Supplementary data

Amino-Polystyrene Supported Hexaethylene Glycol-Bridged Ionic Liquid as an Efficient Heterogeneous Catalyst for Water-Mediated Nucleophilic Hydroxylation

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1. General remarks

Unless otherwise noted, all reagents and solvents were commercially available. Reaction progress was followed by TLC on 0.25 mm silica gel glass plates containing F-254 indicator. Visualization on TLC was monitored by UV light (254 nm). Flash chromatography was performed with 230-400 mesh silica gel. ^{1}H and ^{13}C NMR spectra were recorded on a 400 MHz spectrometer, and chemical shifts were reported in δ units (ppm) relative to tetramethylsilane. FT-IR spectra were collected using a Bruker Vertex 80v FT-IR spectrometer. Low- and high-resolution electron impact (EI, 70 eV) spectra were obtained on high resolution mass spectrometer (Korea Basic Science Institute). X-ray photoelectron spectroscopy (XPS) measurements were performed using an angle-resolved X-ray photoelectron spectrometer (Theta Probe AR-XPS, Thermo Fisher Scientific, U.K.) equipped with an MXR1 Gun 400-μm 15-keV spectrometer. Thermogravimetric analysis (TGA) measurements were performed with a TG 209 F3 (NETZSCH, Germany) at a 5 ^{o}C/min heating rate between 10 and 800 ^{o}C under an argon atmosphere. Elemental analysis was performed using a FLASH EA 1112 elemental analyzer (Thermo Electron Corporation).

2. Procedure of hydroxylation in Table 1.

Procedure of Entry 1 in Table 1. Prepared according to the typical procedure of hydroxylation (entry 2) except no use of APS-HEGBIL for 180 min. This reaction provided only 85 mg of **5a** in 30% yield.

Procedure of Entry 3 in Table 1. Prepared according to the typical procedure of hydroxylation except (entry 2) no use K_2CO_3 in water (3 mL) for 65 min. This reaction provided 205 mg of **5a** in 72% yield.

Procedure of Entry 4 in Table 1. Prepared according to the typical procedure of hydroxylation (entry 2) except using $NaHCO_3$ (252 mg, 3 mmol) for 55 min instead of using K_2CO_3 (415 mg, 3 mmol). This reaction provided 240 mg of **5a** in 84% yield.

Procedure of Entry 7 in Table 1. Prepared according to the typical procedure of hydroxylation except (entry 2) using [hexaEGmim][OMs] (692 mg, 1.0 mmol) for 75 min instead of using APS-HEGBIL (100 mg). This reaction provided 255mg of **5a** in 90% yield.

Procedure of Entry 8 in Table 1. Prepared according to the typical procedure of hydroxylation except (entry 2) using [bmim][OMs] (235 mg, 1.0 mmol) instead of using APS-HEGBIL (100 mg). This reaction provided 192 mg of **5a** in 55% yield after 80 min.

Procedure of Entry 9 in Table 1. Prepared according to the typical procedure of hydroxylation except (entry 2) using HEGBDVIM (594 mg, 1.0 mmol) for 45 min instead of using APS-HEGBIL (100 mg). This reaction provided 243mg of **5a** in 85% yield.

Procedure of Entry 10 in Table 1. Prepared according to the typical procedure of hydroxylation (entry 2) except using TBAOH·30H_2O (2.40 g, 3.0 mmol) instead of using APS-HEGBIL (100 mg) and K_2CO_3 (415 mg, 3.0 mmol). This reaction provided 141 mg of **5a** in 50% yield after 65 min.

Procedure of Entry 10 in Table 1. Prepared according to the typical procedure of hydroxylation (entry 2) except using 18-crown-6 (264 mg, 1.0 mmol) and KOH (169 mg, 3 mmol) instead of using APS-HEGBIL (100 mg) and K_2CO_3 (415 mg, 3 mmol). This reaction provided 156 mg of **5a** in 55% yield after 75 min.

Procedure of Entry 12 in Table 1. Prepared according to the typical procedure of hydroxylation except (entry 2) using PBS (3 mL) for 35 min instead of using water and K_2CO_3. This reaction provided 255mg of **5a** in 90% yield.

Procedure of Entry 13 in Table 1. Prepared according to the typical procedure of hydroxylation (entry 2) except using the mixture solution of 1,4-dioxane (2 mL) and water (2 mL) for 35 min instead of using water (3 mL). This reaction provided 278 mg of **5a** in 98% yield.

3. Procedure of hydroxylation of various substrates and analytical data of the alcohol products in Scheme 2 and Table 2.

2-(3-hydroxypropoxy)-naphthalene (7, Scheme 2). K_2CO_3 (415 mg, 3 mmol) was added to the mixture of 2-(3-bromopropoxy)naphthalene (**6**, 265 mg, 1.0 mmol) and 100 mg (0.12 equiv of the IL portion) of APS-HEGBIL in water (3 mL). The reaction mixture was stirred over 2 h at 110 °C. After completion of the reaction as indicated by TLC, diethyl ether was added to the reaction mixture the insoluble APS-HEGBIL is filtered and washed three times with acetone after that the APS-HEGBIL is dried under vacuum before reusing. The filtrate was evaporated under reduced pressure. Flash column chromatography (10% EtOAc/hexanes) of the filtrate afforded 198 mg (0.98 mmol, 98%) of 2-(3-hydroxypropoxy)naphthalene (**7**) as a white solid; mp: 103-105 °C; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.79-7.67 (m, 3H), 7.47-7.38 (m, 1H), 7.36-7.29 (m, 1H), 7.18-7.10 (m, 2H), 4.22 (t, J = 6.2 Hz, 2H), 3.89 (t, J = 5.7 Hz, 2H), 2.13-2.05 (m, 2H), 1.93 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.6, 134.4, 129.3, 128.9, 127.5, 126.7, 126.3, 123.6, 118.7, 106.6, 65.6, 60.4, 31.9; *known compound.* Registry No. provided by the author: 7598-29-0.

Table S1. Reusability of the APS-HEGBIL catalyst.

recycle run	1	2	3	4	5	6	7	8	9	10
yield (%)	98	98	98	96	96	95	95	93	92	90

1,3-Propanediol (8, Table 2). Prepared according to the typical procedure (entry 2 in Table 1) of hydroxylation except using 3- bromo-1-propanol (139 mg, 1.0 mmol) for 30 min instead of using hexaEG bromide (**4**). This reaction provided 73 mg (0.96 mmol) of 1,3-propanediol (**8**) in 96% yield as a colorless oil; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 3.86-3.81 (m, 4H), 2.21 (s, 2H), 1.84-1.76 (m, 2H), ^{13}C NMR (100 MHz, $CDCl_3$) δ 62.1, 34.0; *known compound.* Registry No. provided by the author: 504-63-2.

1-(3-Hydroxypropyl)-4-nitroimidazole (9, Table 2). Prepared according to the typical procedure of hydroxylation except using 1-(3-bromopropyl)-4-nitroimidazole (233 mg, 1.0 mmol) for 40 min instead of using **4**. This reaction provided 166 mg (0.97 mmol) of 1-(3-hydroxypropyl)-4-nitroimidazole (**9**) in 97% yield as a white solid; mp: 85-87 °C; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.79 (d, *J* = 1.8 Hz, 1H), 7.45 (d, *J* = 1.8 Hz, 1H), 4.20 (t, *J* = 6.9 Hz, 2H), 3.67 (t, *J* = 4.8 Hz, 2H), 2.08-1.98 (m, 2H), 1.85 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.2, 119.4, 58.0, 44.7, 32.6; *known compound.* Registry No. provided by the author: 13230-17-6.

2-(2-Hydroxypropoxy)naphthalene (10, Table 2). Prepared according to the typical procedure of hydroxylation except using 2-(2-bromopropoxy)naphthalene (264 mg, 1.0 mmol) at 110 ^{o}C for 200 min instead of using **4**. This reaction provided 191 mg (0.94 mmol) of 2-(2-hydroxypropoxy)naphthalene (**10**) in 94% yield as a white solid; mp: 82-85 °C; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.79-7.70 (m, 3H), 7.44 (td, *J* = 7.4, 1.2 Hz, 1H), 7.39-7.32 (m, 1H), 7.15 (td, *J* = 8.7, 2.4 Hz, 2H), 4.30-4.20 (m, 1H), 4.04 (dd, *J* = 9.6, 3.2 Hz, 1H), 3.91 (dd, *J* = 9.1, 7.8 Hz, 1H), 2.56 (s, 1H), 1.32 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.4, 134.4, 129.4, 129.1, 127.6, 126.7, 126.4, 123.7, 118.6, 106.8, 73.2, 66.2, 18.8; *known compound.* Registry No. provided by the author: 108298-91-5.

1-(2-Hydroxyethyl)naphthalene (11, Table 2). Prepared according to the typical procedure of hydroxylation except using 1-(2-bromoethyl)naphthalene (234 mg, 1.0 mmol) at 110 ^{o}C for 250 min instead of using **4**. This reaction provided 166 mg (0.96 mmol) of 1-(2-hydroxyethyl)-naphthalene (**11**) in 96% yield as a white solid; mp: 63-65 °C; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.06 (t, *J* = 8.5 Hz, 1H), 7.89-7.83 (m, 1H), 7.75 (d, *J* = 8.2 Hz, 1H), 7.56-7.45 (m, 2H), 7.44-7.33 (m, 2H), 3.98 (t, *J* = 6.6 Hz, 2H), 3.34 (t, *J* = 6.9 Hz, 2H), 1.50 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 134.3, 133.8, 131.9, 128.7, 127.2, 127.0, 125.9, 125.5, 125.4, 123.5, 62.8, 36.1; *known compound.* Registry No. provided by the author: 773-99-9.

3-(3-Hydroxypropoxy)-2*H*-chromen-2-one (12, Table 2). Prepared according to the typical procedure of hydroxylation except using 3-(3-bromopropoxy)-2*H*-chromen-2-one (285 mg, 1.0 mmol) for 95 min instead of using **4**. This reaction provided 213 mg (0.96 mmol) of 3-(3-hydroxypropoxy)-2*H*-chromen-2-one (**12**) in 96% yield as a white solid; mp: 63-65 °C; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.77 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.54-7.47 (m, 1H), 7.29-7.20 (m, 2H), 5.68 (s, 1H), 4.27 (t, *J* = 5.9 Hz, 2H), 3.89 (t, *J* = 5.9 Hz, 2H), 2.13 (t, *J* = 2.7 Hz, 2H), 1.85 (s, 1H): ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.5, 163.0, 153.2, 132.3, 123.3, 122.8, 116.7, 115.6, 90.5, 66.1, 58.9, 31.3; MS (EI) *m/z* 220 (M^+); HRMS (EI TOF) *m/z* calcd for $C_{12}H_{12}O_4$ (M^+) 220.0736, found 220.0735.

2-(Hydroxymethyl)naphthalene (13, Table 2). Prepared according to the typical procedure of hydroxylation except using 2-(bromomethyl)naphthalene (220 mg, 1.0 mmol) for 35 min at 100 °C instead of using **4**. This reaction provided 155 mg (0.98 mmol) of 2-(hydroxymethyl)naphthalene (**13**) in 98% yield as a white solid; mp: 80-82 °C; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.86-7.75 (m, 3H), 7.73 (s, 1H), 7.48 (td, J = 6.6, 3.5 Hz, 2H), 7.42 (dd, J = 8.5, 1.6 Hz, 1H), 4.76 (s, 2H), 2.73 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.2, 133.2, 132.7, 128.1, 127.7, 127.5, 126.0, 125.7, 125.2, 125.0, 65.0; *known compound.* Registry No. provided by the author: 1592-38-7.

α-Hydroxy-3-picoline-*N*-oxide (14, Table 2). Prepared according to the typical procedure of hydroxylation except using 3-chloro-picoline N-oxide (143 mg, 1.0 mmol) for 60 min at 110 °C instead of using **4**. This reaction provided 120 mg (0.96 mmol) of α-hydroxy-3-picoline-*N*-oxide (**14**) in 96% yield as a white solid; mp: 87−88 °C; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.14 (s, 1H), 7.97 (d, *J* = 6.4 Hz, 1H), 7.29-7.23 (m, 1H), 7.17 (t, *J* = 7.1 Hz, 1H), 5.68 (s,1H), 4.59 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.2, 137.4, 137.3, 126.0, 125.6, 60.9; MS (EI) *m/z* 216 (M^+ + H); HRMS (FAB TOF) *m/z* calcd for $C_6H_8NO_2$ (M^+ + H) 126.0555, found 126.0554.

1,2:3,4-Di-*O*-isopropylidene-6-hydroxy-6-deoxy-α-D-galactopyranose (15, Table 2). Prepared according to the typical procedure of hydroxylation except using 1,2:3,4-Di-*O*-isopropylidene-6-bromo-6-deoxy-α-D-galactopyranose (322 mg, 1.0 mmol) for 180 min instead of using **4**. This reaction provided 249 mg (0.95 mmol) of 1,2:3,4-Di-*O*-isopropylidene-6-hydroxy-6-deoxy-α-D-galactopyranose (**15**) in 95% yield as a colorless oil; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 5.54 (d, *J* = 5.0 Hz, 1H), 4.59 (dd, *J* = 7.8, 2.3 Hz, 1H), 4.31 (q, *J* = 2.4 Hz, 1H), 4.28-4.22 (m, 1H), 3.88-3.79 (m, 2H), 3.76-3.65 (m, 1H), 2.21 (s, 1H), 1.51 (s, 3H), 1.43 (s, 3H), 1.31 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 109.4, 108.6, 96.2, 71.5, 70.7, 70.5, 68.0, 62.3, 26.0, 25.8, 24.9, 24.2; MS (EI) *m/z* 261 (M^+); HRMS (EI TOF) *m/z* calcd for $C_{12}H_{20}O_6$ (M^+) 261.1338, found 261.1340.

