Supporting Information

Copper-Catalyzed sp^3 C-H Aminative Cyclization of 2-Alkyl-*N*-arylbenzamides: An Approach for the Synthesis of *N*-Aryl-isoindolinones

Kanako Nozawa-Kumada, Jun Kadokawa, Takehiro Kameyama and Yoshinori Kondo*

Graduate School of Pharmaceutical Sciences, Tohoku University 6-3, Aoba, Aramaki, Aoba-ku, Sendai 980-8578, Japan

e-mail: ykondo@m.tohoku.ac.jp

1. General Comments

Melting points (mp) were determined with a Yazawa micro melting point apparatus and uncorrected. Infrared (IR) data were recorded on a SHIMADZU IRAffinity. Absorbance frequencies are reported in reciprocal centimeters (cm^{-1}). NMR data were recorded on a JEOL AL400 (400 MHz) or JEOL ECA600 (600 MHz) spectrometer. Chemical shifts are expressed in δ (parts per million, ppm) values and coupling constants are expressed in herts (Hz). ^{1}H NMR spectra were referenced to tetramethylsilane as an internal standard or to a solvent signal ($CDCl_3$: 7.26 ppm). ^{13}C NMR spectra were referenced to a solvent signal ($CDCl_3$: 77.0 ppm). The following abbreviations are used: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = double doublet, br.s. = broad singlet. Low and high resolution mass spectra (LRMS and HRMS) were obtained from Mass Spectrometry Resource, Graduate School of Pharmaceutical Sciences, Tohoku University, on a JEOL JMS-DX 303 and JMS-700/JMS-T 100 GC spectrometer respectively.

2. Materials

Benzoic acids and other commercially available materials including copper salts were purchased from Tokyo Kasei Co., Aldrich Inc. and other commercial suppliers and were used as received. Flash column chromatography was performed with Kanto silica gel 60 N (spherical, neutral, 70−230 mesh).

3. Preparation of Starting Materials (1a−1p)

Method A

Benzoyl chloride (1.0 mmol) was added to a mixture of (substituted) aniline (1.1 mmol), DMAP (0.1 mmol), and triethylamine (1.2 mmol) in DCM (1 mL) at 0 °C. The reaction mixture was stirred at room temperature for 6 h. The reaction was quenched by adding water (10 mL) and extracted with AcOEt (10 mL × 3). The organic layer was washed with brine and dried over $MgSO_4$. The

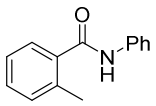
solvent was removed under a reduced pressure and the residue was purified by SiO_2 column chromatography.

Method B

Benzoic acid (1.0 mmol) was added to a mixture of (substituted) aniline (1.1 mmol), EDC·HCl (1.2 mmol), and triethylamine (1.2 mmol) in DCM (1 mL) at 0 °C. The reaction mixture was stirred at room temperature for 6 h. The reaction was quenched by adding water (10 mL) and extracted with AcOEt (10 mL × 3). The organic layer was washed with brine and dried over $MgSO_4$. The solvent was removed under a reduced pressure and the residue was purified by SiO_2 column chromatography.

4. Spectroscopic and Analytical Data for 1a–1p

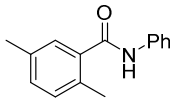
2-Methyl-*N*-phenylbenzamide (1a)



Prepared according to Method A.

Recrystallized from acetone/hexane, colorless needles, mp 125−126 °C (lit.[1] mp 125−126 °C). ^{1}H NMR (400 MHz, $CDCl_3$/TMS) δ (ppm): 2.41 (3H, s), 7.09−7.37 (7H, m), 7.58 (2H, d, J = 6.8 Hz), 7.87 (1H, br.s.); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$/TMS) δ (ppm): 19.6, 119.9, 124.3, 125.7, 126.6, 128.9, 130.0, 131.0, 136.2, 136.3, 138.0, 168.2; LRMS (EI) *m/z*: 211 (M^+); HRMS: Calcd. for $C_{14}H_{13}NO$: 211.0997, found: 211.0972; IR (neat): 2364, 1648, 1539, 1440, 1323, 1269 cm^{-1}.

2,5-Dimethyl-*N*-phenylbenzamide (1b)

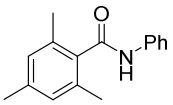


Prepared according to Method B.

Recrystallized from acetone/hexane, colorless needles, mp 148−150 °C (lit.[2] mp 98 °C).

^{1}H NMR (400 MHz, $CDCl_3$/TMS) δ (ppm): 2.34 (3H, s), 2.44 (3H, s), 7.12−7.16 (3H, m), 7.28 (1H, s), 7.35 (2H, t, *J* = 7.8 Hz), 7.50 (1H, s), 7.60 (2H, d, *J* = 7.8 Hz); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$/TMS) δ (ppm): 19.2, 20.7, 119.8, 124.4, 127.2, 129.0, 130.8, 131.0, 133.0, 135.4, 136.2, 138.0, 168.3; LRMS (EI) *m/z*: 225 (M^+); HRMS: Calcd. for $C_{15}H_{15}NO$: 225.1154, found: 225.1151; IR (neat): 3233, 2342, 1640, 1594, 1440, 1322 cm^{-1}.

2,4,6-Trimethyl-*N*-phenylbenzamide (1c)

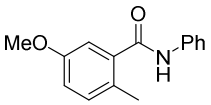


Prepared according to Method B.

Recrystallized from acetone/hexane, colorless needles, mp 170−171 °C (lit.[3] mp 156−157 °C).

^{1}H NMR (400 MHz, $CDCl_3$/TMS) δ (ppm): 2.29 (3H, s), 2.32 (6H, s), 6.86 (2H, s), 7.13 (1H, t, *J* = 7.6 Hz), 7.34 (2H, t, *J* = 7.6 Hz), 7.49 (1H, br.s), 7.58 (2H, d, *J* = 7.6 Hz); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$/TMS) δ (ppm): 19.1, 21.1, 119.8, 124.5, 128.3, 129.0, 134.2, 134.9, 137.9, 138.8, 168.8; LRMS (EI) *m/z*: 239 (M^+); HRMS: Calcd. for $C_{16}H_{17}NO$: 239.1310, found: 239.1322; IR (neat): 3279, 1654, 1541, 1441, 1322, 853 cm^{-1}.

5-Methoxy-2-methyl-*N*-phenylbenzamide (1d)

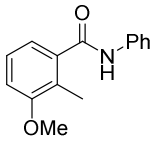


Prepared according to Method B.

Recrystallized from acetone/hexane, colorless needles, mp 105−107 °C.

^{1}H NMR (400MHz, $CDCl_3$/TMS) δ (ppm): 2.40 (3H, s), 3.78 (3H, s), 6.87–6.90 (1H, m), 6.99 (1H, s), 7.12–7.15 (2H, m), 7.35 (2H, t, *J* = 7.8 Hz), 7.58–7.60 (3H, m); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$/TMS) δ (ppm):18.8, 55.4, 112.2, 115.9, 119.9, 124.5, 127.9, 129.1, 132.2, 137.3, 137.9, 157.6, 167.8; LRMS (EI) *m/z:* 241 (M^+); HRMS: Calcd.for $C_{15}H_{15}NO_2$: 241.1103, found: 241.1098; IR (neat): 1653, 1593, 1533, 1498, 1440, 1319, 1282 cm^{-1}.

3-Methoxy-2-methyl-*N*-phenylbenzamide (1e)

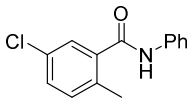


Prepared according to Method B.

Recrystallized from acetone/hexane, colorless needles, mp 156−158 °C.

^{1}H NMR (400MHz, $CDCl_3$/TMS) δ (ppm): 2.32 (3H, s), 3.85 (3H, s), 6.92 (1H, d, J = 8.3 Hz), 7.04 (1H, d, J = 7.3 Hz), 7.14 (1H, t, J = 7.8 Hz), 7.21 (1H, t, J = 8.3 Hz), 7.35 (2H, t, J = 7.8 Hz), 7.49 (1H, br.s.), 7.60 (2H, d, J = 7.8 Hz); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$/TMS) δ (ppm): 12.6, 55.7, 111.7, 118.5, 119.8, 124.5, 124.9, 126.8, 129.1, 138.0, 138.2, 158.2, 168.0; LRMS (EI) *m/z*: 241 (M^+); HRMS: Calcd.for $C_{15}H_{15}NO_2$: 241.1103, found: 241.1097; IR (neat): 1654, 1595, 1530, 1496, 1439, 1323, 1256, 1070 cm^{-1}.

5-Chloro-2-methyl-*N*-phenylbenzamide (1f)

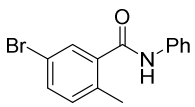


Prepared according to Method B.

Recrystallized from acetone/hexane, colorless needles, mp 172−174 °C.

^{1}H NMR (400 MHz, $CDCl_3$/TMS) δ (ppm): 2.45 (3H, s), 7.15−7.20 (2H, m), 7.30−7.39 (3H, m), 7.44 (1H, s), 7.52 (1H, s), 7.60 (2H, d, J = 7.6 Hz); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$/TMS) δ (ppm): 19.1, 120.1, 124.8, 126.6, 129.1, 130.1, 131.5, 132.5, 134.8, 137.6, 137.7, 166.7; LRMS (EI) *m/z*: 245 (M^+); HRMS: Calcd. for $C_{14}H_{12}{}^{35}ClNO$: 245.0607, found: 245.0587; IR (neat): 3277, 2360, 1653, 1527, 1320 cm^{-1}.

5-Bromo-2-methyl-*N*-phenylbenzamide (1g)

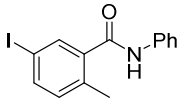


Prepared according to Method B.

Recrystallized from acetone/hexane, colorless needles, mp 158−160 °C.

^{1}H NMR (400 MHz, $CDCl_3$/TMS) δ (ppm): 2.42 (3H, s), 7.11−7.18 (2H, m), 7.36 (2H, t, *J* = 8.0 Hz), 7.45 (1H, dd, *J* = 8.4, 2.0 Hz), 7.58−7.60 (4H, m); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$/TMS) δ (ppm): 19.2, 119.2, 120.0, 124.8, 129.1, 129.5, 132.8, 133.1, 135.3, 137.6, 138.1, 166.5; LRMS (EI) *m/z*: 289 (M^+); HRMS: Calcd. for $C_{14}H_{12}{}^{79}BrNO$: 289.0102, found: 289.0109; IR (neat): 3281, 1652, 1522, 1440, 1260, 819 cm^{-1}.

5-Iodo-2-methyl-*N*-phenylbenzamide (1h)

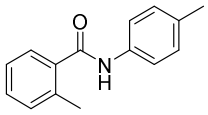


Prepared according to Method B.

Recrystallized from acetone/hexane, colorless needles, mp 145−146 °C.

^{1}H NMR (400 MHz, $CDCl_3$/TMS) δ (ppm): 2.44 (3H, s), 7.02 (1H, d, *J* = 7.8 Hz), 7.17 (1H, t, *J* = 7.3 Hz), 7.36 −7.40 (3H, m), 7.61 (2H, d, *J* = 7.3 Hz), 7.67 (1H, dd, *J* = 8.0, 2.0 Hz), 7.80 (1H, s); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$/TMS) δ (ppm): 19.4, 90.2, 120.0, 124.8, 129.1, 133.1, 135.2, 136.0, 137.6, 138.5, 139.1, 166.3; LRMS (EI) *m/z*: 336 (M^+); HRMS: Calcd. for $C_{14}H_{12}INO$: 336.9964, found: 336.9955; IR (neat): 3280, 1651, 1523, 1494, 1436, 1315, 1255 cm^{-1}.

2-Methyl-*N*-(*p*-tolyl)benzamide (1i)



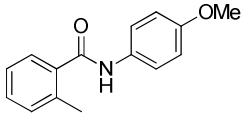
Prepared according to Method A.

Recrystallized from acetone/hexane, colorless needles, mp 145−147 °C (lit.[4] mp 143.5−144 °C).

^{1}H NMR (400 MHz, $CDCl_3$/TMS) δ (ppm): 2.32 (3H, s), 2.46 (3H, s), 7.13−7.20 (2H, m), 7.21−7.24 (2H, m), 7.32 (1H, t, *J* = 7.3 Hz), 7.41 (1H, d, *J* = 7.3 Hz), 7.47 (2H, d, *J* = 7.3 Hz), 7.59 (1H, br.s.); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$/TMS) δ (ppm): 19.8, 20.9, 120.0, 125.8, 126.6, 129.5,

130.0, 131.1, 134.1, 135.5, 136.3, 136.5, 168.1; LRMS (EI) *m/z*: 225 (M^+); HRMS: Calcd. for $C_{15}H_{15}NO$: 225.1154, found: 225.1145; IR (neat): 3227, 2360, 1653, 1558, 1507, 1325 cm^{-1}.

***N*-(4-Methoxyphenyl)-2-methylbenzamide (1j)**

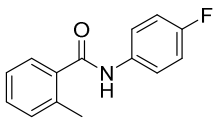


Prepared according to Method A.

Recrystallized from acetone/hexane, colorless needles, mp 147−149 °C (lit.[5] mp 143–144.5 °C).

^1H NMR (400 MHz, $CDCl_3$/TMS) δ (ppm): 2.49 (3H, s), 3.81 (3H, s), 6.90 (2H, d, *J* = 8.8 Hz), 7.23−7.24 (2H, m), 7.33−7.36 (1H, m), 7.43−7.53 (4H, m); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$/TMS) δ (ppm): 19.8, 55.5, 114.2, 121.7, 125.8, 126.6, 130.1, 131.1, 131.2, 136.4, 136.5, 156.6, 167.9; LRMS (EI) *m/z*: 241 (M^+); HRMS: Calcd. for $C_{15}H_{15}NO_2$: 241.1103, found: 241.1134; IR (neat): 3281, 1644, 1512, 1409, 1244, 1031, 824 cm^{-1}.

***N*-(4-fluorophenyl)-2-methylbenzamide (1k)**

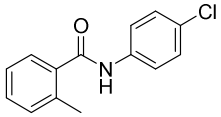


Prepared according to Method A.

Recrystallized from acetone/hexane, colorless needles, mp 143−145 °C.

^1H NMR (400MHz, $CDCl_3$/TMS) δ (ppm): 2.50 (3H, s), 7.06 (2H, t, *J* = 8.8 Hz), 7.25−7.28 (2H, m), 7.37 (1H, t, *J* = 7.1 Hz), 7.46–7.48 (2H, m), 7.58 (2H, s); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$/TMS) δ (ppm): 19.7, 115.6 (d, J_{FC} = 23.0 Hz), 121.8 (d, J_{FC} = 8.2 Hz), 125.8, 126.6, 130.2, 131.2, 134.0 (d, J_{FC} = 2.5 Hz), 136.1, 136.3, 159.5 (d, J_{FC} = 228.0 Hz), 168.4; LRMS (EI) *m/z*: 229 (M^+); HRMS: Calcd.for $C_{14}H_{12}FNO$. 229.0903, found: 229.0912; IR (neat): 2358, 1653, 1647, 1559, 1517, 1490, 1457 cm^{-1}.

***N*-(4-Chlorophenyl)-2-methylbenzamide (1l)**

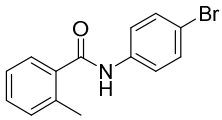


Prepared according to Method A.

Recrystallized from acetone/hexane, colorless needles, mp 135−137 °C (lit.[6] mp 138 °C).

^{1}H NMR (400MHz, $CDCl_3$/TMS) δ (ppm): 2.51 (3H, s), 7.25−7.49 (7H, m), 7.58 (2H, d, J = 8.3 Hz); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$/TMS) δ (ppm): 19.7, 121.1, 125.9, 126.6, 129.0, 129.5, 130.4, 131.3, 136.0, 136.4, 136.6, 168.1; LRMS (EI) *m/z*: 245 (M^+); HRMS: Calcd. for $C_{14}H_{12}{}^{35}ClNO$: 245.0607, found: 245.0615; IR (neat): 2358, 1653, 1507, 1490, 1394, 1309 cm^{-1}.

***N*-(4-Bromophenyl)-2-methylbenzamide (1m)**

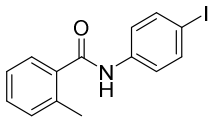


Prepared according to Method A.

Recrystallized from acetone/hexane, colorless needles, mp 144−146 °C.

^{1}H NMR (400MHz, $CDCl_3$/TMS) δ (ppm): 2.51 (3H, s), 7.26−7.29 (2H, m), 7.36−7.52 (7H, m); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$/TMS) δ (ppm): 19.7, 117.0, 121.5, 125.8, 126.6, 130.3, 131.2, 131.9, 135.9, 136.3, 137.1, 168.2; LRMS (EI) *m/z*: 289 (M^+); HRMS: Calcd.for $C_{14}H_{12}{}^{79}BrNO$: 289.0102, found: 289.0112; IR (neat): 3298, 1651, 1585, 1506, 1487, 1388, 1309, 1070 cm^{-1}.

***N*-(4-Iodophenyl)-2-methylbenzamide (1n)**



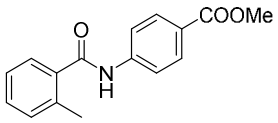
Prepared according to Method A.

Recrystallized from acetone/hexane, colorless needles, mp 160−164 °C.

^{1}H NMR (400MHz, $CDCl_3$/TMS) δ (ppm): 2.49 (3H, s), 7.23−7.28 (2H, m), 7.35−7.49 (5H, m), 7.66 (2H, d, J = 8.8 Hz); $^{13}C\{^1H\}$ NMR (150 MHz, $CDCl_3$/TMS) δ (ppm): 19.8, 87.7, 121.7, 125.9,

126.5, 130.4, 131.3, 136.0, 136.4, 137.7, 137.9, 168.1; LRMS (EI) *m/z*: 337 (M^+); HRMS: Calcd.for $C_{14}H_{12}INO$: 336.9964 , found: 336.9957; IR (neat): 3217, 1651, 1529, 1485, 1390, 1321, 1242, 1004 cm^{-1}.

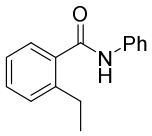
Methyl 4-(2-methylbenzamido)benzoate (1o)



Prepared according to Method A.

Recrystallized from acetone/hexane, colorless prisms, mp 162−165 °C (lit.[7] mp 159.9−160.8 °C). ^1H NMR (400MHz, $CDCl_3$/TMS) δ (ppm): 2.52 (3H, s), 3.92 (3H, s), 7.27−7.30 (2H, m), 7.39 (1H, t, *J* = 7.3 Hz), 7.49 (1H, d, *J* = 7.8 Hz), 7.59 (1H, s), 7.71 (2H, d, *J* = 8.8 Hz), 8.06 (2H, d, *J* = 8.8 Hz); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$/TMS) δ (ppm): 19.8, 52.0, 118.9, 125.8, 126.0, 126.6, 130.6, 130.9, 131.4, 135.9, 136.6, 142.1, 166.5, 168.1; LRMS (EI) *m/z*: 269 (M^+); HRMS: Calcd. for $C_{16}H_{15}NO_3$: 269.1052, found: 269.1055; IR (neat): 3221, 1718, 1653, 1533, 1406, 1282 cm^{-1}.

2-Ethyl-*N*-phenylbenzamide (1p)



Prepared according to Method A.

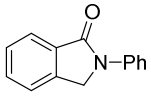
Recrystallized from acetone/hexane, colorless needles, mp 142−145 °C (lit.[8] mp 141−142.5 °C). ^1H NMR (400 MHz, $CDCl_3$/TMS) δ (ppm): 1.28 (3H, t, *J* = 7.8 Hz), 2.86 (2H, q, *J* = 7.8 Hz), 7.16 (1H, t, *J* = 7.3 Hz), 7.24−7.48 (7H, m), 7.61 (2H, d, *J* = 8.8 Hz); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$/TMS) δ (ppm): 15.9, 26.3, 119.9, 124.5, 125.9, 126.6, 129.1, 129.6, 130.3, 136.2, 138.0, 142.6, 168.2; LRMS (EI) *m/z*: 225 (M^+); HRMS: Calcd.for $C_{15}H_{15}NO$: 225.1154, Found: 225.1149; IR (neat): 2360, 1637, 1593, 1539, 1489, 1438, 1325 cm^{-1}.

5. Representative Procedure for 2-phenylisoindolin-1-one Synthesis (Table1, entry 13)

In a glove box, 2-methyl-*N*-phenylbenzamide (**1a**, 21.1 mg, 0.10 mmol), CuI (1.9 mg, 0.010 mmol), DMAP (3.7 mg, 0.030 mmol), and *t*BuOO*t*Bu (29.2 mg, 0.20 mmol) were added in a sealed tube and then DCM (0.5 mL) was added. The mixture was stirred at 100 °C for 24 h. The reaction was quenched by adding water (10 mL) and extracted with AcOEt (10 mL × 3). The organic layer was washed with brine (10 mL) and dried over $MgSO_4$. The solvent was removed under a reduced pressure and the residue was purified by SiO_2 column chromatography to give **2a** (17.3 mg, 82 %).

