Electronic Supplementary Material (ESI) for Catalysis Science & Technology.

Supporting information

Solar and Visible-Light Active Nano Ni/g-C_3N_4 Photocatalyst for Carbon Monoxide (CO) and Ligand-Free Carbonylation Reactions

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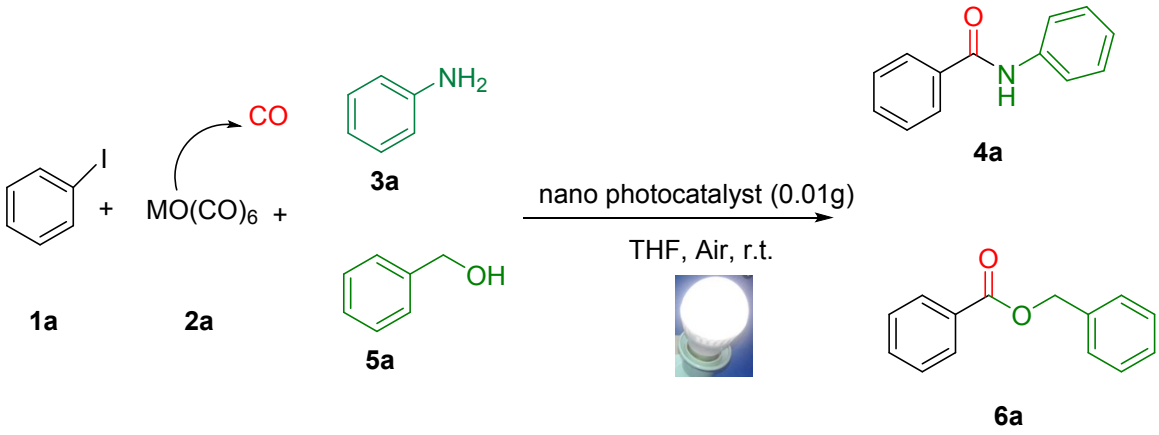
1) Tests for determining the appropriate catalyst for the carbonylation reaction:

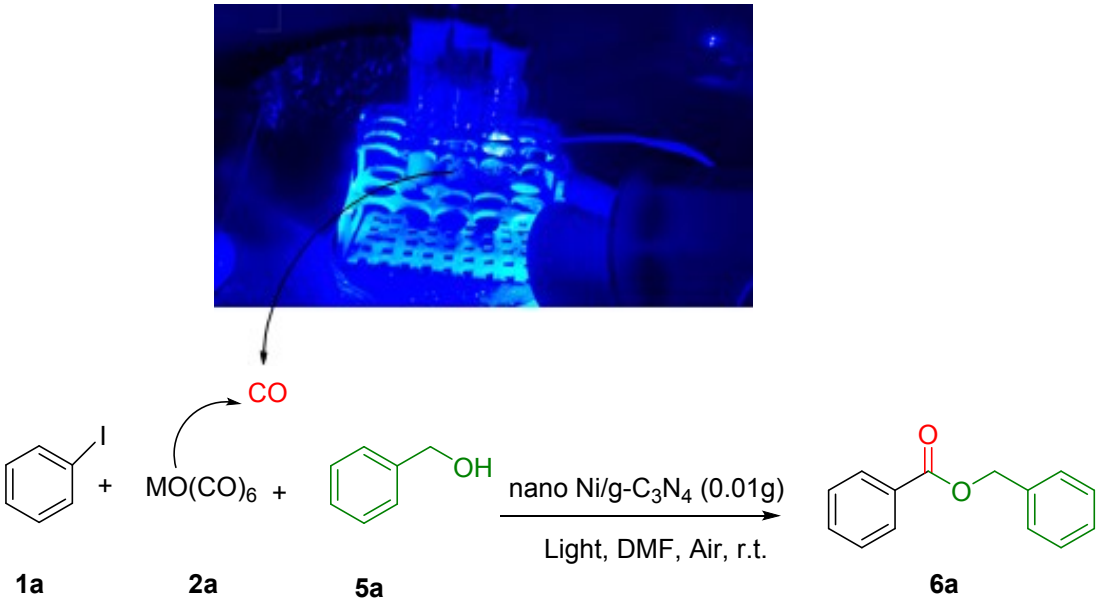
Initially, a model reaction was chosen in attendance of variety photocatalysts in tetrahydrofuran as the solvent and under irradiated-white light for 48 h, at room temperature (Table S1). The key objective of this work was finding suitable support for nickel as photocatalyst but since ZnO and TiO_2 have no absorption in the visible light region, we attempted to use doped Ni on graphitic carbon nitride (band gap: 2.7 eV) to increase light adsorption capacity in the visible region; fortunately, the capability of this model was remarkable. In the following, the activities of other photocatalysts (g-C_3N_4, B-C_3N_4, and also Ni/Sic) and also $NiCl_2$ were also examined, but, the reaction efficiency was notably increased by using them.

2) Optimization

Table S1. The effect of various nano photo-catalyst in the carbonylation reaction.



Entry	Nano photocatalyst	Yield (%)
1	-	0
2	Ni/ZnO	15
3	Ni/TiO_2	15
4	g-C_3N_4	0
5	B-g-C_3N_4	0
6	$NiCl_2$	10
7	Ni/g-C_3N_4	72
8	Ni/Sic	35



Scheme S1: Alkoxycarbinylation

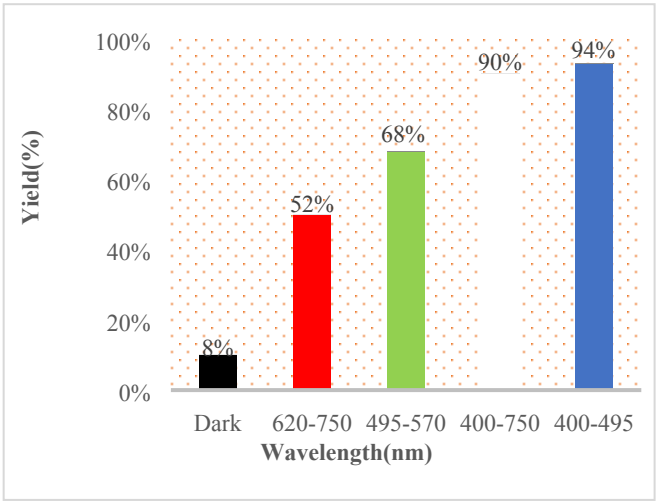


Figure S1. Alkoxycarbonylation under different lights

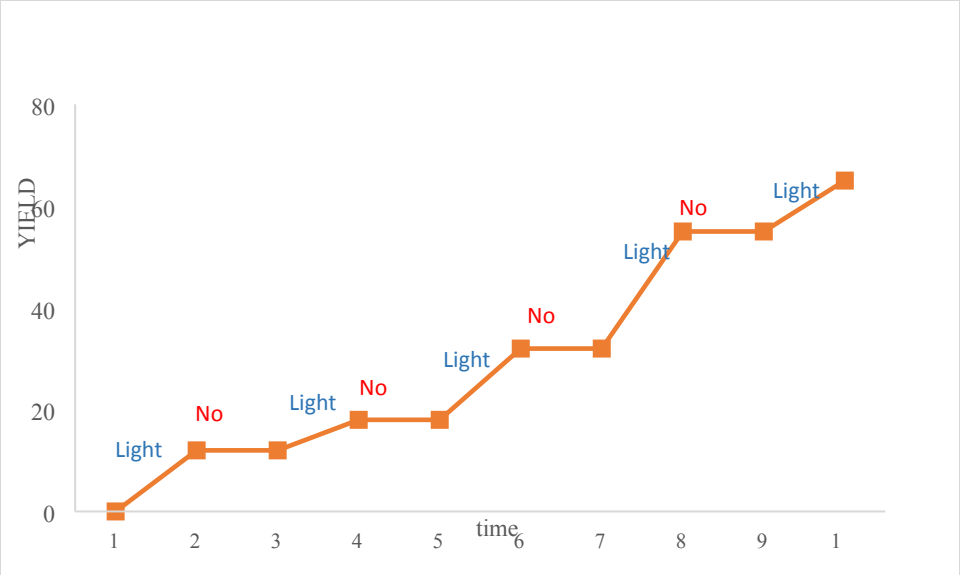


Figure S2. Alcoxycarbonylation reaction under light and no light.

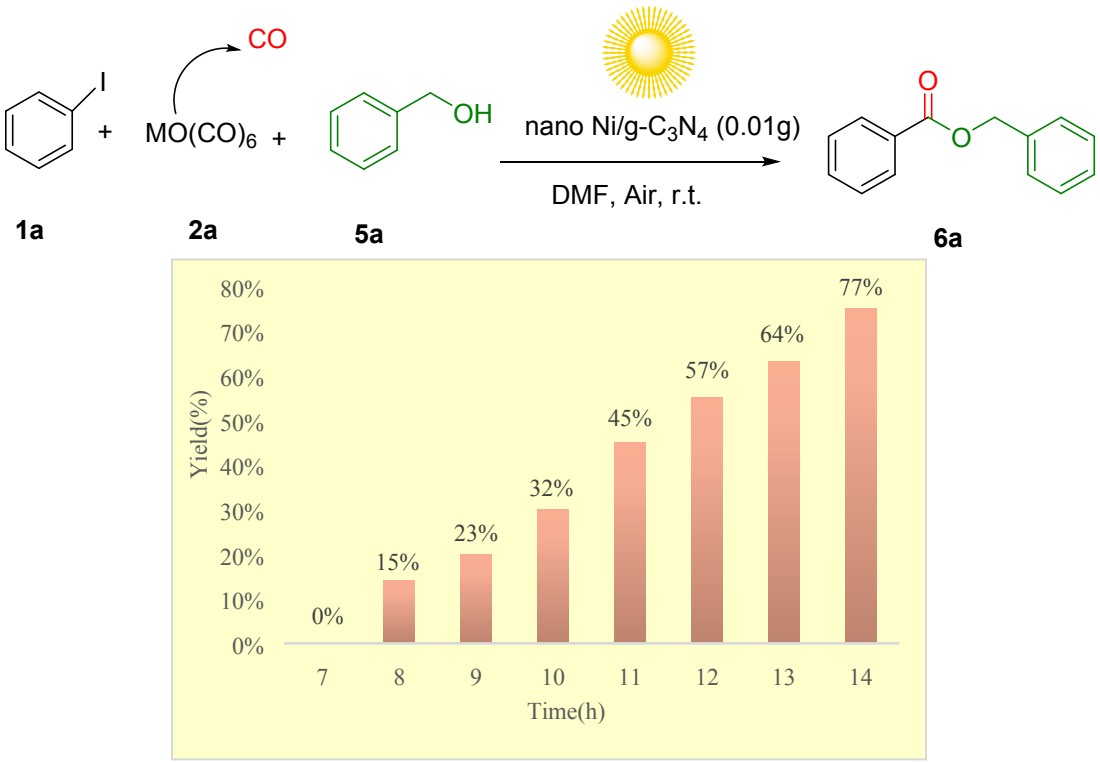


Figure S3. Alkoxycarbonylation under natural sunlight.

Leaching test:

A mixture of iodobenzene (1 mmol), aniline (1 mmol) $Mo(CO)_6$ (1 mmol) nano Ni/g-C_3N_4 (10 mg, 6 wt % Ni), DMF (1.5 mL), was embedded a Pyrex round-bottom flask with a magnetic stir bar and was 3 cm away from the blue LED (11 W), and the reactant mixture was conducted under air condition at normal temperature for 24 h. Then it was centrifuged after complete the reaction for 2 hours to separate the nano Ni/g-C_3N_4 from the solution and the solvent reaction mixture was used to analyze the Ni content using Inductively Coupled Plasma (ICP) spectroscopy, which indicates that no Ni was detected in the filtrate solution. (Table 6 a).

A mixture of iodobenzene (1 mmol), benzyl alcohol (1 mmol), $Mo(CO)_6$ (1 mmol), nano Ni/g-C_3N_4 (10 mg, 6 wt % Ni), DMF (1.5 mL), was embedded a Pyrex round-bottom flask with a magnetic stir bar and was 3 cm away from the blue LED (11 W), and the reactant mixture was conducted under air condition at normal temperature for 24 h. Then it was centrifuged after complete the reaction for 2 hours to separate the nano Ni/g-C_3N_4 from the solution and the solvent reaction mixture was used to analyze the Ni content using Inductively Coupled Plasma (ICP) spectroscopy, which indicates that no Ni was detected in the filtrate solution. (Table 6 b).

3) Experimental information

General Information

Chemicals materials were either made in our laboratories or were acquired from Fluka, Aldrich, Acros, and Merck, and all solvents were obtained from commercial sources and dried employing standard procedures. Crude Products were purified by thin-layer chromatography. Silica gel polygrams SIL G/UV 254 plates were detected process of reactions. Optical characteristics of catalysts were detected by UV/vis spectrophotometer (Shimadzu UV-2450 spectrophotometer). IR spectra were acted on a Shimadzu FT-IR 8300 spectrophotometer and Perkin Elmer spectrum RXI. ^1H and ^{13}C NMR spectra were registered in $CDCl_3$ as solvent using on Bruker, Avance 400 (400 MHz, and 100 MHz respectively) with TMS as an internal standard (multiplicity: s = singlet, d=

doublet, t = triplet, dd = doublet of doublets, m = multiplet). Catalysts were identified by power X-ray diffraction (XRD pattern) on a Bruker D8-advance X-ray diffractometer with Cu Ka (λ=1.54178 A°) incident radiation. The distribution morphology of the catalysis was determined by scanning electron microscope (JEOL, JSM-7610F Fe-SEM) and transmission electron microscopy (JEM-2100F, TEM at 200 kV). Metal contents were investigated by the ICP analyzer (Varian, Vista-pro). The specific surface areas (SSABET; (m2 /g)) of the nano solid were determined with the nitrogen adsorption measurement applying the BET procedure at 77 K (BELsorp-mini II). The fluorescence spectrum of the as-synthesized catalyst was recorded by a spectrofluorometer (Perkin-Elmer model: LS45). The lamps for irradiation were compact fluorescent lamp 11 W white LED (wavelength: 400-750 nm), 11 W blue LED (wavelength: 400-495 nm), 11 W green LED (wavelength: 495-570 nm), 11 W red LED (wavelength: 620-750 nm).

Preparation of the catalysts

Preparation of g-C_3N_4

g-C_3N_4 was prepared according to the procedure reported by Li.[1] Typically, a Coors high-alumina crucible was charged with 10 g of melamine powder, next placed in a 673in³ muffle furnace (115V / 14 Amps) at a heating rate of 20 C /min to 500 C in 2h. In the following for formation better structure, it was put at 520 C for 2 h.

Preparation of nano Ni/g-C_3N_4

The nano Ni/g-C_3N_4 was prepared by altered photodeposition procedure.[2] 10 mg g-C_3N_4, 3 mL triethanolamine (TEOA), 250 μL $NiCl_2$ (0.1 M) solution, 2 mL NaH_2PO_2 (0.1 M) aqueous solution and 4.9 mL H_2O were blended in a round bottom flask at room temperature. The system was stirred for 40 min under nitrogen atmosphere. Then, the mixed solution was irradiated under UV-vis light (300 W Xe lamp) for 30 min. The final point involved washing collected precipitates with distilled water to dispel the residues of reactants.

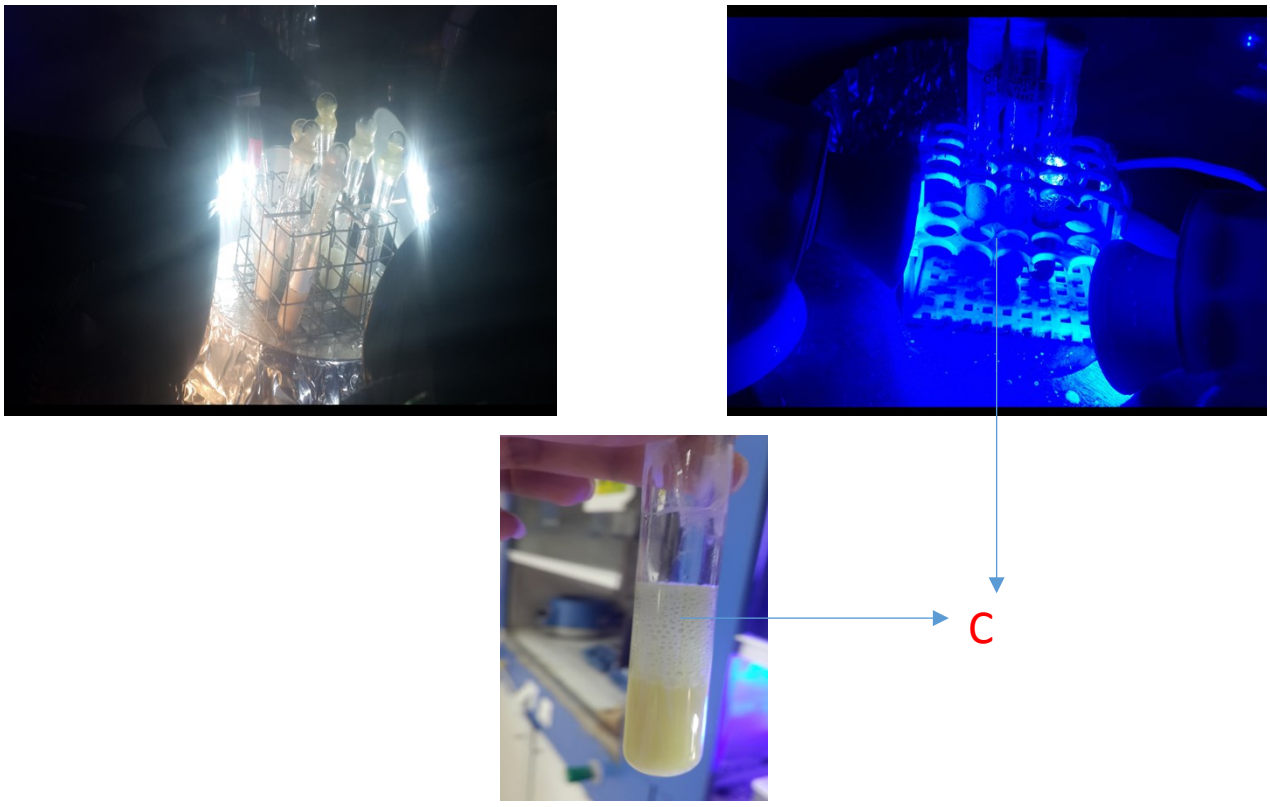
General procedure for aminocarbonylation in the presence of the nano Ni/g-C_3N_4 photocatalyst

To a Pyrex round-bottom flask set with a magnetic stir bar, aryl iodide (1 mmol), amine (1.5 mmol), Mo (CO_6) (1 mmol), nano Ni/g-C_3N_4 (0.01g, 6 wt %) and DMF (1.5 mL) were added and the reactant mixture was irradiated by 11 W blue LED Lamp (λ > 440 nm, distance 3.0 cm) in an

air atmosphere at room temperature. The reaction was stirred for a suitable time at room temperature (25 °C) and checked using TLC. Later the termination of the reaction, ethyl acetate (2 ml) was added to the reaction mixture and heterogeneous nano Ni/g-C_3N_4 were entirely isolated applying centrifugation, then, the organic phases were dehydrated over $MgSO_4$ and the solvent was vaporized under reduced pressure to acquire the crude. Ultimate product purification was done by silica gel column chromatography utilizing petroleum ether/ethyl acetate.

General procedure for alkoxycarbonylation in the presence of the nano Ni/g-C_3N_4 photocatalyst

To a Pyrex round-bottom flask set with a magnetic stir bar, aryl iodide (1 mmol), alcohol (1.5 mmol), Mo (CO_6) (1 mmol), nano Ni/g-C_3N_4 (0.01g, 6 wt %) and DMF (1.5 mL) were added and the reactant mixture was irradiated by 11 W blue LED Lamp ($\lambda > 440$ nm, distance 3.0 cm) in air condition at normal temperature. The reaction was stirred for a suitable time at room temperature (25 °C) and checked using TLC. Later the termination of the reaction, ethyl acetate (2 ml) was added to the reaction mixture and heterogeneous nano Ni/g-C_3N_4 were entirely isolated applying centrifugation, then, the organic phases were dried dehydrated over $MgSO_4$ and the solvent was vaporized under reduced pressure to acquire the crude. Ultimate product purification was done by silica gel column chromatography utilizing petroleum ether/ethyl acetate.



4) Characterization

Photocatalyst characterization

The characterization of the as-prepared photocatalyst was performed applying, UV-Vis absorption, EDX, ICP, XRD, SEM, TEM, and BET. The summary of results from the catalyst characterization is demonstrated in Table S2.

Table S2. Characterization data for Ni/g-C_3N_4 nanoparticles

XRD	Crystallite sizes of nano Ni/g-C_3N_4 is 4.6 nm
TEM	The diameter of catalyst around 5-9 nm
ICP	6 (wt %) of Ni
Pore size distribution	7.98 nm
BET surface area	12.884 $m^2.g^{-1}$

Crystallite sizes of nano Ni/g-C_3N_4 was determined by Scherrer's formula [3].

$$D = \frac{K\,\lambda}{B\,cos\Theta}$$

The K factor in Scherrer's formula = 0.9 or close to unity

λ is the wavelength = 1.54 Å= 0.154 nm

$$B = \frac{FWHM * 3.14}{180}$$

FWHM is the full width of the peak at half the maximum intensity after subtraction of instrumental background. B= 0.0338

Θ = diffraction angle, Cos Θ = 0.99

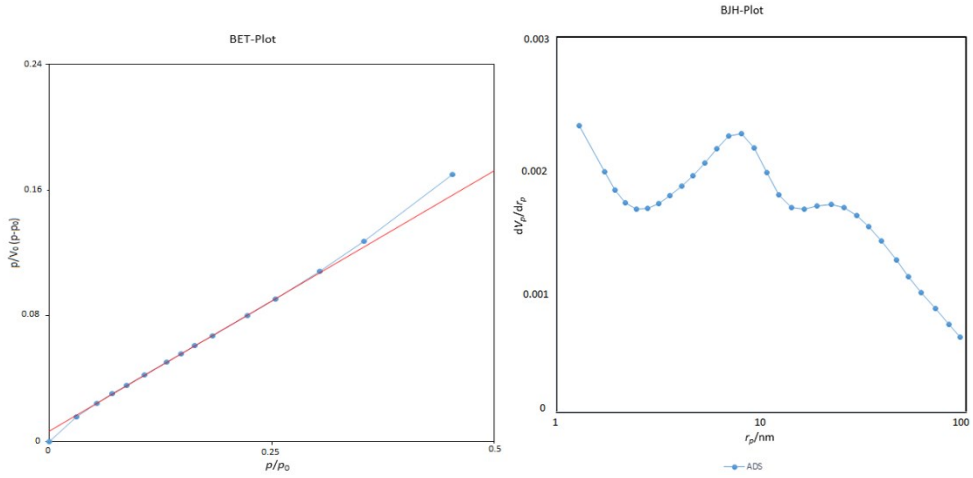
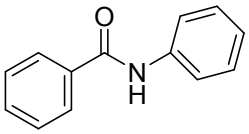
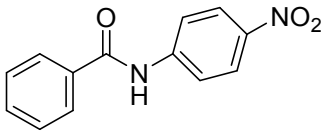


Figure S4. a) BET-Plot; b) BJH-Plot of nano Ni/g-C_3N_4

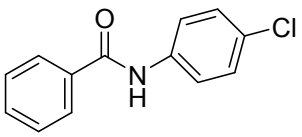
Characterization of compounds



N-phenylbenzamide (4a), 94% yield; white soilid; TLC (Petroleumether:Ethyl acetate, 85:15 v/v) IR (thin film) 3345, 3055, 3027, 1657, 1625, 1538, 1440, 1301, 1179,1002, 885cm^{-1}; ^{1}H NMR (400 MHz, CDCl3) δ (ppm): 7.96 (2H, d, J = 8.4 Hz), 7.87 (2H, d, J = 7.7 Hz), 7.77 (1H, s), 7.55 (1H, t, J = 7.3 Hz), 7.48 (2H, t, J = 8.2 Hz), 7.20 (2H, t. J=7.3), 7.04 (1H, t. J=7.3). ^{13}C NMR (100 MHz, CDCl3) δ (ppm): 166.0, 139.2, 132.1, 130.4, 128.9, 127.2, 124.8, 120.7, 118.0. Anal. Calcd for $C_{13}H_{11}NO$: C, 79.17; H, 5.62; N, 7.10. Found: C, 79.28; H, 5.67; N, 7.19.

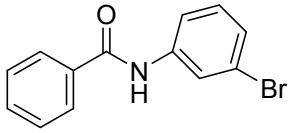


N-(4-nitrophenyl) benzamide (4b), 96% yield; light yellow solid; TLC (Petroleumether:Ethyl acetate, 85:20 v/v) IR (thin film) 3340, 3052, 3023, 1655, 1620, 1548, 1537, 1438, 1347, 1300, 1176,1000, 883cm^{-1}; ^{1}H NMR (400 MHz, CDCl3) δ (ppm): 8.21 (1H, s), 8.03 (2H, d, J = 7.1 Hz), 7.84 (2H, d, J = 8.9 Hz), 7.57 (2H, d, J = 7.3 Hz), 7.17-7.48 (3H, m).^{13}C NMR (100 MHz, CDCl3) δ (ppm): 165.3, 146.2, 134.0, 133.3, 129.8, 128.9, 128.6, 125.2, 119.6. Anal. Calcd for $C_{13}H_{10}N_2O_3$: C, 64.46; H, 4.16; N, 11.56. Found: C, 64.40; H, 4.25; N, 11.51.

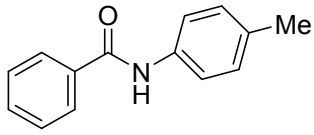


N-(4-chlorophenyl) benzamide (4c), 95% yield; beige solid; TLC (Petroleumether:Ethyl acetate, 85:17 v/v) IR (thin film) 3344, 3054, 3026, 1656, 1622, 1537, 1440, 1302, 1178,1005, 885, 795cm^{-1}; ^{1}H NMR (400 MHz, CDCl3) δ (ppm): 7.86 (2H, d, J = 7.1 Hz), 7.75 (2H, d, J = 8.9 Hz), 7.60

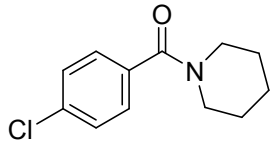
(1H, t, J = 7.3 Hz), 7.38 (2H, t, J = 7.3 Hz), 7.24 (2H, d, J = 8.9 Hz), 7.15 (1H, s). ^{13}C NMR (100 MHz, CDCl3) δ (ppm): 166.8, 132.0, 131.6, 129.2, 128.7, 128.4, 128.1, 126.5, 120.9. Anal. Calcd for $C_{13}H_{10}ClNO$: C, 67.40; H, 4.35; N, 6.05; Found: C, 67.46; H, 4.27; N, 6.00.



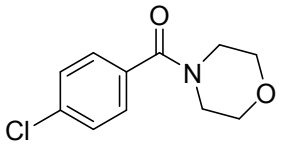
N-(3-bromophenyl) benzamide (4d), 90% yield; beige solid; TLC (Petroleumether:Ethyl acetate, 85:18 v/v) IR (thin film) 3341, 3050, 3026, 1653, 1624, 1539, 1441, 1303, 1179,1003, 881, 670cm^{-1}; ^{1}H NMR (400 MHz, CDCl3) δ (ppm): 8.20 (s,1H), 7.89 (s, 1H), 7.81 (2H, d, J = 6.8 Hz), 7.53 (1H, t, J = 7.1 Hz), 7.34 (2H, t, J = 7.3 Hz), 7.24 (2H, d, J = 7.3 Hz), 7.18 (1H, t, J = 7.1 Hz). ^{13}C NMR (100 MHz, CDCl3) δ (ppm): 166.3, 139.4, 134.6, 132.2, 130.4, 128.9, 127.6, 123.5, 122.7, 119.0. Anal. Calcd for $C_{13}H_{10}BrNO$: C, 56.55; H, 3.65; N, 5.07; O, 5.79; Found: C, 56.46; H, 3.61; N, 5.18.



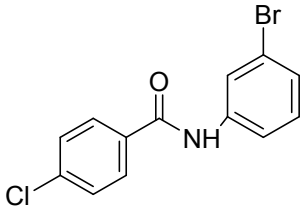
N-(4-methylphenyl) benzamide (4e), 87% yield; white solid; TLC (Petroleumether:Ethyl acetate, 85:15 v/v) IR (thin film) 3341, 3050, 3026, 2920, 1653, 1624, 1539,1449, 1441, 1372,1303, 1179,1003, 881. cm^{-1}; ^{1}H NMR (400 MHz, CDCl3) δ (ppm): 8.26 (s,1H), 7.87 (2H, d, J = 7.2 Hz), 7.55 (2H, d, J = 8.2 Hz), 7.51 (1H, t, J = 7.2 Hz), 7.43 (2H, t, J = 7.2 Hz), 7.16 (2H, d, J = 8.2 Hz), 2.33 (s, 3H).^{13}C NMR (100 MHz, CDCl3) δ (ppm): 165.4, 135.3, 134.9, 133.6, 131.1, 129.0, 127.7, 126.4, 120.2. Anal. Calcd for $C_{14}H_{13}NO$: C, 79.59; H, 6.20; N, 6.63. Found: C, 79.67; H, 6.29; N, 6.55.



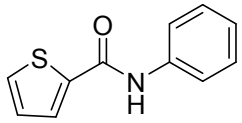
(4-Chlorophenyl)(piperidin-1-yl)methanone(4l), 90% yield; Colorless oil; TLC (Petroleumether:Ethyl acetate, 85:17 v/v) IR (thin film), 3055, 3022, 2922, 1653, 1616, 1539, 1424,1303, 1179,1003, 885, 656cm^{-1}; ^{1}H NMR (400 MHz, CDCl3) δ (ppm): 7.38 (2H, d, J = 8.2 Hz), 7.35 (2H, d, J = 8.2 Hz), 3.34-3.70 (4H, m), 1.40-1.86 (6H, m). ^{13}C NMR (100 MHz, CDCl3) δ (ppm): 169.2, 135.6, 134.8 , 129.2, 128.3, 42.9, 26.2, 25.2. Anal. Calcd for $C_{11}H_{13}NO$: C, 75.40; H, 7.48; N, 7.99. Found: C, 75.33; H, 7.41; N, 7.87.



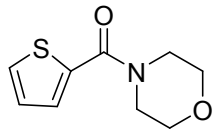
(4-Chlorophenyl)(morpholino)methanone (4m), 92% yield; yellow oil; TLC (Petroleumether:Ethyl acetate, 85:18 v/v) IR (thin film), 3053, 3024, 2921, 1652, 1618, 1537, 1425,1293, 1175,1000, 88o, 650.cm^{-1}; ^{1}H NMR (400 MHz, CDCl3) δ (ppm): 7.40 (2H, d, J = 8.1 Hz), 7.36 (2H, d, J = 8.1 Hz), 3.45-3.73 (8H, m).^{13}C NMR (100 MHz, CDCl3) δ (ppm): 169.8, 136.2, 133.8, 128.8, 128.6, 66.5, 47.9.Anal. Calcd for $C_{11}H_{13}NO$: C, 75.40; H, 7.48; N, 7.99. Found: C, 75.33; H, 7.41; N, 7.87.



