Supporting information

Unexpected C-C Bond Cleavage of Epoxide Motif: Rhodium(I)-Catalyzed Tandem Heterocyclization/[4+1] Cycloaddition of 1-(1-Alkynyl)oxiranyl Ketones.

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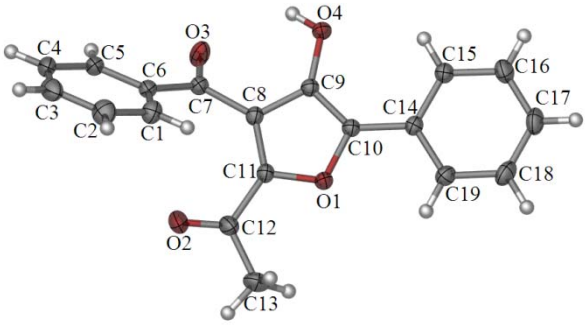
General Information

^{1}H NMR, ^{13}C NMR, ^{19}F NMR spectra were measured at 300 MHz or 400 MHz and 75 MHz or 100 MHz, 376 MHz in $CDCl_3$. Splitting patterns of an apparent multiplet associated with an averaged coupling constant were designed as s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), and br (broadened).

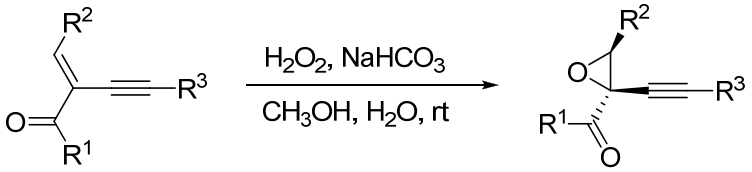

O4
O3
C4
C5
C6
C7
C8
C9
C15
C16
C17
C3
C2
C1
C14
C10
C11
O1
C18
C19
O2
C12
C13

4m

General procedure for synthesis of substrates

2-(1-Alkynyl)-2-alken-1-ones were prepared according to the reference.[1]

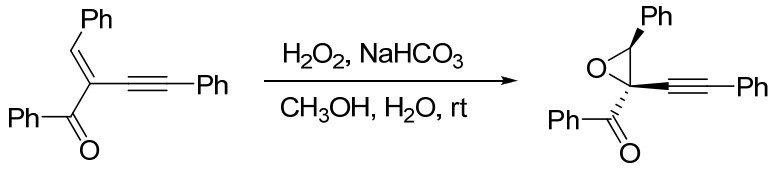
Typical procedure for synthesis of substrates 1.



2-(1-Alkynyl)-2-alken-1-ones **1**

To the solution of the 2-(1-Alkynyl)-2-alken-1-ones (5 mmol) in 200 mL of CH_3OH, a solution of $NaHCO_3$ (2.1 g, 25 mmol) in 20 mL of H_2O was added. The mixtured solution was stired at rt for 10 minutes, then H_2O_2 (30% in water, 5 mL, 50 mmol) was added, and the resulting mixture was stirred at rt until the reaction was completed (monitored by TLC). Then the reaction mixture was extracted with CH_2Cl_2 (3 × 50 mL). The combined organic layers were washed with saturated NaS_2O_3 solution (30 mL), dried over magnesium sulfate and concentrated under vaccuo. The crude residue was purified by flash chromatography on silica gel (hexanes/EtOAc = 10 : 1) to give the desired products.

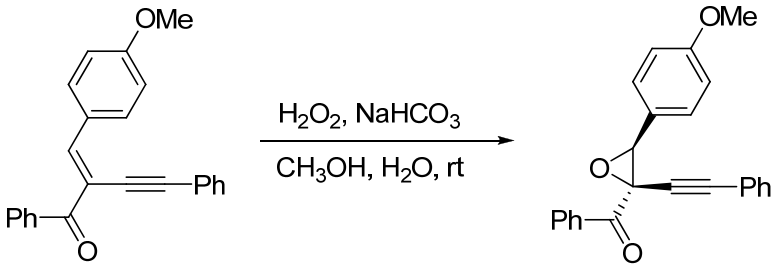
1. **phenyl((2R*,3S*)-3-phenyl-2-(phenylethynyl)oxiran-2-yl)methanone (1a).**



1a

The reaction of (*E*)-2-benzylidene-1,4-diphenylbut-3-yn-1-one (1.54 g, 5 mmol) in 200 mL of CH_3OH, $NaHCO_3$ (2.1 g, 25 mmol) in 20 mL of H_2O and H_2O_2 (30% in water, 5 mL , 50 mmol) at rt for 12 h afforded 1.07 g of **1a** (66% yield) as a white solid. m.p. 118 - 119 ^oC; ^1H NMR (300 MHz, $CDCl_3$): δ = 8.15 (d, 2 H, *J* = 7.5 Hz); 7.64 - 7.52 (m, 3 H); 7.52 - 7.40 (m, 5 H); 7.31 - 7.15 (m, 5 H); 4.37 (s, 1 H). ^{13}C NMR (75.4 MHz, $CDCl_3$): δ = 190.86, 133.97, 133.95, 132.80, 131.78, 129.58, 129.14, 128.53, 128.19, 128.10, 127.21, 121.24, 89.69, 82.16, 64.68, 61.22 ppm; IR (neat): $\tilde{\nu}$ = 3085, 2966, 2231, 1690, 1490, 1449, 1263, 960, 889, 853, 751, 692 cm^{-1}; MS (EI, 70 ev) m/z (%): 324 (M^+, 4.63), 105 (100). Anal calcd for $C_{23}H_{16}O_2$: C, 85.16; H, 4.97; found: C, 85.20; H, 5.24.

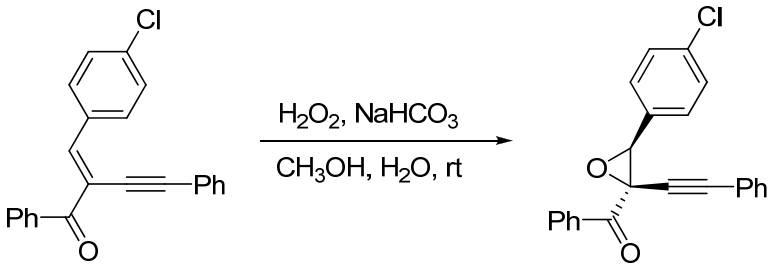
2. ((2R*,3S*)-3-(4-methoxyphenyl)-2-(phenylethynyl)oxiran-2-yl)(phenyl)methanone (1b).



1b

The reaction of (*E*)-2-(4-methoxybenzylidene)-1,4-diphenylbut-3-yn-1-one (1.69 g, 5 mmol) in 200 mL of CH_3OH, $NaHCO_3$ (2.1 g, 25 mmol) in 20 mL of H_2O and H_2O_2 (30% in water, 5 mL , 50 mmol) at rt for 10 h afforded 1.22 g of **1b** (69 % yield) as a yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.14 (d, 2 H, *J* = 7.5 Hz); 7.60 (t, 2 H, *J* = 7.2 Hz); 7.52 - 7.46 (m, 4 H); 7.32 - 7.20 (m, 5 H); 6.97 (d, 2 H, *J* = 8.7 Hz); 4.31 (s, 1 H); 3.84 (s, 3 H). ^{13}C NMR (75.4 MHz, $CDCl_3$): δ = 191.05, 160.43, 134.10, 133.86, 131.77, 129.55, 129.10, 128.66, 128.49, 128.21, 124.76, 121.38, 113.62, 89.79, 82.45, 64.76, 61.35, 55.32 ppm; IR (neat): $\tilde{\nu}$ = 3062, 3034, 3003, 2959, 2934, 2912, 2838, 2226, 2192, 1691, 1611, 1597, 1514, 1306, 1250, 1172, 1029, 826, 756, 689 cm^{-1}; MS (EI, 70 ev) m/z (%): 354 (M^+, 9.20), 77 (100). HRMS calcd for $C_{24}H_{18}O_3$: 354.1256, found: 354.1259.

3. ((2R*,3S*)-3-(4-chlorophenyl)-2-(phenylethynyl)oxiran-2-yl)(phenyl)methanone (1c).

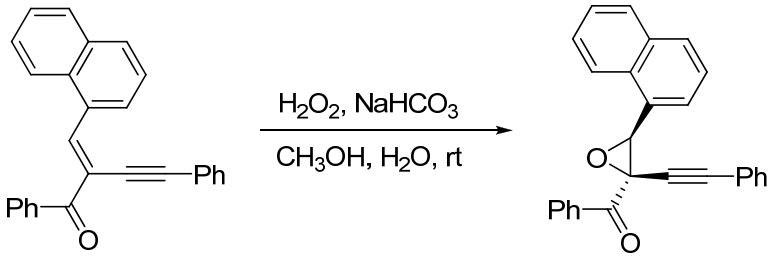


1c

The reaction of (*E*)-2-(4-chlorobenzylidene)-1,4-diphenylbut-3-yn-1-one (1.71 g, 5 mmol) in 200 mL of CH_3OH, $NaHCO_3$ (2.1 g, 25 mmol) in 20 mL of H_2O and H_2O_2 (30% in water, 5 mL , 50 mmol) at rt for 12 h afforded 1.35 g of **1c** (75 % yield) as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.13 (d, 2 H, *J* = 8.0 Hz); 7.60 (t, 1 H, *J* = 7.4 Hz); 7.51 - 7.46 (m, 4 H); 7.41 (d, 2 H, *J* = 8.0 Hz); 7.27 - 7.17 (m, 5 H); 4.34 (s, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 190.42, 135.00, 134.02, 133.72, 131.69, 131.34, 129.49, 129.24, 128.51, 128.29, 128.21, 120.89, 89.97, 81.74, 63.88, 61.12 ppm; IR (neat): $\tilde{\nu}$ = 3029, 3002, 2955, 2924, 2852, 1733, 1669, 1599, 1436, 1252, 1172, 1068, 723, 697 cm^{-1};

MS (EI, 70 ev) m/z (%): 358 (M^+, 5.08), 105 (100). HRMS calcd for $C_{23}H_{15}ClO_2$: 358.0761, found: 358.0763.

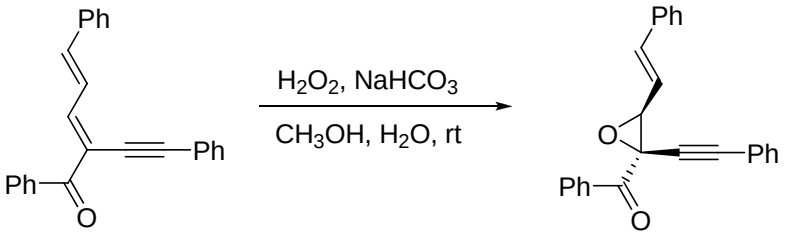
4. ((2R*,3S*)-3-(naphthalen-1-yl)-2-(phenylethynyl)oxiran-2-yl)(phenyl)methanone (1d).



1d

The reaction of (*E*)-2-(naphthalen-1-ylmethylene)-1,4-diphenylbut-3-yn-1-one (1.79 g, 5 mmol) in 200 mL of CH_3OH, $NaHCO_3$ (2.1 g, 25 mmol) in 20 mL of H_2O and H_2O_2 (30% in water, 5 mL , 50 mmol) at rt for 11 h afforded 1.39 g of **1d** (74 % yield) as a white solid. m.p. 157 - 159 ^oC; ^1H NMR (400 MHz, $CDCl_3$): δ = 8.29 (d, 2 H, *J* = 8.0 Hz); 8.14 (d, 1 H, *J* = 7.2 Hz); 7.98 - 7.92 (m, 2 H); 7.78 (d, 1 H, *J* = 6.8 Hz); 7.66 (t, 1 H, *J* = 7.2 Hz); 7.61 - 7.50 (m, 5 H); 7.21 (t, 1 H, *J* = 7.2 Hz); 7.13 (t, 2 H, *J* = 7.6 Hz); 6.97 (d, 2 H, *J* = 7.6 Hz); 5.06 (s, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 190.97, 134.16, 133.91, 133.23, 131.76, 131.33, 129.77, 129.07, 129.01, 128.90, 128.58, 128.05, 126.73, 125.99, 125.05, 124.46, 122.55, 121.09, 89.01, 82.32, 63.09, 61.29 ppm; IR (neat): $\tilde{\nu}$ = 3028, 3000, 2955, 2926, 2852, 1733, 1669, 1436, 1252, 1201, 1173, 1069, 722, 696 cm^{-1}; MS (EI, 70 ev) m/z (%): 374 (M^+, 7.02), 105 (100). HRMS calcd for $C_{27}H_{18}O_2$: 374.1307, found: 374.1309.

5. phenyl((2R*,3S*)-2-(phenylethynyl)-3-styryloxiran-2-yl)methanone (1e).

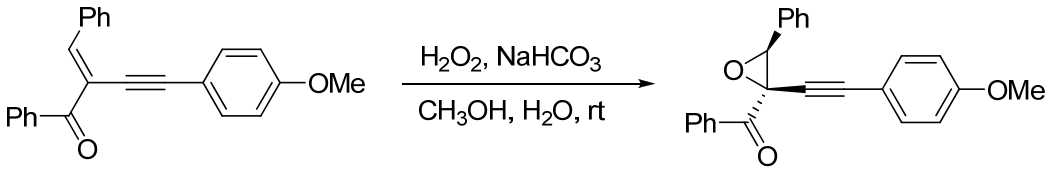


1e

The reaction of (2*E*,4*E*)-1,5-diphenyl-2-(phenylethynyl)penta-2,4-dien-1-one (1.67 g, 5 mmol) in 200 mL of CH_3OH, $NaHCO_3$ (2.1 g, 25 mmol) in 20 mL of H_2O and H_2O_2 (30% in water, 5 mL , 50 mmol) at rt for 40 h afforded 0.88 g of **1e** (50 % yield) as a yellow oil. ^1H NMR (400 MHz, $CDCl_3$): δ = 8.16 (d, 2 H, *J* = 7.6 Hz); 7.62 (t, 1 H, *J* = 7.4 Hz); 7.52 - 7.46 (m, 4 H); 7.40 - 7.24 (m,

8 H); 7.01 (d, 2 H, *J* = 16.4 Hz); 6.37 (dd, 1 H, *J* = 16.4 Hz, 8.4 Hz); 3.94 (d, 1 H, *J* = 8.4 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 190.87, 138.74, 135.53, 133.97, 131.88, 129.58, 129.25, 128.78, 128.75, 128.51, 128.32, 126.84, 122.07, 121.28, 89.65, 82.39, 64.38, 60.01 ppm; IR (neat): $\tilde{\nu}$ = 3002, 2955, 2922, 2852, 1733, 1667, 1435, 1253, 1201, 1172, 1056, 723, 635 cm^{-1}; MS (EI, 70 ev) m/z (%): 350 (M^+, 2.31), 77 (100). HRMS calcd for $C_{25}H_{18}O_2$: 350.1307, found: 350.1305.

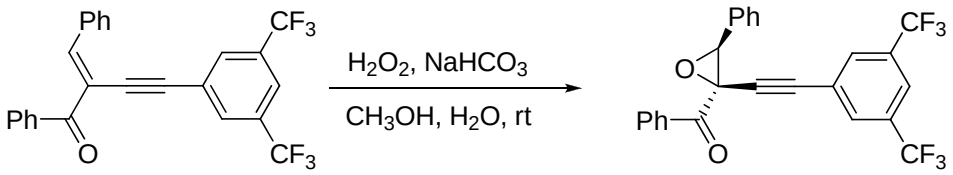
6. ((2R*,3S*)-2-((4-methoxyphenyl)ethynyl)-3-phenyloxiran-2-yl)(phenyl)methanone (1f).



1f

The reaction of (*E*)-2-benzylidene-4-(4-methoxyphenyl)-1-phenylbut-3-yn-1-one (1.69 g, 5 mmol) in 200 mL of CH_3OH, $NaHCO_3$ (2.1 g, 25 mmol) in 20 mL of H_2O and H_2O_2 (30% in water, 5 mL , 50 mmol) at rt for 12 h afforded 1.45 g of **1f** (82 % yield) as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.16 (d, 2 H, *J* = 7.2 Hz); 7.64 - 7.54 (m, 3 H); 7.54 - 7.41 (m, 5 H); 7.12 (d, 2 H, *J* = 8.8 Hz); 6.74 (d, 2 H, *J* = 8.8 Hz); 4.36 (s, 1 H); 3.76 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 191.01, 160.25, 134.08, 133.88, 133.39, 133.04, 129.63, 129.06, 128.51, 128.07, 127.27, 113.88, 113.34, 89.94, 80.93, 64.64, 61.40, 55.24 ppm; IR (neat): $\tilde{\nu}$ = 3063, 3036, 3004, 2960, 2936, 2838, 2223, 1693, 1604, 1509, 1449, 1290, 1249, 1173, 1028, 832, 730, 695 cm^{-1}; MS (EI, 70 ev) m/z (%): 354 (M^+, 7.21), 77 (100). HRMS calcd for $C_{24}H_{18}O_3$: 354.1256, found: 354.1255.

7. ((2R*,3S*)-2-((3,5-bis(trifluoromethyl)phenyl)ethynyl)-3-phenyloxiran-2-yl)(phenyl)methanone (1g).

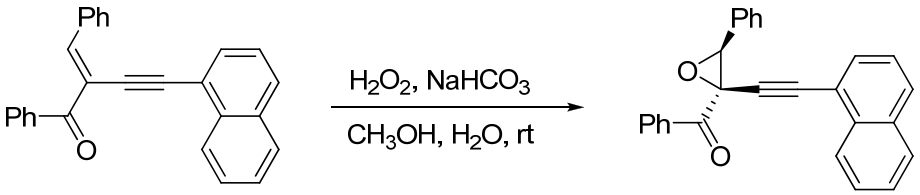


1g

The reaction of (*E*)-2-benzylidene-4-(3,5-bis(trifluoromethyl)phenyl)-1-phenylbut-3-yn-1-one (2.22 g, 5 mmol) in 200 mL of CH_3OH, $NaHCO_3$ (2.1 g, 25 mmol) in 20 mL of H_2O and H_2O_2 (30% in water, 5 mL , 50 mmol) at rt for 12 h afforded 1.70 g of **1g** (74 % yield) as a white solid. m.p. 124 - 126 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.13 (d, 2 H, *J* = 7.2 Hz); 7.77 (s, 1 H); 7.66 (t, 1 H, *J* =

7.2 Hz); 7.57 - 7.48 (m, 9 H); 4.44 (s, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 190.24, 134.30, 133.77, 132.09, 129.50, 129.46, 128.74, 128.28, 127.02, 123.97, 123.52, 122.56 (hep, *J* = 3.7 Hz), 121.26, 86.11, 85.87, 64.79, 60.89 ppm. ^{19}F NMR (376 MHz, $CDCl_3$): δ = -63.25 ppm; IR (neat): $\tilde{\nu}$= 3028, 3001, 2955, 2925, 2852, 1733, 1669, 1436, 1252, 1203, 1173, 1068, 721, 635 cm^{-1}; MS (EI, 70 ev) m/z (%): 460 (M^+, 4.24), 105 (100). HRMS calcd for $C_{25}H_{14}F_6O_2$: 460.0898, found: 460.0899.

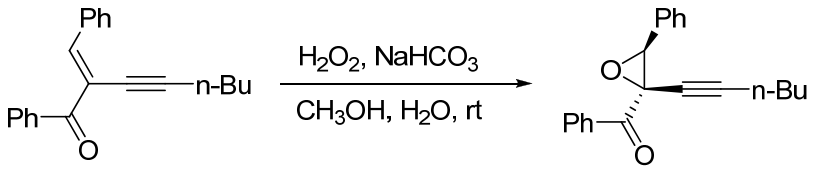
8. ((2R*,3S*)-2-(naphthalen-1-ylethynyl)-3-phenyloxiran-2-yl)(phenyl)methanone (1h).



1h

The reaction of (*E*)-2-benzylidene-4-(naphthalen-1-yl)-1-phenylbut-3-yn-1-one (1.71 g, 5 mmol) in 200 mL of CH_3OH, $NaHCO_3$ (2.1 g, 25 mmol) in 20 mL of H_2O and H_2O_2 (30% in water, 5 mL , 50 mmol) at rt for 17 h afforded 1.14 g of **1h** (61 % yield) as a white solid. m.p. 99 - 101 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.20 (d, 2 H, *J* = 8.0 Hz); 7.67 - 7.61 (m, 4 H); 7.56 - 7.48 (m, 2 H); 7.45 - 7.40 (m, 5 H); 7.35 (t, 2 H, *J* = 7.8 Hz); 7.27 - 7.17 (m, 2 H); 4.46 (s, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 190.81, 133.90, 133.85, 132.92, 132.85, 132.59, 131.08, 129.53, 129.44, 129.00, 128.45, 128.17, 127.97, 126.98, 126.68, 126.25, 125.46, 124.72, 118.53, 88.04, 86.64, 64.55, 61.42 ppm; IR (neat): $\tilde{\nu}$= 3027, 3002, 2955, 2925, 2853, 1733, 1669, 1436, 1253, 1203, 1173, 1068, 723, 698 cm^{-1}; MS (EI, 70 ev) m/z (%): 374 (M^+, 11.94), 239 (100). HRMS calcd for $C_{27}H_{18}O_2$: 374.1307, found: 374.1308.

9. ((2R*,3S*)-2-(hex-1-ynyl)-3-phenyloxiran-2-yl)(phenyl)methanone (1i).

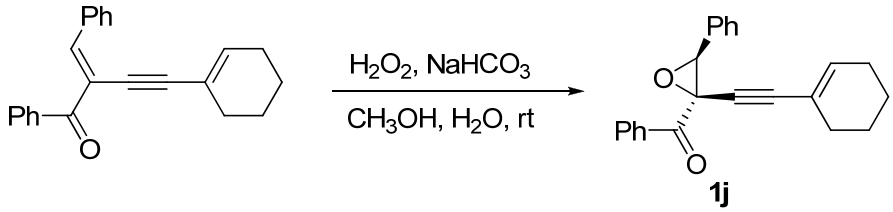


1i

The reaction of (*E*)-2-benzylidene-1-phenyloct-3-yn-1-one (1.44 g, 5 mmol) in 200 mL of CH_3OH, $NaHCO_3$ (2.1 g, 25 mmol) in 20 mL of H_2O and H_2O_2 (30% in water, 5 mL , 50 mmol) at rt for 17 h afforded 0.70 g of **1i** (46% yield) as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.10 (d, 2

H, *J* = 7.6 Hz); 7.60 (t, 1 H, *J* = 7.4 Hz); 7.52 - 7.38 (m, 7 H); 4.23 (s, 1 H); 2.06 (t, 2 H, *J* = 6.4 Hz); 1.31 - 1.23 (m, 2 H); 1.18 - 1.08 (m, 2 H); 0.75 (t, 3 H, *J* = 7.2 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 191.35, 133.92, 133.72, 133.02, 129.52, 128.82, 128.36, 127.94, 127.10, 91.92, 73.11, 63.91, 61.09, 29.70, 21.40, 18.35, 13.31 ppm; IR (neat): $\tilde{\nu}$ = 3064, 3033, 2957, 2932, 2871, 2239, 1694, 1598, 1450, 1260, 1174, 853, 756, 695 cm^{-1}; MS (EI, 70 ev) m/z (%): 304 (M^+, 2.43), 105 (100). HRMS calcd for $C_{21}H_{20}O_2$: 304.1463, found: 304.1465.

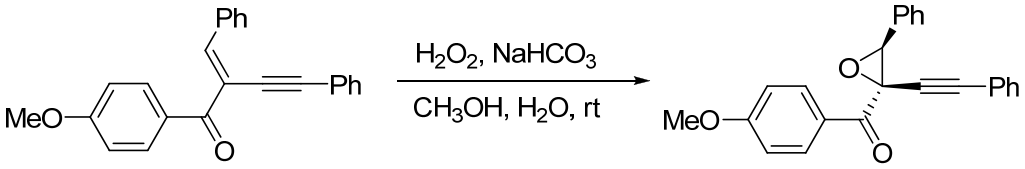
10. ((2R*,3S*)-2-(cyclohexenylethynyl)-3-phenyloxiran-2-yl)(phenyl)methanone (1j).



1j

The reaction of (*E*)-2-benzylidene-4-cyclohexenyl-1-phenylbut-3-yn-1-one (1.56 g, 5 mmol) in 200 mL of CH_3OH, $NaHCO_3$ (2.1 g, 25 mmol) in 20 mL of H_2O and H_2O_2 (30% in water, 5 mL , 50 mmol) at rt for 30 h afforded 1.31 g of **1j** (80 % yield) as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.10 (m, 2 H); 7.60 (t, 1 H, *J* = 7.4 Hz); 7.55 - 7.38 (m, 7 H); 5.95 - 5.92 (m, 1 H); 4.29 (s, 1 H); 2.01 - 1.98 (m, 2 H); 1.87 - 1.85 (m, 2 H); 1.54 - 1.45 (m, 4 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 191.10, 137.40, 133.95, 133.80, 132.94, 129.58, 128.95, 128.40, 127.96, 127.25, 119.30, 91.82, 79.37, 64.53, 61.30, 28.16, 25.49, 21.85, 21.11 ppm; IR (neat): $\tilde{\nu}$ = 3063, 3031, 2933, 2861, 2840, 2215, 1693, 1597, 1449, 1260, 1178, 850, 751, 696 cm^{-1}; MS (EI, 70 ev) m/z (%): 328 (M^+, 3.53), 105 (100). HRMS calcd for $C_{23}H_{20}O_2$: 328.1463, found: 328.1462.

11. (4-methoxyphenyl)((2R*,3S*)-3-phenyl-2-(phenylethynyl)oxiran-2-yl)methanone (1k)

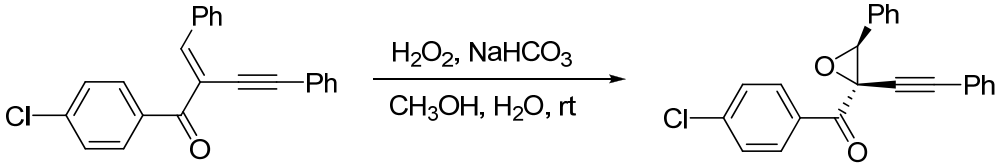


1k

The reaction of (*E*)-2-benzylidene-1-(4-methoxyphenyl)-4-phenylbut-3-yn-1-one (1.69 g, 5 mmol) in 200 mL of CH_3OH, $NaHCO_3$ (2.1 g, 25 mmol) in 20 mL of H_2O and H_2O_2 (30% in water, 5 mL , 50 mmol) at rt for 9 h afforded 1.01 g of **1k** (57% yield) as a yellow oil. ^{1}H NMR (300 MHz,

$CDCl_3$): δ = 8.16 (d, 2 H, *J* = 8.7 Hz); 7.56 (d, 2 H, *J* = 6.6 Hz); 7.47 - 7.40 (m, 3 H); 7.27 - 7.18 (m, 5 H); 6.95 (d, 2 H, *J* = 8.7 Hz); 4.34 (s, 1 H); 3.84 (s, 3 H). ^{13}C NMR (75.4 MHz, $CDCl_3$): δ = 189.03, 164.19, 132.99, 131.97, 131.73, 129.02, 128.98, 128.13, 128.01, 127.12, 126.76, 121.29, 113.80, 89.31, 82.55, 64.41, 61.09, 55.43 ppm; IR (neat): $\tilde{\nu}$ = 3062, 3034, 3010, 2968, 2935, 2840, 2226, 1683, 1597, 1510, 1314, 1257, 1170, 1205, 858, 757, 690 cm^{-1}; MS (EI, 70 ev) m/z (%): 354 (M^+, 2.67), 135 (100). HRMS calcd for $C_{24}H_{18}O_3$: 354.1256, found: 354.1254.

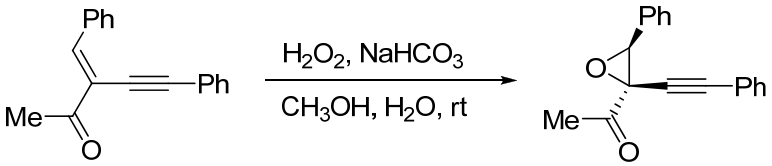
12. (4-chlorophenyl)((2R*,3S*)-3-phenyl-2-(phenylethynyl)oxiran-2-yl)methanone (1l)



1l

The reaction of (*E*)-2-benzylidene-1-(4-chlorophenyl)-4-phenylbut-3-yn-1-one (1.71 g, 5 mmol) in 200 mL of CH_3OH, $NaHCO_3$ (2.1 g, 25 mmol) in 20 mL of H_2O and H_2O_2 (30% in water, 5 mL, 50 mmol) at rt for 13 h afforded 0.90 g of **1l** (50% yield) as a white solid. m.p. 104 - 105 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.10 (d, 2 H, *J* = 8.8 Hz); 7.58 - 7.54 (m, 2 H); 7.50 - 7.40 (m, 5 H); 7.30 - 7.15 (m, 5 H); 4.36 (s, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 189.72, 140.50, 132.60, 132.25, 131.78, 130.96, 129.26, 129.22, 128.90, 128.24, 128.12, 127.19, 121.03, 89.91, 81.86, 64.69, 61.06 ppm; IR (neat): $\tilde{\nu}$ = 3072, 2980, 2231, 1698, 1588, 1489, 1449, 1258, 1090, 960, 833, 758, 693 cm^{-1}; MS (EI, 70 ev) m/z (%): 358 (M^+, 7.34), 139 (100). Anal calcd for $C_{23}H_{15}ClO_2$: C, 76.99; H, 4.21; found: C, 77.15; H, 4.05.

13. 1-((2R*,3S*)-3-phenyl-2-(phenylethynyl)oxiran-2-yl)ethanone (1m).

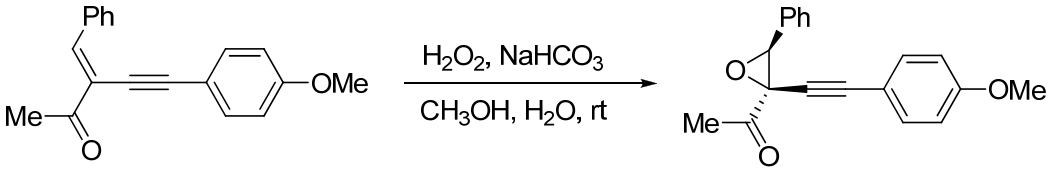


1m

The reaction of (*E*)-3-benzylidene-5-phenylpent-4-yn-2-one (1.23 g, 5 mmol) in 200 mL of CH_3OH, $NaHCO_3$ (2.1 g, 25 mmol) in 20 mL of H_2O and H_2O_2 (30% in water, 5 mL, 50 mmol) at rt for 12 h afforded 891.8 mg of **1m** (68% yield) as a yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.52

- 7.45 (m, 2 H); 7.45 - 7.39 (m, 3 H); 7.35 - 7.24 (m, 5 H); 4.38 (s, 1 H); 2.48 (s, 3 H). ^{13}C NMR (75.4 MHz, $CDCl_3$): δ = 200.15, 132.59, 131.74, 129.01, 128.97, 128.14, 127.81, 127.13, 121.15, 88.04, 81.37, 65.41, 61.14, 26.37 ppm; IR (neat): $\tilde{\nu}$ = 3063, 3033, 2924, 2868, 2233, 1722, 1491, 1358, 1246, 1215, 1120, 858, 753, 691 cm^{-1}; MS (EI, 70 ev) m/z (%): 262 (M^+, 10.65), 128 (100). HRMS calcd for $C_{18}H_{14}O_2$: 262.0994, found: 262.0996.

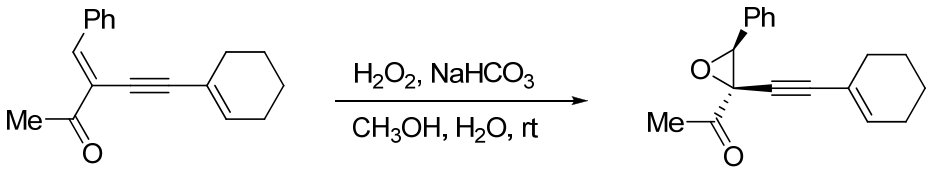
14. 1-((2R*,3S*)-2-((4-methoxyphenyl)ethynyl)-3-phenyloxiran-2-yl)ethanone (1n).



1n

The reaction of (*E*)-3-benzylidene-5-(4-methoxyphenyl)pent-4-yn-2-one (1.38 g, 5 mmol) in 200 mL of CH_3OH, $NaHCO_3$ (2.1 g, 25 mmol) in 20 mL of H_2O and H_2O_2 (30% in water, 5 mL , 50 mmol) at rt for 36 h afforded 0.99 g of **1n** (68 % yield) as a yellow solid. m.p. 47 - 48 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.50 - 7.37 (m, 5 H); 7.20 (d, 2 H, *J* = 8.7 Hz); 6.78 (d, 2 H, *J* = 8.7 Hz); 4.33 (s, 1 H); 3.79 (s, 3 H); 2.48 (s, 3 H). ^{13}C NMR (75.4 MHz, $CDCl_3$): δ = 200.69, 160.20, 133.49, 132.83, 129.07, 127.92, 127.31, 113.91, 113.30, 88.34, 80.08, 65.67, 61.40, 55.27, 26.75 ppm; IR (neat): $\tilde{\nu}$ = 2973, 2903, 2840, 2227, 1711, 1603, 1509, 1289, 1249, 1112, 1020, 841, 760, 699 cm^{-1}; MS (EI, 70 ev) m/z (%): 292 (M^+, 13.61), 158 (100). HRMS calcd for $C_{19}H_{16}O_3$: 292.1099, found: 292.1100.

15. 1-((2R*,3S*)-2-(cyclohexenylethynyl)-3-phenyloxiran-2-yl)ethanone (1o).

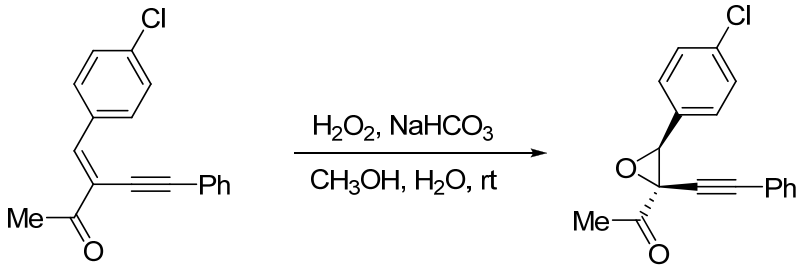


1o

The reaction of (*E*)-3-benzylidene-5-cyclohexenylpent-4-yn-2-one (1.25 g, 5 mmol) in 200 mL of CH_3OH, $NaHCO_3$ (2.1 g, 25 mmol) in 20 mL of H_2O and H_2O_2 (30% in water, 5 mL, 50 mmol) at rt for 18 h afforded 0.71 g of **1o** (53% yield) as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.39 - 7.35 (m, 5 H); 6.03 (br, 1 H); 4.26 (s, 1 H); 2.41 (s, 3 H); 2.02 – 1.94 (m, 4 H); 1.54 – 1.52 (m, 4 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.59, 137.33, 132.72, 128.82, 127.69, 127.18, 119.19, 90.09, 78.53, 65.44, 61.16, 28.21, 26.55, 25.43, 21.82, 21.07 ppm; IR (neat): $\tilde{\nu}$ = 3001, 2957, 2849, 1736, 1669, 1442, 1258, 1199, 1087, 1066, 722, 657 cm^{-1}; MS (EI, 70 ev) m/z (%): 266 (M^+, 20.65), 43 (100). HRMS calcd for $C_{18}H_{18}O_2$: 266.1307, found: 266.1305.

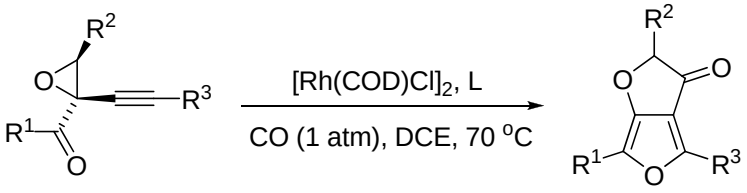
16. 1-((2R*,3S*)-3-(4-chlorophenyl)-2-(phenylethynyl)oxiran-2-yl)ethanone (1p).



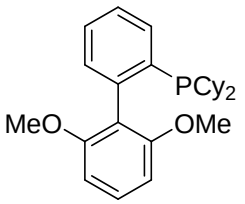
1p

The reaction of (*E*)-3-(4-chlorobenzylidene)-5-phenylpent-4-yn-2-one (1.40 g, 5 mmol) in 200 mL of CH_3OH, $NaHCO_3$ (2.1 g, 25 mmol) in 20 mL of H_2O and H_2O_2 (30% in water, 5 mL, 50 mmol) at rt for 28 h afforded 1.04 g of **1p** (70 % yield) as a yellow solid. m.p. 89 - 90 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.42 - 7.25 (m, 9 H); 4.31 (s, 1 H); 2.48 (s, 3 H). ^{13}C NMR (75.4 MHz, $CDCl_3$): δ = 199.93, 135.08, 131.90, 131.36, 129.32, 128.63, 128.37, 128.24, 121.09, 88.56, 81.07, 64.90, 61.13, 26.65 ppm; IR (neat): $\tilde{\nu}$ = 3081, 2966, 2234, 1710, 1490, 1255, 1114, 1086, 1016, 866, 841, 799, 749, 683 cm^{-1}; MS (EI, 70 ev) m/z (%): 296 (M^+, 4.40), 128 (100). Anal calcd for $C_{18}H_{13}ClO_2$: C, 72.85; H, 4.42; found: C, 72.93; H, 4.15.

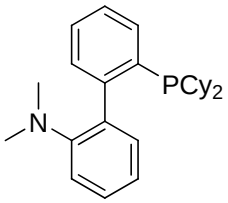
Typical procedure for Rhodium(I)-Catalyzed Tandem Heterocyclization/[4+1] Cycloaddition of 1-(1-Alkynyl)oxiranyl Ketones.



1 (racemic) **3**



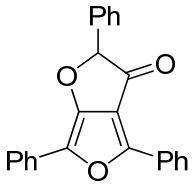
L1



L3

The $[Rh(COD)Cl]_2$ (9.9 mg, 0.02 mmol) and **L1** (8.2 mg, 0.02 mmol) were allowed to stir together over 1 hour in 2 mL of anhydrous CH_2ClCH_2Cl in the presence of 1 atm of CO (balloon). The catalyst solution was then transferred to the solution of **1** (0.4 mmol) in 2 mL of anhydrous CH_2ClCH_2Cl in the presence of 1 atm of CO (balloon). The combined solution was then stired at 70 ^oC until the reaction was completed (monitored by TLC). After evaporation, the residue was purified by column chromatography on silica gel (hexanes/ EtOAc = 20:1) afforded the desired product.

17. 2,4,6-triphenylfuro[3,4-b]furan-3(2H)-one (3a).

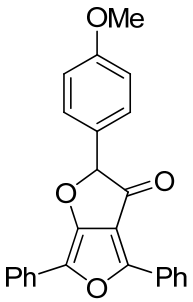


3a

The reaction of **1a** (129.7 mg, 0.4 mmol), $[Rh(COD)Cl]_2$ (9.9 mg, 0.02 mmol) and **L1** (8.2 mg, 0.02 mmol) in $ClCH_2CH_2Cl$ (4 mL) was carried out in the presence of 1 atm of CO (balloon) at 70 ^oC for 11 hours to afford **3a** (139.8 mg) in 99% yield as a yellow solid. m.p. 185 - 187 ^oC; ^1H NMR (400 MHz, $CDCl_3$): δ = 8.16 (d, 2 H, J = 8.0 Hz); 7.74 (d, 2 H, J = 7.6 Hz); 7.51 - 7.37 (m, 10 H); 7.24 (t, 1 H, J = 7.6 Hz); 6.06 (s, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 190.18, 156.76, 148.82, 134.49, 130.57, 130.12, 129.08, 129.02, 128.99, 128.88, 128.85, 128.40, 126.48, 126.03, 125.80, 122.66, 114.17, 96.11 ppm; IR (neat): $\tilde{\nu}$ = 3028, 3002, 2955, 2929, 2852, 1733, 1669, 1598, 1580, 1437,

1254, 1203, 1172, 1068, 967, 722 cm^{-1}; MS (EI, 70 ev) m/z (%): 352 (18.32), 324 (6.59), 77 (100). HRMS calcd for $C_{24}H_{16}O_3$: 352.1099, found: 352.1100.

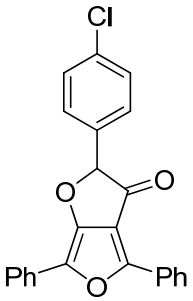
18. 2-(4-methoxyphenyl)-4,6-diphenylfuro[3,4-b]furan-3(2H)-one (3b).



3b

The reaction of **1b** (141.8 mg, 0.4 mmol), $[Rh(COD)Cl]_2$ (9.9 mg, 0.02 mmol) and **L1** (8.2 mg, 0.02 mmol) in $ClCH_2CH_2Cl$ (4 mL) was carried out in the presence of 1 atm of CO (balloon) at 70 ^oC for 12 hours to afford **3b** (110.1 mg) in 72% yield as a yellow solid. m.p. 184 - 186 ^oC; ^1H NMR (400 MHz, $CDCl_3$): δ = 8.15 (d, 2 H, *J* = 7.6 Hz); 7.71 (d, 2 H, *J* = 7.6 Hz); 7.47 - 7.37 (m, 7 H); 7.22 (t, 1 H, *J* = 7.0 Hz); 6.92 (d, 2 H, *J* = 8.0 Hz); 5.98 (s, 1 H); 3.79 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 190.68, 160.21, 156.66, 148.65, 130.49, 130.03, 129.08, 128.96, 128.82, 128.41, 127.84, 126.70, 126.38, 125.76, 122.60, 114.32, 114.27, 96.14, 55.29 ppm; IR (neat): $\tilde{\nu}$ = 3029, 3002, 2955, 2925, 2852, 1733, 1669, 1598, 1580, 1436, 1259, 1203, 1171, 1068, 967, 722 cm^{-1}; MS (EI, 70 ev) m/z (%): 382 (M^+, 26.14), 354 (16.71), 77 (100). HRMS calcd for $C_{25}H_{18}O_4$: 382.1205, found: 382.1208.

19. 2-(4-chlorophenyl)-4,6-diphenylfuro[3,4-b]furan-3(2H)-one (3c).