6. Spectroscopic and Analytical Data for 2a–p

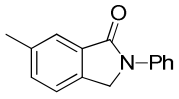
2-Phenylisoindolin-1-one (2a)



2a was obtained in 82% yield (17.3 mg), recrystallized from acetone/hexane, colorless prisms, mp 168−170 °C (lit.[9] mp 162–163 °C).

^{1}H NMR (400 MHz, $CDCl_3$/TMS) δ (ppm): 4.84 (2H, s), 7.17 (1H, t, J = 7.6 Hz), 7.42 (2H, t, J = 8.3 Hz), 7.50 (2H, t, J = 7.2 Hz), 7.59 (1H, t, J = 7.2 Hz), 7.86 (2H, d, J = 8.3 Hz), 7.91 (1H, d, J = 7.2 Hz); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$/TMS) δ (ppm): 50.7, 119.4, 122.6, 124.1, 124.4, 128.3, 129.1, 132.0, 133.2, 139.5, 140.1, 167.5; LRMS (EI) *m/z*: 209 (M^+); HRMS: Calcd. for $C_{14}H_{11}NO$: 209.0841, found: 209.0864; IR (neat): 1681, 1595, 1387, 1151, 750 cm^{-1}.

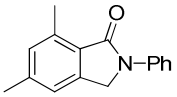
6-Methyl-2-phenylisoindolin-1-one (2b)



2b was obtained in 84% yield (18.7 mg), recrystallized from acetone/hexane, colorless needles, mp 189−191 °C (lit.[10] mp 190–191 °C).

^{1}H NMR (400 MHz, $CDCl_3$/TMS) δ (ppm): 2.45 (3H, s), 4.80 (2H, s), 7.16 (1H, t, *J* = 7.3 Hz), 7.39−7.44 (4H, m), 7.72 (1H, s), 7.86 (2H, d, *J* = 8.8 Hz); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$/TMS) δ (ppm): 21.3, 50.5, 119.4, 122.3, 124.27, 124.31, 129.1, 133.1, 133.3, 137.3, 138.4, 139.6, 167.6; LRMS (EI) *m/z*: 223 (M^+); HRMS: Calcd. for $C_{15}H_{13}NO$: 223.0997, found: 223.0991; IR (neat): 2360, 1680, 1387, 1141, 825 cm^{-1}.

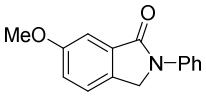
5,7-Dimethyl-2-phenylisoindolin-1-one (2c)



2c was obtained in 70% yield (16.7 mg), recrystallized from acetone/hexane, colorless needles, mp 158−159 °C.

^{1}H NMR (400 MHz, $CDCl_3$/TMS) δ (ppm): 2.41 (3H, s), 2.72 (3H, s), 4.72 (2H, s), 7.03 (1H, s), 7.10 (1H, s), 7.14 (1H, t, *J* = 7.8 Hz), 7.40 (2H, t, *J* = 7.8 Hz), 7.85 (2H, d, *J* = 7.8 Hz); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$/TMS) δ (ppm): 17.3, 21.7, 49.9, 119.2, 120.5, 124.0, 127.7, 129.0, 131.4, 138.0, 139.8, 141.1, 142.3, 168.4; LRMS (EI) *m/z*: 237 (M^+); HRMS: Calcd. for $C_{16}H_{15}NO$: 237.1154, found: 237.1136; IR (neat):2358, 1675, 1501, 1374, 1275 cm^{-1}.

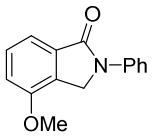
6-Methoxy-2-phenylisoindolin-1-one (2d)



2d was obtained in quantitative yield (23.4 mg), recrystallized from acetone/hexane, colorless needles, mp 175−178 °C.

^{1}H NMR (600MHz, $CDCl_3$/TMS) δ (ppm): 3.90 (3H, s), 4.81 (2H, s), 7.15−7.20 (2H, m), 7.40−7.45 (4H, m), 7.86 (2H, d, *J* = 8.3 Hz); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$/TMS) δ (ppm): 50.3, 55.7, 106.6, 119.4, 120.7, 123.5, 124.4, 129.1, 132.3, 134.5, 139.6, 160.2, 167.5; LRMS (EI) *m/z*: 239 (M^+); HRMS: Calcd.for $C_{15}H_{13}NO_2$: 239.0946, found: 239.0946; IR (neat): 1669, 1654, 1559, 1490, 1457, 1387, 1282, 1248, 1145 cm^{-1}.

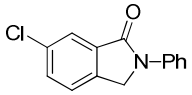
4-Methoxy-2-phenylisoindolin-1-one (2e)



Recrystallized from acetone/hexane, colorless needles, mp 142–144 °C (lit.[11] mp 145–146 °C).

^{1}H NMR (600MHz, $CDCl_3$/TMS) δ (ppm): 3.94 (3H, s), 4.79 (2H, s), 7.05 (1H, d, *J* = 8.2 Hz), 7.17 (1H, t, *J* = 7.5 Hz), 7.42 (2H, t, *J* = 5.0 Hz), 7.46 (1H, t, *J* = 5.0 Hz), 7.52 (1H, d, *J* = 7.5 Hz), 7.88 (2H, d, *J* = 8.2 Hz); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$/TMS) δ (ppm): 48.6, 55.5, 113.2, 116.2, 119.4, 124.4, 128.4, 129.1, 130.0, 134.9, 139.7, 154.5, 167.5; LRMS (EI) *m/z*: 239 (M^+); HRMS: Calcd.for $C_{15}H_{13}NO_2$: 239.0946, found: 239.0976; IR (neat); 1683, 1492, 1444, 1381, 1260, 1104, 1048 cm^{-1}.

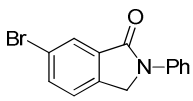
6-Chloro-2-phenylisoindolin-1-one (2f)



2f was obtained in 57% yield (42.1 mg, 0.3 mmol scale), recrystallized from acetone/hexane, colorless needles, mp 209−210 °C.

^{1}H NMR (400 MHz, $CDCl_3$/TMS) δ (ppm): 4.84 (2H, s), 7.20–7.22 (1H, m), 7.41–7.47 (3H, m), 7.57 (1H, dd, *J* = 8.3, 2.0 Hz), 7.83–7.85 (2H, m), 7.89 (1H, d, *J* = 2.0 Hz); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$/TMS) δ (ppm): 50.4, 119.6, 123.9, 124.3, 124.8, 129.2, 132.3, 134.8, 135.0, 138.2, 139.1, 164.6; LRMS (EI) *m/z*: 243 (M^+); HRMS: Calcd. for $C_{14}H_{10}{}^{35}ClNO$: 243.0451, found: 243.0444; IR (neat): 2362, 1684, 1595, 1386, 1215, 1155 cm^{-1}.

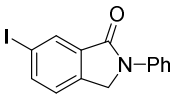
6-Bromo-2-phenylisoindolin-1-one (2g)



2g was obtained in 50% yield (14.1 mg), recrystallized from acetone/hexane, colorless prisms, mp 224–226°C.

^{1}H NMR (400 MHz, $CDCl_3$/TMS) δ (ppm): 4.83 (2H, s), 7.20 (1H, t, *J* = 7.6 Hz), 7.39–7.46 (3H, m), 7.71 (1H, dd, *J* = 8.0, 2.0 Hz), 7.85 (2H, d, *J* = 7.8 Hz), 8.06 (1H, d, *J* = 2.0 Hz); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$/TMS) δ (ppm): 50.5, 119.6, 122.5, 124.2, 124.8, 127.3, 129.2, 135.1, 135.3, 138.7, 139.1, 166.0; LRMS (EI) *m/z*: 287 (M^+); HRMS: Calcd. for $C_{14}H_{10}{}^{79}BrNO$: 286.9946, found: 286.9912; IR (neat): 1681, 1507, 1490, 1450, 1387, 1198 cm^{-1}.

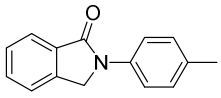
6-Iodo-2-phenylisoindolin-1-one (2h)



2h was obtained in 44% yield (14.9 mg), recrystallized from acetone/hexane, colorless prisms, mp 236–238 °C.

^{1}H NMR (600 MHz, $CDCl_3$/TMS) δ (ppm): 4.80 (2H, s), 7.20 (1H, t, *J* = 7.6 Hz), 7.28 (1H, d, *J* = 8.2 Hz), 7.43 (2H, t, *J* = 7.6 Hz), 7.83 (2H, d, *J* = 8.2 Hz), 7.90 (1H, d, *J* = 8.2 Hz), 8.26 (1H, s); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$/TMS) δ (ppm): 50.5, 93.4, 119.6, 124.4, 124.8, 129.2, 133.3, 135.3, 139.1, 139.4, 140.8, 165.8; LRMS (EI) *m/z*: 335 (M^+); HRMS: Calcd. for $C_{14}H_{10}INO$: 334.9807, found: 334.9800; IR (neat): 1676, 1597, 1502, 1448, 1387, 1324, 1203, 1197, 1151 cm^{-1}.

2-(4-Tolyl)-isoindolin-1-one (2i)

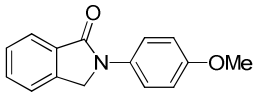


2i was obtained in 70% yield (15.6 mg), recrystallized from acetone/hexane, colorless prisms, mp136−138 °C (lit.[12] mp 134–135 °C).

^{1}H NMR (400MHz, $CDCl_3$/TMS) δ (ppm): 2.35 (3H, s), 4.83 (2H, s), 7.24 (2H, d, *J* = 8.3 Hz), 7.48–7.51 (2H, m), 7.56–7.60 (1H, m), 7.73 (2H, d, *J* = 8.3 Hz), 7.91 (1H, d, *J* = 6.8 Hz); ^{13}C{^{1}H}

NMR (100 MHz, $CDCl_3$/TMS) δ (ppm): 20.8, 50.7, 119.6, 122.4, 124.1, 128.3, 129.7, 131.9, 133.4, 134.3, 137.1, 140.2, 167.4; LRMS (EI) *m/z*: 223 (M^+); HRMS: Calcd. for $C_{15}H_{13}NO$: 223.0997, found: 223.0991; IR (neat): 1669, 1559, 1507, 1386, 1304, 1264, 1157 cm^{-1}.

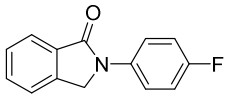
2-(4-Methoxyphenyl)-isoindolin-1-one (2j)



2j was obtained in 66% yield (15.7 mg), recrystallized from acetone/hexane, colorless prisms, mp 144−145 °C (lit.[13] mp 130–134 °C).

^1H NMR (600 MHz, $CDCl_3$/TMS) δ (ppm): 3.83 (3H, s), 4.83 (2H, s), 6.97 (2H, d, *J* = 8.9 Hz), 7.49–7.51 (2H, m), 7.57–7.60 (1H, m), 7.74 (2H, d, *J* = 8.9 Hz), 7.92 (1H, d, *J* = 7.6 Hz); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$/TMS) δ (ppm): 51.1, 55.3, 114.3, 121.5, 122.5, 124.1, 128.3, 131.8, 132.7, 133.3, 140.1, 156.7, 167.2; LRMS (EI) *m/z*: 239 (M^+); HRMS: Calcd. for $C_{15}H_{13}NO_2$: 239.0946, found: 239.0946; IR (neat): 2360, 1700, 1683, 1513, 1393, 828 cm^{-1}.

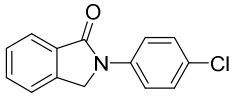
2-(4-Fluorophenyl)isoindolin-1-one (2k)



2k was obtained in quantitative yield (22.7 mg), recrystallized from acetone/hexane, colorless prisms, mp 176–178 °C.

^1H NMR (400MHz, $CDCl_3$/TMS) δ (ppm): 4.84 (2H, s), 7.12 (2H, t, *J* = 9.2 Hz), 7.50–7.53 (2H, m), 7.58−7.62 (1H, m), 7.80−7.84 (2H, m), 7.92 (1H, d, *J* = 8.3 Hz); $^{13}C\{^1H\}$ NMR (150 MHz, $CDCl_3$/TMS) δ (ppm): 51.0, 115.8 (d, J_{FC} = 22.9 Hz), 121.3 (d, J_{FC} = 8.6 Hz), 122.6, 124.2, 128.5, 132.1, 133.0 , 135.6 (d, J_{FC} = 2.9 Hz), 140.0, 159.5 (d, J_{FC} = 243.5 Hz), 167.4; LRMS (EI) *m/z*: 227 (M^+); HRMS: Calcd.for $C_{14}H_{10}FNO$: 227.0746, found: 227.0768; IR (neat): 1676, 1559, 1507, 1490, 1386, 1222, 1153 cm^{-1}.

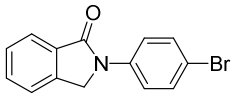
2-(4-Chlorophenyl)isoindolin-1-one (2l)



2l was obtained in 70% yield (17.0 mg), recrystallized from acetone/hexane, colorless needles, mp 180–183 °C (lit.[14] mp 184–185 °C).

^{1}H NMR (400MHz, $CDCl_3$/TMS) δ (ppm): 4.80 (2H, s), 7.36 (2H, d, *J* = 8.8 Hz), 7.50 (2H, d, *J* = 6.8 Hz), 7.59 (1H, t, *J* = 7.3 Hz), 7.82 (2H, d, *J* = 8.8 Hz), 7.89 (1H, d, *J* = 7.3 Hz); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$/TMS) δ (ppm): 50.5, 120.3, 122.6, 124.1, 128.4, 129.1, 129.4, 132.2, 132.8, 138.0, 139.8, 167.4; LRMS (EI) *m/z*: 243 (M^+); HRMS: Calcd.for $C_{14}H_{10}ClNO$: 243.0451, found: 243.0451; IR (neat): 1679, 1594, 1465, 1381, 1334, 1303, 1153, 1091 cm^{-1}.

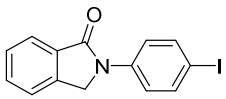
2-(4-Bromophenyl)isoindolin-1-one (2m)



2m was obtained in 57% yield (16.4 mg), recrystallized from acetone/hexane, colorless needles, mp 180–184 °C (lit.[14] mp 183 °C).

^{1}H NMR (400MHz, $CDCl_3$/TMS) δ (ppm): 4.82 (2H, s), 7.49–7.53 (4H, m), 7.59–7.63 (1H, m), 7.76–7.79 (2H, m), 7.92 (1H, d, *J* = 8.3 Hz); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$/TMS) δ (ppm): 50.5, 117.2, 120.7, 122.6, 124.2, 128.5, 132.1, 132.3, 132.9, 138.6, 139.8, 167.5; LRMS (EI) *m/z*: 287 (M^+); HRMS: Calcd.for $C_{14}H_{10}{}^{79}BrNO$: 286.9946 , found: 286.9938; IR (neat): 1680, 1490, 1466, 1380, 1335, 1304, 1147, 1078 cm^{-1}.

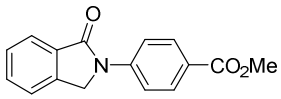
2-(4-Iodophenyl)isoindolin-1-one (2n)



2n was obtained in quantitative yield (33.4 mg), recrystallized from acetone/hexane, colorless prisms, mp 196−199 °C (lit.[15] mp 200–203 °C).

^{1}H NMR (400MHz, $CDCl_3$/TMS) δ (ppm): 4.79 (2H, s), 7.50 (2H, d, *J* = 5.4 Hz), 7.57–7.61 (1H, m), 7.64–7.71 (4H, m), 7.88 (1H, d, *J* = 7.3 Hz); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$/TMS) δ (ppm): 50.4, 87.9, 121.0, 122.6, 124.2, 128.5, 132.3, 132.9, 138.0, 139.3, 139.8, 167.5; LRMS (EI) *m/z*: 335 (M^+); HRMS: Calcd. for $C_{14}H_{10}INO$: 334.9807, found: 334.9795; IR (neat): 1683, 1576, 1490, 1377, 1333, 1303, 1158, 1149 cm^{-1}.

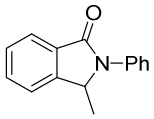
Methyl 4-(1-oxoisoindolin-2-yl)benzoate (2o)



2o was obtained in 74% yield (19.8 mg), recrystallized from acetone/hexane, colorless prisms, mp 218–220 °C (lit.[16] mp 201 °C).

^{1}H NMR (600MHz, $CDCl_3$/TMS) δ (ppm): 3.92 (3H, s), 4.90 (2H, s), 7.51–7.54 (2H, m), 7.63 (1H, t, *J* = 7.6 Hz), 7.93 (1H, d, *J* = 7.6 Hz), 8.00 (2H, d, *J* = 8.2 Hz), 8.10 (2H, d, *J* = 8.9 Hz); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$/TMS) δ (ppm): 50.5, 52.0, 118.0, 122.7, 124.4, 125.5, 128.6, 130.9, 132.6, 132.8, 139.9, 143.5, 166.6, 167.8; LRMS (EI) *m/z*: 267 (M^+); HRMS: Calcd.for $C_{16}H_{13}NO_3$: 267.0895, found: 267.0864; IR (neat): 1684, 1601, 1558, 1517, 1383, 1266, 1148 cm^{-1}.

3-Methyl-2-phenylisoindolin-1-one (2p)



2p was obtained as colorless oil (46% yield, 10.2 mg).

^{1}H NMR (400MHz, $CDCl_3$/TMS) δ (ppm): 1.46 (3H, d, *J* = 6.8 Hz), 5.21 (1H, q, *J* = 6.8 Hz), 7.22–7.24 (1H, m), 7.46 (2H, t, *J* = 8.3 Hz), 7.51 (2H, t, *J* = 8.3 Hz), 7.58–7.63 (3H, m), 7.93 (1H, d, *J* = 7.8 Hz); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$/TMS) δ (ppm): 18.7, 56.9, 121.9, 123.3, 124.1, 125.3, 128.3, 129.0, 131.7, 132.0, 137.0, 146.2, 166.8; LRMS (EI) *m/z*: 223 (M^+); HRMS: Calcd. for $C_{15}H_{13}NO$: 223.0997, found: 223.1003; IR (neat): 1684, 1597, 1497, 1367, 1298, 1107 cm^{-1}.