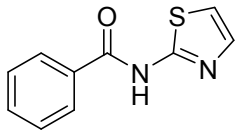
N-(3-bromophenyl)-4-chlorobenzamide (4n), 92% yield; light yellow solid; TLC (Petroleumether:Ethyl acetate, 85:18 v/v) IR (thin film) 3387, 3286, 3047, 1660, 1620, 1534, 1448, 1300, 1175,1001, 883, 795, 680cm^{-1}. ^{1}H NMR (400 MHz, CDCl3) δ (ppm): 8.45 (s, 1H), 8.01 (2H, d, J = 7.8 Hz), 7.83 (1H, s), 7.72 (2H, d, J = 7.8 Hz), 7.32 (1H, d, J = 7.6 Hz), 7.23 (1H, t, J = 7.7 Hz), 7.11 (1H, d, J = 7.6 Hz). ^{13}C NMR (100 MHz, CDCl3) δ (ppm): 165.3 139.1, 132.0, 130.4, 129.5, 129.1, 128.7, 127.9, 123.7, 122.7, 119.3.Anal. Calcd for $C_{13}H_{9}BrClNO$: C, 50.28; H, 2.92; N, 4.51. Found: C, 50.33; H, 2.99; N, 4.46.



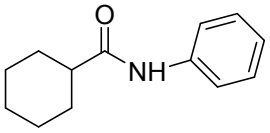
N-phenylthiophene-2-carboxamide (4o), 95% yield; light yellow solid; TLC (Petroleumether:Ethyl acetate, 85:17 v/v) IR (thin film) 3410, 3286, 3047, 1660, 1622, 1534, 1430, 1322, 1210,1011,795cm^{-1}. ^{1}H NMR (400 MHz, CDCl3) δ (ppm): 7.70 (s,1H), 7.68 (1H, d, J = 7.7 Hz), 7.59 (1H, d, J = 7.7 Hz), 7.52 (2H, d, J = 7.6 Hz), 7.35 (2H, t, J = 7.5 Hz), 7.16 (1H, t, J = 7.5 Hz), 7.08 (1H, t, J = 7.6 Hz).^{13}C NMR (100 MHz, CDCl3) δ (ppm): 166.2, 137.7, 133.9, 130.8, 129.0, 128.6, 127.8, 124.6, 120.4. Anal. Calcd for $C_{11}H_9NOS$: C, 65.00; H, 4.46; N, 6.89. Found: C, 71.00; H, 4.37; N, 6.79.



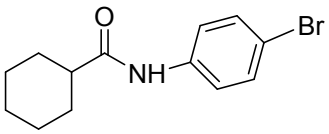
Morpholino (thiophen-2-yl) methanone (4p), 89% yield; colorless oil; TLC (Petroleumether:Ethyl acetate, 85:19 v/v) IR (thin film) 3106, 3070, 2940, 2830, 1620, 1395, 1346, 1251,1081, 1034, 871, 714. cm^{-1}. ^{1}H NMR (400 MHz, CDCl3) δ (ppm): 7.39 (d, 1H, J= 8 Hz), 7.19 (t, 1H, J= 8 Hz), 6.93 (d, 1H, J= 8 Hz), 3.71-3.87 (4H, m), 3.54-3.69 (4H, m). ^{13}C NMR (100 MHz, CDCl3) δ (ppm): 163.6, 136.7, 129.0, 128.9, 126.9, 67.0, 46.1. Anal. Calcd for $C_9H_{11}NO_2S$: C, 54.80; H, 5.62; N, 7.10. Found: C, 54.71; H, 5.55; N, 7.15.



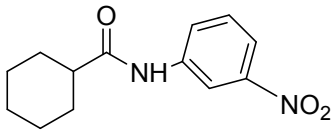
N-(thiazol-2-yl)benzamide (4q), 89% yield; yellowish oil; TLC (Petroleumether:Ethyl acetate, 85:20 v/v) IR (thin film) 3118, 3064, 2952, 2830, 1619,1481, 1361, 1319, 1241,1222, 1074, 882. cm^{-1}. ^{1}H NMR (400 MHz, CDCl3) δ (ppm): 8.15 (d, 2H, J= 7.5 Hz), 7.65 (t, 1H, J= 7.7 Hz), 7.52 (t, 2H, J= 7.7 Hz), 7.28 (d, 1H, J= 7.8 Hz), 7.03 (d, 1H, J= 7.8 Hz), 6.90 (s, 1H). ^{13}C NMR (100 MHz, CDCl3) δ (ppm): 171.3, 165.3, 160.5, 135.9, 131.9, 128.2, 127.9, 112.7. Anal. Calcd for $C_{10}H_8N_2OS$: C, 58.81; H, 3.95; N, 13.72. Found: C, 58.89; H, 3.88; N, 13.81.



N-phenylcyclohexanecarboxamide (4r), 80% yield; white solid; TLC (Petroleumether:Ethyl acetate, 85:15 v/v) IR (thin film) 3110, 3064, 2926, 2830, 2553, 2690, 1960, 1817, 1660,1450, 1257, 1241,1039, 1015, 852. cm^{-1}. ^{1}H NMR (400 MHz, CDCl3) δ (ppm): 7.61 (1H, s), 7.58 (d, 2H, J= 7.9 Hz), 7.32 (t, 2H, J= 8.12 Hz), 7.11 (t, 1H, J= 7.4 Hz), 2.25-2.30 (1H, m), 1.83 – 1.98 (2H, m), 1.52-1.78 (2H, m), 1.20-1.33 (6H, m). ^{13}C NMR (100 MHz, CDCl3) δ (ppm): 174.8, 138.4, 128.7, 124.0, 120.3, 46.7, 29.9, 26.1, 24.8. Anal. Calcd for $C_{13}H_{17}NO$: C, 76.81; H, 8.43; N, 6.89. Found: C, 76.74; H, 8.36; N, 6.80.

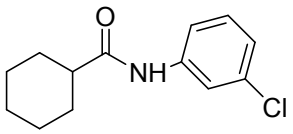


N-(4-bromophenyl)cyclohexanecarboxamide (4s), 84% yield; colorless oil; TLC (Petroleumether:Ethyl acetate, 85:17 v/v) IR (thin film) 3112, 3061, 2830, 2550, 2692, 1958, 1819, 1662,1448, 1256, 1244,1037, 1018, 852, 680 cm^{-1}. ^{1}H NMR (400 MHz, CDCl3) δ (ppm): 7.26-7.45 (4H, m), 7.20 (1H, s), 2.17-2.35 (1H, m), 1.86 – 2.08 (2H, m), 1.58-1.80 (2H, m), 1.29-1.50 (6H, m). ^{13}C NMR (100 MHz, CDCl3) δ (ppm): 174.4, 137.1, 131.5, 121.7, 116.6, 46.2, 29.4, 26.1, 25.7. Anal. Calcd for $C_{13}H_{16}BrNO$: C, 55.33; H, 5.72; N, 4.96. Found: C, 55.40; H, 5.65; N, 4.89.

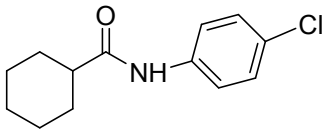


N-(3-nitrophenyl)cyclohexanecarboxamide (4t), 84% yield; yellow solid; TLC (Petroleumether:Ethyl acetate, 85:18 v/v) IR (thin film) 3110, 3059, 2830, 2552, 2694, 1956, 1817, 1660, 1552, 1448, 1352, 1255, 1240,1035, 1016, 855 cm^{-1}. ^{1}H NMR (400 MHz, CDCl3) δ (ppm):

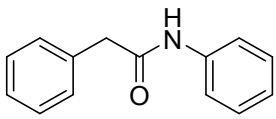
8.45 (s, 1H), 7.98 (t, 1H, J= 7.9 Hz), 7.94 (s, 1H), 7.87 (d, 1H, J= 7.7 Hz), 7.49 (t, 1H, J= 7.9 Hz), 2.17-2.34 (1H, m), 1.73 – 2.09 (2H, m), 1.47-1.67 (2H, m), 1.26-1.43 (6H, m).
^{13}C NMR (100 MHz, CDCl3) δ (ppm): 174.4, 138.6, 128.6, 124.9, 118.3, 114.2, 46.1, 28.9, 24.8, 24.3. Anal. Calcd for $C_{13}H_{16}N_2O_3$: C, 62.89; H, 6.50; N, 11.28. Found: C, 62.96; H, 6.41; N, 11.21.



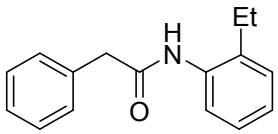
N-(3-chlorophenyl)cyclohexanecarboxamide (4u), 87% yield; white solid; TLC (Petroleumether:Ethyl acetate, 85:17 v/v) IR (thin film) 3114, 3056, 2827, 2551, 2690, 1955, 1818, 1668,1447, 1254, 1244,1035, 1017, 854, cm^{-1}.^{1}H NMR (400 MHz, CDCl3) δ (ppm): 7.71 (s, 1H), 7.53 (t, 1H, J= 7.9 Hz), 7.40 (s, 1H), 7.24 (d, 1H, J= 7.7 Hz), 7.09 (t, 1H, J= 7.9 Hz), 2.01-2.27 (1H, m), 1.73 – 1.95 (2H, m), 1.44-1.59 (2H, m), 1.22-1.34 (6H, m). ^{13}C NMR (100 MHz, CDCl3) δ (ppm): 174.4, 131.8, 127.7, 123.6, 119.8, 117.3, 45.9, 28.9, 25.7, 24.8. Anal. Calcd for $C_{13}H_{16}ClNO$: C, 65.68; H, 6.78; N, 5.89. Found: C, 65.76; H, 6.68; N, 5.80.



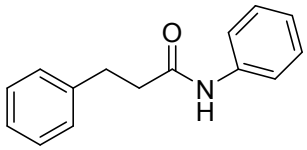
N-(4-chlorophenyl)cyclohexanecarboxamide (4v), 87% yield; white solid; TLC (Petroleumether:Ethyl acetate, 85:17 v/v) IR (thin film) 3113, 3054, 2821, 2547, 2687, 1959, 1821, 1665,1442, 1253, 1244,1037, 1011, 852, cm^{-1}.^{1}H NMR (400 MHz, CDCl3) δ (ppm): 7.43 (d, 2H, J= 7.6 Hz), 7.10-7.25 (3H, m), 2.08-2.38 (1H, m), 1.81 – 2.06 (2H, m), 1.50-1.69 (2H, m), 1.08-1.17 (6H, m). ^{13}C NMR (100 MHz, CDCl3) δ (ppm): 174.7, 136.5, 130.2, 129.0, 121.1, 46.5, 29.6, 26.4, 25.7.Anal. Calcd for $C_{13}H_{16}ClNO$: C, 65.68; H, 6.78; N, 5.89. Found: C, 65.78; H, 6.66; N, 5.78.



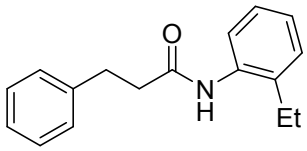
N,2-diphenylacetamide (4w), 90% yield; white solid; TLC (Petroleumether:Ethyl acetate, 85:15 v/v) IR (thin film) 3345, 3055, 3027, 1657, 1625, 1538, 1440, 1301, 1179,1002, 885cm^{-1}; ^{1}H NMR (400 MHz, CDCl3) δ (ppm): 7.50 (2H, d, J = 8.2 Hz), 7.45 (2H, d, J = 7.74 Hz), 7.42 (1H, s), 7.37 (1H, t, J = 7.2 Hz), 7.30 (2H, t, J = 8.3 Hz), 7.09 (2H, t. J=7.2), 3.75 (2H, s). ^{13}C NMR (100 MHz, CDCl3) δ (ppm): 169.2, 137.5, 134.2, 129.8, 129.2, 128.9, 127.7, 124.3, 119.9, 44.6. Anal. Calcd for $C_{14}H_{13}NO$: C, 79.59; H, 6.20; N, 6.63. Found: C, 79.66; H, 6.12; N, 6.69.



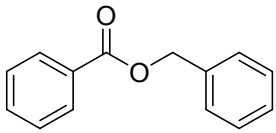
N-(2-ethylphenyl)-2-phenylacetamide (4x), 86% yield; light yellow powder; TLC (Petroleumether:Ethyl acetate, 85:17 v/v) IR (thin film) 3345, 3055, 3027, 1657, 1625, 1538, 1440, 1301, 1179,1002, 885cm^{-1}; ^{1}H NMR (400 MHz, CDCl3) δ (ppm): 7.98 (2H, d, J = 8.2 Hz), 7.44 (2H, d, J = 7.70 Hz), 7.39 (1H, s), 7.27-7.32 (3H, m), 7.22 (1H, t, J = 8.1 Hz), 7.13 (1H, t, J=7.1). 3.82 (1H, s), 2.24 (2H, q, J=7.6), 0.92 (3H, t, J=7.5). ^{13}C NMR (100 MHz, CDCl3) δ (ppm): 169.2, 135.1, 134.8, 130.1, 129.8, 129.5, 128.9, 128.0, 126.8, 125.1, 122.5, 44.9, 24.4, 14.02. Anal. Calcd for $C_{16}H_{17}NO$: C, 80.30; H, 7.16; N, 5.85. Found: C, 80.21; H, 7.22; N, 5.79.



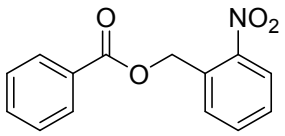
N,3-diphenylpropanamide (4y), 88% yield; white powder; TLC (Petroleumether:Ethyl acetate, 85:16 v/v) IR (thin film) 3345, 3055, 3027, 1657, 1625, 1538, 1440, 1301, 1179,1002, 885cm^{-1}; ^{1}H NMR (400 MHz, CDCl3) δ (ppm): 7.51 (2H, d, J = 8.4 Hz), 7.46 (2H, d, J = 7.75 Hz), 7.35 (1H, s), 7.24 (1H, t, J = 7.1 Hz), 7.14 (2H, t, J = 8.5 Hz), 7.09 (2H, t. J=7.4), 3.09 (2H, t, J=7.9). 2.68 (2H, t, J=7.7). ^{13}C NMR (100 MHz, CDCl3) δ (ppm): 170.6, 140.8, 129.2, 129.0, 128.7, 128.2, 126.5, 124.5, 119.9, 39.3, 31.4. Anal. Calcd for $C_{15}H_{15}NO$: C, 79.97; H, 6.71; N, 6.22. Found: C, 79.89; H, 6.63; N, 6.15.



N-(2-ethylphenyl)-3-phenylpropanamide (4z), 86% yield; light yellow powder; TLC (Petroleumether:Ethyl acetate, 85:17 v/v) IR (thin film) 3345, 3055, 3027, 1657, 1625, 1538, 1440, 1301, 1179,1002, 885cm^{-1}; ^{1}H NMR (400 MHz, CDCl3) δ (ppm): 7.72 (2H, d, J = 8.0 Hz), 7.40 (2H, d, J = 7.71 Hz), 7.32 (1H, s), 7.23-7.29 (3H, m), 7.20 (1H, t, J = 7.99 Hz), 7.14 (1H, t, J=7.0). 3.10 (2H, t, J = 7.35 Hz), 2.75 (2H, t, J =7.62 Hz), 2.42 (2H, q, J=7.70). 1.14 (3H, t, J=7.60). ^{13}C NMR (100 MHz, CDCl3) δ (ppm): 170.4, 140.6, 134.8, 129.0, 127.9, 127.6, 126.5, 126.0, 124.3, 39.1, 31.9, 23.9, 13.7. Anal. Calcd for $C_{17}H_{19}NO$: C, 80.60; H, 7.56; N, 5.53. Found: C, 80.71; H, 7.49; N, 5.58.

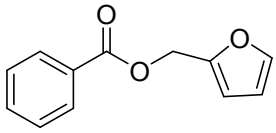


Benzyl benzoate (6a), 98% yield; white solid; TLC (Petroleumether:Ethyl acetate, 85:15 v/v) IR (thin film) 3091, 3034, 2963, 1718, 1603, 1584, 1497, 1451, 1371,1110, 753cm^{-1}; ^{1}H NMR (400 MHz, CDCl3) δ (ppm): 8.16 (2H, d, J = 8.4 Hz), 7.57 (1H, t, J = 7.6 Hz), 7.51 (2H, t, J = 7.9 Hz), 7.43-7.48 (4H, m), 7.39 (1H, t. J=7.6), 5.44 (2H, s). ^{13}C NMR (100 MHz, CDCl3) δ (ppm): 166.5, 136.4, 133.2, 130.4, 129.9, 128.8, 128.6, 128.4, 66.9. Anal. Calcd for $C_{14}H_{12}O_2$: C, 79.23; H, 5.70. Found: C, 79.14; H, 5.76.

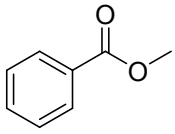


2-Nitrobenzyl benzoate (6b), 94% yield; white solid; TLC (Petroleumether:Ethyl acetate, 85:17 v/v) IR (thin film) 3088, 3032, 2962, 1709, 1602, 1584, 1548, 1496, 1449, 1371, 1344, 1107, 751cm^{-1}; ^{1}H NMR (400 MHz, CDCl3) δ (ppm): 8.18 (2H, d, J = 8.2 Hz), 8.11 (1H, t, J = 7.4 Hz), 7.68 (2H, t, J = 7.7 Hz), 7.62 (1H, d. J=7.4), 7.46-7.58 (3H, m), 5.78 (2H, s). ^{13}C NMR (100 MHz,

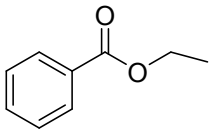
CDCl3) δ (ppm): 166.0, 147.8, 133.9, 133.5, 132.5, 130.2, 129.9, 129.1, 128.9, 128.7, 125.2, 63.4. Anal. Calcd for $C_{14}H_{11}NO_4$: C, 65.37; H, 4.31; N, 5.45. Found: C, 65.29; H, 4.25; N, 5.40.



Furan-2-ylmethyl benzoate (6c), 83% yield; colorless liquid; TLC (Petroleumether:Ethyl acetate, 85:17 v/v) IR (thin film) 3150, 3121, 3030, 2957, 1700, 1563, 1584, 1485, 1440, 1172, 1061, 749cm^{-1}; ^{1}H NMR (400 MHz, CDCl3) δ (ppm): 8.05 (2H, d, J = 7.6 Hz), 7.54 (1H, t, J = 7.4 Hz), 7.45 (1H, d, J = 7.8 Hz), 7.41 (2H, t. J=7.6), 6.58 (1H, d. J=7.4), 6.48 (1H, t. J=7.8), 5.31 (2H, s). ^{13}C NMR (100 MHz, CDCl3) δ (ppm): 166.3, 149.6, 143.4, 133.2, 130.0, 129.9, 128.5, 110.9, 110.7. Anal. Calcd for $C_{12}H_{10}O_3$: C, 71.28; H, 4.98. Found: C, 71.19; H, 4.91.

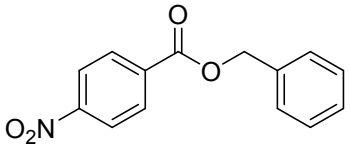


Methyl benzoate (6d), 92% yield; yellowish oil; TLC (Petroleumether:Ethyl acetate, 85:15 v/v) IR (thin film) 3066, 3036, 2952, 1728, 1603, 1492,1272, 1177, 1115, 970cm^{-1}; ^{1}H NMR (400 MHz, CDCl3) δ (ppm): 8.01 (2H, d, J = 7.4 Hz), 7.50 (1H, t, J = 7.9 Hz), 7.38 (2H, t, J = 8.3 Hz), 3.81 (3H, s). ^{13}C NMR (100 MHz, CDCl3) δ (ppm): 166.9, 132.7, 130.3, 129.8, 128.4, 52.0. Anal. Calcd for $C_8H_8O_2$: C, 70.58; H, 5.92. Found: C, 70.46; H, 5.85.

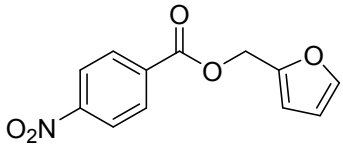


Ethyl benzoate (6e), 90% yield; white oil; TLC (Petroleumether:Ethyl acetate, 85:15 v/v) IR (thin film) 3091, 3064, 2989, 1719, 1585, 1491,1393, 1277, 1109, 711cm^{-1}; ^{1}H NMR (400 MHz, CDCl3) δ (ppm): 8.04 (2H, d, J = 7.4 Hz), 7.50 (1H, t, J = 7.9 Hz), 7.38 (2H, t, J = 8.3 Hz), 4.35 (q, 2H, J = 6.2 Hz), 1.36 (t, 3H, J = 6.2 Hz). ^{3}C NMR (100 MHz, CDCl3) δ (ppm): 165.9, 132.4,

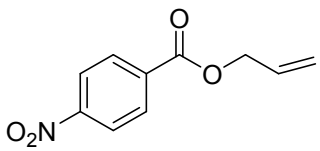
130.0, 128.9, 128.9, 128.0, 60.3, 13.5. Anal. Calcd for $C_9H_{10}O_2$: C, 71.98; H, 6.71. Found: C, 71.83; H, 6.79.



Benzyl 4-nitrobenzoate (6f), 85% yield; white solid; TLC (Petroleumether:Ethyl acetate, 85:18 v/v) IR (thin film) 3089, 3032, 2961, 1710, 1603, 1583, 1550, 1495, 1451, 1379, 1352,1107, 752cm^{-1}; ^{1}H NMR (400 MHz, CDCl3) δ (ppm): 8.3 (2H, d, J = 8.4 Hz), 8.25 (2H, d, J = 8.4 Hz), 7.34-7.51 (5H, m), 5.45 (2H, s).^{13}C NMR (100 MHz, CDCl3) δ (ppm): 164.1, 150.6, 135.5, 135.2, 130.8, 128.7, 128.7, 128.4, 123.5, 67.91. Anal. Calcd for $C_{14}H_{11}NO_4$: C, 65.37; H, 4.31; N, 5.45. Found: C, 65.43; H, 4.26; N, 5.39.

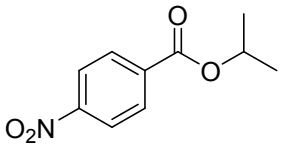


Furan-2-ylmethyl 4-nitrobenzoate (6g), 85% yield; clear brown liquid; TLC (Petroleumether:Ethyl acetate, 85:20 v/v) IR (thin film) 3149, 3119, 3032, 2959, 1706, 1603, 1579, 1550, 1490, 1448, 1379, 1352,1159, 747cm^{-1}; ^{1}H NMR (400 MHz, CDCl3) δ (ppm): 8.23 (4H, d, J = 7.8 Hz), 7.48 (1H, d, J = 8 Hz), 6.52 (1H, t, J = 8 Hz), 6.42 (1H, d. J=8), 5.35 (2H, s). ^{13}C NMR (100 MHz, CDCl3) δ (ppm): 166.0, 139.2, 132.1, 130.4, 128.9, 127.2, 124.8, 120.7, 118.0. Anal. Calcd for $C_{12}H_9NO_5$: C, 58.30; H, 3.67; N, 5.67. Found: C, 58.22; H, 3.61; N, 5.65.

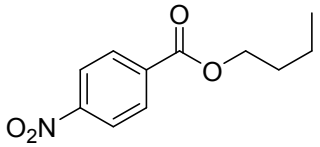


Allyl 4-nitrobenzoate (6h), 89% yield; yellow solid; TLC (Petroleumether:Ethyl acetate, 85:17 v/v) IR (thin film) 3092, 3062, 2986, 1722, 1655, 1582, 1488,1391, 1272, 1105, 715cm^{-1}; ^{1}H NMR (400 MHz, CDCl3) δ (ppm): 8.22 (2H, d, J = 7.6 Hz), 8.16 (2H, d, J = 7.6 Hz), 5.96-6.04 (1H, m), 5.40 (1H, d, J = 4.6 Hz), 5.34 (2H, d, J = 4.6 Hz), 4.81 (2H, d, J=2.8). ^{13}C NMR (100 MHz, CDCl3)

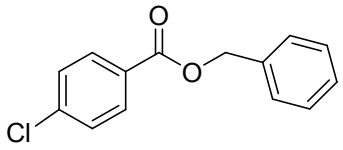
δ (ppm): 164.3, 150.6, 135.6, 131.7, 130.8, 123.6, 119.1, 66.3. Anal. Calcd for $C_{10}H_9NO_4$: C, 57.97; H, 4.38; N, 6.76. Found: C, 57.92; H, 4.34; N, 6.82.



Isopropyl 4-nitrobenzoate (6i), 88% yield; white solid; TLC (Petroleumether:Ethyl acetate, 85:17 v/v) IR (thin film) 3086, 3055, 2993, 1723, 1575, 1481,1393, 1270, 1106, 712cm^{-1}; ^{1}H NMR (400 MHz, CDCl3) δ (ppm): 8.26 (2H, d, J = 7.8 Hz), 8.18 (2H, d, J = 7.8 Hz), 5.25-5.30 (1H, m), 1.38 (6H, d, J=7.2).^{13}C NMR (100 MHz, CDCl3) δ (ppm): 164.4, 150.5, 136.5, 130.8, 123.6, 70.0 , 21.9. Anal. Calcd for $C_{10}H_{11}NO_4$: C, 57.41; H, 5.30; N, 6.70; Found: C, 57.34; H, 5.39; N, 6.66.

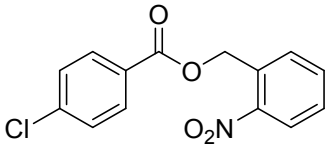


Butyl benzoate (6j), 89% yield; white solid; TLC (Petroleumether:Ethyl acetate, 85:17 v/v) IR (thin film) 3091, 3064, 2961, 1721, 1602, 1555,1492, 1387, 1275, 1110, 739cm^{-1}; ^{1}H NMR (400 MHz, CDCl3) δ (ppm): 8.25 (2H, d, J = 7.9 Hz), 8.19 (2H, d, J = 7.9 Hz), 4.36 (2H, t, J = 6.3 Hz), 1.56-1.90 (2H, m), 1.36-1.49 (2H, m), 0.98 (3H, t, J = 7.5 Hz). ^{13}C NMR (100 MHz, CDCl3) δ (ppm): 164.7, 150.6, 136.2, 130.2, 123.9, 65.9, 30.8, 19.2, 13.9. Anal. Calcd for $C_{11}H_{14}O_2$: C, 74.13; H, 7.92. Found: C, 74.22; H, 7.87.

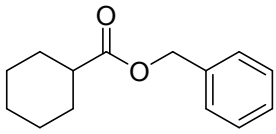


Benzyl 4-chlorobenzoate (6l), 94% yield; white solid; TLC (Petroleumether:Ethyl acetate, 85:17 v/v) IR (thin film) 3095, 3034, 2966, 1719, 1607, 1589, 14958, 1456, 1383,1109, 756cm^{-1}; ^{1}H NMR (400 MHz, CDCl3) δ (ppm): 8.12 (2H, d, J = 8.4 Hz), 8.05 (2H, d, J = 8.4 Hz), 7.40-7.55 (5H, m,), 5.41 (2H, s).^{13}C NMR (100 MHz, CDCl3) δ (ppm): 165.0, 139.0, 131.4, 130.6, 128.8,

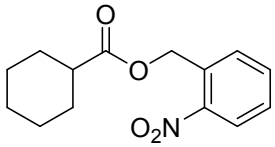
128.2, 128.1, 127.8, 127.7, 66.5. Anal. Calcd for $C_{14}H_{11}ClO_2$: C, 68.16; H, 4.49. Found: C, 68.26; H, 4.55.



2-Nitrobenzyl 4-chlorobenzoate (6m), 90% yield; white solid; TLC (Petroleumether:Ethyl acetate, 85:18 v/v) IR (thin film) 3083, 3031, 2959, 1709, 1602, 1582, 1553, 1494, 1452, 1377, 1351,1106, 755cm^{-1}; ^{1}H NMR (400 MHz, CDCl3) δ (ppm): 8.15 (1H, d, J = 8.3 Hz), 8.08 (2H, d, J = 8.5 Hz), 8.01 (1H, t, J=8.2), 7.68 (2H, d, J = 8.3 Hz), 7.28-7.50 (2H, m), 5.75 (2H, s). ^{13}C NMR (100 MHz, CDCl3) δ (ppm): 165.3, 140.0, 133.9, 132.1, 132.0, 131.2, 129.5, 129.2, 129.1, 125.2, 63.7. Anal. Calcd for $C_{14}H_{10}ClNO_4$: C, 57.65; H, 3.46; N, 4.80. Found: C, 57.58; H, 3.40; N, 4.72.

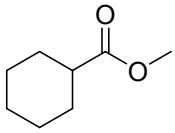


Benzyl cyclohexanecarboxylate (6n), 81% yield; white solid; TLC (Petroleumether:Ethyl acetate, 85:15 v/v) IR (thin film) 3112, 3063, 2921, 2833, 2550, 2692, 1961, 1815, 1720,1452, 1256, 1240,1038, 1014, 851. cm^{-1}. ^{1}H NMR (400 MHz, CDCl3) δ (ppm): 7.33-7.50 (5H, m), 5.13 (2H, s), 1.97-2.38 (1H, m), 1.66-1.95 (2H, m), 1.49-1.57 (2H, m), 1.23-1.46 (6H, m). ^{13}C NMR (100 MHz, CDCl3) δ (ppm): 175.7, 136.5, 128.7, 128.6, 128.2, 128.1, 65.9, 43.0, 28.9, 25.9, 25.5. Anal. Calcd for $C_{13}H_{17}NO$: C, 76.81; H, 8.43; N, 6.89. Found: C, 76.74; H, 8.36; N, 6.80.

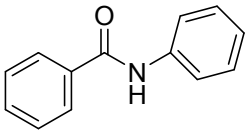


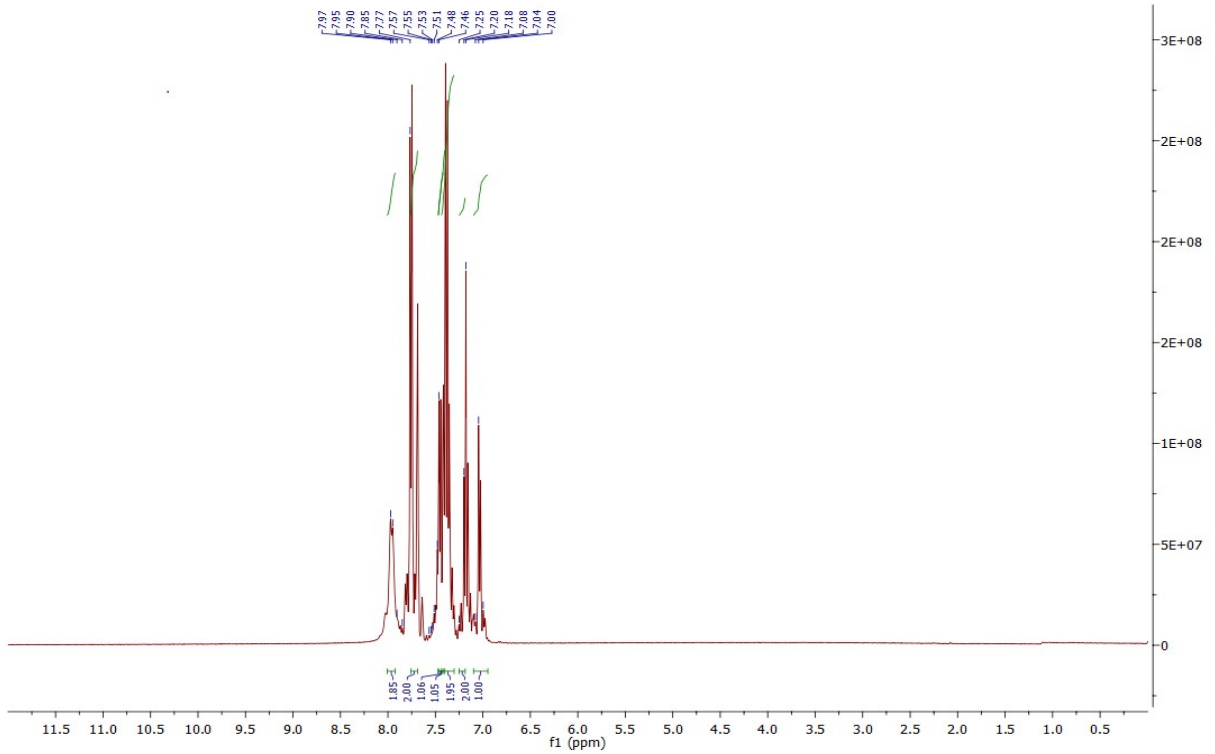
2-Nitrobenzyl cyclohexanecarboxylate (6o), 80% yield; colorless oil; TLC (Petroleumether:Ethyl acetate, 85:18 v/v) IR (thin film) 3114, 3061, 2925, 2836, 2556, 2687, 1960, 1815, 1717, 1547,1450, 1349, 1250, 1243,1035, 1011, 852 cm^{-1}. ^{1}H NMR (400 MHz, CDCl3) δ (ppm): 8.02 (1H, d, J= 7.6 Hz), 7.61 (1H, t, J= 7.6 Hz), 7.53 (1H, t, J= 7.5 Hz), 7.43 (1H, d, J= 7.7 Hz), 5.40 (2H, s), 2.33-2.38 (1H, m), 1.71-1.92 (2H, m), 1.42-1.70 (2H, m), 1.17-1.29 (6H, m).