N-Fmoc-2-hydroxymethylaniline (16, Table 2). A mixture of N-Fmoc-2-chloromethylaniline (363 mg, 1.0 mmol) was taken in 100 mg of APS-HEGBIL in 3 mL of water and stirred at 110 °C for 120 min. After completion of the reaction, the product was extracted with 20 mL of diethyl ether, dried on sodium sulfate, and concentrated under reduced pressure on rotary evaporator. Flash column chromatography (15% EtOAc/hexanes) of the residue afforded 319 mg (0.92mmol, 92%) of *N*-Fmoc-2-hydroxymethylaniline (**16**) as a white solid; mp: 158-160 °C; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.93 (d, *J* = 48.9 Hz, 1H), 7.76 (d, *J* = 7.3 Hz, 2H), 7.61 (d, *J* = 7.8 Hz, 2H), 7.40 (t, *J* = 7.3 Hz, 2H), 7.31 (td, *J* = 7.4, 1.1 Hz, 3H), 7.16 (dd, *J* = 7.8, 1.4 Hz, 1H), 7.04 (td, *J* = 7.4, 1.1 Hz, 1H), 4.70 (d, *J* = 5.5 Hz, 2H), 4.47 (d, *J* = 6.9 Hz, 2H), 4.28 (t, *J* = 7.1 Hz, 1H), 2.18 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.9, 143.7, 141.3, 137.4, 137.4, 129.2, 128.8, 127.7, 127.0, 125.0, 123.6, 120.0, 67.1, 64.3, 47.1; MS (EI) *m/z* 345 (M^+); HRMS (EI TOF) *m/z* calcd for $C_{22}H_{19}NO_3$ (M^+) 345.1365, found 345.1362.

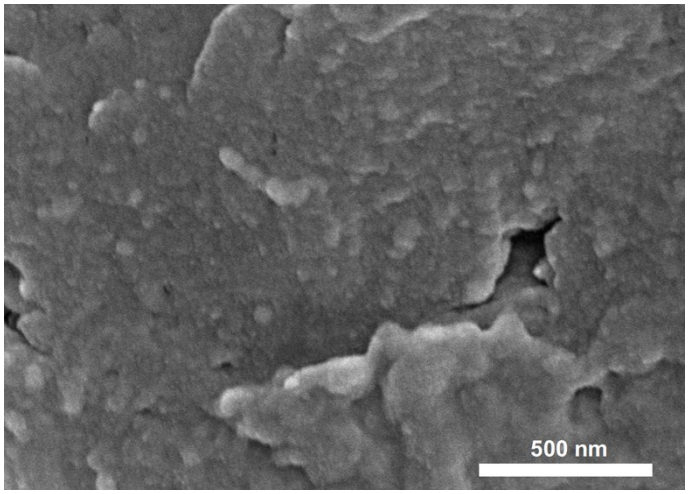
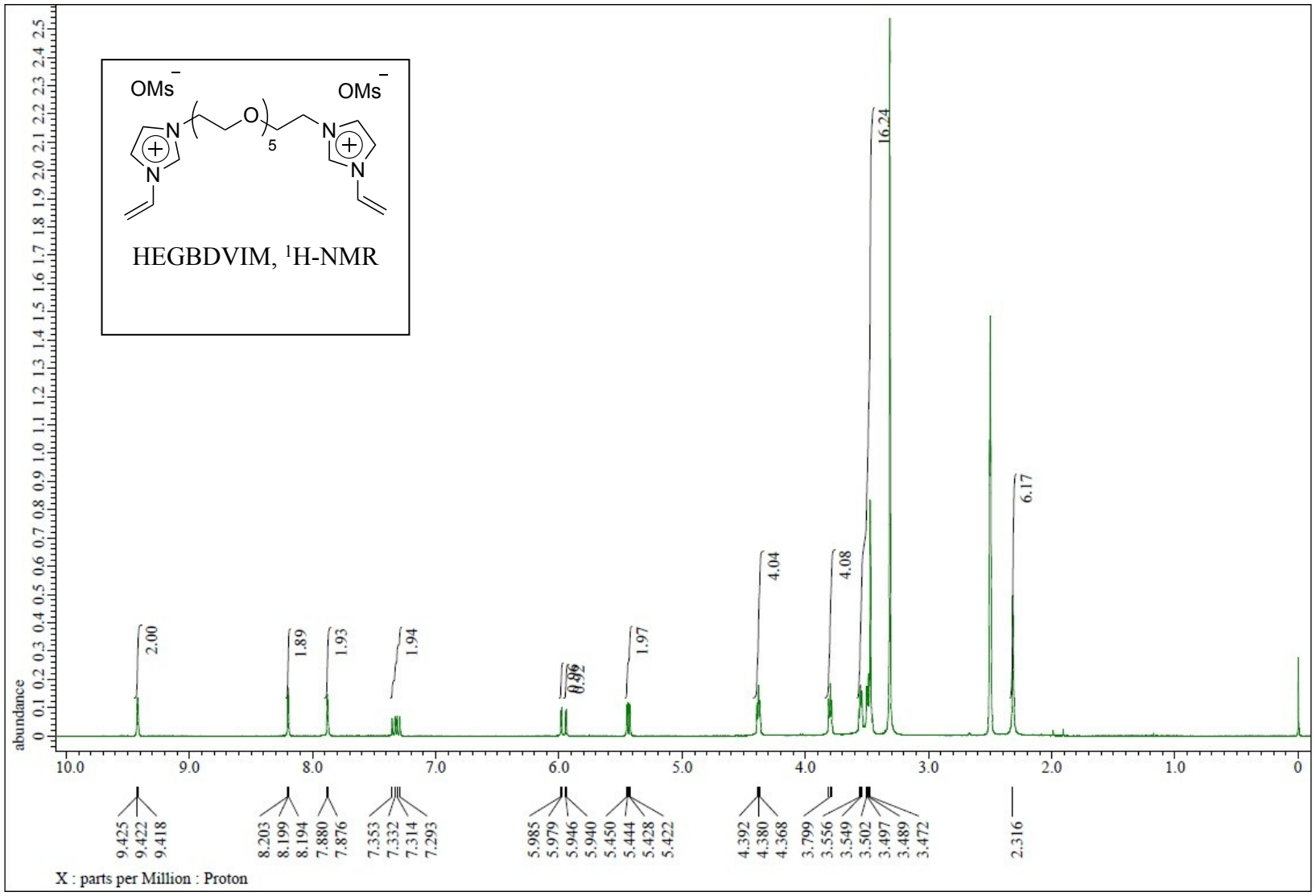
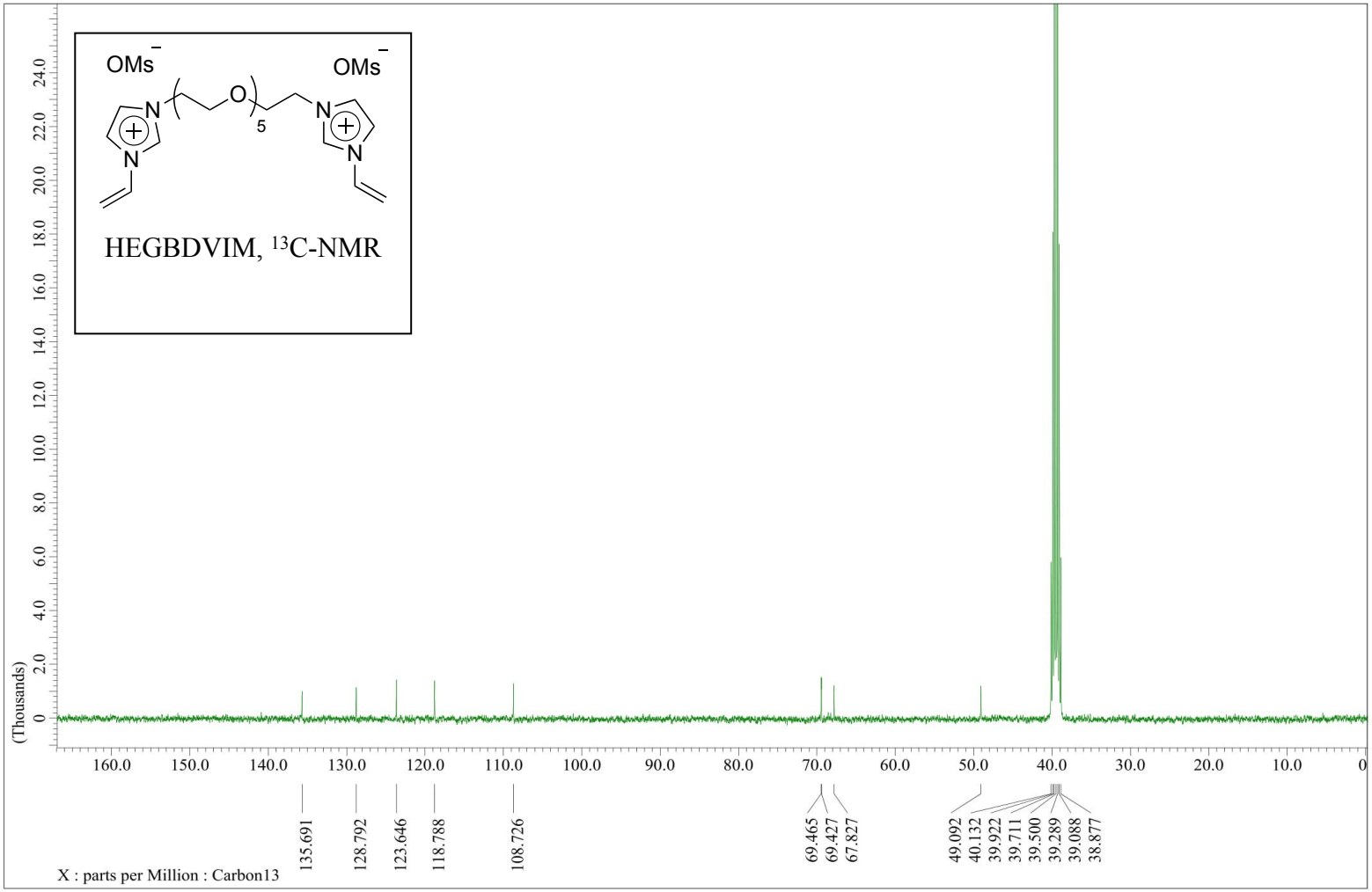


Fig. S1. SEM image of APS-HEGBIL.

4. ^{1}H and ^{13}C NMR spectra of HEGBDVIM.




OMs⁻
OMs⁻
N
O
5
N
N
N
HEGBDVIM, ^{13}C-NMR
(Thousands)
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22.0
20.0
18.0
16.0
14.0
12.0
10.0
8.0
6.0
4.0
2.0
0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
135.691
128.792
123.646
118.788
108.726
69.465
69.427
67.827
49.092
40.132
39.922
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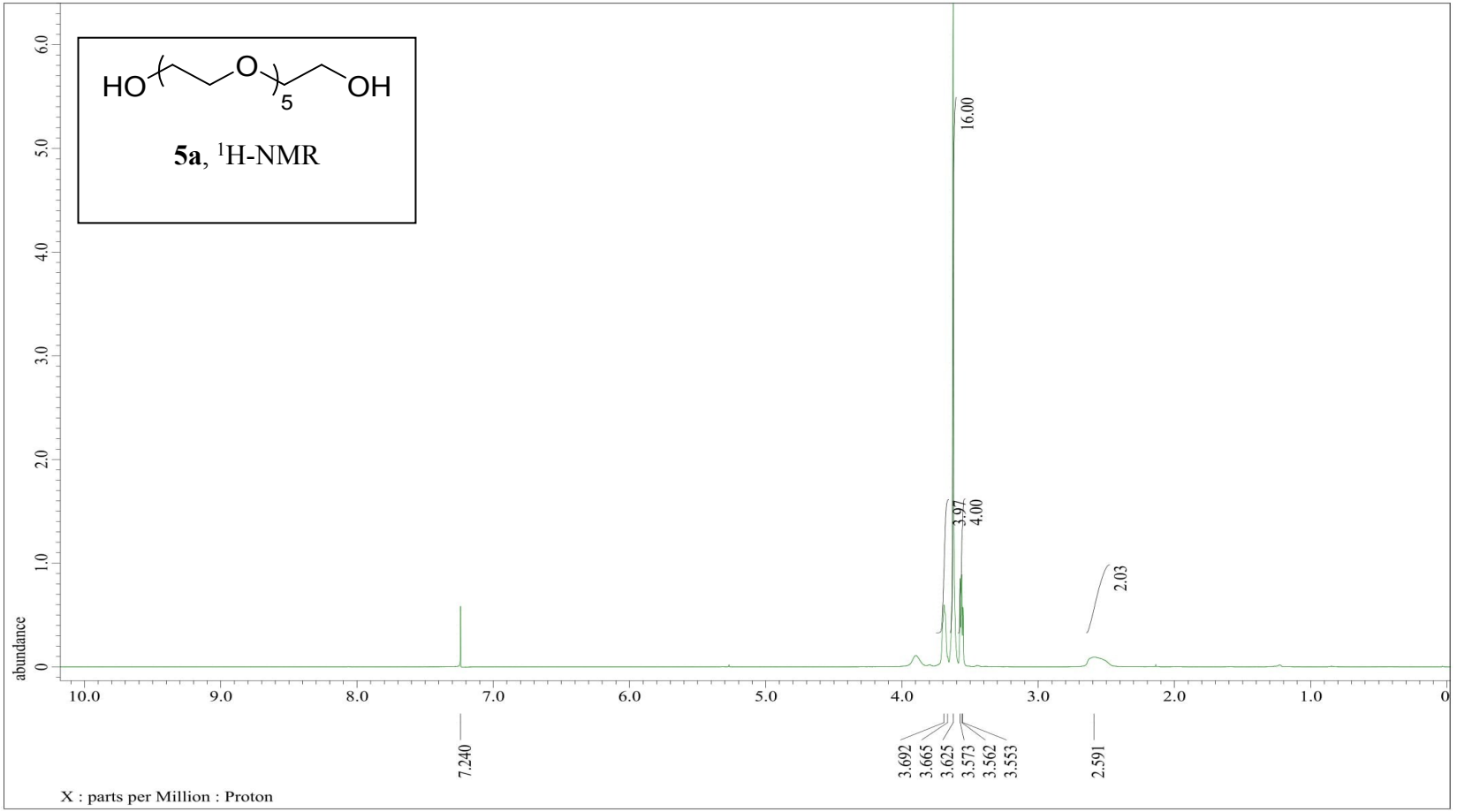
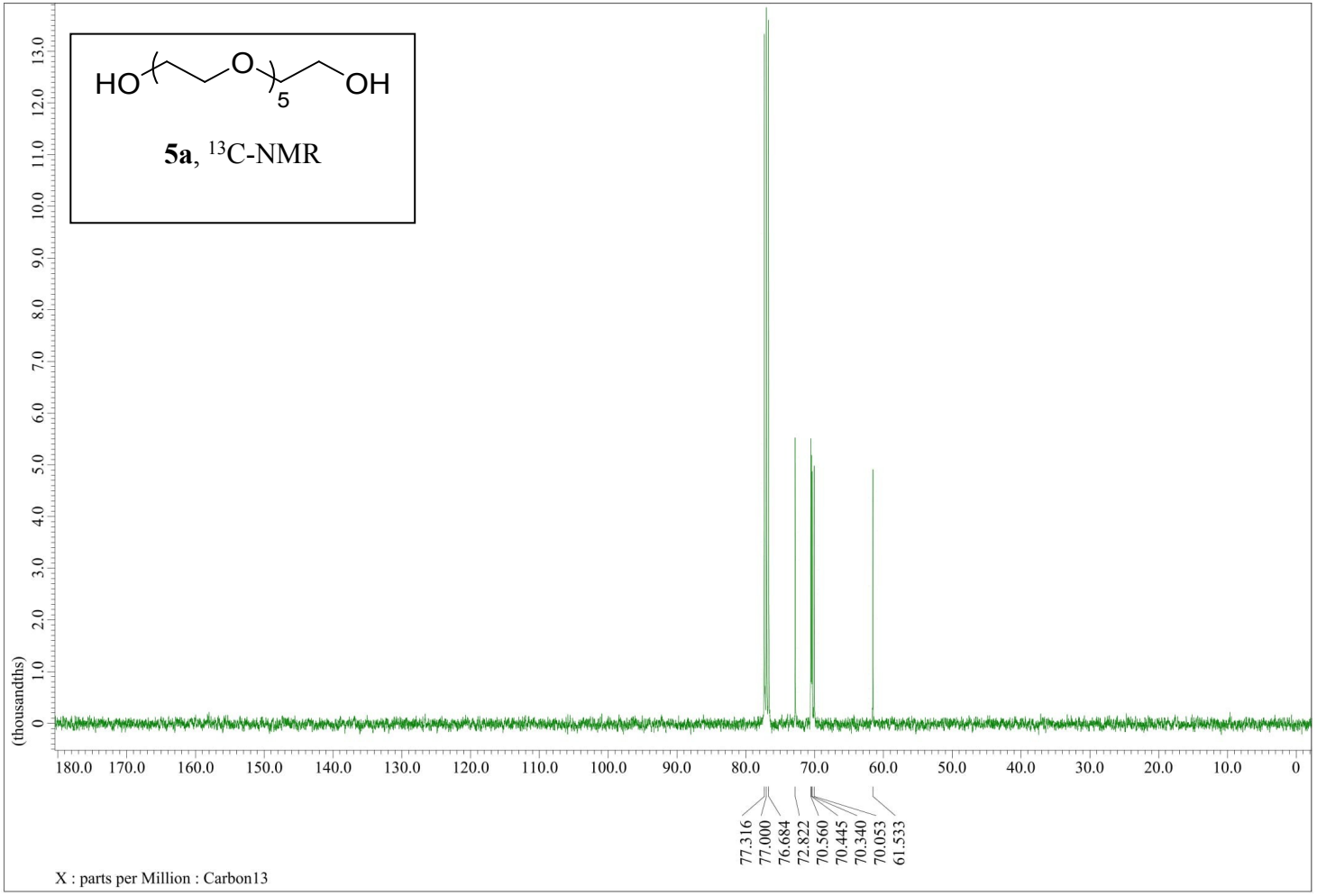
39.088
38.877
X : parts per Million : Carbon13

