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Supporting Information

Synthesis of 4-Ynamides and Cyclization by the Vilsmeier Reagent to Dihydrofuran-2(3*H*)-ones

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I. General information

All reagents were purchased from commercial sources and used without treatment, unless otherwise indicated. The products were purified by column chromatography over silica gel. ^{1}H-NMR and ^{13}C-NMR spectra were recorded on a Varian 400 MHz or 500 MHz and 125 MHz respectively and TMS was used as internal standard. Mass spectra were recorded on BRUKER AutoflexIII Smartbeam MS-spectrometer. High resolution mass spectra (HRMS) were recorded on Brucker microTof by using ESI method.

II. Crystallography of the compounds 3nb and 4d

The structure of 4-ynamide **3nb** and 5-benzylidenedihydrofuran-2(*3H)*-one **4d** was confirmed by the X-ray diffraction. Single-crystal X-ray diffraction data for the reported compounds was recorded at a temperature of 293(2) on an Oxford Diffraction Gemini R Ultra diffractometer, using a ω scan technique with Mo-Kα radiation (λ = 0.71073 Å). The structure was solved by Direct Method of SHELXS-97 and refined by full-matrix least-squares techniques using the SHELXL-97 program.[1] Non-hydrogen atoms were refined with anisotropic temperature parameters and hydrogen atoms of the compounds were refined as rigid groups. Basic information pertaining to crystal parameters and structure refinement is summarized in Table S1 and Table S2.

Ref: 1 (*a*) G. M. Sheldrick, *SHELXS-97, Program for Solution of Crystal Structures, University of Gottingen,* Germany, 1997; (*b*) G. M. Sheldrick, *SHELXL-97, Program for Refinement of Crystal Structures, University of Gottingen,* Germany, 1997.

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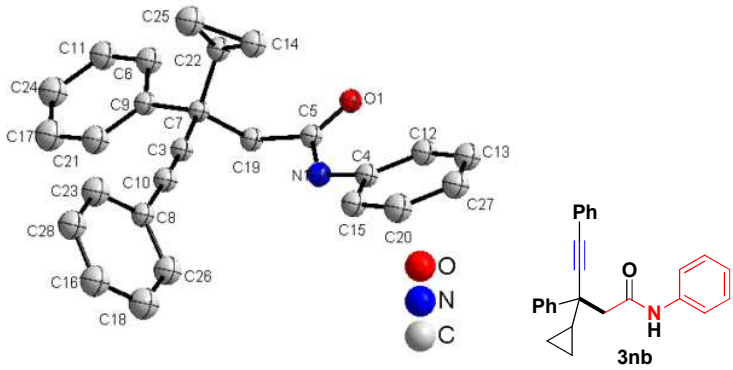
Table S1 Crystal data of 3nb and structure refinement



Empirical formula	$C_{26}H_{23}NO$
Temperature	293(2) K
Wavelength	0.71073 Å
Unit cell dimensions	a = 9.0840(9) Å b = 9.3124(9) Å c = 23.625(2) Å alpha = 90.000 deg. beta = 90.000 deg. gamma = 90.000 deg.
Volume	1997.7(3) $Å^3$
Z	40
Calculated density	1.431 Mg/m^3
Absorption coefficient	0.129 mm^{-1}
F(000)	880
Crystal size	0.1 x 0.1x 0.1 mm
Theta range for data collection	1.72 to 26.05 deg.
Reflections collected / unique	12128 / 3952 [R(int) = 0.0498]
Data / restraints / parameters	3952 / 0 / 113
Goodness-of-fit on F^2	1.643
Final R indices [I>2sigma(I)]	R1 = 0.0984, wR2 = 0.2547
R indices (all data)	R1 = 0.1173, wR2 = 0.2652

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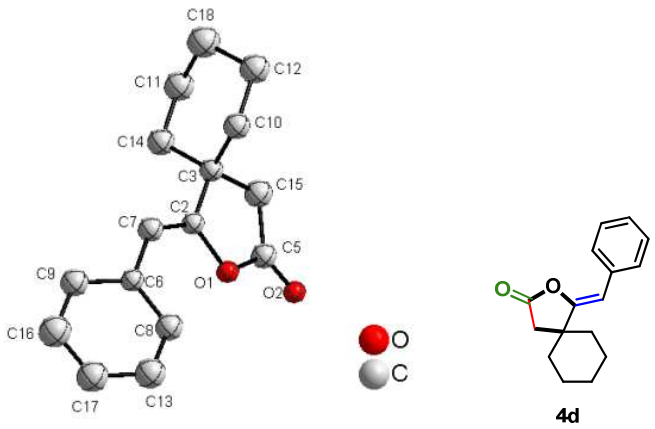
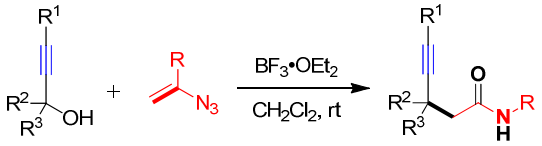
Table S2 Crystal data of 4d and structure refinement

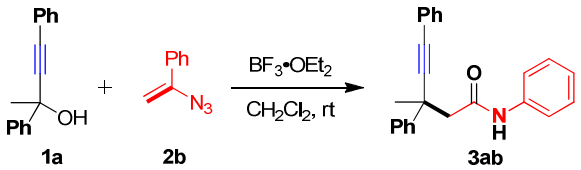


Empirical formula	$C_{16}H_{18}O_2$
Temperature	293(2)K
Wavelength	0.71073 Å
Unit cell dimensions	a = 26.558(3) Å b = 7.6785(7) Å c = 15.0400(15) Å alpha = 90.000 deg. beta = 121.8490(10) deg. gamma = 90.000 deg.
Volume	2605.3(4) $Å^3$
Z	76
Calculated density	0.154 Mg/m^3
Absorption coefficient	0.010 mm^{-1}
F(000)	130
Crystal size	0.1 x 0.1x 0.1 mm
Theta range for data collection	2.71 to 25.03 deg.
Reflections collected / unique	7113 / 2293 [R(int) = 0.0297]
Data / restraints / parameters	2293 / 0 / 73
Goodness-of-fit on F^2	3.048
Final R indices [I>2sigma(I)]	R1 = 0.1478, wR2 = 0.4050
R indices (all data)	R1 = 0.1684, wR2 = 0.4292

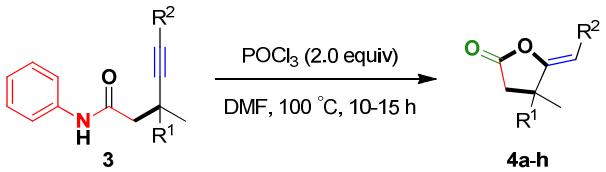
III. Synthesis and analytical data of compounds 3 and 4



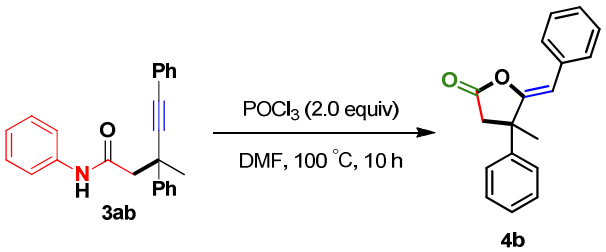
Synthesis of the compound 3ab :



A mixture of 2,4-diphenylbut-3-yn-2-ol (**1a**) (222.2 mg, 1.0 mmol) and 1-azidovinyl-benzene (**2b**) (72.5 mg, 0.5 mmol) in CH_2Cl_2 (3.0 mL) were stirred at 25 °C under N_2 atmosphere, then added BF_3•Et_2O (18 µL, 0.15 mmol), stirring continued until the substrate **2b** was consumed as indicated by TLC (about 10 min). After the completion of reaction, quenched with aqueous saturated $NaHCO_3$ solution (10 mL). The resulting mixture was extracted three times with DCM (3 x 10 mL). The organic layer was washed with brine, dried over $MgSO_4$ and concentrated. Purification of the crude product by column chromatography (silica gel; Hexane : Ethyl acetate = 20 : 1) afforded **3ab** (135.4 mg, 80%) as a colorless oil.

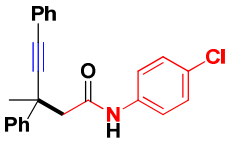


Synthesis of the compound 4b :



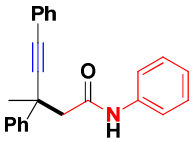
The Vilsmeier reagent was prepared by adding $POCl_3$ (92.0 mg, 0.6 mmol) dropwise to ice-cold dry DMF (3.0 mL) while stirring under air atmophere. After 30 minutes, to the resulting Vilsmeier reagent was added 3-methyl-*N*,3,5-triphenylpent-4-ynamide **3ab** (101.8 mg, 0.3 mmol). Later, the reaction mixture was stirred at 100 °C for 10 h under air atmophere. After complete consumption of **3ab** as indicated by TLC, the reaction was quenched with water (10 mL). Resulting mixture was extracted three times with

DCM (3 x 10 mL). The organic layer was washed with brine, dried over $MgSO_4$ and concentrated. Purification of the crude product by column chromatography (silica gel; Hexane : Ethyl acetate = 20: 1) afforded **4b** (64.0 mg, 81%) as colorless oil.



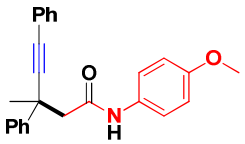
3aa

(**3aa**) Starting materials: 2,4-diphenylbut-3-yn-2-ol **1a** (222.2 mg, 1.0 mmol), 1-(1-azidovinyl)-4-chlorobenzene **2a** (89.8 mg, 0.5 mmol), BF_3•Et_2O (18 μL, 0.15 mmol), and CH_2Cl_2 (3.0 mL); Reaction temperature = 25 ^oC; Reaction time = 10 min under N_2 atmosphere; Column chromatography on silica gel = 20 : 1 Hexane : Ethylacetate; The product **3aa**: Yield = 155.3 mg, 83%; White solid, m.p. 91-92 ^oC; ^{1}**H-NMR** (500 MHz, $CDCl_3$) δ 1.81 (s, 3H), 2.98 (AB, *J* = 14.0 Hz, 2H), 7.17 (d, *J* = 9.0 Hz, 2H), 7.23 (d, *J* = 9.0 Hz, 2H), 7.29 (t, *J* = 7.5 Hz, 1H), 7.34 (d, *J* = 6.5 Hz, 3H), 7.39 (t, *J* = 7.5 Hz, 2H), 7.47 (dd, *J* = 1.5 Hz, 7.0 Hz, 2H), 7.65 (d, *J* = 7.5 Hz, 2H), 7.7 (s, 1H); ^{13}**C-NMR** ($CDCl_3$, 125 MHz) δ 30.0, 39.4, 51.8, 86.0, 93.3, 120.8, 122.2, 125.8, 127.3, 128.5, 128.6, 128.7, 128.8, 129.0, 131.6, 136.2, 143.5, 167.8; **HRMS** (ESI) m/z calculated for $C_{24}H_{21}ClNO$ $[M+H]^+$: 374.1312, found : 374.1306.



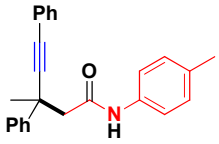
3ab

(**3ab**) Starting materials: 2,4-diphenylbut-3-yn-2-ol **1a** (222.2 mg, 1.0 mmol), 1-azidovinyl-benzene **2b** (72.5 mg, 0.5 mmol), BF_3•Et_2O (18 μL, 0.15 mmol), and CH_2Cl_2 (3.0 mL); Reaction temperature = 25 ^oC; Reaction time = 10 min under N_2 atmosphere; Column chromatography on silica gel = 20 : 1 Hexane : Ethylacetate; The product **3ab**: Yield = 135.4 mg, 80%; Colorless oil; ^{1}**H-NMR** (500 MHz, $CDCl_3$) δ 1.81 (s, 3H), 2.97 (AB, *J* = 14.0 Hz, 2H), 7.03 (t, *J* = 7.5 Hz, 1H), 7.22 (t, *J* = 7.0 Hz, 2H), 7.25-7.35 (m, 6H), 7.38 (t, *J* = 7.5 Hz, 2H), 7.47 (dd, *J* = 1.5 Hz, 6.5 Hz, 2H), 7.66 (d, *J* = 7.5 Hz, 2H), 7.80 (s, 1H); ^{13}**C-NMR** ($CDCl_3$, 125 MHz) δ 29.9, 39.4, 51.8, 85.8, 93.4, 119.7, 122.4, 124.0, 125.8, 127.2, 128.4, 128.5, 128.7, 128.8, 131.6, 137.7, 143.7, 167.7; **FT-IR** (neat) ν = 3310, 3199, 3186, 3058, 3030, 2976, 2931, 1658, 1599, 1542, 1494, 1442, 1380, 1341, 1257, 1157, 1072, 1028, 911, 755, 693 cm^{-1}; **HRMS** (ESI) m/z calculated for $C_{24}H_{22}NONa$ $[M+Na]^+$: 362.1521, found : 362.1515.



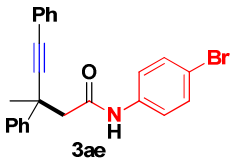
3ac

(**3ac**) Starting materials: 2,4-diphenylbut-3-yn-2-ol **1a** (222.2 mg, 1.0 mmol), 1-(1-azidovinyl)-4-methoxybenzene **2c** (87.5 mg, 0.5 mmol), BF_3•Et_2O (18 μL, 0.15 mmol), and CH_2Cl_2 (3.0 mL); Reaction temperature = 25 ^oC; Reaction time = 10 min under N_2 atmosphere; Column chromatography on silica gel = 20 : 1 Hexane : Ethylacetate; The product **3ac**: Yield = 153.4 mg, 83%; White solid, m.p. 102-103 ^oC; **^{1}H-NMR** (500 MHz, $CDCl_3$) δ 1.81 (s, 3H), 2.96 (AB, *J* = 14.0 Hz, 2H), 3.74 (s, 3H), 6.77 (d, *J* = 9.0 Hz, 2H), 7.20 (d, *J* = 9.0 Hz, 2H), 7.29 (t, *J* = 7.5 Hz, 1H), 7.31-7.35 (m, 3H), 7.39 (t, *J* = 7.5 Hz, 2H), 7.48 (dd, *J* = 2.0 Hz, 6.5 Hz, 2H), 7.65 (s, 1H), 7.67 (d, *J* = 8.0 Hz, 2H); **^{13}C-NMR** ($CDCl_3$, 125 MHz) δ 29.8, 39.4, 51.7, 55.4, 85.8, 93.5, 114.0, 121.6, 122.5, 125.8, 127.2, 128.4, 128.5, 128.7, 130.8, 131.6, 143.8, 156.3, 167.6; **HRMS** (ESI) m/z calculated for $C_{25}H_{24}NO_2$ $[M+H]^+$: 370.1807, found : 370.1802.



3ad

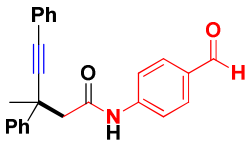
(**3ad**) Starting materials: 2,4-diphenylbut-3-yn-2-ol **1a** (222.2 mg, 1.0 mmol), 1-(1-azidovinyl)-4-methylbenzene **2d** (79.5 mg, 0.5 mmol), BF_3•Et_2O (18 μL, 0.15 mmol), and CH_2Cl_2 (3.0 mL); Reaction temperature = 25 ^oC; Reaction time = 10 min under N_2 atmosphere; Column chromatography on silica gel = 20 : 1 Hexane : Ethylacetate; The product **3ad**: Yield = 145.2 mg, 82%; White solid, m.p. 101-102 ^oC; **^{1}H-NMR** (500 MHz, $CDCl_3$) δ 1.81 (s, 3H), 2.26 (s, 3H), 2.97 (AB, *J* = 14.0 Hz, 2H), 7.03 (d, *J* = 8.5 Hz, 2H), 7.18 (d, *J* = 8.5 Hz, 2H), 7.28 (t, *J* = 7.5 Hz, 1H), 7.31-36 (m, 3H), 7.38 (t, *J* = 7.5, 2H), 7.48 (dd, *J* = 2.0 Hz, 7.0 Hz, 2H), 7.67 (d, *J* = 7.5 Hz, 2H), 7.69 (s, 1H); **^{13}C-NMR** ($CDCl_3$, 125 MHz) δ 20.7, 29.8, 39.4, 51.9, 85.8, 93.5, 119.8, 122.5, 125.8, 127.2, 128.4, 128.5, 128.7, 129.3, 131.6, 133.7, 135.1, 143.8, 167.6; **HRMS** (ESI) m/z calculated for $C_{25}H_{24}NO$ $[M+H]^+$: 354.1858, found : 354.1852.



3ae

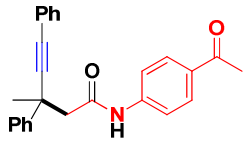
(**3ae**) Starting materials: 2,4-diphenylbut-3-yn-2-ol **1a** (222.2 mg, 1.0 mmol), 1-(1-azidovinyl)-4-bromobenzene **2e** (112.0 mg, 0.5 mmol), BF_3•Et_2O (18 μL, 0.15 mmol), and CH_2Cl_2 (3.0 mL); Reaction temperature = 25 ^oC; Reaction time = 10 min under N_2

atmosphere; Column chromatography on silica gel = 20 : 1 Hexane : Ethylacetate; The product **3ae**: Yield = 177.9 mg, 85%; White solid, m.p. 103-104 °C; **^{1}H-NMR** (500 MHz, $CDCl_3$) δ 1.80 (s, 3H), 2.97 (AB, *J* = 14.0 Hz, 2H), 7.18 (d, *J* = 8.5 Hz, 2H), 7.27-7.36 (m, 6H), 7.39 (t, *J* = 7.5, 2H), 7.47 (dd, *J* = 2.0 Hz, 7.5 Hz, 2H), 7.65 (d, *J* = 7.5 Hz, 2H), 7.75 (s, 1H); **^{13}C-NMR** ($CDCl_3$, 125 MHz) δ 30.0, 39.4, 51.8, 86.0, 93.3, 116.6, 121.2, 122.2, 125.8, 127.3, 128.5, 128.6, 128.8, 131.6, 131.8, 136.7, 143.5, 167.7; **HRMS** (ESI) m/z calculated for $C_{24}H_{21}BrNO$ $[M+H]^+$: 418.0807, found : 418.0801.



3af

(**3af**) Starting materials: 2,4-diphenylbut-3-yn-2-ol **1a** (222.2 mg, 1.0 mmol), 4-(1-azidovinyl)benzaldehyde **2f** (86.5 mg, 0.5 mmol), BF_3•Et_2O (18 μL, 0.15 mmol), and CH_2Cl_2 (3.0 mL); Reaction temperature = 25 °C; Reaction time = 10 min under N_2 atmosphere; Column chromatography on silica gel = 20 : 1 Hexane : Ethylacetate; The product **3af**: Yield = 135.7 mg, 74%; White solid, m.p. 115-116 °C; **^{1}H-NMR** (500 MHz, $CDCl_3$) δ 1.82 (s, 3H), 3.02 (AB, *J* = 14.0 Hz, 2H), 7.30 (t, *J* = 7.5 Hz, 1H), 7.32-7.37 (m, 3H), 7.39 (t, *J* = 7.5 Hz, 2H), 7.44-7.50 (m, 4H), 7.66 (d, *J* = 7.5 Hz, 2H), 7.74 (d, *J* = 8.5 Hz, 2H), 8.07 (s, 1H), 9.85 (s, 1H); **^{13}C-NMR** ($CDCl_3$, 125 MHz) δ 30.0, 39.4, 52.0, 86.2, 93.1, 119.1, 122.1, 125.7, 127.4, 128.5, 128.74, 128.79, 128.8, 130.8, 131.0, 131.6, 132.1, 143.1, 143.3, 168.1, 190.8; **HRMS** (ESI) m/z calculated for $C_{25}H_{22}NO_2$ $[M+H]^+$: 368.1651, found : 387.1645.



3ag

(**3ag**) Starting materials: 2,4-diphenylbut-3-yn-2-ol **1a** (222.2 mg, 1.0 mmol), 1-(4-(1-azidovinyl)phenyl)ethanone **2g** (93.5 mg, 0.5 mmol), BF_3•Et_2O (18 μL, 0.15 mmol), and CH_2Cl_2 (3.0 mL); Reaction temperature = 25 °C; Reaction time = 10 min under N_2 atmosphere; Column chromatography on silica gel = 20 : 1 Hexane : Ethylacetate; The product **3ag**: Yield = 151.0 mg, 79%; White solid, m.p. 125-126 °C; **^{1}H-NMR** (500 MHz, $CDCl_3$) δ 1.81 (s, 3H), 2.50 (s, 3H), 2.99 (AB, *J* = 14.0 Hz, 2H), 7.28 (t, *J* = 7.5 Hz, 1H), 7.30-7.34 (m, 3H), 7.37 (t, *J* = 7.5 Hz, 2H), 7.43 (d, *J* = 8.5 Hz, 2H), 7.45 (d, *J* = 7.5 Hz, 2H), 7.64 (d, *J* = 7.5 Hz, 2H), 7.82 (d, *J* = 8.5 Hz, 2H), 8.21 (s, 1H); **^{13}C-NMR** ($CDCl_3$, 125 MHz) δ 26.3, 29.8, 39.3, 51.7, 85.9, 93.0, 118.6, 122.2, 125.6, 127.2, 128.4, 128.5, 128.6, 129.5, 131.5, 132.5, 142.0, 143.4, 168.1, 196.8; **HRMS** (ESI) m/z calculated for $C_{26}H_{24}NO_2$ $[M+H]^+$: 382.1762, found : 382.1785.

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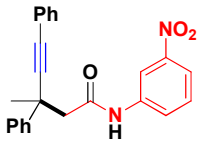
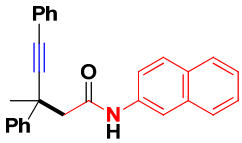


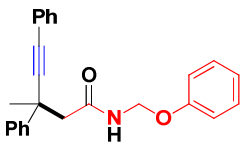
3ah

(**3ah**) Starting materials: 2,4-diphenylbut-3-yn-2-ol **1a** (222.2 mg, 1.0 mmol), 1-(1-azidovinyl)-3-nitrobenzene **2h** (95.0 mg, 0.5 mmol), BF_3•Et_2O (18 μL, 0.15 mmol), and CH_2Cl_2 (3.0 mL); Reaction temperature = 25 ^oC; Reaction time = 10 min under N_2 atmosphere; Column chromatography on silica gel = 20 : 1 Hexane : Ethylacetate; The product **3ah**: Yield = 146.2 mg, 76%; White solid, m.p. 112-113 ^oC; ^{1}**H-NMR** (500 MHz, $CDCl_3$) δ 1.82 (s, 3H), 3.03 (AB, *J* = 14.0 Hz, 2H), 7.31 (t, *J* = 7.5 Hz, 1H), 7.35-7.38 (m, 4H), 7.40 (t, *J* = 7.5 Hz, 2H), 7.48-7.52 (m, 2H), 7.67 (d, *J* = 8.0 Hz, 2H), 7.70 (d, *J* = 9.0 Hz, 1H), 7.85-7.90 (m, 1H), 8.03 (s, 1H), 8.1 (t, *J* = 2.0 Hz, 1H); ^{13}**C-NMR** ($CDCl_3$, 125 MHz) δ 30.1, 39.4, 51.7, 86.3, 93.1, 114.3, 118.6, 122.0, 125.2, 125.7, 127.5, 128.6, 128.84, 128.89, 129.7, 131.6, 138.7, 143.3, 148.4, 168.1; **HRMS** (ESI) m/z calculated for $C_{24}H_{21}N_2O_3$ $[M+H]^+$: 385.1552, found : 385.1547.



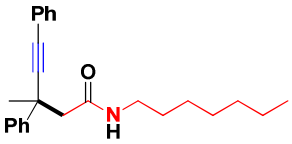
3ai

(**3ai**) Starting materials: 2,4-diphenylbut-3-yn-2-ol **1a** (222.2 mg, 1.0 mmol), 2-(1-azidovinyl)naphthalene **2i** (97.6 mg, 0.5 mmol), BF_3•Et_2O (18 μL, 0.15 mmol), and CH_2Cl_2 (3.0 mL); Reaction temperature = 25 ^oC; Reaction time = 10 min under N_2 atmosphere; Column chromatography on silica gel = 15 : 1 Hexane : Ethylacetate; The product **3ai**: Yield = 145.8 mg, 75%; White solid, m.p. 94-95 ^oC; ^{1}**H-NMR** (500 MHz, $CDCl_3$) δ 1.84 (s, 3H), 3.03 (AB, *J* = 14.0 Hz, 2H), 7.20 (dd, *J* = 2.0 Hz, 9.0 Hz, 1H), 7. 29 (t, *J* = 7.0 Hz, 1H), 7.32-7.44 (m, 7H), 7.51 (dd, *J* = 1.5 Hz, 7. 0 Hz, 2H), 7.64-7.74 (m, 5H), 7.97 (s, 1H), 8.04 (s, 1H); ^{13}**C-NMR** ($CDCl_3$, 125 MHz) δ 29.8, 39.4, 52.0, 86.0, 93.5, 116.3, 119.7, 122.3, 124.8, 125.8, 126.3, 127.3, 127.4, 127.5, 128.4, 128.5, 128.6, 128.7, 130.4, 131.7, 133.7, 135.1, 143.6, 167.9; **HRMS** (ESI) m/z calculated for $C_{28}H_{24}NO$ $[M+H]^+$: 390.1858, found : 390.1852.



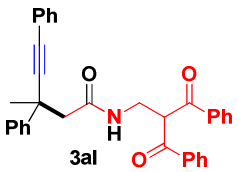
3aj

(**3aj**) Starting materials: 2,4-diphenylbut-3-yn-2-ol **1a** (222.2 mg, 1.0 mmol), ((2-azidoallyl)oxy)benzene **2j** (87.5 mg, 0.5 mmol), BF_3•Et_2O (18 μL, 0.15 mmol), and CH_2Cl_2 (3.0 mL); Reaction temperature = 25 °C; Reaction time = 10 min under N_2 atmosphere; Column chromatography on silica gel = 20 : 1 Hexane : Ethylacetate; The product **3aj**: Yield = 140.7 mg, 76%; Colorless oil; **^{1}H-NMR** (500 MHz, $CDCl_3$) δ 1.66 (s, 3H), 3.64 (AB, *J* = 5.5 Hz, 1H), 3.87 (AB, *J* = 7.0 Hz, 1H), 4.47 (AB, *J* = 15.0 Hz, 2H), 6.78 (d, *J* = 8.0 Hz, 2H), 6.89 (s, 1H), 6.98 (t, *J* = 7.5 Hz, 1H), 7.22 (t, *J* = 8.0 Hz, 2H), 7.28 (d, *J* = 7.5 Hz, 1H), 7.30-7.37 (m, 5H), 7.40-7.45 (m, 2H), 7.55 (d, *J* = 7.5 Hz, 2H); **^{13}C-NMR** ($CDCl_3$, 125 MHz) δ 26.7, 42.0, 49.4, 67.2, 85.1, 92.0, 114.5, 122.0, 122.8, 126.2, 127.2, 128.24, 128.29, 128.5, 129.7, 131.6, 142.3, 157.1, 168.2; **HRMS** (ESI) m/z calculated for $C_{25}H_{24}NO_2$ $[M+H]^+$: 370.1807, found : 370.1802.



3ak

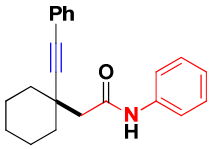
(**3ak**) Starting materials: 2,4-diphenylbut-3-yn-2-ol **1a** (222.2 mg, 1.0 mmol), 2-azidonon-1-ene **2k** (83.6 mg, 0.5 mmol), BF_3•Et_2O (18 μL, 0.15 mmol), and CH_2Cl_2 (3.0 mL); Reaction temperature = -80 °C; Reaction time = 10 min under N_2 atmosphere; Column chromatography on silica gel = 20 : 1 Hexane : Ethylacetate; The product **3ak**: Yield = 111.7 mg, 62%; Colorless oil; **^{1}H-NMR** (500 MHz, $CDCl_3$) δ 0.81 (t, *J* = 7.0 Hz, 3H), 1.08-1.15 (m, 5H), 1.22-1.30 (m, 5H), 1.76 (s, 3H), 2.84 (AB, *J* = 14.5 Hz, 2H), 3.08-3.21 (m, 2H), 5.95 (s, 1H), 7.27 (d, *J* = 7.0 Hz, 1H), 7.32-7.40 (m, 5H), 7.45-7.50 (m, 2H), 7.63 (d, *J* = 7.5 Hz, 2H); **^{13}C-NMR** ($CDCl_3$, 100 MHz) δ 13.9, 22.4, 26.5, 29.3, 29.6, 30.3, 31.4, 39.2, 39.4, 50.6, 85.3, 93.8, 122.7, 125.8, 127.0, 128.3, 128.6, 131.5, 144.0, 169.4, 198.7; **HRMS** (ESI) m/z calculated for $C_{25}H_{32}NO$ $[M+H]^+$: 362.2484, found : 362.2483.



3al

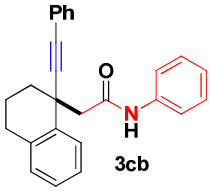
(**3al**) Starting materials: 2,4-diphenylbut-3-yn-2-ol **1a** (222.2 mg, 1.0 mmol), 2-(2-azidoallyl)-1,3-diphenylpropane-1,3-dione **2l** (152.6 mg, 0.5 mmol), BF_3•Et_2O (18 μL, 0.15 mmol), and CH_2Cl_2 (3.0 mL); Reaction temperature = -80 °C; Reaction time = 2 h under N_2 atmosphere; Column chromatography on silica gel = 15 : 1 Hexane : Ethylacetate; The product **3al**: Yield = 187.8 mg, 75%; White solid, m.p. 140-141 °C; **^{1}H-NMR** (500 MHz, $CDCl_3$) δ 1.76 (s, 3H), 3.07-3.27 (m, 4H), 5.77 (t, *J* = 6.5 Hz, 1H), 7.18-7.27 (m, 4H), 7.29-7.39 (m, 8H), 7.50 (q, *J* = 2.5 Hz, 7.5 Hz, 2H), 7.58 (d, *J* = 7.5 Hz, 2H), 7.85 (d, *J* = 8.0 Hz, 4H); **^{13}C-NMR** ($CDCl_3$, 125 MHz) δ 30.2, 38.8, 42.8, 51.3, 55.5, 84.7, 93.2, 123.0, 125.7, 126.8, 127.9, 128.1, 128.43, 128.49, 128.7,

131.5, 133.4, 135.31, 135.38, 144.2, 195.5, 195.6, 204.4; **HRMS** (ESI) m/z calculated for $C_{34}H_{30}NO_3$ $[M+H]^+$: 500.2226, found : 500.2215.



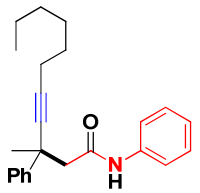
3bb

(**3bb**) Starting materials: 1-(phenylethynyl)cyclohexanol **1b** (200.2 mg, 1.0 mmol), 1-azidovinyl-benzene **2b** (72.5 mg, 0.5 mmol), BF_3•Et_2O (18 μL, 0.15 mmol), and CH_2Cl_2 (3.0 mL); Reaction temperature = 25 ^oC; Reaction time = 10 min under N_2 atmosphere; Column chromatography on silica gel = 20 : 1 Hexane : Ethylacetate; The product **3bb**: Yield = 114.2 mg, 72%; White solid, m.p. 95-96 ^oC; **^{1}H-NMR** (500 MHz, $CDCl_3$) δ 1.15-1.24 (m, 1H), 1.37-1.46 (m, 2H), 1.61-1.75 (m, 5H), 1.95 (d, *J* = 12.5 Hz, 2H), 2.57 (s, 2H), 7.06 (t, *J* = 7.0 Hz, 1H), 7.26 (t, *J* = 7.5 Hz, 2H), 7.29-7.35 (m, 3H), 7.43 (dd, *J* = 2.0 Hz, 5.5 Hz, 2H), 7.46 (d, *J* = 8.0 Hz, 2H), 8.28 (s, 1H); **^{13}C-NMR** ($CDCl_3$, 125 MHz) δ 22.8, 25.6, 36.1, 37.6, 51.0, 85.5, 94.0, 119.6, 122.7, 123.9, 128.2, 128.3, 128.8, 131.5, 137.9, 168.5; **HRMS** (ESI) m/z calculated for $C_{22}H_{24}NO$ $[M+H]^+$: 318.1858, found : 318.1845.



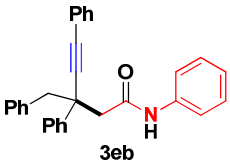
3cb

(**3cb**) Starting materials: 1-(phenylethynyl)-1,2,3,4-tetrahydronaphthalen-1-ol **1c** (248.3 mg, 1.0 mmol), 1-azidovinyl-benzene **2b** (72.5 mg, 0.5 mmol), BF_3•Et_2O (18 μL, 0.15 mmol), and CH_2Cl_2 (3.0 mL); Reaction temperature = 25 ^oC; Reaction time = 10 min under N_2 atmosphere; Column chromatography on silica gel = 20 : 1 Hexane : Ethylacetate; The product **3cb**: Yield = 144.3 mg, 79%; White solid, m.p. 110-111 ^oC; **^{1}H-NMR** (500 MHz, $CDCl_3$) δ 1.91-2.00 (m, 2H), 2.19-2.31 (m, 2H), 2.81-2.90 (m, 3H), 3.07 (d, *J* = 14.0 Hz, 1H), 7.07 (t, *J* = 7.5 Hz, 1H), 7.13 (d, *J* = 7.5 Hz, 1H), 7.17-7.33 (m, 7H), 7.37-7.44 (m, 4H), 7.63 (d, *J* = 7.5 Hz, 1H), 7.97 (s, 1H); **^{13}C-NMR** ($CDCl_3$, 125 MHz) δ 19.0, 29.3, 34.6, 37.9, 50.9, 83.9, 95.4, 119.6, 122.5, 124.1, 126.4, 127.1, 127.8, 128.4, 128.9, 129.8, 131.6, 136.0, 137.8, 139.1, 167.8; **HRMS** (ESI) m/z calculated for $C_{26}H_{24}NO$ $[M+H]^+$: 366.1858, found : 366.1846.



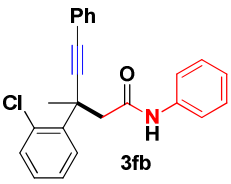
3db

(**3db**) Starting materials: 2-phenyldec-3-yn-2-ol **1d** (230.3 mg, 1.0 mmol), 1-azidovinyl-benzene **2b** (72.5 mg, 0.5 mmol), BF_3•Et_2O (18 μL, 0.15 mmol), and CH_2Cl_2 (3.0 mL); Reaction temperature = 25 ^oC; Reaction time = 10 min under N_2 atmosphere; Column chromatography on silica gel = 20 : 1 Hexane : Ethylacetate; The product **3db**: Yield = 137.2 mg, 79%; Colorless oil; **^{1}H-NMR** (500 MHz, $CDCl_3$) δ 0.95 (t, *J* = 7.0 Hz, 3H), 1.29-1.40 (m, 4H), 1.43-1.54 (m, 2H), 1.60-1.70 (m, 2H), 1.76 (s, 3H), 2.41 (t, *J* = 7.0 Hz, 2H), 2.94 (AB, *J* = 14.0 Hz, 2H), 7.12 (t, *J* = 7.0 Hz, 1H), 7.32 (t, *J* = 7.5, 3H), 7.41 (t, *J* = 7.5 Hz, 4H), 7.67 (d, *J* = 8.0 Hz, 2H), 8.05 (s, 1H); **^{13}C-NMR** ($CDCl_3$, 125 MHz) δ 13.9, 18.7, 22.3, 28.5, 28.8, 30.4, 31.1, 38.7, 51.7, 84.4, 86.2, 119.7, 123.9, 125.7, 126.9, 128.4, 128.7, 137.7, 144.2, 168.0; **FT-IR** (neat) ν = 3415, 3249, 3192, 3133, 3079, 3034, 2929, 1653, 1595, 1549, 1490, 1443, 1374, 1357, 1246, 1154, 1077, 1027, 909, 761, 695 cm^{-1}; **HRMS** (ESI) m/z calculated for $C_{24}H_{30}NO$ $[M+H]^+$: 348.2283, found : 348.2264.



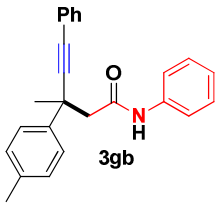
3eb

(**3eb**) Starting materials: 1,2,4-triphenylbut-3-yn-2-ol **1e** (298.3 mg, 1.0 mmol), 1-azidovinyl-benzene **2b** (72.5 mg, 0.5 mmol), BF_3•Et_2O (18 μL, 0.15 mmol), and CH_2Cl_2 (3.0 mL); Reaction temperature = 25 ^oC; Reaction time = 10 min under N_2 atmosphere; Column chromatography on silica gel = 20 : 1 Hexane : Ethylacetate; The product **3eb**: Yield = 164.1 mg, 79%; White solid, m.p. 138-139 ^oC; **^{1}H-NMR** (500 MHz, $CDCl_3$) δ 3.14 (AB, *J* = 14.5 Hz, 2H), 3.26 (AB, *J* = 13.0 Hz, 2H), 7.00 (t, *J* = 7.0 Hz, 1H), 7.03-7.07 (m, 2H), 7.15-7.23 (m, 7H), 7.27 (d, *J* = 7.5 Hz, 1H), 7.31-7.35 (m, 5H), 7.45 (dd, *J* = 1.5 Hz, 7.5 Hz, 2H), 7.57 (d, *J* = 7.5 Hz, 2H), 7.84 (s, 1H); **^{13}C-NMR** ($CDCl_3$, 125 MHz) δ 45.1, 48.9, 49.8, 88.8, 91.7, 119.7, 122.2, 124.0, 126.7, 127.4, 127.5, 128.4, 128.5, 128.6, 128.7, 130.8, 131.5, 136.1, 137.6, 141.1, 167.8; **HRMS** (ESI) m/z calculated for $C_{30}H_{26}NO$ $[M+H]^+$: 416.1970, found : 416.1982.

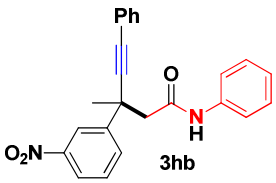


3fb

(**3fb**) Starting materials: 2-(2-chlorophenyl)-4-phenylbut-3-yn-2-ol **1f** (256.7 mg, 1.0 mmol), 1-azidovinyl-benzene **2b** (72.5 mg, 0.5 mmol), BF_3•Et_2O (18 μL, 0.15 mmol), and CH_2Cl_2 (3.0 mL); Reaction temperature = 25 ^oC; Reaction time = 10 min under N_2 atmosphere; Column chromatography on silica gel = 20 : 1 Hexane : Ethylacetate; The product **3fb**: Yield = 151.3 mg, 81%; White solid, m.p. 94-95 ^oC; ^{1}**H-NMR** (500 MHz, $CDCl_3$) δ 2.01 (s, 3H), 3.28 (AB, *J* = 14.0 Hz, 2H), 7.05 (t, *J* = 7.5 Hz, 1H), 7.21-7.27 (m, 4H), 7.29-7.34 (m, 5H), 7.41-7.48 (m, 3H), 7.71 (s, 1H), 7.80 (d, *J* = 7.5 Hz, 1H); ^{13}**C-NMR** ($CDCl_3$, 125 MHz) δ 27.6, 39.6, 48.0, 85.6, 93.1, 119.8, 122.6, 124.1, 127.2, 128.40, 128.47, 128.8, 131.6, 132.1, 133.0, 137.6, 139.3, 167.8; **HRMS** (ESI) m/z calculated for $C_{24}H_{21}ClNO$ $[M+H]^+$: 374.1204, found : 375.1221.

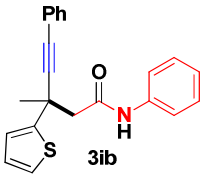


(**3gb**) Starting materials: 4-phenyl-2-(*p*-tolyl)but-3-yn-2-ol **1g** (236.3 mg, 1.0 mmol), 1-azidovinyl-benzene **2b** (72.5 mg, 0.5 mmol), BF_3•Et_2O (18 μL, 0.15 mmol), and CH_2Cl_2 (3.0 mL); Reaction temperature = 25 ^oC; Reaction time = 10 min under N_2 atmosphere; Column chromatography on silica gel = 20 : 1 Hexane : Ethylacetate; The product **3gb**: Yield = 134.2 mg, 76%; White solid, m.p. 105-106 ^oC; ^{1}**H-NMR** (500 MHz, $CDCl_3$) δ 1.79 (s, 3H), 2.34 (s, 3H), 2.97 (AB, *J* = 14.5 Hz, 2H), 7.04 (t, *J* = 7.5 Hz, 1H), 7.19 (d, *J* = 8.0 Hz, 2H), 7.24 (d, *J* = 8.0 Hz, 2H), 7.28-7.37 (m, 5H), 7.48 (dd, *J* = 2.0 Hz, 7.0 Hz, 2H), 7.54 (d, *J* = 8.0 Hz, 2H), 7.81 (s, 1H); ^{13}**C-NMR** ($CDCl_3$, 125 MHz) δ 20.9, 30.0, 39.0, 51.9, 85.7, 93.7, 119.7, 122.4, 124.0, 125.7, 128.4, 128.5, 128.8, 129.4, 131.6, 136.9, 137.7, 140.7, 167.8; **FT-IR** (KBr) ν = 3408, 3138, 3056, 3024, 2974, 2925, 1660, 1599, 1543, 1497, 1441, 1383, 1339, 1257, 1156, 1021, 913, 816, 754, 691 cm^{-1}; **HRMS** (ESI) m/z calculated for $C_{25}H_{24}NO$ $[M+H]^+$: 354.1813, found : 354.1835.



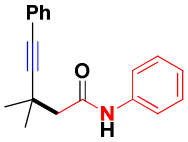
(**3hb**) Starting materials: 2-(3-nitrophenyl)-4-phenylbut-3-yn-2-ol **1h** (267.2 mg, 1.0 mmol), 1-azidovinyl-benzene **2b** (72.5 mg, 0.5 mmol), BF_3•Et_2O (18 μL, 0.15 mmol), and CH_2Cl_2 (3.0 mL); Reaction temperature = 25 ^oC; Reaction time = 10 min under N_2 atmosphere; Column chromatography on silica gel = 20 : 1 Hexane : Ethylacetate; The product **3hb**: Yield = 142.2 mg, 74%; White solid, m.p. 117-118 ^oC; ^{1}**H-NMR** (500 MHz, $CDCl_3$) δ 1.87 (s, 3H), 3.00 (s, 2H), 7.07 (t, *J* = 7.5 Hz, 1H), 7.25 (t, *J* = 7.5

Hz, 2H), 7.31-7.42 (m, 5H), 7.50 (d, J = 6.5 Hz, 2H), 7.52 (t, J = 7.5 Hz, 1H), 7.90 (s, 1H), 7.98 (d, J = 8.0 Hz, 1H), 8.14 (d, J = 8.0 Hz, 1H), 8.58 (s, 1H); **^{13}C-NMR** ($CDCl_3$, 125 MHz) δ 29.9, 39.7, 51.3, 86.9, 92.0, 119.8, 121.3, 121.8, 122.3, 124.4, 128.6, 128.9, 129.6, 131.7, 132.0, 137.4, 146.0, 148.4, 166.9; **HRMS** (ESI) m/z calculated for $C_{24}H_{21}N_2O_3$ $[M+H]^+$: 385.1507, found : 385.1525.



