# Palladium-Catalyzed Electrochemical C($sp^3$)–H Acetoxylation of Alcohol Derivatives with an *exo*-Directing Group

Daisuke Ogawa,[1] Ayumu Sasaki,[1] Takuya Kochi[1] and Fumitoshi Kakiuchi[1,2,*]

[1] *Department of Chemistry, Faculty of Science and Technology, Keio University, 3-14-1 Hiyoshi, Kohoku-ku, Yokohama, Kanagawa 223-8522, Japan.*

[2] *JST, CREST, JPMJCR20R1, 4-1-8 Honcho, Kawaguchi, Saitama, 332-0012, Japan.*

kakiuchi@chem.keio.ac.jp

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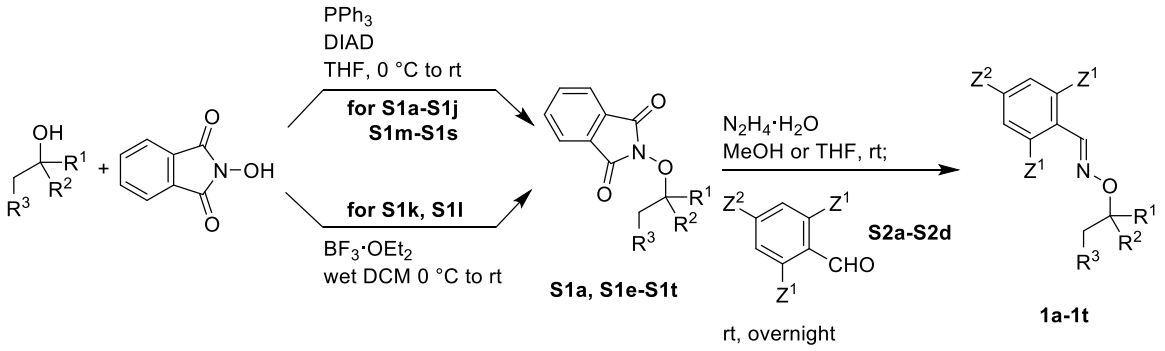
## I. General Information.

$^{1}H$, $^{13}C\{^{1}H\}$, and $^{19}F$ NMR spectra were recorded on a JEOL ECS-400, AL-400, or ECX-400 spectrometer. IR spectra were recorded on a JASCO FT/IR-410 infrared spectrometer. ESI-MS analyses were performed on a JEOL JMS-T100LCS instrument. Flash chromatography was carried out with silica gel 60N (Kanto Chemical Co., Inc.). Melting points were determined on a Stanford Research Systems MPA 100 instrument. Gel permeation chromatography (GPC) was carried out with a JAI LaboACE LC-5060 instrument.

## II. Solvents and Materials

Alcohols (except for 8-nonen-2-ol), AcOH, $Ac_2O$, MeOH, THF, boron trifluoride ethyl ether complex, 7-bromo-1-heptene, diisopropyl azodicarboxylate (40% in Toluene, ca. 1.9 mol/L), *N*-hydroxyphthalimide, dichloromethane, hydrazine monohydrate, $Pd(OAc)_2$, NaOAc, $PPh_3$ were purchased from commercial suppliers and used as received. Neosepta CSE was used as a cation-exchange membrane and washed with water and acetone prior to use.

## III. Preparation of Oxime Ether 1



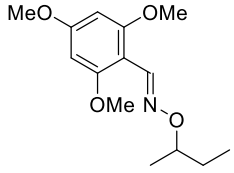

Compounds **S1a**, **S1e-S1l**, **1a**, **1d-1l**, **1s**, and **1t** were prepared according to the literature procedures.[1] Compound **1m** was prepared according to the literature procedures.[2] Compounds **S1n** and **S1o** were prepared according to the literature procedures.[3] Aldehydes **S2a** ($Z^1$ = OMe, $Z^2$ = H), **S2b** ($Z^1$ = OMe, $Z^2$ = OMe), and **S2d** ($Z^1$ = Cl, $Z^2$ = H) were commercially available and used without purification.

### General Procedure A for the Synthesis of *N*-Alkoxyphthalimide

To a solution of the alcohol in THF was added *N*-hydroxyphthalimide and $PPh_3$, diisopropyl azodicarboxylate (40% in toluene, ca. 1.9 mol/L) dropwise at 0 °C. The reaction mixture was allowed to warm to room temperature and stirred overnight. Volatile materials were removed under reduced pressure, and the residue was purified by flash column chromatography on silica gel to afford *N*-alkoxypthalimide.

### General procedure B for the Directing Group Installation Step.

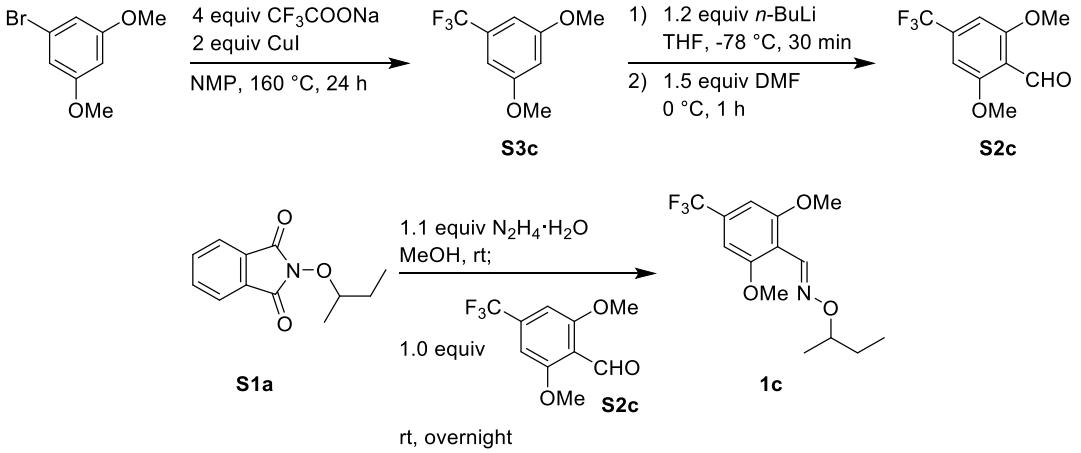
To a solution of the *N*-alkoxypthtalimide in MeOH or THF was added hydrazine monohydrate at room temperature. The progress of the reaction was monitored by TLC (usually completed in 2 h). After complete consumption of the *N*-alkoxypthtalimide was observed, 2,6-dimethoxybenzaldehyde was added to the mixture, which was then stirred overnight. The resulting mixture was filtered, if any precipitate was formed, and the filtrate was concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel to give oxime ether **1**.



**1b**

**Oxime ether 1b.** General procedure B was followed with 1.094 g (5.0 mmol) of **S1a**, 25 mL of MeOH, 249 mg of hydrazine monohydrate (5.0 mmol, 1.0 equiv) and 981 mg of 2,4,6-trimethoxybenzaldehyde **S2b** (5.0 mmol, 1.0 equiv). Silica gel chromatography (hexane/EtOAc 4:1) of the crude material afforded 1.205 g of **1b** (4.50 mmol, 91% yield) as a yellow oil. $^{1}$H NMR (400 MHz, $CDCl_3$): δ 8.36 (s, 1H), 6.12 (s, 1H), 4.30-4.22 (m, 1H), 3.83 (s, 9H), 1.81-1.71 (m, 1H), 1.61-1.50 (m, 1H), 1.27 (d, $J$ = 6.3 Hz, 3H), 0.96 (t, $J$ = 7.5 Hz, 3H); $^{13}$C{$^{1}$H} NMR (100 MHz, $CDCl_3$): δ 162.0, 160.0, 143.2, 103.2, 90.8, 80.0, 56.0, 55.3, 28.5, 19.2, 9.7; IR (NaCl): 2967 s, 2937 s, 2879 s, 2840 s, 1735 m, 1605 s, 1496 s, 1455 s, 1438 s, 1415 s, 1373 m, 1328 s, 1207 s, 1186 m, 1157 s, 1127 s, 1061 m, 1033 s, 993 s, 942 s, 900 m, 813 s, 791 w, 690 w, 636 w, 609 w, 588 w, 550 w cm$^{-1}$; HRMS (ESI-TOF) *m/z* $[M + H]^+$ calcd for $C_{14}H_{22}NO_4^+$ 268.1543, found 268.1541.

**Synthesis of Oxime ether 1c**




1,3-Dimethoxy-5-(trifluoromethyl)benzene **S3c** was prepared according to the literature procedure[4] with slight modifications.

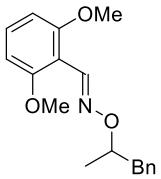
A solution of sodium trifluoroacetate (2.758 g, 20 mmol), CuI (1.920 g, 10 mmol) and 1-bromo-3,5-dimethoxybenzene (1.095 g, 5.0 mmol) in NMP (40 mL) in an oven-dried round bottom flask was stirred at 160 °C (oil bath temperature) for 24 h under $N_2$. After the oil bath temperature was lowered to 120 °C, the reaction flask was connected to a vacuum system with a trap immersed in liquid $N_2$ to condense the product. The material in the trap was collected with ether and washed once with an aqueous HCl solution (10%) and 3 times with water. The ether layer was dried over $MgSO_4$, filtered, and concentrated in vacuo. Purification by GPC afforded 354 mg of 1,3-dimethoxy-5-(trifluoromethyl)benzene **S3c** (1.7 mmol, 34% yield) as a colorless oil: $^{1}$H NMR (400 MHz, $CDCl_3$):

δ 6.74 (d, $J$ = 2.3 Hz, 2H), 6.59 (t, $J$ = 2.3 Hz, 1H). 3.83 (s, 6H); $^{19}$ F NMR (MHz, $CDCl_3$); δ -62.8. The analytical data of **S3c** are in good agreement with those reported in literature.[5]

Ortho lithiation[6] and formylation[7] were conducted according to the literature procedure with slight modifications.

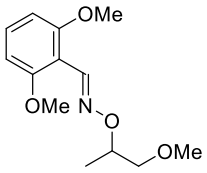
To a THF solution of 1,3-dimethoxy-5-(trifluoromethyl)benzene **S3c** (354 mg, 1.7 mmol in 3.4 mL THF) in an oven-dried 100 mL round bottom flask under $N_2$ was slowly added at –78 °C a solution of *n*-BuLi (1.30 mL, 1.95 mmol, 1.5 M in hexane), and the reaction mixture was stirred at –78 °C for 30 min. Dimethyl formamide (0.23 mL, 2.9 mmol) was added dropwise to the resulting mixture at 0 °C and the solution was stirred for 1 h. The reaction mixture was quenched by addition of a saturated aqueous solution of $NH_4Cl$ (10 mL) and extracted with $Et_2O$. The combined organic portions were washed with brine, dried over anhydrous $MgSO_4$, and concentrated. The crude material was purified by silica gel column chromatography (hexane/EtOAc 5:1) to afford 275 mg of **S2c** as a white solid (1.2 mmol, 69% yield): Mp. 106-109 °C; $^1$H NMR (400 MHz, $CDCl_3$): δ 10.50 (s, 1H), 6.81 (s, 2H), 3.95 (s, 6H); $^{13}$C{$^1$H} NMR (100 MHz, $CDCl_3$): δ 188.5, 162.0, 136.5 (q, $J$ = 32.7 Hz), 123.1 (q, $J$ = 273.1 Hz), 116.0, 100.9 (q, $J$ = 3.8 Hz), 56.3; $^{19}$F NMR (370 MHz, $CDCl_3$) δ -63.7; IR (KBr): 2955 w, 2876 w, 2782 w, 1696 m, 1617 w, 1585 m, 1484 m, 1470 m, 1409 m, 1353 s, 1262 s, 1239 s, 1181 m, 1168 m, 1128 s, 1100 m, 926 w, 899 w, 839 m, 796 w, 714 w, 679 w cm$^{-1}$; HRMS (ESI-TOF) *m/z* [M + Na]$^+$ calcd for $C_{10}H_9F_3NaO_3^+$ 257.0396, found 257.0395.

General procedure B was followed with 308.4 mg of **S1a** (1.4 mmol) in MeOH (7 mL), 76.2 mg of hydrazine monohydrate (1.5 mmol, 1.1 equiv) and 330 mg of **S2b** (1.4 mmol, 1.0 equiv). Silica gel chromatography (hexane/EtOAc 10:1) of the crude material afforded 379 mg of **1c** (1.2 mmol, 88% yield) as colorless oil. $^1$H NMR (400 MHz, $CDCl_3$): δ 8.33 (s, 1H), 6.71 (s, 2H), 4.33-4.25 (m, 1H), 3.88 (s, 6H), 1.82-1.71 (m, 1H), 1.63-1.52 (m, 1H), 1.27 (d, $J$ = 6.3 Hz, 3H), 0.97 (t, $J$ = 7.5 Hz, 3H); $^{13}$C{$^1$H} NMR (100 MHz, $CDCl_3$): δ 159.1, 142.1, 132.2 (q, $J$ = 32.6 Hz), 123.9 (q, $J$ = 272.5 Hz), 113.2, 101.1 (q, $J$ = 4.1 Hz), 80.7, 56.3, 28.7, 19.3, 9.8; $^{19}$F NMR (370 MHz, $CDCl_3$) δ -62.9; IR (NaCl): 2971 s, 2940 m, 2880 m, 2846 m, 1606 s, 1579 s, 1493 m, 1461 s, 1415 s, 1355 s, 1300 w, 1258 s, 1242 s, 1208 s, 1185 s, 1167 s, 1127 s, 1031 m, 993 s, 952 s, 897 s, 839 s, 762 w, 742 w, 711 m, 676 m, 605 w, 542 w cm$^{-1}$; HRMS (ESI-TOF) *m/z* [M + Na]$^+$ calcd for $C_{14}H_{18}F_3NNaO_3^+$ 328.1131, found 328.1132.



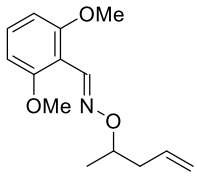
**1n**

**Oxime ether 1n**. General procedure B was followed with 2.750 g (9.8 mmol) of **S1n**, 20 mL of THF, 584 mg of hydrazine monohydrate (11.7 mmol, 1.2 equiv), and 1.951 g of 2,6-dimethoxybenzaldehyde (11.7 mmol, 1.2 equiv). Silica gel chromatography (hexane/EtOAc 5:1) of the crude material afforded 2.711 g of **1n** (9.1 mmol, 93% yield) as a colorless oil. $^{1}$H NMR (400 MHz, $CDCl_3$): δ 8.42 (s, 1H), 7.31-7.19 (m, 6H), 6.57 (d, *J* = 8.3 Hz, 2H), 4.64-4.56 (m, 1H), 3.85 (s, 6H), 3.18 (dd, *J* = 13.5, 5.4 Hz, 1H), 2.83 (dd, *J* = 13.5, 7.7 Hz, 1H), 1.25 (d, *J* = 6.3 Hz, 3H); $^{13}$C{$^{1}$H} NMR (100 MHz, $CDCl_3$): δ 159.0, 143.7, 138.6, 130.6, 129.7, 128.1, 126.0, 109.8, 104.1, 79.4. 56.1, 41.9, 18.9; IR (NaCl): 3086 w, 3061 w, 3026 m, 3002 m, 2969 s, 2934 s, 2838 m, 1596 s, 1470 s, 1433 s, 1373 w, 1340 m, 1307 m, 1285 w, 1256 s, 1208 s, 1184 w, 1114 s, 1034 m, 958 s, 888 w, 815 w, 778 s, 746 m, 729 m, 702 m, 633 w cm$^{-1}$; HRMS (ESI-TOF) *m/z* [M + Na]$^{+}$ calcd for $C_{18}H_{21}NNaO_3^{+}$ 322.1414, found 322.1412.



**1o**

**Oxime ether 1o.** General procedure B was followed with 1.012 g (4.3 mmol) of **S1o**, 22 mL of MeOH, 222 mg of hydrazine monohydrate (4.4 mmol, 1.0 equiv) and 716 mg of 2,6-dimethoxybenzaldehyde (4.3 mmol, 1.0 equiv). Silica gel chromatography (hexane/EtOAc 3:1) of the crude material afforded 1.01 g of **1o** (3.67 mmol, 94% yield) as colorless oil. $^{1}$H NMR (400 MHz, $CDCl_3$): δ 8.44 (s, 1H), 7.25 (t, *J* = 8.3 Hz, 1H), 6.61 (d, *J* = 8.3 Hz, 2H), 4.48-4.55 (m, 1H), 3.84 (s, 6H), 3.61 (dd, *J* = 10.3, 5.4 Hz, 1H), 3.54 (dd, *J* = 10.3, 4.5 Hz, 1H), 3.42 (s, 3H), 1.38 (d, *J* = 6.7 Hz, 2H); $^{13}$C{$^{1}$H} NMR (100 MHz, $CDCl_3$): δ 159.0, 144.1, 130.6, 109.7, 104.1, 77.6, 75.1, 59.3, 56.0, 16.7; IR (NaCl): 2975 s, 2934 s, 2838 s, 2813 m, 1596 s, 1470 s, 1434 s, 1371 m, 1337 m, 1306 s, 1284 m, 1257 s, 1206 s, 1186 m, 1148 m, 1114 s, 1034 m, 975 s, 891 s, 831 w, 819 m, 779 w, 730 m, 632 m cm$^{-1}$; HRMS (ESI-TOF) *m/z* [M + Na]$^{+}$ calcd for $C_{13}H_{19}NNaO_4^{+}$ 276.1206, found 276.1204.

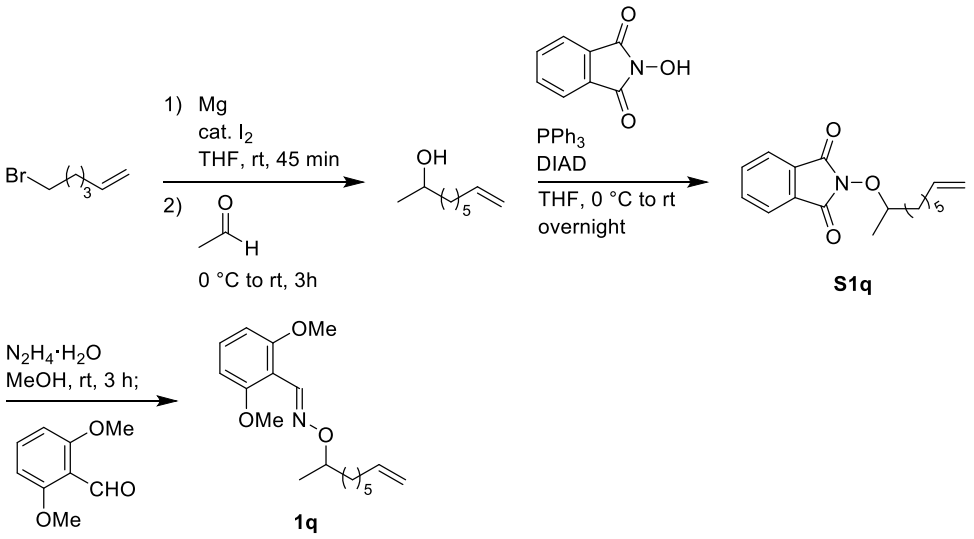


**1p**

**Oxime ether 1p.** General procedure A was followed with 363.3 mg of pent-4-en-2-ol (4.2 mmol), 986 mg of *N*-hydroxyphthalimide (6.0 mmol, 1.4 equiv), 1578 mg of $PPh_3$ (6.0 mmol, 1.4 equiv), 24 mL of THF, and diisopropyl azodicarboxylate (40% in toluene, ca. 1.9 mol/L, 3.2 mL, 1.4 equiv). Silica gel chromatography (hexane/EtOAc 5:1) of the crude material afforded 905 mg of **S1p** (3.9 mmol, 93% yield). $^{1}$H NMR (391.78 MHz, $CDCl_3$): δ 7.87-7.82 (m, 2H), 7.77-7.73 (m, 2H), 5.95-5.84 (m, 1H), 5.19-5.10 (m, 2H), 4.50-4.42 (m, 1H), 2.62-2.55 (m, 1H), 2.45-2.38 (m, 1H), 1.36 (d, *J* = 6.3 Hz, 3H). The analytical data for this product are in good agreement with those reported in literature.[8] General procedure B was followed with 838 mg (3.6 mmol) of **S1p** in MeOH (18 mL), 190 mg of hydrazine monohydrate (3.8 mmol, 1.1 equiv) and 606 mg of 2,6-dimethoxybenzaldehyde (3.6 mmol, 1.0 equiv). Silica gel chromatography (hexane/EtOAc 15:1) of the crude material afforded 817 mg of **1p** (3.3 mmol, 91% yield) as colorless oil. $^{1}$H NMR (400 MHz, $CDCl_3$): δ 8.39 (s, 1H), 7.25 (t,

$J$ = 8.5 Hz, 1H), 6.56 (d, $J$ = 8.5 Hz, 2H), 5.94-5.84 (m, 1H), 5.14-5.05 (m, 2H), 4.46-4.38 (m, 1H), 3.84 (s, 6H), 2.59-2.52 (m, 1H), 2.38-2.30 (m, 1H), 1.30 (d, $J$ = 6.3 Hz, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 158.8, 143.5, 134.8, 130.5, 116.8, 109.7, 104.0, 78.0, 56.0, 40.2, 19.1; IR (NaCl): 3075 w, 2972 s, 2935 s, 2839 m, 1641 w, 1596 m, 1470 s, 1433 m, 1374 w, 1330 m, 1306 m, 1256 s, 1207 s, 1185 w, 1114 s, 1033 m, 996 m, 962 s, 886 m, 778 s, 729 m, 633 w, 590 w $cm^{-1}$; HRMS (ESI-TOF) $m/z$ $[M + H]^+$ calcd for $C_{14}H_{20}NO_3^+$ 250.1438, found 250.1440.

**Synthesis of oxime ether 1q**

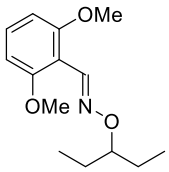



8-Nonen-2-ol was prepared according to the literature procedures[9] with slight modification.

To a suspension of Mg turnings (368 mg, 15 mmol, 1.1 equiv) and iodine (72.1 mg, 0.284 mmol, 0.02 equiv) in dry THF (30 mL) in an oven-dried 200 mL round-bottom flask under $N_2$ was slowly added 7-bromo-1-heptene (2.65 g, 15 mmol), and the mixture was stirred for 45 min at room temperature. The reaction mixture was then cooled to 0 °C in an ice bath, and acetaldehyde (0.84 mL, 1 equiv) was slowly added to the mixture. The reaction mixture was gradually warmed to room temperature by removing the ice bath with stirring for 3 h. The resulting mixture was quenched by addition of a saturated aqueous solution of $NH_4Cl$ and extracted with $Et_2O$. The combined organic portions were then washed with water, dried with $MgSO_4$, and filtered. After removing the volatile materials in vacuo, the crude product was purified by silica gel column chromatography to give the alcohol as a clear oil (82% purity, containing 18% of AcOEt, ca. 12.7 mmol), which was directly used for the subsequent step without further purification.

General Procedure A was followed with the alcohol, 2.490 g of *N*-hydroxyphthalimide (15 mmol, 1.2 equiv), 4.010 g of $PPh_3$ (15 mmol, 1.2 equiv), 50 mL of THF, and diisopropyl azodicarboxylate (40% in toluene, 8.0 mL, 1.2 equiv). Silica gel chromatography (hexane/EtOAc 5:1) of the crude material afforded 980 mg of **S1q** (3.4 mmol) as a colorless oil.

General Procedure B was followed with **S1q**, 173 mg of hydrazine monohydrate (3.5 mmol, 1 equiv), 569 mg of 2,6-dimethoxybenzaldehyde (3.4 mmol, 1.0 equiv), and 17 mL of MeOH. Silica gel chromatography (hexane/EtOAc 20:1) of the crude material afforded 615 mg of **1q** (2.0 mmol, 13% yield over 3 steps) as a colorless oil: $^{1}$H NMR (400 MHz, $CDCl_3$): δ 8.38 (s, 1H), 7.25 (t, *J* = 8.6 Hz, 1H), 6.56 (d, *J* = 8.6 Hz, 2H), 5.87-5.76 (m, 1H), 5.02-4.91 (m, 2H), 4.38-4.30 (m, 1H), 3.84 (s, 6H), 2.08-2.02 (m, 2H), 1.79-1.69 (m, 1H), 1.55-1.32 (m, 7H), 1.29 (d, *J* = 6.3 Hz, 3H); $^{13}$C{$^{1}$H} NMR (100 MHz, $CDCl_3$): δ 158.8, 143.0, 140.0, 130.2, 114.0, 109.8, 103.9, 78.8, 55.9, 35.6, 33.6, 29.1, 28.7, 25.2, 19.7; IR (NaCl): 3075 w, 2968 s, 1932 s, 2856 s, 2840 s, 1640 w, 1596 s, 1471 s, 1433 s, 1372 m, 1335 m, 1307 m, 1284 m, 1255 s, 1207 s, 1185 w, 1115 s, 1036 m, 996 m, 953 s, 908 m, 777 s, 727 m, 632 w cm$^{-1}$; HRMS (ESI-TOF) *m/z* [M + Na]$^{+}$ calcd for $C_{18}H_{27}NNaO_3^{+}$ 328.1883, found 328.1883.



**1r**

**Oxime ether 1r.** General Procedure A was followed with 445 mg of 3-pentanol (5.0 mmol), 984 mg of *N*-hydroxyphthalimide (6.0 mmol, 1.2 equiv), 1.575 g of $PPh_3$ (6.0 mmol, 1.2 equiv), 20 mL of THF, and diisopropyl azodicarboxylate (40% in toluene, ca. 1.9 mol/L, 3.2 mL, 1.4 equiv). After the reaction, hexanes were added to the reaction mixture, and the precipitate was collected to directly use for the next step without further purification. General Procedure B was then followed with 256 mg of hydrazine monohydrate (5.1 mmol, 1.0 equiv), 831 mg of 2,6-dimethoxybenzaldehyde (5.0 mmol, 1.0 equiv), and 25 mL of MeOH. Silica gel chromatography (hexane/EtOAc 10:1) of the crude material afforded 265 mg of **1r** (1.05 mmol, 21% yield over 2 steps) as a white solid: Mp. 53-55 °C. $^{1}$H NMR (400 MHz, $CDCl_3$): δ 8.40 (s, 1H), 7.24 (t, *J* = 8.2 Hz, 1H), 6.56 (d, *J* = 8.2 Hz, 2H), 4.13-4.07 (m, 1H), 3.84 (s, 6H), 1.74-1.62 (m, 4H), 0.97 (t, *J* = 7.5 Hz, 3H); $^{13}$C{$^{1}$H} NMR (100 MHz, $CDCl_3$): δ 158.9, 142.9, 130.3, 110.0, 104.1, 85.0, 56.0, 25.9, 9.6; IR (KBr): 3091 w, 3006 m, 2971 m, 2935 m, 2882 m, 2833 m, 1596 s, 1575 m, 1469 s, 1432 s, 1382 w, 1349 m, 1335 m, 1305 m, 1255 s, 1209 m, 1187 m, 1133 m, 1111 s, 1098 s, 1029 m, 1009 w, 982 m, 964 s, 938 s, 889 m, 859 w, 837 w, 789 s, 741 w, 635 m, 584 w cm$^{-1}$; HRMS (ESI-TOF) *m/z* [M + Na]$^{+}$ calcd for $C_{14}H_{21}NNaO_3^{+}$ 274.1414, found 274.1415.

