Supporting Information

Nickel-Catalyzed Reductive Cleavage of Aryl Alkyl Ethers to Arenes in the Absence of an External Reductant

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CONTENTS

I. General Information

^{1}H and ^{13}C NMR spectra were recorded on a JEOL ECS-400 spectrometer (JEOL, Tokyo, Japan) or VARIAN UNITY INOVA-600 spectrometer in $CDCl_3$ with tetramethylsilane as an internal reference standard. NMR data have been reported as follows: chemical shift (δ) in ppm, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, and m = multiplet), coupling constant (*J*) in Hz, and integration. Infrared spectra (IR) were obtained on a JASCO FT/IR-4000 spectrometer, and the absorptions have been reported in reciprocal centimeters with the following relative intensities: s (strong), m (medium), or w (weak). Mass spectra were obtained on a Shimadzu GCMS-QP 5000 or GCMS-QP 2010 instrument with an ionization voltage of 70 eV. High resolution mass spectra (HRMS) were obtained on a JEOL JMS-DX303. Analytical gas chromatography (GC) was carried out on Shimadzu GC-2014, equipped with a flame ionization detector. Melting points were determined using a Yamato melting point apparatus. Column chromatography was performed over SiO_2 (Silycycle Silica Flash F60 (230-400 mesh)) and NH Silica (Silica Gel 60 (spherical) NH_2 (40-50 μm)). Some of the compounds were purified by LC-908 HPLC (GPC).

II. Materials

Toluene (for Organic Synth.) was purchased from Wako Chemicals and used as received. $Ni(cod)_2$ was purchased from Strem Chemicals and used as received. PCy_3 (Sigma-Aldrich), SIPr·HCl (TCI), IPr·HCl (TCI), IMes·HCl (TCI), I^{i}Pr·HCl (TCI), I(1-Ad)·HCl (Sigma-Aldrich) and NaO^{t}Bu (TCI) were purchased from commercial suppliers and used as received. I^{t}Bu[1] and ICy·HCl[2] were prepared according to the literature procedures. Aryl ethers **1a** (TCI), **1b** (TCI), **14** (TCI), **19** (TCI) and **20** (TCI) were purchased from commercial suppliers and purified by distillation over CaH_2 prior to being used. Aryl ethers **1a-*d*$_3$** (CAS 97073-37-5),[3] **1c** (CAS 70617-42-4),[4] **1d** (CAS 15052-09-2),[5] **1e** (CAS 613-62-7),[6] **1f** (CAS 19420-29-2),[7] **3** (CAS 5085-74-5),[8] **4** (CAS 5328-01-8),[9] **14** (CAS 93359-83-2),[10] **16** (CAS 2584-79-4),[11] **17** (CAS 613-40-1),[12] and **18** (CAS

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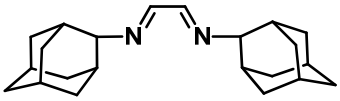
54852-73-2)[13] were prepared from the corresponding phenols or alcohols by treatment with NaH followed by the addition of an alkyl halide. Ethers **6** (CAS 858458-42-1),[14] **8** (CAS 6297-10-5),[15] **10** (CAS 123871-53-4),[16] **19** (CAS 611-94-9),[17] **20** (CAS 6136-7-0)[18] and **23** (CAS 4357-28-2)[19] were prepared according to the literature procedures.

III. Preparation of I(2-Ad)·HCl

***N, N'*-Bis(adamantan-2-yl)ethane-1,2-diimine.**



2-Adamantanamine hydrochloride (14 g, 75 mmol), Et_2O (200 mL), and a saturated aqueous solution of K_2CO_3 (300 mL) were added to a 500 mL flask, and the mixture was stirred for 1 h. The mixture was then partitioned between Et_2O and water. The organic layer was washed with water, dried ($MgSO_4$) and concentrated *in vacuo* to give 2-adamantanamine as a white solid (11 g, 98%). The resulting 2-adamantanamine (11 g, 73 mmol) and an aqueous solution of glyoxal (8.8 M, 4.0 mL, 35 mmol) were dissolved in THF (100 mL), and the mixture was stirred at room temperature for 12 h. The resulting mixture was partitioned between Et_2O and a saturated aqueous solution of $NaHCO_3$. The organic layer was washed with a saturated aqueous solution of $NaHCO_3$ and water, dried ($MgSO_4$) and concentrated *in vacuo* to give the title compound as a white solid (12 g, 100%).

White solid (12 g, 98 %). R_f 0.83 (NH Silica, Et_2O). Mp: 159−162 °C. ^{1}H NMR ($CDCl_3$, 399.78 MHz): 1.54 (s, 2H), 1.90-1.77 (m, 22H), 2.24 (d, J = 12.0 Hz, 4H), 3.42 (s, 2H), 8.04 (s, 2H). ^{13}C NMR ($CDCl_3$, 100.53 MHz): 27.3, 28.0, 31.8, 35.0, 37.2, 37.9, 74.3, 160.5. IR (ATR): 2901 s, 2849 s, 2823 m, 1633 m, 1467 w, 1449 m, 1364 w, 1336 w, 1288 w, 1101 m, 1049 w, 1011 w, 966 w, 930 m, 902 w, 877 w, 809 w, 669 w. Exact Mass (EI) Calcd for $C_{22}H_{32}N_2$: 324.2565. Found: 324.2566.

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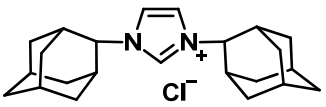
1,3-Bis(adamantan-2-yl)imidazolin-2-ylidene chloride [I(2-Ad)·HCl].



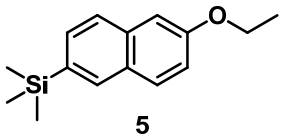
N,*N*,*N*',*N*'-Tetramethyldiaminomethane (6.6 mL, 5.0 g, 49 mmol) and CH_2Cl_2 (60 mL) were added to a dry 500 mL three-necked flask; the mixture was degassed before cooling to 0 °C. Acetyl chloride (3.7 mL, 4.1 g, 52 mmol) was added dropwise to the reaction mixture over 20 min, and the resulting mixture was stirred at 0 °C for 1 h.

A solution of *N*,*N*'-bis(adamantan-2-yl)ethane-1,2-diimine (12 g, 37 mmol) in CH_2Cl_2 (60 mL) was added dropwise to the reaction mixture over 30 min at -78 °C. The reaction mixture was then warmed to room temperature and stirred for 14 h. Et_2O (400 mL) was added to the resulting white suspension, and the white precipitate was collected by filtration. The precipitate was purified by recrystallization (H_2O) to give the title compound as a white powder (9.0 g, 65%).

White powder (9.0 g, 65%). Mp: 318−320 °C. ^{1}H NMR ($CDCl_3$, 399.78 MHz): 1.69-1.64 (m, 4H), 1.80-1.77 (m, 8H), 1.89 (br, 2H), 2.08-2.00 (m, 10H), 2.85 (s, 4H), 4.60 (s, 2H), 7.37 (t, J = 1.4 Hz, 2 H), 10.53 (s, 1H). ^{13}C NMR ($CDCl_3$, 100.53 MHz): 26.56, 26.62, 30.99, 31.10, 36.6, 36.9, 64.0, 119.2, 137.5. IR (ATR): 3298 w, 3091 w, 3039 m, 2920 s, 2901 s, 2855 m, 1736 w, 1544 m, 1470 w, 1449 m, 1390 w, 1372 m, 1342 m, 1282 w, 1244 w, 1188 w, 1159 s, 1106 m, 1075 w, 964 m, 864 s, 821 m, 792 w, 782 w, 765 w, 704 m. Exact Mass (FAB) Calcd for $C_{23}H_{33}N_2$ ([M-Cl]$^+$): 337.2644. Found: 337.2642.

IV. Synthesis of Starting Materials

(6-Ethoxynaphthalen-2-yl)trimethylsilane (5).



5

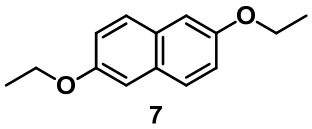
n-BuLi (1.6 M in hexanes, 5.6 mL, 9 mmol) was added to a solution of 2-bromo-6-ethoxynaphthalene (1.5 g, 6.0 mmol)[20] in THF (10 mL) in a dropwise manner at -78 °C, and the mixture was stirred at -78 °C for 1 h. TMSCl (1.5 mL, 12 mmol) was then added dropwise at

[20] Matsunaga,N.; Kaku, T.; Ojida, A.; Tanaka, T.; Hara, T.; Yamaoka, M.; Kusaka, M.; Tasaka, A. *Bioorg. Med. Chem.* **2004**, *12*, 4313.

-78 °C, and the resulting mixture was allowed to warm to room temperature and stirred for 15 h. The reaction was quenched by the addition of an aqueous solution of HCl (1M, 10 mL), and the resultant mixture was extracted with Et_2O. The combined extracts were washed with brine, dried ($MgSO_4$), and concentrated to give a residue, which was purified by column chromatography (hexane/EtOAc = 20/1) to give **5** as a white solid (1.2 g, 84%).

White solid (1.2 g, 84%). Mp 86.9−87.6 °C. R_f 0.40 (SiO_2, hexane/EtOAc = 20/1). ^{1}H NMR ($CDCl_3$, 399.78 MHz) δ: 0.33 (s, 9H), 1.49 (t, *J* = 6.9 Hz, 3H), 4.15 (q, *J* = 6.9 Hz, 2H), 7.10 (s, 1H), 7.14 (d, *J* = 9.2 Hz, 1H), 7.55 (d, *J* = 8.2 Hz, 1H), 7.70 (d, *J* = 8.2 Hz, 1H), 7.73 (d, *J* = 9.2 Hz, 1H), 7.9 (s, 1H). ^{13}C NMR ($CDCl_3$, 100.53 MHz) δ: -1.0, 14.8, 63.4, 106.3, 119.0, 125.9, 128.4, 129.5, 130.3, 133.5, 134.8, 134.9, 157.2. IR (ATR): 3050 w, 2990 w, 2952 w, 2361 w, 1624 w, 1590 , 1469 w, 1393 m, 1320 w, 1263 m, 1250 m, 1215 m, 1172 w, 1132 w, 1113 w, 1089 w, 1045 m, 971 w, 899 w, 854 s, 835 s, 769 w, 750 m, 690 w. MS, *m/z* (relative intensity, %): 244 (40), 230 (20), 229 (100), 201 (16). Exact Mass (EI) Calcd for $C_{15}H_{20}OSi$: 244.1283. Found: 244.1284.

2,6-Diethoxynaphthalene (7).

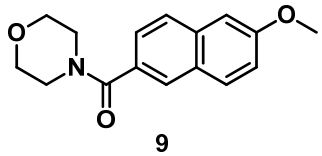


7

2,6-Dihydroxynaphthalene (1.6 g, 10 mmol) was added at 0 °C to a suspension of NaH (60% in mineral oil, 48 mmol) in DMF under an atmosphere of nitrogen, and the resulting mixture was stirred at room temperature for 1 h. EtI (2.6 mL, 32 mmol) was then added to the reaction in a dropwise manner at 0 °C, and the resulting mixture was allowed to warm to room temperature and stirred for 12 h. The reaction was then quenched by the addition of deionized water, and the resulting mixture was extracted with EtOAc (twice). The combined extracts were then washed with brine, dried ($MgSO_4$), and concentrated to give a residue, which was purified by column chromatography (hexane/EtOAc = 20/1) to give **7** as a white solid (0.46 g, 21%).

White solid (0.46 g, 21%). Mp 162.9−163.6 °C. R_f 0.23 (SiO_2, hexane/EtOAc = 20/1). ^{1}H NMR ($CDCl_3$, 399.78 MHz) δ: 1.48 (t, *J* = 6.9 Hz, 6H), 4.12 (q, *J* = 6.9 Hz, 4H), 7.08 (s, *J* = 2.3 Hz, 2H), 7.12 (d, *J* = 2.3, 8.7 Hz, 2H), 7.62 (d, *J* = 8.7 Hz, 2H). ^{13}C NMR ($CDCl_3$, 100.53 MHz) δ: 14.9, 63.4, 106.9, 119.2, 128.0, 129.7, 155.3. IR (ATR): 2983 m, 2934 w, 2883 w, 2154 w, 1602 s, 1508 m, 1474 w, 1458 w, 1442 w, 1393 m, 1381 s, 1336 w, 1268 w, 1231 s, 1164 m, 1113 s, 1043 s, 959 m, 900 w, 856 s, 826 m, 808 w, 759 w, 688 w, 664 w. MS, *m/z* (relative intensity, %):217 (11), 216 (89), 188 (17), 187 (24), 160 (73), 159 (46), 132 (11), 131 (100), 115 (16), 114 (11), 103 (16), 102 (17), 77 (34). Exact Mass (EI) Calcd for $C_{14}H_{16}O_2$: 216.1150. Found: 216.1151.

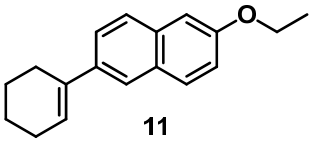
(6-Methoxynaphthalen-2-yl)(morpholino)methanone (9).



9

White solid. Mp 149.0-149.5 °C. R_f 0.52 (NH Silica, hexane/EtOAc = 1/1). ^{1}H NMR ($CDCl_3$, 399.78 MHz) δ: 3.73 (br, 8H), 3.92 (s, 3H), 7.12 (s, 1H), 7.17 (d, *J* = 8.7 Hz, 1H), 7.44 (d, *J* = 8.7 Hz, 1H), 7.74 (d, *J* = 8.7 Hz, 2H), 7.83 (s, 1H). ^{13}C NMR ($CDCl_3$, 100.53 MHz) δ: 42.7 (br), 48.5 (br), 55.3, 66.9, 105.6, 119.7, 124.9, 127.1, 128.0, 129.9, 130.1, 130.2, 135.2, 158.6, 170.6. IR (ATR): 2984 w, 1737 s, 1633 w, 1485 w, 1428 w, 1373 m, 1300 w, 1238 s, 1165 w, 1115 w, 1044 s, 938 w, 847 w, 815 w, 787 w, 745 w. MS, *m/z* (relative intensity, %): 271 (29), 270 (17), 186 (13), 185 (100), 157 (26), 142 (13), 114 (21). Exact Mass (APCI) Calcd for $C_{16}H_{17}NO_3$+H^+: 271.1208. Found: 272.1282.

2-(Cyclohex-1-en-1-yl)-6-ethoxynaphthalene (11).

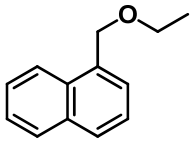


11

A dry three-necked flask was charged with 2-bromo-6-ethoxynaphthalene (0.93 g, 3.7 mmol),[19] 2-(1-cyclohexenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1.0g, 4.8 mmol), K_2CO_3 (2.0 g, 15 mmol), $Pd(PPh_3)_4$ (430 mg, 0.37 mmol), and 1,4-dioxane (6.0 mL) under a nitrogen atmosphere, and the resulting mixture was heated at reflux for 12 h. The mixture was then cooled to room temperature and treated with deionized water, and the resulting mixture was extracted with EtOAc. The combined extracts were then washed with brine, dried ($MgSO_4$) and concentrated to give a residue, which was purified by column chromatography (hexane/EtOAc = 20/1) to give **11** as a white solid, which was further purified by GPC (0.37 g, 40%).

White solid (0.37 g, 40%). Mp 105.6−106.2 °C. R_f 0.31 (SiO_2, hexane/EtOAc = 20/1). ^{1}H NMR ($CDCl_3$, 399.78 MHz) δ: 1.49 (t, *J* = 6.8 Hz, 3H), 1.73-1.71 (m, 2H). 1.86-1.83 (m, 2H), 2.27-2.25 (m, 2H), 2.55-2.52 (m, 2H), 4.15 (q, *J* = 6.8 Hz, 2H), 6.28-6.26 (m, 1H), 7.14-7.11 (m, 2H), 7.57 (d, *J* = 8.7 Hz, 1H), 7.66 (d, *J* = 8.7 Hz, 1H), 7.72-7.70 (m, 2H). ^{13}C NMR ($CDCl_3$, 100.53 MHz) δ: 14.8, 22.2, 23.1, 26.0, 27.3, 63.4, 106.3, 118.9, 122.9, 124.1, 124.5, 126.4, 128.8, 129.5, 133.4, 136.2, 137.5, 156.6. IR (ATR): 2979 w, 2929 m, 2832 w, 2250 w, 1675 w, 1626 w, 1600 m, 1502 w, 1473 m, 1391 m, 1263 m, 1233 m, 1195 m, 1174 m, 1114 w, 1045 m, 970 w, 951 w, 908 s, 854 m, 816 m, 730 s, 665 w. MS, *m/z* (relative intensity, %):253 (20), 252 (100), 224 (21), 223 (25), 196 (14), 195 (24), 165 (13), 152 (10), 115(14). Exact Mass (EI) Calcd for $C_{18}H_{20}O$: 252.1514. Found: 252.1516.

1-(Ethoxymethyl)naphthalene (15).

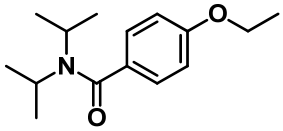


15

1-Naphthalenemethanol (0.79 g, 5.0 mmol) was added to a suspension of NaH (60% in mineral oil, 12 mmol) in DMF at 0 °C under a nitrogen atmosphere, and the resulting mixture was stirred at room temperature for 1 h. EtI (0.64 mL, 8.0 mmol) was then added to the reaction in a dropwise manner at 0 °C, and the resulting mixture was allowed to warm to room temperature and stirred for 12 h. The reaction was then quenched by the addition of deionized water, and the resulting mixture was extracted with EtOAc (twice). The combined extracts were then washed with brine, dried ($MgSO_4$) and concentrated to give a residue, which was purified by column chromatography (hexane/EtOAc = 20/1) to give **15** as a colorless oil (0.78 g, 84%).

Colorless oil (0.78 g, 84%). R_f 0.40 (SiO_2, hexane/EtOAc = 20/1). ^{1}H NMR ($CDCl_3$, 600.13 MHz) δ: 1.30 (t, *J* = 4.6 Hz, 3H), 3.65 (q, *J* = 4.6 Hz, 2H), 4.98 (s, 2H), 7.45 (d, *J* = 4.6 Hz, 1H), 7. 53-7.50 (m, 2H), 7.56 (dd, *J* = 4.6, 5.6 Hz, 1H), 7.82 (d, *J* = 5.6 Hz, 1H), 7.88 (d, *J* = 5.4 Hz, 1H), 8.14 (d, *J* = 5.6 Hz, 1H). ^{13}C NMR ($CDCl_3$, 150.92 MHz) δ: 15.3, 65.8, 71.2, 124.0, 125.2, 125.7, 126.0, 126.2, 128.45, 128.47, 131.7, 133.7, 134.0. IR (ATR): 3047 w, 2975 m, 2930 w, 2866 m, 2349 w, 1691 w, 1598 w, 1511 w, 1442 w, 1376 w, 1265 w, 1231 w, 1166 m, 1095 s, 1073 m, 1015 w, 911 w, 893 w, 859 w, 793 s, 773 s, 734 w. MS, *m/z* (relative intensity, %): 186 (29), 143 (11), 142 (100), 141 (95), 139 (16), 129 (78), 128 (39), 127 (28), 115 (53). Exact Mass (EI) Calcd for $C_{13}H_{14}O$: 186.1045. Found: 186.1045.

4-Ethoxy-N,N-diisopropylbenzamide (21).



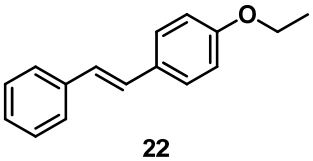
21

Diisopropylamine (3.1 mL, 22 mmol) was added dropwise at 0 °C to a stirred solution of 4-ethoxy benzoylchloride (3.2 mL, 20 mmol) and triethylamine (9.0 mL, 70 mmol) in anhydrous CH_2Cl_2 (50 mL) under an atmosphere of nitrogen. The mixture was stirred at 50 °C for 18 h before cooling and washing with aqueous solution of HCl (1M) and a saturated aqueous solution of ammonium chloride. The organic layer was separated, and the solvents were evaporated under reduced pressure to give a residue, which was purified by column chromatography (NH silica, hexane/EtOAc = 10/1) to give 21 as a colorless oil (4.3 g, 85%).

White solid (4.3 g, 85%). Mp 58.5−59.0 °C. R_f 0.11 (NH Silica, hexane/EtOAc = 10/1). ^{1}H NMR

($CDCl_3$, 399.78 MHz) δ: 1.42-1.30 (m, 15H), 3.68 (br, 2H), 4.01 (q, *J* = 6.9 Hz, 2H), 6.87-6.82 (m, 2H), 7.26-7.20 (m, 2H). ^{13}C NMR ($CDCl_3$, 100.53 MHz) δ: 14.6, 20.7, 46.0 (br), 50.2 (br), 63.3, 114.0, 127.3, 131.0, 159.1, 170.8. IR (ATR): 2979 w, 1739 m, 1630 w, 1609 m, 1512 w, 1440 w, 1372 m, 1336 w, 1296 w, 1242 s, 1174 w, 1114 w, 1045 m, 919 w, 842 w, 764 w, 735 w, 698 w. MS, *m/z* (relative intensity, %): 149 (100), 121 (58), 93 (19), 18 (65). Exact Mass (EI) Calcd for $C_{15}H_{23}O_2$: 249.1729. Found: 249.1728.

(E)-1-Ethoxy-4-styrylbenzene (22).



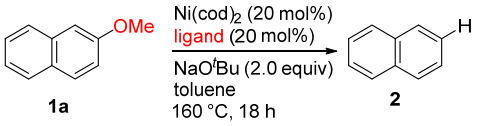
22

A dry three-necked flask was charged with 4-bromoethoxybenzene (2.3 mL, 17 mmol), *trans*-2-phenylvinylboronic acid (3.1 g, 21 mmol), K_2CO_3 (8.2 g, 59 mmol), $Pd(PPh_3)_4$ (700 mg, 0.60 mmol), and THF (70 mL) under a nitrogen atmosphere, and the resulting mixture was heated at reflux for 12 h. The mixture was cooled to room temperature and treated with deionized water, and the resulting mixture was extracted with EtOAc. The combined extracts were then washed with brine, dried ($MgSO_4$) and concentrated to give a residue, which was purified by column chromatography (hexane/EtOAc = 20/1) to give **22** as a white solid, which was further purified by GPC (1.1 g, 29%). White solid (1.1 g, 29%). Mp 133.8−134.3 °C. R_f 0.51 (SiO_2, hexane/EtOAc = 20/1). ^1H NMR ($CDCl_3$, 399.78 MHz) δ: 1.43 (t, *J* = 6.9 Hz, 3H), 4.06 (q, *J* = 6.9 Hz, 2H), 6.89 (d, *J* = 8.7 Hz, 2H), 6.97 (d, *J* = 16.0 Hz, 1H), 7.07 (d, *J* = 16.0 Hz, 1H), 7.23 (dd, *J* = 7.6, 7.6 Hz, 1H), 7.35 (d, *J* = 7.3 Hz, 2H), 7.45 (d, *J* = 8.7 Hz, 2H), 7.49 (d, *J* = 7.3 Hz, 2H). ^{13}C NMR ($CDCl_3$, 100.53 MHz) δ: 14.8, 63.4, 114.6, 126.2, 126.4, 127.2, 127.7, 128.2, 128.6, 129.9, 137.6, 158.6. IR (ATR): 2984 w, 2359 w, 1738 s, 1446 w, 1372 m, 1301 w, 1235 s, 1098 w, 1044 s, 937 w, 847 w, 786 w, 696 w, 667 w. MS, *m/z* (relative intensity, %): 225 (16), 224 (100), 196 (42), 195 (40), 177 (12), 167 (22), 165 (25), 152 (18), 115 (11), 89 (12). Exact Mass (EI) Calcd for $C_{16}H_{16}O$: 224.1201. Found: 224.1203.

V. Optimization Studies

The effect of the ligand was initially examined using **1a** (0.25 mmol), $Ni(cod)_2$ (0.050 mmol), ligand (0.050 mmol), NaO^tBu (0.50 mmol) in toluene (0.50 mL) at 160 °C for 18 h (Table 1). Under these conditions, I(2-Ad) was found to be an optimal ligand with a reduced product **2** being formed in 84% yield (Entry 9).

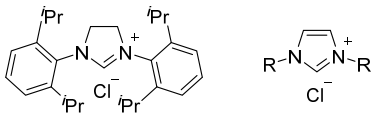
Table 1 Ni-catalyzed reductive cleavage of **1a**: effect of ligands[a]



1a **2**

Entry	Ligand	GC yield of **2** (%)
1	PCy_3	0
2	SIPr·HCl	5
3	IPr·HCl (R = 2,6-$^{i}Pr_2C_6H_4$)	31
4	IMes·HCl (R = 2,4,6-$Me_3C_6H_2$)	38
5	I^{i}Pr·HCl (R = ^{i}Pr)	2
6	I^{t}Bu·HCl (R = ^{t}Bu)	3
7	ICy·HCl (R = cyclohexyl)	50
8	I(1-Ad)·HCl (R = 1-adamantyl)	45
9	I(2-Ad)·HCl (R = 2-adamantyl)	84

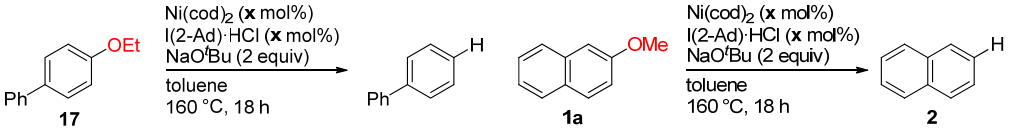
ligand:



SIPr·HCl

[a] Reaction conditions: **1a** (0.25 mmol), $Ni(cod)_2$ (0.050 mmol), ligand (0.050 mmol), NaO^{t}Bu (0.50 mmol) in toluene (0.50 mL) at 160 °C for 18 h.

The catalyst loading can be reduced to 10 mol% without a significant decrease in the yield of the reduced product when ethyl ethers, such as **17** were used as substrates (see below left). However, the decrease in yield was significant in the case of methyl ethers, such as **1a** (see below right).

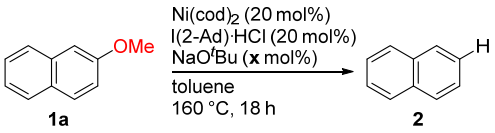


17 **1a** **2**

x (mol%)	GC yield of **2** (%)
10	69
20	78

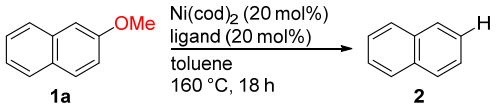
x (mol%)	GC yield of **2** (%)
10	52
20	84

We have also examined the amount of the base, as shown in the Table below. The reaction proceeded when the amount of NaO^{t}Bu was reduced to a catalytic amount (25 mol%), although the yield of the product was slightly decreased. Therefore, we set 2.0 equivalents as the standard amount of NaO^{t}Bu to maximize the yield of the product. In the cases of ketone **19** and **20**, the use of 2 equivalents of NaO^{t}Bu complicated the reaction, probably due to undesirable reactions of the ketone moiety with NaO^{t}Bu. The use of 0.50 equivalents of NaO^{t}Bu was found to give the optimal yield of the reduced product.



x (mol%)	GC yield of **2** (%)
25	63
50	80
200	84

To gain further insight into the role of NaO^{t}Bu, we have conducted the reaction in the absence of NaO^{t}Bu using pure IMes (we were unable to isolate pure I(2-Ad) due to its unstability). As a result, the reaction proceeded in the absence of NaO^{t}Bu (see below). These results suggest that the key elementary steps involved in this reaction, *i.e.*, the oxidative addition of a C-OMe bond and β-hydrogen elimination of Ni-OMe, can basically proceed in the absence of NaO^{t}Bu. The slight improvement in yield by the presence of NaO^{t}Bu can be attributed to the accelerating effect of these elementary steps and/or the assistance of catalyst turnover.

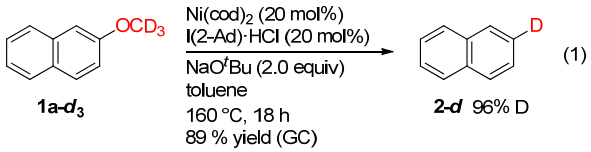


ligand	GC yield of **2** (%)
IMes	22
IMes·HCl [NaO^{t}Bu (2 equiv) was used.]	38

VI. Typical Procedure for Ni/I(2-Ad)-Catalyzed Reductive Cleavage of Aryl Methyl Ethers (Tables 2 and 3)

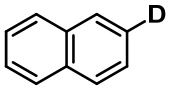
$Ni(cod)_2$ (14 mg, 0.050 mmol), I(2-Ad)·HCl (13 mg, 0.050 mmol), NaO^{t}Bu (48 mg, 0.50 mmol) and toluene (0.25 mL) were added to a 10 mL-sample vial with a Teflon-sealed screwcap in a glovebox filled with nitrogen, and the resulting mixture was stirred at room temperature for 3 min. An aryl methyl ether (0.25 mmol) in toluene (0.25 mL) was then added to the vial and the cap was closed. The contents of the vial were stirred at 160 °C for 18 h. The reaction mixture was cooled to room temperature, and the crude mixture was filtered through a pad of silica gel, and then analyzed by GC. The filtrate was concentrated *in vacuo* to give a residue, which was purified by flash column chromatography over silica gel.

VII. Labeling Studies (Eq 1)



Compound **1a-*d*$_3$** was subjected to the typical reaction conditions described in section V (eq 1). The yield of **2-*d*** was determined by GC analysis using undecane as an internal substance due to its sublimability. The deuterium content of **2-*d*** obtained under these conditions was determined to be 96% by ^{1}H NMR spectroscopy.

2-Deuteronaphthalene (2-*d*). [CAS: 2430-34-4]

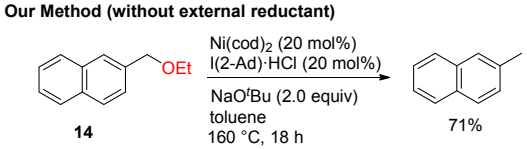


2-*d*

White solid (90%). R_f 0.66 (SiO_2, hexane/EtOAc = 20/1).^{1}H NMR ($CDCl_3$, 399.78 MHz) δ: 7.50-7.48 (s, 3H), 7.87-7.85 (m, 4H). ^{13}C NMR ($CDCl_3$, 100.53 MHz) δ: 125.7, 125.8, 127.7, 127.9, 133.4. ^{2}D NMR (acetone-d_6, 399.78 MHz) δ: 7.44. Exact Mass (EI) Calcd for $C_{10}H_7D$: 129.0689. Found: 129.0687.

VIII. Comparison with Reported Catalytic Systems (Scheme 2)

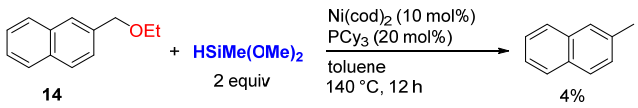
Benzyl ether **14** underwent the reductive cleavage reaction under the current conditions to give the reduced product in 71% yield.



In contrast, the reduced product was formed in only 4% yield when the previously reported catalytic conditions using hydrosilane[21] was directly applied to this substrate.

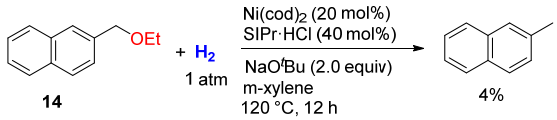
[21] Tobisu, M.; Yamakawa, K.; Shimasaki, T.; Chatani, N. *Chem. Commun.* **2011**, *47*, 2946.

Previous Method Using Hydrosilane (ref 21)



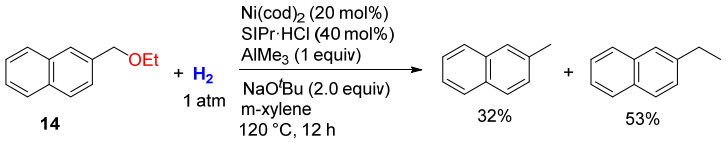
The reported conditions using hydrogen[22] could not promote the reductive cleavage of **14**, either.

Previous Method Using Hydrogen (ref 22)



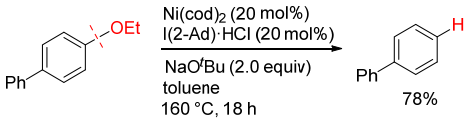
The other condition using stoichiometric $AlMe_3$, which was also reported by Hartwig, was also tested for the reaction of **14** and the product was obtained in 32% yield, and 53% of a methylated product, *i.e.*, 2-ethylbenzene, was also formed. This result indicated that organoaluminum reagents[23] can be used for cross-coupling of aryl and benzyl ethers, and studies along this line is in progress in our laboratory.

Previous Method Using Hydrogen + 1 equiv of $AlMe_3$ (ref 22)

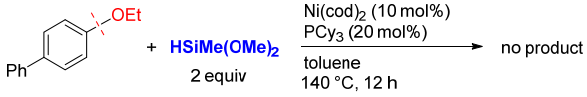


The method using hydrosilane as the reductant (see refs 21) is not applicable to the substrates that do not contain a fused aromatic ring, and therefore the scope is much narrower than the current method.

Our Method (without external reductant)



Previous Method Using Hydrosilane (ref 21)



The method using hydrogen as a reductant (ref 22) cannot be applied to substrates bearing reducible functionalities, such as alkenes and keotones, which are compatible with our methods (See compounds **10**, **19**, **20**, and **22**).

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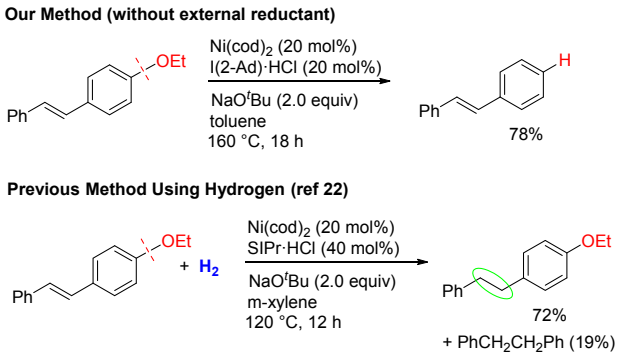
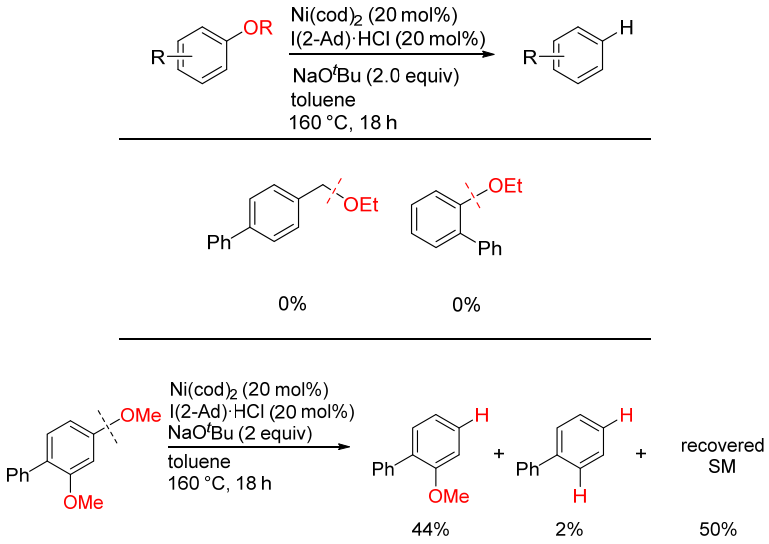
[22] Hartwig, J. F.; Sergeev, A. G. *Science* **2011**, *332*, 439.
[23] Wang, C.; Ozaki, T.; Takita, R.; Uchiyama, M. *Chem.– Eur. J.* **2012**, *18*, 3482.



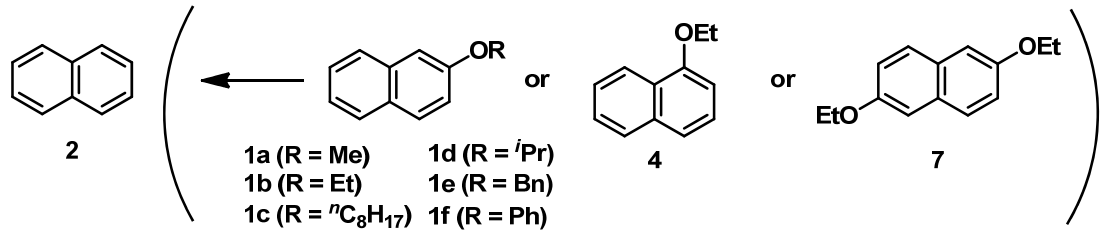
IX. Inapplicable Substrates

Benzyl ethers that do not contain a fused aromatic ring are unreactive under these conditions. Although ortho substitution cannot be tolerated, this provides us with the opportunity to achieve the selective cleavage of the less hindered C-O bond of polyalkoxyarenes. Indeed, 2,4-dimethoxy-1,1'-biphenyl underwent demethoxylation at the less hindered site in a selective manner, as shown below.



X. Spectroscopic Data of Products

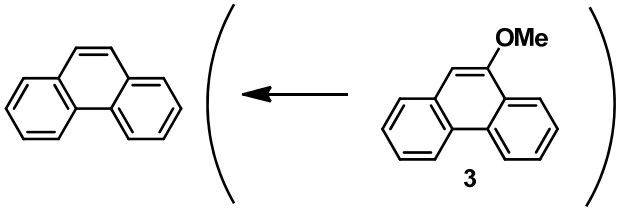
Naphthalene (2) [CAS: 91-20-3].



