Supplementary Information
for
Controlled Degradation of Chemically Stable Poly(aryl ethers) via Directing Group-Assisted Catalysis

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CONTENTS

I. General Information

^{1}H, ^{13}C, ^{11}B and ^{19}F NMR spectra were recorded on a JEOL ECS-400 spectrometer in $CDCl_3$. The chemical shifts in ^{1}H NMR spectra were recorded relative to $CHCl_3$ (δ 7.26) unless otherwise noted. The chemical shifts in ^{13}C NMR spectra were recorded relative to $CDCl_3$ (δ 77.0). The chemical shifts in ^{11}B NMR spectra were recorded relative to boron trifluoride diethyl etherate (δ 0.00). The chemical shifts in ^{19}F NMR spectra were recorded relative to hexafluorobenzene (δ –163.00). The data is reported as follows: chemical shift (δ) in ppm, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, and m = multiplet), coupling constant (Hz), and integration. Infrared spectra (IR) were obtained using a JASCO FT/IR-4200 spectrometer. Absorption is reported in reciprocal centimeters (cm^{-1}) with the following relative intensities: s (strong), m (medium), or w (weak). High resolution mass spectra (HRMS) were obtained using a JEOL JMS-T100LP spectrometer, JEOL JMS-700 or JEOL JMS-S3000. Analytical gas chromatography (GC) was carried out on a Shimadzu GC-2014 gas chromatograph, equipped with a flame ionization detector. Melting points were determined using a Yamato melting point apparatus. Column chromatography was performed with Biotage Isolera® equipped with Biotage® Sfär. Microwave synthesis was performed by Biotage® Initiator+. Analytical SEC was performed on a Hitachi High-Technologies Corporation L-2420/L-2130 equipped with a Shodex K-803L using chloroform ($CHCl_3$) as an eluent (40 °C, 1 ml/min) calibrated with polystyrene standards. Thermogravimetric analyses (TGA) were performed on a Hitachi High-Tech NEXTA STA200RV at 10 °C/min under N_2 flow. The glass transition temperatures (T_g) of the polymers were recorded on a Hitachi High-Tech NEXTA DSC2000.

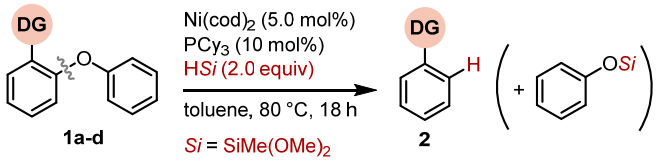
II. Materials

All commercially available reagents and solvents were supplied from TCI, WAKO, Aldrich and Strem Chemicals. $Ni(cod)_2$ was purchased from Strem Chemicals and used as received. PEEK (pellet forms) was purchased from Sigma–Aldrich Japan. 1,4-Bis((trimethylsilyl)oxy)benzene [CAS: 2117-24-0] was prepared from hydroquinone [CAS: 123-31-9] according to the literature method[1]. Ethyl 2-phenoxybenzoate [CAS: 41755-76-4][2], 2-(2-phenoxyphenyl)pyridine [CAS: 358741-46-5][3] and *N*,*N*-diethyl-2-phenoxybenzamide [CAS: 76279-43-1][4] were prepared according to the literature methods.

III. Effect of Directing Group in Ni-Catalyzed Reductive Cleavage of Model Substrate

Several functional groups have been tested as a directing group for use in Ni-catalyzed reductive cleavage of diphenyl ether derivatives.

A typical procedure: In a glovebox filled with nitrogen, diphenyl ether **1b** (58.3 mg, 0.24 mmol), $HSiMe(OMe)_2$ (54.8 mg, 0.52 mmol), $Ni(cod)_2$ (3.3 mg, 0.012 mmol), PCy_3 (6.7 mg, 0.024 mmol) and toluene (0.75 mL) were added to a 5 mL vial with a Teflon-sealed screwcap. The mixture was stirred at 80 °C for 18 h. The resulting mixture was then evaporated to dryness, and the residue was purified by flash column chromatography (eluent: hexane/EtOAc = 96/4 to 68/32) to give 2-phenylpyridine **2b** (74%).



Entry	DG	Yield of **2**[a] (%)	Recovered **1** (%)
1	H (**1a**)	0	89
2	2-pyridyl (**1b**)	74[b]	0
3	CO_2Et (**1c**)	54	0
4	$CONEt_2$ (**1d**)	27	48
5[c]	2-pyridyl (**1b**)	0	>99
6[d]	2-pyridyl (**1b**)	<5	>99
7[e]	2-pyridyl (**1b**)	<5	>99
8[f]	2-pyridyl (**1b**)	28	72

Fig. S1. Effect of directing group in Ni-catalyzed reductive cleavage of model substrate **1**.

[a]The yields were determined by GC analysis using dodecane as an internal standard.

[b]Isolated yield.

[c]Without $Ni(cod)_2$.

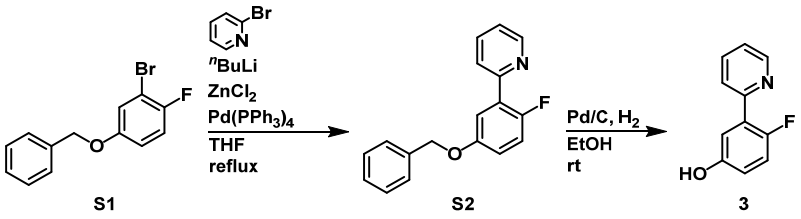
[d]Without PCy_3.

[e]Without $HSiMe(OMe)_2$.

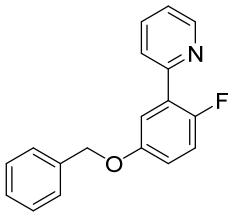
[f]At 40 °C.

IV. Preparation of Starting Materials

Synthesis of 4-fluoro-3-(pyridin-2-yl)phenol (3)



2-(5-(Benzyloxy)-2-fluorophenyl)pyridine (S2). This compound was synthesized using a literature procedure[5], with minor modifications. **S1**[6] (2.17 g, 7.7 mmol) was cooled to −78 ºC in THF (12 mL) under N_2. ^{n}BuLi (5.3mL of a 1.6 M solution in hexane, 8.5 mmol) was added dropwise and stirred for 30 min. $ZnCl_2$ (5.29 g, 38.5 mmol) was cannulated into the reaction mixture as a THF solution (90 mL), and the mixture was stirred for an additional 30 min. 2-bromopyridine (1.52 g, 9.6 mmol) and $Pd(PPh_3)_4$ (428 mg, 5 mol%) were dissolved in THF (12 mL) and cannulated into the reaction mixture. The reaction mixture was degassed then refluxed for 16 hours. The reaction mixture was cooled to rt and diluted with water. After the removing THF in vacuum, The aqueous phase was extracted with EtOAc. The combined extracts were dried over Na_2SO_4 and concentrated. The crude product was purified by flash column chromatography (eluent: hexane/EtOAc = 96/4 to 68/32) to give **S2** as a white solid (1.17 g, 54%).



White solid. R_f 0.43 (Hexane/EtOAc = 5/1). M.p. 99.4–100.2 ºC.

^{1}H NMR ($CDCl_3$, 400 MHz) δ: 8.72 (d, J = 4.6 Hz, 1H), 7.81–7.79 (m, 1H), 7.76–7.72 (m, 1H), 7.64 (dd, J =6.0, 3.1 Hz, 1H), 7.46–7.44 (m, 2H), 7.40–7.37 (m, 2H), 7.34–7.31 (m, 1H), 7.26–7.24 (m, 1H), 7.10–7.05 (m, 1H), 6.98–6.94 (m, 1H), 5.10 (s, 2H).

The chemical shifts in ^{1}H NMR spectra were recorded relative to tetramethylsilane (δ 0.00).

^{13}C NMR ($CDCl_3$, 101 MHz) δ: 155.1, 155.0 (d, J = 242.5 Hz), 153.1 (d, J = 2.9 Hz), 149.6, 136.8, 136.4, 128.5, 128.0, 127.7 (d, J = 13.4 Hz), 127.5, 124.5 (d, J = 10.5 Hz), 122.5, 117.3 (d, J = 8.6 Hz), 117.1 (d, J = 25.9 Hz), 115.5 (d, J = 2.9 Hz), 70.6.

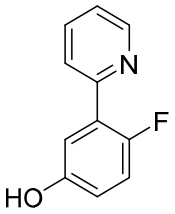
^{19}F NMR (CDCl3, 376 MHz) δ: −128.6 (s, 1F).

IR (KBr): 3071 w, 3033 w, 2950 w, 2876 w, 1584 s, 1569 m, 1501 s, 1460 s, 1442 s, 1406 s, 1383 s, 1328 s, 1292 m, 1269 m, 1254 m, 1240 m, 1222 s, 1195 s, 1006 s, 993 s, 904 m, 843 m, 816 s, 776 m, 754 s, 725 s, 696 s, 554 m.

MS, *m/z* (relative intensity, %): 280 (M^+, 6), 279 (31), 92 (19), 91 (100), 65 (22).

HRMS (DART+, $[M+H]^+$): Calcd for $C_{18}H_{15}NOF$: 280.1132. Found: 280.1137

4-Fluoro-3-(pyridin-2-yl)phenol (3). This compound was synthesized using a literature procedure[5], with minor modifications. **S2** (2.31 g, 8.3 mmol), Pd/C (2.02 g, 0.83 mmol) and EtOH were added to a 500 mL round-bottom flask. The reaction mixture was flushed with H_2 and stirred for 16 h, then filtered through celite and rinsed with EtOAc. After removing the solvent in vacuo, the crude product was purified by flash column chromatography (eluent: hexane/EtOAc = 90/10 to 20/80) to give **3** as a white solid (1.34 g, 86%).



White solid. R_f 0.50 (Hexane/EtOAc = 1/1). M.p. 150.0–151.1 ºC.

^{1}H NMR ($CDCl_3$, 400 MHz) δ: 8.71–8.69 (m, 1H), 7.82–7.77 (m, 2H), 7.52–7.49 (m, 1H), 7.33–7.28 (m, 1H), 7.05–7.00 (m, 2H), 6.84 (dt, J = 8.7, 3.6 Hz, 1H).

The chemical shifts in ^{1}H NMR spectra were recorded relative to tetramethylsilane (δ 0.00).

^{13}C NMR ($CDCl_3$, 101 MHz) δ: 154.0 (d, J = 241.5 Hz), 153.2 (d, J = 1.9 Hz), 153.1 (d, J = 1.9 Hz), 149.0, 137.3, 126.7 (d, J = 14.4 Hz), 125.3 (d, J = 8.6 Hz), 122.8, 118.0 (d, J = 1.9 Hz), 117.1 (d, J = 24.9 Hz), 117.0.

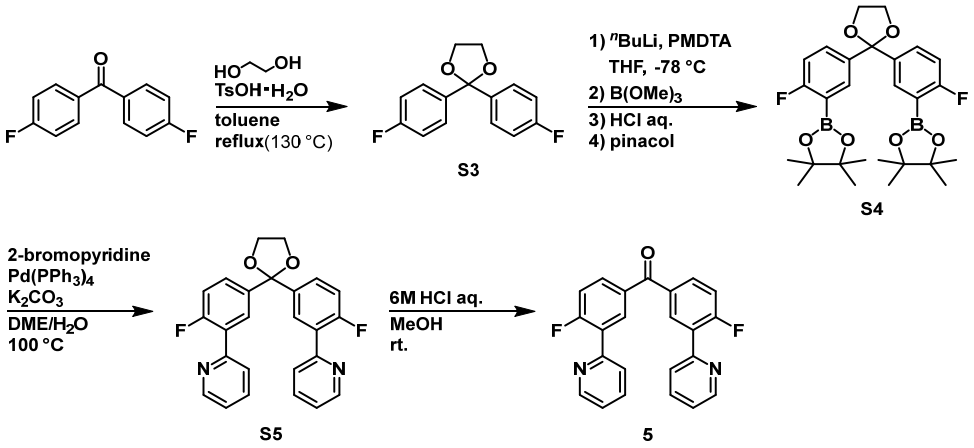
^{19}F NMR (CDCl3, 376 MHz) δ: −130.3 (s, 1F).

IR (KBr): 3081 br, 1615 m, 1593 s, 1566 m, 1508 s, 1471 s, 1454 s, 1423 s, 1354 s, 1327 m, 1256 m, 1237 s, 1227 s, 1202 s, 1155 m, 1059 m, 1001 m, 869 m, 828 m, 799 m, 792 m, 763 s, 751 s, 728 s, 714 m, 627 m, 611 m.

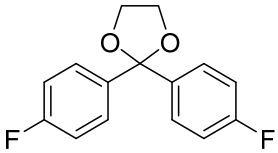
MS, *m/z* (relative intensity, %): 190 (14), 189 (M^+,100), 161 (43), 160 (13), 135 (30), 133 (14).

HRMS (DART+, $[M+H]^+$): Calcd for $C_{11}H_9NOF$: 190.0663. Found: 190.0665

Synthesis of bis(4-fluoro-3-(pyridin-2-yl)phenyl)methanone (5)



2,2-Bis(4-fluorophenyl)-1,3-dioxolane (S3). CAS[1616861-44-9] This compound was synthesized using a literature procedure[7], with minor modifications. 4,4'-Difluorobenzophenone (21.3 g, 97.7 mmol), ethylene glycol (27.6 g, 444.1 mmol), *p*-toluenesulfonic acid monohydrate (190 mg, 1.1 mmol) and toluene (100 mL) were added to a 300 mL round-bottom flask. The mixture was refluxed for 2 days with a Dean–Stark apparatus for azeotropic removal of H_2O. The resulting mixture was cooled to rt and washed with saturated aqueous solution of $NaHCO_3$ and with water. The aqueous phase was extracted with Et_2O twice. After drying the combined organic extracts with Na_2SO_4 and removing the solvent in vacuo, **S3** was obtained as a colorless oil (25.3 g, 99%). This compound was used in the next step without further purification.



Colorless oil. R_f 0.62 (Hexane/EtOAc = 1/1).

^{1}H NMR ($CDCl_3$, 400 MHz) δ: 7.48–7.44 (m, 4H), 7.04–6.99 (m, 4H), 4.06 (s, 4H).

^{13}C NMR ($CDCl_3$, 101 MHz) δ: 162.6 (d, J = 247.3 Hz), 137.7 (d, J = 2.9 Hz), 128.1 (d, J = 7.7 Hz), 115.0 (d, J = 21.1 Hz), 108.7, 64.9.

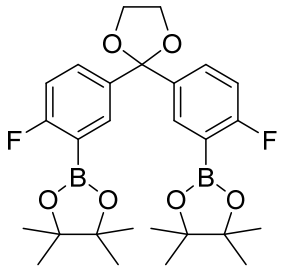
^{19}F NMR ($CDCl_3$, 376 MHz) δ: −115.4.

IR (KBr): 2979 w, 2893 w, 1604 m, 1507 s, 1269 w, 1223 s, 1155 m, 1080 s, 1014 m, 992 m, 946 m, 834 s, 602 w, 577 s, 545 w.

MS, *m/z* (relative intensity, %): 262 (M^+, 14), 231 (18), 202 (14), 201 (54), 190 (31), 168 (73), 167 (100), 124 (28), 123 (100), 95 (94), 75 (31).

HRMS (DART+, $[M+H]^+$) Calcd for $C_{15}H_{13}O_2F_2$: 263.0878. Found: 263.0878.

2,2'-((1,3-Dioxolane-2,2-diyl)bis(6-fluoro-3,1-phenylene))bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (S4). This compound was synthesized using a literature procedure[8,9,10], with minor modifications. *N*,*N*,*N'*,*N''*,*N''*-pentamethyldiethylenetriamine (PMDTA) (2.9 mL, 13.8 mmol) was added at −78 ºC under N_2 to ^{n}BuLi (8.6 mL of a 1.6 M solution in hexane, 13.8 mmol) followed by dropwise addition of 2,2-bis(4-fluorophenyl)-1,3-dioxolane (1.49 g, 5.7 mmol) in THF (6 mL). The reaction mixture was stirred at −78 ºC for 1 h, then $B(OMe)_3$ (4.67 g, 44.9 mmol) was slowly added. The reaction mixture was warmed to rt and then stirred for 19 h. THF (18 mL) was added, then the reaction mixture was stirred for an additional 1 h. To the resulting mixture, water (10 mL) was added, followed by aqueous HCl (5%, 25 mL). The reaction mixture was stirred for 4 h. THF was removed under reduced pressure. The remaining aqueous phase was extracted with EtOAc. After drying the organic phase with Na_2SO_4 and removing the solvent in vacuo, the boronic acid product was obtained as a white solid. Pinacol (1.78 g, 15.1 mmol) was added to a stirred solution of this boronic acid in hexane (20 mL) and the reaction mixture was stirred for 4 h at rt. The resulting hexane solution was dried with over Na_2SO_4, filtered with EtOAc, and the solvent was removed in vacuo. The crude product was purified by flash column chromatography (eluent: hexane/EtOAc = 100/0 to 75/25) to give **S4** as a white solid (1.17 g, 40%).



White solid. R_f 0.58 (Hexane/EtOAc = 1/1). M.p. 143.8–145.1 ºC.

^{1}H NMR ($CDCl_3$, 400 MHz) δ: 7.91 (dd, J = 5.5, 2.3 Hz, 2H), 7.52–7.48 (m, 2H), 6.97 (t, J = 8.9 Hz, 2H), 4.05 (s, 4H), 1.35 (s, 24H).

^{13}C NMR ($CDCl_3$, 101 MHz) δ: 167.0 (d, J = 252.1 Hz), 137. 2 (d, J = 2.9 Hz), 134.36 (d, J = 8.6 Hz), 131.5 (d, J = 9.6 Hz), 115.06 (d, J = 24.9 Hz), 108.8, 83.9, 64.9, 24.8.

^{19}F NMR ($CDCl_3$, 376 MHz) δ: −103.9.

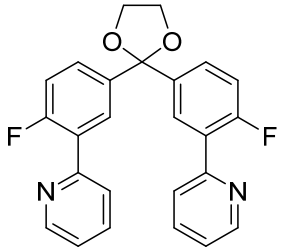
^{11}B NMR ($CDCl_3$, 128 MHz) δ: 30.1.

IR (KBr): 2979 m, 2893 w, 1614 s, 1585 w, 1488 m, 1419 s, 1372 s, 1346 s, 1328 s, 1289 m, 1269 m, 1254 m, 1215 s, 1200 s, 1144 s, 1091 m, 1069 s, 998 m, 962 m, 851 m, 743 w, 673 w, 607 m.

MS, *m/z* (relative intensity, %): 514 (M^+, 3), 294 (41), 293 (100), 292 (65), 249 (14), 193 (11), 149 (13), 101 (14), 83 (16), 43 (15).

HRMS (DART+, $[M+H]^+$) Calcd for $C_{27}H_{35}O_6B_2F_2$: 515.2582. Found: 515.2607.

2,2'-((1,3-Dioxolane-2,2-diyl)bis(6-fluoro-3,1-phenylene))dipyridine (S5). This compound was synthesized using a literature procedure[11], with minor modifications. To a 50 mL two-necked flask, 2-bromopyridine (1.09 g, 6.9 mmol), $Pd(PPh_3)_4$ (261 mg, 9.8 mol%), K_2CO_3 (967 mg, 7.0 mmol), 2,2'-((1,3-dioxolane-2,2-diyl)bis(6-fluoro-3,1-phenylene))bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (1.17 g, 2.3 mmol), water (8 mL) and DME (8 mL) were added. The reaction mixture was degassed then refluxed for 19 h. The reaction mixture was cooled to rt and diluted with H_2O and extracted with EtOAc. The combined extracts were dried over Na_2SO_4 and concentrated. The crude product was purified by flash column chromatography (eluent: hexane/EtOAc = 50/50 to 10/90) to give **S5** as a yellow oil (669 mg, 71%).



Yellow oil. R_f 0.20 (Hexane/EtOAc = 1/1).

^{1}H NMR ($CDCl_3$, 400 MHz) δ: 8.72 (td, J = 3.0, 1.5 Hz, 2H), 8.18 (dd, J = 7.6, 2.5 Hz, 2H), 7.74–7.72 (m, 4H), 7.55–7.51 (m, 2H), 7.26–7.22 (m, 2H), 7.12 (dd, J = 11.0, 8.7 Hz, 2H), 4.10 (s, 4H).

^{13}C NMR ($CDCl_3$, 101 MHz) δ: 160.1 (d, J = 250 Hz), 153.3, 149.7, 138.4 (d, J = 3.8 Hz), 136.2, 128.8 (d, J = 2.9 Hz), 128.5 (d, J = 8.6 Hz), 127.2 (d, J = 12.5 Hz), 124.5 (d, J = 8.6 Hz), 122.4, 116.0 (d, J = 24.0 Hz), 108.6, 65.1.

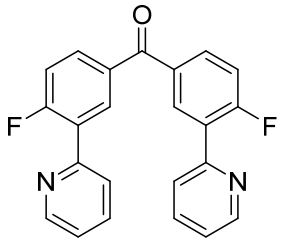
^{19}F NMR ($CDCl_3$, 376 MHz) δ: −118.2.

IR (KBr): 3055 m, 3006 m, 2895 m, 1611 m, 1587 s, 1567 m, 1496 s, 1463 s, 1441 m, 1407 m, 1295 m, 1250 m, 1212 m, 1184 m, 1089 m, 1061 m, 1038 m, 1013 m, 996 s, 946 m, 907 m, 828 s, 790 s, 746 s, 648 m, 638 m, 627 m, 614 m, 474 m.

MS, *m/z* (relative intensity, %): 416 (M^+, 2), 245 (19), 244 (100), 200 (21), 172 (20).

HRMS (DART+, $[M+H]^+$) Calcd for $C_{25}H_{19}N_2O_2F_2$: 417.1409. Found: 417.1414.

Bis(4-fluoro-3-(pyridin-2-yl)phenyl)methanone (5). This compound was synthesized using a literature procedure[12], with minor modifications. An aqueous HCl (10%, 2 mL) was added to a solution of 2,2'-((1,3-dioxolane-2,2-diyl)bis(6-fluoro-3,1-phenylene))dipyridine (160 mg, 0.38 mmol) in MeOH (2 mL) and the mixture was stirred at rt for 16 h. An aqueous solution of NaOH (10%, 2 mL) was added to the reaction mixture, and the aqueous layer was extracted with EtOAc. The combined extracts were dried over Na_2SO_4 and concentrated. The crude product was purified by flash column chromatography (eluent: hexane/EtOAc = 80/20 to 50/50) to give **5** as a white solid (132 mg, 92%).



White solid. R_f 0.40 (Hexane/EtOAc = 1/1). M.p. 139.2–139.9 ºC.

^{1}H NMR ($CDCl_3$, 400 MHz) δ: 8.72–8.71 (m, 2H), 8.45 (dd, J = 7.8, 2.3 Hz, 2H), 7.90–7.86 (m, 2H), 7.83–7.76 (m, 4H), 7.32–7.27 (m, 4H).

^{13}C NMR ($CDCl_3$, 101 MHz) δ: 193.7, 162.9 (d, J = 257.8 Hz), 152.3, 149.9, 136.5, 134.1 (d, J = 3.8 Hz), 133.5 (d, J = 4.8 Hz), 132.2 (d, J = 9.6 Hz), 127.8 (d, J = 12.5 Hz), 124.4 (d, J = 8.6 Hz), 122.8, 116.6 (d, J = 24.0 Hz).

^{19}F NMR ($CDCl_3$, 376 MHz) δ: −111.8.

IR (KBr): 3079 w, 3056 w, 3005 w, 1657 s, 1604 m, 1588 s, 1570 m, 1497 m, 1463 s, 1442 m, 1392 m, 1328 m, 1306 m, 1294 m, 1285 m, 1254 s, 1220 s, 1139 m, 1119 m, 1061 m, 971 m, 930 m, 842 s, 788 s, 761 s, 752 s, 742 s, 706 m, 607 s.

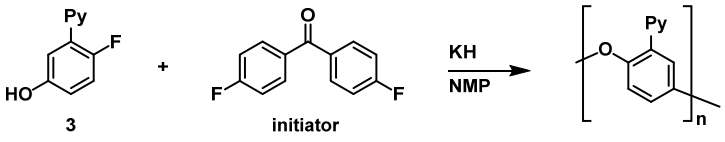
MS, *m/z* (relative intensity, %): 373 (15), 372 (M^+, 59), 371 (37), 352 (20), 351 (14), 343 (14), 295 (22), 294 (100), 200 (18), 172 (38), 145 (10), 125 (14), 78 (11).

HRMS (DART+, $[M+H]^+$): Calcd for $C_{23}H_{15}N_2OF_2$: 373.1147. Found: 373.1152.

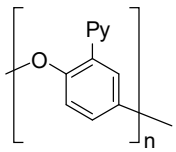
V. Synthesis and Characterization of DG-PPE, DG-PEEK1 and DG-PEEK2

V-1. Synthesis of DG-polymers

Synthesis of DG-PPE



This compound was synthesized using a literature procedure[13], with minor modifications. In a glovebox filled with nitrogen, **3** (764.3 mg, 4.0 mmol), 4,4'-difluorobenzophenone (5.2 mg, 0.024 mmol), KH (168.0 mg, 4.2 mmol) and NMP (2.7 mL) were added to a 5 mL pressure-resistant vial and it was closed with a resealable septum. The mixture was stirred at 200 °C for 4 h using a microwave reactor. After cooling to rt, the polymer is precipitated by adding the viscous reaction mixture slowly to vigorously stirred MeOH. The polymer is purified by stirring with acetone. After drying in a vacuum oven, **DG-PPE** was obtained as a white powder (426.2 mg, 62%).



M_w = 4980, M_w/M_n = 3.01.

^{1}H NMR ($CDCl_3$, 400 MHz) δ: 8.62 (s, 1H), 7.87 (d, J = 7.8 Hz, 1H), 7.63 (t, J = 7.1 Hz, 1H), 7.57 (s, 1H), 7.17−7.16 (m, 1H), 6.96 (d, J = 8.7 Hz, 1H), 6.86 (dd, J = 8.7, 2.7 Hz, 1H).

The chemical shifts in ^{1}H NMR spectra were recorded relative to tetramethylsilane (δ 0.00).

^{13}C NMR ($CDCl_3$, 101 MHz) δ: 154.2, 153.5, 149.8, 149.5, 136.1, 132.9, 124.9, 122.3, 121.0, 120.6, 119.6.

IR (ATR): 2359 w, 2340 w, 1585 m, 1567 w, 1483 s, 1458 s, 1442 m, 1399 m, 1311 w, 1289 w, 1235 m, 1197 s, 1176 s, 1119 w, 992 w, 901 w, 890 w, 842 w, 833 w, 812 w, 786 m, 772 m, 742 m, 618 w.

The observed peak interval in the MALDI-TOF MS spectrum was 169, which is consistent with the mass of the repeating unit of **DG-PPE**.

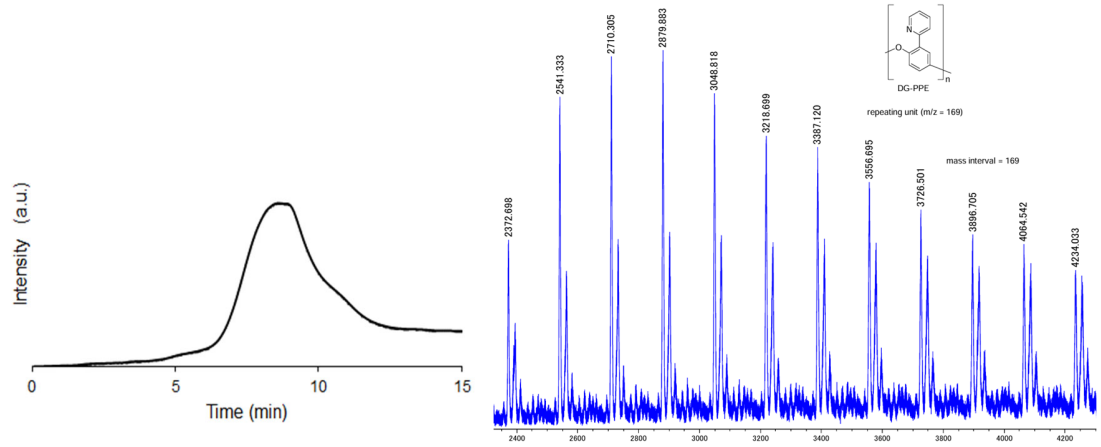


Fig. S2. SEC and MALDI-TOF spectrum of **DG-PPE**.

The relatively high dispersity of **DG-PPE** (M_w/M_n = 3.01) compared with the value reported in Yokozawa's work (M_w/M_n = 1.1)[13] can be attributed to the higher polymerization temperature used in our study. While Yokozawa conducted polymerization at 150 °C, our monomer **3** required 200 °C for efficient polymerization due to the pyridine ring being a weaker electron-withdrawing group compared to a cyano group used in the Yokozawa's work. The elevated temperature likely led to main-chain cleavage reactions as side reactions under our polymerization conditions, resulting in the higher dispersity observed. Specifically, aryloxide can attack the carbon ortho to the pyridine group (Fig. S3). We confirmed that this side reaction pathway occurred using a model substrate **1d** by reacting with *p*-cresol under the conditions used for polymerization (i.e., in NMP at 200 °C).

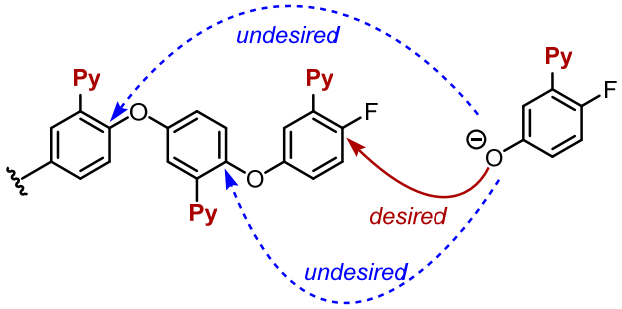
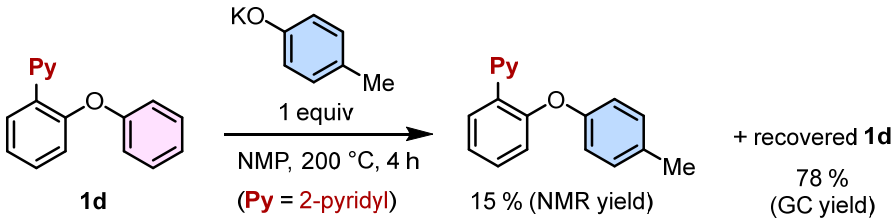


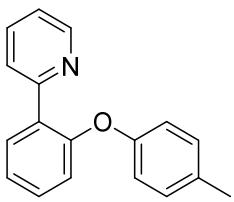
Fig. S3. One of the possible side reactions under our polymerization conditions.

Proof of side reaction pathway



In a glovebox filled with nitrogen, *p*-cresol (113 mg, 1.0 mmol), KH (43.2 mg, 1.1 mmol) and NMP (0.70 mL) were added to a 5 mL pressure-resistant vial. After stirring the reaction mixture at rt for 30 min, **1d** (244 mg, 1.0 mmol) and NMP (0.30 mL) were added to the vial and it was closed with a resealable septum. The mixture was stirred at 200 °C for 4 h using a microwave reactor. Water (20 mL) was added to the reaction mixture, and the aqueous layer was extracted with EtOAc. The combined extracts were dried over Na_2SO_4 and concentrated to give crude product. The crude product was analyzed by ^{1}H NMR with 1,1,2,2-tetrachloroethane as an internal standard, which revealed that 2-(2-(*p*-tolyloxy)phenyl)pyridine was formed in 15 % yield. Yield of recovered **1d** was determined by GC using dodecane as an internal standard. These results indicate that main chain cleavage can occur under the polymerization conditions, which leads to high dispersity of the resulting polymer.

2-(2-(*p*-Tolyloxy)phenyl)pyridine.



Colorless oil. R_f 0.58 (Hexane/EtOAc = 1/1).

^{1}H NMR ($CDCl_3$, 400 MHz) δ: 8.69–8.68 (m, 1H), 7.92 (dd, J = 7.8, 1.8 Hz, 1H), 7.88–7.86 (m, 1H), 7.65 (td, J = 7.7, 2.0 Hz, 1H), 7.33 (td, J = 7.8, 1.8 Hz, 1H), 7.26–7.17 (m, 2H), 7.09 (d, J = 8.2 Hz, 2H), 6.96 (dd, J = 8.2, 0.9 Hz, 1H), 6.89–6.85 (m, 2H), 2.30 (s, 3H).

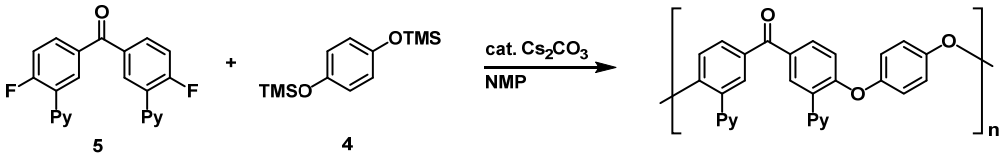
^{13}C NMR ($CDCl_3$, 101 MHz) δ: 155.2, 155.0, 154.6, 149.4, 135.9, 132.5, 131.6, 131.4, 130.2, 130.0, 124.9, 123.8, 122.0, 119.4, 118.4, 20.6.

IR (KBr): 3058 w, 3033 w, 2924 w, 1604 w, 1585 m, 1506 s, 1489 s, 1462 w, 1453 w, 1423 w, 1227 s, 913 w, 876 w, 831 w, 745 s.

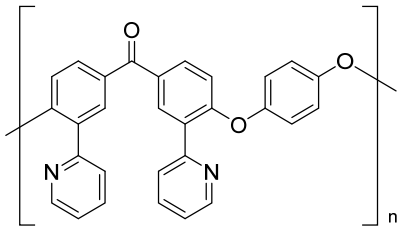
MS, *m/z* (relative intensity, %): 262 (13), 261 (M^+, 73), 260 (100), 244 (35), 127 (12), 109 (17).

HRMS (DART+, $[M+H]^+$): Calcd for $C_{18}H_{16}NO$: 262.1226. Found: 262.1230.

Synthesis of DG-PEEK1



The synthesis of **DG-PEEK1** was performed following a modified procedure from reported literature.[14] To a 100 mL two-necked flask, **5** (6.14 g, 16.5 mmol), 1,4-bis((trimethylsilyl)oxy)benzene (**4**, 4.21 g, 16.5 mmol), and NMP (33 mL) were added. The flask is equipped with a reflux condenser, and the reaction mixture is stirred under N_2 atmosphere at rt. After the reactants were completely dissolved, Cs_2CO_3 (184 mg, 3.4 mol%) were added, and the mixture was heated to 150 °C using an oil bath. After stirring the reaction mixture at 150 ˚C for 18 h, it was stirred at 180 °C for further 2.5 h. After cooling to rt, the polymer was precipitated by pouring the viscous reaction mixture slowly to a vigorously stirred MeOH. The polymer is purified by stirring in acetone. After drying in a vacuum oven, **DG-PEEK1** was obtained as a white powder (6.73 g, 92%).



M_w = 27200, M_w/M_n = 1.81.

^{1}H NMR ($CDCl_3$, 400 MHz) δ: 8.68 (d, J = 4.6 Hz, 2H), 8.38 (d, J = 2.4 Hz, 2H), 7.86 (d, J = 7.8 Hz, 2H), 7.81 (dd, J = 8.5, 2.1 Hz, 2H), 7.70 (td, J = 7.8, 1.8 Hz, 2H), 7.23 (td, J = 6.2, 1.1 Hz, 2H), 7.05 (s, 4H), 6.97 (d, J = 8.2 Hz, 2H).

^{13}C NMR ($CDCl_3$, 101 MHz) δ: 194.0, 158.3, 154.3, 152.1, 149.6, 136.1, 133.9, 133.0, 131.9, 130.8, 124.8, 122.4, 121.2, 117.5.