## IV. Experimental Apparatus for Electrochemical Acetoxylation

**Figure S1.** Components of experimental apparatus prior to assembly.

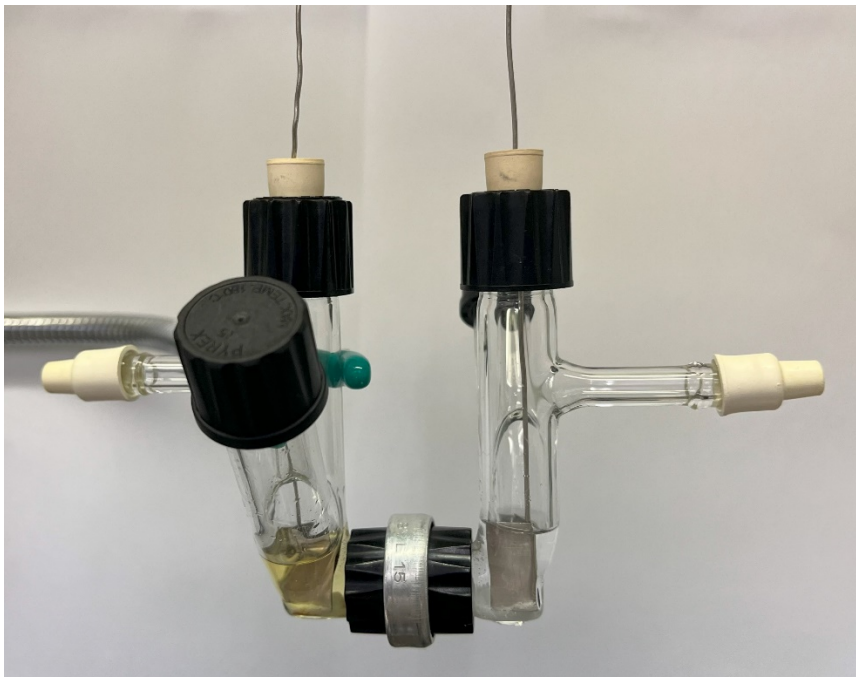


**Figure S2.** Experimental apparatus after assembly of components.

**V. References**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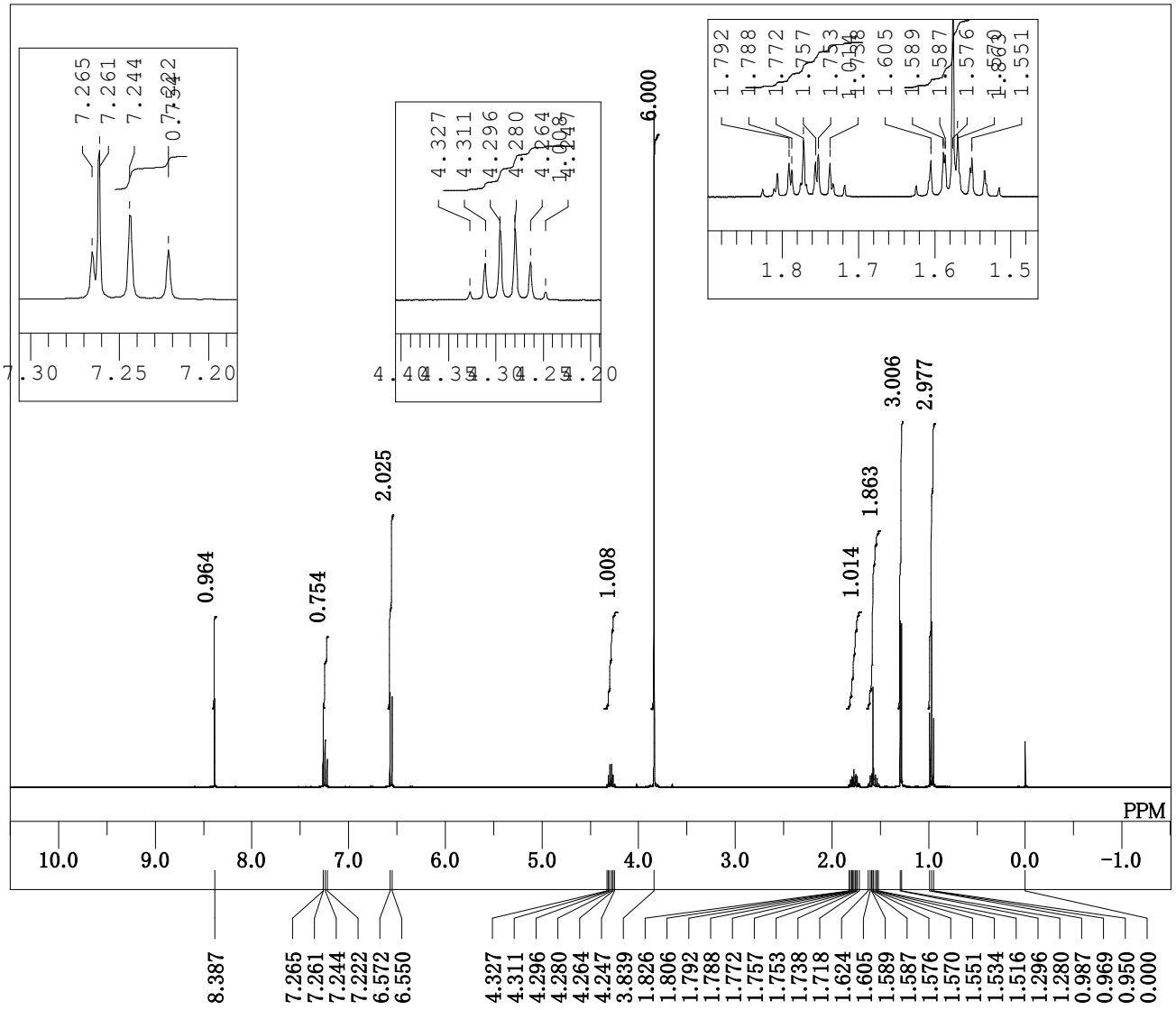
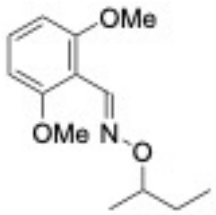
# VI. NMR Spectra

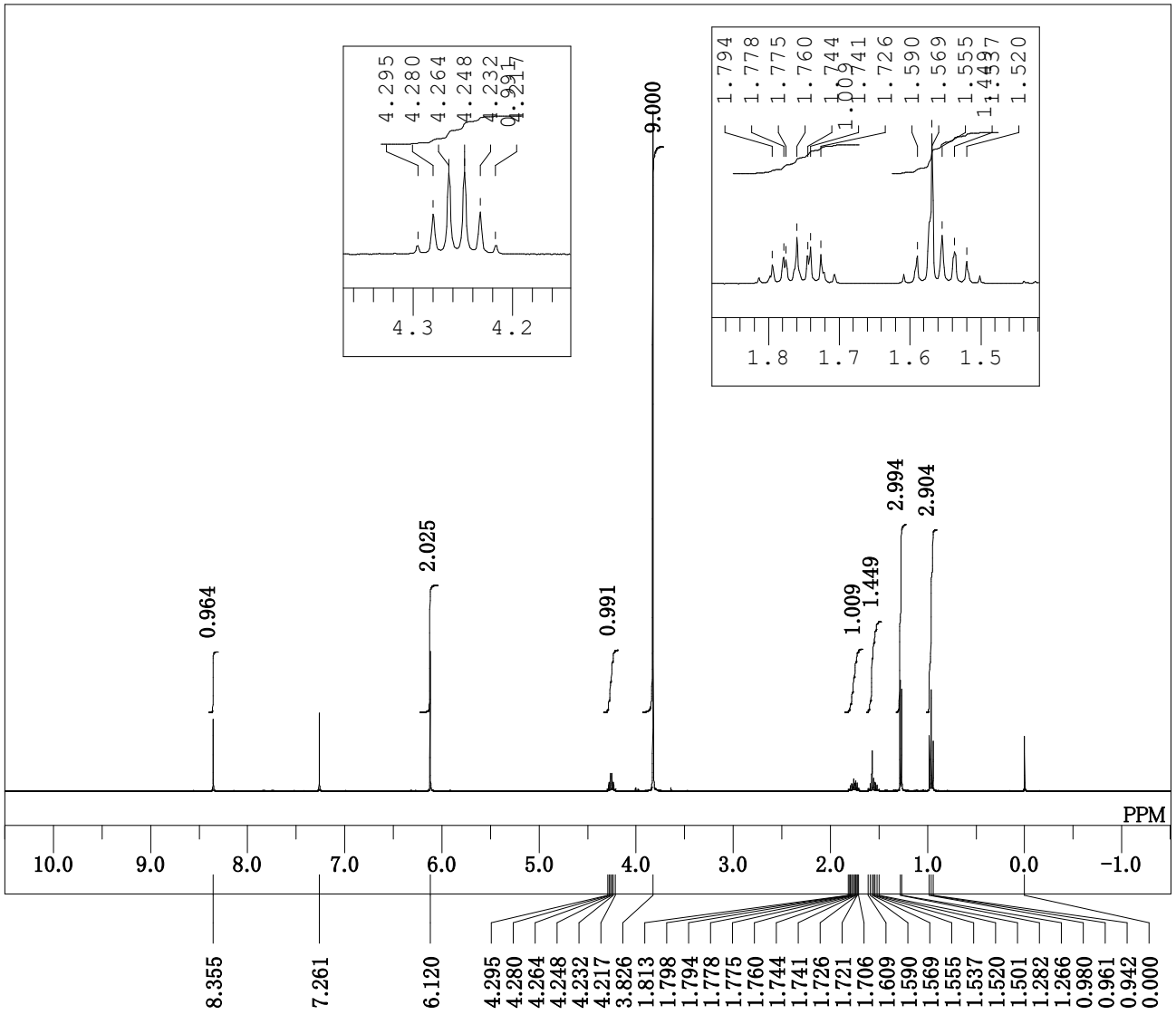
$^1$H NMR (395.88 MHz, Solvent=$CDCl_3$)

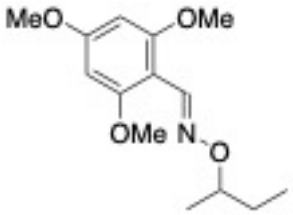



**1a**

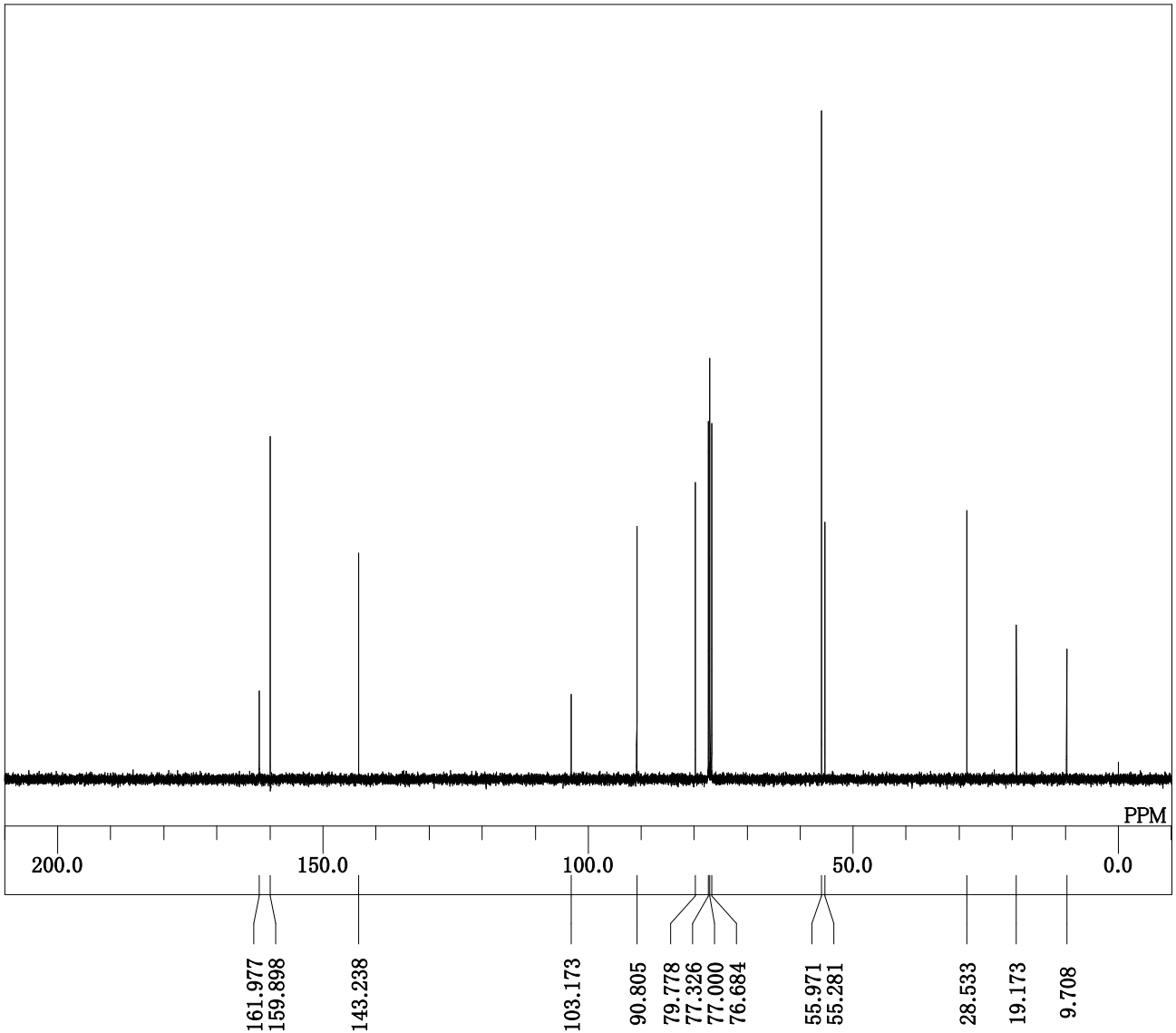
$^1$H NMR (395.88 MHz, Solvent=$CDCl_3$)

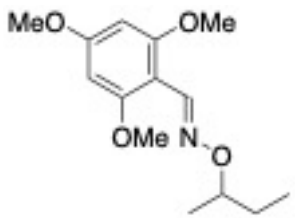



**1b**

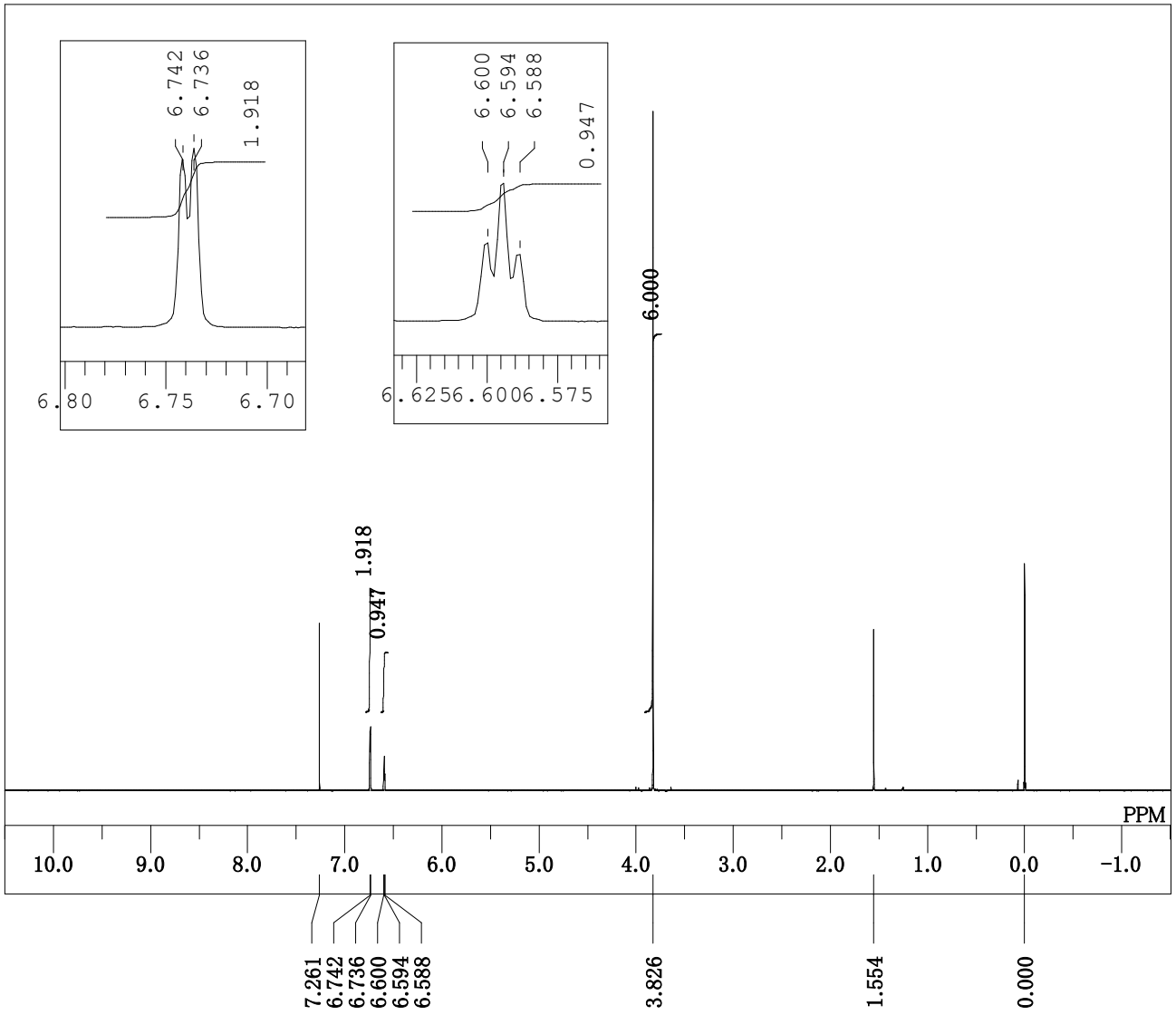
$^{13}$C NMR (99.55 MHz, Solvent=$CDCl_3$)

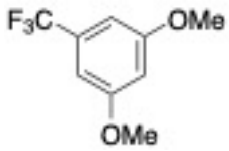



**1b**

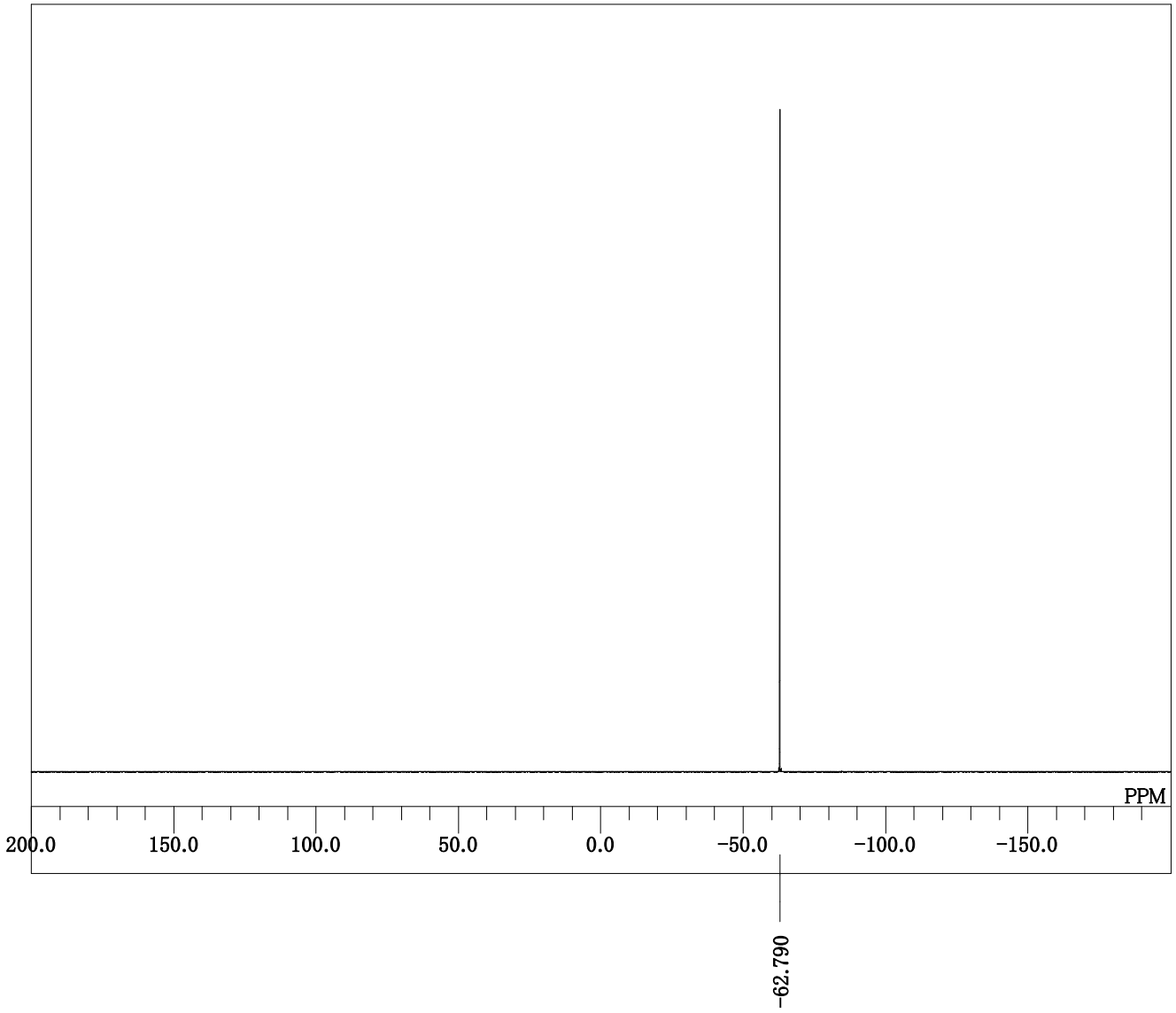
$^1$H NMR (395.88 MHz, Solvent=$CDCl_3$)

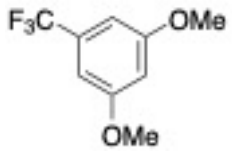



**S3c**

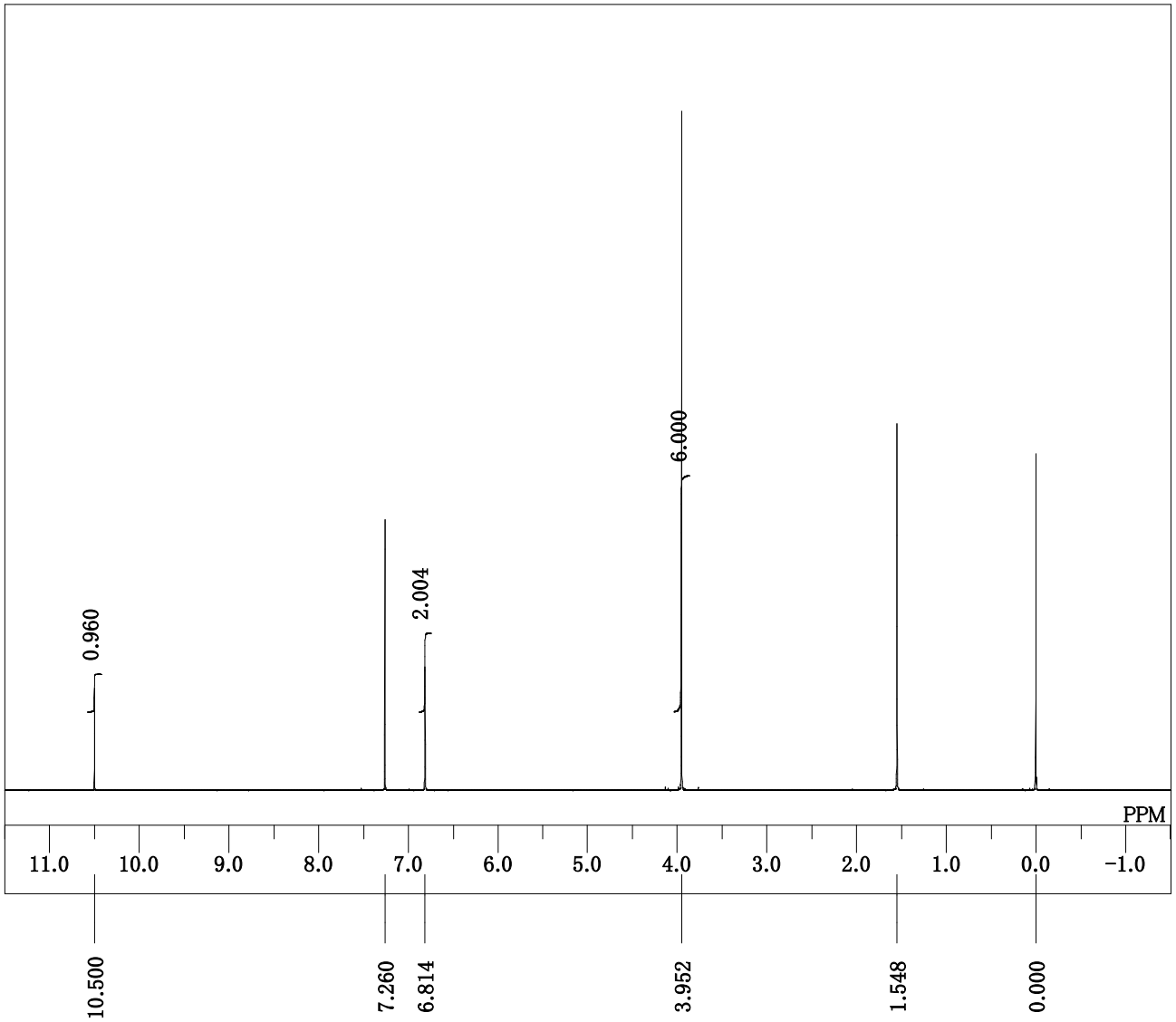
$^{19}F$ NMR (372.50 MHz, Solvent=$CDCl_3$)

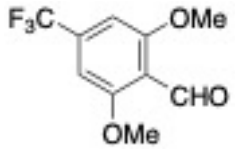



**S3c**

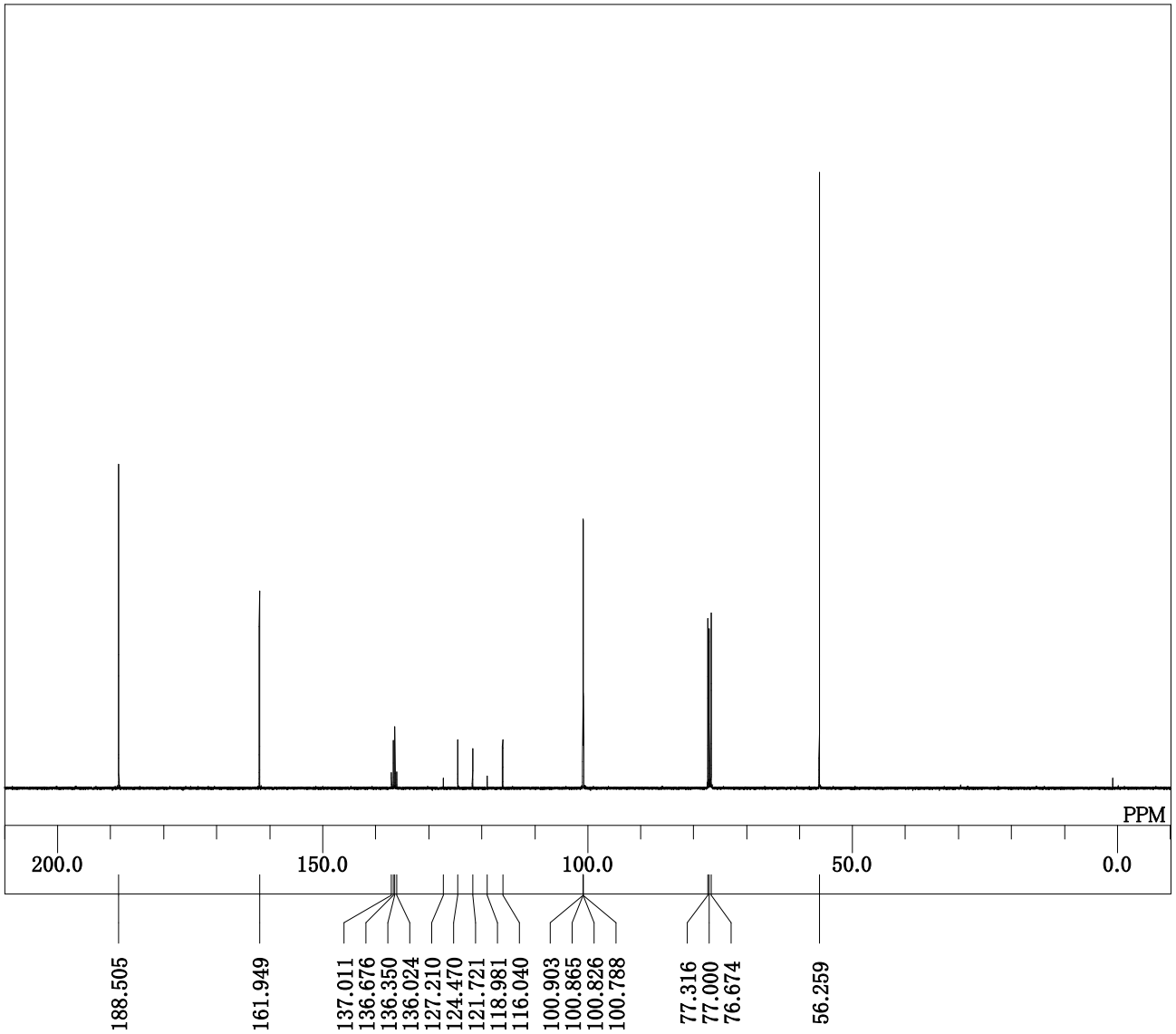
$^1$H NMR (395.88 MHz, Solvent=$CDCl_3$)

