Chemoselective Deoxygenation of Ether-substituted Alcohols and Carbonyl Compounds by $B(C_6F_5)_3$-catalyzed Reduction with $(HMe_2SiCH_2)_2$

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Supporting Information

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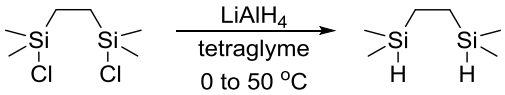
1. General Methods

Commercial reagents were used without any purification. $B(C_6F_5)_3$ was purchased from J&K Scientific. $(ClMe_2SiCH_2)_2$ was purchased from Duodian Chemistry (¥2200/200g). All reactions were performed using common anhydrous, inert atmosphere techniques. Reactions were monitored by TLC which was performed on glass-backed silica plates and visualized using UV, $KMnO_4$ stains, H_3PO_4•$12MoO_3$/EtOH stains, H_2SO_4(conc.)/anisaldehyde/ EtOH stains. Column chromatography was performed using silica gel (200-300 and 300-400 mesh) eluting with EtOAc/petroleum ether. ^1H NMR spectra were recorded at 400 MHz (Varian and Bruker) and 600 MHz (Agilent), and ^{13}C NMR spectra were recorded at 100 MHz (Varian) and 150 MHz (Agilent) using $CDCl_3$ (except where noted) with TMS or residual solvent as standard. Dibromomethane and 1,3,5-trimethoxybenzene were used as internal standard to calculate NMR yields. Infrared spectra were obtained using KCl plates on a VECTOR22. High-resolution mass spectral analyses performed on Waters Q-TOF. DMF, CH_2Cl_2 and Et_3N were distilled from CaH_2. Et_2O and THF were distilled from sodium. All spectral data obtained for new compounds are reported here.

2. General Procedure and Spectral Data

2.1. Preparations of $(HMe_2SiCH_2)_2$



To a suspension of $LiAlH_4$ (3 g, 78.9 mmol) in tetraglyme (35 mL) was slowly added 1,2-bis-(chlorodimethylsilyl)ethane (10 g, 46.5 mmol) at 0 ℃. Then the resulting mixture was stirred at 50 ℃ for 5 h. Purification by direct distillation from the resultant suspension under reduced pressure (64 ℃/90 Torr) gave $(HMe_2SiCH_2)_2$ (5.6 g, 83% yield) as a colorless liquid.[1]

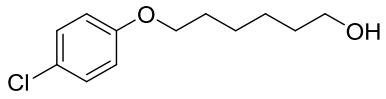
2.2. Preparations and Spectral Data of Alcohols

Alcohols **1a**, **1b, 1k** and **1o** are commercially available; **1e-1j**[2-6], **1l**[7] and **1p-1r**[10, 12-13] are

1. (*a*) S. Hanada, Y. Motoyama and H. Nagashima, *Eur. J. Org. Chem.*, 2008, 4097; (*b*) M. G. Steinmetz and B. S. Udayakumar, *J. Organomet. Chem.,* 1989, **378**, 1.

known compounds, which were prepared by the previously reported procedure.

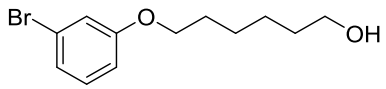
Preparation of 1c



1c

p-Chlorophenol (498 mg, 2.75 mmol), 6-bromo-1-hexanol (0.36 mL, 4.13 mmol) and K_2CO_3 (571 mg, 4.13 mmol) were stirred in DMF (8 mL) at 80 ℃ and for 4 h. After cooling to room temperature, the reaction was quenched with H_2O (15 mL) and extracted with ethyl acetate (3 × 10 mL). The combined organic layer was washed with sat. aq. NaCl (30 mL), dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by silica gel flash column chromatography (gradient eluent: 0-20% of EtOAc/petroleum ether) to afford **1c** (464 mg, 74% yield) as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 (d, *J* = 8.8 Hz, 2H), 6.81 (d, *J* = 8.8 Hz, 2H), 3.92 (t, *J* = 6.4 Hz, 2H), 3.66 (t, *J* = 6.4 Hz, 2H), 1.83–1.72 (m, 2H), 1.64–1.57 (m, 2H), 1.54–1.37 (m, 4H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 157.6, 129.2, 125.3, 115.7, 68.1, 62.8, 32.6, 29.1, 25.8, 25.5; IR (neat) cm^{-1} 3356, 2936, 2861, 1597, 1580, 1491, 1472, 1284, 1264, 1242, 1169, 1092, 1057, 1005, 823, 736; HRMS (ESI-TOF, m/z) calcd for $C_{12}H_{18}O_2Cl$ $(M+H)^+$: 229.0990, found 229.0986.

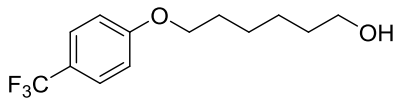
Preparation of 1d



1d

Using the same procedure as that used for **1c** afforded **1d** as a yellow oil (613 mg 82% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.12 (t, *J* = 8.0 Hz, 1H), 7.06 (d, *J* = 8.0 Hz, 1H), 7.05 (s, 1H), 6.82 (d, *J* = 8.0 Hz, 1H) 3.93 (t, *J* = 6.4 Hz, 2H), 3.66 (t, *J* = 6.4 Hz, 2H), 1.82–1.75 (m, 2H), 1.64–1.57 (m, 2H), 1.51–1.41 (m, 4H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 159.8, 130.4, 123.5, 122.7, 117.7, 113.5, 68.0, 62.8, 32.6, 29.1, 25.8, 25.5; IR (neat) cm^{-1} 3347, 2934, 2859, 1588, 1572, 1467, 1424, 1390, 1284, 1243, 1227, 1158, 1011, 991, 861, 765, 736, 680; HRMS (ESI-TOF, m/z) calcd for $C_{12}H_{18}O_2Br$ $(M+H)^+$: 273.0485, found 273.0488.

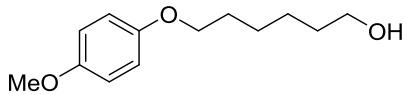
Preparation of 1e



1e

Using the same procedure as that used for **1c** afforded **1e**[2] as a yellow oil (995 mg, 71% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (d, *J* = 8.4 Hz, 2H), 6.94 (d, *J* = 8.4 Hz, 2H), 3.99 (t, *J* = 6.4 Hz, 2H), 3.66 (t, *J* = 6.4 Hz, 2H), 1.83–1.78 (m, 2H), 1.65–1.58 (m, 2H), 1.55–1.44 (m, 4H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 161.5, 126.8 (q, J_1 = 3.75 Hz), 124.4 (q, J_2 = 267.45 Hz), 122.6 (q, J_3 = 32.3 Hz), 114.4, 68.0, 62.8, 32.6, 29.0, 25.8, 25.5.

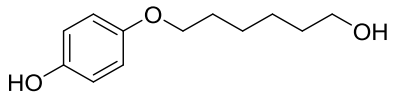
Preparation of 1f



1f

Using the same procedure as that used for **1c** afforded **1f**[2] as a white solid (529 mg, 85% yield, mp = 60–62 ℃). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.82 (s, 4H), 3.91 (t, *J* = 6.4 Hz, 2H), 3.76 (s, 3H), 3.65 (t, *J* = 6.4 Hz, 2H), 1.80–1.74 (m, 2H), 1.62–1.57 (m, 2H), 1.51–1.39 (m, 4H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 153.6, 153.2, 115.4, 114.6, 68.4, 62.8, 55.7, 32.6, 29.3, 25.8, 25.5.

Preparation of 1g



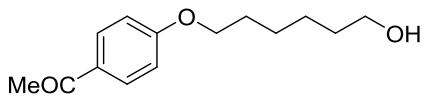
1g

Using the same procedure as that used for **1c** afforded **1g**[3] as a white solid (327 mg, 56% yield, mp = 87–88 ℃). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.79–6.73 (m, 4H), 4.76 (s, 1H), 3.89 (t, *J* = 6.4 Hz, 2H), 3.66 (t, *J* = 6.4 Hz, 2H), 1.80–1.73 (m, 2H), 1.62–1.56 (m, 2H), 1.49–1.42 (m, 4H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 153.2, 149.4, 116.0, 115.6, 685, 62.9, 32.7, 29.3, 25.9, 25.5.

Preparation of 1h

2. W. Yu, T. Gill, L. Wang, Y. Du, H. Ye, X. Qu, J. Guo, A. Cuconati, K. Zhao, T. M. Block, X. Xu and J. Chang, *J. Med. Chem.*2012, **55**, 6061.

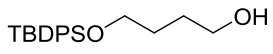
3. J. Lenoble, N. Maringa, S. Campidelli, B. Donnio, D. Guillon and R. Deschenaux, *Org. Lett.*, 2006, **8**, 1851.



1h

Using the same procedure as that used for **1c** afforded **1h**[4] as a yellow solid (455 mg, 70% yield, mp = 57–59 ℃). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (d, *J* = 8.4 Hz, 2H), 6.91 (d, *J* = 8.4 Hz, 2H), 4.02 (t, *J* = 6.4 Hz, 2H), 3.66 (t, *J* = 6.4 Hz, 2H), 2.55 (s, 3H), 1.87–1.76 (m, 2H), 1.63–1.57 (m, 2H), 1.53–1.42 (m, 4H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 196.9, 163.0, 130.5, 130.0, 114.1, 68.0, 62.7, 32.6, 29.0, 26.3, 25.8, 25.5.

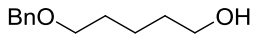
Preparation of 1i



1i

1i was prepared according to a known procedure.[5] **1i**: colorless oil, 1.33 g, 81% yield. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.67 (d, *J* = 7.2 Hz, 4H), 7.44–7.38 (m, 6H), 3.70 (t, *J* = 6.0 Hz, 2H), 3.67 (q, *J* = 6.0 Hz, 2H), 2.01 (t, *J* = 5.4 Hz, 1H), 1.71–1.64 (m, 4H), 1.06 (s, 9H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 135.5, 133.6, 129.6, 127.6, 64.0, 62.7, 29.8, 29.2, 26.8, 19.1.

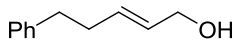
Preparation of 1j



1j

1j was prepared according to a known procedure.[6] **1j**: colorless oil, 776 mg, 80% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35–7.26 (m, 5H), 4.50 (s, 2H), 3.64 (t, *J* = 6.8 Hz, 2H), 3.48 (t, *J* = 6.8 Hz, 2H), 1.69–1.55 (m, 4H), 1.49–1.43 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 138.4, 128.2, 127.5, 127.4, 72.8, 70.2, 62.4, 32.3, 29.3, 22.3.

Preparation of 1l

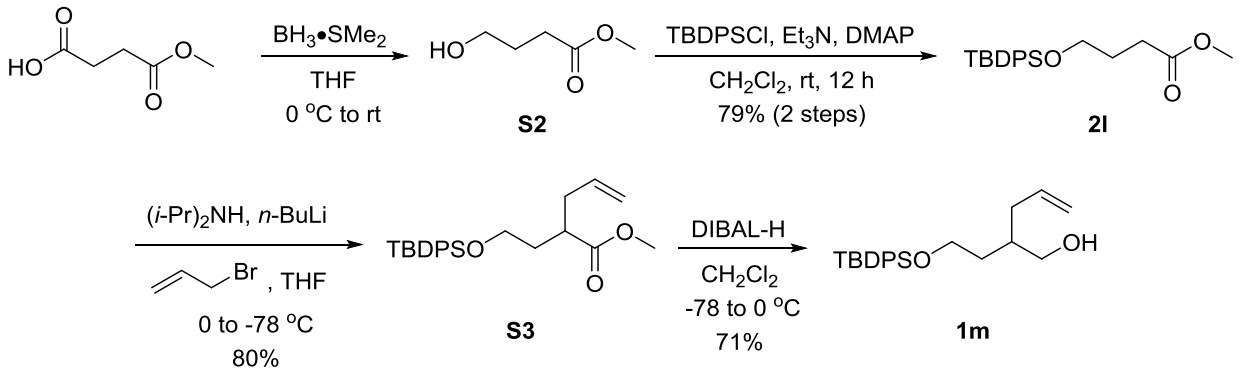


1l

4. I. Buslov, J. Becouse, S. Mazza, M. Montandon-Clerc and X. Hu, *Angew. Chem., Int. Ed.*, 2015, **54**, 14523.
5. A. W. J. Logan, J. S. Parker, M. S. Hallside and J. W. Burton, *Org. Lett.*, 2012, **14**, 2940.
6. K. Kubota, E. Yamamoto and H. Ito, *J. Am. Chem. Soc.*, 2015, **137**, 420.

1l was prepared according to a known procedure.[7] **1l**: colorless oil, 675 mg, 85% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 (t, *J* = 7.6 Hz, 2H), 7.21–7.18 (m, 3H), 5.78–5.64 (m, 2H), 4.09 (d, *J* = 4.8 Hz, 2H), 2.71 (t, *J* = 8.0 Hz, 2H), 2.38 (q, *J* = 8.0 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 141.6, 131.9, 129.5, 128.3, 128.2, 125.7, 63.4, 35.4, 33.8.

Preparation of 1m



To a solution of methyl hydrogen succinate (5 g, 37.8 mmol) in THF (30 mL) was added BH_3•SMe_2 (4.9 mL of 10 M solution in THF, 49.2 mmol) at 0 ℃. The reaction mixture was stirred for 12 h at room temperature before quenching slowly with H_2O and K_2CO_3. The mixture was then filtered by Celite and concentrated under reduced pressure to give crude **S2** (4.45 g), which was used in the next reaction without further purification.

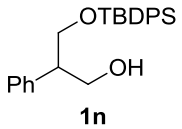
To a solution of **S2** (2.8 g, 23.7 mmol), Et_3N (6.6 mL, 47.4 mmol) and DMAP (290 mg, 2.37 mmol) in CH_2Cl_2 (30 mL) was added *tert*-butyldiphenylsilylchloride (7.14 g, 26.0 mmol) at 0 ℃. The reaction mixture was stirred at room temperature for 12 h before quenching with sat. aq. NH_4Cl (15 mL) and then extraction with CH_2Cl_2 (3 × 20 mL). The combined organic layers were then dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by silica gel flash column chromatography (gradient eluent: 0-10% of EtOAc/petroleum ether) to afford **2l** as a colorless liquid (6.6 g, 79% yield)[8]. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (d, *J* = 6.8 Hz, 4H), 7.45–7.37 (m, 6H), 3.69 (t, *J* = 6.0 Hz, 2H), 3.66 (s, 3H), 2.47 (t, *J* = 7.6 Hz, 2H), 1.92–1.86 (m, 2H), 1.06 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 174.0, 135.5, 133.7, 129.6, 127.6, 62.8, 51.5, 30.6, 27.7, 26.8, 19.2.

7. I. Franzoni, L. Guénée and C. Mazet, *Chem. Sci.,* 2013, **4**, 2619.
8. Y. Hayashi, J. Yamaguchi and M. Shoji, *Tetrahedron,* 2002, **58**, 9839.

To a solution of $(i\text{-Pr})_2NH$ (0.48 mL, 3.4 mmol) in THF (10 mL) at 0 ℃ was added *n*-BuLi (1.36 mL of 2.5 M solution in hexane, 3.4 mmol). The resulting solution was stirred for 15 min then cooled to -78 ℃. A solution of **2l** (0.6 g, 1.7 mmol) in THF (5 mL) was added to the above mixture dropwise. The resulting mixture was stirred at -78 ℃ for 1 h before adding allylbromide (0.3 mL, 3.4 mmol). The reaction was stirred at -78 ℃ for 2 h. and quenched with sat. aq. NH_4Cl (5 mL). The aqueous layer was extracted with Et_2O (3 × 5 mL). The combined organic layers were then dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by silica gel flash column chromatography (gradient eluent: 0-5% of EtOAc/petroleum ether) to afford **S3** as a colorless oil (530 mg, 80% yield).[9]

To a solution of **S3** (380 mg, 0.96 mmol) in CH_2Cl_2 (10 mL) was added DIBAL-H (1.1 mL of 1.0 M solution in hexane, 1.1 mmol) at 0 ℃. The reaction mixture was stirred for 30 min before quenching with sat. aq. potassium sodium tartrate (10 mL). The mixture was extracted with CH_2Cl_2 (3 × 5 mL). The combined organic layers were then dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by silica gel flash column chromatography (gradient eluent: 0-20% of EtOAc/petroleum ether) to afford **1m** as a light yellow oil (251 mg, 71% yield). ^1H NMR (600 MHz, $CDCl_3$) δ 7.67 (d, *J* = 7.2 Hz, 4H), 7.45–7.38 (m, 6H), 5.80–5.73 (m, 1H), 5.02 (d, *J* = 15.0 Hz, 1H), 5.00 (d, *J* = 8.4 Hz, 1H), 3.78–3.75 (m, 1H), 3.72–3.68 (m, 1H), 3.63–3.61 (m, 1H), 3.55–3.53 (m, 1H), 2.61 (s, 1H), 2.14–2.10 (m, 1H), 2.06–2.01 (m, 1H), 1.82–1.78 (m, 1H), 1.67–1.63 (m, 2H), 1.06 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 136.8, 135.5, 133.2, 129.7, 127.7, 116.3, 65.8, 62.6, 38.7, 36.2, 34.3, 26.8, 19.1; IR (neat) cm^{-1} 3379, 3072, 2930, 2857, 1640, 1589, 1472, 1427, 1390, 1106, 1083, 1030, 997, 912, 822, 736, 699, 613; HRMS (ESI-TOF, m/z) calcd for $C_{23}H_{33}O_2Si$ $(M+H)^+$: 369.2244, found 369.2242.

Preparation of 1n



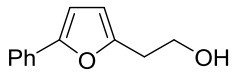
1n

Using the same procedure as that used for **1i** afforded **1n** as a colorless oil (1.54 g, 79% yield). ^1H NMR (400 MHz, $CDCl_3$) δ 7.63 (t, *J* = 7.2 Hz, 4H), 7.45–7.36 (m, 6H), 7.30–7.22 (m, 3H), 7.15 (d,

9. A. Joosten, E. Lambert, J. Vasse and J. Szymoniak, *Org. Lett.*, 2010, **12**, 5128.

J = 7.2 Hz, 2H), 4.15–4.10 (m, 1H), 3.99–3.88 (m, 3H), 3.16–3.10 (m, 1H), 2.35 (t, *J* = 6.0 Hz, 1H), 1.06 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 139.5, 135.6, 135.5, 133.0, 132.97, 129.8, 129.75, 128.5, 128.1, 127.74, 127.73, 127.0, 67.3, 65.8, 49.8, 26.8, 19.1; IR (neat) cm^{-1} 3417, 3070, 2930, 2857, 1589, 1494, 1472, 1427, 1390, 1361, 1265, 1109, 1029, 822, 758, 737, 698, 611; HRMS (ESI-TOF, m/z) calcd for $C_{25}H_{31}O_2Si$ $(M+H)^+$: 391.2088, found 391.2084.

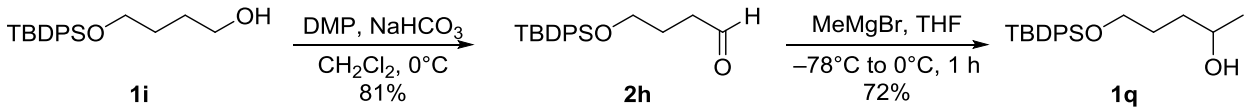
Preparation of 1p



1p

1p was prepared according to a known procedure[10]. **1p**: yellow oil, 670 mg, 56% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (d, *J* = 7.6 Hz, 2H), 7.37 (t, *J* = 7.6 Hz, 2H), 7.23 (d, *J* = 7.6 Hz, 1H), 6.58 (d, *J* = 3.2 Hz, 1H), 6.20 (d, *J* = 3.2 Hz, 1H), 3.93 (t, *J* = 6.0 Hz, 2H), 2.97 (t, *J* = 6.0 Hz, 2H), 1.83 (s, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 152.9, 152.4, 130.8, 128.6, 127.0, 123.4, 108.7, 105.7, 61.0, 31.8.

Preparation of 1q



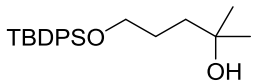
1i **2h** **1q**

To a solution of **1i** (660 mg, 2 mmol) in CH_2Cl_2 (20 mL) was added Dess-Martin periodinane (1.0 g, 2.4 mmol) and $NaHCO_3$ (840 mg, 10 mmol). The resulting mixture was stirred at 0 ℃ until the starting material was completely consumed (monitored by TLC analysis). The reaction mixture was quenched with sat. aq. $Na_2S_2O_3$ (15 mL). The aqueous phase was extracted with CH_2Cl_2 (3 × 10 mL). The combined organic layers were then dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by silica gel flash column chromatography (gradient eluent: 0-10% of EtOAc/petroleum ether) to afford **2h**[11] as a colorless oil (528 mg, 81% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.80 (s, 1H), 7.66 (d, *J* = 7.2 Hz, 4H), 7.46–7.38 (m, 6H), 3.70 (t, *J* = 6.0 Hz, 2H), 2.56 (t, *J* = 6.8 Hz, 2H), 1.93–1.86 (m, 2H), 1.06 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.6, 135.5, 133.5, 129.7, 127.7, 62.9, 40.7, 26.8, 25.2, 19.1.

10. J. Li, L. Lu, Q. Pan, Y. Ren, B. Liu and B. Yin, *Adv. Synth. Catal.*, 2017, **359**, 2001.
11. J. Luo, H. Li, J. Wu, X. Xing and W. Dai, *Tetrahedron,* 2009, **65**, 6828.

To a stirred solution of **2h** (260 mg, 0.8 mmol) in THF (12 mL) was added MeMgBr (1.2 mL of 1.0 M solution in THF, 1.2 mmol) at -78 ℃, then the mixture was allowed to warm to 0 ℃. After further stirring for 1 h, the reaction was quenched with sat. aq. NH_4Cl (5 mL) and the aqueous phase was extracted with EtOAc (3 × 10 mL). The combined organic layers were then dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by silica gel flash column chromatography (gradient eluent: 0-10% of EtOAc/petroleum ether) to afford **1q**[12] as a colorless oil (196 mg, 72% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69–7.67 (m, 4H), 7.44–7.37 (m, 6H), 3.86–3.81 (m, 1H), 3.70 (t, *J* = 6.4 Hz, 2H), 2.19 (d, *J* = 4.0 Hz, 1H), 1.67–1.51 (m, 4H), 1.20 (d, *J* = 6.4 Hz, 3H), 1.06 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 135.6, 133.6, 129.6, 127.6, 67.8, 64.2, 36.3, 28.9, 26.8, 23.4, 19.1.

Preparation of 1r



1r

To a stirred solution of **2l** (178 mg, 0.5 mmol) in dry Et_2O (5 mL) was added MeMgBr (1.5 mL of 1.0 M solution in THF, 1.5 mmol) at -20 ℃. Then the mixture was allowed to warm to 0 ℃ and stirred until the starting material was completely consumed (monitored by TLC analysis). The reaction mixture was quenched with sat. aq. NH_4Cl (5 mL). The aqueous phase was extracted with EtOAc (3 × 5 mL). The combined organic layers were then dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by silica gel flash column chromatography (gradient eluent: 0-10% of EtOAc/petroleum ether) to afford **1r**[13] as a colorless oil (150 mg, 84% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, *J* = 6.8 Hz, 4H), 7.45–7.37 (m, 6H), 3.69 (t, *J* = 6.4 Hz, 2H), 1.82 (s, 1H), 1.70–1.55 (m, 4H), 1.23 (s, 6H), 1.06 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 135.6, 133.7, 129.6, 127.6, 70.5, 64.6, 40.5, 29.3, 27.5, 26.8, 19.2.

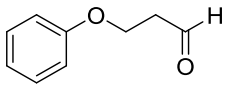
2.3. Preparations and Spectral Data of Carbonyl Compounds

Aldehydes **2a**-**2b**[14], **2h**[11] and **2j**-**2l**[7-8, 15] are known compounds, which were prepared by the previously reported procedures.

12. G. Pattenden, D. A. Stoker and N. M. Thomson, *Org. Biomol. Chem.*, 2007, **5**, 1776.

13. K. Masutani, T. Minowa, Y. Hagiwara and T. Mukaiyama, *Bull. Chem. Soc. Jpn.*, 2006, **79**, 1106.

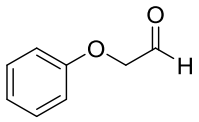
Preparation of 2a



2a

Using the same procedure as that used for **2h** afforded **2a**[14] as a colorless oil from **1a** (125 mg, 83% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.87 (s, 1H), 7.30 (t, *J* = 7.6 Hz, 2H), 6.98 (t, *J* = 7.6 Hz, 1H), 6.91 (d, *J* = 7.6 Hz, 2H), 4.32 (t, *J* = 6.0 Hz, 2H), 2.91 (t, *J* = 6.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.3, 158.3, 129.5, 121.1, 114.5, 61.4, 43.2.

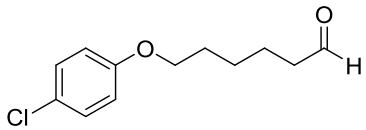
Preparation of 2b



2b

Using the same procedure as that used for **2h** afforded **2b**[14] as a colorless oil from **1b** (199 mg, 73% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.87 (s, 1H), 7.33 (t, *J* = 7.6 Hz, 2H), 7.03 (t, *J* = 7.6 Hz, 1H), 6.91 (d, *J* = 7.6 Hz, 2H), 4.58 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.4, 157.6, 129.7, 121.9, 114.5, 72.6.

Preparation of 2c

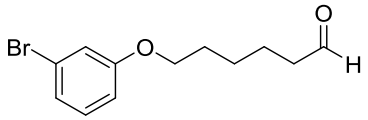


2c

Using the same procedure as that used for **2h** afforded **2c** as a colorless oil from **1c** (175 mg, 71% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.78 (s, 1H), 7.22 (d, *J* = 8.8 Hz, 2H), 6.80 (d, *J* = 8.8 Hz, 2H), 3.92 (t, *J* = 6.4 Hz, 2H), 2.48 (t, *J* = 7.2 Hz, 2H), 1.83–1.76 (m, 2H), 1.74–1.67 (m, 2H), 1.54–1.47 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 202.4, 157.5, 129.2, 125.3, 115.7, 67.8, 43.7, 28.9, 25.6, 21.7; IR (neat) cm^{-1} 2937, 28656, 2720, 1721, 1588, 1571, 1467, 1424, 1390, 1284, 1243, 1227, 1157, 1092, 1064, 1022, 991, 859, 767, 680; HRMS (ESI-TOF, m/z) calcd for $C_{12}H_{16}O_2Cl$ $(M+H)^+$: 227.0833, found 227.0831.

Preparation of 2d

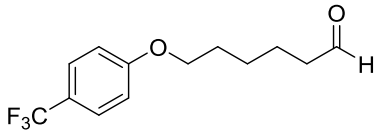
14. G. Foyer, B. H. Chanfi, B. Boutevin, S. Caillol and G. David, *Eur. Polym. J.*, 2016, **74**, 296.



2d

Using the same procedure as that used for **2h** afforded **2d** as a light yellow oil from **1d** (191 mg, 77% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.78 (s, 1H), 7.12 (t, *J* = 8.4 Hz, 1H), 7.07 (d, *J* = 8.4 Hz, 1H), 7.04 (s, 1H), 6.81 (d, *J* = 8.4 Hz, 1H), 3.94 (t, *J* = 6.6 Hz, 2H), 2.48 (t, *J* = 7.2 Hz, 2H), 1.82–1.77 (m, 2H), 1.73–1.68 (m, 2H), 1.53–1.48 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 202.3, 159.7, 130.5, 123.6, 122.7, 117.7, 113.4, 67.7, 43.7, 28.9, 25.6, 21.7; IR (neat) cm^{-1} 2937, 2866, 2720, 1721, 1588, 1571, 1467, 1424, 1390, 1284, 1242. 1227, 1157, 1064, 1022, 991, 859, 767, 680; HRMS (ESI-TOF, m/z) calcd for $C_{12}H_{15}BrNaO_2$ $(M+Na)^+$: 293.0148, found 293.0151.

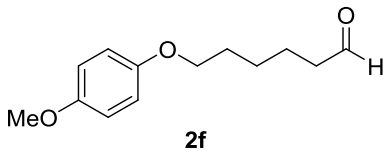
Preparation of 2e



2e

Using the same procedure as that used for **2h** afforded **2e** as a light yellow oil from **1e** (140 mg, 89% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.79 (s, 1H), 7.53 (d, *J* = 8.8 Hz, 2H), 6.93 (d, *J* = 8.8 Hz, 2H), 4.00 (t, *J* = 6.4 Hz, 2H), 2.49 (t, *J* = 7.2 Hz, 2H), 1.86–1.79 (m, 2H), 1.75–1.68 (m, 2H), 1.58–1.48 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 202.4, 161.4, 126.8 (q, J_1 = 3.75 Hz), 124.4 (q, J_2 = 269.25 Hz), 122.7 (q, J_3 = 32.4 Hz), 114.4, 67.7, 43.7, 28.8, 25.6, 21.7; IR (neat) cm^{-1} 2941, 2870, 2723, 1723, 1615, 1590, 1519, 1324, 1310, 12559, 1177, 1158, 1107, 1066, 100, 835, 813, 736; HRMS (ESI-TOF, m/z) calcd for $C_{13}H_{15}F_3NaO_2$ $(M+Na)^+$: 283.0916, found 283.0920.

Preparation of 2f

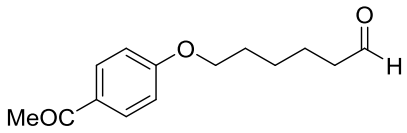


2f

Using the same procedure as that used for **2h** afforded **2f** as a light yellow oil from **1f** (141 mg, 71% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.78 (s, 1H), 6.82 (s, 4H), 3.91 (t, *J* = 6.4 Hz, 2H), 3.76 (s, 3H), 2.47 (t, *J* = 7.2 Hz, 2H), 1.82–1.67 (m, 4H), 1.55–1.47 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 202.5, 153.7, 153.1, 115.3, 114.6, 68.1, 55.7, 43.7, 29.1, 25.7, 21.8; IR (neat) cm^{-1} 2937, 2865, 2722,

1721, 1506, 1466, 1442, 1391, 1288, 1226, 1180, 1107, 1036, 824, 736; HRMS (ESI-TOF, m/z) calcd for $C_{13}H_{19}O_3$ $(M+H)^+$: 223.1329, found 223.1326.

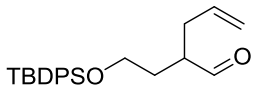
Preparation of 2g



2g

Using the same procedure as that used for **2h** afforded **2g** as a white solid from **1h** (142 mg, 73% yield, mp = 49–51 ℃). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.78 (s, 1H), 7.91 (d, *J* = 8.8 Hz, 2H), 6.90 (d, *J* = 8.8 Hz, 2H), 4.02 (t, *J* = 6.4 Hz, 2H), 2.54 (s, 3H), 2.48 (t, *J* = 7.2 Hz, 2H), 1.86–1.79 (m, 2H), 1.75–1.67 (m, 2H), 1.55–1.48 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 202.3, 196.7, 162.9, 130.5, 130.1, 114.0, 67.7, 43.7, 28.8, 26.2, 25.5, 21.6; IR (neat) cm^{-1} 2941, 2867, 2825, 2724, 1721, 1710, 1671, 1599, 1575, 1509, 1474, 1358, 1305, 1251, 1169, 1116, 1045, 1008, 958, 834, 817, 734; HRMS (ESI-TOF, m/z) calcd for $C_{14}H_{19}O_3$ $(M+H)^+$: 235.1329, found 235.1325.

