Supporting Information

Catalyst-controlled selective synthesis of pyridines and pyrroles

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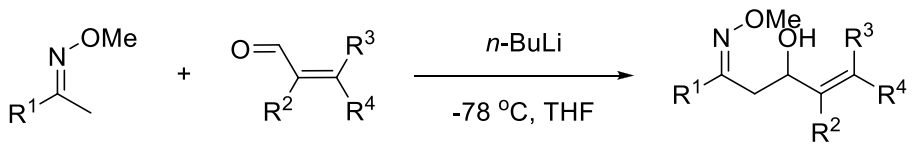
General methods: All reactions were carried out in flame or oven-dried glassware under nitrogen atmosphere with freshly distilled dry solvents under anhydrous conditions unless otherwise indicated. Flash column chromatography was performed with silica gel 60 (230 – 400 mesh). Chromatograms were visualized by fluorescence quenching with UV light at 254 nm or by staining with base solution of potassium permanganate and molybdate. NMR spectra were recorded at RT on 400 MHz Bruker spectrometers. The residual solvent signals were taken as the reference (0.00 ppm for ^{1}H NMR spectra and 77.0 ppm for ^{13}C NMR spectra in $CDCl_3$). Chemical shift (δ) is reported in ppm, coupling constants (J) are given in Hz. The following abbreviations classify the multiplicity: s = singlet, d = doublet, t = triplet, m = multiplet, dd = doublet of doublet, q = quartet and br = broad signal. HRMS (ESI) spectra were recorded on a Waters Q-Tof premierTM mass spectrometer.

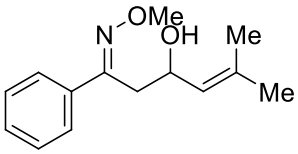
Materials: All solvents were distilled under nitrogen atmosphere from the following drying agents immediately before use: acetonitrile and chlorobenzene were distilled from P_2O_5, THF were distilled from Na.

General procedure for *β*-hydroxy oxime ethers



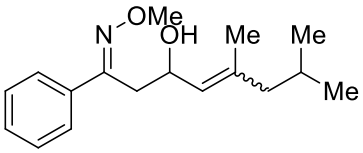
To a solution of oxime ether (2.0 mmol, 1.0 eq.) in THF (10 mL) at -78 ℃ was added *n*-BuLi (2.4 mmol, 1.2 eq.) dropwisely, and the mixture was stirred at -78 ℃ for 0.5 h. A solution of aldehyde (2.4 mmol, 1.2 eq.) in THF (2 mL) was added to the solution over 5 min. Upon completion as indicated by TLC, the reaction was quenched with saturated NH_4Cl and extracted with ethyl acetate. The combined organic layers were washed with water, brine, and dried over Na_2SO_4. The crude material was purified by flash chromatography using hexane - ethyl acetate (9:1).

(*E*)-3-hydroxy-5-methyl-1-phenylhex-4-en-1-one *O*-methyl oxime:



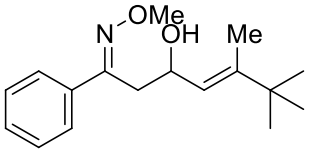
The title compound was prepared according to general procedure. The product was obtained as colorless oil. Yield: 87%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 - 7.64 (m, 2H), 7.37 - 7.35 (m, 3H), 5.22 (d, J = 8.8 Hz, 1H), 4.77 - 4.73 (m, 1H), 4.00 (s, 3H), 3.11 (dd, J_1 = 13.2 Hz, J_2 = 8.4 Hz, 1H), 2.88 (dd, J_1 = 13.2 Hz, J_2 = 5.2 Hz, 1H), 2.03 (d, J = 4.0 Hz, 1H), 1.66 (s, 3H), 1.60 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.2, 136.0, 135.1, 129.1, 128.4, 127.5, 126.6, 66.7, 62.0, 35.3, 25.6, 18.1; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{14}H_{20}NO_2$: 234.1494. Found: 234.1496.

(1*E*)-3-hydroxy-5,7-dimethyl-1-phenyloct-4-en-1-one O-methyl oxime:



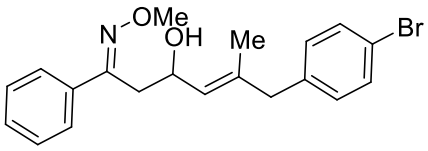
The title compound was prepared according to general procedure. The product was obtained as colorless oil in a *cis* : *trans* ratio of 17 : 83. Yield: 74%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 - 7.64 (m, 2H), 7.36 - 7.34 (m, 3H), 5.29 & 5.20 (d & dd, J = 9.2 Hz & J_1 = 8.8 Hz, J_2 = 0.8 Hz, 1H), 4.79 - 4.74 (m, 1H), 4.00 & 3.99 (s & s, 3H), 3.11 - 3.06 (m, 1H), 2.96 - 2.91 (m, 1H), 2.05 & 1.81 (m & m, 1H), 1.80 - 1.69 (m, 3H), 1.65 & 1.54 (d & d, J = 1.2 Hz & 1.2 Hz, 3H), 0.86 & 0.77 (d & d, J = 6.4 Hz & 6.4 Hz, 3H), 0.83 & 0.82 (d & d, J = 6.4 Hz & 6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.3, 156.0, 138.4, 137.8, 136.1, 136.0, 129.1, 128.9, 128.4, 128.4, 126.7, 126.6, 66.6, 66.2, 62.0, 62.0, 49.2, 41.4, 35.5, 35.2, 26.4, 25.9, 23.6, 22.6, 22.6, 22.4, 22.2, 16.4; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{17}H_{26}NO_2$: 276.1964. Found: 276.1964.

(1*E*, 4*E*)-3-hydroxy-5,6,6-trimethyl-1-phenylhept-4-en-1-one *O*-methyl oxime:



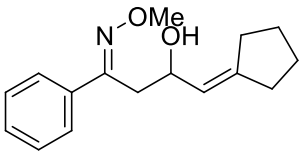
The title compound was prepared according to general procedure. The product was obtained as colorless oil. Yield: 79%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 - 7.56 (m, 2H), 7.29 - 7.26 (m, 3H), 5.15 (dd, J_1 = 8.8 Hz, J_2 = 1.2 Hz, 1H), 4.74 - 4.69 (m, 1H), 3.93 (s, 3H), 3.05 (dd, J_1 = 13.2 Hz, J_2 = 7.2 Hz, 1H), 2.87 (dd, J_1 = 12.8 Hz, J_2 = 6.0 Hz, 1H), 1.99 (s, 1H), 1.49 (d, J = 1.2 Hz, 3H), 0.85 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.0, 146.1, 136.0, 129.1, 128.4, 126.6, 124.2, 66.9, 62.0, 36.0, 35.1, 28.7, 13.0; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{17}H_{26}NO_2$: 276.1964. Found: 276.1967.

(1*E*, 4*E*)-6-(4-bromophenyl)-3-hydroxy-5-methyl-1-phenylhex-4-en-1-one *O*-methyl oxime:



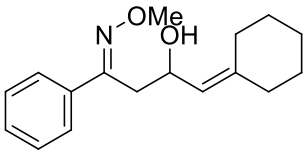
The title compound was prepared according to general procedure. The product was obtained as colorless oil. Yield: 85%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 - 7.62 (m, 2H), 7.38 - 7.33 (m, 5H), 6.88 (d, J = 8.4 Hz, 2H), 5.23 (dd, J_1 = 8.8 Hz, J_2 = 1.2 Hz, 1H), 4.81 - 4.78 (m, 1H), 4.00 (s, 3H), 3.16 - 3.10 (m, 3H), 2.94 (dd, J_1 = 13.2 Hz, J_2 = 6.0 Hz, 1H), 2.05 (d, J = 4.0 Hz, 1H), 1.51 (d, J = 1.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.7, 138.1, 137.4, 135.9, 131.4, 130.7, 129.3, 129.2, 128.5, 126.5, 120.0, 66.5, 62.1, 45.2, 35.1, 16.4; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{20}H_{23}NO_2Br$: 388.0912. Found: 388.0909.

(*E*)-4-cyclopentylidene-3-hydroxy-1-phenylbutan-1-one *O*-methyl oxime:



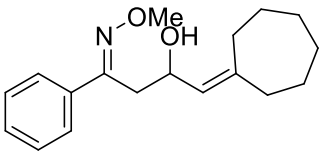
The title compound was prepared according to general procedure. The product was obtained as yellow oil. Yield: 82%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 - 7.63 (m, 2H), 7.37 - 7.33 (m, 3H), 5.33 - 5.30 (m, 1H), 4.67 - 4.60 (m, 1H), 4.00 (s, 3H), 3.13 (dd, J_1 = 13.2 Hz, J_2 = 8.0 Hz, 1H), 2.89 (dd, J_1 = 13.2 Hz, J_2 = 5.6 Hz, 1H), 2.26 - 2.19 (m, 2H), 2.13 (m, 1H), 2.09 - 2.02 (m, 2H), 1.64 - 1.51 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.2, 146.6, 136.0, 129.1, 128.4, 126.6, 122.7, 68.3, 62.0, 35.1, 33.6, 28.7, 26.3, 25.9; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{16}H_{22}NO_2$: 260.1651. Found: 260.1651.

(*E*)-4-cyclohexylidene-3-hydroxy-1-phenylbutan-1-one *O*-methyl oxime:



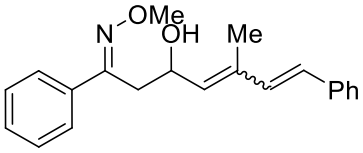
The title compound was prepared according to general procedure. The product was obtained as colorless oil. Yield: 90%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 - 7.64 (m, 2H), 7.36 - 7.33 (m, 3H), 5.15 (d, J = 8.8 Hz, 1H), 4.83 - 4.77 (m, 1H), 3.99 (s, 3H), 3.11 (dd, J_1 = 13.2 Hz, J_2 = 7.6 Hz, 1H), 2.88 (dd, J_1 = 13.2 Hz, J_2 = 5.6 Hz, 1H), 2.14 - 1.98 (m, 5H), 1.52 - 1.37 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.0, 142.8, 136.0, 129.1, 128.4, 126.6, 124.2, 65.8, 62.0, 36.9, 35.6, 29.2, 28.2, 27.7, 26.6; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{17}H_{24}NO_2$: 274.1807. Found: 274.1803.

(*E*)-4-cycloheptylidene-3-hydroxy-1-phenylbutan-1-one *O*-methyl oxime:



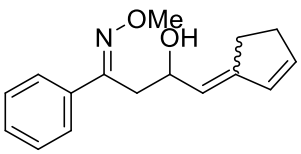
The title compound was prepared according to general procedure. The product was obtained as colorless oil. Yield: 70%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 - 7.65 (m, 2H), 7.38 - 7.35 (m, 3H), 5.22 (d, J = 8.8 Hz, 1H), 4.79 - 4.72 (m, 1H), 4.00 (s, 3H), 3.11 (dd, J_1 = 13.2 Hz, J_2 = 8.0 Hz, 1H), 2.91 (dd, J_1 = 13.2 Hz, J_2 = 5.2 Hz, 1H), 2.24 - 2.17 (m, 2H), 2.15 - 2.14 (m, 2H), 1.96 (d, J = 3.6 Hz, 1H), 1.55 - 1.45 (m, 8H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.2, 144.7, 136.0, 129.2, 128.4, 127.6, 126.6, 66.3, 62.0, 37.7, 35.3, 30.0, 29.5, 28.9, 28.6, 27.3; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{18}H_{26}NO_2$: 288.1964. Found: 288.1967.

(1*E*, 4*E*)-3-hydroxy-5-methyl-1,7-diphenylhepta-4,6-dien-1-one *O*-methyl oxime:



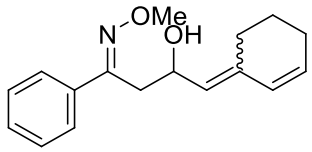
The title compound was prepared according to general procedure. The product was obtained as colorless oil in a *cis* : *trans* ratio 33 : 67. Yield: 89%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 - 7.66 (m, 2H), 7.40 - 7.29 (m, 7H), 7.25 - 7.22 (m, 1H), 7.07 & 6.72 (d & d, J = 16.0 Hz & 16.0 Hz, 1H), 6.58 & 6.53 (d & d, J = 16.0 Hz & 16.4 Hz, 1H), 5.65 & 5.48 (d & d, J = 8.4 Hz & 8.4 Hz, 1H), 5.14 - 5.13 & 4.94 - 4.93 (m & m, 1H), 4.03 & 4.02 (s & s, 3H), 3.19 - 3.13 (m, 1H), 2.98 (dd, J_1 = 13.6 Hz, J_2 = 5.2 Hz, 1H), 2.18 (s, 1H), 1.91 & 1.85 (d & d, J = 0.8 Hz & 0.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.0, 137.4, 137.4, 135.9, 135.4, 134.5, 134.1, 133.0, 132.8, 130.2, 129.3, 129.2, 128.6, 128.5, 128.3, 127.7, 127.4, 126.7, 126.6, 126.6, 126.4, 125.3, 66.8, 66.0, 62.2, 62.1, 35.6, 35.2, 20.2, 12.9; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{21}H_{24}NO_2$: 322.1807. Found: 322.1812.

(1*E*)-4-(cyclopent-2-enylidene)-3-hydroxy-1-phenylbutan-1-one *O*-methyl oxime:



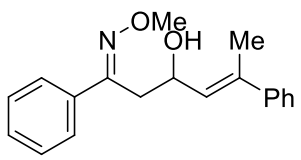
The title compound was prepared according to general procedure. The product was obtained as colorless oil in a *cis* : *trans* ratio of 38 :62. Yield 80%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 - 7.64 (m, 2H), 7.36 - 7.31 (m, 3H), 6.32 & 6.10 (d & d, J = 5.6 Hz & 5.2 Hz, 1H), 6.17 & 6.06 (d & d, J = 3.2 Hz & 5.6 Hz, 1H), 5.37 & 5.20 (d & d, J = 8.8 Hz & 9.2 Hz, 1H), 4.83 - 4.81 & 4.69 - 4.65 (m & m, 1H), 4.00 (s, 3H), 3.18 - 3.11 (m, 1H), 2.98 - 2.92 (m, 1H), 2.53 - 2.37 (m, 4H), 2.15 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.2, 156.2, 149.4, 148.4, 140.7, 139.1, 136.1, 136.0, 134.2, 129.6, 129.2, 129.1, 128.4, 126.7, 126.6, 120.9, 119.6, 68.4, 67.9, 62.0, 35.7, 35.1, 32.2, 31.2, 29.3, 25.8; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{16}H_{20}NO_2$: 258.1494. Found: 258.1498.

(1*E*)-4-(cyclohex-2-enylidene)-3-hydroxy-1-phenylbutan-1-one *O*-methyl oxime:



The title compound was prepared according to general procedure. The product was obtained as yellow oil in a *cis* : *trans* ratio of 33 : 67. Yield: 79%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 - 7.63 (m, 2H), 7.37 - 7.33 (m, 3H), 6.31 & 5.98 (d & d, J = 10.0 Hz & 9.6 Hz, 1H), 5.89 - 5.84 & 5.83 - 5.78 (m & m, 1H), 5.27 & 5.13 (d & d, J = 8.8 Hz & 8.8 Hz, 1H), 4.96 - 4.91 & 4.87 - 4.81 (m & m, 1H), 4.01 & 4.00 (s, 3H), 3.16 - 3.11 (m, 1H), 2.94 - 2.88 (m, 1H), 2.22 - 2.08 (m, 5H), 1.68 - 1.61 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.1, 156.0, 136.7, 136.0, 135.8, 132.1, 130.4, 130.3, 129.2, 128.5, 128.4, 128.3, 126.7, 126.6, 123.7, 66.0, 65.3, 62.1, 35.5, 35.2, 32.0, 26.0, 25.5, 25.4, 22.8, 22.2; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{17}H_{22}NO_2$: 272.1651. Found: 272.1653.

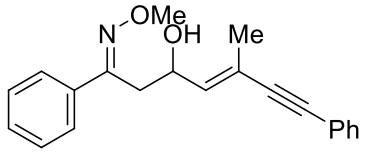
(1*E*, 4*E*)-3-hydroxy-1,5-diphenylhex-4-en-1-one *O*-methyl oxime:



The title compound was prepared according to general procedure. The product was obtained as colorless oil. Yield 87%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 - 7.67 (m, 2H), 7.37 - 7.35 (m, 3H), 7.30 - 7.23 (m, 5H), 5.77 (dd, J_1 = 8.4 Hz, J_2 = 1.2 Hz, 1H), 4.95 - 4.93 (m, 1H), 4.03 (s, 3H), 3.20 (dd, J_1 = 13.2 Hz, J_2 = 4.0 Hz, 1H), 3.04 (dd, J_1 = 13.2 Hz, J_2 = 5.6 Hz, 1H),

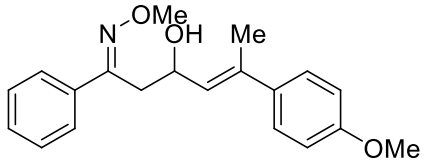
2.18 (d, *J* = 2.8 Hz, 1H), 2.00 (d, *J* = 1.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.9, 142.9, 137.2, 136.0, 130.2, 129.3, 128.5, 128.2, 127.3, 126.6, 125.9, 67.1, 62.1, 35.1, 16.3; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{19}H_{22}NO_2$: 296.1651. Found: 296.1656.

(1*E*, 4*E*)-3-hydroxy-5-methyl-1,7-diphenylhept-4-en-6-yn-1-one *O*-methyl oxime:



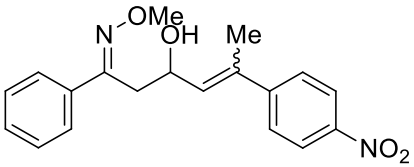
The title compound was prepared according to general procedure. The product was obtained as colorless oil. Yield: 73%. 1H NMR (400 MHz, CDCl3) δ 7.69 - 7.67 (m, 2H), 7.42 - 7.37 (m, 5H), 7.31 - 7.25 (m, 3H), 5.96 (dd, J_1 = 8.8 Hz, J_2 = 1.6 Hz, 1H), 4.87 - 4.81 (m, 1H), 4.02 (s, 3H), 3.12 (dd, J_1 = 13.6 Hz, J_2 = 8.8 Hz, 1H), 3.00 (dd, J_1 = 13.6 Hz, J_2 = 8.8 Hz, 1H), 2.22 (d, *J* = 4.0 Hz, 1H), 1.87 (d, *J* = 1.6 Hz, 3H); 13C NMR (100 MHz, CDCl3) δ 155.7, 138.9, 135.8, 131.6, 129.3, 128.6, 128.3, 128.2, 126.6, 123.3, 120.2, 91.4, 87.8, 66.6, 62.2, 34.8, 17.9; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{21}H_{22}NO_2$: 320.1651. Found: 320.1644.

(1*E*, 4*E*)-3-hydroxy-5-(4-methoxyphenyl)-1-phenylhex-4-en-1-one *O*-methyl oxime:



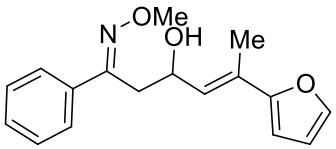
The title compound was prepared according to general procedure. The product was obtained as yellow oil. Yield 90%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 - 7.66 (m, 2H), 7.38 - 7.35 (m, 3H), 7.21 (dd, J_1 = 6.4 Hz, J_2 = 2.0 Hz, 2H), 6.82 (dd, J_1 = 6.4 Hz, J_2 = 2.0 Hz, 2H), 5.71 (dd, J_1 = 8.4 Hz, J_2 = 1.2 Hz, 1H), 4.96 - 4.90 (m, 1H), 4.02 (s, 3H), 3.80 (s, 3H), 3.20 (dd, J_1 = 13.2 Hz, J_2 = 8.0 Hz, 1H), 3.03 (dd, J_1 = 13.2 Hz, J_2 = 5.2 Hz, 1H), 2.16 (s, 1H), 1.98 (d, *J* = 1.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.9, 155.9, 136.5, 135.9, 135.2, 129.1, 128.6, 128.4, 126.8, 126.6, 113.5, 67.1, 62.0, 55.2, 35.2, 16.2; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{19}H_{21}N_2O_4$: 341.1501. Found: 341.1505.

(1*E*)-3-hydroxy-5-(4-nitrophenyl)-1-phenylhex-4-en-1-one *O*-methyl oxime:



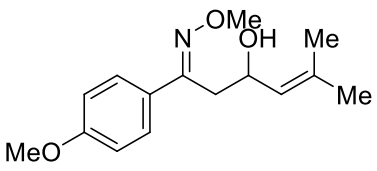
The title compound was prepared according to general procedure. The product was obtained as yellow oil in a cis : trans ratio of 25 : 75. Yield 68%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 - 8.10 (m, 2H), 7.68 - 7.66 & 7.53 - 7.51 (m & m, 2H), 7.39 - 7.28 (m, 5H), 5.86 & 5.65 (dd & dd, J_1 = 8.8 Hz & 9.6 Hz, J_2 = 1.2 Hz & 1.2 Hz, 1H), 5.00 - 4.94 & 4.37 - 4.36 (m & m, 1H), 4.03 & 3.92 (s & s, 3H), 3.22 & 2.92 (dd & dd, J_1 = 13.2 Hz, J_2 = 8.0 Hz, 1H), 3.10 - 3.05 (m, 1H), 2.39 & 1.66 (s & s, 1H), 2.02 (d, J = 1.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.5, 155.4, 149.4, 147.8, 146.9, 137.6, 135.8, 135.2, 133.8, 131.4, 129.4, 129.4, 128.8, 128.6, 128.5, 126.5, 126.4, 123.5, 123.4, 67.1, 67.0, 62.2, 62.1, 35.0, 34.9, 25.1, 16.2; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{19}H_{21}N_2O_4$: 341.1501. Found: 341.1505.

(1*E*, 4*E*)-5-(furan-2-yl)-3-hydroxy-1-phenylhex-4-en-1-one *O*-methyl oxime:



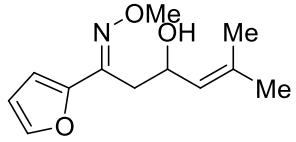
The title compound was prepared according to general procedure. The product was obtained as colorless oil. Yield: 75%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 - 7.67 (m, 2H), 7.37 - 7.33 (m, 4H), 6.36 (dd, J_1 = 3.2 Hz, J_2 = 2.0 Hz, 1H), 6.26 (d, J = 3.2 Hz, 1H), 6.11 (dd, J_1 = 8.8 Hz, J_2 = 0.8 Hz, 1H), 4.96 - 4.89 (m, 1H), 4.01 (s, 3H), 3.19 (dd, J_1 = 13.6 Hz, J_2 = 8.8 Hz, 1H), 3.00 (dd, J_1 = 13.2 Hz, J_2 = 8.8 Hz, 1H), 2.12 (d, J = 4.0 Hz, 1H), 1.92 (d, J = 1.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.9, 155.1, 141.9, 135.9, 129.2, 128.5, 126.7, 126.6, 126.5, 111.1, 106.4, 66.6, 62.1, 35.3, 13.6; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{17}H_{20}NO_3$: 286.1443. Found: 286.1442.

(*E*)-3-hydroxy-3-(4-methoxyphenyl)-1-phenylpropan-1-one *O*-methyl oxime:



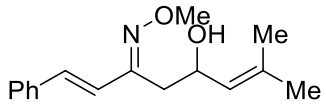
The title compound was prepared according to general procedure. The product was obtained as colorless oil. Yield: 85%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61 - 7.59 (m, 2H), 6.89 - 6.86 (m, 2H), 5.23 (d, J = 8.8 Hz, 1H), 4.76 - 4.70 (m, 1H), 3.97 (s, 3H), 3.82 (s, 3H), 3.07 (dd, J_1 = 13.2 Hz, J_2 = 8.4 Hz, 1H), 2.85 (dd, J_1 = 13.6 Hz, J_2 = 5.2 Hz, 1H), 2.14 (s, 1H), 1.66 (s, 3H), 1.61 (d, J = 0.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.4, 155.8, 135.0, 128.5, 127.9, 127.6, 113.8, 66.8, 61.9, 55.3, 35.2, 25.6, 18.2; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{15}H_{22}NO_3$: 264.1600. Found: 264.1600.

(*E*)-1-(furan-2-yl)-3-hydroxy-5-methylhex-4-en-1-one *O*-methyl oxime:



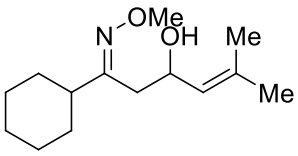
The title compound was prepared according to general procedure. The product was obtained as yellow oil. Yield: 68%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 (d, J = 1.2 Hz, 1H), 6.66 (d, J = 3.2 Hz, 1H), 6.44 (dd, J_1 = 3.6 Hz, J_2 = 2.0 Hz, 1H), 5.25 - 5.22 (m, 1H), 4.80 - 4.76 (m, 1H), 4.01 (s, 3H), 2.96 (dd, J_1 = 13.2 Hz, J_2 = 8.4 Hz, 1H), 2.79 (dd, J_1 = 12.8 Hz, J_2 = 4.8 Hz, 1H), 1.98 (d, J = 4.0 Hz. 1H), 1.68 (s, 3H), 1.65 (d, J = 0.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.9, 148.2, 143.8, 135.4, 127.2, 111.4, 110.9, 66.8, 62.4, 34.9, 25.7, 18.2; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{12}H_{18}NO_3$: 224.1287. Found: 224.1288.

(1*E*, 3*E*)-5-hydroxy-7-methyl-1-phenylocta-1,6-dien-3-one *O*-methyl oxime:



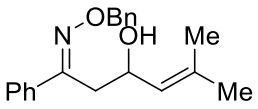
The title compound was prepared according to general procedure. The product was obtained as colorless oil. Yield: 72%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 - 7.45 (m, 2H), 7.36 - 7.33 (m, 2H), 7.30 - 7.26 (m, 1H), 6.92 (d, J = 16.4 Hz, 1H), 6.81 (d, J = 16.4 Hz, 1H), 5.30 - 5.27 (m, 1H), 4.77 - 4.75 (m, 1H), 3.96 (s, 3H), 2.93 (dd, J_1 = 13.2 Hz, J_2 = 8.0 Hz, 1H), 2.77 (dd, J_1 = 13.2 Hz, J_2 = 5.2 Hz, 1H), 2.08 (d, J = 3.2 Hz, 1H), 1.71 (d, J = 1.2 Hz, 3H), 1.68 (d, J = 1.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.7, 136.4, 135.2, 133.8, 128.8, 128.5, 127.5, 126.9, 125.4, 66.9, 62.0, 33.1, 25.7, 18.2; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{16}H_{22}NO_2$: 260.1651. Found: 260.1648.

(*E*)-1-cyclohexyl-3-hydroxy-5-methylhex-4-en-1-one *O*-methyl oxime:



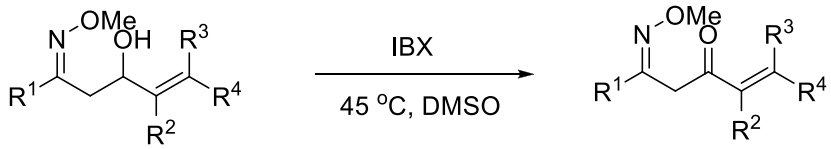
The title compound was prepared according to general procedure. The product was obtained as colorless oil. Yield: 82%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.22 - 5.19 (m, 1H), 4.71 - 4.65 (m, 1H), 3.83 (s, 3H), 2.69 (dd, J_1 = 13.2 Hz, J_2 = 8.8 Hz, 1H), 2.58 (d, J = 0.4 Hz, 1H), 2.26 (dd, J_1 = 12.8 Hz, J_2 = 4.4 Hz, 1H), 2.17 - 2.12 (m, 1H), 1.82 - 1.74 (m, 4H), 1.71 (d, J = 1.2 Hz, 3H), 1.69 (d, J = 1.2 Hz, 3H), 1.69 - 1.65 (m, 1H), 1.35 - 1.16 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.4, 134.5, 128.0, 66.7, 61.2, 44.5, 35.9, 30.4, 30.4, 26.2, 26.2, 26.0, 25.7, 18.2; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{14}H_{26}NO_2$: 240.1964. Found: 240.1964.

(*E*)-3-hydroxy-5-methyl-1-phenylhex-4-en-1-one *O*-benzyl oxime:



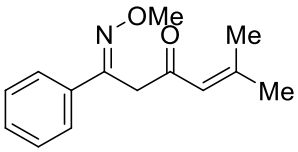
The title compound was prepared according to general procedure. The product was obtained as colorless oil. Yield: 80%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 - 7.64 (m, 2H), 7.43 - 7.23 (m, 8H), 5.24 (s, 2H), 5.20 - 5.17 (m, 1H), 4.76 - 4.73 (m, 1H), 3.09 (dd, J_1 = 13.2 Hz, J_2 = 8.0 Hz, 1H), 2.93 (dd, J_1 = 13.2 Hz, J_2 = 5.2 Hz, 1H), 2.00 (d, J = 4.0 Hz, 1H), 1.61 (d, J = 1.2 Hz, 3H), 1.49 (d, J = 1.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.4, 137.6, 136.1, 135.2, 129.2, 128.5, 128.4, 128.0, 127.5, 126.7, 76.5, 66.7, 35.5, 25.6, 18.1; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{20}H_{24}NO_2$: 310.1807. Found: 310.1798.

General procedure for β-keto oxime ethers



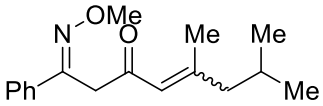
To a solution of *β*-oximino alcohol (1.0 mmol, 1.0 eq.) in DMSO (5 mL) at RT was added IBX (1.2 mmol, 1.2 eq.) in one portion, and the reaction mixture was stirred for 4h at 45 ℃. Upon completion as indicated by TLC, the reaction was quenched with 20 mL water and extracted with ethyl acetate, and dried over anhydrous Na_2SO_4. The crude material was purified by column chromatography using hexane: ethyl acetate (19:1).

(*E*)-1-(methoxyimino)-5-methyl-1-phenylhex-4-en-3-one:



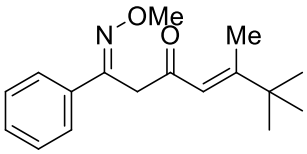
The title compound was prepared according to general procedure. The product was obtained as colorless oil. Yield: 87%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 - 7.63 (m, 2H), 7.36 - 7.33 (m, 3H), 6.15 (t, J = 1.2 Hz, 1H), 4.00 (s, 3H), 3.86 (s, 2H), 2.13 (d, J = 0.8 Hz, 3H), 1.87 (d, J = 0.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.6, 157.3, 152.4, 135.6, 129.2, 128.5, 126.3, 122.6, 62.2, 42.9, 27.8, 20.9; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{14}H_{18}NO_2$: 232.1338. Found: 232.1343.

(1*E*, 4*E*)-1-(methoxyimino)-5,7-dimethyl-1-phenyloct-4-en-3-one:



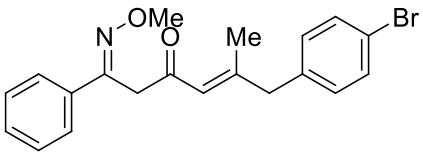
The title compound was prepared according to general procedure. The product was obtained as colorless oil in a cis : trans ratio of 21 : 79. Yield: 92%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 - 7.62 (m, 2H), 7.37 - 7.33 (m, 3H), 6.17 & 6.10 (s & d, J = 0.8 Hz, 1H), 4.00 & 3.99 (s & s, 3H), 3.86 & 3.84 (s & s, 2H), 2.53 & 1.97 (d & d, J = 7.2 Hz & 6.8 Hz, 2H), 2.09 & 1.87 (d & s, J = 1.2 Hz, 3H), 1.85 - 1.82 (m, 1H), 0.85 (d, J = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.8, 194.3, 160.4, 159.9, 152.5, 135.6, 129.2, 128.6, 128.5, 126.3, 126.3, 123.7, 123.2, 62.1, 50.8, 43.2, 43.1, 42.0, 27.5, 26.3, 25.9, 22.4, 19.5; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{17}H_{24}NO_2$: 274.1807. Found: 274.1812.

(1*E*, 4*E*)-1-(methoxyimino)-5,6,6-trimethyl-1-phenylhept-4-en-3-one:



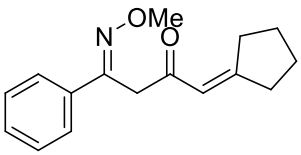
The title compound was prepared according to General procedure. The product was obtained as colorless oil. Yield: 92%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 - 7.63 (m, 2H), 7.36 - 7.34 (m, 3H), 6.23 (d, J = 0.8 Hz, 1H), 4.01 (s, 3H), 3.90 (s, 2H), 2.09 (d, J = 0.8 Hz, 3H), 1.07 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.7, 167.6, 152.5, 135.7, 129.2, 128.5, 126.3, 119.2, 62.1, 43.5, 38.1, 28.5, 15.9; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{17}H_{24}NO_2$: 274.1807. Found: 274.1808.

(1*E*, 4*E*)-6-(4-bromophenyl)-1-(methoxyimino)-5-methyl-1-phenylhex-4-en-3-one:



The title compound was prepared according to general procedure. The product was obtained as yellow oil. Yield: 89%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 - 7.60 (m, 2H), 7.41 - 7.39 (m, 2H), 7.36 - 7.34 (m, 3H), 6.96 (d, J = 8.4 Hz, 2H), 6.04 (d, J = 0.8 Hz, 1H), 3.95 (s, 3H), 3.84 (s, 2H), 3.33 (s, 2H), 2.05 (d, J = 0.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.9, 157.8, 152.1, 136.5, 135.5, 131.7, 131.1, 129.4, 128.6, 126.2, 123.7, 120.7, 62.2, 46.4, 43.1, 19.5; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{20}H_{21}NO_2Br$: 386.0756. Found: 386.0760.

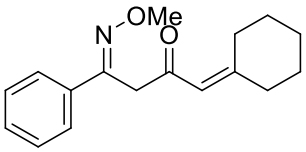
(*E*)-1-cyclopentylidene-4-(methoxyimino)-4-phenylbutan-2-one:



The title compound was prepared according to general procedure. The product was obtained as colorless oil. Yield: 84%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 - 7.64 (m, 2H), 7.37 - 7.34 (m, 3H), 6.32 (t, J = 2.4 Hz, 1H), 4.00 (s, 3H), 3.88 (s, 2H), 2.78 - 2.74 (m, 2H), 2.43 - 2.39 (m, 2H), 1.74 - 1.69 (m, 2H), 1.65 - 1.61 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.9,

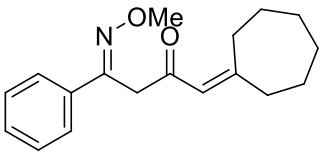
170.8, 152.5, 135.6, 129.2, 128.5, 126.3, 118.1, 62.2, 42.5, 36.4, 33.7, 26.5, 25.3; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{16}H_{20}NO_2$: 258.1494. Found: 258.1490.

(*E*)-1-cyclohexylidene-4-(methoxyimino)-4-phenylbutan-2-one:



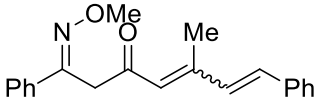
The title compound was prepared according to general procedure. The product was obtained as colorless oil. Yield: 95%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 - 7.62 (m, 2H), 7.37 - 7.33 (m, 3H), 6.05 (s, 1H), 4.00 (s, 3H), 3.86 (s, 2H), 2.77 (s, 2H), 2.16 - 2.13 (m, 2H), 1.69 - 1.63 (m, 2H), 1.57 - 1.54 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.4, 163.9, 152.5, 135.7, 129.2, 128.5, 126.3, 119.9, 62.2, 43.2, 38.1, 30.1, 28.8, 27.9, 26.2; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{17}H_{22}NO_2$: 272.1651. Found: 272.1655.

(*E*)-1-cycloheptylidene-4-(methoxyimino)-4-phenylbutan-2-one:



The title compound was prepared according to general procedure. The product was obtained as colorless oil. Yield: 85%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 - 7.63 (m, 2H), 7.37 - 7.34 (m, 3H), 6.14 (s, 1H), 4.00 (s, 3H), 3.86 (s, 2H), 2.84 - 2.81 (m, 2H), 2.35 - 2.32 (m, 2H), 1.63 - 1.61 (m, 4H), 1.50 - 1.48 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.5, 167.9, 152.6， 135.7, 129.2, 128.5, 126.3, 122.2, 62.1, 43.0, 39.2, 33.1, 29.8, 29.2, 28.1, 26.4; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{18}H_{24}NO_2$: 286.1807. Found: 286.1803.

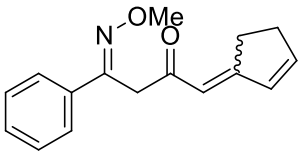
(1*E*, 4*E*)-1-(methoxyimino)-5-methyl-1,7-diphenylhepta-4,6-dien-3-one:



The title compound was prepared according to general procedure. The product was obtained as yellow oil in a cis : trans ratio of 56 :44. Yield: 86%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.38 & 6.76 (d & d, J = 16.4 Hz & 16.4 Hz, 1H), 7.68 - 7.66 (m, 2H), 7.53 - 7.45 (m, 2H), 7.37 -

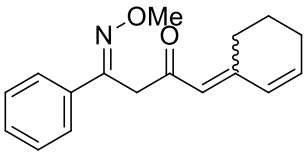
7.25 (m, 6H), 7.00 & 6.98 (d & d, *J* = 16.0 Hz & 16.4 Hz, 1H), 6.33 & 6.18 (s & s, 1H), 4.02 & 4.01 (d & d, *J* = 2.0 Hz & 2.0 Hz, 3H), 4.03 & 4.01 (s & s, 2H), 2.37 & 2.12 (s & s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 195.2, 194.7, 152.4, 152.4, 152.2, 150.7, 137.2, 136.6, 136.3, 135.9, 135.6, 132.2, 129.3, 129.3, 128.9, 128.8, 128.7, 128.6, 127.6, 127.1, 126.8, 126.3, 126.3, 125.6, 123.7, 62.2, 43.4, 43.3, 21.0, 14.3; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{21}H_{22}NO_2$: 320.1651. Found: 320.1648.

(4*E*)-1-(cyclopent-2-en-1-ylidene)-4-(methoxyimino)-4-phenylbutan-2-one:



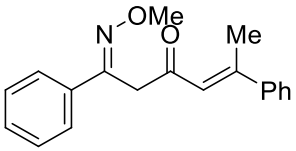
The title compound was prepared according to general procedure. The product was obtained as colorless oil in a cis : trans ratio of 33 : 67. Yield: 94%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 - 7.65 (m, 2H), 7.51 - 7.50 & 6.29 - 6.27 (m & m, 1H), 7.37 - 7.33 (m, 3H), 6.75 - 6.73 (m, 1H), 6.29 & 6.11 (s & s, 1H), 4.01 (s, 3H), 3.92 & 3.89 (s & s, 2H), 3.03 - 3.00 & 2.50 - 2.49 (m & m, 2H), 2.65 - 2.59 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.4, 193.4, 168.9, 166.7, 152.6, 151.8, 150.7, 135.7, 135.0, 132.9, 129.2, 128.5, 126.3, 115.1, 113.8, 62.2, 42.5, 42.5, 33.7, 31.4, 31.1; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{16}H_{18}NO_2$: 256.1338. Found: 256.1341.

(4*E*)-1-(cyclohex-2-en-1-ylidene)-4-(methoxyimino)-4-phenylbutan-2-one:



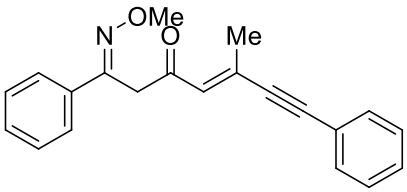
The title compound was prepared according to general procedure. The product was obtained as yellow oil in a cis : trans ratio of 37 : 63. Yield 90%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 - 7.63 (m, 2H), 7.50 - 7.47 & 6.08 - 6.05 (m & m, 1H), 7.37 - 7.33 (m, 3H), 6.33 - 6.25 (m, 1H), 6.01 & 5.91 (s & s, 1H), 4.00 (s, 3H), 3.90 & 3.89 (s & s, 2H), 2.95 - 2.91 & 2.37 - 2.33 (m & m, 2H), 2.23 - 2.16 (m, 2H), 1.81 - 1.75 & 1.69 - 1.62 (m & m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.1, 194.6, 154.1, 152.5, 152.4, 140.5, 140.3, 135.6, 130.5, 129.2, 128.5, 126.3, 126.0, 121.1, 119.2, 62.2, 43.2, 43.2, 32.5, 26.9, 26.4, 25.7, 22.7, 21.7; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{17}H_{20}NO_2$: 270.1494. Found: 270.1499.

(1*E*, 4*E*)-1-(methoxyimino)-1,5-diphenylhex-4-en-3-one:



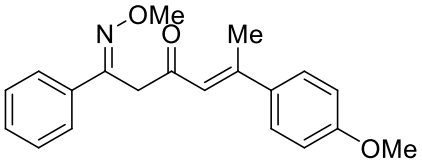
The title compound was prepared according to general procedure. The product was obtained as yellow oil. Yield 89%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 - 7.66 (m, 2H), 7.46 - 7.44 (m, 2H), 7.39 - 7.36 (m, 6H), 6.61 (d, J = 1.6 Hz, 1H), 4.02 (s, 3H), 3.99 (s, 2H), 2.52 (d, J = 1.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.1, 155.6, 152.3, 142.3, 135.6, 129.4, 129.3, 128.6, 128.6, 126.5, 126.3, 123.1, 62.3, 43.5, 18.6; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{19}H_{20}NO_2$: 294.1494. Found: 294.1498.

(1*E*, 4*E*)-1-(methoxyimino)-5-methyl-1,7-diphenylhept-4-en-6-yn-3-one:



The title compound was prepared according to general procedure. The product was obtained as yellow oil. Yield: 90%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 - 7.64 (m, 2H), 7.48 - 7.46 (m, 2H), 7.38 - 7.33 (m, 6H), 6.60 (d, J = 1.2 Hz, 1H), 4.02 (s, 3H), 3.92 (s, 2H), 2.35 (d, J = 1.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.3, 152.0, 138.0, 135.4, 132.0, 129.4, 129.3, 129.2, 128.6, 128.5, 126.3, 122.2, 94.7, 91.9, 62.3, 43.1, 20.6; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{21}H_{20}NO_2$: 318.1494. Found: 318.1497.

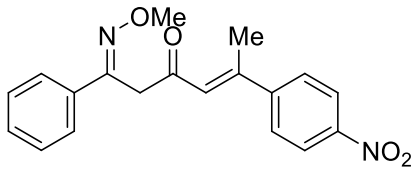
(1*E*, 4*E*)-1-(methoxyimino)-5-(4-methoxyphenyl)-1-phenylhex-4-en-3-one:



The title compound was prepared according to general procedure. The product was obtained as yellow oil. Yield 83%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 - 7.67 (m, 2H), 7.43 (dd, J_1 = 6.8 Hz, J_2 = 2.4 Hz, 2H), 7.37 - 7.35 (m, 3H), 6.81 (dd, J_1 = 6.8 Hz, J_2 = 2.0 Hz, 2H), 6.59 (d,

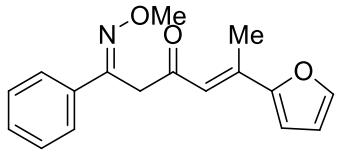
J = 0.8 Hz, 1H), 4.02 (s, 3H), 3.97 (s, 2H), 3.81 (s, 3H), 2.51 (d, *J* = 0.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.9, 160.8, 155.0, 152.5, 135.7, 134.3, 129.3, 128.6, 128.0, 126.3, 121.3, 114.0, 62.2, 55.4, 43.6, 18.3; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{19}H_{19}N_2O_4$: 339.1345. Found: 339.1342.

(1*E*, 4*E*)-1-(methoxyimino)-5-(4-nitrophenyl)-1-phenylhex-4-en-3-one:



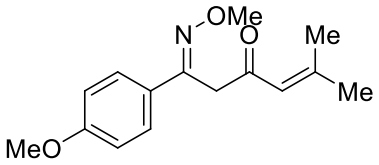
The title compound was prepared according to general procedure. The product was obtained as yellow oil. Yield 60%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.24 - 8.21 (m, 2H), 7.69 - 7.66 (m, 2H), 7.58 - 7.56 (m, 2H), 7.40 - 7.38 (m, 3H), 6.63 (d, *J* = 1.2 Hz, 1H), 4.03 (s, 3H), 4.01 (s, 2H), 2.51 (d, *J* = 1.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.9, 152.4, 151.9, 148.7, 148.0, 135.3, 129.5, 128.7, 127.4, 126.2, 125.6, 123.8, 62.3, 43.6, 18.4; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{19}H_{19}N_2O_4$: 339.1345. Found: 339.1342.

(1*E*, 4*E*)-5-(furan-2-yl)-1-(methoxyimino)-1-phenylhex-4-en-3-one:



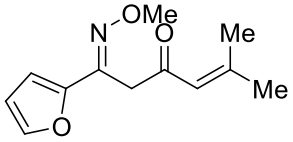
The title compound was prepared according to general procedure. The product was obtained as yellow oil. Yield: 87%. ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.68 - 7.66 (m, 2H), 7.46 (d, *J* = 1.6 Hz, 1H), 7.38 - 7.35 (m, 3H), 6.85 (s, 1H), 6.71 (d, *J* = 3.6 Hz, 1H), 6.47 (dd, J_1 = 3.6 Hz, J_2 = 1.6 Hz, 1H), 4.03 (s, 3H), 3.99 (s, 2H), 2.42 (d, *J* = 1.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 194.9, 154.4, 152.4, 144.3, 141.9, 135.6, 129.3, 128.5, 126.3, 117.9, 112.9, 112.3, 62.2, 43.5, 15.4; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{17}H_{18}NO_3$: 284.1287. Found: 284.1288.

(*E*)-1-(methoxyimino)-1-(4-methoxyphenyl)-5-methylhex-4-en-3-one:



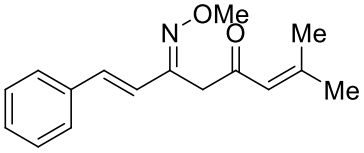
The title compound was prepared according to general procedure. The product was obtained as colorless oil. Yield: 92%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61 - 7.58 (m, 2H), 6.89 - 6.86 (m, 2H), 6.15 (t, J = 0.8 Hz, 1H), 3.98 (s, 3H), 3.84 (s, 2H), 3.81 (s, 3H), 2.13 (d, J = 1.2 Hz, 3H), 1.87 (d, J = 1.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.8, 160.5, 157.3, 151.9, 128.1, 127.6, 122.6, 113.9, 62.0, 55.3, 42.9, 27.8, 21.0; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{15}H_{20}NO_3$: 262.1443. Found: 262.1437.

(*E*)-1-(furan-2-yl)-1-(methoxyimino)-5-methylhex-4-en-3-one:



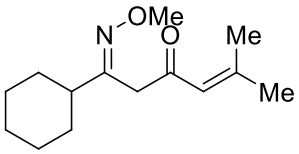
The title compound was prepared according to general procedure. The product was obtained as yellow oil. Yield: 94%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 (d, J = 1.2 Hz, 1H), 6.65 - 6.64 (m, 1H), 6.43 (dd, J_1 = 3.6 Hz, J_2 = 2.0 Hz, 1H), 6.13 (d, J = 1.2 Hz, 1H), 4.01 (s, 3H), 3.75 (s, 2H), 2.14 (d, J = 1.2 Hz, 3H), 1.88 (d, J = 1.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.8, 157.7, 149.4, 144.7, 143.8, 122.3, 111.5, 110.8, 62.5, 42.0, 27.8, 21.0; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{12}H_{16}NO_3$: 222.1130. Found: 222.1130.

(6*E*, 7*E*)-6-(methoxyimino)-2-methyl-8-phenylocta-2,7-dien-4-one:



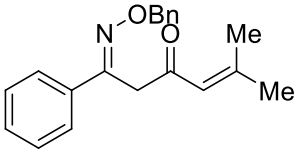
The title compound was prepared according to general procedure. The product was obtained as yellow oil. Yield: 88%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 - 7.44 (m, 2H), 7.35 - 7.31 (m, 2H), 7.28 - 7.26 (m, 1H), 6.89 (d, J = 16.4 Hz, 1H), 6.82 (d, J = 16.4 Hz, 1H), 6.13 (s, 1H), 3.97 (s, 3H), 3.73 (s, 2H), 2.15 (s, 3H), 1.89 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.3, 157.5, 153.3, 136.3, 133.9, 128.7, 128.5, 127.0, 125.0, 122.4, 62.2, 40.9, 27.8, 21.0; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{16}H_{20}NO_2$: 258.1494. Found: 258.1494.

(*E*)-1-cyclohexyl-1-(methoxyimino)-5-methylhex-4-en-3-one:



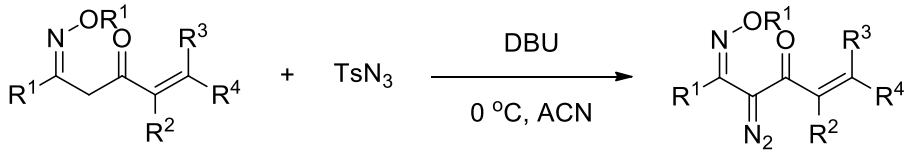
The title compound was prepared according to general procedure. The product was obtained as yellow oil. Yield: 88%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.10 (t, J = 1.2 Hz, 1H), 3.81 (s, 3H), 3.36 (s, 2H), 2.24 - 2.18 (m, 1H), 2.13 (d, J = 0.8 Hz, 3H), 1.89 (d, J = 1.2 Hz, 3H), 1.79 - 1.74 (m, 4H), 1.68 - 1.65 (m, 1H), 1.29 - 1.24 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.9, 158.8, 156.2, 123.1, 61.2, 43.9, 42.6, 30.1, 27.7, 26.0, 25.9, 20.8; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{14}H_{24}NO_2$: 238.1807. Found: 238.1805.

(*E*)-1-(benzyloxyimino)-5-methyl-1-phenylhex-4-en-3-one:



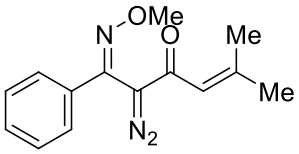
The title compound was prepared according to general procedure. The product was obtained as yellow oil. Yield: 89%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 - 7.63 (m, 2H), 7.40 - 7.27 (m, 8H), 6.11 (t, J = 1.2 Hz, 1H), 5.26 (s, 2H), 3.88 (s, 2H), 2.10 (d, J = 0.8 Hz, 3H), 1.79 (d, J = 0.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.7, 157.3, 152.7, 137.7, 135.6, 129.3, 128.5, 128.4, 128.3, 127.9, 126.4, 122.8, 76.5, 43.2, 27.7, 21.0; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{20}H_{22}NO_2$: 308.1651. Found: 308.1646.

General procedure for *α*-diazo-β-keto oxime ethers



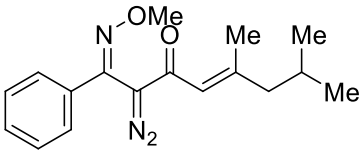
To a solution of *β*-oximino ketones (0.5 mmol, 1.0 eq.) and 4-methylbenzenesulfonyl azide (0.55 mmol, 1.1 eq.) in CH_3CN (5 mL) was added DBU (0.55 mmol, 1.1 eq.) dropwise at 0 °C. The resulting orange color solution was stirred at 0 °C for 3 h and slowly brought to RT. Upon completion as indicated by TLC, the solvent was removed under reduced pressure, and the crude material was purified by flash chromatography using hexane - ethyl acetate (19:1).

(*Z*)-2-diazo-1-(methoxyimino)-5-methyl-1-phenylhex-4-en-3-one (1a):



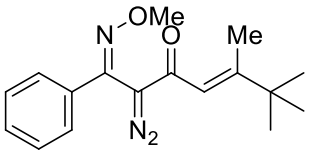
The title compound was prepared according to the general procedure. The product was obtained as yellow oil. Yield: 94%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 - 7.57 (m, 2H), 7.41 - 7.36 (m, 3H), 5.58 (s, 1H), 4.07 (s, 3H), 2.08 (d, J = 0.8 Hz, 3H), 1.62 (d, J = 0.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 183.7, 154.2, 144.5, 134.1, 130.0, 128.7, 127.7, 121.3, 62.7, 27.4, 20.7; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{14}H_{16}N_3O_2$: 258.1243. Found: 258.1245.

(1*Z*, 4*E*)-2-diazo-1-(methoxyimino)-5,7-dimethyl-1-phenyloct-4-en-3-one (1b):



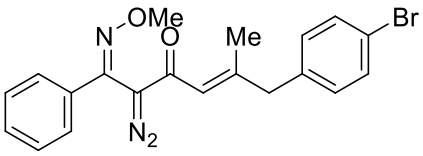
The title compound was prepared according to the general procedure. The product was obtained as yellow oil. Yield: 69%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 - 7.57 (m, 2H), 7.39 - 7.37 (m, 3H), 5.56 (s, 1H), 4.07 (s, 3H), 2.48 (d, J = 6.8 Hz, 2H), 1.86 - 1.80 (m, 1H), 1.57 (s, 3H), 0.87 (d, J = 6.4 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 183.4, 157.5, 144.5, 134.2, 130.0, 128.7, 127.8, 122.5, 62.7, 42.1, 27.4, 25.4, 22.5; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{17}H_{22}N_3O_2$: 300.1712. Found: 300.1706.

(1*Z*, 4*E*)-2-diazo-1-(methoxyimino)-5,6,6-trimethyl-1-phenylhept-4-en-3-one (1c):



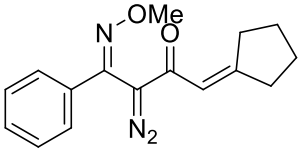
The title compound was prepared according to the general procedure. The product was obtained as yellow oil. Yield: 88%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 - 7.55 (m, 2H), 7.40 - 7.34 (m, 3H), 5.50 (s, 1H), 4.08 (s, 3H), 2.02 (d, J = 1.2 Hz, 3H), 0.72 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 185.0, 163.8, 144.7, 134.6, 130.0, 128.7, 128.0, 119.1, 62.7, 37.6, 28.1, 15.6; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{17}H_{22}N_3O_2$: 300.1712. Found: 300.1706.

(1*Z*, 4*E*)-6-(4-bromophenyl)-2-diazo-1-(methoxyimino)-5-methyl-1-phenylhex-4-en-3-one (1d):



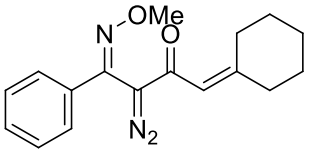
The title compound was prepared according to the general procedure. The product was obtained as yellow oil. Yield: 83%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 - 7.52 (m, 2H), 7.45 - 7.39 (m, 3H), 7.29 - 7.27 (m, 2H), 6.58 (d, J = 8.4 Hz, 1H), 5.41 (s, 1H), 4.07 (s, 3H), 3.06 (s, 2H), 2.04 (d, J = 0.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 183.9, 155.0, 144.1, 136.2, 134.2, 131.5, 130.8, 130.0, 128.8, 127.7, 122.6, 120.5, 62.8, 46.2, 19.4; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{20}H_{19}N_3O_2Br$: 412.0661. Found: 412.0662.

(*Z*)-1-cyclopentylidene-3-diazo-4-(methoxyimino)-4-phenylbutan-2-one (1e):



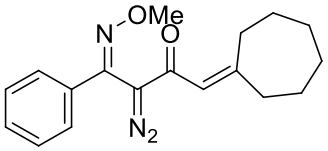
The title compound was prepared according to the general procedure. The product was obtained as yellow oil. Yield: 90%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61 - 7.59 (m, 2H), 7.40 - 7.38 (m, 3H), 5.76 (s, 1H), 4.07 (s, 3H), 2.78 - 2.75 (m, 2H), 2.17 - 2.14 (m, 2H), 1.70 - 1.67 (m, 2H), 1.61 - 1.55 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 182.9, 167.9, 144.5, 134.1, 130.0, 128.8, 127.7, 116.4, 62.7, 36.2, 33.4, 26.5, 25.2; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{16}H_{18}N_3O_2$: 284.1399. Found: 284.1398.

(*Z*)-1-cyclohexylidene-3-diazo-4-(methoxyimino)-4-phenylbutan-2-one (1f):



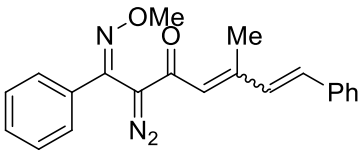
The title compound was prepared according to the general procedure. The product was obtained as yellow oil. Yield: 94%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 - 7.57 (m, 2H), 7.41 - 7.35 (m, 3H), 5.46 (s, 1H), 4.07 (s, 3H), 2.67 (t, J = 5.6 Hz, 2H), 1.84 (t, J = 7.5 Hz, 2H), 1.55 - 1.50 (m, 4H), 1.41 - 1.38 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 184.4, 160.6, 144.6, 134.2, 129.9, 128.6, 127.9, 119.0, 62.7, 37.8, 30.3, 28.5, 27.8, 26.1; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{17}H_{20}N_3O_2$: 298.1556. Found: 298.1555.

(*Z*)-1-cycloheptylidene-3-diazo-4-(methoxyimino)-4-phenylbutan-2-one (1g):



The title compound was prepared according to the general procedure. The product was obtained as yellow oil. Yield: 97%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 - 7.58 (m, 2H), 7.40 - 7.35 (m, 3H), 5.53 (s, 1H), 4.07 (s, 3H), 2.82 - 2.78 (m, 2H), 2.02 - 2.00 (m, 2H), 1.68 - 1.59 (m, 2H), 1.43 - 1.33 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 183.5, 164.8, 144.7, 134.3, 129.9, 128.7, 127.8, 121.2, 62.7, 38.8, 32.7, 29.5, 29.1, 28.0, 26.4; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{18}H_{22}N_3O_2$: 312.1712. Found: 312.1712.

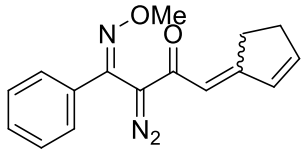
(1*Z*)-2-diazo-1-(methoxyimino)-5-methyl-1,7-diphenylhepta-4,6-dien-3-one (1h):



The title compound was prepared according to the general procedure. The product was obtained as yellow oil in cis : trans ratio of 31 : 69. Yield: 90%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.27 & 6.80 (d & d, J = 16.4 Hz & 16.0 Hz, 1H), 7.81 - 7.59 (m, 2H), 7.53 - 7.27 (m, 8H), 6.86 & 6.38 (d & d, J = 16.4 Hz & 16.0 Hz, 1H), 5.77 & 5.57 (s, 1H), 4.08 (s, 3H), 2.34 & 1.81 (d & d , J = 1.2 Hz & 0.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 183.6, 183.4, 149.9,

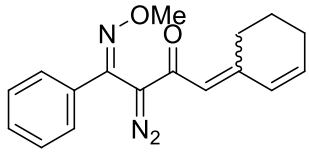
148.1, 144.4, 144.3, 136.7, 136.3, 136.3, 134.8, 134.1, 134.1, 131.9, 130.2, 130.1, 129.8, 128.8, 128.7, 127.9, 127.8, 127.5, 127.0, 126.7, 124.5, 123.0, 66.8, 62.8, 62.8, 21.6, 20.7, 14.7, 14.5; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{21}H_{20}N_3O_2$: 346.1556. Found: 346.1561.

(4*Z*)-1-(cyclopent-2-enylidene)-3-diazo-4-(methoxyimino)-4-phenylbutan-2-one (1i):



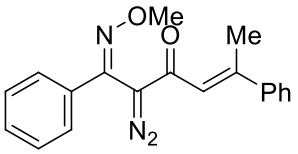
The title compound was prepared according to the general procedure. The product was obtained as yellow oil in a cis : trans ratio of 39 : 61. Yield: 95%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 - 7.60 (m, 2H), 7.48 - 7.39 & 6.07 - 5.99 (m & m, 1H), 7.38 - 7.36 (m, 3H), 6.69 - 6.67 & 6.63 - 6.60 (m & m, 1H), 5.72 & 5.54 (s & s, 1H), 4.07 (s, 3H), 3.06 - 3.03 & 2.44 - 2.42 (m & m, 2H), 2.59 - 2.57 & 2.38 - 2.37 (m & m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 183.1, 182.4, 166.6, 164.3, 151.0, 149.7, 144.6, 135.0, 134.2, 134.1, 132.8, 130.0, 130.0, 129.0, 128.8, 128.7, 128.6, 127.8, 127.7, 113.6, 112.4, 62.7, 33.7, 31.3, 31.0, 30.8; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{16}H_{16}N_3O_2$: 282.1243. Found: 282.1238.

(4*Z*)-1-(cyclohex-2-enylidene)-3-diazo-4-(methoxyimino)-4-phenylbutan-2-one (1j):



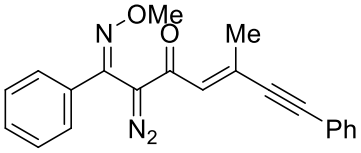
The title compound was prepared according to the general procedure. The product was obtained as yellow oil in a cis : trans ratio of 37: 63. Yield 91%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61 - 7.58 (m, 2H), 7.42 - 7.35 & 5.70 (m & d, J = 10.0 Hz, 3H & 1H), 6.24 - 6.19 & 6.63 - 6.60 (m & m, 1H), 5.45 & 5.32 (s & s, 1H), 4.07 (s, 3H), 2.94 - 2.91 & 2.04 - 2.01 (m & m, 2H), 2.17 - 2.13 (m, 2H), 1.67 - 1.62 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 183.5, 183.3, 152.0, 150.1, 144.6, 144.5, 139.5, 139.2, 134.1, 130.3, 130.1, 130.0, 128.7, 128.7, 127.8, 127.7, 125.8, 119.8, 118.3, 62.7, 32.4, 26.9, 26.2, 25.6, 22.6, 21.8; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{17}H_{18}N_3O_2$: 296.1399. Found: 296.1407.

(1*Z*, 4*E*)-2-diazo-1-(methoxyimino)-1,5-diphenylhex-4-en-3-one (1k):



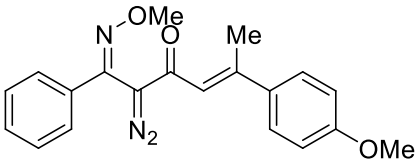
The title compound was prepared according to the general procedure. The product was obtained as yellow solid. Mp: 61 - 62 ℃. Yield 92%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 - 7.65 (m, 2H), 7.44 - 7.42 (m, 3H), 7.25 - 7.23 (m, 1H), 7.20 - 7.19 (m, 2H), 6.85 (d, J = 7.2 Hz, 2H), 5.94 (s, 1H), 4.08 (s, 3H), 2.48 (d, J = 1.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 183.8, 152.6, 144.3, 142.2, 134.4, 130.2, 128.9, 128.9, 128.3, 128.0, 126.2, 122.5, 62.8, 18.4; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{19}H_{18}N_3O_2$: 320.1399. Found: 320.1395.

(1*Z*, 4*E*)-2-diazo-1-(methoxyimino)-5-methyl-1,7-diphenylhept-4-en-6-yn-3-one (1l):



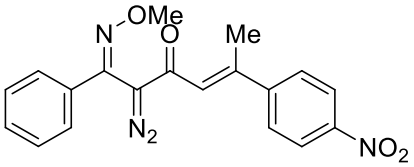
The title compound was prepared according to the general procedure. The product was obtained as yellow oil. Yield: 83%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 - 7.61 (m, 2H), 7.43 - 7.39 (m, 3H), 7.37 - 7.30 (m, 5H), 6.07 (s, 1H), 4.08 (s, 3H), 2.33 (d, J = 1.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 182.7, 143.9, 135.5, 133.7, 131.9, 130.3, 129.1, 128.9, 128.4, 128.0, 127.7, 122.3, 93.6, 91.7, 62.8, 20.4; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{21}H_{18}N_3O_2$: 344.1399. Found: 344.1391.

(1*E*, 4*E*)-1-(methoxyimino)-5-(4-methoxyphenyl)-1-phenylhex-4-en-3-one (1m):



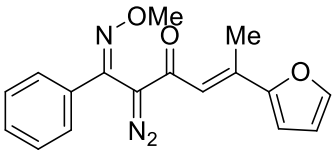
The title compound was prepared according to general procedure. The product was obtained as yellow oil. Yield 97%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 - 7.65 (m, 2H), 7.43 - 7.42 (m, 3H), 6.83 (d, J = 7.2 Hz, 2H), 6.70 (dd, J_1 = 6.8 Hz, J_2 = 2.4 Hz, 2H), 5.91 (s, 1H), 4.08 (s, 3H), 3.77 (s, 3H), 2.48 (d, J = 1.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 183.6, 160.4, 152.2, 144.5, 134.5, 134.3, 130.1, 128.9, 128.0, 127.7, 120.8, 113.6, 62.8, 55.3, 18.2; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{19}H_{19}N_2O_4$: 339.1345. Found: 339.1342.

(1*Z*, 4*E*)-2-diazo-1-(methoxyimino)-5-(4-nitrophenyl)-1-phenylhex-4-en-3-one (1n):



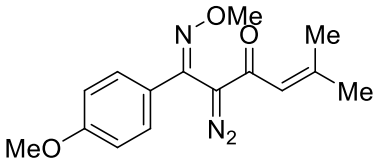
The title compound was prepared according to the general procedure. The product was obtained as yellow solid. Mp: 123 - 124 ℃. Yield 83%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 - 8.02 (m, 2H), 7.67 - 7.64 (m, 2H), 7.46 - 7.45 (m, 3H), 6.91 (d, *J* = 8.8 Hz, 2H), 5.92 (d, *J* = 1.2 Hz, 1H), 4.10 (s, 3H), 2.46 (d, *J* = 1.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 183.3, 149.1, 148.6, 147.7, 143.8, 134.5, 130.3, 129.1, 128.1, 127.0, 125.3, 123.5, 62.9, 18.2; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{19}H_{17}N_4O_4$: 365.1250. Found: 365.1245.