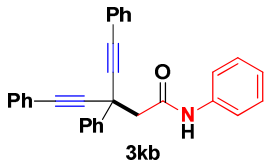
3ib

(**3ib**) Starting materials: 4-phenyl-2-(thiophen-2-yl)but-3-yn-2-ol **1i** (228.3 mg, 1.0 mmol), 1-azidovinyl-benzene **2b** (72.5 mg, 0.5 mmol), BF_3•Et_2O (18 μL, 0.15 mmol), and CH_2Cl_2 (3.0 mL); Reaction temperature = 25 ^oC; Reaction time = 10 min under N_2 atmosphere; Column chromatography on silica gel = 20 : 1 Hexane : Ethylacetate; The product **3ib**: Yield = 120.8 mg, 70%; Colorless oil; **^{1}H-NMR** (400 MHz, $CDCl_3$) δ 1.88 (s, 3H), 3.00 (d, J = 14.5 Hz, 2H), 6.97 (t, J = 5.0 Hz, 1H), 7.07 (t, J = 9.0 Hz, 1H), 7.15 (d, J = 3.0 Hz, 1H), 7.21-7.29 (m, 3H), 7.31-7.40 (m, 5H), 7.47 (d, J = 7.0 Hz, 2H), 7.79 (s, 1H); **^{13}C-NMR** ($CDCl_3$, 100 MHz) δ 30.8, 37.0, 52.9, 84.8, 92.9, 119.8, 123.9, 124.2, 124.4, 126.9, 128.4, 128.6, 128.9, 131.7, 137.7, 149.4, 167.2; **FT-IR** (neat) ν = 3310, 3199, 3138, 3059, 2975, 2927, 1663, 1599, 1542, 1496, 1441, 1376, 1340, 1234, 1199, 1027, 903, 848, 754, 691 cm^{-1}; **HRMS** (ESI) m/z calculated for $C_{22}H_{20}NOS$ $[M+H]^+$: 346.1221, found : 346.1235.



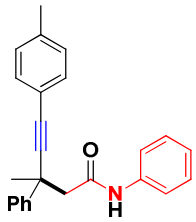
3jb

(**3jb**) Starting materials: 2-methyl-4-phenylbut-3-yn-2-ol **1j** (160.2 mg, 1.0 mmol), 1-azidovinyl-benzene **2b** (72.5 mg, 0.5 mmol), BF_3•Et_2O (18 μL, 0.15 mmol), and CH_2Cl_2 (3.0 mL); Reaction temperature = 25 ^oC; Reaction time = 10 min under N_2 atmosphere; Column chromatography on silica gel = 20 : 1 Hexane : Ethylacetate; The product **3jb**: Yield = 112.3 mg, 81%; White solid, m.p. 120-121 ^oC; **^{1}H-NMR** (500 MHz, $CDCl_3$) δ 1.45 (s, 6H), 2.58 (s, 2H), 7.08 (t, J = 7.5 Hz, 1H), 7.25-7.33 (m, 2H), 7.30-7.34 (m, 3H), 7.40-7.42 (m, 2H), 7.46 (d, J = 8.0 Hz, 2H), 8.18 (s, 1H); **^{13}C-NMR** ($CDCl_3$, 125 MHz) δ 29.3, 30.7, 51.1, 82.7, 95.5, 119.6, 122.5, 124.0, 128.3, 128.4, 128.9, 131.5, 137.8, 168.6; **HRMS** (ESI) m/z calculated for $C_{19}H_{20}NO$ $[M+H]^+$: 278.1500, found : 278.1503.



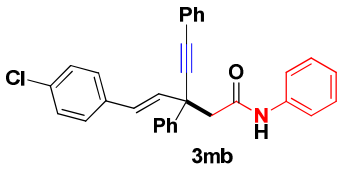
3kb

(**3kb**) Starting materials: 1,3,5-triphenylpenta-1,4-diyn-3-ol **1k** (308.3 mg, 1.0 mmol), 1-azidovinyl-benzene **2b** (72.5 mg, 0.5 mmol), BF_3•Et_2O (18 μL, 0.15 mmol), and CH_2Cl_2 (3.0 mL); Reaction temperature = 25 ^oC; Reaction time = 10 min under N_2 atmosphere; Column chromatography on silica gel = 20 : 1 Hexane : Ethylacetate; The product **3kb**: Yield = 136.1 mg, 64%; White solid, m.p. 131-132 ^oC; **^{1}H-NMR** (500 MHz, $CDCl_3$) δ 3.20 (s, 2H), 7.07 (t, *J* = 7.5 Hz, 1H), 7.27 (t, *J* = 8.5 Hz, 2H), 7.29-7.38 (m, 7H), 7.42 (d, *J* = 7.5 Hz, 4H), 7.50 (d, *J* = 6.5 Hz, 4H), 7.86 (d, *J* = 8.0 Hz, 2H), 8.13 (s, 1H); **^{13}C-NMR** ($CDCl_3$, 125 MHz) δ 39.0, 53.8, 85.4, 88.3, 119.8, 121.9, 124.2, 126.2, 128.0, 128.3, 128.80, 128.83, 128.9, 131.8, 137.7, 140.7, 166.1; **HRMS** (ESI) m/z calculated for $C_{31}H_{24}NO$ $[M+H]^+$: 426.1858, found : 426.1845.



3lb

(**3lb**) Starting materials: 2-phenyl-4-(*p*-tolyl)but-3-yn-2-ol **1l** (236.3 mg, 1.0 mmol), 1-azidovinyl-benzene **2b** (72.5 mg, 0.5 mmol), BF_3•Et_2O (18 μL, 0.15 mmol), and CH_2Cl_2 (3.0 mL); Reaction temperature = 25 ^oC; Reaction time = 10 min under N_2 atmosphere; Column chromatography on silica gel = 20 : 1 Hexane : Ethylacetate; The product **3lb**: Yield = 132.5 mg, 75%; White solid, m.p. 94-95 ^oC; **^{1}H-NMR** (500 MHz, $CDCl_3$) δ 1.80 (s, 3H), 2.37 (s, 3H), 2.98 (AB, *J* = 14.0 Hz, 2H), 7.04 (t, *J* = 7.5 Hz, 1H), 7.15 (d, *J* = 8.5 Hz, 2H), 7.23 (t, *J* = 7.5 Hz, 2H), 7.30 (t, *J* = 8.0 Hz, 3H), 7.35-7.42 (m, 4H), 7.67 (d, *J* = 7.0 Hz, 2H), 7.86 (s, 1H); **^{13}C-NMR** ($CDCl_3$, 125 MHz) δ 21.4, 30.0, 39.4, 51.9, 86.1, 92.7, 119.7, 124.0, 125.8, 127.2, 128.7, 128.8, 129.2, 131.5, 137.7, 138.7, 143.8, 167.8; **HRMS** (ESI) m/z calculated for $C_{25}H_{24}NO$ $[M+H]^+$: 354.1858, found : 354.1849.



3mb

(**3mb**) Starting materials: (*E*)-1-(4-chlorophenyl)-3,5-diphenylpent-1-en-4-yn-3-ol **1m** (344.8 mg, 1.0 mmol), 1-azidovinyl-benzene **2b** (72.5 mg, 0.5 mmol), BF_3•Et_2O (18 μL, 0.15 mmol), and CH_2Cl_2 (3.0 mL); Reaction temperature = 25

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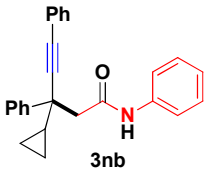
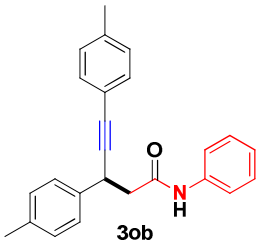
^{o}C; Reaction time = 10 min under N_2 atmosphere; Column chromatography on silica gel = 20 : 1 Hexane : Ethylacetate; The product **3mb**: Yield = 184.7 mg, 80%; White solid, m.p. 127-128 ^{o}C; **^{1}H-NMR** (500 MHz, $CDCl_3$) δ 2.86 (AB, *J* = 7.5 Hz, 1H), 2.93 (AB, *J* = 7.5 Hz, 1H), 4.74 (q, *J* = 7.5 Hz, 9.0 Hz, 1H), 6.54 (d, *J* = 9.5 Hz, 1H), 7.04 (t, *J* = 7.0 Hz, 1H), 7.22 (t, *J* = 7.5 Hz, 2H), 7.25-7.40 (m, 13H), 7.51 (t, *J* = 3.5 Hz, 2H), 7.62 (d, *J* = 7.5 Hz, 2H); **^{13}C-NMR** ($CDCl_3$, 125 MHz) δ 43.4, 43.9, 86.0, 97.0, 120.1, 122.7, 124.4, 124.8, 126.2, 128.1, 128.4, 128.6, 128.7, 128.8, 128.9, 131.6, 132.6, 137.3, 137.5, 137.9, 140.9, 168.5; **HRMS** (ESI) m/z calculated for $C_{31}H_{25}ClNO$ $[M+H]^+$: 462.1580, found : 462.1597.



3nb

(**3nb**) Starting materials: 1-cyclopropyl-1,3-diphenylprop-2-yn-1-ol **1n** (248.3 mg, 1.0 mmol), 1-azidovinyl-benzene **2b** (72.5 mg, 0.5 mmol), BF_3•Et_2O (18 μL, 0.15 mmol), and CH_2Cl_2 (3.0 mL); Reaction temperature = 25 ^{o}C; Reaction time = 10 min under N_2 atmosphere; Column chromatography on silica gel = 20 : 1 Hexane : Ethylacetate; The product **3nb**: Yield = 164.4 mg, 90%; White solid, m.p. 87-88 ^{o}C; **^{1}H-NMR** (500 MHz, $CDCl_3$) δ 0.42-0.50 (m, 1H), 0.50-0.58 (m, 1H), 0.61-0.70 (m, 1H), 0.76-0.83 (m, 1H), 1.39-1.80 (m, 1H), 3.15 (AB, *J* = 14.5 Hz, 2H), 7.03 (t, *J* = 7.0 Hz, 1H), 7.21 (t, *J* = 7.5 Hz, 2H), 7.26 (d, *J* = 9.0 Hz, 2H), 7.28-7.36 (m, 4H), 7.40 (t, *J* = 7.5Hz, 2H), 7.46 (dd, *J* = 2.0 Hz, 7.5 Hz, 2H), 7.68 (s, 1H), 7.70 (d, *J* = 8.0 Hz, 2H); **^{13}C-NMR** ($CDCl_3$, 125 MHz) δ 2.03, 3.51, 20.8, 45.5, 51.2, 87.4, 89.0, 119.6, 122.1, 124.0, 126.4, 127.3, 128.4, 128.7, 128.8, 131.7, 137.7, 143.1, 167.7; **HRMS** (ESI) m/z calculated for $C_{26}H_{24}NO$ $[M+H]^+$: 366.1813, found : 366.1824.



3ob

(**3ob**) Starting materials: 1,3-di-*p*-tolylprop-2-yn-1-ol **1o** (236.3 mg, 1.0 mmol), 1-azidovinyl-benzene **2b** (72.5 mg, 0.5 mmol), BF_3•Et_2O (18 μL, 0.15 mmol), and CH_2Cl_2 (3.0 mL); Reaction temperature = 25 ^{o}C; Reaction time = 10 min under N_2 atmosphere; Column chromatography on silica gel = 20 : 1 Hexane : Ethylacetate; The product **3ob**: Yield = 84.8 mg, 48%; White solid, m.p. 98-99 ^{o}C; **^{1}H-NMR** (500 MHz, $CDCl_3$) δ 2.33 (s, 6H), 2.76-2.83 (m, 1H), 2.83-2.90 (m, 1H), 4.42 (t, *J* = 6.5 Hz, 1H), 7.08 (d, *J* = 8.0 Hz, 3H), 7.15 (d, *J* = 8.0 Hz, 2H), 7.25-7.32 (m, 4H), 7.36 (d, *J* = 8.0 Hz, 2H), 7.43 (d, *J* = 8.0 Hz, 2H), 7.50 (s, 1H);

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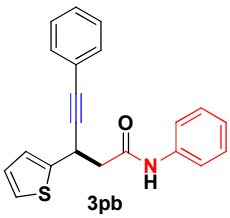
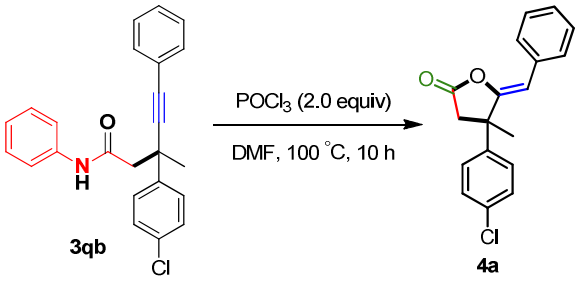
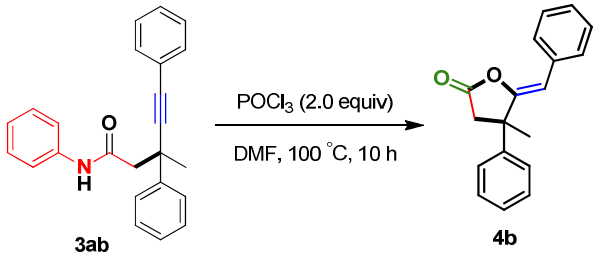
^{13}C-NMR ($CDCl_3$, 125 MHz) δ 21.0, 21.4, 34.9, 46.8, 84.6, 89.2, 119.8, 120.0, 124.3, 127.2, 127.8, 128.9, 129.0, 129.4, 129.5, 131.5, 136.9, 137.3, 137.6, 138.2, 168.4; **HRMS** (ESI) m/z calculated for $C_{25}H_{24}NO$ $[M+H]^+$: 354.1813, found : 354.1822.



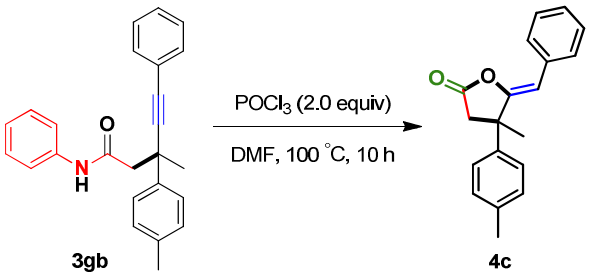
(**3pb**) Starting materials: 3-phenyl-1-(thiophen-2-yl)prop-2-yn-1-ol **1p** (214.2 mg, 1.0 mmol), 1-azidovinyl-benzene **2b** (72.5 mg, 0.5 mmol), BF_3•Et_2O (18 μL, 0.15 mmol), and CH_2Cl_2 (3.0 mL); Reaction temperature = 25 °C; Reaction time = 10 min under N_2 atmosphere; Column chromatography on silica gel = 20 : 1 Hexane : Ethylacetate; The product **3pb**: Yield = 66.2 mg, 40%; White solid, m.p. 113-114 °C; **^{1}H-NMR** (500 MHz, $CDCl_3$) δ 2.79-2.93 (m, 2H), 4.72 (t, *J* = 7.0 Hz, 1H), 6.86 (t, *J* = 5.0 Hz, 1H), 6.98-7.04 (m, 2H), 7.12 (d, *J* = 5.0 Hz, 1H), 7.18-7.22 (m, 5H), 7.33 (d, *J* = 8.0 Hz, 2H), 7.38 (d, *J* = 7.5 Hz, 2H), 7.50 (s, 1H); **^{13}C-NMR** ($CDCl_3$, 125 MHz) δ 30.5, 46.7, 84.0, 89.2, 120.1, 122.6, 124.4, 125.0, 126.9, 128.2, 128.3, 128.9, 131.6, 137.5, 143.6, 167.8; **HRMS** (ESI) m/z calculated for $C_{22}H_{18}NOS$ $[M+H]^+$: 346.1221, found : 346.1234.



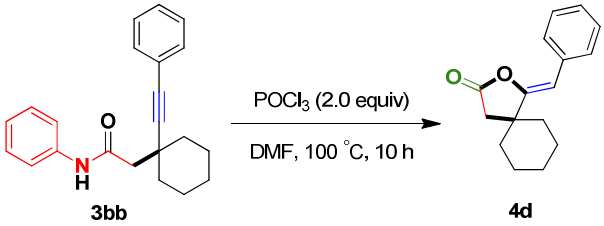
(**4a**) Starting materials: 3-(4-chlorophenyl)-3-methyl-*N*,5-diphenylpent-4-ynamide **3qb** (112.1 mg, 0.3 mmol), $POCl_3$ (92.0 mg, 0.6 mmol), and dry DMF (3.0 mL); Reaction temperature = 100 °C; Reaction time = 10 h under air atmosphere; Column chromatography on silica gel = 25 : 1 Hexane : Ethylacetate; The product **4a**: Yield = 75.2 mg, 84%; Colorless oil; **^{1}H-NMR** (500 MHz, $CDCl_3$) δ 1.76 (s, 3H), 2.84 (AB, *J* = 17.5 Hz, 2H), 5.54 (s, 1H), 7.22 (t, *J* = 7.0 Hz, 1H), 7.28-7.37 (m, 6H), 7.56 (d, *J* = 8.0 Hz, 2H); **^{13}C-NMR** ($CDCl_3$, 125 MHz) δ 26.9, 44.2, 47.5, 105.6, 127.1, 127.4, 128.4, 128.5, 128.8, 133.2, 133.3, 142.5, 156.0, 172.0; **FT-IR** (neat) ν = 3443, 2924, 1805, 1673, 1636, 1560, 1491, 1384, 1154, 1130, 1038, 954, 827, 752, 617 cm^{-1}; **HRMS** (ESI) m/z calculated for $C_{18}H_{16}ClO_2$ $[M+H]^+$: 397.0665, found : 397.0675.



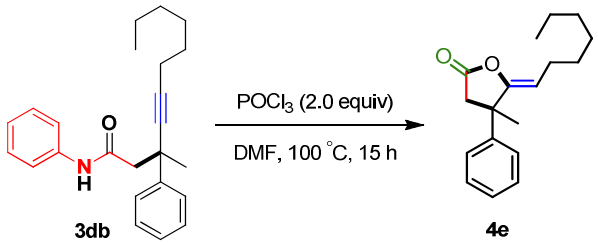
(**4b**) Starting materials: 3-methyl-*N*,3,5-triphenylpent-4-ynamide **3ab** (101.8 mg, 0.3 mmol), $POCl_3$ (92.0 mg, 0.6 mmol), and dry DMF (3.0 mL); Reaction temperature = 100 °C; Reaction time = 10 h under air atmosphere; Column chromatography on silica gel = 25 : 1 Hexane : Ethylacetate; The product **4b**: Yield = 64.0 mg, 81%; Colorless oil; **^{1}H-NMR** (500 MHz, $CDCl_3$) δ 1.79 (s, 3H), 2.83 (AB, *J* = 18.0 Hz, 2H), 5.46 (s, 1H), 7.21 (t, *J* = 7.5 Hz, 1H), 7.28 (t, *J* = 7.5 Hz, 1H), 7.30-7.38 (m, 4H), 7.42 (d, *J* = 8.0 Hz, 2H), 7.58 (d, *J* = 8.0 Hz, 2H); **^{13}C-NMR** ($CDCl_3$, 125 MHz) δ 27.0, 44.5, 47.9, 105.4, 125.9, 127.0, 127.3, 128.4, 128.5, 128.8, 133.5, 144.0, 156.6, 172.4; **FT-IR** (neat) ν = 3427, 3057, 3025, 2974, 2923, 1807, 1679, 1599, 1494, 1446, 1382, 1181, 1132, 1038, 955, 758, 696 cm^{-1}; **HRMS** (ESI) m/z calculated for $C_{18}H_{17}O_2$ $[M+H]^+$: 265.1184, found: 265.1192.



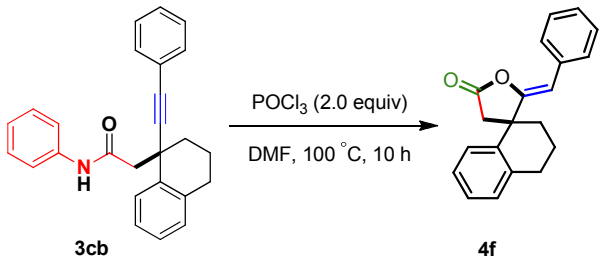
(**4c**) Starting materials: 3-methyl-*N*,5-diphenyl-3-(*p*-tolyl)pent-4-ynamide **3gb** (106.0 mg, 0.3 mmol), $POCl_3$ (92.0 mg, 0.6 mmol), and dry DMF (3.0 mL); Reaction temperature = 100 °C; Reaction time = 10 h under air atmosphere; Column chromatography on silica gel = 25 : 1 Hexane : Ethylacetate; The product **4c**: Yield = 61.9 mg, 74%; Colorless oil; **^{1}H-NMR** (500 MHz, $CDCl_3$) δ 1.76 (s, 3H), 2.33 (s, 3H), 2.90 (AB, *J* = 18.0 Hz, 2H), 5.45 (s, 1H), 7.15 (d, *J* = 8.0 Hz, 2H), 7.20 (t, *J* = 7.5 Hz, 1H), 7.27-7.35 (m, 4H), 7.57 (d, *J* = 7.5 Hz, 2H); **^{13}C-NMR** ($CDCl_3$, 125 MHz) δ 20.8, 27.0, 44.4, 47.6, 105.2, 125.8, 126.9, 128.4, 128.5, 129.4, 133.6, 137.0, 141.01, 156.8, 172.5; **FT-IR** (neat) ν = 3436, 3055, 3025, 2971, 2872, 1811, 1679, 1512, 1493, 1448, 1383, 1181, 1131, 1039, 955, 755, 694 cm^{-1}; **HRMS** (ESI) m/z calculated for $C_{19}H_{19}O_2$ $[M+H]^+$: 279.1340, found : 279.1356.



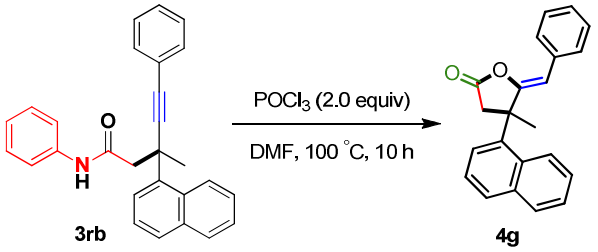
(**4d**) Starting materials: *N*-phenyl-2-(1-(phenylethynyl)cyclohexyl)acetamide **3bb** (95.2 mg, 0.3 mmol), $POCl_3$ (92.0 mg, 0.6 mmol), and dry DMF (3.0 mL); Reaction temperature = 100 °C; Reaction time = 10 h under air atmosphere; Column chromatography on silica gel = 25 : 1 Hexane : Ethylacetate; The product **4d**: Yield = 58.2 mg, 80%; White solid, m.p. 72-73 °C; **^{1}H-NMR** (500 MHz, $CDCl_3$) δ 1.26-1.37 (m, 3H), 1.57-1.66 (m, 2H), 1.72-1.80 (m, 5H), 2.60 (s, 2H), 5.54 (s, 1H), 7.19 (t, *J* = 7.5 Hz, 1H), 7.32 (t, *J* = 7.5 Hz, 2H), 7.56 (d, *J* = 8.0 Hz, 2H); **^{13}C-NMR** ($CDCl_3$, 125 MHz) δ 22.8, 25.2, 36.8, 37.8, 44.3, 102.6, 126.6, 128.40, 128.43, 133.9, 158.5, 173.4; **HRMS** (ESI) m/z calculated for $C_{16}H_{19}O_2$ $[M+H]^+$: 243.1340, found : 243.1334.



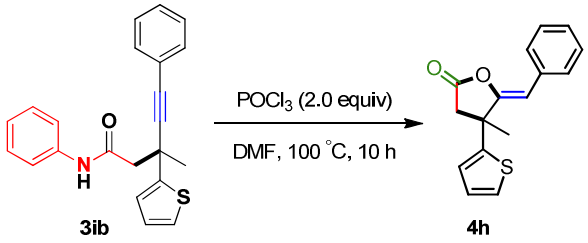
(**4e**) Starting materials: 3-methyl-*N*,3-diphenylundec-4-ynamide **3db** (104.2 mg, 0.3 mmol), $POCl_3$ (92.0 mg, 0.6 mmol), and dry DMF (3.0 mL); Reaction temperature = 100 °C; Reaction temperature = 100 °C; Reaction time = 15 h under air atmosphere; Column chromatography on silica gel = 25 : 1 Hexane : Ethylacetate; The product **4e**: Yield = 59.8 mg, 73%; Colorless oil; **^{1}H-NMR** (500 MHz, $CDCl_3$) δ 0.88 (t, *J* = 8.0 Hz, 3H), 1.26-1.34 (m, 6H), 1.31-1.40 (m, 2H), 1.67 (s, 3H), 2.15-2.25 (m, 2H), 2.87 (AB, *J* = 17.5 Hz, 2H), 4.59 (t, *J* = 7.5 Hz, 1H), 7.26 (t, *J* = 7.0 Hz, 1H), 7.31-7.39 (m, 4H); **^{13}C-NMR** ($CDCl_3$, 125 MHz) δ 14.0, 22.5, 25.1, 27.0, 28.8, 29.3, 31.5, 43.8, 45.3, 46.8, 106.2, 125.3, 125.8, 127.1, 128.6, 144.4, 156.0, 172.9; **HRMS** (ESI) m/z $C_{18}H_{25}O_2$ $[M+H]^+$: 273.1810, found : 273.1816.



(**4f**) Starting materials: *N*-phenyl-2-(1-(phenylethynyl)-1,2,3,4-tetrahydronaphthalen-1-yl)acetamide **3cb** (109.6 mg, 0.3 mmol), $POCl_3$ (92.0 mg, 0.6 mmol), and dry DMF (3.0 mL); Reaction temperature = 100 °C; Reaction time = 10 h under air atmosphere; Column chromatography on silica gel = 25 : 1 Hexane : Ethylacetate; The product **4f**: Yield = 73.1 mg, 84%; White solid, m.p. 93-94 °C; **^{1}H-NMR** (500 MHz, $CDCl_3$) δ 1.73-1.84 (m, 1H), 1.97-2.07 (m, 2H), 2.07-2.15 (m, 1H), 2.81-2.92 (m, 3H), 3.01 (d, *J* = 18.0 Hz, 1H), 5.26 (s, 1H), 7.13 (dd, *J* = 2.0 Hz, 7.5 Hz, 1H), 7.15-7.22 (m, 3H), 7.25-7.33 (m, 3H), 7.52 (d, *J* = 7.0 Hz, 2H); **^{13}C-NMR** ($CDCl_3$, 125 MHz) δ 19.6, 29.4, 37.6, 44.9, 48.0, 105.5, 126.7, 126.8, 127.2, 127.9, 128.3, 128.4, 129.3, 133.7, 137.1, 138.2, 158.4, 172.7; **HRMS** (ESI) m/z calculated for $C_{20}H_{19}O_2$ $[M+H]^+$: 291.1340, found : 291.1342.



(**4g**) Starting materials: 3-methyl-3-(naphthalen-1-yl)-*N*,5-diphenylpent-4-ynamide **3rb** (116.8 mg, 0.3 mmol), $POCl_3$ (92.0 mg, 0.6 mmol), and dry DMF (3.0 mL); Reaction temperature = 100 °C; Reaction time = 10 h under air atmosphere; Column chromatography on silica gel = 25 : 1 Hexane : Ethylacetate; The product **4g**: Yield = 74.4 mg, 79%; White solid, m.p. 89-90 °C; **^{1}H-NMR** (500 MHz, $CDCl_3$) δ 1.91 (s, 3H), 3.03 (AB, *J* = 17.5 Hz, 2H), 5.46 (s, 1H), 7.23 (t, *J* = 7.5 Hz, 1H), 7.34 (t, *J* = 7.5 Hz, 2H), 7.46-7.52 (m, 3H), 7.58 (d, *J* = 7.5 Hz, 2H), 7.80-7.86 (m, 3H), 7.88 (s, 1H); **^{13}C-NMR** ($CDCl_3$, 125 MHz) δ 27.0, 44.3, 48.0, 105.6, 124.43, 124.47, 126.4, 126.6, 127.0, 127.5, 128.0, 128.4, 128.6, 128.9, 132.3, 132.9, 133.6, 141.0, 156.7, 172.5; **HRMS** (ESI) m/z calculated for $C_{22}H_{19}O_2$ $[M+H]^+$: 315.1340, found : 315.1334.

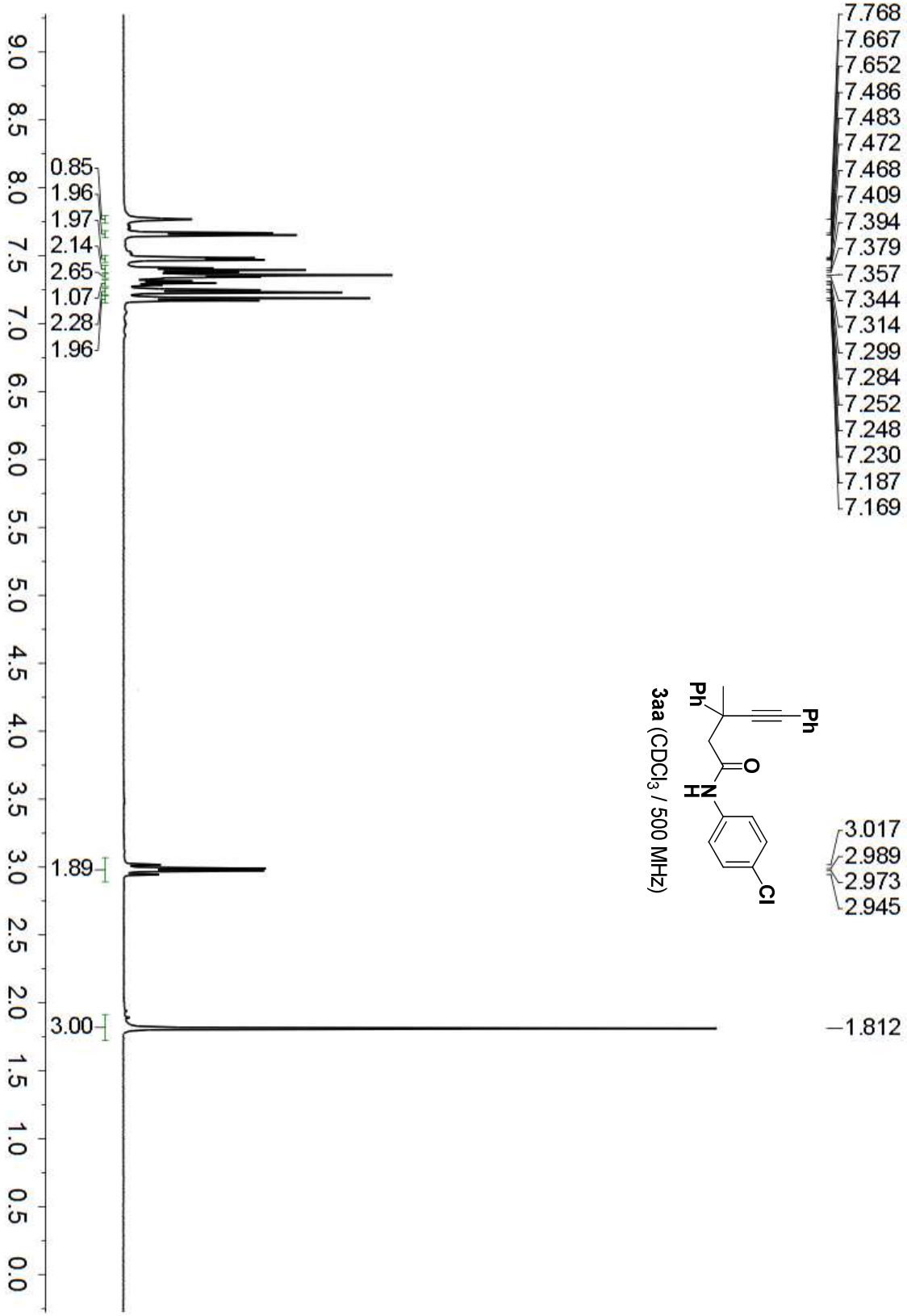


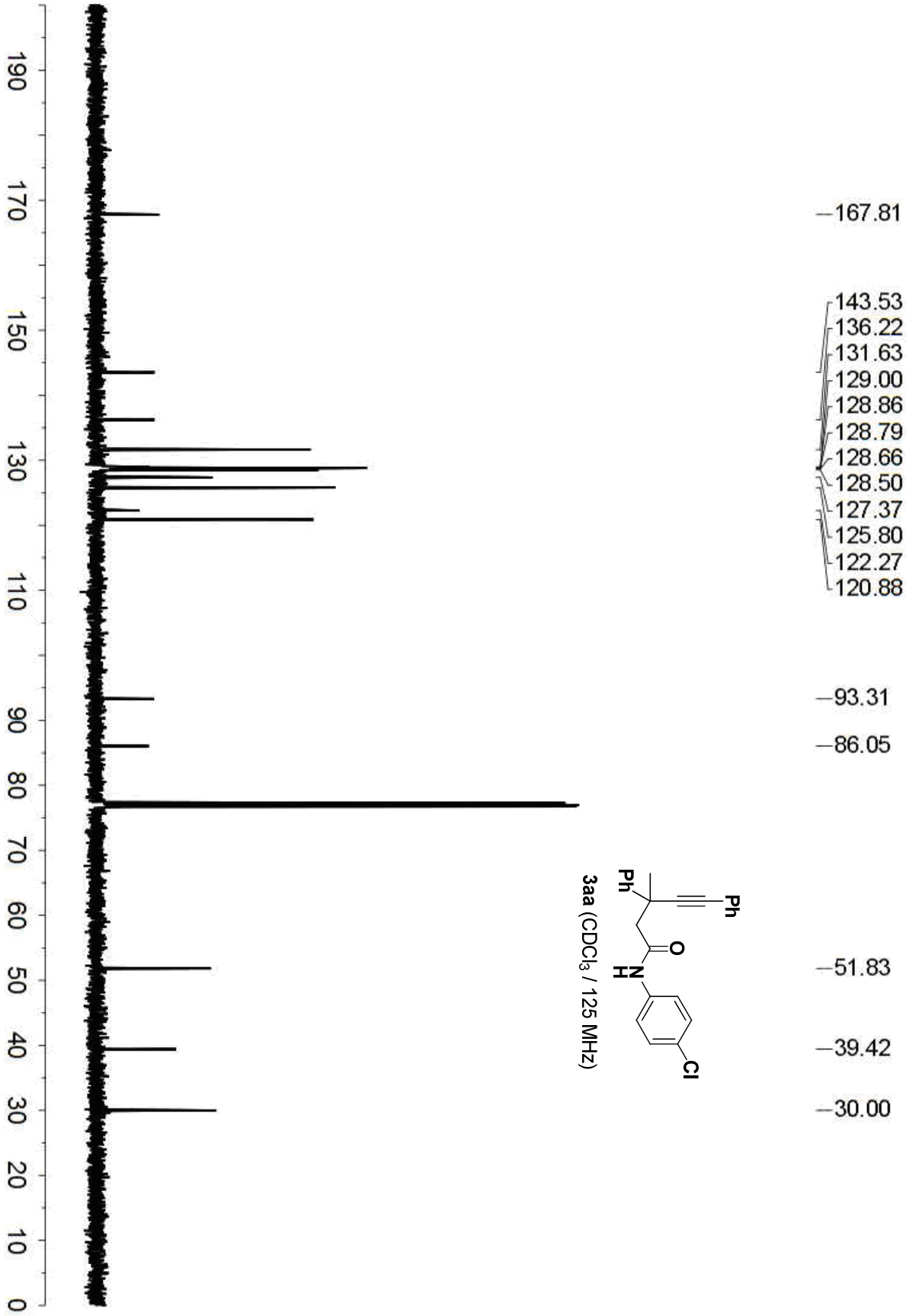
(**4h**) Starting materials: 3-methyl-*N*,5-diphenyl-3-(thiophen-2-yl)pent-4-ynamide **3ib** (103.6 mg, 0.3 mmol), $POCl_3$ (92.0 mg, 0.6 mmol), and dry DMF (3.0 mL); Reaction temperature = 100 °C; Reaction time = 10 h under air atmosphere; Column chromatography on silica gel = 25 : 1 Hexane : Ethylacetate; The product **4h**: Yield = 66.4 mg, 82%; Colorless oil; **^{1}H-NMR** (500

`

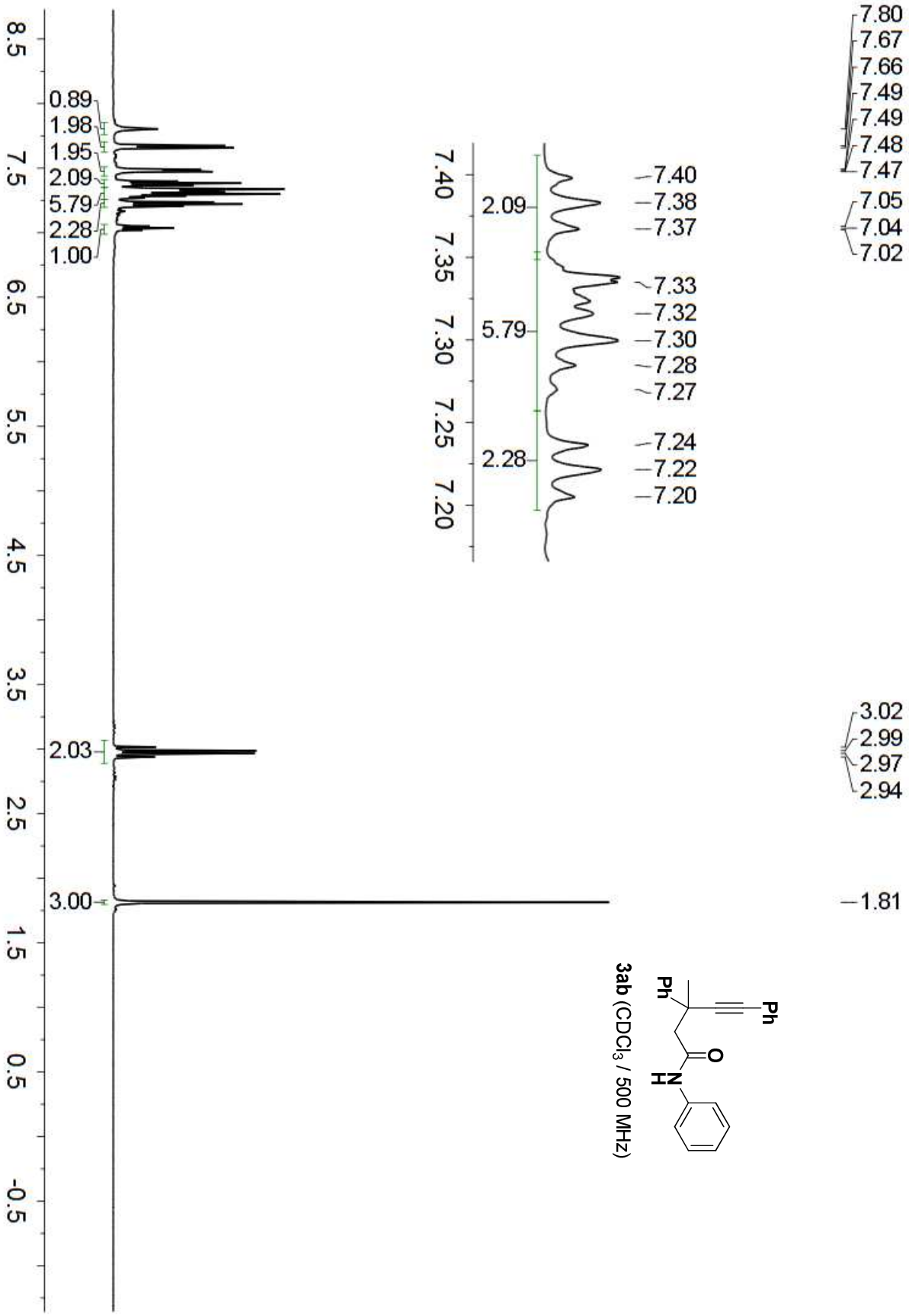
MHz, $CDCl_3$) δ 1.83 (s, 3H), 2.98 (AB, *J* = 17.5 Hz, 2H), 5.59 (s, 1H), 6.95 (t, *J* = 5.0 Hz, 1H), 7.01 (d, *J* = 8.0 Hz, 1H), 7.18-7.25 (m, 2H), 7.32 (t, *J* = 7.5 Hz, 2H), 7.56 (d, *J* = 7.5 Hz, 2H); **^{13}C-NMR** ($CDCl_3$, 125 MHz) δ 28.4, 44.8, 45.7, 104.9, 124.4, 124.8, 126.9, 127.1, 128.4, 128.6, 133.3, 148.7, 155.9, 171.7; **FT-IR** (neat) ν = 3424, 3025, 2966, 2854, 1806, 1674, 1510, 1492, 1446, 1384, 1172, 1040, 951, 753, 692 cm^{-1}; **HRMS** (ESI) m/z calculated for $C_{16}H_{15}O_2S$ $[M+H]^+$: 271.0748, found : 271.0757.