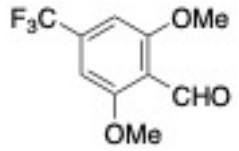



**S2c**

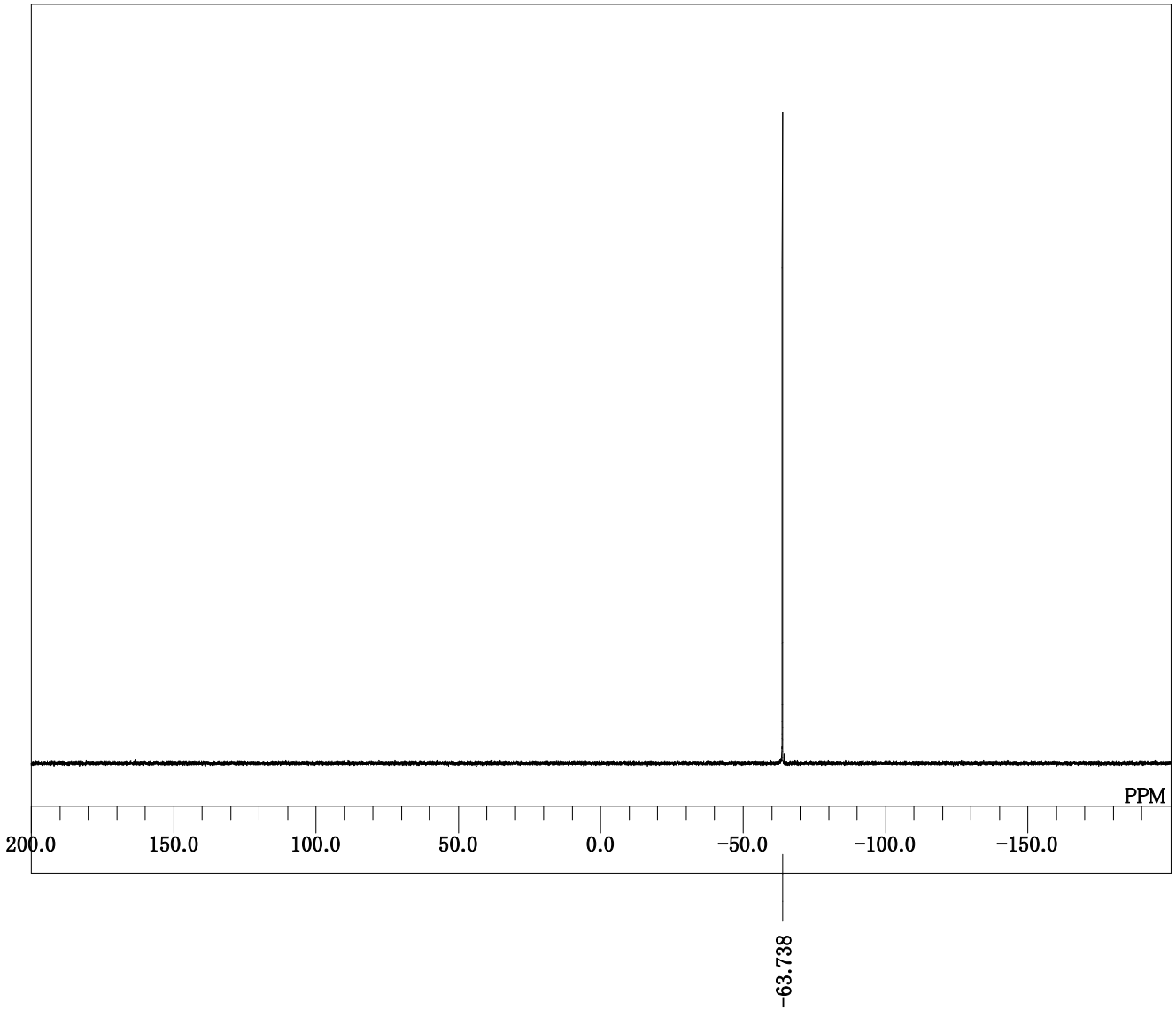
$^{13}$C NMR (99.55 MHz, Solvent=$CDCl_3$)

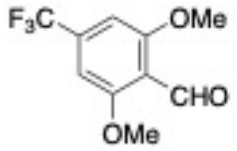



**S2c**

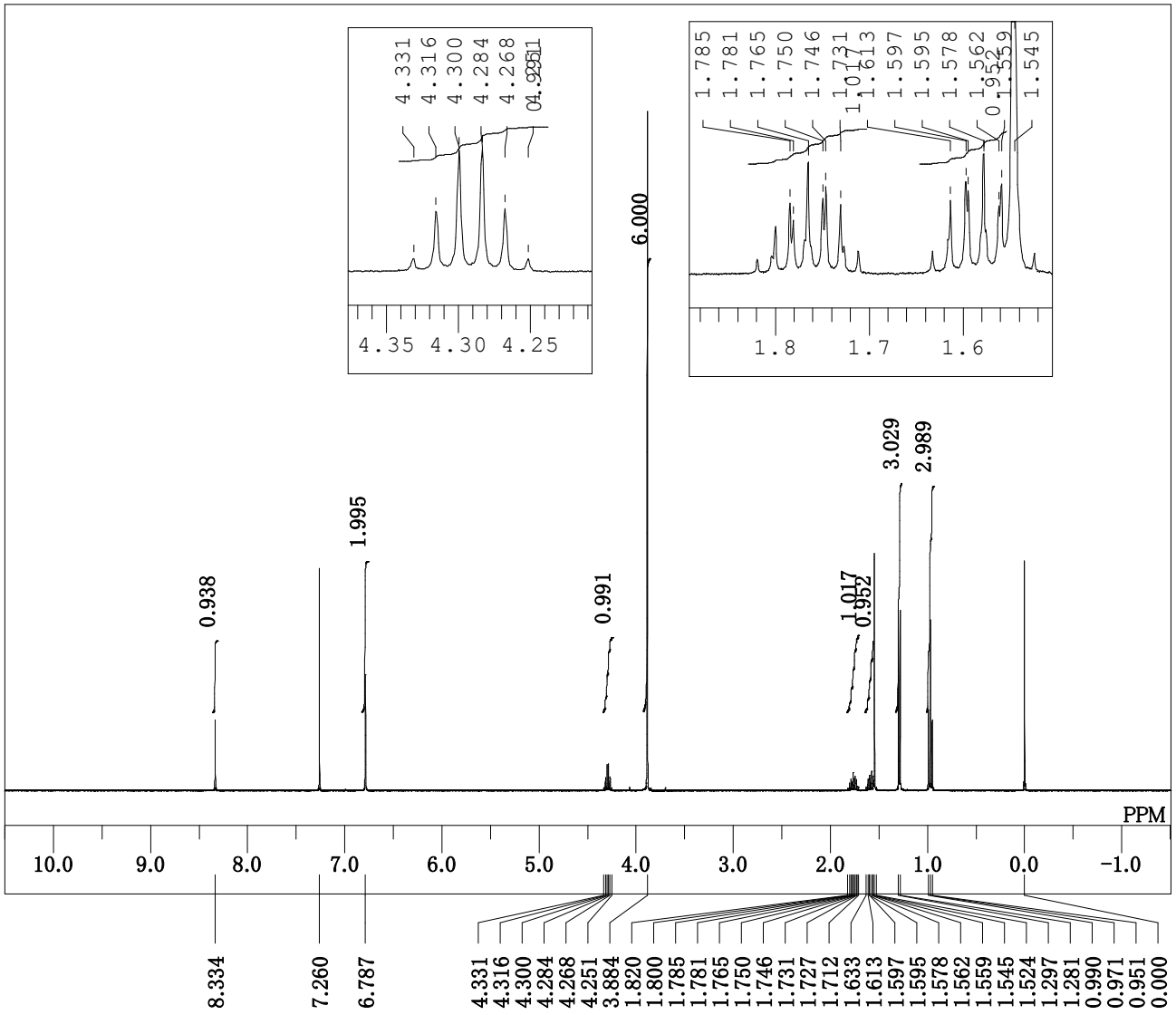
$^{19}F$ NMR (372.50 MHz, Solvent=$CDCl_3$)

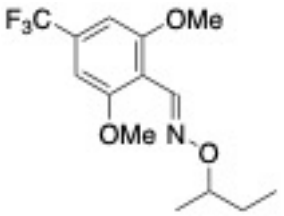



**S2c**

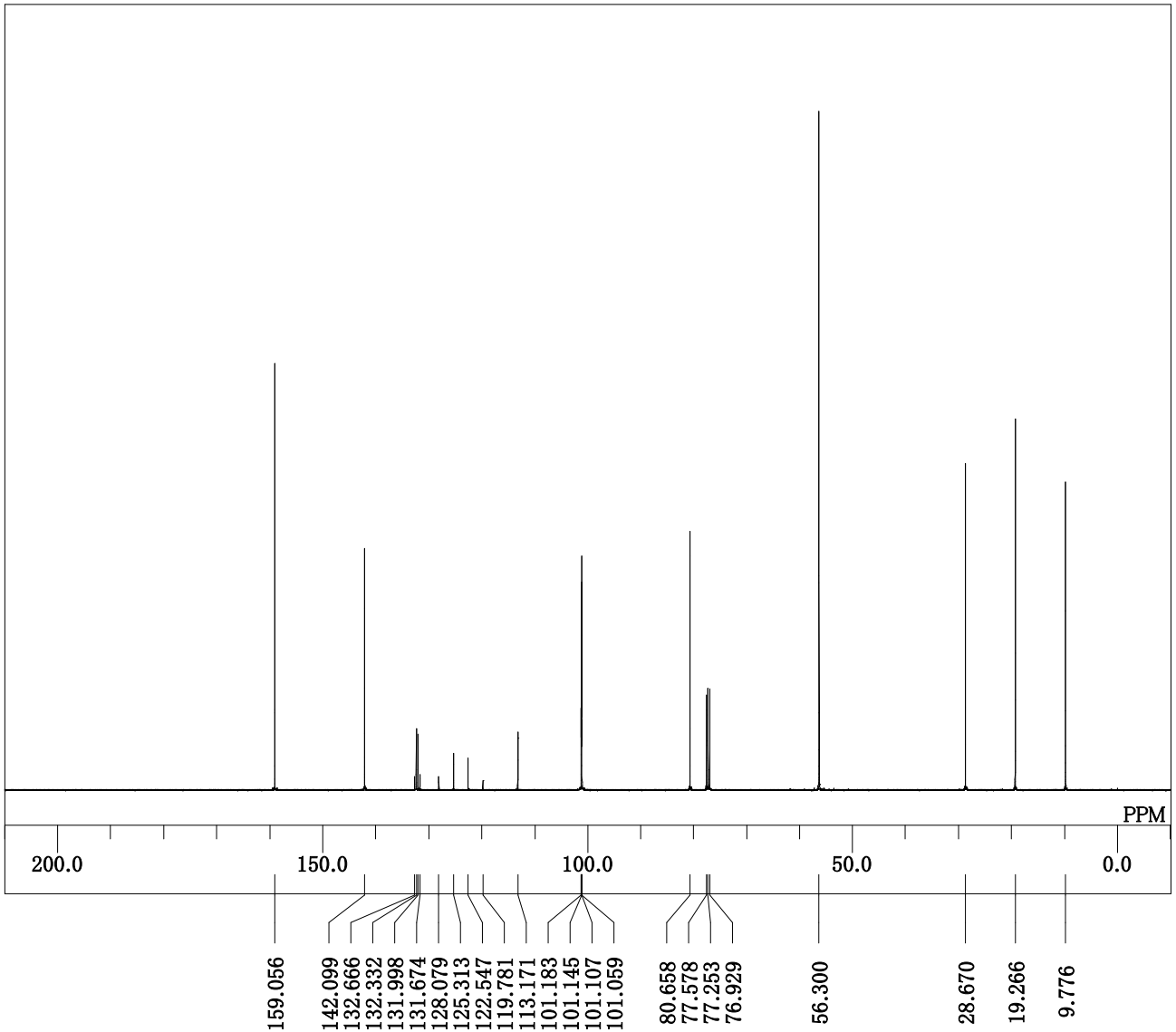
$^1$H NMR (395.88 MHz, Solvent=$CDCl_3$)

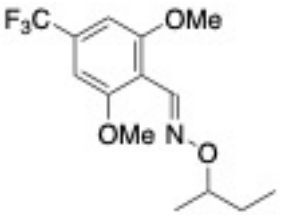



**1c**

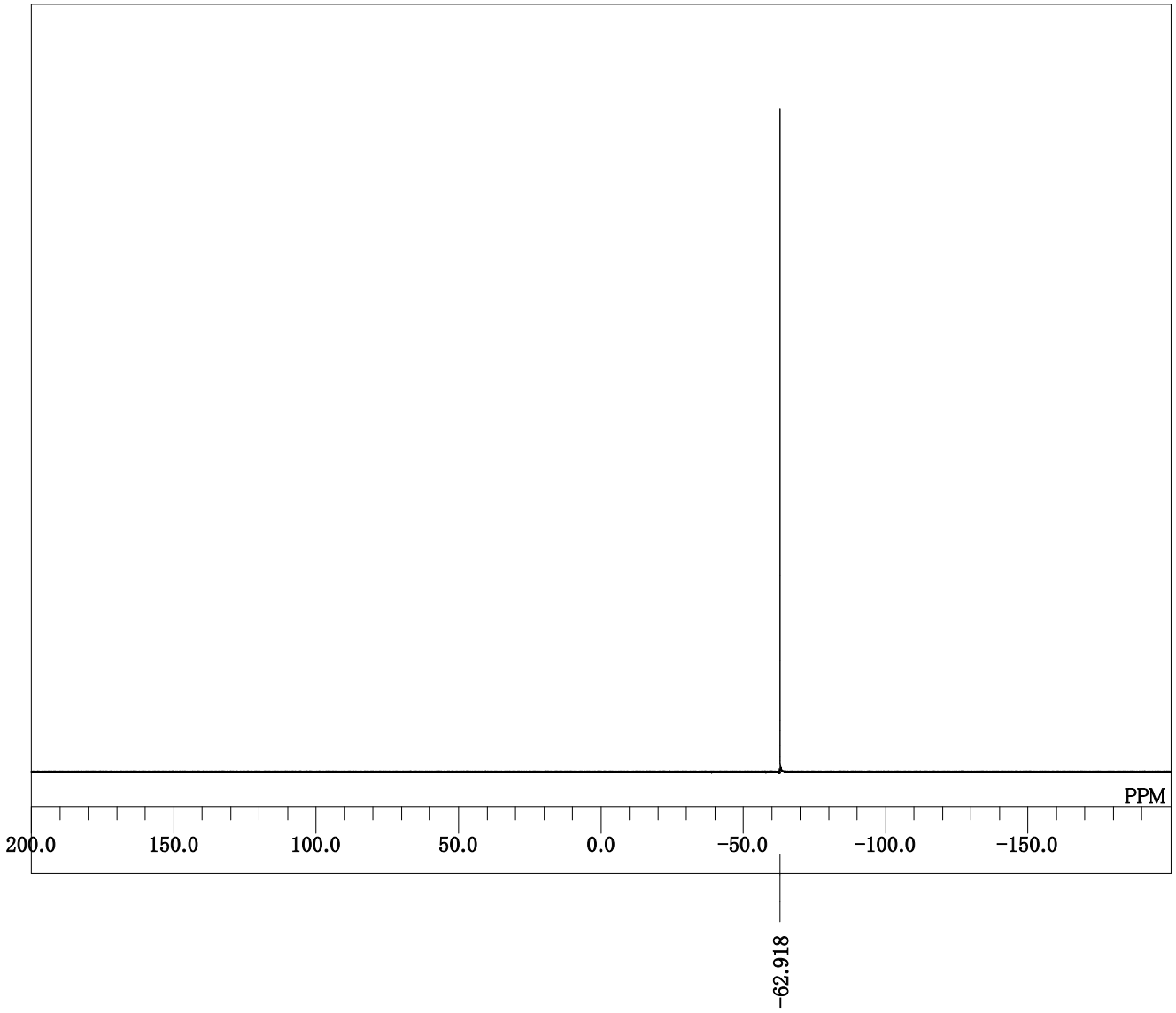
$^{13}C$ NMR (98.52 MHz, Solvent=$CDCl_3$)

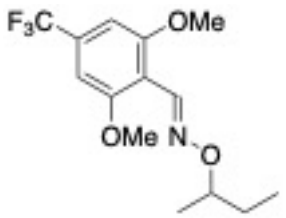



**1c**

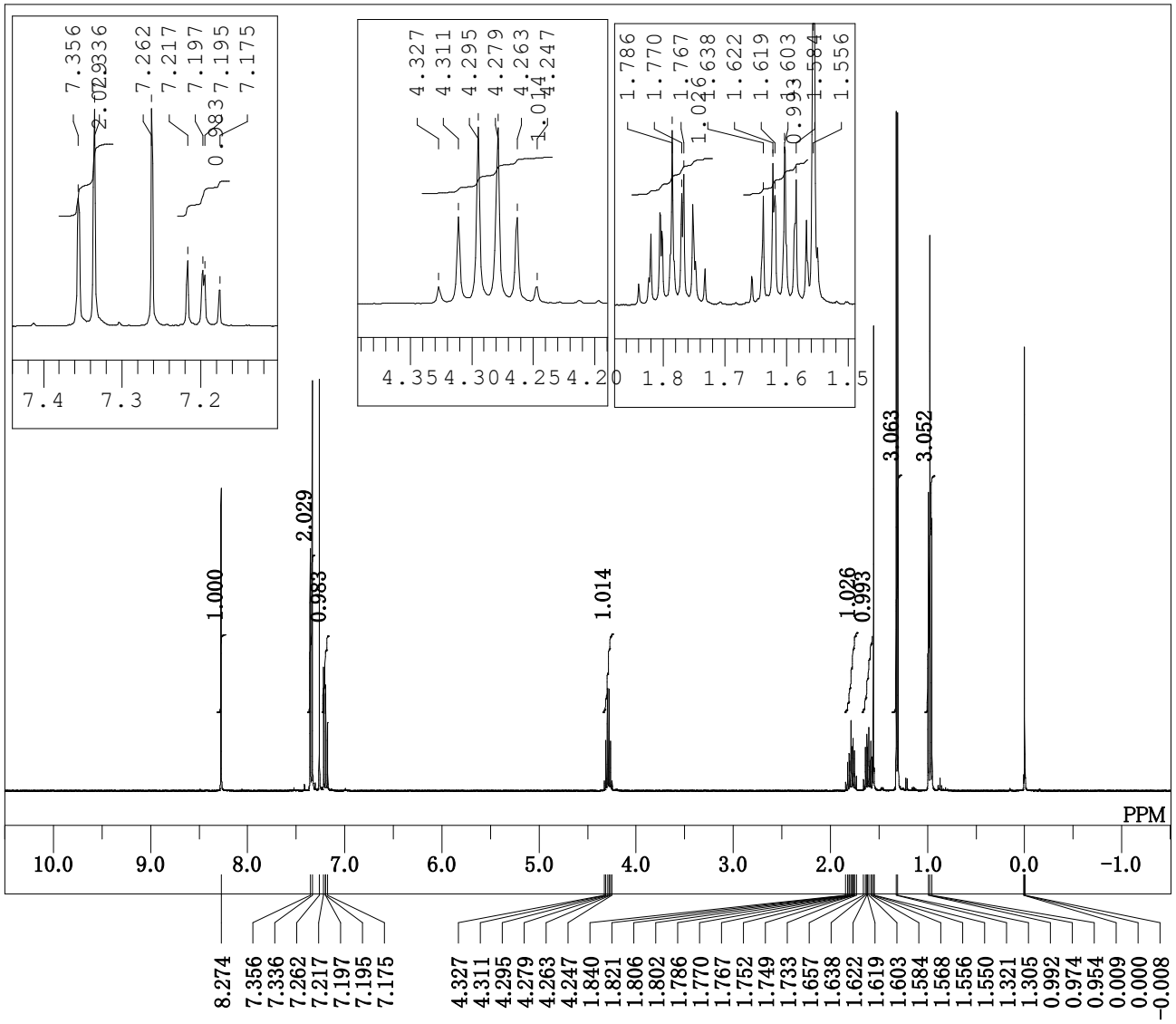
$^{19}F$ NMR (368.64 MHz, Solvent=$CDCl_3$)

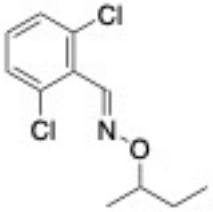



**1c**

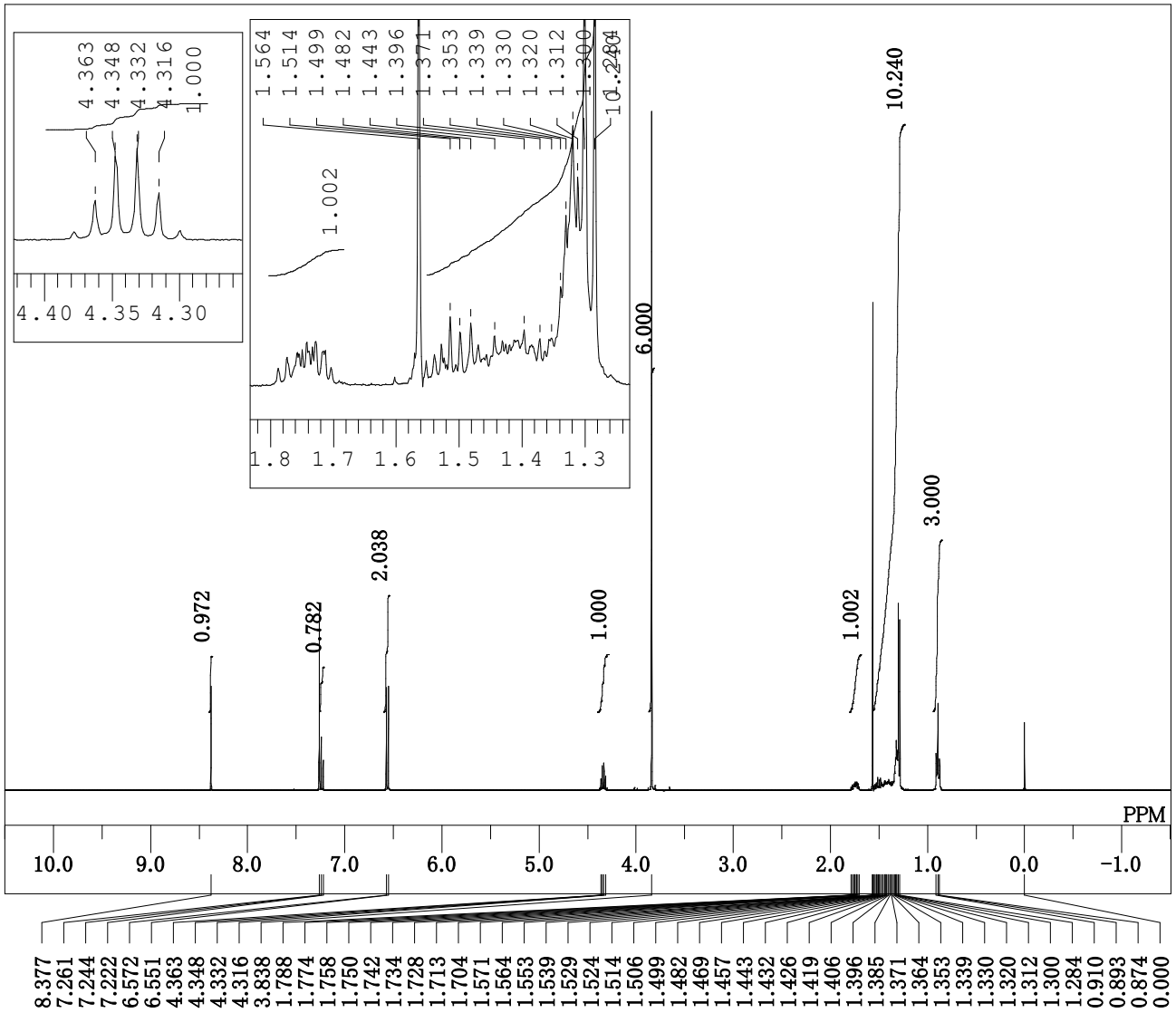
$^1$H NMR (395.88 MHz, Solvent=$CDCl_3$)

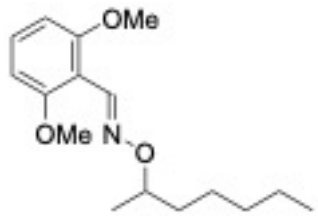



**1d**

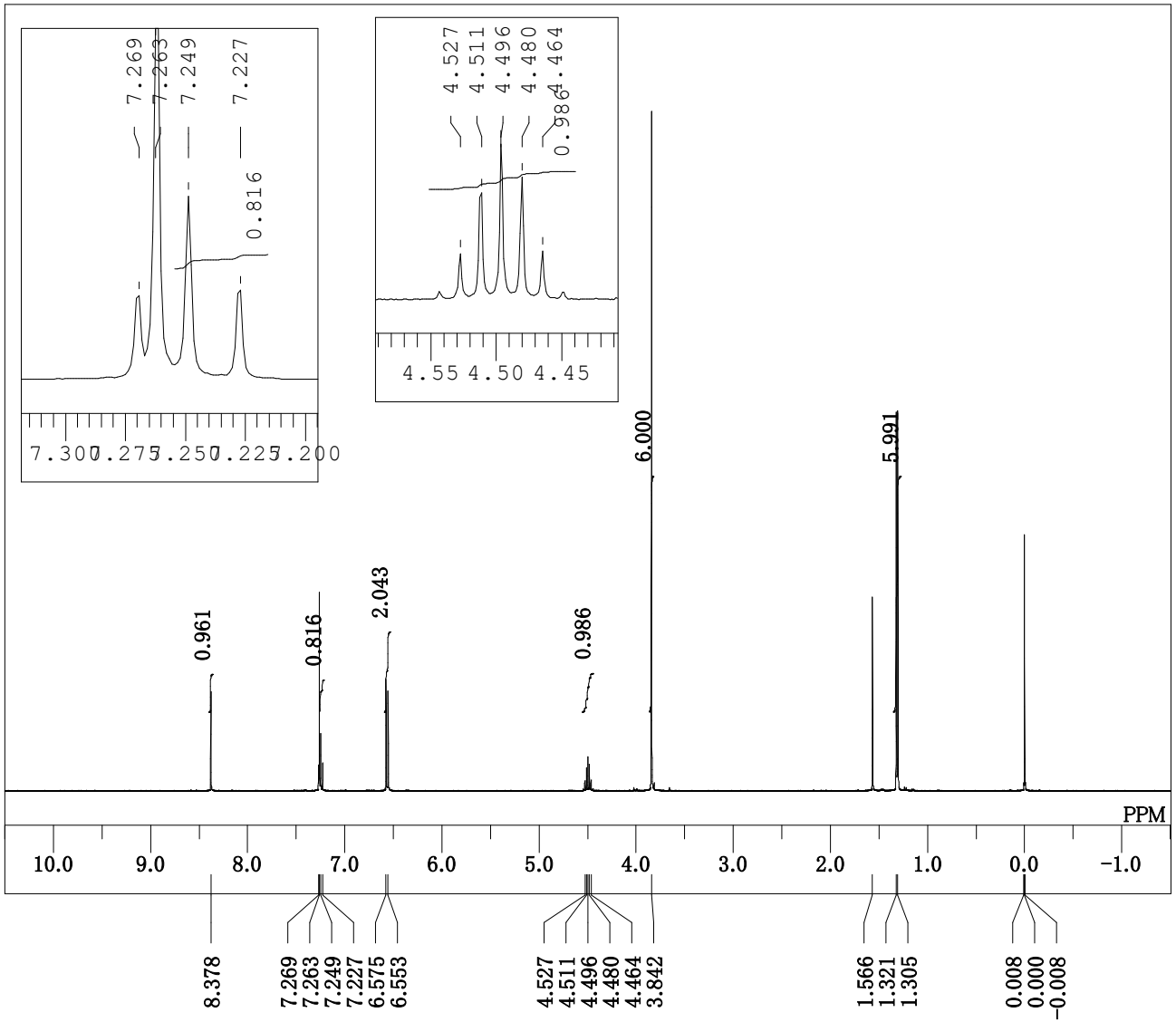
$^1$H NMR (395.88 MHz, Solvent=$CDCl_3$)