5. ^{1}H and ^{13}C NMR spectra of hexaethylene glycol (5a).




HO
O
5
OH
5a, 13C-NMR
(thousandths)
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1.0
2.0
3.0
4.0
5.0
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8.0
9.0
10.0
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120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
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77.000
76.684
72.822
70.560
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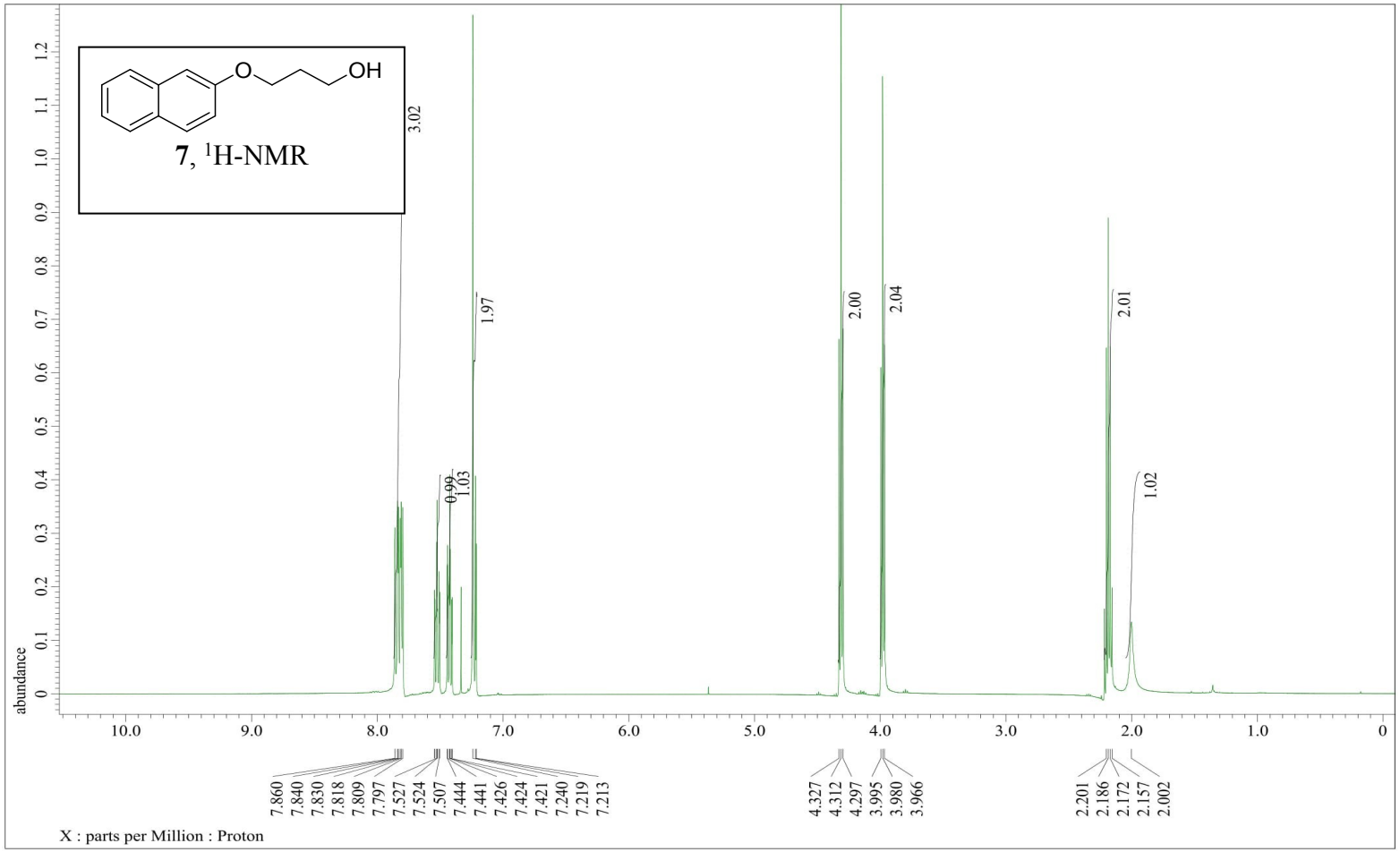
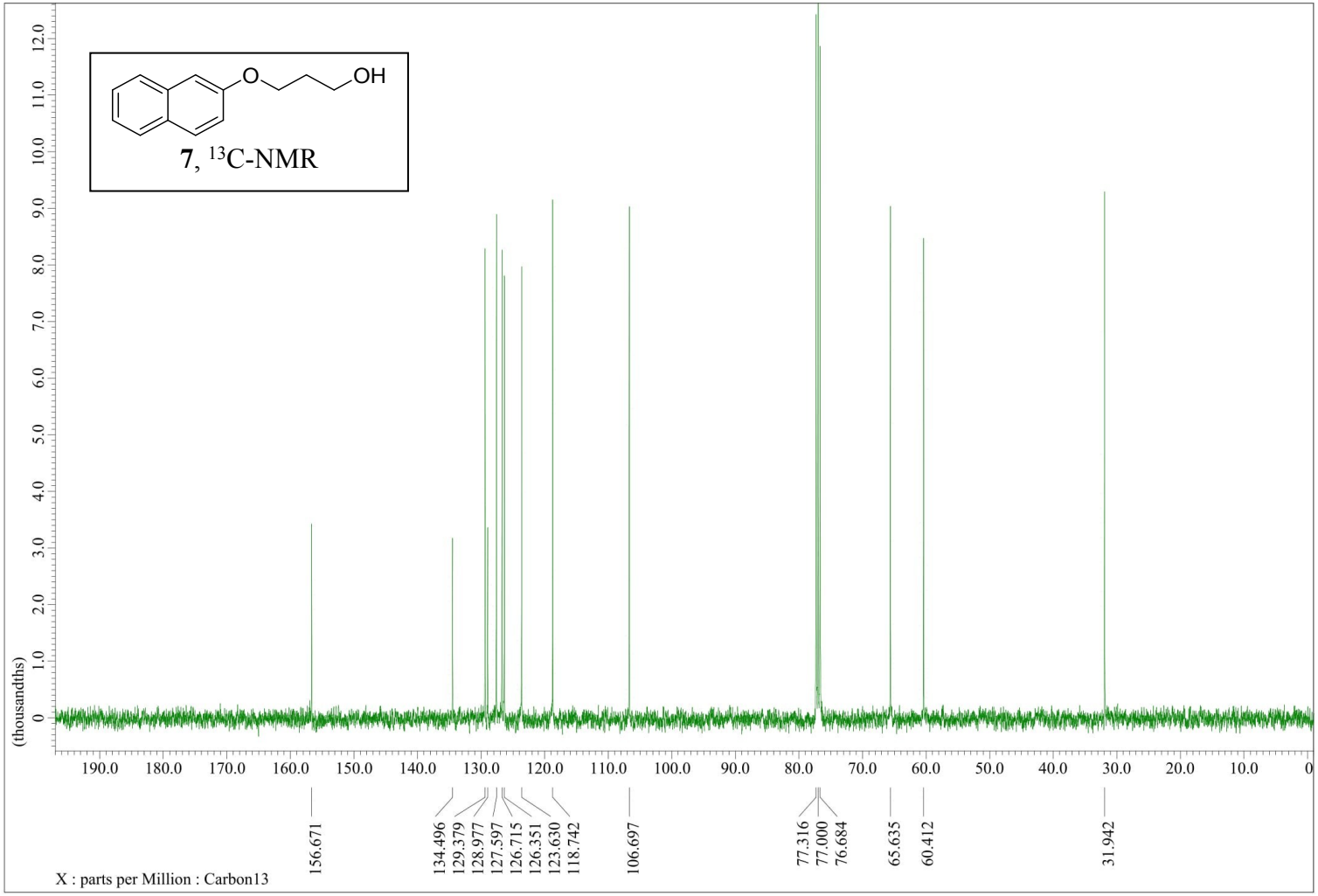
61.533
X : parts per Million : Carbon13

6. ^{1}H and ^{13}C NMR spectra of products (7-16) in Scheme 2 and Table 2.




