

SUPPORTING INFORMATION

for

Chemoselective Reduction of α -Keto Amides by Nickel Catalyst

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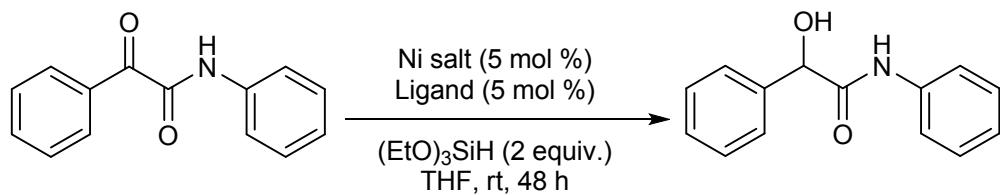
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Table 1: Optimization of ligands and nickel salts in the reduction of α -keto amides^a



Entry	Ligand	Nickel salt	Yield (%)
1	L1	Ni(OAc) ₂	30
2	L2	Ni(OAc)₂	32
3	L3	Ni(OAc) ₂	16
4	L4	Ni(OAc) ₂	20
5	L5	Ni(OAc) ₂	10
6	L6	Ni(OAc) ₂	22
7	L7	Ni(OAc) ₂	18
8	L8	Ni(OAc) ₂	5
9	L9	Ni(OAc) ₂	12
10	L2	NiCl ₂	10
11	L2	NiBr ₂	8
12	L2	Ni(acac) ₂	10
13	L2	Ni(OAc) ₂ .4H ₂ O	8
14	L2	NiCl ₂ .6H ₂ O	6
15	L2	NiBr ₂ .3H ₂ O	4

^aReaction condition. 0.5 mmol of **1a** in THF. ^bIsolated yield.

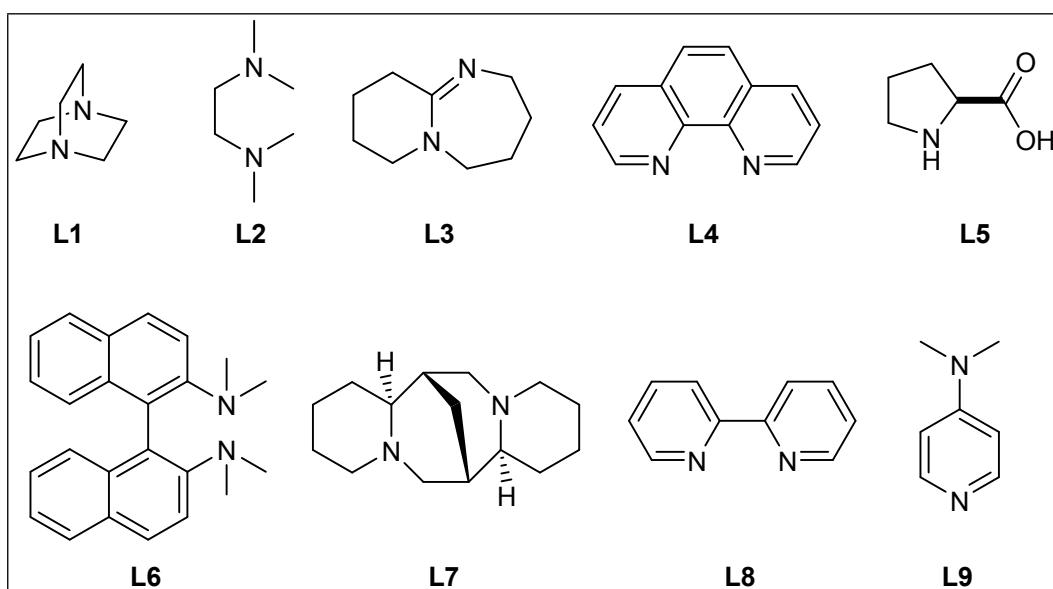


Figure 1: Ligands screened for the reduction of α -ketoamides

General considerations

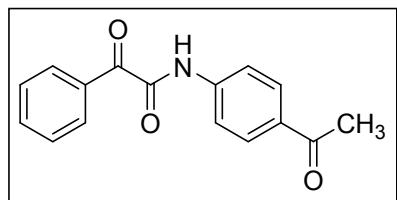
Nickel(II) acetate (98% pure), TMEDA (99% pure), DBU (99% pure), NaOAc (99% pure), NaOtBu (97% pure) and KOtBu (98% pure) were purchased from SpectrochemPvt. Ltd. India. Hydrosilanes, nickel(II) bromide (98% pure), nickel(II) chloride (98%), DABCO (>99% pure) and 1,10-phenanthroline (\geq 99% pure) were purchased from Sigma aldrich chemicals, USA. Thin-layer chromatography (TLC) was performed using Merck silica gel 60 F254 precoated plates (0.25 mm) and visualized by UV fluorescence quenching. Silica gel for column chromatography (particle size 100-200 mesh) was purchased from SRL India. 1 H and 13 C NMR spectra were recorded on a Bruker 400 MHz instrument. 1 H NMR spectra were reported relative to residual CHCl₃ (δ 7.26 ppm) or DMSO (δ 2.50 ppm). 13 C NMR were reported relative to CDCl₃ (δ 77.16 ppm) or DMSO-d₆ (δ 39.52 ppm). FTIR spectra were recorded on a JASCO spectrometer and are reported in frequency of absorption (cm⁻¹). High resolution mass spectra (HRMS) were recorded on Q-ToF Micro mass spectrometer.

General experimental procedure for synthesis of α -keto amides

Thionyl chloride (0.3 ml, 4 mmol) was added dropwise to a stirred mixture of benzyl formic acid (0.300 g, 2 mmol) and Et₃N (0.5 ml, 4 mmol) in CH₂Cl₂ (10 mL) at 0°C under nitrogen atmosphere. The stirring was continued for 20 min. and then a suspension of corresponding amine (2 mmol) in CH₂Cl₂ (10 mL) was added slowly to the reaction mixture at 0 °C under nitrogen flow. The stirring was continued in the room temperature and the completion of the reaction monitored through TLC. A saturated aqueous solution of NaHCO₃ (20 mL) was added slowly under stirring to the reaction mixture. The organic layer separated, washed with water (3 \times 15 mL) and evaporated under reduced pressure. The solid residue purified through silica gel column chromatography.

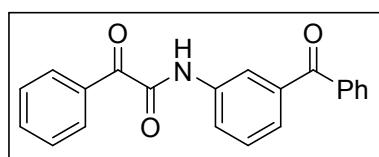
Spectral data for α -keto amides

N-(4-acetylphenyl)-2-oxo-2-phenylacetamide



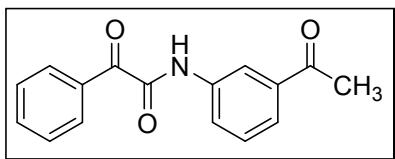
Green colour solid, mp = 163-162 °C R_f 0.36; (hexanes : ethyl acetate, 90:10 v/v): 1 H NMR (400 MHz, CDCl₃): δ 9.18 (bs, 1H), 8.41 (d, *J* = 7.6 Hz, 2H), 8.01 (d, *J* = 8.8 Hz, 2H), 7.81 (d, *J* = 8.8 Hz, 2H), 7.67 (t, *J* = 7.6 Hz, 1H), 7.52 (t, *J* = 7.6 Hz, 2H), 2.60 (s, 3H); 13 C NMR (100 MHz, CDCl₃): δ 197.0, 186.9, 159.1, 140.9, 135.0, 133.9, 132.9, 131.6, 130.0, 128.8, 119.5, 26.6; IR (neat) 3315, 1675, 1597, 1526, 1442, 1167 cm⁻¹; HRMS (*m/z*): [M+H]⁺ calcd for C₁₆H₁₄NO₃, 268.0974; found, 268.0966.

N-(3-benzoylphenyl)-2-oxo-2-phenylacetamide



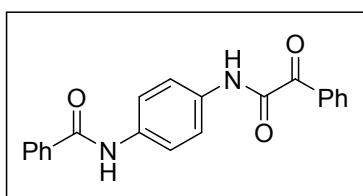
Yellow colour solid, mp = 148-149 °C R_f 0.35; (hexanes : ethyl acetate, 90:10 v/v): 1 H NMR (400 MHz, CDCl₃): δ 9.11 (bs, 1H), 8.40 (dd, *J* = 7.2, 1.2 Hz, 2H), 8.05 (dd, *J* = 9.8, 2.0 Hz, 2H), 7.83 (dd, *J* = 7.2, 1.6 Hz, 2H), 7.66 (t, *J* = 7.6, 1.2 Hz, 1H), 7.60 (t, *J* = 7.6, 1.2 Hz, 2H), 7.54-7.47 (m, 5H); 13 C NMR (100 MHz, CDCl₃): δ 196.1, 187.2, 159.2, 138.8, 137.3, 136.9, 134.9, 133.0, 131.6, 130.2, 129.4, 128.8, 128.5, 126.9, 123.8, 121.4; IR (neat) 3252, 1676, 1636, 1591, 1552, 1169 cm⁻¹; HRMS (*m/z*): [M+H]⁺ calcd for C₂₁H₁₆NO₃, 330.1130; found, 330.1119.

N-(3-acetylphenyl)-2-oxo-2-phenylacetamide



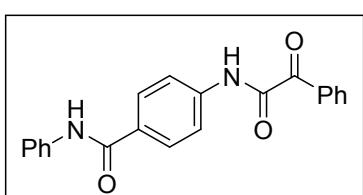
Pale yellow colour solid, mp = 88-89 °C R_f 0.36; (hexanes : ethyl acetate, 90:10 v/v): ¹H NMR (400 MHz, CDCl₃): δ 9.12 (bs, 1H), 8.42 (d, J = 7.6 Hz, 2H), 8.28 (s, 1H), 7.97 (d, J = 8.0 Hz, 1H), 7.78 (t, J = 7.6 Hz, 1H), 7.67 (t, J = 7.6 Hz, 1H), 7.56-7.46 (m, 3H), 2.63 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 197.6, 187.1, 159.2, 138.2, 137.3, 134.9, 133.1, 131.6, 129.7, 128.8, 125.2, 124.4, 119.8, 26.8; IR (neat) 3291, 1672, 1599, 1543, 1486, 1170 cm⁻¹; HRMS (m/z): [M+H]⁺ calcd for C₁₆H₁₄NO₃, 268.0974; found, 268.0967.

N-(4-(2-oxo-2-phenylacetamido)phenyl)benzamide



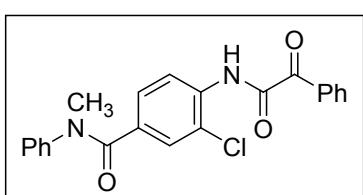
Lite Green colour solid, mp = 195-196 °C ; R_f 0.35; (hexanes : ethyl acetate, 80:20 v/v): ¹H NMR (400 MHz, CDCl₃:DMSO-d₆, 8:2): δ 9.61 (bs, 1H), 9.26 (bs, 1H), 8.20 (dd, J = 17.2, 8.0 Hz, 2H), 7.84 (t, J = 8.0 Hz, 2H), 7.60-7.75 (m, 4H), 7.59-7.49 (m, 1H), 7.48-7.30 (m, 5H); ¹³C NMR (100 MHz, CDCl₃:DMSO-d₆, 8:2): δ 188.1, 165.8, 160.5, 135.6, 134.9, 134.1, 133.0, 132.9, 131.1, 130.5, 128.2, 128.0, 127.3, 120.7, 120.3; IR (KBr) 3989, 1659, 1537, 1402 cm⁻¹; HRMS (m/z): [M+Na]⁺ calcd for C₂₁H₁₆N₂O₃Na₁, 367.1059; found, 367.1057.

4-(2-oxo-2-phenylacetamido)-N-phenylbenzamide



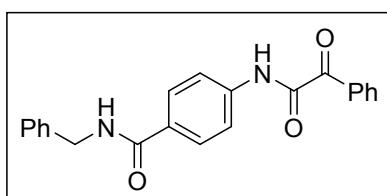
Green colour solid, mp = 180-181 °C ; R_f 0.49; (hexanes : ethyl acetate, 70:30 v/v): ¹H NMR (400 MHz, DMSO-d₆): δ 11.20 (bs, 1H), 10.17 (bs, 1H), 8.07 (d, J = 7.6 Hz, 2H), 8.01 (d, J = 8.0 Hz, 2H), 7.89 (d, J = 8.0 Hz, 2H), 7.78 (d, J = 8.0 Hz, 3H), 7.62 (t, J = 7.6 Hz, 2H) 7.35 (t, J = 7.6 Hz, 2H) 7.10 (t, J = 7.2 Hz, 1H); ¹³C NMR (100 MHz, DMSO-d₆): δ 189.1, 164.8, 163.5, 140.5, 139.2, 134.9, 132.5, 130.7, 130.0, 129.1, 128.7, 128.6, 123.6, 120.4, 119.5; IR (KBr) 3333, 1671, 1650, 1595, 1525, 1180 cm⁻¹; HRMS (m/z): [M+Na]⁺ calcd for C₁₅H₁₂N₂O₃Na₁, 291.0746; found, 291.0759.

3-chloro-N-methyl-4-(2-oxo-2-phenylacetamido)-N-phenylbenzamide



Pale Green colour solid, mp = 155-156 °C ; R_f 0.55; (hexanes : ethyl acetate, 50:50 v/v): ¹H NMR (400 MHz, DMSO-d₆): δ 10.56 (bs, 1H), 8.05 (d, J = 8.0, 2H), 7.76 (dd, J = 8.0, 13.2 Hz, 2H), 7.60 (t, J = 8.0 Hz, 2H), 7.46 (bs, 1H), 7.32 (t, J = 7.6 Hz, 2H), 7.29-7.18 (m, 4H), 3.38 (s, 3H); ¹³C NMR (100 MHz, DMSO-d₆): δ 188.7, 167.4 163.5, 144.2, 135.0, 134.8, 134.2, 132.6, 130.0, 129.7, 129.3, 129.0, 127.7, 126.9, 125.9, 124.7, 38.0; IR (KBr) 3358, 1705, 1678, 1637, 1600, 1569, 1525, 1054 cm⁻¹; HRMS (m/z): [M+H]⁺ calcd for C₂₂H₁₈N₂O₃Cl, 393.1006; found, 393.1016.

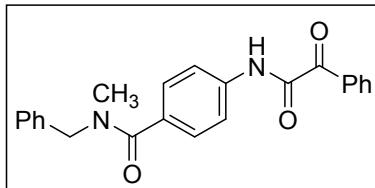
N-benzyl-4-(2-oxo-2-phenylacetamido)benzamide



Pale Green colour solid, mp = 142-143 °C ; R_f 0.55; (hexanes : ethyl acetate, 50:50 v/v): ¹H NMR (400 MHz, DMSO-d₆): δ 11.17 (bs, 1H), 9.00 (t, J = 5.6 Hz, 1H), 8.06 (d, J = 7.6 Hz, 2H), 7.94 (d, J = 8.0 Hz, 2H), 7.83 (d, J = 8.0 Hz, 2H), 7.77 (t, J = 7.2, 1H), 7.62 (t, J = 7.6 Hz, 2H), 7.32 (bs, 4H), 7.25 (d, J = 4.4 Hz, 1H), 4.49 (d, J = 5.6 Hz,

2H); ^{13}C NMR (100 MHz, DMSO- d_6): δ 189.2, 165.6, 163.5, 140.3, 139.8, 135.0, 132.5, 130.2, 130.0, 129.1, 128.3, 127.3, 126.8, 126.3, 119.5, 42.7; IR (KBr) 3326, 1803, 1666, 1630, 1168 cm^{-1} ; HRMS (m/z): [M+H] $^+$ calcd for $\text{C}_{22}\text{H}_{19}\text{N}_2\text{O}_3$, 359.1396; found, 359.1390.

N-benzyl-N-methyl-4-(2-oxo-2-phenylacetamido)benzamide



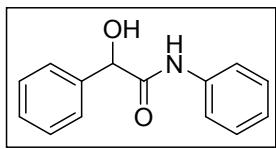
Pale Green colour solid, mp = 120-121 $^\circ\text{C}$; R_f 0.40; (hexanes : ethyl acetate, 70:30 v/v): ^1H NMR (400 MHz, DMSO- d_6): δ 10.85 (bs, 1H), 8.07 (dd, J = 1.2 Hz, 5.6 Hz, 2H), 7.81 (d, J = 6.4 Hz, 2H), 7.74 (t, J = 6.0 Hz, 1H), 7.60 (t, J = 6.4 Hz, 2H), 7.49 (d, J = 6.8 Hz, 2H), 7.38 (t, J = 6.4 Hz, 2H), 7.29 (t, J = 5.2 Hz, 3H), 4.62 (s, 2H), 2.90 (s, 3H); ^{13}C NMR (100 MHz, DMSO- d_6 , temp. 90 $^\circ\text{C}$): δ 188.6, 169.8, 162.9, 138.1, 136.9, 134.0, 132.6, 132.2, 129.3, 128.4, 128.0, 127.2, 126.8, 126.6, 119.6, 51.7, 34.7; IR (KBr) 3069, 1694, 1688, 1682, 1632, 1612, 1071 cm^{-1} ; HRMS (m/z): [M+H] $^+$ calcd for $\text{C}_{23}\text{H}_{21}\text{N}_2\text{O}_3$, 373.1552; found, 367.1569.

General experimental procedure for chemo selective reduction of α -keto amides

A mixture of TMEDA (4 μL , 0.025 mmol), nickel(II)acetate (4.4 mg, 0.025 mmol), NaOAc (4 mg, 0.05 mmol) and α -keto amide (0.5 mmol) in 1.5 mL of dry THF were taken in a reaction tube fitted with rubber septum under nitrogen atmosphere. The reaction mixture was stirred at room temperature for 10 min., then PMHS (120 μL , 2 mmol) was slowly added to the reaction mixture. Then the rubber septum was replaced with glass stopper under nitrogen flow and the reaction was stirred at 60 $^\circ\text{C}$. The progress of the reaction was monitored by TLC. After complete disappearance of starting material, 5 mL of 2N aq. NaOH was added and the resulting reaction mixture was stirred for 30 min. Then the reaction mixture was extracted with ethyl acetate (2x10 mL). The combined organic layers was washed with brine, dried over anhydrous MgSO_4 , filtered off and the solvent was removed under reduced pressure. The resulting residue was purified by silica gel column chromatography (eluents: hexanes-ethyl acetate, 80:20) to get pure α -hydroxy amide.

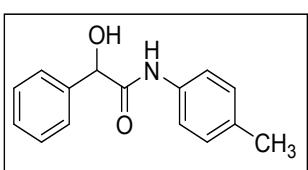
Spectral data for α -hydroxy amides

2-Hydroxy-N, 2-diphenylacetamide¹



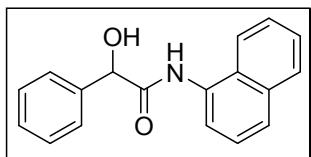
Colorless solid, mp = 152-153 $^\circ\text{C}$ (Lit.¹ mp = 150-151 $^\circ\text{C}$); R_f 0.56; (hexanes : ethyl acetate, 80:20 v/v): ^1H NMR (400 MHz, CDCl_3): δ 8.11 (bs, 1H), 7.55-7.46 (m, 4H), 7.43-7.35 (m, 3H), 7.32 (t, J = 7.6 Hz, 2H), 7.12 (t, J = 7.6 Hz, 2H), 5.19 (s, 1H), 3.42 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 170.1, 139.1, 137.2, 129.2, 129.1, 129.00, 127.0, 124.9, 120.0, 74.9; IR (KBr) 3675, 3409, 1655, 1602, 1544, 1191 cm^{-1} ; HRMS (m/z): [M+Na] $^+$ calcd for $\text{C}_{14}\text{H}_{13}\text{NO}_2\text{Na}_1$, 250.0844; found, 250.0833

2-Hydroxy-2-phenyl-N-p-tolylacetamide²



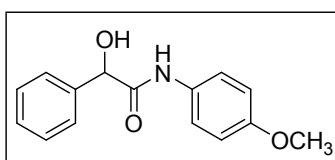
Colorless solid, mp = 168-169 $^\circ\text{C}$ (Lit.² mp = 169-170 $^\circ\text{C}$); R_f 0.46; (hexanes : ethyl acetate, 70:30 v/v): ^1H NMR (400 MHz, CDCl_3): δ 8.00 (bs, 1H), 7.48 (dd, J = 8.0 Hz, 1.6 Hz, 2H), 7.43-7.31 (m, 5H), 7.11 (d, J = 8.4 Hz, 2H), 5.17 (d, J = 3.2 Hz, 1H), 3.44 (d, J = 3.2 Hz 1H), 2.30 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 169.9, 139.2, 134.6, 134.5, 129.7, 129.2, 129.1, 127.1, 120.0, 74.8, 21.0; IR (KBr) 3322, 1646, 1602, 1546, 1527, 1495 cm^{-1} ; HRMS (m/z): [M+H] $^+$ calcd for $\text{C}_{15}\text{H}_{16}\text{NO}_2$, 242.1181; found, 242.1192

2-Hydroxy-N-(naphthalen-1-yl)-2-phenylacetamide



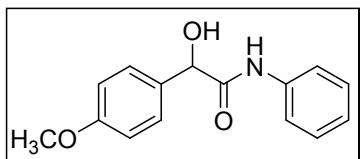
Colorless solid, mp = 114-115 °C ; R_f 0.55; (hexanes : ethyl acetate, 90:10 v/v): ^1H NMR (400 MHz, CDCl_3): δ 8.72 (bs, 1H), 7.96 (d, J = 7.6, 1H), 7.84 (d, J = 5.2 Hz, 1H), 7.67 (d, J = 8.4 Hz, 2H), 7.64 (d, J = 3.2 Hz 1H), 7.56-7.34 (m, 8H), 5.26 (d, J = 3.2 Hz, 1H), 3.92 (d, J = 3.2 Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 171.1, 139.3, 134.1, 131.5, 129.0, 128.9, 126.9, 126.8, 126.6, 126.1, 126.0, 125.8, 120.3, 120.1, 74.9; IR (KBr) 3360, 3265, 1659, 1592, 1523, 1514, 1497 cm^{-1} ; HRMS (m/z): [M+H] $^+$ calcd for $\text{C}_{18}\text{H}_{15}\text{NO}_2$, 300.1000; found, 300.1001.

2-Hydroxy-N-(4-methoxyphenyl)-2-phenylacetamide³



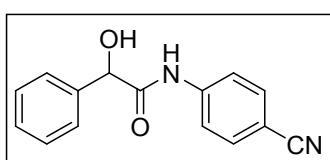
Colorless solid, mp = 154-155 °C (Lit.³ mp= 153-154 °C); R_f 0.34; (hexanes : ethyl acetate, 80:20 v/v): ^1H NMR (400 MHz, CDCl_3): δ 8.01 (bs, 1H), 7.48 (d, J = 7.2 Hz, 3H), 7.45-7.33 (m, 5H), 6.84 (d, J = 2.8 Hz, 1H), 5.17 (d, J = 2.8 Hz, 1H), 3.77 (s, 3H), 3.49 (d, J = 3.2 Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 170.0, 156.8, 139.2, 130.3, 129.0, 128.9, 127.0, 121.7, 114.3, 74.7, 55.6; IR (KBr) 3655, 1644, 1599, 1556, 1545, 1192 cm^{-1} ; HRMS (m/z): [M+H] $^+$ calcd for $\text{C}_{15}\text{H}_{16}\text{NO}_3$, 258.1130; found, 258.1133.

2-Hydroxy-2-(4-methoxyphenyl)-N-phenylacetamide



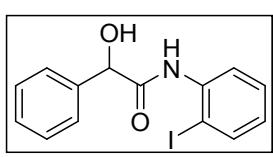
Colorless solid, mp = 94-95 °C; R_f 0.32; (hexanes : ethyl acetate, 80:20 v/v): ^1H NMR (400 MHz, CDCl_3): δ 8.25 (bs, 1H), 7.51 (d, J = 7.6 Hz, 2H), 7.35 (d, J = 8.4 Hz, 1H), 7.30 (t, J = 8.4 Hz, 2H), 7.11 (t, J = 7.6 Hz, 1H), 6.89 (d, J = 8.4 Hz, 2H), 5.07 (d, J = 2.8 Hz, 1H), 3.79 (s, 1H), 3.65 (d, J = 3.2 Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 170.6, 160.0, 137.2, 131.3, 129.1, 128.3, 124.7, 119.9, 114.4, 74.3, 55.4; IR (KBr) 3359, 2923, 1662, 1602, 1530, 1523, 1513 cm^{-1} ; HRMS (m/z): [M+Na] $^+$ calcd for $\text{C}_{15}\text{H}_{15}\text{NO}_3\text{Na}_1$, 280.0950; found, 280.0954.