^{13}C NMR (100 MHz, CDCl3) δ (ppm): 147.7, 133.7, 132.5, 129.1, 128.8, 125.0, 62.7, 43.0, 28.9, 26.1, 25.2. Anal. Calcd for $C_{13}H_{17}NO$: C, 76.81; H, 8.43; N, 6.89. Found: C, 76.74; H, 8.36; N, 6.80.

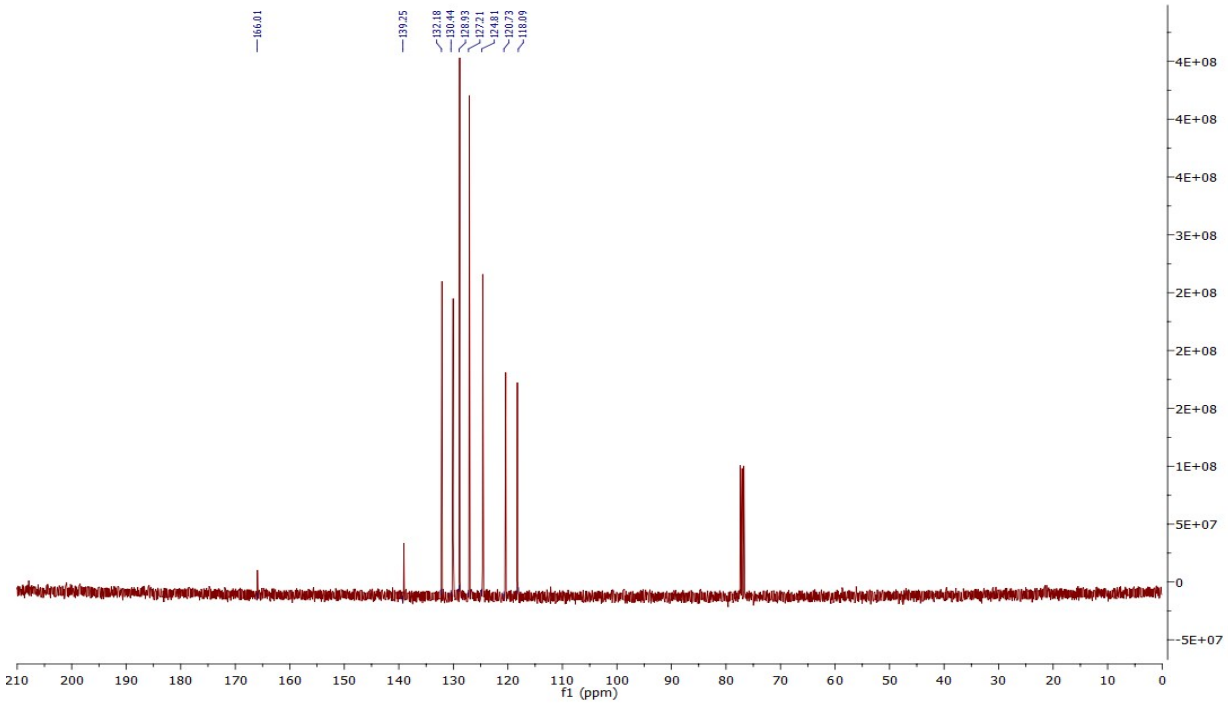


Methyl cyclohexanecarboxylate (6p), 77% yield; yellow oil; TLC (Petroleumether:Ethyl acetate, 85:15 v/v) IR (thin film), 2935, 2855, 2668, 1735,1452, 1436, 1355, 1230,1133, 986 cm^{-1}. ^{1}H NMR (400 MHz, CDCl3) δ (ppm): ^{13}C NMR (100 MHz, CDCl3) δ (ppm): 182.5, 45.9, 43.0, 28.8, 25.8, 25.4. Anal. Calcd for $C_8H_{14}O_2$: C, 67.57; H, 9.92. Found: C, 67.48; H, 9.98.

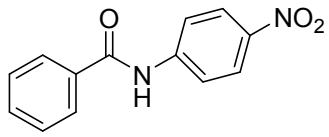


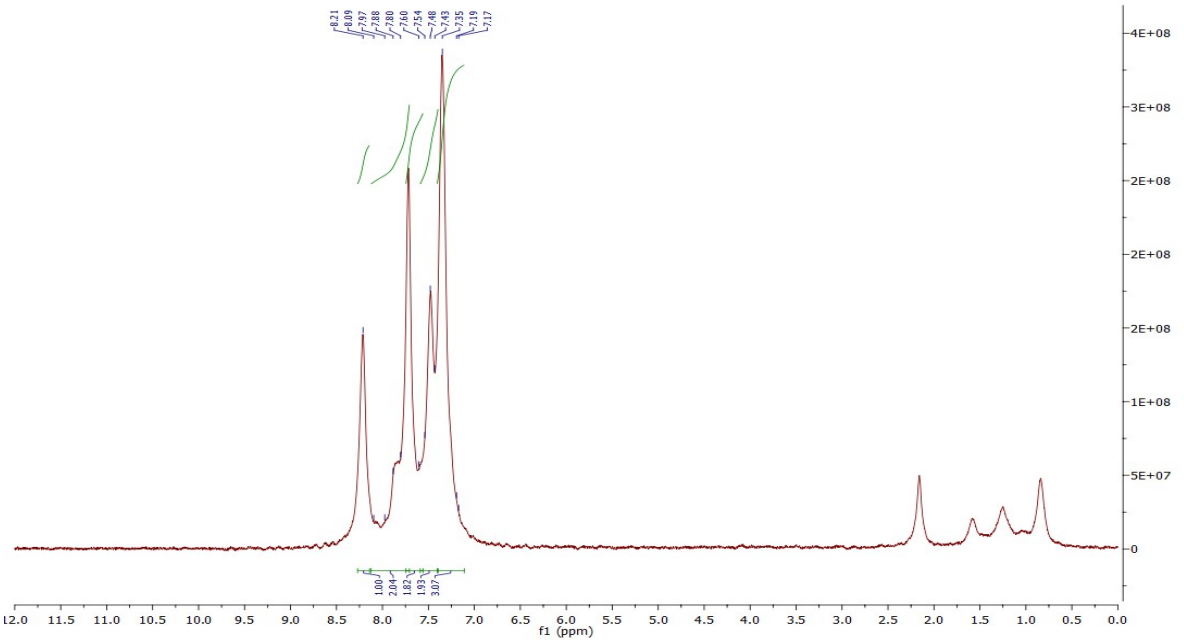


^{1}HNMR (4a)

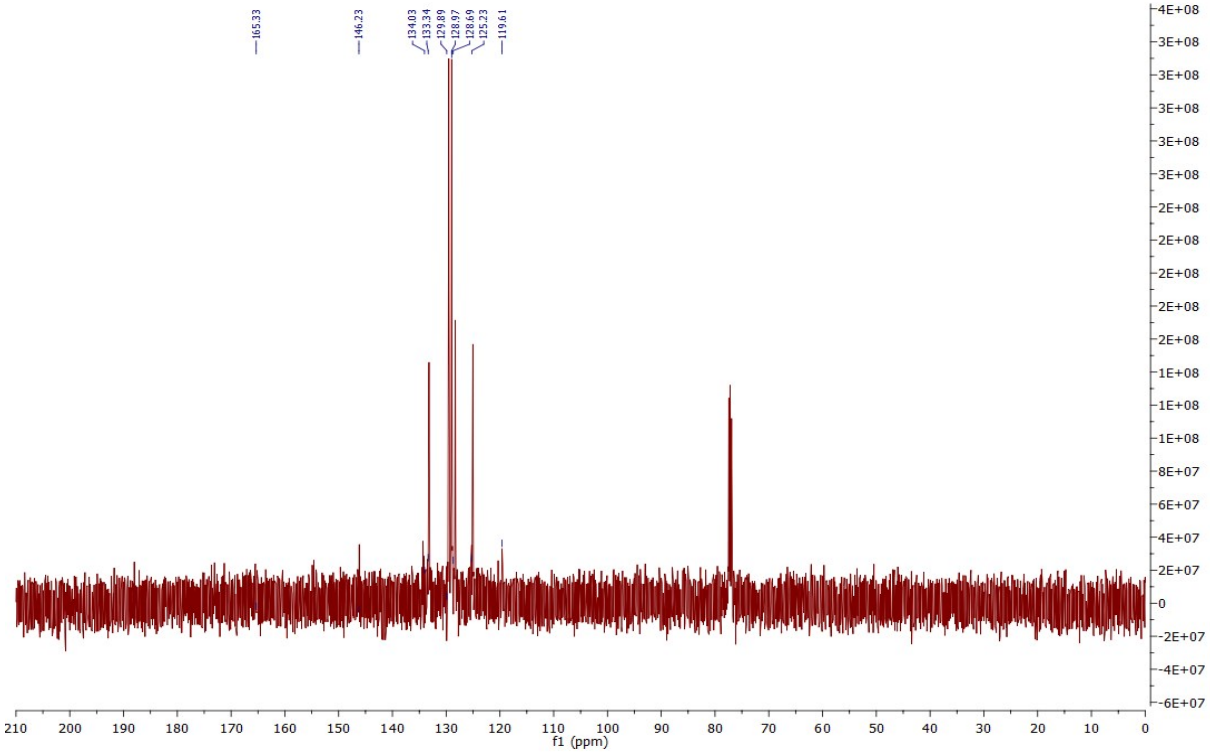


^{13}CNMR (4a)





[1]HNMR (4b)



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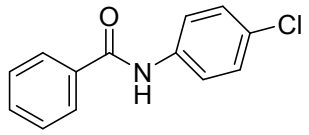

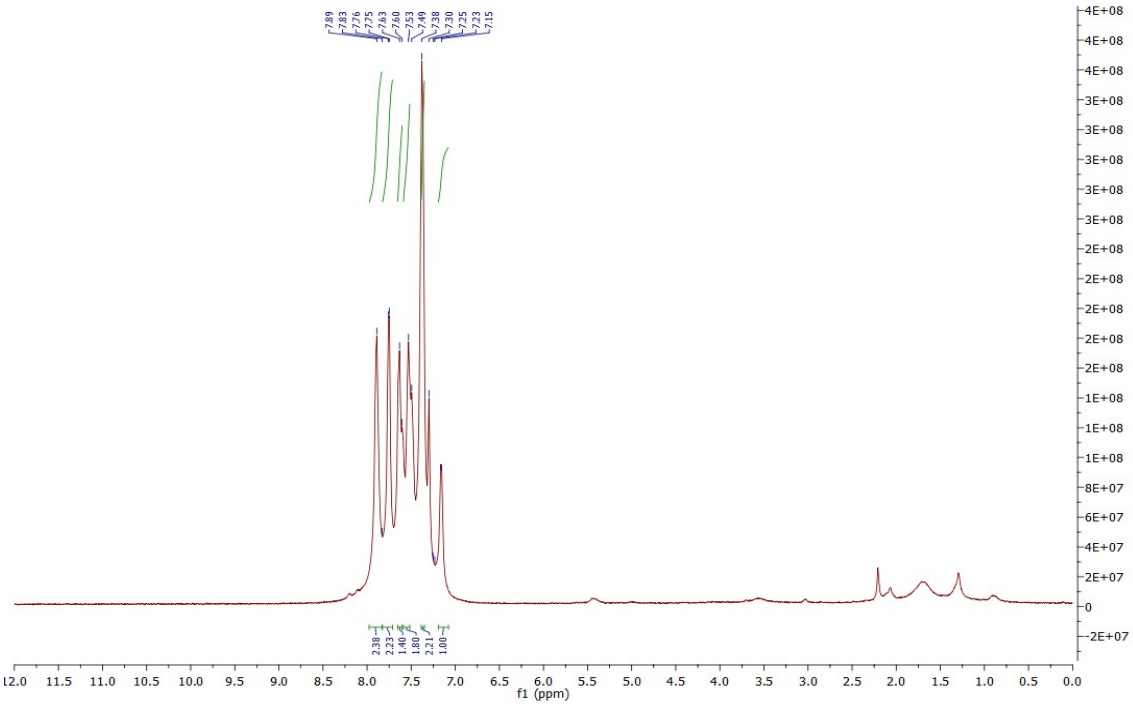

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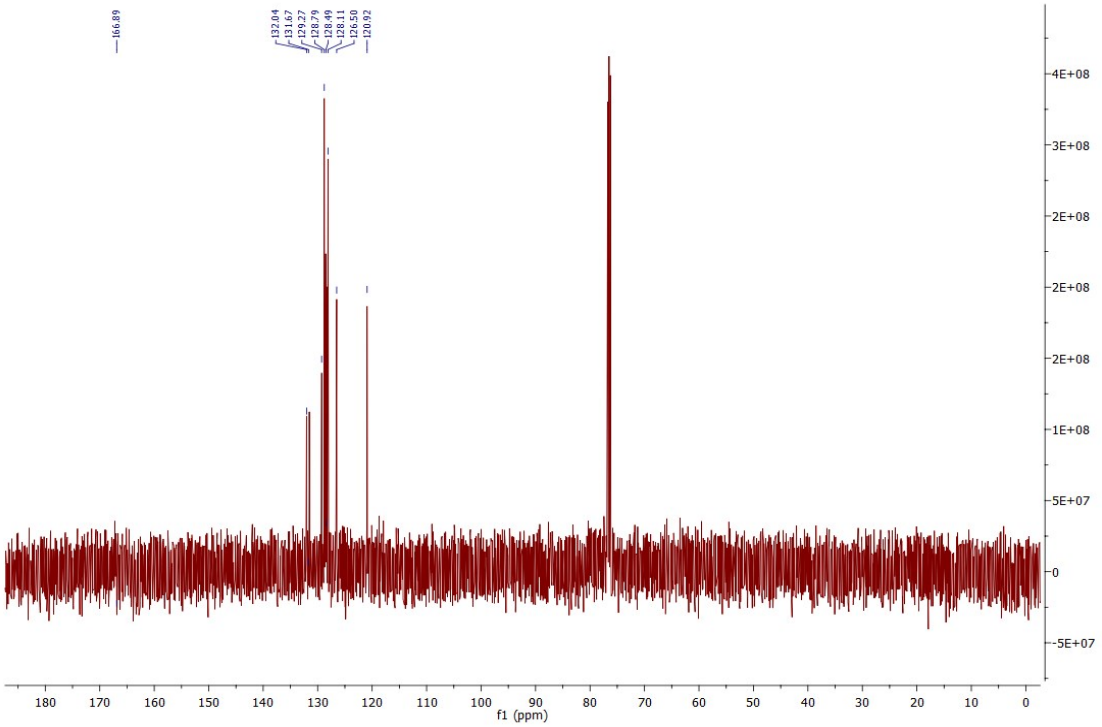

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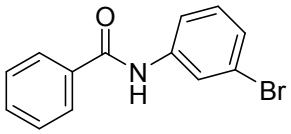

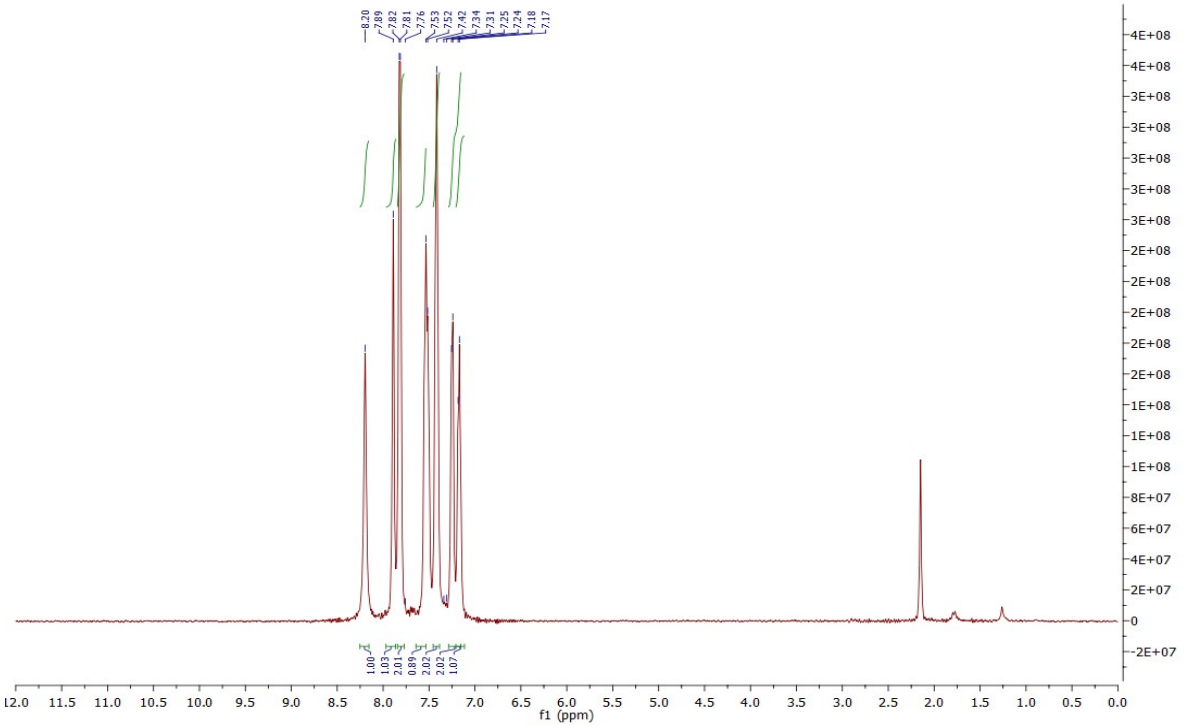

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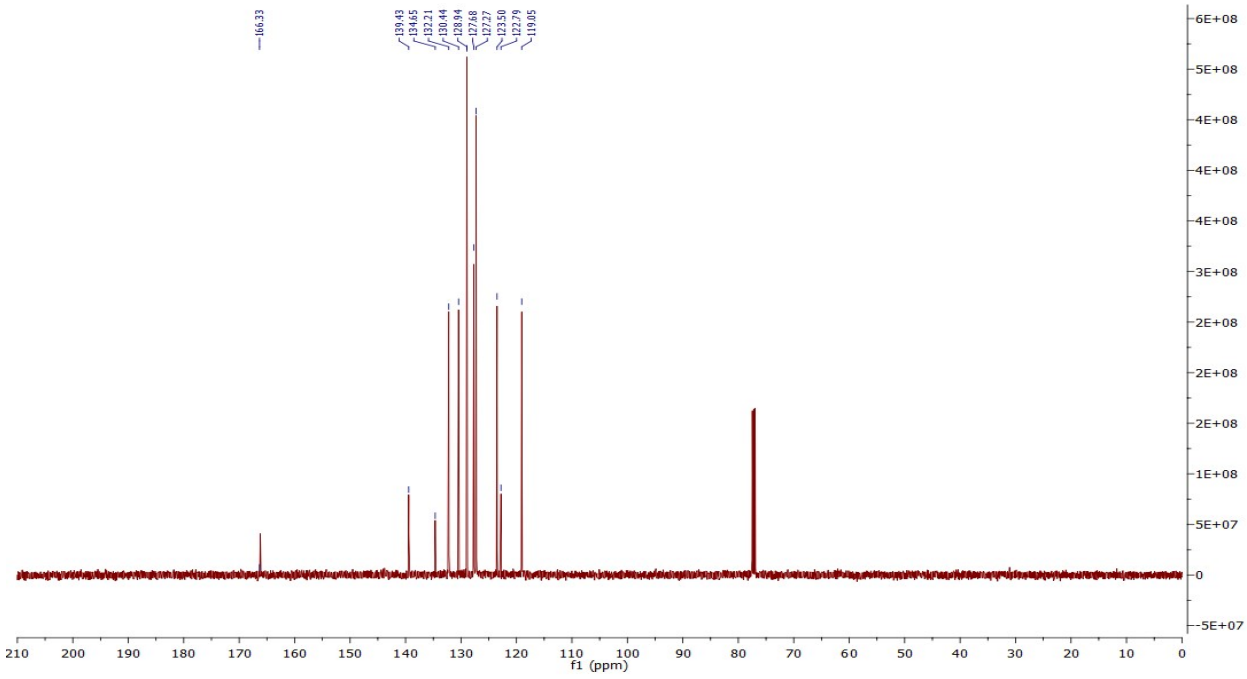

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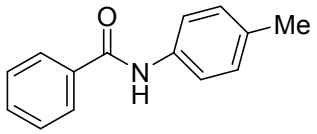

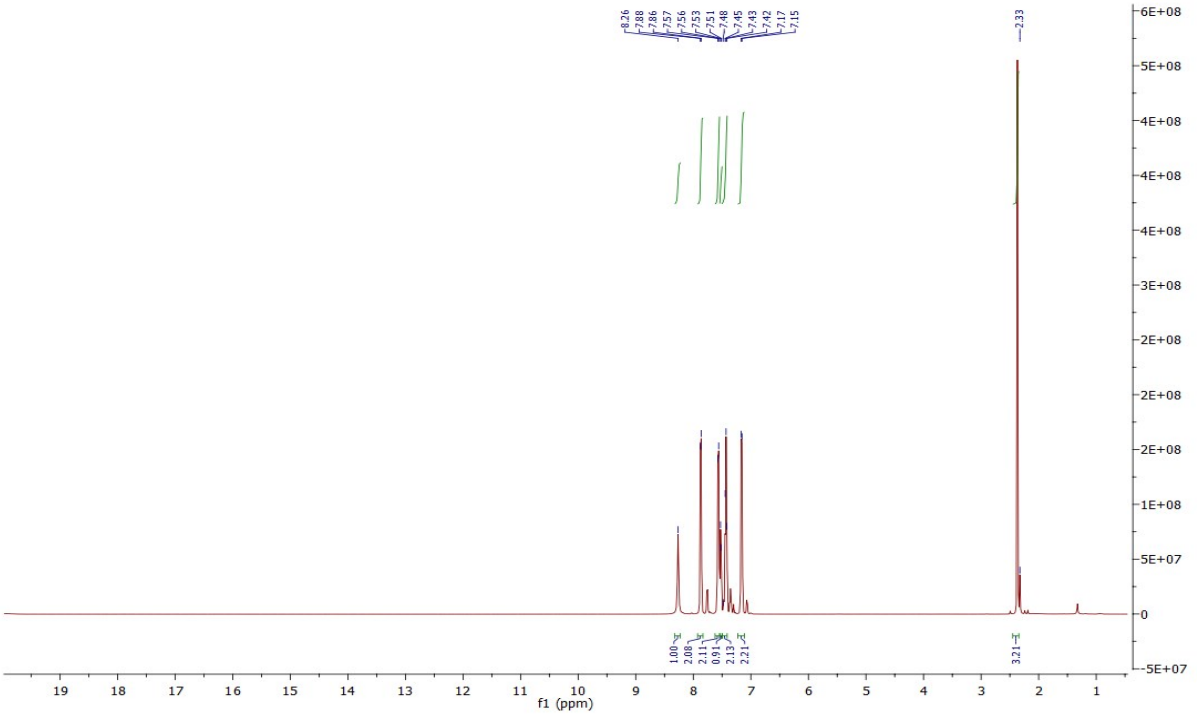

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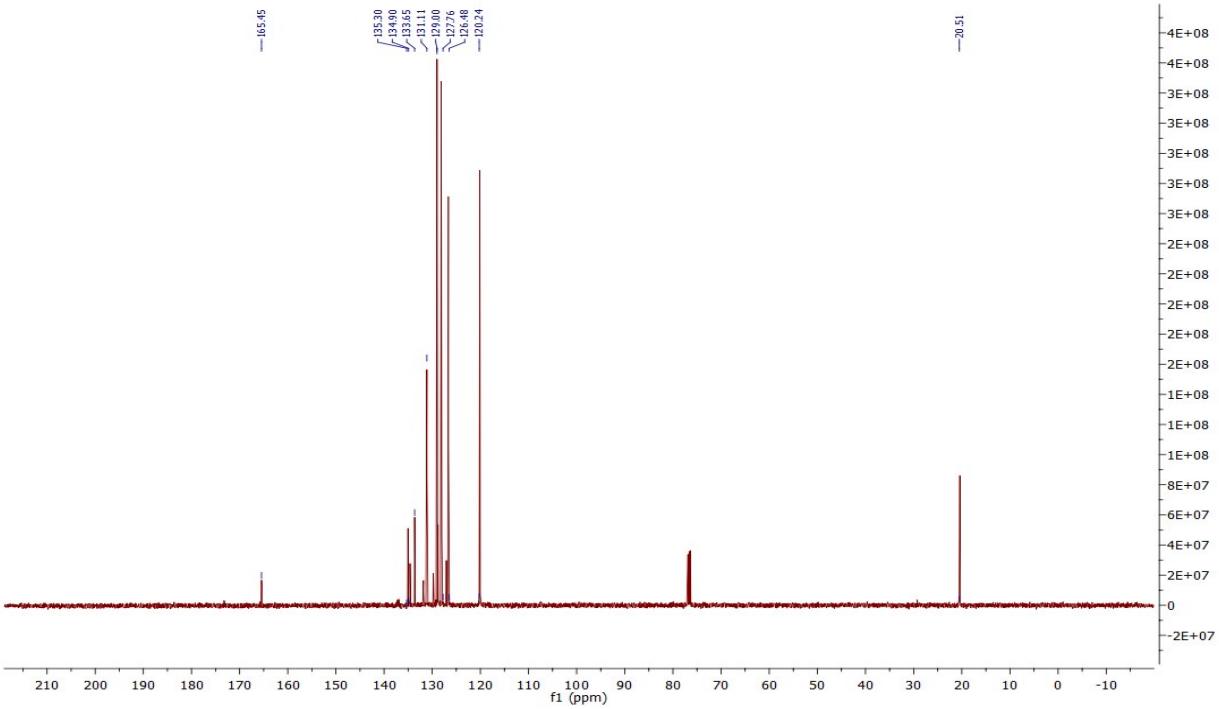

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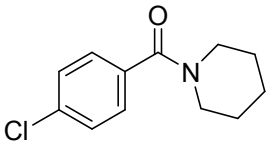

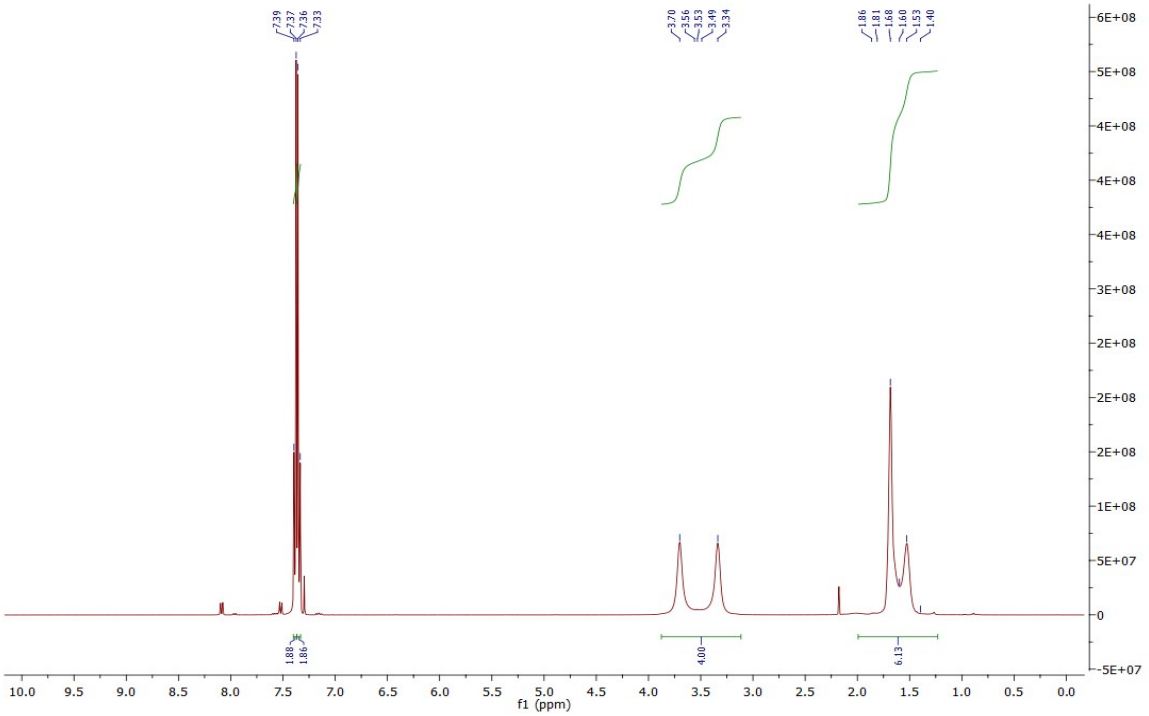

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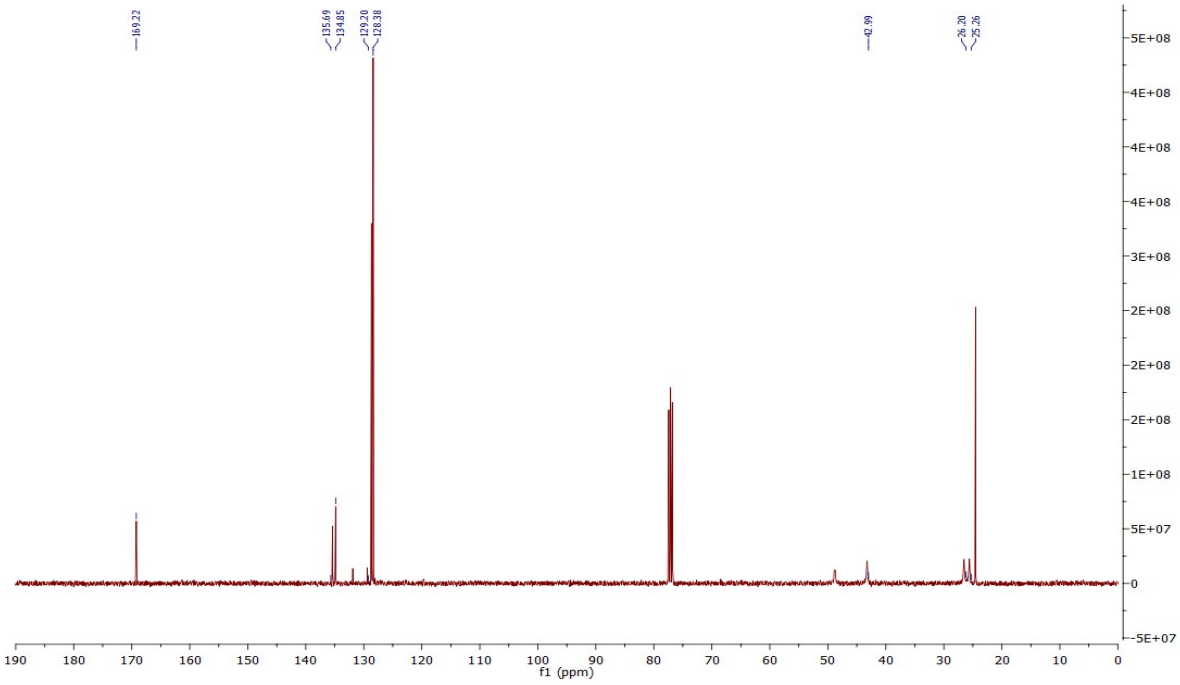