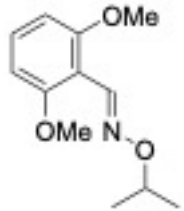



**1e**

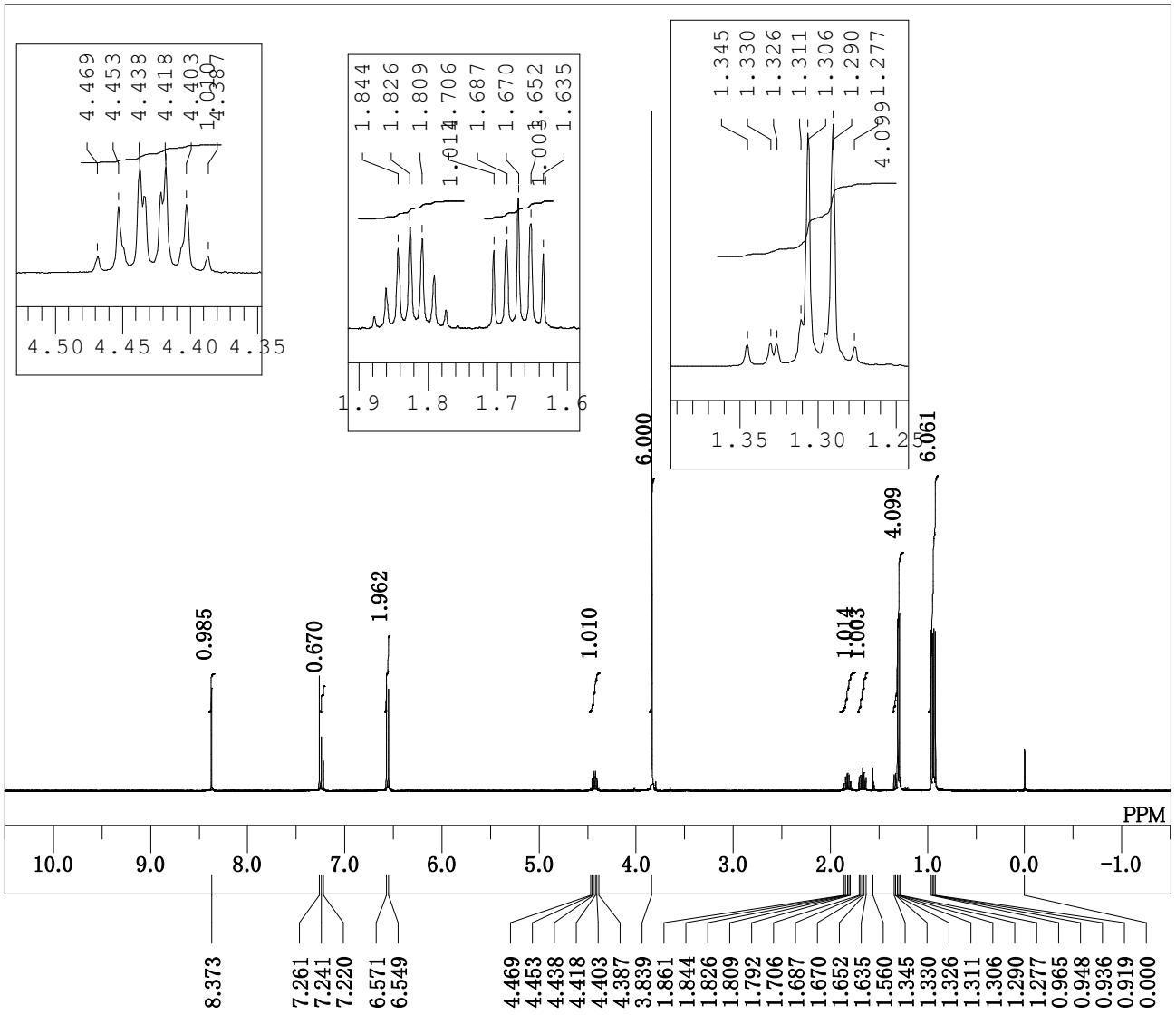
$^1$H NMR (395.88 MHz, Solvent=$CDCl_3$)

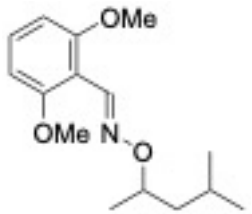



**1f**

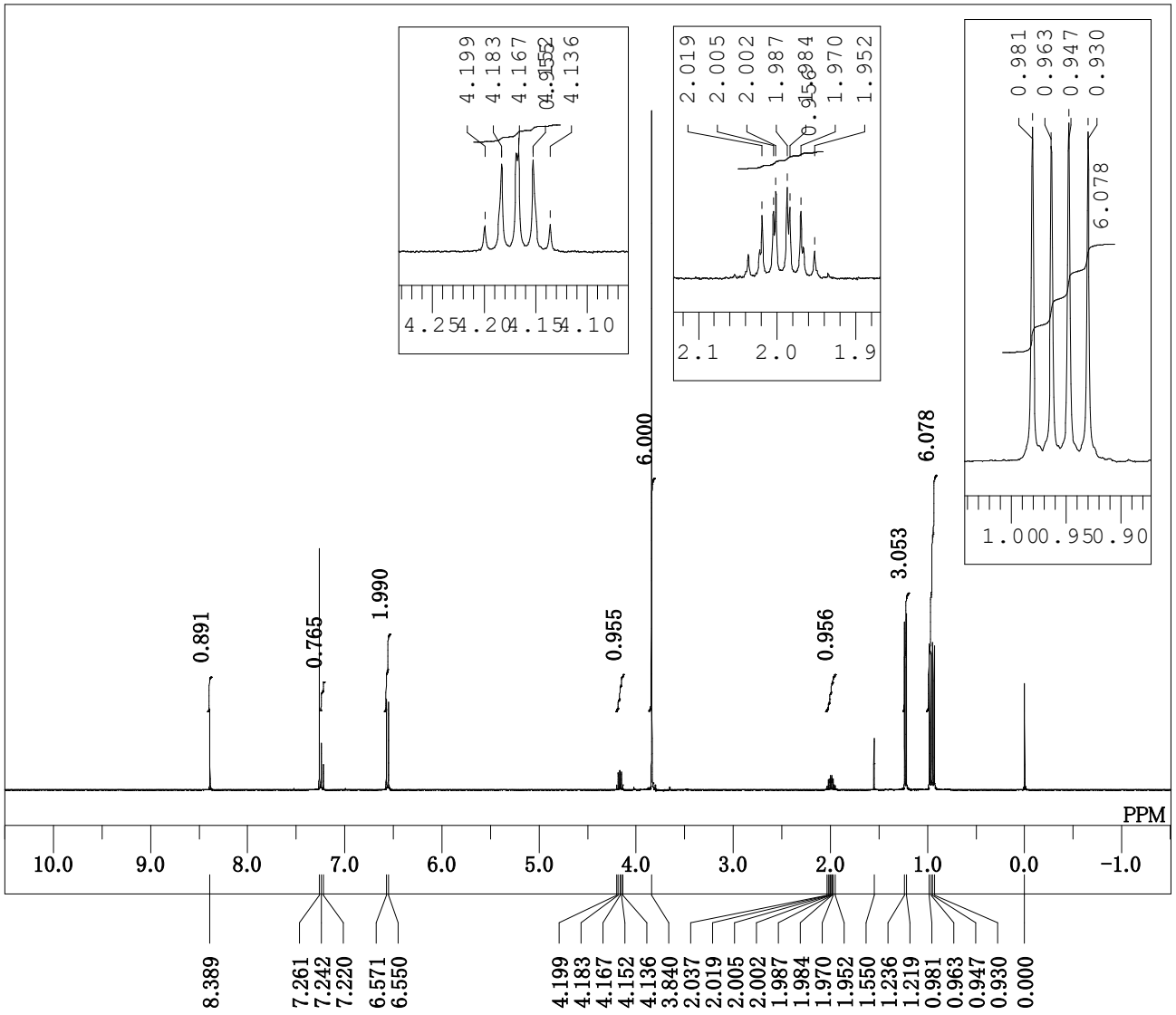
$^1$H NMR (395.88 MHz, Solvent=$CDCl_3$)

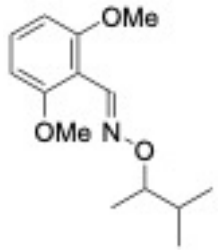



**1g**

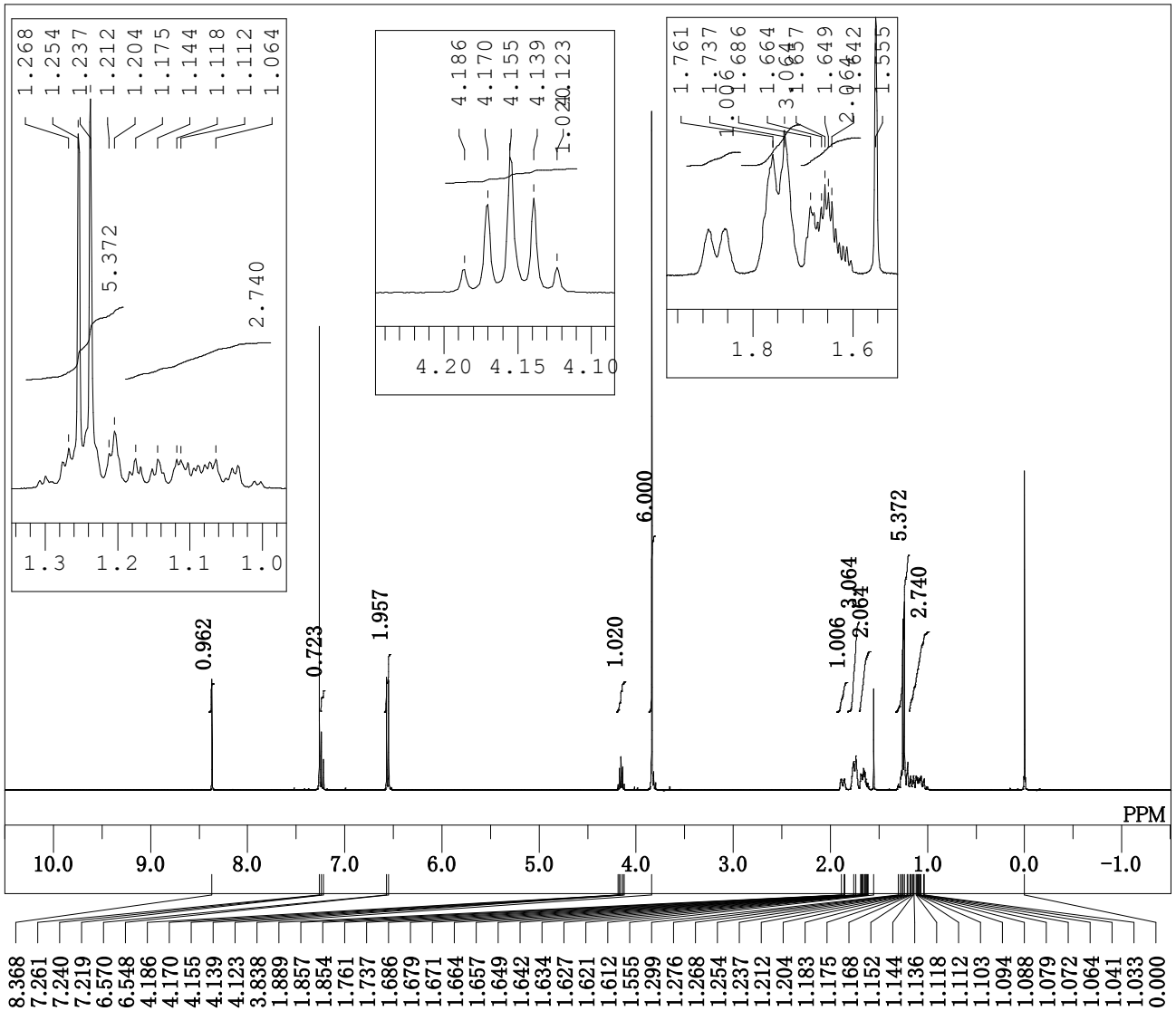
$^1$H NMR (395.88 MHz, Solvent=$CDCl_3$)

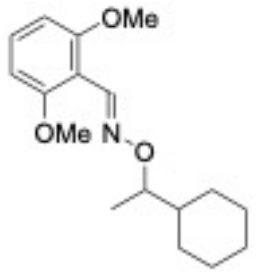



**1h**

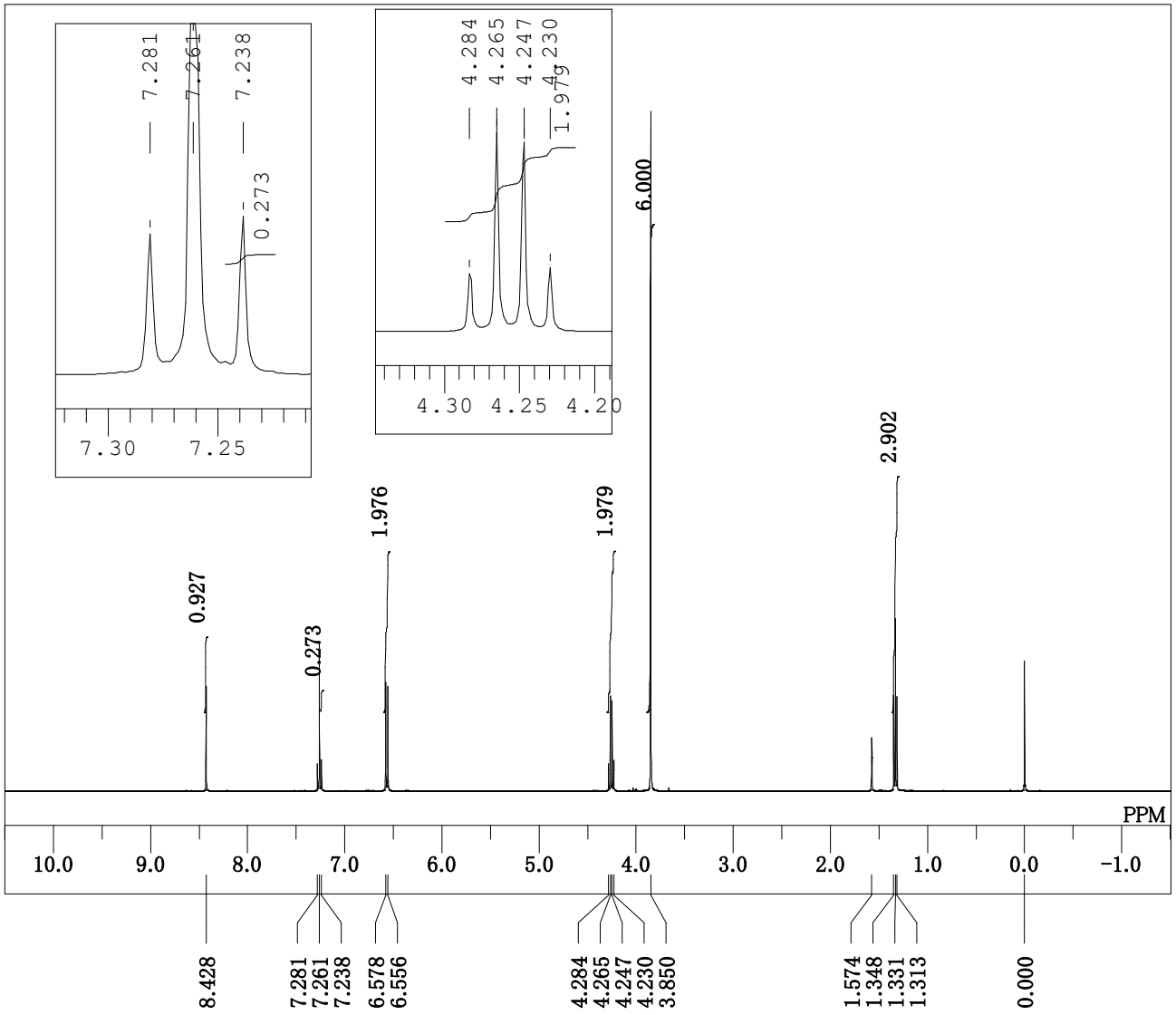
$^1$H NMR (391.78 MHz, Solvent=$CDCl_3$)

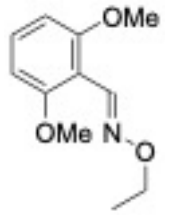



**1i**

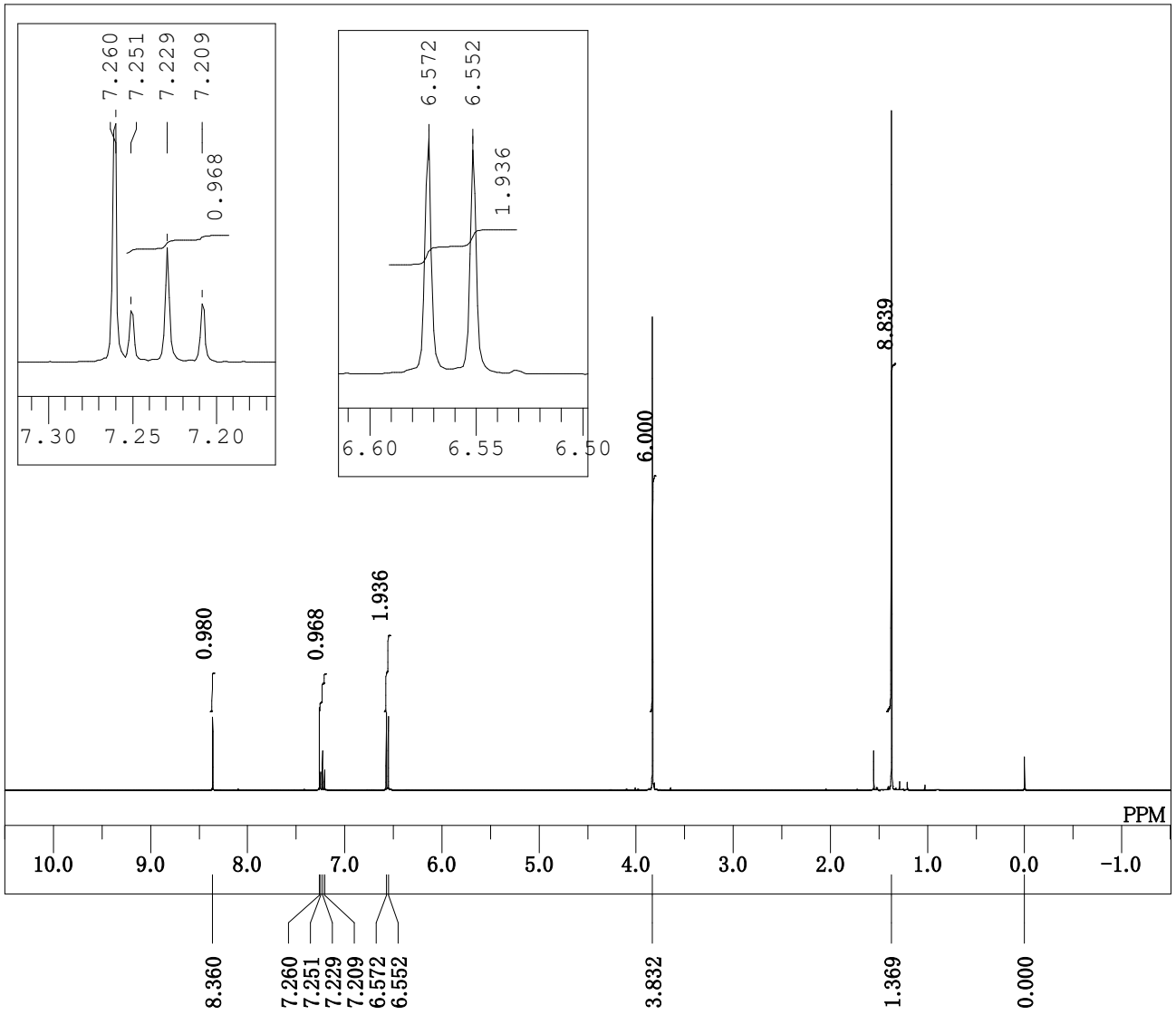
$^1$H NMR (395.88 MHz, Solvent=$CDCl_3$)

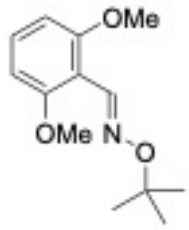



**1j**

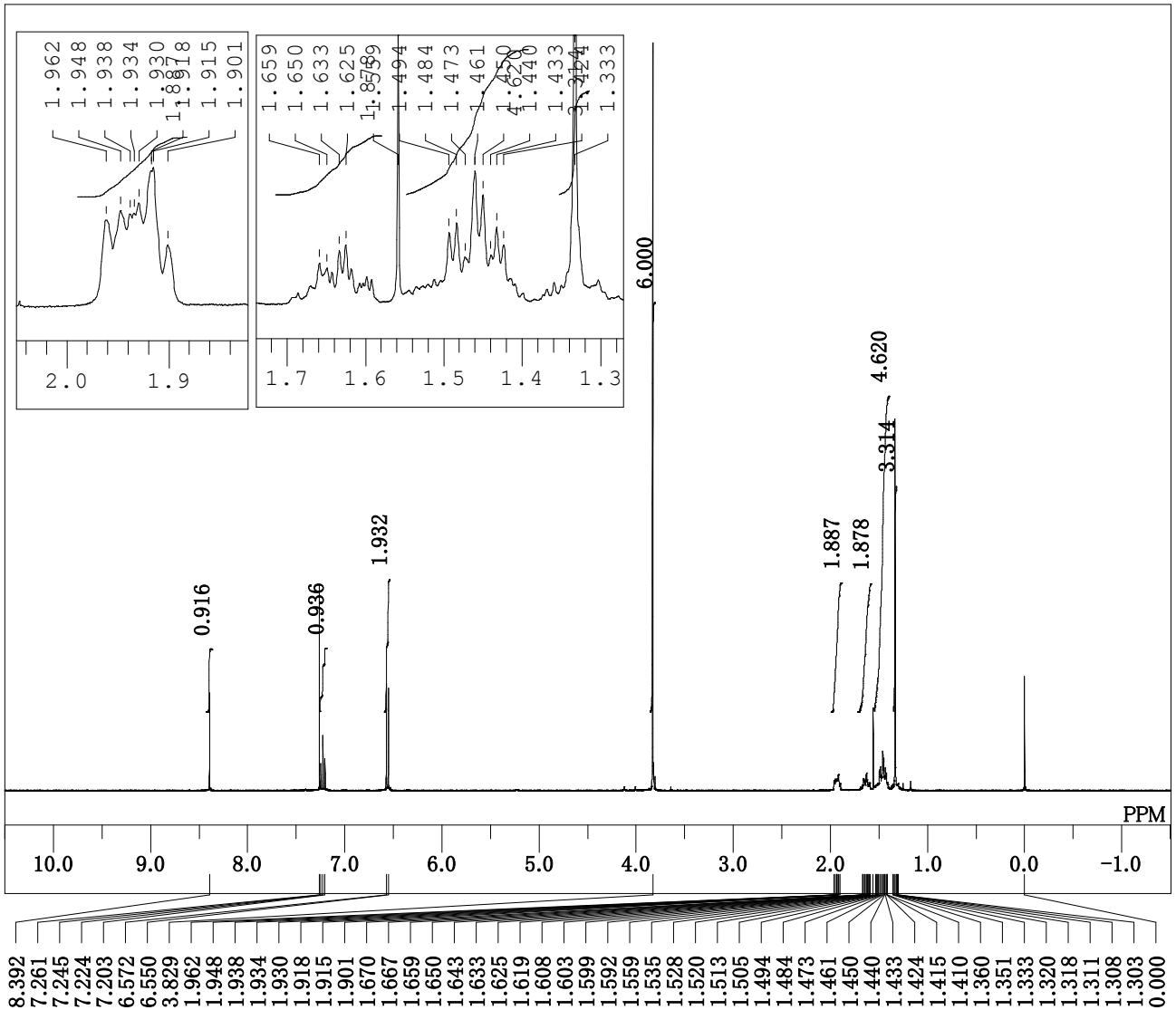
$^1$H NMR (395.88 MHz, Solvent=$CDCl_3$)

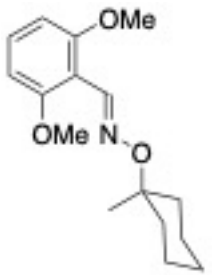



**1k**

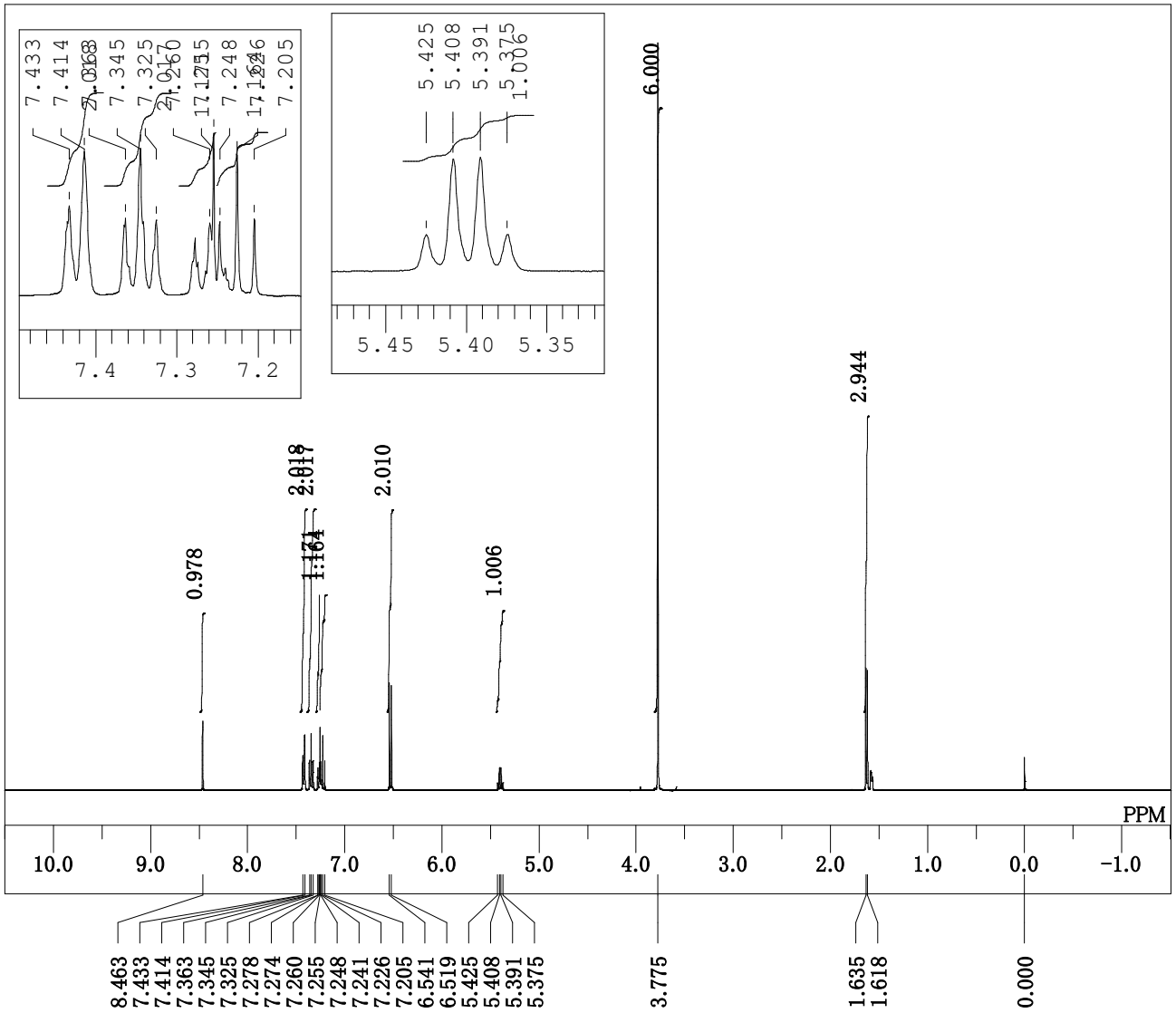
$^1$H NMR (391.78 MHz, Solvent=$CDCl_3$)

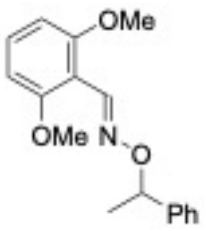



**1l**

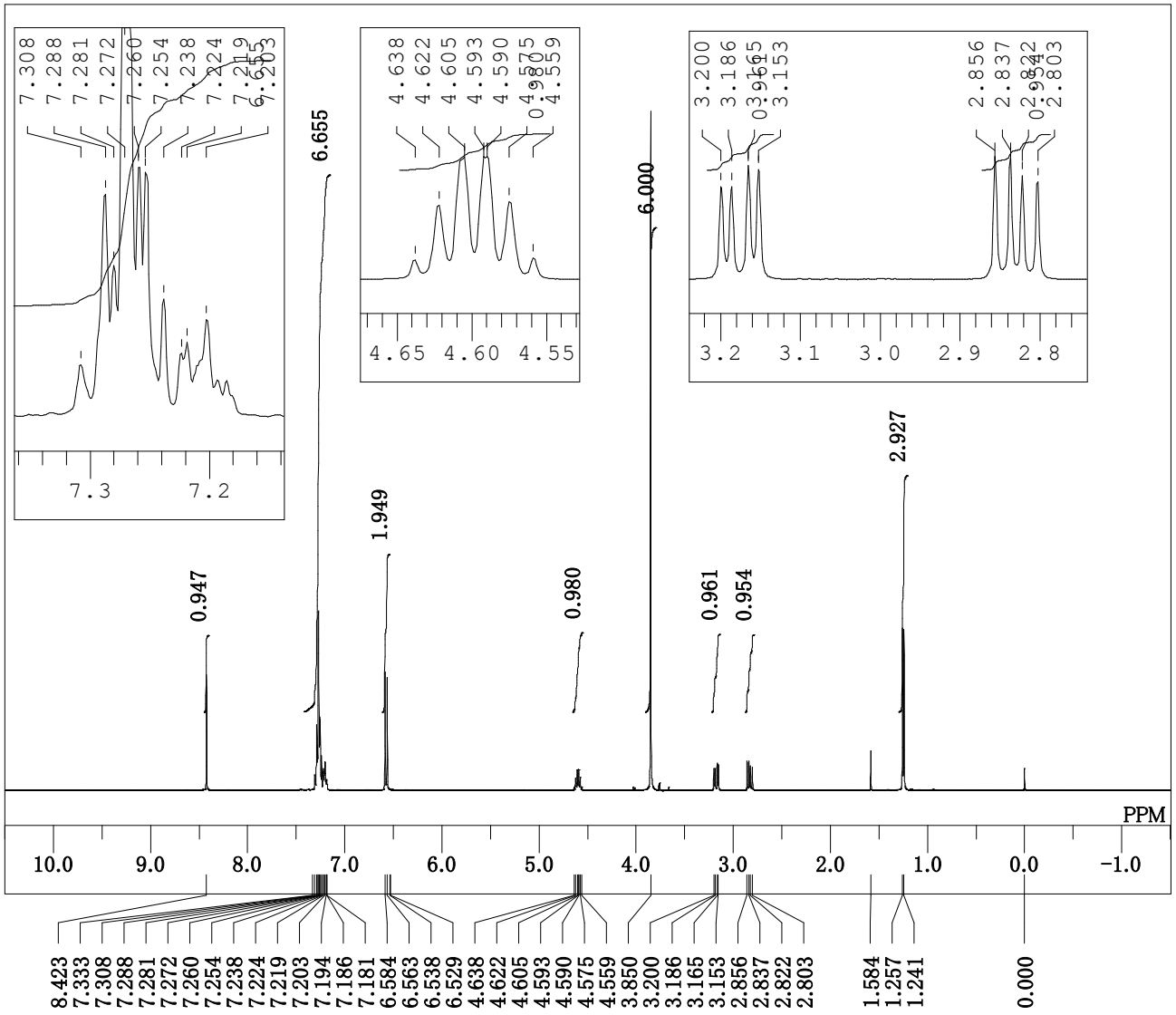
$^1$H NMR (395.88 MHz, Solvent=$CDCl_3$)