N-(4-Cyanophenyl)-2-hydroxy-2-phenylacetamide



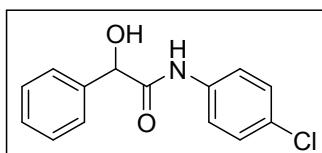
Colorless solid, mp= 130-131°C; R_f 0.37; (hexanes : ethyl acetate, 80:20 v/v): ^1H NMR (400 MHz, CDCl_3): δ 8.53 (s, 1H), 7.70 (d, J = 8.8 Hz 2H), 7.61 (d, J = 8.8 Hz, 2H), 7.49 (dd, J = 8.0, 1.6 Hz 2H), 7.45-7.37 (m, 3H), 5.26 (d, J = 3.2 Hz, 1H), 3.13 (d, J = 3.2 Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ 170.3, 141.3, 138.6, 133.5, 131.3, 129.3, 129.2, 126.9, 119.8, 119.6, 75.0; IR (KBr) 3289, 3109, 2227, 1744, 1703, 1667, 1604, 1597 cm^{-1} ; HRMS (m/z): [M+H] $^+$ calcd for $\text{C}_{15}\text{H}_{13}\text{O}_2\text{N}_2$, 253.0977; found, 253.0978

2-Hydroxy-N-(2-iodophenyl)-2-phenylacetamide



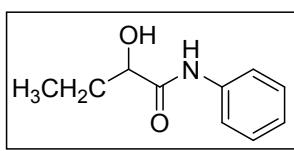
Colorless solid, mp = 75-76 °C ; R_f 0.35; (hexanes : ethyl acetate, 80:20 v/v): ^1H NMR (400 MHz, CDCl_3): δ 8.57 (bs, 1H), 8.22 (dd, J = 8.4, 1.2 Hz, 1H), 7.76 (dd, J = 8.0,1.6 Hz, 1H), 7.52 (d, J = 7.2 Hz, 2H), 7.45-7.35 (m, 3H), 7.35-7.29(m, 1H), 6.84 (td, J = 7.8, 0.8 Hz, 1H), 5.21 (d, J = 1.2 Hz, 1H), 3.69 (d, J = 2.4 Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 170.6, 139.0, 138.0, 137.7, 129.4, 129.2, 129.1, 127.1, 126.4, 121.6, 89.8, 75.1; IR (KBr) 3445, 2958, 1731, 1183, 1067 cm^{-1} ; HRMS (m/z): [M+H] $^+$ calcd for $\text{C}_{14}\text{H}_{13}\text{NO}_2\text{I}$, 353.9991; found, 354.0004.

N-(4-Chlorophenyl)-2-hydroxy-2-phenylacetamide⁴



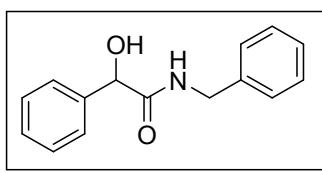
Colorless solid, mp = 160-161 °C (Lit.⁴ mp = 161-164 °C); R_f 0.58; (hexanes : ethyl acetate, 70:30 v/v): ¹H NMR (400 MHz, CDCl₃): δ 8.18 (bs, 1H), 7.49-7.42 (m, 4H), 7.41-7.32 (m, 3H), 7.25 (d, J = 2.4 Hz, 2H), 5.18 (d, J = 2.8 Hz, 1H), 3.24 (d, J = 2.8 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 169.9, 138.9, 135.8, 129.9, 129.2, 129.19, 127.0, 121.2, 74.9; IR (KBr) 3297, 1650, 1596, 1540, 1068 cm⁻¹; HRMS (m/z): [M+Na]⁺ calcd for C₁₄H₁₂NO₂Na₁Cl, 284.0454; found, 284.0443.

2-Hydroxy-N-phenylpentanamide



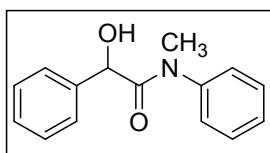
Colorless solid mp = 129-130 °C; R_f 0.30; (hexanes : ethyl acetate, 80:20 v/v): ¹H NMR (400 MHz, CDCl₃): δ 8.38 (bs, 1H), 7.57 (dd, J = 8.4, 1.2 Hz, 2H), 7.31-7.37 (m, 2H), 7.15-7.37 (m, 1H), 4.23 (m, 1H), 2.58 (d, J = 4.4 Hz, 1H), 2.05-1.93 (m, 1H), 1.87-1.74 (m, 1H), 1.04 (t, J = 7.6 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 172.3, 137.2, 129.2, 124.7, 120.0, 73.6, 30.0, 9.3; IR (KBr) 3402, 3392, 1647, 1601, 1531, 1444, 1120 cm⁻¹; HRMS (m/z): [M+H]⁺ calcd for C₁₀H₁₄NO₂, 180.1025; found, 180.1033.

N-Benzyl-2-hydroxy-2-phenylacetamide⁵



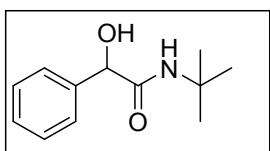
Colorless solid, mp = 133-134 °C (Lit.⁵ mp = 133-135 °C); R_f 0.35; (hexanes : ethyl acetate, 80:20 v/v): ¹H NMR (400 MHz, CDCl₃): δ 7.43-7.33 (m, 5H), 7.33-7.27 (m, 3H), 7.18 (dd, J = 6.8, 1.2 Hz, 1H), 6.47 (bs, 1H), 5.08 (d, J = 3.2 Hz, 1H), 4.52-4.38 (m, 2H), 3.60 (d, J = 3.2 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 172.2, 139.5, 137.8, 129.0, 128.9, 127.7, 127.0, 74.4, 43.7; IR (KBr) 3568, 3281, 2921, 1626, 1534, 1348, 1028 cm⁻¹; HRMS (m/z): [M+Na]⁺ calcd for C₁₄H₁₁NO₂Na₁, 248.0687; found, 248.0691.

2-Hydroxy-N-methyl-N, 2-diphenylacetamide



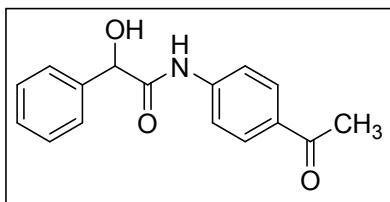
Colorless solid, mp = 89-90 °C; R_f 0.40; (hexanes : ethyl acetate, 70:30 v/v): ¹H NMR (400 MHz, CDCl₃): δ 7.34-7.22 (m, 3H), 7.22-7.10 (m, 3H), 6.86-6.77 (m, 4H), 5.01 (d, J = 6.8 Hz, 1H), 4.49 (d, J = 7.2 Hz, 1H), 3.30 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 172.9, 141.6, 139.3, 129.7, 128.4, 128.2, 128.1, 127.4, 71.7, 38.4; IR (KBr) 3423, 2952, 1655, 1596, 1585, 1496, 1020 cm⁻¹; HRMS (m/z): [M+Na]⁺ calcd for C₁₅H₁₅NO₂Na₁, 264.1010; found, 264.1000.

N-tert-Butyl-2-hydroxy-2-phenylacetamide⁶



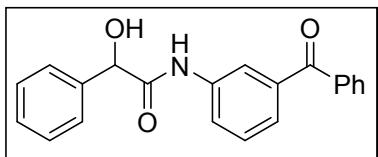
Colorless solid, mp = 103-104 °C; R_f 0.31; (hexanes : ethyl acetate, 70:30 v/v): ¹H NMR (400 MHz, CDCl₃): δ 7.39-7.31 (m, 5H), 5.76 (s, 1H), 4.90 (d, J = 3.6 Hz, 1H), 3.64 (s, 1H), 1.32 (s, 9H); ¹³C NMR (100 MHz, CDCl₃): δ 171.4, 140.0, 129.0, 128.6, 127.0, 74.3, 51.6, 28.8; IR (KBr) 3225, 1645, 1538, 1409, 1452, 1065 cm⁻¹; HRMS (m/z): [M+Na]⁺ calcd for C₁₂H₁₇NO₂Na₁, 230.1157; found, 230.1167

N-(4-Acetylphenyl)-2-hydroxy-2-phenylacetamide



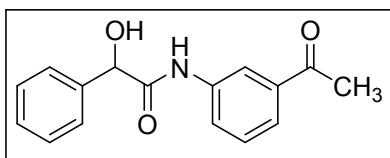
Colourless solid, mp = 148-149 °C R_f 0.39; (hexanes : ethyl acetate, 70:30 v/v): ¹H NMR (400 MHz, DMSO-d₆): δ 10.78 (bs, 1H), 8.42 (d, J = 8.8 Hz, 2H), 8.37 (d, J = 8.8 Hz, 2H), 8.04 (d, J = 8.0 Hz, 2H), 7.87 (t, J = 7.2 Hz, 2H), 7.84-7.78 (m, 1H), 7.02 (d, J = 4.4 Hz, 1H), 5.66 (d, J = 4.4 Hz, 1H), 3.03 (s, 3H); ¹³C NMR (100 MHz, DMSO-d₆): δ 196.6, 171.8, 142.9, 140.6, 132.0, 129.3, 128.2, 127.8, 126.6, 119.0, 74.1, 26.5; IR (neat) 3285, 1674, 1657, 1598, 1543, 1183 cm⁻¹; HRMS (m/z): [M+H]⁺ calcd for C₁₆H₁₆NO₃, 270.1130; found, 270.1118.

N-(3-Benzoylphenyl)-2-hydroxy-2-phenylacetamide



Colourless solid, mp = 126-127 °C R_f 0.35; (hexanes : ethyl acetate, 70:30 v/v): ¹H NMR (400 MHz, CDCl₃): δ 8.52 (bs, 1H), 7.94-7.90 (m 1H), 7.81 (t, J = 1.6 Hz, 1H), 7.77 (dd, J = 7.2, 1.6 Hz, 2H), 7.61-7.55 (m 1H), 7.51-7.41 (m, 5H), 7.41-7.32 (m, 4H), 5.17 (d, J = 3.6 Hz, 1H), 3.76 (d, J = 3.6 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 196.5, 170.5, 138.9, 138.5, 137.5, 137.3, 132.9, 130.2, 129.2, 129.1, 129.0, 128.5, 126.9, 126.4, 123.9, 121.1, 74.8; IR (neat) 3324, 1659, 1591, 1532, 1499, 1180 cm⁻¹; HRMS (m/z): [M+H]⁺ calcd for C₂₁H₁₈NO₃, 332.1287; found, 332.1286.

N-(3-Acetylphenyl)-2-hydroxy-2-phenylacetamide



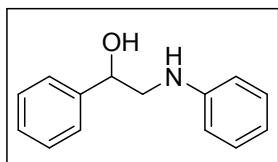
Yellow colour solid, mp = 118-119 °C, R_f 0.38; (hexanes : ethyl acetate, 70:30 v/v): ¹H NMR (400 MHz, DMSO-d₆): δ 10.16 (bs, 1H), 8.32 (t, J = 1.6 Hz, 1H), 7.98 (dd, J = 8.2, 1.2 Hz, 1H), 7.65 (d, J = 8.0 Hz, 1H), 7.53 (d, J = 7.2 Hz, 2H), 7.44 (t, J = 8.0 Hz, 1H), 7.36 (t, J = 7.2 Hz, 2H), 7.33-7.26 (m 1H), 6.48 (d, J = 4.8 Hz, 1H), 5.13 (d, J = 4.4 Hz, 1H), 2.54 (s, 3H); ¹³C NMR (100 MHz, DMSO-d₆): δ 197.7, 171.6, 140.8, 139.0, 137.3, 129.1, 128.2, 127.7, 126.6, 124.2, 123.4, 119.3, 74.1, 26.8; IR (neat) 3335, 1674, 1604, 1542, 1485, 1192 cm⁻¹; HRMS (m/z): [M+H]⁺ calcd for C₁₆H₁₆NO₃, 270.1130; found, 270.1130.

General experimental procedure for complete reduction of α -keto amides.

A mixture of TMEDA (4 μ L, 0.025 mmol), nickel(II)acetate (4.4 mg, 0.025 mmol), KO^tBu (6 mg, 0.05 mmol) in 1.5 mL of dry THF were taken in a reaction tube fitted with rubber septum under nitrogen atmosphere. The reaction mixture was stirred at room temperature for 10 min. Then the α -keto amide (0.5 mmol) was added to the reaction mixture and stirred for 10 min. then Ph₂SiH₂ (370 μ L, 2 mmol) was added slowly to the reaction mixture. After that, the rubber septum was replaced with glass stopper under nitrogen flow. The resulting reaction mixture was stirred at room temperature. The progress of the reaction was monitored by TLC. After complete disappearance of substrate, 5 mL of 2N aq. NaOH was added to the reaction and the resulting mixture was stirred for 30 min. The reaction mixture was extracted with ethyl acetate (2x10 mL). The combined organic layers was washed with brine, dried over anhydrous MgSO₄, filtered off and the solvents was removed under reduced pressure and the resulting residue was purified by neutral aluminium oxide column chromatography (eluents: hexanes-ethyl acetate, 90:10) to obtain pure β -amino alcohol.

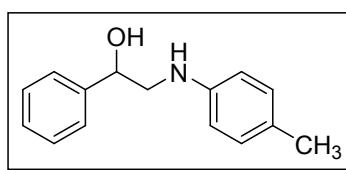
Spectral data for β -amino alcohols

1-Phenyl-2-(phenylamino)ethanol⁷



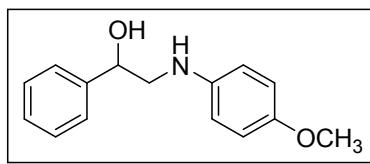
Colourless oily liqued; R_f 0.36; (hexanes : ethyl acetate, 80:20 v/v): ¹H NMR (400 MHz, CDCl₃): δ 7.45-7.37 (m, 4H), 7.36-7.31 (m, 1H), 7.21 (t, J = 8.4 Hz, 2H), 6.77 (t, J = 7.6 Hz, 1H), 6.69 (d, J = 7.6 Hz, 2H), 4.93 (dd, J = 8.8, 4.0 Hz, 1H), 3.44 (dd, J = 13.2, 4.0 Hz, 1H), 3.30 (dd, J = 13.2, 8.8 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 148.0, 142.1, 129.5, 128.8, 128.1, 126.0, 118.3, 113.6, 72.6, 51.9; IR (Neat) 3395, 3113, 1603, 1559, 1508, 1454 cm⁻¹; HRMS (*m/z*): [M+H]⁺ calcd for C₁₄H₁₆NO, 214.1232; found, 214.1240

1-Phenyl-2-(p-tolylamino)ethanol⁸



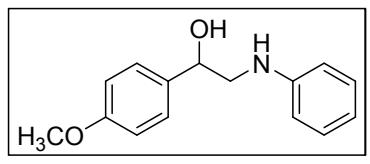
Colourless oil; R_f 0.39; (hexanes : ethyl acetate, 80:20 v/v): ¹H NMR (400 MHz, CDCl₃): δ 7.45-7.30 (m, 5H), 7.02 (d, J = 8.0 Hz, 2H), 6.62 (t, J = 8.0 Hz, 2H), 4.91 (dd, J = 8.6, 3.6 Hz, 1H), 3.41 (dd, J = 13.2, 4.0 Hz, 1H), 3.27 (dd, J = 13.2, 8.4 Hz, 1H), 2.26 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 145.6, 142.2, 130.0, 128.7, 128.0, 127.6, 126.0, 113.8, 72.5, 52.4, 20.5; IR (Neat) 3451, 3444, 1646, 1547, 1407, 1258 cm⁻¹; HRMS (*m/z*): [M+H]⁺ calcd for C₁₅H₁₈NO, 228.1388; found, 228.1378

2-(4-Methoxyphenylamino)-1-phenylethanol⁹



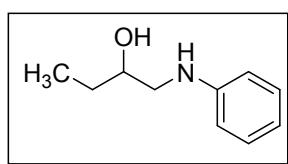
Pale yellow oil; R_f 0.34; (hexanes : ethyl acetate, 80:20 v/v): ¹H NMR (400 MHz, CDCl₃): δ 7.44-7.36 (m, 4H), 7.35-7.29 (m, 1H), 6.80 (d, J = 9.2 Hz, 2H), 6.66 (d, J = 8.8 Hz, 2H), 4.92 (dd, J = 8.8, 3.6 Hz, 1H), 3.75 (s, 3H), 3.38 (dd, J = 12.8, 4.0 Hz, 1H), 3.25 (dd, J = 13.2, 8.8 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 152.8, 142.2, 142.1, 128.8, 128.1, 126.0, 115.2, 115.1, 72.6, 55.9, 53.1; IR (Neat) 3363, 3061, 1653, 1510, 1450, 1034 cm⁻¹; HRMS (*m/z*): [M+H]⁺ calcd for C₁₅H₁₈NO₂, 244.1338; found, 244.1332

2-(Methyl(phenyl)amino)-1-phenylethanol⁷



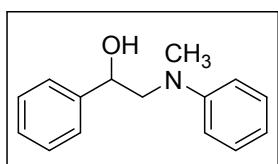
Pale yellow oil; R_f 0.37; (hexanes : ethyl acetate, 80:20 v/v): ¹H NMR (400 MHz, CDCl₃): δ 7.33 (d, J = 7.6 Hz, 2H), 7.20 (t, J = 7.6 Hz, 2H), 6.92 (d, J = 7.6 Hz, 1H), 6.76 (t, J = 7.2 Hz, 1H), 6.67 (d, J = 7.6 Hz, 2H), 4.86 (dd, J = 8.4, 3.6 Hz, 1H), 3.82 (s, 3H), 3.38 (dd, J = 13.0, 4.0 Hz, 1H), 3.29 (dd, J = 12.6, 8.4 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 159.5, 148.0, 134.3, 129.4, 127.3, 118.2, 114.1, 113.6, 72.2, 55.4, 51.8; IR (Neat) 3395, 3113, 1603, 1559, 1508, 1454 cm⁻¹; HRMS (*m/z*): [M+H]⁺ calcd for C₁₅H₁₈NO₂, 244.1338; found, 244.1340

1-(Phenylamino)butan-2-ol¹⁰



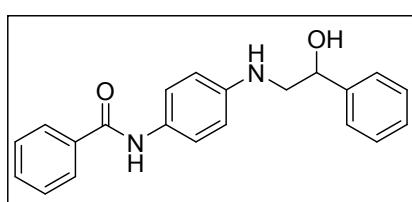
Colourless oil; R_f 0.40; (hexanes : ethyl acetate, 80:20 v/v): ¹H NMR (400 MHz, CDCl₃): δ 7.21-7.16 (m, 2H), 6.74 (tt, J = 6.0, 0.8 Hz, 1H), 6.66 (dd, J = 8.5, 1.0 Hz, 2H), 3.80-3.74 (m, 1H), 3.28 (dd, J = 13.0, 3.0 Hz, 1H), 3.01 (dd, J = 13.0, 8.5 Hz, 1H), 1.65-1.50 (m, 2H), 1.02 (t, J = 7.5 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 148.4, 129.4, 118.0, 113.4, 71.9, 50.0, 28.1, 10.1; IR (Neat) 3281, 3054, 1677, 1605, 1503, 1461 cm⁻¹; HRMS (*m/z*): [M+H]⁺ calcd for C₁₀H₁₆NO, 166.1232; found, 166.1235

2-(methyl(phenyl)amino)-1-phenylethanol



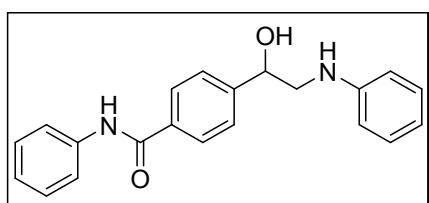
Colourless oily liquid; R_f 0.62; (hexanes : ethyl acetate, 80:20 v/v): ^1H NMR (400 MHz, CDCl_3): δ 7.47-7.38 (m, 4H), 7.37-7.27 (m, 3H), 6.89 (d, $J = 8.0$ Hz, 2H), 6.82 (t, $J = 7.2$ Hz, 1H), 5.01 (dd, $J = 8.8, 4.4$ Hz, 1H), 3.54 (dd, $J = 14.6, 8.4$ Hz, 1H), 3.46 (dd, $J = 14.8, 4.4$ Hz, 1H), 2.96 (s, 3H), 2.66 (bs, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 150.0, 142.1, 129.4, 128.7, 127.9, 126.0, 117.7, 113.4, 71.8, 62.1, 39.6; IR (Neat) 3405, 3043, 1598, 1498, 1357, 1043 cm^{-1} ; HRMS (m/z): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{15}\text{H}_{18}\text{NO}$, 228.1388; found, 228.1385

N-(4-(1-Hydroxy-2-(phenylamino)ethyl)phenyl)benzamide



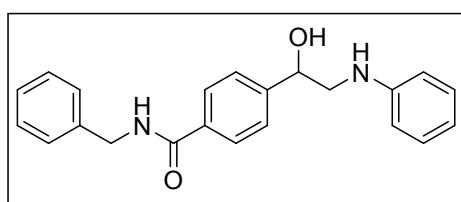
Colourless oil; R_f 0.33; (hexanes : ethyl acetate, 80:20 v/v): ^1H NMR (400 MHz, CDCl_3): δ 7.96 (s, 1H), 7.82 (d, $J = 6.8$ Hz, 2H), 7.50 (t, $J = 7.2$ Hz, 1H), 7.42 (t, $J = 7.6$ Hz, 2H), 7.39-7.33 (m, 6H), 7.33-7.29 (m, 1H), 6.58 (d, $J = 8.8$ Hz, 2H), 4.85 (dd, $J = 8.9, 3.6$ Hz, 1H), 3.35 (dd, $J = 13.2, 4.8$ Hz, 1H), 3.23 (dd, $J = 13.2, 8.8$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 166.0, 145.4, 142.3, 135.1, 131.7, 128.8, 128.7, 128.6, 128.0, 127.2, 126.0, 122.9, 113.8, 72.3, 52.1; IR (Neat) 3385, 3060, 1643, 1523, 1474, 1407, 1061 cm^{-1} ; HRMS (m/z): $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{21}\text{H}_{20}\text{N}_2\text{O}_2$, 355.1422; found, 355.1429

4-(1-Hydroxy-2-(phenylamino)ethyl)-N-phenylbenzamide



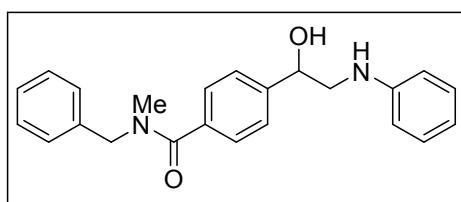
Pale yellow oil; R_f 0.40; (hexanes : ethyl acetate, 60:40 v/v): ^1H NMR (400 MHz, DMSO-d_6): δ 9.76 (s, 1H), 7.77 (t, $J = 8.8$ Hz, 4H), 7.42 (d, $J = 7.6$ Hz, 2H), 7.38-7.24 (m, 5H), 7.04 (t, $J = 7.6$ Hz, 1H), 6.69 (d, $J = 8.0$ Hz, 2H), 4.77 (t, $J = 6.8$ Hz, 1H), 3.31 (dd, $J = 13.2, 4.4$ Hz, 2H), 3.22 (dd, $J = 13.0, 8.0$ Hz, 1H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 165.3, 151.6, 144.0, 139.8, 129.3, 128.5, 128.1, 127.1, 126.1, 122.9, 121.2, 120.2, 111.0, 70.8, 50.9; IR (Neat) 3548, 3412, 3344, 1645, 1603, 1516, 1063 cm^{-1} ; HRMS (m/z): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{21}\text{H}_{21}\text{N}_2\text{O}_2$, 333.1615; found, 333.1603

N-Benzyl-4-(1-hydroxy-2-(phenylamino)ethyl)benzamide



Colourless oil; R_f 0.32; (hexanes : ethyl acetate, 60:40 v/v): ^1H NMR (400 MHz, DMSO-d_6): δ 8.62 (t, $J = 6.0$ Hz, 1H), 7.68 (d, $J = 8.4$ Hz, 2H), 7.41 (d, $J = 7.2$ Hz, 2H), 7.37-7.26 (m, 8H), 6.65 (d, $J = 8.4$ Hz, 6H), 4.74 (dd, $J = 7.8, 4.8$ Hz, 1H), 4.43 (d, $J = 6.0$ Hz, 1H), 3.27 (dd, $J = 13.0, 4.4$ Hz, 1H), 3.18 (dd, $J = 13.0, 8.0$ Hz, 1H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 166.2, 151.1, 144.0, 140.4, 128.8, 128.2, 128.1, 127.2, 127.1, 126.6, 126.1, 121.2, 111.2, 70.8, 51.0, 42.4; IR (Neat) 3361, 3062, 3032, 1728, 1608, 1512, 1059 cm^{-1} ; HRMS (m/z): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{22}\text{H}_{23}\text{N}_2\text{O}_2$, 347.1760; found, 347.1756

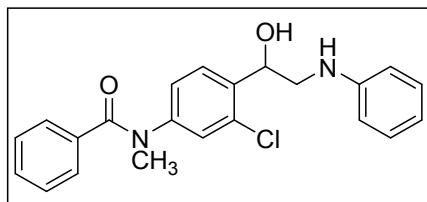
N-Benzyl-4-(1-hydroxy-2-(phenylamino)ethyl)-N-methylbenzamide



Yellow oil; R_f 0.35; (hexanes : ethyl acetate, 60:40 v/v): ^1H NMR (400 MHz, DMSO-d_6): δ 7.42-7.29 (m, 7H), 7.28-7.22 (m, 5H), 6.64 (d, $J = 8.4$ Hz, 2H), 4.74 (dd, $J = 7.8, 4.8$ Hz, 1H), 4.60 (s, 2H), 3.25 (dd, $J = 13.2, 4.4$ Hz, 1H) 3.15