IR (ATR): 3055 w, 3006 w, 1655 w, 1599 m, 1586 m, 1498 m, 1485 s, 1463 w, 1440 w, 1323 w, 1248 m, 1218 s, 1186 s, 1136 w, 1058 w, 969 w, 748 m.

The observed peak interval in the MALDI-TOF MS spectrum was 442, which is consistent with the mass of the repeating unit of **DG-PEEK1**.

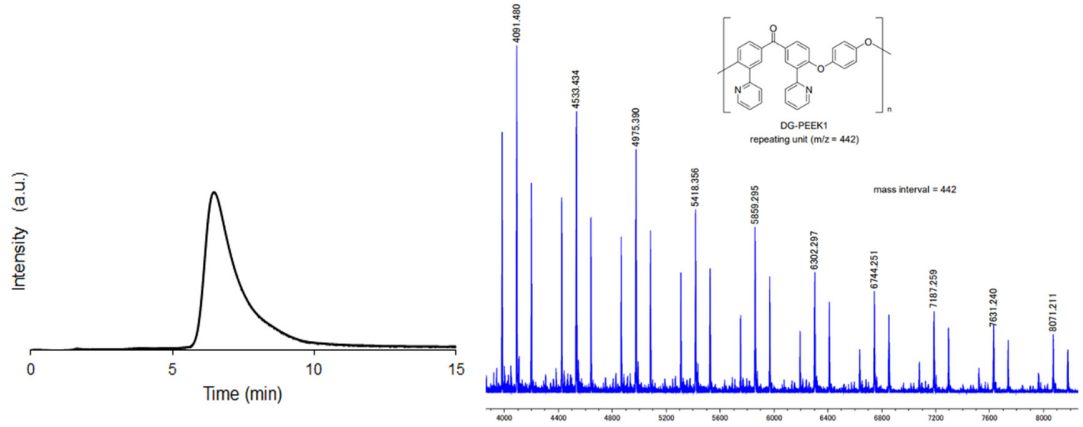
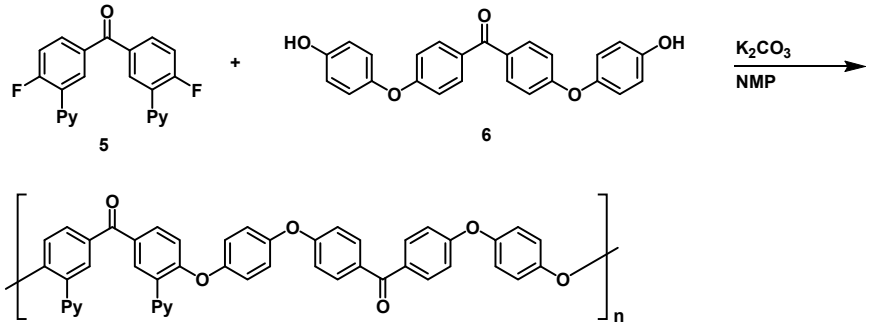


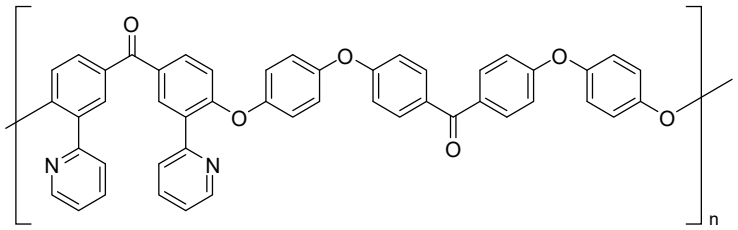
Fig. S4. SEC and MALDI-TOF spectrum of **DG-PEEK1**.

Synthesis of DG-PEEK2



To a 30 mL two-necked flask, **5** (391 mg, 0.98 mmol), **6**[15] (368 mg, 0.99 mmol), K_2CO_3 (302 mg, 2.2 mmol) and NMP (4 mL) were added. The flask is equipped with a reflux condenser, and the reaction mixture is stirred under N_2 atmosphere at rt. After the reactants are completely dissolved, the reaction mixture was stirred at 180 °C for 18 h. After cooling to rt, the polymer is precipitated by adding the viscous reaction

mixture slowly to vigorously stirred MeOH. The polymer was purified by stirring with acetone. After drying in a vacuum oven, **DG-PEEK2** was obtained as a white powder (619 mg, 86%).



M_w = 59000, M_w/M_n = 2.21.

^{1}H NMR ($CDCl_3$, 396 MHz) δ: 8.69 (d, J = 4.1 Hz, 2H), 8.40 (d, J = 1.8 Hz, 2H), 7.88 (d, J = 8.2 Hz, 2H), 7.84 (dd, J = 8.6, 2.3 Hz, 2H), 7.79 (d, J = 8.6 Hz, 4H), 7.71 (td, J = 7.7, 1.5 Hz, 2H), 7.24 (dd, J = 7.0, 5.2 Hz, 2H), 7.12–6.99 (m, 14H).

^{13}C NMR ($CDCl_3$, 101 MHz) δ: 194.1, 194.0, 161.4, 158.2, 154.4, 152.4, 151.7, 149.7, 136.0, 133.9, 133.1, 132.22, 132.18, 131.9, 131.0, 124.8, 122.4, 121.7, 121.1, 117.6, 116.8.

IR (ATR): 3065 w, 3004 w, 1653 w, 1598 m, 1487 s, 1464 w, 1306 w, 1279 w, 1247 m, 1221 s, 1188 s, 1161 m, 929 w, 850 w, 751 m.

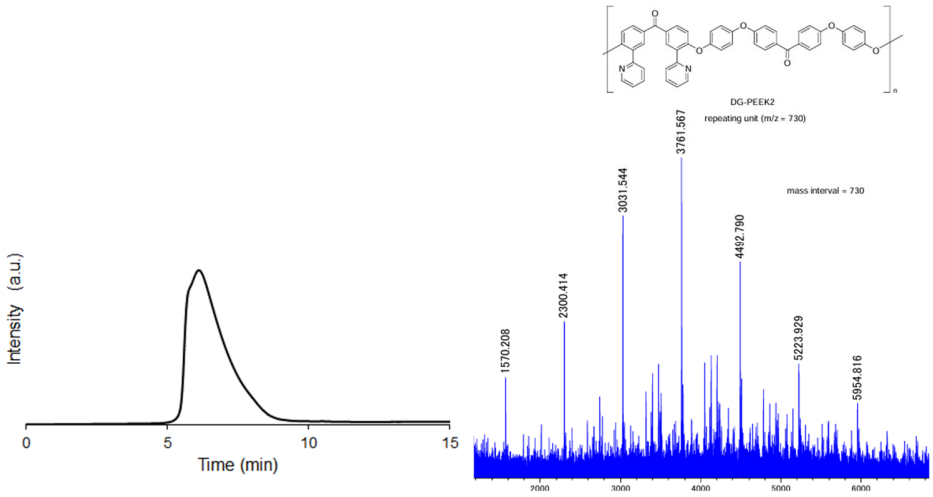


Fig. S5. SEC chart of **DG-PEEK2**.

V-2. Thermal properties of DG-polymers

Differential Scanning Calorimetry (DSC) Thermograms

DG-PPE: Thermal transitions were assessed with differential scanning calorimetry using a Hitachi High-Tech NEXTA DSC2000 and 2–3 mg of polymer material. Samples were heated to 350 °C and then cooled to 30 °C at 10 °C/min. Samples were again heated to 350 °C at 10 °C/min. Thermal data was assessed from the second heating cycle. T_g value of **DG-PPE** was determined to be 143 °C (Fig. S6).

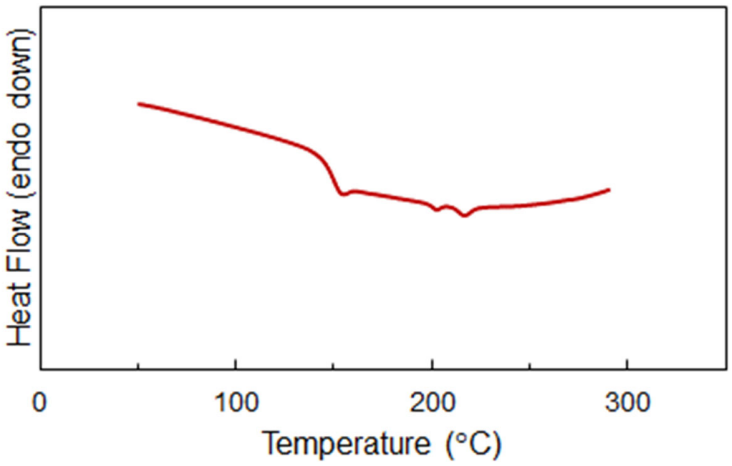


Fig. S6. DSC second heating thermogram of **DG-PPE**.

DG-PEEK1, DG-PEEK2, PEEK: Thermal transitions were assessed with differential scanning calorimetry using a Hitachi High-Tech NEXTA DSC2000 and 1–10 mg of polymer material. Samples were heated to 400 °C and then cooled to 30 °C at 10 °C/min. Samples were again heated to 400 °C at 10 °C/min. Thermal data was assessed from the second heating cycle. PEEK without DG was purchased from Sigma–Aldrich Japan (pellet forms, Cat. No. 456640.). T_g values for **DG-PEEK1** and **DG-PEEK2** were determined to be 182 and 172 °C, respectively, with no T_m being observed (Fig. S7 and S8). The parent PEEK without DGs has T_g of 143 °C and T_m of 344 °C (Fig. S9).

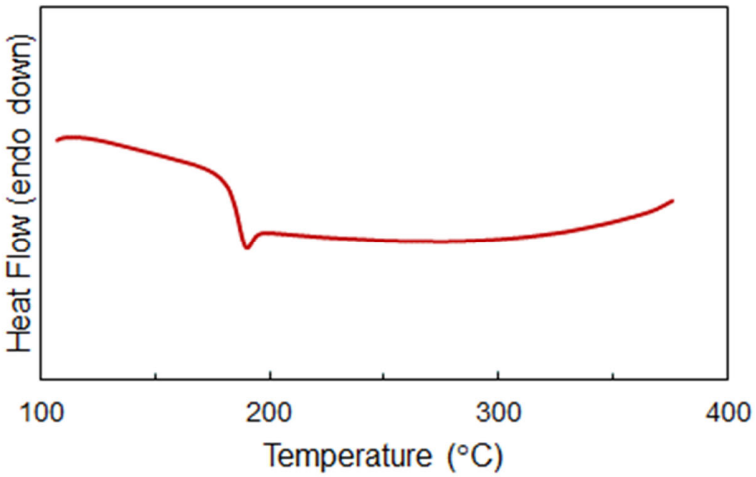


Fig. S7. DSC second heating thermogram of **DG-PEEK1**.

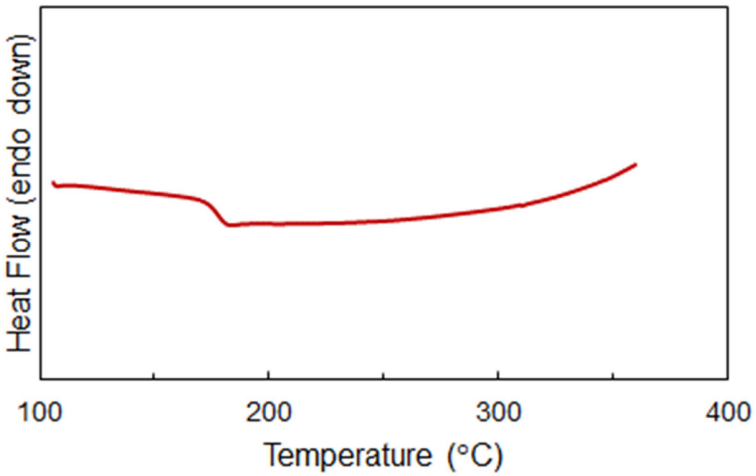


Fig. S8. DSC second heating thermogram of **DG-PEEK2**.

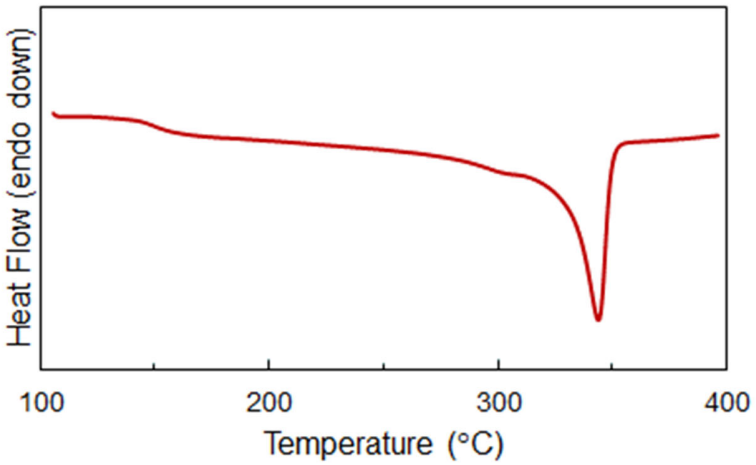


Fig. S9. DSC second heating thermogram of PEEK.

Thermogravimetric Analysis (TGA)

DG-PPE: Thermogravimetric analysis was performed using a Hitachi High-Tech NEXTA STA200RV and 3–6 mg of polymer material. Samples were heated at a rate of 10 °C/min from 40 °C to 600 °C under N_2 or air flow. Decomposition temperatures are reported at 5% mass loss (T_{d5}). T_{d5} of **DG-PPE** was determined to be 399 °C (N_2) and 419 °C (air) (Fig. S10).

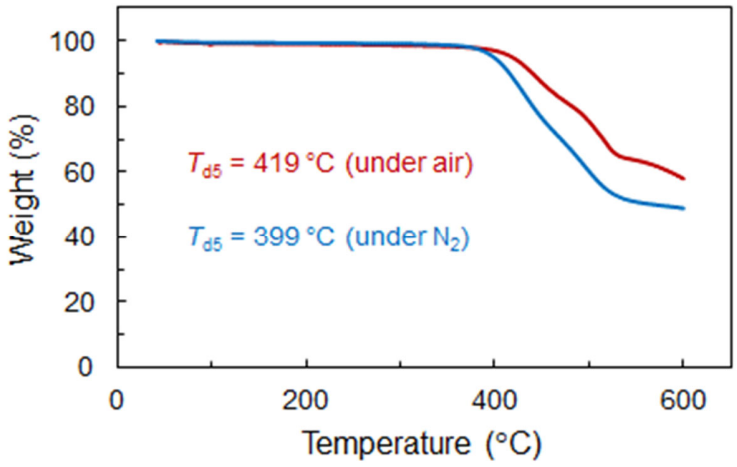


Fig. S10. TGA of **DG-PPE**.

PEEK, **DG-PEEK1** and **DG-PEEK2**: Thermogravimetric analysis was performed using a Hitachi High-Tech NEXTA STA200RV and 1–10 mg of polymer material. Samples were heated at a rate of 10 °C/min from 30 °C to 600 °C under N_2 or air flow. Decomposition temperatures are reported at 5% mass loss (T_{d5}). T_{d5} of **DG-PEEK1** was determined to be 465 (N_2) and 473 °C (air) (Fig. S11). T_{d5} of **DG-PEEK2** was determined to be 437 (N_2) and 451 °C (air) (S12). The parent PEEK without DGs had T_{d5} of 576 °C (Fig. S13).

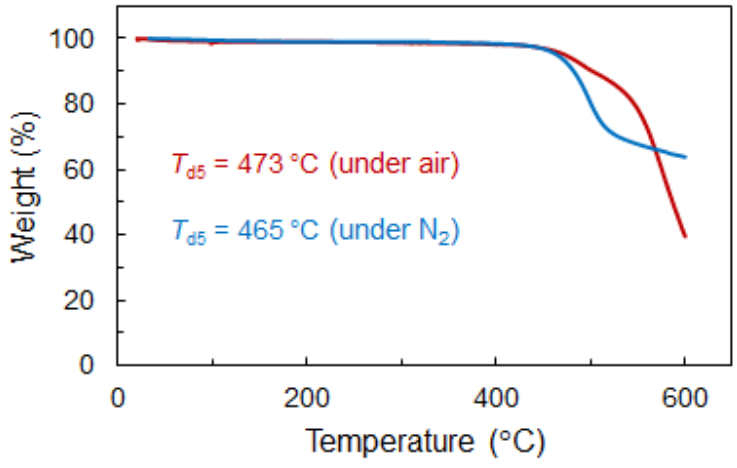


Fig. S11. TGA of **DG-PEEK1**.

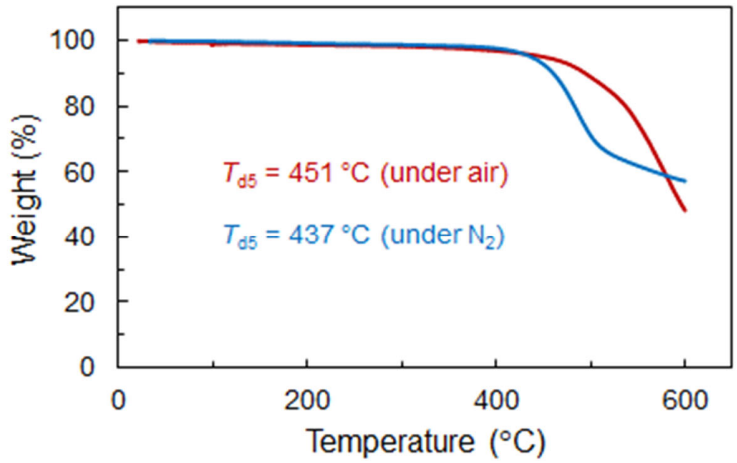


Fig. S12. TGA of **DG-PEEK2**.

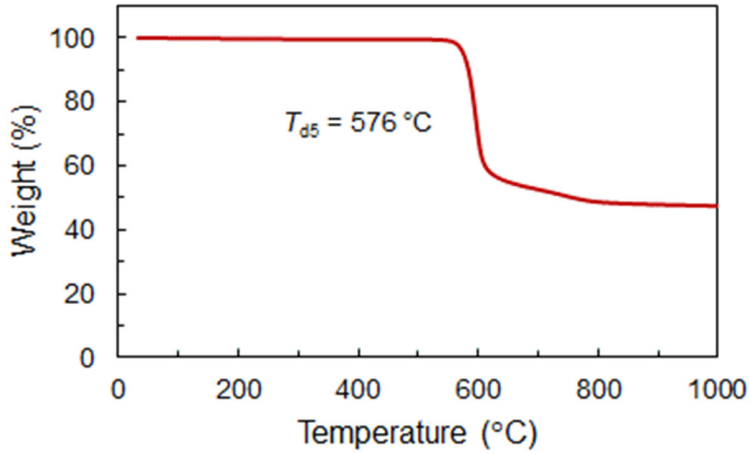
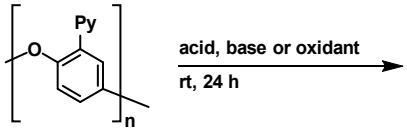


Fig. S13. TGA of PEEK.

VI. Chemical Stability of DG-PPE and DG-PEEK1

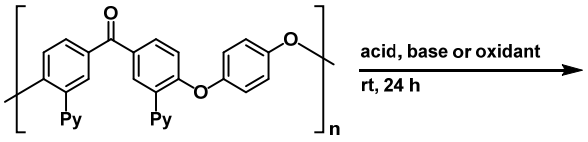
VI-1. Stability of DG-PPE in different solutions



DG-PPE (5.0 mg) was introduced into a 10 mL vial with a Teflon-sealed screwcap, along with a magnetic stirring bar. Subsequently, a solution (1.0 mL) containing HCl (12 M), NaOH (10 M), H_2O_2 (30%), AcOH

or water was added to the vial, and the mixture was stirred for 24 h at rt (or heated to 100°C in the case of water). Following neutralization, the mixture was extracted with $CHCl_3$ (2 mL × 3). The combined extracts were dried, and the solid obtained was subjected to FT-IR spectroscopic analysis using an ATR detector. A portion of the solid was dissolved in HPLC-grade $CHCl_3$, filtered through a syringe filter, and subsequently analyzed via SEC analysis.

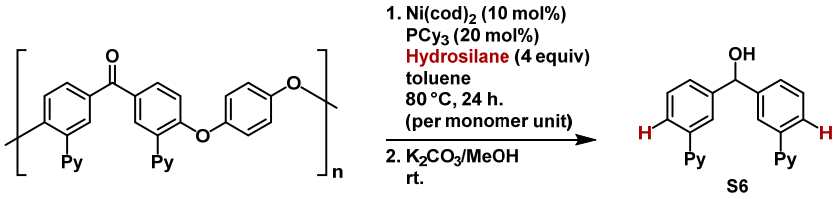
VI-2. Stability of DG-PEEK1 in different solutions



DG-PEEK1 (5.0 mg) was introduced into a 10 mL vial with a Teflon-sealed screwcap, along with a magnetic stirring bar. Subsequently, a solution (1.0 mL) containing HCl (12 M), NaOH (10 M), H_2O_2 (30%), AcOH or water was added to the vial, and the mixture was stirred for 24 h at rt (or heated to 100°C in the case of water). Following neutralization, the mixture was extracted with $CHCl_3$ (2 mL × 3). The combined extracts were dried, and the solid obtained was subjected to FT-IR spectroscopic analysis using an ATR detector. A portion of the solid was dissolved in HPLC-grade $CHCl_3$, filtered through a syringe filter, and subsequently analyzed via SEC analysis.

VII. Optimization of Depolymerization Conditions

VII-1. Optimization of C–O bond cleavage in DG-PEEK1



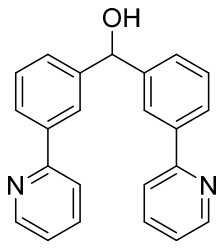
S6

Entry	Hydrosilane	Yield [%]
1	$HSiEt_3$	0
2[a]	$(HSiMe_2)O$	32
3	$HSiMe(OMe)_2$	36
4	$HSiMe(OEt)_2$	51 (89)[b]
5	$HSiMe(OTMS)_2$	0
6	$HSi(OMe)_3$	25
7	$HSi(OEt)_3$	26

Fig. S14. Screening of hydrosilane in C–O bond cleavage reaction with **DG-PEEK1**.
[a]3 Equiv of hydrosilane was used. [b]With 20 mol% catalyst.

In a glovebox filled with nitrogen, **DG-PEEK1** (0.20 mmol), $Ni(cod)_2$ (0.020 mmol), PCy_3 (0.04 mmol), hydrosilane (0.80 mmol) and toluene (1.0 mL) were added to a 5 mL vial with a Teflon-sealed screwcap. The mixture was stirred at 80 °C for 24 h. The resulting mixture was then evaporated to dryness, and the residue was hydrolyzed with saturated solution of K_2CO_3 in MeOH (1.0 mL) at rt for 4 h. After removing MeOH in vacuo, the crude mixture was purified by flash column chromatography (eluent: hexane/EtOAc = 80/20 to 30/70) to give **S6** as a white solid. Among the hydrosilanes examined, $HSiMe(OEt)_2$ resulted in the formation of a degraded product in highest yield.

Bis(3-(pyridin-2-yl)phenyl)methanol (S6).



White solid. R_f 0.12 (Hexane/EtOAc = 1/1). M.p. 134.1–136.3 ºC.

^{1}H NMR ($CDCl_3$, 400 MHz) δ: 8.66 (d, J = 5.0 Hz, 2H), 8.06 (s, 2H), 7.87 (d, J = 7.3 Hz, 2H), 7.75–7.68 (m, 4H), 7.46–7.40 (m, 4H), 7.23–7.20 (m, 2H), 6.01 (s, 1H), 2.90 (br, 1H).

^{13}C NMR ($CDCl_3$, 101 MHz) δ: 157.3, 149.5, 144.5, 139.4, 136.8, 128.9, 127.3, 126.1, 125.3, 122.2, 120.9, 76.0.

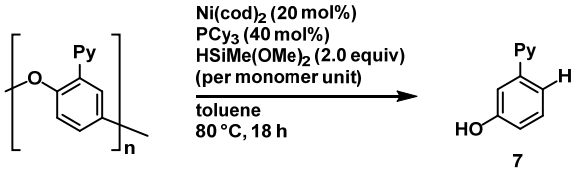
IR (KBr): 3331 br, 3061 m, 2960 m, 2927 m, 1652 w, 1586 s, 1567 s, 1464 s, 1435 s, 1417 m, 1268 m, 1173 m, 1153 m, 1096 m, 1054m, 998 m, 911 w, 775 s, 753 s, 703 m, 640 w, 617 w.

MS, *m/z* (relative intensity, %): 338 (M^+, 26), 337 (24), 260 (18), 182 (12), 157 (13), 156 (100), 155 (17), 154 (29), 127 (14), 78 (23).

HRMS (DART+, $[M+H]^+$): Calcd for $C_{23}H_{19}N_2O$: 339.1492. Found: 339.1493.

VIII. Typical Procedures for Depolymerization

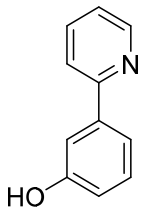
VIII-1. Depolymerization of DG-PPE



In a glovebox filled with nitrogen, **DG-PPE** (21.1 mg, 0.13 mmol), $Ni(cod)_2$ (6.9 mg, 0.025 mmol), PCy_3 (16.4 mg, 0.058 mmol), $HSiMe(OMe)_2$ (27.7 mg, 0.26 mmol) and toluene (0.5 mL) were added to a 5 mL vial with a Teflon-sealed screwcap. The mixture was stirred at 80 °C for 18 h. The resulting mixture was then evaporated to dryness, and the residue was purified by flash column chromatography (eluent: hexane/EtOAc = 90/10 to 50/50) to give **7** as a white solid (13.6 mg, 64%).

Note: When $HSiMe(OEt)_2$ was used, instead of $HSiMe(OMe)_2$, **7** was obtained in lower yield of 30%, along with its silylated derivative (5%).

3-(Pyridin-2-yl)phenol (7).



White solid. R_f 0.50 (Hexane/EtOAc = 1/1). M.p. 101.3–102.7 ºC.

^{1}H NMR ($CDCl_3$, 400 MHz) δ: 8.64–8.63 (m, 1H), 7.74 (td, J = 7.8, 1.8 Hz, 1H), 7.59 (d, J = 8.2 Hz, 1H), 7.50 (t, J = 1.8 Hz, 1H), 7.36 (d, J = 7.8 Hz, 1H), 7.27–7.22 (m, 2H), 6.85 (dd, J = 8.0, 1.6 Hz, 1H).

The chemical shifts in ^{1}H NMR spectra were recorded relative to tetramethylsilane (δ 0.00).

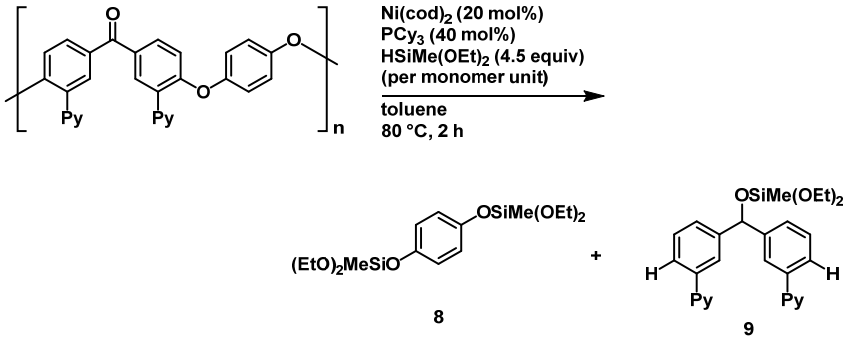
^{13}C NMR ($CDCl_3$, 101 MHz) δ: 157.5, 157.1, 149.0, 140.2, 137.4, 130.0, 122.4, 121.6, 118.8, 116.7, 114.6.

IR (KBr): 2794 br, 2667 s, 2607 s, 1593 s, 1567 s, 1468 s, 1430 s, 1352 s, 1313 s, 1281 s, 1243 m, 1216 s, 1156 s, 1082 m, 1006 s, 999 s, 897 s, 872 m, 807 m, 771 s, 746 m, 729 m, 700 s, 631 m, 618 m, 538 m.

MS, *m/z* (relative intensity, %): 172 (16), 171 (M^+, 100), 170 (24), 154 (13), 143 (40), 142 (19), 141 (14), 117 (49), 115 (23), 89 (12), 63 (12), 51 (13).

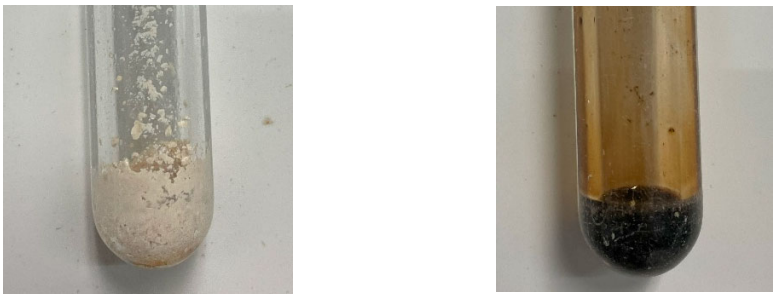
HRMS (DART+, $[M+H]^+$): Calcd for $C_{11}H_{10}NO$: 172.0757. Found: 172.0758

VIII-2. Depolymerization of DG-PEEK1



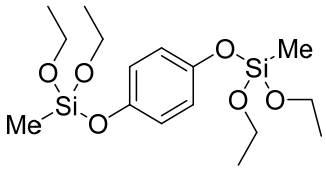
In a glovebox filled with nitrogen, **DG-PEEK1** (87.1 mg, 0.20 mmol), $Ni(cod)_2$ (11.7 mg, 0.043 mmol), PCy_3 (22.5 mg, 0.080 mmol), hydrosilane (115.0 mg, 0.86 mmol) and toluene (0.40 mL) were added to a 5 mL vial with a Teflon-sealed screwcap. The mixture was stirred at 80 °C for 2 h. The resulting mixture was then evaporated to dryness, and the residue was purified by flash column chromatography (eluent: hexane/EtOAc = 100/0 to 60/40) to give **8** as a colorless oil (57.7 mg, 78%) and **9** as a yellow oil (80.4 mg, 87%). Column chromatography was performed with SiO_2 (Silica Gel 60N (spherical, neutral) (40-50 μm, KANTO CHEMIXAL CO., INC.).

(A) (B)



Pictures of the reaction mixture before heating (A) and after heating at 80 °C for 24 h (B).

1,4-Bis((diethoxy(methyl)silyl)oxy)benzene (8).



Colorless oli. R_f 0.73 (Hexane/EtOAc = 1/1).

^{1}H NMR ($CDCl_3$, 400 MHz) δ: 6.84 (s, 4H), 3.87 (q, J = 7.0 Hz, 8H), 1.23 (t, J = 7.1 Hz, 12H), 0.23 (s, 6H).

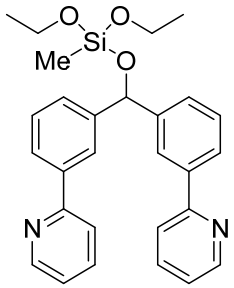
^{13}C NMR ($CDCl_3$, 101 MHz) δ: 148.5, 120.0, 58.7, 18.1, −6.8.

IR (KBr): 2976 m, 2927 w, 2888 w, 1506 s, 1267 m, 1241 m, 1167 m, 1107 s, 1082 s, 954 s, 839 m, 784 m, 775m.

MS, *m/z* (relative intensity, %): 376 (11), 375 (28), 374 (M^+, 100), 226 (19), 133 (37), 105 (16), 89 (16), 77 (26).

HRMS (EI+, M^+) Calcd for $C_{16}H_{30}O_6Si_2$: 374.1581. Found: 374.1588.

2,2'-((((Diethoxy(methyl)silyl)oxy)methylene)bis(3,1-phenylene))dipyridine (9).



Yellow oil. R_f 0.64 (Hexane/EtOAc = 1/1).

^{1}H NMR ($CDCl_3$, 400 MHz) δ: 8.68–8.66 (m, 2H), 8.04 (s, 2H), 7.90–7.88 (m, 2H), 7.74–7.68 (m, 4H), 7.51 (d, J = 7.8 Hz, 2H), 7.43 (t, J = 7.8 Hz, 2H), 7.22–7.18 (m, 2H), 6.20 (s, 1H), 3.73 (q, J = 7.0 Hz, 4H), 1.14 (t, J = 7.1 Hz, 6H), 0.10 (s, 3H).

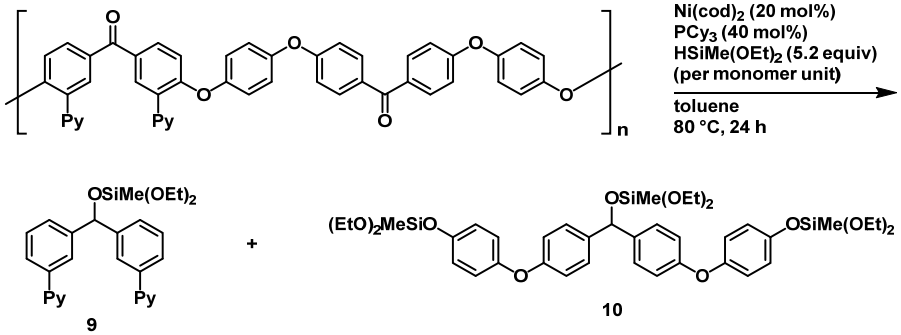
^{13}C NMR ($CDCl_3$, 101 MHz) δ: 157.4, 149.6, 144.6, 139.4, 136.6, 128.8, 127.2, 125.9, 125.1, 122.0, 120.6, 76.1, 58.4, 18.1, −6.4.

IR (KBr): 2973 m, 2924 w, 2886 w, 1584 m, 1566 w, 1462 m, 1436 m, 1417 w, 1265 w, 1167 w, 1099 s, 1074 s, 957 m, 876 w, 824 w, 773 m, 746 m.

MS, *m/z* (relative intensity, %): 472 (12), 471 (39), 470 (M^+, 100), 322 (15), 321 (36), 320 (41), 319 (12), 316 (10), 302 (17), 288 (18), 244 (13), 242 (16), 226 (23), 190 (14), 133 (27), 105 (21), 89 (24), 77 (39).

HRMS (ESI+, $[M+Na]^+$): Calcd for $C_{28}H_{30}N_2O_3NaSi$: 493.1918. Found: 493.1925.

VIII-3. Depolymerization of DG-PEEK2

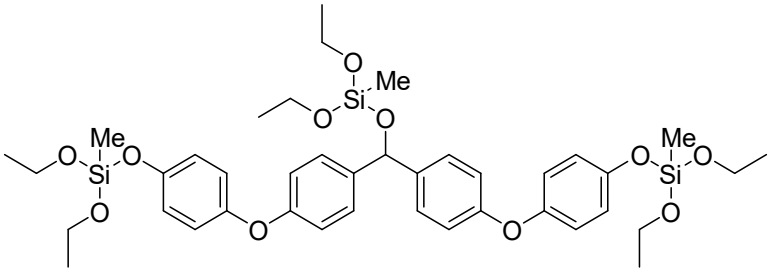


In a glovebox filled with nitrogen, **DG-PEEK2** (77.9 mg, 0.11 mmol), $Ni(cod)_2$ (6.2 mg, 0.023 mmol), PCy_3 (12.0 mg, 0.043 mmol), $HSiMe(OEt)_2$ (69.4 mg, 0.52 mmol) and toluene (0.4 mL) were added to a 5 mL vial with a Teflon-sealed screwcap. The mixture was stirred at 80 °C for 2 h. The resulting mixture was then evaporated to dryness, and the residue was purified by flash column chromatography (eluent: hexane/EtOAc = 100/0 to 60/40) to give **9** as a yellow oil (39.9 mg, 80%) and **10** as a colorless oil (57.6 mg, 68%). Column chromatography was performed with SiO_2 (Silica Gel 60N (spherical, neutral) (40-50 μm, KANTO CHEMIXAL CO., INC.).

(A) (B) (C)

Pictures of the reaction mixture before heating (A) and after heating at 80 °C for 2 h (B) and 24 h (C).

(((((((Diethoxy(methyl)silyl)oxy)methylene)bis(4,1-phenylene))bis(oxy))bis(4,1-phenylene))bis(oxy))bis(diethoxy(methyl)silane) (10).