The product was obtained from either **1a**-**1f**, **4** or **7** under the typical conditions. The yield was determined by GC analysis using undecane as an internal substance due to sublimability of the product.

White solid. R_f 0.63 (Hexane/EtOAc = 50/1). ^{1}H NMR ($CDCl_3$, 399.78 MHz) δ: 7.55-7.51 (m, 4H), 7.90-7.87 (m, 4H). ^{13}C NMR ($CDCl_3$, 100.53 MHz) δ: 125.8, 127.8, 133.4. Exact Mass (EI) Calcd for $C_{10}H_8$: 128.0626. Found: 128.0624.

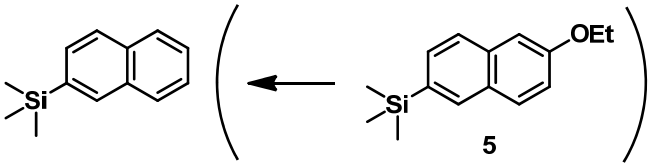
Phenanthrene [CAS: 85-01-8].



The typical procedure was followed using **3** as the substrate.

White solid (36 mg, 80%). R_f 0.43 (SiO_2, hexane/EtOAc = 50/1). ^{1}H NMR ($CDCl_3$, 399.78 MHz) δ: 7.61 (dd, *J* = 6.9, 7.8 Hz, 2H), 7.67 (dd, *J* = 6.9, 8.2 Hz, 2H), 7.76 (s, 2H), 7.91 (d, *J* = 8.2 Hz, 2H), 8.71 (d, *J* = 7.8 Hz, 2H). ^{13}C NMR ($CDCl_3$, 100.53 MHz) δ: 122.6, 126.54, 125.55, 126.9, 128.5, 130.3, 132.0. Exact Mass (EI) Calcd for $C_{14}H_{10}$: 178.07825. Found: 178.0785.

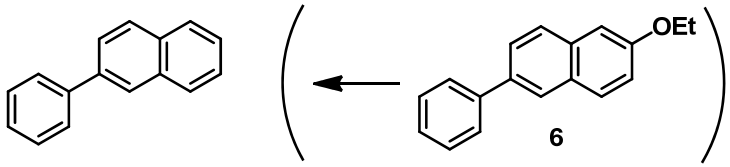
Trimethyl(naphthalen-2-yl)silane [CAS: 18052-85-2].



The typical procedure was followed using **5** as the substrate.

White solid (47 mg, 93%). R_f 0.63 (SiO_2, hexane/EtOAc = 20/1). ^{1}H NMR ($CDCl_3$, 399.78 MHz) δ: 0.38 (s, 9H), 7.50 (dd, *J* = 6.0, 6.8 Hz, 2H), 7.64 (d, *J* = 8.2 Hz, 1H), 7.86-7.83 (m, 3H), 8.03 (s, 1H). ^{13}C NMR ($CDCl_3$, 100.53 MHz) δ: −1.1, 125.8, 126.2, 126.9, 127.7, 128.0, 129.8, 132.9, 133.6, 133.7, 137.9. Exact Mass (EI) Calcd for $C_{13}H_{16}Si$: 200.1021. Found: 200.1020.

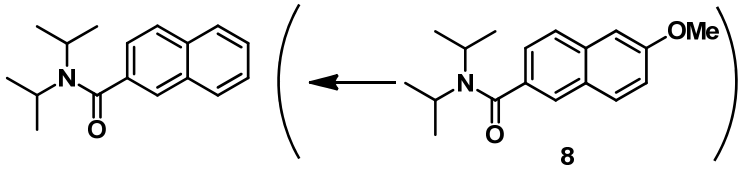
2-Phenylnaphthalene [CAS: 612-94-2].



The typical procedure was followed using **6** as the substrate.

White solid (50 mg, 97%). R_f 0.51 (SiO_2, hexane/EtOAc = 20/1). ^1H NMR ($CDCl_3$, 399.78 MHz) δ: 7.43 (d, *J* = 7.8 Hz, 1H), 7.57-7.51 (m, 4H), 7.80-7.76 (m, 3H), 7.97-7.90 (m, 3H), 8.09 (s, 1H). ^{13}C NMR ($CDCl_3$, 100.53 MHz) δ: 125.6, 125.8, 125.9, 126.3, 127.3, 127.4, 127.6, 128.2, 128.4, 128.8, 132.6, 133.6, 138.5, 141.1. Exact Mass (EI) Calcd for $C_{11}H_{10}$: 204.0939. Found: 204.0941.

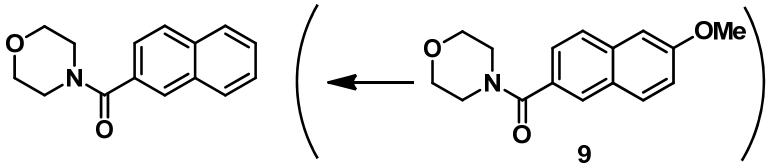
***N*,*N*-Diisopropyl-6-methoxy-2-naphthamide [CAS: 31609-22-0].**



The typical procedure was followed using **8** as the substrate.

White solid (53 mg, 83%). R_f 0.2 (NH Silica, hexane/EtOAc = 10/1). ^1H NMR ($CDCl_3$, 399.78 MHz) δ: 1.56-1.18 (m, 12H), 3.87-3.60 (m, 2H), 7.42 (d, *J* = 8.2 Hz, 1H), 7.53-7.49 (m, 2H), 7.80 (s, 1H), 7.87-7.83 (m, 3H). ^{13}C NMR ($CDCl_3$, 100.53 MHz) δ: 20.7, 46.0 (br), 50.9 (br), 123.4, 124.8, 126.50, 126.51, 127.7, 128.20, 128.25, 132.9, 133.1, 136.2, 171.0. Exact Mass (EI) Calcd for $C_{17}H_{21}NO$: 255.1623. Found: 255.1625.

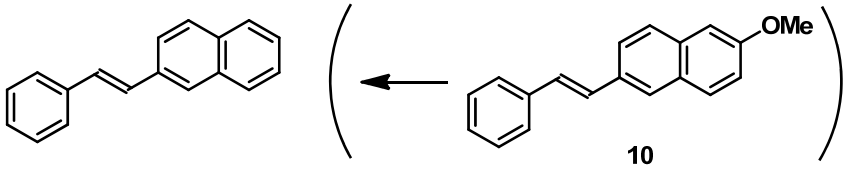
Morpholino(naphthalen-2-yl)methanone [CAS: 26162-88-9].



The typical procedure was followed using **9** as the substrate.

White solid (41 mg, 53%). R_f 0.40 (NH Silica, hexane/EtOAc = 5/1). ^1H NMR ($CDCl_3$, 399.78 MHz) δ: 3.79-3.53 (m, 8H), 7.49 (d, *J* = 8.2 Hz, 1H), 7.57-7.52 (m, 2H), 7.91-7.85 (m, 4H). ^{13}C NMR ($CDCl_3$, 100.53 MHz) δ: 42.7 (br), 48.3 (br), 66.9, 124.2, 126.7, 127.0, 127.2, 127.8, 128.37, 128.42, 132.5, 132.6, 133.7, 170.5. Exact Mass (EI) Calcd for $C_{15}H_{15}NO_2$: 241.1101. Found: 241.1100.

(E)-2-Styrylnaphthalene [CAS: 2039-70-5].

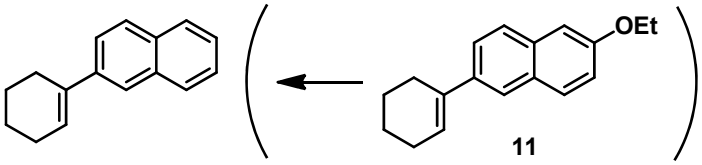


10

The typical procedure was followed using **10** as the substrate.

White solid (49 mg, 86%). R_f 0.40 (SiO_2, hexane/EtOAc = 50/1). ^1H NMR ($CDCl_3$, 399.78 MHz) δ: 7.36-7.28 (m, 3H), 7.44 (dd, *J* = 7.3, 7.8 Hz, 2H), 7.51 (dd, *J* = 6.9, 7.8 Hz, 2H), 7.62 (d, *J* = 7.3 Hz, 2H), 7.79 (d, *J* = 8.7 Hz, 1H), 7.90-7.84 (m, 4H). ^{13}C NMR ($CDCl_3$, 100.53 MHz) δ: 123.5, 125.9, 126.3, 126.5, 126.6, 127.6, 127.7, 128.0, 128.3, 128.7, 128.7, 129.0, 133.0, 133.7, 134.8, 137.3. Exact Mass (EI) Calcd for $C_{18}H_{14}$: 230.1096. Found: 230.1094.

2-(Cyclohex-1-en-1-yl)naphthalene. [CAS: 54607-03-3]

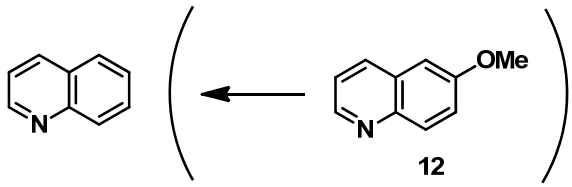


11

The typical procedure was followed using **11** as the substrate.

White solid (52 mg, 99%). R_f 0.57 (SiO_2, hexane/EtOAc = 100/1). ^1H NMR ($CDCl_3$, 399.78 MHz) δ: 1.76-1.72 (m, 2H). 1.90-1.84 (m, 2H), 2.32-2.28 (m, 2H), 2.59-2.55 (m, 2H), 6.33-6.31 (m, 1H), 7.43 (dd, *J* = 6.9, 7.4 Hz, 1H), 7.47 (dd, *J* = 6.9, 7.8 Hz, 1H), 7.62 (d, *J* = 8.7 Hz, 1H), 7.84-7.78 (m, 4H). ^{13}C NMR ($CDCl_3$, 100.53 MHz) δ: 22.2, 23.1, 26.0, 27.3, 123.1, 123.8, 125.3, 125.5, 125.9, 127.4, 127.5, 128.0, 132.4, 133.5, 136.3, 139.7. Exact Mass (EI) Calcd for $C_{16}H_{16}$: 208.1252. Found: 208.1253.

Quinoline [CAS: 91-22-5].

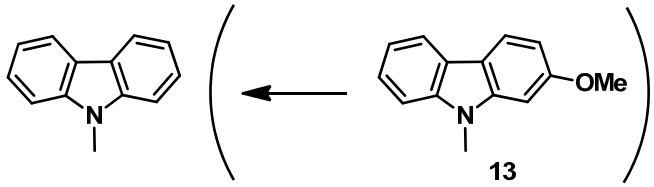


12

The typical procedure was followed using **12** as the substrate. The yield was determined by GC analysis using undecane as an internal substance due to volatility of the product.

Colorless oil (85%). R_f 0.11 (SiO_2, hexane/EtOAc = 20/1). ^1H NMR ($CDCl_3$, 399.78 MHz) δ: 7.31 (dd, *J* = 4.1, 8.2 Hz, 1H), 7.49 (dd, *J* = 6.9, 8.2 Hz, 1H), 7.66 (dd, *J* = 6.9, 8.2 Hz, 1H), 7.75 (d, *J* = 8.2 Hz, 1H), 8.13 (d, *J* = 8.2 Hz, 2H), 8.88 (d, *J* = 4.1 Hz, 1H). ^{13}C NMR ($CDCl_3$, 100.53 MHz) δ: 120.9, 126.4, 127.6, 128.1, 129.29, 129.32, 135.9, 148.1, 150.2. Exact Mass (EI) Calcd for C_9H_7N: 129.0579. Found: 129.0579.

9-Methyl-9H-carbazolel [CAS: 1484-12-4].

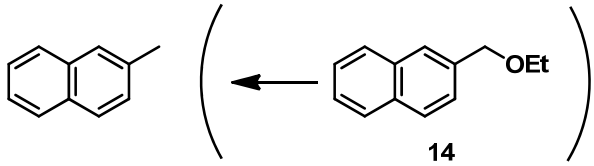


13

The typical procedure was followed using **13** as the substrate.

White solid (40 mg, 89%). R_f 0.26 (NH Silica, hexane/EtOAc = 50/1). ^{1}H NMR ($CDCl_3$, 399.78 MHz) δ: 3.86 (s, 3H), 7.27 (dd, *J* = 7.3 Hz, 7.8 Hz, 2H), 7.42 (d, *J* = 8.2 Hz, 2H), 7.52 (dd, *J* = 7.3, 8.2 Hz, 2H), 8.14 (d, *J* = 7.82 Hz, 2H). ^{13}C NMR ($CDCl_3$, 100.53 MHz) δ: 29.0, 108.4, 118.8, 120.3, 122.7, 125.6, 140.9. Exact Mass (EI) Calcd for $C_{11}H_{10}$: 181.0892. Found: 181.0894.

2-Methylnaphthalene [CAS: 91-57-6].

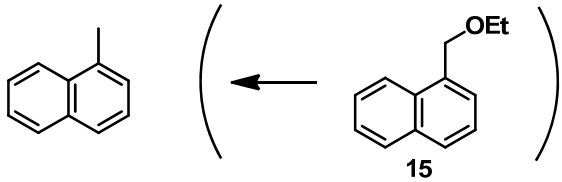


14

The typical procedure was followed using **14** as the substrate. The yield was determined by GC analysis using undecane as an internal substance due to volatility of the product.

Colorless oil (71%). R_f 0.74 (SiO_2, hexane/EtOAc = 20/1). ^{1}H NMR ($CDCl_3$, 399.78 MHz) δ: 2.53 (s, 3H), 7.33 (d, *J* = 8.3 Hz, 1H), 7.41 (dd, *J* = 7.3, 7.3 Hz, 1H), 7.44 (dd, *J* = 7.3, 7.8 Hz, 1H), 7.65 (s, 1H), 7.75 (d, *J* = 7.8 Hz, 1H), 7.77 (d, *J* = 8.3 Hz, 1H), 7.81 (d, *J* = 7.8 Hz, 1H). ^{13}C NMR ($CDCl_3$, 100.53 MHz) δ: 27.7, 124.9, 125.8, 126.8, 127.2, 127.56, 127.65, 128.1, 131.6, 133.6, 135.4. Exact Mass (EI) Calcd for $C_{11}H_{10}$: 142.07825. Found: 142.0782.

1-Methylnaphthalene [CAS: 90-12-0].



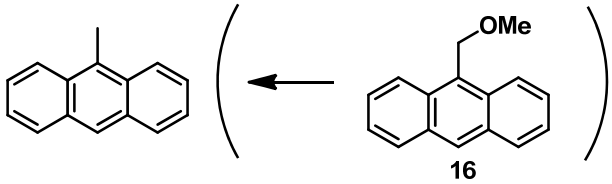
15

The typical procedure was followed using **15** as the substrate. The yield was determined by GC analysis using undecane as an internal substance due to volatility of the product.

Colorless oil (69%). R_f 0.49 (SiO_2, hexane/EtOAc = 50/1). ^{1}H NMR ($CDCl_3$, 399.78 MHz) δ: 2.73 (s, 3H), 7.35 (d, *J* = 6.9 Hz, 1H), 7.41 (dd, *J* = 6.8, 7.8 Hz, 1H), 7.51 (dd, *J* = 6.8, 7.8 Hz, 1H), 7.55 (dd, *J* = 6.9, 8.2 Hz, 1H), 7.74 (d, *J* = 7.8 Hz, 1H), 7.88 (d, *J* = 7.8 Hz, 1H), 8.03 (d, *J* = 8.2 Hz, 1H). ^{13}C NMR ($CDCl_3$, 100.53 MHz) δ: 19.4, 124.1, 125.50, 125.54, 125.7, 126.3, 126.5, 128.5, 132.5, 133.4,

134.2. Exact Mass (EI) Calcd for $C_{11}H_{10}$: 142.07825. Found: 142.0783.

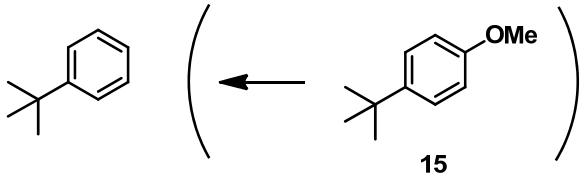
9-Methylanthracene [CAS: 779-02-2].



The typical procedure was followed using **16** as the substrate.

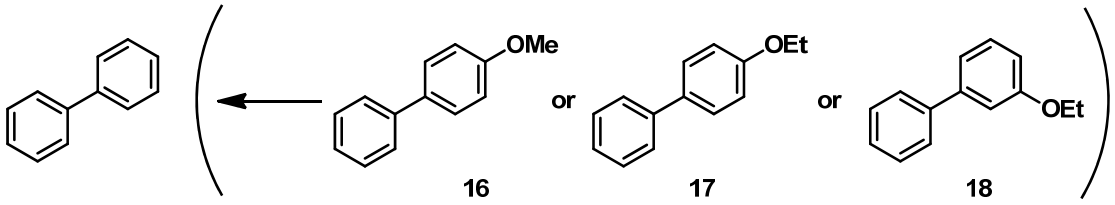
White solid (38 mg, 78%). R_f 0.60 (SiO_2, hexane/EtOAc = 20/1). ^{1}H NMR ($CDCl_3$, 399.78 MHz) δ: 3.12 (s, 3H), 7.50 (dd, *J* = 6.9, 8.2 Hz, 2H), 7.54 (dd, *J* = 6.9, 8.7 Hz, 2H), 8.03 (d, *J* = 8.2 Hz, 2H), 8.32 (d, *J* = 8.7 Hz, 2H), 8.36 (s, 1H). ^{13}C NMR ($CDCl_3$, 100.53 MHz) δ: 13.9, 124.7, 124.8, 125.20, 125.25, 129.0, 130.0, 130.1, 131.4. Exact Mass (EI) Calcd for $C_{15}H_{12}$: 192.09390. Found: 192.0940.

tert-Butylbenzene [CAS: 98-06-6].



The typical procedure was followed using **15** as the substrate. The yield was determined by GC analysis using undecane as an internal substance due to volatility of the product (27%).

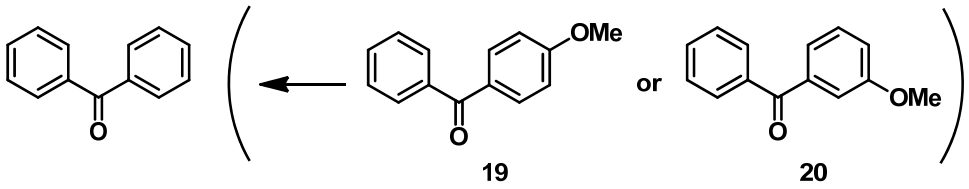
Biphenyl [CAS: 92-52-4].



The product was obtained from either **16**, **17** or **18** under the typical conditions. The yield was determined by GC analysis using undecane as an internal substance due to sublimability of the product.

White solid. R_f 0.60 (SiO_2, hexane/EtOAc = 50/1). ^{1}H NMR ($CDCl_3$, 399.78 MHz) δ: 7.37 (d, *J* = 7.3, 2H), 7.47 (dd, *J* = 7.3, 7.9 Hz, 4H), 7.62 (d, *J* = 7.9 Hz, 4H). ^{13}C NMR ($CDCl_3$, 100.53 MHz) δ: 127.1, 127.2, 128.7, 141.2. Exact Mass (EI) Calcd for $C_{12}H_{10}$: 154.0783. Found: 154.0783.

Benzophenone [CAS: 119-61-9].

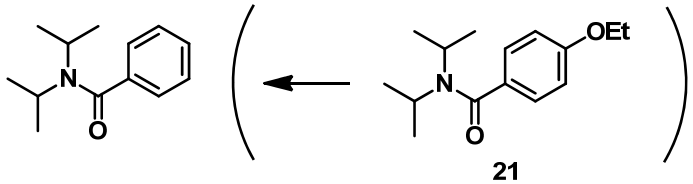


19 20

The product was obtained from either **19** or **20** under the typical conditions, except that NaO^{t}Bu (12 mg, 0.13 mmol) was used.

White solid. R_f 0.77 (NH Silica, hexane/EtOAc = 20/1). ^{1}H NMR ($CDCl_3$, 399.78 MHz) δ: 7.48 (dd, *J* = 6.9, 7.8 Hz, 4H), 7.60 (dd, *J* = 7.8, 7.8 Hz, 2H), 7.80 (d, *J* = 6.9 Hz, 4H). ^{13}C NMR ($CDCl_3$, 100.53 MHz) δ: 128.2, 130.0, 132.4, 137.5, 196.7. Exact Mass (EI) Calcd for $C_{13}H_{10}O$: 182.0732. Found: 182.0730.

N,N-Diisopropylbenzamide [CAS: 20383-28-2].

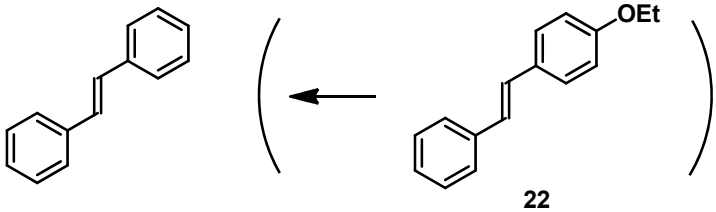


21

The typical procedure was followed using **21** as the substrate.

White solid (34 mg, 67%). R_f 0.34 (NH Silica, hexane/EtOAc = 5/1). ^{1}H NMR ($CDCl_3$, 399.78 MHz) δ: 1.53-1.14 (m, 12H), 3.82-3.52 (m, 2H), 7.30 (dd, *J* = 5.5, 6.9 Hz, 2H), 7.38-7.34 (m, 3H). ^{13}C NMR ($CDCl_3$, 100.53 MHz) δ: 20.7, 45.7 (br), 50.8 (br), 125.5, 128.4, 128.6, 138.9, 171.0. Exact Mass (EI) Calcd for $C_{13}H_{19}NO$: 205.1467. Found: 205.1468.

(E)-1,2-Diphenylethene [CAS: 103-30-0].

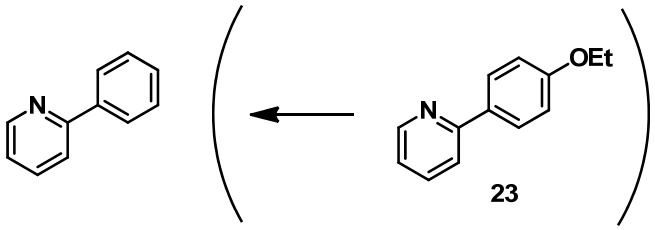


22

The typical procedure was followed using **22** as the substrate.

White solid (35 mg, 78%). R_f 0.69 (SiO_2, hexane/EtOAc = 20/1). ^{1}H NMR ($CDCl_3$, 399.78 MHz) δ: 7.12 (s, 2H), 7.26 (dd, *J* = 8.4 Hz, 2H), 7.37 (dd, *J* = 7.3, 8.4 Hz, 4H), 7.52 (d, *J* = 7.3 Hz, 4H). ^{13}C NMR ($CDCl_3$, 100.53 MHz) δ: 126.5, 127.6, 128.65, 128.66, 137.3. Exact Mass (EI) Calcd for $C_{14}H_{12}$: 180.0939. Found: 180.0980.

2-Phenylpyridine [CAS: 1008-89-5].



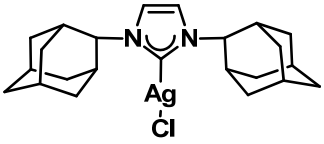
23

The typical procedure was followed using **23** as the substrate.

White solid (29 mg, 75%). R_f 0.14 (SiO_2, hexane/EtOAc = 20/1). ^1H NMR ($CDCl_3$, 399.78 MHz) δ: 7.21-7.23 (m, 1H), 7.43 (d, *J* = 6.0 Hz, 1H), 7.49 (dd, *J* = 6.0, 8.2 Hz, 2H), 7.78-7.73 (m, 2H), 8.00 (d, *J* = 8.2 Hz, 2H), 8.71 (d, *J* = 5.2 Hz, 1H). ^{13}C NMR ($CDCl_3$, 100.53 MHz) δ: 120.6, 122.1, 126.9, 128.8, 129.0, 136.9, 139.2, 149.6, 157.4. Exact Mass (EI) Calcd for $C_{11}H_9N$: 155.0735. Found: 155.0737.

XI. Determination of Tolman Electronic Parameter and Buried Volume of [Ir(I(2-Ad))(CO)$_2$Cl]

Chloro[1,3-bis(adamantan-2-yl)imidazolin-2-yliden]silver (I) ([I(2-Ad)]AgCl).[24]

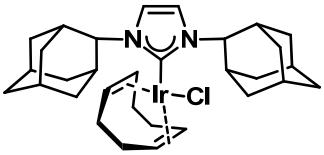


1,3-Bis(adamantan-2-yl)imidazolin-2-ylidene chloride [I(2-Ad)·HCl, 310 mg, 0.80 mmol] and Ag_2O (220 mg, 0.95 mmol) were dissolved in CH_2Cl_2 (45 mL). The reaction mixture was refluxed under an atmosphere of nitrogen for 12 h, and then filtered through a Celite pad; the filtrate volume was reduced to about 2 mL by evaporation. The reaction mixture was cooled to 0 °C, and then hexane was slowly added to this solution until white precipitate appeared. The solid was filtered, washed with cold hexane, and dried *in vacuo* to give title compound as a white solid (340 mg, 86%). This compound was used for the subsequent reaction without further purification.

White solid (340 mg, 86%). R_f 0.00 (SiO_2, CH_2Cl_2). Mp: 252−255 °C. ^1H NMR ($CDCl_3$, 399.78 MHz) δ: 1.74-1.70 (m, 4H), 1.80 (s, 4H), 2.00-1.87 (m, 16H), 2.70 (s, 4H), 4.43 (s, 2H), 7.31 (s, 2H). ^{13}C NMR ($CDCl_3$, 100.53 MHz) δ: 27.01, 27.07, 31.4, 33.3, 37.3, 37.9, 65.4, 118.3. IR (ATR): 2915 s, 2888 s, 2853 m, 2361 m, 2336 m, 1554 w, 1467 w, 1451 m, 1425 w, 1382 w, 1353 w, 1306 w, 1232 w, 1200 m, 1172 w, 1127 w, 1102 w, 1071 w, 1054 w, 1028 w, 980 w, 956 w, 907 w, 822 w, 792 w, 767 w, 719 s, 668 w. Exact Mass (FAB^+) Calcd for $C_{23}H_{32}N_2ClAg$ (M-HCl): 443.1616. Found: 443.1617.

[24] Frémont, P. D.; Scott, N. M.; Stevens, E. D.; Ramnial, T.; Lightbody, O. C.; Macdonald, C. L. B.; Clyburne, J. A. C.; Abernethy, C. D.; Nolan, S. P. *Organometallics*, **2005**, *24*, 6301.

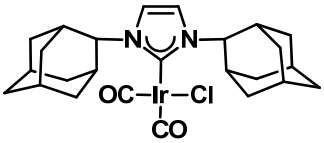
Chloro(η^4-1,5-cyclooctadiene)[1,3-bis(adamantan-2-yl)-imidazolin-2-ylidene]iridium(I) ([I(2-Ad)]Ir(COD)Cl).[25]



[I(2-Ad)]AgCl (230 mg, 0.47 mmol) and $[Ir(COD)Cl]_2$ (160 mg, 0.24 mmol) were dissolved in CH_2Cl_2 (13 mL). The reaction mixture was stirred under an atmosphere of nitrogen for 2.5 h, and then filtered through a Celite pad; the filtrate volume was reduced to about 1 mL by evaporation. The reaction mixture was cooled to 0 °C, and then Et_2O was slowly added to the solution until a white precipitate appeared. The solid was filtered, and the residue was concentrated *in vacuo* to give the title compound as a yellow powder (310 mg, 94%). This compound was used for the subsequent reaction without further purification.

Yellow powder (310 mg, 94%). R_f 0.66 (SiO_2, CH_2Cl_2). ^{1}H NMR ($CDCl_3$, 399.78 MHz) δ: 1.80-1.56 (m, 12H), 2.08-1.93 (m, 14H), 2.18-2.15 (m, 6H), 2.32 (br, 2H), 2.78 (br, 2H), 2.91-2.90 (m, 2H), 4.55 (t, J = 2.8 Hz, 2H), 5.67 (br, 2H), 7.30 (s, 2H). ^{13}C NMR ($CDCl_3$, 100.53 MHz) δ: 27.1, 27.6, 29.7, 30.9, 32.3, 33.40, 33.48, 33.90, 38.0, 38.8, 39.4, 50.6, 65.2, 81.8, 118.7, 181.1. IR (ATR): 2906 s, 2850 m, 2825 w, 2360 s, 2339 w, 2057 w, 2975 w, 1736 w, 1650 w, 1554 w, 1541 w, 1517 w, 1474 w, 1455 w, 1432 w, 1404 w, 1353 w, 1336 w, 1246 w, 1215 w, 1192 m, 1098 w, 1068 w, 1026 w, 959 w, 904 w, 883 w, 864 w, 810 m, 708 s, 669 m. Exact Mass (FAB^+) Calcd for $C_{31}H_{44}N_2ClIr$: 672.2822. Found: 672.2794.

Dicarbonylchloro[1,3-bis(adamantan-2-yl)-imidazolin-2-ylidene]iridium(I) [I(2-Ad)]Ir(CO)$_2$Cl[26]



A CH_2Cl_2 (5 mL) solution of [I(2-Ad)]Ir(COD)Cl (220 mg, 0.33 mmol) was placed under 1 atm of CO. The reaction mixture was stirred for 2 h. The color of the solution was changed from yellow-brown to pale yellow. The solvent was removed under reduced pressure. Hexane (5 mL) was added, and the collected precipitate was washed with Et_2O (0.5 mL) and pentane (2 mL X 3), and dried under vacuum to give the title compound as a yellow powder (170 mg, 84%).

Yellow powder (170 mg, 84%). R_f 0.83 (SiO_2, CH_2Cl_2). ^{1}H NMR ($CDCl_3$, 399.78 MHz) δ: 1.81-1.71

[25] Frey,G. D.; Rentzsch,C. F.; Preysing, D. V.; Scherg, T.; Mu¨hlhofer, M.; Herdtweck, E.; Herrmann, W. *J. Organomet. Chem.*, **2006**, *691*, 5725.

[26] Kelly、R. E. III.; Clavier, H.; Giudice, S.; Scott, N. M.; Stevens, E. D.; Bordner, J.; Samardjiev, I.; Hoff, C. D.; Cavallo, L.; Nolan, S. P. *Organometallic*s, **2008**, *27*, 202.

(m, 8H), 2.05-1.93 (m, 16H), 2.71 (s, 4H), 5.11 (s, 2H), 7.40 (s, 2H). ^{13}C NMR ($CDCl_3$, 100.53 MHz) δ: 27.1, 27.3, 31.46, 31.51, 33.1, 38.0, 38.2, 38.3, 65.5, 119.3, 168.1, 174.6, 181.1. IR (ATR): 3155 w, 3014 w, 2970 w, 2911 m, 2855 m, 2361 w, 2339 w, 2055 s, 1964 s, 1739 s, 1561 w, 1542 w, 1452 m, 1416 m, 1366 s, 1215 s, 1173 m, 1097 w, 1058 w, 1028 w, 982 w, 958 w, 904 w, 810 w, 727 m, 703 m, 667 w. Exact Mass (FAB^+) Calcd for $C_{25}H_{32}O_2N_2ClIr$: 620.1782. Found: 620.1776.

The infrared carbonyl stretching frequencies of [Ir(I(2-Ad))$(CO)_2$Cl] (ν_{co}^{Ir}) in CH_2Cl_2 appeared at 2062.50 and 1980.54 cm^{-1}. The average of the frequencies for the iridium complex ($\nu_{co}^{av/Ir}$) is therefore 2021.52. The TEP value for I(2-Ad) was determined to be 2049.4, using the following equation:[27]

$$TEP = 0.8475\ \nu_{co}^{av/Ir} + 336.2$$

The buried volume, $\%V_{bur}$, was calculated with SambV_{ca}, using the crystal structures of [Ir(I(2-Ad))(cod)Cl] (data are shown in the next section), [Ir(I(1-Ad)(cod)Cl] (CSD Refcode: QIWMEP), [Ir(ICy)(cod)Cl] (CSD Refcode: QIWLVE), [Ir(IMes)(cod)Cl] (CSD Refcode: CIVNOL), [Ir(IPr)(cod)Cl] (CSD Refcode: QIWMAL), and [Ir(PCy_3)(COD)(pyridine)PF_6] (CSD Refcode: JAFFOL) deposited with the Cambridge Structural Database (CSD). The parameters used for the SambV_{ca} calculations were as follows:[28] 3.5 Å was selected as the value for the sphere radius, 2.00 Å was used as the distance between the coordinated atom center of the ligand and the metal atom center, hydrogen atoms were included, and Bondi radii scaled by 1.17 were used. These parameters applied are identical to those in literature examples[29] allowing a direct comparison with the calculated values.

XII. X-Ray Crystallographic Structure Analysis of [Ir(I(2-Ad))$(CO)_2$Cl]

[27] a) Kelly Iii, R. A.; Clavier, H.; Giudice, S.; Scott, N. M.; Stevens, E. D.; Bordner, J.; Samardjiev, I.; Hoff, C. D.; Cavallo, L.; Nolan, S. P. *Organometallics* **2008**, *27*, 202. b) Dröge, T.; Glorius, F. *Angew. Chem., Int. Ed.* **2010**, *49*, 6940.

[28] Poater, A.; Cosenza, B.; Correa, A.; Giudice, S.; Ragone, F.; Scarano, V.; Cavallo, L. *Eur. J. Inorg. Chem.* **2009**, *2009*, 1759.

[29] (a) dunsford, J. J.; Tromp, D. S.; Cavell, K. J.; Elsevier, C. J.; Mariuki, B. N. *Dalton Trans.*, **2013**, *42*, 7318. (b) Weerdenburg, B. J. A.; Glögger, S.; Eshuis, N.; Engwerda, A. H. J. (T).; Smits, J. M. M.; Gelder, R.; Appelt, S.; wymenga, S. S.; Tessari, M.; Feiters, M.; Blümich, B.; Rutjes, F. P. J. *Chem. Commun.*, **2013**, *49*, 7388.