(1*Z*, 4*E*)-2-diazo-5-(furan-2-yl)-1-(methoxyimino)-1-phenylhex-4-en-3-one (1o):



The title compound was prepared according to the general procedure. The product was obtained as yellow oil. Yield: 93%. ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.64 - 7.62 (m, 2H), 7.38 - 7.36 (m, 3H), 7.28 (d, *J* = 1.2 Hz, 1H), 6.47 (d, *J* = 3.2 Hz, 1H), 6.36 (dd, J_1 = 3.2 Hz, J_2 = 1.6 Hz, 1H), 6.28 (s, 1H), 4.08 (s, 3H), 2.40 (d, *J* = 0.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 183.3, 154.4, 144.5, 143.8, 139.8, 134.1, 130.0, 128.7, 127.8, 117.0, 112.0, 111.7, 62.7, 15.2; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{17}H_{16}N_3O_3$: 310.1192. Found: 310.1197.

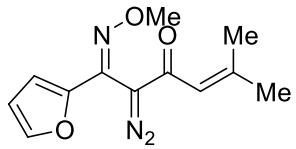
(*Z*)-2-diazo-1-(methoxyimino)-1-(4-methoxyphenyl)-5-methylhex-4-en-3-one (1p):



The title compound was prepared according to the general procedure. The product was obtained as yellow oil. Yield: 92%. ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.53 (dd, J_1 = 6.8 Hz, J_2 = 2.0 Hz, 2H), 6.90 (dd, J_1 = 6.8 Hz, J_2 = 2.0 Hz, 2H), 5.62 (s, 1H), 4.04 (s, 3H), 3.83 (s, 3H), 2.10 (d, *J* = 1.2 Hz, 3H), 1.65 (d, *J* = 0.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 183.8,

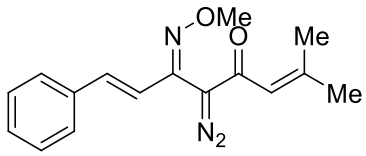
161.1, 154.2, 144.0, 129.1, 126.4, 121.2, 114.1, 62.6, 55.4, 27.5, 20.8; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{15}H_{18}N_3O_3$: 288.1348. Found: 288.1361.

(*E*)-2-diazo-1-(furan-2-yl)-1-(methoxyimino)-5-methylhex-4-en-3-one (1q):



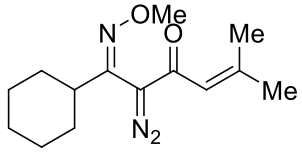
The title compound was prepared according to the general procedure. The product was obtained as yellow oil. Yield: 94%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (dd, J_1 = 2.0 Hz, J_2 = 0.8 Hz, 1H), 6.67 (dd, J_1 = 3.6 Hz, J_2 = 0.8 Hz, 1H), 6.47 (dd, J_1 = 3.6 Hz, J_2 = 2.0 Hz, 1H), 5.71 (s, 1H), 4.07 (s, 3H), 2.15 (d, J = 1.2 Hz, 3H), 1.76 (d, J = 0.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 183.2, 154.7, 147.4, 144.4, 135.9, 120.4, 112.5, 111.8, 63.0, 27.6, 20.8; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{12}H_{14}N_3O_3$: 248.1035. Found: 248.1041.

(6*Z*, 7*E*)-5-diazo-6-(methoxyimino)-2-methyl-8-phenylocta-2,7-dien-4-one (1r):



The title compound was prepared according to the general procedure. The product was obtained as yellow oil. Yield: 90%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 - 7.43 (m, 2H), 7.37 - 7.30 (m, 3H), 6.92 (d, J = 16.4 Hz, 1H), 6.86 (d, J = 16.4 Hz, 1H), 5.91 (s, 1H), 4.03 (s, 3H), 2.20 (d, J = 0.8 Hz, 3H), 1.81 (d, J = 1.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 183.7, 154.9, 144.0, 136.5, 135.8, 128.9, 128.8, 127.1, 122.9, 120.6, 62.8, 27.7, 20.9; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{16}H_{18}N_3O_2$: 314.1116. Found: 314.1111.

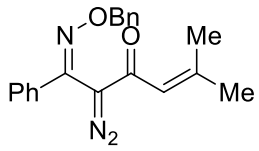
(*Z*)-1-cyclohexyl-2-diazo-1-(methoxyimino)-5-methylhex-4-en-3-one (1s):



The title compound was prepared according to the general procedure. The product was obtained as yellow oil. Yield: 88%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.96 (t, J = 1.2 Hz, 1H),

3.88 (s, 3H), 2.67 - 2.61 (m, 1H), 2.13 (d, *J* = 0.8 Hz, 3H), 1.94 - 1.90 (m, 2H), 1.90 (d, *J* = 1.2 Hz, 3H), 1.79 - 1.76 (m, 2H), 1.69 - 1.66 (m, 1H), 1.41 - 1.18 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 183.9, 154.3, 148.7, 120.0, 61.9, 41.5, 31.3, 27.5, 26.3, 26.1, 20.7; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{14}H_{22}N_3O_2$: 264.1712. Found: 264.1714.

(*Z*)-1-((benzyloxy)imino)-2-diazo-5-methyl-1-phenylhex-4-en-3-one (1t):

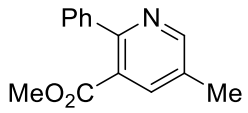


The title compound was prepared according to the general procedure. The product was obtained as yellow oil. Yield: 93%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 - 7.56 (m, 2H), 7.40 - 7.33 (m, 8H), 5.56 (s, 1H), 5.28 (s, 2H), 2.05 (d, *J* = 0.8 Hz, 3H), 1.59 (d, *J* = 2.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 183.7, 154.2, 144.8, 136.9, 134.1, 130.0, 128.6, 128.5, 128.4, 128.2, 127.8, 121.3, 76.7, 27.4, 20.8; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{20}H_{20}N_3O_2$: 334.1556. Found: 334.1551.

General procedure for pyridines

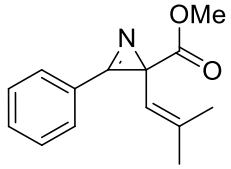
A solution of diazo compound (0.2 mmol) and $Rh_2(OAc)_4$ (0.004 mmol, 2 mol%) in chlorobenzene (2.0 mL) was stirred at 130 ℃ until the starting material was fully consumed. The reaction mixture was concentrated under reduced pressure to give the crude material which was purified by flash chromatography using hexane - ethyl acetate (9:1) to give desired product.

Methyl 5-methyl-2-phenylnicotinate (2a):



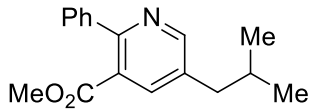
The title compound was prepared according to the general procedure. The product was obtained as colorless oil. Yield: 74%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.60 (d, J = 1.6 Hz, 1H), 7.90 (d, J = 1.6 Hz, 1H), 7.53 - 7.50 (m, 2H), 7.45 - 7.39 (m, 3H), 3.68 (s, 3H), 2.42 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.8, 156.1, 151.8, 140.0, 138.2, 131.3, 128.5, 128.1, 126.4, 52.3, 17.9; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{14}H_{14}NO_2$: 228.1025. Found: 228.1027.

Methyl 2-(2-methylprop-1-enyl)-3-phenyl-2*H*-azirine-2-carboxylate (1a'):



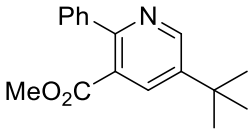
The title compound was prepared according to the general procedure. The product was obtained as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 - 7.86 (m, 2H), 7.63 - 7.55 (m, 3H), 5.65 (t, J = 1.2 Hz, 1H), 3.71 (s, 3H), 1.76 (d, J = 1.2 Hz, 3H), 1.68 (d, J = 0.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.6, 161.7, 140.5, 133.5, 130.1, 129.4, 122.9, 118.9, 52.6, 37.9, 26.2, 19.5; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{14}H_{16}NO_2$: 230.1181. Found: 230.1180.

Methyl 5-isobutyl-2-phenylnicotinate (2b):



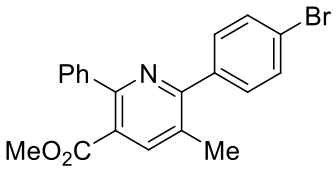
The title compound was prepared according to the general procedure. The product was obtained as colorless oil. Yield: 65%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.56 (d, J = 2.4 Hz, 1H), 7.87 (d, J = 2.0 Hz, 1H), 7.54 - 7.52 (m, 2H), 7.45 - 7.40 (m, 3H), 3.70 (s, 3H), 2.56 (d, J = 6.8 Hz, 2H), 1.97 - 1.91 (m, 1H), 0.96 (d, J = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.9, 156.4, 152.0, 140.0, 138.2, 135.0, 128.5, 128.1, 126.4, 52.3, 41.7, 30.0, 22.2; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{17}H_{20}NO_2$: 270.1494. Found: 270.1498.

Methyl 5-tert-butyl-2-phenylnicotinate (2c):



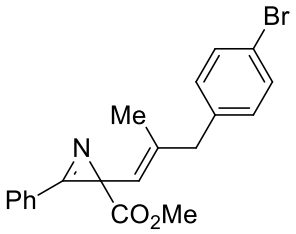
The title compound was prepared according to the general procedure. The product was obtained as colorless oil. Yield: 72%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.81 (d, J = 2.4 Hz, 1H), 8.06 (d, J = 2.4 Hz, 1H), 7.54 - 7.52 (m, 2H), 7.43 - 7.41 (m, 3H), 3.69 (s, 3H), 1.41 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.1, 155.9, 149.2, 144.2, 140.0, 134.7, 128.5, 128.1, 126.2, 52.3, 33.6, 30.9; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{17}H_{20}NO_2$: 270.1494. Found: 270.1492.

Methyl 6-(4-bromophenyl)-5-methyl-2-phenylnicotinate (2d):



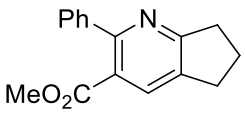
The title compound was prepared according to the general procedure. The product was obtained as colorless oil. Yield: 77%. 1H NMR (400 MHz, CDCl3) δ 8.00 (d, J = 0.4 Hz, 1H), 7.60 - 7.57 (m, 4H), 7.56 - 7.49 (m, 2H), 7.42 - 7.40 (m, 3H), 3.71 (s, 3H), 2.44 (s, 3H); ^{13}C NMR (100 MHz, CDCl3) δ 168.6, 158.9, 156.1, 140.6, 139.9, 138.7, 131.4, 130.9, 128.9, 128.7, 128.6, 128.1, 125.3, 122.9, 52.3, 19.6; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{20}H_{17}NO_2Br$: 382.0443. Found: 382.0447.

(E)-methyl 2-(3-(4-bromophenyl)-2-methylprop-1-en-1-yl)-3-phenyl-2H-azirine-2-carboxylate (1d'):



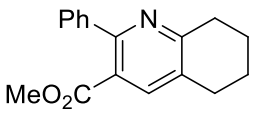
The title compound was prepared according to the general procedure at 60°C for 12h. The product was obtained as colorless oil. Yield: 92%. 1H NMR (400 MHz, CDCl3) δ 7.87 - 7.85 (m, 2H), 7.65 - 7.63 (m, 1H), 7.62 - 7.55 (m, 2H), 7.39 - 7.37 (m, 2H), 7.06 - 7.04 (m, 2H), 5.77 (d, *J* = 1.2 Hz, 1H), 3.71 (s, 3H), 3.29 (s, 2H), 1.59 (s, 3H); ^{13}C NMR (100 MHz, CDCl3) *δ* 172.3, 161.4, 142.1, 138.2, 133.6, 131.4, 130.6, 130.1, 129.4, 122.8, 121.3, 120.1, 52.7, 45.7, 37.8, 17.5; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{20}H_{19}BrNO_2$: 384.0599. Found: 384.0602.

Methyl 2-phenyl-6,7-dihydro-5H-cyclopenta[b]pyridine-3-carboxylate (2e):



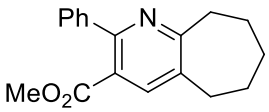
The title compound was prepared according to the general procedure. The product was obtained as yellow oil. Yield: 75%. ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.91 (s, 1H), 7.50 - 7.41 (m, 2H), 7.39 - 7.38 (m, 3H), 3.65 (s, 3H), 3.10 (t, *J* = 7.6 Hz, 2H), 3.02 (t, *J* = 7.6 Hz, 2H), 2.22 - 2.18 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 169.2, 168.5, 157.7, 140.7, 135.3, 133.6, 128.5, 128.2, 128.1, 124.6, 52.2, 34.6, 30.3, 23.2; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{16}H_{16}NO_2$: 254.1181. Found: 254.1188.

Methyl 2-phenyl-5,6,7,8-tetrahydroquinoline-3-carboxylate (2f):



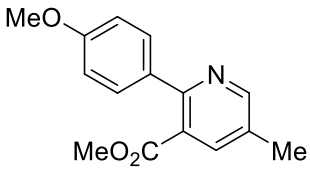
The title compound was prepared according to the general procedure. The product was obtained as yellow oil. Yield: 80%. ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.81 (s, 1H), 7.50 - 7.40 (m, 2H), 7.39 - 7.37 (m, 3H), 3.66 (s, 3H), 3.02 - 2.99 (m, 2H), 2.86 - 2.83 (m, 2H), 1.95 - 1.91 (m, 2H), 1.88 - 1.84 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 168.7, 160.1, 156.2, 140.5, 138.5, 130.5, 128.5, 128.2, 128.1, 124.0, 52.1, 32.9, 28.3, 22.9, 22.6; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{17}H_{18}NO_2$: 268.1338. Found: 268.1336.

Methyl 2-phenyl-6,7,8,9-tetrahydro-5H-cyclohepta[b]pyridine-3-carboxylate (2g):



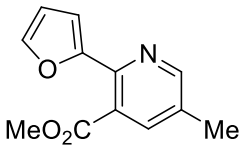
The title compound was prepared according to the general procedure. The product was obtained as colorles oil. Yield: 73%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (s, 1H), 7.53 - 7.50 (m, 2H), 7.43 - 7.35 (m, 3H), 3.67 (s, 3H), 3.13 (t, J = 5.6 Hz, 2H), 2.86 (dd, J_1 = 6.8 Hz, J_2 = 4.0 Hz, 2H), 1.91 - 1.89 (m, 2H), 1.74 - 1.71 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.9, 165.8, 155.6, 140.4, 138.1, 136.3, 128.6, 128.3, 128.1, 124.0, 52.1, 39.6, 34.7, 32.4, 27.9, 26.4; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{18}H_{20}NO_2$: 282.1494. Found: 282.1496.

Methyl 2-(4-methoxyphenyl)-5-methylnicotinate (2h):



The title compound was prepared according to the general procedure. The product was obtained as yellow oil. Yield: 72%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.57 (d, J = 1.2 Hz, 1H), 7.86 (d, J = 1.2 Hz, 1H), 7.48 (d, J = 8.8 Hz, 2H), 6.95 (d, J = 8.8 Hz, 2H), 3.85 (s, 3H), 3.72 (s, 3H), 2.40 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.1, 160.0, 155.6, 151.7, 138.2, 132.4, 130.8, 129.9, 126.1, 113.6, 55.3, 52.3, 17.9; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{15}H_{16}NO_3$: 258.1130. Found: 258.1128.

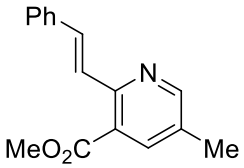
Methyl 2-(furan-2-yl)-5-methylnicotinate (2i):



The title compound was prepared according to the general procedure. The product was obtained as yellow solid. Mp: 96 - 97 ℃. Yield: 64%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.52 (d, J = 2.0 Hz, 1H), 7.70 (d, J = 1.2 Hz, 1H), 7.51 (d, J = 0.8 Hz, 1H), 6.98 (d, J = 3.2 Hz, 1H), 6.52 (dd, J_1 = 3.6 Hz, J_2 = 2.0 Hz, 1H), 3.90 (s, 3H), 2.38 (s, 3H); ^{13}C NMR (100 MHz,

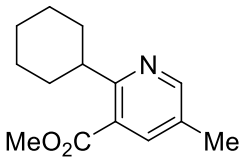
$CDCl_3$) δ 168.8, 152.3, 151.4, 144.3, 143.6, 137.1, 131.4, 125.2, 111.9, 110.5, 52.6, 18.0; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{12}H_{12}NO_3$: 218.0817. Found: 218.0820.

(E)-Methyl 5-methyl-2-styrylnicotinate (2j):



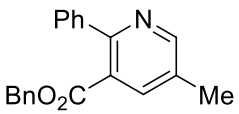
The title compound was prepared according to the general procedure. The product was obtained as colorless oil. Yield: 70%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.56 (d, J = 2.0 Hz, 1H), 8.10 (d, J = 15.6 Hz, 1H), 8.01 (d, J = 0.8 Hz, 1H), 7.87 (d, J = 15.6 Hz, 1H), 7.64 - 7.62 (m, 2H), 7.39 - 7.35 (m, 2H), 7.31 - 7.26 (m, 1H), 3.96 (s, 3H), 2.38 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.1, 152.8, 138.9, 137.0, 135.0, 131.2, 128.6, 128.4, 127.5, 125.0, 123.5, 52.4, 18.1; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{16}H_{16}NO_2$: 254.1181. Found: 254.1179.

Methyl 2-cyclohexyl-5-methylnicotinate (2k):



The title compound was prepared according to the general procedure. The product was obtained as yellow oil. Yield: 65%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.52 (d, J = 1.6 Hz, 1H), 7.88 (d, J = 2.0 Hz, 1H), 3.94 (s, 3H), 3.47 - 3.40 (m, 1H), 2.35 (s, 3H), 1.88 - 1.82 (m, 4H), 1.74 - 1.64 (m, 3H), 1.48 - 1.38 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.8, 163.7, 152.2, 138.3, 129.8, 124.7, 52.3, 42.3, 32.5, 26.7, 26.1, 17.8; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{14}H_{20}NO_2$: 234.1494. Found: 234.1490.

Benzyl 5-methyl-2-phenylnicotinate (2l):



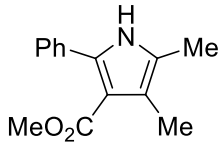
The title compound was prepared according to the general procedure. The product was obtained as yellow oil. Yield: 78%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.59 (d, J = 2.0 Hz, 1H),

7.91 (d, J = 1.6 Hz, 1H), 7.50 - 7.48 (m, 2H), 7.39 - 7.36 (m, 3H), 7.28 - 7.25 (m, 3H), 7.03 - 7.00 (m, 2H), 5.12 (s, 2H), 2.41 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.3, 156.2, 151.8, 140.1, 138.2, 134.9, 131.4, 128.5, 128.4, 128.3, 128.3, 128.3, 126.5, 67.4, 17.9; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{20}H_{18}NO_2$: 304.1338. Found: 304.1334.

General procedure for pyrroles:

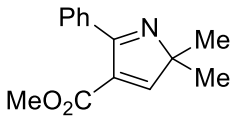
A solution of diazo compound (0.2 mmol) and $NiCl_2(PPh_3)_2$ (0.02 mmol, 10 mol%) in chlorobenzene (2.0 mL) was stirred at 130 ℃ until the starting material was fully consumed. The reaction mixture was concentrated under reduced pressure to give the crude material which was purified by column chromatography using hexane - ethyl acetate (9:1) to give the corresponding product.

Methyl 4,5-dimethyl-2-phenyl-1*H*-pyrrole-3-carboxylate (3a):



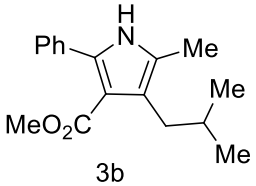
The title compound was prepared according to the general procedure. The product was obtained as white solid. Mp: 168 - 170 ℃. Yield 82%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (s, 1H), 7.48 - 7.45 (m, 2H), 7.40 - 7.30 (m, 3H), 3.69 (s, 3H), 2.21 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.3, 135.3, 133.1, 128.8, 128.1, 127.7, 124.6, 117.6, 111.3, 50.5, 10.9, 10.8; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{14}H_{16}NO_2$: 230.1181. Found: 230.1178.

Methyl 2,2-dimethyl-5-phenyl-2H-pyrrole-4-carboxylate (3a'):

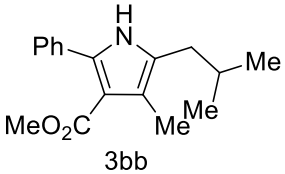


The title compound was prepared according to the general procedure. The product was obtained as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (s, 1H), 7.68 - 7.66 (m, 2H), 7.42 - 7.40 (m, 3H), 3.78 (s, 3H), 1.46 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.4, 168.2, 164.0, 134.4, 131.3, 129.7, 128.5, 127.9, 127.8, 76.7, 51.9, 22.8; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{14}H_{16}NO_2$: 230.1181. Found: 230.1176.

Methyl 4-isobutyl-5-methyl-2-phenyl-1*H*-pyrrole-3-carboxylate (3b) & Methyl 5-isobutyl-4-methyl-2-phenyl-1H-pyrrole-3-carboxylate (3bb):



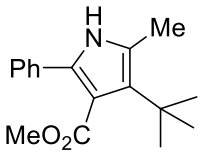
3b



3bb

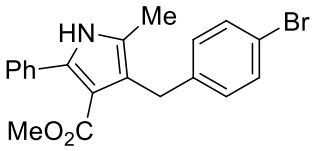
The title compound was prepared according to the general procedure. The product was obtained as yellow oil in a ratio of **3b**:**3bb** = 1.7:1. Yield 82%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 & 7.91 (s & s, 1H), 7.48 - 7.44 (m, 2H), 7.40 - 7.29 (m, 3H), 3.68 & 3.66 (s & s, 3H), 2.52 & 2.43 (d & d, *J* = 6.8 Hz & 7.2 Hz, 2H), 2.21 & 2.20 (s & s, 1H), 1.86 - 1.83 (m, 1H), 0.93 & 0.91 (d & d, *J* = 6.8 Hz & 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.5, 166.4, 135.3, 135.3, 133.2, 133.2, 128.8, 128.7, 128.1, 128.0, 127.7, 127.6, 125.3, 121.8, 118.1, 111.2, 111.0, 50.5, 50.5, 34.7, 34.3, 30.3, 29.6, 22.6, 22.4, 11.2, 11.1; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{17}H_{22}NO_2$: 272.1651. Found: 272.1653.

Methyl 4-tert-butyl-5-methyl-2-phenyl-1*H*-pyrrole-3-carboxylate (3c):



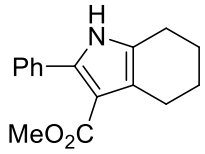
The title compound was prepared according to the general procedure. The product was obtained as colorless oil. Yield 67%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (s, 1H), 7.35 - 7.33 (m, 4H), 7.26 - 7.25 (m, 1H), 3.69 (s, 3H), 2.39 (s, 3H), 1.39 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.1, 132.5, 129.7, 128.6, 127.7, 127.0, 126.7, 123.3, 114.2, 51.7, 32.6, 31.6, 15.4 ; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{17}H_{22}NO_2$: 272.1651. Found: 272.1656.

Methyl 4-(4-bromobenzyl)-5-methyl-2-phenyl-1*H*-pyrrole-3-carboxylate (3d):



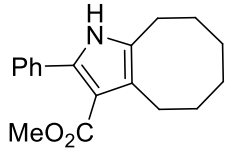
The title compound was prepared according to the general procedure. The product was obtained as yellow oil. Yield 74%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (s, 1H), 7.48 - 7.45 (m, 2H), 7.40 - 7.33 (m, 5H), 7.09 - 7.07 (m, 2H), 4.03 (s, 2H), 3.58 (s, 3H), 2.21 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.8, 141.3, 135.8, 132.9, 131.1, 130.0, 128.8, 128.1, 127.9, 125.8, 120.0, 119.1, 110.9, 50.5, 30.4, 11.1; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{20}H_{19}NO_2Br$: 384.0599. Found: 384.0594.

Methyl 5-phenyl-2-(phenylethynyl)-1H-pyrrole-3-carboxylate (3e):



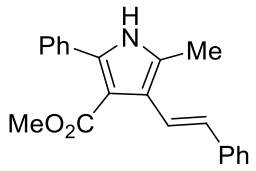
The title compound was prepared according to the general procedure. The product was obtained as white solid. Mp: 126 - 127 ℃. Yield 70%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (s, 1H), 7.52 - 7.50 (m, 2H), 7.40 - 7.36 (m, 2H), 7.34 - 7.31 (m, 1H), 3.70 (s, 3H), 2.76 (t, *J* = 6.0 Hz, 2H), 2.58 (t, *J* = 6.0 Hz, 2H), 1.84 - 1.78 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.1, 135.8, 133.0, 128.9, 128.1, 127.8, 127.8, 120.2, 109.9, 50.5, 23.4, 23.4, 22.9, 22.6; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{19}H_{18}NO_2$: 292.1338. Found: 292.1343.

Methyl 2-phenyl-4,5,6,7,8,9-hexahydro-1*H*-cycloocta[b]pyrrole-3-carboxylate (3f):



The title compound was prepared according to general procedure. The product was obtained as white solid. Mp: 129 - 130 ℃. Yield 58%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (s, 1H), 7.50 - 7.47 (m, 2H), 7.40 - 7.36 (m, 2H), 7.33 - 7.31 (m, 1H), 3.69 (s, 3H), 2.86 (t, *J* = 6.0 Hz, 2H), 2.68 (t, *J* = 6.0 Hz, 2H), 1.72 - 1.70 (m, 2H), 1.69 - 1.61 (m, 2H), 1.53 - 1.48 (m, 2H), 1.42 - 1.38 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.3, 134.7, 133.2, 130.3, 128.7, 128.1, 127.6, 121.9, 110.6, 50.5, 30.7, 29.8, 26.0, 25.9, 25.8, 23.0; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{18}H_{22}NO_2$: 284.1651. Found: 284.1651.

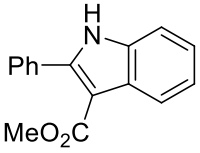
(*E*)-Methyl 5-methyl-2-phenyl-4-styryl-1*H*-pyrrole-3-carboxylate (3g):



The title compound was prepared according to the general procedure. The product was obtained as white solid. Mp: 174 - 175 °C. Yield 84%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 (s, 1H), 7.56 (d, *J* = 16.4 Hz, 1H), 7.51 - 7.47 (m, 4H), 7.42 - 7.40 (m, 2H), 7.38 - 7.32 (m, 3H), 7.24 - 7.20 (m, 1H), 6.65 (d, *J* = 16.8 Hz, 1H), 3.71 (s, 3H), 2.46 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.1, 138.5, 135.4, 132.6, 129.1, 128.8, 128.6, 128.2, 128.1, 126.8, 126.1,

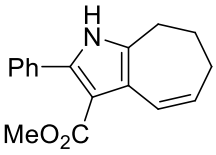
122.9, 120.1, 110.8, 50.9, 13.2; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{21}H_{20}NO_2$: 318.1494. Found: 318.1497.

Methyl 2-phenyl-1*H*-indole-3-carboxylate (3h):



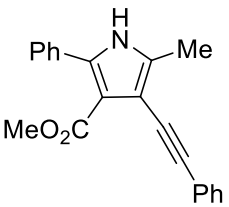
The title compound was prepared according to the general procedure. The product was obtained as white solid. Mp: 150 - 151 ℃. Yield 44%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.46 (s, 1H), 8.23 - 8.21 (m, 1H), 7.69 - 7.66 (m, 2H), 7.48 - 7.46 (m, 3H), 7.42 - 7.39 (m, 1H), 7.30 - 7.28 (m, 2H), 3.85 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.7, 144.5, 135.1, 132.0, 129.5, 129.3, 128.2, 127.6, 123.3, 122.2, 122.2, 110.9, 104.6, 50.9; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{16}H_{14}NO_2$: 252.1025. Found: 252.1025.

Methyl 2-phenyl-1,6,7,8-tetrahydrocyclohepta[b]pyrrole-3-carboxylate (3i):



The title compound was prepared according to the general procedure (reaction at 60 °C). The product was obtained as yellow solid. Mp: 134 - 135 ℃. Yield 78%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (s, 1H), 7.42 - 7.39 (m, 2H), 7.37 - 7.35 (m, 3H), 6.93 (d, *J* = 12.0 Hz, 1H), 5.74 - 5.69 (m, 1H), 3.65 (s, 3H), 2.87 (t, *J* = 6.0 Hz, 2H), 2.47 - 2.43 (m, 2H), 2.00 - 1.94 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.2, 135.2, 132.8, 131.6, 128.7, 128.1, 127.8, 127.4, 121.5, 119.4, 110.5, 50.8, 30.9, 29.1, 23.3; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{17}H_{18}NO_2$: 268.1338. Found: 268.1333.

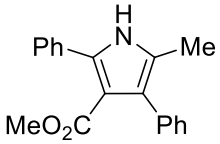
Methyl 5-methyl-2-phenyl-4-(phenylethynyl)-1*H*-pyrrole-3-carboxylate (3j):



The title compound was prepared according to the general procedure. The product was obtained as yellow solid. Mp: 180 - 181 ^{o}C. Yield 92%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.23 (s,

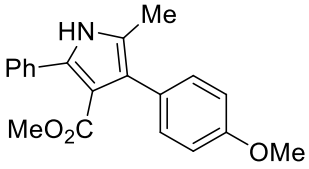
1H), 7.54 - 7.51 (m, 4H), 7.43 - 7.27 (m, 6H), 3.79 (s, 3H), 2.43 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.8, 135.8, 133.6, 131.8, 131.3, 128.8, 128.4, 128.3, 128.2, 127.5, 124.4, 112.5, 104.7, 92.5, 83.7, 51.1, 12.0; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{21}H_{18}NO_2$: 316.1338. Found: 316.1333.

Methyl 5-methyl-2,4-diphenyl-1*H*-pyrrole-3-carboxylate (3k):



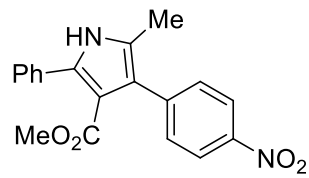
The title compound was prepared according to the general procedure. The product was obtained as white solid. Mp: 206 - 208 ℃. Yield 80%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16 (s, 1H), 7.55 - 7.54 (m, 2H), 7.43 - 7.39 (m, 5H), 7.38 - 7.27 (m, 3H), 3.51 (s, 3H), 2.23 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.0, 135.6, 134.9, 132.5, 130.1, 128.6, 128.3, 127.9, 127.7, 126.2, 125.8, 123.7, 111.5, 50.7, 11.5; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{19}H_{18}NO_2$: 292.1338. Found: 292.1336.

Methyl 4-(4-methoxyphenyl)-5-methyl-2-phenyl-1*H*-pyrrole-3-carboxylate (3l):



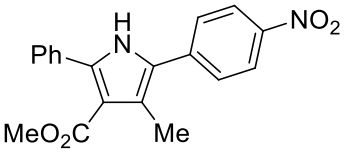
The title compound was prepared according to general procedure. The product was obtained as yellow solid. Mp: 186 - 187 ℃. Yield 78%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 (s, 1H), 8.54 - 7.52 (m, 2H), 7.40 - 7.39 (m, 2H), 7.26 - 7.26 (m, 1H), 7.25 - 7.23(m, 2H), 6.93 (d, *J* = 8.8 Hz, 2H), 3.84 (s, 3H), 3.53 (s, 3H), 2.21 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.1, 158.1, 134.8, 132.7, 131.1, 129.6, 128.5, 128.2, 127.9, 125.6, 123.3, 114.7, 113.1, 111.5, 55.2, 50.7, 11.5; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{19}H_{17}N_2O_4$ 337.1188. Found: 337.1194.

Methyl 5-methyl-4-(4-nitrophenyl)-2-phenyl-1*H*-pyrrole-3-carboxylate (3m):



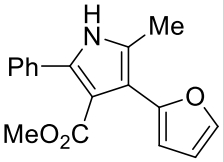
The title compound was prepared according to general procedure. The product was obtained as yellow solid. Mp: 220 - 221 ℃. Yield 57%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 (s, 1H), 8.24 (d, *J* = 8.8 Hz, 2H), 7.55 - 7.53 (m, 2H), 7.48 - 7.36 (m, 5H), 3.52 (s, 3H), 2.24 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.5, 146.2, 143.1, 136.1, 132.0, 130.8, 128.8, 128.4, 128.3, 126.7, 123.0, 121.8, 50.9, 11.5; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{19}H_{17}N_2O_4$ 337.1188. Found: 337.1194.

Methyl 4-methyl-5-(4-nitrophenyl)-2-phenyl-1H-pyrrole-3-carboxylate (3mm):



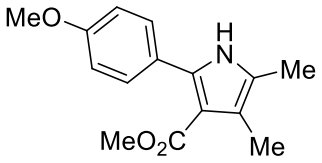
The title compound was prepared according to General procedure. The product was obtained as yellow solid. Mp: 199 - 201 ℃. Yield 32%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.45 (s, 1H), 8.29 (d, *J* = 8.8 Hz, 2H), 7.60 - 7.58 (m, 2H), 7.52 - 7.51 (m, 2H), 7.43 - 7.41 (m, 3H), 3.72 (s, 3H), 2.49 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.8, 146.0, 138.9, 132.1, 128.9, 128.7, 128.3, 127.4, 127.1, 124.3, 122.2, 113.9, 50.9, 12.1; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{19}H_{17}N_2O_4$ 337.1190. Found: 337.1194.

Methyl 4-(furan-2-yl)-5-methyl-2-phenyl-1*H*-pyrrole-3-carboxylate (3n):



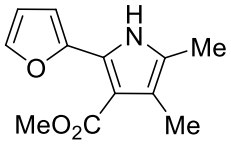
The title compound was prepared according to the general procedure. The product was obtained as yellow solid. Mp: 165 - 166 ^{o}C. Yield 72%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.34 (s, 1H), 7.47 - 7.44 (m, 3H), 7.38 - 7.31 (m, 3H), 6.44 (d, J_1 = 3.2 Hz, J_2 = 1.6 Hz 1H), 6.38 (d, *J* = 3.2 Hz, 1H), 3.60 (s, 3H), 2.30 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.9, 149.1, 141.2, 134.9, 132.2, 128.4, 128.3, 128.0, 127.9, 113.1, 111.2, 110.7, 107.6, 51.1, 12.1; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{17}H_{16}NO_3$: 282.1130. Found: 282.1133.

Methyl 2-(4-methoxyphenyl)-4,5-dimethyl-1H-pyrrole-3-carboxylate (3o):



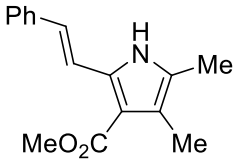
The title compound was prepared according to the general procedure. The product was obtained as white solid. Mp: 159 - 160 ^{o}C. Yield 81%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (s, 1H), 7.40 (d, *J* = 8.4 Hz, 2H), 6.91 (d, *J* = 8.8 Hz, 2H), 3.83 (s, 3H), 3.69 (s, 3H), 2.21 (s, 3H), 2.20 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.4, 159.3, 135.4, 130.1, 125.6, 124.1, 117.3, 113.5, 110.8, 55.3, 50.5, 11.0, 10.8; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{15}H_{18}NO_3$: 260.1287. Found: 260.1286.

Methyl 2-(furan-2-yl)-4,5-dimethyl-1*H*-pyrrole-3-carboxylate (3p):



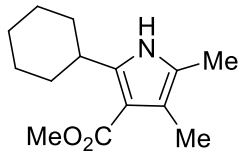
The title compound was prepared according to the general procedure. The product was obtained as green oil. Yield 68%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.53 (s, 1H), 7.37 (d, *J* = 1.2 Hz, 1H), 7.23 (d, *J* = 3.6 Hz, 1H), 6.48 (dd, J_1 = 3.2 Hz, J_2 = 1.6 Hz, 1H), 3.85 (s, 3H), 2.21 (s, 3H), 2.18 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.9, 146.6, 140.8, 125.8, 124.7, 117.6, 112.1, 110.3, 109.1, 50.7, 11.2, 10.9; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{12}H_{14}NO_3$: 220.0974. Found: 220.0973.

(*E*)-Methyl 4,5-dimethyl-2-styryl-1*H*-pyrrole-3-carboxylate (3q):



The title compound was prepared according to the general procedure. The product was obtained as white solid. Mp: 150 - 151 ^{o}C. Yield 84%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.29 (s, 1H), 7.80 (d, *J* = 16.8 Hz, 1H), 7.48 - 7.46 (m, 2H), 7.35 - 7.31 (m, 2H), 7.25 - 7.21 (m, 1H), 6.68 (d, *J* = 16.8 Hz, 1H), 3.86 (s, 3H), 2.21 (s, 3H), 2.18 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.4, 137.2, 133.0, 128.7, 127.5, 126.3, 125.8, 125.5, 118.5, 118.0, 113.1, 50.8, 11.0; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{16}H_{18}NO_2$: 256.1338. Found: 256.1335.

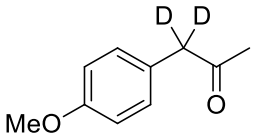
Methyl 2-cyclohexyl-4,5-dimethyl-1*H*-pyrrole-3-carboxylate (3r):



The title compound was prepared according to the general procedure. The product was obtained as white solid. Mp: 127 - 129 ^{o}C. Yield 77%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (s, *J* = 16.0 Hz, 1H), 3.79 (s, 3H), 3.44 - 3.36 (m, 1H), 2.13 (s, 6H), 1.98 - 1.95 (m, 2H), 1.83 - 1.73 (m, 3H), 1.48 - 1.41 (m, 2H), 1.40 - 1.19 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.7, 143.2, 121.9, 115.8, 109.2, 50.3, 36.0, 32.9, 26.6, 26.2, 11.1, 10.7; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{14}H_{22}NO_2$: 236.1651. Found: 236.1646.

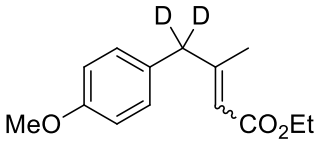
General procedure for deuterium labeling experiment:

1-(4-methoxyphenyl)-(1,1-(2)D)propan-2-one:



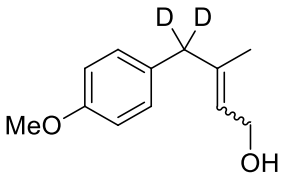
The title compound was prepared according to reported method[1]. The product was obtained as yellow oil. Yield: 95%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.12 - 7.09 (m, 2H), 6.88 - 6.85 (m, 2H), 3.78 (s, 3H), 2.12 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 206.8, 158.6, 130.2, 126.1, 114.1, 55.1, 50.1, 28.9; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{10}H_{11}D_2O_2$: 167.1041. Found: 167.1039.

Ethyl 4-(4-methoxyphenyl)-(4,4-(2)D)-3-methylbut-2-enoate:



The title compound was prepared according to reported method[2]. The product was obtained as colorless oil in a *cis* : *trans* ratio of 24 : 76. Yield: 92%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.17 - 7.15 & 7.09 - 7.07 (m & m, 2H), 6.85 - 6.81 (m, 2H), 5.74 & 5.66 (d & d, J = 0.4 & 0.8 Hz, 1H), 4.22 - 4.12 (m, 2H), 3.79 & 3.78 (s & s, 3H), 2.11 & 1.78 (s & d, J = 0.4 Hz, 3H), 1.32 - 1.25 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.8, 166.5, 158.7, 158.4, 130.1, 129.9, 129.7, 116.9, 116.8, 114.0, 113.8, 59.7, 59.6, 55.2, 55.2, 24.4, 18.5, 14.3; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{14}H_{17}D_2O_3$: 237.1460. Found: 237.1457.

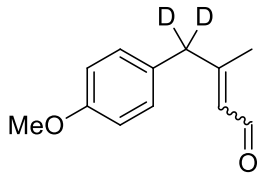
4-(4-methoxyphenyl)-(4,4-(2)D)-3-methylbut-2-en-1-ol:



The title compound was prepared according to reported method[2]. The product was obtained as colorless oil in a *cis* : *trans* ratio of 20 : 80. Yield: 98%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.08 - 7.04 (m, 2H), 6.81 - 6.79 (m, 2H), 5.54 - 5.51 & 5.49 - 5.45 (m & m, 1H), 4.22 & 4.13 (d & d, J = 7.2 & 6.8 Hz, 2H), 3.73 (s, 3H), 2.61 (s, 1H), 1.64 & 1.57 (s & s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.0, 157.9, 138.6, 138.3, 131.6, 131.5, 129.9, 129.5, 125.3, 125.2,

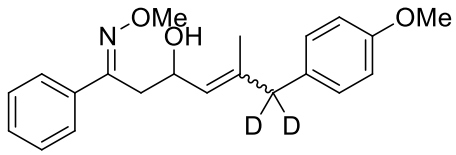
113.9, 113.8, 59.2, 59.0, 55.2, 23.3, 16.0; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{12}H_{15}D_2O_2$: 195.1354. Found: 195.1348.

4-(4-methoxyphenyl)-(4,4-(2)D)-3-methylbut-2-en-1-ol:



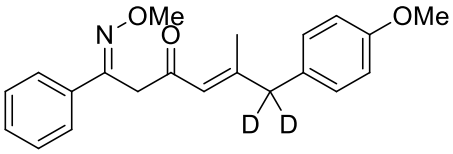
The title compound was prepared according to general IBX oxidation procedure. The product was obtained as colorless oil in a *cis* : *trans* ratio of 19 : 81. Yield: 82%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.11 & 9.99 (d & d, J = 8.0 & 8.0 Hz, 1H), 7.11 - 7.06 (m, 2H), 6.87 - 6.83 (m, 2H), 6.02 - 6.00 & 5.89 - 5.87 (m & m, 1H), 3.79 (s, 3H), 2.11 & 1.88 (d & d, J = 1.2 Hz & 0.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 191.4, 190.8, 162.8, 158.6, 158.6, 130.1, 129.6, 129.0, 128.9, 128.2, 114.2, 114.1, 114.0, 55.3, 17.2; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{12}H_{15}D_2O_2$: 195.1354. Found: 195.1348.

(1*E*)-3-hydroxy-6-(4-methoxyphenyl)-6,6-(2)D-5-methyl-1-phenylhex-4-en-1-one *O*-methyl oxime:



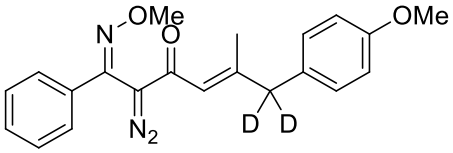
The title compound was prepared according to general procedure. The product was obtained as colorless oil in a *cis* : *trans* ratio of 20 : 80. Yield: 72%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 - 7.63 (m, 2H), 7.36 - 7.35 (m, 3H), 7.05 - 7.03 & 6.95 - 6.93 (m & m, 2H), 6.81 - 6.77 (m, 2H), 5.38 & 5.25 (d & d, J = 8.8 & 8.8 Hz, 1H), 4.90 - 4.82 & 4.80 - 4.76 (m & m, 1H), 4.00 & 3.99 (s & s, 3H), 3.78 (s, 3H), 3.16 - 3.10 (m, 1H), 2.96 - 2.91 (m, 1H), 2.09 & 2.02 (m & m, 1H), 1.60 & 1.51 (s & s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.0, 156.1, 155.9, 138.3, 136.0, 136.0, 131.3, 131.2, 129.9, 129.5, 129.2, 129.1, 128.8, 128.6, 128.5, 126.6, 126.6, 113.9, 113.7, 66.6, 66.6, 55.3, 35.6, 35.2, 23.2, 16.3, 16.3; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{21}H_{24}D_2NO_3$: 342.2038. Found: 342.2029.

(1*E*, 4*E*)-1-(methoxyimino)-6-(4-methoxyphenyl)-6,6-(2)D-5-methyl-1-phenylhex-4-en-3-one:



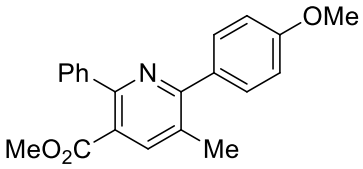
The title compound was prepared according to reported method. The product was obtained as colorless oil. Yield: 54%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 - 7.61 (m, 2H), 7.36 - 7.34 (m, 3H), 7.02 - 7.00 (m, 2H), 6.84 - 6.82 (m, 2H), 6.09 (d, J = 0.8 Hz, 1H), 3.96 (s, 3H), 3.85 (s, 2H), 3.79 (s, 3H), 2.06 (d, J = 0.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.9, 159.0, 158.4, 152.2, 135.6, 130.1, 129.5, 129.2, 128.5, 126.2, 123.2, 113.9, 62.1, 55.2, 43.1, 19.3; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{21}H_{22}D_2NO_3$: 340.1882. Found: 340.1886.

(1*Z*,4*E*)-2-diazo-1-(methoxyimino)-6-(4-methoxyphenyl)-6,6-(2)D-5-methyl-1-phenylhex-4-en-3-one (1m-D_2):



The title compound was prepared according to reported method. The product was obtained as yellow oil. Yield: 79%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 - 7.55 (m, 2H), 7.47 - 7.37 (m, 3H), 6.73 - 6.70 (m, 2H), 6.66 - 6.64 (m, 2H), 5.49 (s, 1H), 4.06 (s, 3H), 3.79 (s, 3H), 2.03 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 184.0, 158.3, 156.3, 144.3, 134.3, 130.0, 129.4, 128.7, 127.8, 113.9, 62.8, 46.1, 45.5, 19.2; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{21}H_{20}D_2N_3O_3$: 366.1787. Found: 366.1781.

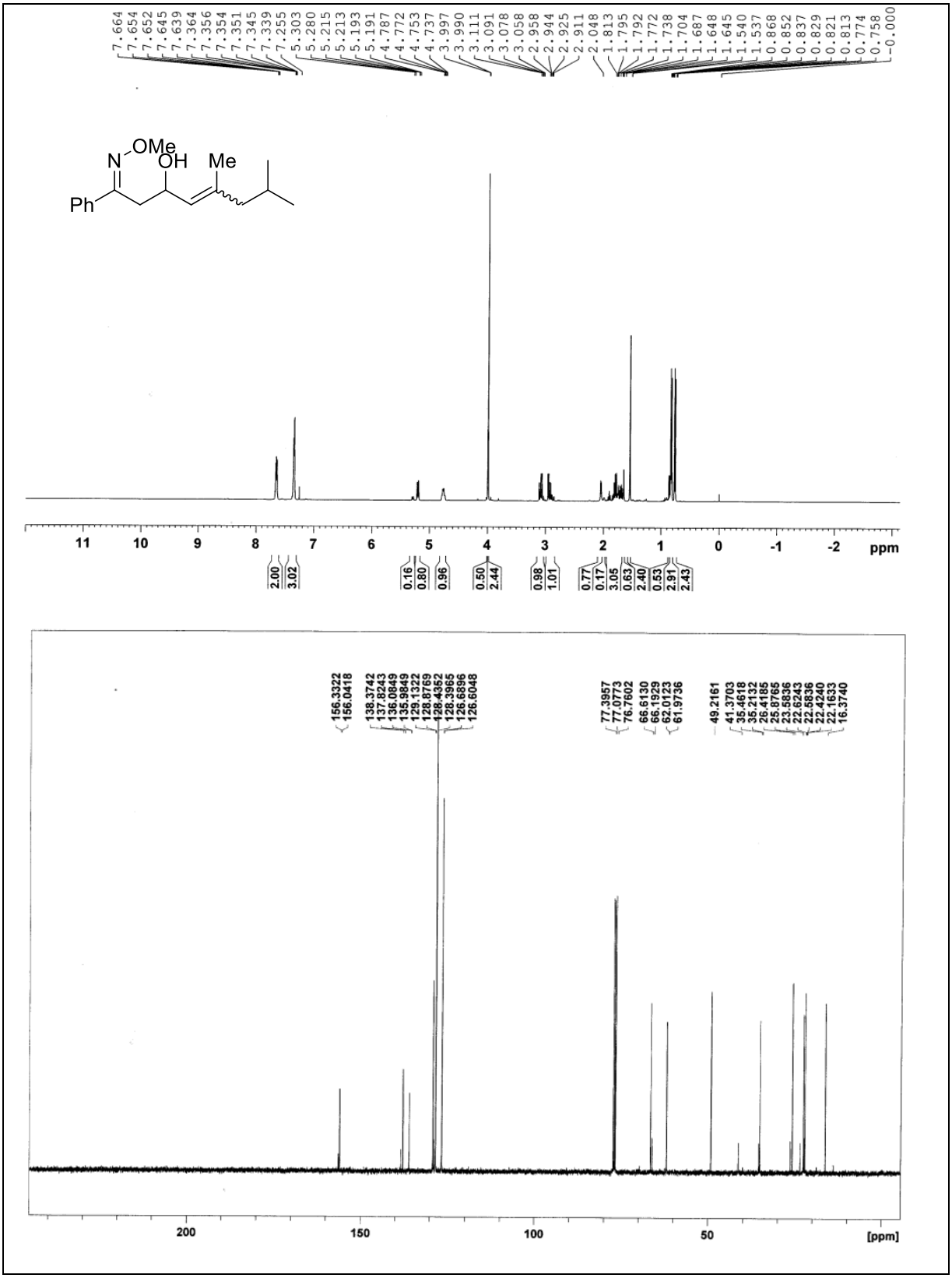
Methyl 6-(4-methoxyphenyl)-5-methyl-2-phenylnicotinate:



The title compound was prepared according to reported method. The product was obtained as colorless oil. Yield: 75%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 (s, 1H), 7.61 - 7.57 (m, 4H), 7.43 - 7.38 (m, 3H), 7.00 - 6.97 (m, 2H), 3.86 (s, 3H), 3.71 (s, 3H), 2.47 (s, 3H) ; ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.8, 159.9, 159.7, 155.9, 140.5, 140.2, 132.4, 130.7, 128.8, 128.6, 128.3, 128.0, 124.4, 113.6, 55.4, 52.2, 19.9; HRMS (ESI) m/z $[M+H]^+$: Calcd for $C_{21}H_{20}NO_3$: 334.1443. Found: 334.1447.

(1). Winnik, M. A. *Synth. Commun.* **1973**, *3*, 299.
(2). M. Cavero, W. B. Motherwell, P. Potier , J-M. Weibel. *Chem. Commun.* **2002**, 2394