Colorless oil. R_f 0.75 (Hexane/EtOAc = 1/1).

^{1}H NMR ($CDCl_3$, 400 MHz) δ: 7.29 (d, J = 8.6 Hz, 4H), 6.94-6.87 (m, 12H), 5.94 (s, 1H), 3.89 (q, J = 6.9 Hz, 8H), 3.70 (q, J = 7.2 Hz, 4H), 1.24 (t, J = 7.0 Hz, 12H), 1.14 (t, J = 7.0 Hz, 6H), 0.25 (s, 6H), 0.05 (s, 3H).

^{13}C NMR ($CDCl_3$, 101 MHz) δ: 157.3, 151.0, 150.0, 138.6, 127.7, 120.5, 120.4, 117.5, 75.1, 58.8, 58.4, 18.1, −6.5, −6.8.

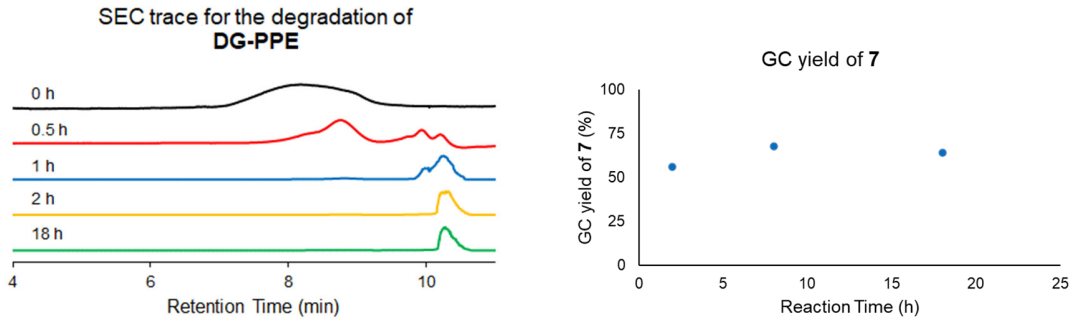
IR (KBr): 2975 m, 2927 w, 2889 w, 1497 s, 1267 m, 1227 s, 1197 w, 1166 m, 1104 s, 1079 s, 954 m, 875 w, 827 m, 807 m, 786 m.

HRMS (FAB+, M^+): Calcd for $C_{40}H_{56}O_{11}Si_3$: 796.3130. Found: 796.3141.

IX. Time-course Study

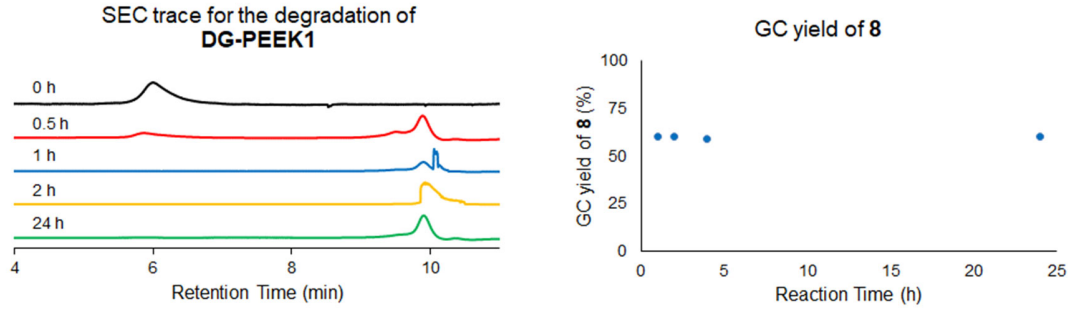
IX -1. Time-course of the degradation of DG-PPE

Reactions were set up according to the typical Procedure outlined above on a 0.13 mmol scale. Time points were taken as separate reactions. To track degradation of DG-PPE via molecular weight, a portion of resulting mixture was subjected to SEC analysis. The yields of **7** was determined by GC using pentadecane as an internal standard. SEC analysis showed that the polymeric materials disappeared within 2 h, but the yield of monomer **7** increased slightly with extended reaction time (50% at 2 hours; 64% at 18 hours).



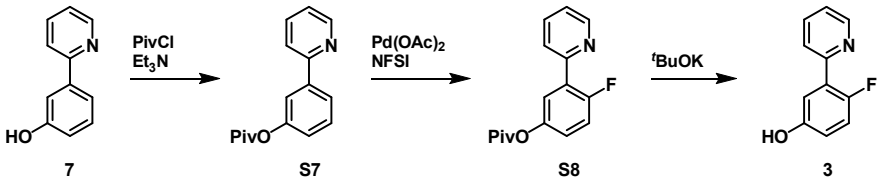
IX -2. Time-course of the degradation of DG-PEEK1

Reactions were set up according to the typical Procedure outlined above on a 0.10 mmol scale. Time points were taken as separate reactions. To track degradation of DG-PEEK1 via molecular weight, a portion of resulting mixture was subjected to SEC analysis. The yields of **7** was determined by GC using pentadecane as an internal standard. SEC traces and GC measurements showed that degradation of **DG-PEEK1** were completed within 2 h.

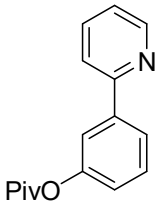


X. Repolymerization of Monomeric Products into Original DG-Polymers

X -1. Conversion of 7 to 3 for Repolymerization to DG-PPE



3-(Pyridin-2-yl)phenyl pivalate (S7). To a 50 mL two-necked flask, **7** (700.2 mg, 4.1 mmol), pivaloyl chloride (0.54 mL, 4.4 mmol) , Et_3N (1.2 mL, 8.7 mmol) and THF (16 mL) were added at 0 °C. The reaction mixture was warmed to rt and then stirred for 13 h. The resulting mixture was filtered, and THF was evaporated in vacuo. The crude product was purified by flash column chromatography (eluent: hexane/EtOAc = 80/20 to 70/30) to give **S7** as a pale yellow oil (1.03 g, 99%).



Pale yellow oil. R_f 0.54 (Hexane/EtOAc = 1/1).

^{1}H NMR ($CDCl_3$, 400 MHz) δ: 8.70–8.68 (m, 1H), 7.84–7.81 (m, 1H), 7.78–7.72 (m, 3H), 7.48 (t, J = 7.8 Hz, 1H), 7.26–7.22 (m, 1H), 7.13–7.11 (m, 1H), 1.38 (s, 9H).

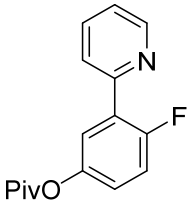
^{13}C NMR ($CDCl_3$, 101 MHz) δ: 177.0, 156.3, 151.6, 149.6, 140.9, 136.8, 129.6, 123.9, 122.5, 122.1, 120.7, 120.1, 39.1, 27.2.

IR (KBr): 3066 w, 2975 w, 2872 w, 1749 s, 1584 m, 1567 w, 1472 m, 1460 m, 1277 m, 1181 m, 1167 m, 1117 s, 767 m.

MS, *m/z* (relative intensity, %): 255 (M^+, 20), 172 (14), 171 (100), 143 (27), 117 (12), 85 (24), 57 (96), 41 (14).

HRMS (ESI+, $[M+H]^+$): Calcd for $C_{16}H_{18}NO_2$: 256.1332. Found: 256.1339.

4-Fluoro-3-(pyridin-2-yl)phenyl pivalate (S8). This compound was synthesized using a literature procedure,[16] with minor modifications. In a glovebox filled with nitrogen, 3-(pyridin-2-yl)phenyl pivalate (26.1 mg, 0.10 mmol), $Pd(OAc)_2$ (2.3 mg, 0.010 mmol), NFSI (79.3 mg, 0.25 mmol), acetonitrile (0.1 mL) and benzotrifluoride (4.3 mL) were added to a 5 mL pressure-resistant vial and it was closed with a resealable septum. The mixture was stirred at 170 °C for 2 h using a microwave reactor. After cooling to rt, the resulting mixture was evaporated to dryness, and the residue was purified by flash column chromatography (eluent: hexane/EtOAc = 95/5 to 50/50) to give **S8** as a colorless oil (12.7 mg, 45%).



Colorless oil. R_f 0.54 (Hexane/EtOAc = 1/1).

^{1}H NMR ($CDCl_3$, 400 MHz) δ: 8.72–8.70 (m, 1H), 7.84–7.81 (m, 1H), 7.77–7.71 (m, 2H), 7.27–7.24 (m, 1H), 7.16 (dd, J = 10.3, 8.9 Hz, 1H), 7.09–7.05 (m, 1H), 1.35 (s, 9H).

^{13}C NMR ($CDCl_3$, 101 MHz) δ: 177.0, 157.7 (d, J = 248.2 Hz), 152.3 (d, J = 2.9 Hz), 149.6, 147.2 (d, J = 2.9 Hz), 136.5, 128.0 (d, J = 13.4 Hz), 124.5 (d, J = 10.5 Hz), 123.5 (d, J = 3.8 Hz), 123.4 (d, J = 9.6 Hz), 122.7, 116.9 (d, J = 25.9 Hz), 39.0, 27.1.

^{19}F NMR ($CDCl_3$, 376 MHz) δ: −122.5.

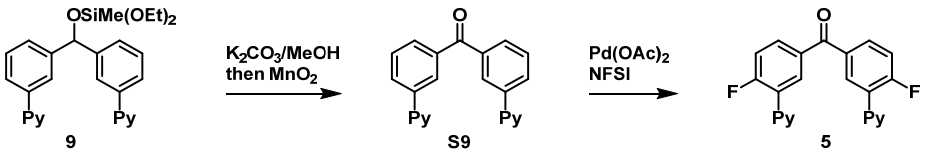
IR (KBr): 3073 w, 2977 s, 2939 m, 2875 m, 1749 s, 1586 s, 1571 m, 1496 s, 1462 s, 1408 m, 1272 m, 1239 m, 1171 m, 1120 s, 1057 m, 1027 m, 916 m, 831 m, 791 m, 770 s, 615 m.

MS, *m/z* (relative intensity, %): 273 (M^+, 20), 190 (14), 189 (100), 161 (28), 85 (34), 57 (95), 41 (14).

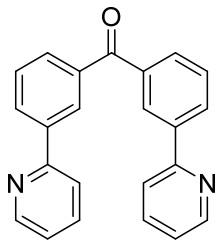
HRMS (DART+, $[M+H]^+$): Calcd for $C_{16}H_{17}FNO_2$: 274.1238. Found: 274.1244.

4-Fluoro-3-(pyridin-2-yl)phenol (3). This compound was synthesized using a literature procedure[17], with minor modifications. To a 10 mL round-bottom flask, 4-fluoro-3-(pyridin-2-yl)phenyl pivalate (52.3 mg, 0.19 mmol), ^{t}BuONa (58.5 mg, 0.61 mmol) and MeOH (1 mL) were added at 0 °C. After stirring at rt for 4 h, the reaction was quenched by adding saturated aqueous solution of NH_4Cl and the resulting mixture was extracted with EtOAc. The combined extracts were dried over Na_2SO_4 and concentrated. The crude product was purified by flash column chromatography (eluent: hexane/EtOAc = 95/5 to 50/50) to give 4-fluoro-3-(pyridin-2-yl)phenol (**3**) as a white solid (29.3 mg, 81%).

X -2. Conversion of 9 to 5 for Repolymerization to DG-PEEK1



Bis(3-(pyridin-2-yl)phenyl)methanone (S9). CAS[851540-82-4] Compound **9** (97.8 mg, 0.21 mmol) was hydrolyzed with saturated solution of K_2CO_3 in MeOH (1.0 mL) at rt for 30 min. After removing MeOH in vacuo, toluene (2.0 mL) and MnO_2 (263.3 mg, 3.0 mmol) were added. The mixture was stirred at rt for 17 h. The resulting mixture was filtered, and toluene was evaporated in vacuo to give **S9** as a white solid (70.2 mg, 100%).



White solid. R_f 0.28 (Hexane/EtOAc = 1/1). M.p. 140.6–141.0 °C.

^{1}H NMR ($CDCl_3$, 400 MHz) δ: 8.70 (dd, J = 4.6, 0.9 Hz, 2H), 8.44 (t, J = 1.8 Hz, 2H), 8.29 (dd, J = 7.8, 1.4 Hz, 2H), 7.87 (d, J = 7.8 Hz, 2H), 7.80–7.75 (m, 4H), 7.62 (t, J = 7.8 Hz, 2H), 7.28–7.25 (m, 2H).

^{13}C NMR ($CDCl_3$, 101 MHz) δ: 196.5, 156.4, 149.8, 139.7, 138.1, 137.0, 131.0, 130.5, 128.8, 128.4, 122.6, 120.7.

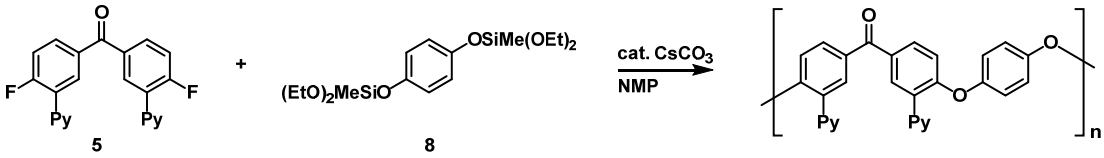
IR (KBr): 3064 w, 3049 w, 1972 w, 1921 w, 1650 s, 1596 s, 1581 s, 1563 s, 1473 m, 1433 s, 1313 s, 1298 m, 1246 s, 1168 m, 1144 m, 1086 m, 992 m, 976 m, 914 m, 816 m, 780 s, 756 s, 748 s, 736 s, 692 m, 649 s, 618 m.

MS, *m/z* (relative intensity, %): 336 (M^+, 67), 335 (53), 307 (16), 258 (100), 207 (63), 182 (13), 154 (51), 127 (34).

HRMS (DART+, $[M+H]^+$): Calcd for $C_{23}H_{17}N_2O$: 337.1335. Found: 337.1335.

Bis(4-fluoro-3-(pyridin-2-yl)phenyl)methanone (5). This compound was synthesized using a literature procedure,[16] with minor modifications. In a glovebox filled with nitrogen, bis(3-(pyridin-2-yl)phenyl)methanone (67.9 mg, 0.20 mmol), $Pd(OAc)_2$ (6.1 mg, 0.027 mmol), NFSI (334.5 mg, 1.1 mmol), acetonitrile (0.36 mL) and benzotrifluoride (18.0 mL) were added to a 20 mL pressure-resistant vial and it was closed with a resealable septum. The mixture was stirred at 160 °C for 2 h using a microwave reactor. After cooling to rt, saturated solution of K_2CO_3 in MeOH (1.0 mL) was added. The resulting mixture was then evaporated to dryness, and the residue was purified by flash column chromatography (eluent: hexane/EtOAc = 100/0 to 52/48) to give **5** as a white solid (35.1 mg, 47%).

Repolymerization to DG-PEEK1.



DG-PEEK1 was synthesized using monomers obtained from depolymerization experiments. To a 20 mL two-necked flask, compound **5** (743.6 mg, 2.0 mmol), compound **8** (750.2 mg, 2.0 mmol), and NMP (4 mL) were added. The flask is equipped with a reflux condenser, and the reaction mixture is stirred under N_2 atmosphere at rt. After the reactants were completely dissolved, Cs_2CO_3 (23.4 mg, 3.6 mol%) was added, and the mixture was heated to 150˚C using an oil bath. After stirring the reaction mixture at 150˚C for 18 h, it was stirred at 180˚C for further 3 h. After cooling to rt, the polymer was precipitated by adding the viscous reaction mixture slowly to vigorously stirred MeOH. The polymer was purified by stirring with acetone. After drying in a vacuum oven, **DG-PEEK1** was obtained as a white powder (806.1 mg, 92%). The spectroscopic data of the repolymerized **DG-PEEK1** was in agreement with that of the original **DG-PEEK1**. The values of M_w, T_g and T_{d5} were comparable to those obtained for the original **DG-PEEK1** (T_{d5} = 474 °C, T_g = 179 °C, M_w = 35400, M_w/M_n = 2.37).

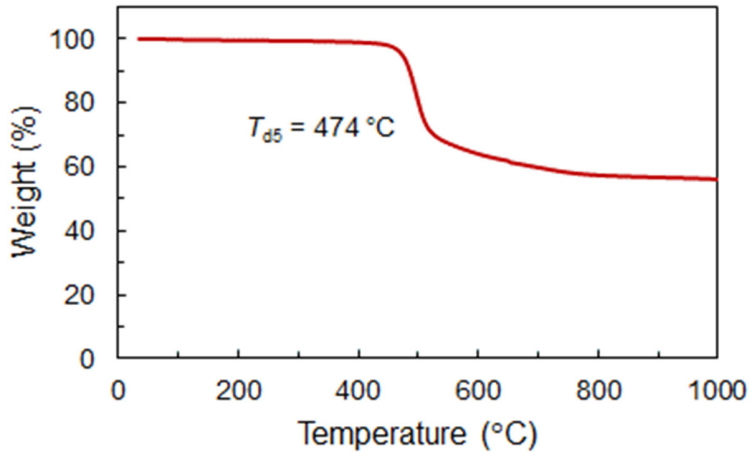


Fig. S15. TGA of **DG-PEEK1** obtained by repolymerization.

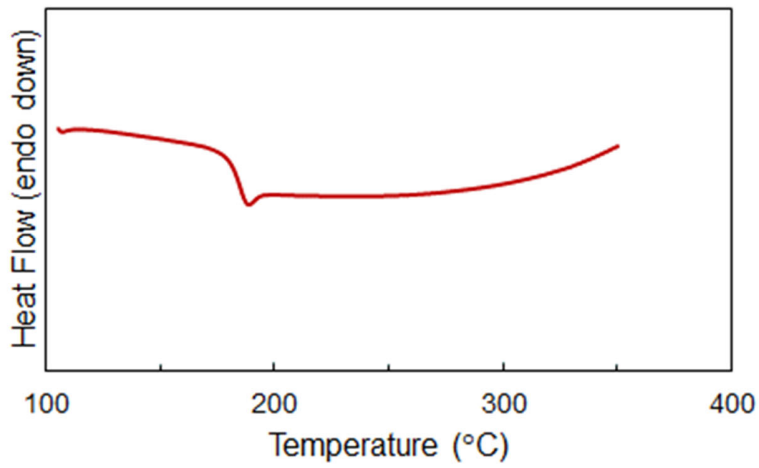


Fig. S16. DSC second heating thermogram of **DG-PEEK1** obtained by repolymerization.

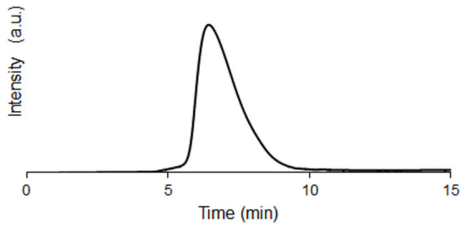
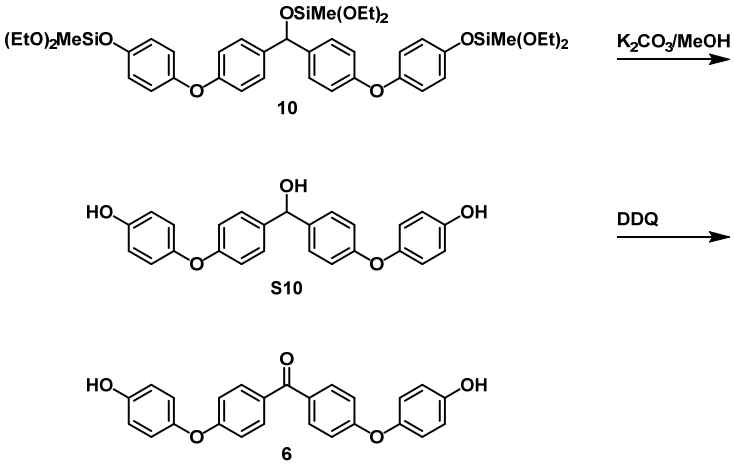
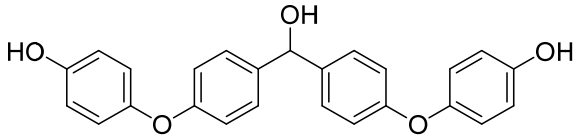


Fig. S17. SEC chart of **DG-PEEK1** obtained by repolymerization.

X-3. Conversion of 10 to 6 for repolymerization to DG-PEEK2



4,4'-(((Hydroxymethylene)bis(4,1-phenylene))bis(oxy))diphenol (S10). Compound **10** (75.1 mg, 0.094 mmol) was hydrolyzed with a saturated solution K_2CO_3 in MeOH (3.0 mL) at rt for 1 h. After neutralizing using HCl aq, the mixture was extracted with EtOAc. The combined extracts were dried over Na_2SO_4 and concentrated. The crude product was purified by flash column chromatography (eluent: hexane/EtOAc = 60/40 to 50/50) to give **S10** as a white solid (35.5 mg, 94%).



White solid. R_f 0.24 (Hexane/EtOAc = 1/1). M.p. 168.8–169.9 ºC.

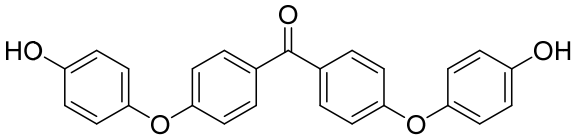
^{1}H NMR (DMSO-d_6, 400 MHz) δ: 9.33 (br, 2H), 7.27 (d, J = 8.7 Hz, 4H), 6.85-6.79 (m, 8H), 6.74 (m, 4H), 5.78 (br, 1H), 5.61 (s, 1H).

^{13}C NMR (DMSO-d_6, 101 MHz) δ: 157.1, 153.8, 148.0, 139.8, 127.6, 120.8, 116.6, 116.2, 73.3.

IR (KBr): 3302 br, 1606 s, 1504 s, 1457 s, 1374 m, 1337 m, 1262 s, 1228 s, 1165 s, 1103 m, 1010 m, 988 m, 879 m, 830 s, 758 m, 604 m, 561 m, 502 m.

HRMS (MALDI+, $[M]^+$): Calcd for $C_{25}H_{20}O_5$: 400.1305. Found: 400.1298.

Bis(4-(4-hydroxyphenoxy)phenyl)methanone (6). [CAS: 42592-70-1] This compound was synthesized using a literature procedure,[18] with minor modifications. DDQ (46.7 mg, 0.21 mmol) was added to a solution of 4,4'-(((hydroxymethylene)bis(4,1-phenylene))bis(oxy))diphenol (77.8 mg, 0.19 mmol) in dioxane (1.2 mL). The reaction mixture immediately turned deep green. The mixture was stirred at rt for 14 h. The resulting white precipitate was filtered off, and dioxane was evaporated in vacuo. The obtained crude compound was purified by flash column chromatography (eluent: hexane/EtOAc = 60/40 to 35/65) to give **6** as a white solid (65.8 mg, 85%).



White solid. R_f 0.32 (Hexane/EtOAc = 1/1).

^{1}H NMR (DMSO-d_6, 400 MHz) δ: 9.50 (s, 1H), 7.70 (d, J = 8.7 Hz, 2H), 6.98-6.95 (m, 4H), 6.82 (d, J = 9.2 Hz, 2H).

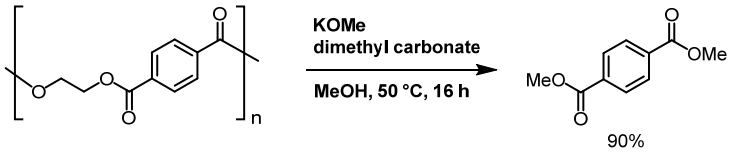
^{13}C NMR (DMSO-d_6, 101 MHz) δ: 193.1, 162.1, 154.7, 146.5, 132.1, 131.1, 121.8, 116.5, 115.9.

HRMS (EI+, M^+): Calcd for $C_{25}H_{18}O_5$: 398.1154. Found: 398.1162.

XI. Orthogonal Depolymerization

Post-consumer polyethylene terephthalate (PET) in beverage bottles was used for this study. PET beverage bottles were washed with water, dried, and shredded to flake form (ca. 1 mm squares) prior to use.

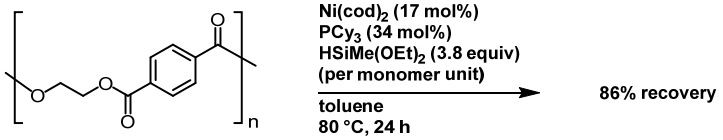
Depolymerization of PET under basic conditions



This reaction was conducted based on the literature procedure.[19]

PET (108.3 mg, 0.53 mmol), KOMe (10.8 mg, 0.15 mmol), dimethyl carbonate (1.5 mL) and MeOH (0.20 mL) were added to a 10 mL vial with a Teflon-sealed screwcap with a magnetic stirring bar. The reaction mixture was degassed and stirred at 50 °C for 16 h. The resulting mixture was then evaporated to dryness, and the residue was purified by flash column chromatography (eluent: hexane/EtOAc = 85/15 to 50/50) to give dimethyl terephthalate as a white solid (98.9 mg, 90%). The spectroscopic data of the product was in agreement with that reported in the literature.

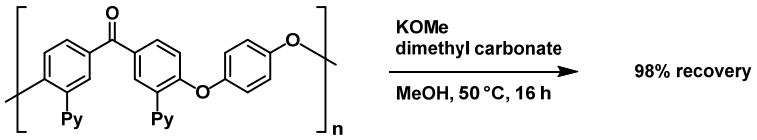
Attempted depolymerization of PET under Ni-catalyzed conditions



In a glovebox filled with nitrogen, PET (52.2 mg, 0.27 mmol), $Ni(cod)_2$ (13.0 mg, 0.047 mmol), PCy_3 (25.6 mg, 0.091 mmol), hydrosilane (137.2 mg, 1.02 mmol) and toluene (0.50 mL) were added to a 5 mL vial

with a Teflon-sealed screwcap. The mixture was stirred at 80 °C for 24 h. The resulting mixture was filtered, and the residue was washed with MeOH. PET was recovered (45.0 mg, 86%). This result is consistent with the fact that in Ni-catalyzed reductive C–O bond cleavage reactions with hydrosilane, an ester group remains unreacted.[20]

Attempted depolymerization of DG-PEEK1 under basic conditions



DG-PEEK1 (90.3 mg, 0.20 mmol), KOMe (4.4 mg, 0.063 mmol), dimethyl carbonate (0.6 mL) and MeOH (0.080 mL) were added to a 5 mL vial with a Teflon-sealed screwcap with a magnetic stirring bar. The reaction mixture was degassed and stirred at 50 °C for 18 h. The resulting mixture was filtered, and the residue was washed with MeOH. **DG-PEEK1** was recovered (88.2 mg, 98%).

XII. References

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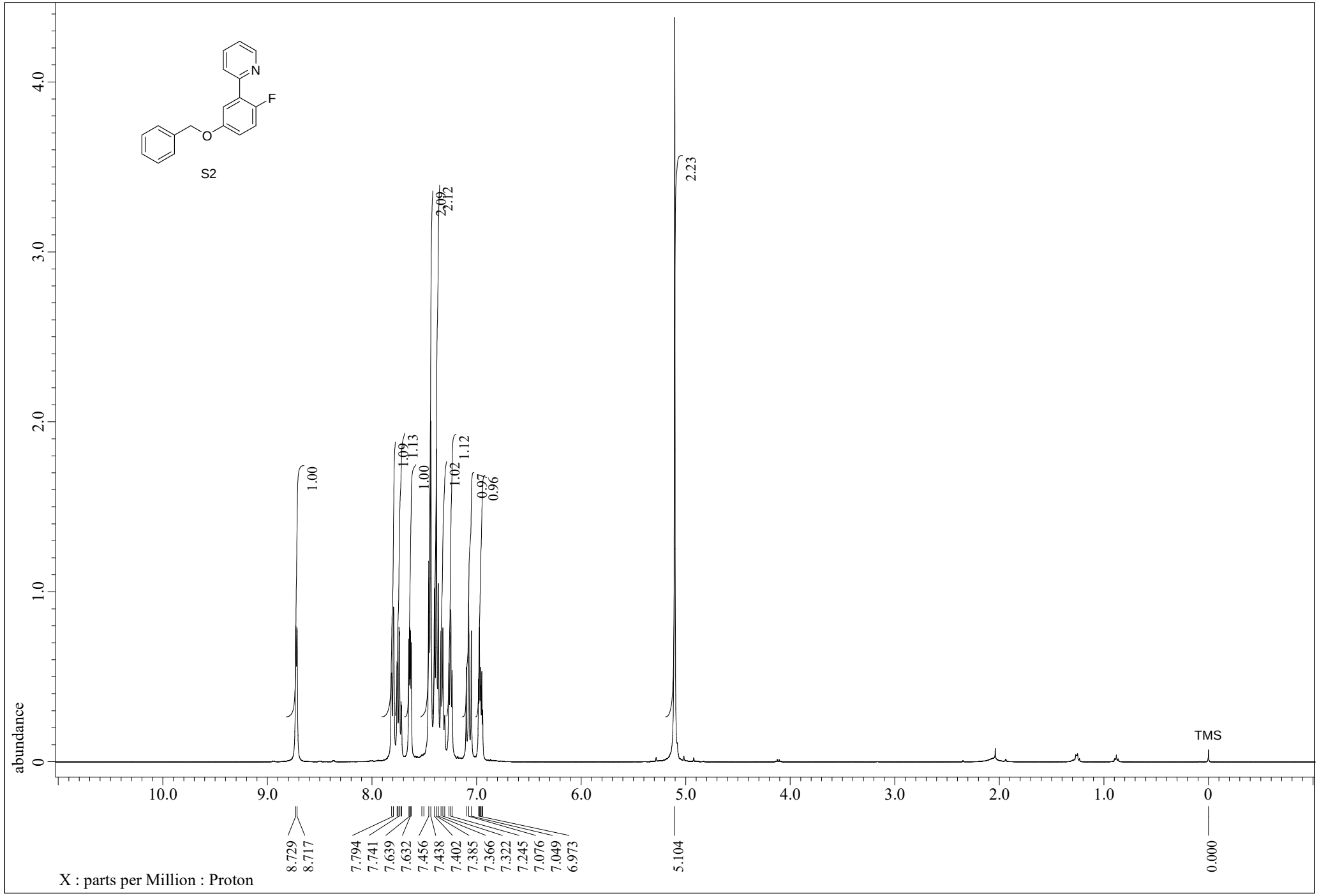
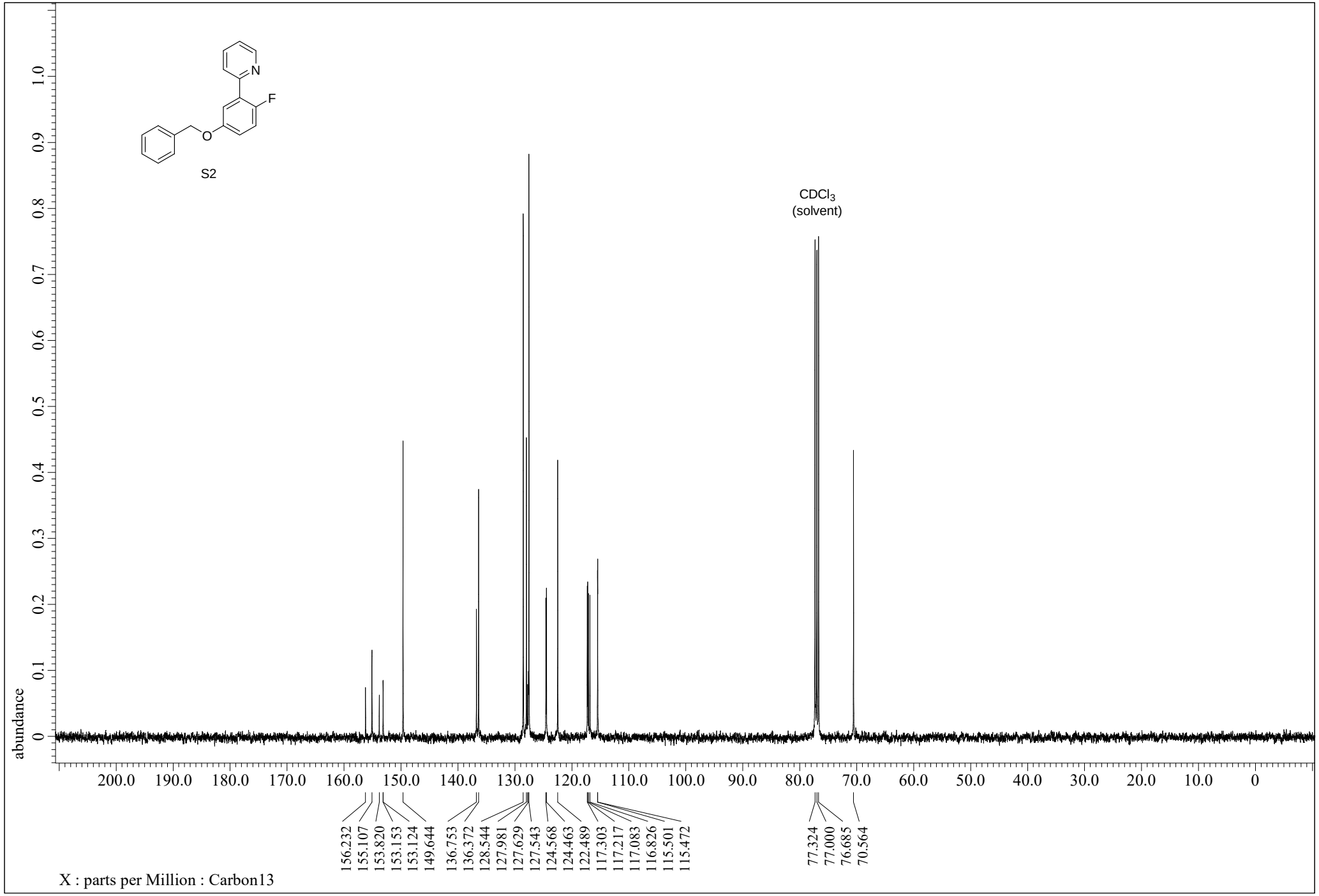
XIII. Copies of NMR spectra

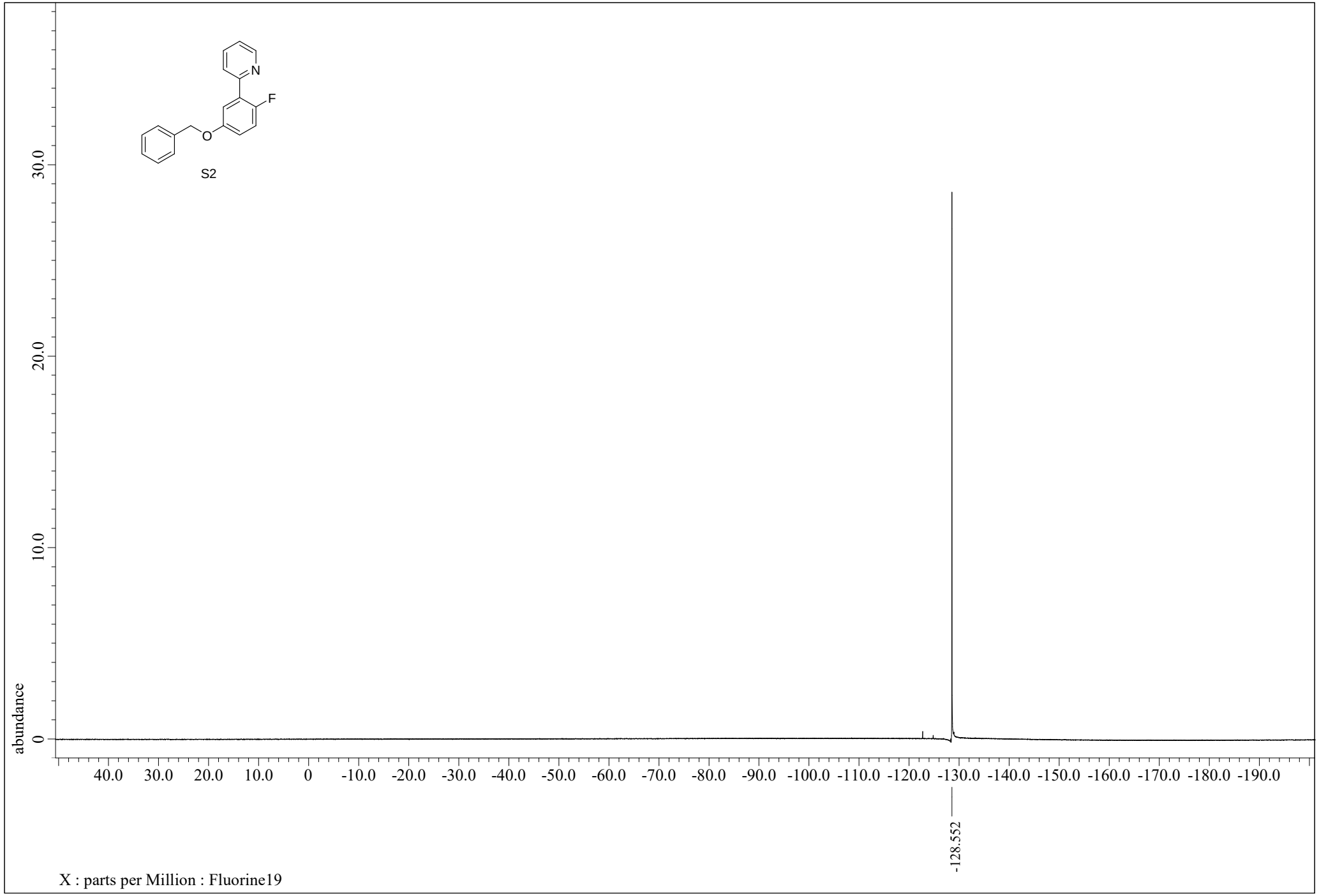
^{1}H NMR (400 MHz, $CDCl_3$)