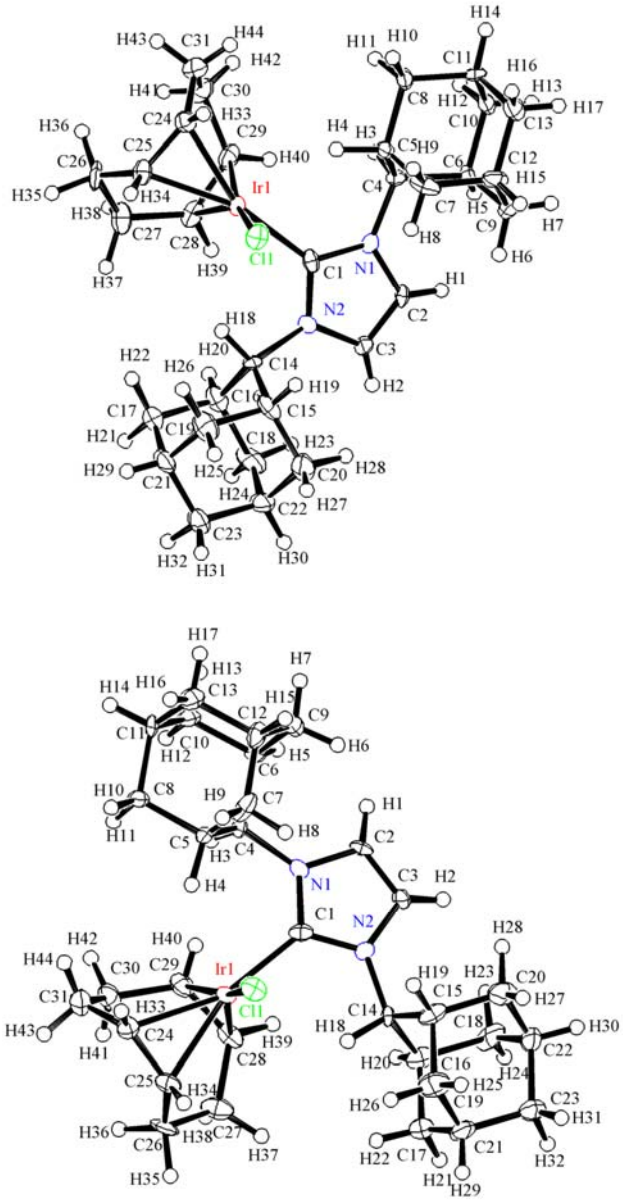


Table S1. Crystal Data and structure refinement for compound [Ir(I(2-Ad))$(CO)_2$Cl]

Empirical Formula	$C_{31}H_{44}N_2ClIr$
Formula Weight	672.37
Crystal Color, Habit	yellow, platelet
Crystal Dimensions	0.150 X 0.100 X 0.010 mm
Crystal System	monoclinic
Lattice Type	Primitive
Lattice Parameters	a = 10.4452(2) Å
	b = 7.3064(2) Å
	c = 34.6675(7) Å

	β = 91.7266(7) ^{o}
	V = 2644.52(9) Å^3
Space Group	$P2_1/n$ (#14)
Z value	4
D_{calc}	1.689 g/cm^3
F000	1352.00
μ(CuKα)	106.429 cm^{-1}
Diffractometer	R-AXIS RAPID
Radiation	CuKα (λ = 1.54187 Å)
	graphite monochromated
Voltage, Current	50kV, 100mA
Temperature	-150.0^{o}C
Detector Aperture	460 x 256 mm
Data Images	60 exposures
ω oscillation Range (χ=54.0, ϕ=0.0)	80.0 - 260.0^{o}
Exposure Rate	135.0 sec./^{o}
ω oscillation Range (χ=54.0, ϕ=60.0)	80.0 - 260.0^{o}
Exposure Rate	135.0 sec./^{o}
ω oscillation Range (χ=54.0, ϕ=120.0)	80.0 - 260.0^{o}
Exposure Rate	135.0 sec./^{o}
ω oscillation Range (χ=54.0, ϕ=180.0)	80.0 - 260.0^{o}
Exposure Rate	135.0 sec./^{o}
ω oscillation Range (χ=54.0, ϕ=240.0)	80.0 - 260.0^{o}
Exposure Rate	135.0 sec./^{o}
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{max}$	136.4^{o}
No. of Reflections Measured	Total: 27352
	Unique: 4831 (R_{int} = 0.0891)
Corrections	Lorentz-polarization
	Absorption
	(trans. factors: 0.704 - 0.899)

Table S2. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
Ir1	Cl1	2.3744(14)	Ir1	C1	2.062(6)
Ir1	C24	2.173(7)	Ir1	C25	2.182(7)
Ir1	C28	2.101(7)	Ir1	C29	2.131(7)
N1	C1	1.364(8)	N1	C2	1.391(8)
N1	C4	1.494(8)	N2	C1	1.363(8)
N2	C3	1.385(8)	N2	C14	1.491(8)
C2	C3	1.333(9)	C4	C5	1.533(9)
C4	C6	1.538(9)	C5	C7	1.537(10)
C5	C8	1.543(9)	C6	C9	1.533(9)
C6	C10	1.562(9)	C7	C12	1.510(10)
C8	C11	1.532(9)	C9	C12	1.528(10)
C10	C11	1.521(9)	C11	C13	1.529(10)
C12	C13	1.530(10)	C14	C15	1.528(9)
C14	C16	1.534(9)	C15	C19	1.532(10)
C15	C20	1.531(11)	C16	C17	1.539(10)
C16	C18	1.545(10)	C17	C21	1.518(11)
C18	C22	1.516(11)	C19	C21	1.526(11)
C20	C22	1.534(11)	C21	C23	1.526(10)
C22	C23	1.532(11)	C24	C25	1.403(10)
C24	C31	1.511(10)	C25	C26	1.526(10)
C26	C27	1.529(11)	C27	C28	1.506(11)
C28	C29	1.430(10)	C29	C30	1.510(10)
C30	C31	1.543(11)			

Table S3. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C2	H1	0.91(6)	C3	H2	0.87(6)
C4	H3	0.95(6)	C5	H4	1.05(7)
C6	H5	0.95(6)	C7	H8	0.97(8)
C7	H9	0.93(6)	C8	H10	0.96(6)
C8	H11	1.02(6)	C9	H6	0.98(6)
C9	H7	0.94(7)	C10	H12	1.044(7)
C10	H13	0.99(7)	C11	H14	0.96(6)

C12	H15	0.98(6)	C13	H16	0.92(7)
C13	H17	0.97(6)	C14	H18	1.000
C15	H19	0.95(6)	C16	H20	0.94(7)
C17	H21	1.08(7)	C17	H22	0.97(7)
C18	H23	1.02(7)	C18	H24	1.124(7)
C19	H25	1.01(7)	C19	H26	0.98(8)
C20	H27	0.92(7)	C20	H28	1.03(7)
C21	H29	0.90(6)	C22	H30	0.98(7)
C23	H31	1.01(7)	C23	H32	1.04(6)
C24	H33	0.92(6)	C25	H34	0.94(6)
C26	H35	1.05(6)	C26	H36	1.00(6)
C27	H37	1.09(9)	C27	H38	0.78(7)
C28	H39	0.99(6)	C29	H40	0.88(6)
C30	H41	0.88(8)	C30	H42	1.01(7)
C31	H43	0.99(6)	C31	H44	0.93(7)

Table S4. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
Cl1	Ir1	C1	90.73(17)	Cl1	Ir1	C24	91.00(17)
Cl1	Ir1	C25	90.98(18)	Cl1	Ir1	C28	153.78(18)
Cl1	Ir1	C29	166.29(18)	C1	Ir1	C24	159.6(3)
C1	Ir1	C25	162.7(3)	C1	Ir1	C28	90.2(3)
C1	Ir1	C29	92.9(3)	C24	Ir1	C25	37.6(3)
C24	Ir1	C28	97.2(3)	C24	Ir1	C29	81.0(3)
C25	Ir1	C28	80.8(3)	C25	Ir1	C29	89.5(3)
C28	Ir1	C29	39.5(3)	C1	N1	C2	109.2(5)
C1	N1	C4	124.5(5)	C2	N1	C4	126.2(5)
C1	N2	C3	109.7(5)	C1	N2	C14	121.6(5)
C3	N2	C14	128.4(5)	Ir1	C1	N1	129.4(5)
Ir1	C1	N2	124.7(5)	N1	C1	N2	105.7(5)
N1	C2	C3	107.8(6)	N2	C3	C2	107.5(6)
N1	C4	C5	111.0(5)	N1	C4	C6	111.9(5)
C5	C4	C6	108.8(5)	C4	C5	C7	110.7(5)
C4	C5	C8	108.2(6)	C7	C5	C8	109.8(6)

C4	C6	C9	112.8(6)	C4	C6	C10	106.6(5)
C9	C6	C10	108.1(5)	C5	C7	C12	109.9(6)
C5	C8	C11	109.0(5)	C6	C9	C12	109.6(6)
C6	C10	C11	109.6(5)	C8	C11	C10	109.3(5)
C8	C11	C13	109.2(6)	C10	C11	C13	110.4(5)
C7	C12	C9	109.6(6)	C7	C12	C13	110.3(6)
C9	C12	C13	109.2(6)	C11	C13	C12	109.3(6)
N2	C14	C15	113.5(5)	N2	C14	C16	115.0(5)
C15	C14	C16	109.5(5)	C14	C15	C19	105.7(6)
C14	C15	C20	113.1(6)	C19	C15	C20	108.4(6)
C14	C16	C17	105.9(6)	C14	C16	C18	113.6(6)
C17	C16	C18	107.9(6)	C16	C17	C21	109.9(6)
C16	C18	C22	109.2(6)	C15	C19	C21	110.1(6)
C15	C20	C22	109.8(6)	C17	C21	C19	110.3(6)
C17	C21	C23	109.0(6)	C19	C21	C23	109.3(7)
C18	C22	C20	109.7(6)	C18	C22	C23	109.9(6)
C20	C22	C23	109.7(6)	C21	C23	C22	108.9(6)
Ir1	C24	C25	71.6(4)	Ir1	C24	C31	109.6(5)
C25	C24	C31	125.9(6)	Ir1	C25	C24	70.9(4)
Ir1	C25	C26	112.7(5)	C24	C25	C26	121.9(6)
C25	C26	C27	110.6(6)	C26	C27	C28	112.7(6)
Ir1	C28	C27	111.5(5)	Ir1	C28	C29	71.4(4)
C27	C28	C29	125.8(6)	Ir1	C29	C28	69.1(4)
Ir1	C29	C30	114.8(5)	C28	C29	C30	124.4(6)
C29	C30	C31	111.9(6)	C24	C31	C30	113.5(6)

Table S5. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
N1	C2	H1	118(4)	C3	C2	H1	134(4)
N2	C3	H2	126(4)	C2	C3	H2	127(4)
N1	C4	H3	108(4)	C5	C4	H3	109(4)
C6	C4	H3	108(4)	C4	C5	H4	108(4)
C7	C5	H4	115(4)	C8	C5	H4	105(4)
C4	C6	H5	109(4)	C9	C6	H5	112(4)

C10	C6	H5	108(4)	C5	C7	H8	111(5)
C5	C7	H9	105(4)	C12	C7	H8	114(5)
C12	C7	H9	115(4)	H8	C7	H9	101(6)
C5	C8	H10	111(4)	C5	C8	H11	105(4)
C11	C8	H10	115(4)	C11	C8	H11	115(4)
H10	C8	H11	102(5)	C6	C9	H6	108(4)
C6	C9	H7	108(4)	C12	C9	H6	114(4)
C12	C9	H7	110(4)	H6	C9	H7	107(6)
C6	C10	H12	104.2(5)	C6	C10	H13	106(4)
C11	C10	H12	115.8(6)	C11	C10	H13	114(4)
H12	C10	H13	107(4)	C8	C11	H14	106(4)
C10	C11	H14	111(4)	C13	C11	H14	111(4)
C7	C12	H15	109(4)	C9	C12	H15	106(4)
C13	C12	H15	113(4)	C11	C13	H16	115(4)
C11	C13	H17	106(4)	C12	C13	H16	111(4)
C12	C13	H17	109(4)	H16	C13	H17	107(6)
N2	C14	H18	106.0	C15	C14	H18	106.0
C16	C14	H18	106.0	C14	C15	H19	108(4)
C19	C15	H19	112(4)	C20	C15	H19	110(4)
C14	C16	H20	113(4)	C17	C16	H20	109(4)
C18	C16	H20	107(4)	C16	C17	H21	108(4)
C16	C17	H22	104(4)	C21	C17	H21	114(3)
C21	C17	H22	116(4)	H21	C17	H22	105(5)
C16	C18	H23	104(4)	C16	C18	H24	109.6(6)
C22	C18	H23	116(4)	C22	C18	H24	125.9(7)
H23	C18	H24	89(4)	C15	C19	H25	108(4)
C15	C19	H26	111(4)	C21	C19	H25	112(4)
C21	C19	H26	110(4)	H25	C19	H26	105(5)
C15	C20	H27	102(4)	C15	C20	H28	110(4)
C22	C20	H27	112(4)	C22	C20	H28	107(4)
H27	C20	H28	116(6)	C17	C21	H29	107(4)
C19	C21	H29	111(4)	C23	C21	H29	109(4)
C18	C22	H30	111(4)	C20	C22	H30	109(4)
C23	C22	H30	107(4)	C21	C23	H31	110(4)
C21	C23	H32	104(4)	C22	C23	H31	116(4)
C22	C23	H32	113(3)	H31	C23	H32	105(5)

Ir1	C24	H33	103(4)	C25	C24	H33	119(4)
C31	C24	H33	113(4)	Ir1	C25	H34	107(4)
C24	C25	H34	117(4)	C26	C25	H34	117(4)
C25	C26	H35	110(4)	C25	C26	H36	111(4)
C27	C26	H35	110(3)	C27	C26	H36	116(4)
H35	C26	H36	99(5)	C26	C27	H37	110(5)
C26	C27	H38	110(5)	C28	C27	H37	111(5)
C28	C27	H38	104(6)	H37	C27	H38	108(7)
Ir1	C28	H39	110(4)	C27	C28	H39	113(4)
C29	C28	H39	116(4)	Ir1	C29	H40	99(4)
C28	C29	H40	118(4)	C30	C29	H40	116(4)
C29	C30	H41	108(5)	C29	C30	H42	105(4)
C31	C30	H41	109(5)	C31	C30	H42	111(4)
H41	C30	H42	112(7)	C24	C31	H43	109(4)
C24	C31	H44	108(4)	C30	C31	H43	104(4)
C30	C31	H44	113(4)	H43	C31	H44	109(5)

Table S6. Torsion Angles(o)

(Those having bond angles > 160 or < 20 degrees are excluded.)

atom1	atom2	atom3	atom4	angle
Cl1	Ir1	C1	N1	-105.1(5)
Cl1	Ir1	C1	N2	80.7(5)
Cl1	Ir1	C24	C25	-90.3(3)
Cl1	Ir1	C24	C31	147.2(4)
Cl1	Ir1	C25	C24	90.4(3)
Cl1	Ir1	C25	C26	-152.1(4)
Cl1	Ir1	C28	C27	51.6(6)
Cl1	Ir1	C28	C29	173.64(17)
C1	Ir1	C24	C25	174.9(6)
C1	Ir1	C24	C31	52.4(9)
C24	Ir1	C1	N1	-10.3(10)
C24	Ir1	C1	N2	175.5(5)
C1	Ir1	C28	C27	143.5(4)

C1	Ir1	C28	C29	-94.4(3)
C28	Ir1	C1	N1	101.1(5)
C28	Ir1	C1	N2	-73.1(5)
C1	Ir1	C29	C28	86.7(3)
C1	Ir1	C29	C30	-153.9(4)
C29	Ir1	C1	N1	61.7(5)
C29	Ir1	C1	N2	-112.5(5)
C24	Ir1	C25	C24	0.0(3)
C24	Ir1	C25	C26	117.5(6)
C25	Ir1	C24	C25	0.0(3)
C25	Ir1	C24	C31	-122.5(6)
C24	Ir1	C28	C27	-55.6(4)
C24	Ir1	C28	C29	66.5(3)
C28	Ir1	C24	C25	64.7(3)
C28	Ir1	C24	C31	-57.8(4)
C24	Ir1	C29	C28	-112.9(3)
C24	Ir1	C29	C30	6.4(4)
C29	Ir1	C24	C25	100.9(3)
C29	Ir1	C24	C31	-21.6(4)
C25	Ir1	C28	C27	-21.6(4)
C25	Ir1	C28	C29	100.4(3)
C28	Ir1	C25	C24	-114.7(3)
C28	Ir1	C25	C26	2.8(4)
C25	Ir1	C29	C28	-76.1(3)
C25	Ir1	C29	C30	43.2(4)
C29	Ir1	C25	C24	-75.9(3)
C29	Ir1	C25	C26	41.6(4)
C28	Ir1	C29	C28	-0.0(3)
C28	Ir1	C29	C30	119.4(6)
C29	Ir1	C28	C27	-122.1(6)
C29	Ir1	C28	C29	-0.0(3)
C1	N1	C2	C3	-0.8(7)
C2	N1	C1	Ir1	-174.0(5)
C2	N1	C1	N2	1.0(6)
C1	N1	C4	C5	47.8(7)
C1	N1	C4	C6	169.5(5)

C4	N1	C1	Ir1	8.1(9)
C4	N1	C1	N2	-176.8(5)
C2	N1	C4	C5	-129.7(6)
C2	N1	C4	C6	-8.0(8)
C4	N1	C2	C3	177.0(5)
C1	N2	C3	C2	0.4(7)
C3	N2	C1	Ir1	174.5(5)
C3	N2	C1	N1	-0.9(6)
C1	N2	C14	C15	-111.1(6)
C1	N2	C14	C16	121.7(5)
C14	N2	C1	Ir1	-11.4(8)
C14	N2	C1	N1	173.2(5)
C3	N2	C14	C15	61.8(7)
C3	N2	C14	C16	-65.4(7)
C14	N2	C3	C2	-173.2(5)
N1	C2	C3	N2	0.3(7)
N1	C4	C5	C7	67.4(6)
N1	C4	C5	C8	-172.3(4)
N1	C4	C6	C9	-68.0(6)
N1	C4	C6	C10	173.5(4)
C5	C4	C6	C9	55.0(6)
C5	C4	C6	C10	-63.5(6)
C6	C4	C5	C7	-56.2(6)
C6	C4	C5	C8	64.1(6)
C4	C5	C7	C12	60.8(7)
C4	C5	C8	C11	-61.6(6)
C7	C5	C8	C11	59.3(7)
C8	C5	C7	C12	-58.5(7)
C4	C6	C9	C12	-56.6(6)
C4	C6	C10	C11	62.1(6)
C9	C6	C10	C11	-59.4(6)
C10	C6	C9	C12	61.0(6)
C5	C7	C12	C9	-61.3(7)
C5	C7	C12	C13	58.9(7)
C5	C8	C11	C10	60.3(6)
C5	C8	C11	C13	-60.6(6)

C6	C9	C12	C7	58.7(7)
C6	C9	C12	C13	-62.2(7)
C6	C10	C11	C8	-61.0(6)
C6	C10	C11	C13	59.1(6)
C8	C11	C13	C12	60.8(6)
C10	C11	C13	C12	-59.3(7)
C7	C12	C13	C11	-60.2(7)
C9	C12	C13	C11	60.2(7)
N2	C14	C15	C19	163.6(4)
N2	C14	C15	C20	-77.9(6)
N2	C14	C16	C17	-164.5(4)
N2	C14	C16	C18	77.3(6)
C15	C14	C16	C17	66.2(6)
C15	C14	C16	C18	-51.9(7)
C16	C14	C15	C19	-66.3(6)
C16	C14	C15	C20	52.1(7)
C14	C15	C19	C21	61.2(7)
C14	C15	C20	C22	-57.3(7)
C19	C15	C20	C22	59.7(7)
C20	C15	C19	C21	-60.3(7)
C14	C16	C17	C21	-60.9(7)
C14	C16	C18	C22	56.8(7)
C17	C16	C18	C22	-60.2(7)
C18	C16	C17	C21	61.0(7)
C16	C17	C21	C19	58.4(7)
C16	C17	C21	C23	-61.6(7)
C16	C18	C22	C20	-59.7(7)
C16	C18	C22	C23	60.9(7)
C15	C19	C21	C17	-58.7(8)
C15	C19	C21	C23	61.1(7)
C15	C20	C22	C18	60.5(7)
C15	C20	C22	C23	-60.2(7)
C17	C21	C23	C22	60.5(8)
C19	C21	C23	C22	-60.2(7)
C18	C22	C23	C21	-60.7(7)
C20	C22	C23	C21	60.0(8)

Ir1	C24	C25	Ir1	0.0
Ir1	C24	C25	C26	-105.4(5)
Ir1	C24	C31	C30	33.6(7)
C25	C24	C31	C30	-47.4(9)
C31	C24	C25	Ir1	101.2(7)
C31	C24	C25	C26	-4.1(10)
Ir1	C25	C26	C27	16.1(7)
C24	C25	C26	C27	97.0(7)
C25	C26	C27	C28	-34.8(9)
C26	C27	C28	Ir1	37.6(8)
C26	C27	C28	C29	-44.3(10)
Ir1	C28	C29	Ir1	-0.0
Ir1	C28	C29	C30	-106.4(6)
C27	C28	C29	Ir1	103.5(7)
C27	C28	C29	C30	-2.9(11)
Ir1	C29	C30	C31	9.9(8)
C28	C29	C30	C31	90.8(8)
C29	C30	C31	C24	-29.0(9)

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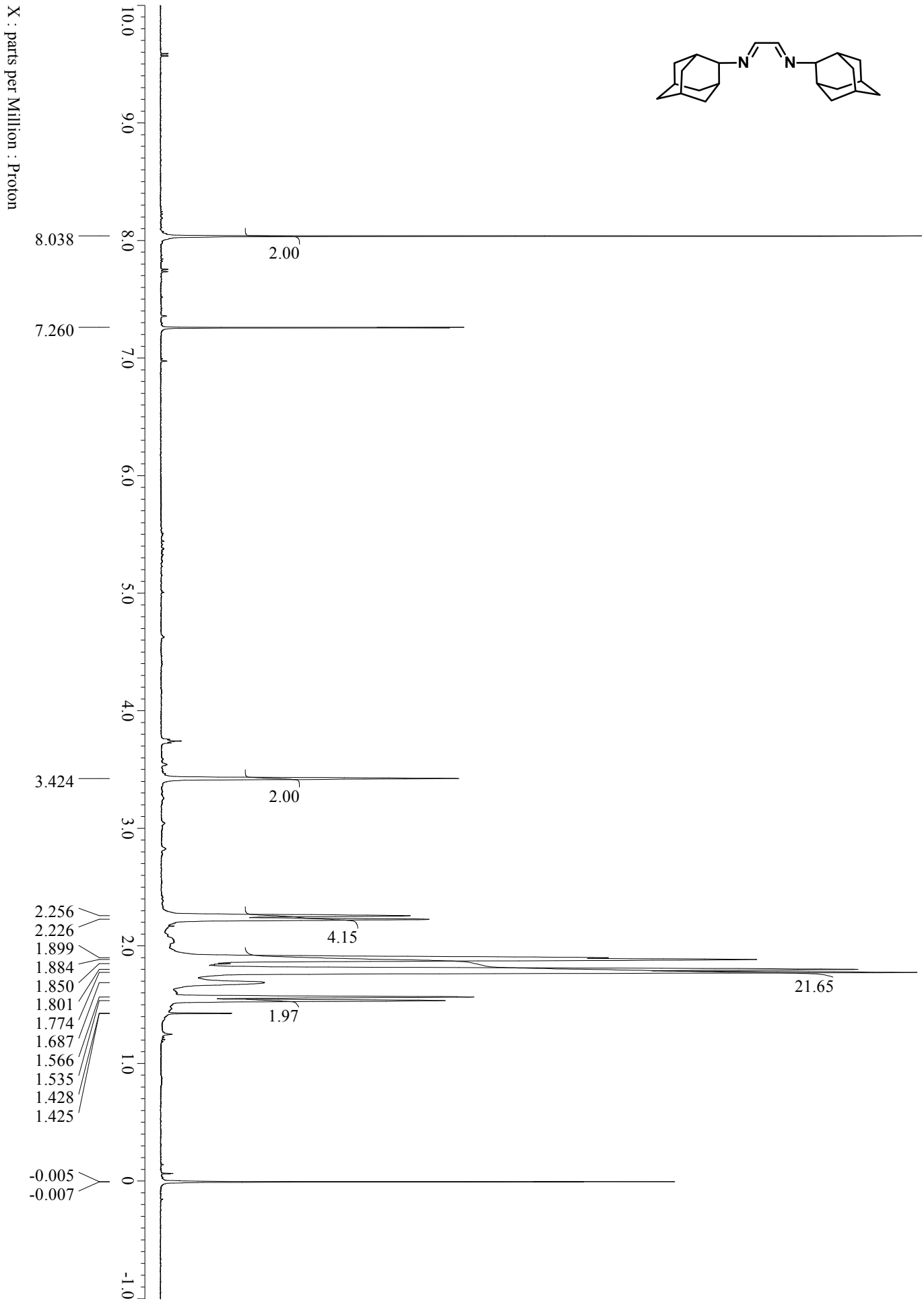
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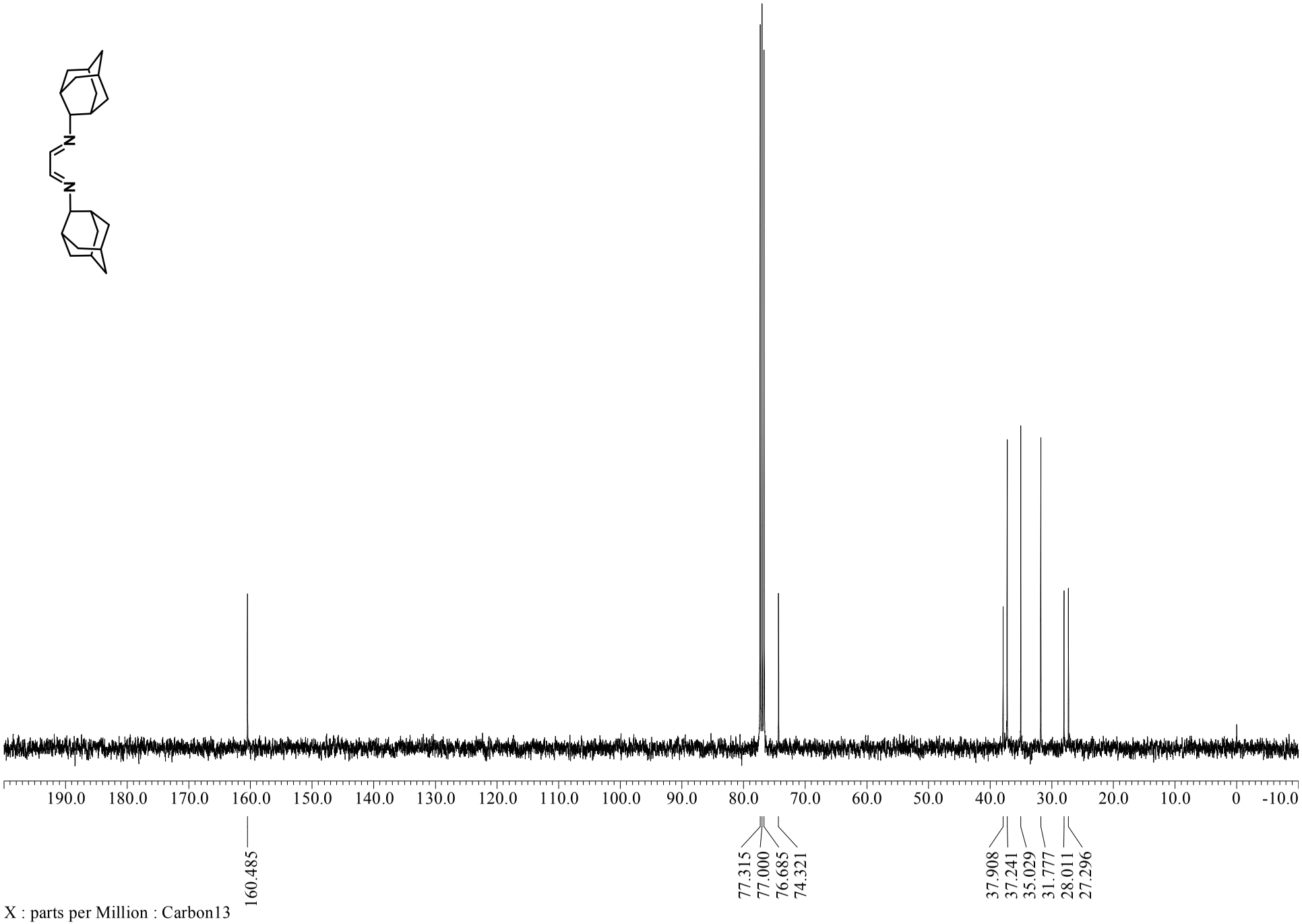
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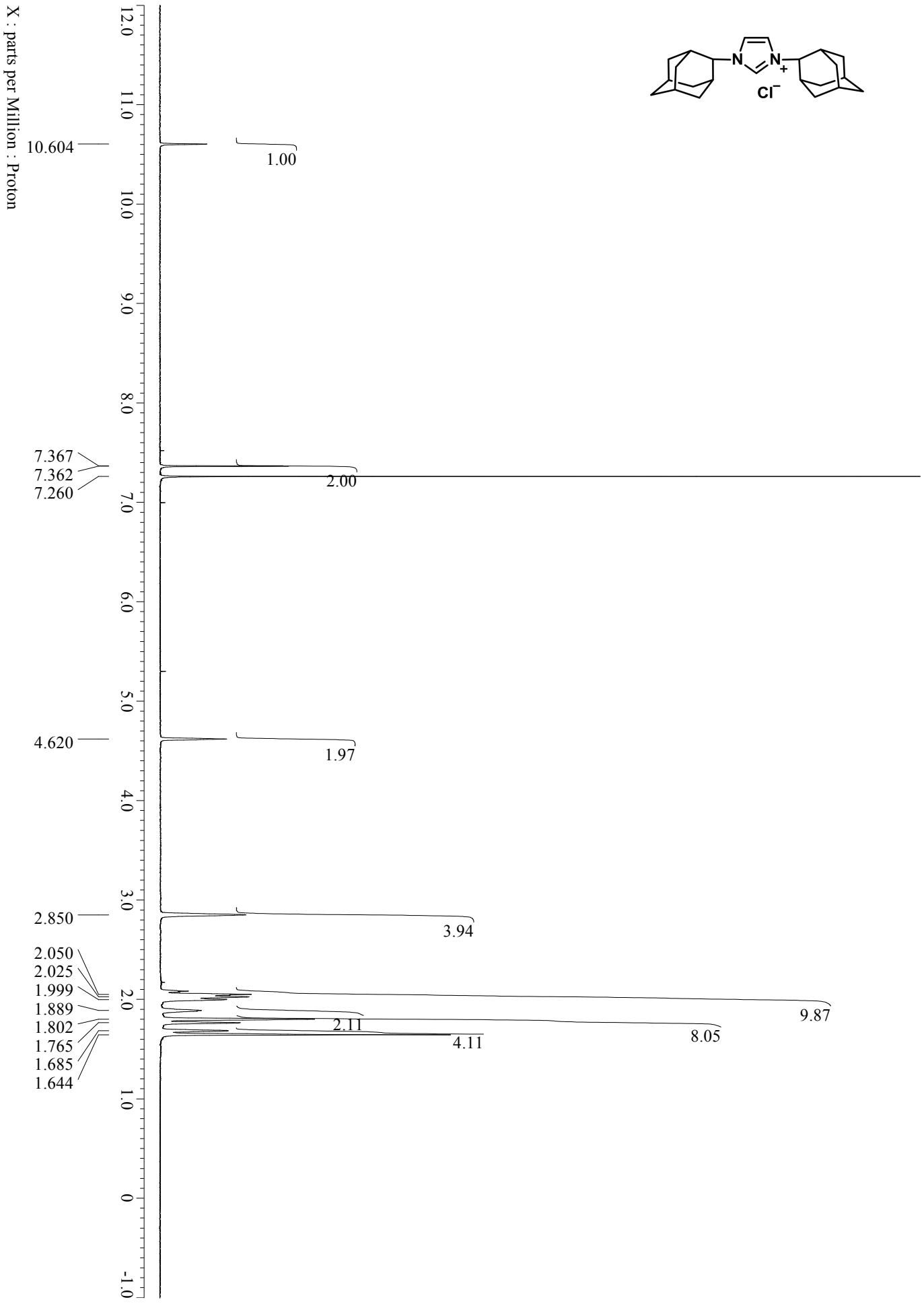
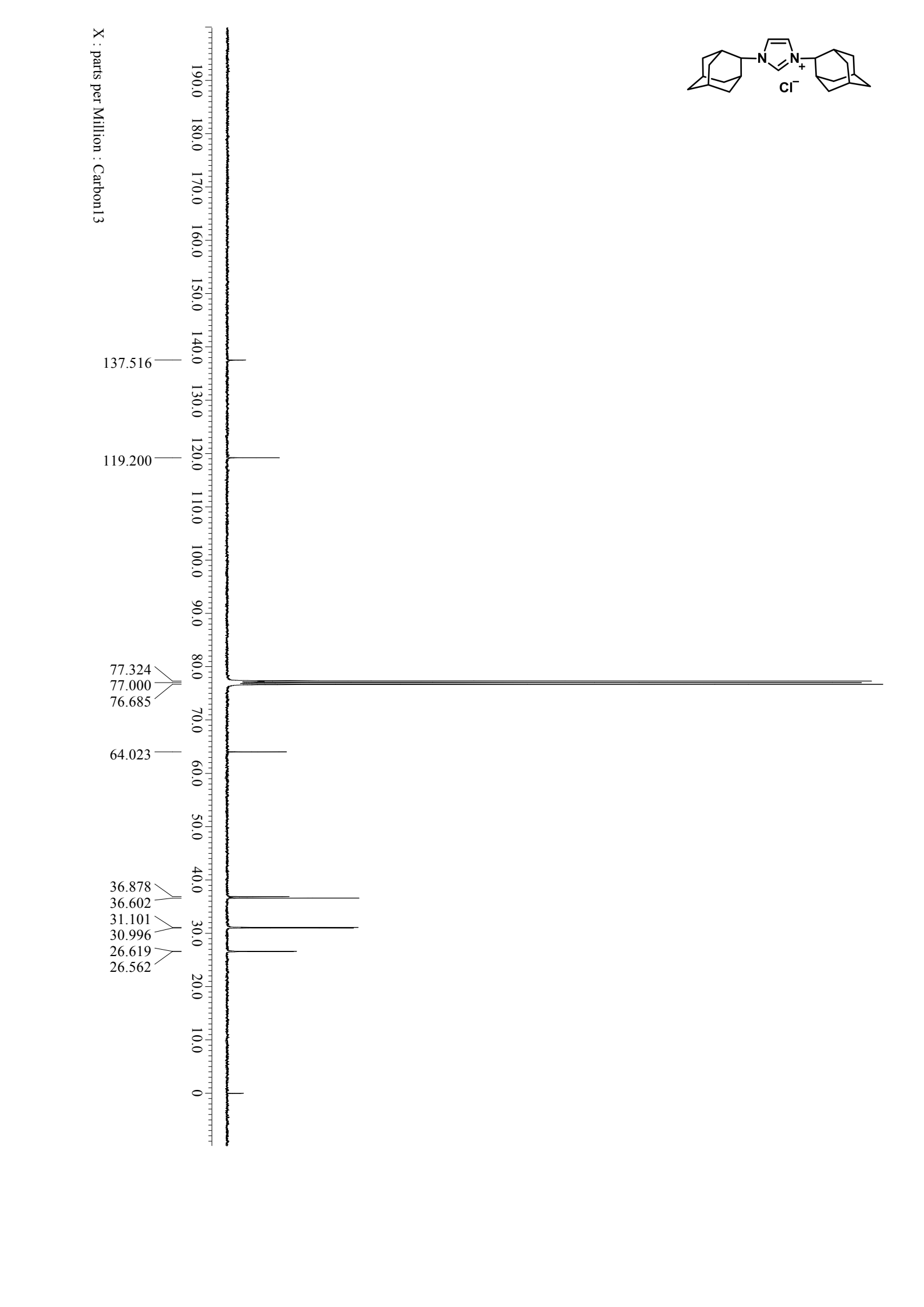
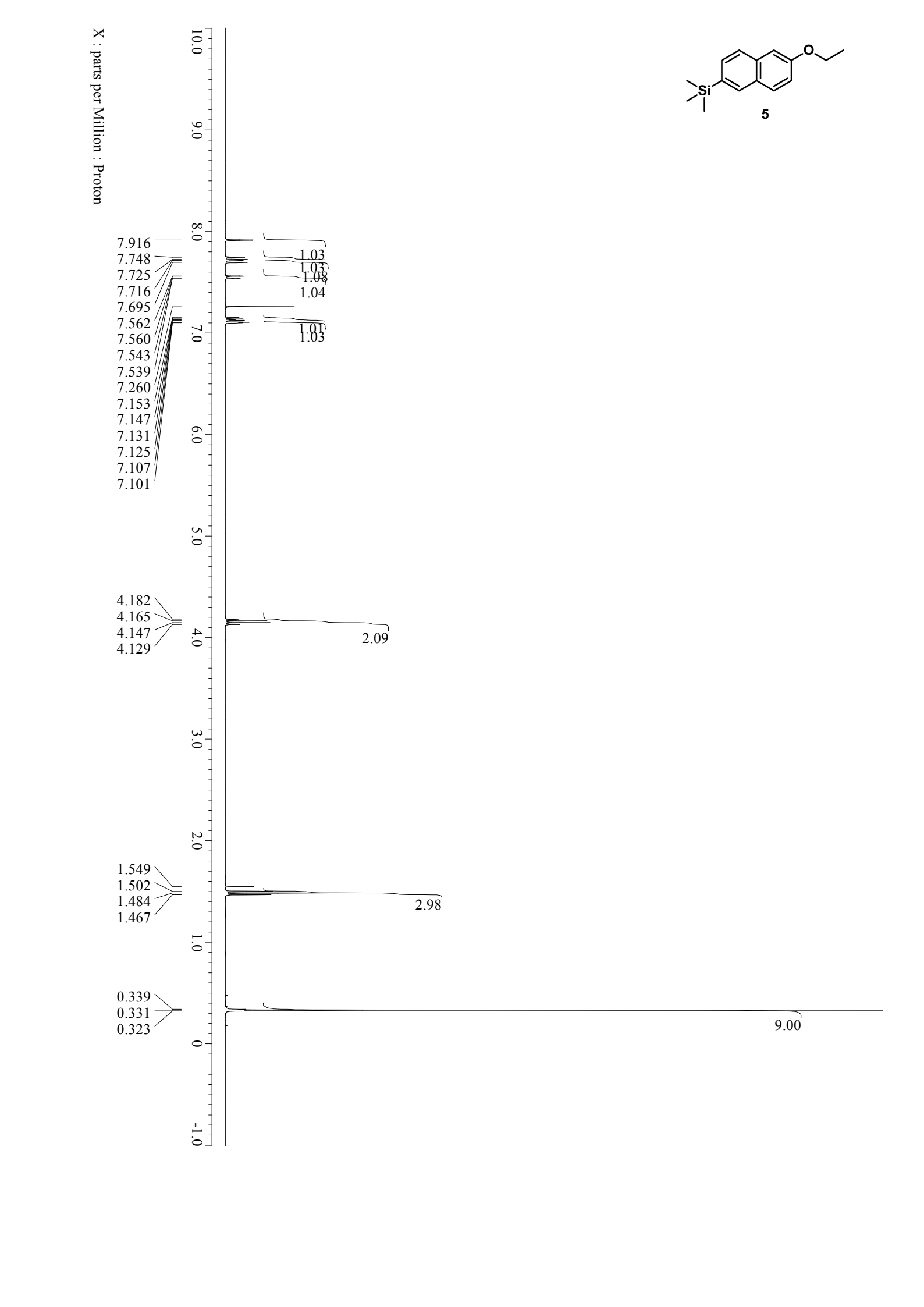
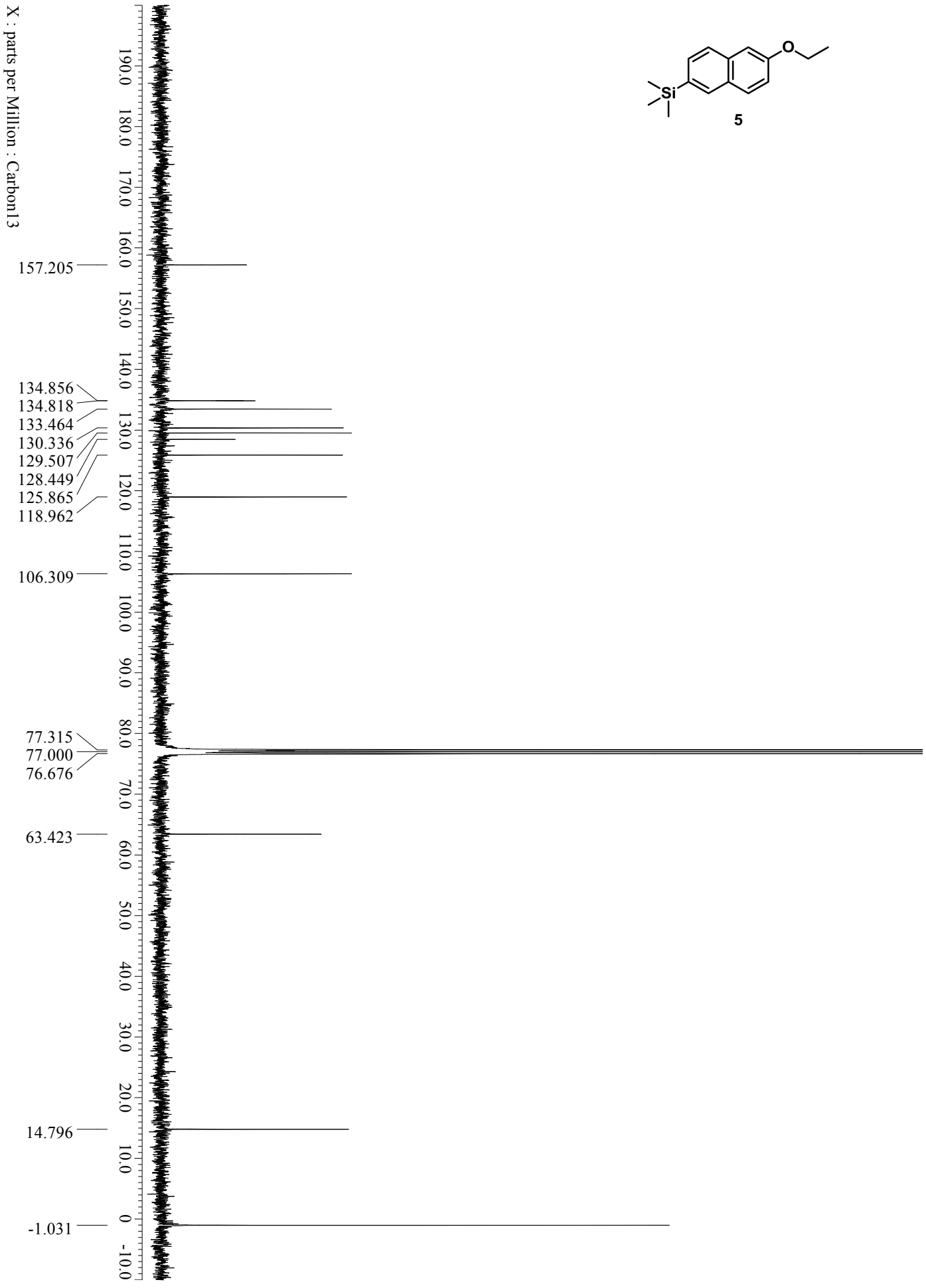
X : parts per Million : Carbon13