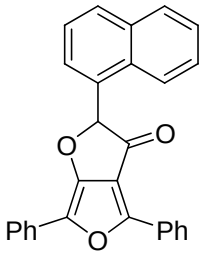


3c

The reaction of **1c** (143.5 mg, 0.4 mmol), $[Rh(COD)Cl]_2$ (9.9 mg, 0.02 mmol) and **L1** (8.2 mg, 0.02 mmol) in $ClCH_2CH_2Cl$ (4 mL) was carried out in the presence of 1 atm of CO (balloon) at 70 ^oC

for 17 hours to afford **3c** (140.5 mg) in 91% yield as a yellow solid. m.p. 192 - 194 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.15 (d, 2 H, *J* = 7.6 Hz); 7.73 (d, 2 H, *J* = 7.6 Hz); 7.49 - 7.38 (m, 9 H); 7.26 (t, 1 H, *J* = 7.2 Hz); 6.03 (s, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 189.62, 156.51, 149.03, 135.00, 132.94, 130.71, 130.26, 129.05, 128.96, 128.92, 128.32, 127.32, 126.63, 125.83, 122.71, 113.89, 95.24 ppm; IR (neat): $\tilde{\nu}$ = 3029, 3004, 2955, 2929, 2852, 1733, 1669, 1598, 1580, 1437, 1254, 1203, 1172, 1068, 967, 722 cm^{-1}; MS (EI, 70 ev) m/z (%): 386 (M^+, 22.37), 358 (7.38), 105 (100). HRMS calcd for $C_{24}H_{15}ClO_3$: 386.0710, found: 386.0711.

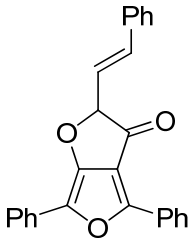
20. 2-(naphthalen-1-yl)-4,6-diphenylfuro[3,4-b]furan-3(2H)-one (3d).



3d

The reaction of **1d** (149.8 mg, 0.4 mmol), $[Rh(COD)Cl]_2$ (9.9 mg, 0.02 mmol) and **L1** (8.2 mg, 0.02 mmol) in $ClCH_2CH_2Cl$ (4 mL) was carried out in the presence of 1 atm of CO (balloon) at 70 ^{o}C for 22 hours to afford **3d** (130.8 mg) in 81% yield as a yellow solid. m.p. 215 - 217 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.18 (d, 2 H, *J* = 7.6 Hz); 8.11 (d, 1 H, *J* = 8.4 Hz); 7.88 (t, 2 H, *J* = 6.8 Hz); 7.74 (d, 2 H, *J* = 7.6 Hz); 7.61 - 7.51 (m, 3 H); 7.47 - 7.37 (m, 6 H); 7.23 (t, 1 H, *J* = 7.2 Hz); 6.76 (s, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 190.31, 156.39, 148.70, 134.07, 130.95, 130.56, 130.36, 130.31, 129.97, 129.08, 128.99, 128.87, 128.79, 128.44, 126.81, 126.50, 126.13, 125.83, 125.16, 123.92, 122.70, 114.51, 94.69 ppm; IR (neat): $\tilde{\nu}$ = 3028, 3002, 2955, 2924, 2852, 1733, 1669, 1598, 1580, 1437, 1254, 1203, 1173, 1069, 967, 721 cm^{-1}; MS (EI, 70 ev) m/z (%): 402 (M^+, 19.64), 105 (100). HRMS calcd for $C_{28}H_{18}O_3$: 402.1256, found: 402.1257.

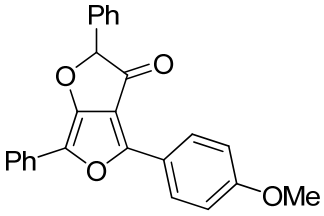
21. (E)-4,6-diphenyl-2-styrylfuro[3,4-b]furan-3(2H)-one (3e).



3e

The reaction of **1e** (140.2 mg, 0.4 mmol), $[Rh(COD)Cl]_2$ (9.9 mg, 0.02 mmol) and **L1** (8.2 mg, 0.02 mmol) in $ClCH_2CH_2Cl$ (4 mL) was carried out in the presence of 1 atm of CO (balloon) at 70 ^oC for 13 hours to afford **3e** (87.8 mg) in 58% yield as a yellow solid. m.p. 172 - 174 ^oC; ^1H NMR (400 MHz, $CDCl_3$): δ = 8.15 (d, 2 H, *J* = 7.6 Hz); 7.71 (d, 2 H, *J* = 7.6 Hz); 7.49 -7.38 (m, 7 H); 7.34 - 7.21 (m, 5 H); 6.91 (d, 1 H, *J* = 16.0 Hz); 6.36 (dd, 1 H, *J* = 16.0 Hz, 6.4 Hz); 5.72 (d, 1 H, *J* = 6.4 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 190.19, 156.39, 148.53, 135.60, 134.31, 130.51, 130.26, 129.07, 128.99, 128.84, 128.61, 128.42, 126.86, 126.43, 125.76, 122.61, 121.31, 114.27, 95.73 ppm; IR (neat): $\tilde{\nu}$ = 3028, 3003, 2955, 2923, 2852, 1733, 1669, 1598, 1580, 1436, 1254, 1203, 1172, 1067, 966, 722 cm^{-1}; MS (EI, 70 ev) m/z (%): 378 (M^+, 34.94), 77 (100). HRMS calcd for $C_{26}H_{18}O_3$: 378.1256, found: 378.1255.

22. 4-(4-methoxyphenyl)-2,6-diphenylfuro[3,4-b]furan-3(2H)-one (3f).

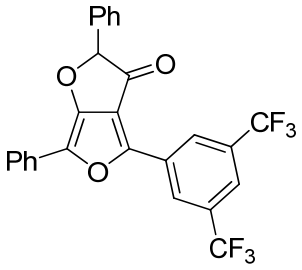


3f

The reaction of **1f** (141.8 mg, 0.4 mmol), $[Rh(COD)Cl]_2$ (9.9 mg, 0.02 mmol) and **L1** (8.2 mg, 0.02 mmol) in $ClCH_2CH_2Cl$ (4 mL) was carried out in the presence of 1 atm of CO (balloon) at 70 ^oC for 11 hours to afford **3f** (145.8 mg) in 98% yield as a yellow solid. m.p. 194 - 196 ^oC; ^1H NMR (400 MHz, $CDCl_3$): δ = 8.08 (d, 2 H, *J* = 7.6 Hz); 7.68 (d, 2 H, *J* = 7.6 Hz); 7.48 (d, 2 H, *J* = 7.2 Hz); 7.43 - 7.33 (m, 5 H); 7.20 (t, 1 H, *J* = 7.6 Hz); 6.93 (d, 2 H, *J* = 7.6 Hz); 5.99 (s, 1 H); 3.81 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 189.81, 161.54, 156.53, 149.41, 134.69, 129.24, 129.07, 128.90, 128.78, 127.75, 126.05, 126.01, 122.38, 121.39, 114.40, 112.59, 96.09, 55.35 ppm; IR (neat): $\tilde{\nu}$ =

3028, 3002, 2955, 2922, 2853, 1733, 1670, 1598, 1580, 1447, 1436, 1251, 1203, 1171, 1068, 967, 722 cm^{-1}; MS (EI, 70 ev) m/z (%): 382 (M^+, 34.92), 354 (8.53), 105 (100). HRMS calcd for $C_{25}H_{18}O_4$: 382.1205, found: C, 382.1202.

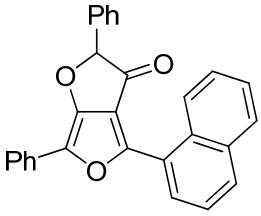
23. 4-(3,5-bis(trifluoromethyl)phenyl)-2,6-diphenylfuro[3,4-b]furan-3(2H)-one (3g).



3g

The reaction of **1g** (184.1 mg, 0.4 mmol), $[Rh(COD)Cl]_2$ (9.9 mg, 0.02 mmol) and **L1** (8.2 mg, 0.02 mmol) in $ClCH_2CH_2Cl$ (4 mL) was carried out in the presence of 1 atm of CO (balloon) at 70 ^oC for 40 hours to afford **3g** (168.0 mg) in 86% yield as a yellow solid. m.p. 227 - 229 ^oC; ^1H NMR (400 MHz, $CDCl_3$): δ = 8.56 (s, 2 H); 7.87 (s, 1 H); 7.79 (d, 2 H, *J* = 7.6 Hz); 7.51 - 7.39 (m, 7 H); 7.32 (t, 1 H, *J* = 7.2 Hz); 6.13 (s, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 190.38, 156.83, 144.41, 133.74, 132.87, 132.54, 132.46, 130.08, 129.32, 129.07, 129.02, 128.30, 127.48, 125.91, 125.05, 124.30, 123.13, 123.03 (hep, *J* = 3.9 Hz), 121.58, 116.50, 96.27 ppm, ^{19}F NMR (376 MHz, $CDCl_3$): δ = -63.14 ppm; IR (neat): $\tilde{\nu}$ = 3027, 3003, 2955, 2925, 2852, 1734, 1669, 1598, 1580, 1437, 1253, 1203, 1172, 1069, 968, 722 cm^{-1}; MS (EI, 70 ev) m/z (%): 488 (M^+, 13.61), 460 (5.37), 43 (100). HRMS calcd for $C_{26}H_{14}F_6O_3$: 488.0847, found: 488.0846.

24. 4-(naphthalen-1-yl)-2,6-diphenylfuro[3,4-b]furan-3(2H)-one (3h).

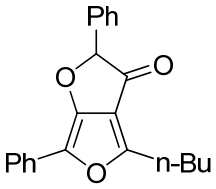


3h

The reaction of **1h** (149.8 mg, 0.4 mmol), $[Rh(COD)Cl]_2$ (9.9 mg, 0.02 mmol) and **L1** (8.2 mg, 0.02 mmol) in $ClCH_2CH_2Cl$ (4 mL) was carried out in the presence of 1 atm of CO (balloon) at 70 ^oC for 40 hours to afford **3h** (144.9 mg) in 90% yield as a yellow solid. m.p. 215 - 216 ^oC; ^1H NMR

(400 MHz, $CDCl_3$): δ = 8.97 (d, 2 H, *J* = 8.8 Hz); 8.53 (d, 2 H, *J* = 7.6 Hz); 7.92 (t, 2 H, *J* = 7.0 Hz); 7.80 (d, 2 H, *J* = 8.0 Hz); 7.70 (t, 1 H, *J* = 7.6 Hz); 7.59 - 7.36 (m, 9 H); 7.30 - 7.25 (m, 1 H); 6.11 (s, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 190.00, 156.99, 150.65, 134.56, 134.13, 131.77, 130.70, 129.29, 129.12, 129.07, 129.03, 129.01, 128.87, 127.86, 126.55, 126.37, 126.06, 125.54, 125.51, 124.88, 122.66, 115.32, 96.20 ppm; IR (neat): $\tilde{\nu}$ = 3028, 3002, 2955, 2922, 2852, 1733, 1670, 1598, 1580, 1436, 1253, 1203, 1172, 1068, 967, 721 cm^{-1}; MS (EI, 70 ev) m/z (%): 402 (M^+, 17.34), 105 (100). HRMS calcd for $C_{28}H_{18}O_3$: 402.1256, found: 402.1255.

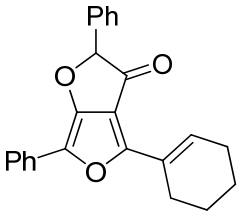
25. 4-butyl-2,6-diphenylfuro[3,4-b]furan-3(2H)-one (3i).



3i

The reaction of **1i** (121.8 mg, 0.4 mmol), $[Rh(COD)Cl]_2$ (9.9 mg, 0.02 mmol) and **L1** (8.2 mg, 0.02 mmol) in $ClCH_2CH_2Cl$ (4 mL) was carried out in the presence of 1 atm of CO (balloon) at 70 °C for 48 hours to afford **3i** (91.7 mg) in 69% yield as a yellow oil. ^1H NMR (400 MHz, $CDCl_3$): δ = 7.61 (d, 2 H, *J* = 8.0 Hz); 7.43 (d, 2 H, *J* = 7.6 Hz); 7.39 - 7.30 (m, 5 H); 7.16 (t, 1 H, *J* = 7.6 Hz); 5.88 (s, 1 H); 2.81 (t, 2 H, *J* = 7.6 Hz); 1.83 - 1.75 (m, 2 H); 1.44 - 1.34 (m, 2 H); 0.93 (t, 3 H, *J* = 7.4 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 190.40, 155.12, 154.15, 134.58, 129.51, 129.29, 128.79, 128.70, 125.89, 122.18, 114.67, 95.70, 28.81, 28.68, 22.24, 13.57 ppm; IR (neat): $\tilde{\nu}$ = 3027, 3001, 2955, 2922, 2852, 1733, 1669, 1598, 1580, 1438, 1253, 1203, 1173, 1067, 967, 722 cm^{-1}; MS (EI, 70 ev) m/z (%): 332 (M^+, 7.91), 304 (3.61), 43 (100). HRMS calcd for $C_{22}H_{20}O_3$: 332.1412, found: 332.1411.

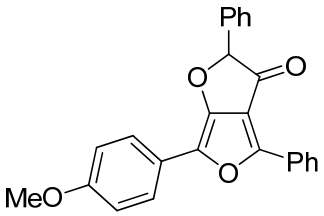
26. 4-cyclohexenyl-2,6-diphenylfuro[3,4-b]furan-3(2H)-one (3j).



3j

The reaction of **1j** (131.4 mg, 0.4 mmol), $[Rh(COD)Cl]_2$ (9.9 mg, 0.02 mmol) and **L1** (8.2 mg, 0.02 mmol) in $ClCH_2CH_2Cl$ (4 mL) was carried out in the presence of 1 atm of CO (balloon) at 70 ^oC for 11 hours to afford **3j** (124.9 mg) in 88% yield as a yellow solid. m.p. 155 - 157 ^oC; ^1H NMR (400 MHz, $CDCl_3$): δ = 7.62 (d, 2 H, *J* = 7.6 Hz); 7.45 (d, 2 H, *J* = 7.2 Hz); 7.40 - 7.32 (m, 5 H); 7.22 - 7.15 (m, 1 H); 7.11 (s, 1 H); 5.94 (s, 1 H); 2.51 (br, 2 H)； 2.28 (br, 2 H); 1.74 - 1.64 (m, 4 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 189.75, 156.59, 151.53, 134.75, 134.42, 129.32, 128.80, 128.72, 128.52, 127.97, 125.93, 125.91, 122.29, 112.41, 95.93, 25.94, 23.69, 21.77, 21.64 ppm; IR (neat): $\tilde{\nu}$ = 3027, 3002, 2955, 2922, 2851, 1733, 1669, 1598, 1580, 1438, 1253, 1203, 1173, 1067, 967, 723 cm^{-1}; 356 (M^+, 32.82), 328 (7.07), 105 (100). HRMS calcd for $C_{24}H_{20}O_3$: 356.1412, found: 356.1411.

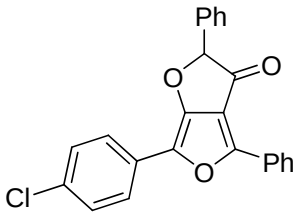
27. 6-(4-methoxyphenyl)-2,4-diphenylfuro[3,4-b]furan-3(2H)-one (3k).



3k

The reaction of **1k** (141.8 mg, 0.4 mmol), $[Rh(COD)Cl]_2$ (9.9 mg, 0.02 mmol) and **L1** (8.2 mg, 0.02 mmol) in $ClCH_2CH_2Cl$ (4 mL) was carried out in the presence of 1 atm of CO (balloon) at 70 ^oC for 16 hours to afford **3k** (120.8 mg) in 79% yield as a yellow solid. m.p. 190 - 192 ^oC; ^1H NMR (400 MHz, $CDCl_3$): δ = 8.13 (d, 2 H, *J* = 7.6 Hz); 7.67 (d, 2 H, *J* = 7.6 Hz); 7.52 - 7.37 (m, 8 H); 6.99 (d, 2 H, *J* = 7.6 Hz); 6.03 (s, 1 H); 3.85 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 190.33, 158.28, 155.36, 147.88, 134.61, 130.20, 130.17, 128.93, 128.91, 128.80, 128.50, 126.01, 125.54, 124.13, 122.11, 114.39, 114.16, 95.94, 55.31 ppm; IR (neat): $\tilde{\nu}$ = 3026, 3001, 2955, 2924, 2853, 1733, 1670, 1598, 1580, 1437, 1259, 1203, 1171, 1065, 967, 722 cm^{-1}; MS (EI, 70 ev) m/z (%): 382 (M^+, 12.48), 43 (100). HRMS calcd for $C_{25}H_{18}O_4$: 382.1205, found: 382.1208.

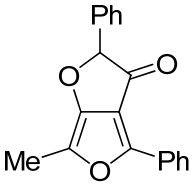
28. 6-(4-chlorophenyl)-2,4-diphenylfuro[3,4-b]furan-3(2H)-one (3l).



3l

The reaction of **1l** (143.5 mg, 0.4 mmol), $[Rh(COD)Cl]_2$ (9.9 mg, 0.02 mmol) and **L1** (8.2 mg, 0.02 mmol) in $ClCH_2CH_2Cl$ (4 mL) was carried out in the presence of 1 atm of CO (balloon) at 70 ^oC for 17 hours to afford **3l** (151.6 mg) in 98% yield as a yellow solid. m.p. 214 - 216 ^oC; ^1H NMR (400 MHz, $CDCl_3$): δ = 8.14 (d, 2 H, *J* = 8.0 Hz); 7.64 (d, 2 H, *J* = 8.0 Hz); 7.50 - 7.39 (m, 10 H); 6.05 (s, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 189.94, 156.93, 149.10, 134.30, 131.89, 130.76, 129.17, 129.12, 129.09, 129.02, 128.89, 128.20, 127.53, 126.07, 125.83, 123.79, 114.18, 96.26 ppm; IR (neat): $\tilde{\nu}$ = 3028, 3002, 2955, 2927, 2852, 1733, 1669, 1599, 1580, 1438, 1254, 1203, 1172, 1066, 951, 722 cm^{-1}; MS (EI, 70 ev) m/z (%): 386 (M^+, 35.86), 358 (12.64), 118 (100). HRMS calcd for $C_{24}H_{15}ClO_3$: 386.0710, found: 386.0710.

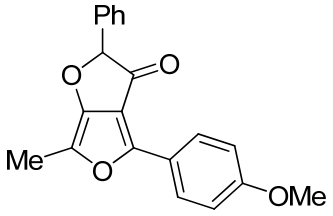
29. 6-methyl-2,4-diphenylfuro[3,4-b]furan-3(2H)-one (3m).



3m

The reaction of **1m** (104.9 mg, 0.4 mmol), $[Rh(COD)Cl]_2$ (19.8 mg, 0.04 mmol) and **L3** (15.7 mg, 0.04 mmol) in $ClCH_2CH_2Cl$ (4 mL) was carried out in the presence of 1 atm of CO (balloon) at 70 ^oC for 18 hours to afford **3m** (99.8 mg) in 86% yield as a yellow solid. m.p. 115 - 117 ^oC; ^1H NMR (400 MHz, $CDCl_3$): δ = 8.09 (d, 2 H, *J* = 7.6 Hz); 7.51 - 7.35 (m, 8 H); 5.93 (s, 1 H); 2.45 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 190.62, 155.51, 148.38, 134.97, 129.89, 128.78, 128.75, 128.68, 128.64, 127.01, 125.99, 125.26, 113.03, 95.41, 10.56 ppm; IR (neat): $\tilde{\nu}$ = 3027, 3002, 2955, 2929, 2852, 1733, 1673, 1598, 1579, 1436, 1253, 1203, 1172, 1067, 967, 723 cm^{-1}; MS (EI, 70 ev) m/z (%): 290 (M^+, 11.41), 262 (2.29), 43 (100). HRMS calcd for $C_{19}H_{14}O_3$: 290.0943, found: 290.0944.

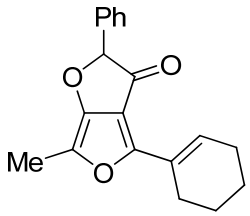
30. 4-(4-methoxyphenyl)-6-methyl-2-phenylfuro[3,4-b]furan-3(2H)-one (3n).



3n

The reaction of **1n** (116.8 mg, 0.4 mmol), $[Rh(COD)Cl]_2$ (19.8 mg, 0.04 mmol) and **L3** (15.7 mg, 0.04 mmol) in $ClCH_2CH_2Cl$ (4 mL) was carried out in the presence of 1 atm of CO (balloon) at 70 ^oC for 36 hours to afford **3n** (85.8 mg) in 67% yield as a yellow solid. m.p. 117 - 119 ^oC; ^1H NMR (400 MHz, $CDCl_3$): δ = 8.03 (d, 2 H, *J* = 7.6 Hz); 7.49 - 7.34 (m, 5 H); 6.94 (d, 2 H, *J* = 8.0 Hz); 5.91 (s, 1 H); 3.83 (s, 3 H); 2.41 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 190.35, 161.04, 155.14, 149.04, 135.21, 128.71, 128.68, 127.23, 126.03, 125.91, 121.81, 114.26, 111.53, 95.47, 55.29, 10.54 ppm; IR (neat): $\tilde{\nu}$ = 3026, 3002, 2955, 2920, 2853, 1733, 1676, 1660, 1598, 1578, 1436, 1250, 1203, 1172, 1068, 967, 719 cm^{-1}; MS (EI, 70 ev) m/z (%): 320 (M^+, 26.59), 43 (100). HRMS calcd for $C_{20}H_{16}O_4$: 320.1049, found: 320.1049.

31. 4-cyclohexenyl-6-methyl-2-phenylfuro[3,4-b]furan-3(2H)-one (3o).

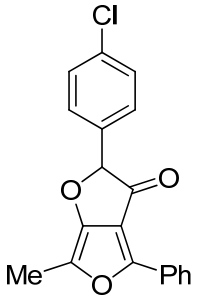


3o

The reaction of **1o** (106.5 mg, 0.4 mmol), $[Rh(COD)Cl]_2$ (19.8 mg, 0.04 mmol) and **L3** (15.7 mg, 0.04 mmol) in $ClCH_2CH_2Cl$ (4 mL) was carried out in the presence of 1 atm of CO (balloon) at 70 ^oC for 42 hours to afford **3o** (68.3 mg) in 58% yield as a yellow oil. ^1H NMR (400 MHz, $CDCl_3$): δ = 7.42 - 7.29 (m, 5 H); 6.95 (br, 1 H); 5.80 (s, 1 H); 2.42 (br, 2 H); 2.32 (s, 3 H); 2.23 (br, 2 H); 1.71 - 1.61 (m, 4 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 190.25, 155.14, 151.08, 135.29, 132.22, 128.58, 127.93, 125.92, 125.28, 111.31, 95.28, 25.70, 23.81, 21.82, 21.68, 10.41 ppm; IR (neat): $\tilde{\nu}$ = 3028, 3002, 2955, 2924, 2853, 1733, 1669, 1598, 1580, 1436, 1253, 1203, 1172, 1068, 967, 722 cm^{-1};

MS (EI, 70 ev) m/z (%): 294 (6.64), 266 (17.21), 43 (100). HRMS calcd for $C_{19}H_{18}O_3$: 294.1256, found: 294.1255.

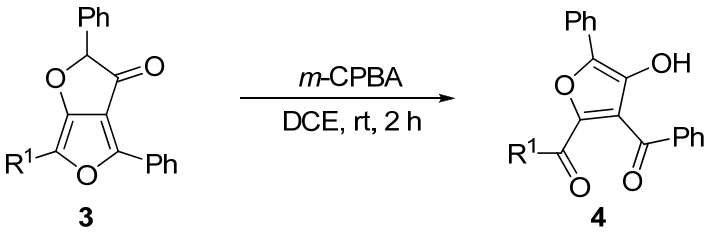
32. 2-(4-chlorophenyl)-6-methyl-4-phenylfuro[3,4-b]furan-3(2H)-one (3p).



3p

The reaction of **1p** (118.4 mg, 0.4 mmol), $[Rh(COD)Cl]_2$ (19.8 mg, 0.04 mmol) and **L3** (15.7 mg, 0.04 mmol) in $ClCH_2CH_2Cl$ (4 mL) was carried out in the presence of 1 atm of CO (balloon) at 70 ^oC for 36 hours to afford **3p** (78.8 mg) in 61% yield as a yellow solid. m.p. 128 - 130 ^oC; ^1H NMR (400 MHz, $CDCl_3$): δ = 8.03 (d, 2 H, J = 7.6 Hz); 7.44 - 7.33 (m, 7 H); 5.86 (s, 1 H); 2.42 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 190.16, 155.37, 148.71, 134.72, 133.43, 130.09, 128.90, 128.87, 128.58, 127.30, 127.20, 125.35, 112.74, 94.58, 10.62 ppm; IR (neat): $\tilde{\nu}$ = 3028, 3002, 2955, 2927, 2852, 1733, 1671, 1599, 1579, 1437, 1254, 1203, 1172, 1067, 967, 722 cm^{-1}; MS (EI, 70 ev) m/z (%): 324 (M^+, 23.44), 296 (1.74), 152 (100). HRMS calcd for $C_{19}H_{13}ClO_3$: 324.0553, found: 324.0554.

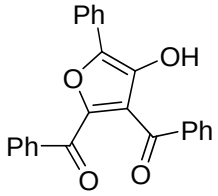
Synthsis of tetrasubstituted furans 4.



3 **4**

To a stirred solution of **3** (0.4 mmol) in $ClCH_2CH_2Cl$ (4 mL) at rt, *m*-CPBA (85%, 89.4 mg, 0.44 mmol) was added. The result solution was then stired at rt for 2 hours. After evaporation, the residue was purified by column chromatography on silica gel (hexanes/ EtOAc = 20:1) afforded the desired products **4**.

33. (4-hydroxy-5-phenylfuran-2,3-diyl)bis(phenylmethanone) (4a).

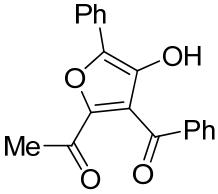


4a

The reaction of **3a** (141.0 mg, 0.4 mmol), *m*-CPBA (85%, 89.4 mg, 0.44 mmol) in $ClCH_2CH_2Cl$ (4 mL) at rt for 2 hours to afford **4a** (126.7 mg) in 86% yield as a yellow solid. m.p. 201 - 203 ^oC; ^1H NMR (400 MHz, $CDCl_3$): δ = 7.93 (d, 2 H, *J* = 7.6 Hz); 7.88 (s, 1 H); 7.82 (d, 2 H, *J* = 7.6 Hz); 7.70 (d, 2 H, *J* = 7.6 Hz); 7.54 (t, 1 H, *J* = 7.2 Hz); 7.49 - 7.38 (m, 5 H); 7.33 - 7.28 (m, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 194.02, 182.22, 147.54, 143.24, 139.24, 137.97, 136.80, 133.37, 133.15, 129.69, 128.83, 128.78, 128.43, 128.00, 124.32, 121.00 ppm; IR (neat): $\tilde{\nu}$ = 3469, 3029, 3003, 2955, 2924, 2852, 1733, 1669, 1599, 1580, 1436, 1254, 1202, 1173, 1067, 967, 724 cm^{-1}; MS (EI, 70 ev) m/z (%): 368 (M^+, 8.25), 41 (100). HRMS calcd for $C_{24}H_{16}O_4$: 368.1049, found: 368.1052.

Keeping the solution of **3a** in THF stirring in the air for 24 hours afforded **4a** in 16% yield. When 1 equivalent of 2.6-lutidine was added, 48% of **4a** could be obtained within 4 hours.

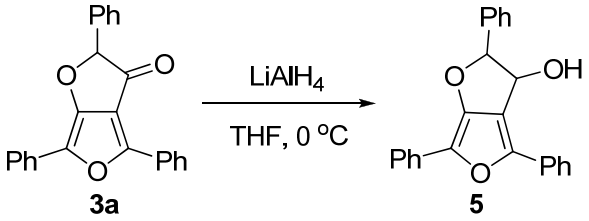
34. 1-(3-benzoyl-4-hydroxy-5-phenylfuran-2-yl)ethanone (4m).



4m

The reaction of **3m** (116.1 mg, 0.4 mmol), *m*-CPBA (85%, 89.4 mg, 0.44 mmol) in ClCH2CH2Cl (4 mL) at rt for 2 hours to afford **4m** (101.7 mg) in 83% yield as a yellow solid. m.p. 171 - 173 ^oC; ^1H NMR (400 MHz, $CDCl_3$): δ = 7.96 (d, 2 H, *J* = 7.6 Hz); 7.82 (d, 2 H, *J* = 7.6 Hz); 7.63 - 7.59 (m, 2 H); 7.50 - 7.43 (m, 4 H); 7.35 (t, 1 H, *J* = 7.4 Hz); 2.50 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 193.98, 186.62, 146.11, 143.18, 139.35, 137.53, 133.84, 129.28, 128.85, 128.80, 128.47, 128.24, 124.41, 120.19, 26.40 ppm; IR (neat): $\tilde{\nu}$ = 3469, 3029, 3003, 2955, 2924, 2852, 1733, 1670, 1598, 1581, 1436, 1254, 1202, 1173, 1068, 967, 722 cm^{-1}; MS (EI, 70 ev) m/z (%): 306 (M^+, 25.37), 105 (100). HRMS calcd for $C_{19}H_{14}O_4$: 306.0892, found: 306.0896.

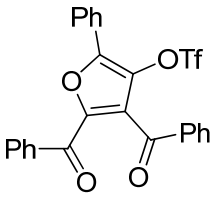
35. Synthsis of 5.



3a **5**

To a stirred solution of **3a** (141.0 mg, 0.4 mmol) in THF (4 mL) at 0 ^{o}C, $LiAlH_4$ (22.8 mg, 0.6 mmol) was added. The result solution was then stired at 0 ^{o}C for 1 hour. Then the reaction mixture was added water (4 mL), and extracted with diethyl ether (3 × 4 mL). The combined organic layers were washed with brine, dried over magnesium sulfate and concentrated under vaccuo. The crude residue was purified by flash chromatography on silica gel (hexanes : EtOAc = 10 : 1) to give the desired product **5** (113.6 mg, 80% yield) as mixtured yellow solid (major / minor = 7.3 / 1). ^{1}H NMR (400 MHz, $CDCl_3$): δ = [7.77 (d, 1.76 H, *J* = 7.6 Hz), 7.73 (d, 0.24 H, *J* = 7.6 Hz)]; 7.69 (d, 2 H, *J* = 7.6 Hz); 7.50 - 7.36 (m, 9 H); 7.28 (t, 1 H, *J* = 7.2 Hz); 7.17 (t, 1 H, *J* = 7.2 Hz); [6.08 (d, 0.88 H, *J* = 5.6 Hz), 5.97 (br, 0.12 H)]; [5.38 (d, 0.88 H, *J* = 5.6 Hz), 5.26 (br, 0.12 H)]; [1.57 (br, 0.88 H), 1.42 (br, 0.12 H)]. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 150.86, 144.39, 134.19, 130.01, 129.87, 128.79, 128.67, 128.61, 127.90, 127.02, 125.70, 125.25, 124.46, 124.41, (122.65, 122.57), (120.33, 119.53), (102.88, 98.22), (76.02, 69.48) ppm, IR (neat): $\tilde{\nu}$ = 3465, 3029, 3003, 2955, 2929, 2852, 1733, 1670, 1598, 1580, 1439, 1254, 1203, 1173, 1086, 1071, 1058, 951, 726, 700 cm^{-1}; MS (EI, 70 ev) m/z (%): 354 (M^+, 22.66), 105 (100). HRMS calcd for $C_{24}H_{18}O_3$: 354.1256, found: 354.1260.

36. 4,5-dibenzoyl-2-phenylfuran-3-yl trifluoromethanesulfonate (6).



6

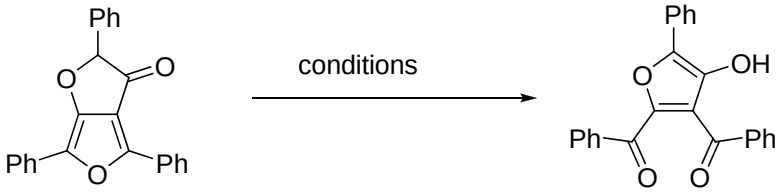
From 3: To a stirred solution of diisopropylamine (0.5 mL, 3.3 mmol) in anhydrous THF (10 mL) was added n-BuLi (1.1 mL, 2.5 M solution in hexane, 2.7 mmol) at -78 ^{o}C and stirred for 30 minutes at same temperature under N_2. To the resulting mixture was added a solution of **3a** (634.3 mg, 1.8 mmol) in anhydrous THF (5 mL) dropwise with syringe. The reaction mixture was stirred at

the same temperature for 2 hours under N_2 and to this was added solid N-phenyltrifluoromethanesulfonimide ($PhNTf_2$) (971.1 mg, 2.7 mmol) in one portion. The resulting solution was allowed to stir at -78 ^oC for 3 h and then at room temperature for 16 h in the air. After removing the solvent under reduced pressure, the crude residue was purified by column chromatography on silica gel (hexanes : EtOAc = 20 : 1) to afford the product **6** (603.8 mg, 67% yield) as a yellow solid. m.p. 152 - 154 ^oC; ^1H NMR (400 MHz, $CDCl_3$): δ = 7.91 - 7.87 (m, 4 H); 7.83 (d, 2 H, *J* = 7.2 Hz); 7.59 - 7.53 (m, 5 H); 7.46 - 7.37 (m, 4 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 187.38, 181.67, 147.41, 147.14, 136.85, 135.95, 133.80, 133.50, 131.39, 130.85, 129.50, 129.24, 129.14, 128.60, 128.55, 127.13, 126.35, 126.00, [(122.99, 119.80, 116.60, 113.41), J_{CF} = 239.5 Hz] ppm; ^{19}F NMR (376 MHz, $CDCl_3$): δ = -72.94 ppm; IR (neat): $\tilde{\nu}$ = 3468, 3029, 3002, 2953, 2925, 2852, 1733, 1668, 1599, 1580, 1446, 1254, 1200, 1067, 968, 723 cm^{-1}; MS (EI, 70 ev) m/z (%): 500 (M^+, 1.88), 262 (51.12), 105 (100). HRMS calcd for $C_{25}H_{15}F_3O_6S$: 500.0541, found: 500.0542.

From 4a: To a stirred solution of **4a** (110.5 mg, 0.3 mmol) and DMAP (55.0 mg, 0.45 mmol) in anhydrous DCM (3 mL) was added Tf_2O (0.15 mL, 0.9 mmol) at 0 ^oC. The resulting solution was allowed to stir for 20 min. After removing the solvent under reduced pressure, the crude residue was purified by column chromatography on silica gel (hexanes : EtOAc = 20 : 1) to afford the product **6** (137.6 mg, 92% yield) as a yellow solid.

37. Experiments supporting the point that in the conversion of 3 to 6, air plays the role of oxidant.

Table 1. Experiments to prove that this kind of 2,5-diphenylfurans could be easily oxidated by air, especially in the presence of base, which could result in the enolization of **3a** and make it more electron-rich.[a]

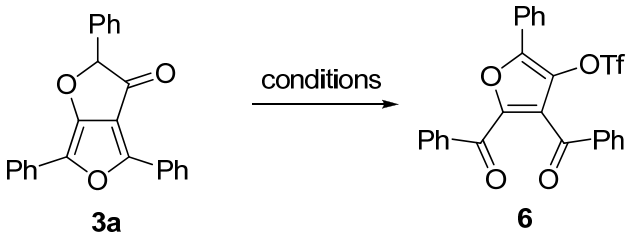


3a **4a**

Entry	Conditions	Convertion (**3a**, %)[b]	Yield (**4a**, %)[c]
1	THF, air, rt, 24 h	20	16
2	2,6-lutidine (1 eq.), THF, air, rt, 4 h	100	48[d]
3	2,6-lutidine (1 eq.), DCM, air, rt, 3.5 h	100	43[d]

[a] The reactions were performed with 0.3 mmol of **3a** in the air in THF or DCM at rt. [b] Determined by NMR analysis. [c] Isolated yield. [d] With some by-pruducts whose structures have not been established.

Table 2. Experiments to prove that the corresponding heterobicyclic furo[3,4-b]furan-3-yl triflate is easily converted into product **6** in the air, even in the presence of the trace amount of oxygen dissolved in the solvents.



3a **6**

Entry	Conditions	Yield (**6**, %)[a]
1[b]	2,6-lutidine (2 eq.), Tf_2O (2 eq.), DCM, N_2, 0 ^oC - rt, 24 h	61
2[c]	2,6-lutidine (2 eq.), Tf_2O (2 eq.), DCM, N_2, 0 ^oC - rt, 24 h	55[d]
3[e]	LDA (2.7 eq.), $PhNTf_2$ (2.7 eq.), THF, -78 ^oC – rt, 16 h	64

[a] Isolated yield. [b] without strict 'oxygen-free' operation. [c] The reaction was performed in strict 'oxygen-free' conditions. [d] By TLC analysis, an intermediate product could be observed obviously, which was observed just trace amount in entry 1, but after working up the reaction, we could not obtain the intermediate product, only product **6** in 55% yield.[e] A similar result with entry 2.

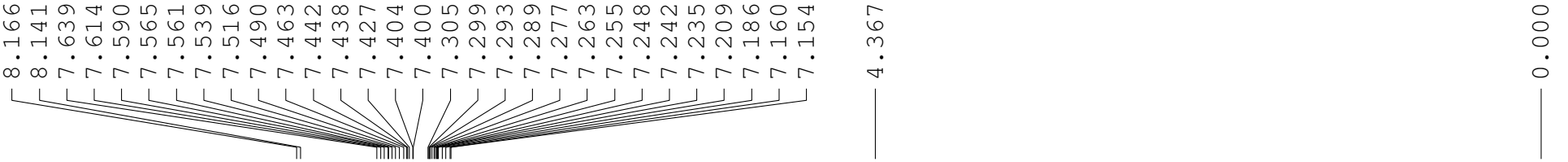
Entry 1: To a stirred solution of **3a** (105.7 mg, 0.3 mmol) and 2,6-lutidine (70 µL, 0.6 mmol) in anhydrous DCM (3 mL) was added Tf_2O (0.1 mL, 0.6 mmol) under N_2 at 0 ^oC and stirred for 30 minutes at same temperature. The resulting solution was then allowed to stir at rt for 24 hours. After removing the solvent under reduced pressure, the crude residue was purified by column chromatography on silica gel (hexanes : EtOAc = 20 : 1) to afford the product **6** (91.5 mg, 61% yield) as a yellow solid.

Entry 2: The mixed solution of **3a** (105.7 mg, 0.3 mmol), 2,6-lutidine (70 µL, 0.6 mmol) and Tf_2O (0.1 mL, 0.6 mmol) in anhydrous DCM (3 mL) was placed to liquid nitrogen until it was completely solidified. Then N_2 was charged after depressurization under vaccuo. The solution was allowed to warm slowly to become liquid state. Then the same operation was repeated 3 times. After that the solution was allowed to stir at rt for 24 hours. After removing the solvent under reduced pressure, the crude residue was purified by column chromatography on silica gel (hexanes : EtOAc = 20 : 1) to afford the product **6** (82.3 mg, 55% yield) as a yellow solid.

Entry 3: The similar operation with entry 2. 64% yield of **6** was obtained.