^{1}H and ^{13}C NMR spectra of β-hydroxy oxime ethers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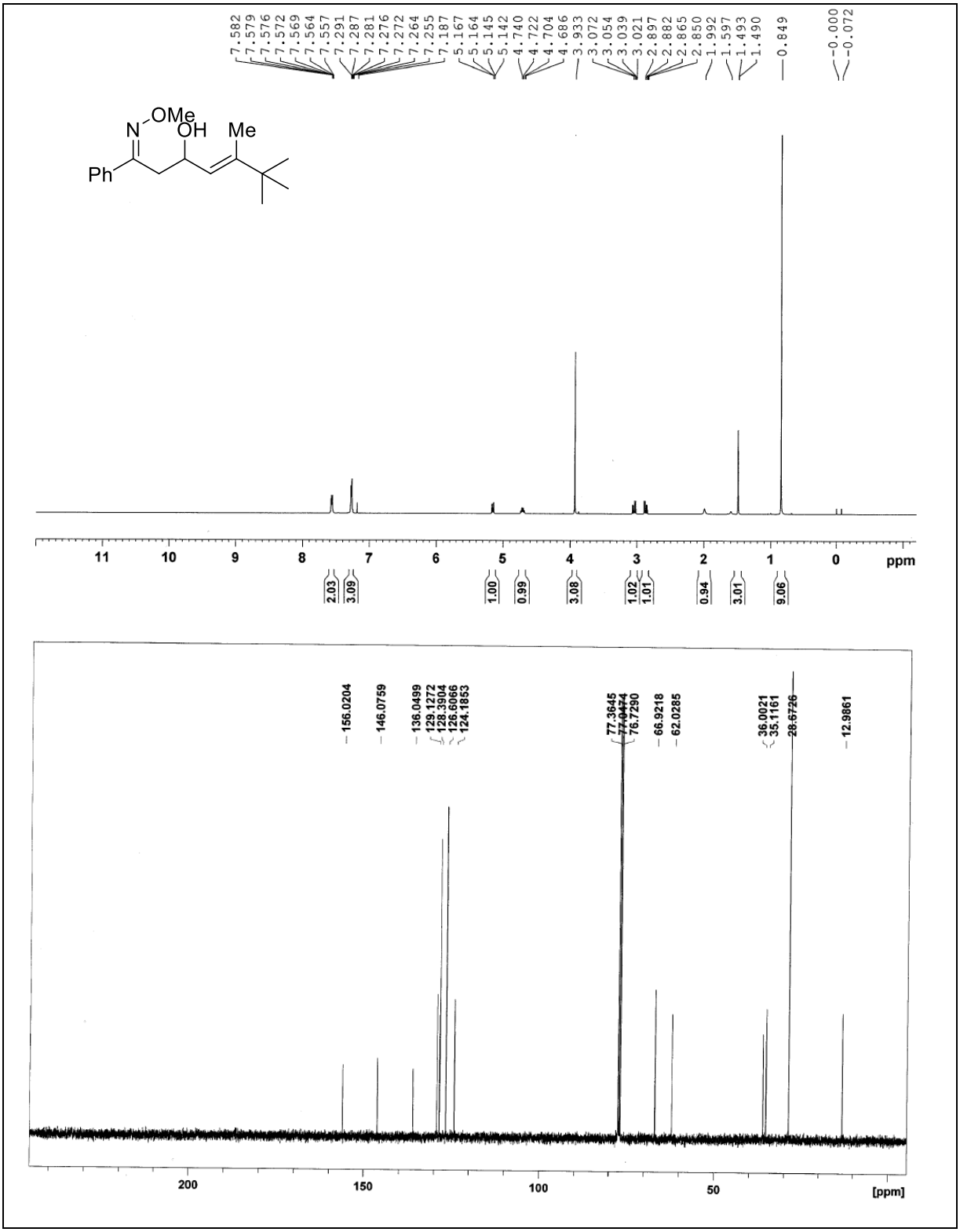
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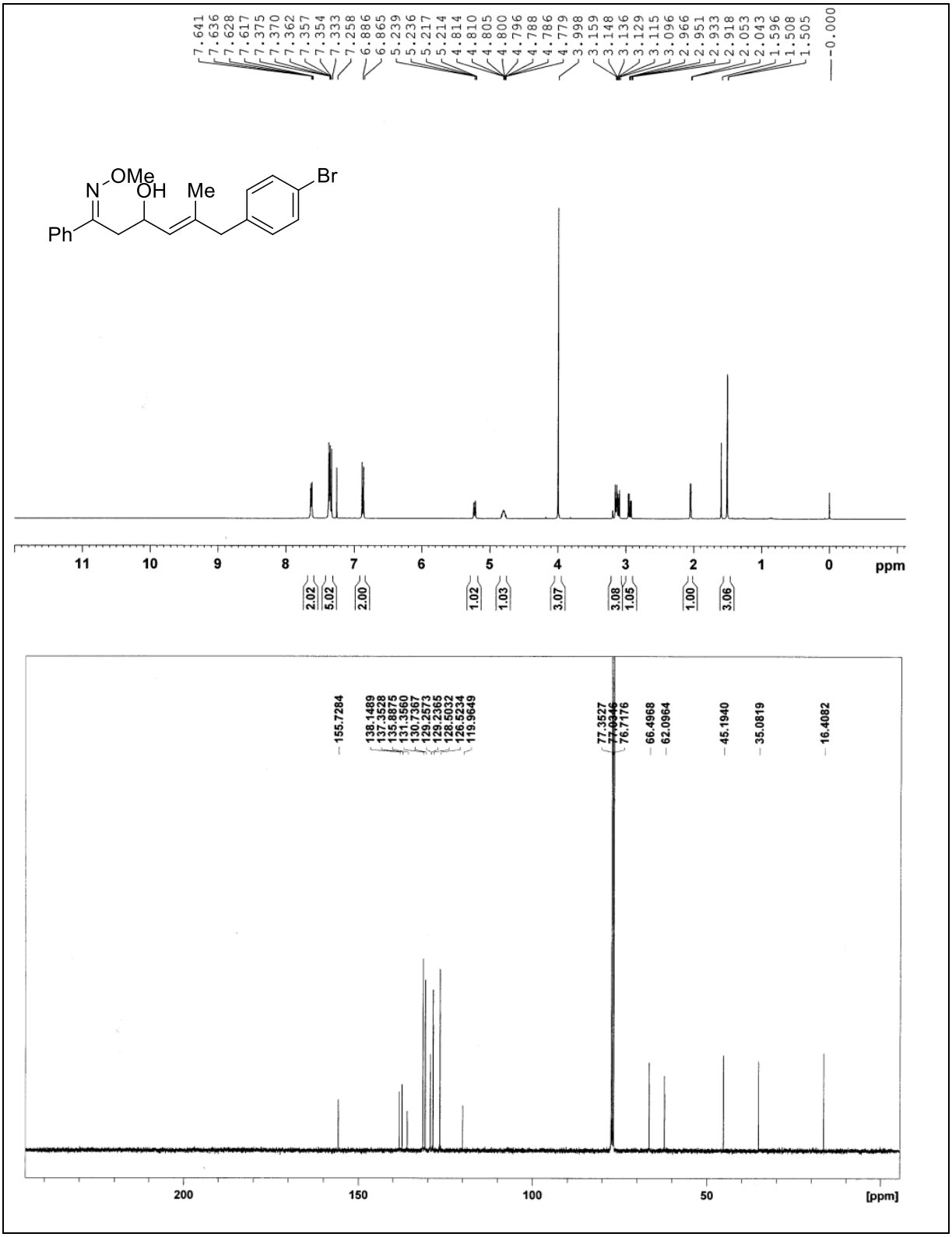
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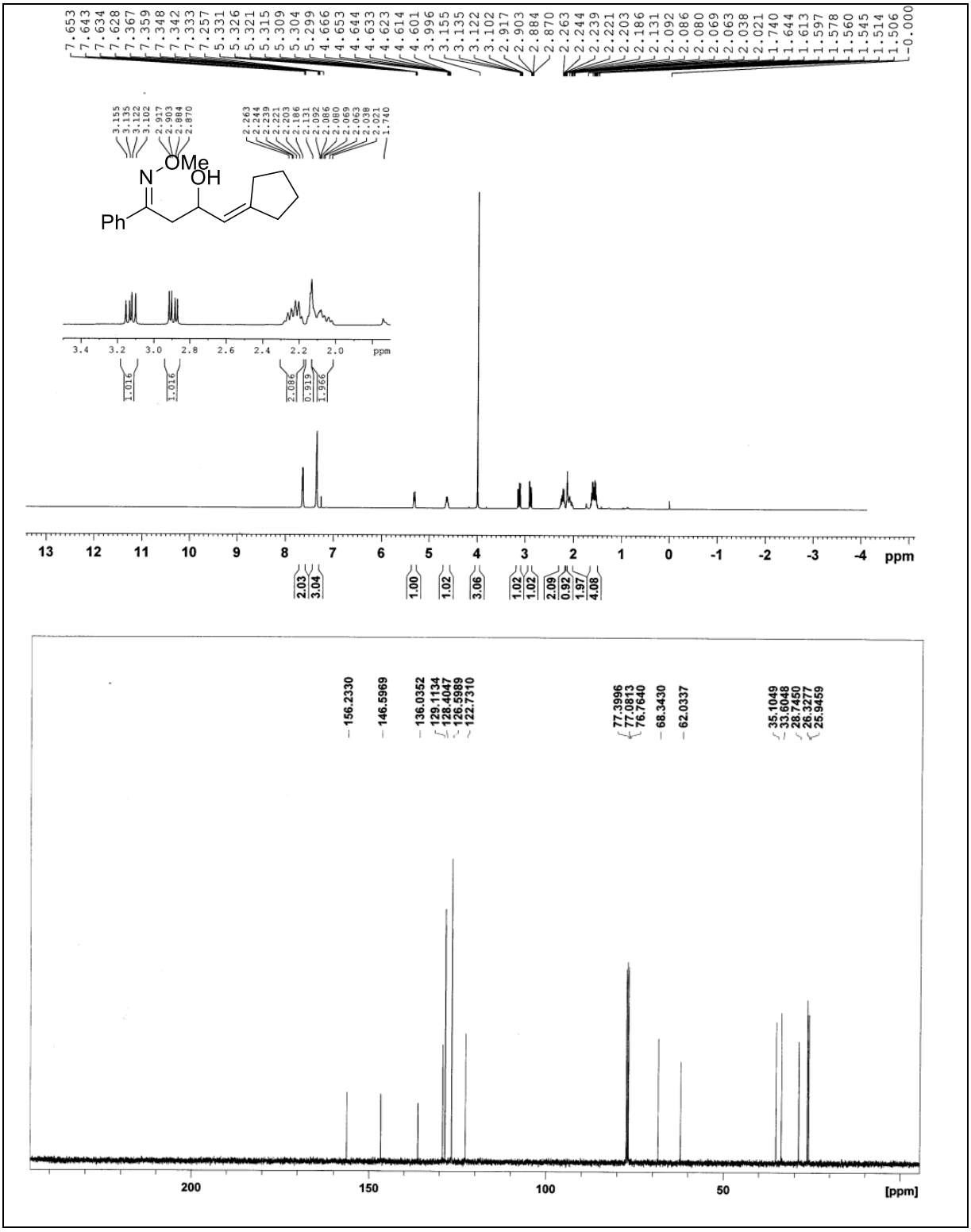
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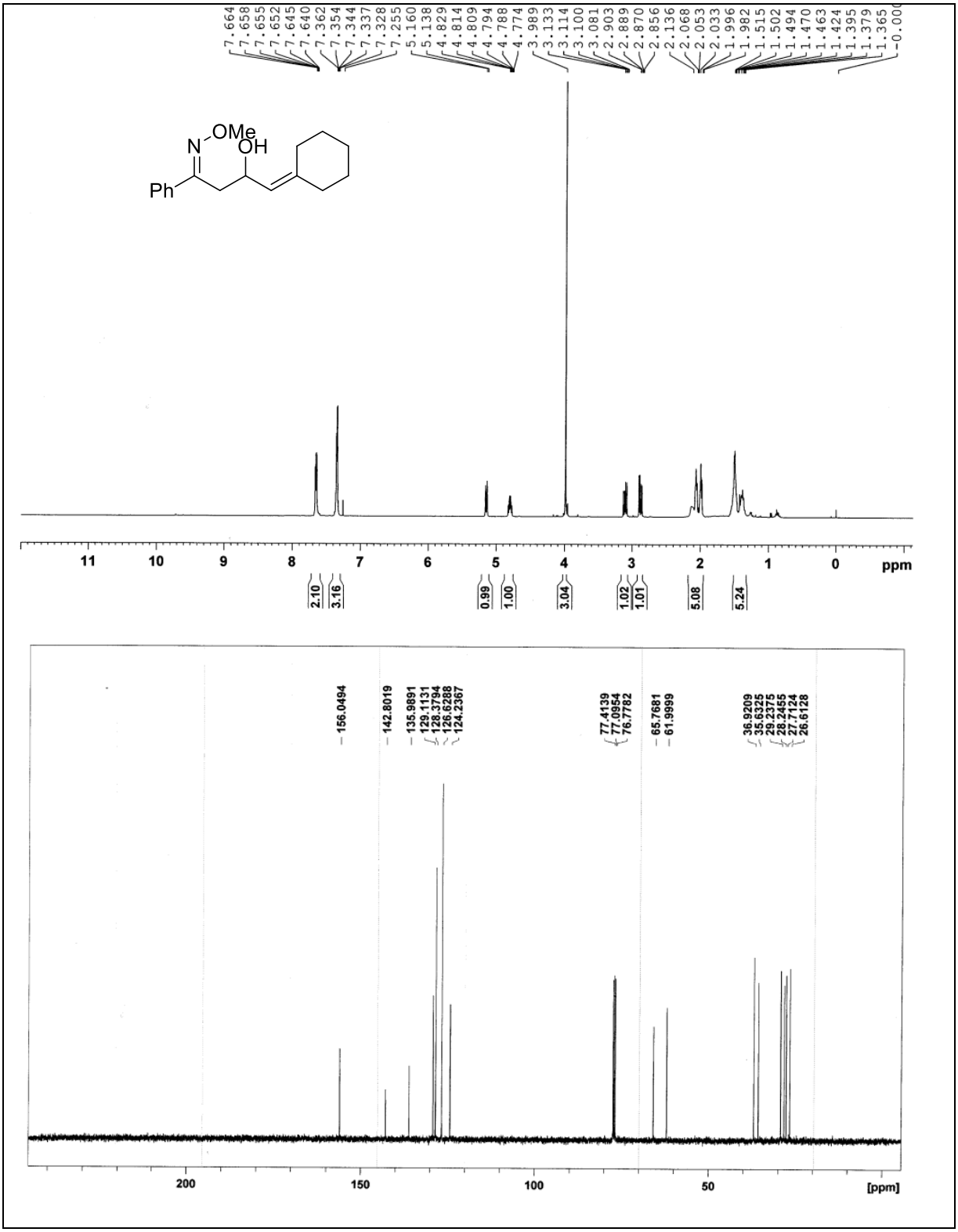
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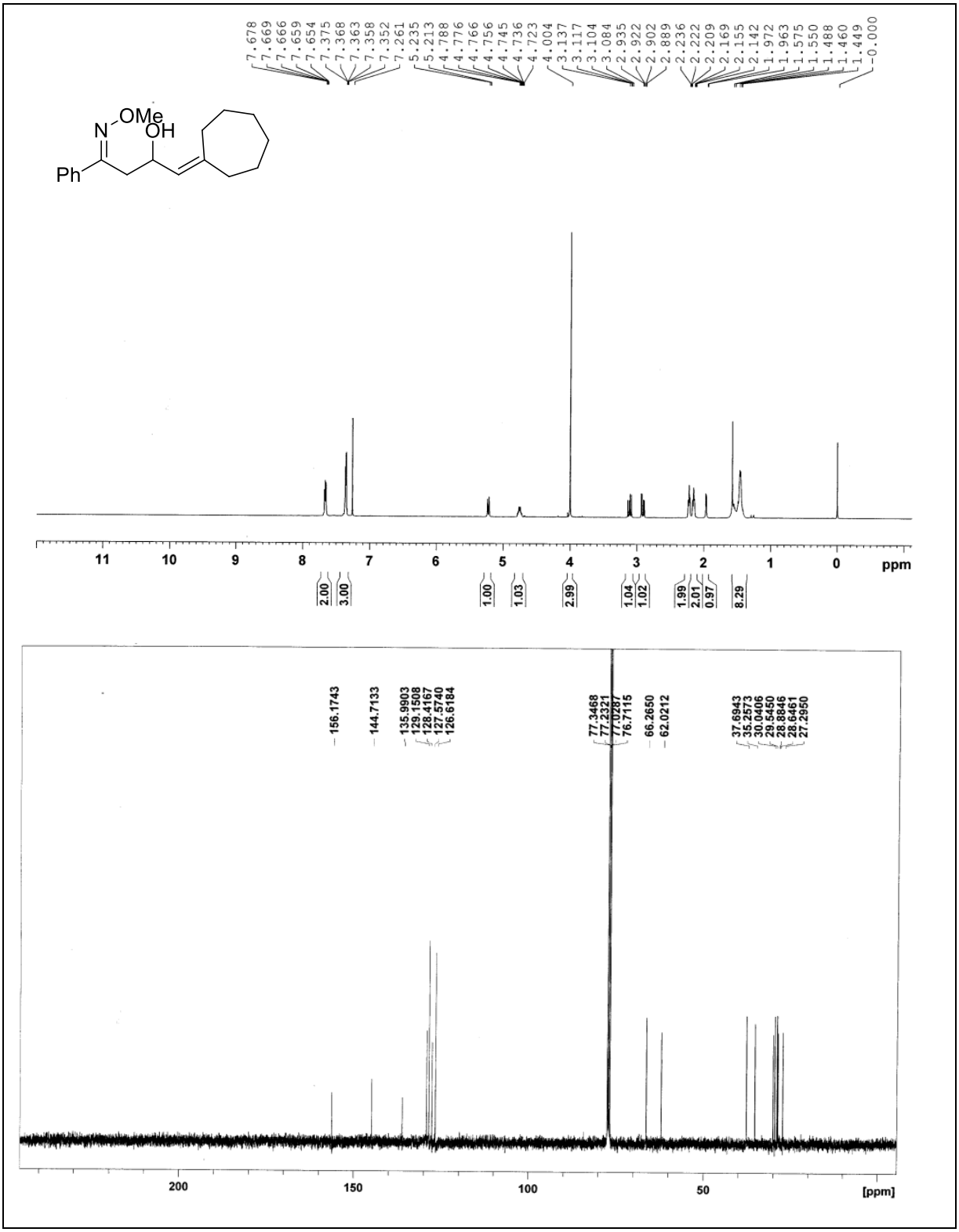
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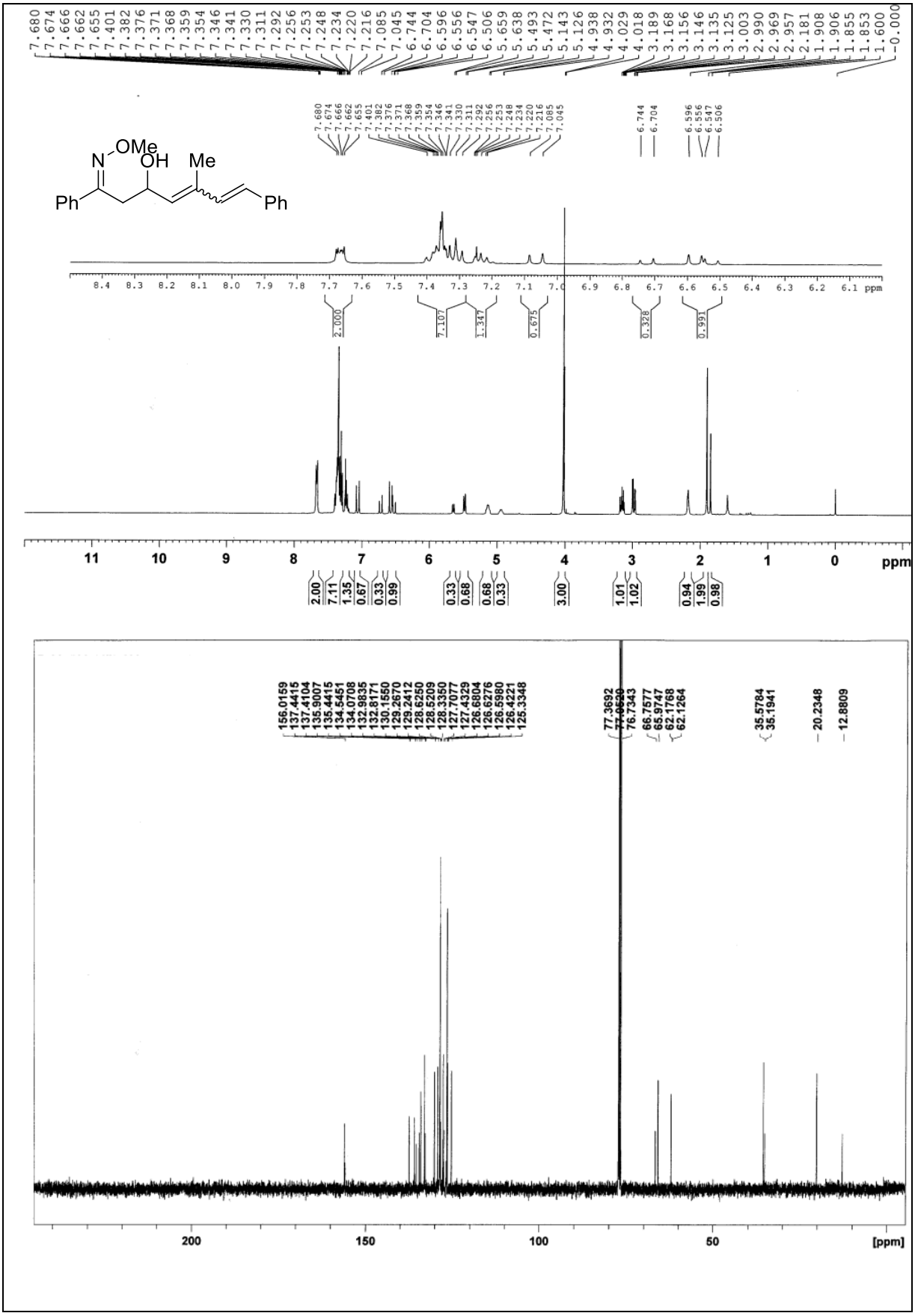
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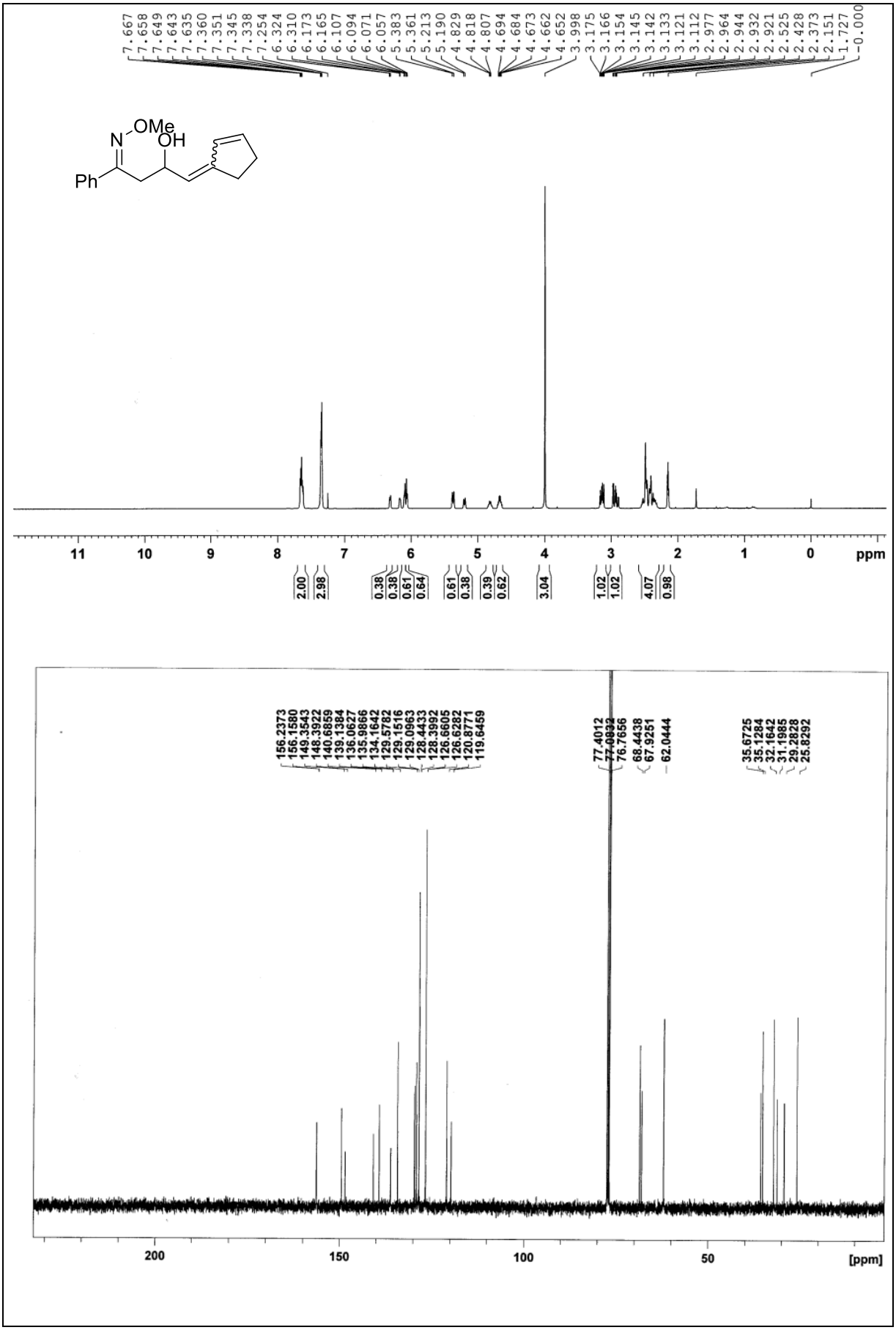
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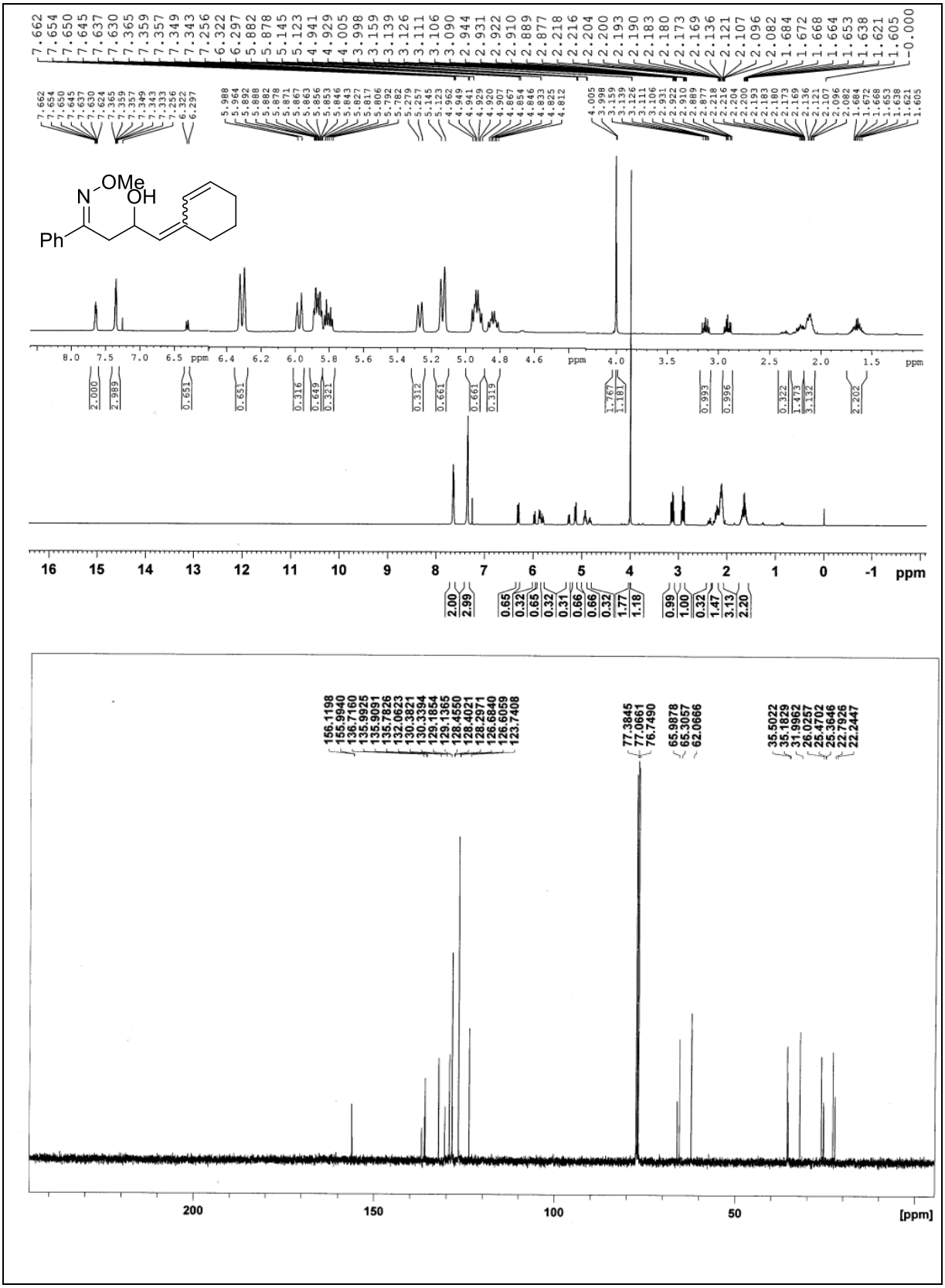
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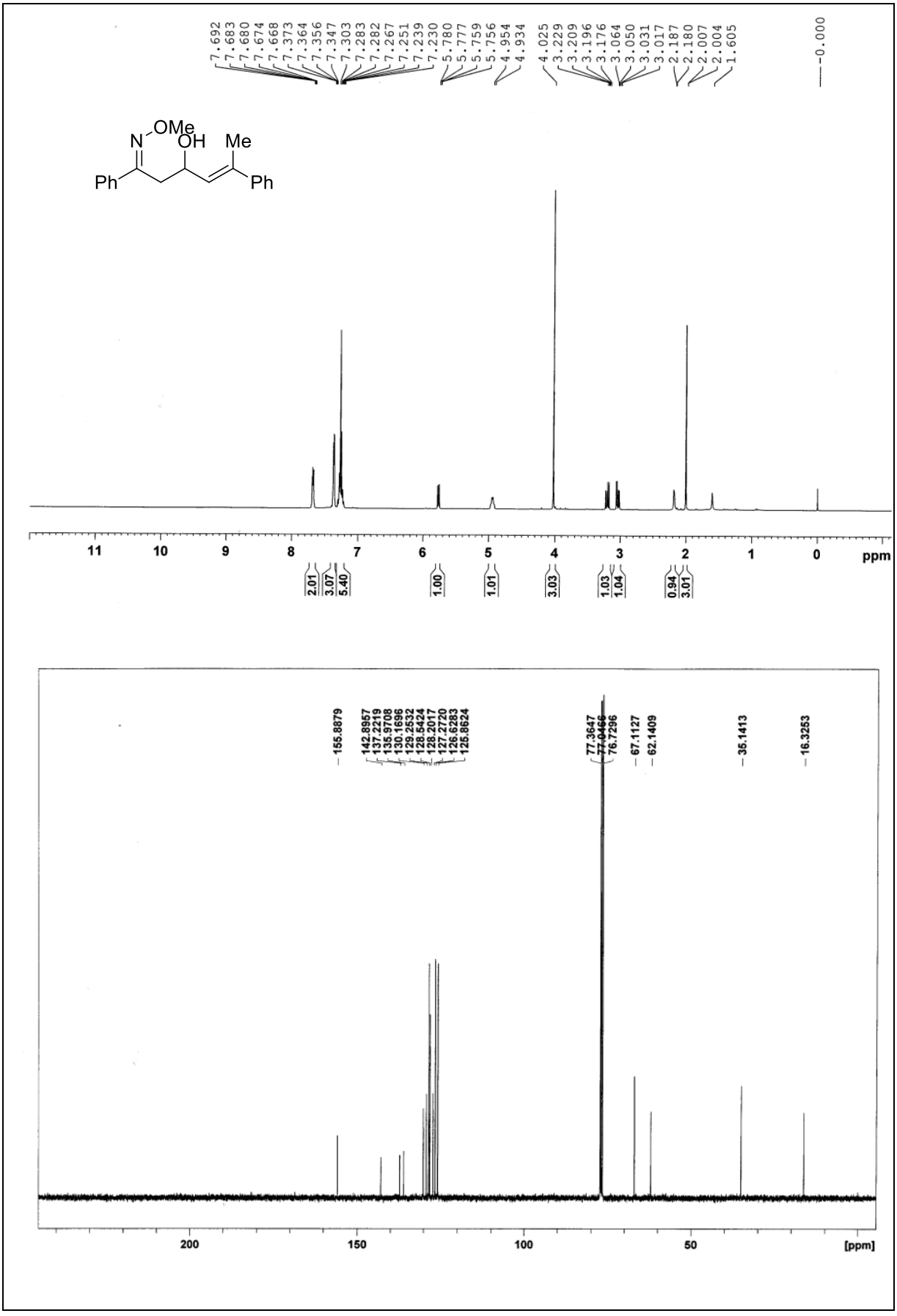
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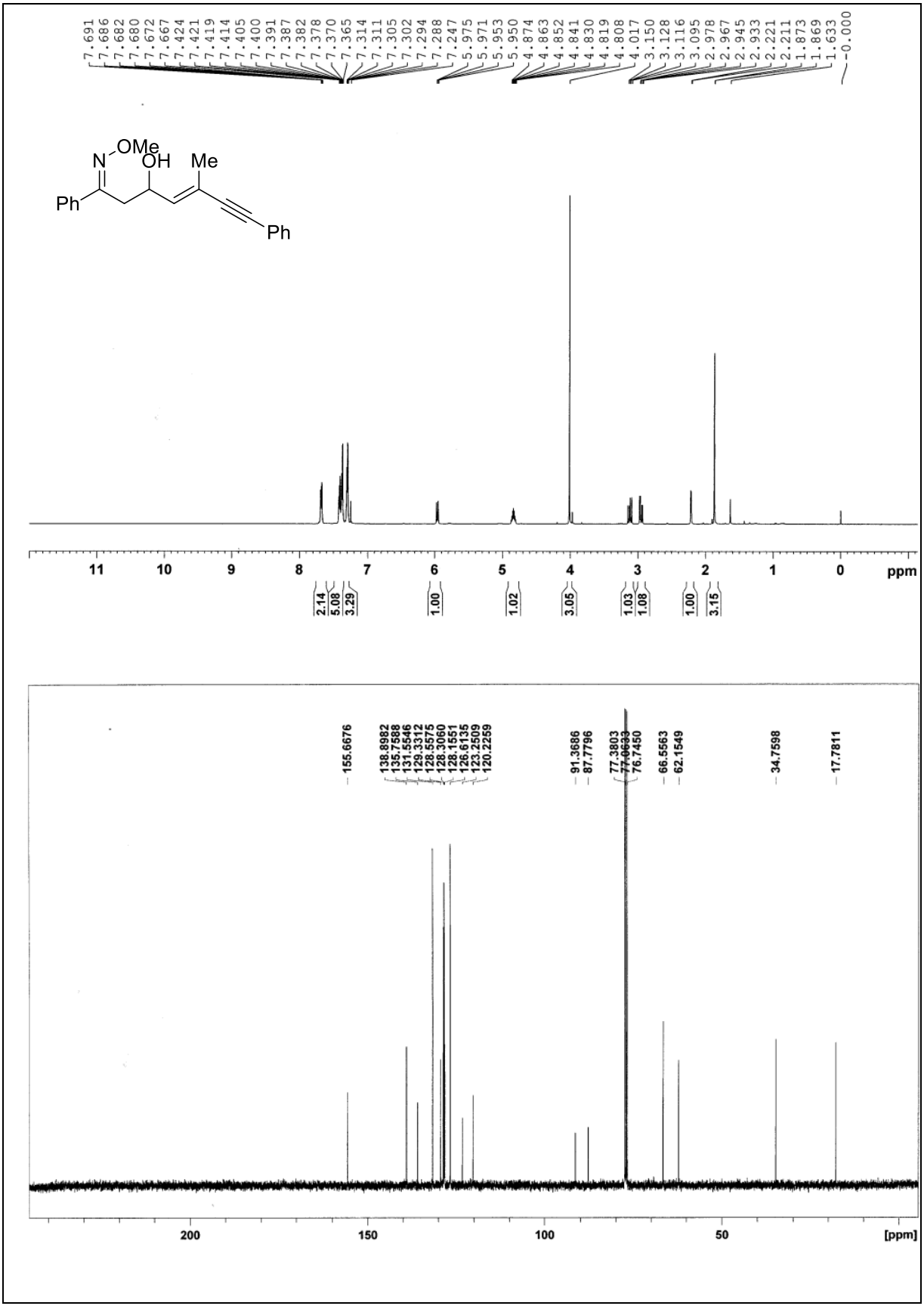
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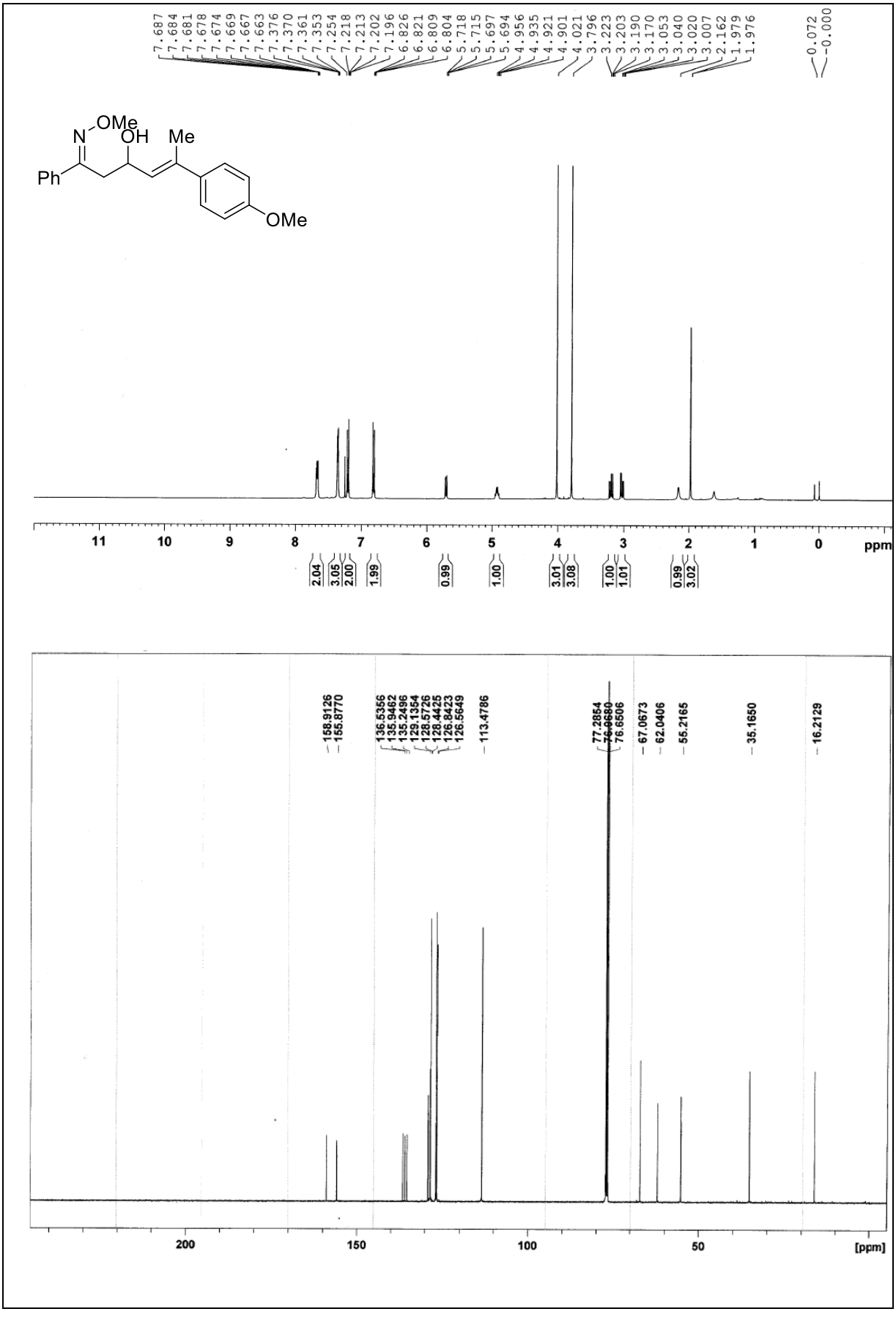
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Ph
OMe
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1.00
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1.01
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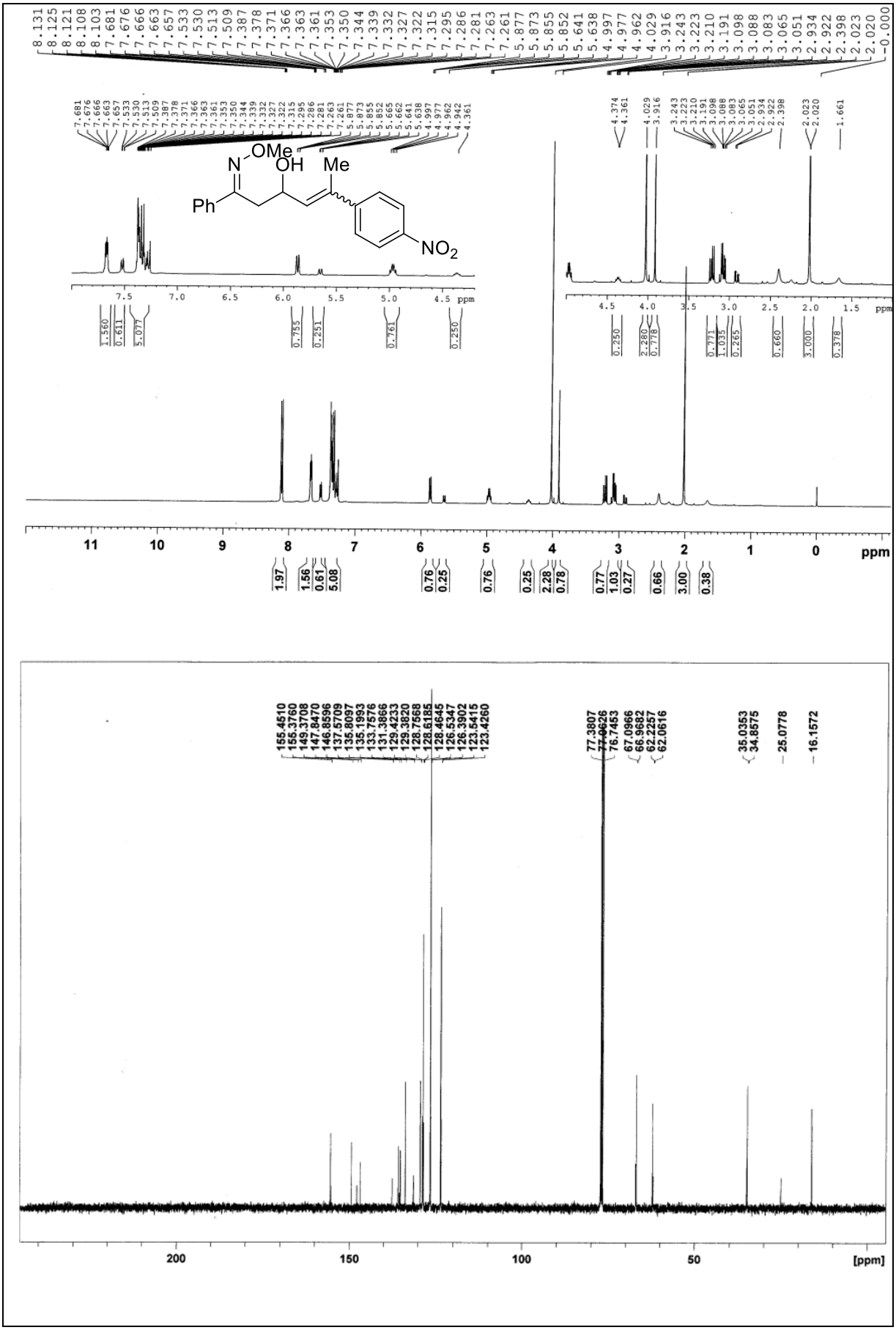
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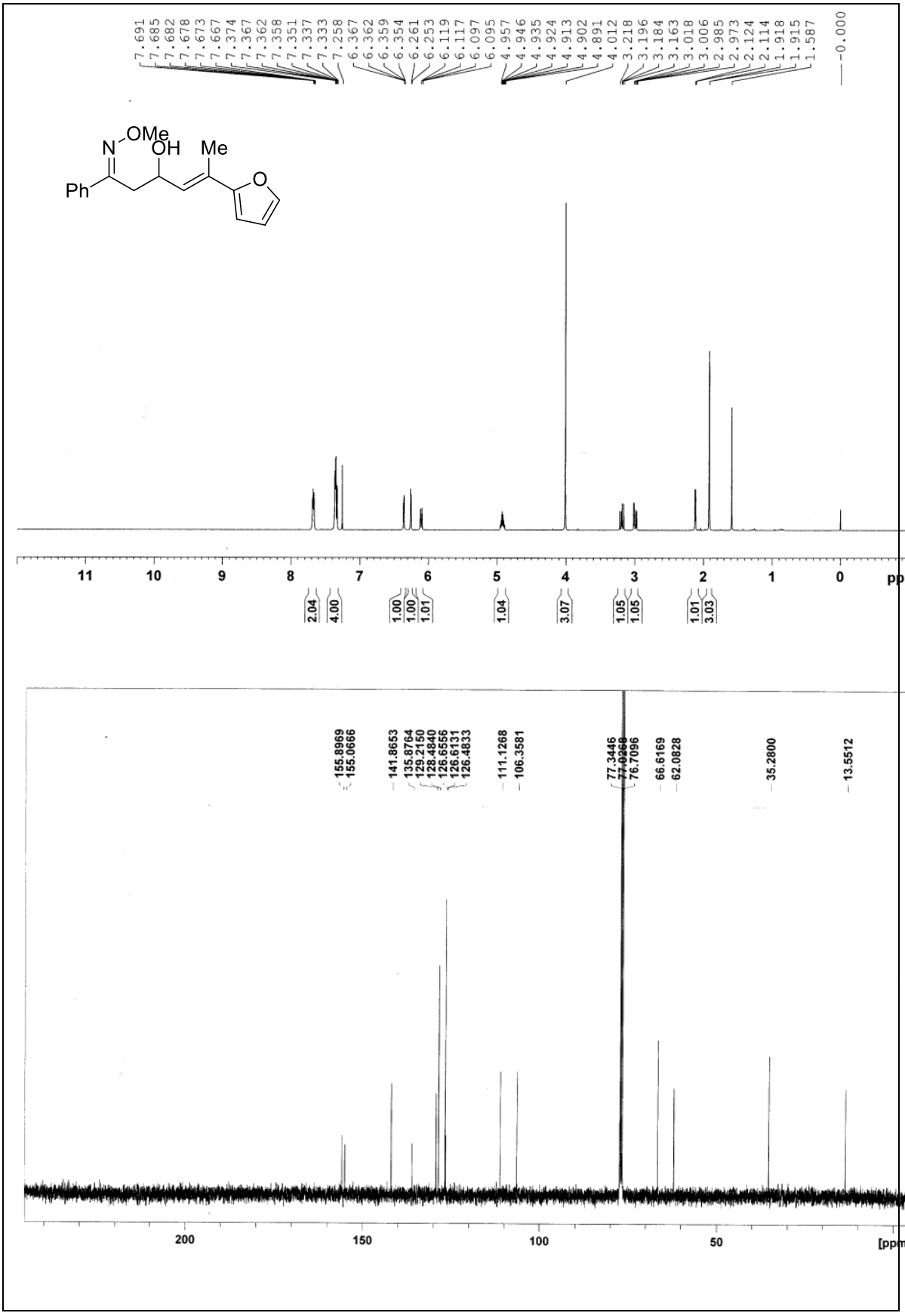
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OH
Me
Ph
O
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ppm
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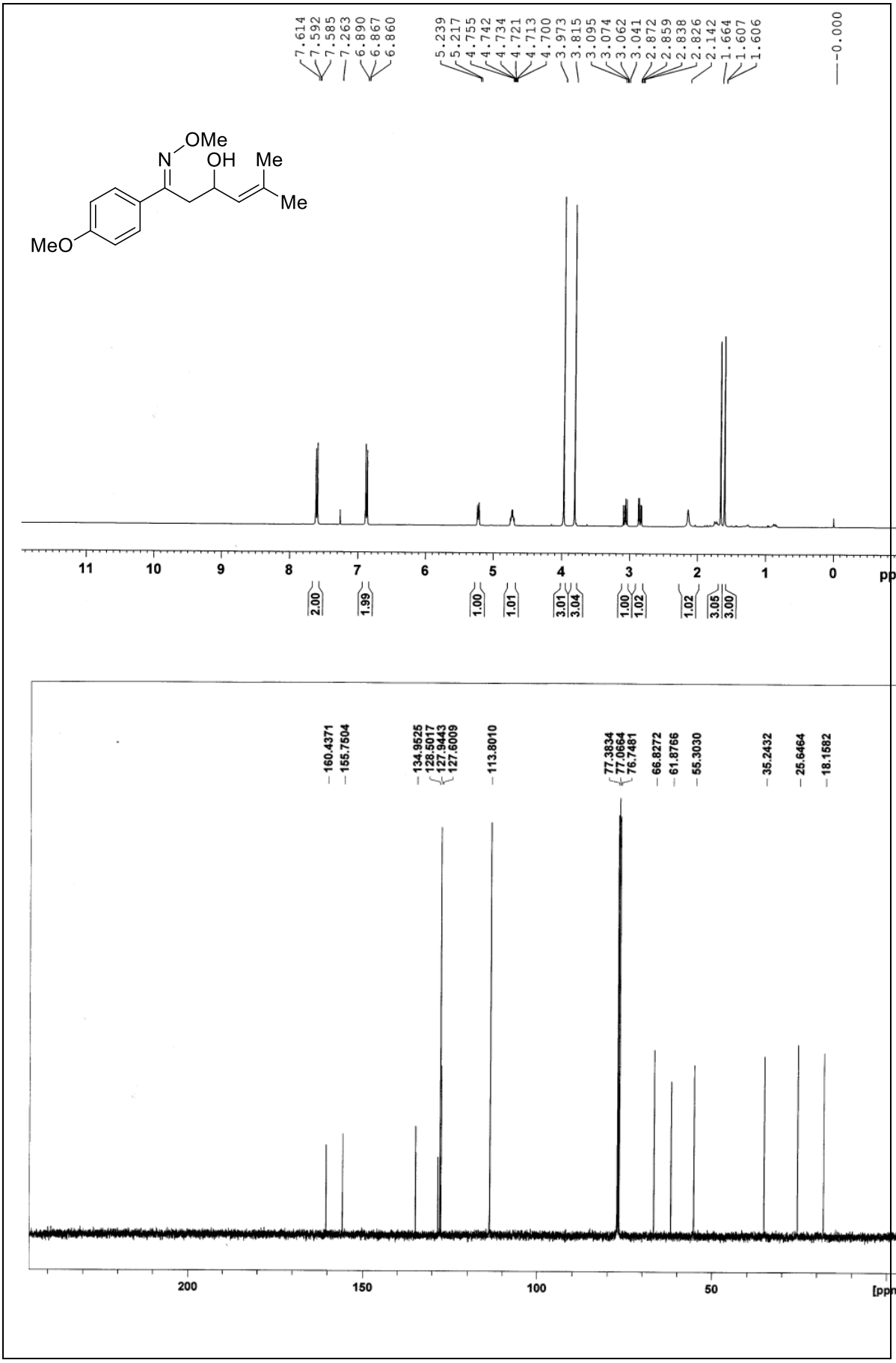
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Me
Me
MeO
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1.02
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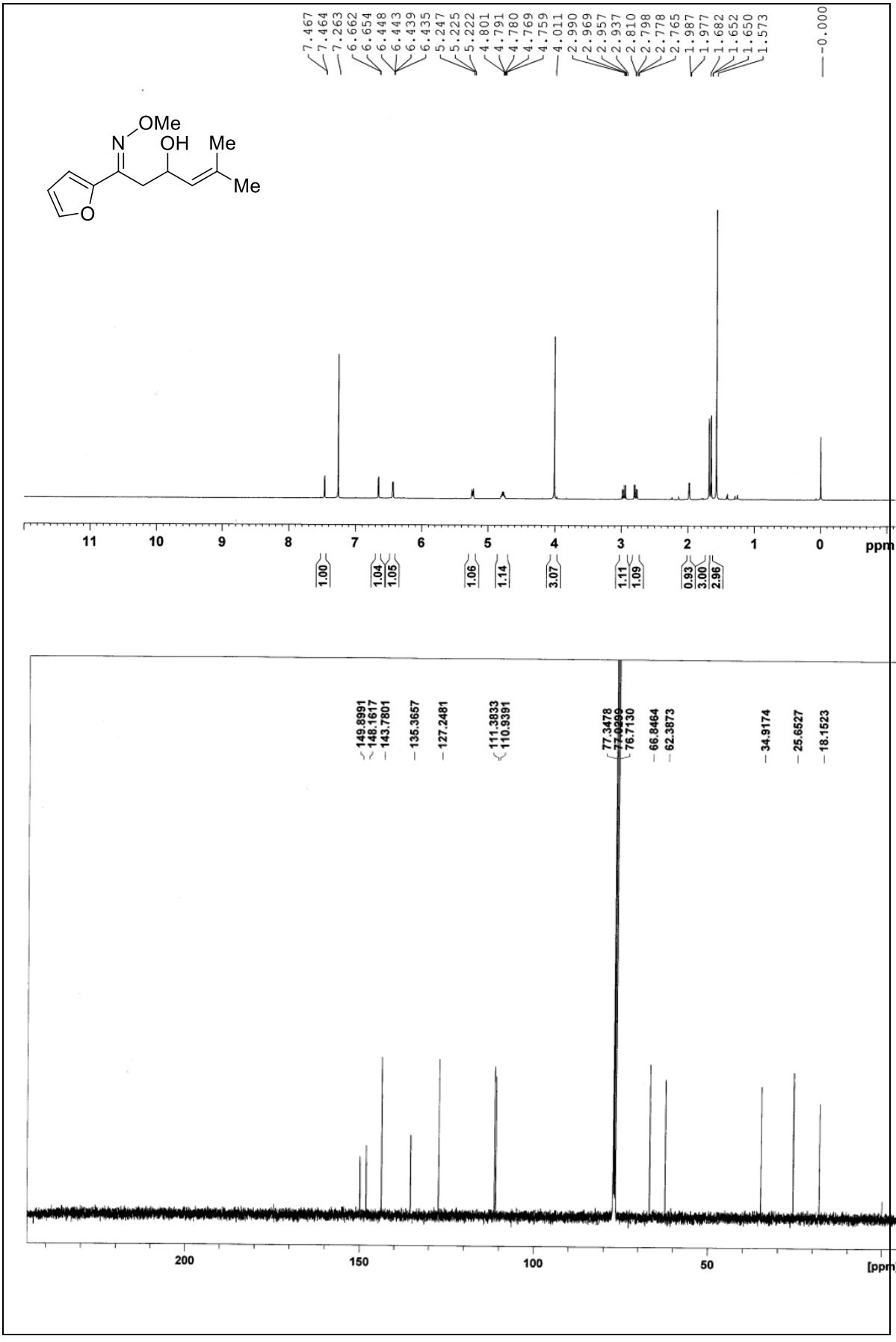
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OMe
N
OH
Me
Me
O
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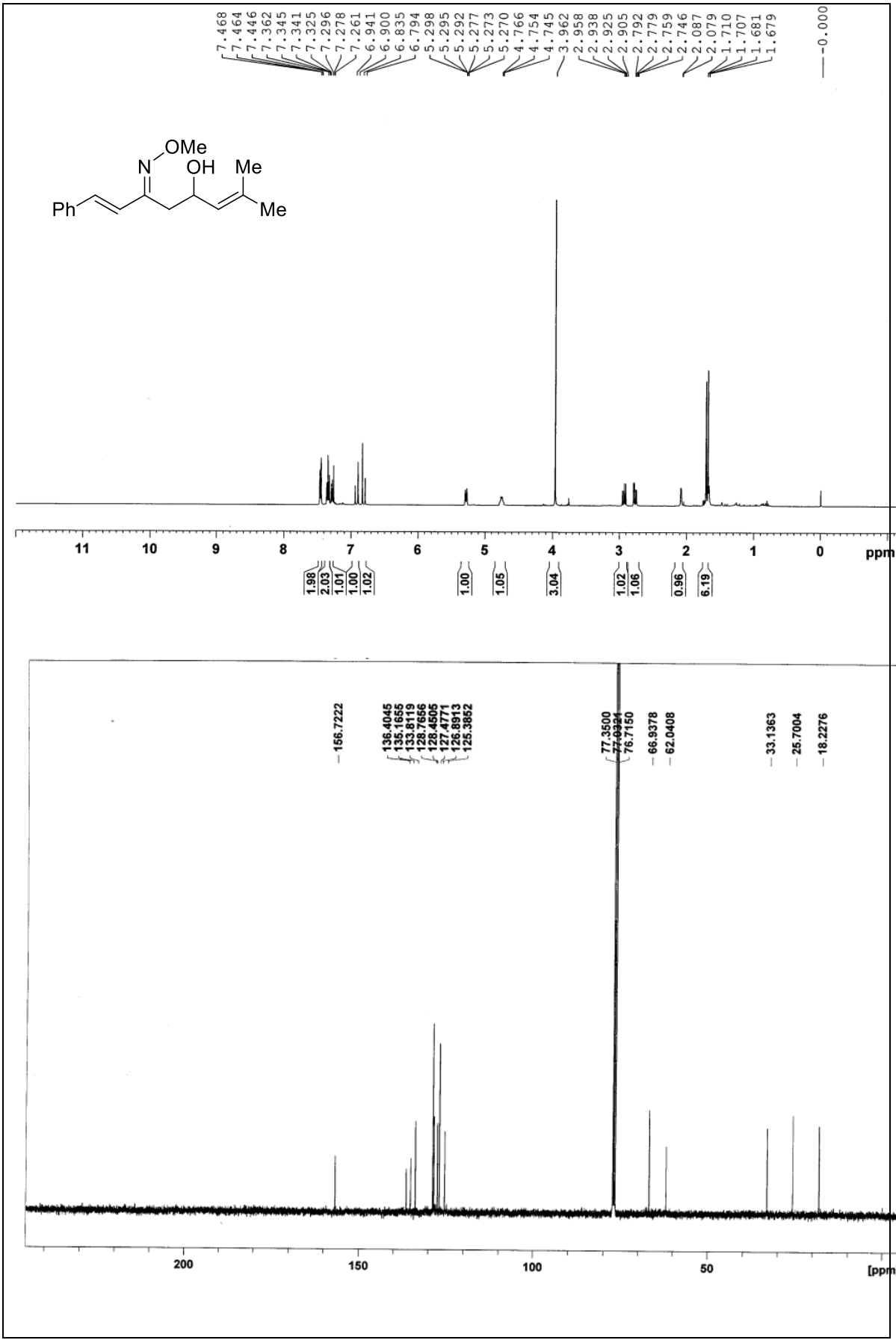
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5.277
5.273
5.270
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4.745
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2.925
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2.779
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2.746
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N
OH
Me
Ph
Me
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5
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ppm
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1.00
1.02
1.00
1.05
3.04
1.02
1.06
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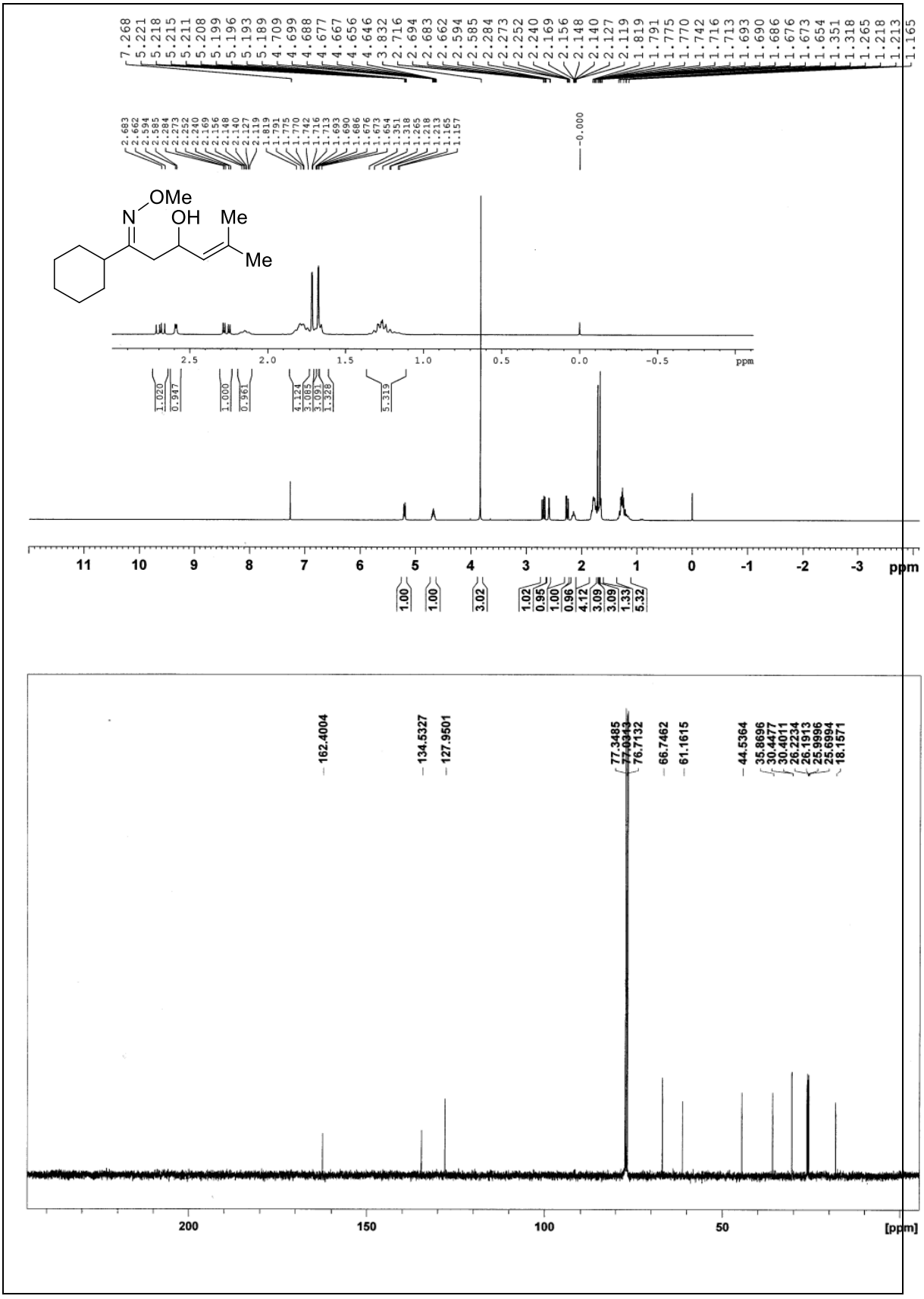
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Me
ppm
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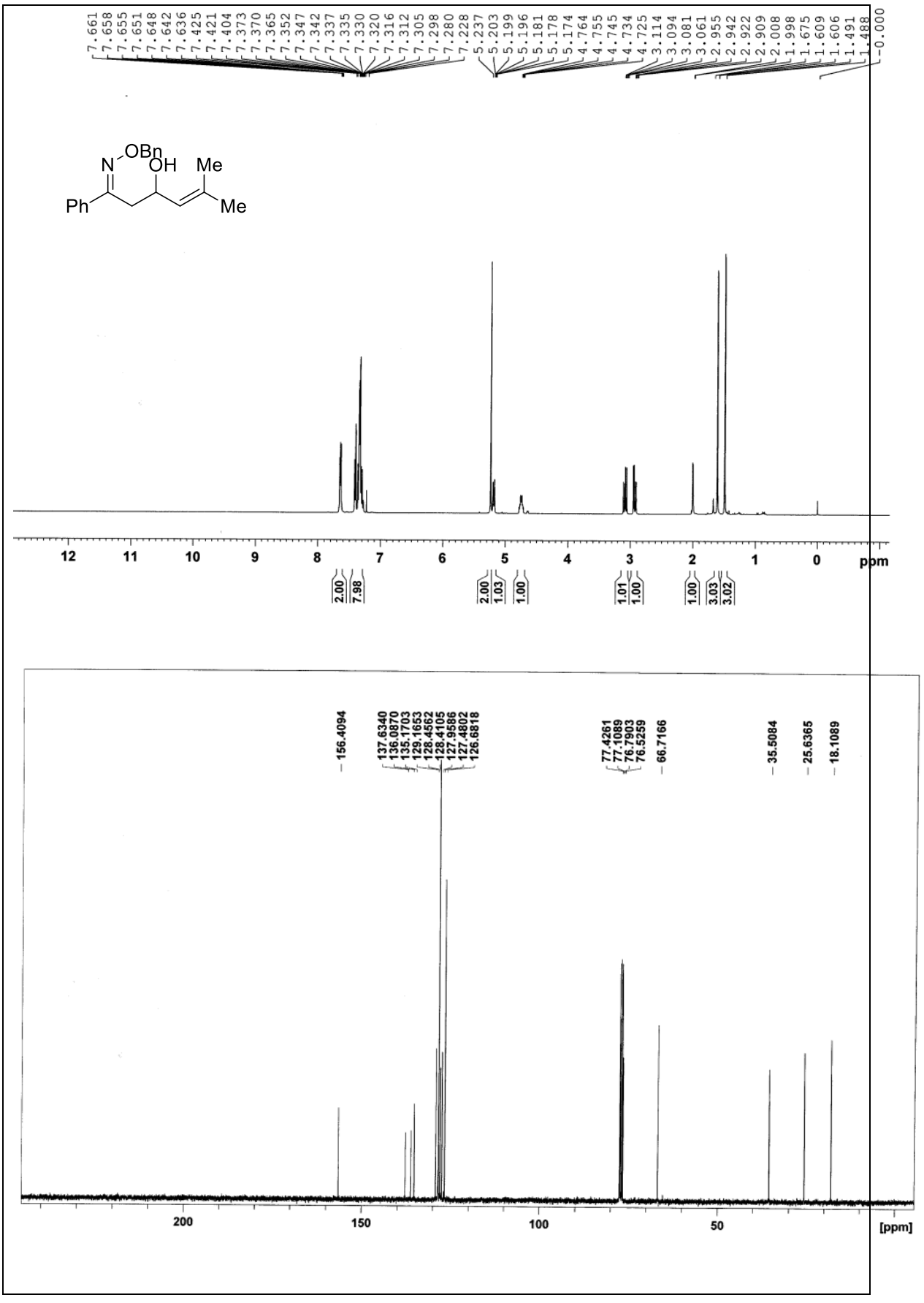
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OBn
OH
Me
Ph
Me
12
11
10
9
8
7
6
5
4
3
2
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0
ppm
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1.00
1.01
1.00
1.00
3.03
3.02
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76.7903
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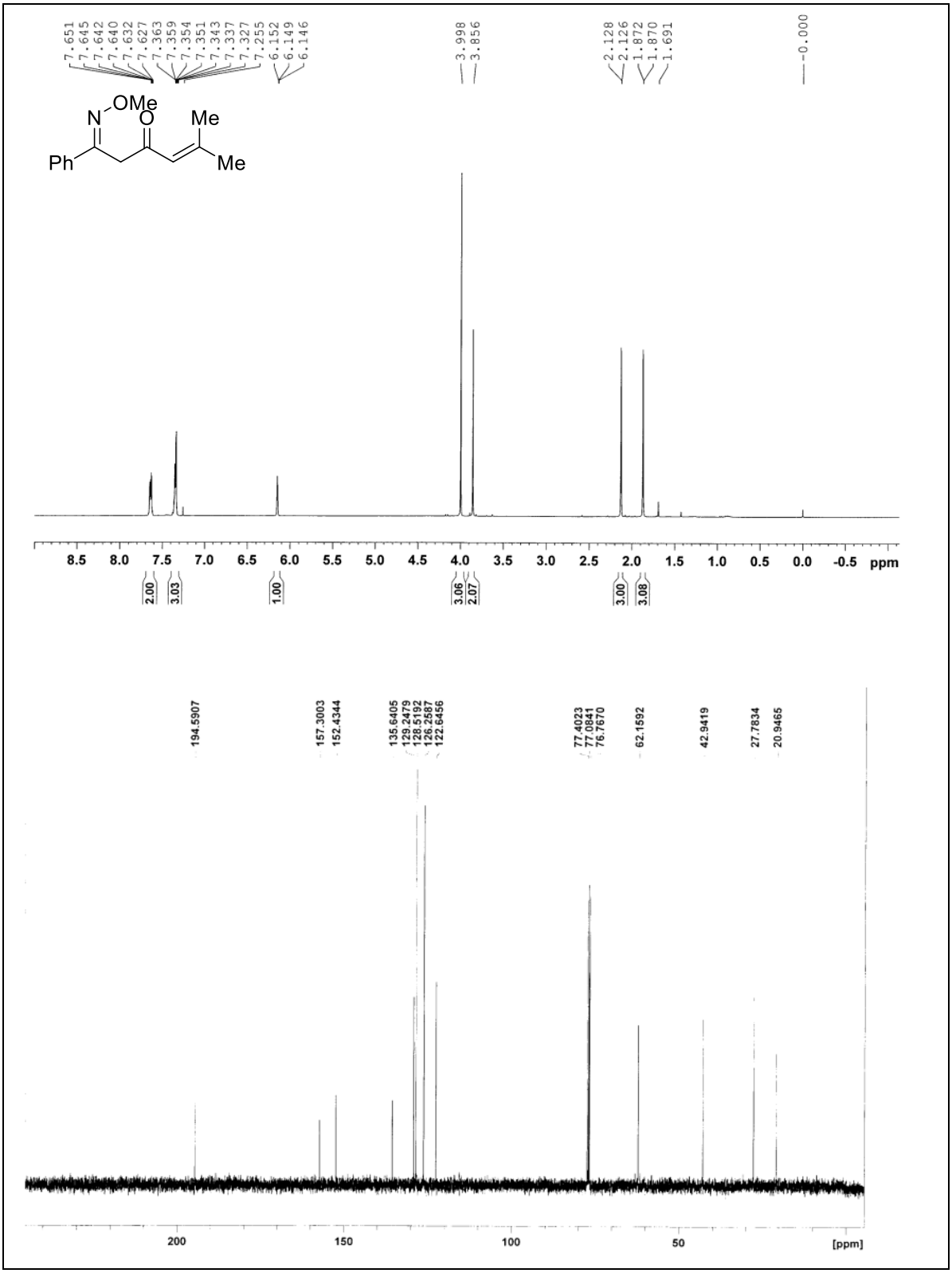
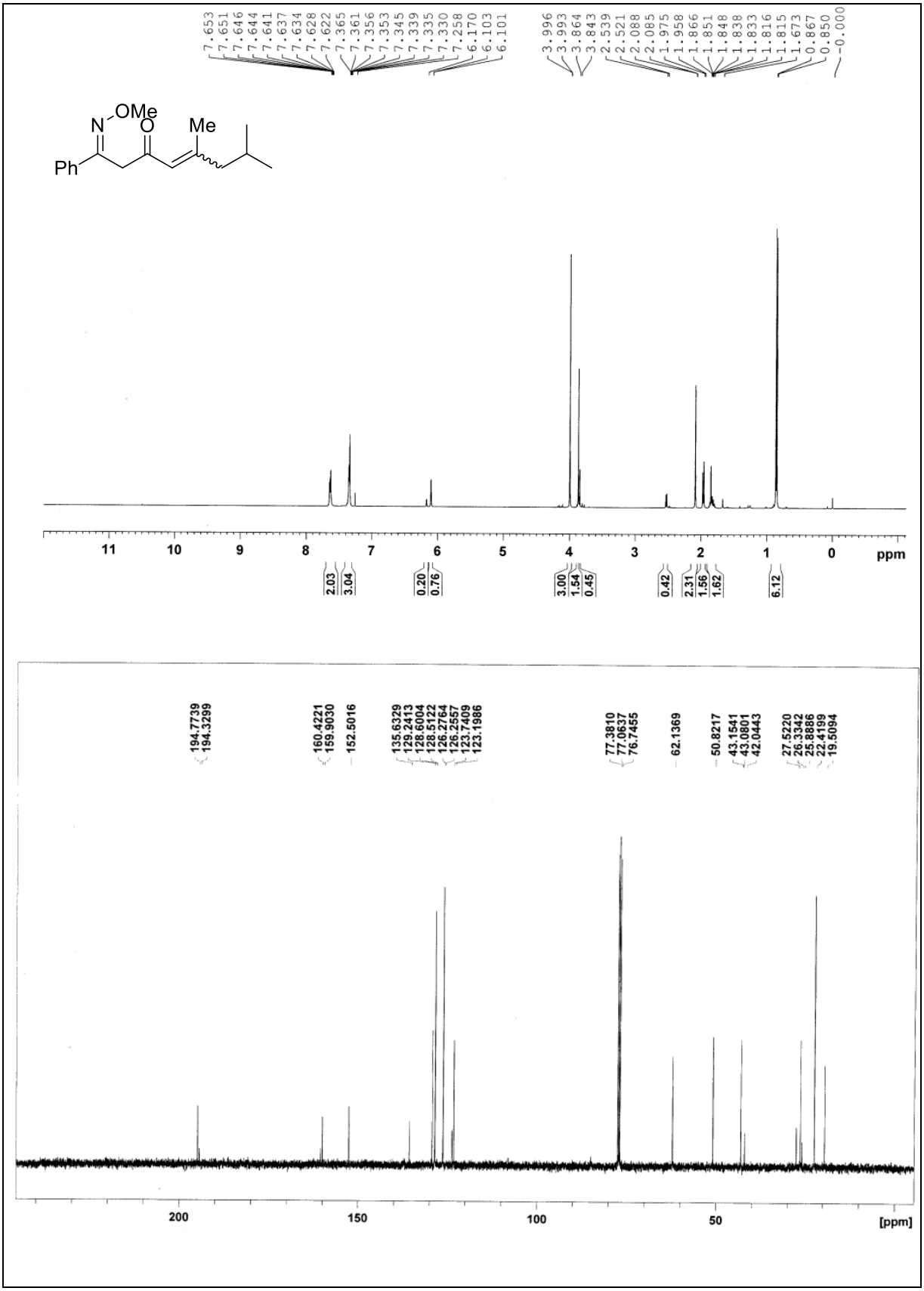
[ppm]

