date : 7/7/2016 3:35:13 PM Inj : 1
Inj Volume : 5 µl
Acq. Method : D:\LC\DATA\LJ\LJ-11-90\LJ-11-90-4 2016-07-07 14-51-05\0DH-20-80-1ML-254NM-30MIN.M
Last changed : 1/1/2006 1:57:18 AM by LHC
Analysis Method : D:\LC\DATA\LJ\LJ-11-90\LJ-11-90-4 2016-07-07 14-51-05\096-0301.D\DA.M (0DH-20-80-1ML-254NM-30MIN.M)
Last changed : 10/5/2016 6:33:57 PM by LHC
(modified after loading)



==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	11.002	BB	0.5043	2667.27710	81.46521	50.5340
2	21.019	BB	1.0547	2610.91016	36.42158	49.4660

Totals : 5278.18726 117.88679

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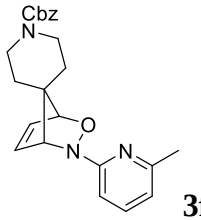
Instrument 1 10/5/2016 6:34:03 PM LHC Page 1 of 1



3f

Data File D:\LC\DATA\LJ\LJ-11-90\LJ-11-90-4 2016-07-07 14-51-05\098-0201.D
Sample Name: LJ-11-90-2

```
==========================================================================
Acq. Operator   : LHC                              Seq. Line :   2
Acq. Instrument : Instrument 1                      Location : Vial 98
Injection Date  : 7/7/2016 3:03:37 PM                  Inj :   1
                                                  Inj Volume : 5 µl
Acq. Method     : D:\LC\DATA\LJ\LJ-11-90\LJ-11-90-4 2016-07-07 14-51-05\0DH-20-80-1ML-254NM-
                  30MIN.M
Last changed    : 1/1/2006 1:57:18 AM by LHC
Analysis Method : D:\LC\DATA\LJ\LJ-11-90\LJ-11-90-4 2016-07-07 14-51-05\098-0201.D\DA.M (
                  0DH-20-80-1ML-254NM-30MIN.M)
Last changed    : 10/5/2016 6:44:17 PM by LHC
                  (modified after loading)
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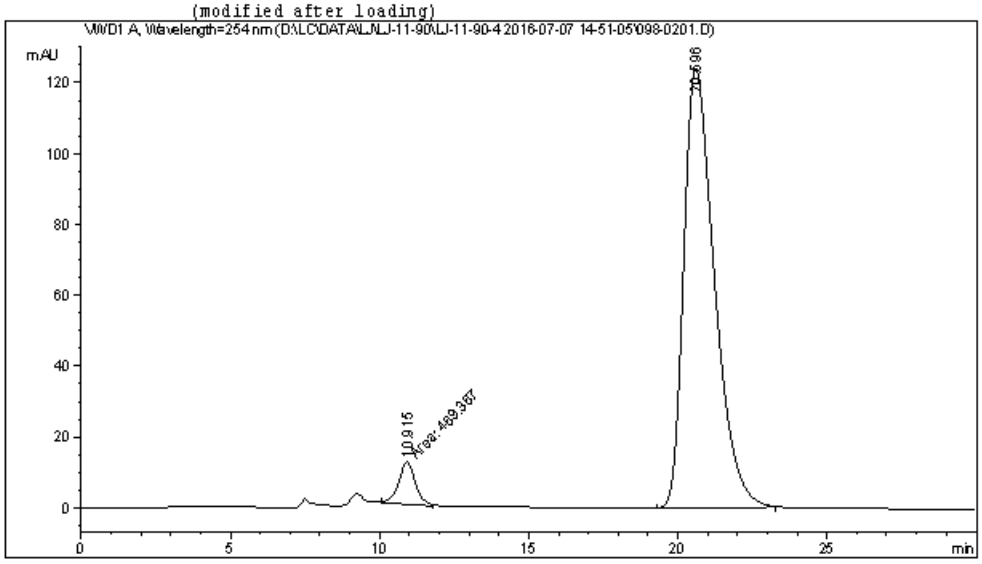
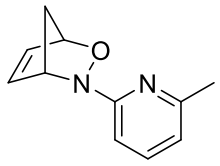



```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s    [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  10.915 MM    0.6517  469.36676   12.00426   5.0319
   2  20.596 BB    1.0799 8858.53125  124.16433  94.9681