Reference:

[1] (a) T. Yao, X. Zhang and R. C. Larock, *J. Am. Chem. Soc.* 2004, **126**, 11164; (b) G. Zhang, X. Huang, G. Li and L. Zhang, *J. Am. Chem. Soc.* 2008, **130**, 1814; (c) F. Liu, Y. Yu and J. Zhang, *Angew. Chem. Int. Ed.* 2009, **48**, 5505; (d) H. Gao, X. Zhao, Y. Yu and J. Zhang, *Chem. Eur. J.* 2010, **16**, 456; (e) Y. Zhang, F. Liu and J. Zhang, *Chem. Eur. J.* 2010, **16**, 6146.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7.299
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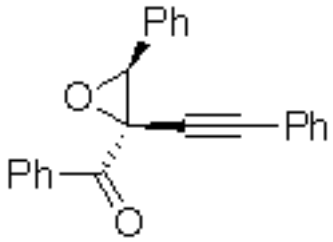
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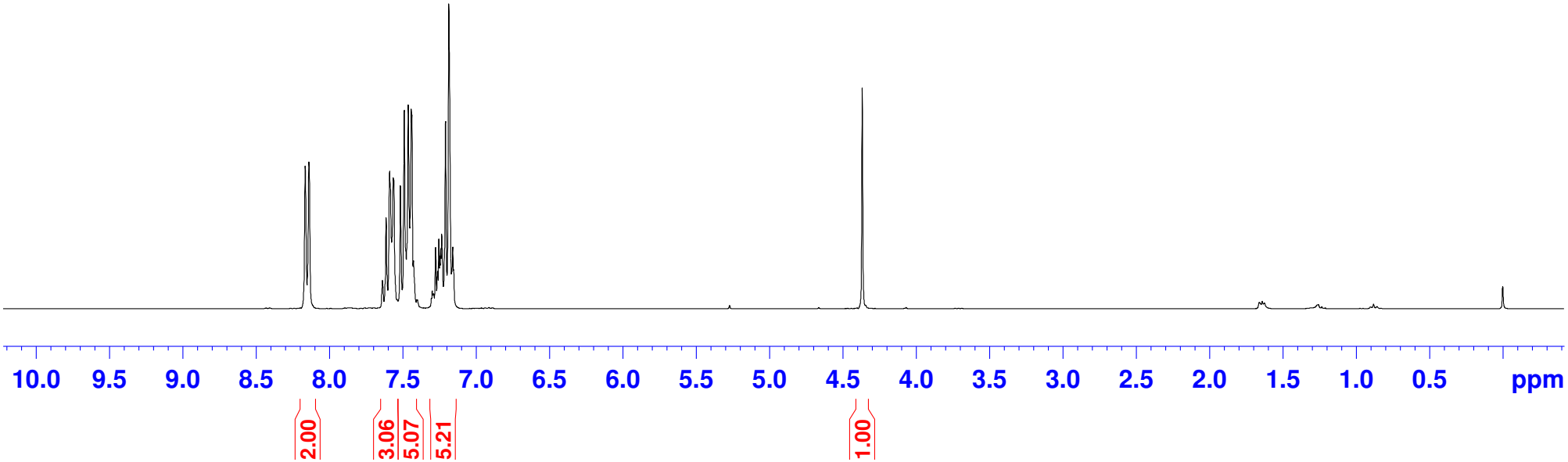
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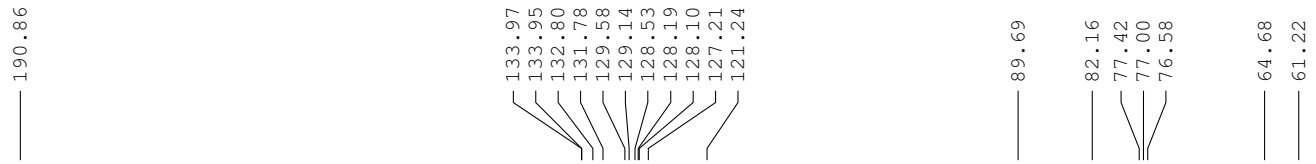
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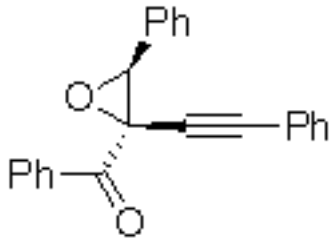
76.58
64.68
61.22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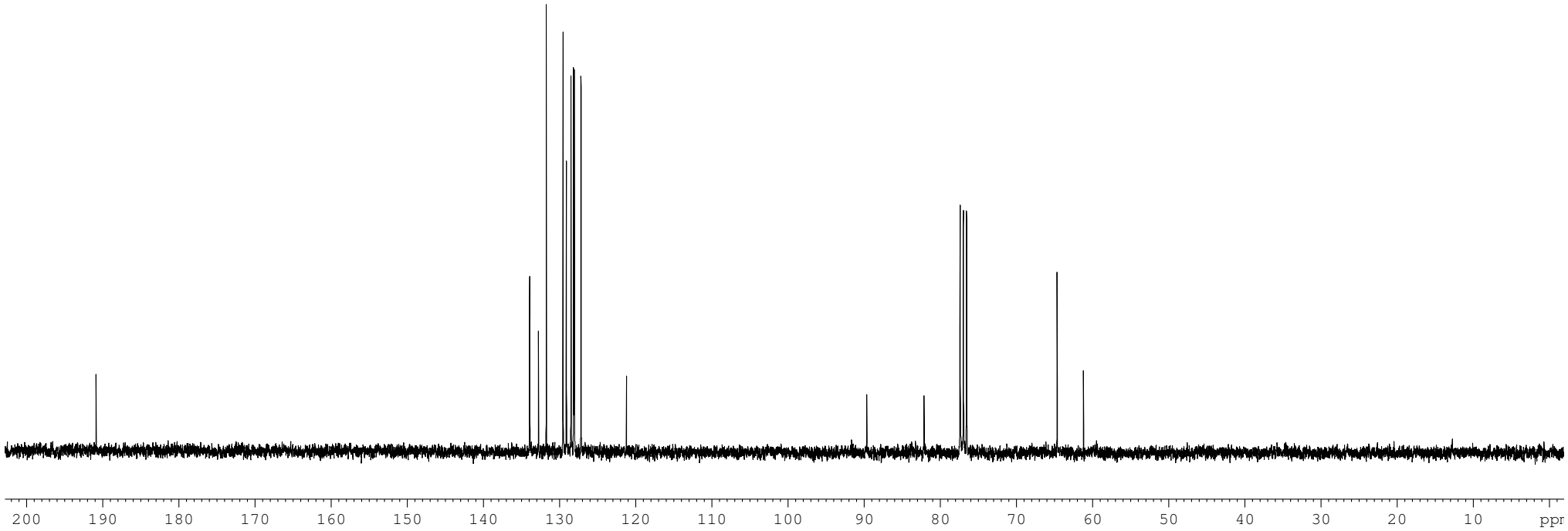
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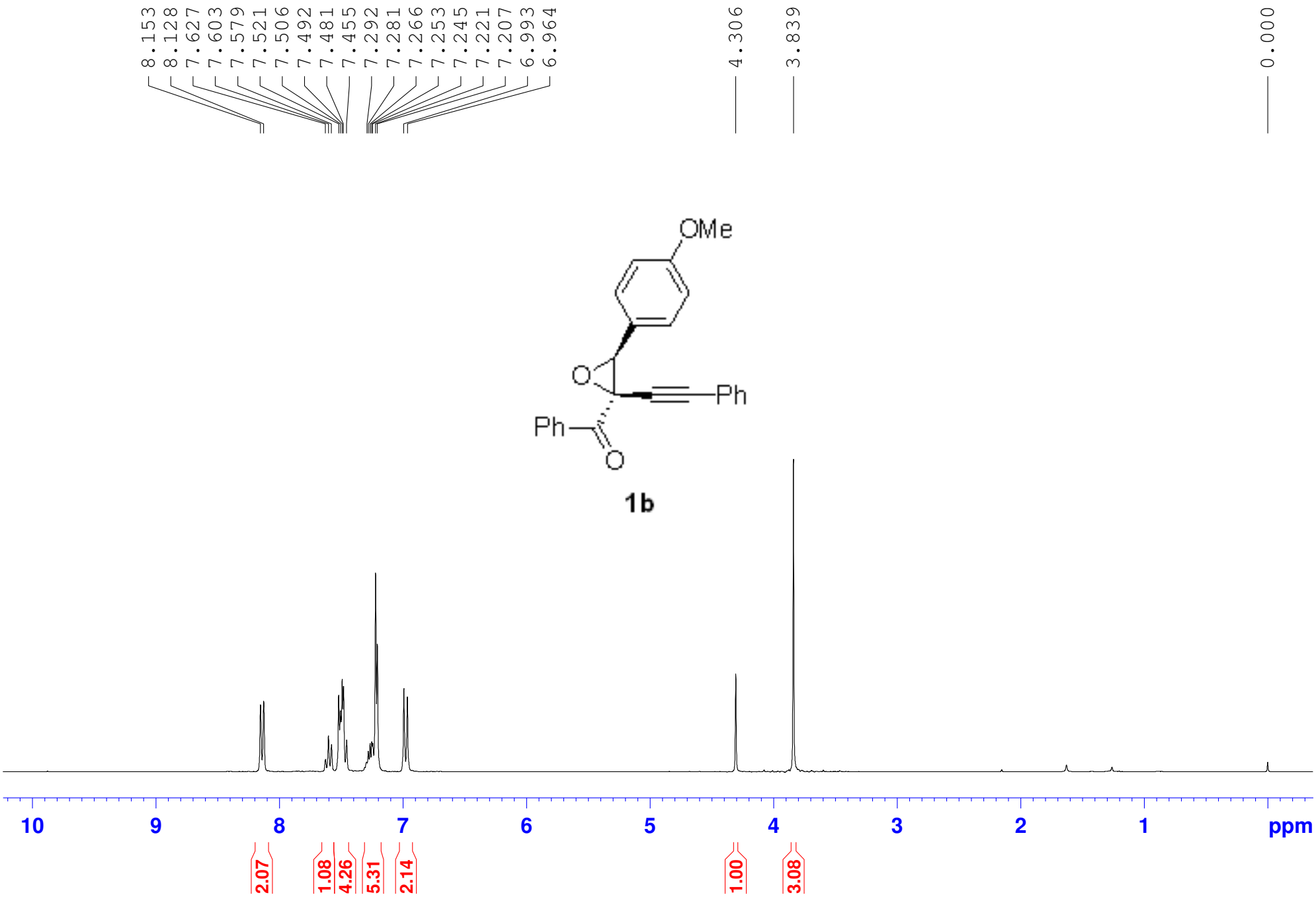
ppm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7.221
7.207
6.993
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4.306
3.839
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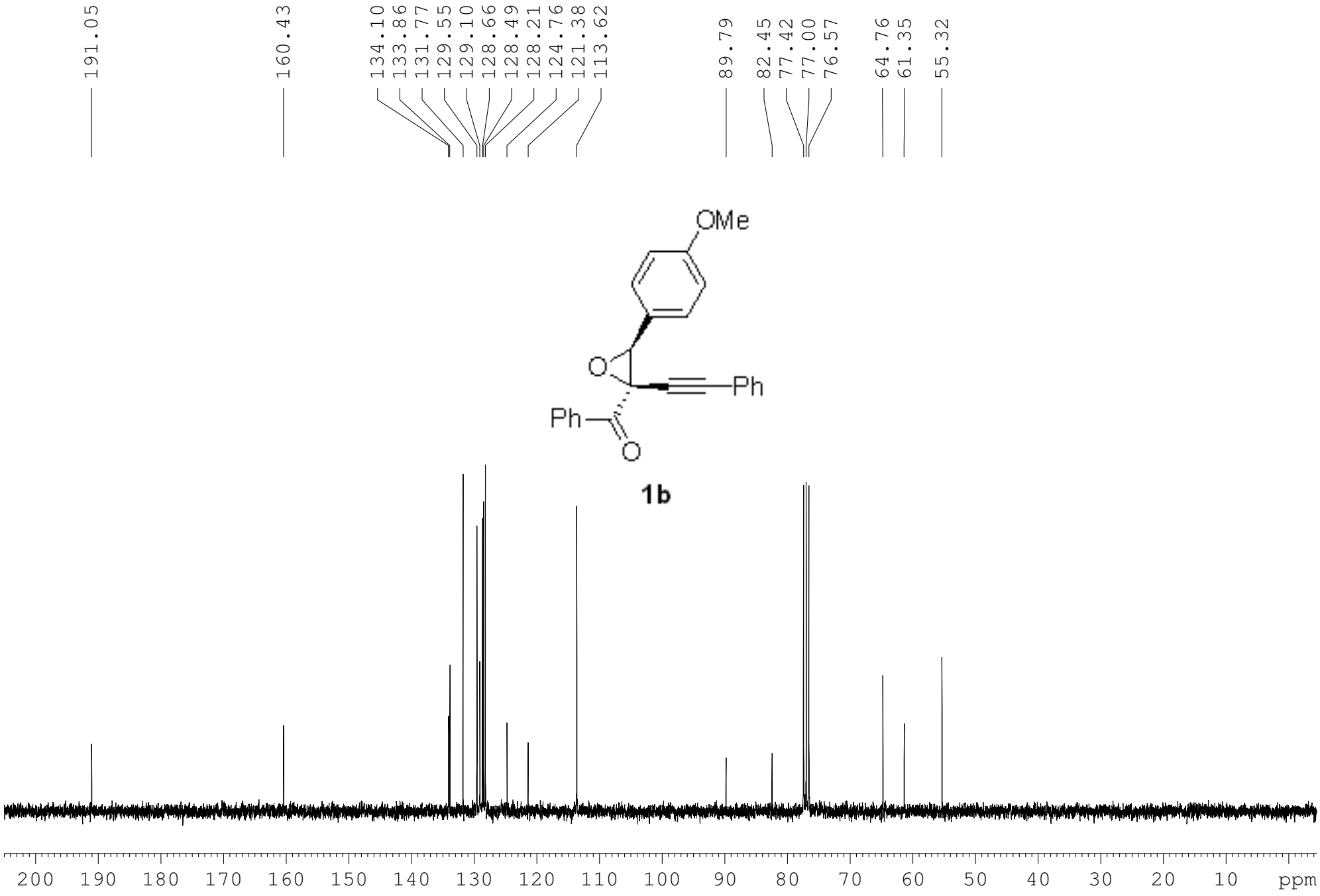
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77.42
77.00
76.57
64.76
61.35
55.32
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Ph
Ph
O
1b
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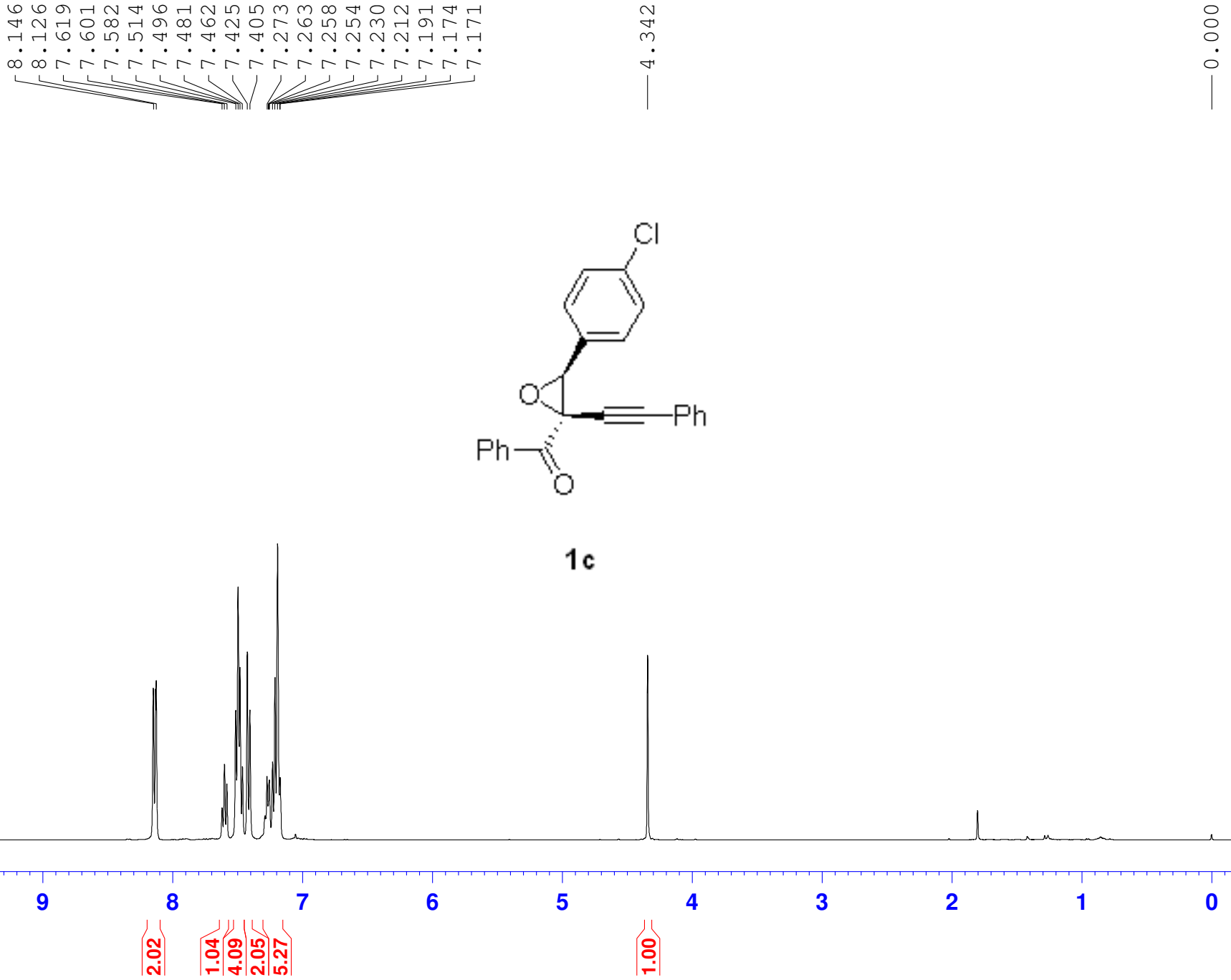
30
20
10
ppm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7.258
7.254
7.230
7.212
7.191
7.174
7.171
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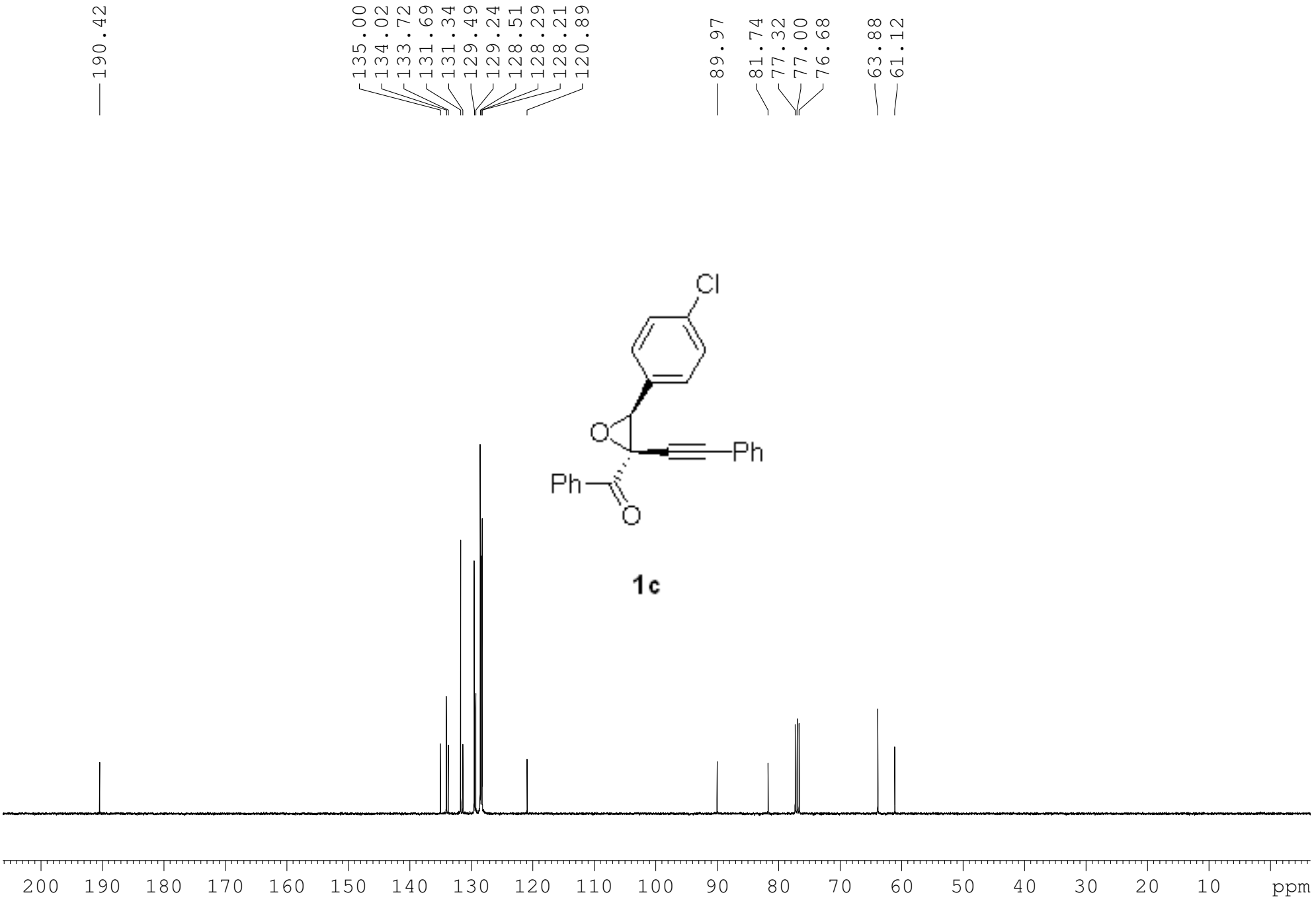
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77.00
76.68
63.88
61.12
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Ph
Ph
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ppm

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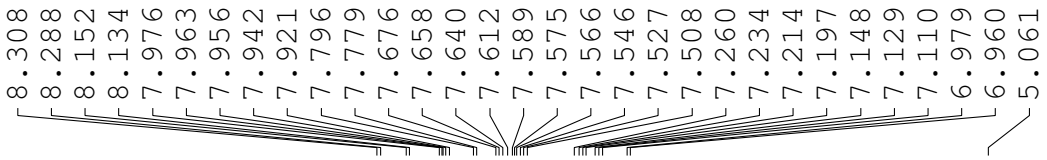
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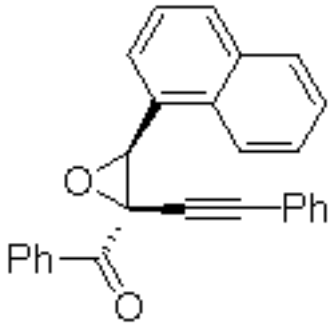
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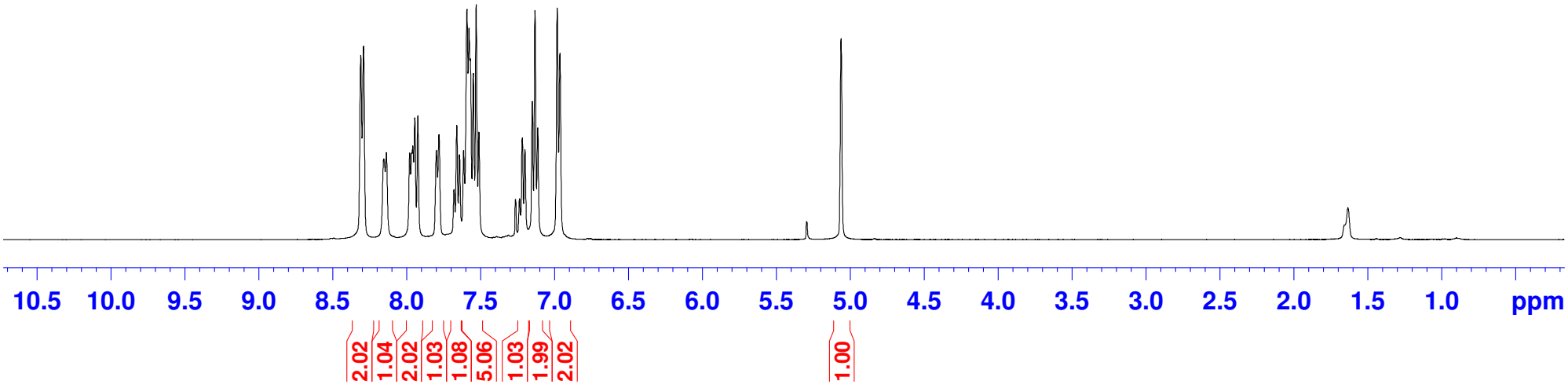
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1.03
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2.02
1.00

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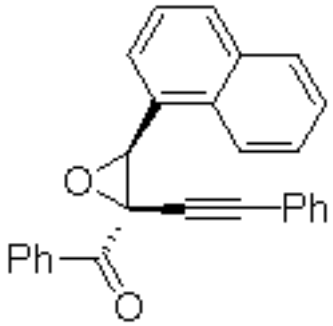

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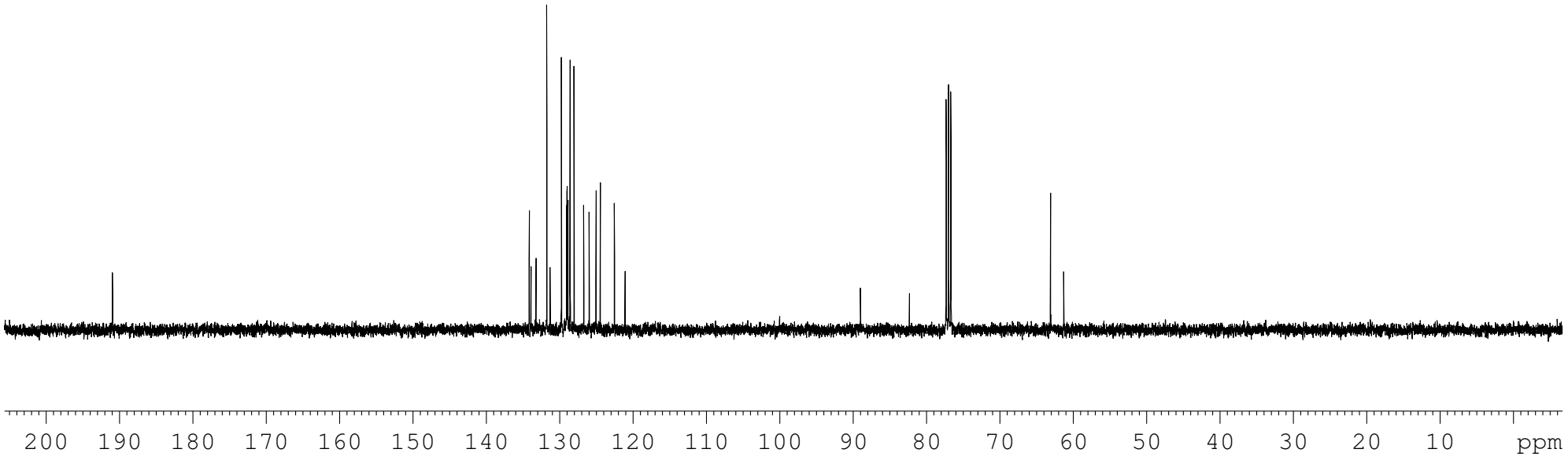

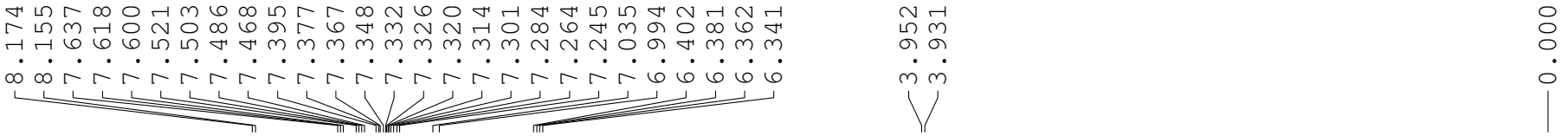
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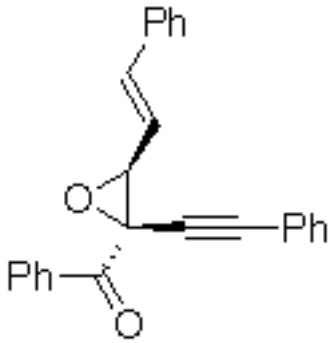
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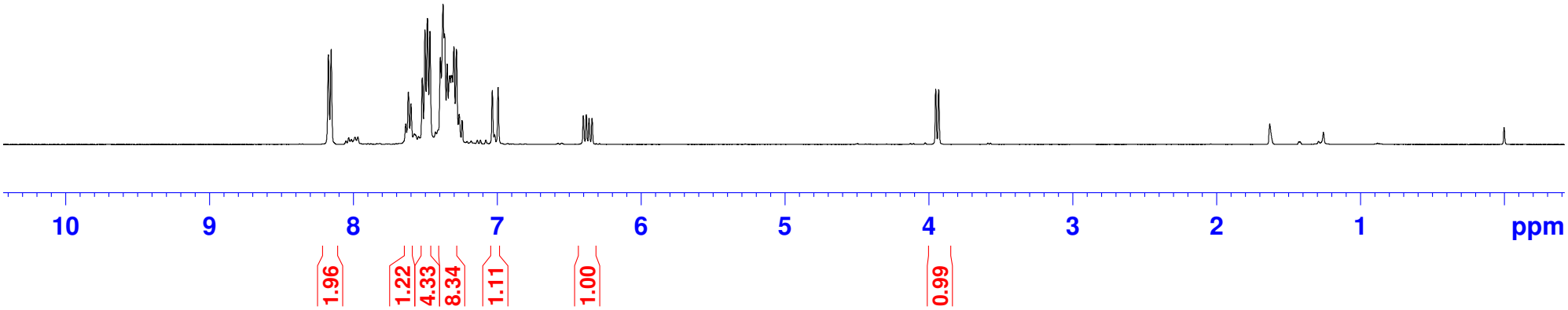
1e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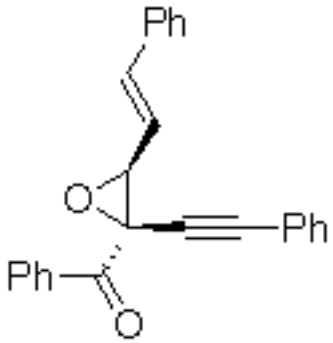

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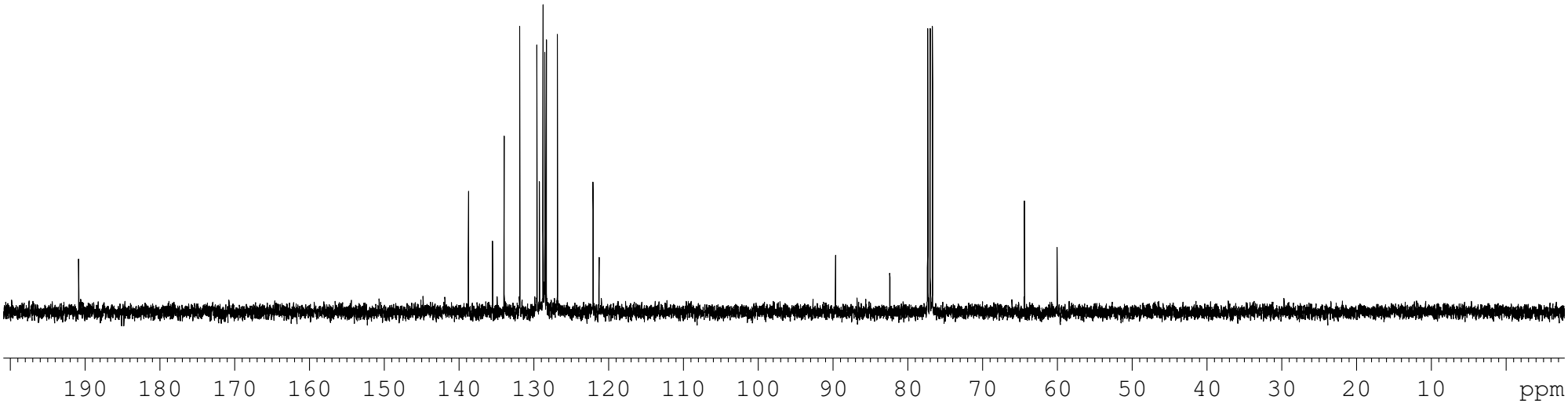

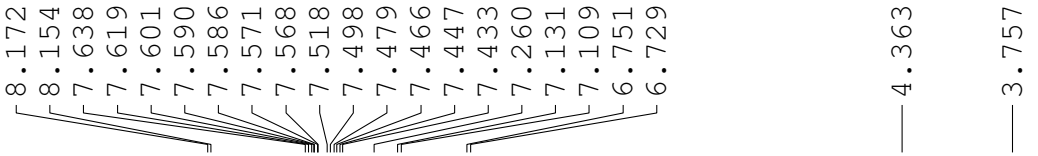
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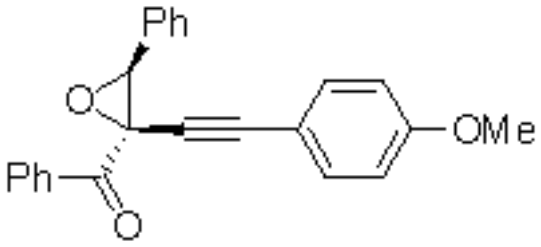
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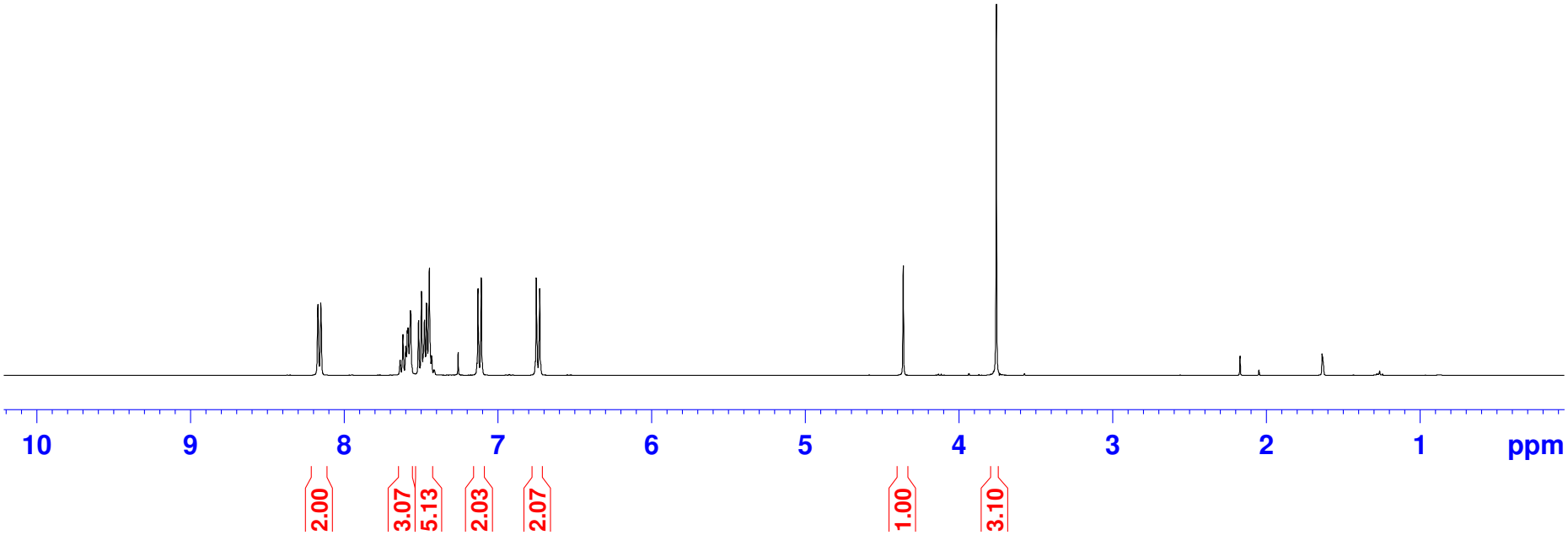
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3.10

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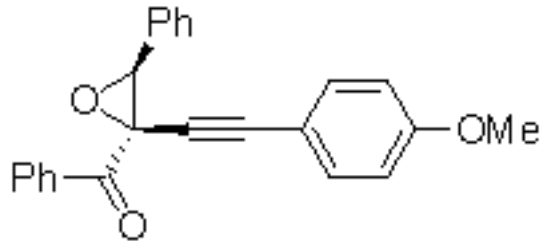

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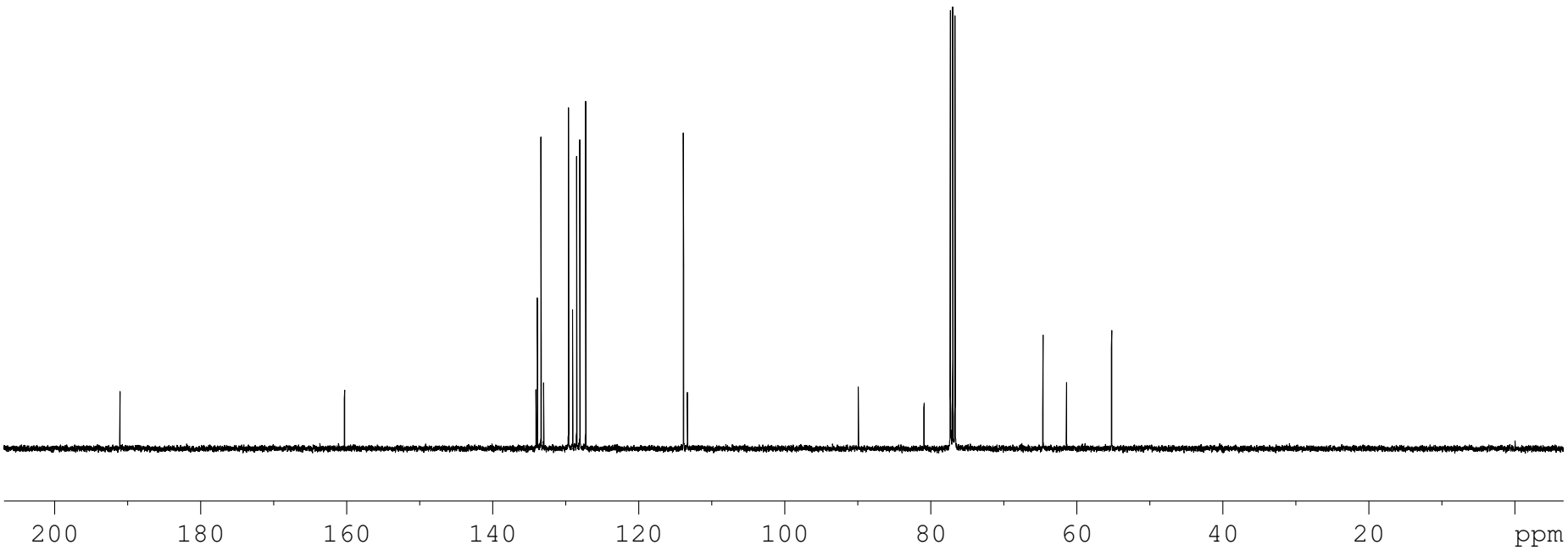

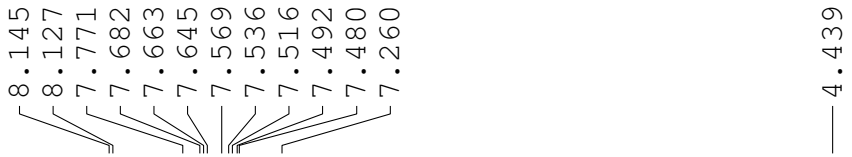
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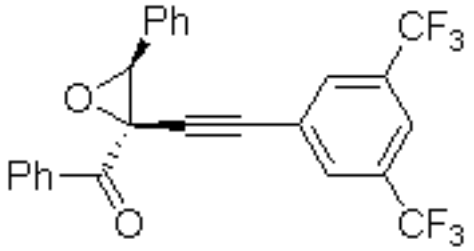
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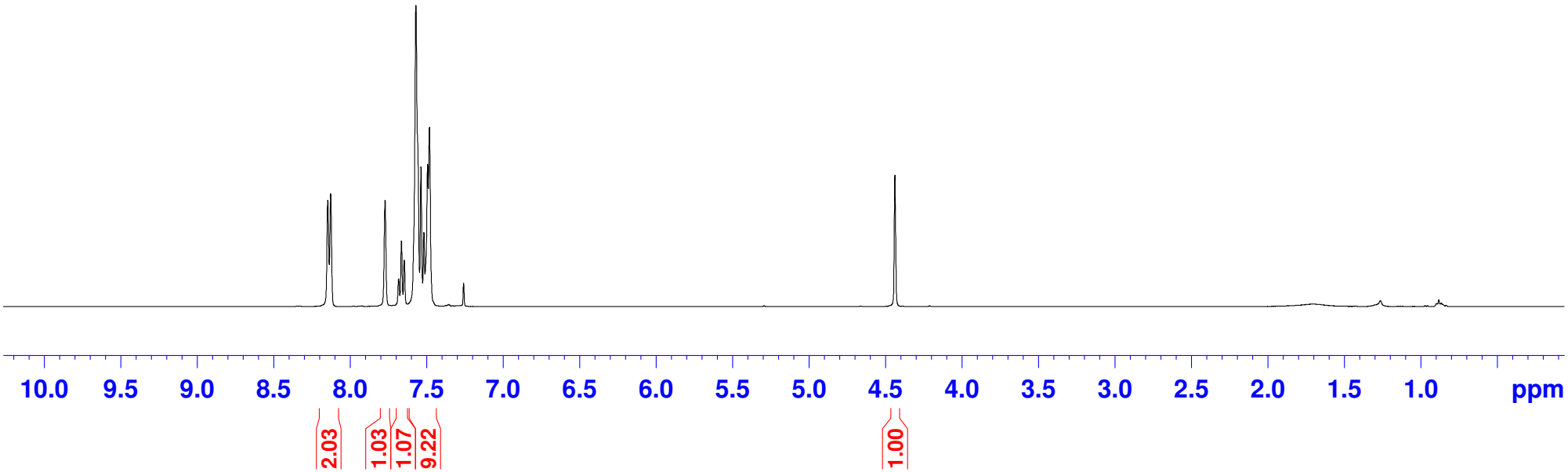
CF3