IV. NMR spectra copies

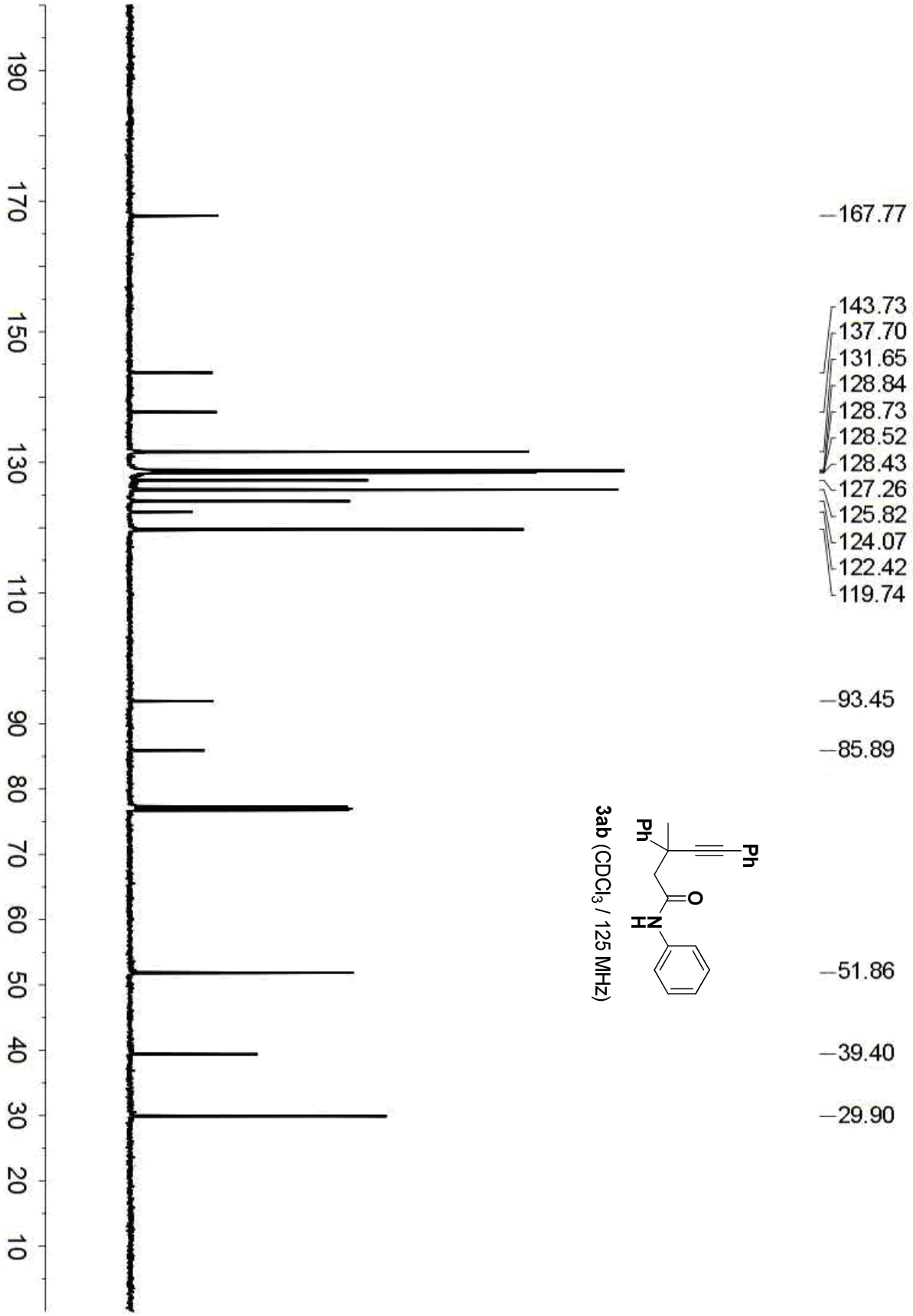


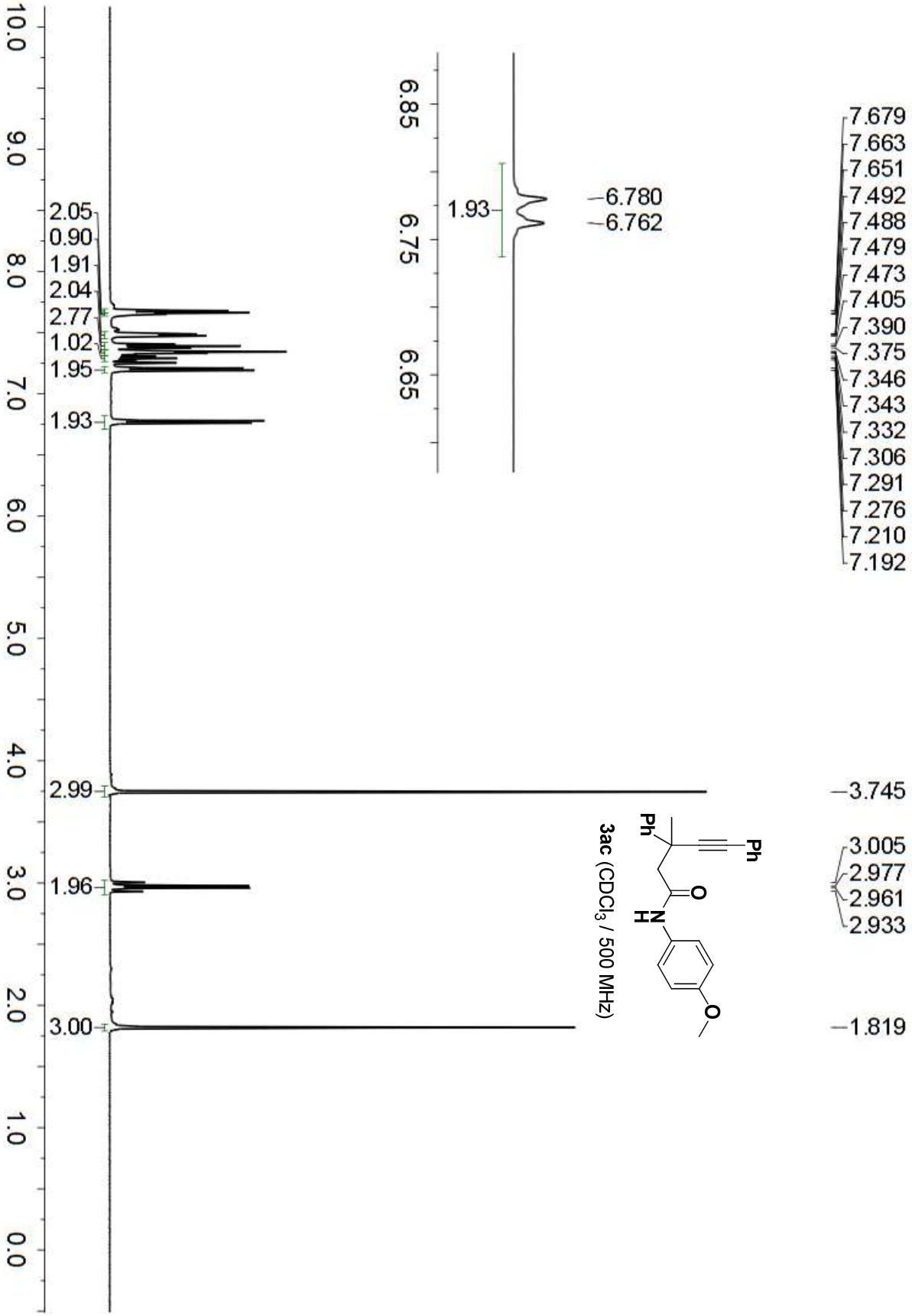


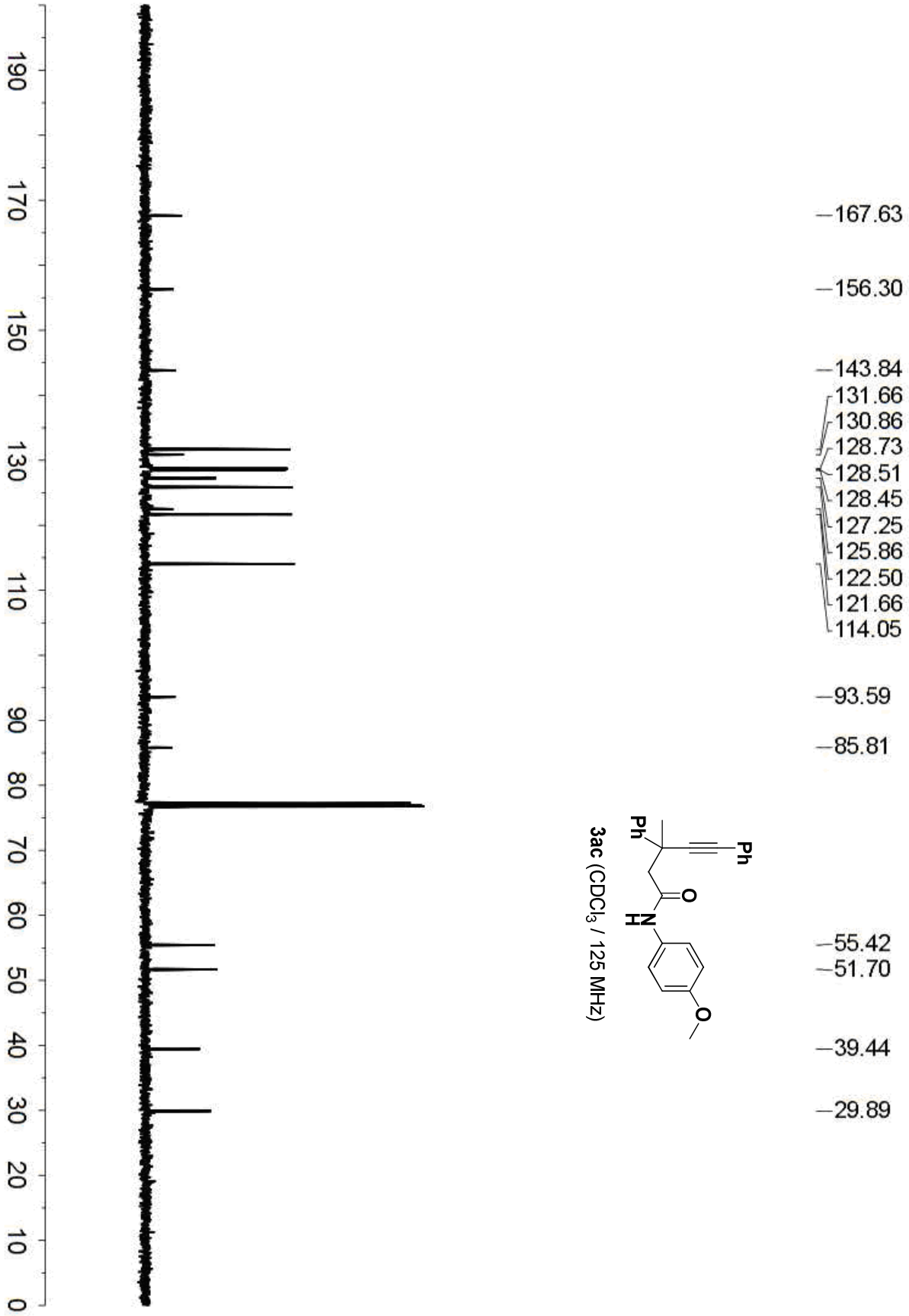
3aa ($CDCl_3$ / 125 MHz)

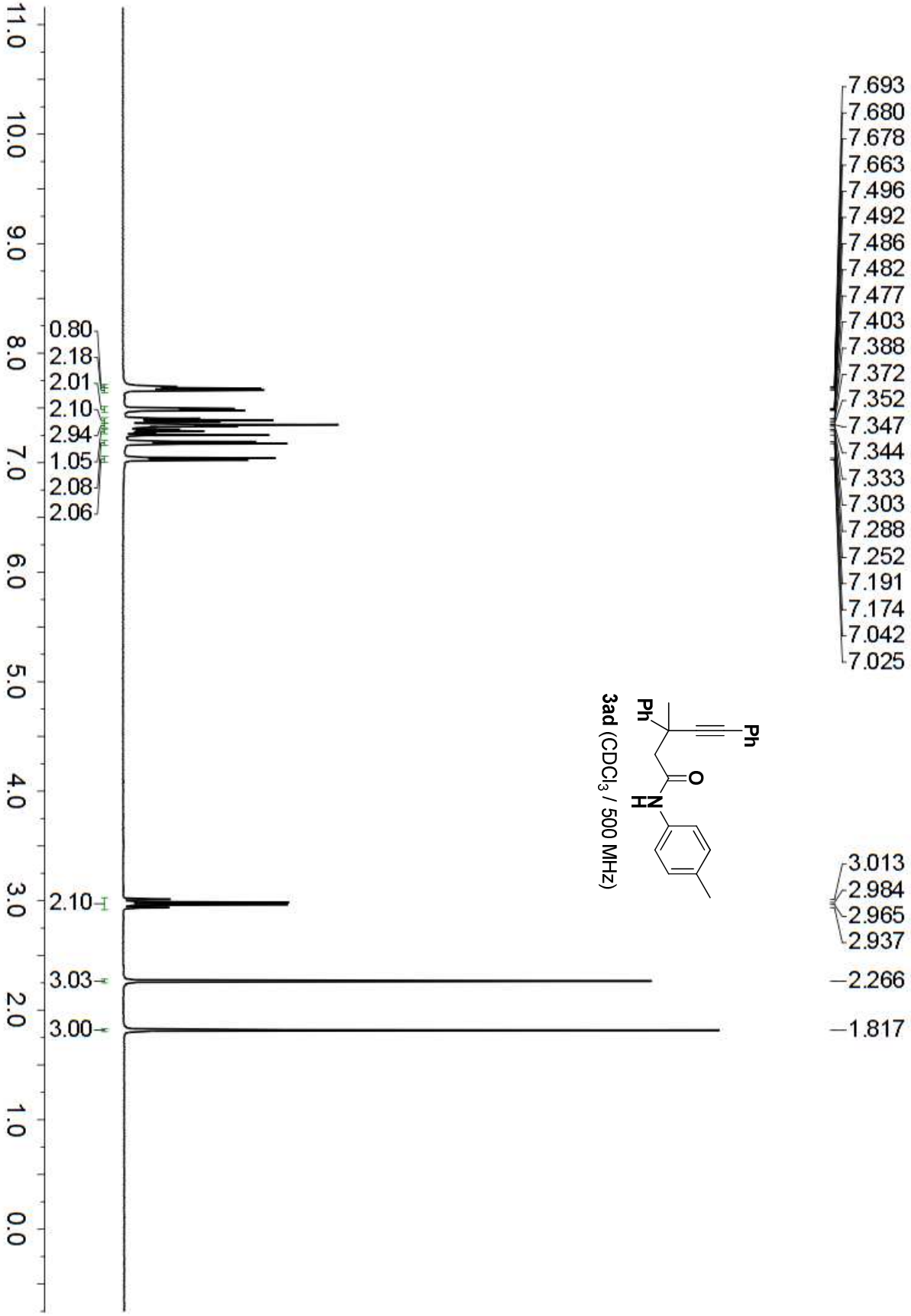


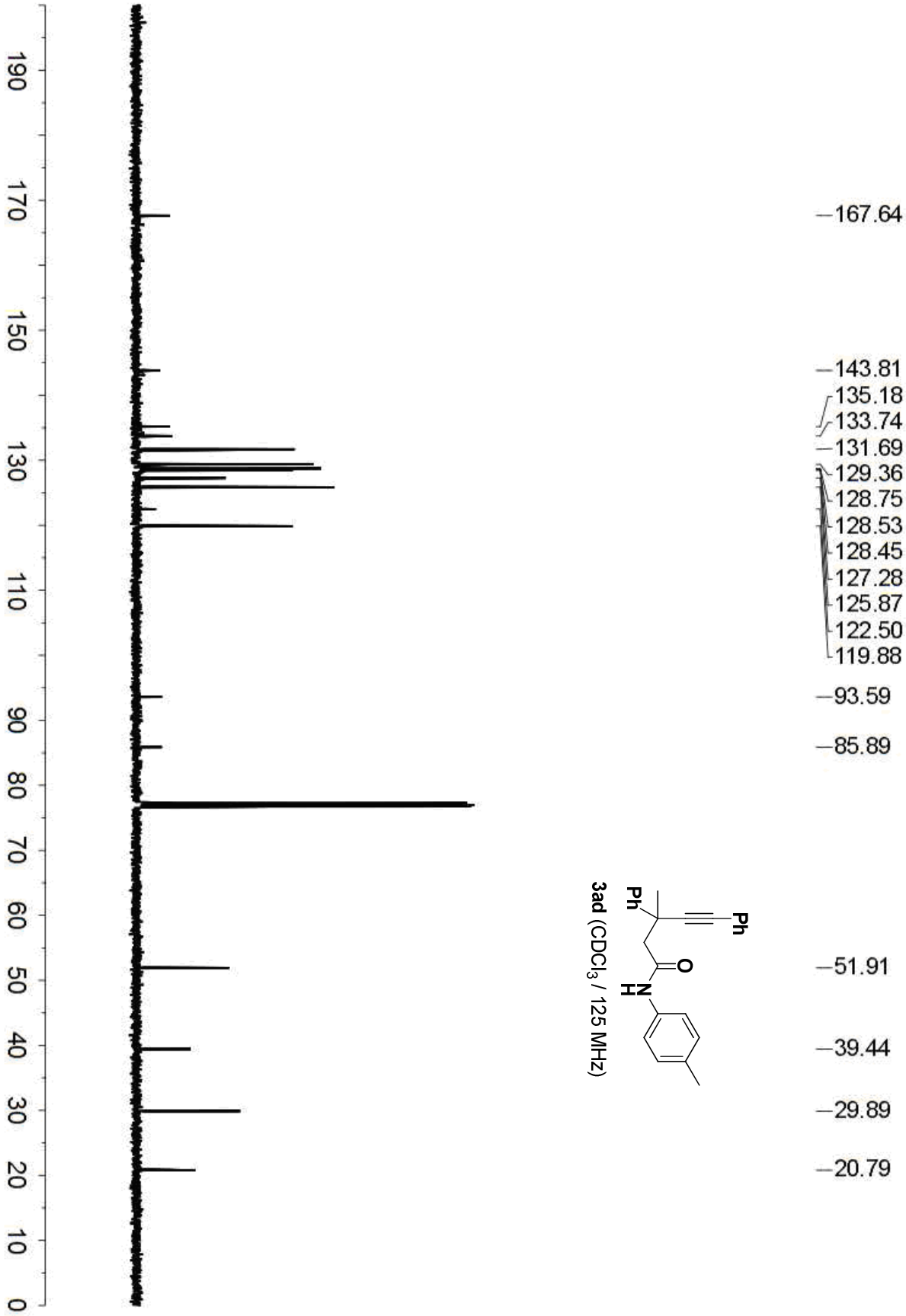
3ab ($CDCl_3$ / 500 MHz)

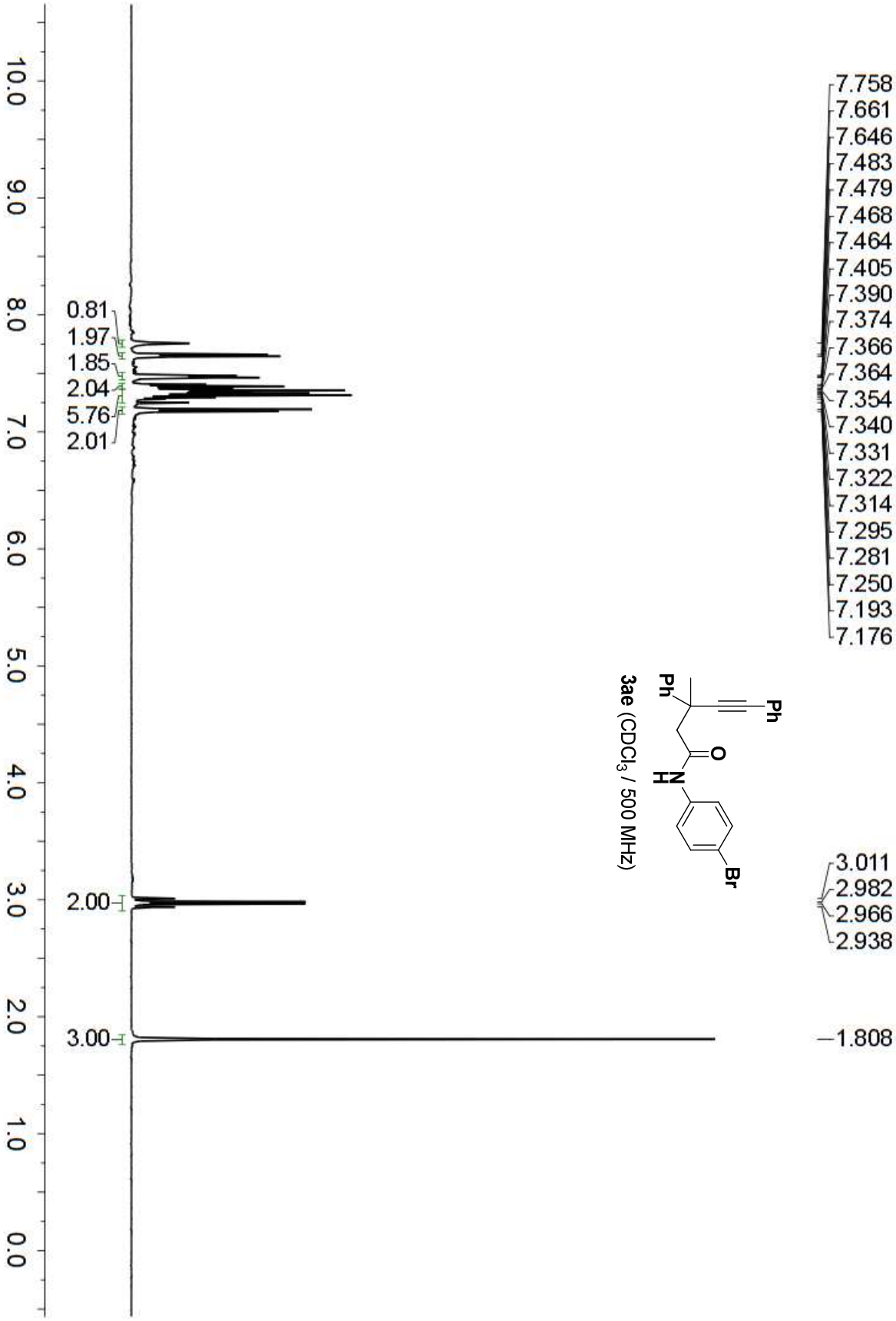

167.77
143.73
137.70
131.65
128.84
128.73
128.52
128.43
127.26
125.82
124.07
122.42
119.74
93.45
85.89
51.86
39.40
29.90
Ph
Ph
O
N
H
3ab (CDCl3 / 125 MHz)
190
170
150
130
110
90
80
70
60
50
40
30
20
10


7.679
7.663
7.651
7.492
7.488
7.479
7.473
7.405
7.390
7.375
7.346
7.343
7.332
7.306
7.291
7.276
7.210
7.192
6.780
6.762
3.745
3.005
2.977
2.961
2.933
1.819
2.05
0.90
1.91
2.04
2.77
1.02
1.95
1.93
2.99
1.96
3.00
1.93
Ph
Ph
O
N
H
O
3ac (CDCl3 / 500 MHz)

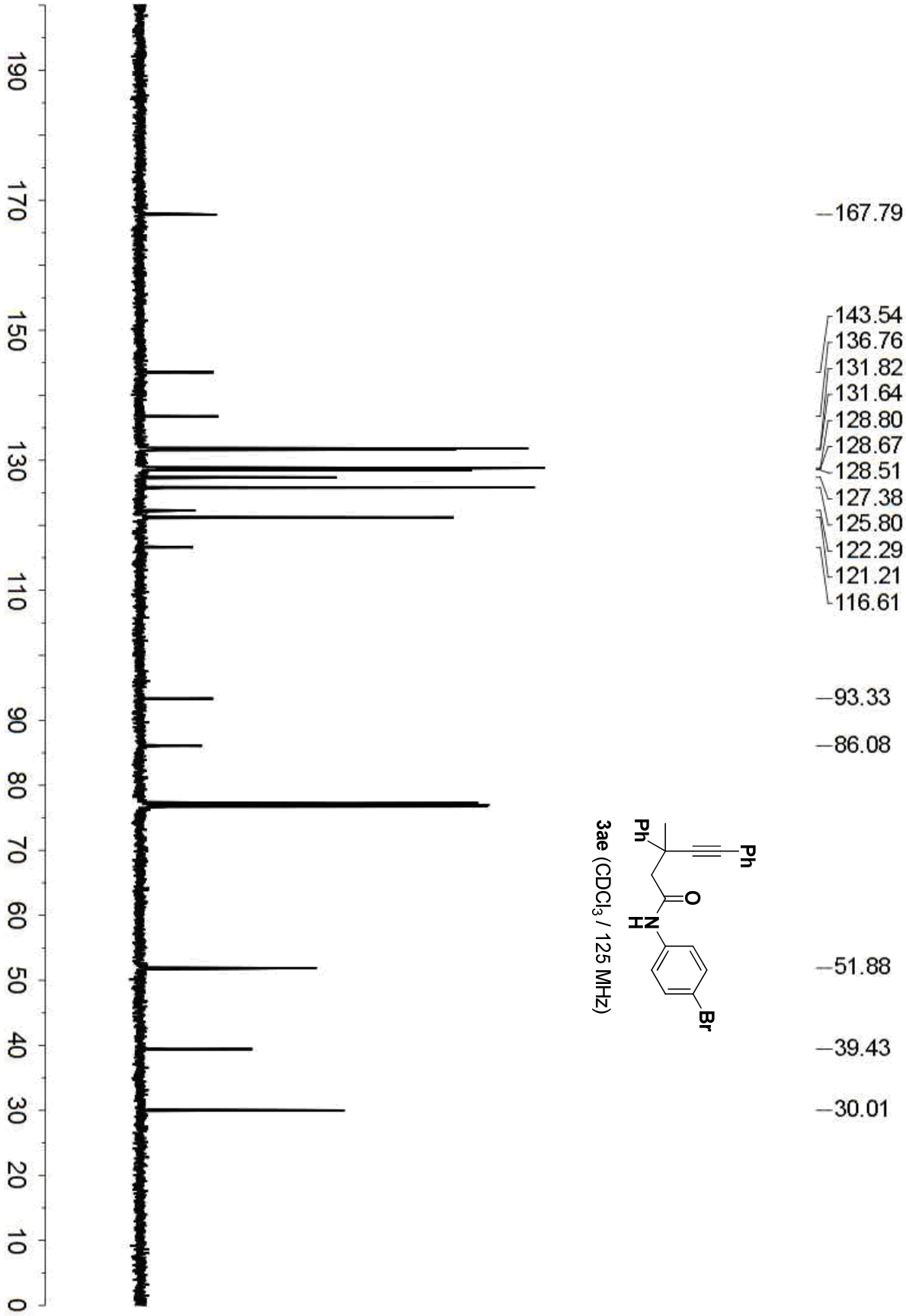

167.63
156.30
143.84
131.66
130.86
128.73
128.51
128.45
127.25
125.86
122.50
121.66
114.05
93.59
85.81
55.42
51.70
39.44
29.89
Ph
Ph
O
N
H
O
3ac (CDCl3 / 125 MHz)
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0

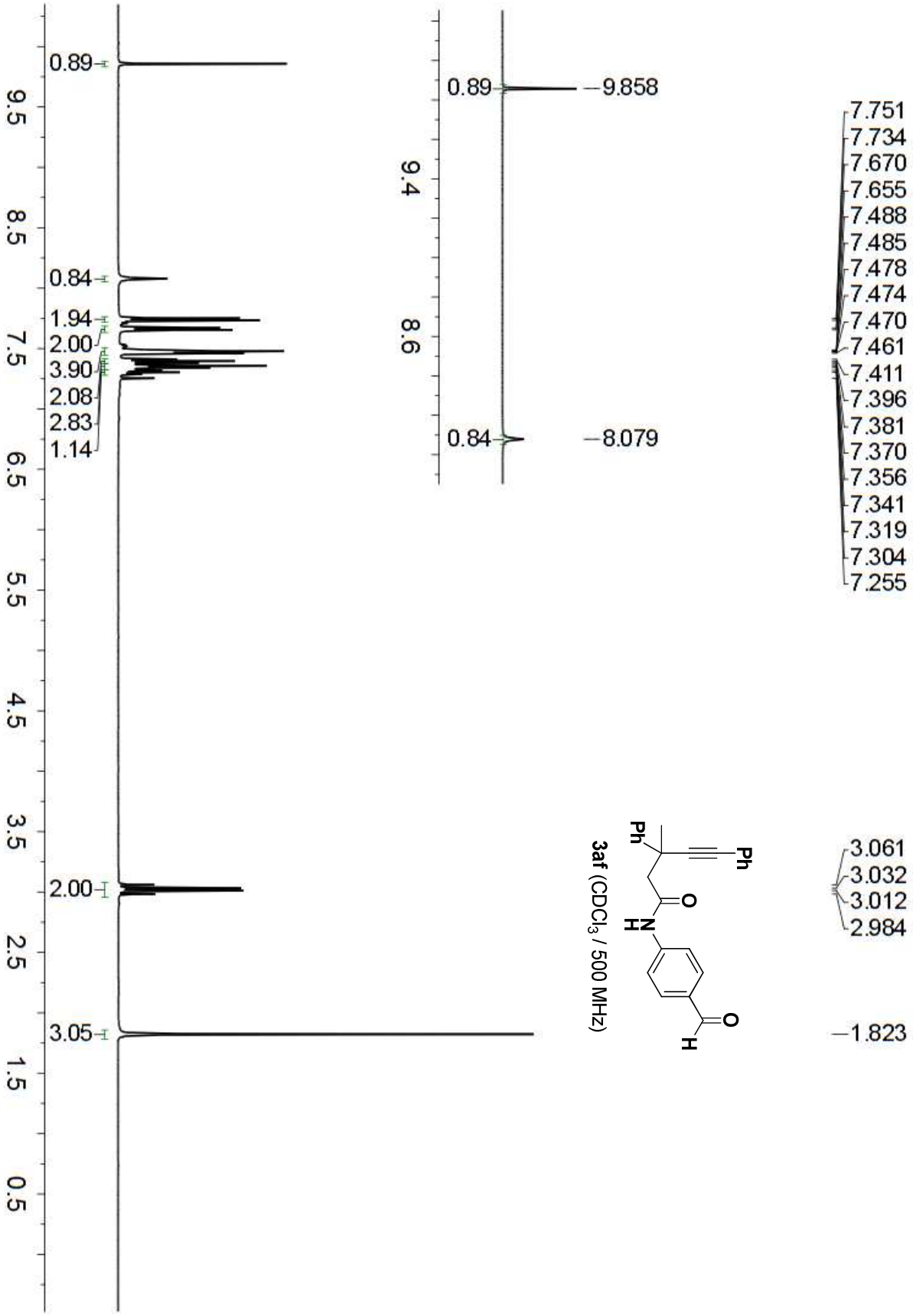

7.693
7.680
7.678
7.663
7.496
7.492
7.486
7.482
7.477
7.403
7.388
7.372
7.352
7.347
7.344
7.333
7.303
7.288
7.252
7.191
7.174
7.042
7.025
3.013
2.984
2.965
2.937
2.266
1.817
0.80
2.18
2.01
2.10
2.94
1.05
2.08
2.06
2.10
3.03
3.00
Ph
Ph
O
N
H
3ad (CDCl3 / 500 MHz)
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0

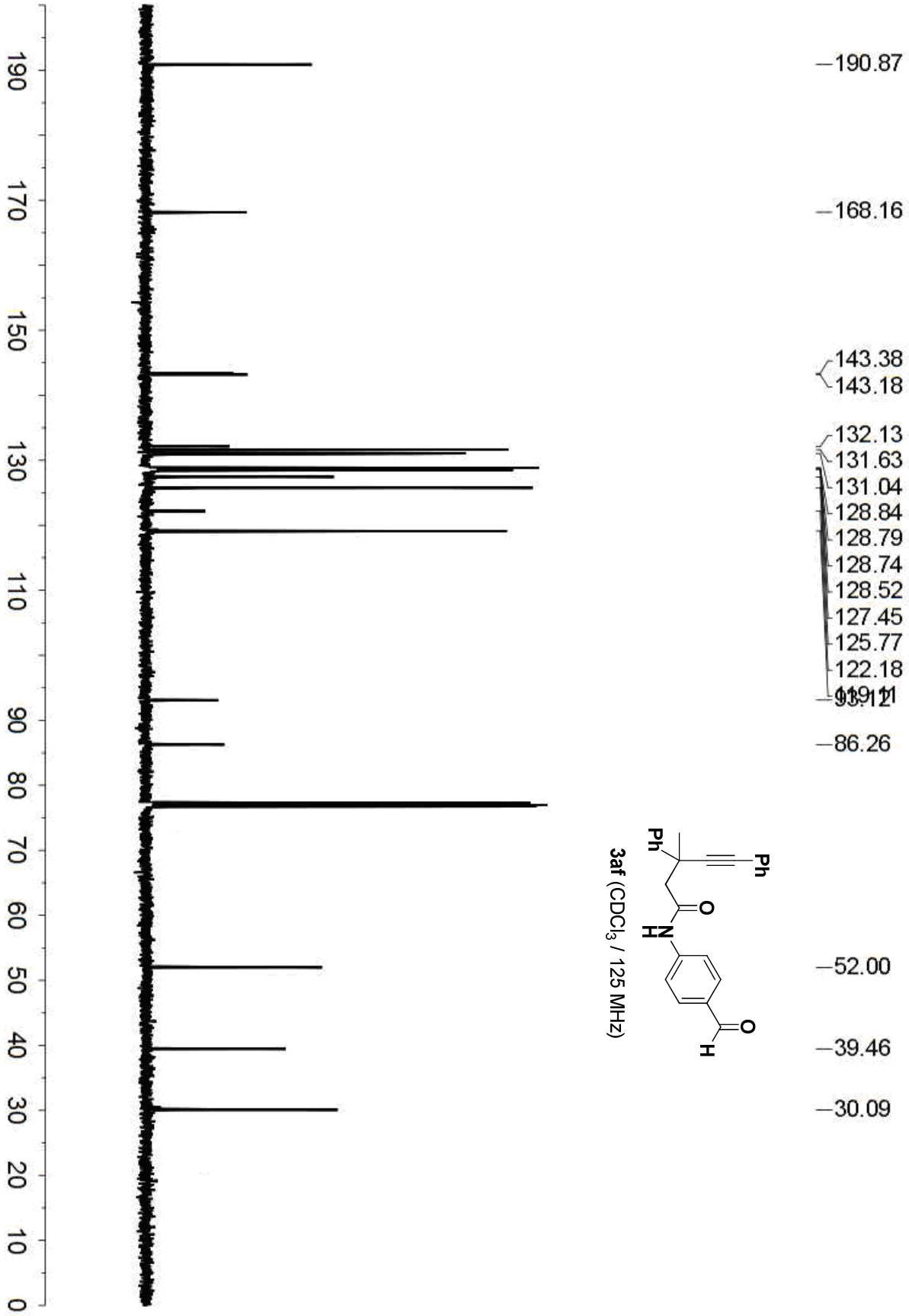

167.64
143.81
135.18
133.74
131.69
129.36
128.75
128.53
128.45
127.28
125.87
122.50
119.88
93.59
85.89
51.91
39.44
29.89
20.79
Ph
Ph
O
N
H
3ad (CDCl3 / 125 MHz)
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0



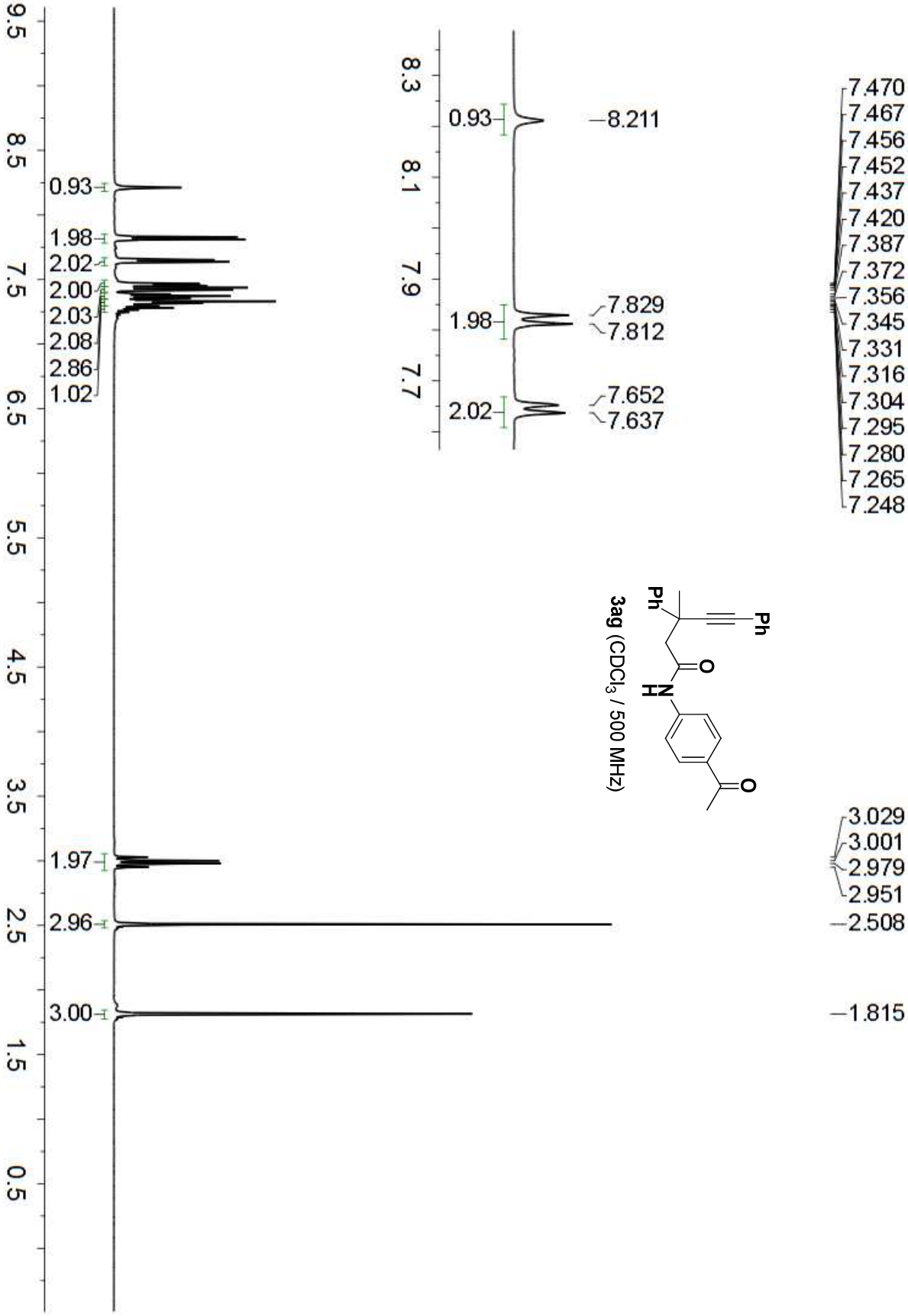
3ae ($CDCl_3$ / 500 MHz)

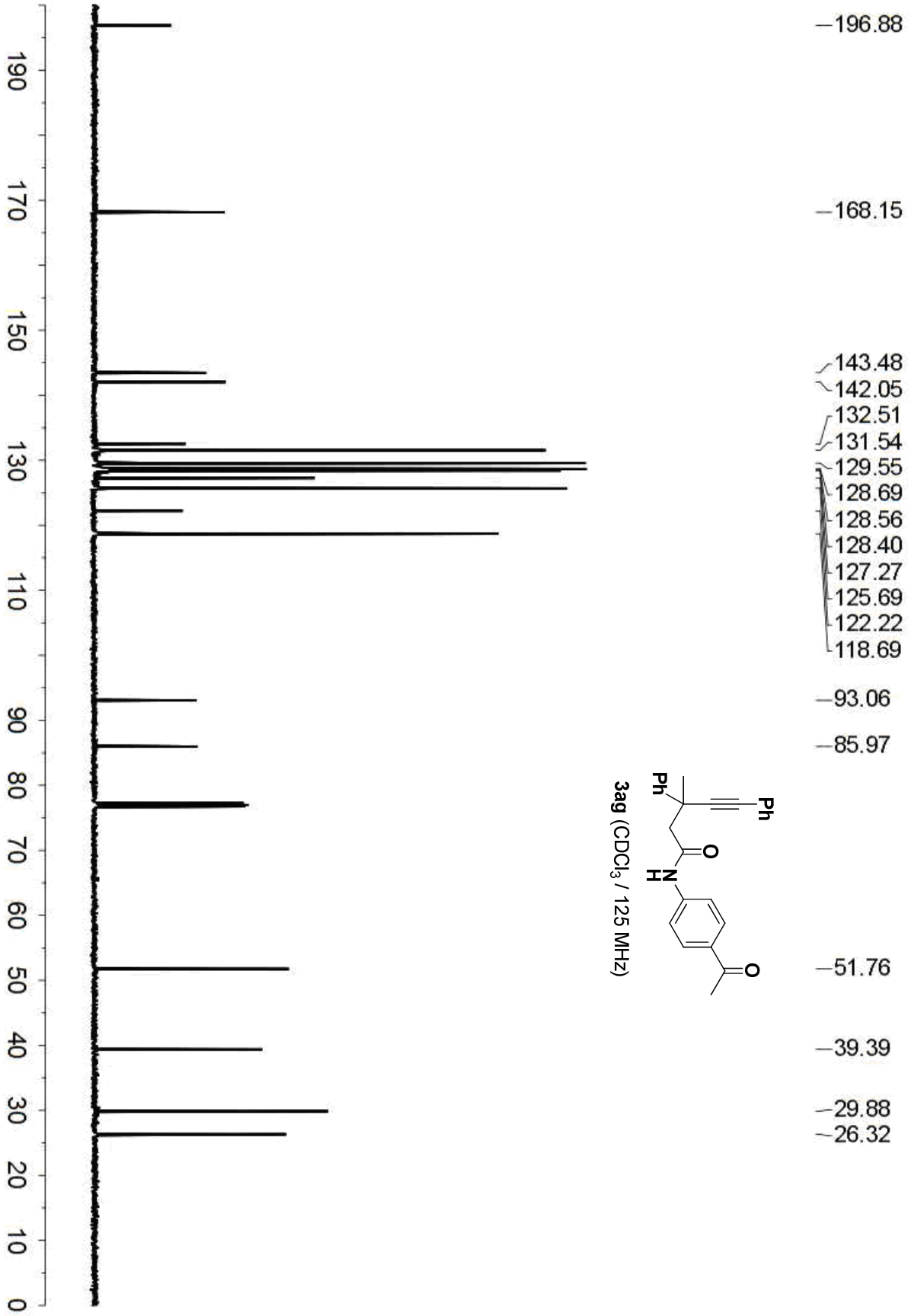

167.79
143.54
136.76
131.82
131.64
128.80
128.67
128.51
127.38
125.80
122.29
121.21
116.61
93.33
86.08
51.88
39.43
30.01
Ph
Ph
O
N
H
Br
3ae (CDCl3 / 125 MHz)
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0

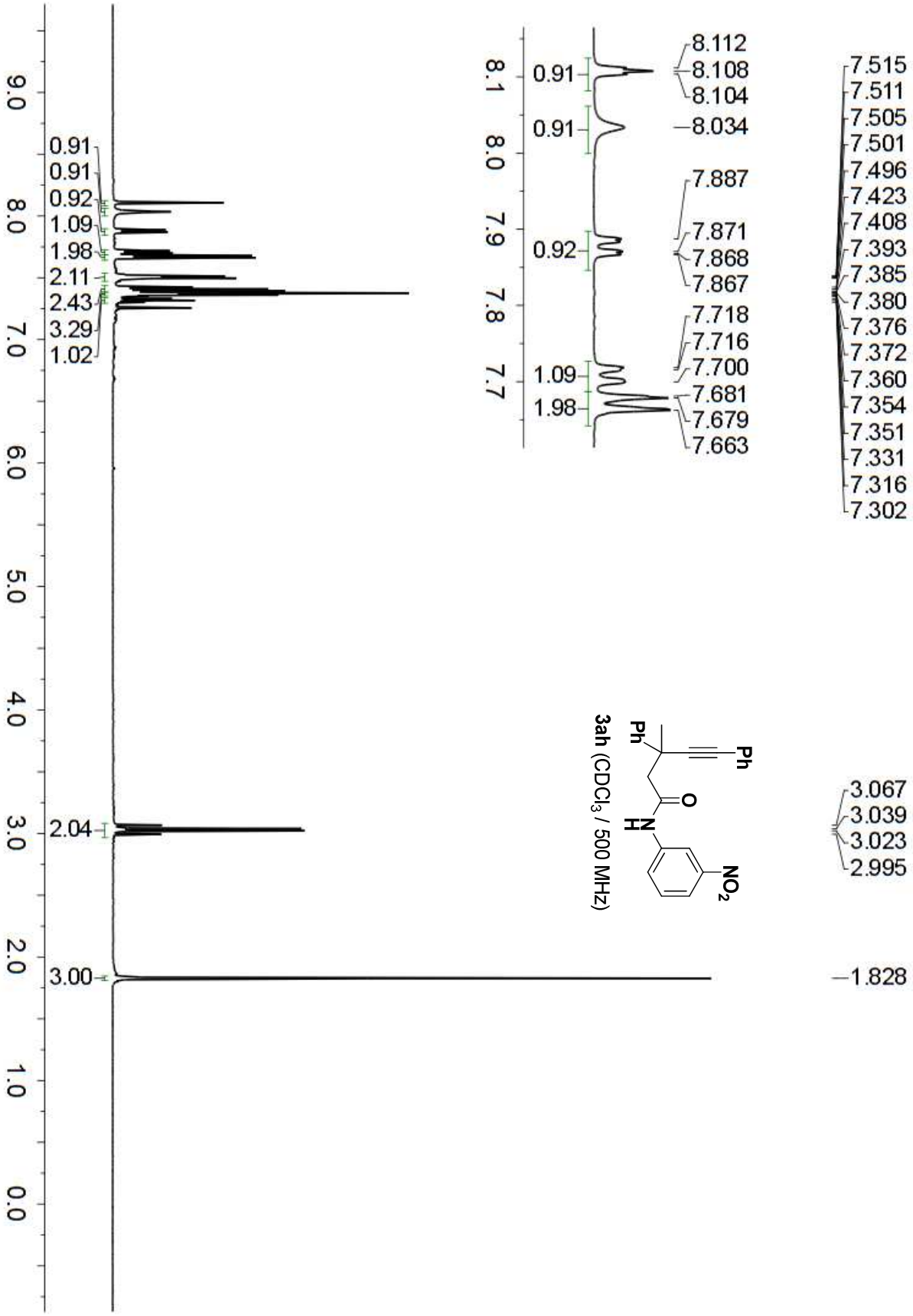

3af (CDCl3 / 500 MHz)
Ph
Ph
O
N
H
O
H
7.751
7.734
7.670
7.655
7.488
7.485
7.478
7.474
7.470
7.461
7.411
7.396
7.381
7.370
7.356
7.341
7.319
7.304
7.255
3.061
3.032
3.012
2.984
1.823
0.89
9.858
0.84
8.079
0.89
0.84
1.94
2.00
3.90
2.08
2.83
1.14
2.00
3.05
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
9.4
8.6