^{1}H and ^{13}C NMR spectra of β-keto oxime ethers

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7.339
7.335
7.330
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O
Me
Ph
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10
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7
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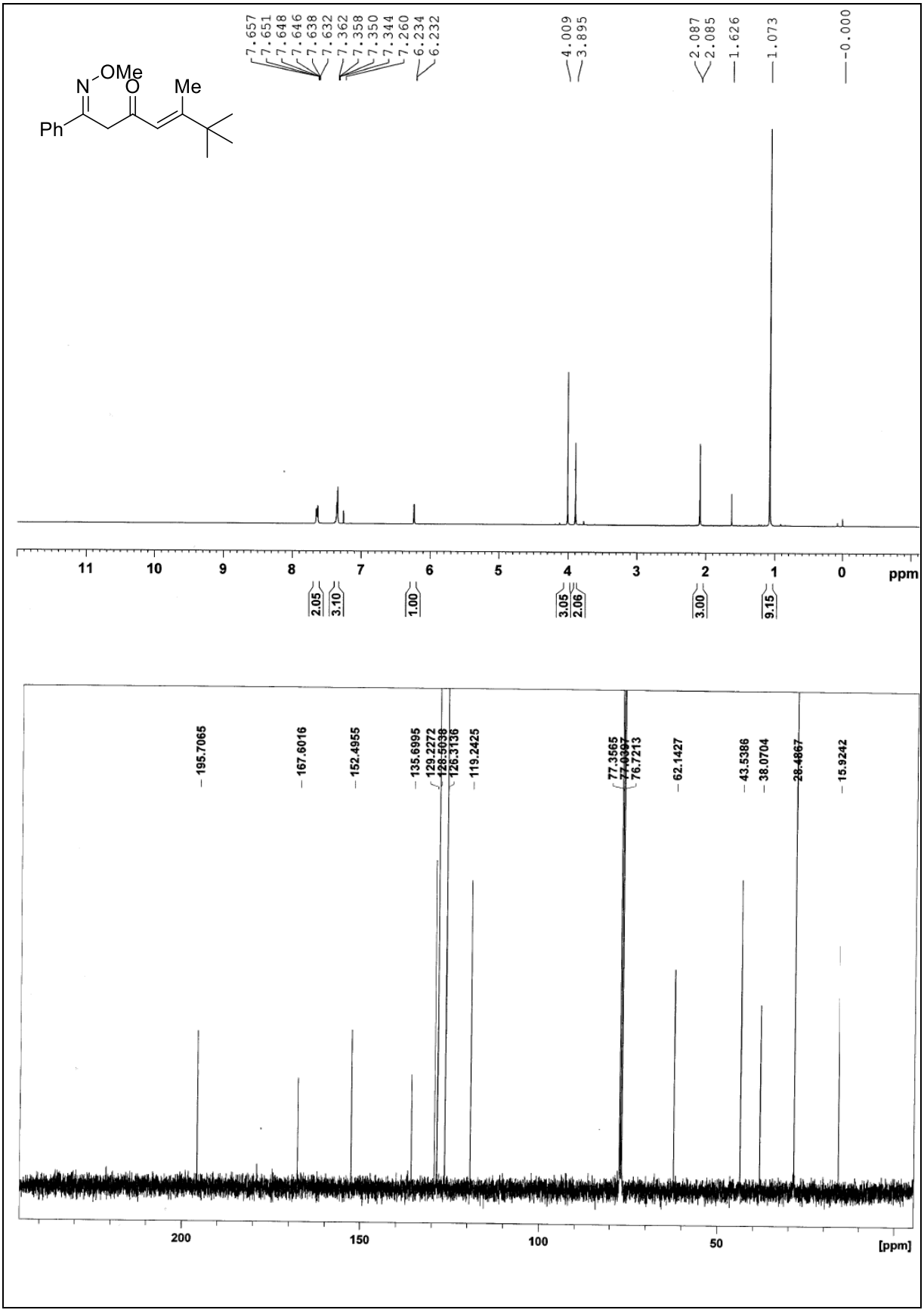
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8
7
6
5
4
3
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ppm
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3.10
1.00
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2.06
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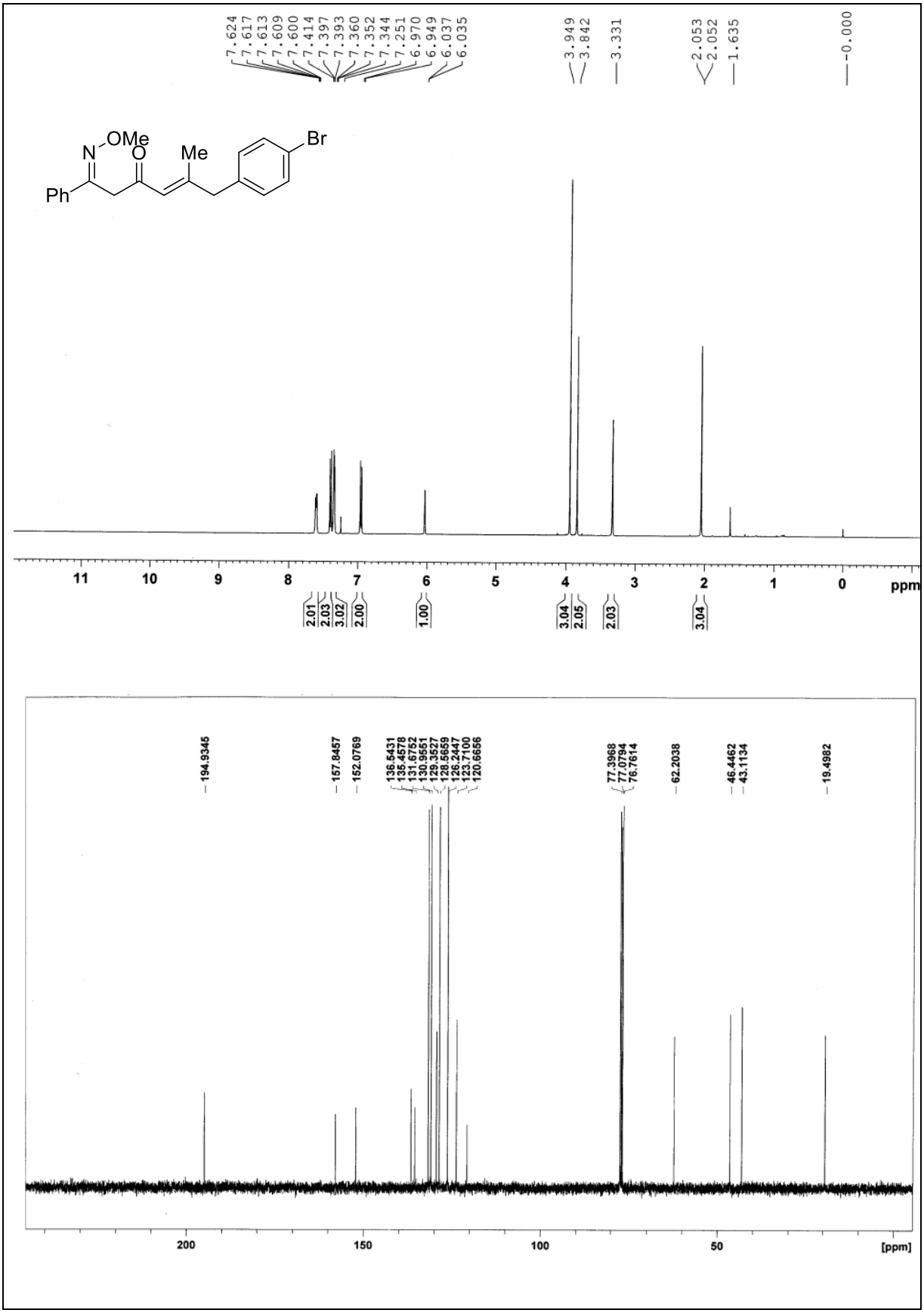
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O
Me
Br
Ph
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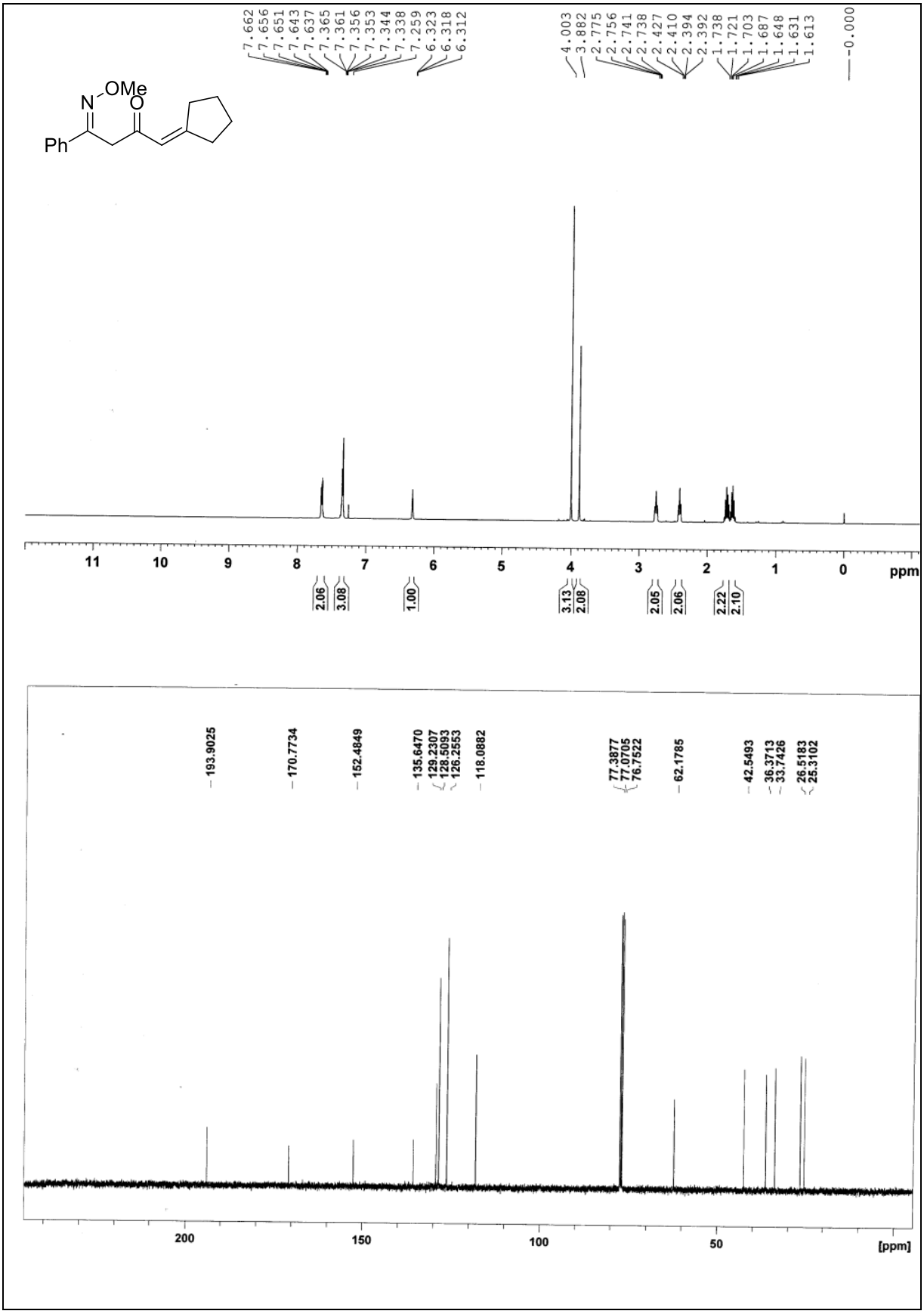
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OMe
N
O
Ph
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ppm
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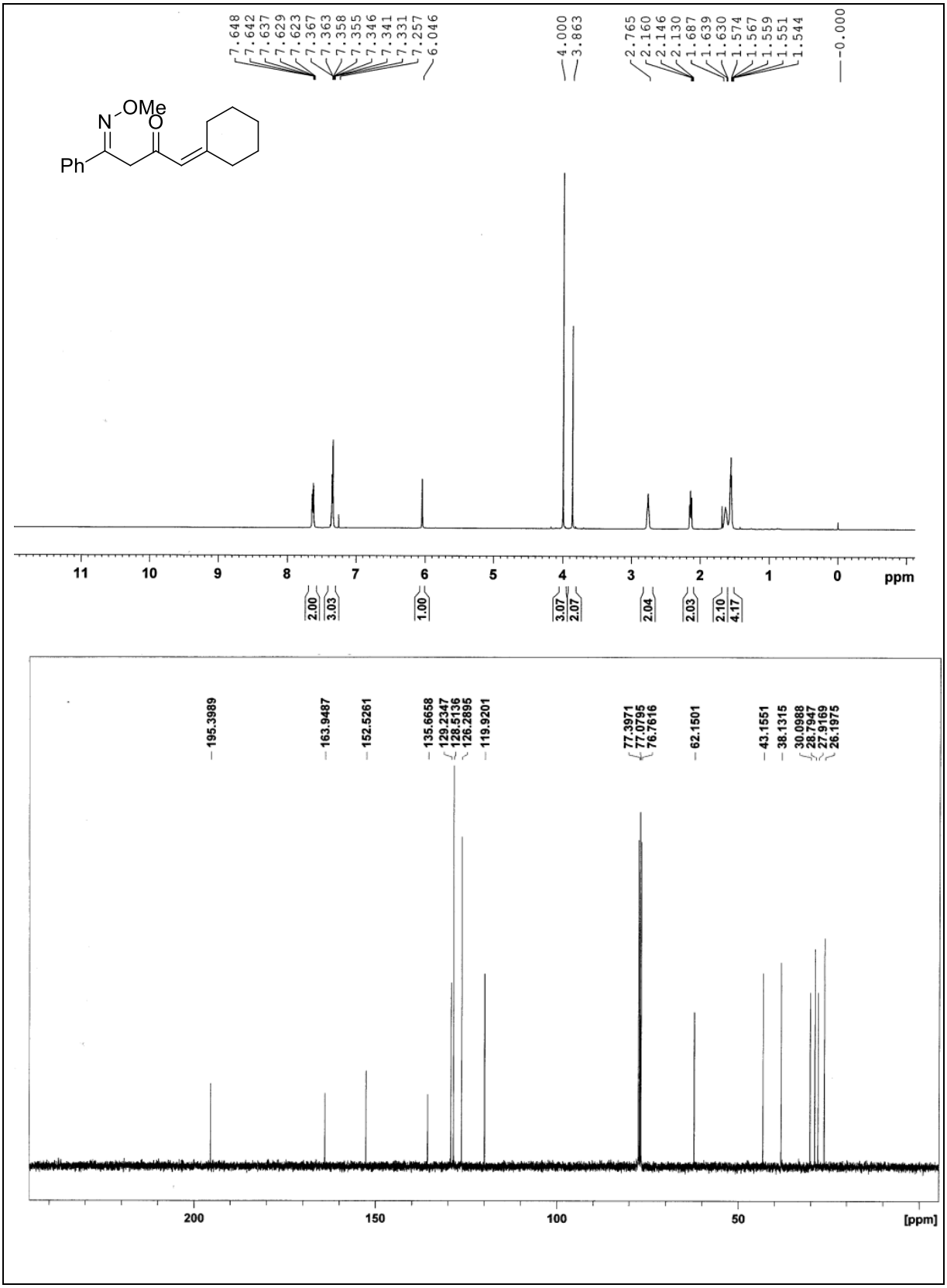
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OMe
N
O
Ph
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10
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8
7
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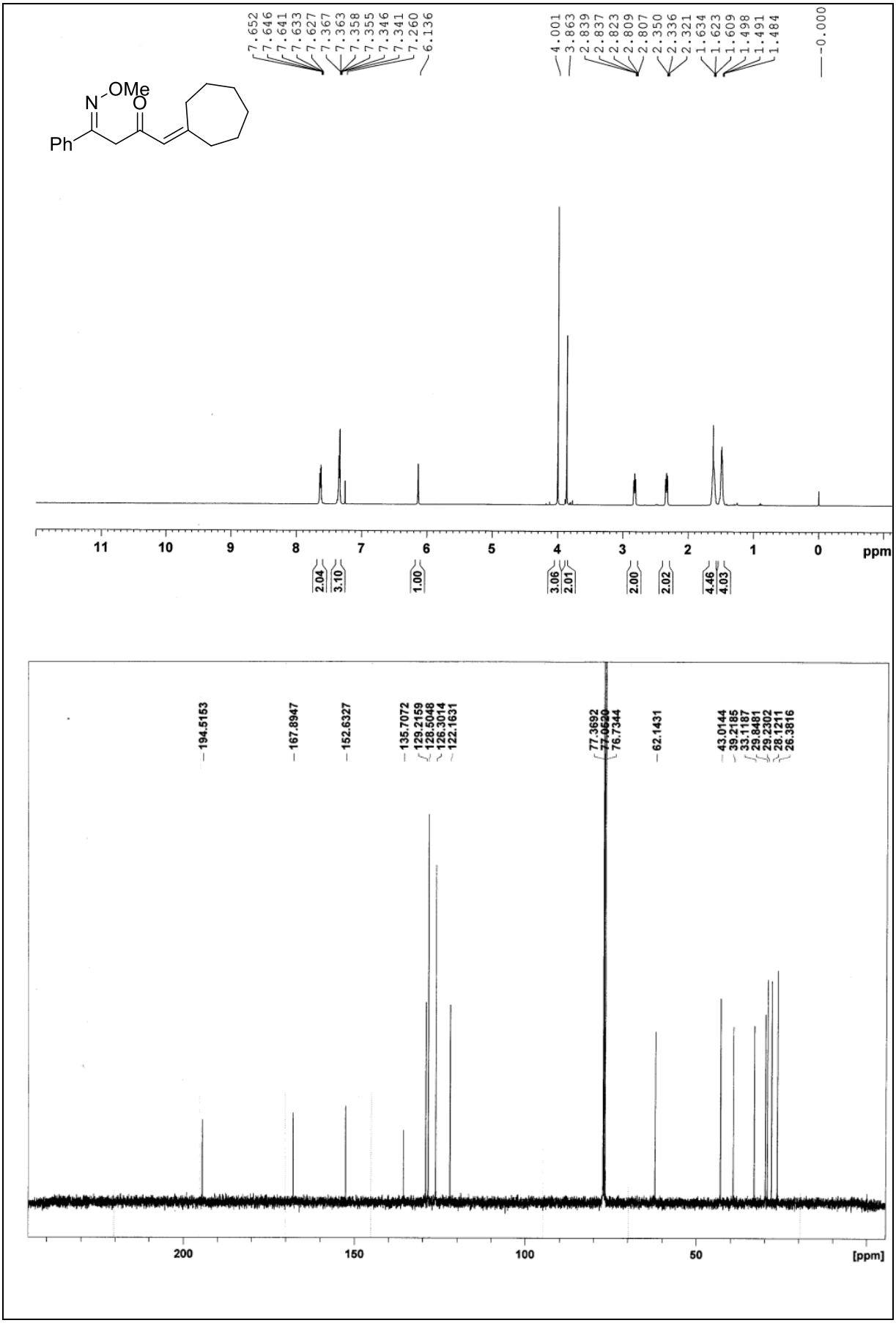
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O
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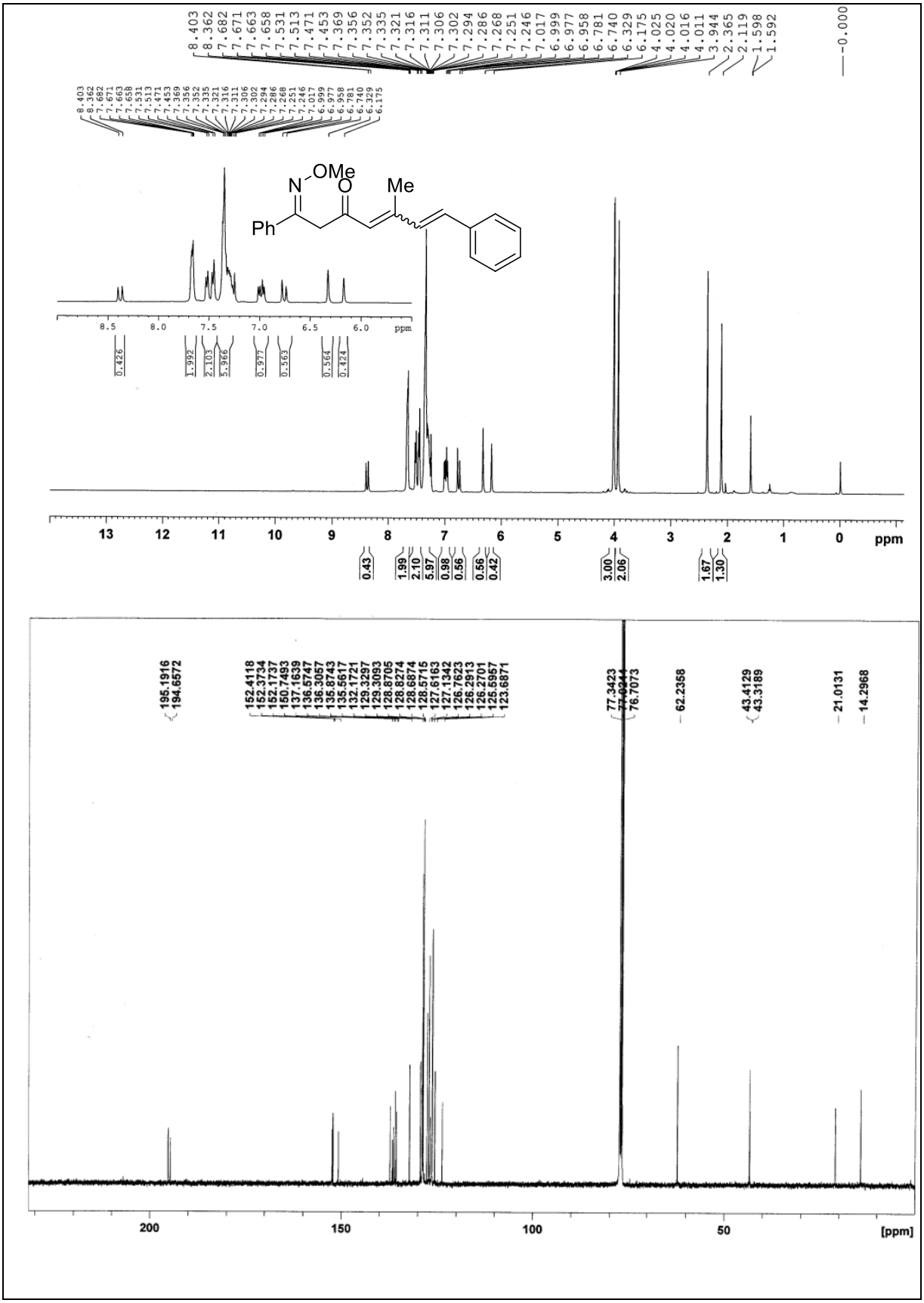
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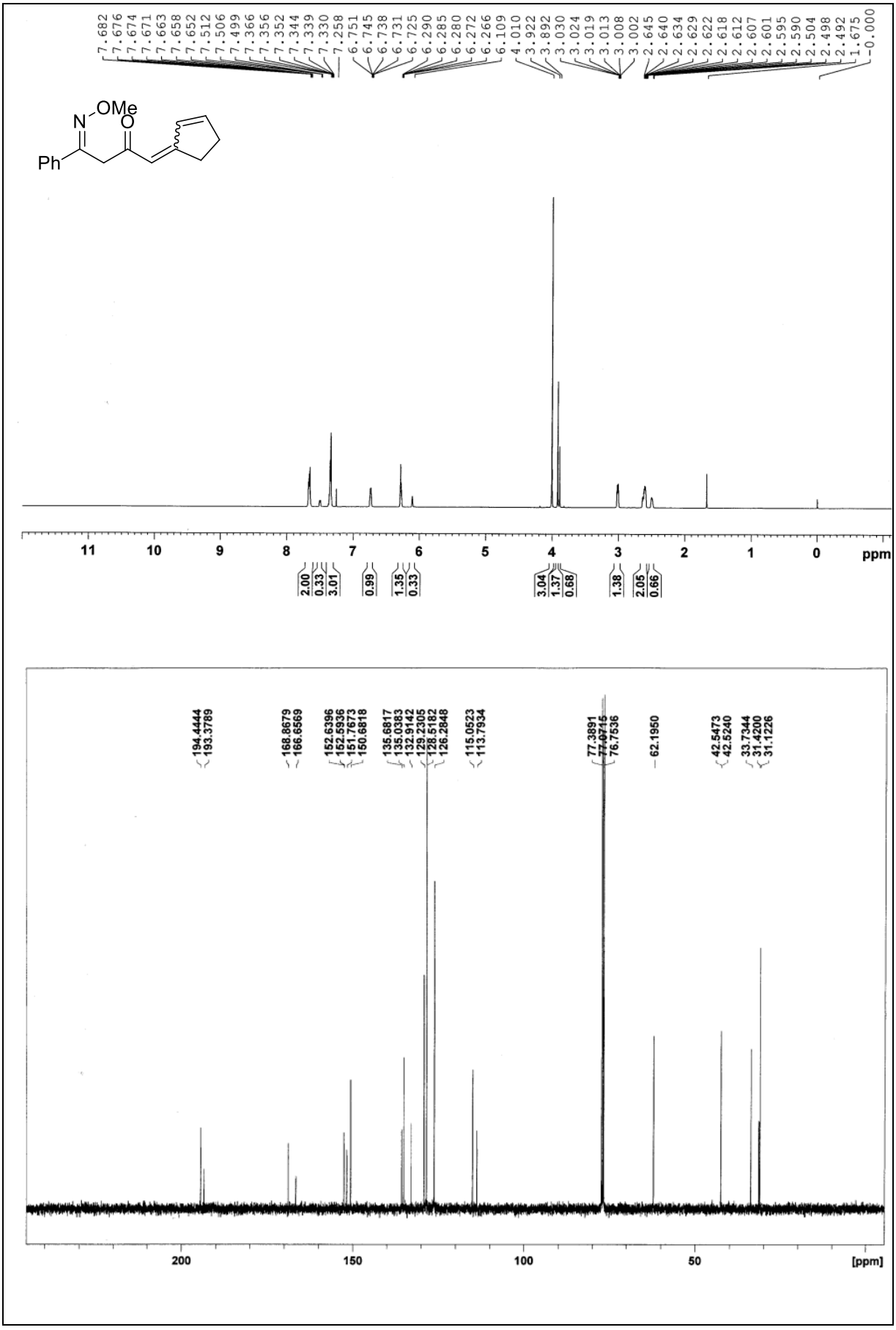
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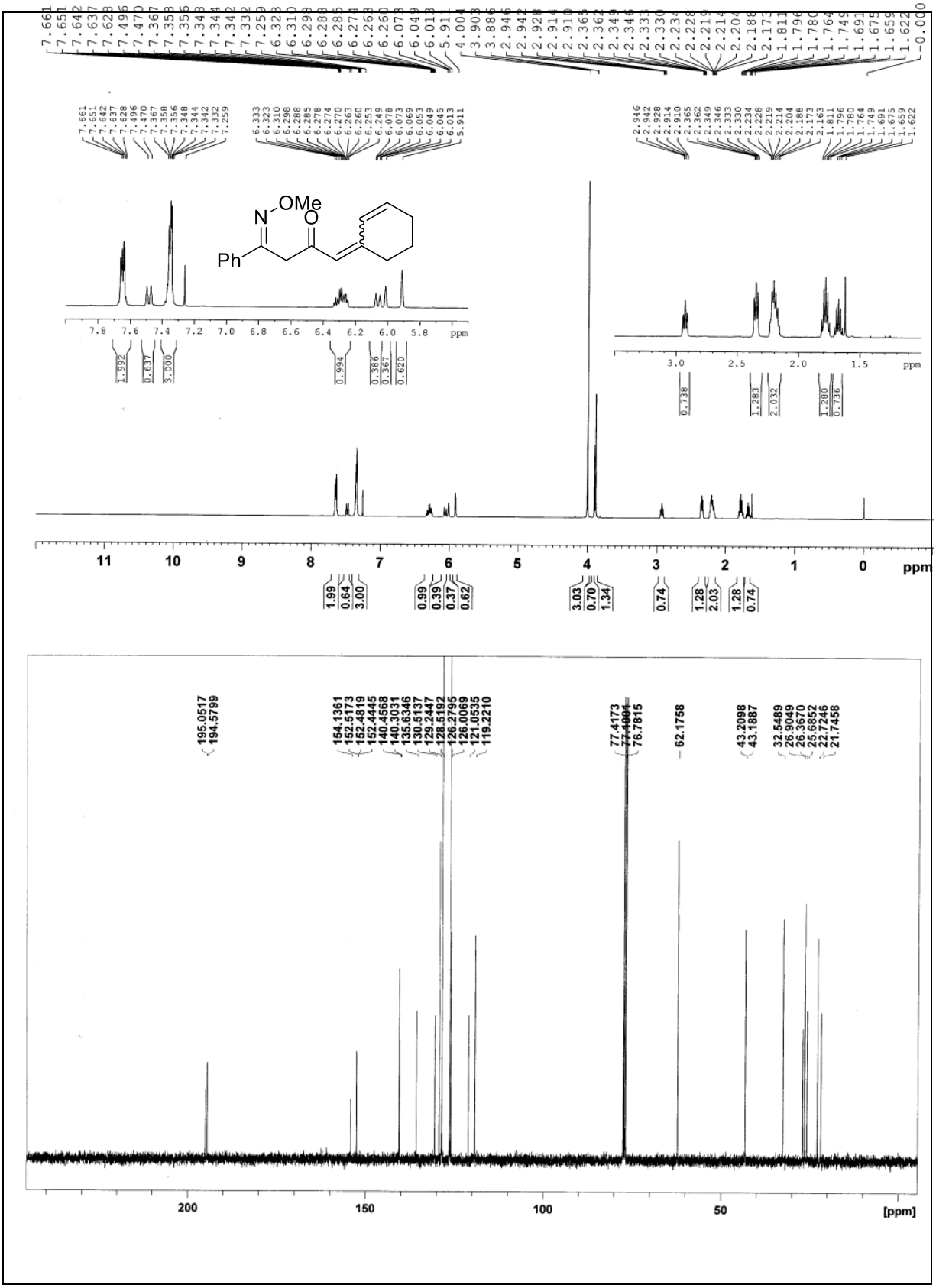
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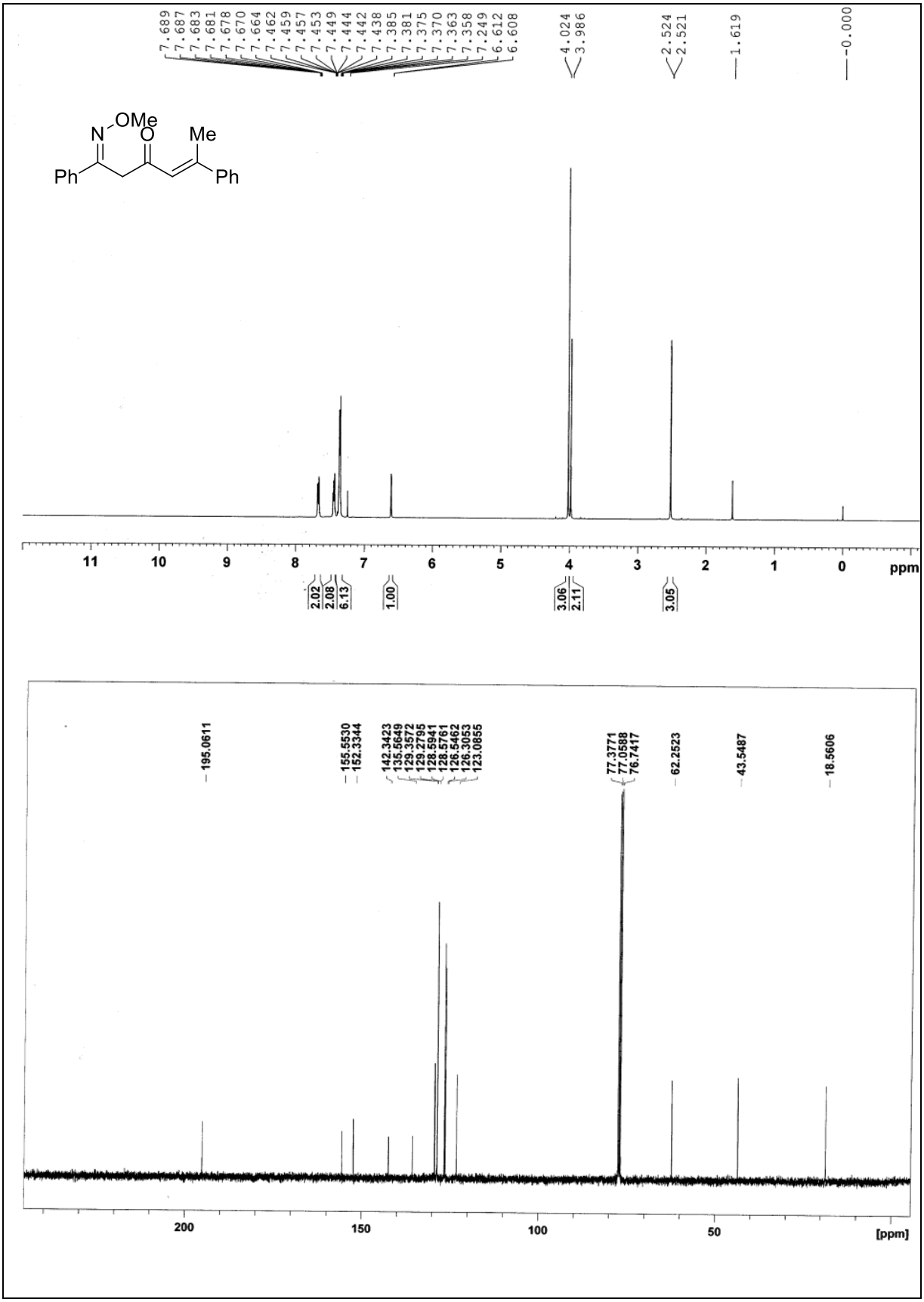
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2.521
1.619
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OMe
O
Me
Ph
Ph
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10
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6
5
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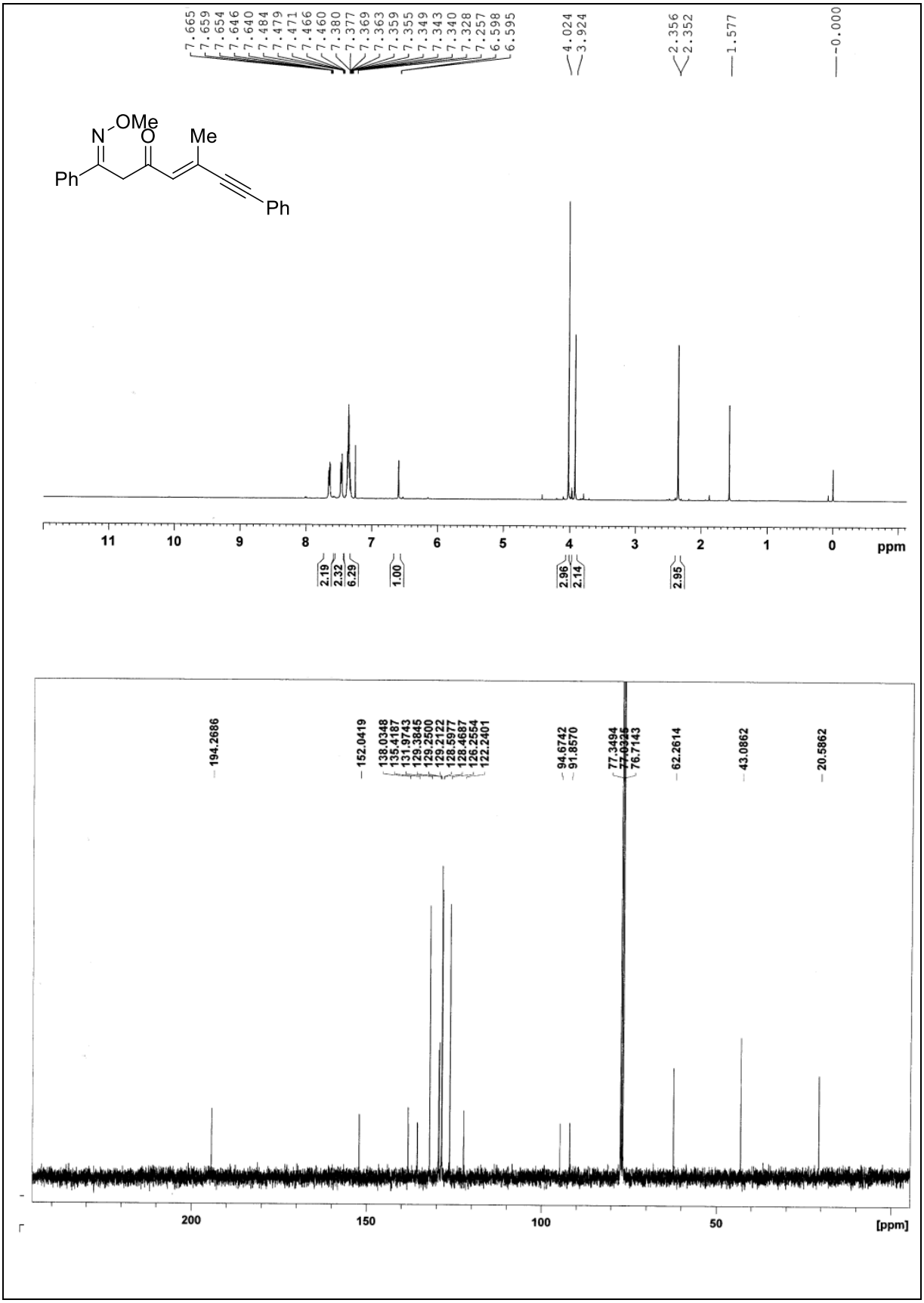
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2.352
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OMe
O
Me
Ph
Ph
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8
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6
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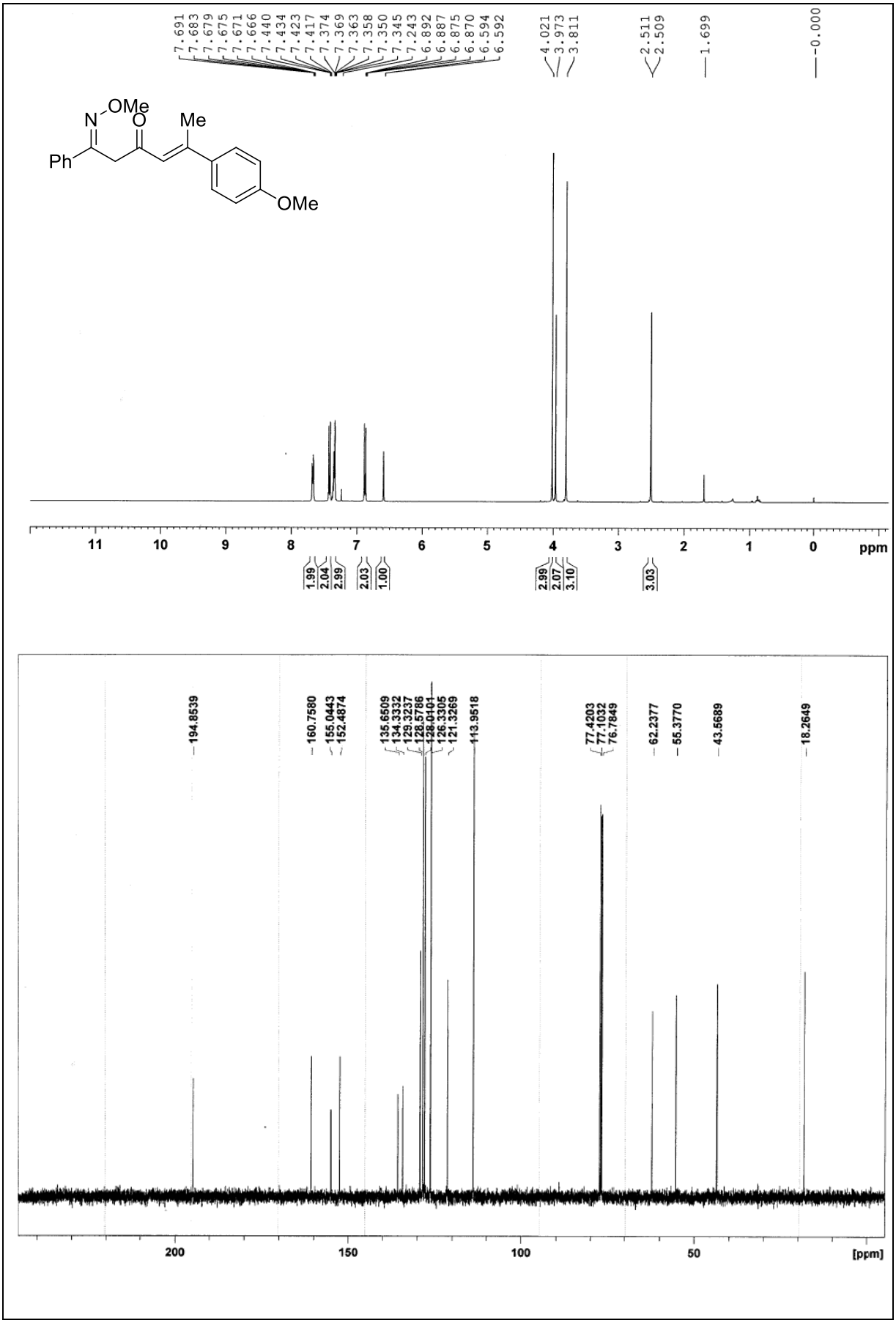
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3.973
3.811
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N
OMe
O
Me
Ph
OMe
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10
9
8
7
6
5
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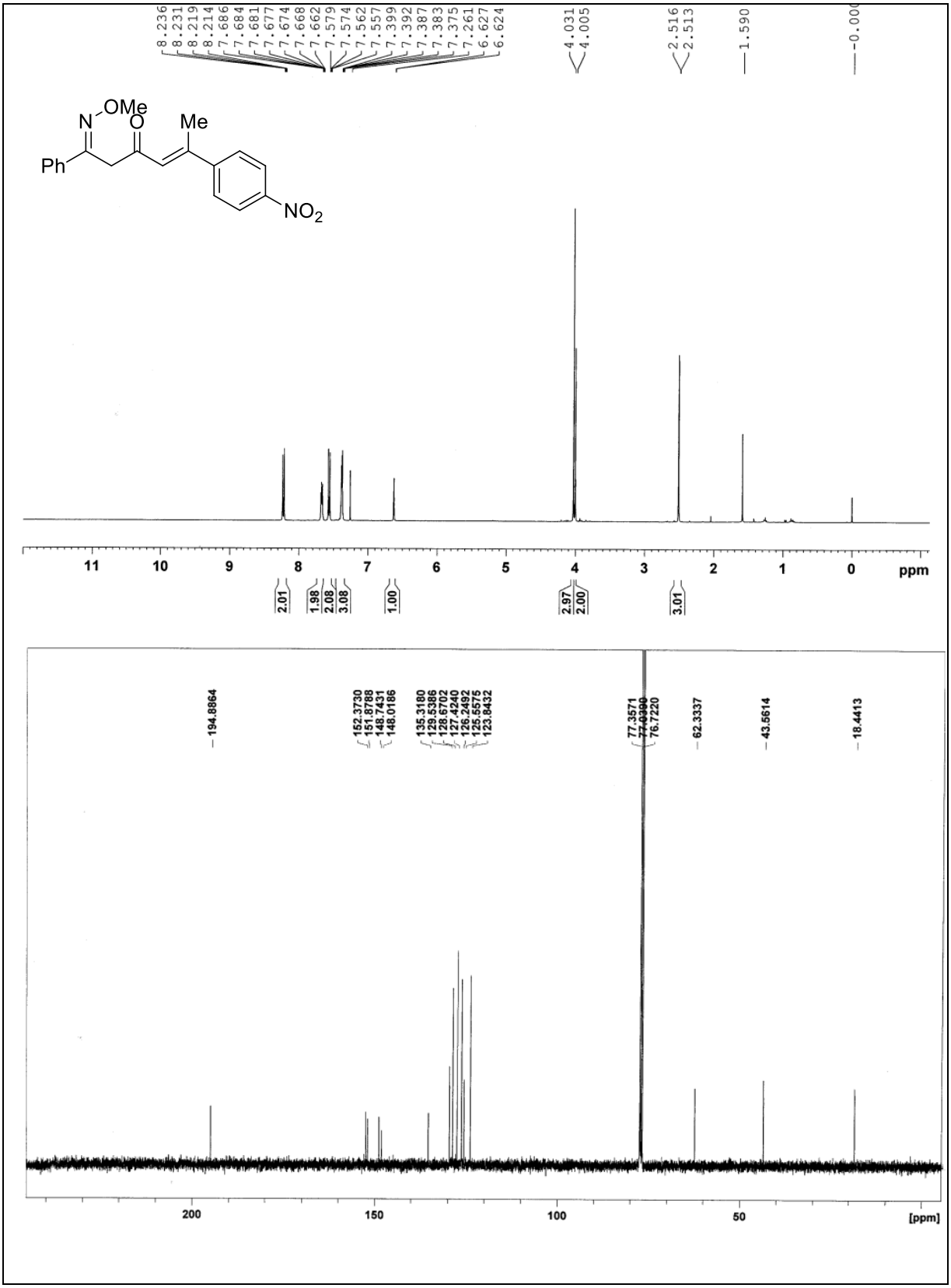
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7.662
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7.562
7.557
7.399
7.392
7.387
7.383
7.375
7.261
6.627
6.624
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4.005
2.516
2.513
1.590
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OMe
O
Me
Ph
NO2
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10
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8
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5
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0
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3.08
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2.00
3.01
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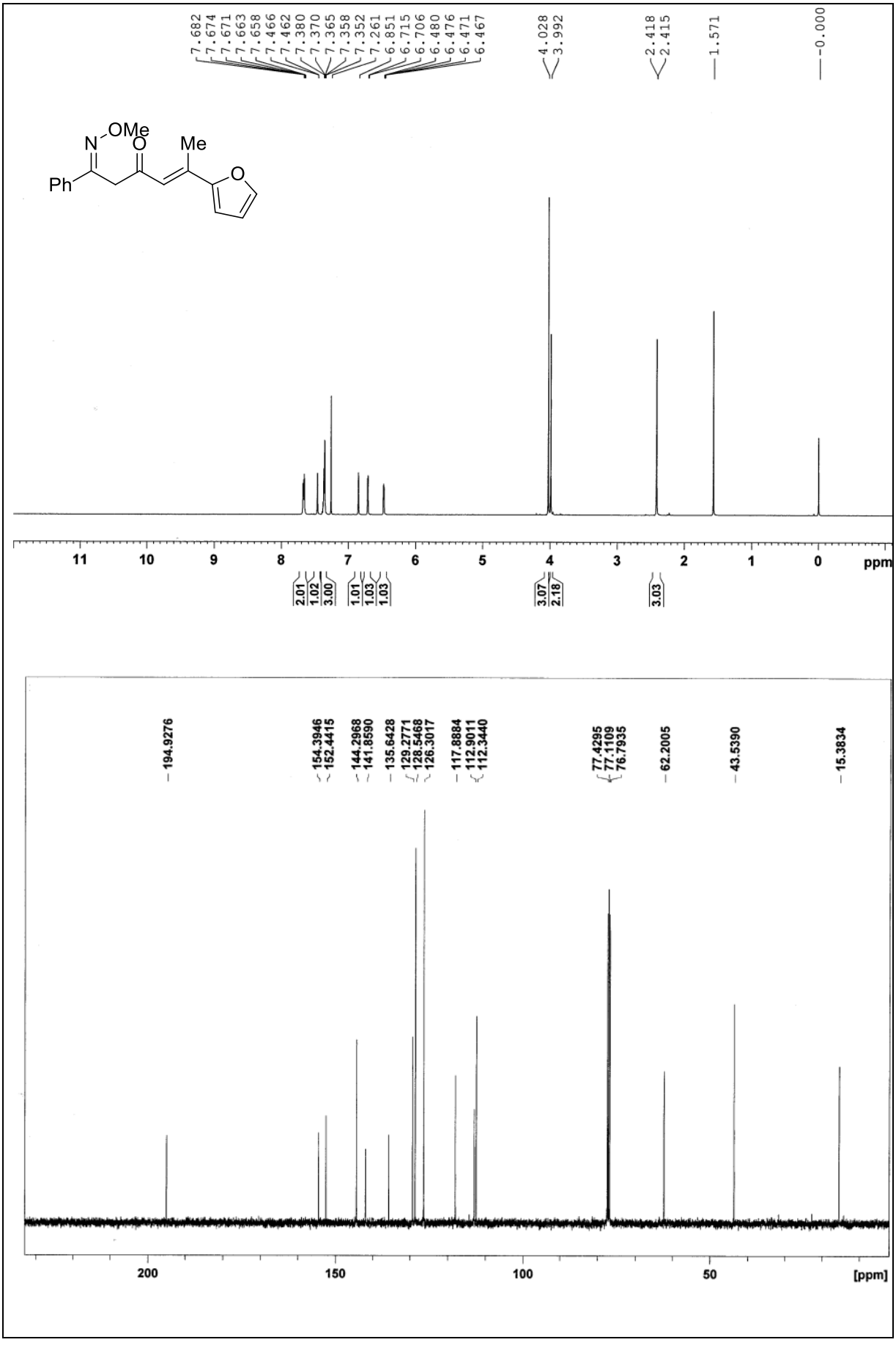
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7.358
7.352
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6.715
6.706
6.480
6.476
6.471
6.467
4.028
3.992
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2.415
1.571
0.000
N
OMe
O
Me
Ph
O
11
10
9
8
7
6
5
4
3
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1
0
ppm
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1.02
3.00
1.01
1.03
1.03
3.07
2.18
3.03
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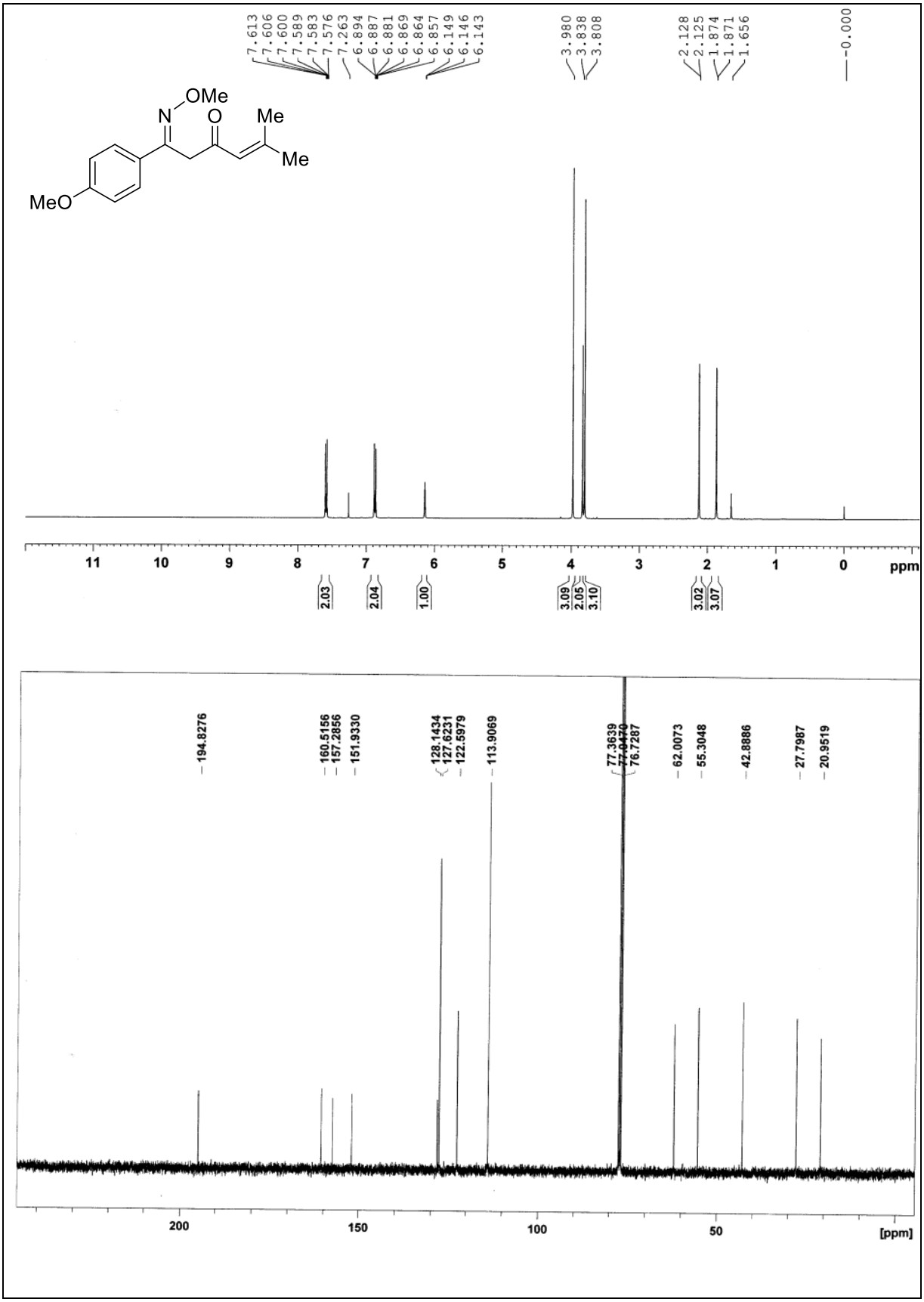
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6.857
6.149
6.146
6.143
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3.838
3.808
2.128
2.125
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1.871
1.656
0.000
OMe
N
O
Me
Me
MeO
11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.03
2.04
1.00
3.09
2.05
3.10
3.02
3.07
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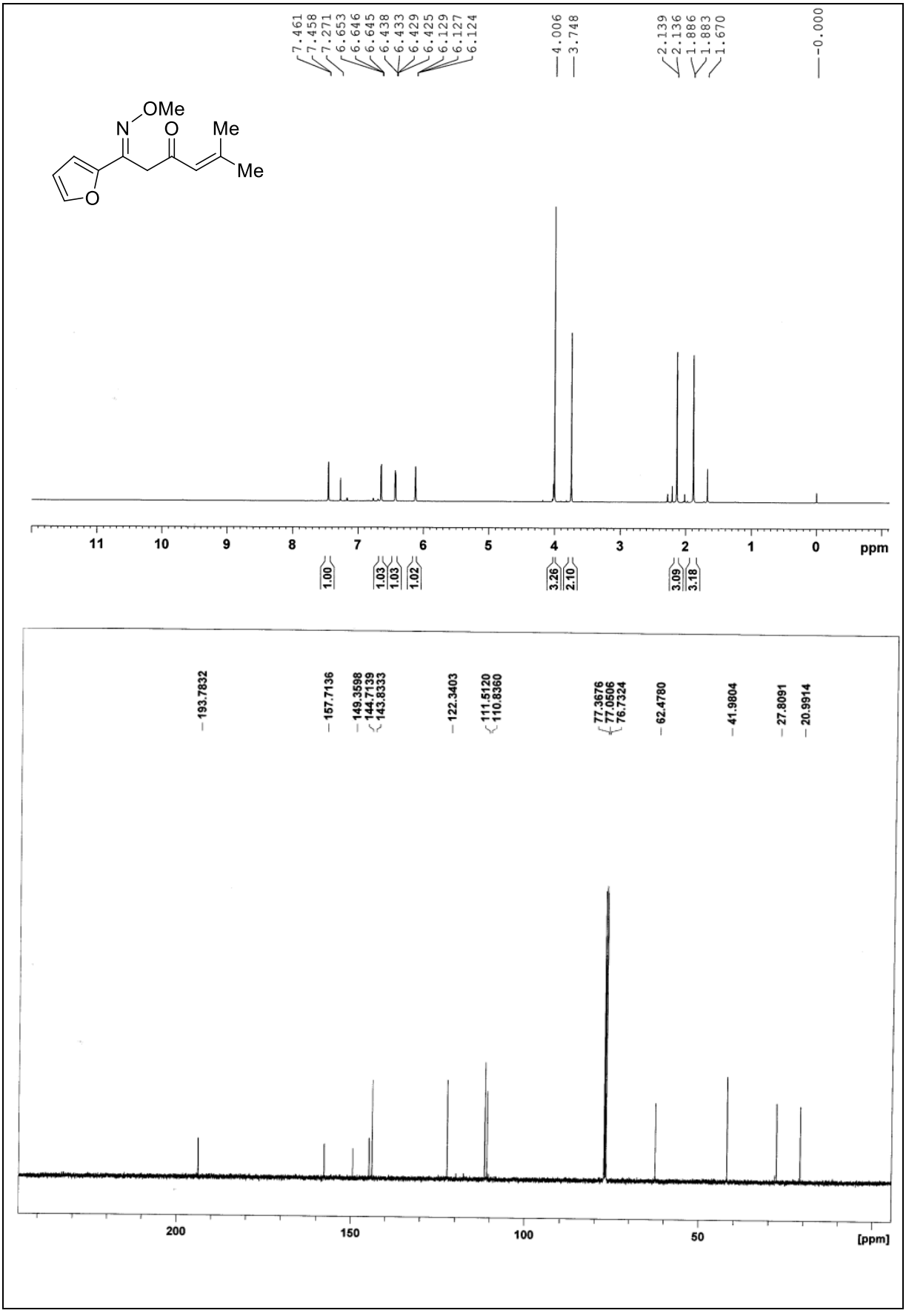
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6.127
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2.136
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OMe
N
O
Me
Me
O
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.03
1.03
1.02
3.26
2.10
3.09
3.18
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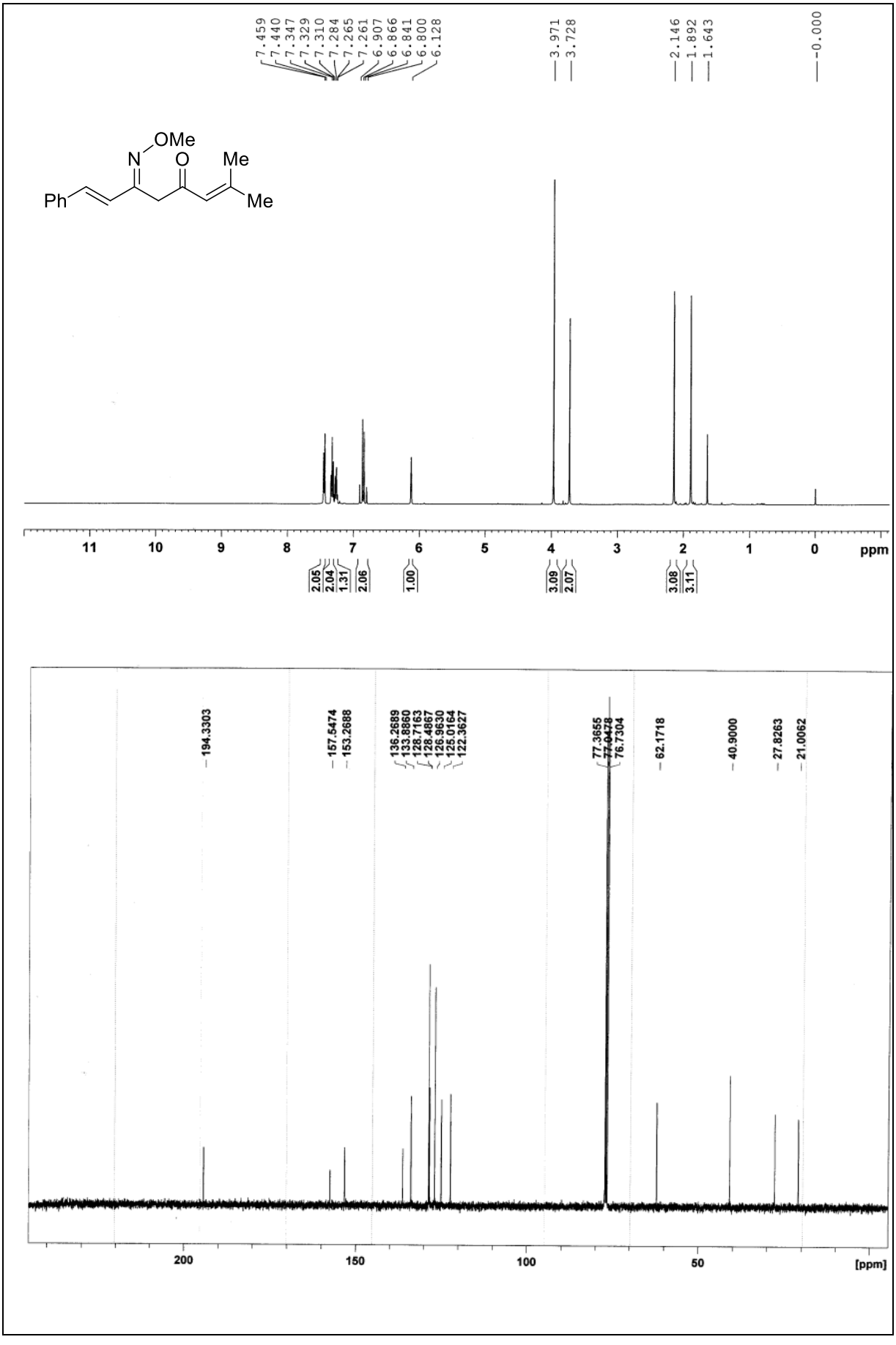
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2.146
1.892
1.643
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OMe
N
O
Me
Ph
Me
11
10
9
8
7
6
5
4
3
2
1
0
ppm
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2.04
1.31
2.06
1.00
3.09
2.07
3.08
3.11
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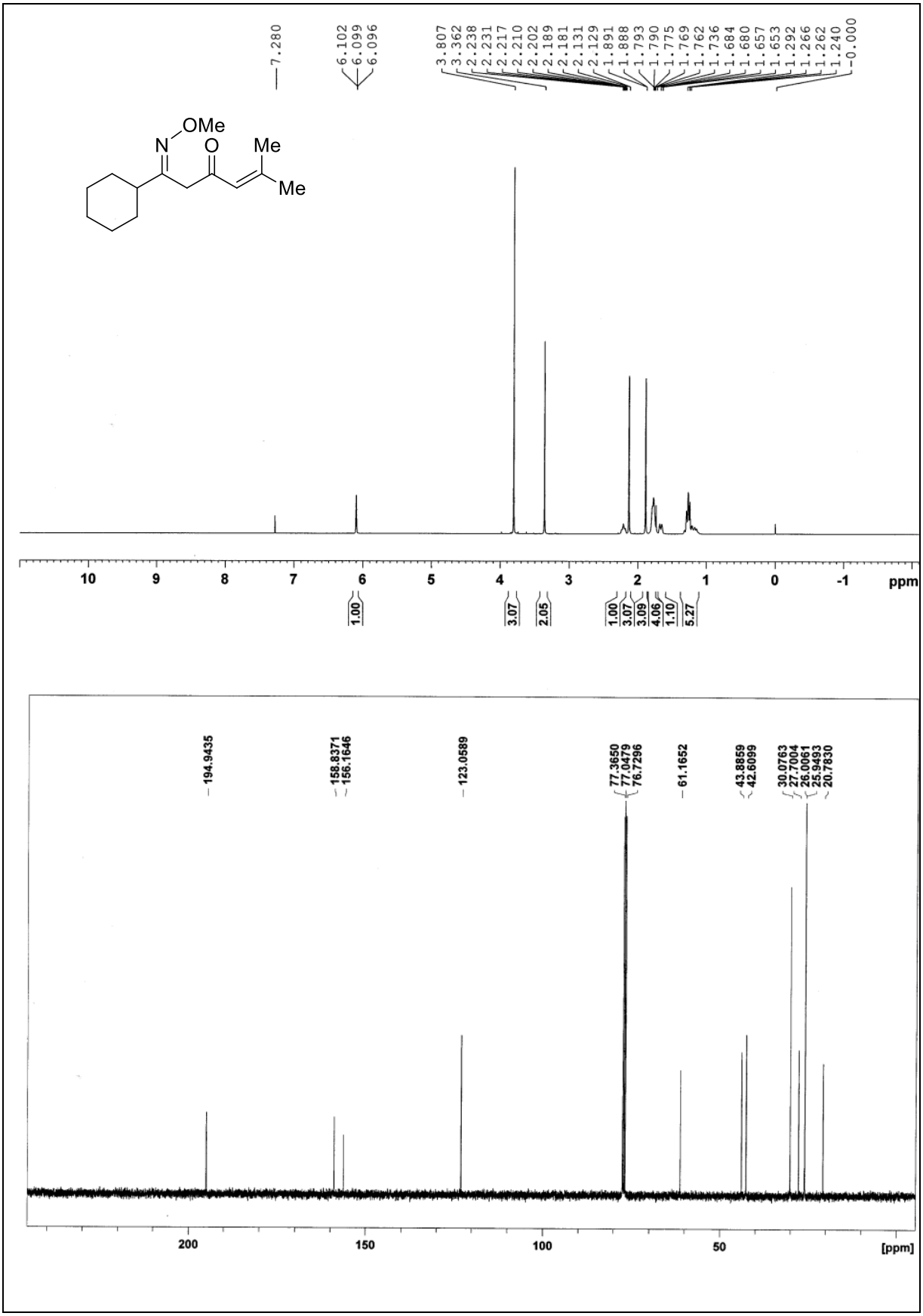
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2.129
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1.888
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1.790
1.775
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1.762
1.736
1.684
1.680
1.657
1.653
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1.266
1.262
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OMe
N
O
Me
Me
10
9
8
7
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4
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-1
ppm
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3.07
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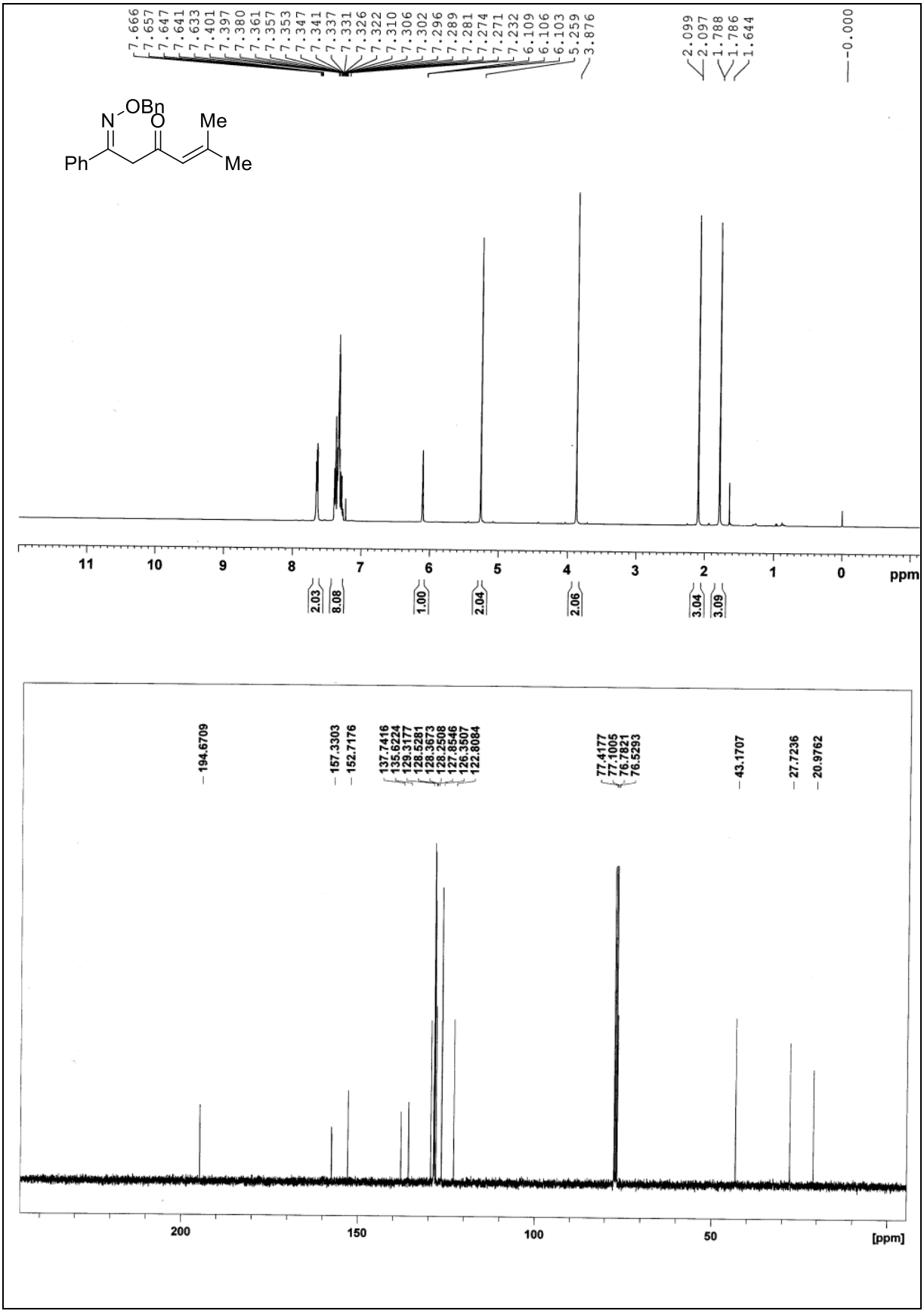
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7.322
7.310
7.306
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7.271
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OBn
N
O
Me
Ph
Me
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7
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5
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3.09
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77.1005
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76.5293
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100
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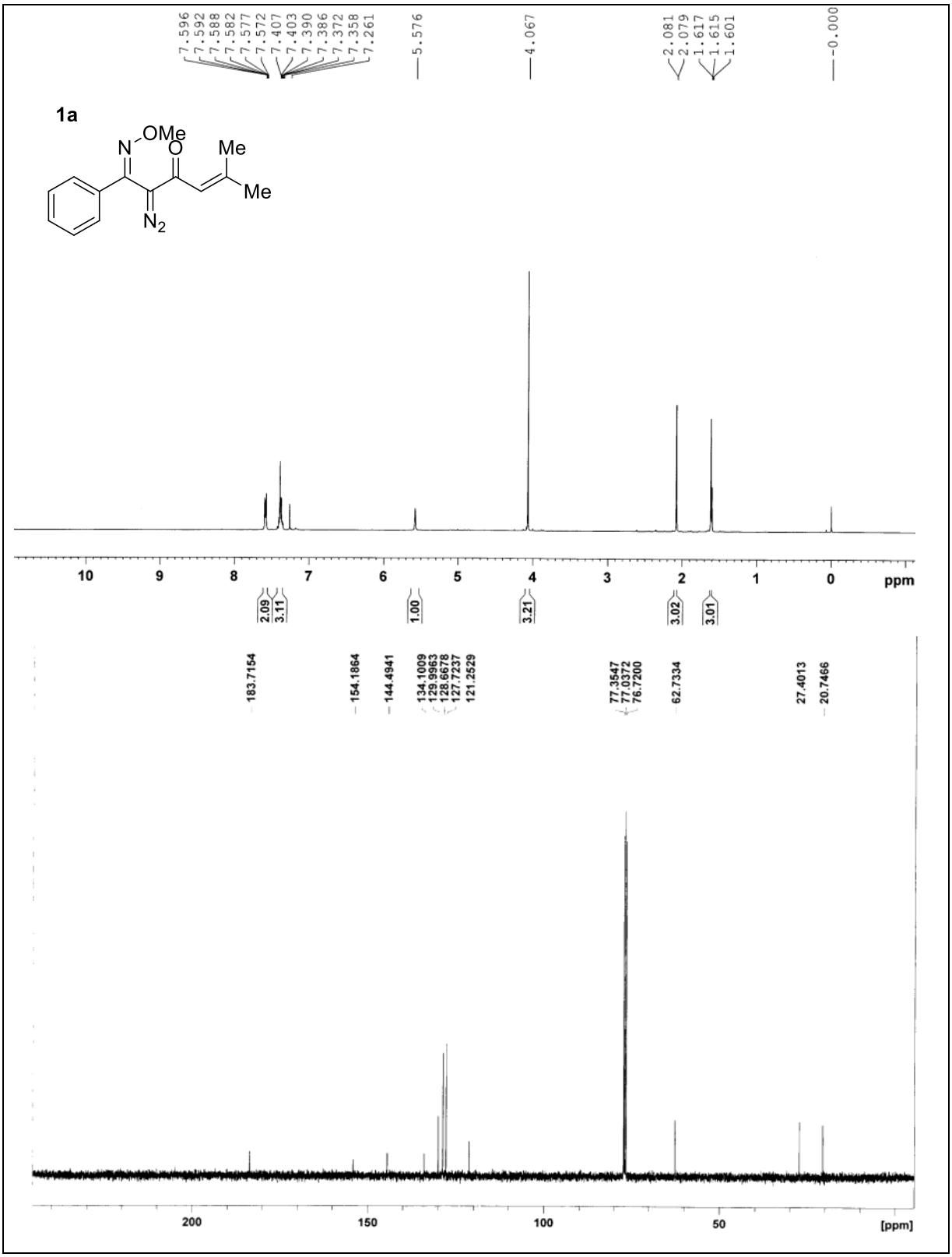
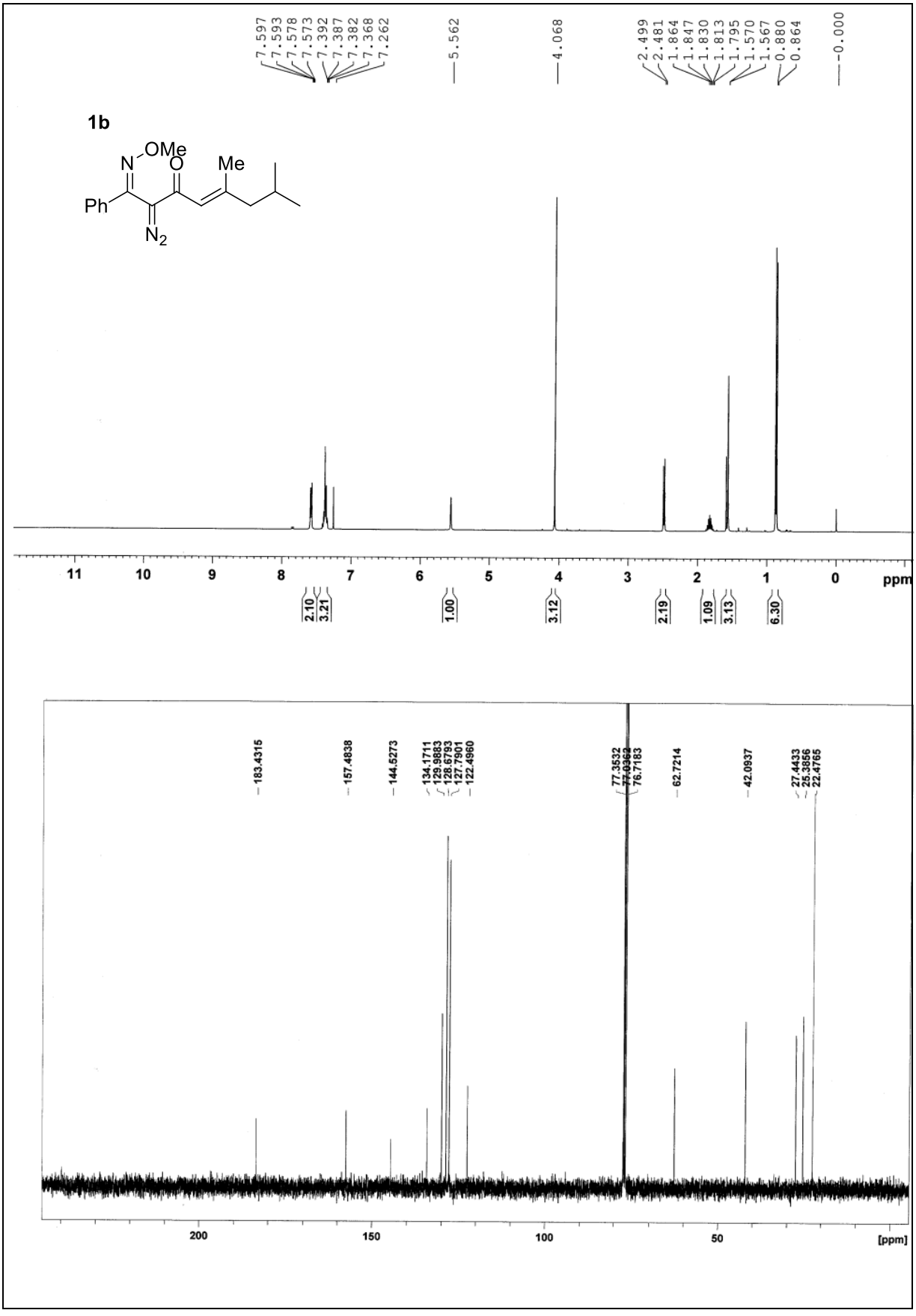
[ppm]

