Supporting Information

Pd-Catalyzed Cascade Reaction of N-H Insertion and Oxidative Dehydrogenative Aromatization: A New Entry to 2-Amino-phenols

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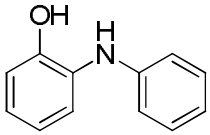
General Information

All experiments were conducted under air unless otherwise noted. Flasks were flame dried and cooled under nitrogen before use. All solvents were dried appropriately. 1,4-dioxane was dried by refluxing from sodium under nitrogen. For column chromatography, 200-300 mesh silica gel was employed. ^{1}H NMR and ^{13}C NMR were recorded on 400MHz or 500 MHz spectrometer in $CDCl_3$ or DMSO-d_6 solution and the chemical shifts were reported in parts per million (δ) relative to internal standard TMS(0 ppm). HRMS were performed using atmospheric pressure chemical ionization (APCI) with a ion-trap analyzer or electron impact mode (EI) with a TOF mass analyzer. IR spectra were recorded on a FTIR-8400S FTIR spectrometer. Melting points were not corrected. Unless otherwise noted, materials obtained from commercial suppliers were used without further purification. All the diazo compounds were prepared according to the references.[1],[2]

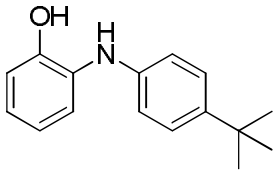
Typical procedure for Pd-catalyzed cascade reaction of N-H insertion and oxidative dehydrogenative aromatization

A reaction tube was charged with palladium(II) catalyst (10 mol%), aniline (0.5 mmol) and 1,4-dioxane (3 mL), then 6-diazo-2cyclohexenone (0.75 mmol) was added, and the reaction mixture was stirred at 60 ^{o}C for 3 h under air. After the mixture was cooled to room temperature, solvents were removed under vacuum. The residue was purified by column chromatography on silica gel to give the desired product.



2-(phenylamino)phenol (3a) [3]

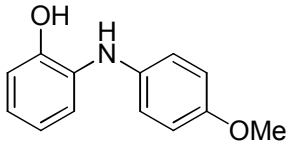
The title compound was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1) to afford pure product as dark brown oil (yield 90%, 83.3 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25-7.18 (m, 3H), 7.10 (t, *J* = 7.7 Hz, 1H), 6.99 (d, *J* = 8.0 Hz, 1H), 6.89 (t, *J* = 6.9 Hz, 2H), 6.78 (d, *J* = 8.0 Hz, 2H), 5.79 (s, 1H), 5.26 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.1, 145.5, 129.5, 129.1, 126.1, 124.7, 121.0, 120.3, 115.9, 115.4. MS (ESI) 186.2 $(M+H)^{+}$.



2-((4-(tert-butyl)phenyl)amino)phenol (3b)

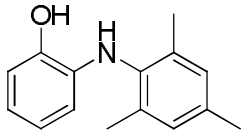
The title compound was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1) to afford pure product as brown oil (yield 83%, 100.2 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 (s, 2H), 7.18 (d, *J* = 7.4 Hz, 1H), 7.12 – 7.05 (m, 1H), 6.98 (d, *J* = 7.0 Hz, 1H), 6.88 (t, *J* = 6.8 Hz, 1H), 6.74 (d, *J* = 7.6 Hz, 2H), 5.77 (s, 1H), 5.20 (s, 1H), 1.29 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 150.8, 143.4, 142.8, 129.7, 126.2, 125.7, 124.3, 121.0, 115.9, 115.3, 77.3, 77.0, 76.8, 34.1, 31.5. HRMS

(EI) calcd for $C_{16}H_{19}NO$ 241.1467, found: 241.1470. IR (KBr, cm^{-1}): 2960, 1607, 1516, 827, 746.



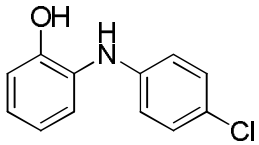
2-((4-methoxyphenyl)amino)phenol (3c) [4]

The title compound was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1) to afford pure product as brown oil (yield 85%, 91.5 mg); ^{1}H NMR (400 MHz, DMSO) δ 9.39 (s, 1H), 7.00 (d, *J* = 8.8 Hz, 2H), 6.97-6.95 (m, 1H), 6.82-6.77 (m, 4H), 6.64-6.61 (m, 2H), 3.68 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 154.7, 149.6, 139.5, 132.1, 124.8, 122.4, 121.1, 119.1, 115.5, 114.9, 55.7. MS (ESI) 216.2 $(M+H)^+$



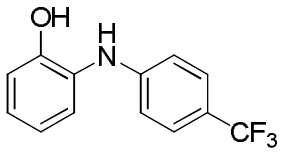
2-(mesitylamino)phenol (3d) [5]

The title compound was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1) to afford pure product as brown oil (yield 80%, 90.9 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.93 (s, 2H), 6.84 (s, 1H), 6.71 (s, 2H), 6.27 (s, 1H), 2.30 (s, 3H), 2.16 (s, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.7, 134.5, 129.3, 121.4, 114.8, 77.3, 77.0, 76.8, 20.9, 18.1. MS (ESI) 228.2 $(M+H)^+$



2-((4-chlorophenyl)amino)phenol (3e)

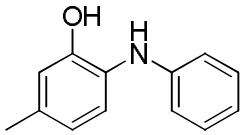
The title compound was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1) to afford pure product as a brown solid (yield 74%, 81.3 mg); Mp: 79-81 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.16 (t, *J* = 7.2 Hz, 3H), 7.10 (t, *J* = 7.7 Hz, 1H), 6.98 (d, *J* = 7.9 Hz, 1H), 6.90 (t, *J* = 7.5 Hz, 1H), 6.72 (d, *J* = 8.6 Hz, 2H), 5.66 (s, 1H), 5.30 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.8, 144.0, 129.3, 128.8, 126.2, 125.1, 124.4, 121.2, 117.1, 115.5. HRMS (APCI) calcd for $C_{12}H_{11}ClNO$ $(M+H)^+$ 220.0524, found: 220.0531. IR (KBr, cm^{-1}): 2924, 1591, 1456, 1091, 820, 748.



2-((4-(trifluoromethyl)phenyl)amino)phenol (3f)

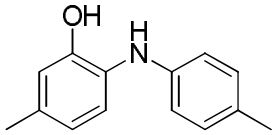
The title compound was purified by column chromatography on silica gel (petroleum ether/dichloromethane = 3/1) to afford pure product as a brown solid (yield 46%, 58.2 mg); Mp: 100-102 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (d, *J* = 8.2 Hz, 2H),

7.21-7.14 (m, 2H), 7.01 (d, J = 8.1 Hz, 1H), 6.93 (t, J = 7.6 Hz, 1H), 6.80 (d, J = 8.2 Hz, 2H), 5.59 (s, 1H), 5.53 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.2, 148.5, 127.5, 127.1, 126.8, 125.9, 125.4, 123.2, 121.3, 115.8, 114.7. HRMS (APCI) calcd for $C_{13}H_{11}F_3NO$ $(M+H)^+$ 254.0787, found: 254.0789. IR (KBr, cm^{-1}): 2926, 1620, 1458, 1327, 1113, 1067, 831, 750.



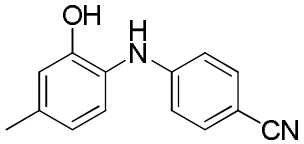
5-methyl-2-(phenylamino)phenol (3g) [6]

The title compound was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1) to afford pure product as a brown solid (yield 88%, 87.7 mg); mp: 63-65 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20 (t, J = 7.8 Hz, 2H), 7.04 (d, J = 7.9 Hz, 1H), 6.86-6.83 (m, 2H), 6.70 (d, J = 8.0 Hz, 3H), 5.84 (s, 1H), 5.06 (s, 1H), 2.32 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.1, 146.4, 137.4, 129.5, 126.1, 125.8, 121.7, 121.7, 119.8, 115.7, 115.1. MS (ESI) 200.2 $(M+H)^+$



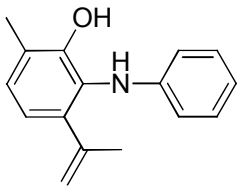
5-methyl-2-(p-tolylamino)phenol (3h) [7]

The title compound was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1) to afford pure product as a brown solid (yield 85%, 90.6 mg); mp: 63-65 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.01 (d, J = 7.4 Hz, 3H), 6.82 (s, 1H), 6.69 (d, J = 7.6 Hz, 1H), 6.63 (d, J = 7.8 Hz, 2H), 5.89 (s, 1H), 4.97 (s, 1H), 2.32 (s, 3H), 2.26 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 151.8, 143.7, 136.9, 129.8, 129.3, 126.4, 125.4, 121.4, 115.7, 115.5, 21.1, 20.4. MS (ESI) 214.2 $(M+H)^+$.



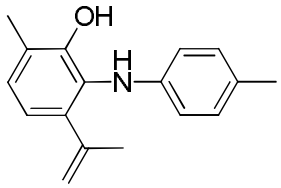
4-((2-hydroxy-4-methylphenyl)amino)benzonitrile (3i)

The title compound was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to afford pure product as a grey solid (yield 53%, 59.4 mg); mp: 139-141 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 (d, J = 8.2 Hz, 2H), 7.06 (d, J = 7.9 Hz, 1H), 6.85 (s, 1H), 6.77 – 6.72 (m, 3H), 5.58 (s, 1H), 5.49 (s, 1H), 2.34 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 150.9, 133.7, 133.6, 125.2, 120.9, 120.4, 117.4, 117.3, 114.1, 114.0, 21.1. HRMS (EI) calcd for $C_{14}H_{12}N_2O$ 224.0950, found: 224.0948. IR (KBr, cm^{-1}): 2924, 1595, 1458, 1377, 752.



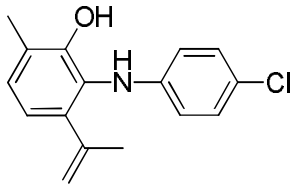
6-methyl-2-(phenylamino)-3-(prop-1-en-2-yl)phenol (3j)

The title compound was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 100/1) to afford pure product as a yellow solid (yield 70%, 83.8 mg); mp: 54-56 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.19 (t, *J* = 7.6 Hz, 2H), 7.03 (d, *J* = 7.7 Hz, 1H), 6.84 (t, *J* = 7.3 Hz, 1H), 6.72 (d, *J* = 7.8 Hz, 1H), 6.62 (d, *J* = 7.8 Hz, 2H), 6.08 (s, 1H), 5.16 (s, 1H), 5.09 (s, 1H), 4.81 (s, 1H), 2.31 (s, 3H), 1.86 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.2, 146.3, 143.2, 139.2, 129.6, 128.4, 124.2, 123.2, 119.9, 119.3, 115.3, 114.5, 24.4, 15.8. HRMS (EI) calcd for $C_{16}H_{17}NO$ 239.1310, found: 239.1311. IR (KBr, cm^{-1}): 3337, 2920, 1601, 1495, 1418, 1306, 1209, 1036, 908, 743, 689.



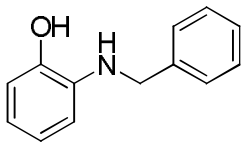
6-methyl-3-(prop-1-en-2-yl)-2-(p-tolylamino)phenol (3k)

The title compound was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 100/1) to afford pure product as brown oil (yield 73%, 92.5 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.00 (t, *J* = 7.7 Hz, 3H), 6.71 (d, *J* = 7.7 Hz, 1H), 6.52 (d, *J* = 7.7 Hz, 2H), 6.08 (s, 1H), 5.28 (s, 1H), 5.08 (s, 2H), 4.81 (s, 1H), 2.29 (s, 3H), 2.24 (s, 3H), 1.86 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 151.1, 143.8, 143.3, 138.9, 130.0, 129.2, 128.1, 124.5, 123.1, 119.3, 115.3, 114.7, 77.3, 77.0, 76.8, 24.2, 20.5, 15.9. HRMS (EI) calcd for $C_{17}H_{19}NO$ 253.1467, found: 253.1469. IR (KBr, cm^{-1}): 3379, 2920, 1614, 1514, 1427, 1219, 1036, 895, 812.



2-((4-chlorophenyl)amino)-6-methyl-3-(prop-1-en-2-yl)phenol (3l)

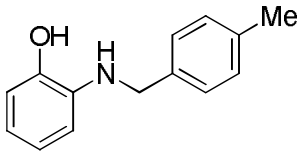
The title compound was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 100/1) to afford pure product as brown oil (yield 60%, 82.1 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.16 (d, *J* = 8.0 Hz, 2H), 7.06 (d, *J* = 7.8 Hz, 1H), 6.75 (d, *J* = 7.7 Hz, 1H), 6.56 (d, *J* = 8.0 Hz, 2H), 6.02 (s, 1H), 5.32 (s, 1H), 5.18 (s, 1H), 5.11 (s, 1H), 4.81 (s, 1H), 2.32 (s, 3H), 1.88 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 151.1, 145.0, 143.0, 139.2, 129.3, 128.7, 124.7, 123.7, 123.4, 119.5, 115.8, 115.5, 77.3, 77.0, 76.8, 24.2, 15.9. HRMS (EI) calcd for $C_{16}H_{16}ClNO$ 273.0920, found: 273.0925. IR (KBr, cm^{-1}): 3370, 2920, 1608, 1504, 1210, 1036, 899.



2-(benzylamino)phenol (5a) [8]

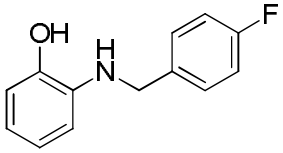
The title compound was purified by column chromatography on silica gel (petroleum

ether/ethyl acetate = 50/1) to afford pure product as light green solid (yield 80%, 79.7 mg). Mp: 69-71 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.33 (m, 4H), 7.28 (d, *J* = 6.8 Hz, 1H), 6.83 (t, *J* = 7.0 Hz, 1H), 6.73 (d, *J* = 7.2 Hz, 1H), 6.68-6.63 (m, 2H), 4.59 (s, 1H), 4.35 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.4, 139.3, 136.9, 128.6, 127.6, 127.2, 121.8, 117.8, 114.3, 112.5, 48.6. MS (ESI) 200.2 $(M+H)^+$.



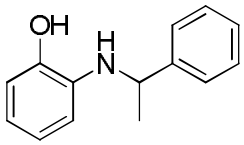
2-((4-methylbenzyl)amino)phenol (5b)

The title compound was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1) to afford pure product as light green solid (yield 82%, 87.4 mg). Mp: 71-73 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 (d, *J* = 7.6 Hz, 1H), 7.16 (d, *J* = 7.6 Hz, 1H), 6.84 (t, *J* = 7.0 Hz, 1H), 6.74-6.62 (m, 3H), 4.61 (s, 1H), 4.31 (s, 2H), 2.35 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 143.5, 137.1, 136.8, 136.4, 129.3, 127.6, 121.7, 117.7, 114.3, 112.5, 48.3, 21.1. HRMS (EI) calcd for $C_{14}H_{15}NO$ 213.1154, found: 213.1156. IR (KBr, cm^{-1}): 2922, 1610, 1514, 1487, 1250, 1111, 802, 741. IR (KBr, cm^{-1}): 2924, 1622, 1529, 1454, 1242, 1126, 798, 689.



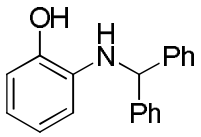
2-((4-fluorobenzyl)amino)phenol (5c)

The title compound was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1) to afford pure product as light green solid (yield 75%, 81.5 mg). Mp: 75-77 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37-7.33 (m, 2H), 7.03 (t, *J* = 8.6 Hz, 2H), 6.83 (t, *J* = 7.4 Hz, 1H), 6.73 (d, *J* = 8.0 Hz, 1H), 6.63 (d, *J* = 6.8 Hz, 2H), 4.60 (s, 1H), 4.32 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 163.0, 161.1, 143.3, 136.8, 129.1, 129.0, 121.7, 117.8, 115.5, 115.3, 114.3, 112.3, 47.8. HRMS (EI) calcd for $C_{13}H_{12}FNO$ 217.0903, found: 217.0907. IR (KBr, cm^{-1}): 2926, 1603, 1510, 1223, 1155, 822, 743.



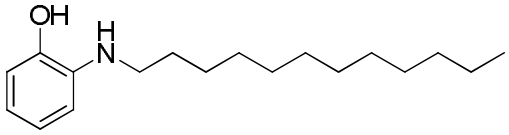
2-((1-phenylethyl)amino)phenol (5d) [9]

The title compound was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1) to afford pure product as light green solid (yield 71%, 75.7 mg). Mp: 122-124 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (d, *J* = 7.6 Hz, 2H), 7.31 (t, *J* = 7.4 Hz, 2H), 7.24-7.20 (m, 1H), 6.69 (s, 2H), 6.57 (s, 1H), 6.44 (d, *J* = 7.2 Hz, 1H), 4.46 (s, 2H), 1.53 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.2, 143.5, 135.9, 128.6, 126.9, 125.9, 121.5, 117.7, 114.1, 113.7, 53.9, 24.9. MS (ESI) 214.2 $(M+H)^+$.



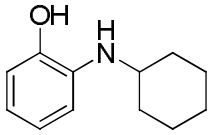
2-(benzhydrylamino)phenol (5e) [10]

The title compound was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1) to afford pure product as light green solid (yield 66%, 90.9 mg). Mp: 128-130 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 (d, J = 7.2 Hz, 4H), 7.34 (t, J = 7.2 Hz, 4H), 7.26-7.23 (m, 2H), 6.71 (d, J = 7.6 Hz, 2H), 6.59 (t, J = 7.2 Hz, 1H), 6.45 (d, J = 7.6 Hz, 1H), 5.50 (s, 1H), 4.68 (s, 1H), 4.58 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.1, 143.0, 136.3, 128.7, 127.5, 127.3, 121.7, 117.6, 114.2, 113.2, 63.1. MS (ESI) 276.2 $(M+H)^+$.



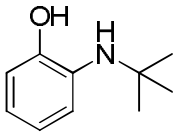
2-(dodecylamino)phenol (5f)

The title compound was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1) to afford pure product as light green solid (yield 79%, 109.6 mg). Mp: 70-72 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.86 (s, 1H), 6.69 (d, J = 7.2 Hz, 2H), 6.62 (s, 1H), 4.38 (s, 2H), 3.10 (s, 2H), 1.64 (t, J = 7.2 Hz, 2H), 1.27 (s, 16H), 0.88 (t, J = 6.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.7, 137.3, 121.6, 117.4, 114.3, 112.4, 44.5, 31.9, 29.7, 29.6, 29.5, 29.4, 27.2, 22.7, 14.1. HRMS (EI) calcd for $C_{18}H_{31}NO$ 277.2406, found: 277.2403. IR (KBr, cm^{-1}): 2920, 1605, 1512, 1230, 825, 749.



2-(cyclohexylamino)phenol (5g) [11]

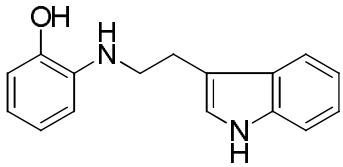
The title compound was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1) to afford pure product as a brown solid (yield 84%, 80.3 mg). Mp: 65-67 ^{o}C; ^{1}H NMR (400 MHz, DMSO) δ 9.19 (s, 1H), 6.61 (dd, J = 16.7, 7.7 Hz, 2H), 6.49 (d, J = 7.7 Hz, 1H), 6.36 (t, J = 7.5 Hz, 1H), 4.15 (s, 1H), 3.17 (s, 1H), 1.91 (d, J = 10.9 Hz, 2H), 1.67 (d, J = 13.0 Hz, 2H), 1.57 (d, J = 12.4 Hz, 1H), 1.32 (dd, J = 24.4, 12.2 Hz, 2H), 1.21-1.09 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.0, 135.5, 128.4, 121.3, 118.4, 114.9, 52.8, 33.4, 26.0, 25.0. MS (ESI) 192.2 $(M+H)^+$.



2-(tert-butylamino)phenol (5h) [12]

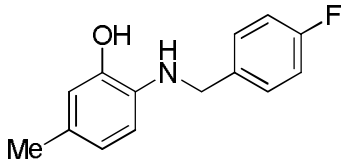
The title compound was purified by column chromatography on silica gel (petroleum

ether/ethyl acetate = 50/1) to afford pure product as a yellow solid (yield 60%, 49.6 mg). Mp: 80-82 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.05-7.00 (m, 2H), 6.89 (d, *J* = 8.0 Hz, 1H), 6.78 (t, *J* = 7.4 Hz, 1H), 1.21 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 153.1, 131.0, 126.9, 125.6, 119.4, 113.9, 53.3, 29.7. MS (ESI) 166.2 $(M+H)^+$.



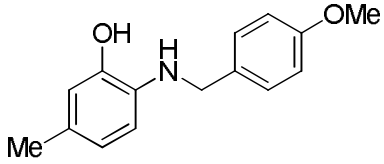
2-((2-(1H-indol-3-yl)ethyl)amino)phenol (5i)

The title compound was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1) to afford pure product as a white solid (yield 77%, 97.1 mg). Mp: 84-86 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (s, 1H), 7.64 (d, *J* = 8.0 Hz, 1H), 7.38 (d, *J* = 8.0 Hz, 1H), 7.21 (t, *J* = 7.4 Hz, 1H), 7.13 (t, *J* = 7.4 Hz, 1H), 7.07 (s, 1H), 6.87 (t, *J* = 6.4 Hz, 1H), 6.73 (dd, *J* = 7.2 Hz, 2H), 6.64 (d, *J* = 7.2 Hz, 1H), 4.42 (s, 2H), 3.47 (s, 2H), 3.12 (t, *J* = 6.8 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 143.9, 136.4, 127.4, 122.1, 122.0, 119.4, 118.8, 117.9, 114.4, 113.5, 112.8, 111.2, 44.5, 25.3. HRMS (EI) calcd for $C_{16}H_{16}N_2O$ 252.1263, found: 252.1269. IR (KBr, cm^{-1}): 3396, 2912, 1599, 1510, 1456, 1231, 1128, 750, 739.



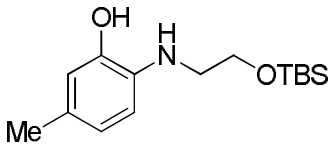
2-((4-fluorobenzyl)amino)-5-methylphenol (5j)

The title compound was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1) to afford pure product as a yellow solid (yield 70%, 80.9 mg). Mp: 117-119 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34-7.31 (m, 2H), 7.01 (t, *J* = 8.4 Hz, 2H), 6.61-6.55 (m, 3H), 4.26 (s, 2H), 2.21 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.4, 160.9, 144.4, 136.2, 134.0, 129.1, 121.8, 115.5, 115.3, 113.7, 48.5, 20.6. HRMS (EI) calcd for $C_{14}H_{14}FNO$ 231.1059, found: 231.1062. IR (KBr, cm^{-1}): 3039, 1614, 1522, 1244, 1126, 818.



2-((4-methoxybenzyl)amino)-5-methylphenol (5k)

The title compound was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1) to afford pure product as a yellow solid (yield 78%, 94.9 mg). Mp: 100-102 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 (d, *J* = 8.4 Hz, 2H), 6.87 (d, *J* = 8.4 Hz, 2H), 6.62 (s, 1H), 6.57 (s, 1H), 4.22 (s, 2H), 3.80 (s, 3H), 2.21 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 163.3, 160.8, 144.1, 129.2, 129.1, 121.7, 115.5, 115.3, 113.6, 102.1. HRMS (EI) calcd for $C_{15}H_{17}NO_2$ 243.1259, found: 243.1255. IR (KBr, cm^{-1}): 3390, 1620, 1529, 1512, 1236, 1128, 1018, 825, 798.

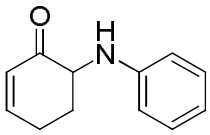


2-((2-((tert-butyldimethylsilyl)oxy)ethyl)amino)-5-methylphenol (5l)

The title compound was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1) to afford pure product as a white solid (yield 88%, 123.8 mg). Mp: 83-85 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.69-6.59 (m, 3H), 3.80 (t, J = 5.2 Hz, 2H), 3.16 (s, 2H), 2.22 (s, 3H), 0.92 (s, 9H), 0.09 (s, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 146.1, 133.9, 129.7, 121.2, 115.8, 115.6, 61.9, 48.0, 25.9, 20.7, 18.3, -5.34. HRMS (EI) calcd for $C_{15}H_{27}NO_2Si$ 281.1811, found: 281.1812. IR (KBr, cm^{-1}): 3033, 2161, 1624, 1510, 1210, 1110, 815, 780.

Procedure for the synthesis of 3a' (the controlled experiment)

Under a nitrogen atmosphere, a reaction tube was charged with palladium(II) catalyst (10 mol%), aniline (0.5 mmol) and 1,4-dioxane (3 mL), then 6-diazo-2cyclohexenone (0.75 mmol) was added, and the reaction mixture was stirred at 60 ^{o}C for 3 h. After the mixture was cooled to room temperature, solvents were removed under vacuum. The residue was purified by column chromatography on silica gel to give the desired product.



6-(phenylamino)cyclohex-2-enone (3a')

The title compound was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1) to afford pure product as brown oil (yield 93%, 87.1 mg, Scheme 3); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20 (t, J = 7.9 Hz, 2H), 7.03 – 6.99 (m, 1H), 6.74 (t, J = 7.3 Hz, 1H), 6.67 (d, J = 7.9 Hz, 2H), 6.14 (dd, J = 10.0, 2.6 Hz, 1H), 5.02 (s, 1H), 4.01 (dd, J = 13.6, 4.4 Hz, 1H), 2.69-2.49 (m, 3H), 1.89-1.78 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.6, 150.2, 147.0, 129.8, 128.2, 118.1, 112.9, 59.5, 30.4, 26.0. HRMS (EI) calcd for $C_{12}H_{13}NO$ 187.0997, found: 187.0995.

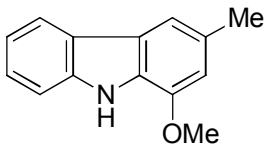
Procedure for the reaction of 1a with phenol

A reaction tube was charged with palladium(II) catalyst (10 mol%), phenol (0.5 mmol) and 1,4-dioxane (3 mL), then 6-diazo-2cyclohexenone (0.75 mmol) was added, and the reaction mixture was stirred at 60 ^{o}C for 12 h under air.

Procedure for the synthesis of Murrayafoline A[6], [13]

A reaction tube was charged with palladium(II) chloride (1 mmol), aniline (**2a**) (10 mmol) and 1,4-dioxane (20 mL), then 6-diazo-2cyclohexenone (15 mmol) was added, and the reaction mixture was stirred at 60 ^{o}C for 5 h under air. After the mixture was cooled to room temperature, solvents were removed under vacuum. The residue was purified by column chromatography on silica gel to give the desired product. A

mixture of the purified product (**3g**), MeI (3 equiv.) and K_2CO_3 (5 equiv.) in anhydrous acetone (35 mL) was heated to 60 ^oC for 2.5 h. After cooling to room temperature, the resulting mixture was filtered through a pad of celite, eluting with ethyl acetate. The solvent was removed under vacuum to give the methylation intermediates in quantitive yield. Then the methylation intermediates, K_2CO_3 (0.1 equiv.), $Pd(OAc)_2$ (0.05 equiv.) and pivalic acid were added to a reaction tube. The tube was placed in an oil bath (110 ^oC) and the mixture was stirred under air for 14h. The solution is then cooled to rt, diluted with CH_2Cl_2, washed with a saturated aqueous solution of Na_2CO_3, dried over $MgSO_4$, filtered, and evaporated under vacuum. The crude product was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 100/1) to give the desired product.



1-methoxy-3-methyl-9H-carbazole[6],[13]

The title compound was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 100/1) to afford pure product as yellow oil (yield 57%, 1.2 g); ^1H NMR (400 MHz, $CDCl_3$) δ 8.15 (s, 1H), 8.01 (d, *J* = 7.8 Hz, 1H), 7.47 (s, 1H), 7.43-7.38 (m, 2H), 7.22-7.19 (m, 1H), 6.73 (s, 1H), 3.98 (s, 3H), 2.53 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.5, 139.5, 129.4, 128.1, 125.6, 124.3, 123.6, 120.4, 119.1, 112.5, 110.8, 107.6, 55.4, 21.7.