Totals :                  9327.89801  136.16859

==========================================================================
```



3g

Data File D:\LC\DATA\LJ\LJ-10-9\LJ-10-9 2015-12-16 20-46-49\005-0201.D
Sample Name: LJ-10-9A

```
    ====================================================================
    Acq. Operator   : LHC                          Seq. Line :   2
    Acq. Instrument : Instrument 1                  Location : Vial 5
    Injection Date  : 12/16/2015 8:59:31 PM            Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\DATA\LJ\LJ-10-9\LJ-10-9 2015-12-16 20-46-49\ODH-5-95-1ML-254NM-
                      20MIN.M
    Last changed    : 12/16/2015 3:05:20 PM by LHC
    Analysis Method : D:\LC\DATA\LJ\LJ-10-9\LJ-10-9 2015-12-16 20-46-49\005-0201.D\DA.M (ODH-5-
                      95-1ML-254NM-20MIN.M)
    Last changed    : 6/30/2016 3:55:15 PM by LHC
                      (modified after loading)
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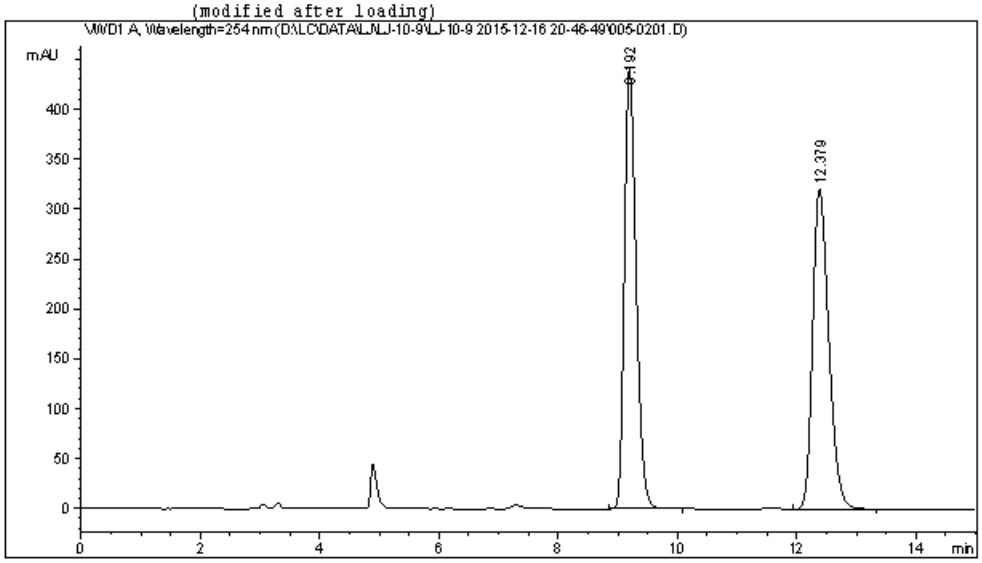



```
    ====================================================================
                            Area Percent Report
    ====================================================================

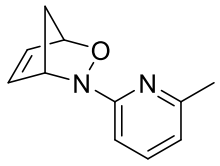
    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   9.192 BB    0.2117 6031.13477  441.73480  50.0634
       2  12.379 VB    0.2902 6015.87012  320.68643  49.9366

    Totals :                  1.20470e4   762.42123


    ====================================================================
```