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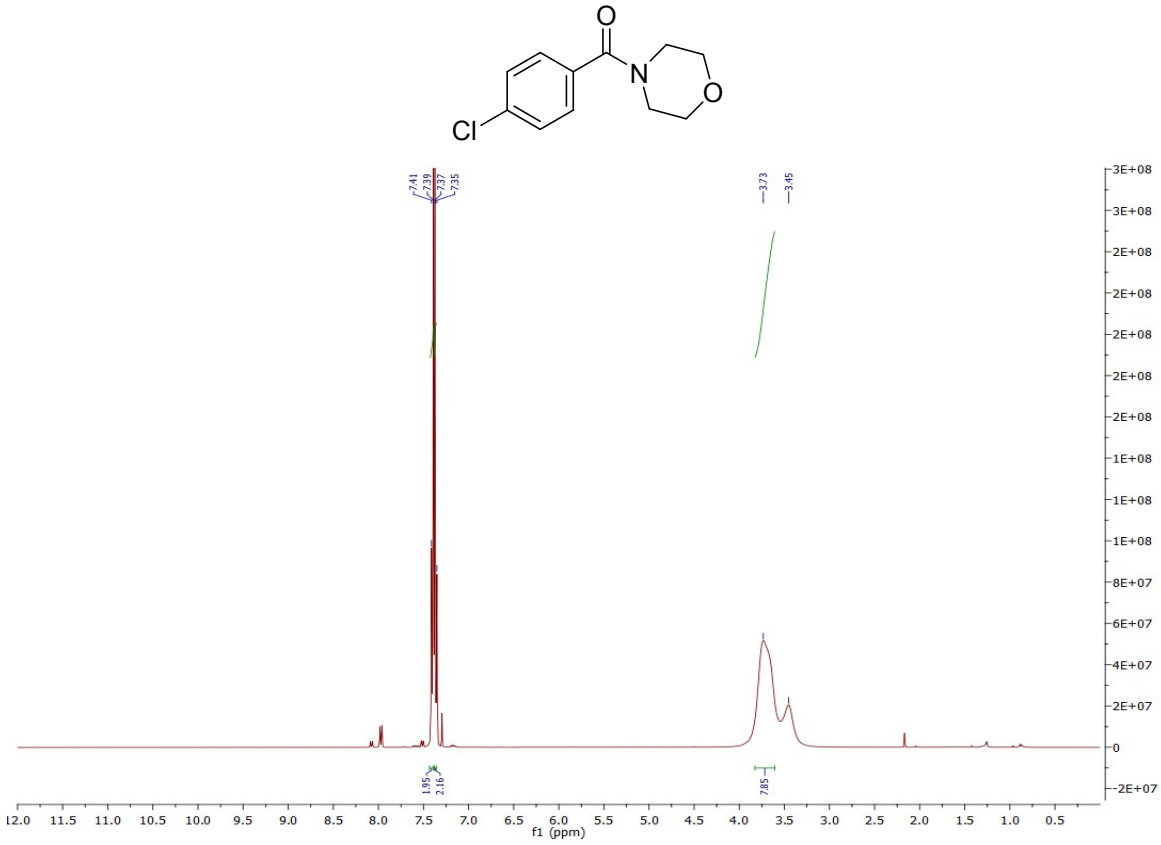

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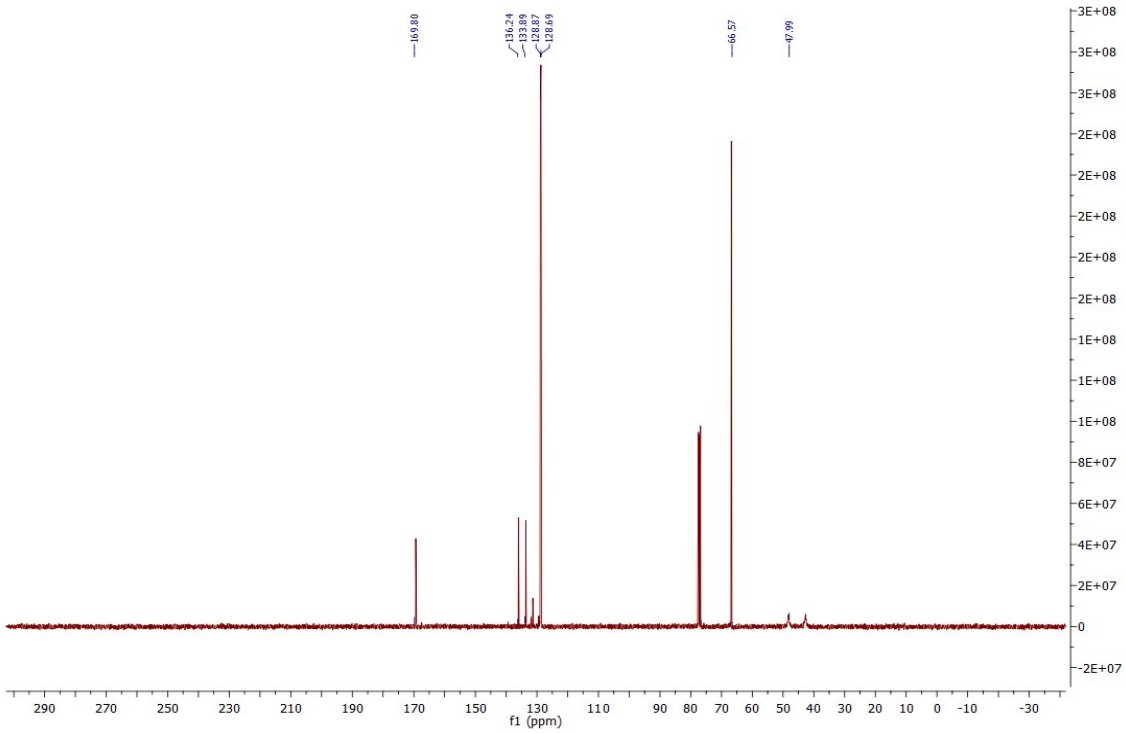

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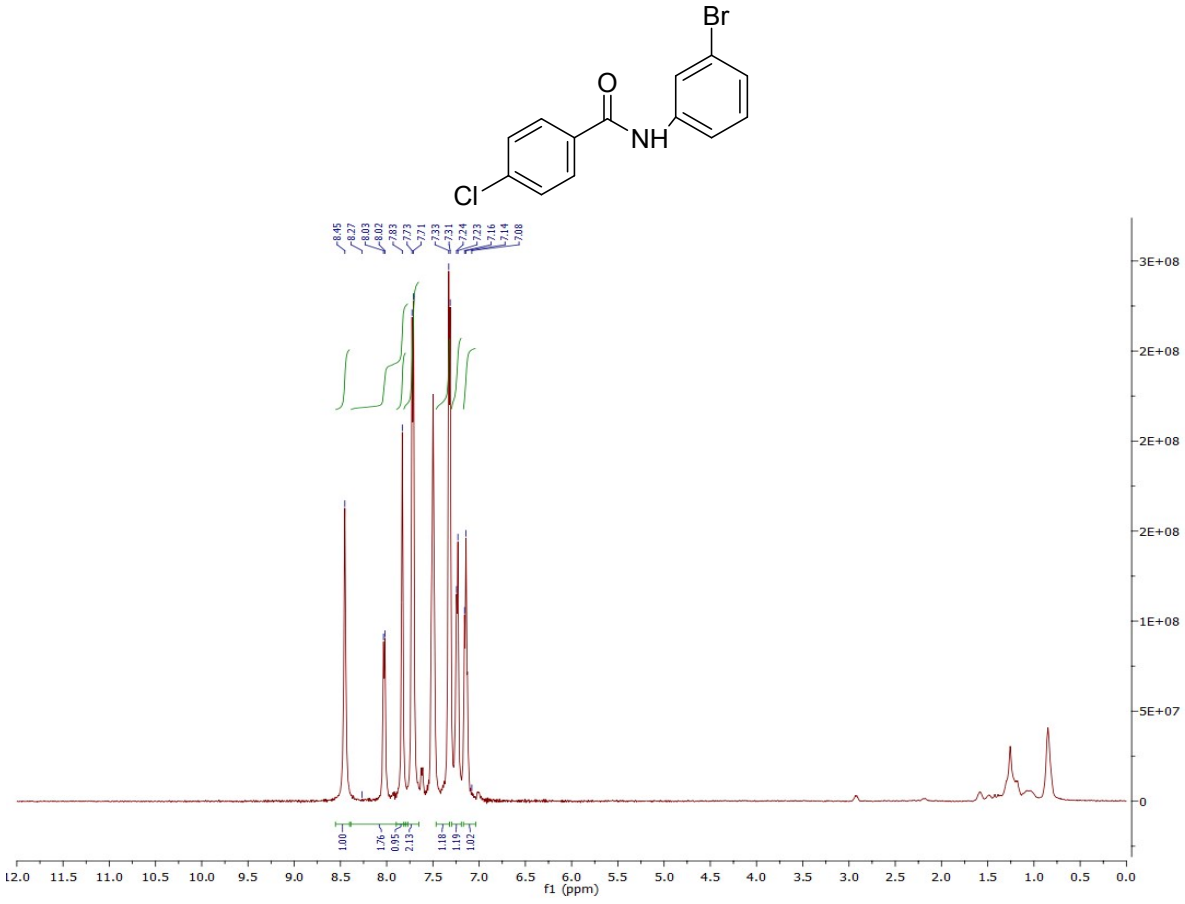

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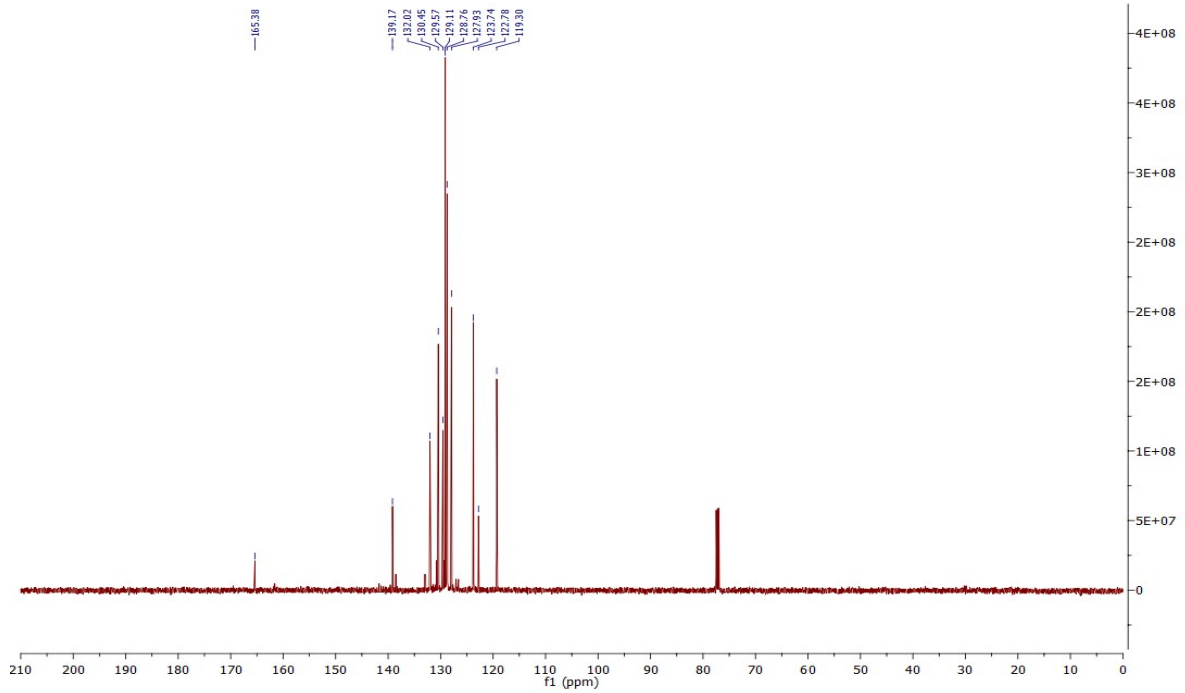

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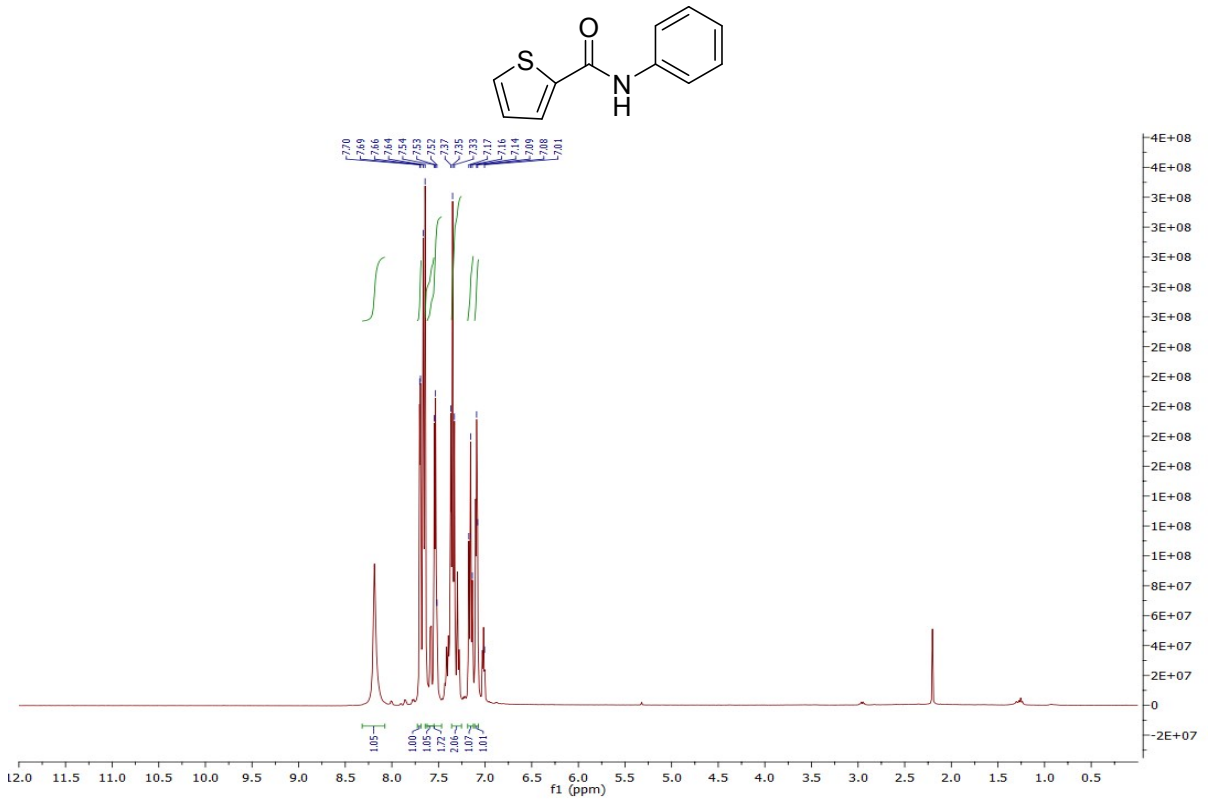

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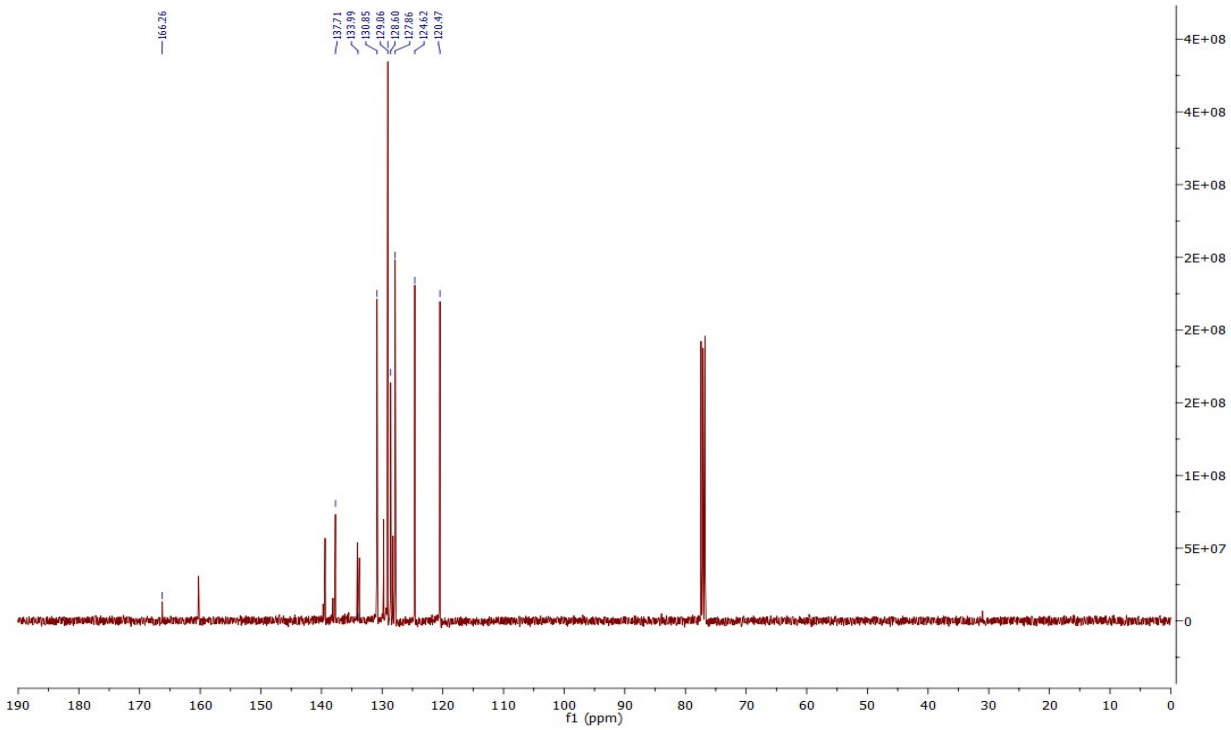

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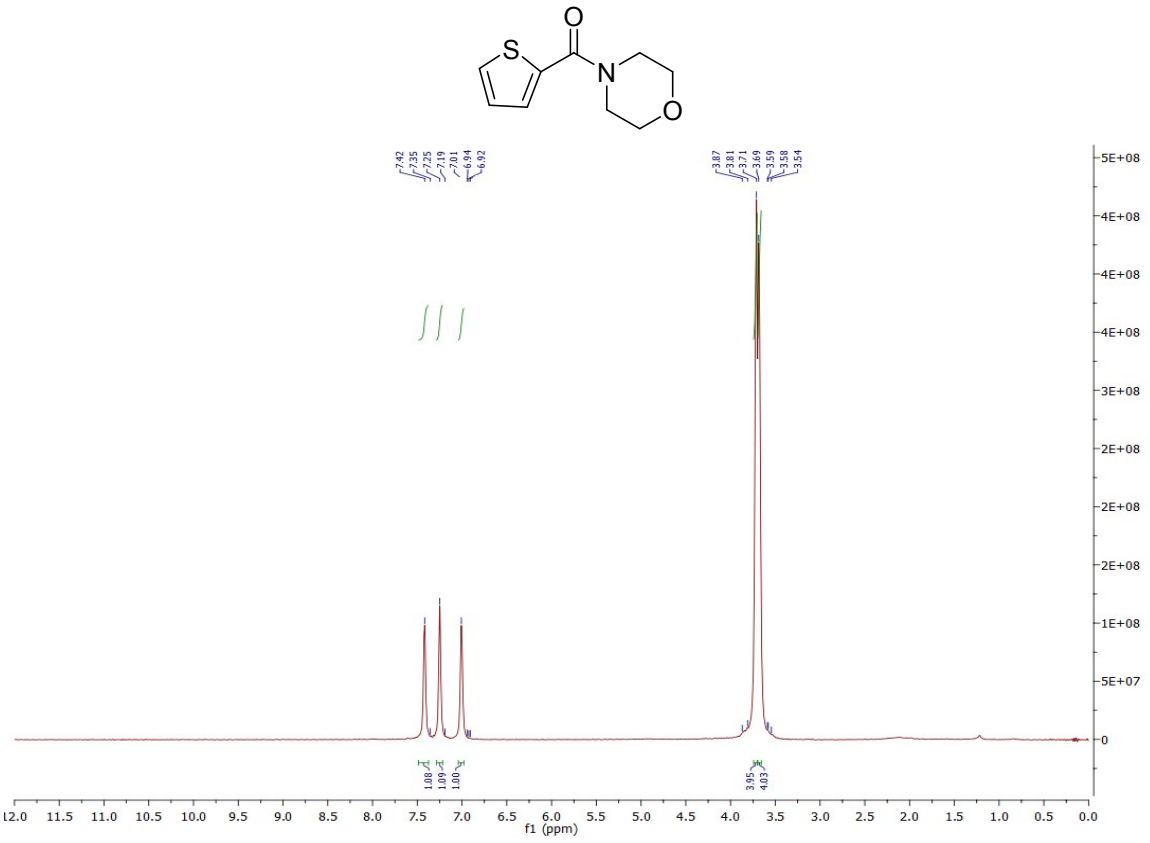

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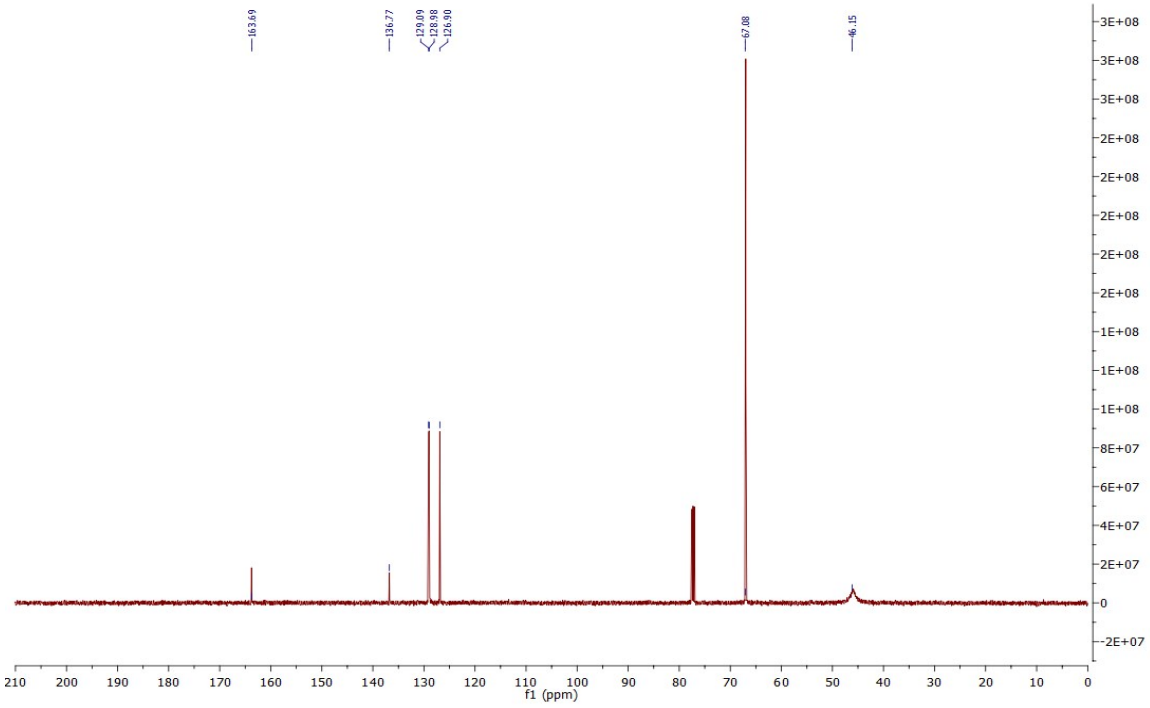

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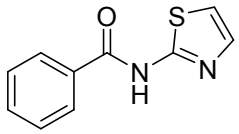

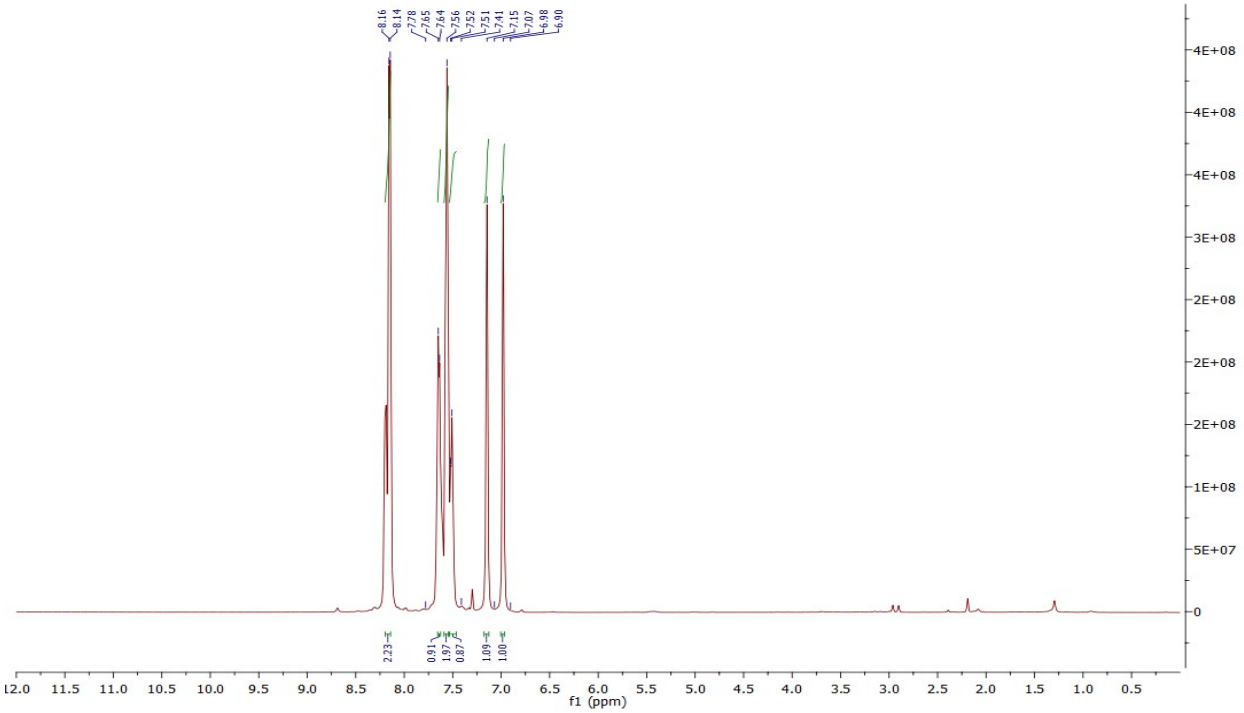

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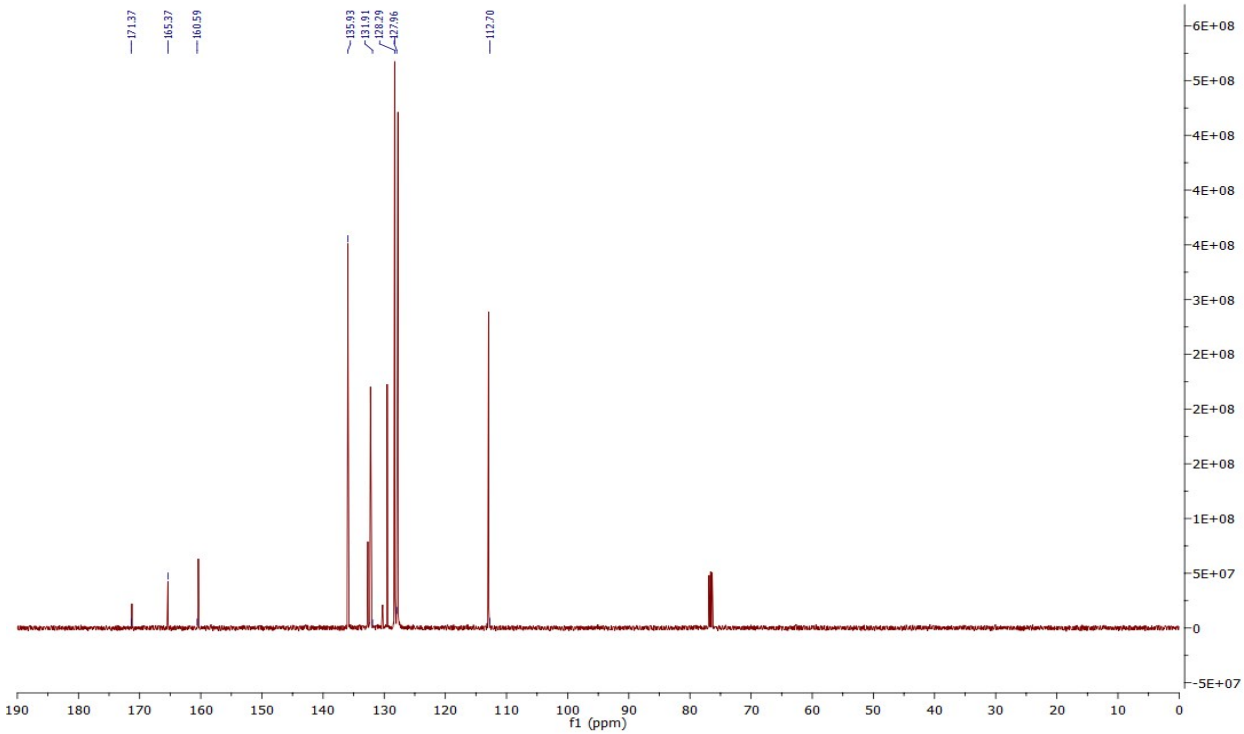