References:

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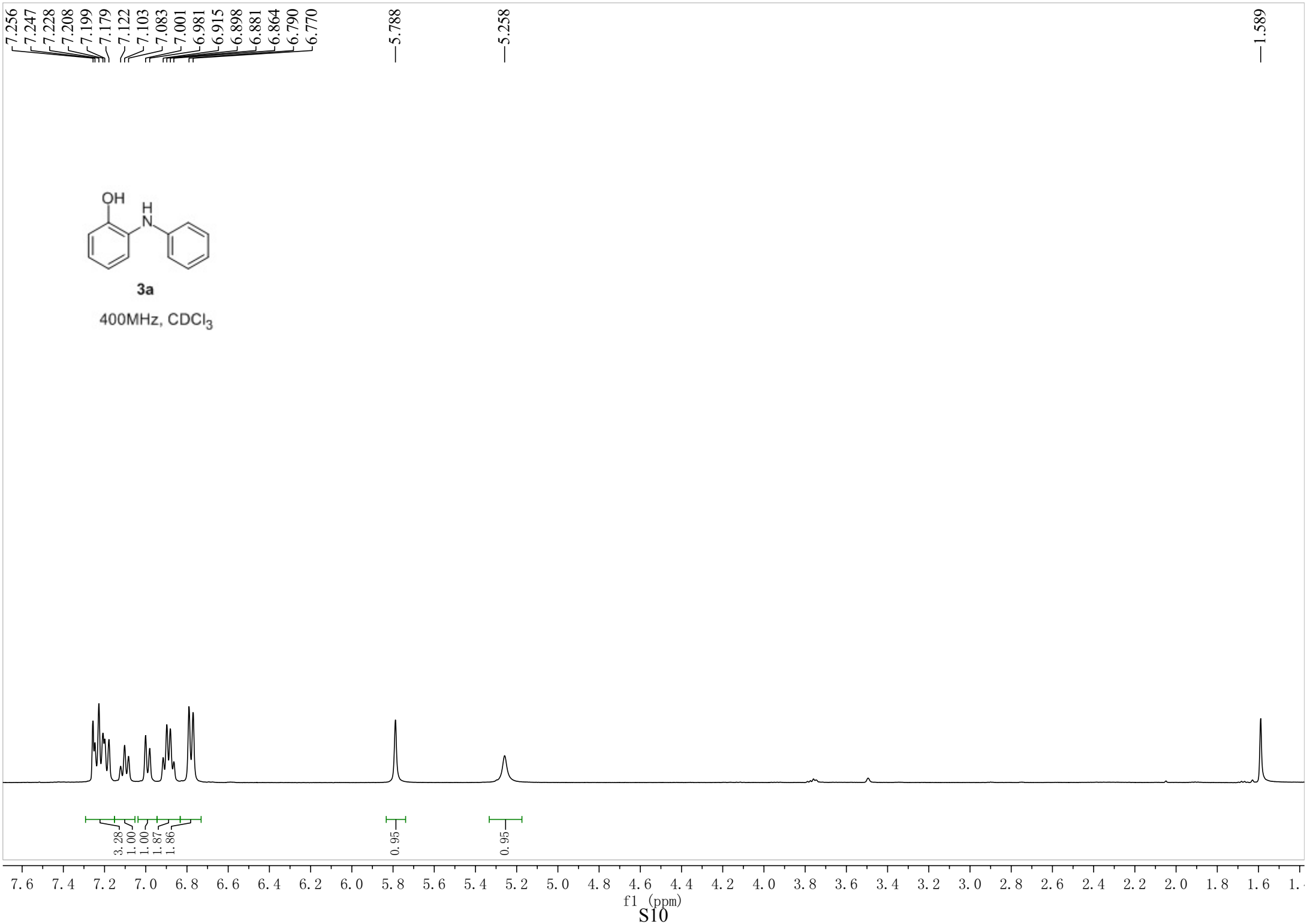
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6.790
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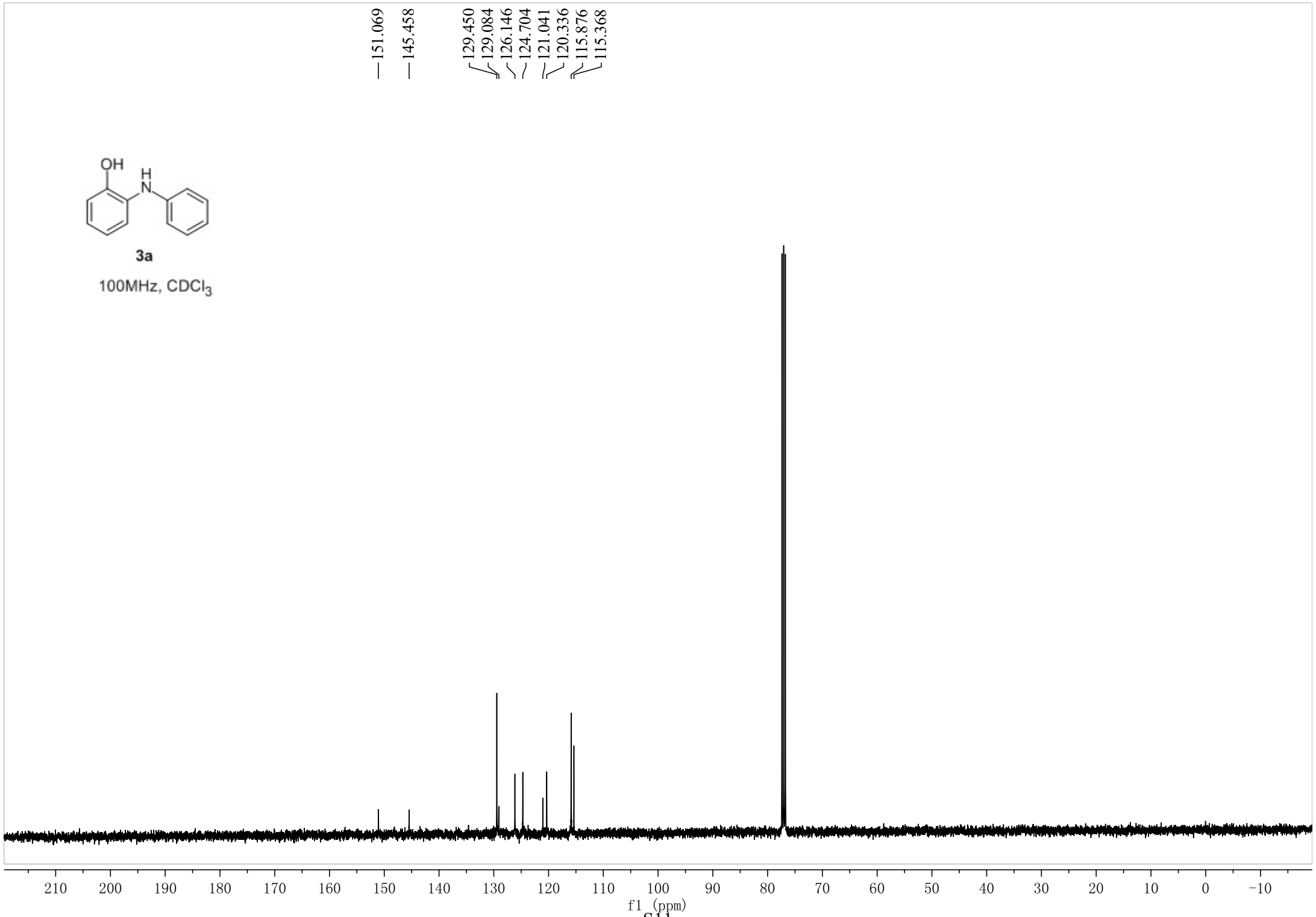
5.788
5.258
1.589
OH
H
N
3a
400MHz, CDCl3
3.28
1.00
1.00
1.87
1.86
0.95
0.95
7.6 7.4 7.2 7.0 6.8 6.6 6.4 6.2 6.0 5.8 5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2 4.0 3.8 3.6 3.4 3.2 3.0 2.8 2.6 2.4 2.2 2.0 1.8 1.6
f1 (ppm)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200
190
180
170
160
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100
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80
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40
30
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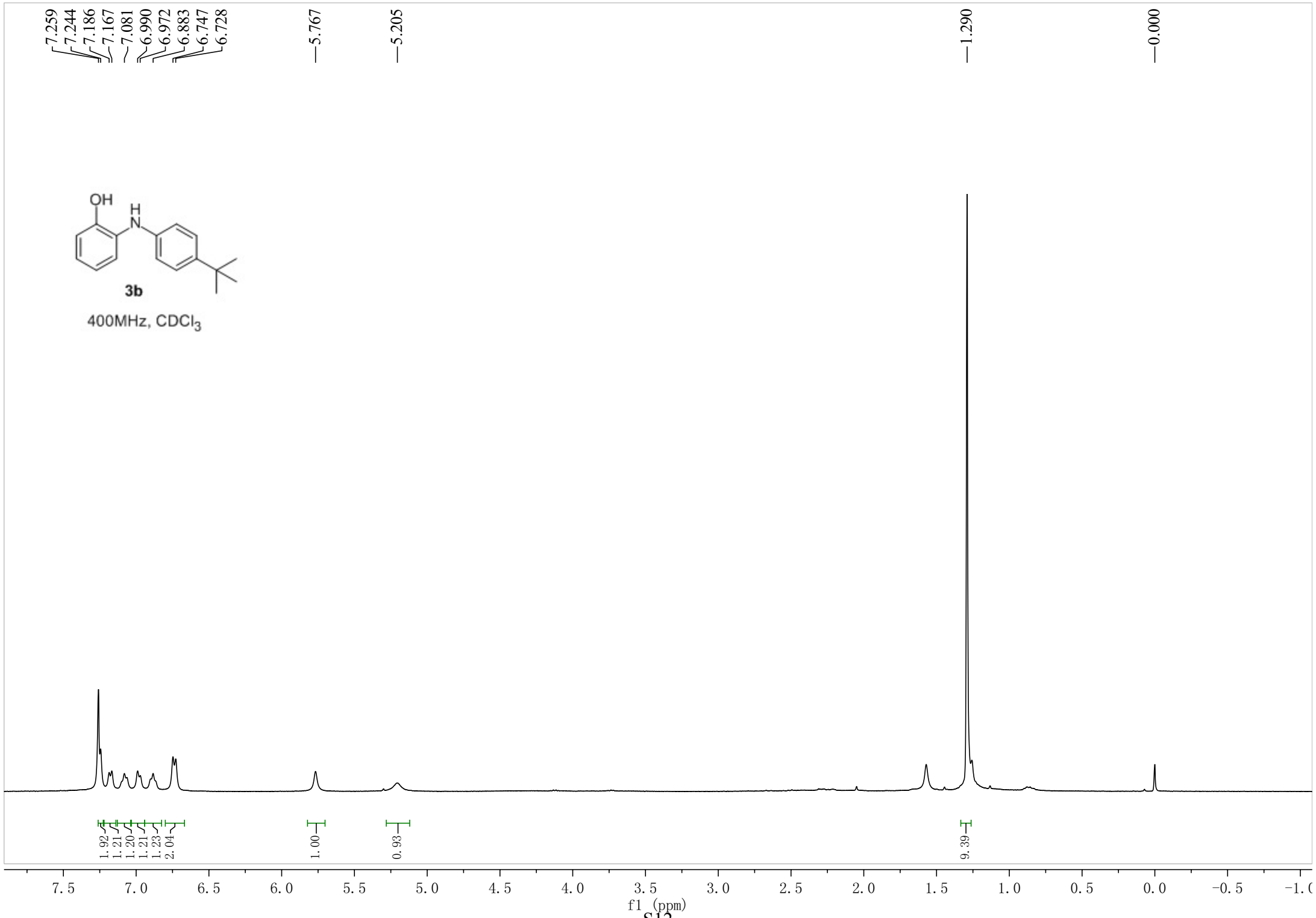
f1 (ppm)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3b
400MHz, CDCl3
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2.04
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6.0
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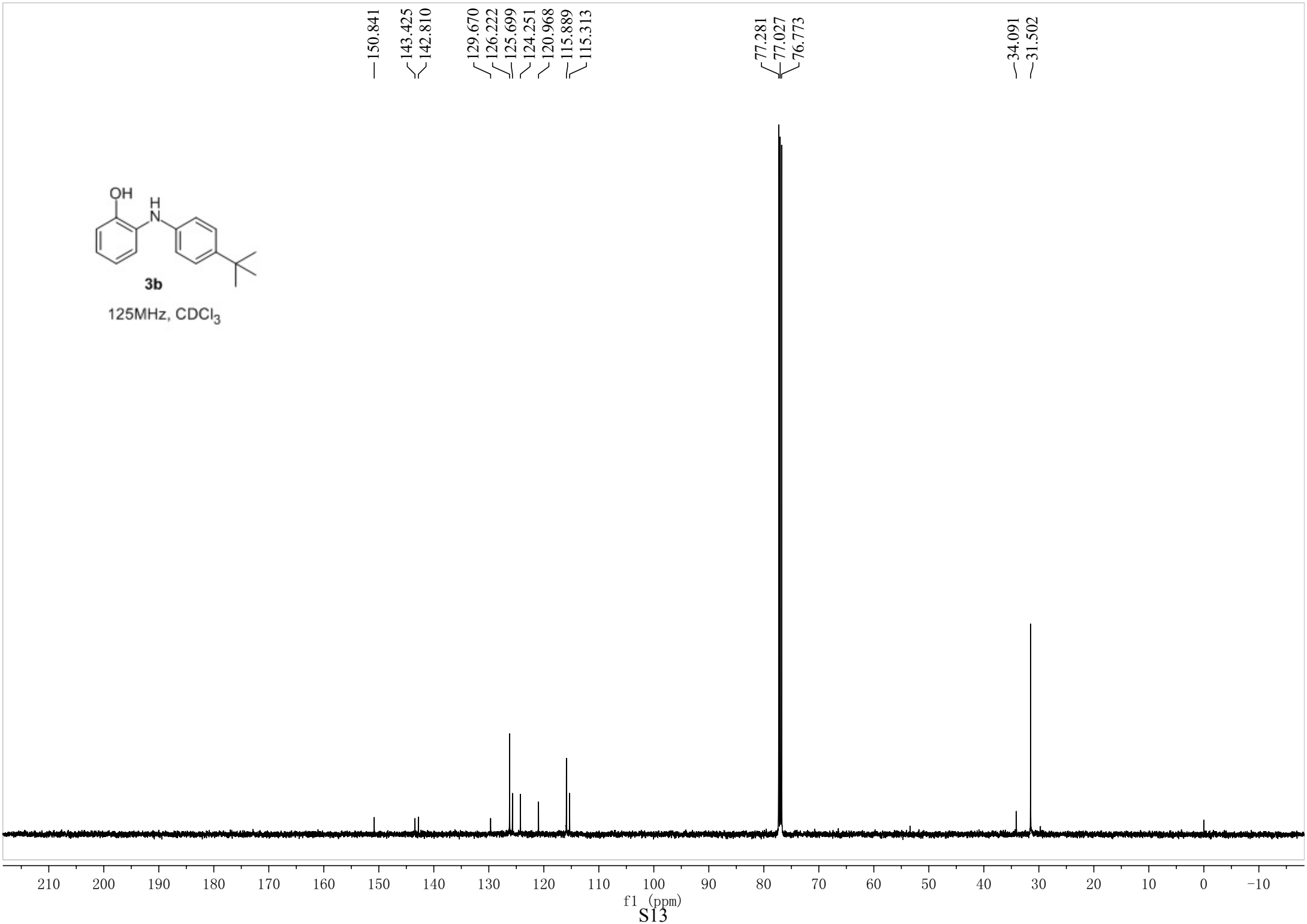
f1 (ppm)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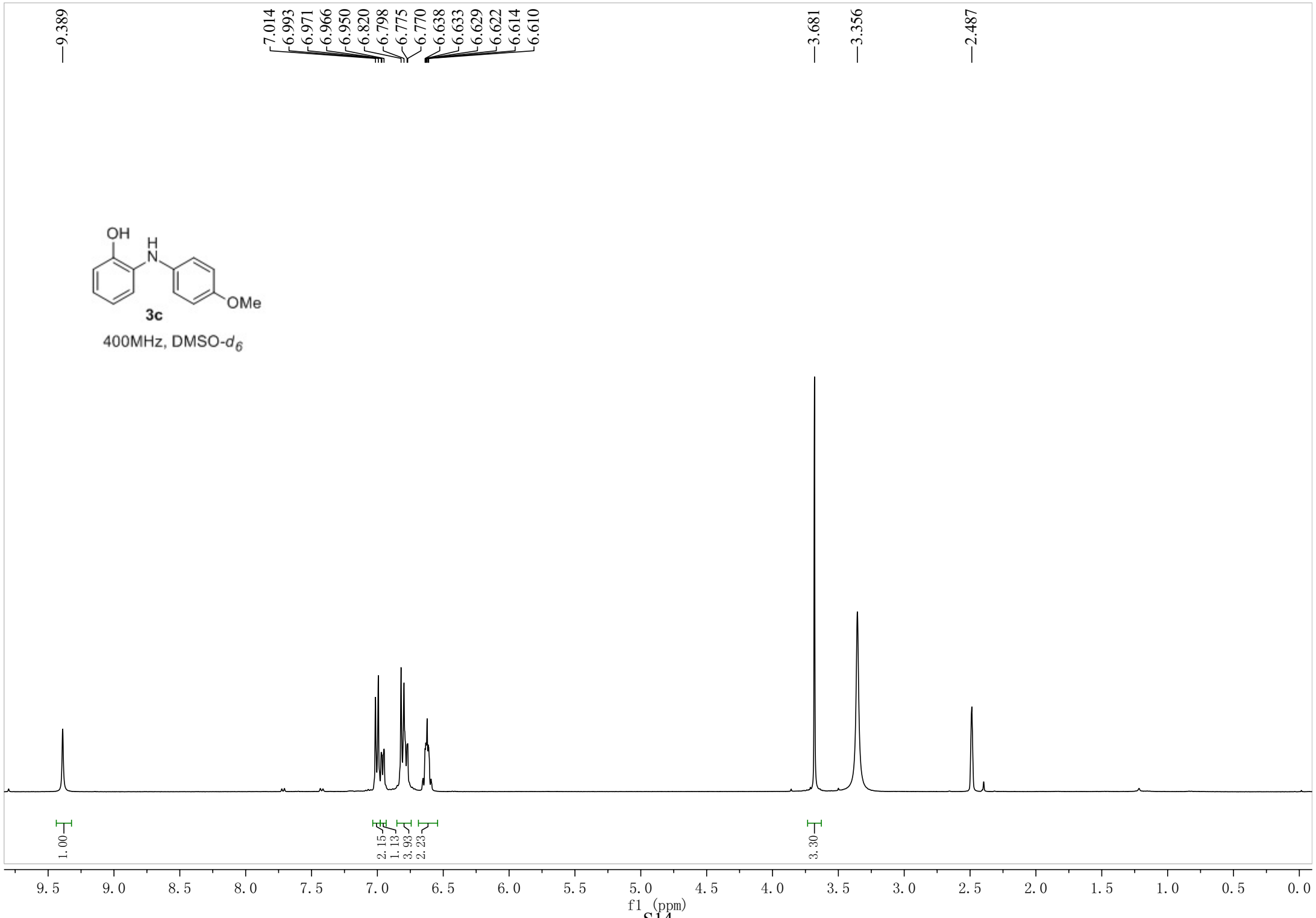
f1 (ppm)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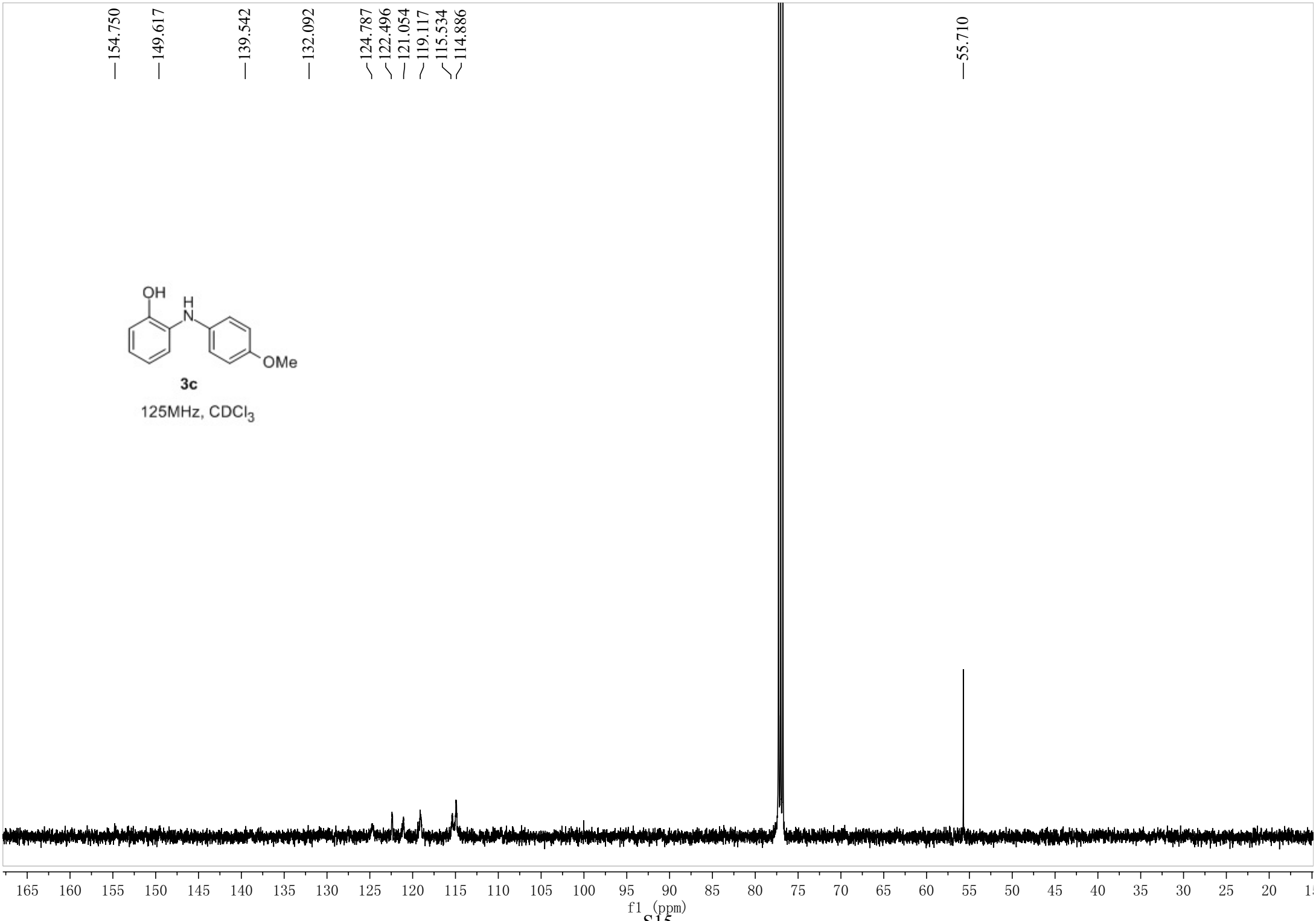
f1 (ppm)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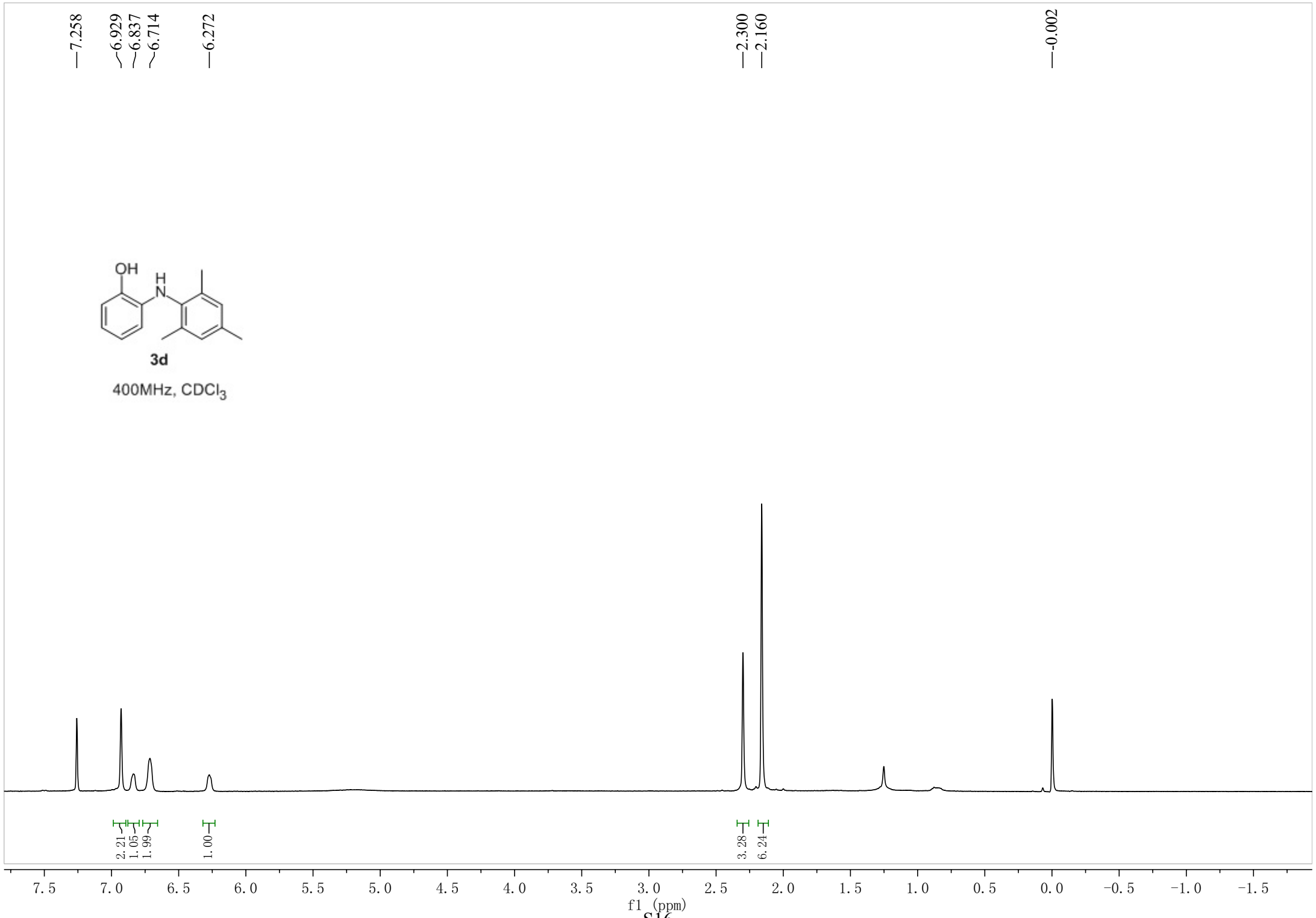
f1 (ppm)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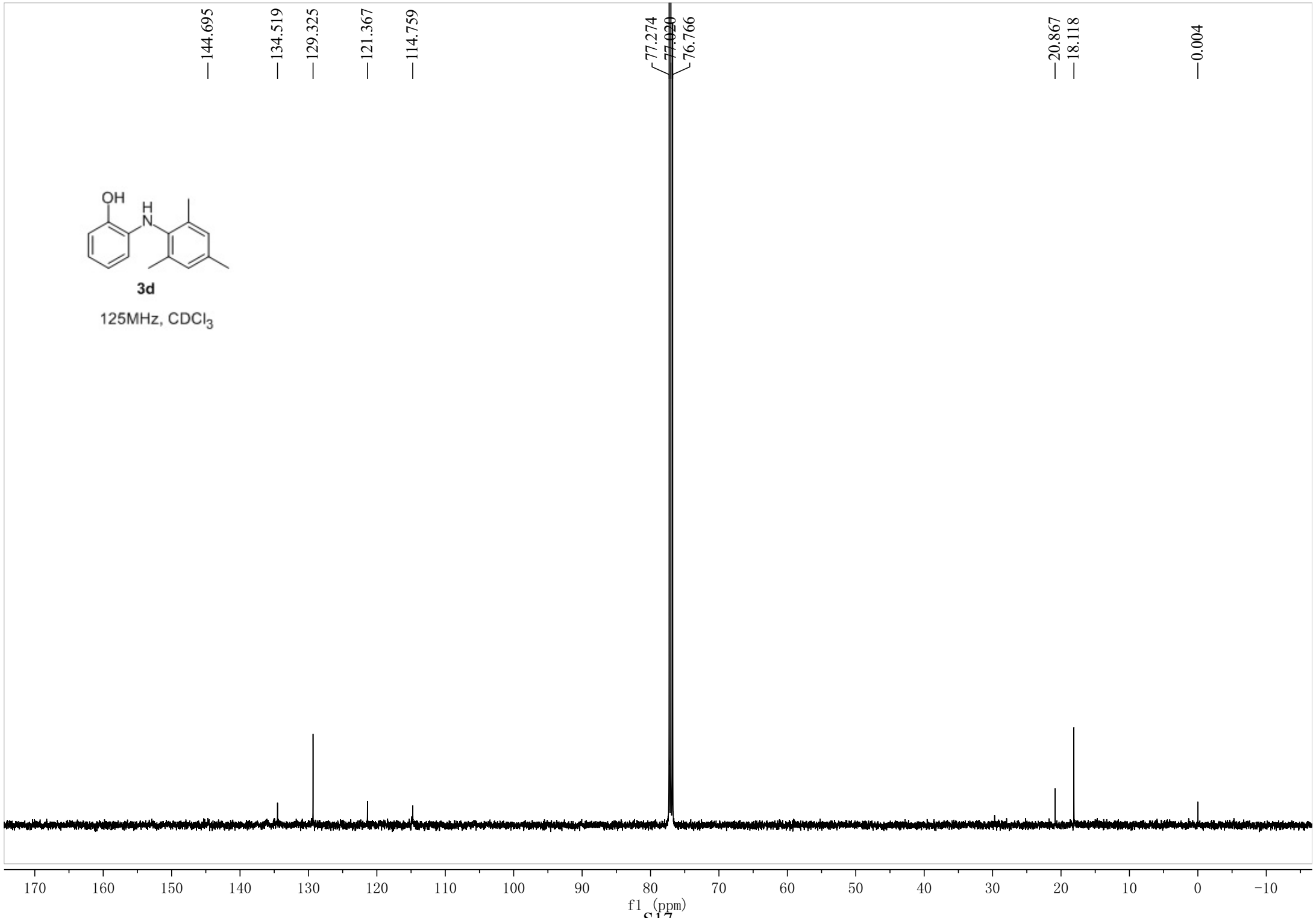