1g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8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
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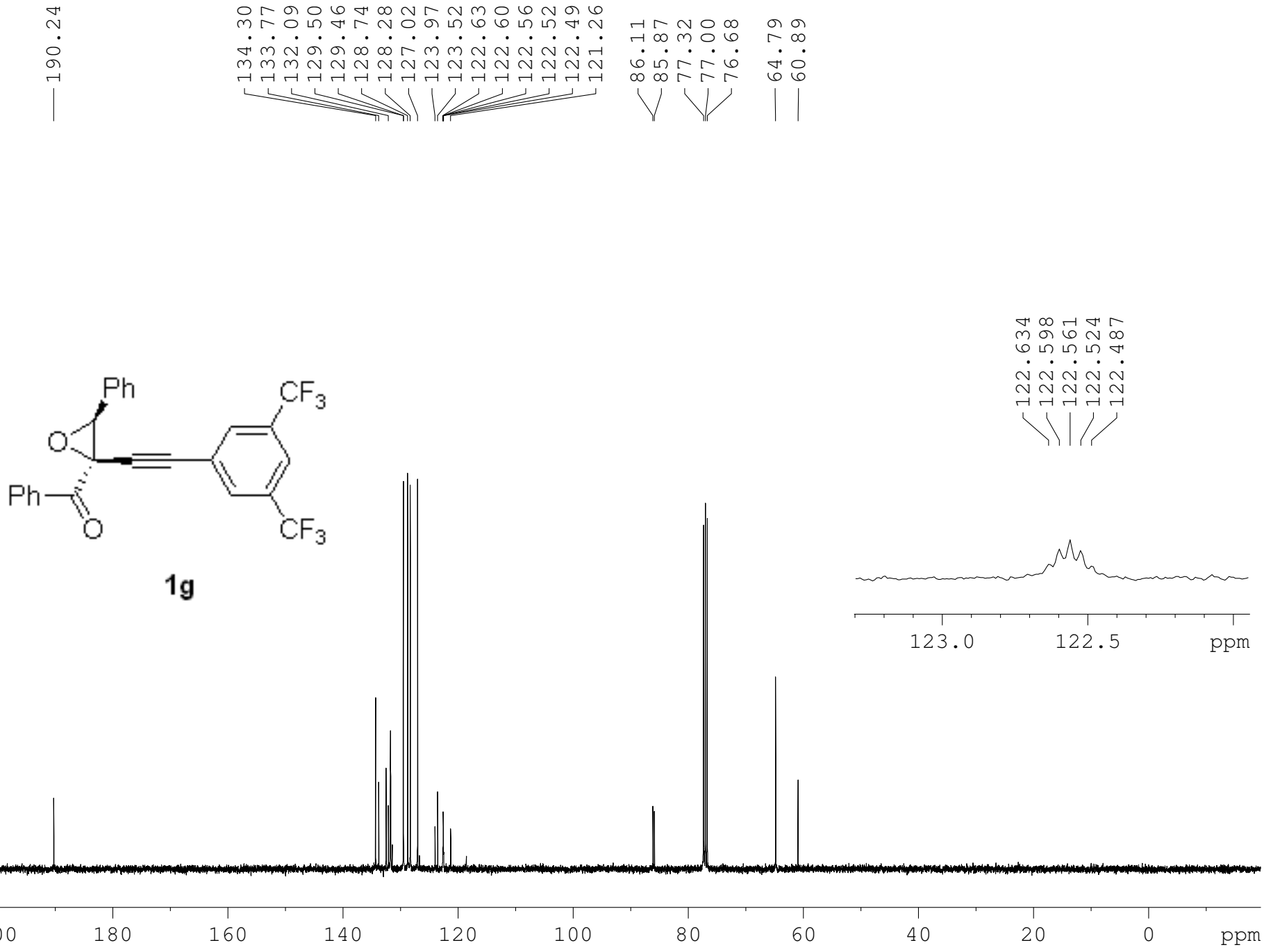
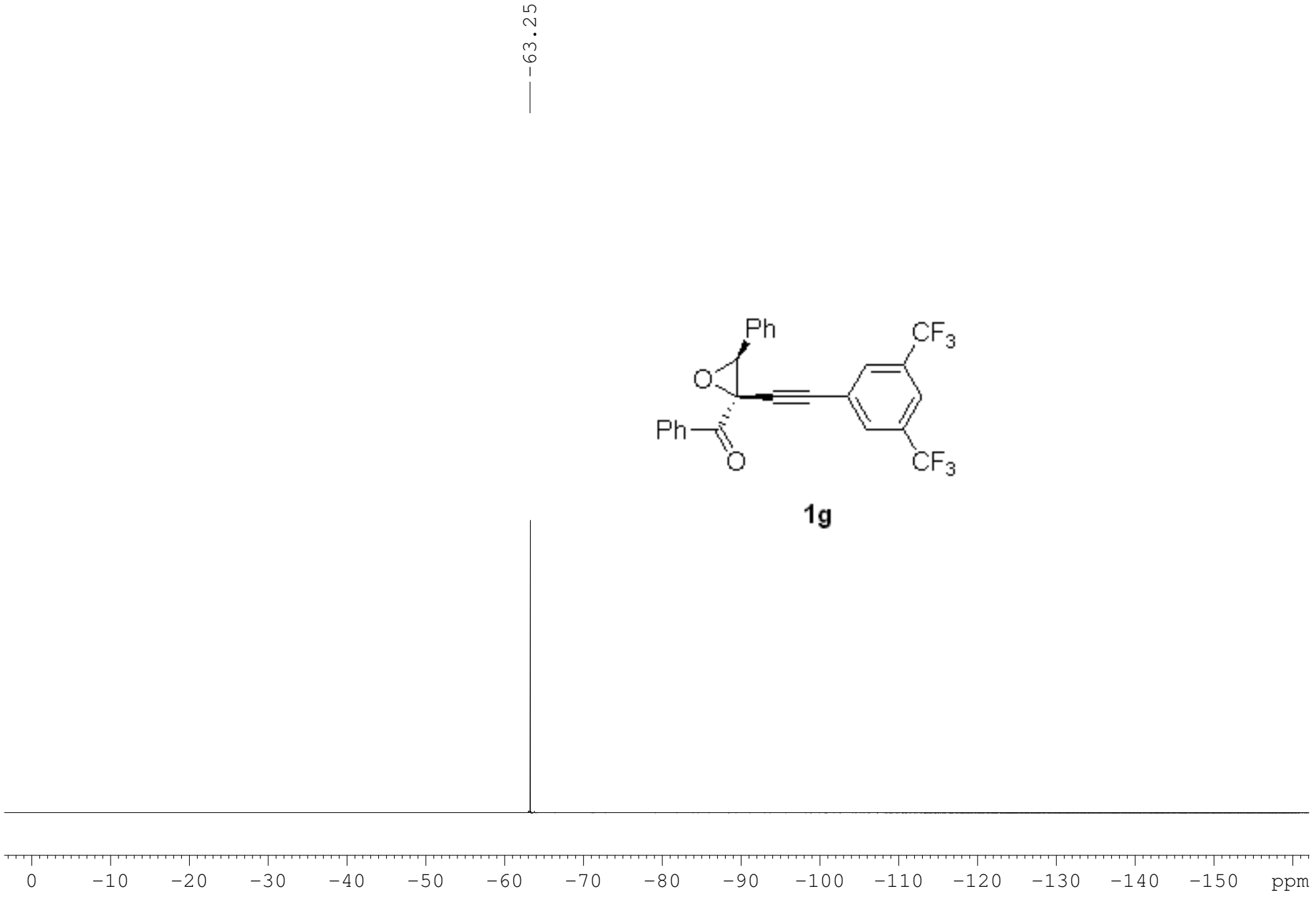
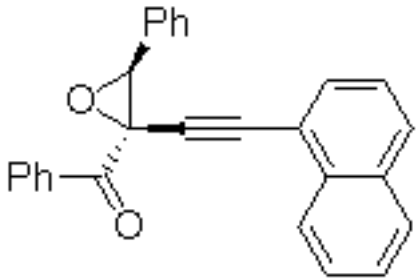
2.5
2.0
1.5
1.0
ppm
2.03
1.03
1.07
9.22
1.00

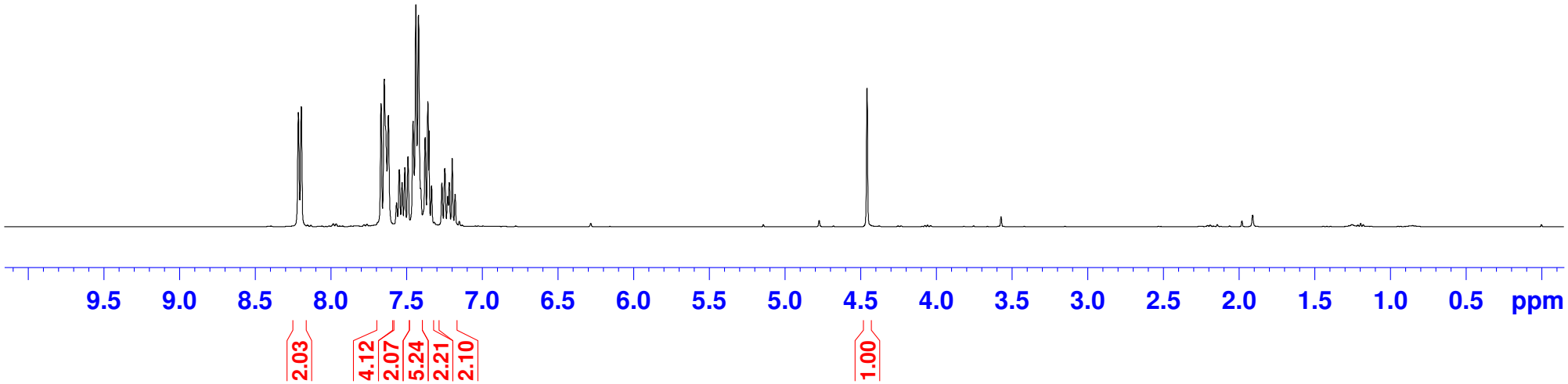

190.24
134.30
133.77
132.09
129.50
129.46
128.74
128.28
127.02
123.97
123.52
122.63
122.60
122.56
122.52
122.49
121.26
86.11
85.87
77.32
77.00
76.68
64.79
60.89
122.634
122.598
122.561
122.524
122.487
Ph
CF3
O
Ph
O
CF3
1g
123.0
122.5
ppm
200
180
160
140
120
100
80
60
40
20
0
ppm


-63.25
Ph
CF3
O
Ph
O
CF3
1g
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
ppm


8.212
8.192
7.665
7.644
7.617
7.563
7.545
7.527
7.508
7.488
7.454
7.436
7.418
7.404
7.374
7.356
7.335
7.266
7.247
7.228
7.218
7.198
7.179
4.457
0.000


Ph
O
Ph
O

1h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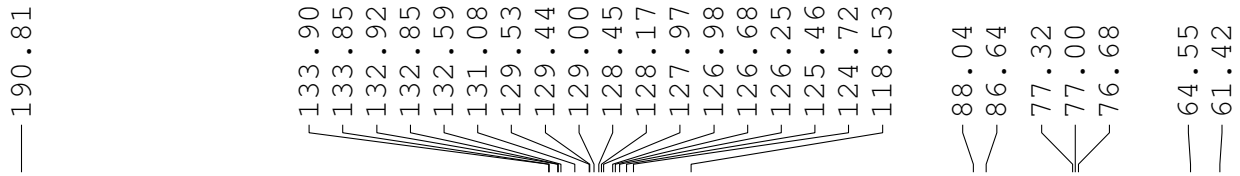
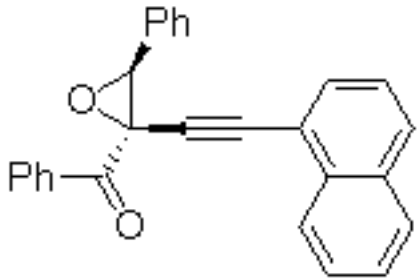
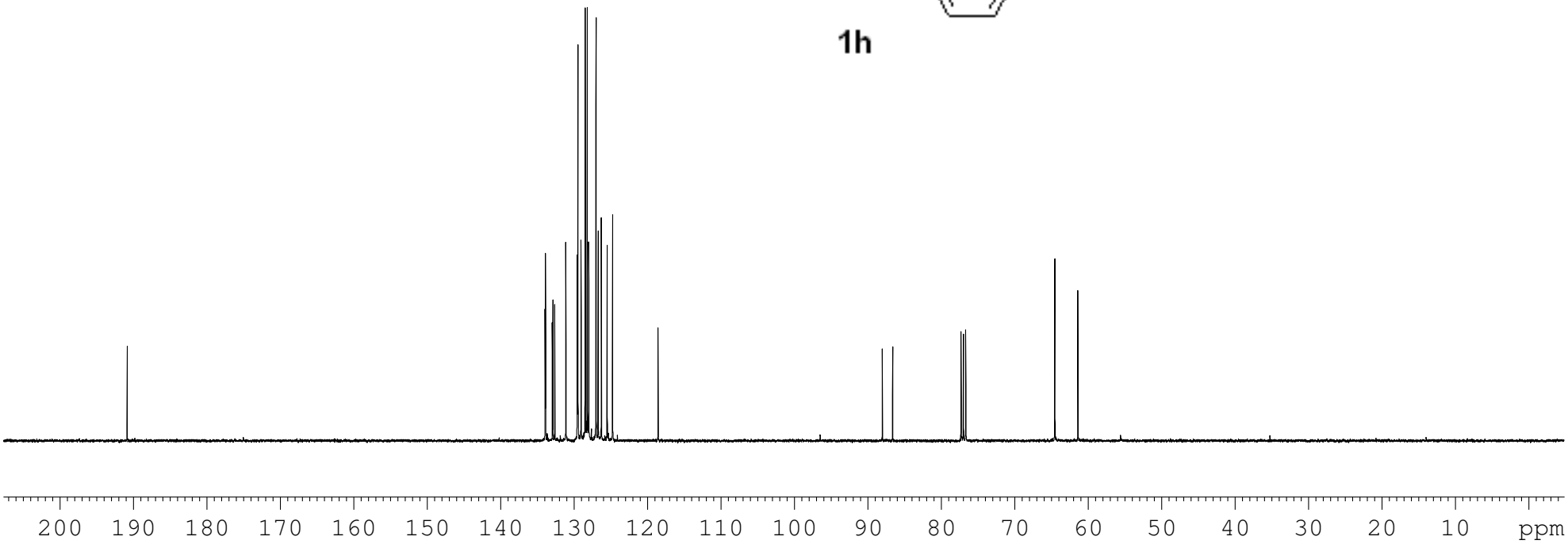
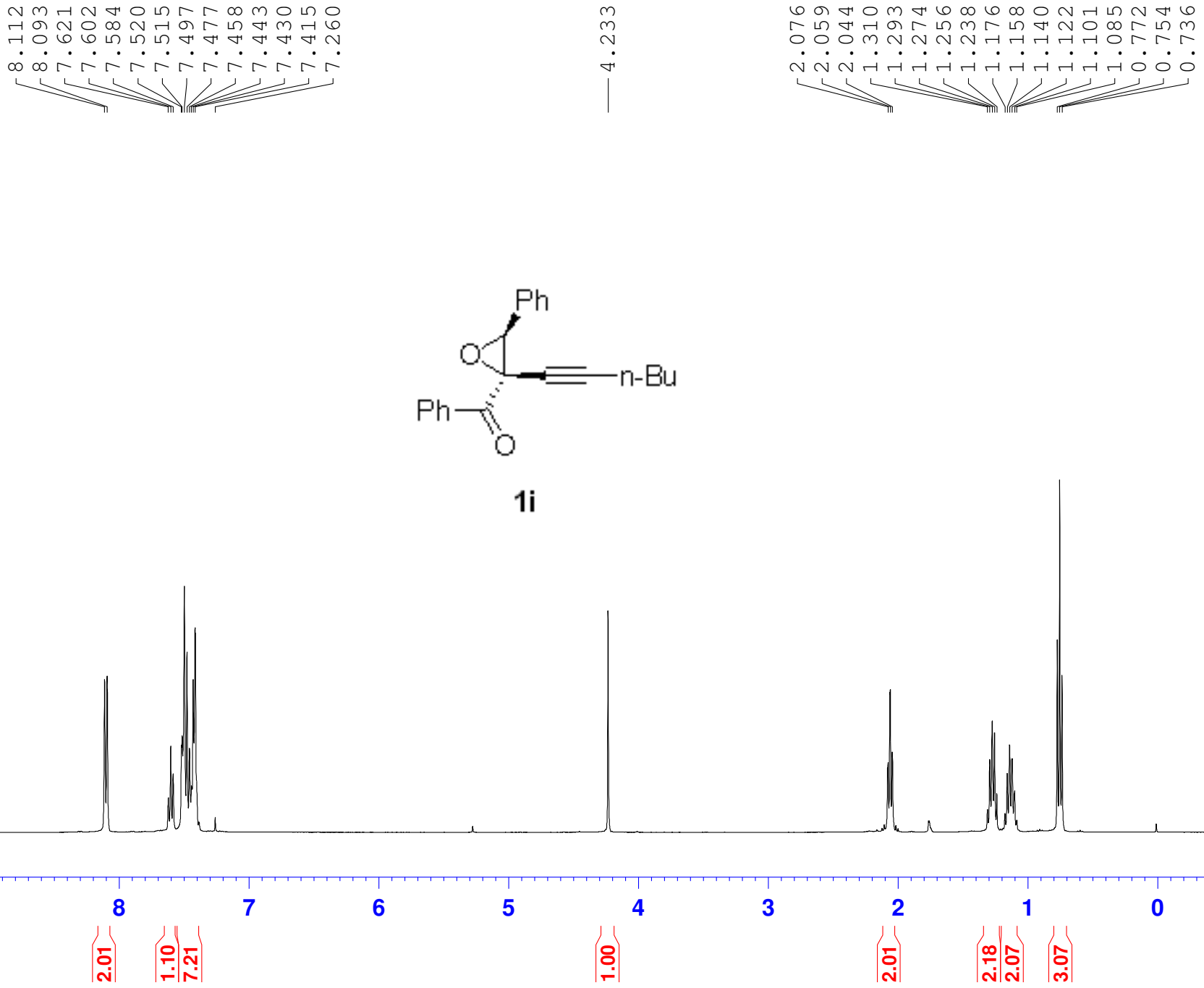
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.03
4.12
2.07
5.24
2.21
2.10
1.00


190.81
133.90
133.85
132.92
132.85
132.59
131.08
129.53
129.44
129.00
128.45
128.17
127.97
126.98
126.68
126.25
125.46
124.72
118.53
88.04
86.64
77.32
77.00
76.68
64.55
61.42

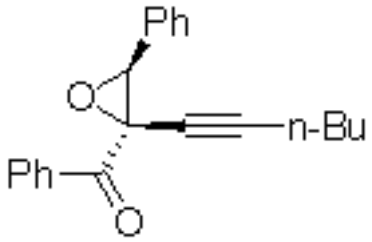

Ph
O
Ph
O

1h

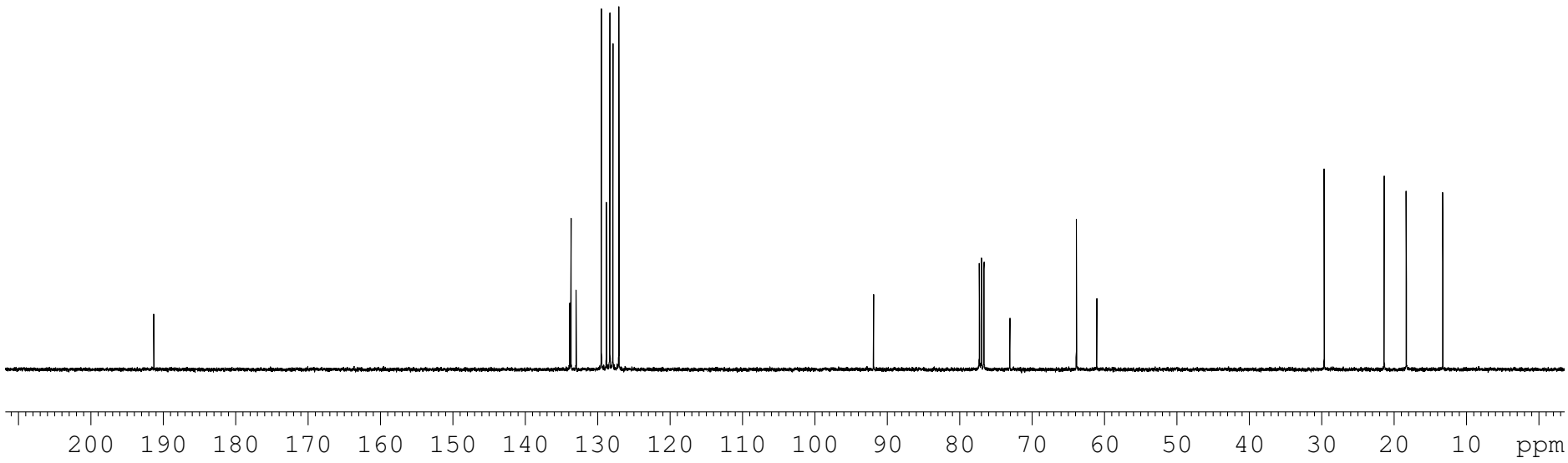

200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

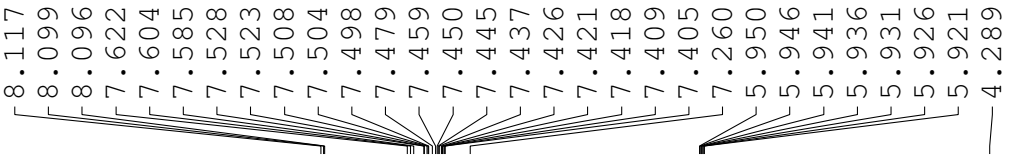

8.112
8.093
7.621
7.602
7.584
7.520
7.515
7.497
7.477
7.458
7.443
7.430
7.415
7.260
4.233
2.076
2.059
2.044
1.310
1.293
1.274
1.256
1.238
1.176
1.158
1.140
1.122
1.101
1.085
0.772
0.754
0.736
Ph
O
n-Bu
Ph
O
1i
10
9
8
7
6
5
4
3
2
1
0
ppm
2.01
1.10
7.21
1.00
2.01
2.18
2.07
3.07

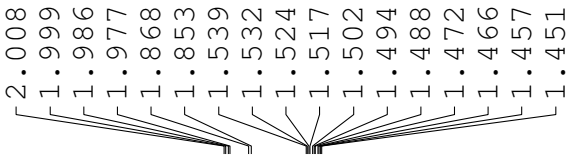
191.35
133.92
133.72
133.02
129.52
128.82
128.36
127.94
127.10
91.92
77.32
77.00
76.68
73.11
63.91
61.09
29.70
21.40
18.35
13.31

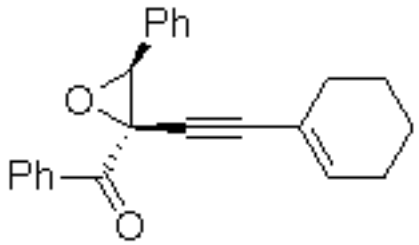


1i

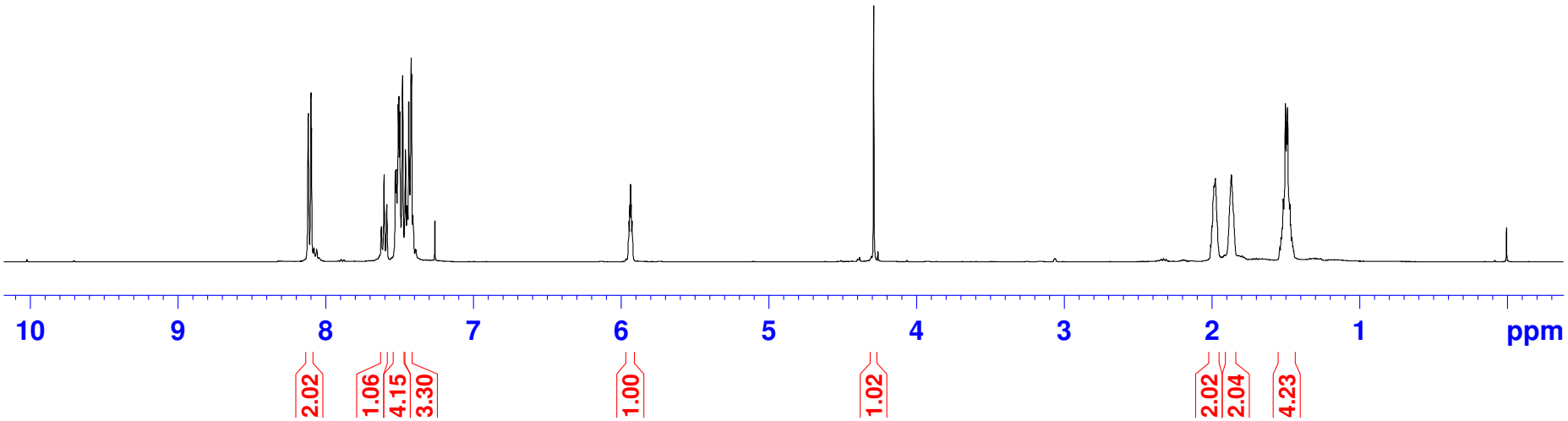



8.117
8.099
8.096
7.622
7.604
7.585
7.528
7.523
7.508
7.504
7.498
7.479
7.459
7.450
7.445
7.437
7.426
7.421
7.418
7.409
7.405
7.260
5.950
5.946
5.941
5.936
5.931
5.926
5.921
4.289

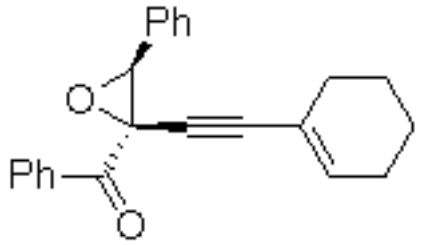

2.008
1.999
1.986
1.977
1.868
1.853
1.539
1.532
1.524
1.517
1.502
1.494
1.488
1.472
1.466
1.457
1.451


Ph
O
Ph
O

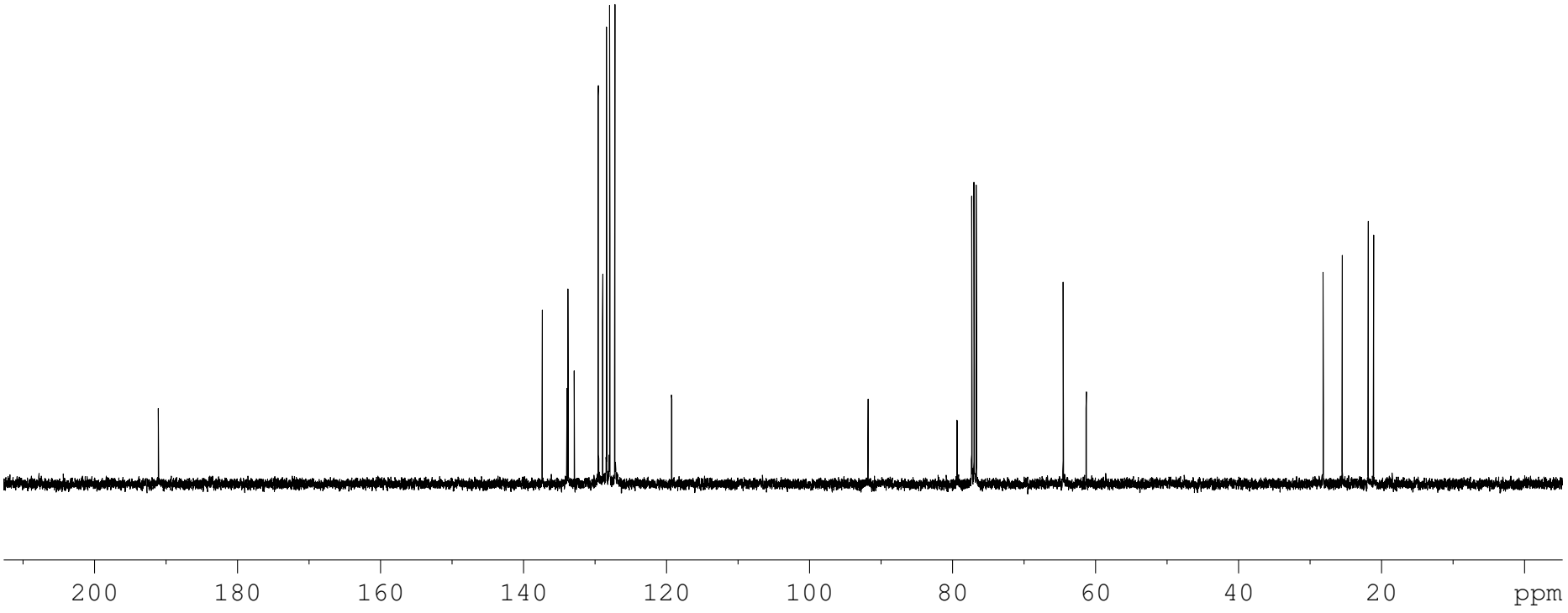
1j

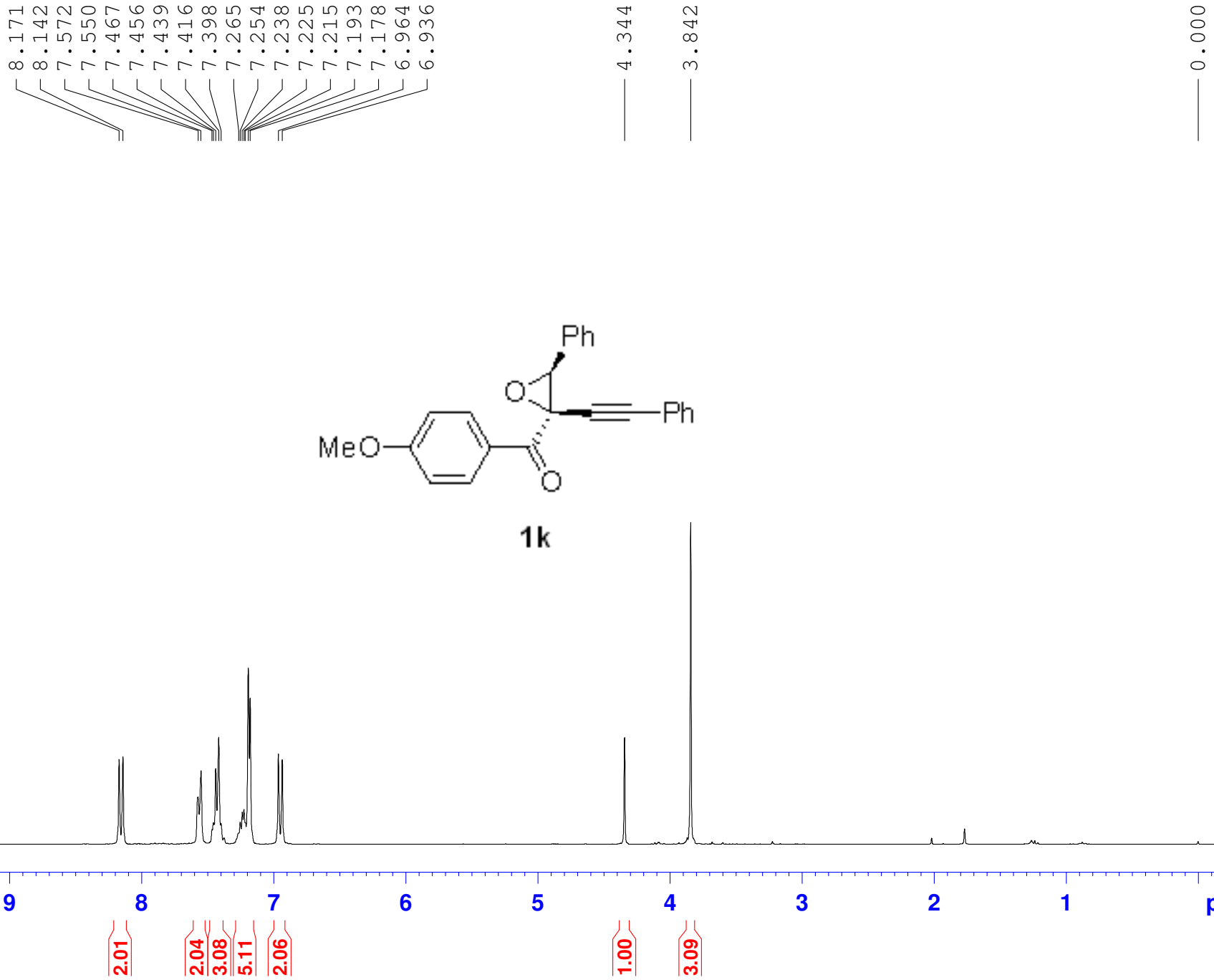

10
9
8
7
6
5
4
3
2
1
ppm
2.02
1.06
4.15
3.30
1.00
1.02
2.02
2.04
4.23

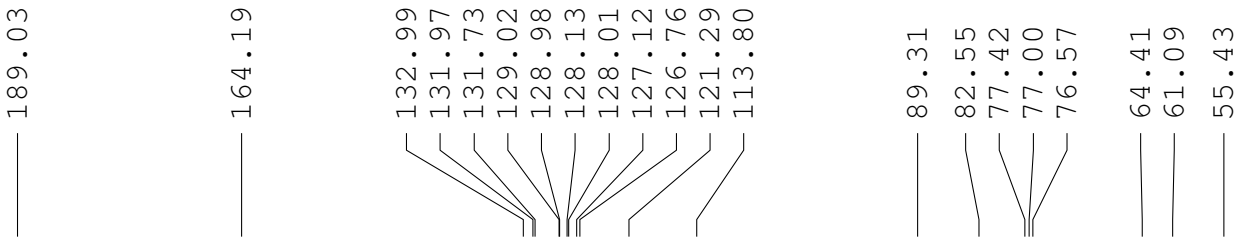

191.10
137.40
133.95
133.80
132.94
129.58
128.95
128.40
127.96
127.25
119.30
91.82
79.37
77.32
77.00
76.68
64.53
61.30
28.16
25.49
21.85
21.11

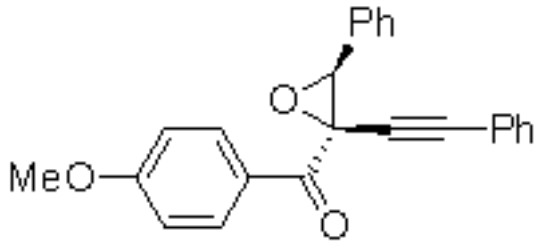

Ph
O
Ph
O

1j

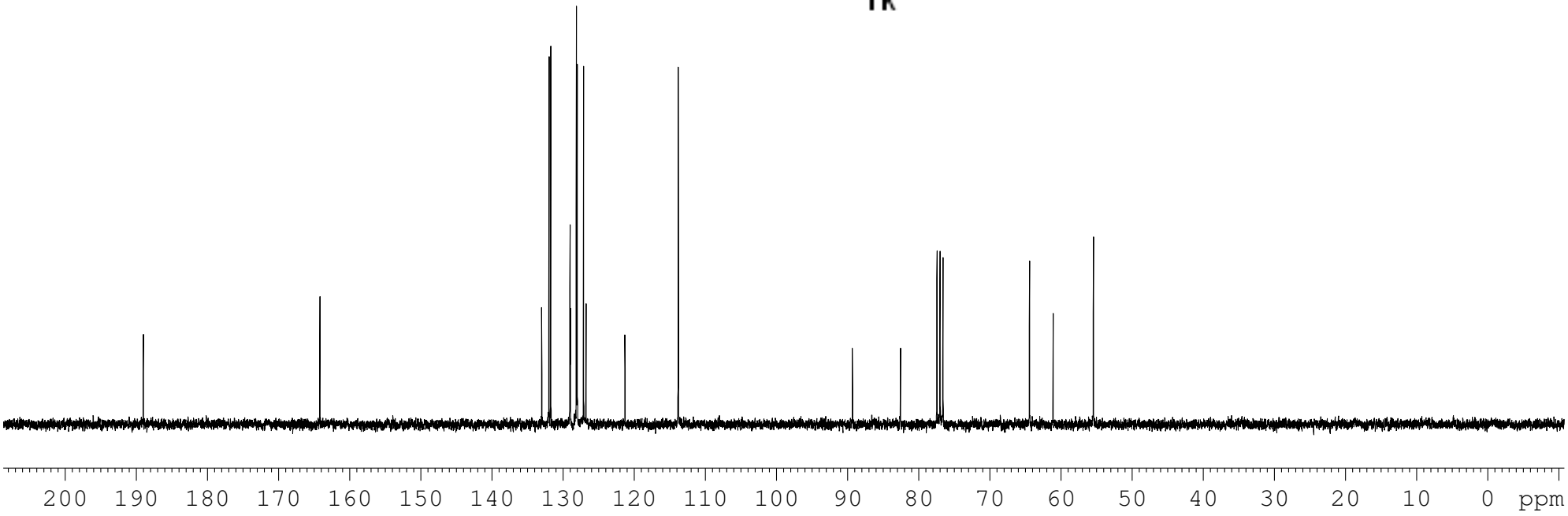

200
180
160
140
120
100
80
60
40
20
ppm

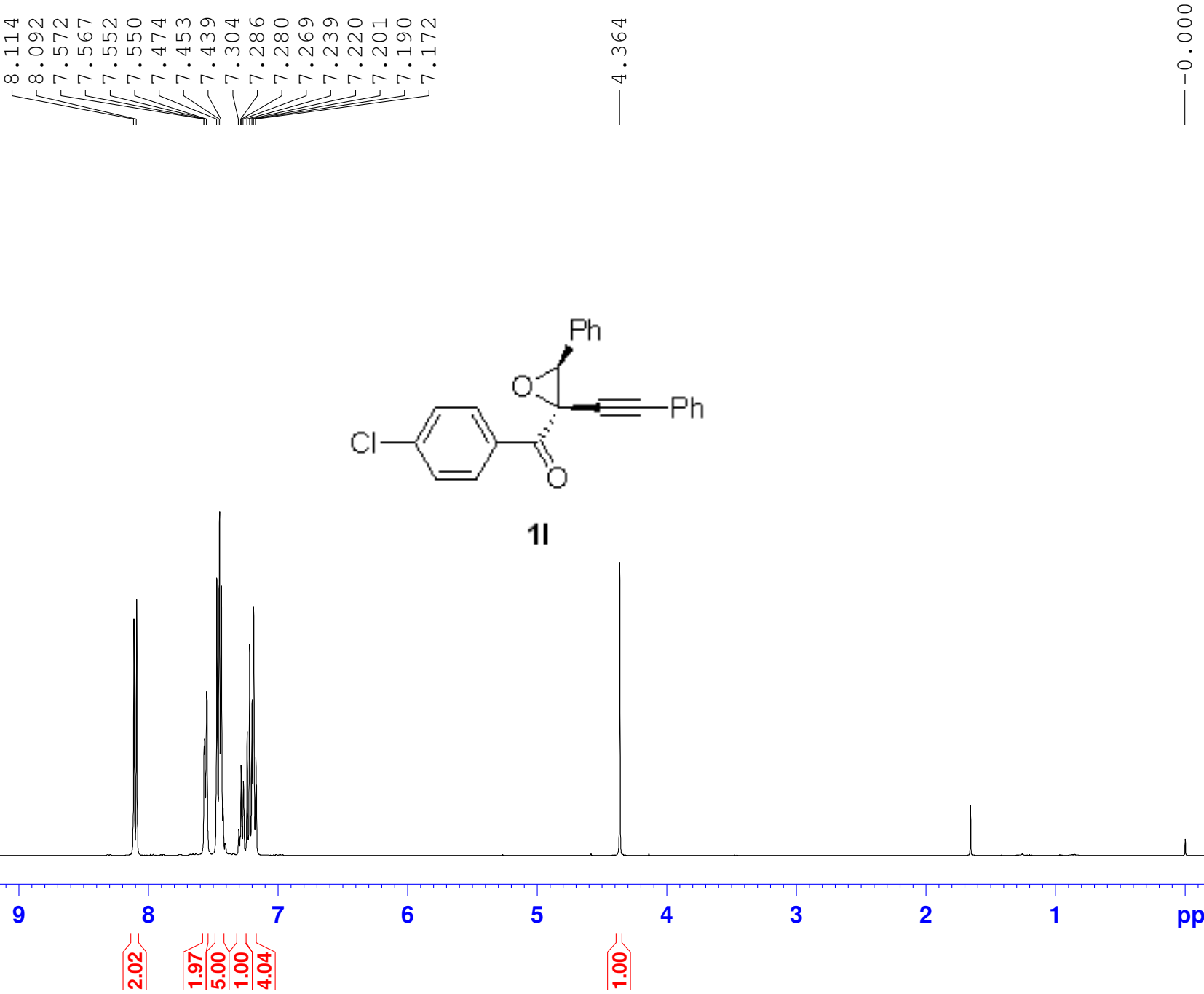

8.171
8.142
7.572
7.550
7.467
7.456
7.439
7.416
7.398
7.265
7.254
7.238
7.225
7.215
7.193
7.178
6.964
6.936
4.344
3.842
0.000
Ph
O
Ph
MeO
O
1k
10
9
8
7
6
5
4
3
2
1
ppm
2.01
2.04
3.08
5.11
2.06
1.00
3.09


189.03
164.19
132.99
131.97
131.73
129.02
128.98
128.13
128.01
127.12
126.76
121.29
113.80
89.31
82.55
77.42
77.00
76.57
64.41
61.09
55.43


Ph
O
Ph
MeO
O

1k


200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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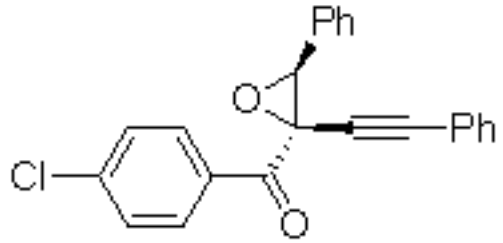
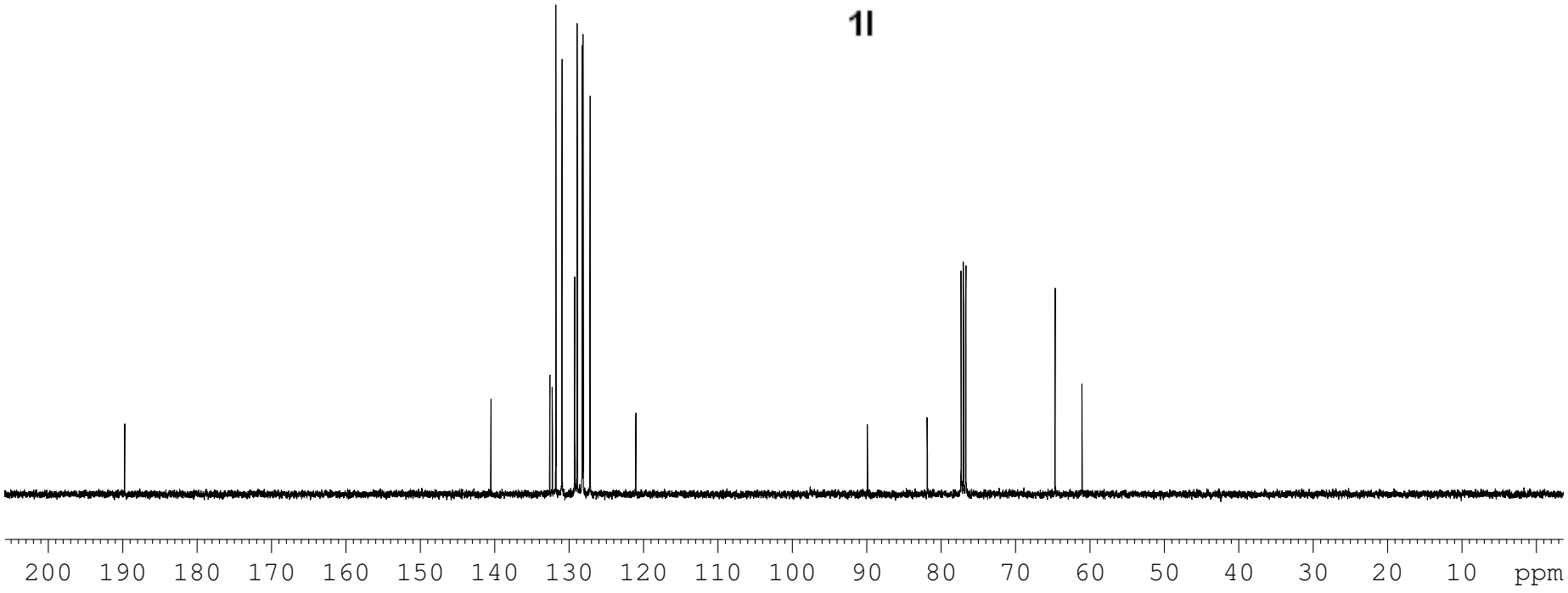
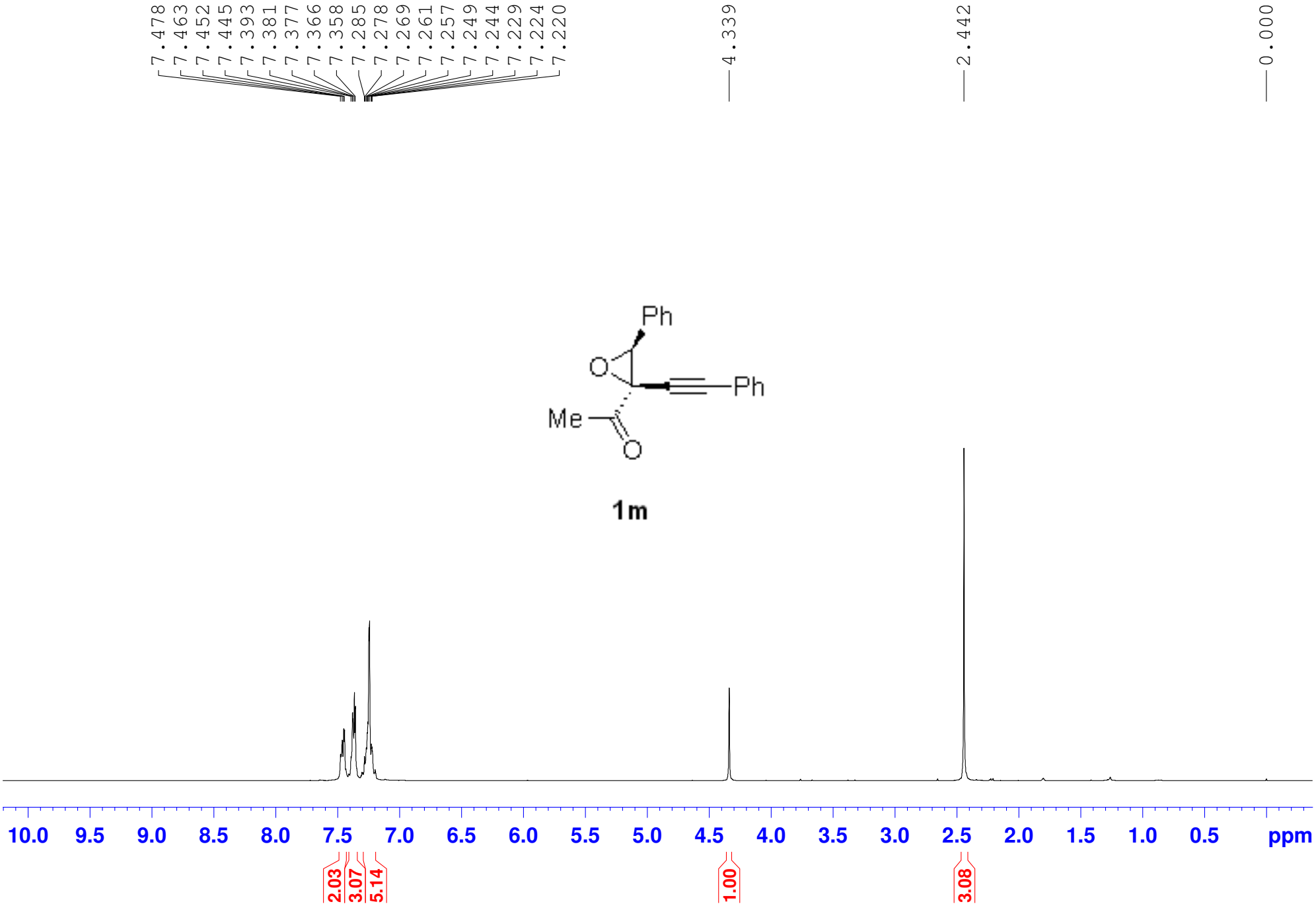
8.092
7.572
7.567
7.552
7.550
7.474
7.453
7.439
7.304
7.286
7.280
7.269
7.239
7.220
7.201
7.190
7.172
4.364
0.000
Ph
O
Ph
Cl
O
1l
10
9
8
7
6
5
4
3
2
1
ppm
2.02
1.97
5.00
1.00
4.04
1.00