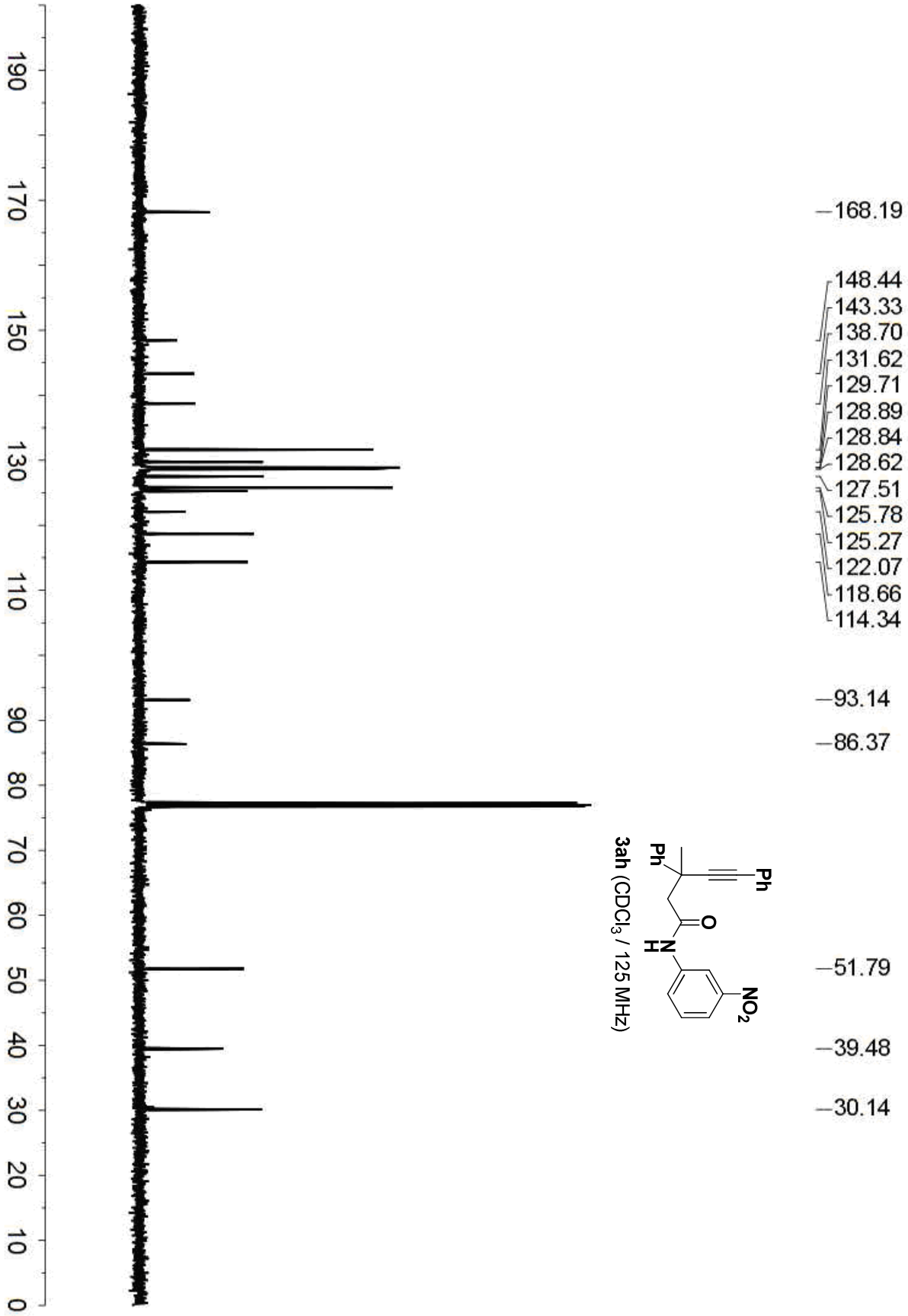


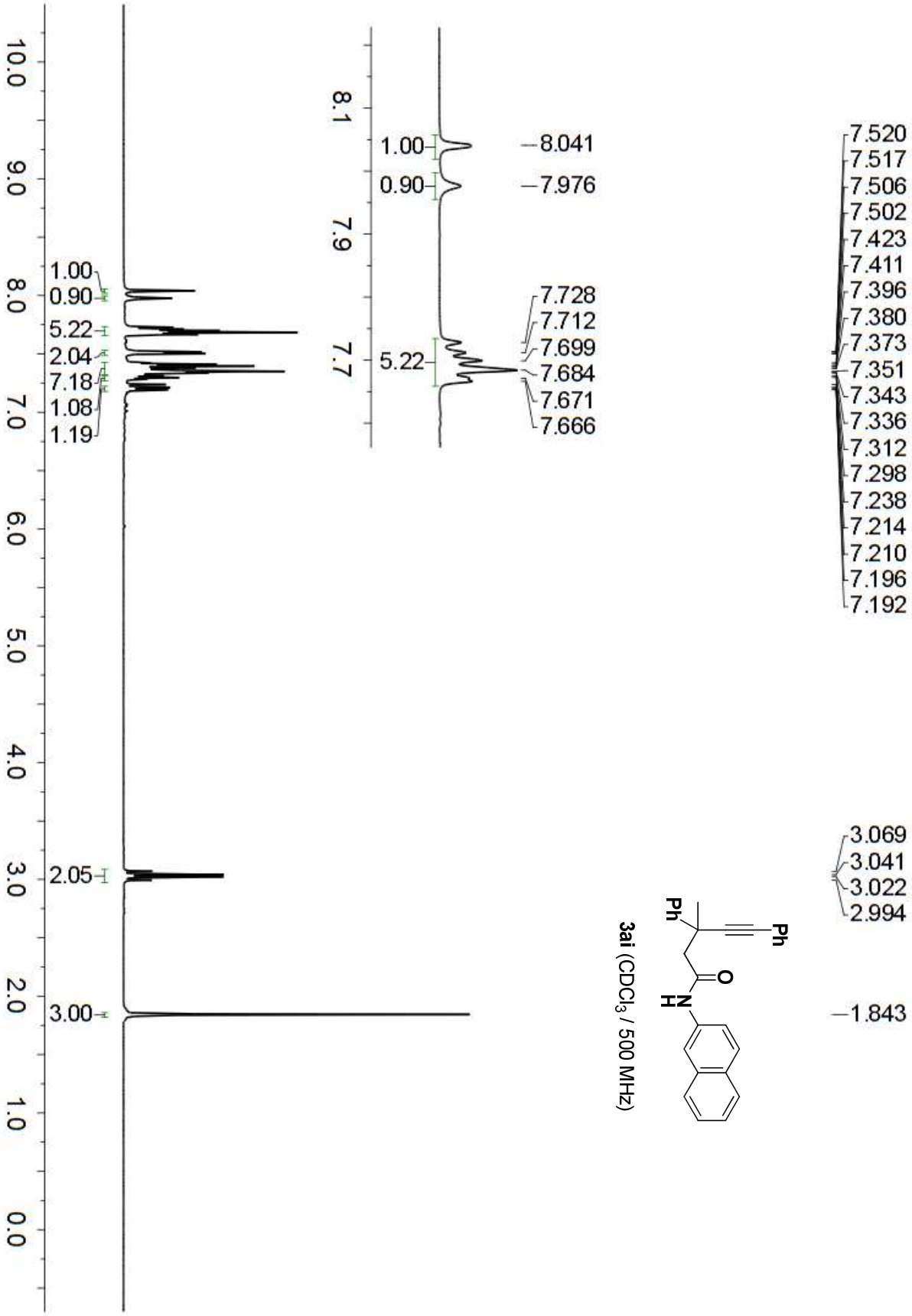
3af ($CDCl_3$ / 125 MHz)


7.470
7.467
7.456
7.452
7.437
7.420
7.387
7.372
7.356
7.345
7.331
7.316
7.304
7.295
7.280
7.265
7.248
8.211
7.829
7.812
7.652
7.637
0.93
1.98
2.02
3.029
3.001
2.979
2.951
2.508
1.815
Ph
Ph
O
N
H
O
3ag (CDCl3 / 500 MHz)
0.93
1.98
2.02
2.00
2.03
2.08
2.86
1.02
1.97
2.96
3.00
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
8.3
8.1
7.9
7.7

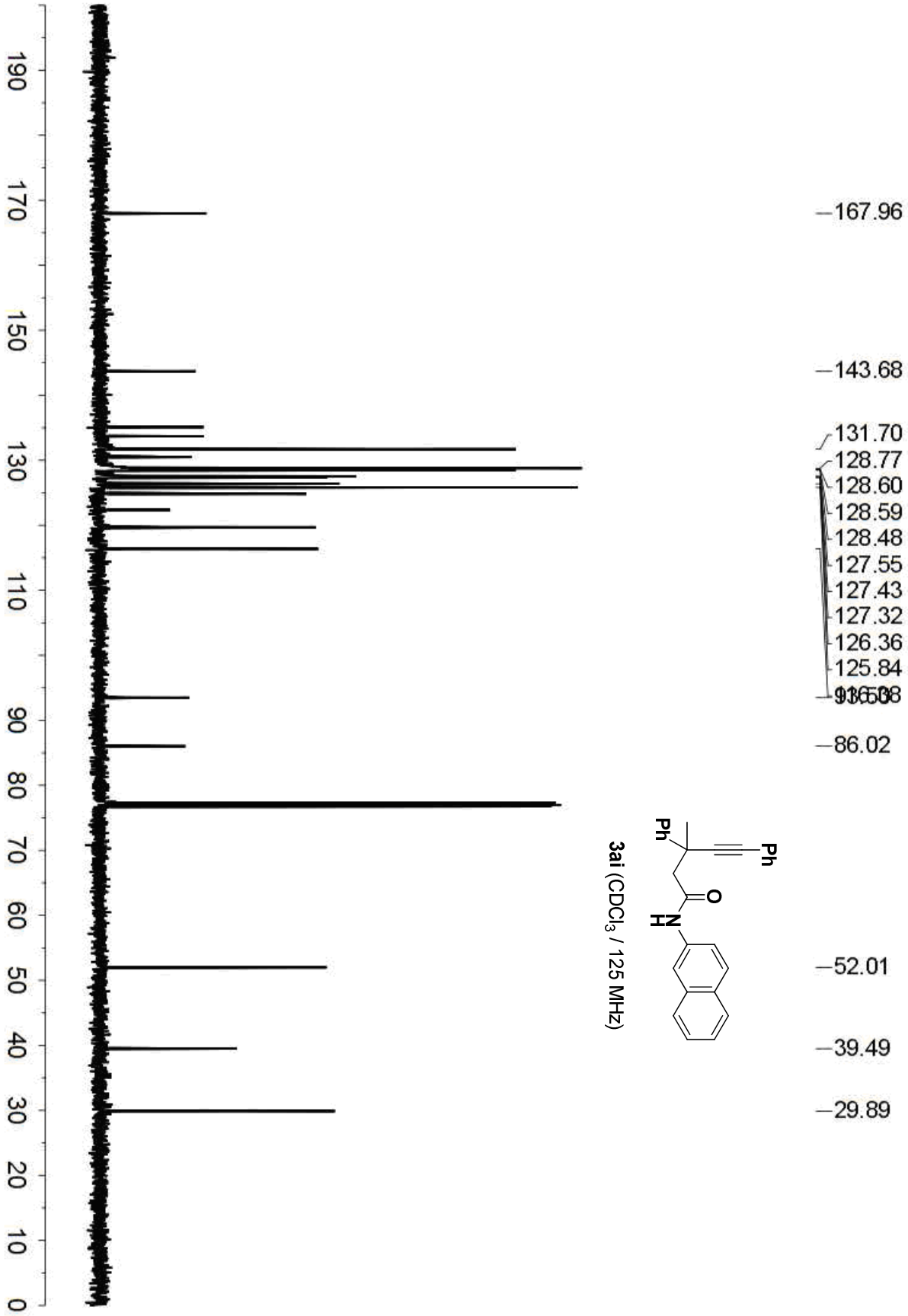

196.88
168.15
143.48
142.05
132.51
131.54
129.55
128.69
128.56
128.40
127.27
125.69
122.22
118.69
93.06
85.97
51.76
39.39
29.88
26.32
Ph
Ph
O
N
H
O
3ag (CDCl3 / 125 MHz)
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0

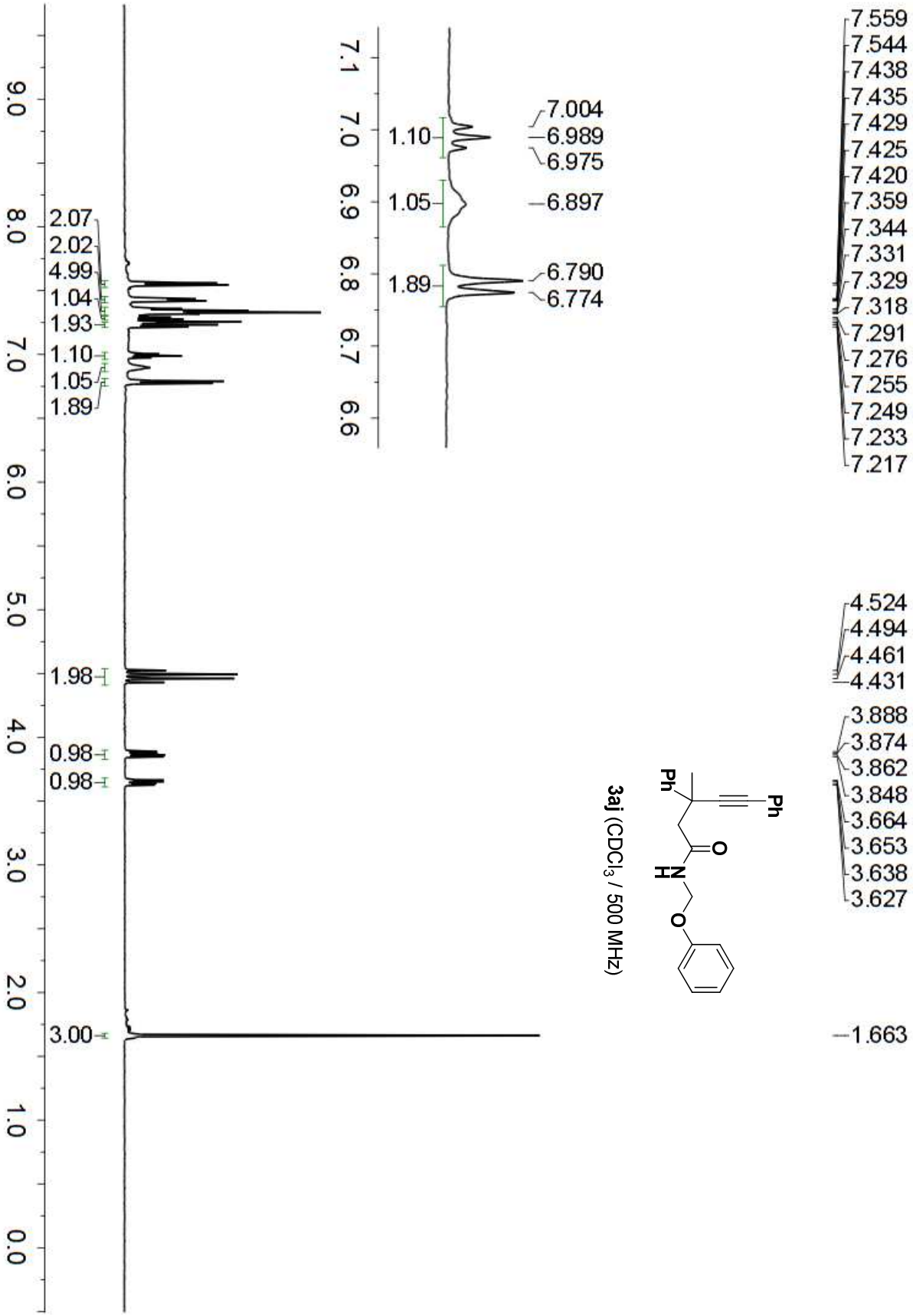

3ah (CDCl3 / 500 MHz)
Ph
Ph
O
N
H
NO2
8.112
8.108
8.104
8.034
7.887
7.871
7.868
7.867
7.718
7.716
7.700
7.681
7.679
7.663
7.515
7.511
7.505
7.501
7.496
7.423
7.408
7.393
7.385
7.380
7.376
7.372
7.360
7.354
7.351
7.331
7.316
7.302
3.067
3.039
3.023
2.995
1.828
0.91
0.91
0.92
1.09
1.98
2.11
2.43
3.29
1.02
2.04
3.00


168.19
148.44
143.33
138.70
131.62
129.71
128.89
128.84
128.62
127.51
125.78
125.27
122.07
118.66
114.34
93.14
86.37
51.79
39.48
30.14
Ph
Ph
O
N
H
NO2
3ah (CDCl3 / 125 MHz)
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0

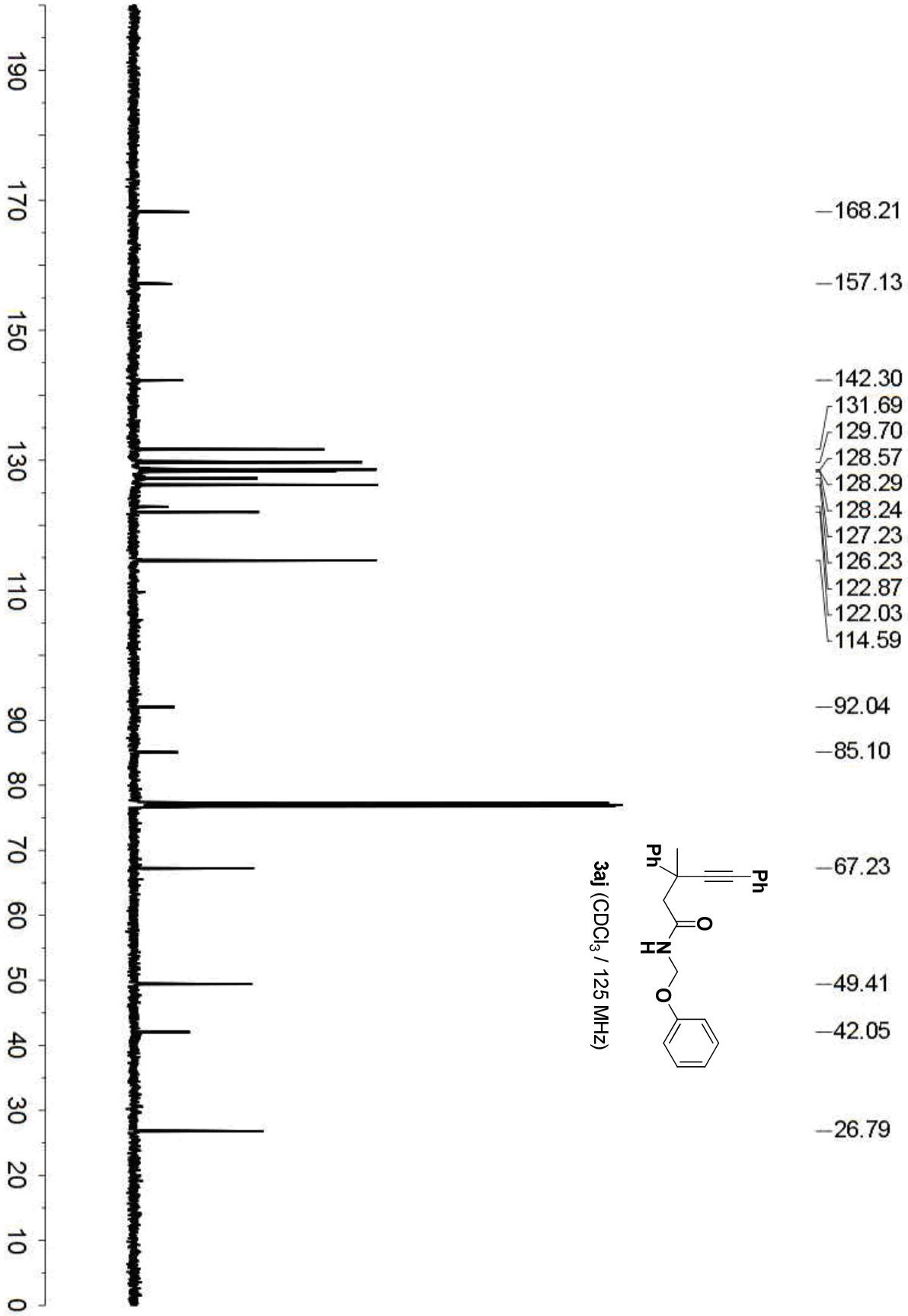


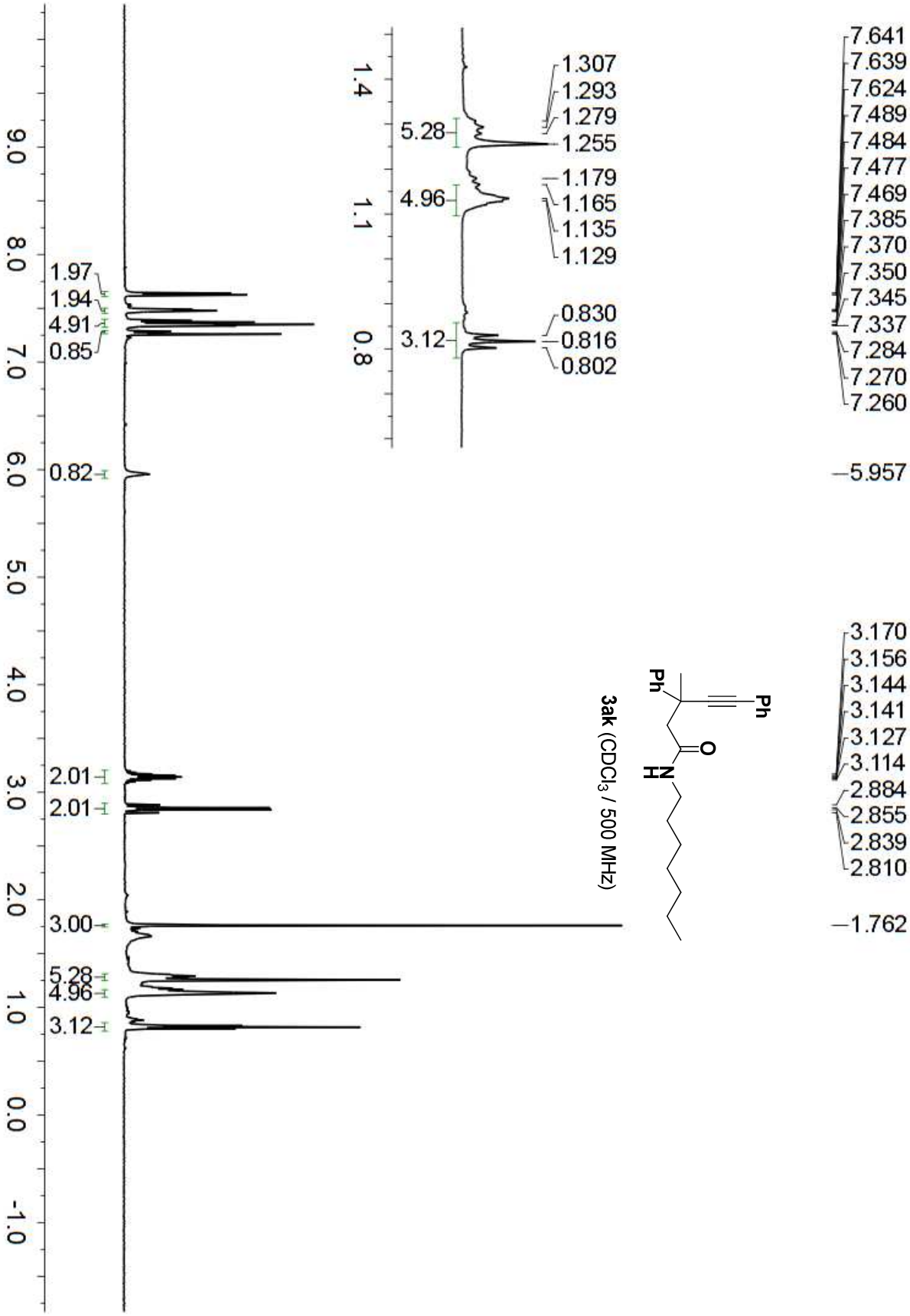
3ai ($CDCl_3$ / 500 MHz)

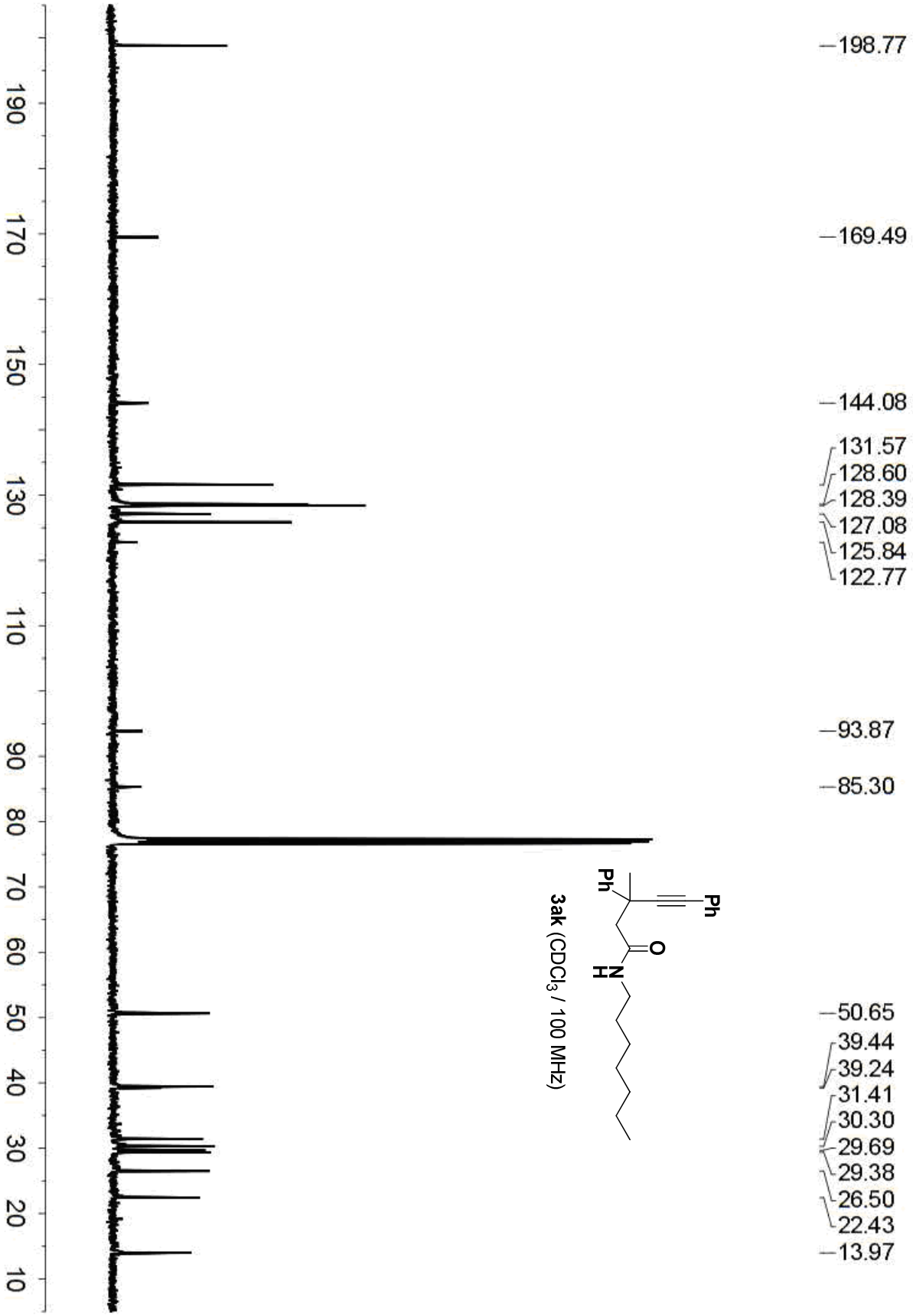

167.96
143.68
131.70
128.77
128.60
128.59
128.48
127.55
127.43
127.32
126.36
125.84
86.02
52.01
39.49
29.89
Ph
Ph
O
N
H
3ai (CDCl3 / 125 MHz)
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0

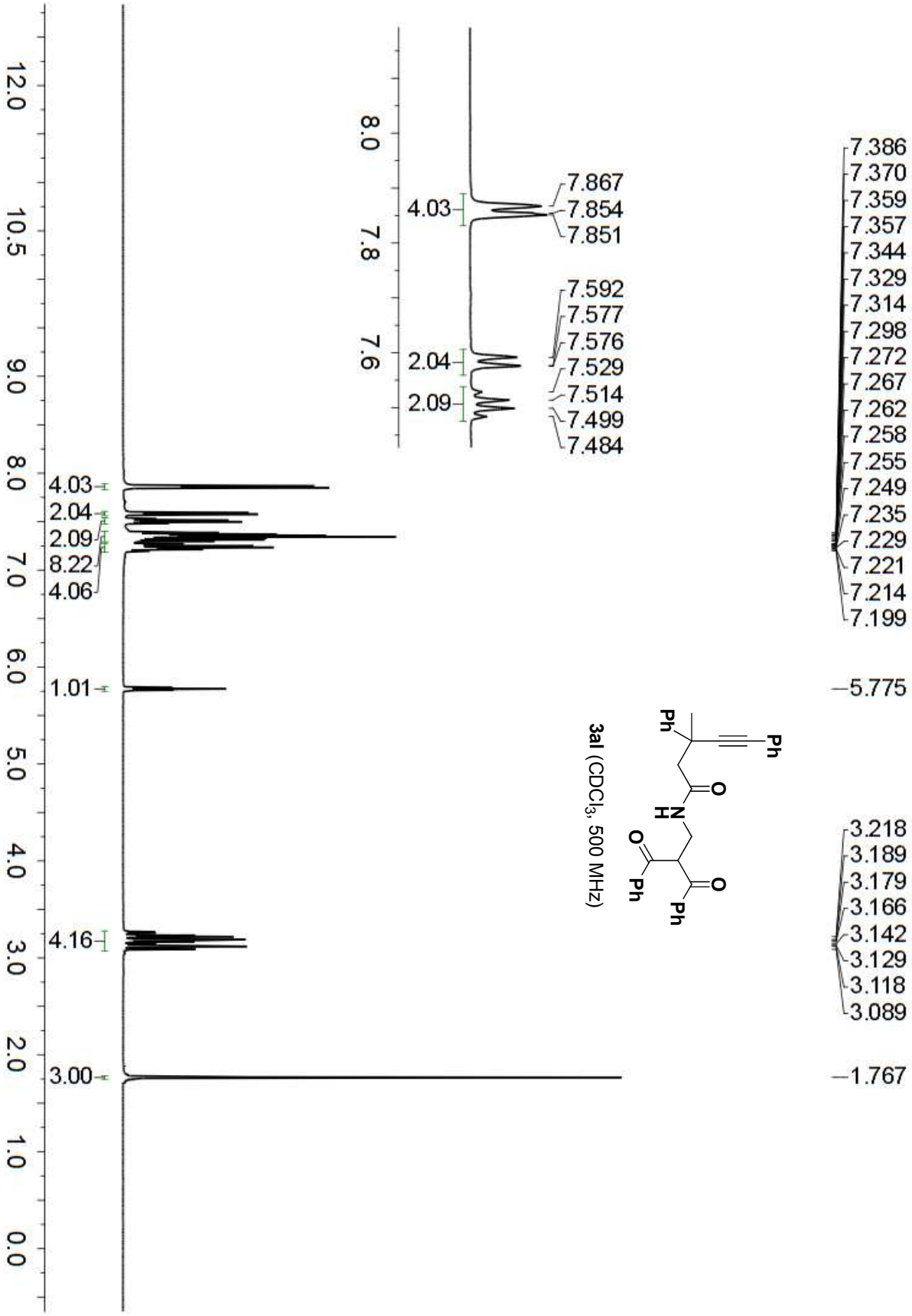


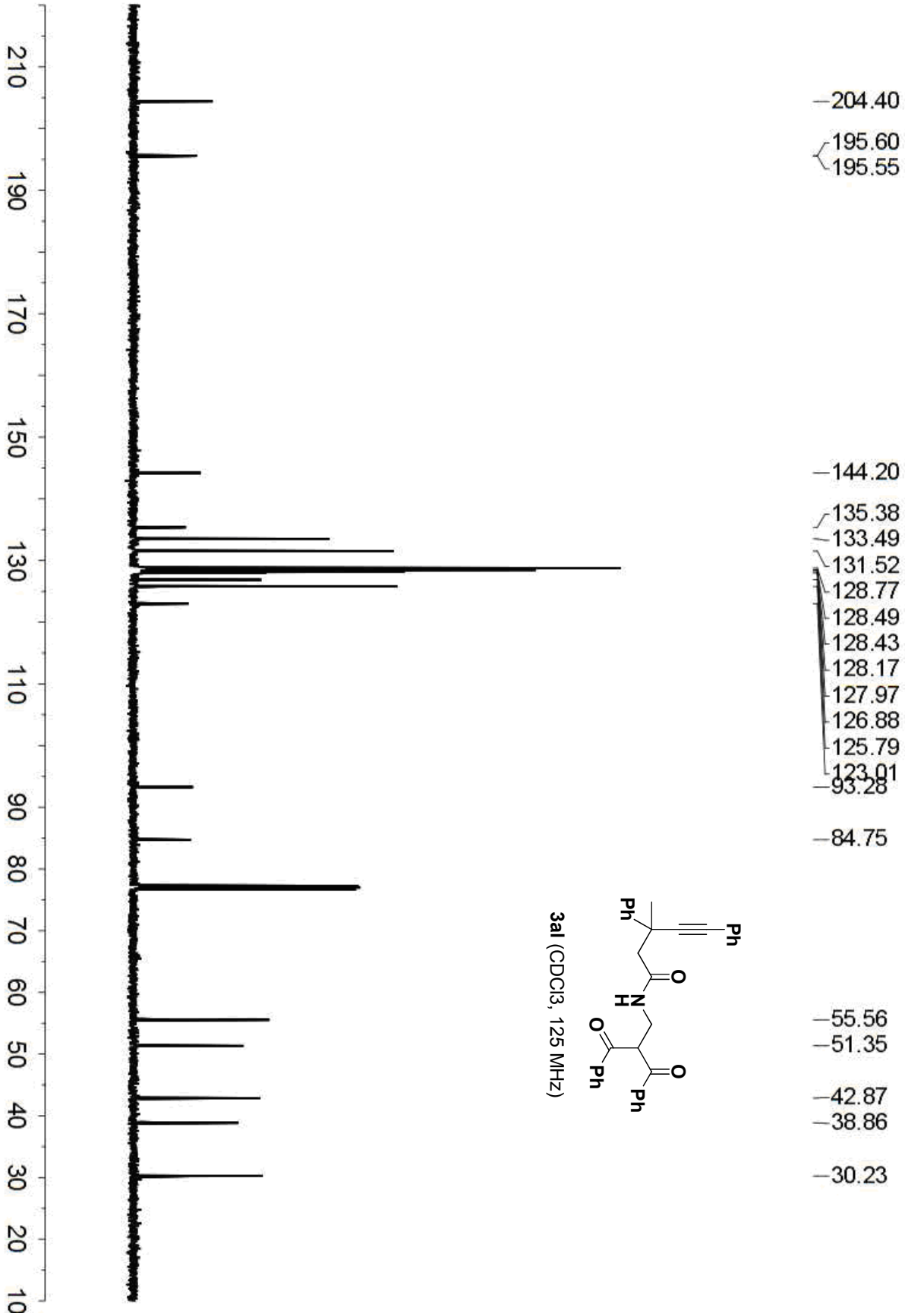
3aj ($CDCl_3$ / 500 MHz)


168.21
157.13
142.30
131.69
129.70
128.57
128.29
128.24
127.23
126.23
122.87
122.03
114.59
92.04
85.10
67.23
49.41
42.05
26.79
Ph
Ph
O
N
H
O
3aj (CDCl3 / 125 MHz)

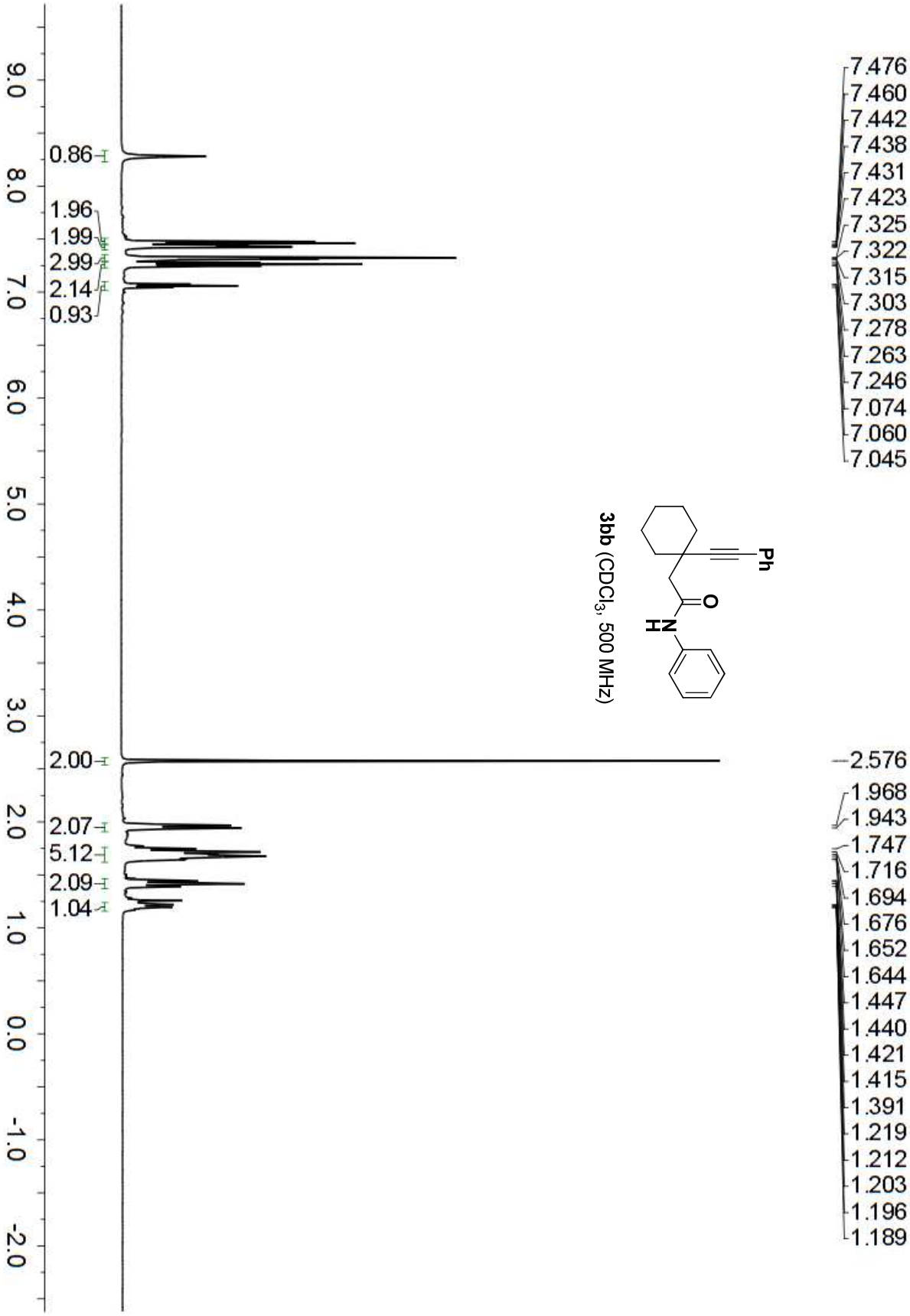

7.641
7.639
7.624
7.489
7.484
7.477
7.469
7.385
7.370
7.350
7.345
7.337
7.284
7.270
7.260
5.957
3.170
3.156
3.144
3.141
3.127
3.114
2.884
2.855
2.839
2.810
1.762
1.307
1.293
1.279
1.255
1.179
1.165
1.135
1.129
0.830
0.816
0.802
5.28
4.96
3.12
1.97
1.94
4.91
0.85
0.82
2.01
2.01
3.00
5.28
4.96
3.12
Ph
Ph
O
N
H
3ak (CDCl3 / 500 MHz)


198.77
169.49
144.08
131.57
128.60
128.39
127.08
125.84
122.77
93.87
85.30
50.65
39.44
39.24
31.41
30.30
29.69
29.38
26.50
22.43
13.97
190
170
150
130
110
90
80
70
60
50
40
30
20
10
Ph
Ph
O
N
H
3ak (CDCl3 / 100 MHz)