^{1}H and ^{13}C NMR spectra of α-diazo β-keto oxime ethers

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O
Me
Ph
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7.382
7.368
7.262
5.562
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1.830
1.813
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3.13
6.30
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128.6793
127.7901
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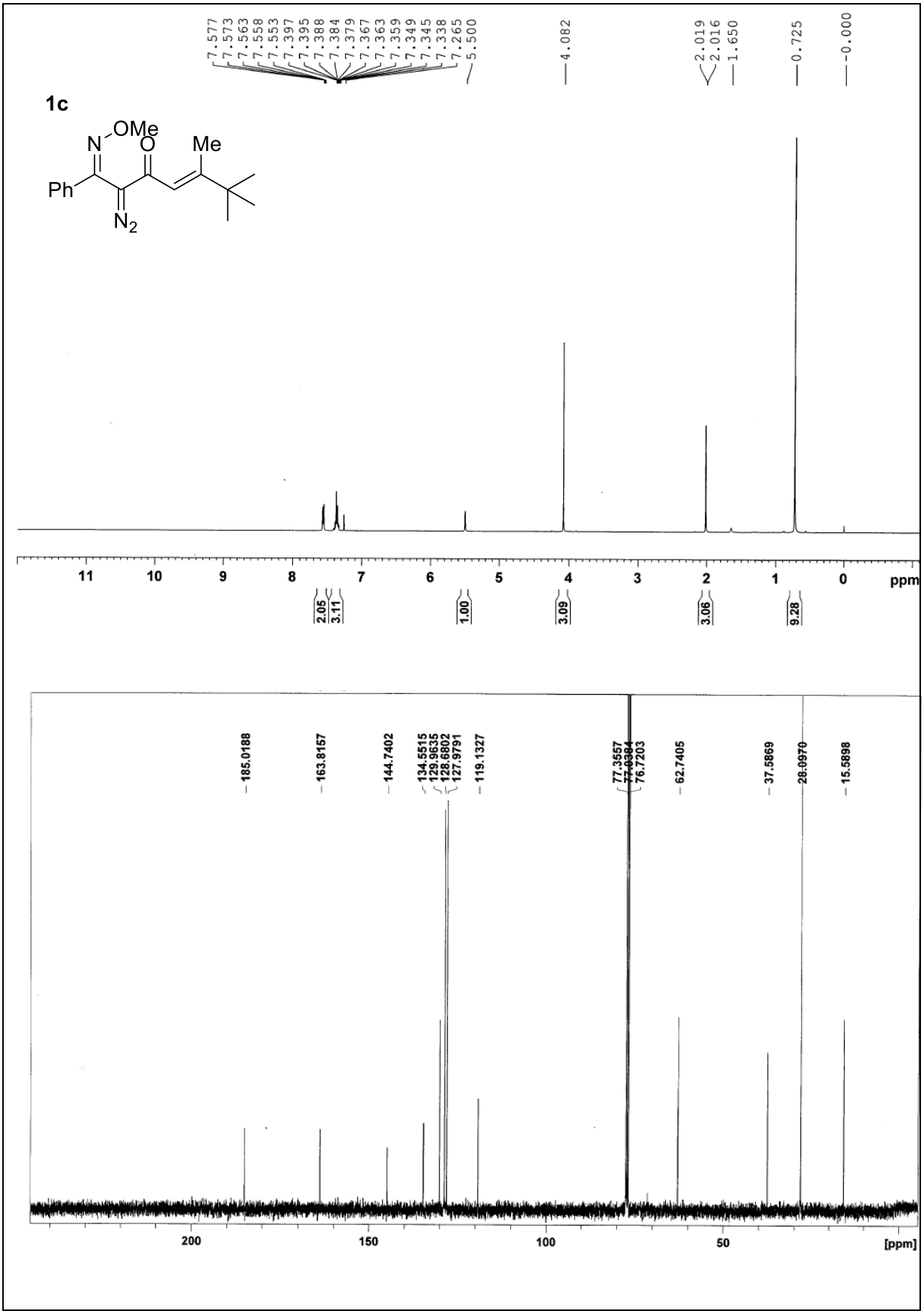
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7.367
7.363
7.359
7.349
7.345
7.338
7.265
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2.016
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-0.000
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10
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6
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0
ppm
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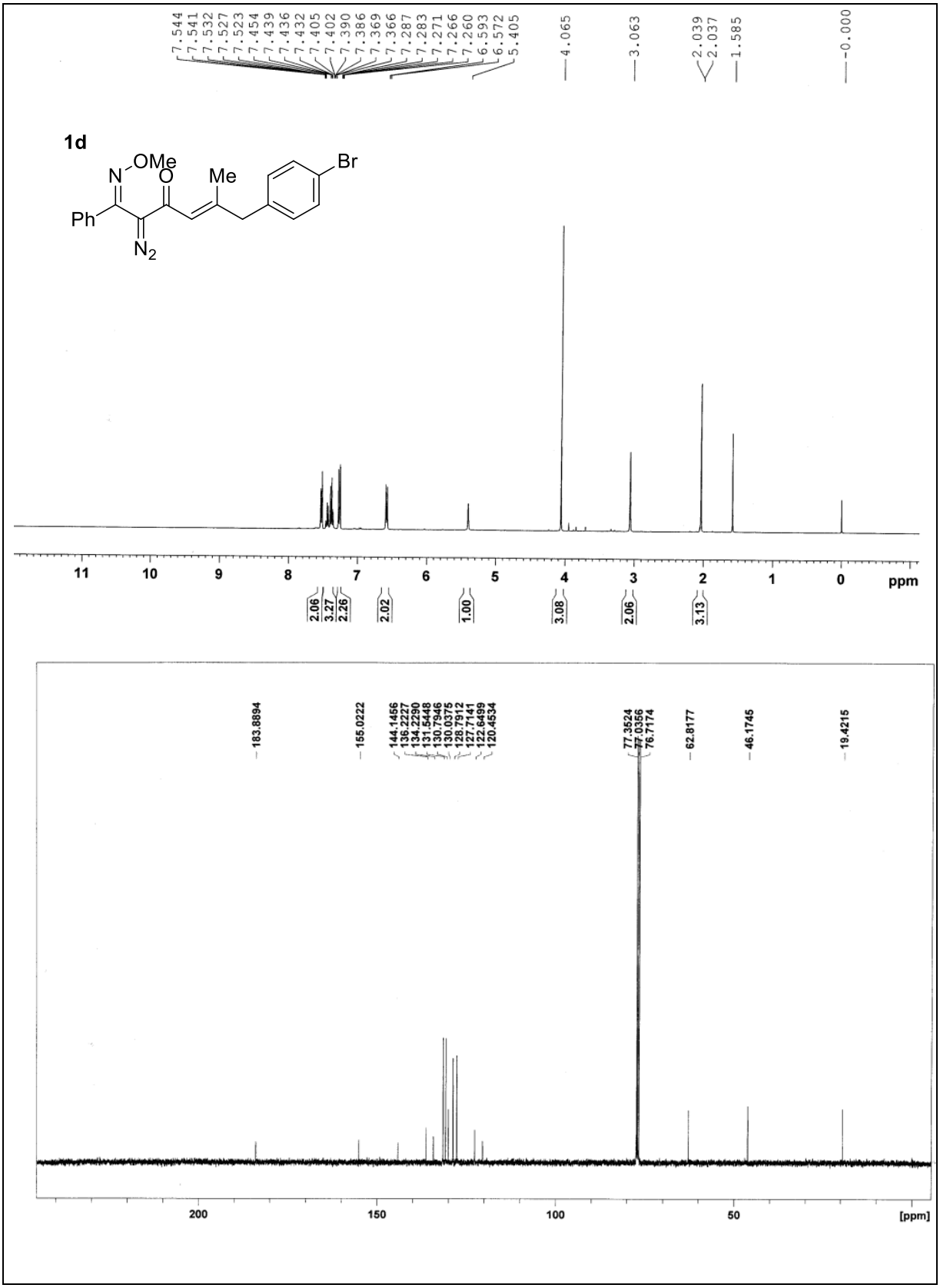
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7.366
7.287
7.283
7.271
7.266
7.260
6.593
6.572
5.405
4.065
3.063
2.039
2.037
1.585
0.000
1d
N
OMe
O
Me
Br
Ph
N2
11
10
9
8
7
6
5
4
3
2
1
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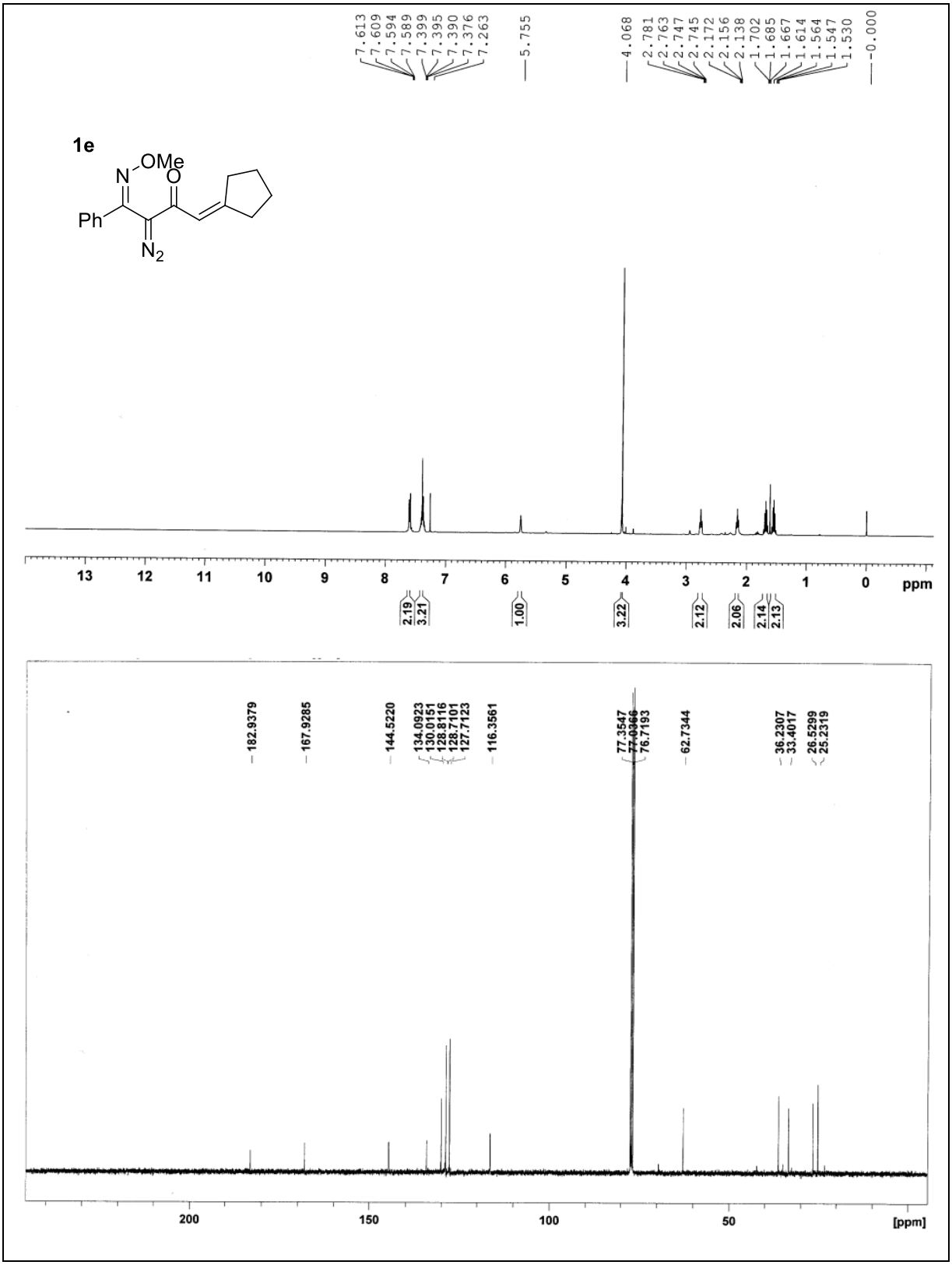
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Ph
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7.395
7.390
7.376
7.263
5.755
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1.00
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2.06
2.14
2.13
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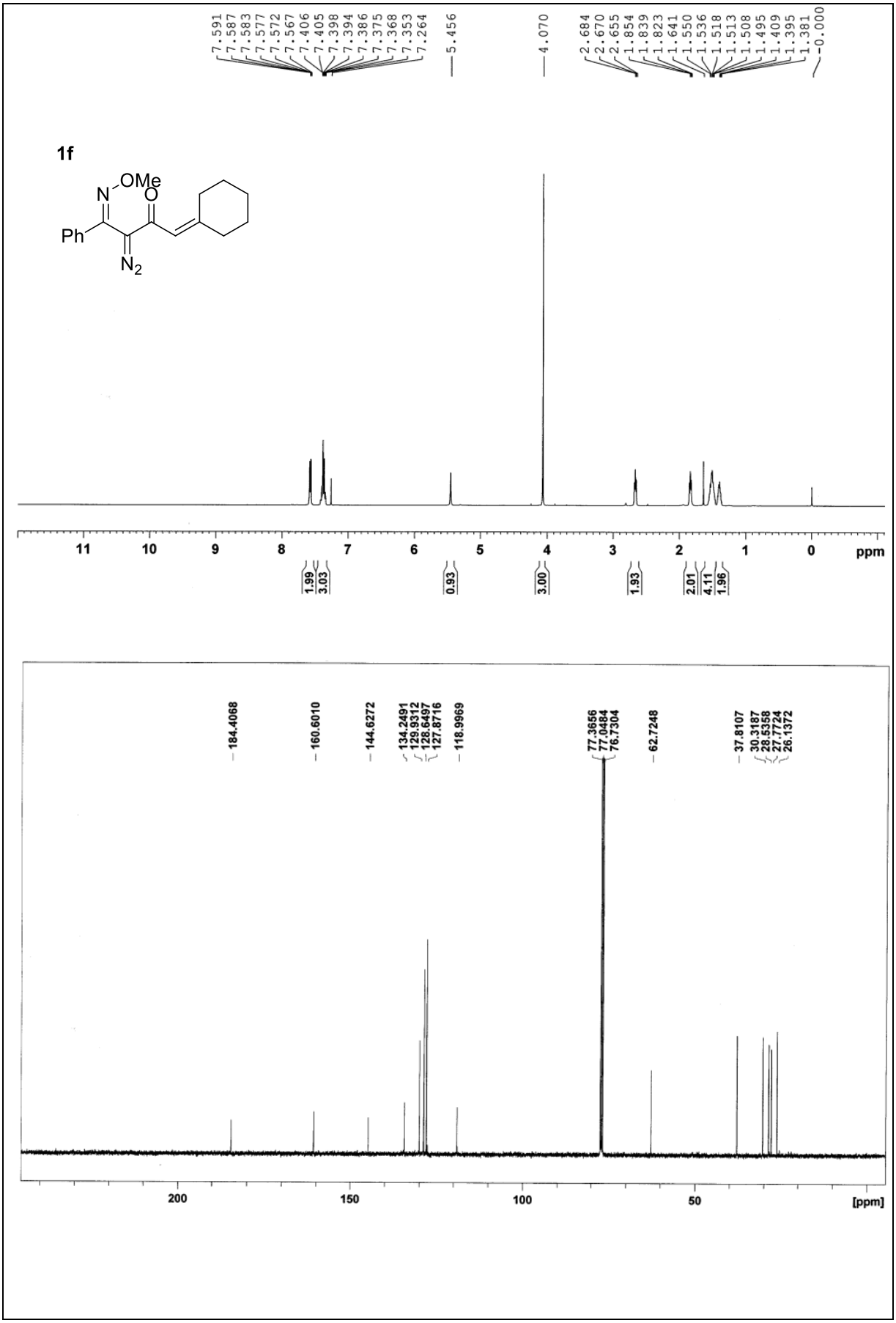
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1.823
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1.550
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O
Ph
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10
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7
6
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128.6497
127.8716
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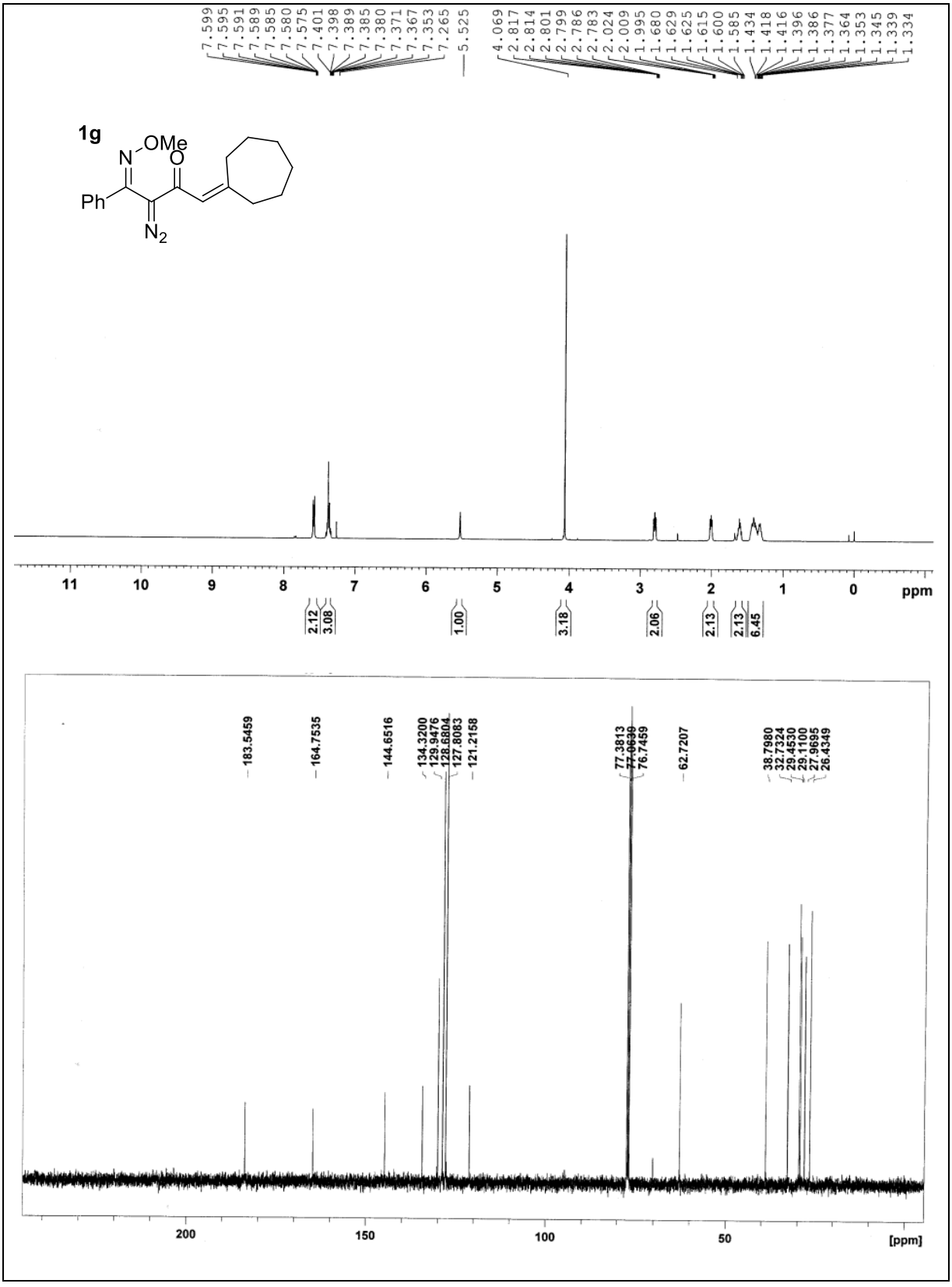
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2.009
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1.615
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1.386
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1.364
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1.339
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1g
OMe
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O
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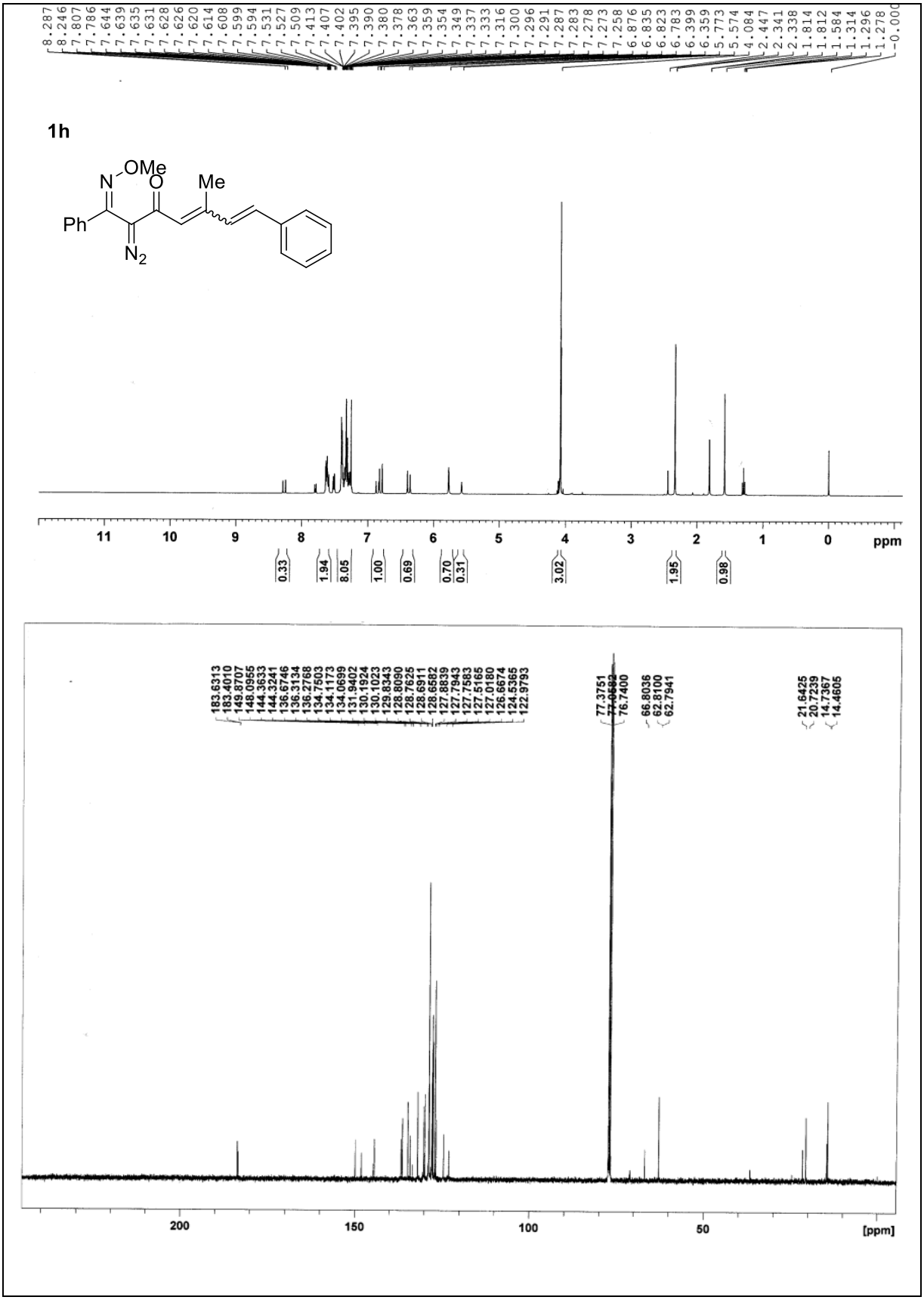
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7.626
7.620
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7.608
7.599
7.594
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7.509
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7.407
7.402
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7.380
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7.363
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7.354
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7.333
7.316
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7.291
7.287
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1.296
1.278
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OMe
N
O
Me
Ph
N2
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134.0699
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128.7625
128.6911
128.6582
127.8839
127.7943
127.7583
127.5165
127.0180
126.6674
124.5365
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77.3751
77.0682
76.7400
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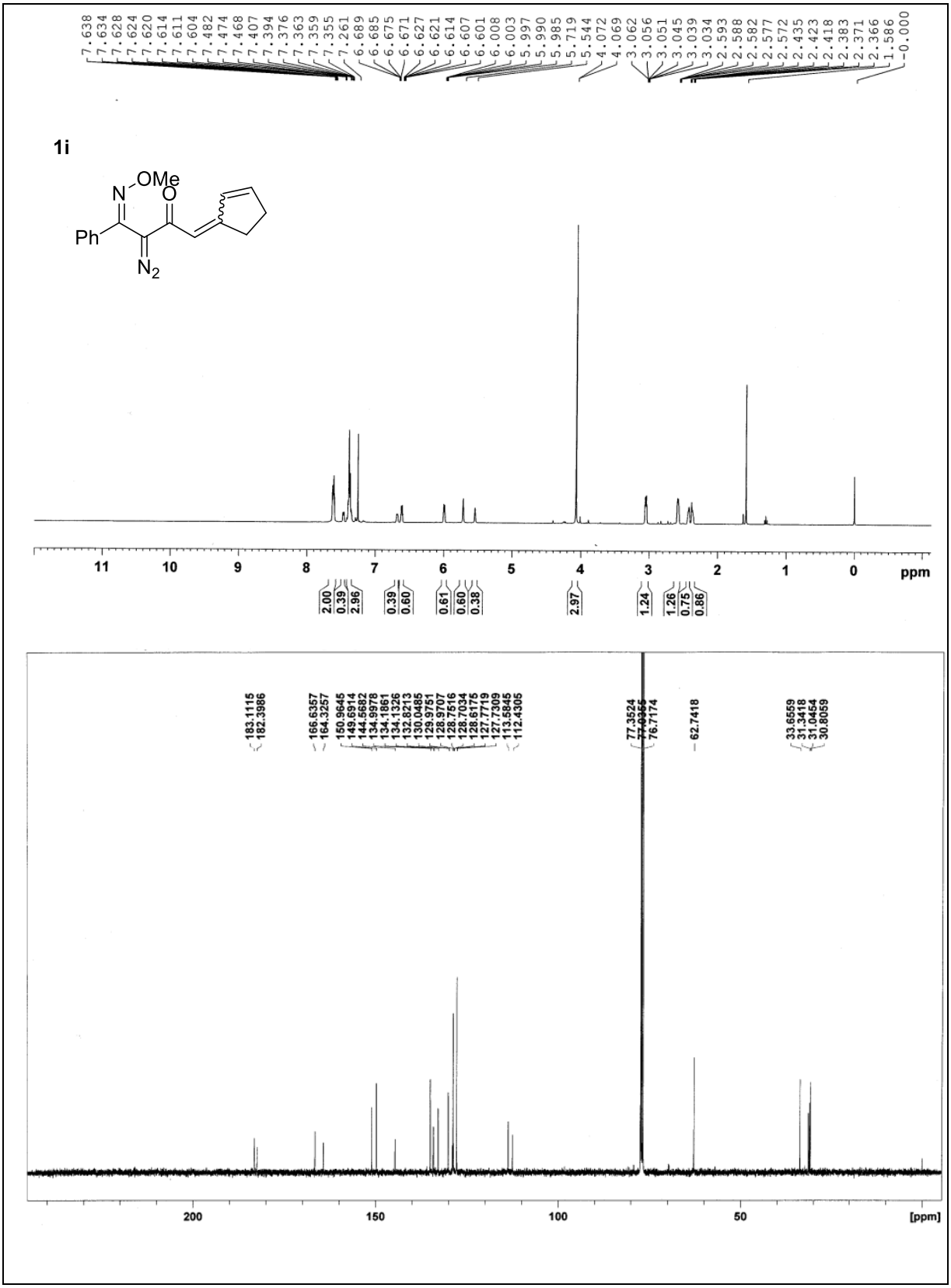
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7.363
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7.355
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6.671
6.627
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6.614
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6.601
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6.003
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5.719
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4.069
3.062
3.056
3.051
3.045
3.039
3.034
2.593
2.588
2.582
2.577
2.572
2.435
2.423
2.418
2.383
2.371
2.366
1.586
0.000
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N
OMe
O
Ph
N2
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10
9
8
7
6
5
4
3
2
1
0
ppm
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2.97
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1.26
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134.1861
134.1326
132.8213
130.0485
129.9751
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112.4305
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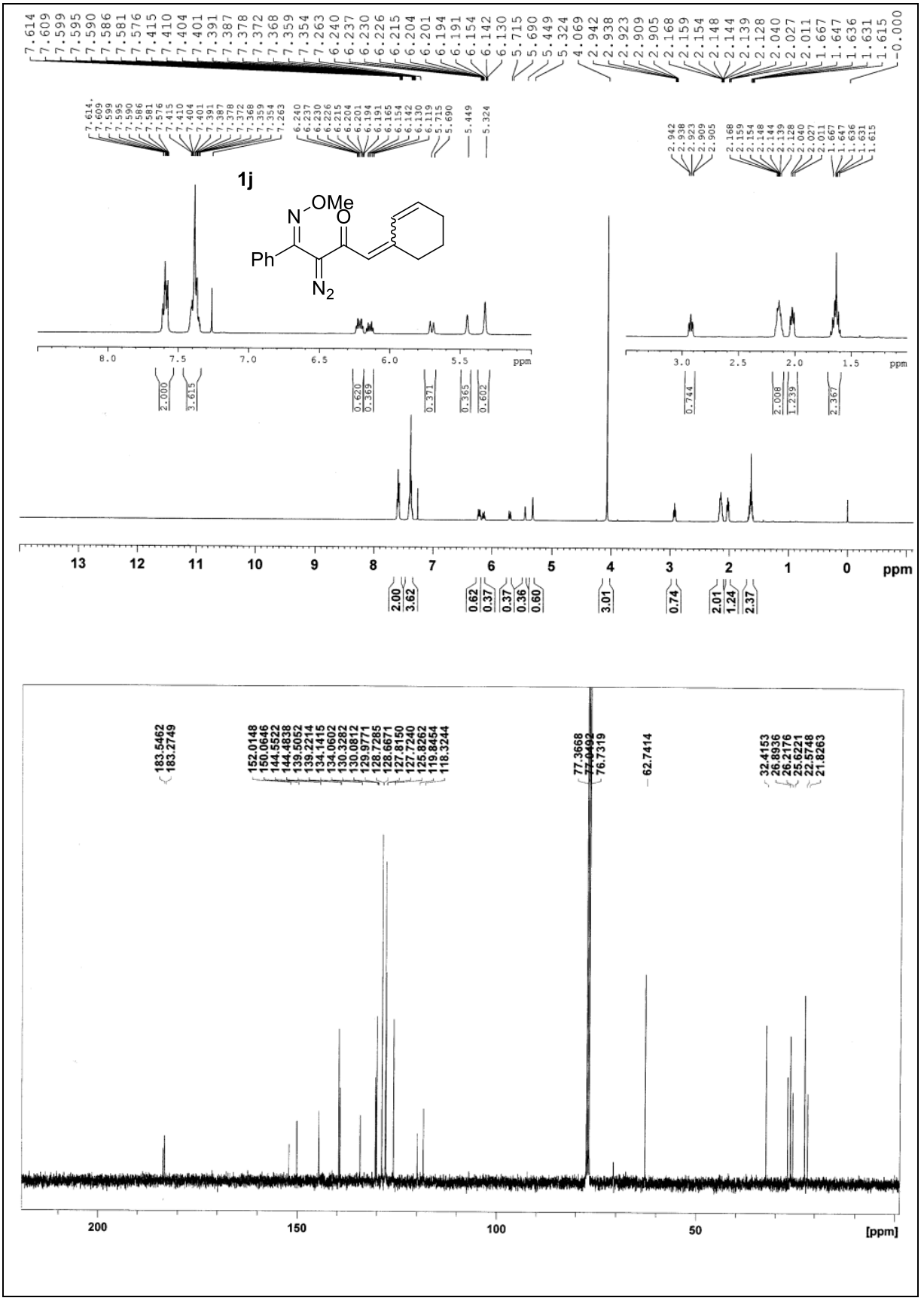
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O
Ph
N2
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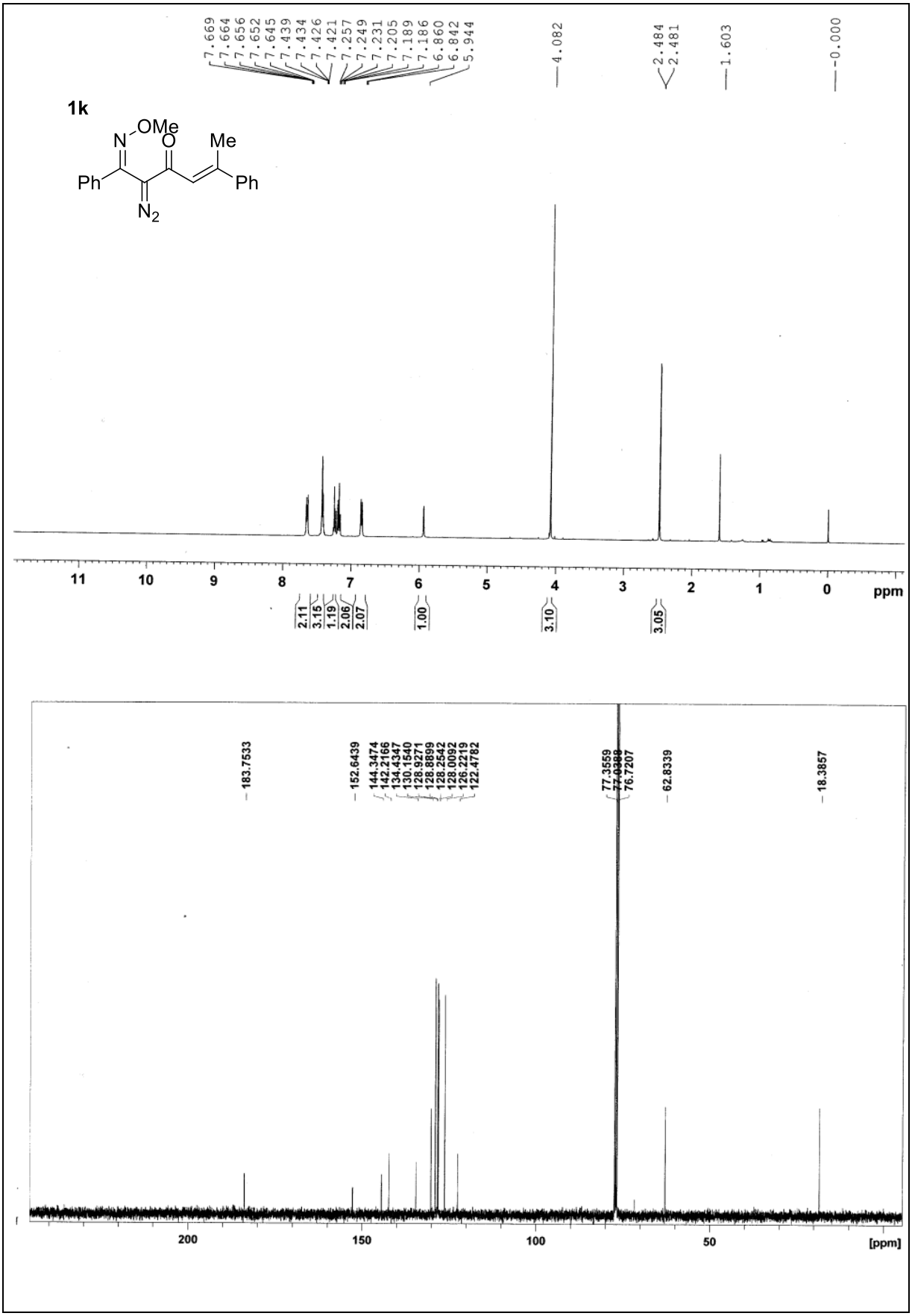
[ppm]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Ph
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7.421
7.257
7.249
7.231
7.205
7.189
7.186
6.860
6.842
5.944
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2.484
2.481
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0.000
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10
9
8
7
6
5
4
3
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0
ppm
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3.15
1.19
2.06
2.07
1.00
3.10
3.05
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144.3474
142.2166
134.4347
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128.9271
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128.2542
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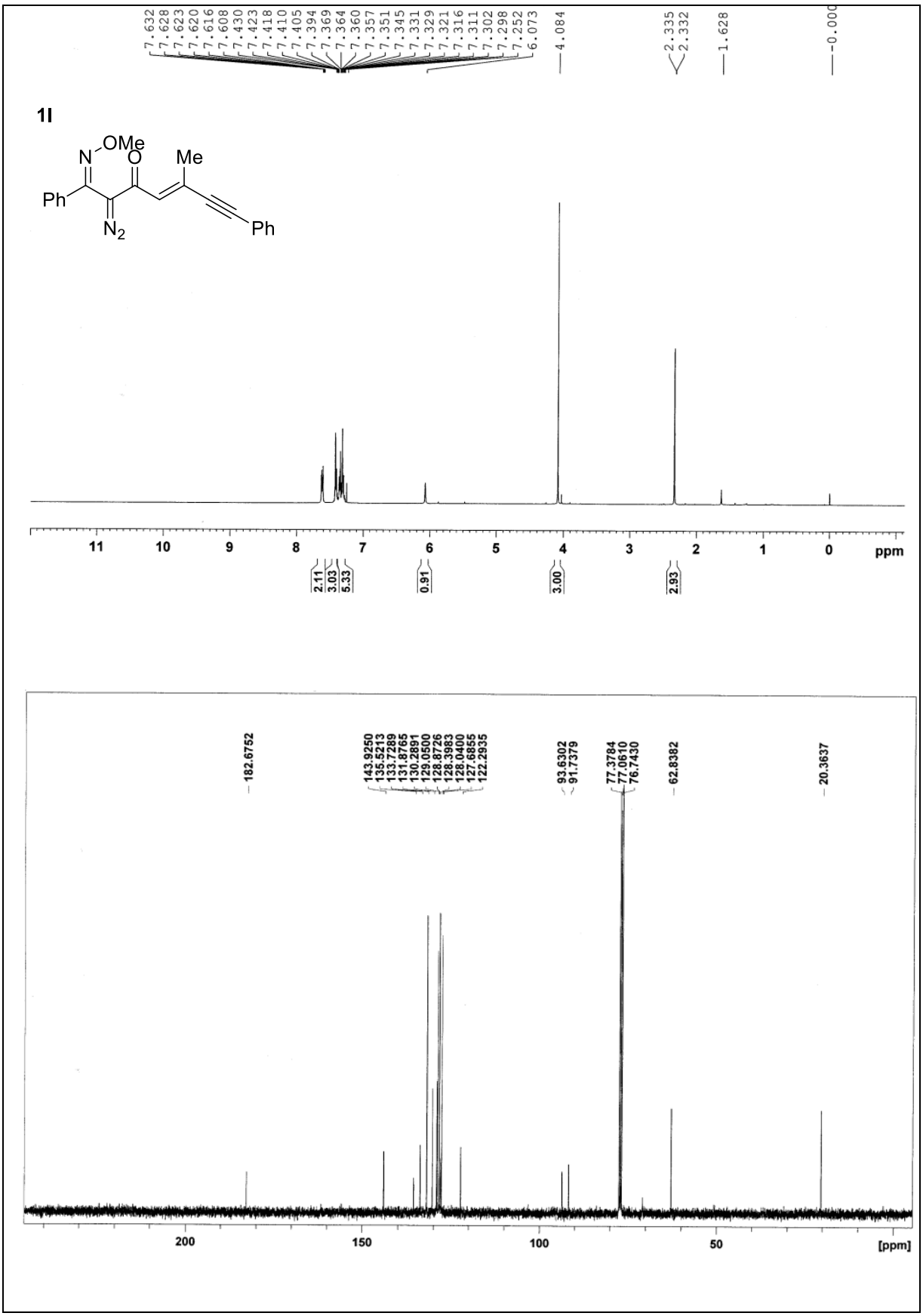
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7.423
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7.410
7.405
7.394
7.369
7.364
7.360
7.357
7.351
7.345
7.331
7.329
7.321
7.316
7.311
7.302
7.298
7.252
6.073
4.084
2.335
2.332
1.628
0.000
1l
OMe
N
O
Me
Ph
N2
Ph
11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.11
3.03
5.33
0.91
3.00
2.93
182.6752
143.9250
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131.8765
130.2891
129.0500
128.8726
128.3983
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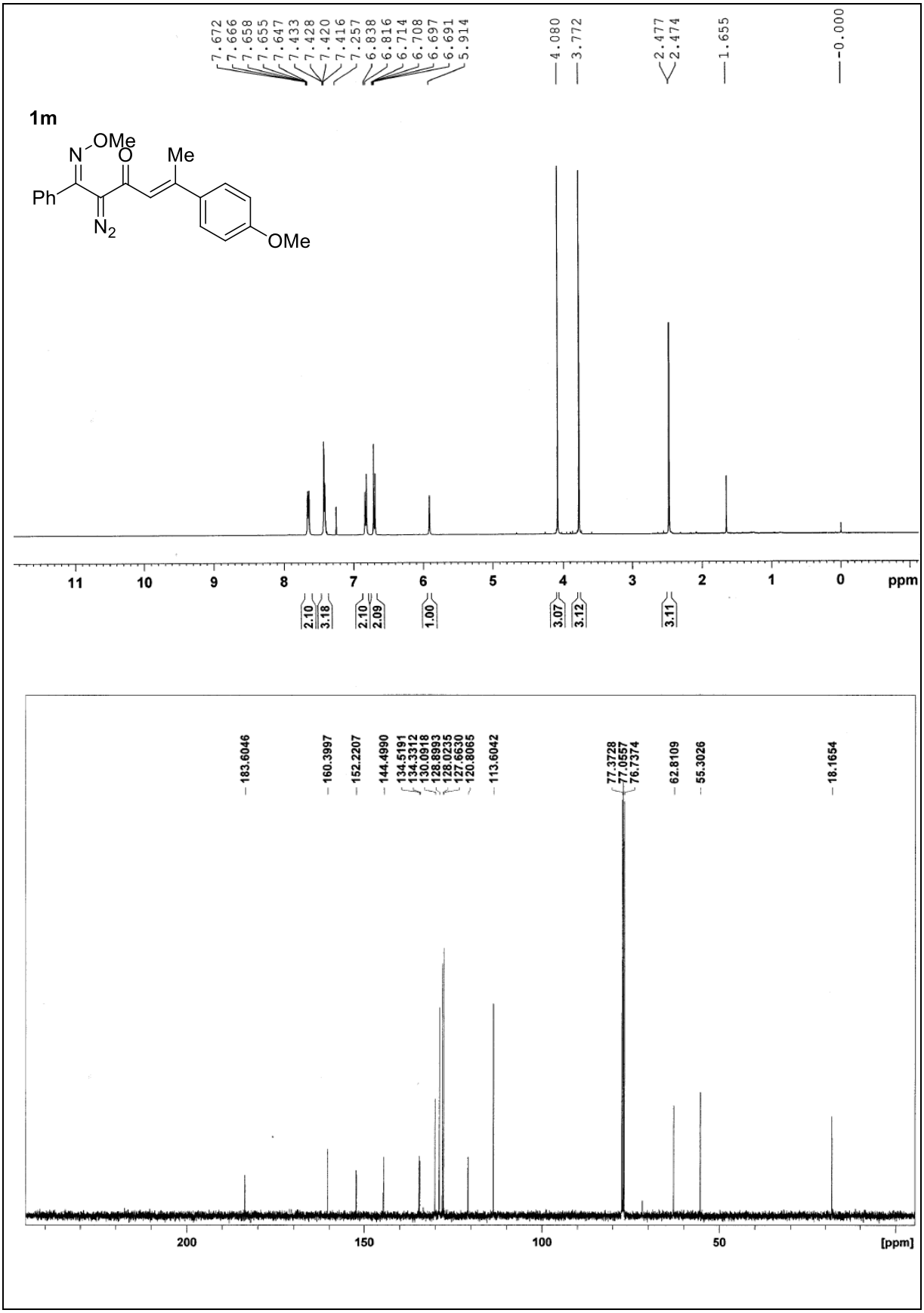
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7.416
7.257
6.838
6.816
6.714
6.708
6.697
6.691
5.914
4.080
3.772
2.477
2.474
1.655
0.000
OMe
N
O
Me
Ph
N2
OMe
11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.10
3.18
2.10
2.09
1.00
3.07
3.12
3.11
183.6046
160.3997
152.2207
144.4990
134.5191
134.3312
130.0918
128.8993
128.0235
127.6630
120.8065
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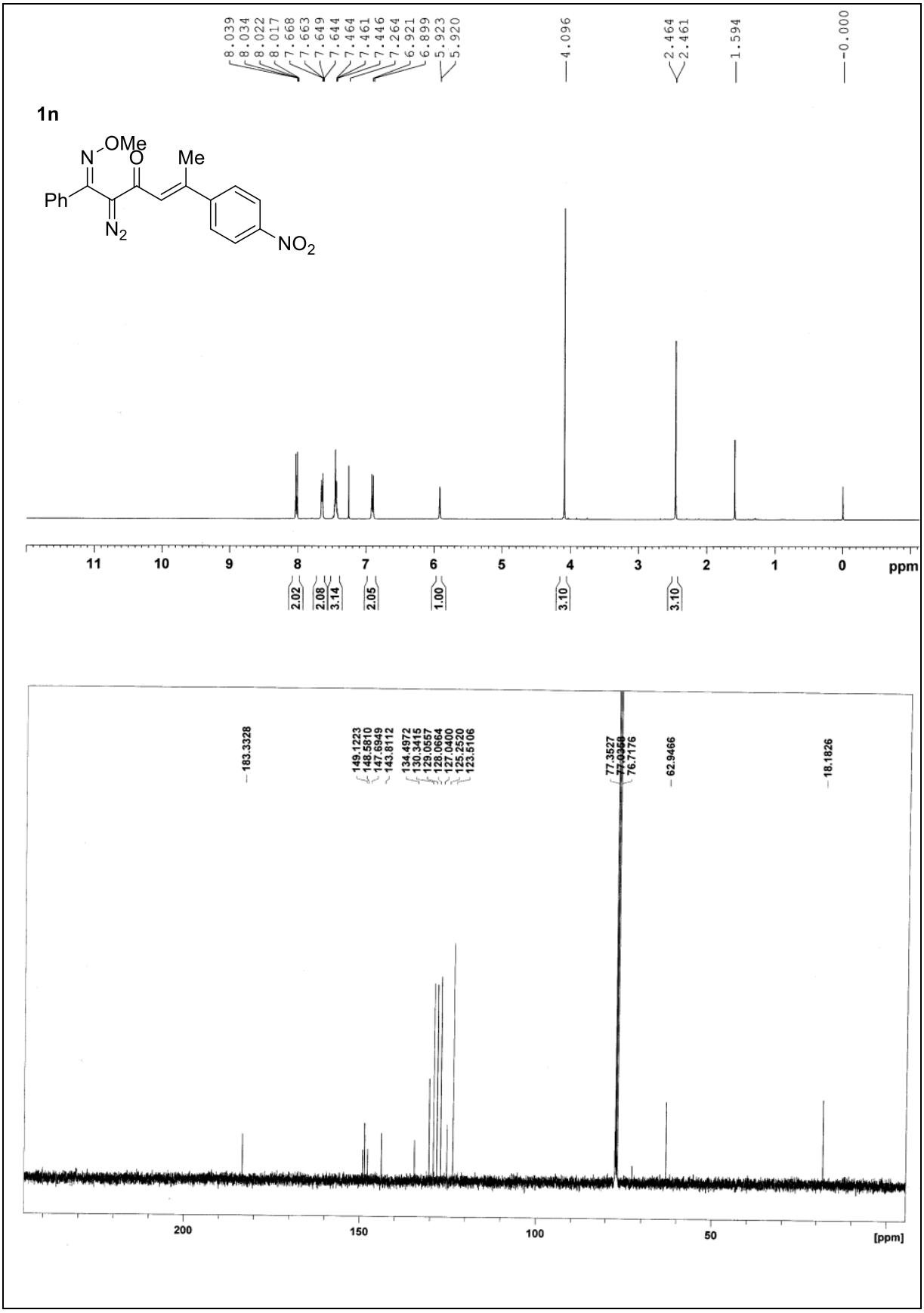
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8.034
8.022
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7.663
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7.644
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7.461
7.446
7.264
6.921
6.899
5.923
5.920
4.096
2.464
2.461
1.594
0.000
1n
OMe
N
O
Me
Ph
N2
NO2
11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.02
2.08
3.14
2.05
1.00
3.10
3.10
183.3328
149.1223
148.5810
147.6949
143.8112
134.4972
130.3415
129.0557
128.0664
127.0400
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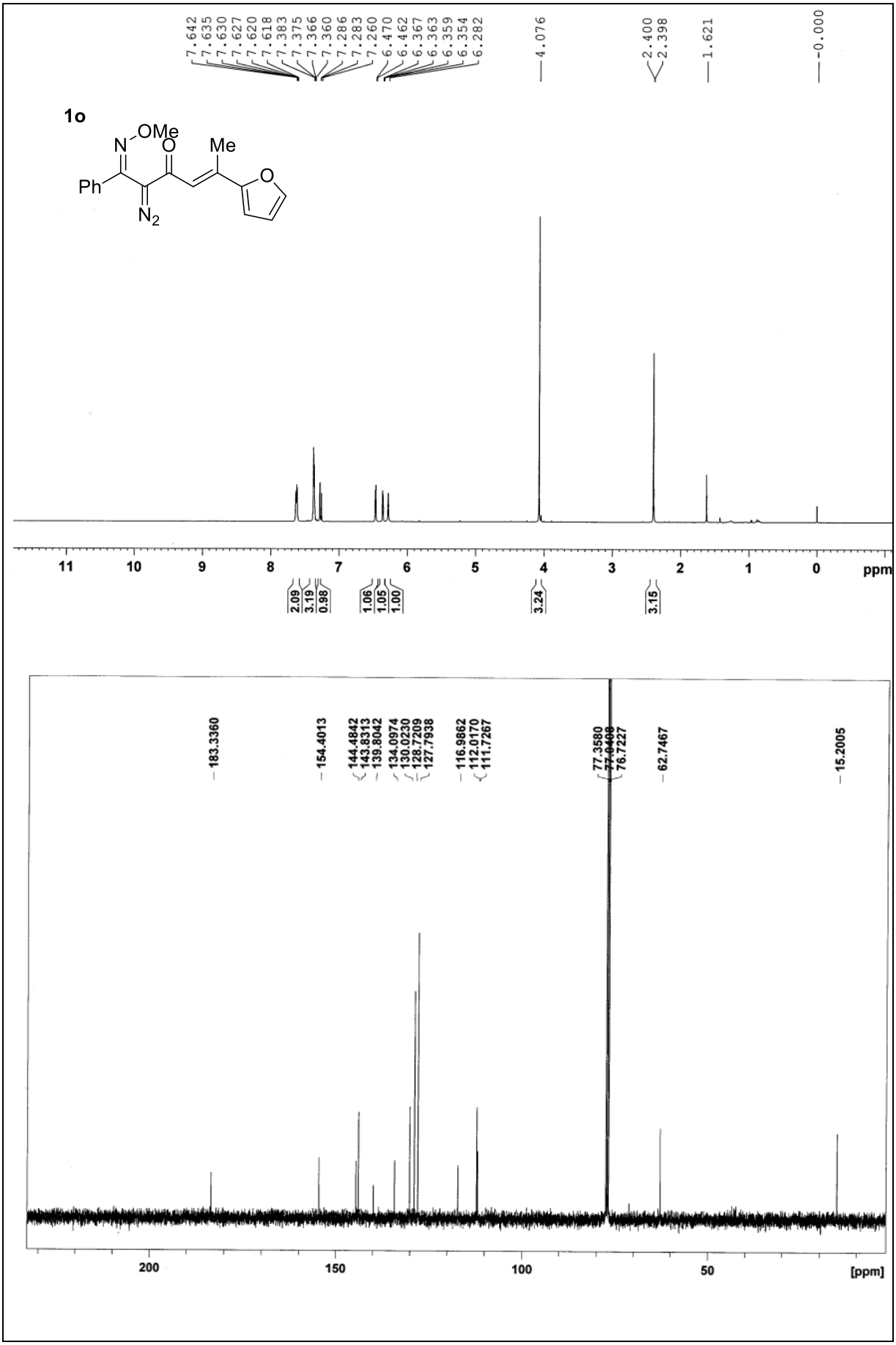
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7.360
7.286
7.283
7.260
6.470
6.462
6.367
6.363
6.359
6.354
6.282
4.076
2.400
2.398
1.621
0.000
1o
N
OMe
O
Me
Ph
O
N2
11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.09
3.19
0.98
1.06
1.05
1.00
3.24
3.15
183.3360
154.4013
144.4842
143.8313
139.8042
134.0974
130.0230
128.7209
127.7938
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77.3580
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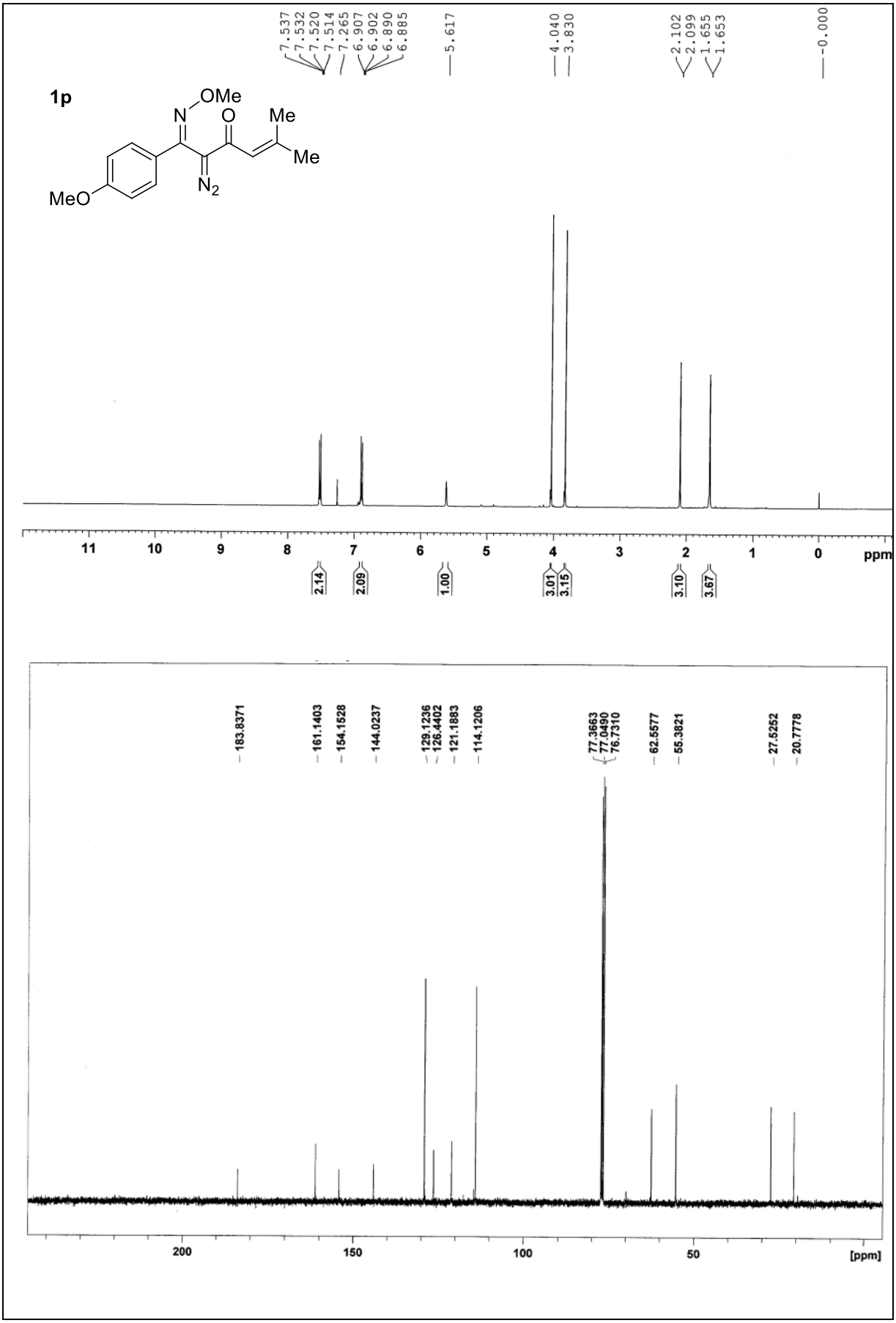
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O
Me
Me
N2
MeO
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7.265
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6.902
6.890
6.885
5.617
4.040
3.830
2.102
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1.655
1.653
0.000
11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.14
2.09
1.00
3.01
3.15
3.10
3.67
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161.1403
154.1528
144.0237
129.1236
126.4402
121.1883
114.1206
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20.7778
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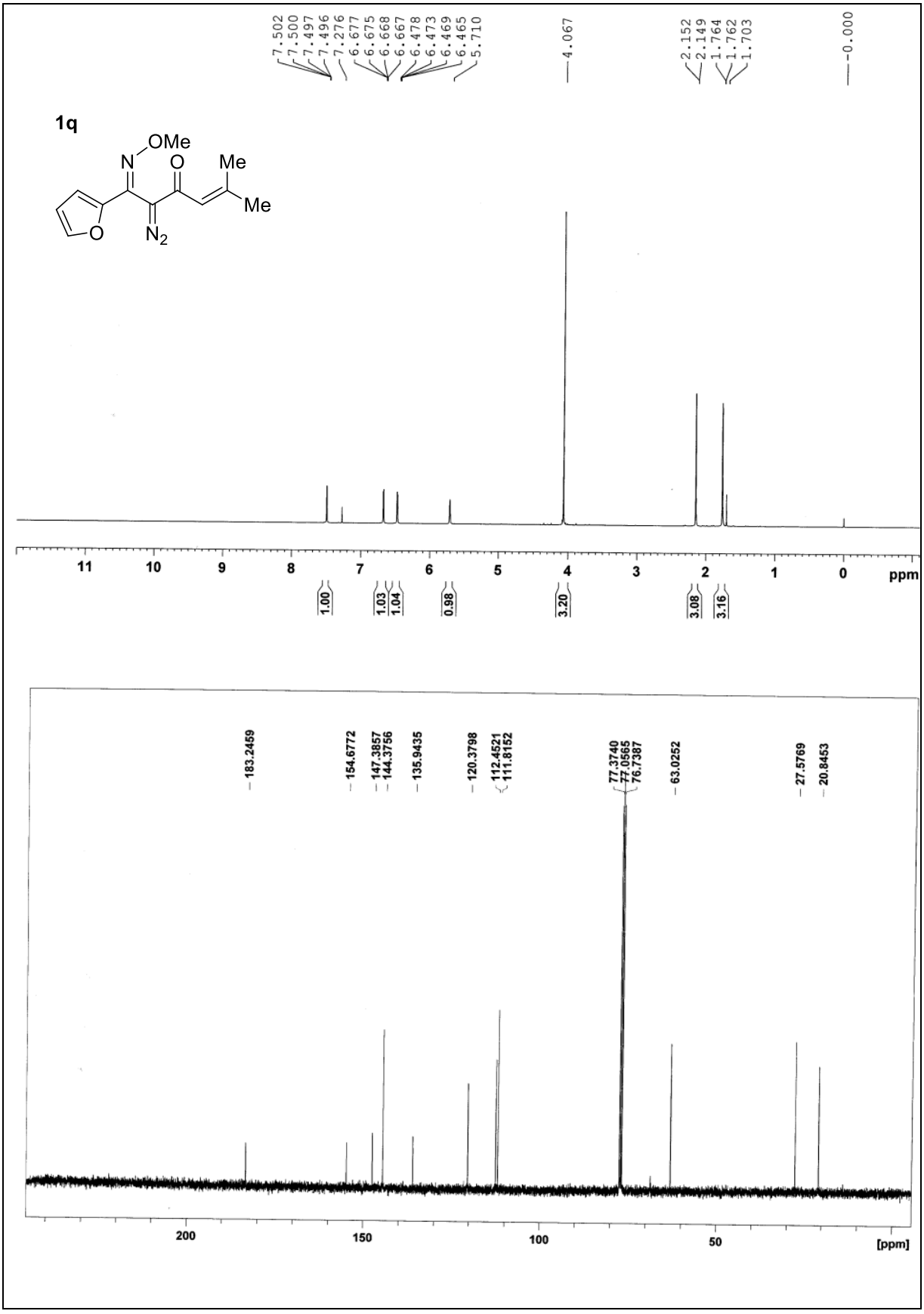
150
100
50
[ppm]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O
Me
Me
N2
O
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6.667
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6.469
6.465
5.710
4.067
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2.149
1.764
1.762
1.703
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ppm
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1.04
0.98
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3.08
3.16
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144.3756
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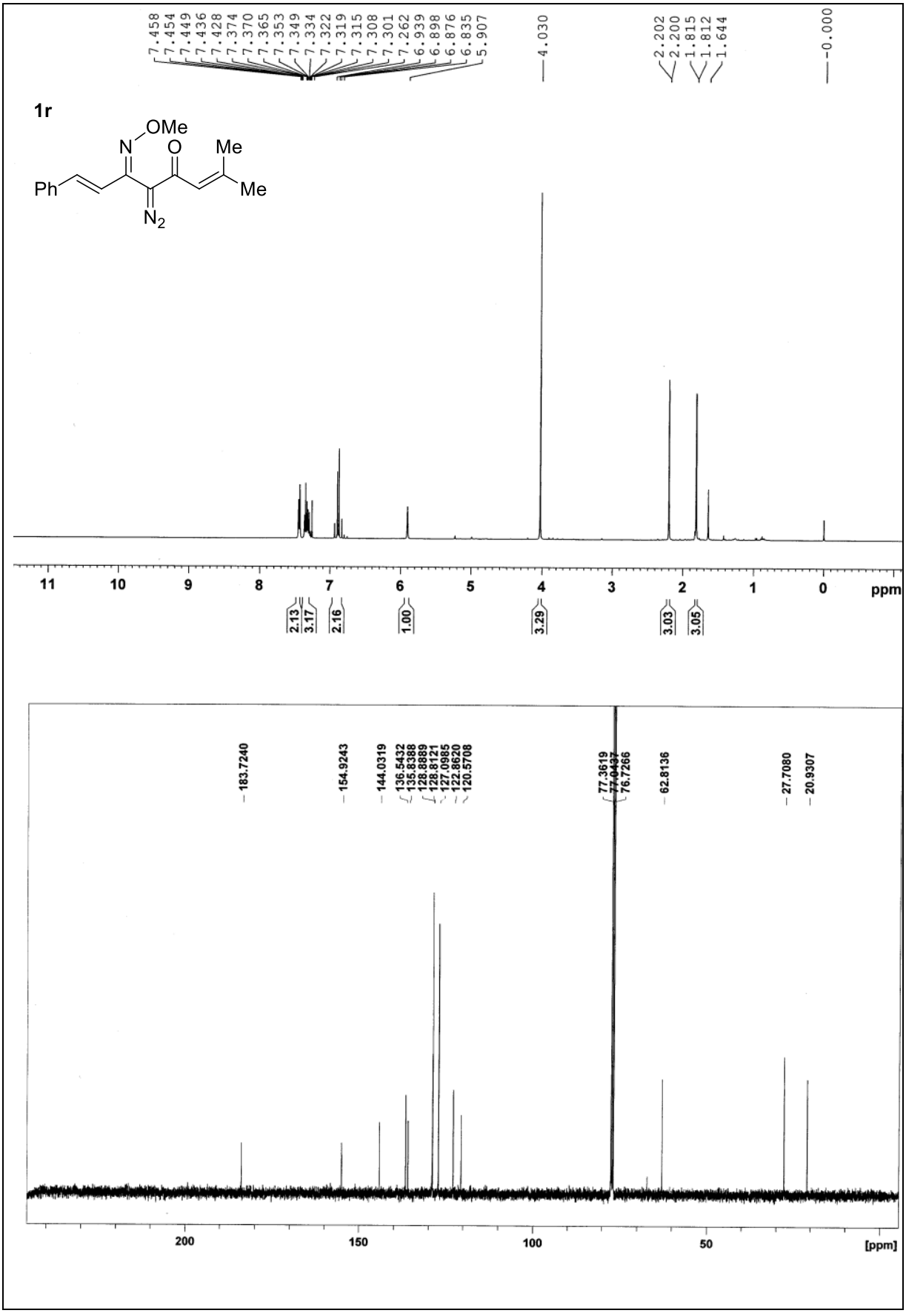
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7.365
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7.349
7.334
7.322
7.319
7.315
7.308
7.301
7.262
6.939
6.898
6.876
6.835
5.907
4.030
2.202
2.200
1.815
1.812
1.644
0.000
1r
OMe
N
O
Me
Ph
Me
N2
11
10
9
8
7
6
5
4
3
2
1
0
ppm
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3.17
2.16
1.00
3.29
3.03
3.05
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144.0319
136.5432
135.8388
128.8889
128.8121
127.0985
122.8620
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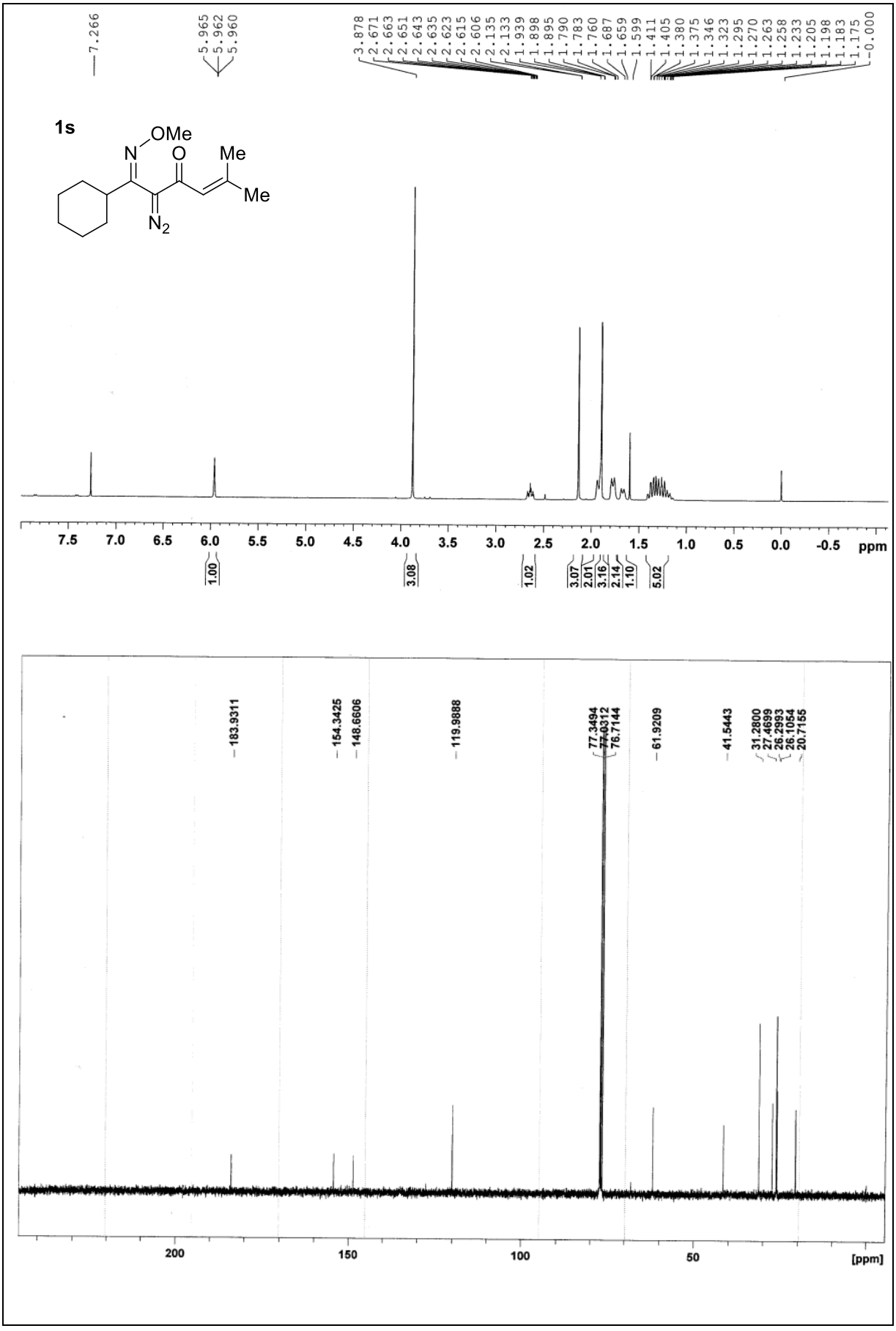
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O
Me
Me
N2
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2.643
2.635
2.623
2.615
2.606
2.135
2.133
1.939
1.898
1.895
1.790
1.783
1.760
1.687
1.659
1.599
1.411
1.405
1.380
1.375
1.346
1.323
1.295
1.270
1.263
1.258
1.233
1.205
1.198
1.183
1.175
0.000
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 ppm
1.00
3.08
1.02
3.07
2.01
3.16
2.14
1.10
5.02
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148.6606
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77.0312
76.7144
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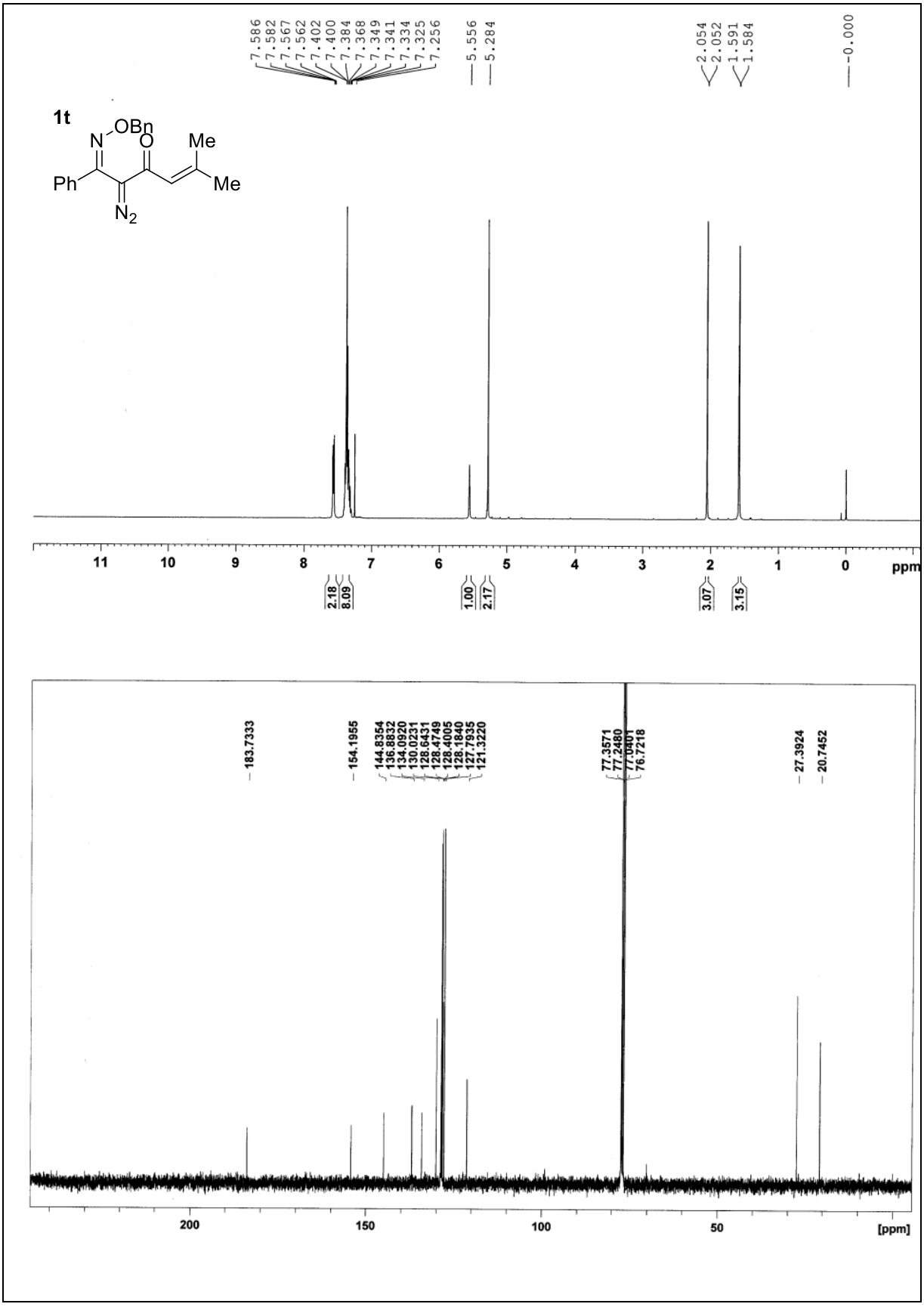
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OBn
N
O
Me
Ph
Me
N2
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7.349
7.341
7.334
7.325
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0.000
11
10
9
8
7
6
5
4
3
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1
0
ppm
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2.17
3.07
3.15
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134.0920
130.0231
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128.4749
128.4005
128.1840
127.7935
121.3220
77.3571
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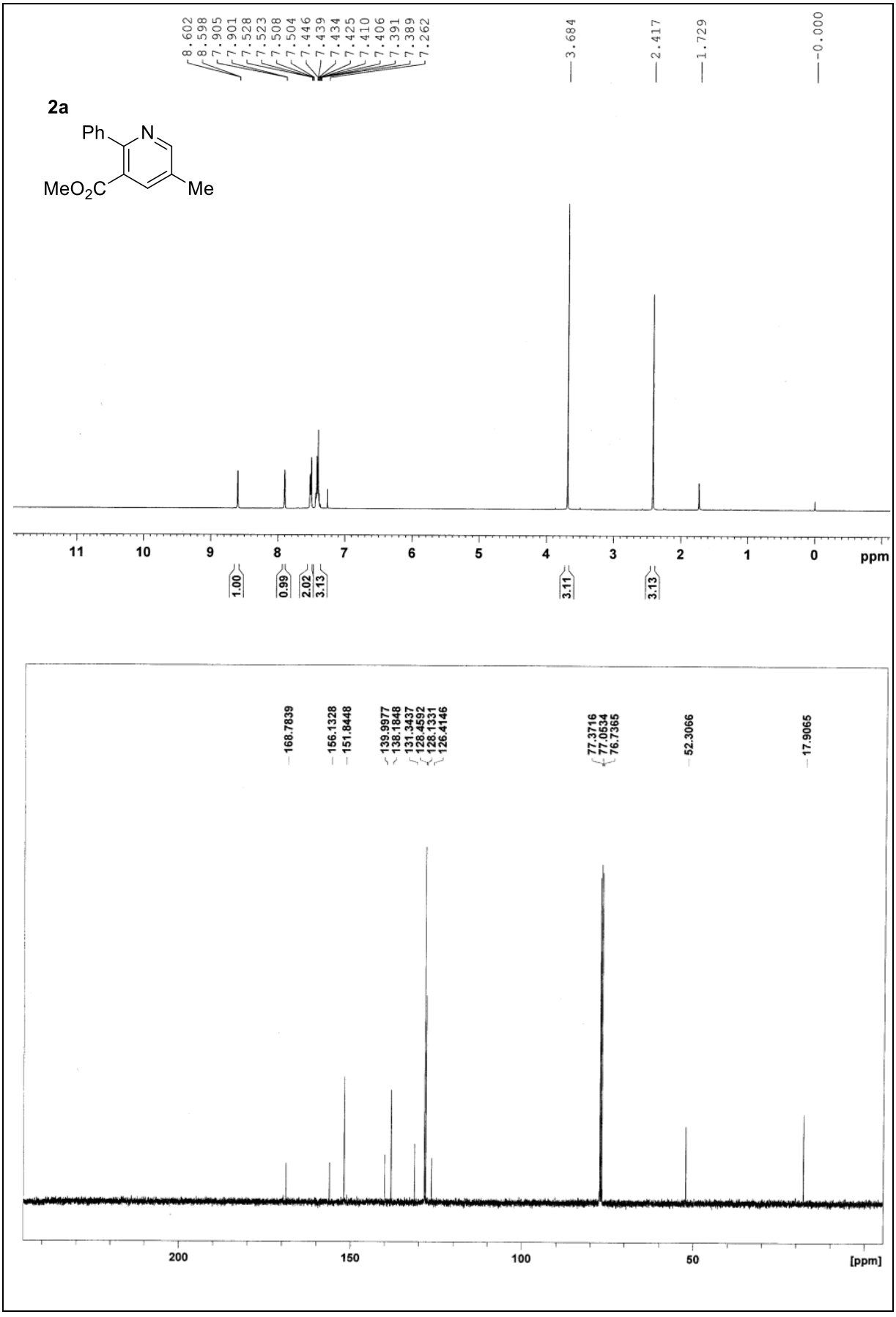
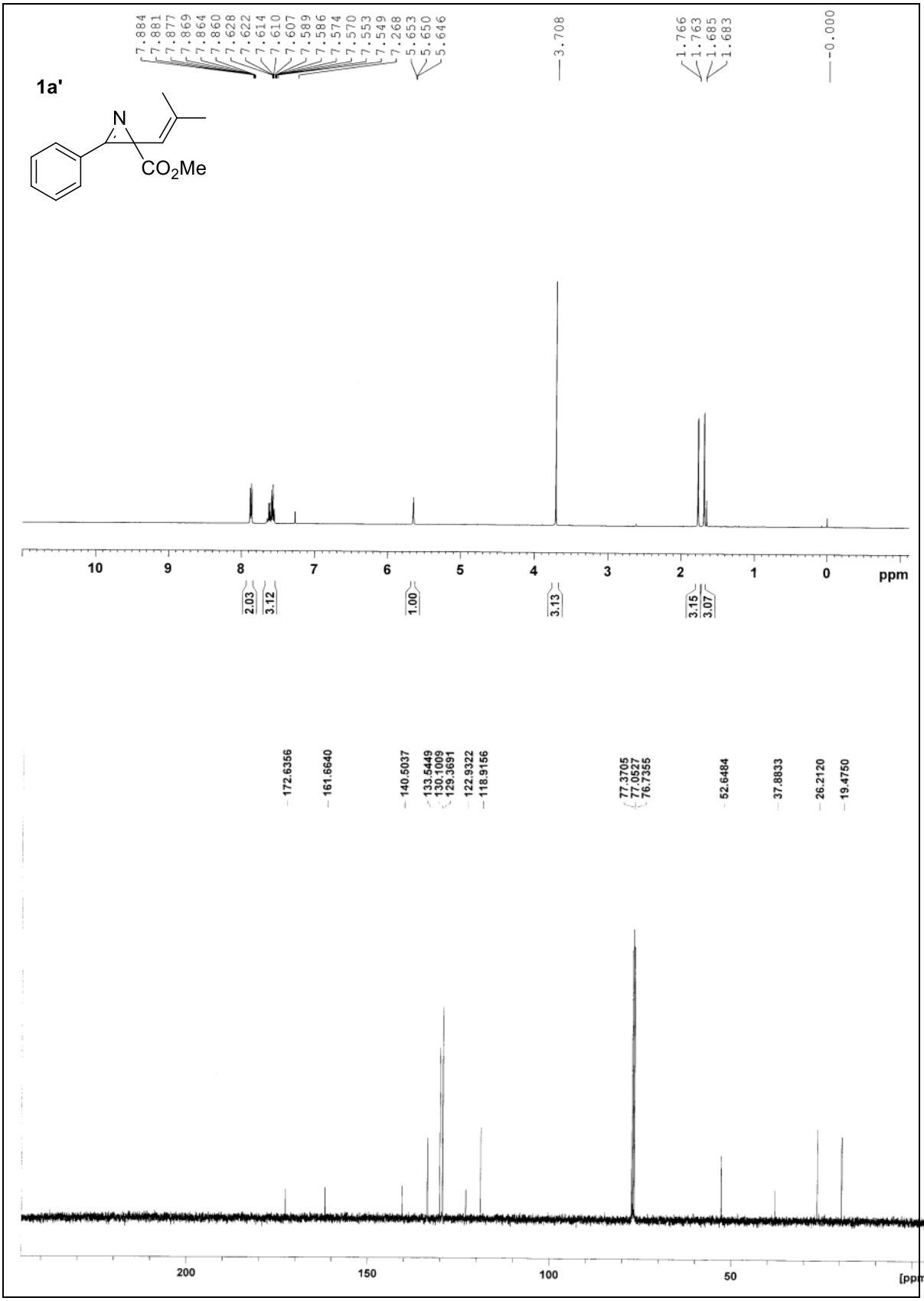
150
100
50
[ppm]