7. References

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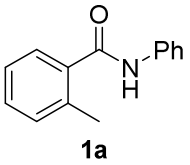
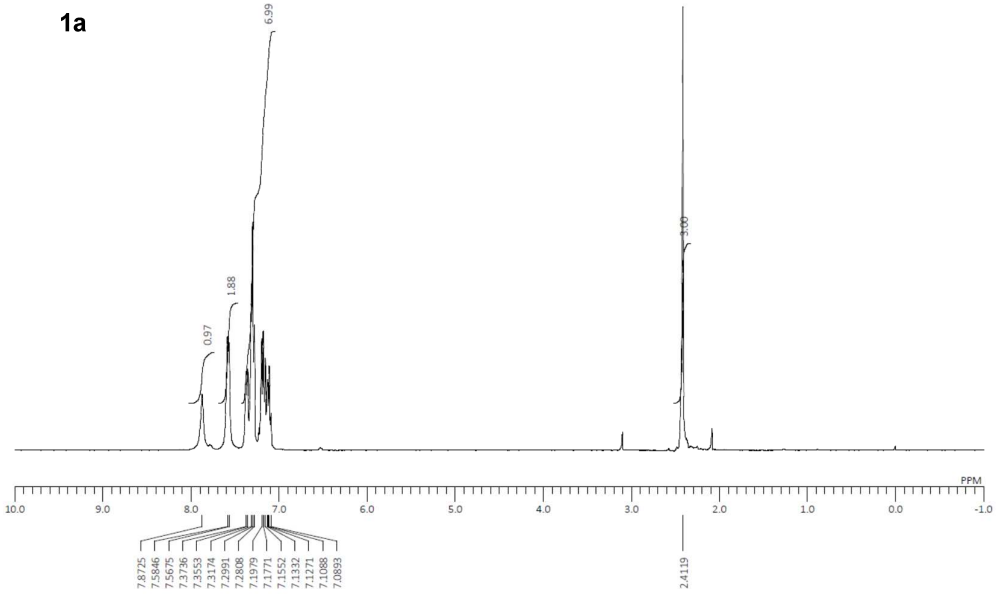
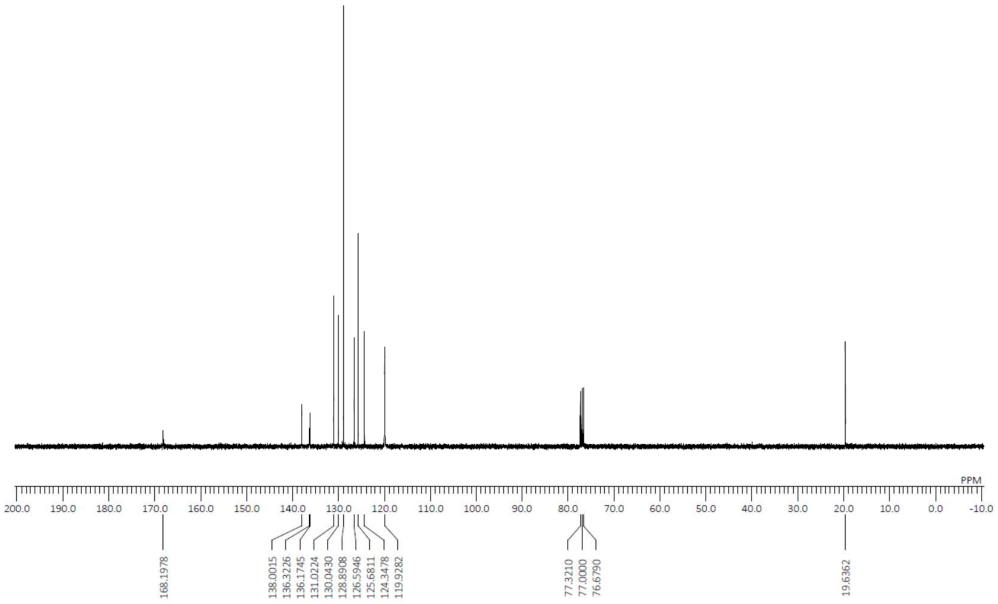
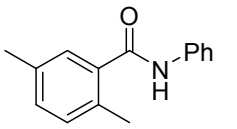
8. ^{1}H- and ^{13}C-NMR Spectra of 1a–p and 2a–p



1a







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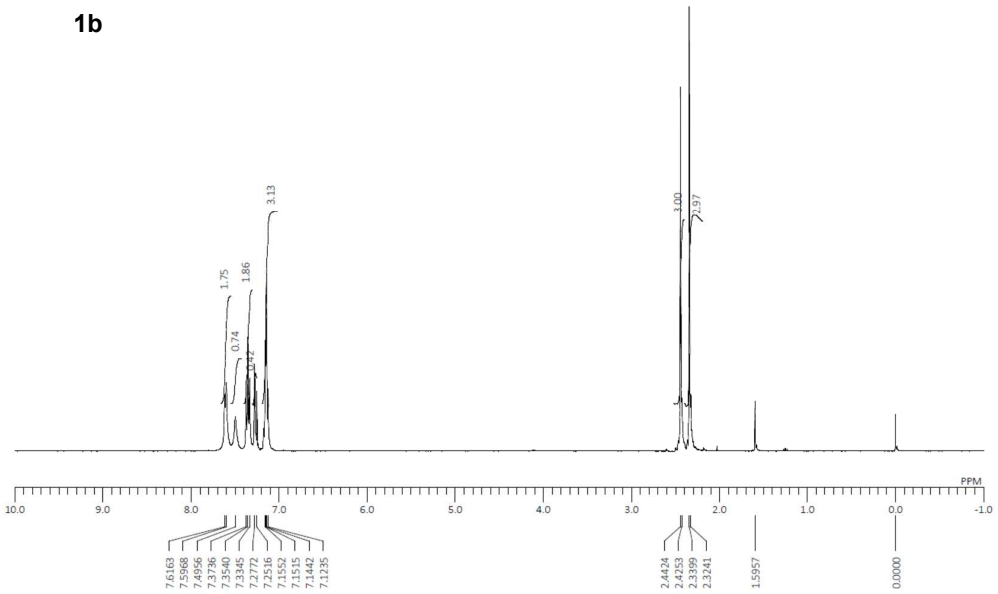
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6.0
5.0
4.0
3.0
2.0
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0.0
-1.0
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7.5968
7.4956
7.3736
7.3540
7.3345
7.2772
7.2516
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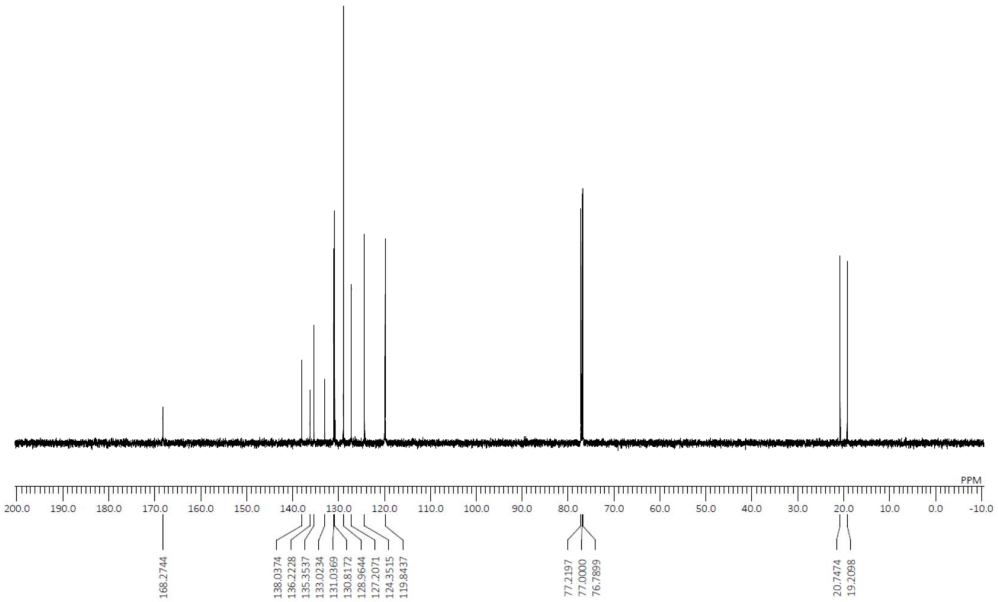
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80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0
-10.0
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138.0374
136.2228
135.3537
133.0234
131.0369
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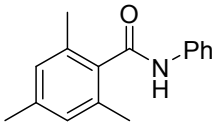
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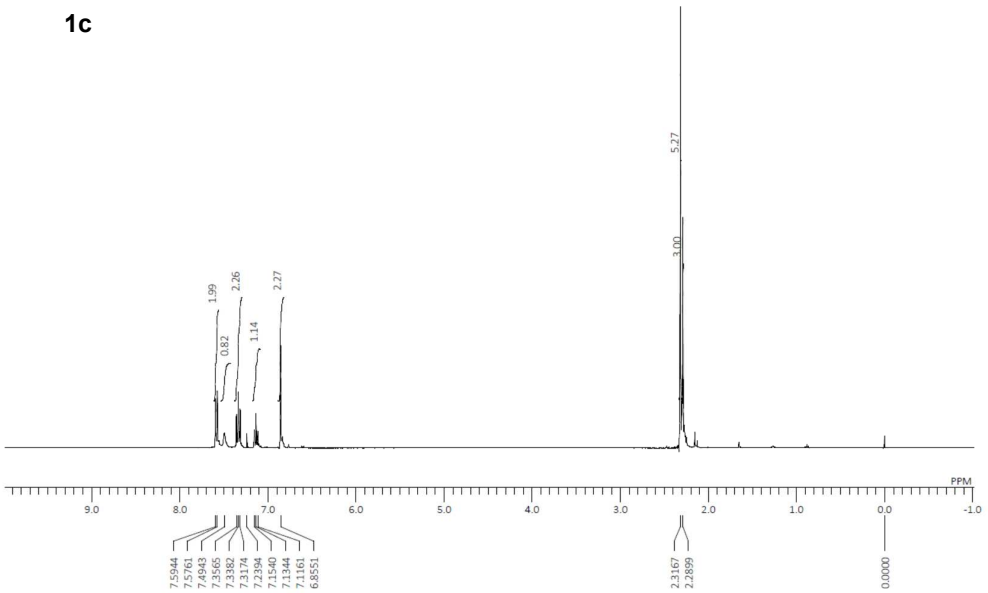
Ph

1c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3.0
2.0
1.0
0.0
-1.0
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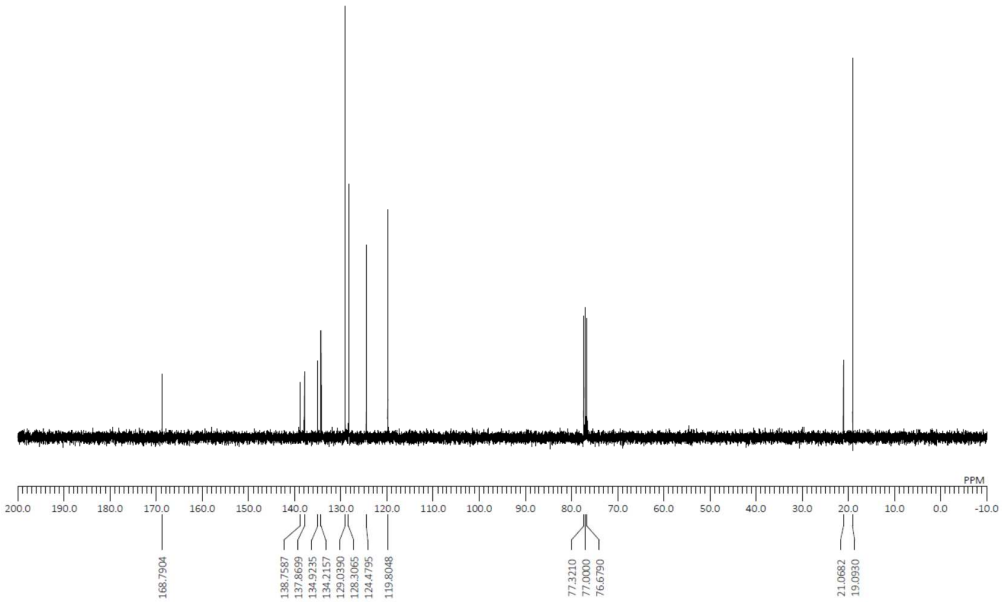
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70.0
60.0
50.0
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10.0
0.0
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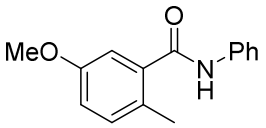
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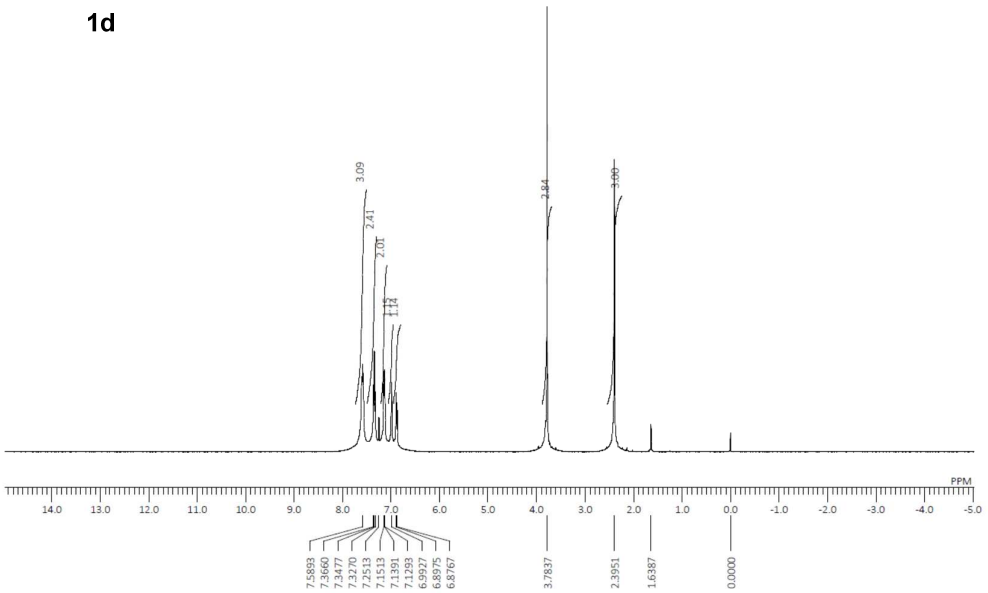
MeO
N
H
Ph

1d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1.14
3.84
3.00
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7.3477
7.3270
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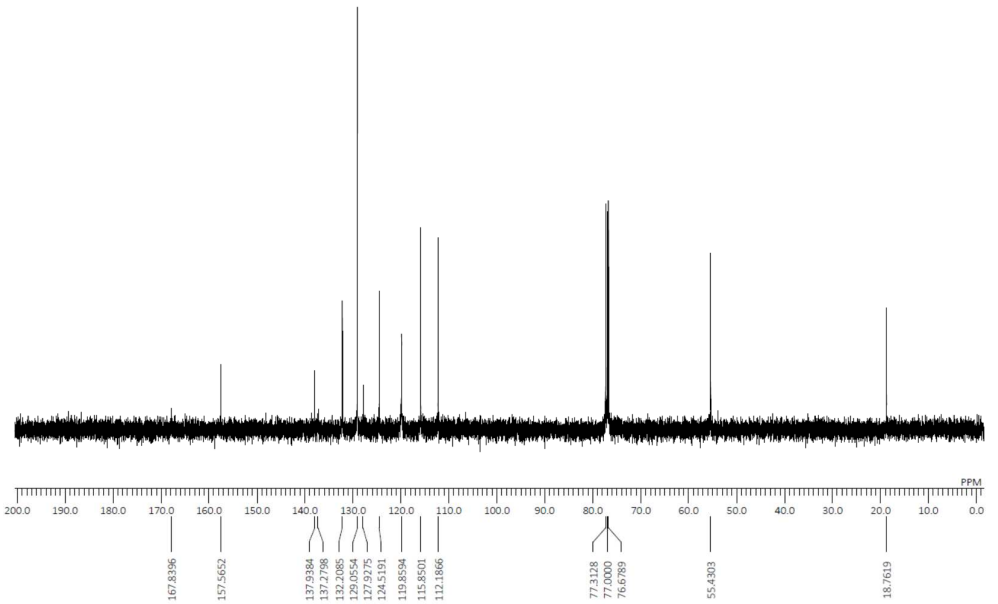
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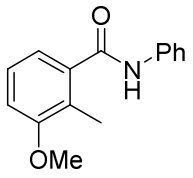
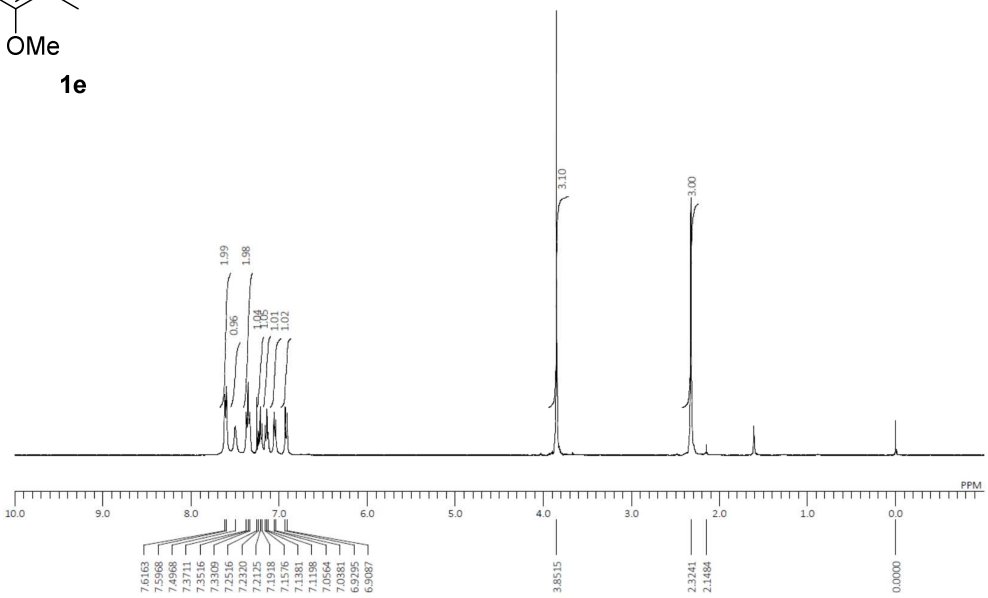

O
N
H
Ph
OMe

1e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3.10
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8.0
7.0
6.0
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2.0
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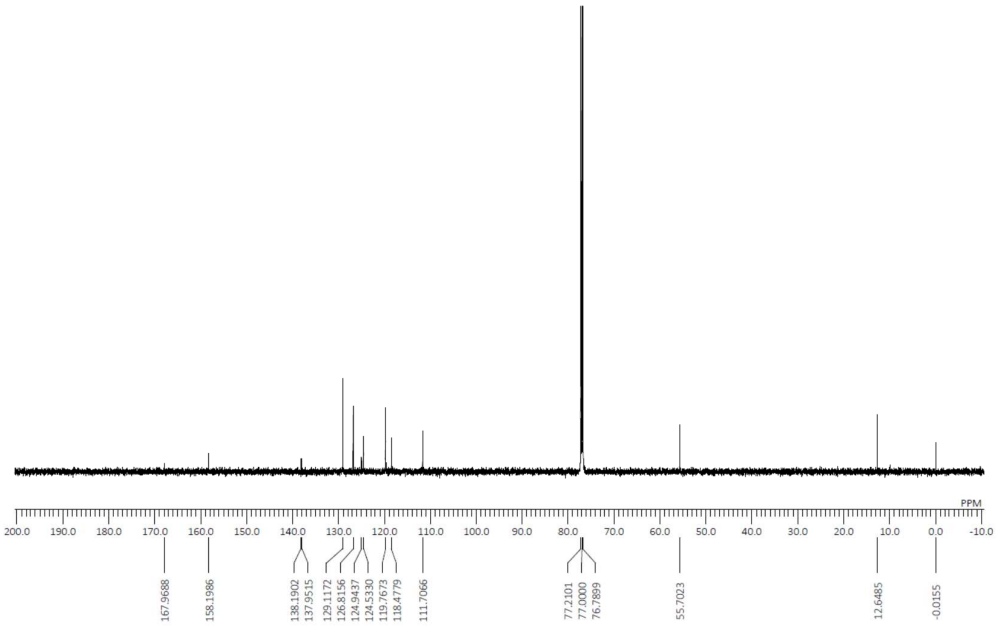
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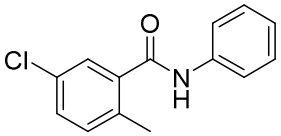
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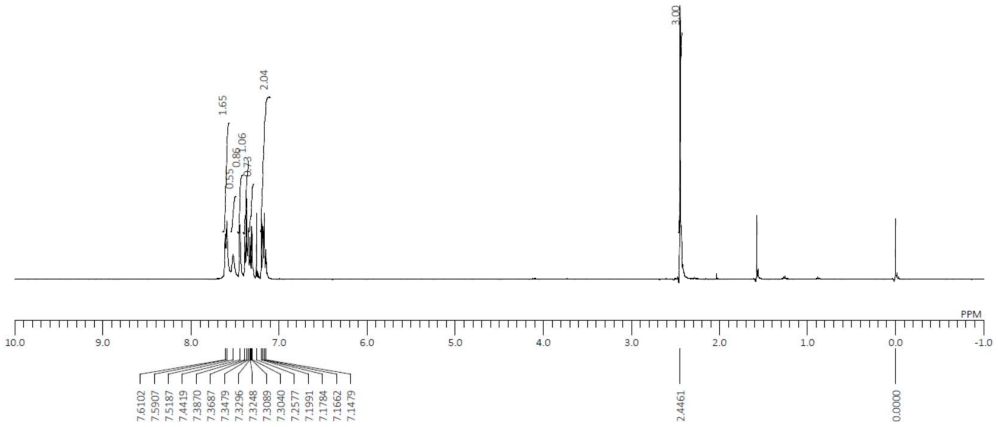
O
N
H