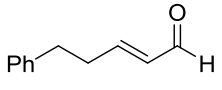
Preparation of 2i



2i

Using the same procedure as that used for **2h** afforded **2i** as a light yellow oil from **1m** (272 mg, 81% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.68 (s, 1H), 7.64 (d, *J* = 7.1 Hz, 4H), 7.44–7.38 (m, 6H), 5.75–5.69 (m, 1H), 5.06–5.03 (m, 2H), 3.71–3.66 (m, 2H), 2.60–2.58 (m, 1H), 2.44–2.39 (m, 1H), 2.25–2.20 (m, 1H), 1.98–1.92 (m, 1H), 1.76–1.71 (m, 1H), 1.04 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 204.3, 135.5, 134.9, 133.4, 129.7, 127.7, 117.3, 61.3, 48.3, 32.8, 31.2, 26.8, 19.1; IR (neat) cm^{-1} 3071, 2930, 2857, 2713, 1724, 1641, 1589, 1472, 1427, 1390, 1361, 1260, 1107, 997, 917, 822, 739, 701, 614; HRMS (ESI-TOF, m/z) calcd for $C_{23}H_{31}O_2Si$ $(M+H)^+$: 367.2088, found 367.2083.

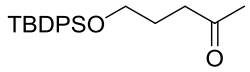
Preparation of 2j



2j

Using the same procedure as that used for **2h** afforded **2j**[7] as a light yellow oil from **1l** (155 mg, 86% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.49 (d, *J* = 7.8 Hz, 1H), 7.31 (t, *J* = 7.2 Hz, 2H), 7.22 (t, *J* = 7.2 Hz, 1H), 7.19 (d, *J* = 7.2 Hz, 2H), 6.88–6.84 (m, 1H), 6.14 (dd, *J* = 15.6, 7.8 Hz, 1H), 2.84 (t, *J* = 7.2 Hz, 2H), 2.68 (q, *J* = 7.2 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 193.9, 157.3, 140.2, 133.3, 128.5, 128.3, 126.3, 34.2, 34.0.

Preparation of 2k

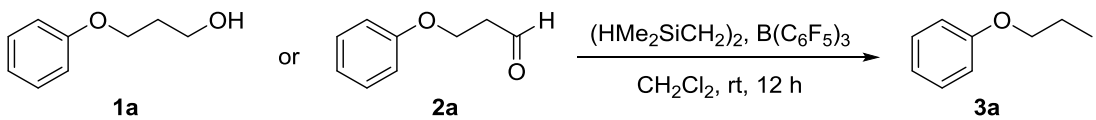


2k

Using the same procedure as that used for **2h** afforded **2k**[15] as a light yellow oil from **1q** (123 mg, 90% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.65 (d, *J* = 7.8 Hz, 4H), 7.44–7.37 (m, 6H), 3.67 (t, *J* = 6.0 Hz, 2H), 2.55 (t, *J* = 7.2 Hz, 2H), 2.13 (s, 3H), 1.85–1.82 (m, 2H), 1.05 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 208.9, 135.5, 133.7, 129.6, 127.6, 62.9, 40.1, 29.9, 26.8, 26.6, 19.2.

2.4. General Procedure of Deoxygenation to Synthesize Alkane

Preparation of 3a



1a **2a** **3a**

Procedure A (from alcohol): To a solution of **1a** (30.4 mg, 0.2 mmol) and $B(C_6F_5)_3$ (5.1 mg, 0.01 mmol) in anhydrous CH_2Cl_2 (4 mL) under argon atmosphere was added $(HMe_2SiCH_2)_2$ (35.1 mg, 0.24 mmol) at room temperature. The resulting mixture was stirred for 12 h before quenching with H_2O (2 mL) and extraction with CH_2Cl_2 (3 × 3 mL). The combined organic layers were dried over Na_2SO_4, filtered and concentrated under reduced pressure to afford **3a**[16] (70%. The yield was determined by ^{1}H NMR analysis using dibromomethane as an internal standard).

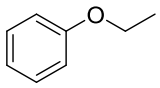
Procedure B (from aldehyde): To a solution of **2a** (30 mg, 0.2 mmol) and $B(C_6F_5)_3$ (5.1 mg, 0.01 mmol) in anhydrous CH_2Cl_2 (4 mL) under argon atmosphere was added $(HMe_2SiCH_2)_2$ (35.1 mg, 0.24 mmol) at room temperature. The resulting mixture was stirred for 12 h before quenching with H_2O (2 mL) and extraction with CH_2Cl_2 (3 × 3 mL). The combined organic layers were dried

15. A. Rühling, H. J. Galla and F. Glorius, *Chem. Eur. J.,* 2015, **21**, 12291.
16. Y. Zheng, P. Ye, B. Chen, Q. Meng, K. Feng, W. Wang, L. Wu and C. Tung, *Org. Lett.*, 2017, **19**, 2206.

over Na_2SO_4, filtered and concentrated under reduced pressure to afford **3a** (68%. The yield was determined by ^1H NMR analysis using dibromomethane as an internal standard).

3a : ^1H NMR (400 MHz, $CDCl_3$) δ 7.28 (t, *J* = 7.6 Hz, 2H), 6.96–6.90 (m, 3H), 3.93 (t, *J* = 6.8 Hz, 2H), 1.86–1.78 (m, 2H), 1.05 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 159.1, 129.4, 120.4, 114.5, 69.4, 22.6, 10.5.

Preparation of 3b



3b

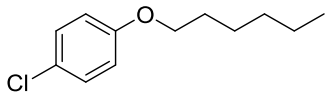
Using the same procedure as that used for **3a**.

Procedure A: 1b (27.6 mg, 0.2 mmol), $B(C_6F_5)_3$ (5.1 mg, 0.01 mmol) and $(HMe_2SiCH_2)_2$ (35.1 mg, 0.24 mmol) in CH_2Cl_2 (4 mL) at room temperature for 12 h to afford **3b**[16] (62%. The yield was determined by ^1H NMR analysis using dibromomethane as an internal standard).

Procedure B: 2b (30 mg, 0.2 mmol), $B(C_6F_5)_3$ (5.1 mg, 0.01 mmol) and $(HMe_2SiCH_2)_2$ (35.1 mg, 0.24 mmol) in CH_2Cl_2 (4 mL) at room temperature for 12 h to afford **3b** (65%. The yield was determined by ^1H NMR analysis using dibromomethane as an internal standard).

3b : ^1H NMR (400 MHz, $CDCl_3$) δ 7.28 (t, *J* = 8.0, 2H), 6.95-6.89 (m, 3H), 4.04 (q, *J* = 7.2 Hz, 2H), 1.42 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 158.9, 129.459, 120.5, 114.4, 63.3, 14.8.

Preparation of 3c



3c

Using the same procedure as that used for **3a**.

Procedure A: 1c (45.6 mg, 0.2 mmol), $B(C_6F_5)_3$ (5.1 mg, 0.01 mmol) and $(HMe_2SiCH_2)_2$ (35.1 mg, 0.24 mmol) in CH_2Cl_2 (4 mL) at room temperature for 12 h. Purification of the crude product by silica gel flash column chromatography to afford **3c**[17] as a colorless oil (34.4 mg, 81% yield).

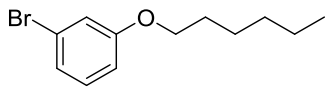
Procedure B: 2c (22.6 mg, 0.1 mmol), $B(C_6F_5)_3$ (2.5 mg, 0.005 mmol) and $(HMe_2SiCH_2)_2$ (17.6

17. I. Chatterjee, D. Porwal and M. Oestreich, *Angew. Chem., Int. Ed.*, 2017, **56**, 3389.

mg, 0.12 mmol) in CH_2Cl_2 (2 mL) at room temperature for 12 h. Purification of the crude product by silica gel flash column chromatography to afford **3c** (17.0 mg, 80% yield).

3c : ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 (d, *J* = 8.8 Hz, 2H), 6.82 (d, *J* = 8.8 Hz, 2H), 3.92 (t, *J* = 6.4 Hz, 2H), 1.80–1.73 (m, 2H), 1.45–1.43 (m, 2H), 1.35–1.33 (m, 4H), 0.91 (t, *J* = 6.4 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 157.7, 129.2, 125.2, 115.7, 68.3, 31.6, 29.2, 25.7, 22.6, 14.0.

Preparation of 3d



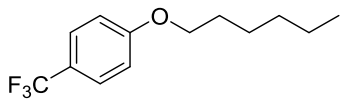
3d

Using the same procedure as that used for **3a**.

Procedure A: 1d (54.4 mg, 0.2 mmol), $B(C_6F_5)_3$ (5.1 mg, 0.01 mmol) and $(HMe_2SiCH_2)_2$ (35.1 mg, 0.24 mmol) in CH_2Cl_2 (4 mL) at room temperature for 12 h. Purification of the crude product by silica gel flash column chromatography to afford **3d**[17] as a colorless oil (42 mg, 82% yield). **Procedure B: 2d** (27 mg, 0.1 mmol), $B(C_6F_5)_3$ (2.5 mg, 0.005 mmol) and $(HMe_2SiCH_2)_2$ (17.6 mg, 0.12 mmol) in CH_2Cl_2 (2 mL) at room temperature for 12 h. Purification of the crude product by silica gel flash column chromatography to afford **3d** (22.3 mg, 87% yield).

3d : ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.13 (t, *J* = 8.0 Hz, 1H), 7.06 (d, *J* = 8.0 Hz, 1H), 7.05 (s, 1H), 6.82 (d, *J* = 8.0 Hz, 1H), 3.93 (t, *J* = 6.8 Hz, 2H), 1.80–1.73 (m, 2H), 1.47–1.43 (m, 2H), 1.35–1.34 (m, 4H), 0.91 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 159.9, 130.4, 123.5, 122.7, 117.7, 113.5, 68.2, 31.5, 29.1, 25.7, 22.6, 14.0.

Preparation of 3e



3e

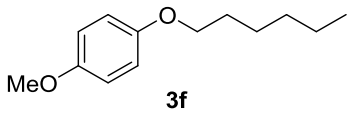
Using the same procedure as that used for **3a**.

Procedure A: 1e (52.4 mg, 0.2 mmol), $B(C_6F_5)_3$ (5.1 mg, 0.01 mmol) and $(HMe_2SiCH_2)_2$ (35.1 mg, 0.24 mmol) in CH_2Cl_2 (4 mL) at room temperature for 12 h. Purification of the crude product by silica gel flash column chromatography to afford **3e**[17] as a colorless oil (37.4 mg, 76% yield). **Procedure B: 2e** (52 mg, 0.2 mmol), $B(C_6F_5)_3$ (5.1 mg, 0.01 mmol) and $(HMe_2SiCH_2)_2$ (35.1 mg,

0.24 mmol) in CH_2Cl_2 (4 mL) at room temperature for 12 h. Purification of the crude product by silica gel flash column chromatography to afford **3e** (35.9 mg, 73% yield).

3e: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 (d, *J* = 8.8 Hz, 2H), 6.95 (d, *J* = 8.8 Hz, 2H), 4.00 (t, *J* = 6.4 Hz, 2H), 1.84–1.77 (m, 2H), 1.49–1.44 (m, 2H), 1.37–1.35 (m, 4H), 0.92 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 161.6, 126.8 (q, J_1 = 3.75 Hz), 124.5 (q, J_2 = 269.55 Hz), 122.6 (q, J_3 = 32.4 Hz), 114.4, 68.2, 31.5, 29.1, 25.7, 22.6, 14.0.

Preparation of 3f



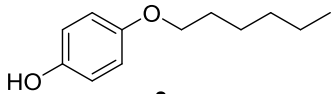
3f

Using the same procedure as that used for **3a**.

Procedure A: 1f (44.8 mg, 0.2 mmol), $B(C_6F_5)_3$ (5.1 mg, 0.01 mmol) and $(HMe_2SiCH_2)_2$ (35.1 mg, 0.24 mmol) in CH_2Cl_2 (4 mL) at room temperature for 12 h. Purification of the crude product by silica gel flash column chromatography to afford **3f**[18] as a colorless oil (24.6 mg, 59% yield). **Procedure B: 2f** (22.2 mg, 0.1 mmol), $B(C_6F_5)_3$ (2.5 mg, 0.005 mmol) and $(HMe_2SiCH_2)_2$ (17.6 mg, 0.12 mmol) in CH_2Cl_2 (2 mL) at room temperature for 12 h. Purification of the crude product by silica gel flash column chromatography to afford **3f** (12.7 mg, 61% yield).

3f: ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.83 (s, 4H), 3.90 (t, *J* = 6.8 Hz, 2H), 3.77 (s, 3H), 1.79–1.72 (m, 2H), 1.46–1.43 (m, 2H), 1.35–1.33 (m, 4H), 0.91 (t, *J* = 6.4 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 153.6, 153.3, 115.4, 114.6, 68.7, 55.7, 31.6, 29.3, 25.7, 22.6, 14.0.

Preparation of 3g



3g

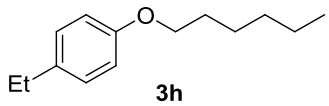
Using the same procedure as that used for **3a**.

Procedure A: 1g (42 mg, 0.2 mmol), $B(C_6F_5)_3$ (5.1 mg, 0.01 mmol) and $(HMe_2SiCH_2)_2$ (70.3 mg, 0.48 mmol) in CH_2Cl_2 (4 mL) at room temperature for 12 h to afford **3g**[17] (36%. The yield was determined by ^{1}H NMR analysis using dibromomethane as an internal standard).

18. Y.-X. Wang, C.-S. Zhou, and R.-H. Wang, *Green Chem.,* 2015, **17**, 3910.

3g: ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.80–6.74 (m, 4H), 4.93 (s, 1H), 3.90 (t, *J* = 6.4 Hz, 2H), 1.79–1.72(m, 2H), 1.46–1.42 (m, 2H), 1.38–1.33 (m, 4H), 0.90 (t, *J* = 6.4 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 153.2, 149.4, 116.0, 115.6, 68.8, 31.6, 29.3, 25.7, 22.6, 14.0.

Preparation of 3h



3h

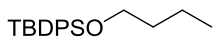
Using the same procedure as that used for **3a**.

Procedure A: 1h (47.2 mg, 0.2 mmol), $B(C_6F_5)_3$ (5.1 mg, 0.01 mmol) and $(HMe_2SiCH_2)_2$ (70.3 mg, 0.48 mmol) in CH_2Cl_2 (4 mL) at room temperature for 12 h. Purification of the crude product by silica gel flash column chromatography to afford **3h** as a colorless oil (33 mg, 80% yield).

Procedure B: 2g (46.8 mg, 0.2 mmol), $B(C_6F_5)_3$ (5.1 mg, 0.01 mmol) and $(HMe_2SiCH_2)_2$ (70.3 mg, 0.48 mmol) in CH_2Cl_2 (4 mL) at room temperature for 12 h. Purification of the crude product by silica gel flash column chromatography to afford **3h** (32.1 mg, 78% yield).

3h: ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.11 (d, *J* = 8.4 Hz, 2H), 6.83 (d, *J* = 8.4 Hz, 2H), 3.94 (t, *J* = 6.6 Hz, 2H), 2.59 (q, *J* = 7.8 Hz, 2H), 1.80–1.75 (m, 2H), 1.47–1.45 (m, 2H), 1.35–1.34 (m, 4H), 1.22 (t, *J* = 7.8 Hz, 3H), 0.91 (t, *J* = 6.6 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 157.2, 136.2, 128.6, 114.4, 68.0, 31.6, 29.3, 28.0, 25.8, 22.6, 15.9, 14.0; IR (neat) cm^{-1} 2958, 2928, 2859, 1612, 1583, 1511, 1468, 1389, 1297, 1238, 1175, 1115, 1032, 938, 826, 801, 750; HRMS (ESI-TOF, m/z) calcd for $C_{14}H_{22}O$ $(M+Na)^+$: 229.1563, found 229.1568.

Preparation of 3i



3i

Using the same procedure as that used for **3a**.

Procedure A: 1i (65.6 mg, 0.2 mmol), $B(C_6F_5)_3$ (5.1 mg, 0.01 mmol) and $(HMe_2SiCH_2)_2$ (35.1 mg, 0.24 mmol) in CH_2Cl_2 (4 mL) at room temperature for 12 h. Purification of the crude product by silica gel flash column chromatography to afford **3i**[19] as a colorless oil (52.4 mg, 84% yield).

Procedure B: 2h (65 mg, 0.2 mmol), $B(C_6F_5)_3$ (5.1 mg, 0.01 mmol) and $(HMe_2SiCH_2)_2$ (35.1 mg,

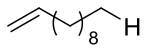
19. R. R. Hill and S. D. Rychnovsky, *J. Org. Chem.,* 2016, **81**, 10707.

0.24 mmol) in CH_2Cl_2 (4 mL) at room temperature for 12 h. Purification of the crude product by silica gel flash column chromatography to afford **3i** (53.7 mg, 86% yield).

Procedure C: 2l (35.6 mg, 0.1 mmol), $B(C_6F_5)_3$ (2.5 mg, 0.005 mmol) and $(HMe_2SiCH_2)_2$ (17.6 mg, 0.12 mmol) in CH_2Cl_2 (2 mL) at room temperature for 12 h. Purification of the crude product by silica gel flash column chromatography to afford **3i** (25.3 mg, 81% yield).

3i : ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, *J* = 6.4 Hz, 4H), 7.45–7.37 (m, 6H), 3.68 (t, *J* = 6.4 Hz, 2H), 1.60–1.53 (m, 2H), 1.45–1.35 (m, 2H), 1.06 (s, 9H), 0.90 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 135.6, 134.2, 129.5, 127.5, 63.7, 34.8, 26.9, 19.2, 19.0, 13.9.

Preparation of 3k



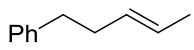
3k

Using the same procedure as that used for **3a**.

Procedure A: 1k (34 mg, 0.2 mmol), $B(C_6F_5)_3$ (5.1 mg, 0.01 mmol) and $(HMe_2SiCH_2)_2$ (35.1 mg, 0.24 mmol) in CH_2Cl_2 (4 mL) at room temperature for 12 h to afford **3k**[20] (73%. The yield was determined by ^{1}H NMR analysis using 1,3,5-trimethoxybenzene as an internal standard).

3k: ^{1}H NMR (600 MHz, $CDCl_3$) δ 5.85–5.78 (m, 1H), 5.00 (d, *J* = 16.8 Hz, 1H), 4.93 (d, *J* = 9.6 Hz, 1H), 2.04 (q, *J* = 6.6 Hz, 2H),1.39–1.27 (m, 14H), 0.88 (t, *J* = 6.6 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 139.3, 114.1, 33.8, 31.9, 29.6, 29.5, 29.3, 29.2, 29.0, 22.7, 14.1.

Preparation of 3l



3l

Using the same procedure as that used for **3a**.

Procedure A: 1l (32.4 mg, 0.2 mmol), $B(C_6F_5)_3$ (5.1 mg, 0.01 mmol) and $(HMe_2SiCH_2)_2$ (35.1 mg, 0.24 mmol) in CH_2Cl_2 (4 mL) at room temperature for 12 h to afford **3l**[21] (80%. The yield was determined by ^{1}H NMR analysis using dibromomethane as an internal standard).

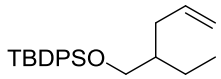
Procedure B: 2j (32 mg, 0.2 mmol), $B(C_6F_5)_3$ (5.1 mg, 0.01 mmol) and $(HMe_2SiCH_2)_2$ (35.1 mg,

20. A. Chatterjee, S. H. Hopen Eliasson, K. W. Törnroos and V. R. Jensen, *ACS Catal.*, 2016, **6**, 7784.
21. M. Movassaghi and O. K. Ahmad, *J. Org. Chem.*, 2007, **72**, 1838.

0.24 mmol) in CH_2Cl_2 (4 mL) at room temperature for 12 h to afford **3l** (70%. The yield was determined by ^1H NMR analysis using dibromomethane as an internal standard).

3l: ^1H NMR (600 MHz, $CDCl_3$) δ 7.29 (t, *J* = 7.6 Hz, 2H), 7.20–7.18 (m, 3H), 5.52–5.44 (m, 2H), 2.67 (t, *J* = 7.6 Hz, 2H), 2.33–2.25 (m, 2H), 1.59 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 142.2, 130.6, 128.4, 128.2, 125.7, 125.4, 36.1, 34.4, 17.9.

Preparation of 3m



3m

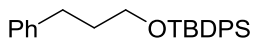
Using the same procedure as that used for **3a**.

Procedure A: 1m (73.6 mg, 0.2 mmol), $B(C_6F_5)_3$ (5.1 mg, 0.01 mmol) and $(HMe_2SiCH_2)_2$ (35.1 mg, 0.24 mmol) in CH_2Cl_2 (4 mL) at room temperature for 12 h. Purification of the crude product by silica gel flash column chromatography to afford **3m**[22] as a colorless oil (51.4 mg, 73% yield).

Procedure B: 2i (36.1 mg, 0.1 mmol), $B(C_6F_5)_3$ (2.5 mg, 0.005 mmol) and $(HMe_2SiCH_2)_2$ (17.6 mg, 0.12 mmol) in CH_2Cl_2 (2 mL) at room temperature for 12 h. Purification of the crude product by silica gel flash column chromatography to afford **3m** (26 mg, 74% yield).

3m: ^1H NMR (600 MHz, $CDCl_3$) δ 7.68 (d, *J* = 6.6 Hz, 4H), 7.44–7.37 (m, 6H), 5.80–5.72 (m, 1H), 5.01 (d, *J* = 16.8 Hz, 1H), 4.97 (d, *J* = 10.2 Hz, 1H), 3.58–3.55 (m, 2H), 2.24–2.20 (m, 1H), 2.12–2.08 (m, 1H), 1.54–1.51 (m, 1H), 1.44–1.33 (m, 2H), 1.07 (s, 9H), 0.84 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 137.3, 135.6, 134.0, 129.5, 127.5, 115.7, 65.4, 42.2, 35.2, 26.9, 23.2, 19.3, 11.3.

Preparation of 3n



3n

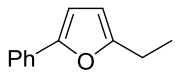
Using the same procedure as that used for **3a**.

Procedure A: 1n (78 mg, 0.2 mmol), $B(C_6F_5)_3$ (5.1 mg, 0.01 mmol) and $(HMe_2SiCH_2)_2$ (35.1 mg, 0.24 mmol) in CH_2Cl_2 (4 mL) at room temperature for 12 h to afford **3n**[17] (70%. The yield was determined by ^1H NMR analysis using dibromomethane as an internal standard).

22. L. A. Paquette, M. Duan, I. Konetzki and C. Kempmann, *J. Am. Chem. Soc.*, 2002, **124**, 4257.

3n : ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 (d, *J* = 7.2 Hz, 4H), 7.42–7.35 (m, 6H), 7.28–7.24 (m, 2H), 7.17–7.15 (m, 3H), 3.69 (t, *J* = 6.4 Hz, 2H), 2.72 (t, *J* = 7.6 Hz, 2H), 1.91–1.84 (m, 2H), 1.07 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 142.2, 135.6, 134.0, 129.5, 128.5, 128.3, 127.6, 125.6, 63.1, 34.2, 32.1, 26.9, 19.3.

Preparation of 3p



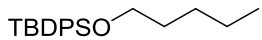
3p

Using the same procedure as that used for **3a**.

Procedure A: 1p (38 mg, 0.2 mmol), $B(C_6F_5)_3$ (5.1 mg, 0.01 mmol) and $(HMe_2SiCH_2)_2$ (35.1 mg, 0.24 mmol) in CH_2Cl_2 (4 mL) at room temperature for 12 h. Purification of the crude product by silica gel flash column chromatography to afford **3p**[23] as a colorless oil (11.9 mg, 35% yield).

3p : ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (d, *J* = 7.6 Hz, 2H), 7.36 (t, *J* = 7.6 Hz, 2H), 7.21 (t, *J* = 7.2 Hz, 1H), 6.55 (d, *J* = 3.2 Hz, 1H), 6.06 (d, *J* = 3.2 Hz, 1H), 2.72 (q, *J* = 7.6 Hz, 2H), 1.29 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 157.6, 152.1, 131.2, 128.6, 126.7, 123.3, 106.1, 105.6, 21.5, 12.2.

Preparation of 3q



3q

Using the same procedure as that used for **3a**.

Procedure A: 1q (51.3 mg, 0.15 mmol), $B(C_6F_5)_3$ (3.8 mg, 0.0075 mmol) and $(HMe_2SiCH_2)_2$ (26.3 mg, 0.18 mmol) in CH_2Cl_2 (3 mL) at room temperature for 12 h. Purification of the crude product by silica gel flash column chromatography to afford **3q**[24] as a colorless oil (31.8 mg, 65% yield).

Procedure B: 2k (51 mg, 0.15 mmol), $B(C_6F_5)_3$ (3.8 mg, 0.0075 mmol) and $(HMe_2SiCH_2)_2$ (26.3 mg, 0.18 mmol) in CH_2Cl_2 (3 mL) at room temperature for 12 h. Purification of the crude product by silica gel flash column chromatography to afford **3q** (33.9 mg, 69% yield).

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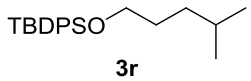
23. J. Izquierdo, S. Rodríguez and F. V. González, *Org. Lett.*, 2011, **13**, 3856.

24. X.-S. Ma and S. B. Herzon, *Chem. Sci.*, 2015, **6**, 6250.

3q: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, *J* = 6.4 Hz, 4H), 7.45–7.37 (m, 6H), 3.67 (t, *J* = 6.8 Hz, 2H), 1.61–1.55 (m, 2H), 1.32–1.31 (m, 4H), 1.06 (s, 9H), 0.89 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 135.6, 134.2, 129.4, 127.5, 64.0, 32.3, 28.0, 26.9, 22.4, 19.2, 14.1.

Preparation of 3r



3r

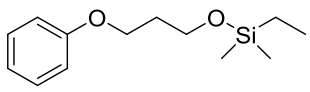
Using the same procedure as that used for **3a**.

Procedure A: 1r (53.4 mg, 0.15 mmol), $B(C_6F_5)_3$ (3.8 mg, 0.0075 mmol) and $(HMe_2SiCH_2)_2$ (26.3 mg, 0.18 mmol) in CH_2Cl_2 (3 mL) at room temperature for 12 h. Purification of the crude product by silica gel flash column chromatography to afford **3r** as a colorless oil (36.1 mg, 71% yield).

3r: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, *J* = 6.4 Hz, 4H), 7.45–7.37 (m, 6H), 3.65 (t, *J* = 6.8 Hz, 2H), 1.59–1.50 (m, 3H), 1.27–1.19 (m, 2H), 1.06 (s, 9H), 0.87 (d, *J* = 6.8 Hz, 6H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 135.6, 134.2, 129.5, 127.5, 64.3, 35.0, 30.4, 27.7, 26.9, 22.6, 19.2; IR (neat) cm^{-1} 2954, 2930, 2896, 2857, 1589, 1471, 1427, 1386, 1304, 1105, 1090, 1008, 938, 822; HRMS (ESI-TOF, m/z) calcd for $C_{22}H_{32}NaOSi$ $(M+Na)^+$: 363.2115, found 363.2114.

2.5 Preparations and Spectral Data of 4a and 4b

Preparation of 4a

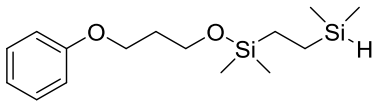


4a

To a solution of **1a** (315 mg, 2.1 mmol), Et_3N (0.36 mL, 2.6 mmol) and DMAP (21 mg, 0.17 mmol) in CH_2Cl_2 (5 mL) was added chlorodimethylethylsilane (210 g, 1.7 mmol) at rt. The reaction mixture was then stirred for 30 min before quenched with sat. NH_4Cl (3 mL). The mixture was extracted with CH_2Cl_2 (3 × 5 mL) and the combined organic layers were then dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by silica gel flash column chromatography (gradient eluent: 0-2% of EtOAc/petroleum ether) to afford **4a** as a colorless liquid (235 mg, 58% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 (t, *J* = 7.8 Hz, 2H), 6.95–6.90 (m, 3H), 4.06 (t, *J* = 6.0 Hz, 2H), 3.79 (t, *J* = 6.0 Hz, 2H), 2.03–1.97 (m, 2H), 0.94 (t, *J* = 8.0 Hz, 3H), 0.58 (q, *J* = 8.0 Hz, 2H), 0.08 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.0, 129.4, 120.5, 114.4, 64.3,

59.0, 32.3, 7.9, 6.7, -2.8; IR (neat) cm^{-1} 2954, 2875, 1601, 1497, 1470, 1395, 1300, 1246, 1172, 1087, 1014, 965, 835, 784, 752, 690; HRMS (ESI-TOF, m/z) calcd for $C_{13}H_{22}NaO_2Si$ $(M+Na)^+$: 261.1281, found 261.1284.

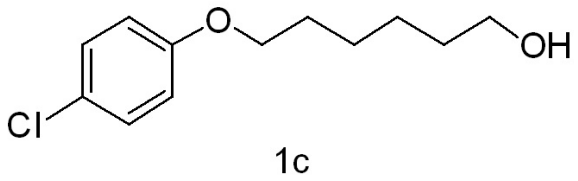
Preparation of 4b



4b

Using the same procedure as that used for **4a** afforded **4b** as a colorless liquid (355 mg, 60% yield). ^1H NMR (600 MHz, $CDCl_3$) δ 7.28 (t, *J* = 7.8 Hz, 2H), 6.95–6.90 (m, 3H), 4.06 (t, *J* = 6.0 Hz, 2H), 3.83–3.81 (m, 1H), 3.79 (t, *J* = 6.0 Hz, 2H), 2.02–1.98 (m, 2H), 0.54–0.49 (m, 4H), 0.09 (s, 6H), 0.05 (d, *J* = 3.6 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.0, 129.4, 120.5, 114.4, 64.2, 59.1, 32.3, 8.8, 5.6, -2.8, -4.9; IR (neat) cm^{-1} 2955, 2875, 2320, 2106, 1601, 1497, 1470, 1403, 1300, 1245, 1172, 1085, 1018, 968, 886, 832, 773, 752, 690; HRMS (ESI-TOF, m/z) calcd for $C_{15}H_{28}NaO_2Si_2$ $(M+Na)^+$: 319.1520, found 319.1520.