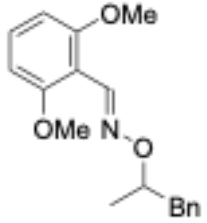



**1m**

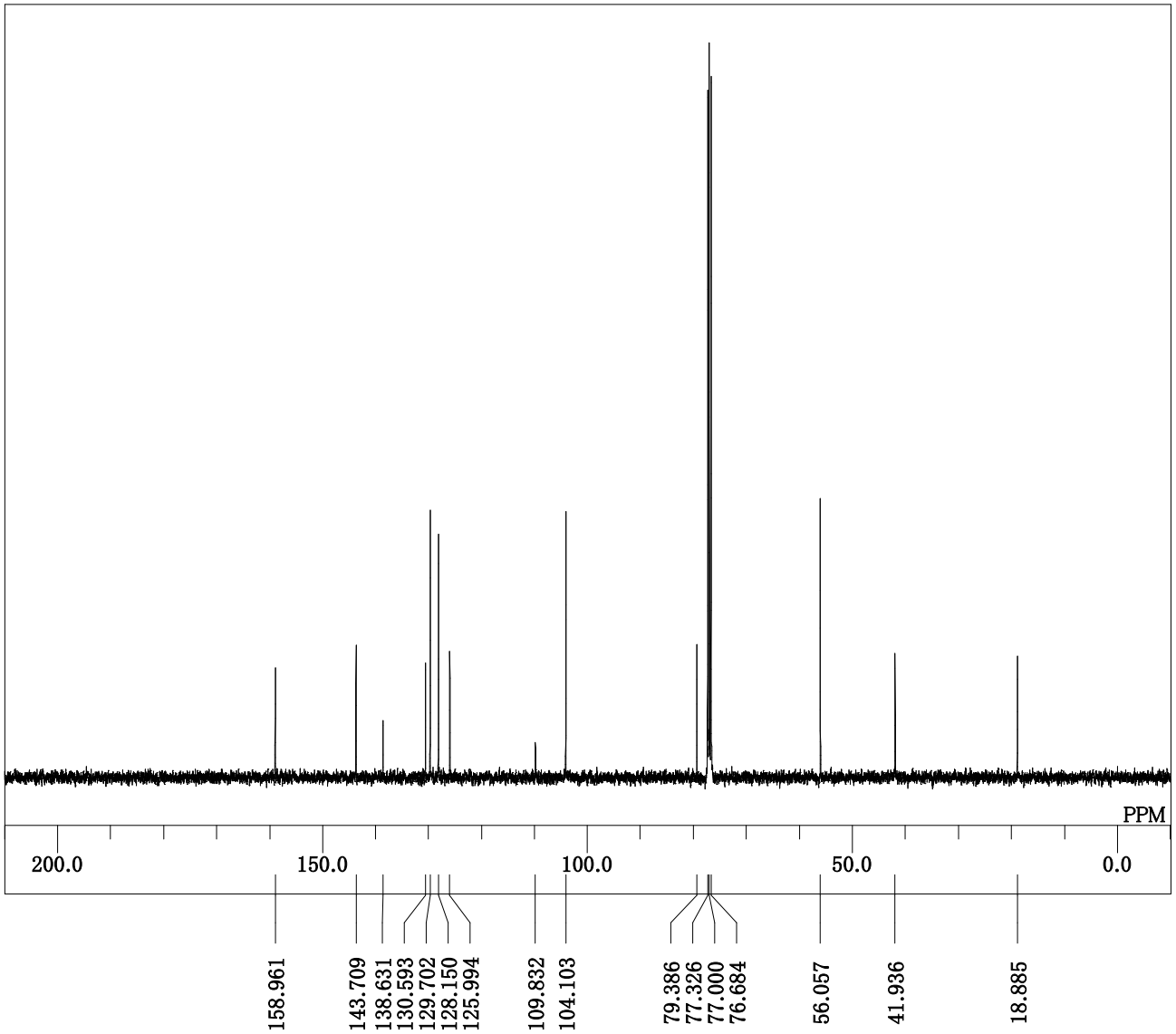
$^1$H NMR (395.88 MHz, Solvent=$CDCl_3$)

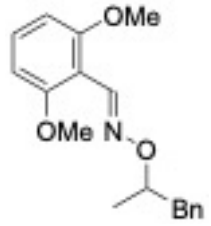



**1n**

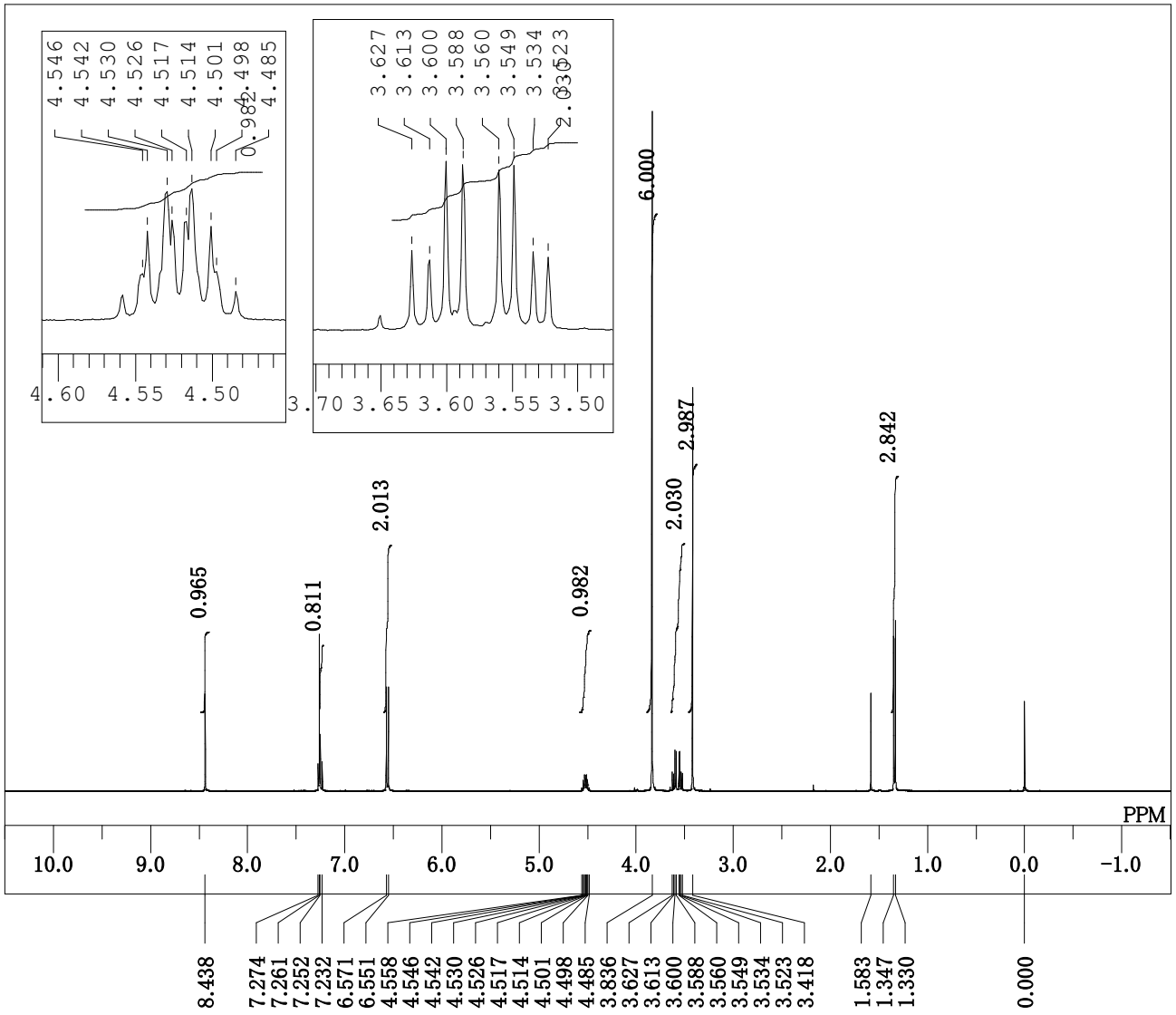
$^{13}$C NMR (99.55 MHz, Solvent=$CDCl_3$)

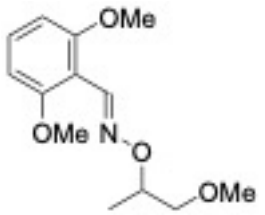



**1n**

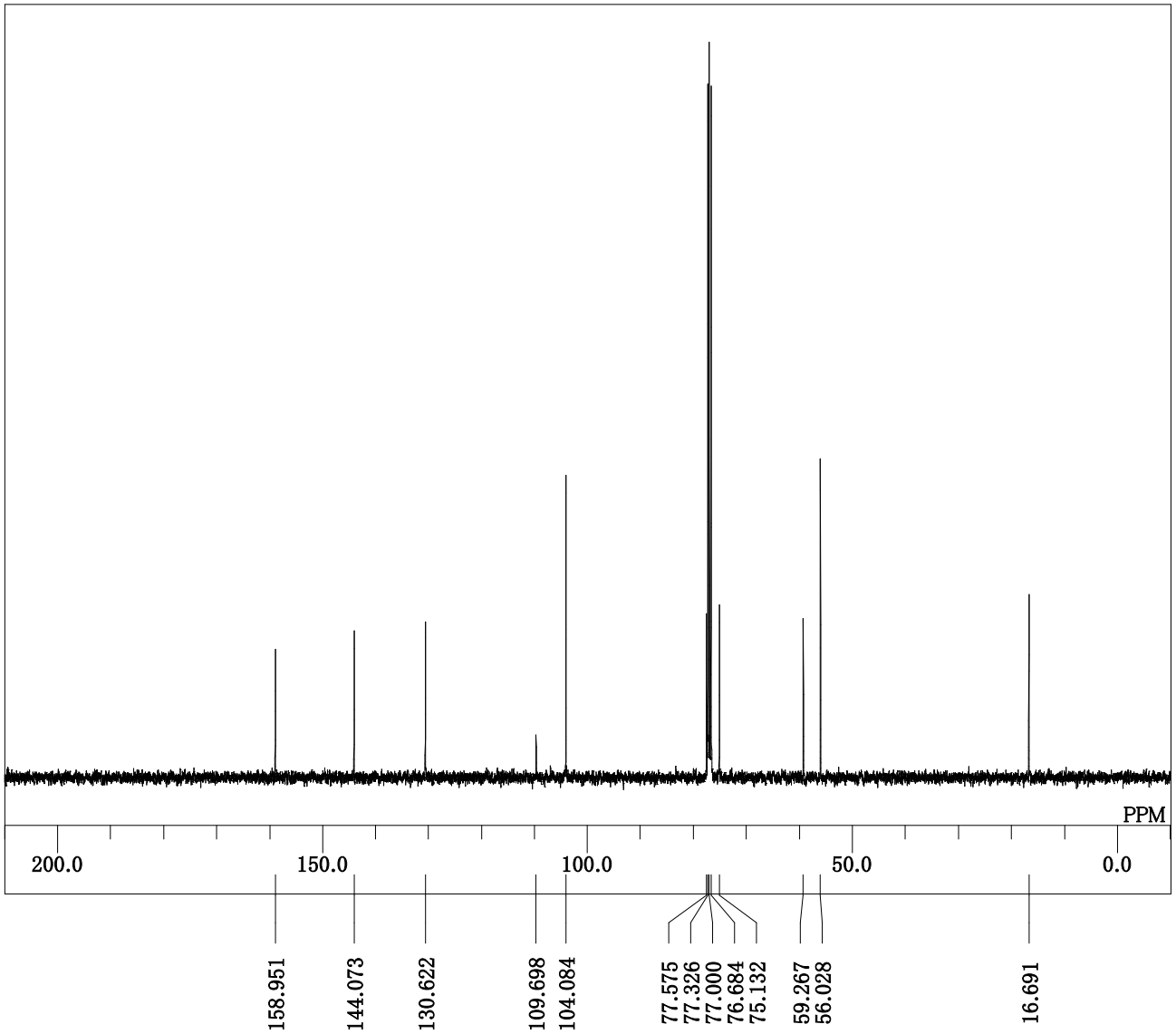
$^1$H NMR (395.88 MHz, Solvent=$CDCl_3$)

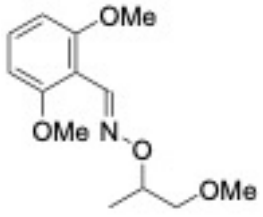



**1o**

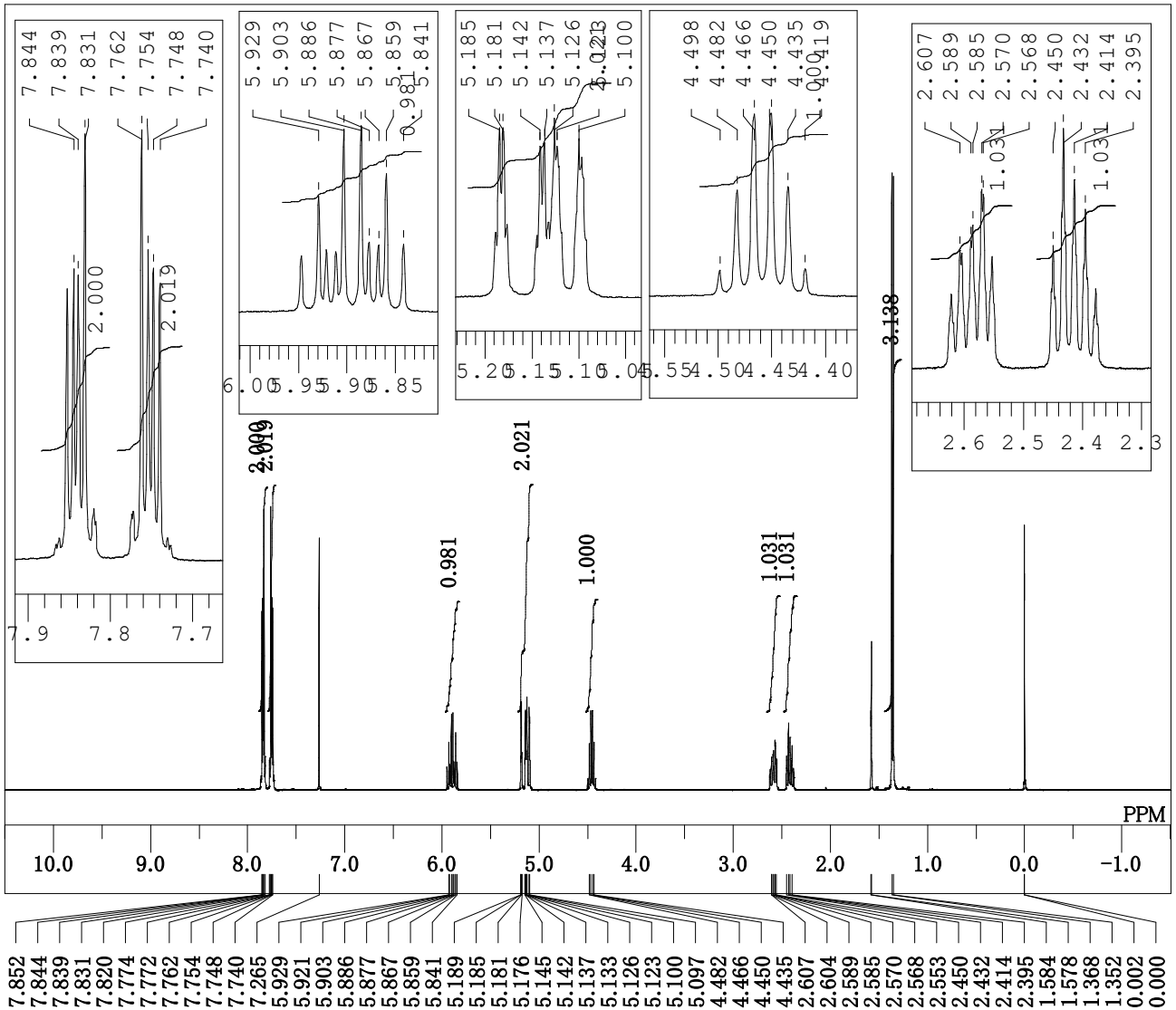
$^{13}$C NMR (99.55 MHz, Solvent=$CDCl_3$)

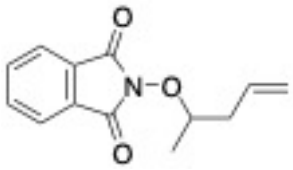



**1o**

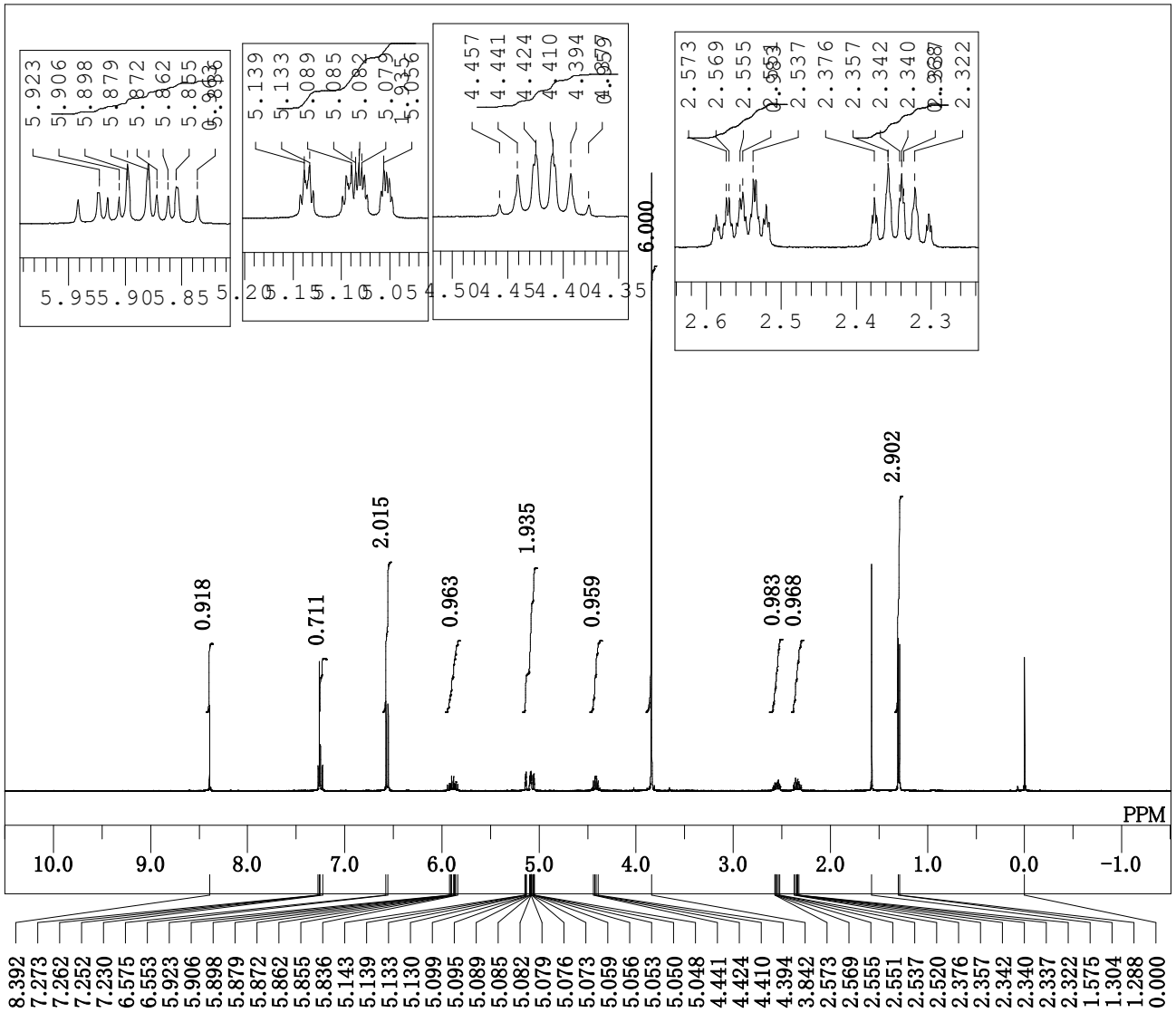
$^1$H NMR (395.78 MHz, Solvent=$CDCl_3$)

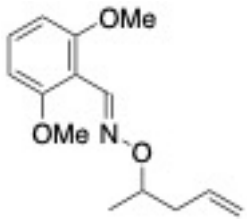




**S1p**

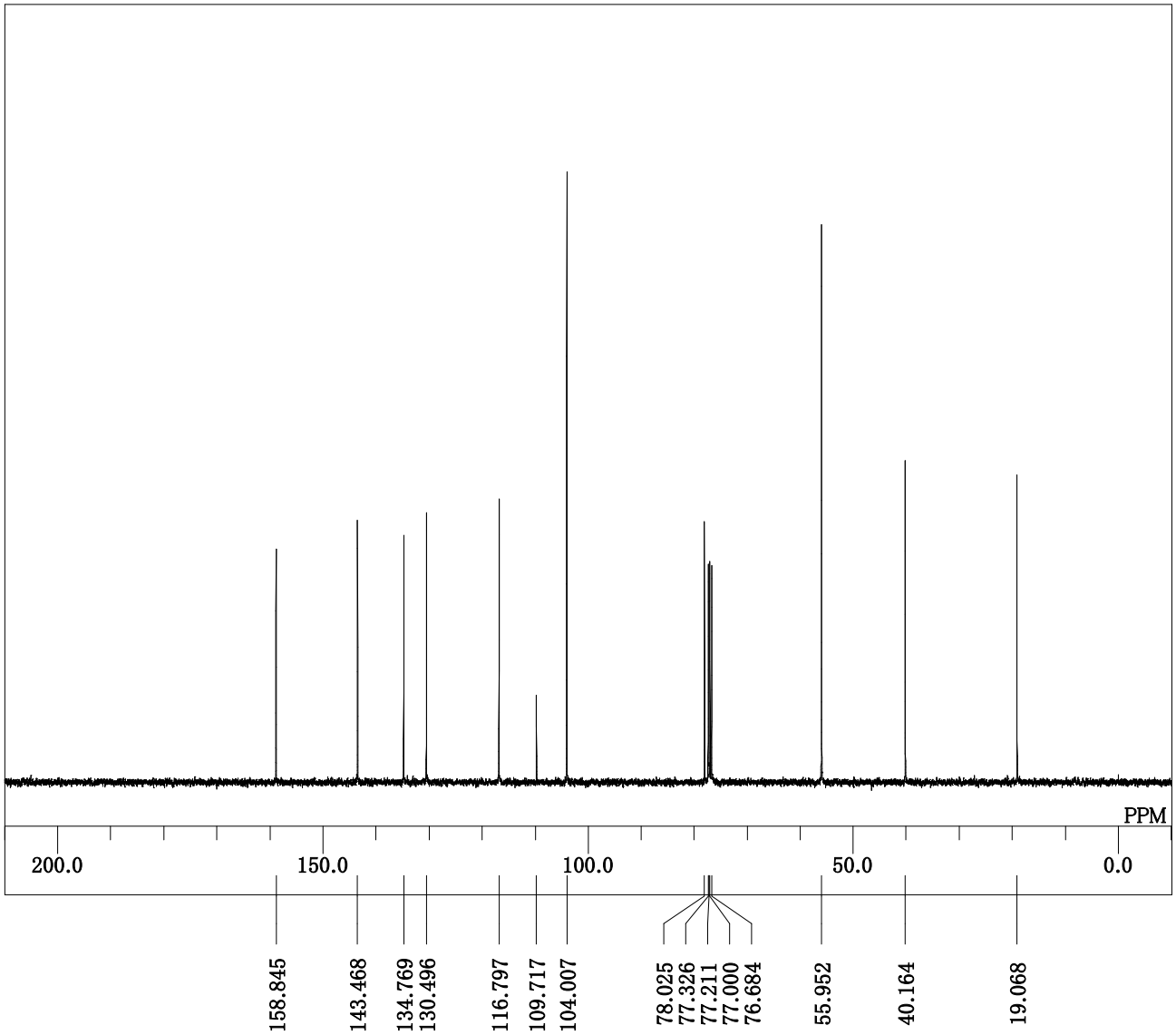
$^1$H NMR (391.78 MHz, Solvent=$CDCl_3$)

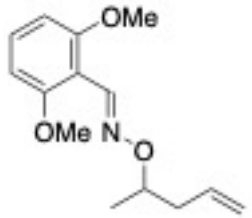



**1p**

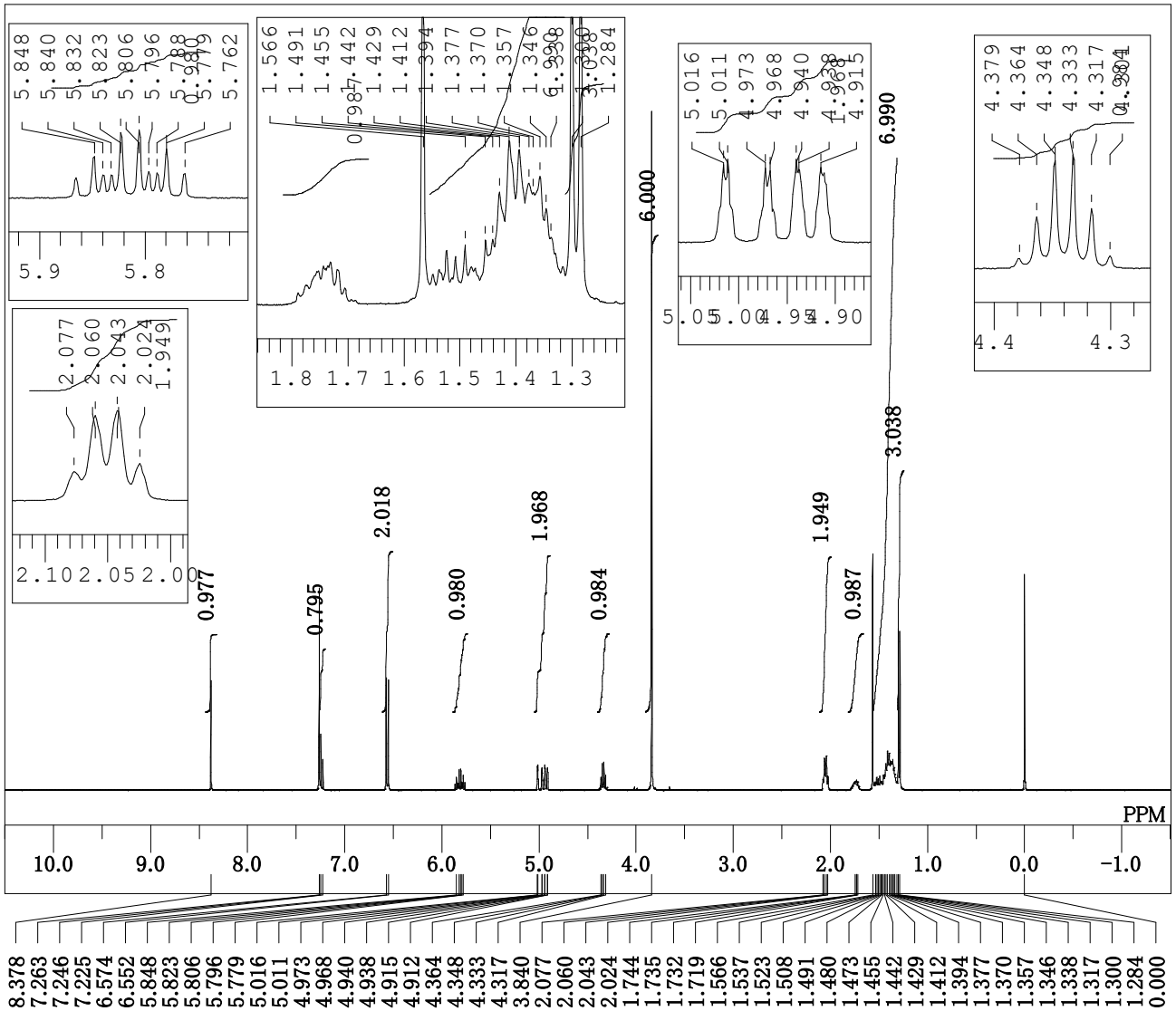
$^{13}C$ NMR (391.78 MHz, Solvent=$CDCl_3$)