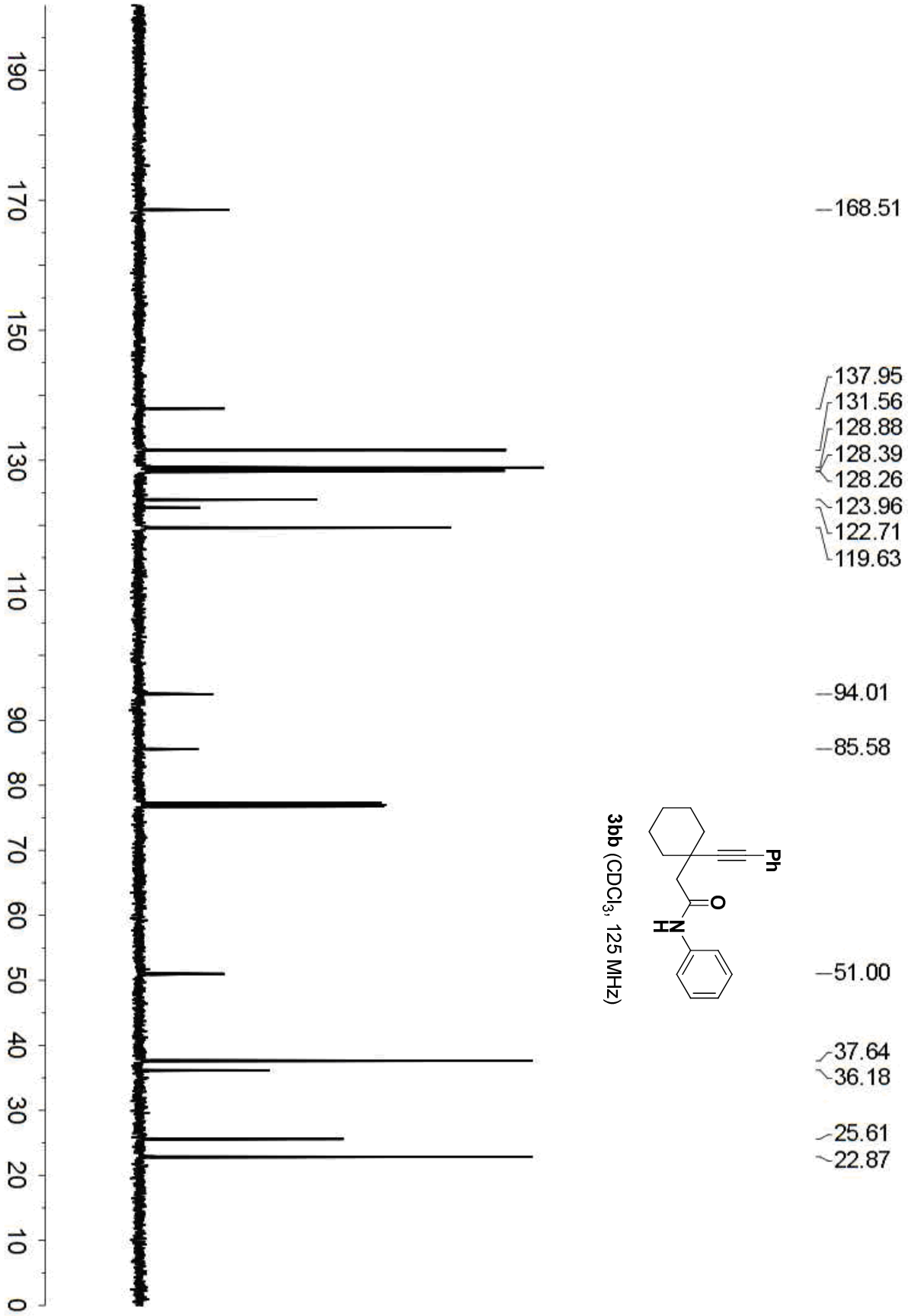

7.867
7.854
7.851
7.592
7.577
7.576
7.529
7.514
7.499
7.484
4.03
2.04
2.09
7.386
7.370
7.359
7.357
7.344
7.329
7.314
7.298
7.272
7.267
7.262
7.258
7.255
7.249
7.235
7.229
7.221
7.214
7.199
5.775
3.218
3.189
3.179
3.166
3.142
3.129
3.118
3.089
1.767
4.03
2.04
2.09
8.22
4.06
1.01
4.16
3.00
Ph
Ph
O
N
H
O
Ph
O
Ph
3al (CDCl3, 500 MHz)



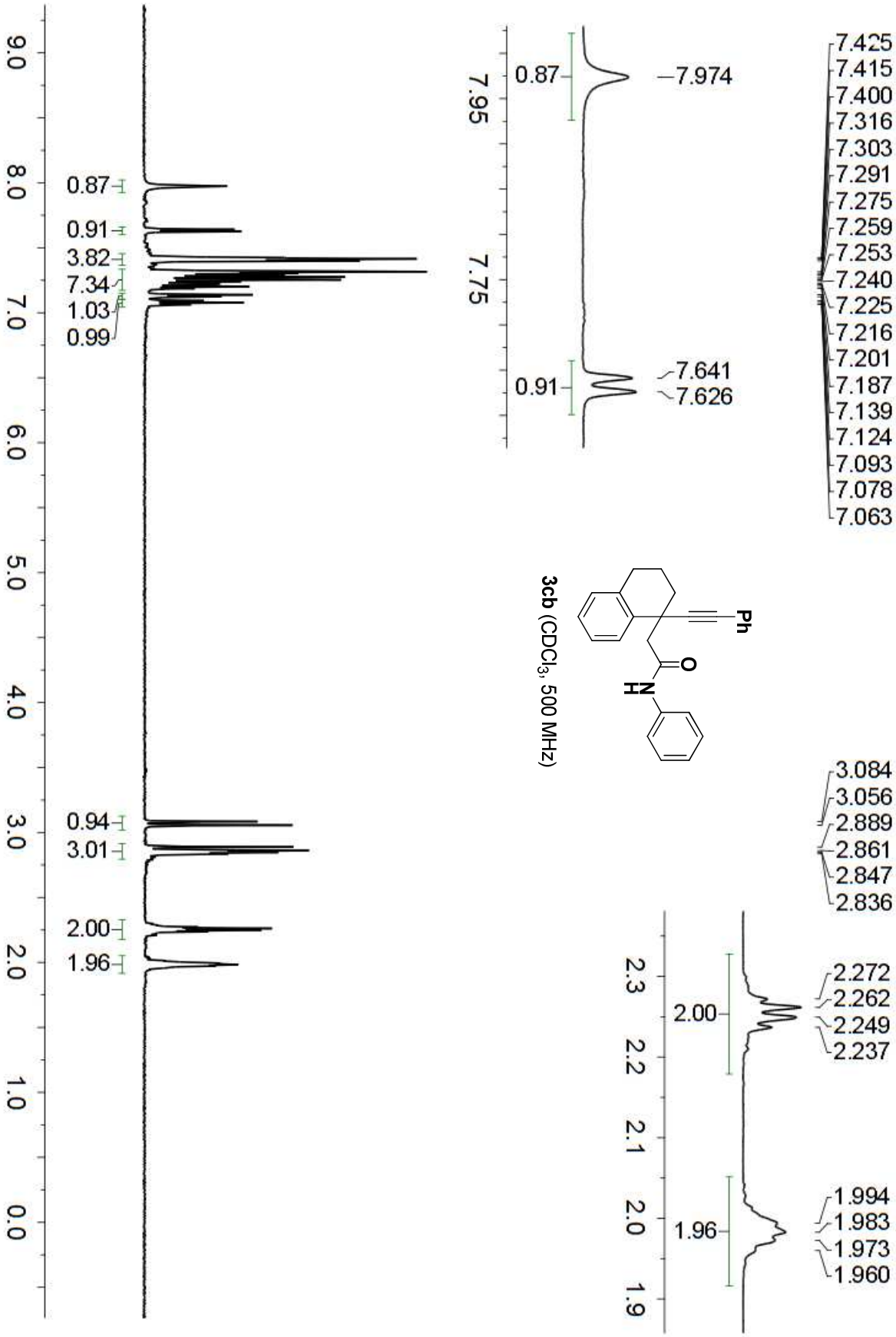
3al (CDCl3, 125 MHz)



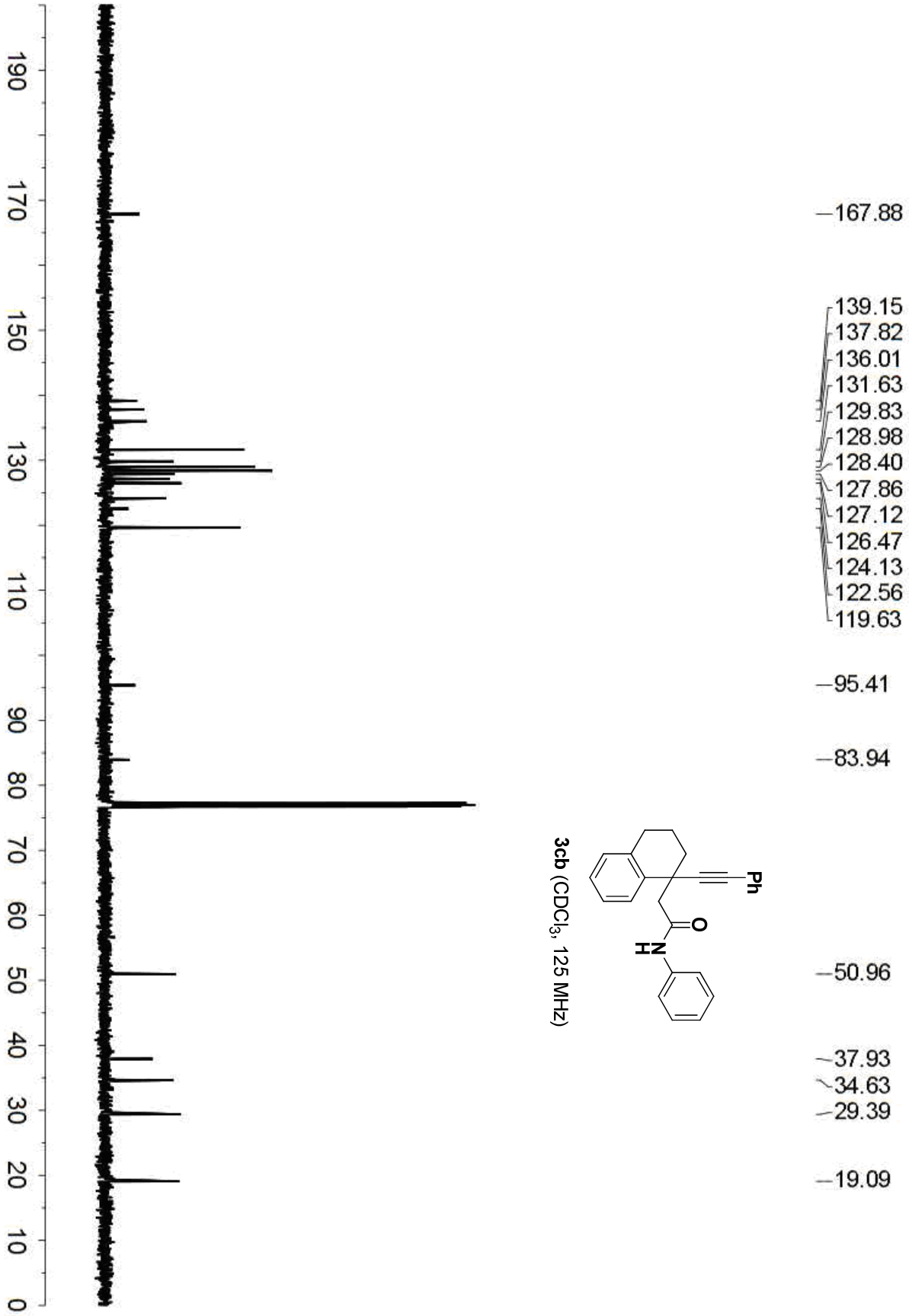
3bb ($CDCl_3$, 500 MHz)



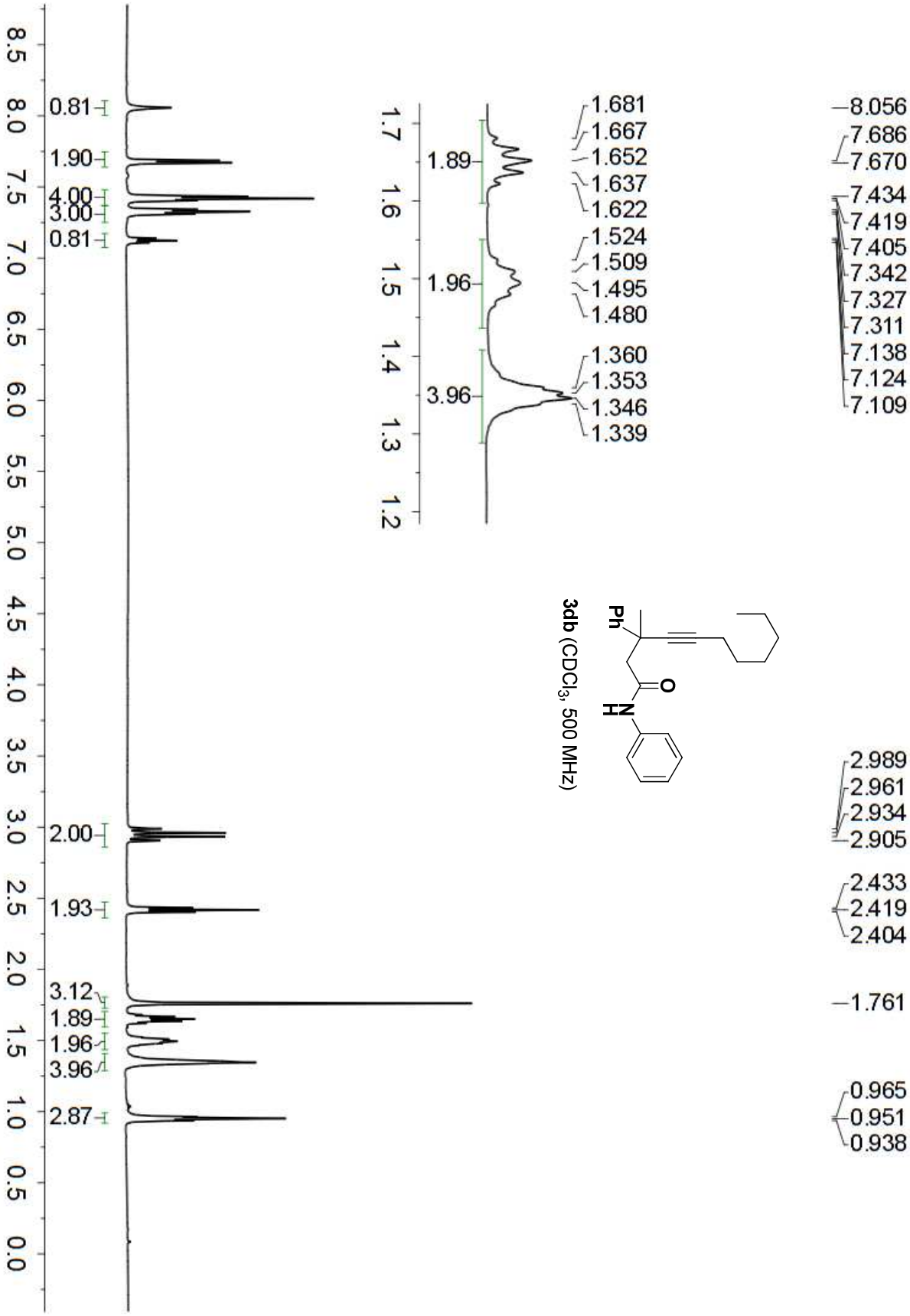
3bb ($CDCl_3$, 125 MHz)



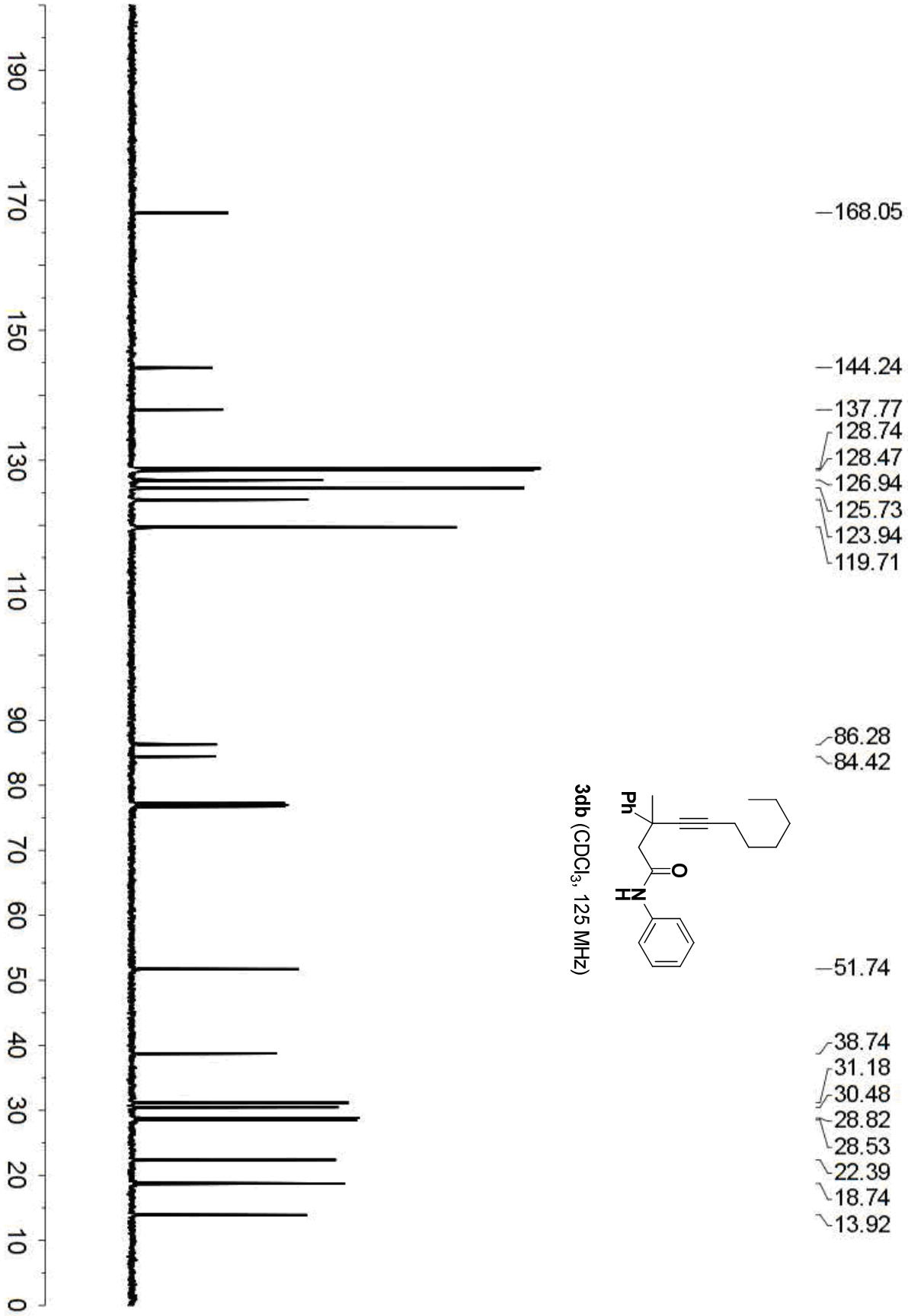
3cb ($CDCl_3$, 500 MHz)

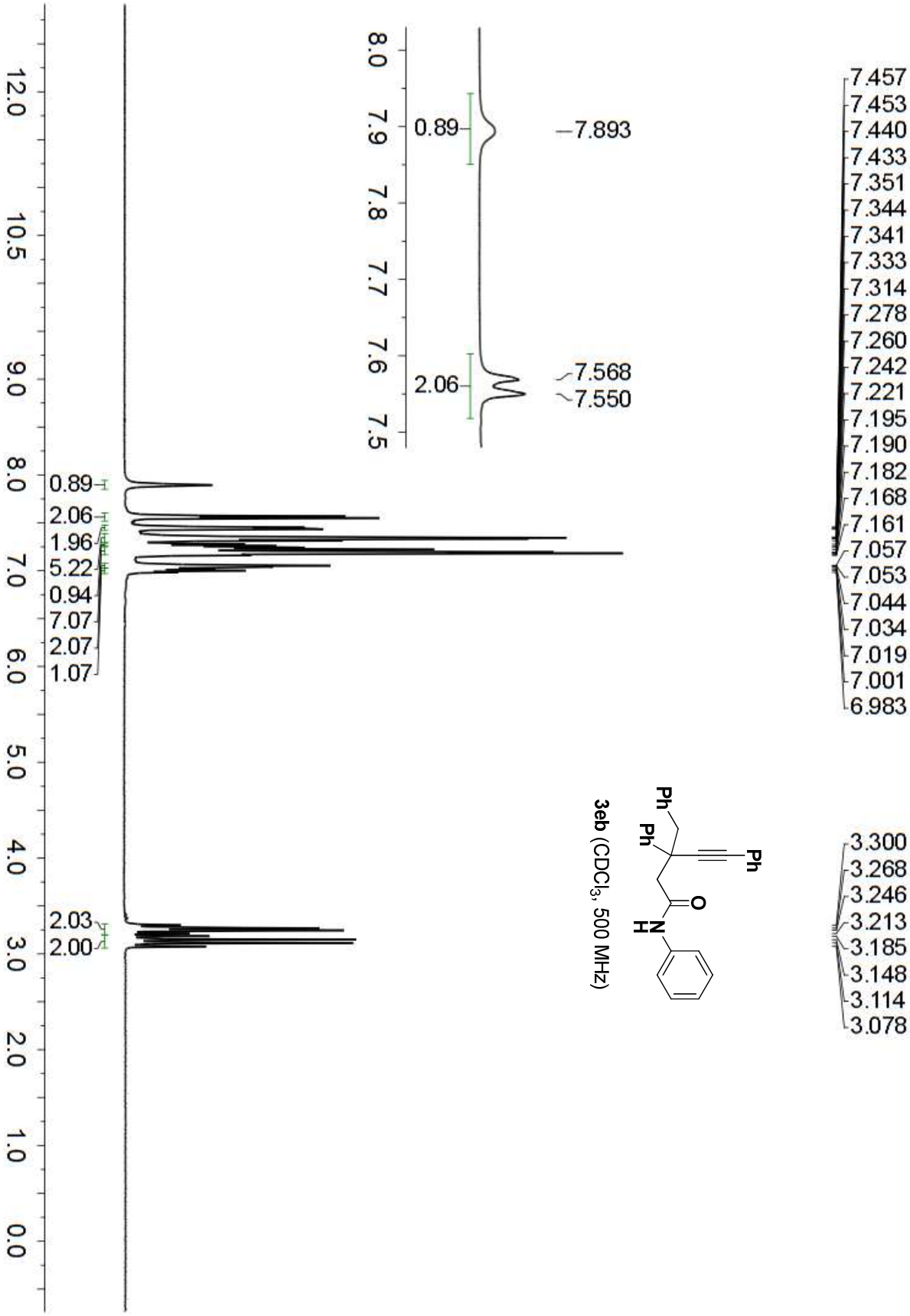


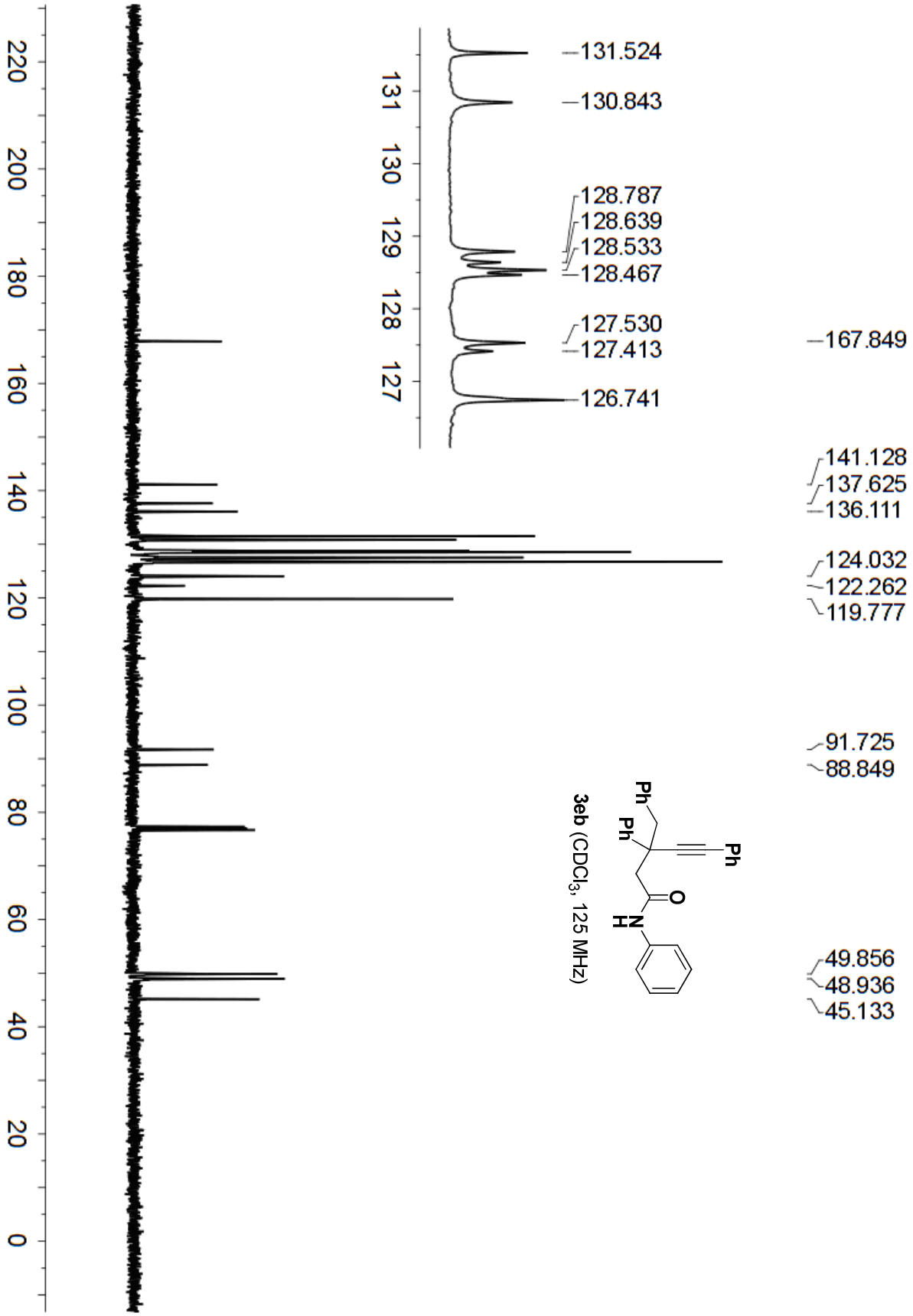
3cb ($CDCl_3$, 125 MHz)

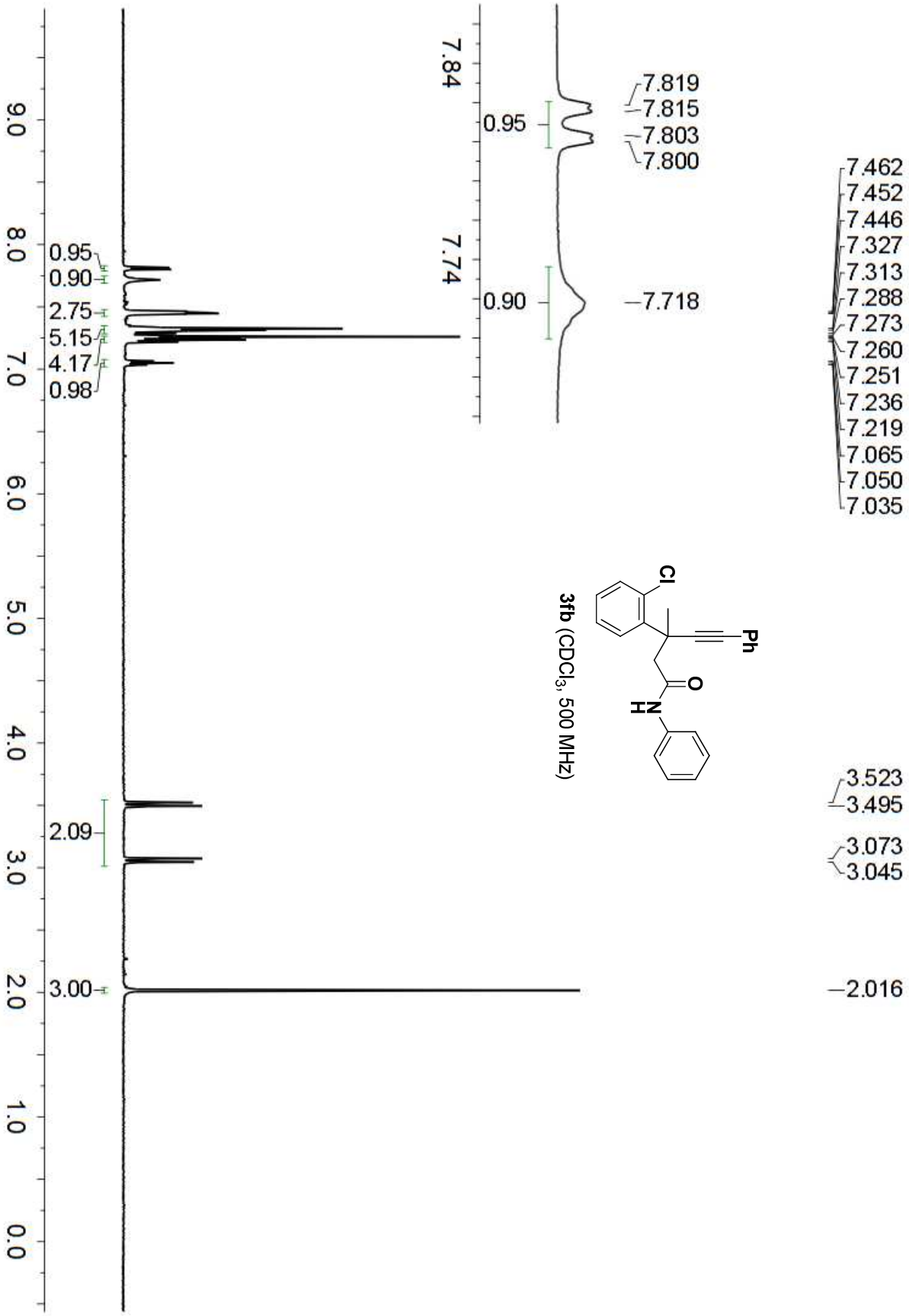


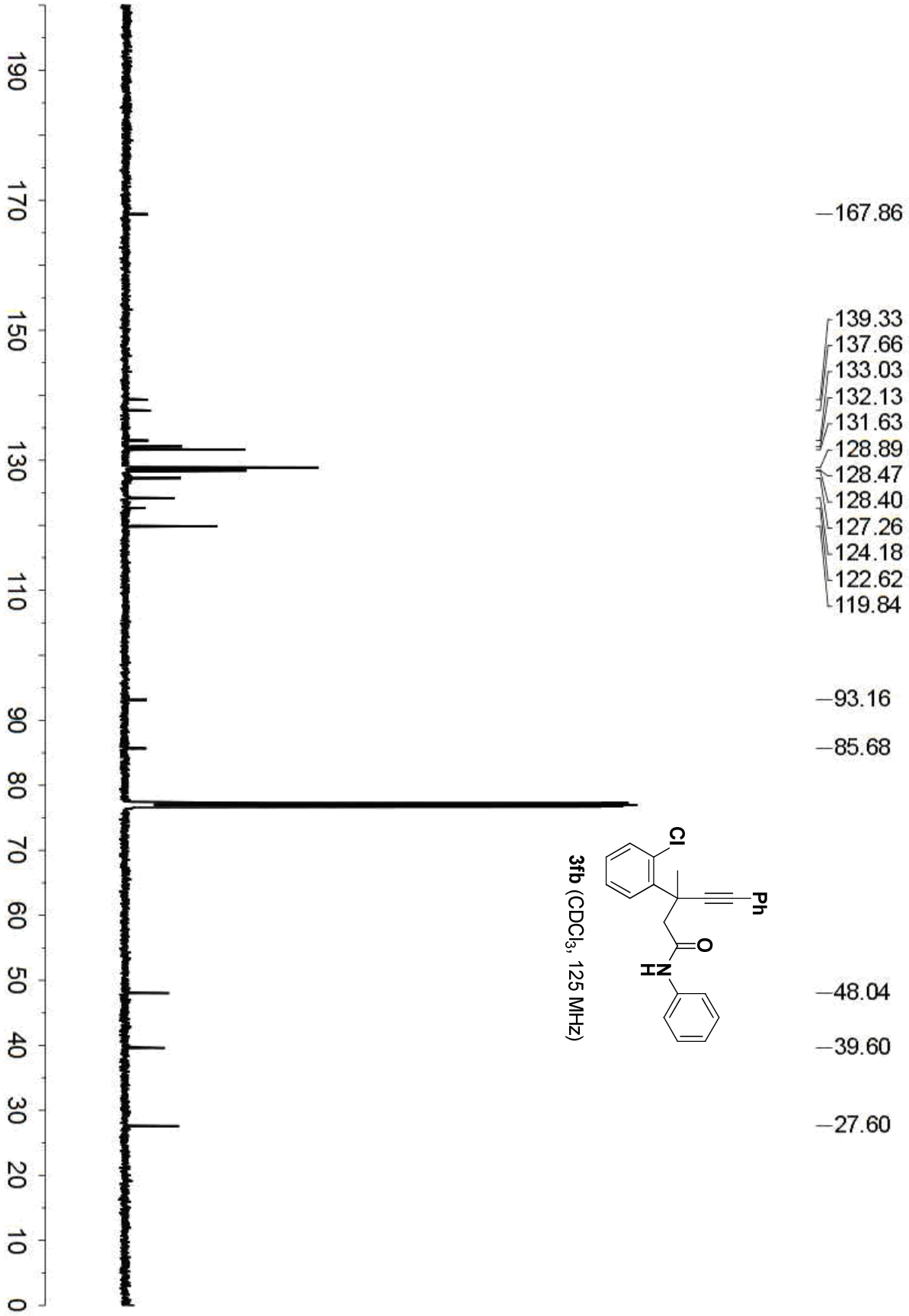
3db ($CDCl_3$, 500 MHz)


168.05
144.24
137.77
128.74
128.47
126.94
125.73
123.94
119.71
86.28
84.42
51.74
38.74
31.18
30.48
28.82
28.53
22.39
18.74
13.92
Ph
O
N
H
3db (CDCl3, 125 MHz)
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0

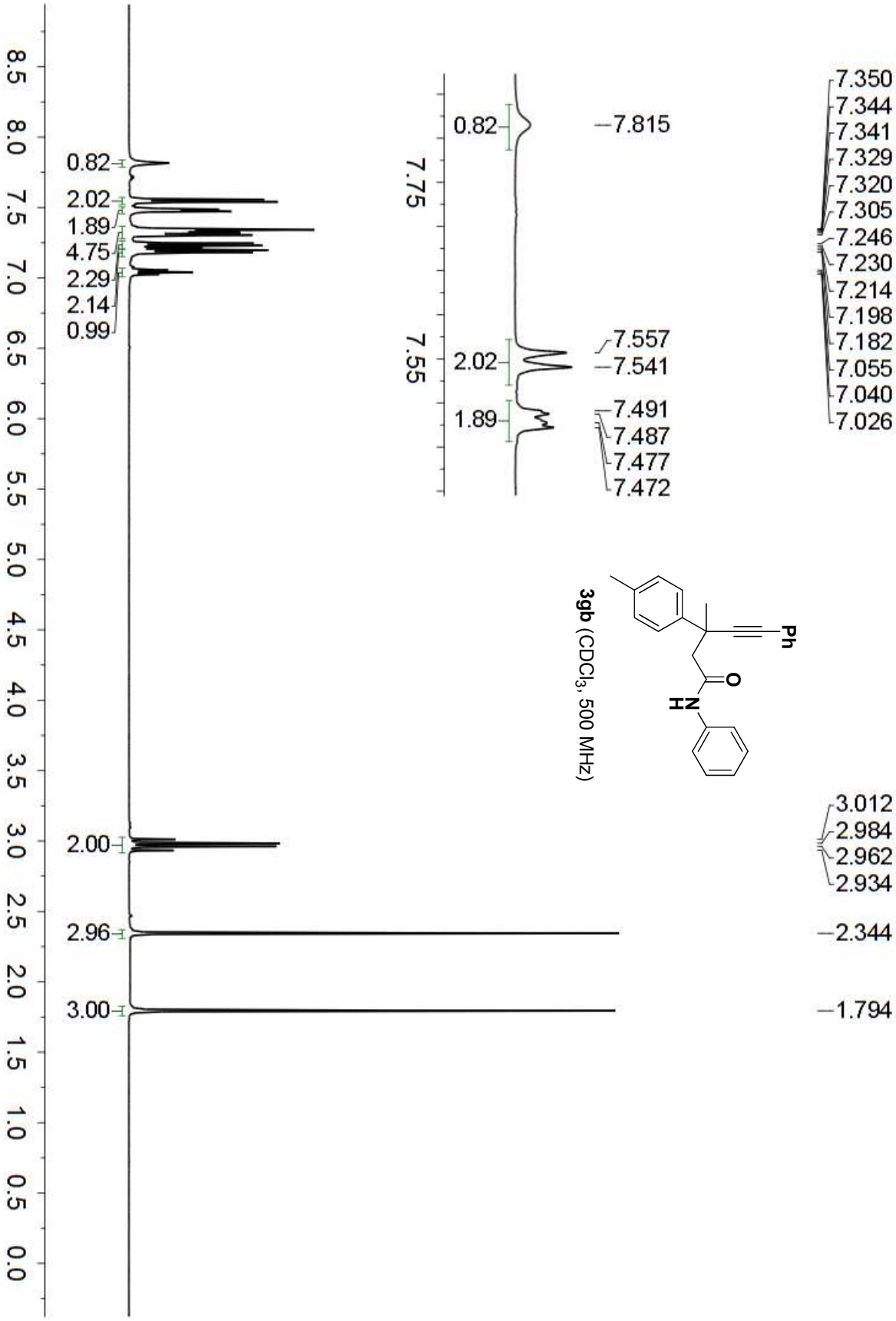

7.457
7.453
7.440
7.433
7.351
7.344
7.341
7.333
7.314
7.278
7.260
7.242
7.221
7.195
7.190
7.182
7.168
7.161
7.057
7.053
7.044
7.034
7.019
7.001
6.983
3.300
3.268
3.246
3.213
3.185
3.148
3.114
3.078
7.893
7.568
7.550
0.89
2.06
0.89
2.06
1.96
5.22
0.94
7.07
2.07
1.07
2.03
2.00
Ph
Ph
Ph
O
N
H
3eb (CDCl3, 500 MHz)

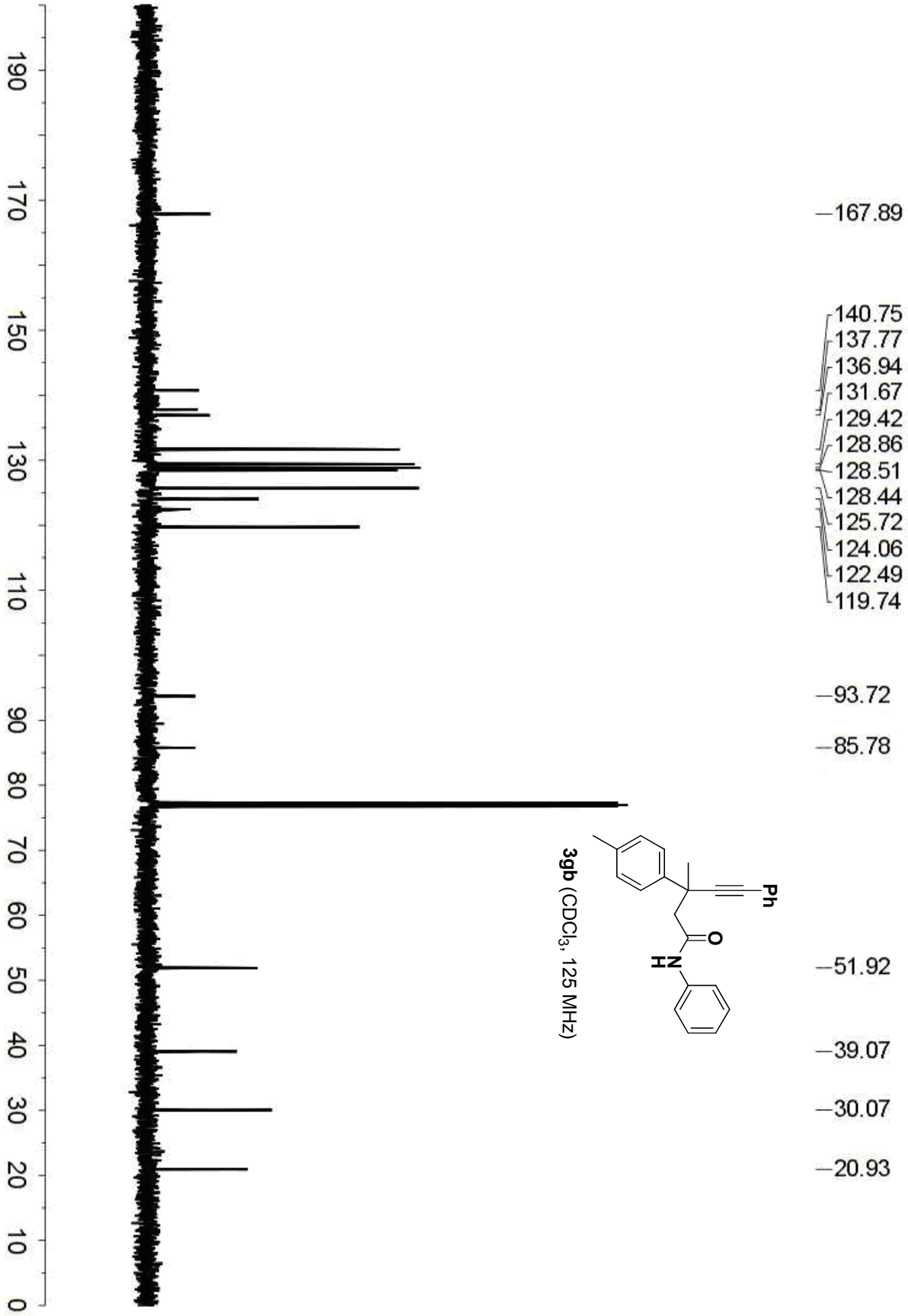

167.849
141.128
137.625
136.111
124.032
122.262
119.777
91.725
88.849
49.856
48.936
45.133
131.524
130.843
128.787
128.639
128.533
128.467
127.530
127.413
126.741
Ph
Ph
Ph
O
N
H
3eb (CDCl3, 125 MHz)


7.819
7.815
7.803
7.800
7.718
7.462
7.452
7.446
7.327
7.313
7.288
7.273
7.260
7.251
7.236
7.219
7.065
7.050
7.035
3.523
3.495
3.073
3.045
2.016
0.95
0.90
2.75
5.15
4.17
0.98
2.09
3.00
7.84
7.74
0.95
0.90
Cl
Ph
O
N
H
3fb (CDCl3, 500 MHz)
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0