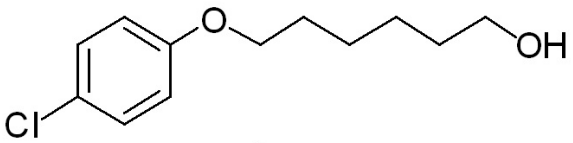
YWY-4-45B1 H1
CDCl3 400MHz



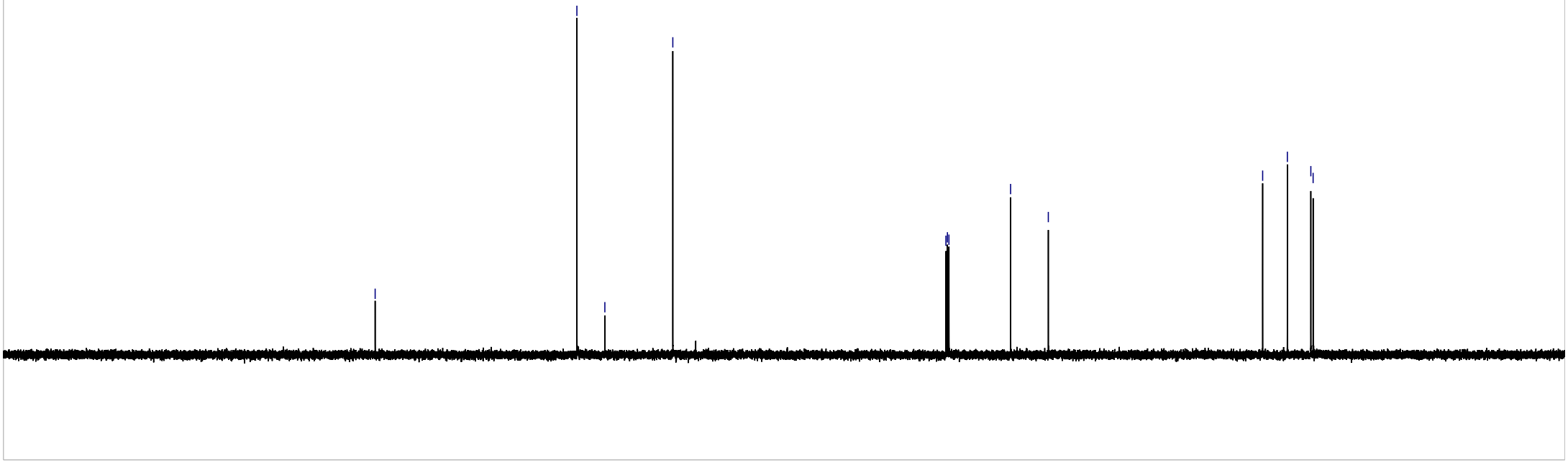
1c

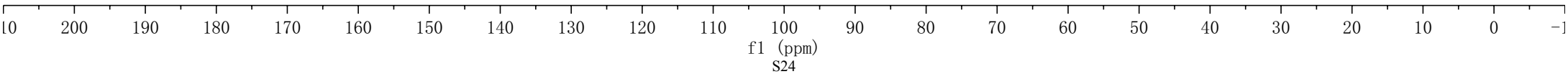
YWY-4-45B1 C13
CDCl3 150MHz

157.622
129.203
125.263
115.688
77.212
77.000
76.788
68.102
62.787
32.591
29.109
25.802
25.481

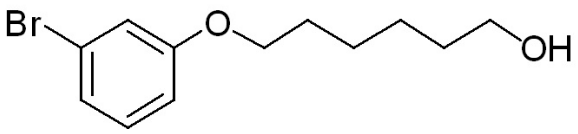


1c





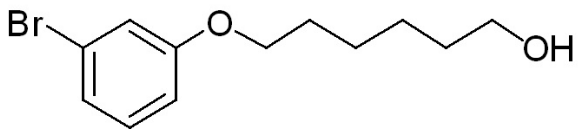
YWY-4-45B2 H1
CDCl3 400MHz



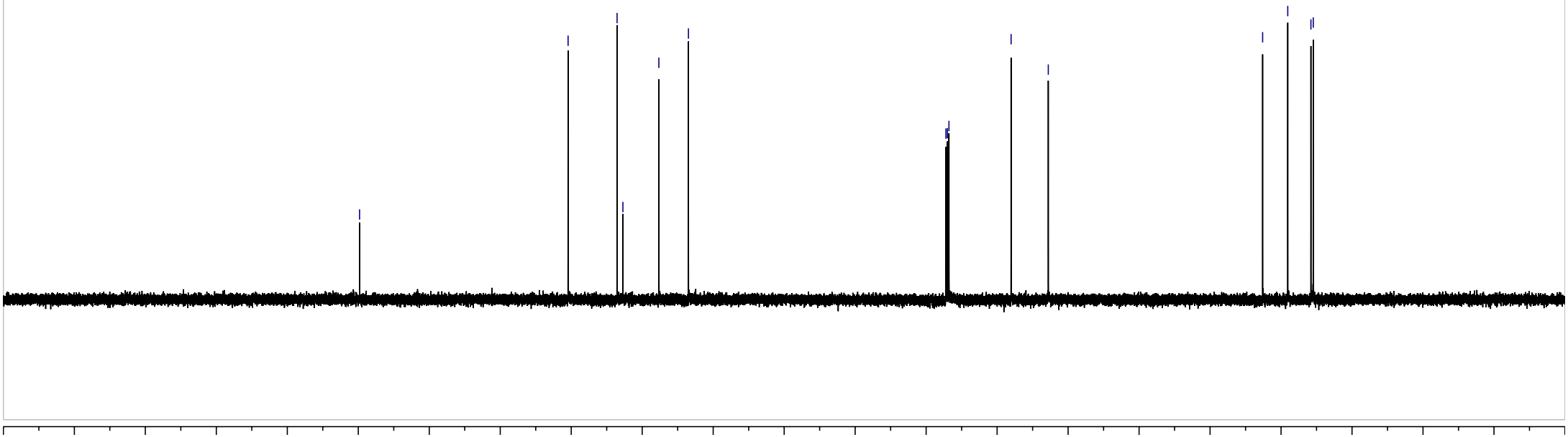
1d

YWY-4-45B2 C13
CDCl3 150MHz

159.818
130.442
123.540
122.718
117.654
113.486
77.211
77.000
76.787
68.031
62.792
32.593
29.054
25.789
25.460

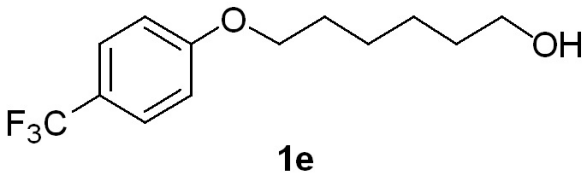


1d



f1 (ppm)

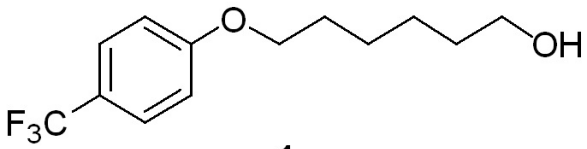
YWY-4-68B1-2 H1
CDCl3 400MHz



1e

YWY-4-68B1-2 C13
CDCl3 150MHz

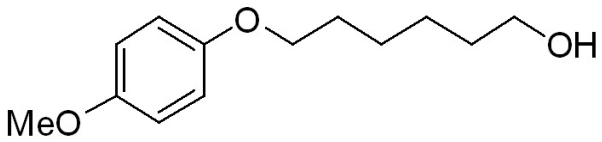
161.488
127.150
126.824
126.799
126.774
126.749
125.339
123.556
122.899
122.684
122.467
122.260
121.740
114.358
77.212
77.000
76.788
68.026
62.765
32.576
29.021
25.788
25.476



1e

f1 (ppm)

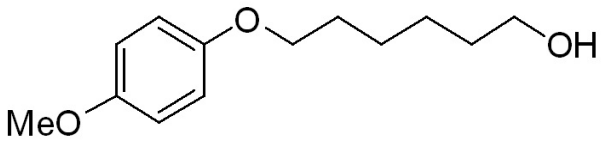
YWY-4-61P H1
CDCl3 400MHz



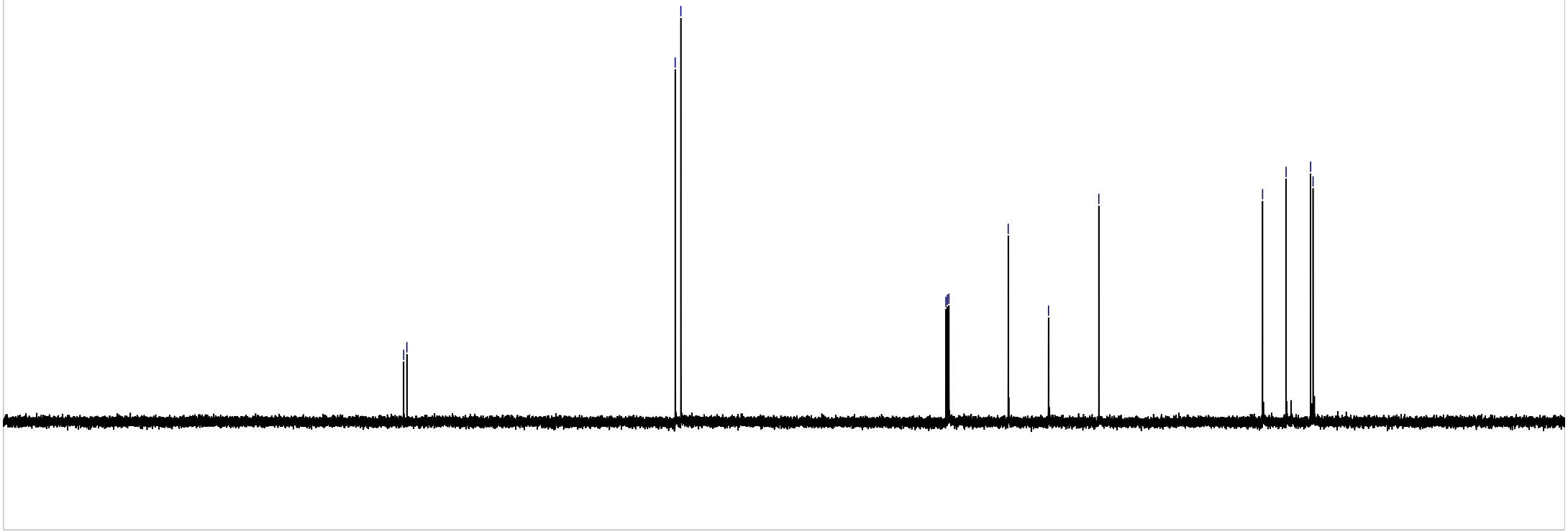
1f

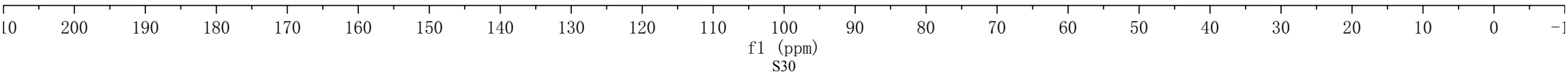
YWY-4-61P C13
CDCl3 150MHz

153.600
153.154
115.356
114.554
77.212
77.000
76.788
68.438
62.759
55.673
32.602
29.284
25.832
25.490

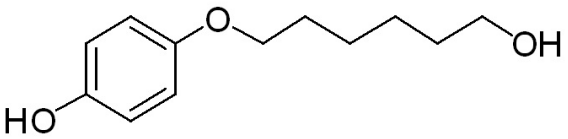


1f





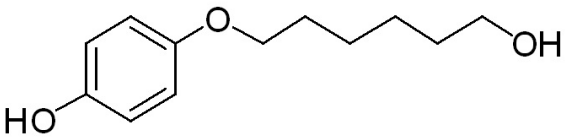
YWY-4-97P-2 H1
CDCl3 400MHz



1g

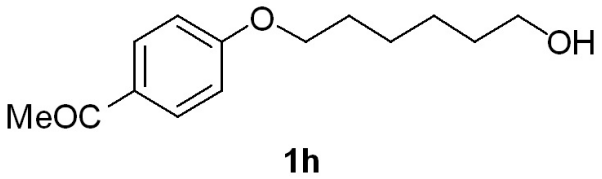
YWY-4-97P-1 C13
CDCl3 150MHz

153.216
149.414
115.969
115.573
77.211
77.000
76.788
68.516
62.938
32.651
29.322
25.875
25.522



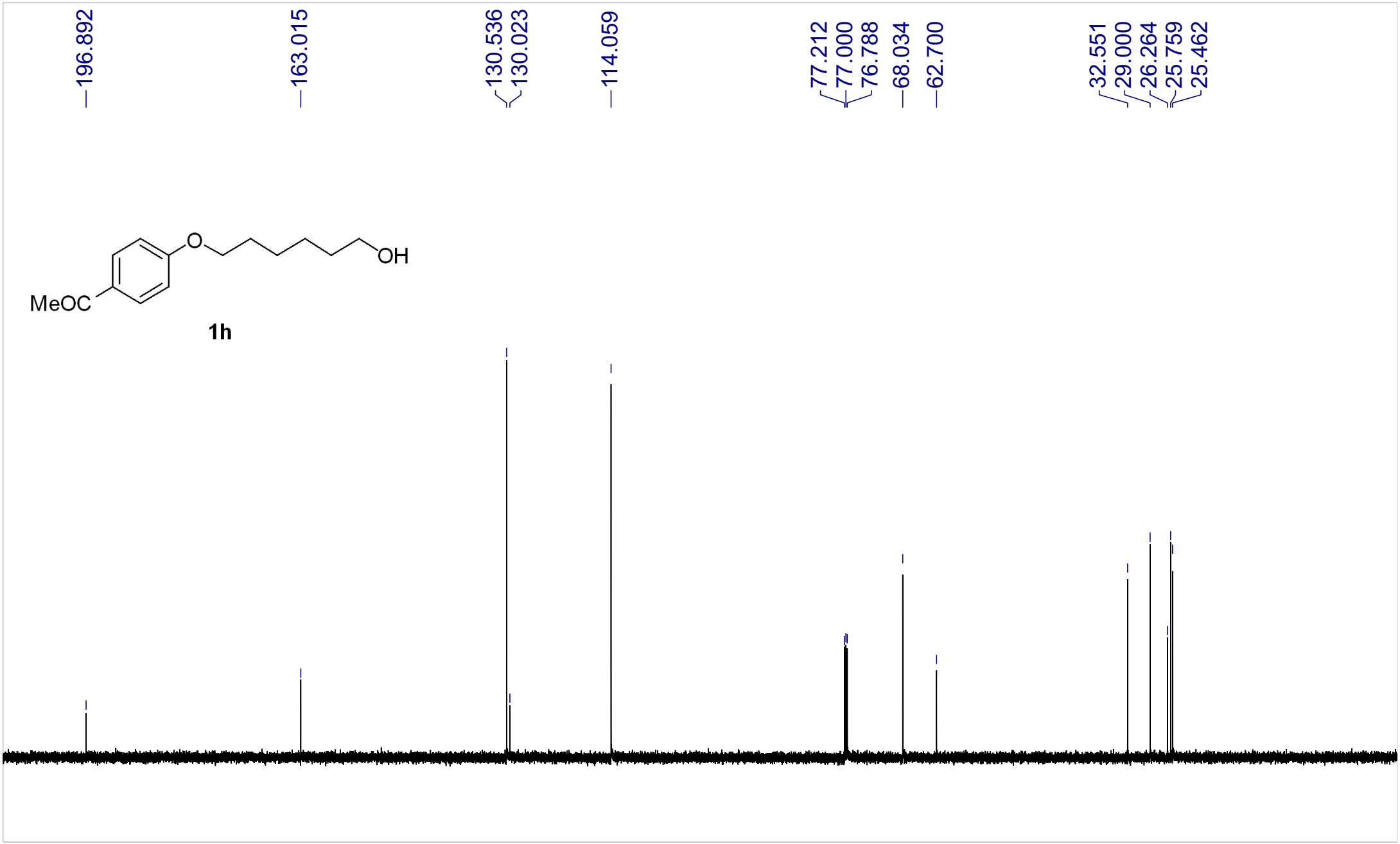
1g

YWY-4-53P H1
CDCl3 400MHz

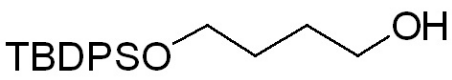


1h

YWY-4-53P C13
CDCl3 150MHz

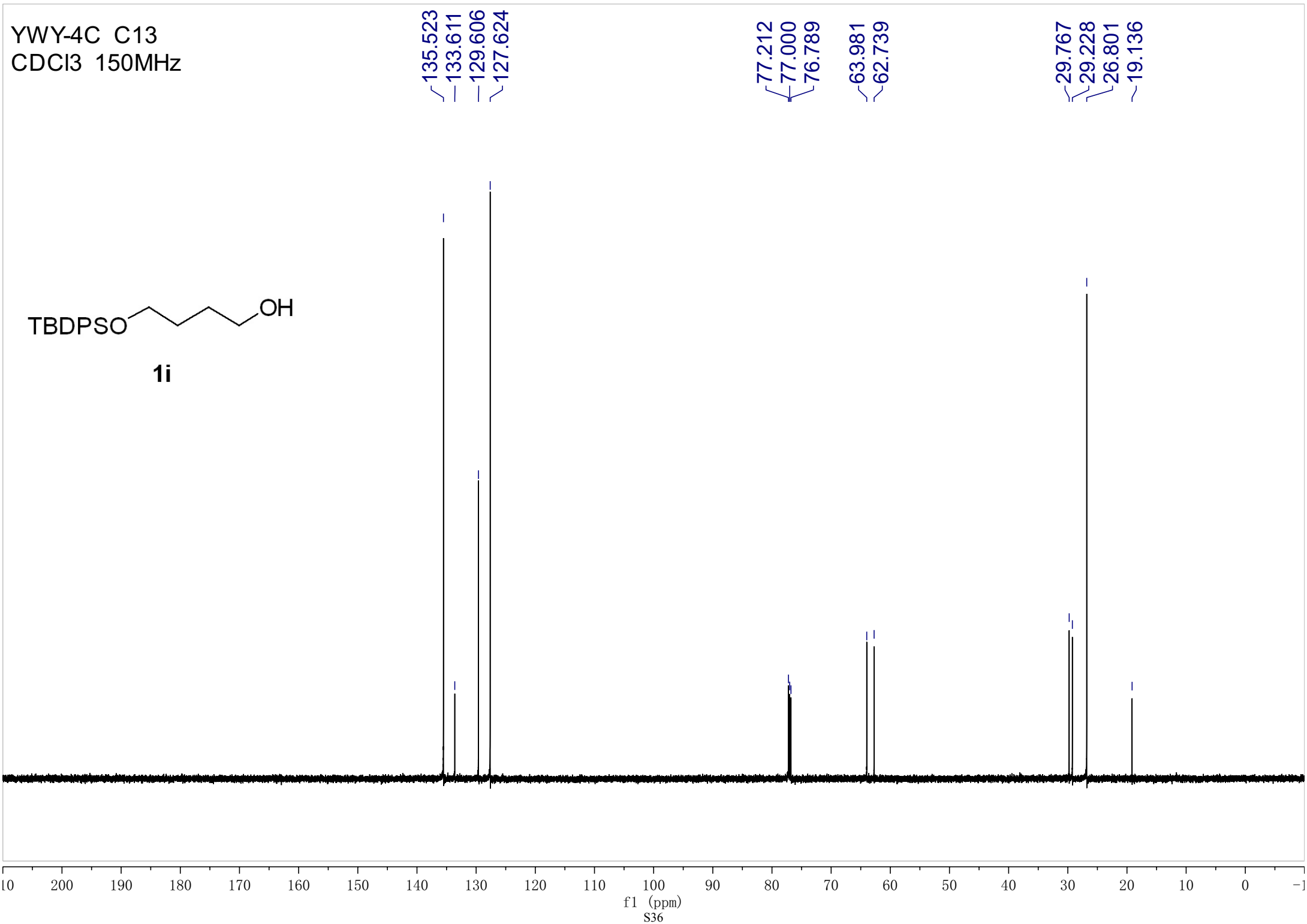


YWY-4C H1
CDCl3 600MHz

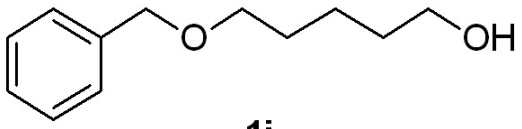


1i

YWY-4C C13
CDCl3 150MHz

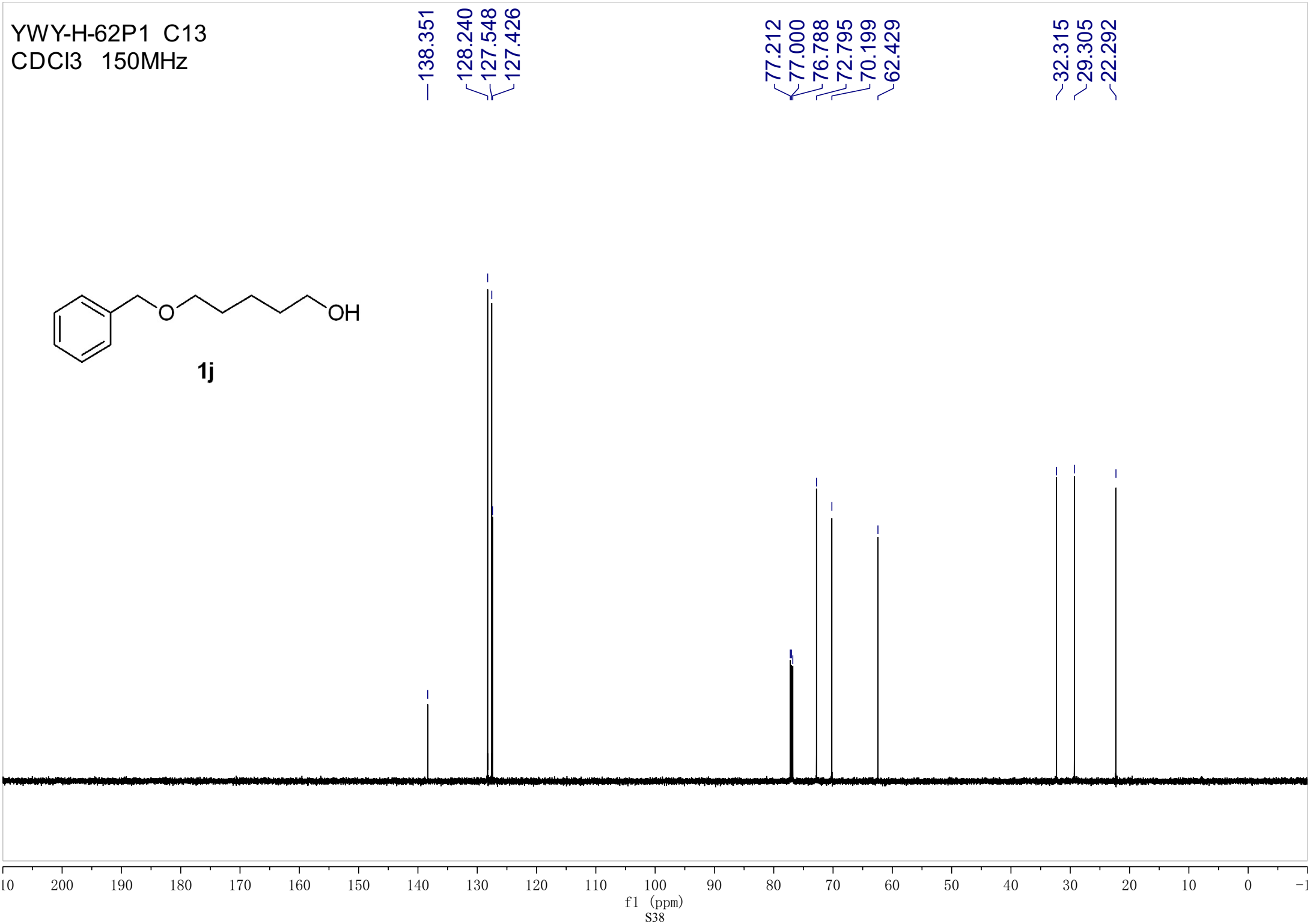


YWY-H-62P1 H1
CDCl3 400MHz

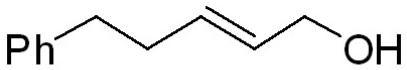


1j

YWY-H-62P1 C13
CDCl3 150MHz



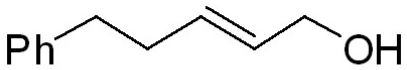
YWY-4-16P2 H1
CDCl3 400MHz



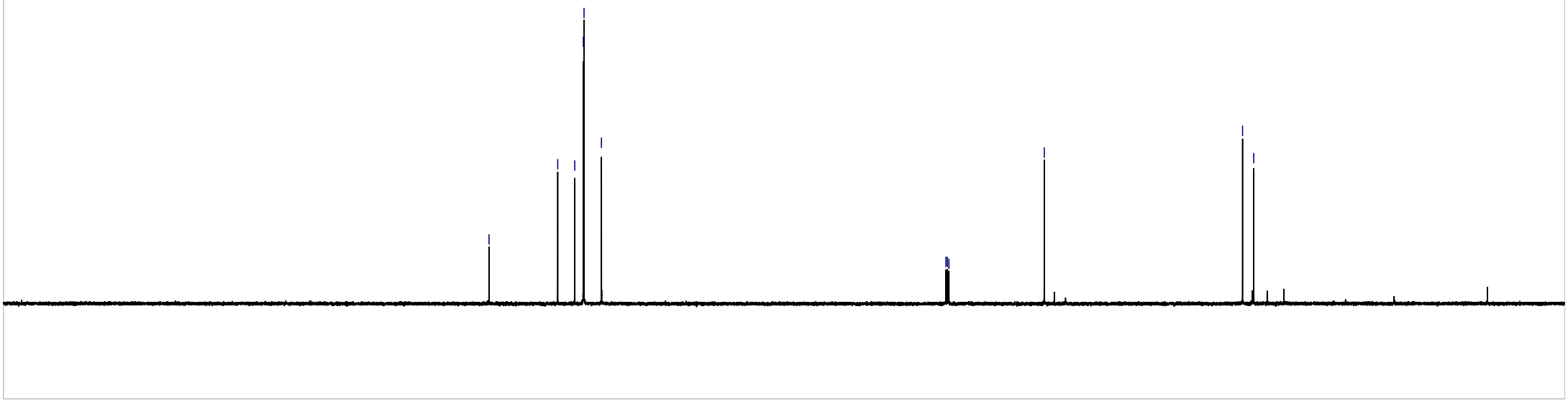
1l

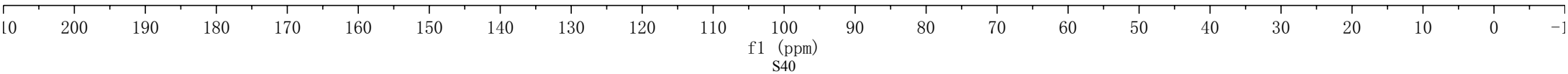
YWY-4-16P2 C13
CDCl3 150MHz

141.579
131.899
129.504
128.290
128.195
125.743
77.212
77.000
76.788
63.354
35.418
33.845

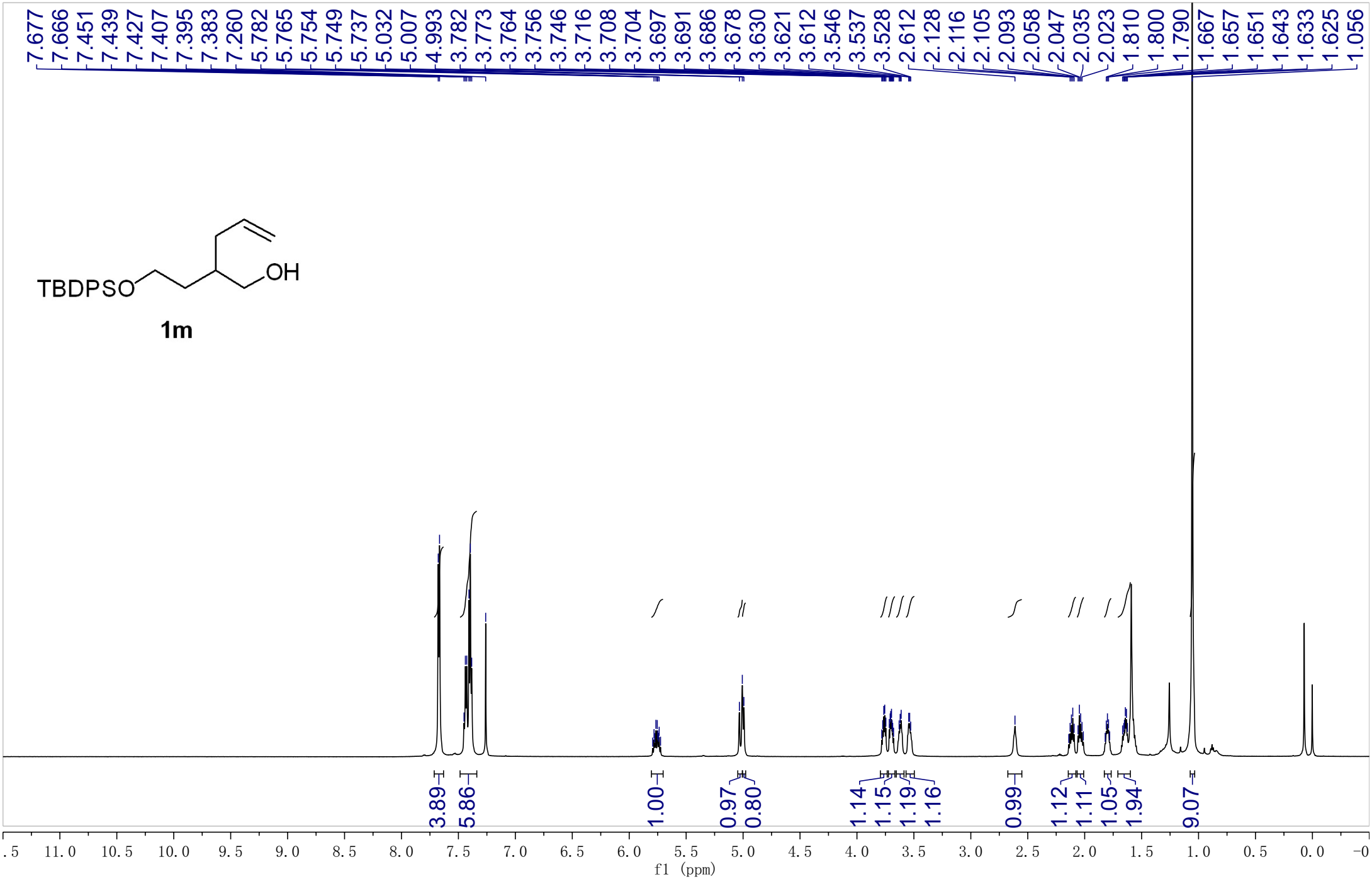


1l



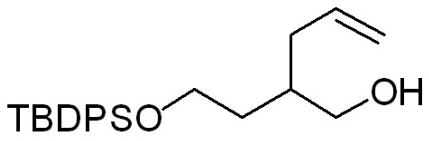


YWY-4-59P1 H1
CDCl3 600MHz



YWY-4-59P C13
CDCl3 150MHz

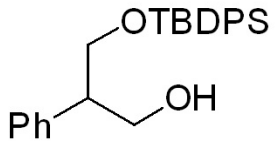
136.802
135.539
133.245
129.728
127.700
116.298
77.211
77.000
76.787
65.792
62.579
38.705
36.201
34.304
26.778
19.079