^{13}C NMR (101 MHz, $CDCl_3$)



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



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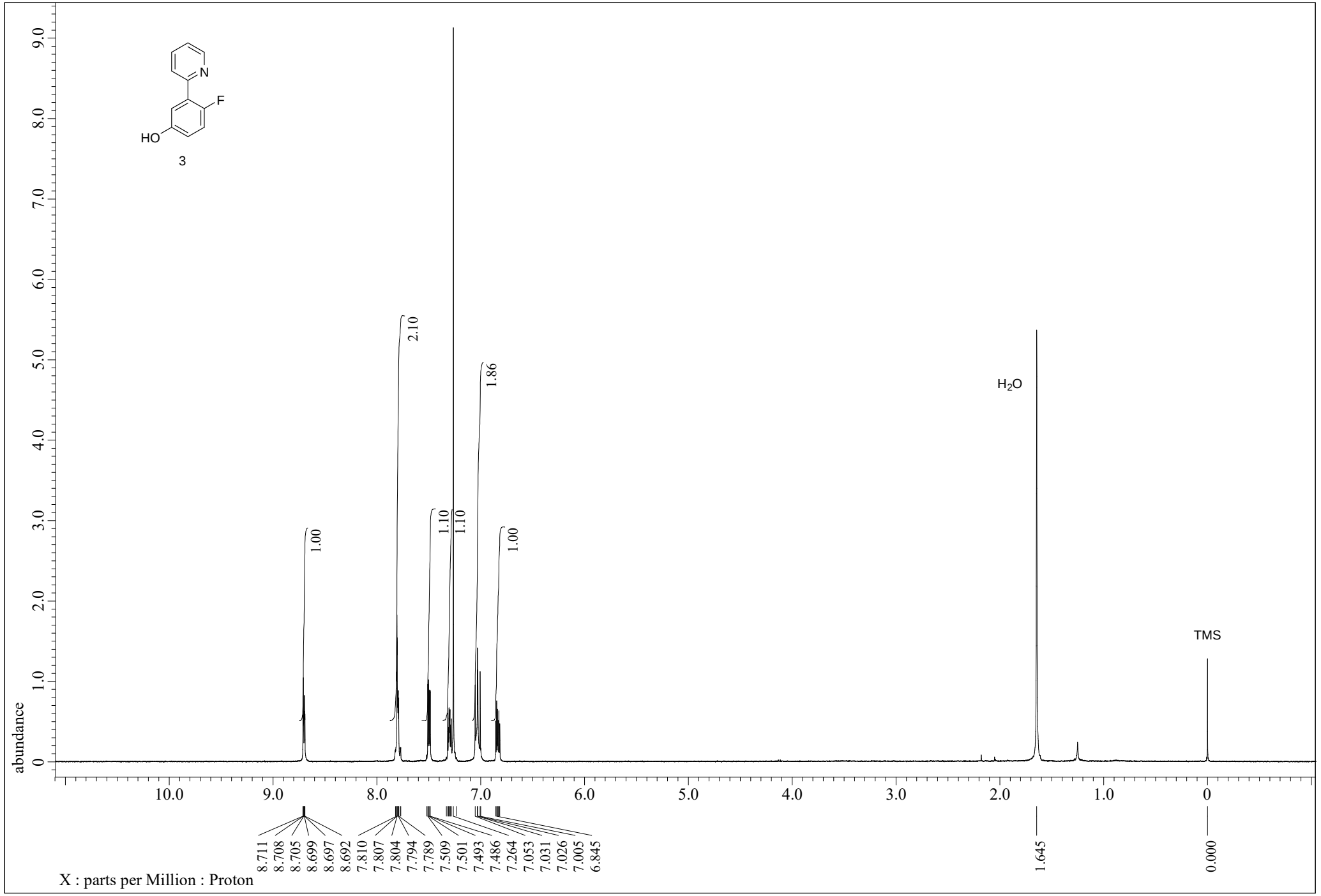

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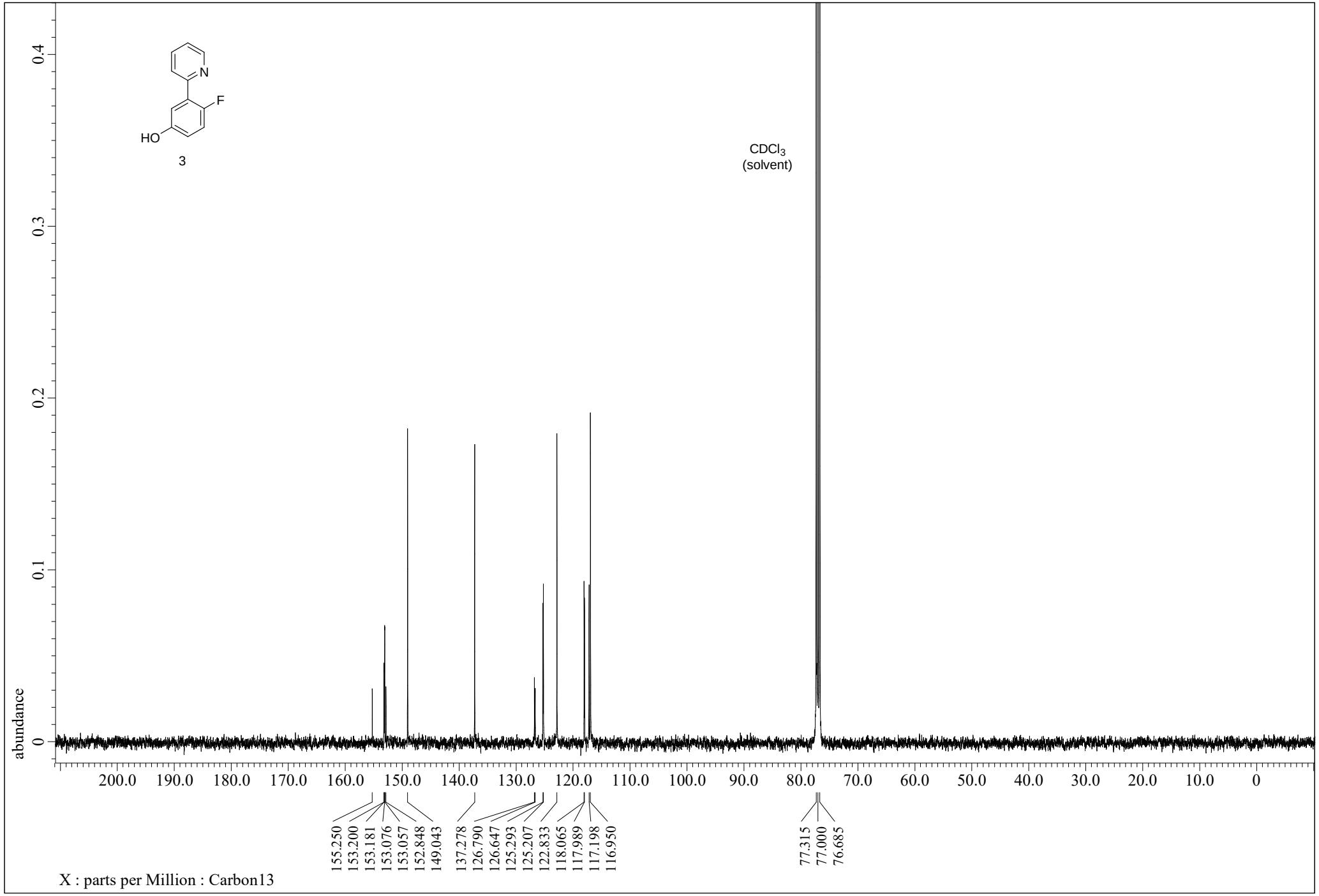

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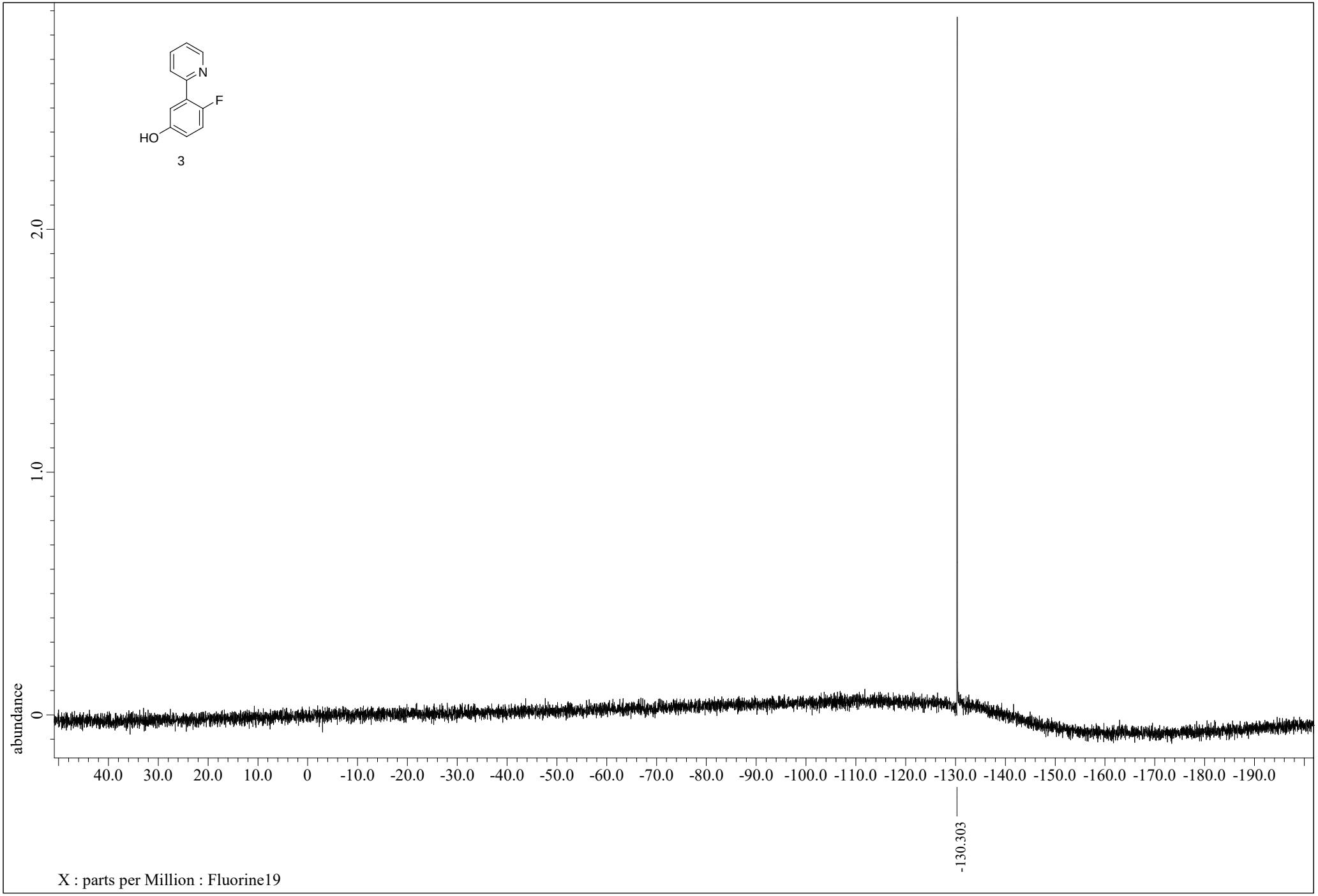
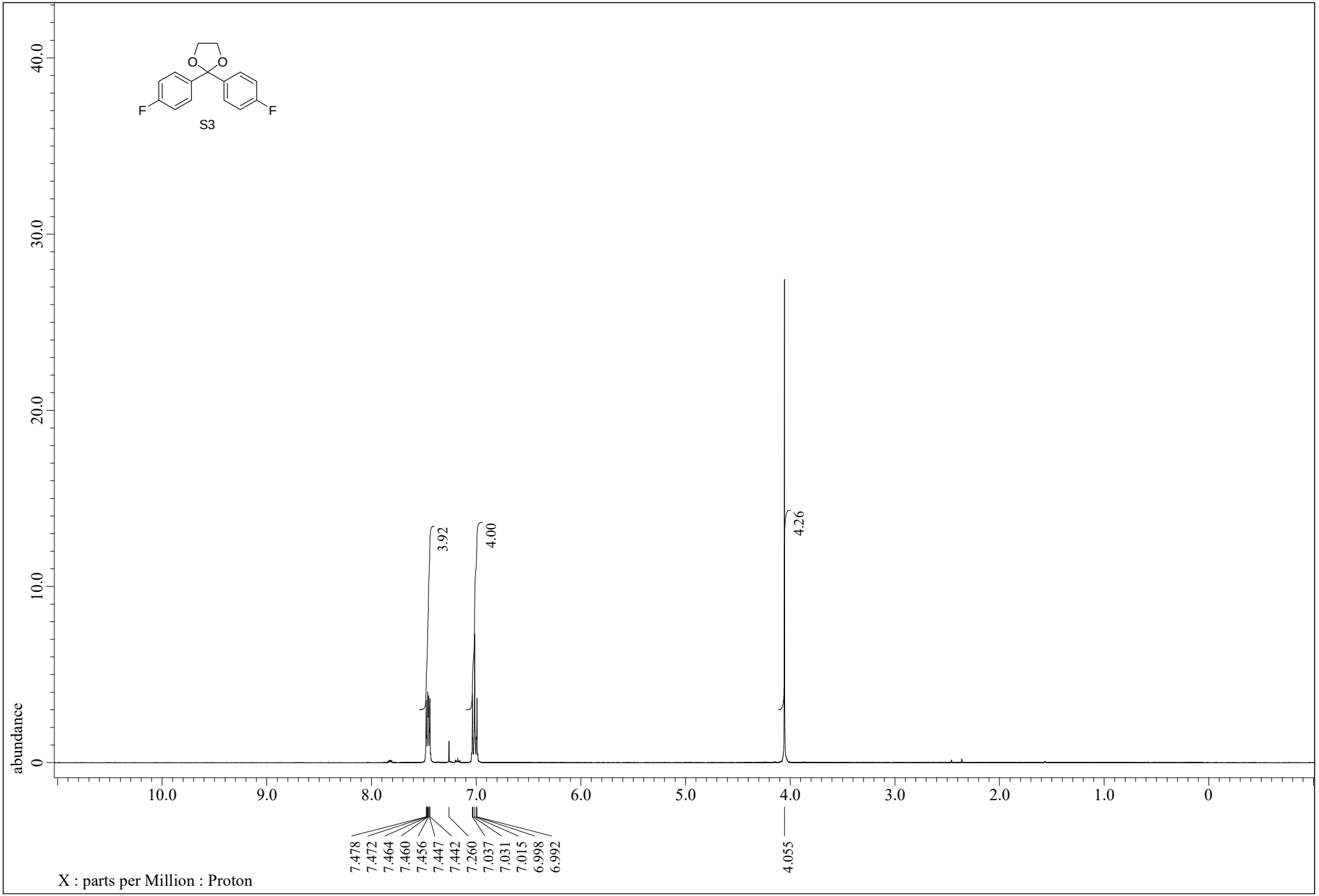
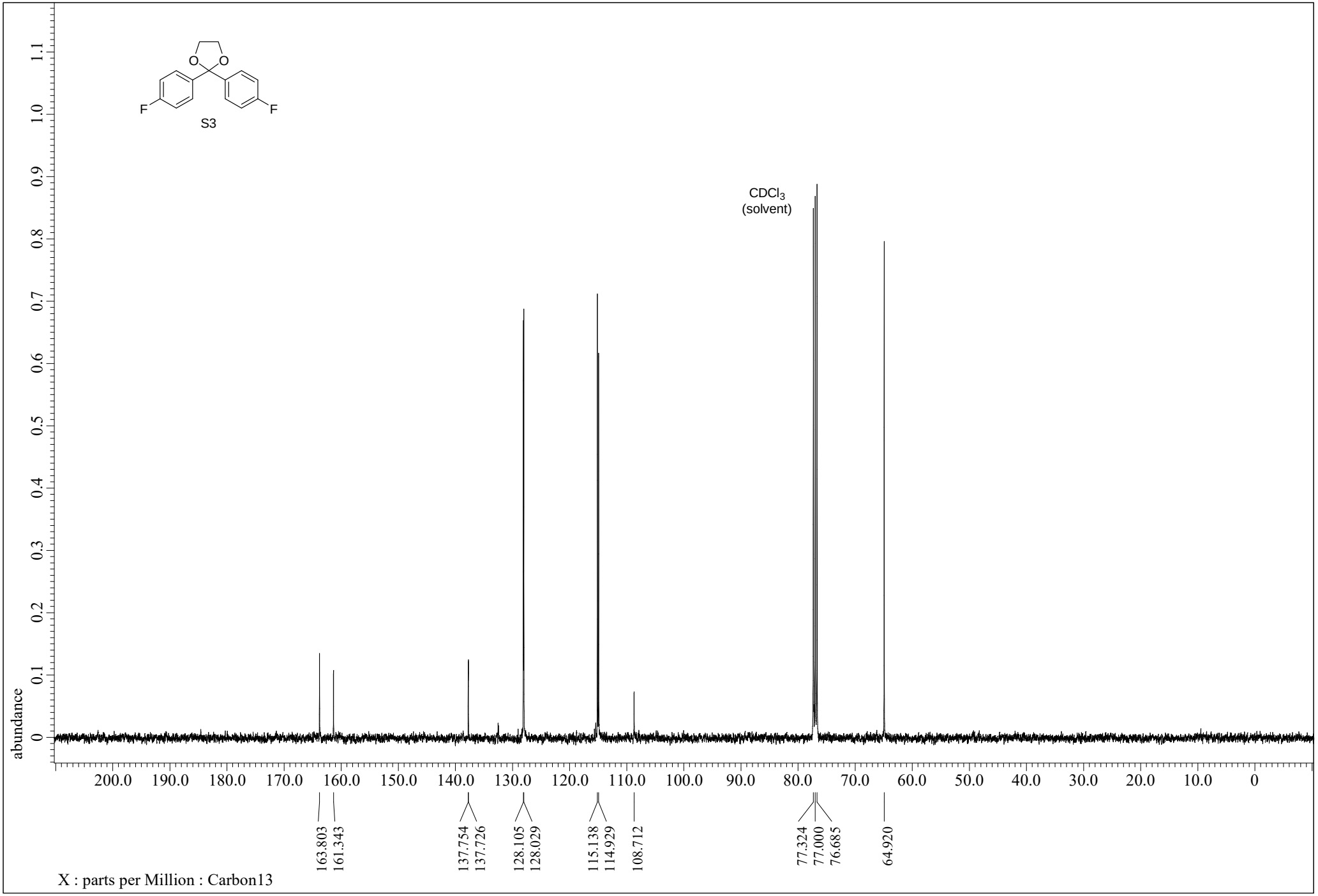


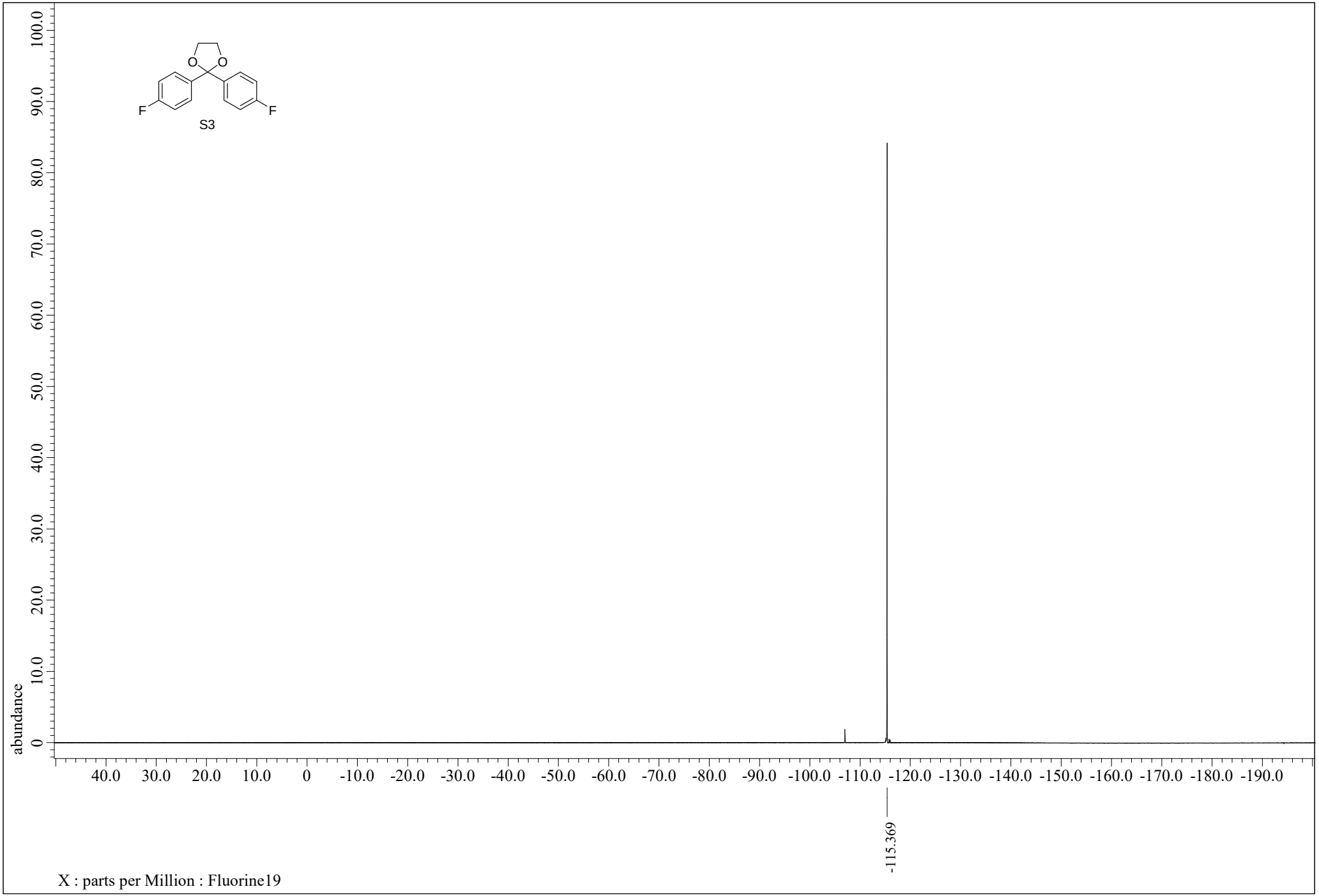
^{1}H NMR (400 MHz, $CDCl_3$)



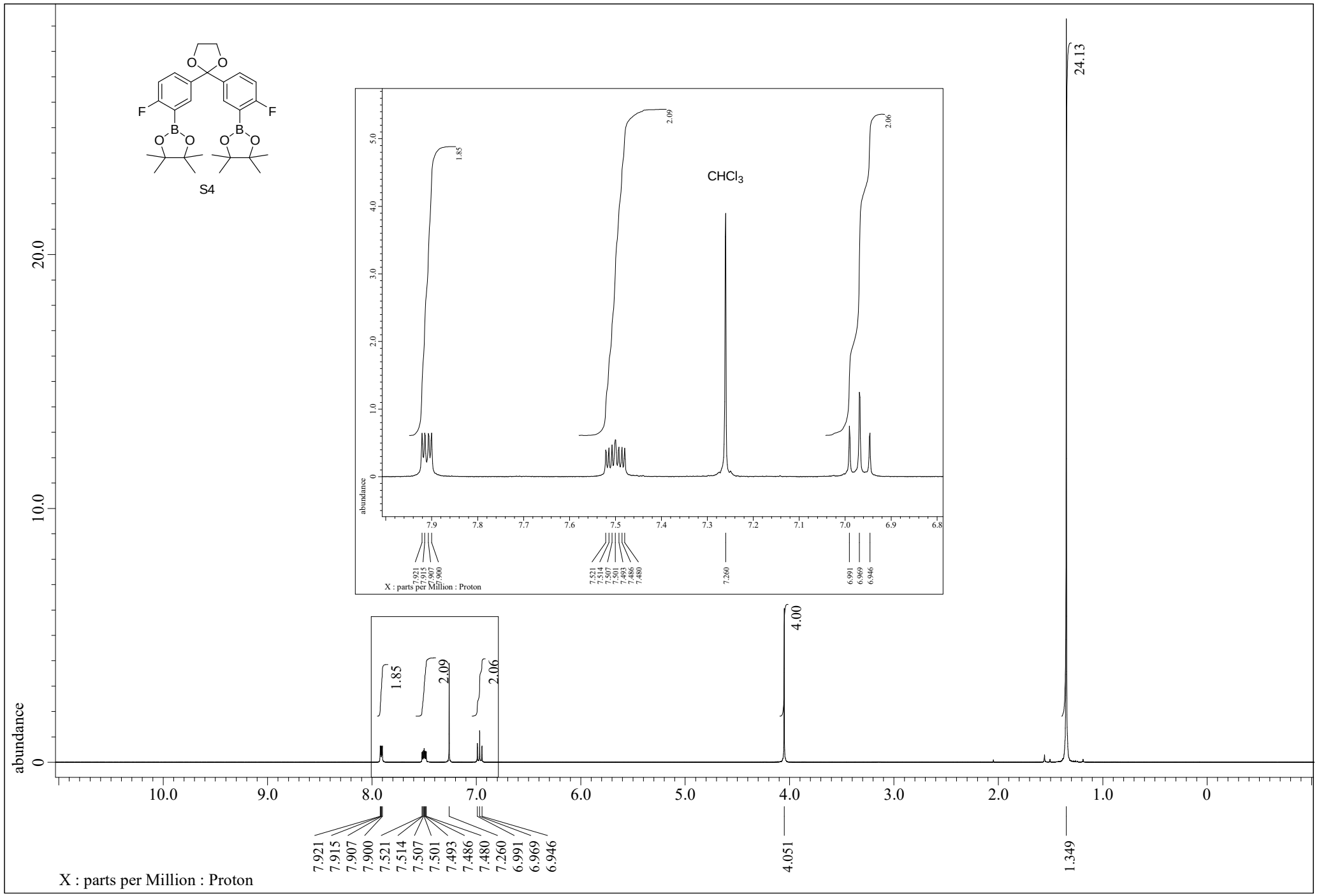
^{13}C NMR (101 MHz, $CDCl_3$)



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



^{1}H NMR (400 MHz, $CDCl_3$)



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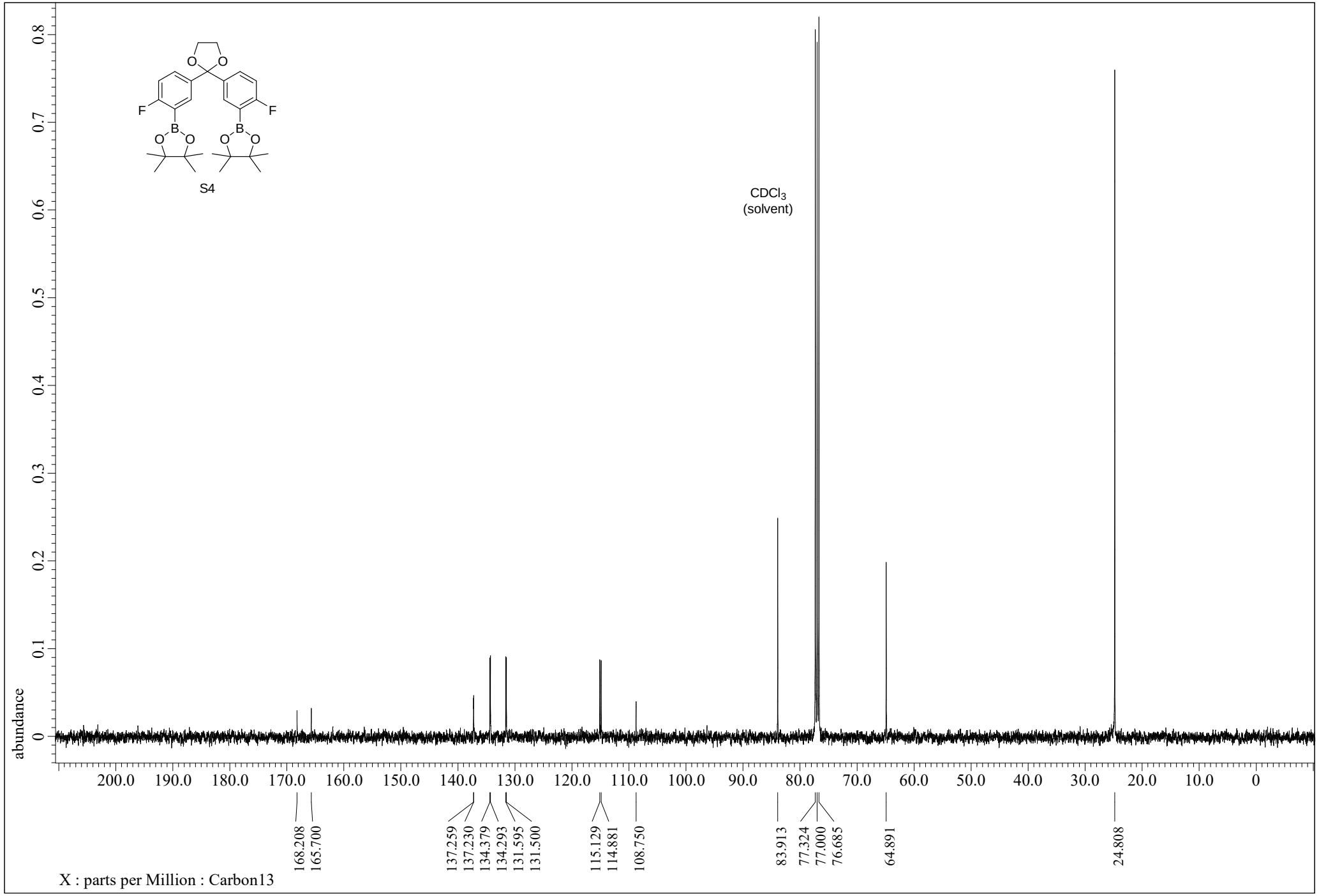
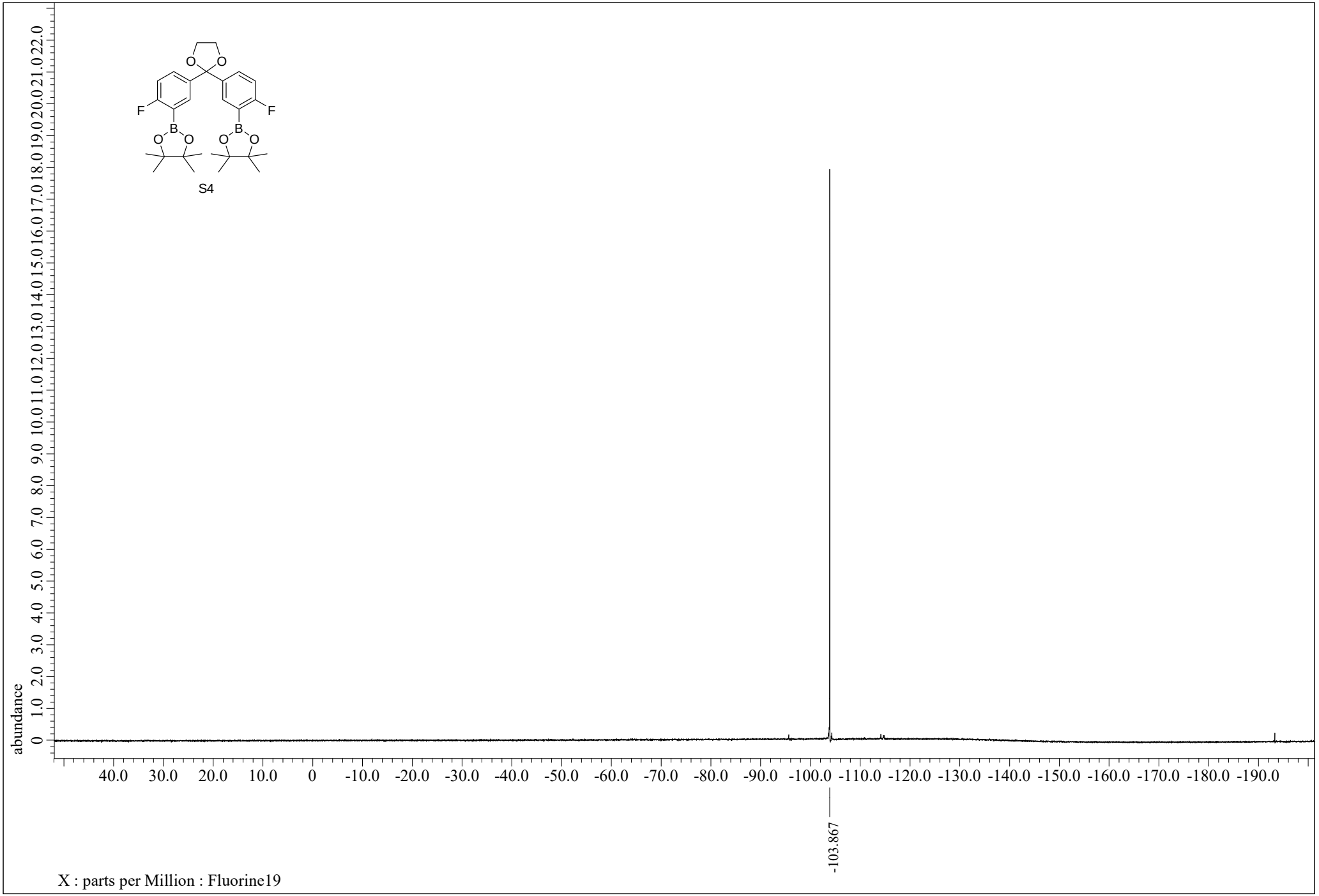
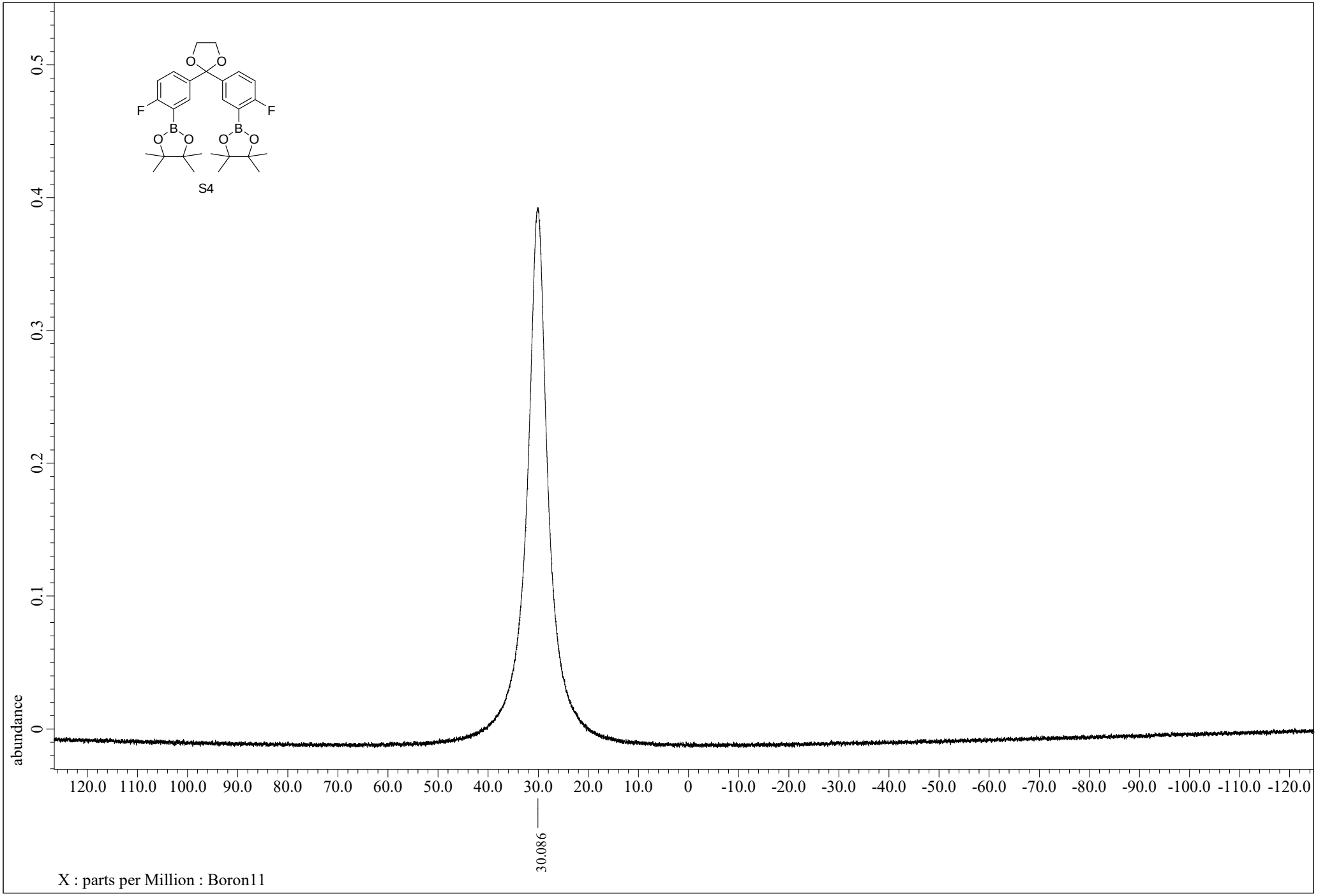