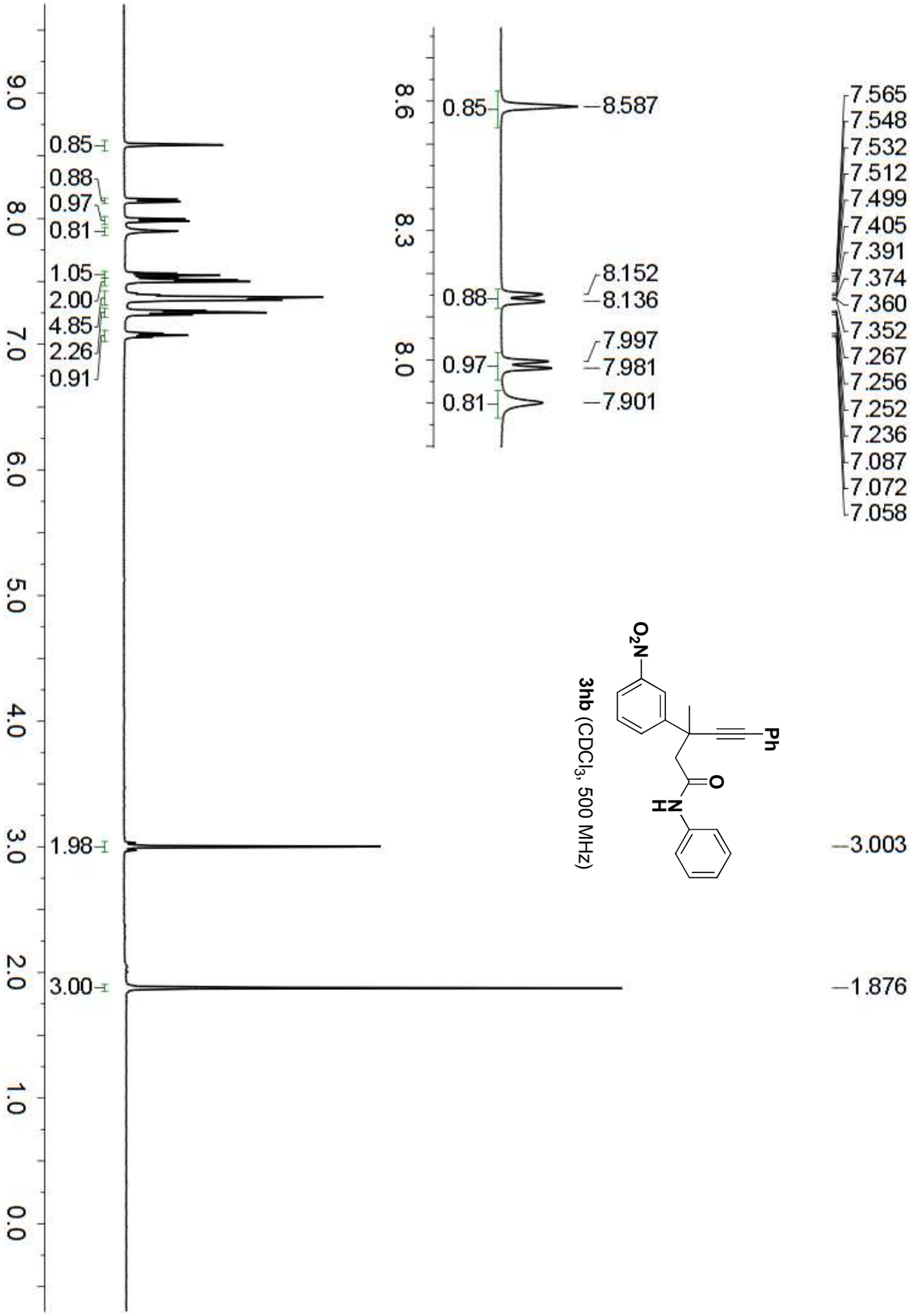


3fb ($CDCl_3$, 125 MHz)

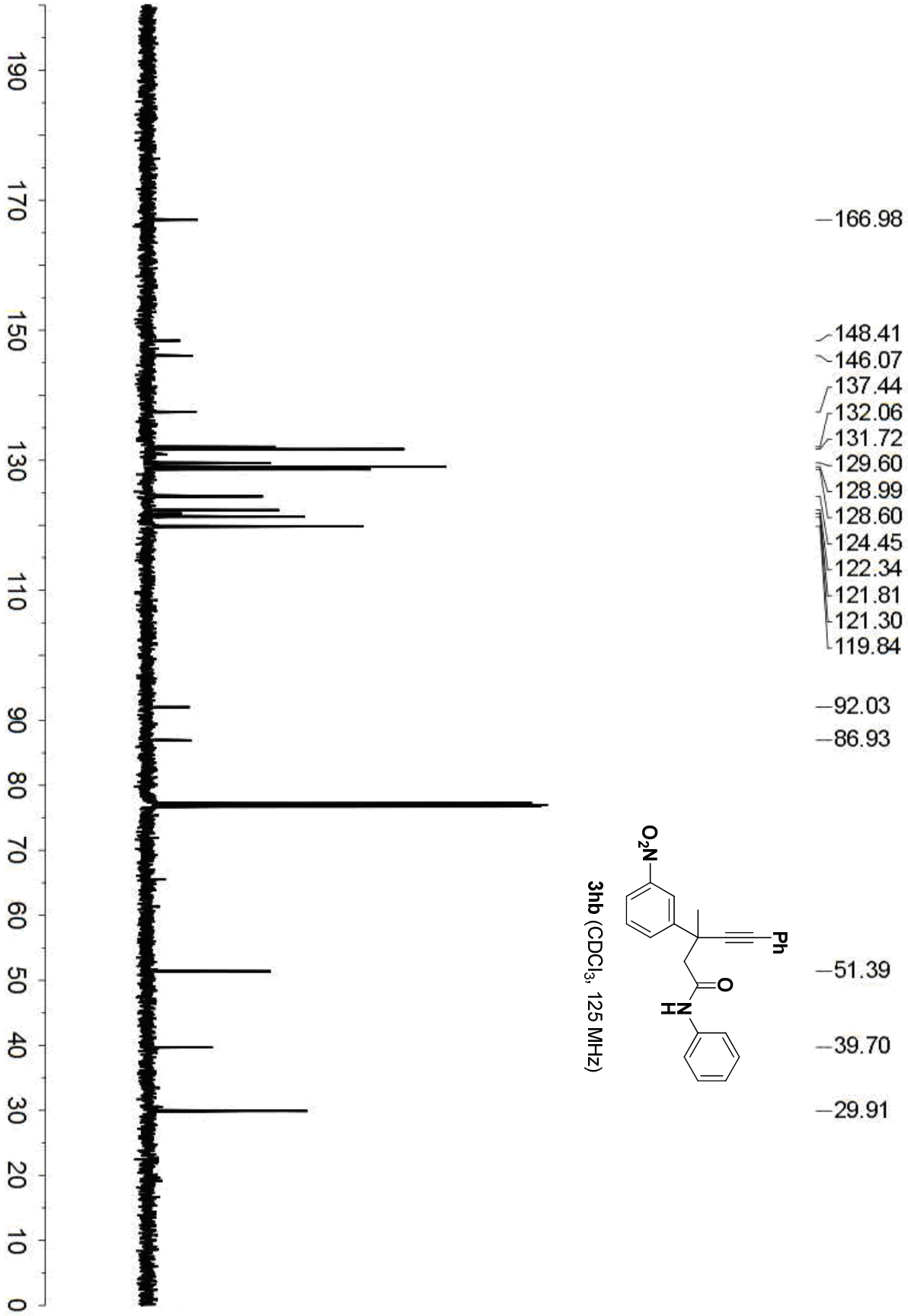

7.350
7.344
7.341
7.329
7.320
7.305
7.246
7.230
7.214
7.198
7.182
7.055
7.040
7.026
0.82
7.815
7.75
7.557
7.541
7.55
2.02
1.89
7.491
7.487
7.477
7.472
Ph
O
N
H
3gb (CDCl3, 500 MHz)
3.012
2.984
2.962
2.934
2.344
1.794
0.82
2.02
1.89
4.75
2.29
2.14
0.99
2.00
2.96
3.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

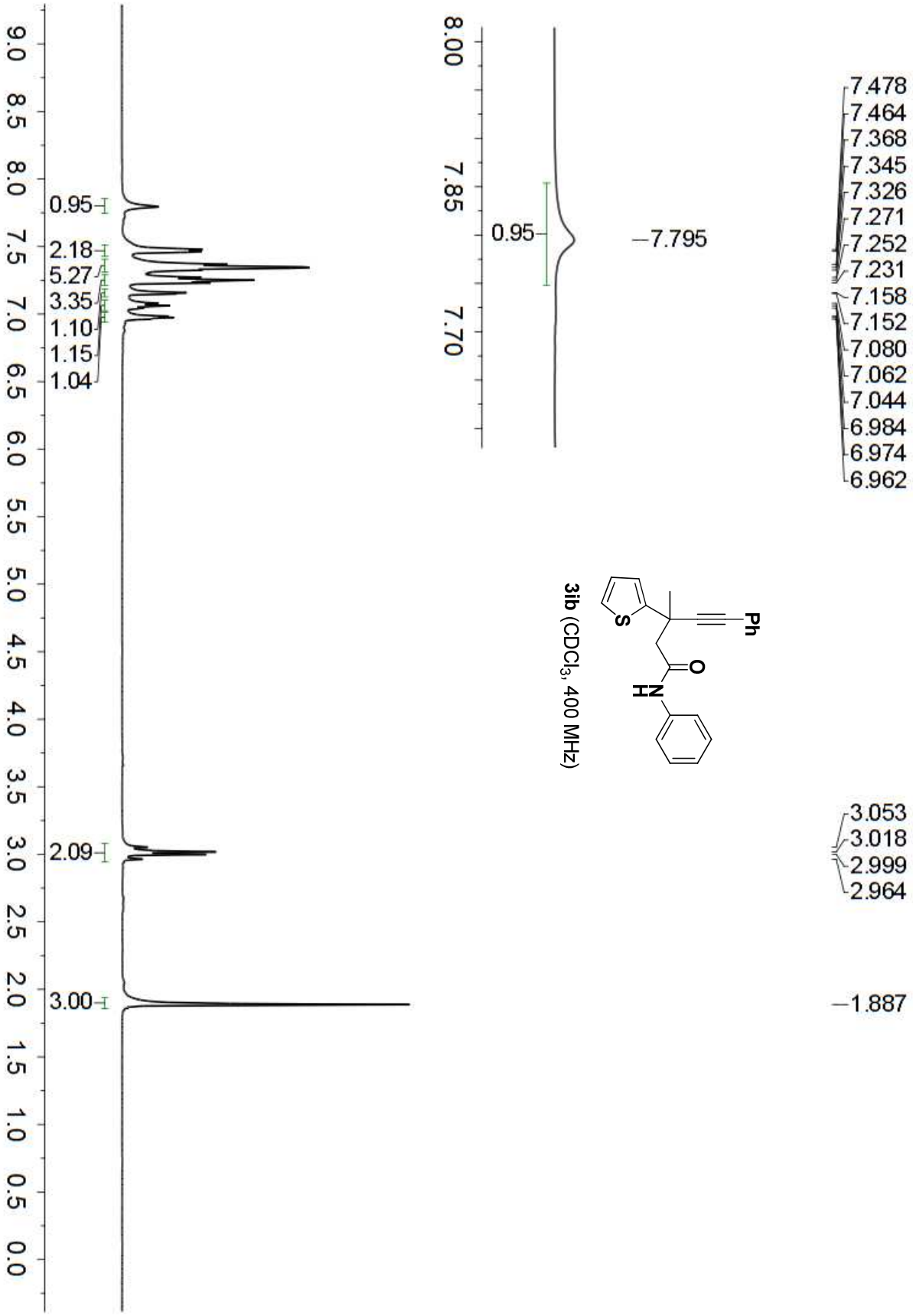


3gb ($CDCl_3$, 125 MHz)

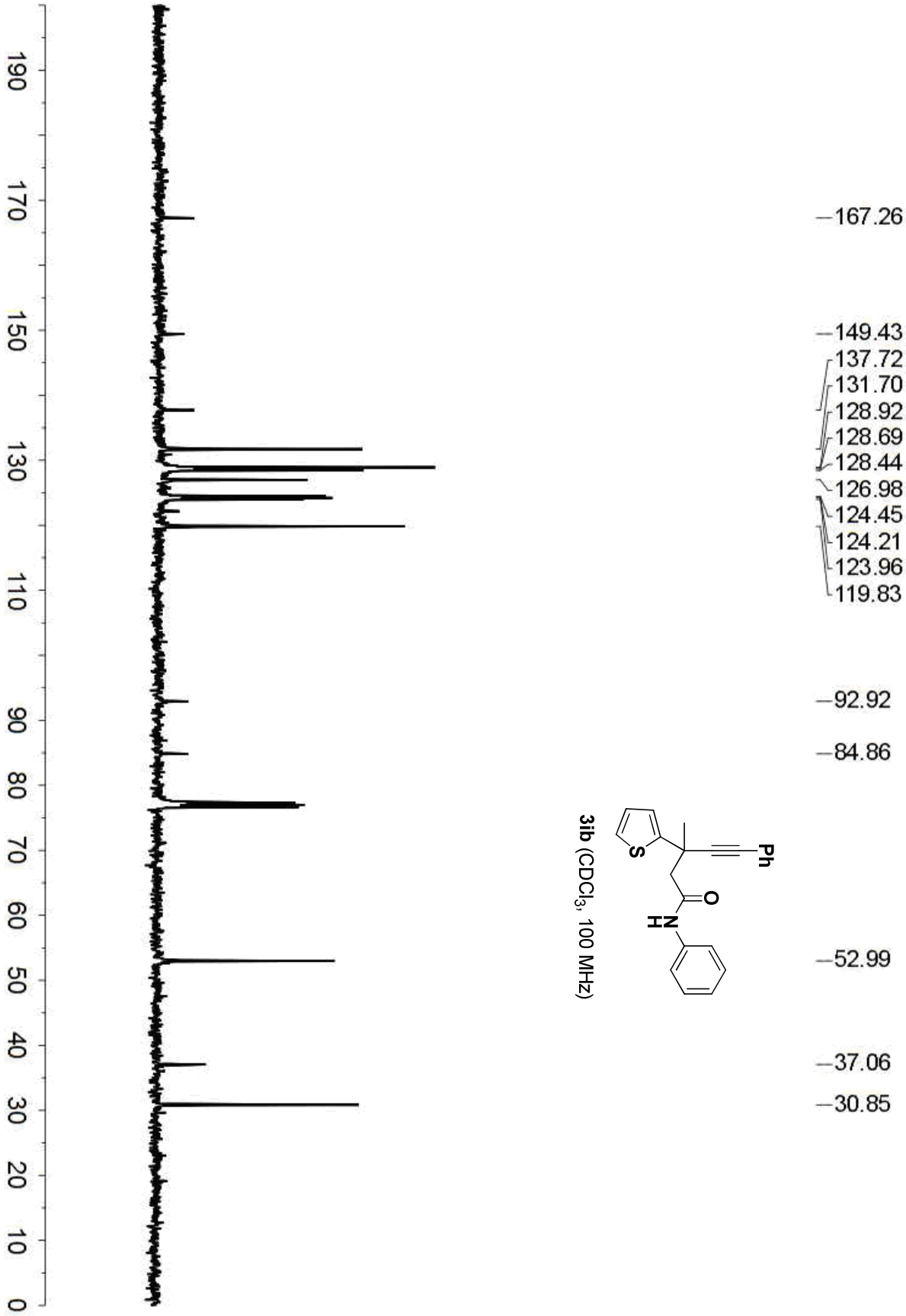


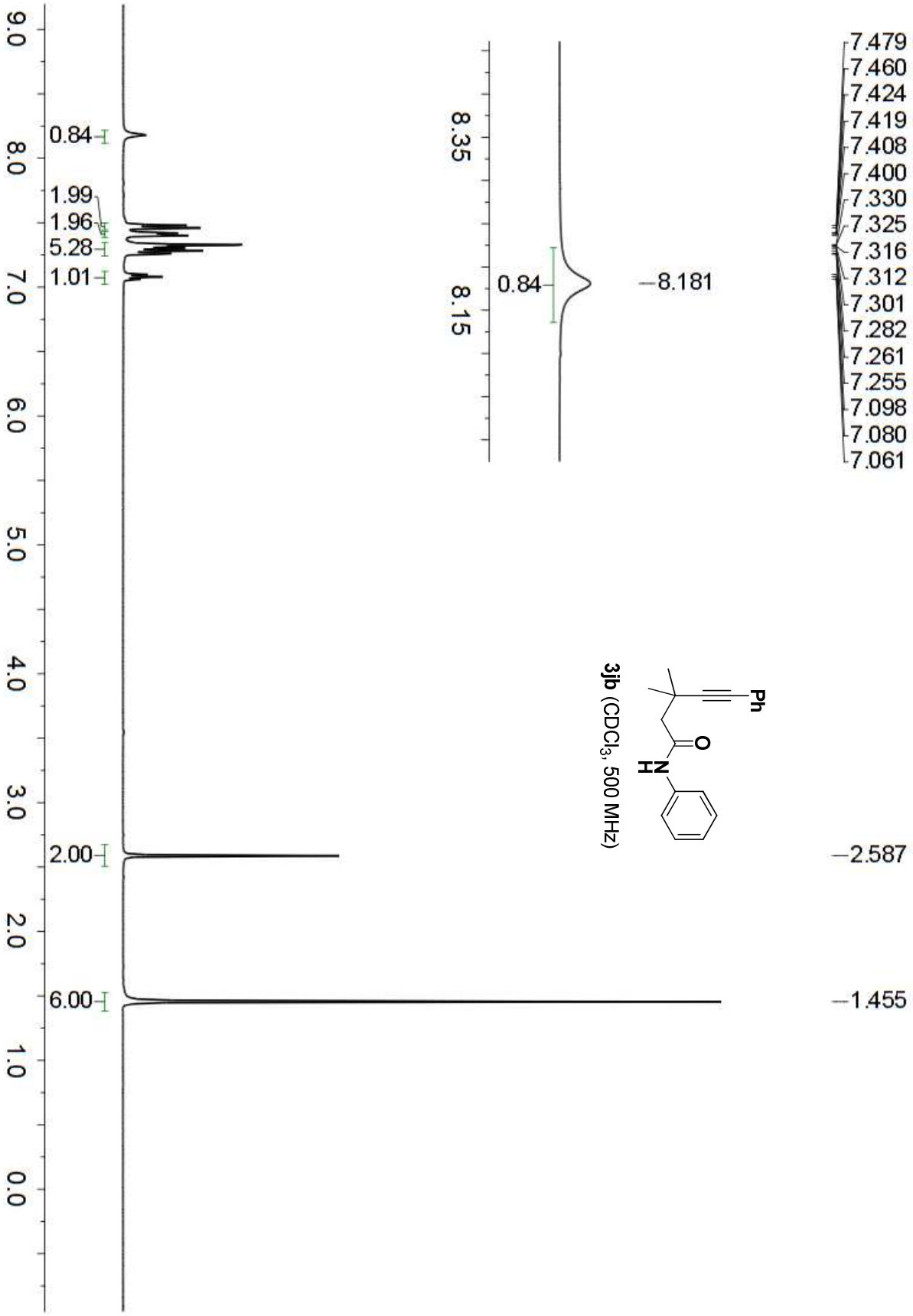
3hb ($CDCl_3$, 500 MHz)


166.98
148.41
146.07
137.44
132.06
131.72
129.60
128.99
128.60
124.45
122.34
121.81
121.30
119.84
92.03
86.93
51.39
39.70
29.91
O2N
Ph
O
N
H
3hb (CDCl3, 125 MHz)
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0

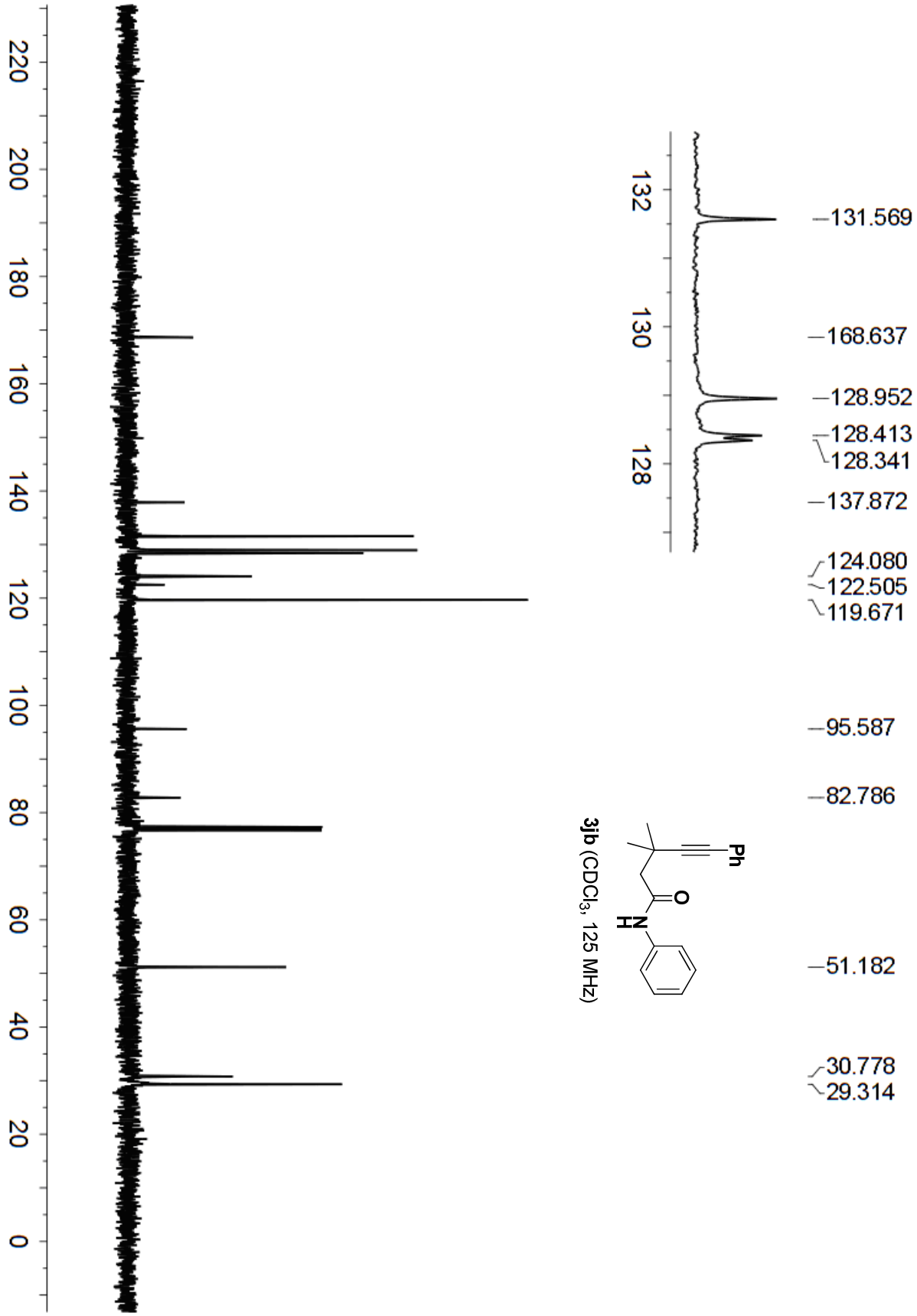


3ib ($CDCl_3$, 400 MHz)

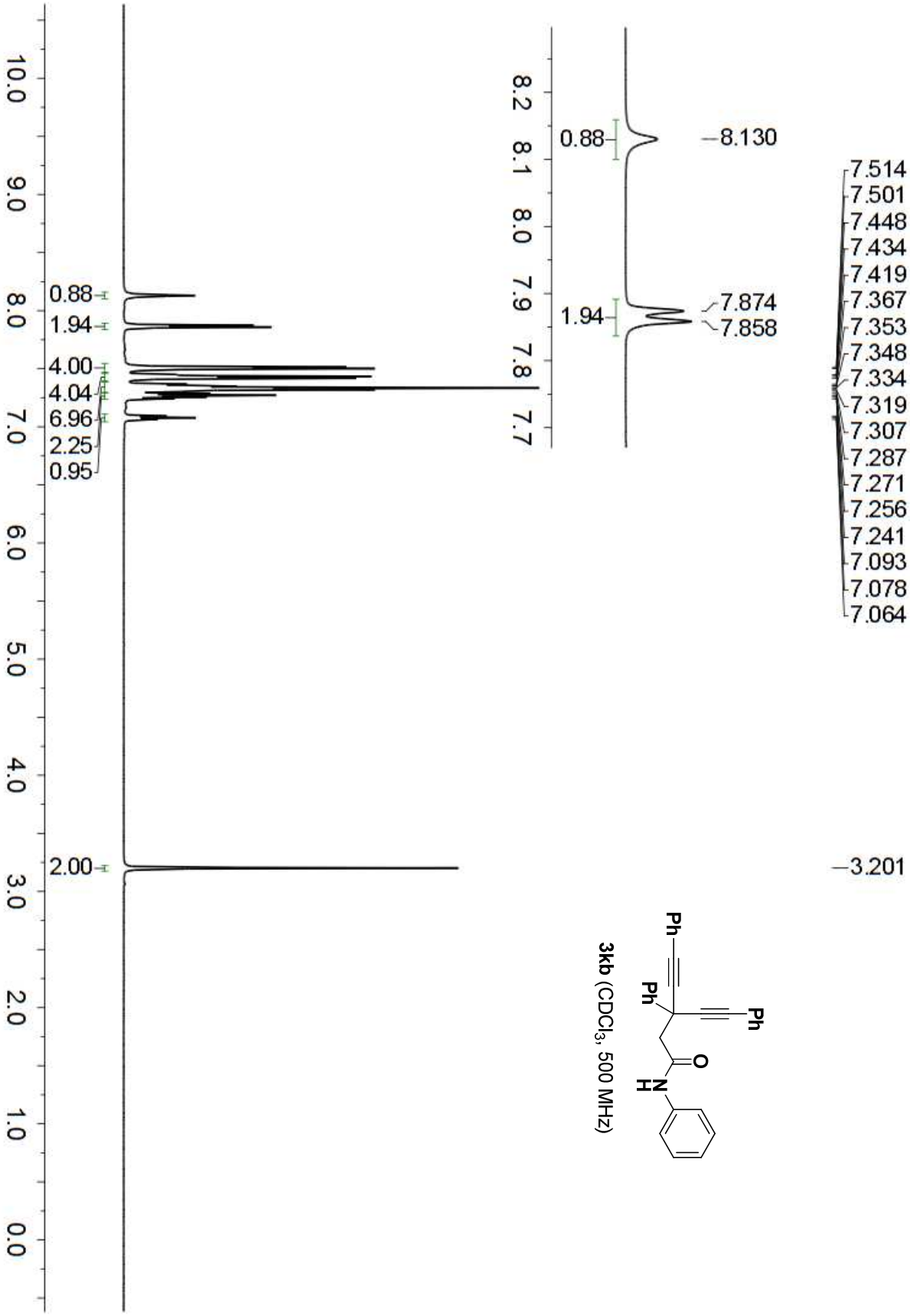

167.26
149.43
137.72
131.70
128.92
128.69
128.44
126.98
124.45
124.21
123.96
119.83
92.92
84.86
52.99
37.06
30.85
Ph
S
O
N
H
3ib (CDCl3, 100 MHz)
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0

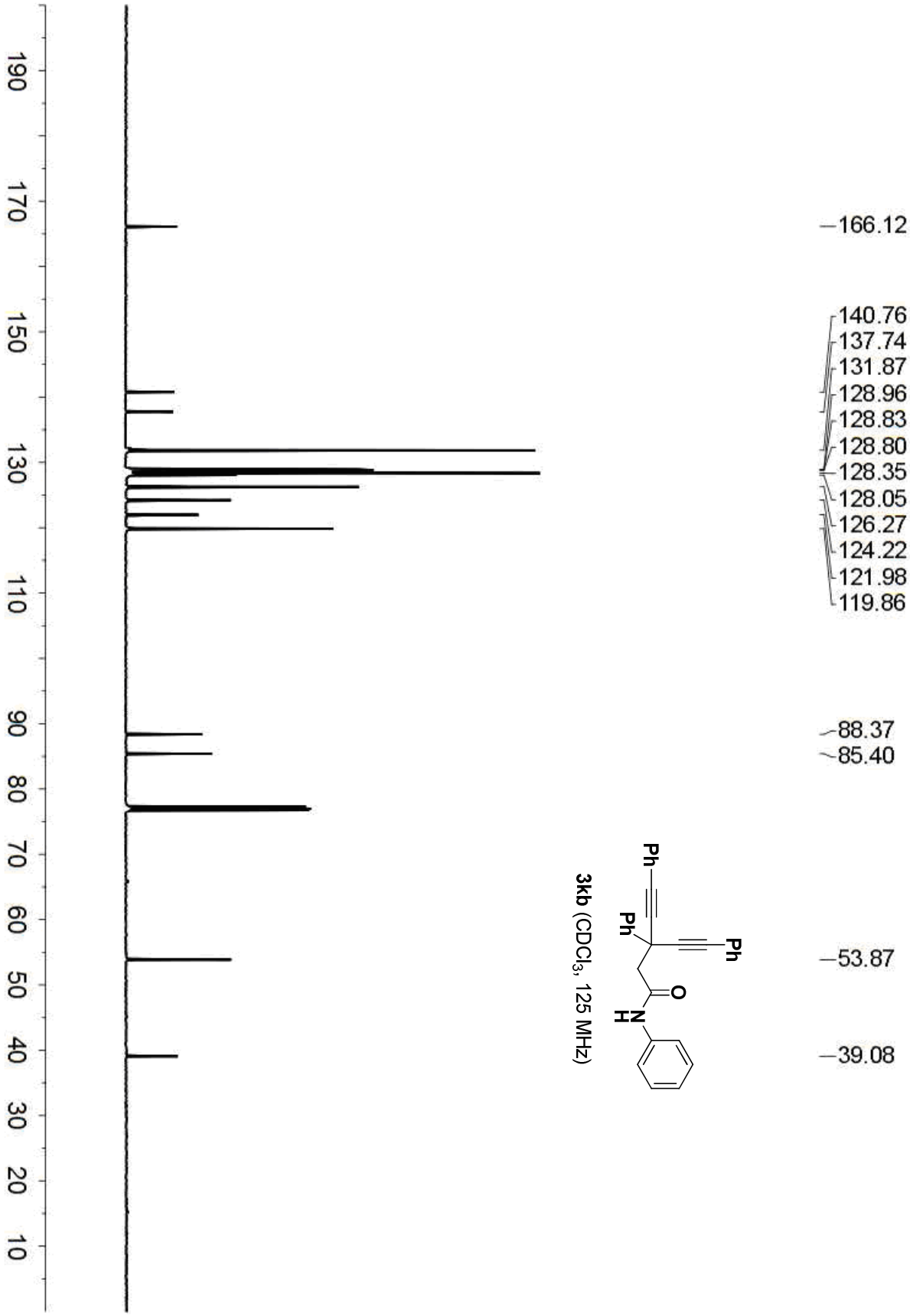


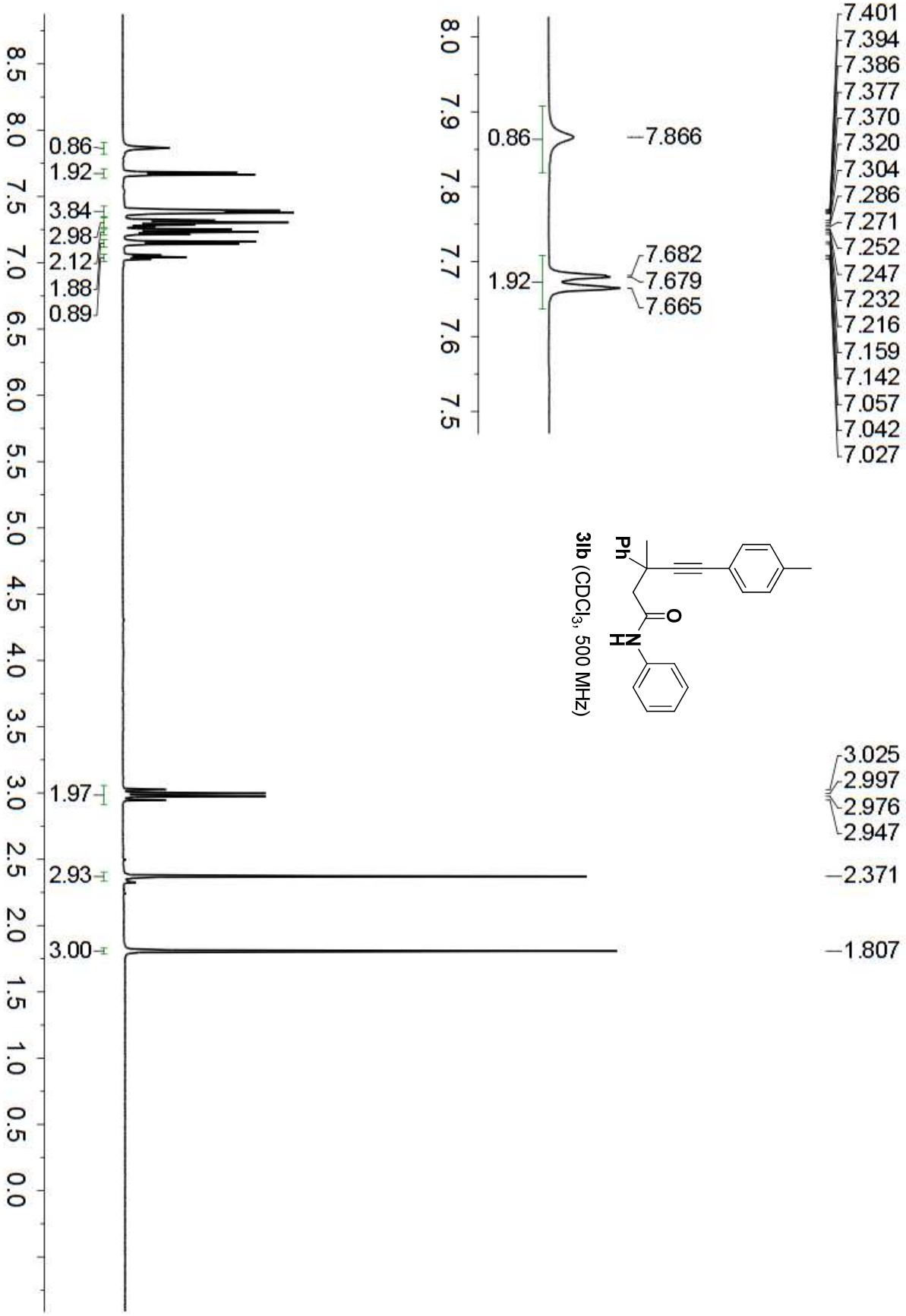
3jb ($CDCl_3$, 500 MHz)



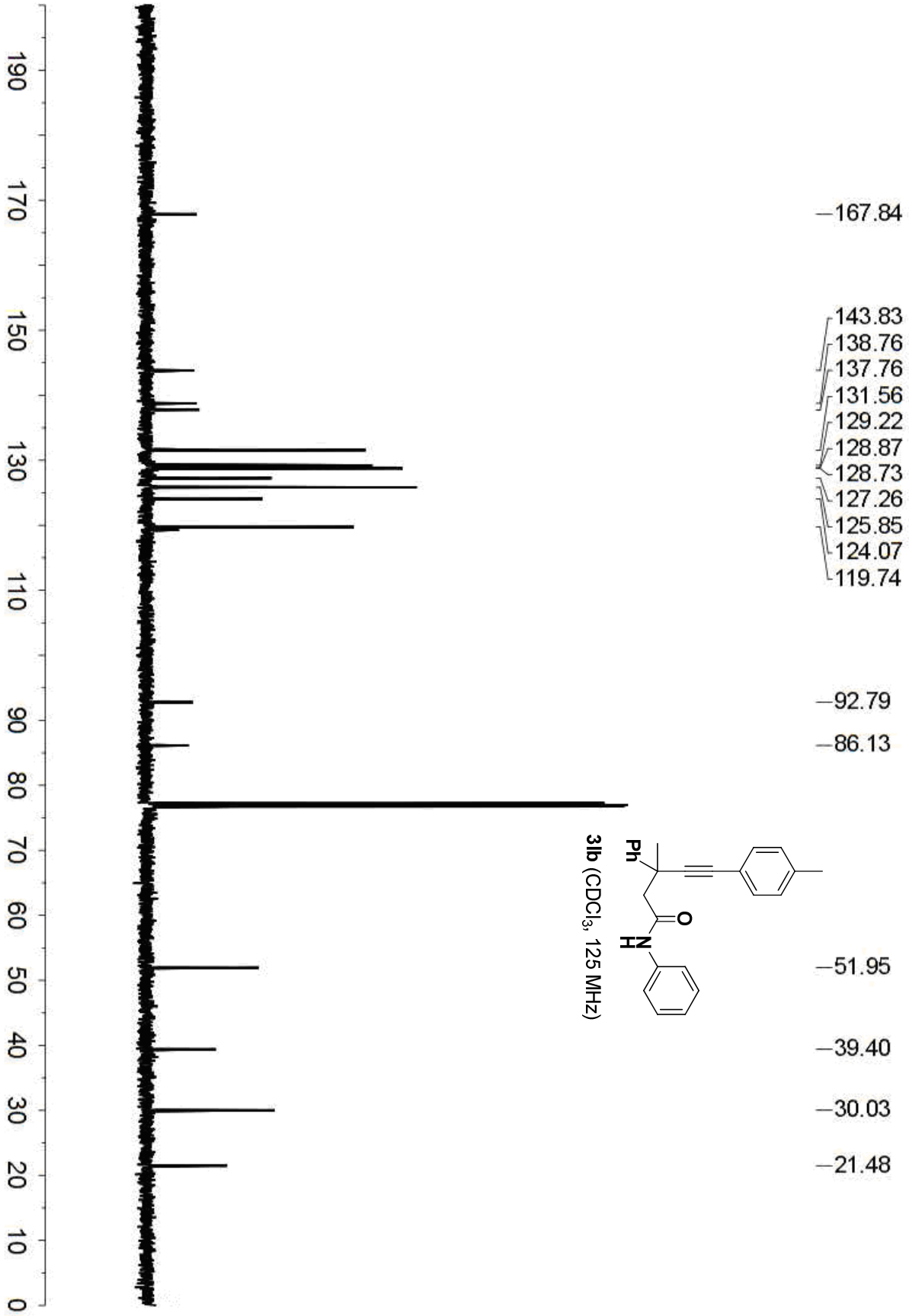
3jb ($CDCl_3$, 125 MHz)

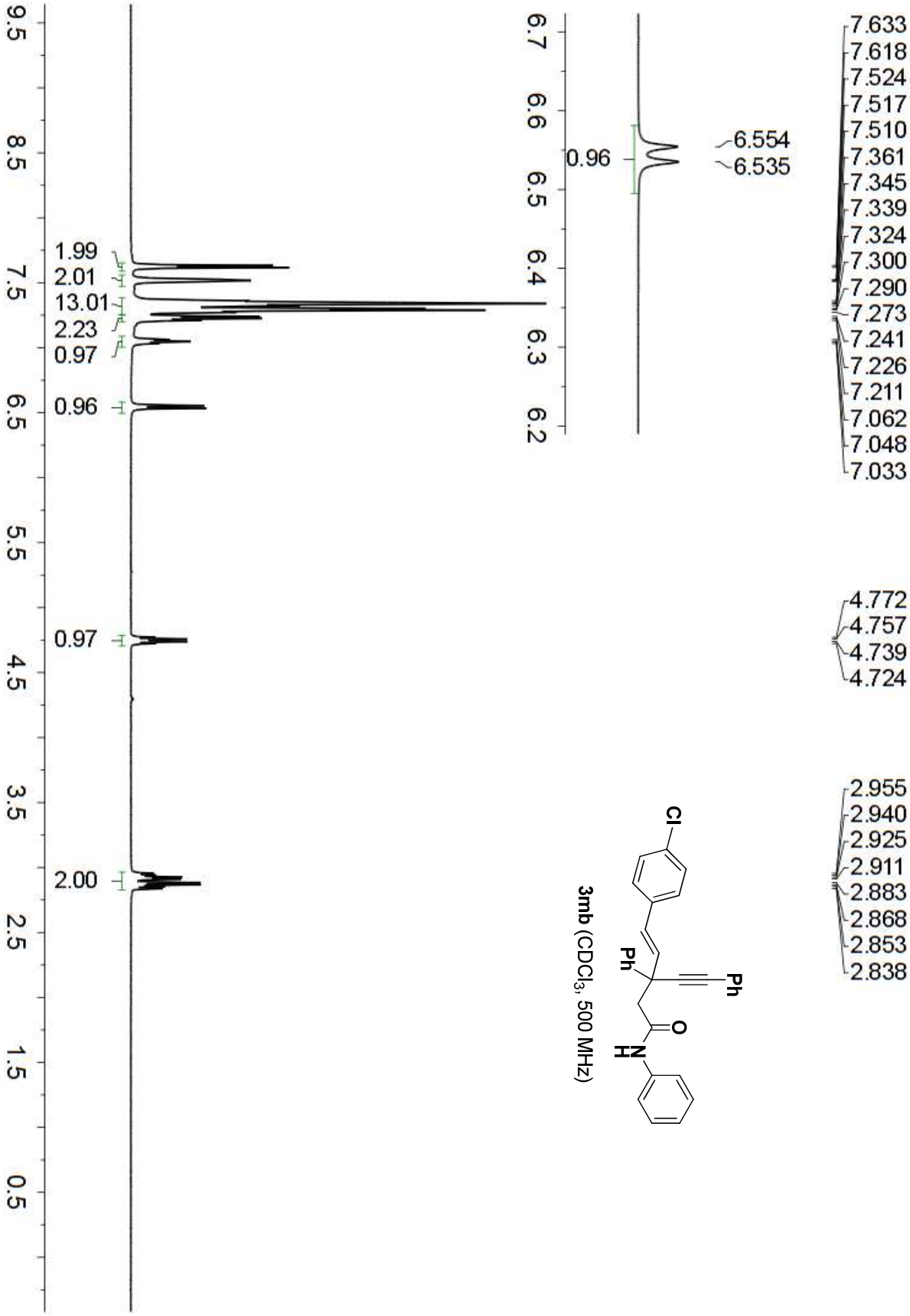

Ph
Ph
Ph
O
N
H
3kb (CDCl3, 500 MHz)
8.130
0.88
7.874
7.858
1.94
7.514
7.501
7.448
7.434
7.419
7.367
7.353
7.348
7.334
7.319
7.307
7.287
7.271
7.256
7.241
7.093
7.078
7.064
3.201
0.88
1.94
4.00
4.04
6.96
2.25
0.95
2.00


166.12
140.76
137.74
131.87
128.96
128.83
128.80
128.35
128.05
126.27
124.22
121.98
119.86
88.37
85.40
53.87
39.08
Ph
Ph
Ph
O
N
H
3kb (CDCl3, 125 MHz)
190
170
150
130
110
90
80
70
60
50
40
30
20
10