1m

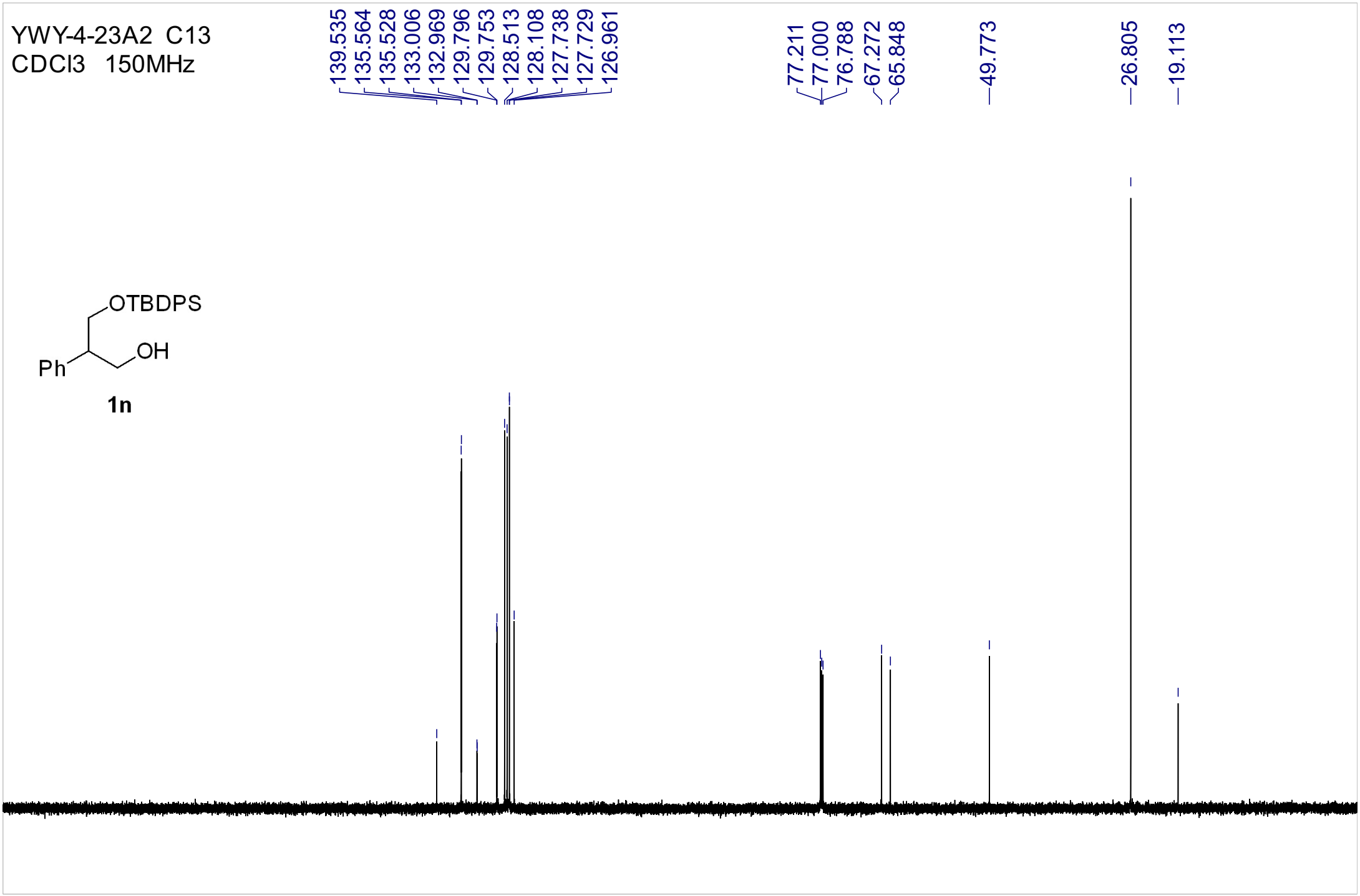
f1 (ppm)

YWY-4-23A2 H1
CDCl3 400MHz

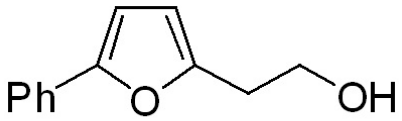


1n

YWY-4-23A2 C13
CDCl3 150MHz

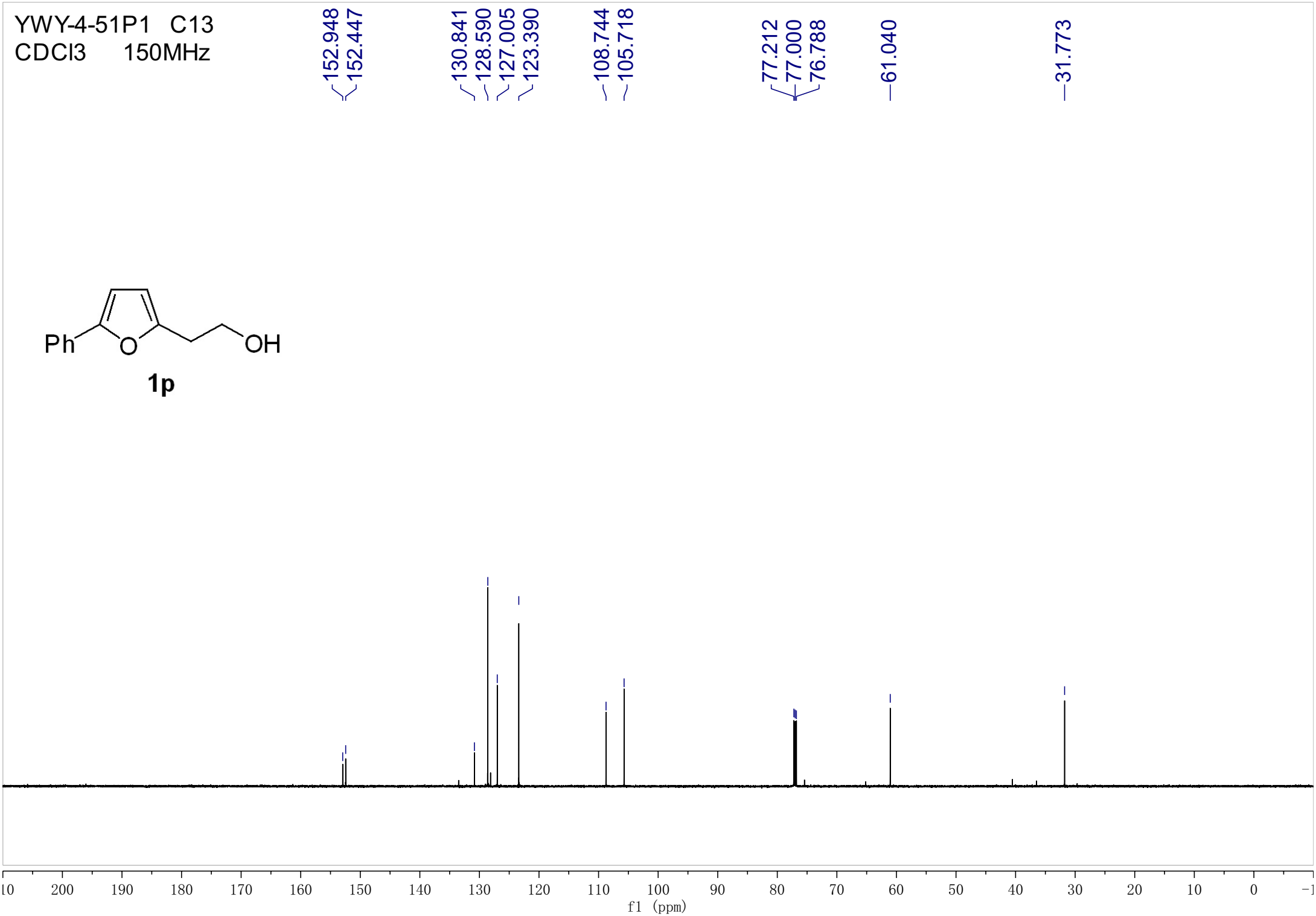


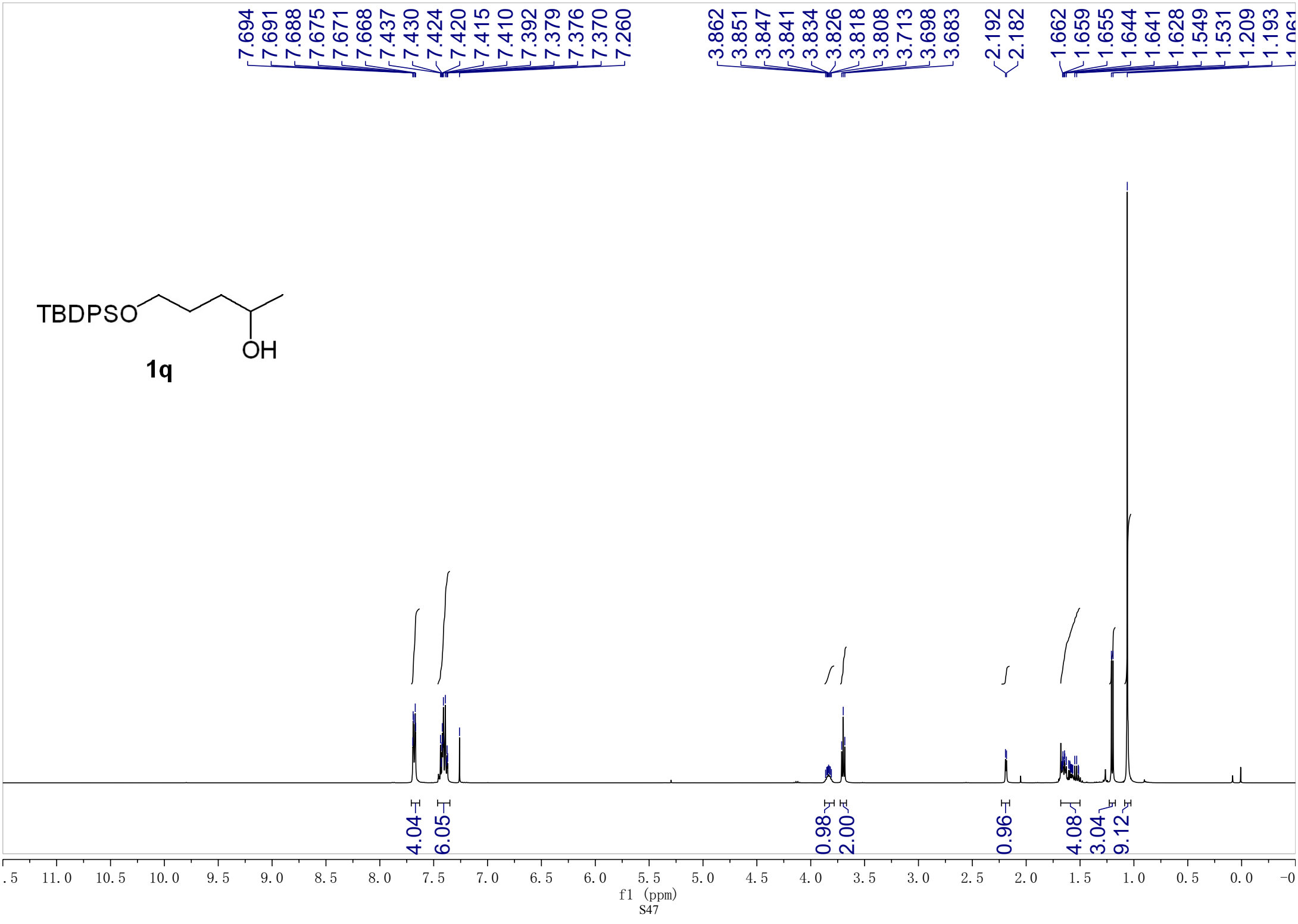
YWY-4-51P1 H1
CDCl3 400MHz



1p

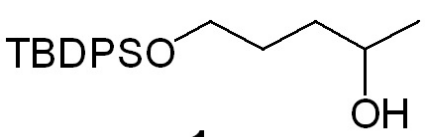
YWY-4-51P1 C13
CDCl3 150MHz




7.694
7.691
7.688
7.675
7.671
7.668
7.437
7.430
7.424
7.420
7.415
7.410
7.392
7.379
7.376
7.370
7.260
3.862
3.851
3.847
3.841
3.834
3.826
3.818
3.808
3.713
3.698
3.683
2.192
2.182
1.662
1.659
1.655
1.644
1.641
1.628
1.549
1.531
1.209
1.193
TBDPSO
OH
1q
4.04
6.05
0.98
2.00
0.96
4.08
3.04
9.12
f1 (ppm)

YWY-5-13A1-1 C13
CDCl3 150MHz

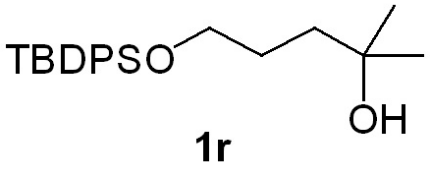
135.552
133.610
129.614
127.634
77.212
77.000
76.788
67.806
64.201
36.258
28.930
26.806
23.438
19.141



1q

f1 (ppm)

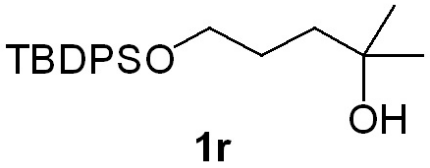
YWY-5-14P H1
CDCl3 400MHz



1r

YWY-5-14P C13
CDCl3 100MHz

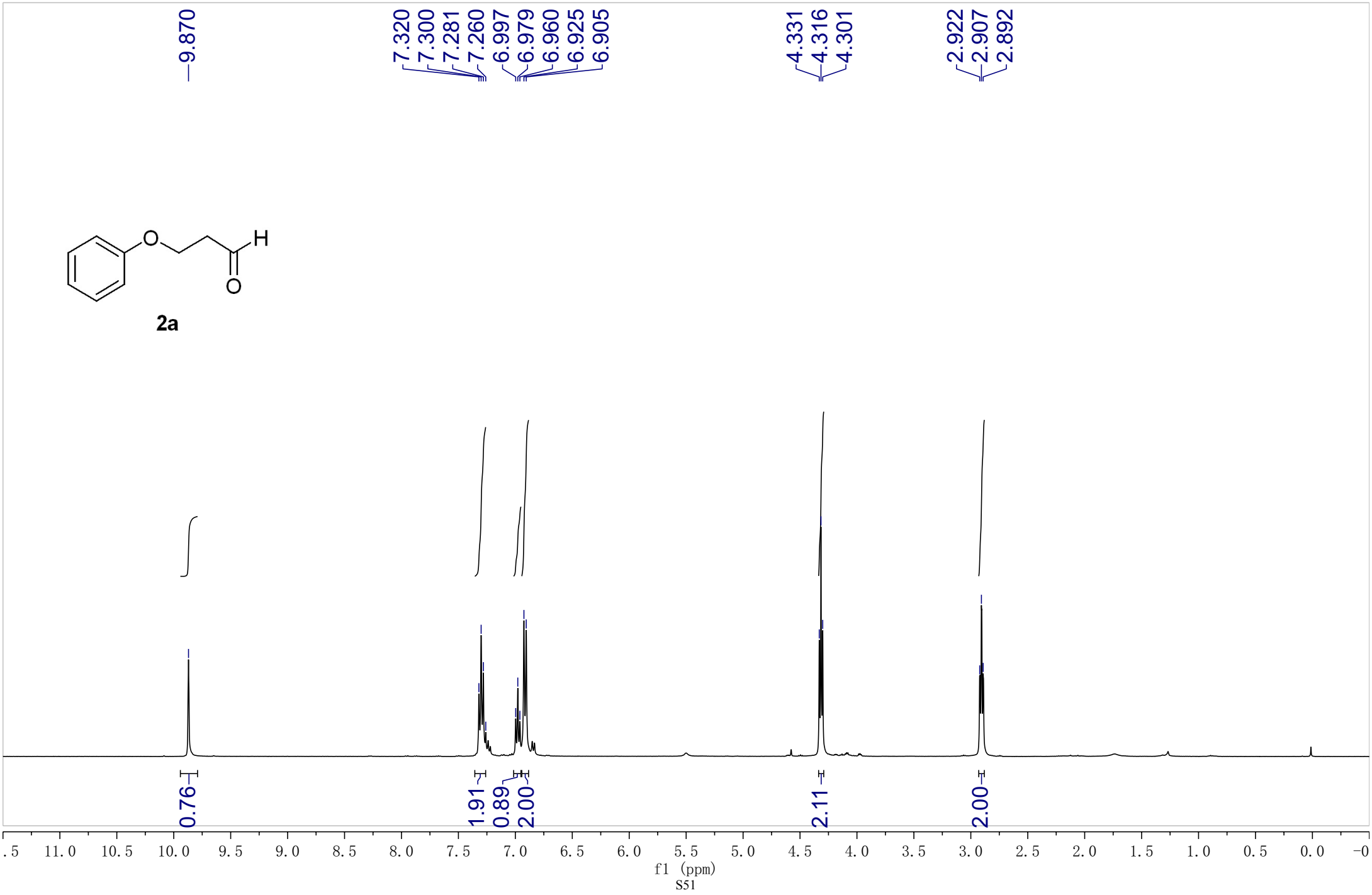
135.561
133.686
129.597
127.626
77.318
77.000
76.683
70.494
64.554
40.506
29.276
27.481
26.813
19.152



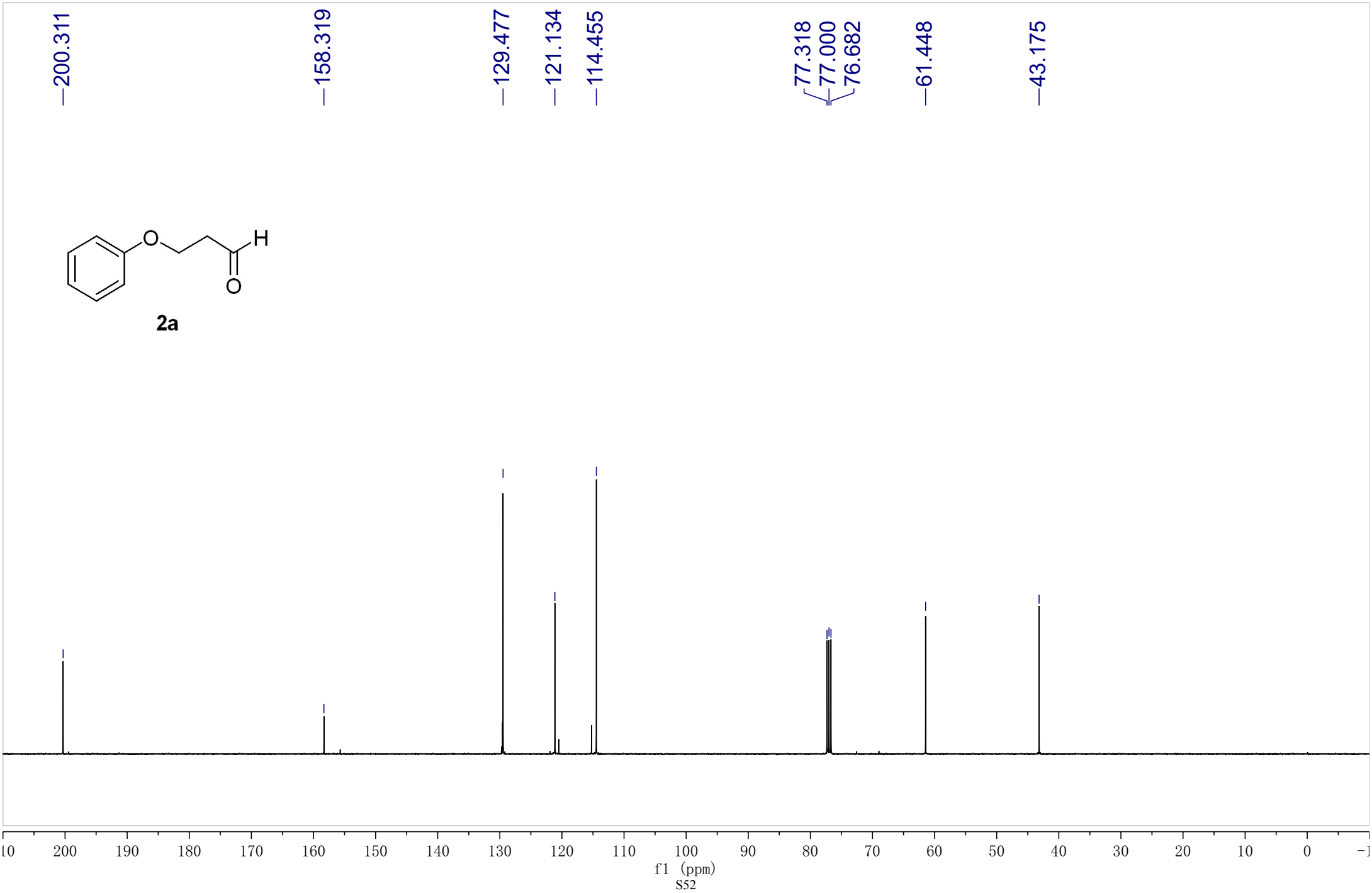
1r

f1 (ppm)