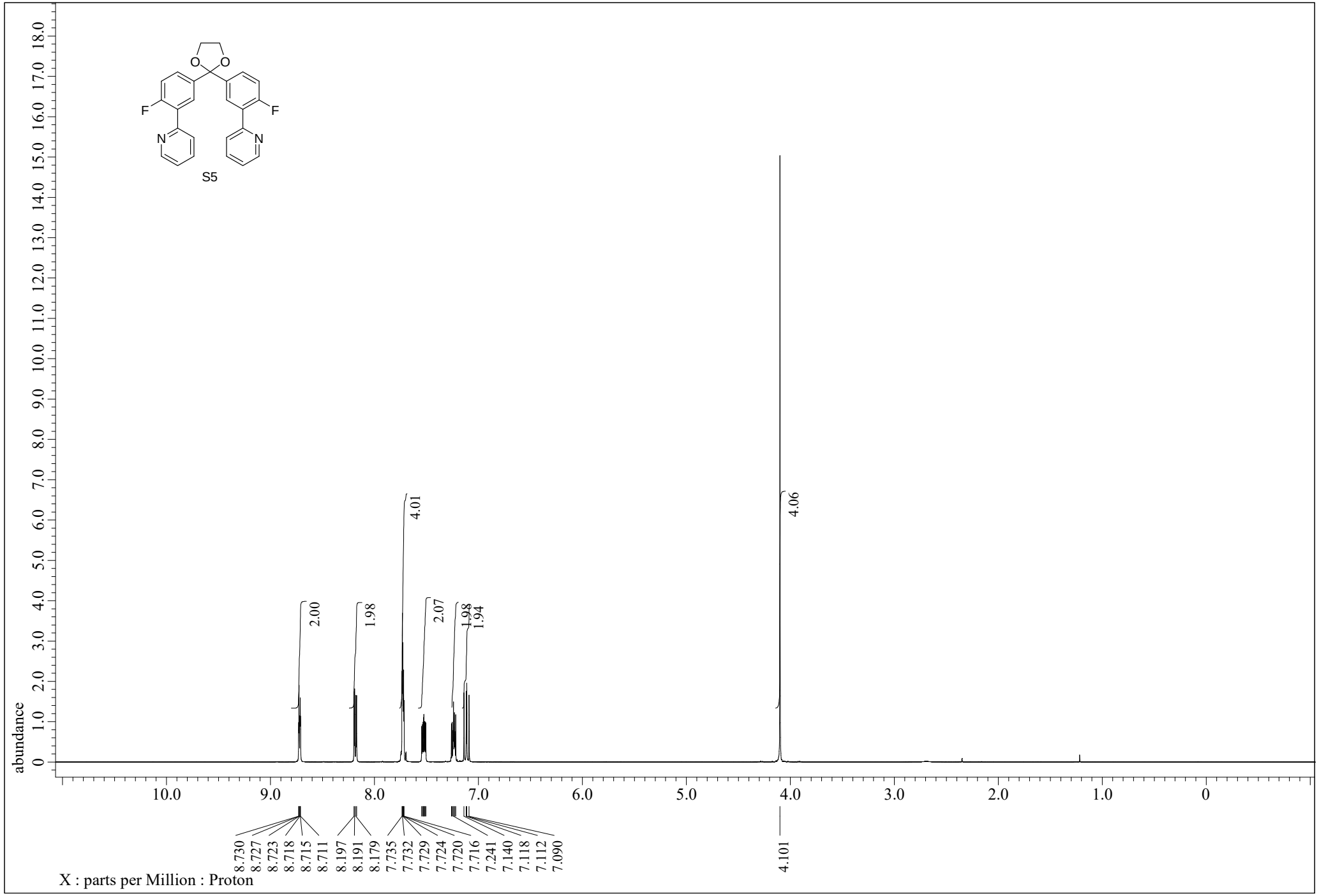
^{19}F NMR (376 MHz, $CDCl_3$)



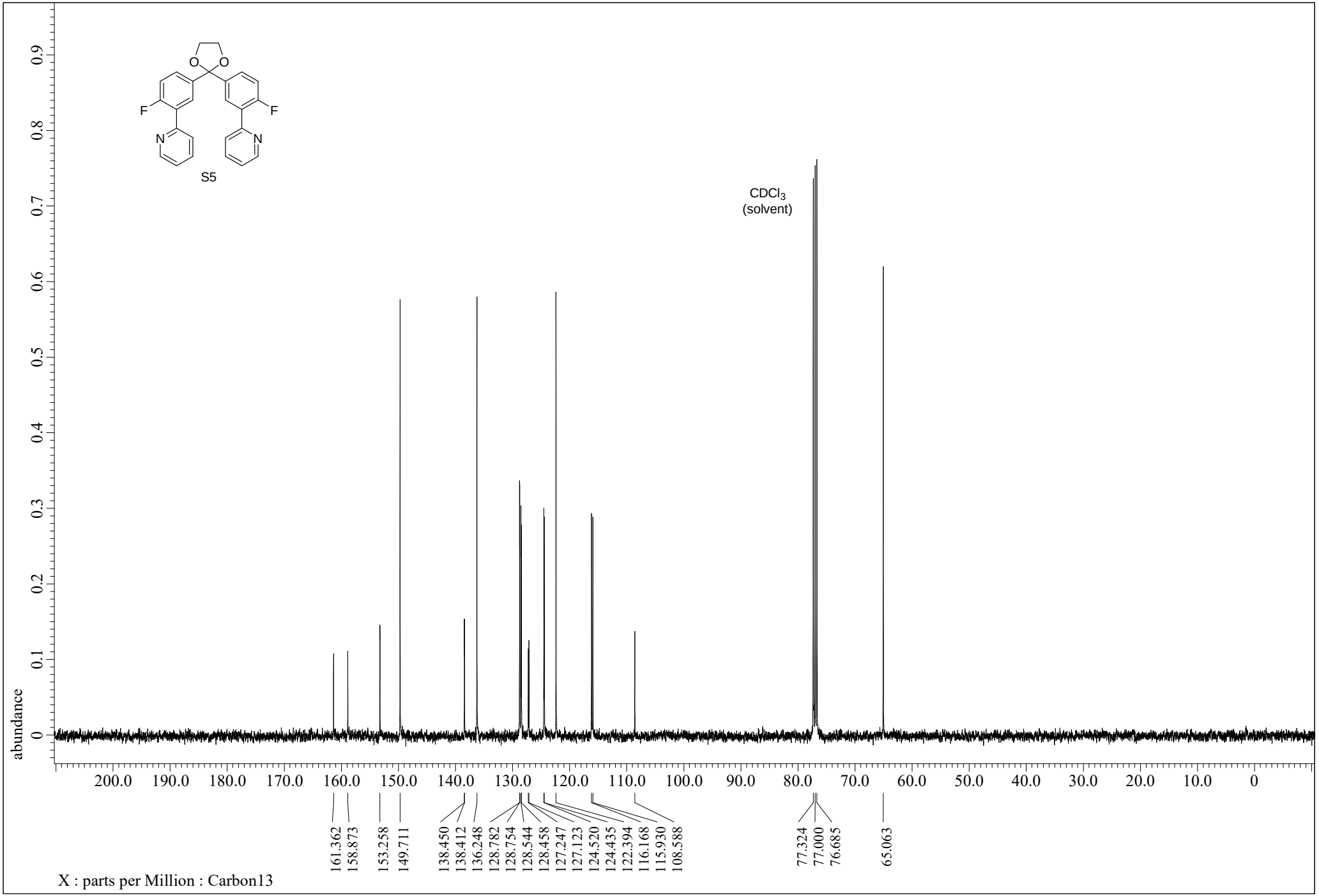
^{11}B NMR (128 MHz, $CDCl_3$)



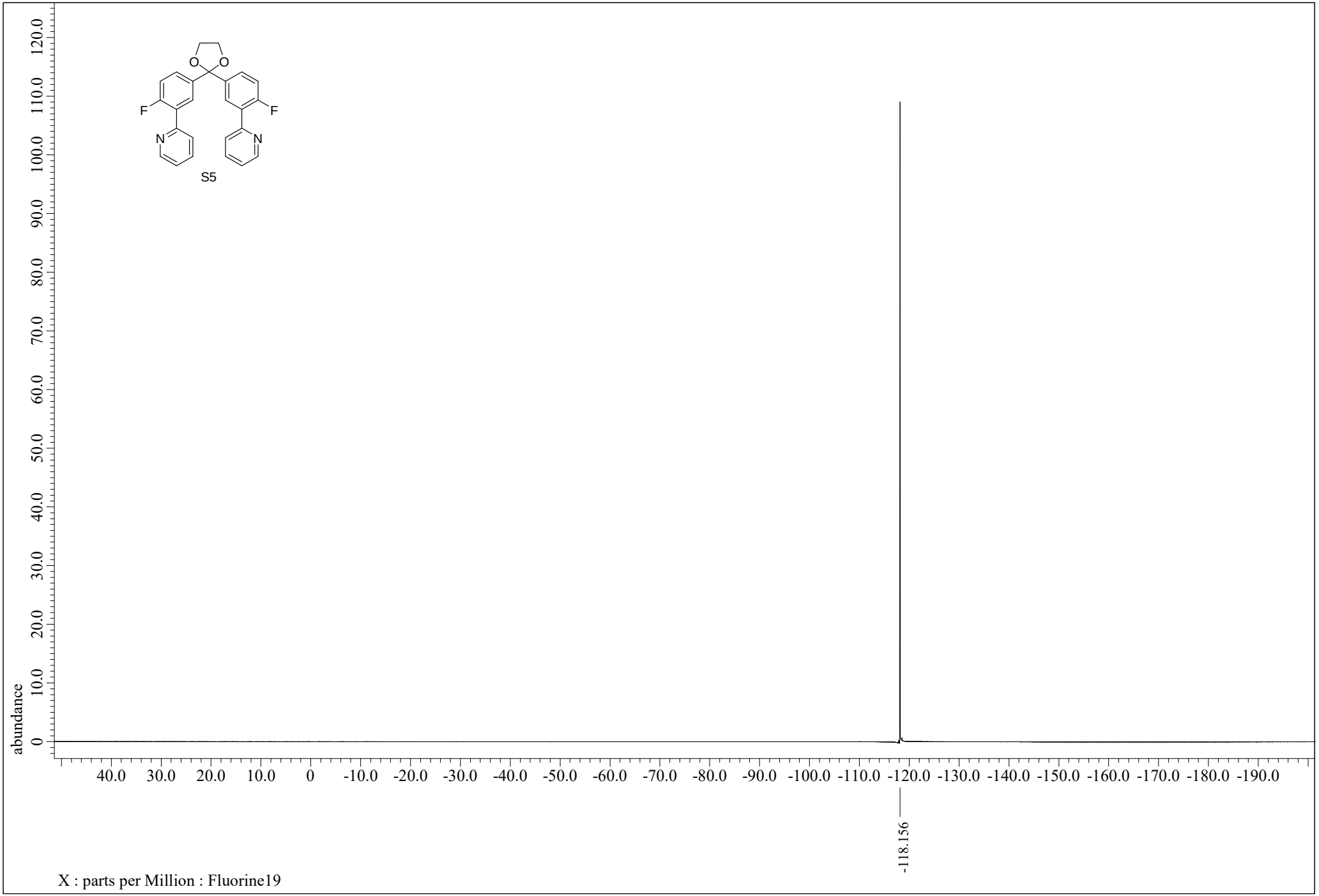
^{1}H NMR (400 MHz, $CDCl_3$)



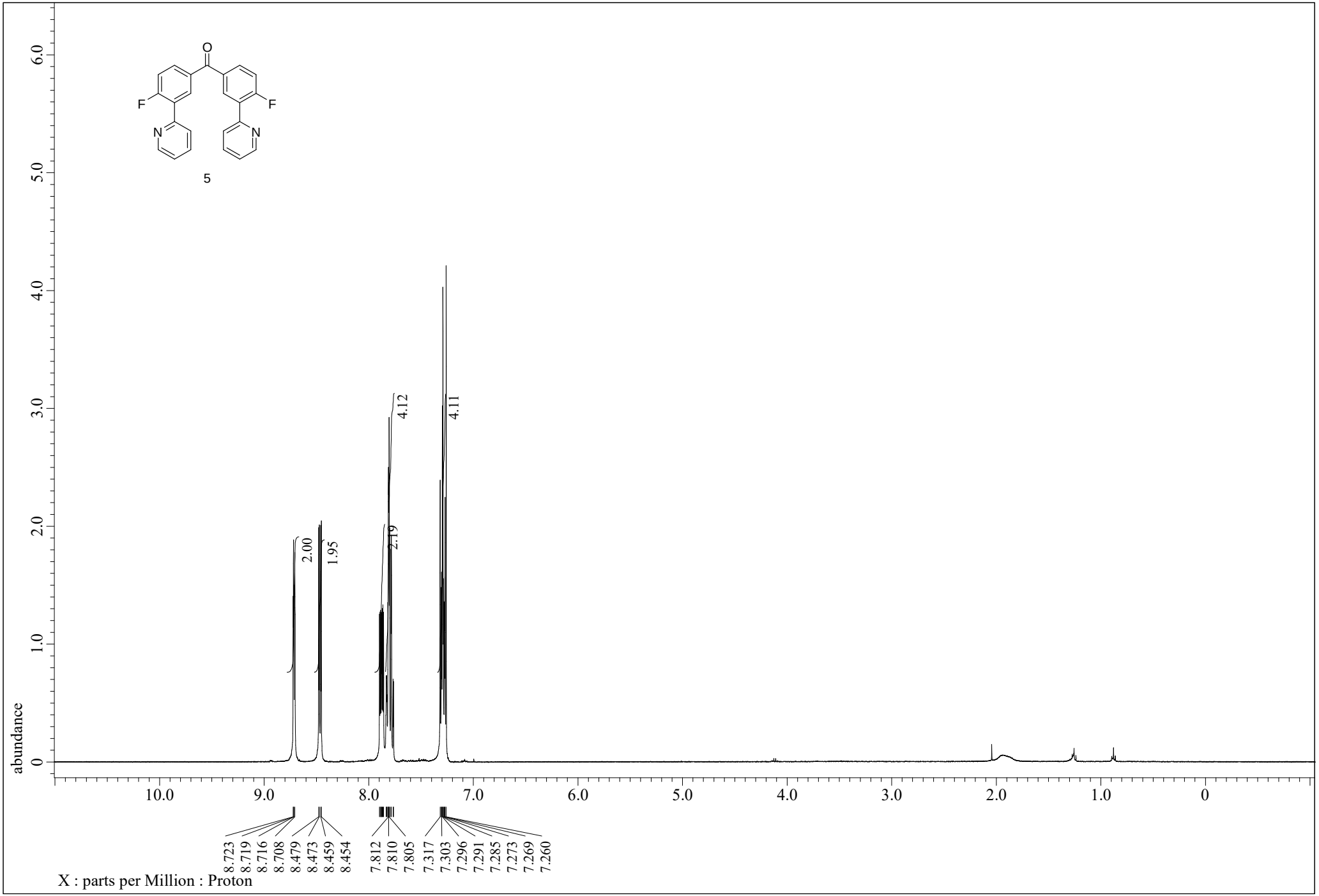
^{13}C NMR (101 MHz, $CDCl_3$)



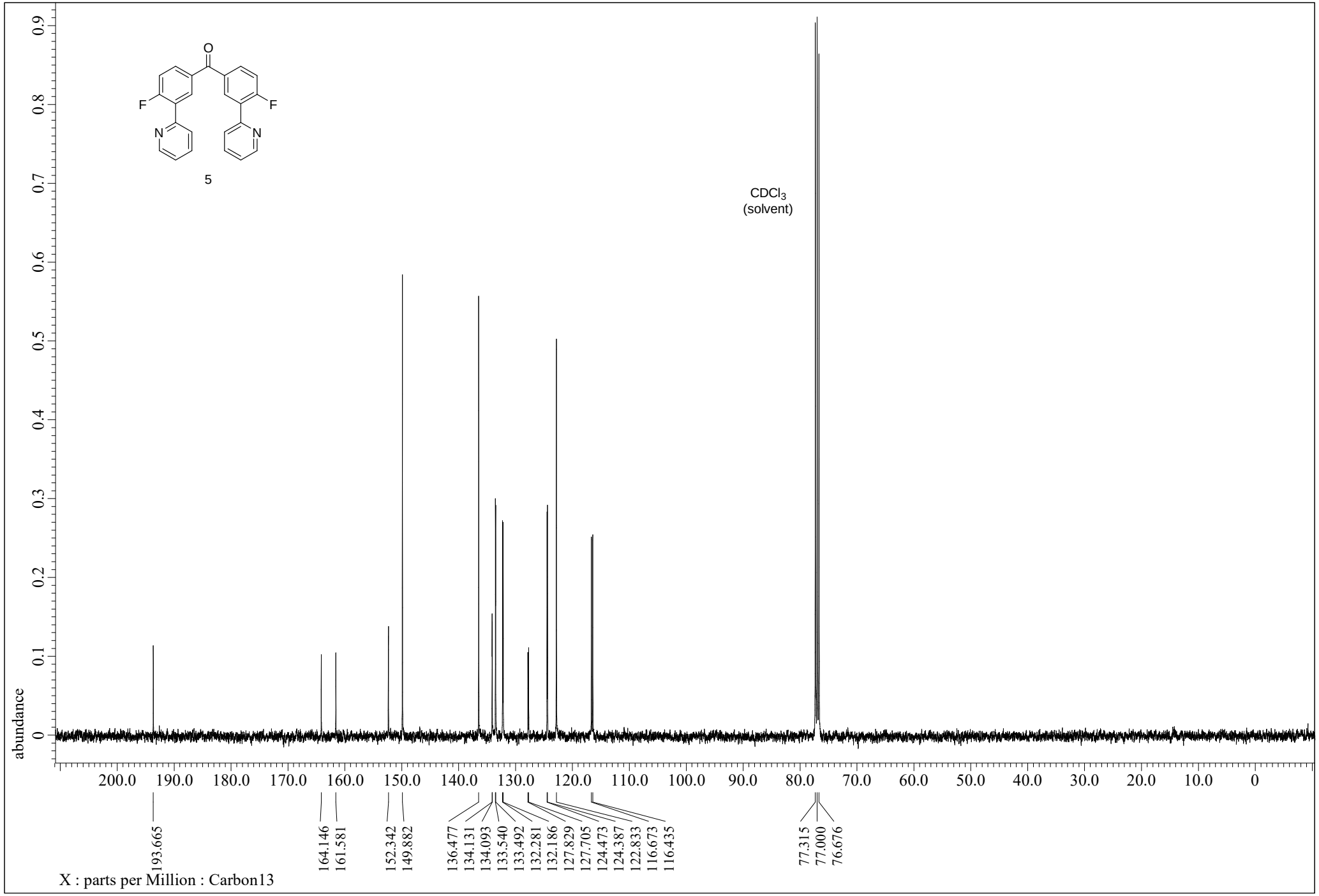
^{19}F NMR (376 MHz, $CDCl_3$)



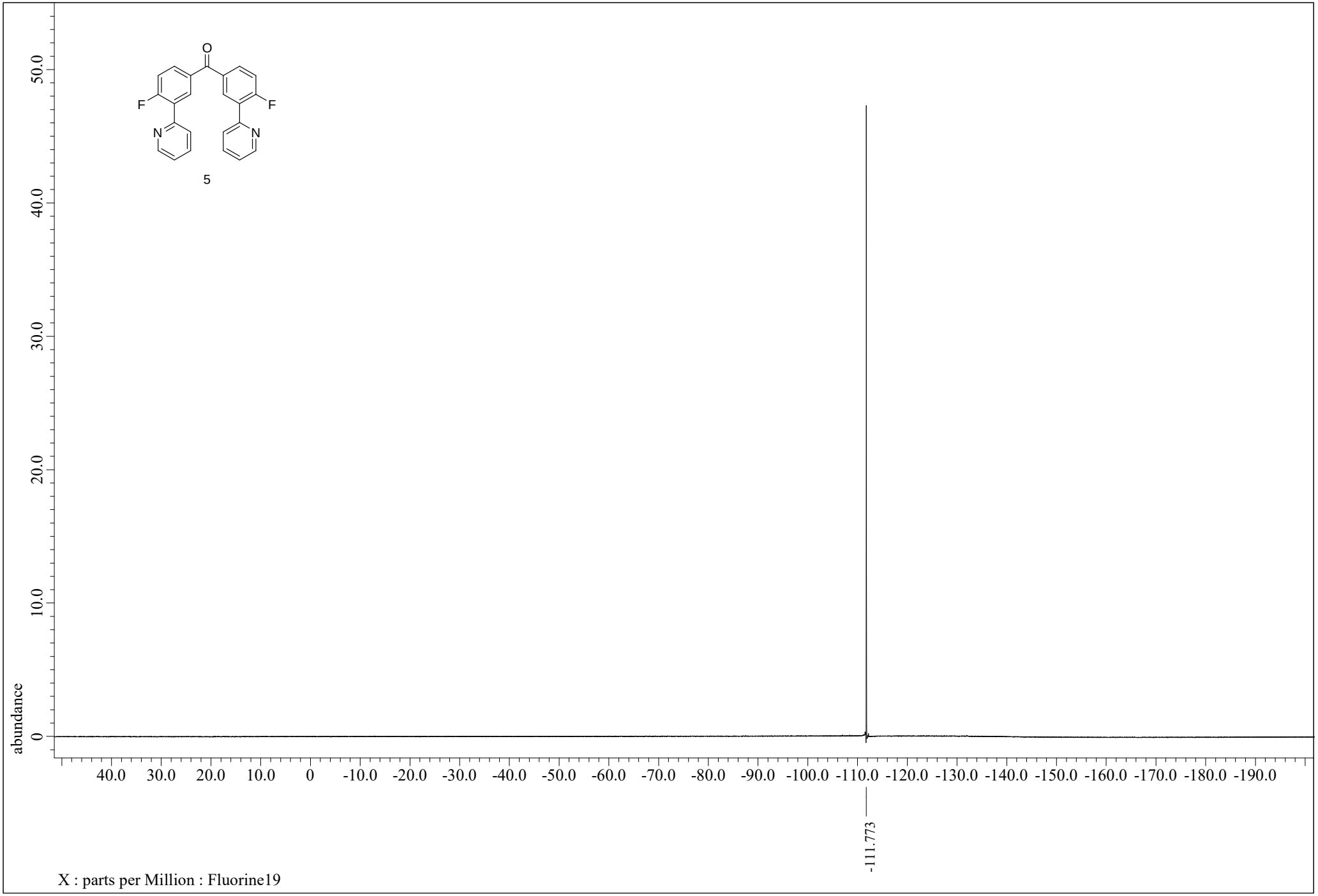
^{1}H NMR (400 MHz, $CDCl_3$)



^{13}C NMR (101 MHz, $CDCl_3$)



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



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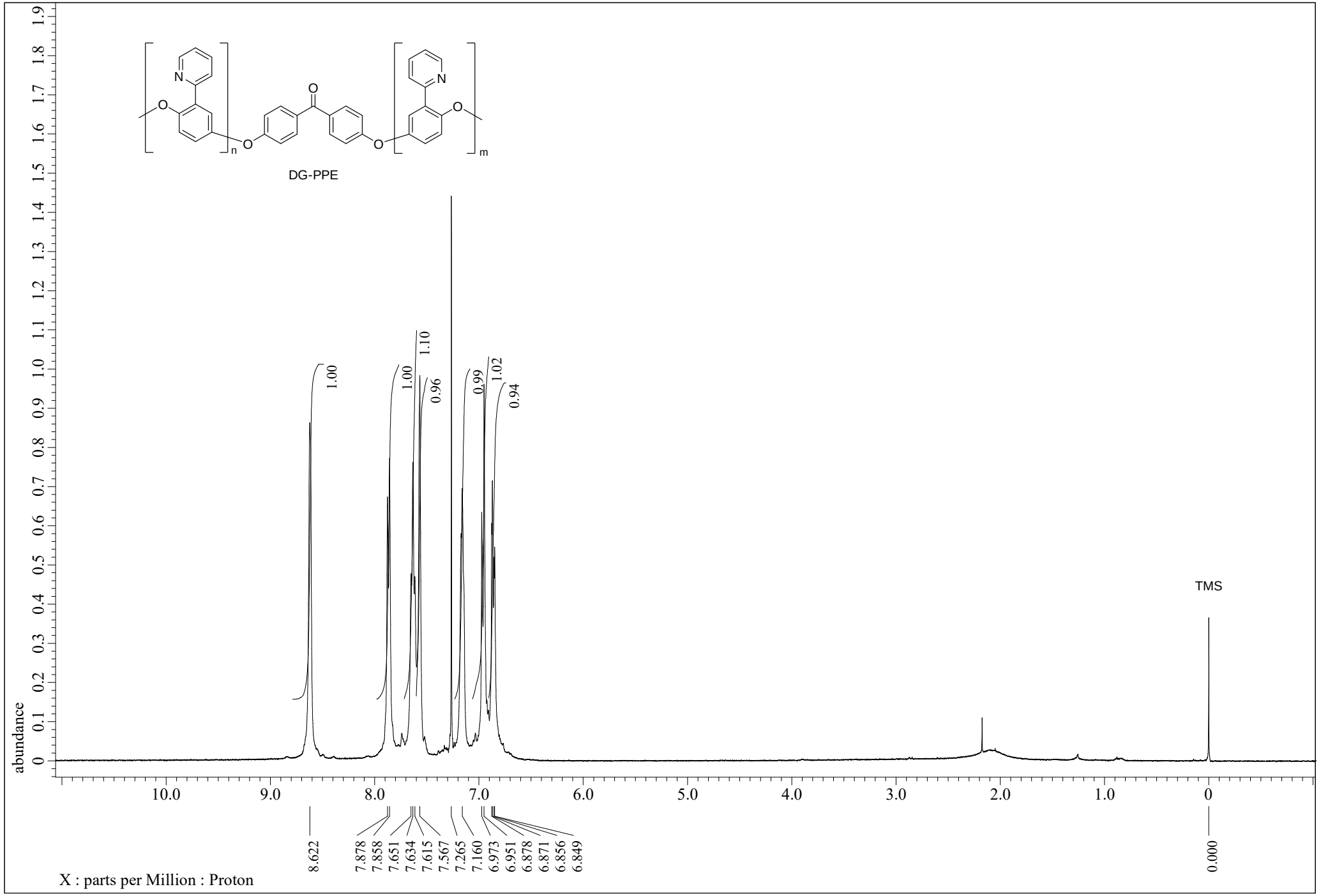

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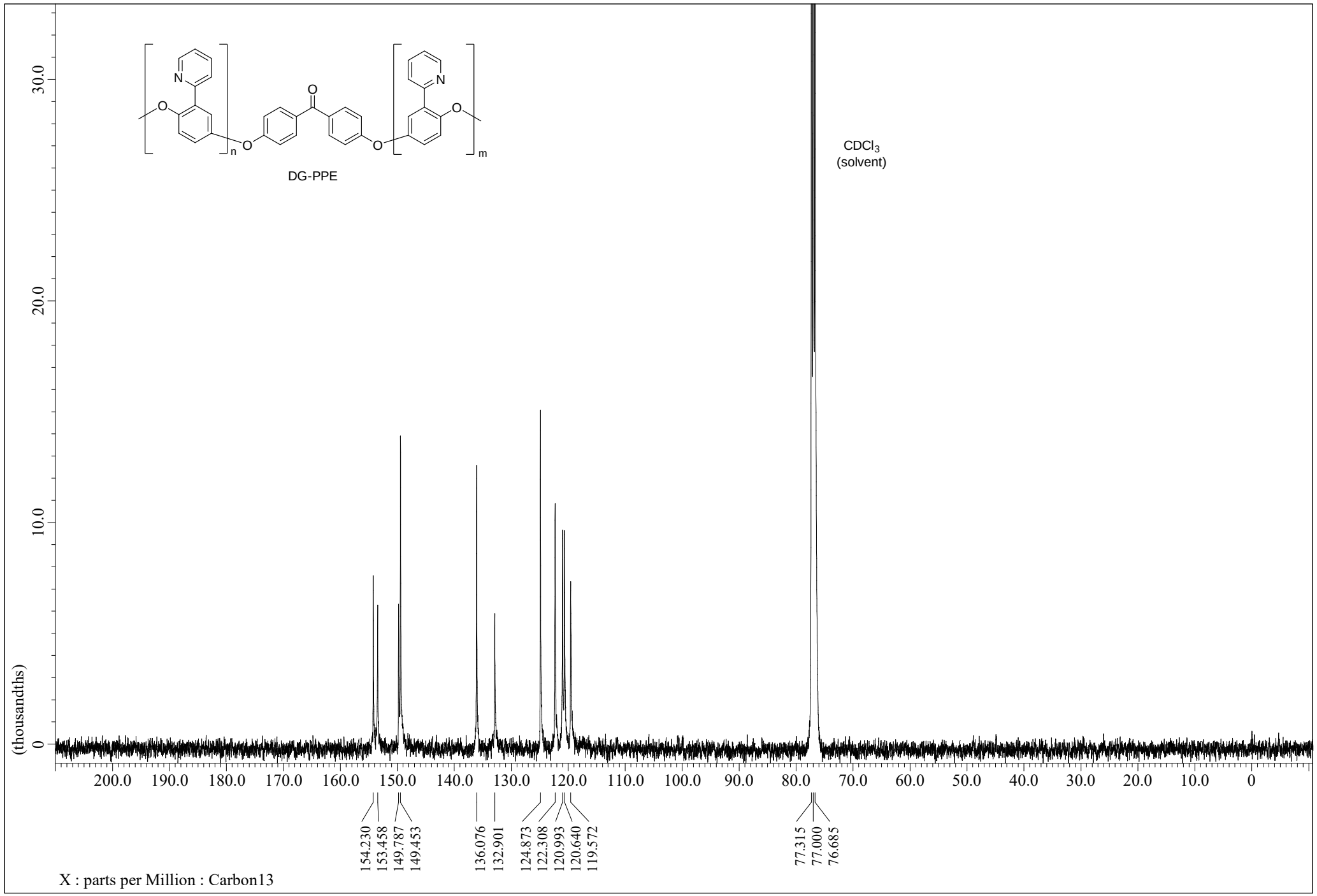
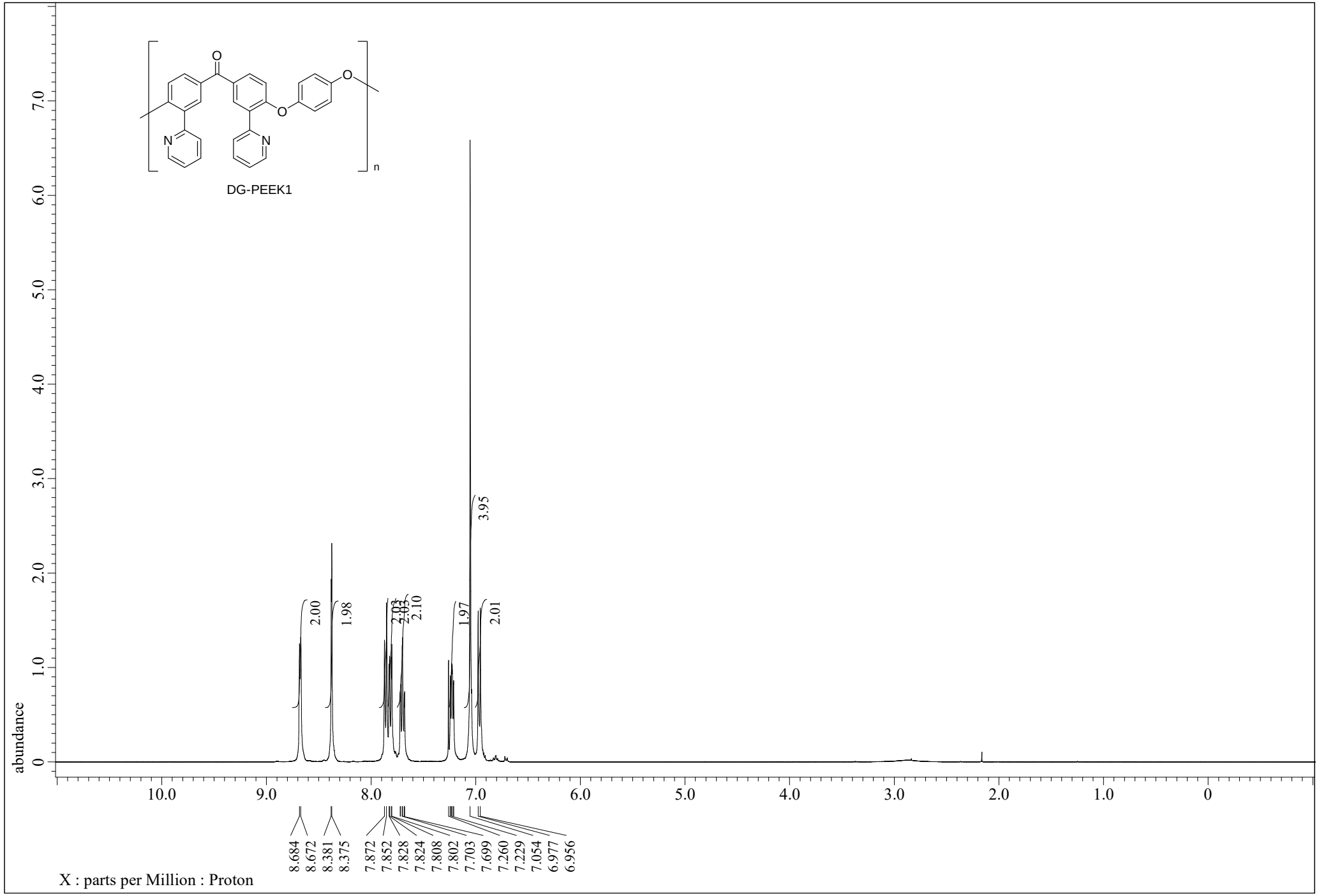
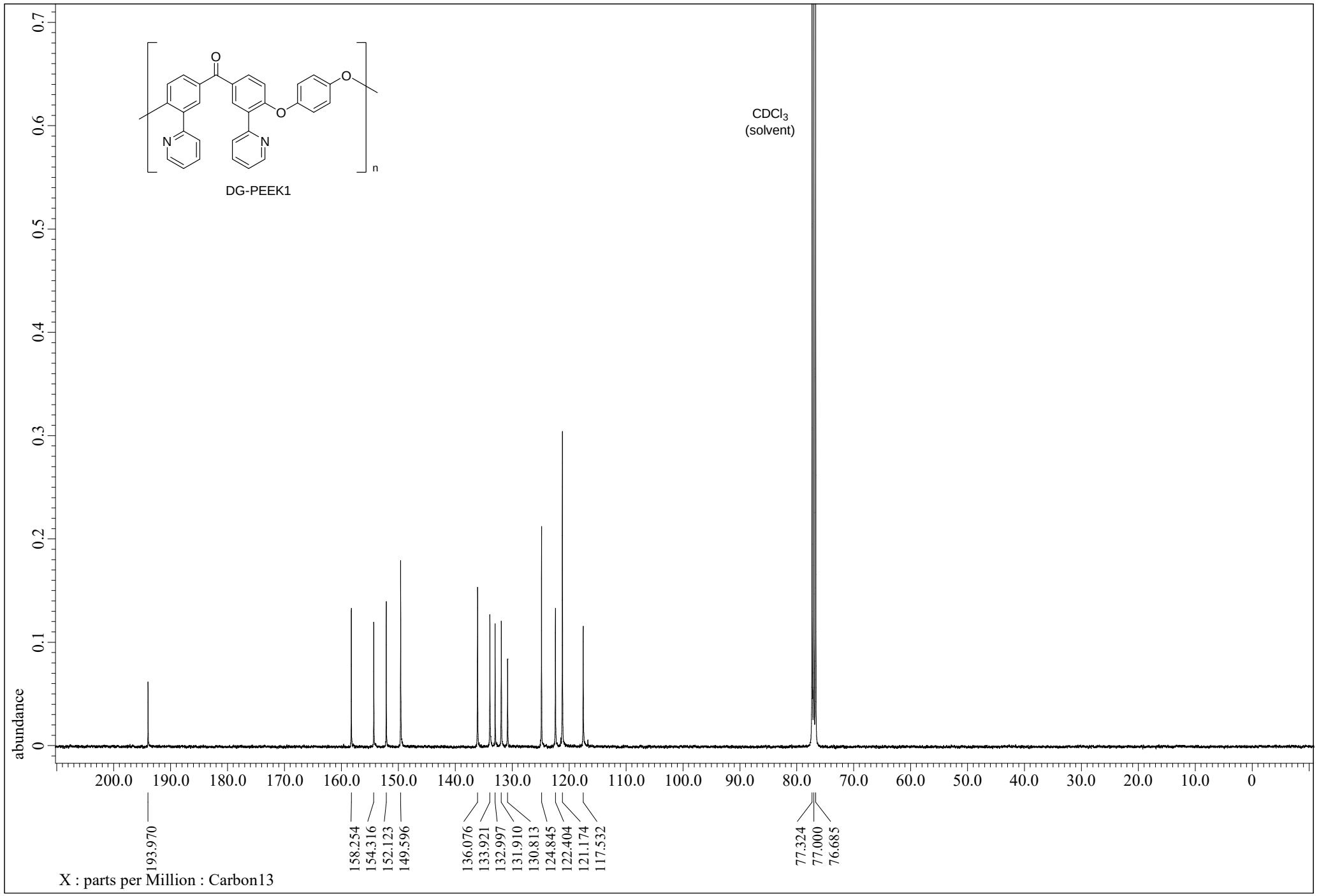