(dd, $J = 13.2, 8.0$ Hz, 1H), 2.87 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d₆): δ 170.9, 149.6, 143.5, 137.4, 128.3, 128.1, 127.6, 126.8, 126.6, 126.5, 125.7, 122.8, 111.1, 70.7, 51.9, 50.8, 35.0; IR (Neat) 3489, 3402, 1610, 1529, 1491, 1070 cm⁻¹; HRMS (m/z): [M+H]⁺ calcd for C₂₃H₂₅N₂O₂, 361.1916; found, 361.1911

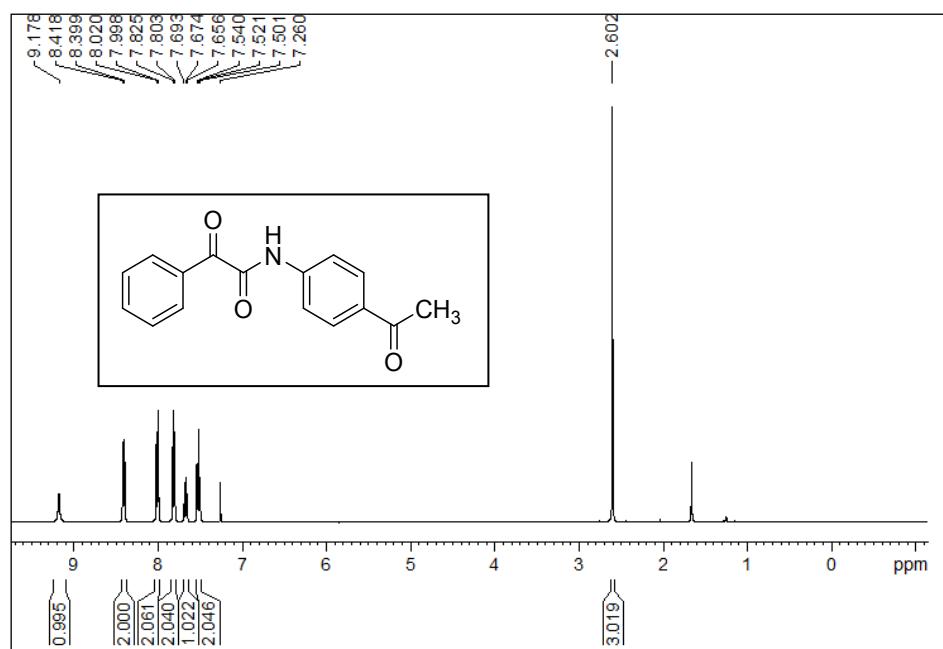
N-(3-Chloro-4-(1-hydroxy-2-(phenylamino)ethyl)phenyl)-N-methylbenzamide



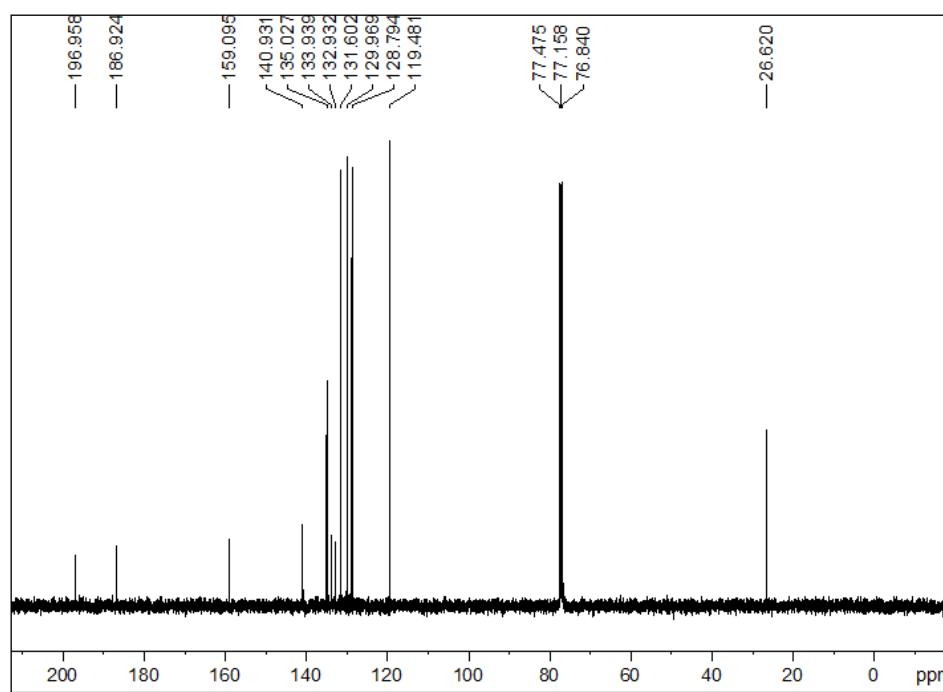
Colourless oil; R_f 0.43; (hexanes : ethyl acetate, 60:40 v/v): ^1H NMR (400 MHz, DMSO-d₆): δ 7.47 (d, $J = 7.6$ Hz, 2H), 7.38 (d, $J = 7.6$ Hz, 2H), 7.33 (t, $J = 6.8$ Hz, 3H), 7.15 (t, $J = 7.6$ Hz, 4H), 7.00 (d, $J = 8.4$ Hz 1H), 6.55 (d, $J = 8.4$ Hz 1H), 5.64 (d, $J = 4.4$ Hz, 1H), 5.44 (t, $J = 5.2$ Hz, 1H), 4.79-4.71 (m, 1H), 3.45 (s, 3H), 3.20-3.10 (m, 1H); ^{13}C NMR (100 MHz, CDCl₃): δ 168.1, 145.3, 145.0, 143.5, 136.0, 134.8, 134.0, 129.2, 128.1, 127.5, 126.9, 126.0, 123.4, 116.4, 110.0, 70.5, 50.4, 38.3; IR (Neat) 3404, 3064, 1600, 1526, 1495, 1459, 1060 cm⁻¹; HRMS (m/z): [M+H]⁺ calcd for C₂₂H₂₂N₂O₂Cl, 381.1370; found, 381.1355.

References:

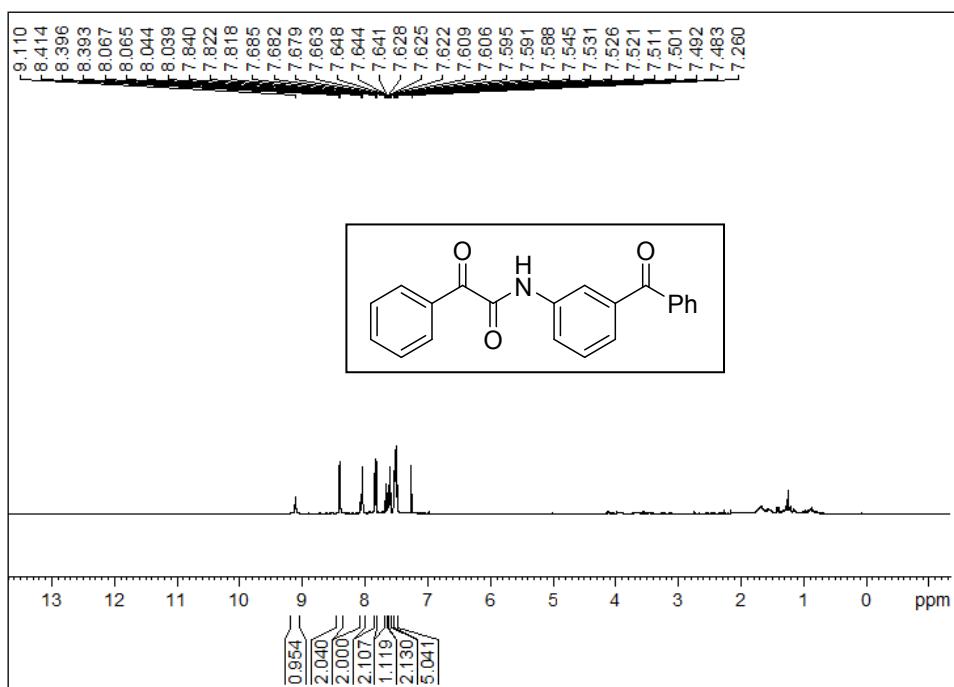
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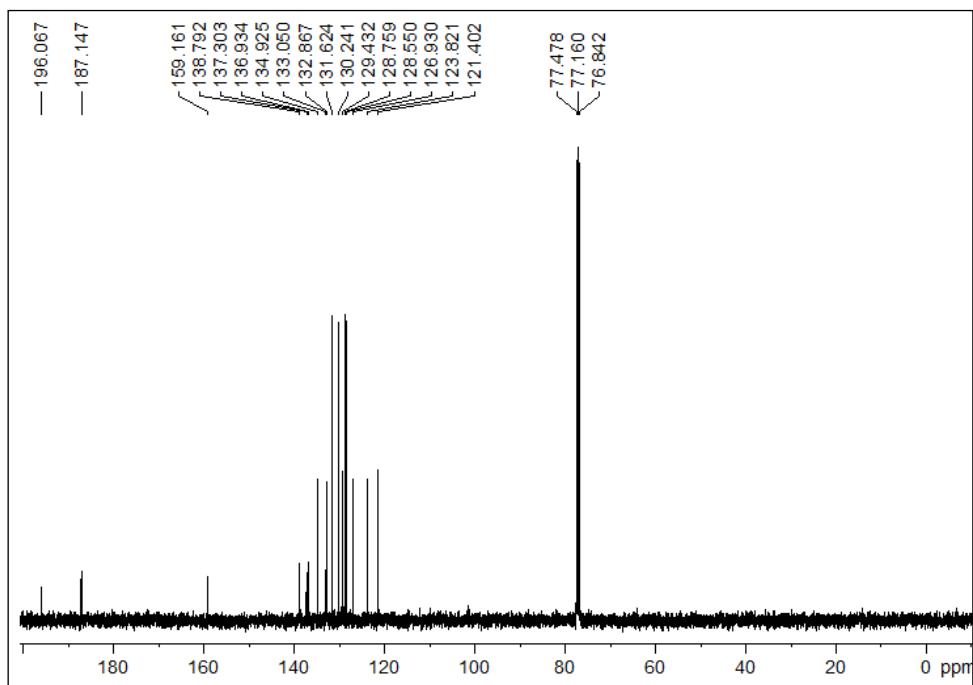
400 Mz ^1H -NMR spectra of **4a** in CDCl_3



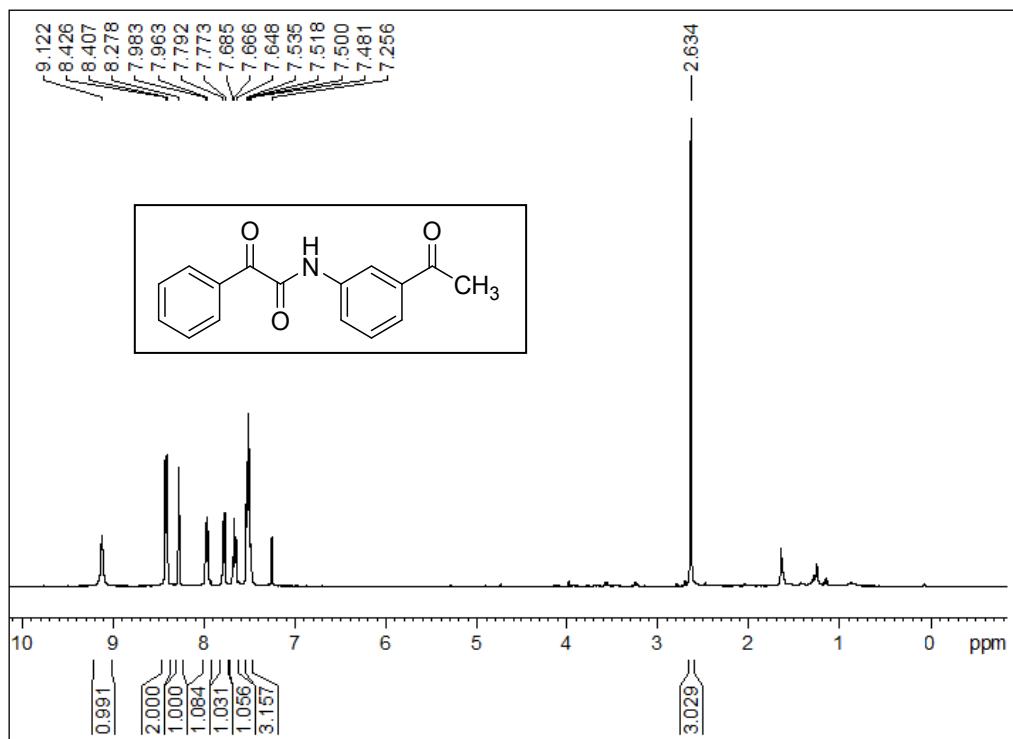
100 Mz ^{13}C -NMR spectra of **4a** in CDCl_3



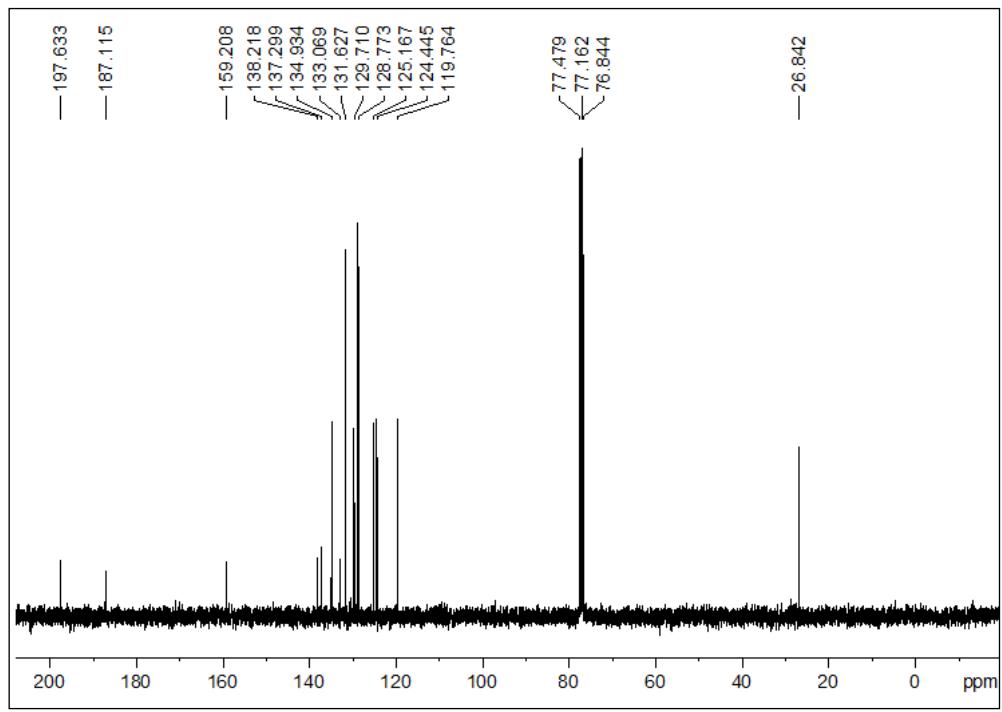
400 Mz ¹H-NMR spectra of **4b** in CDCl₃



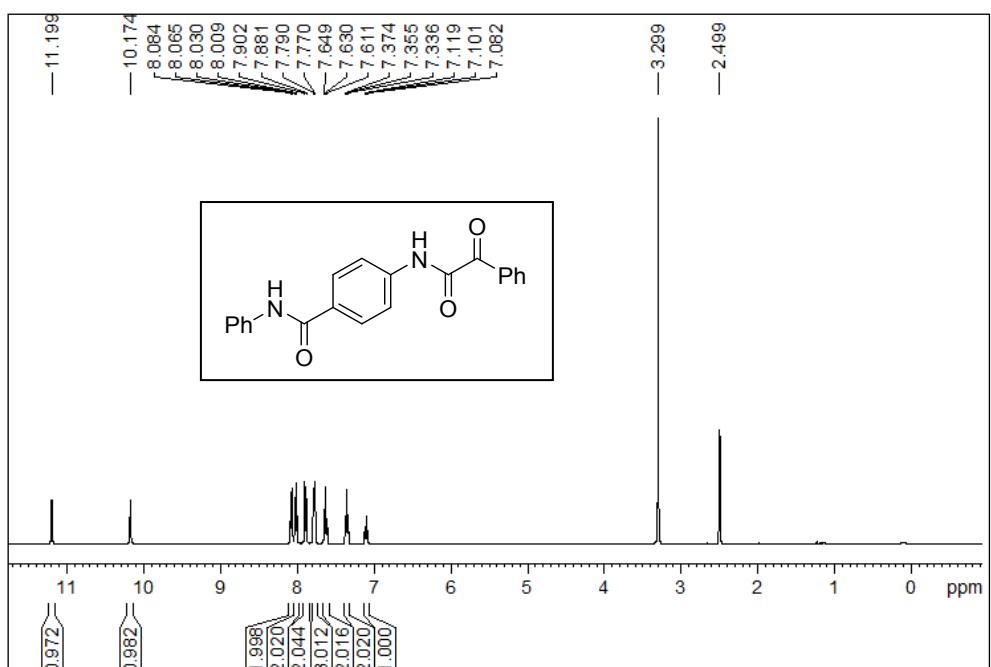
100 Mz ¹³C-NMR spectra of **4b** in CDCl₃



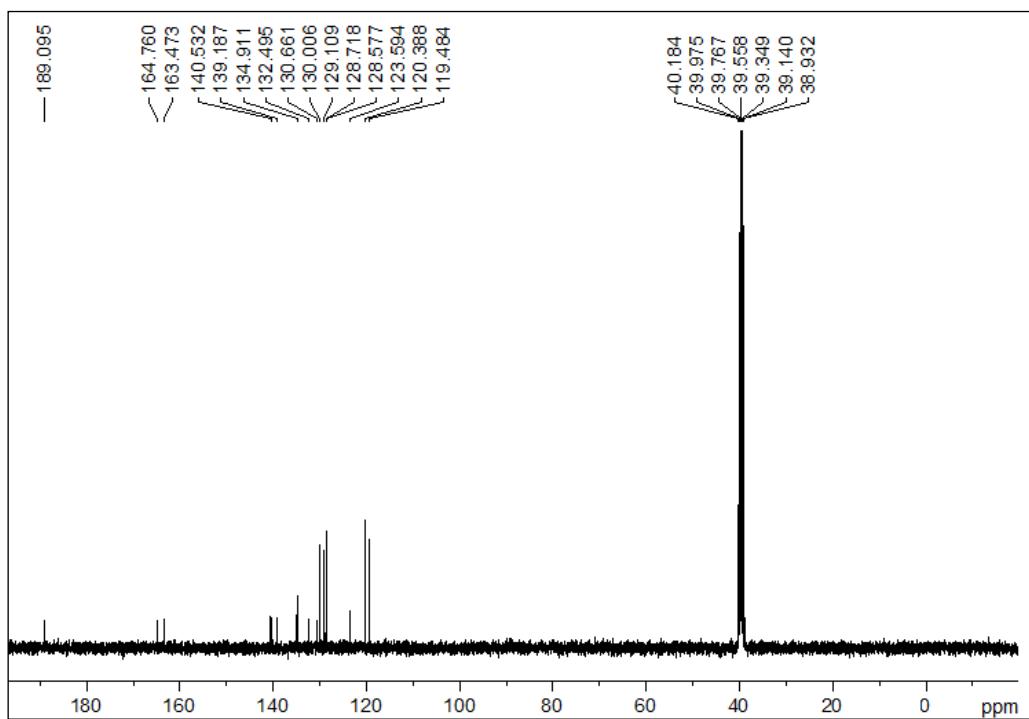
400 Mz ^1H -NMR spectra of **4c** in CDCl_3



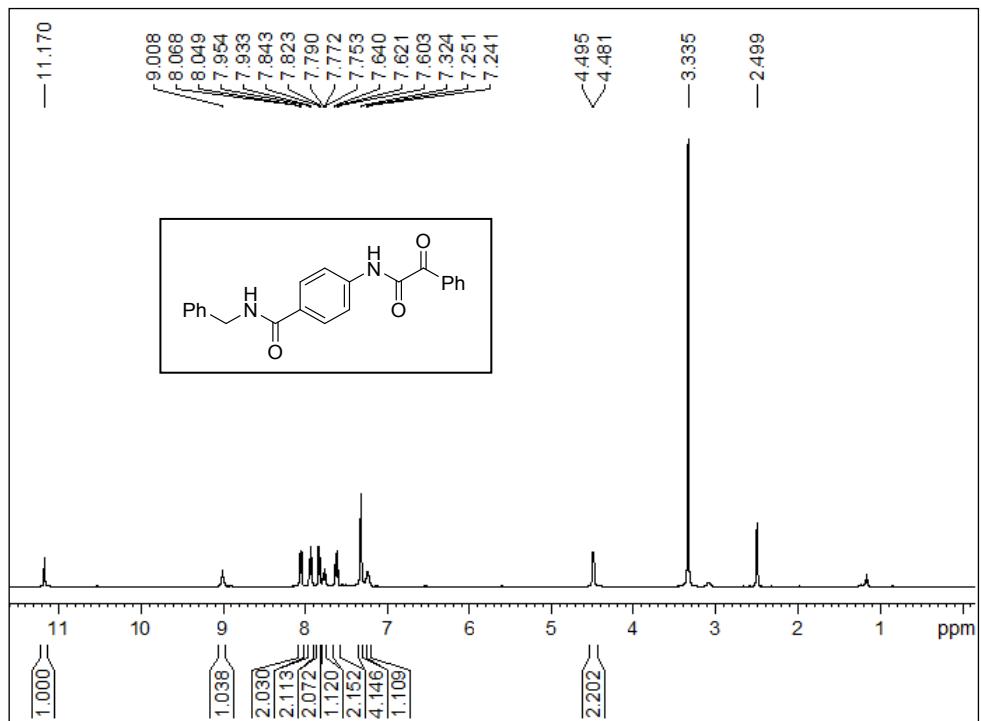
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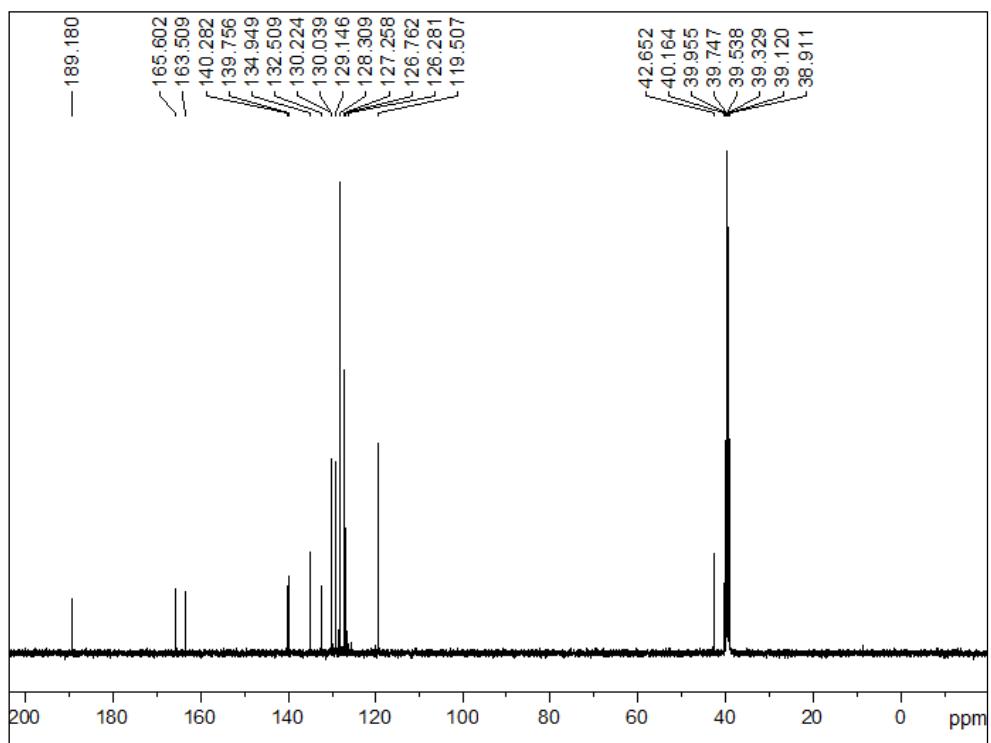
400 Mz ^1H -NMR spectra of **6a** in DMSO-d_6