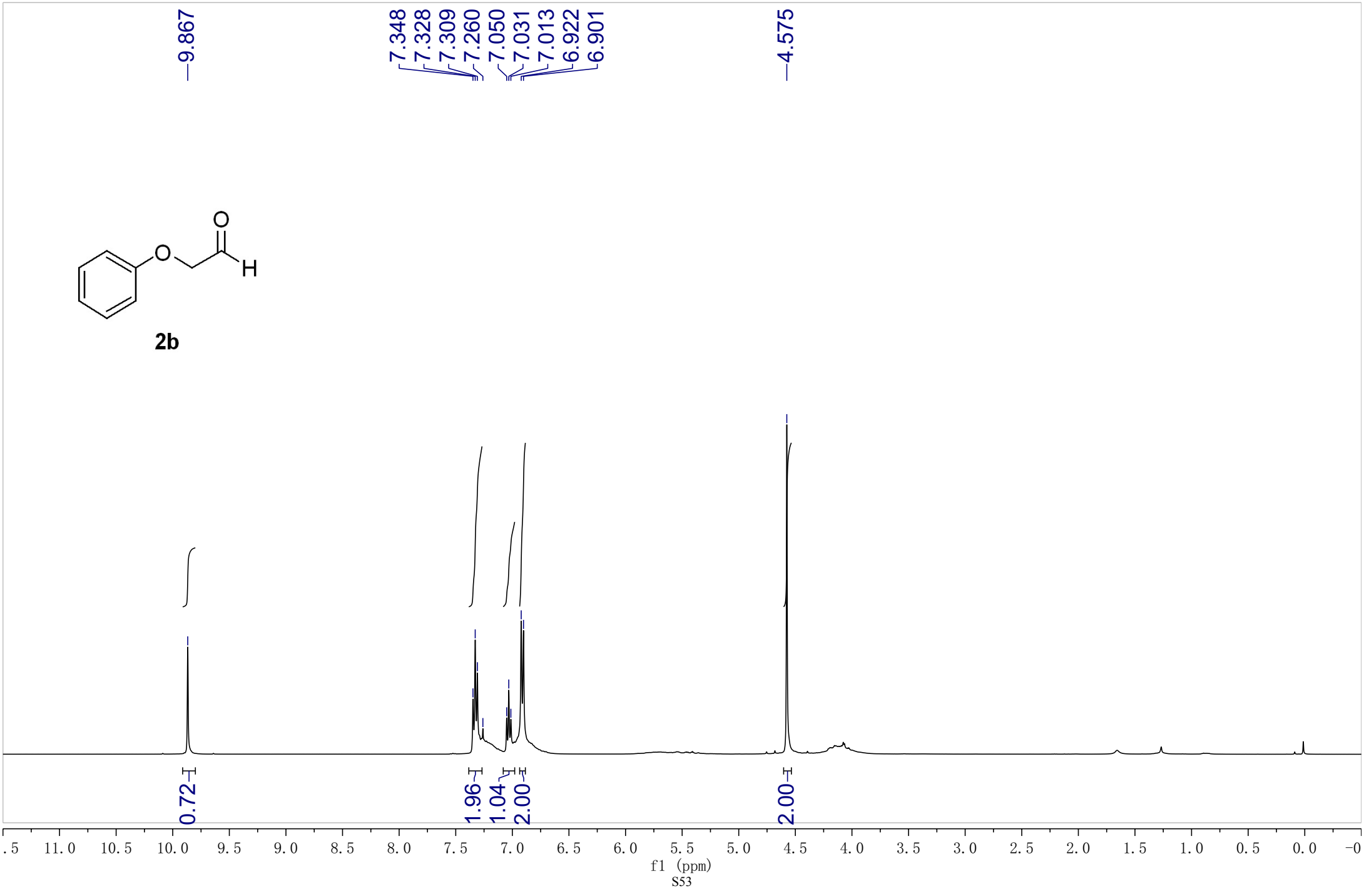
YWY-4-105A2 H1
CDCl3 400MHz



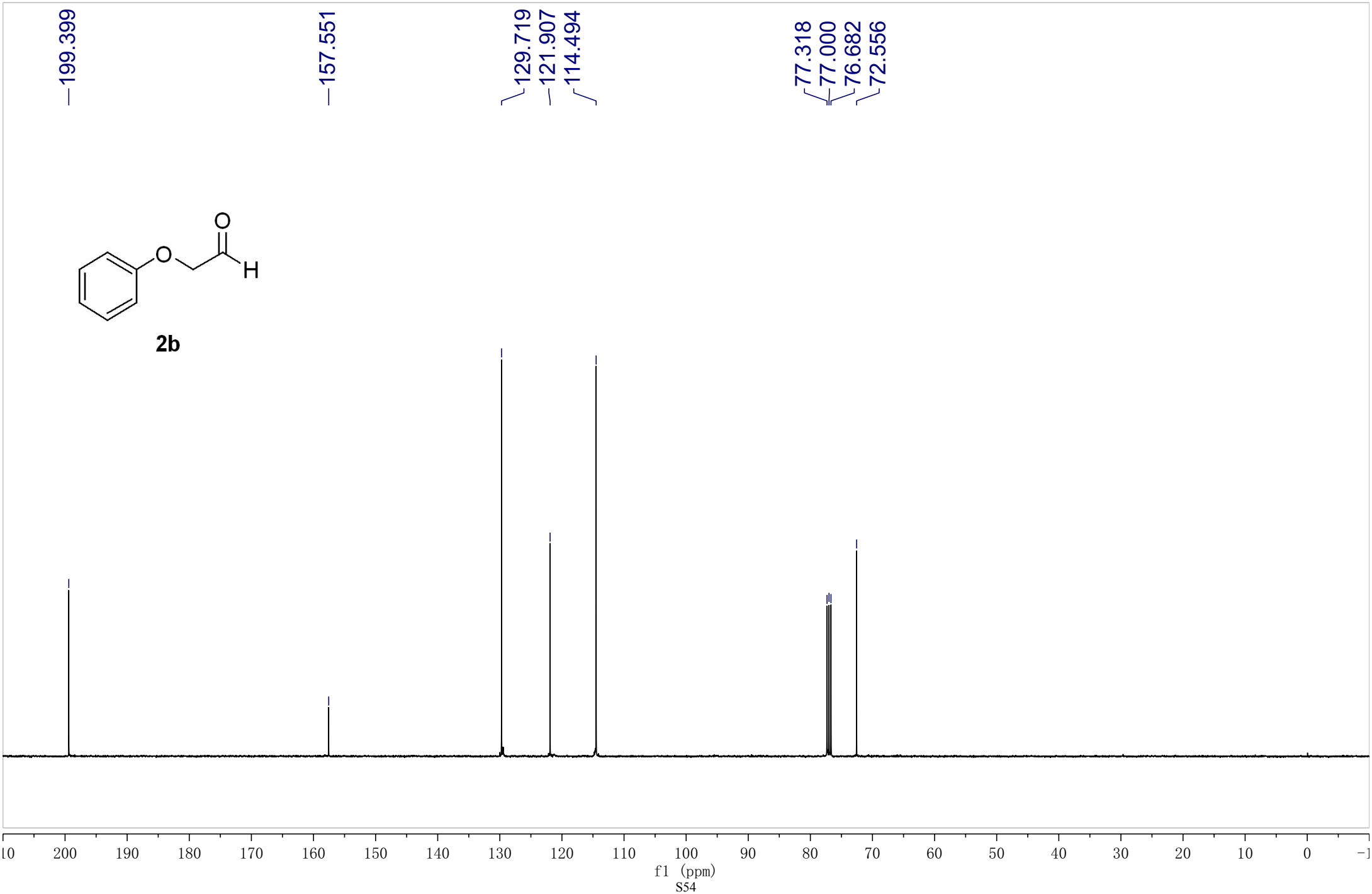
YWY-4-105A2 H1
CDCl3 100MHz



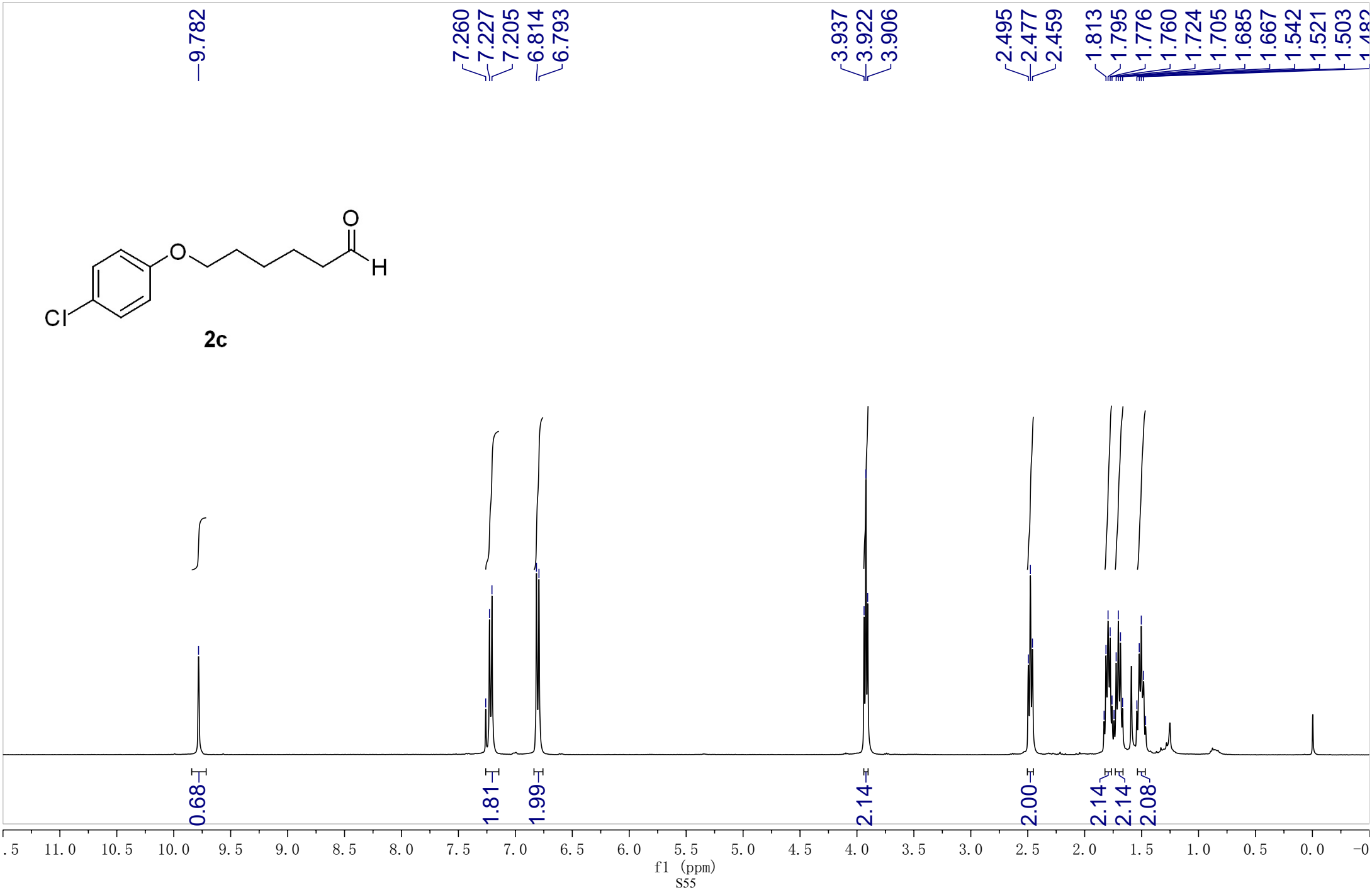
YWY-4-105A3P H1
CDCl3 400MHz



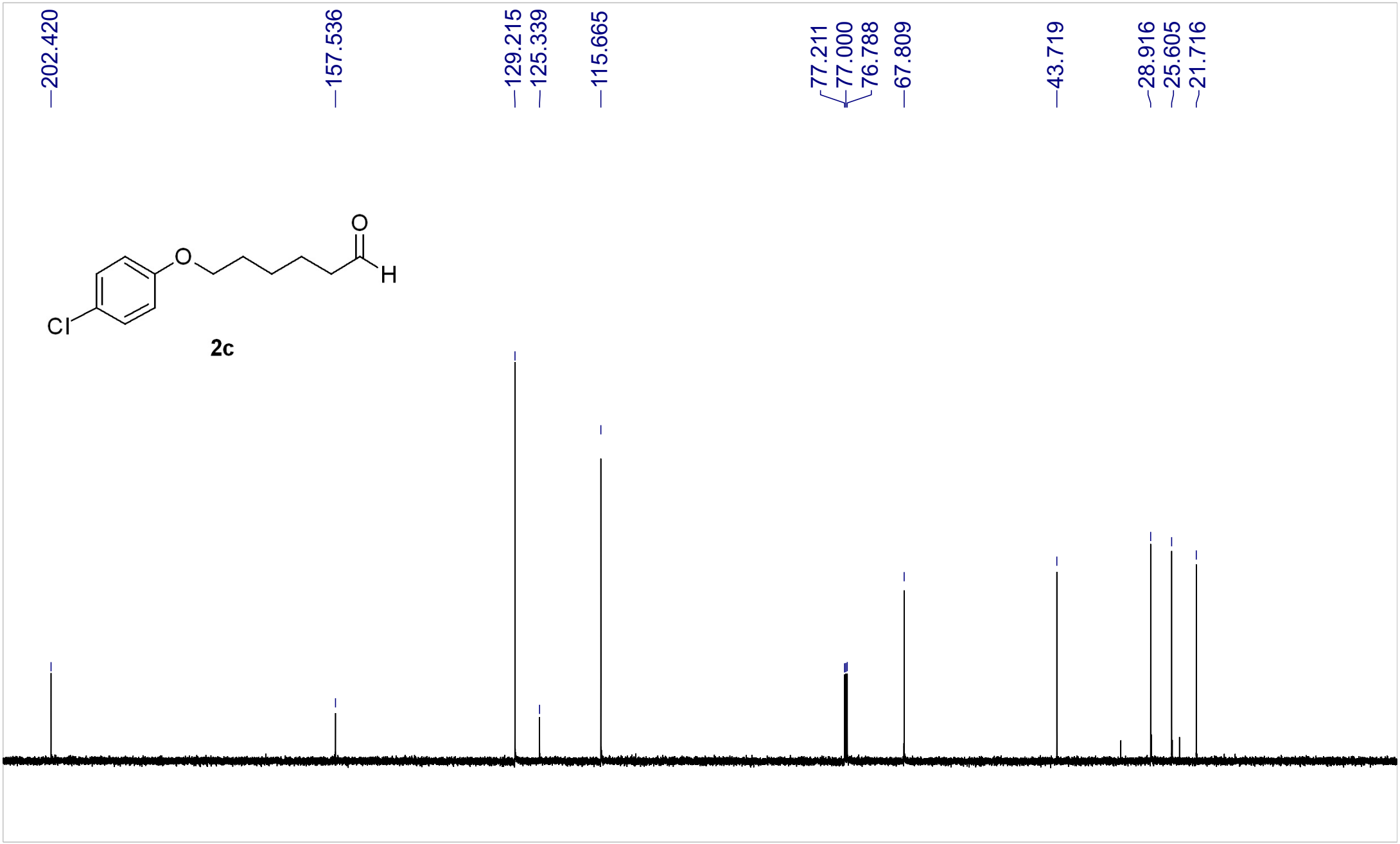
YWY-4-105A3P H1
CDCl3 100MHz



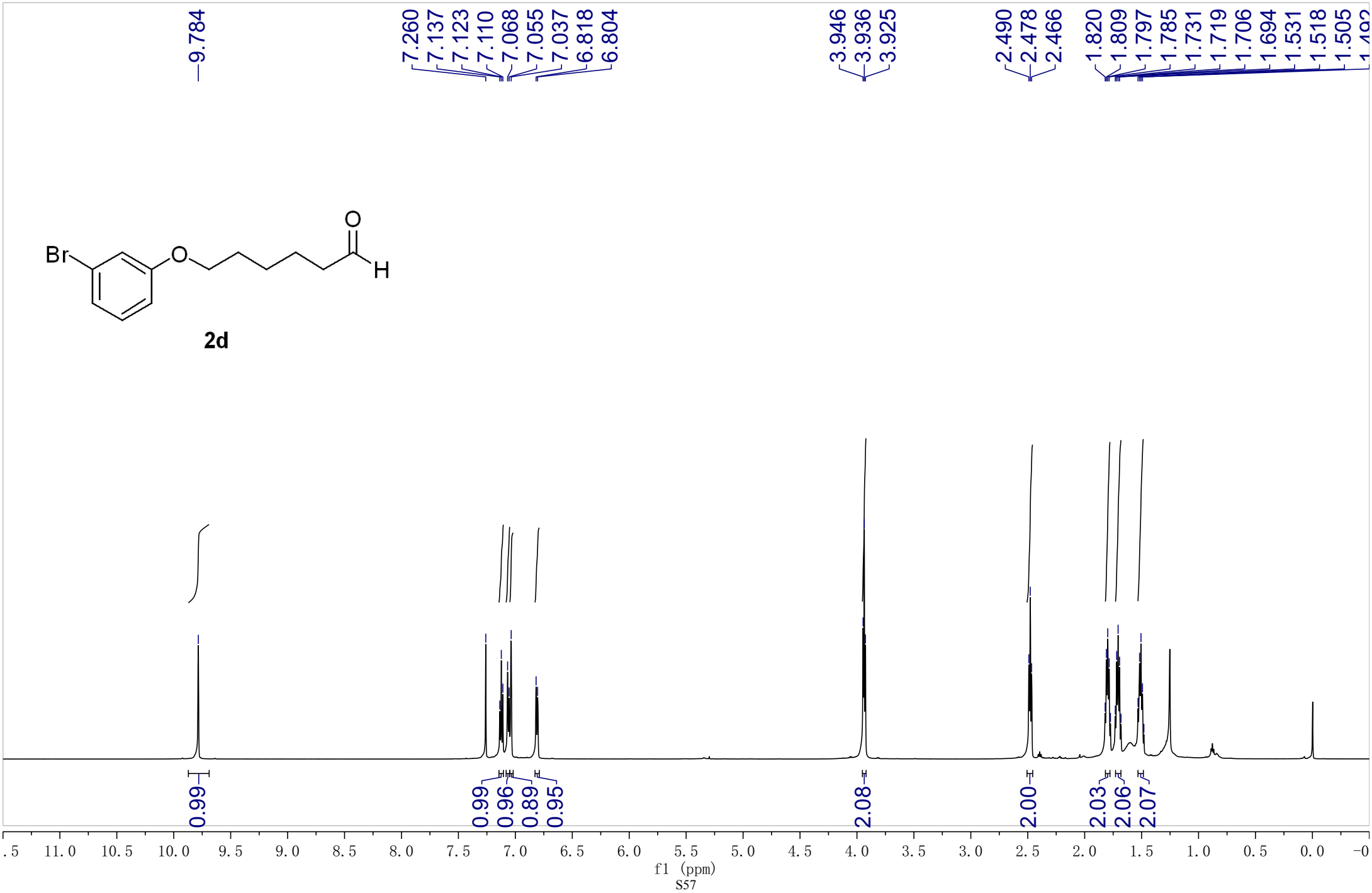
YWY-4-46A1 H1
CDCl3 400MHz



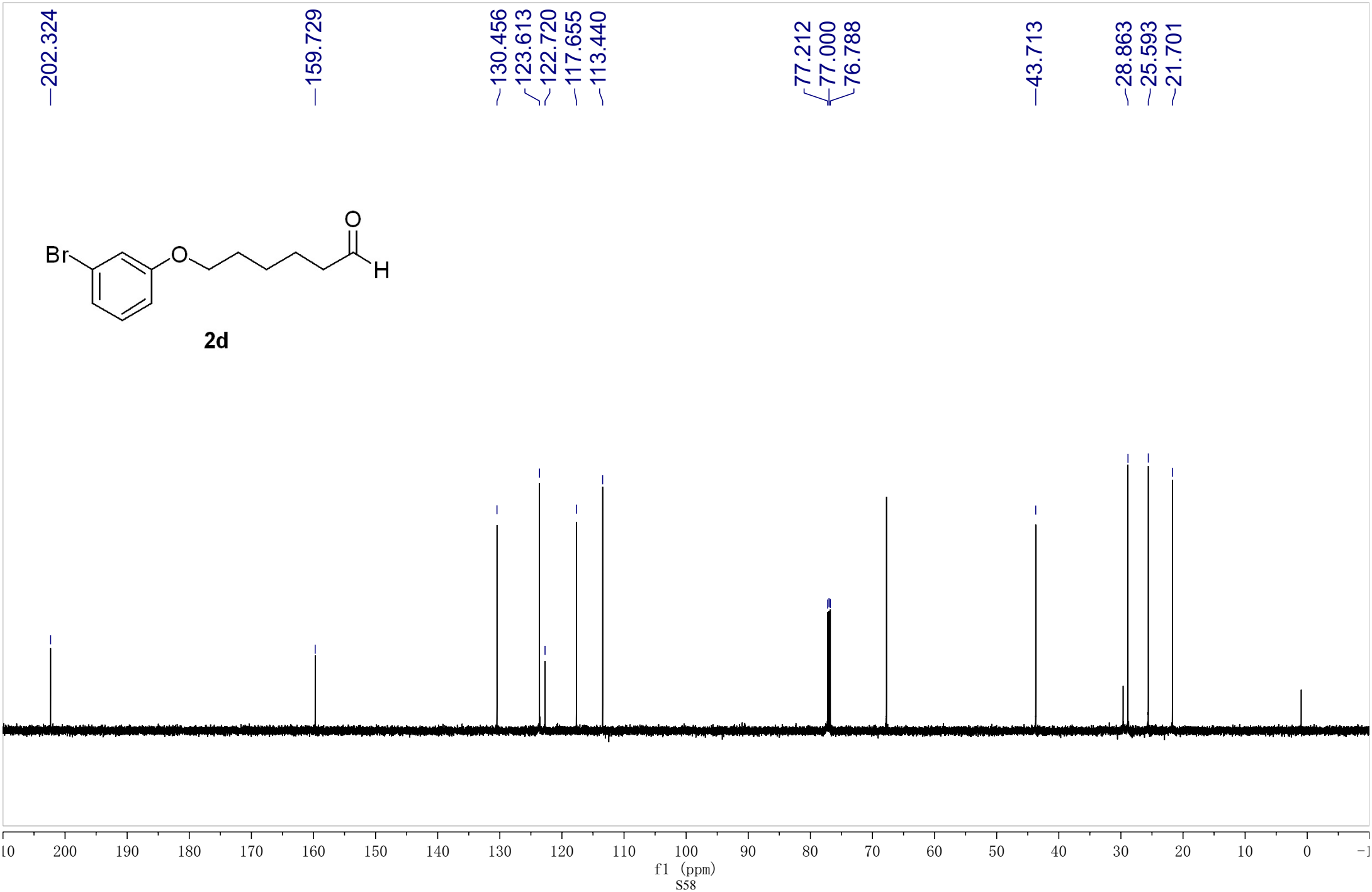
YWY-4-46A1 C13
CDCl3 150MHz



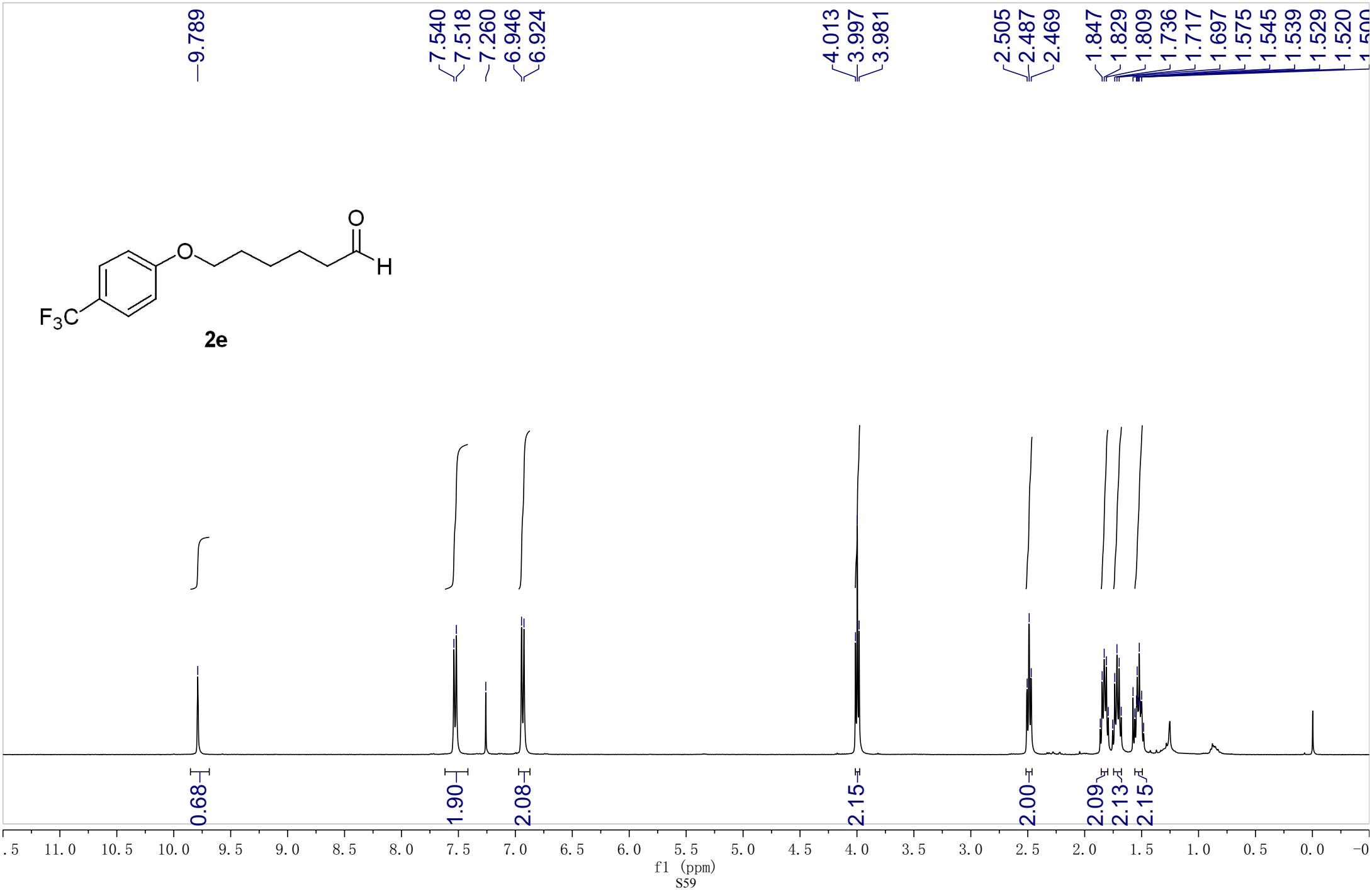
YWY-4-46A2-1 H1
CDCl3 600MHz



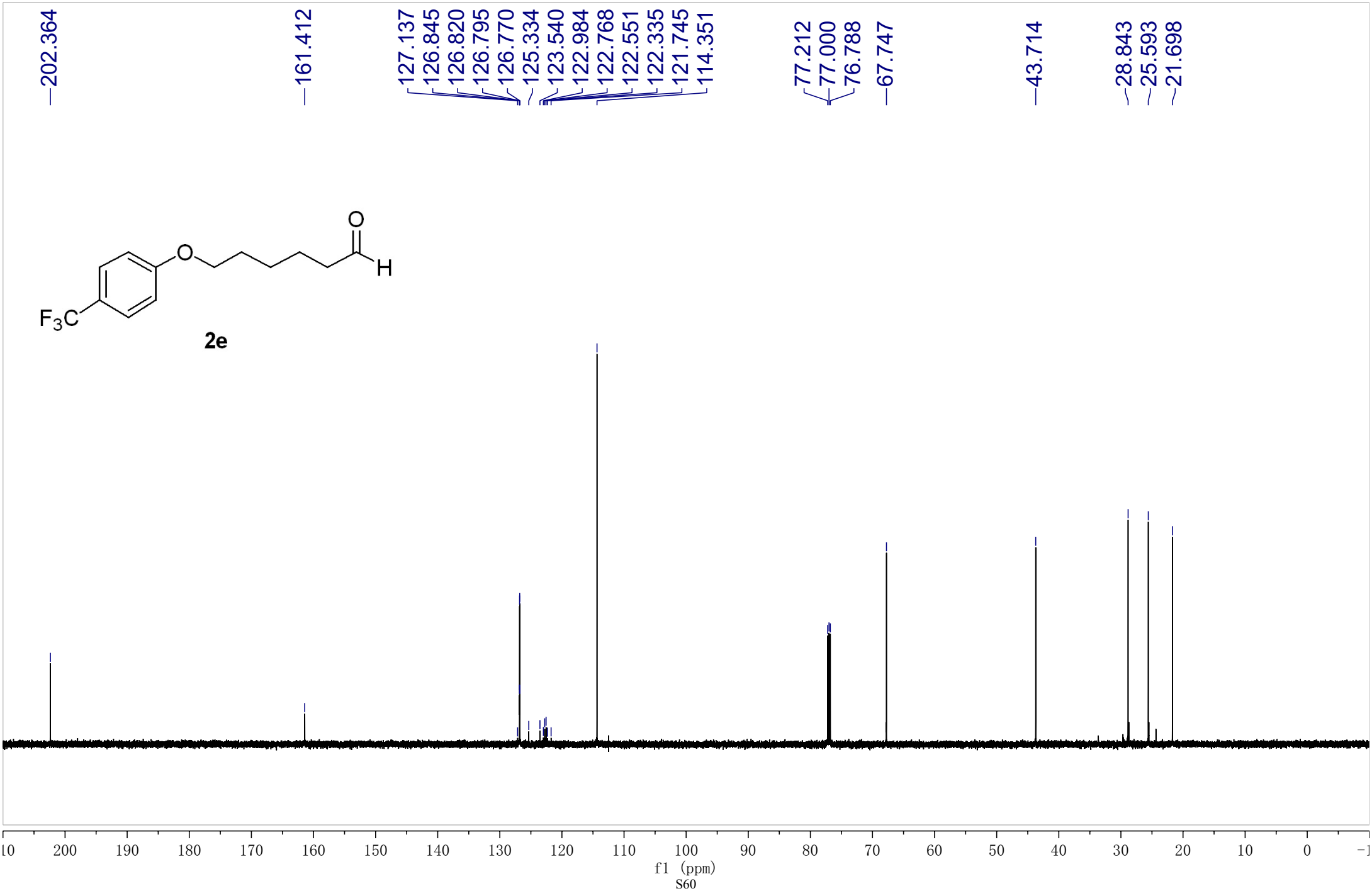
YWY-4-46A2-1 C13
CDCl3 150MHz



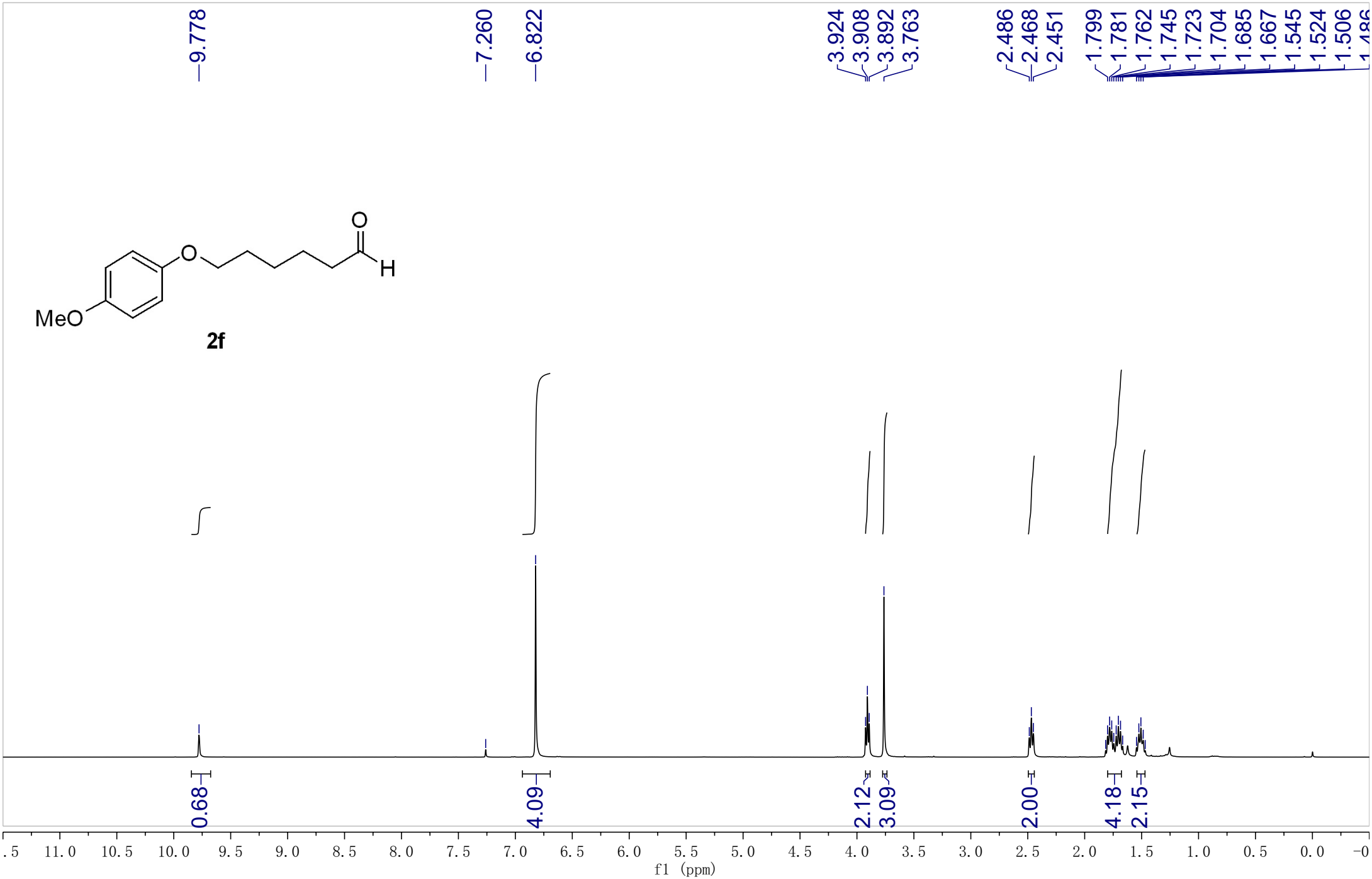
YWY-4-39A2 H1
CDCl3 400MHz



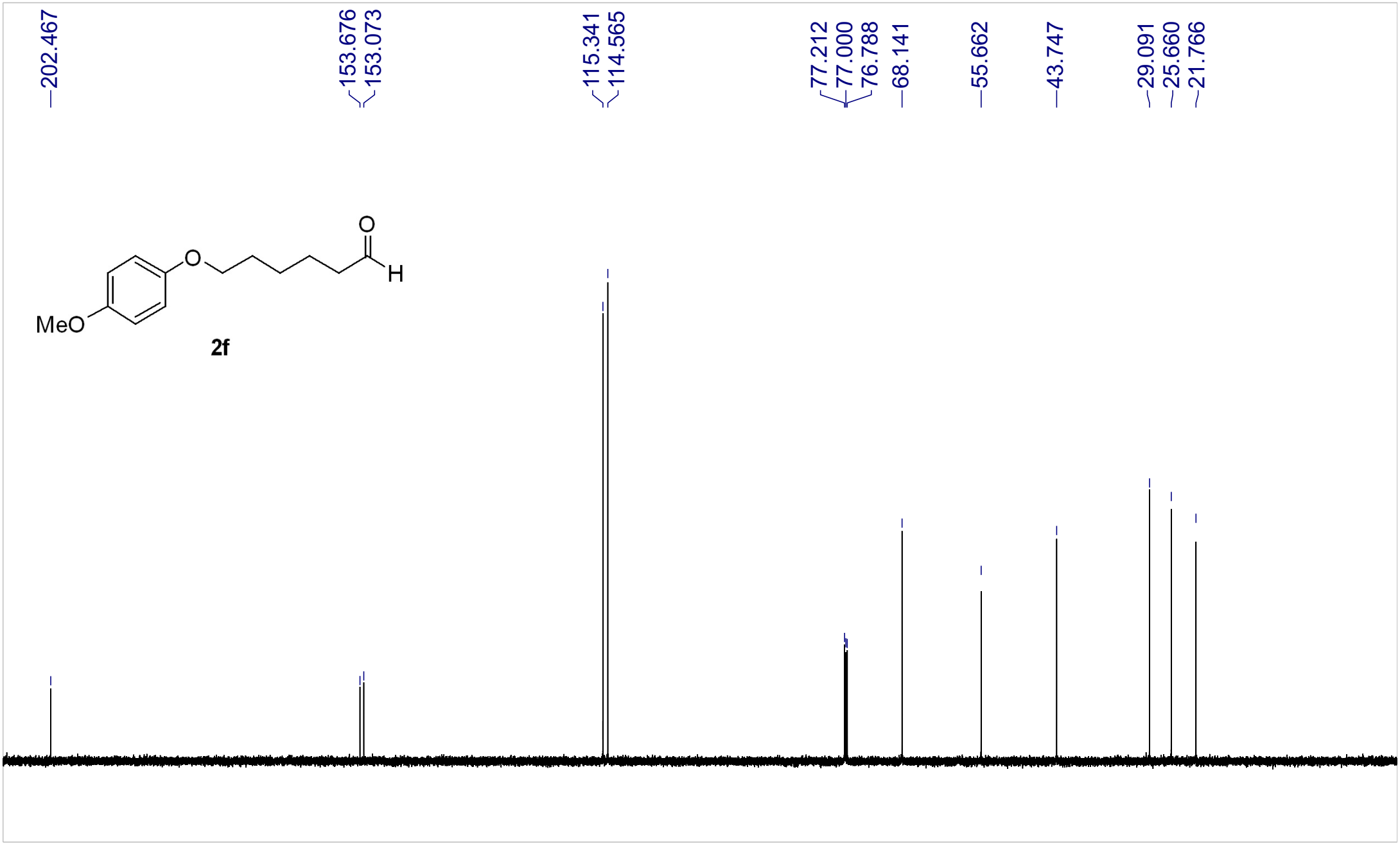
YWY-4-70A1 C13
CDCl3 150MHz



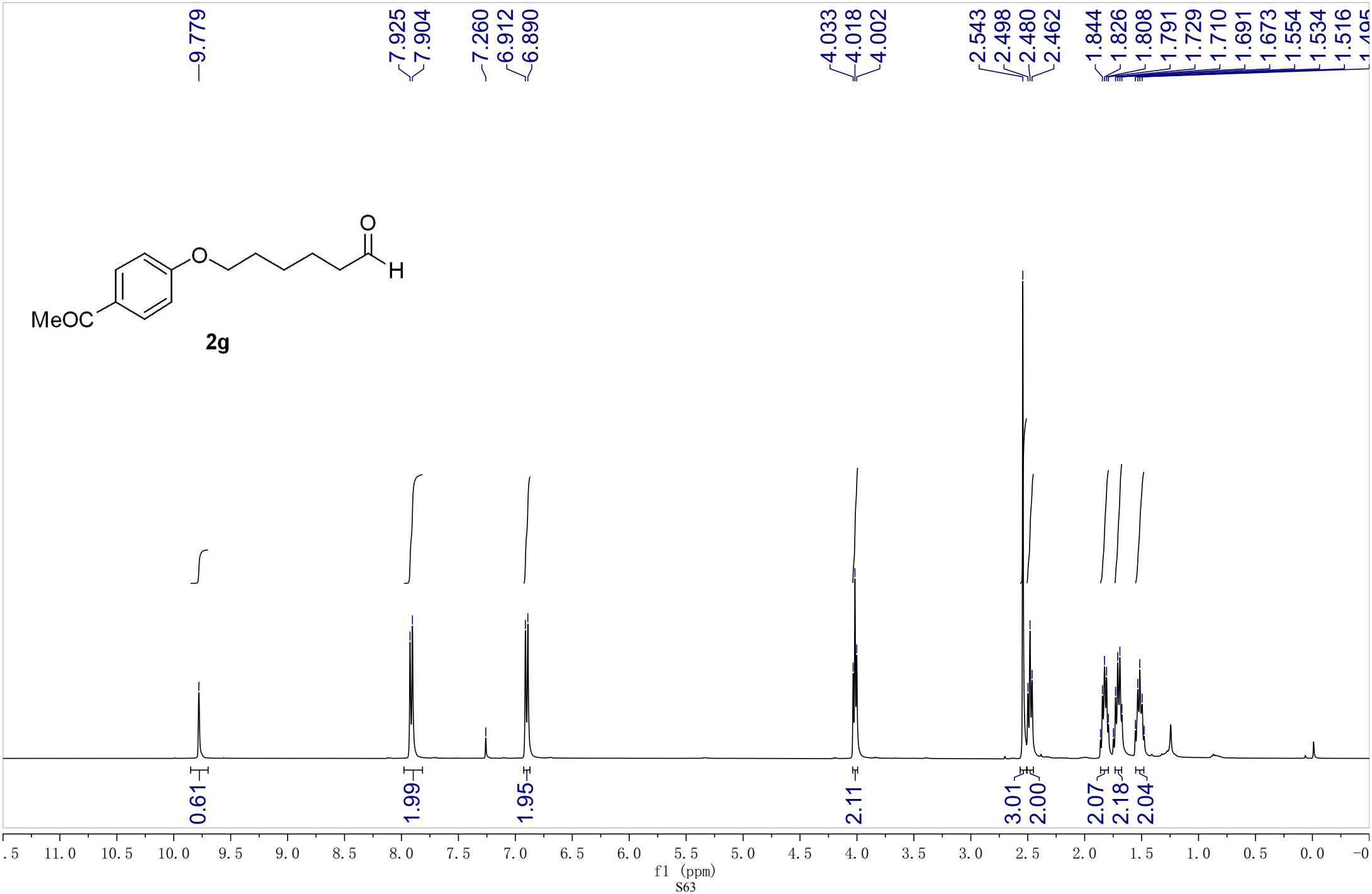
YWY-4-70A2 H1
CDCl3 400MHz



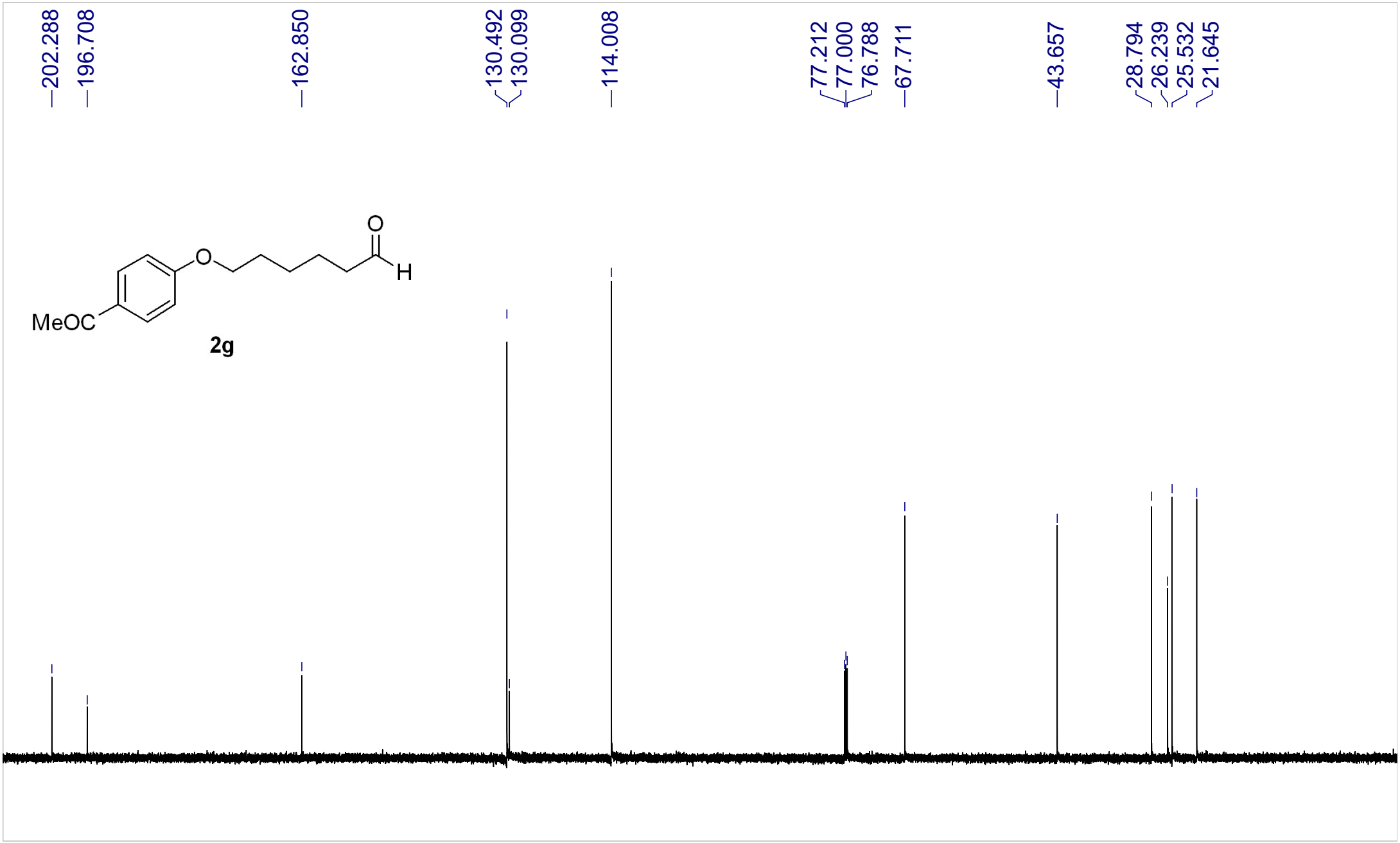
YWY-4-70A2 C13
CDCl3 150MHz



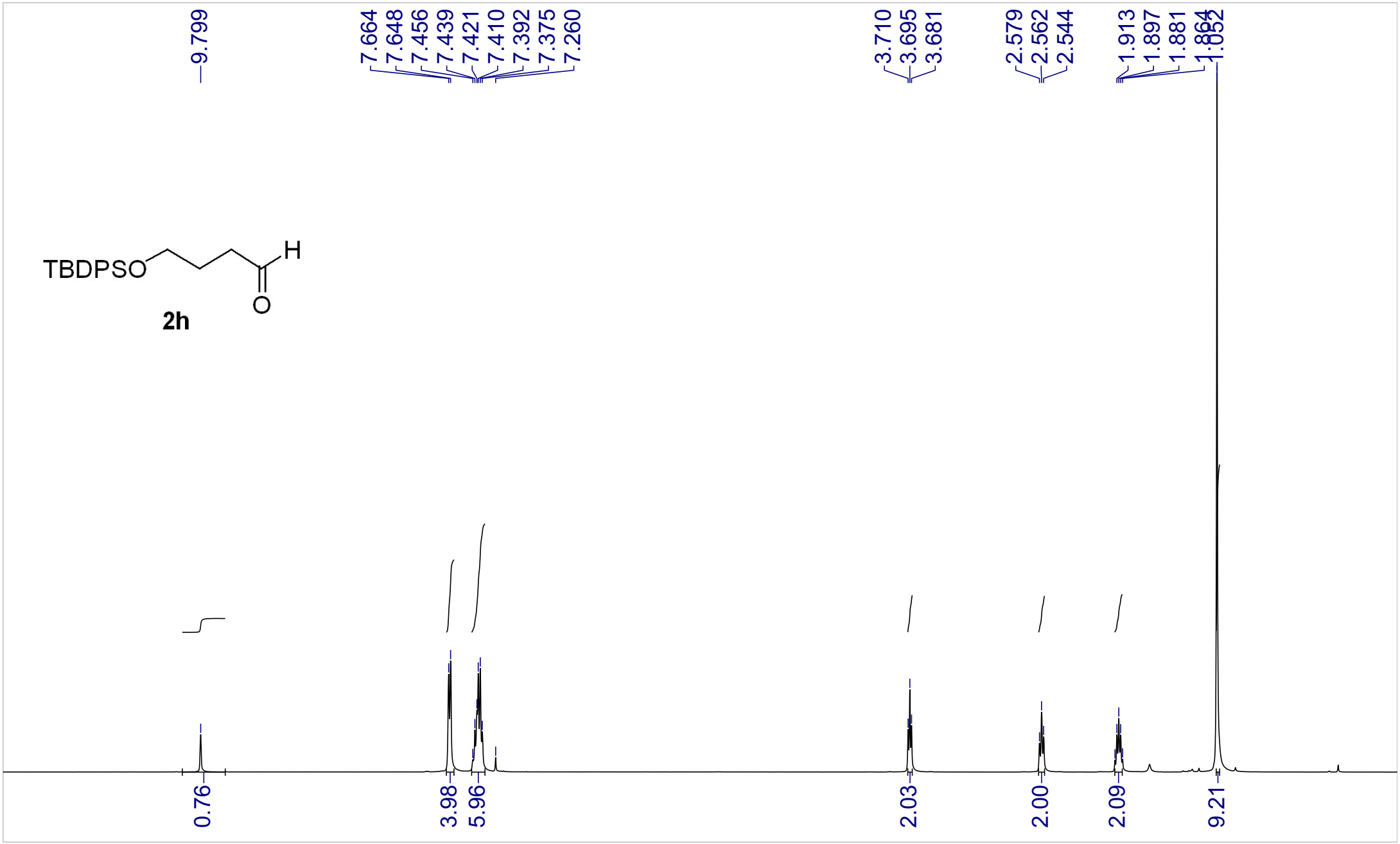
YWY-4-55P H1
CDCl3 400MHz



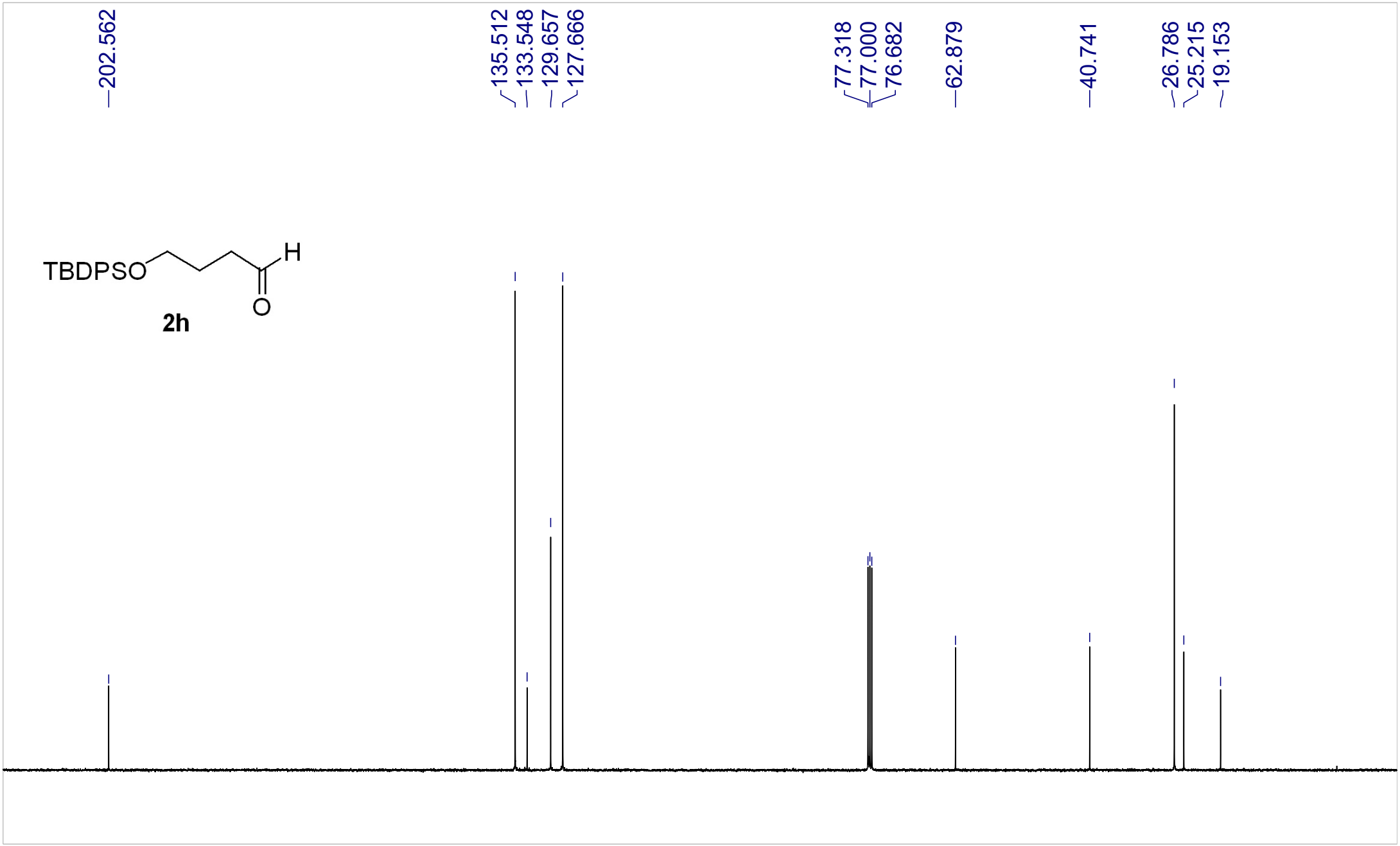
YWY-4-55P C13
CDCl3 150MHz



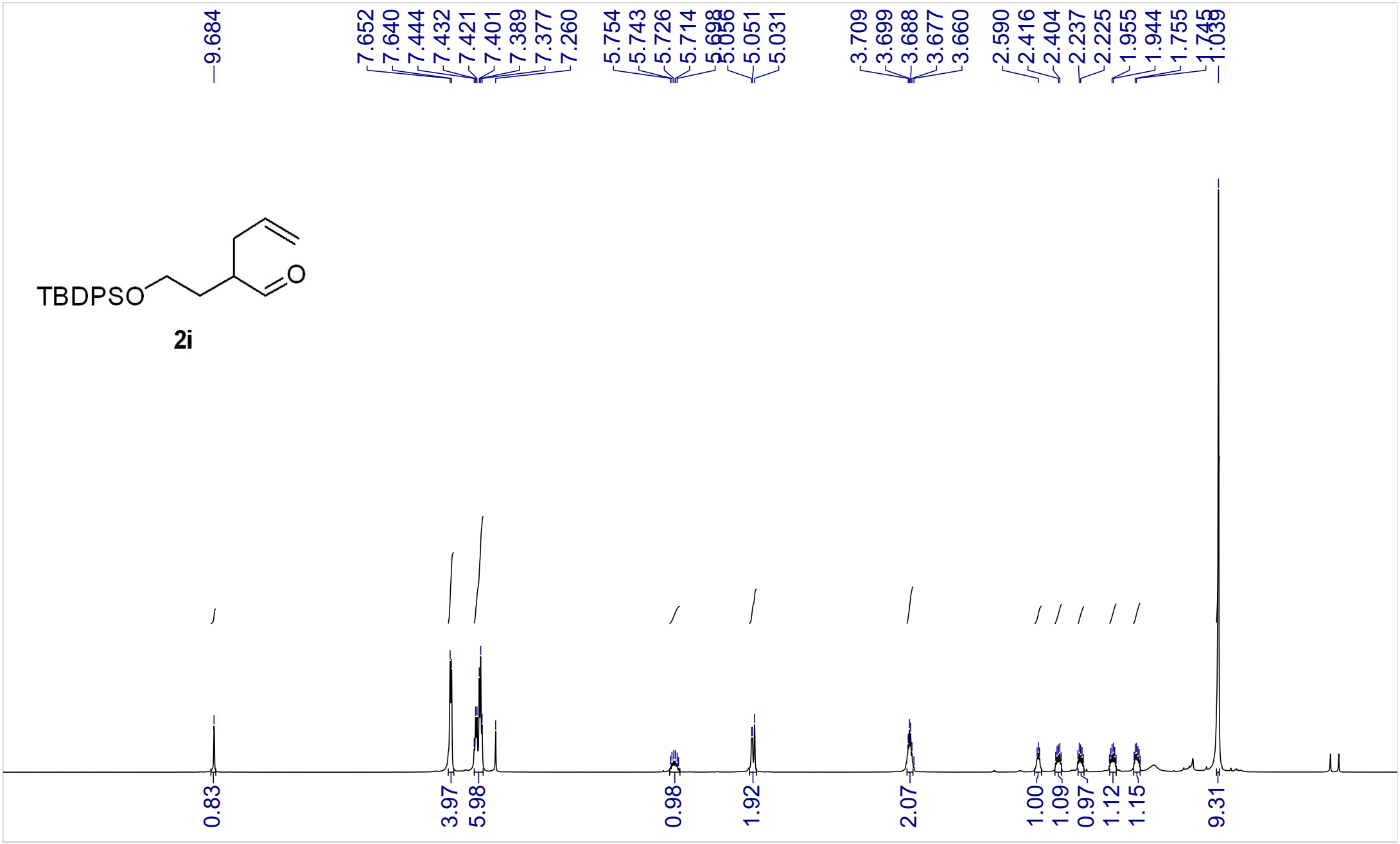
YWY-4-105A1 H1
CDCl3 400MHz



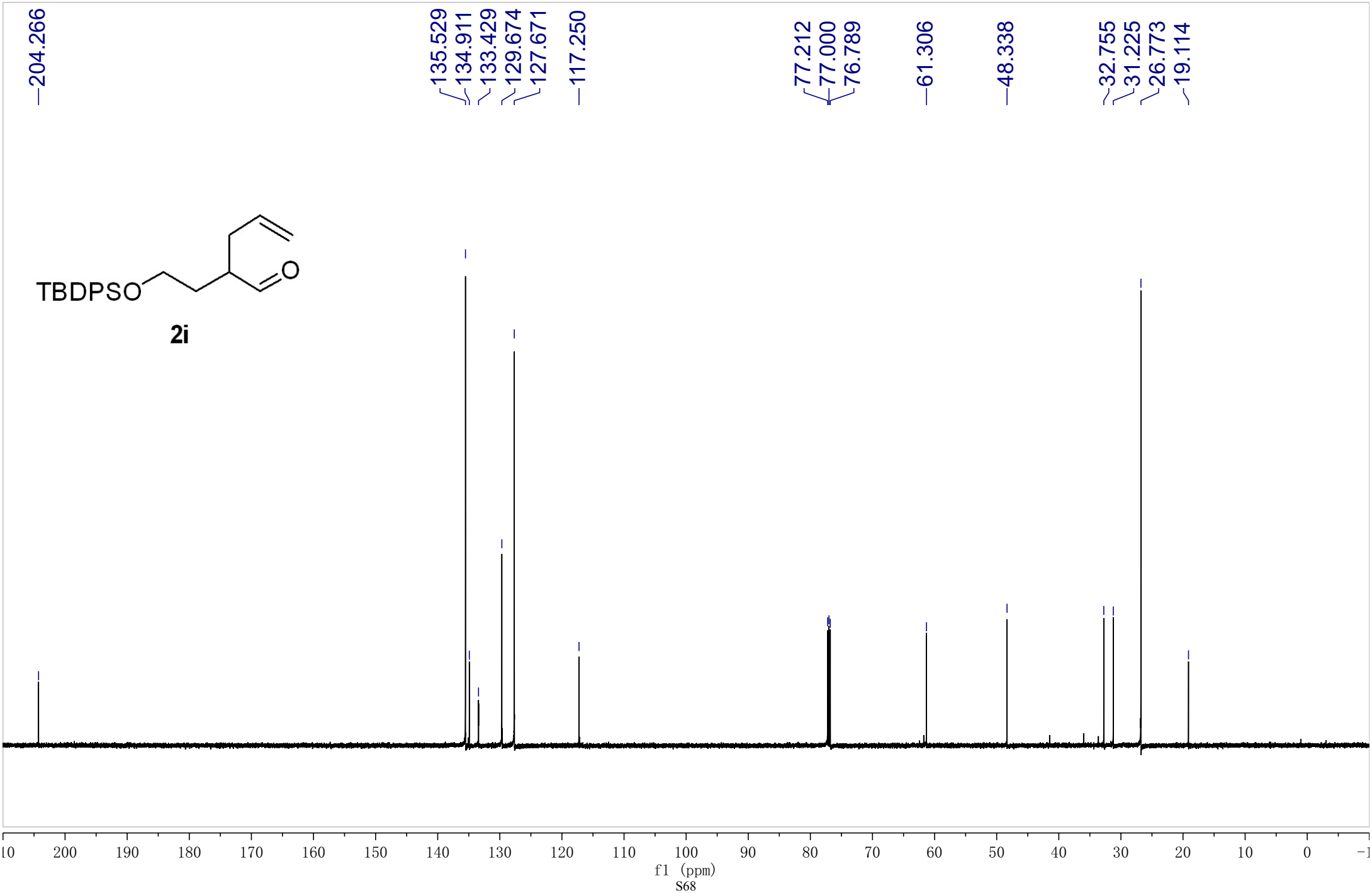
YWY-4-105A1 C13
CDCl3 100MHz



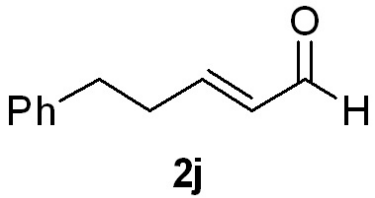
YWY-4-60P H1
CDCl3 600MHz



YWY-4-60P C13
CDCl3 150MHz

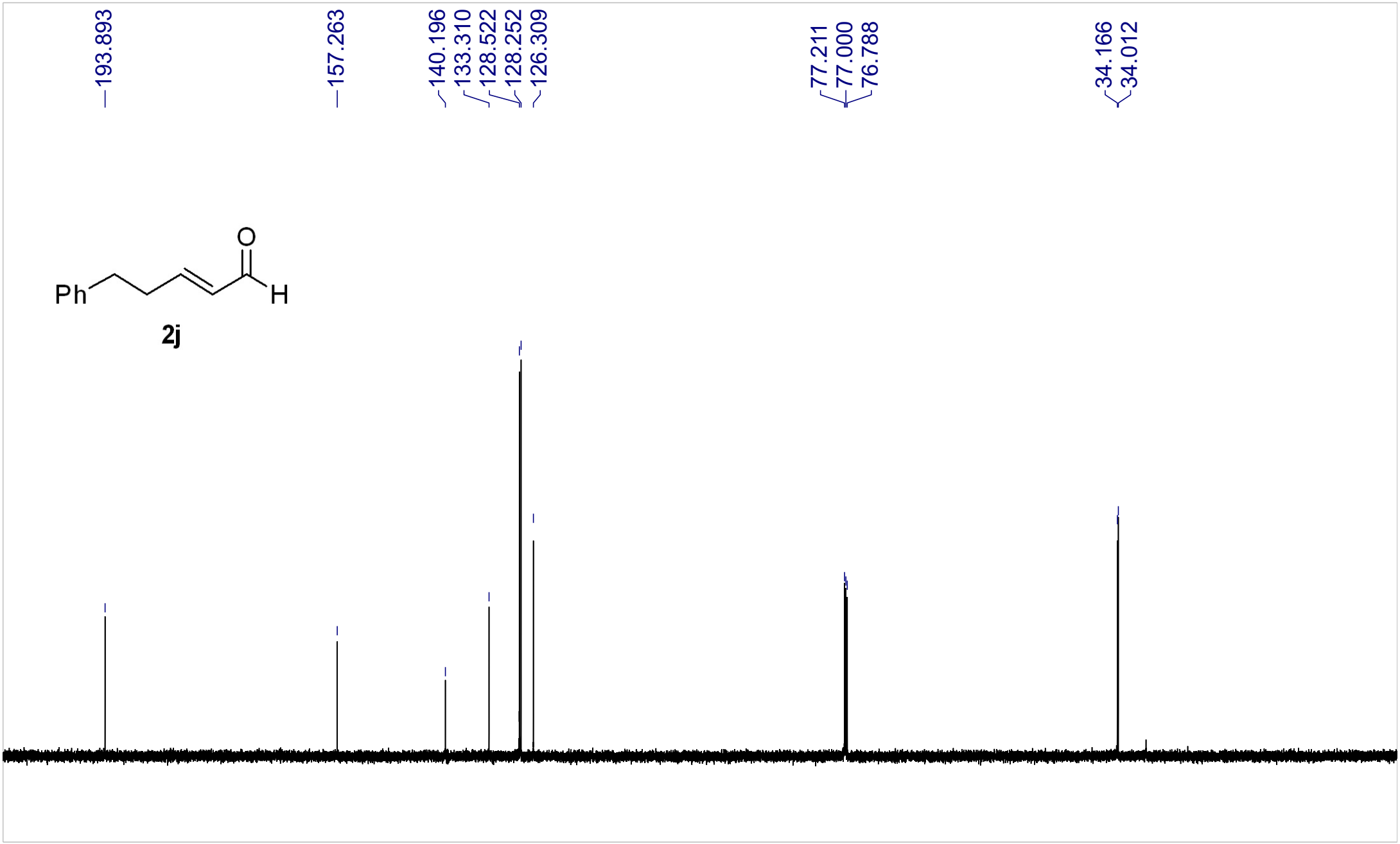


YWY-4-73A2 H1
CDCl3 600MHz

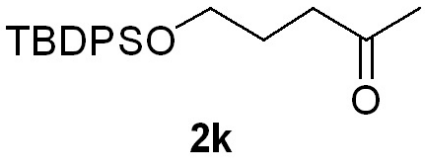


2j

YWY-4-73A2 C13
CDCl3 150MHz