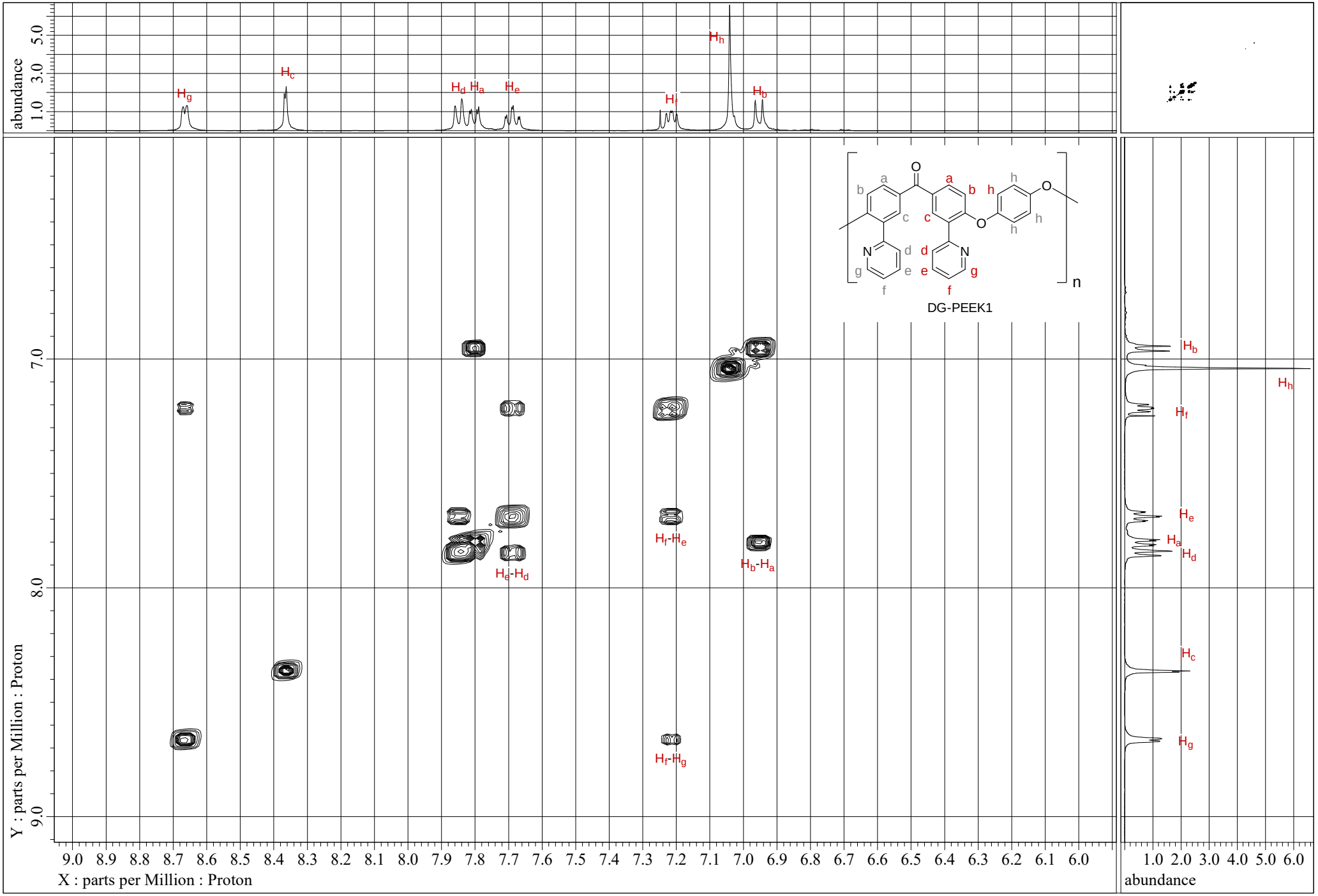
^{1}H NMR (400 MHz, $CDCl_3$)



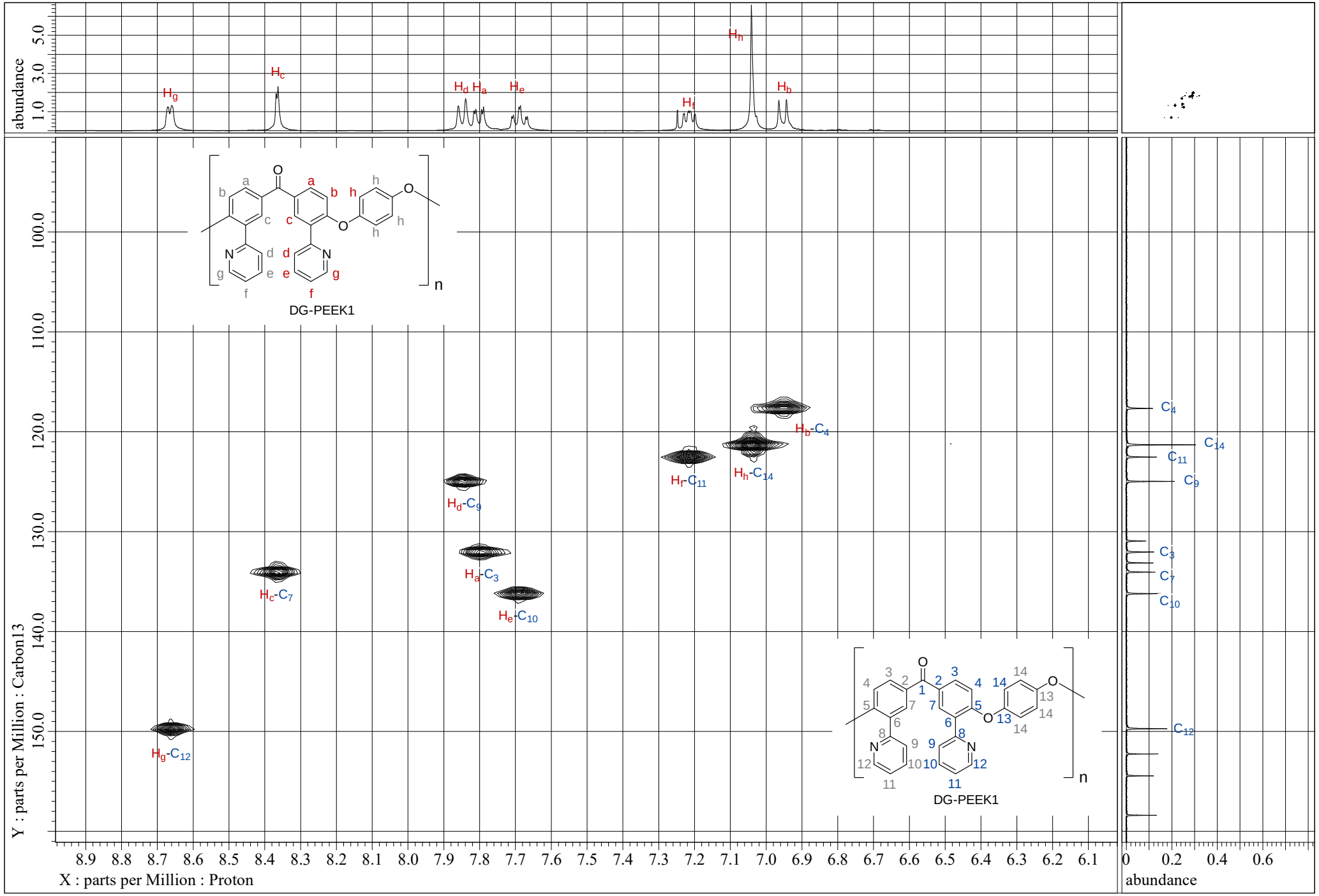
^{13}C NMR (101 MHz, $CDCl_3$)



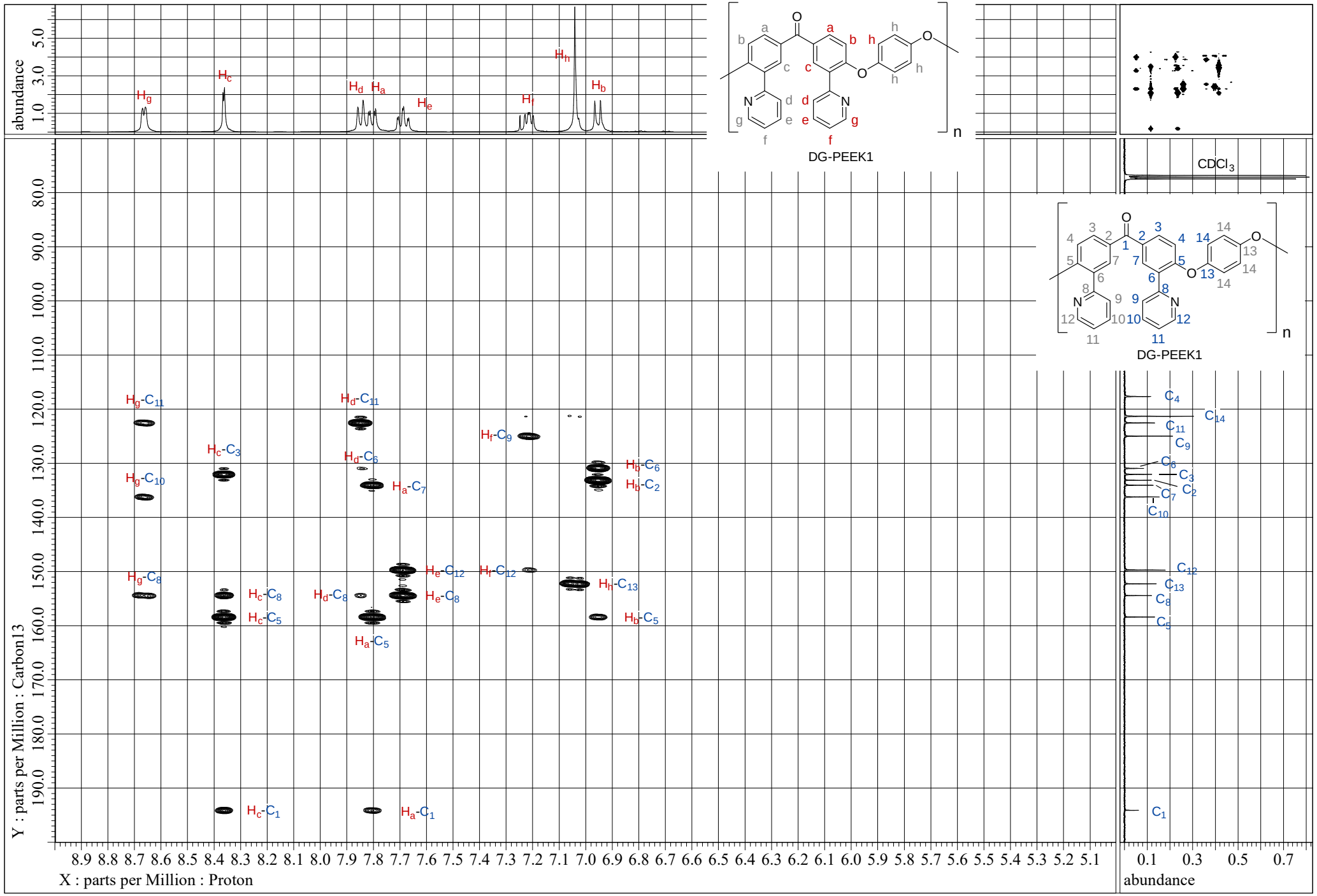
^{1}H-^{1}H COSY ($CDCl_3$)



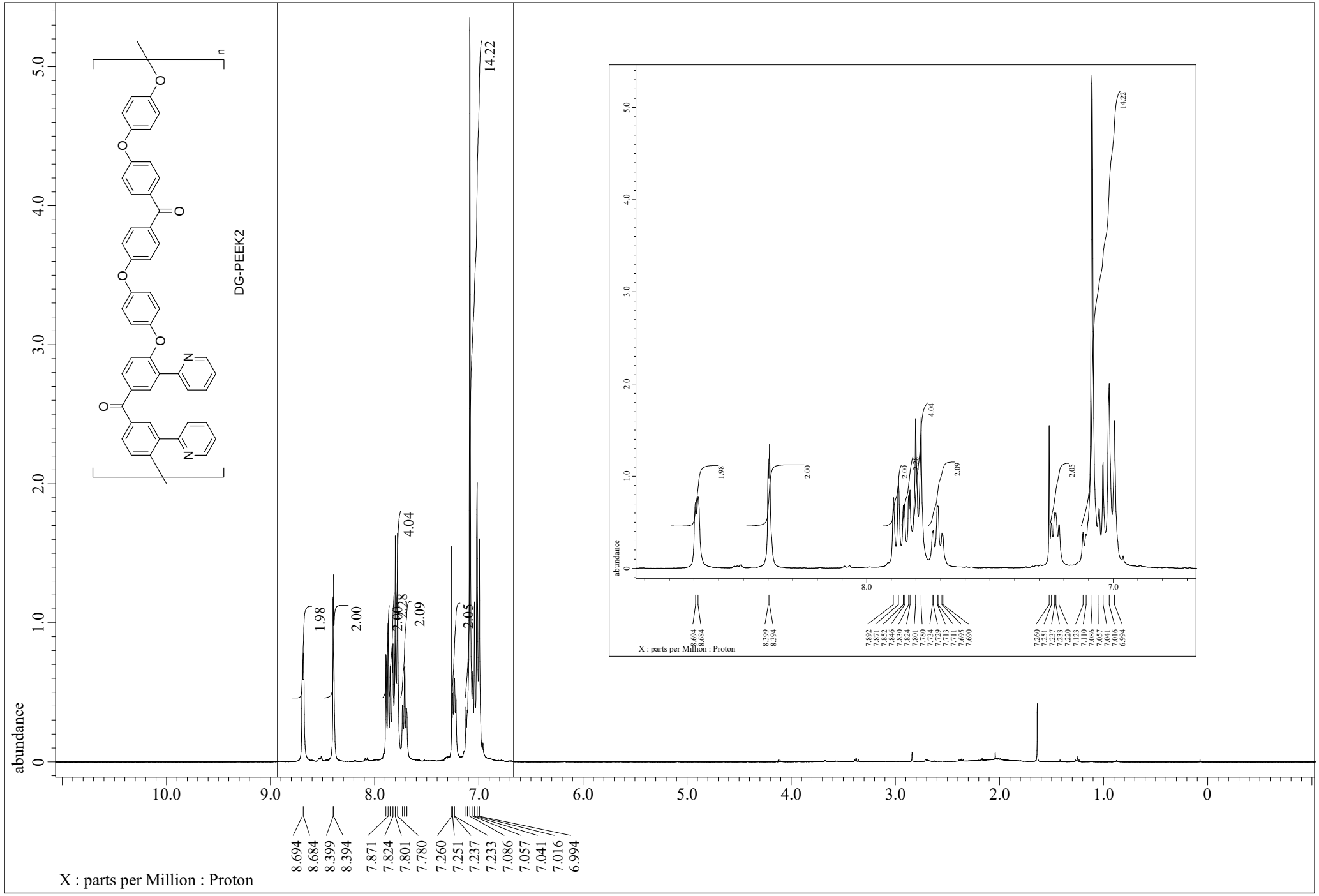
HSQC ($CDCl_3$)


abundance
1.0
3.0
5.0
H_g
H_c
H_d H_a
H_e
H_f
H_h
H_b
DG-PEEK1
n
100.0
110.0
120.0
130.0
140.0
150.0
H_b-C_4
H_f-C_{11}
H_h-C_{14}
H_d-C_9
H_a-C_3
H_c-C_7
H_e-C_{10}
H_g-C_{12}
C_4
C_{14}
C_{11}
C_9
C_3
C_7
C_{10}
C_{12}
DG-PEEK1
n
Y : parts per Million : Carbon13
8.9 8.8 8.7 8.6 8.5 8.4 8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5 6.4 6.3 6.2 6.1
X : parts per Million : Proton
0 0.2 0.4 0.6
abundance

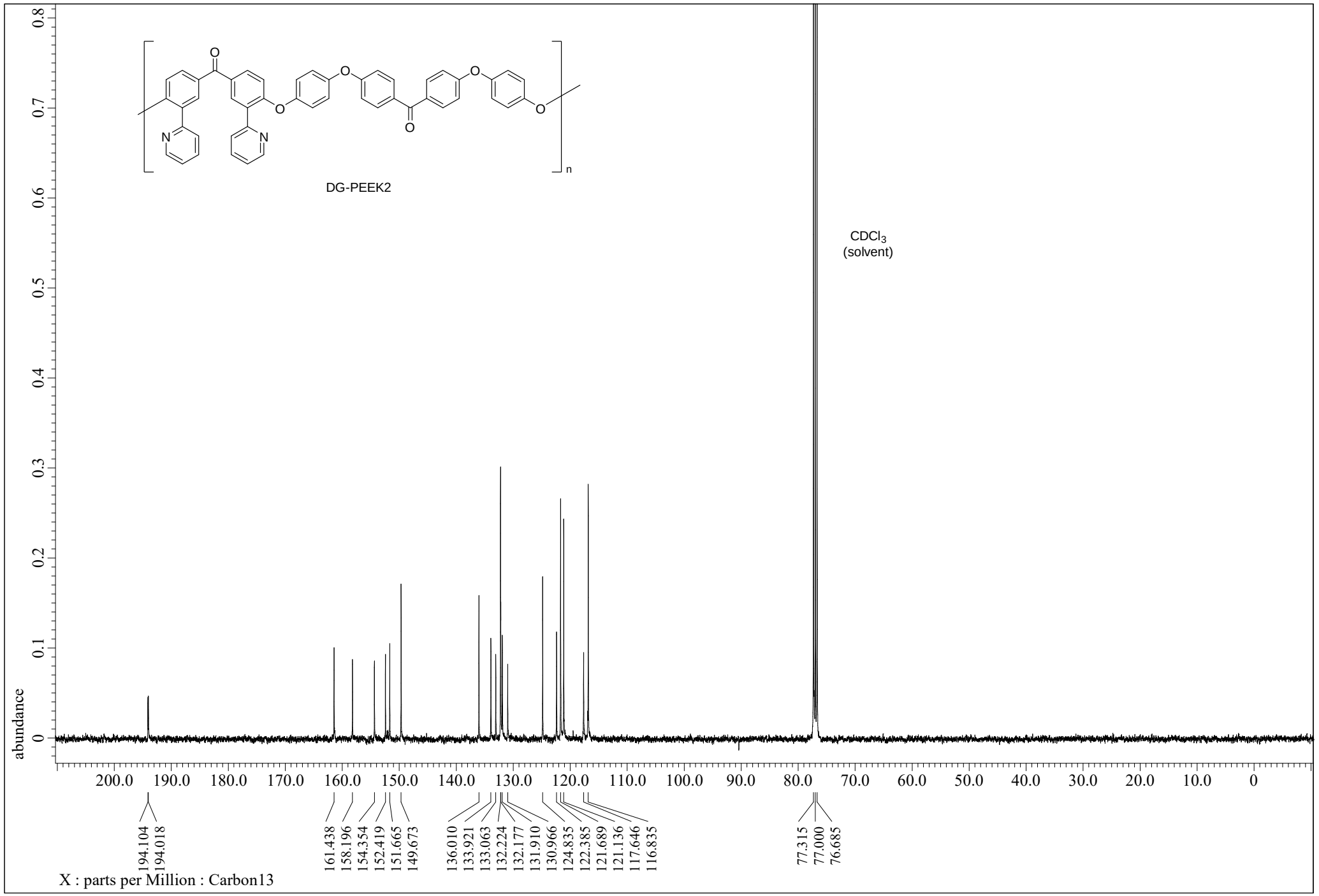
HMBC ($CDCl_3$)


abundance
1.0 3.0 5.0
H_g H_c H_d H_a H_e H_f H_h H_b
DG-PEEK1
$CDCl_3$
H_g-C_{11} H_d-C_{11} H_f-C_9 H_c-C_3 H_d-C_6 H_b-C_6 H_b-C_2 H_g-C_{10} H_a-C_7 H_g-C_8 H_e-C_{12} H_f-C_{12} H_h-C_{13} H_c-C_8 H_d-C_8 H_e-C_8 H_c-C_5 H_b-C_5 H_a-C_5 H_c-C_1 H_a-C_1
C_4 C_{14} C_{11} C_9 C_6 C_3 C_2 C_7 C_{10} C_{12} C_{13} C_8 C_5 C_1
Y : parts per Million : Carbon13
X : parts per Million : Proton
abundance

^{1}H NMR (396 MHz, $CDCl_3$)



^{13}C NMR (101 MHz, $CDCl_3$)



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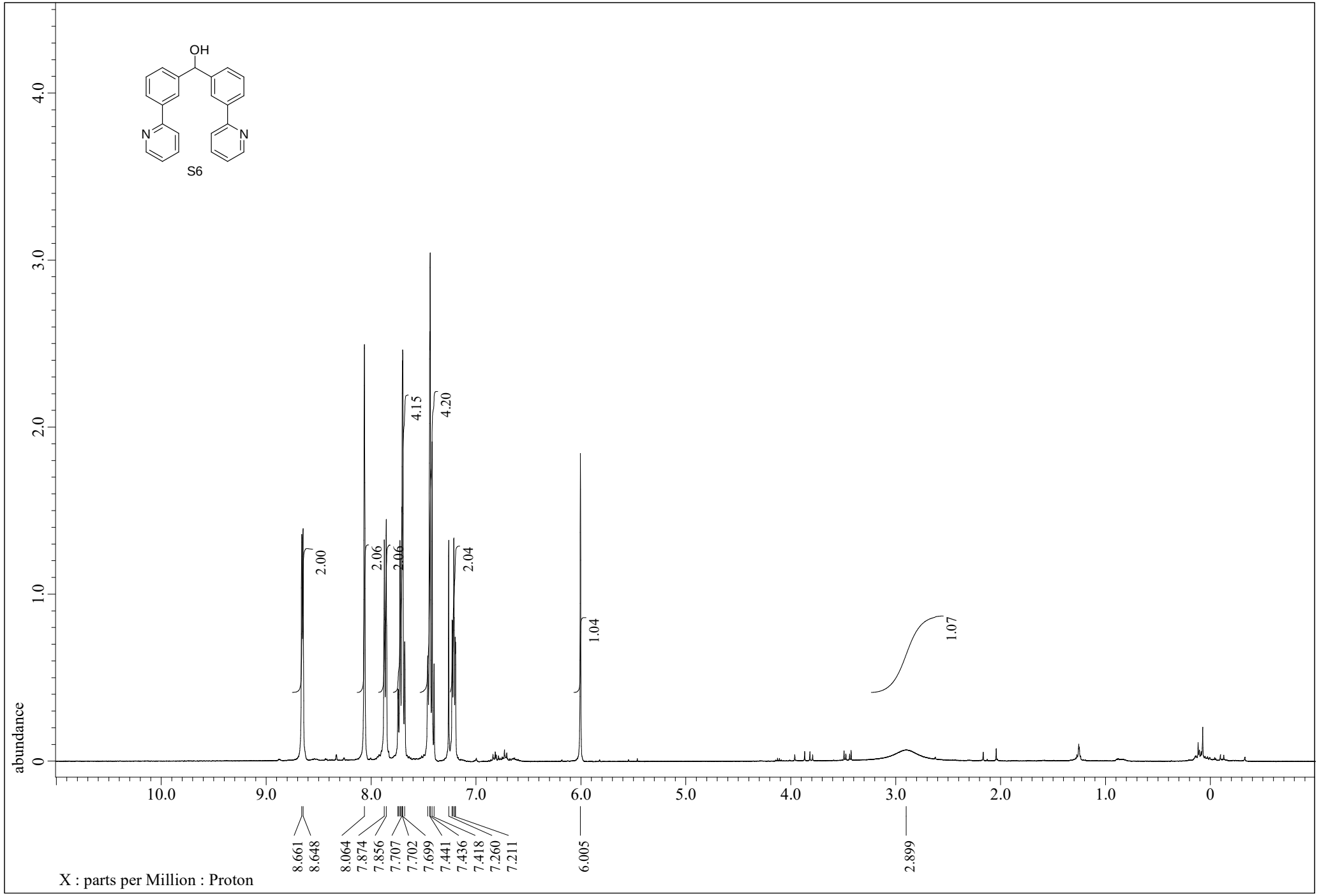
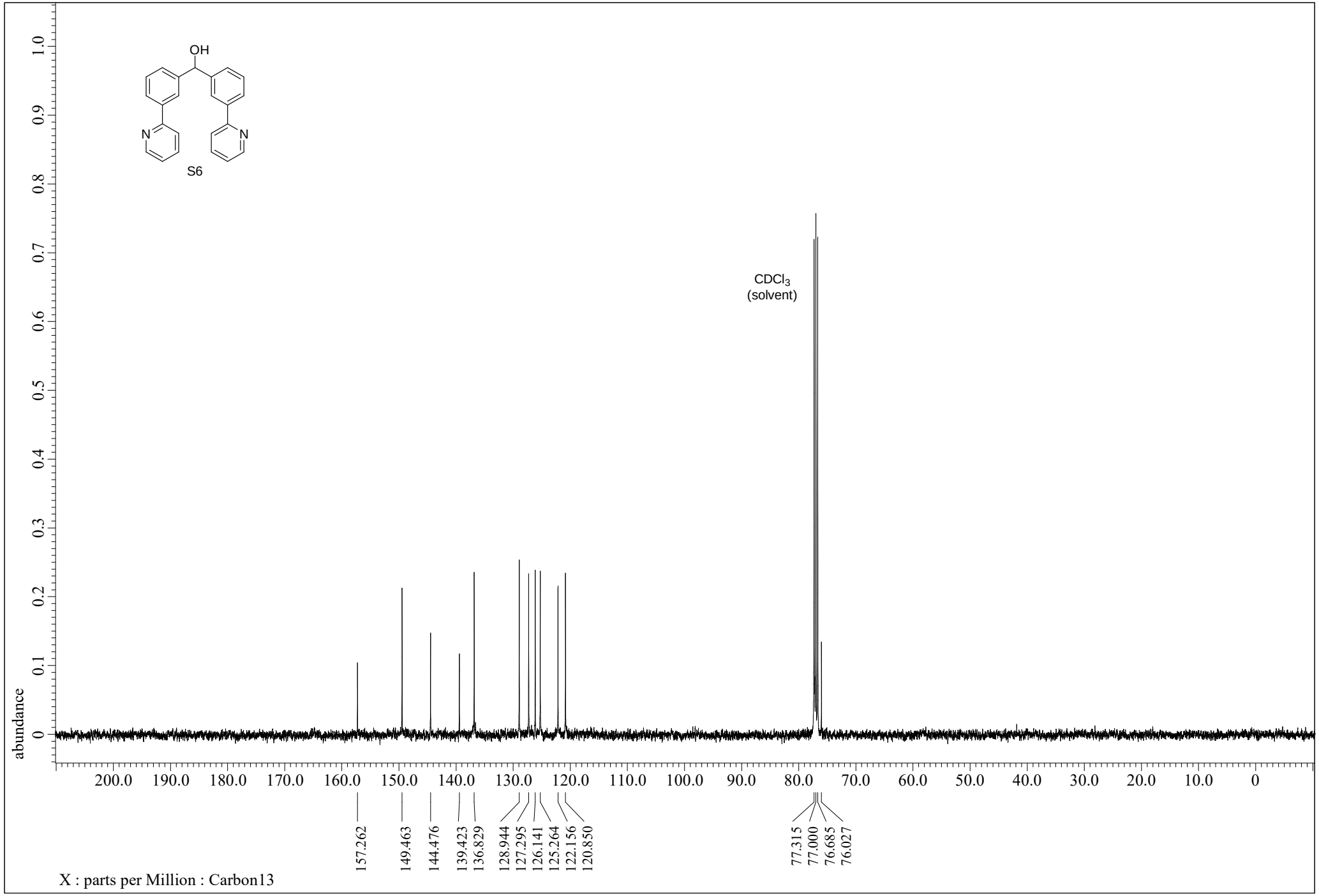
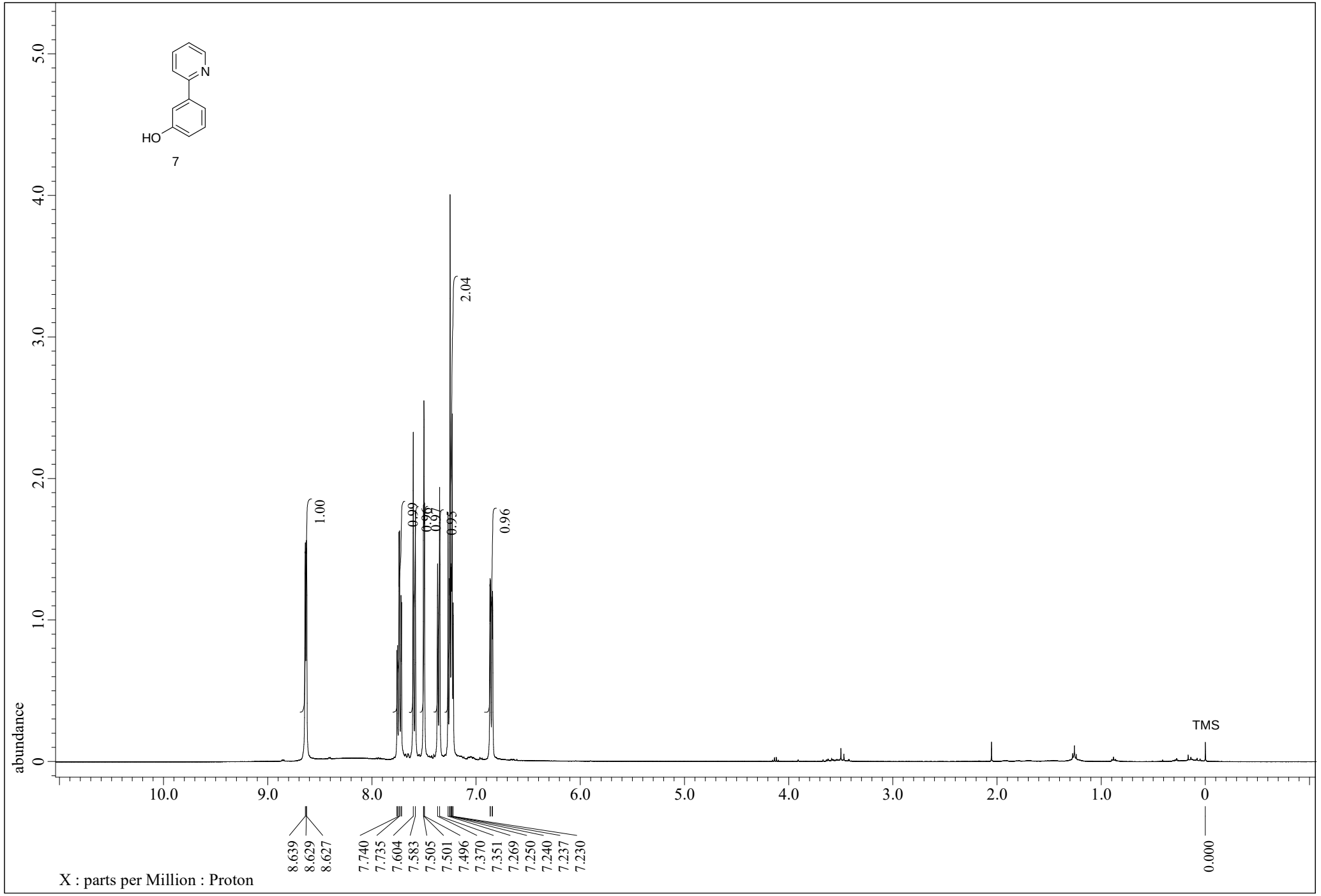