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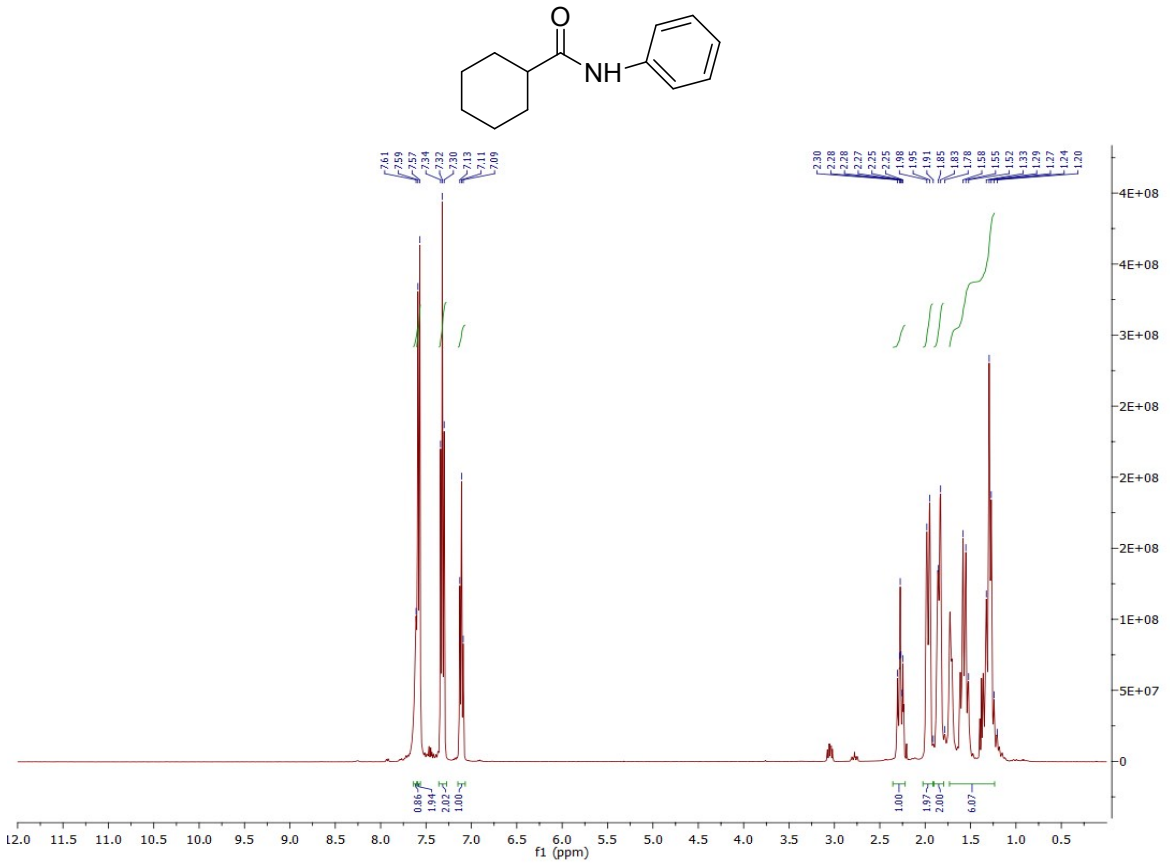

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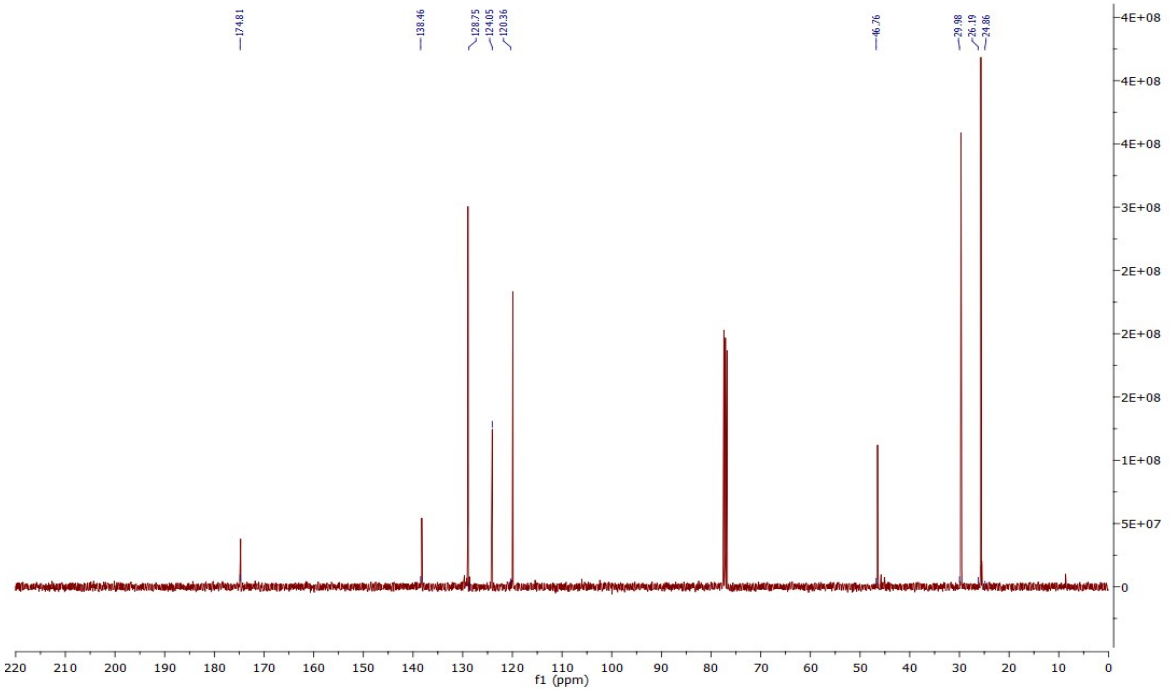

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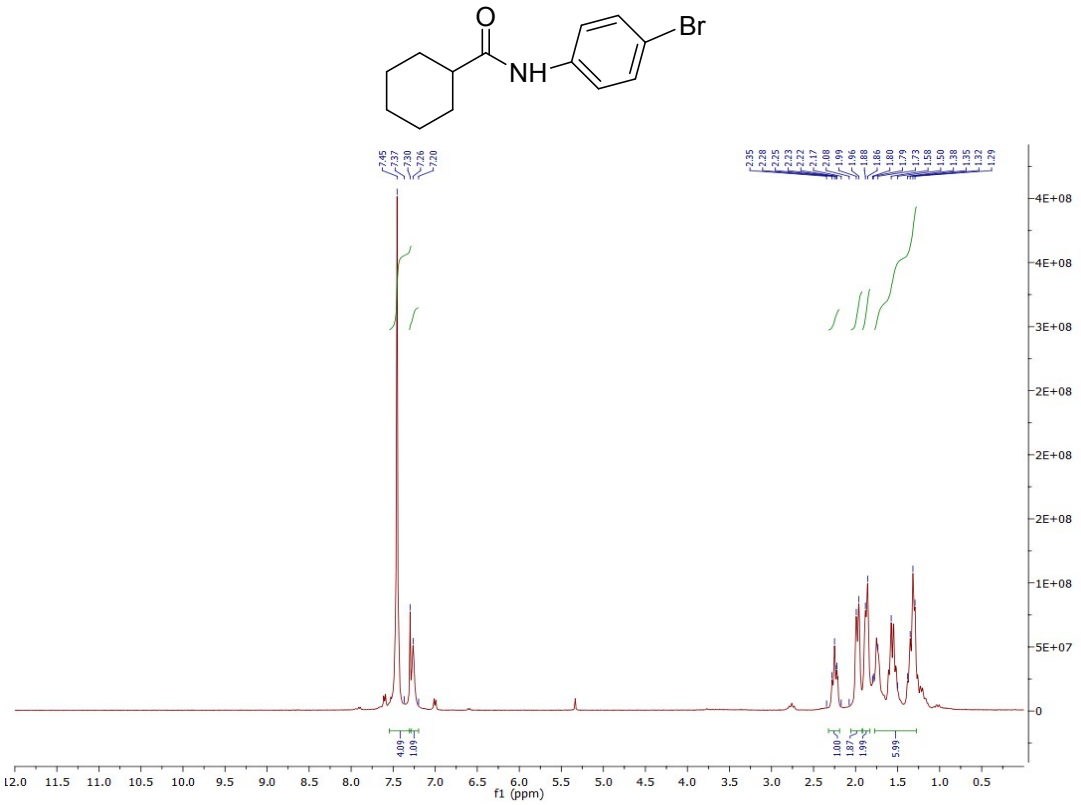

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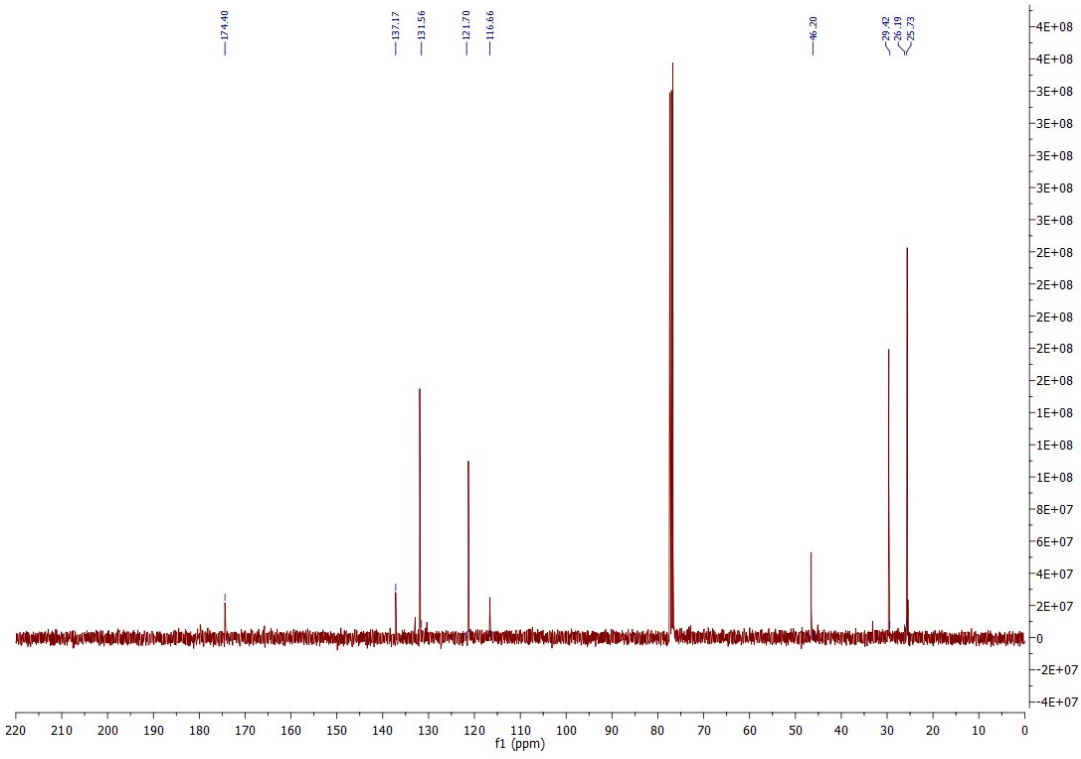

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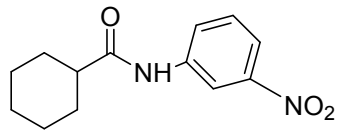

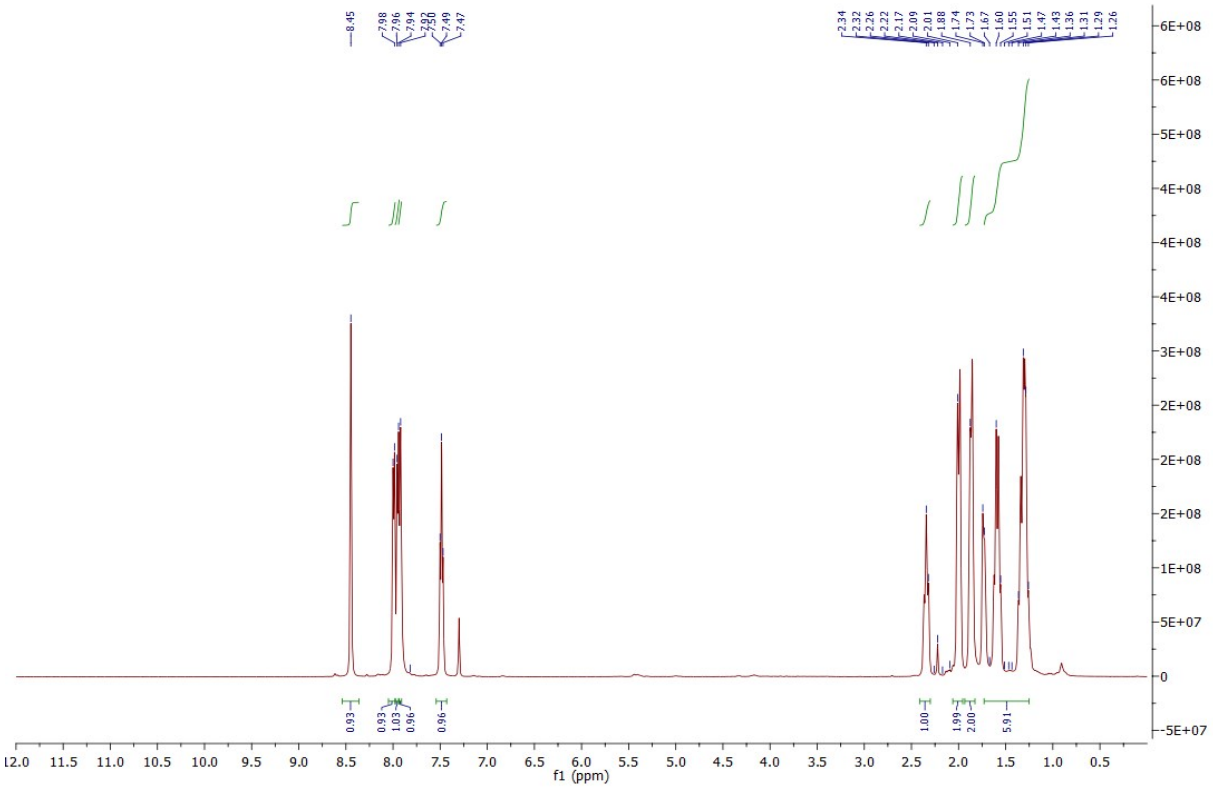

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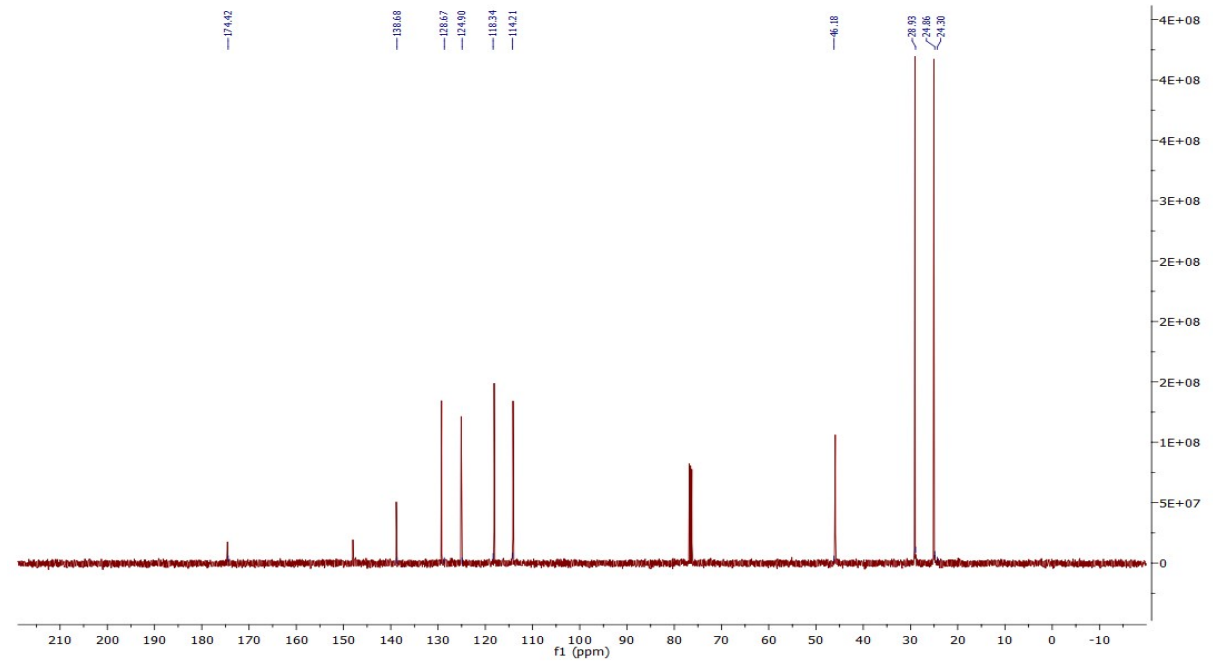

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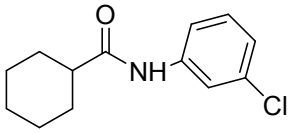
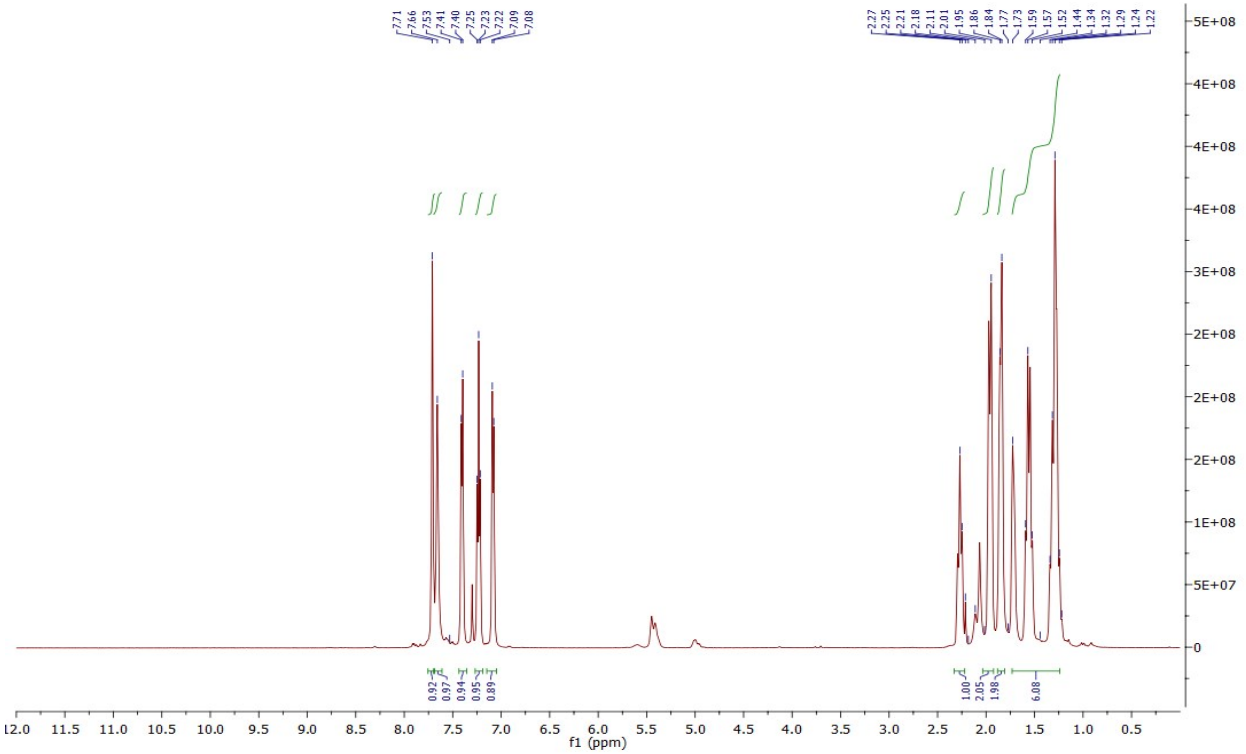
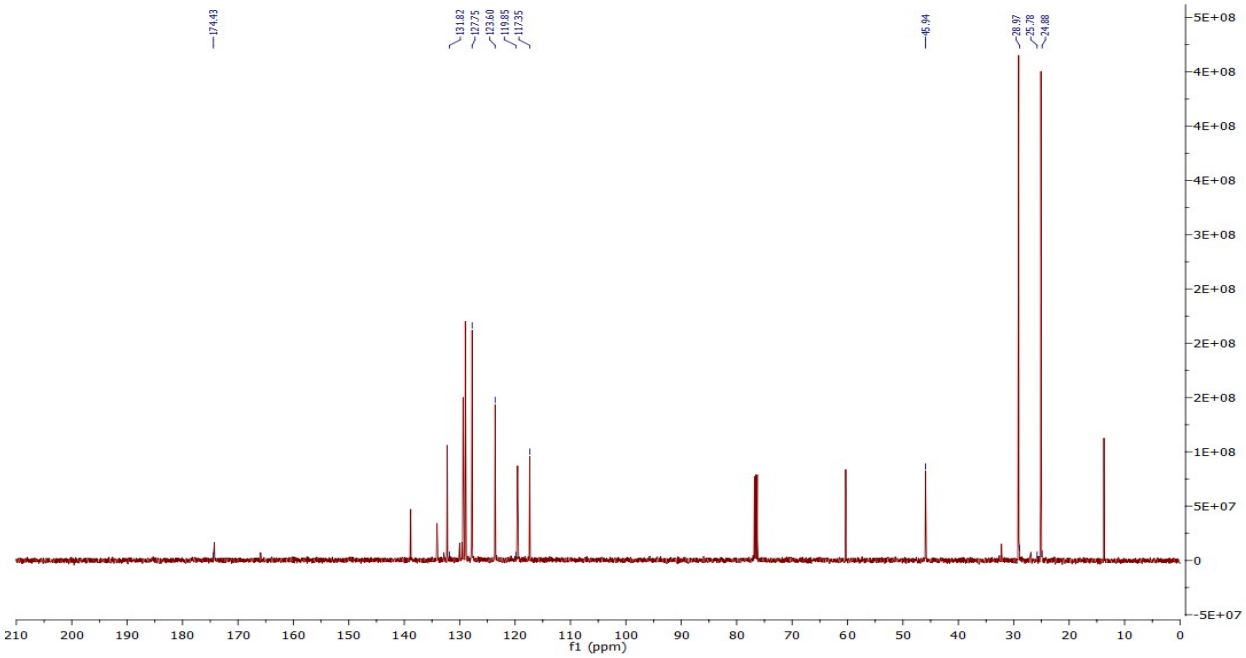
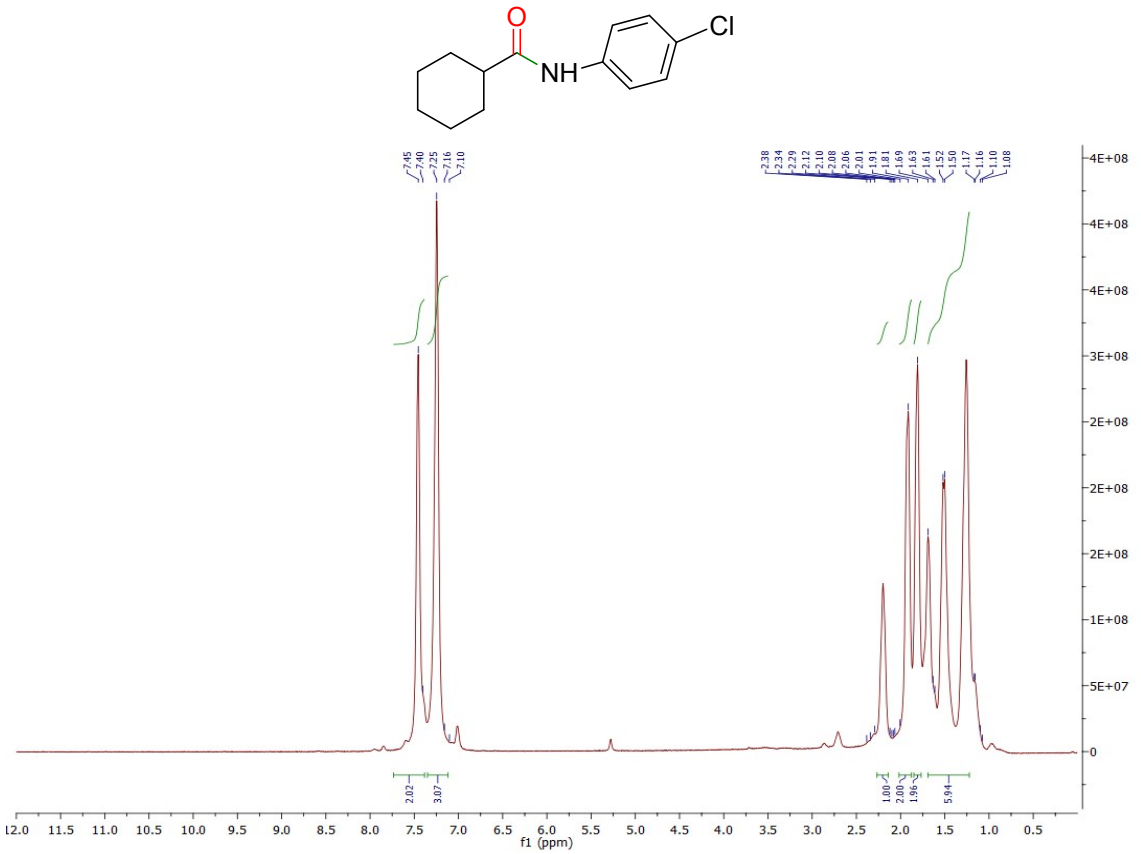




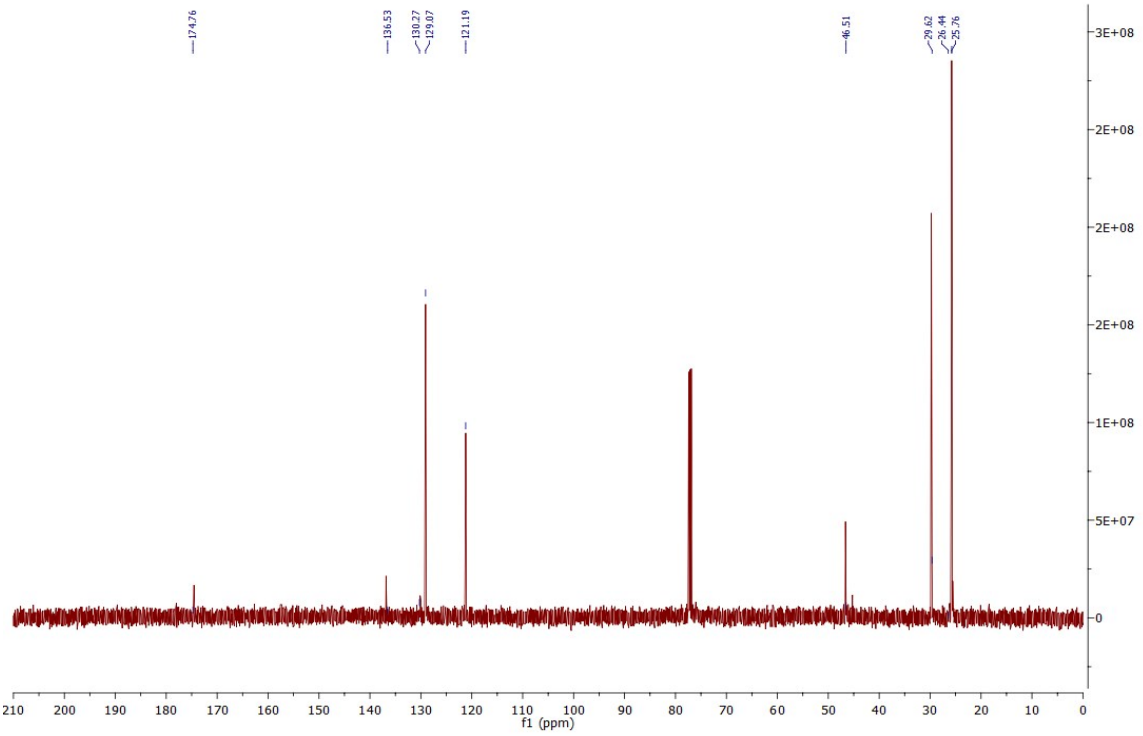
[1]HNMR (4u)



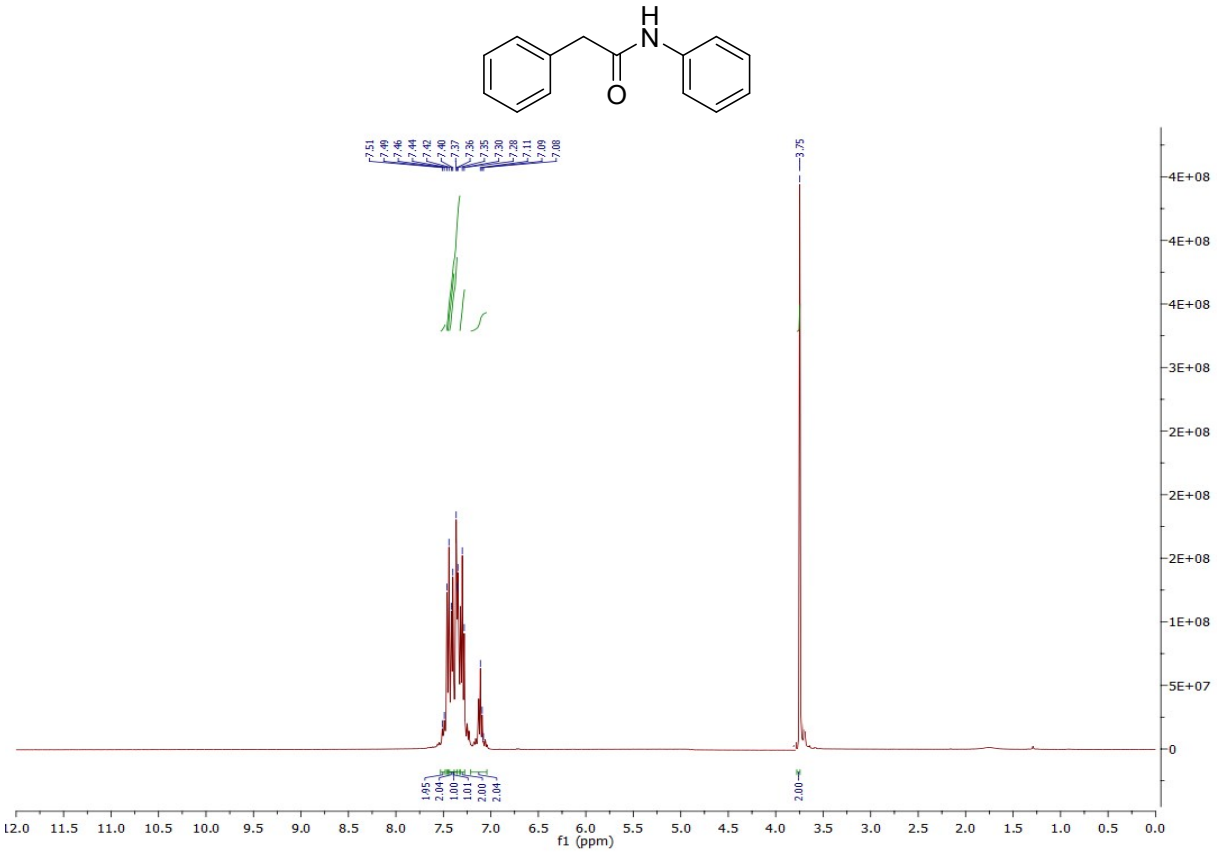
[13]CNMR (4u)