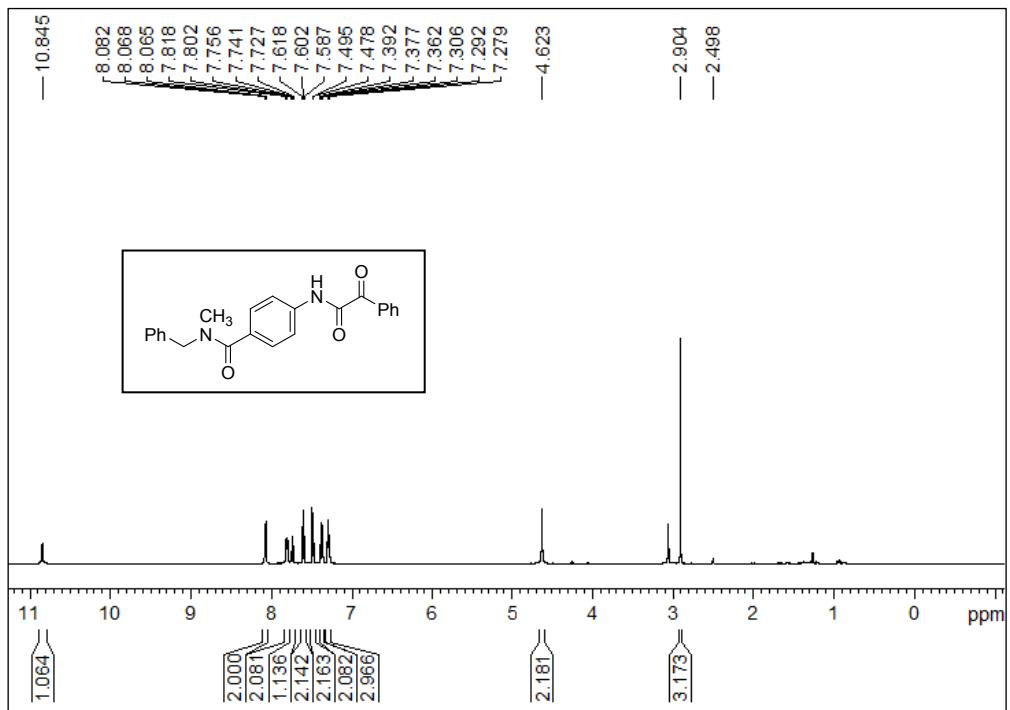
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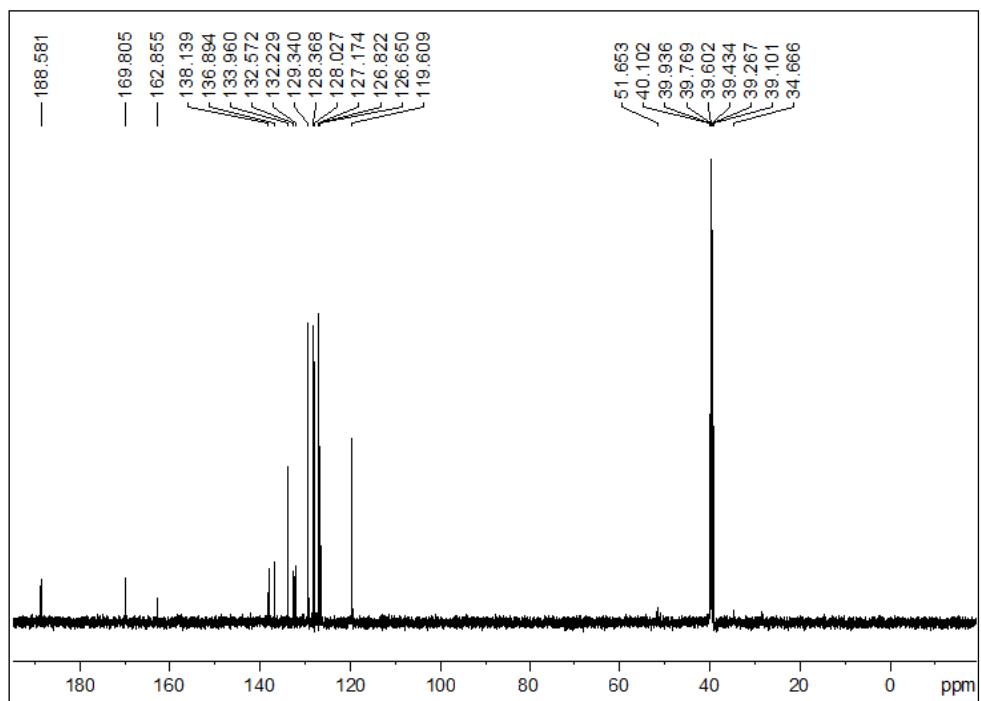
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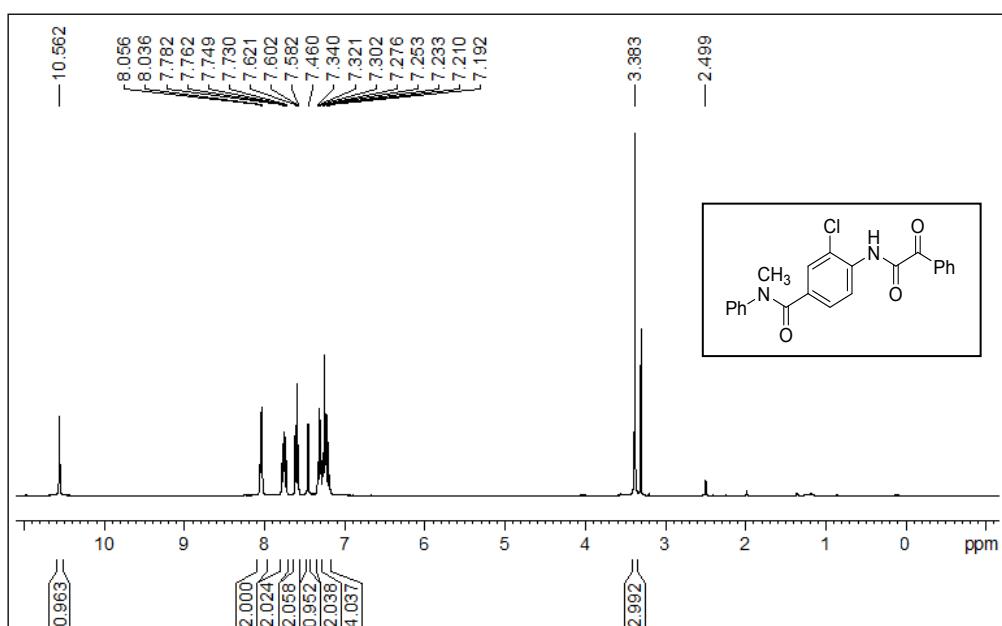
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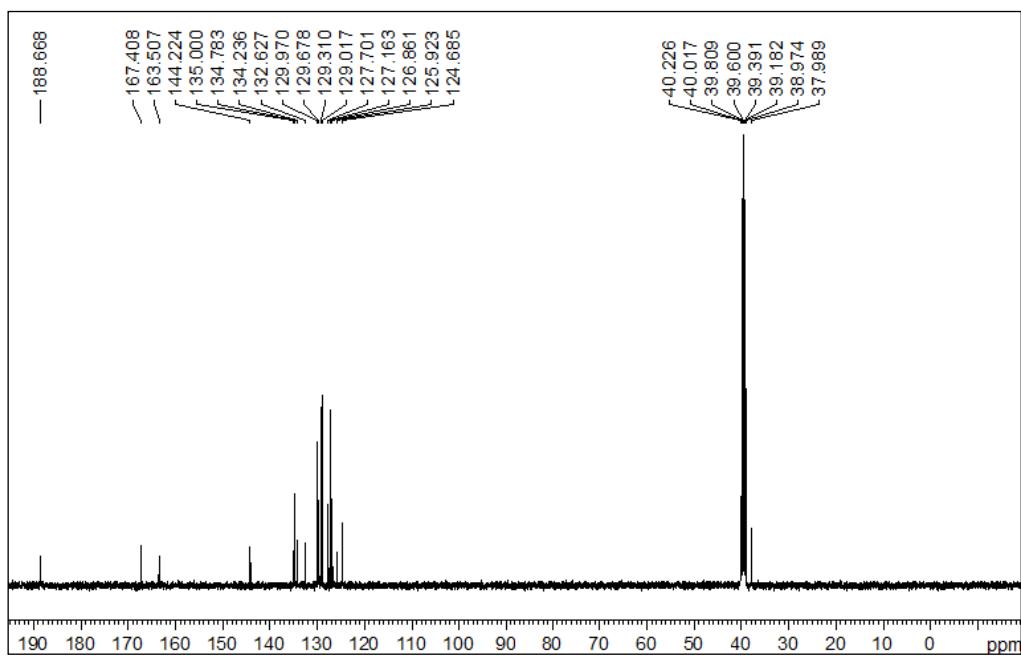
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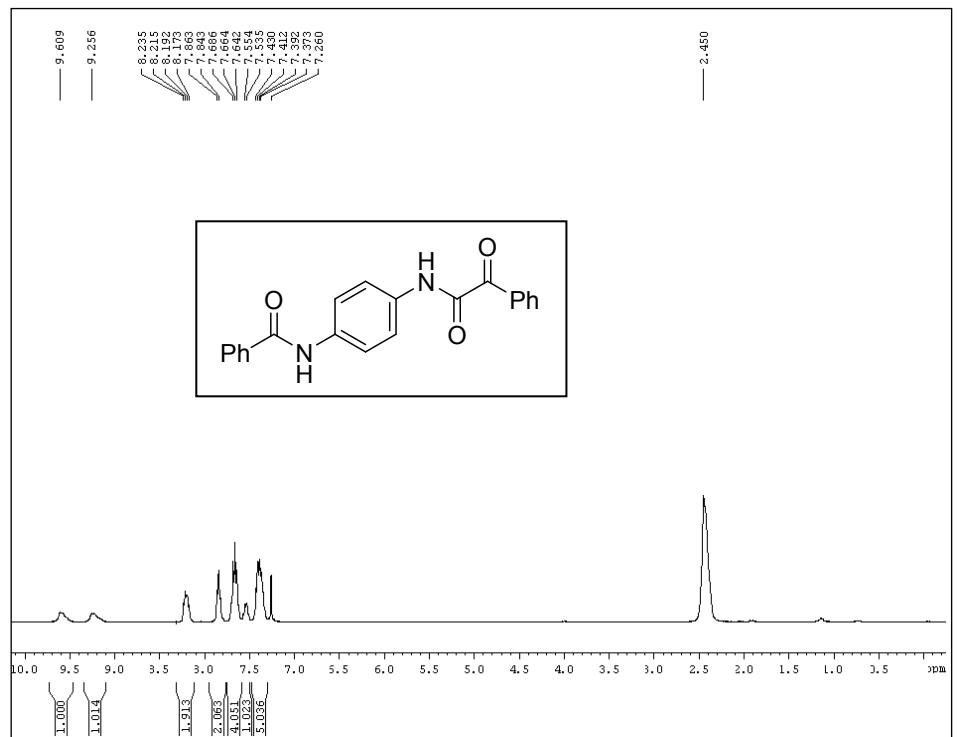
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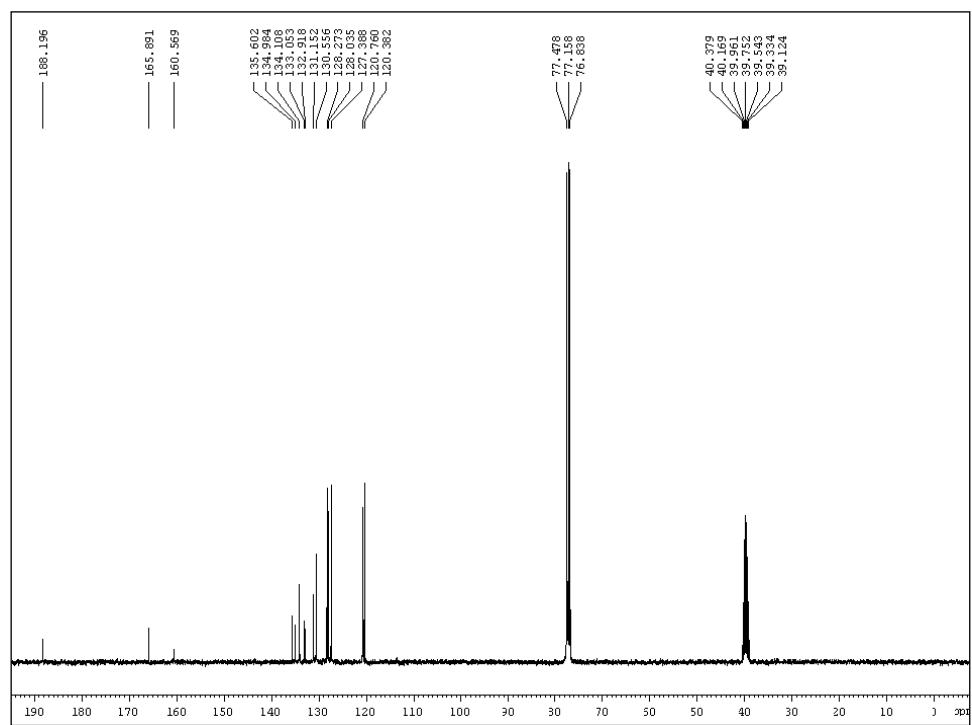
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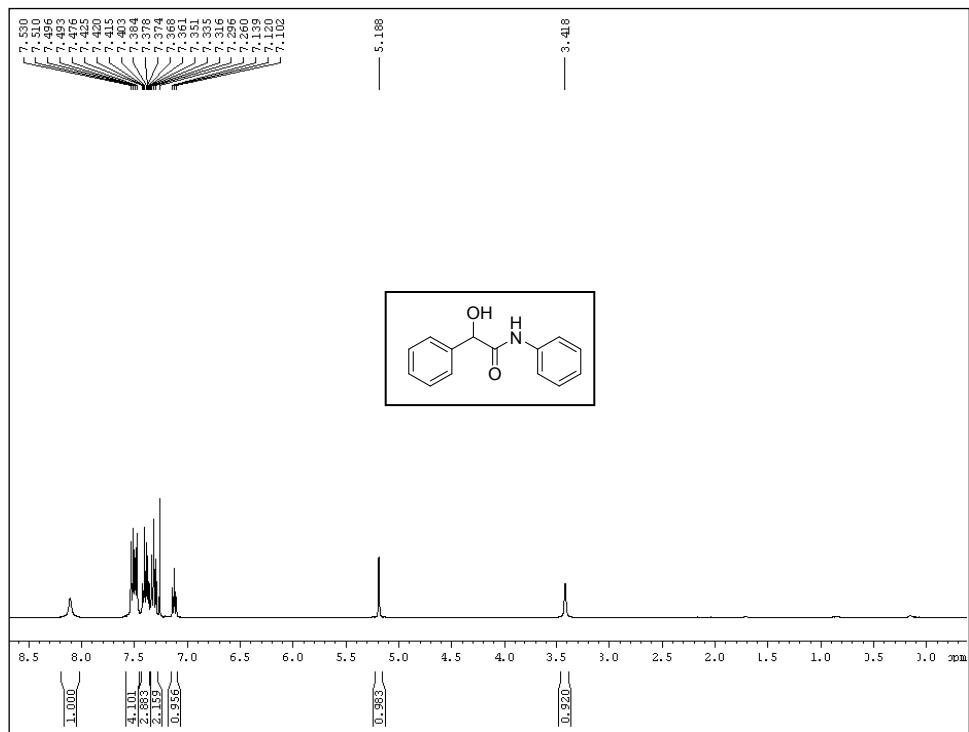
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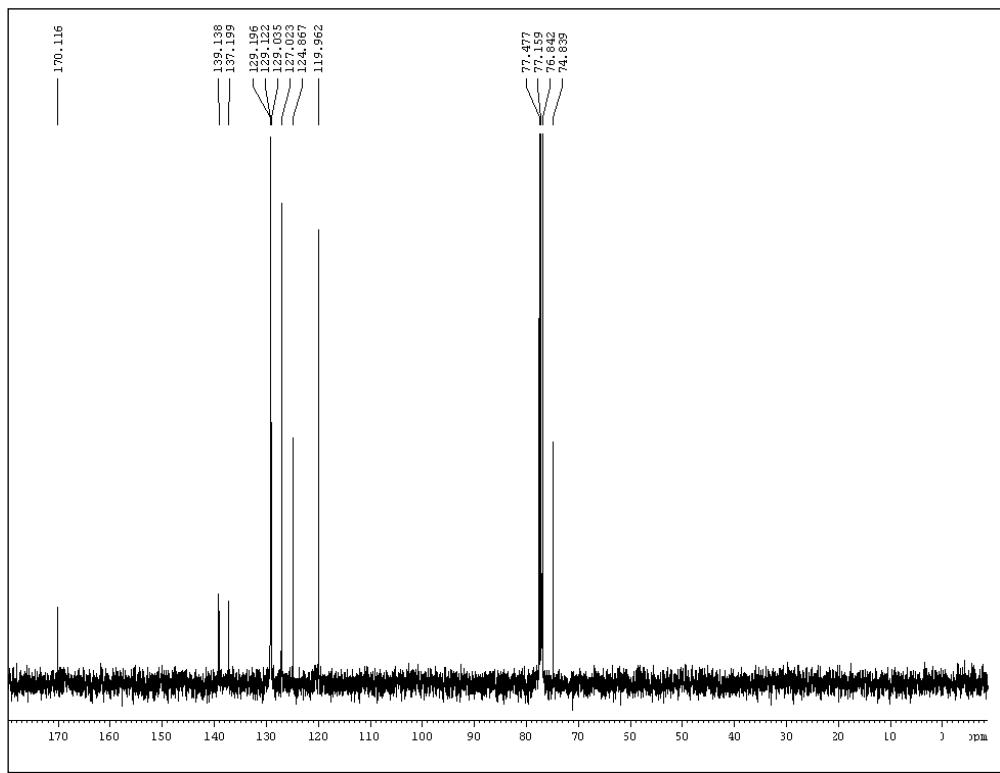
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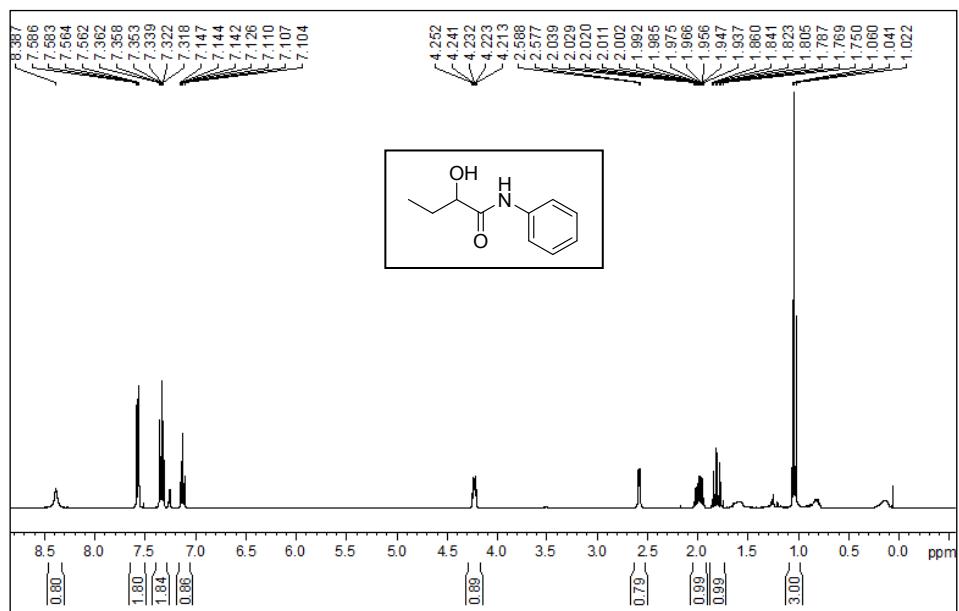
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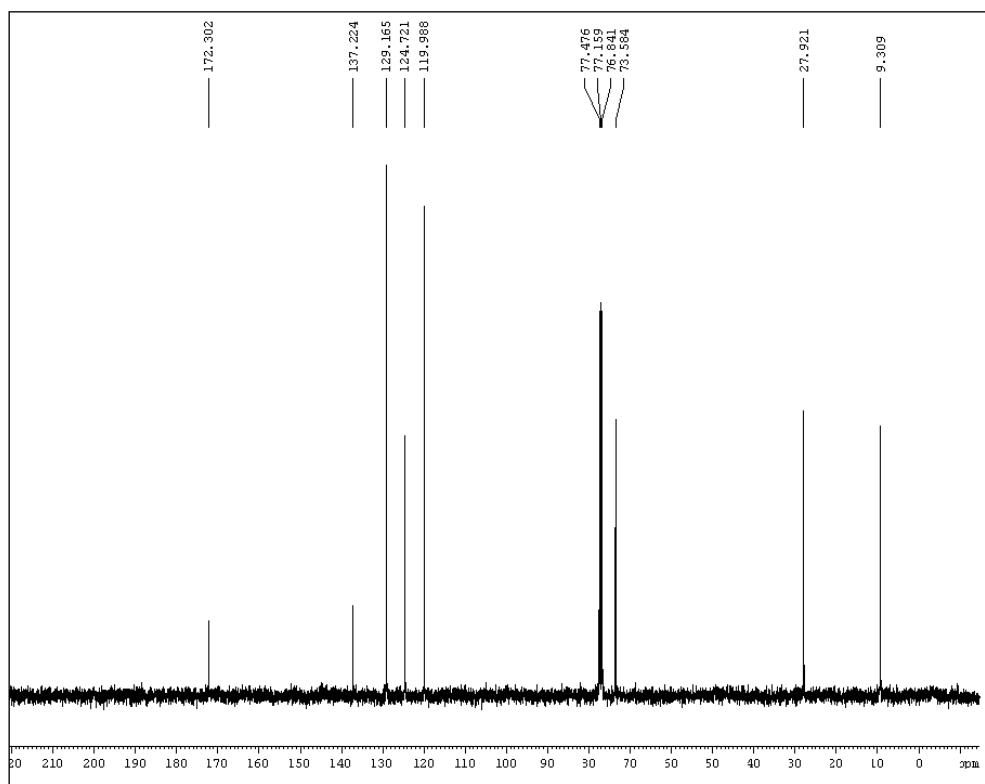
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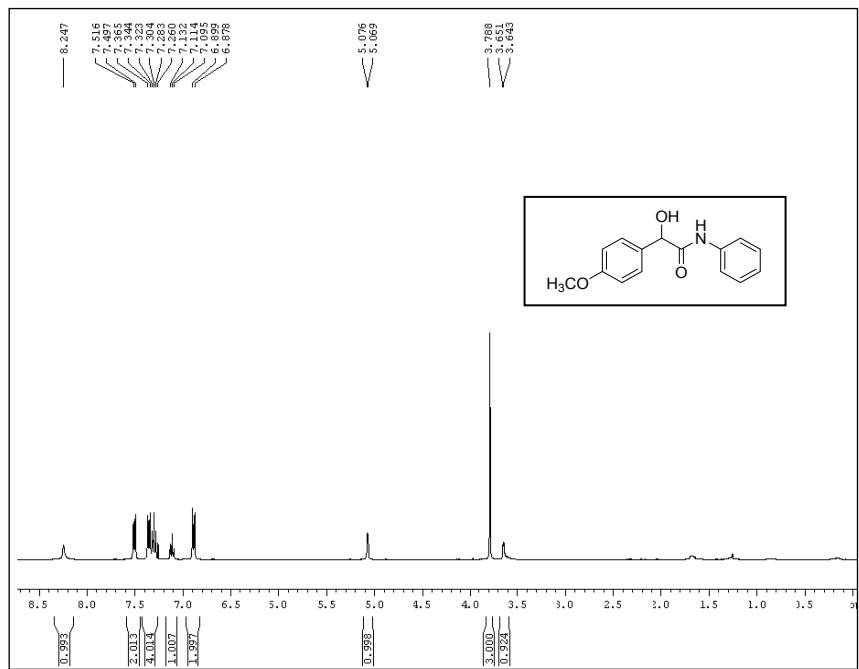
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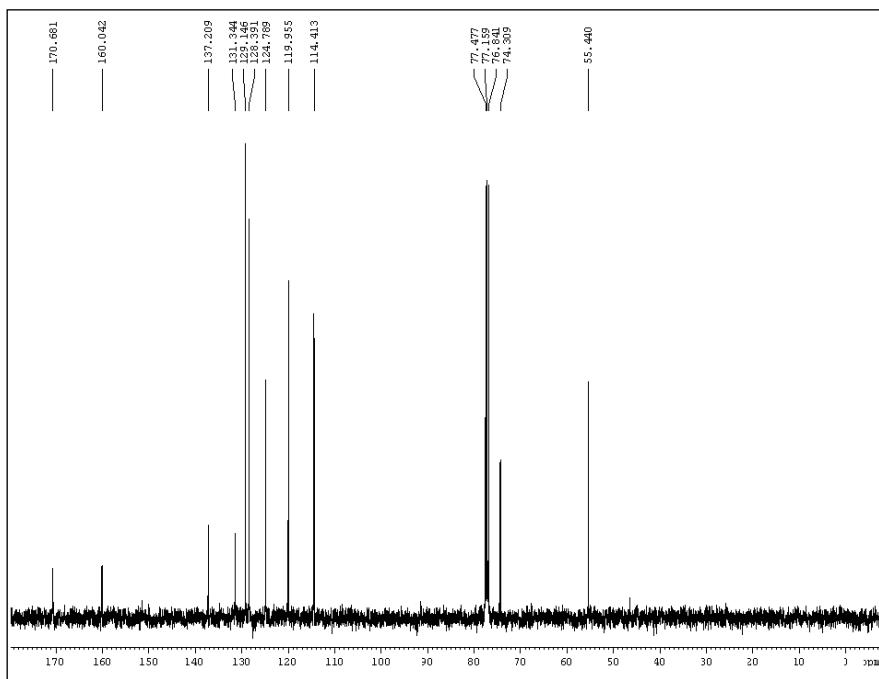
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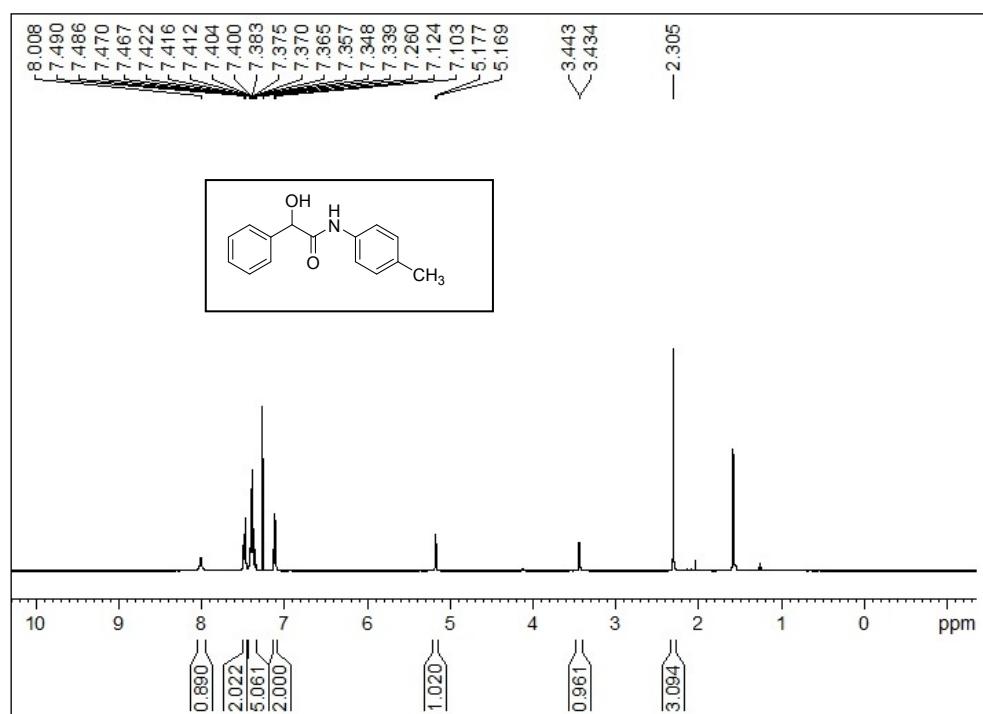
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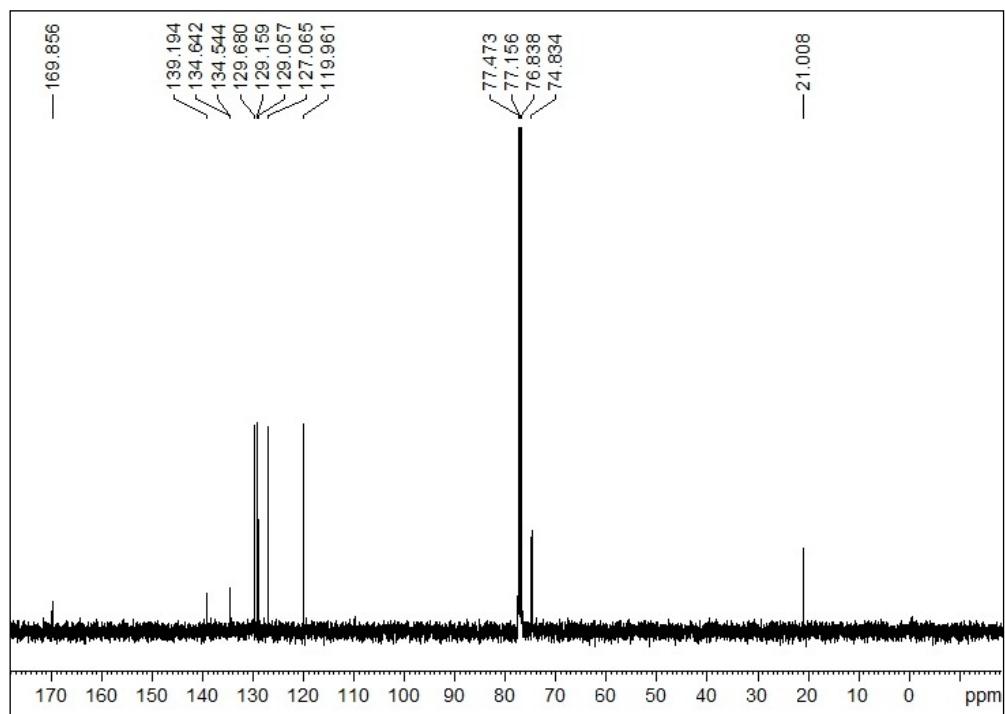
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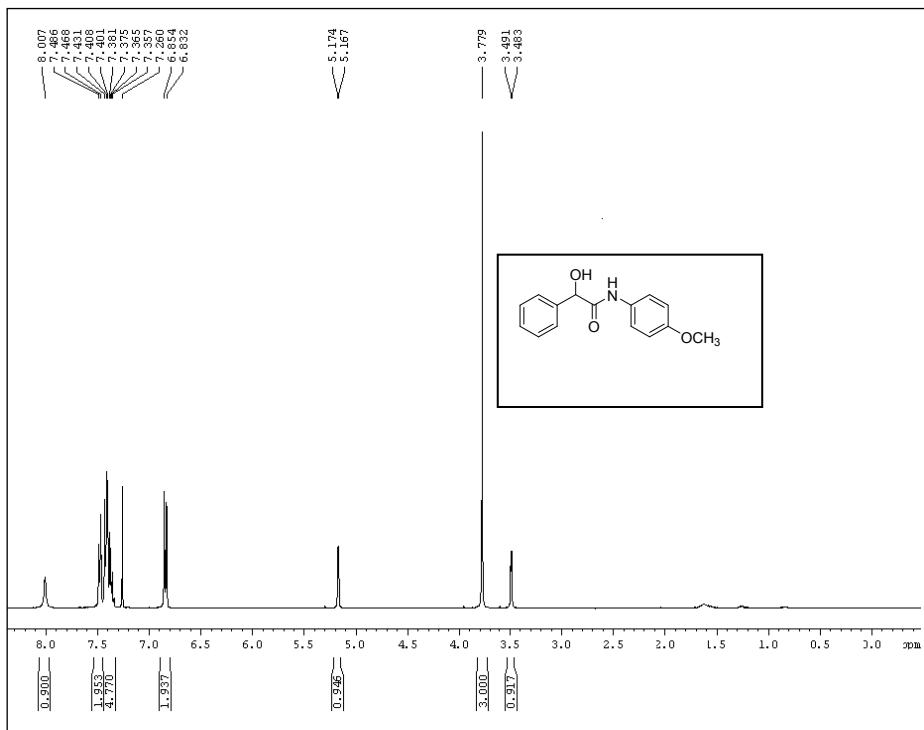
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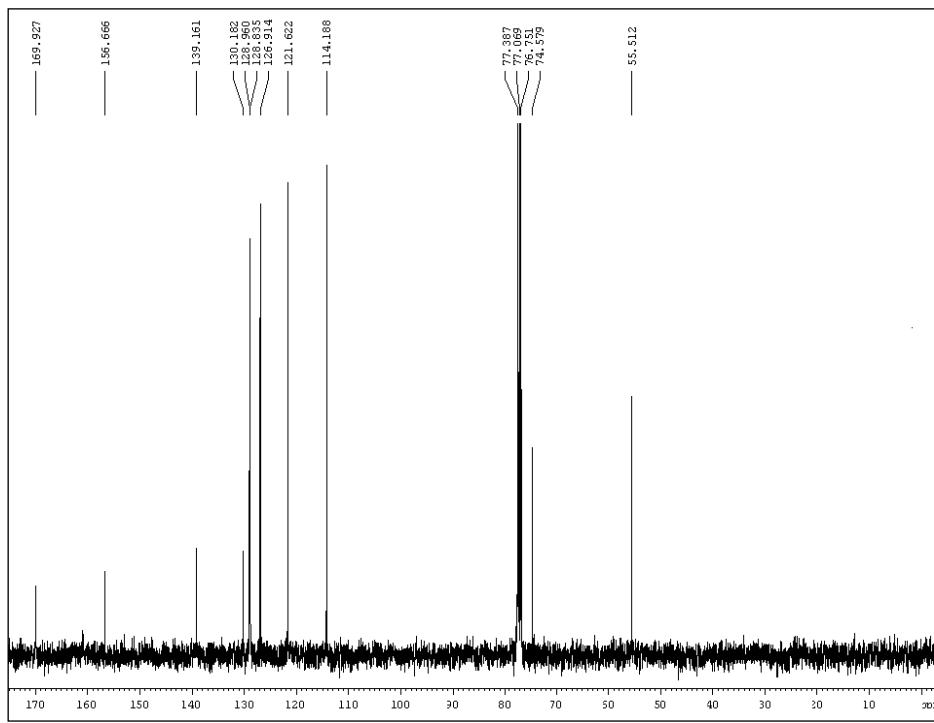
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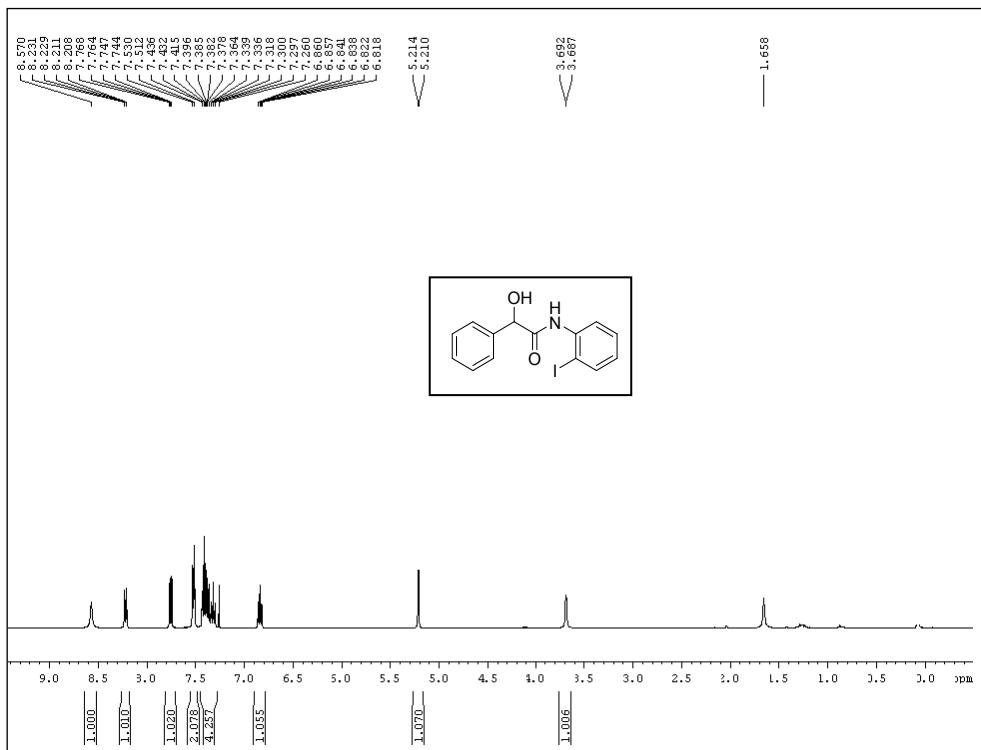
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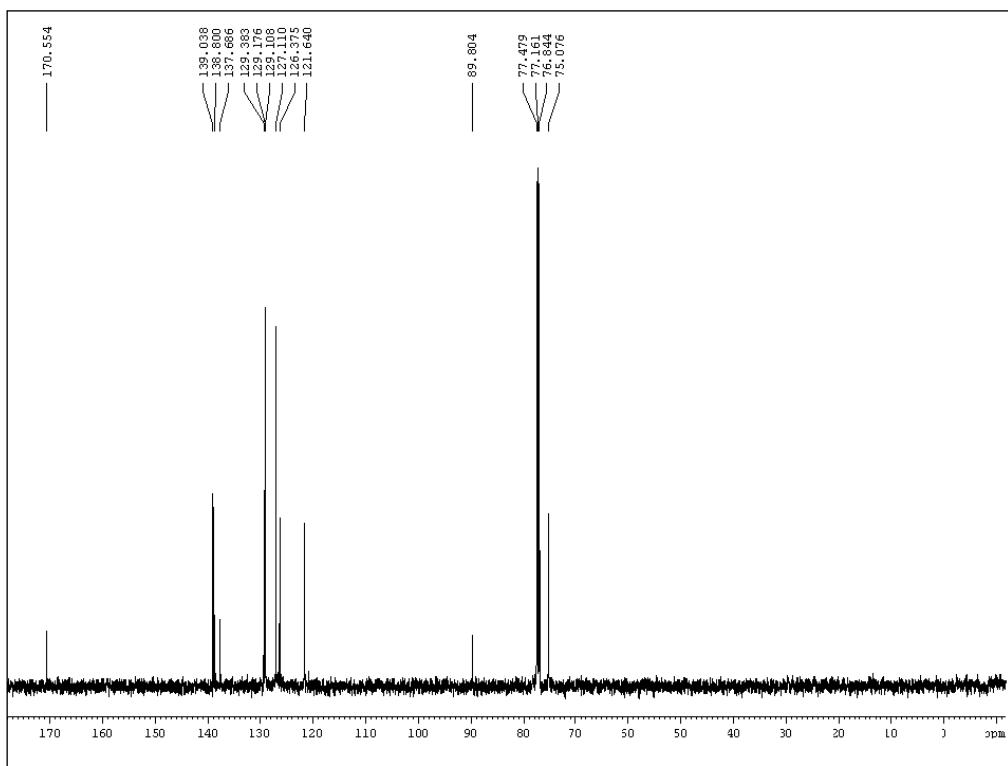
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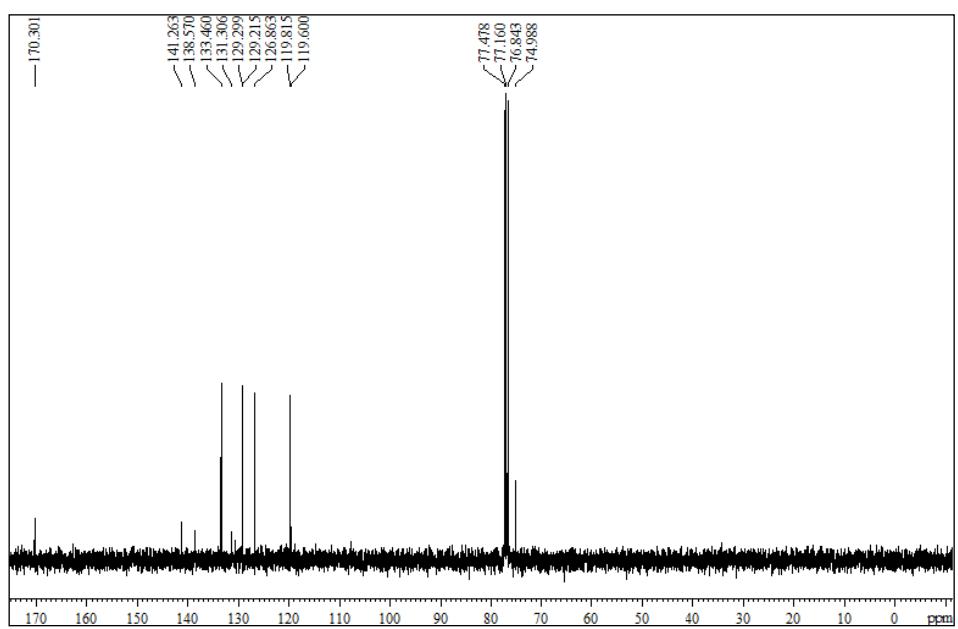
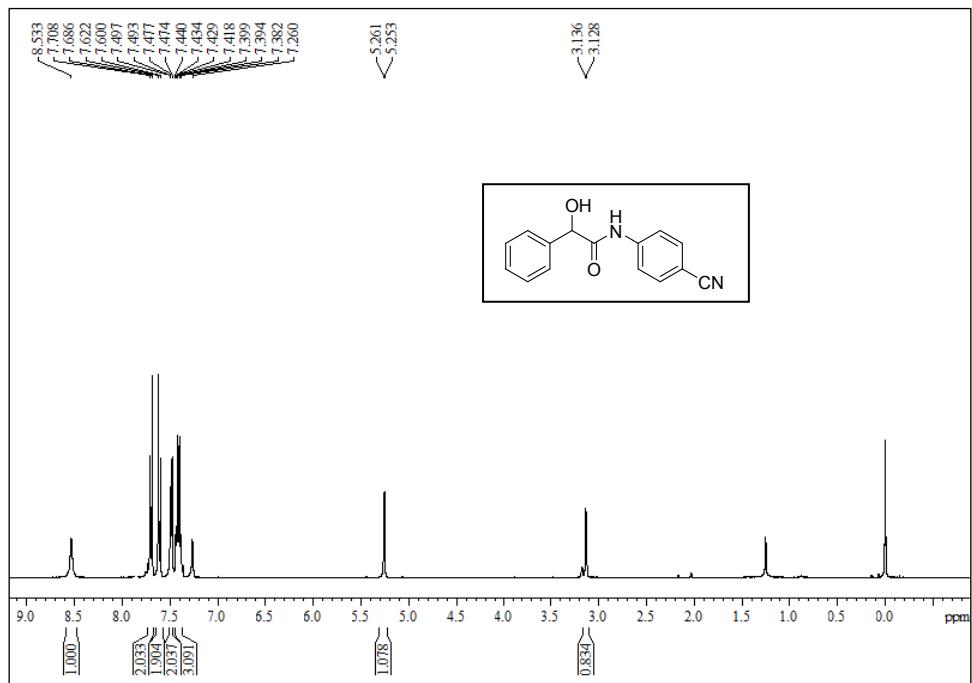
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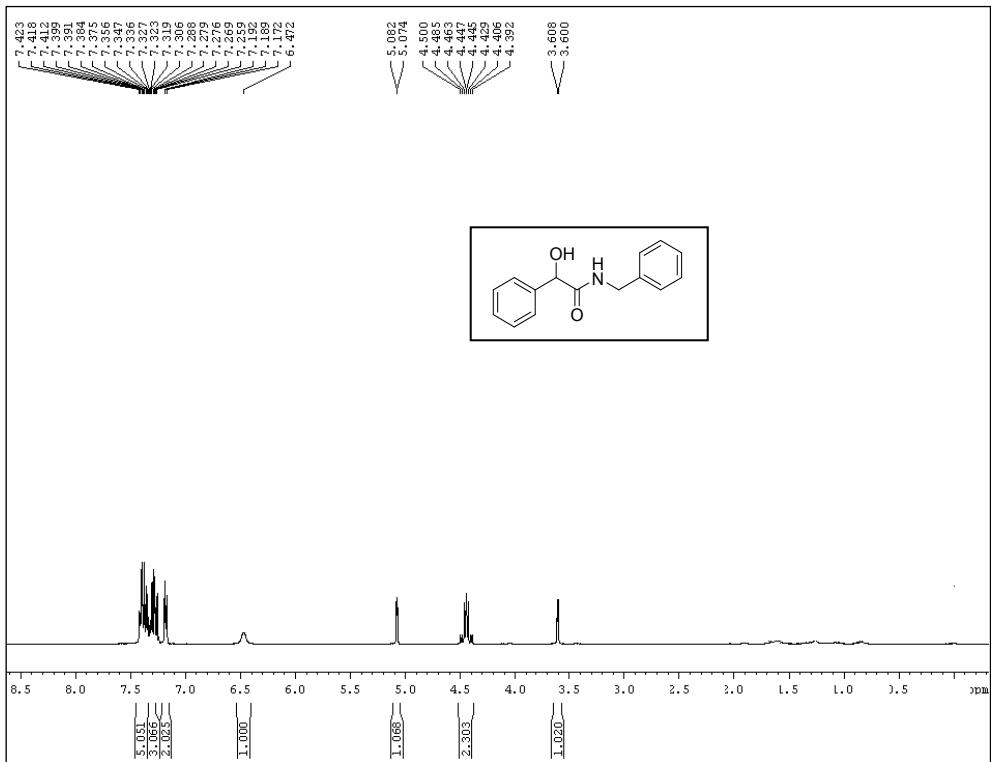