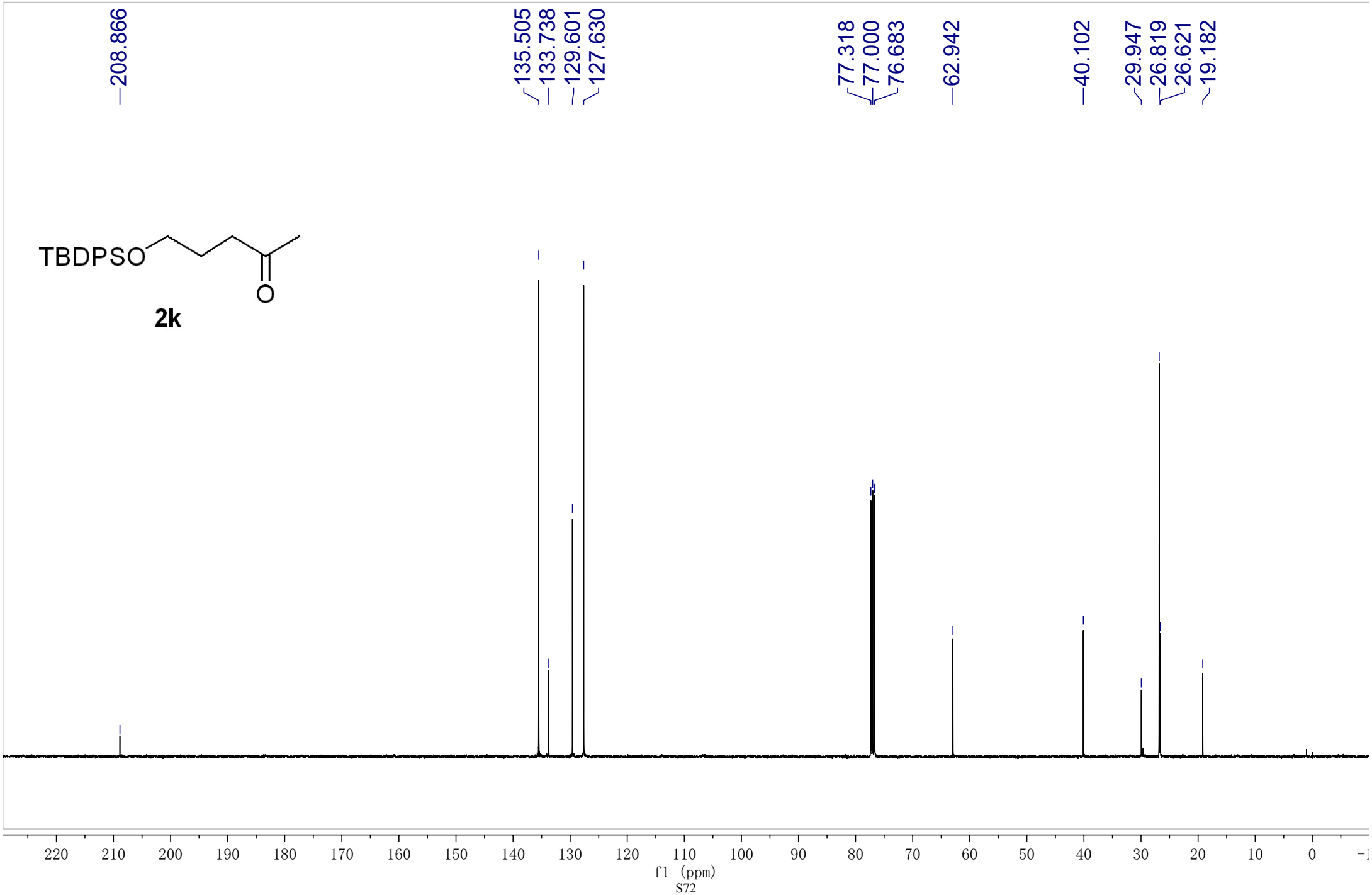


YWY-5-16A1 H1
CDCl3 600MHz

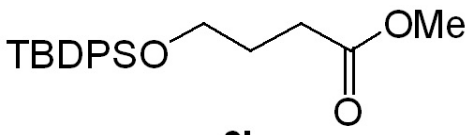


2k

YWY-5-16A1 C13
CDCl3 100MHz

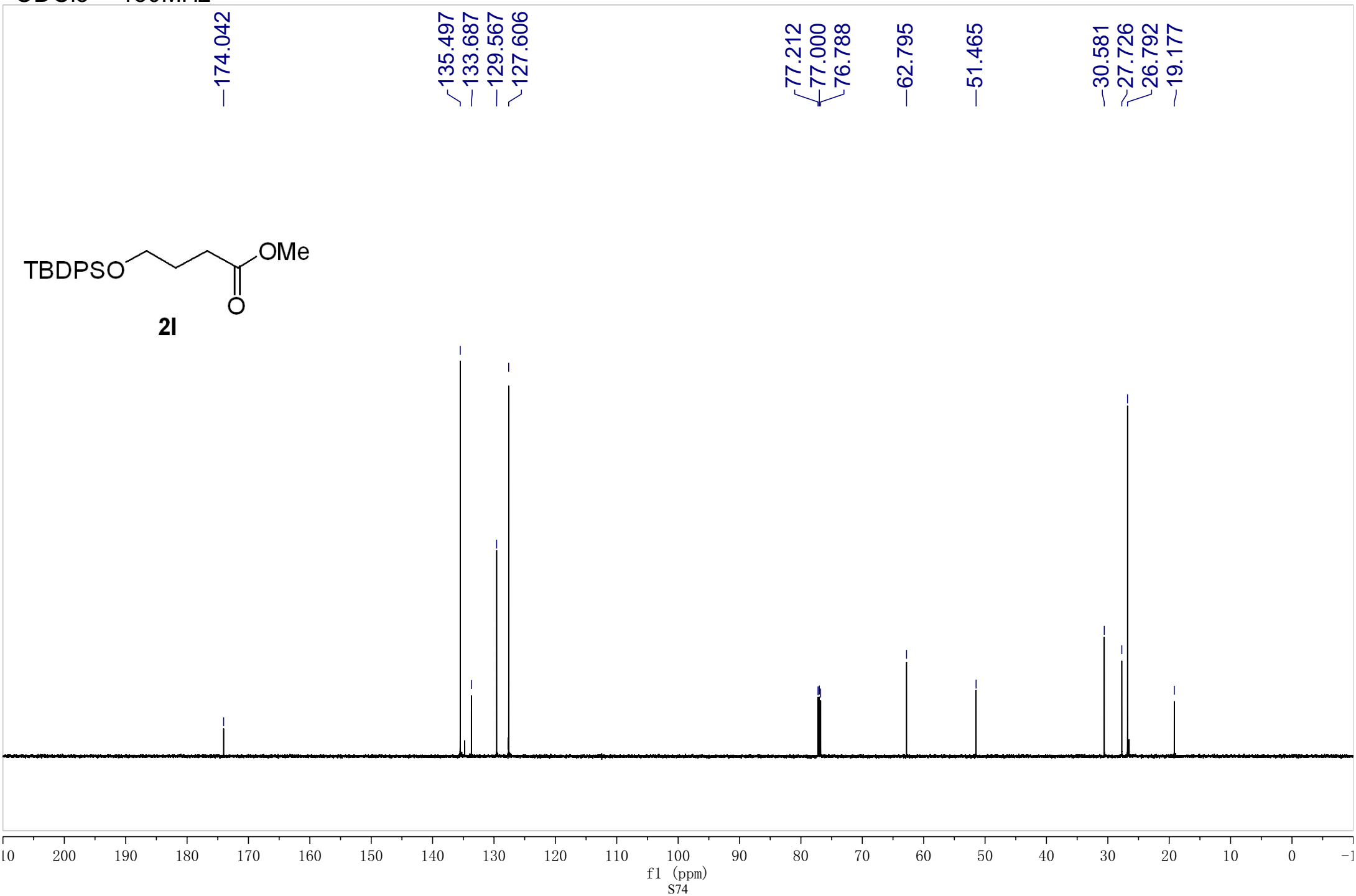


YWY-H-41P H1
CDCl3 400MHz

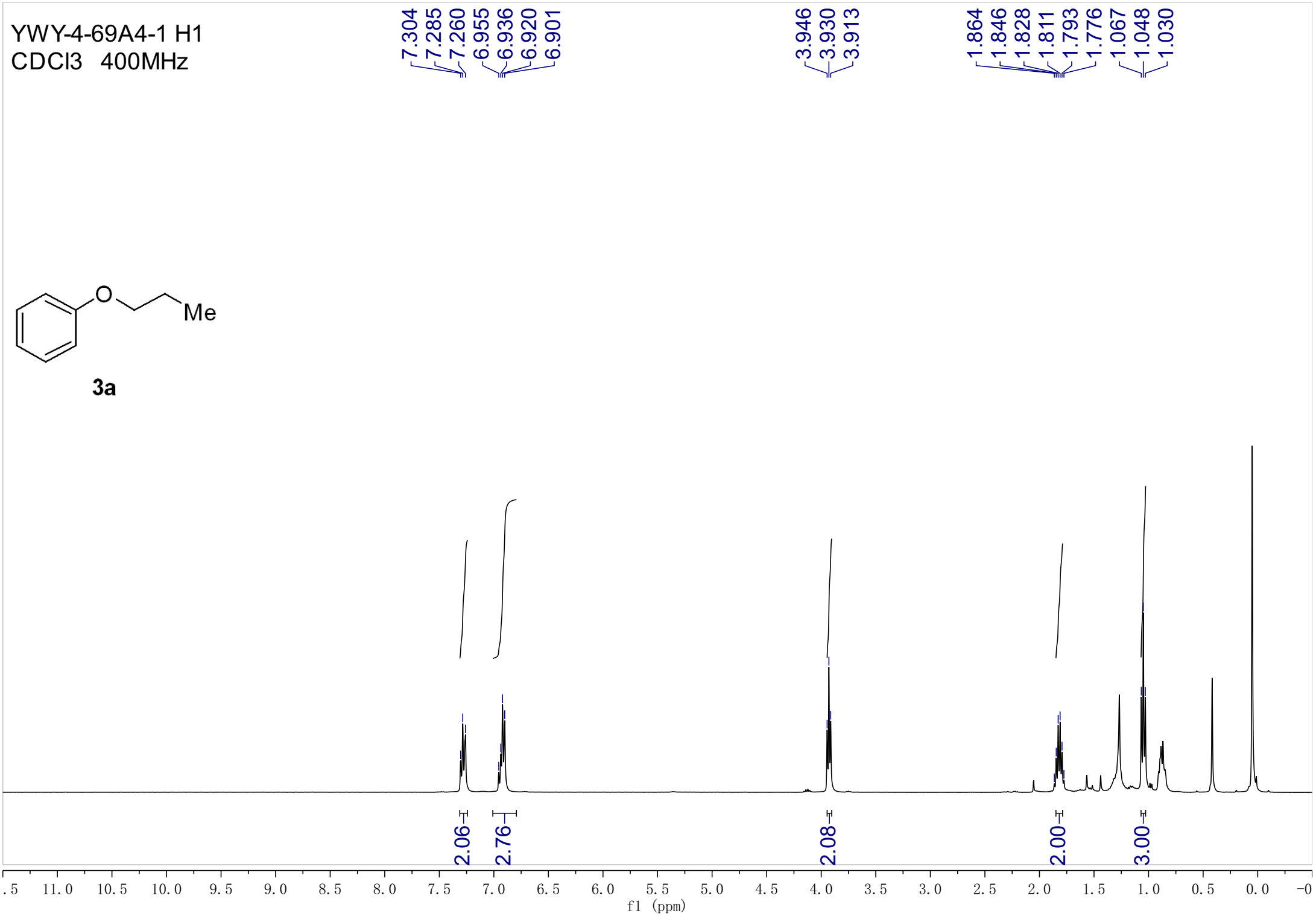


2l

YWY-H-41P C13
CDCl3 150MHz



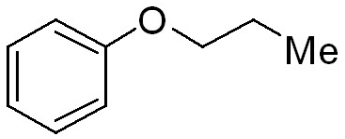
YWY-4-69A4-1 H1
CDCl3 400MHz



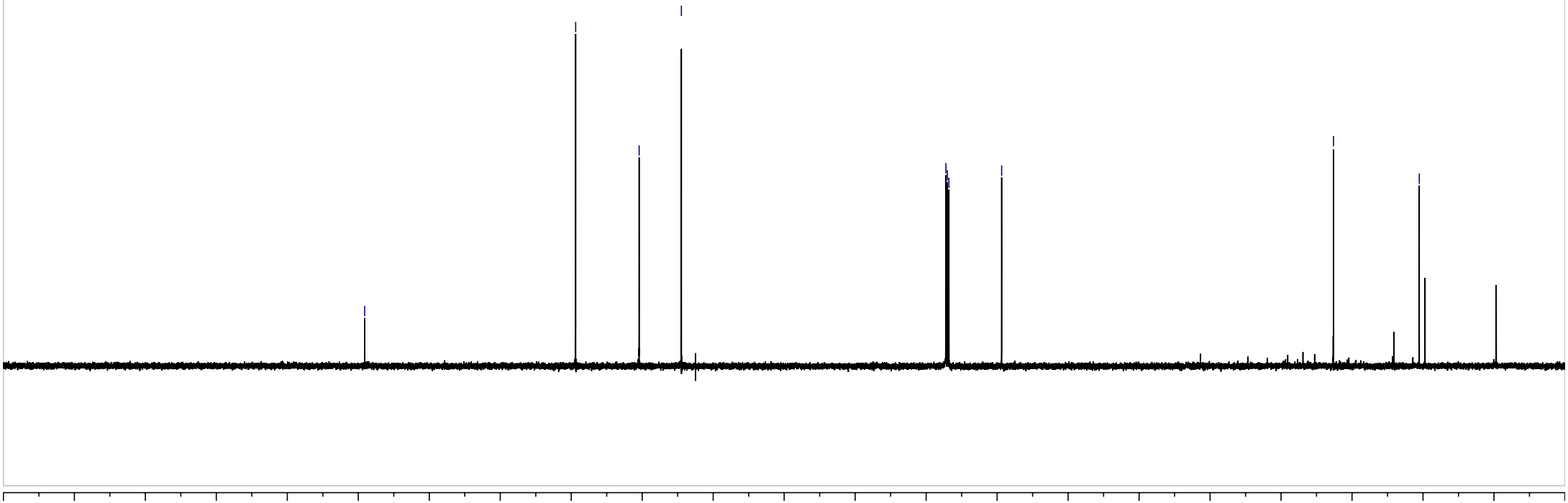
3a

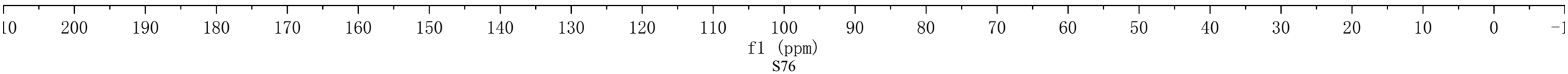
YWY-4-69A4-1 C13
CDCl3 150MHz

159.101
129.374
120.433
114.482
77.211
77.000
76.788
69.363
22.605
10.526

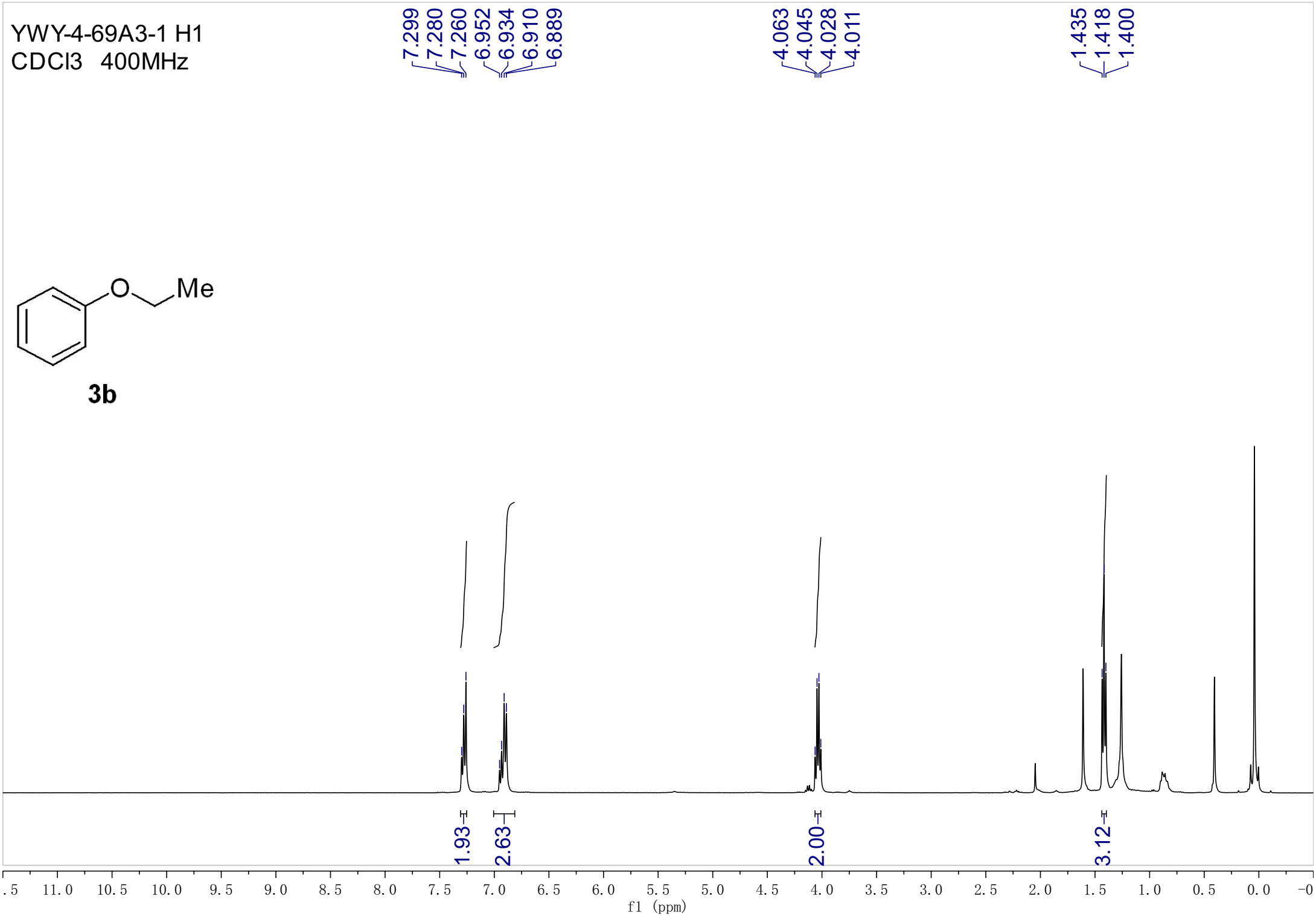


3a





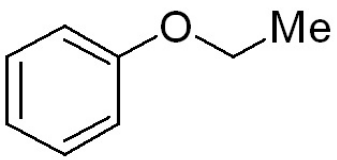
YWY-4-69A3-1 H1
CDCl3 400MHz



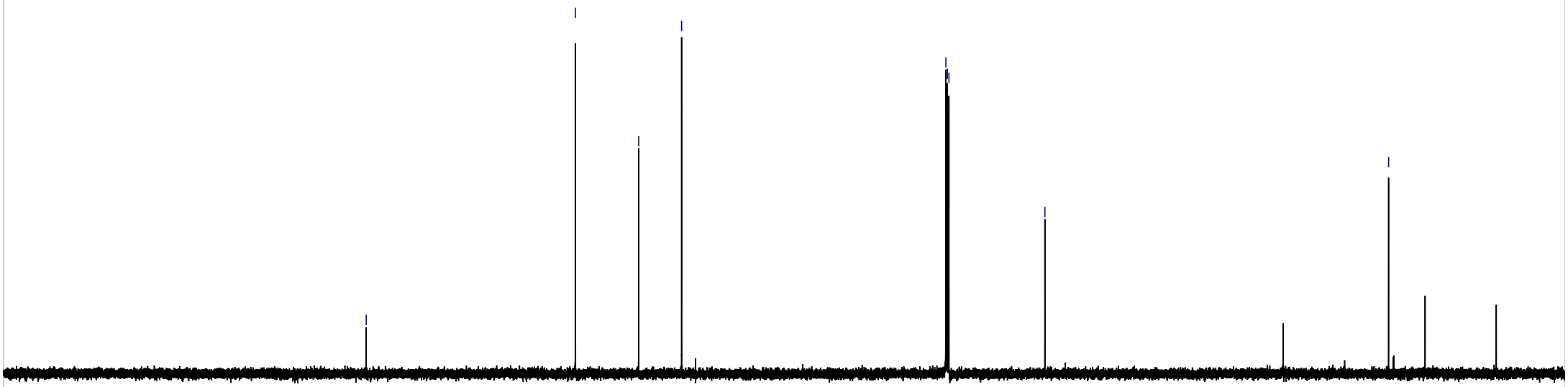
3b

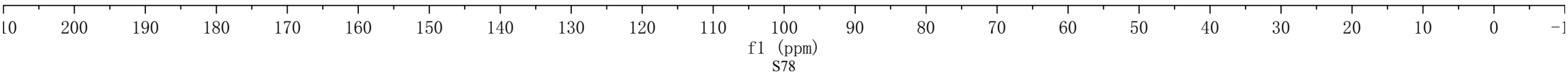
YWY-4-69A3-1 C13
CDCl3 150MHz

158.884
129.395
120.499
114.449
77.211
77.000
76.788
63.267
14.846

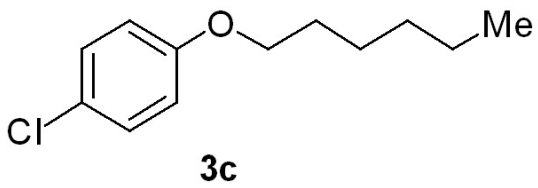


3b





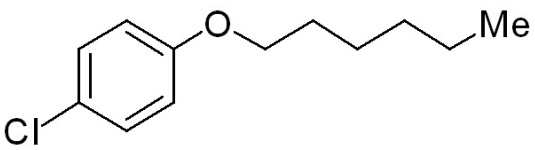
YWY-4-72A1 H1
CDCl3 400MHz



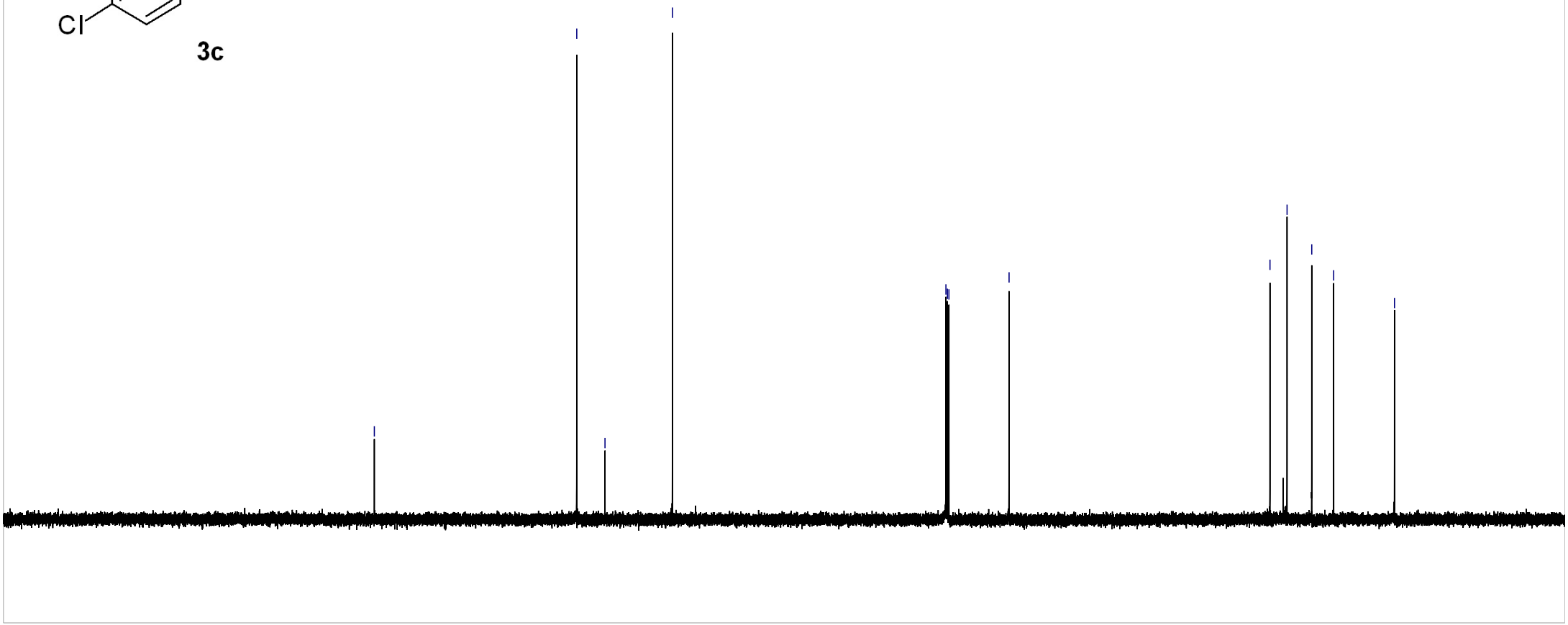
3c

YWY-4-72A1 C13
CDCl3 150MHz

157.737
129.219
125.242
115.734
77.212
77.000
76.788
68.316
31.557
29.152
25.673
22.585
14.015

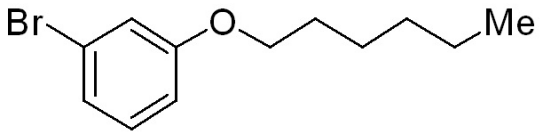


3c



f1 (ppm)

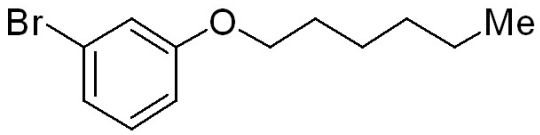
YWY-4-72A2-1 H1
CDCl3 400MHz



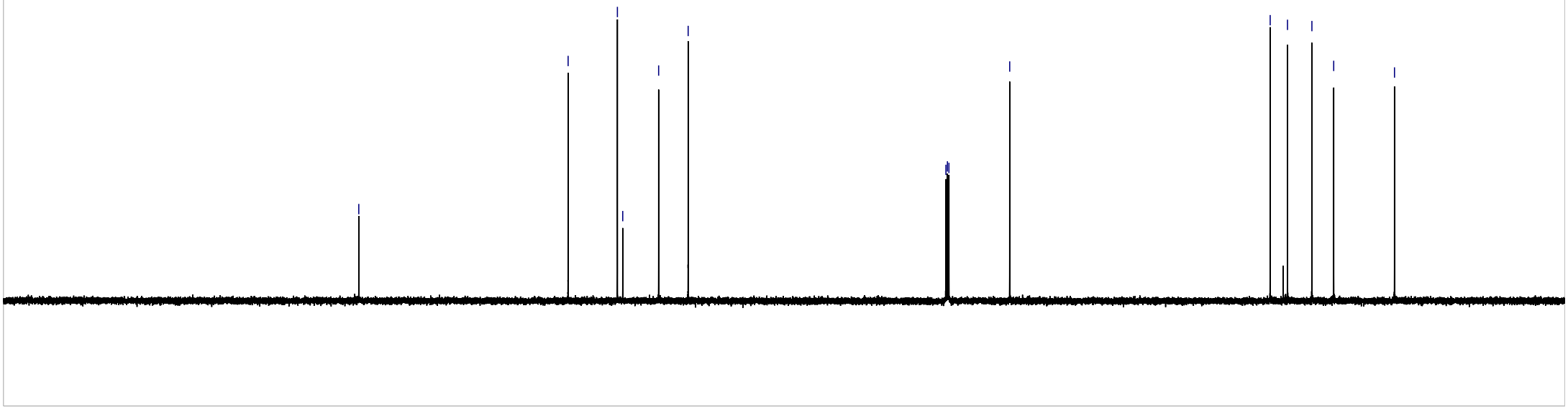
3d

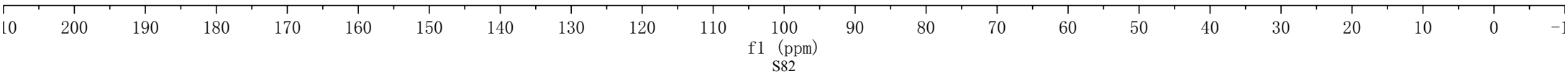
YWY-4-72A2-1 C13
CDCl3 150MHz

159.928
130.437
123.495