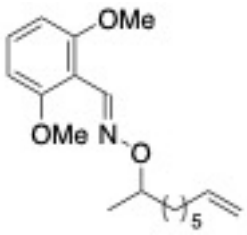



**1p**

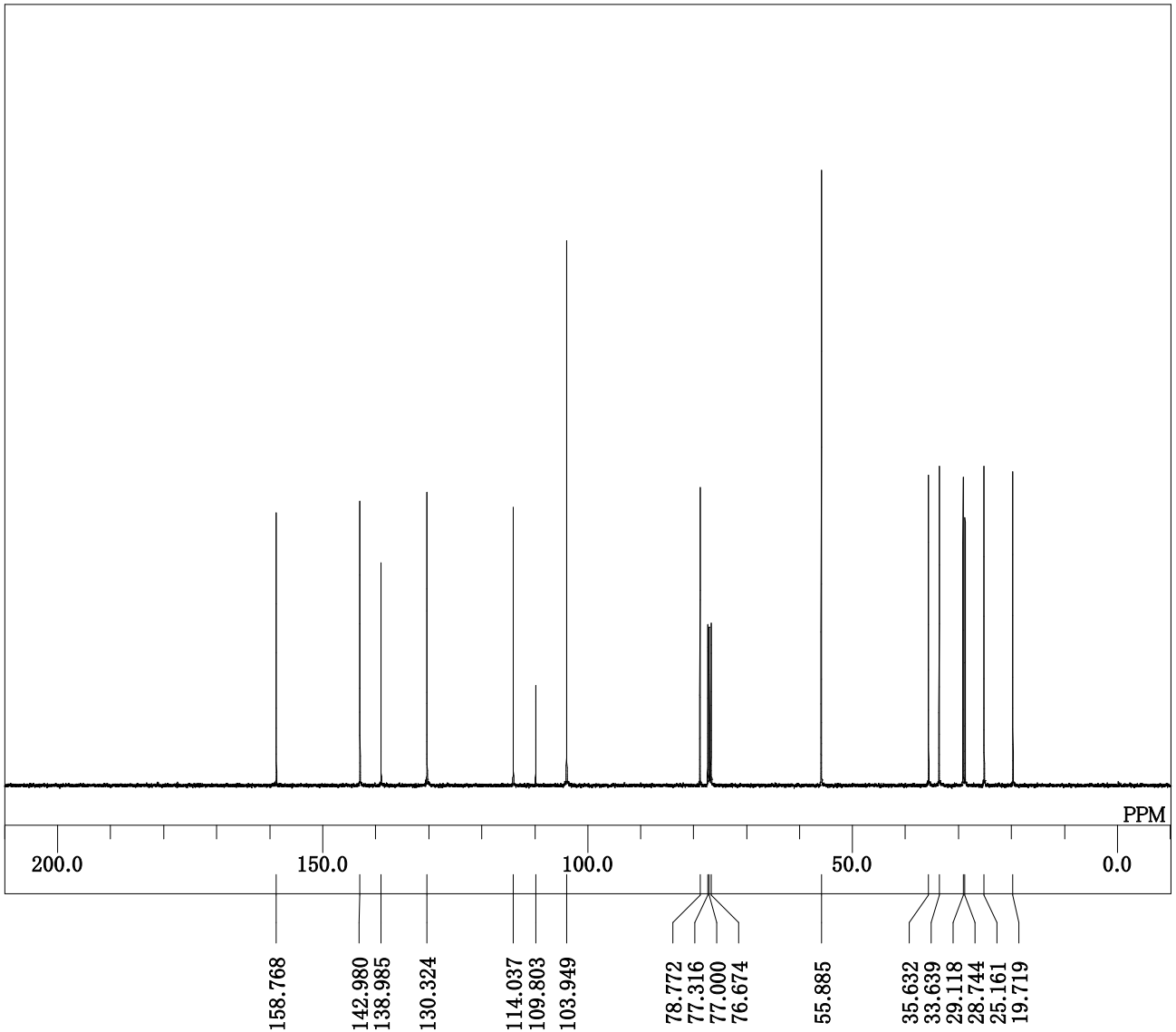
$^1$H NMR (395.88 MHz, Solvent=$CDCl_3$)

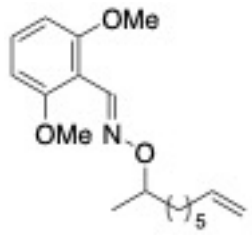



**1q**

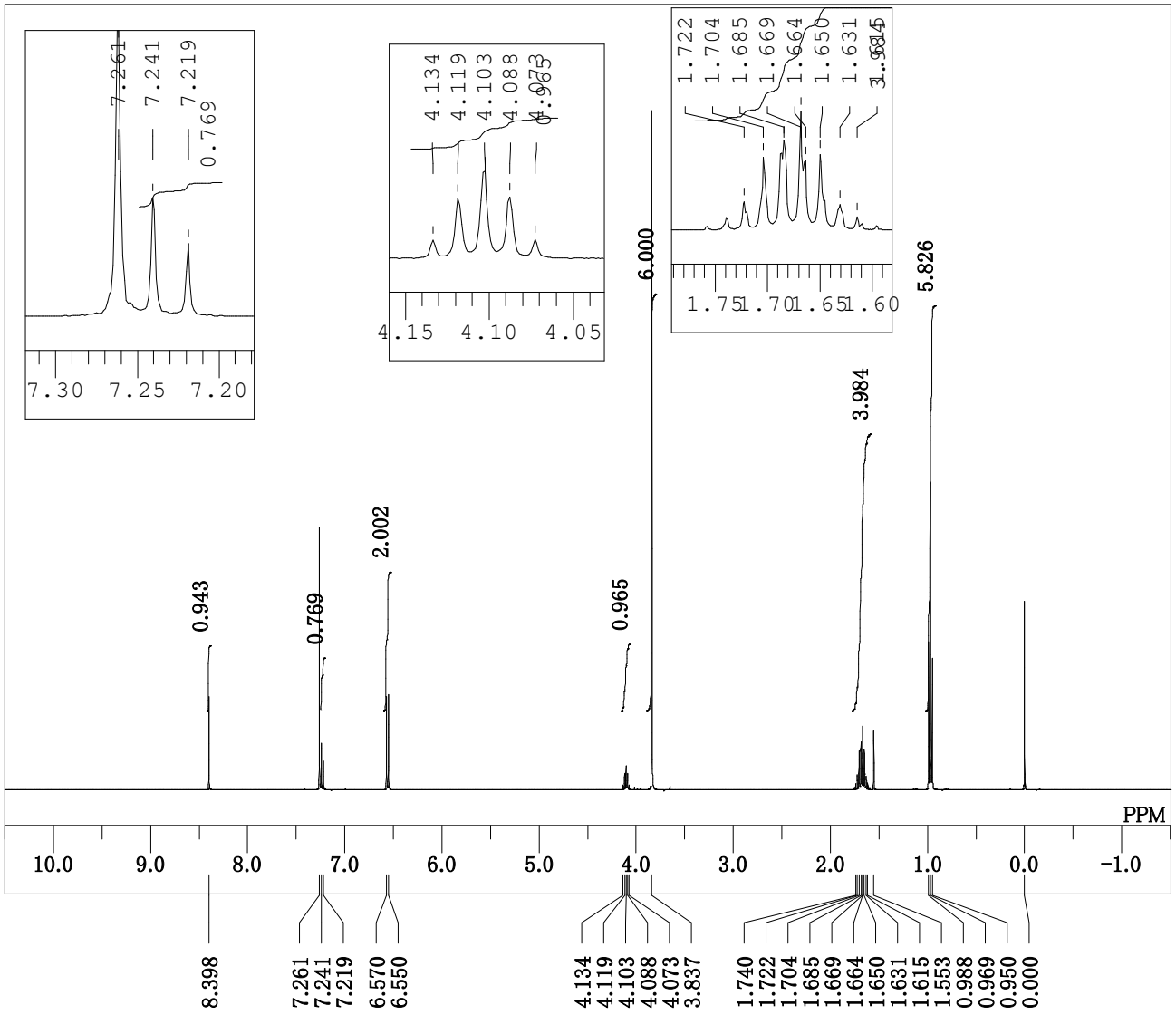
$^1$H NMR (395.88 MHz, Solvent=$CDCl_3$)

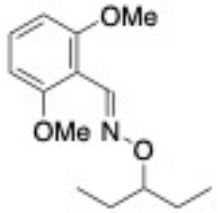



**1q**

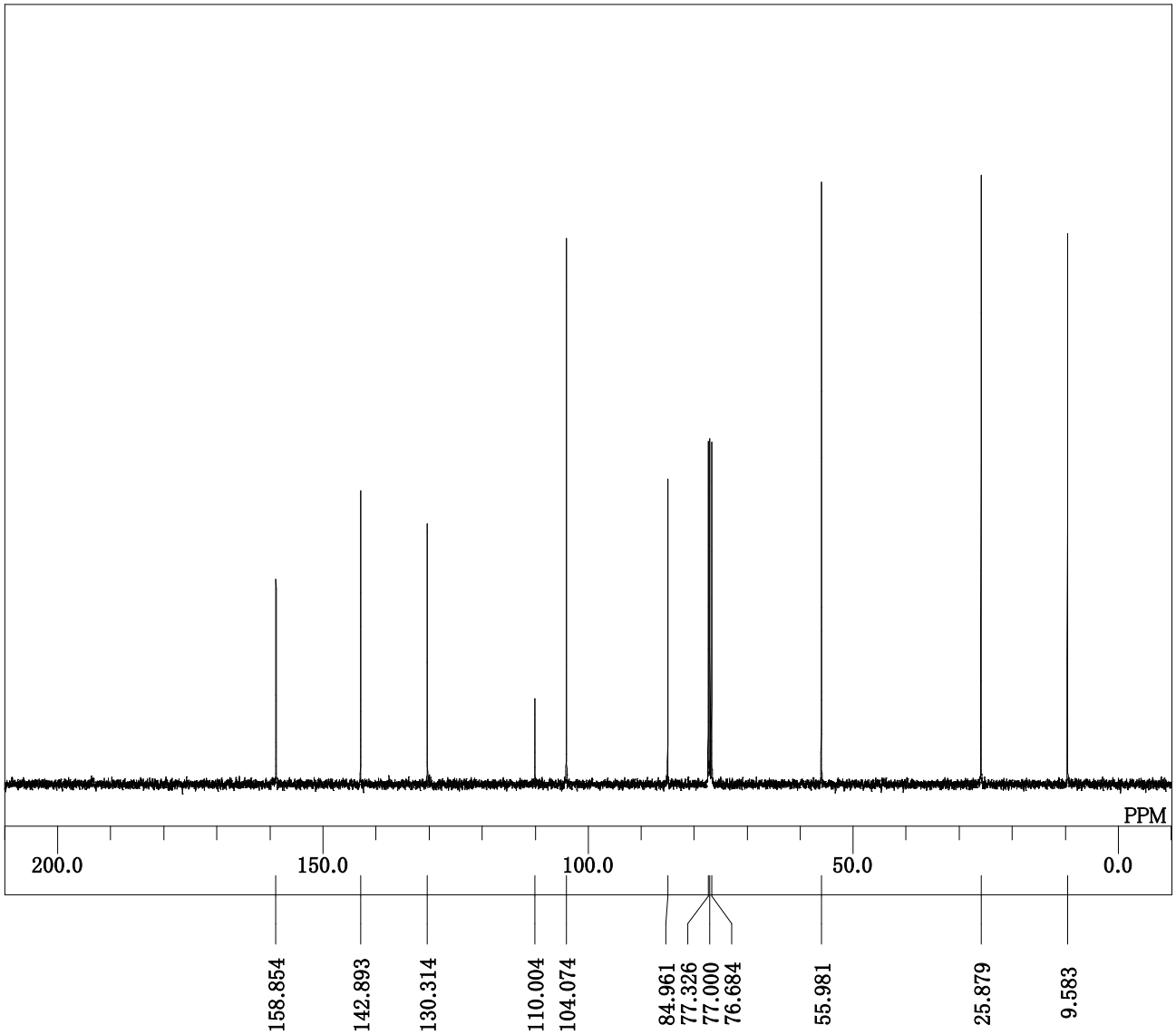
$^1$H NMR (395.88 MHz, Solvent=$CDCl_3$)

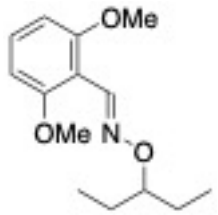



**1r**

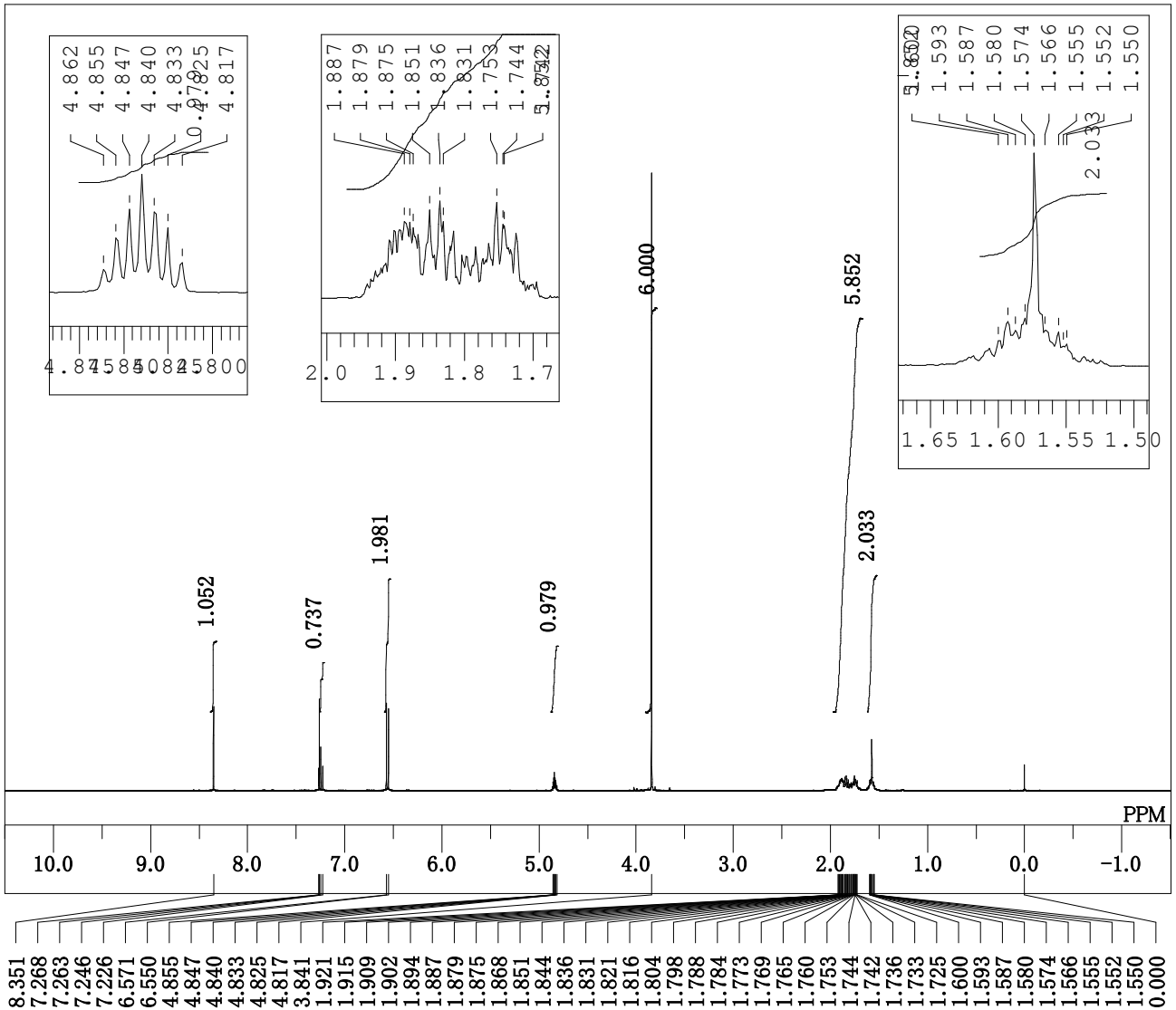
$^{13}C$ NMR (99.55 MHz, Solvent=$CDCl_3$)

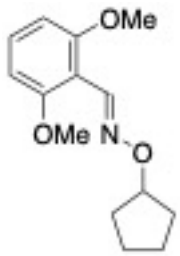



**1r**

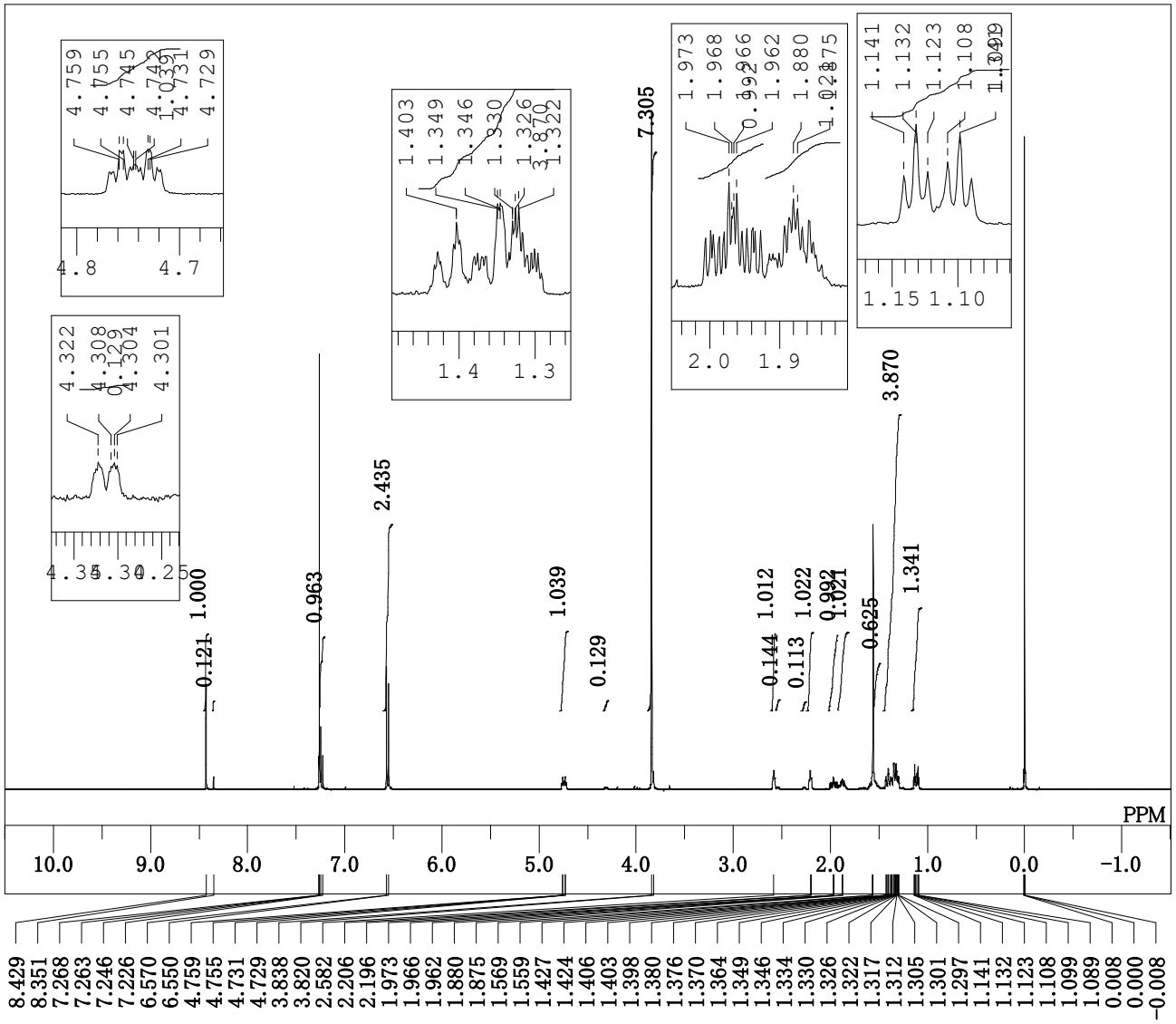
$^1$H NMR (395.88 MHz, Solvent=$CDCl_3$)

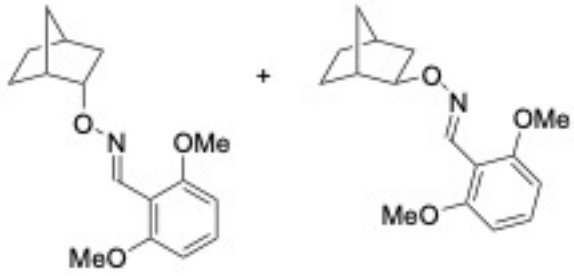



**1s**

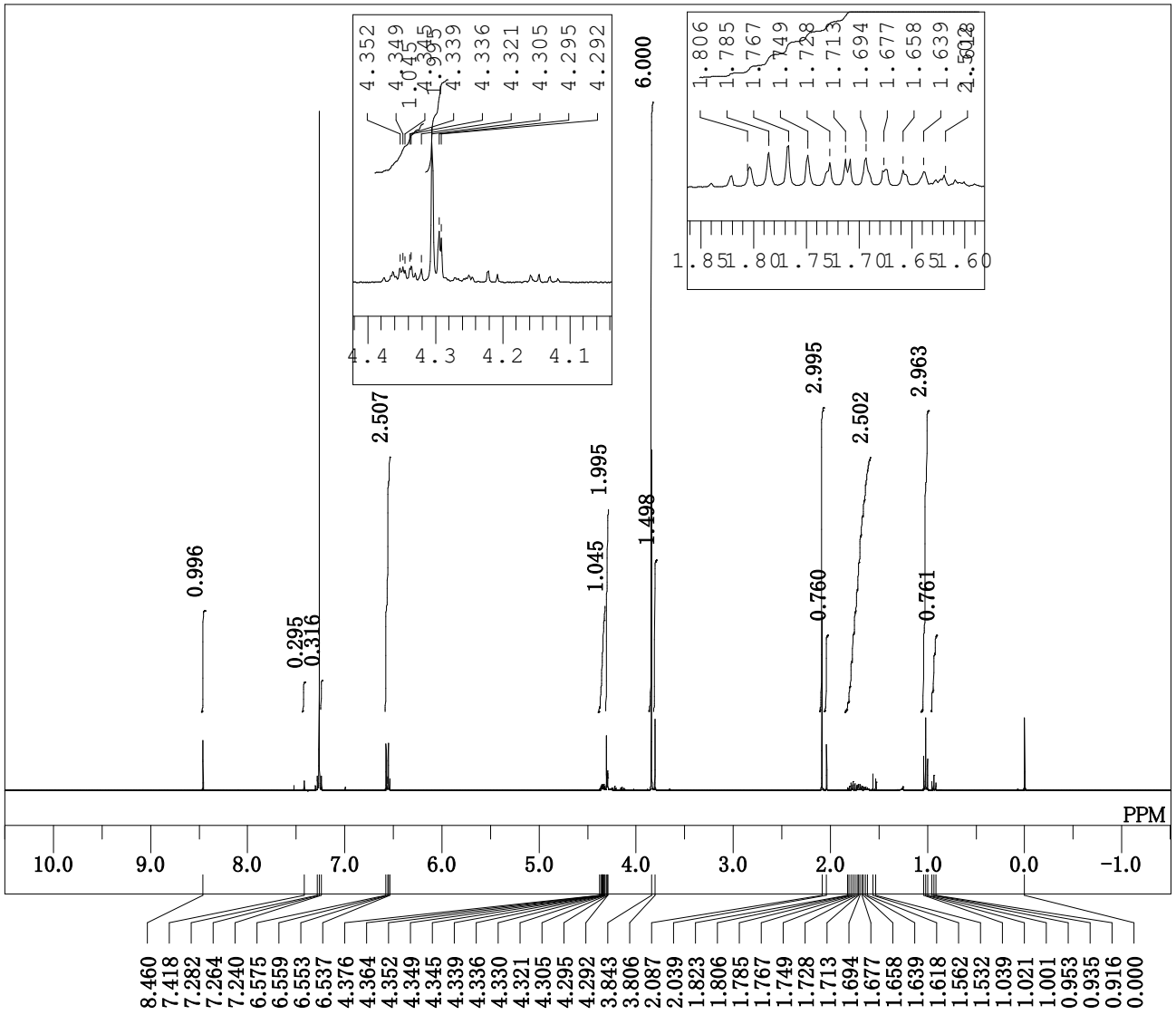
$^1$H NMR (395.88 MHz, Solvent=$CDCl_3$)

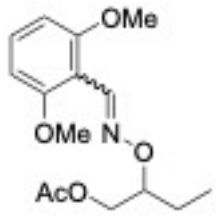



**1t**

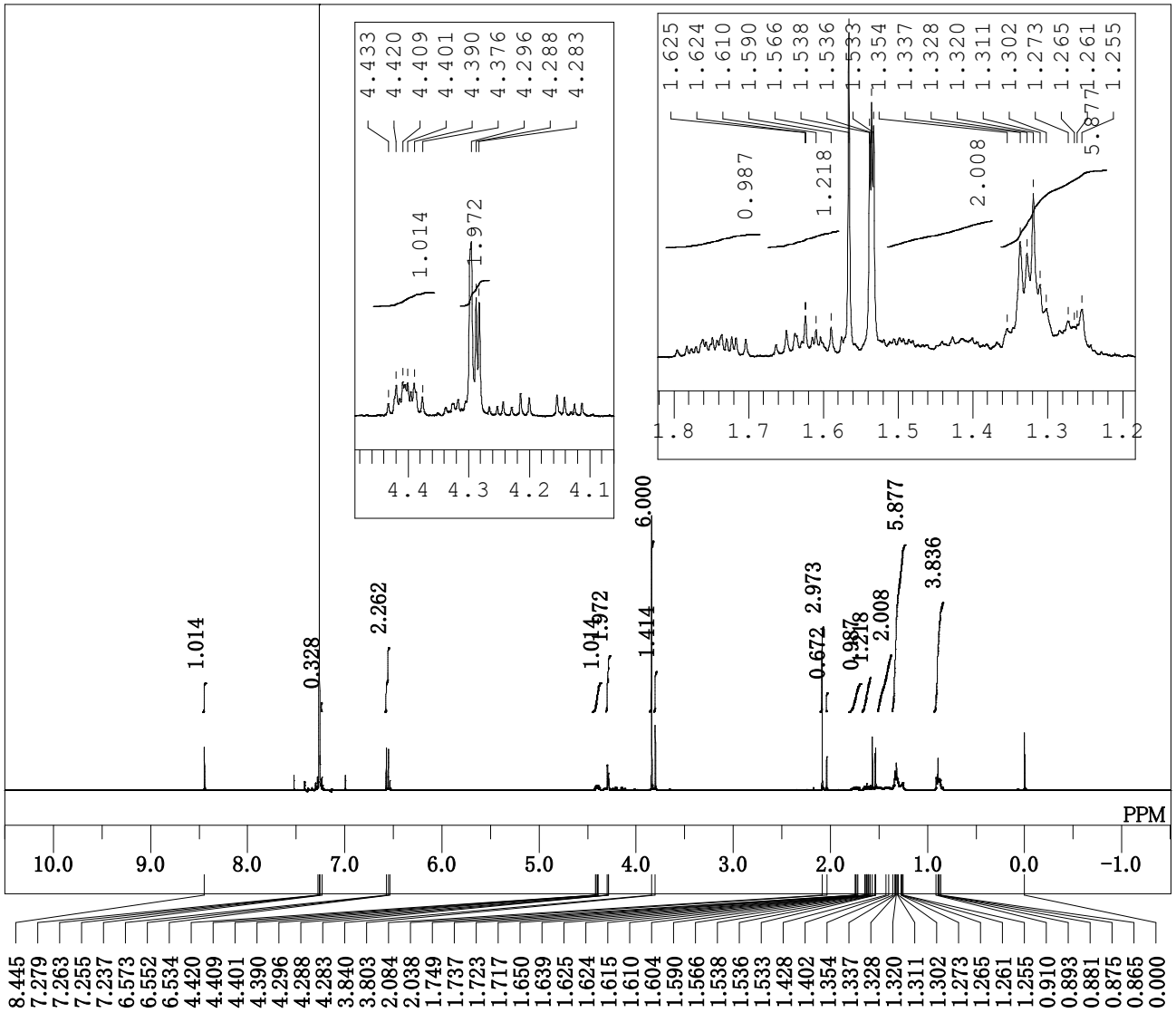
$^1$H NMR (395.88 MHz, Solvent=$CDCl_3$)