O
OH
7, 13C-NMR
(thousandths)
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11.0
10.0
9.0
8.0
7.0
6.0
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120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
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129.379
128.977
127.597
126.715
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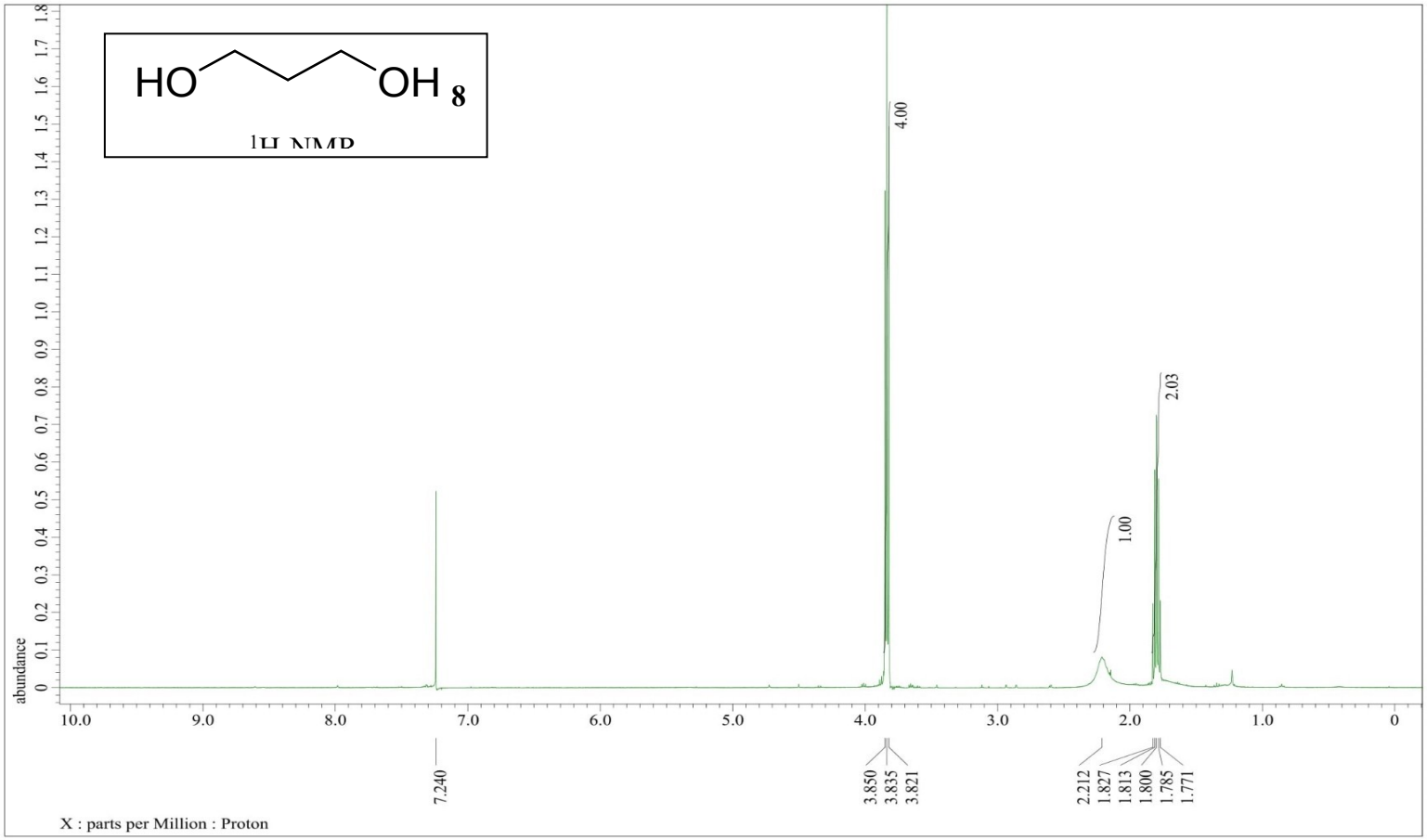
60.412
31.942
X : parts per Million : Carbon13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OH
8
1H NMR
4.00
2.03
1.00
abundance
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.240
3.850
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3.821
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1.827
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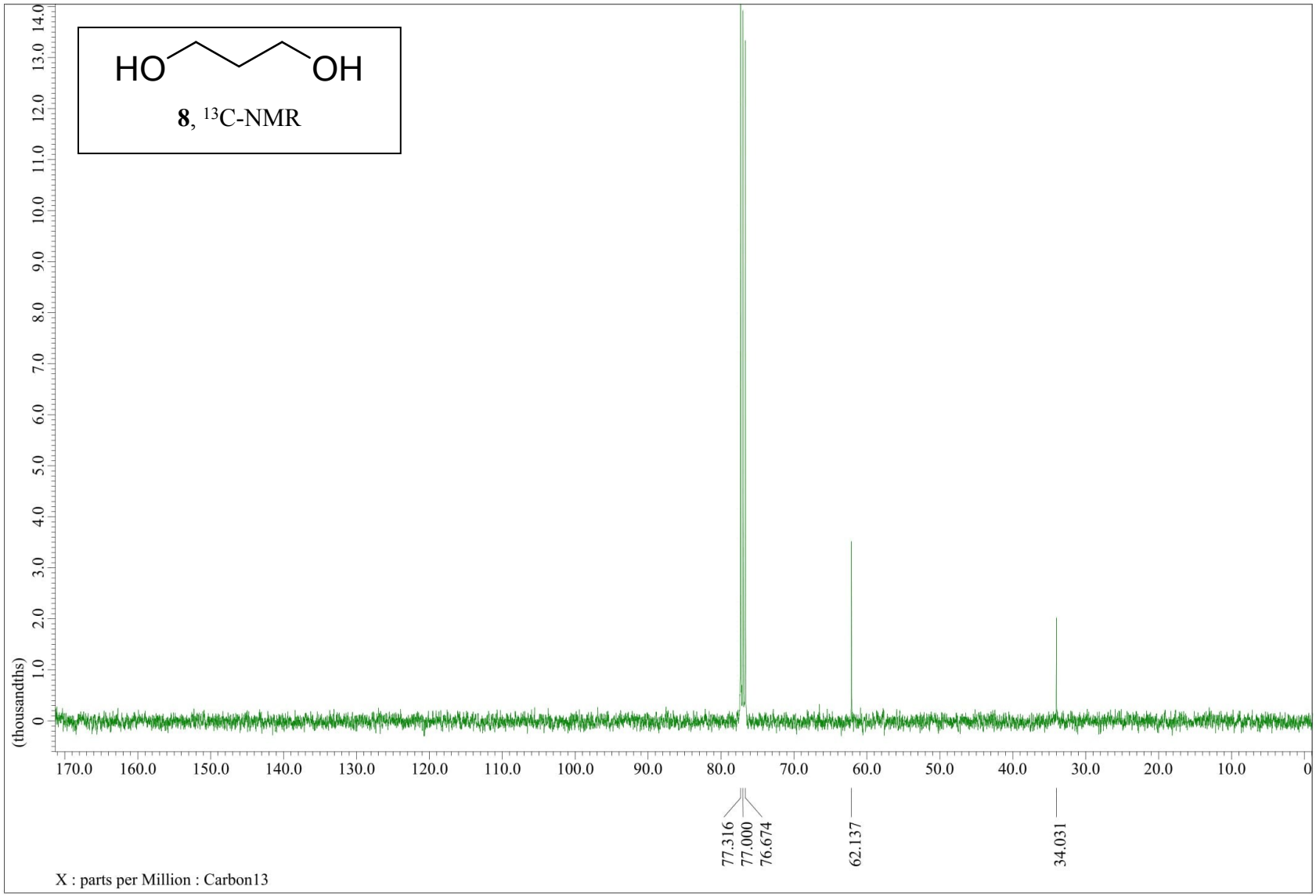
1.800
1.785
1.771
X : parts per Million : Proton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OH
8, 13C-NMR
(thousandths)
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13.0
12.0
11.0
10.0
9.0
8.0
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2.0
1.0
0
170.0
160.0
150.0
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130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
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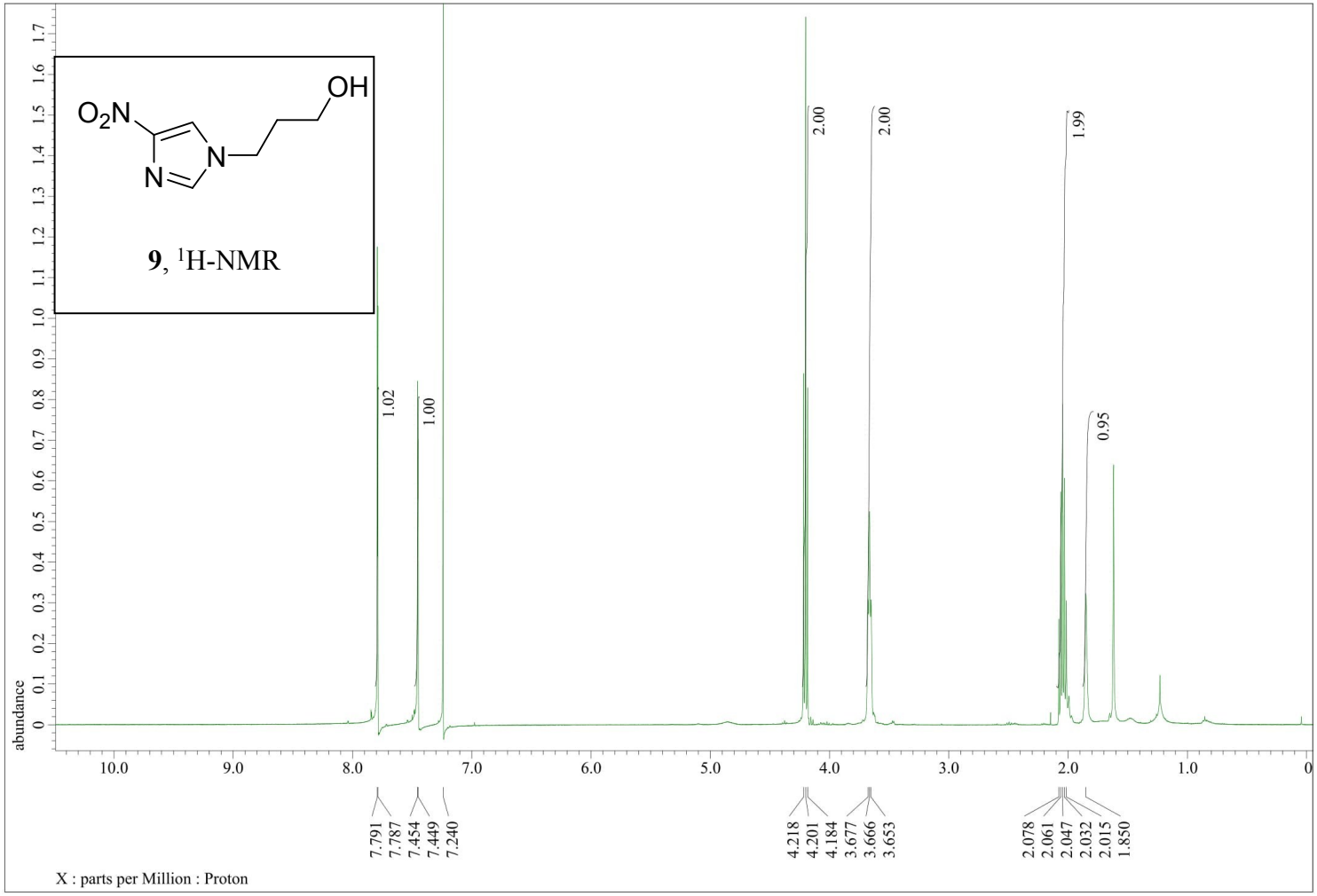
X : parts per Million : Carbon13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OH
N
N
9, 1H-NMR
abundance
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2.00
1.99
0.95
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.791
7.787
7.454
7.449
7.240
4.218
4.201
4.184
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3.653
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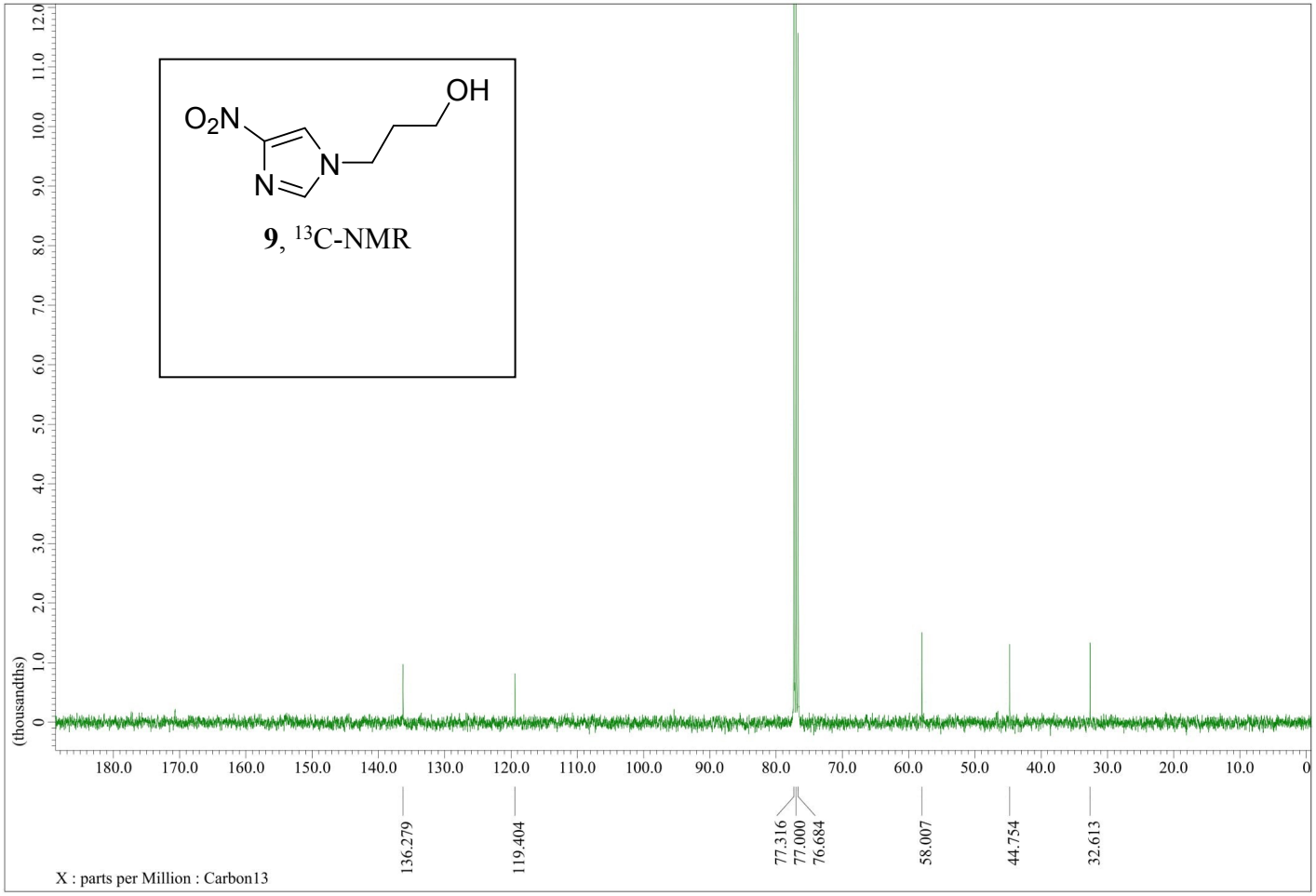
2.047
2.032
2.015
1.850
X : parts per Million : Proton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OH
N
N
9, 13C-NMR
(thousandths)
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11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
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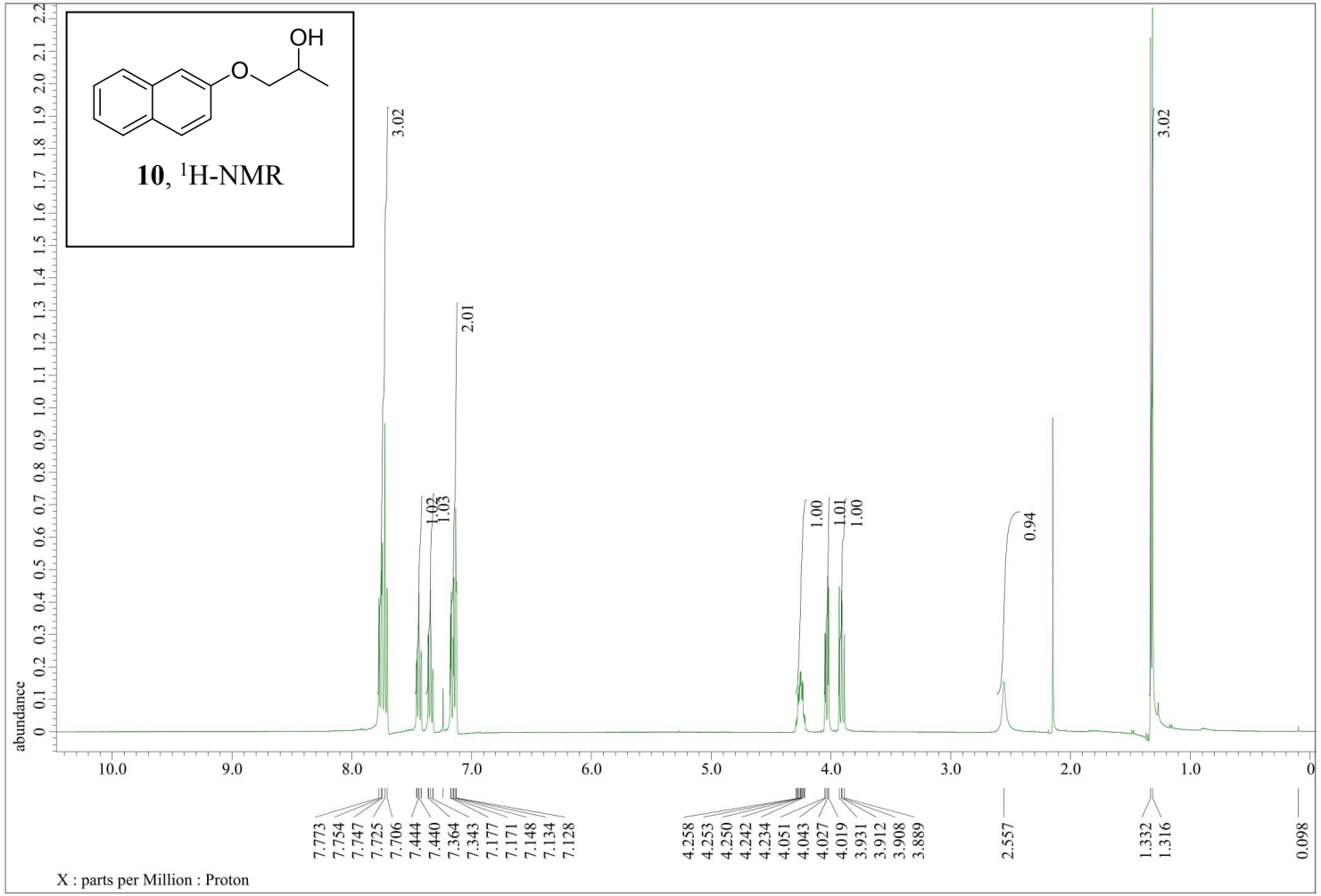
77.000
76.684
58.007
44.754
32.613
X : parts per Million : Carbon13