f1 (ppm)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N
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125MHz, CDCl3
170
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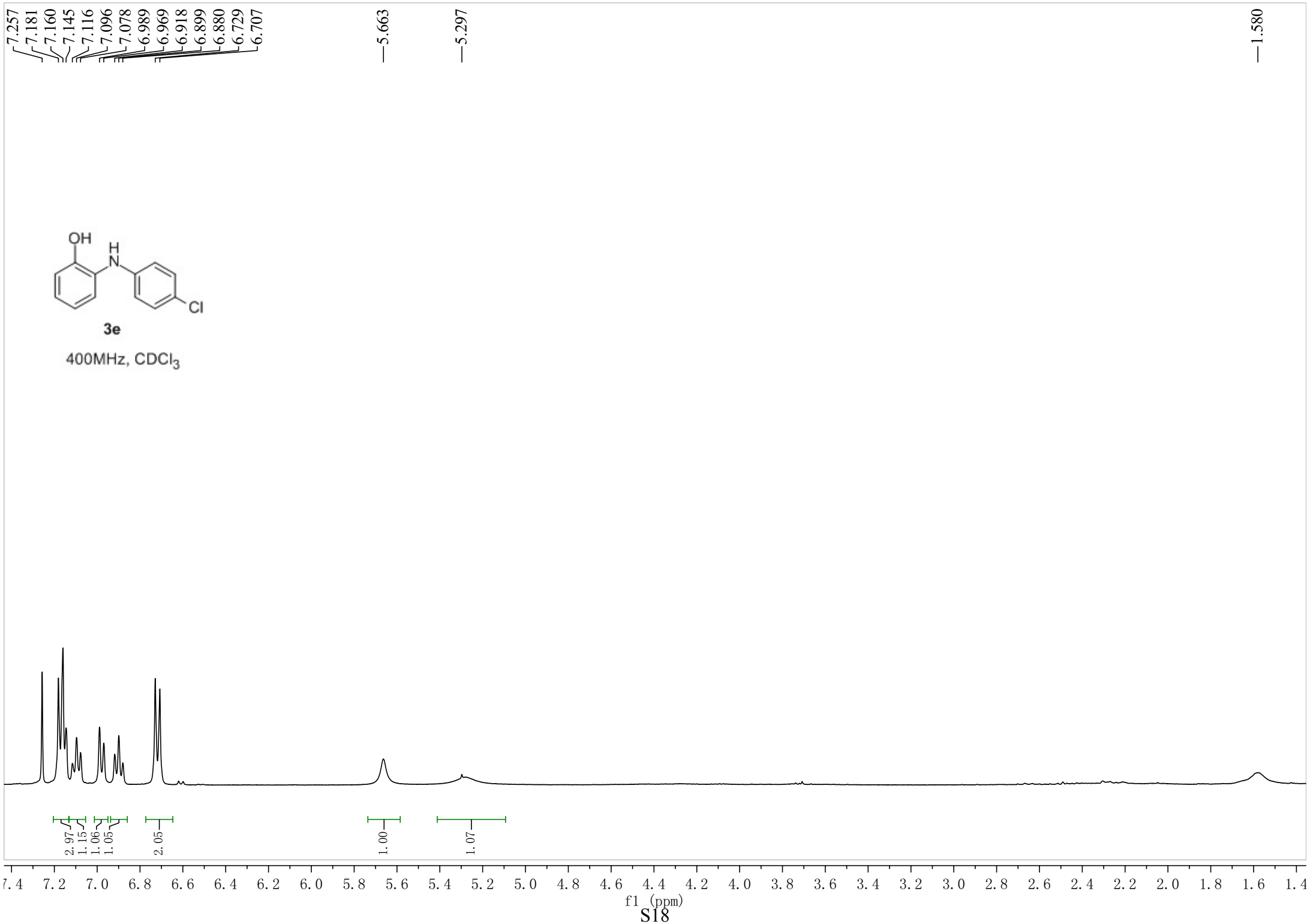
f1 (ppm)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2.2
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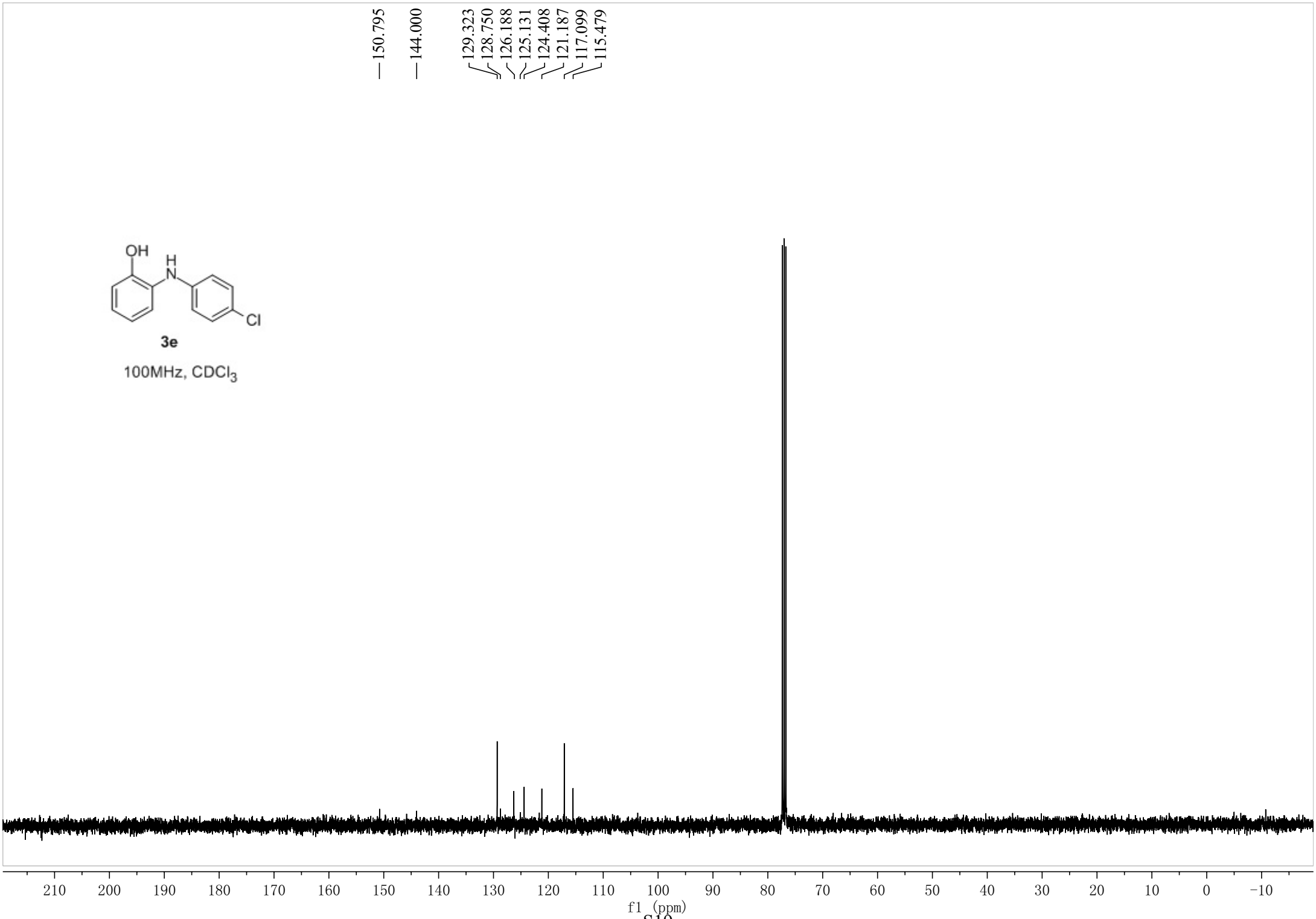
1.6
1.4
f1 (ppm)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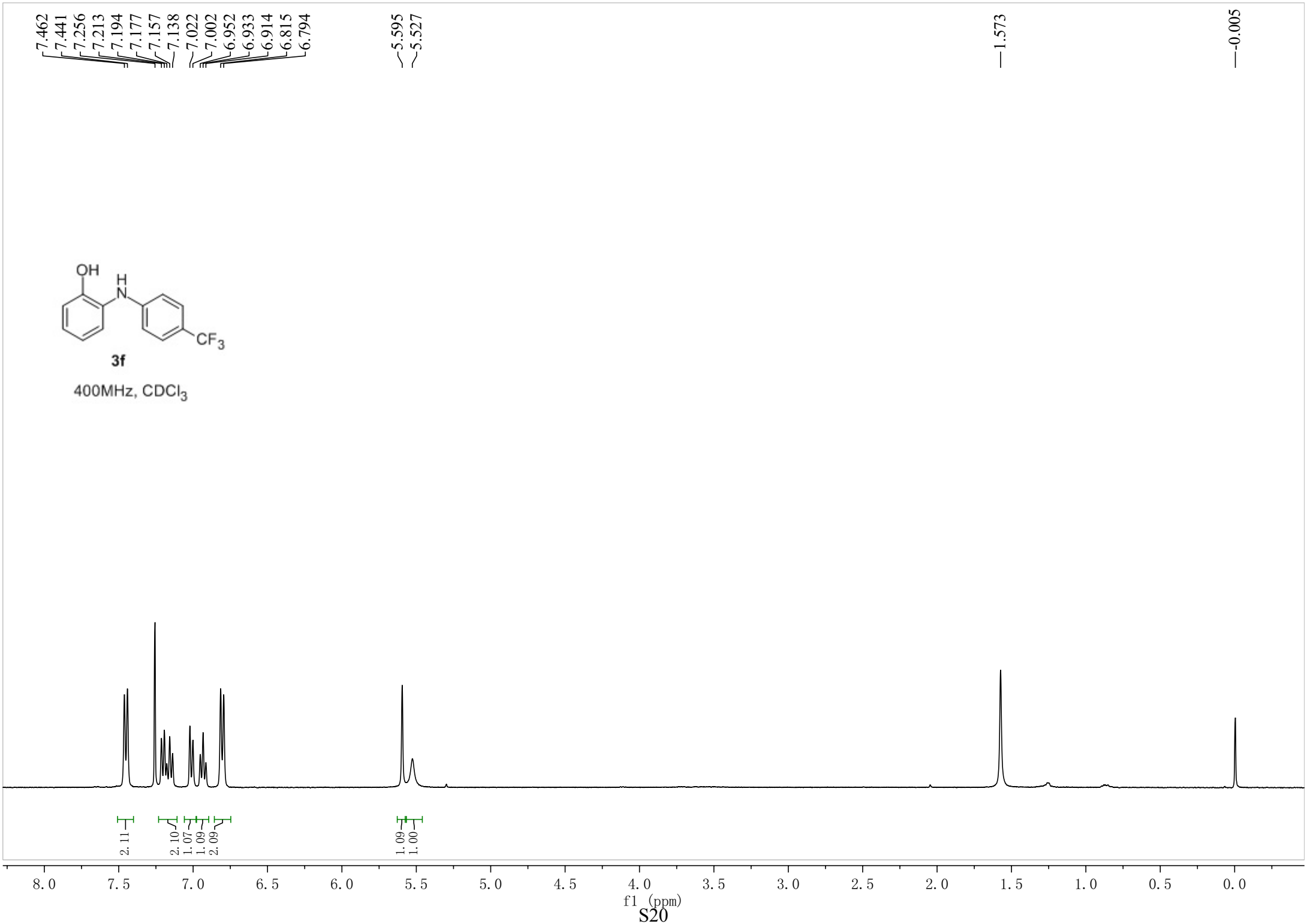
144.000
129.323
128.750
126.188
125.131
124.408
121.187
117.099
115.479
OH
H
N
Cl
3e
100MHz, CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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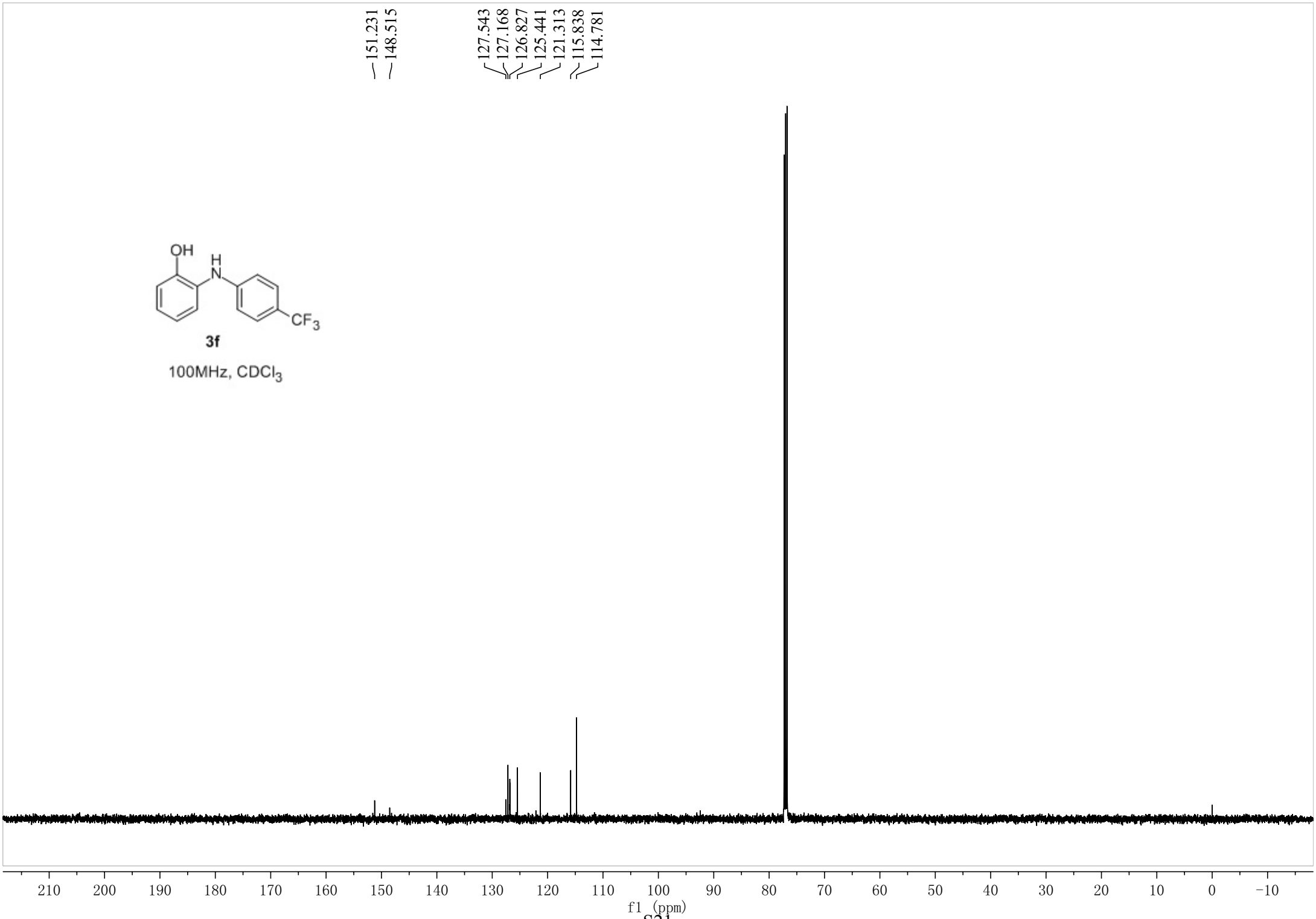
7.441
7.256
7.213
7.194
7.177
7.157
7.138
7.022
7.002
6.952
6.933
6.914
6.815
6.794
5.595
5.527
1.573
0.005
OH
H
N
CF3
3f
400MHz, CDCl3
2.11
2.10
1.07
1.09
2.09
1.09
1.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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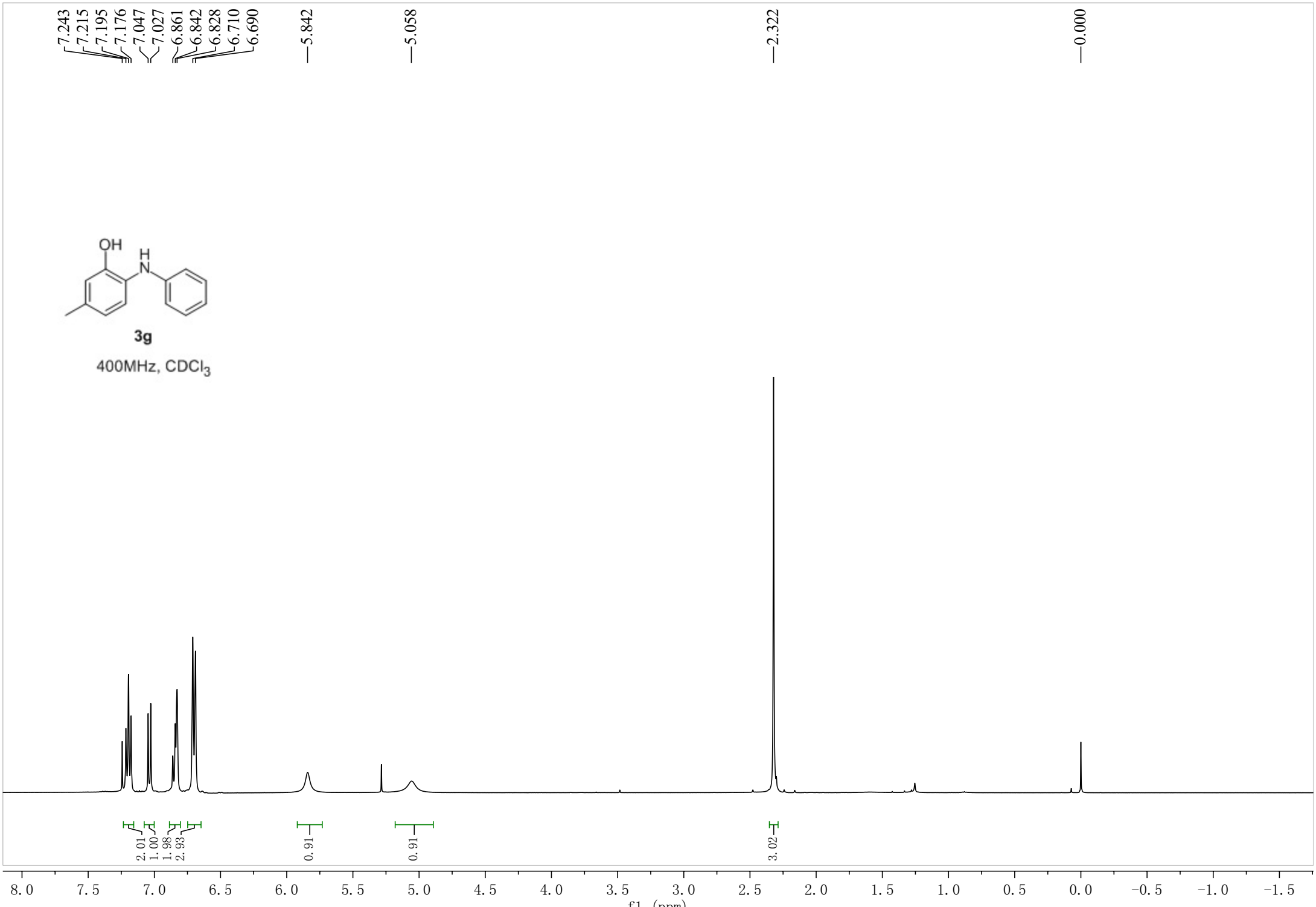
148.515
127.543
127.168
126.827
125.441
121.313
115.838
114.781
OH
H
N
CF3
3f
100MHz, CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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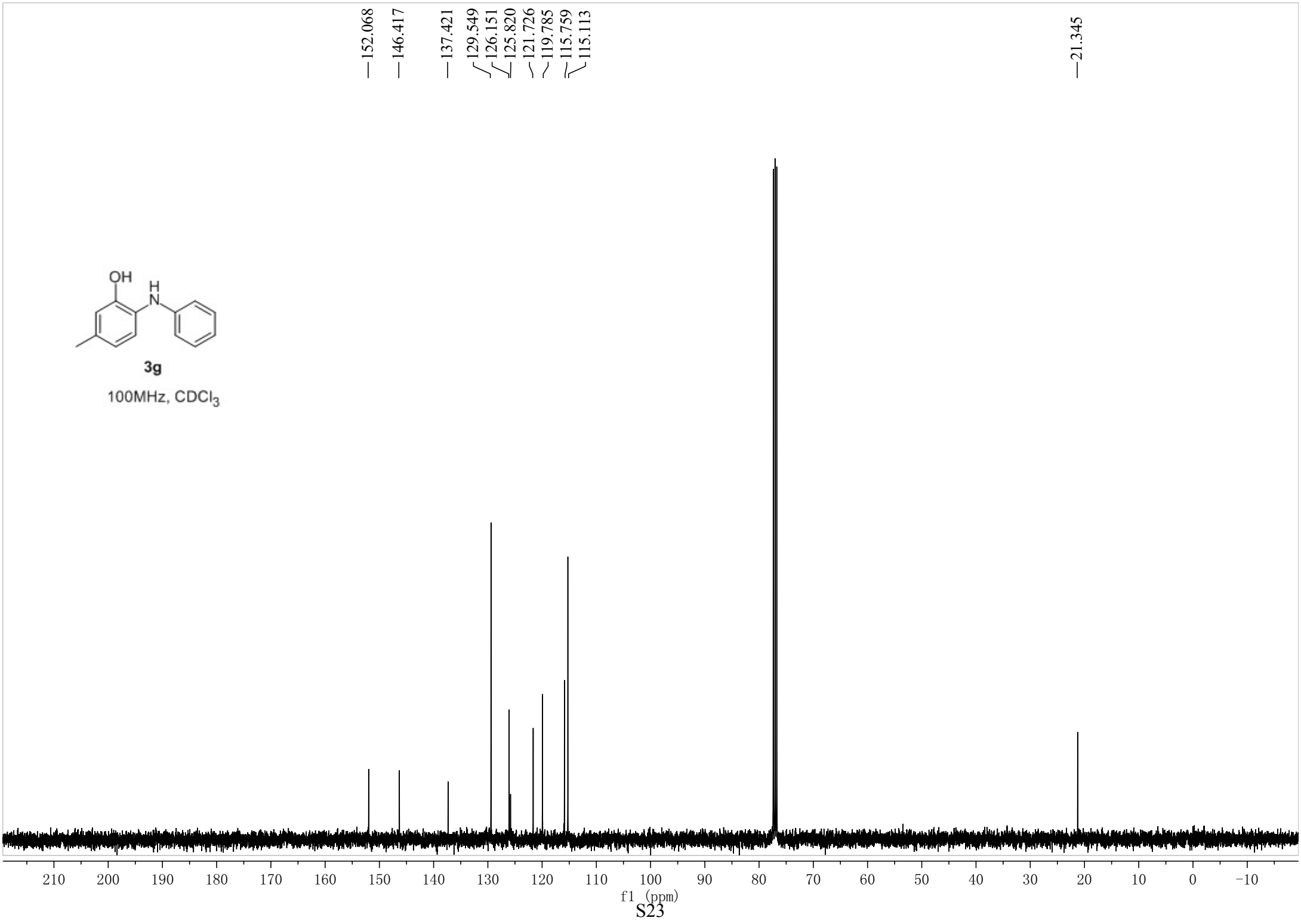
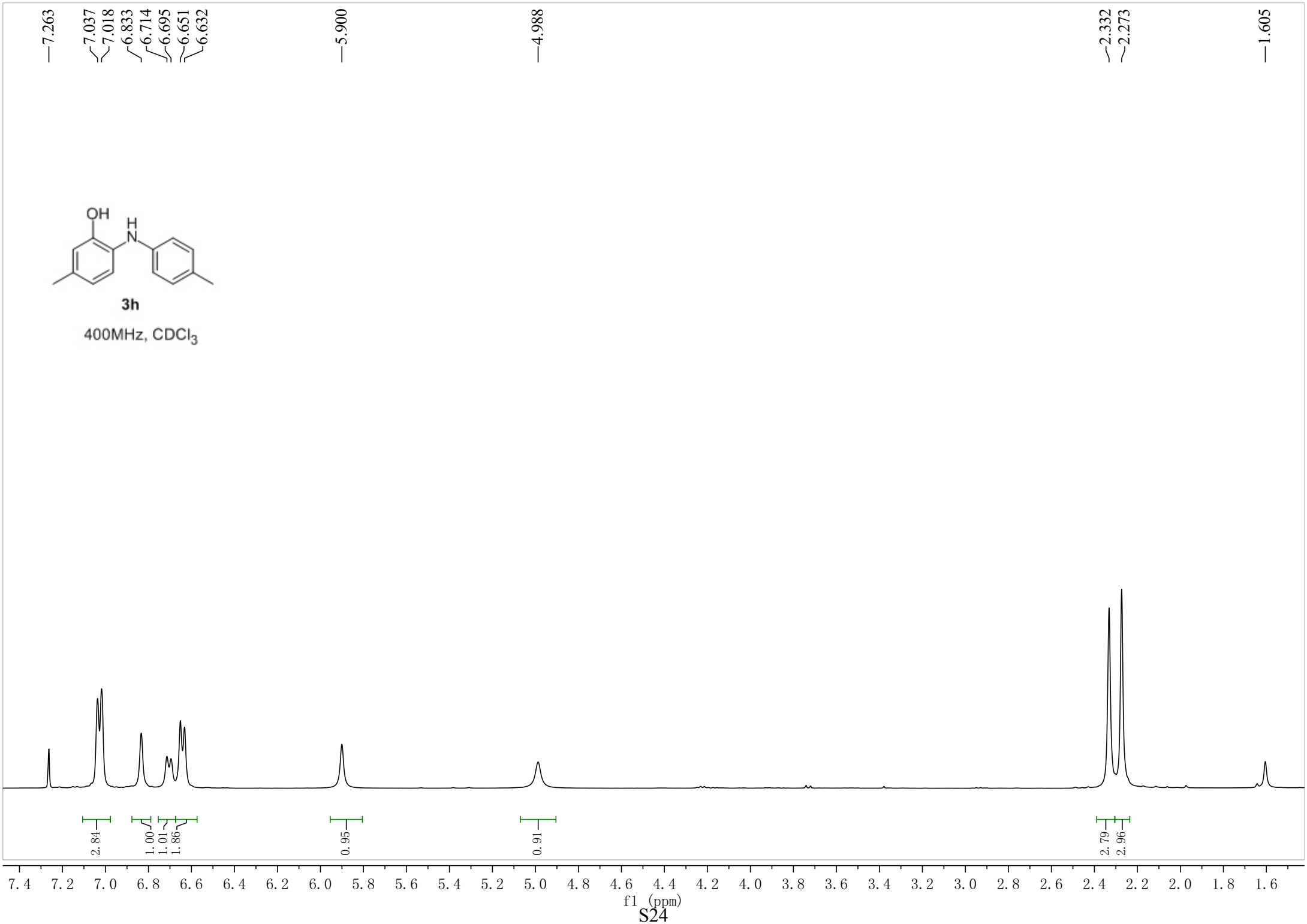
7.215
7.195
7.176
7.047
7.027
6.861
6.842
6.828
6.710
6.690
5.842
5.058
2.322
0.000
OH
H
N
3g
400MHz, CDCl3
2.01
1.00
1.98
2.93
0.91
0.91
3.02
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
-1.5
f1 (ppm)