^{1}H and ^{13}C NMR spectra of pyridines

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7.622
7.614
7.610
7.607
7.589
7.586
7.574
7.570
7.553
7.549
7.268
5.653
5.650
5.646
3.708
1.766
1.763
1.685
1.683
-0.000
10
9
8
7
6
5
4
3
2
1
0
ppm
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3.12
1.00
3.13
3.15
3.07
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140.5037
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130.1009
129.3691
122.9322
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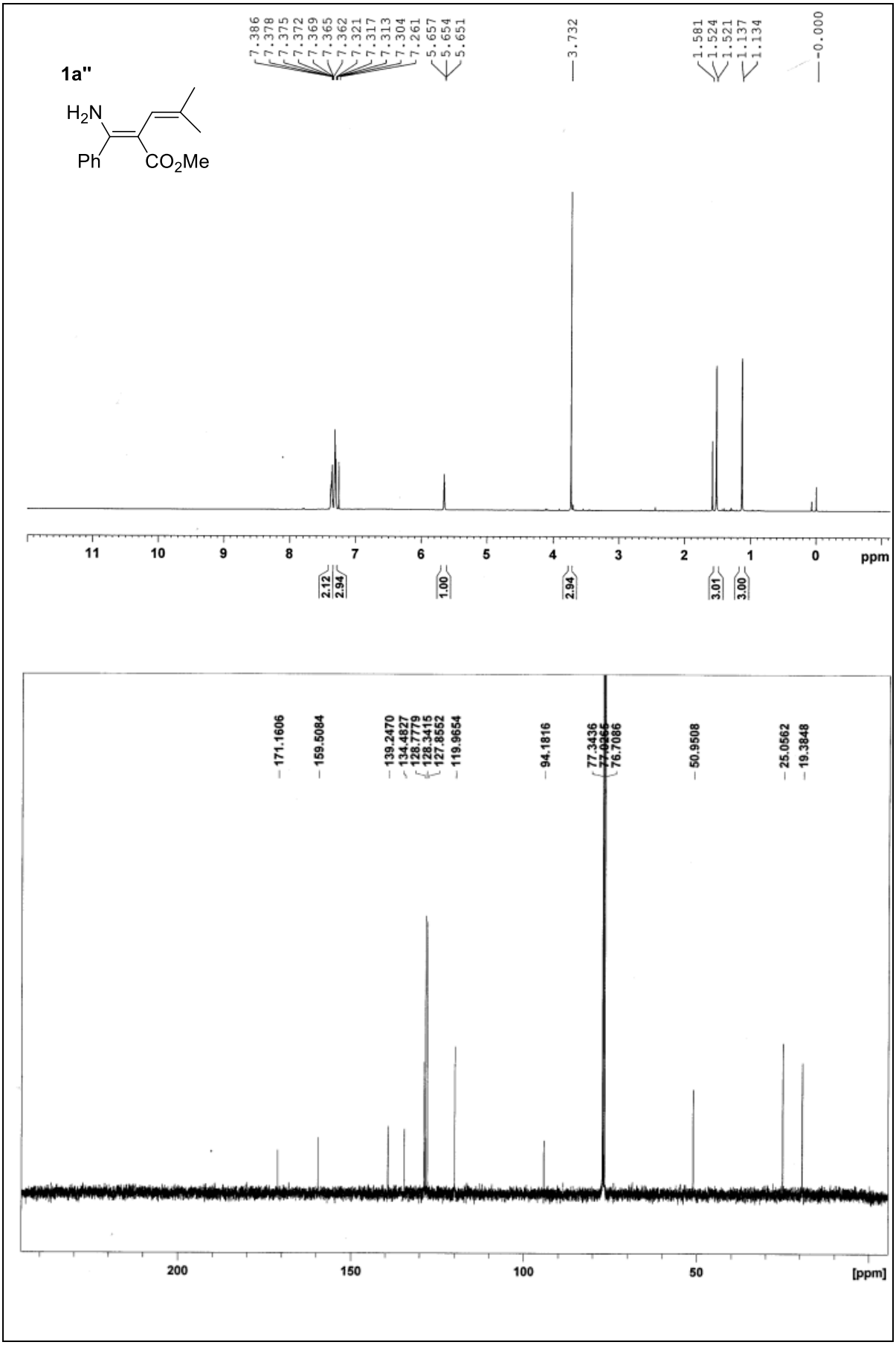
[ppm]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Ph
CO2Me
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7.365
7.362
7.321
7.317
7.313
7.304
7.261
5.657
5.654
5.651
3.732
1.581
1.524
1.521
1.137
1.134
-0.000
11
10
9
8
7
6
5
4
3
2
1
0
ppm
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2.94
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3.00
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128.3415
127.8552
119.9654
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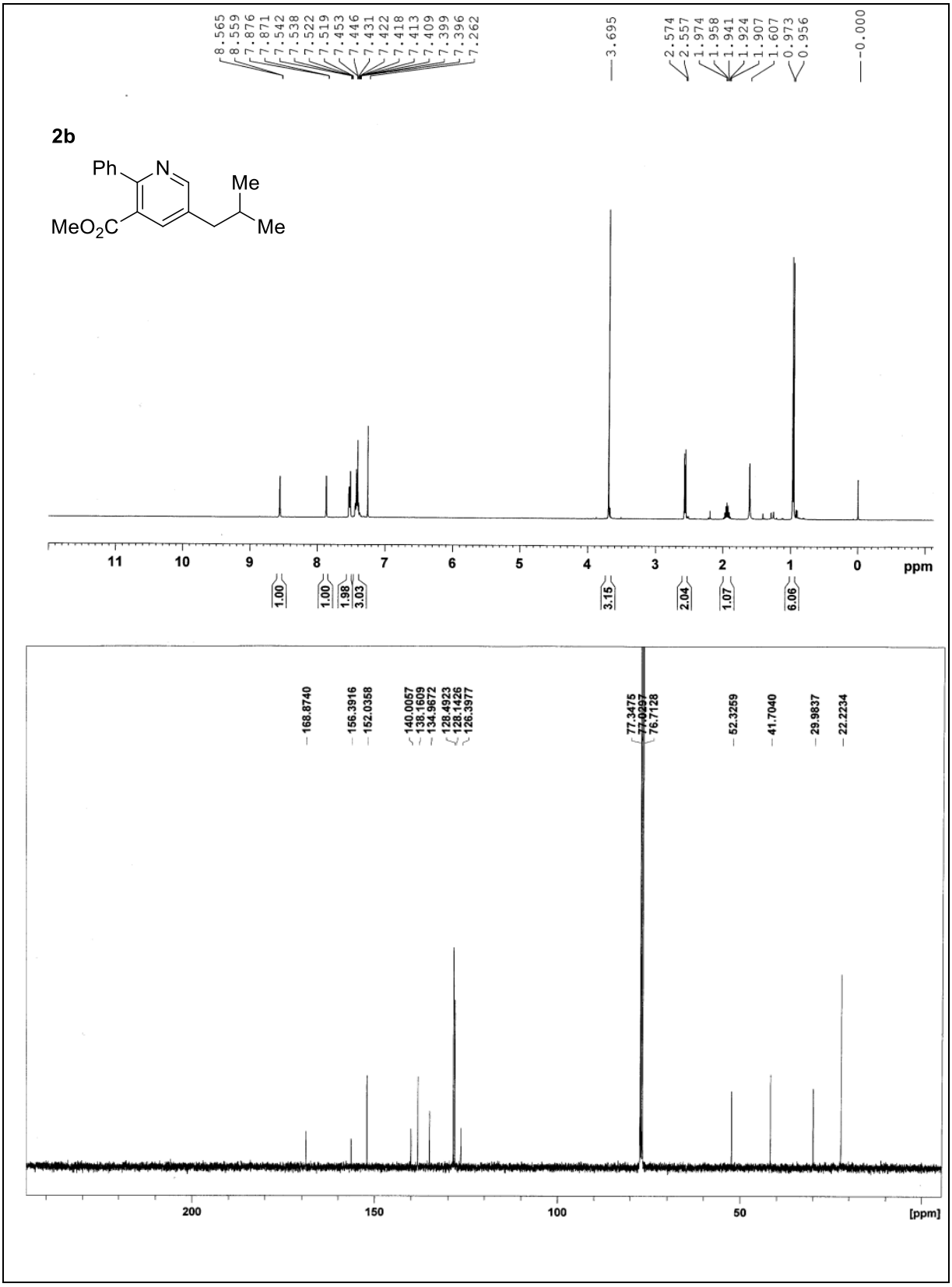
[ppm]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7.538
7.522
7.519
7.453
7.446
7.431
7.422
7.418
7.413
7.409
7.399
7.396
7.262
3.695
2.574
2.557
1.974
1.958
1.941
1.924
1.907
1.607
0.973
0.956
0.000
2b
Ph
N
Me
MeO2C
Me
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.00
1.98
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3.15
2.04
1.07
6.06
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152.0358
140.0057
138.1609
134.9672
128.4923
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22.2234
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150
100
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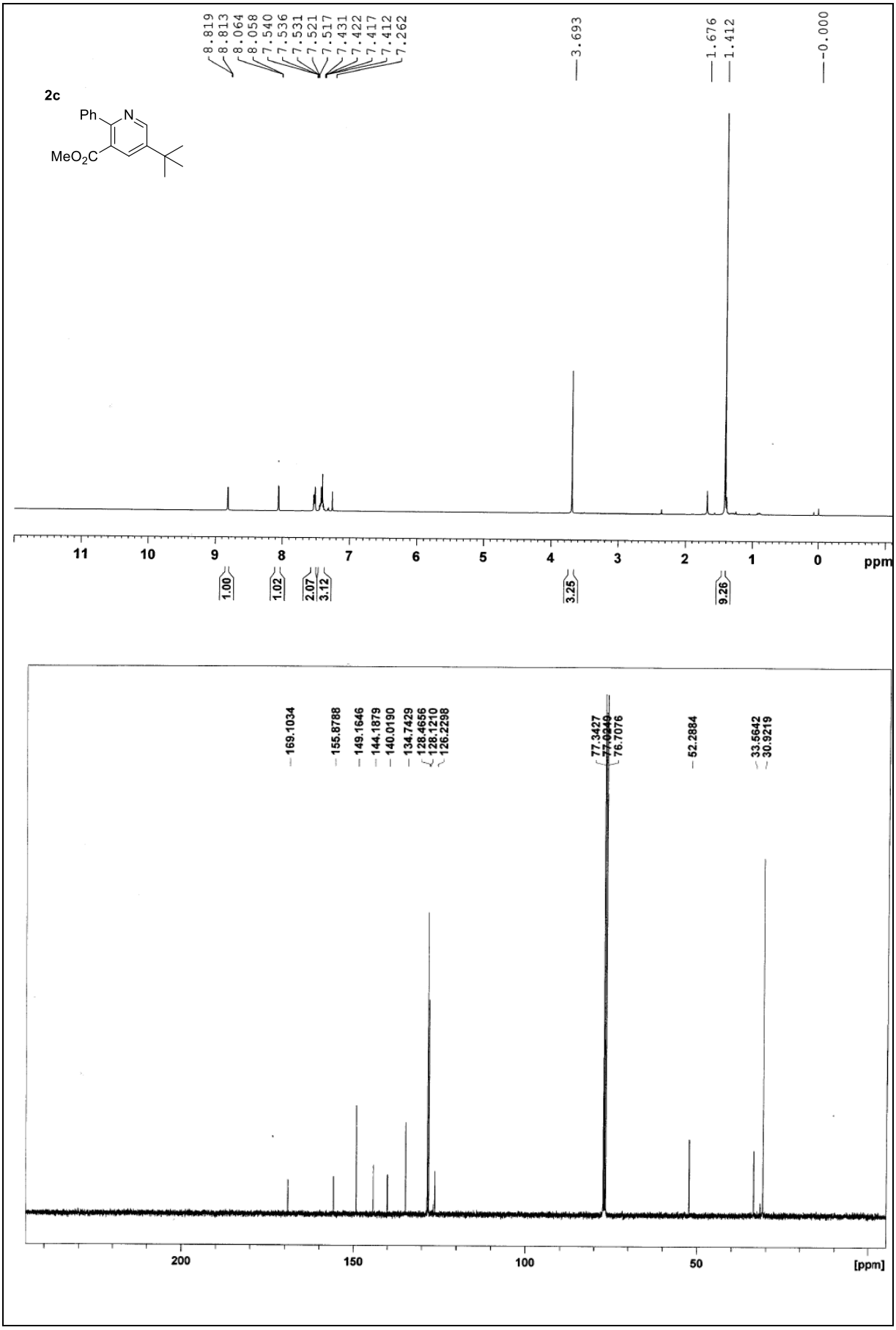
[ppm]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MeO2C
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7.531
7.521
7.517
7.431
7.422
7.417
7.412
7.262
3.693
1.676
1.412
0.000
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.02
2.07
3.12
3.25
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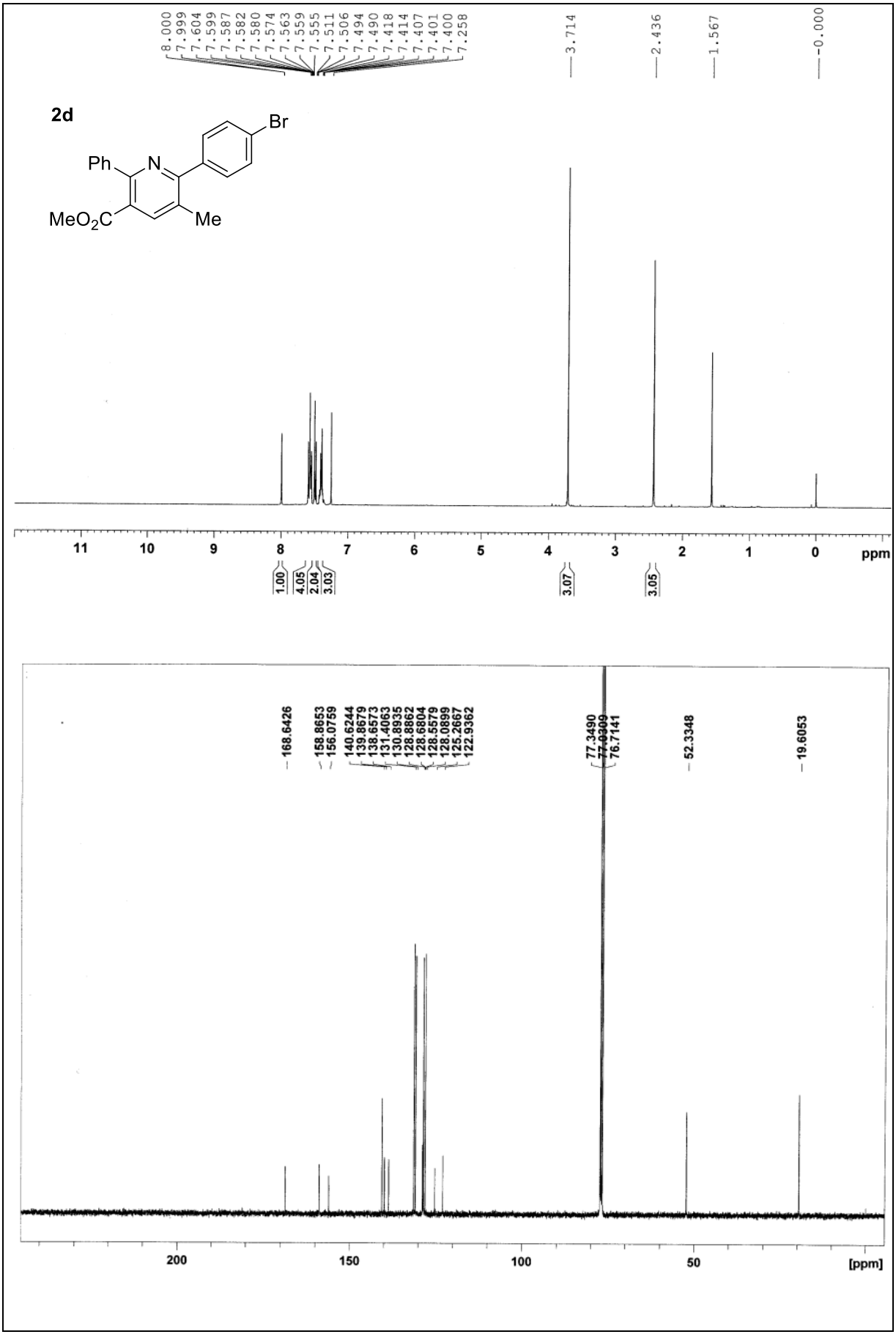
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7.574
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7.511
7.506
7.494
7.490
7.418
7.414
7.407
7.401
7.400
7.258
3.714
2.436
1.567
0.000
2d
Br
Ph
N
MeO2C
Me
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
4.05
2.04
3.03
3.07
3.05
168.6426
158.8653
156.0759
140.6244
139.8679
138.6573
131.4063
130.8935
128.8862
128.6804
128.5579
128.0899
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77.0309
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100
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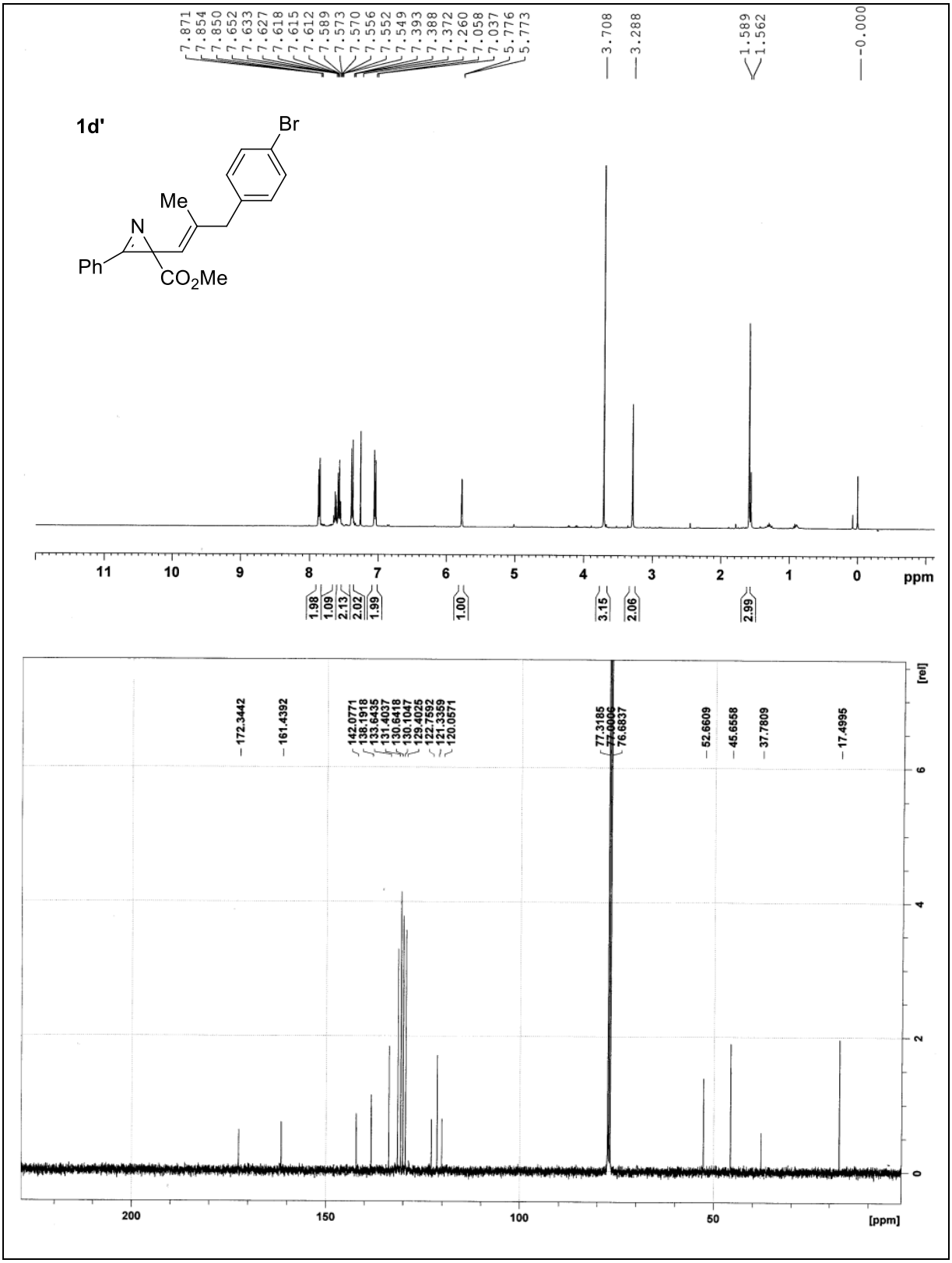
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7.615
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7.570
7.556
7.552
7.549
7.393
7.388
7.372
7.260
7.058
7.037
5.776
5.773
3.708
3.288
1.589
1.562
0.000
1d'
Br
Me
N
Ph
CO2Me
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.98
1.09
2.13
2.02
1.99
1.00
3.15
2.06
2.99
172.3442
161.4392
142.0771
138.1918
133.6435
131.4037
130.6418
130.1047
129.4025
122.7592
121.3359
120.0571
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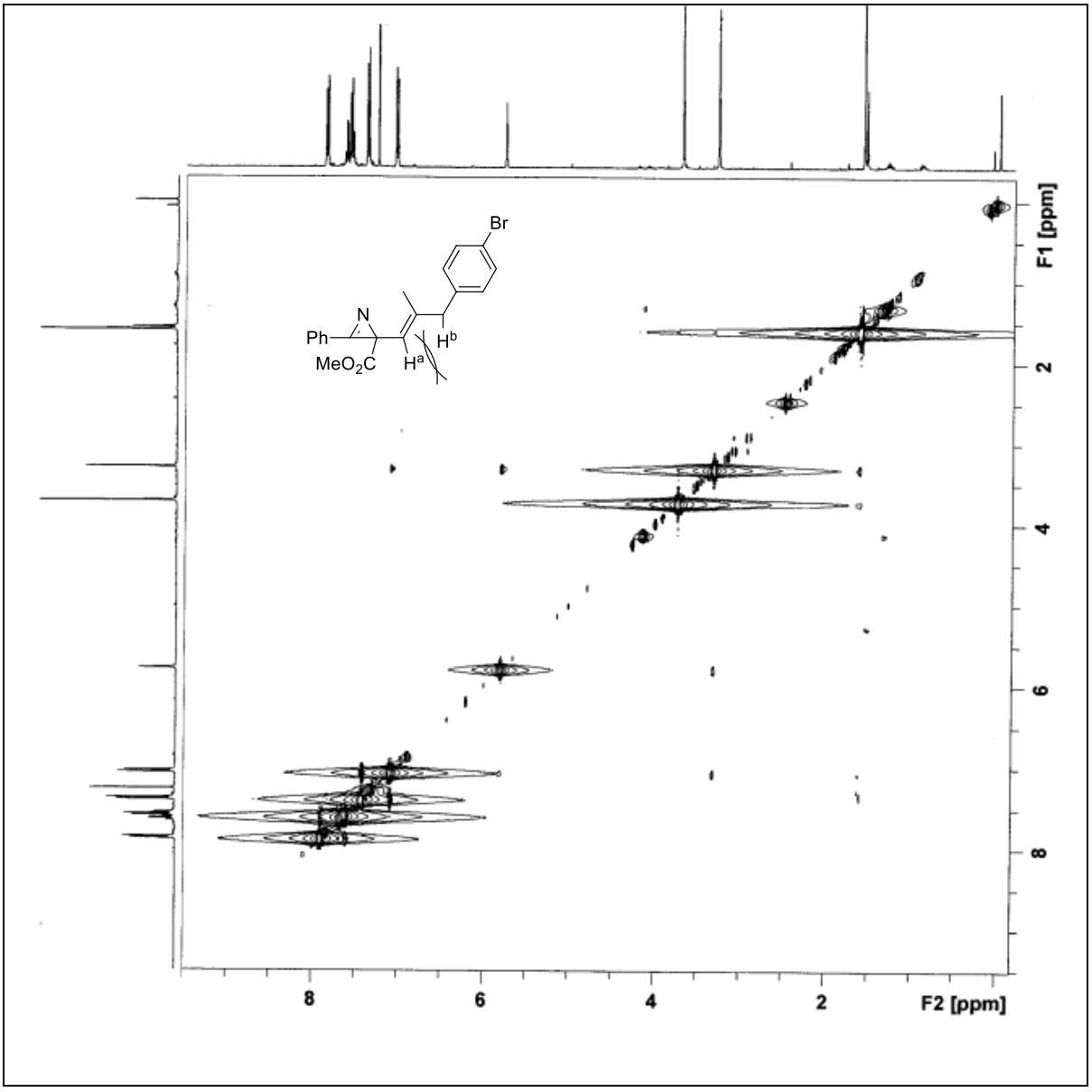
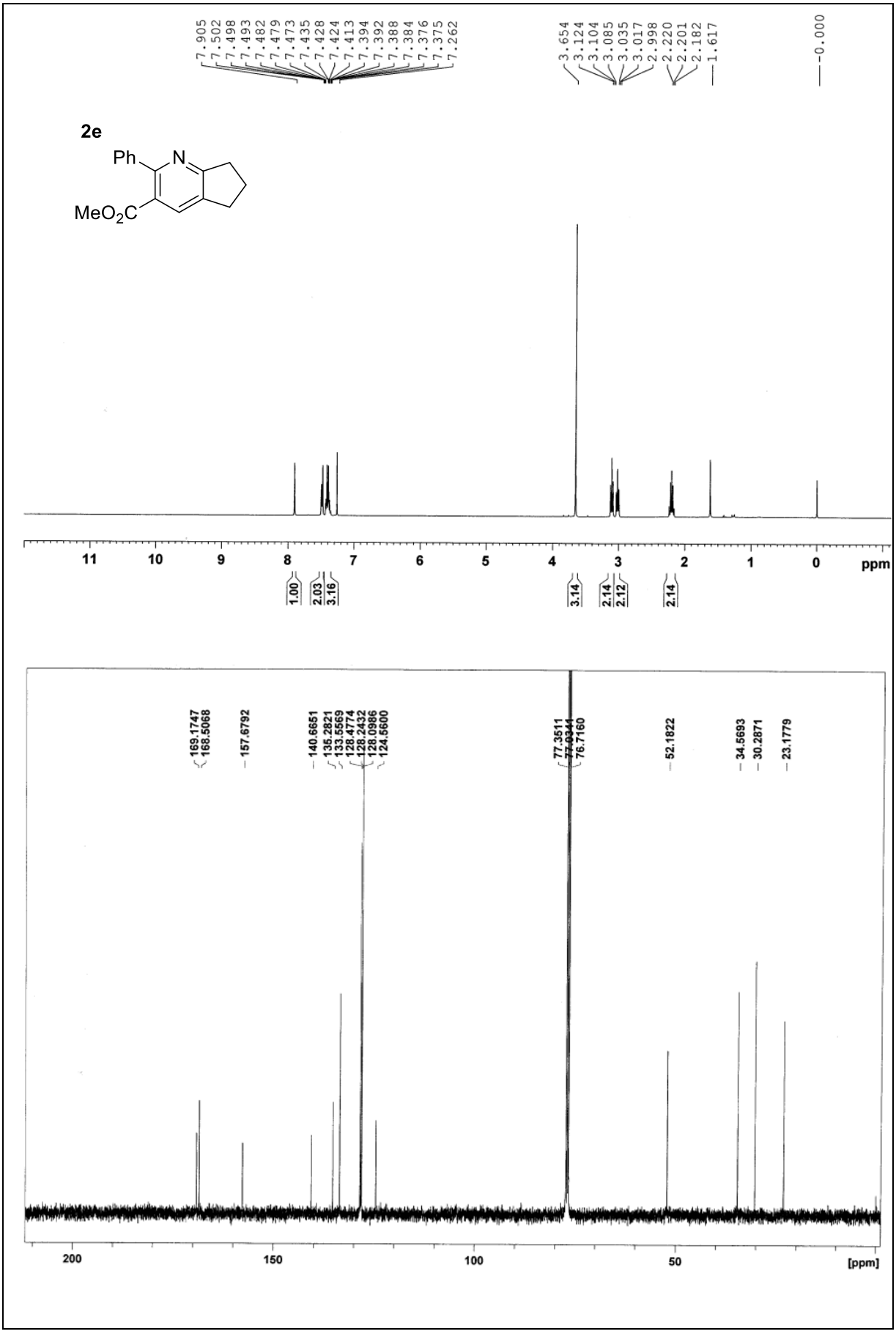

Br
N
Ph
MeO2C
Ha
Hb
F1 [ppm]
F2 [ppm]
2
4
6
8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7.473
7.435
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7.424
7.413
7.394
7.392
7.388
7.384
7.376
7.375
7.262
3.654
3.124
3.104
3.085
3.035
3.017
2.998
2.220
2.201
2.182
1.617
0.000
2e
Ph
N
MeO2C
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.03
3.16
3.14
2.14
2.12
2.14
169.1747
168.5068
157.6792
140.6651
135.2821
133.5569
128.4774
128.2432
128.0986
124.5600
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100
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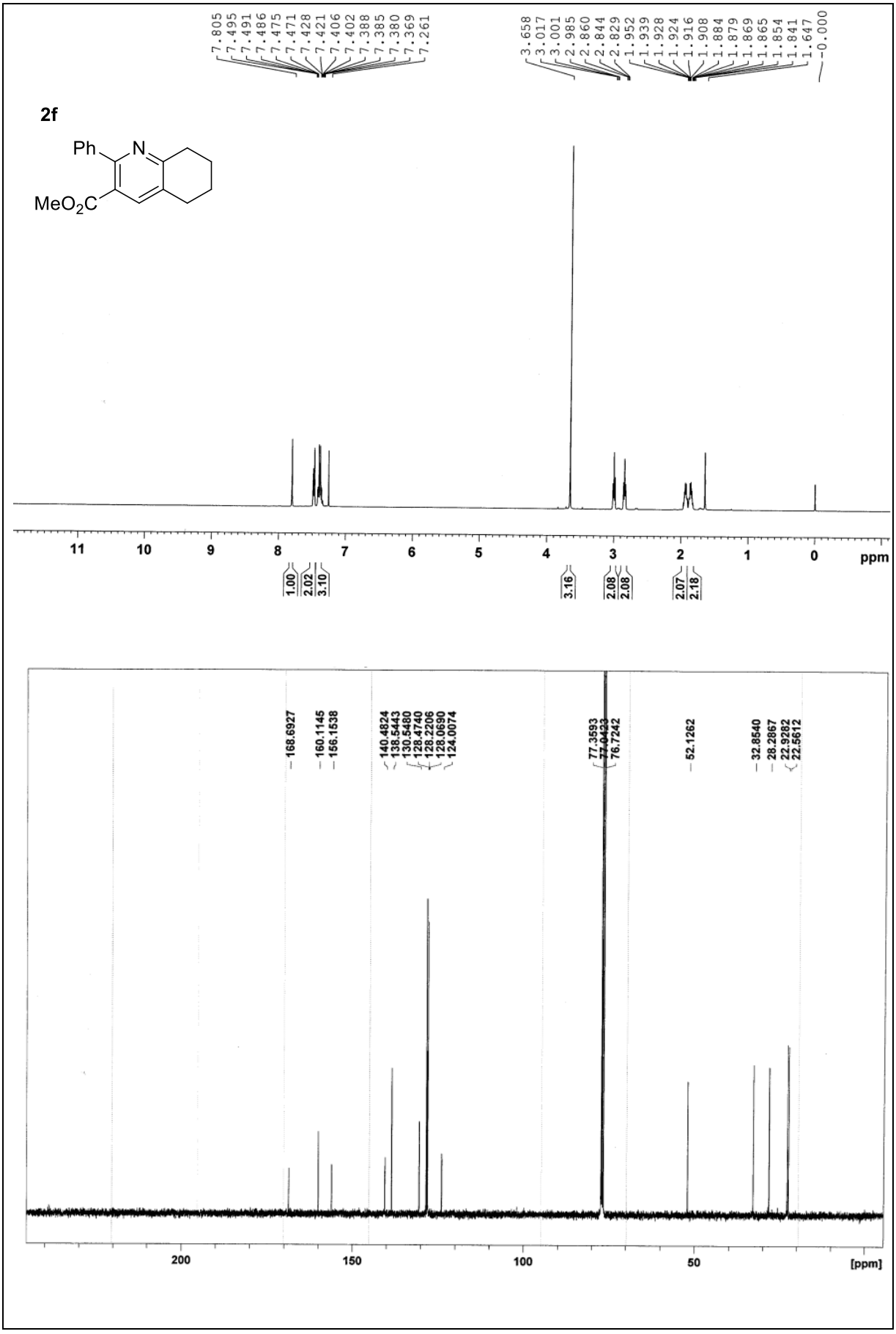
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Ph
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MeO2C
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7.475
7.471
7.428
7.421
7.406
7.402
7.388
7.385
7.380
7.369
7.261
3.658
3.017
3.001
2.985
2.860
2.844
2.829
1.952
1.939
1.928
1.924
1.916
1.908
1.884
1.879
1.869
1.865
1.854
1.841
1.647
-0.000
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.02
3.10
3.16
2.08
2.08
2.07
2.18
168.6927
160.1145
156.1538
140.4824
138.5443
130.5480
128.4740
128.2206
128.0690
124.0074
77.3593
77.0433
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52.1262
32.8540
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22.5612
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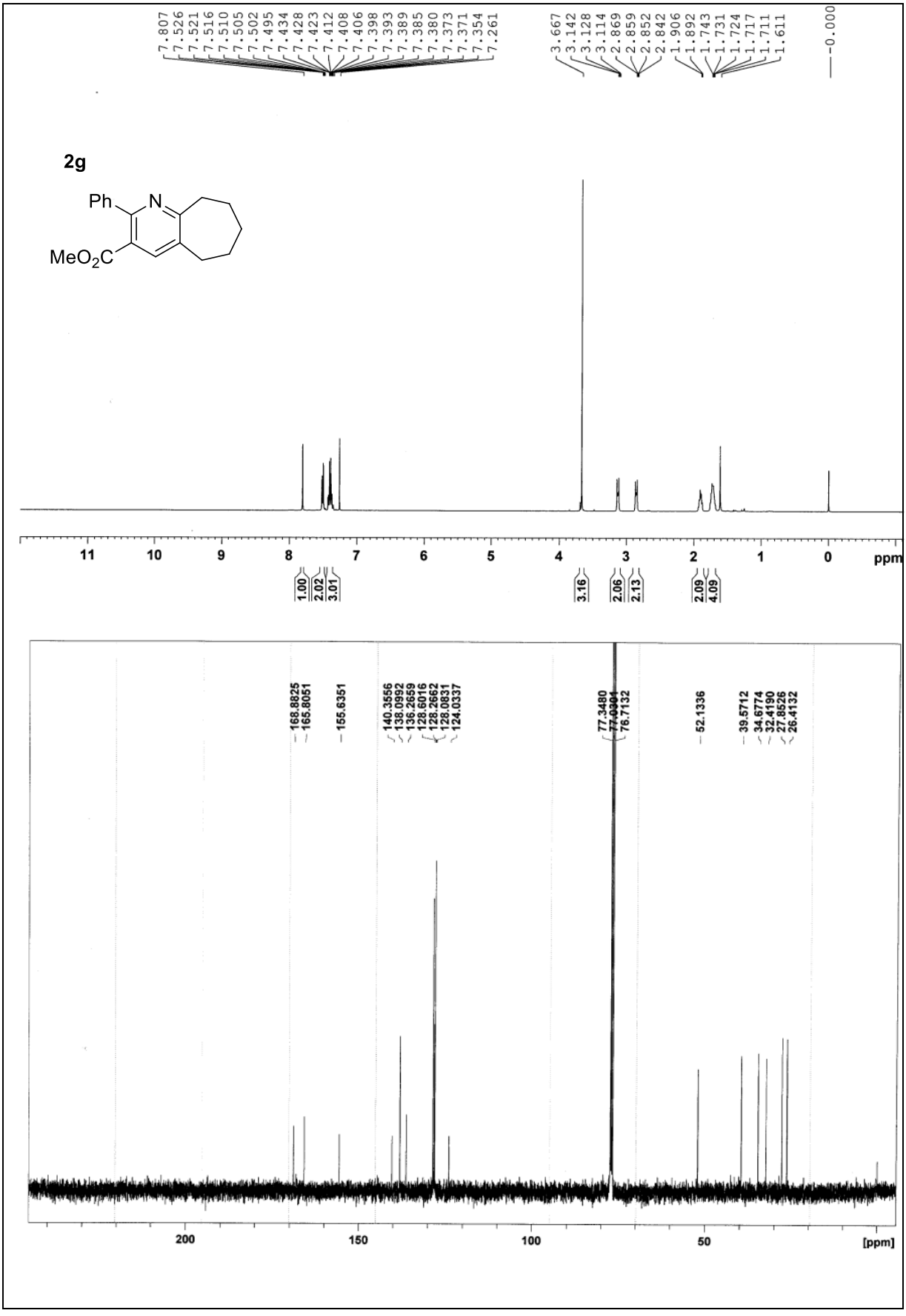
150
100
50
[ppm]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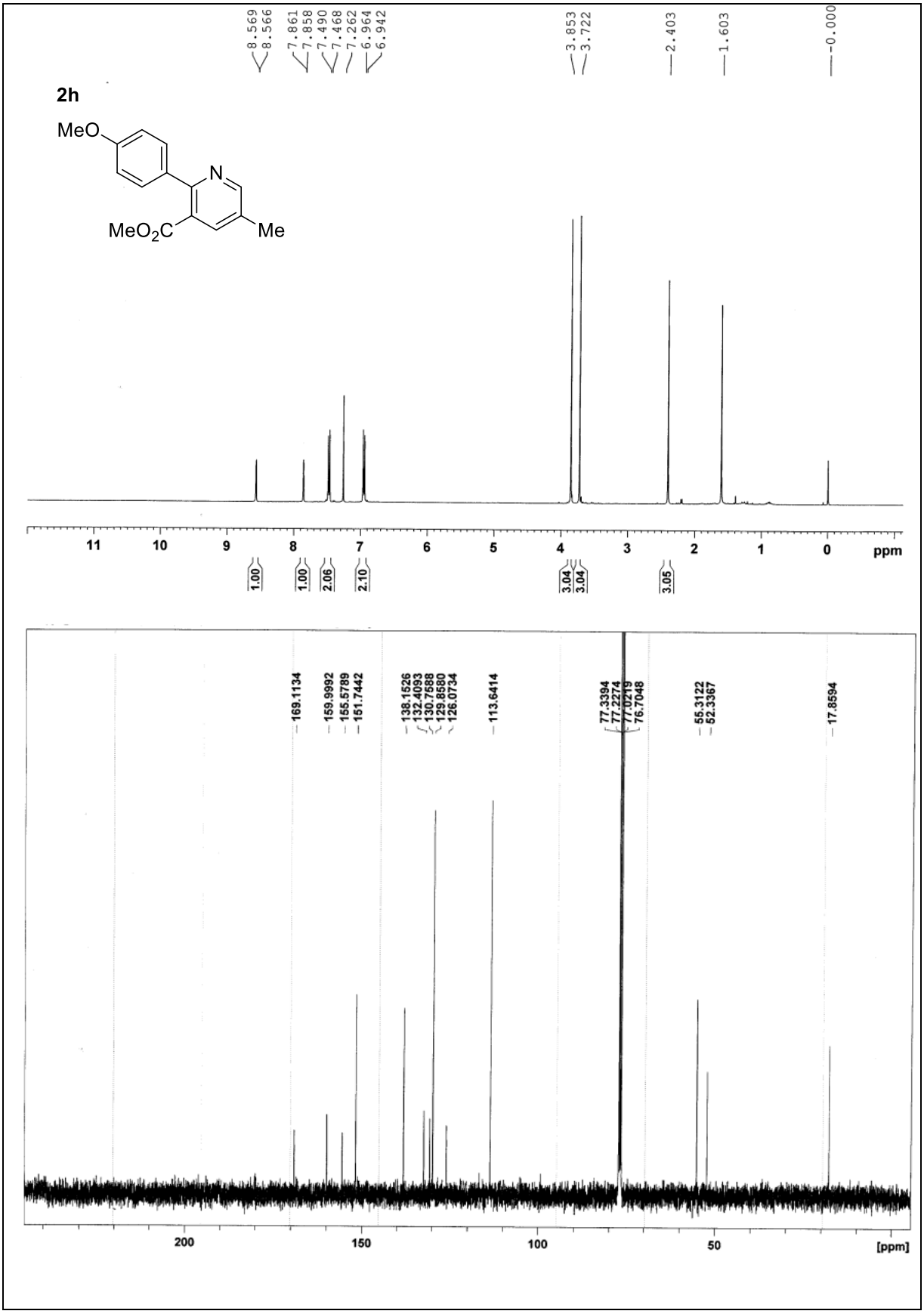
7.521
7.516
7.510
7.505
7.502
7.495
7.434
7.428
7.423
7.412
7.408
7.406
7.398
7.393
7.389
7.385
7.380
7.373
7.371
7.354
7.261
3.667
3.142
3.128
3.114
2.869
2.859
2.852
2.842
1.906
1.892
1.743
1.731
1.724
1.717
1.711
1.611
0.000
2g
Ph
N
MeO2C
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.02
3.01
3.16
2.06
2.13
2.09
4.09
168.8825
165.8051
155.6351
140.3556
138.0992
136.2659
128.6016
128.2662
128.0831
124.0337
77.3480
77.0301
76.7132
52.1336
39.5712
34.6774
32.4190
27.8526
26.4132
200
150
100
50
[ppm]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MeO
N
MeO2C
Me
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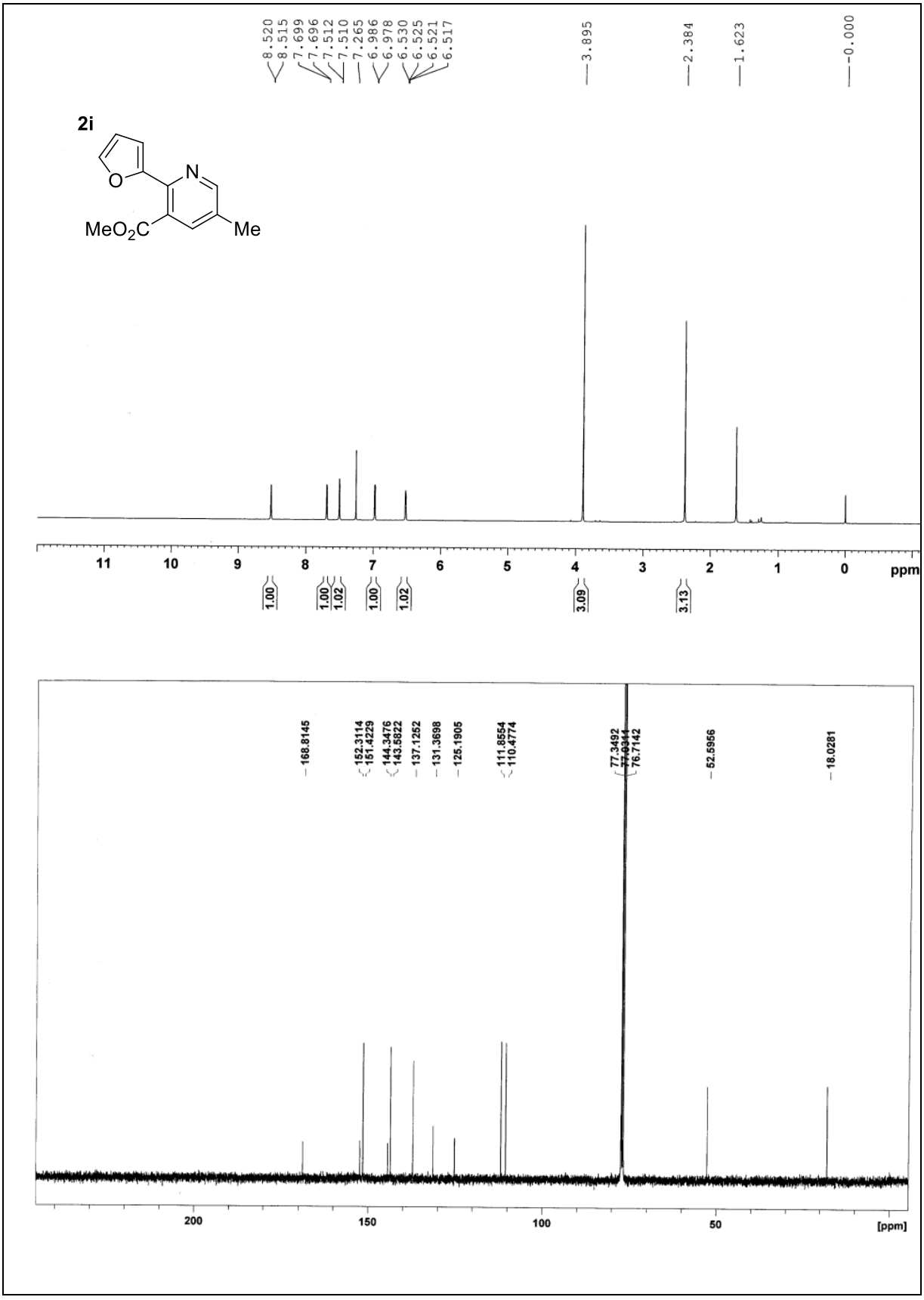
7.858
7.490
7.468
7.262
6.964
6.942
3.853
3.722
2.403
1.603
-0.000
ppm
1.00
1.00
2.06
2.10
3.04
3.04
3.05
169.1134
159.9992
155.5789
151.7442
138.1526
132.4093
130.7588
129.8580
126.0734
113.6414
77.3394
77.2274
77.0219
76.7048
55.3122
52.3367
17.8594
[ppm]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8.515
7.699
7.696
7.512
7.510
7.265
6.986
6.978
6.530
6.525
6.521
6.517
3.895
2.384
1.623
0.000
2i
O
N
MeO2C
Me
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.00
1.02
1.00
1.02
3.09
3.13
168.8145
152.3114
151.4229
144.3476
143.8822
137.1252
131.3698
125.1905
111.8554
110.4774
77.3492
77.0344
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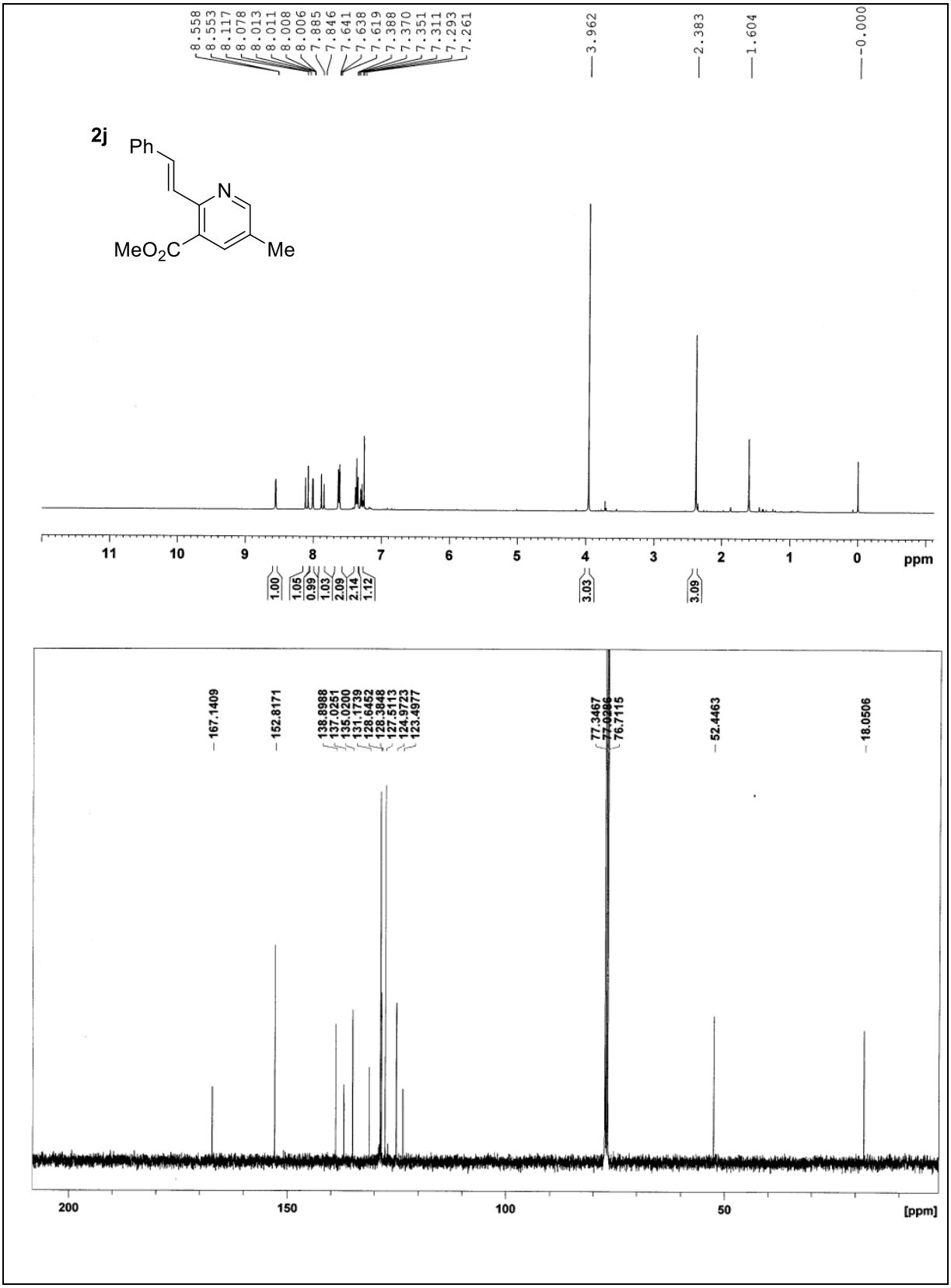
52.5956
18.0281
200
150
100
50
[ppm]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8.117
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8.013
8.011
8.008
8.006
7.885
7.846
7.641
7.638
7.619
7.388
7.370
7.351
7.311
7.293
7.261
3.962
2.383
1.604
0.000
2j
Ph
N
MeO2C
Me
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.05
0.99
1.03
2.09
2.14
1.12
3.03
3.09
167.1409
152.8171
138.8988
137.0251
135.0200
131.1739
128.6452
128.3848
127.5113
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77.3467
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76.7115
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18.0506
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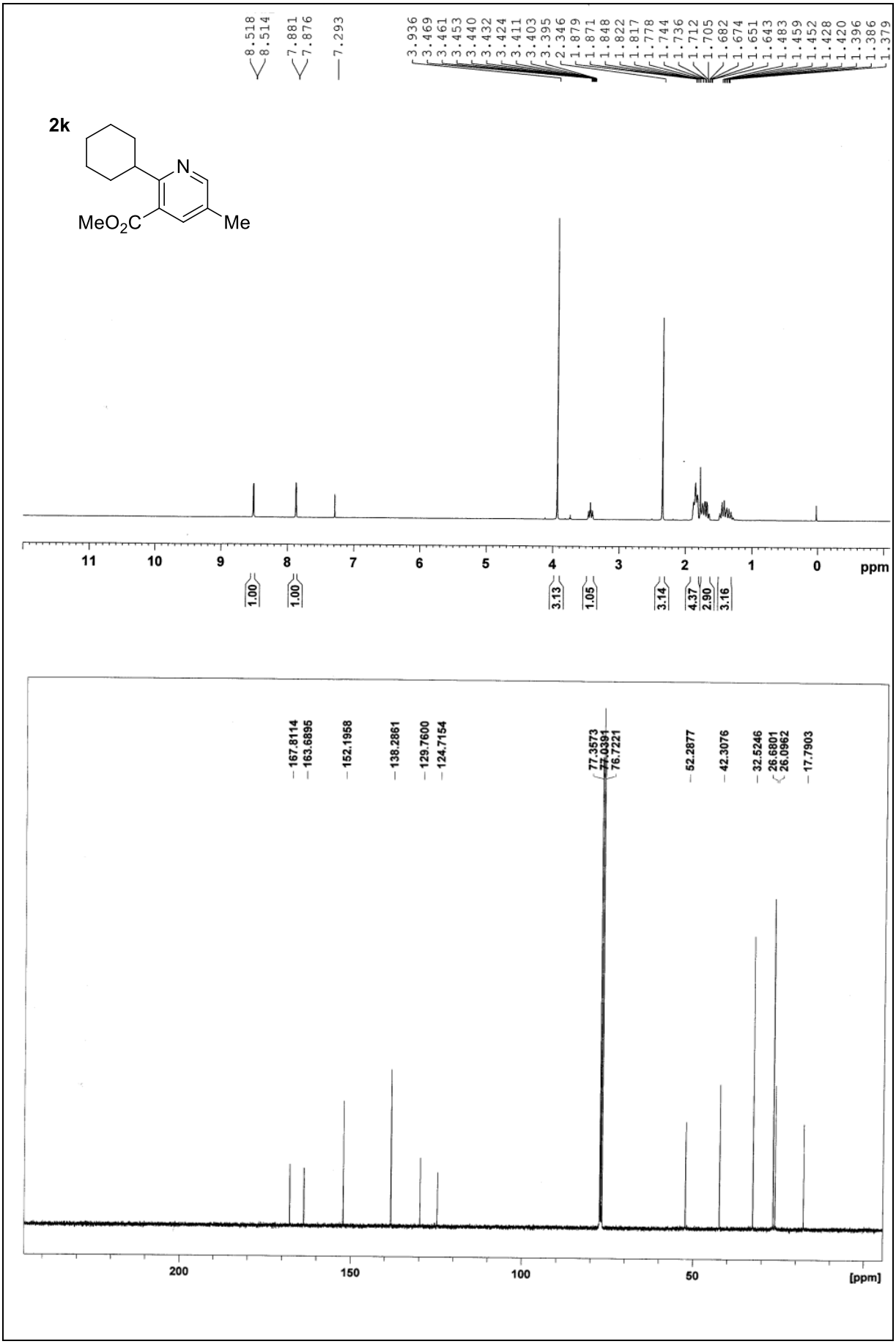
150
100
50
[ppm]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7.876
7.293
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3.469
3.461
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3.440
3.432
3.424
3.411
3.403
3.395
2.346
1.879
1.871
1.848
1.822
1.817
1.778
1.744
1.736
1.712
1.705
1.682
1.674
1.651
1.643
1.483
1.459
1.452
1.428
1.420
1.396
1.386
1.379
2k
N
MeO2C
Me
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.00
3.13
1.05
3.14
4.37
2.90
3.16
167.8114
163.6895
152.1958
138.2861
129.7600
124.7154
77.3573
77.0391
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32.5246
26.6801
26.0962
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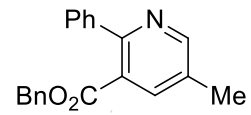
200
150
100
50
[ppm]