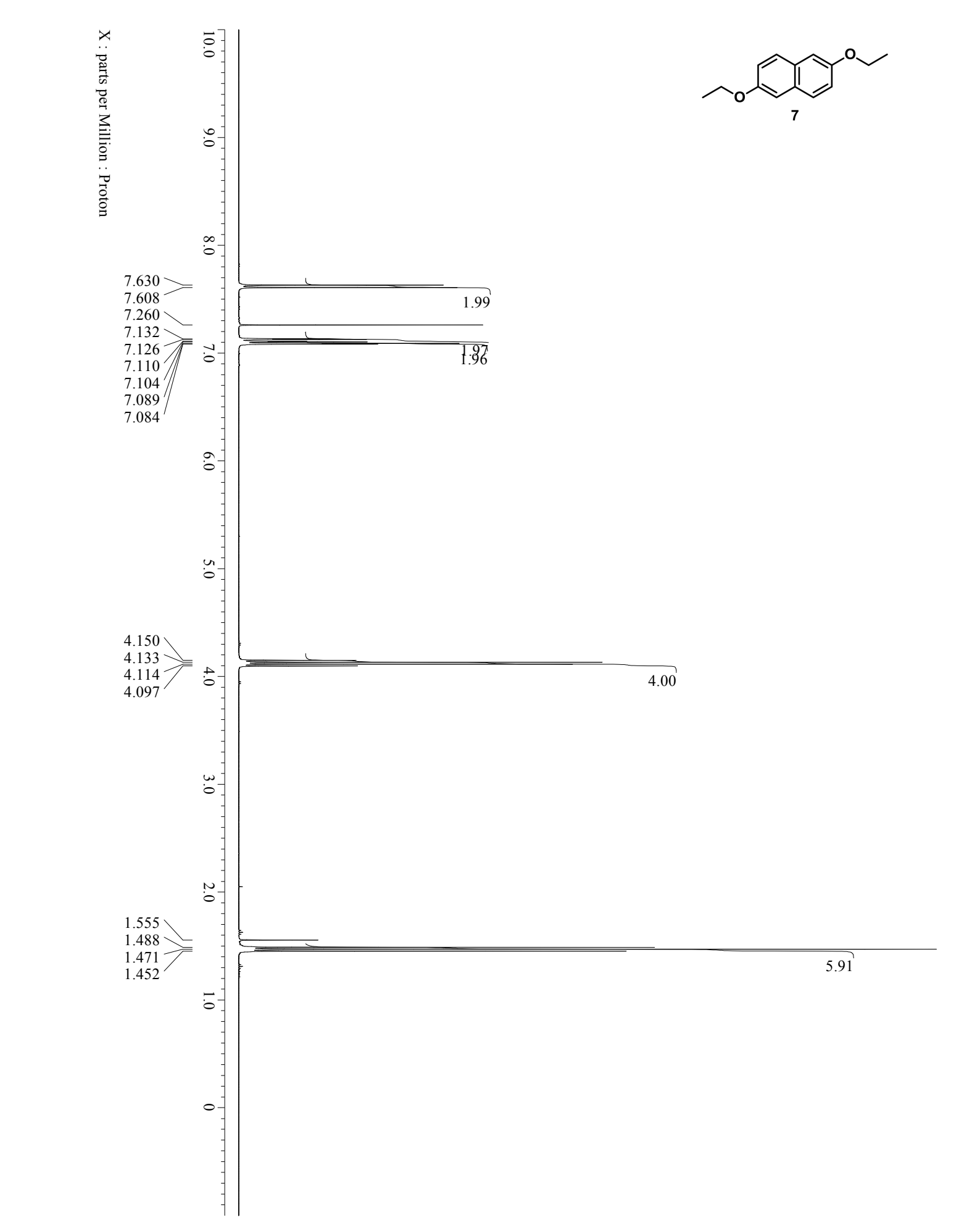

X : parts per Million : Proton
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
10.604
1.00
7.367
7.362
7.260
2.00
4.620
1.97
2.850
3.94
2.050
2.025
1.999
1.889
1.802
1.765
1.685
1.644
9.87
2.11
8.05
4.11
N
N+
Cl−

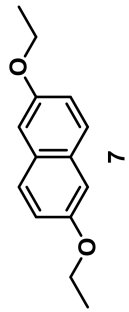

X : parts per Million : Carbon13
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
137.516
119.200
77.324
77.000
76.685
64.023
36.878
36.602
31.101
30.996
26.619
26.562
N
N+
Cl−


X : parts per Million : Proton
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
7.916
7.748
7.725
7.716
7.695
7.562
7.560
7.543
7.539
7.260
7.153
7.147
7.131
7.125
7.107
7.101
1.03
1.03
1.08
1.04
1.01
1.03
4.182
4.165
4.147
4.129
2.09
1.549
1.502
1.484
1.467
2.98
0.339
0.331
0.323
9.00
Si
O
5

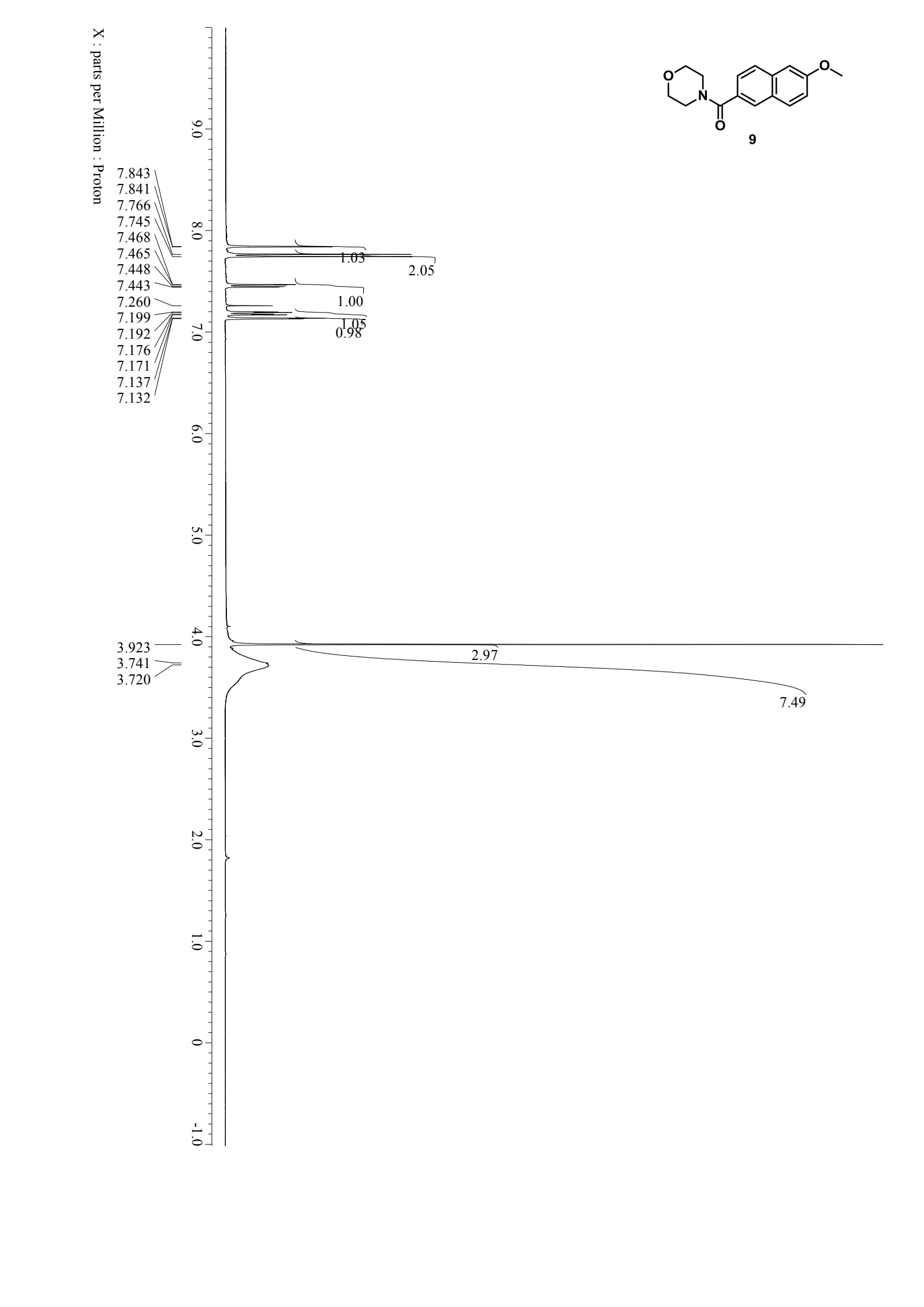

X : parts per Million : Carbon13
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
-10.0
157.205
134.856
134.818
133.464
130.336
129.507
128.449
125.865
118.962
106.309
77.315
77.000
76.676
63.423
14.796
-1.031
Si
O

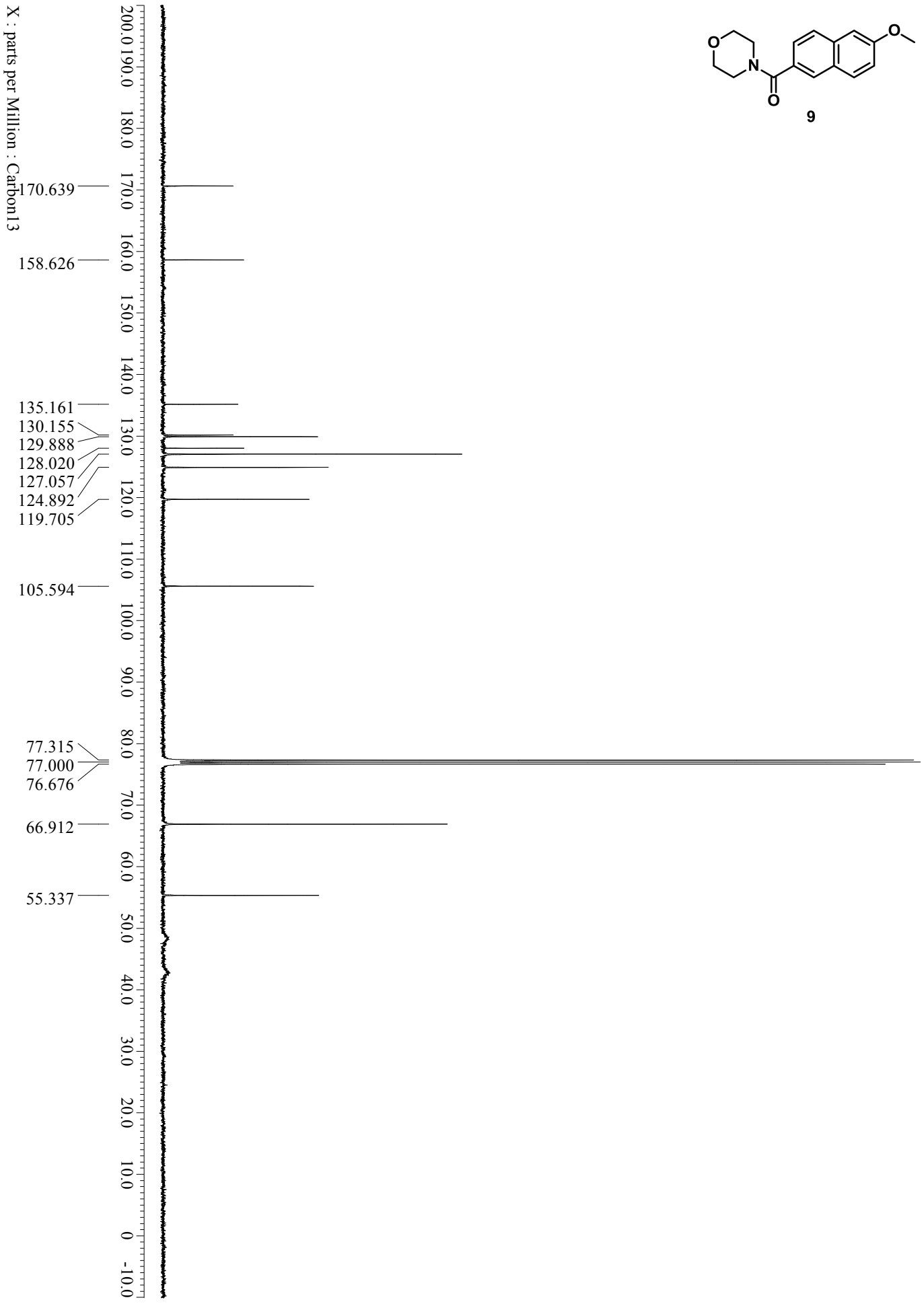
5

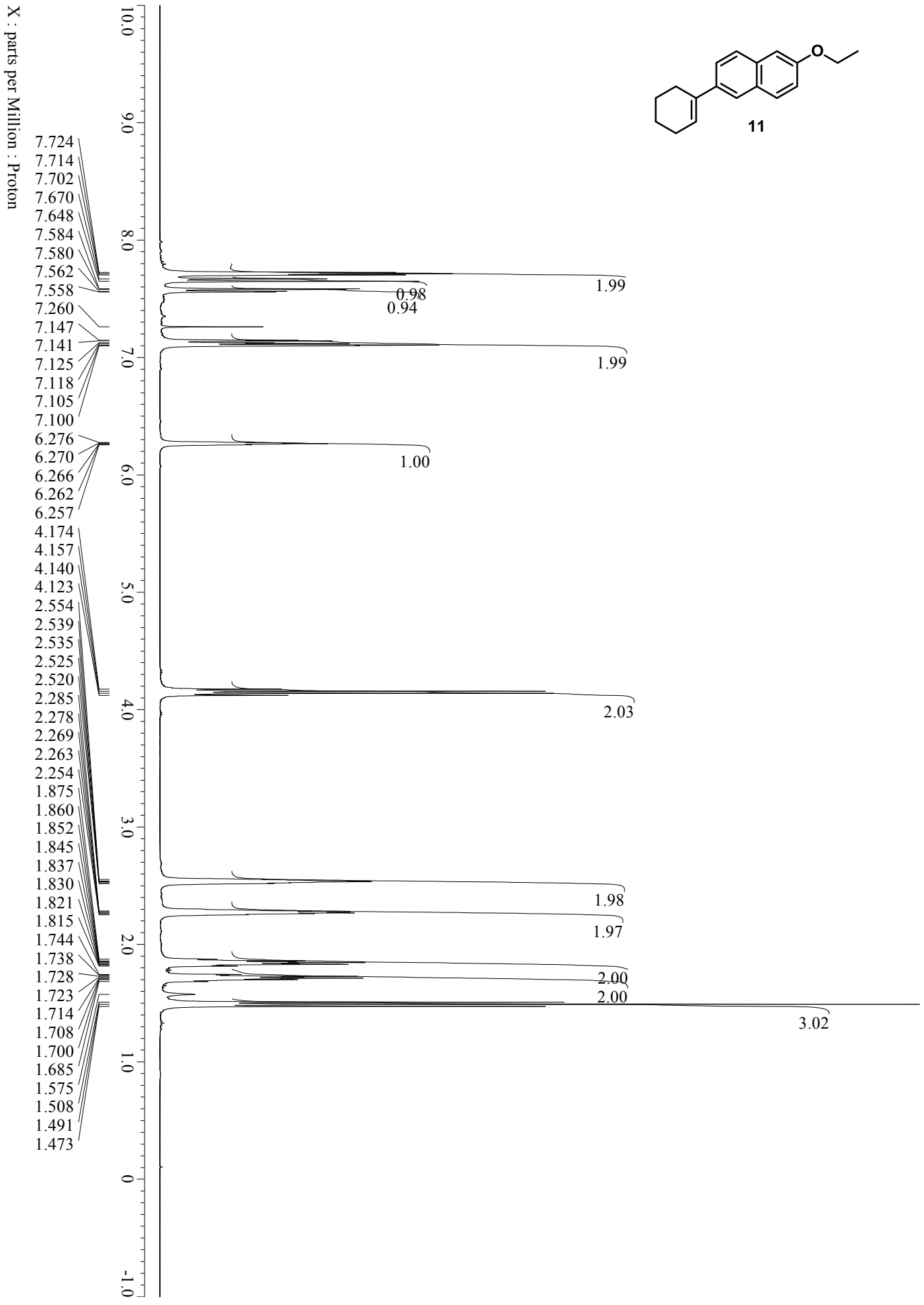

X : parts per Million : Proton
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.630
7.608
7.260
7.132
7.126
7.110
7.104
7.089
7.084
4.150
4.133
4.114
4.097
1.555
1.488
1.471
1.452
1.99
1.97
1.96
4.00
5.91
O
O
7

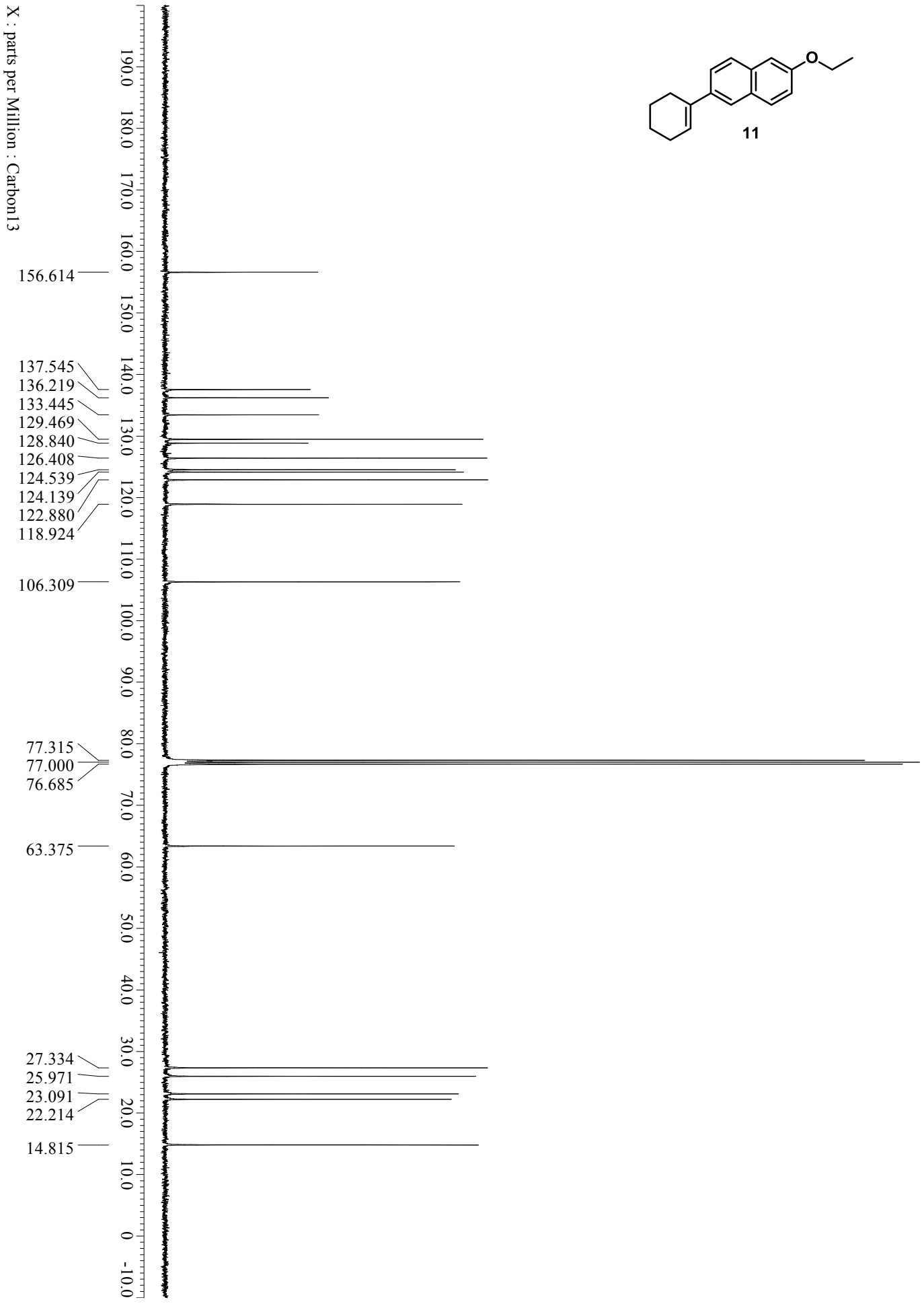

7

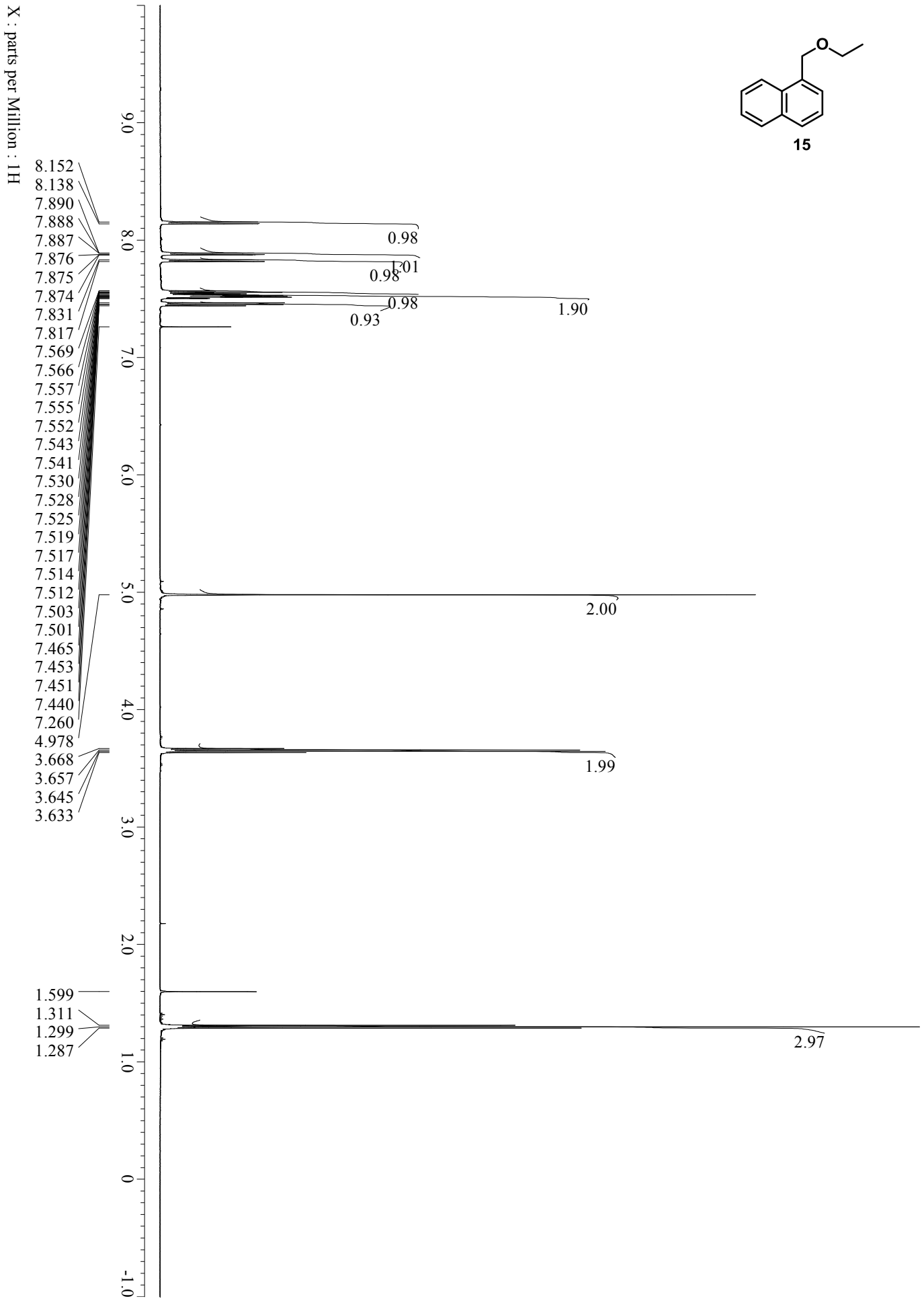
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
155.317
129.679
128.048
119.152
106.862
77.324
77.000
76.685
63.432
14.863
X : parts per Million : Carbon13

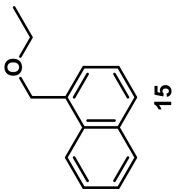

X : parts per Million : Proton
9
7.843
7.841
7.766
7.745
7.468
7.465
7.448
7.443
7.260
7.199
7.192
7.176
7.171
7.137
7.132
3.923
3.741
3.720
1.03
2.05
1.00
1.05
0.98
2.97
7.49
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0


X : parts per Million : Carbon13
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
-10.0
170.639
158.626
135.161
130.155
129.888
128.020
127.057
124.892
119.705
105.594
77.315
77.000
76.676
66.912
55.337
O
N
O
O
9

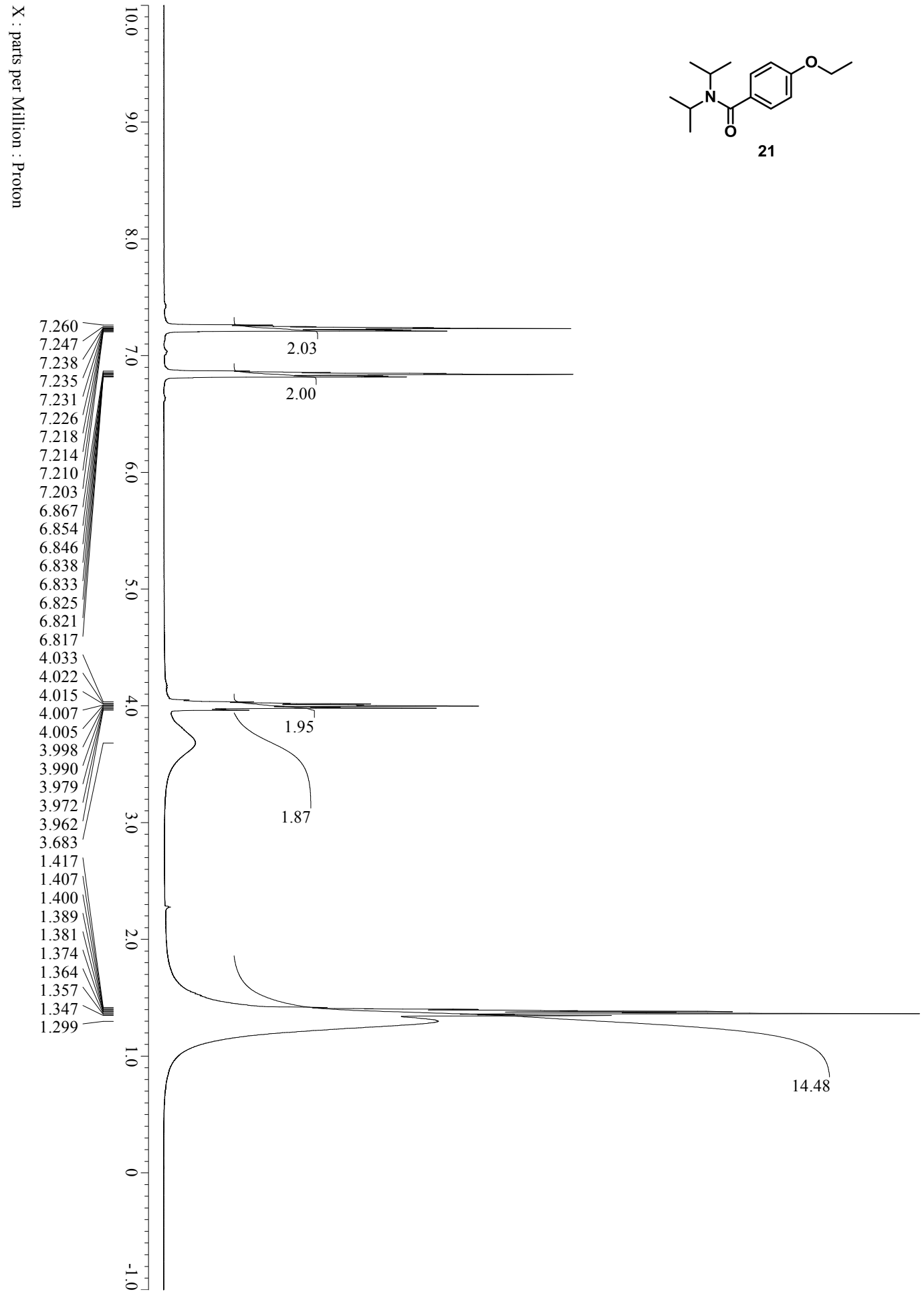

X : parts per Million : Proton
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
7.724
7.714
7.702
7.670
7.648
7.584
7.580
7.562
7.558
7.260
7.147
7.141
7.125
7.118
7.105
7.100
6.276
6.270
6.266
6.262
6.257
4.174
4.157
4.140
4.123
2.554
2.539
2.535
2.525
2.520
2.285
2.278
2.269
2.263
2.254
1.875
1.860
1.852
1.845
1.837
1.830
1.821
1.815
1.744
1.738
1.728
1.723
1.714
1.708
1.700
1.685
1.575
1.508
1.491
1.473
1.99
0.98
0.94
1.99
1.00
2.03
1.98
1.97
2.00
2.00
3.02
O
11

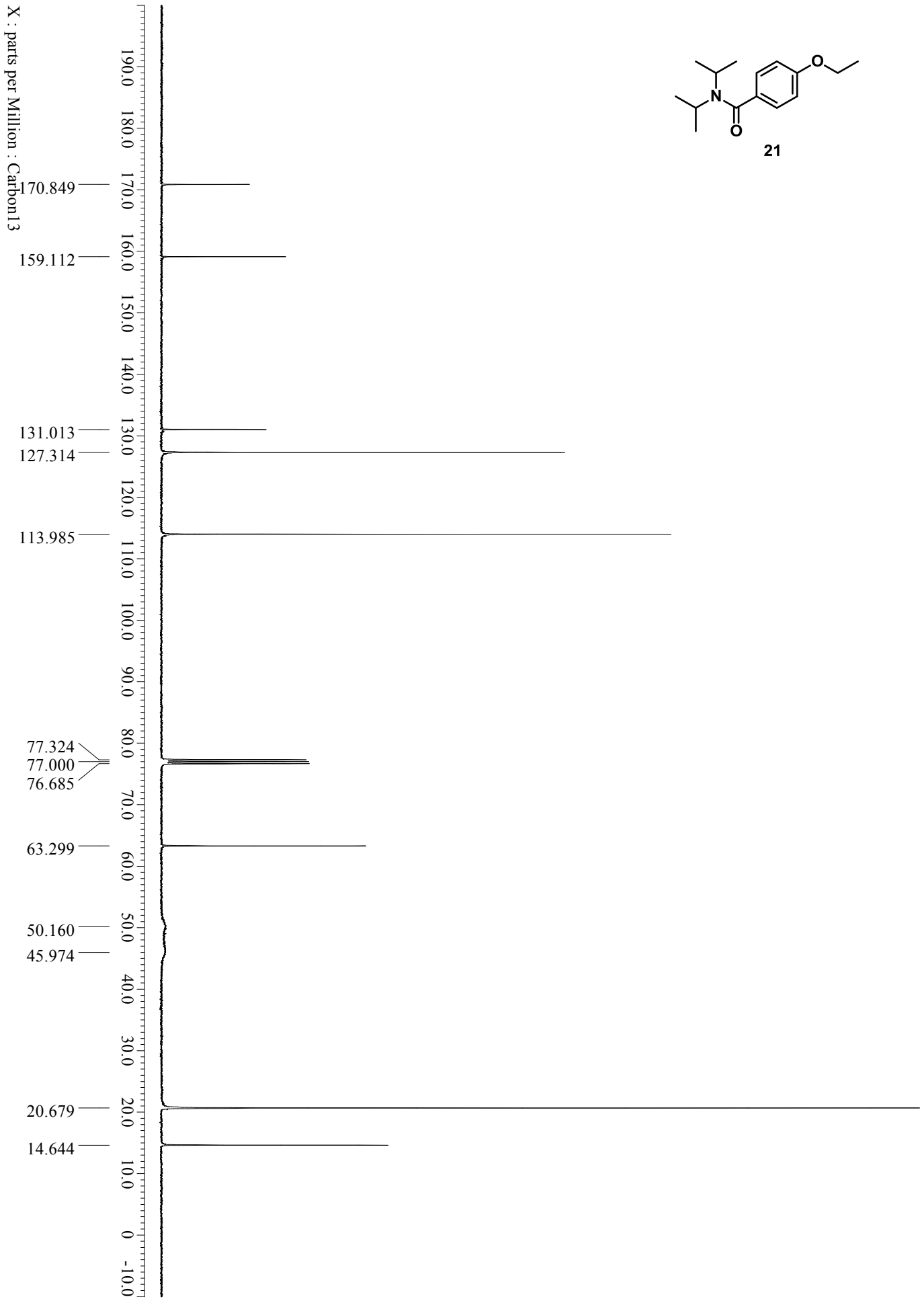

X : parts per Million : Carbon13
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
-10.0
156.614
137.545
136.219
133.445
129.469
128.840
126.408
124.539
124.139
122.880
118.924
106.309
77.315
77.000
76.685
63.375
27.334
25.971
23.091
22.214
14.815
O
11