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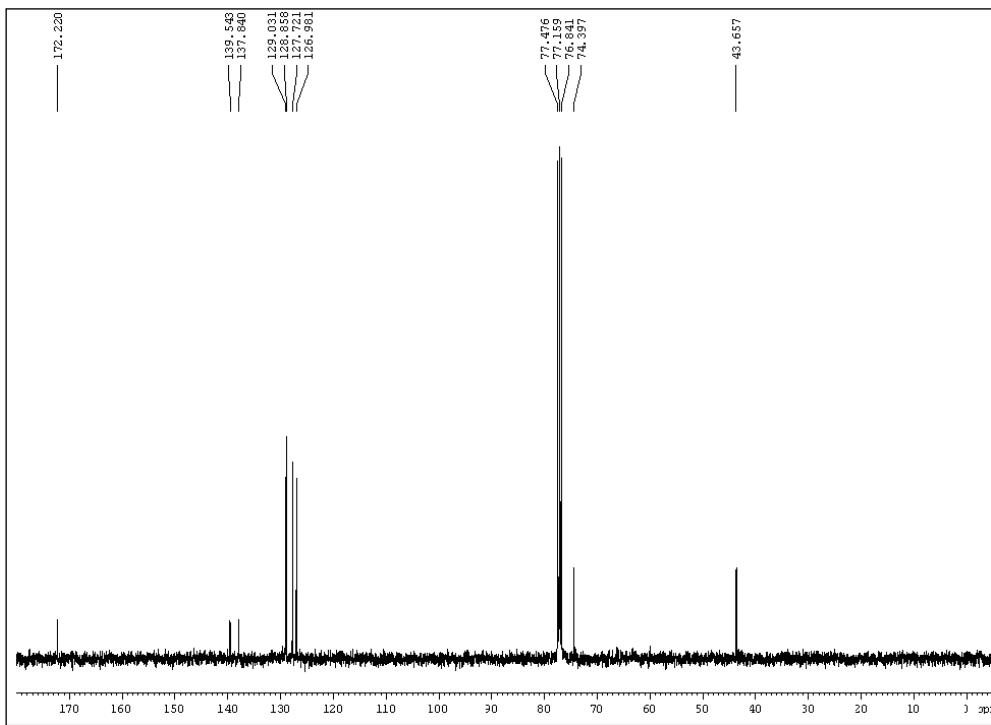


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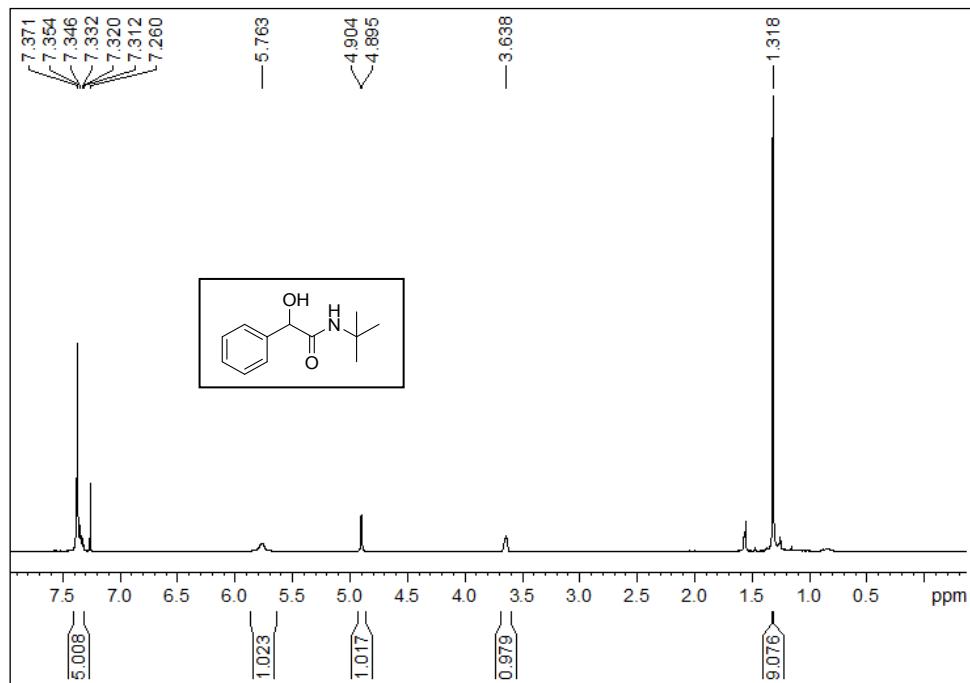




