Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Supporting Information for

Pd/Cu-Catalyzed Cascade Sonogashira coupling/Cyclization Reactions to Highly Substituted 3-Formyl Furans

Jingyu Yang, Chengyu Wang, Xin Xie, Hongfeng Li, Ende Li and Yanzhong Li*

Institute of Medicinal Chemistry, Department of Chemistry, East China Normal University, 3663 North Zhongshan Road, Shanghai, 200062, People's Republic of China

Fax: (+86) 021-62233969, E-mail: yzli@chem.ecnu.edu.cn

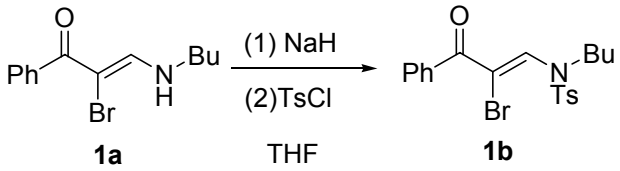
Contents:

Experimental section

General Methods. All reactions were carried out under argon. Toluene and THF were distilled from sodium/benzophenone. Et_3N was distilled from KOH. $Pd(PPh_3)_2Cl_2$ and CuI were used as purchased. Unless noted, all commercial reagents were used without further purification.

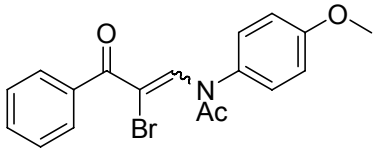
^{1}H NMR spectra was recorded at 300 or 400 MHz, ^{13}C NMR spectra was recorded at 75 or 100 MHz, and in $CDCl_3$ (containing 0.03% TMS) solutions. ^{1}H NMR spectra was recorded with tetramethylsilane (δ = 0.00 ppm) as internal reference; ^{13}C NMR spectra was recorded with $CDCl_3$ (δ = 77.00 ppm) as internal reference. High-resolution mass spectra was obtained by using Waters Micromass GCT mass spectrometer.

Sythesis of 1b.



1a **1b**

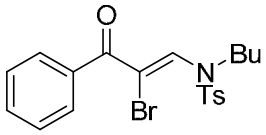
To a solution of **1a** (2.45 g, 8.7 mmol) in THF was added NaH (10.4 mmol) at 0^{o}C and stirred at 0 ^{o}C for 1h, then TsCl (1.99 g, 10.4 mmol) was added at 0^{o}C for 1h, and stirred at room temperature for another 1h. The reaction was quenched with H_2O, the organic layer was separated, and the aqueous layer was extracted with EtOAc. The combined organic layers were dried over Na_2SO_4. The solvent was evaporated under reduced pressure, and the product **1b** was isolated by chromatography on a silica gel column in 71% yield.



1i

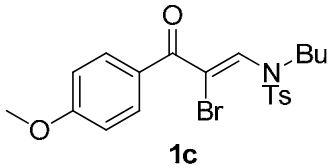
N-(2-bromo-3-oxo-3-phenylprop-1-enyl)-N-(4-methoxyphenyl)acetamide (1i). Column chromatography on silica gel (eluent: petroleum ether / ethyl acetate = 5:1) afforded the title product in 74% isolated yield as a yellow solid. M.p. 109-110 ^{o}C. It was obtained as a mixture of Z/E isomers. Major isomer: ^{1}H NMR (400 MHz, $CDCl_3$,

Me_4Si) δ 1.98 (s, 3H), 3.86 (s, 3H), 6.95-7.00 (m, 2H), 7.16-7.20 (m, 2H), 7.47-7.56 (m, 3H), 7.66-7.69 (m, 2H), 8.64 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, Me_4Si) δ 23.09, 55.37, 107.31, 114.46, 128.25, 129.01, 130.31, 130.70, 131.72, 137.51, 141.04, 159.95, 171.50, 191.37; Minor isomer: ^1H NMR (400 MHz, $CDCl_3$, Me_4Si) δ 1.81 (s, 3H), 3.65 (s, 3H), 6.45-6.49 (m, 2H), 6.73-6.77 (m, 2H), 7.44-7.48 (m, 3H), 7.59-7.61 (m, 2H), 8.03 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, Me_4Si) δ 22.21, 55.18, 101.45, 114.13, 127.95, 129.42, 130.45, 130.67, 131.15, 133.47, 133.93, 159.41, 169.58, 188.90; HRMS calcd for $C_{18}H_{16}NO_3Br$ 373.0314, found 373.0307.



1b

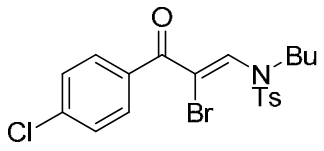
(Z)-N-(2-bromo-3-oxo-3-phenylprop-1-enyl)-N-butyl-4-methylbenzenesulfonamide (1b). Column chromatography on silica gel (eluent: petroleum ether / ethyl acetate = 5:1) afforded the title product in 71% isolated yield as a white solid. M.p. 67-68 ^oC. ^1H NMR (400 MHz, $CDCl_3$, Me_4Si) δ 0.92 (t, J = 7.2 Hz, 3H), 1.29-1.38 (m, 2H), 1.62-1.69 (m, 2H), 2.44 (s, 3H), 3.95-3.99 (m, 2H), 7.33 (d, J = 8.0 Hz, 2H), 7.44-7.48 (m ,2H), 7.53-7.59 (m, 5H), 8.20 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, Me_4Si) δ 13.55, 19.35, 21.54, 31.80, 46.16, 102.94, 127.16, 128.34, 128.84, 130.14, 131.72, 134.83, 137.53, 142.82, 145.22, 190.63; HRMS calcd for $C_{20}H_{22}NO_3SBr$ 435.0504, found 435.0507.



1c

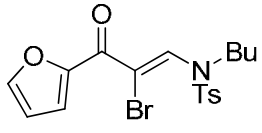
(Z)-N-(2-bromo-3-(4-methoxyphenyl)-3-oxoprop-1-enyl)-N-butyl-4-methylbenzenesulfonamide (1c). Column chromatography on silica gel (eluent: dichloromethane / petroleum ether = 5:1) afforded the title product in 72% isolated yield as a white oil. ^1H NMR (400 MHz, $CDCl_3$, Me_4Si) δ 0.92 (t, J = 7.2 Hz, 3H), 1.29-1.38 (m, 2H),

1.60-1.68 (m, 2H), 2.44 (s, 3H), 3.87 (s, 3H), 3.92-3.96 (m, 2H), 6.93-6.97 (m, 2H), 7.34 (d, J = 8.0 Hz, 2H), 7.61-7.64 (m, 4H), 8.13 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, Me_4Si) δ 13.49, 19.29, 21.45, 31.51, 46.17, 55.31, 103.61, 113.58, 127.06, 129.35, 130.04, 131.45, 134.95, 140.77, 145.02, 162.73, 189.38; HRMS calcd for $C_{21}H_{24}NO_4SBr$ 465.0609, found 465.0615.



1d

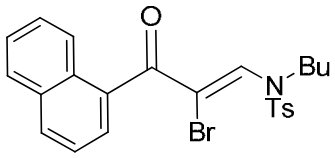
(Z)-N-(2-bromo-3-(4-chlorophenyl)-3-oxoprop-1-enyl)-N-butyl-4-methylbenzenesulfonamide (1d) (the Z-configuration was confirmed by NOESY spectrographic analysis, see S34). Column chromatography on silica gel (eluent: petroleum ether / ethyl acetate = 5:1) afforded the title product in 65% isolated yield as a white solid. M.p. 80-82 ^{o}C. It was obtained as a mixture of Z/E isomers in the ratio of 100:4. ^{1}H NMR (400 MHz, $CDCl_3$, Me_4Si) δ 0.91 (t, J = 7.2 Hz, 3H), 1.30-1.36 (m, 2H), 1.61-1.67 (m, 2H), 2.44 (s, 3H), 3.94-3.98 (m, 2H), 7.33-7.36 (m, 2H), 7.42-7.45 (m, 2H), 7.51-7.55 (m, 2H), 7.58-7.61 (m, 2H), 8.19 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, Me_4Si) δ 13.47, 19.25, 21.47, 31.75, 46.03, 101.90, 127.11, 128.57, 130.12, 130.25, 134.51, 135.72, 137.92, 142.47, 145.31, 189.31; HRMS calcd for $C_{20}H_{21}NO_3SClBr$ 469.0114, found 469.0103.



1e

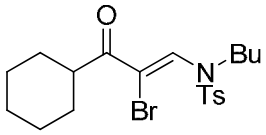
(Z)-N-(2-bromo-3-(furan-2-yl)-3-oxoprop-1-enyl)-N-butyl-4-methylbenzenesulfonamide (1e). Column chromatography on silica gel (eluent: petroleum ether / ethyl acetate = 5:1) afforded the title product in 78% isolated yield as a yellow oil. It was obtained as a mixture of Z/E isomers in the ratio of 100:3. ^{1}H NMR (400 MHz, $CDCl_3$, Me_4Si) δ 0.91 (t, J = 7.2 Hz, 3H), 1.25-1.36 (m, 2H), 1.61-1.69 (m, 2H), 2.44 (s, 3H),

3.96 (t, *J* = 8.0 Hz, 2H), 6.58-6.60 (m, 1H), 7.19 (d, *J* = 3.6 Hz, 1H), 7.39 (d, *J* = 8.0 Hz, 2H), 7.69 (d, *J* = 0.8 Hz, 1H), 7.74 (d, *J* = 8.4 Hz, 2H), 8.82 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, Me_4Si) δ 13.33, 19.12, 21.29, 31.44, 45.98, 101.17, 111.98, 119.33, 126.98, 129.98, 134.67, 140.32 145.02, 146.37, 150.12, 175.69; HRMS calcd for $C_{18}H_{20}NO_4SBr$ 425.0296, found 425.0295.



1f

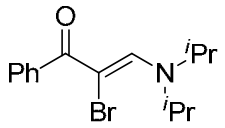
(Z)-N-(2-bromo-3-(naphthalen-1-yl)-3-oxoprop-1-enyl)-N-butyl-4-methylbenzene sulfonamide (1f). Column chromatography on silica gel (eluent: petroleum ether / ethyl acetate = 5:1) afforded the title product in 77% isolated yield as a white solid. M.p. 124 ^{o}C. It was obtained as a mixture of Z/E isomers in the ratio of 100:3. ^{1}H NMR (400 MHz, $CDCl_3$, Me_4Si) δ 0.88 (t, *J* = 7.2 Hz, 3H), 1.27-1.32 (m, 2H), 1.60-1.66 (m, 2H), 2.26 (s, 3H), 3.95 (t, *J* = 8.0 Hz ,2H), 7.05 (d, *J* = 8.0 Hz, 2H), 7.14 (d, *J* = 8.0 Hz, 2H), 7.41-7.50 (m, 4H), 7.76 (d, *J* = 8.0 Hz, 1H), 7.85 (d, *J* = 8.0 Hz, 1H), 7.91 (d, *J* = 8.0 Hz,1H), 8.16 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, Me_4Si) δ 13.32, 19.05, 21.13, 31.88, 45.71, 103.03, 124.31, 124.77, 125.28, 126.10, 126.60, 126.72, 128.17, 129.75, 130.13, 130.16, 133.09, 133.88, 135.87, 144.71, 144.88, 191.13; HRMS calcd for $C_{24}H_{24}NO_3SBr$ 485.0660, found 485.0664.



1g

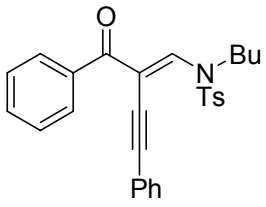
(Z)-N-(2-bromo-3-cyclohexyl-3-oxoprop-1-enyl)-N-butyl-4-methylbenzenesulfonamide (1g). Column chromatography on silica gel (eluent: petroleum ether / ethyl acetate = 20:1) afforded the title product in 77% isolated yield as a white oil. ^{1}H NMR (300 MHz, $CDCl_3$, Me_4Si) δ 0.89 (t, *J* = 7.2 Hz, 3H), 1.20-1.83 (m, 14H), 2.45 (s, 3H), 3.04-3.11 (m, 1H), 3.90 (t, *J* = 7.8 Hz, 2H), 7.38 (d, *J* = 8.1 Hz, 2H), 7.73 (d, *J* = 8.4

Hz, 2H), 8.50 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, Me_4Si) δ 13.56, 19.37, 21.56, 25.69, 25.72, 29.67, 31.59, 45.66, 46.13, 102.47, 130.16, 135.20, 137.51, 145.06, 197.25; HRMS calcd for $C_{20}H_{28}NO_3SBr$ 441.0973, found 441.0980.



1h

(Z)-2-bromo-3-(diisopropylamino)-1-phenylprop-2-en-1-one (1h). Column chromatography on silica gel (eluent: petroleum ether / ethyl acetate = 3:1) afforded the title product in 72% isolated yield as a white solid. M.p. 110-111 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$, Me_4Si) δ 1.12 (s, 12H), 3.61 (br, 1H), 5.56 (br, 1H), 7.36-7.44 (m, 5H), 7.46 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, Me_4Si) δ 22.67 (br), 47.59 (br), 91.42, 127.70, 127.79, 129.40, 140.67, 148.87, 189.66; HRMS calcd for $C_{15}H_{20}NOBr$ 309.0728, found 309.0725.

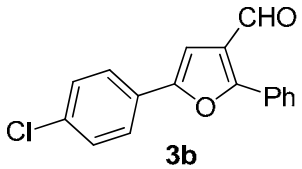


4a

(E)-N-(2-benzoyl-4-phenylbut-1-en-3-ynyl)-N-butyl-4-methylbenzenesulfonamide (4a). Column chromatography on silica gel (eluent: petroleum ether / dichloromethane = 1:1) afforded the title product in 85% isolated yield as a yellow solid. M.p. 94-95 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$, Me_4Si) δ 0.83 (t, J = 7.6 Hz, 3H), 1.25-1.30 (m, 2H), 1.73-1.77 (m, 2H), 2.40 (s, 3H), 4.06 (t, J = 8.0 Hz, 2H), 7.14-7.21 (m, 5H), 7.31 (d, J = 8.4 Hz, 2H), 7.37-7.41 (m, 2H), 7.45-7.47 (m, 1H), 7.68-7.71 (m, 2H), 7.77-7.79 (m, 2H), 8.17 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, Me_4Si) δ 13.64, 19.48, 21.54, 31.90, 46.39, 84.40, 97.73, 101.00, 122.89, 127.27, 127.76, 128.22, 129.00, 130.16, 130.55, 131.67, 135.01, 138.03, 143.45, 145.10, 193.15; HRMS calcd for $C_{28}H_{27}NO_3S$ 457.1712, found 457.1709.