1f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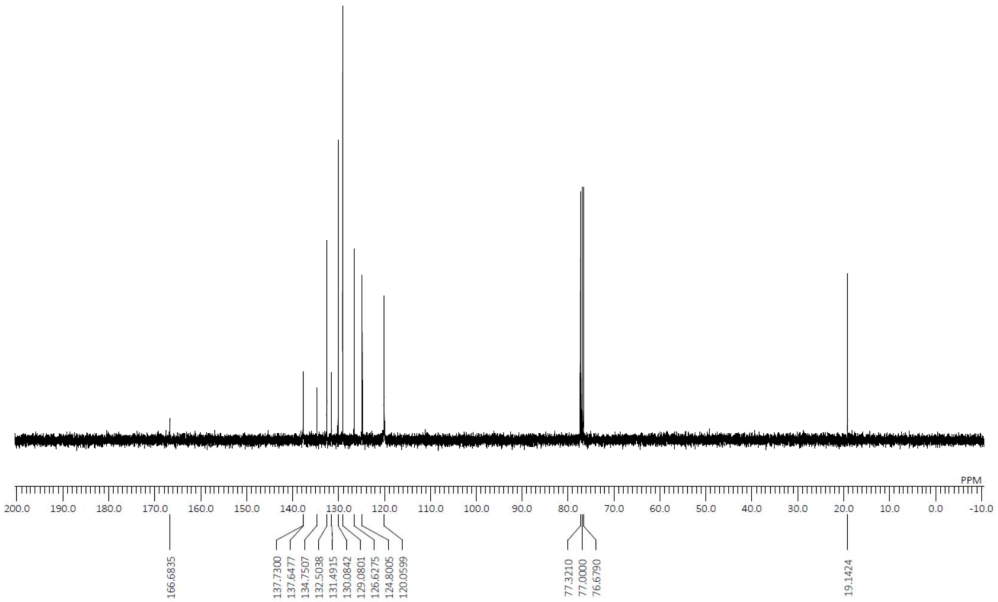
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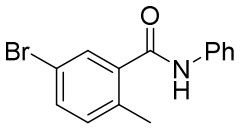
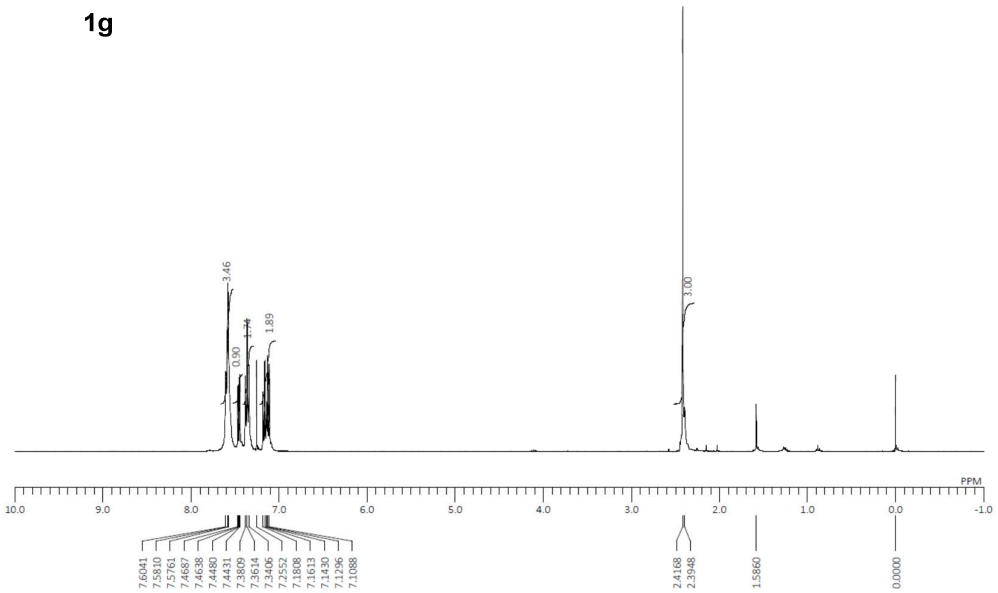

O
Br
N
H
Ph

1g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1.0
0.0
-1.0
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7.5761
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7.4480
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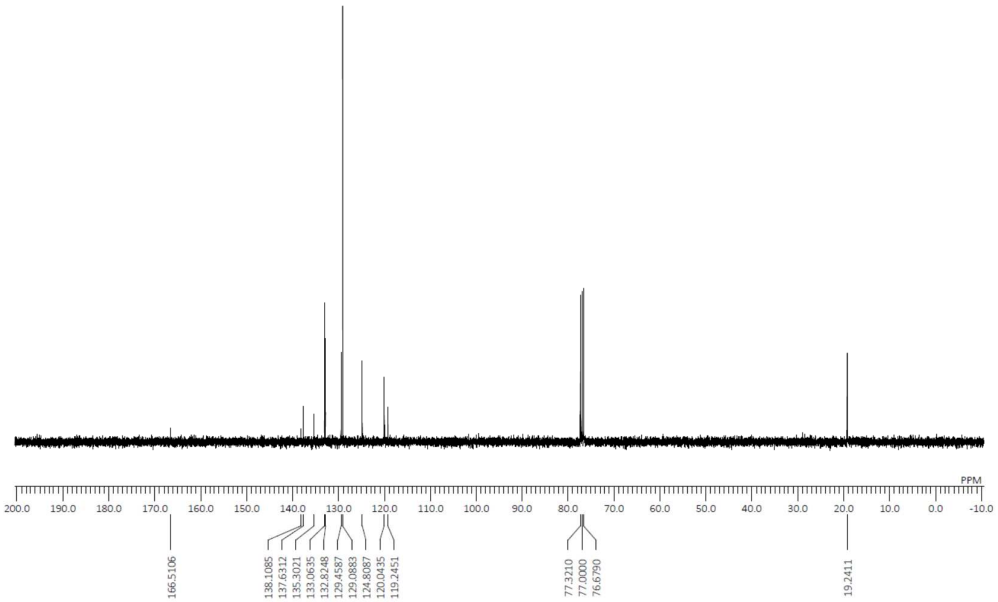
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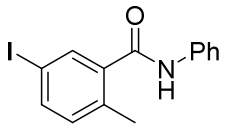
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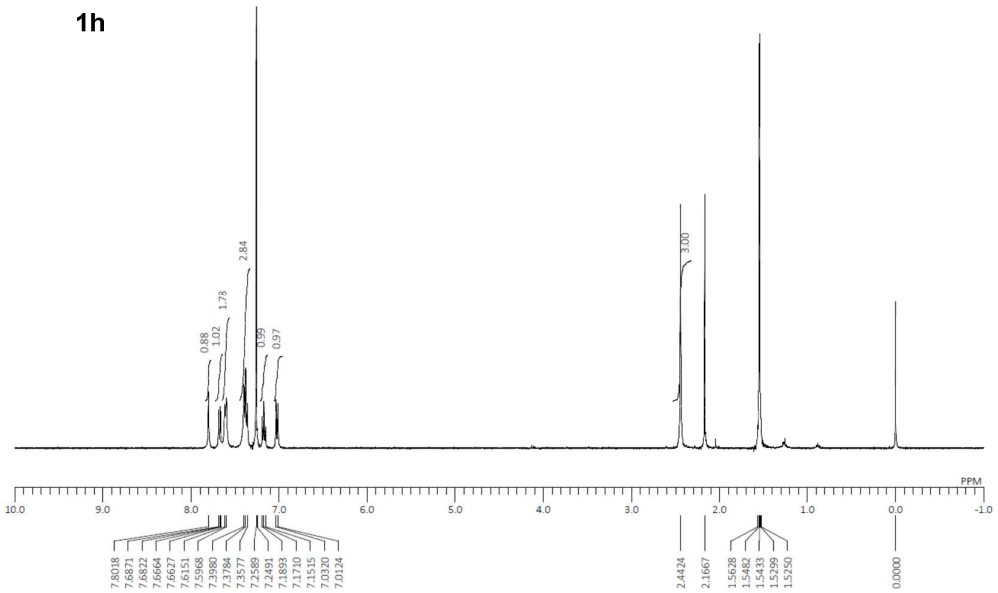
I
N
H
Ph

1h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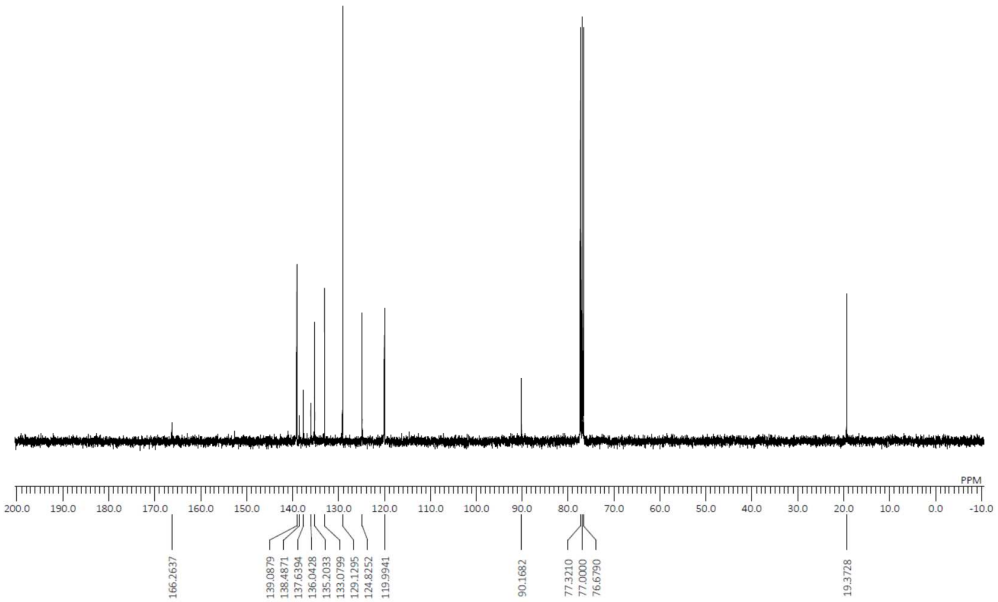
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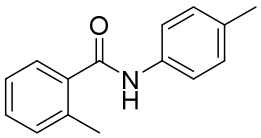

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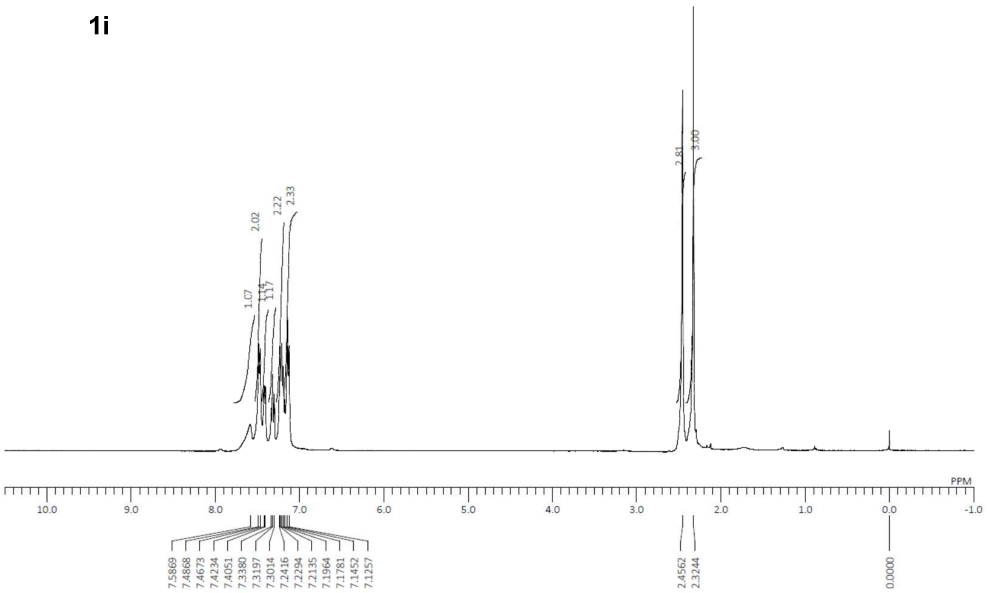
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3.0
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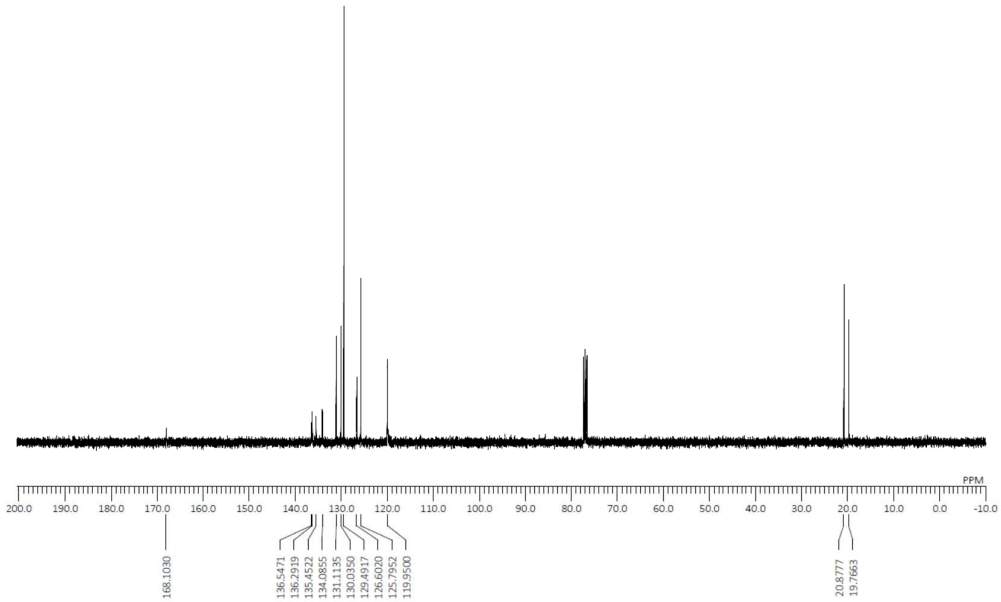
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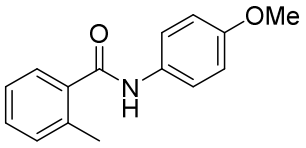
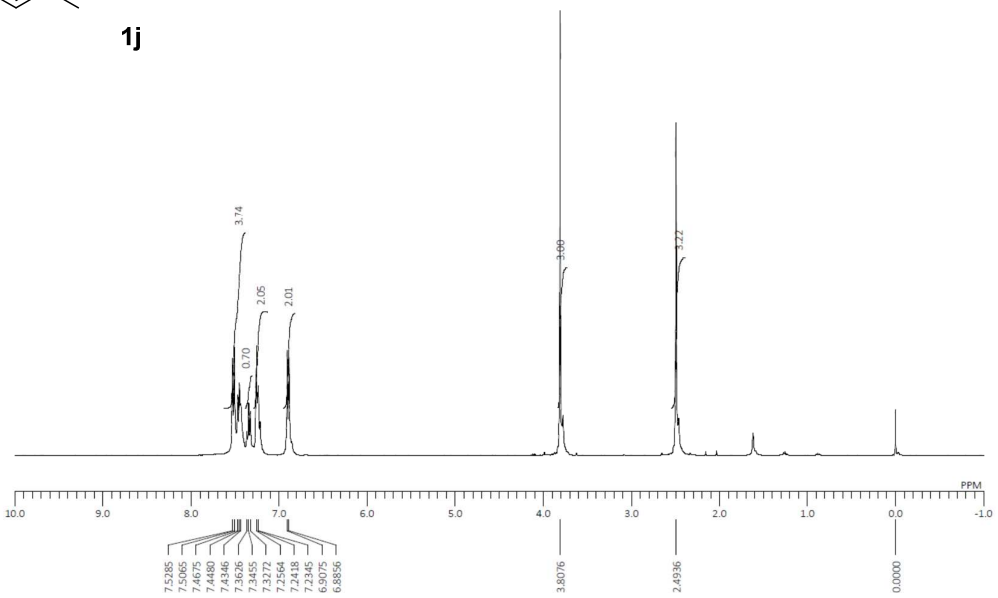

O
N
H
OMe

1j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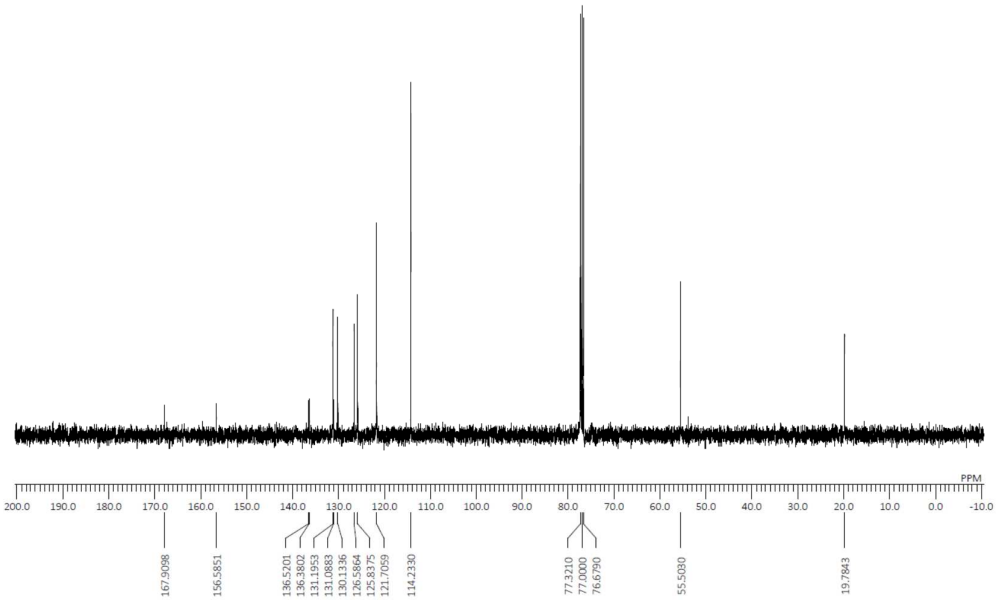
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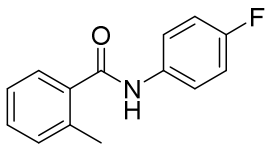
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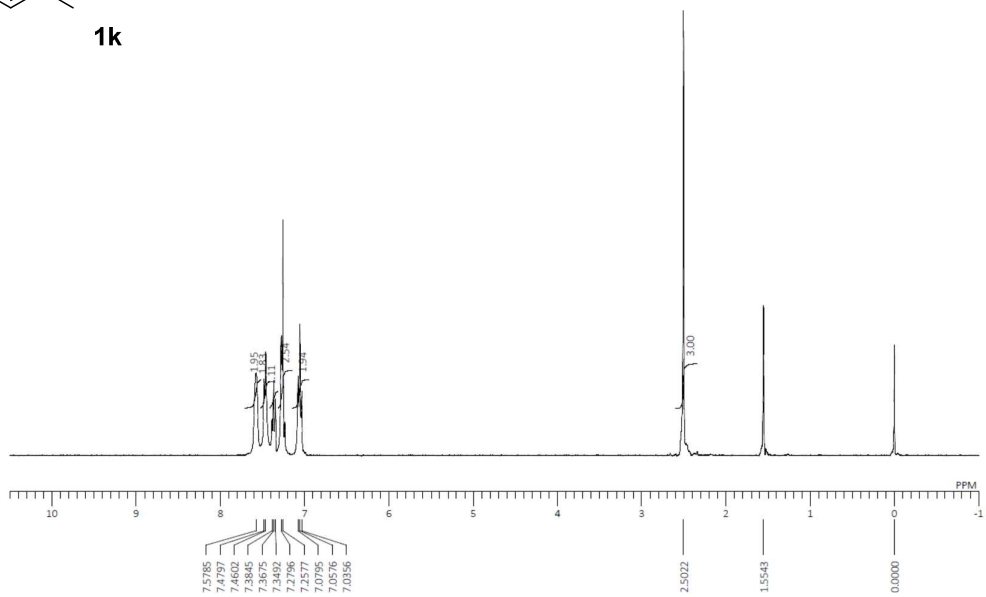
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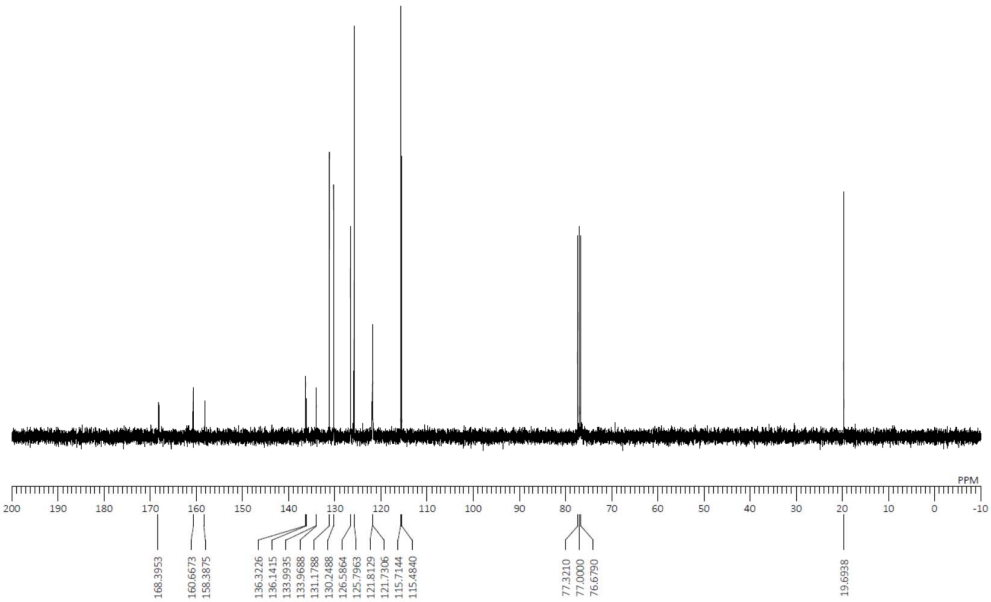
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10
0
-10
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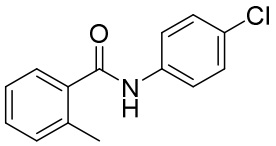
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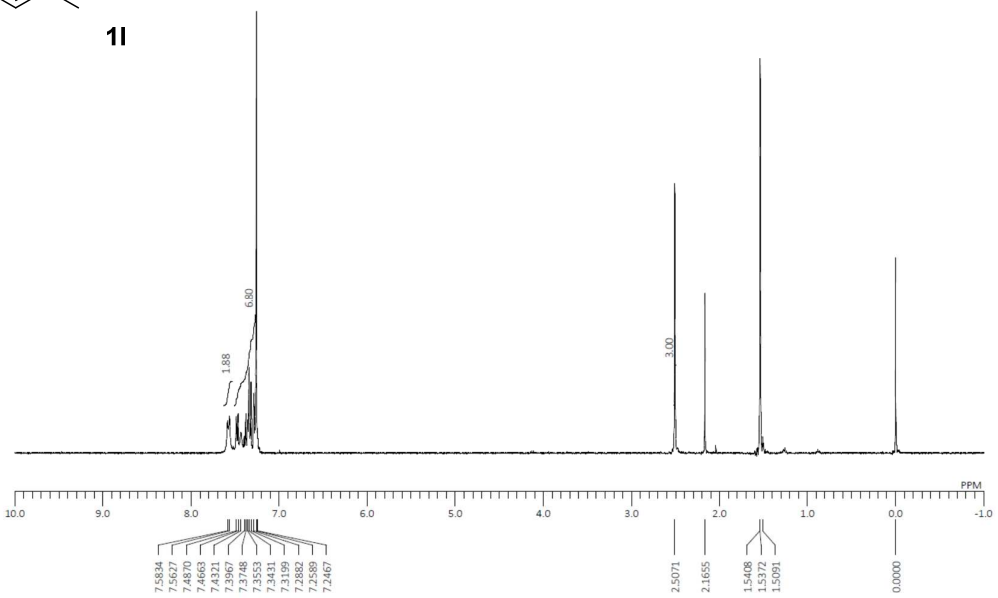
Cl