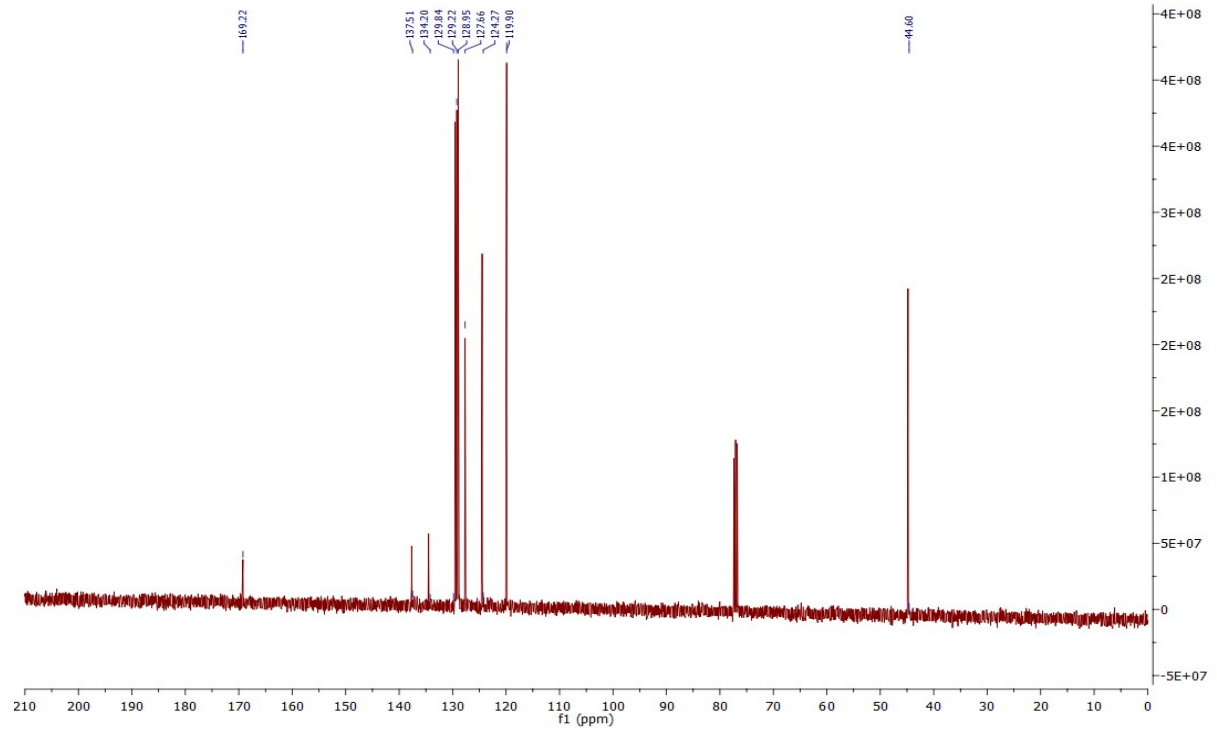
^{1}HNMR (4v)



^{13}CNMR (4v)



[1]HNMR (4w)



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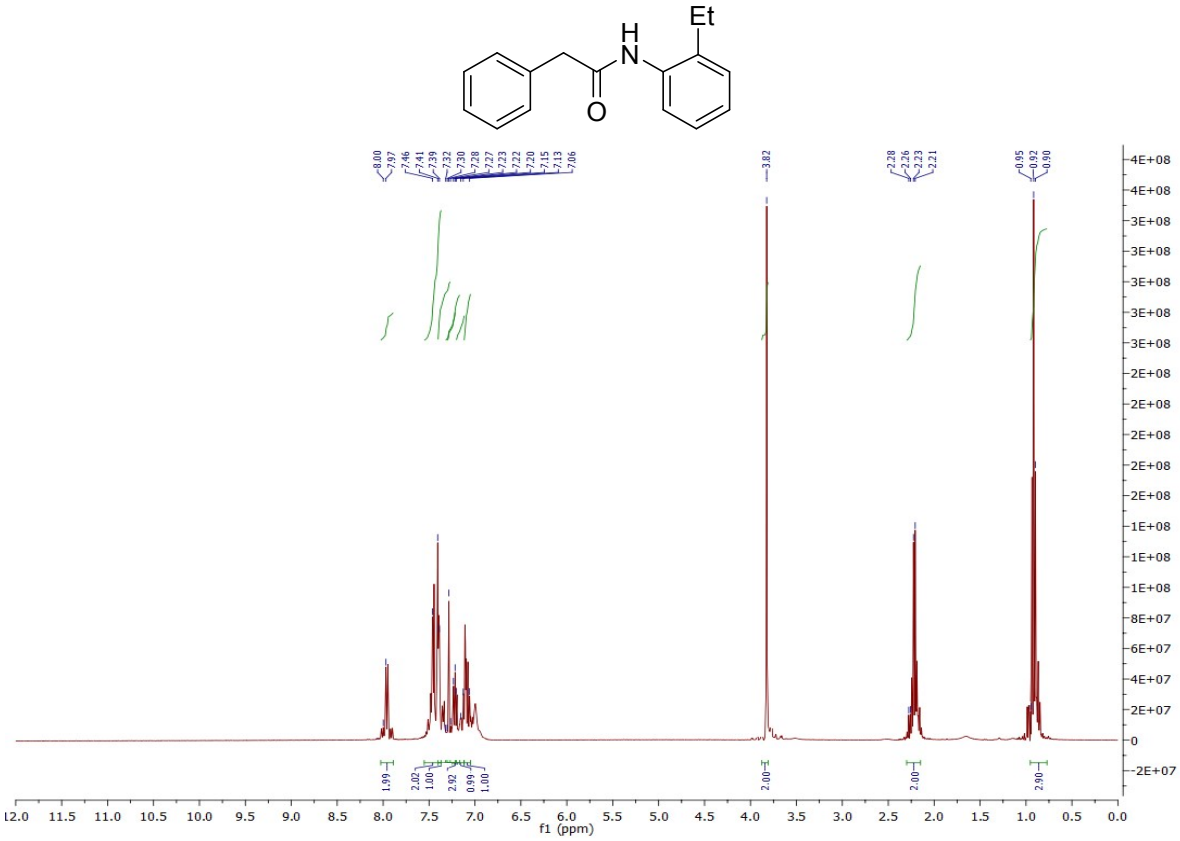

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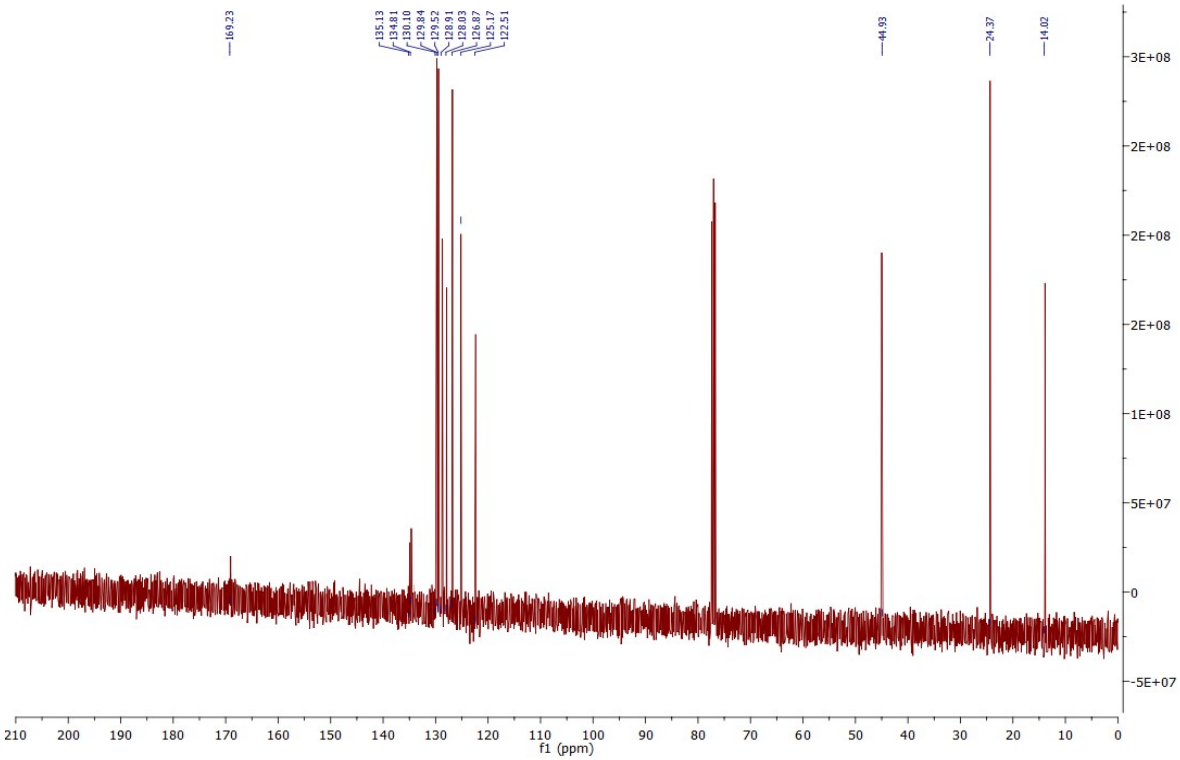
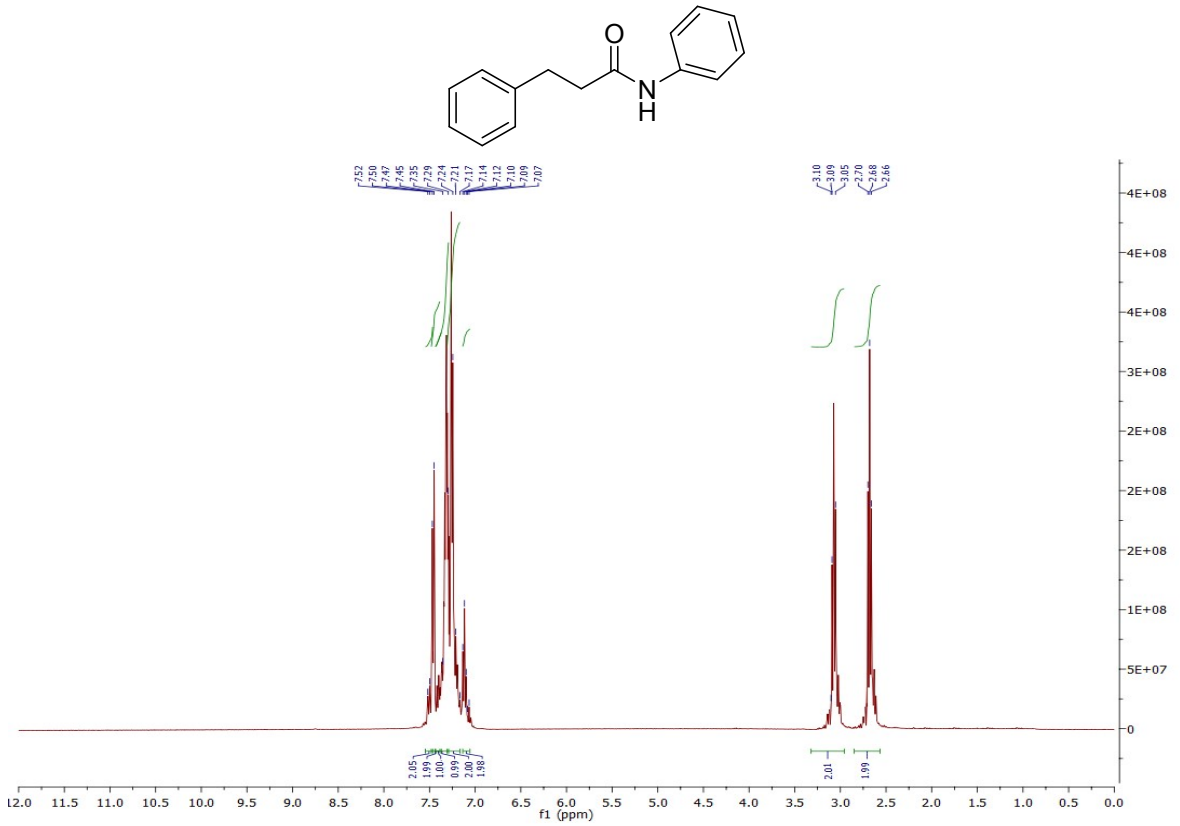


^{13}CNMR (4x)



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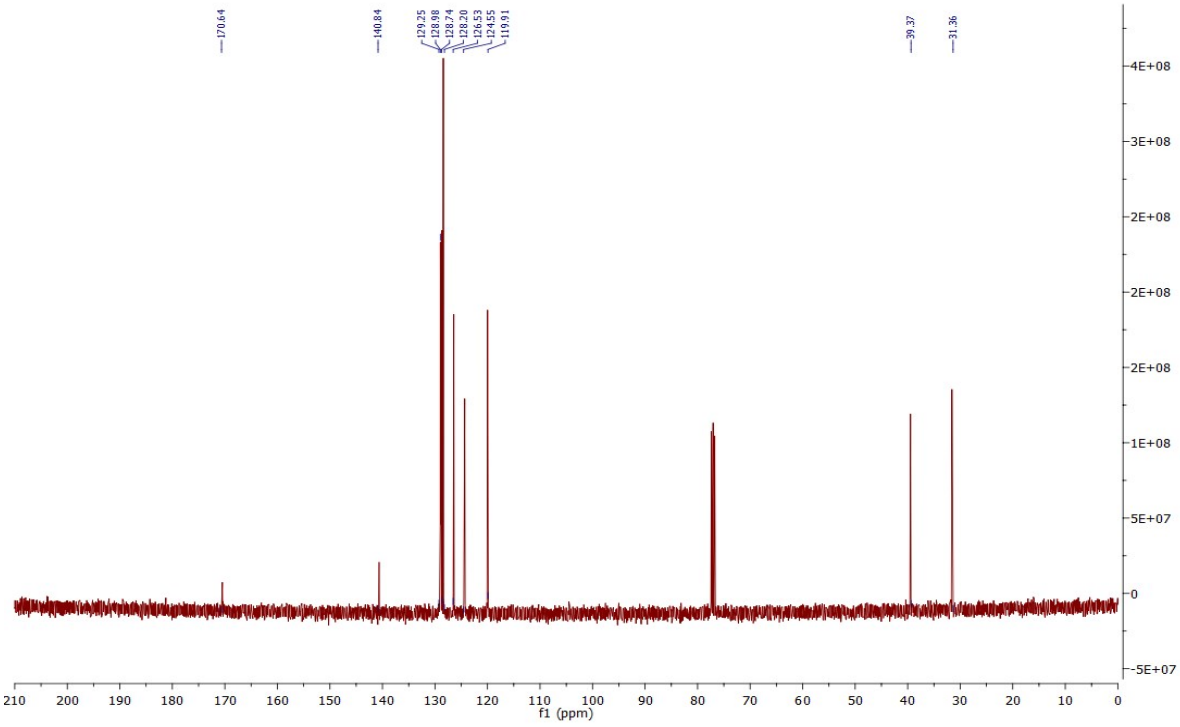

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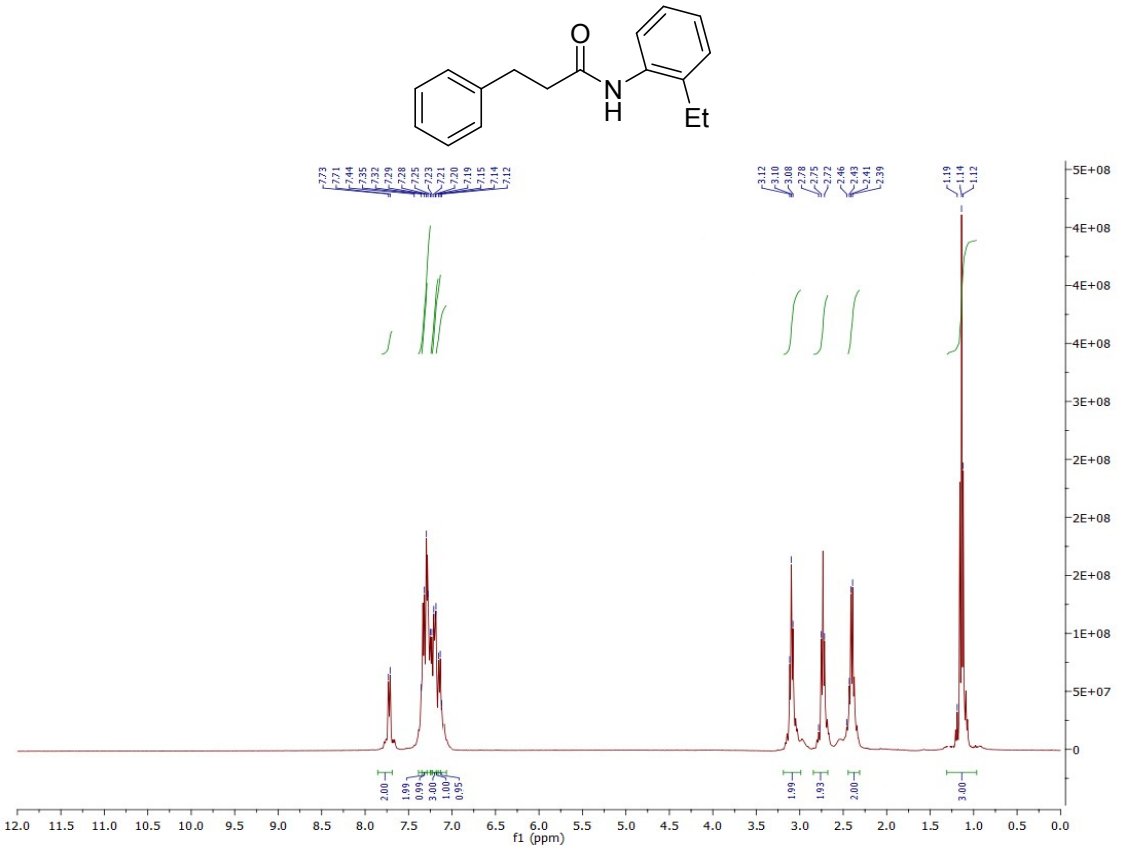

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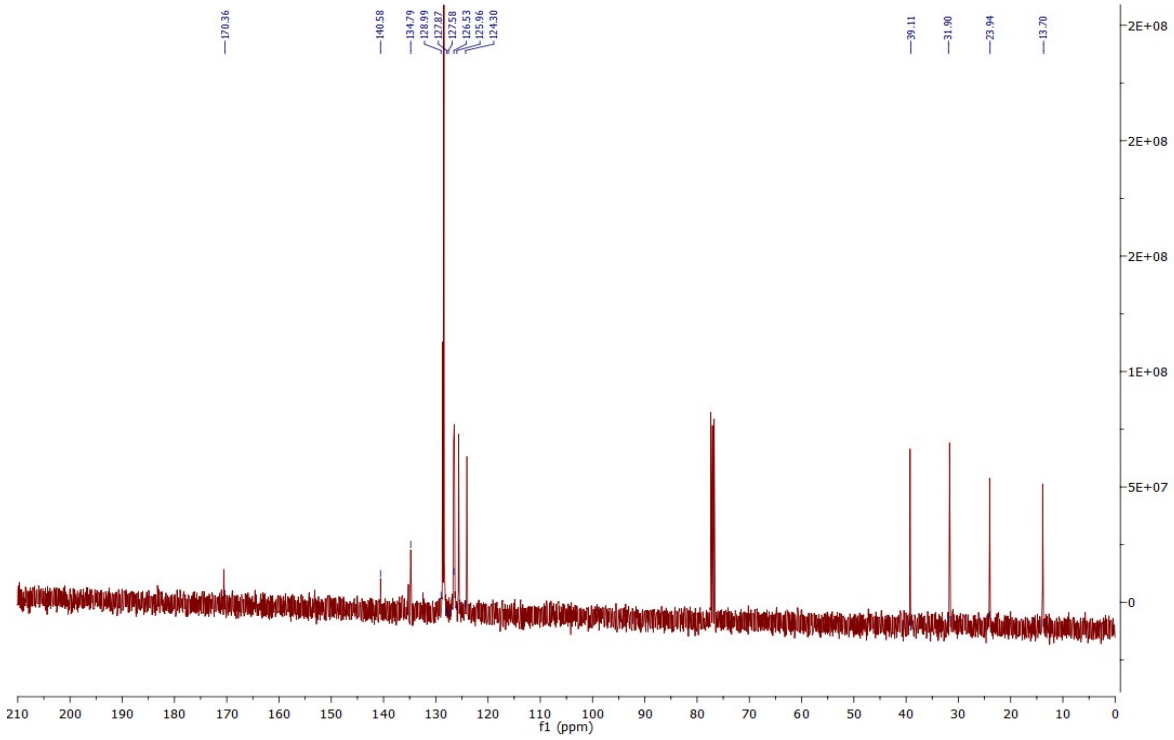

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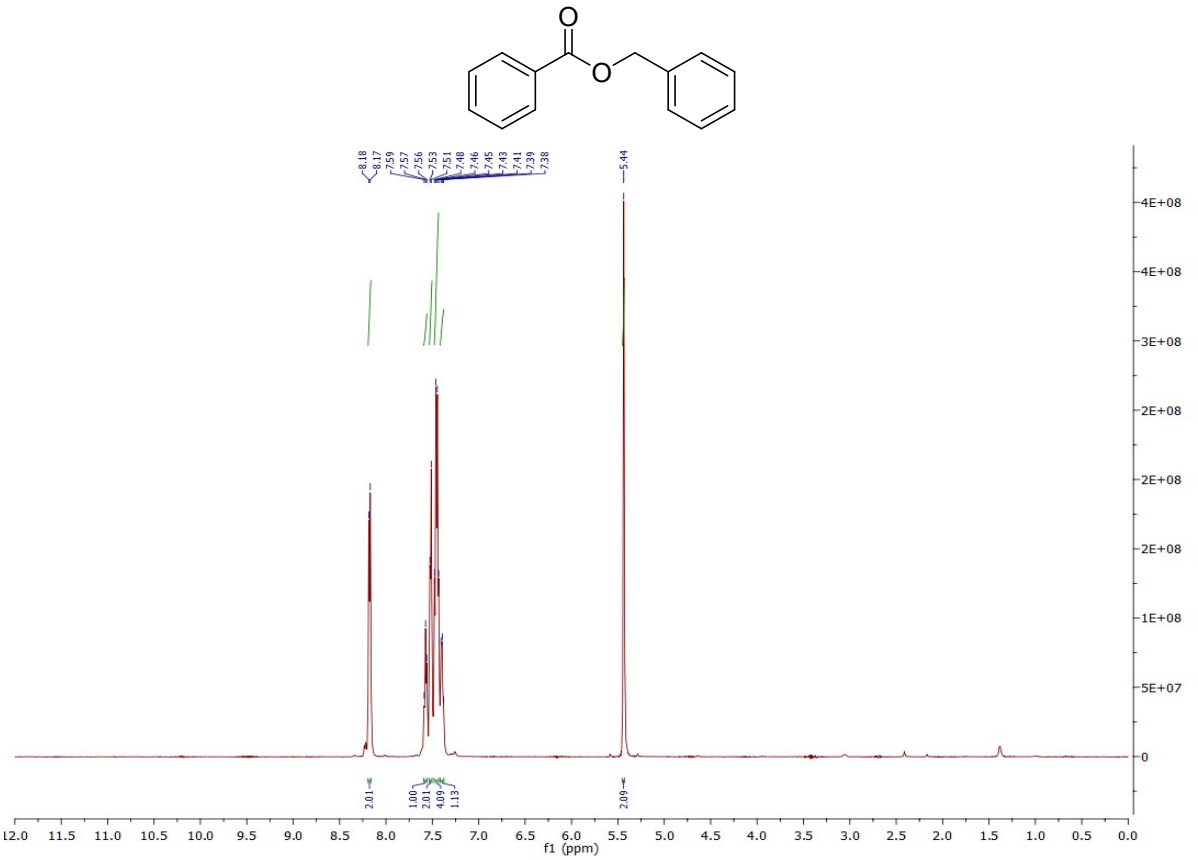
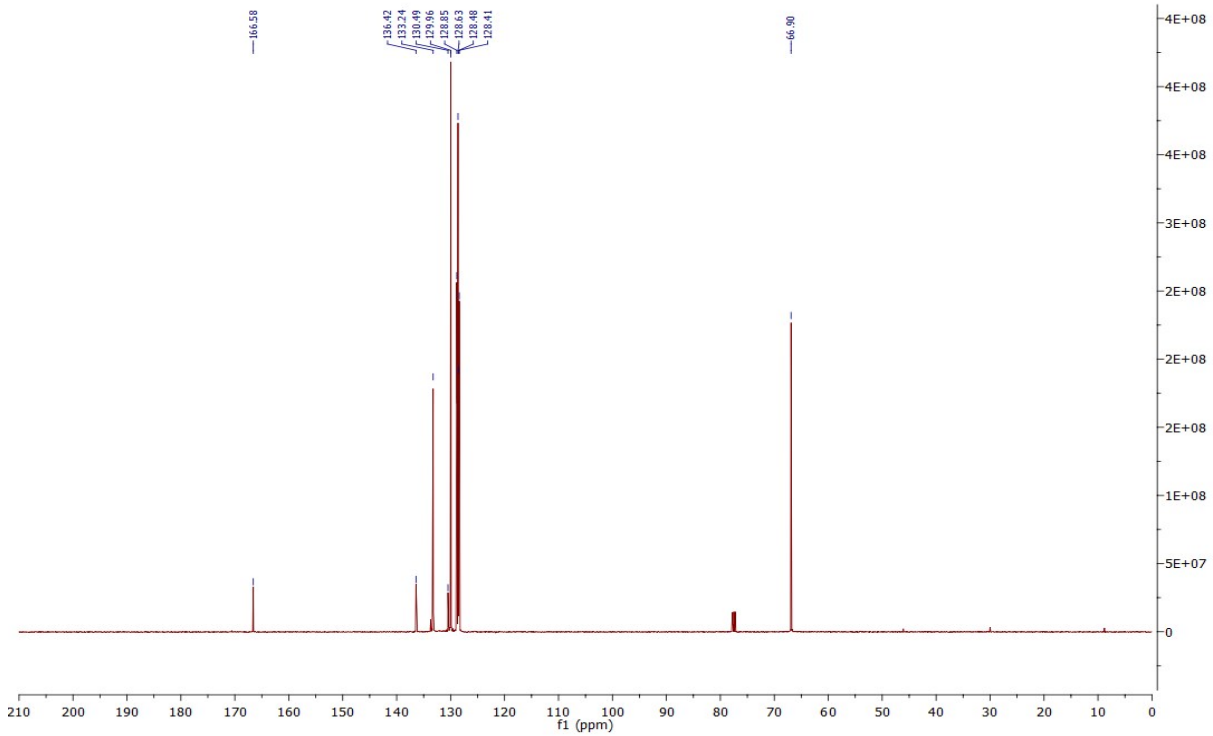


^{1}HNMR (6a)



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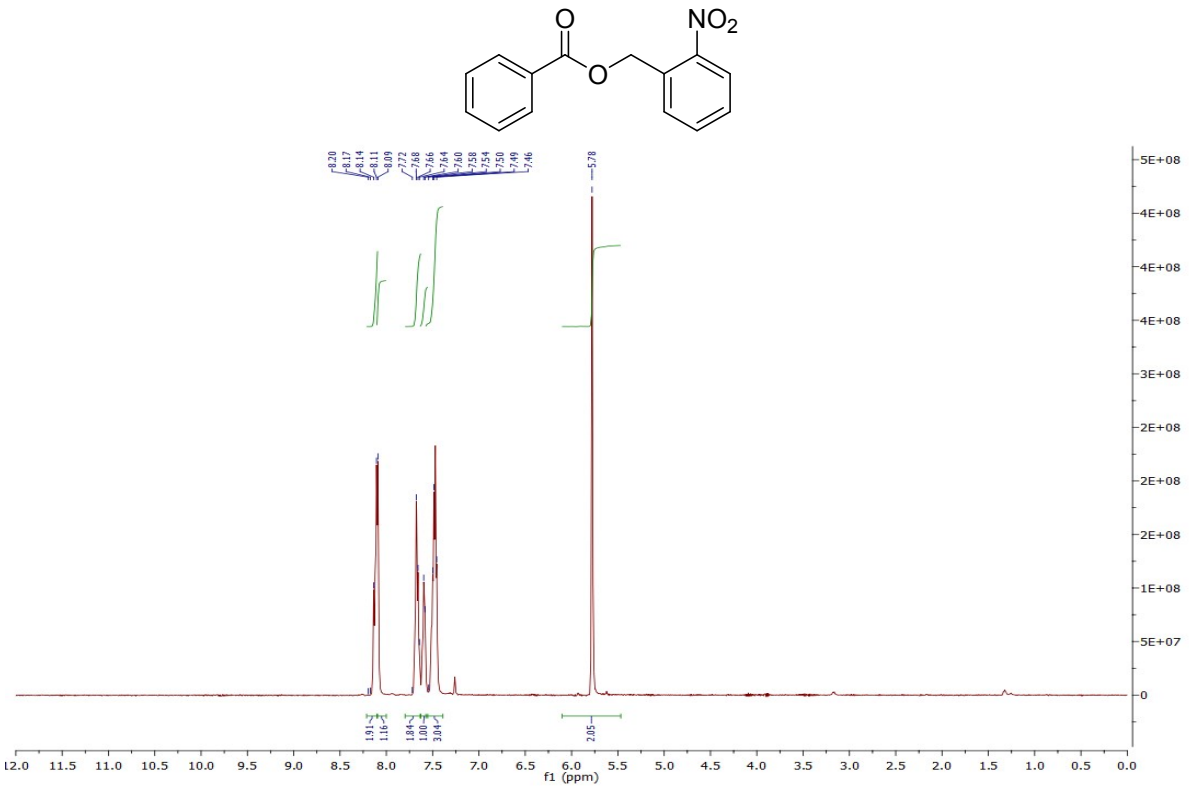
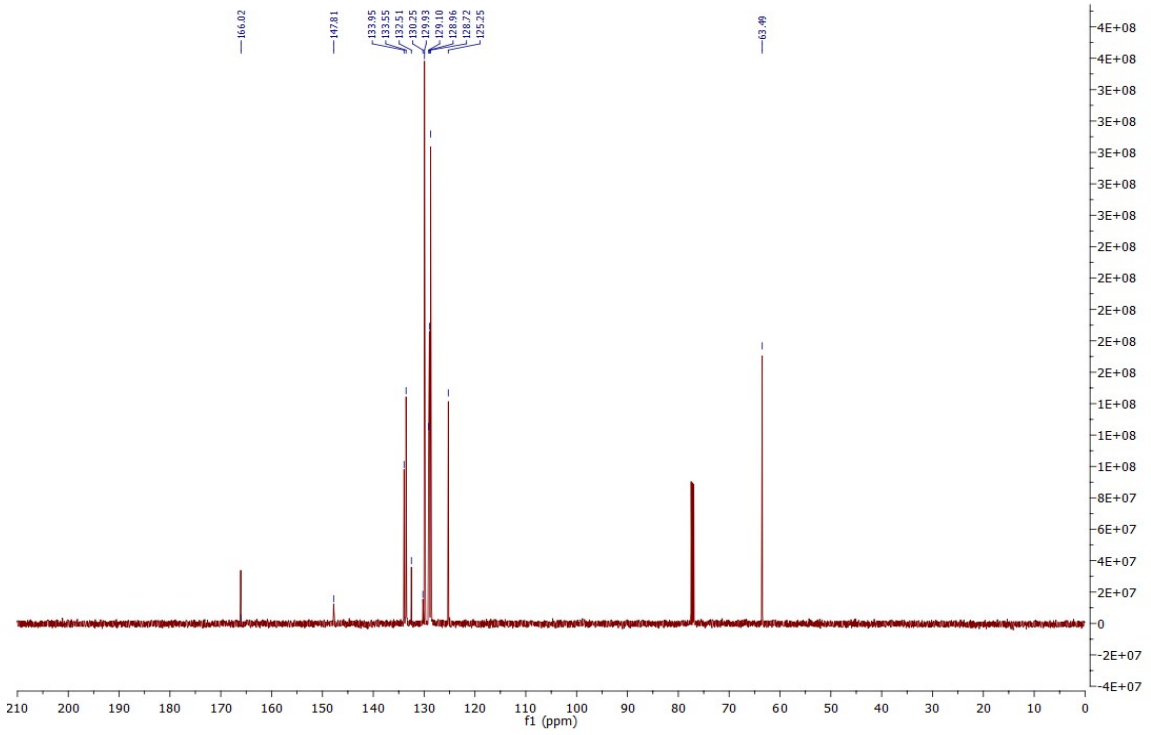
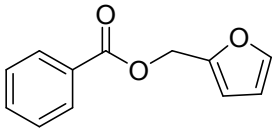
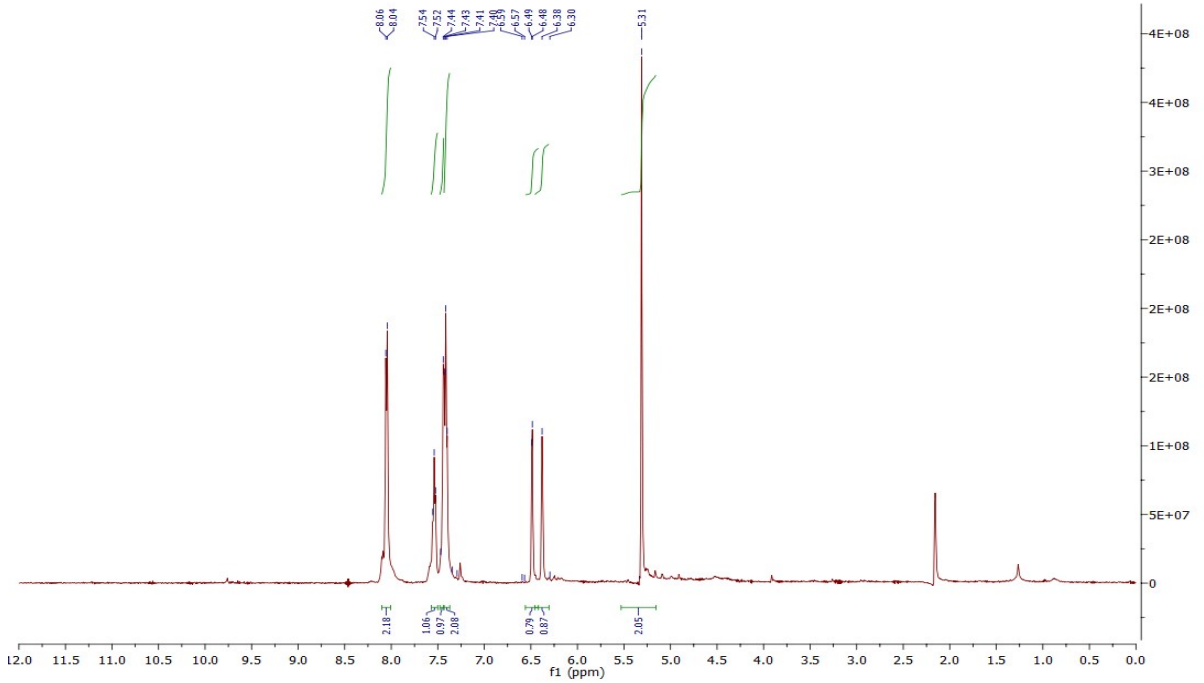