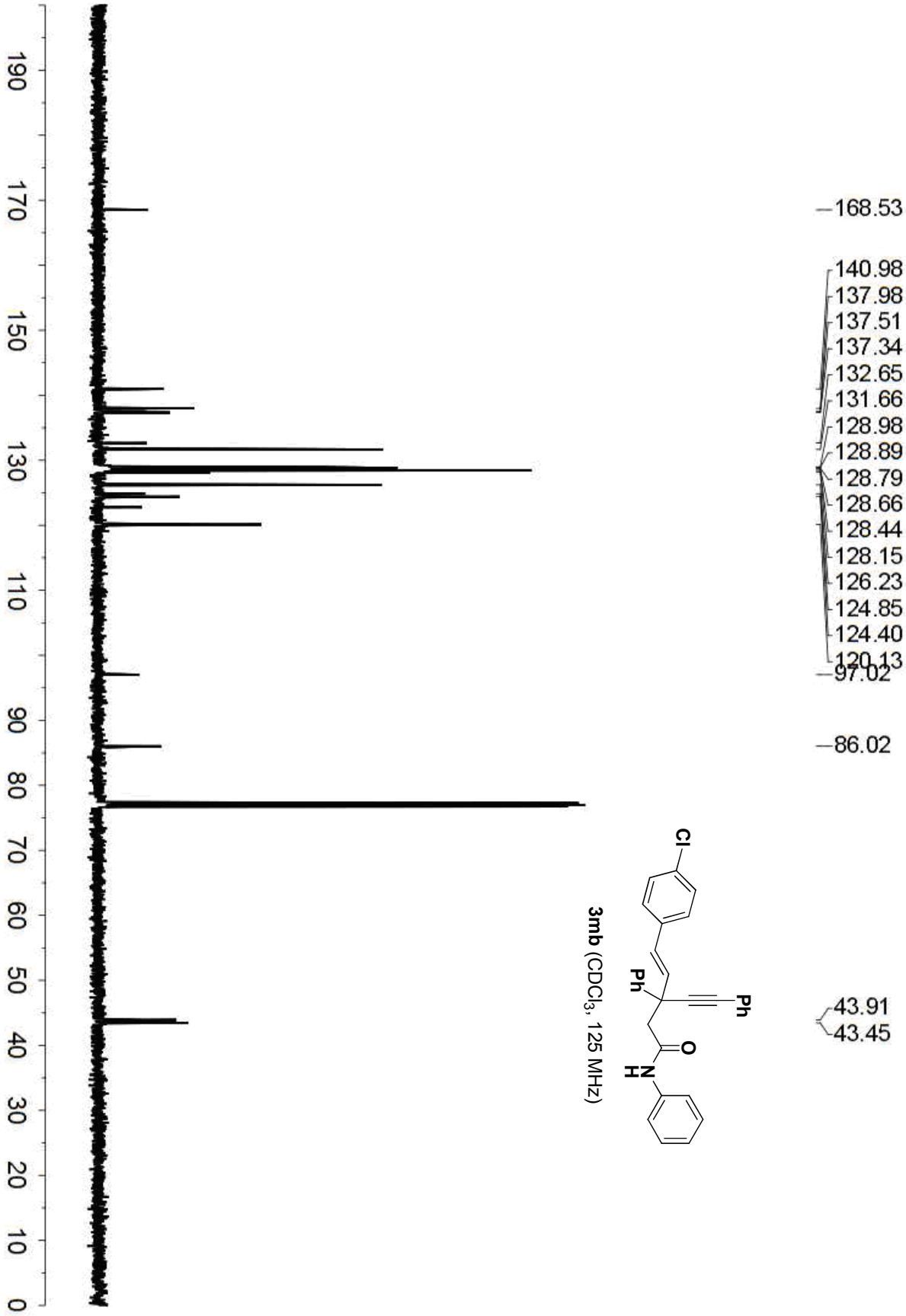


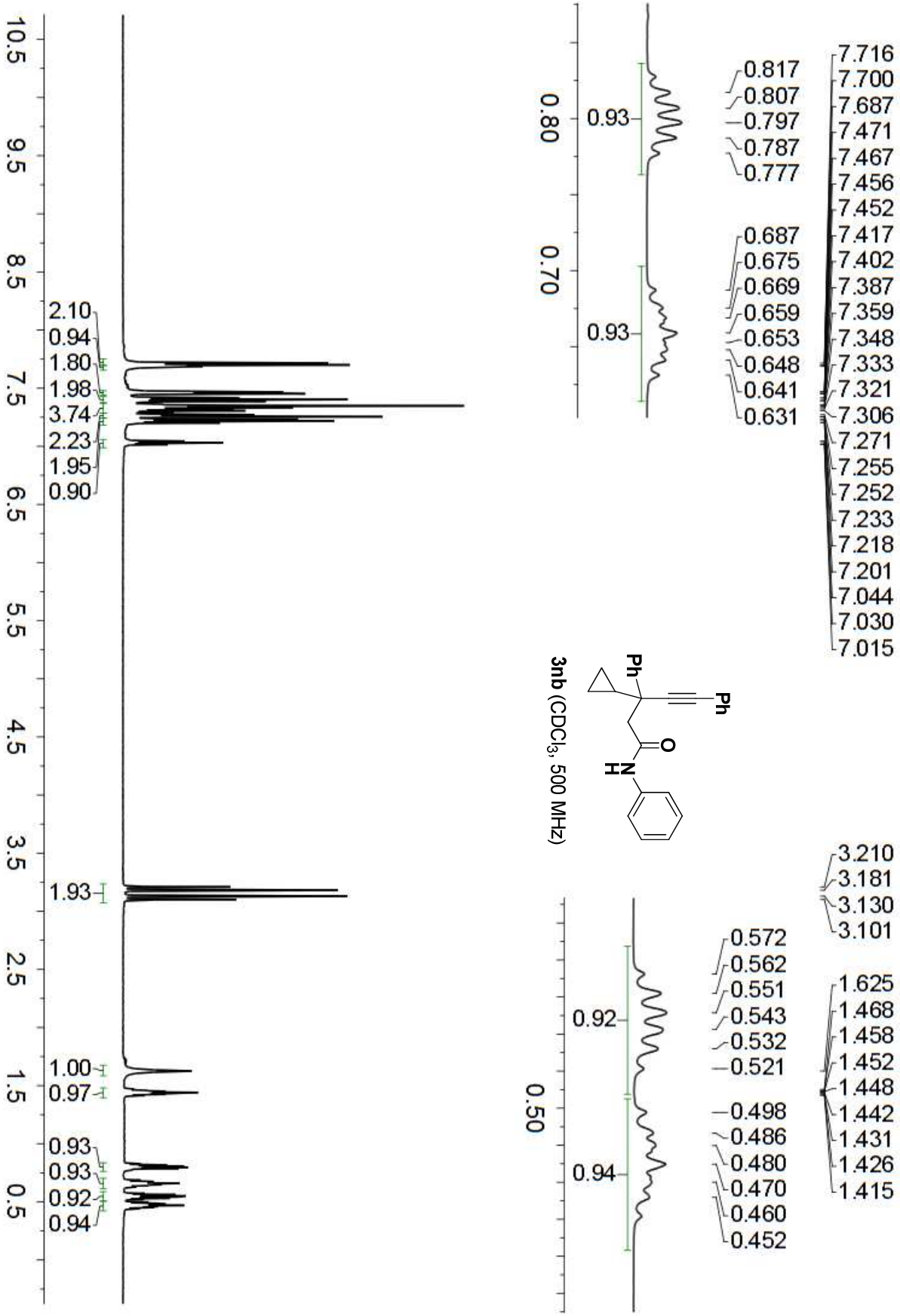
3lb ($CDCl_3$, 500 MHz)


167.84
143.83
138.76
137.76
131.56
129.22
128.87
128.73
127.26
125.85
124.07
119.74
92.79
86.13
51.95
39.40
30.03
21.48
Ph
O
H
N
3lb (CDCl3, 125 MHz)
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0

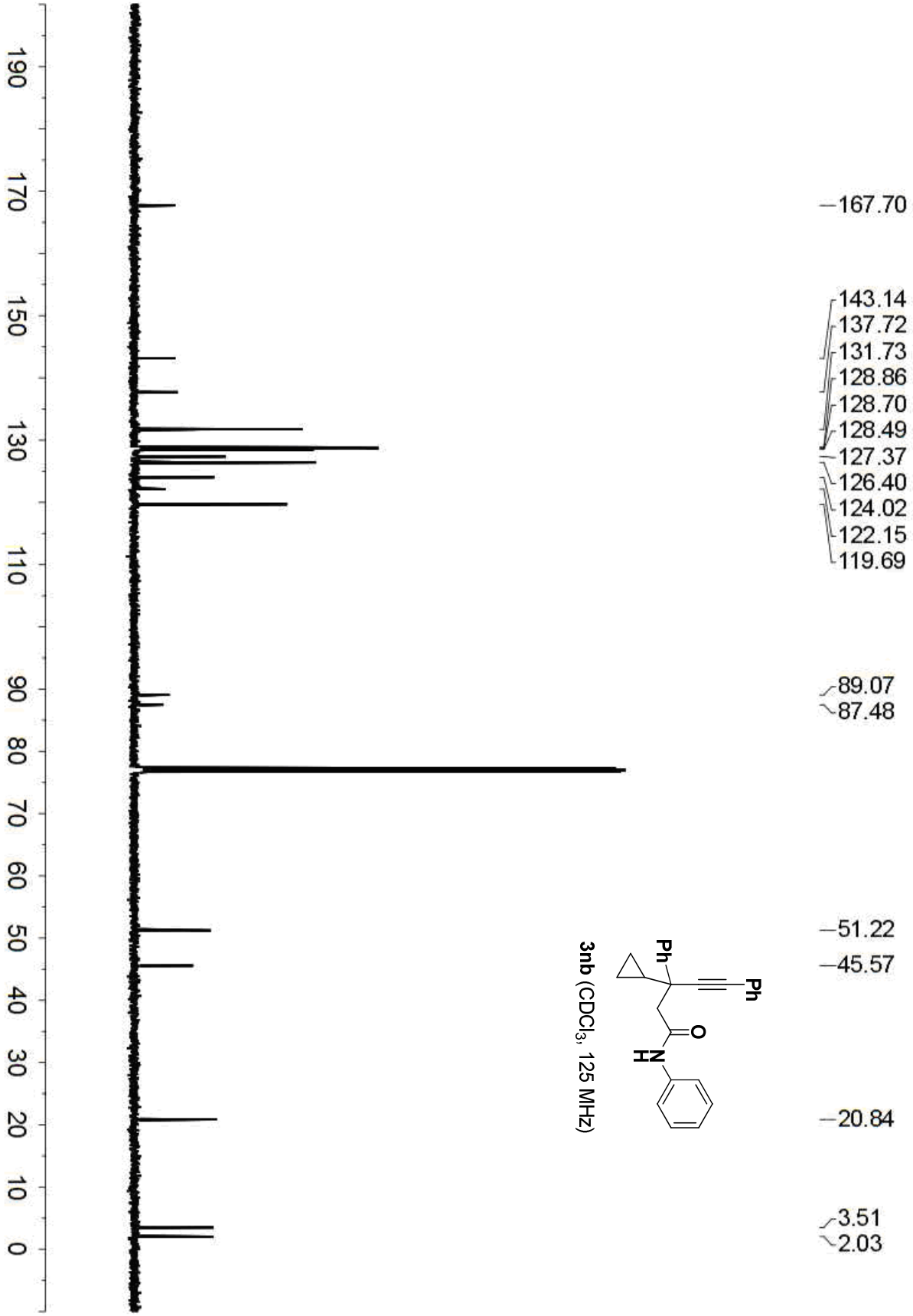


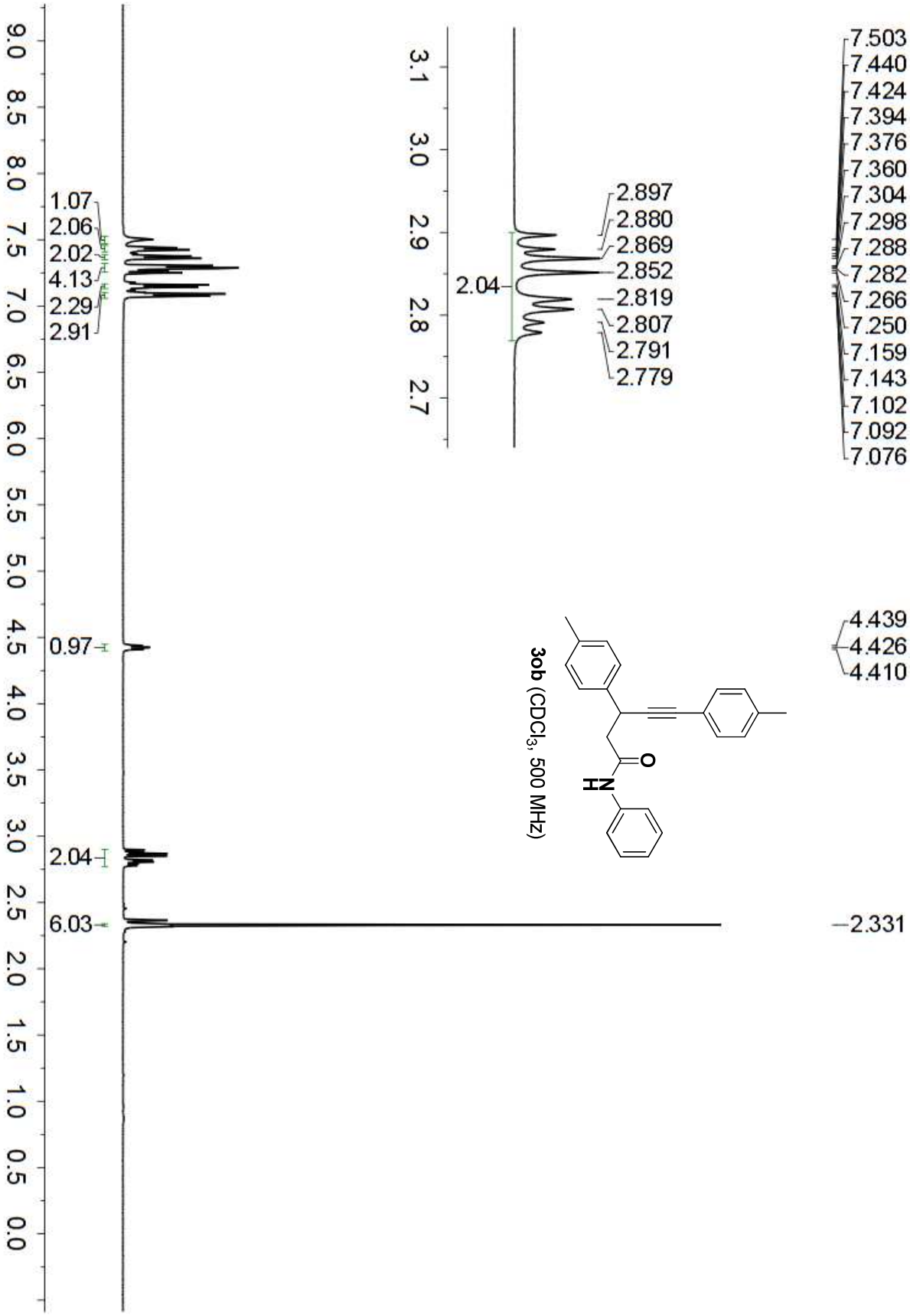
3mb ($CDCl_3$, 500 MHz)

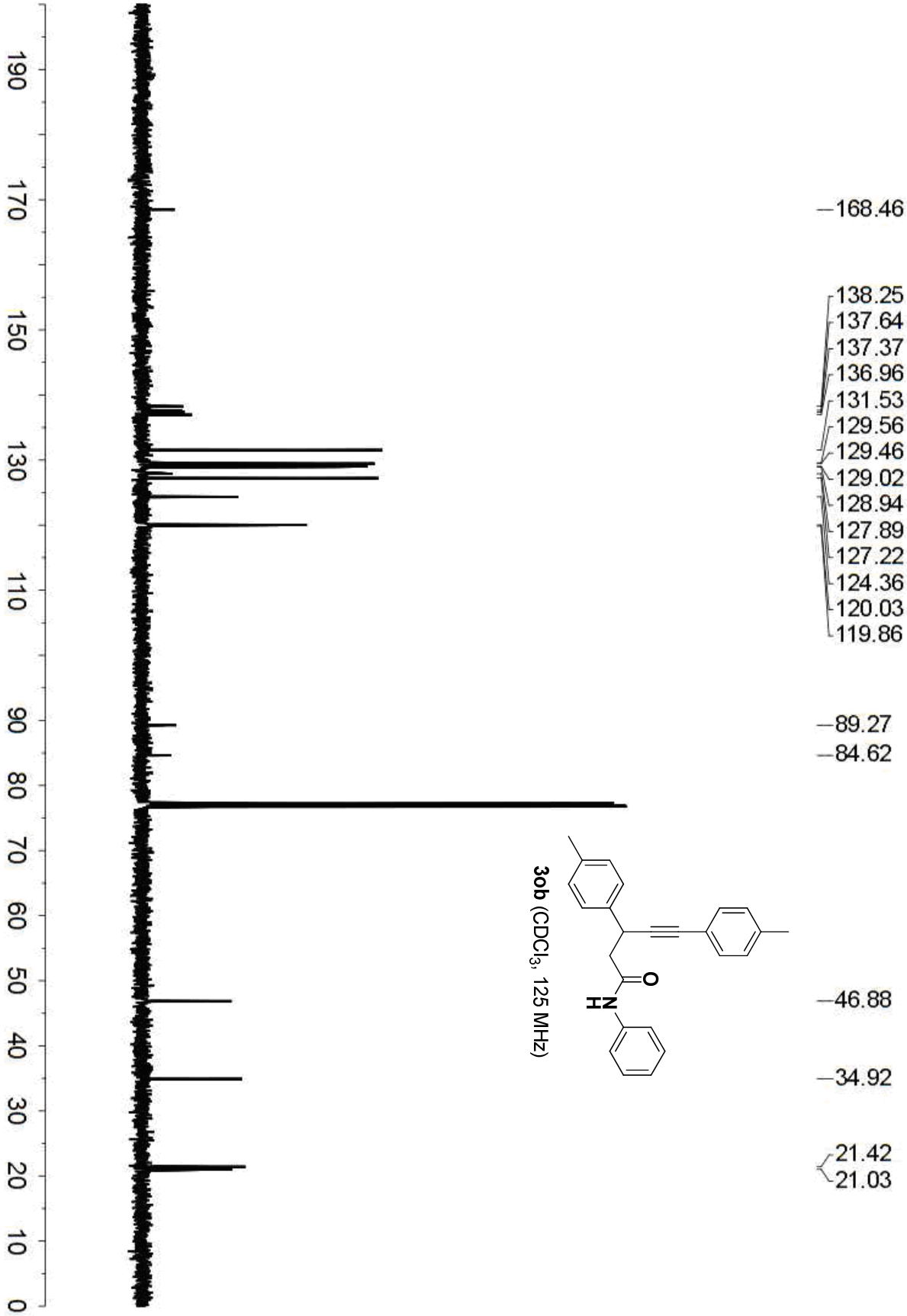

168.53
140.98
137.98
137.51
137.34
132.65
131.66
128.98
128.89
128.79
128.66
128.44
128.15
126.23
124.85
124.40
120.13
97.02
86.02
43.91
43.45
Cl
Ph
Ph
O
N
H
3mb (CDCl3, 125 MHz)
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0



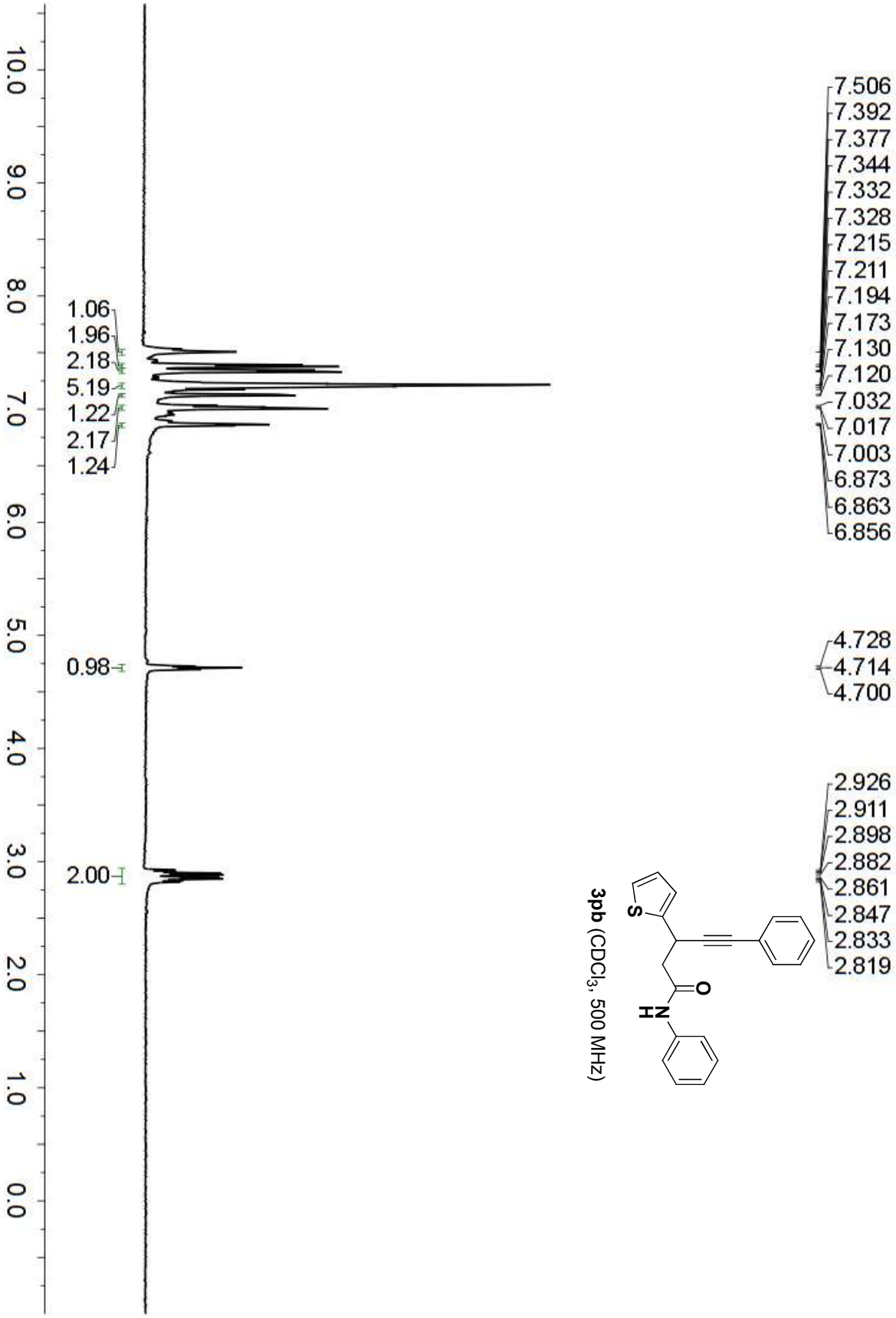
3nb ($CDCl_3$, 500 MHz)


167.70
143.14
137.72
131.73
128.86
128.70
128.49
127.37
126.40
124.02
122.15
119.69
89.07
87.48
51.22
45.57
20.84
3.51
2.03
Ph
Ph
O
N
H
3nb (CDCl3, 125 MHz)
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0

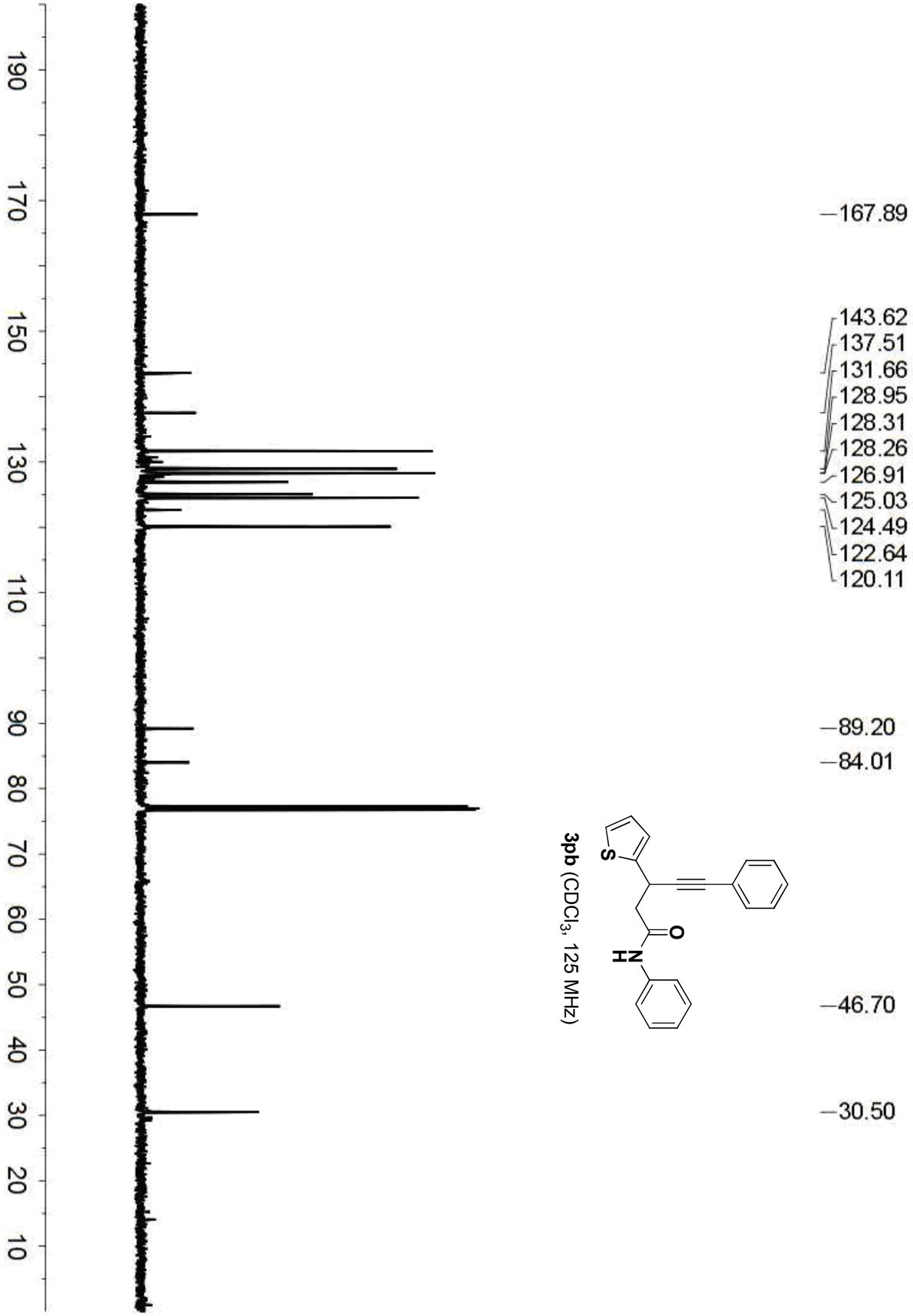

7.503
7.440
7.424
7.394
7.376
7.360
7.304
7.298
7.288
7.282
7.266
7.250
7.159
7.143
7.102
7.092
7.076
4.439
4.426
4.410
2.331
2.897
2.880
2.869
2.852
2.819
2.807
2.791
2.779
2.04
1.07
2.06
2.02
4.13
2.29
2.91
0.97
2.04
6.03
3ob (CDCl3, 500 MHz)
O
H
N



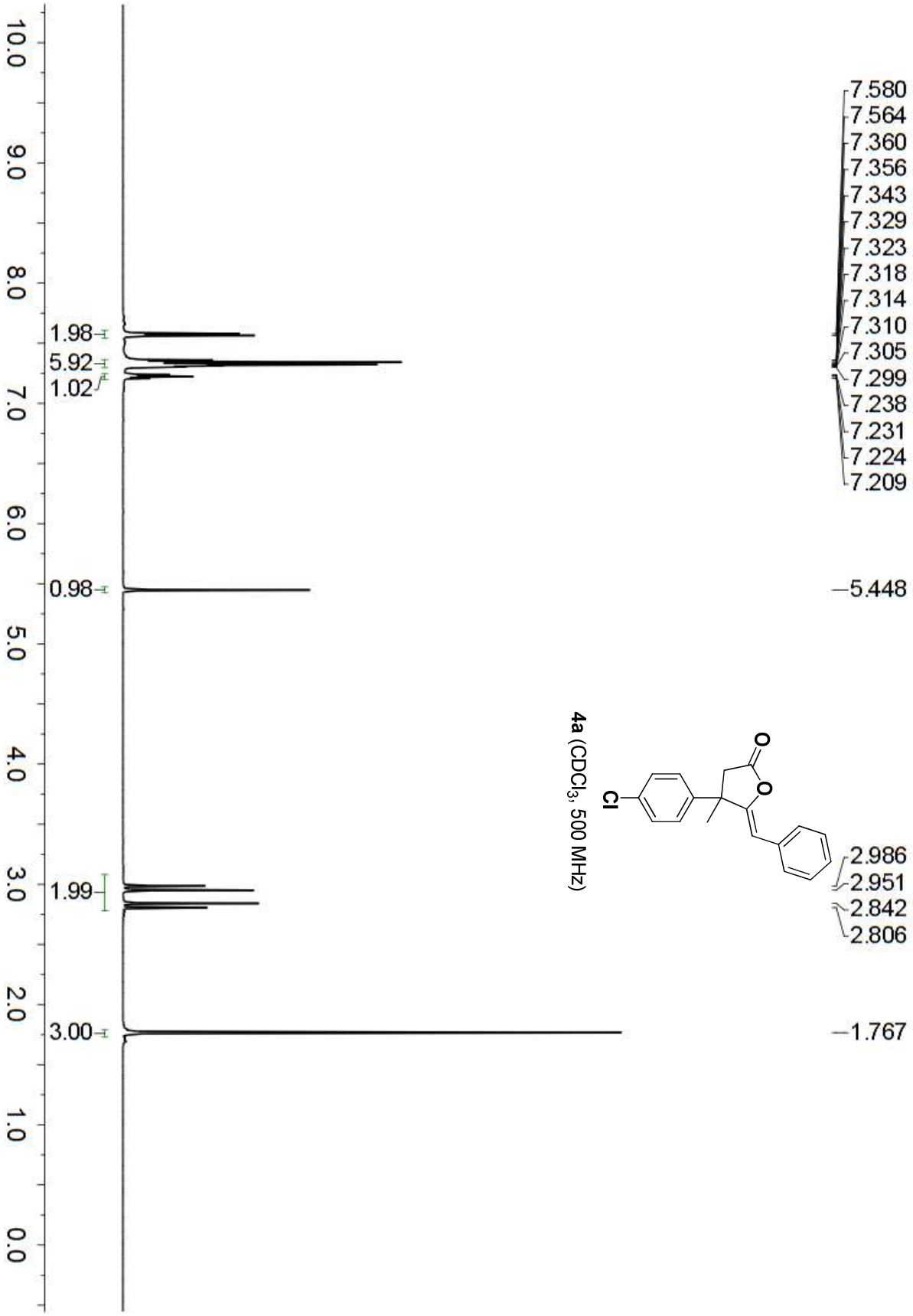
3ob ($CDCl_3$, 125 MHz)



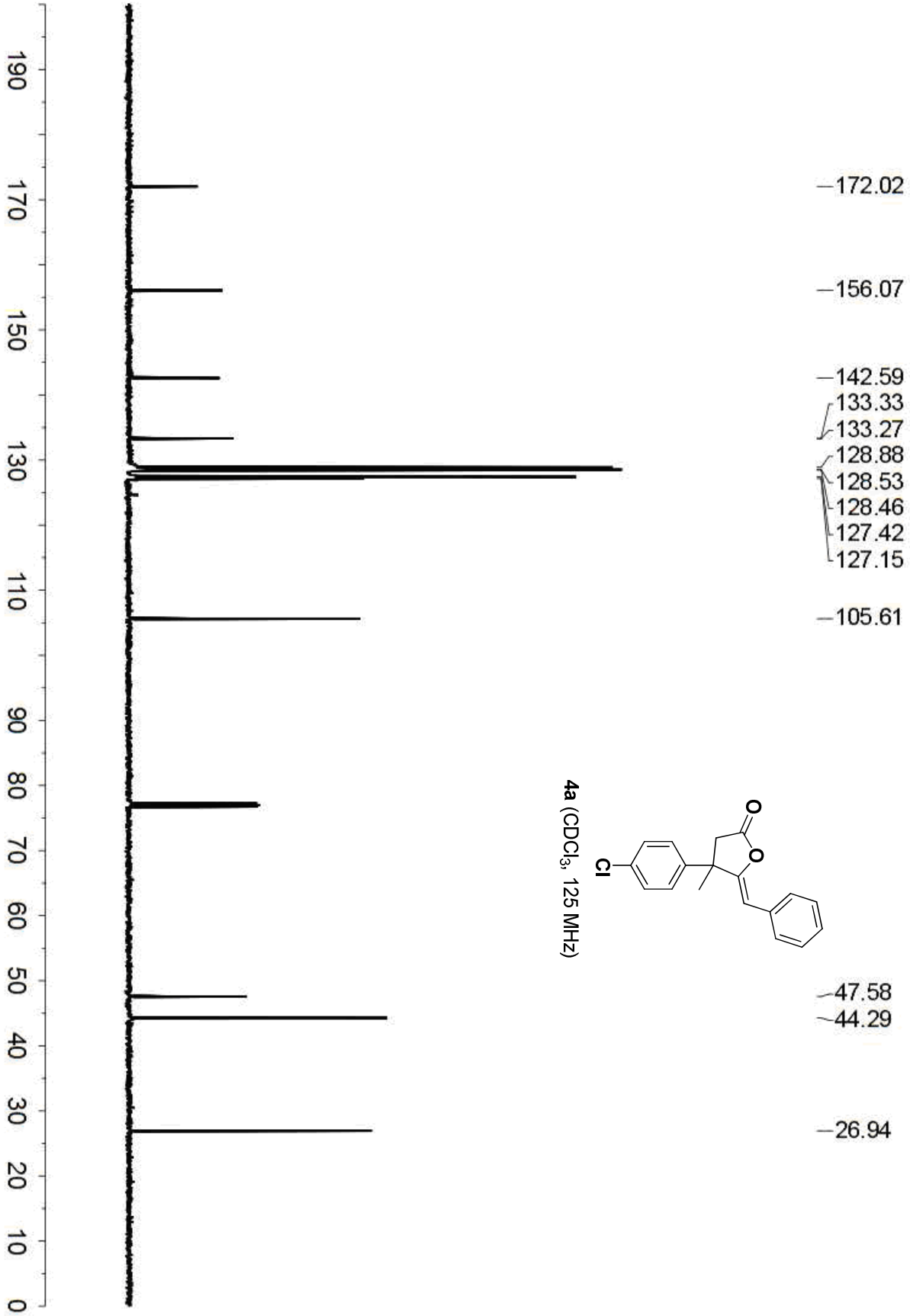
3pb ($CDCl_3$, 500 MHz)

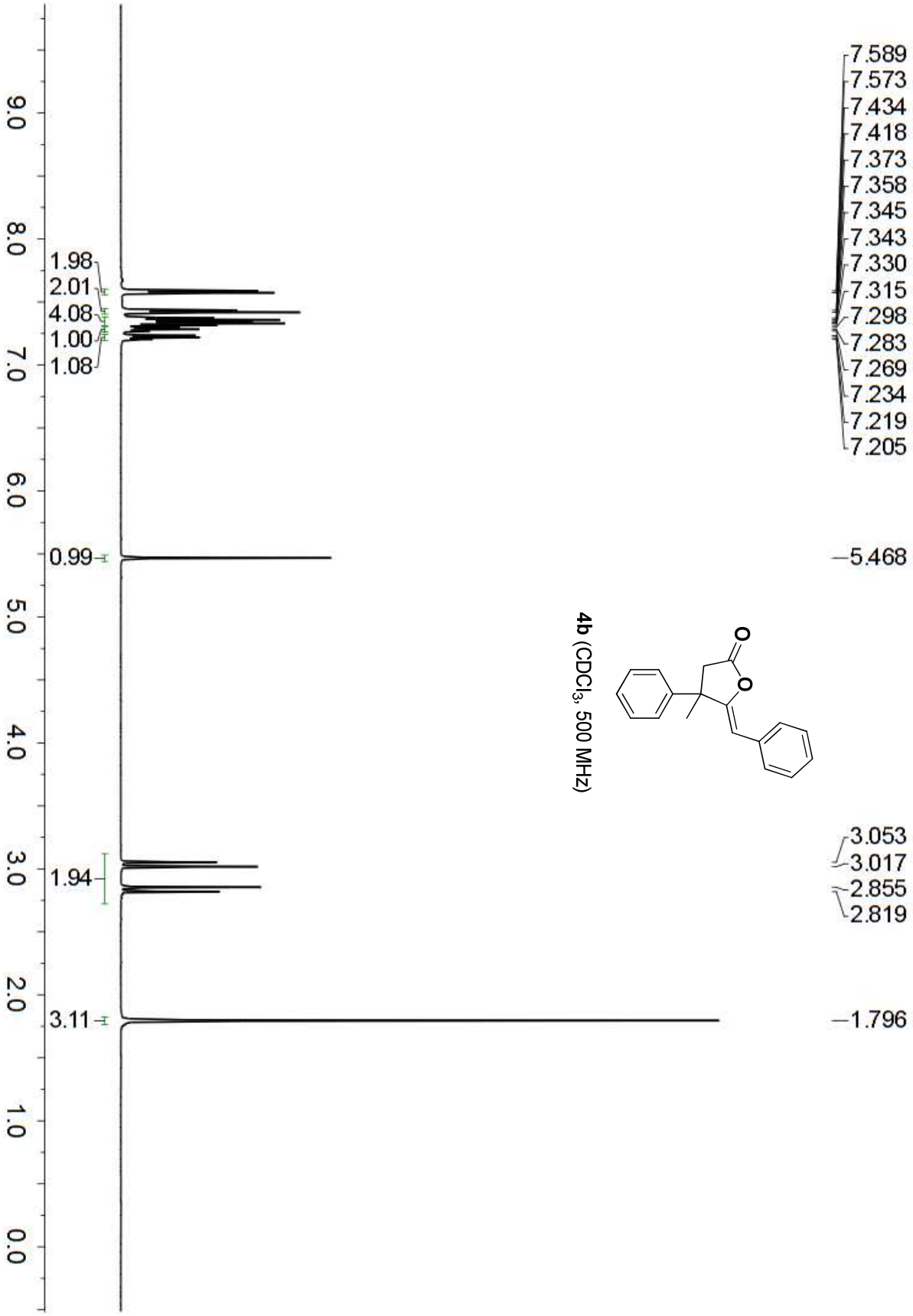


3pb ($CDCl_3$, 125 MHz)

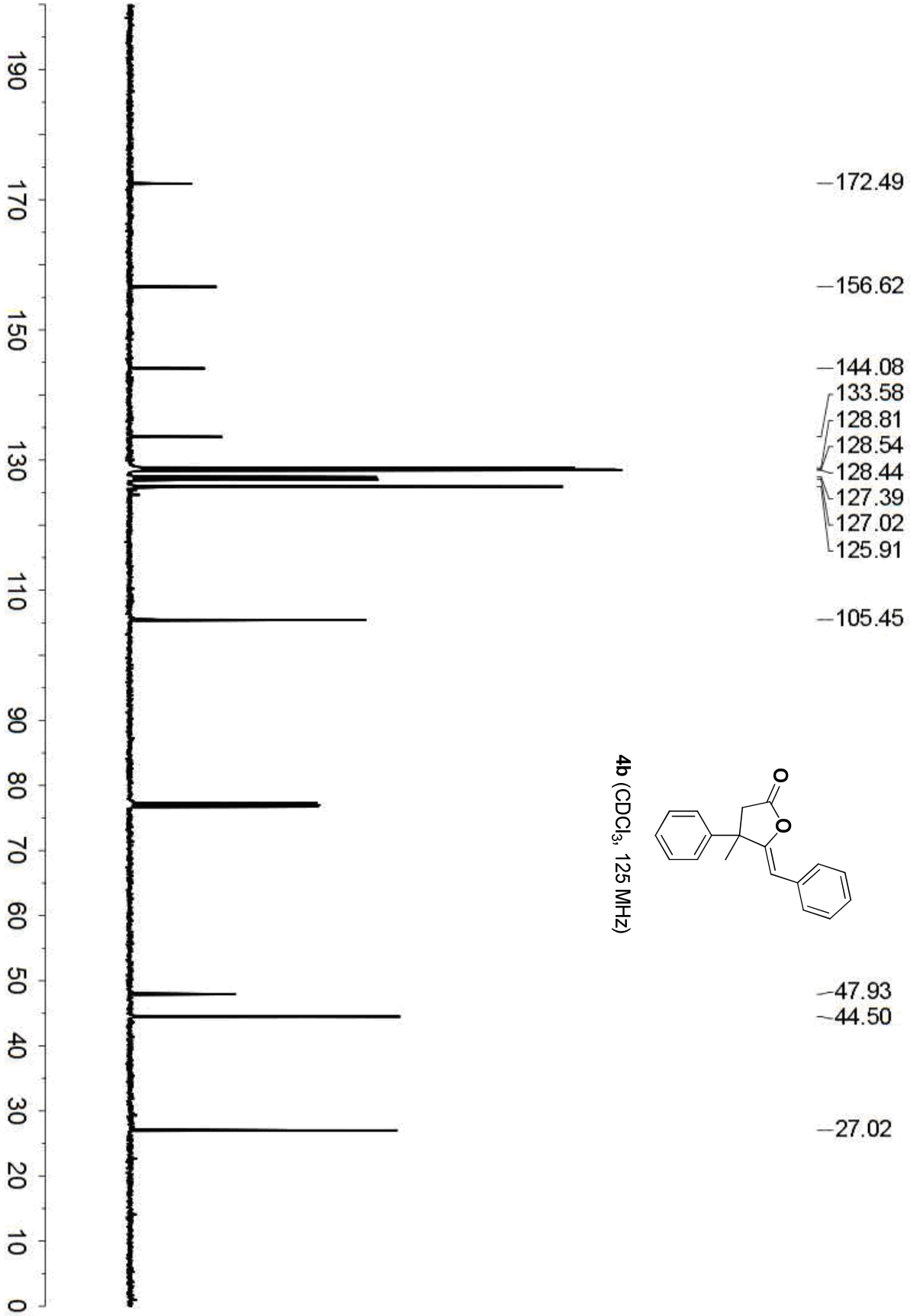


4a ($CDCl_3$, 500 MHz)

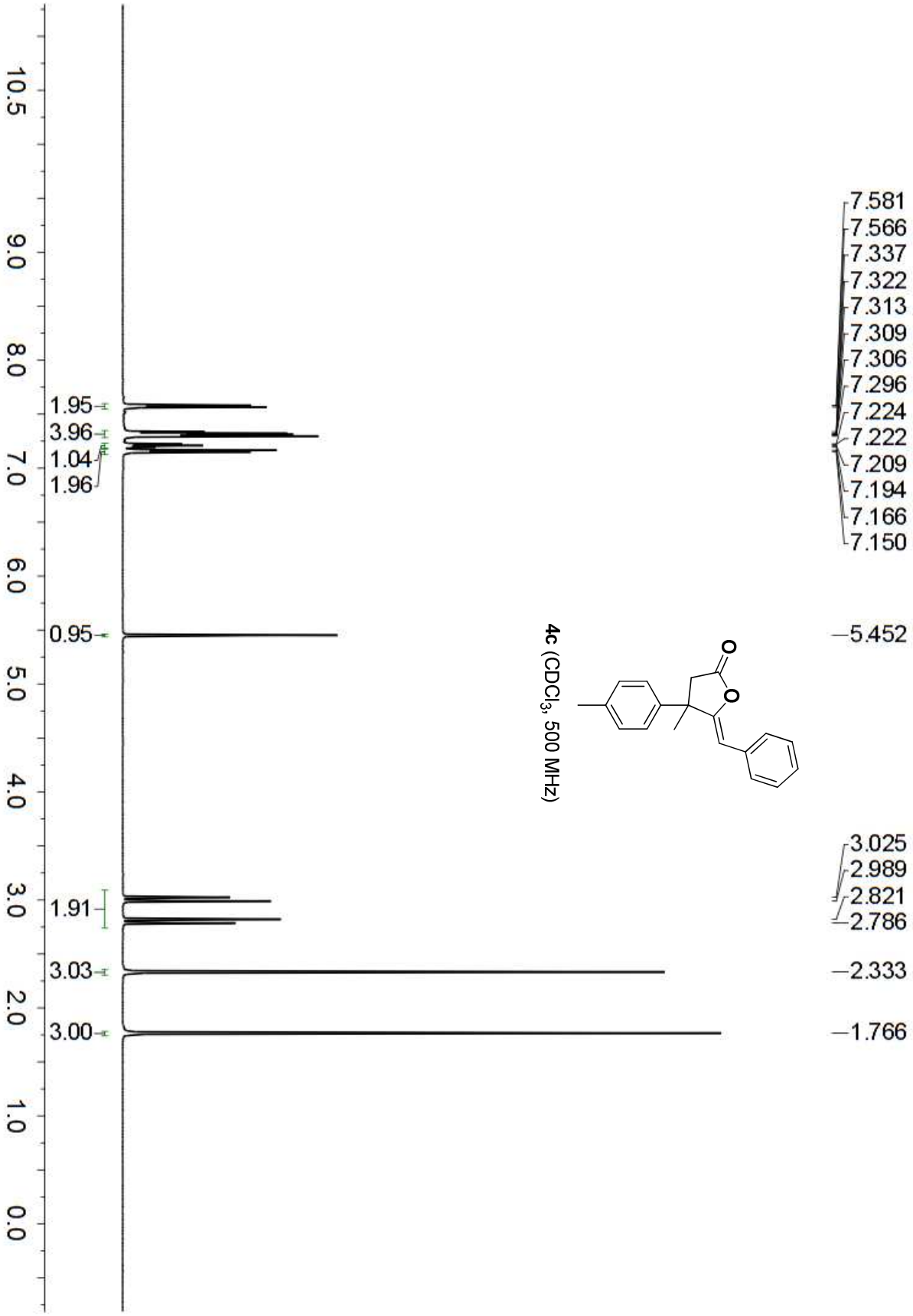

172.02
156.07
142.59
133.33
133.27
128.88
128.53
128.46
127.42
127.15
105.61
47.58
44.29
26.94
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
4a (CDCl3, 125 MHz)
Cl
O
O