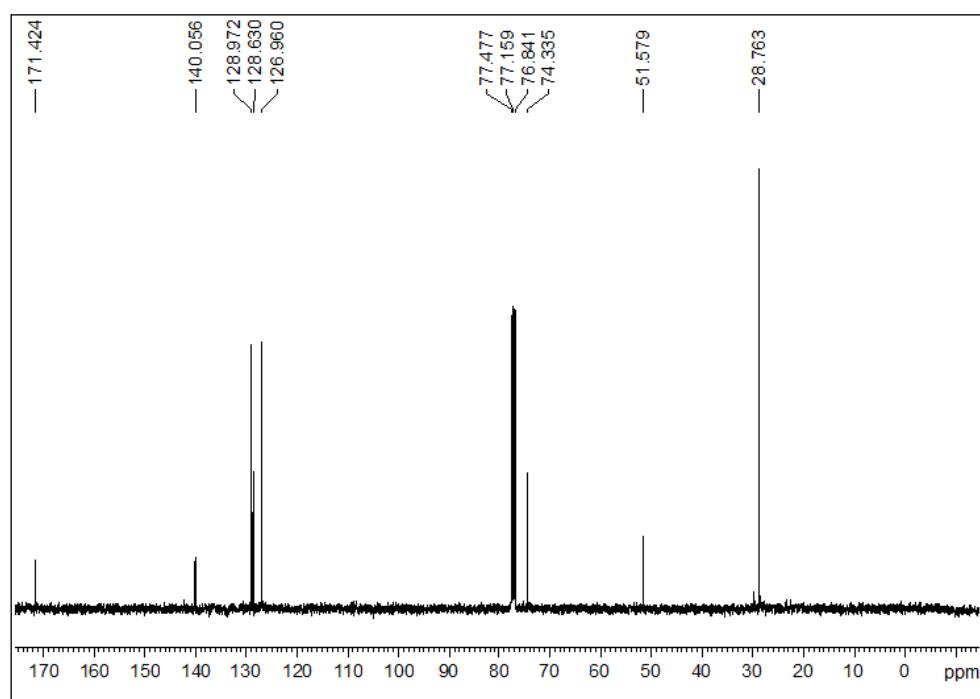
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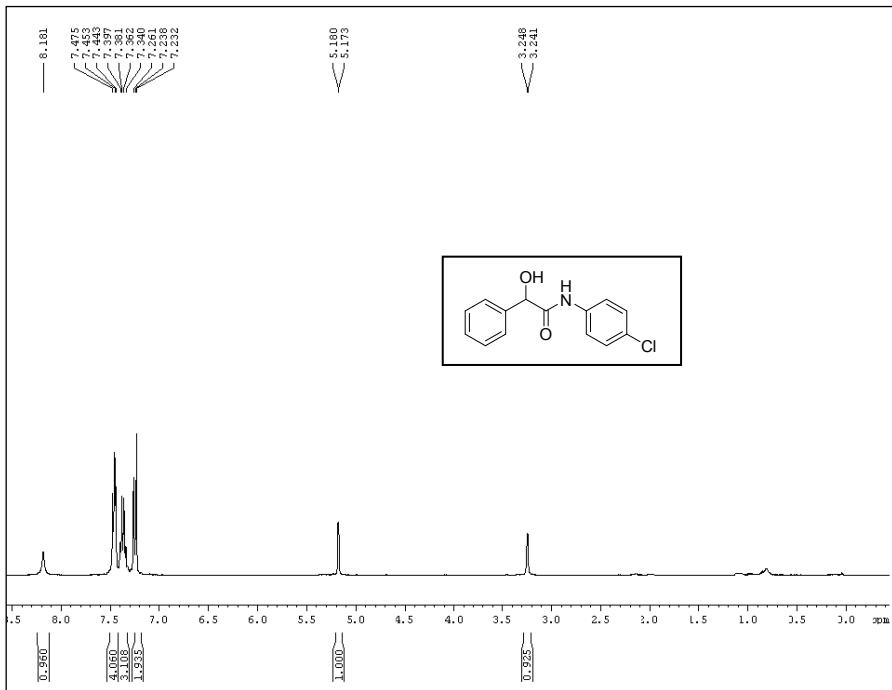
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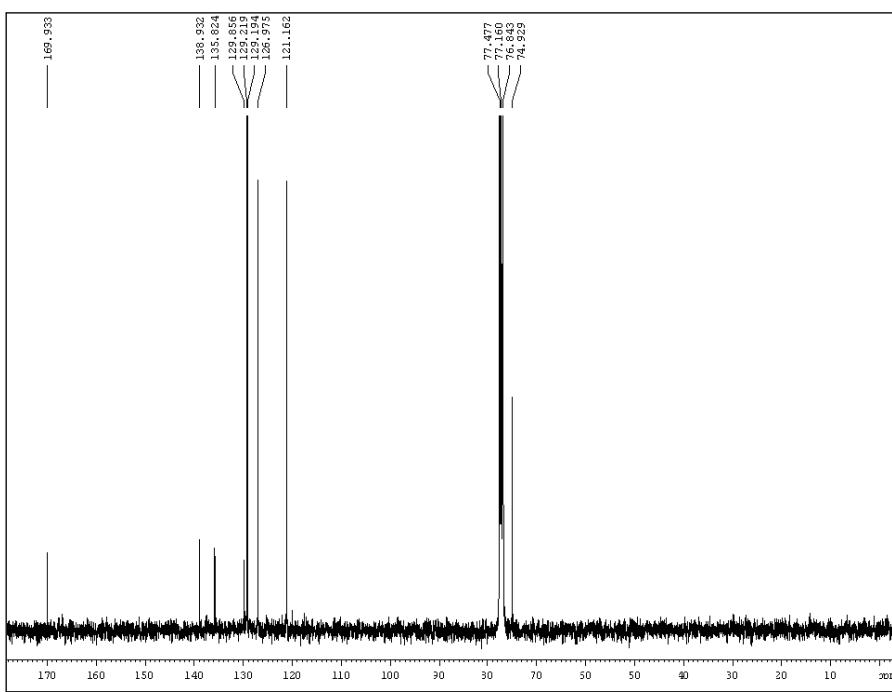
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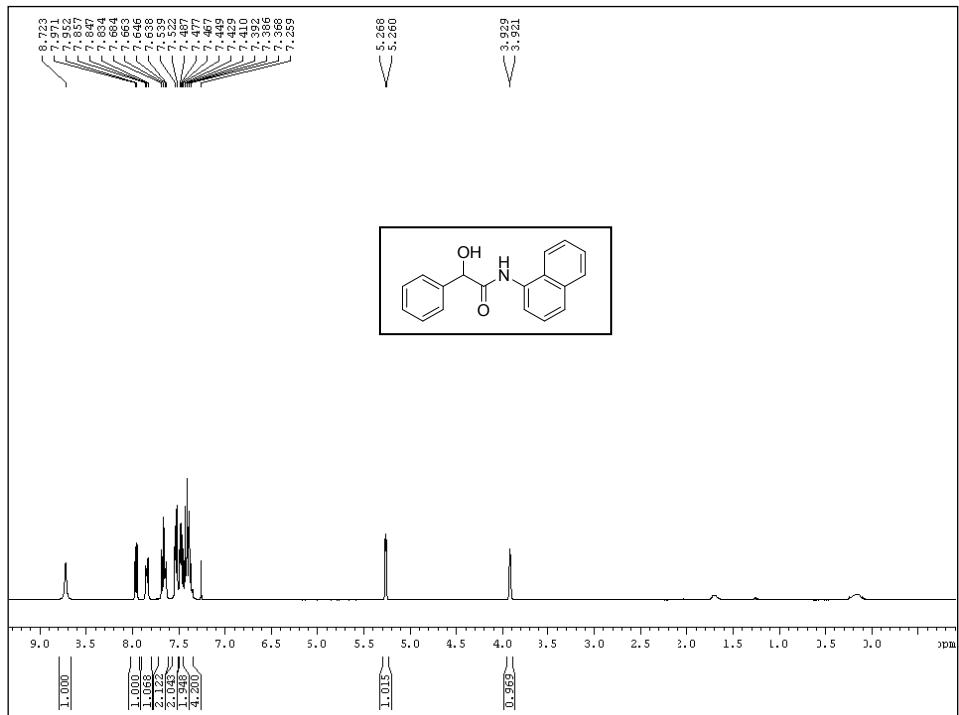
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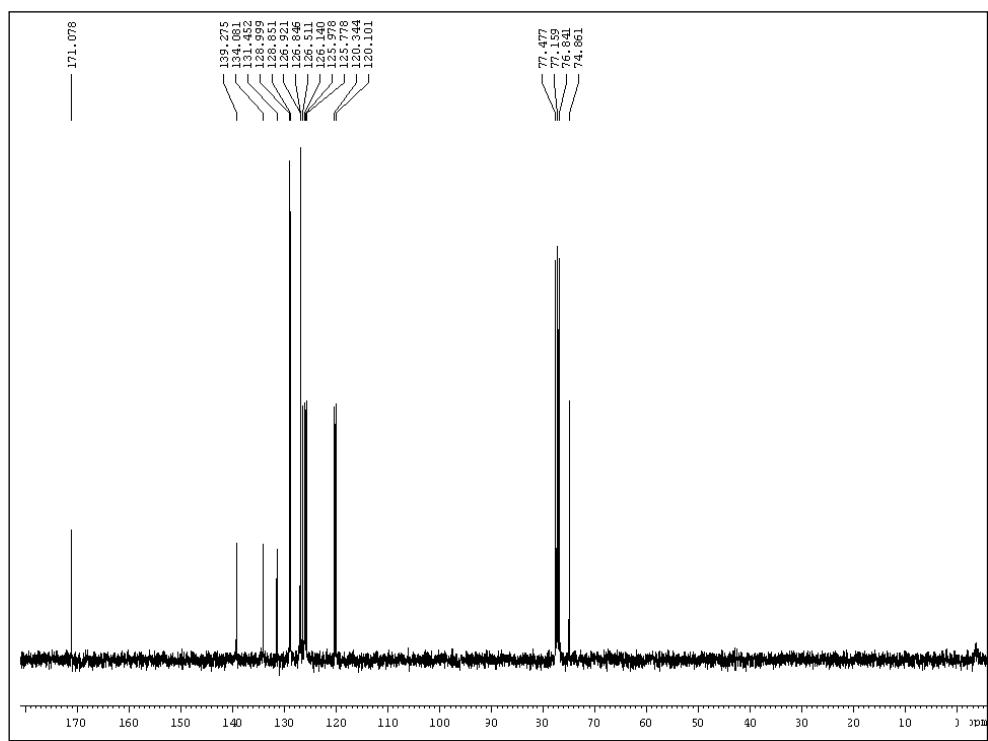
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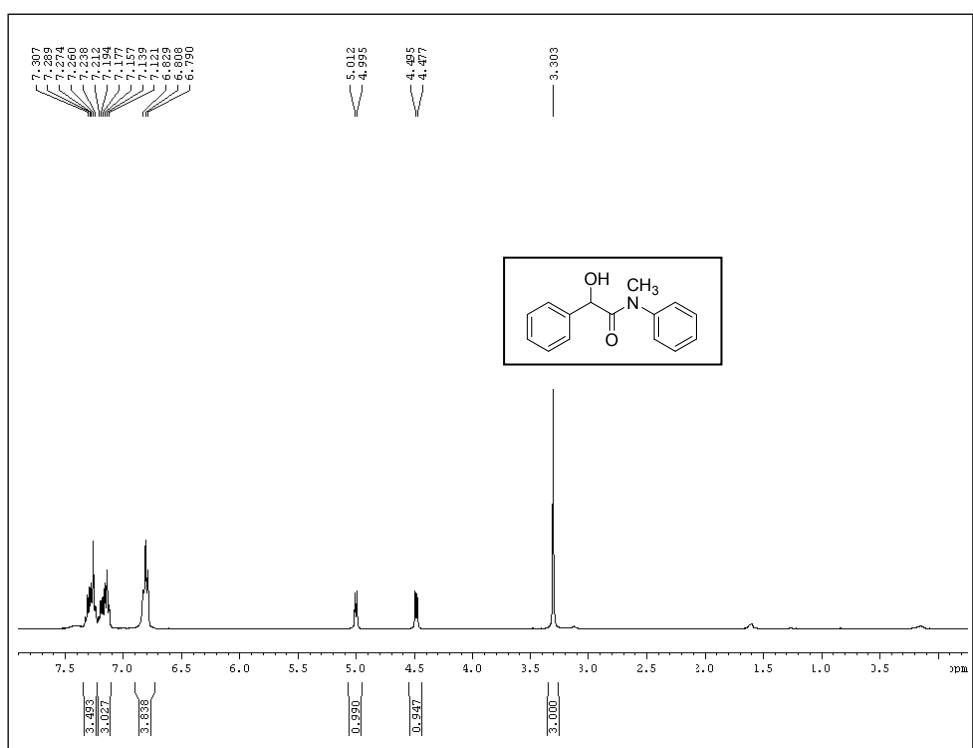
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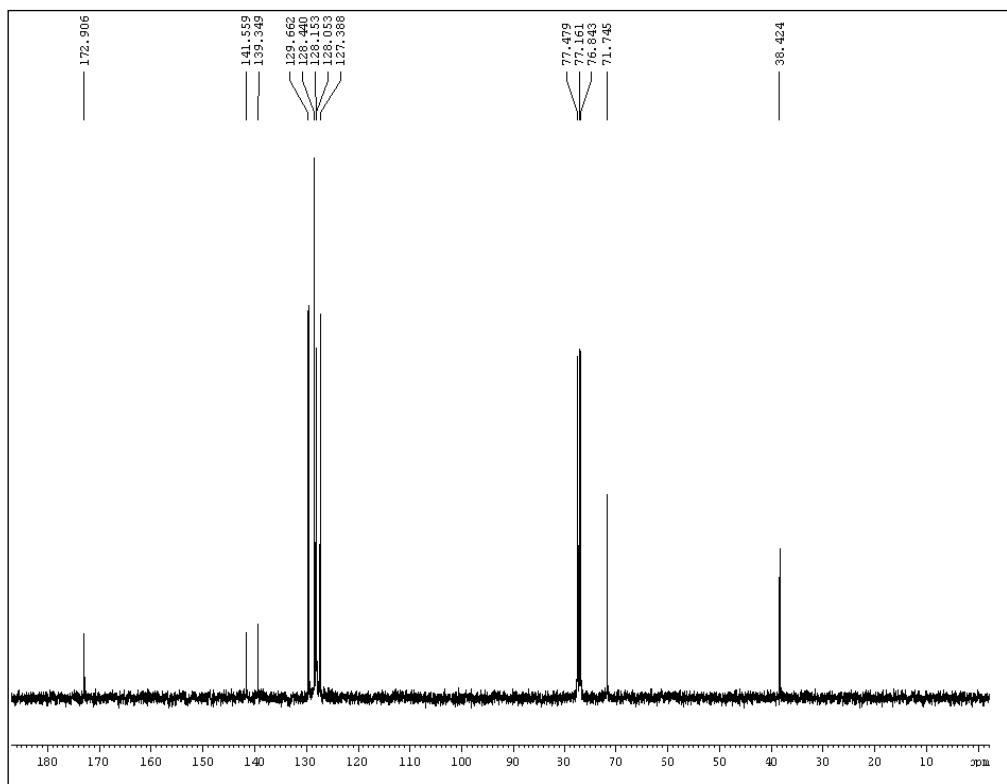
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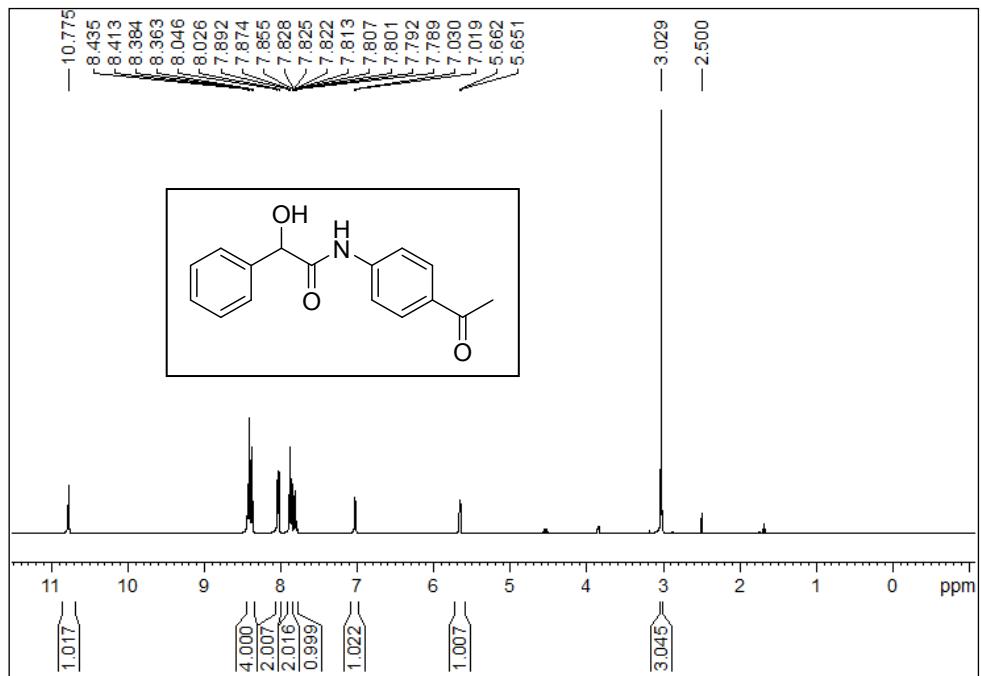
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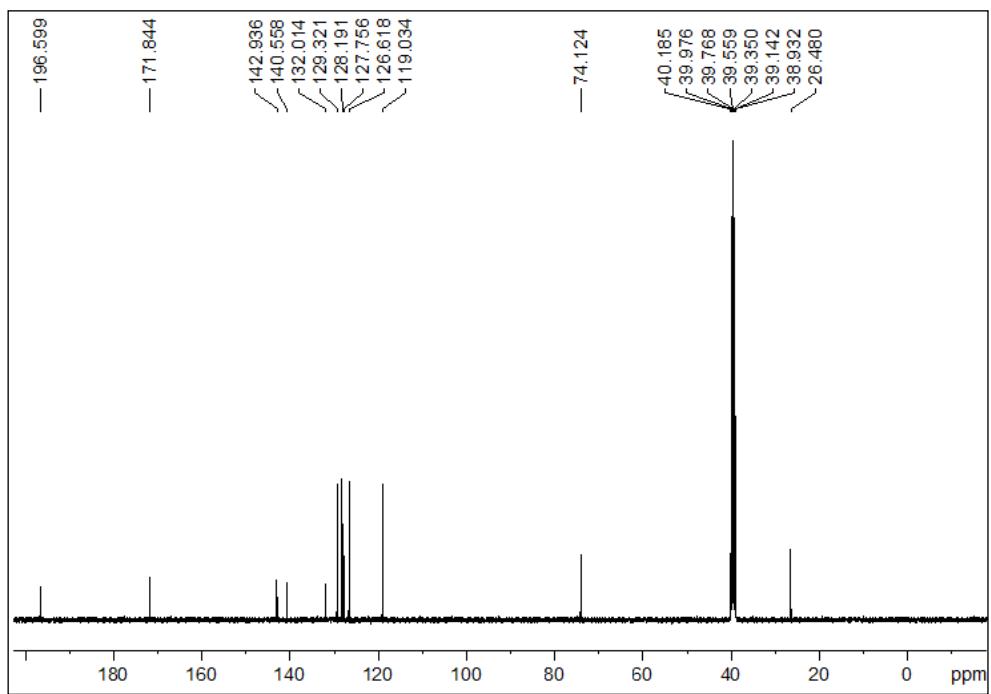
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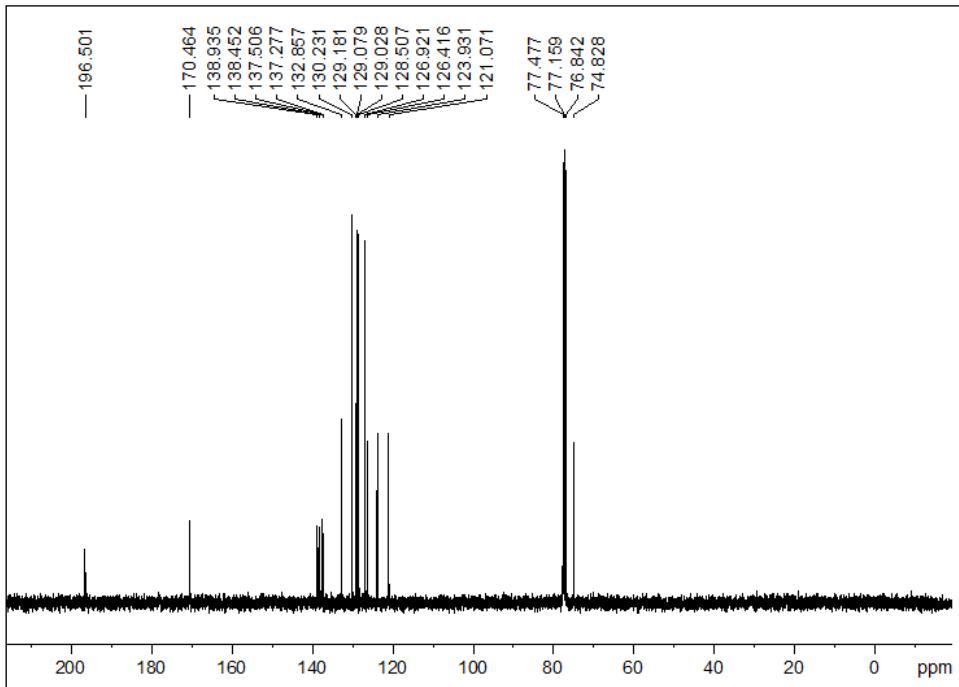
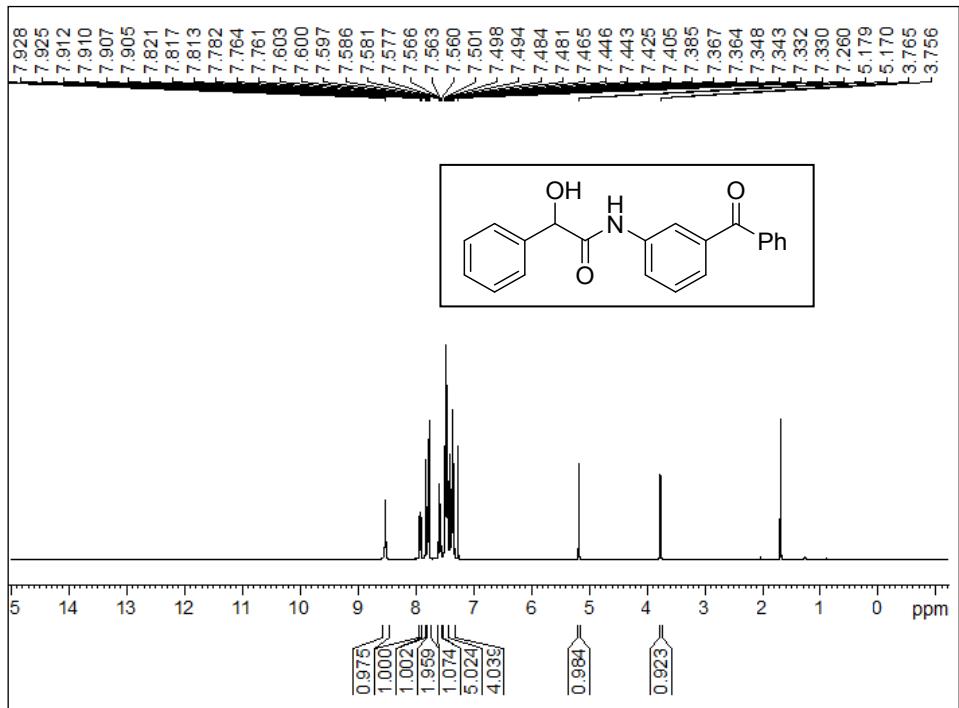
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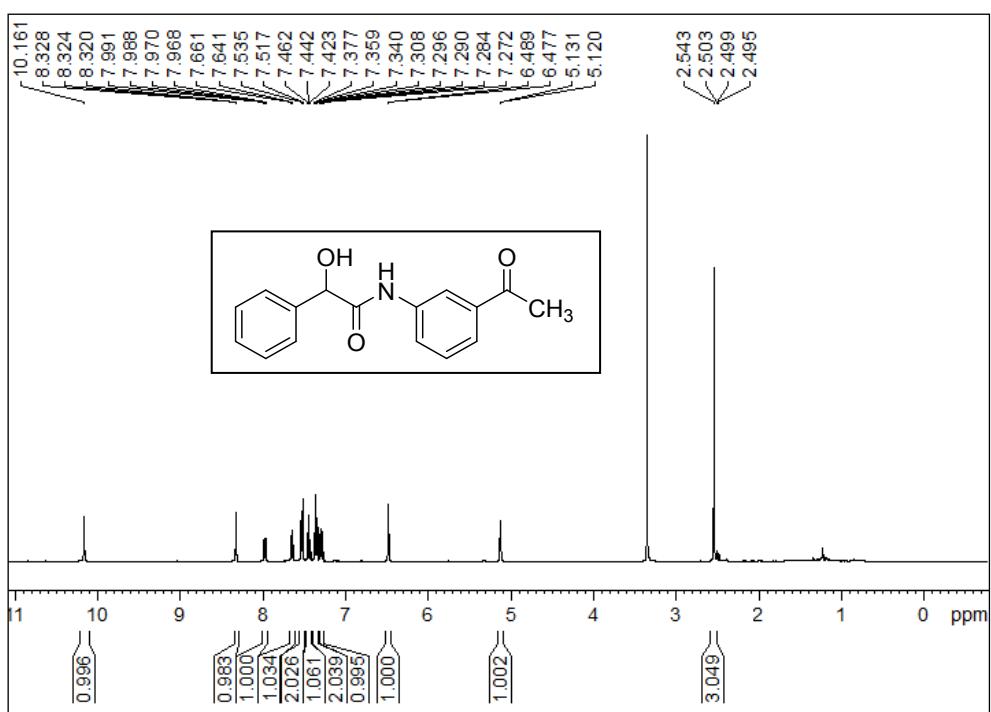