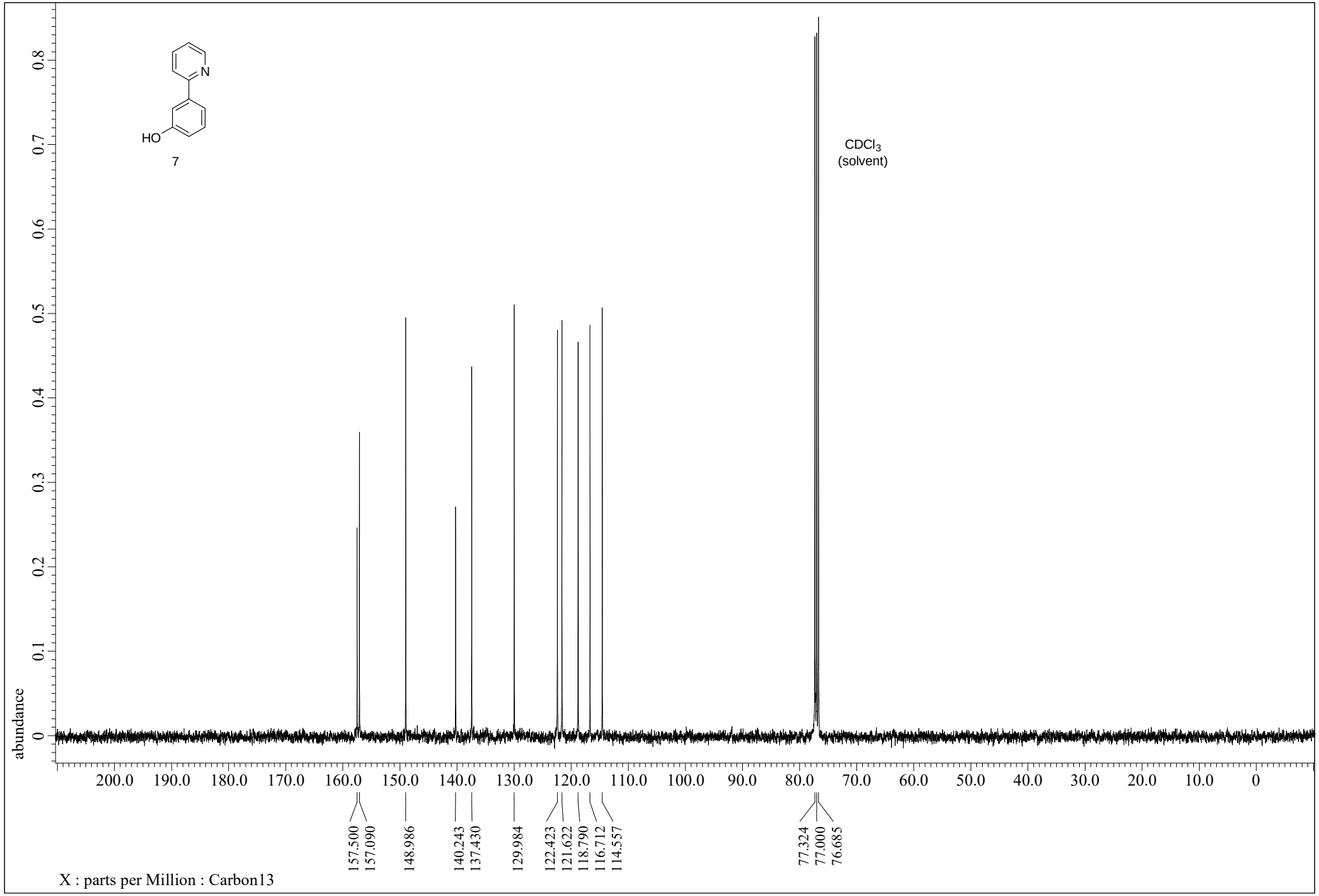
^{13}C NMR (101 MHz, $CDCl_3$)



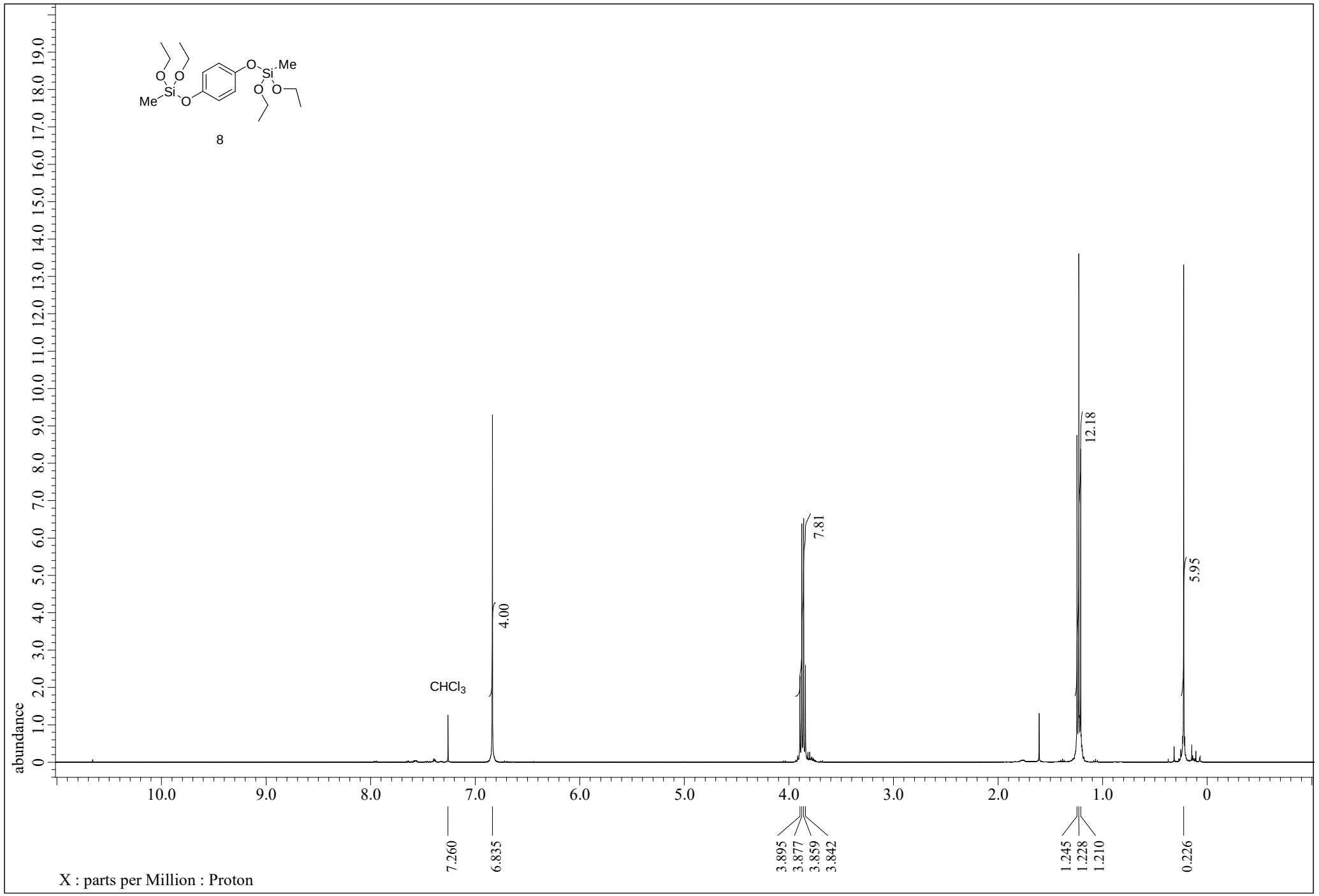
^{1}H NMR (400 MHz, $CDCl_3$)



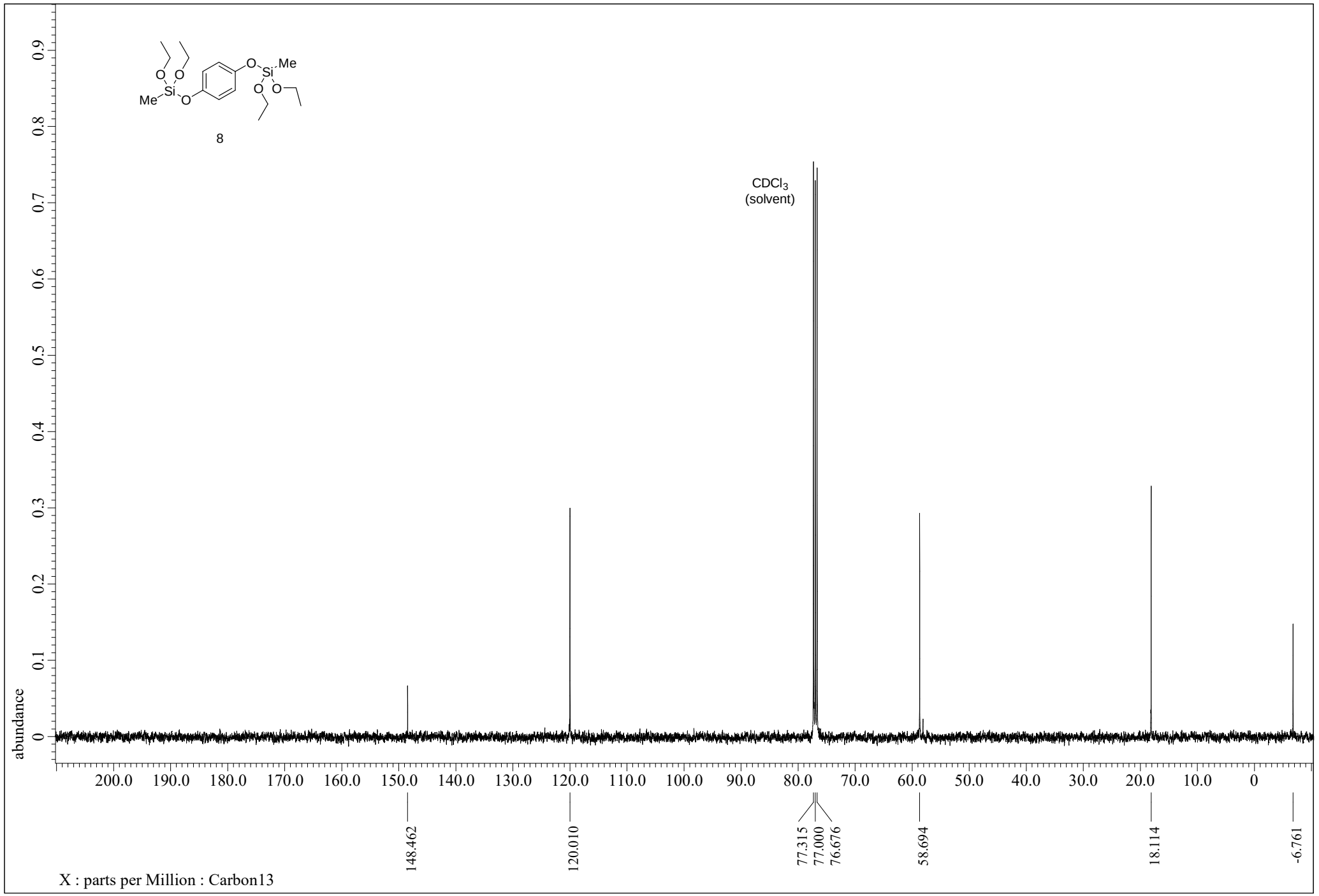
^{13}C NMR (101 MHz, $CDCl_3$)



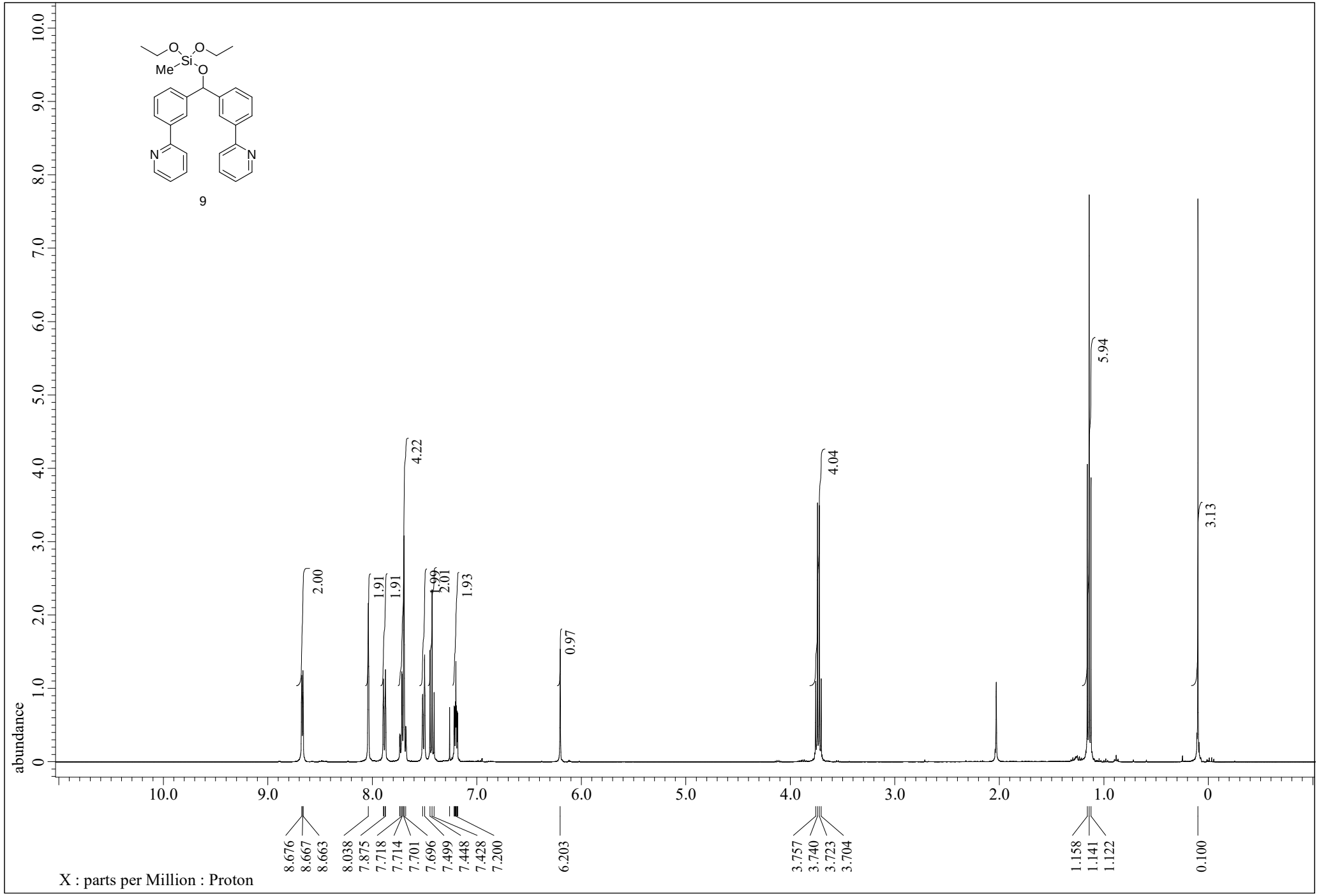
^{1}H NMR (400 MHz, $CDCl_3$)



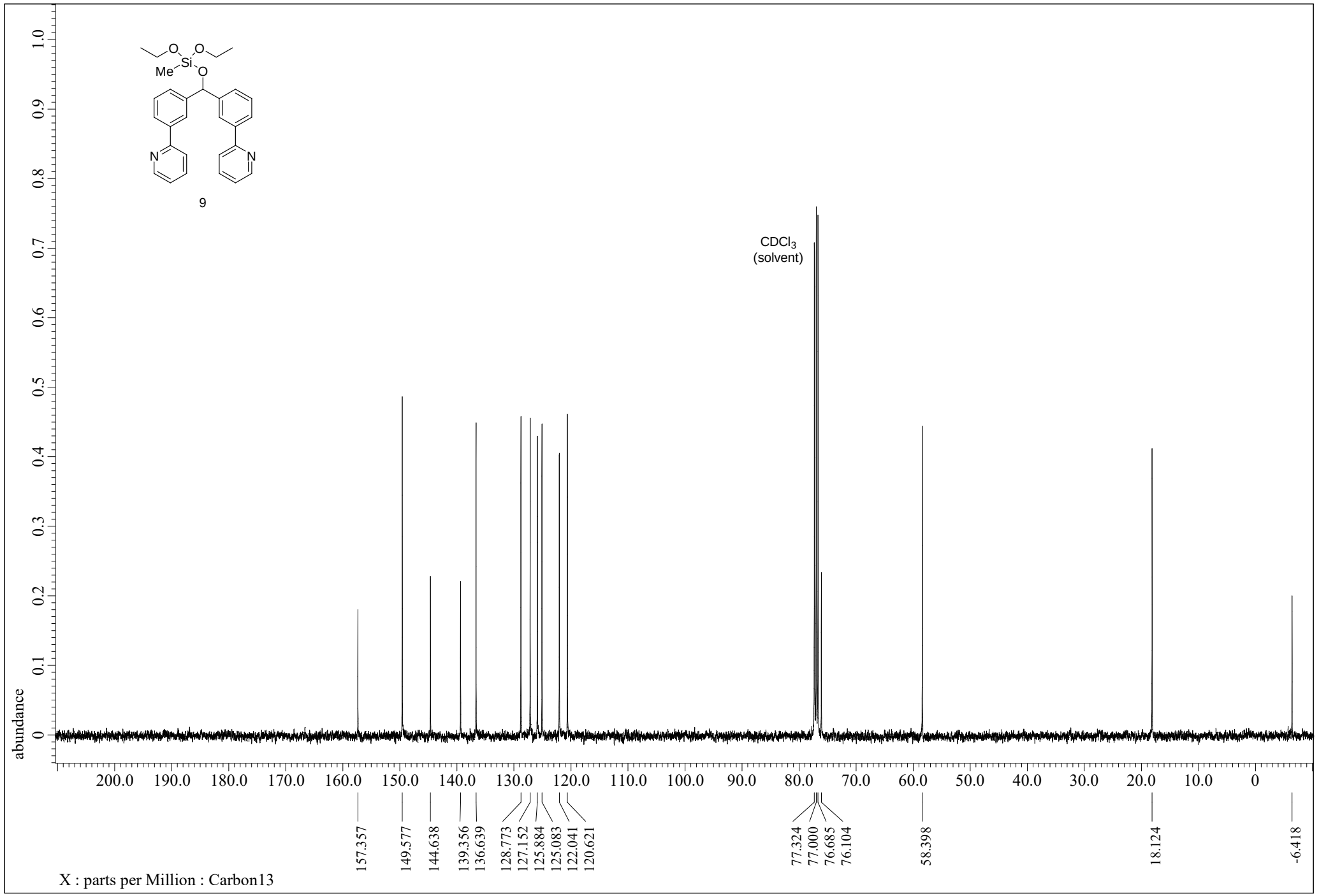
^{13}C NMR (101 MHz, $CDCl_3$)



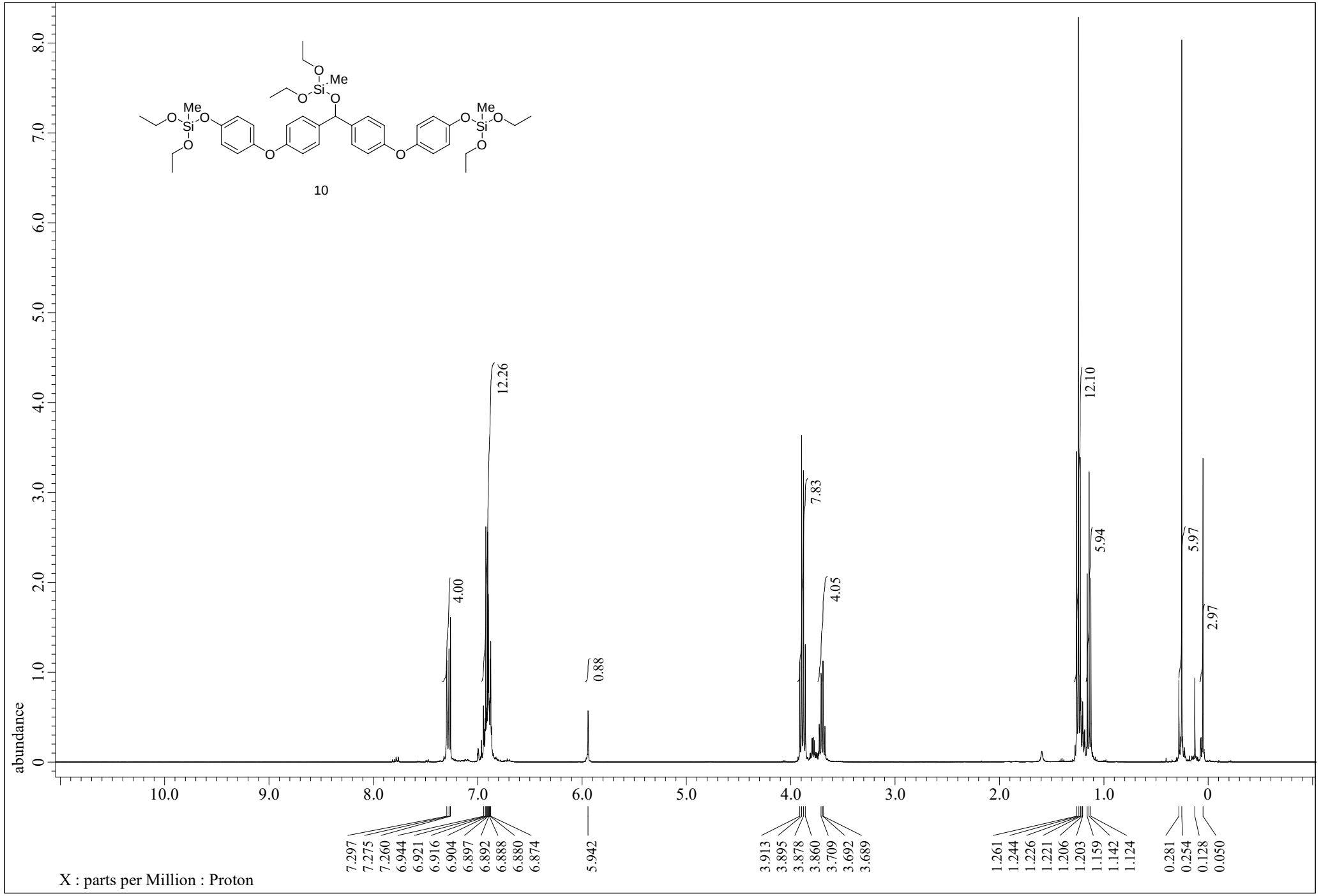
^{1}H NMR (400 MHz, $CDCl_3$)



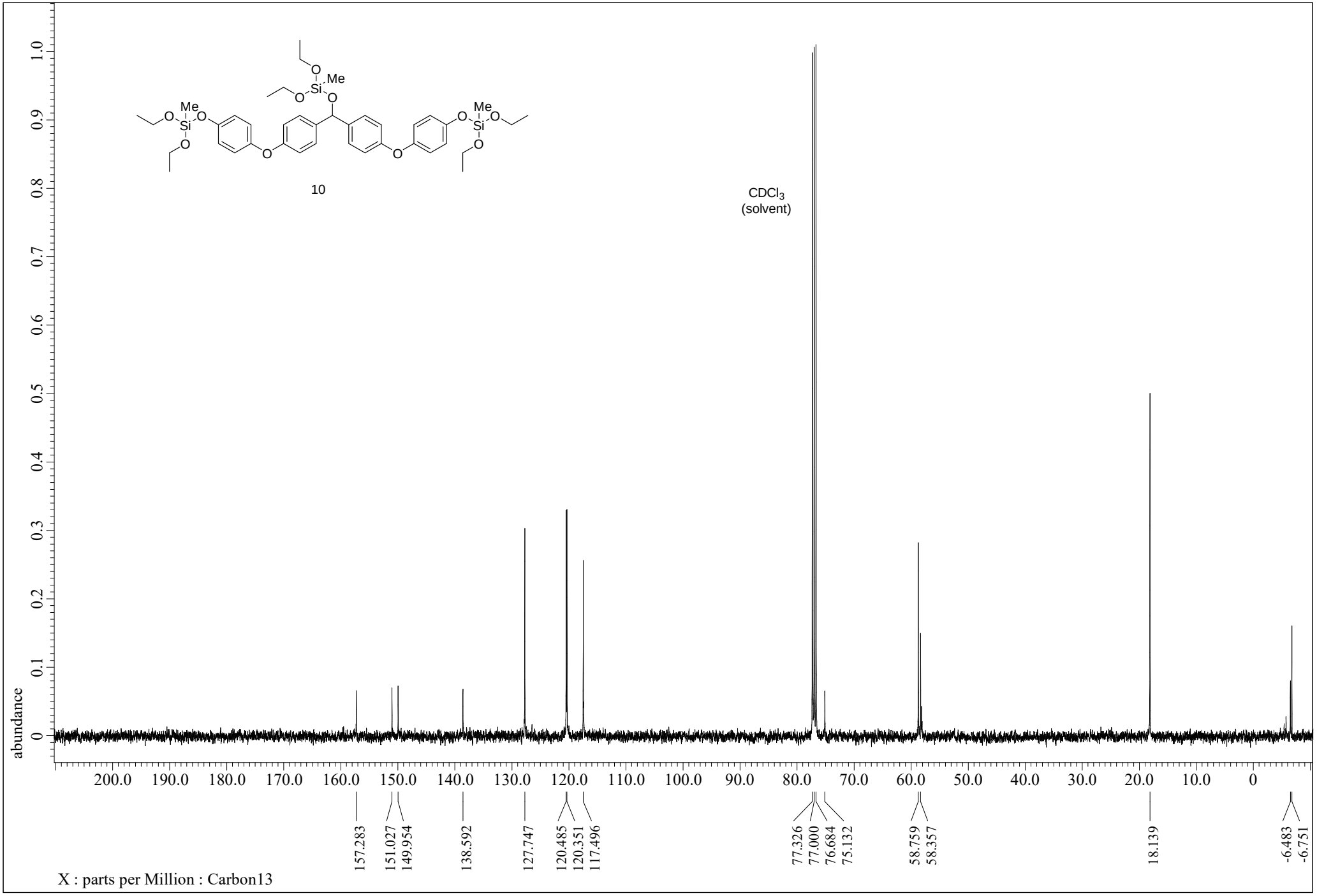
^{13}C NMR (101 MHz, $CDCl_3$)



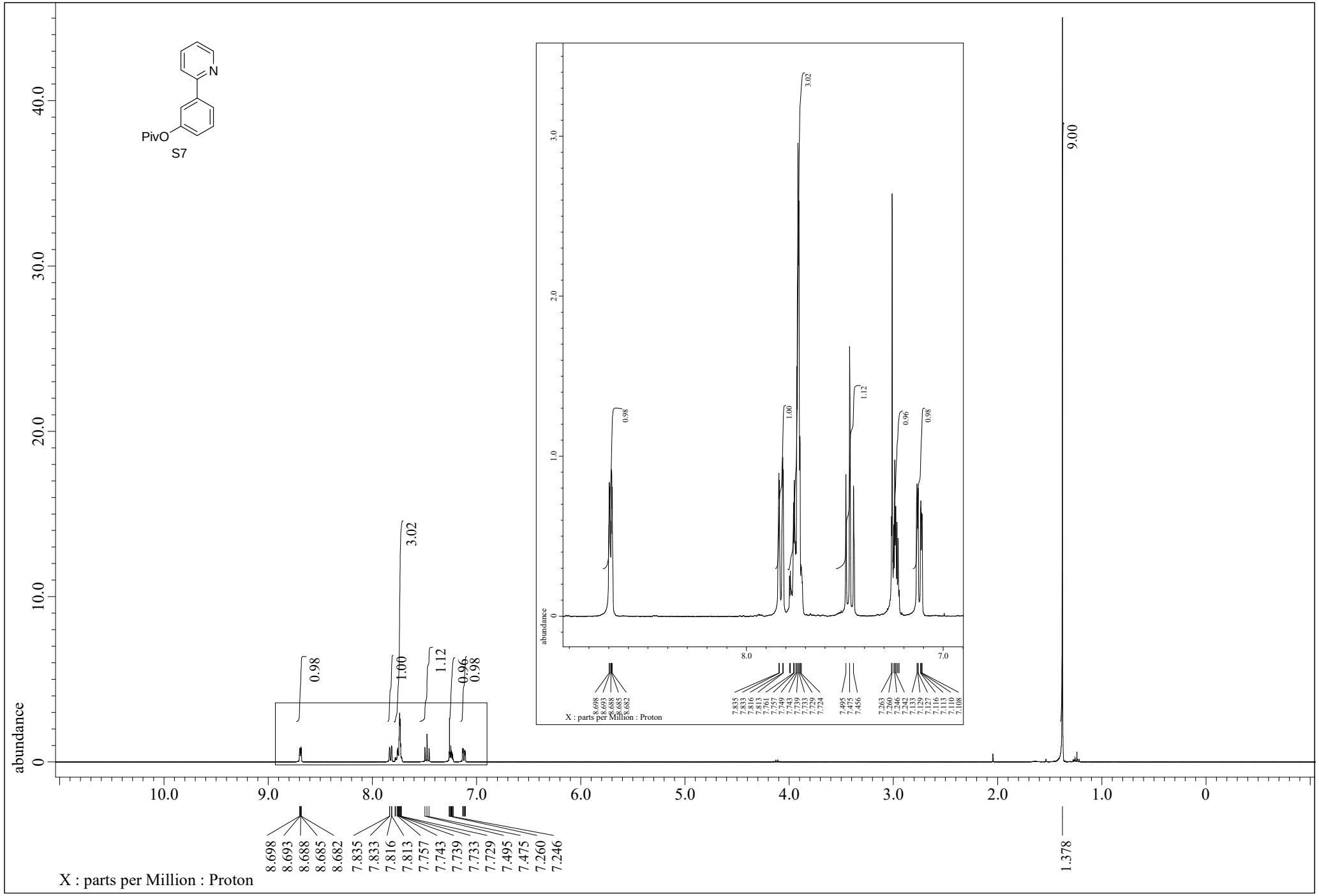
^{1}H NMR (400 MHz, $CDCl_3$)



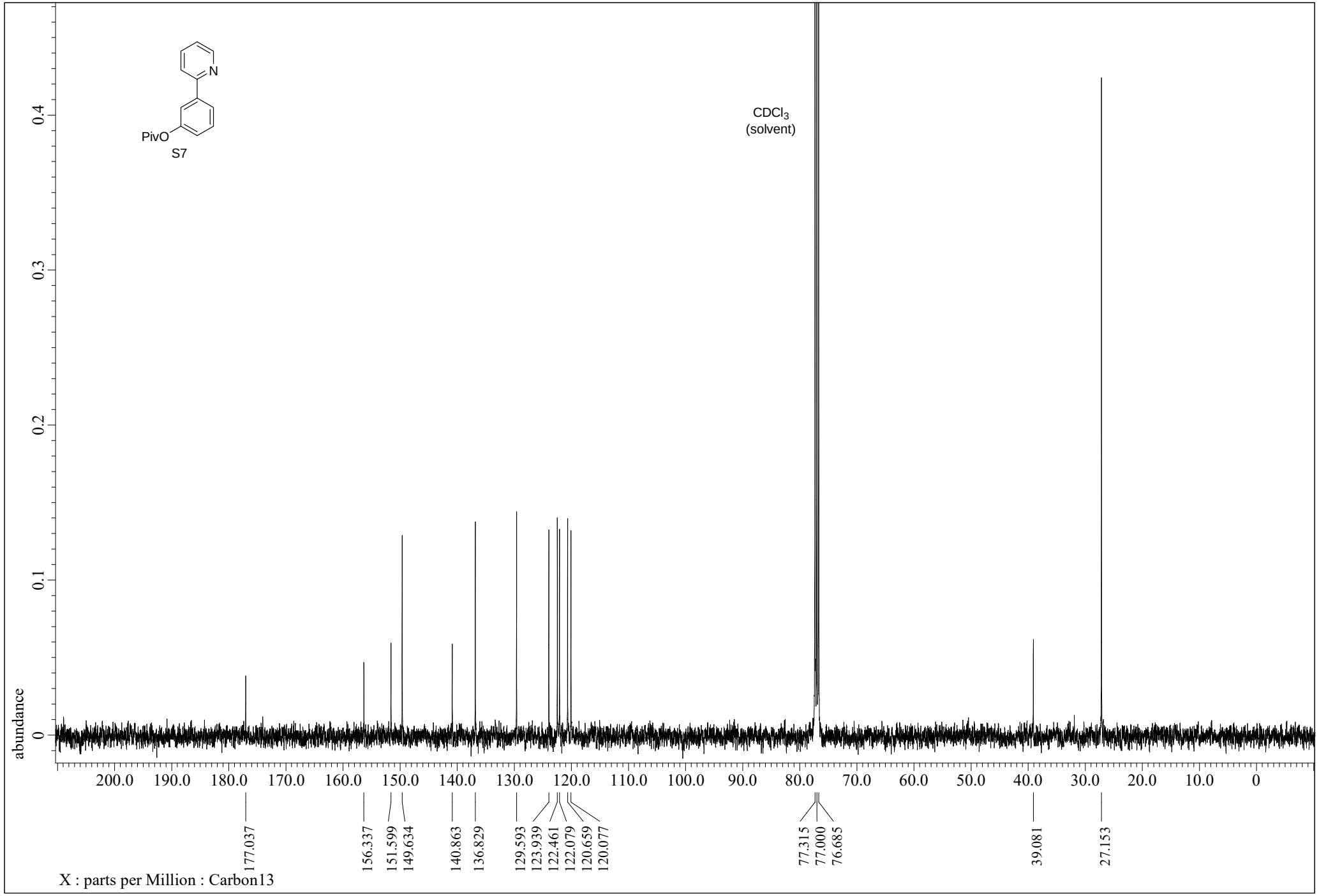
^{13}C NMR (101 MHz, $CDCl_3$)