A typical procedure for the Pd-catalyzed one-pot synthesis of 2,5-diphenylfuran-3-carbaldehyde (3a).

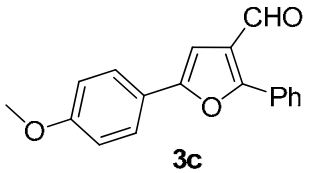
A solution of (Z)-N-(2-bromo-3-oxo-3-phenylprop-1-enyl)-N-butyl-4 -methylbenzenesulfonamide (**1b**) (218.2 mg, 0.5 mmol) in Et_3N(5ML) was added H_2O (0.045 ml, 2.5 mmol), phenylacetylene **2a** (0.082 ml, 0.75 mmol), $Pd(PPh_3)_2Cl_2$ (17.5 mg, 5 mmol %) and CuI (9.5 mg, 10 mmol %) by this sequence. The resulting solution was stirred at 80°C for 14h. The solvent was evaporated under reduced pressure and the residue was diluted with EtOAc and washed with satd. aq. NH_4Cl. The organic layer was separated, and the aqueous layer was extracted with EtOAc. The combined organic layers were dried over Na_2SO_4. The solvent was evaporated under reduced pressure, and the product was isolated by chromatography on a silica gel column to give the yellow solid product **2,5-diphenylfuran-3-carbaldehyde** (**3a**) 101 mg (81%); M.p. 84-85 °C (lit.[1] 84-85 °C). ^{1}H NMR (400 MHz, $CDCl_3$, Me_4Si) δ 7.07 (s, 1H), 7.28-7.32 (m, 1H), 7.37-7.51 (m, 5H), 7.69-7.77 (m, 4H), 10.10 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, Me_4Si) δ 103.85, 124.06, 124.63, 127.80, 128.33, 128.69, 128.73, 128.89, 129.18, 130.00, 153.79, 160.23, 185.42; HRMS calcd for $C_{17}H_{12}O_2$ 248.0837, found 248.0834.



3b

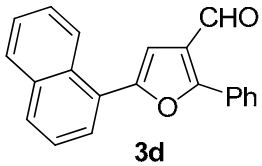
5-(4-chlorophenyl)-2-phenylfuran-3-carbaldehyde (3b). Column chromatography on silica gel (eluent: petroleum ether / ethyl acetate = 20:1) afforded the title product in 76% isolated yield as a yellow solid. M.p. 90-91 °C. ^{1}H NMR (400 MHz, $CDCl_3$, Me_4Si) δ 7.07 (s, 1H), 7.36 (d, J = 8.4 Hz, 2H), 7.49-7.53 (m, 3H), 7.62 (d, J = 8.4 Hz, 2H), 7.76-7.78 (m, 2H), 10.11 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, Me_4Si) δ 104.34,

124.68, 125.31, 127.72, 127.89, 128.59, 129.00, 130.24, 134.15, 152.75, 160.53, 185.37; HRMS calcd for $C_{17}H_{11}O_2Cl$ 282.0448, found 282.0443.



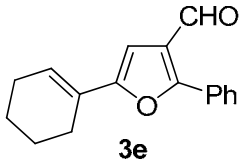
3c

5-(4-methoxyphenyl)-2-phenylfuran-3-carbaldehyde (3c). Column chromatography on silica gel (eluent: petroleum ether / ethyl acetate = 20:1) afforded the title product in 83% isolated yield as a yellow solid. M.p. 77-78 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$, Me_4Si) δ 3.78 (s, 3H), 6.88-6.91 (m, 3H), 7.43-7.49 (m, 3H), 7.59-7.61 (m, 2H), 7.72-7.75 (m, 2H), 10.08 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, Me_4Si) δ 55.08, 102.06, 114.07, 121.99, 124.65, 125.50, 127.66, 128.78, 128.80, 129.77, 153.87, 159.65, 159.68, 185.46; HRMS calcd for $C_{18}H_{14}O_3$: 278.0943, found 278.0944.



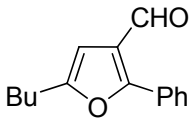
3d

5-(naphthalen-1-yl)-2-phenylfuran-3-carbaldehyde (3d). Column chromatography on silica gel (eluent: petroleum ether / ethyl acetate = 20:1) afforded the title product in 73% isolated yield as a yellow solid. M.p. 99 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$, Me_4Si) δ 7.18 (s, 1H), 7.43-7.54 (m, 6H), 7.76-7.86 (m, 5H), 8.37 (d, J = 8.4 Hz, 1H), 10.18 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, Me_4Si) δ 108.20, 124.41, 124.82, 125.09, 126.06, 126.49, 126.72, 126.92, 127.89, 128.60, 128.73, 128.95, 129.44, 129.97, 130.08, 133.74, 153.28, 160.69, 185.57; HRMS calcd for $C_{21}H_{14}O_2$ 298.0994, found 298.0989.



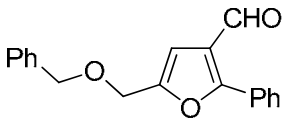
3e

5-cyclohexenyl-2-phenylfuran-3-carbaldehyde (3e). Column chromatography on silica gel (eluent: petroleum ether / ethyl acetate = 20:1) afforded the title product in 69% isolated yield as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$, Me_4Si) δ 1.65-1.76 (m, 4H), 2.22-2.28 (m, 4H), 6.46 (s, 1H), 6.60 (s, 1H), 7.46-7.47 (m, 3H), 7.72 (d, *J* = 4.0 Hz, 2H), 10.07 (s, 1H), ^{13}C NMR (100 MHz, $CDCl_3$, Me_4Si) δ 21.87, 22.03, 24.52, 25.10, 102.25, 124.16, 125.08, 126.13, 127.79, 128.82, 128.92, 129.80, 155.32, 159.84, 185.69; HRMS calcd for $C_{17}H_{16}O_2$ 252.1150, found 252.1147.



3f

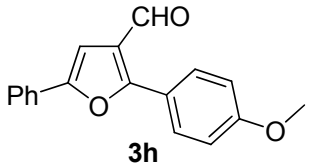
5-butyl-2-phenylfuran-3-carbaldehyde (3f). Column chromatography on silica gel (eluent: petroleum ether / ethyl acetate = 20:1) afforded the title product in 63% isolated yield as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$, Me_4Si) δ 0.95 (t, *J* = 7.2 Hz, 3H), 1.37-1.46 (m, 2H), 1.64-1.72 (m, 2H), 2.68 (t, *J* = 7.6 Hz, 2H), 6.50 (s, 1H), 7.44-7.50 (m, 3H), 7.69-7.71 (m, 2H), 10.06 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, Me_4Si) δ 13.68, 22.08, 27.34, 29.58, 104.14, 123.74, 127.78, 128.84, 129.13, 129.69, 157.21, 160.21, 185.77; HRMS calcd for $C_{15}H_{16}O_2$ 228.1150, found 228.1152.



3g

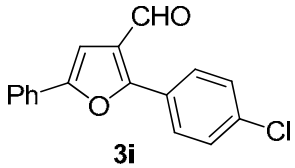
5-(benzyloxymethyl)-2-phenylfuran-3-carbaldehyde (3g). Column chromatography on silica gel (eluent: petroleum ether / ethyl acetate = 20:1) afforded the title product in 35% isolated yield as a oil. ^{1}H NMR (400 MHz, $CDCl_3$, Me_4Si) δ 4.55 (s, 2H), 4.61 (s, 2H), 6.83 (s, 1H), 7.28-7.38 (m, 5H), 7.48-7.52 (m, 3H), 7.73-7.76 (m, 2H), 10.09 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, Me_4Si) δ 63.47, 72.14, 108.67, 123.46, 127.82,

128.07, 128.41, 128.67, 128.91, 130.18, 137.37, 152.08, 161.66, 185.57; HRMS calcd for $C_{19}H_{16}O_3$ 292.1099. 292.1107.



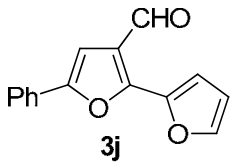
3h

2-(4-methoxyphenyl)-5-phenylfuran-3-carbaldehyde (3h). Column chromatography on silica gel (eluent: petroleum ether / ethyl acetate = 20:1) afforded the title product in 80% isolated yield as a yellow solid. M.p. 105-107 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$, Me_4Si) δ 3.82 (s, 1H), 6.97-7.00 (m, 2H), 7.04 (s, 1H), 7.26-7.31 (m, 1H), 7.36-7.40 (m, 2H), 7.67-7.74 (m, 4H), 10.05 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, Me_4Si) δ 55.25, 103.83, 114.34, 121.36, 123.61, 123.93, 128.13, 128.65, 129.29, 129.33, 153.13, 160.53, 161.08, 185.37; HRMS calcd for $C_{18}H_{14}O_3$ 278.0943, found 278.0949. Anal. Calcd for $C_{18}H_{14}O_3$: C, 77.68; H, 5.07. Found: C, 77.71; H, 5.04.



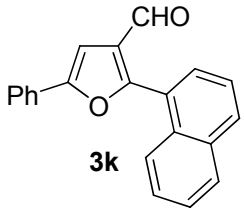
3i

2-(4-chlorophenyl)-5-phenylfuran-3-carbaldehyde (3i). Column chromatography on silica gel (eluent: petroleum ether / ethyl acetate = 20:1) afforded the title product in 60% isolated yield as a yellow solid. M.p. 136-137 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$, Me_4Si) δ 7.07 (s, 1H), 7.30-7.35 (m, 1H), 7.39-7.48 (m, 4H), 7.68-7.77 (m, 4H), 10.09 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, Me_4Si) δ 104.36, 124.14, 124,83, 127.24, 128.57, 128.81, 128.90, 129.04, 129.25, 136.24, 154.05, 158.50, 185.05; HRMS calcd for $C_{17}H_{11}O_2Cl$ 282.0448, found 282.0446.



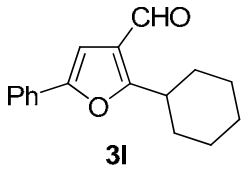
3j

5-phenyl-2,2'-bifuran-3-carbaldehyde (3j). Column chromatography on silica gel (eluent: petroleum ether / ethyl acetate = 20:1) afforded the title product in 83% isolated yield as a solid. M.p. 96 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$, Me_4Si) δ 6.55 (dd, *J* = 3.4, 1.6 Hz, 1H), 6.97 (dd, *J* = 3.6, 0.8 Hz, 1H), 6.99 (s, 1H), 7.26-7.31 (m, 1H), 7.35-7.39 (m, 2H), 7.55 (dd, *J* = 1.8, 0.8 Hz, 1H), 7.62-7.65 (m, 2H), 10.35 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, Me_4Si) δ 103.15, 110.90, 111.91, 123.78, 123.96, 128.34, 128.63, 128.89, 144.51, 145.23, 150.45, 153.56, 185.54; HRMS calcd for $C_{15}H_{10}O_3$ 238.0630, found 238.0626.



3k

2-(naphthalen-1-yl)-5-phenylfuran-3-carbaldehyde (3k). Column chromatography on silica gel (eluent: petroleum ether / ethyl acetate = 20:1) afforded the title product in 78% isolated yield as a solid. M.p. 84-85 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$, Me_4Si) δ 7.22 (s, 1H), 7.30-7.35 (m, 1H), 7.38-7.43 (m, 2H), 7.53-7.58 (m, 3H), 7.65-7.67 (m, 1H), 7.74-7.77 (m, 2H), 7.91-7.95 (m, 1H), 8.00 (d, *J* = 8.4 Hz, 1H), 8.10-8.13 (m, 1H), 9.82 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, Me_4Si) δ 102.75, 124.12, 124.89, 125.17, 125.51, 126.55, 126.60, 127.39, 128.44, 128.47, 128.78, 129.29, 129.84, 130.99, 131.62, 133.69, 154.76, 161.57, 185.91; HRMS calcd for $C_{21}H_{14}O_2$ 298.0994, found 298.0993.



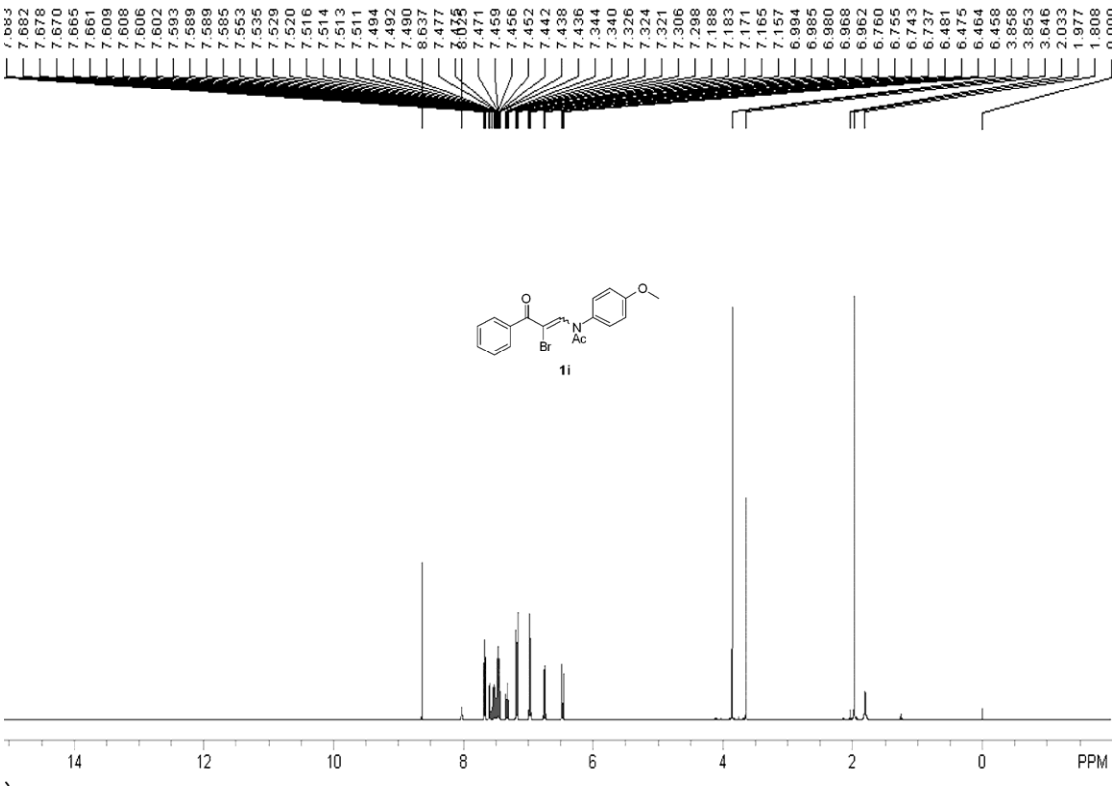
3l

2-cyclohexyl-5-phenylfuran-3-carbaldehyde (3l). Column chromatography on silica

gel (eluent: petroleum ether / ethyl acetate = 20:1) afforded the title product in 41% isolated yield as a solid. M.p. 53-54 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$, Me_4Si) δ 1.29-1.48 (m, 3H), 1.71-1.81 (m, 3H), 1,88-1.98 (m, 4H), 3.20 (tt, *J* = 12.0, 3.6 Hz, 1H), 6.90 (s, 1H), 7.27-7.31 (m, 1H), 7.37-7.41 (m, 2H), 7.64-7.66 (m, 2H), 10.01 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, Me_4Si) δ 25.57, 25.98, 31.69, 36.90, 102.21, 122.83, 123.85, 127.99, 128.70, 129.65, 152.99, 169.29, 184.47; HRMS calcd for $C_{17}H_{18}O_2$ 254.1307, found 254.1304.