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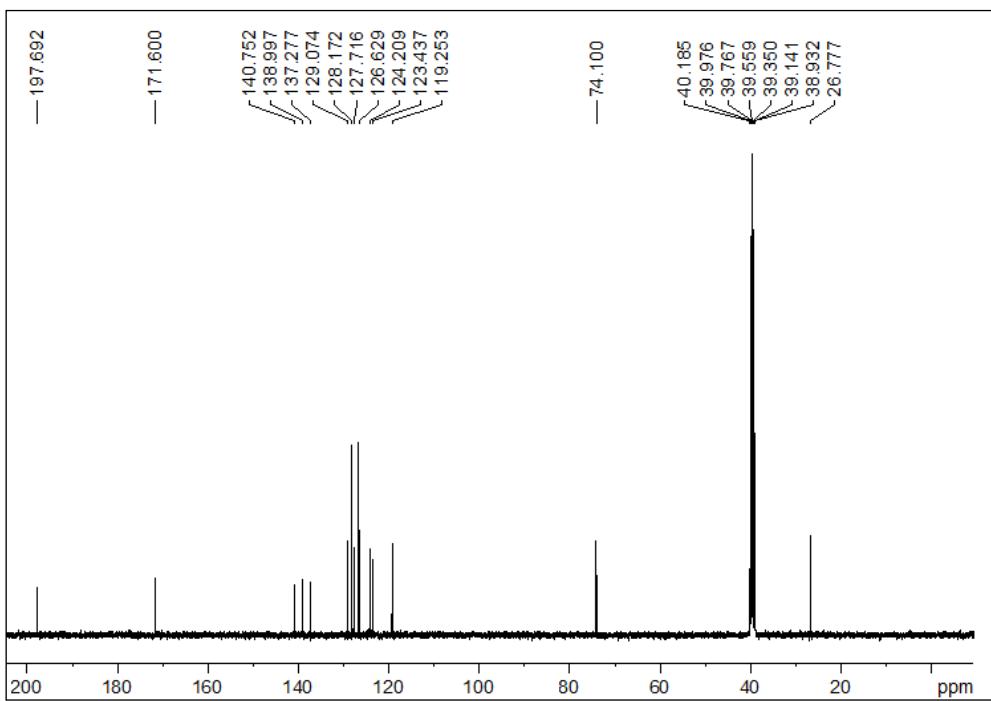


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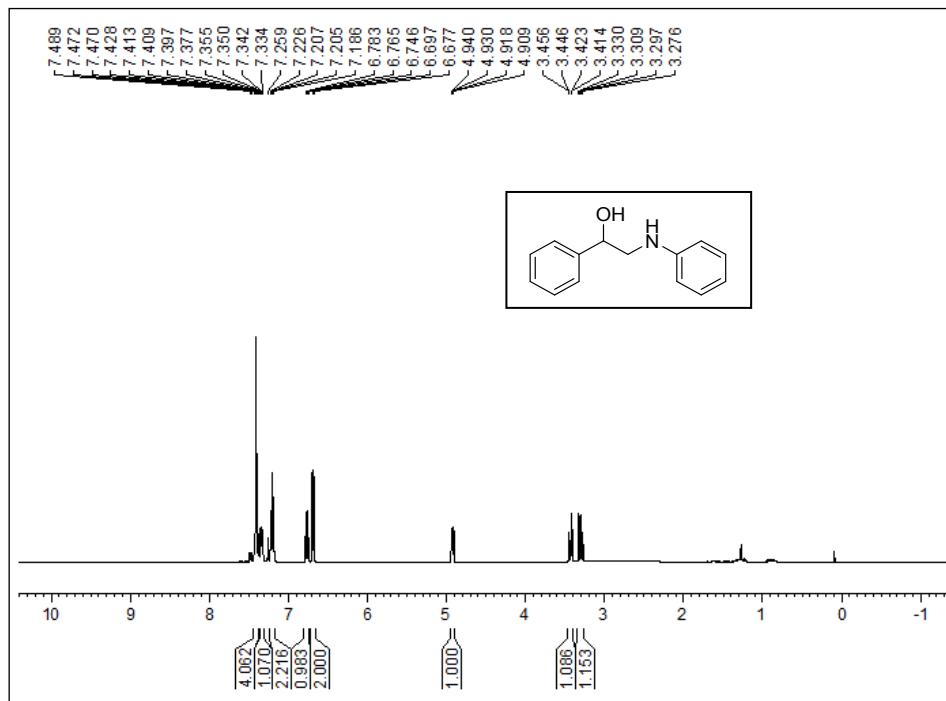