[1]HNMR (6b)

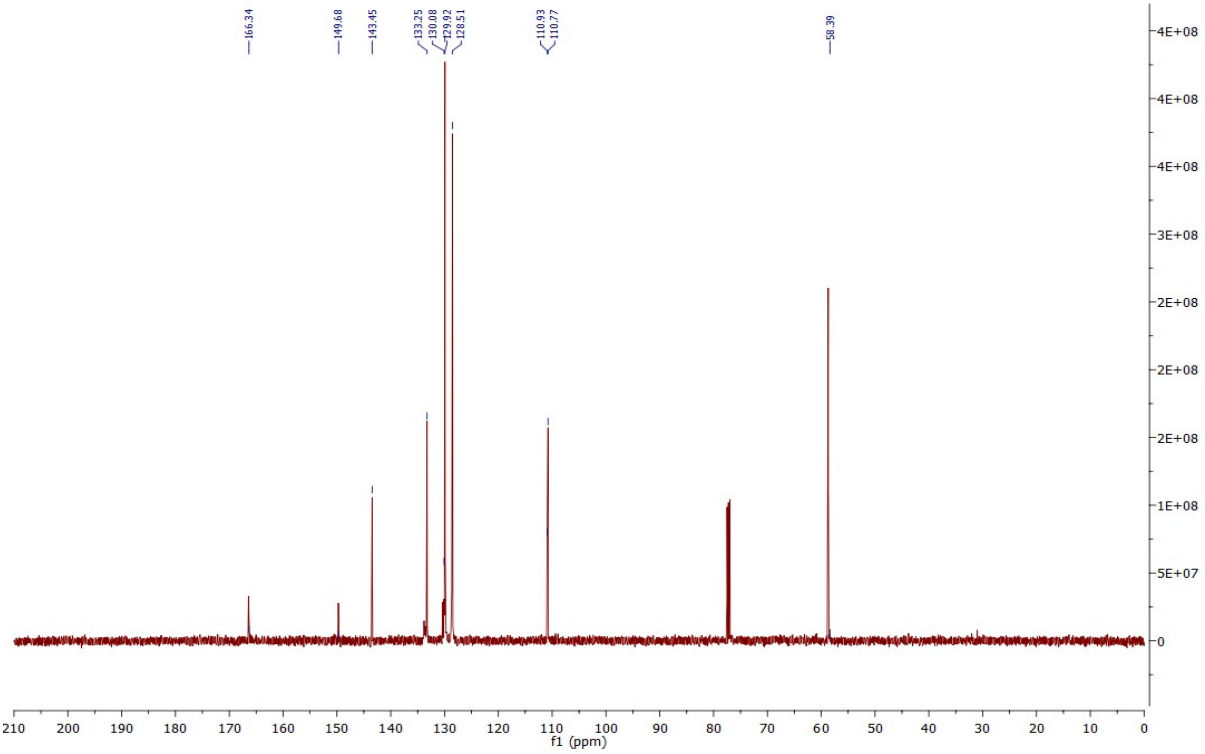


[13]CNMR (6b)





^{1}HNMR (6c)



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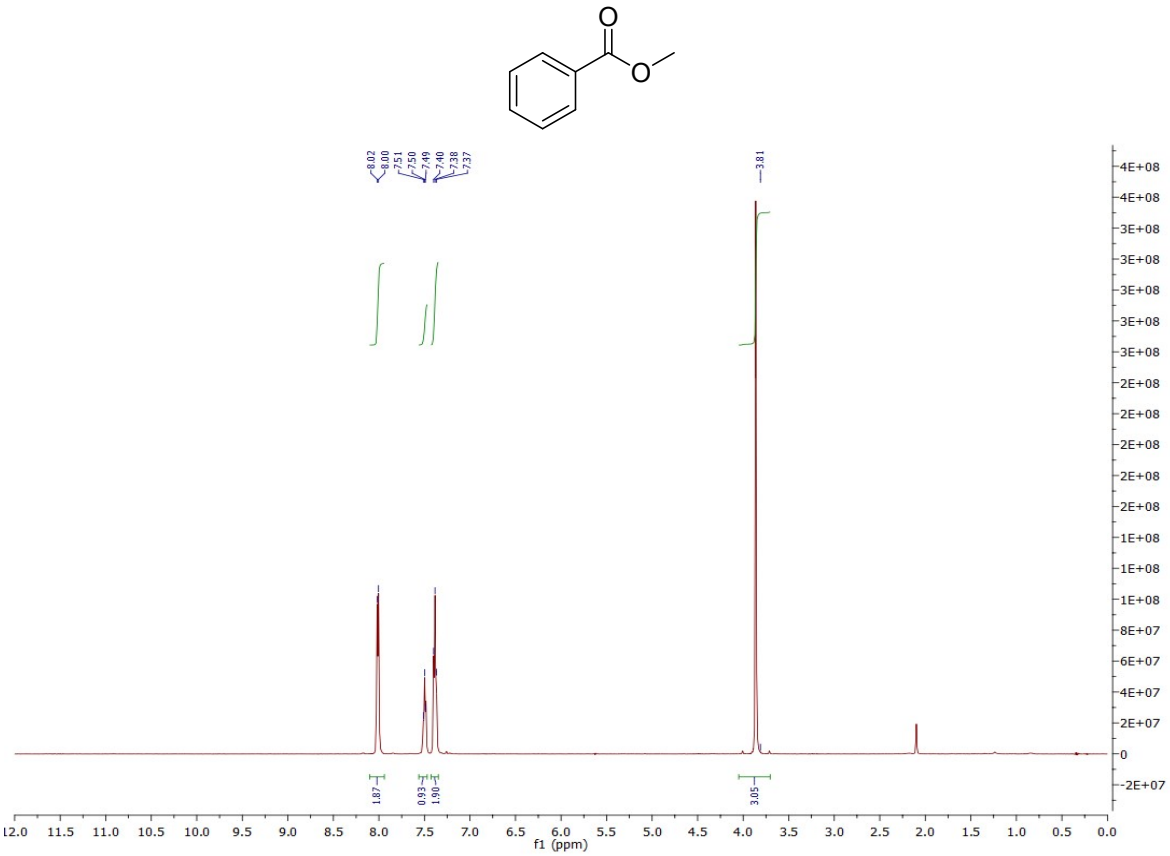
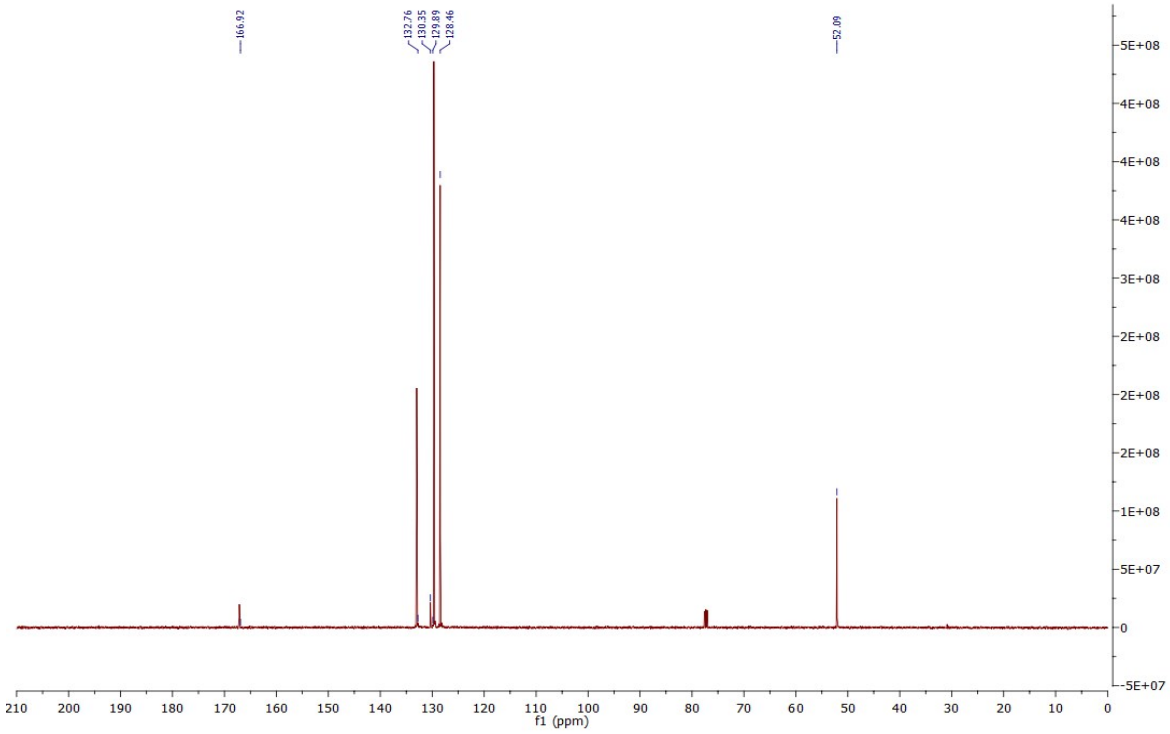
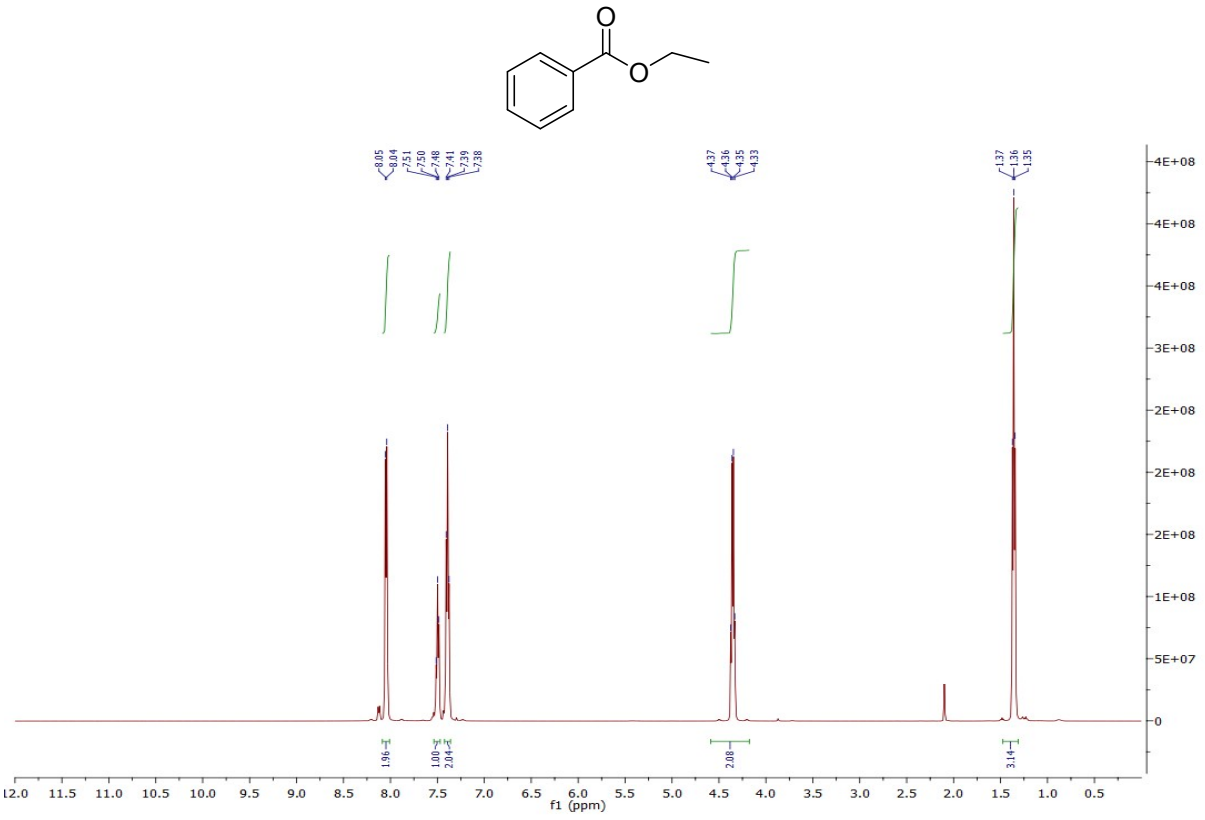


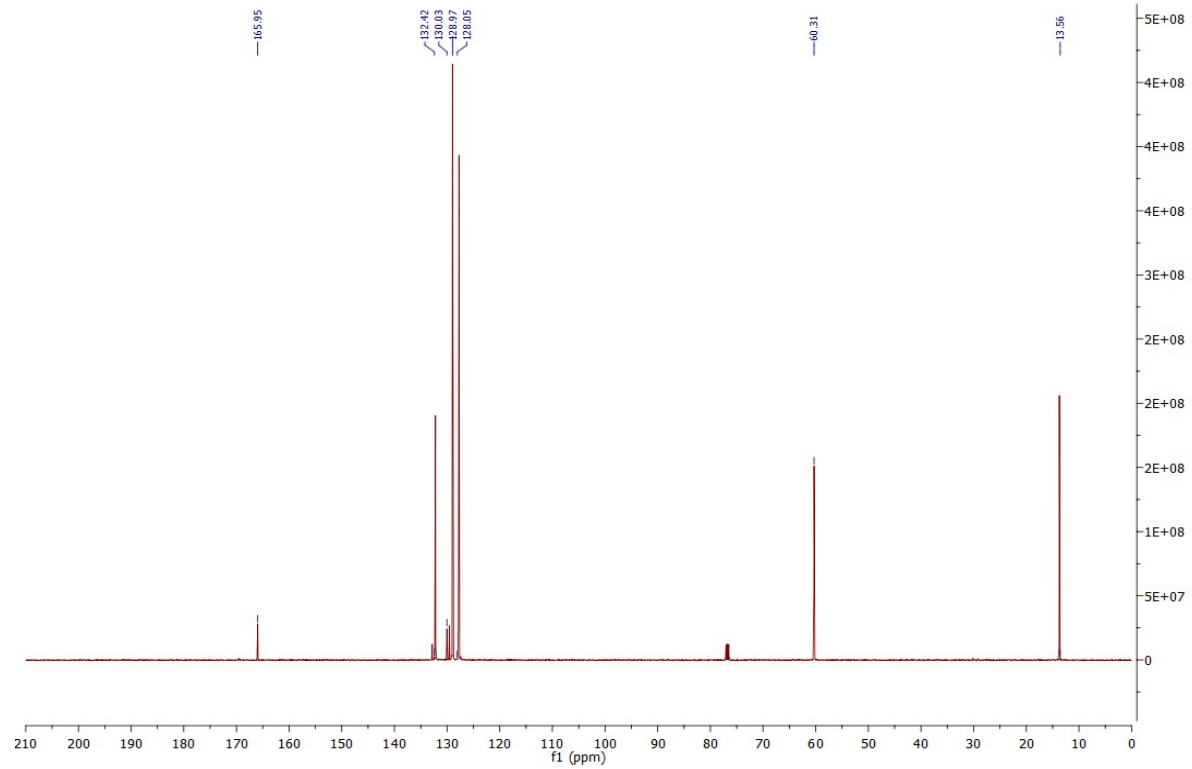
^{1}HNMR (6d)



^{13}CNMR (6d)

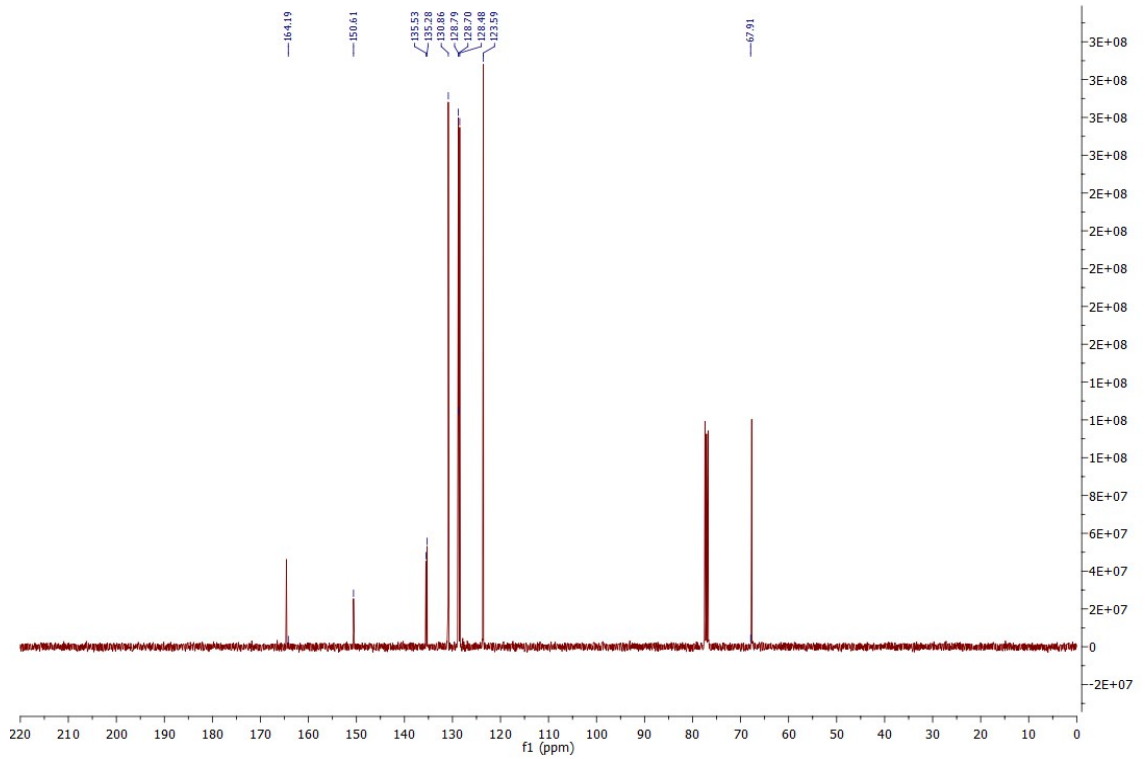


^{1}HNMR (6e)



^{13}CNMR (6e)

^{1}HNMR (6f)



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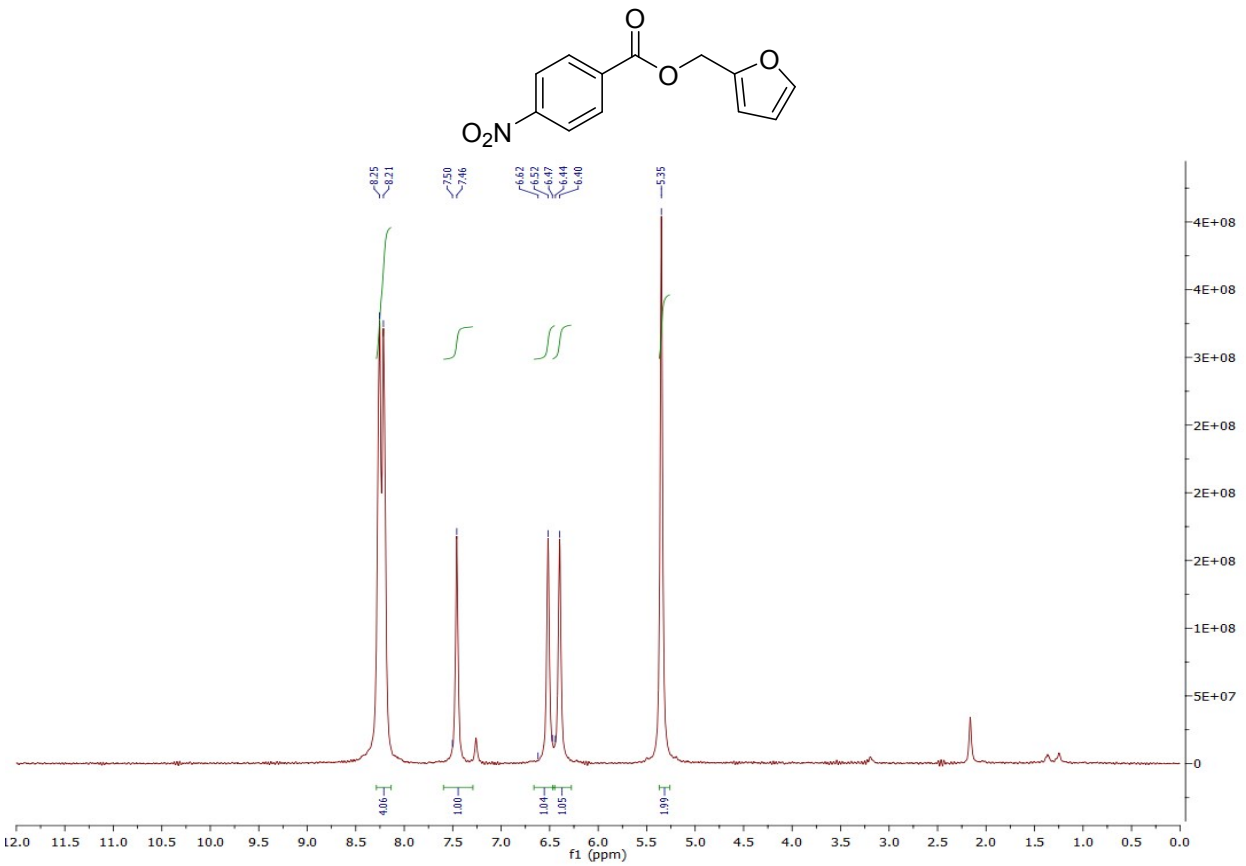
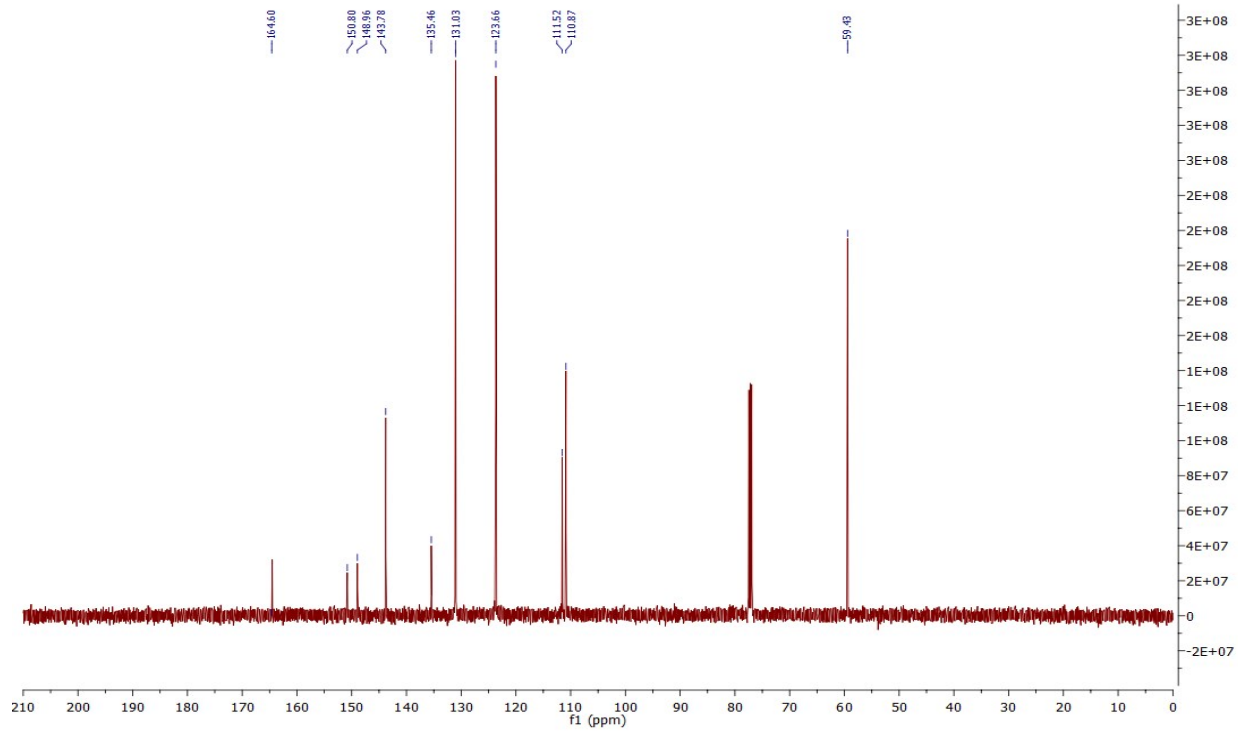
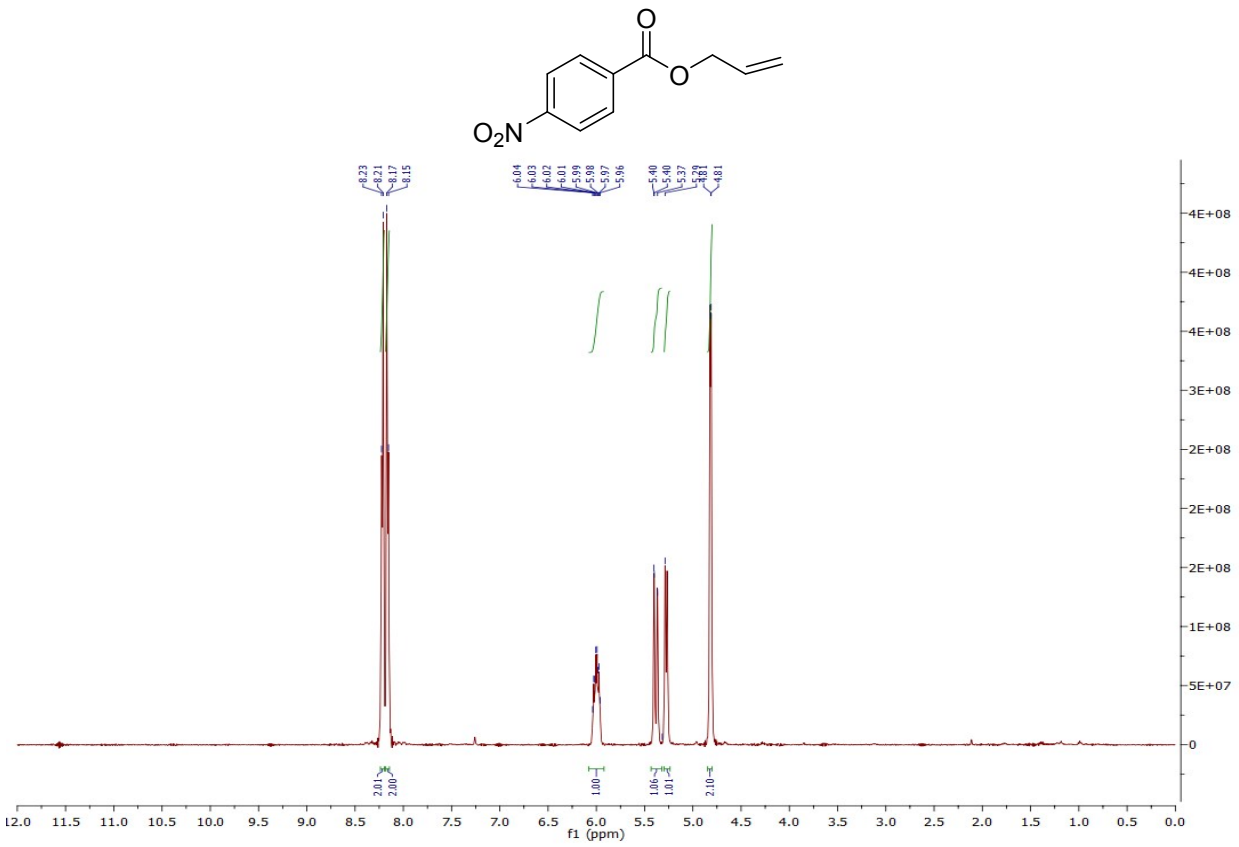