References

(1) P. S. Bailey, J. V. Waggoner, G. Nowlin, G. L. Rushton, *J. Am. Chem. Soc.* **1954**, *76*, 2249.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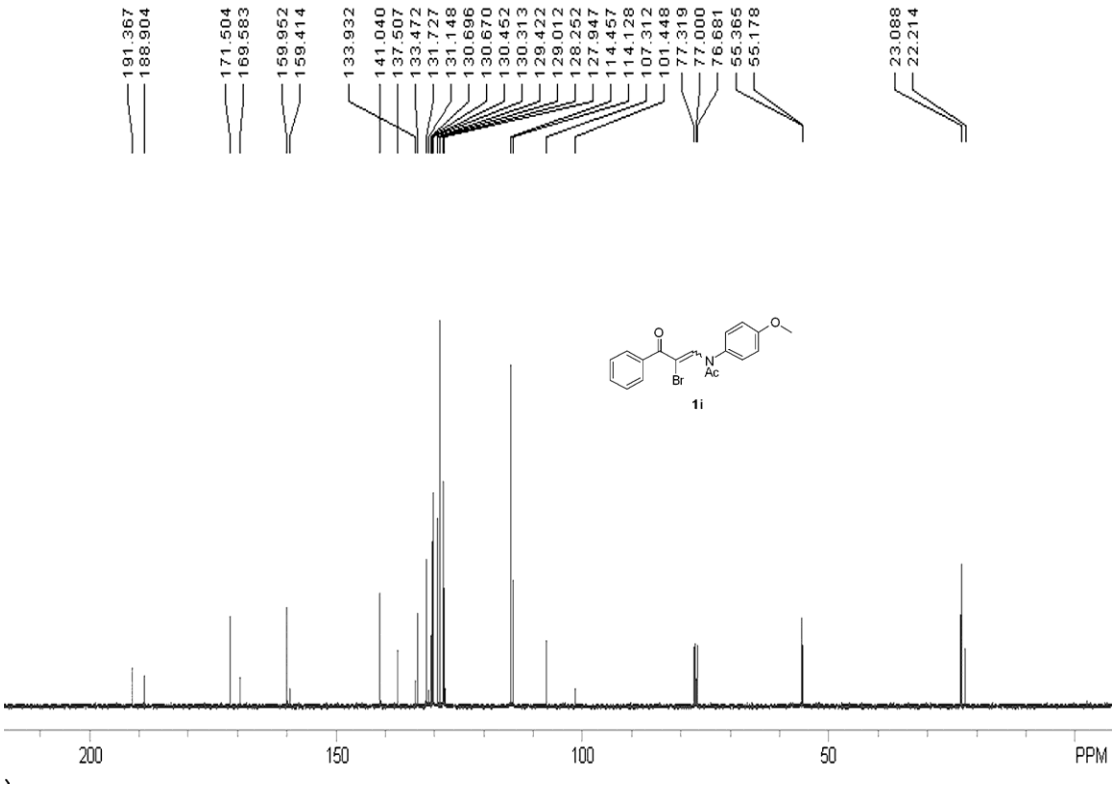
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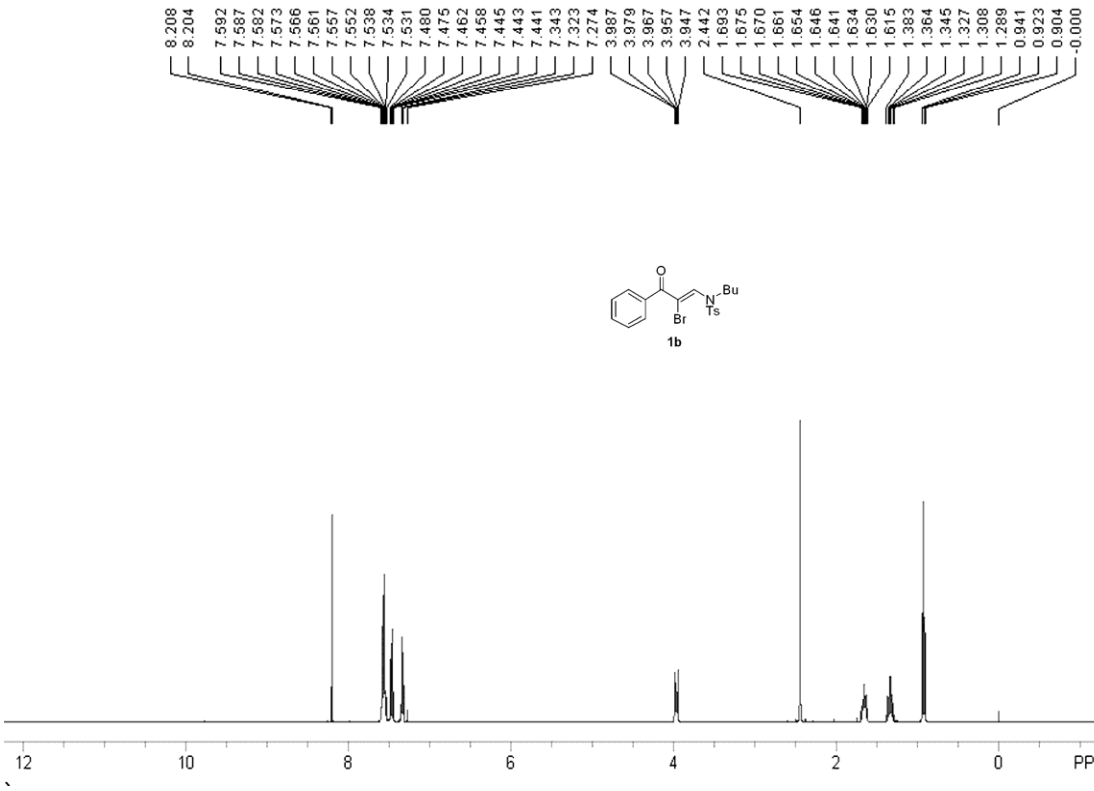
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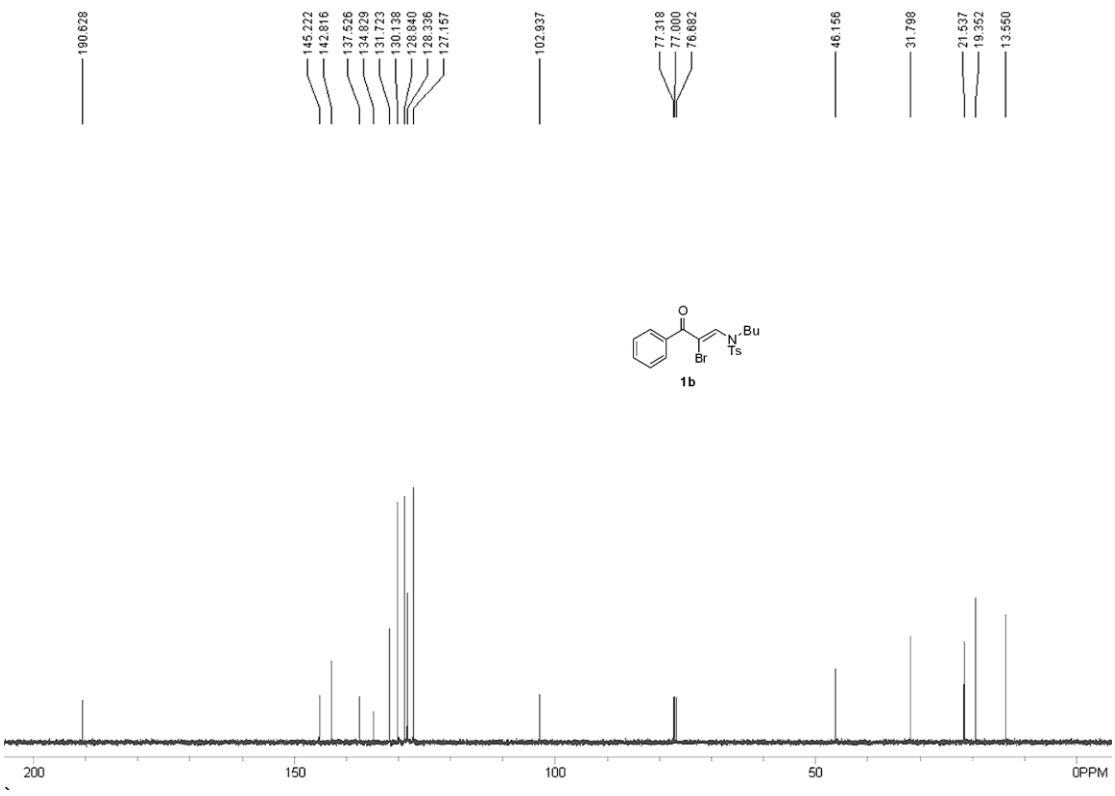
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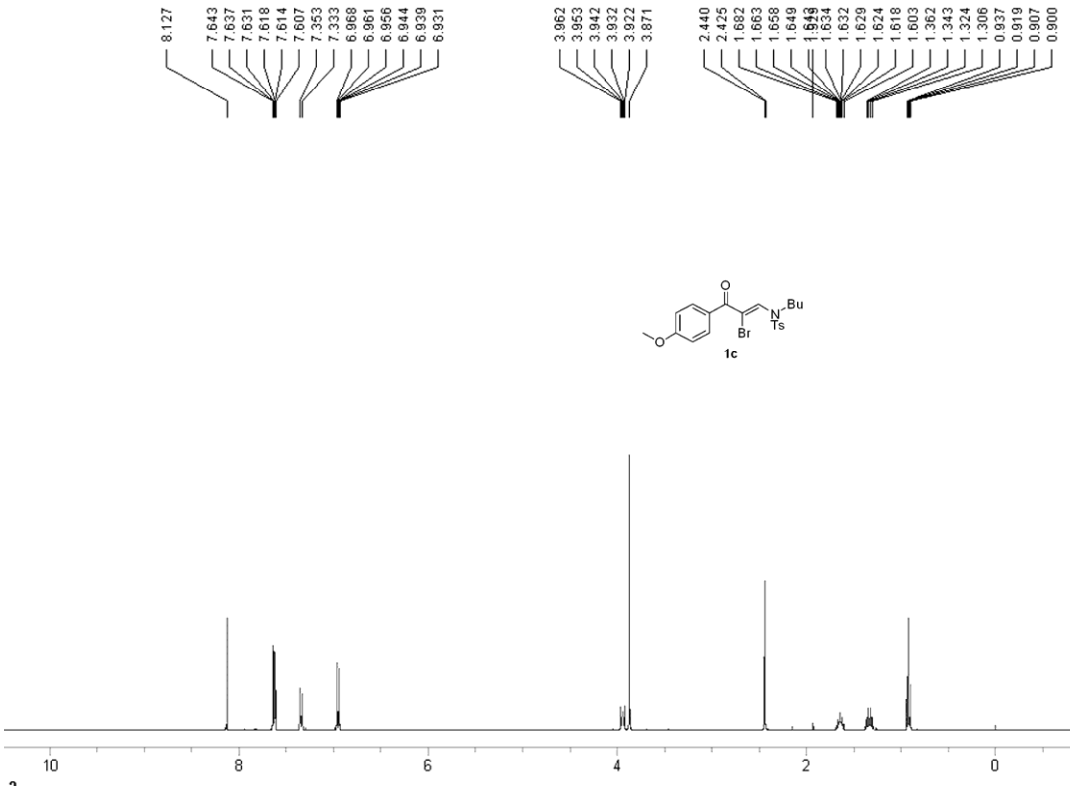
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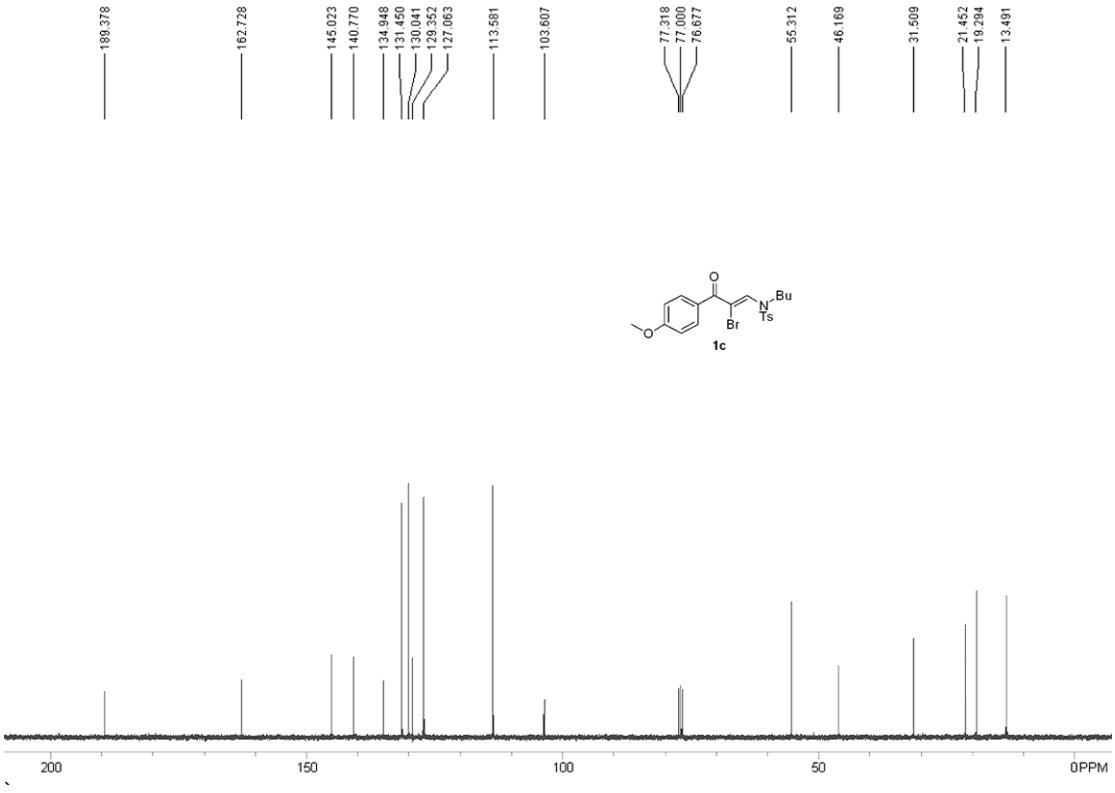
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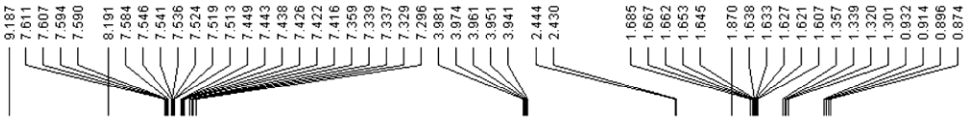
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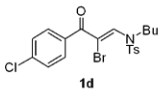
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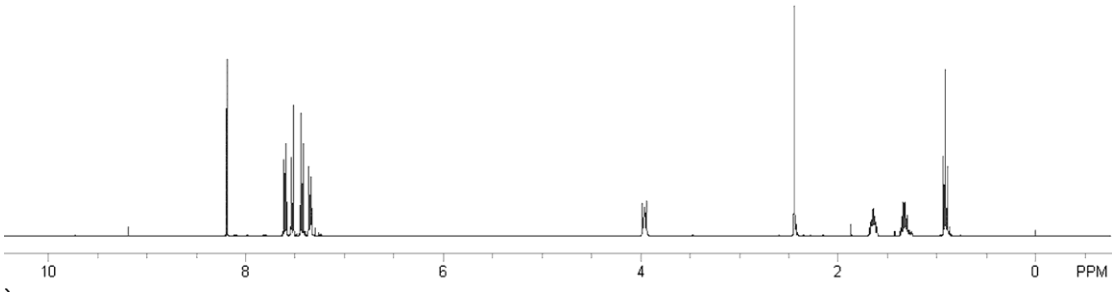
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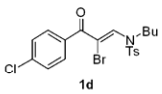
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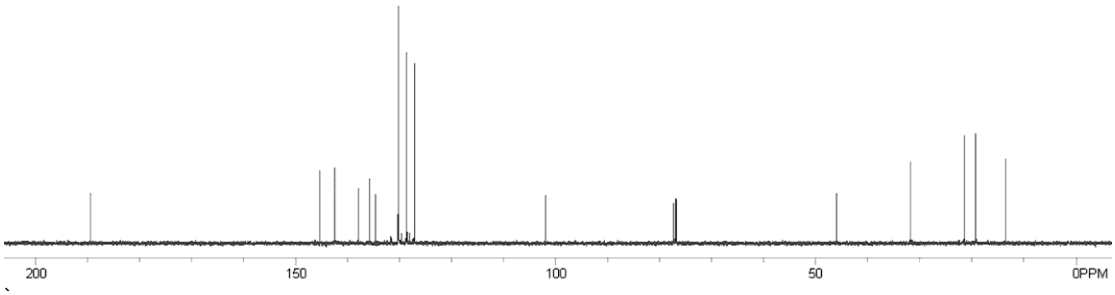
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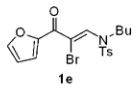