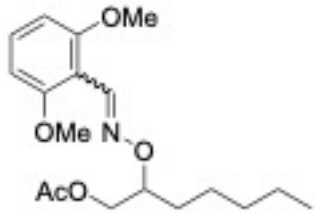



**2a**

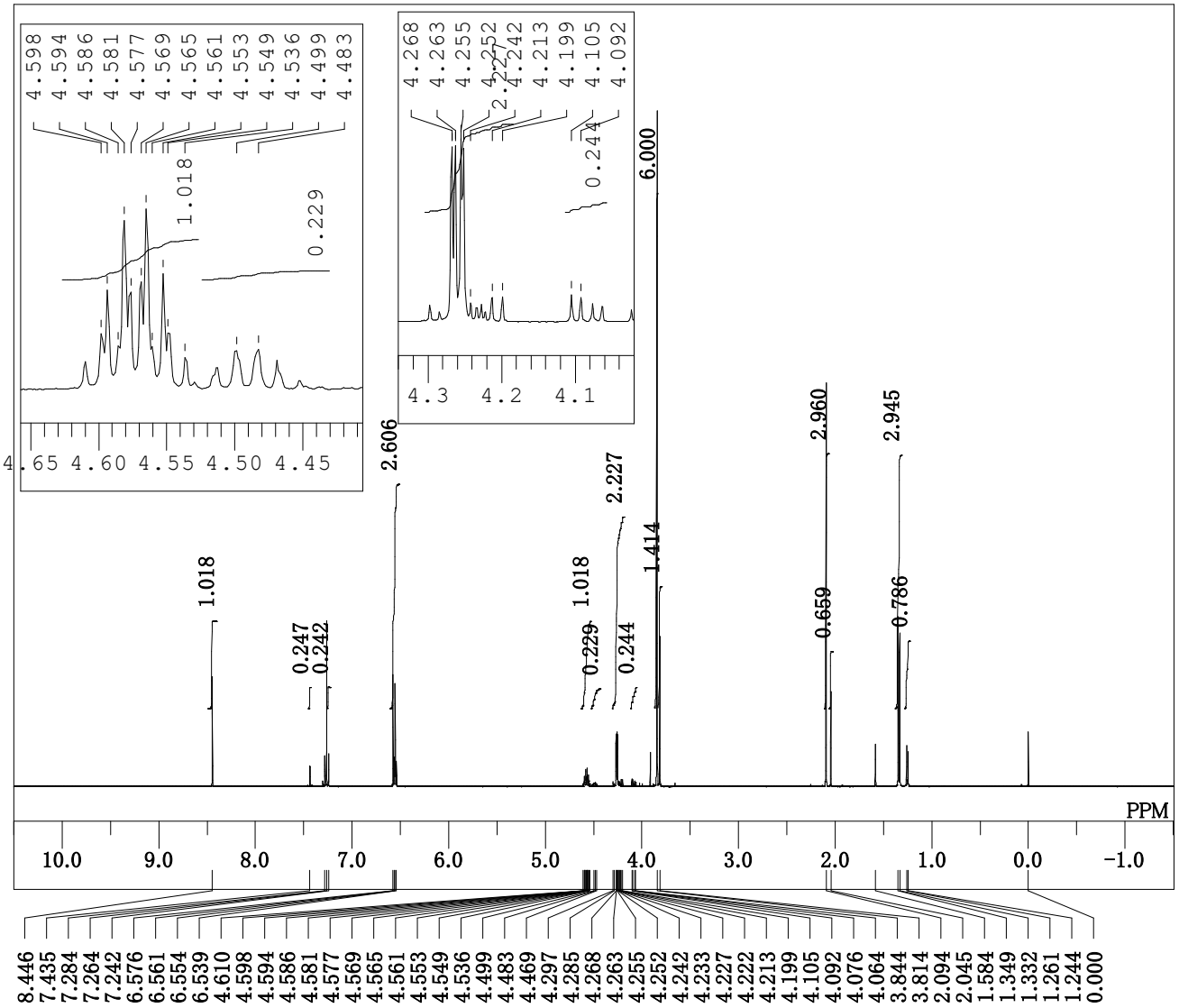
$^1$H NMR (395.88 MHz, Solvent=$CDCl_3$)

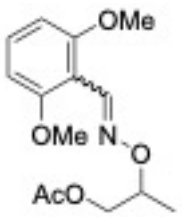



**2e**

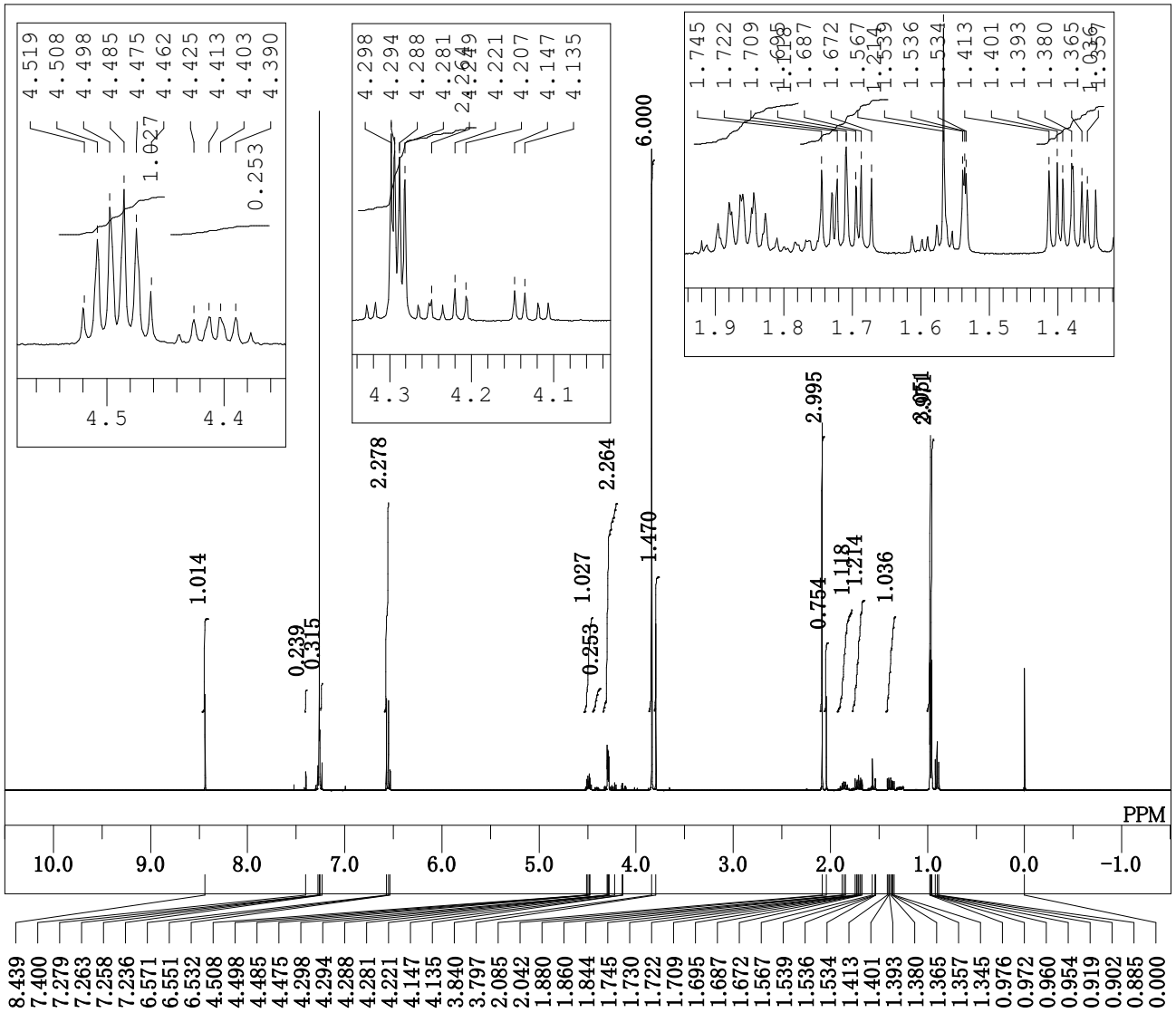
$^1$H NMR (395.88 MHz, Solvent=$CDCl_3$)

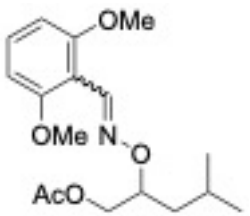



**2f**

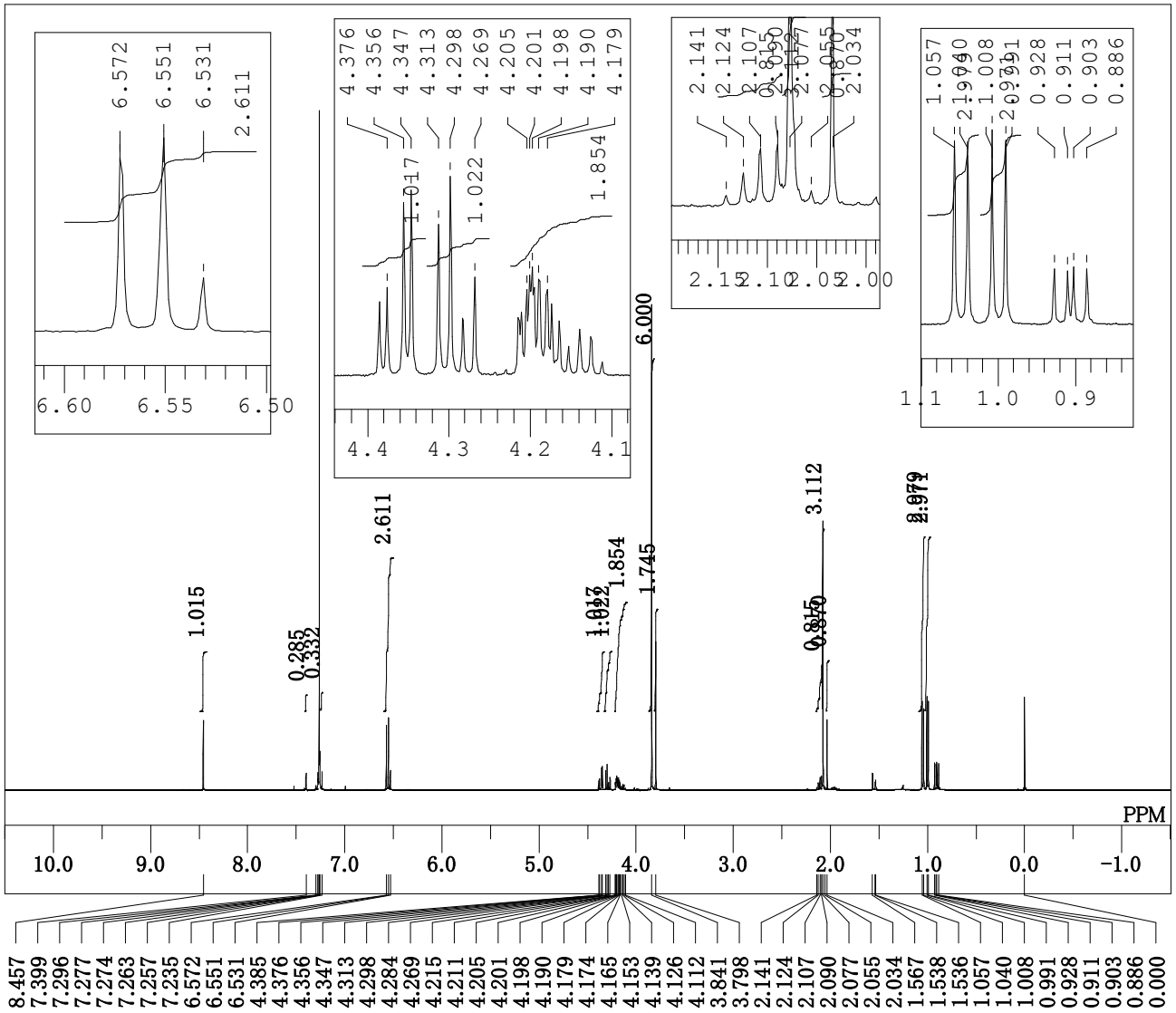
$^1$H NMR (395.88 MHz, Solvent=$CDCl_3$)

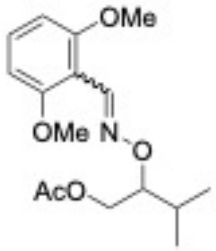





**2g**

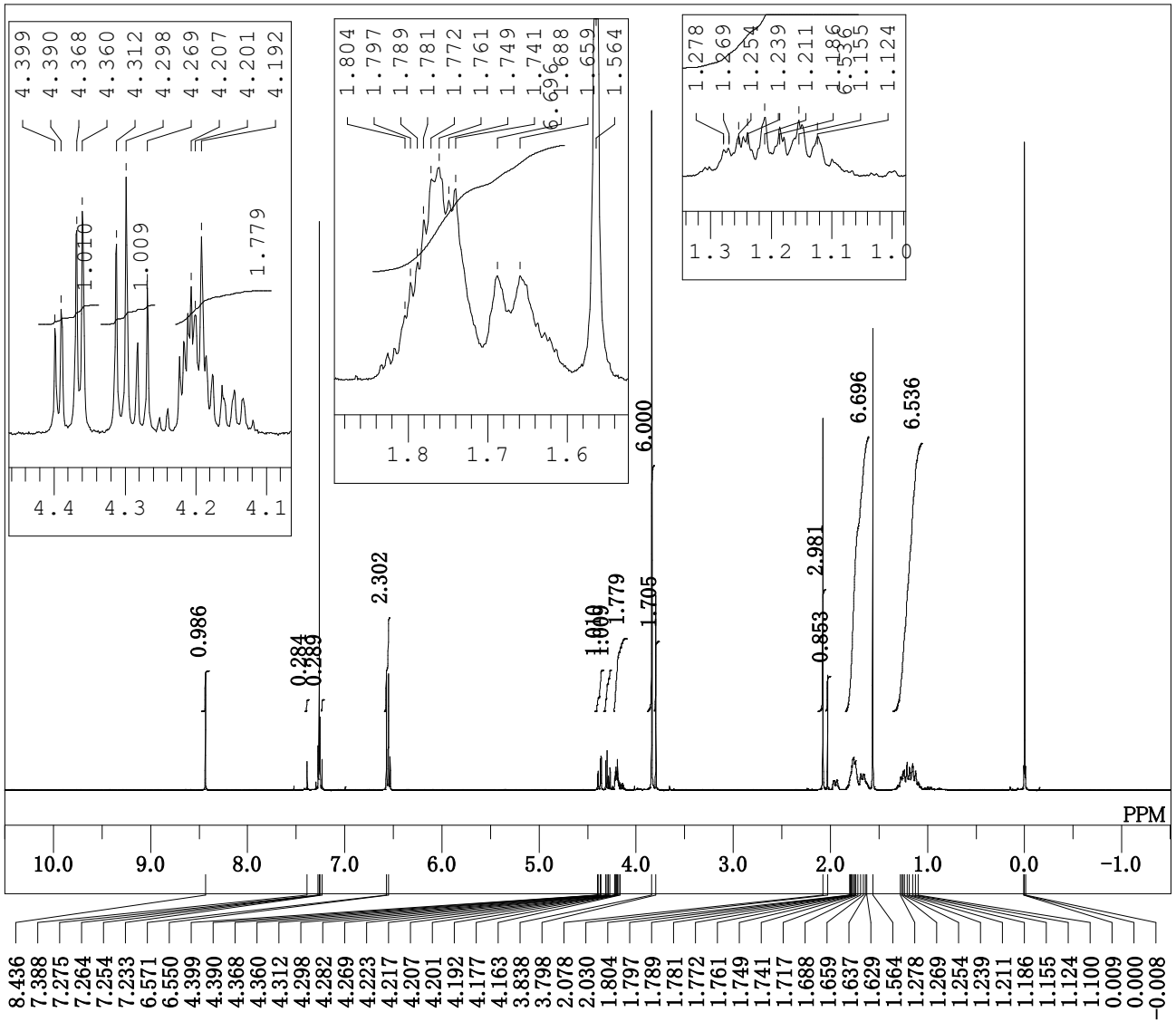
$^1$H NMR (395.88 MHz, Solvent=$CDCl_3$)

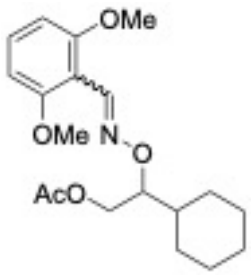





**2h**

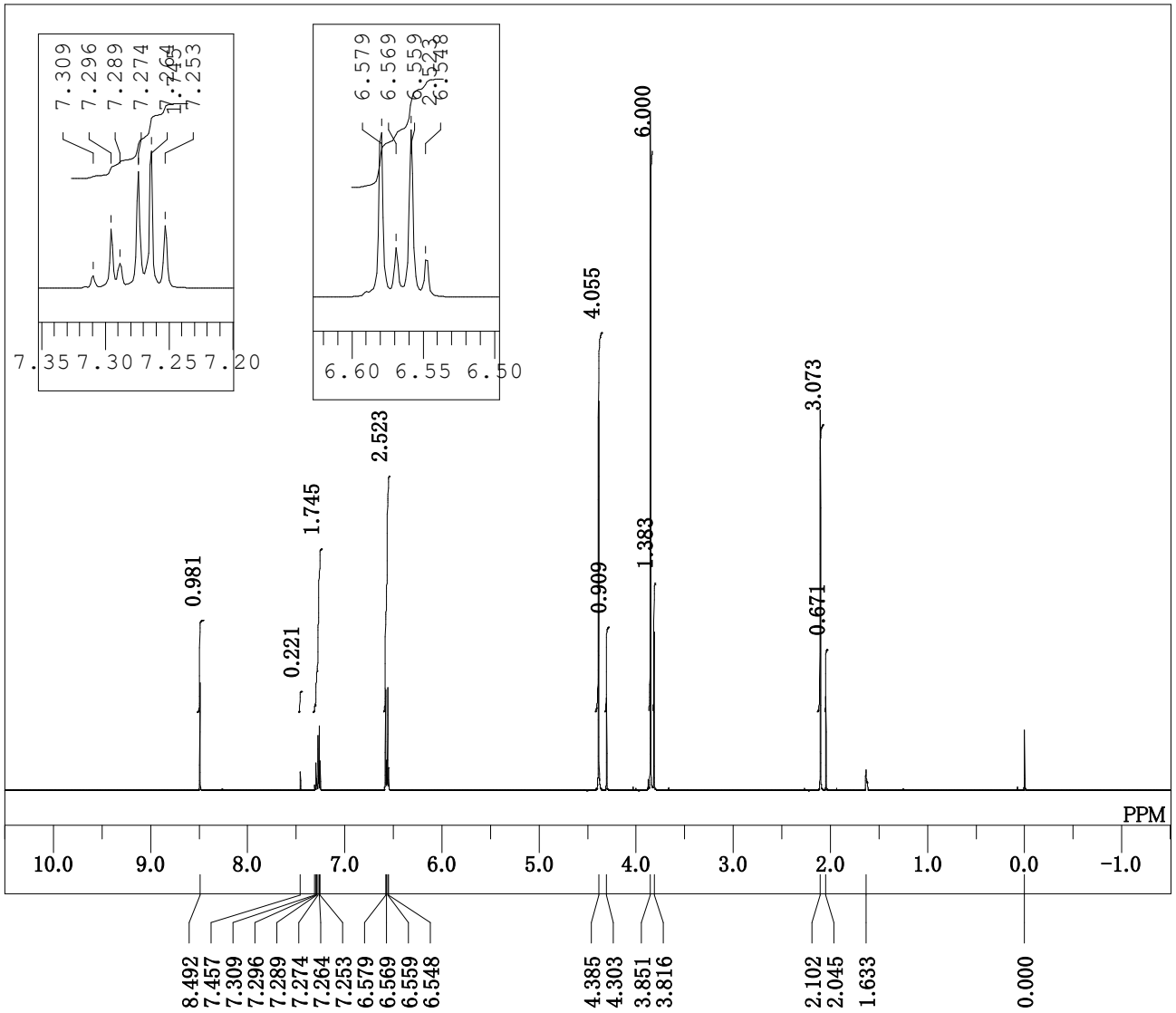
$^1$H NMR (395.88 MHz, Solvent=$CDCl_3$)

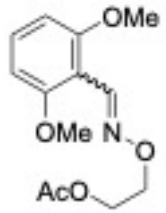





**2i**

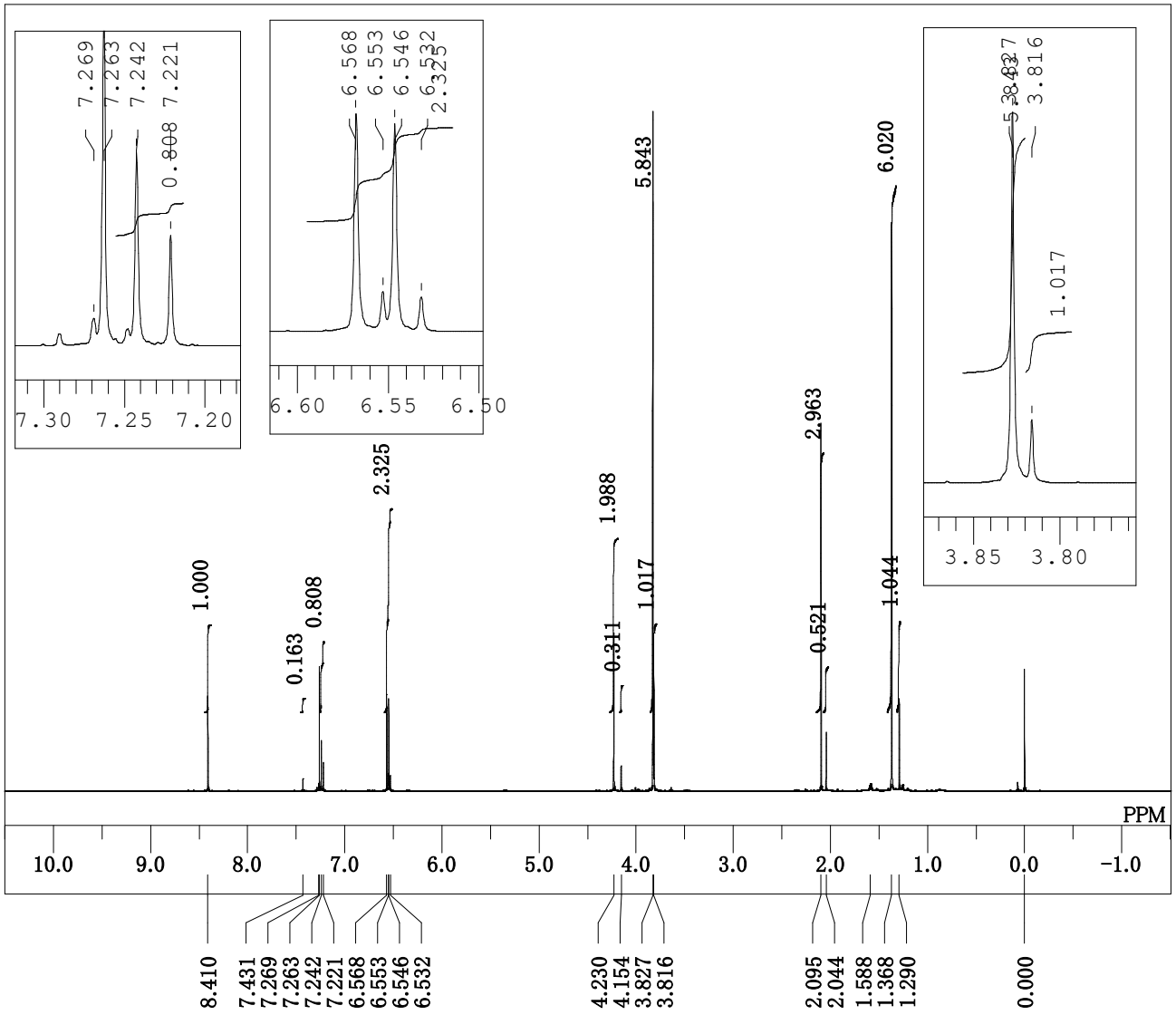
$^1$H NMR (395.88 MHz, Solvent=$CDCl_3$)

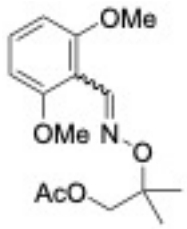



**2j**

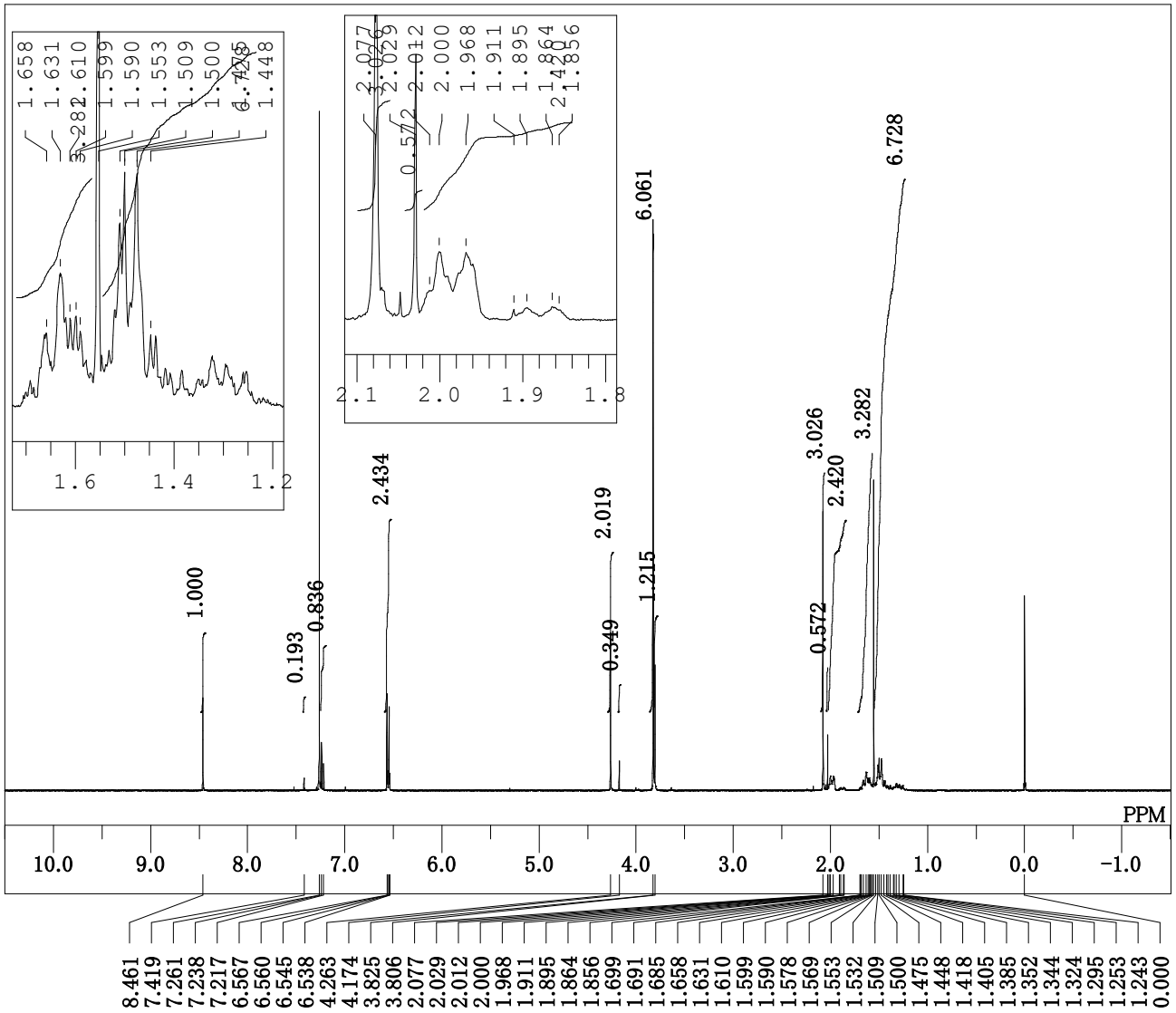
$^1$H NMR (395.88 MHz, Solvent=$CDCl_3$)