3g

Data File D:\LC\DATA\LJ\LJ-10-9\LJ-10-9B 2015-12-16 22-13-24\006-0101.D
Sample Name: LJ-10-9B

```
    ====================================================================
    Acq. Operator   : LHC                          Seq. Line :   1
    Acq. Instrument : Instrument 1                  Location : Vial 6
    Injection Date  : 12/16/2015 10:14:49 PM           Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\DATA\LJ\LJ-10-9\LJ-10-9B 2015-12-16 22-13-24\ODH-5-95-1ML-254NM-
                      20MIN.M
    Last changed    : 12/16/2015 3:05:20 PM by LHC
    Analysis Method : D:\LC\DATA\LJ\LJ-10-9\LJ-10-9B 2015-12-16 22-13-24\006-0101.D\DA.M (ODH-5-
                      95-1ML-254NM-20MIN.M)
    Last changed    : 6/30/2016 3:58:07 PM by LHC
                      (modified after loading)
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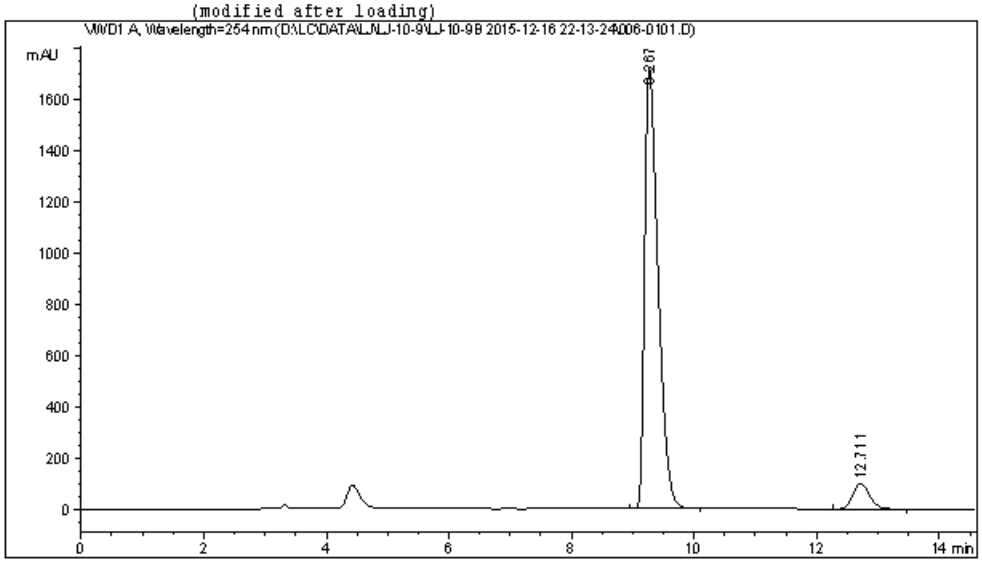
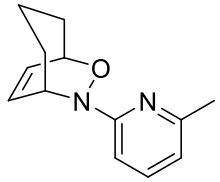



```
    ====================================================================
                            Area Percent Report
    ====================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   9.267 BB    0.2333 2.62811e4  1723.06848  93.2153
       2  12.711 VB    0.2942 1912.88696  100.81892   6.7847

    Totals :                  2.81940e4  1823.88741

    ====================================================================
```

Instrument 1 6/30/2016 3:58:15 PM LHC Page 1 of 1



3h

Data File D:\LC\DATA\LJ\LJ-11-79\LJ-11-79 2016-05-23 18-17-38\003-0801.D
Sample Name: LJ-11-79B

```
    ====================================================================
    Acq. Operator   : WZF                          Seq. Line :   8
    Acq. Instrument : Instrument 1                  Location : Vial 3
    Injection Date  : 5/23/2016 7:54:12 PM             Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\DATA\LJ\LJ-11-79\LJ-11-79 2016-05-23 18-17-38\ODH-5-95-1ML-254NM-
                      20MIN.M
    Last changed    : 5/23/2016 7:52:54 PM by WZF
                      (modified after loading)
    Analysis Method : D:\LC\DATA\LJ\LJ-11-79\LJ-11-79 2016-05-23 18-17-38\003-0801.D\DA.M (ODH-
                      5-95-1ML-254NM-20MIN.M)
    Last changed    : 10/5/2016 6:57:27 PM by LHC
                      (modified after loading)
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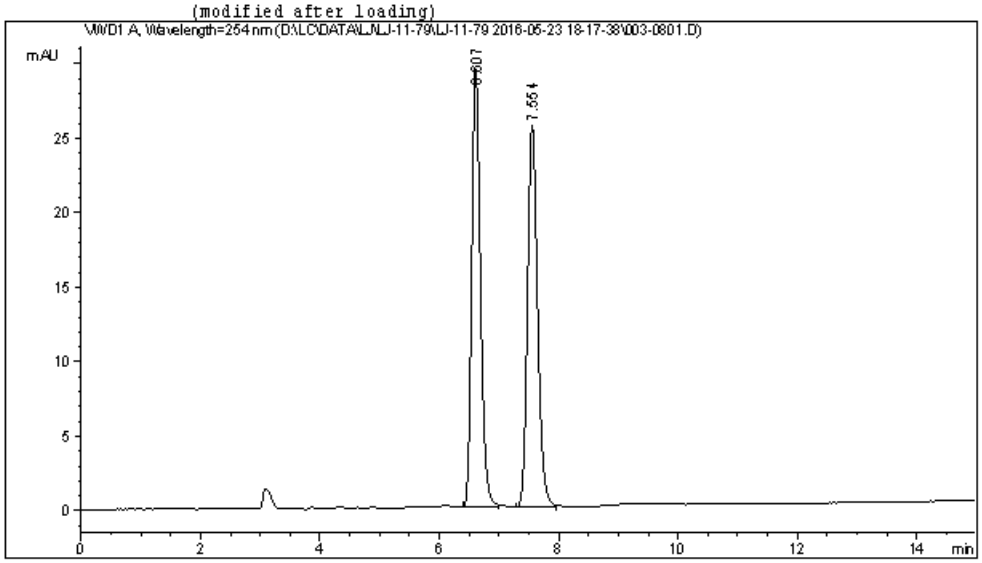