OH
O
10, 1H-NMR
abundance
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2.01
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1.03
1.00
1.01
1.00
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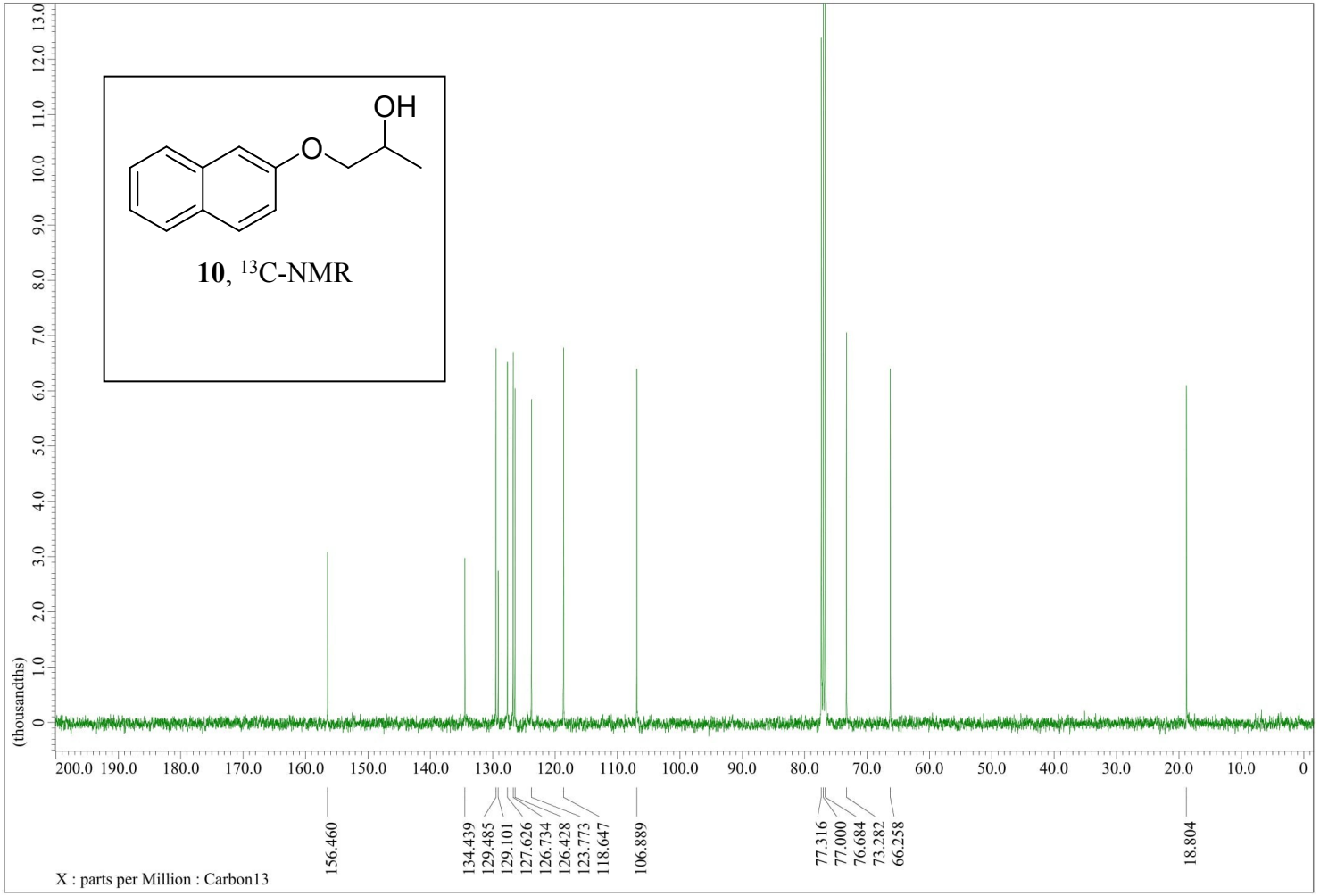
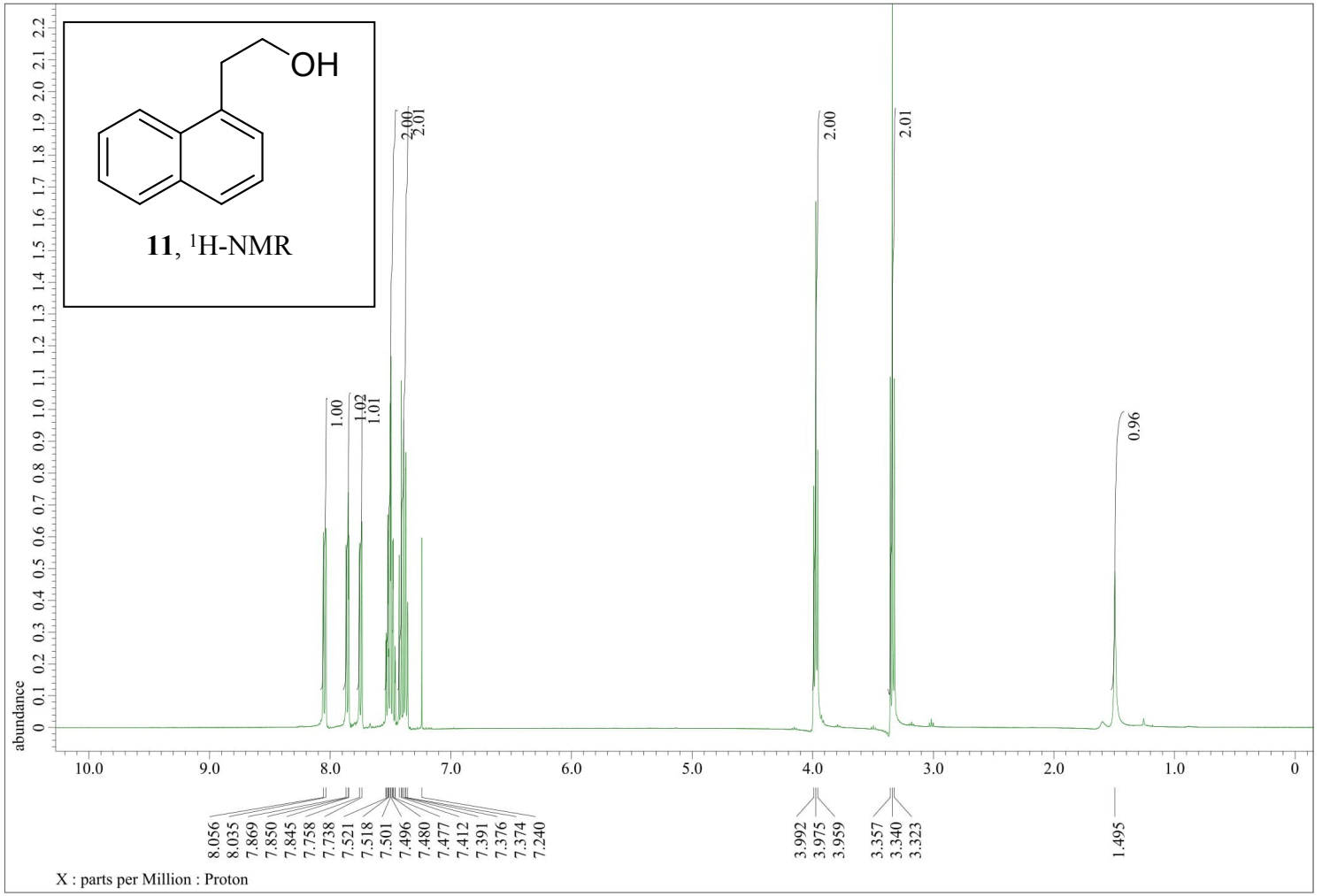
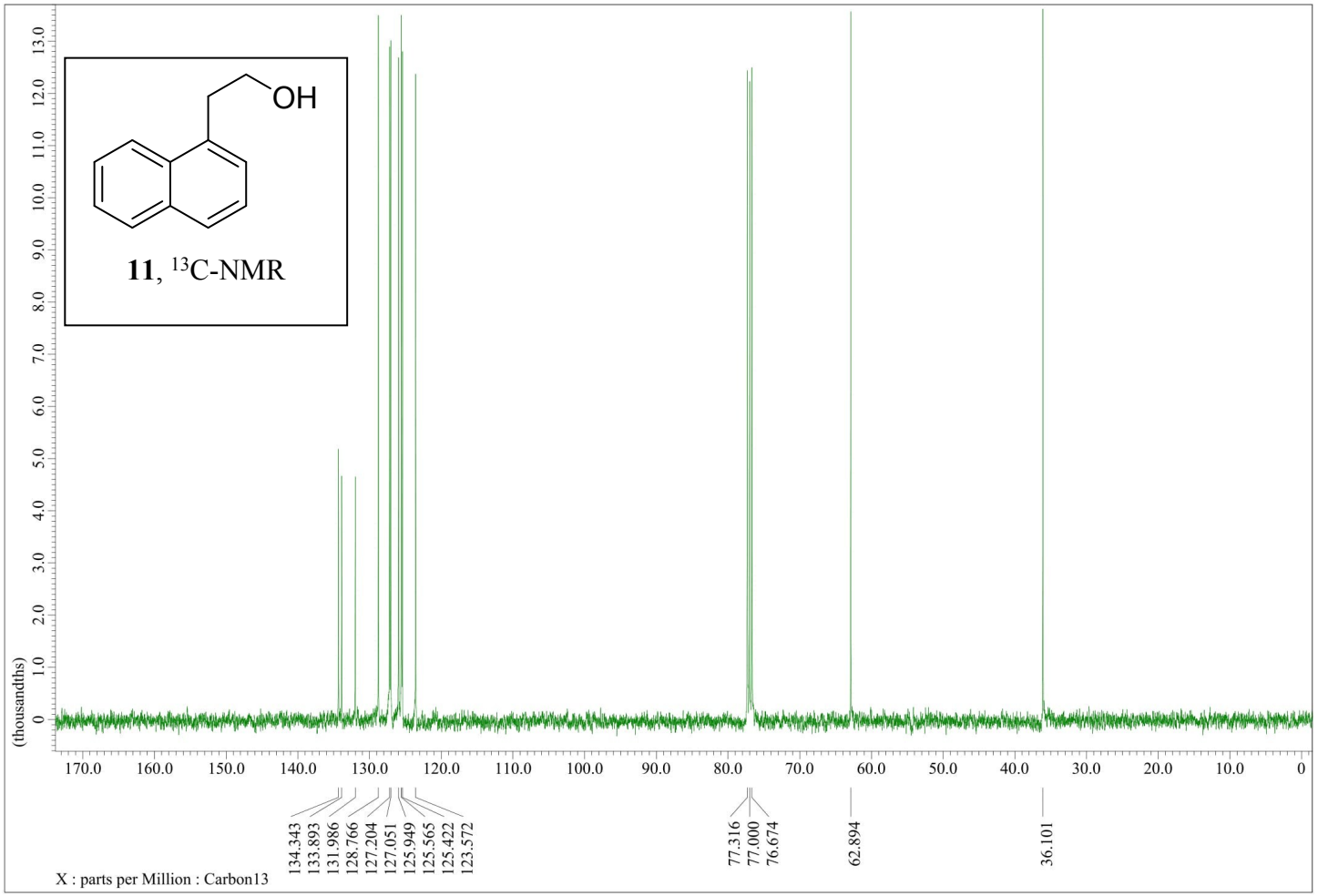
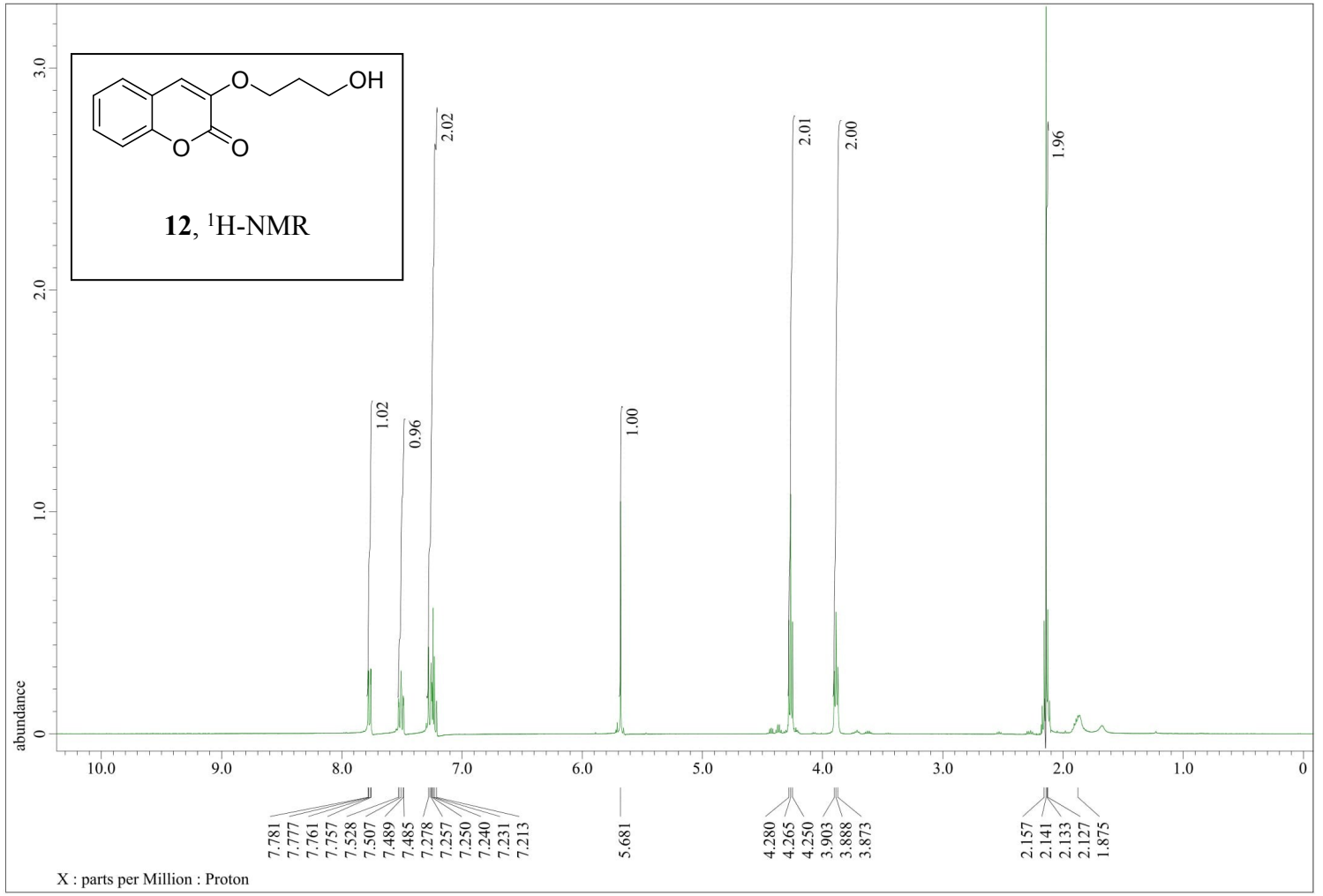
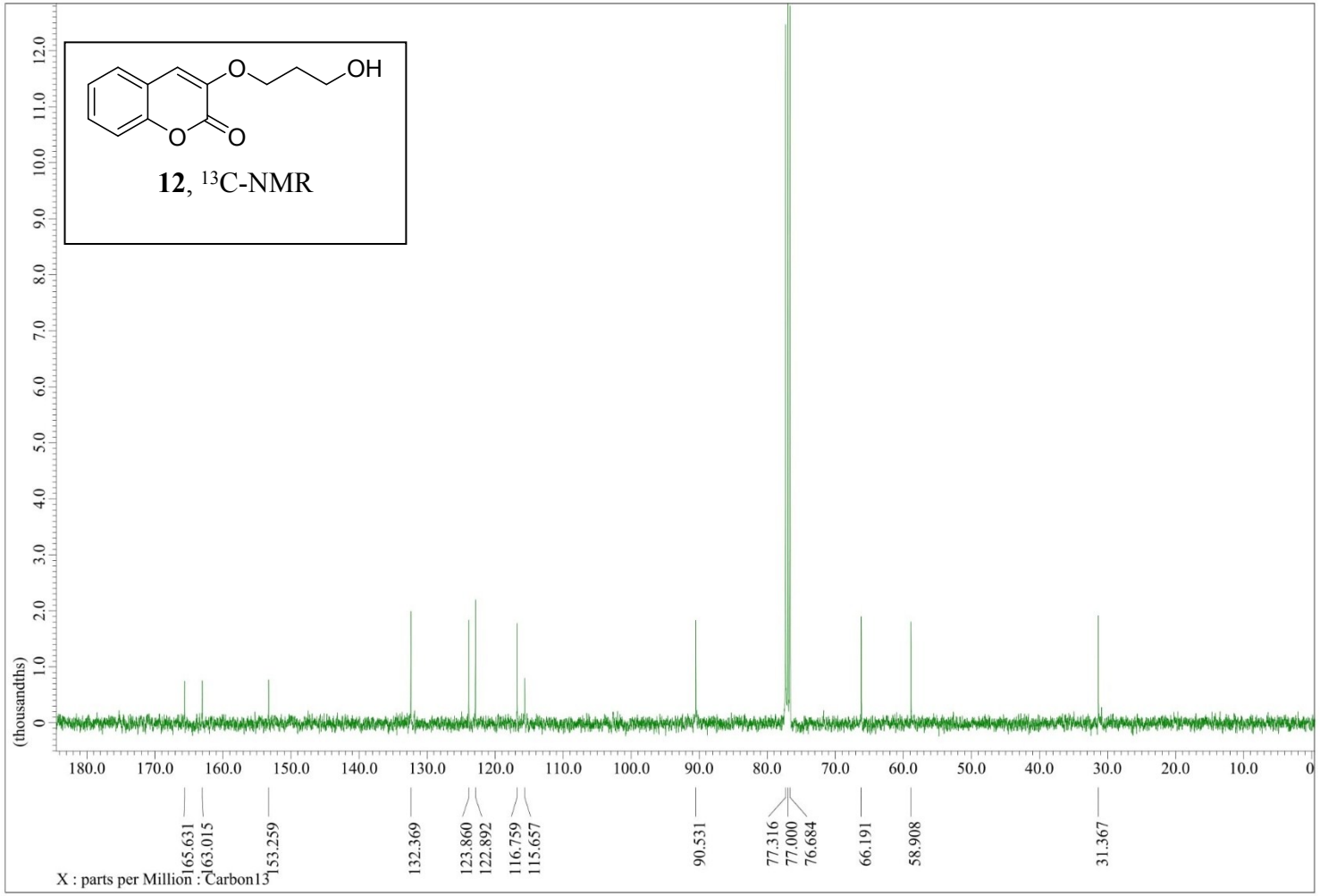
3.02
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.773
7.754
7.747
7.725
7.706
7.444
7.440
7.364
7.343
7.177
7.171
7.148
7.134
7.128
4.258
4.253
4.250
4.242
4.234
4.051
4.043
4.027
4.019
3.931
3.912
3.908
3.889
2.557
1.332
1.316
0.098
X : parts per Million : Proton