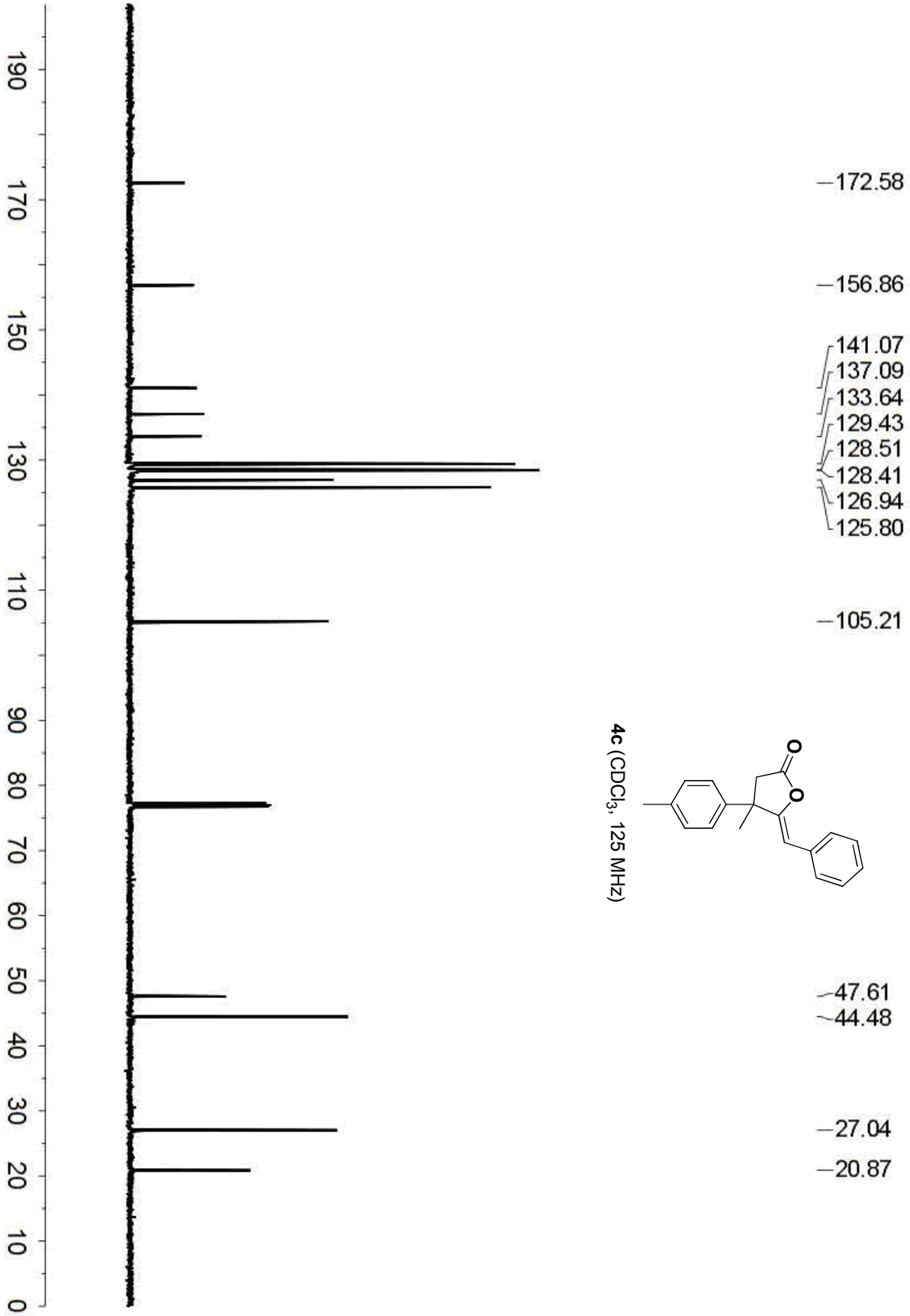
4b ($CDCl_3$, 500 MHz)



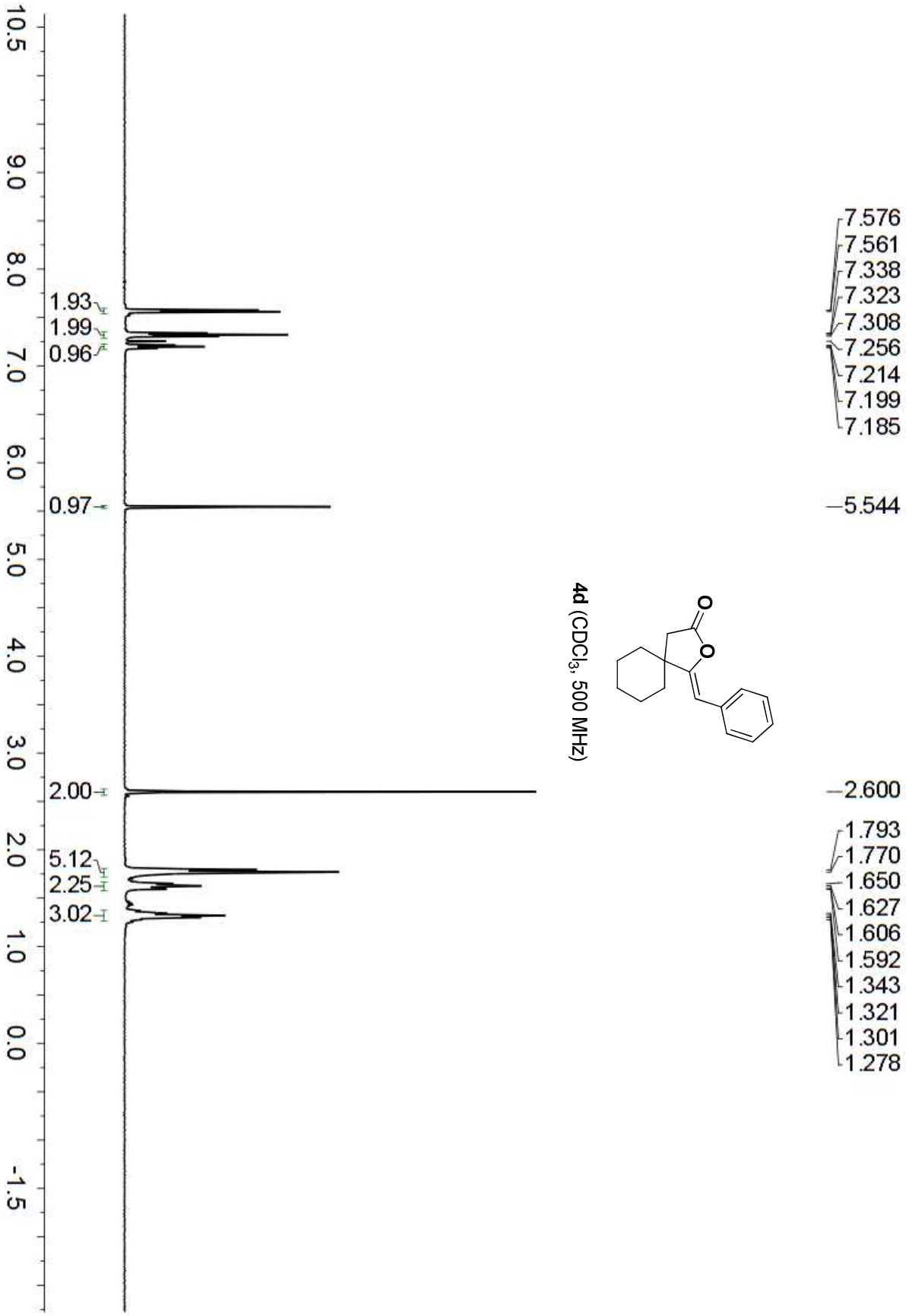
4b ($CDCl_3$, 125 MHz)



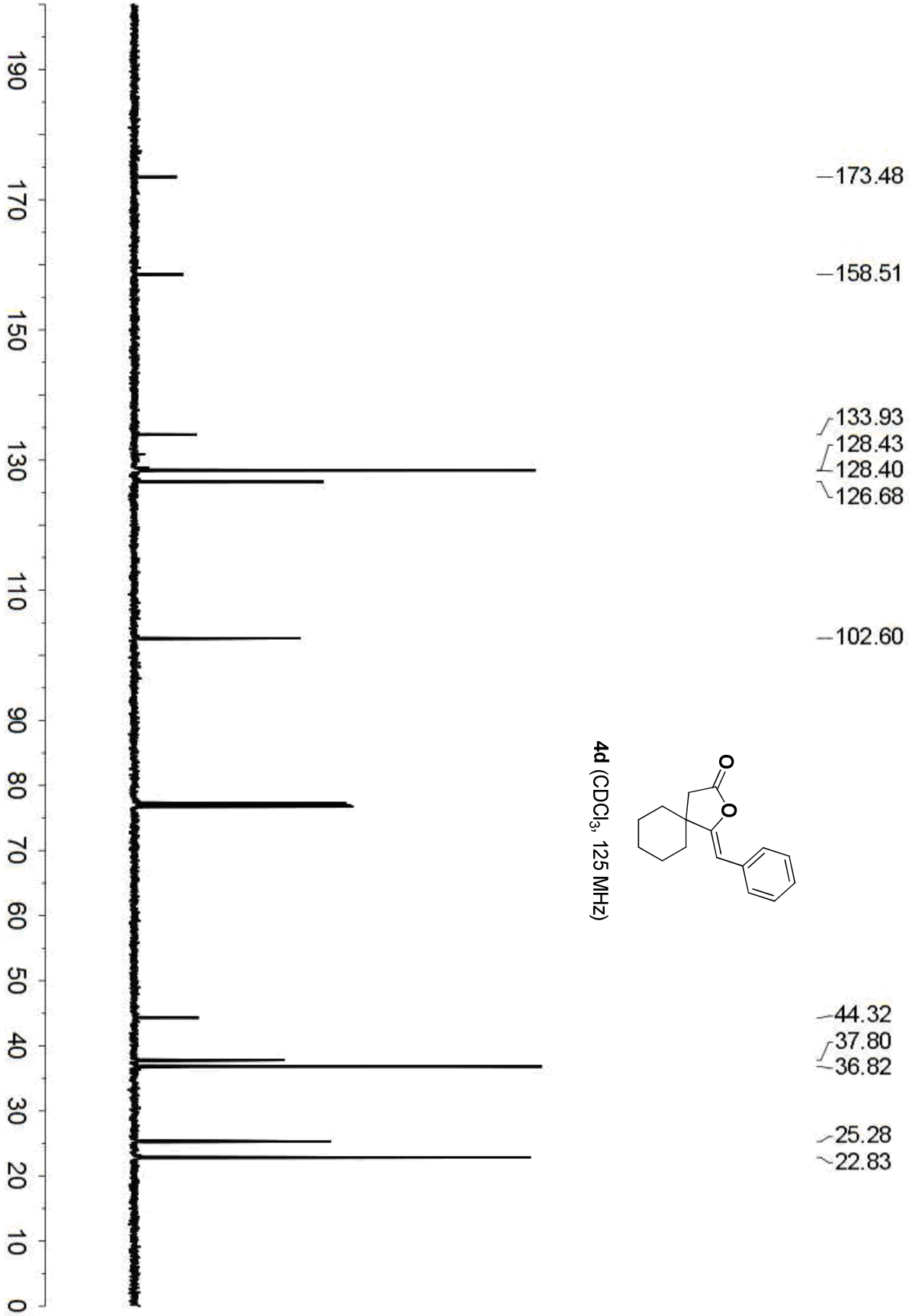
4c ($CDCl_3$, 500 MHz)



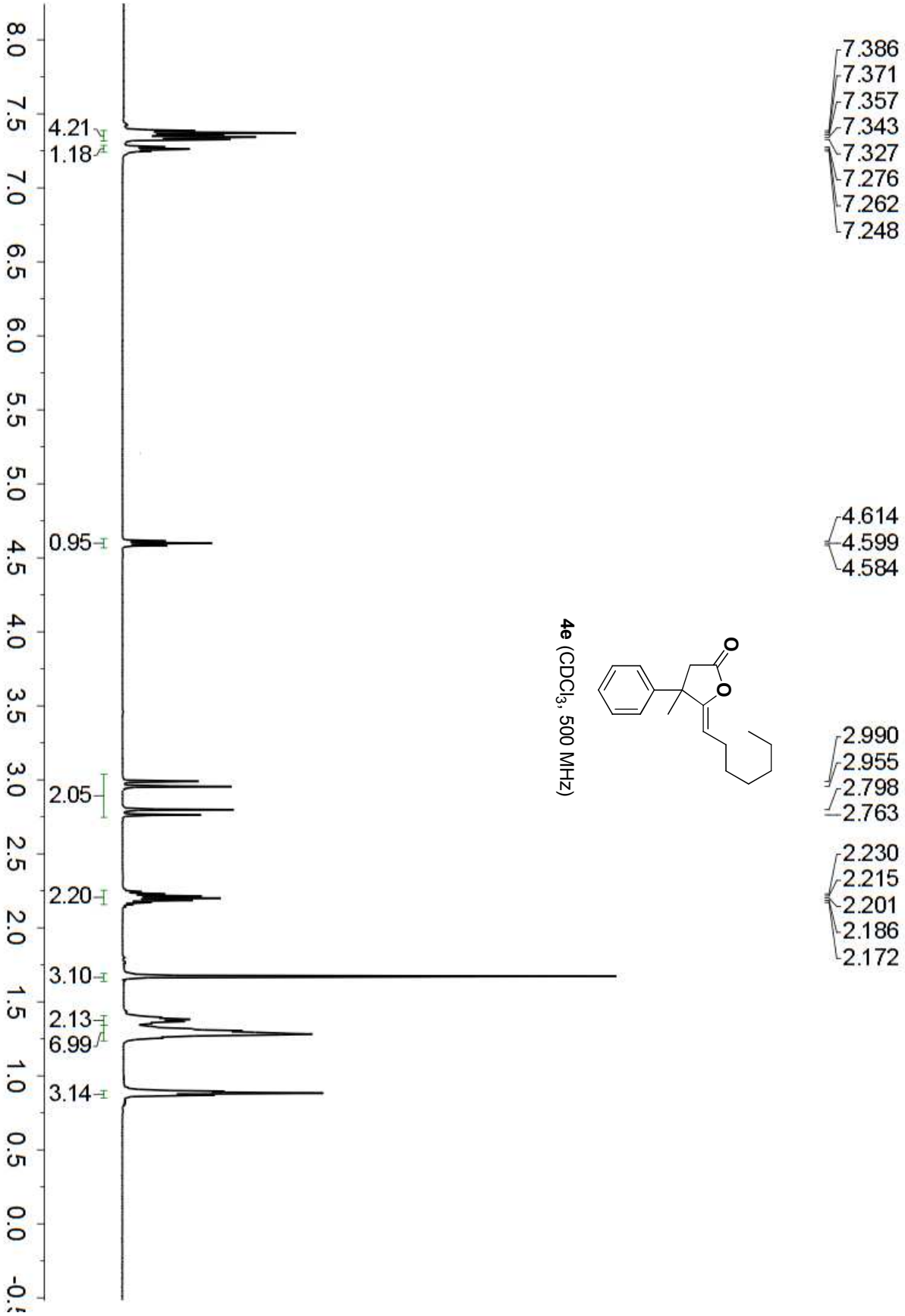
4c ($CDCl_3$, 125 MHz)



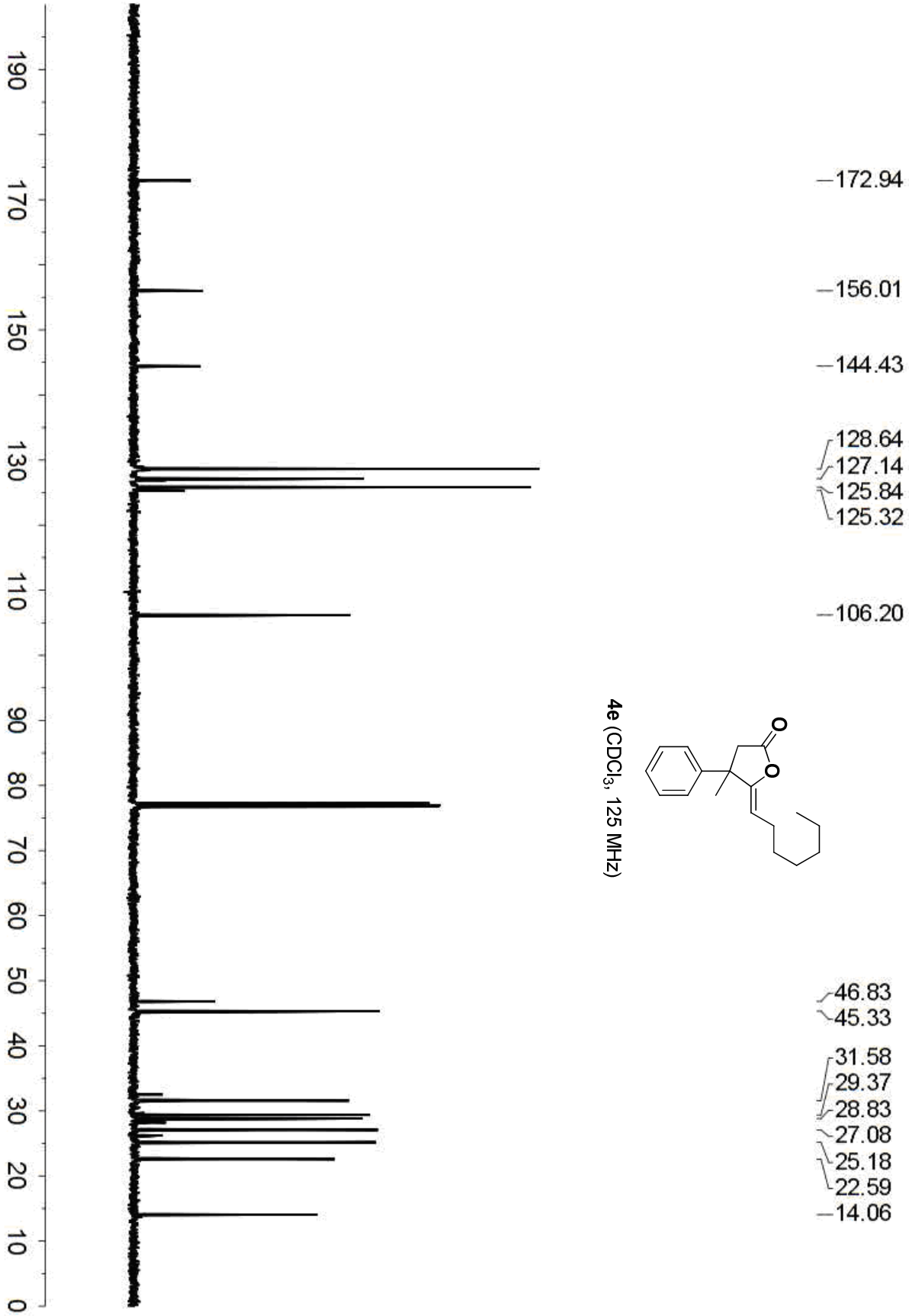
4d ($CDCl_3$, 500 MHz)



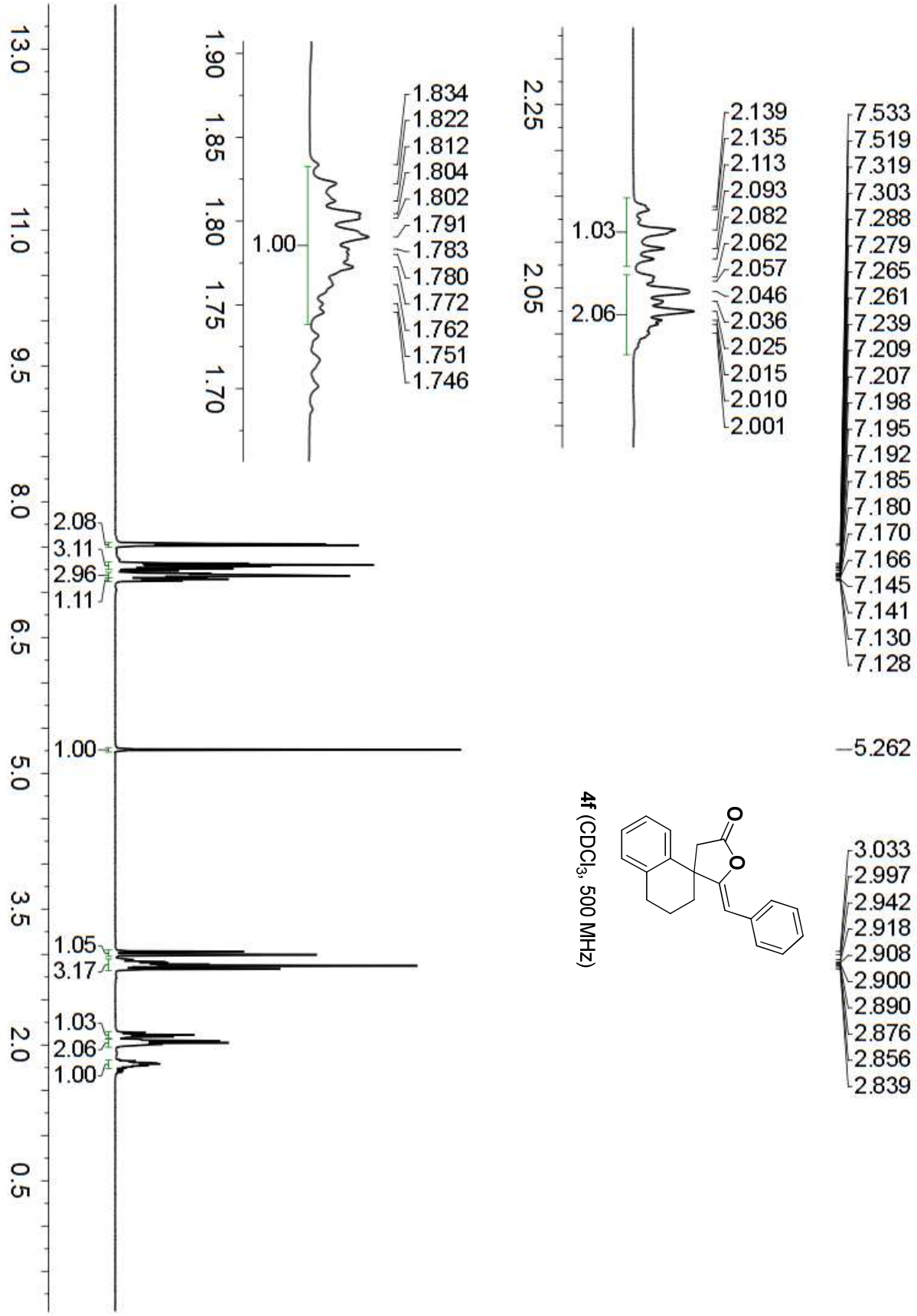
4d ($CDCl_3$, 125 MHz)



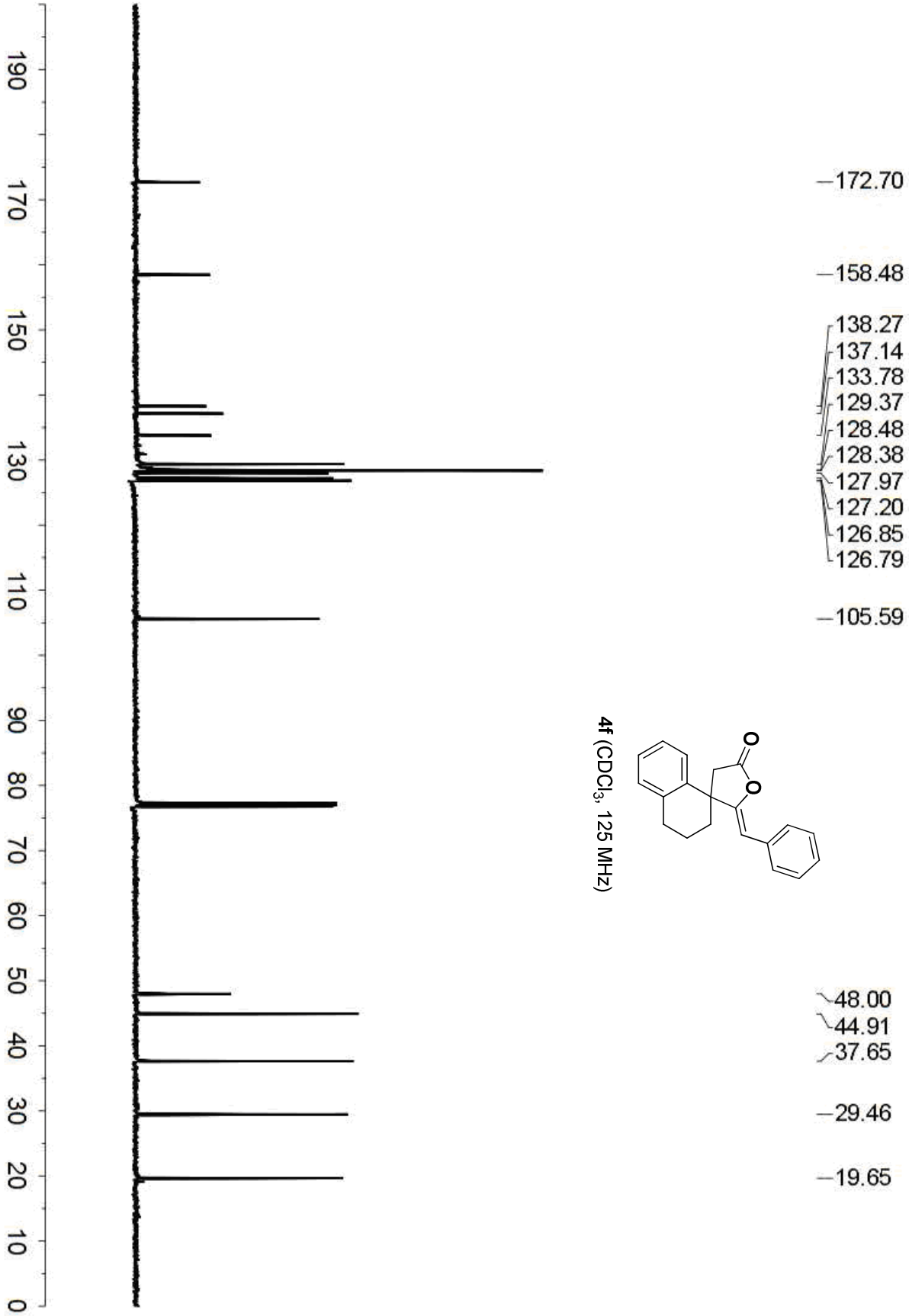
4e ($CDCl_3$, 500 MHz)



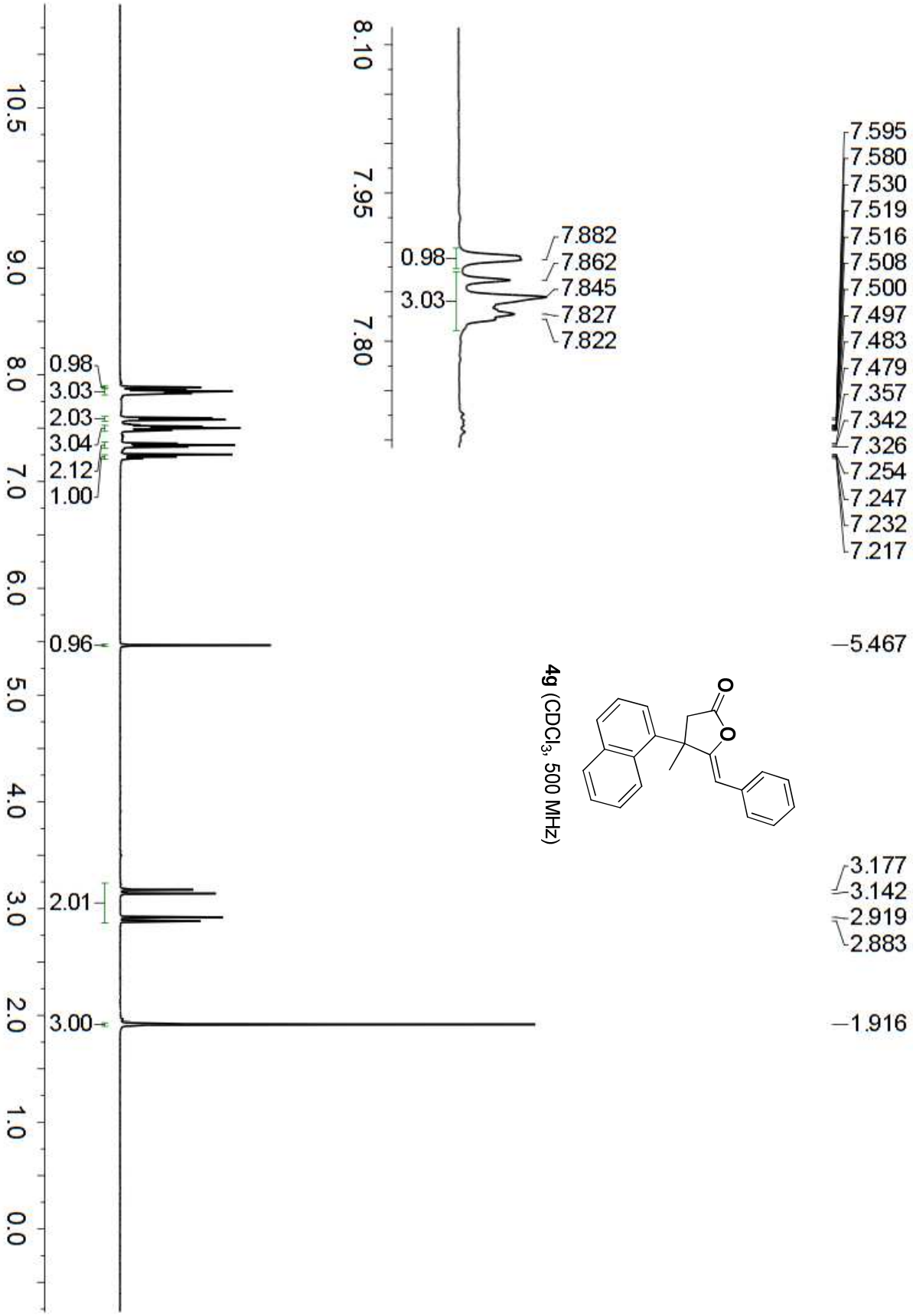
4e ($CDCl_3$, 125 MHz)



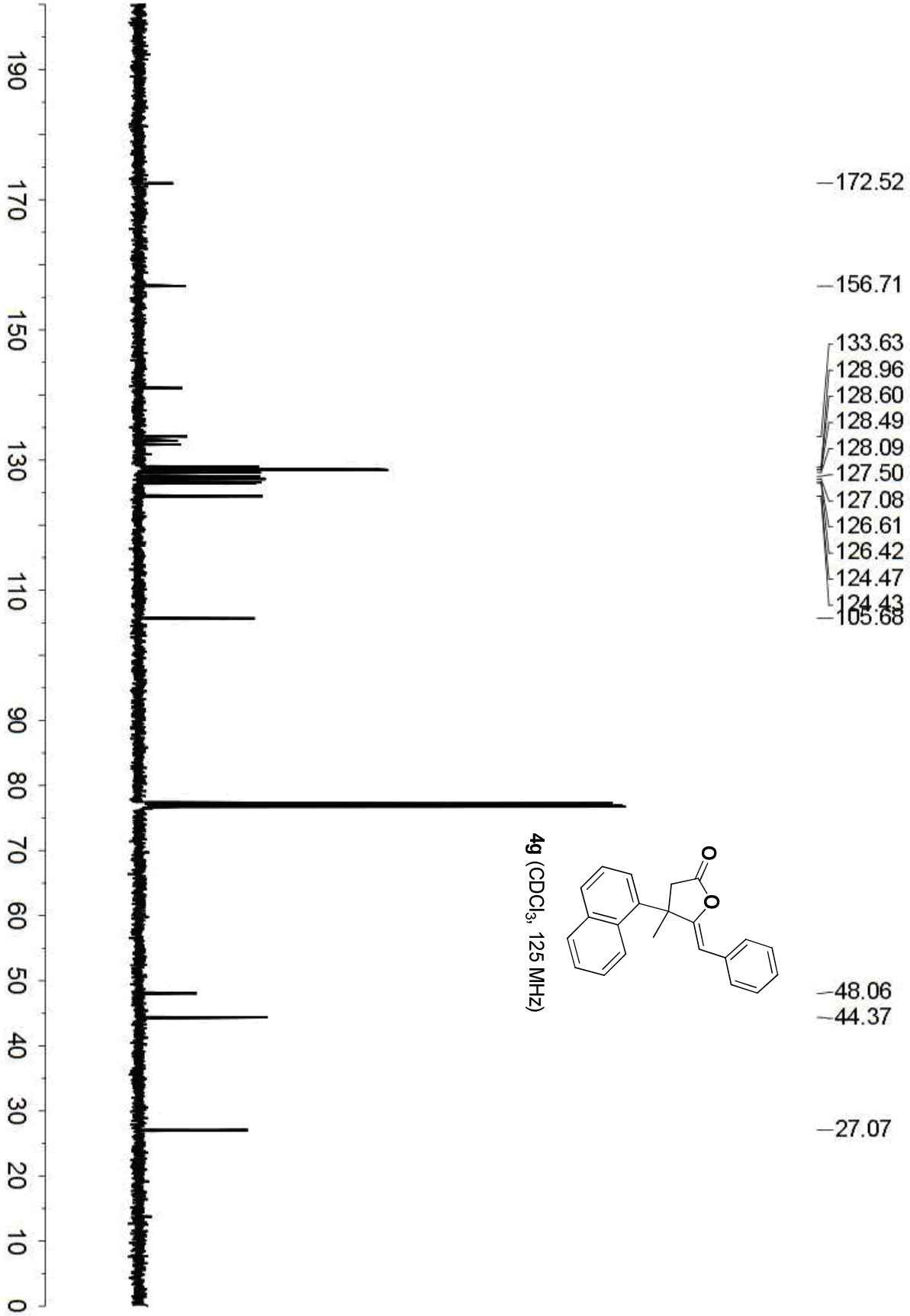
4f ($CDCl_3$, 500 MHz)



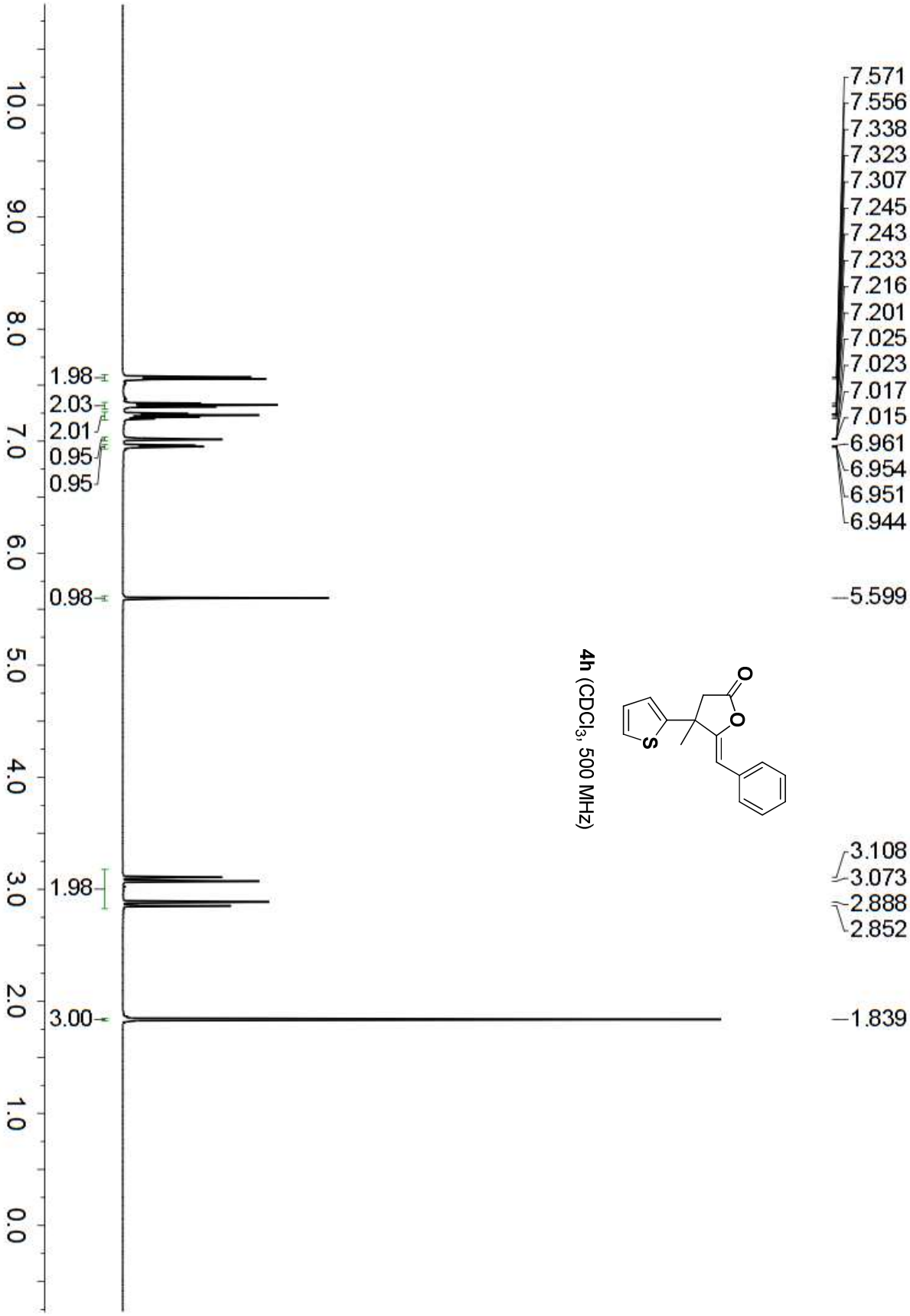
4f ($CDCl_3$, 125 MHz)

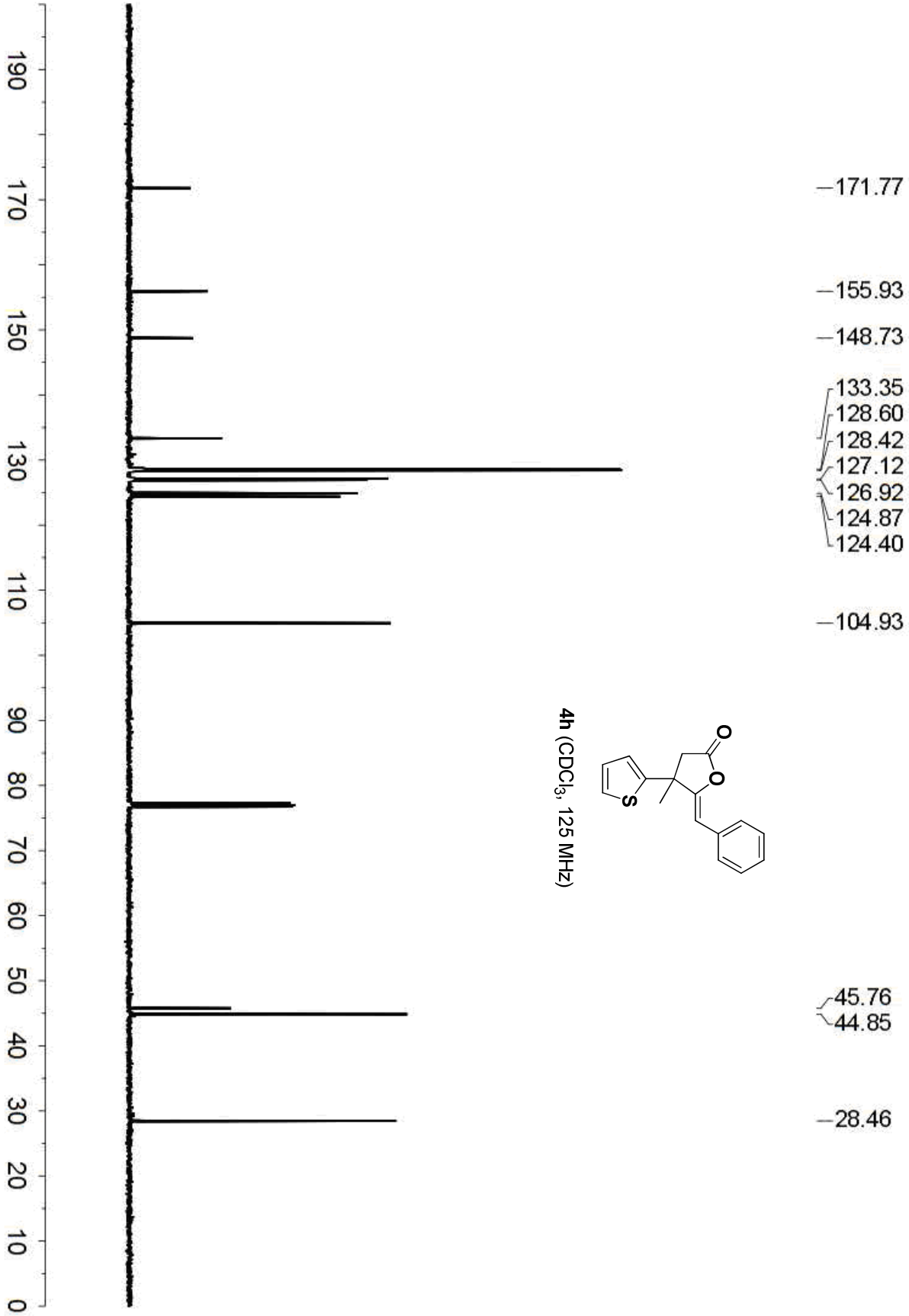


4g ($CDCl_3$, 500 MHz)



4g ($CDCl_3$, 125 MHz)


7.571
7.556
7.338
7.323
7.307
7.245
7.243
7.233
7.216
7.201
7.025
7.023
7.017
7.015
6.961
6.954
6.951
6.944
5.599
3.108
3.073
2.888
2.852
1.839
1.98
2.03
2.01
0.95
0.95
0.98
1.98
3.00
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
S
O
O
4h (CDCl3, 500 MHz)



4h ($CDCl_3$, 125 MHz)