1l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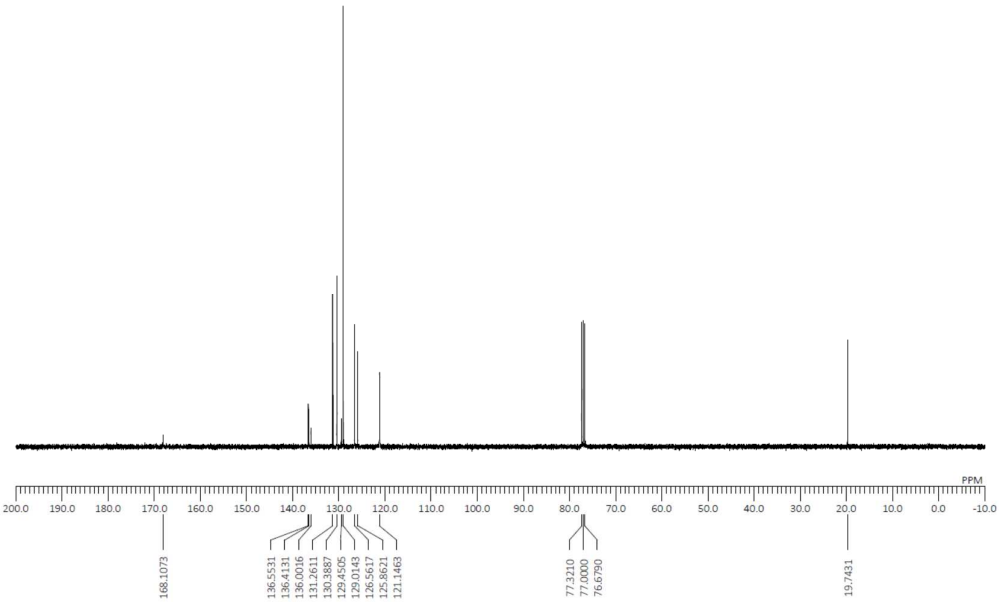
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10.0
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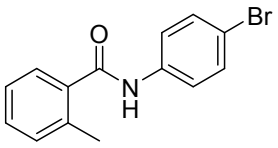
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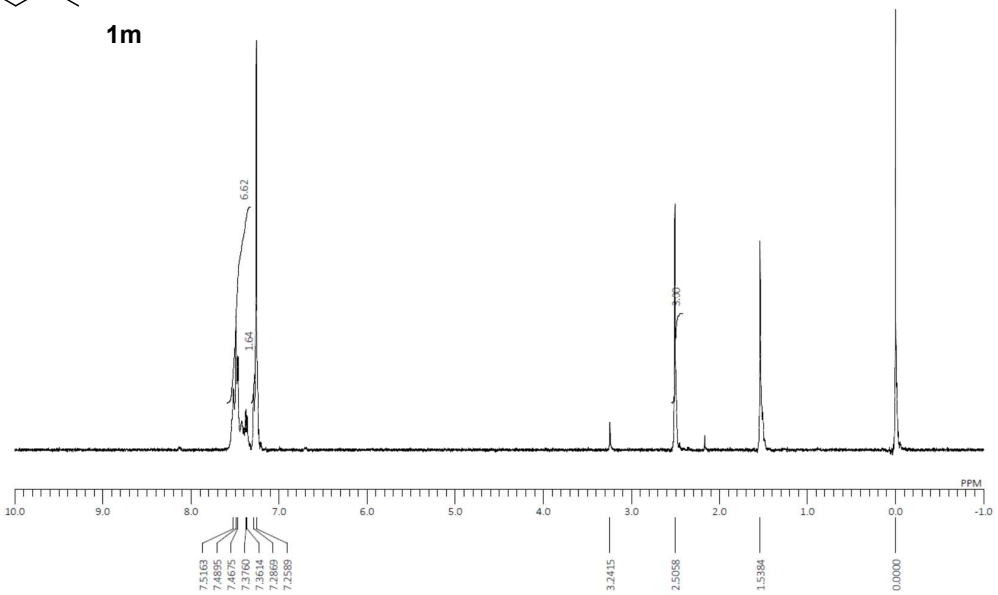
Br

1m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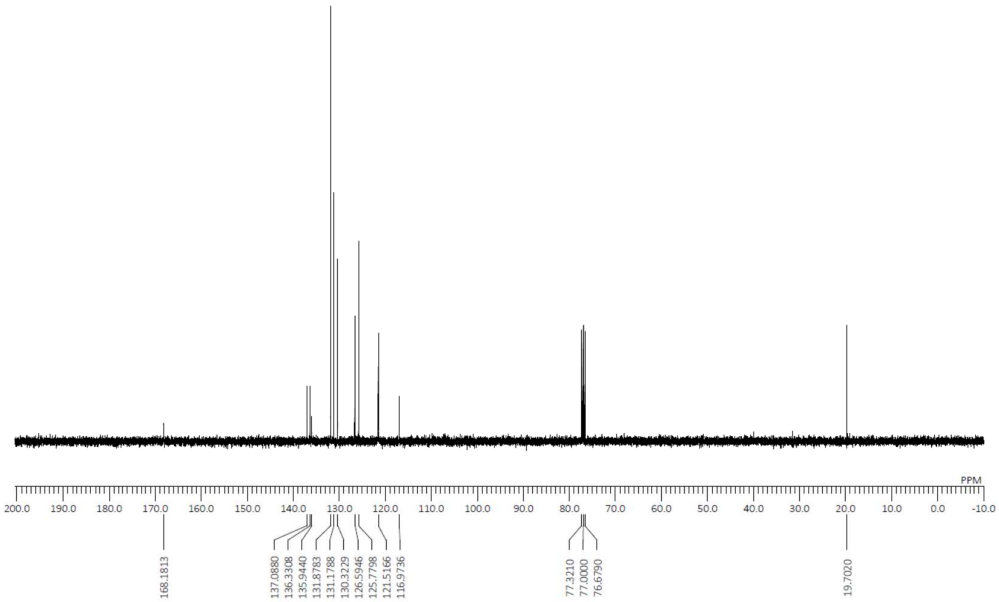
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80.0
70.0
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40.0
30.0
20.0
10.0
0.0
-10.0
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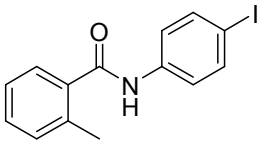
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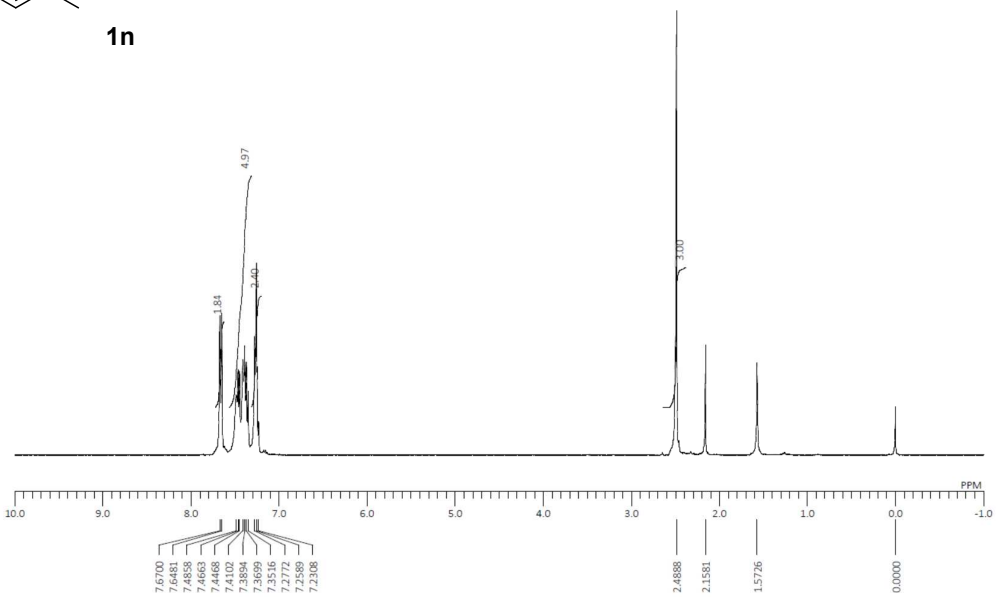
I

1n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1.0
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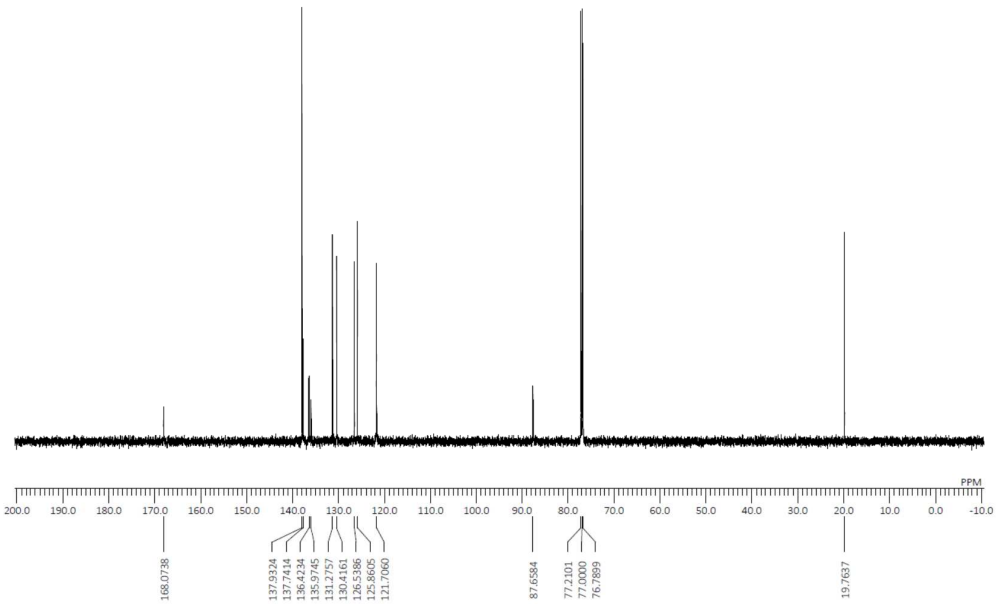
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60.0
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20.0
10.0
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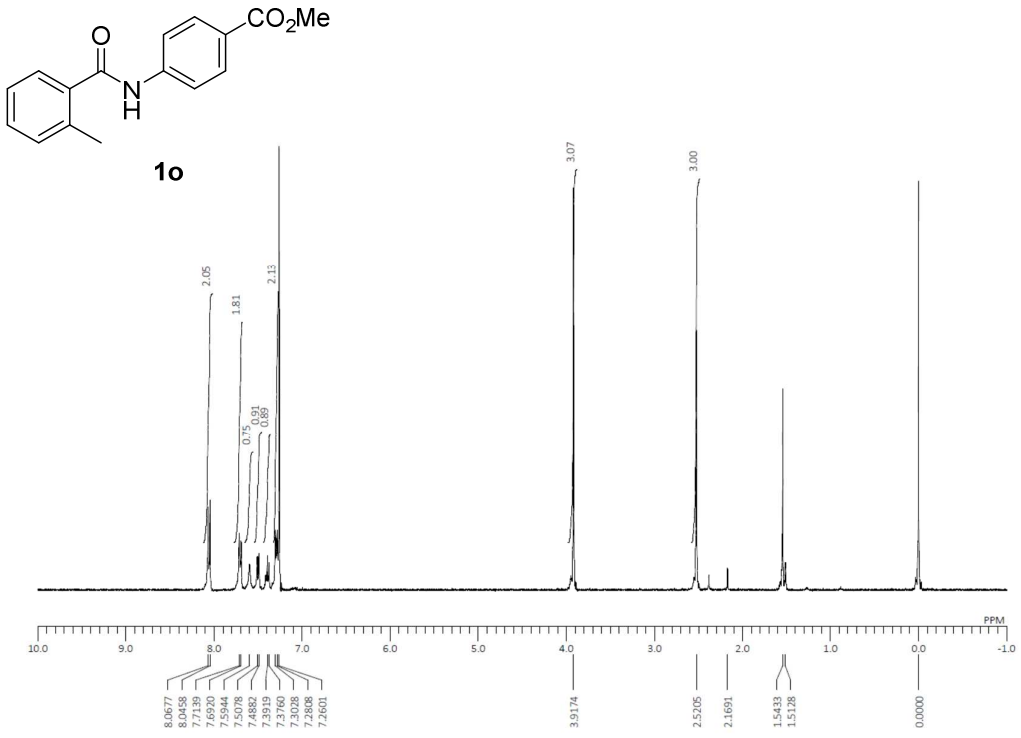
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O
N
H
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3.00
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1.81
2.13
0.75
0.91
0.89
PPM
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8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
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8.0458
7.7139
7.6920
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7.5078
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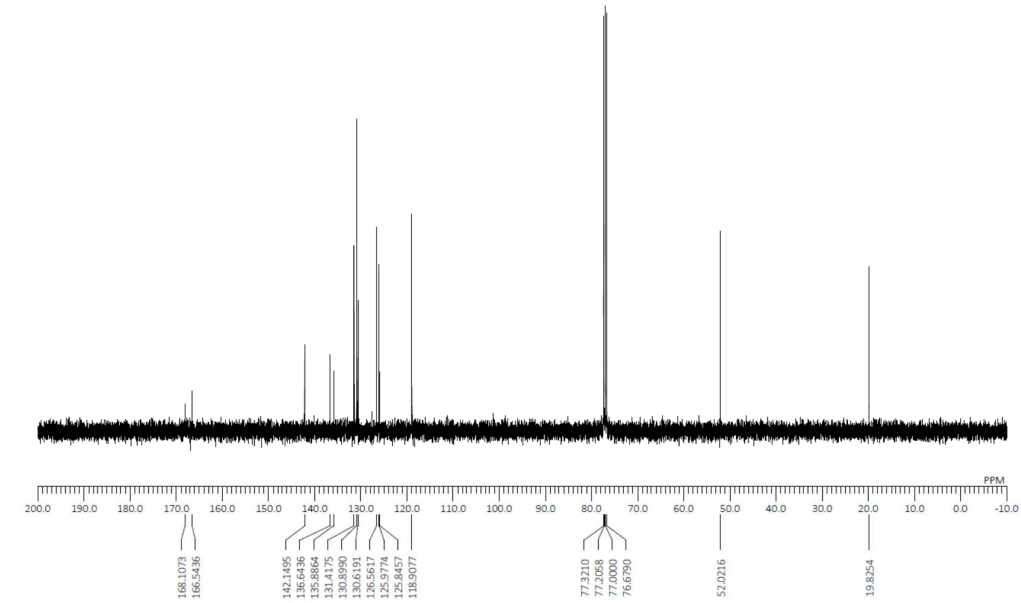
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70.0
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50.0
40.0
30.0
20.0
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0.0
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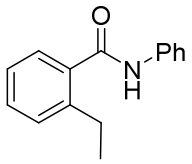
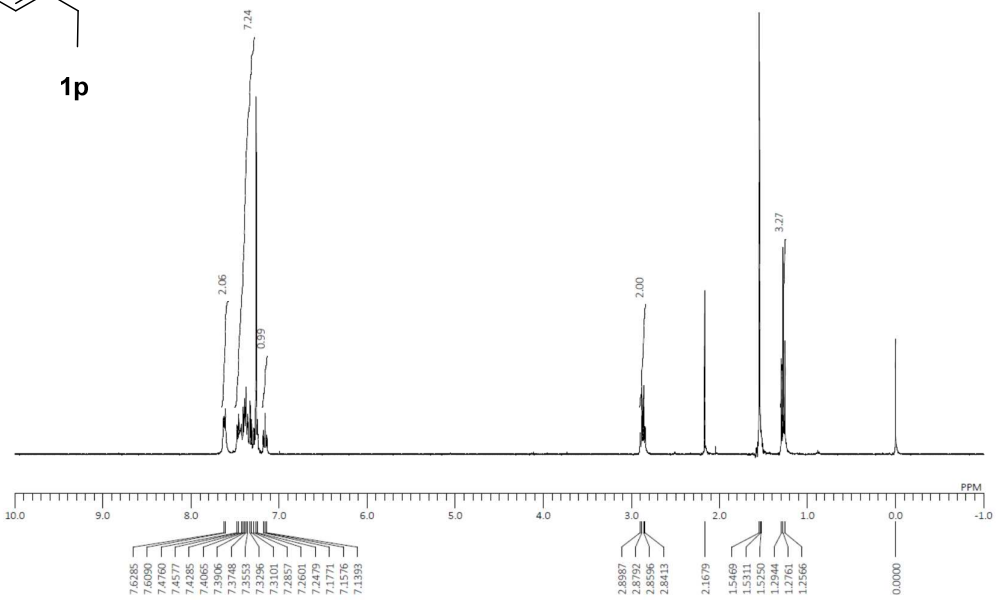

O
N
H
Ph

1p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4.0
3.0
2.0
1.0
0.0
-1.0
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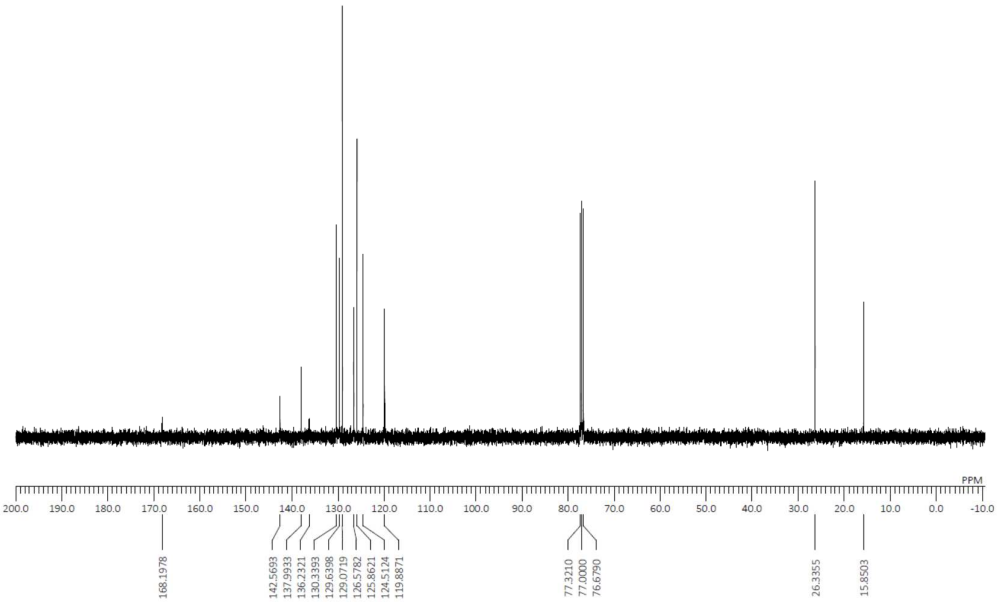
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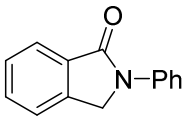
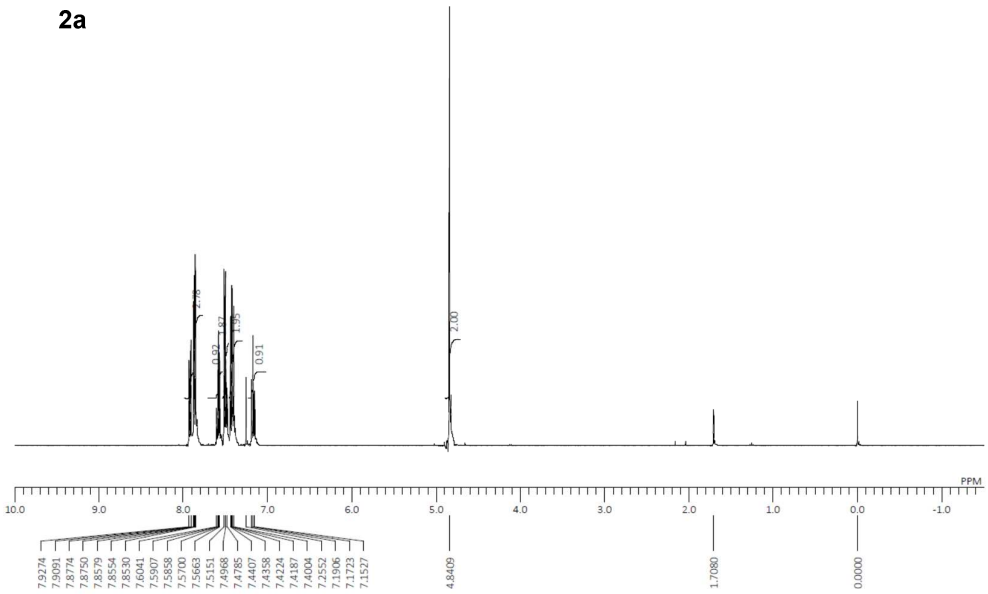