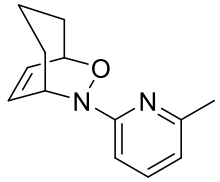

```
    ====================================================================
                            Area Percent Report
    ====================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   6.607 BB    0.1565  298.76682   29.37936  50.0087
       2   7.554 BB    0.1797  298.66275   25.61117  49.9913

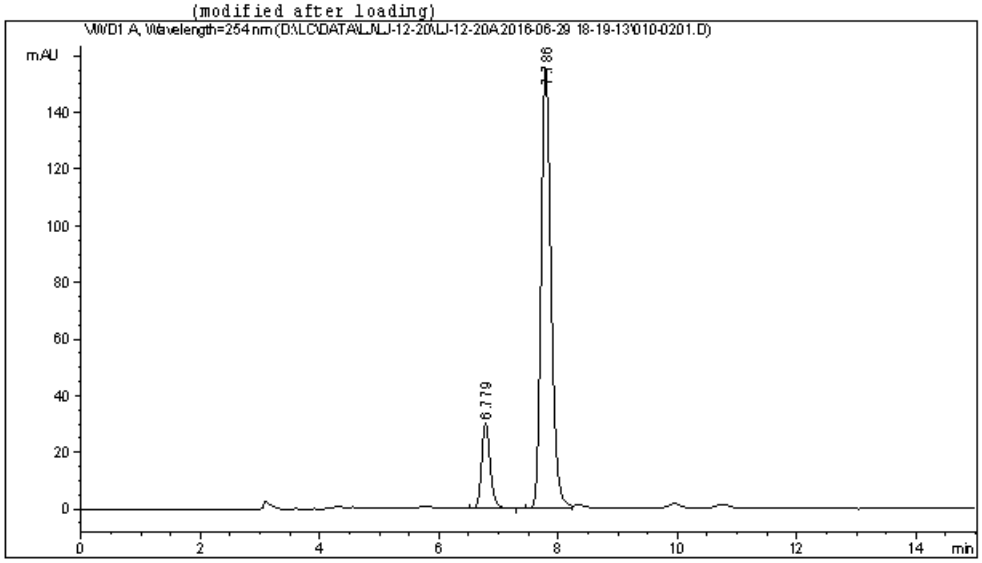
    Totals :                   597.42957   54.99053
```



3h

Data File D:\LC\DATA\LJ\LJ-12-20\LJ-12-20A 2016-06-29 18-19-13\010-0201.D
Sample Name: LJ-12-21A

Acq. Operator	: LHC	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 10
Injection Date	: 6/29/2016 6:31:47 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\DATA\LJ\LJ-12-20\LJ-12-20A 2016-06-29 18-19-13\ODH-5-95-1ML-254NM-20MIN.M		
Last changed	: 12/16/2015 3:05:20 PM by LHC		
Analysis Method	: D:\LC\DATA\LJ\LJ-12-20\LJ-12-20A 2016-06-29 18-19-13\010-0201.D\DA.M (ODH-5-95-1ML-254NM-20MIN.M)		
Last changed	: 10/5/2016 6:59:09 PM by LHC (modified after loading)		

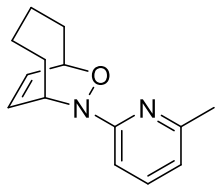


Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.779	VB	0.1572	307.61816	30.07200	14.4776
2	7.786	BV	0.1786	1817.16223	155.50630	85.5224
Totals :				2124.78040	185.57830	