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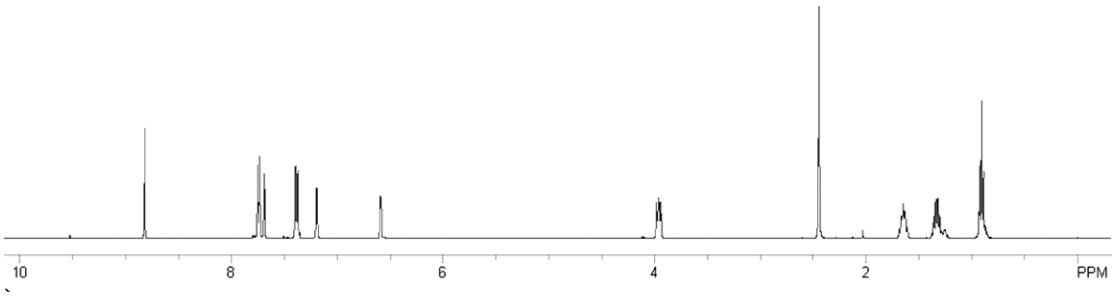
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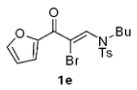
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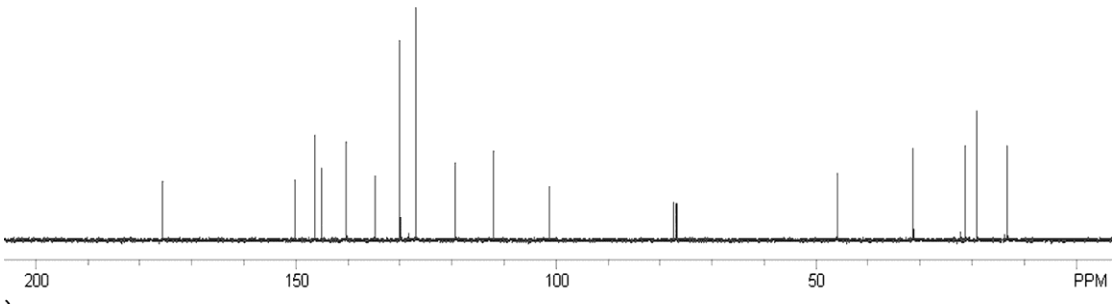
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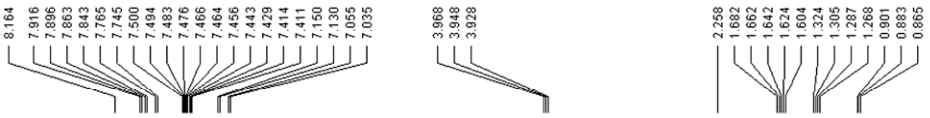
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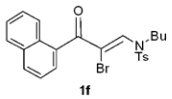
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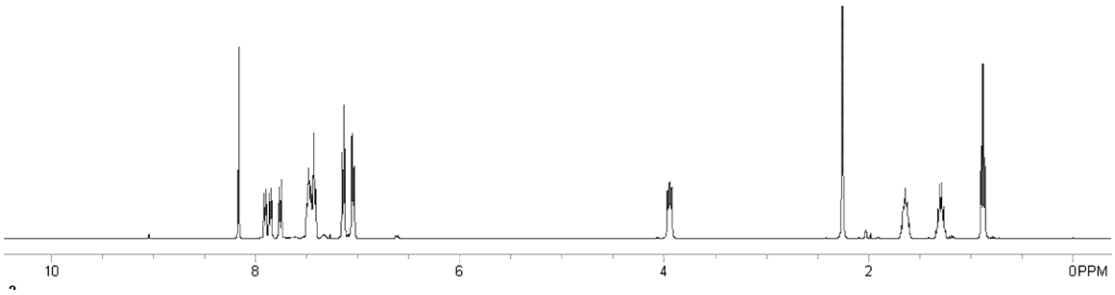
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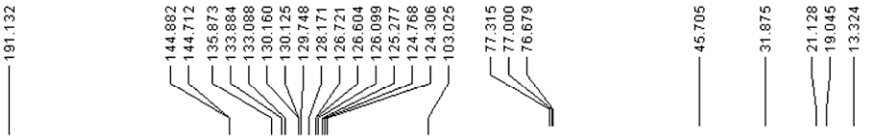
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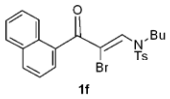
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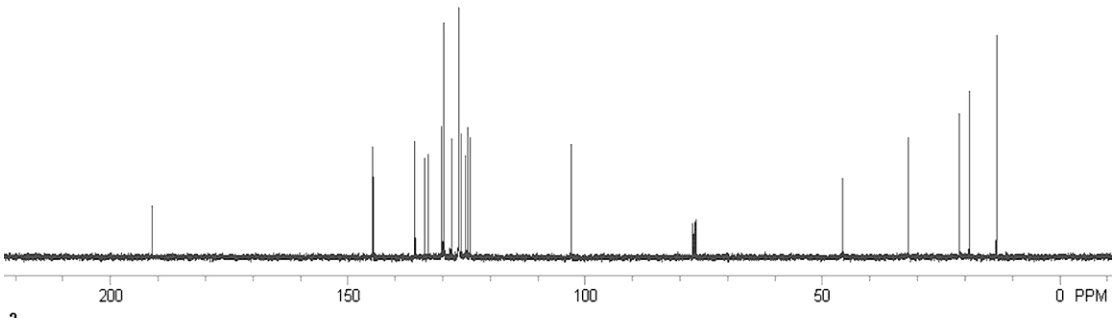
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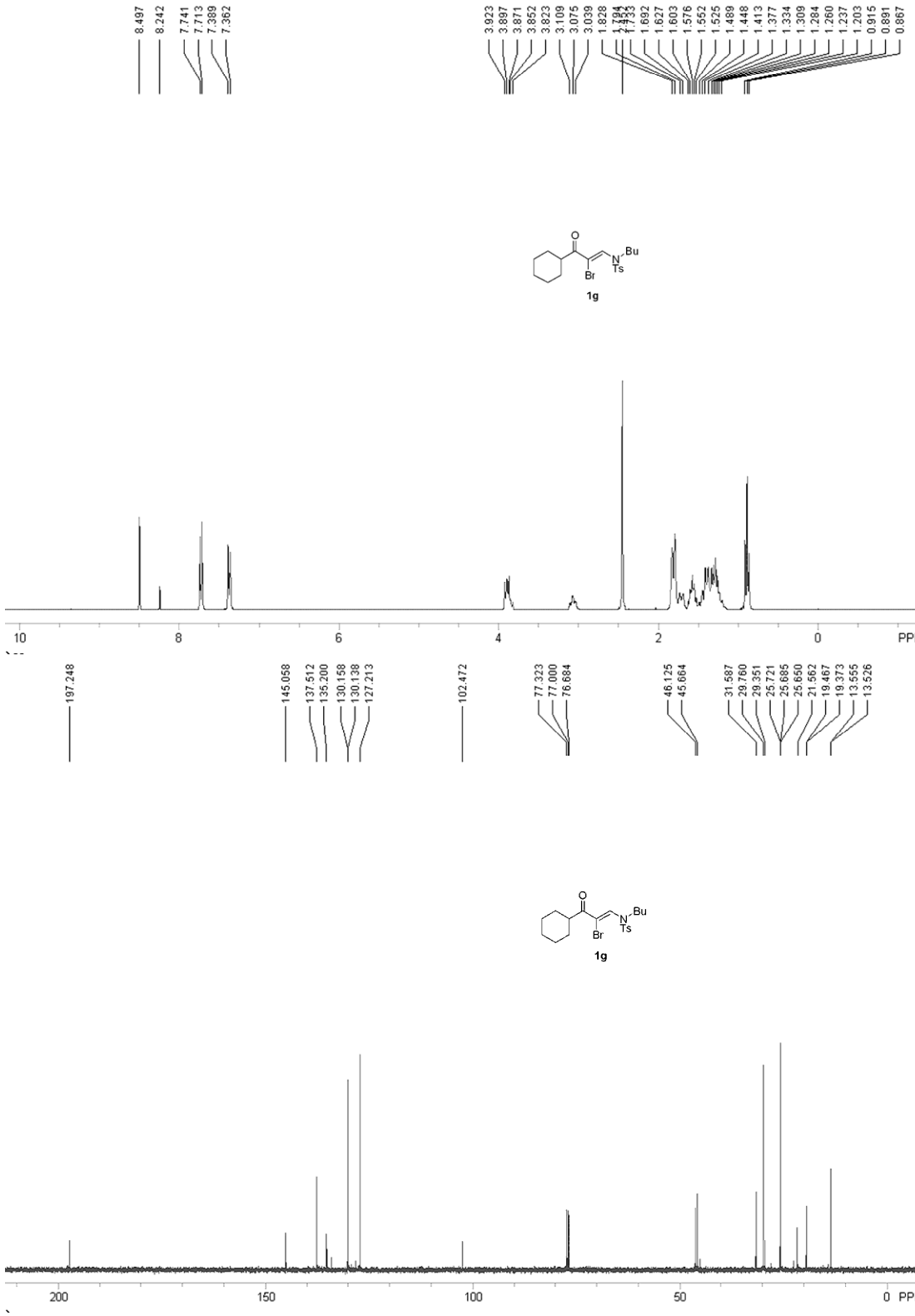
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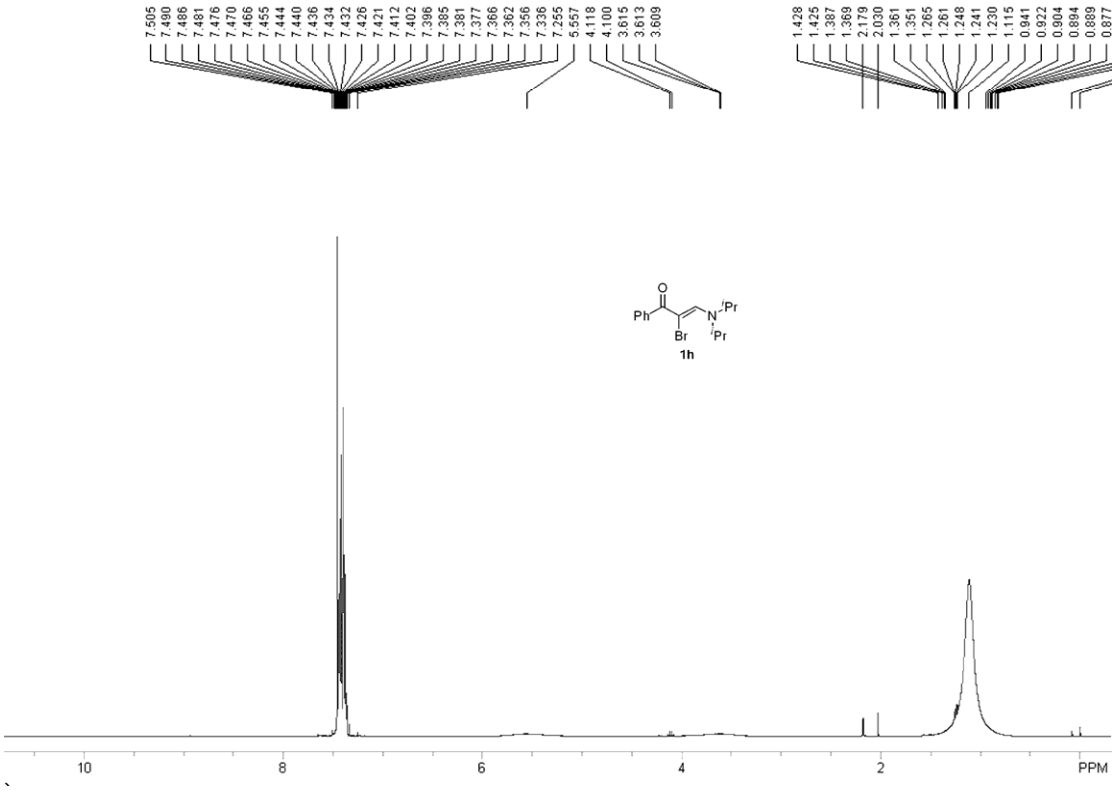
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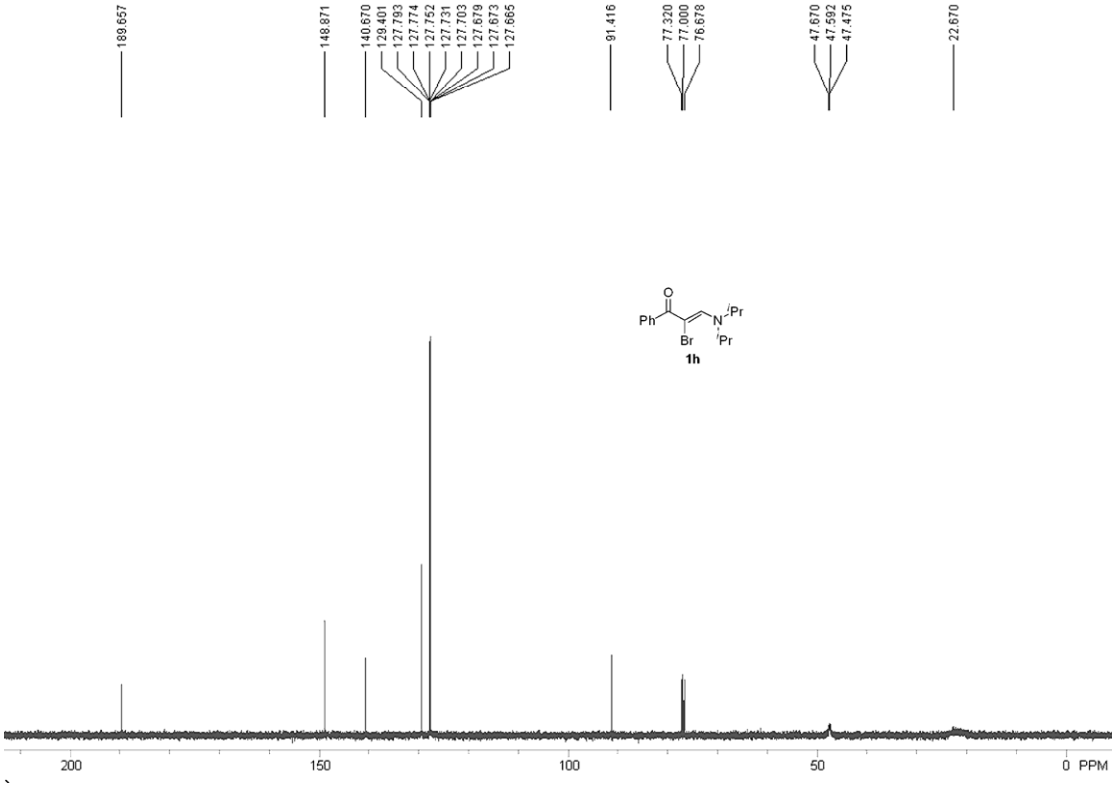
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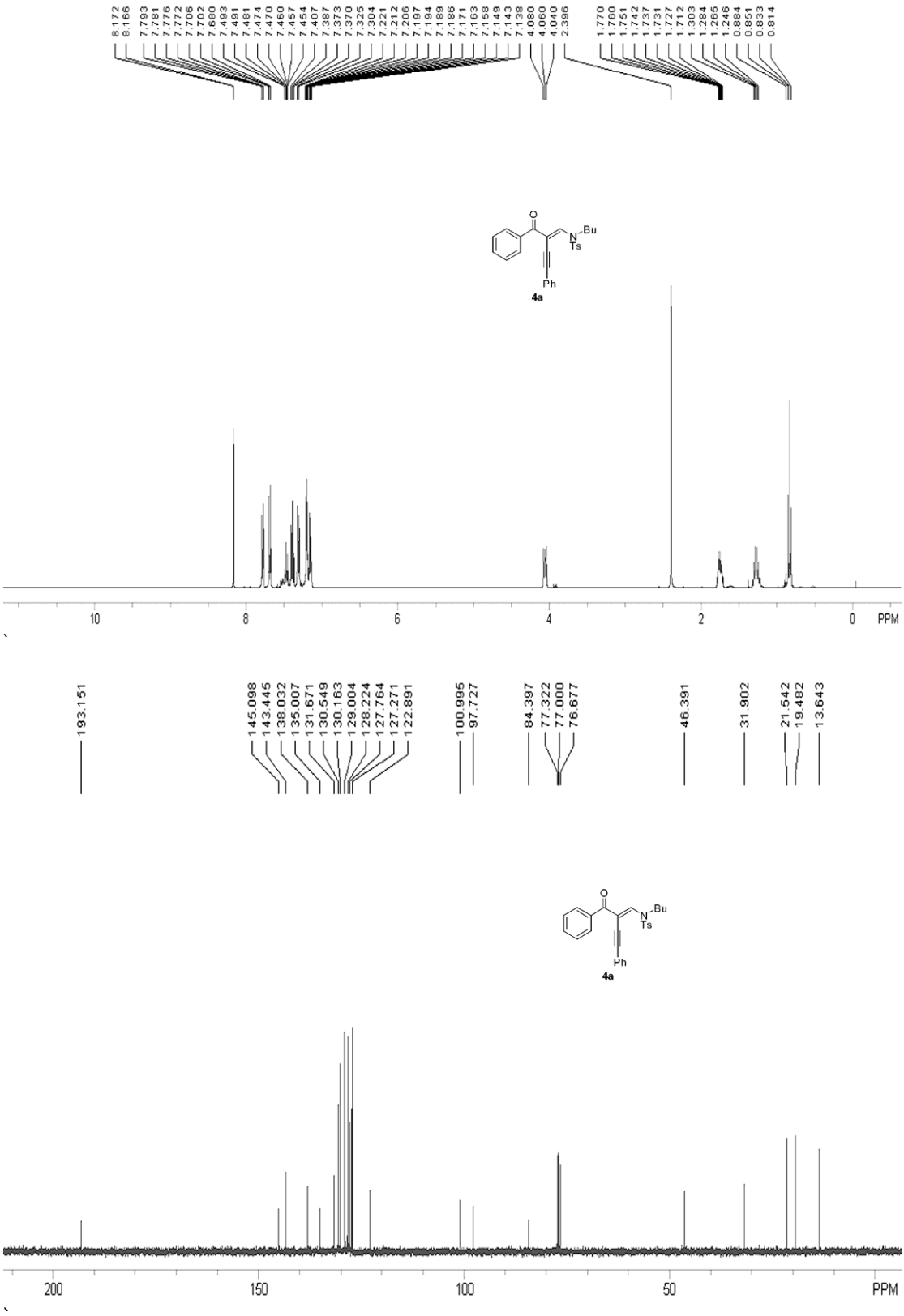