O
N–Ph

2a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7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
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7.9091
7.8774
7.8750
7.8579
7.8554
7.8530
7.6041
7.5907
7.5858
7.5700
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7.4785
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7.1527
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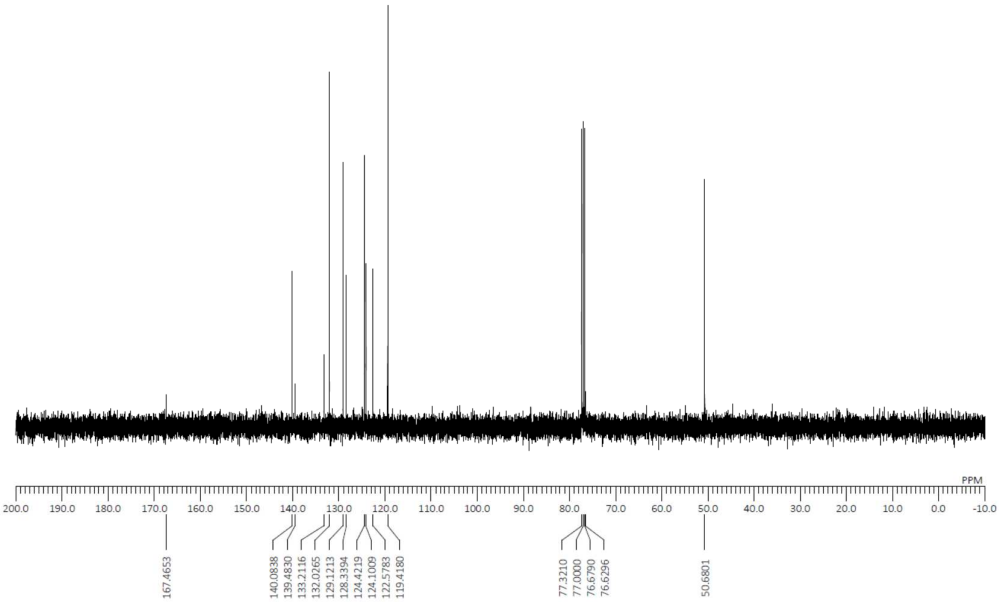
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90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0
-10.0
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139.4830
133.2116
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129.1213
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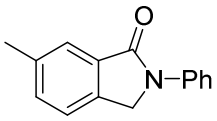
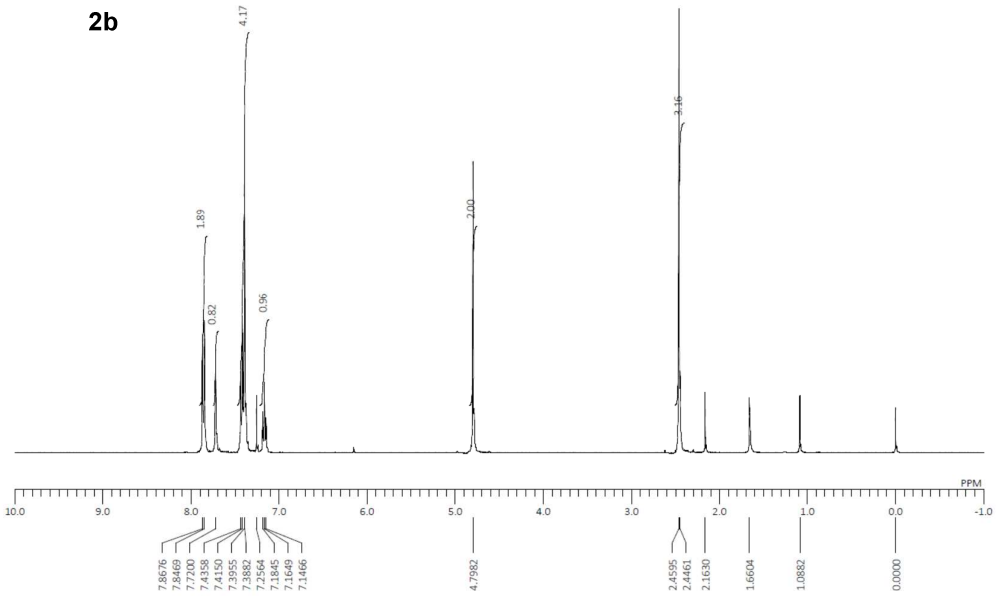

O
N–Ph

2b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7.0
6.0
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4.0
3.0
2.0
1.0
0.0
-1.0
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7.8469
7.7200
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7.3955
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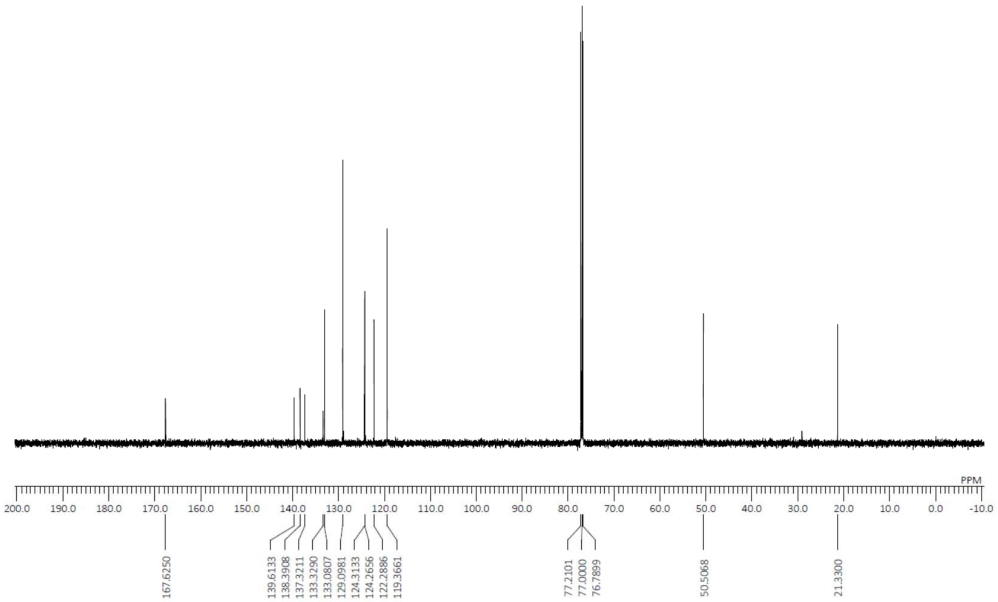
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140.0
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80.0
70.0
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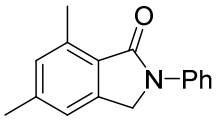
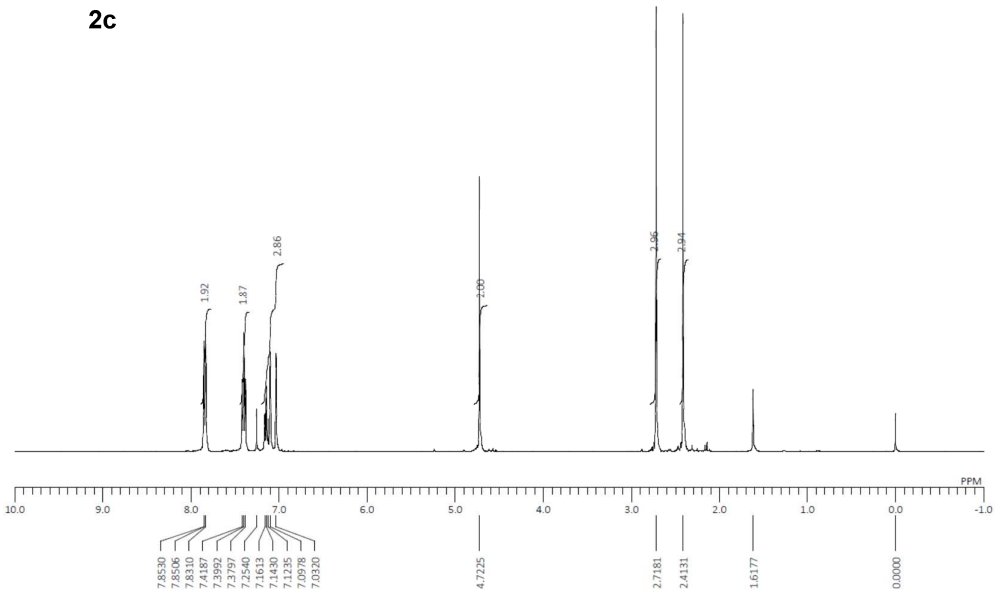

O
N–Ph

2c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2.00
2.96
2.94
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7.0
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5.0
4.0
3.0
2.0
1.0
0.0
-1.0
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7.8506
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7.4187
7.3992
7.3797
7.2540
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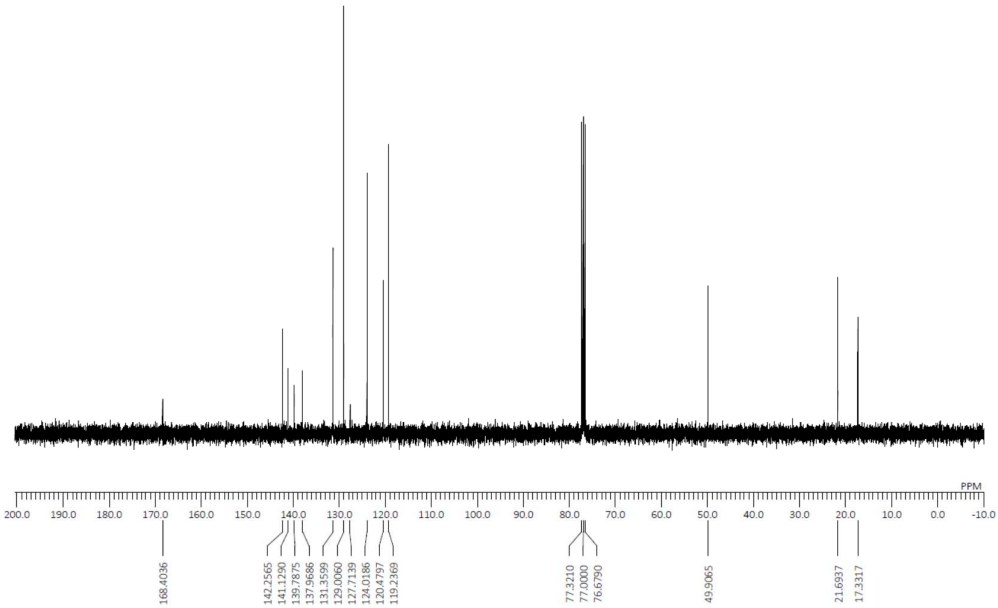
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90.0
80.0
70.0
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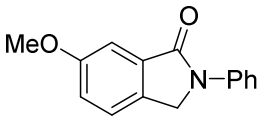
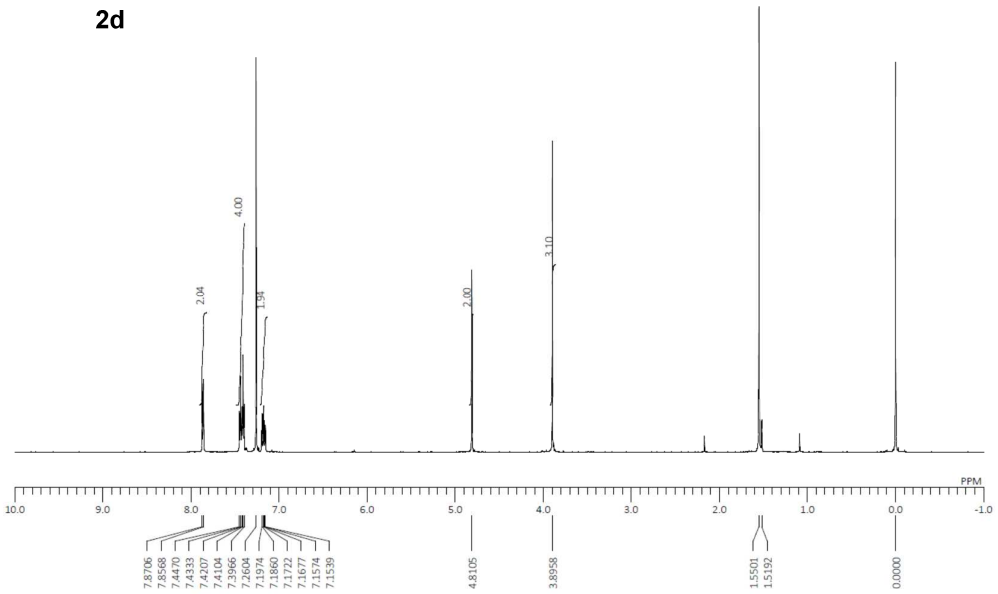

MeO
O
N–Ph

2d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2.00
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4.0
3.0
2.0
1.0
0.0
-1.0
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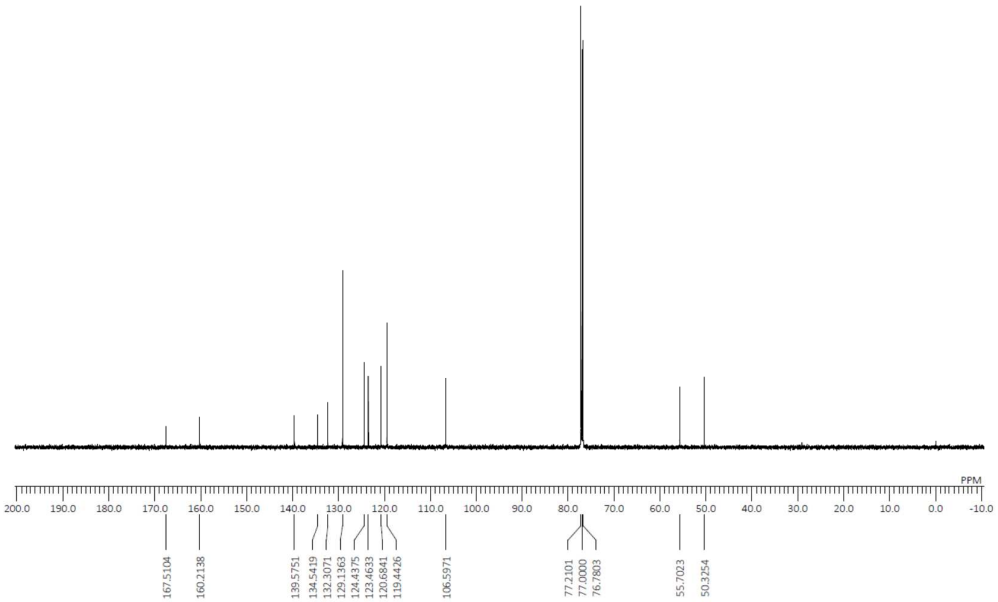
PPM
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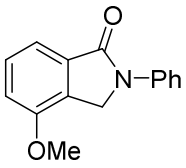
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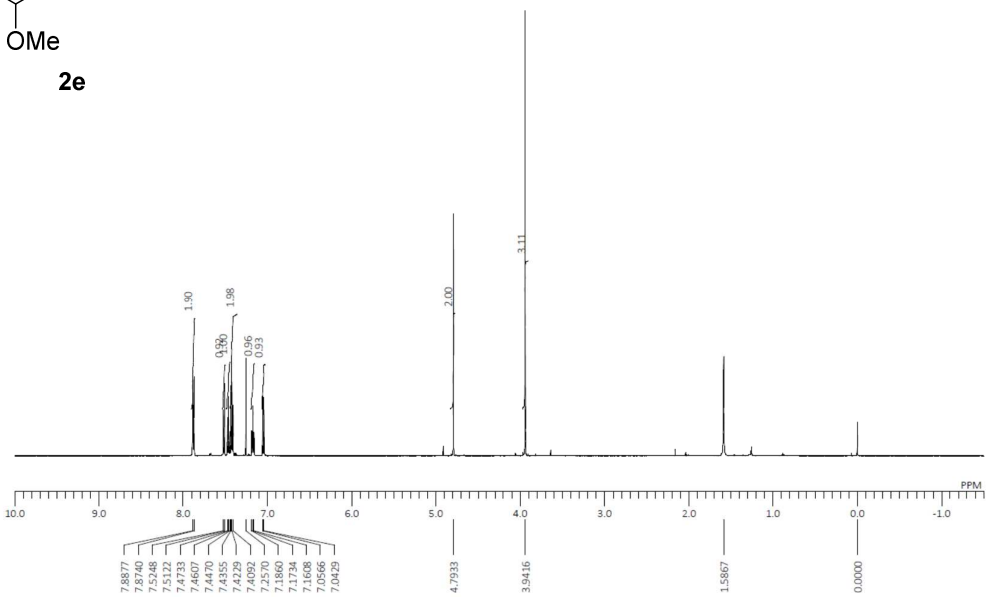
OMe

2e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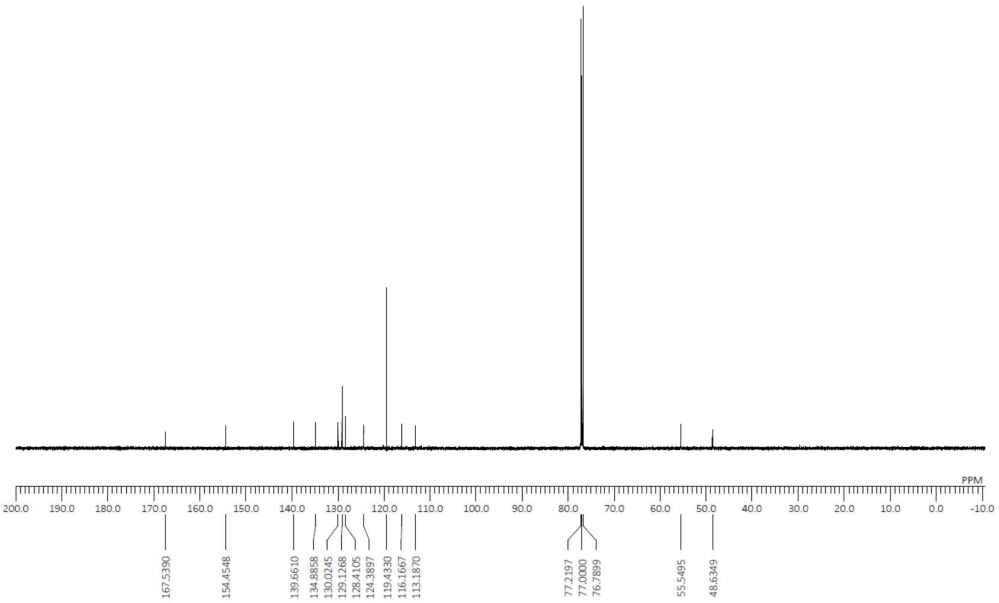
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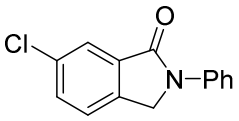
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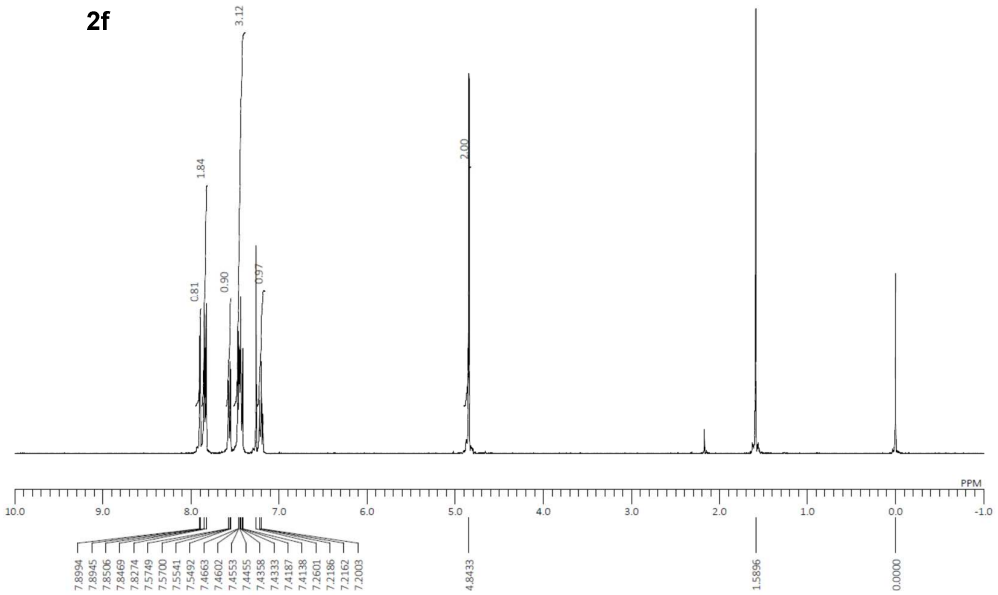
N–Ph