2l


Ph
N
BnO2C
Me

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7.489
7.486
7.480
7.388
7.378
7.372
7.367
7.364
7.362
7.356
7.279
7.271
7.268
7.262
7.253
7.025
7.021
7.014
7.008
7.002
5.119
2.407
1.717
0.000
11
10
9
8
7
6
5
4
3
2
1
0
ppm
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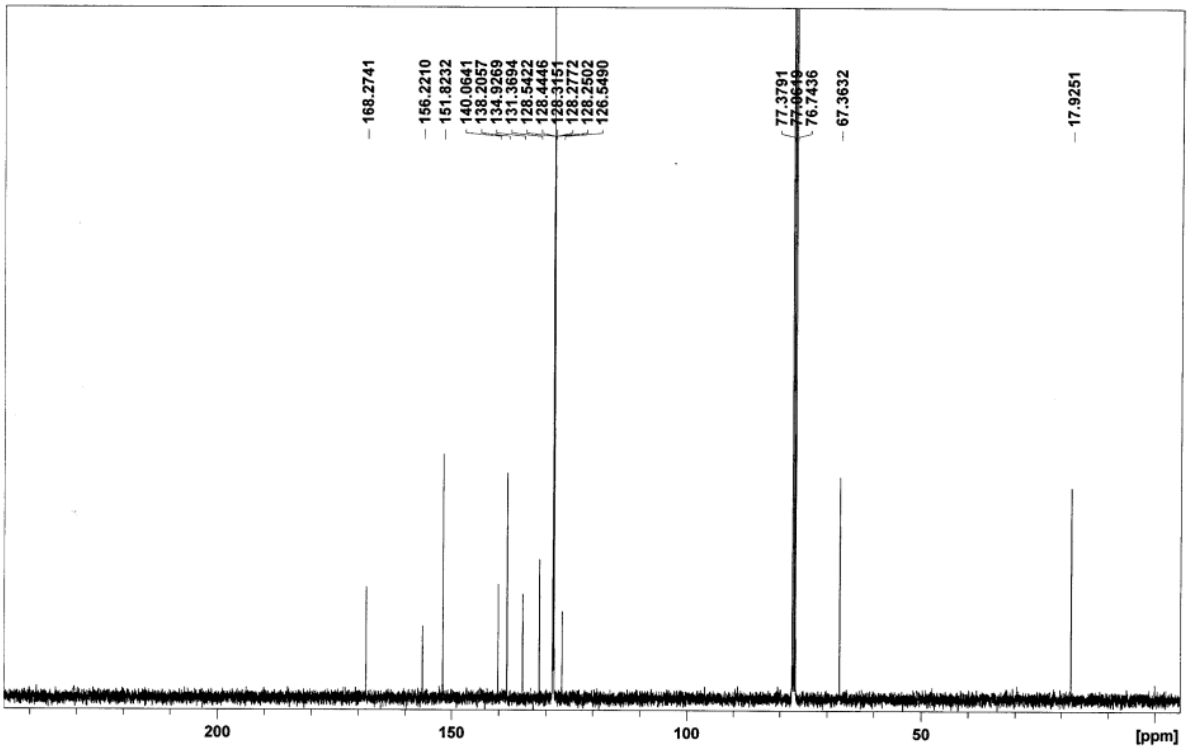
1.01
2.06
3.19
3.23
2.04
2.10
3.09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128.2772
128.2502
126.5490
77.3791
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67.3632
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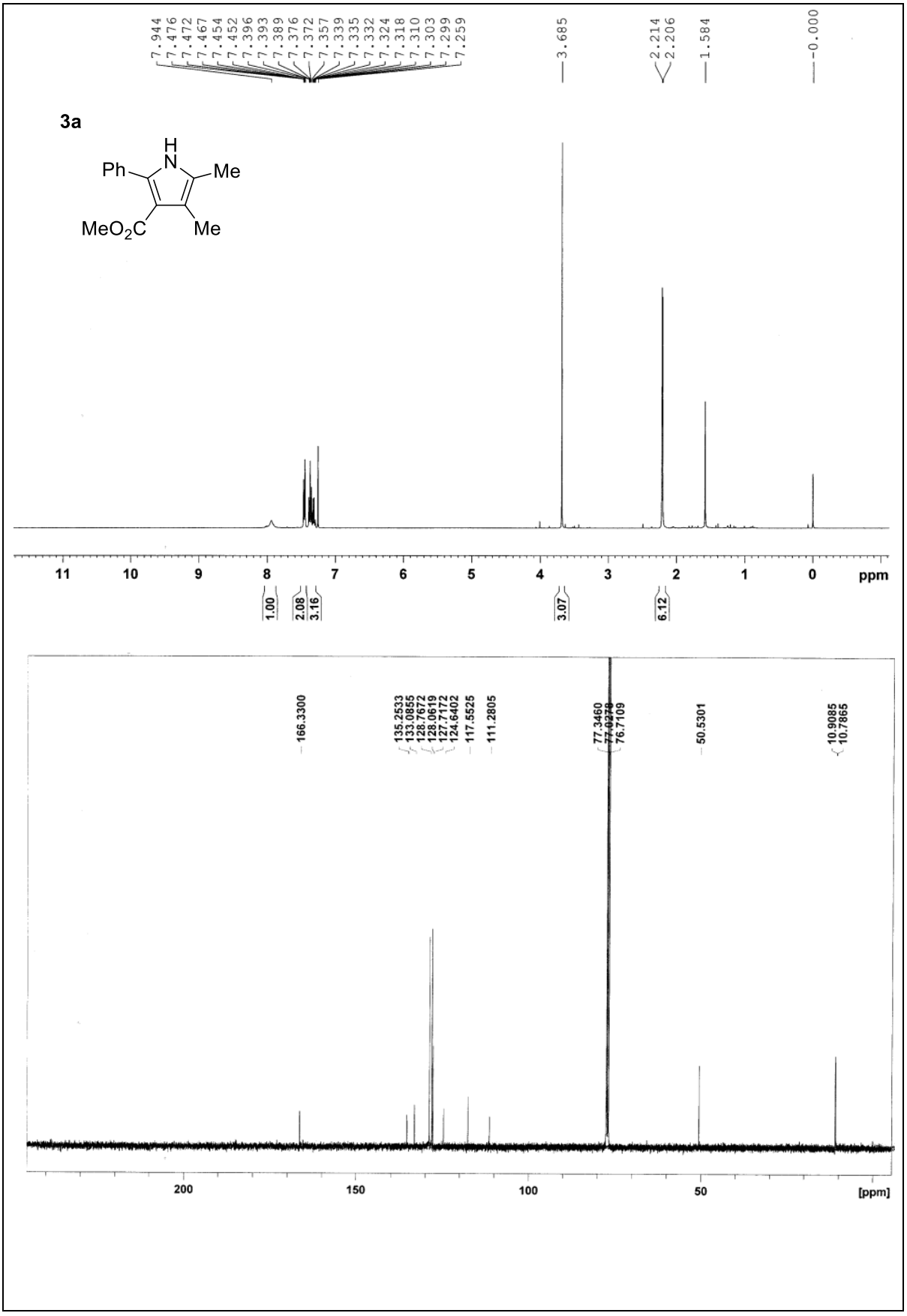
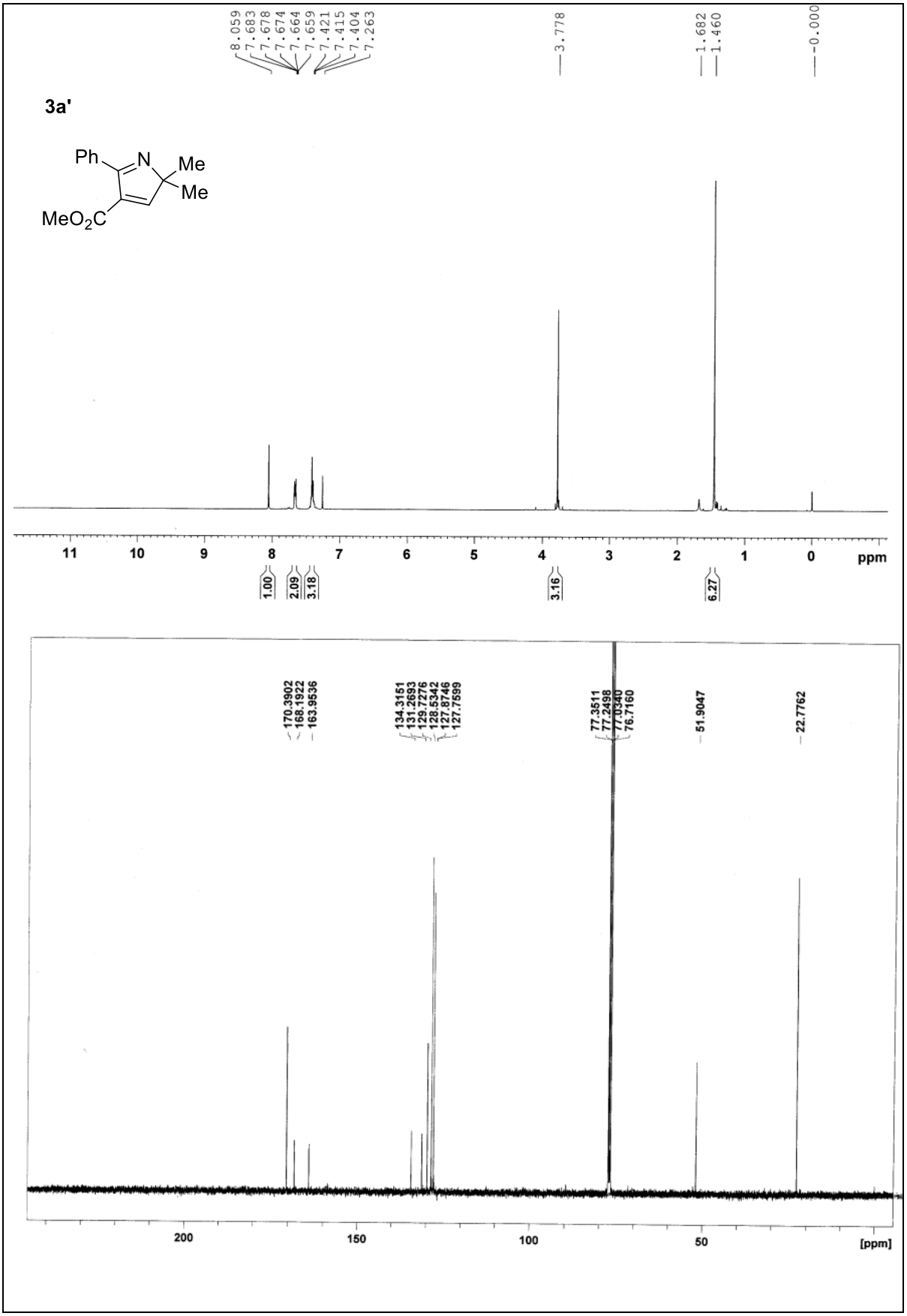
200
150
100
50
[ppm]

^{1}H and ^{13}C NMR spectra of pyrroles

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7.421
7.415
7.404
7.263
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1.682
1.460
0.000
3a'
Ph
N
Me
Me
MeO2C
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.09
3.18
3.16
6.27
170.3902
168.1922
163.9536
134.3151
131.2693
129.7276
128.5342
127.8746
127.7599
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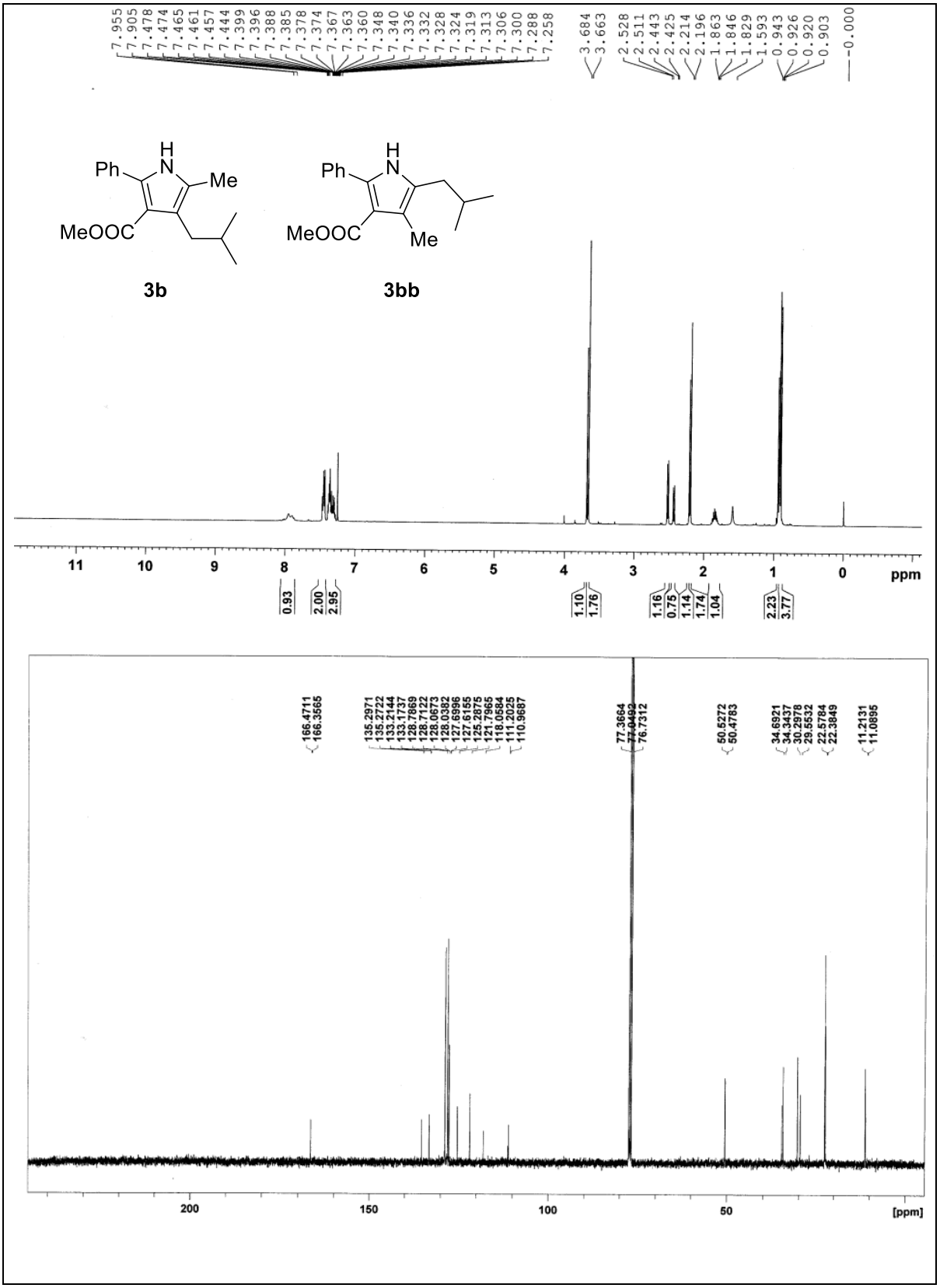
77.2498
77.0340
76.7160
51.9047
22.7762
200
150
100
50
[ppm]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7.399
7.396
7.388
7.385
7.378
7.374
7.367
7.363
7.360
7.348
7.340
7.336
7.332
7.328
7.324
7.319
7.313
7.306
7.300
7.288
7.258
3.684
3.663
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2.511
2.443
2.425
2.214
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1.846
1.829
1.593
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0.926
0.920
0.903
0.000
Ph
N
H
Me
MeOOC
3b
Ph
N
H
MeOOC
Me
3bb
11
10
9
8
7
6
5
4
3
2
1
0
ppm
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1.04
2.23
3.77
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135.2971
135.2722
133.2144
133.1737
128.7869
128.7122
128.0673
128.0382
127.6996
127.6155
125.2875
121.7965
118.0584
111.2025
110.9687
77.3664
77.0492
76.7312
50.5272
50.4783
34.6921
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29.5532
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22.3849
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11.0895
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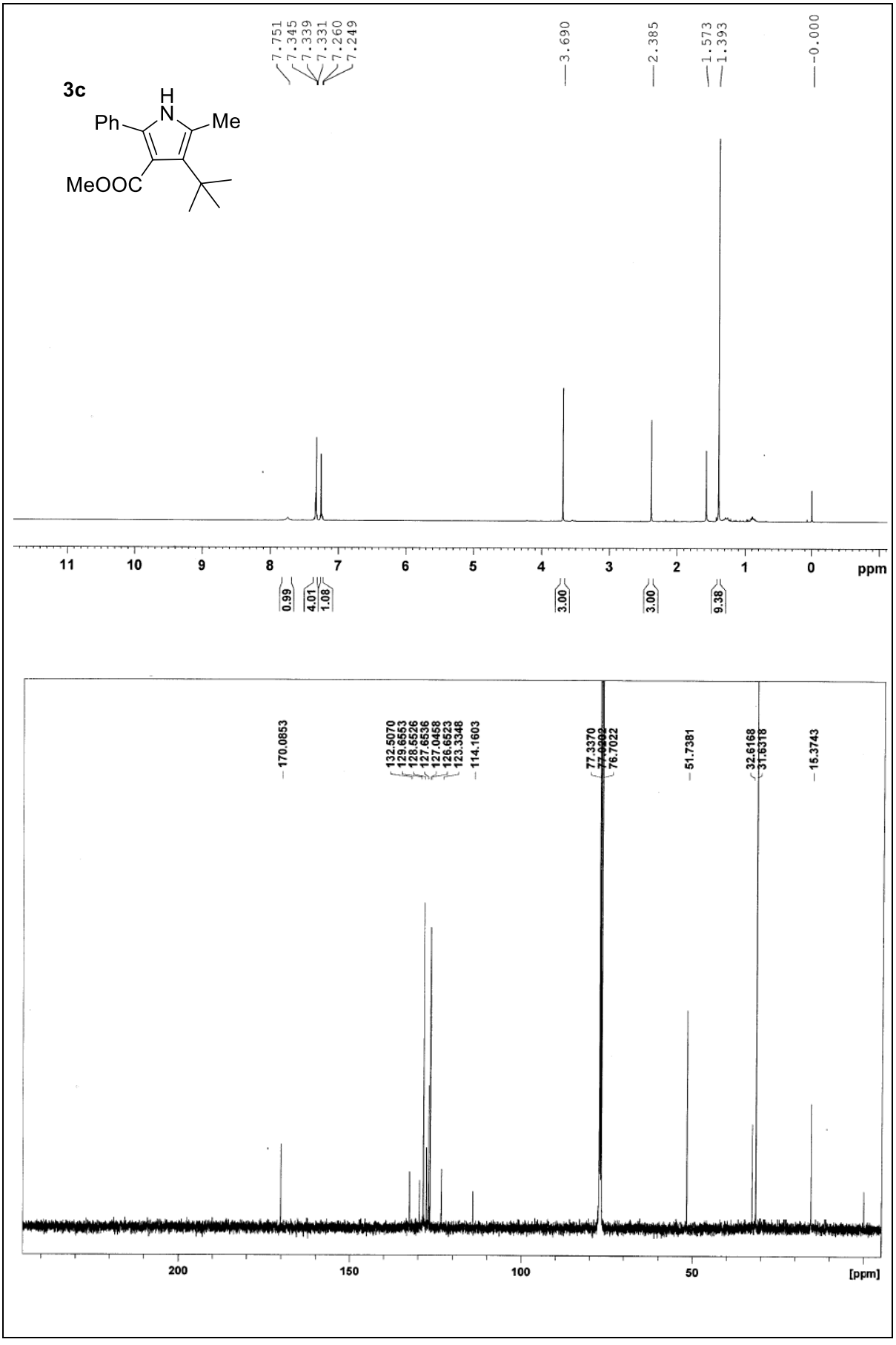
150
100
50
[ppm]


3c
Ph
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H
Me
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1.573
1.393
0.000
11
10
9
8
7
6
5
4
3
2
1
0
ppm
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4.01
1.08
3.00
3.00
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129.6553
128.5526
127.6536
127.0458
126.6523
123.3348
114.1603
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76.7022
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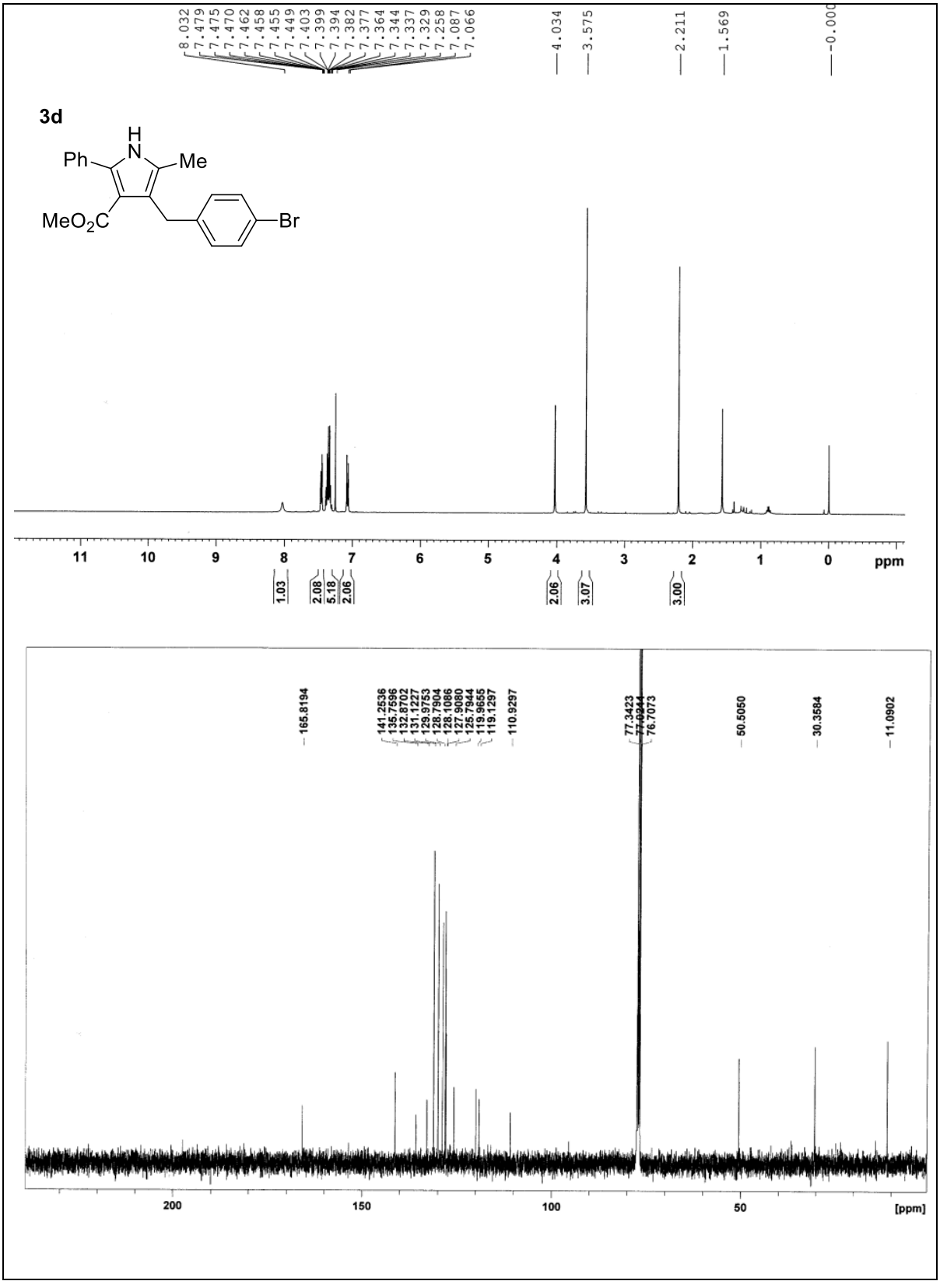
200
150
100
50
[ppm]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7.399
7.394
7.382
7.377
7.364
7.344
7.337
7.329
7.258
7.087
7.066
4.034
3.575
2.211
1.569
-0.000
3d
Ph
N
H
Me
MeO2C
Br
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.03
2.08
5.18
2.06
2.06
3.07
3.00
165.8194
141.2536
135.7596
132.8702
131.1227
129.9753
128.7904
128.1086
127.9080
125.7944
119.9655
119.1297
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77.0244
76.7073
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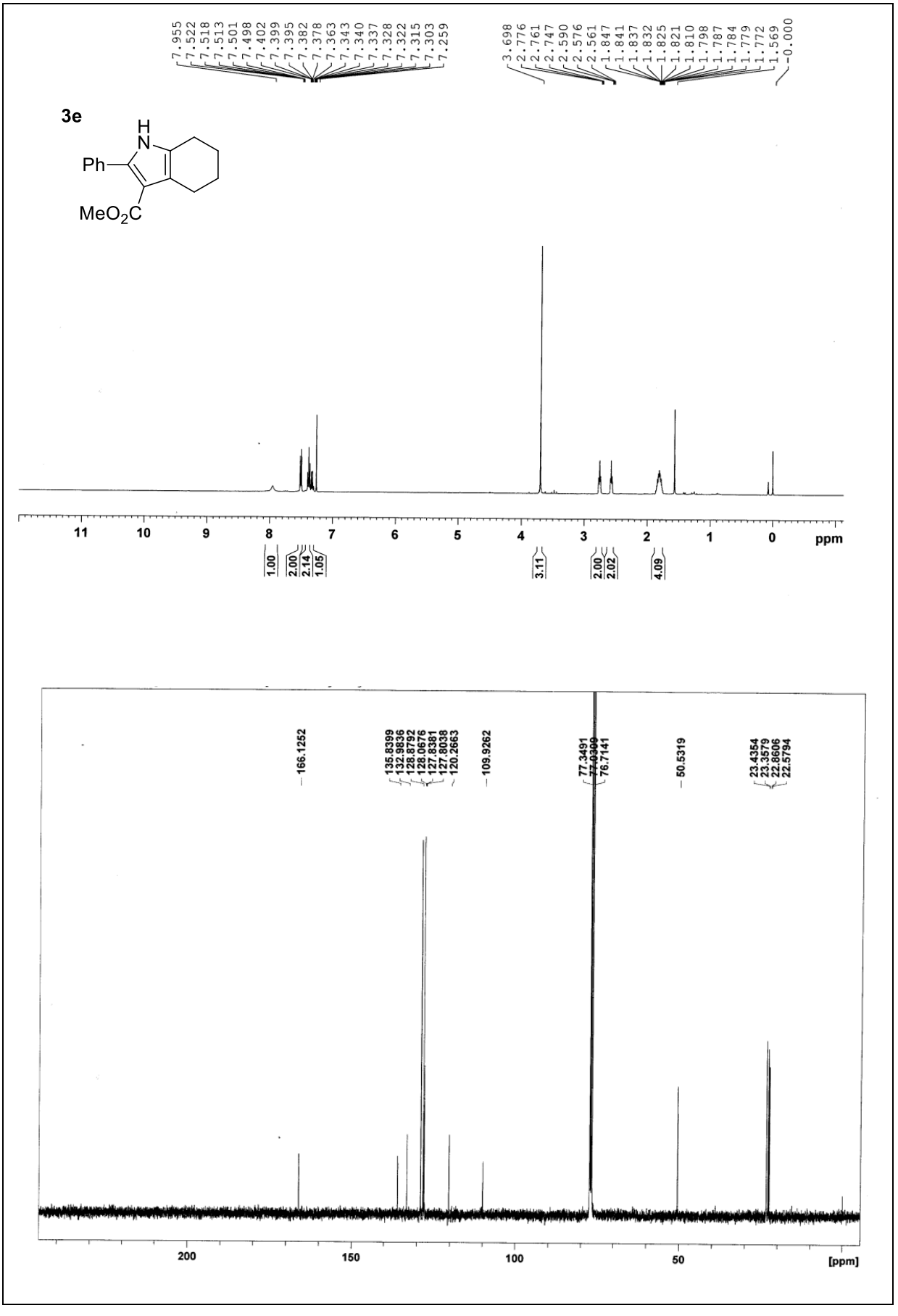
30.3584
11.0902
200
150
100
50
[ppm]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7.513
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7.399
7.395
7.382
7.378
7.363
7.343
7.340
7.337
7.328
7.322
7.315
7.303
7.259
3.698
2.776
2.761
2.747
2.590
2.576
2.561
1.847
1.841
1.837
1.832
1.825
1.821
1.810
1.798
1.787
1.784
1.779
1.772
1.569
0.000
3e
H
N
Ph
MeO2C
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.00
2.14
1.05
3.11
2.00
2.02
4.09
166.1252
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120.2663
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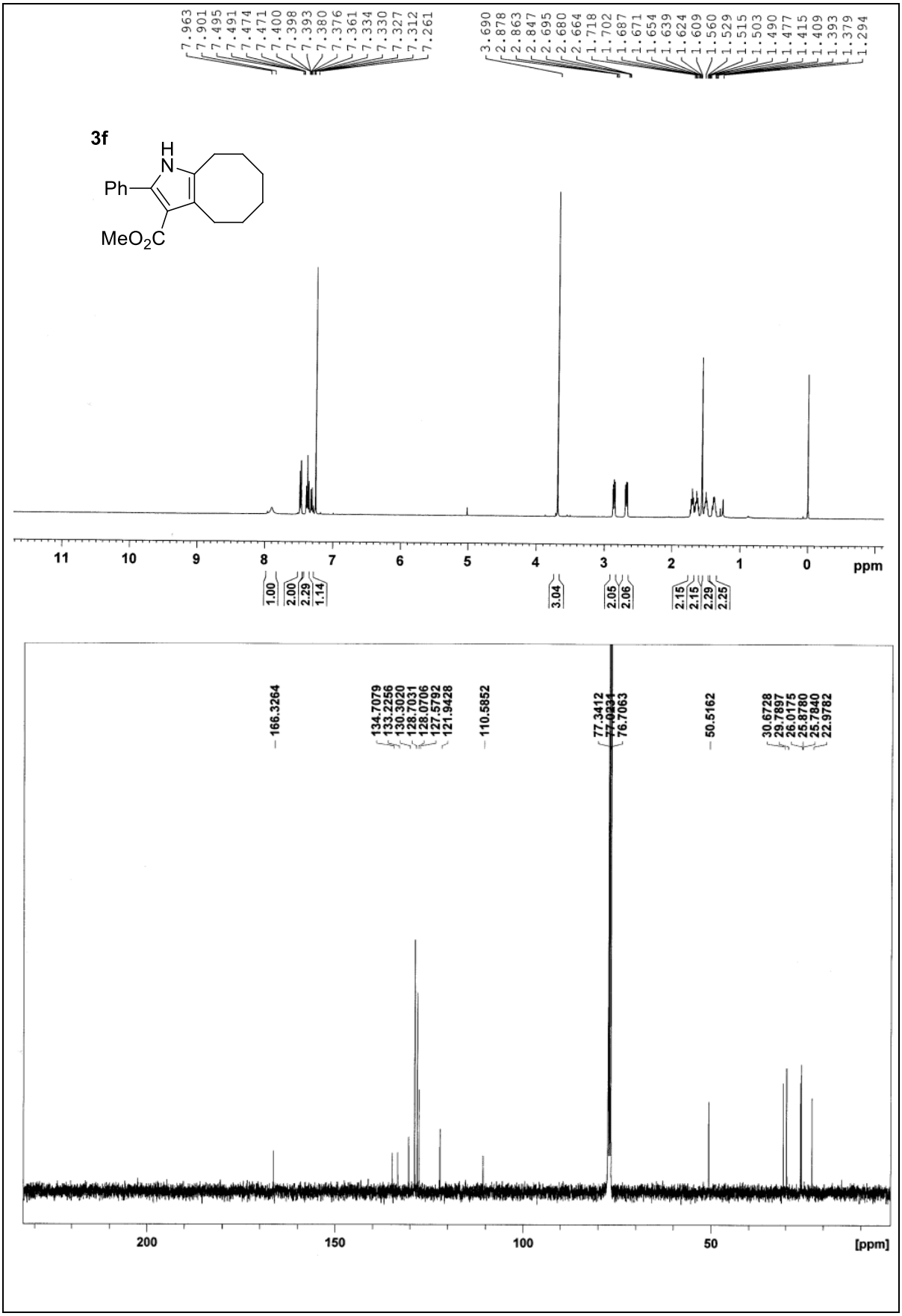
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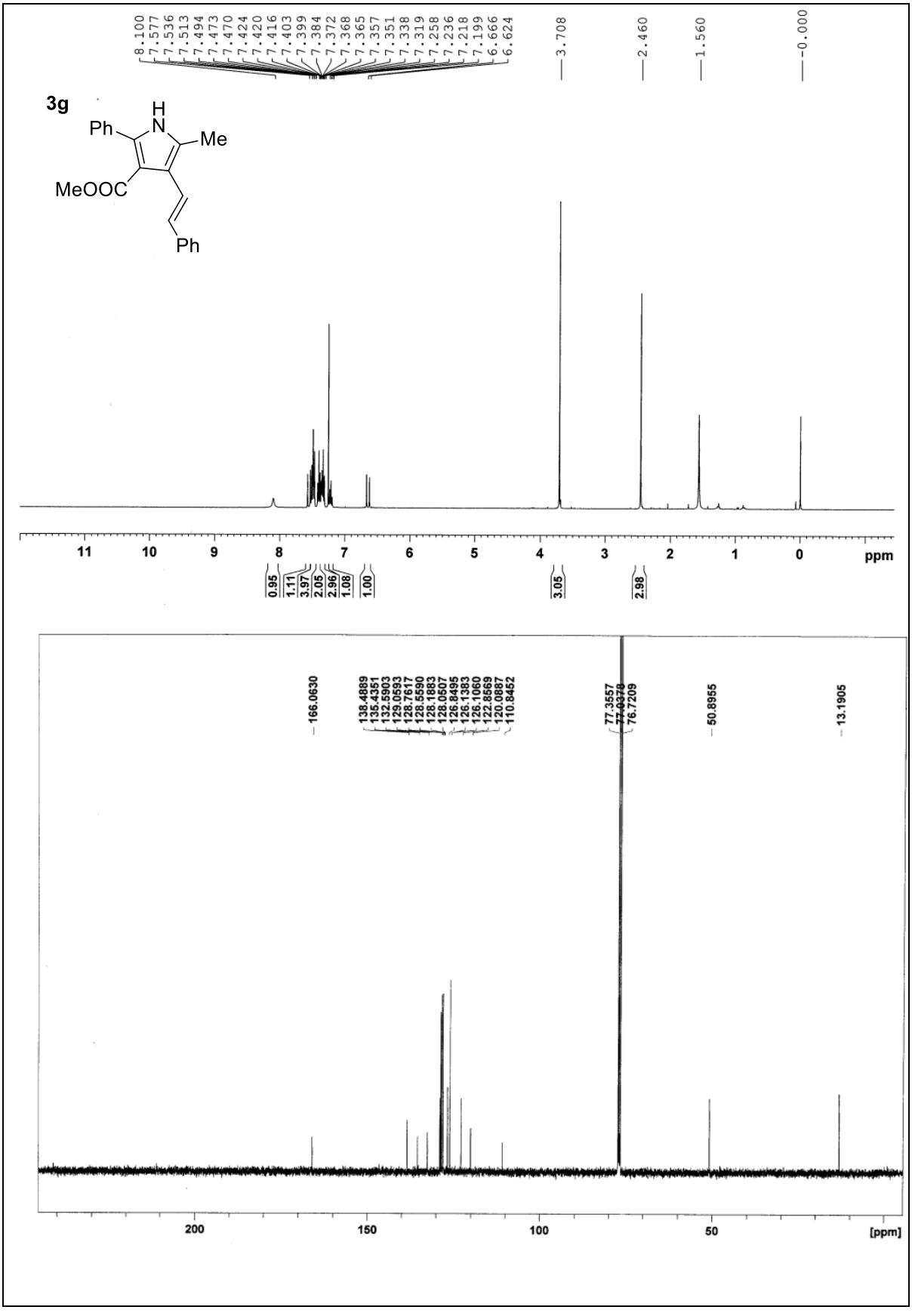
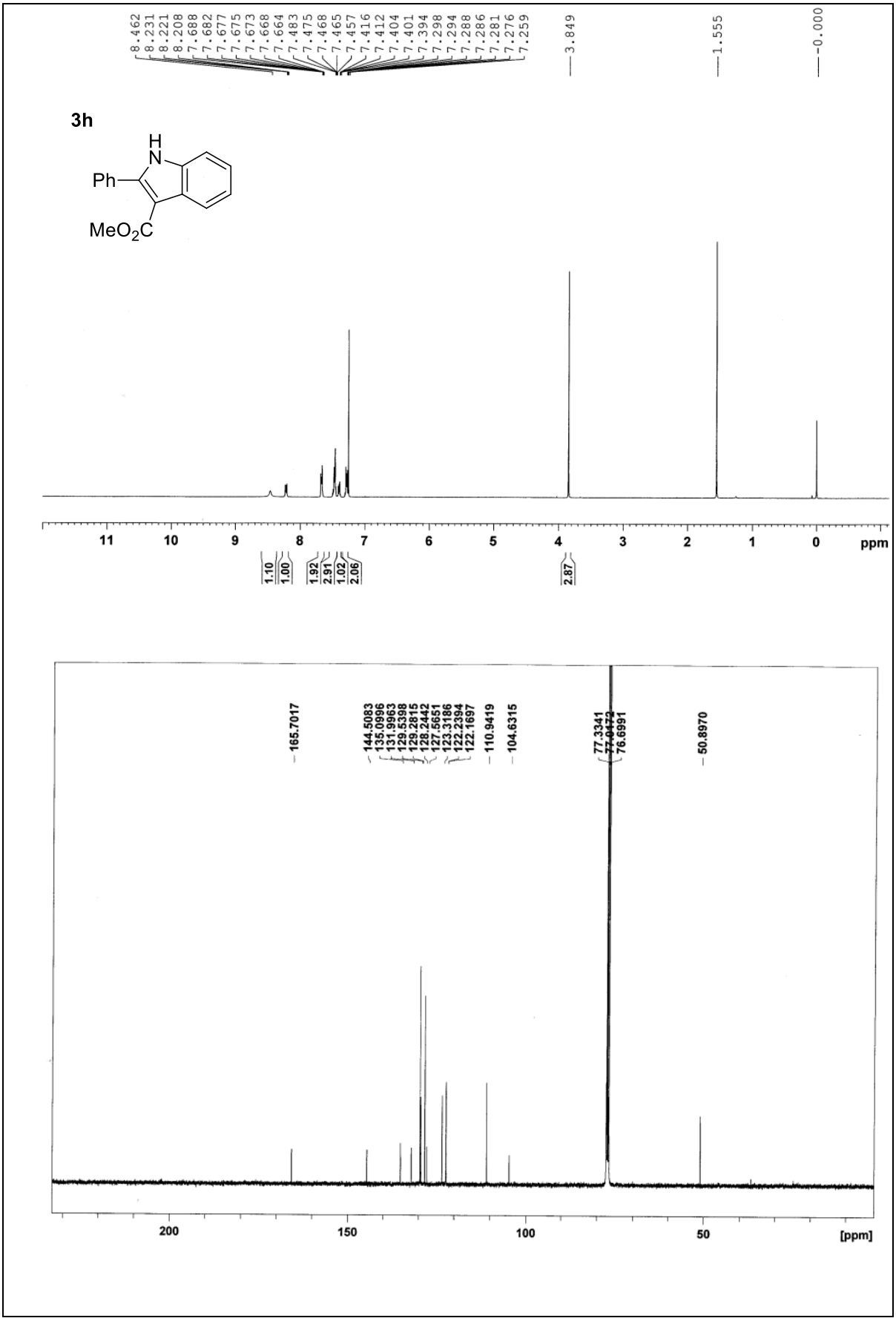
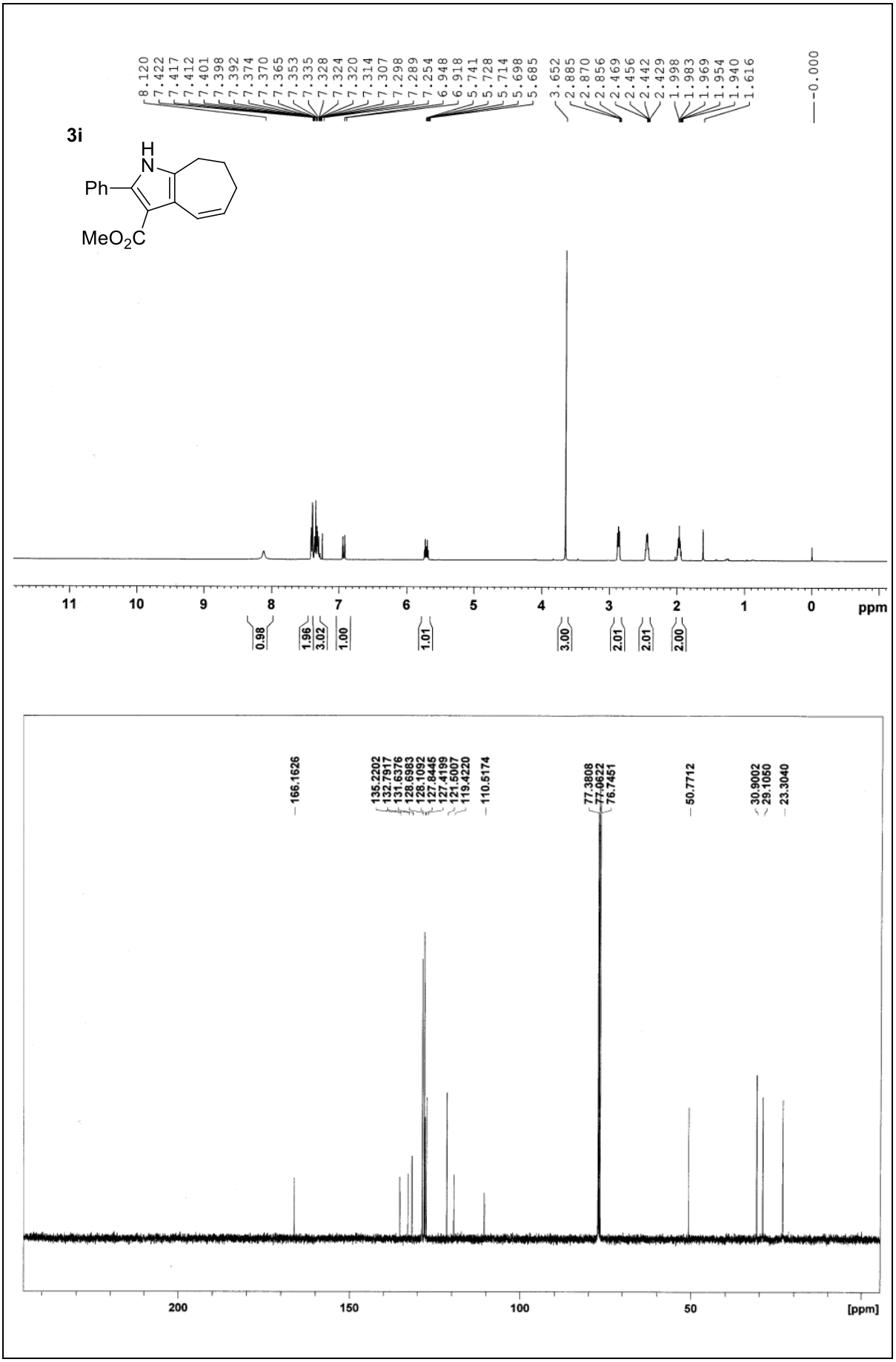
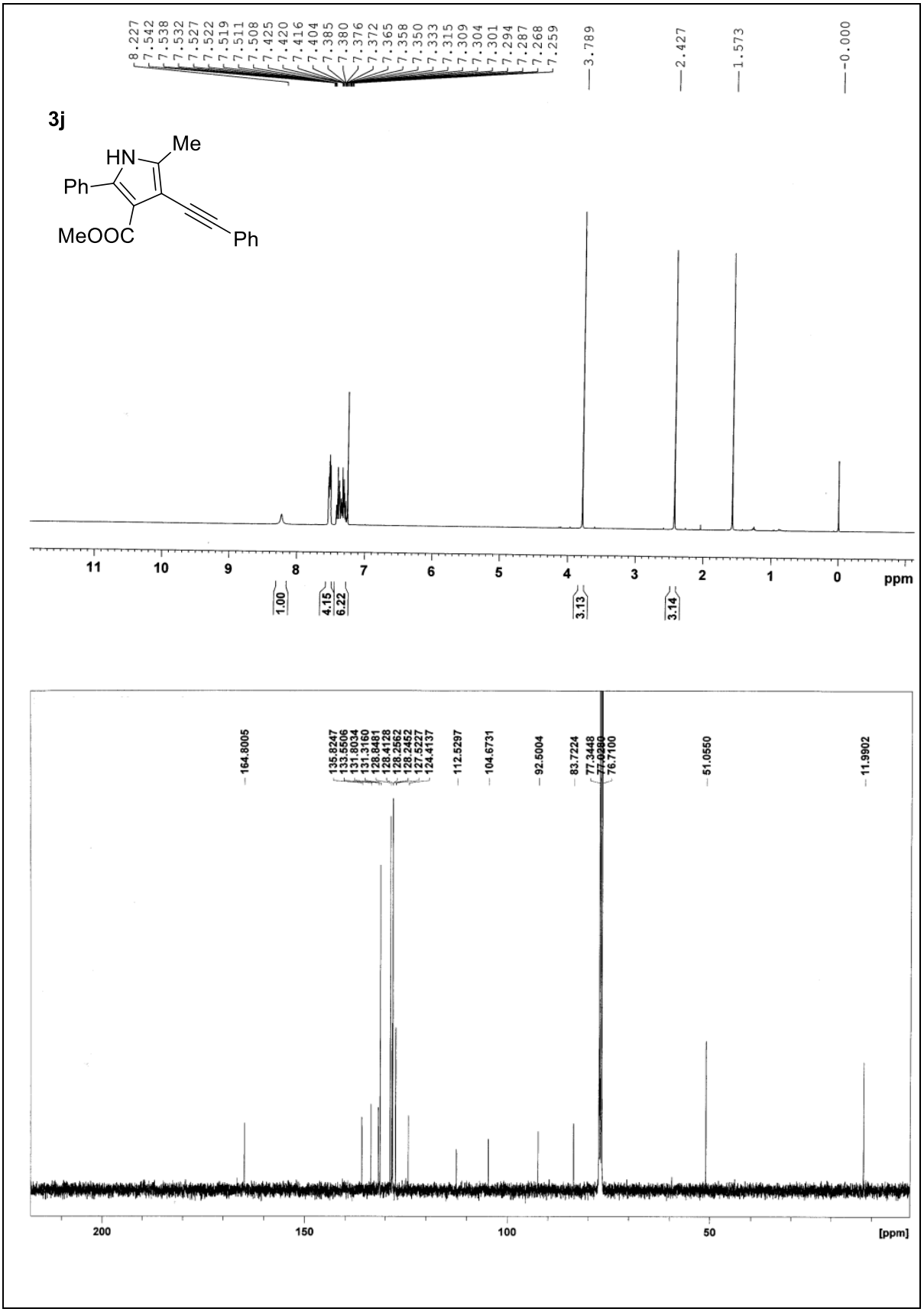
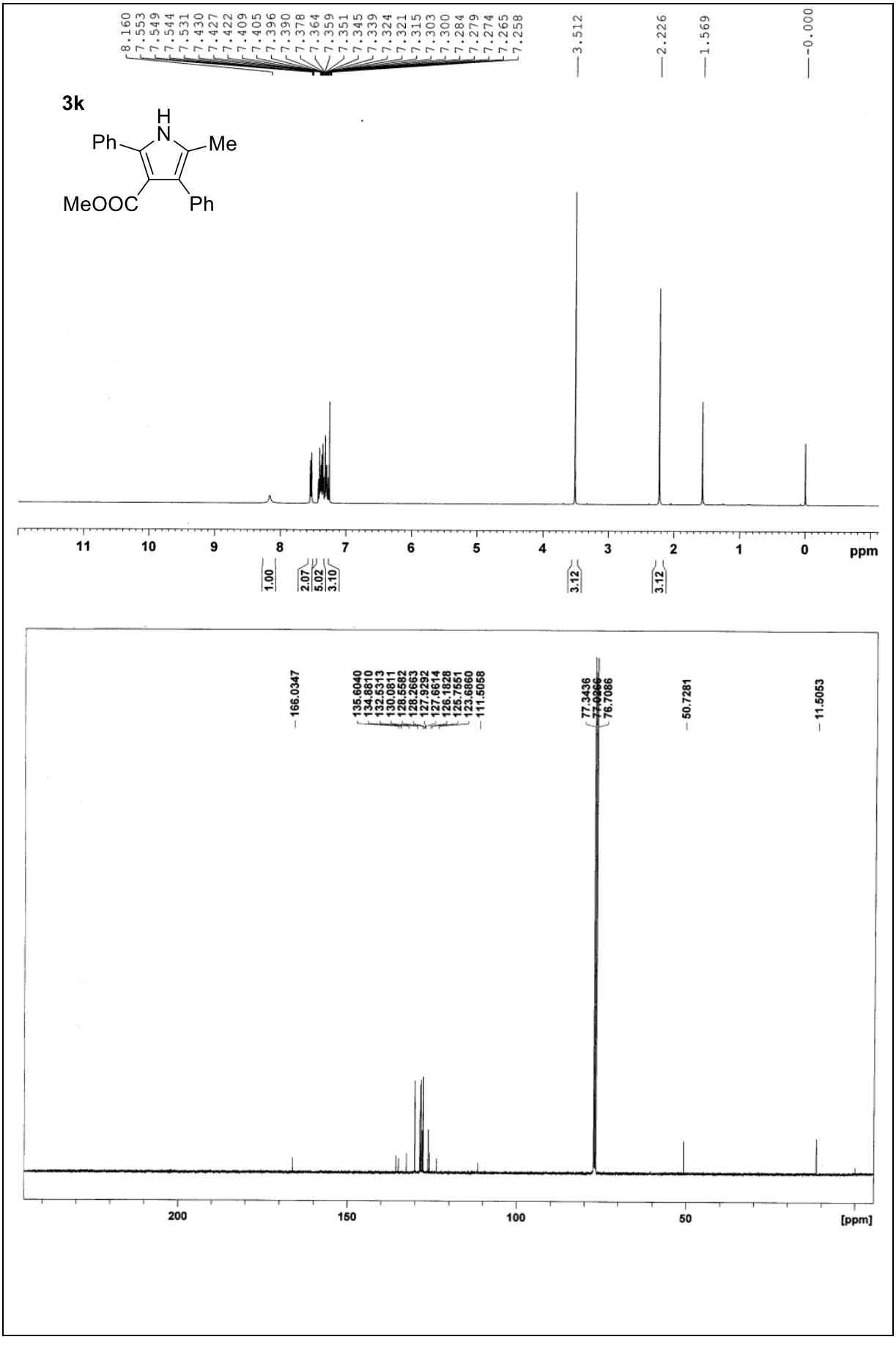
7.361
7.334
7.330
7.327
7.312
7.261
3.690
2.878
2.863
2.847
2.695
2.680
2.664
1.718
1.702
1.687
1.671
1.654
1.639
1.624
1.609
1.560
1.529
1.515
1.503
1.490
1.477
1.415
1.409
1.393
1.379
1.294
3f
H
N
Ph
MeO2C
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.00
2.29
1.14
3.04
2.05
2.06
2.15
2.15
2.29
2.25
166.3264
134.7079
133.2256
130.3020
128.7031
128.0706
127.5792
121.9428
110.5852
77.3412
77.0934
76.7063
50.5162
30.6728
29.7897
26.0175
25.8780
25.7840
22.9782
200
150
100
50
[ppm]


8.100
7.577
7.536
7.513
7.494
7.473
7.470
7.424
7.420
7.416
7.403
7.399
7.384
7.372
7.368
7.365
7.357
7.351
7.338
7.319
7.258
7.236
7.218
7.199
6.666
6.624
3.708
2.460
1.560
0.000
3g
H
N
Ph
Me
MeOOC
Ph
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.95
1.11
3.97
2.05
2.96
1.08
1.00
3.05
2.98
166.0630
138.4889
135.4351
132.5903
129.0593
128.7617
128.5590
128.1883
128.0507
126.8495
126.1383
126.1060
122.8569
120.0887
110.8452
77.3557
77.0378
76.7209
50.8955
13.1905
200
150
100
50
[ppm]

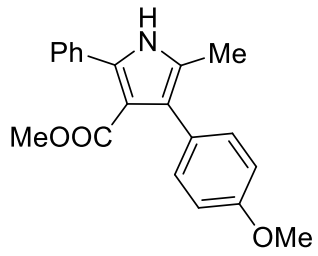

8.462
8.231
8.221
8.208
7.688
7.682
7.677
7.675
7.673
7.668
7.664
7.483
7.475
7.468
7.465
7.457
7.416
7.412
7.404
7.401
7.394
7.298
7.294
7.288
7.286
7.281
7.276
7.259
3.849
1.555
0.000
3h
H
N
Ph
MeO2C
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.10
1.00
1.92
2.91
1.02
2.06
2.87
165.7017
144.5083
135.0996
131.9963
129.5398
129.2815
128.2442
127.5651
123.3186
122.2394
122.1697
110.9419
104.6315
77.3341
77.0472
76.6991
50.8970
200
150
100
50
[ppm]


3i
Ph
MeO2C
8.120
7.422
7.417
7.412
7.401
7.398
7.392
7.374
7.370
7.365
7.353
7.335
7.328
7.324
7.320
7.314
7.307
7.298
7.289
7.254
6.948
6.918
5.741
5.728
5.714
5.698
5.685
3.652
2.885
2.870
2.856
2.469
2.456
2.442
2.429
1.998
1.983
1.969
1.954
1.940
1.616
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.98 1.96 3.02 1.00 1.01 3.00 2.01 2.01 2.00
166.1626
135.2202
132.7917
131.6376
128.6983
128.1092
127.8445
127.4199
121.5007
119.4220
110.5174
77.3808
77.0622
76.7451
50.7712
30.9002
29.1050
23.3040
200 150 100 50 [ppm]

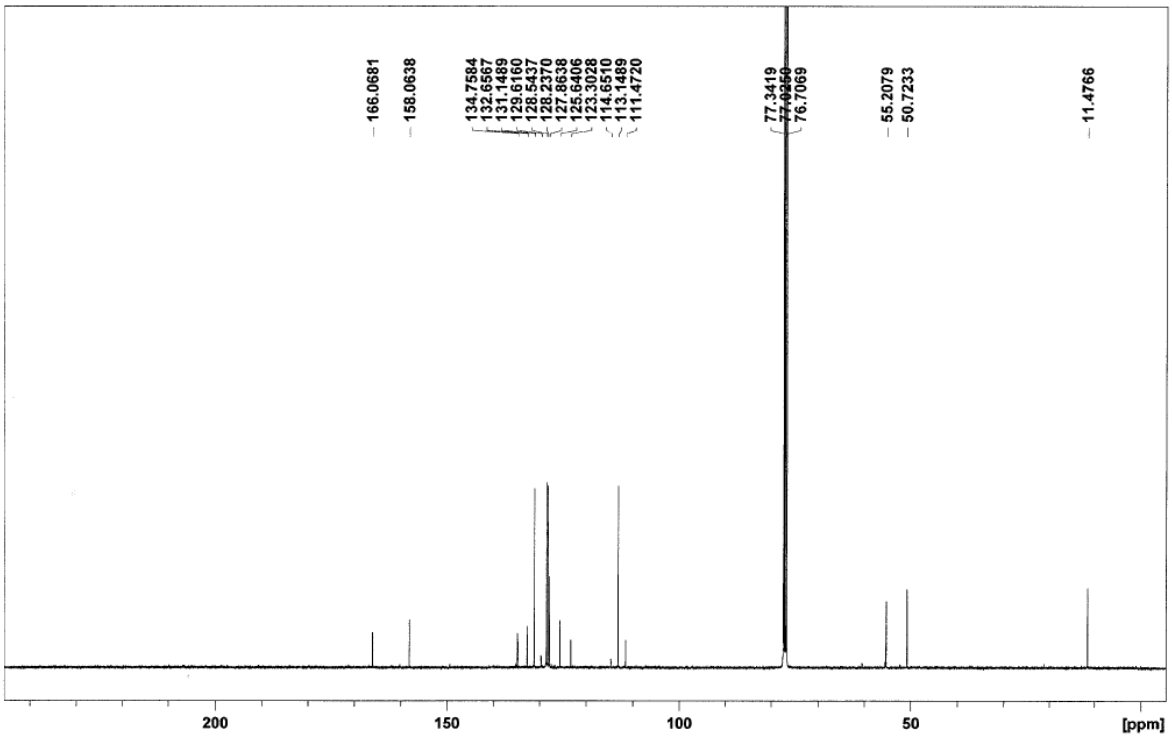

3j
HN
Me
Ph
MeOOC
Ph
8.227
7.542
7.538
7.532
7.527
7.522
7.519
7.511
7.508
7.425
7.420
7.416
7.404
7.385
7.380
7.376
7.372
7.365
7.358
7.350
7.333
7.315
7.309
7.304
7.301
7.294
7.287
7.268
7.259
3.789
2.427
1.573
0.000
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
4.15
6.22
3.13
3.14
164.8005
135.8247
133.5506
131.8034
131.3160
128.8481
128.4128
128.2562
128.2452
127.5227
124.4137
112.5297
104.6731
92.5004
83.7224
77.3448
77.0380
76.7100
51.0550
11.9902
200
150
100
50
[ppm]