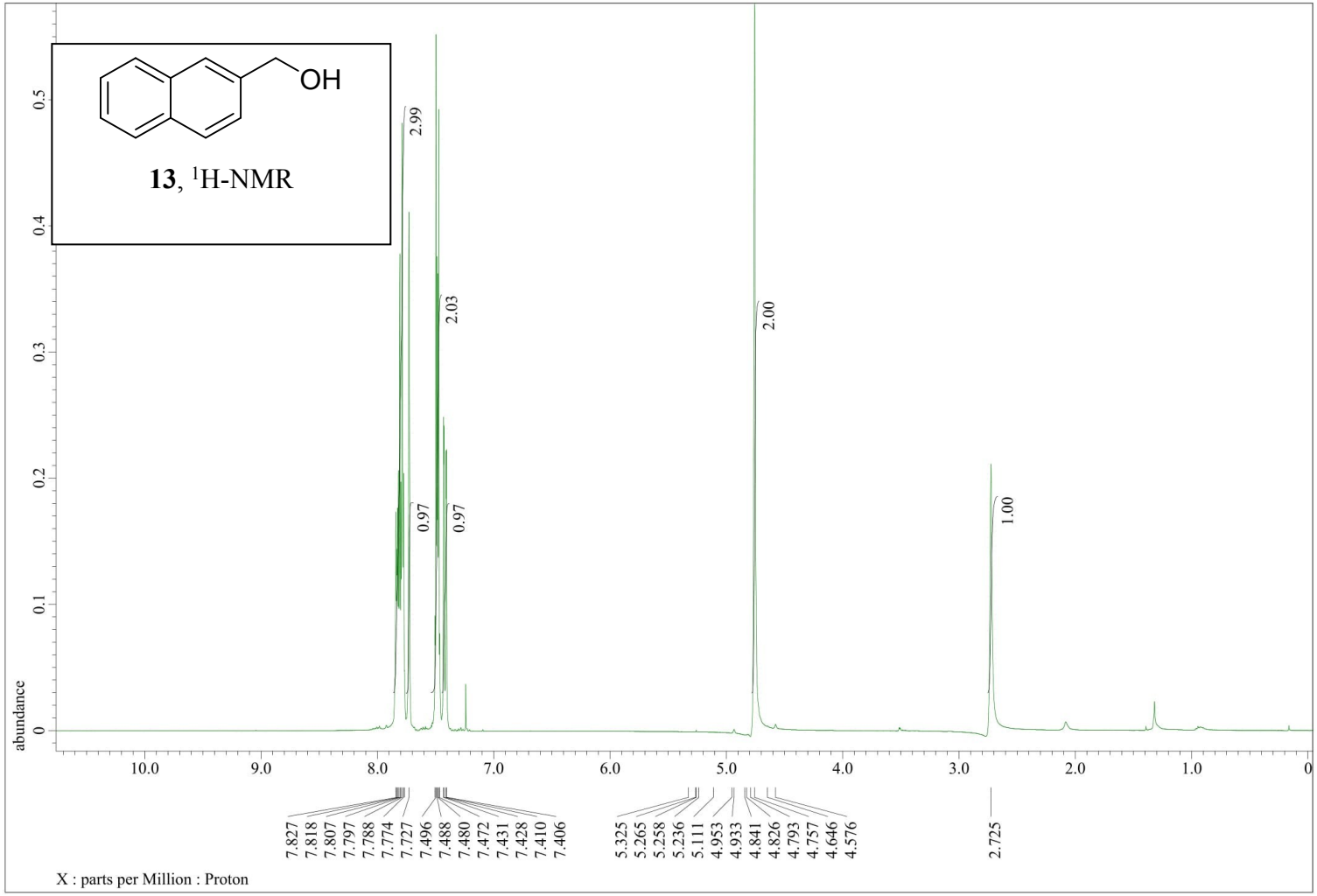

OH
O
10, ^{13}C-NMR
(thousandths)
0 1.0 2.0 3.0 4.0 5.0 6.0 7.0 8.0 9.0 10.0 11.0 12.0 13.0
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
156.460
134.439
129.485
129.101
127.626
126.734
126.428
123.773
118.647
106.889
77.316
77.000
76.684
73.282
66.258
18.804
X : parts per Million : Carbon13