189.72
140.50
132.60
132.25
131.78
130.96
129.26
129.22
128.90
128.24
128.12
127.19
121.03
89.91
81.86
77.32
77.00
76.68
64.69
61.06

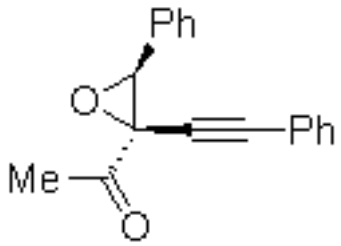


1l

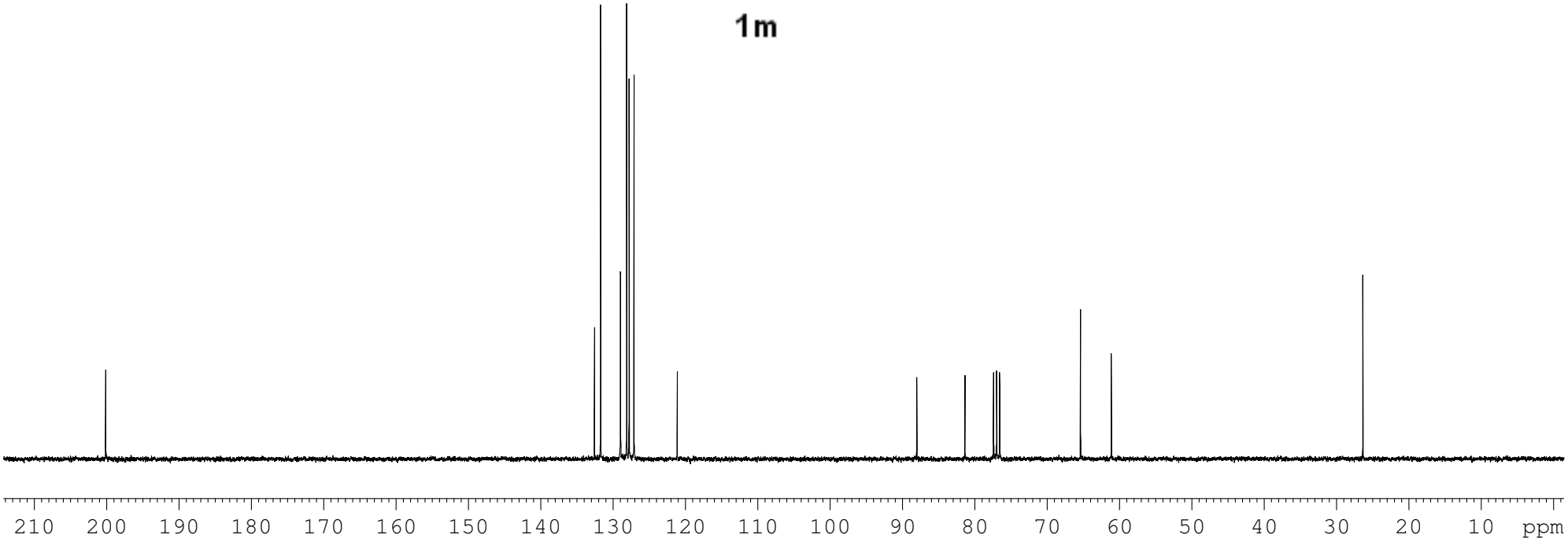



7.478
7.463
7.452
7.445
7.393
7.381
7.377
7.366
7.358
7.285
7.278
7.269
7.261
7.257
7.249
7.244
7.229
7.224
7.220
4.339
2.442
0.000
Ph
O
Ph
Me
O
1m
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.03
3.07
5.14
1.00
3.08

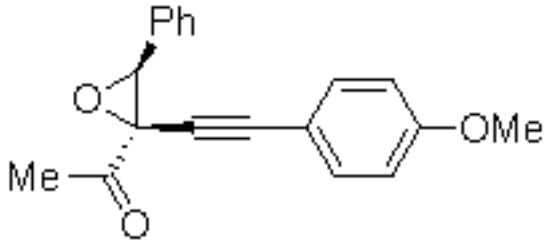
200.15
132.59
131.74
129.01
128.97
128.14
127.81
127.13
121.15
88.04
81.37
77.43
77.00
76.58
65.41
61.14
26.37



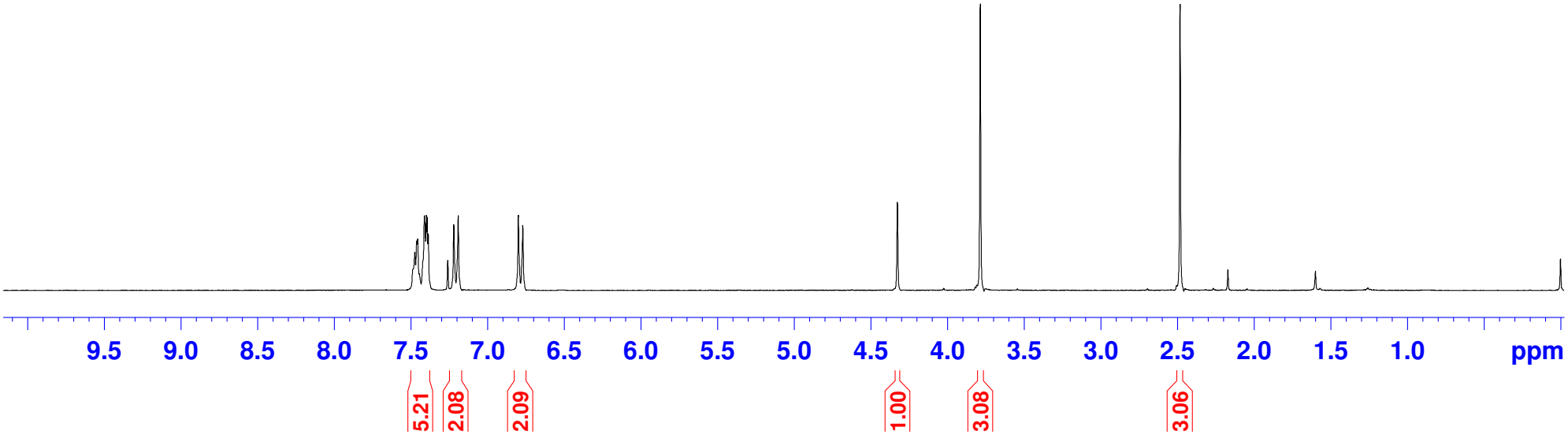
1m

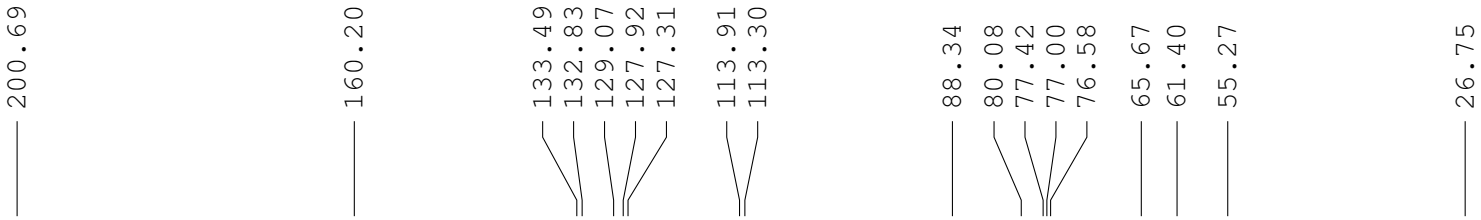


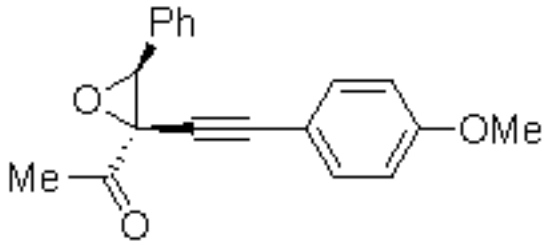

7.472
7.460
7.454
7.443
7.409
7.396
7.388
7.260
7.221
7.192
6.800
6.771
4.327
3.786
2.483


Ph
O
OMe
Me
O

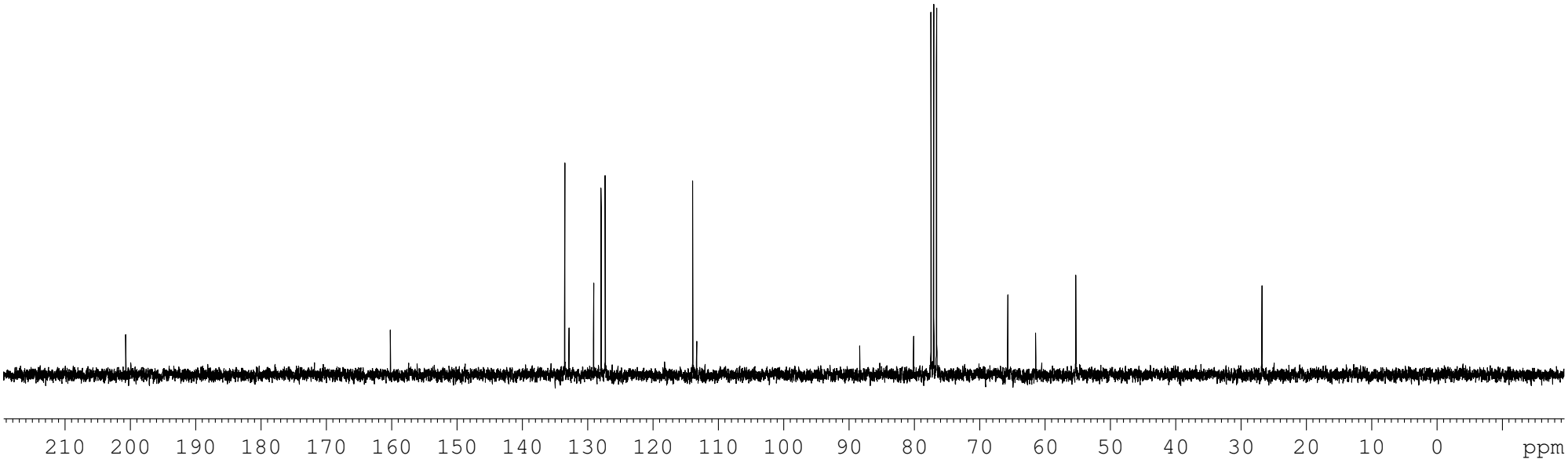
1n

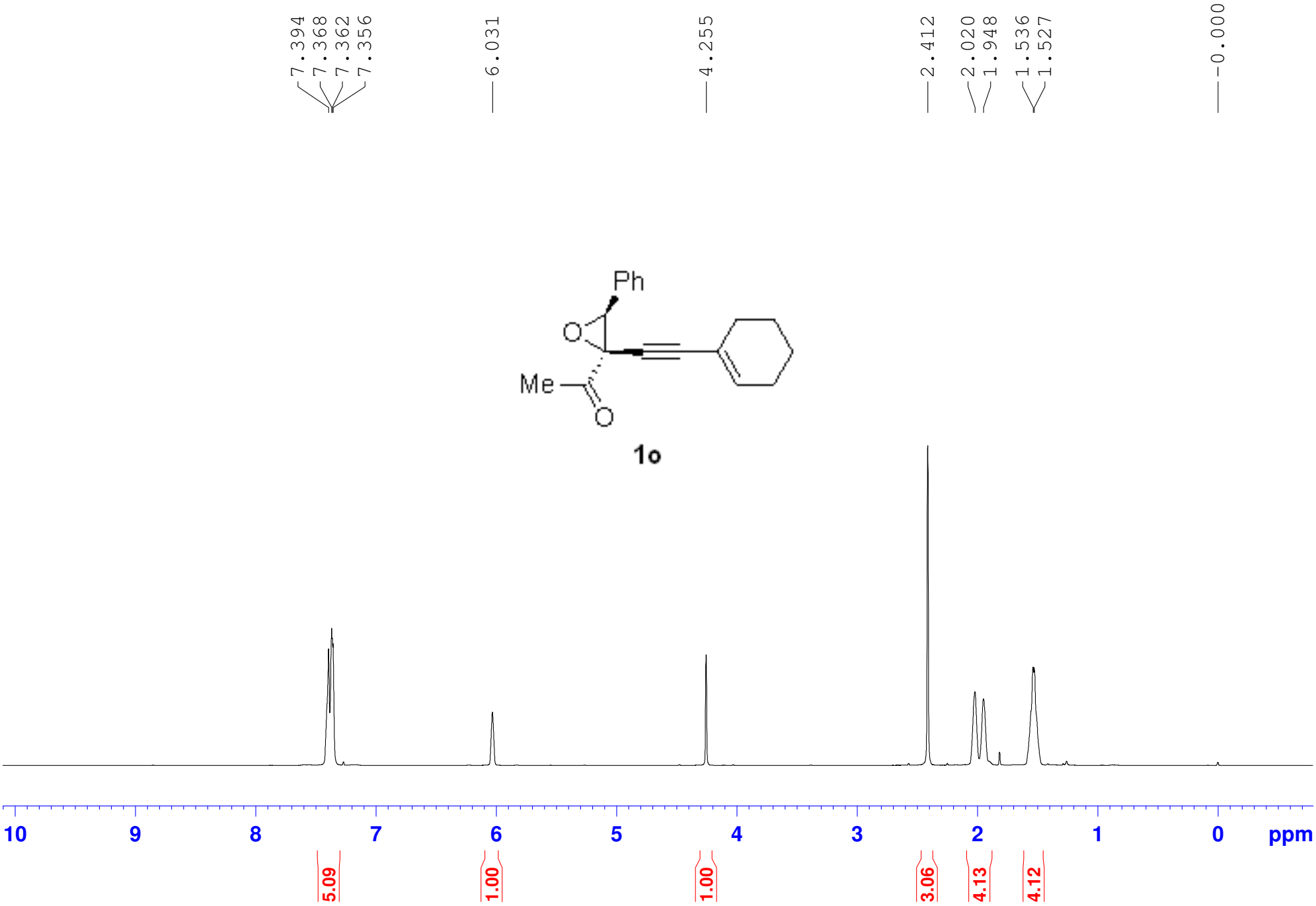

9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
5.21
2.08
2.09
1.00
3.08
3.06


200.69
160.20
133.49
132.83
129.07
127.92
127.31
113.91
113.30
88.34
80.08
77.42
77.00
76.58
65.67
61.40
55.27
26.75


Ph
O
OMe
Me
O

1n


210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.394
7.368
7.362
7.356
6.031
4.255
2.412
2.020
1.948
1.536
1.527
0.000
Ph
O
Me
O
1o
10
9
8
7
6
5
4
3
2
1
0
ppm
5.09
1.00
1.00
3.06
4.13
4.12

200.59
137.33
132.72
128.82
127.69
127.18
119.19
90.09
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77.00
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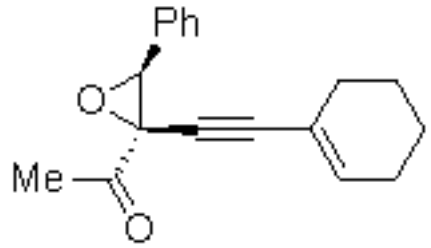
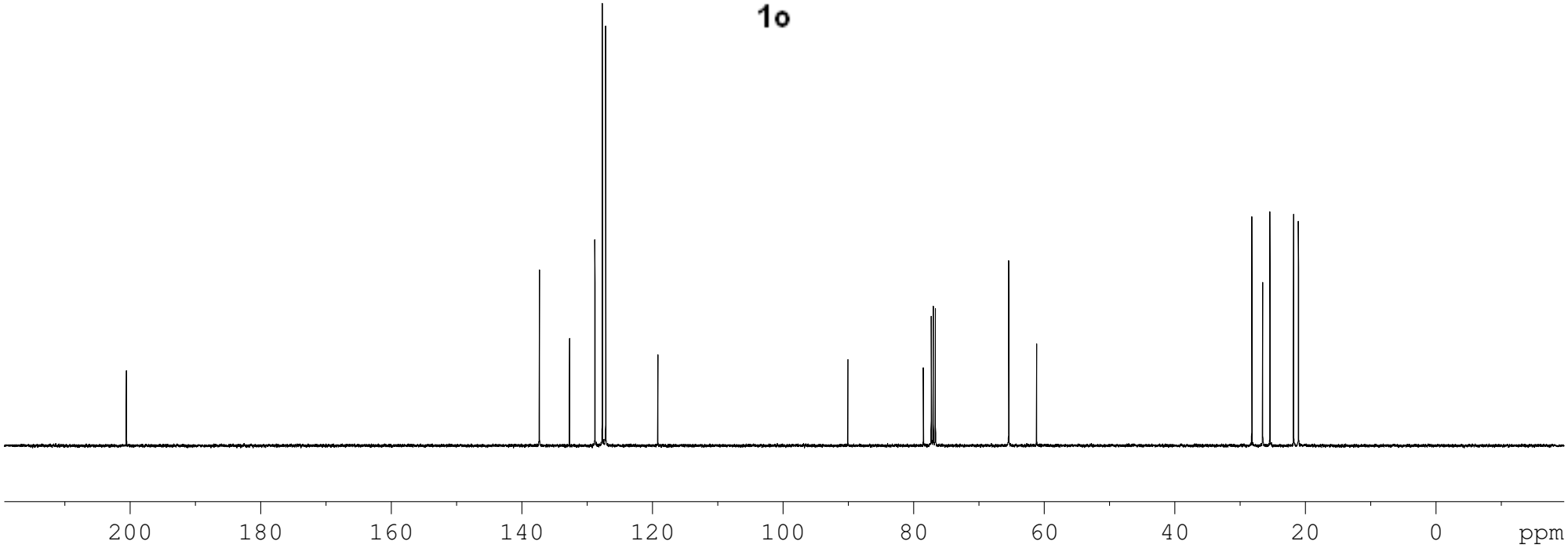
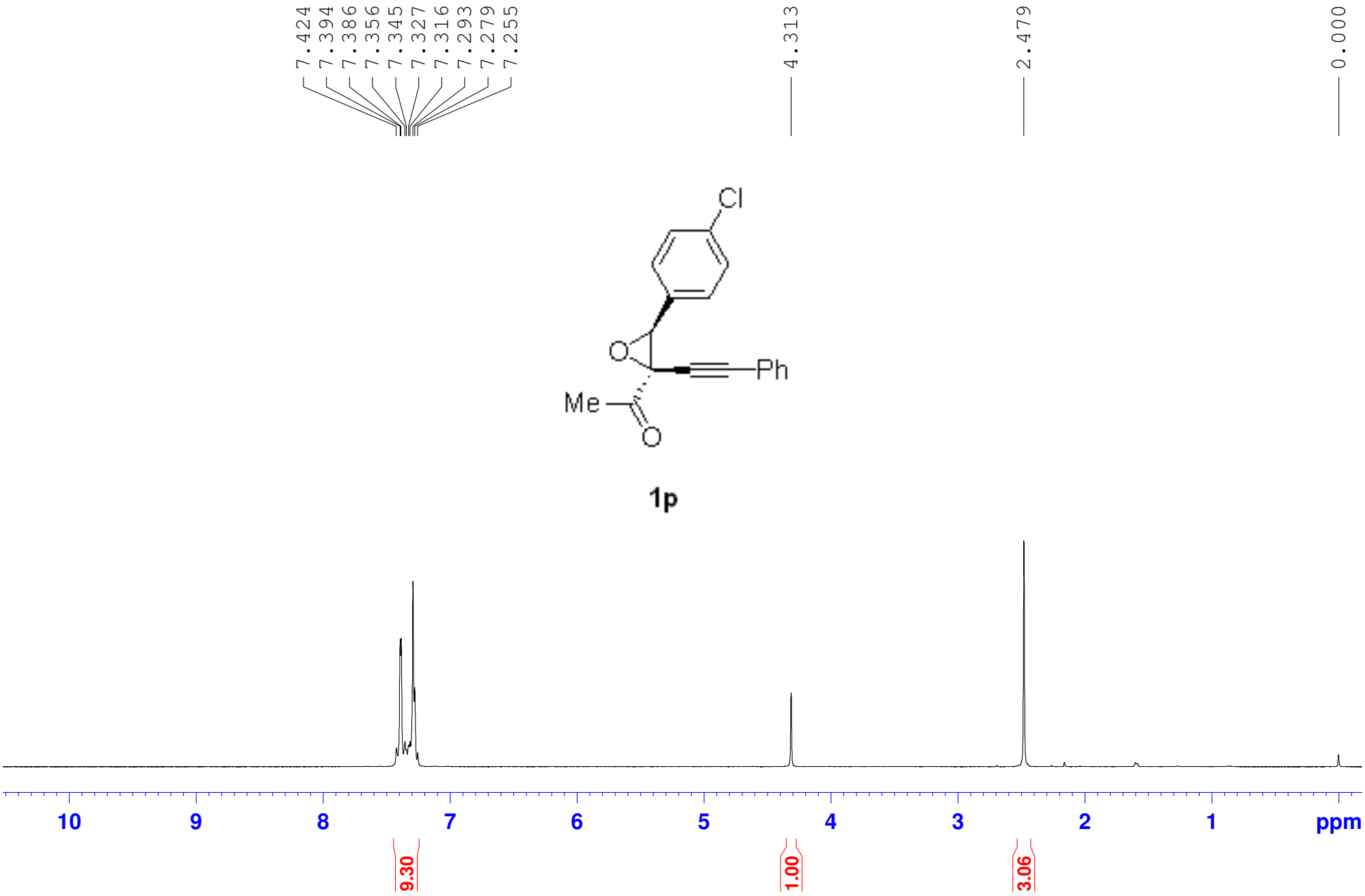
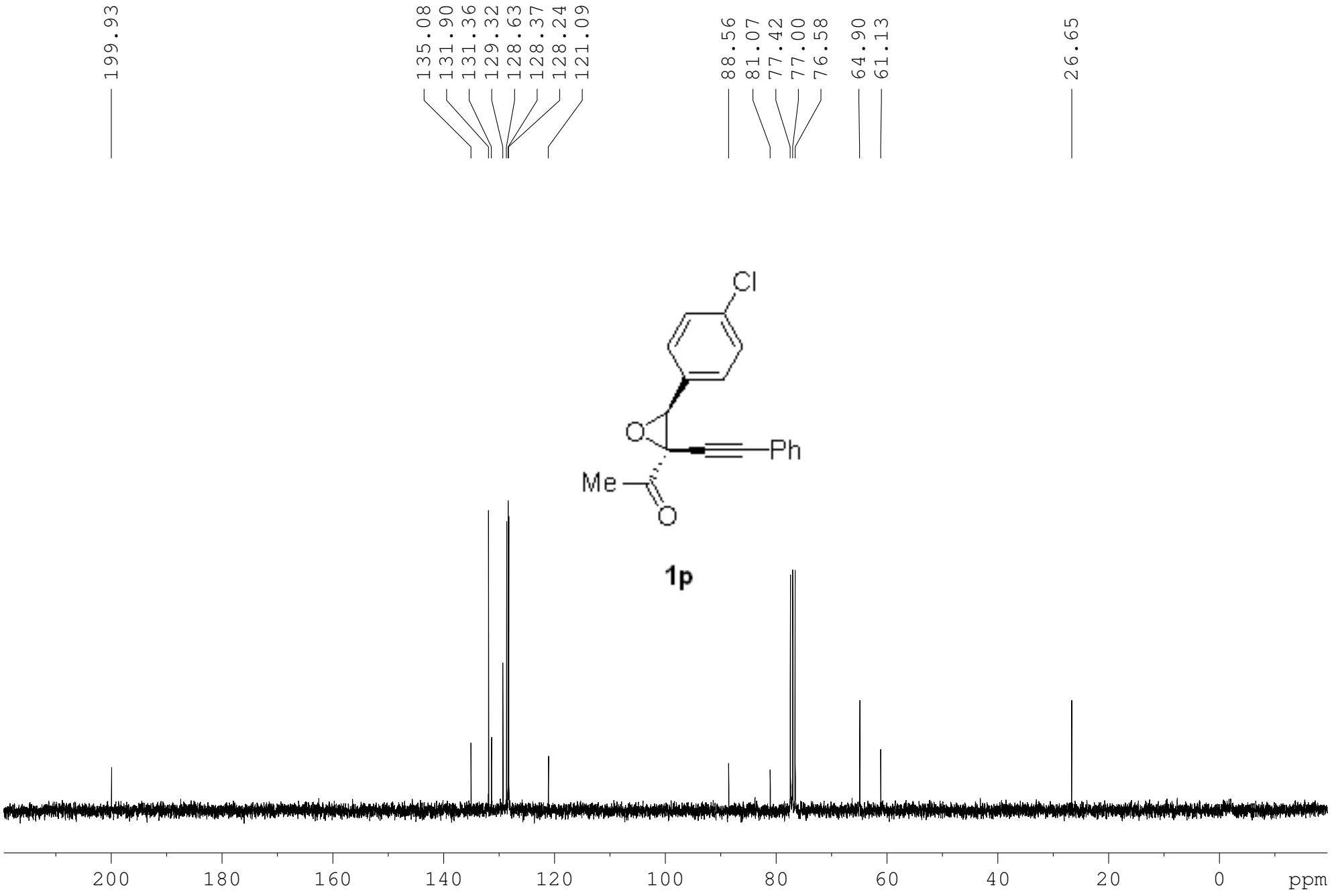
61.16
28.21
26.55
25.43
21.82
21.07



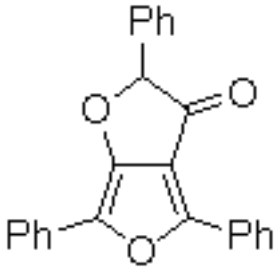
1o



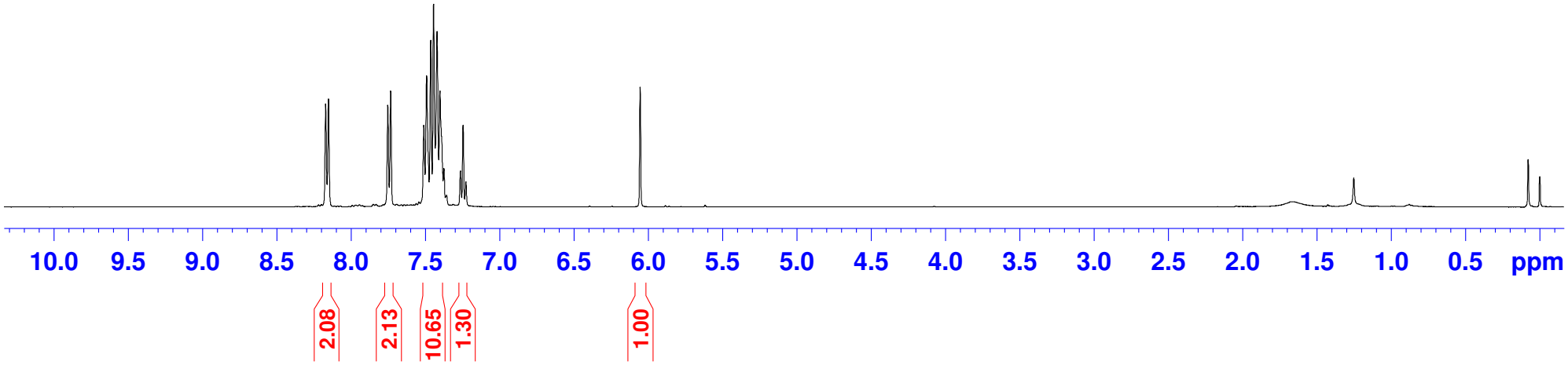

7.424
7.394
7.386
7.356
7.345
7.327
7.316
7.293
7.279
7.255
4.313
2.479
0.000
Cl
O
Ph
Me
O
1p
10
9
8
7
6
5
4
3
2
1
ppm
9.30
1.00
3.06


199.93
135.08
131.90
131.36
129.32
128.63
128.37
128.24
121.09
88.56
81.07
77.42
77.00
76.58
64.90
61.13
26.65
Cl
O
Ph
Me
O
1p
200
180
160
140
120
100
80
60
40
20
0
ppm


8.172
8.152
7.753
7.734
7.511
7.492
7.464
7.445
7.422
7.402
7.375
7.264
7.246
7.227
6.055
0.000


Ph
O
O
Ph
Ph
O

3a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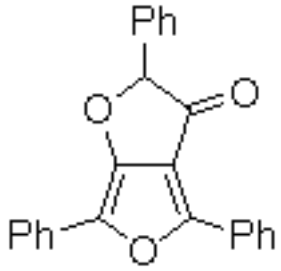
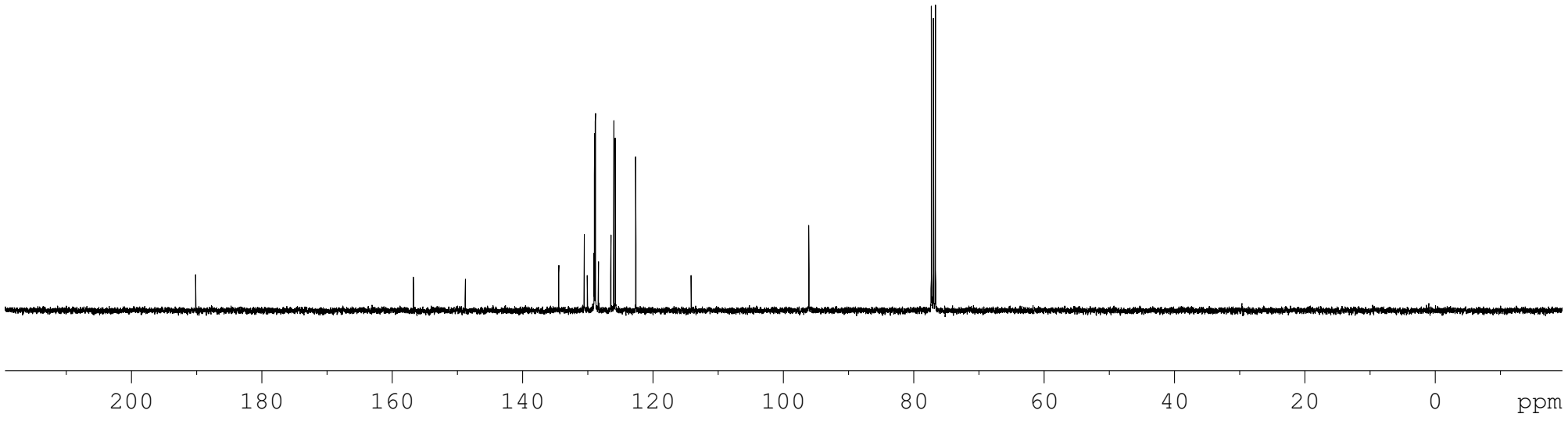
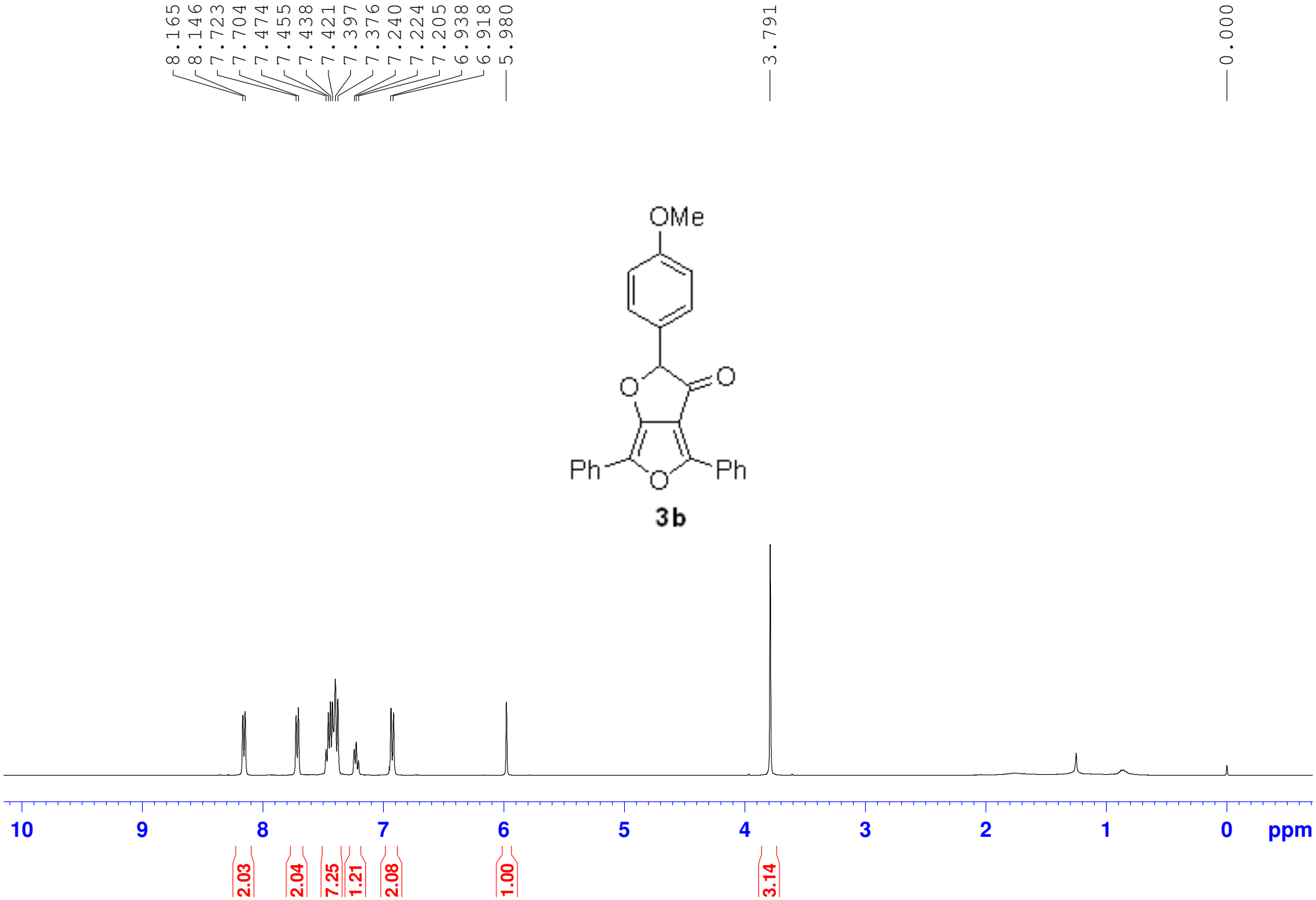
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.08
2.13
10.65
1.30
1.00

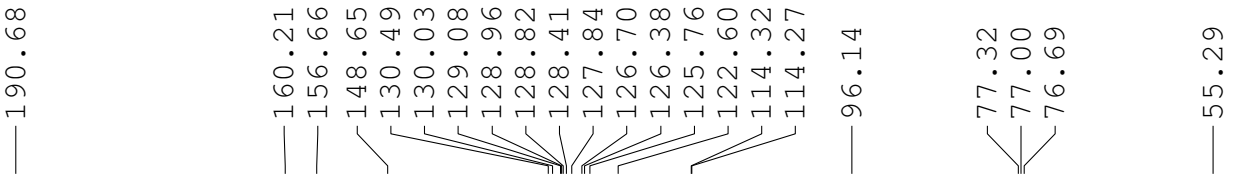
190.18
156.76
148.82
134.49
130.57
130.12
129.08
129.02
128.99
128.88
128.85
128.40
126.48
126.03
125.80
122.66
114.17
96.11
77.32
77.00
76.68



3a




8.165
8.146
7.723
7.704
7.474
7.455
7.438
7.421
7.397
7.376
7.240
7.224
7.205
6.938
6.918
5.980
3.791
0.000
OMe
O
O
Ph
Ph
O
3b
10
9
8
7
6
5
4
3
2
1
0
ppm
2.03
2.04
7.25
1.21
2.08
1.00
3.14


190.68
160.21
156.66
148.65
130.49
130.03
129.08
128.96
128.82
128.41
127.84
126.70
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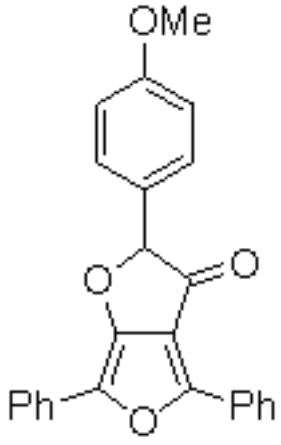
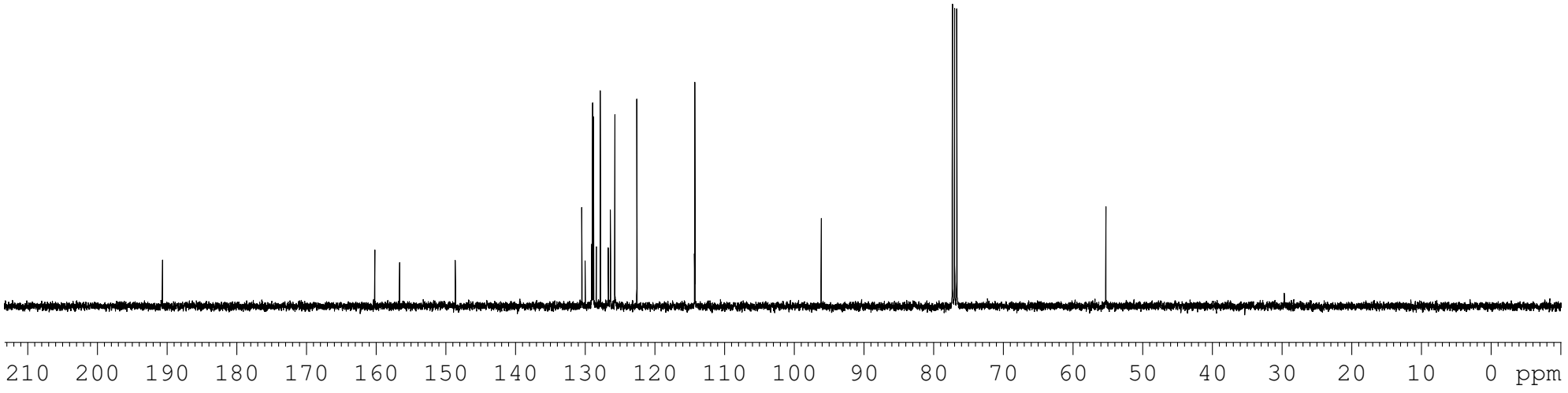
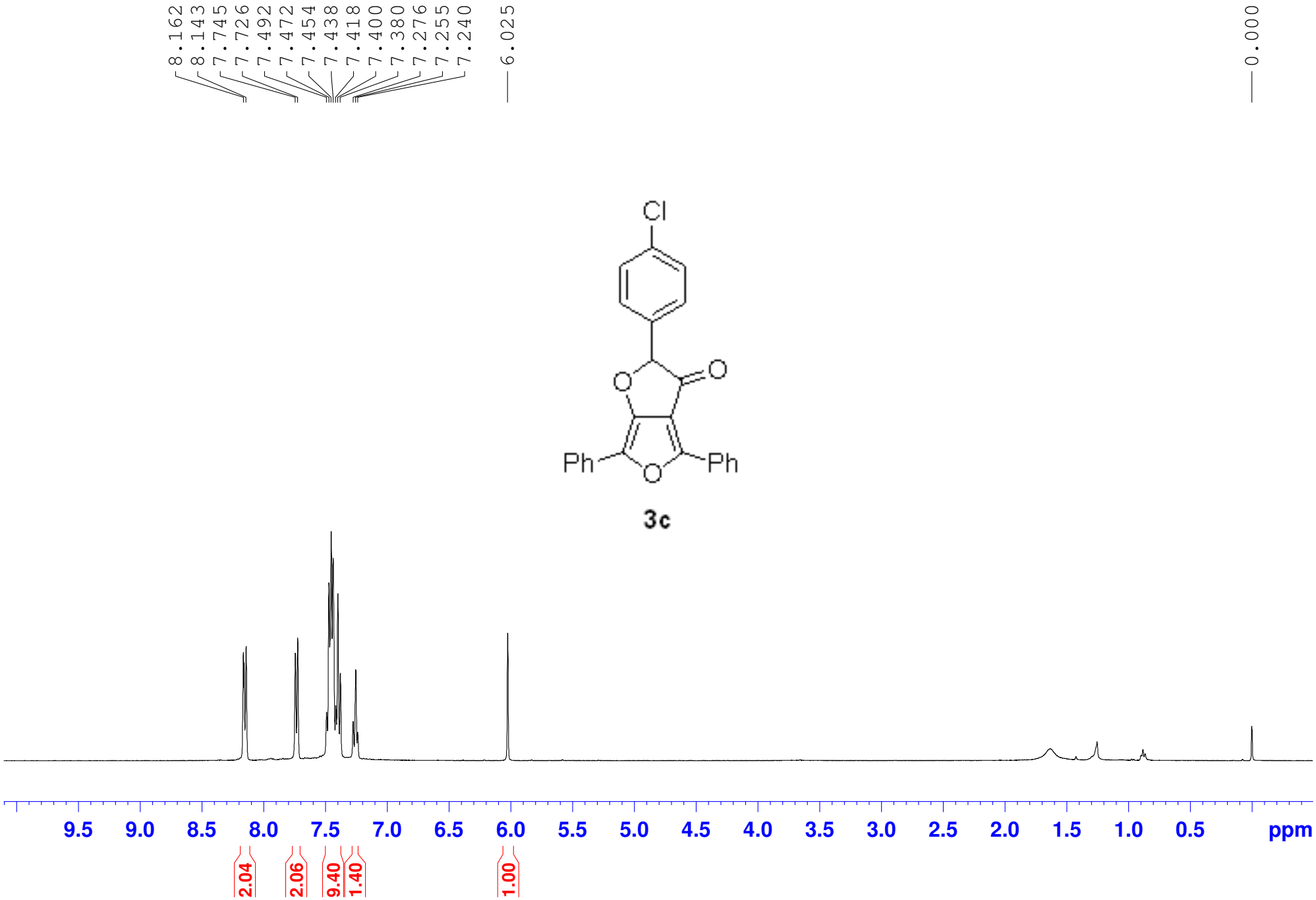
114.27
96.14
77.32
77.00
76.69
55.29