152.068
146.417
137.421
129.549
126.151
125.820
121.726
119.785
115.759
115.113
21.345
OH
H
N
3g
100MHz, CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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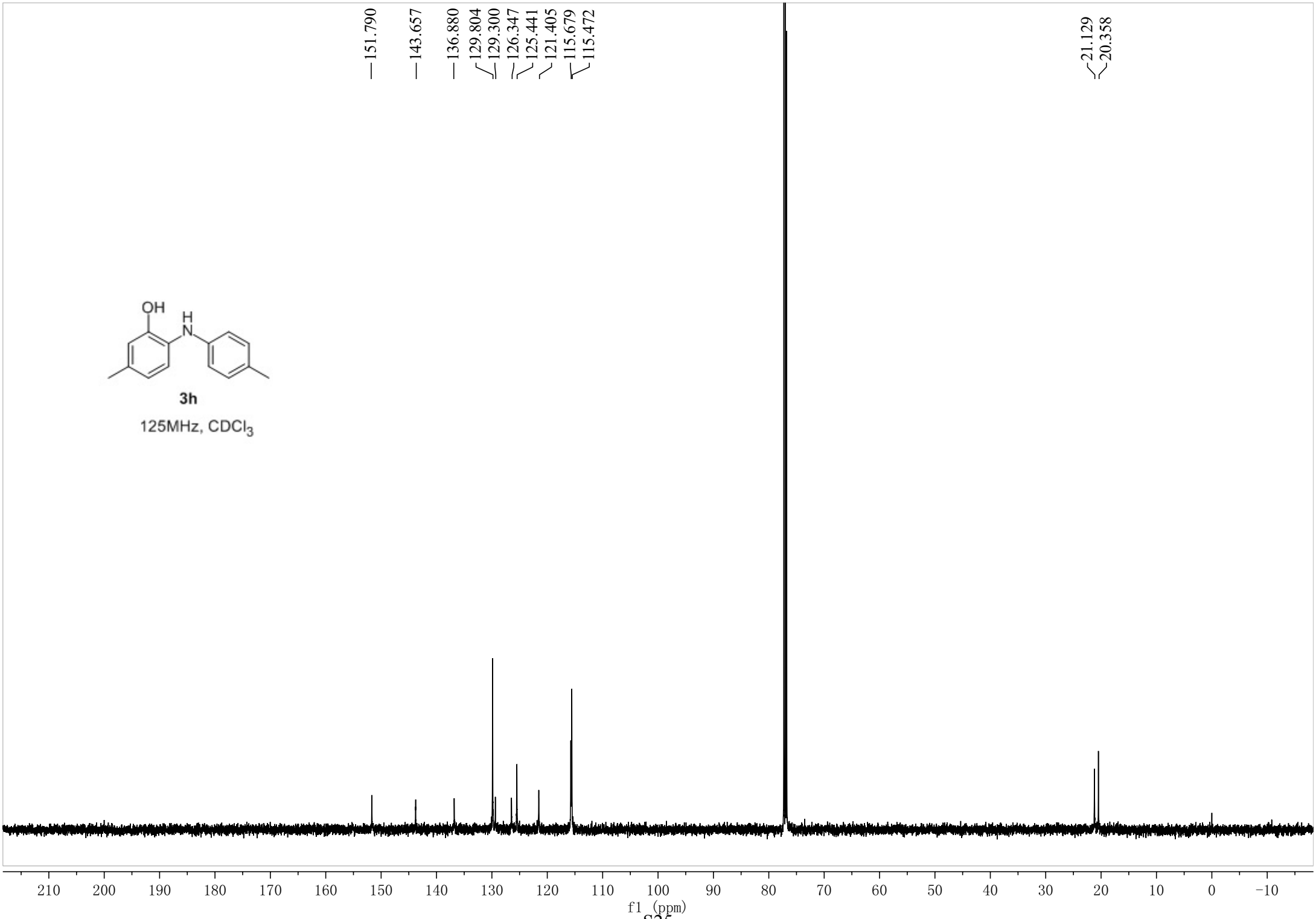
7.037
7.018
6.833
6.714
6.695
6.651
6.632
5.900
4.988
2.332
2.273
1.605
OH
H
N
3h
400MHz, CDCl3
2.84
1.00
1.01
1.86
0.95
0.91
2.79
2.96
7.4 7.2 7.0 6.8 6.6 6.4 6.2 6.0 5.8 5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2 4.0 3.8 3.6 3.4 3.2 3.0 2.8 2.6 2.4 2.2 2.0 1.8 1.6
f1 (ppm)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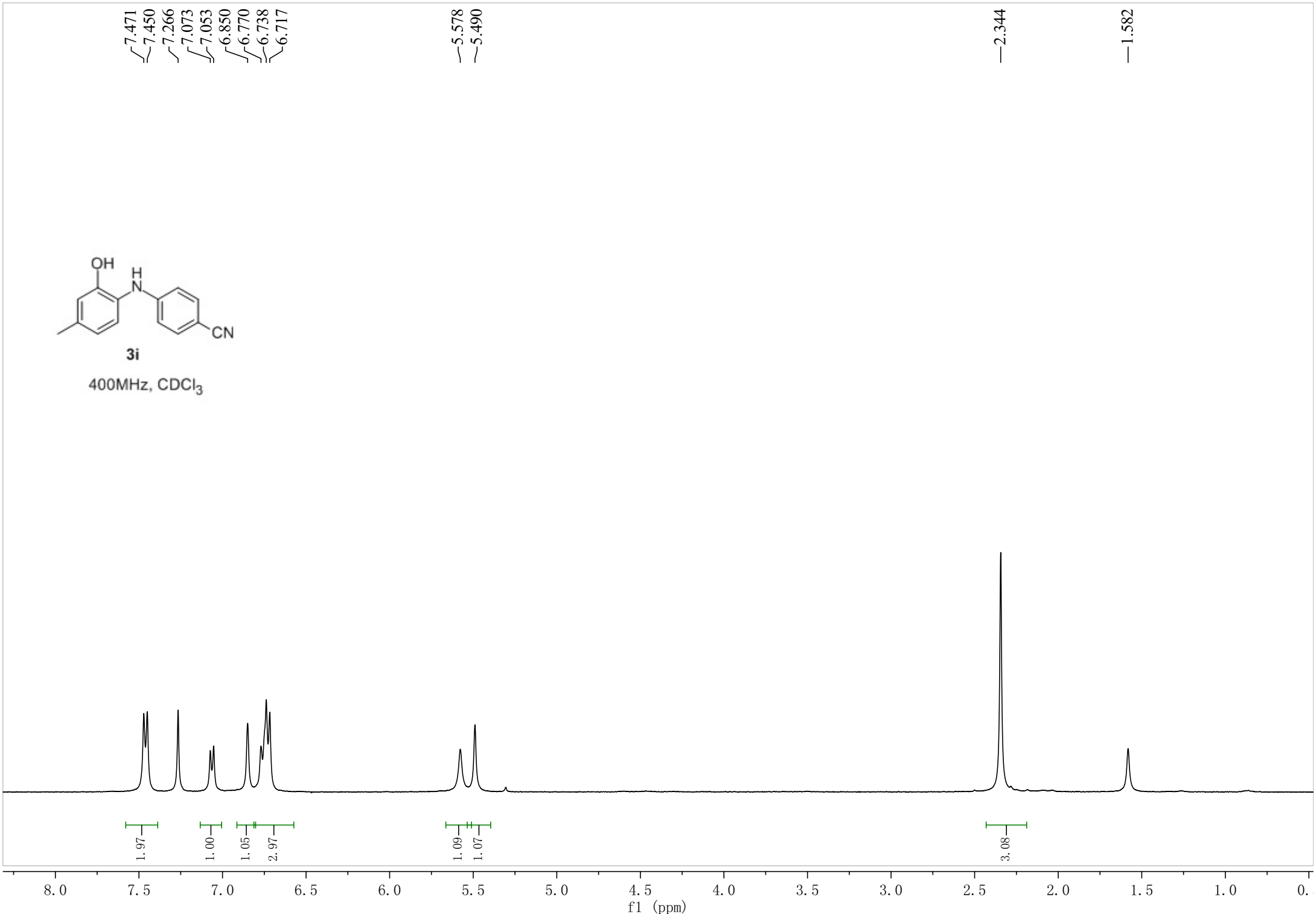
143.657
136.880
129.804
129.300
126.347
125.441
121.405
115.679
115.472
21.129
20.358
OH
H
N
3h
125MHz, CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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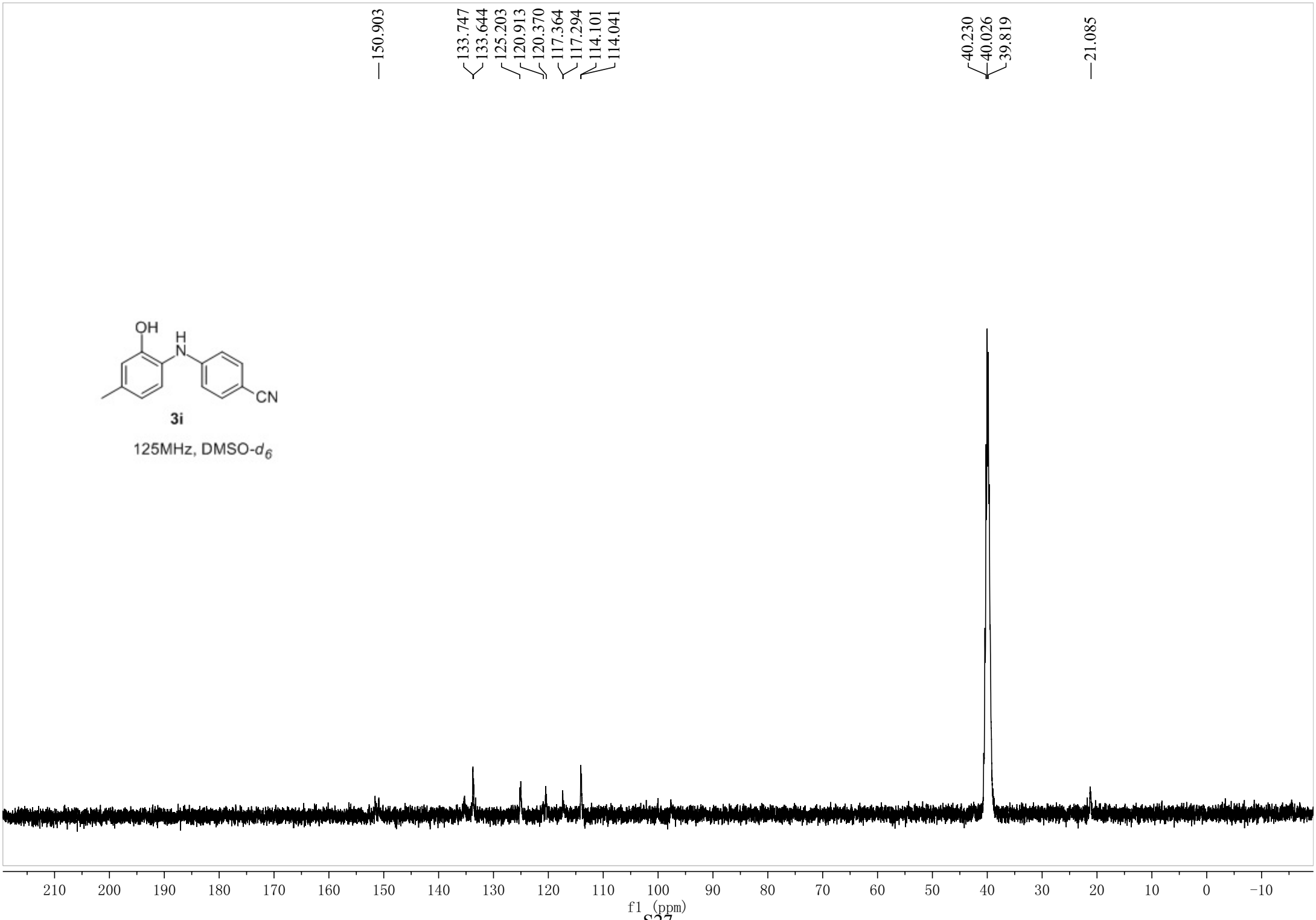
7.450
7.266
7.073
7.053
6.850
6.770
6.738
6.717
5.578
5.490
2.344
1.582
OH
H
N
CN
3i
400MHz, CDCl3
1.97
1.00
1.05
2.97
1.09
1.07
3.08
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.
f1 (ppm)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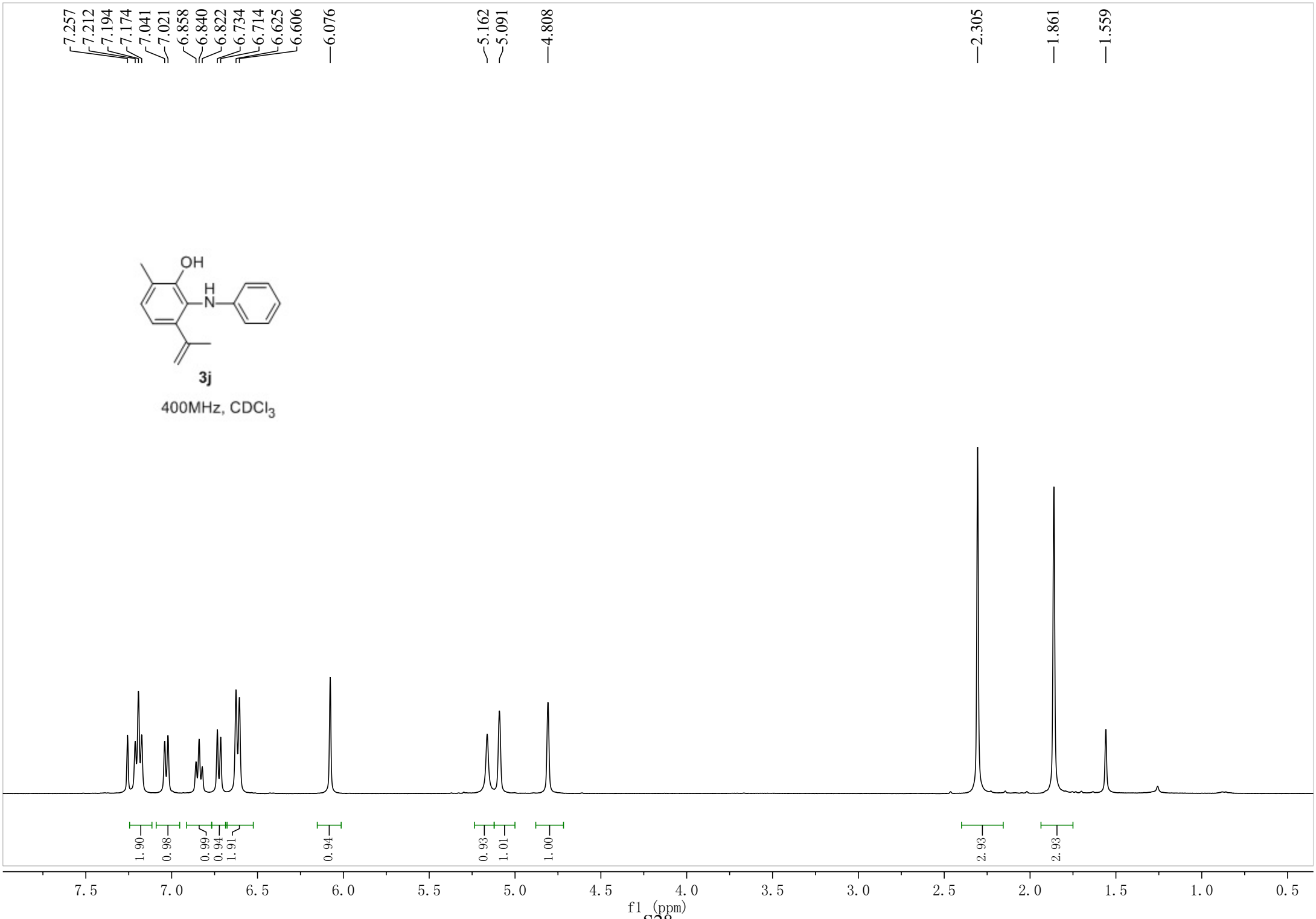
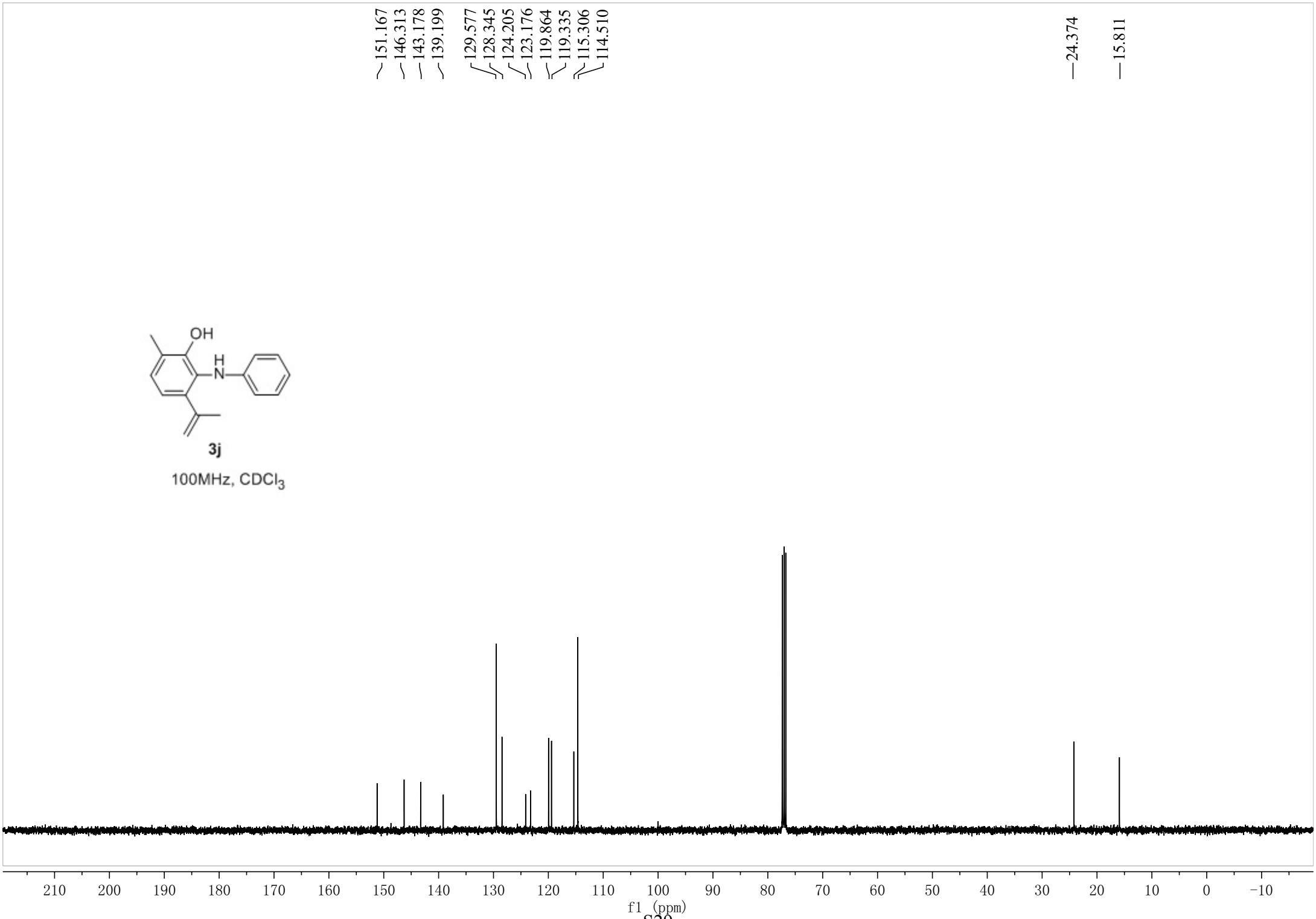
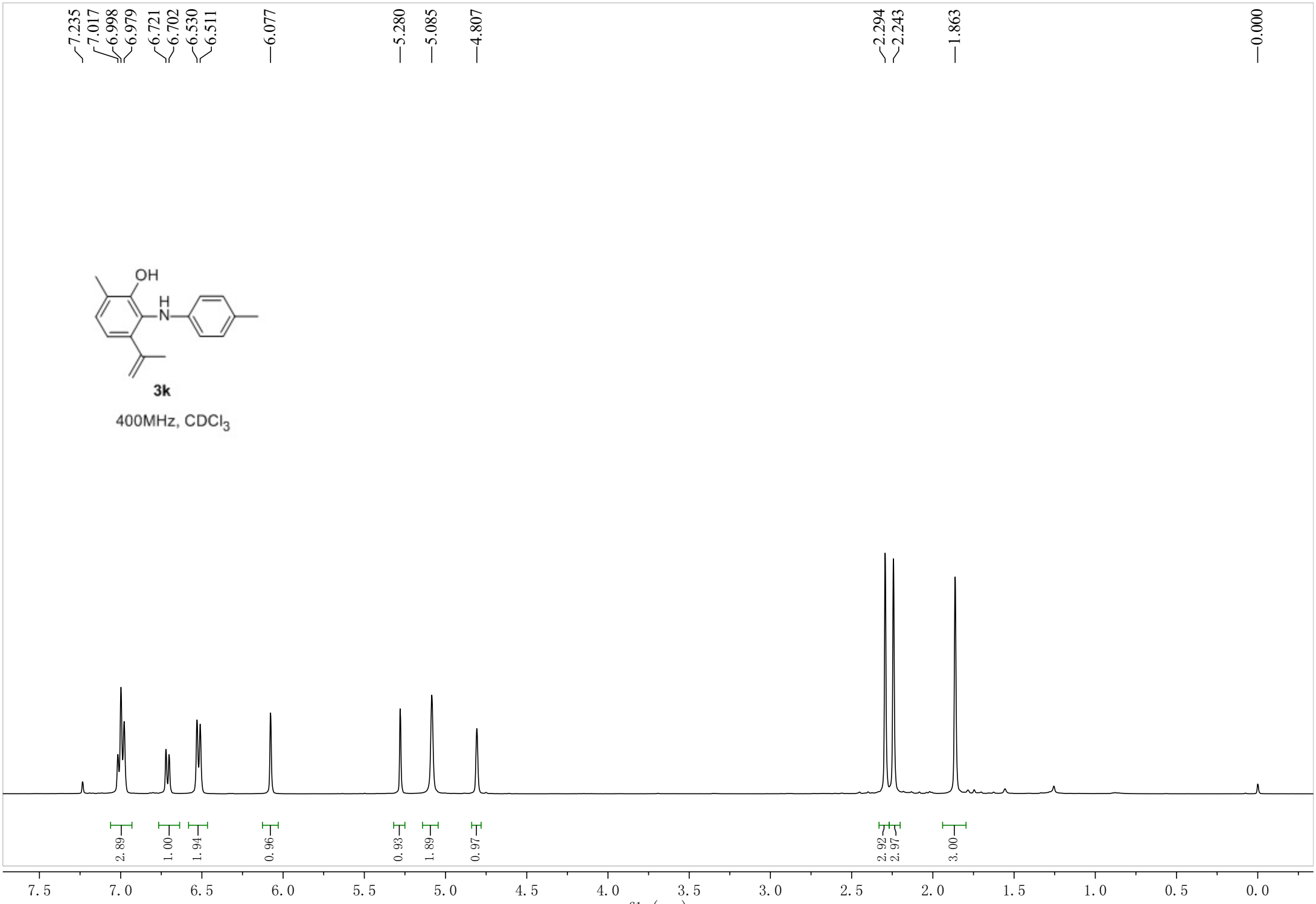
133.747
133.644
125.203
120.913
120.370
117.364
117.294
114.101
114.041
40.230
40.026
39.819
21.085
OH
H
N
CN
3i
125MHz, DMSO-d_6
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.257
7.212
7.194
7.174
7.041
7.021
6.858
6.840
6.822
6.734
6.714
6.625
6.606
6.076
5.162
5.091
4.808
2.305
1.861
1.559
OH
H
N
3j
400MHz, CDCl3
1.90
0.98
0.99
0.94
1.91
0.94
0.93
1.01
1.00
2.93
2.93
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