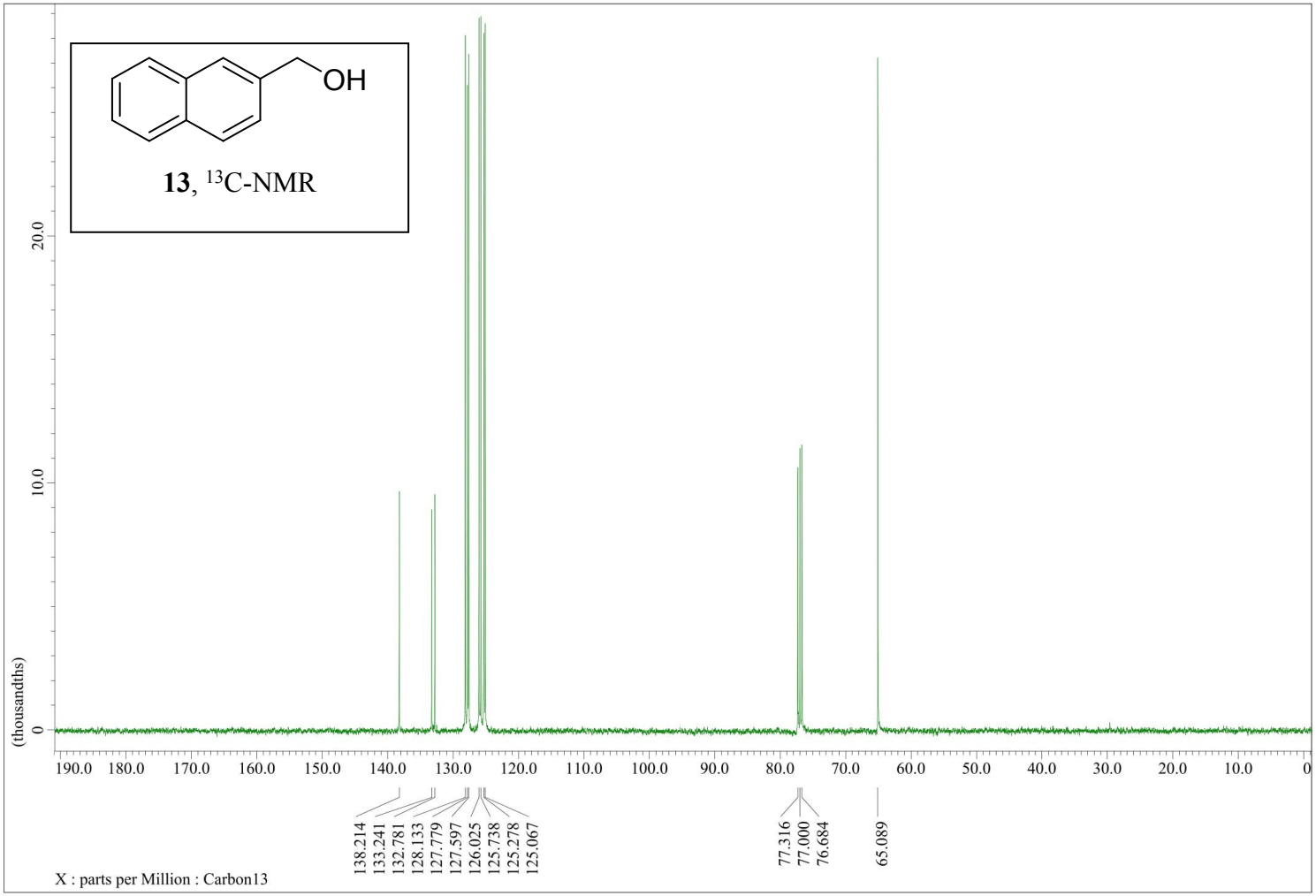

OH
11, 1H-NMR
abundance
X : parts per Million : Proton
1.00
1.02
1.01
2.00
2.01
2.00
2.01
0.96
8.056
8.035
7.869
7.850
7.845
7.758
7.738
7.521
7.518
7.501
7.496
7.480
7.477
7.412
7.391
7.376
7.374
7.240
3.992
3.975
3.959
3.357
3.340
3.323
1.495

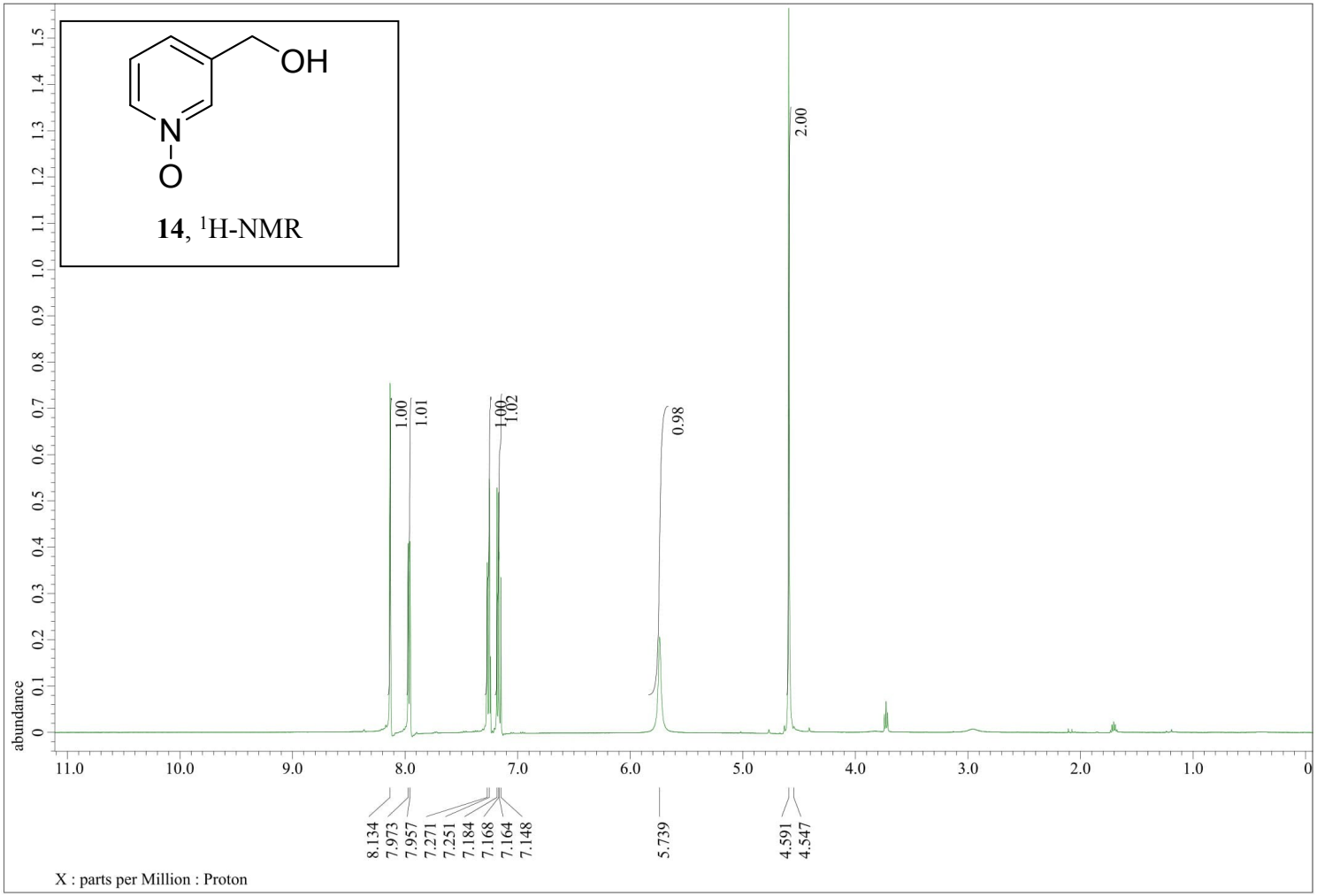

OH
11, 13C-NMR
(thousandths)
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
134.343
133.893
131.986
128.766
127.204
127.051
125.949
125.565
125.422
123.572
77.316
77.000
76.674
62.894
36.101
X : parts per Million : Carbon13

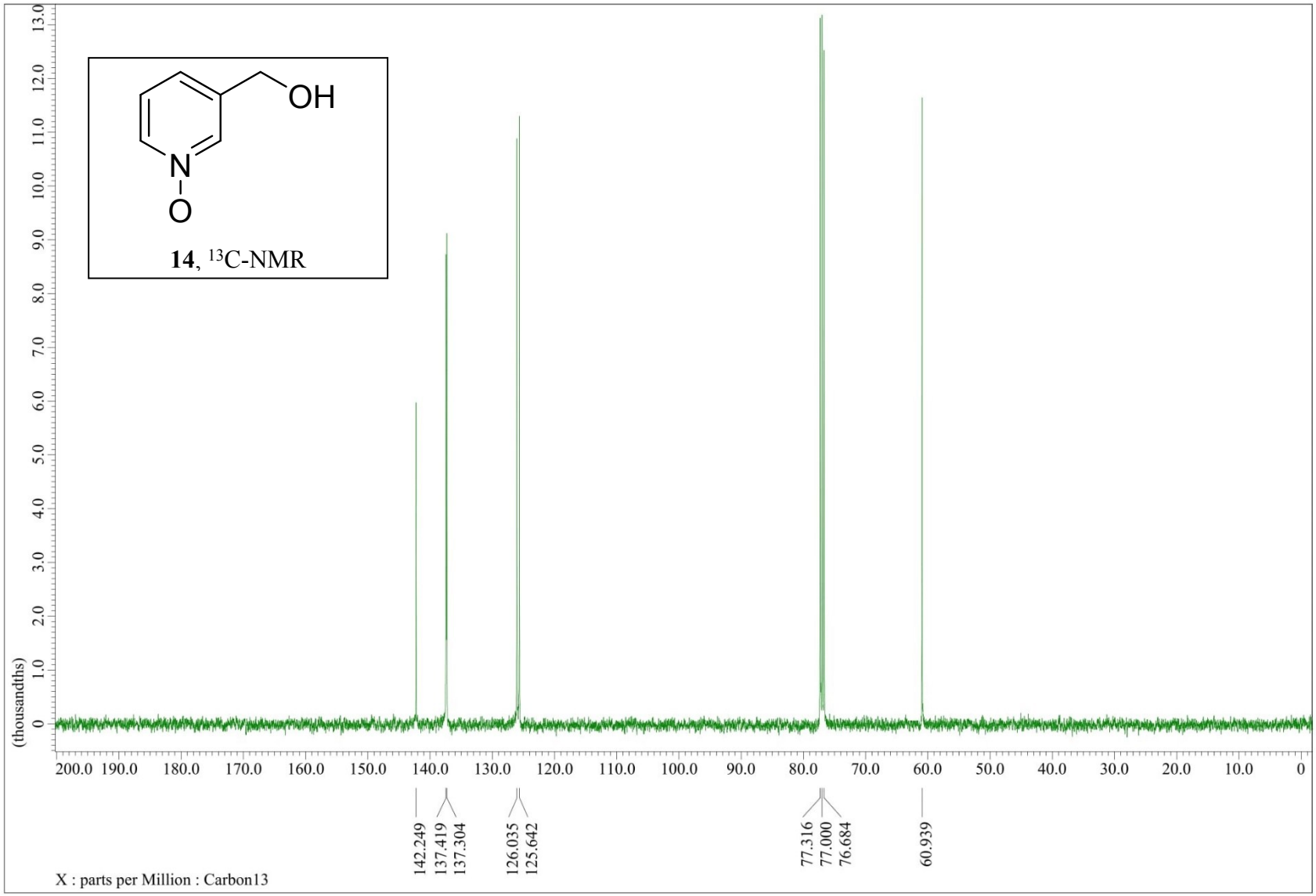

O
OH
O
O
12, 1H-NMR
abundance
3.0
2.0
1.0
0
1.02
0.96
2.02
1.00
2.01
2.00
1.96
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.781
7.777
7.761
7.757
7.528
7.507
7.489
7.485
7.278
7.257
7.250
7.240
7.231
7.213
5.681
4.280
4.265
4.250
3.903
3.888
3.873
2.157
2.141
2.133
2.127
1.875
X : parts per Million : Proton

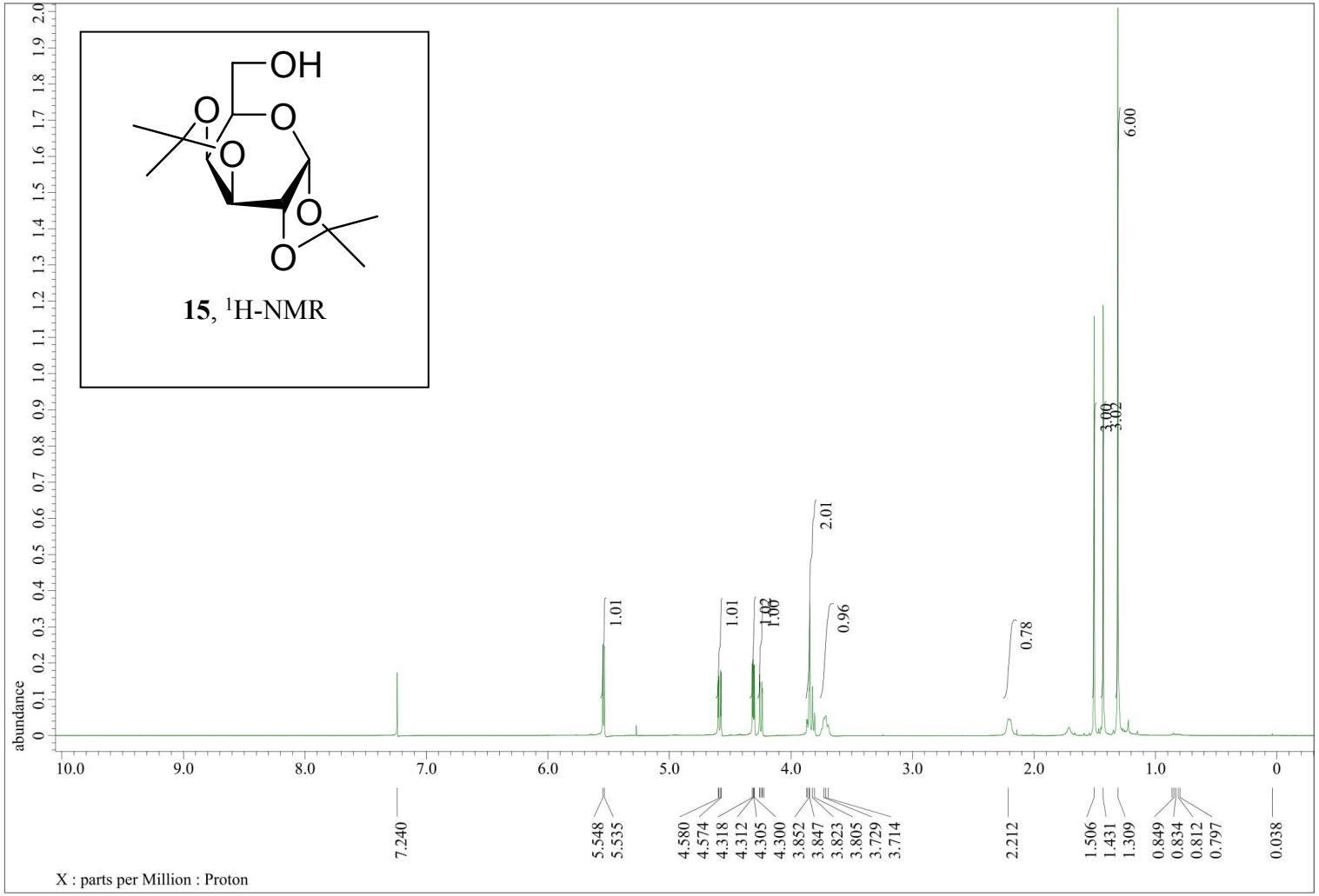

O
OH
O
O
12, 13C-NMR
(thousandths)
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
165.631
163.015
153.259
132.369
123.860
122.892
116.759
115.657
90.531
77.316
77.000
76.684
66.191
58.908
31.367
X : parts per Million : Carbon13