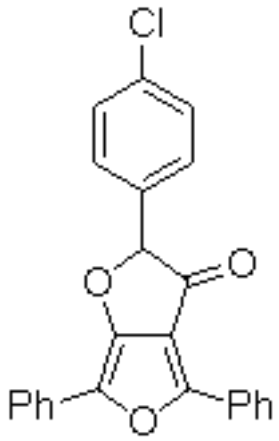

OMe
O
O
Ph
O
Ph

3b

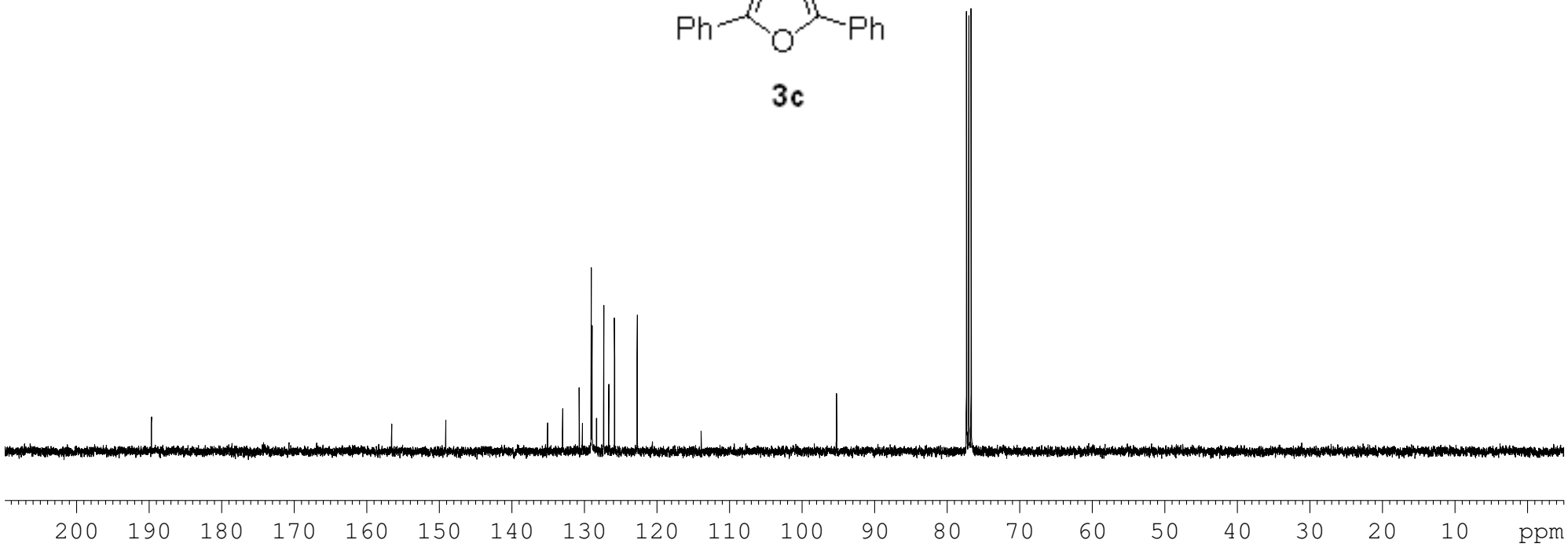

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


8.162
8.143
7.745
7.726
7.492
7.472
7.454
7.438
7.418
7.400
7.380
7.276
7.255
7.240
6.025
0.000
Cl
O
O
Ph
O
Ph
3c
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.04
2.06
9.40
1.40
1.00

189.62
156.51
149.03
135.00
132.94
130.71
130.26
129.05
128.96
128.92
128.32
127.32
126.63
125.83
122.71
113.89
95.24
77.32
77.00
76.69

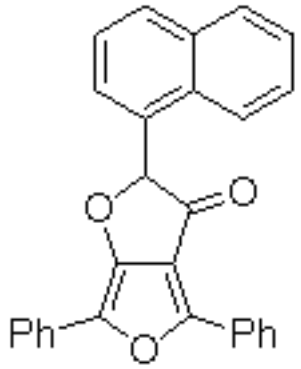


3c

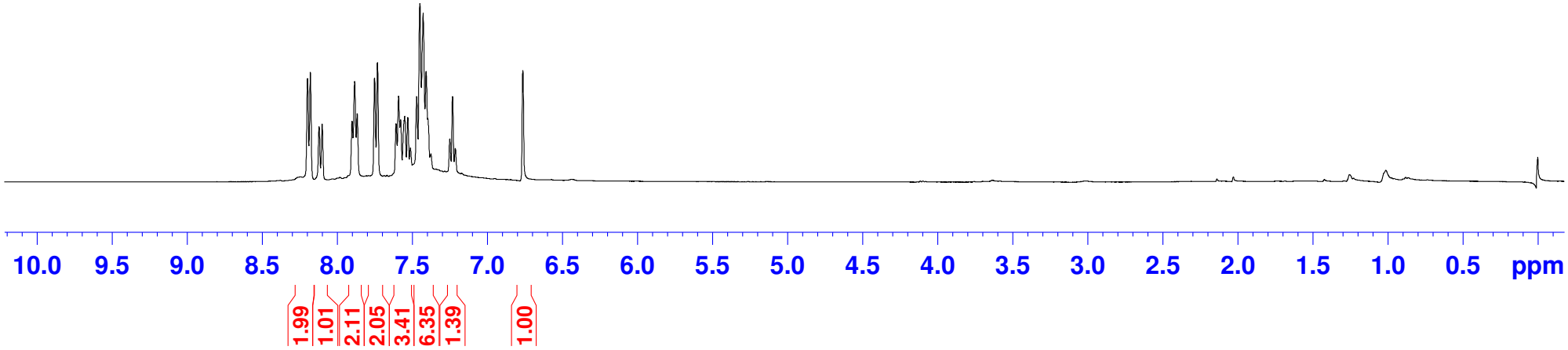


8.198
8.179
8.121
8.100
7.901
7.884
7.867
7.751
7.732
7.608
7.591
7.577
7.554
7.551
7.530
7.512
7.470
7.449
7.427
7.407
7.396
7.375
7.249
7.231
7.213
6.763

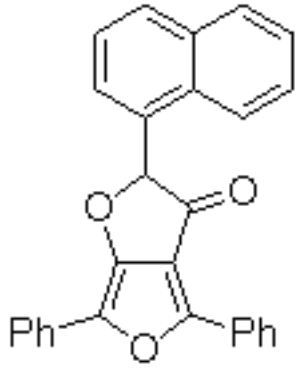
0.000



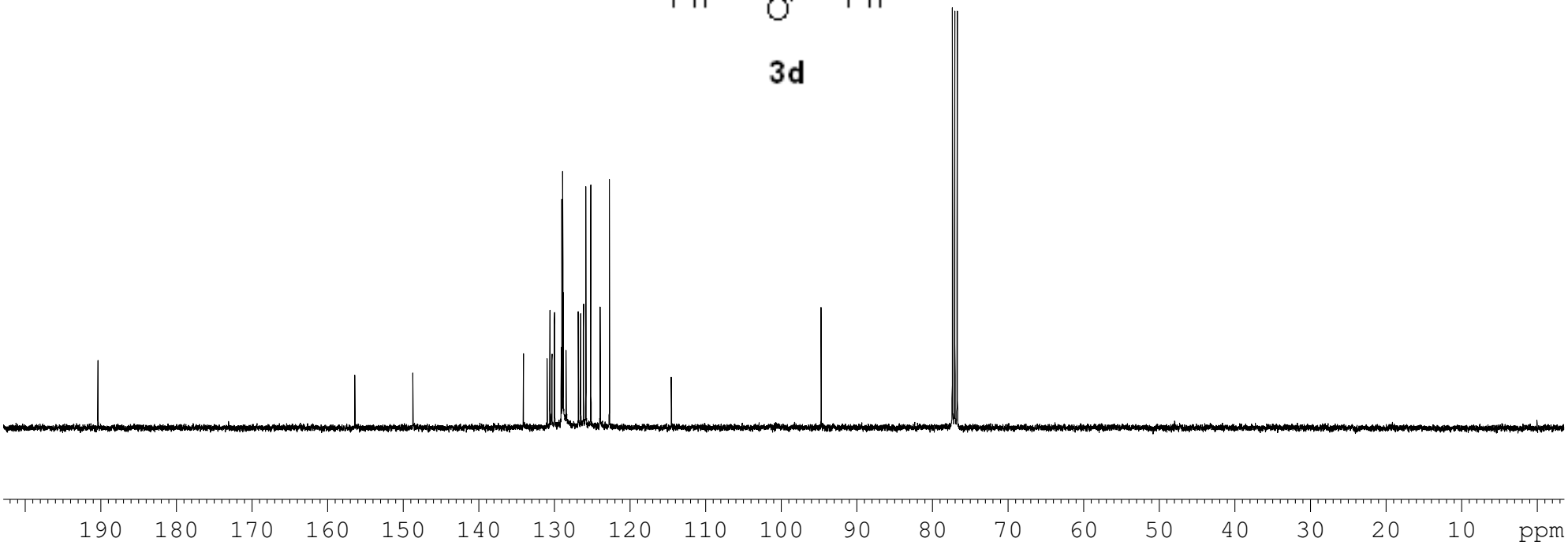
3d

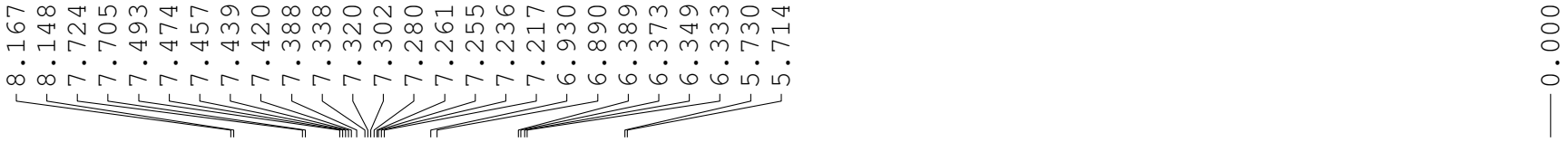


190.31
156.39
148.70
134.07
130.95
130.56
130.36
130.31
129.97
129.08
128.99
128.87
128.79
128.44
126.81
126.50
126.13
125.83
125.16
123.92
122.70
114.51
94.69
77.32
77.00
76.69



3d

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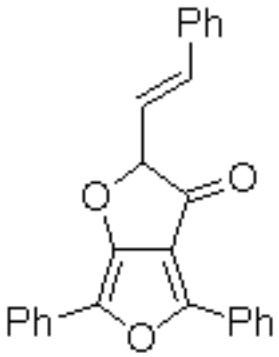
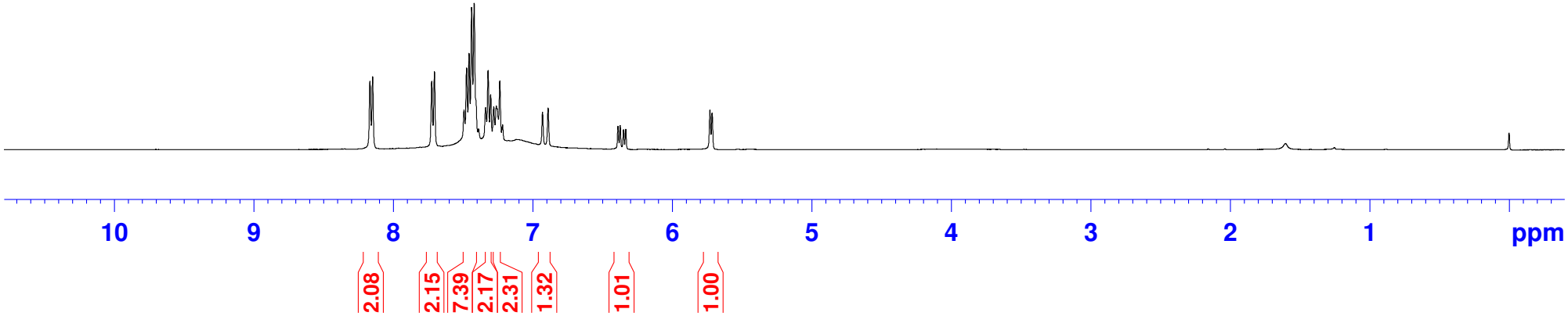
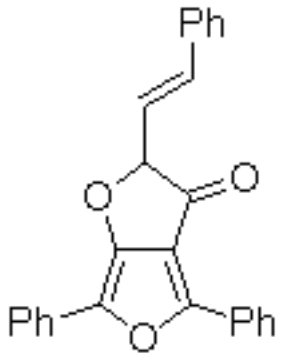
8.148
7.724
7.705
7.493
7.474
7.457
7.439
7.420
7.388
7.338
7.320
7.302
7.280
7.261
7.255
7.236
7.217
6.930
6.890
6.389
6.373
6.349
6.333
5.730
5.714
0.000


Ph
O
O
Ph
Ph
O

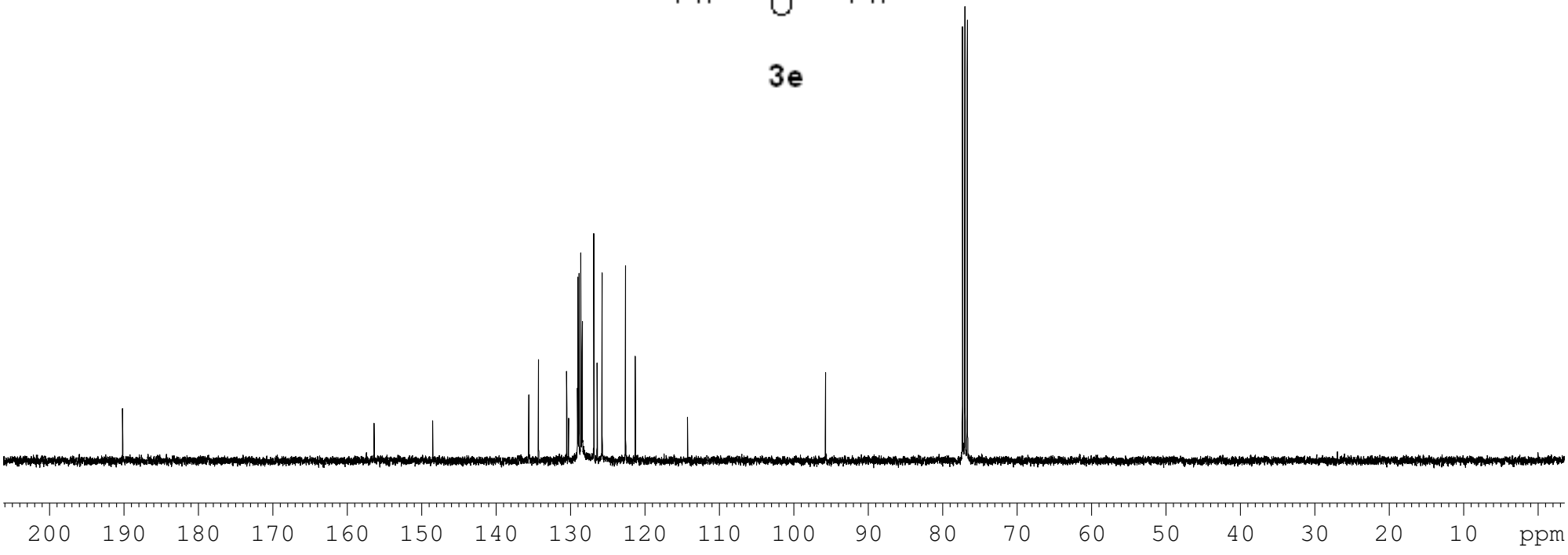
3e


10
9
8
7
6
5
4
3
2
1
ppm
2.08
2.15
7.39
2.17
2.31
1.32
1.01
1.00

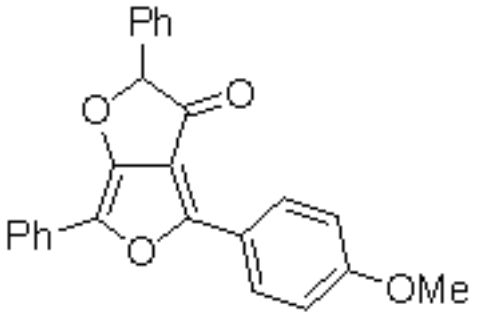
190.19
156.39
148.53
135.60
134.31
130.51
130.26
129.07
128.99
128.84
128.61
128.42
126.86
126.43
125.76
122.61
121.31
114.27
95.73
77.32
77.00
76.68



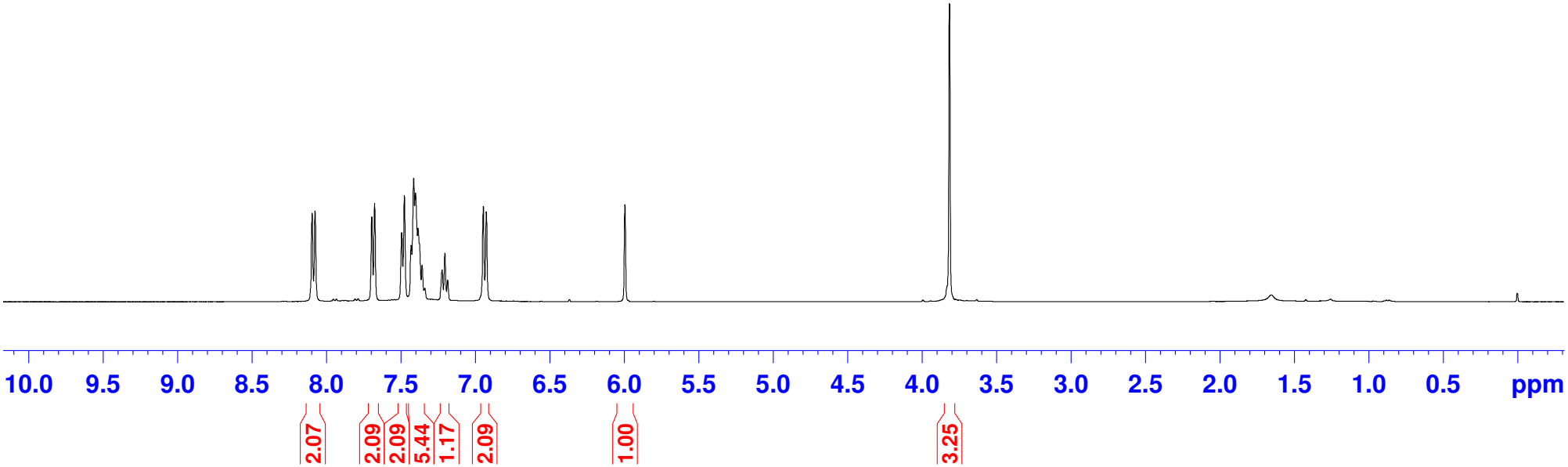
3e



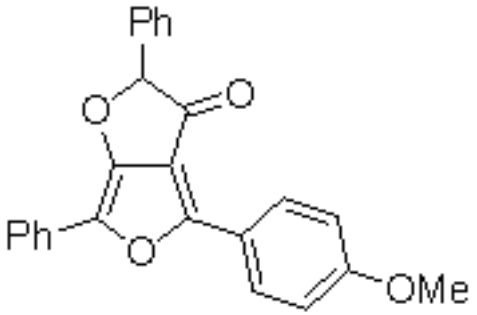

8.093
8.074
7.693
7.674
7.492
7.474
7.429
7.411
7.399
7.383
7.355
7.336
7.221
7.202
7.183
6.943
6.924
5.992
3.813
0.000


Ph
O
O
Ph
O
OMe

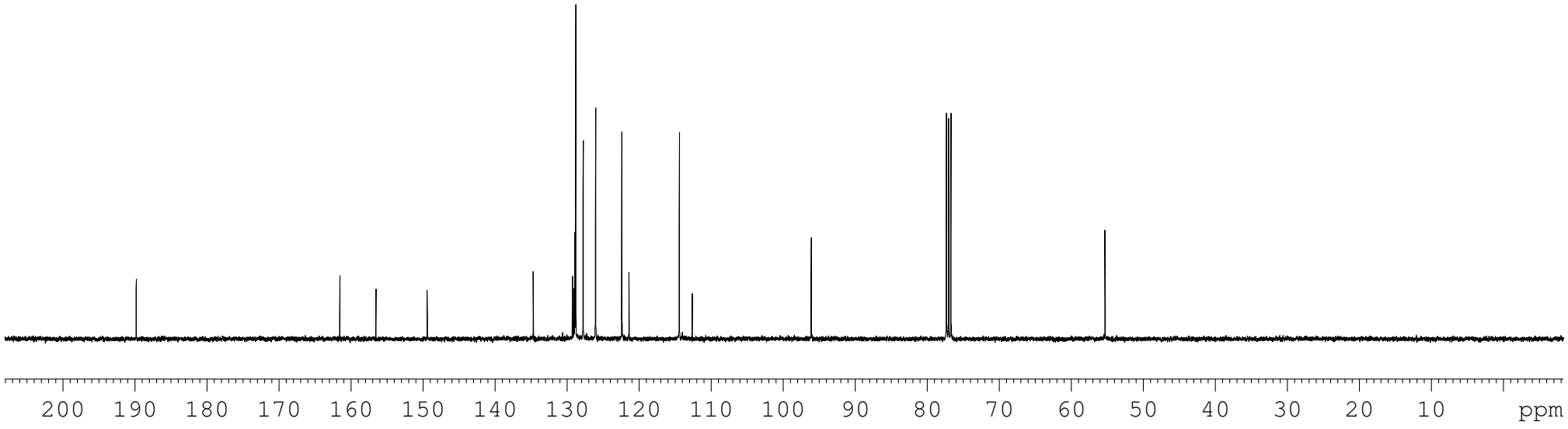
3f

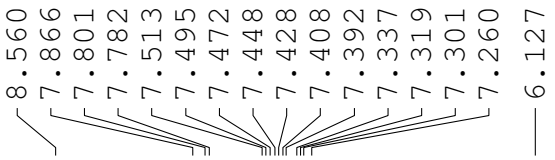

10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.07
2.09
2.09
5.44
1.17
2.09
1.00
3.25

189.81
161.54
156.53
149.41
134.69
129.24
129.07
128.90
128.78
127.75
126.05
126.01
122.38
121.39
114.40
112.59
96.09
77.32
77.00
76.69
55.35



3f

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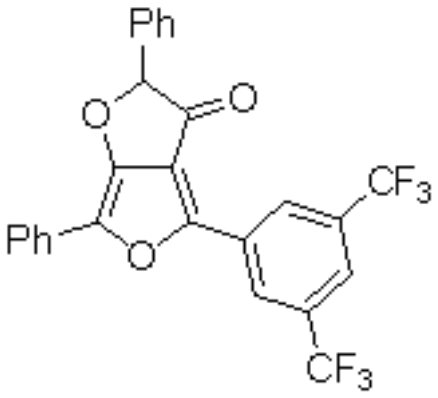
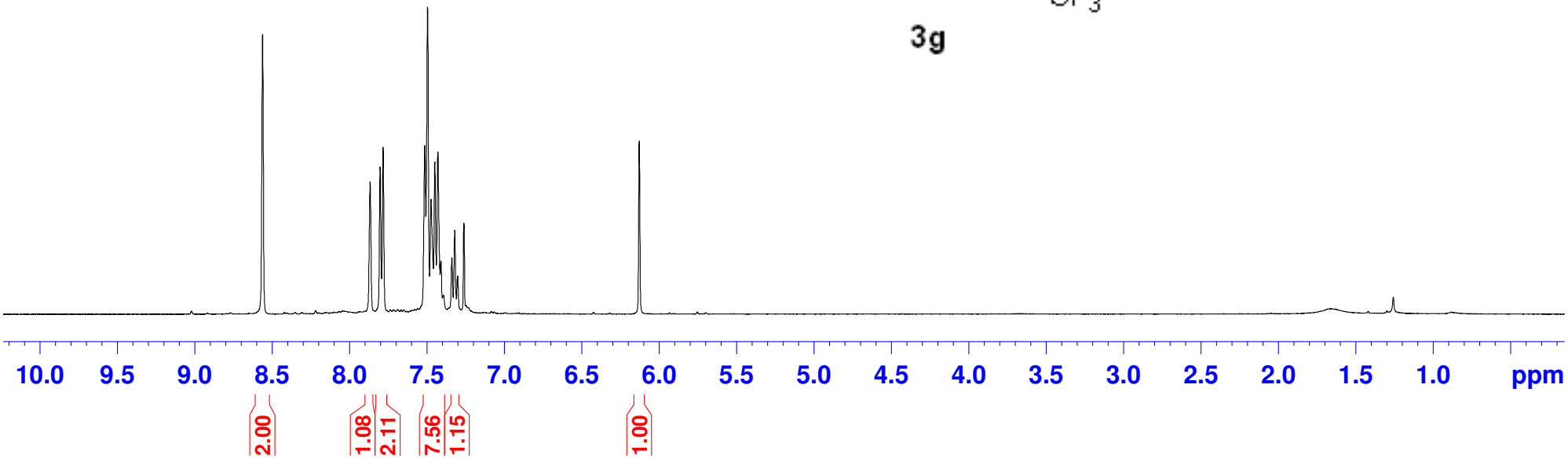
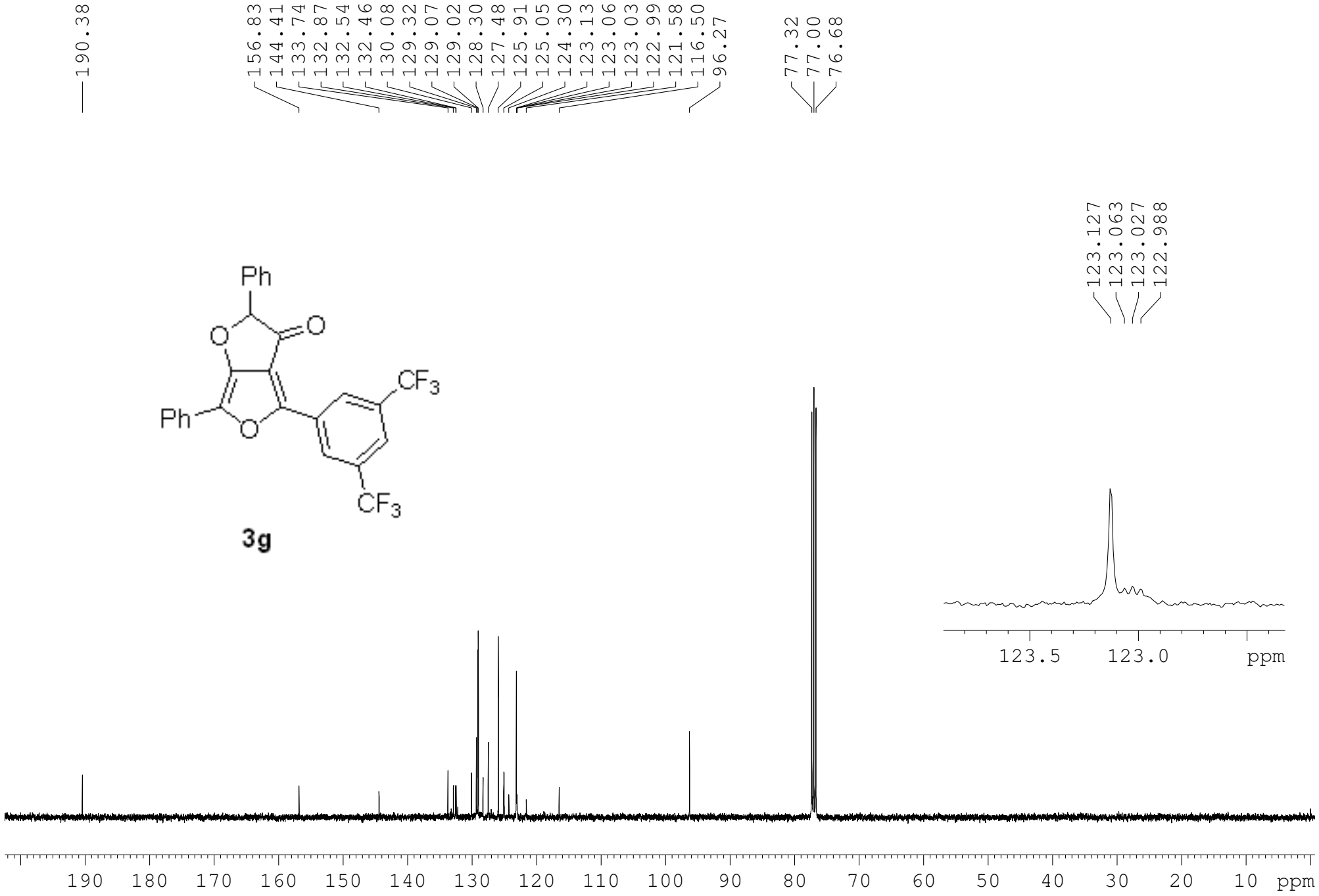
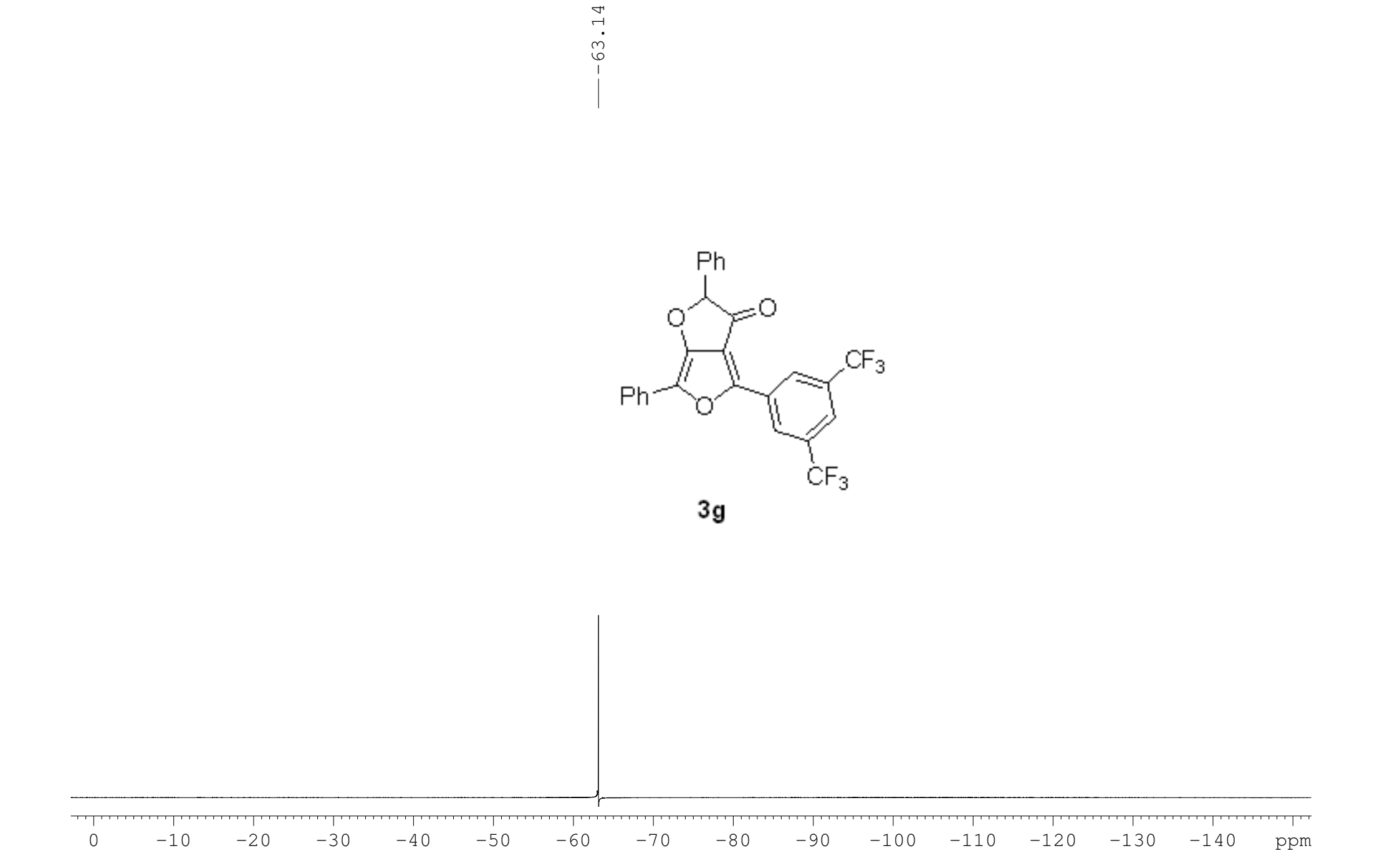
7.801
7.782
7.513
7.495
7.472
7.448
7.428
7.408
7.392
7.337
7.319
7.301
7.260
6.127


Ph
O
O
Ph
O
CF3
CF3

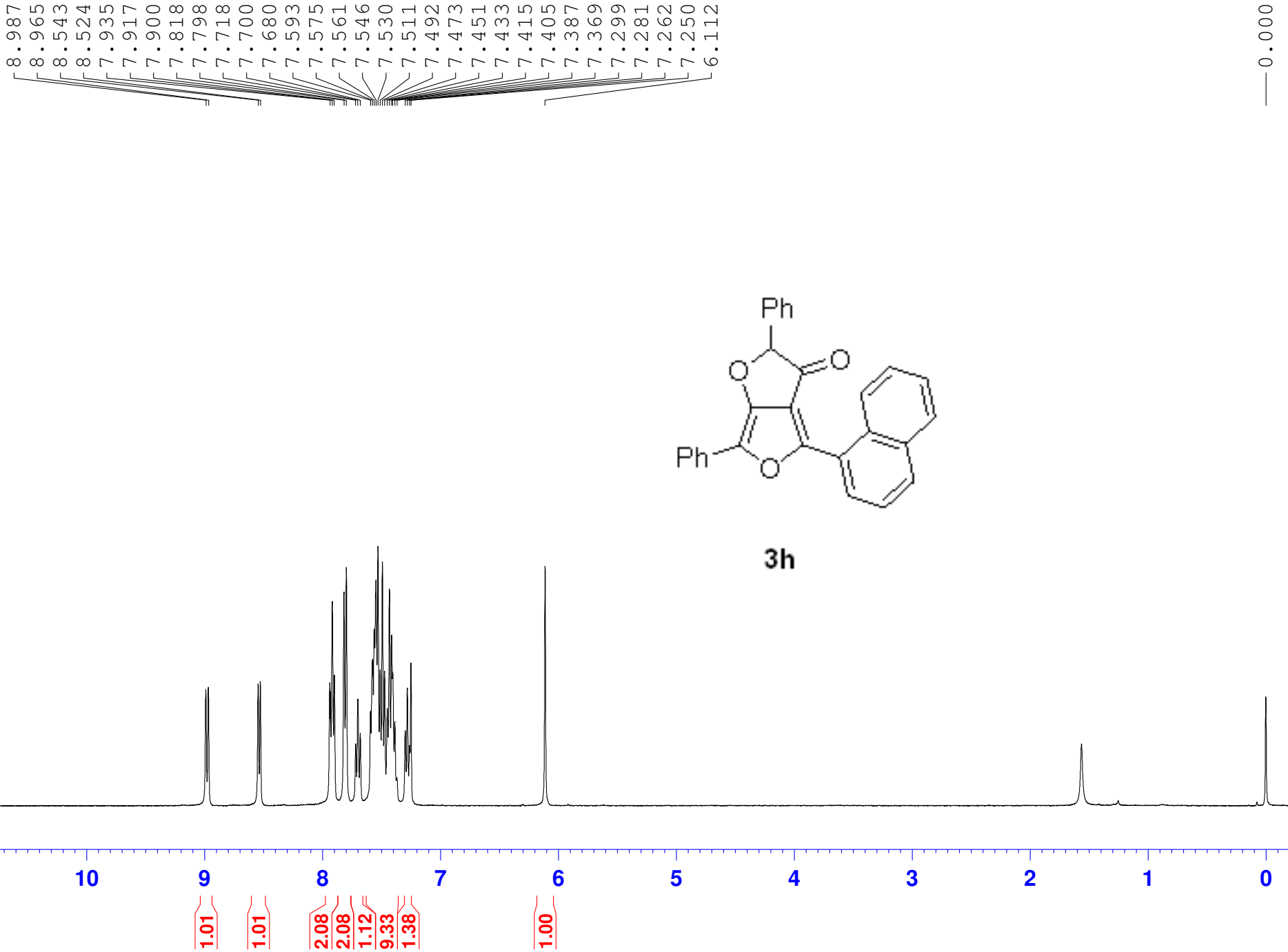
3g

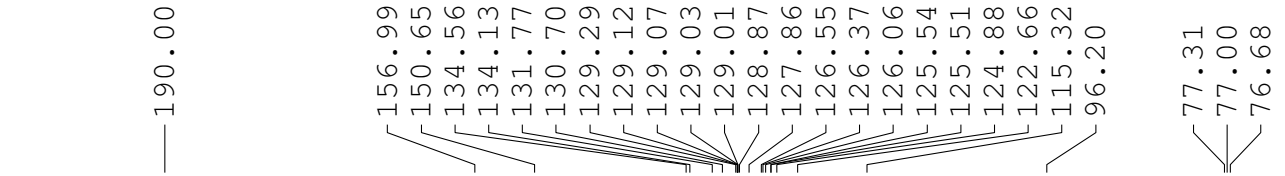

10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
2.00
1.08
2.11
7.56
1.15
1.00

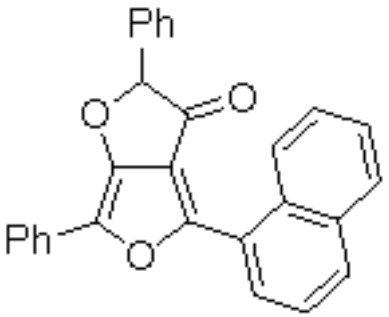

190.38
156.83
144.41
133.74
132.87
132.54
132.46
130.08
129.32
129.07
129.02
128.30
127.48
125.91
125.05
124.30
123.13
123.06
123.03
122.99
121.58
116.50
96.27
77.32
77.00
76.68
123.127
123.063
123.027
122.988
Ph
O
O
CF3
Ph
O
CF3
3g
123.5
123.0
ppm
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


-63.14
Ph
O
O
Ph
O
CF3
CF3
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
ppm

3g

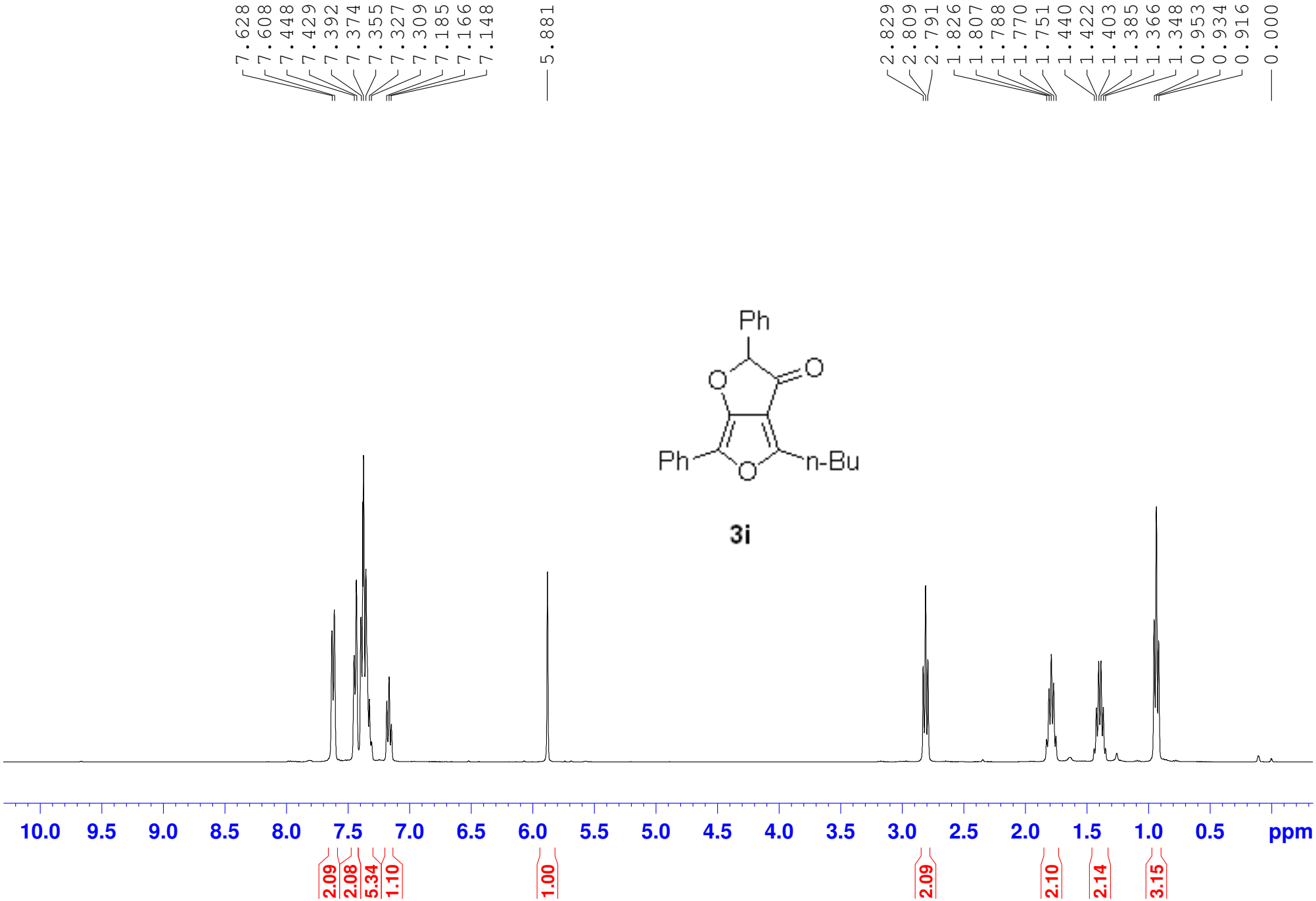

8.987
8.965
8.543
8.524
7.935
7.917
7.900
7.818
7.798
7.718
7.700
7.680
7.593
7.575
7.561
7.546
7.530
7.511
7.492
7.473
7.451
7.433
7.415
7.405
7.387
7.369
7.299
7.281
7.262
7.250
6.112
0.000
Ph
O
O
Ph
O
3h
10
9
8
7
6
5
4
3
2
1
0
ppm
1.01
1.01
2.08
2.08
1.12
9.33
1.38
1.00