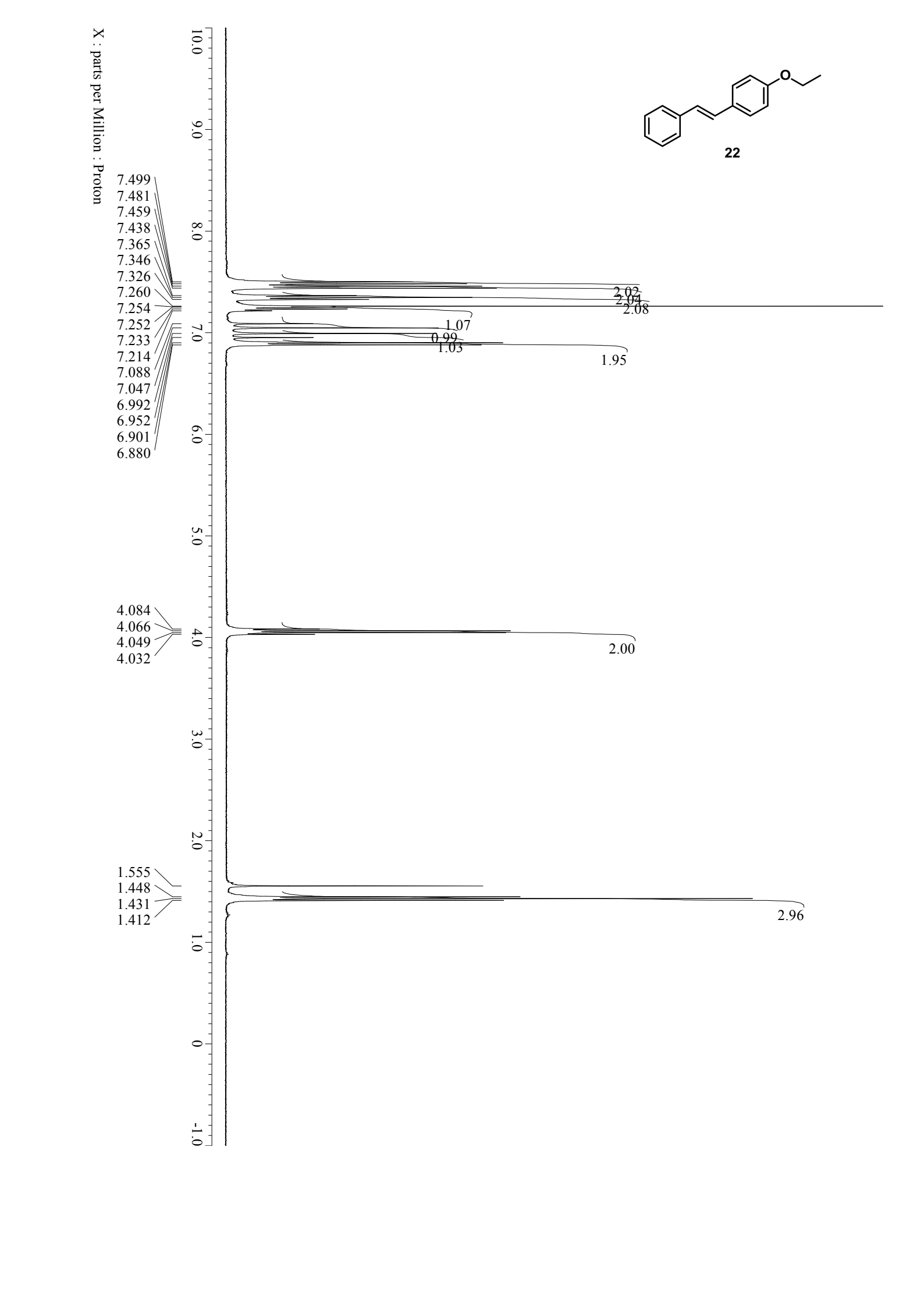

X : parts per Million : 1H
8.152
8.138
7.890
7.888
7.887
7.876
7.875
7.874
7.831
7.817
7.569
7.566
7.557
7.555
7.552
7.543
7.541
7.530
7.528
7.525
7.519
7.517
7.514
7.512
7.503
7.501
7.465
7.453
7.451
7.440
7.260
4.978
3.668
3.657
3.645
3.633
1.599
1.311
1.299
1.287
0.98
1.01
0.98
0.98
0.93
1.90
2.00
1.99
2.97
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
O
15

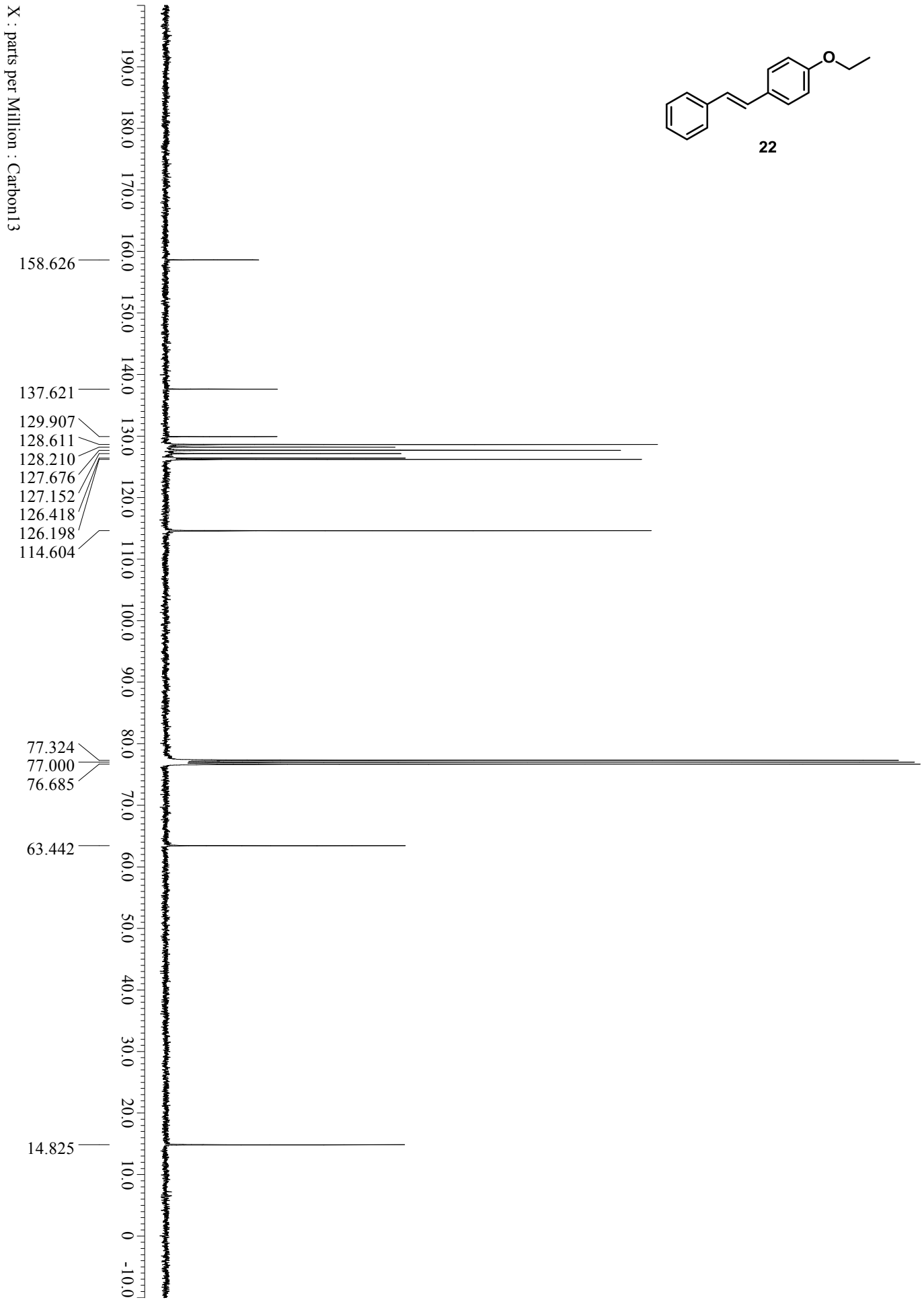


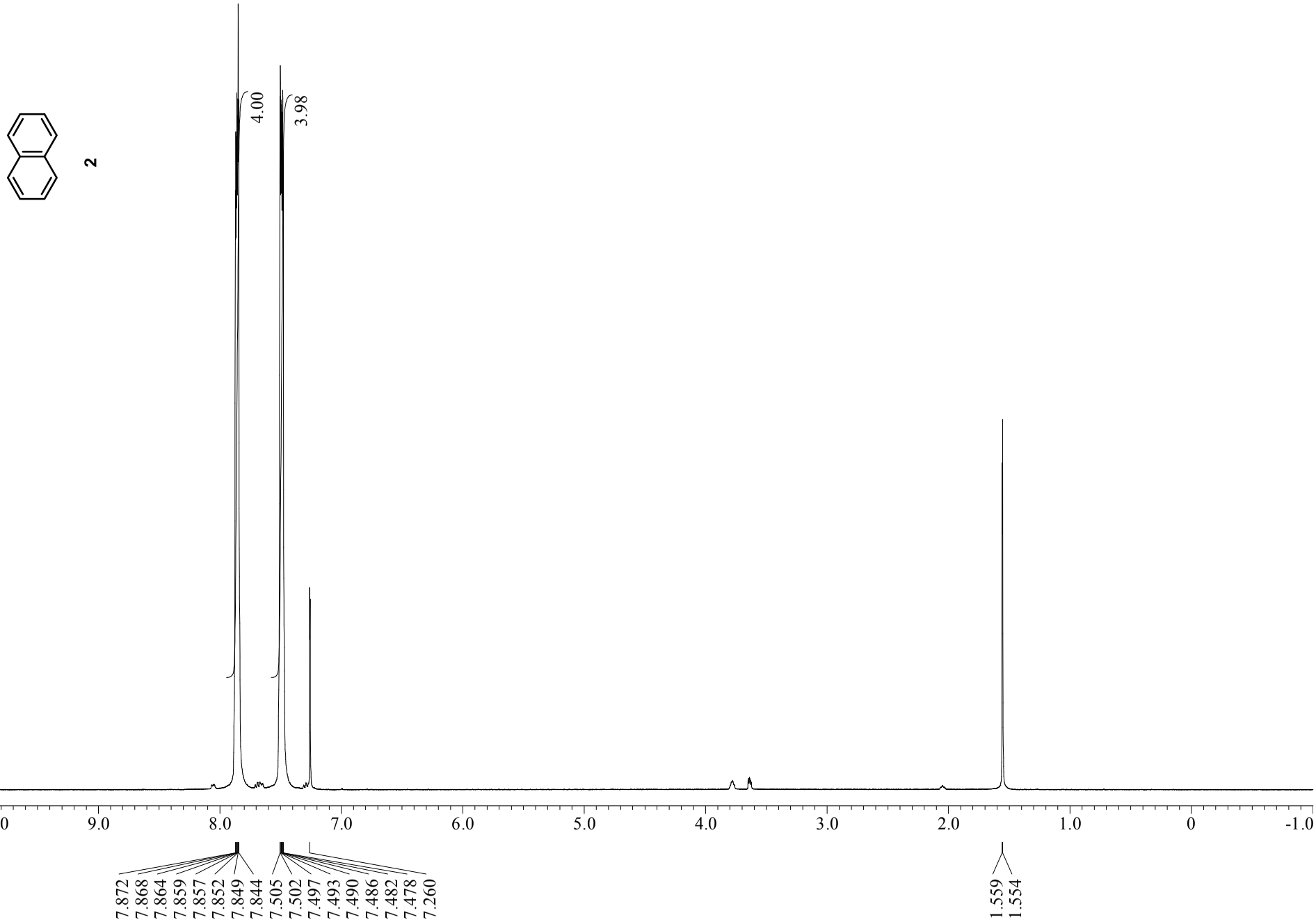
X : parts per Million : 13C


X : parts per Million : Proton
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
7.260
7.247
7.238
7.235
7.231
7.226
7.218
7.214
7.210
7.203
6.867
6.854
6.846
6.838
6.833
6.825
6.821
6.817
4.033
4.022
4.015
4.007
4.005
3.998
3.990
3.979
3.972
3.962
3.683
1.417
1.407
1.400
1.389
1.381
1.374
1.364
1.357
1.347
1.299
2.03
2.00
1.95
1.87
14.48
O
N
O
21

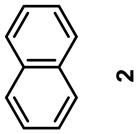

X : parts per Million : Carbon13
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
-10.0
170.849
159.112
131.013
127.314
113.985
77.324
77.000
76.685
63.299
50.160
45.974
20.679
14.644
N
O
O
21


X : parts per Million : Proton
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
7.499
7.481
7.459
7.438
7.365
7.346
7.326
7.260
7.254
7.252
7.233
7.214
7.088
7.047
6.992
6.952
6.901
6.880
4.084
4.066
4.049
4.032
1.555
1.448
1.431
1.412
2.02
2.04
2.08
1.07
0.99
1.03
1.95
2.00
2.96
O
22


X : parts per Million : Carbon13
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
-10.0
158.626
137.621
129.907
128.611
128.210
127.676
127.152
126.418
126.198
114.604
77.324
77.000
76.685
63.442
14.825
O
22

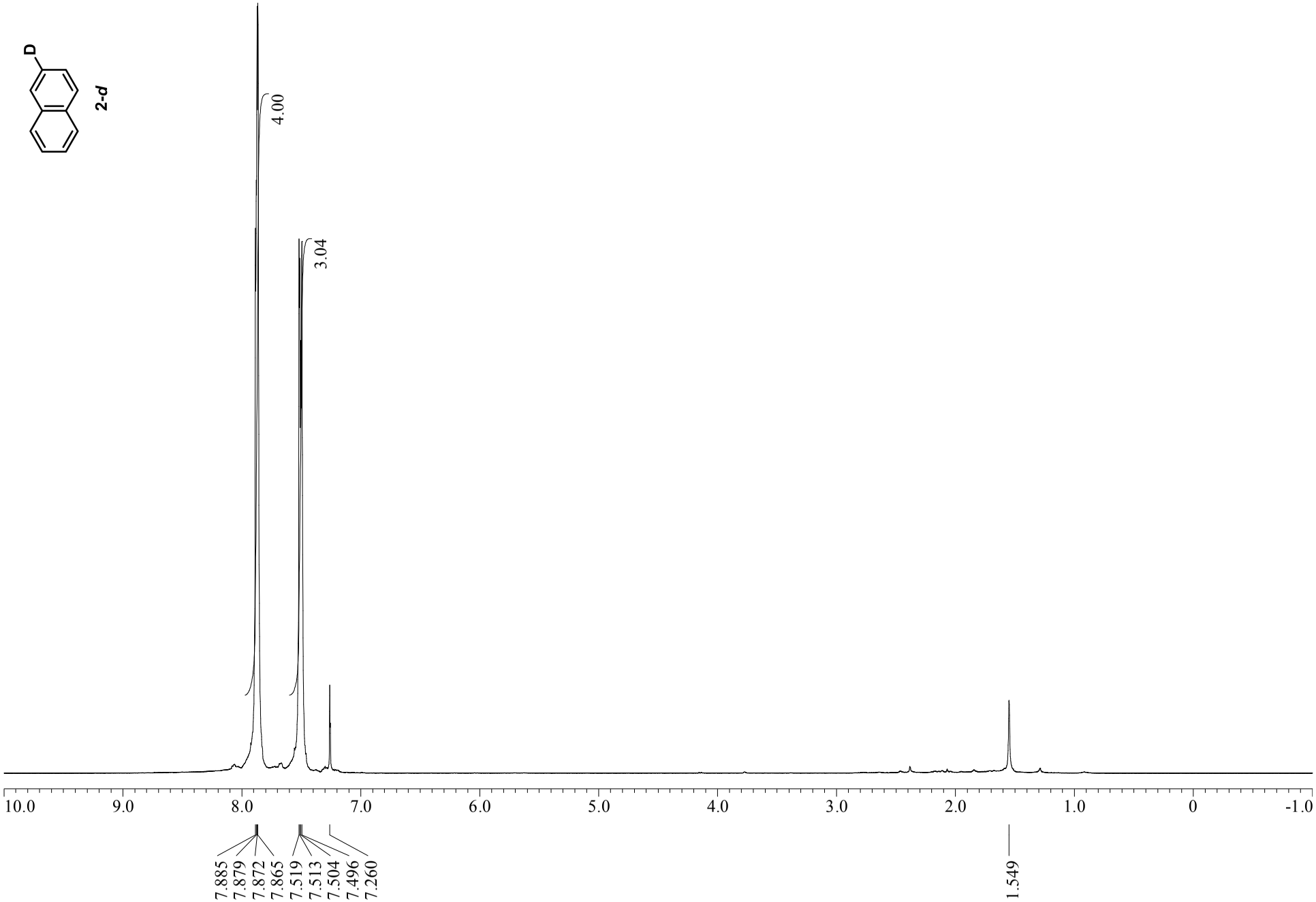


X : parts per Million : Proton

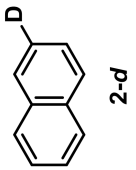


2

X : parts per Million : Carbon13

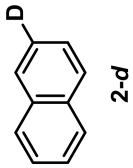


X : parts per Million : Proton



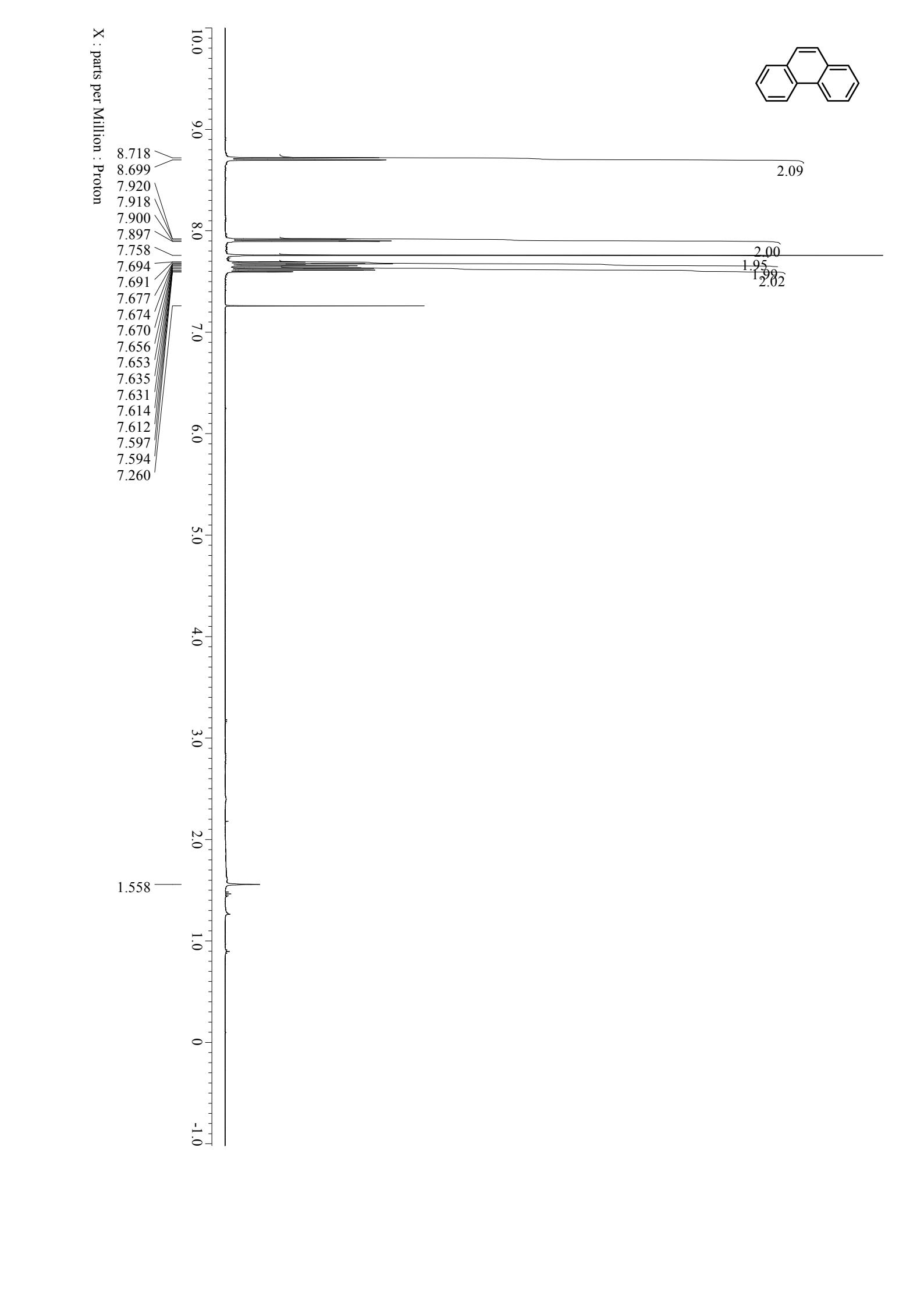
2-*d*

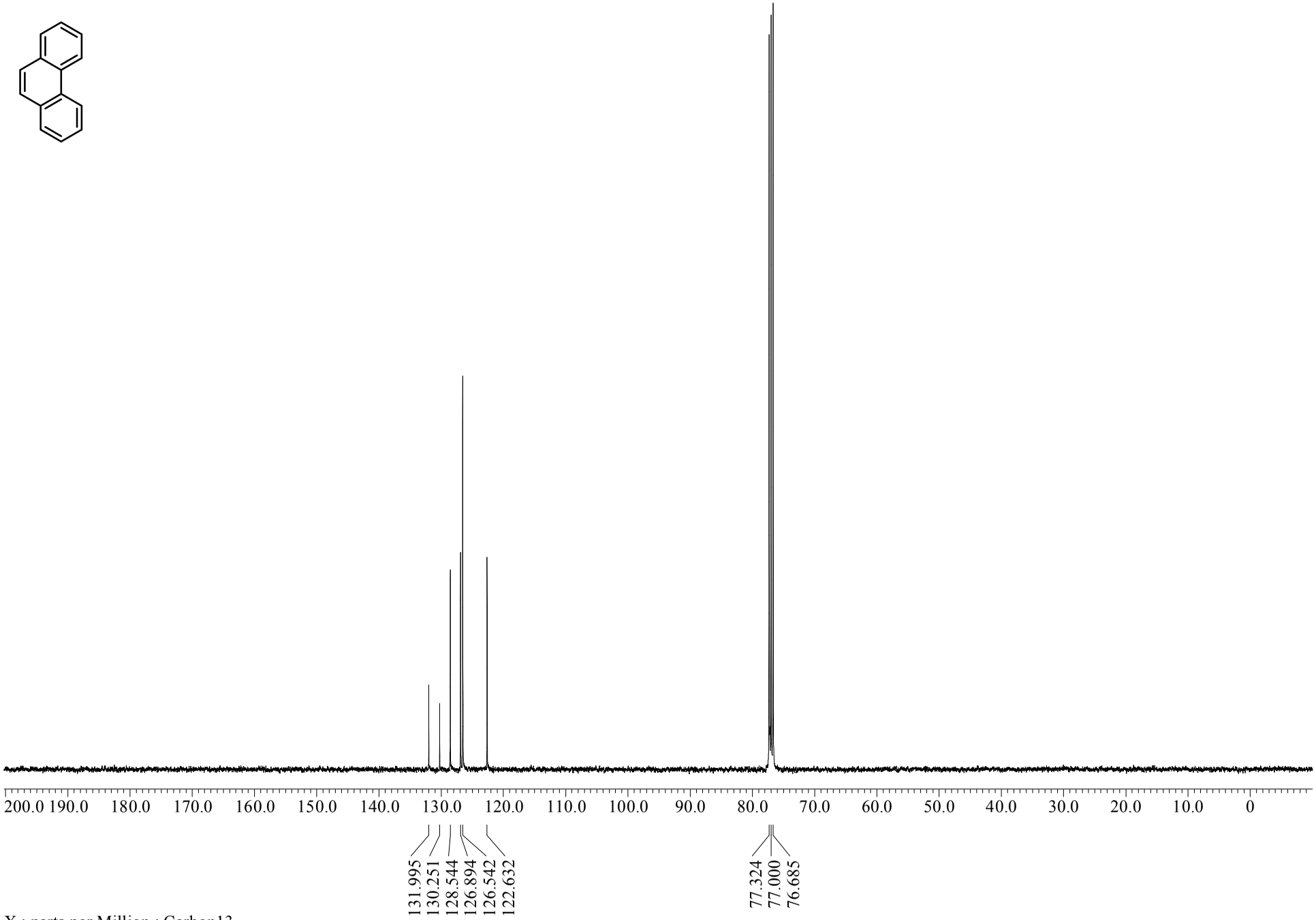
X : parts per Million : Carbon13



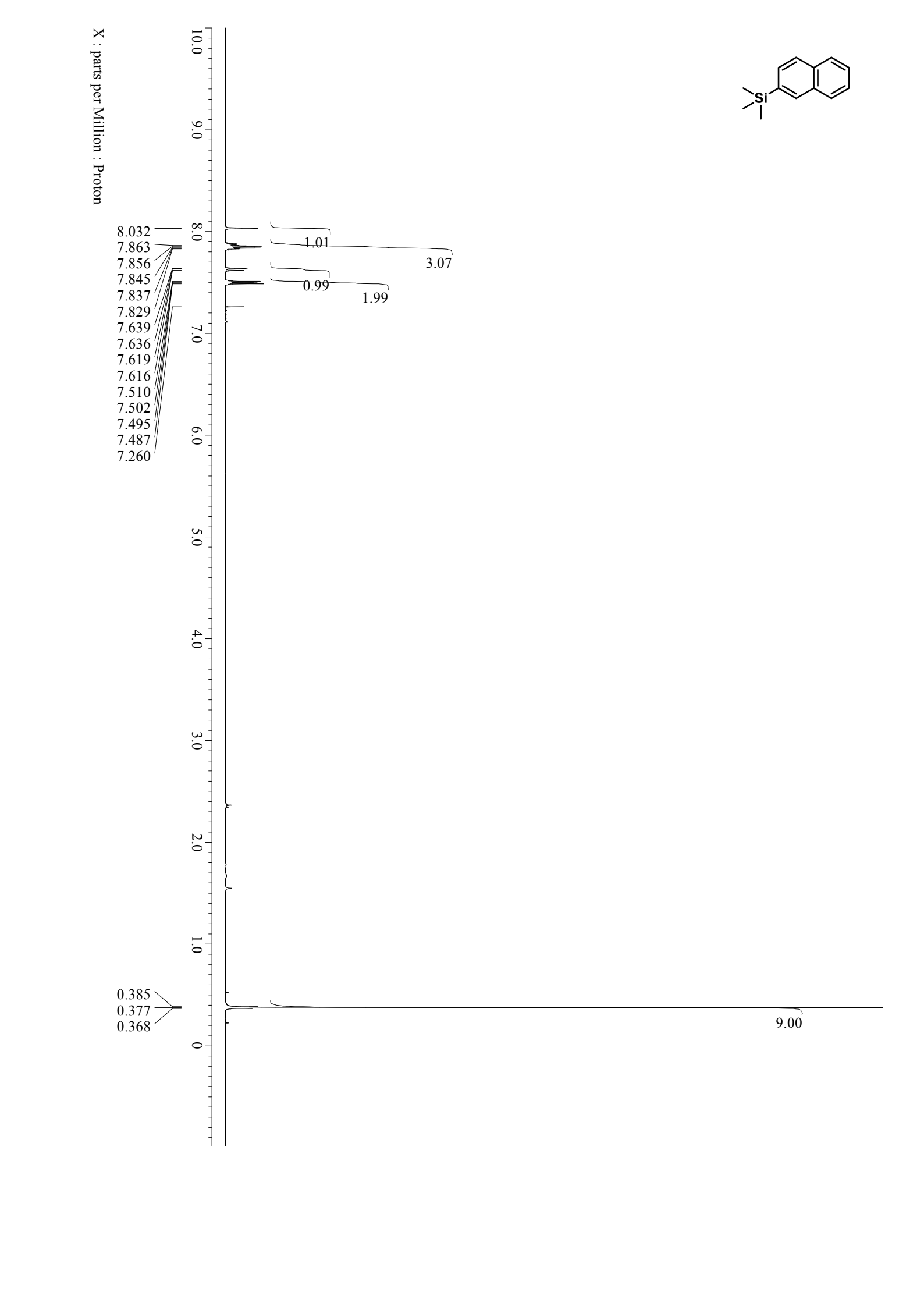
2-*d*

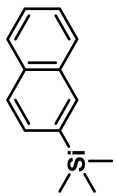
X : parts per Million : Deuterium


X : parts per Million : Proton
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
8.718
8.699
7.920
7.918
7.900
7.897
7.758
7.694
7.691
7.677
7.674
7.670
7.656
7.653
7.635
7.631
7.614
7.612
7.597
7.594
7.260
1.558
2.09
2.00
1.95
1.99
2.02

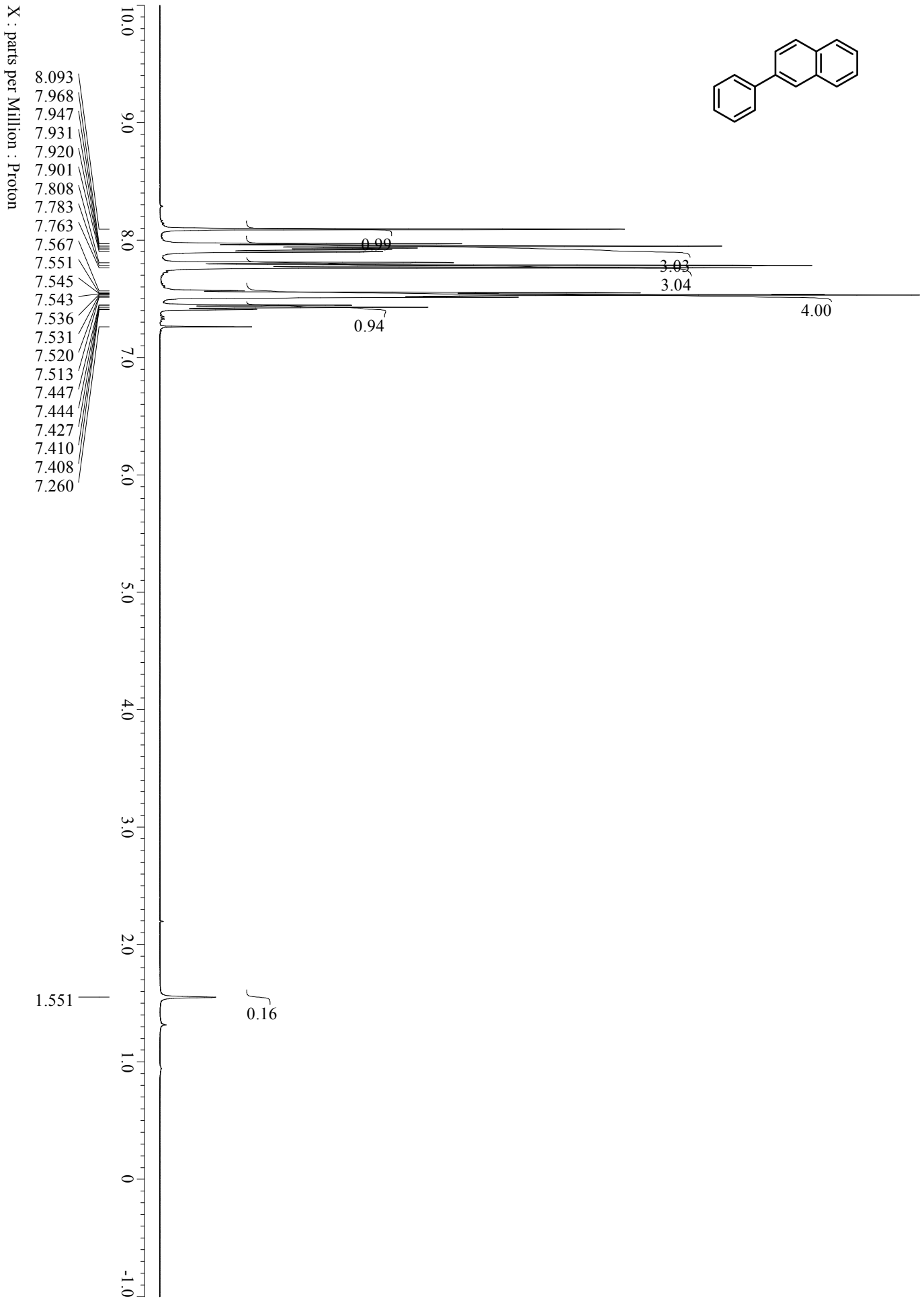


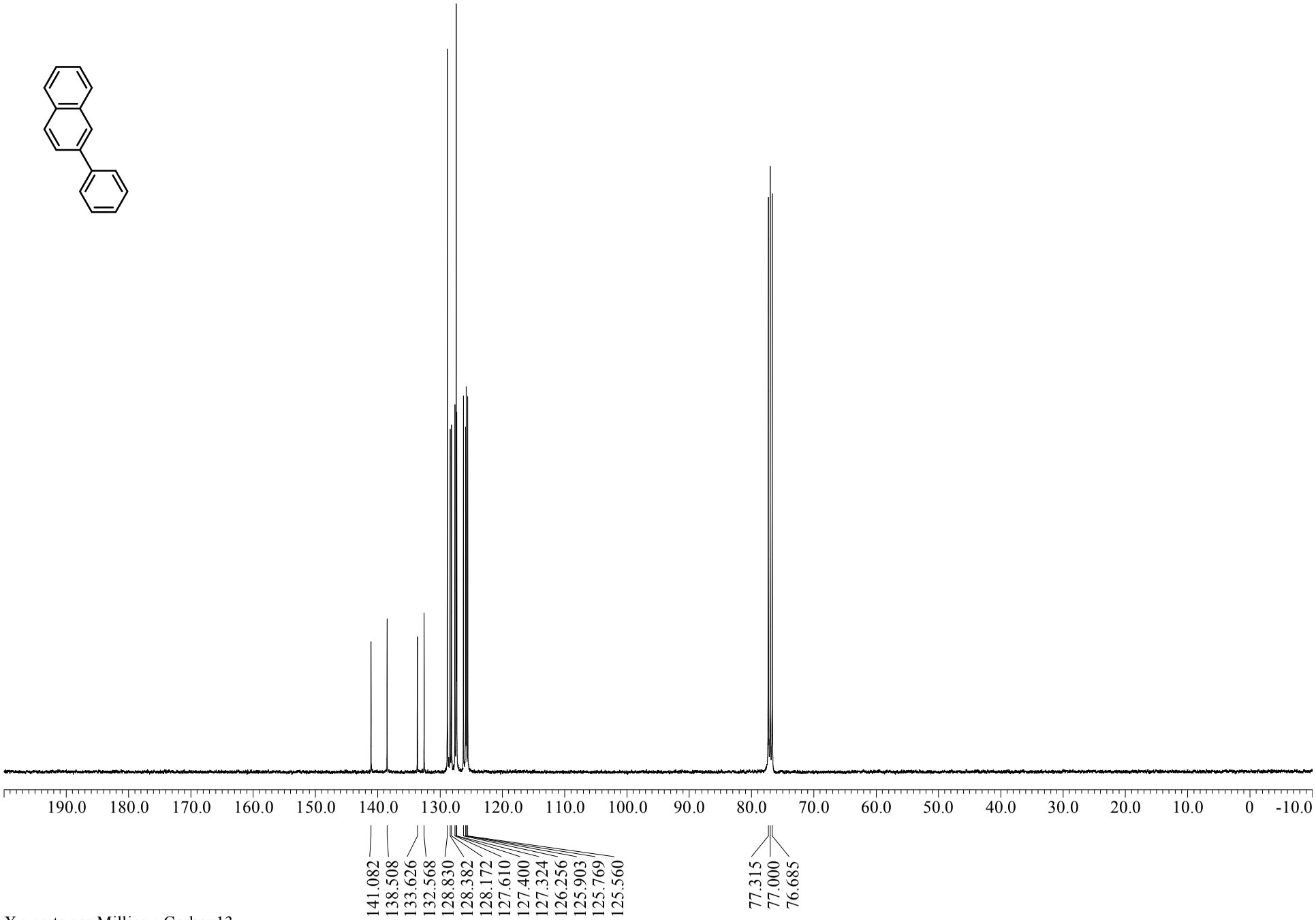
X : parts per Million : Carbon13


X : parts per Million : Proton
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
8.032
7.863
7.856
7.845
7.837
7.829
7.639
7.636
7.619
7.616
7.510
7.502
7.495
7.487
7.260
0.385
0.377
0.368
1.01
3.07
0.99
1.99
9.00
Si