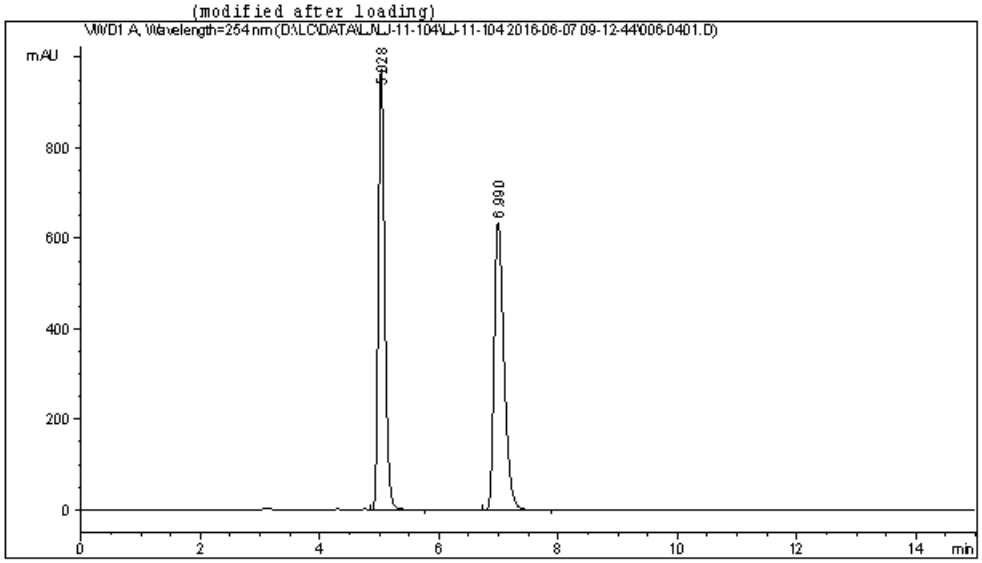


3i

Data File D:\LC\DATA\LJ\LJ-11-104\LJ-11-104 2016-06-07 09-12-44\006-0401.D
Sample Name: LJ-11-103-1

==========

Acq. Operator : HR Seq. Line : 4
Acq. Instrument : Instrument 1 Location : Vial 6
Injection Date : 6/7/2016 10:08:23 AM Inj : 1
Inj Volume : 5 µl
Acq. Method : D:\LC\DATA\LJ\LJ-11-104\LJ-11-104 2016-06-07 09-12-44\0DH-5-95-1ML-254NM-30MIN.M
Last changed : 6/7/2016 10:07:06 AM by HR
(modified after loading)
Analysis Method : D:\LC\DATA\LJ\LJ-11-104\LJ-11-104 2016-06-07 09-12-44\006-0401.D\DA.M (0DH-5-95-1ML-254NM-30MIN.M)
Last changed : 7/6/2016 4:26:51 PM by LHC
(modified after loading)



==========

Area Percent Report

==========

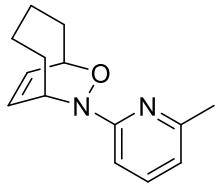
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	5.028	VB	0.1144	7243.91504	974.32288	49.8337
2	6.990	BB	0.1760	7292.25342	636.03137	50.1663