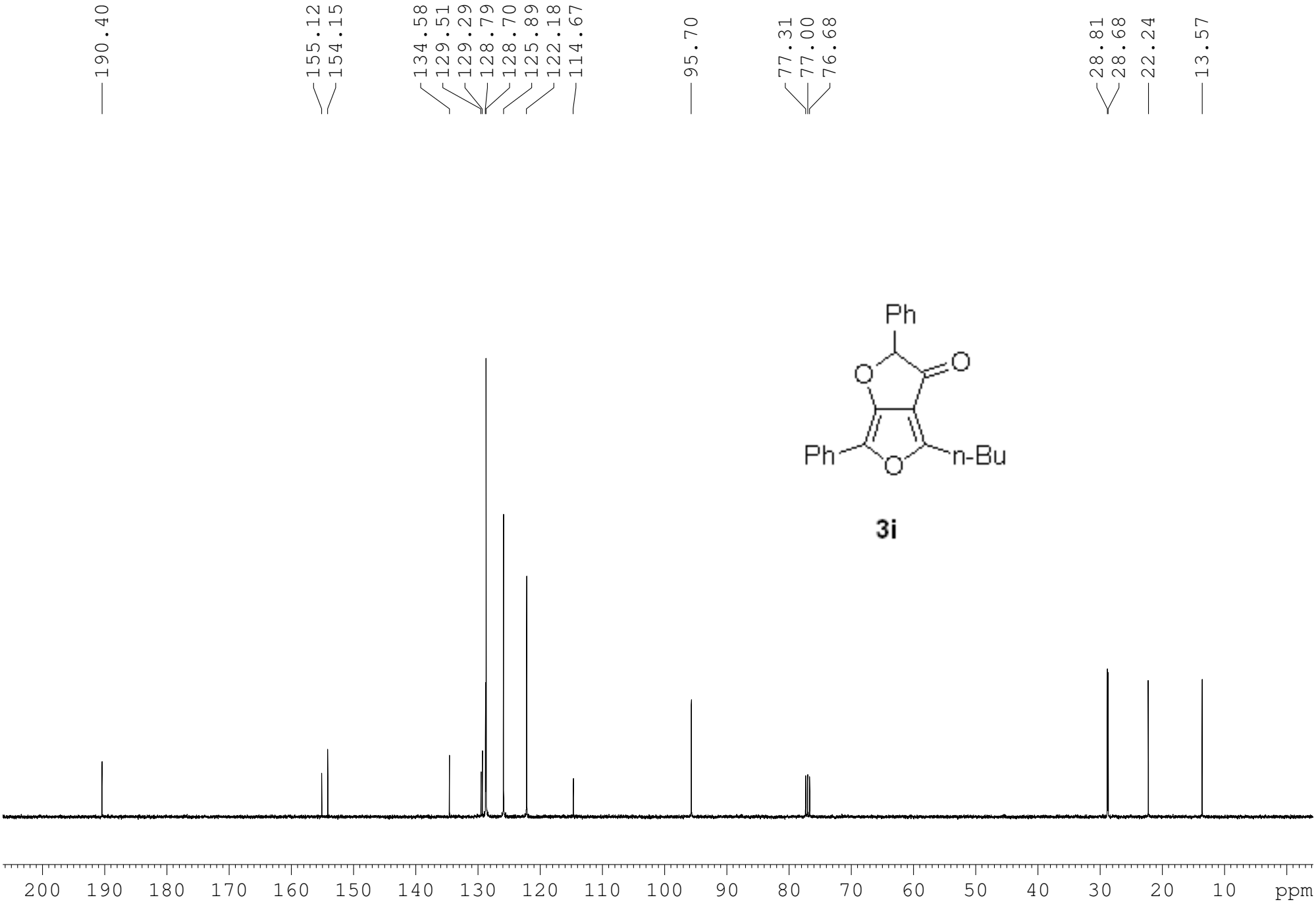

190.00
156.99
150.65
134.56
134.13
131.77
130.70
129.29
129.12
129.07
129.03
129.01
128.87
127.86
126.55
126.37
126.06
125.54
125.51
124.88
122.66
115.32
96.20
77.31
77.00
76.68


Ph
O
O
Ph
O

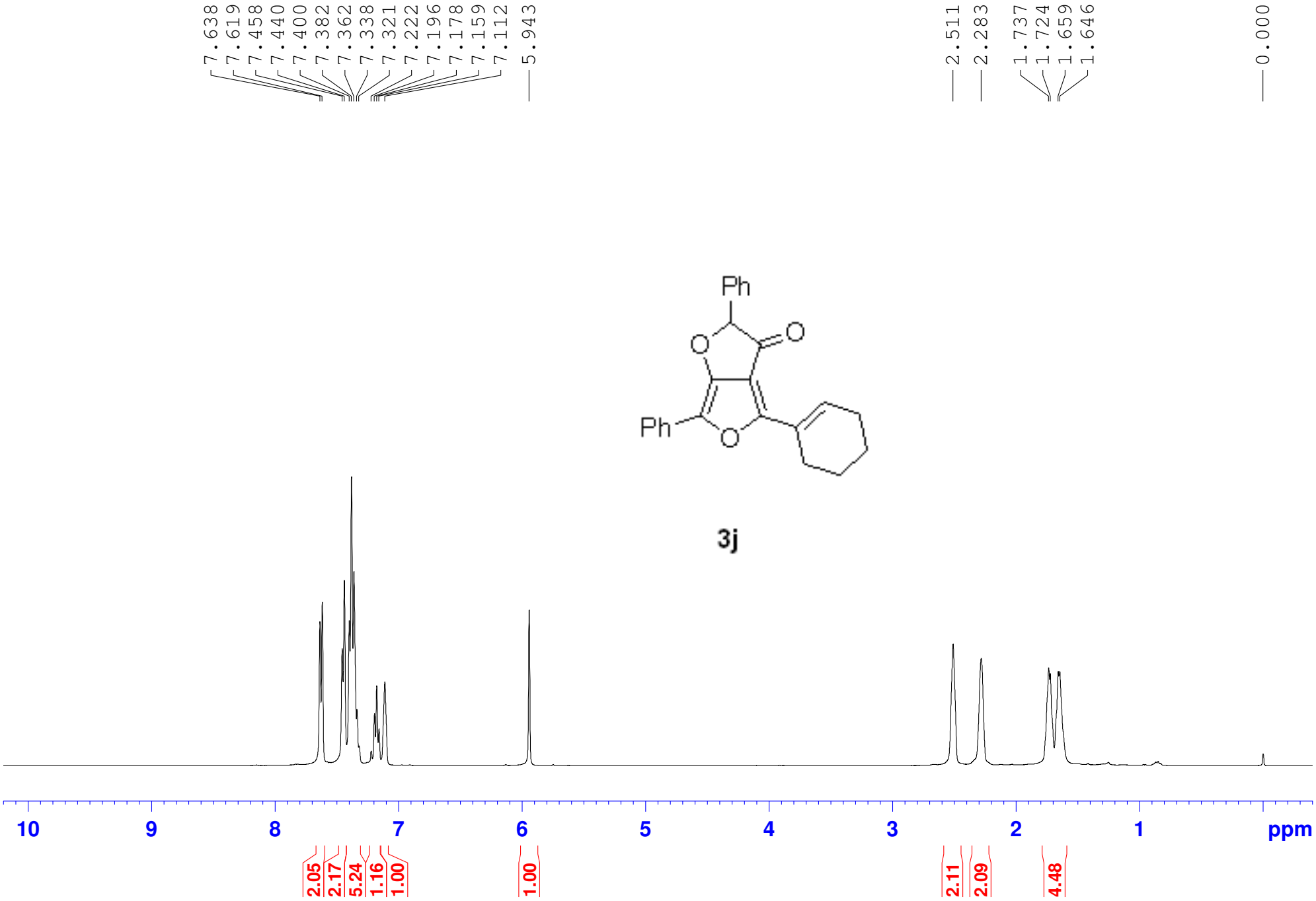
3h

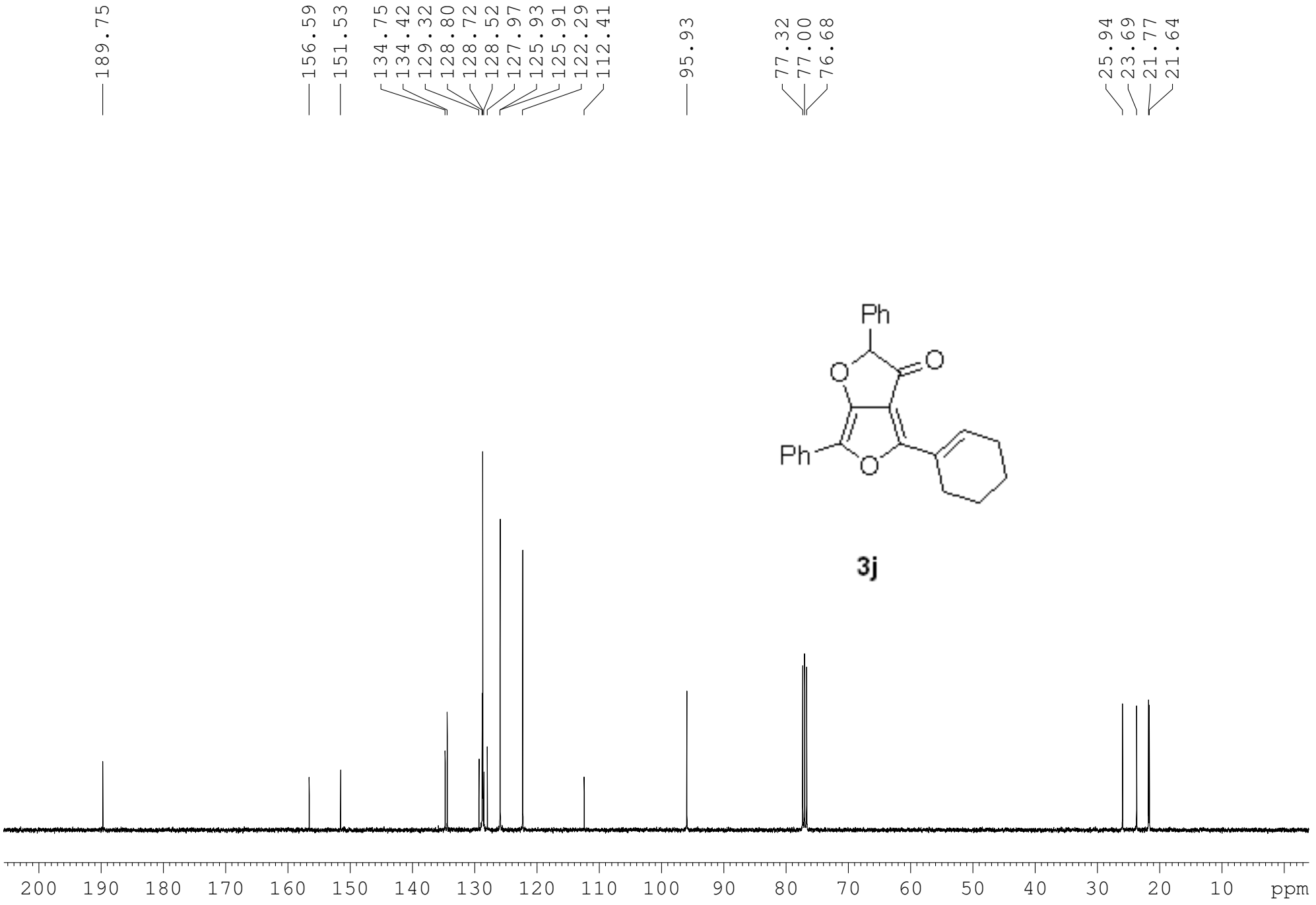
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


7.628
7.608
7.448
7.429
7.392
7.374
7.355
7.327
7.309
7.185
7.166
7.148
5.881
2.829
2.809
2.791
1.826
1.807
1.788
1.770
1.751
1.440
1.422
1.403
1.385
1.366
1.348
0.953
0.934
0.916
0.000
Ph
O
O
Ph
O
n-Bu
3i
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.09
2.08
5.34
1.10
1.00
2.09
2.10
2.14
3.15


190.40
155.12
154.15
134.58
129.51
129.29
128.79
128.70
125.89
122.18
114.67
95.70
77.31
77.00
76.68
28.81
28.68
22.24
13.57
Ph
O
O
Ph
O
n-Bu
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

3i


7.638
7.619
7.458
7.440
7.400
7.382
7.362
7.338
7.321
7.222
7.196
7.178
7.159
7.112
5.943
2.511
2.283
1.737
1.724
1.659
1.646
0.000
Ph
O
O
Ph
O
3j
10
9
8
7
6
5
4
3
2
1
ppm
2.05
2.17
5.24
1.16
1.00
1.00
2.11
2.09
4.48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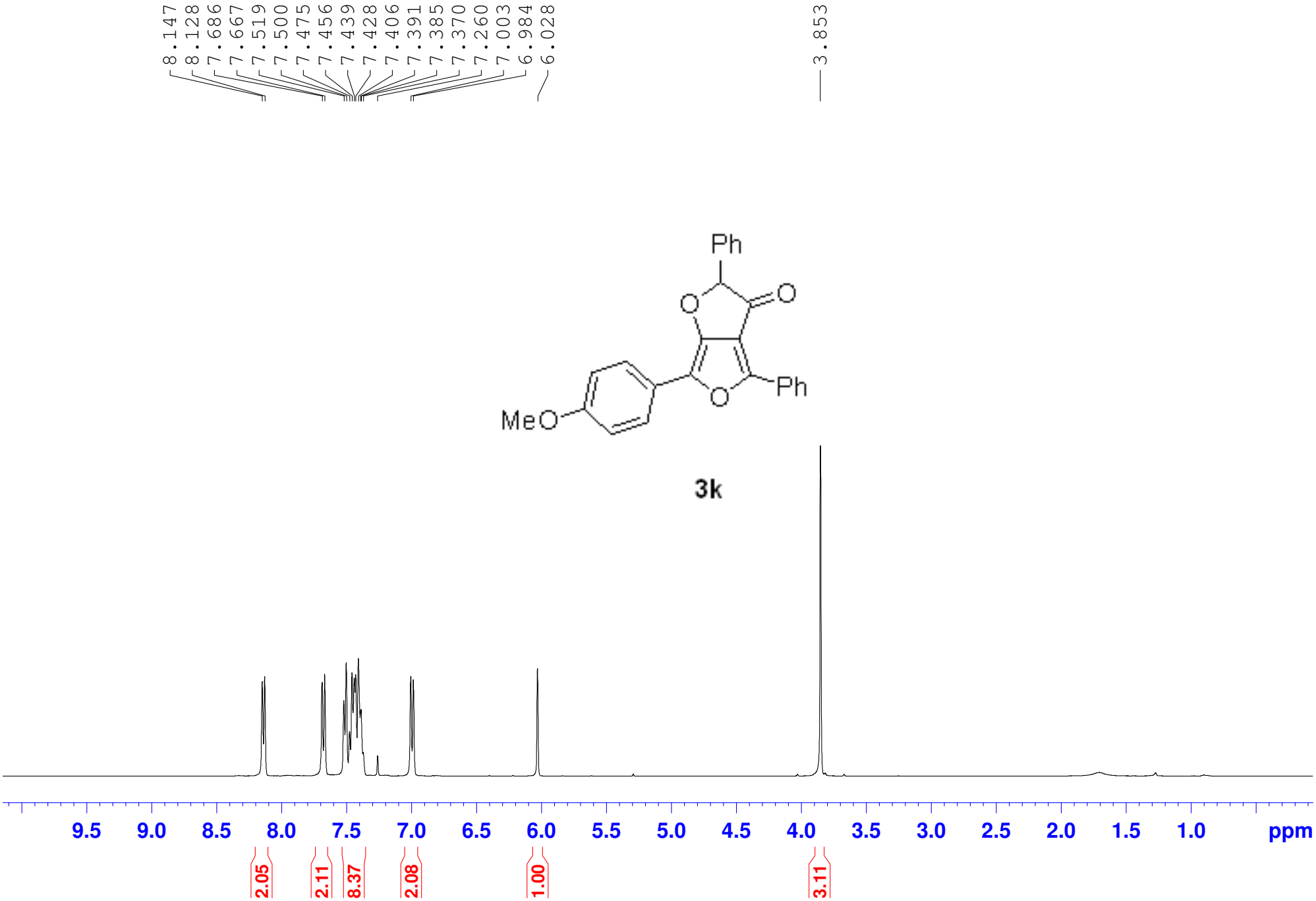
156.59
151.53
134.75
134.42
129.32
128.80
128.72
128.52
127.97
125.93
125.91
122.29
112.41
95.93
77.32
77.00
76.68
25.94
23.69
21.77
21.64
Ph
O
O
Ph
O
3j
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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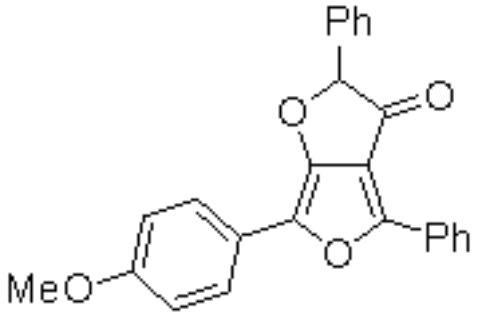
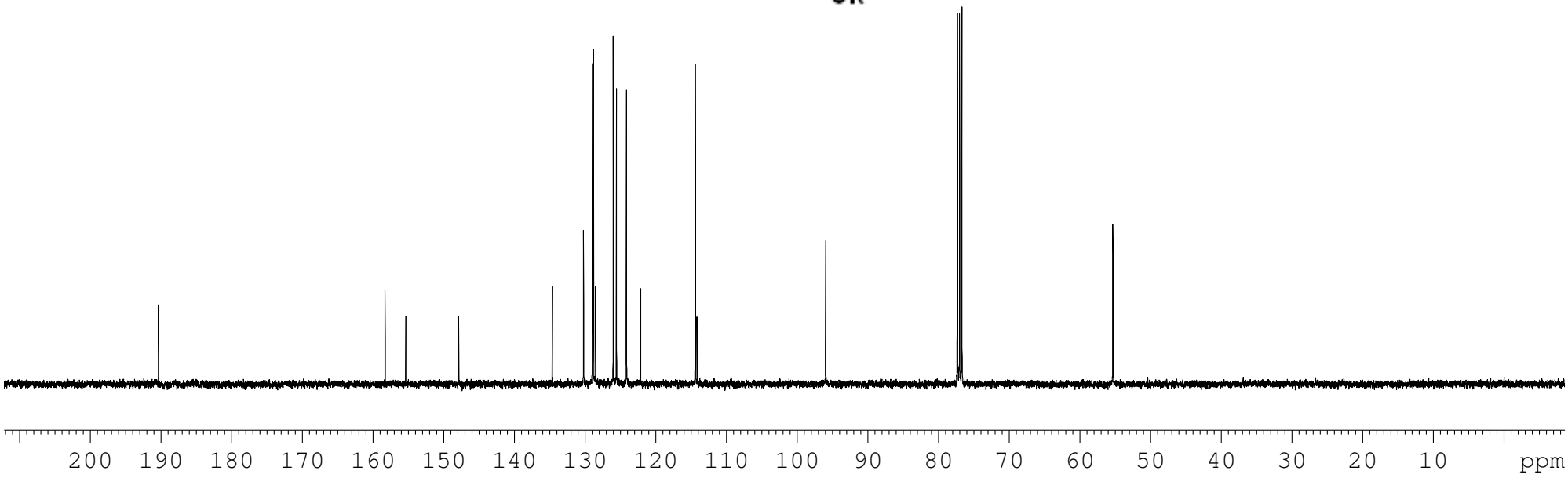
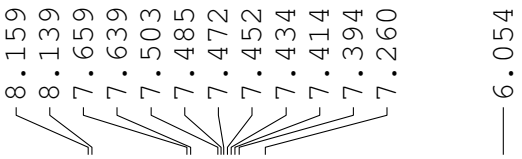
8.128
7.686
7.667
7.519
7.500
7.475
7.456
7.439
7.428
7.406
7.391
7.385
7.370
7.260
7.003
6.984
6.028
3.853
Ph
O
O
Ph
O
MeO
3k
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
2.05
2.11
8.37
2.08
1.00
3.11

190.33
158.28
155.36
147.88
134.61
130.20
130.17
128.93
128.91
128.80
128.50
126.01
125.54
124.13
122.11
114.39
114.16
95.94
77.31
77.00
76.68
55.31



3k

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8.139
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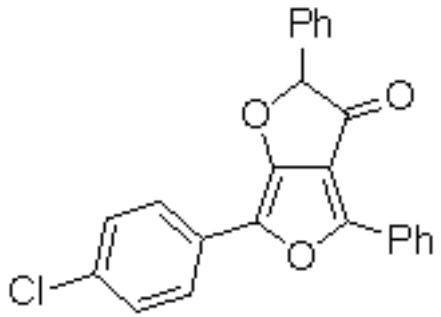
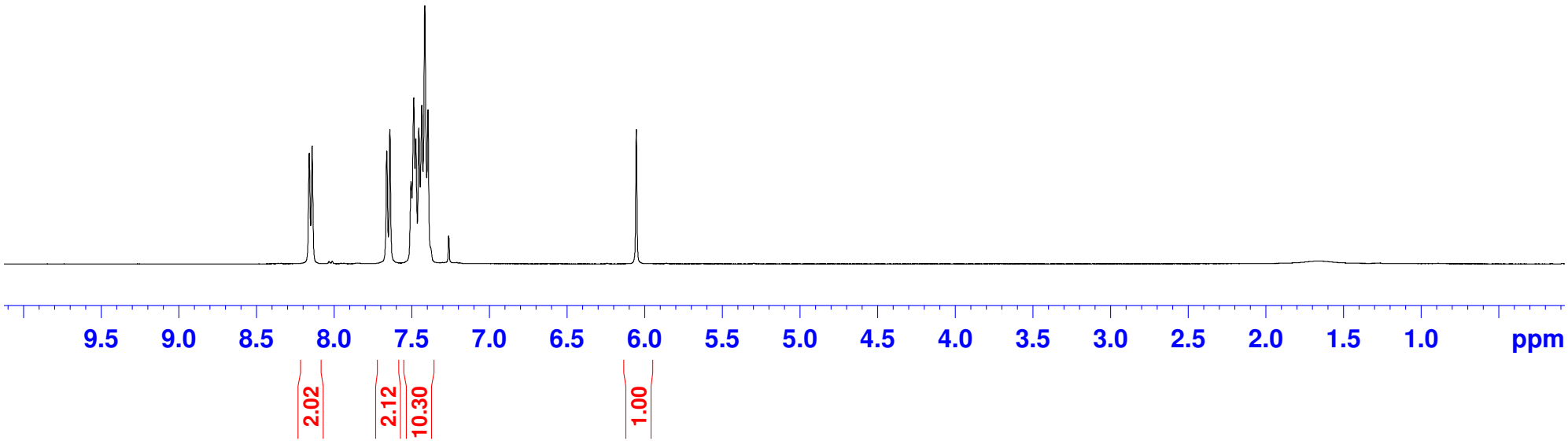
7.639
7.503
7.485
7.472
7.452
7.434
7.414
7.394
7.260
6.054


Ph
O
O
Ph
O
Cl

3l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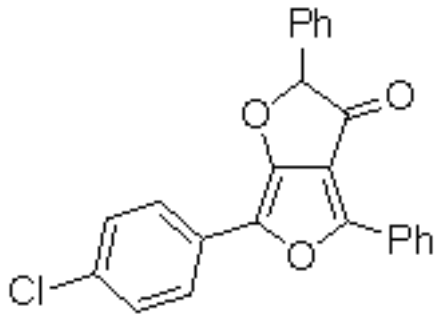
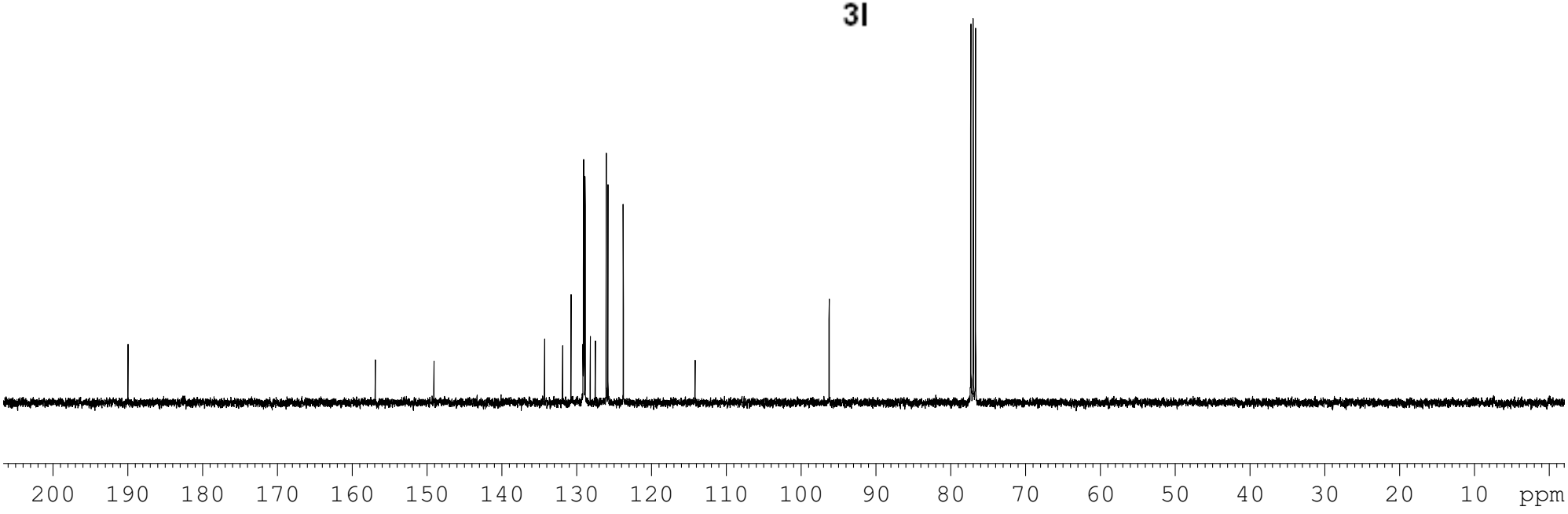
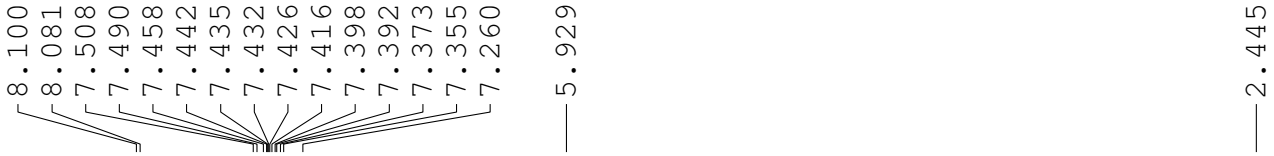
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
2.02
2.12
10.30
1.00

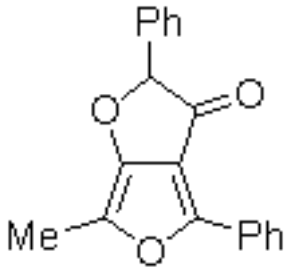
189.94
156.93
149.10
134.30
131.89
130.76
129.17
129.12
129.09
129.02
128.89
128.20
127.53
126.07
125.83
123.79
114.18
96.26
77.32
77.00
76.68



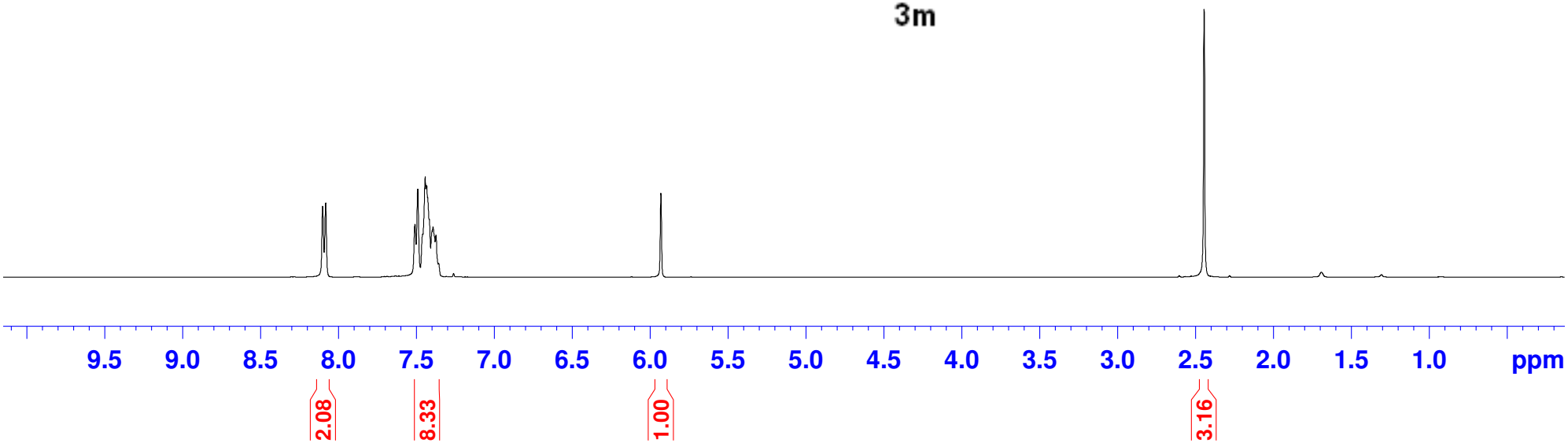
3l




8.100
8.081
7.508
7.490
7.458
7.442
7.435
7.432
7.426
7.416
7.398
7.392
7.373
7.355
7.260
5.929
2.445


Ph
O
O
Me
O
Ph

3m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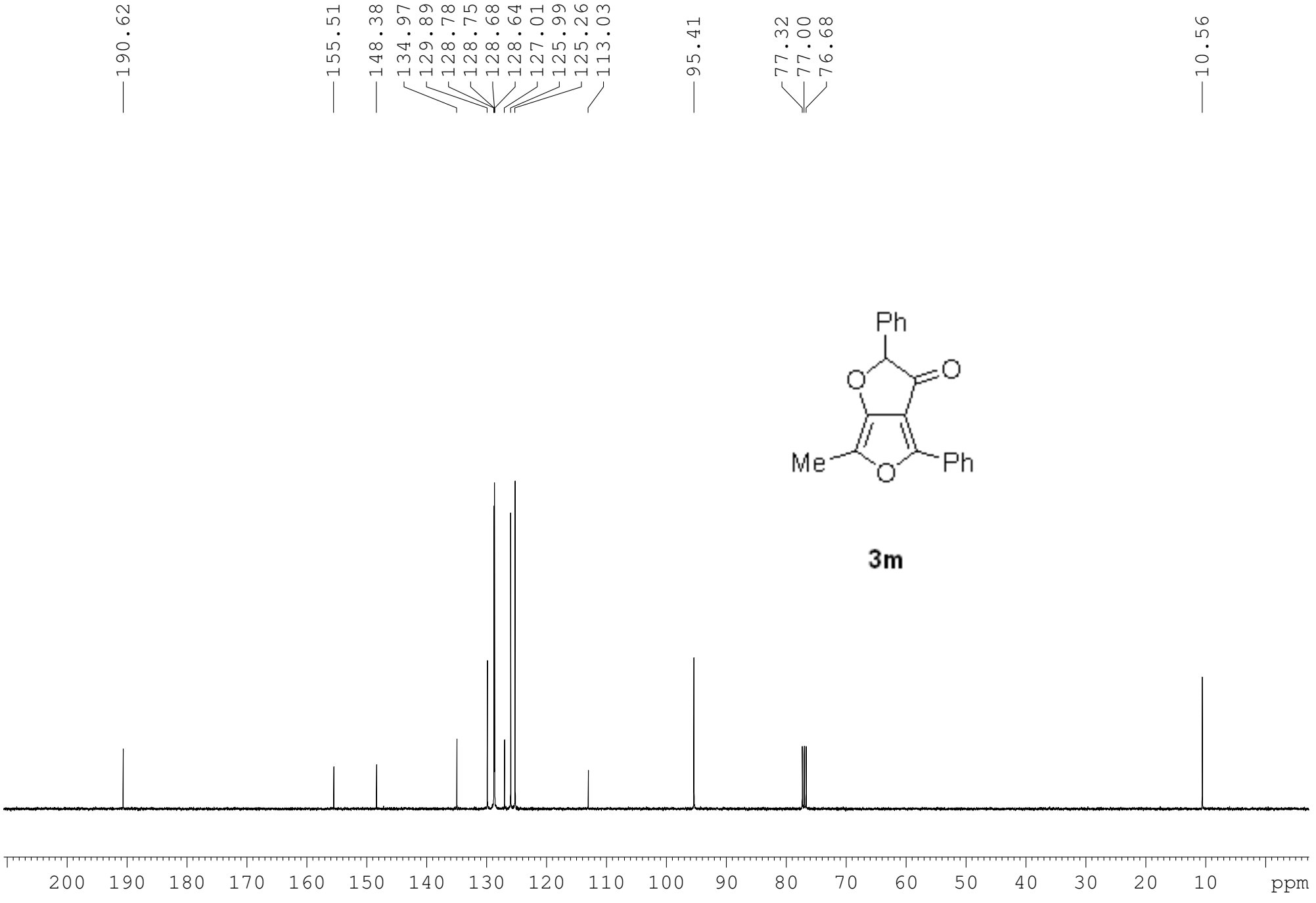
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
2.08
8.33
1.00
3.16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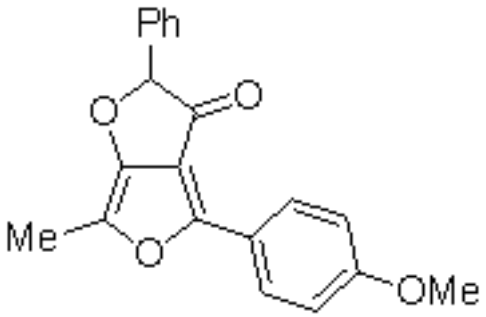
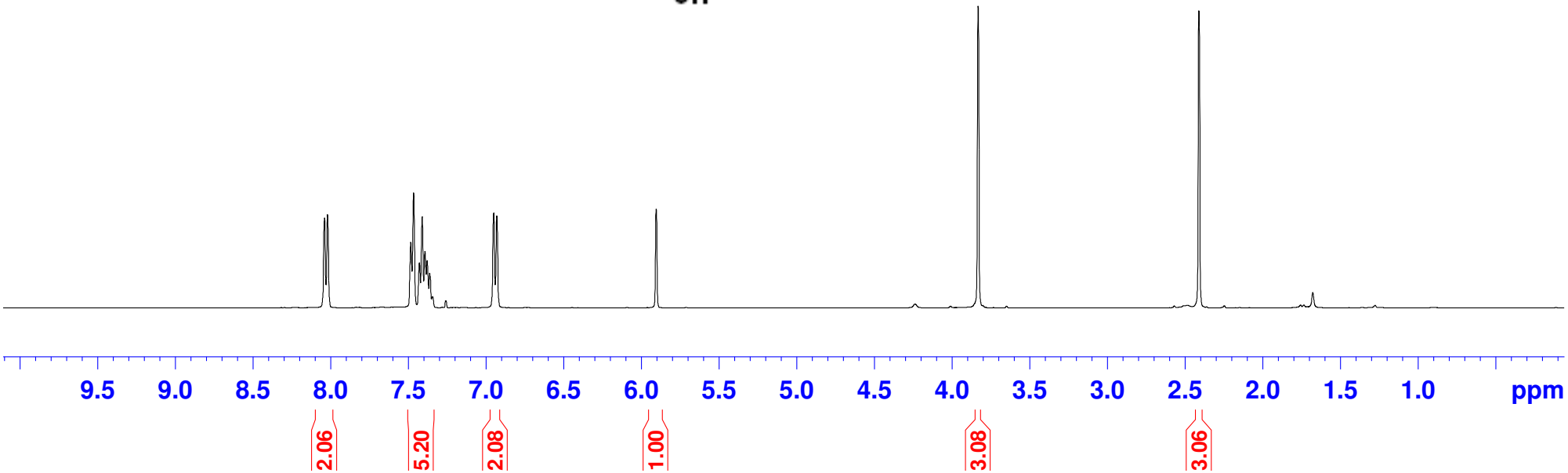
155.51
148.38
134.97
129.89
128.78
128.75
128.68
128.64
127.01
125.99
125.26
113.03
95.41
77.32
77.00
76.68
10.56
Ph
O
O
Me
O
Ph
3m
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


8.040
8.021
7.485
7.467
7.429
7.411
7.394
7.379
7.364
7.344
7.260
6.951
6.931
5.905
3.832
2.411


Ph
O
O
Me
O
OMe

3n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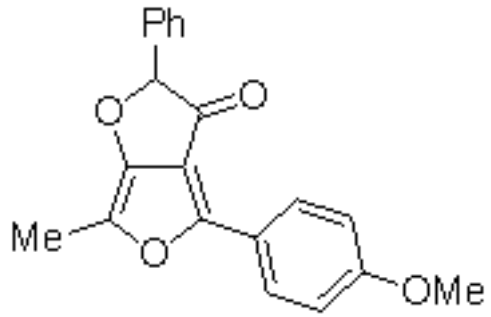
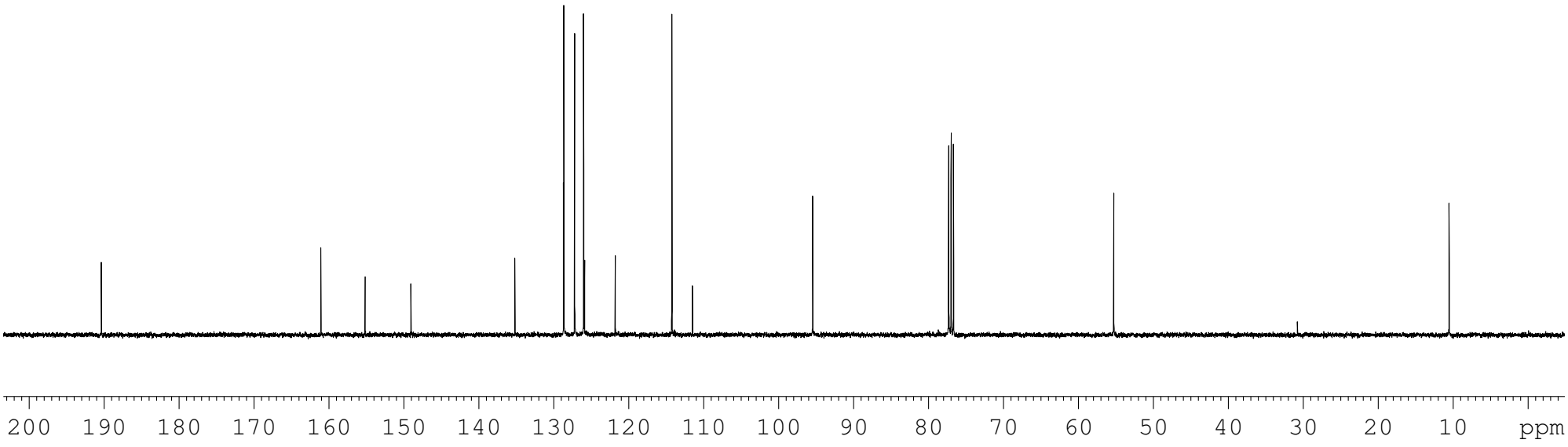
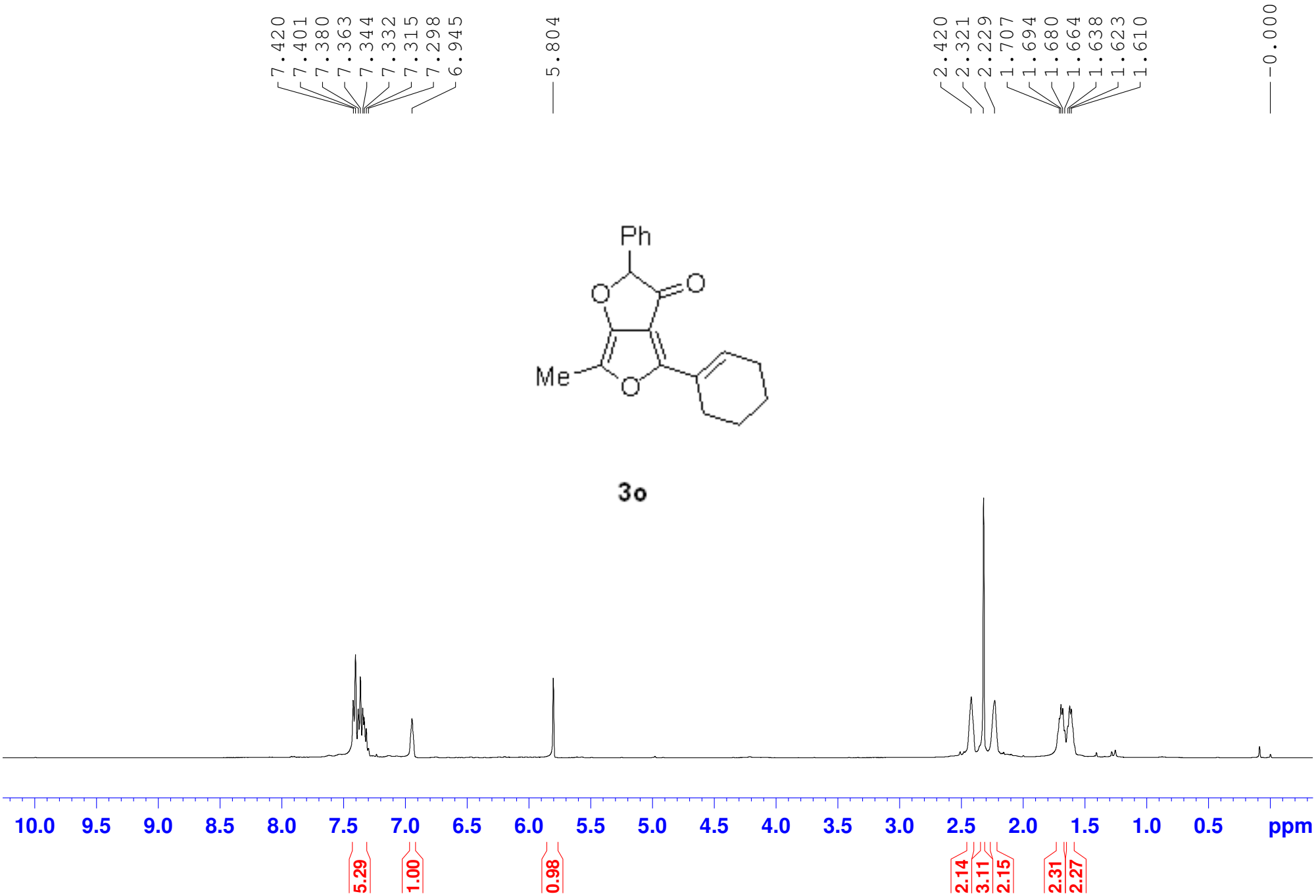
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
2.06
5.20
2.08
1.00
3.08
3.06