2f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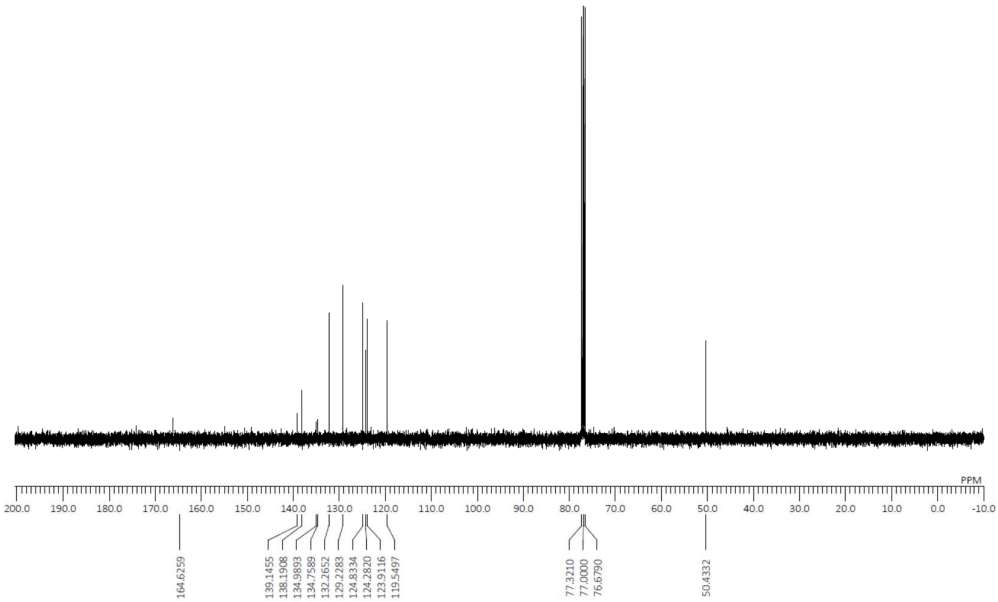
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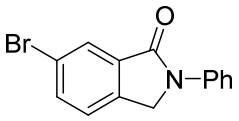
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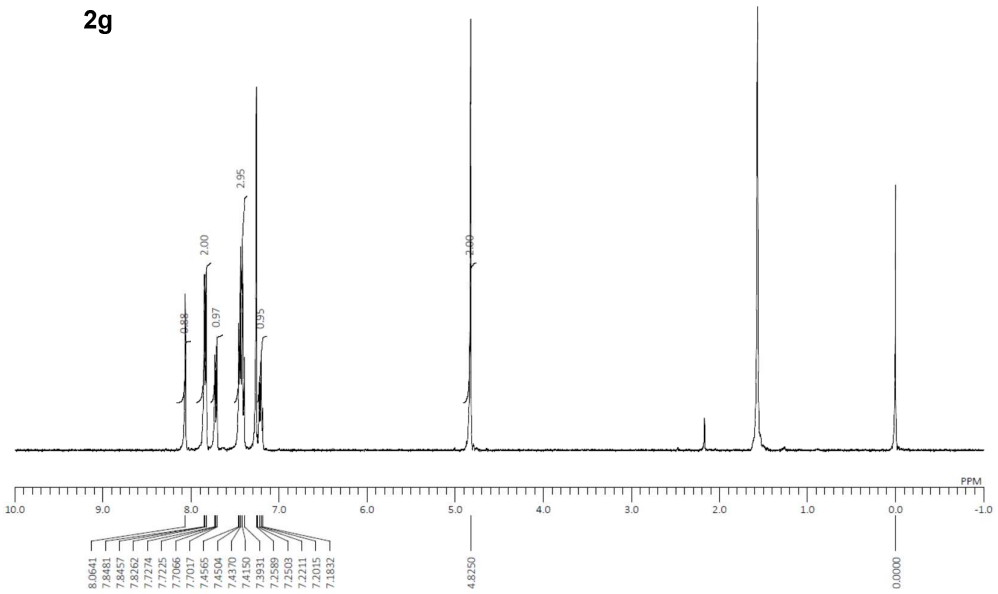
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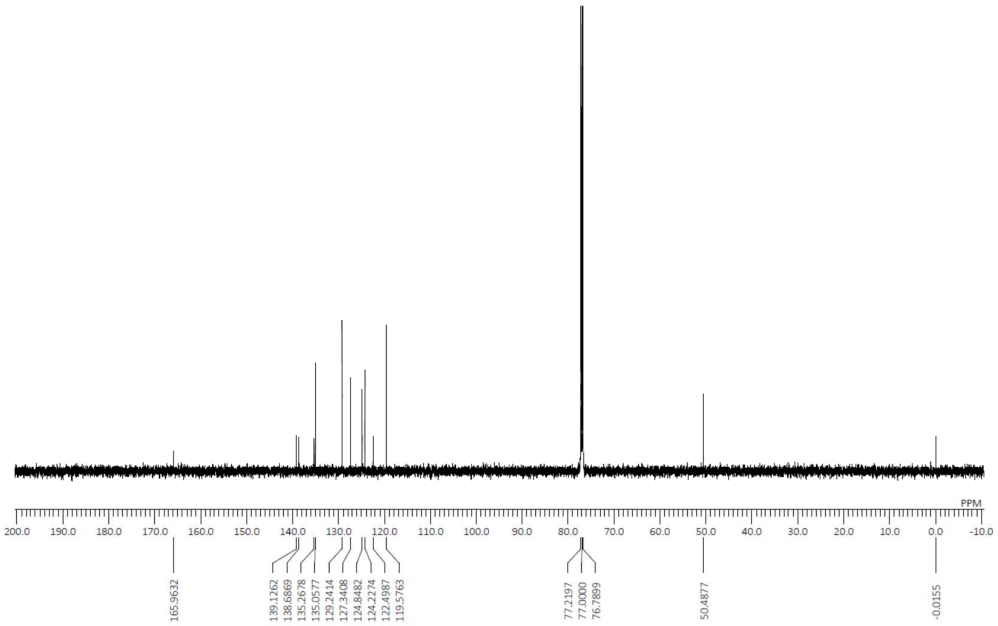
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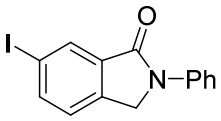
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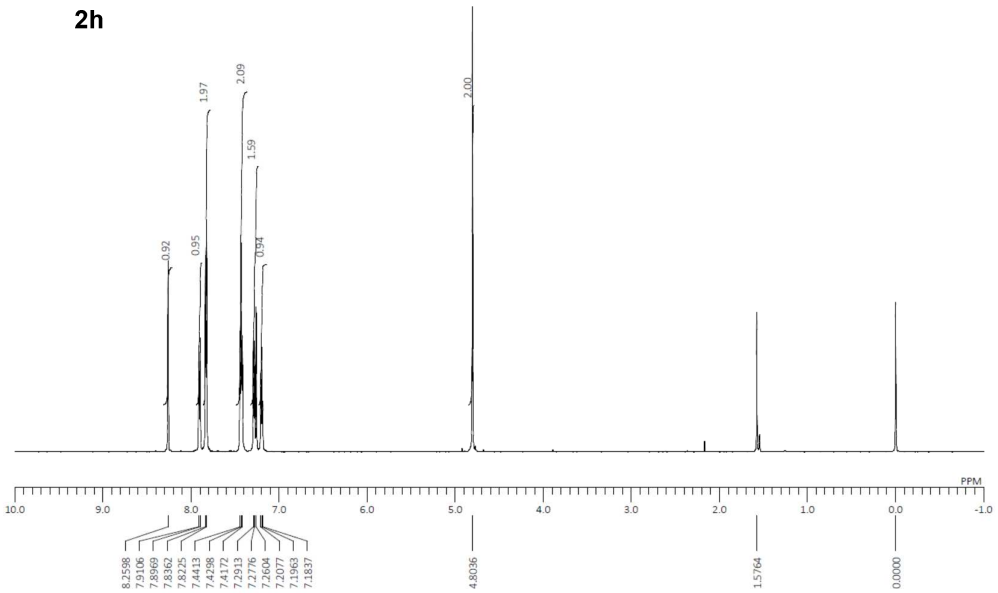
I
N–Ph

2h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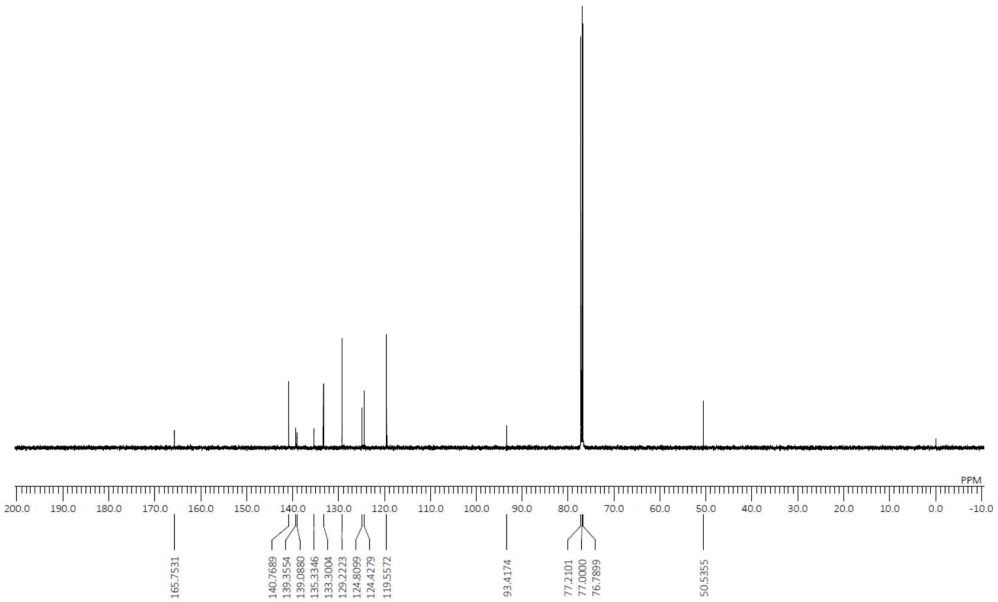
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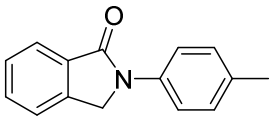

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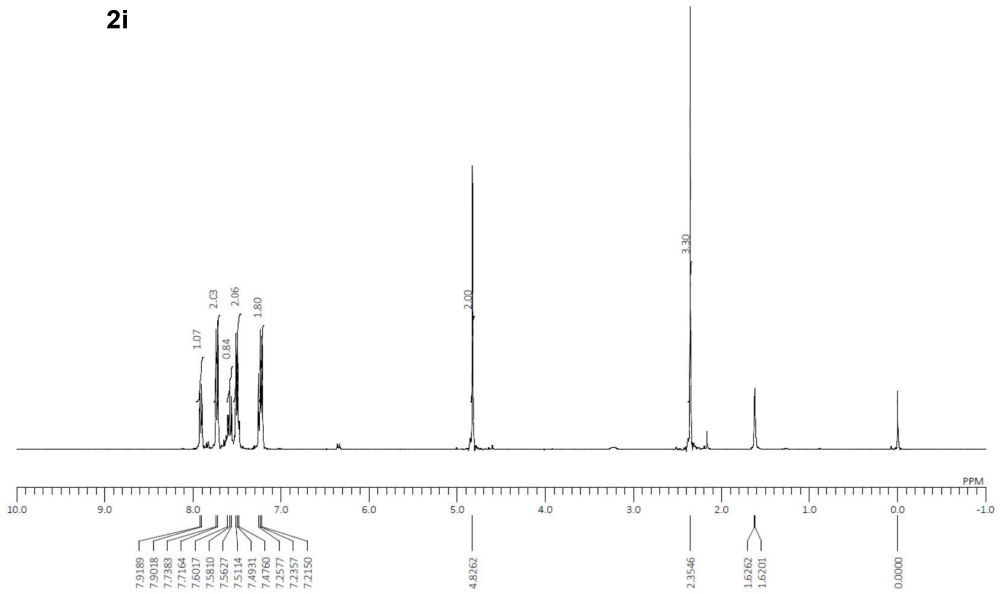
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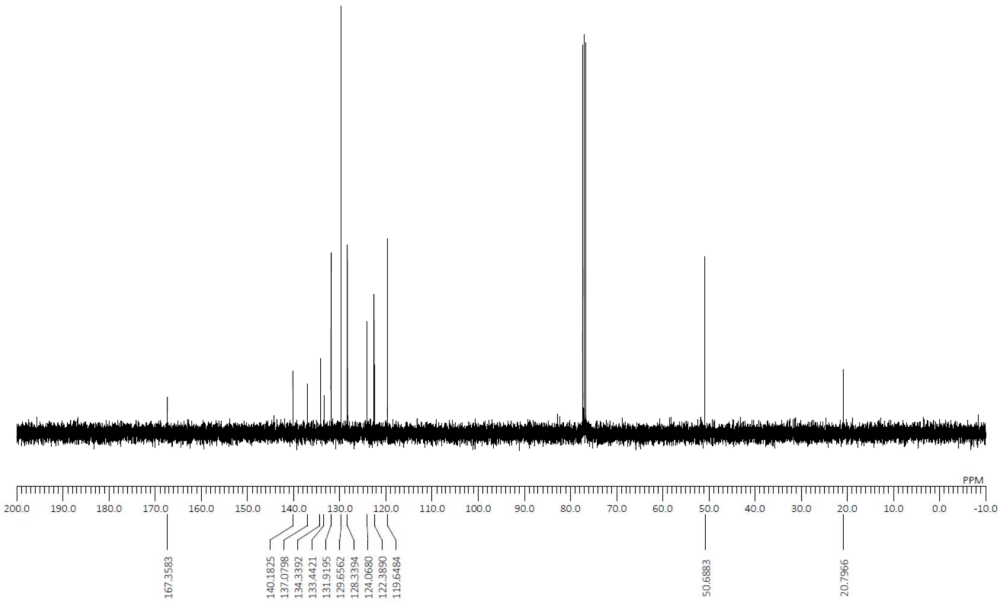
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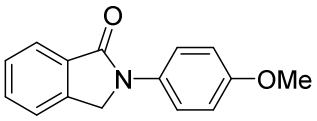
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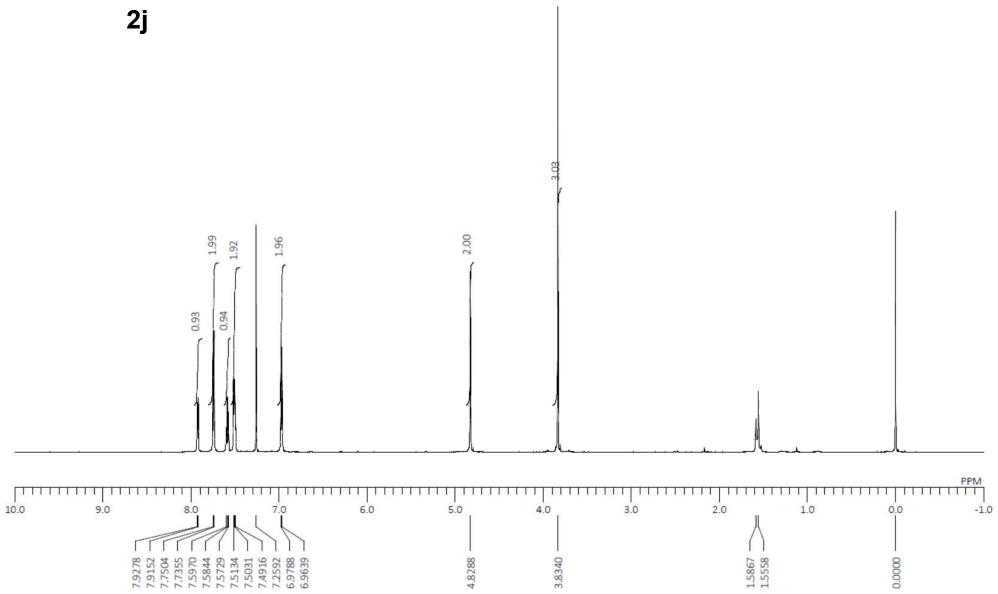
OMe