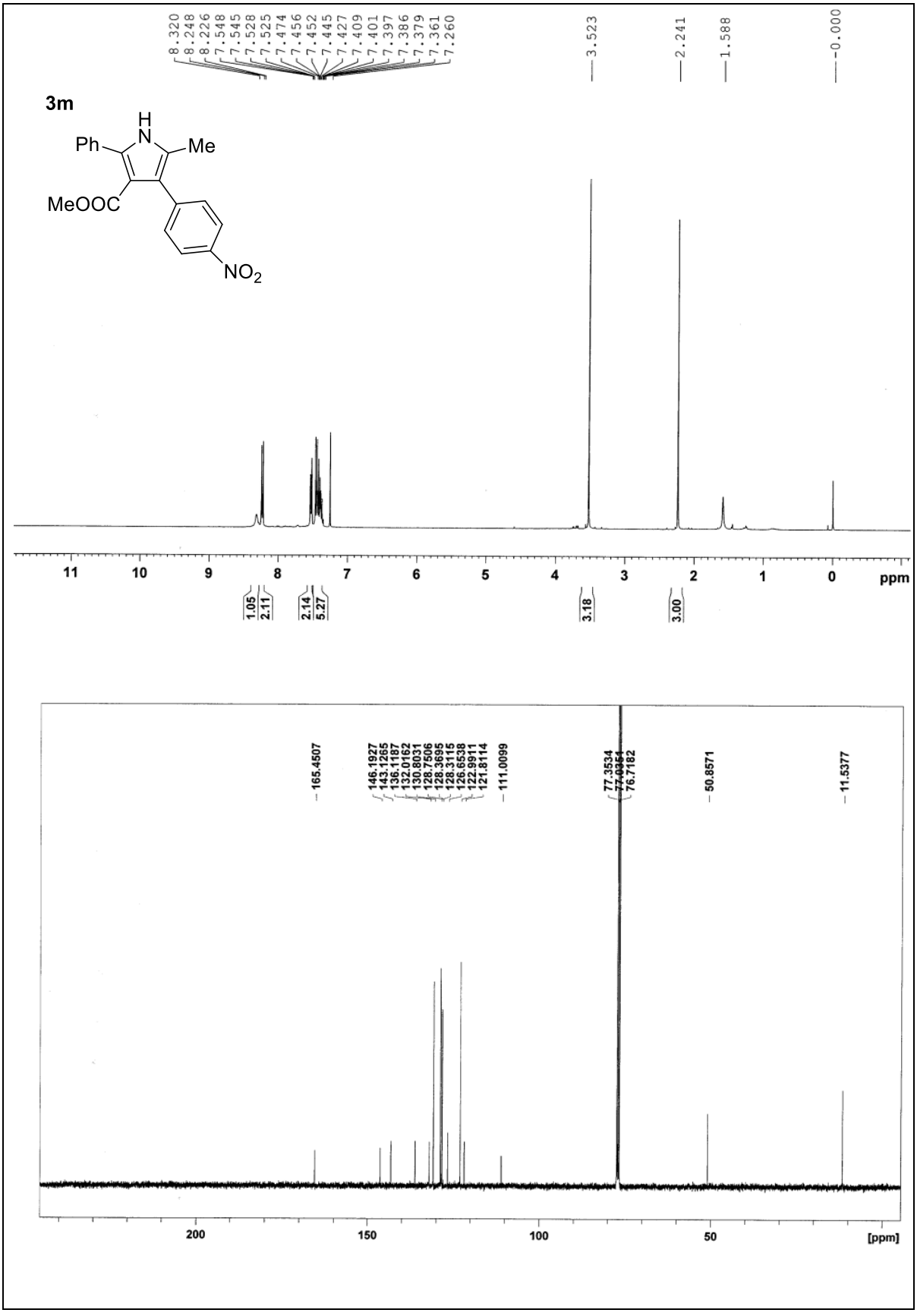

8.160
7.553
7.549
7.544
7.531
7.430
7.427
7.422
7.409
7.405
7.396
7.390
7.378
7.364
7.359
7.351
7.345
7.339
7.324
7.321
7.315
7.303
7.300
7.284
7.279
7.274
7.265
7.258
3.512
2.226
1.569
0.000
3k
H
N
Ph
Me
MeOOC
Ph
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.07
5.02
3.10
3.12
3.12
166.0347
135.6040
134.8810
132.5313
130.0811
128.5582
128.2663
127.9292
127.6614
126.1828
125.7551
123.6860
111.5058
77.3436
77.0266
76.7086
50.7281
11.5053
200
150
100
50
[ppm]

3l

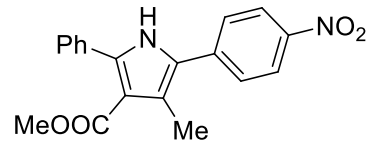

H
N
Ph
Me
MeOOC
OMe

8.132
7.539
7.535
7.517
7.515
7.404
7.385
7.258
7.253
7.232
6.938
6.916
3.843
3.525
2.212
1.570
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.94
2.08
2.14
1.05
2.07
2.07
3.11
2.96
3.00

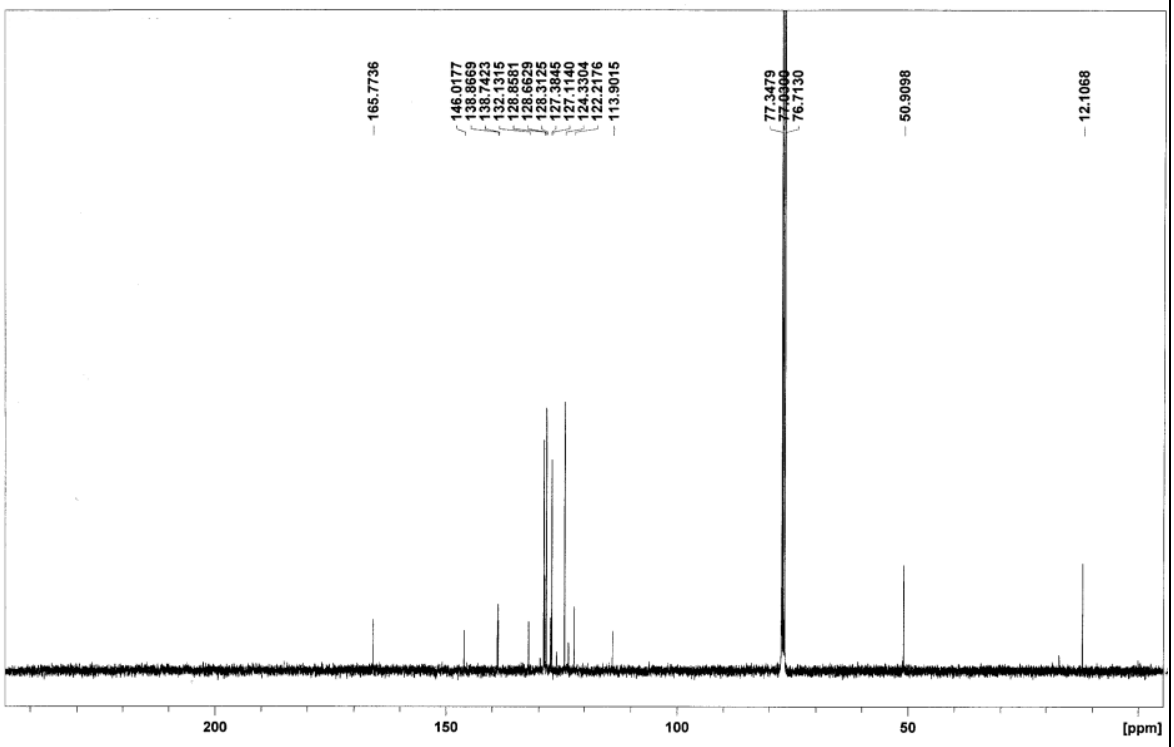

166.0681
158.0638
134.7584
132.6567
131.1489
129.6160
128.5437
128.2370
127.8638
125.6406
123.3028
114.6510
113.1489
111.4720
77.3419
77.0250
76.7069
55.2079
50.7233
11.4766
200 150 100 50 [ppm]

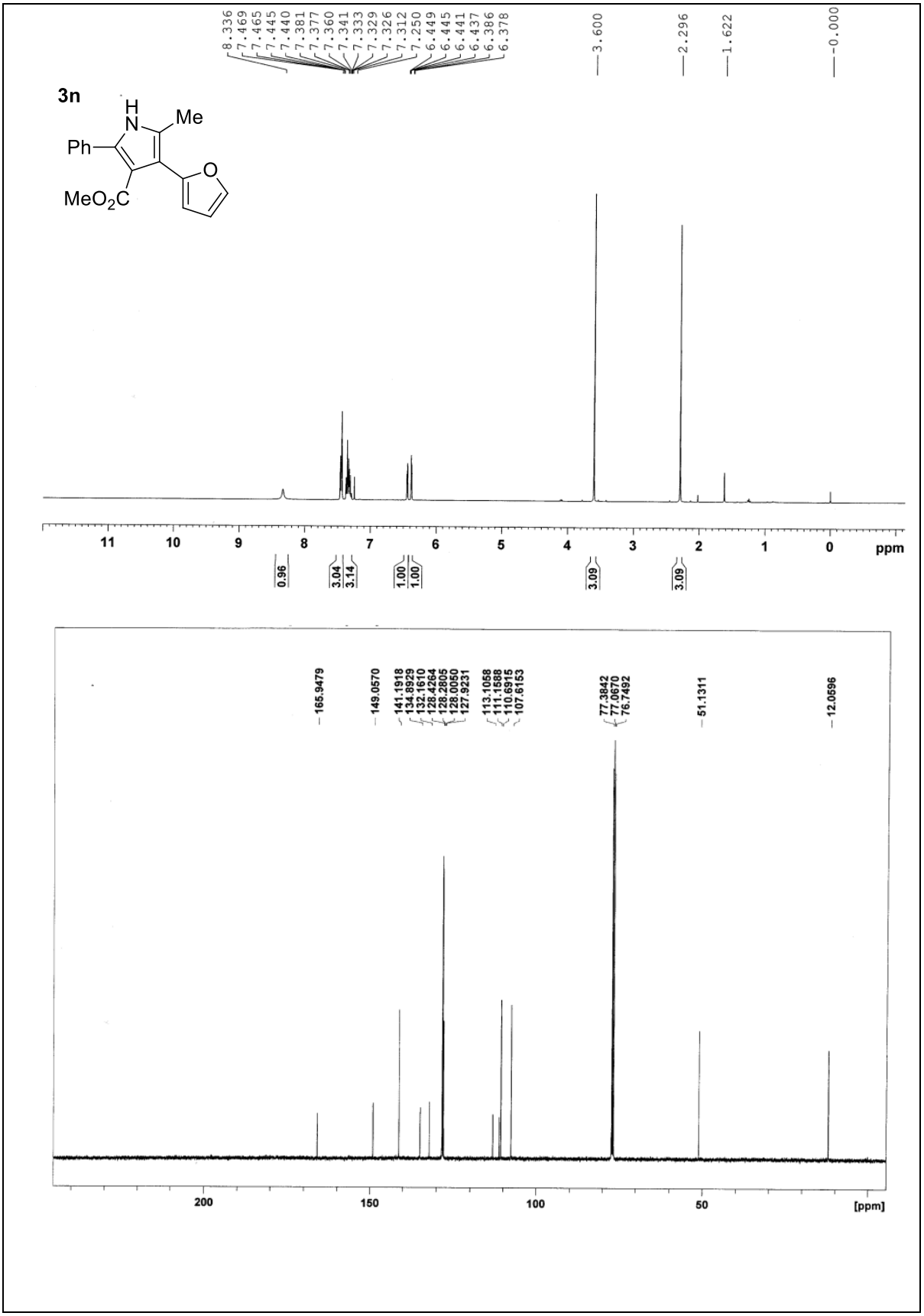

3m
Ph
N
H
Me
MeOOC
NO2
8.320
8.248
8.226
7.548
7.545
7.528
7.525
7.474
7.456
7.452
7.445
7.427
7.409
7.401
7.397
7.386
7.379
7.361
7.260
3.523
2.241
1.588
0.000
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.05
2.11
2.14
5.27
3.18
3.00
165.4507
146.1927
143.1265
136.1187
132.0162
130.8031
128.7506
128.3695
128.3115
126.6538
122.9911
121.8114
111.0099
77.3534
77.0351
76.7182
50.8571
11.5377
200
150
100
50
[ppm]

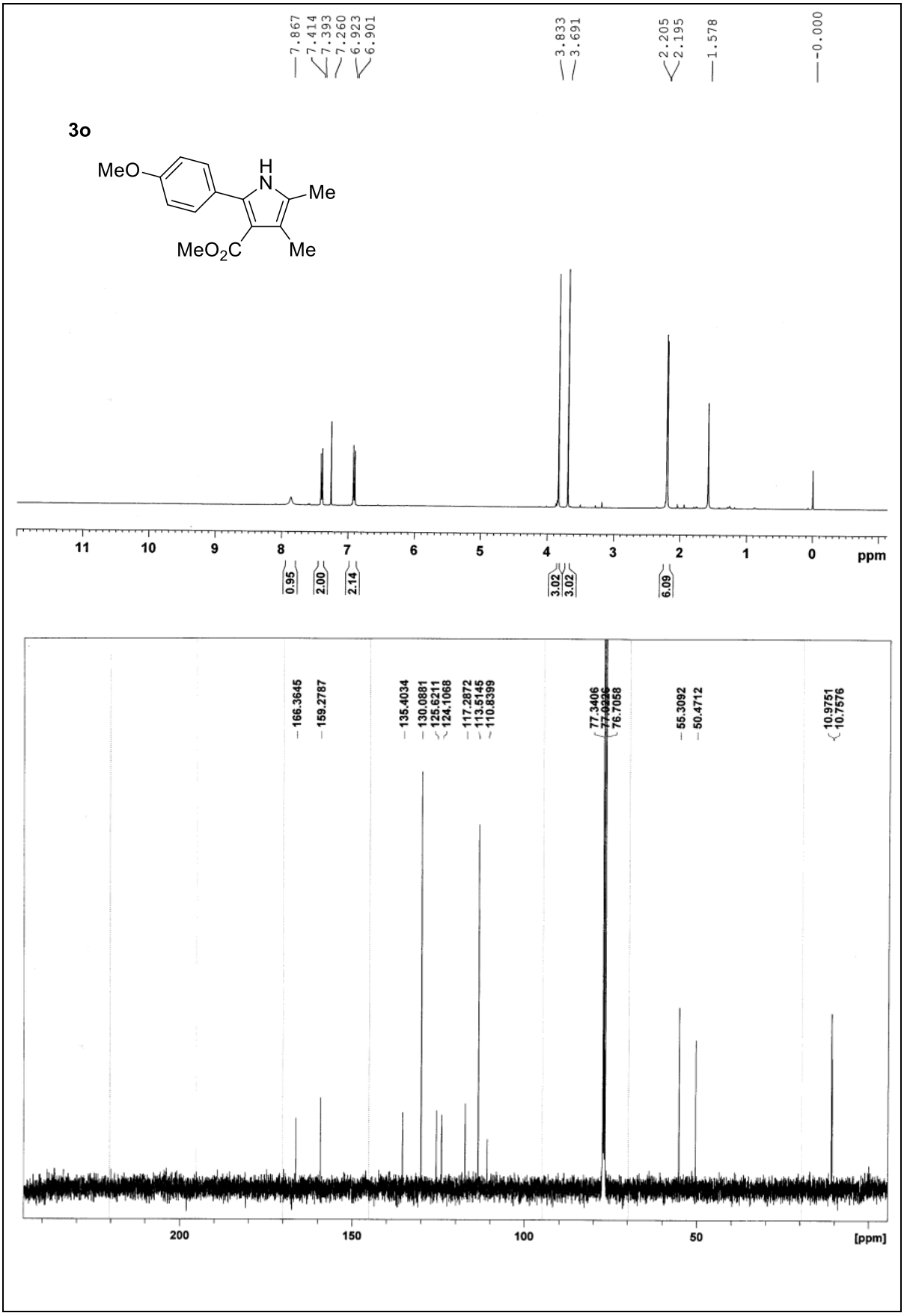
3mm

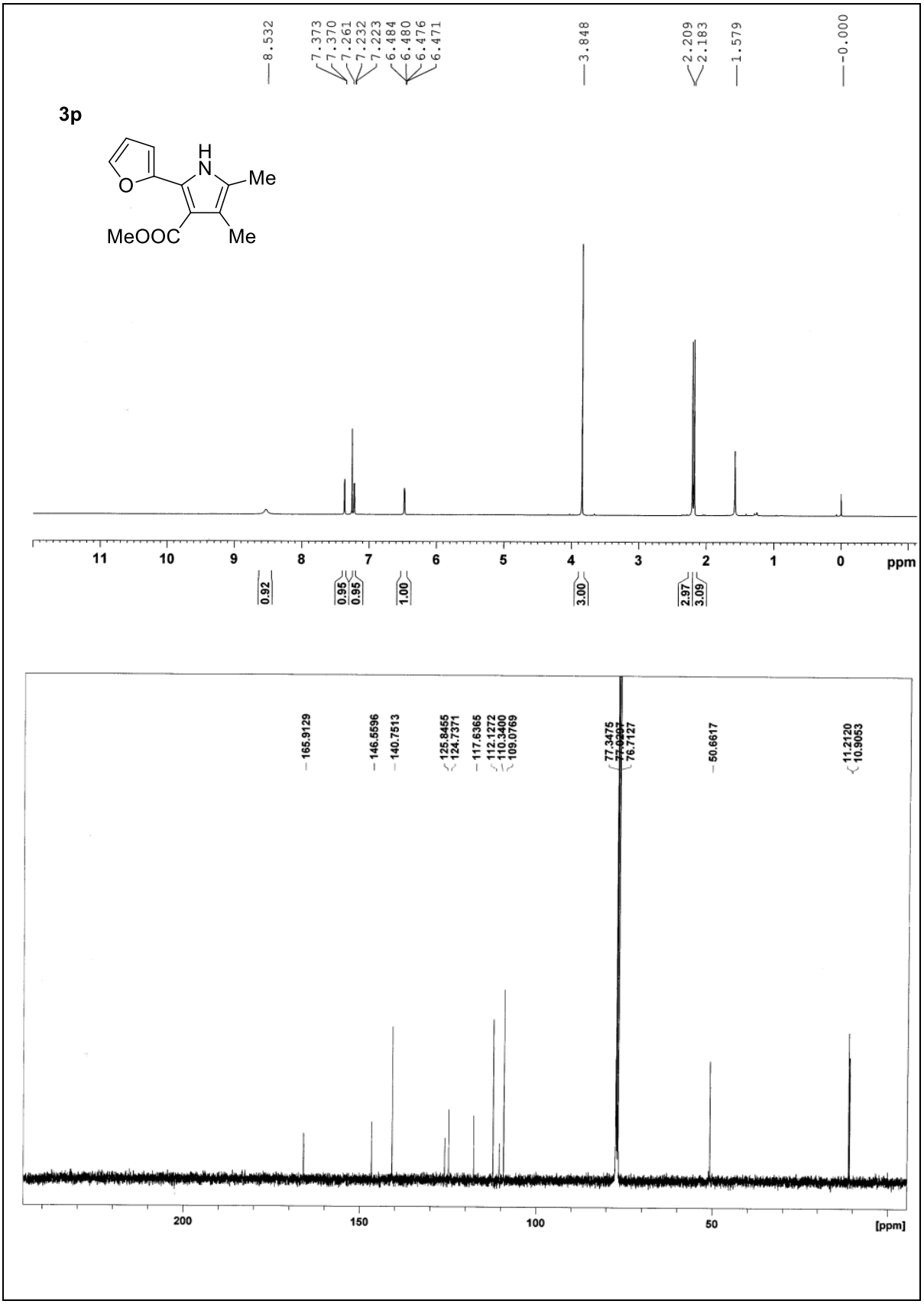

H
N
Ph
NO2
MeOOC
Me

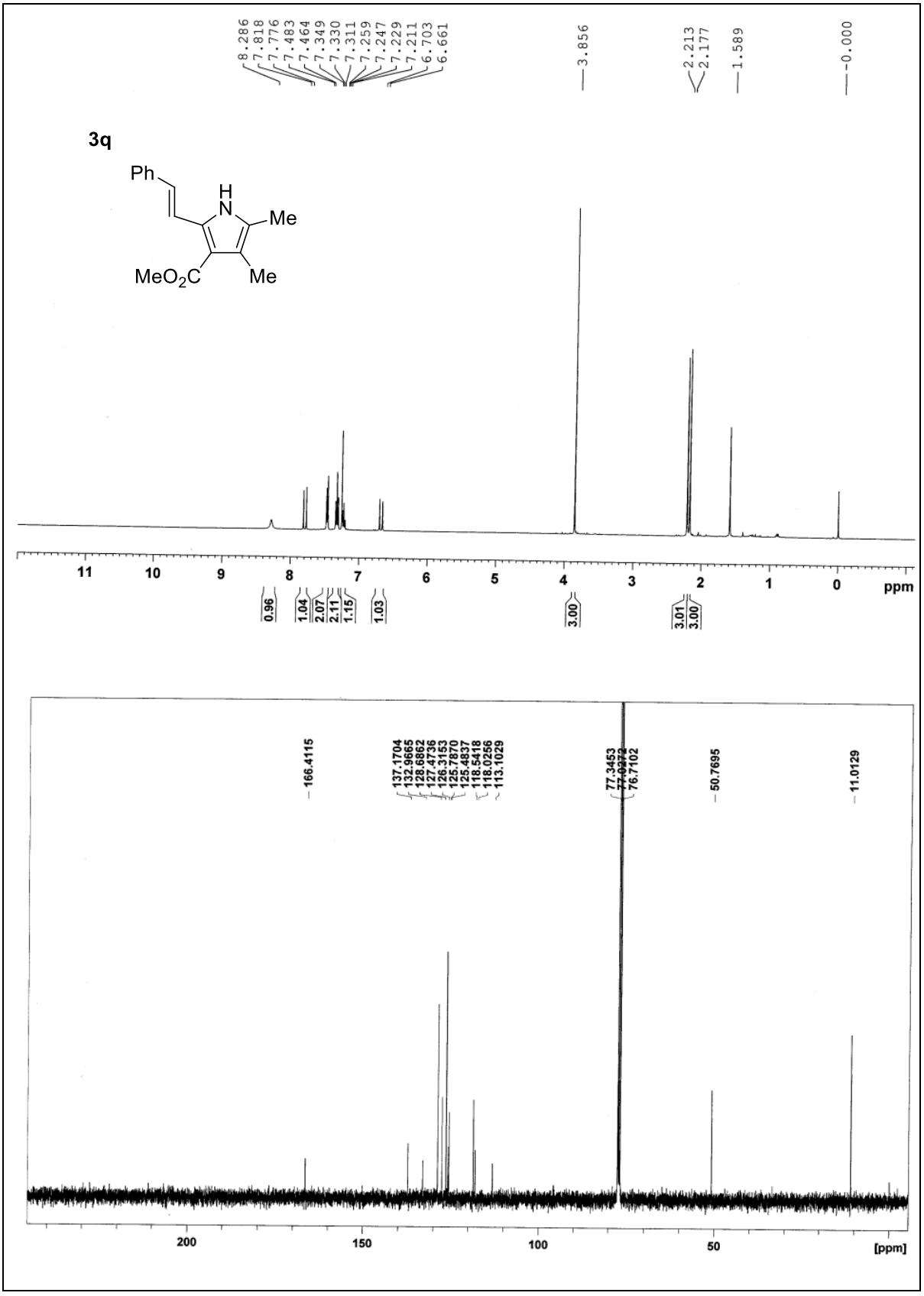
8.451
8.298
8.276
7.600
7.578
7.514
7.511
7.432
7.413
7.261
3.720
2.487
1.580
0.000
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.08
2.09
2.03
3.29
3.03
3.02

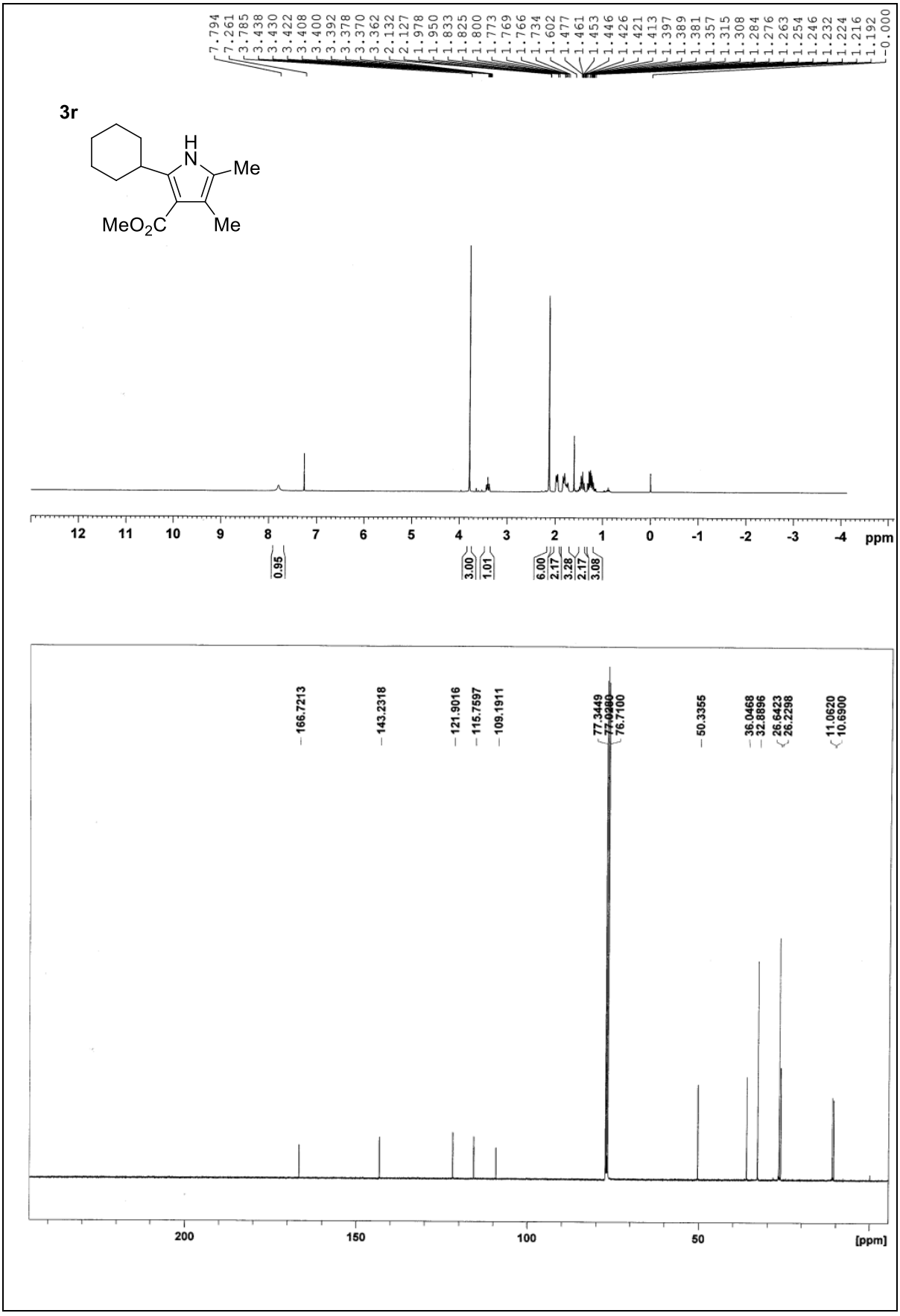

165.7736
146.0177
138.8669
138.7423
132.1315
128.8581
128.6629
128.3125
127.3845
127.1140
124.3304
122.2176
113.9015
77.3479
77.0000
76.7130
50.9098
12.1068
200
150
100
50
[ppm]


3n
H
N
Me
Ph
O
MeO2C
8.336
7.469
7.465
7.445
7.440
7.381
7.377
7.360
7.341
7.333
7.329
7.326
7.312
7.250
6.449
6.445
6.441
6.437
6.386
6.378
3.600
2.296
1.622
0.000
ppm
0.96
3.04
3.14
1.00
1.00
3.09
3.09
165.9479
149.0570
141.1918
134.8929
132.1610
128.4264
128.2805
128.0050
127.9231
113.1058
111.1588
110.6915
107.6153
77.3842
77.0670
76.7492
51.1311
12.0596
[ppm]

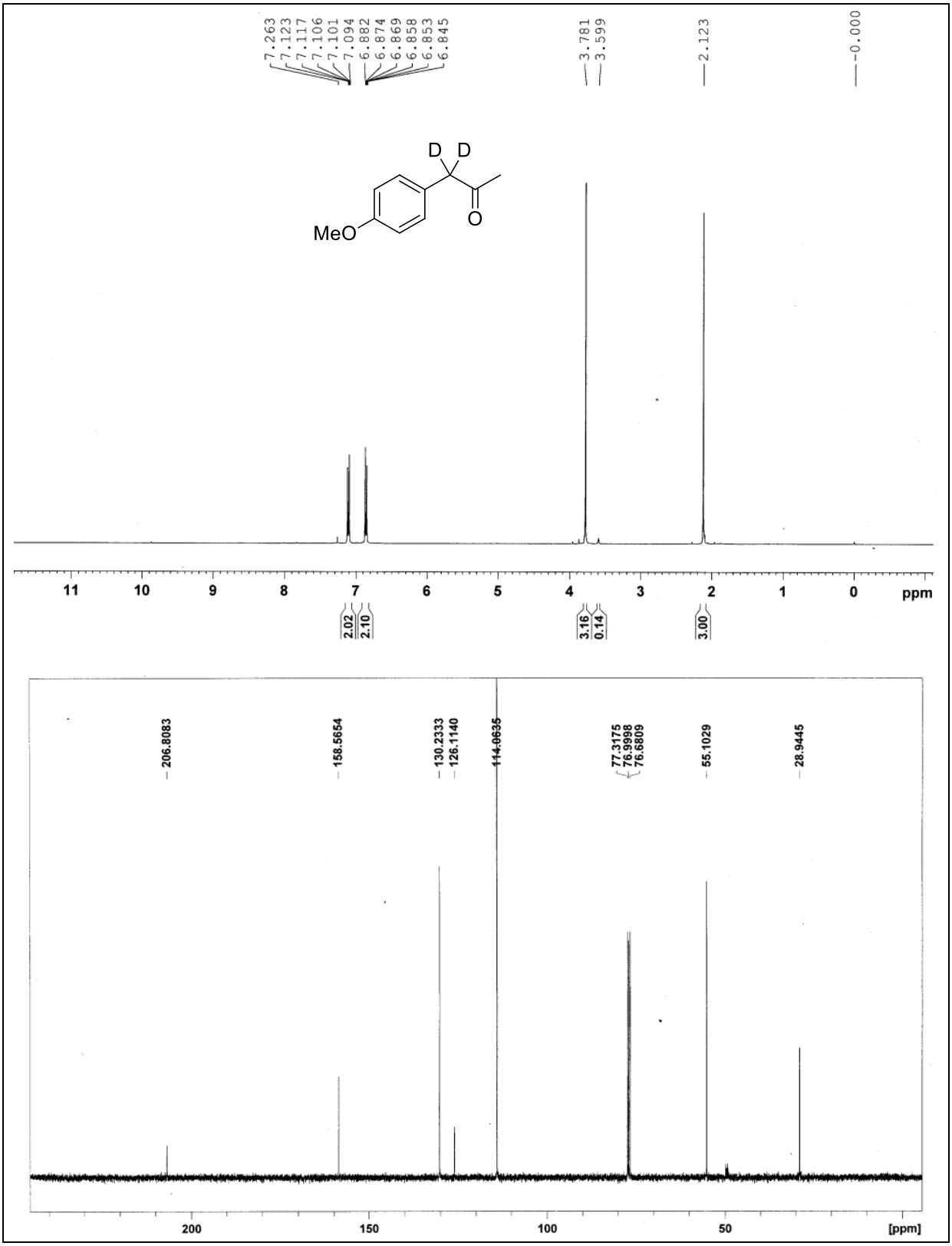

7.867
7.414
7.393
7.260
6.923
6.901
3.833
3.691
2.205
2.195
1.578
-0.000
3o
MeO
H
N
Me
MeO2C
Me
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.95
2.00
2.14
3.02
3.02
6.09
166.3645
159.2787
135.4034
130.0881
125.6211
124.1068
117.2872
113.5145
110.8399
77.3406
77.0226
76.7058
55.3092
50.4712
10.9751
10.7576
200
150
100
50
[ppm]

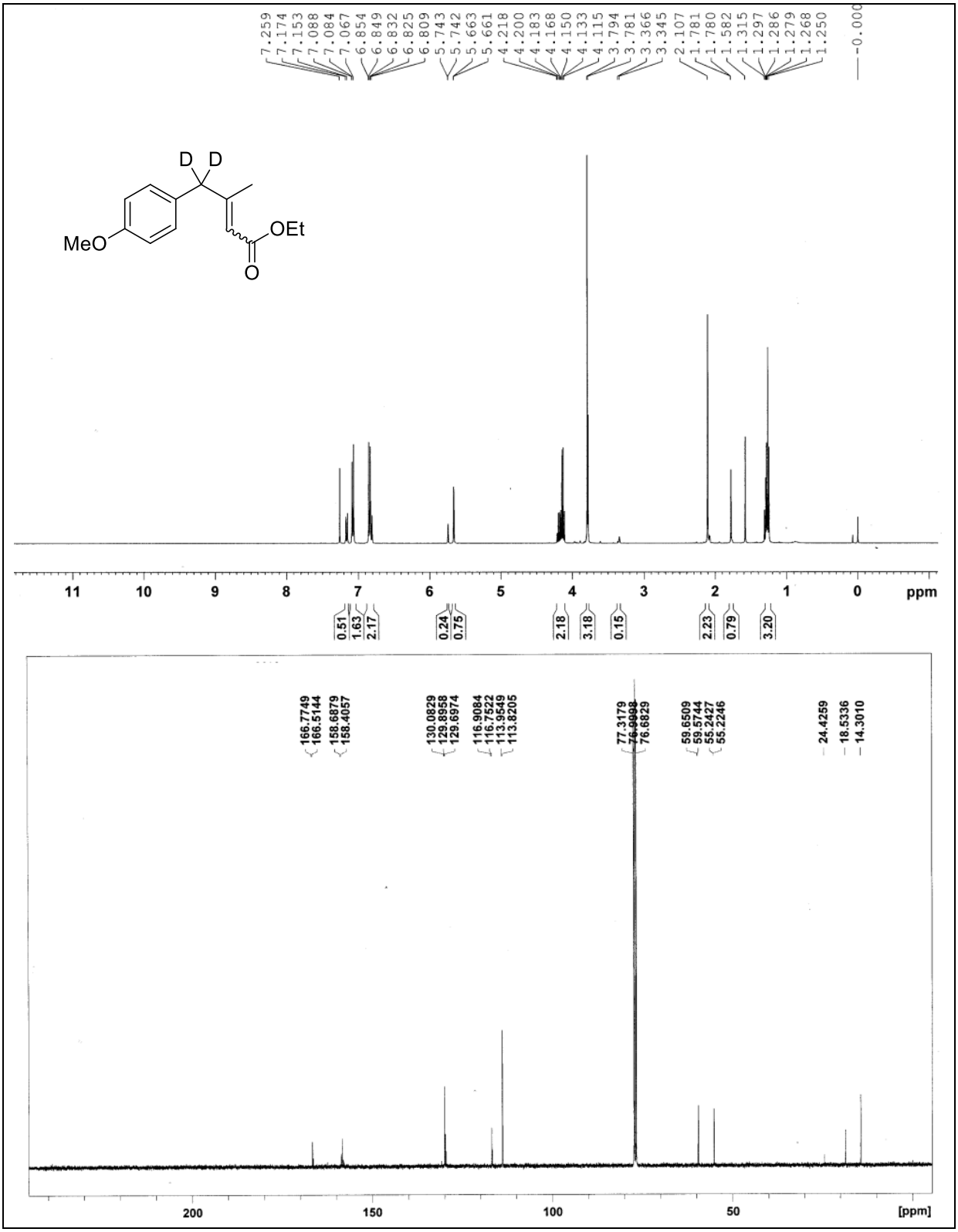

3p
8.532
7.373
7.370
7.261
7.232
7.223
6.484
6.480
6.476
6.471
3.848
2.209
2.183
1.579
0.000
Me
Me
MeOOC
ppm
0.92
0.95
0.95
1.00
3.00
2.97
3.09
165.9129
146.5596
140.7513
125.8455
124.7371
117.6365
112.1272
110.3400
109.0769
77.3475
77.0307
76.7127
50.6617
11.2120
10.9053
[ppm]

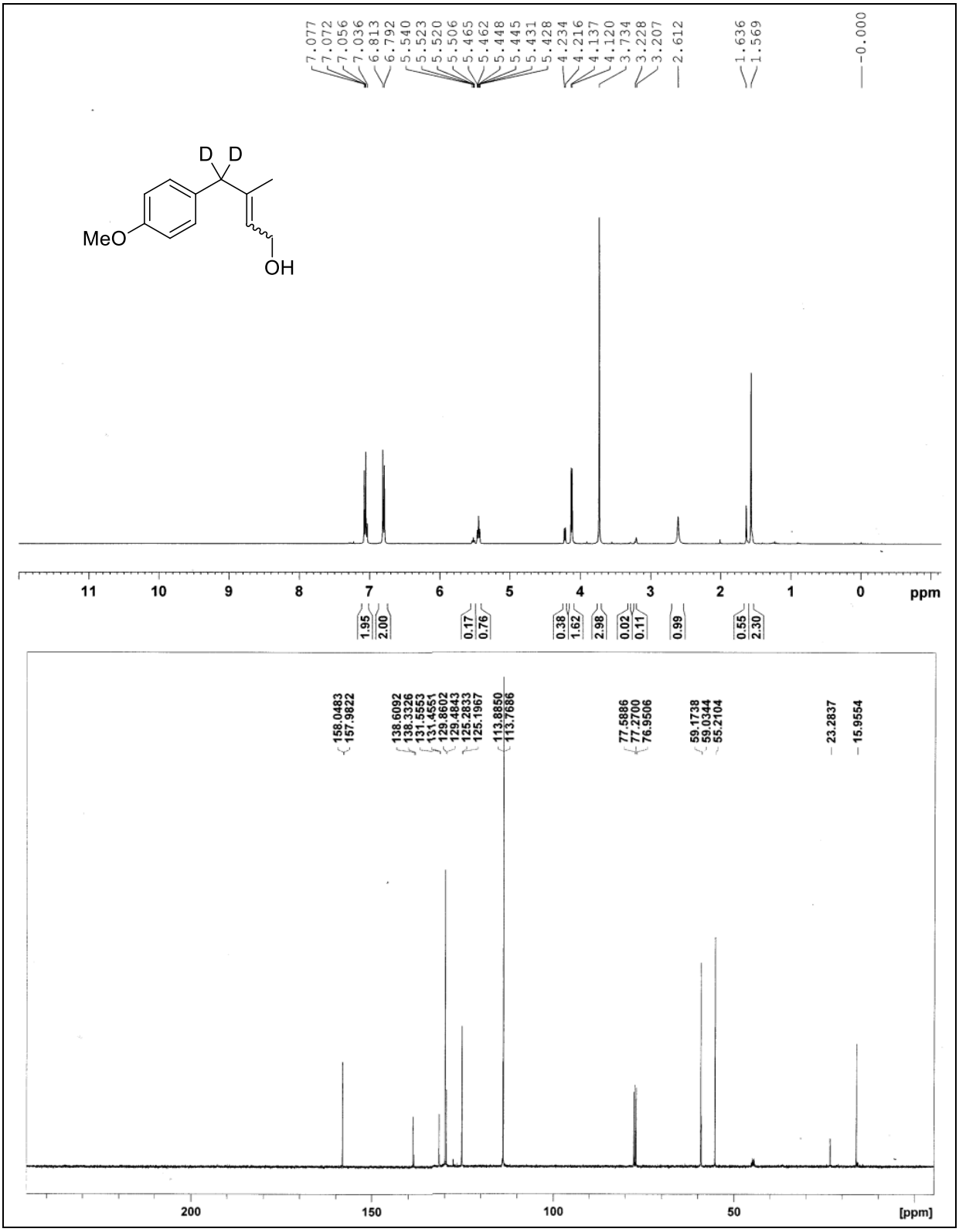

3q
Ph
H
N
Me
MeO2C
Me
8.286
7.818
7.776
7.483
7.464
7.349
7.330
7.311
7.259
7.247
7.229
7.211
6.703
6.661
3.856
2.213
2.177
1.589
0.000
ppm
0.96
1.04
2.07
2.11
1.15
1.03
3.00
3.01
3.00
166.4115
137.1704
132.9665
128.6862
127.4736
126.3153
125.7870
125.4837
118.5418
118.0256
113.1029
77.3453
77.0272
76.7102
50.7695
11.0129
[ppm]

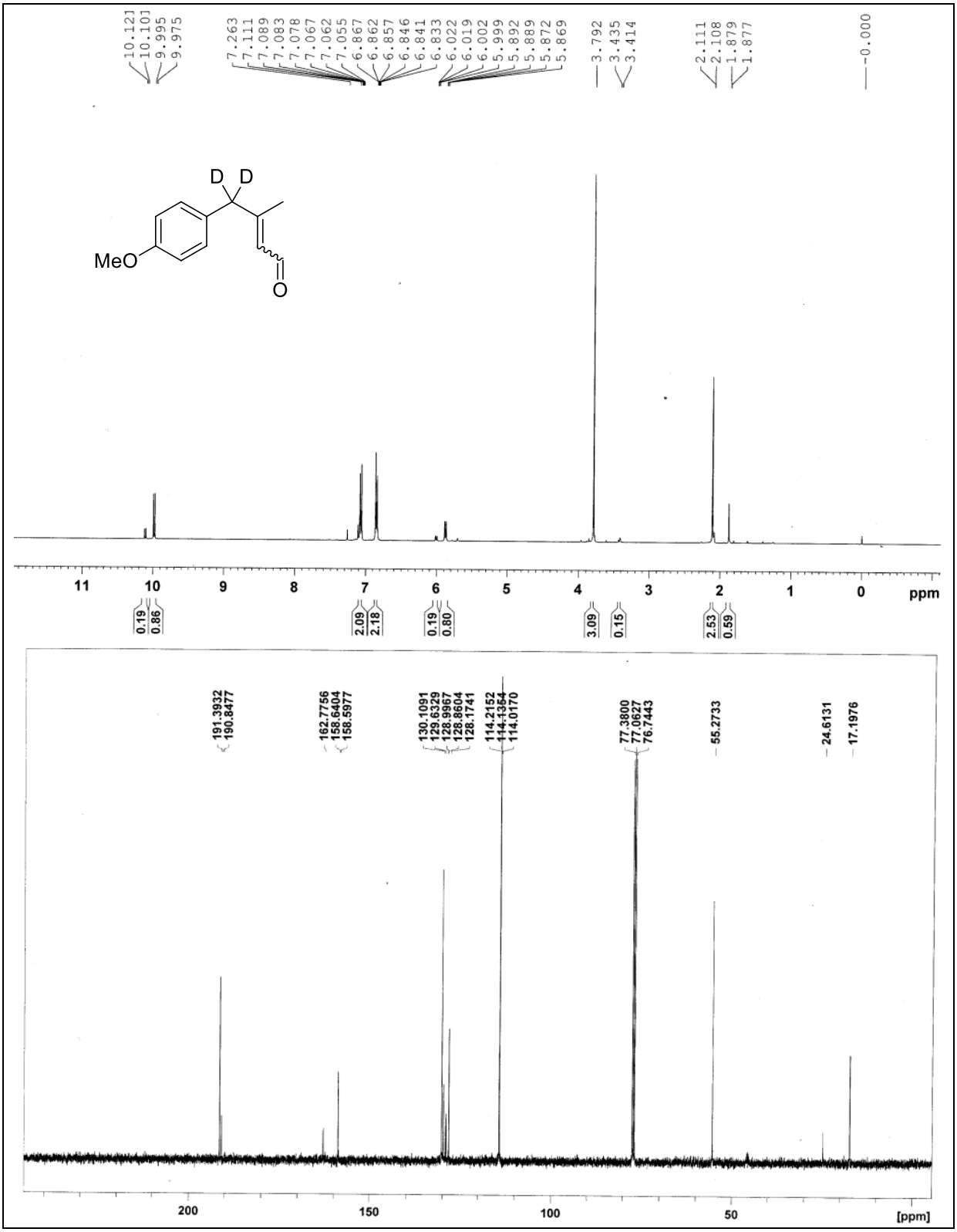

7.794
7.261
3.785
3.438
3.430
3.422
3.408
3.400
3.392
3.378
3.370
3.362
2.132
2.127
1.978
1.950
1.833
1.825
1.800
1.773
1.769
1.766
1.734
1.602
1.477
1.461
1.453
1.446
1.426
1.421
1.413
1.397
1.389
1.381
1.357
1.315
1.308
1.284
1.276
1.263
1.254
1.246
1.232
1.224
1.216
1.192
0.000
3r
H
N
Me
MeO2C
Me
12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4 ppm
0.95
3.00
1.01
6.00
2.17
3.28
2.17
3.08
166.7213
143.2318
121.9016
115.7597
109.1911
77.3449
77.0280
76.7100
50.3355
36.0468
32.8896
26.6423
26.2298
11.0620
10.6900
200 150 100 50 [ppm]

^{1}H and ^{13}C NMR spectra of deuterium labeling experiment




7.259
7.174
7.153
7.088
7.084
7.067
6.854
6.849
6.832
6.825
6.809
5.743
5.742
5.663
5.661
4.218
4.200
4.183
4.168
4.150
4.133
4.115
3.794
3.781
3.366
3.345
2.107
1.781
1.780
1.582
1.315
1.297
1.286
1.279
1.268
1.250
0.000
D D
MeO
OEt
O
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.51 1.63 2.17 0.24 0.75 2.18 3.18 0.15 2.23 0.79 3.20
166.7749
166.5144
158.6879
158.4057
130.0829
129.8958
129.6974
116.9084
116.7522
113.9549
113.8205
77.3179
76.8829
59.6509
59.5744
55.2427
55.2246
24.4259
18.5336
14.3010
200 150 100 50 [ppm]


7.077
7.072
7.056
7.036
6.813
6.792
5.540
5.523
5.520
5.506
5.465
5.462
5.448
5.445
5.431
5.428
4.234
4.216
4.137
4.120
3.734
3.228
3.207
2.612
1.636
1.569
0.000
D D
MeO
OH
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.95 2.00 0.17 0.76 0.38 1.62 2.98 0.02 0.11 0.99 0.55 2.30
158.0483
157.9822
138.6092
138.3326
131.5553
131.4551
129.8602
129.4843
125.2833
125.1967
113.8850
113.7686
77.5886
77.2700
76.9506
59.1738
59.0344
55.2104
23.2837
15.9554
200 150 100 50 [ppm]


10.121
10.101
9.995
9.975
7.263
7.111
7.089
7.083
7.078
7.067
7.062
7.055
6.867
6.862
6.857
6.846
6.841
6.833
6.022
6.019
6.002
5.999
5.892
5.889
5.872
5.869
3.792
3.435
3.414
2.111
2.108
1.879
1.877
0.000
D D
MeO
O
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.19 0.86 2.09 2.18 0.19 0.80 3.09 0.15 2.53 0.59
191.3932
190.8477
162.7756
158.6404
158.5977
130.1091
129.6329
128.9967
128.8604
128.1741
114.2152
114.1364
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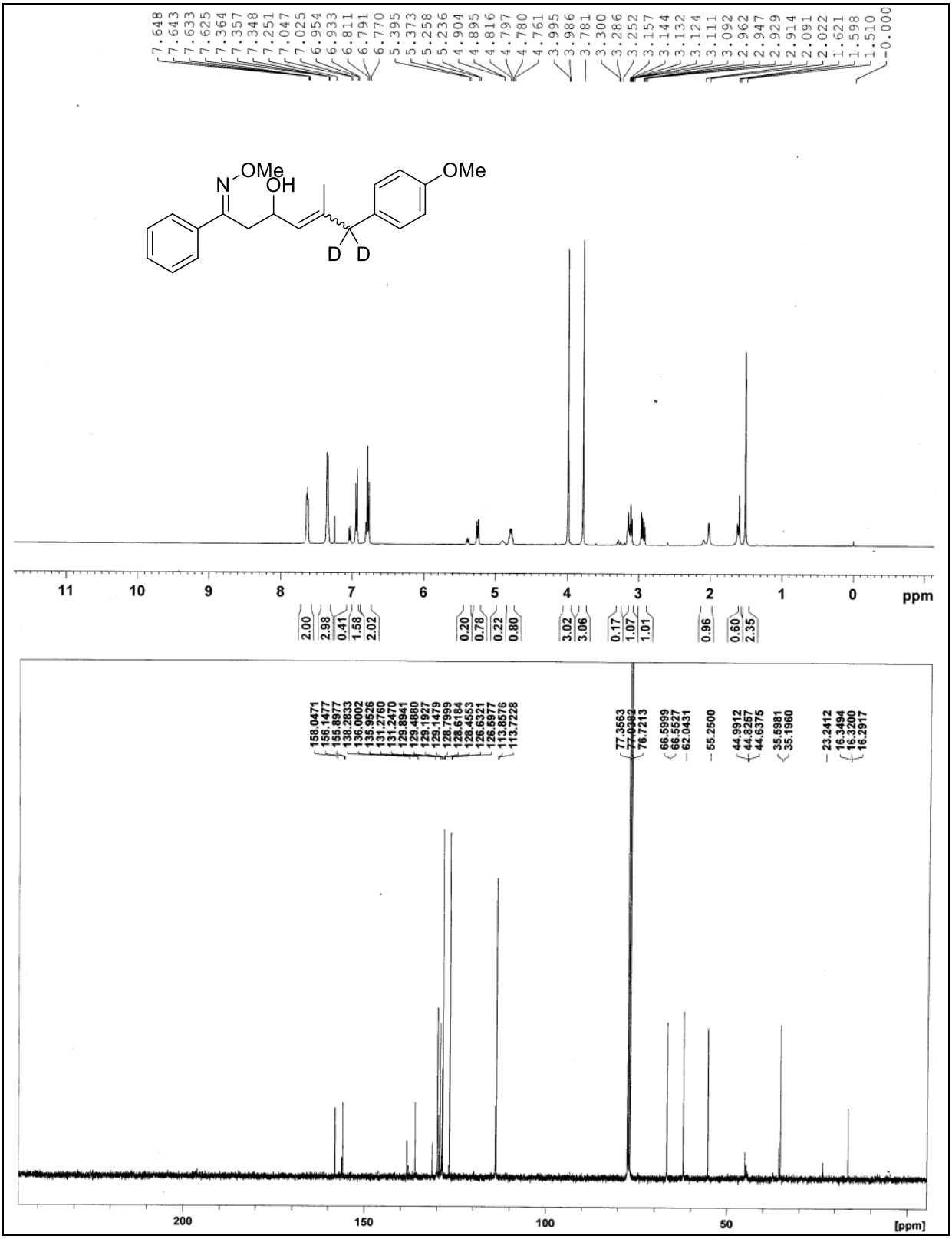
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5.236
4.904
4.895
4.816
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4.780
4.761
3.995
3.986
3.781
3.300
3.286
3.252
3.157
3.144
3.132
3.124
3.111
3.092
2.962
2.947
2.929
2.914
2.091
2.022
1.621
1.598
1.510
-0.000
N
OMe
OH
OMe
D
D
11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.00
2.98
0.41
1.58
2.02
0.20
0.78
0.22
0.80
3.02
3.06
0.17
1.07
1.01
0.96
0.60
2.35
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155.8977
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76.7213
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66.5527
62.0431
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44.9912
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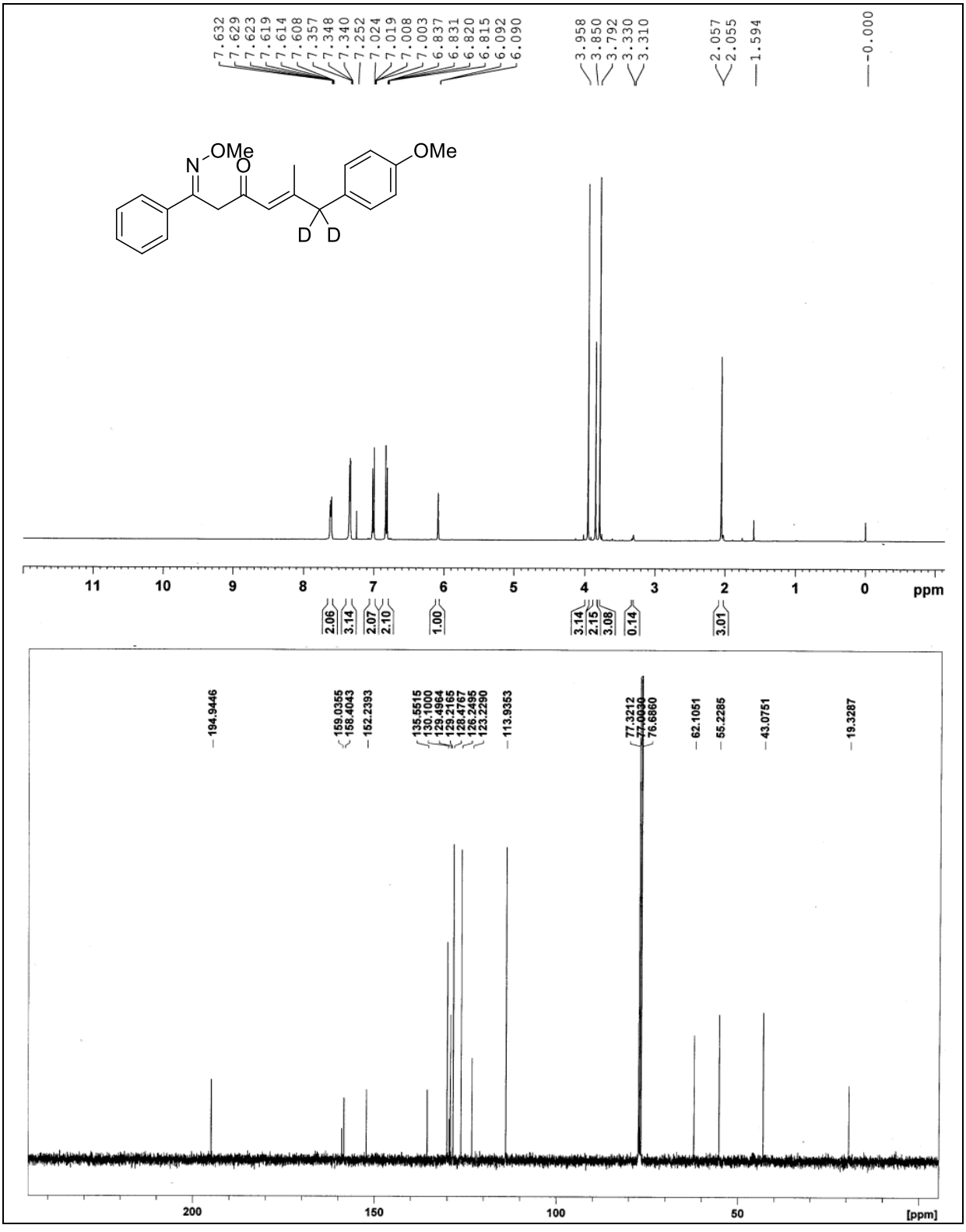
50
[ppm]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7.008
7.003
6.837
6.831
6.820
6.815
6.092
6.090
3.958
3.850
3.792
3.330
3.310
2.057
2.055
1.594
0.000
OMe
N
O
OMe
D D
11 10 9 8 7 6 5 4 3 2 1 0 ppm
2.06 3.14 2.07 2.10 1.00 3.14 2.15 3.08 0.14 3.01
194.9446
159.0355
158.4043
152.2393
135.5515
130.1000
129.4964
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128.4767
126.2495
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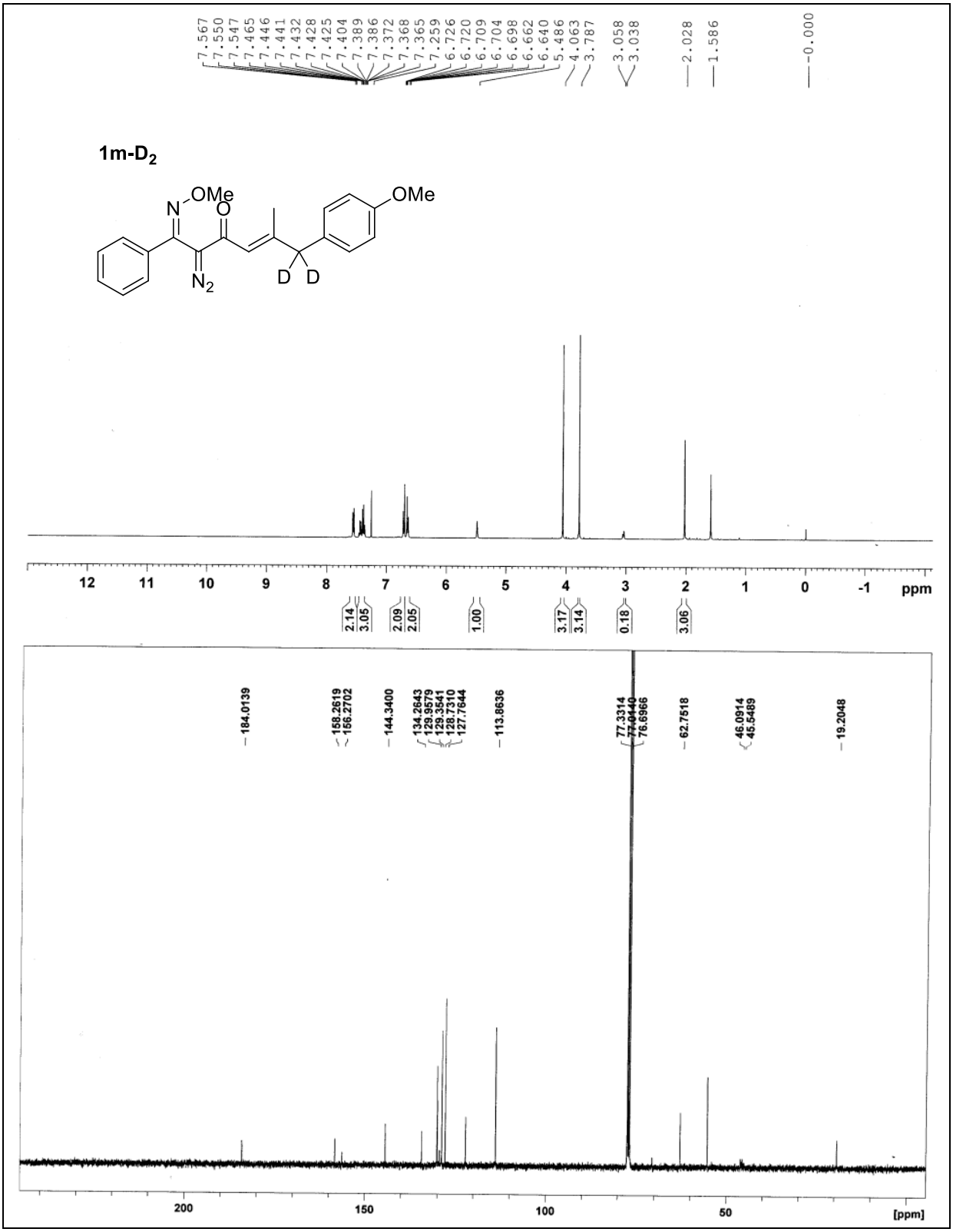
200 150 100 50 [ppm]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7.386
7.372
7.368
7.365
7.259
6.726
6.720
6.709
6.704
6.698
6.662
6.640
5.486
4.063
3.787
3.058
3.038
2.028
1.586
0.000
1m-D2
N
OMe
O
N2
D
D
OMe
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
2.14
3.05
2.09
2.05
1.00
3.17
3.14
0.18
3.06
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156.2702
144.3400
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129.9579
129.3541
128.7310
127.7644
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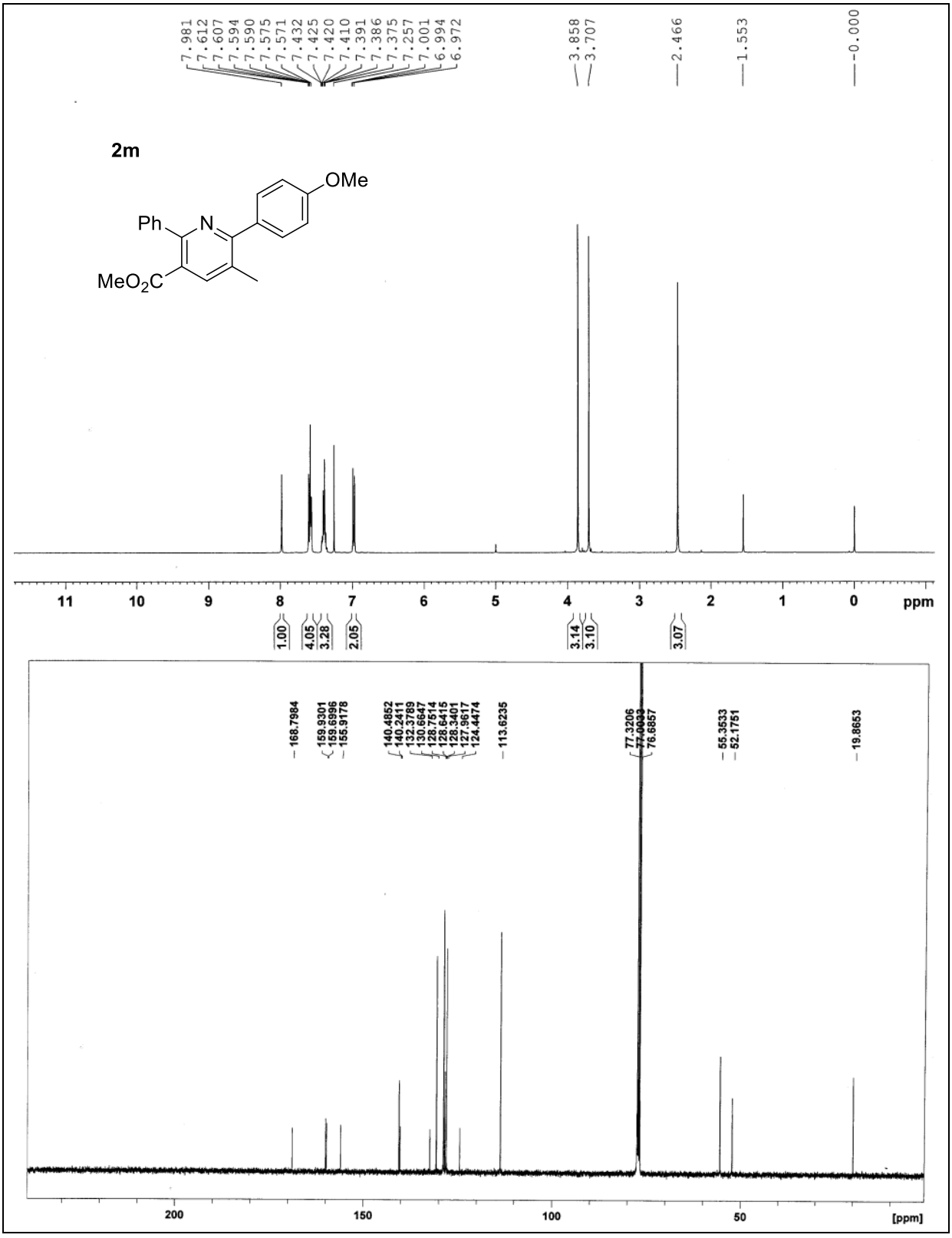
100
50
[ppm]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7.432
7.425
7.420
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7.386
7.375
7.257
7.001
6.994
6.972
3.858
3.707
2.466
1.553
0.000
2m
OMe
Ph
N
MeO2C
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
4.05
3.28
2.05
3.14
3.10
3.07
168.7984
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159.6996
155.9178
140.4852
140.2411
132.3789
130.6647
128.7514
128.6415
128.3401
127.9617
124.4474
113.6235
77.3206
77.0033
76.6857
55.3533
52.1751
19.8653
200
150
100
50
[ppm]