122.743
117.684
113.526
77.212
77.000
76.789
68.226
31.528
29.091
25.653
22.581
14.017

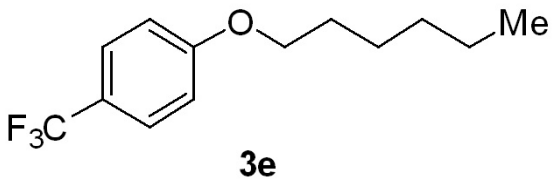


3d





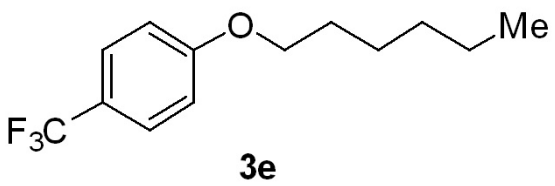
YWY-4-71A3 H1
CDCl3 400MHz



3e

YWY-4-71A3-1 C13
CDCl3 150MHz

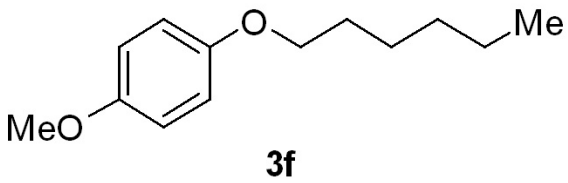
161.593
127.197
126.839
126.815
126.790
126.765
125.399
123.603
122.881
122.666
122.449
122.233
121.806
114.392
77.212
77.000
76.788
68.227
31.537
29.056
25.655
22.584
14.002



3e

f1 (ppm)

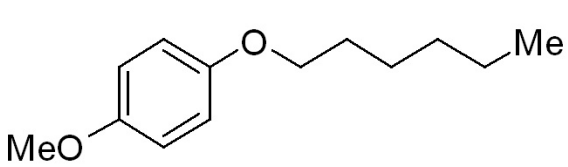
YWY-4-66A5 H1
CDCl3 400MHz



3f

YWY-4-66A5 C13
CDCl3 150MHz

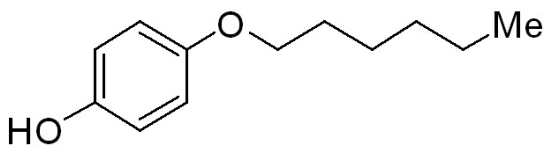
153.627
153.290
115.411
114.588
77.203
76.991
76.780
68.664
55.723
31.600
29.349
25.723
22.596
14.019



3f

f1 (ppm)

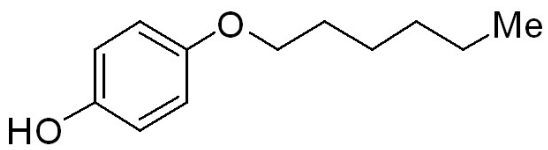
YWY-4-82A2 H1
CDCl3 400MHz



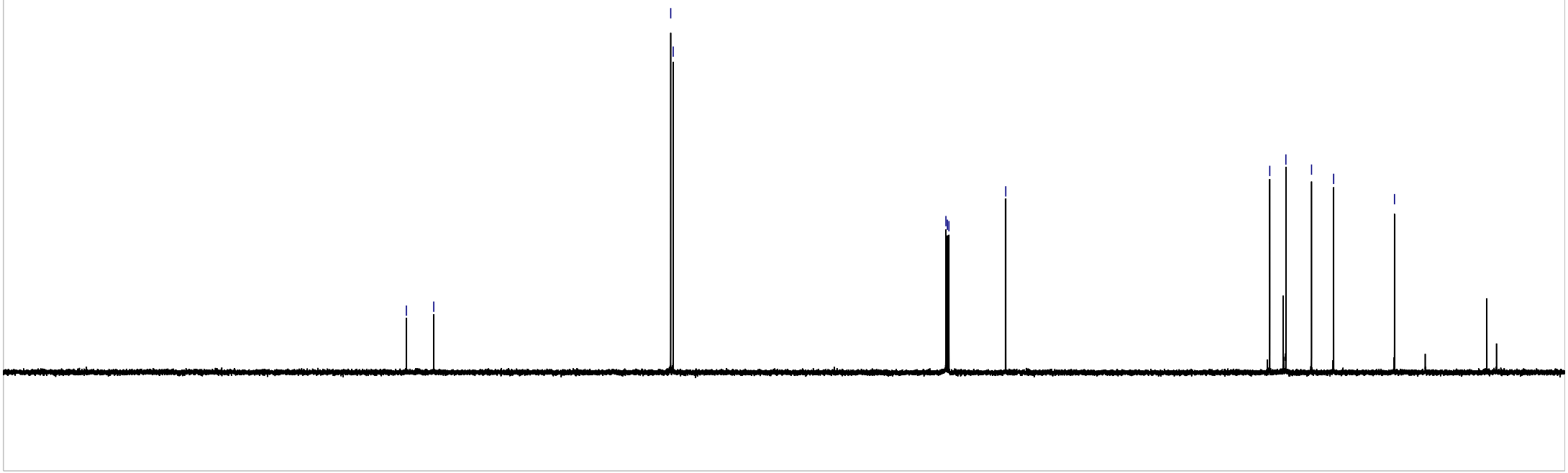
3g

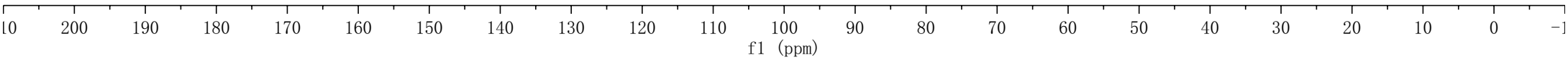
YWY-4-82A2 C13
CDCl3 150MHz

153.225
149.368
115.983
115.632
77.212
77.000
76.788
68.782
31.590
29.318
25.704
22.591
14.018

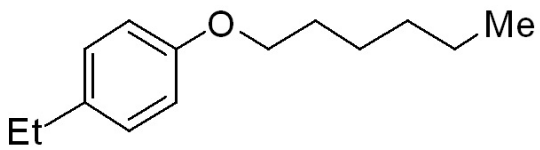


3g





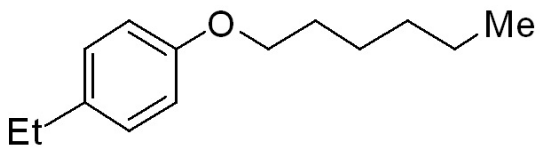
YWY-4-76A2 H1
CDCl3 600MHz



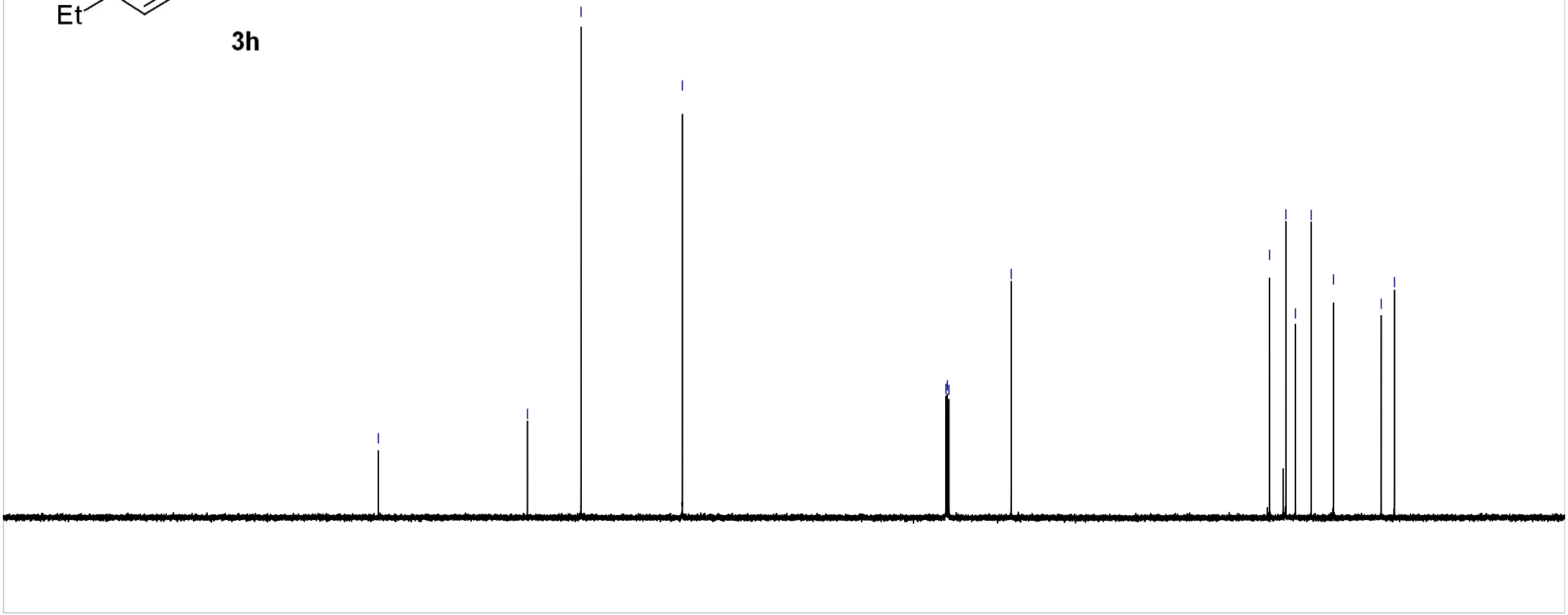
3h

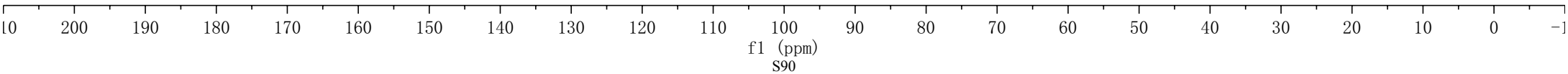
YWY-4-76A2 C13
CDCl3 150MHz

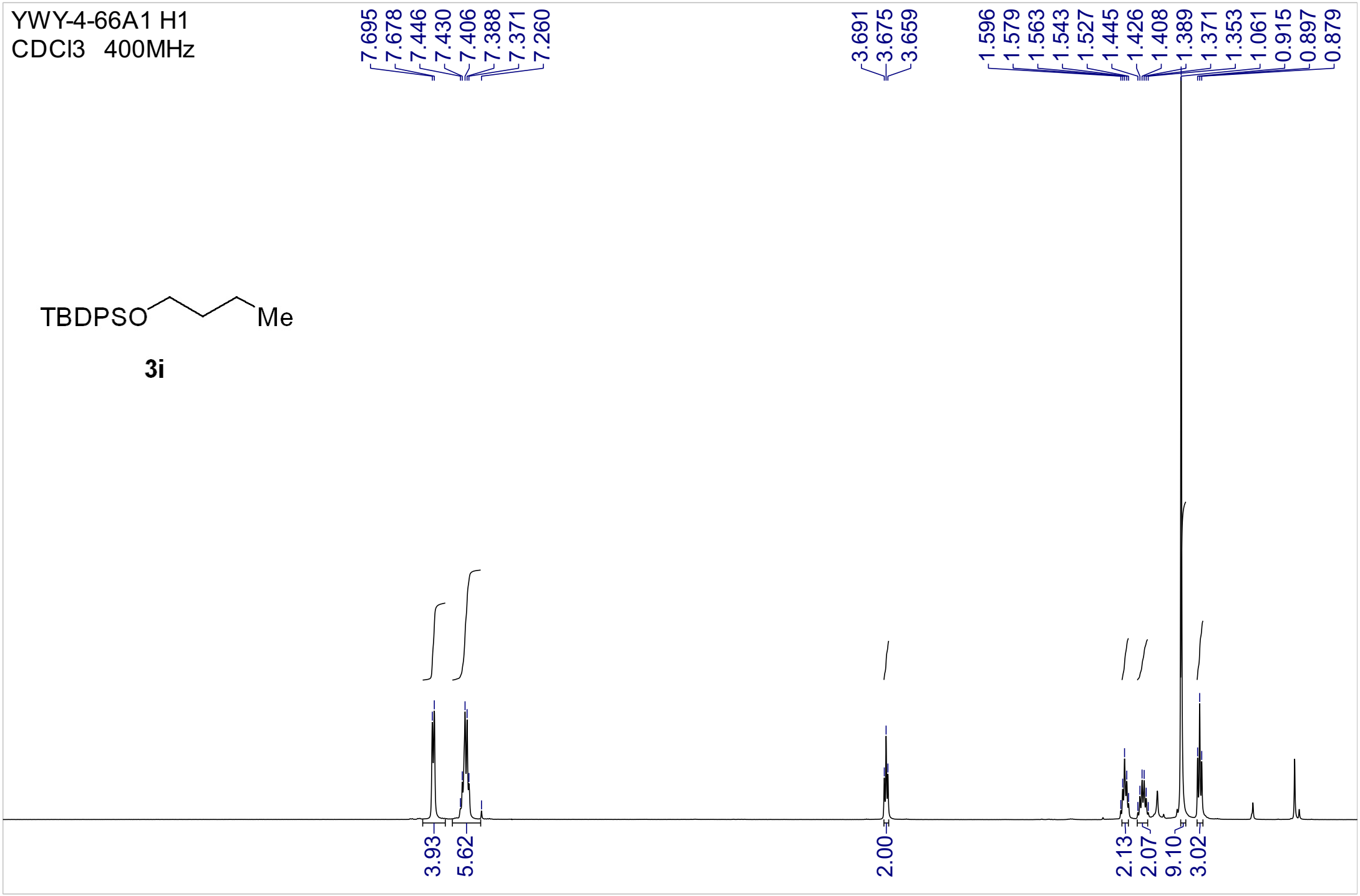
157.167
136.155
128.619
114.351
77.212
77.000
76.788
68.014
31.608
29.321
27.965
25.751
22.613
15.880
14.028