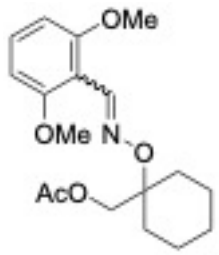



**2k**

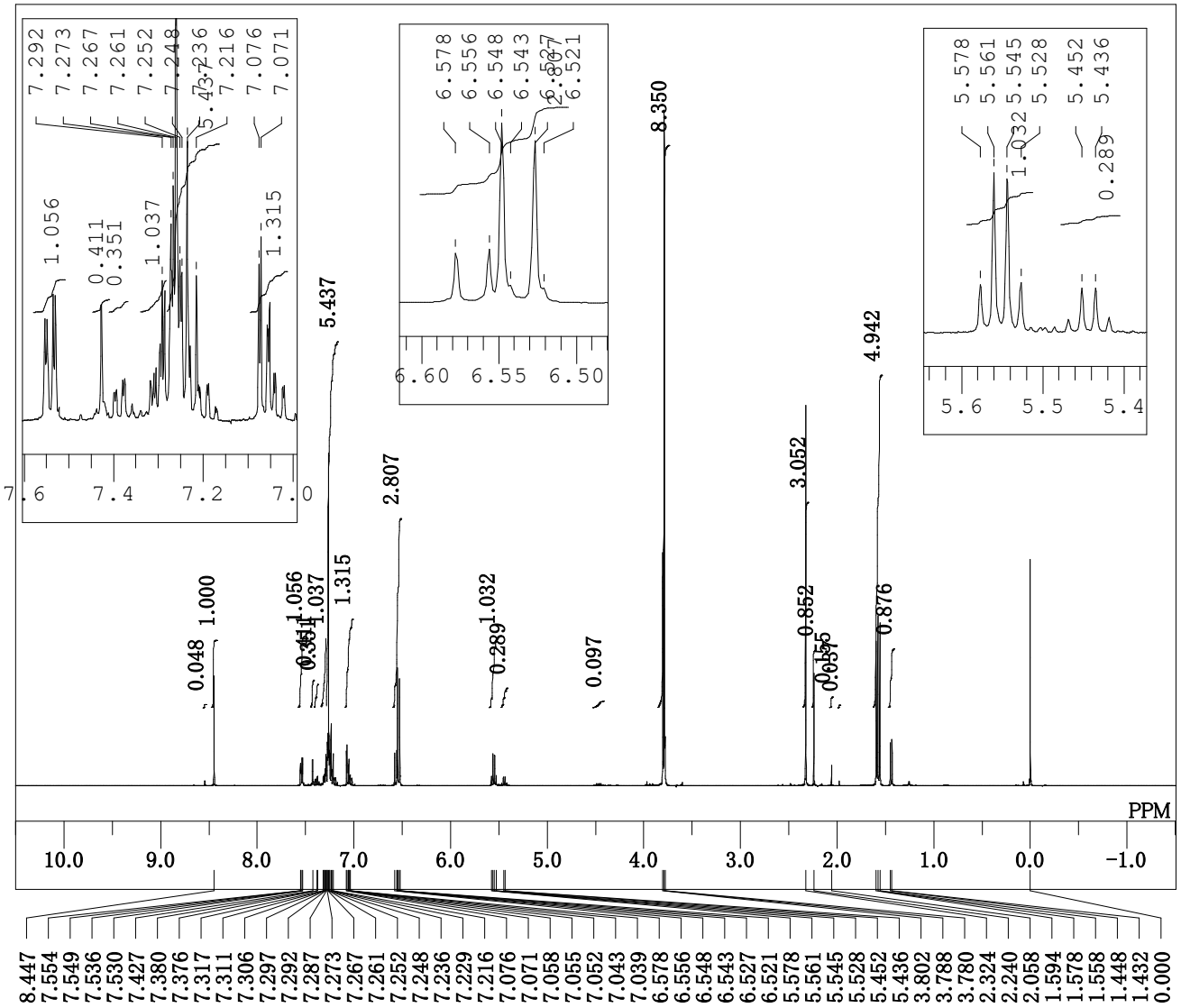
$^1$H NMR (395.88 MHz, Solvent=$CDCl_3$)

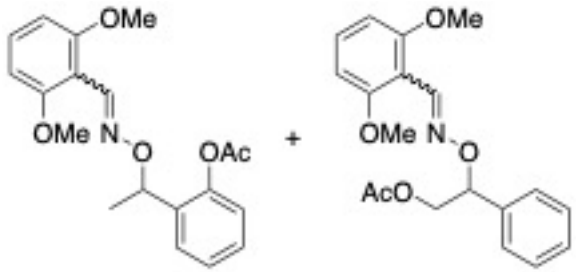



**2l**

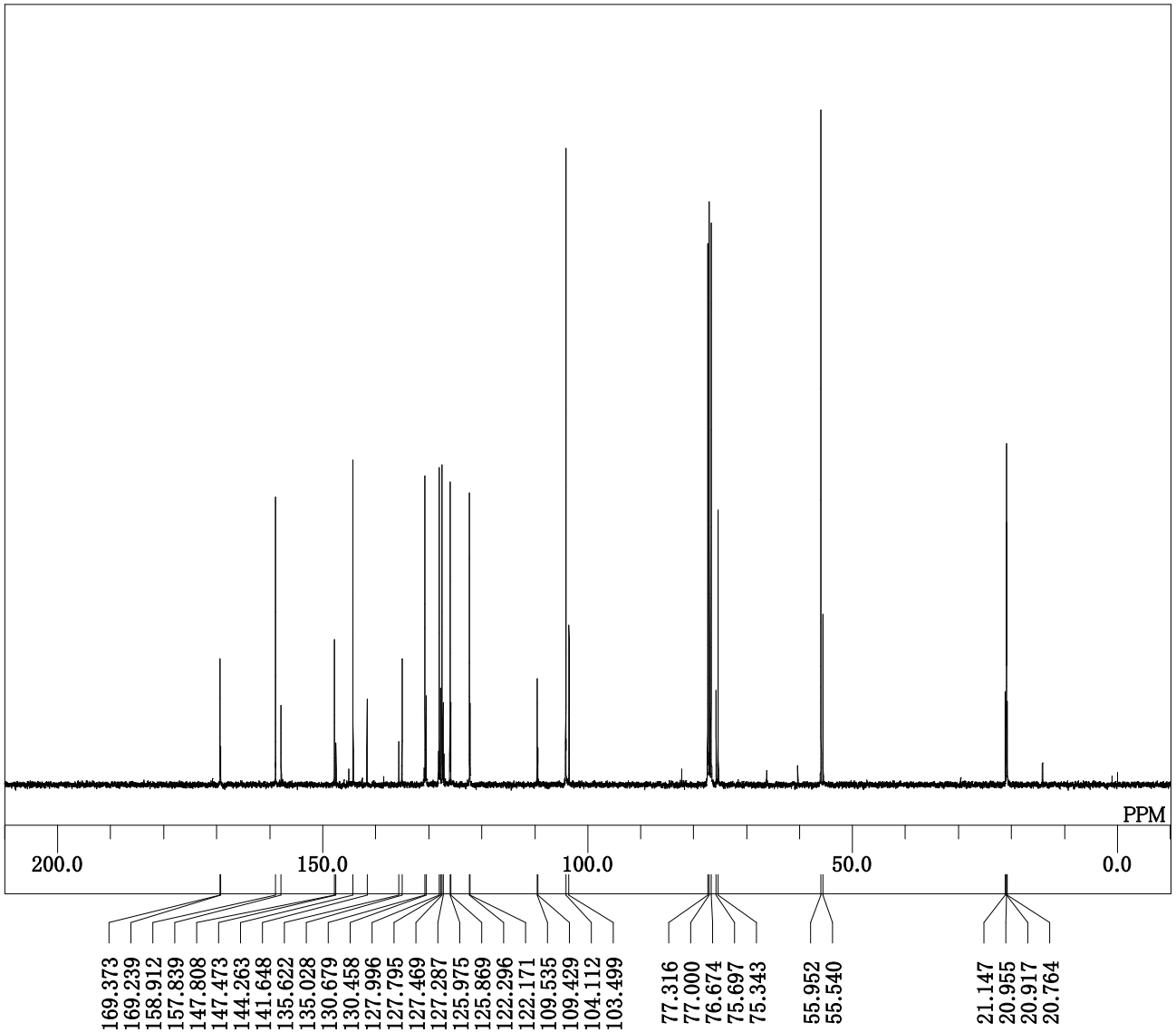
$^1$H NMR (395.88 MHz, Solvent=$CDCl_3$)

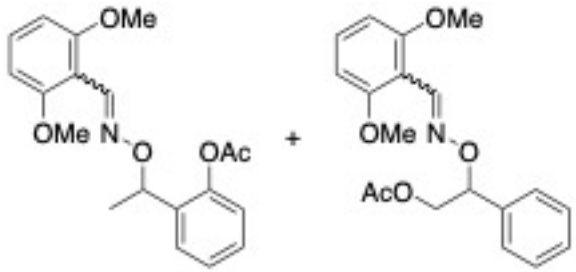




**2m**

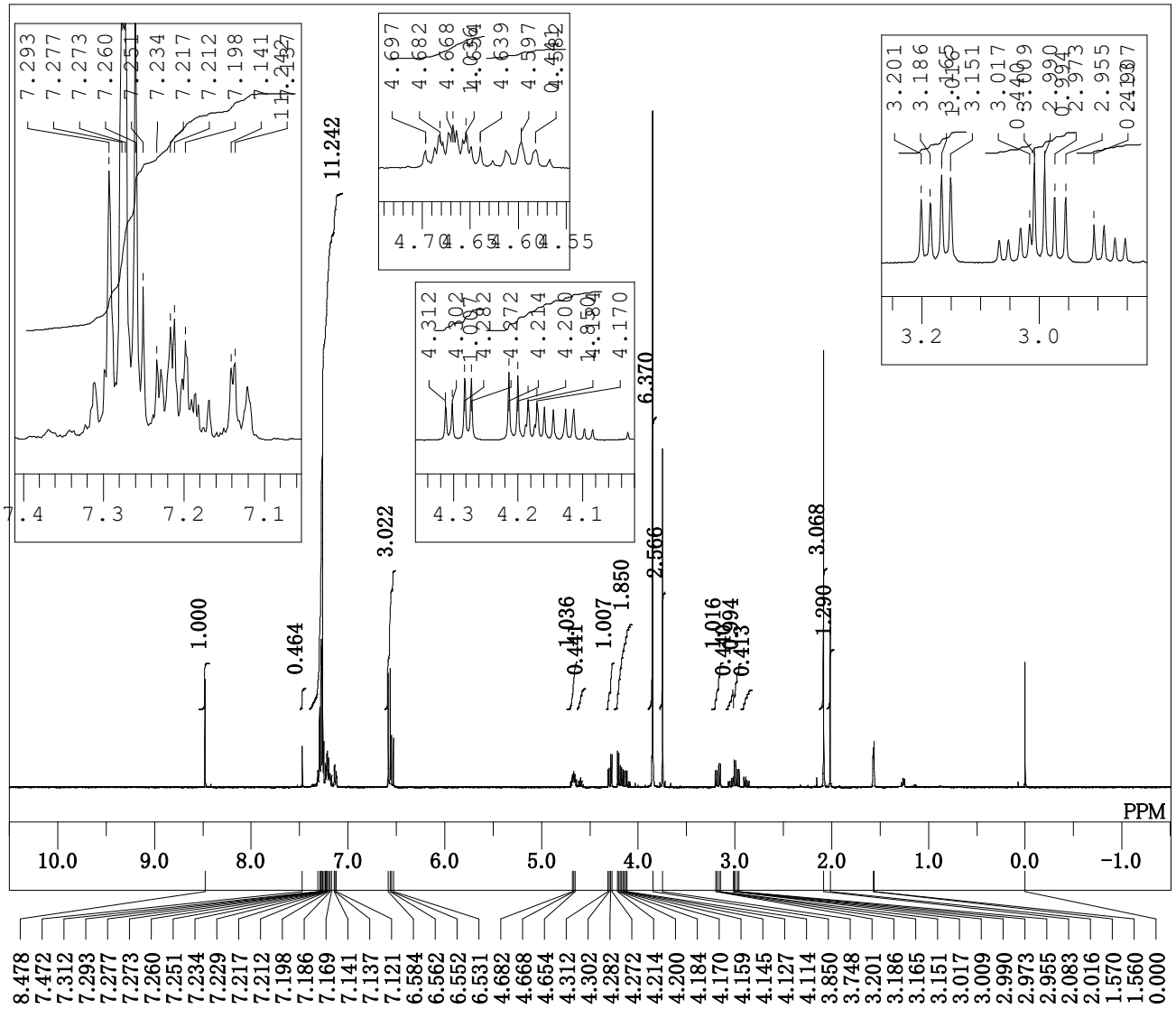
$^{13}$C NMR (99.55 MHz, Solvent=$CDCl_3$)

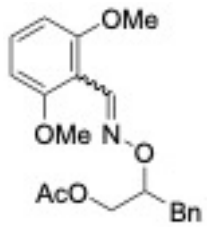



**2m**

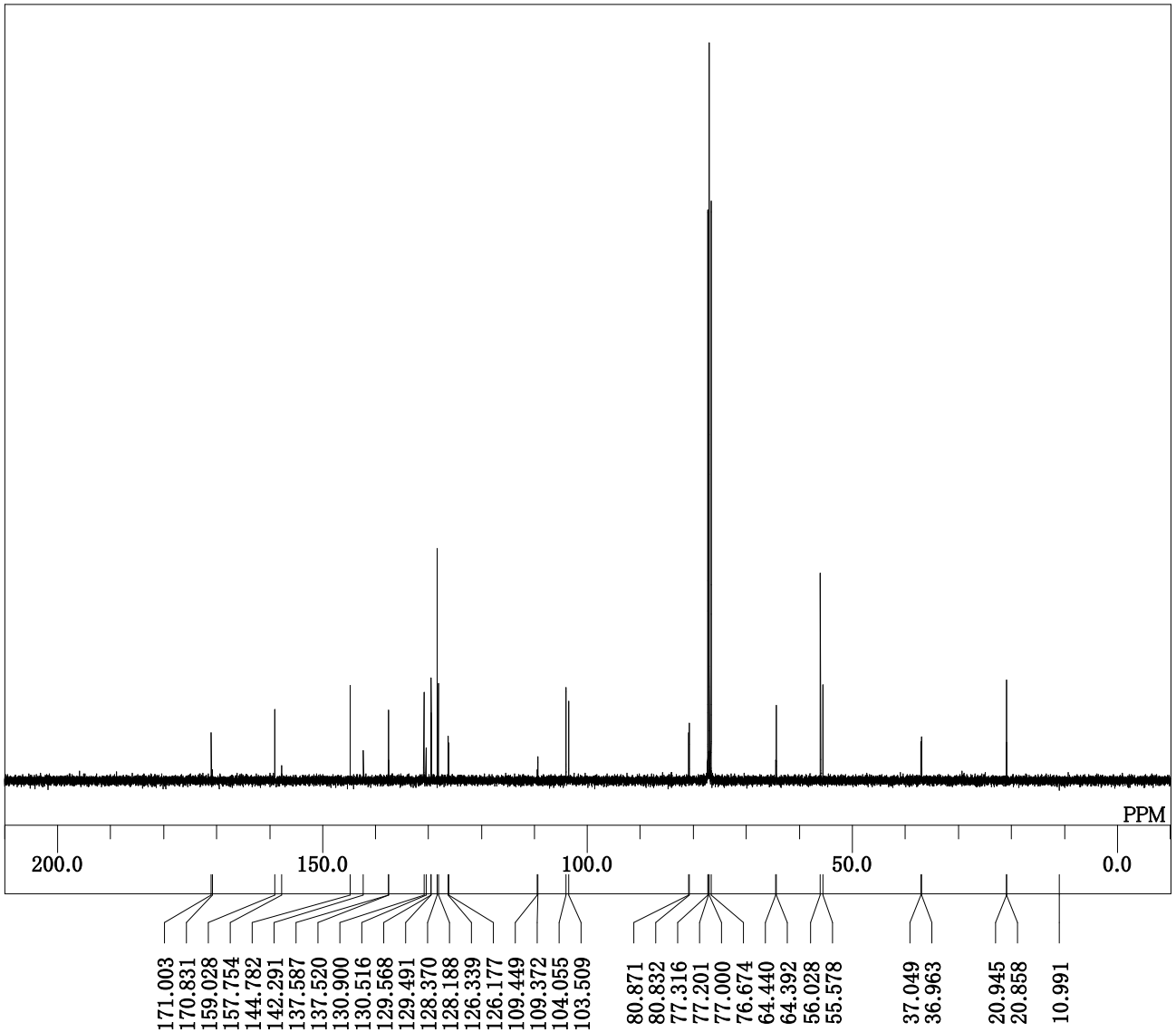
$^1$H NMR (395.88 MHz, Solvent=$CDCl_3$)

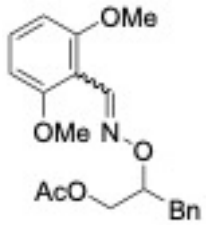





**2n**

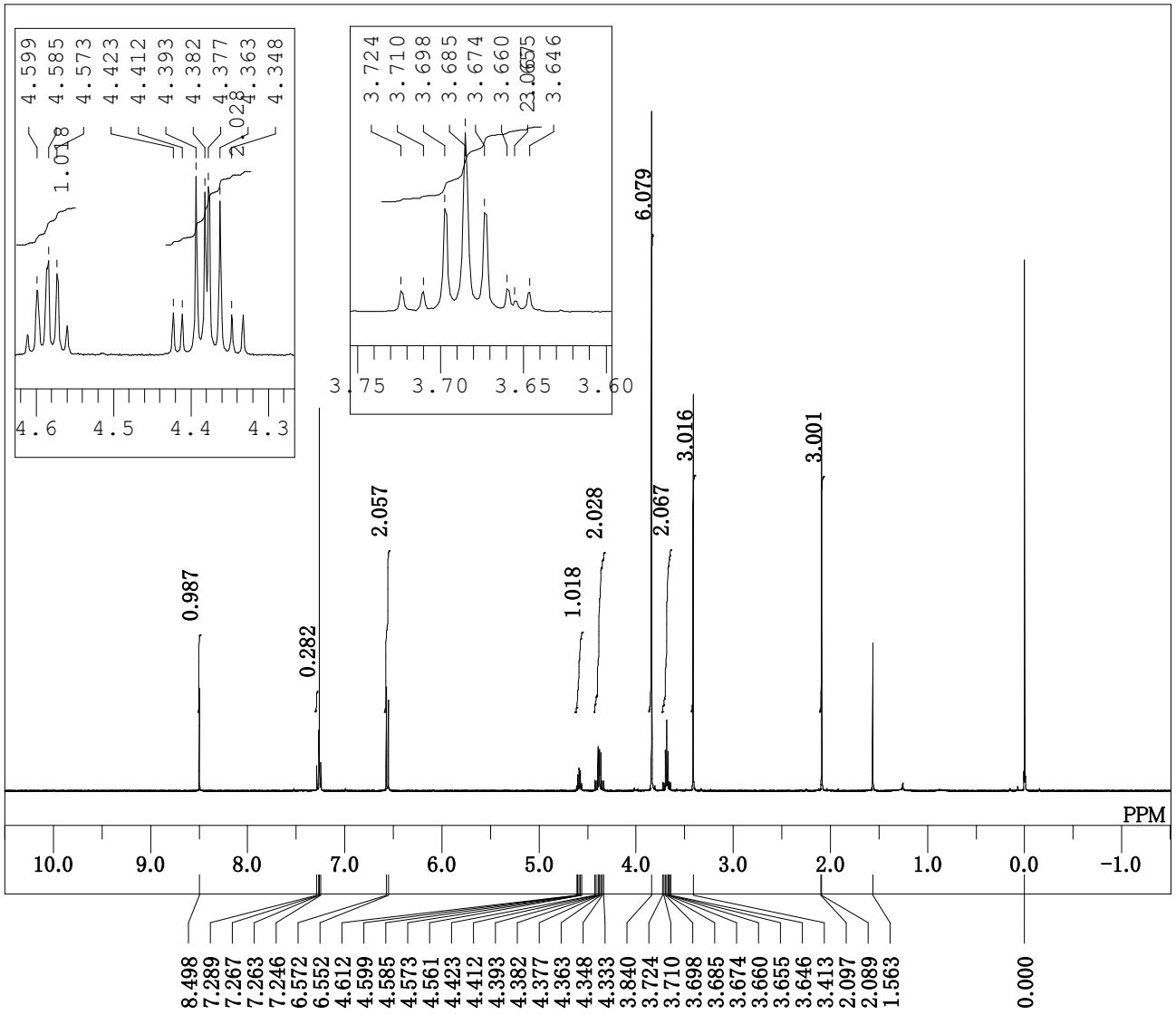
$^{13}$C NMR (99.55 MHz, Solvent=$CDCl_3$)

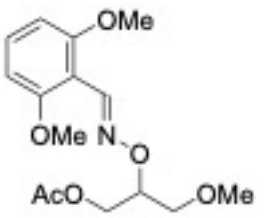



**2n**

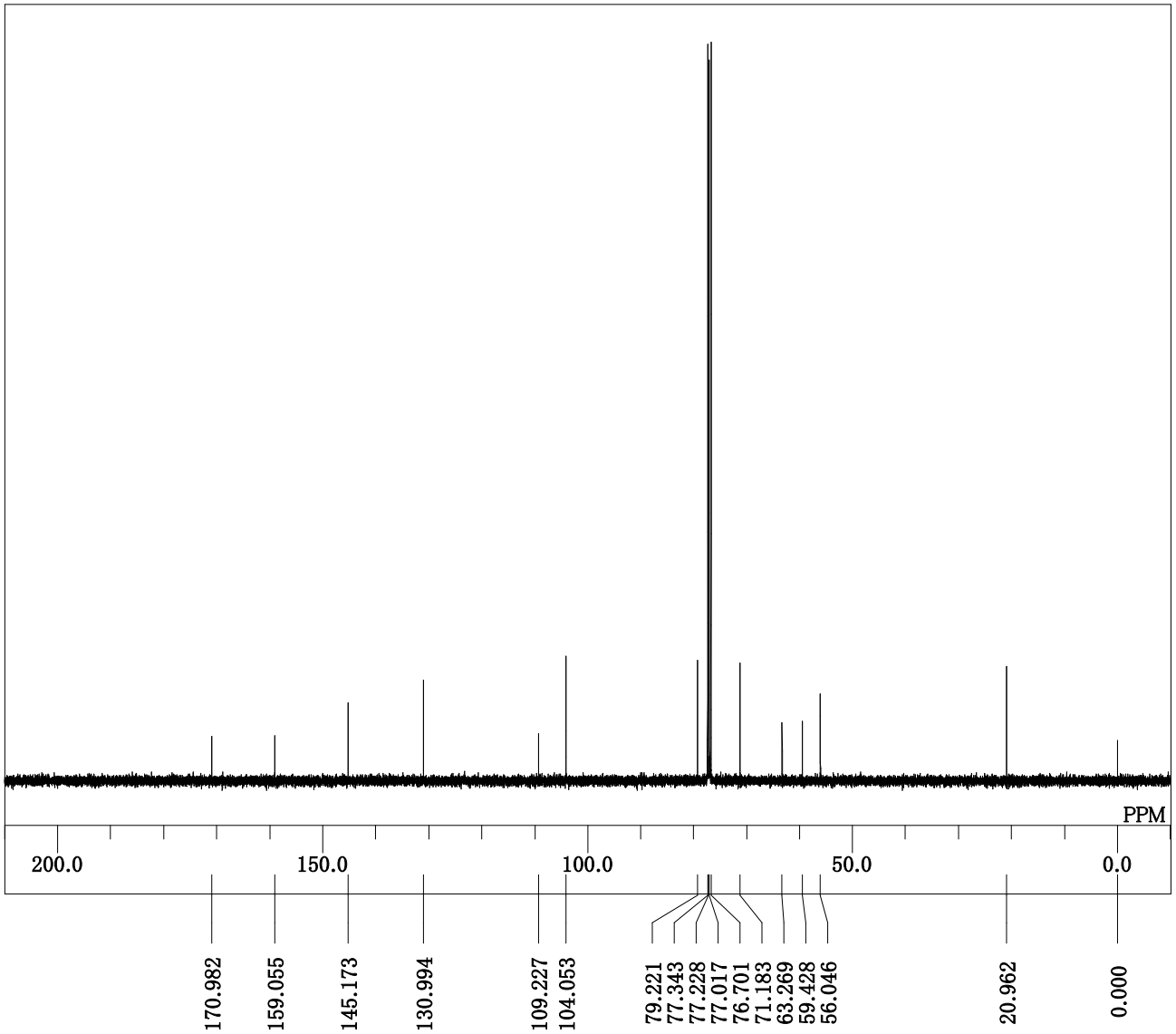
$^1$H NMR (395.88 MHz, Solvent=$CDCl_3$)

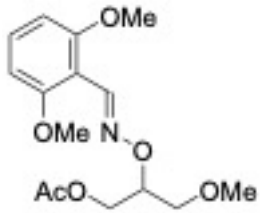



**2o**

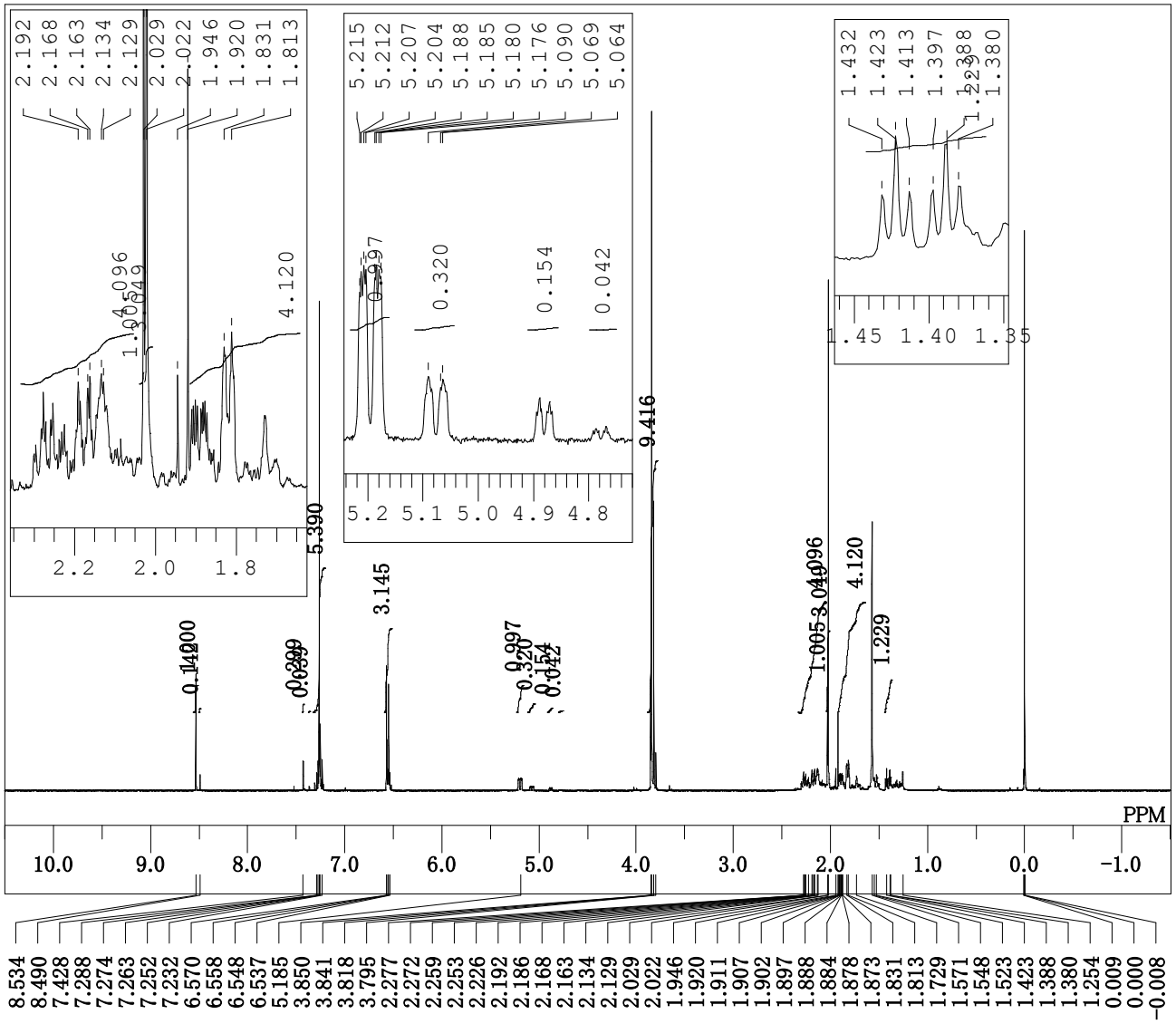
$^{13}$C NMR (99.55 MHz, Solvent=$CDCl_3$)

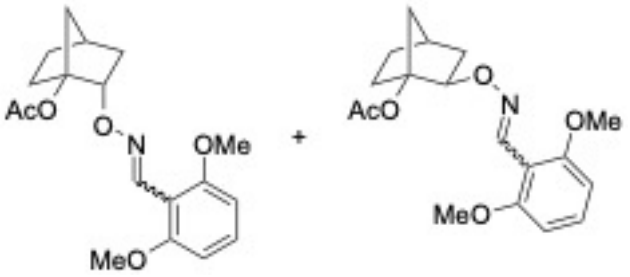



**2o**

$^1$H NMR (395.88 MHz, Solvent=$CDCl_3$)






**2t**