151.167
146.313
143.178
139.199
129.577
128.345
124.205
123.176
119.864
119.335
115.306
114.510
24.374
15.811
OH
H
N
3j
100MHz, CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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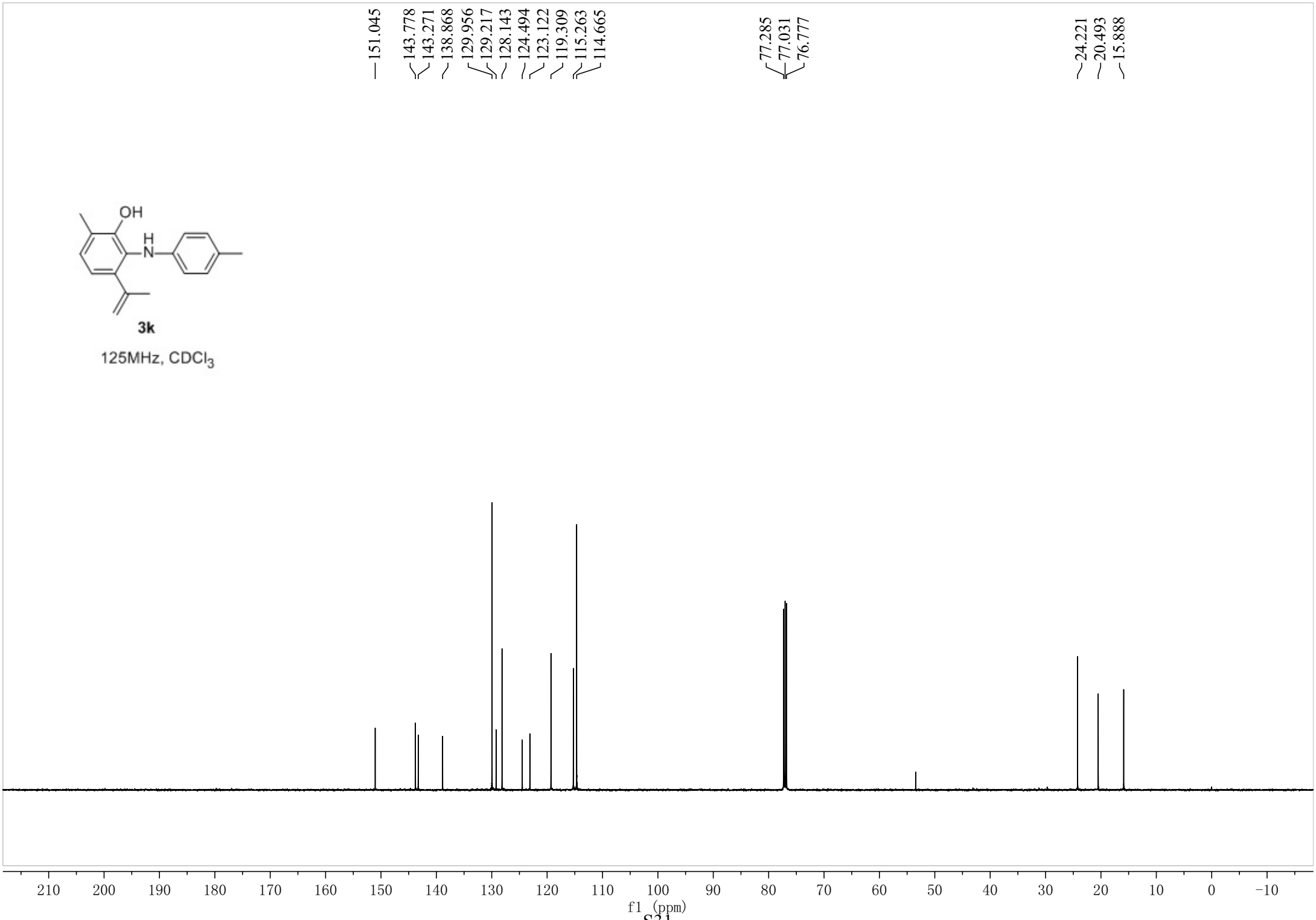
6.998
6.979
6.721
6.702
6.530
6.511
6.077
5.280
5.085
4.807
2.294
2.243
1.863
0.000
OH
H
N
3k
400MHz, CDCl3
2.89
1.00
1.94
0.96
0.93
1.89
0.97
2.92
2.97
3.00
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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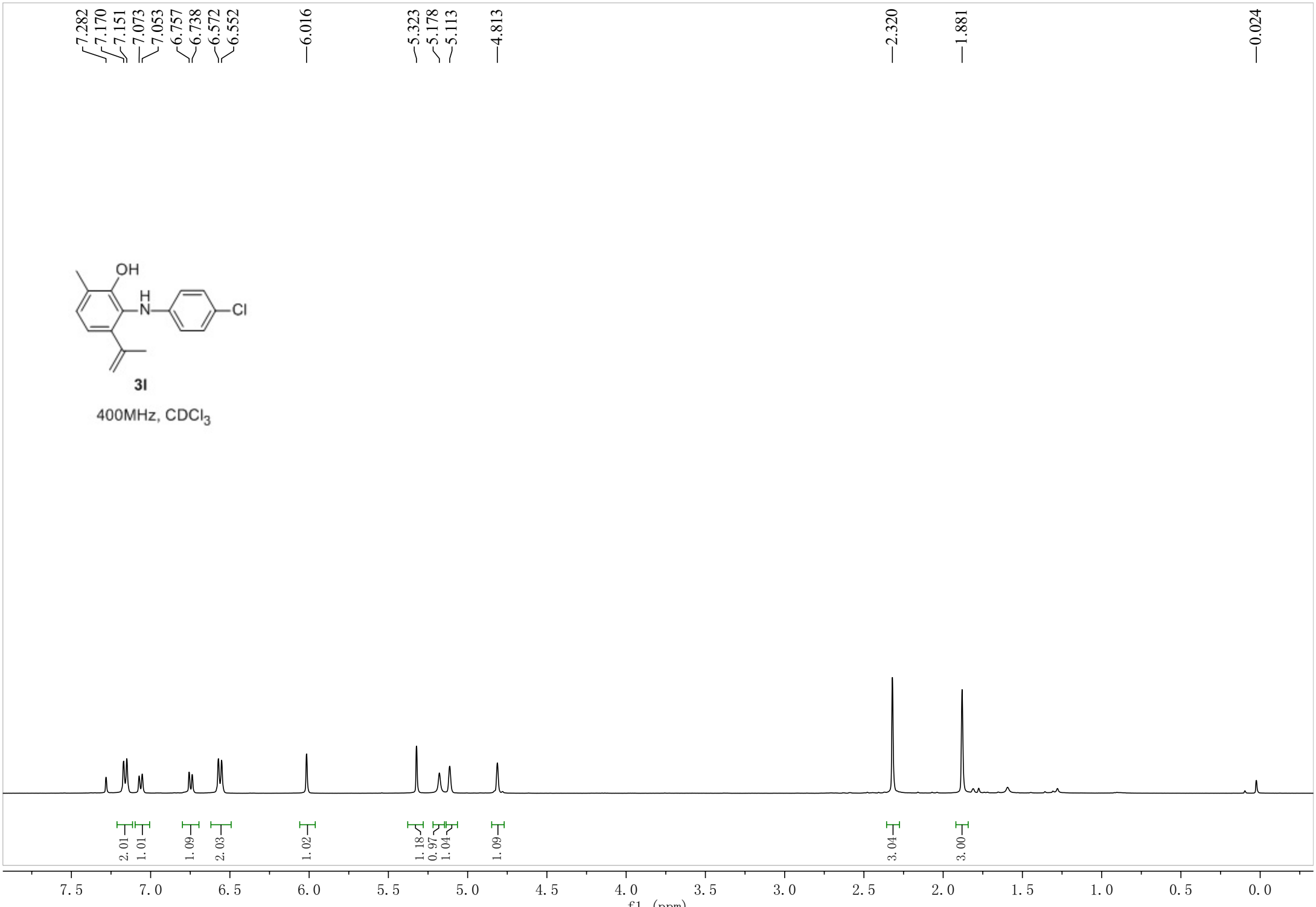
143.271
138.868
129.956
129.217
128.143
124.494
123.122
119.309
115.263
114.665
77.285
77.031
76.777
24.221
20.493
15.888
OH
H
N
3k
125MHz, CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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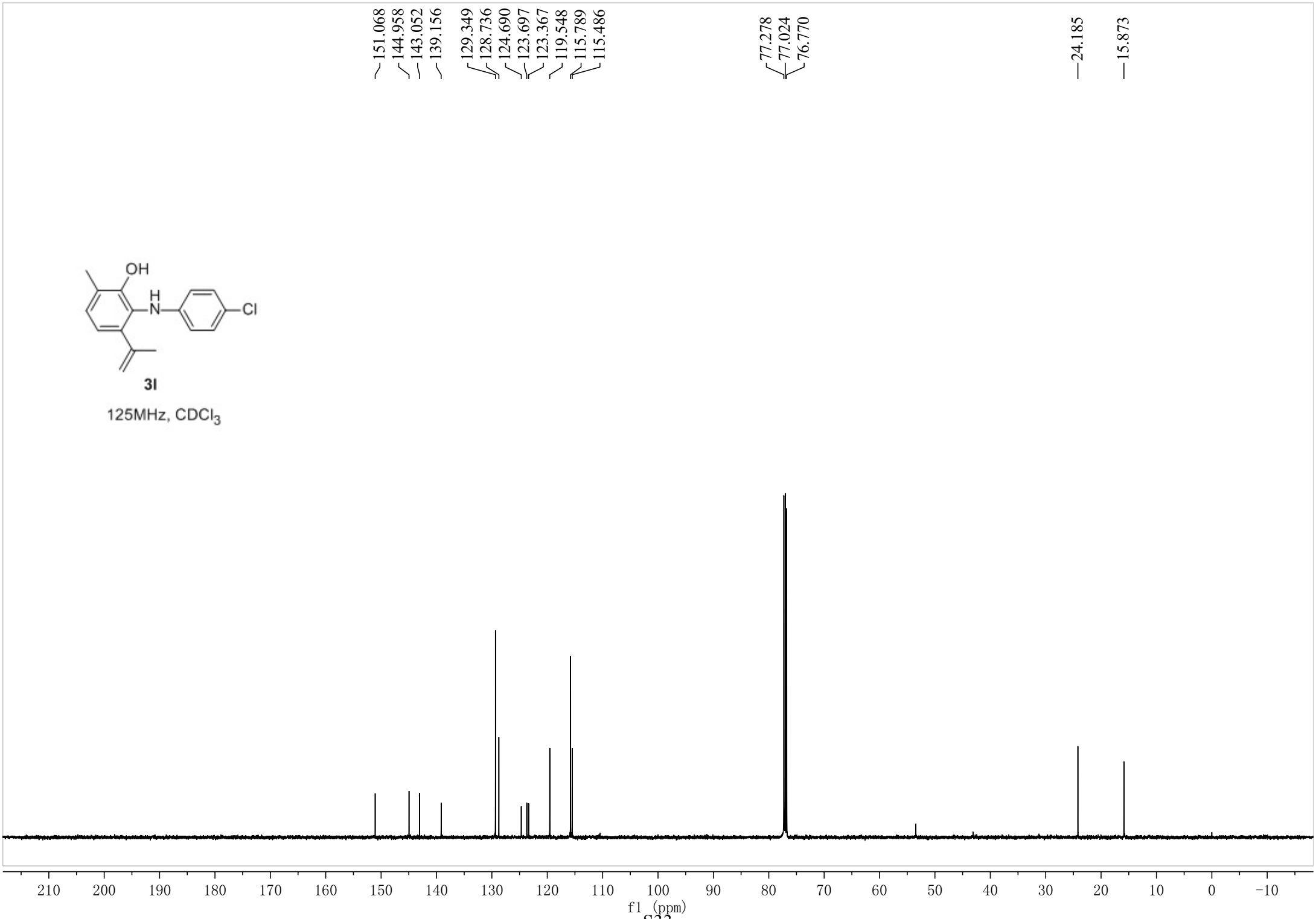
7.170
7.151
7.073
7.053
6.757
6.738
6.572
6.552
6.016
5.323
5.178
5.113
4.813
2.320
1.881
0.024
OH
H
N
Cl
3l
400MHz, CDCl3
2.01
1.01
1.09
2.03
1.02
1.18
0.97
1.04
1.09
3.04
3.00
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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128.736
124.690
123.697
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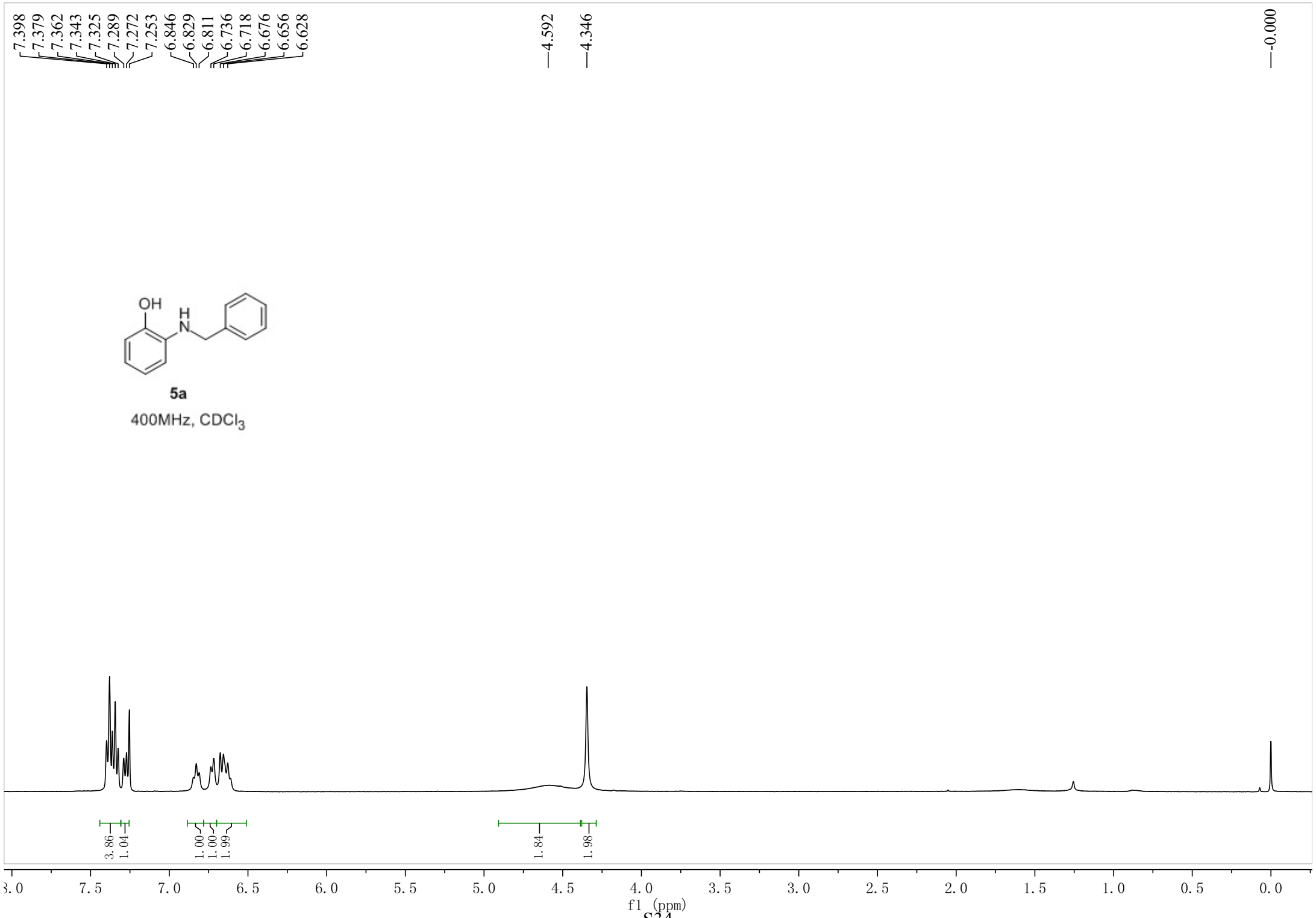
119.548
115.789
115.486
77.278
77.024
76.770
24.185
15.873
OH
H
N
Cl
3l
125MHz, CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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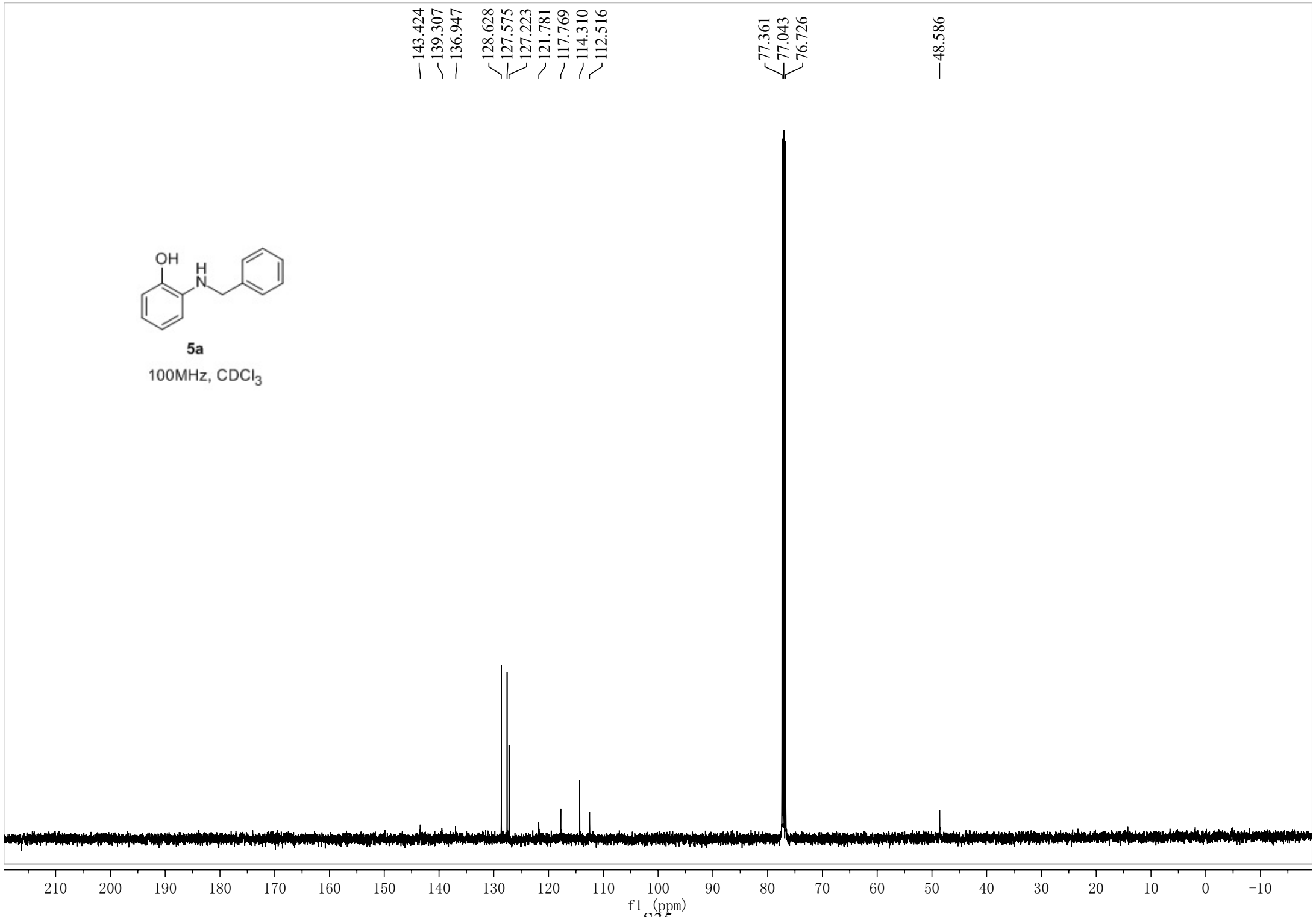
7.325
7.289
7.272
7.253
6.846
6.829
6.811
6.736
6.718
6.676
6.656
6.628
4.592
4.346
0.000
OH
H
N
5a
400MHz, CDCl3
3.86
1.04
1.00
1.00
1.99
1.84
1.98
f1 (ppm)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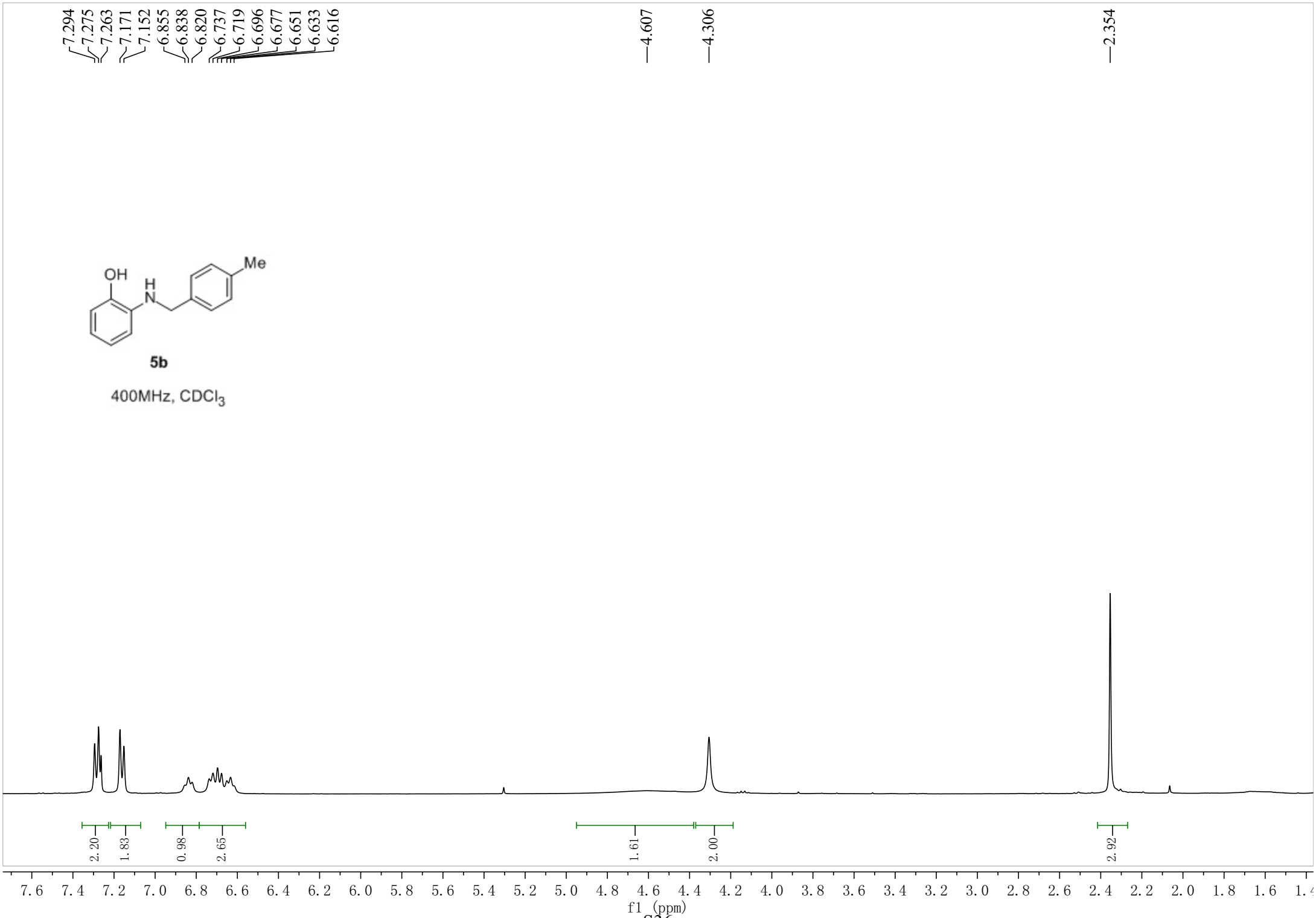
136.947
128.628
127.575
127.223
121.781
117.769
114.310
112.516
77.361
77.043
76.726
48.586
OH
H
N
5a
100MHz, CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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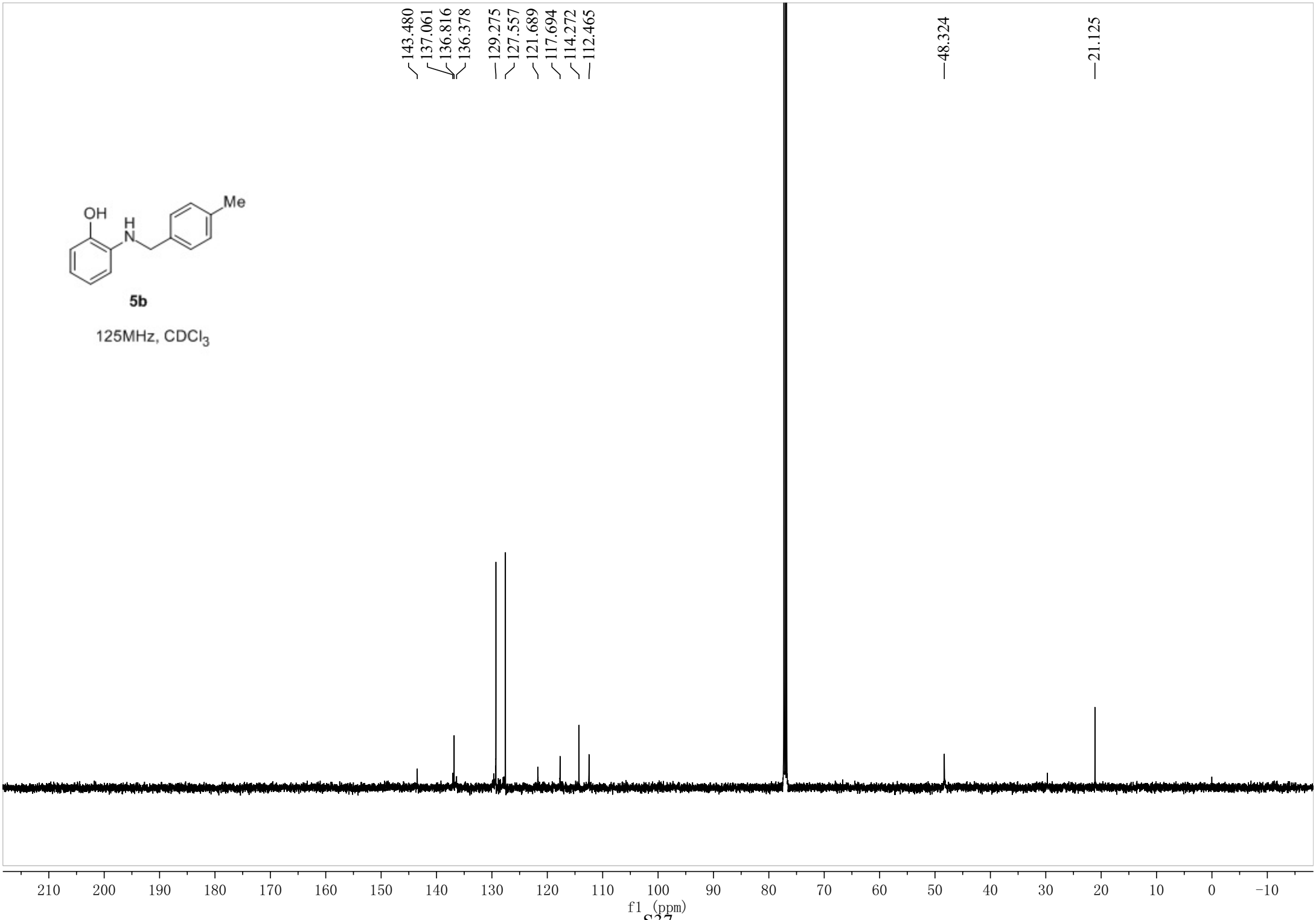
6.855
6.838
6.820
6.737
6.719
6.696
6.677
6.651
6.633
6.616
4.607
4.306
2.354
OH
H
N
Me
5b
400MHz, CDCl3
2.20
1.83
0.98
2.65
1.61
2.00
2.92
7.6 7.4 7.2 7.0 6.8 6.6 6.4 6.2 6.0 5.8 5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2 4.0 3.8 3.6 3.4 3.2 3.0 2.8 2.6 2.4 2.2 2.0 1.8 1.6
f1 (ppm)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136.378
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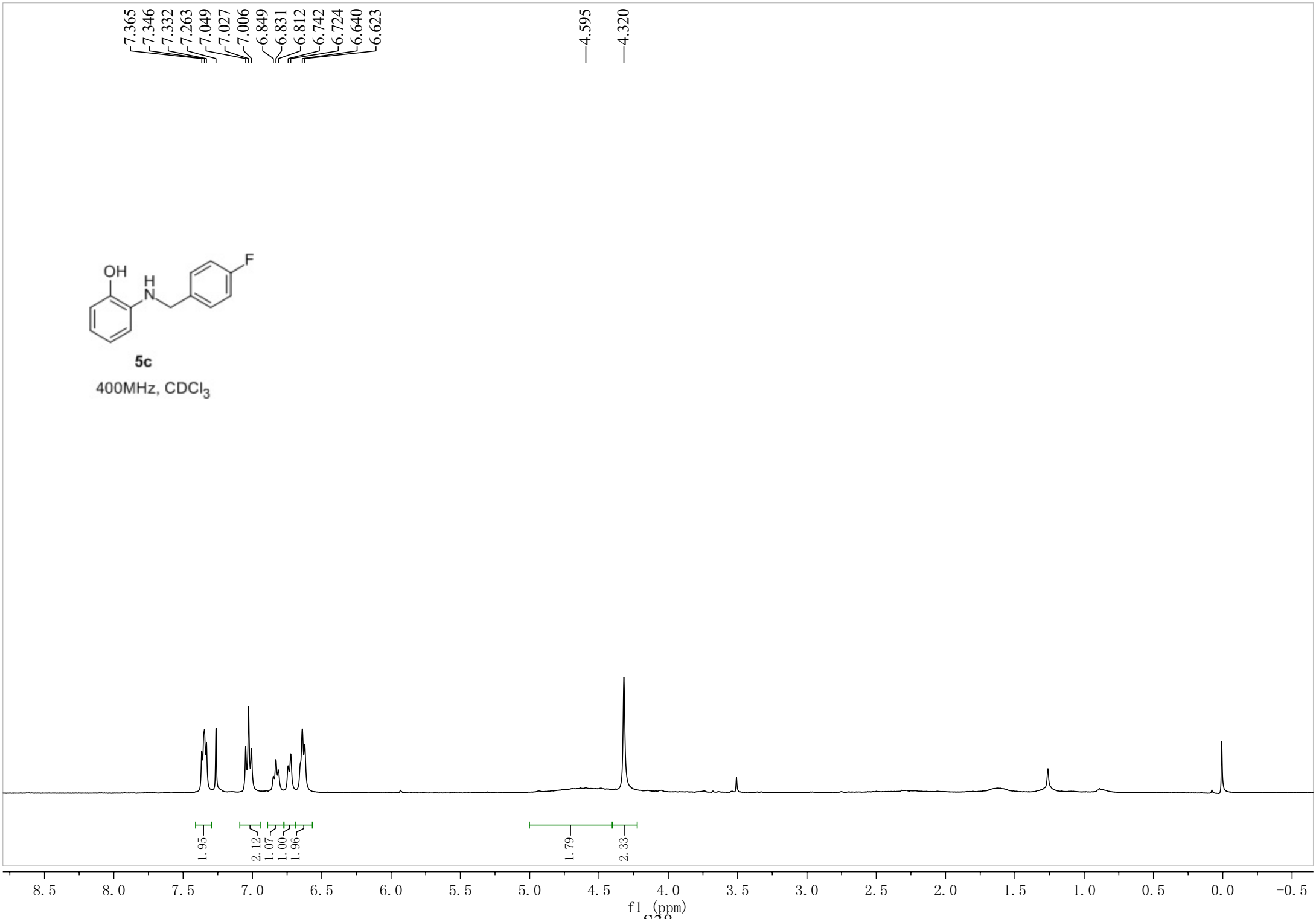
127.557
121.689
117.694
114.272
112.465
48.324
21.125
OH
H
N
Me
5b
125MHz, CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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7.027
7.006
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6.812
6.742
6.724
6.640
6.623
4.595
4.320
OH
H
N
F
5c
400MHz, CDCl3
1.95
2.12
1.07
1.00
1.96
1.79
2.33
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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3.0
2.5
2.0
1.5
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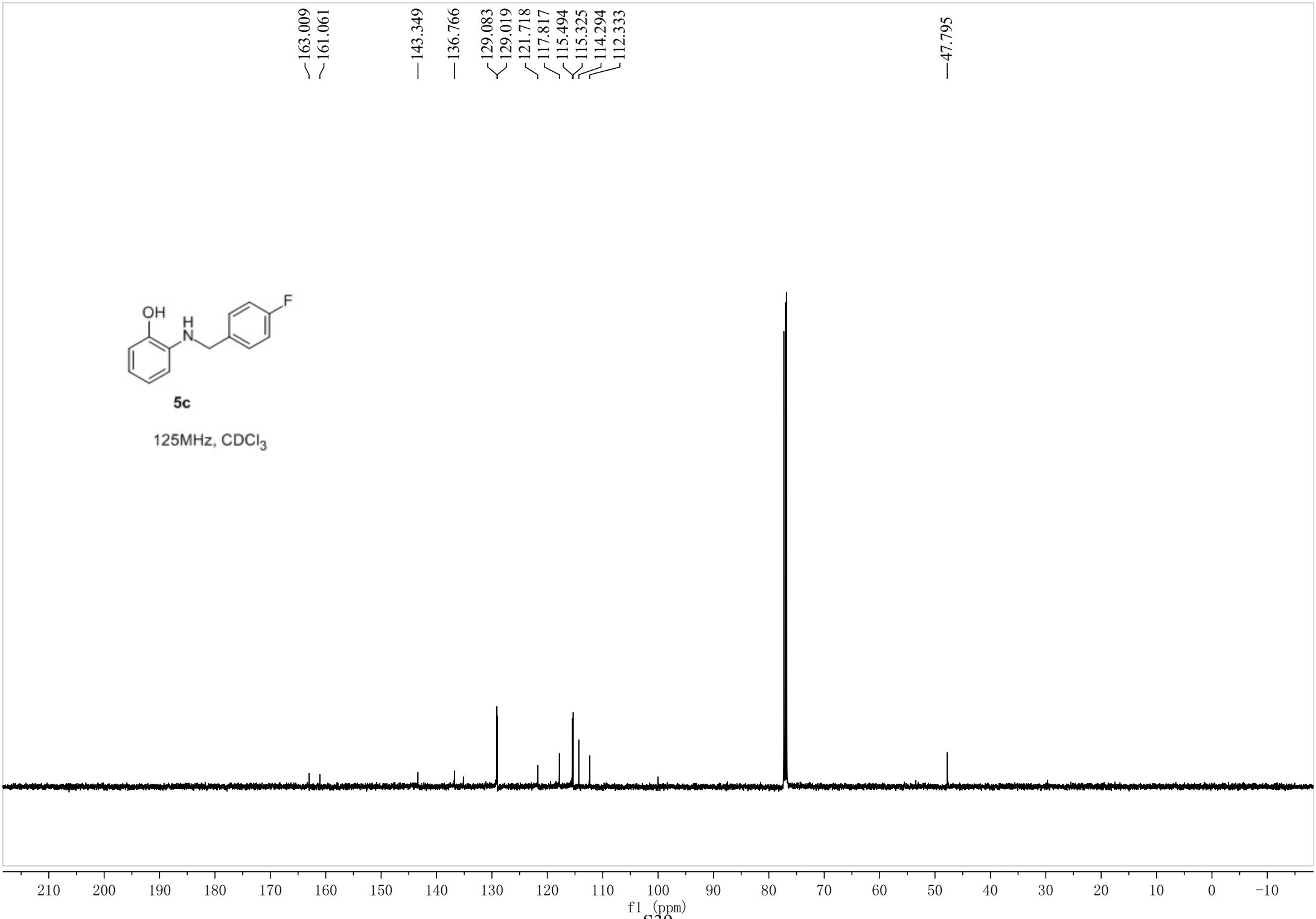
0.5
0.0
-0.5
f1 (ppm)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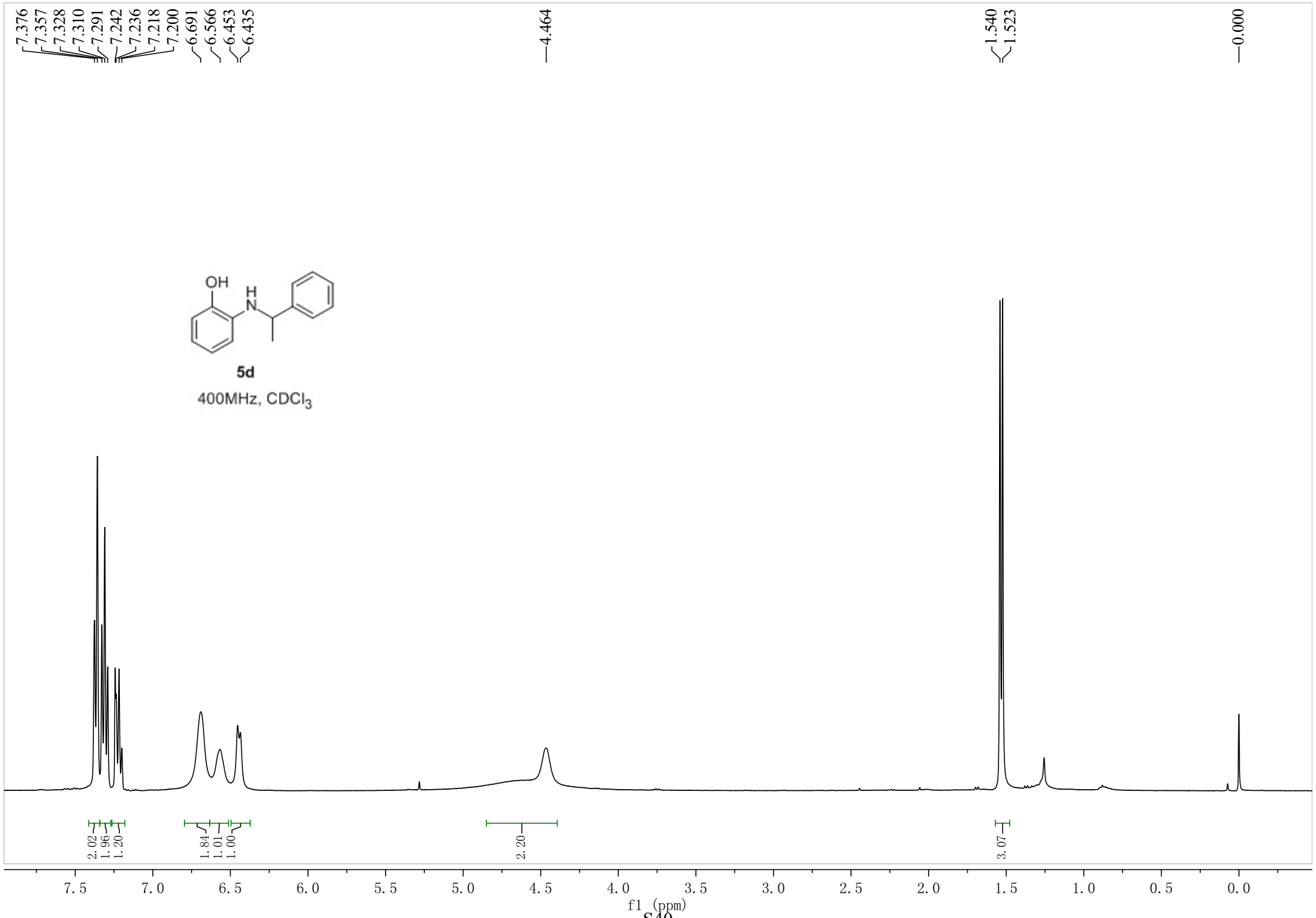
129.019
121.718
117.817
115.494
115.325
114.294
112.333
47.795
OH
H
N
F
5c
125MHz, CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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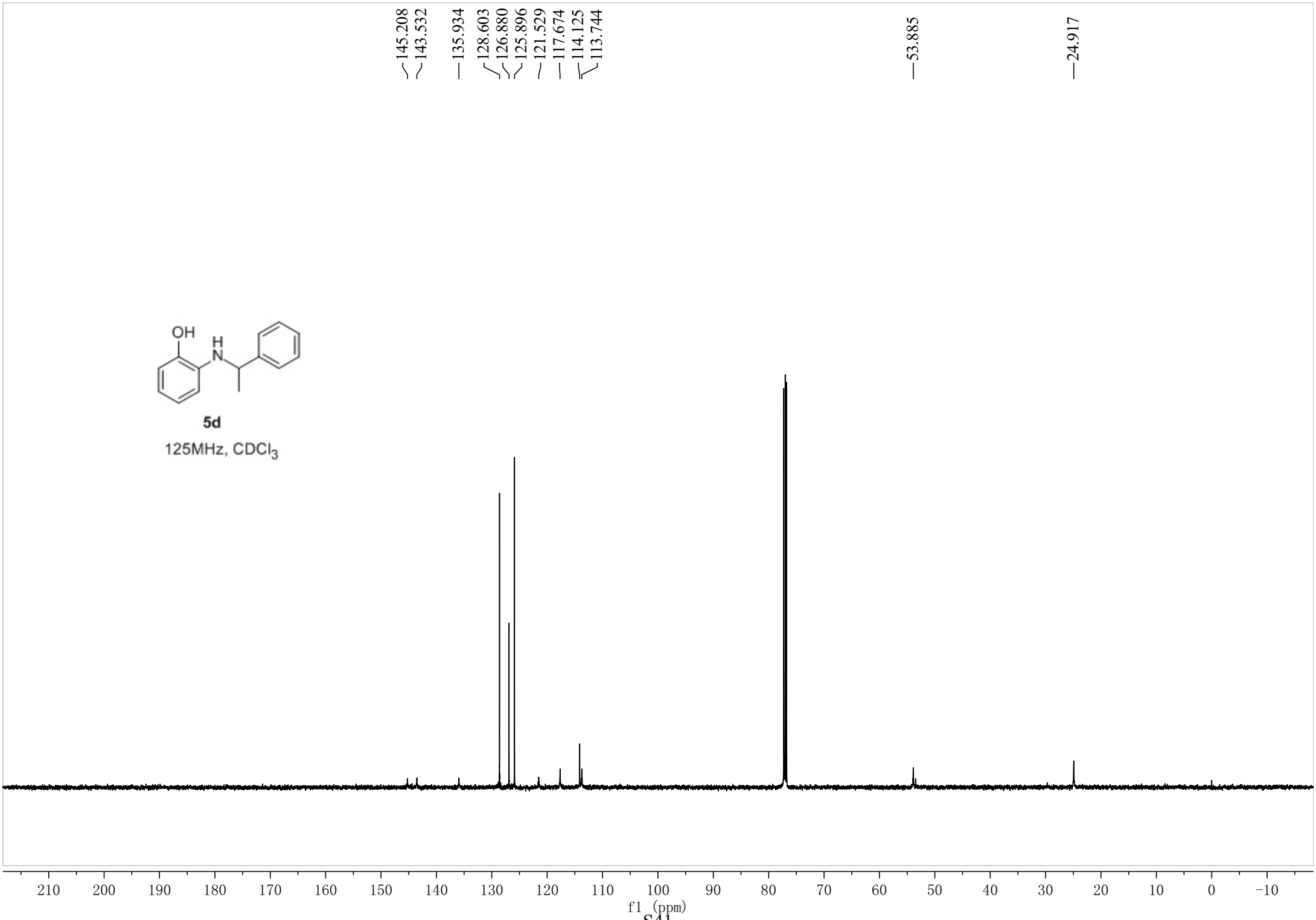
7.310
7.291
7.242
7.236
7.218
7.200
6.691
6.566
6.453
6.435
4.464
1.540
1.523
0.000
OH
H
N
5d
400MHz, CDCl3
2.02
1.96
1.20
1.84
1.01
1.00
2.20
3.07
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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121.529
117.674
114.125
113.744
53.885
24.917
OH
H
N
5d
125MHz, CDCl3
210
200
190
180
170
160
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140
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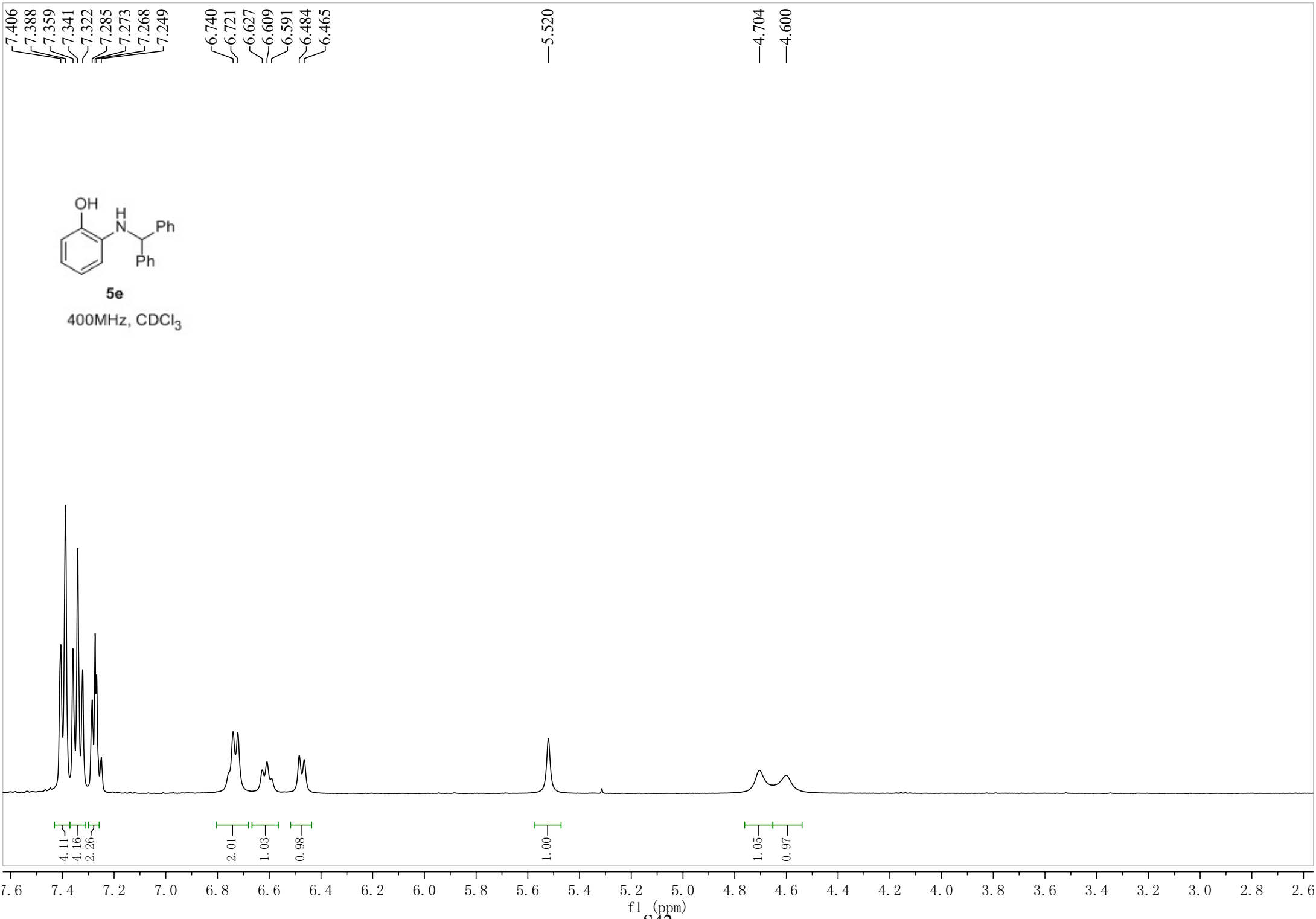
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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7.285
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7.268
7.249
6.740
6.721
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6.609
6.591
6.484
6.465
5.520
4.704
4.600
OH
H
N
Ph
Ph
5e
400MHz, CDCl3
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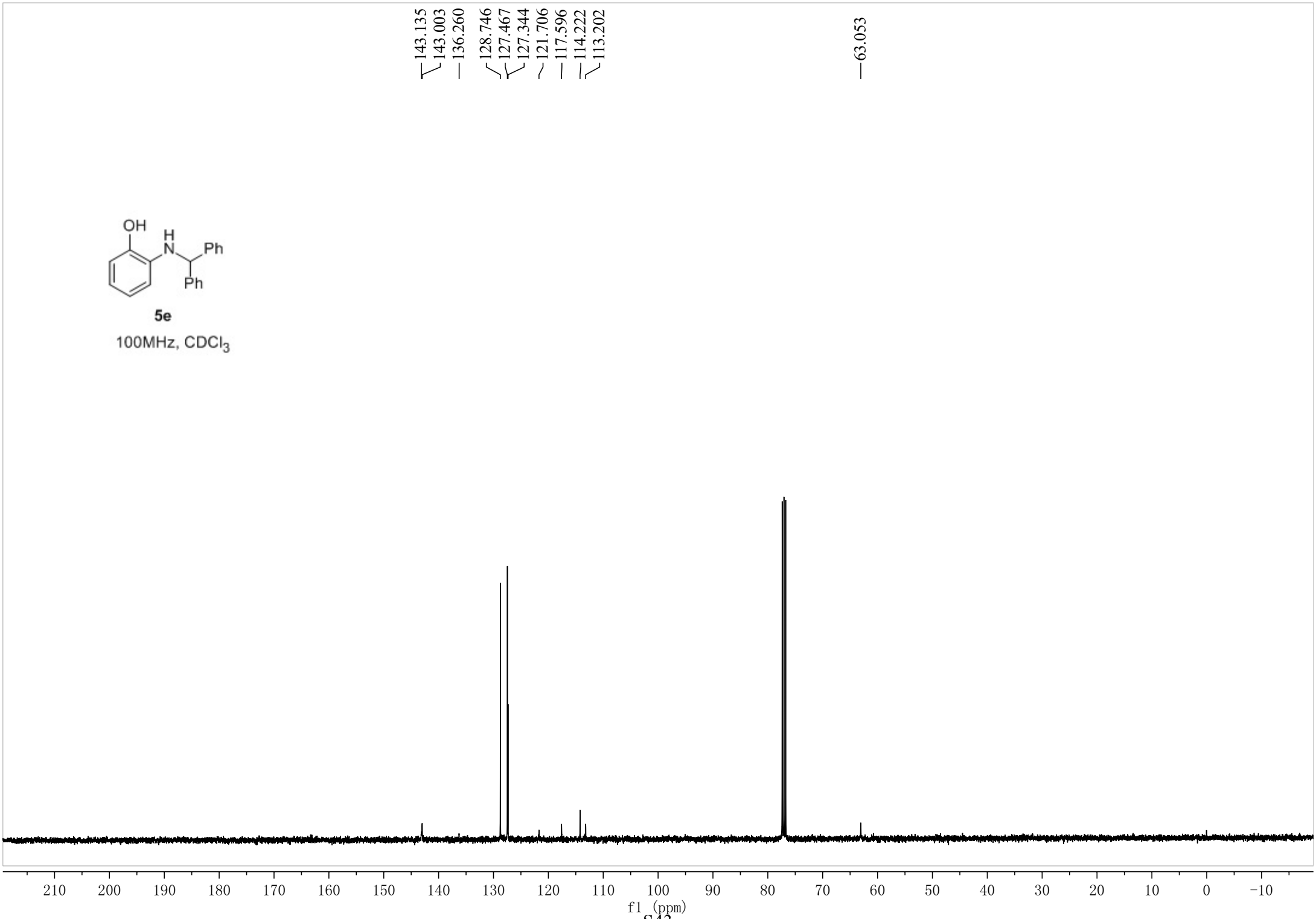
4.16
2.26
2.01
1.03
0.98
1.00
1.05
0.97
f1 (ppm)