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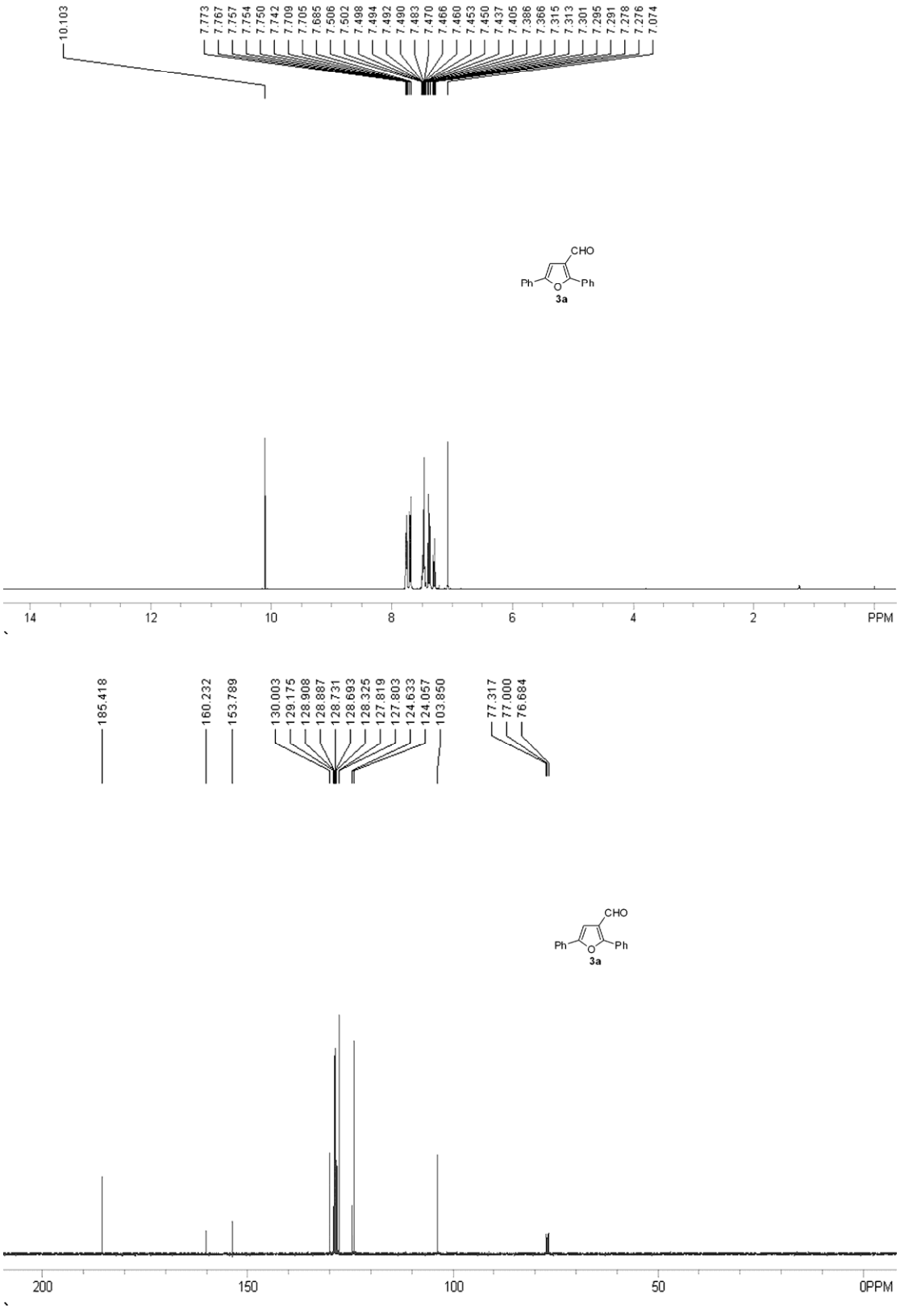
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7.483
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Ph
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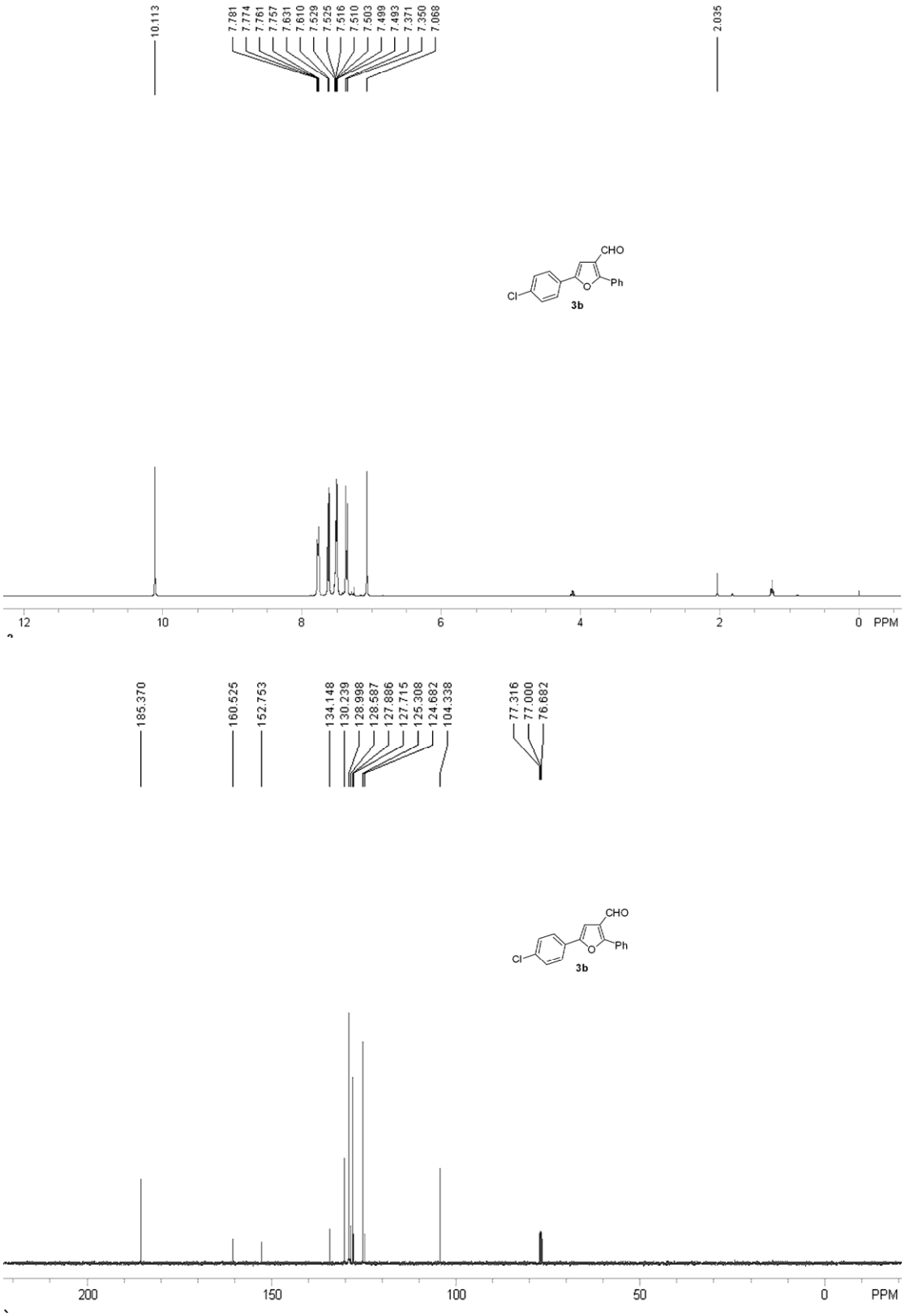
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4
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0 PPM
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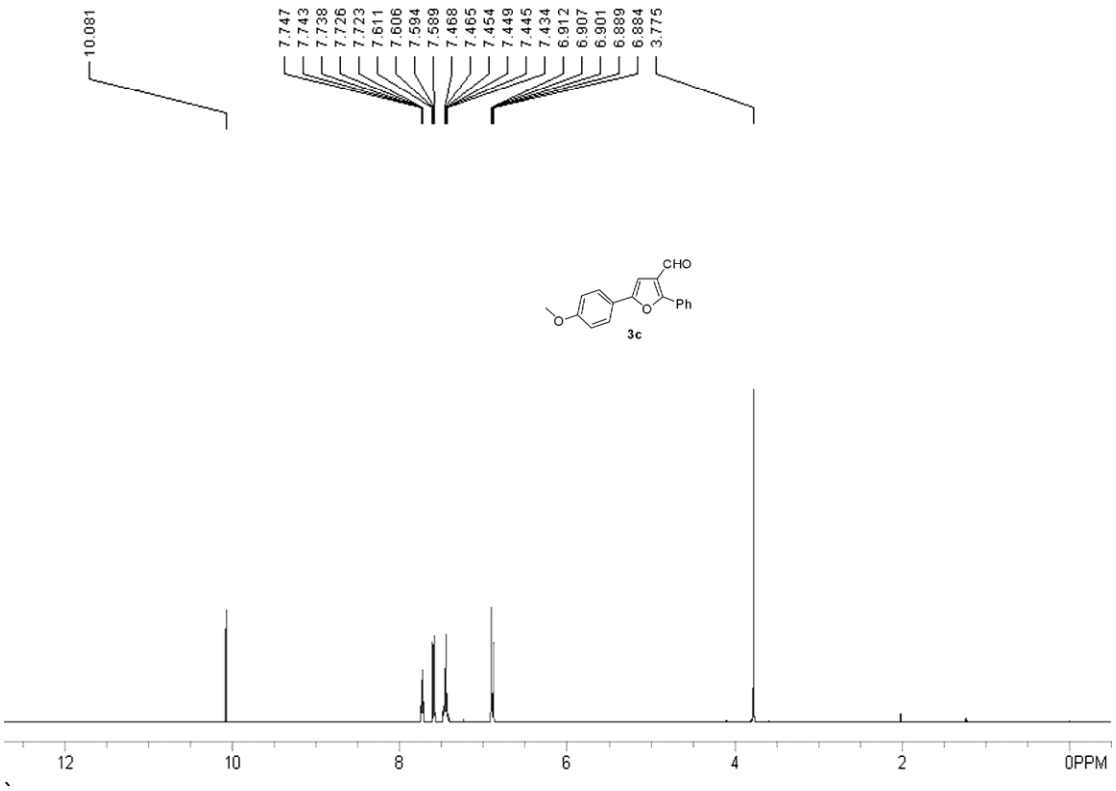
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7.445
7.434
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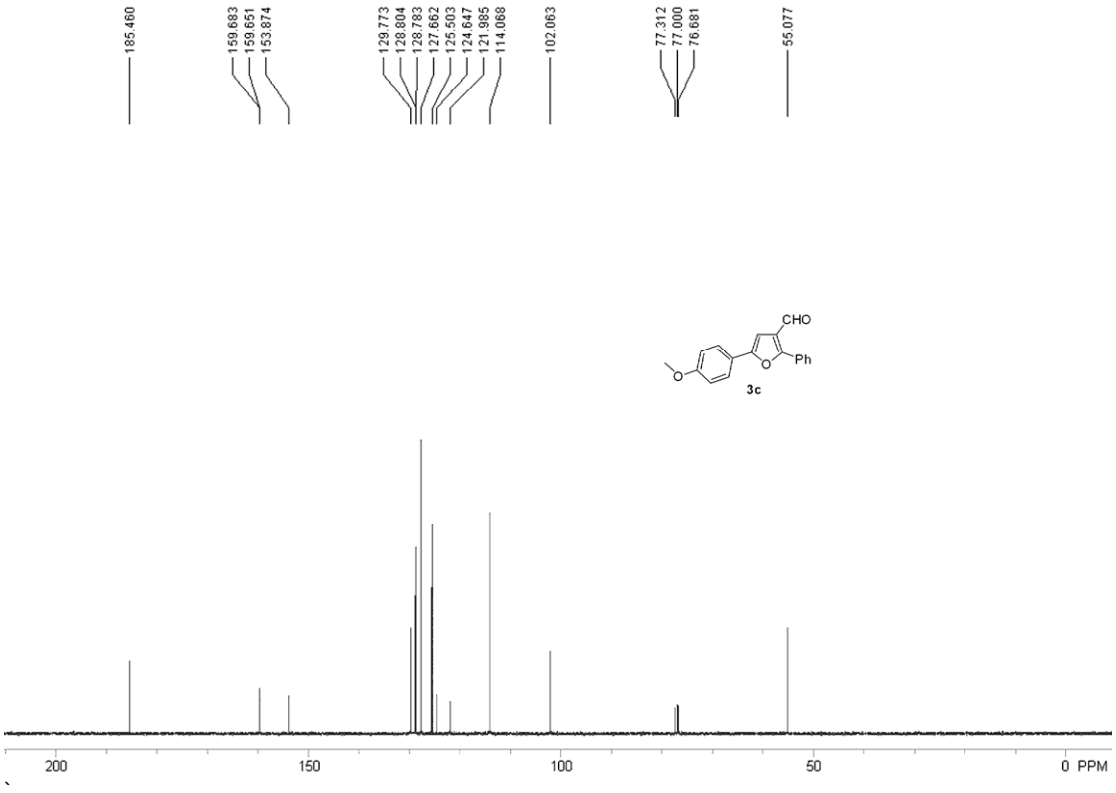
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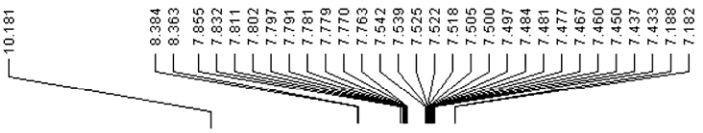
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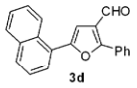
7.182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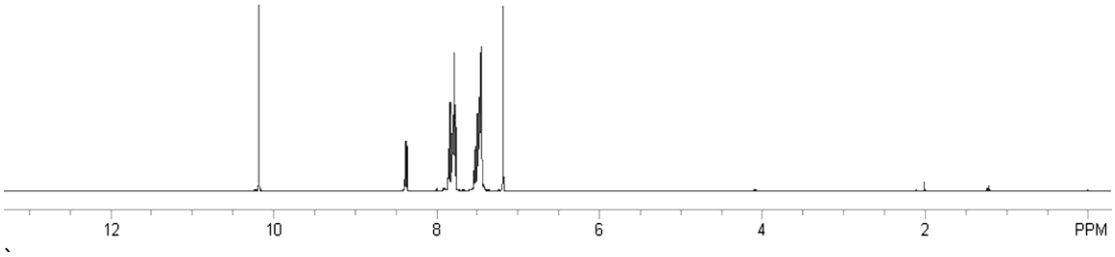
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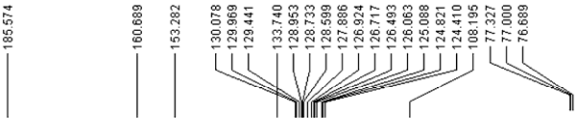
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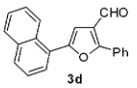