3h

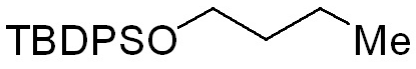




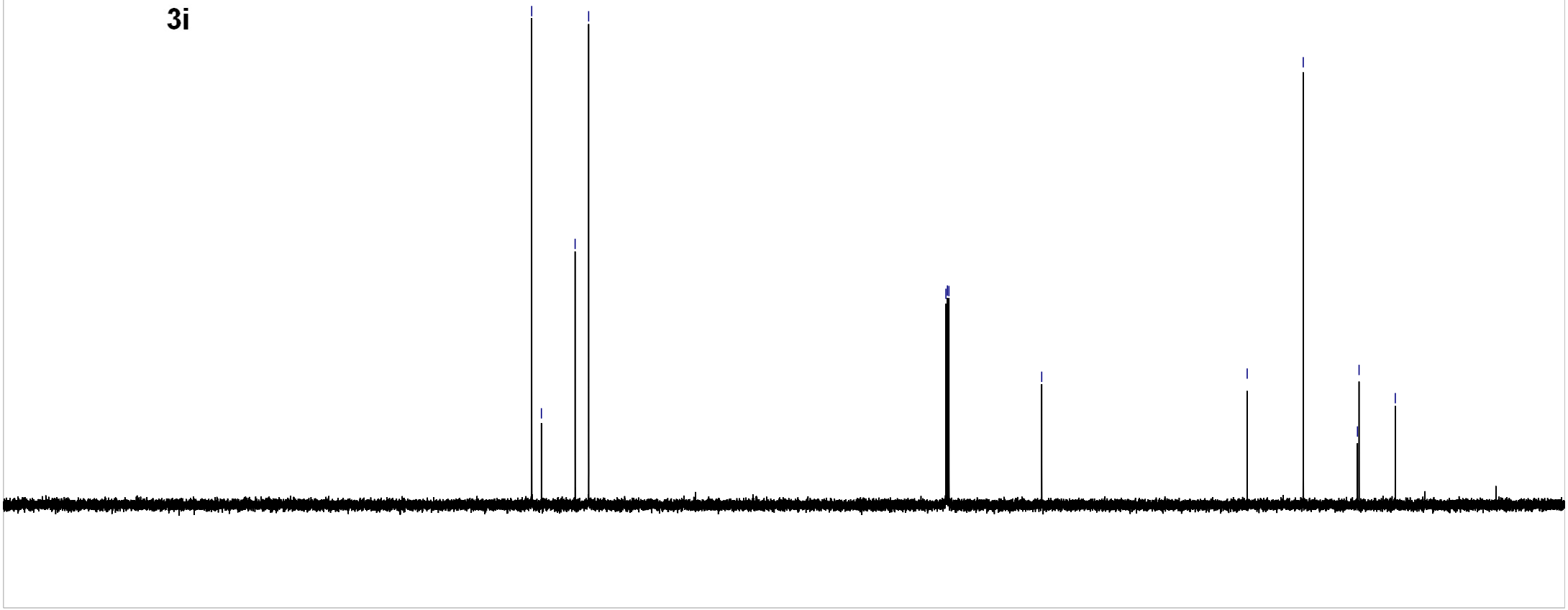

YWY-4-66A1 H1
CDCl3 400MHz
7.695
7.678
7.446
7.430
7.406
7.388
7.371
7.260
3.691
3.675
3.659
1.596
1.579
1.563
1.543
1.527
1.445
1.426
1.408
1.389
1.371
1.353
1.061
0.915
0.897
0.879
TBDPSO
Me
3i
3.93
5.62
2.00
2.13
2.07
9.10
3.02

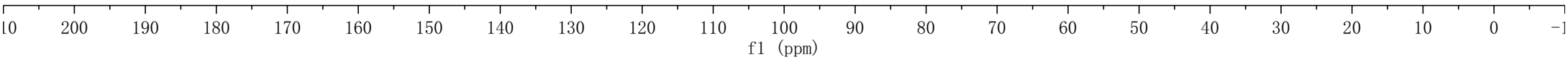
YWY-4-66A1 C13
CDCl3 150MHz

135.568
134.201
129.451
127.546
77.212
77.000
76.788
63.717
34.768
26.871
19.235
19.008
13.903

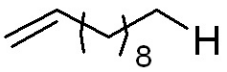


3i





YWY-4-76A3 H1
CDCl3 600MHz



3k

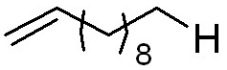
7.260
5.850
5.839
5.833
5.822
5.811
5.804
5.794
5.782
5.008
4.980
4.937
4.921
2.059
2.048
2.036
2.024
1.388
1.377
1.367
1.268
0.894
0.883
0.872

0.83
0.83
0.81
2.00
14.15
3.00

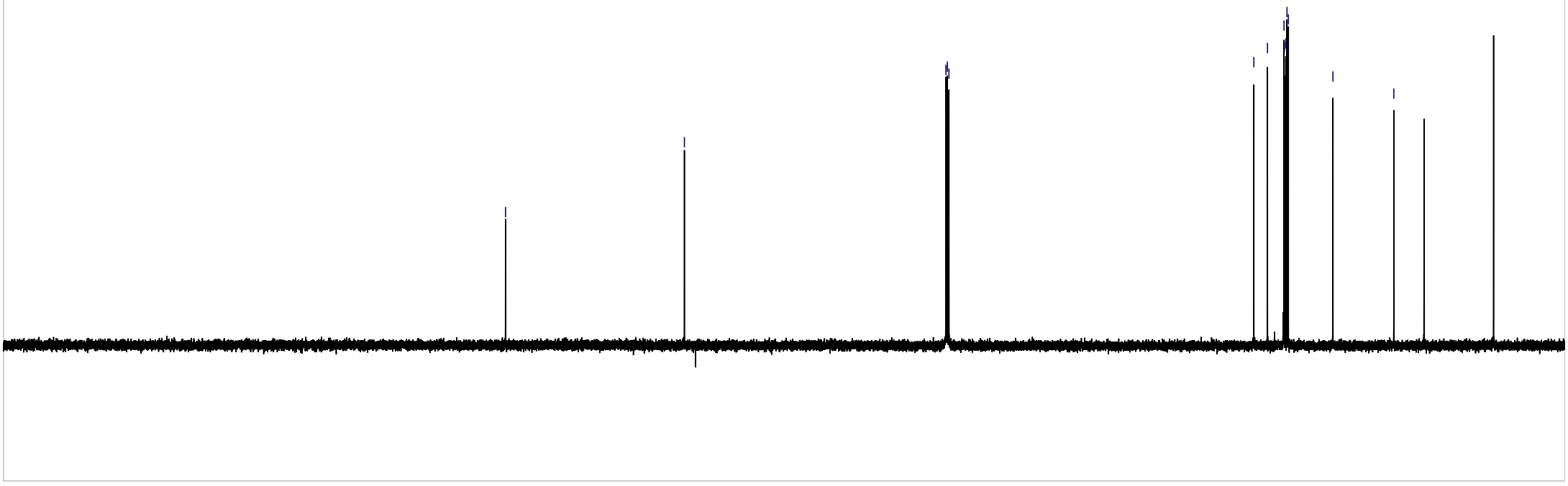
f1 (ppm)

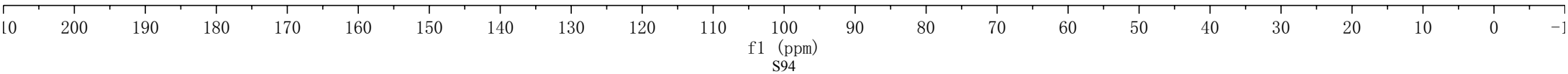
YWY-4-76A3 C13
CDCl3 150MHz

139.257
114.054
77.211
77.000
76.788
33.828
31.912
29.587
29.521
29.337
29.165
28.962
22.687
14.109

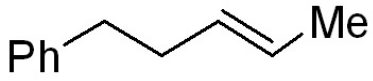


3k





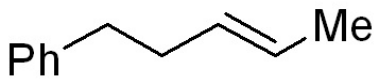
YWY-4-78A3 H1
CDCl3 400MHz



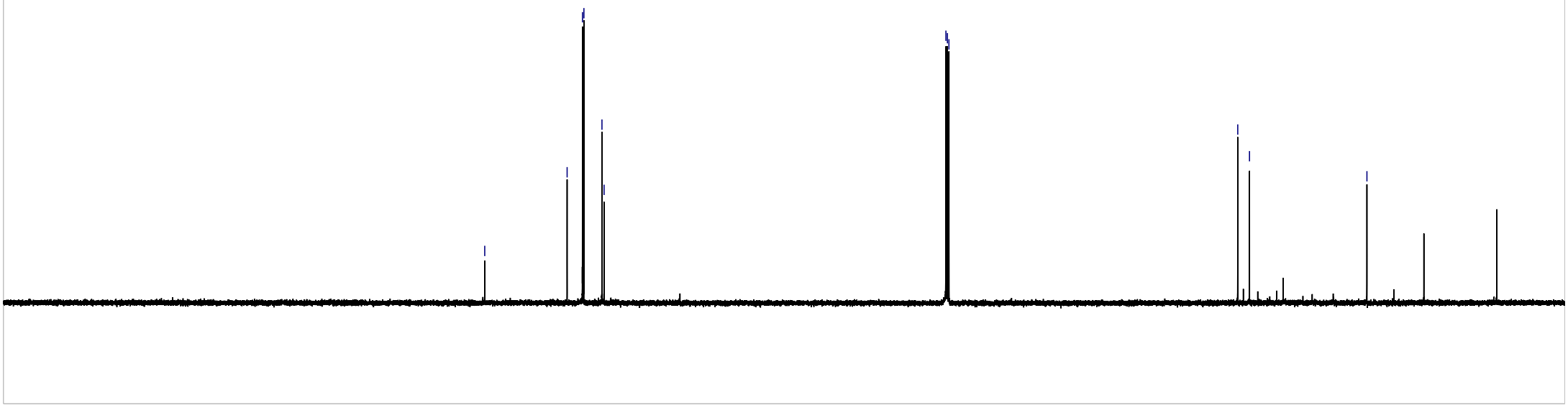
3l

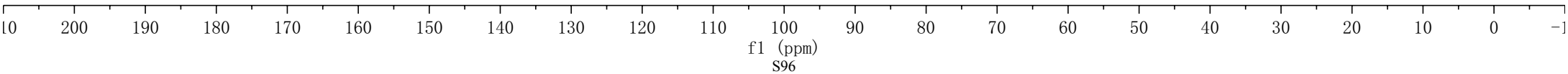
YWY-4-78A3 C13
CDCl3 150MHz

142.184
130.577
128.414
128.212
125.670
125.370
77.212
77.000
76.789
36.098
34.444
17.900

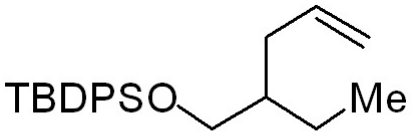


3l





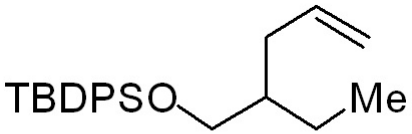
YWY-4-76A1 H1
CDCl3 600MHz



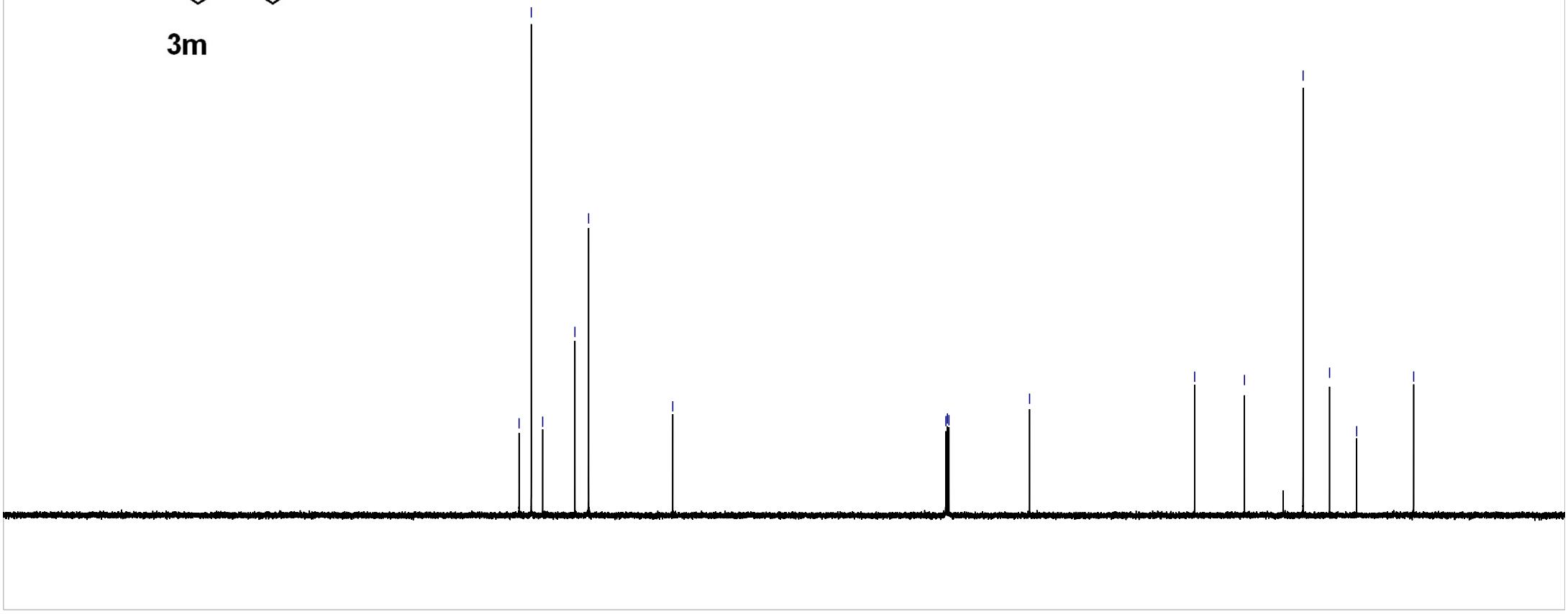
3m

YWY-4-76A1 C13
CDCl3 150MHz

137.345
135.627
134.021
129.479
127.549
115.693
77.212
77.000
76.789
65.427
42.172
35.152
26.891
23.163
19.341
11.312

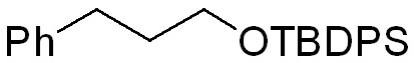


3m



f1 (ppm)

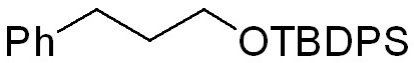
YWY-4-71A5 H1
CDCl3 400MHz



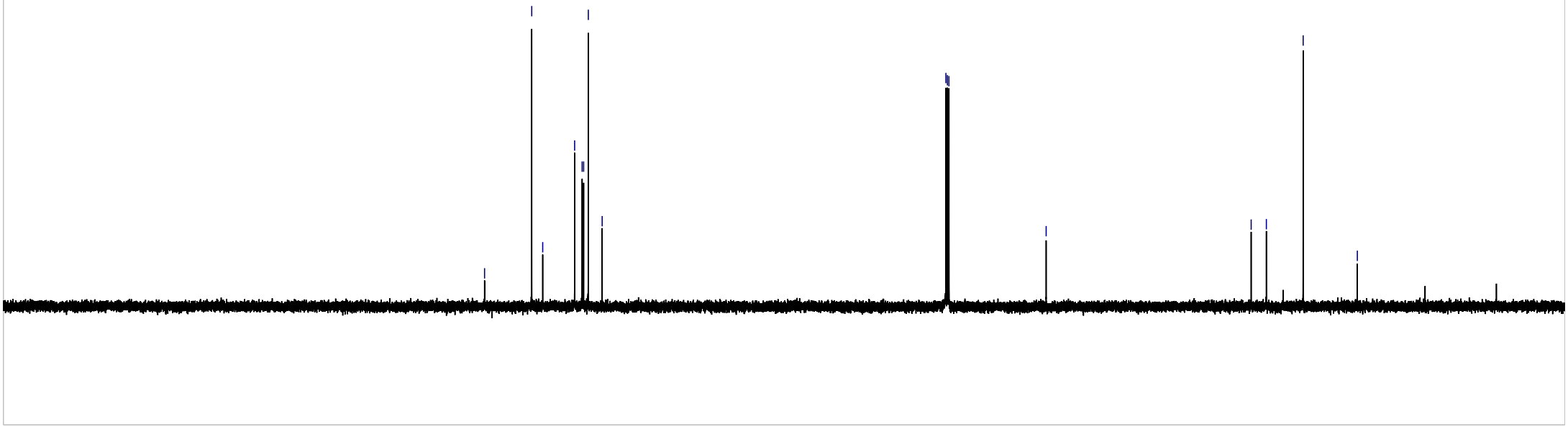
3n

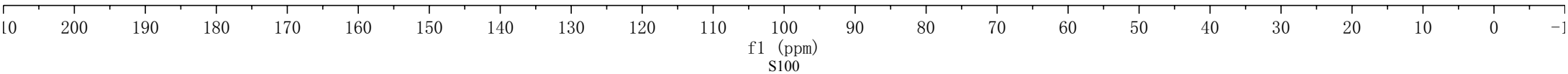
YWY-4-71A5 C13
CDCl3 150MHz

142.208
135.562
134.017
129.519
128.462
128.252
127.591
125.641
77.212
77.000
76.789
63.091
34.209
32.068
26.884
19.252



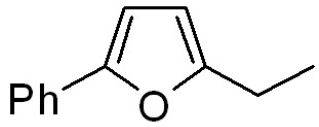
3n





S100

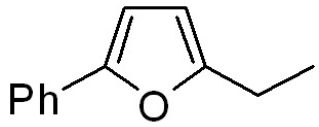
YWY-5-26A3-3 H1
CDCl3 400MHz



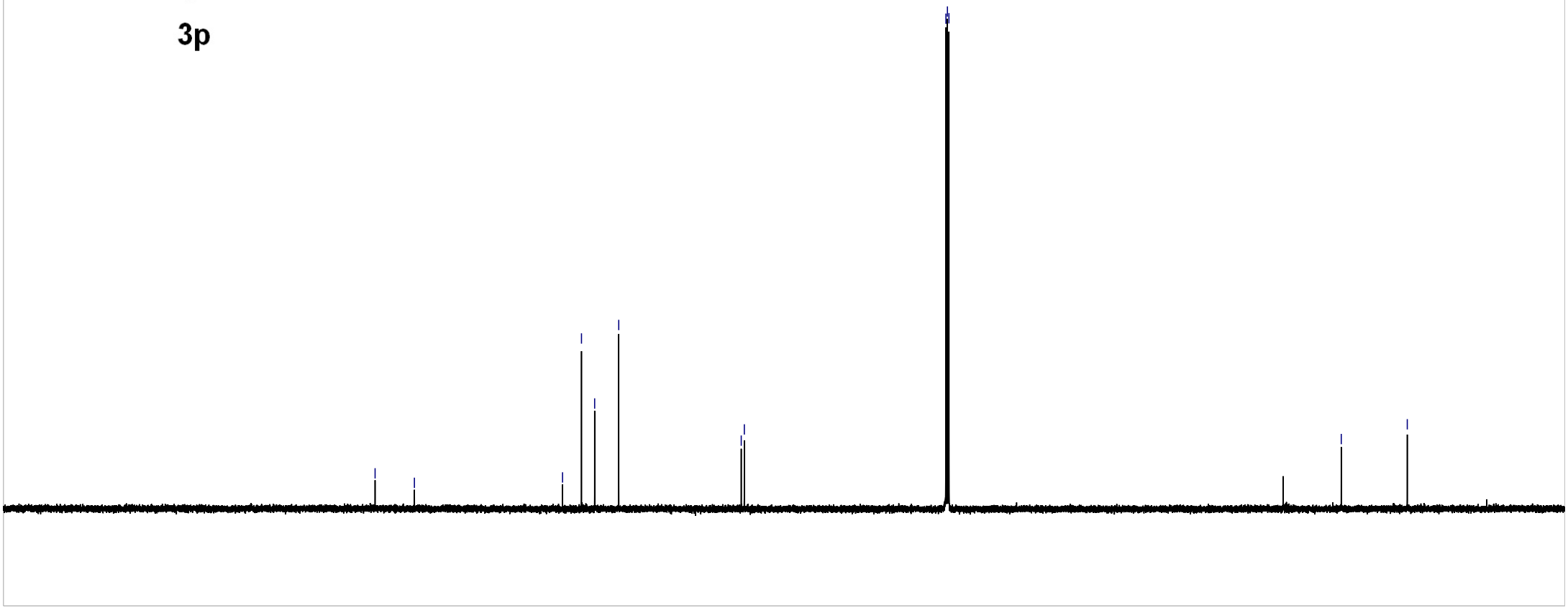
3p

YWY-5-26A3-3 C13
CDCl3 150MHz

157.631
152.093
131.222
128.555
126.699
123.305
106.050
105.612
77.212
77.000
76.788
21.517
12.216

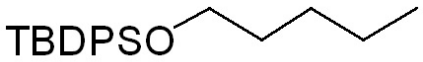


3p



f1 (ppm)

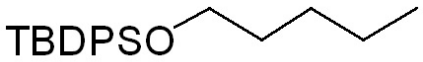
YWY-5-17A3-1 H1
CDCl3 400MHz



3q

YWY-5-17A3-1 C13
CDCl3 150MHz

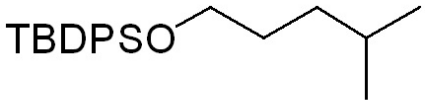
135.560
134.182
129.447
127.540
77.211
77.000
76.788
64.017
32.292
27.991
26.865
22.448
19.220
14.088



3q

f1 (ppm)

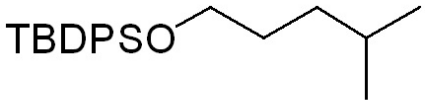
YWY-5-17A4-1 H1
CDCl3 400MHz



3r

YWY-5-17A4-1 C13
CDCl3 150MHz

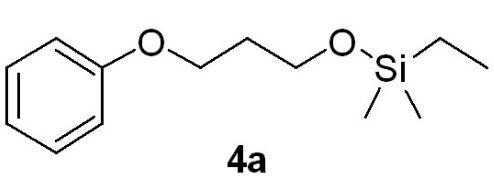
135.564
134.176
129.453
127.542
77.211
77.000
76.788
64.318
34.957
30.432
27.746
26.868
22.608
19.214



3r

f1 (ppm)

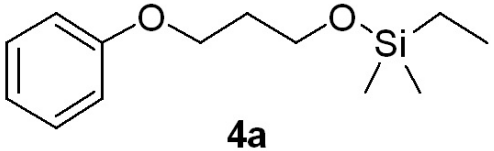
YWY-4-102P2 H1
CDCl3 400MHz



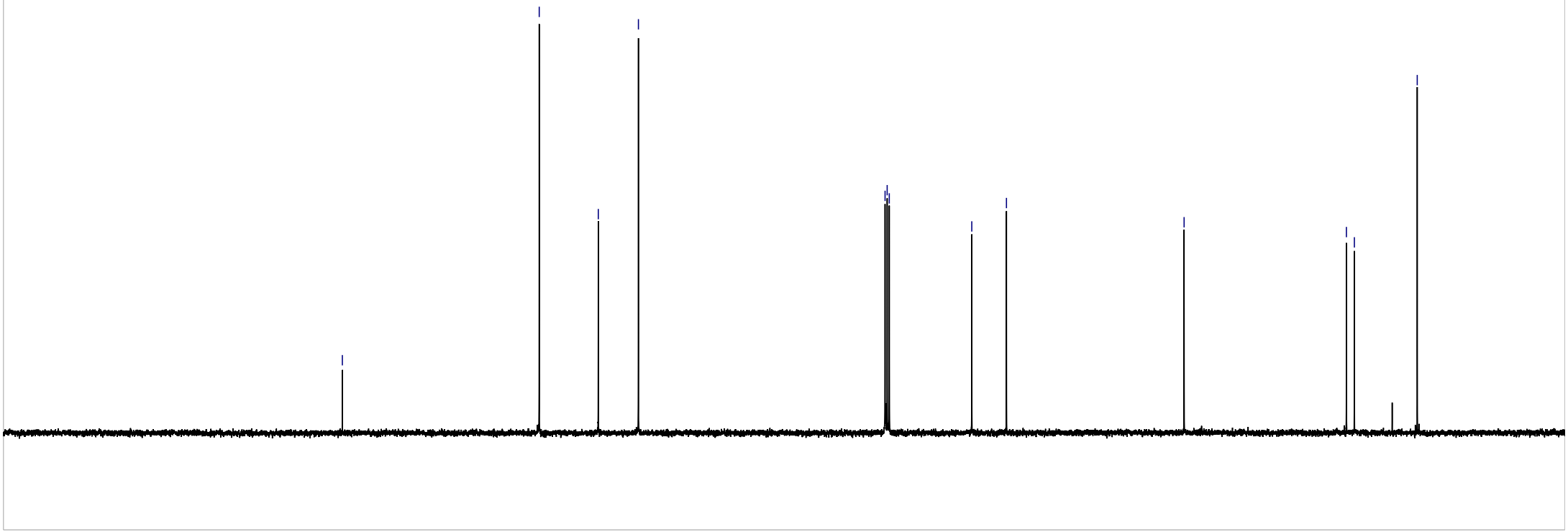
4a

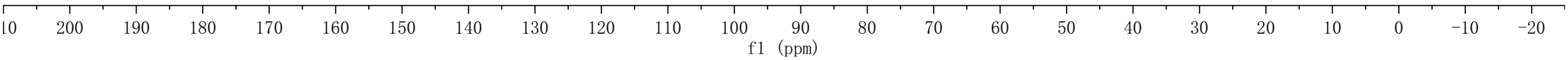
YWY-4-102P2 C13
CDCl3 100MHz

158.987
129.360
120.470
114.433
77.318
77.000
76.682
64.267
59.049
32.315
7.890
6.692
-2.768

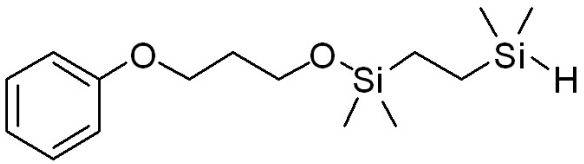


4a





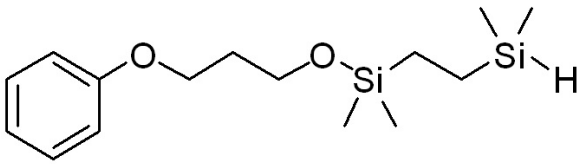
YWY-5-1P H1
CDCl3 400MHz



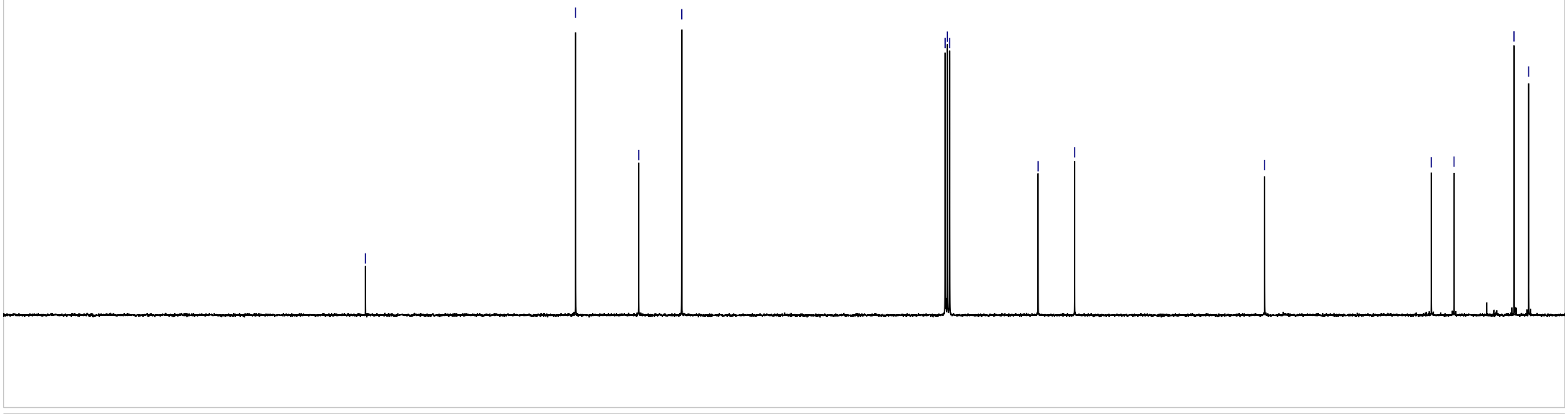
4b

YWY-5-1P C13
CDCl3 100MHz

158.985
129.378
120.491
114.431
77.318
77.000
76.683
64.226
59.082
32.310
8.827
5.630
-2.820
-4.891



4b



f1 (ppm)