Totals : 1.45362e4 1610.35425

Instrument 1 7/6/2016 4:26:56 PM LHC Page 1 of 2

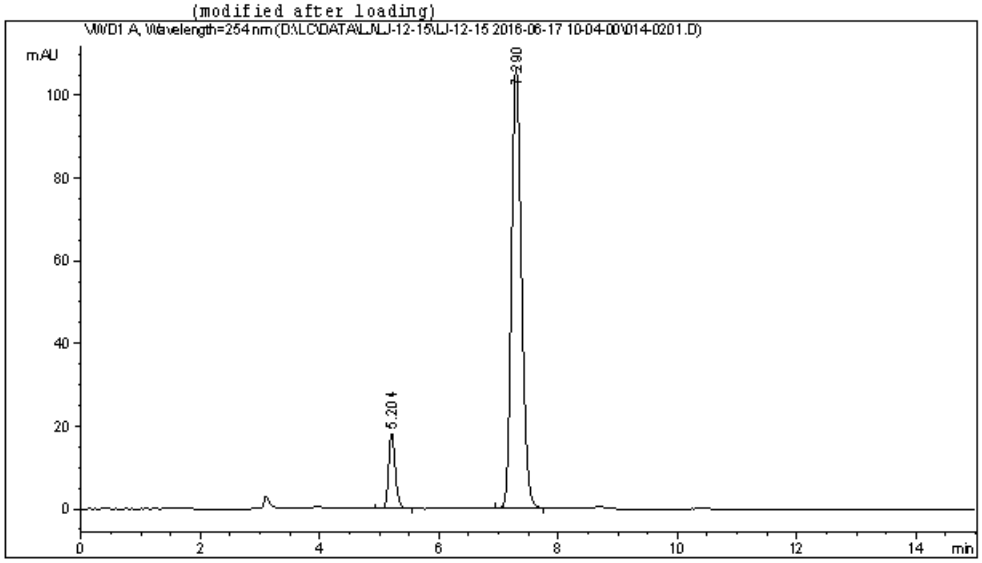


3i

Data File D:\LC\DATA\LJ\LJ-12-15\LJ-12-15 2016-06-17 10-04-00\014-0201.D
Sample Name: LJ-12-15-3

==========

Acq. Operator : LHC Seq. Line : 2
Acq. Instrument : Instrument 1 Location : Vial 14
Injection Date : 6/17/2016 10:16:29 AM Inj : 1
Inj Volume : 5 µl
Acq. Method : D:\LC\DATA\LJ\LJ-12-15\LJ-12-15 2016-06-17 10-04-00\0DH-5-95-1ML-254NM-20MIN.M
Last changed : 12/16/2015 3:05:20 PM by LHC
Analysis Method : D:\LC\DATA\LJ\LJ-12-15\LJ-12-15 2016-06-17 10-04-00\014-0201.D\DA.M (0DH-5-95-1ML-254NM-20MIN.M)
Last changed : 7/6/2016 4:24:51 PM by LHC
(modified after loading)



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Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	5.204	BB	0.1205	142.33124	18.16833	10.2667
2	7.290	VB	0.1800	1244.01306	106.50106	89.7333

Totals : 1386.34430 124.66940

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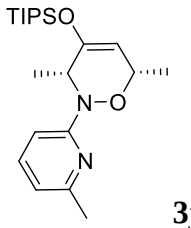
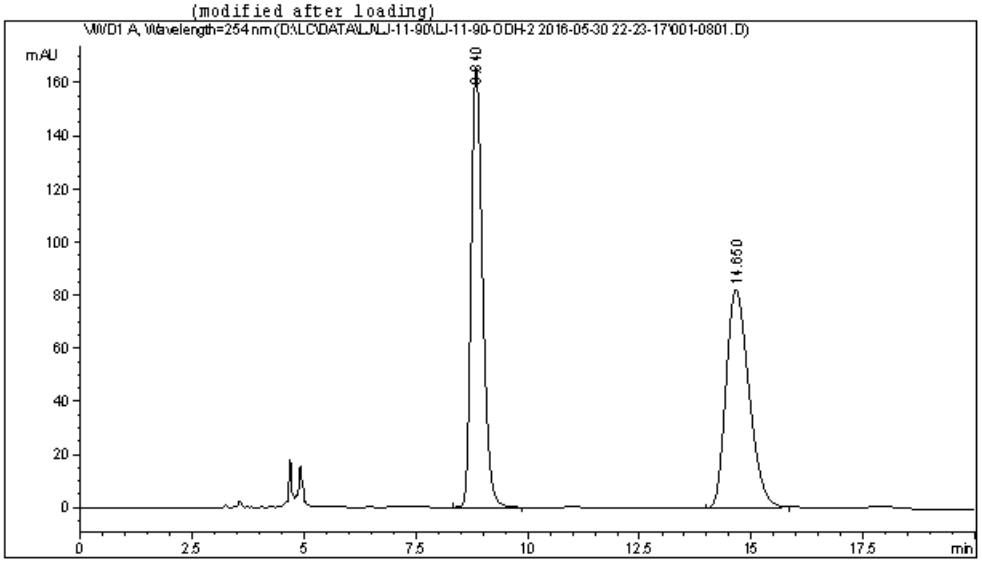
Instrument 1 7/6/2016 4:24:56 PM LHC Page 1 of 1



3j

Data File D:\LC\DATA\LJ\LJ-11-90\LJ-11-90-ODH-2 2016-05-30 22-23-17\001-0801.D
Sample Name: LJ-11-79A