143.135
143.003
136.260
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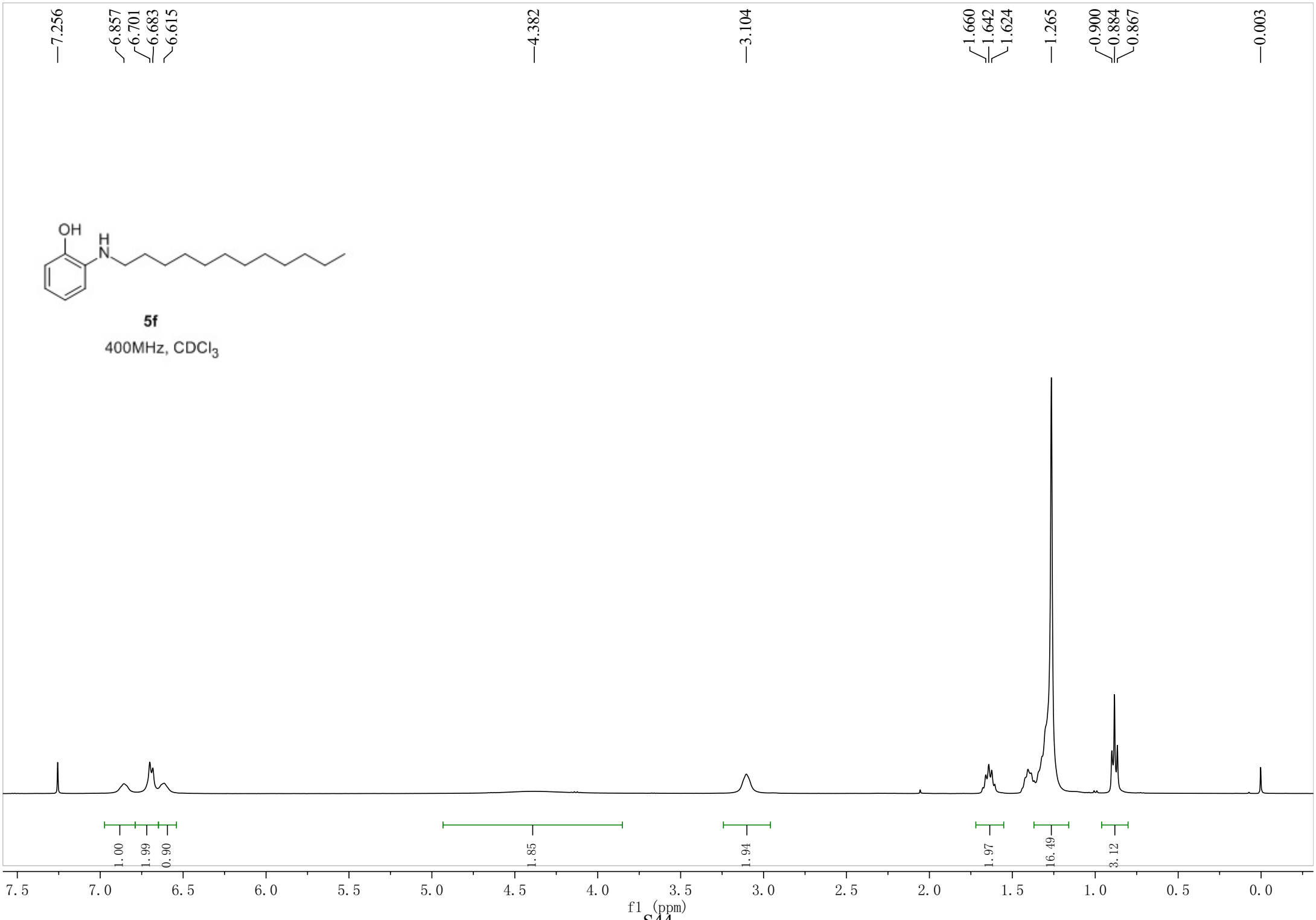
127.467
127.344
121.706
117.596
114.222
113.202
63.053
OH
H
N
Ph
Ph
5e
100MHz, CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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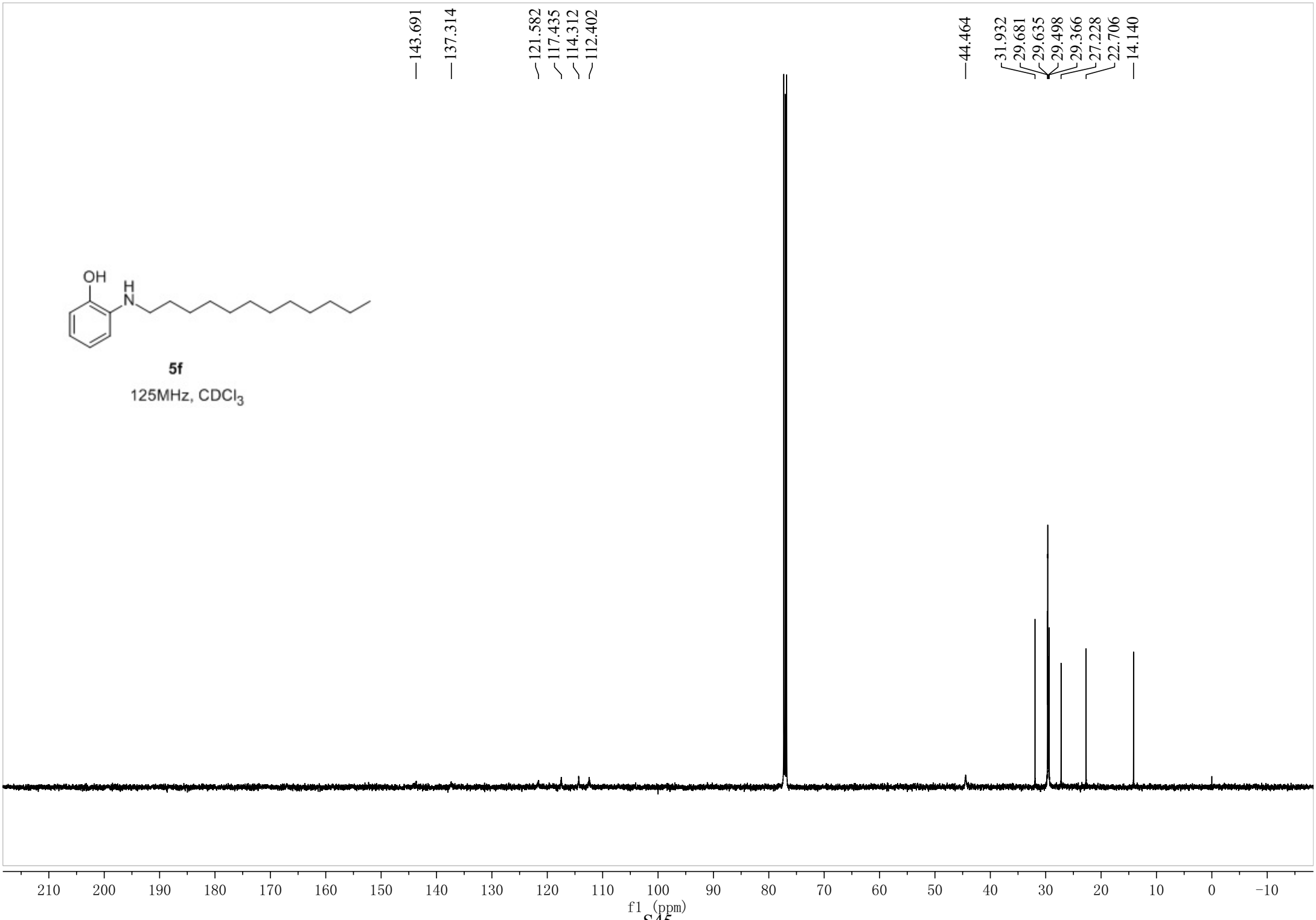
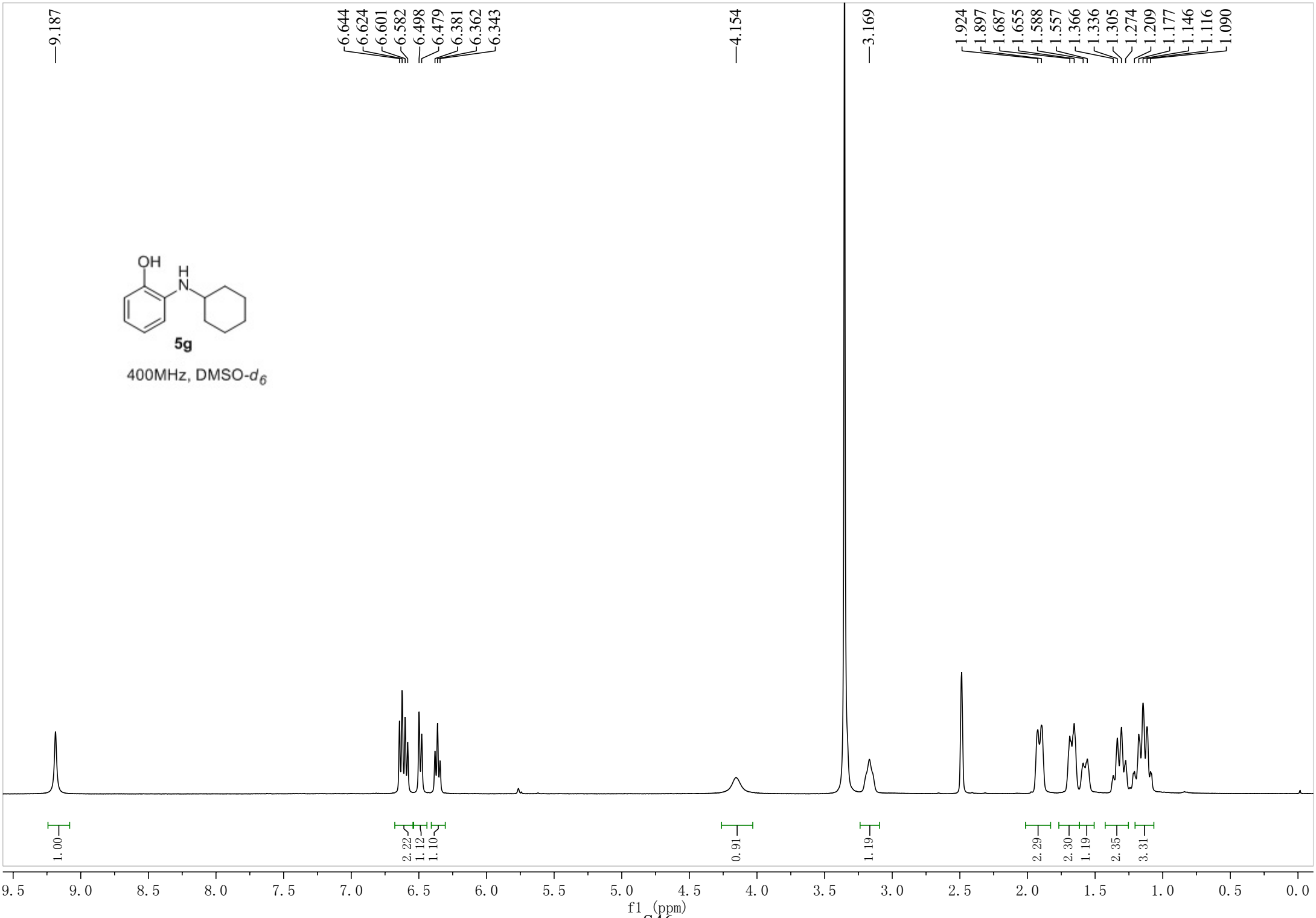
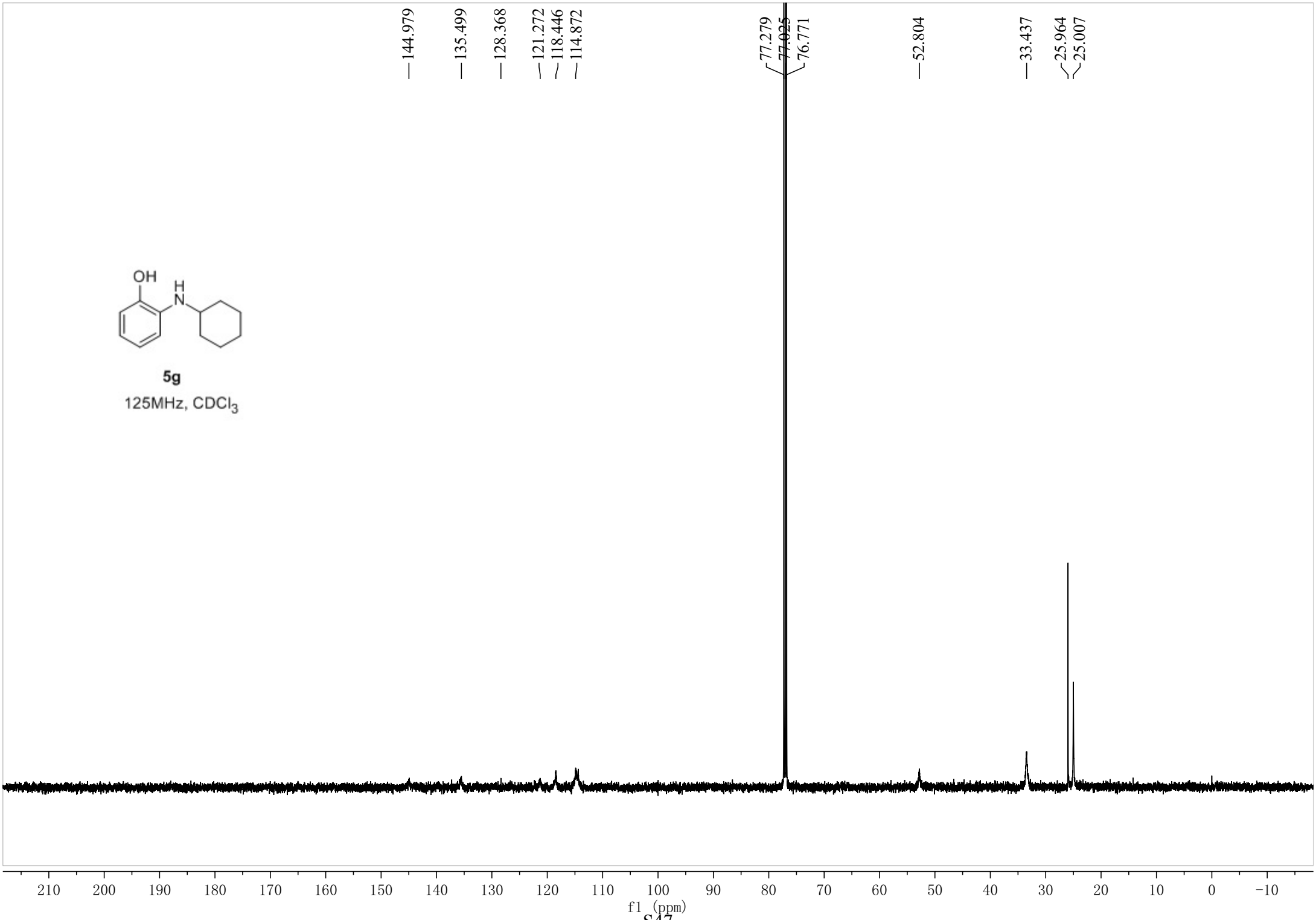
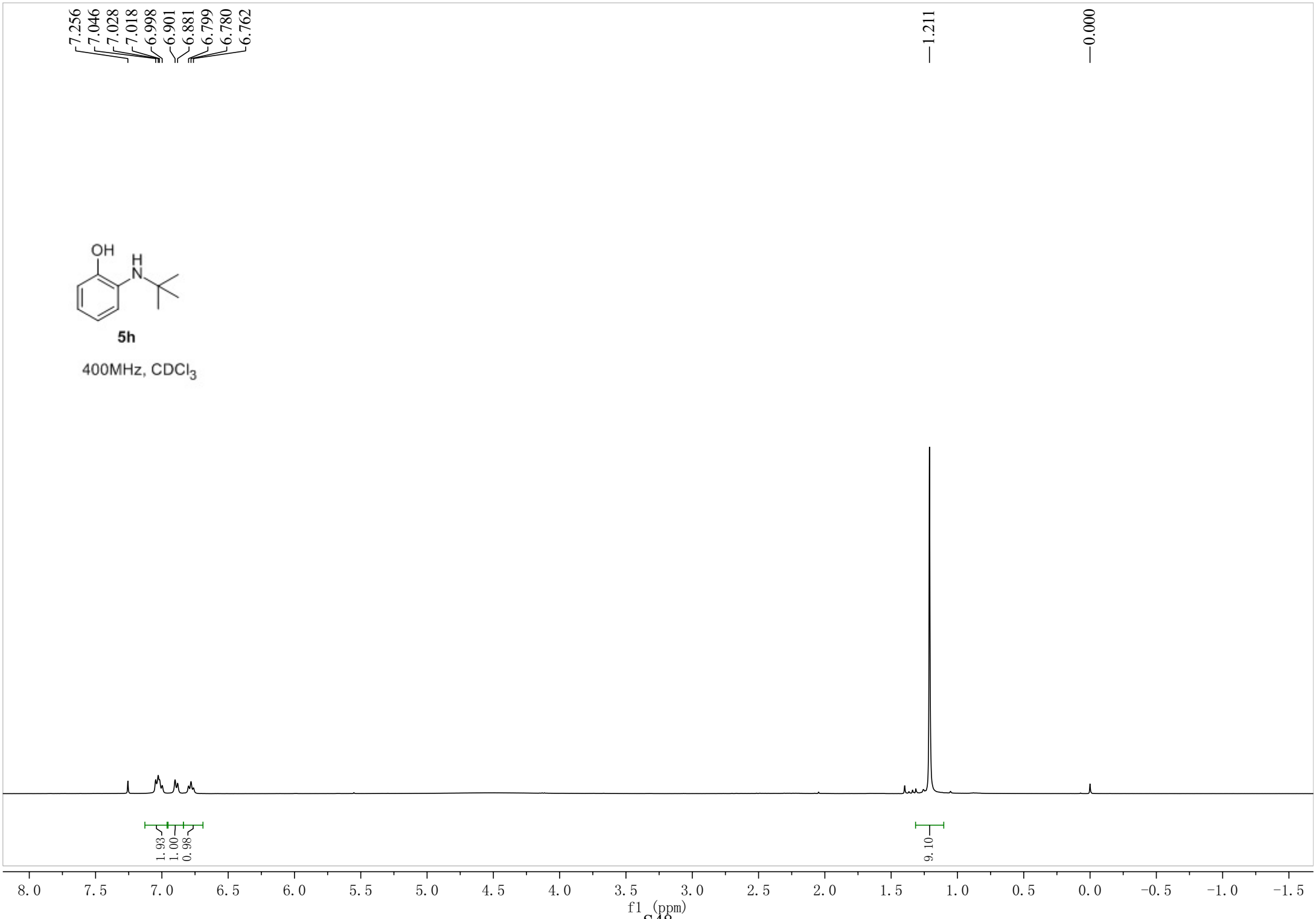
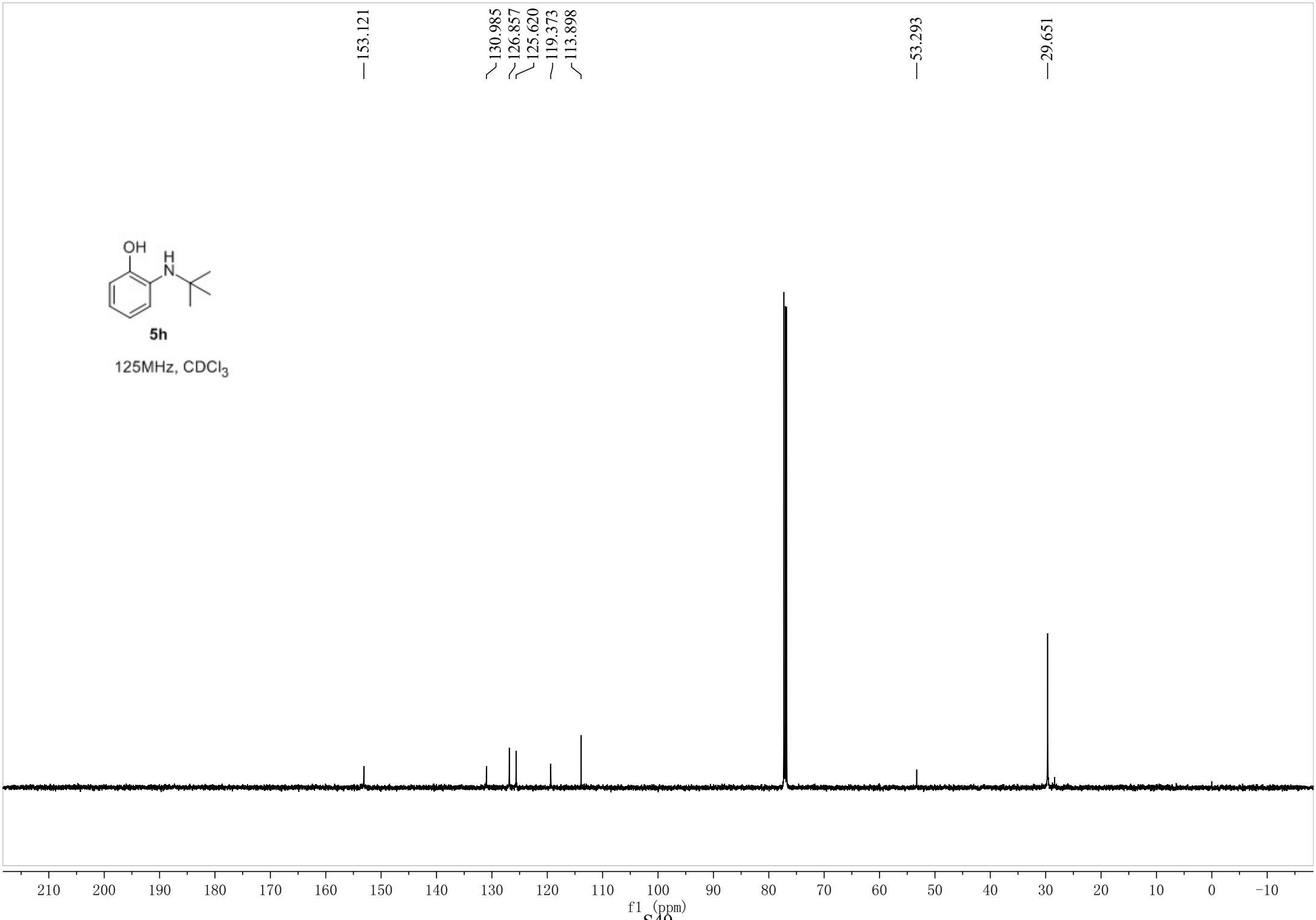
6.857
6.701
6.683
6.615
4.382
3.104
1.660
1.642
1.624
1.265
0.900
0.884
0.867
0.003
OH
H
N
5f
400MHz, CDCl3
1.00
1.99
0.90
1.85
1.94
1.97
16.49
3.12
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