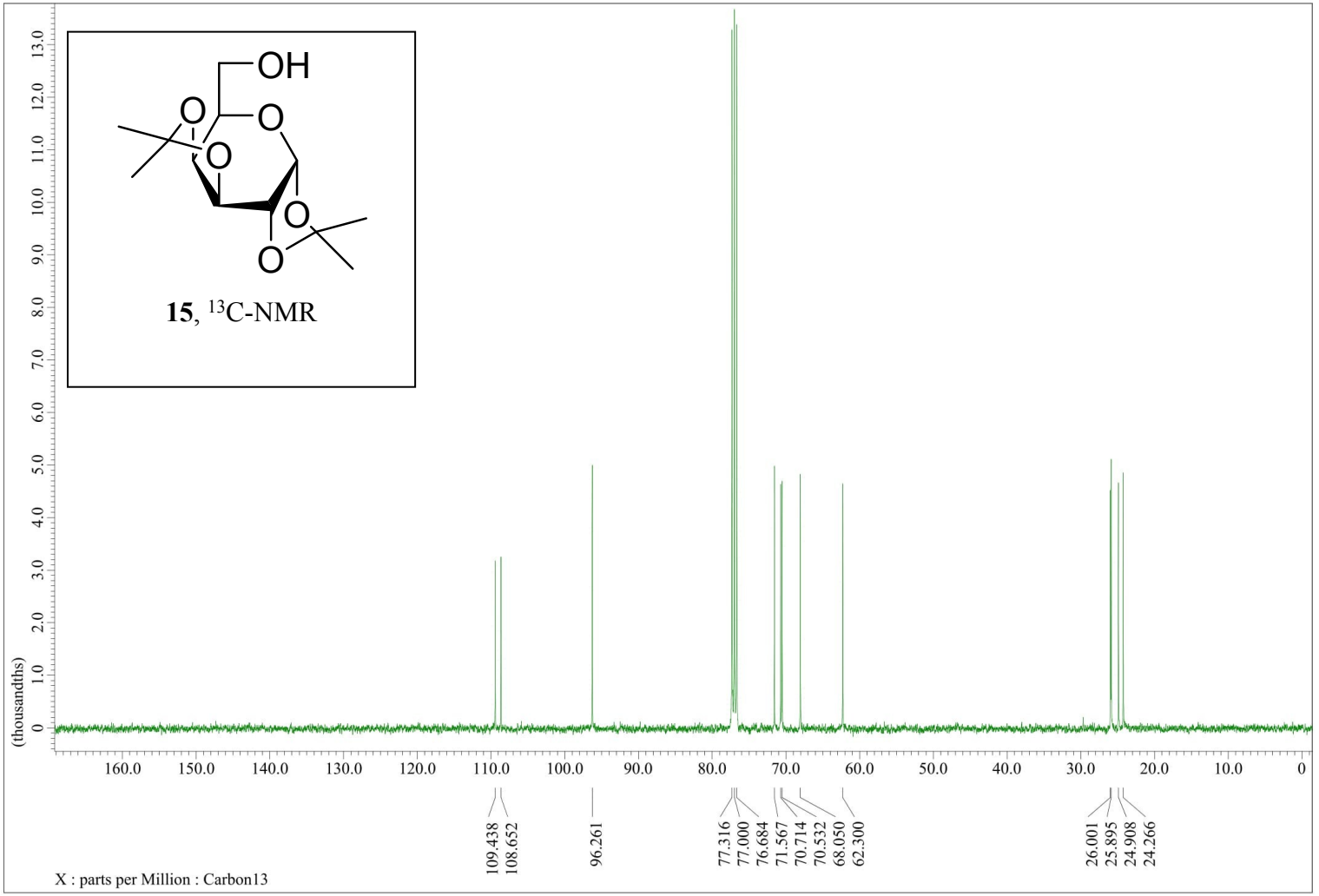

OH
13, 1H-NMR
abundance
0
0.1
0.2
0.3
0.4
0.5
2.99
0.97
2.03
0.97
2.00
1.00
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.827
7.818
7.807
7.797
7.788
7.774
7.727
7.496
7.488
7.480
7.472
7.431
7.428
7.410
7.406
5.325
5.265
5.258
5.236
5.111
4.953
4.933
4.841
4.826
4.793
4.757
4.646
4.576
2.725
X : parts per Million : Proton

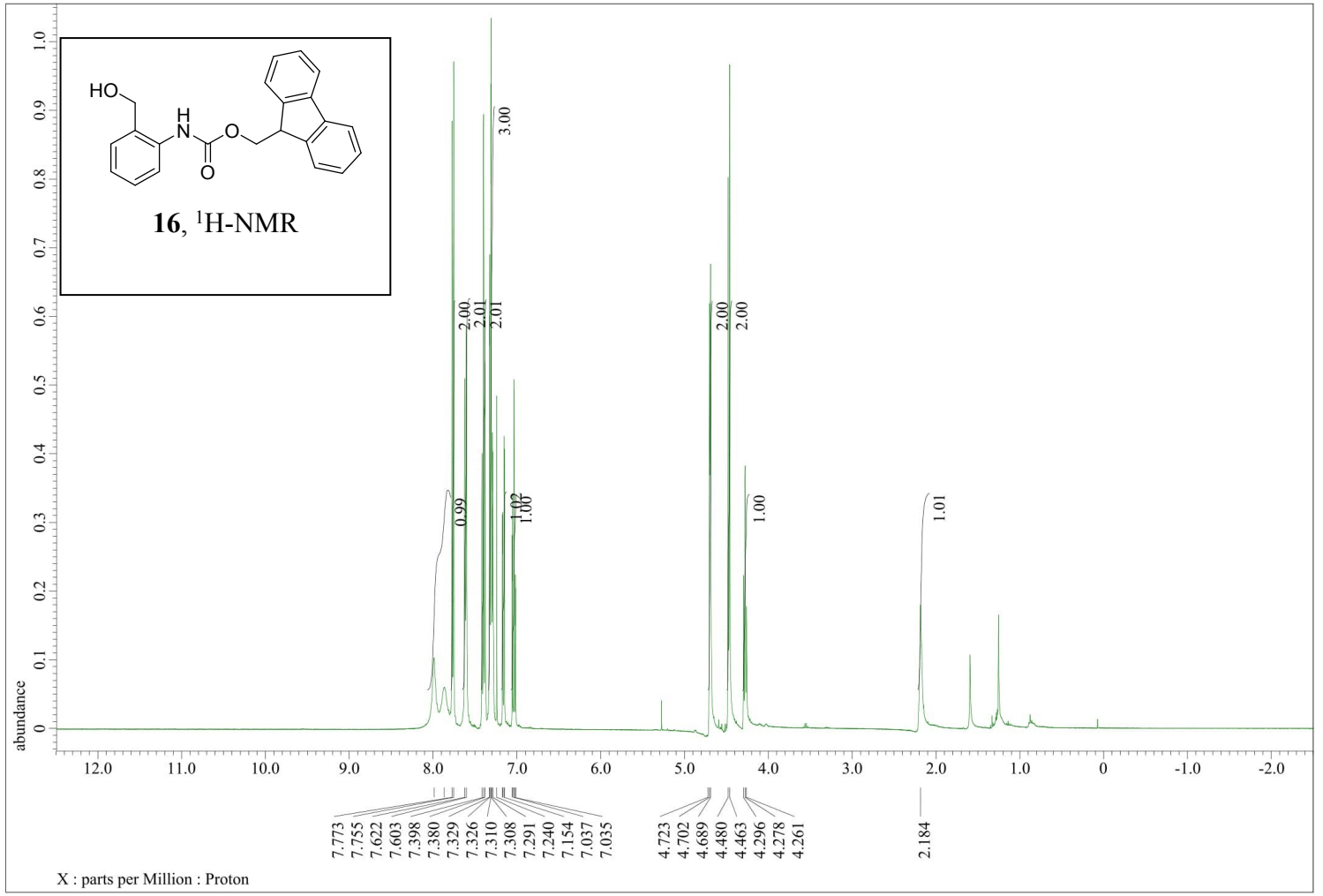

OH
13, 13C-NMR
20.0
10.0
0
(thousandths)
190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
138.214
133.241
132.781
128.133
127.779
127.597
126.025
125.738
125.278
125.067
77.316
77.000
76.684
65.089
X : parts per Million : Carbon13

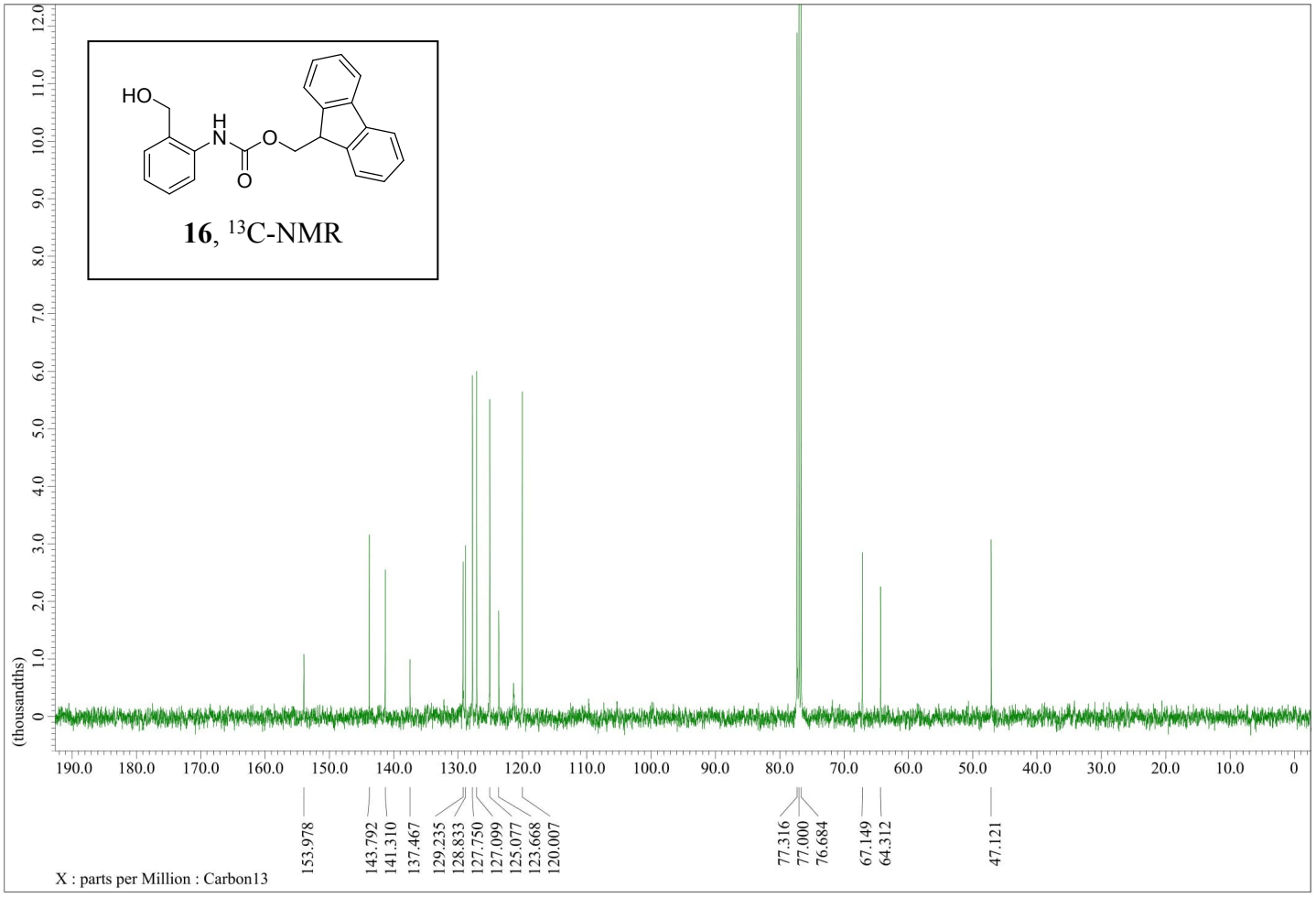

OH
N
O
14, 1H-NMR
abundance
X : parts per Million : Proton
1.00
1.01
1.00
1.02
0.98
2.00
8.134
7.973
7.957
7.271
7.251
7.184
7.168
7.164
7.148
5.739
4.591
4.547


OH
N
O
14, 13C-NMR
(thousandths)
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
142.249
137.419
137.304
126.035
125.642
77.316
77.000
76.684
60.939
X : parts per Million : Carbon13


OH
O
O
O
O
O
15, 1H-NMR
abundance
X : parts per Million : Proton
6.00
3.02
3.00
2.01
1.01
1.01
1.02
1.00
0.96
0.78
7.240
5.548
5.535
4.580
4.574
4.318
4.312
4.305
4.300
3.852
3.847
3.823
3.805
3.729
3.714
2.212
1.506
1.431
1.309
0.849
0.834
0.812
0.797
0.038


OH
O
O
O
O
O
15, 13C-NMR
(thousandths)
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
109.438
108.652
96.261
77.316
77.000
76.684
71.567
70.714
70.532
68.050
62.300
26.001
25.895
24.908
24.266
X : parts per Million : Carbon13


HO
H
N
O
O
16, 1H-NMR
abundance
X : parts per Million : Proton
7.773
7.755
7.622
7.603
7.398
7.380
7.329
7.326
7.310
7.308
7.291
7.240
7.154
7.037
7.035
4.723
4.702
4.689
4.480
4.463
4.296
4.278
4.261
2.184


HO
H
N
O
O
16, 13C-NMR
(thousandths)
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
153.978
143.792
141.310
137.467
129.235
128.833
127.750
127.099
125.077
123.668
120.007
77.316
77.000
76.684
67.149
64.312
47.121
X : parts per Million : Carbon13