Acq. Operator	: HR
Acq. Instrument	: Instrument 1
Injection Date	: 5/31/2016 2:34:49 AM
Acq. Method	: D:\LC\DATA\LJ\LJ-11-90\LJ-11-90-ODH-2 2016-05-30 22-23-17\ODH-0-100-1ML-254NM-40MIN.M
Last changed	: 5/30/2016 10:18:58 PM by HR
Analysis Method	: D:\LC\DATA\LJ\LJ-11-90\LJ-11-90-ODH-2 2016-05-30 22-23-17\001-0801.D\DA.M (ODH-0-100-1ML-254NM-40MIN.M)
Last changed	: 10/5/2016 6:42:55 PM by LHC (modified after loading)
Seq. Line	: 8
Location	: Vial 1
Inj	: 1
Inj Volume	: 5 µl

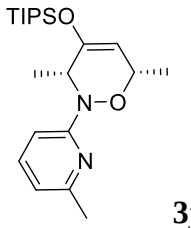


Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.840	BB	0.2755	2958.30957	165.51152	50.2429
2	14.650	BB	0.5571	2929.70850	82.17965	49.7571
Totals :				5888.01807	247.69117	



3j

```
Data File D:\LC\DATA\LJ\LJ-11-90\LJ-11-90-ODH-2 2016-05-30 22-23-17\003-0901.D
Sample Name: LJ-11-76

    ==================================================================
    Acq. Operator   : HR                              Seq. Line :   9
    Acq. Instrument : Instrument 1                     Location : Vial 3
    Injection Date  : 5/31/2016 3:16:23 AM                  Inj :   1
                                                     Inj Volume : 5 µl
    Acq. Method     : D:\LC\DATA\LJ\LJ-11-90\LJ-11-90-ODH-2 2016-05-30 22-23-17\ODH-0-100-1ML-
                      254NM-40MIN.M
    Last changed    : 5/30/2016 10:18:58 PM by HR
    Analysis Method : D:\LC\DATA\LJ\LJ-11-90\LJ-11-90-ODH-2 2016-05-30 22-23-17\003-0901.D\DA.M
                      (ODH-0-100-1ML-254NM-40MIN.M)
    Last changed    : 10/5/2016 6:41:06 PM by LHC
                      (modified after loading)
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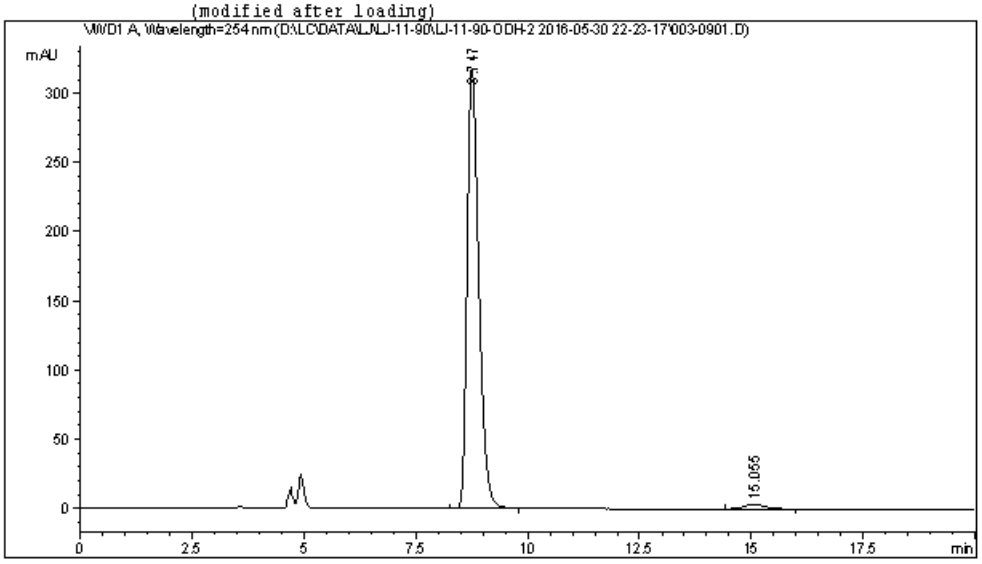


```
    ==================================================================
                            Area Percent Report
    ==================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   8.747 BB    0.2773 5781.29346  318.45264  98.1470
       2  15.055 BB    0.4589  109.15193    3.15551   1.8530

    Totals :                  5890.44539  321.60815


    ==================================================================
```

Instrument 1 10/5/2016 6:41:12 PM LHC Page 1 of 1