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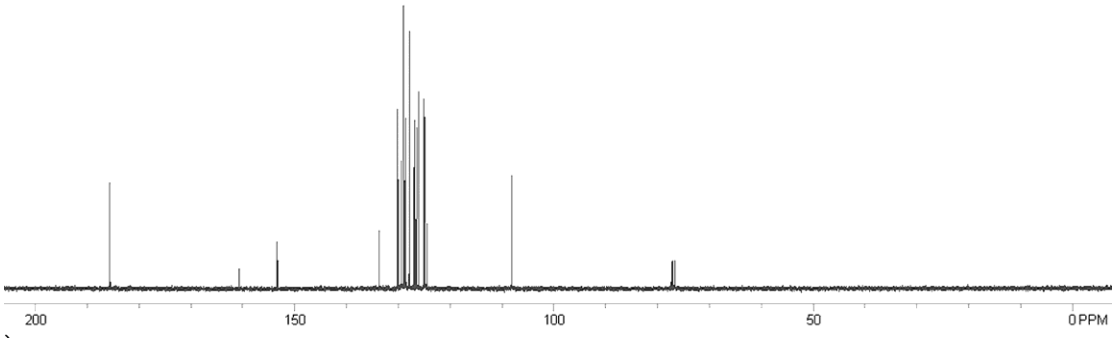
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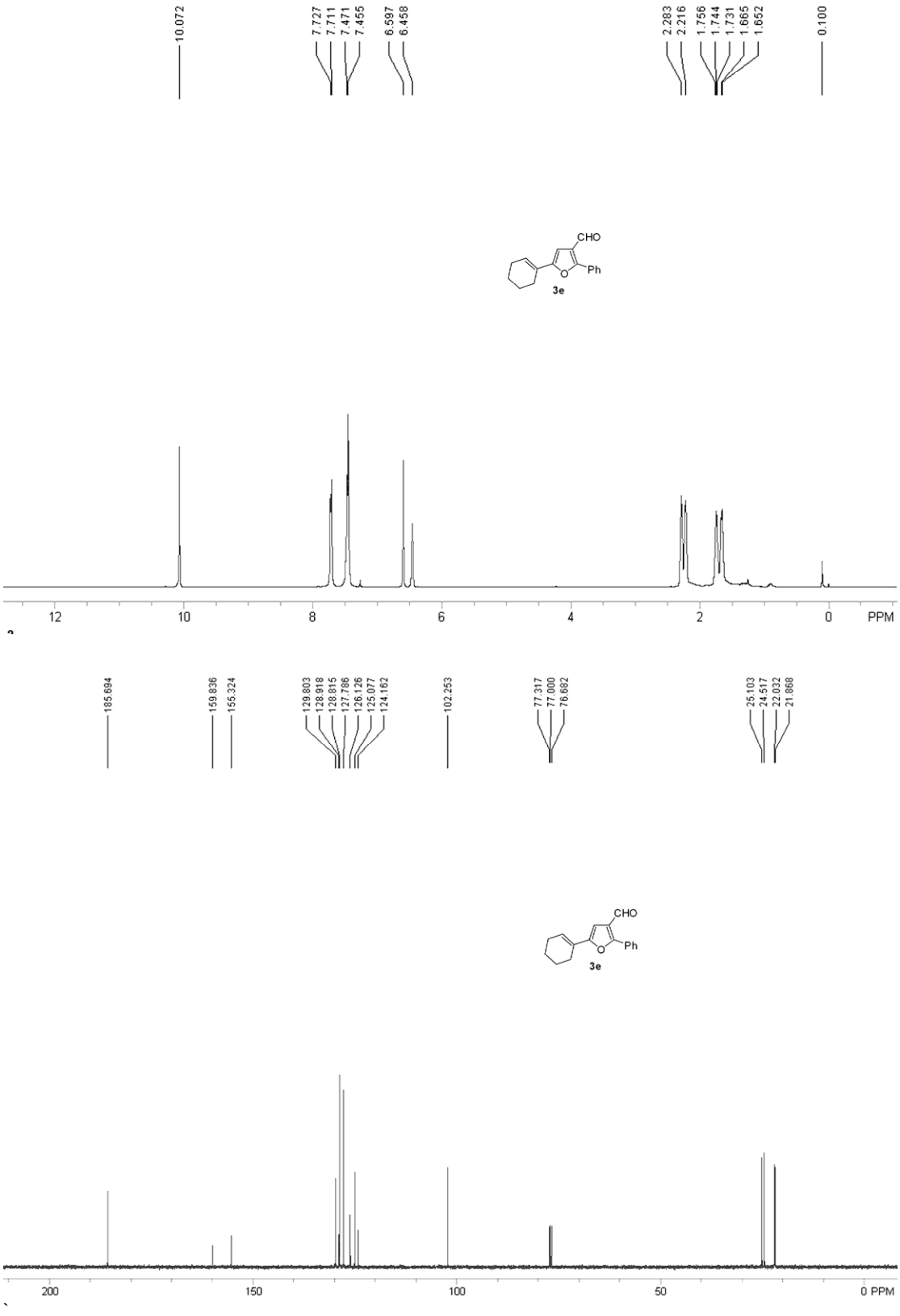
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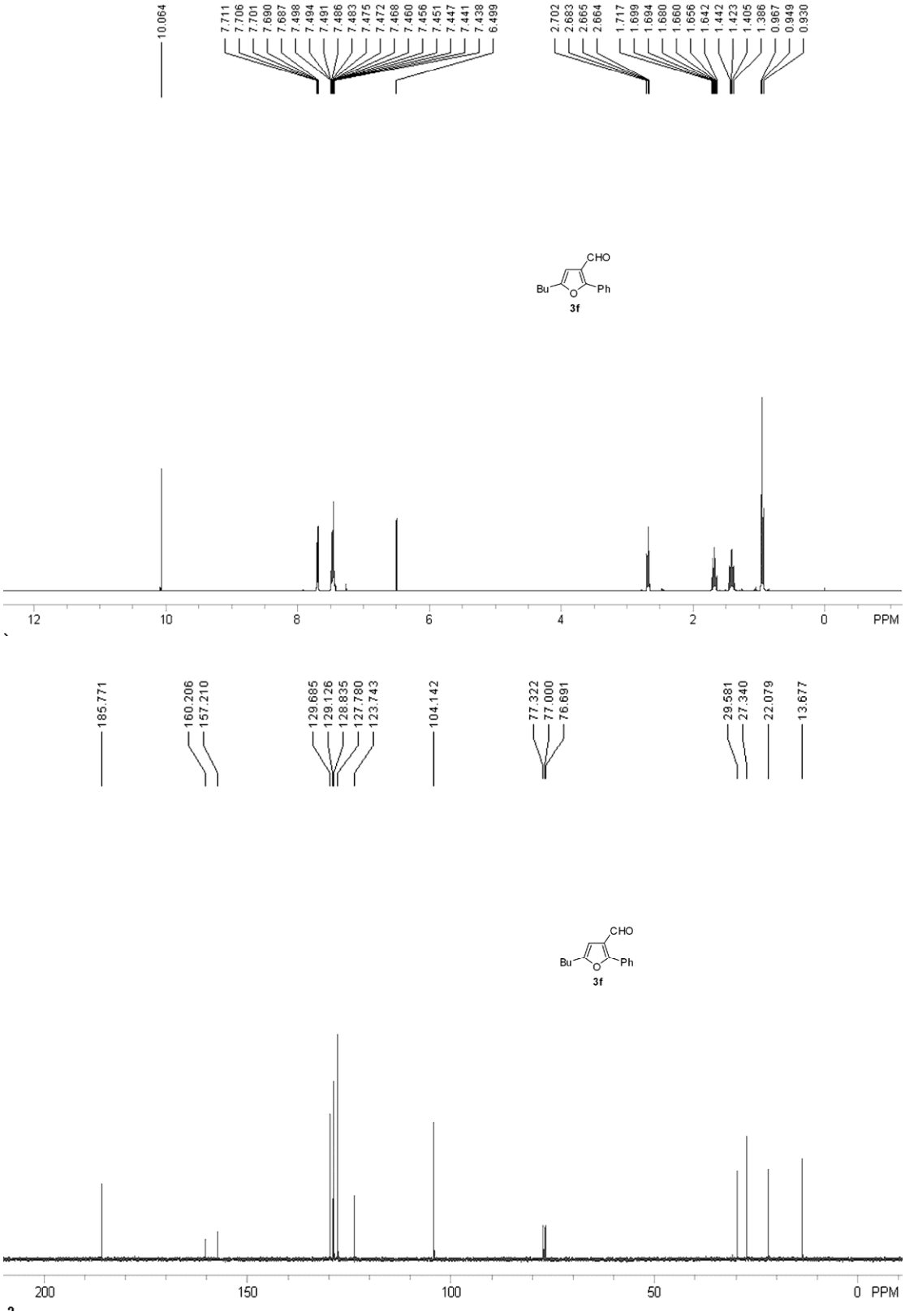
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7.438
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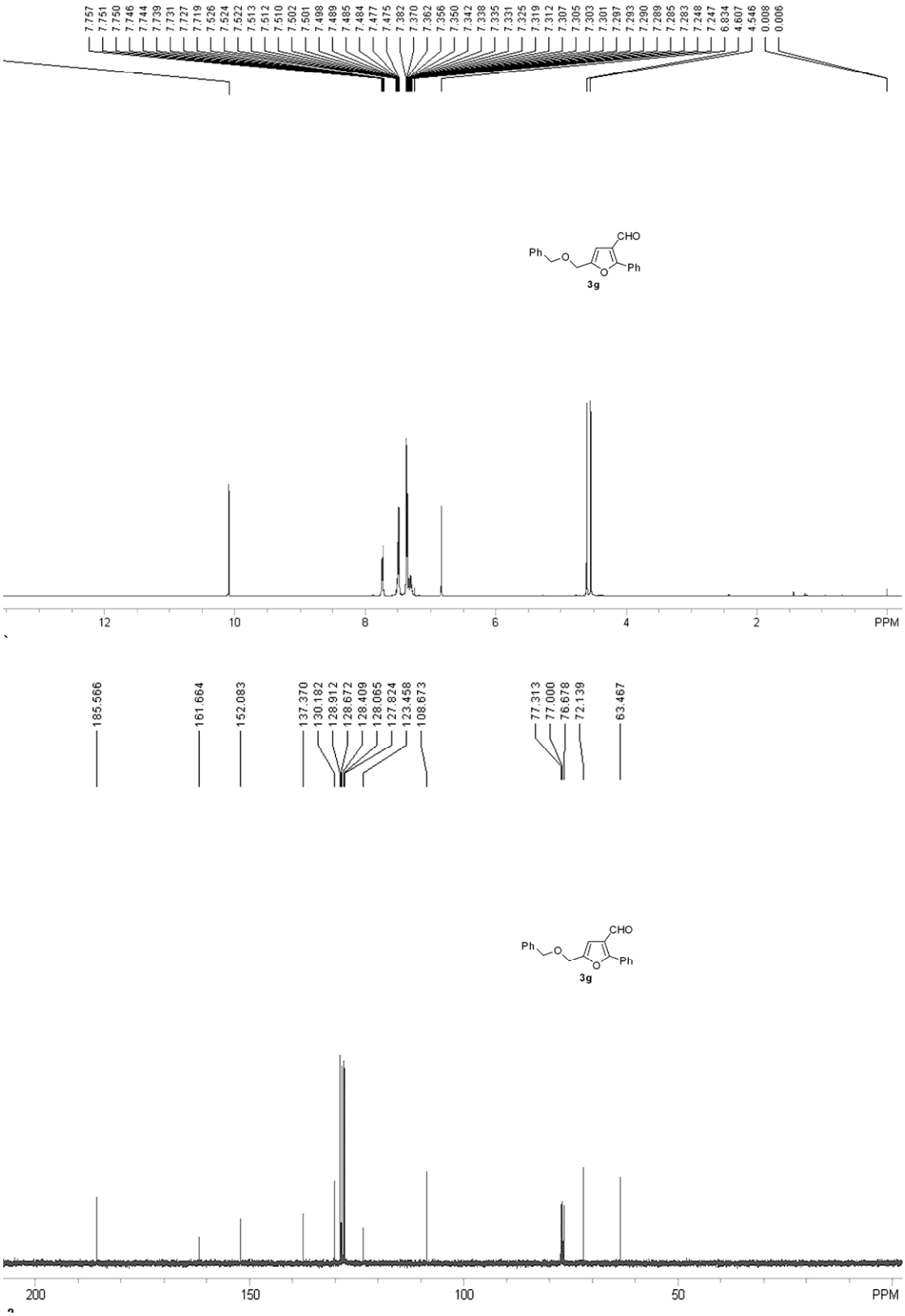
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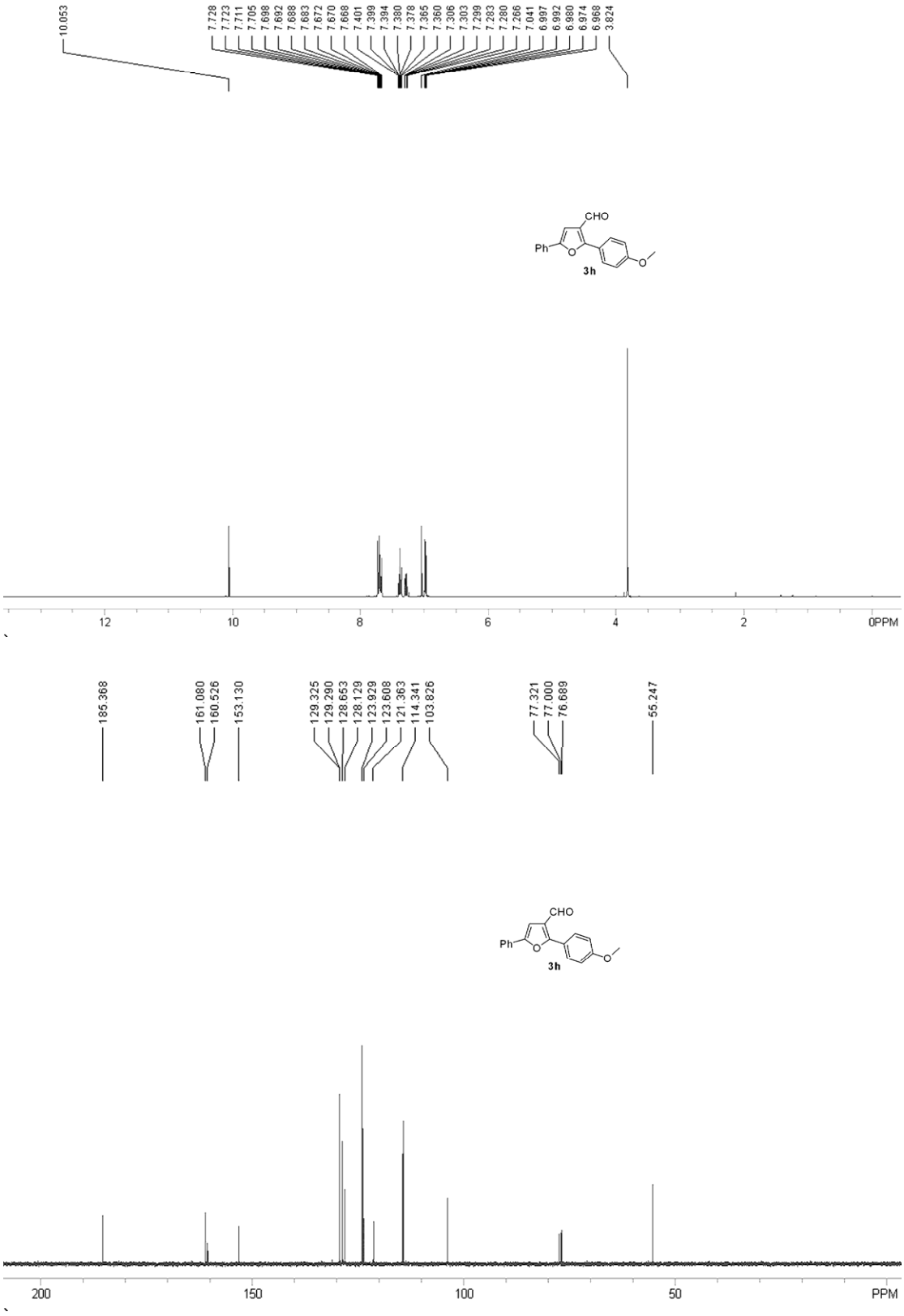
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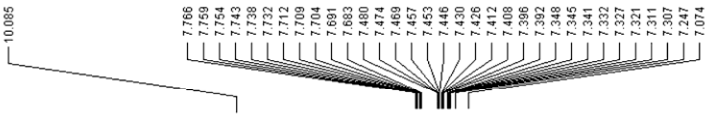
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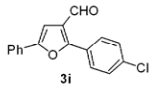
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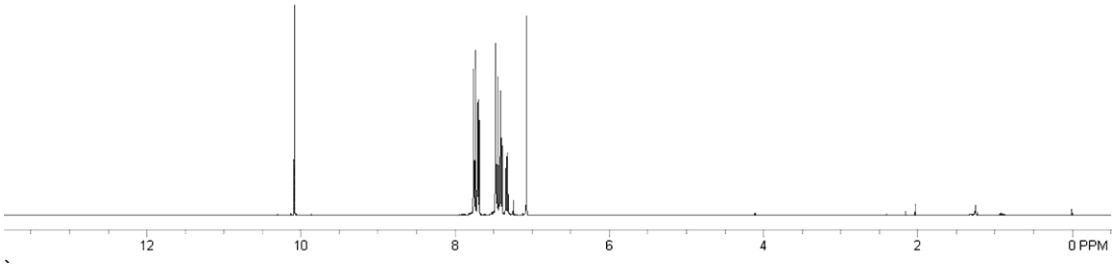
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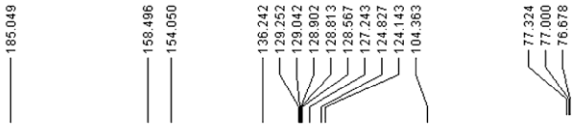
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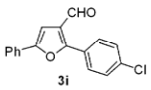
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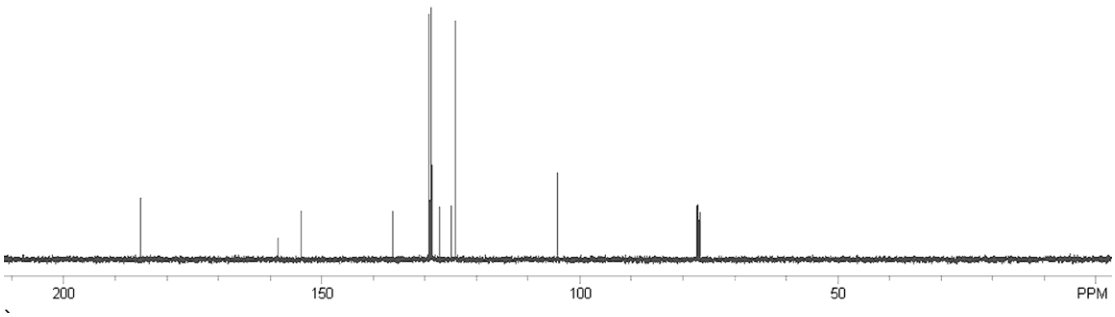
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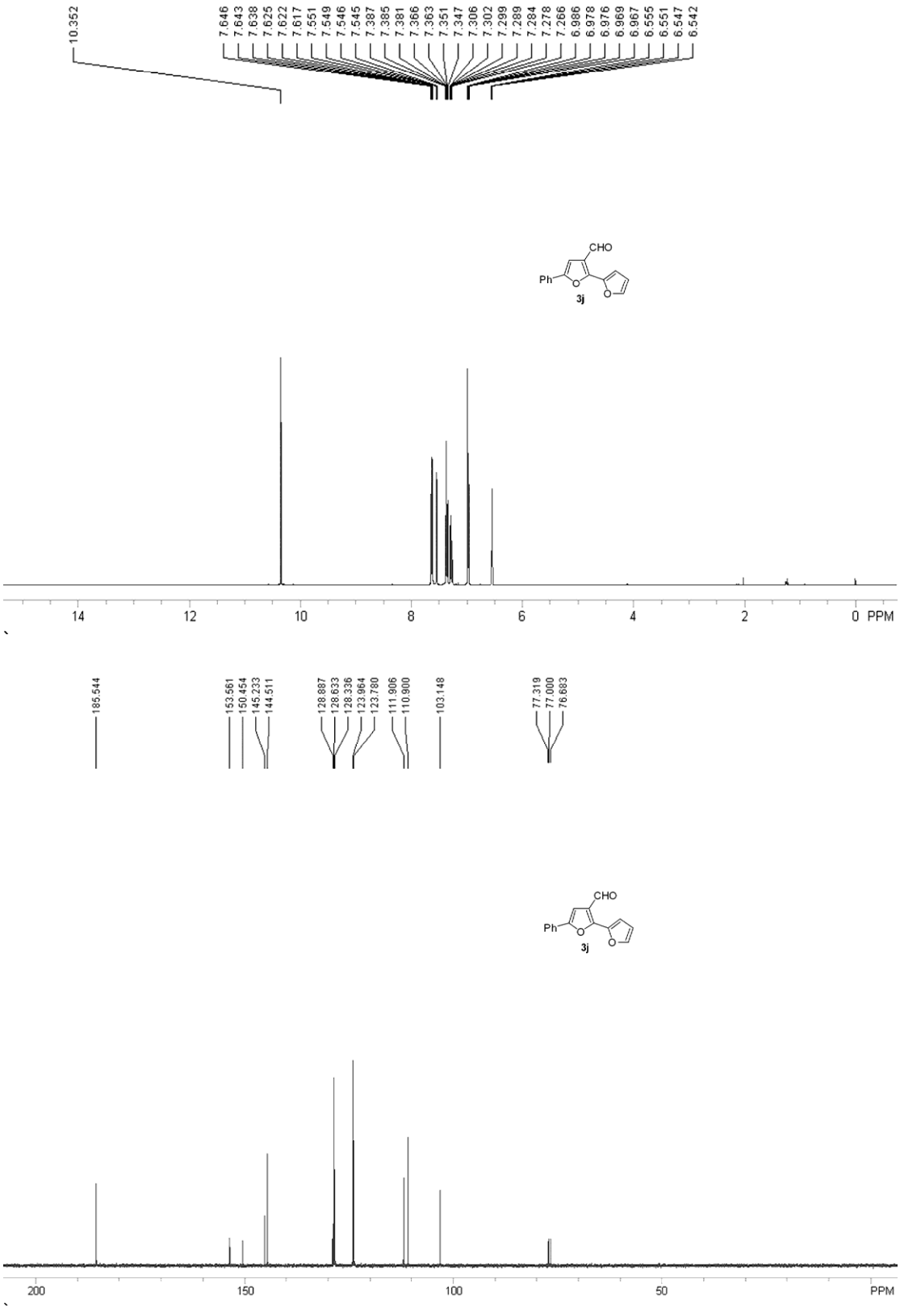