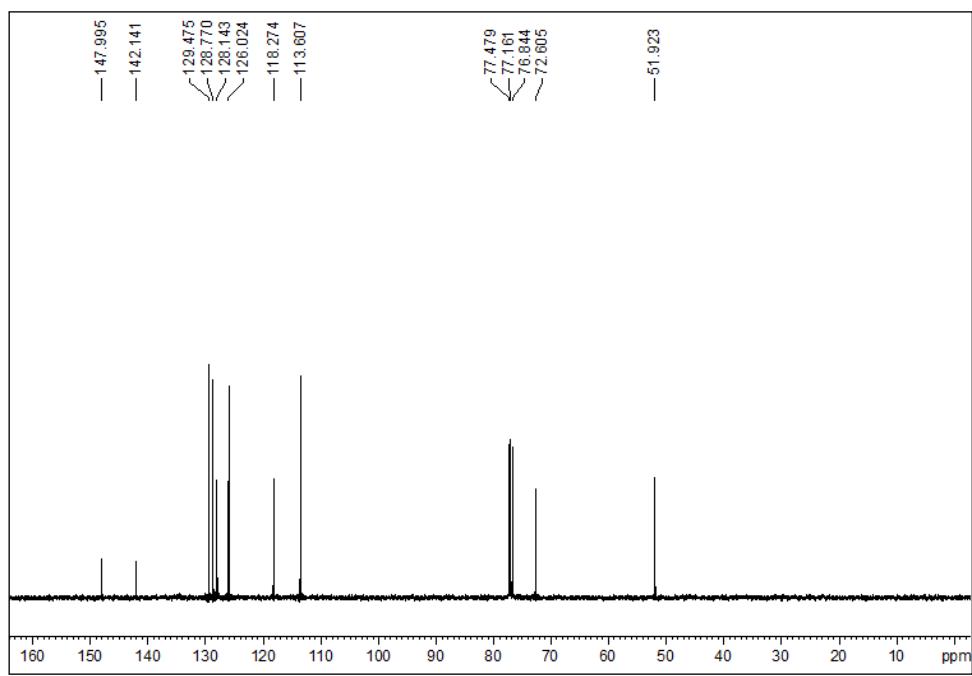
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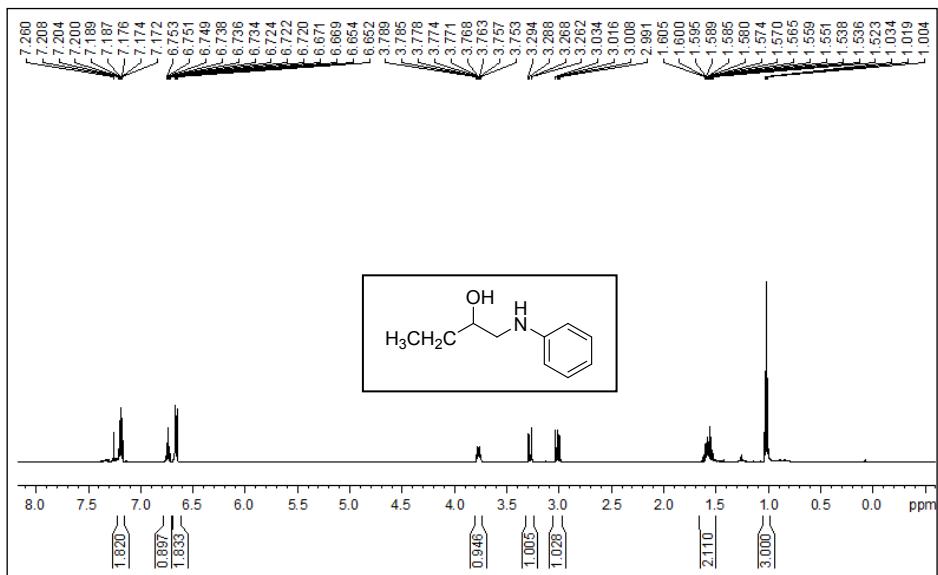
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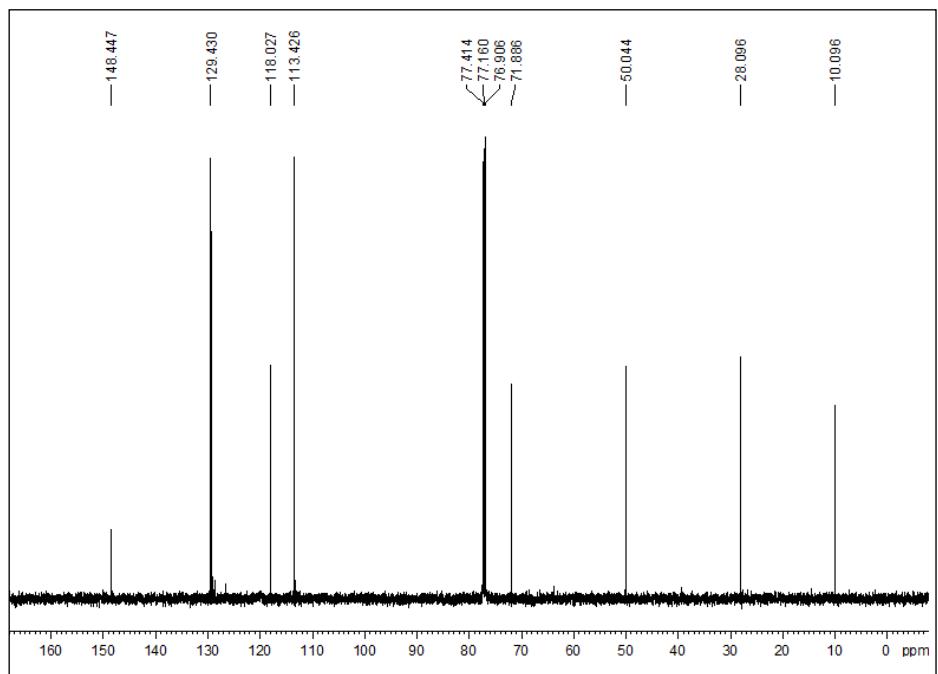
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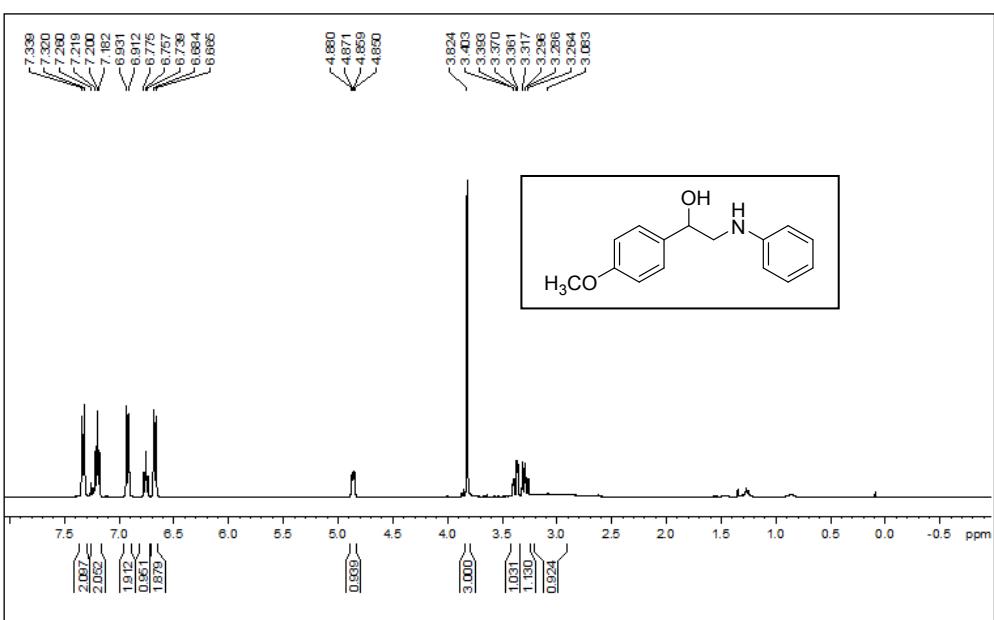
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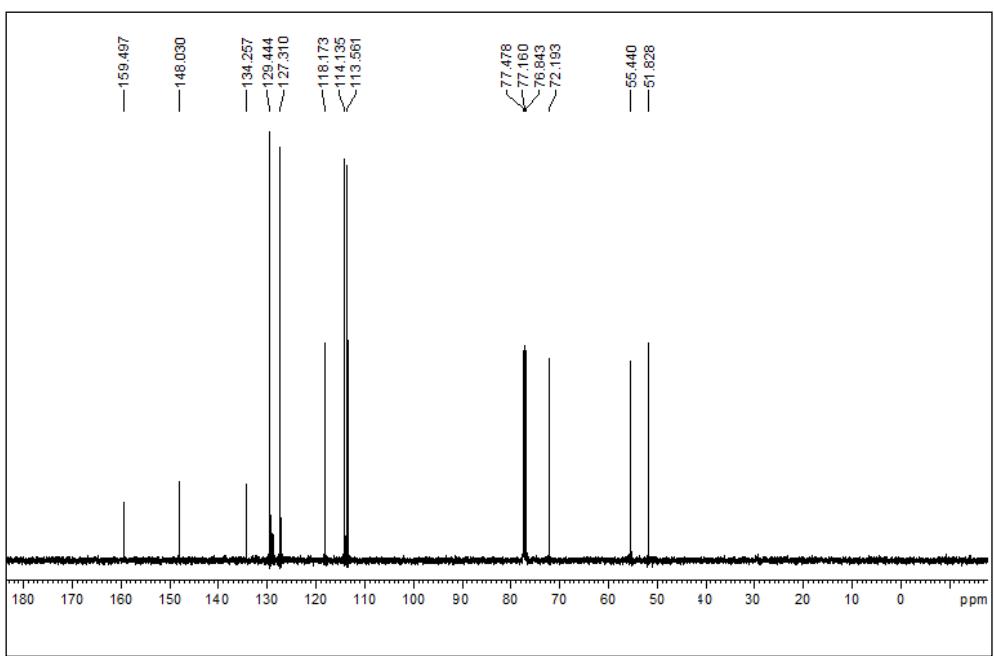
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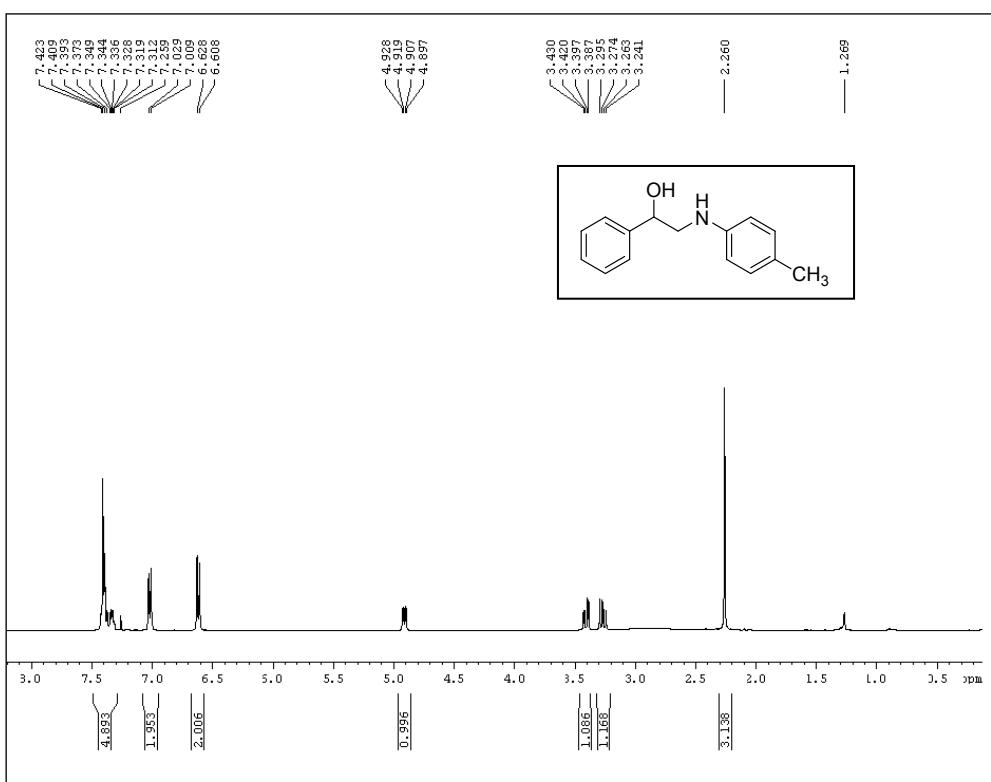
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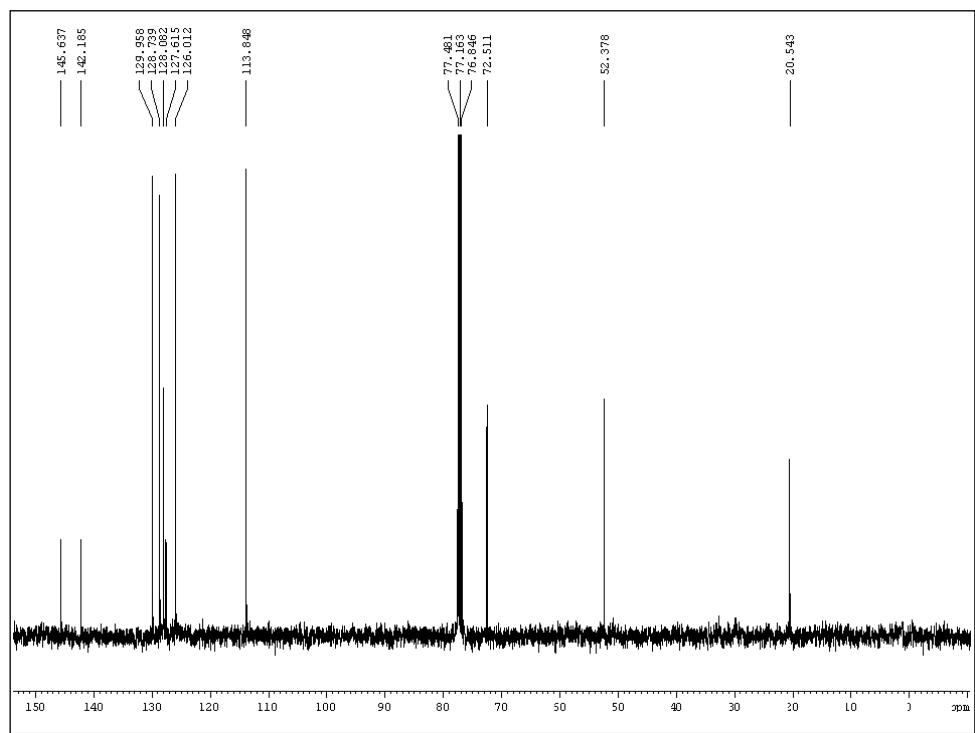
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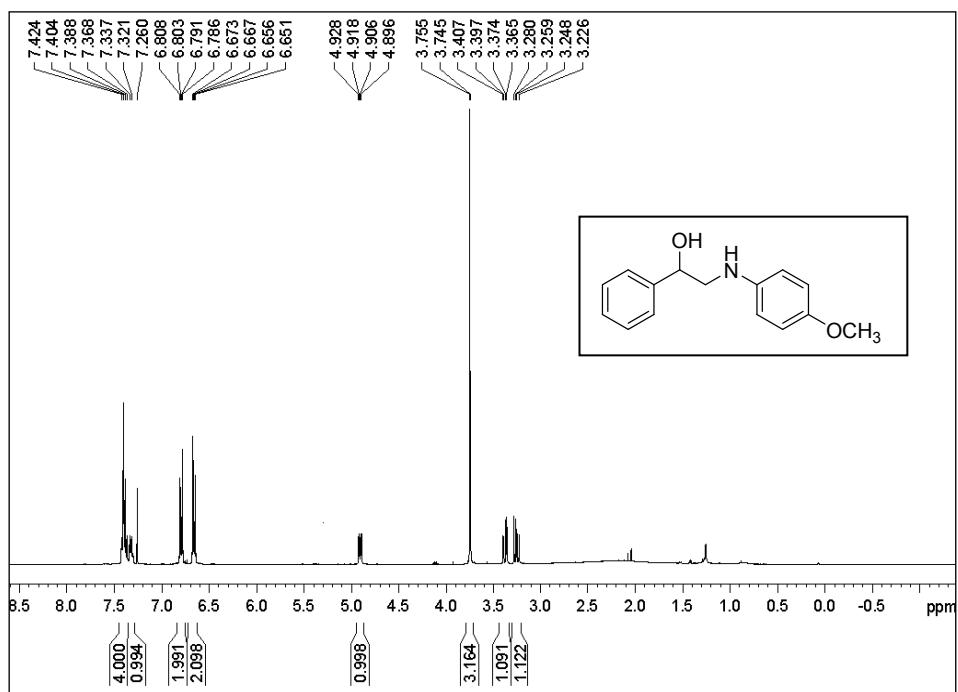
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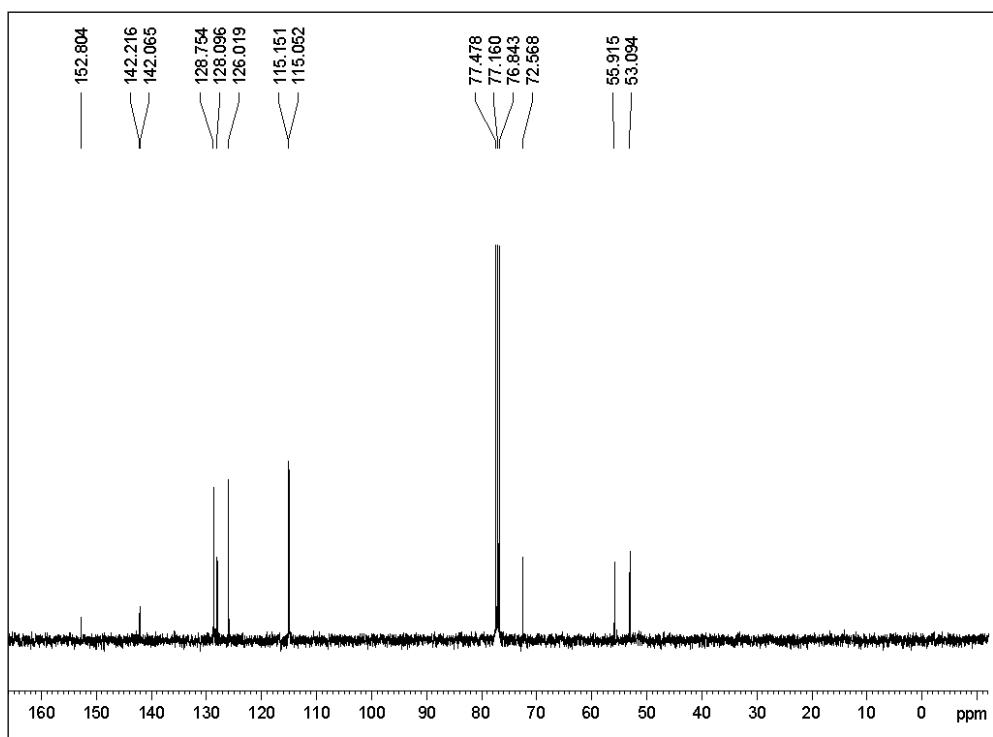
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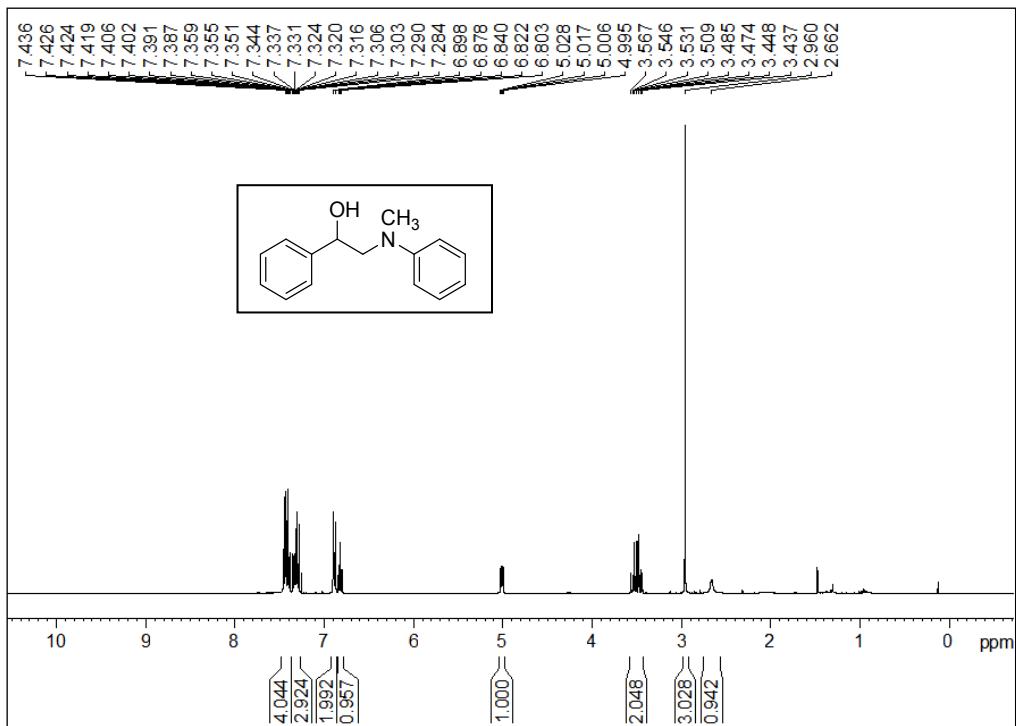
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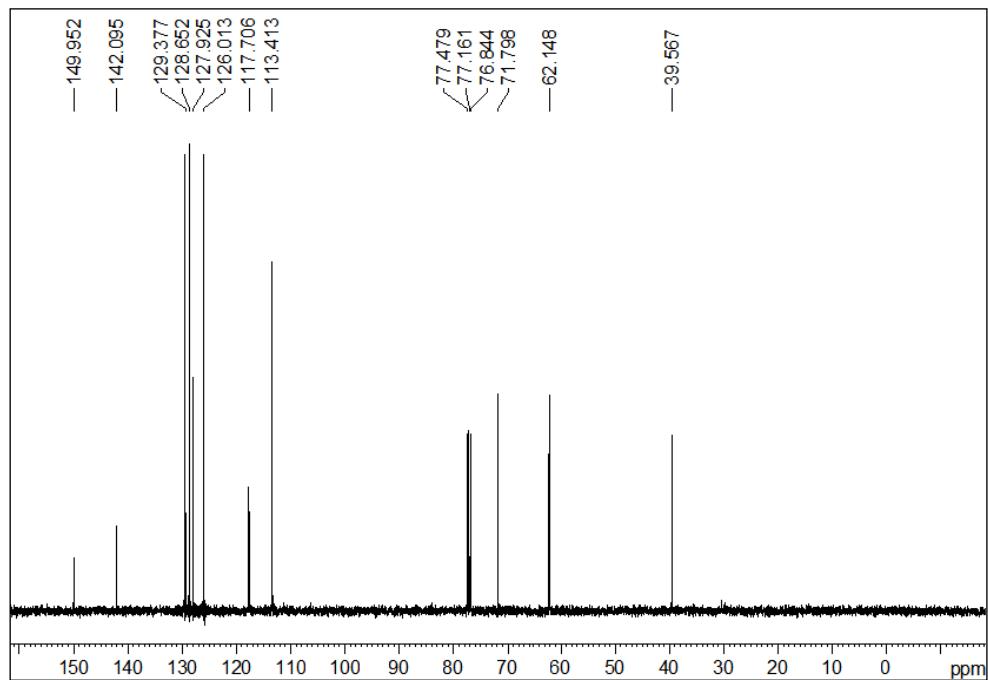
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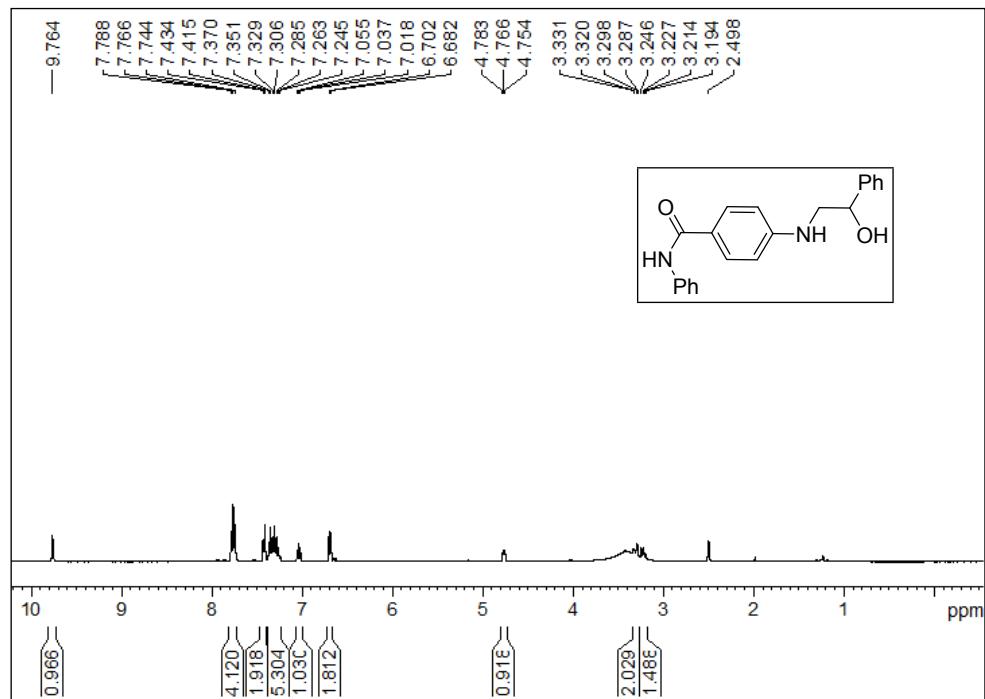
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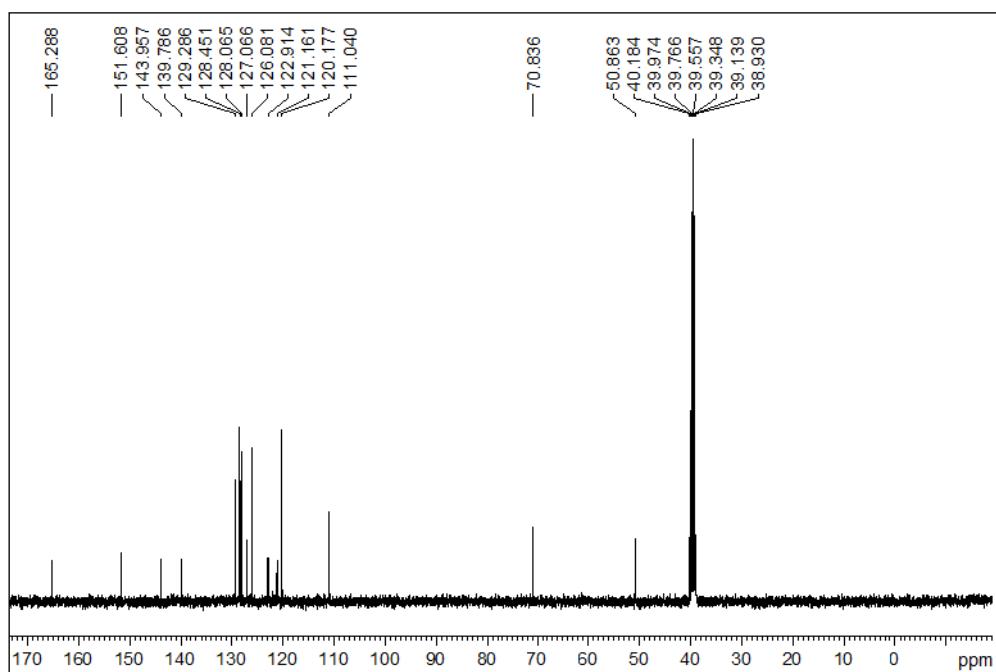
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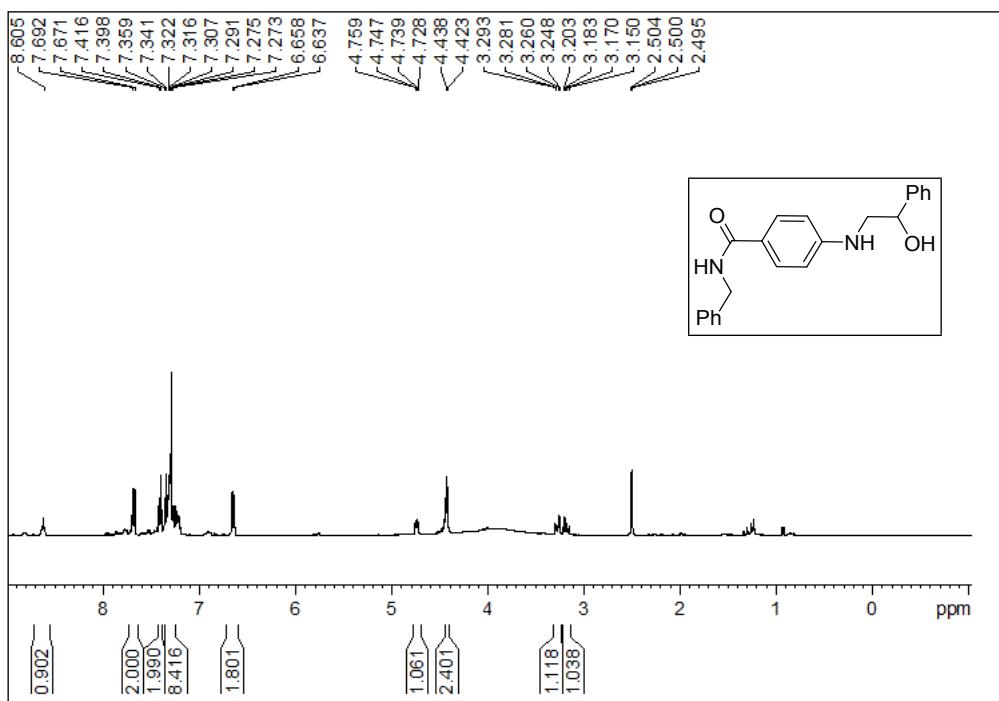
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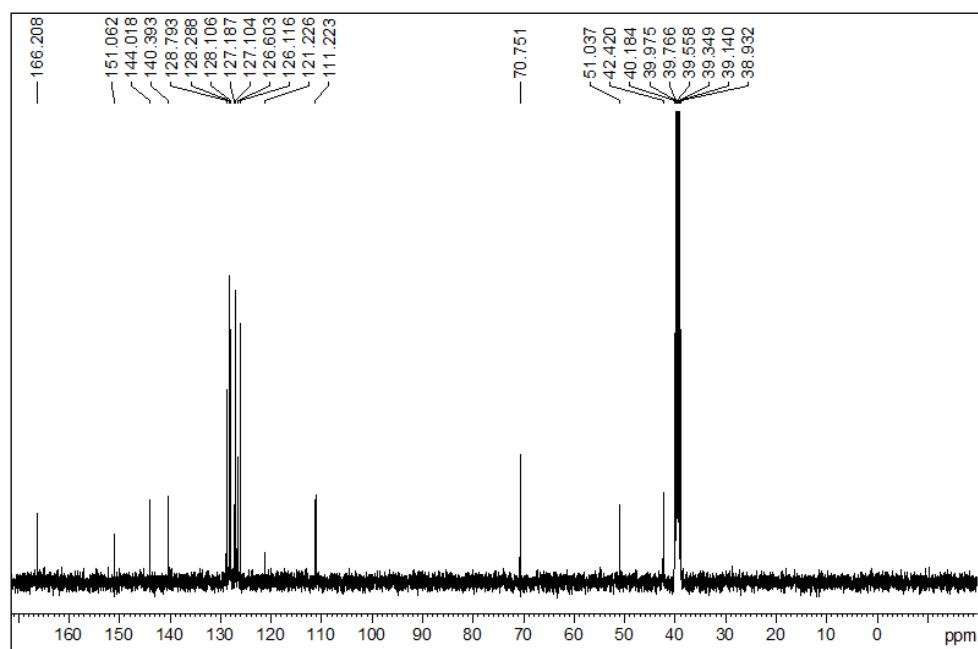
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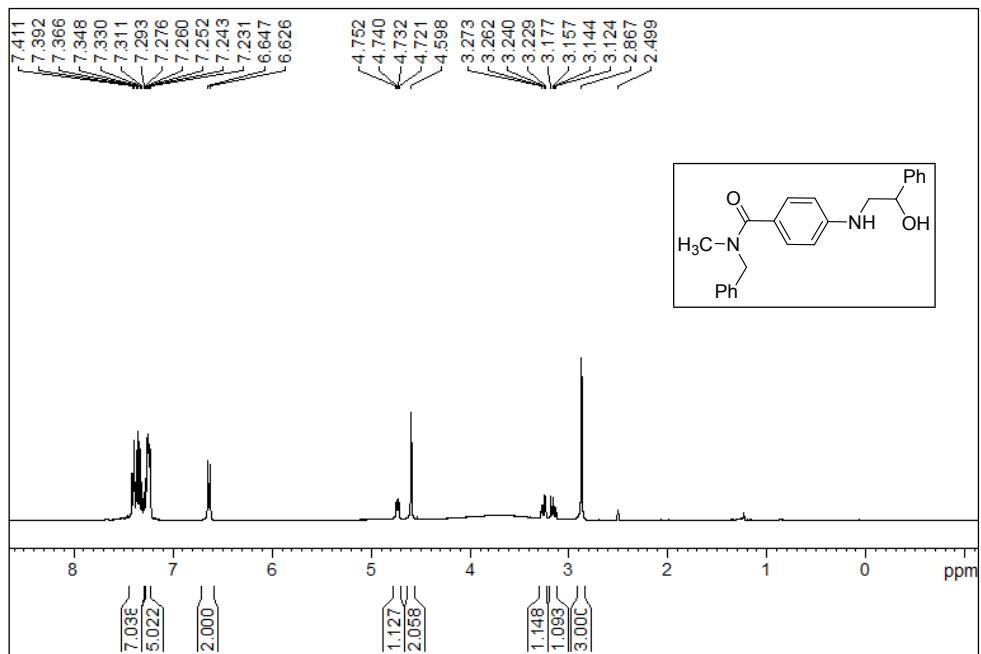
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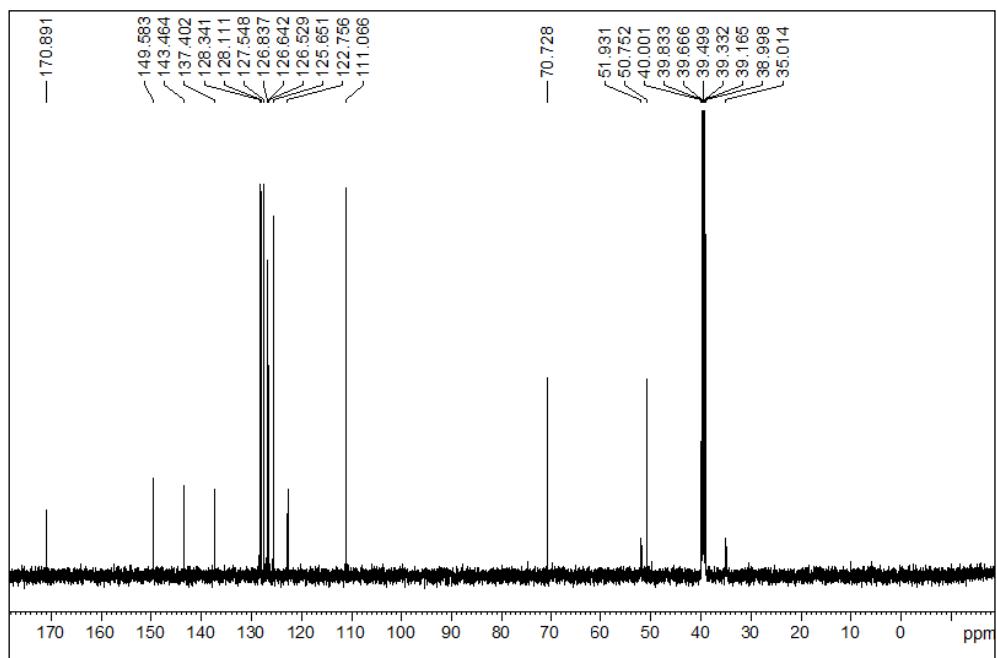
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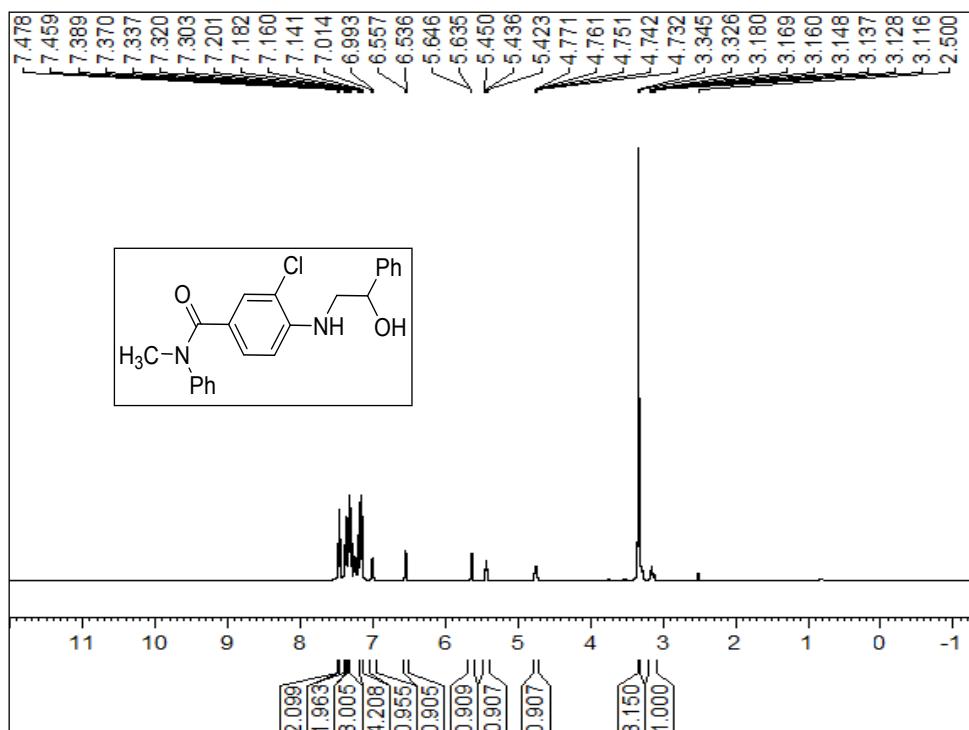
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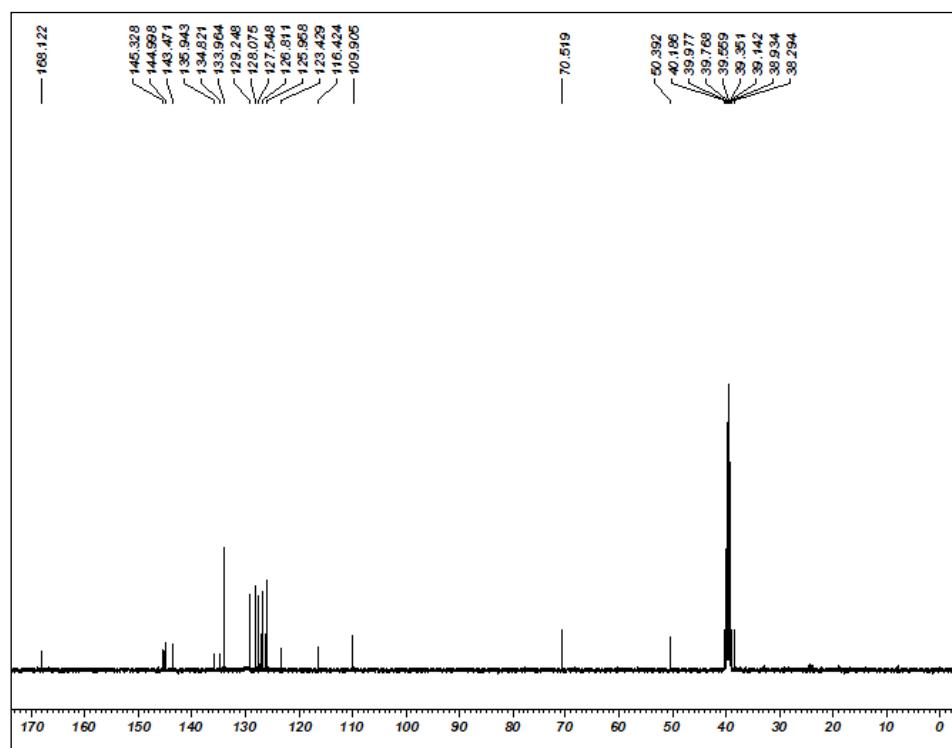
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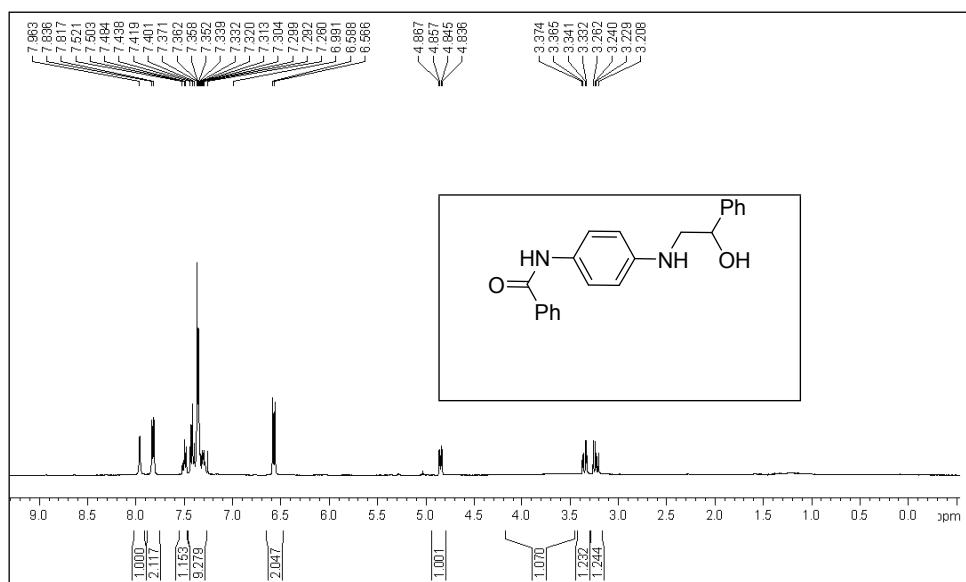
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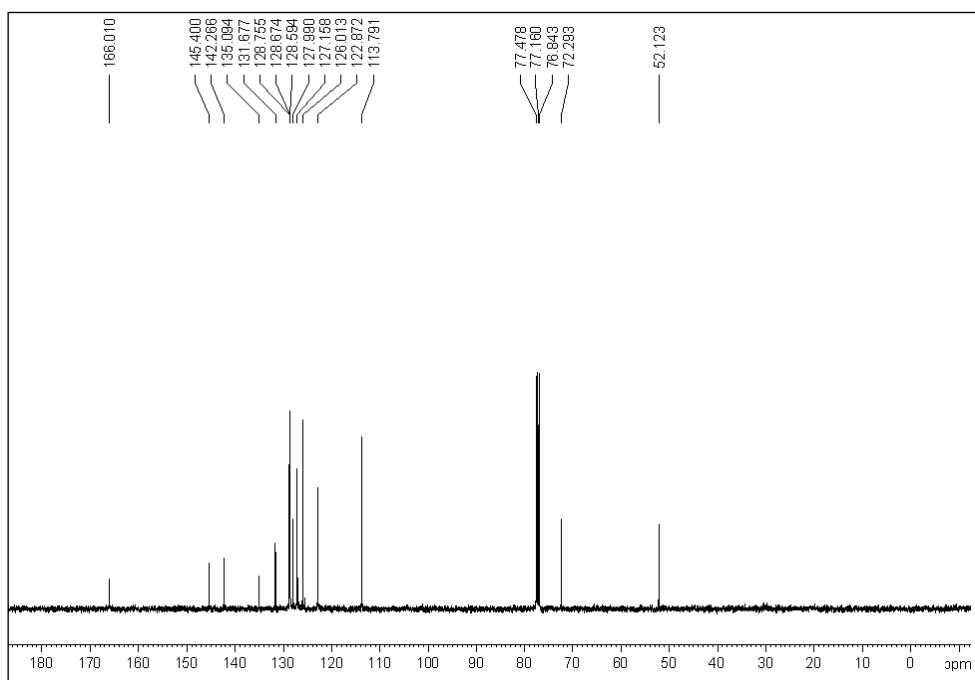
400 MHz ^1H -NMR spectra of **7d** in $\text{DMSO}-d_6$



100 MHz ^{13}C -NMR spectra of **7d** in $\text{DMSO}-d_6$



400 MHz ^1H -NMR spectra of **7e** in CDCl_3



100 MHz ^{13}C -NMR spectra of **7e** in CDCl_3