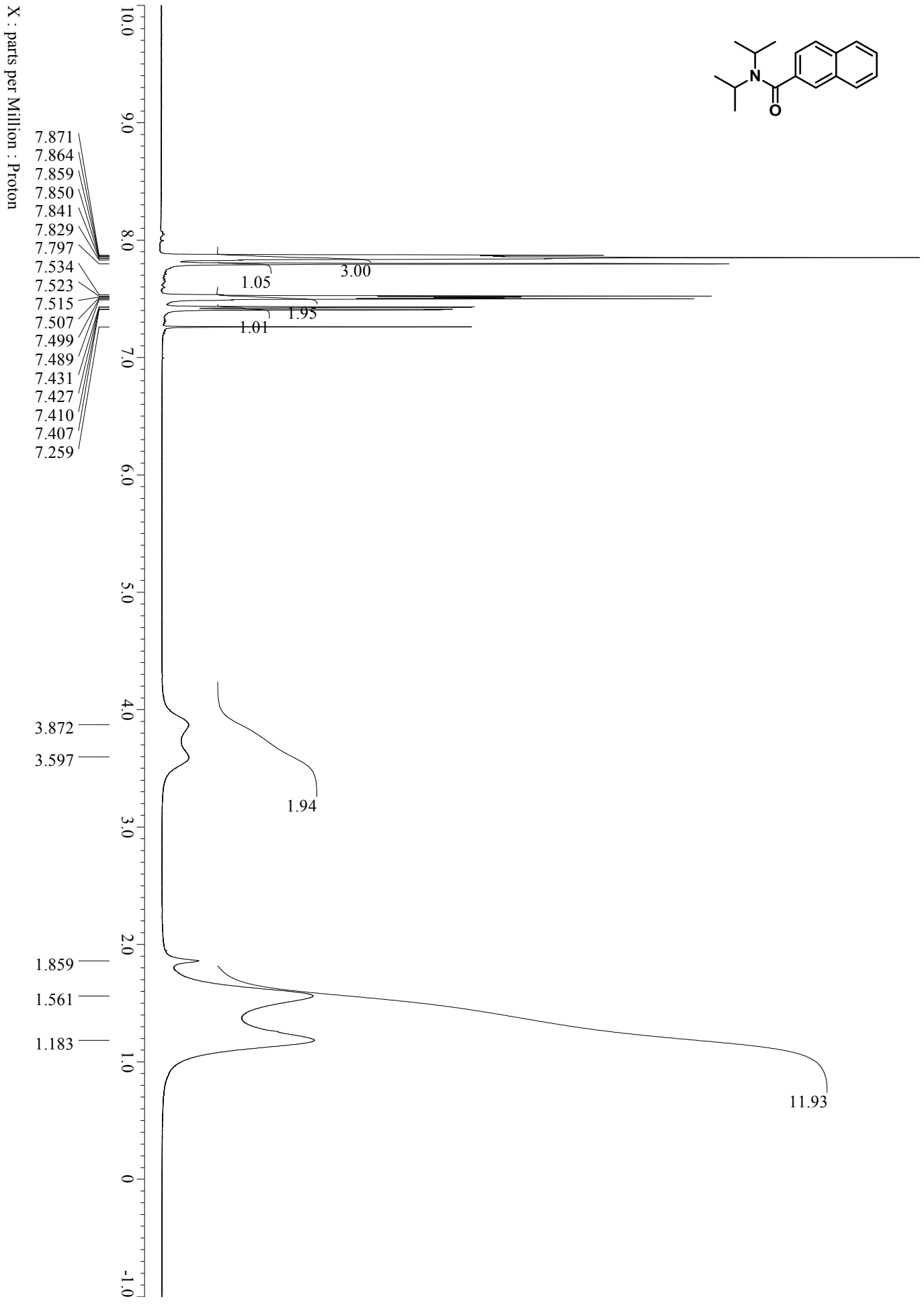


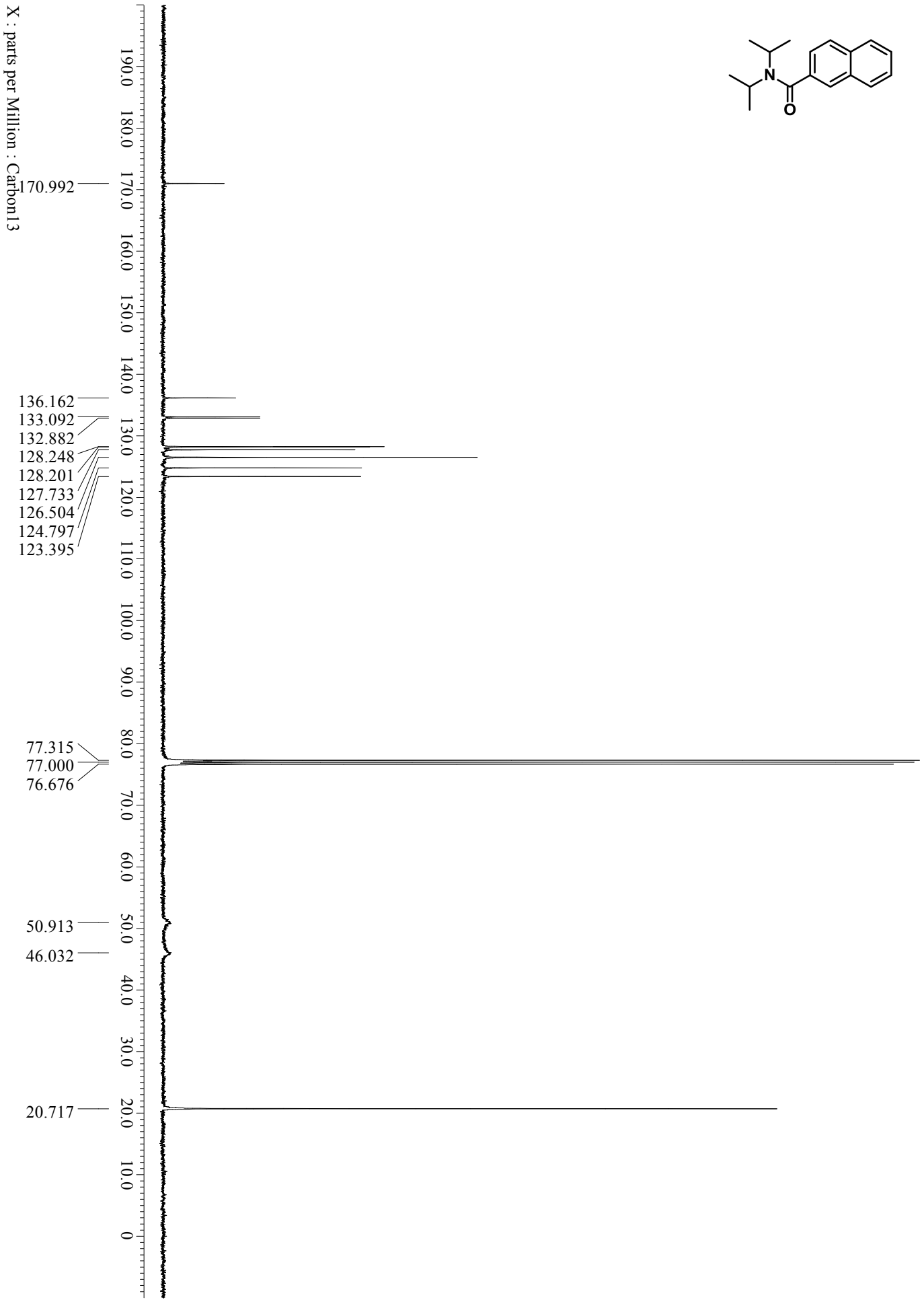
X : parts per Million : Carbon13


X : parts per Million : Proton
8.093
7.968
7.947
7.931
7.920
7.901
7.808
7.783
7.763
7.567
7.551
7.545
7.543
7.536
7.531
7.520
7.513
7.447
7.444
7.427
7.410
7.408
7.260
1.551
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
0.99
3.03
3.04
4.00
0.94
0.16



X : parts per Million : Carbon13


X : parts per Million : Proton
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
7.871
7.864
7.859
7.850
7.841
7.829
7.797
7.534
7.523
7.515
7.507
7.499
7.489
7.431
7.427
7.410
7.407
7.259
3.872
3.597
1.859
1.561
1.183
3.00
1.05
1.95
1.01
1.94
11.93
N
O


X : parts per Million : Carbon13
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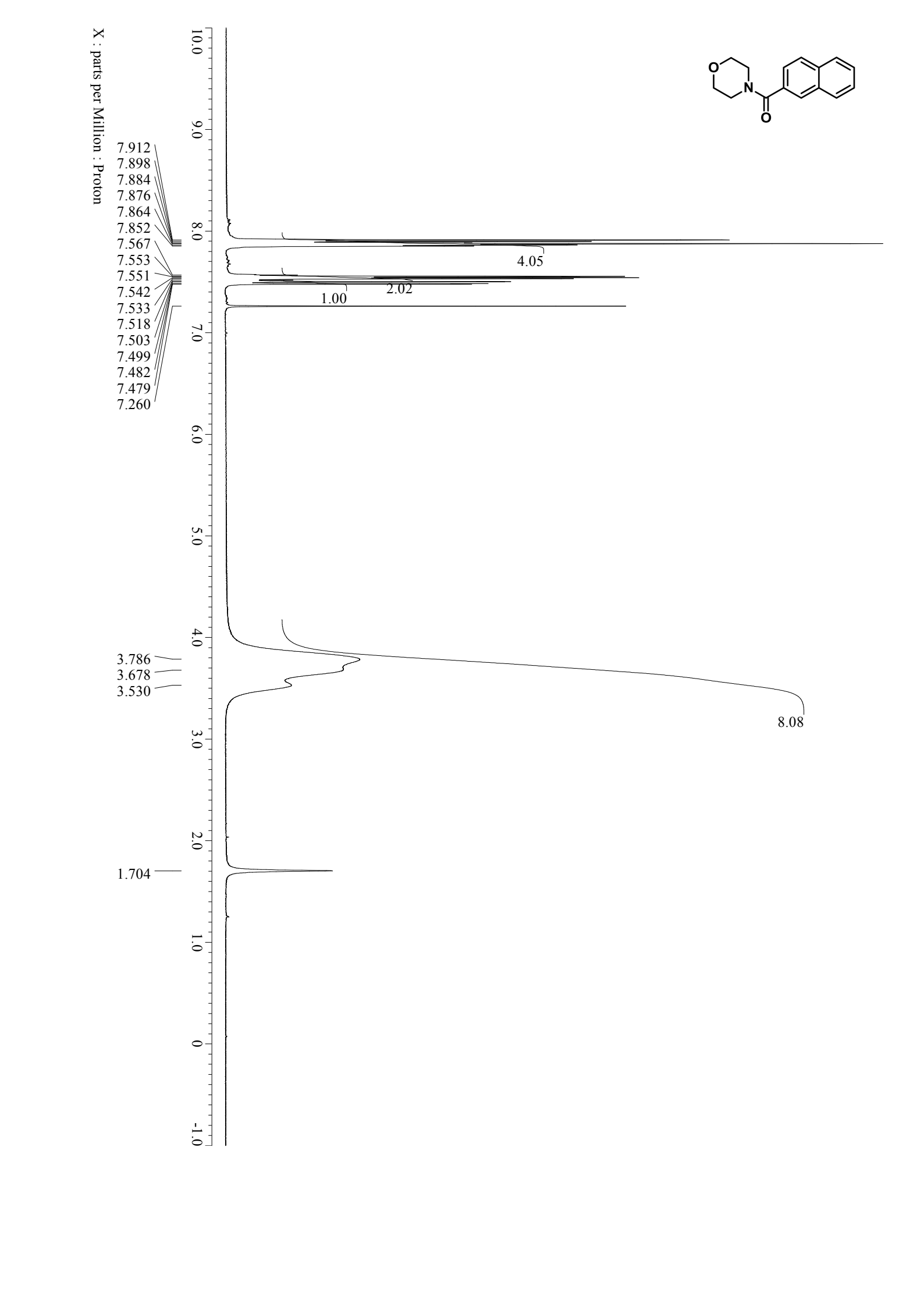
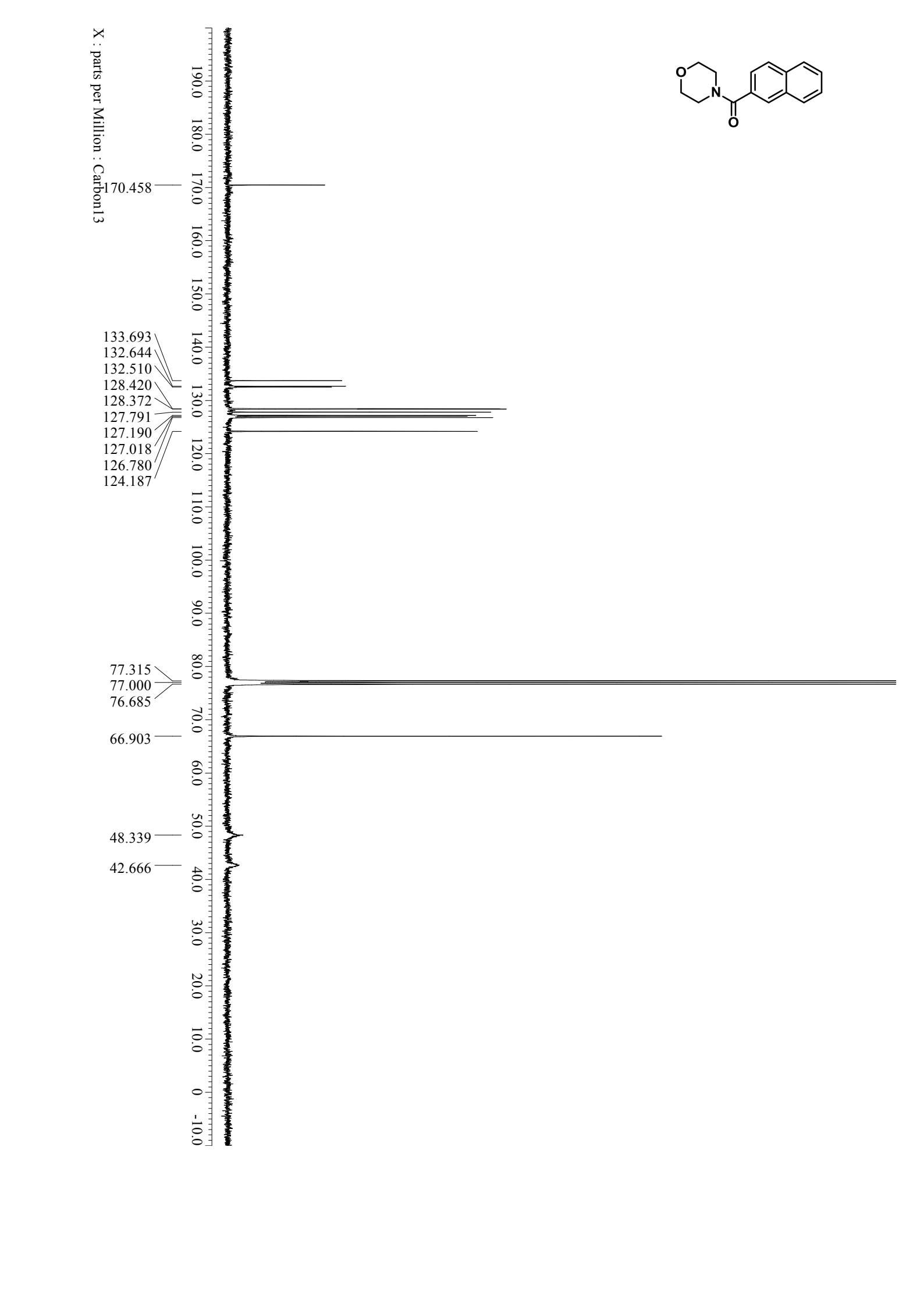
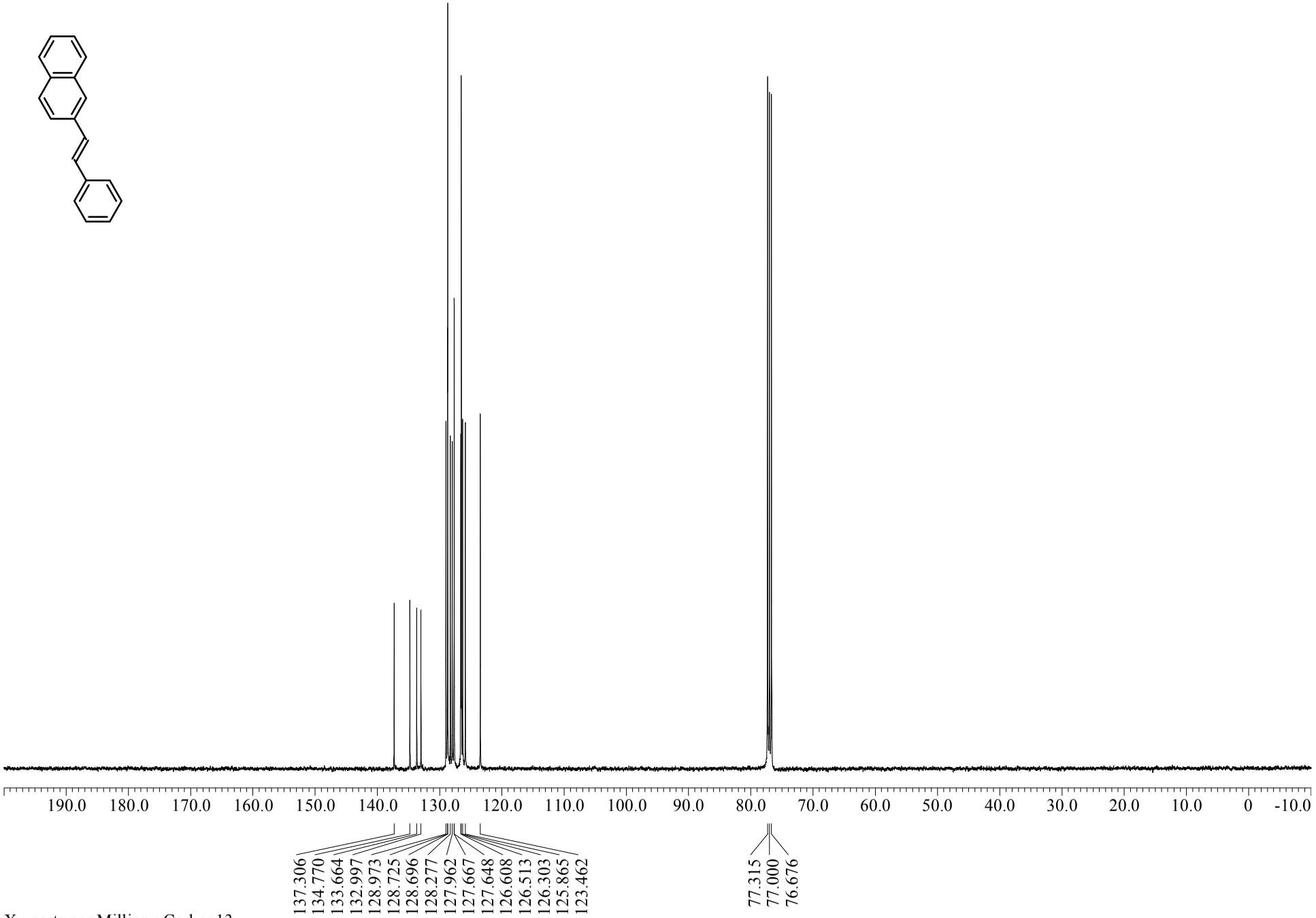
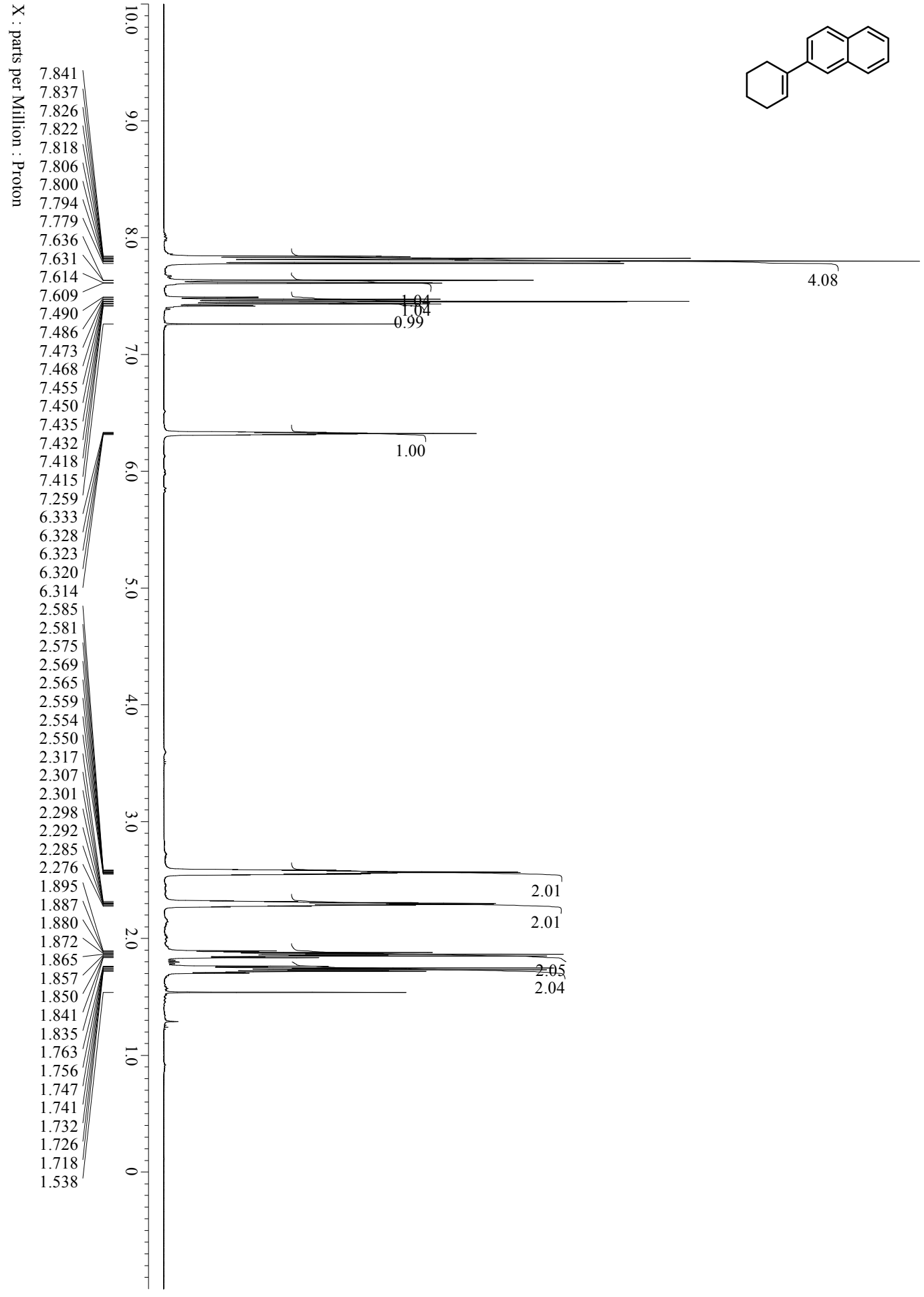
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
170.992
136.162
133.092
132.882
128.248
128.201
127.733
126.504
124.797
123.395
77.315
77.000
76.676
50.913
46.032
20.717
N
O


X : parts per Million : Proton
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
7.912
7.898
7.884
7.876
7.864
7.852
7.567
7.553
7.551
7.542
7.533
7.518
7.503
7.499
7.482
7.479
7.260
3.786
3.678
3.530
1.704
4.05
2.02
1.00
8.08
O
N
O


X : parts per Million : Carbon13
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
-10.0
170.458
133.693
132.644
132.510
128.420
128.372
127.791
127.190
127.018
126.780
124.187
77.315
77.000
76.685
66.903
48.339
42.666
O
N
O

X : parts per Million : Proton
7.903
7.892
7.888
7.883
7.872
7.861
7.806
7.802
7.785
7.780
7.625
7.607
7.548
7.544
7.531
7.527
7.517
7.511
7.497
7.494
7.480
7.476
7.456
7.438
7.418
7.355
7.352
7.333
7.315
7.300
7.278
7.260
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
3.91
1.00
1.97
1.92
1.97
2.93


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
-10.0
137.306
134.770
133.664
132.997
128.973
128.725
128.696
128.277
127.962
127.667
127.648
126.608
126.513
126.303
125.865
123.462
77.315
77.000
76.676
X : parts per Million : Carbon13


X : parts per Million : Proton
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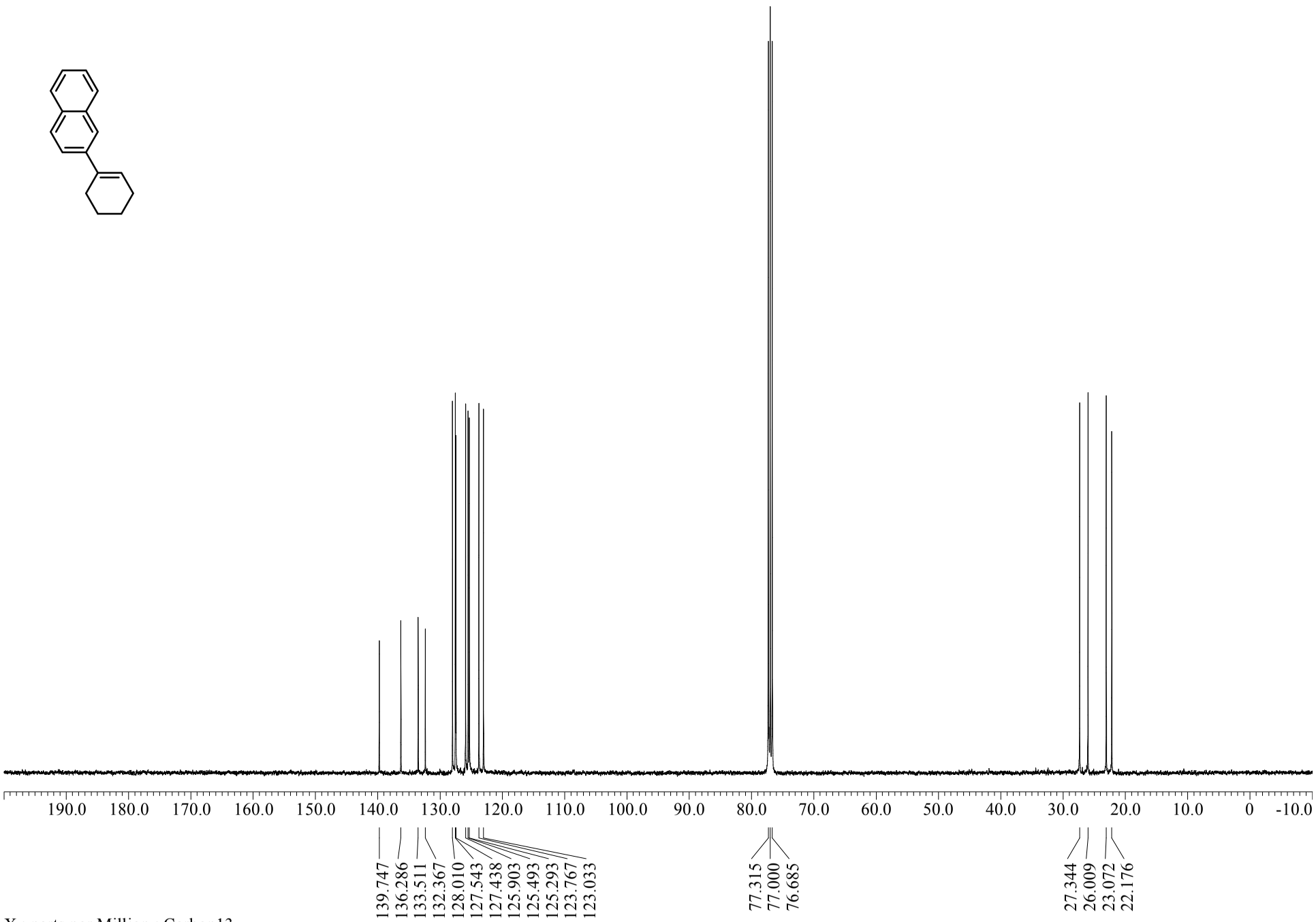
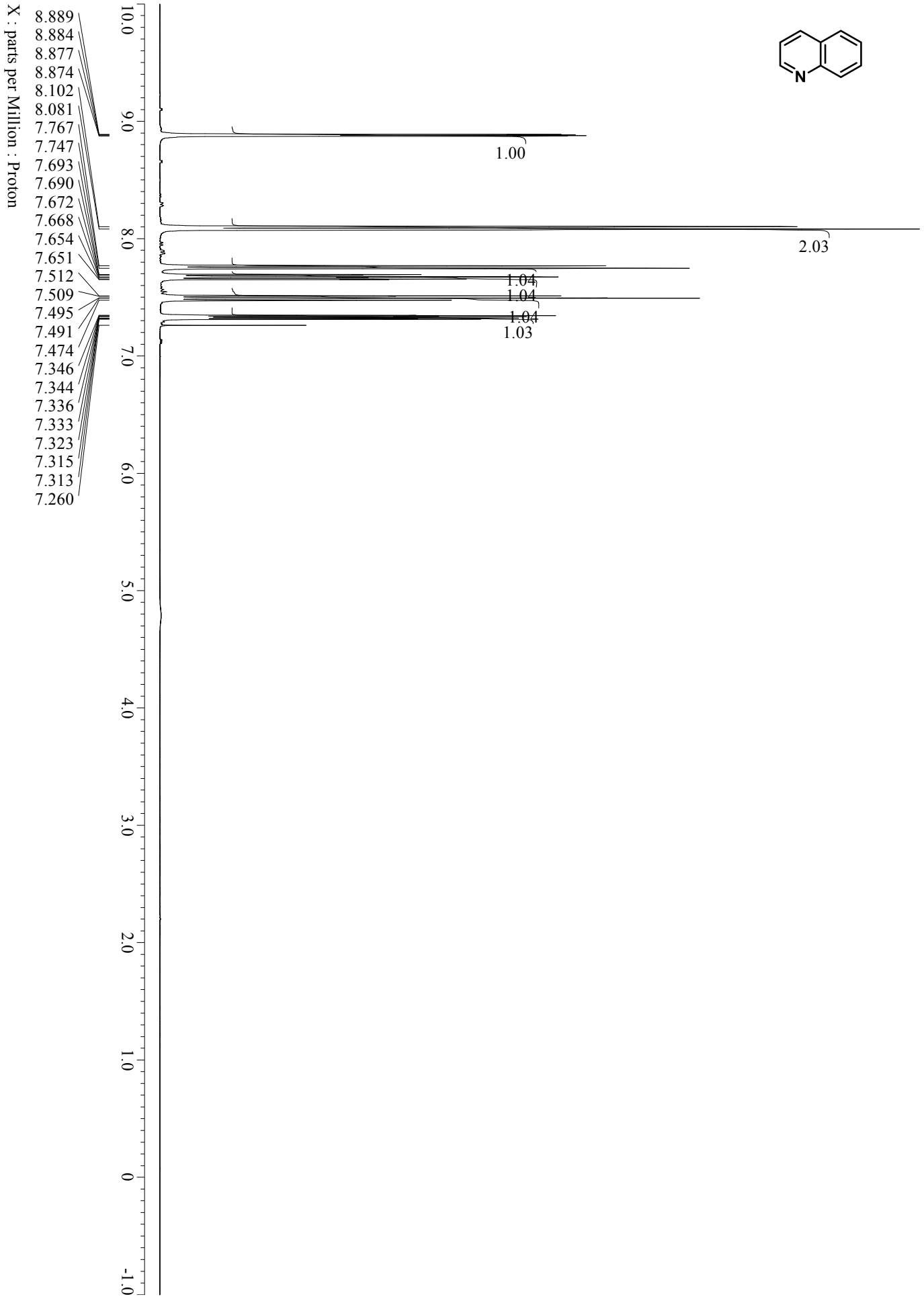
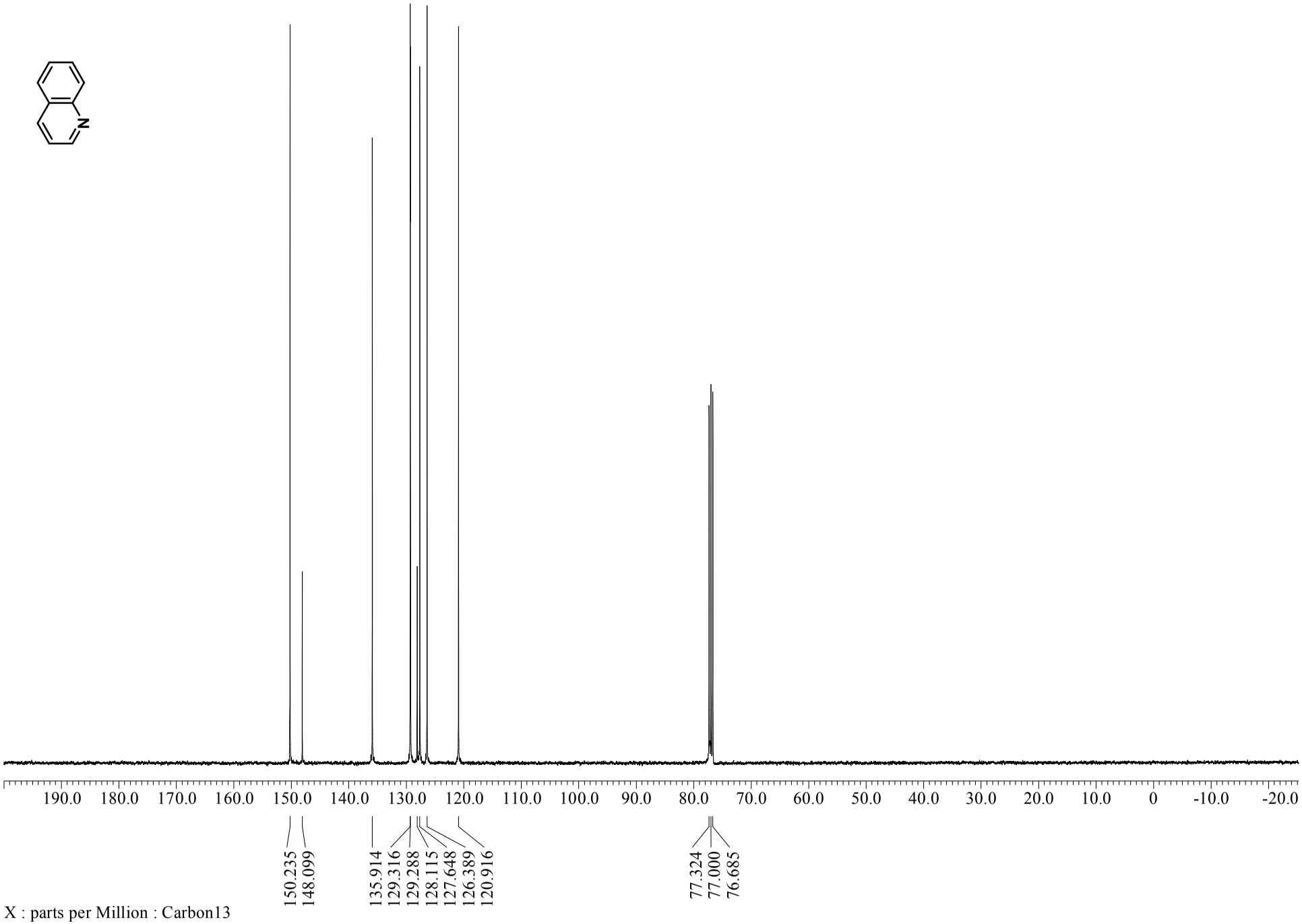
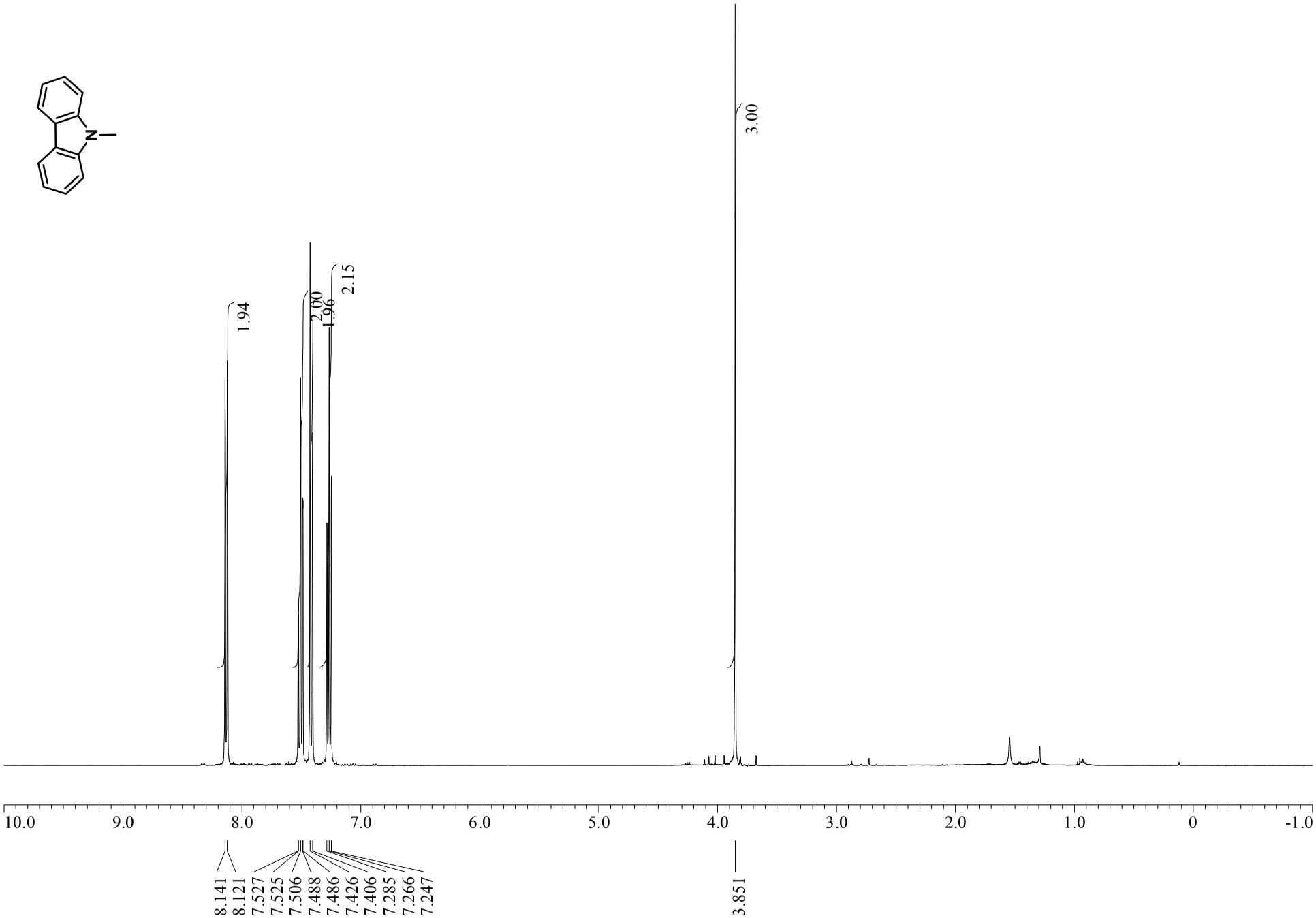
7.837
7.826
7.822
7.818
7.806
7.800
7.794
7.779
7.636
7.631
7.614
7.609
7.490
7.486
7.473
7.468
7.455
7.450
7.435
7.432
7.418
7.415
7.259
6.333
6.328
6.323
6.320
6.314
2.585
2.581
2.575
2.569
2.565
2.559
2.554
2.550
2.317
2.307
2.301
2.298
2.292
2.285
2.276
1.895
1.887
1.880
1.872
1.865
1.857
1.850
1.841
1.835
1.763
1.756
1.747
1.741
1.732
1.726
1.718
1.538
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
4.08
1.04
1.04
0.99
1.00
2.01
2.01
2.05
2.04