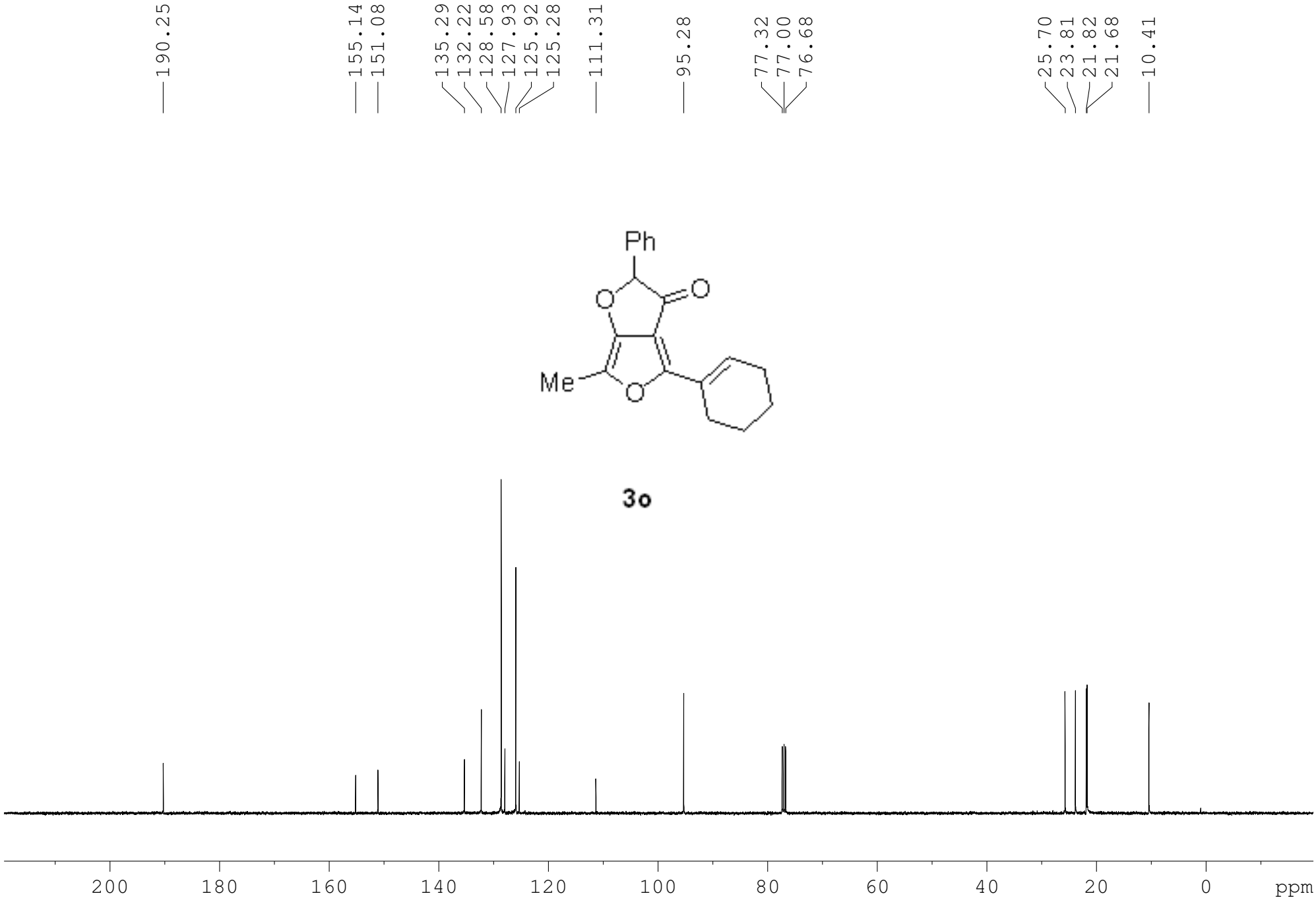

190.35
161.04
155.14
149.04
135.21
128.71
128.68
127.23
126.03
125.91
121.81
114.26
111.53
95.47
77.32
77.00
76.68
55.29
10.54


Ph
O
O
Me
O
OMe

3n


200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


7.420
7.401
7.380
7.363
7.344
7.332
7.315
7.298
6.945
5.804
2.420
2.321
2.229
1.707
1.694
1.680
1.664
1.638
1.623
1.610
0.000
Ph
O
O
Me
O
3o
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
5.29
1.00
0.98
2.14
3.11
2.15
2.31
2.27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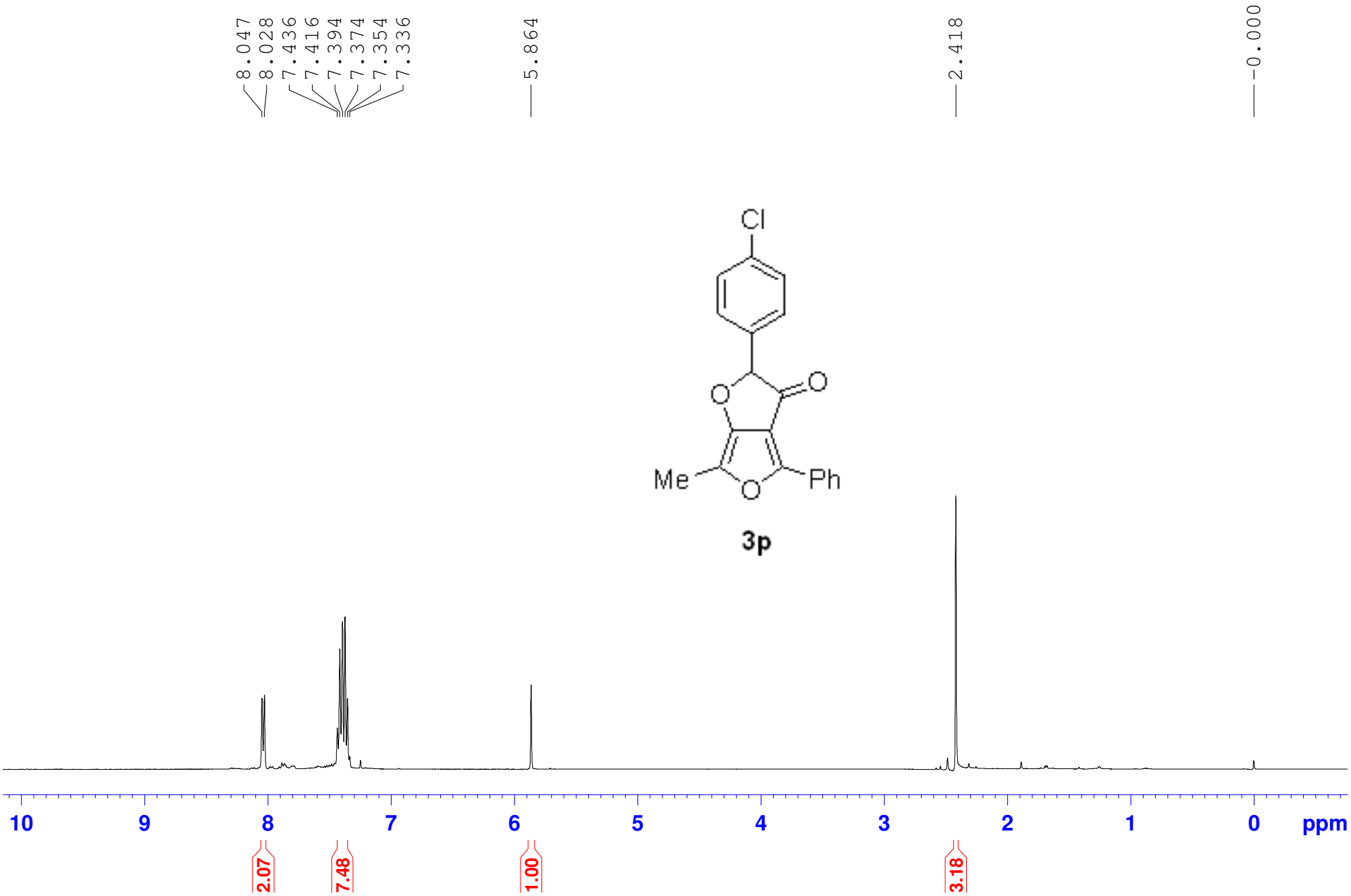
155.14
151.08
135.29
132.22
128.58
127.93
125.92
125.28
111.31
95.28
77.32
77.00
76.68
25.70
23.81
21.82
21.68
10.41
Ph
O
O
Me
O
3o
200
180
160
140
120
100
80
60
40
20
0
ppm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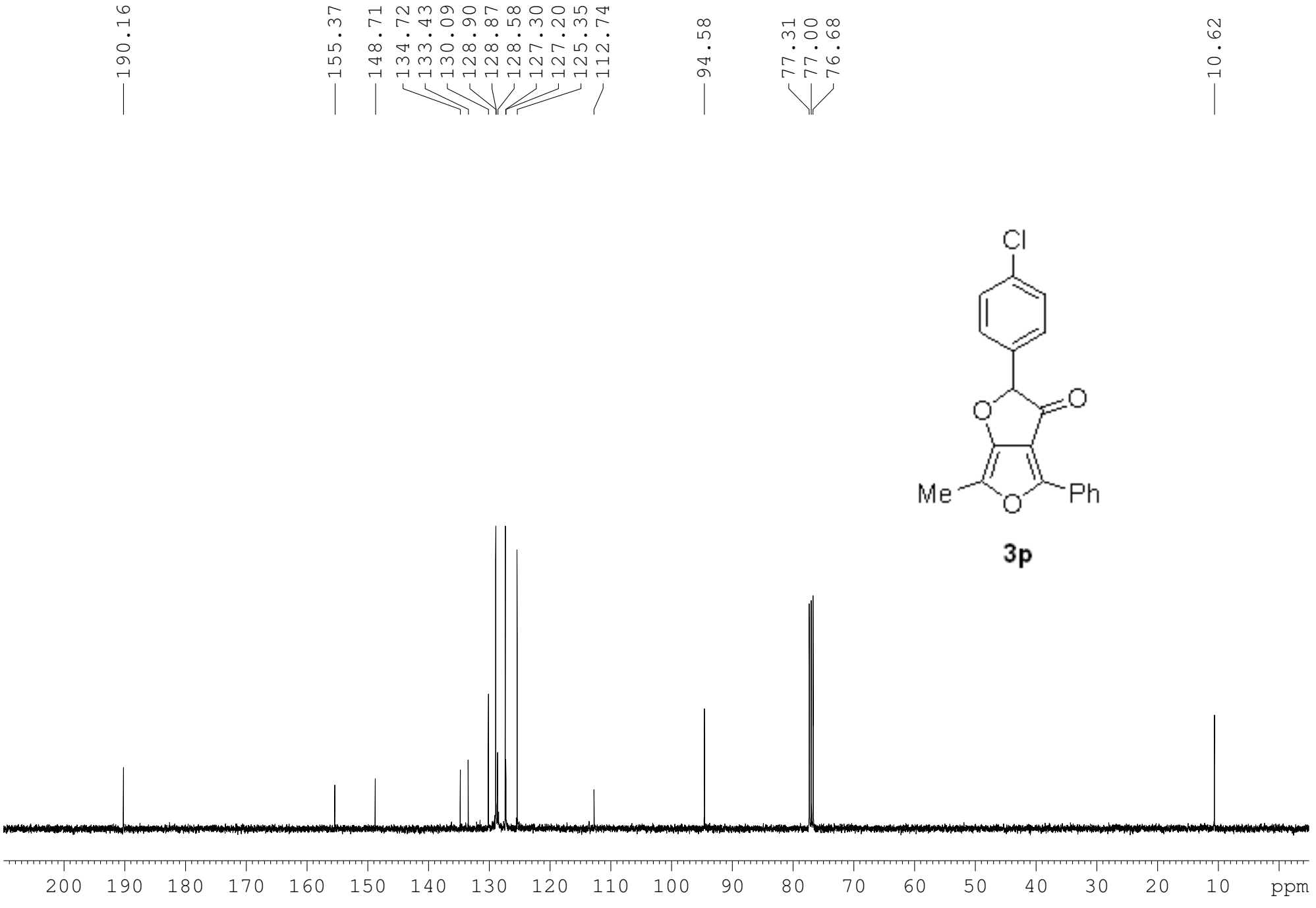
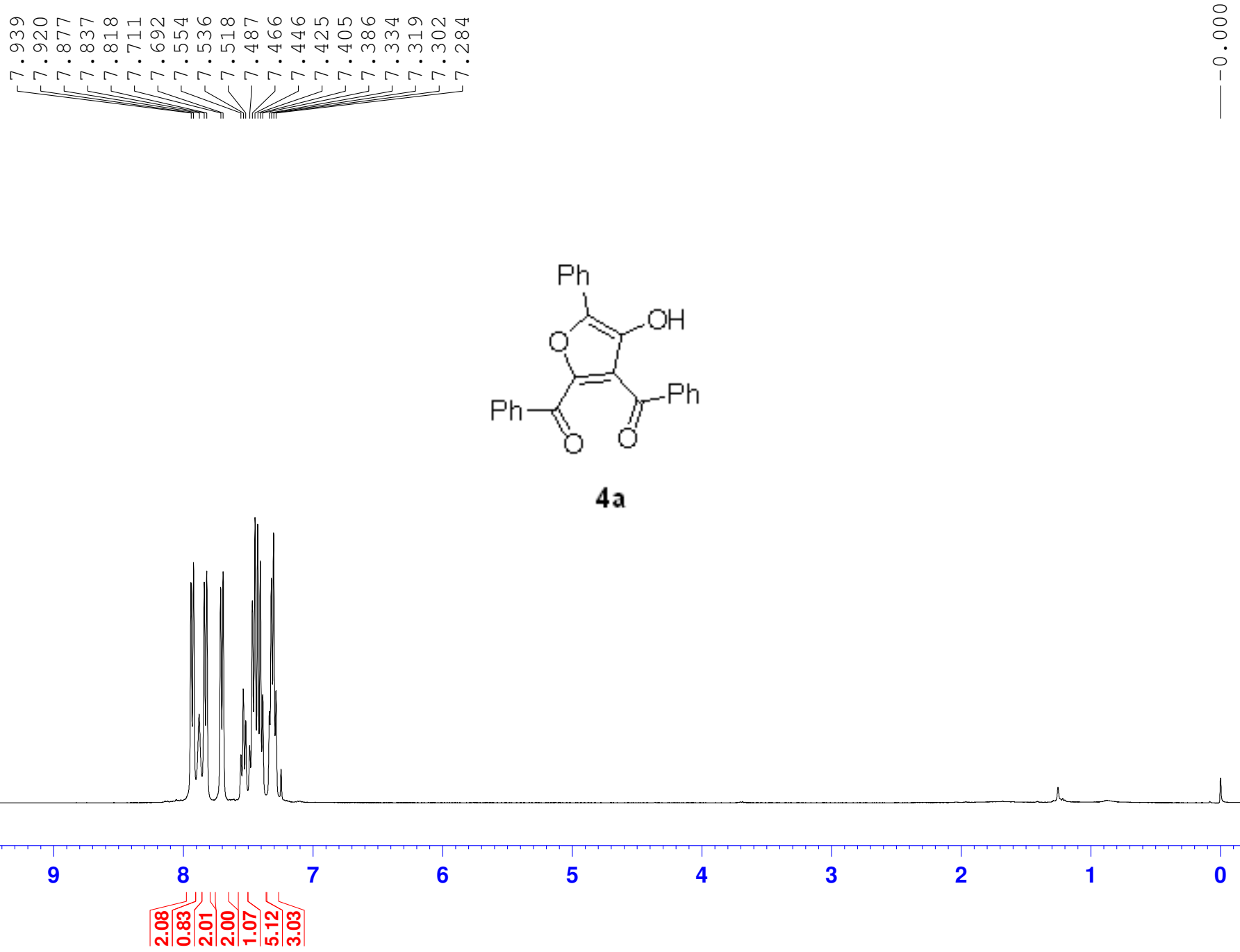
8.028
7.436
7.416
7.394
7.374
7.354
7.336
5.864
2.418
0.000
Cl
O
O
Me
O
Ph
3p
10
9
8
7
6
5
4
3
2
1
0
ppm
2.07
7.48
1.00
3.18


190.16
155.37
148.71
134.72
133.43
130.09
128.90
128.87
128.58
127.30
127.20
125.35
112.74
94.58
77.31
77.00
76.68
10.62
Cl
O
O
Me
O
Ph
3p
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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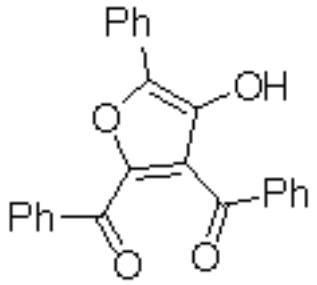
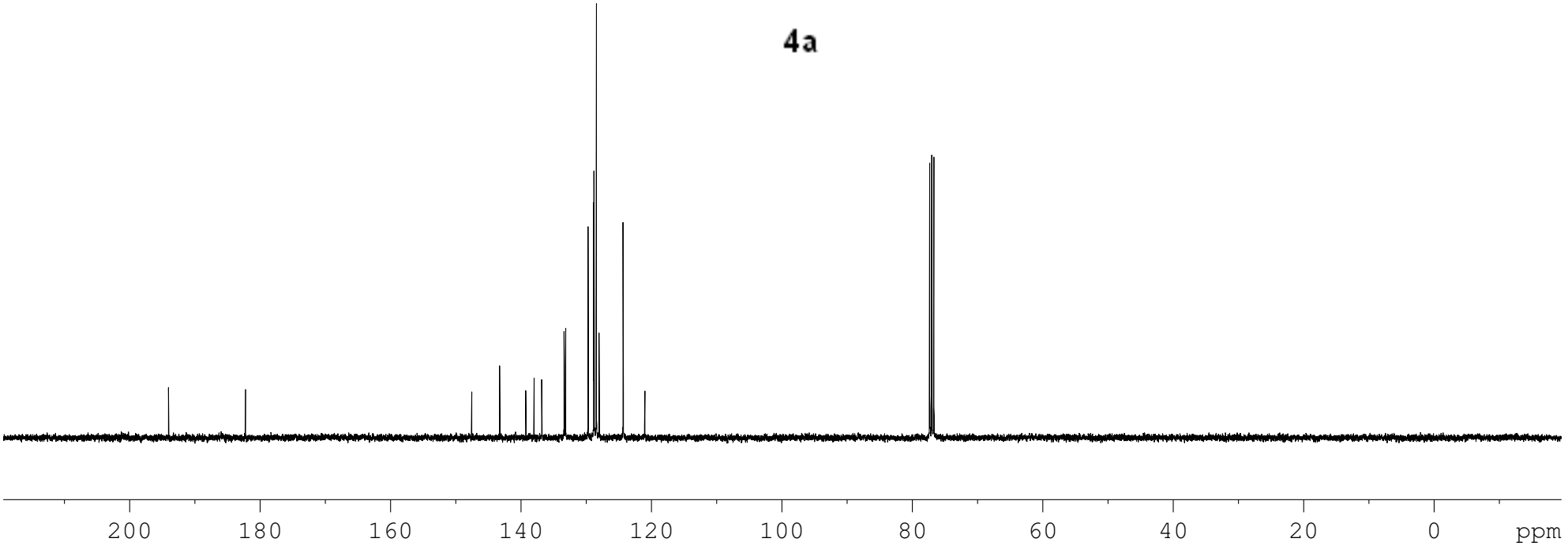
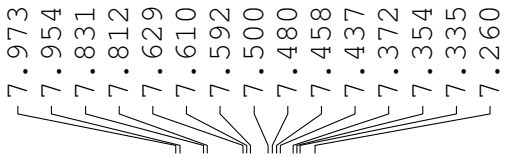
7.837
7.818
7.711
7.692
7.554
7.536
7.518
7.487
7.466
7.446
7.425
7.405
7.386
7.334
7.319
7.302
7.284
0.000
Ph
OH
O
Ph
Ph
O
O
4a
10
9
8
7
6
5
4
3
2
1
0
ppm
2.08
0.83
2.01
2.00
1.07
5.12
3.03

194.02
182.22
147.54
143.24
139.24
137.97
136.80
133.37
133.15
129.69
128.83
128.78
128.43
128.00
124.32
121.00
77.32
77.00
76.68



4a

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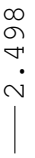
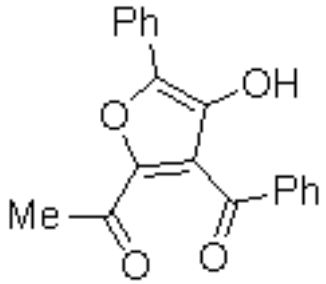
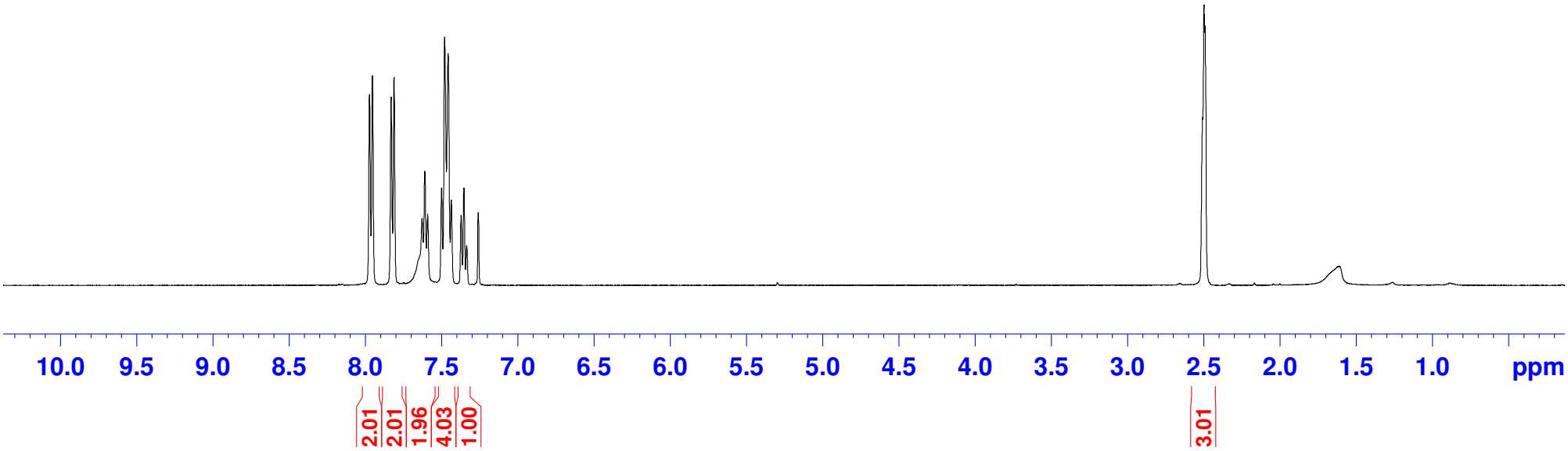
7.831
7.812
7.629
7.610
7.592
7.500
7.480
7.458
7.437
7.372
7.354
7.335
7.260


2.498


Ph
OH
O
Me
Ph
O
O

4m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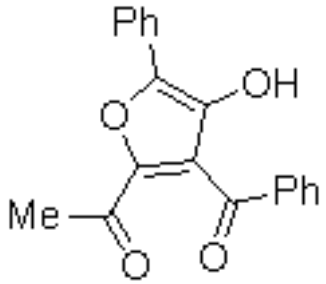
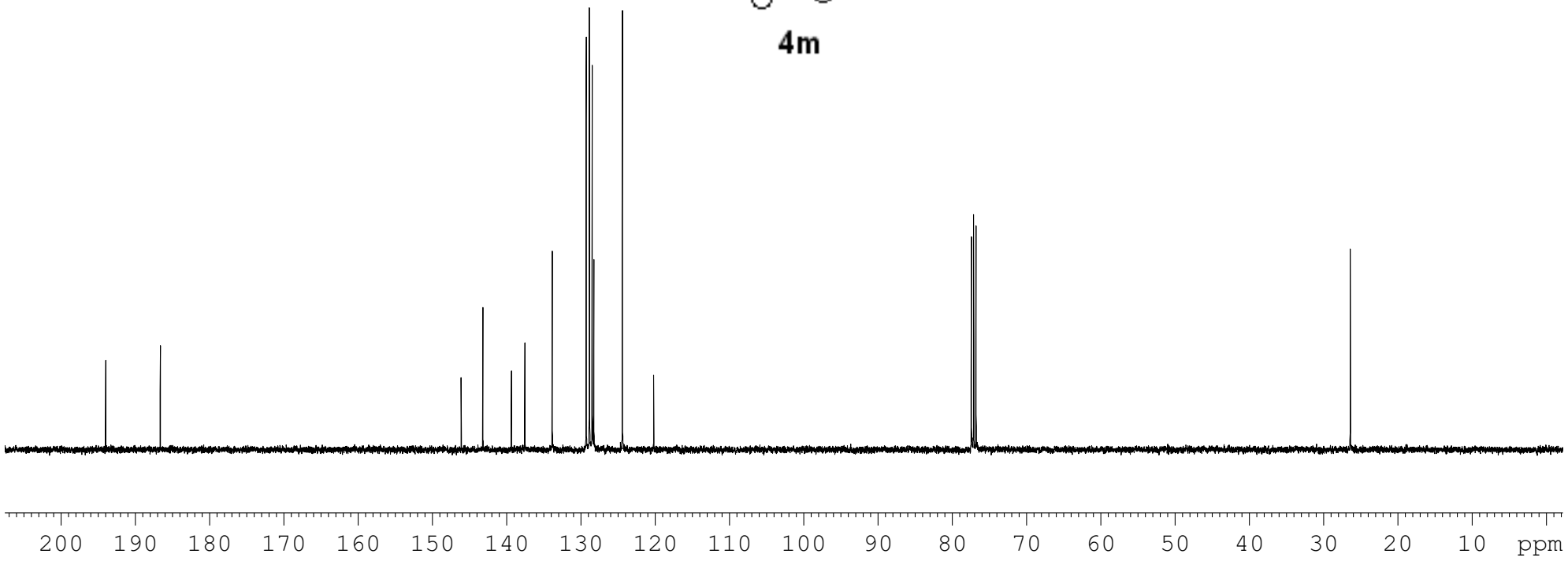
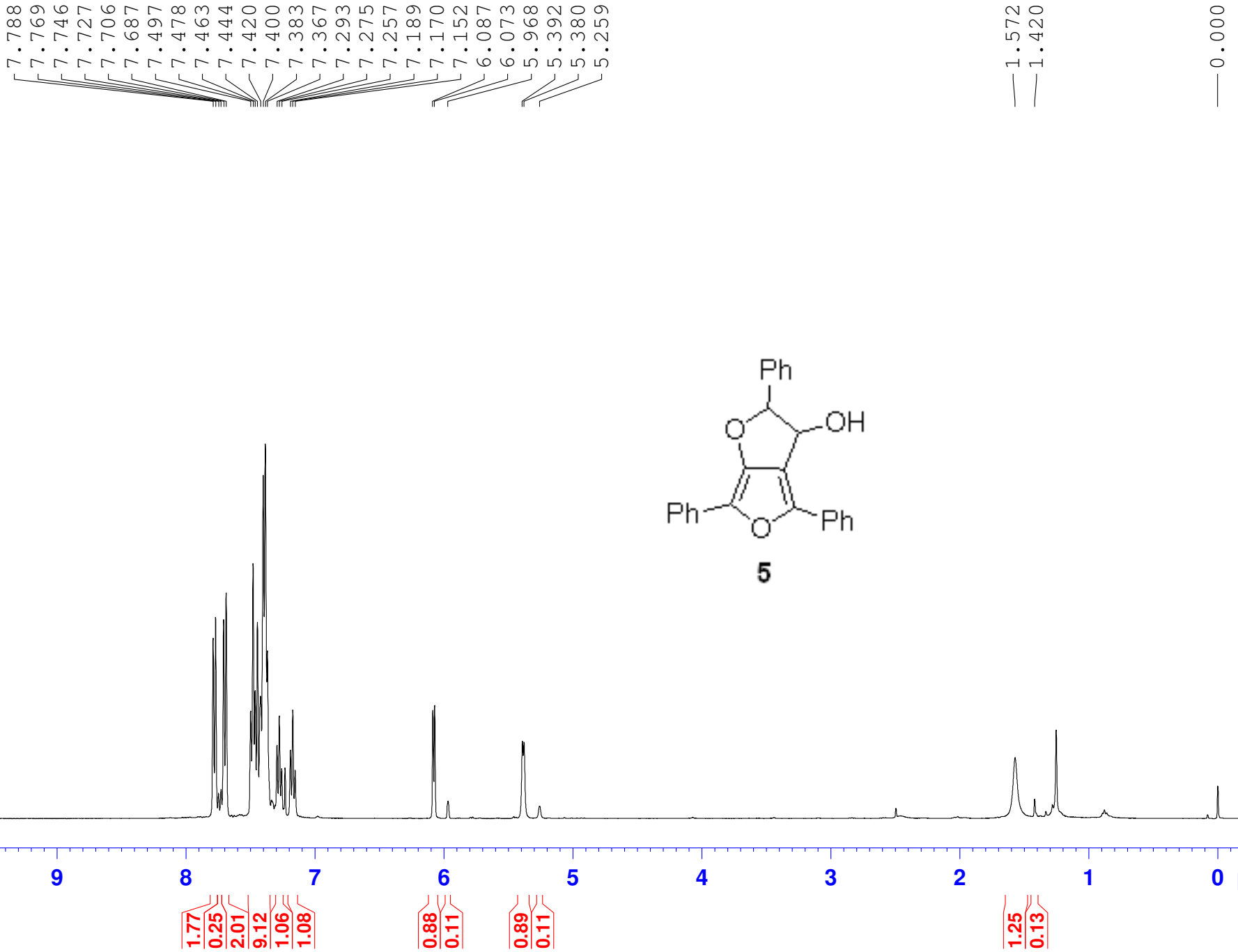
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
2.01
2.01
1.96
4.03
1.00
3.01

193.98
186.62
146.11
143.18
139.35
137.53
133.84
129.28
128.85
128.80
128.47
128.24
124.41
120.19
77.42
77.10
76.78
26.40



4m

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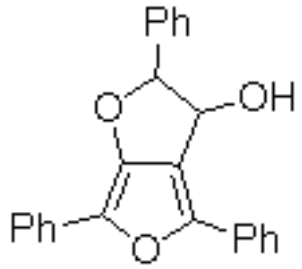
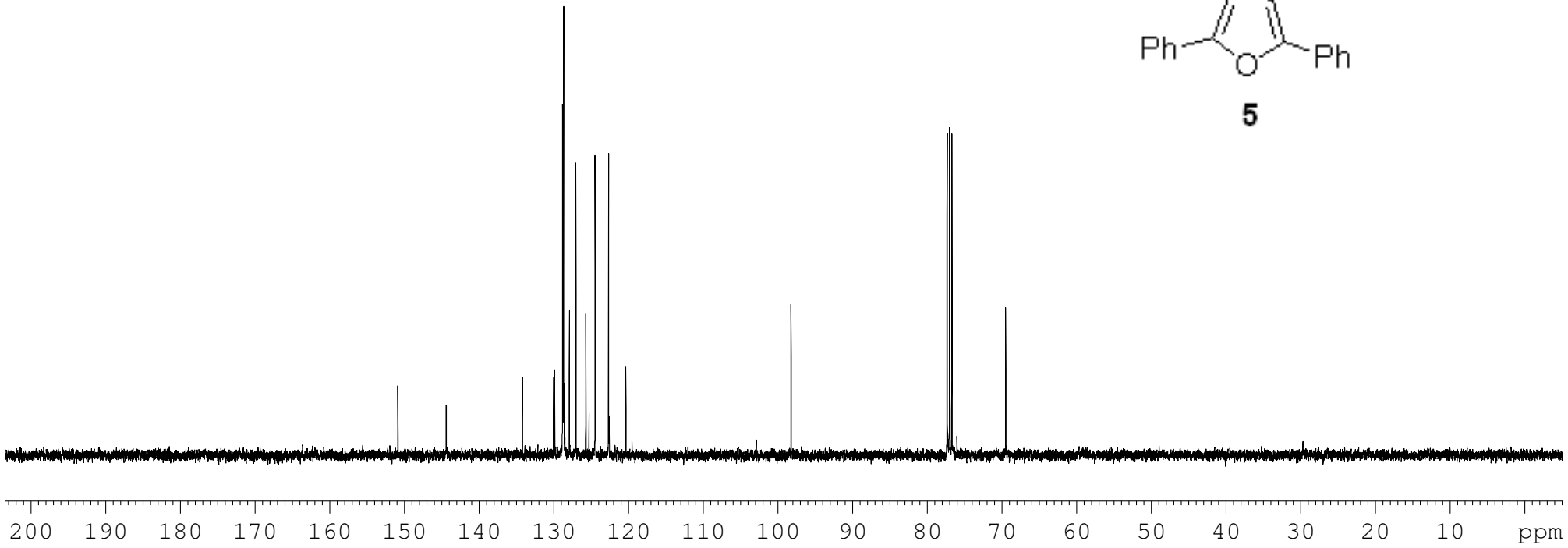
7.746
7.727
7.706
7.687
7.497
7.478
7.463
7.444
7.420
7.400
7.383
7.367
7.293
7.275
7.257
7.189
7.170
7.152
6.087
6.073
5.968
5.392
5.380
5.259
1.572
1.420
0.000
Ph
OH
O
Ph
O
Ph
5
10
9
8
7
6
5
4
3
2
1
0
ppm
1.77
0.25
2.01
9.12
1.06
1.08
0.88
0.11
0.89
0.11
1.25
0.13

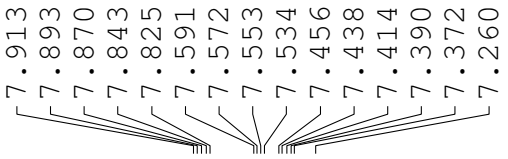
150.86
144.39
134.19
130.01
129.87
128.79
128.67
128.61
127.90
127.02
125.70
125.25
124.46
124.41
122.65
122.57
120.33
119.53
102.88
98.22

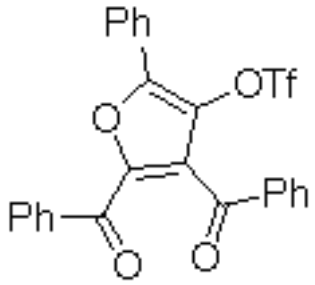
77.32
77.00
76.68
76.02
69.48



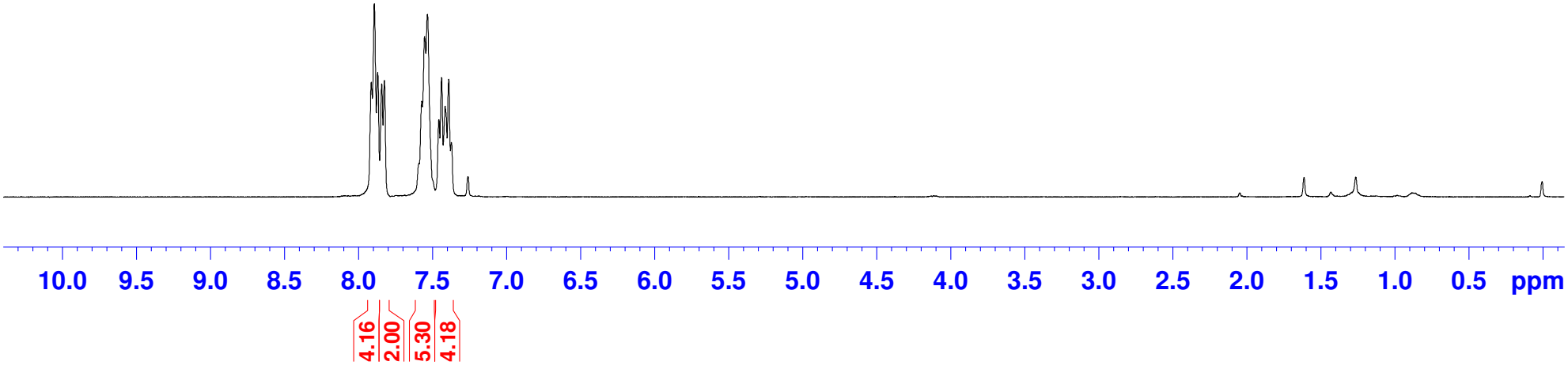
5




7.913
7.893
7.870
7.843
7.825
7.591
7.572
7.553
7.534
7.456
7.438
7.414
7.390
7.372
7.260


Ph
OTf
O
Ph
Ph
O
O

6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3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
4.16
2.00
5.30
4.18

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181.67
147.41
147.14
136.85
135.95
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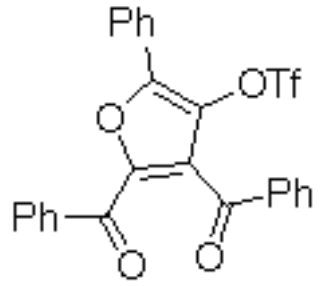

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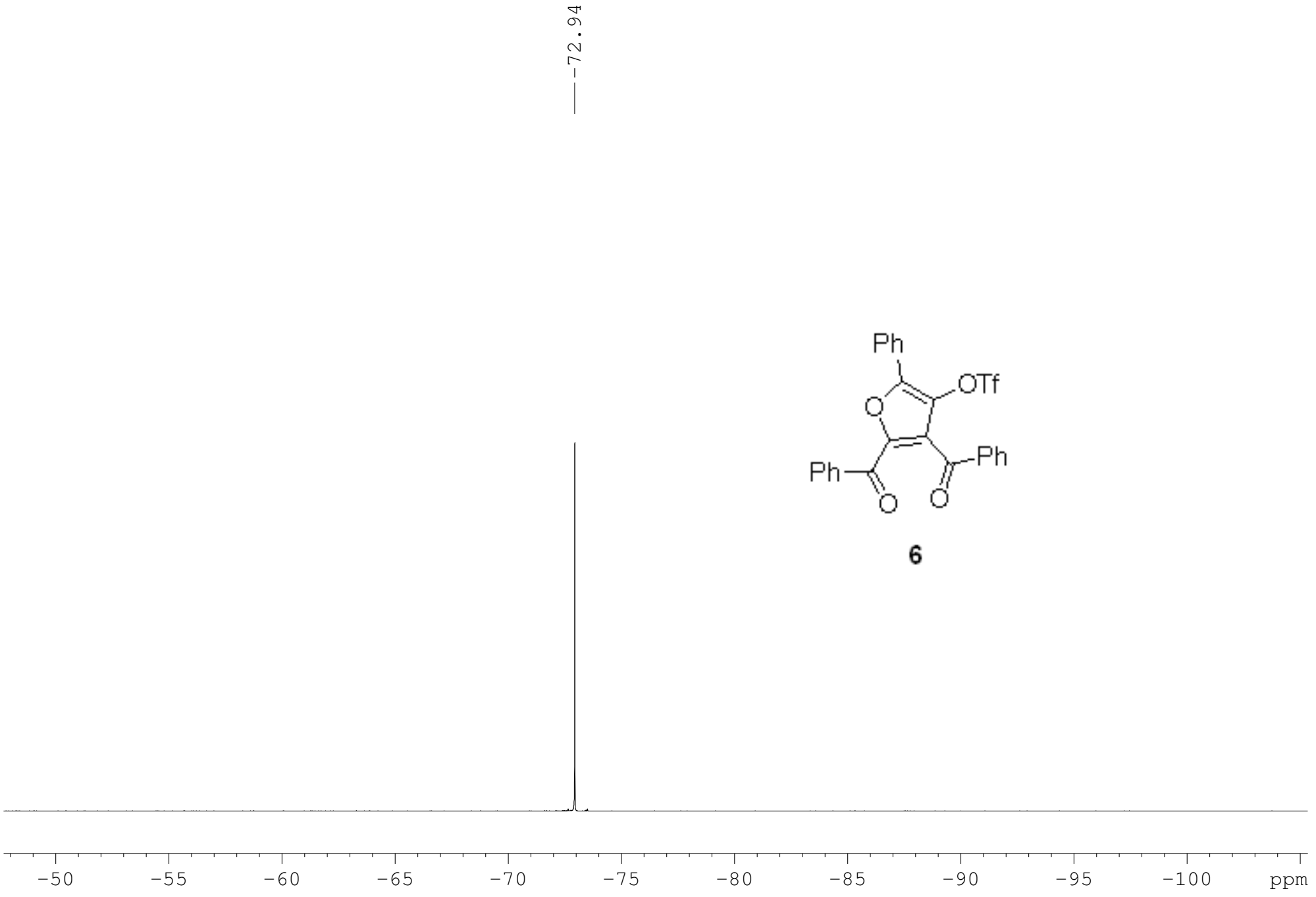

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