2j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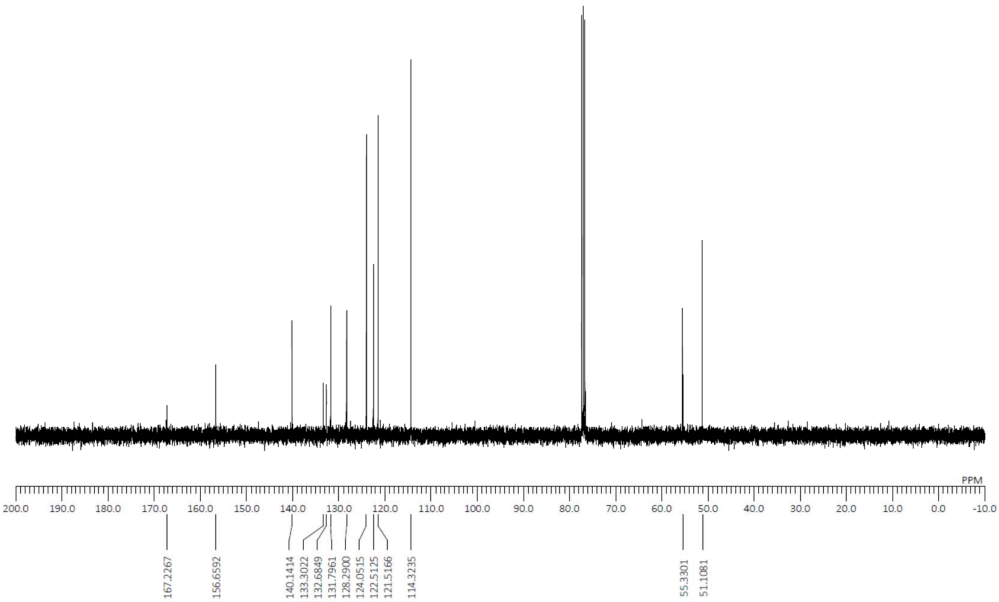
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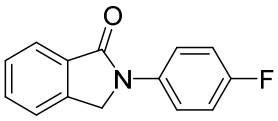
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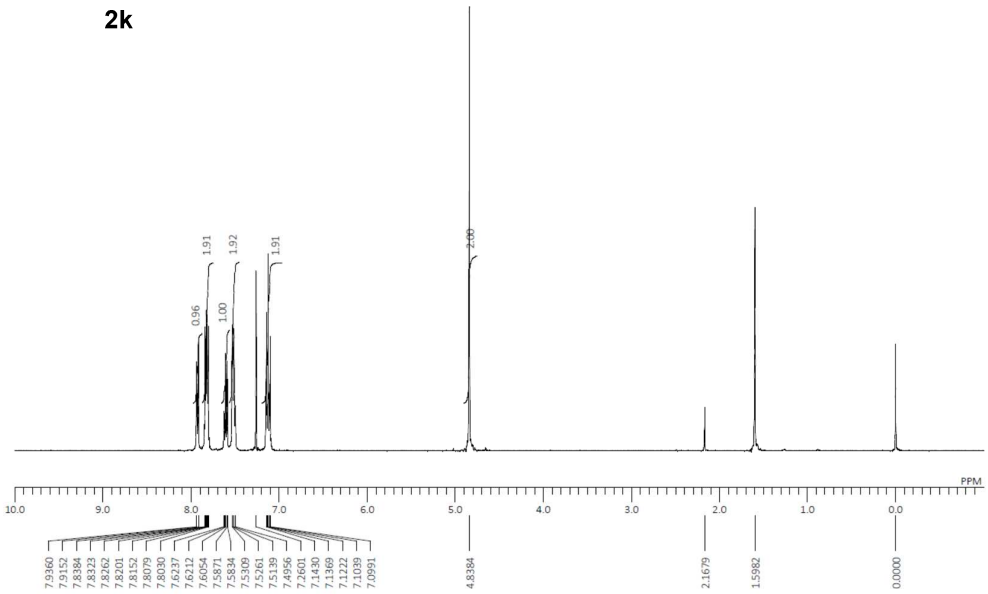
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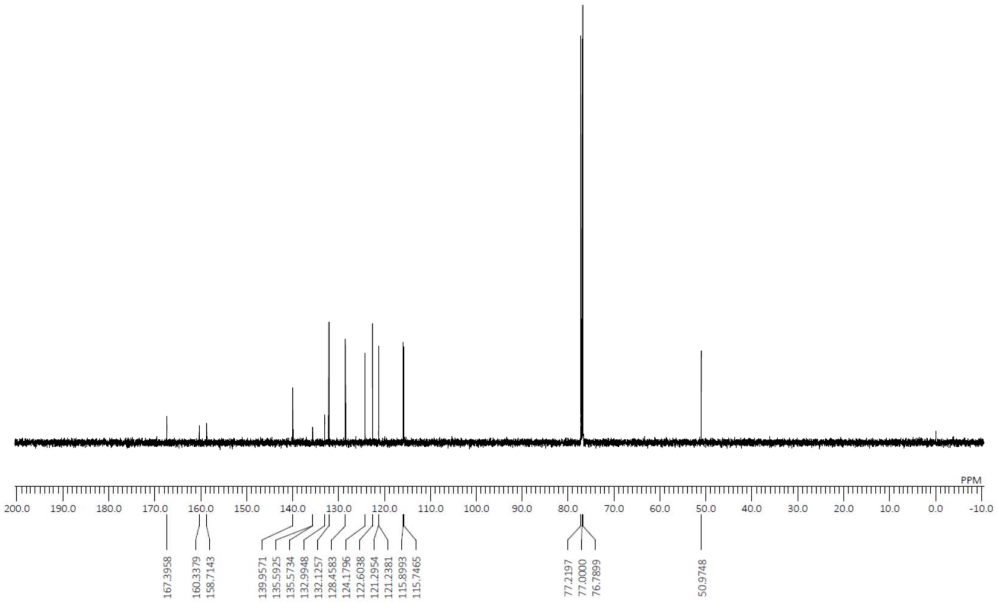
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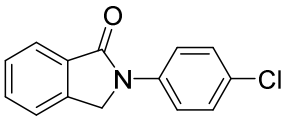
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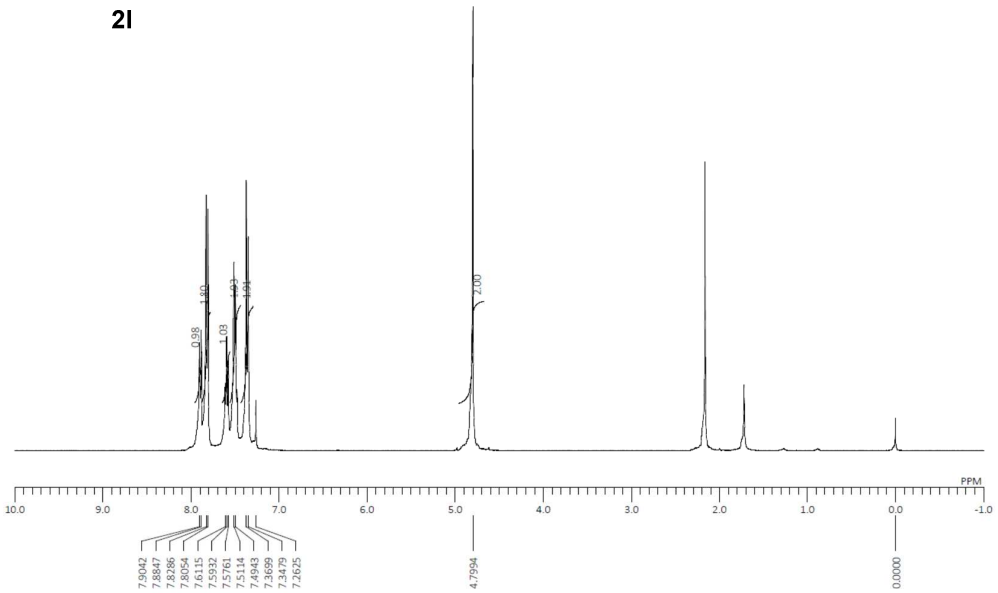
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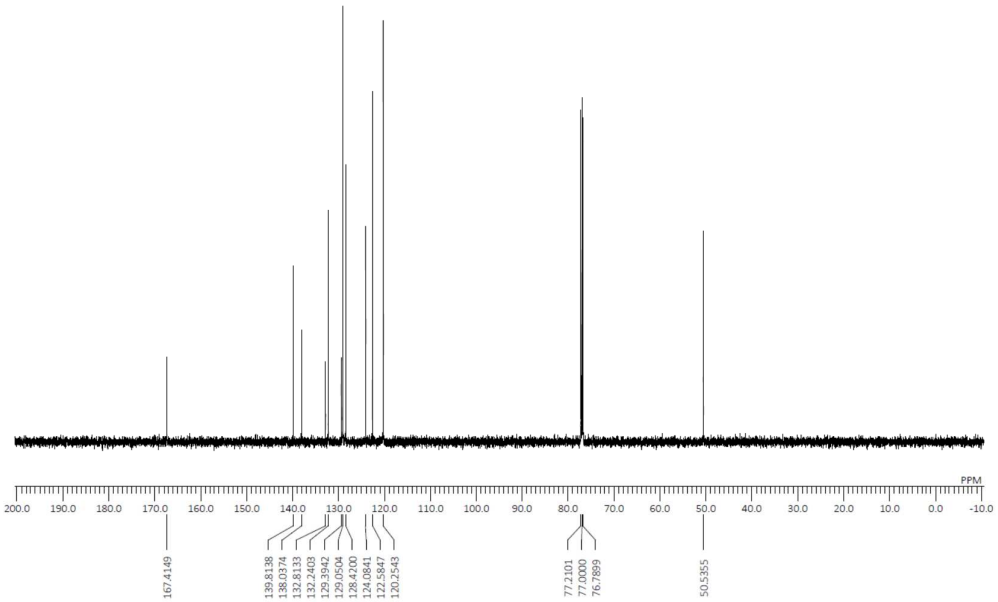
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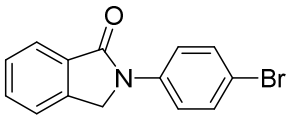
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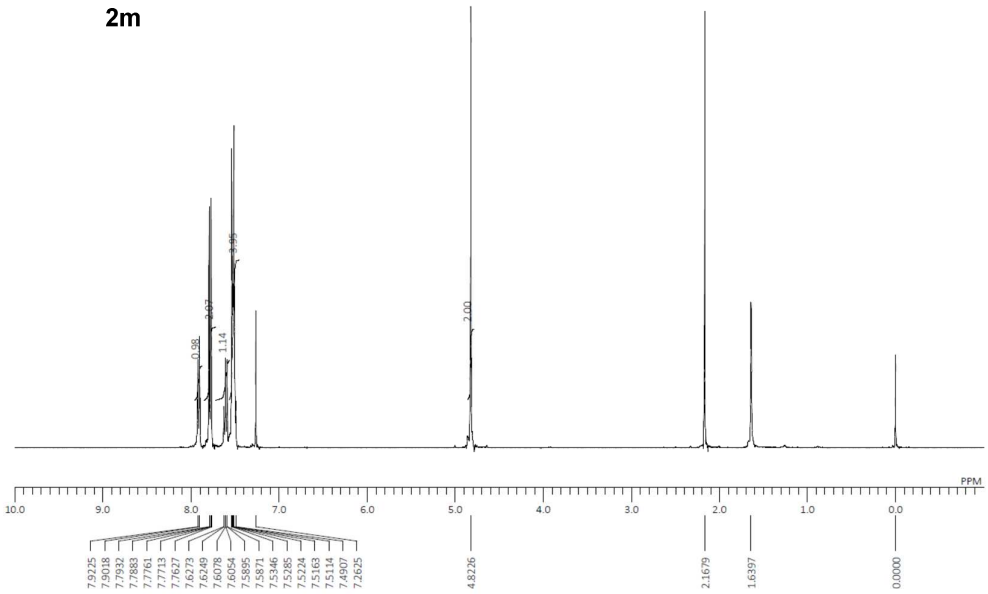
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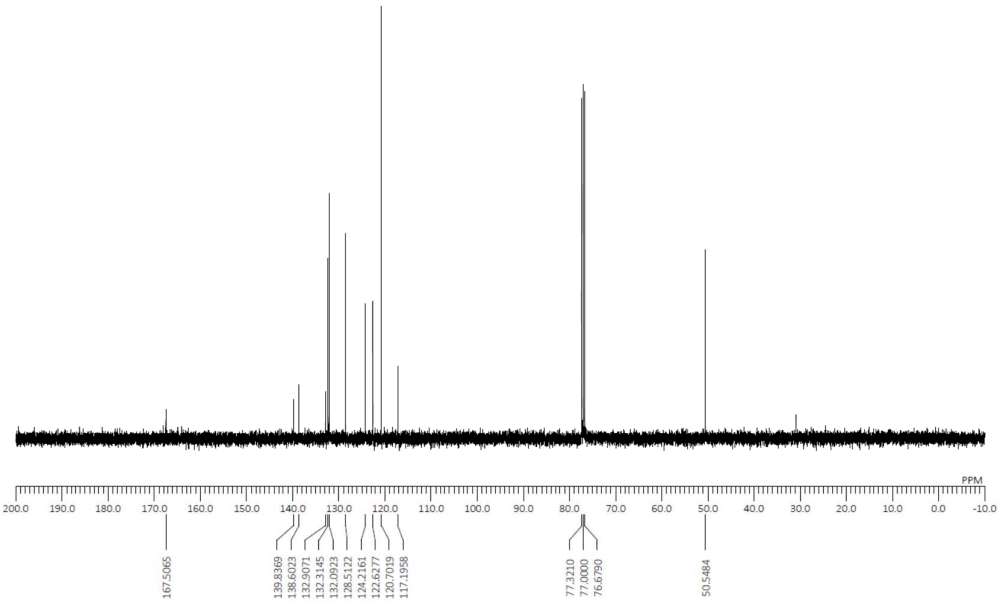
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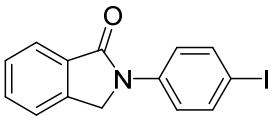
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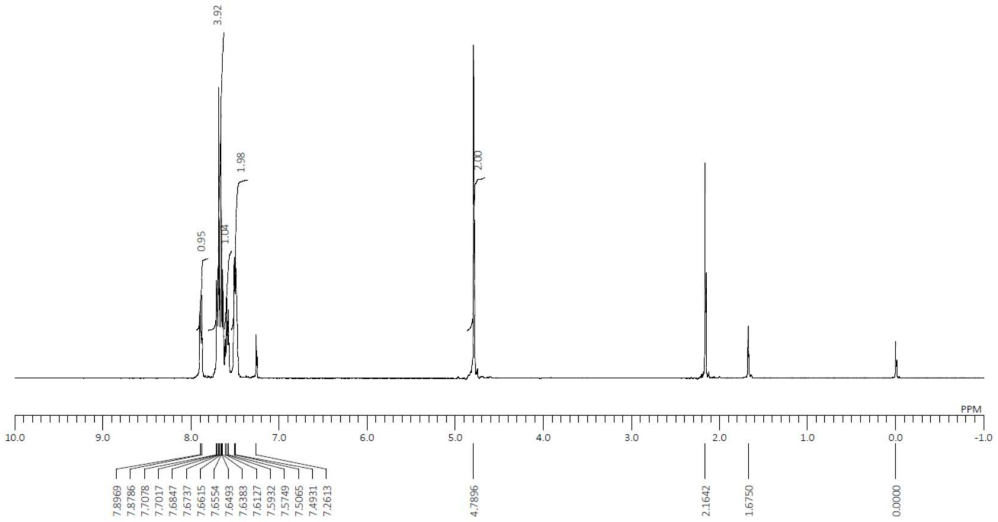
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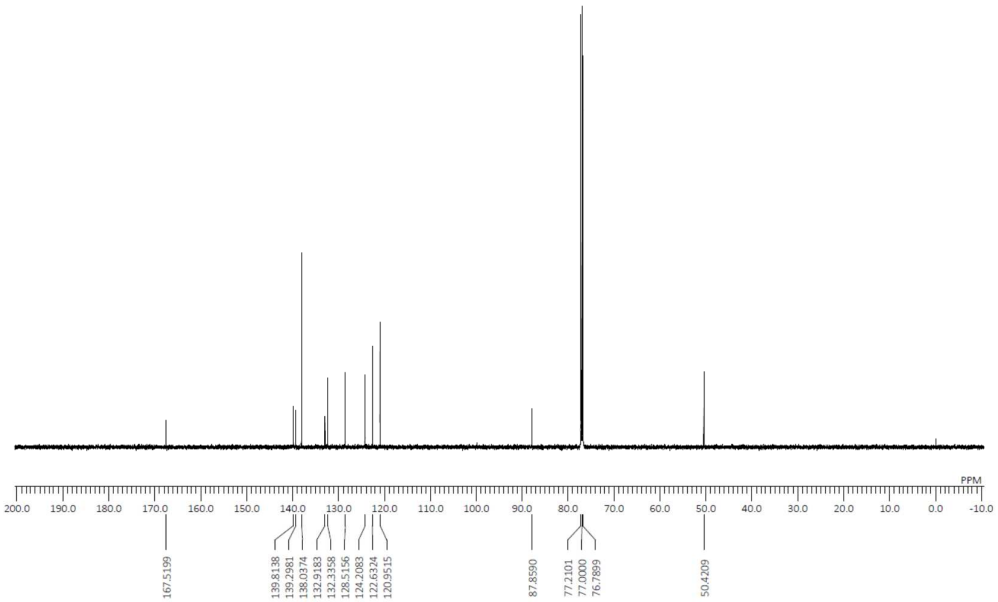
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60.0
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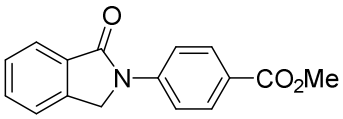
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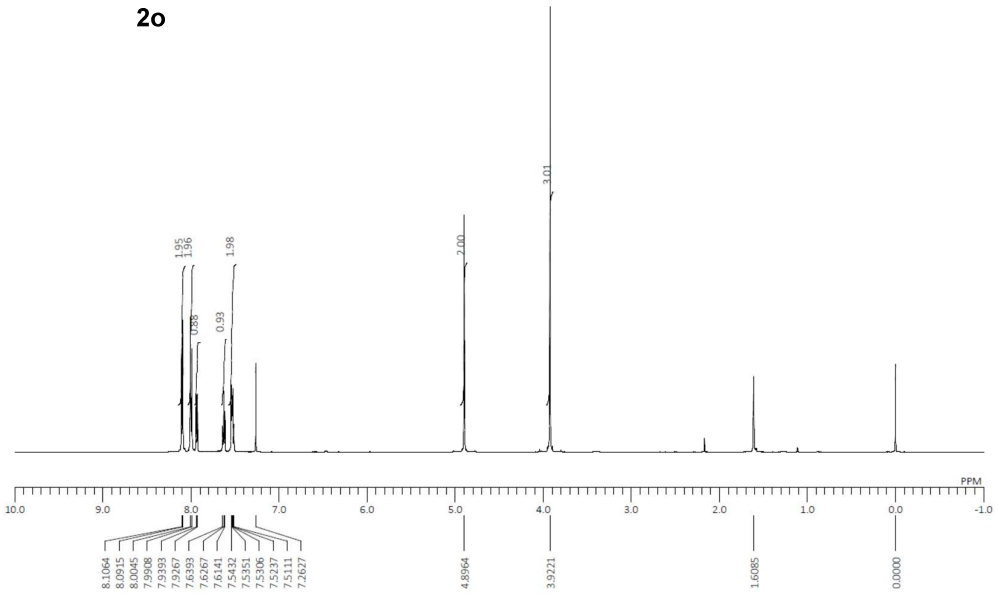
2o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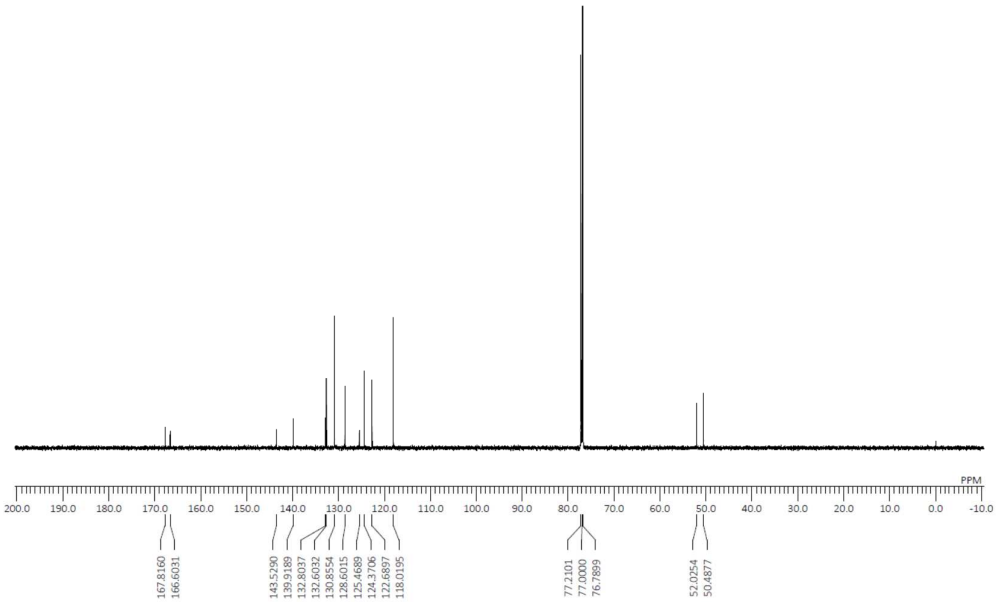
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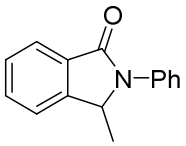
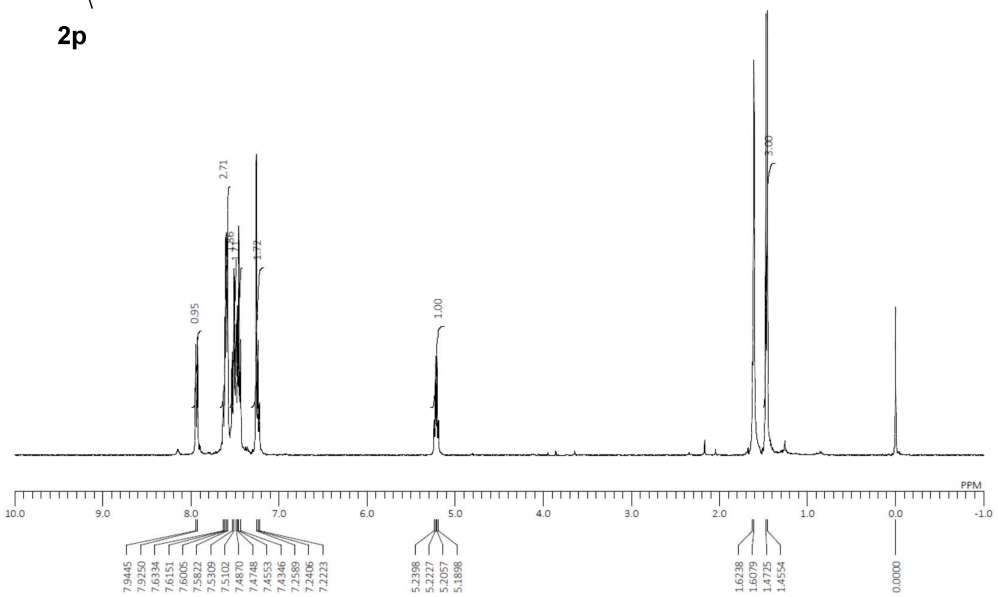

O
N–Ph

2p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2.0
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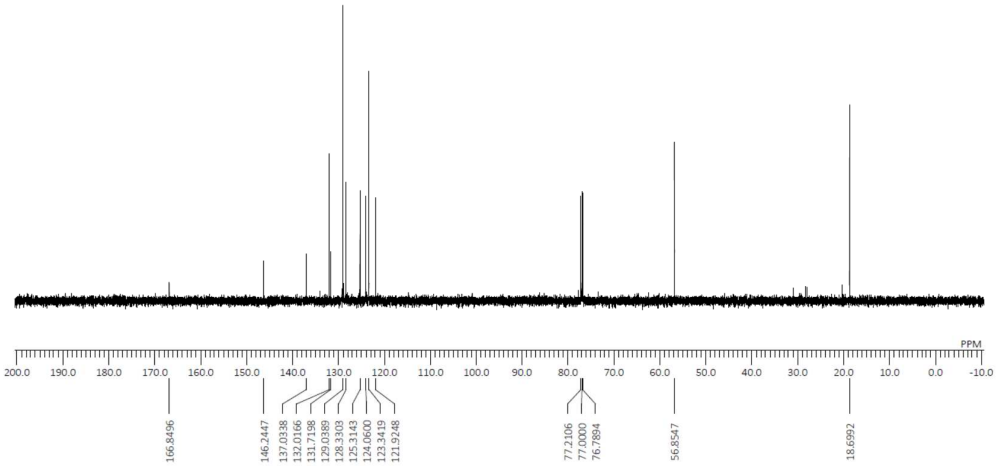
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