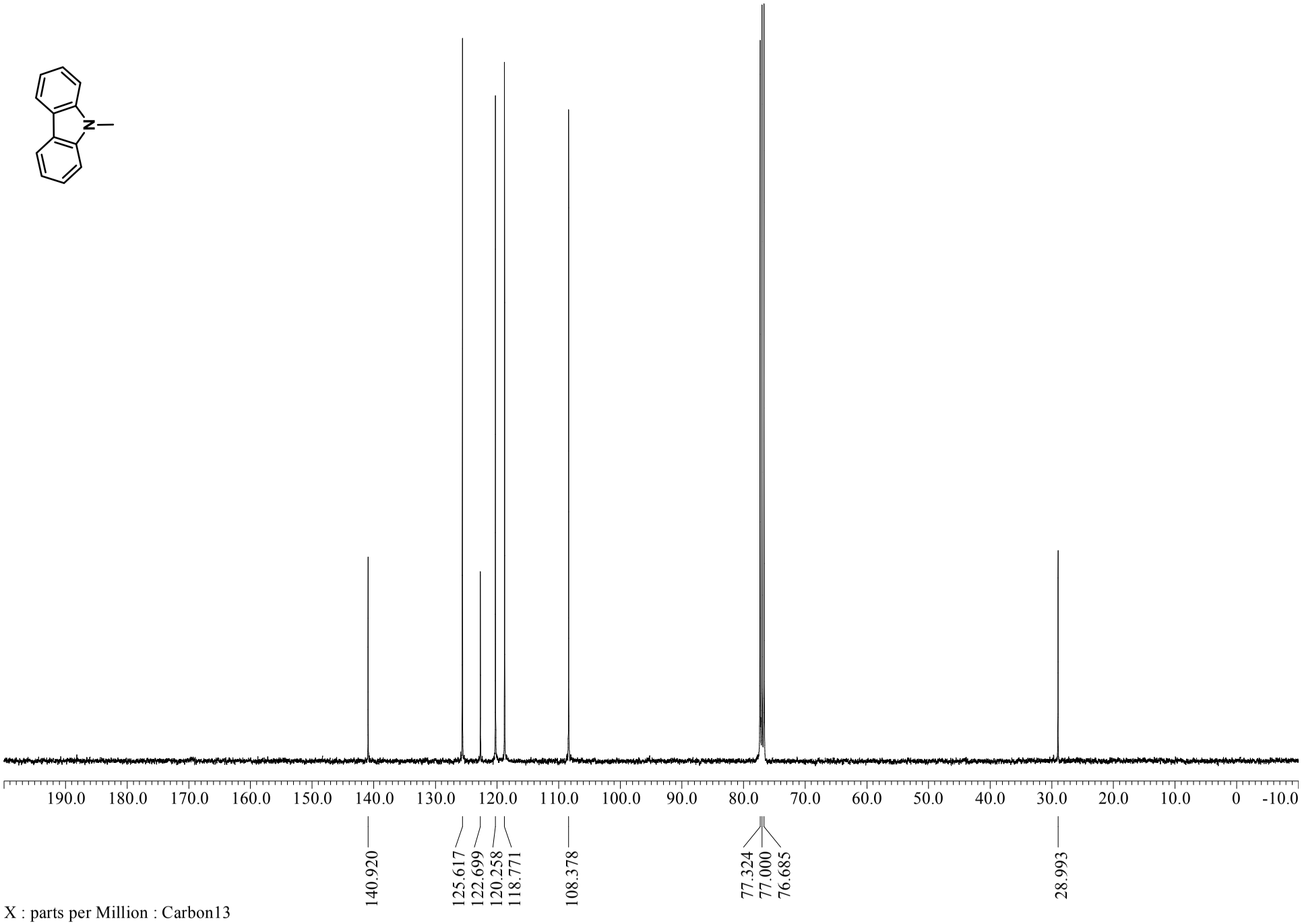
X : parts per Million : Carbon13

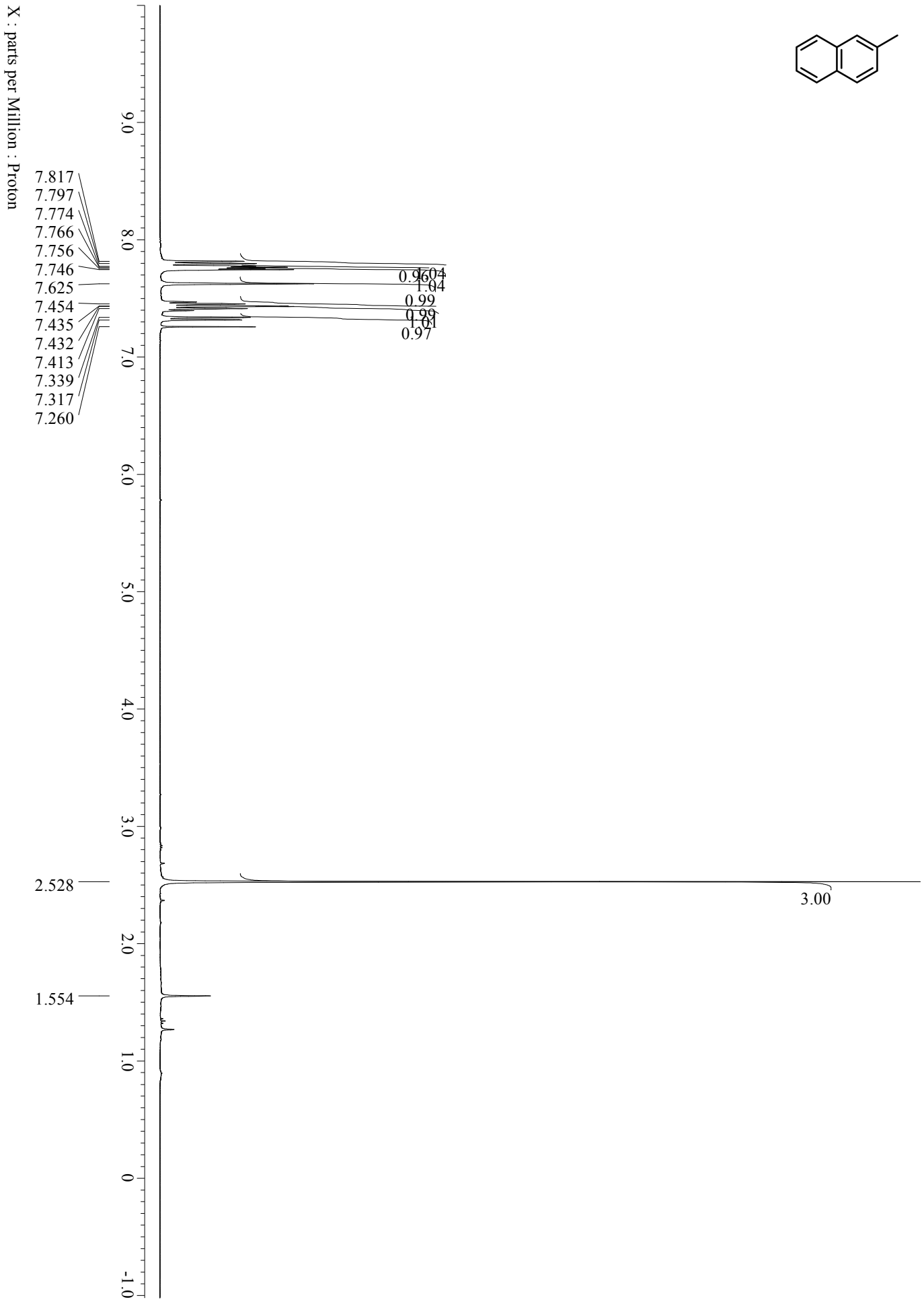

X : parts per Million : Proton
8.889
8.884
8.877
8.874
8.102
8.081
7.767
7.747
7.693
7.690
7.672
7.668
7.654
7.651
7.512
7.509
7.495
7.491
7.474
7.346
7.344
7.336
7.333
7.323
7.315
7.313
7.260
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
1.00
2.03
1.04
1.04
1.04
1.03
N

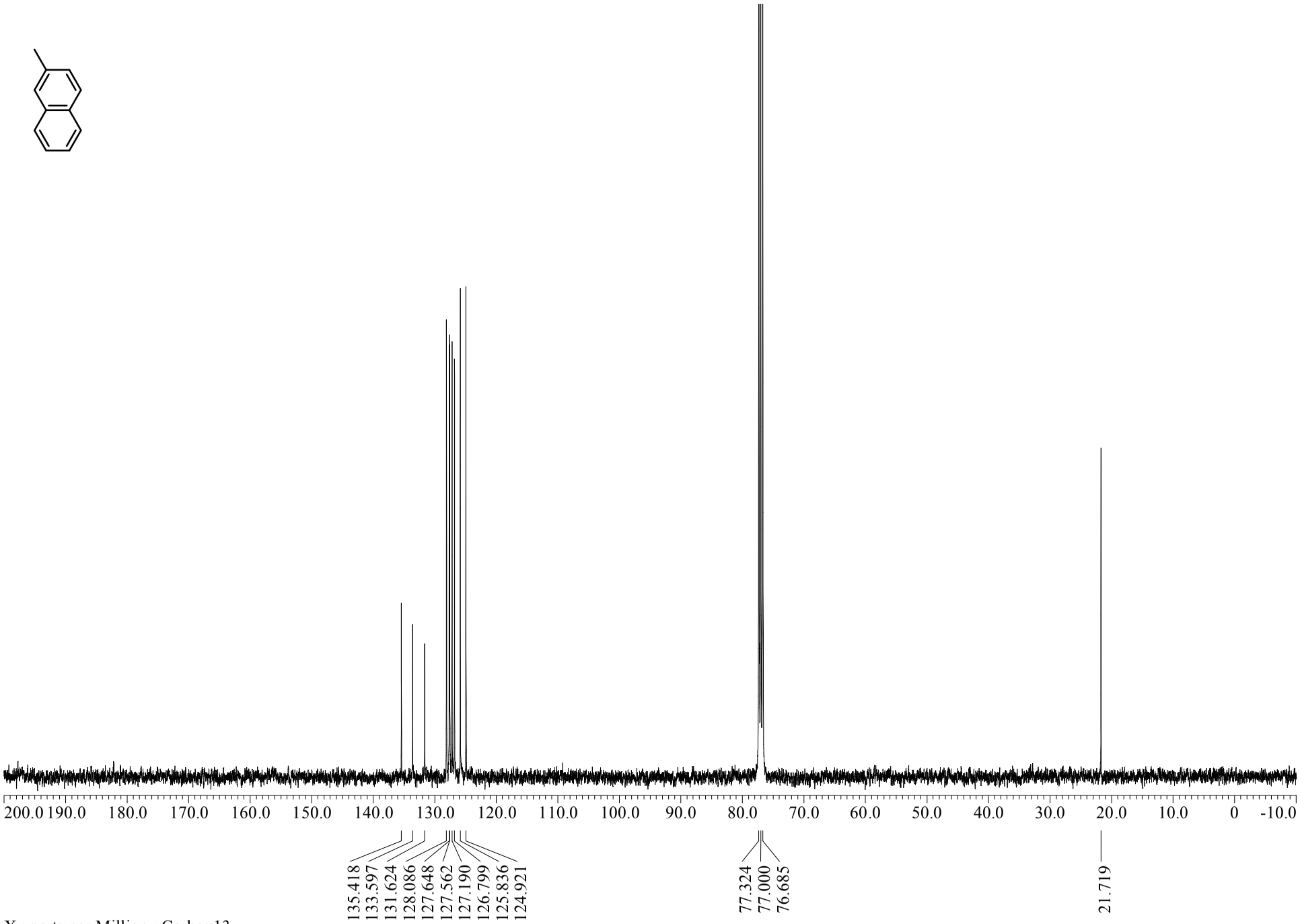

N
190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
150.235
148.099
135.914
129.316
129.288
128.115
127.648
126.389
120.916
77.324
77.000
76.685
X : parts per Million : Carbon13

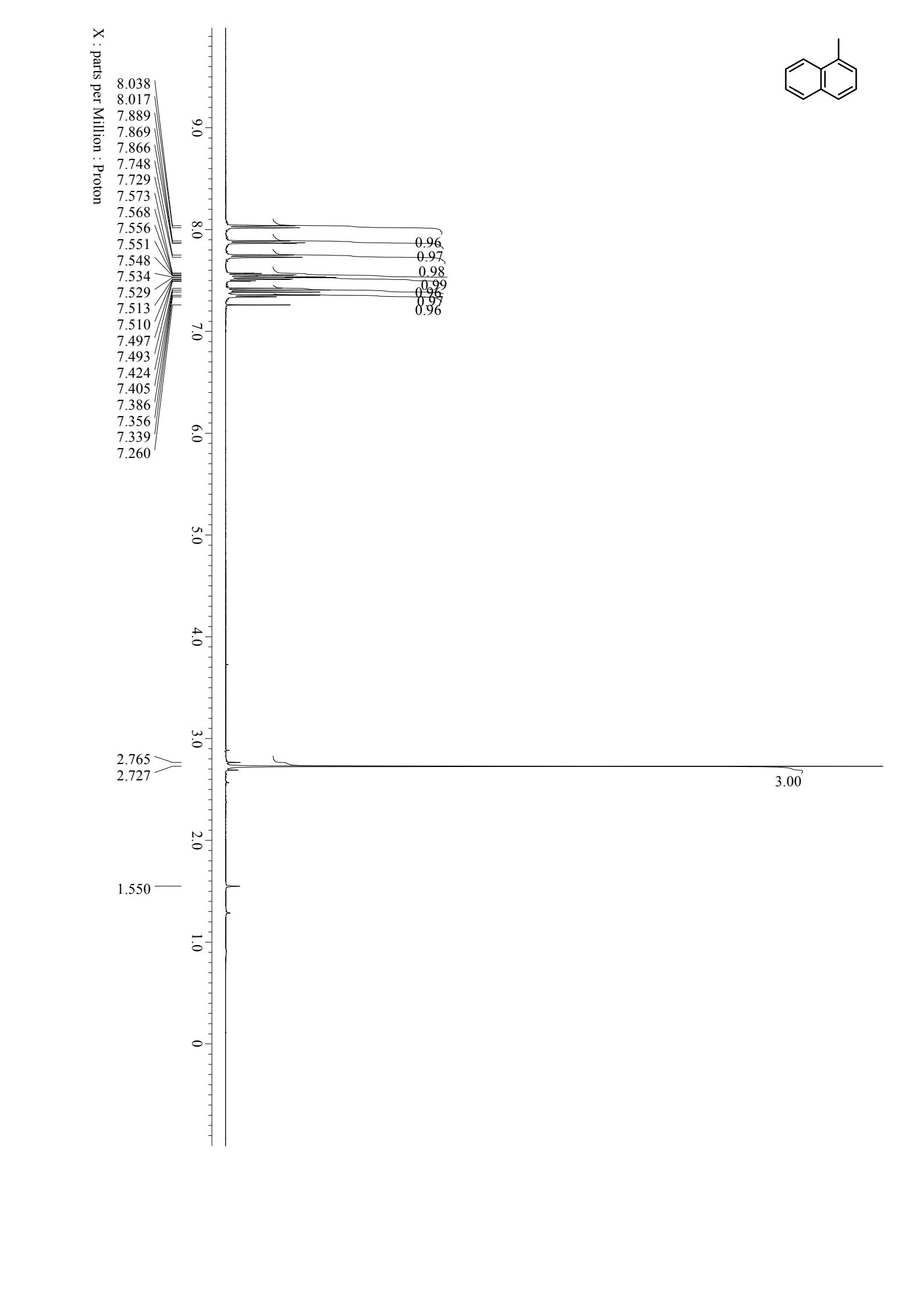


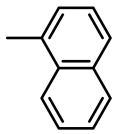
X : parts per Million : Proton


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
-10.0
140.920
125.617
122.699
120.258
118.771
108.378
77.324
77.000
76.685
28.993
X : parts per Million : Carbon13

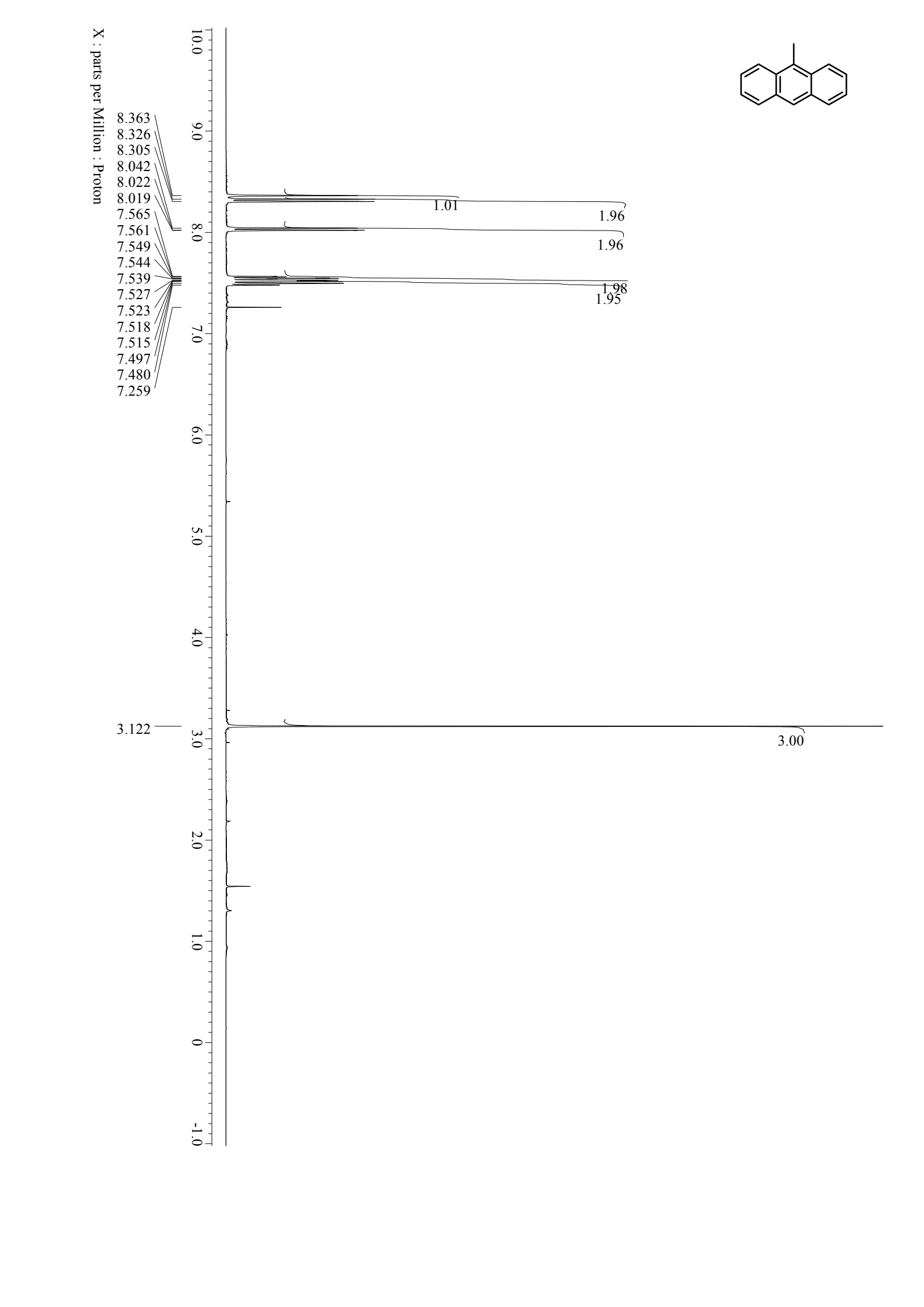

X : parts per Million : Proton
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
7.817
7.797
7.774
7.766
7.756
7.746
7.625
7.454
7.435
7.432
7.413
7.339
7.317
7.260
2.528
1.554
0.96
1.04
0.99
0.99
1.01
0.97
3.00

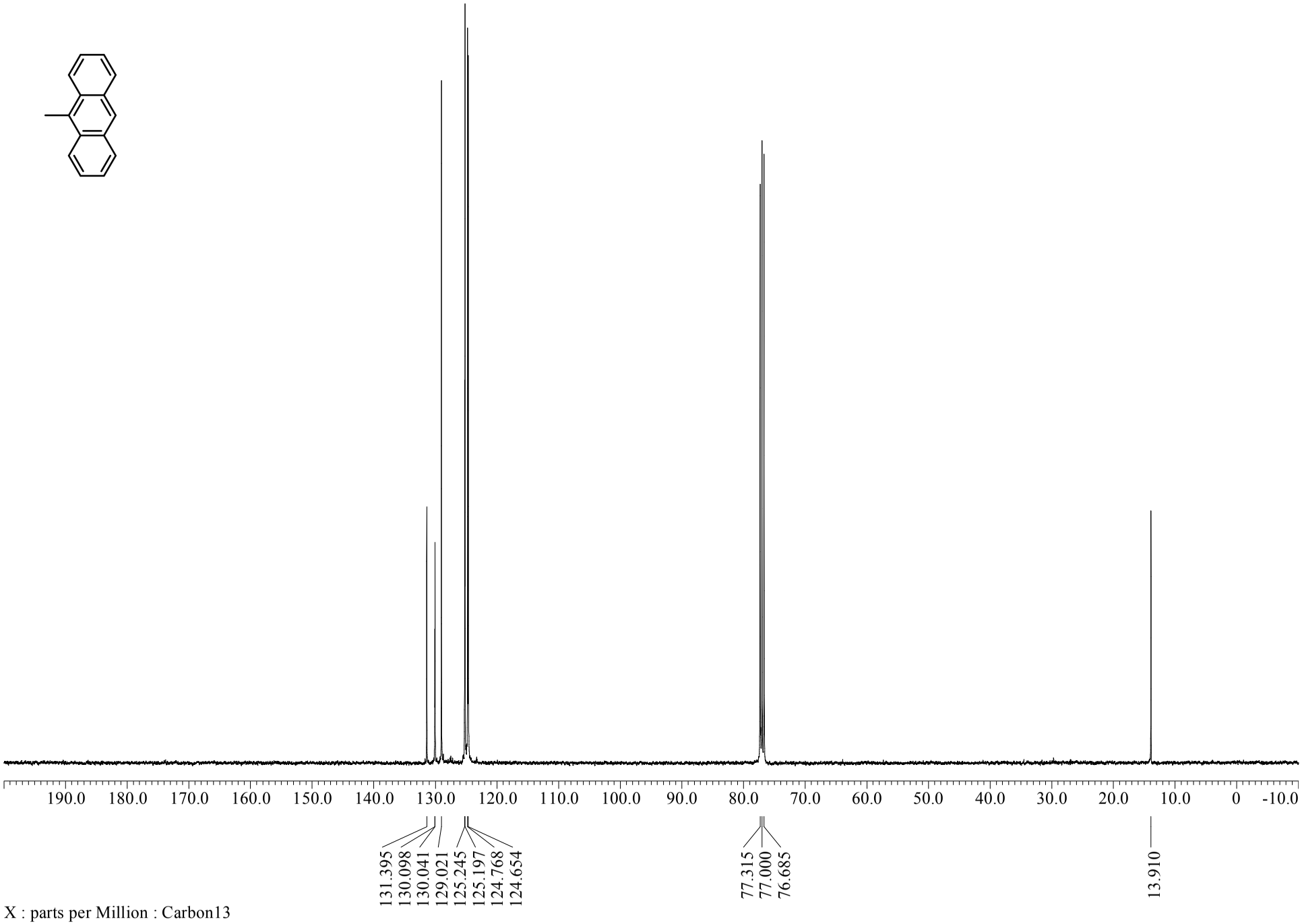

200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0
135.418
133.597
131.624
128.086
127.648
127.562
127.190
126.799
125.836
124.921
77.324
77.000
76.685
21.719
X : parts per Million : Carbon13


X : parts per Million : Proton
8.038
8.017
7.889
7.869
7.866
7.748
7.729
7.573
7.568
7.556
7.551
7.548
7.534
7.529
7.513
7.510
7.497
7.493
7.424
7.405
7.386
7.356
7.339
7.260
0.96
0.97
0.98
0.99
0.96
0.97
0.96
2.765
2.727
1.550
3.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0



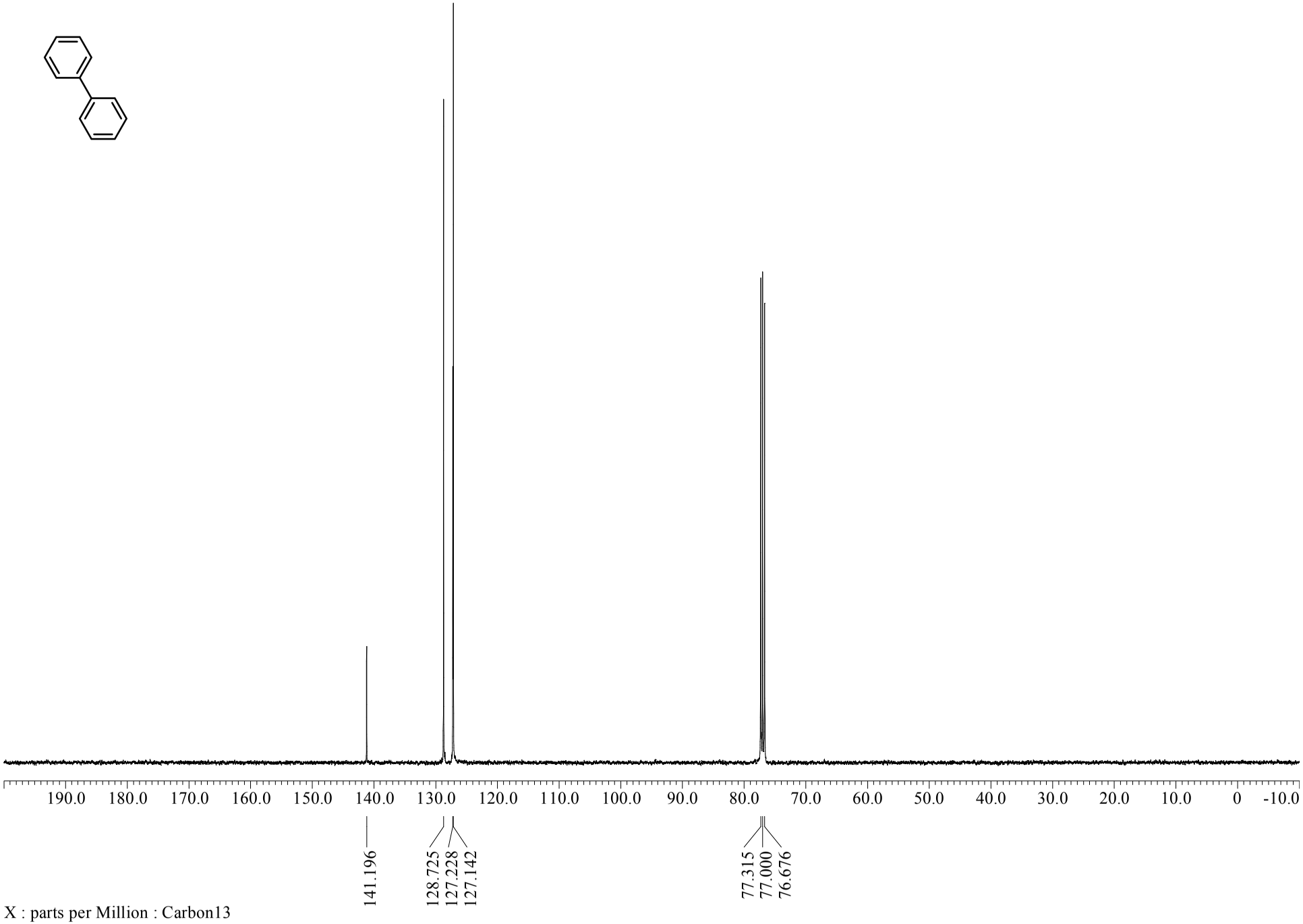
X : parts per Million : Carbon13

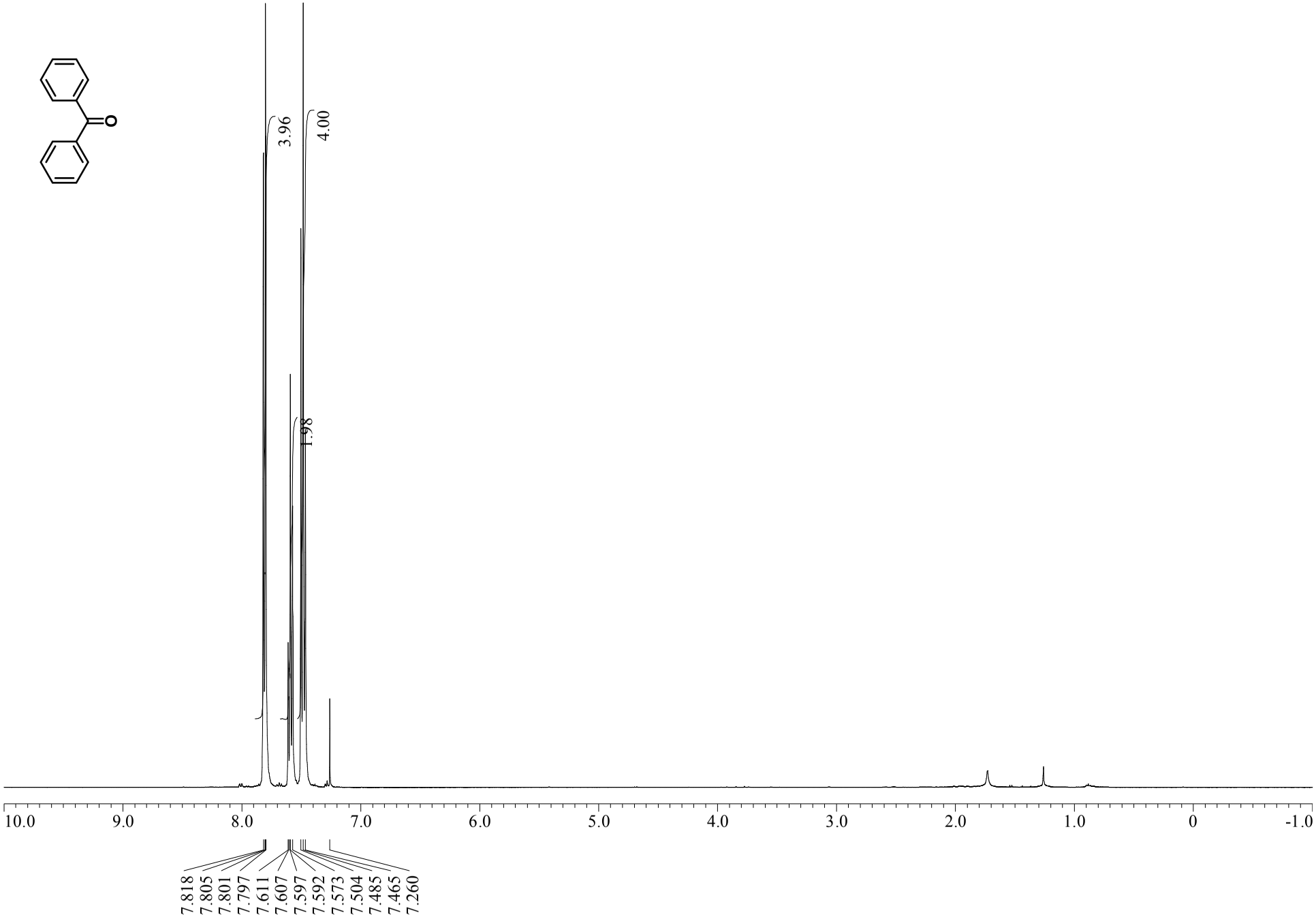

X : parts per Million : Proton
8.363
8.326
8.305
8.042
8.022
8.019
7.565
7.561
7.549
7.544
7.539
7.527
7.523
7.518
7.515
7.497
7.480
7.259
3.122
1.01
1.96
1.96
1.98
1.95
3.00
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0


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
-10.0
131.395
130.098
130.041
129.021
125.245
125.197
124.768
124.654
77.315
77.000
76.685
13.910
X : parts per Million : Carbon13

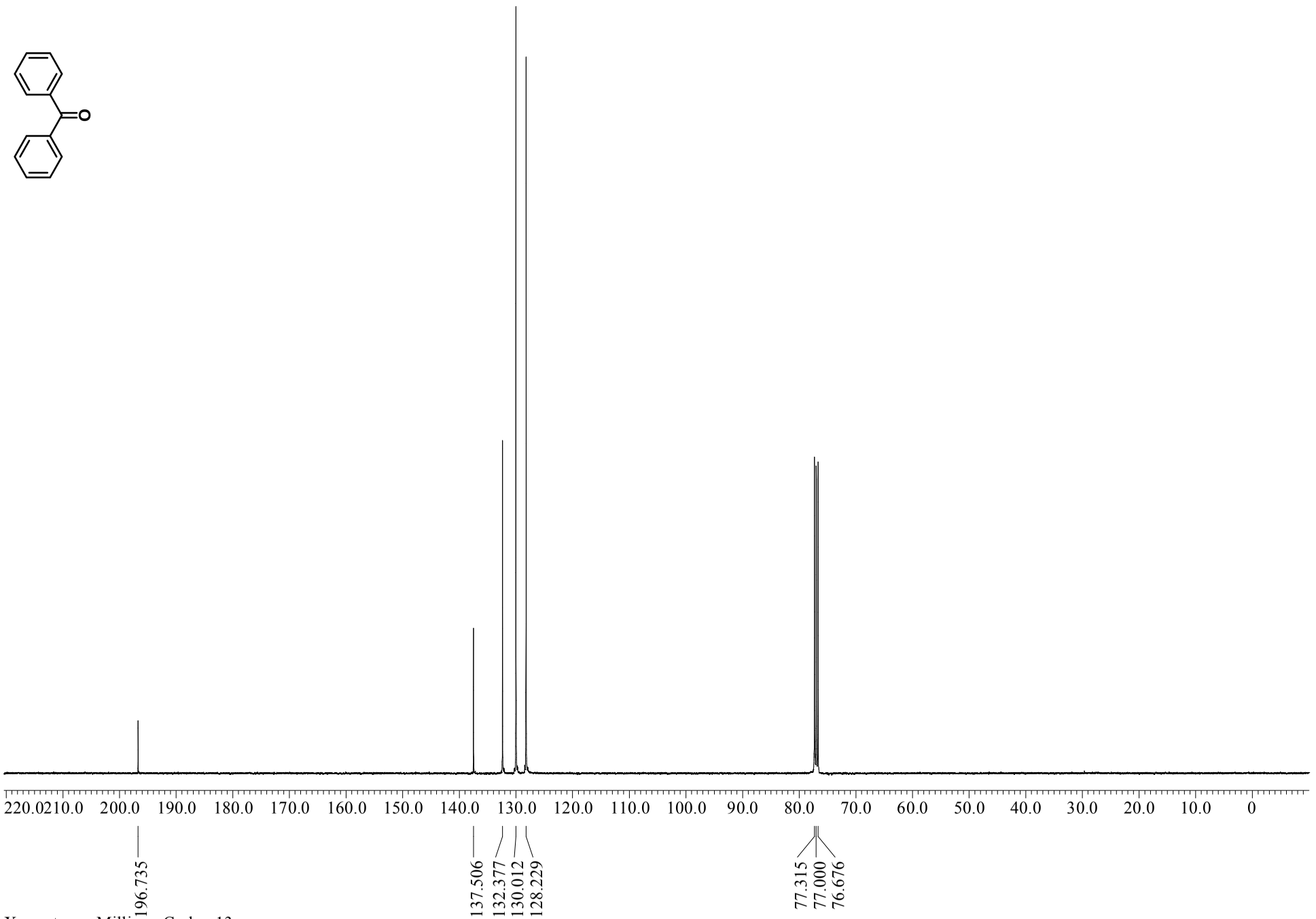
X : parts per Million : Proton

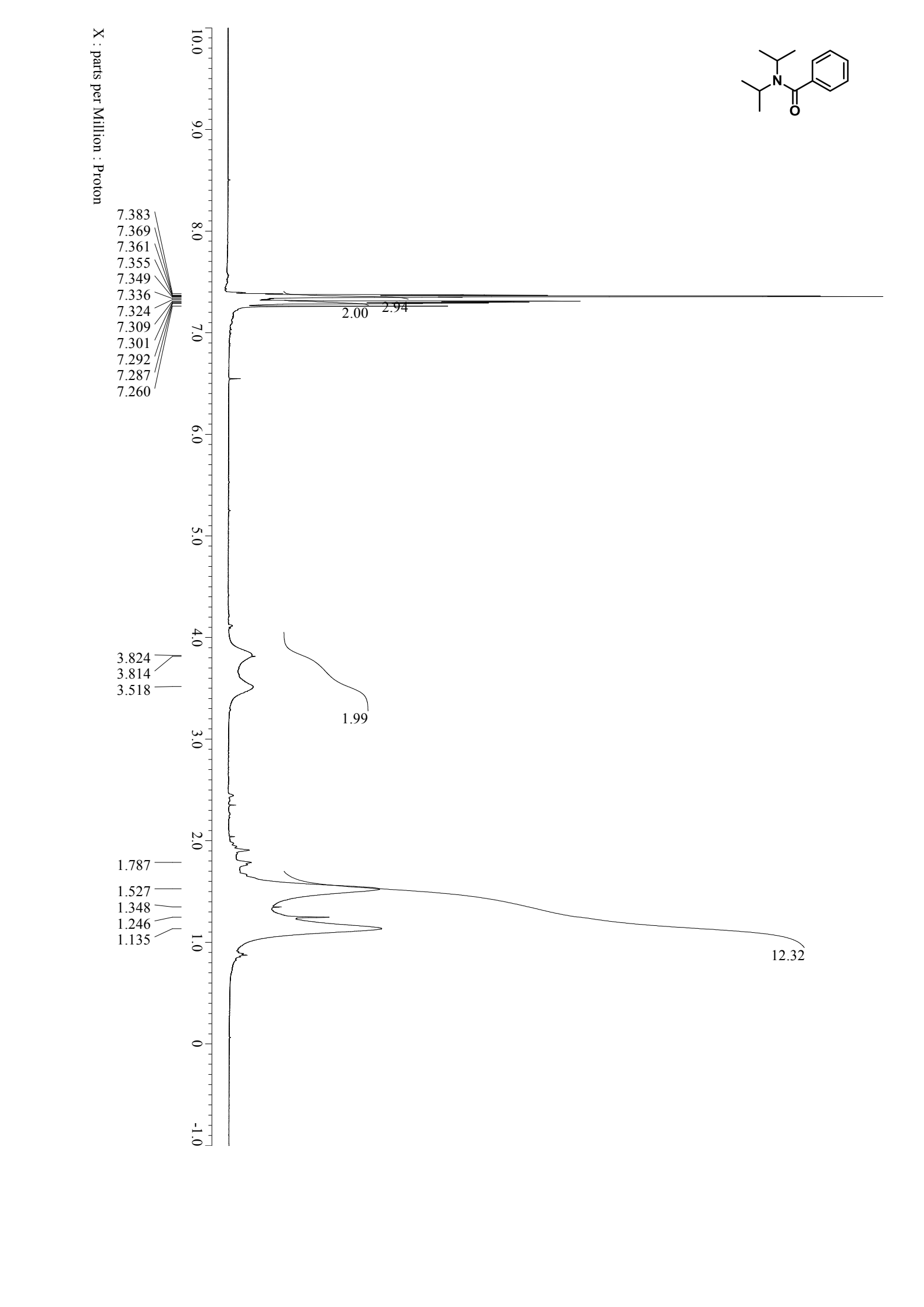
7.631
7.629
7.611
7.485
7.466
7.447
7.388
7.370
7.352
7.260
1.553
4.00
3.97
1.96
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0