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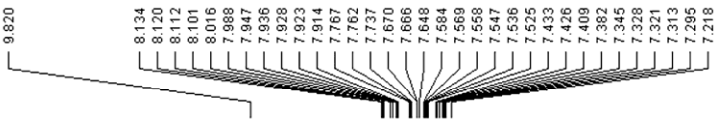
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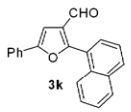
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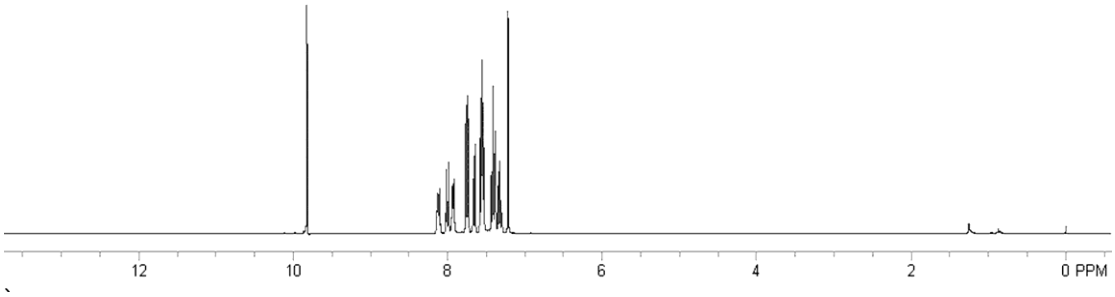
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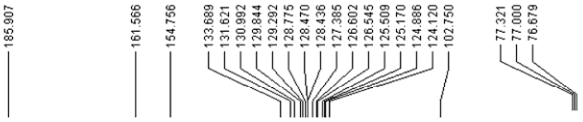
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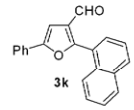
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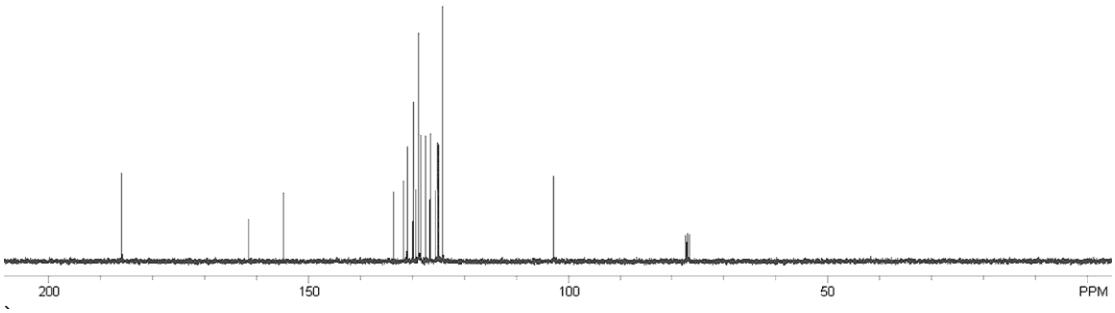
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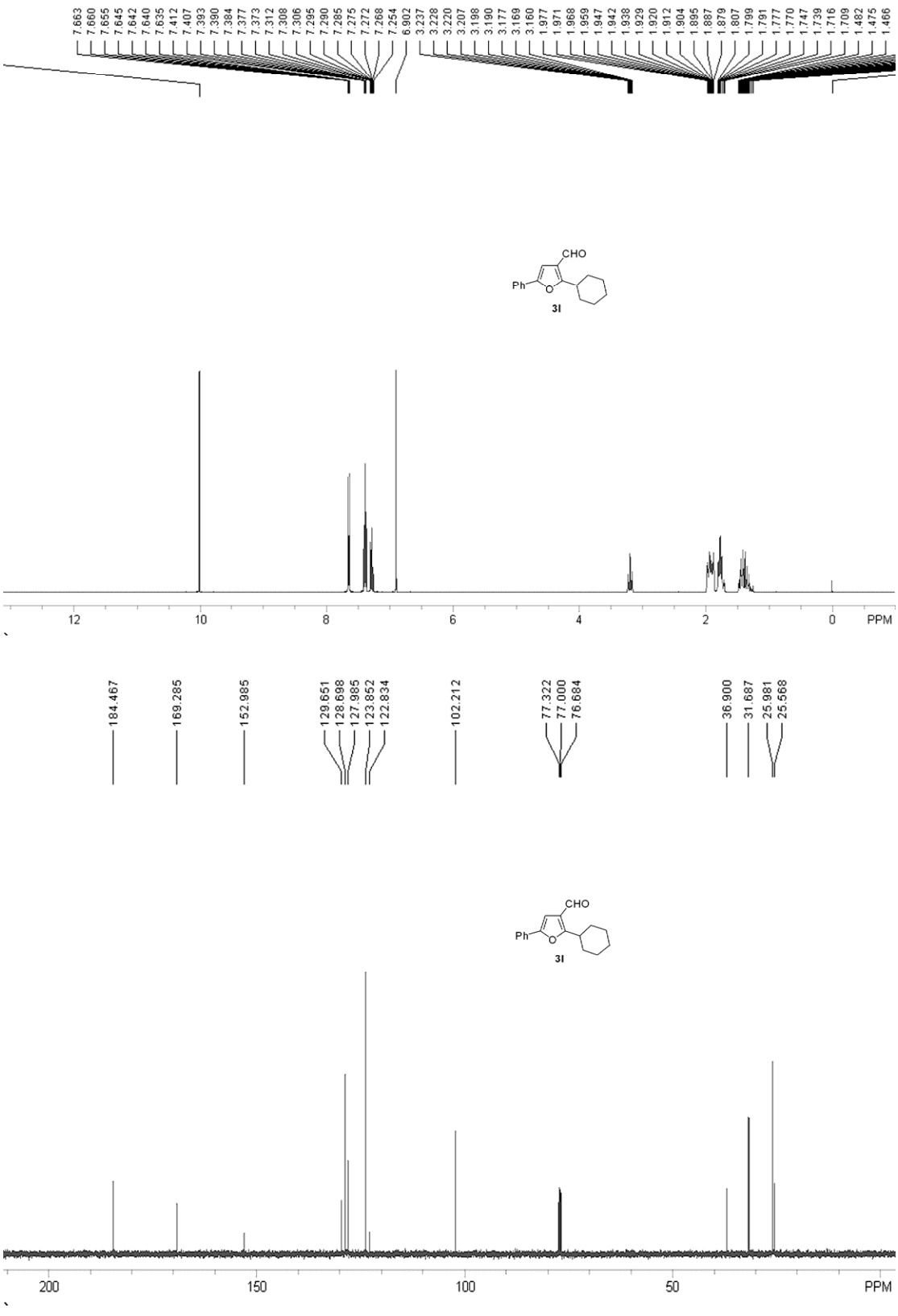
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1.716
1.709
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CHO
Ph
O
3l
12
10
8
6
4
2
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PPM
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3l
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50
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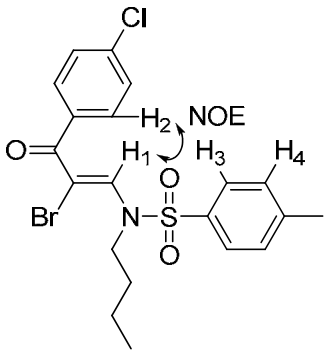
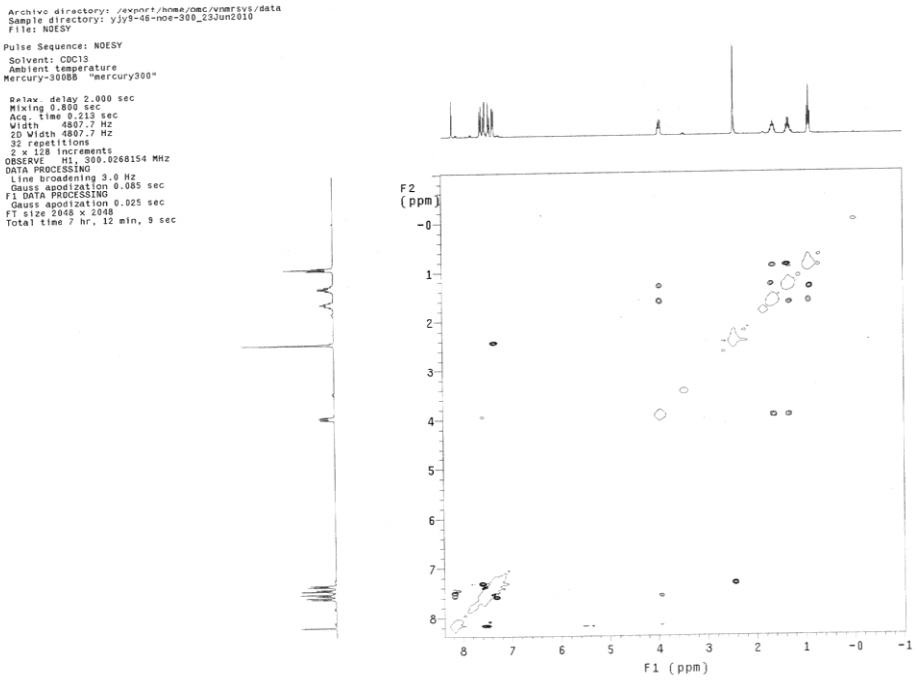
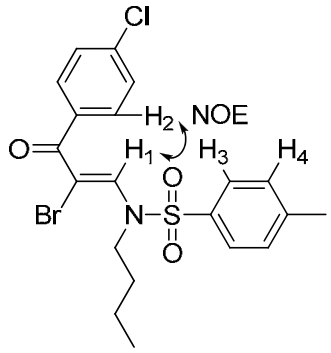