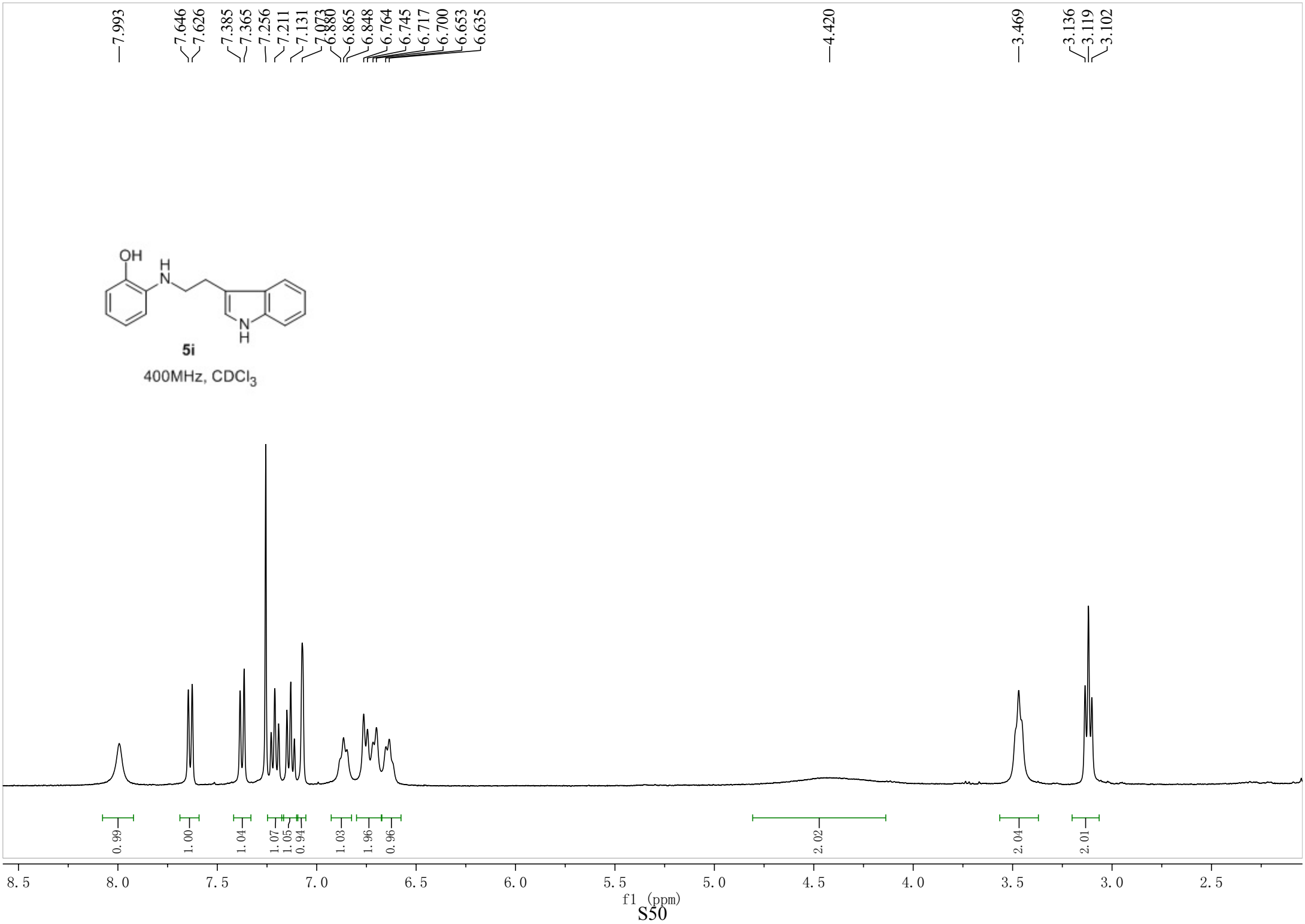

143.691
137.314
121.582
117.435
114.312
112.402
44.464
31.932
29.681
29.635
29.498
29.366
27.228
22.706
14.140
OH
H
N
5f
125MHz, CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

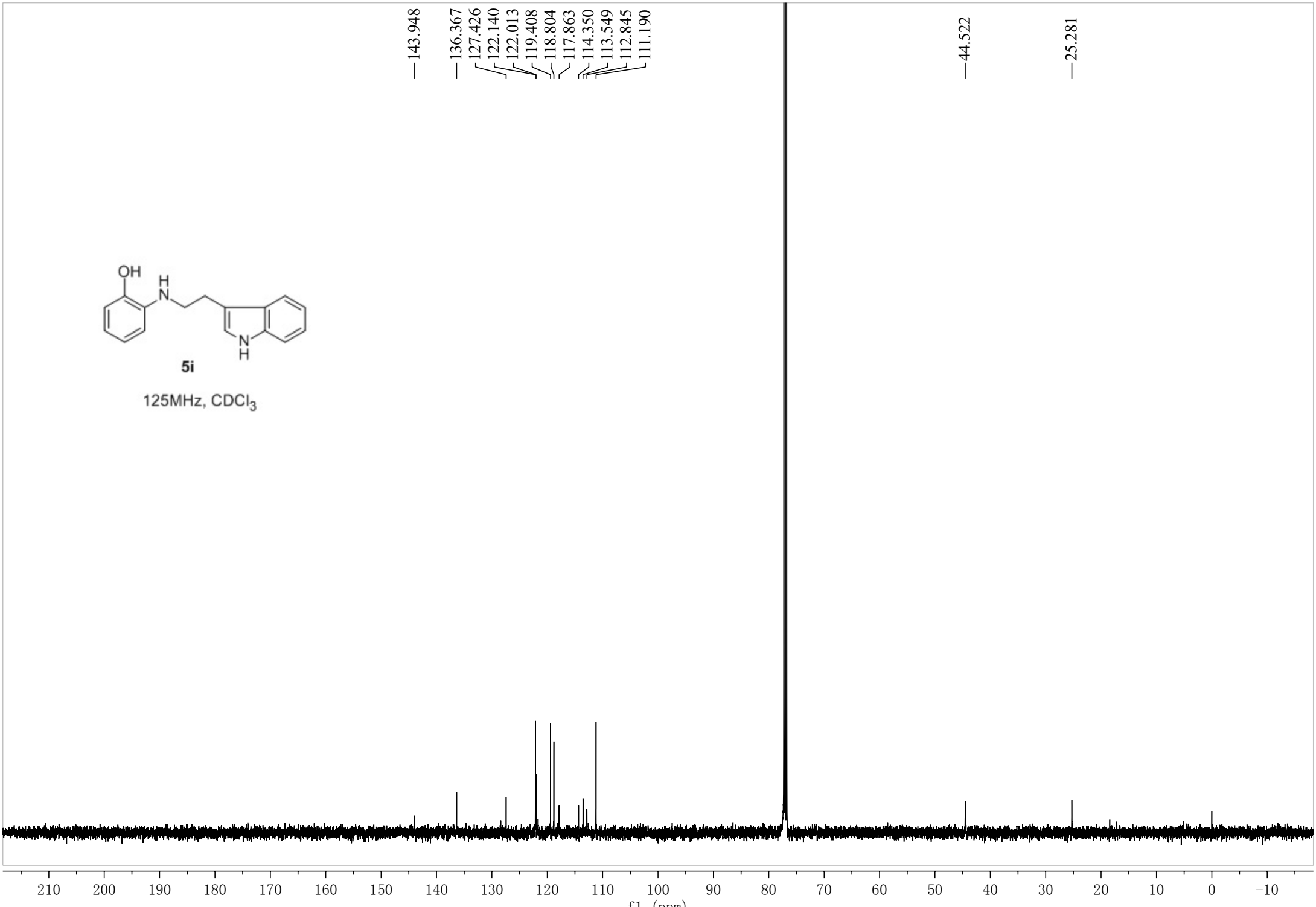

9.187
6.644
6.624
6.601
6.582
6.498
6.479
6.381
6.362
6.343
4.154
3.169
1.924
1.897
1.687
1.655
1.588
1.557
1.366
1.336
1.305
1.274
1.209
1.177
1.146
1.116
1.090
OH
H
N
5g
400MHz, DMSO-d6
1.00
2.22
1.12
1.10
0.91
1.19
2.29
2.30
1.19
2.35
3.31
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

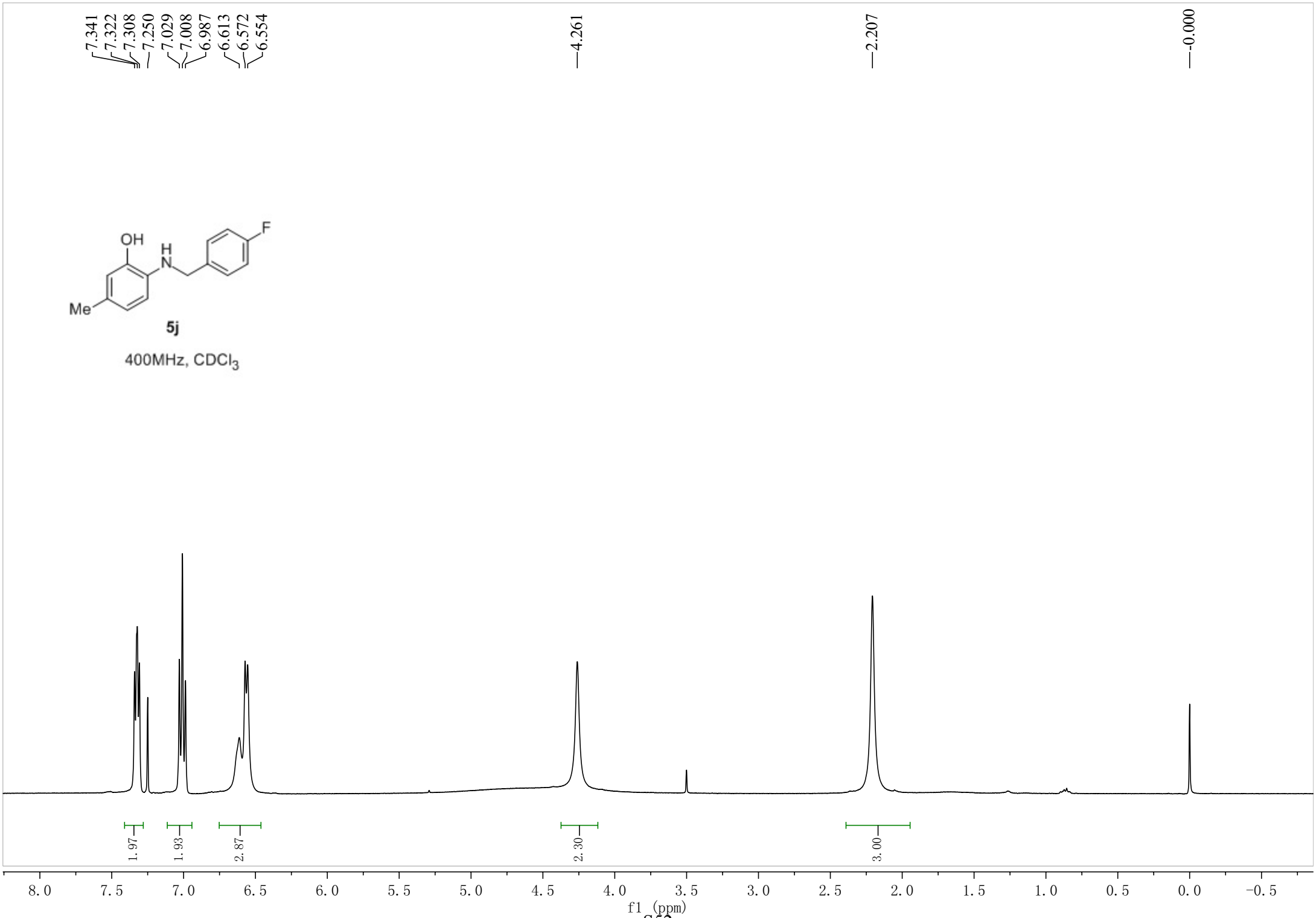

144.979
135.499
128.368
121.272
118.446
114.872
77.279
77.025
76.771
52.804
33.437
25.964
25.007
OH
H
N
5g
125MHz, CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

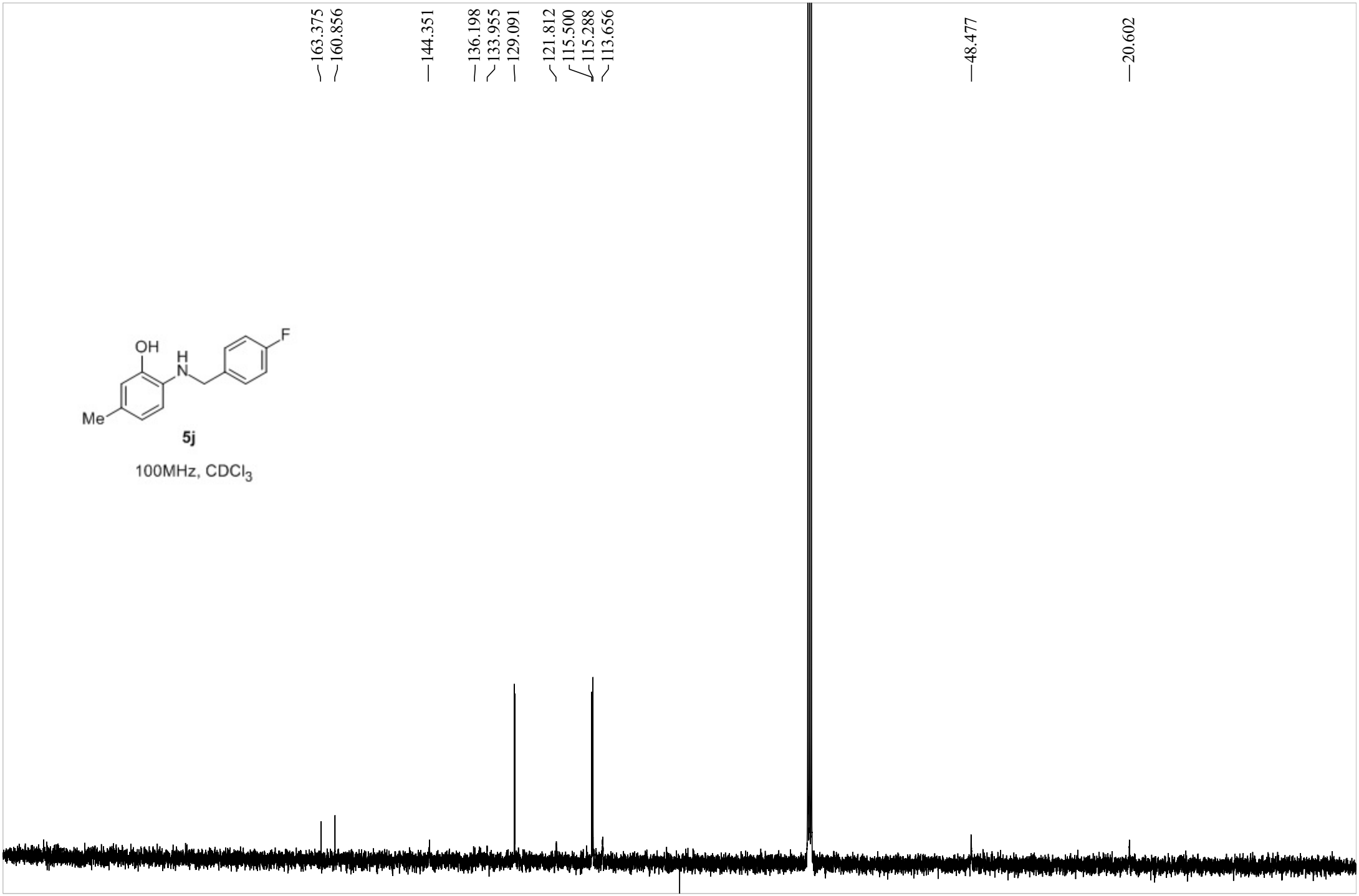

7.256
7.046
7.028
7.018
6.998
6.901
6.881
6.799
6.780
6.762
1.211
0.000
OH
H
N
5h
400MHz, CDCl3
1.93
1.00
0.98
9.10
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
-1.5
f1 (ppm)

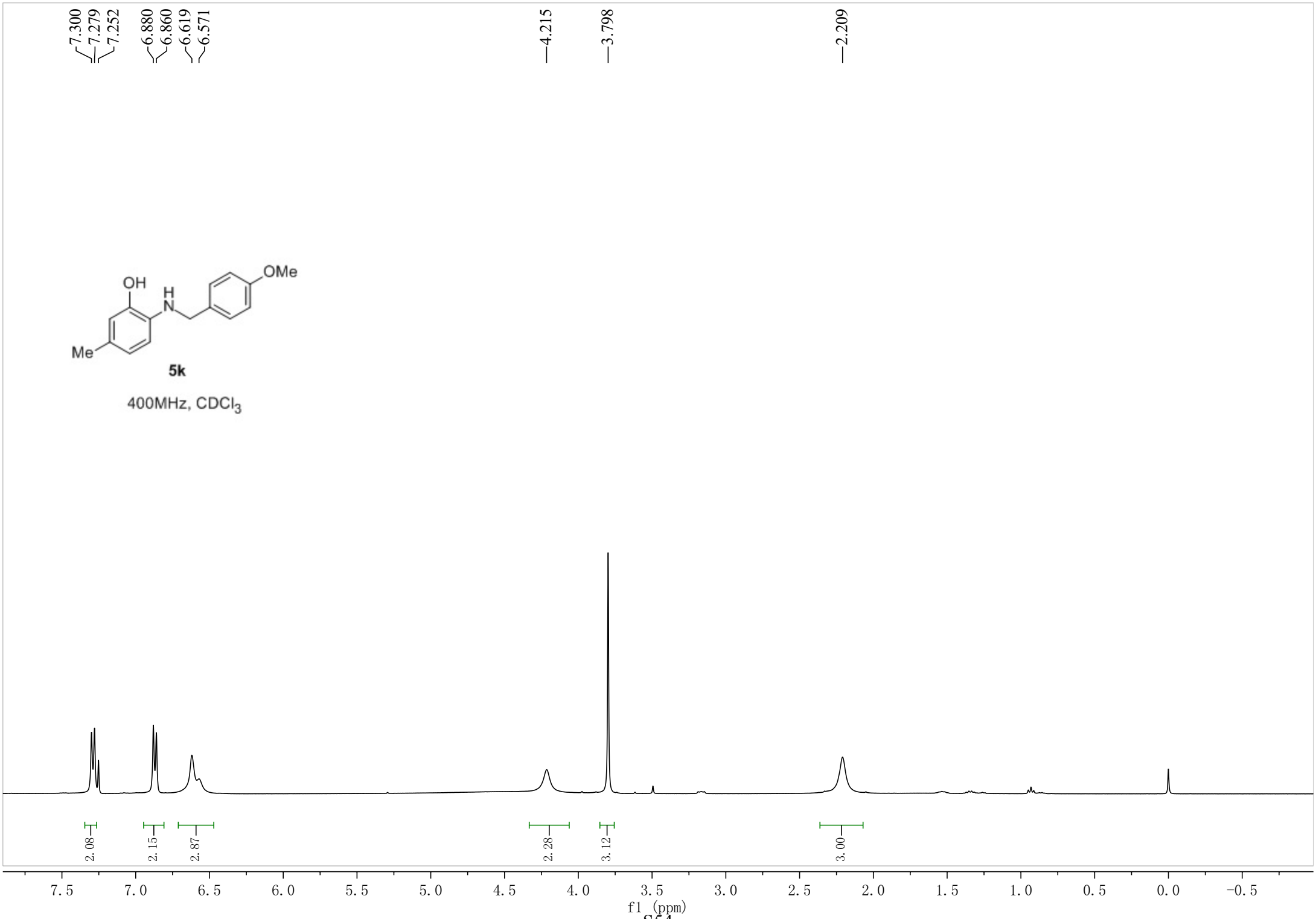

153.121
130.985
126.857
125.620
119.373
113.898
53.293
29.651
OH
H
N
5h
125MHz, CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