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
-10.0
141.196
128.725
127.228
127.142
77.315
77.000
76.676
X : parts per Million : Carbon13



X : parts per Million : Proton


220.0210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
196.735
137.506
132.377
130.012
128.229
77.315
77.000
76.676
X : parts per Million : Carbon13


X : parts per Million : Proton
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
7.383
7.369
7.361
7.355
7.349
7.336
7.324
7.309
7.301
7.292
7.287
7.260
3.824
3.814
3.518
1.787
1.527
1.348
1.246
1.135
2.00
2.94
1.99
12.32
N
O

X : parts per Million : Carbon13
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
-10.0
170.973
138.898
128.553
128.391
125.521
77.315
77.000
76.676
50.828
45.708
20.679
N
O

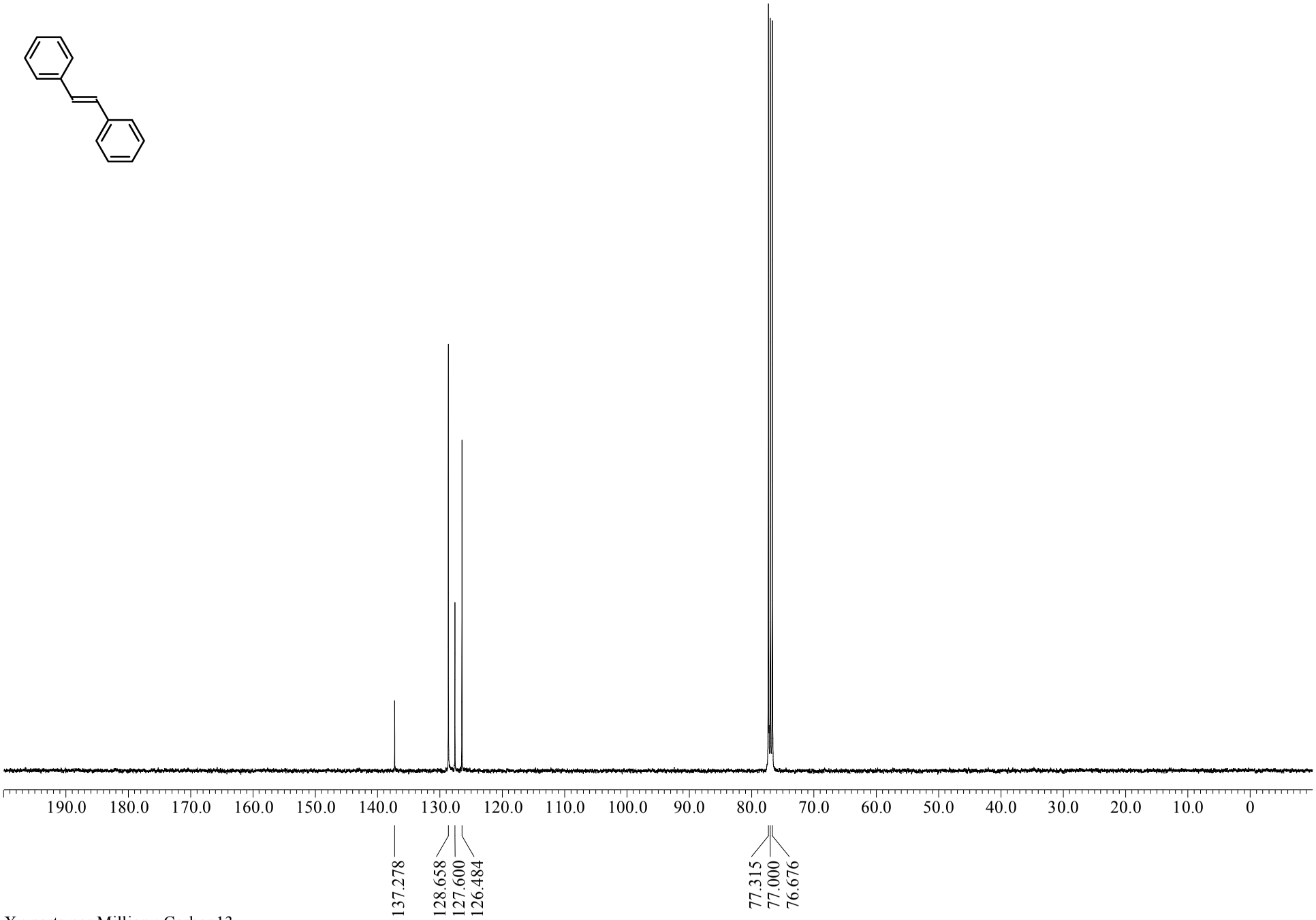
X : parts per Million : Proton

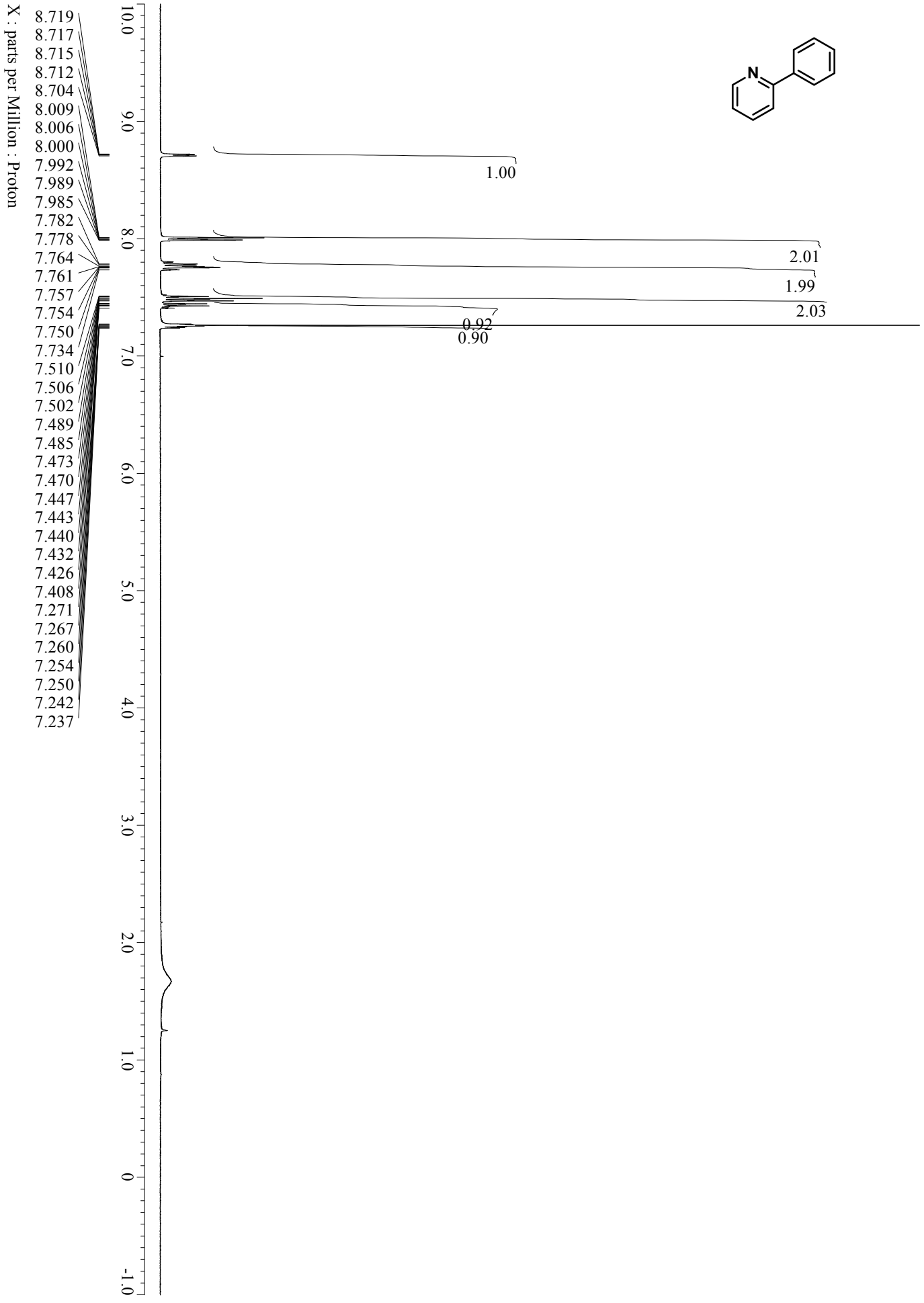
7.541
7.537
7.533
7.519
7.512
7.388
7.384
7.370
7.350
7.346
7.290
7.287
7.284
7.274
7.269
7.263
7.260
7.253
7.251
7.247
7.123

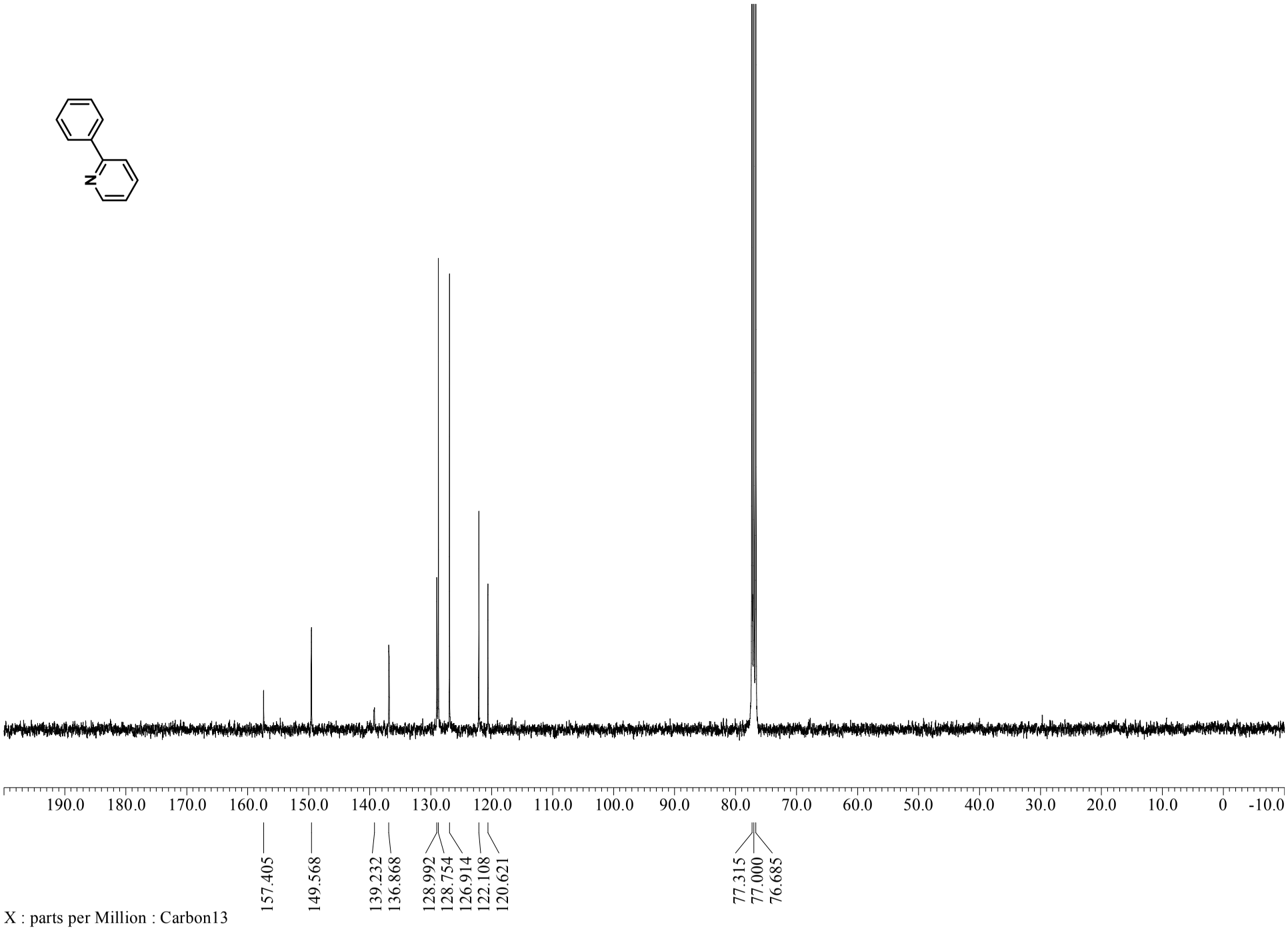
1.565

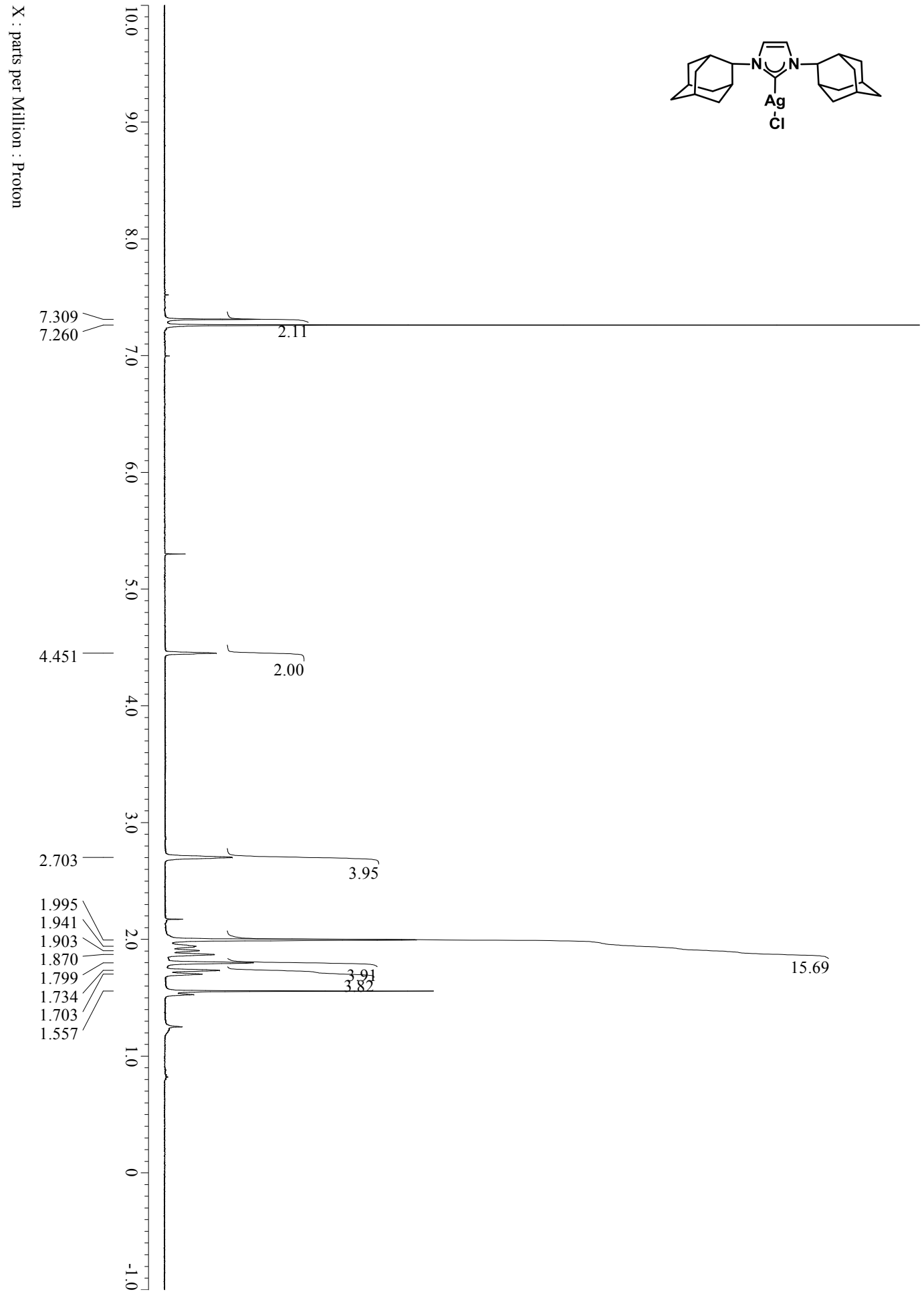
4.00
3.96
2.40
1.94

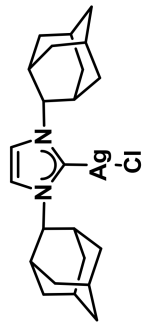
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0


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
137.278
128.658
127.600
126.484
77.315
77.000
76.676
X : parts per Million : Carbon13

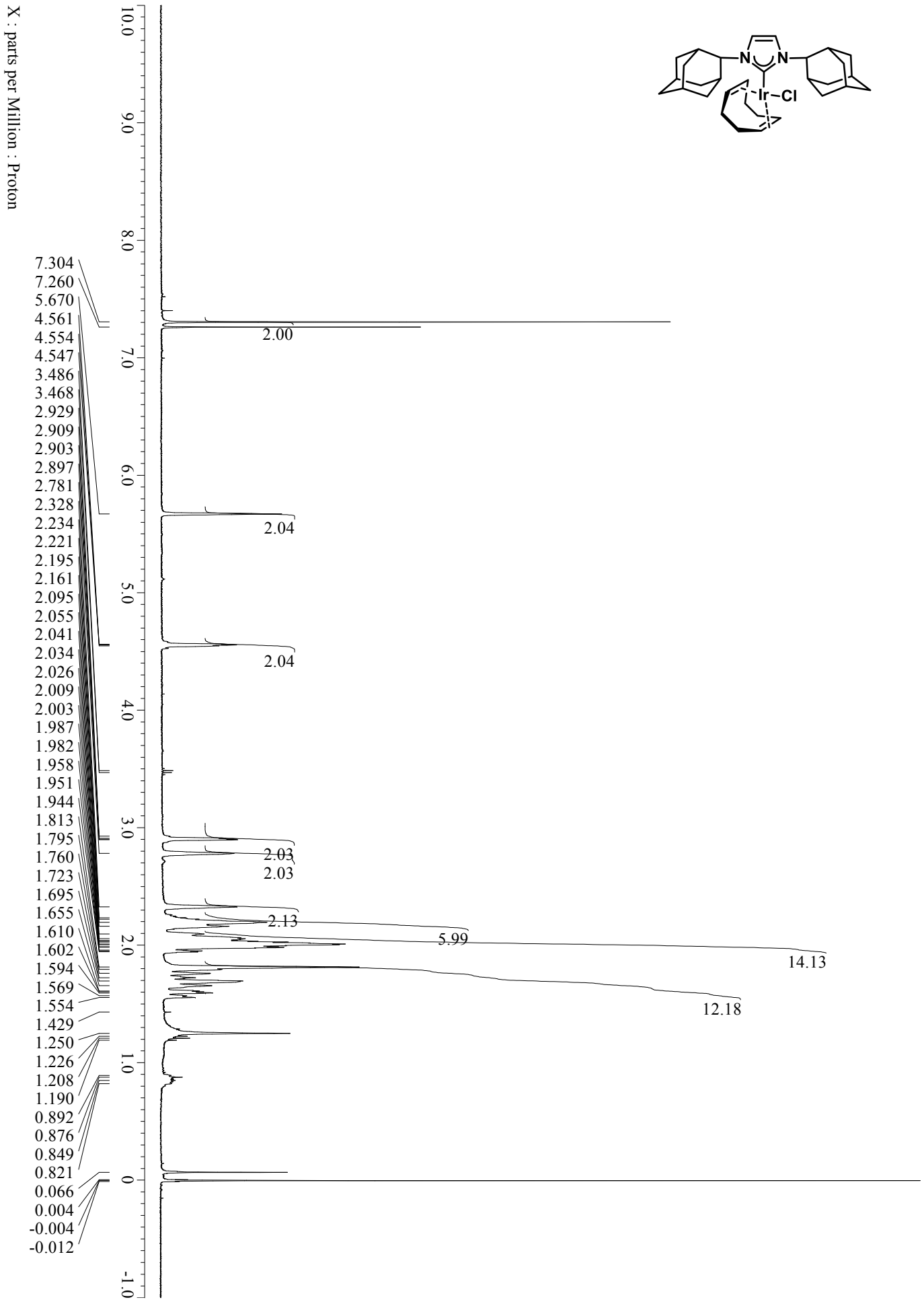

X : parts per Million : Proton
8.719
8.717
8.715
8.712
8.704
8.009
8.006
8.000
7.992
7.989
7.985
7.782
7.778
7.764
7.761
7.757
7.754
7.750
7.734
7.510
7.506
7.502
7.489
7.485
7.473
7.470
7.447
7.443
7.440
7.432
7.426
7.408
7.271
7.267
7.260
7.254
7.250
7.242
7.237
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
1.00
2.01
1.99
2.03
0.92
0.90
N

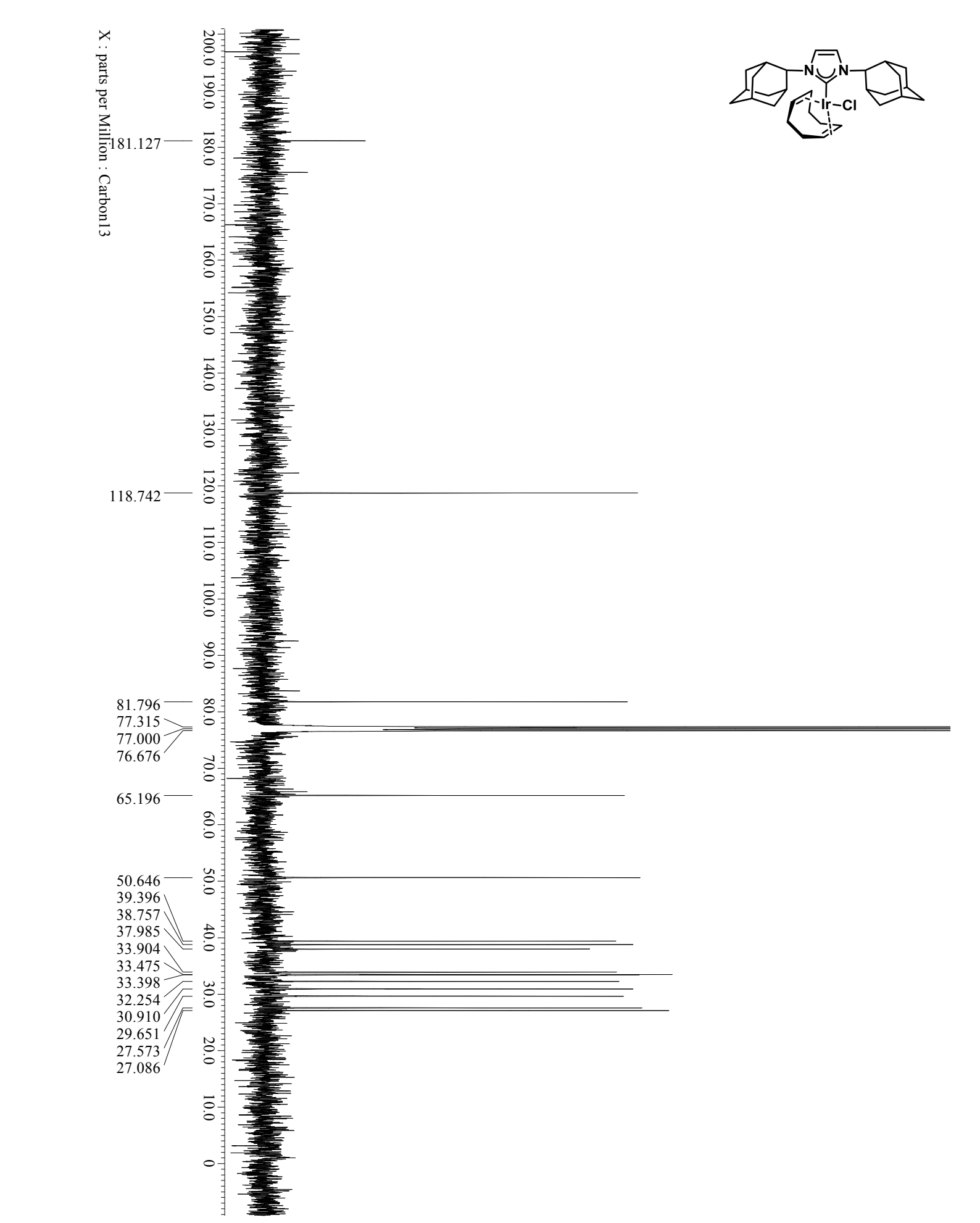

N
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
-10.0
157.405
149.568
139.232
136.868
128.992
128.754
126.914
122.108
120.621
77.315
77.000
76.685
X : parts per Million : Carbon13

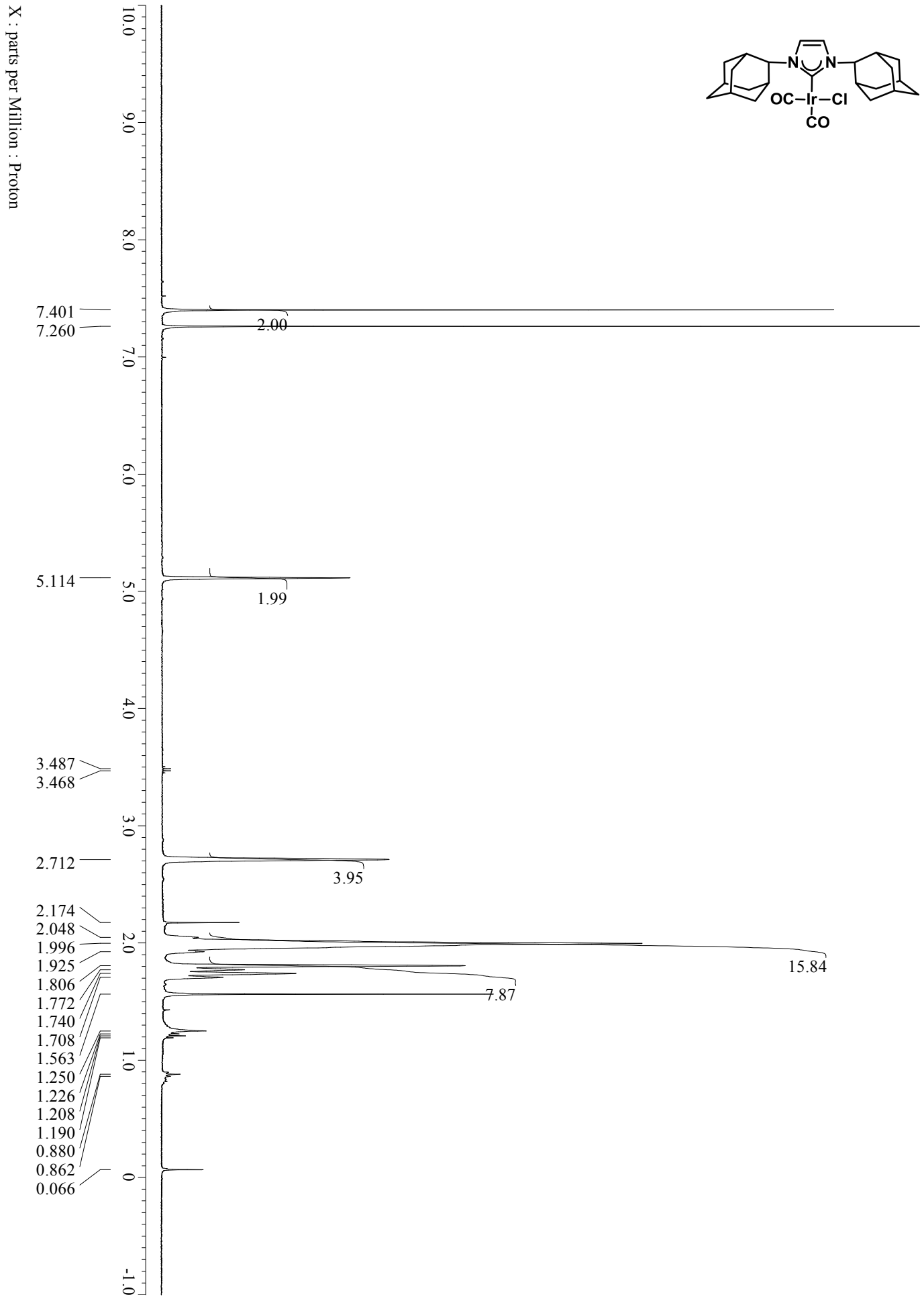

X : parts per Million : Proton
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
7.309
7.260
4.451
2.703
1.995
1.941
1.903
1.870
1.799
1.734
1.703
1.557
2.11
2.00
3.95
15.69
3.91
3.82
N
N
Ag
Cl

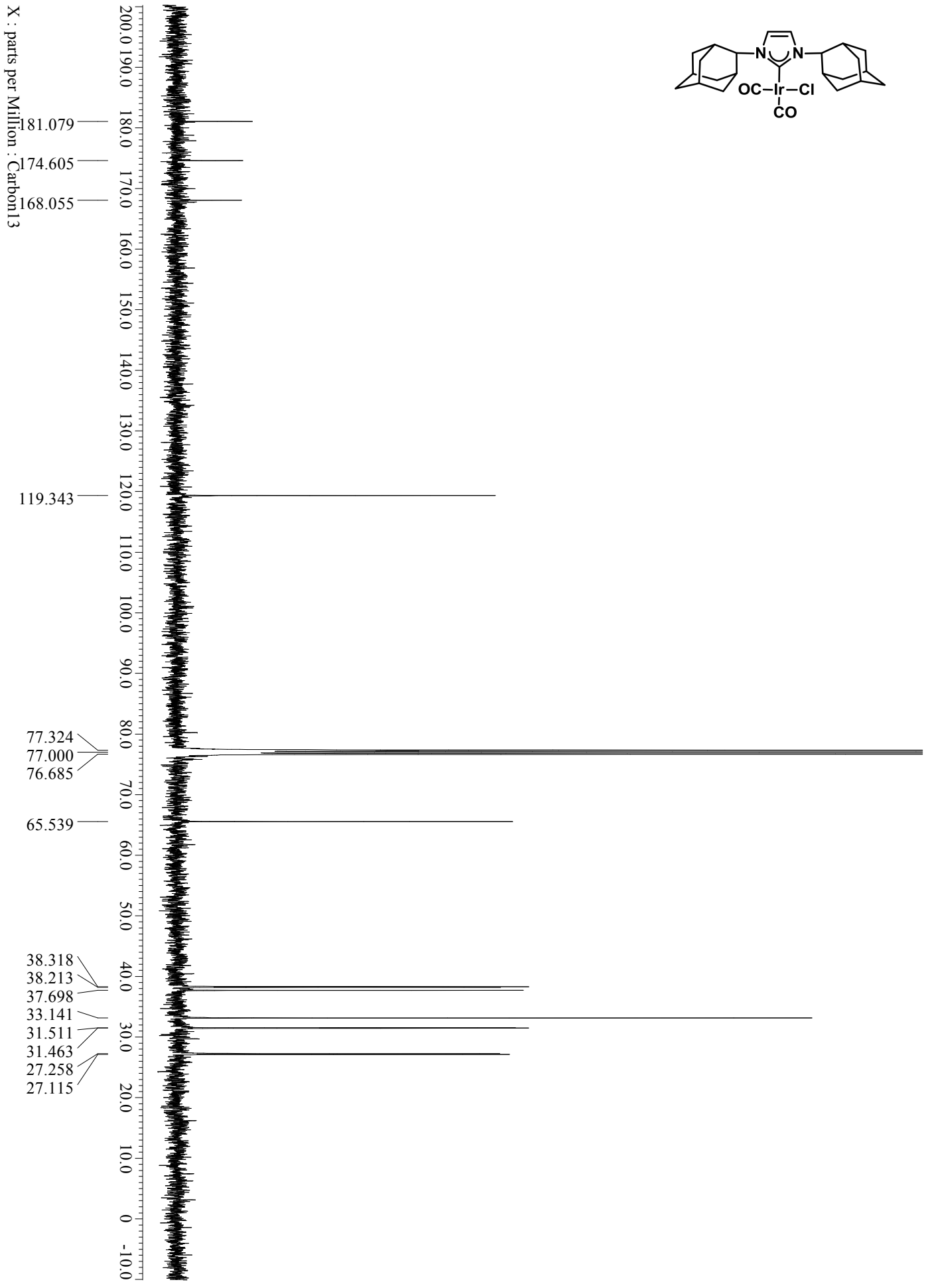


X : parts per Million : Carbon13


X : parts per Million : Proton
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
7.304
7.260
5.670
4.561
4.554
4.547
3.486
3.468
2.929
2.909
2.903
2.897
2.781
2.328
2.234
2.221
2.195
2.161
2.095
2.055
2.041
2.034
2.026
2.009
2.003
1.987
1.982
1.958
1.951
1.944
1.813
1.795
1.760
1.723
1.695
1.655
1.610
1.602
1.594
1.569
1.554
1.429
1.250
1.226
1.208
1.190
0.892
0.876
0.849
0.821
0.066
0.004
-0.004
-0.012
2.00
2.04
2.04
2.03
2.03
2.13
5.99
14.13
12.18
N
N
Ir
Cl


X : parts per Million : Carbon13
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
181.127
118.742
81.796
77.315
77.000
76.676
65.196
50.646
39.396
38.757
37.985
33.904
33.475
33.398
32.254
30.910
29.651
27.573
27.086
N
N
Ir
Cl


X : parts per Million : Proton
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
7.401
7.260
2.00
5.114
1.99
3.487
3.468
2.712
3.95
2.174
2.048
1.996
1.925
1.806
1.772
1.740
1.708
1.563
1.250
1.226
1.208
1.190
0.880
0.862
0.066
15.84
7.87
N
N
OC–Ir–Cl
CO


X : parts per Million : Carbon13
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 -10.0
181.079
174.605
168.055
119.343
77.324
77.000
76.685
65.539
38.318
38.213
37.698
33.141
31.511
31.463
27.258
27.115
N
N
OC–Ir–Cl
CO