1d

^{1}H- ^{1}H NOESY





1d

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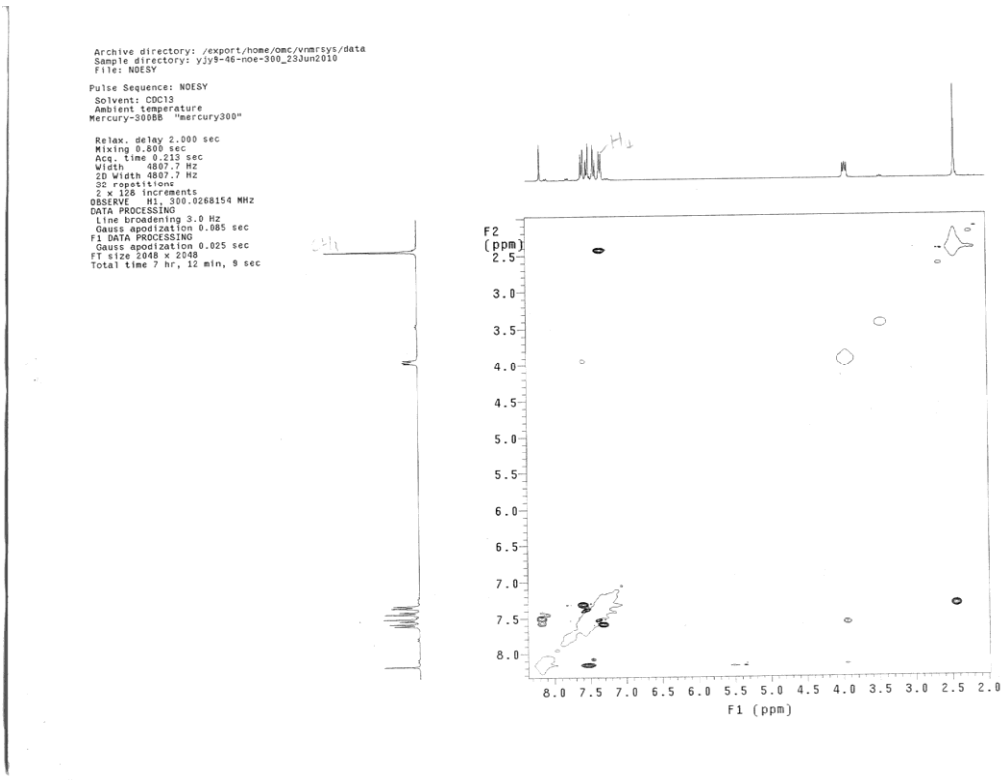

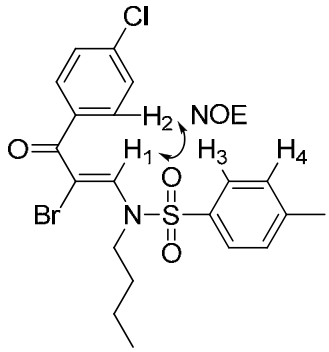

1d

^{1}H- ^{1}H NOESY-enlarged