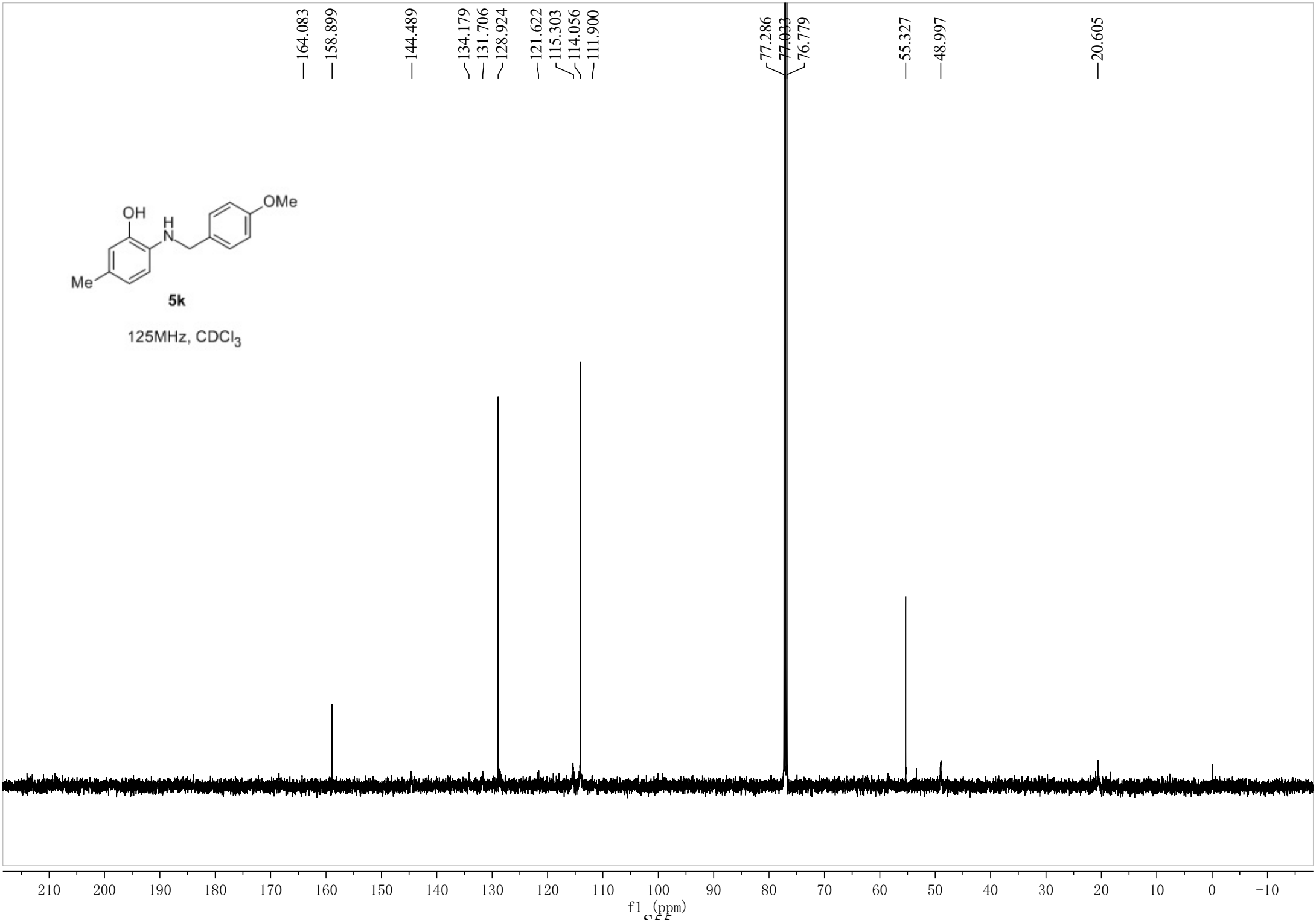

7.993
7.646
7.626
7.385
7.365
7.256
7.211
7.131
7.073
6.880
6.865
6.848
6.764
6.745
6.717
6.700
6.653
6.635
4.420
3.469
3.136
3.119
3.102
OH
H
N
N
H
5i
400MHz, CDCl3
0.99
1.00
1.04
1.07
1.05
0.94
1.03
1.96
0.96
2.02
2.04
2.01
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
f1 (ppm)

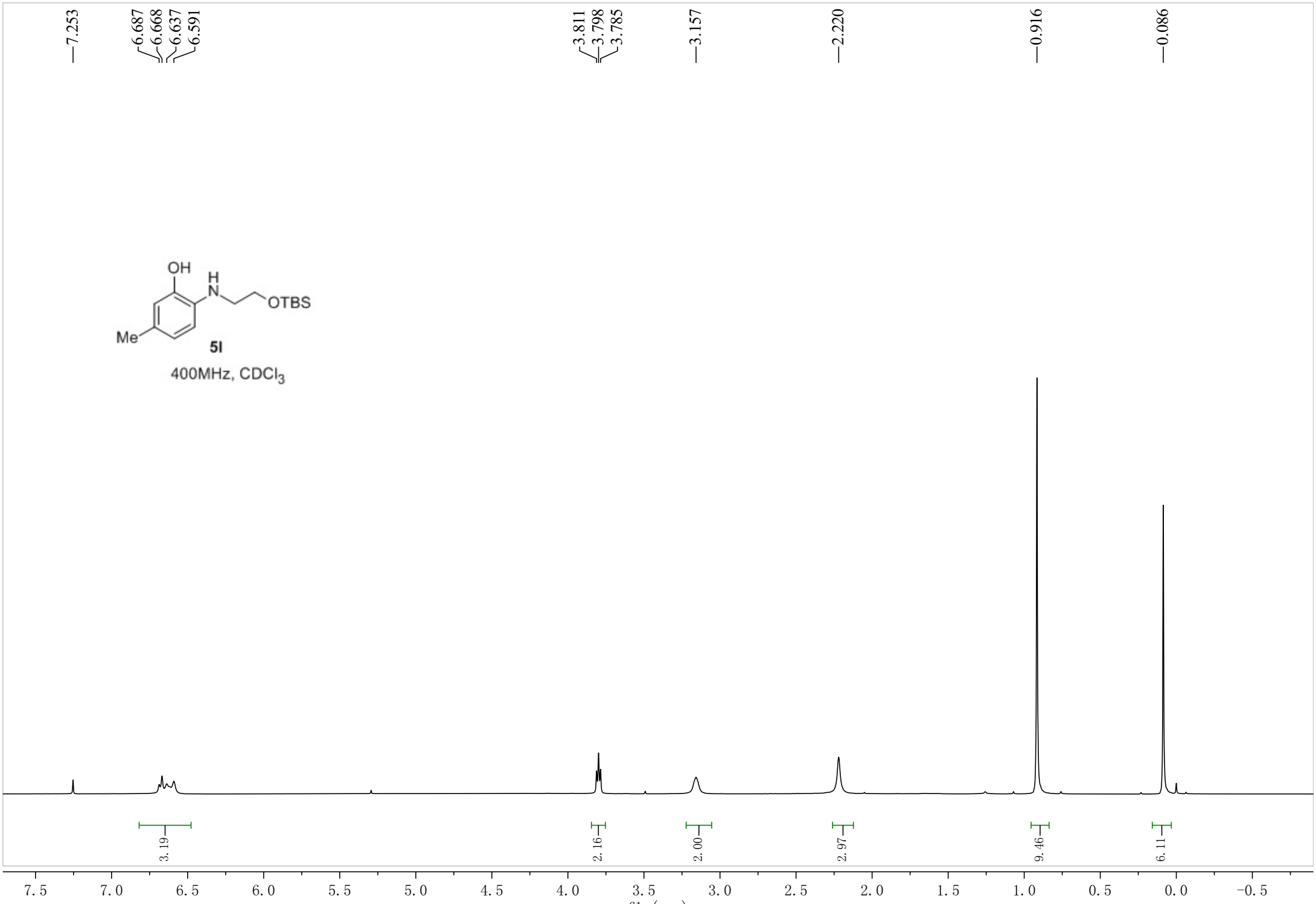

143.948
136.367
127.426
122.140
122.013
119.408
118.804
117.863
114.350
113.549
112.845
111.190
44.522
25.281
OH
H
N
N
H
5i
125MHz, CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

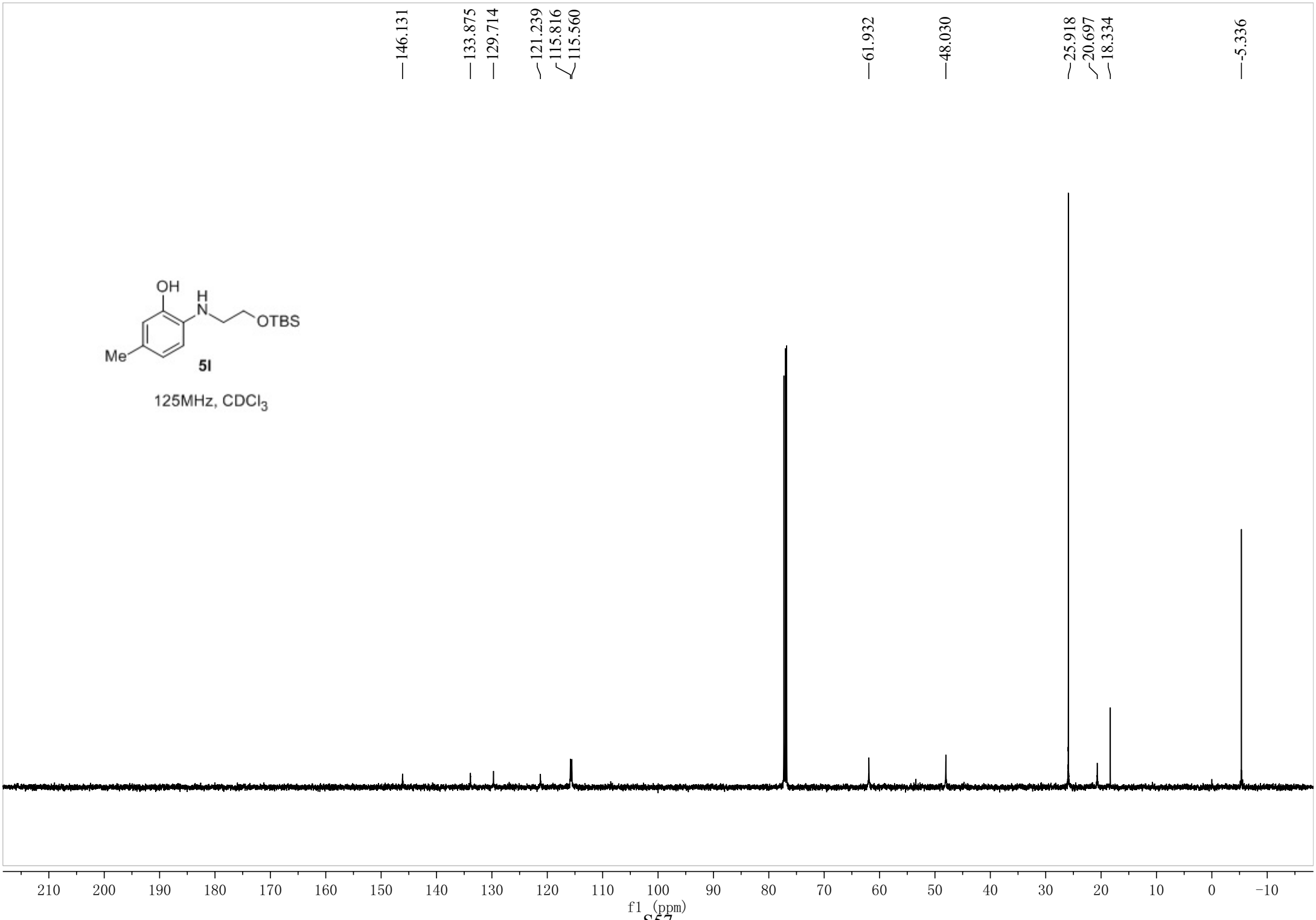

7.341
7.322
7.308
7.250
7.029
7.008
6.987
6.613
6.572
6.554
4.261
2.207
0.000
OH
H
N
F
Me
5j
400MHz, CDCl3
1.97
1.93
2.87
2.30
3.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

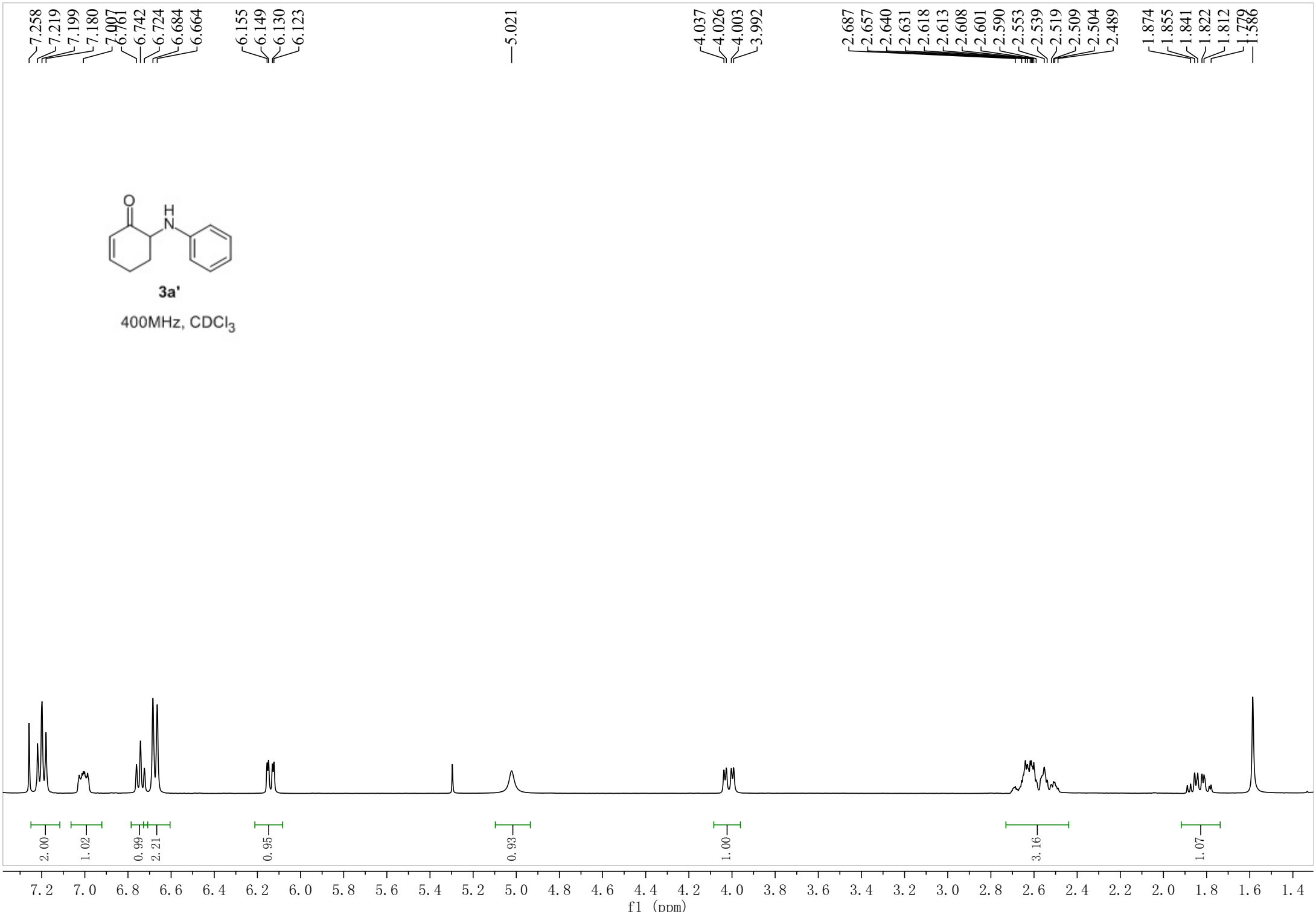

163.375
160.856
144.351
136.198
133.955
129.091
121.812
115.500
115.288
113.656
48.477
20.602
OH
H
N
F
Me
5j
100MHz, CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

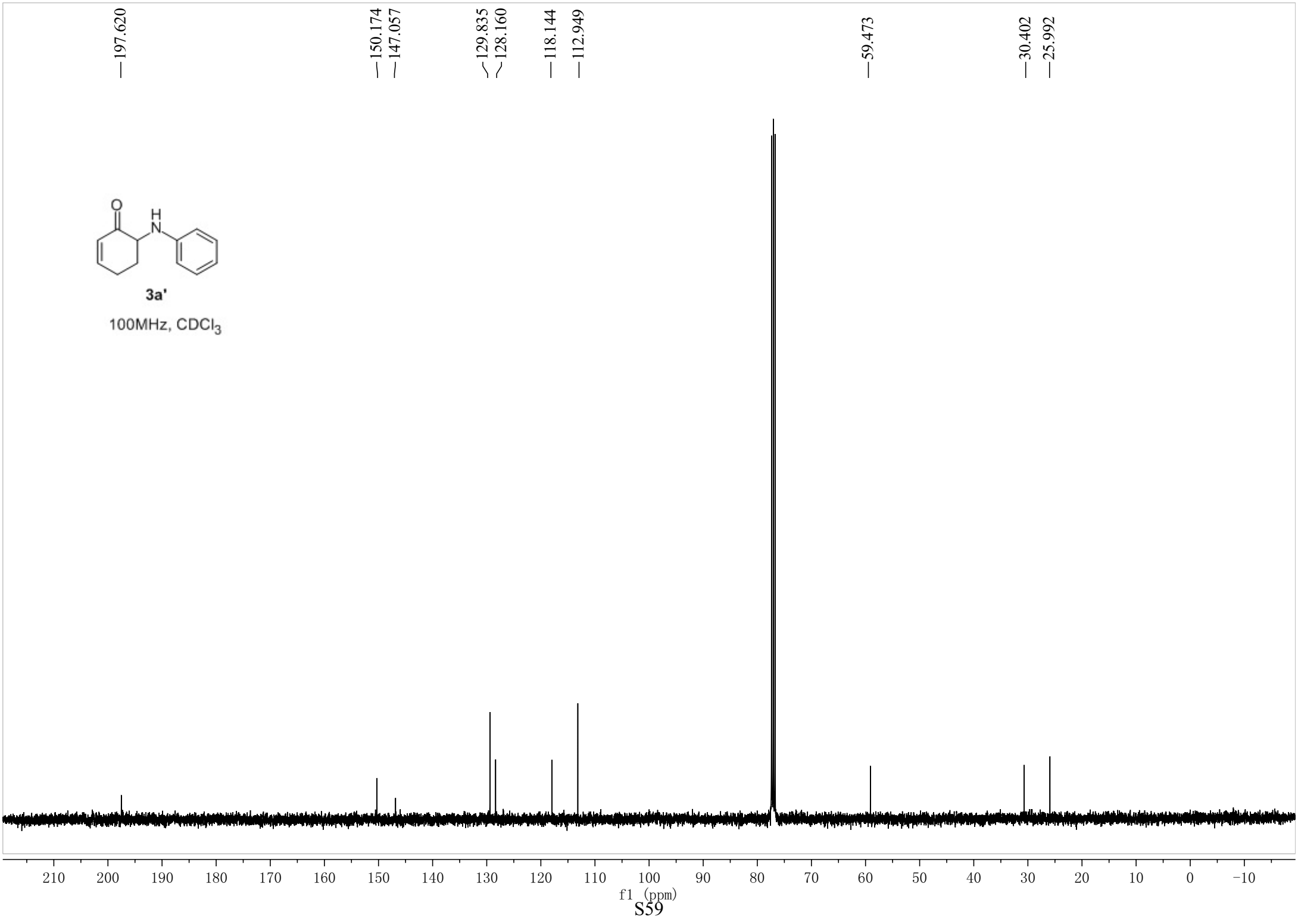

7.300
7.279
7.252
6.880
6.860
6.619
6.571
4.215
3.798
2.209
OH
H
N
OMe
Me
5k
400MHz, CDCl3
2.08
2.15
2.87
2.28
3.12
3.00
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

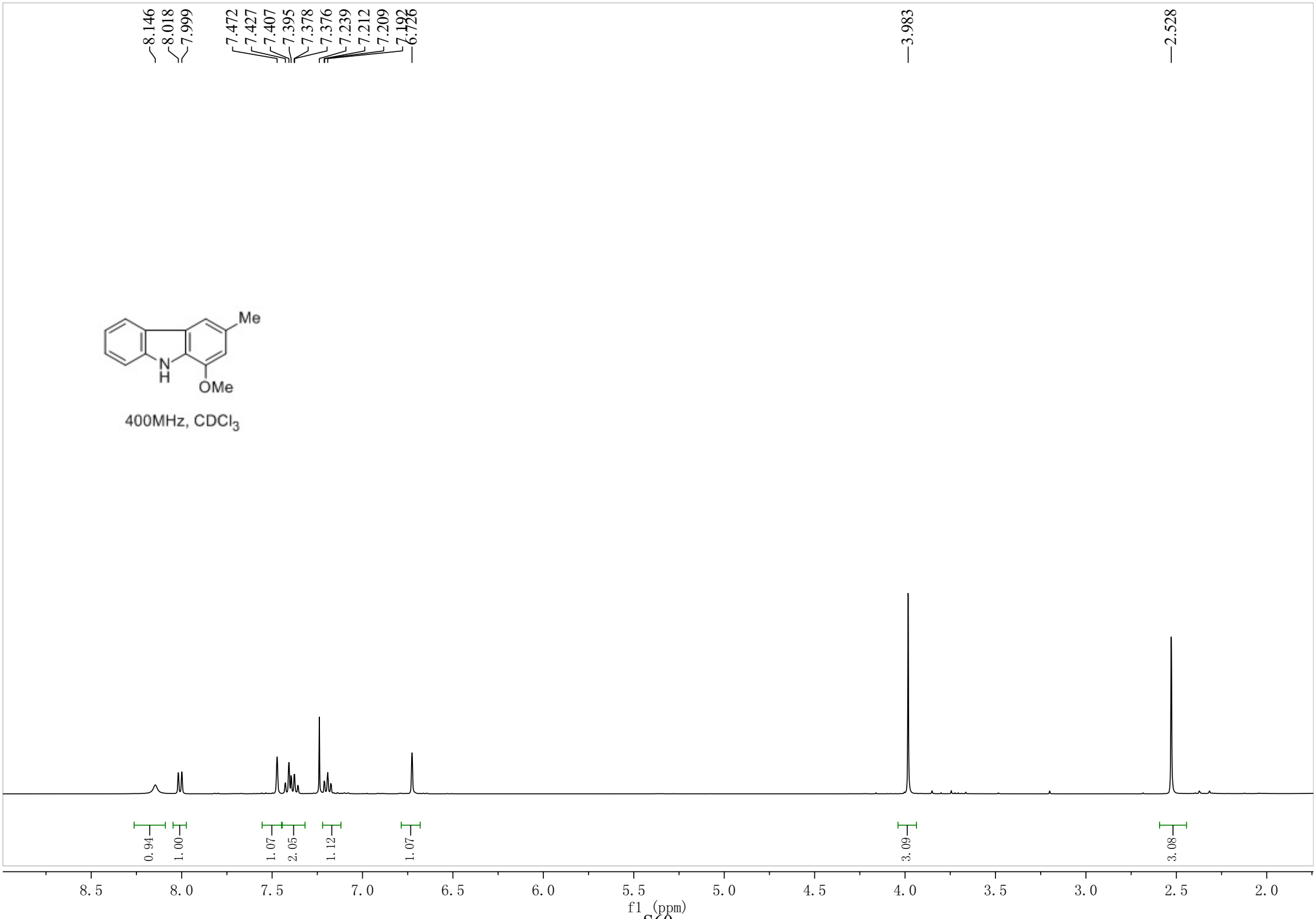

164.083
158.899
144.489
134.179
131.706
128.924
121.622
115.303
114.056
111.900
77.286
77.033
76.779
55.327
48.997
20.605
OH
H
N
OMe
Me
5k
125MHz, CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


7.253
6.687
6.668
6.637
6.591
3.811
3.798
3.785
3.157
2.220
0.916
0.086
OH
H
N
OTBS
Me
5l
400MHz, CDCl3
3.19
2.16
2.00
2.97
9.46
6.11
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


146.131
133.875
129.714
121.239
115.816
115.560
61.932
48.030
25.918
20.697
18.334
-5.336
OH
H
N
OTBS
Me
5l
125MHz, CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


7.258
7.219
7.199
7.180
7.007
6.981
6.742
6.724
6.684
6.664
6.155
6.149
6.130
6.123
5.021
4.037
4.026
4.003
3.992
2.687
2.657
2.640
2.631
2.618
2.613
2.608
2.601
2.590
2.553
2.539
2.519
2.509
2.504
2.489
1.874
1.855
1.841
1.822
1.812
1.779
1.586
O
H
N
3a'
400MHz, CDCl3
2.00
1.02
0.99
2.21
0.95
0.93
1.00
3.16
1.07
7.2
7.0
6.8
6.6
6.4
6.2
6.0
5.8
5.6
5.4
5.2
5.0
4.8
4.6
4.4
4.2
4.0
3.8
3.6
3.4
3.2
3.0
2.8
2.6
2.4
2.2
2.0
1.8
1.6
1.4
f1 (ppm)


197.620
150.174
147.057
129.835
128.160
118.144
112.949
59.473
30.402
25.992
O
H
N
3a'
100MHz, CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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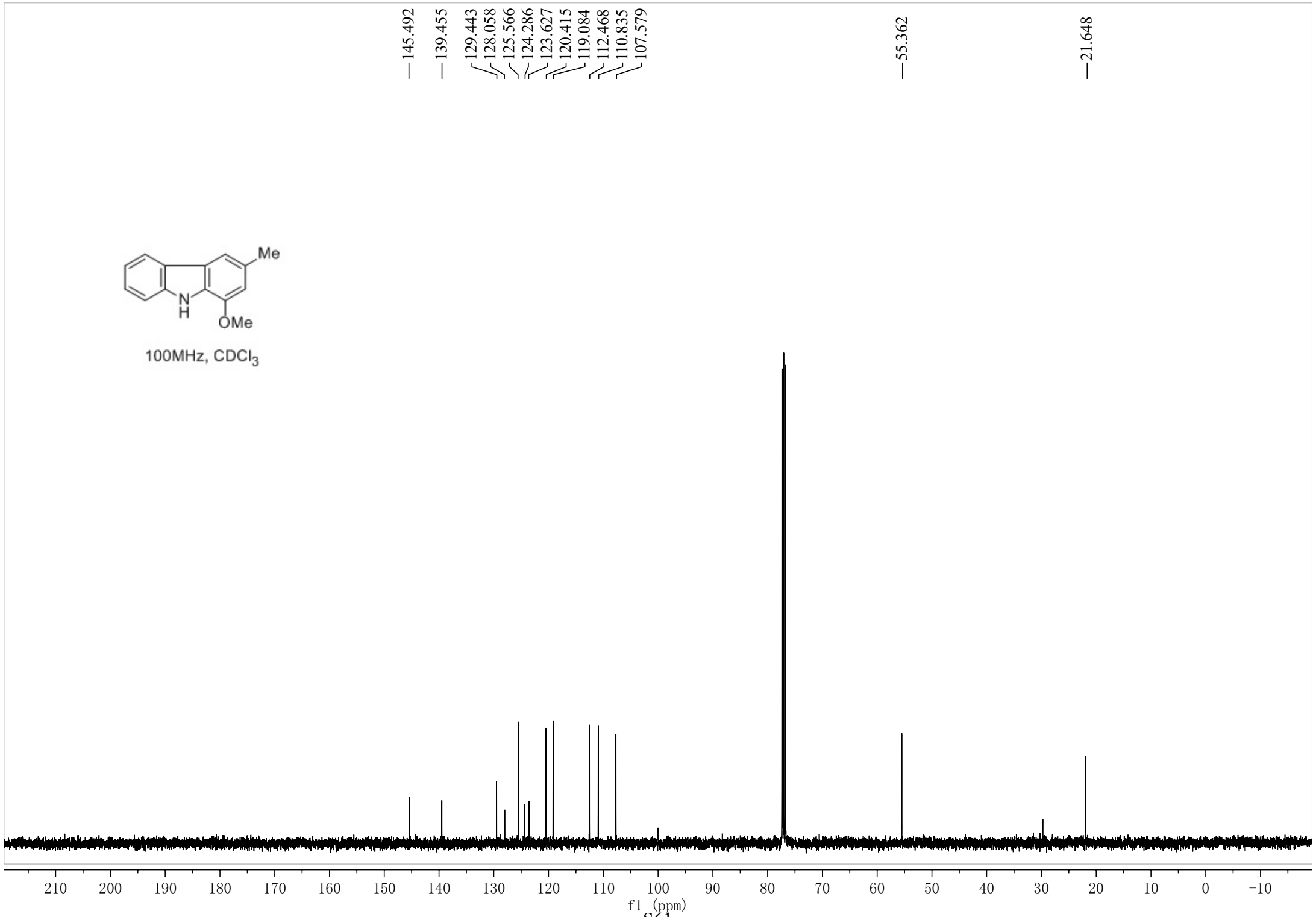
8.018
7.999
7.472
7.427
7.407
7.395
7.378
7.376
7.239
7.212
7.209
7.192
6.726
3.983
2.528
Me
N
H
OMe
400MHz, CDCl3
0.94
1.00
1.07
2.05
1.12
1.07
3.09
3.08
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
f1 (ppm)


145.492
139.455
129.443
128.058
125.566
124.286
123.627
120.415
119.084
112.468
110.835
107.579
55.362
21.648
Me
N
H
OMe
100MHz, CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)