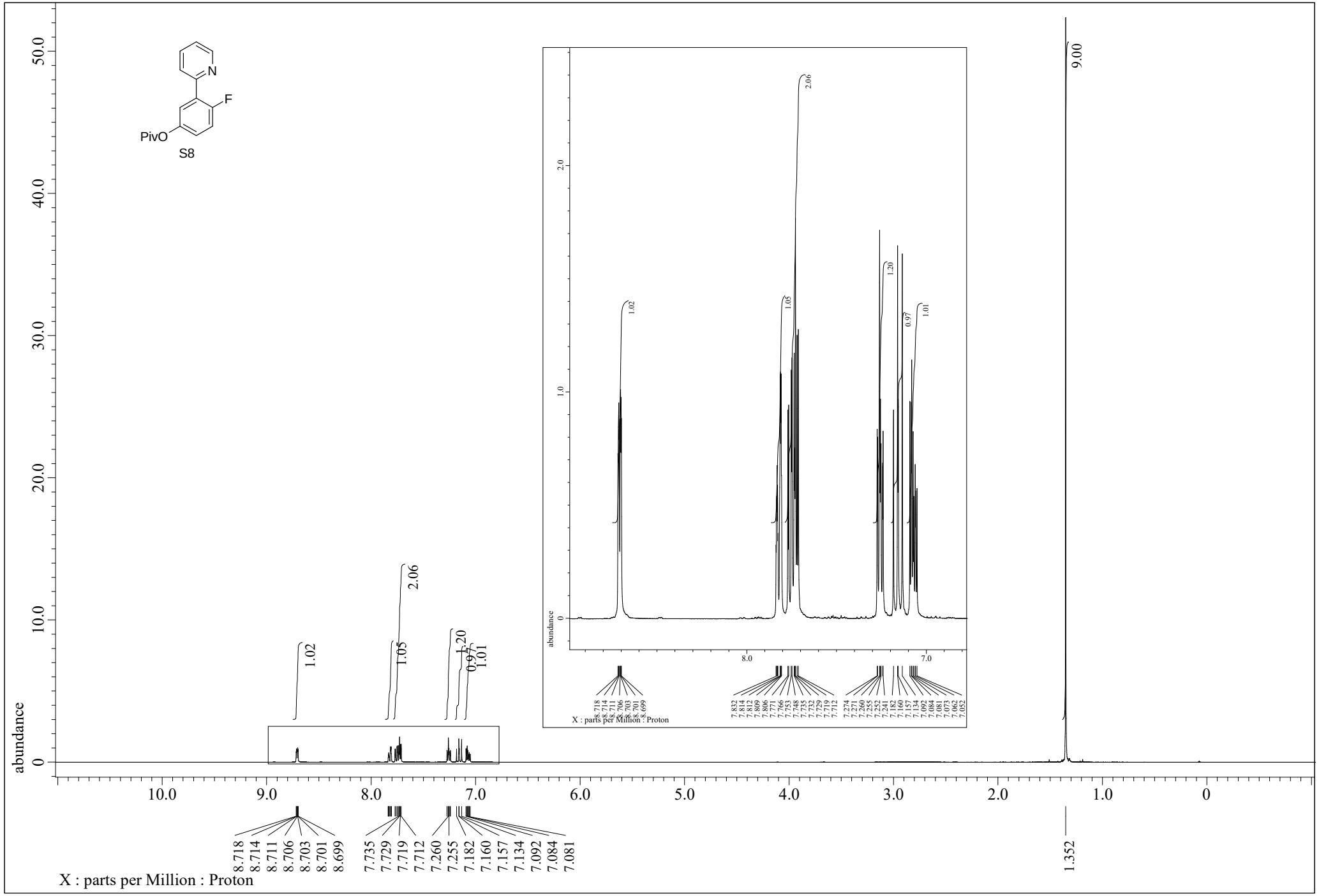
^{1}H NMR (400 MHz, $CDCl_3$)



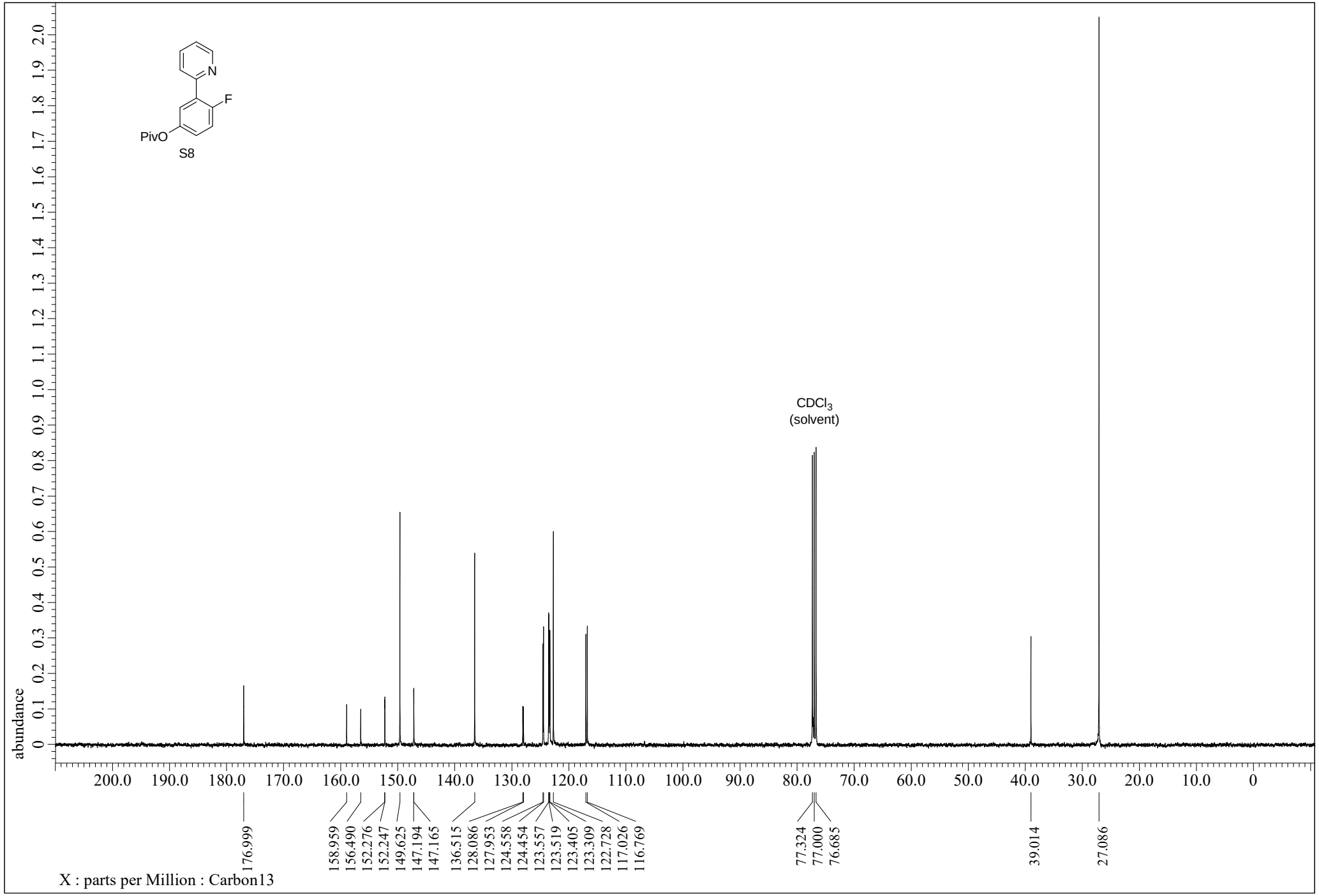
^{13}C NMR (101 MHz, $CDCl_3$)



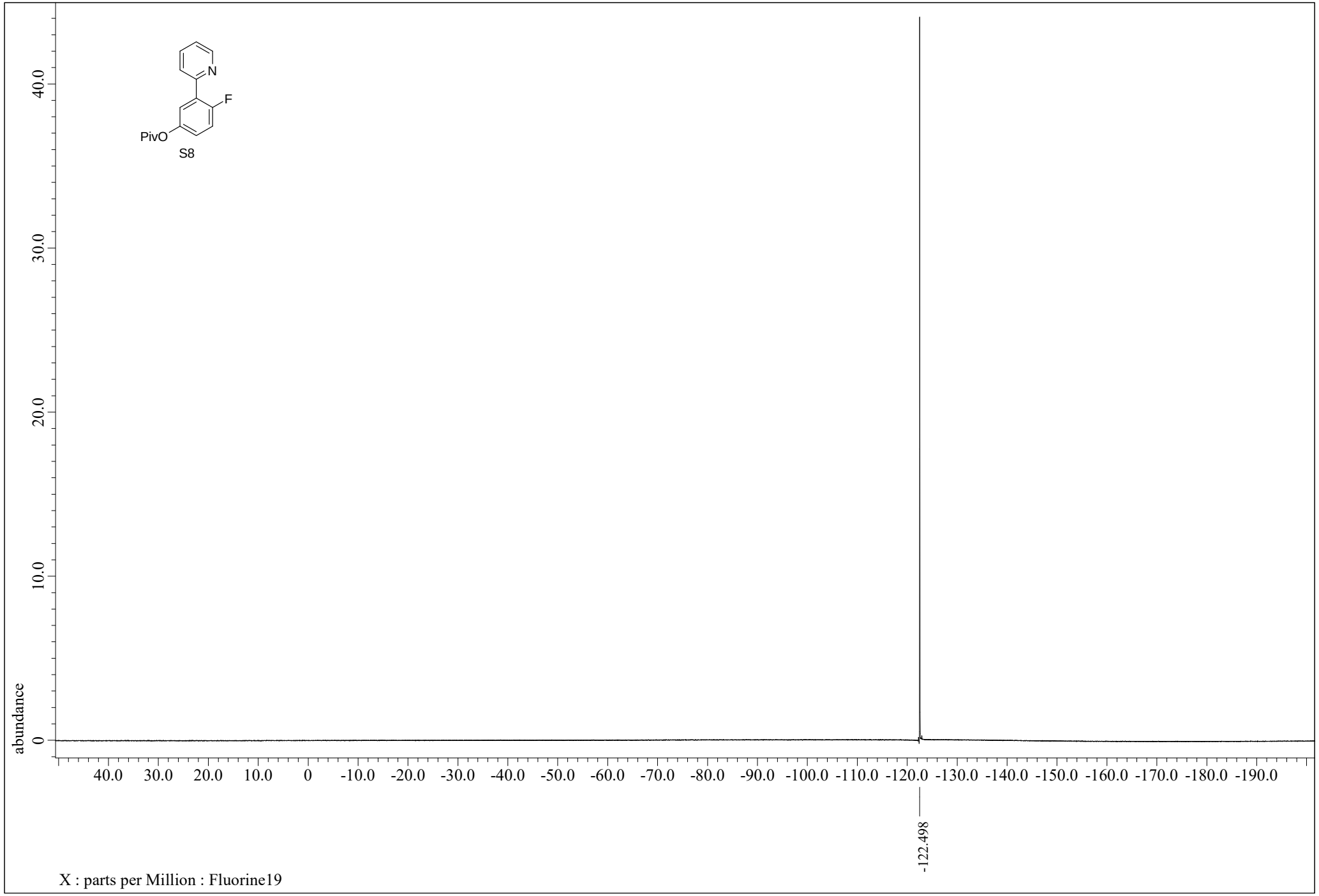
^{1}H NMR (400 MHz, $CDCl_3$)



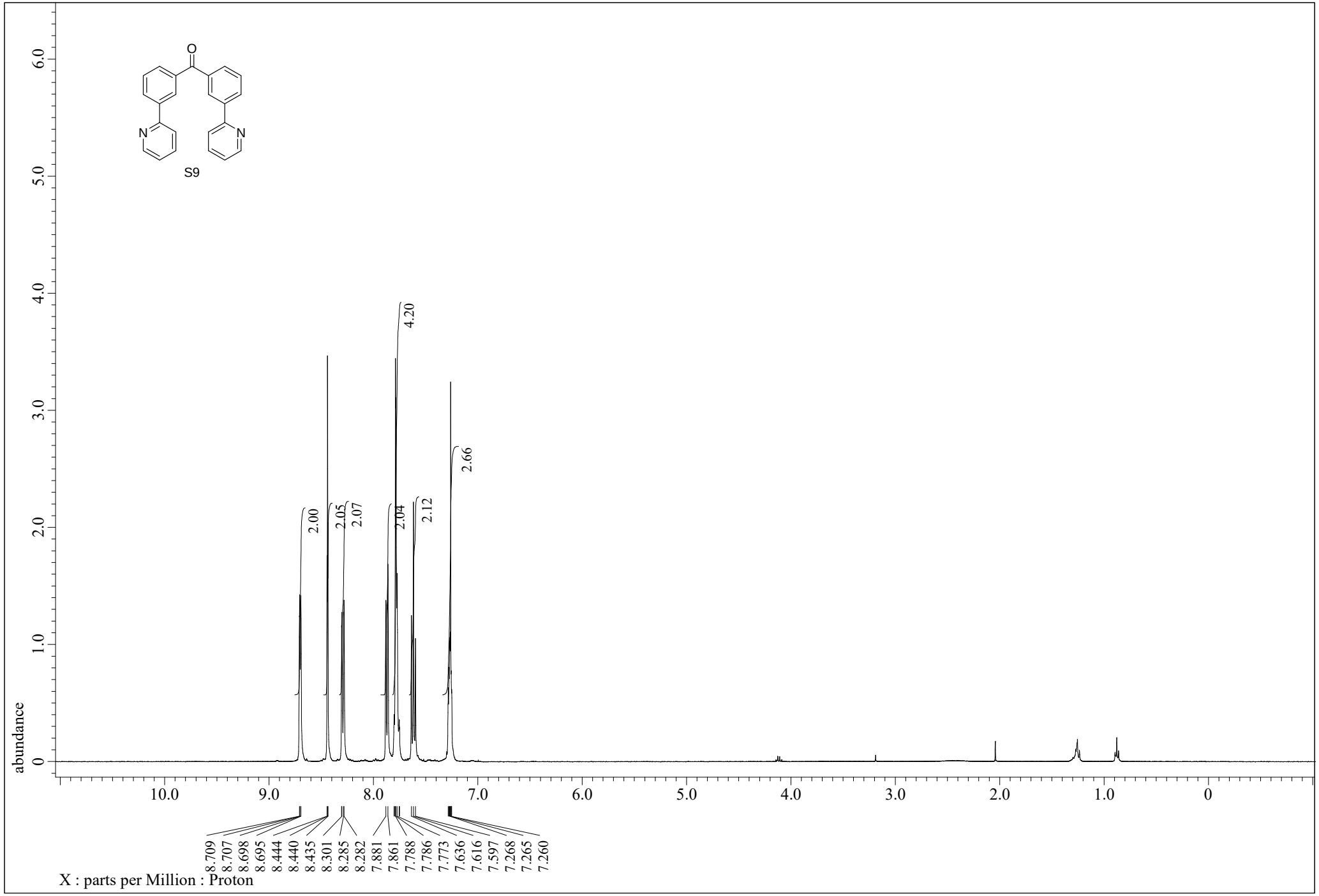
^{13}C NMR (101 MHz, $CDCl_3$)



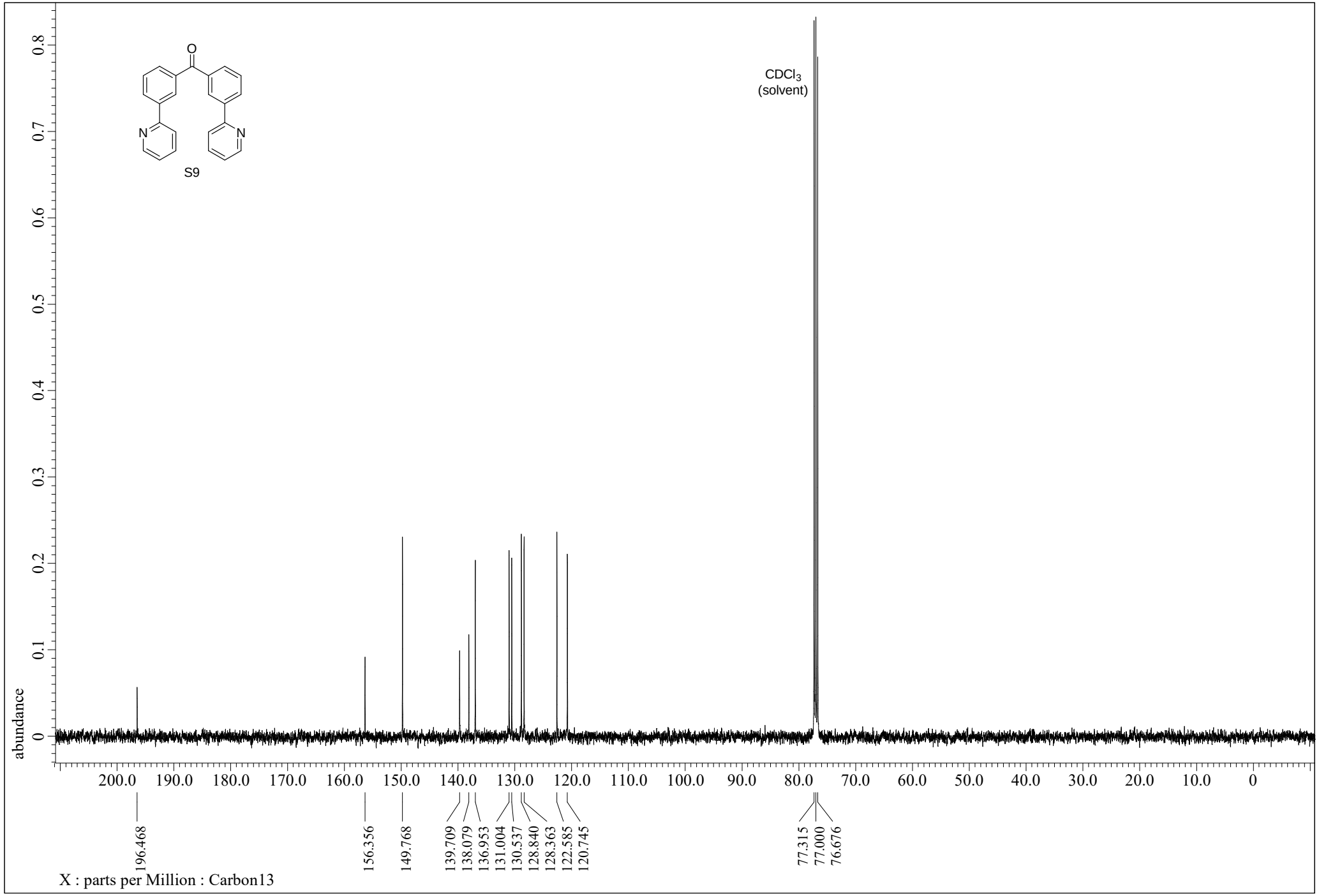
^{19}F NMR (376 MHz, $CDCl_3$)



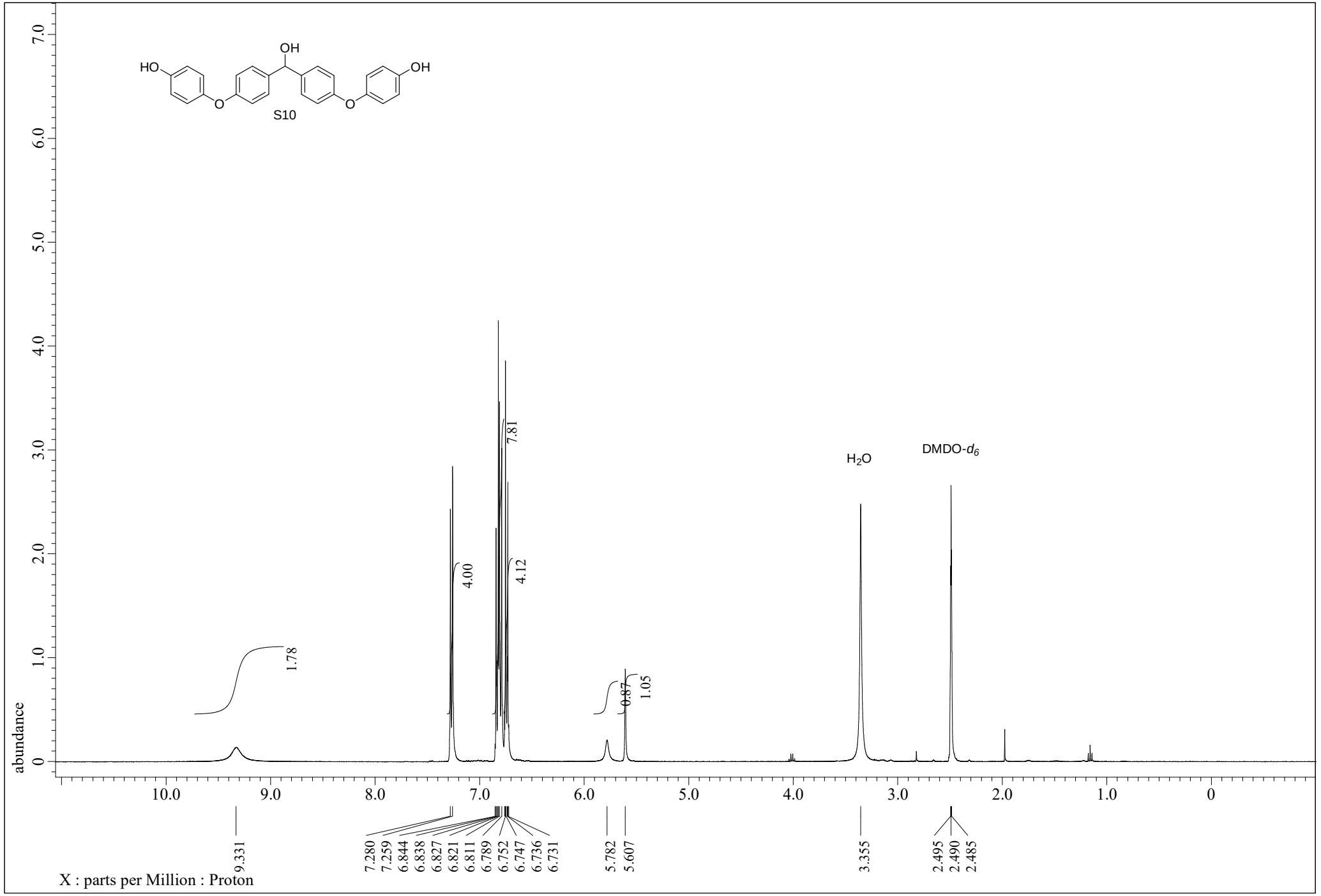
^{1}H NMR (400 MHz, $CDCl_3$)



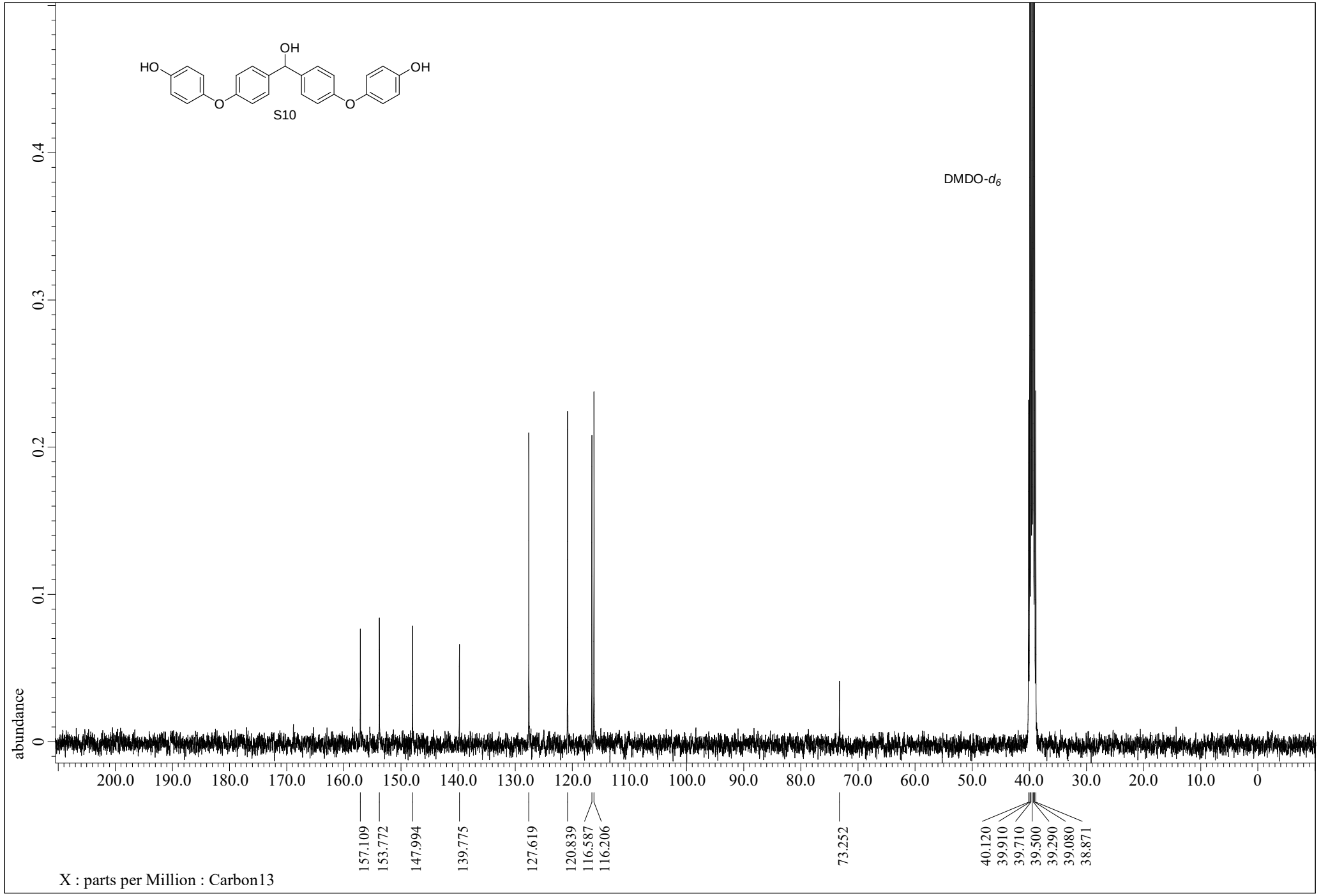
^{13}C NMR (101 MHz, $CDCl_3$)



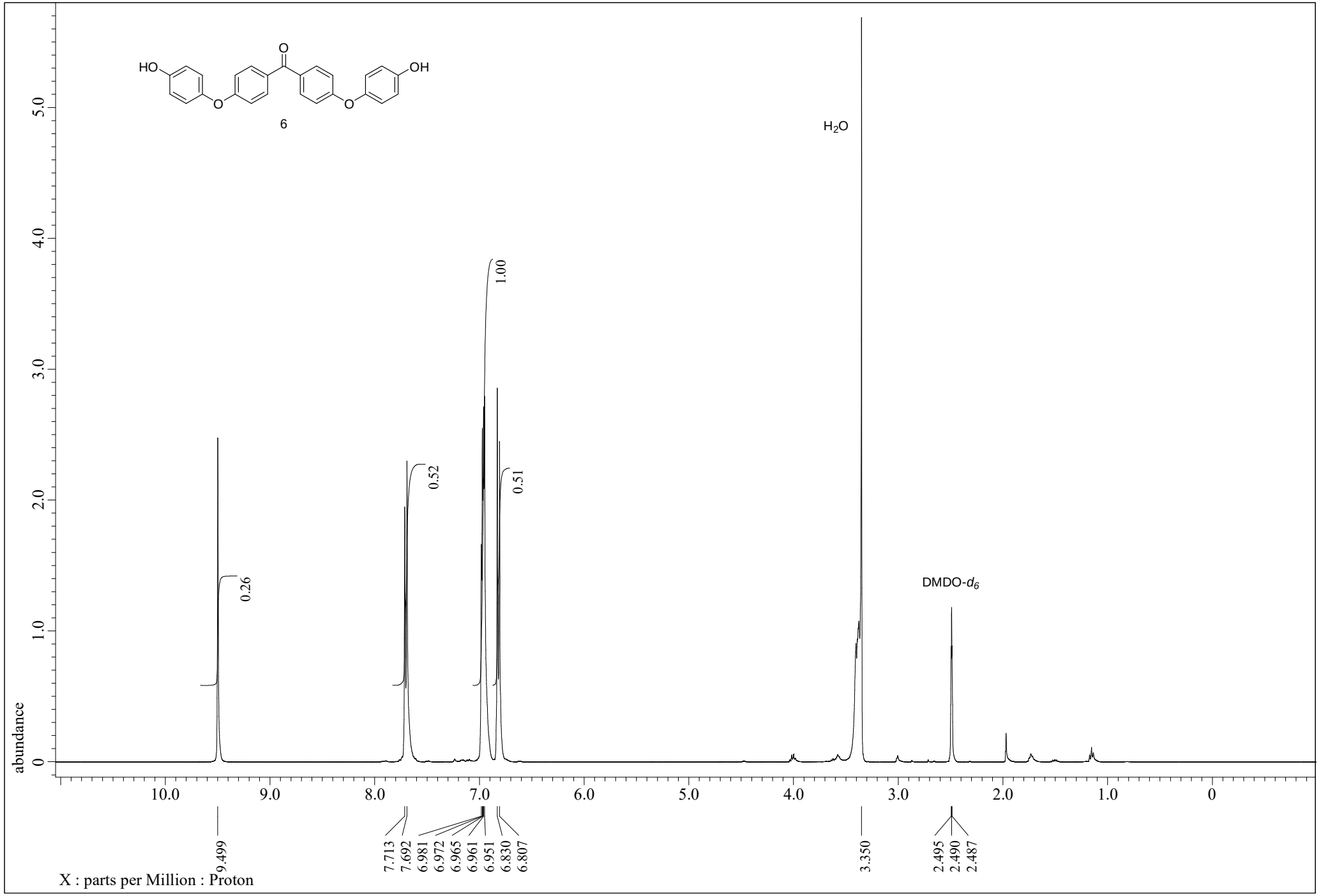
^{1}H NMR (400 MHz, DMSO-d_6)



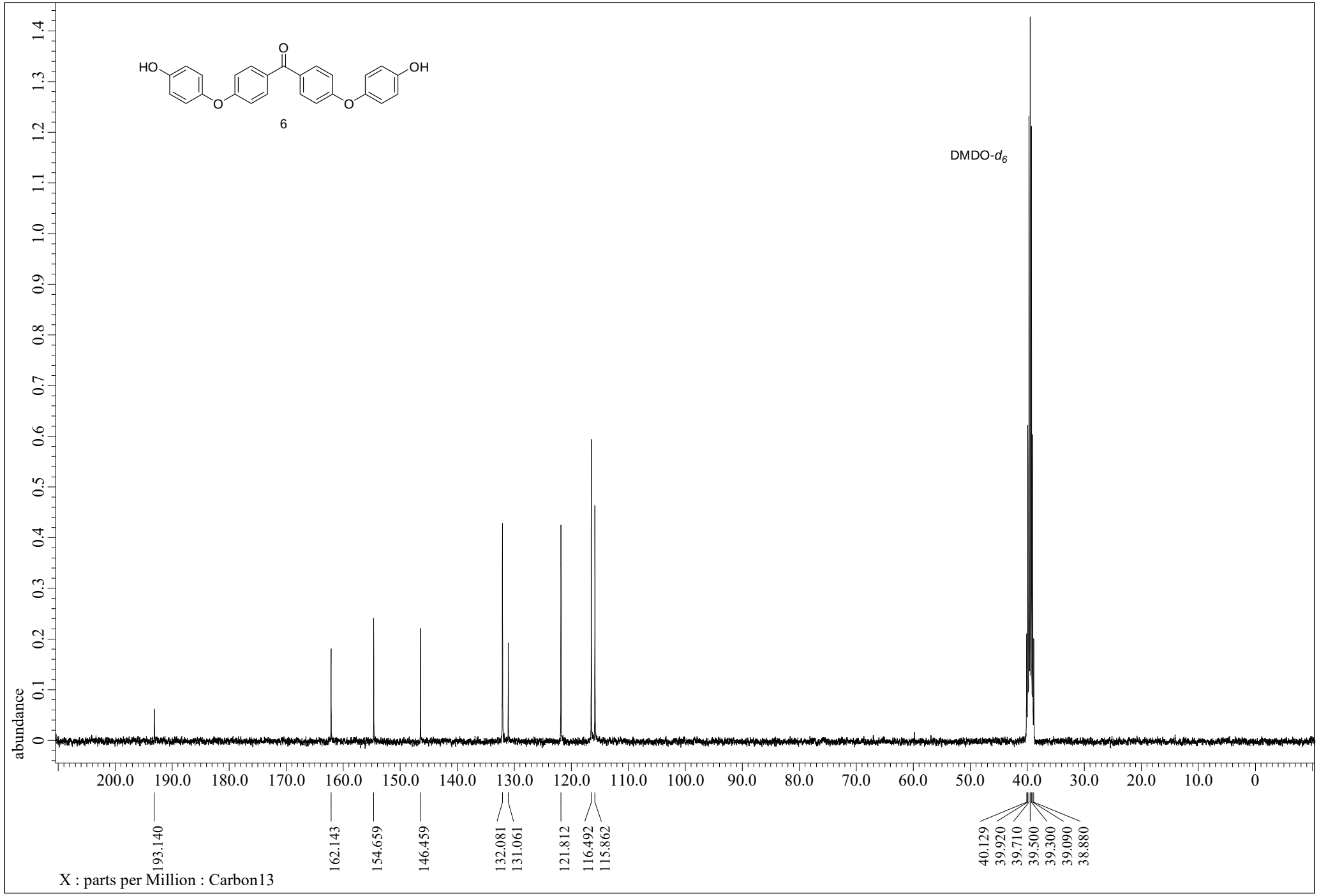
^{13}C NMR (101 MHz, DMSO-d_6)



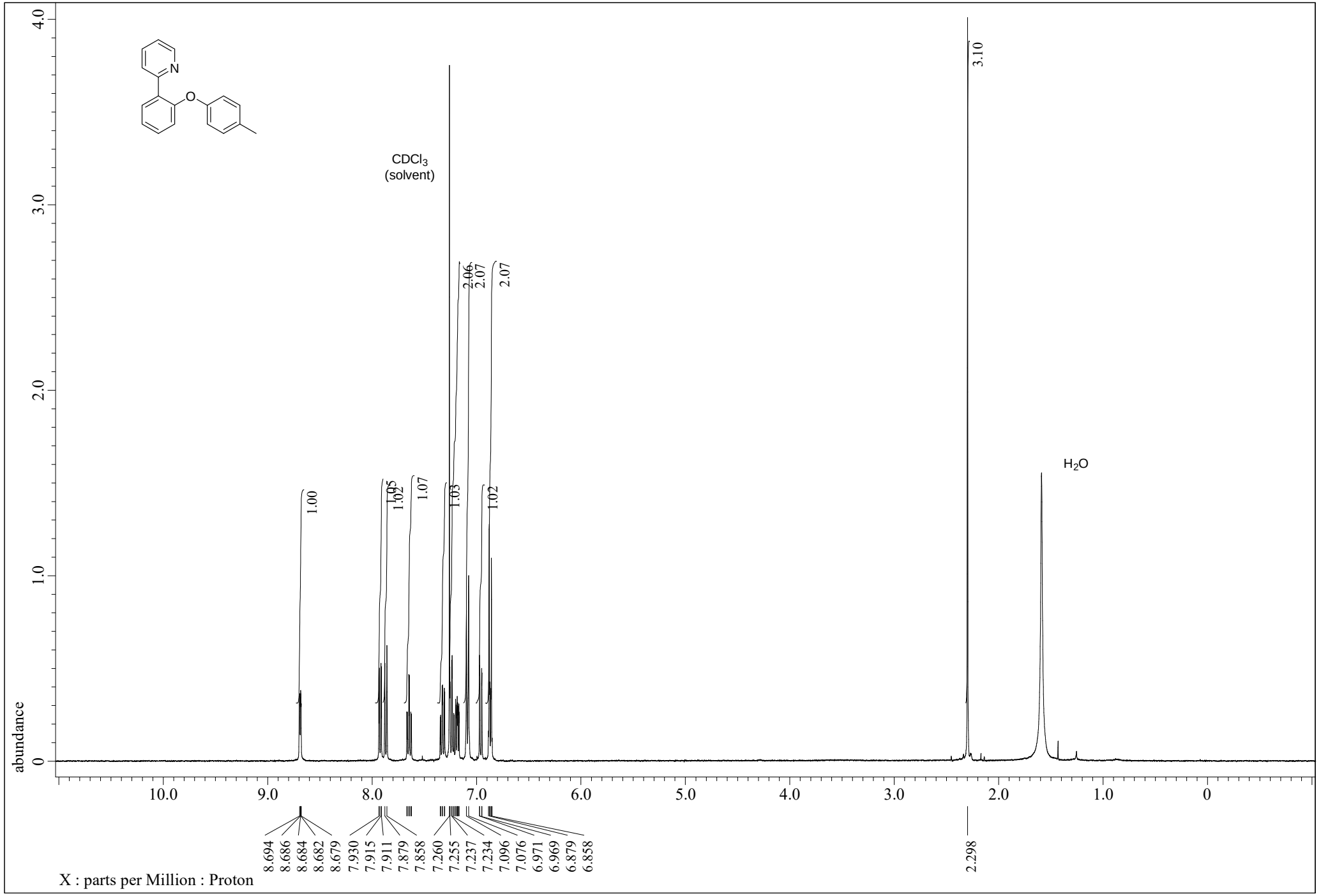
^{1}H NMR (400 MHz, DMSO-d_6)



^{13}C NMR (101 MHz, DMSO-d_6)



^{1}H NMR (400 MHz, $CDCl_3$)



^{13}C NMR (101 MHz, $CDCl_3$)