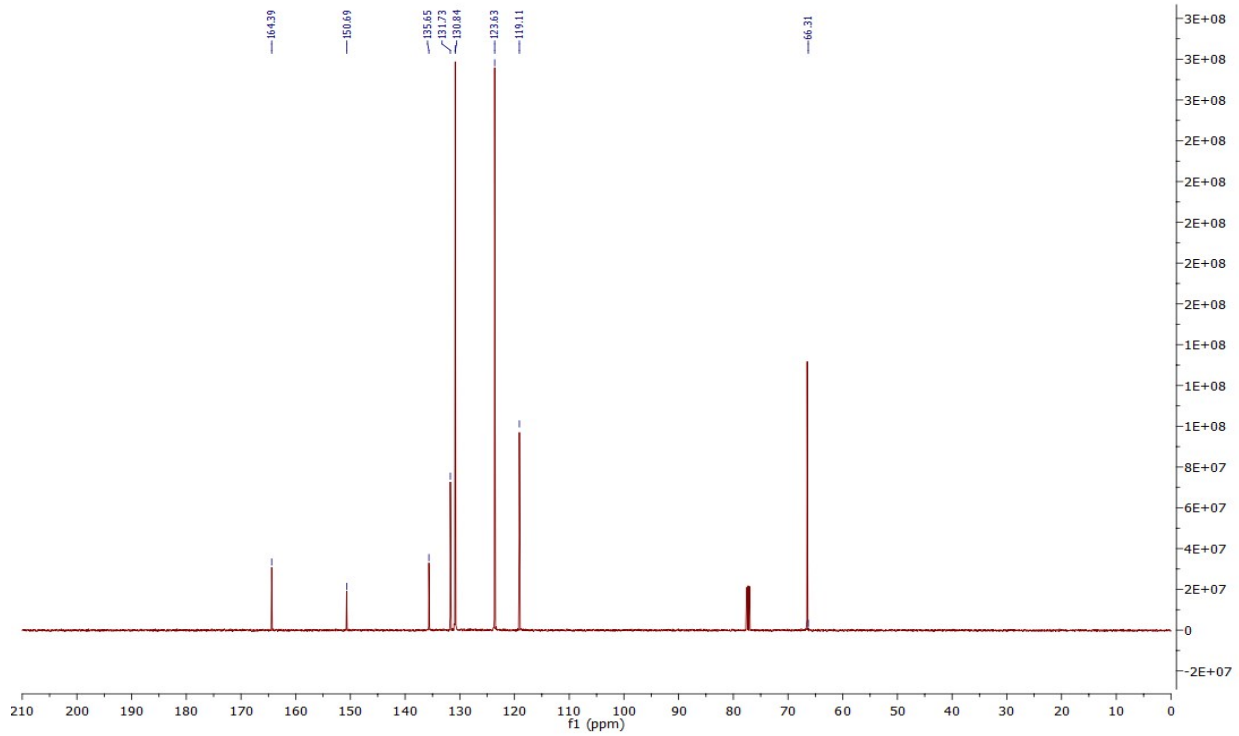
[1]HNMR (6g)



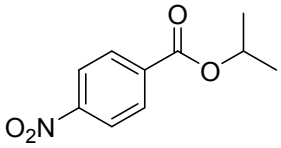
[13]CNMR (6g)

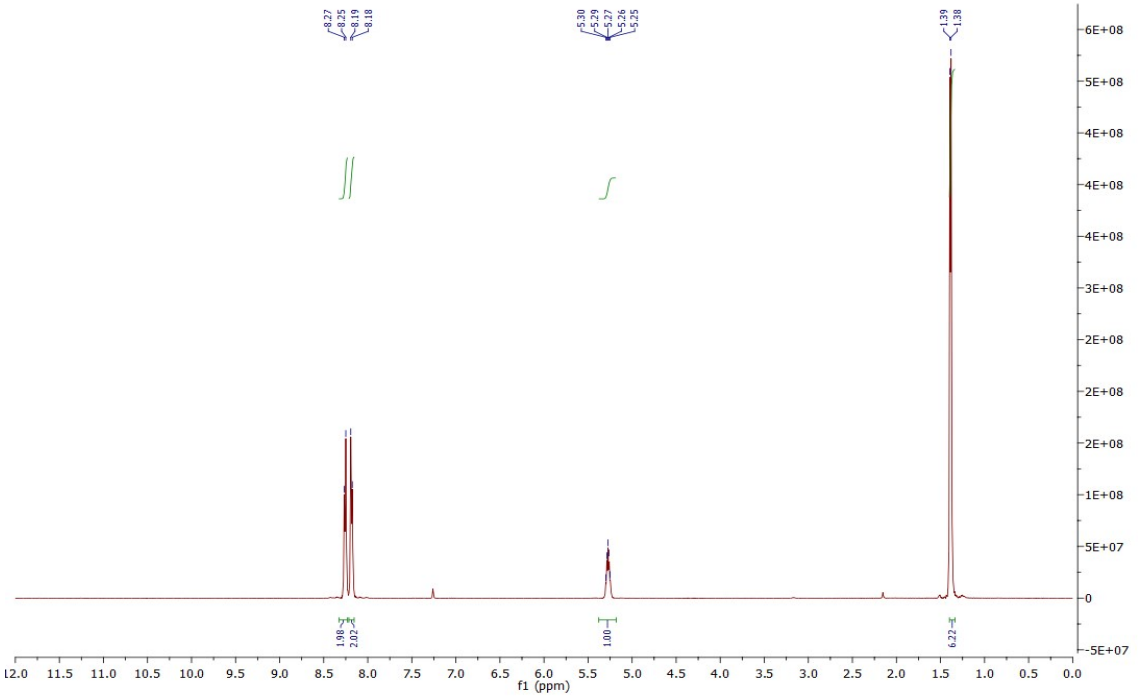


[1]HNMR (6h)

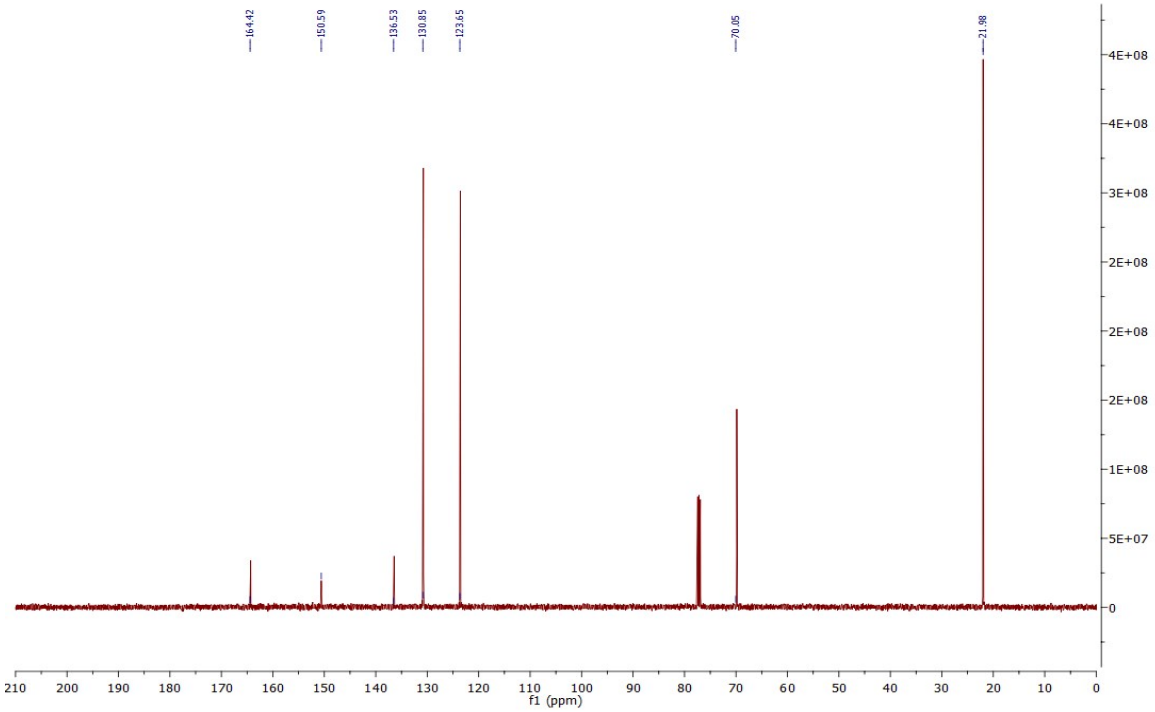


[13]CNMR (6h)





[1]HNMR (6i)



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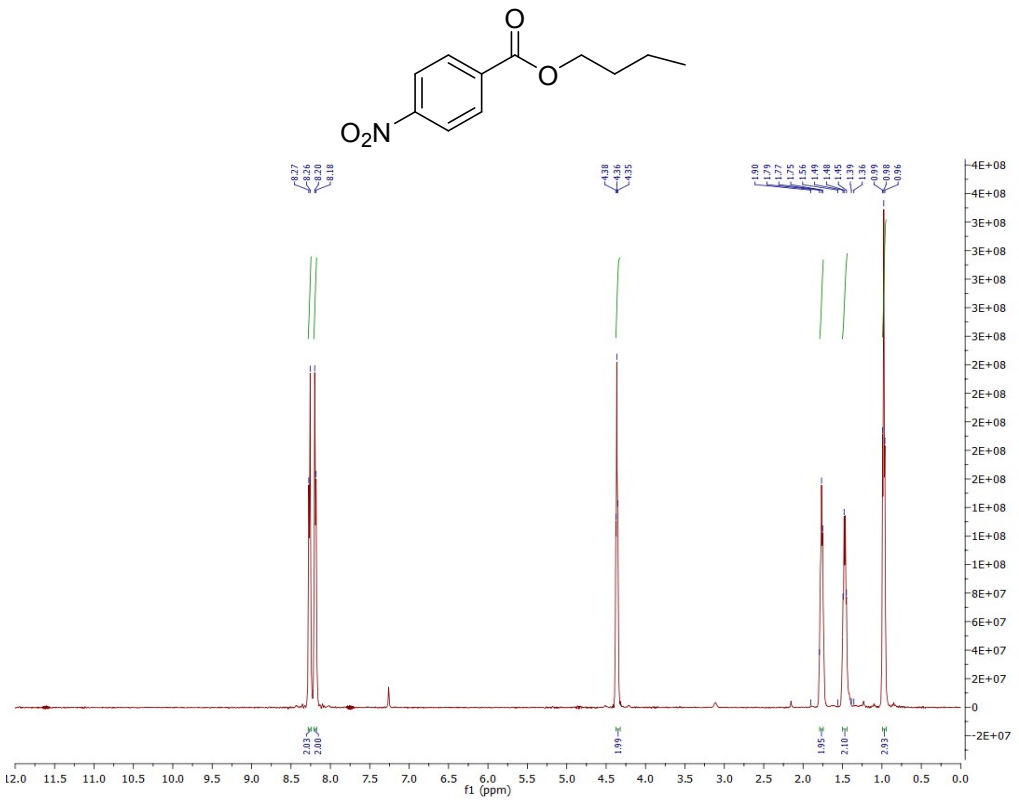
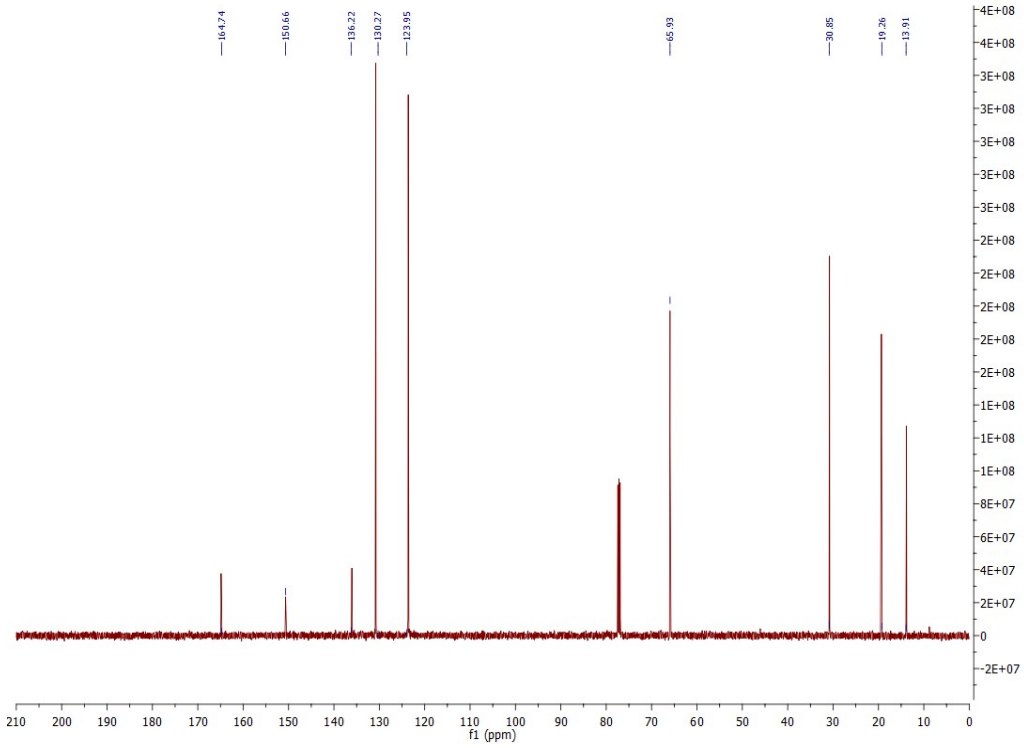


[1]HNMR (6j)



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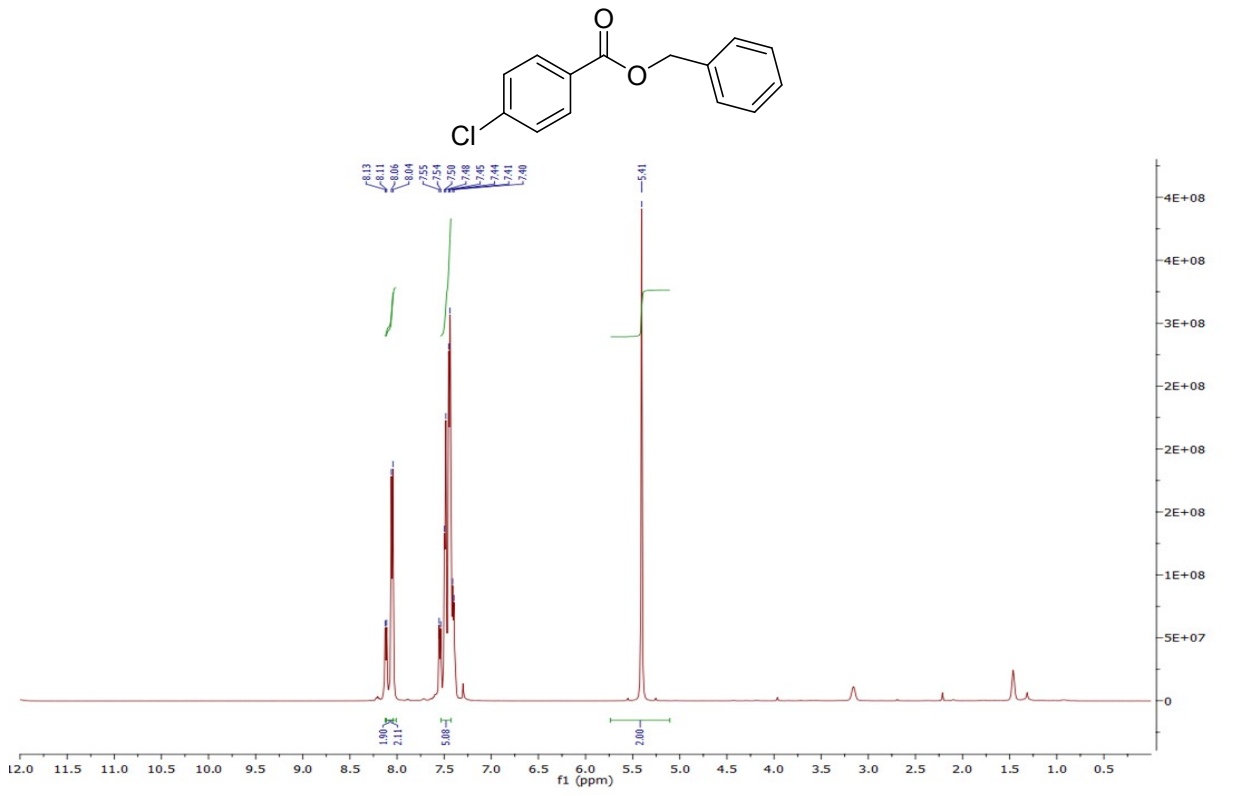
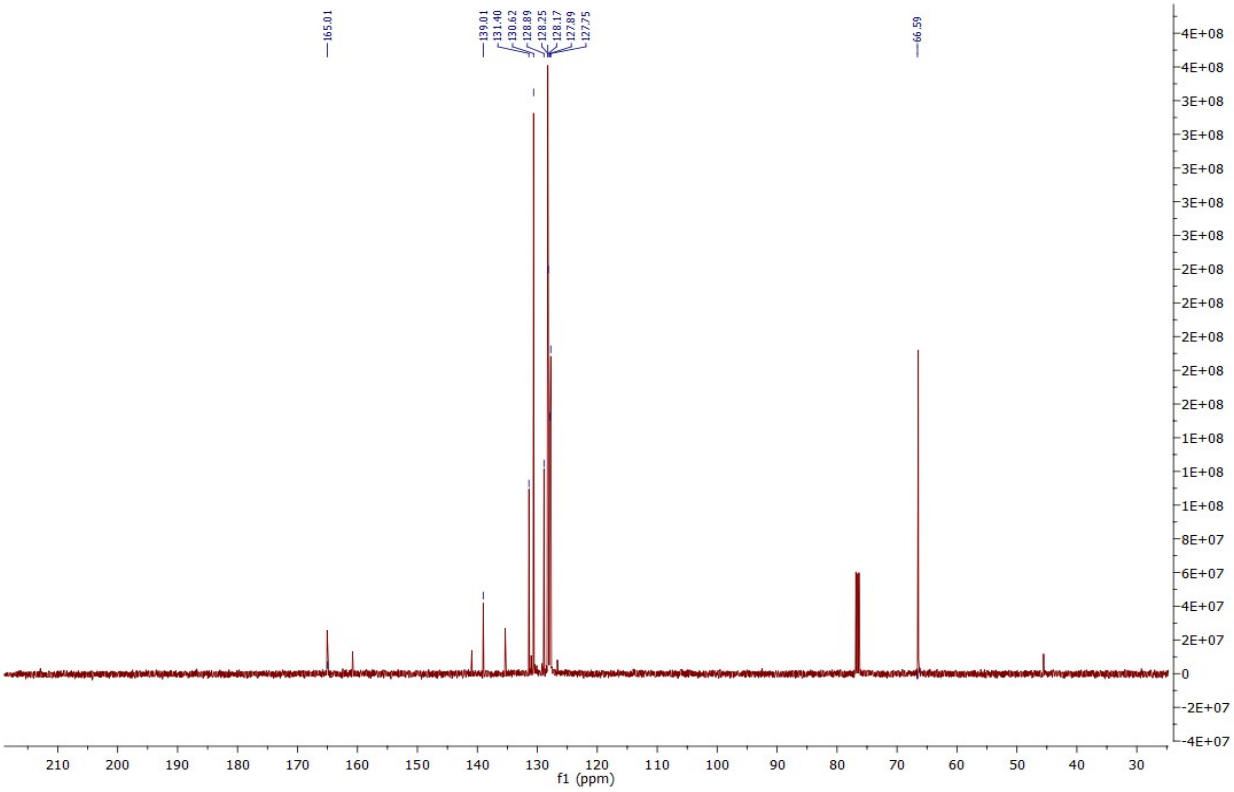
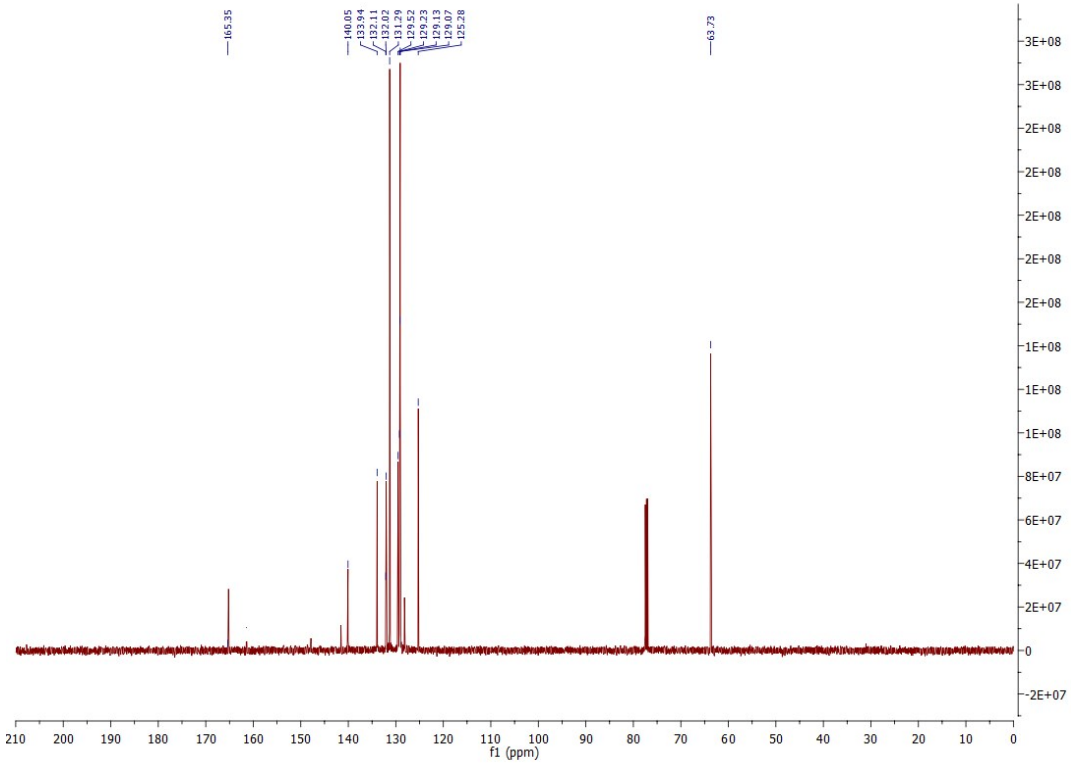


^{1}HNMR (6l)

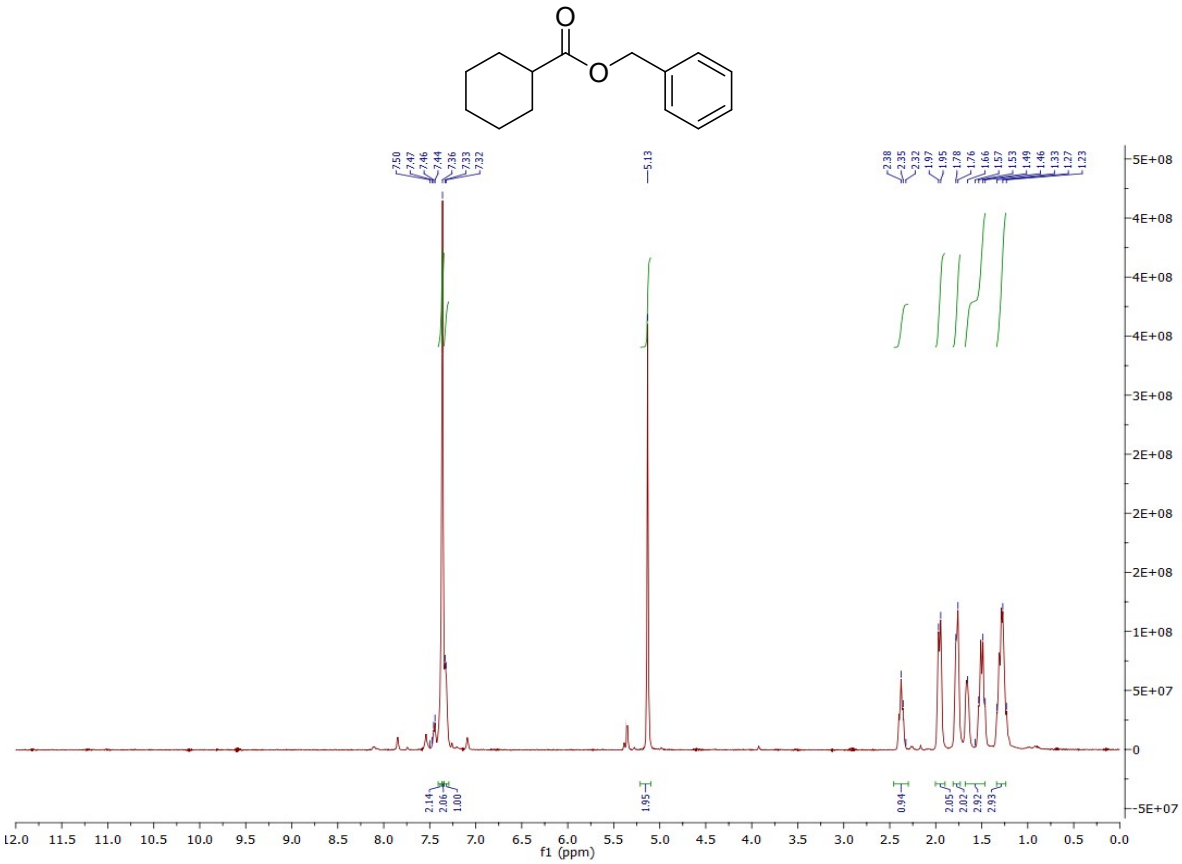


^{13}CNMR (6l)

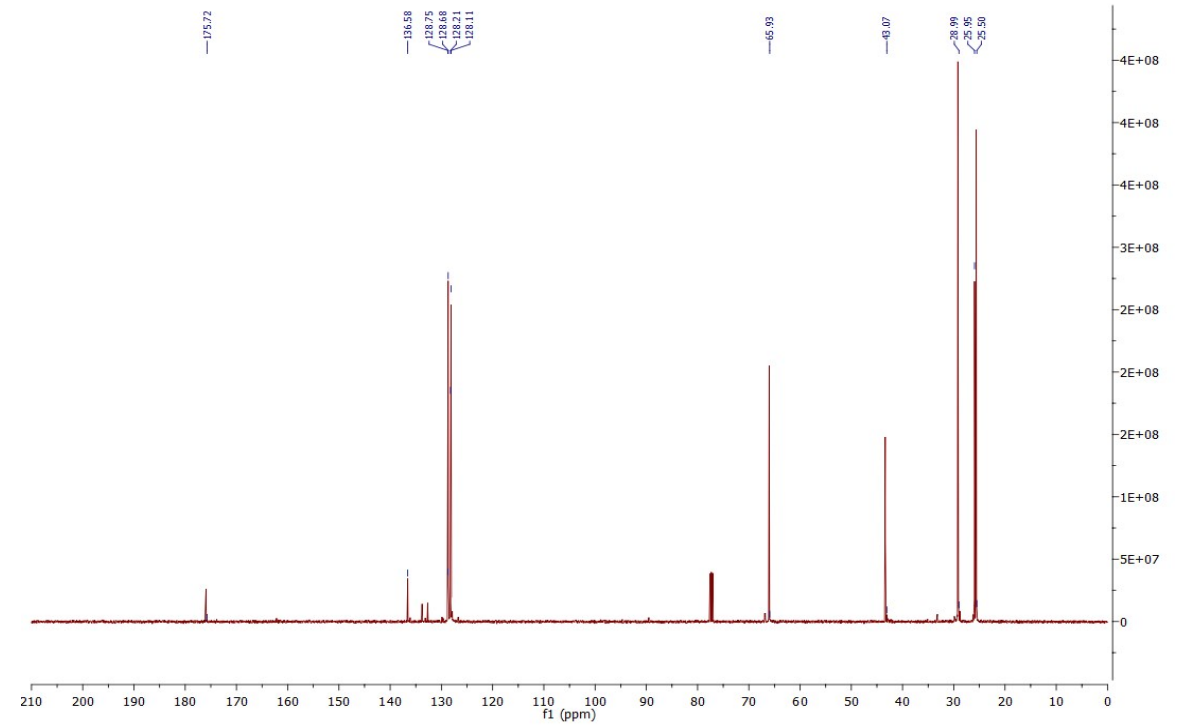
^{1}HNMR (6m)



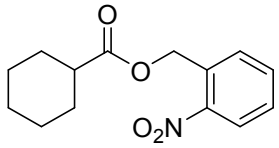
^{13}CNMR (6m)

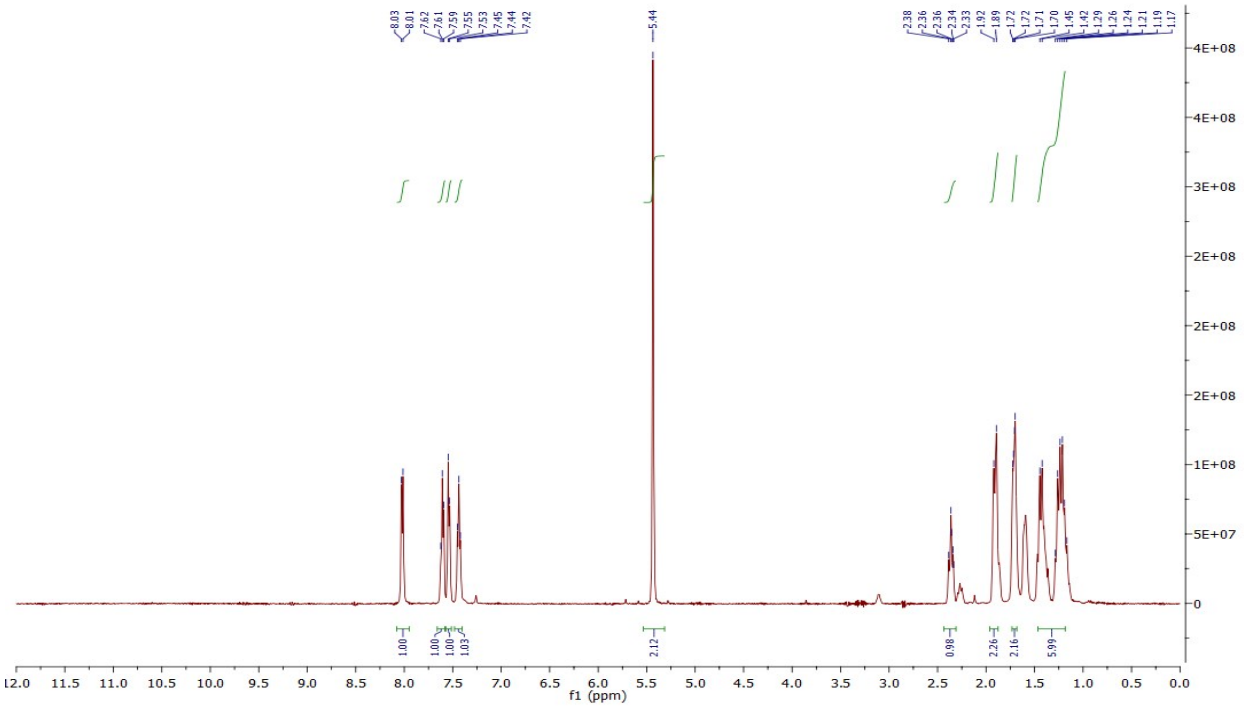


[1]HNMR (6n)

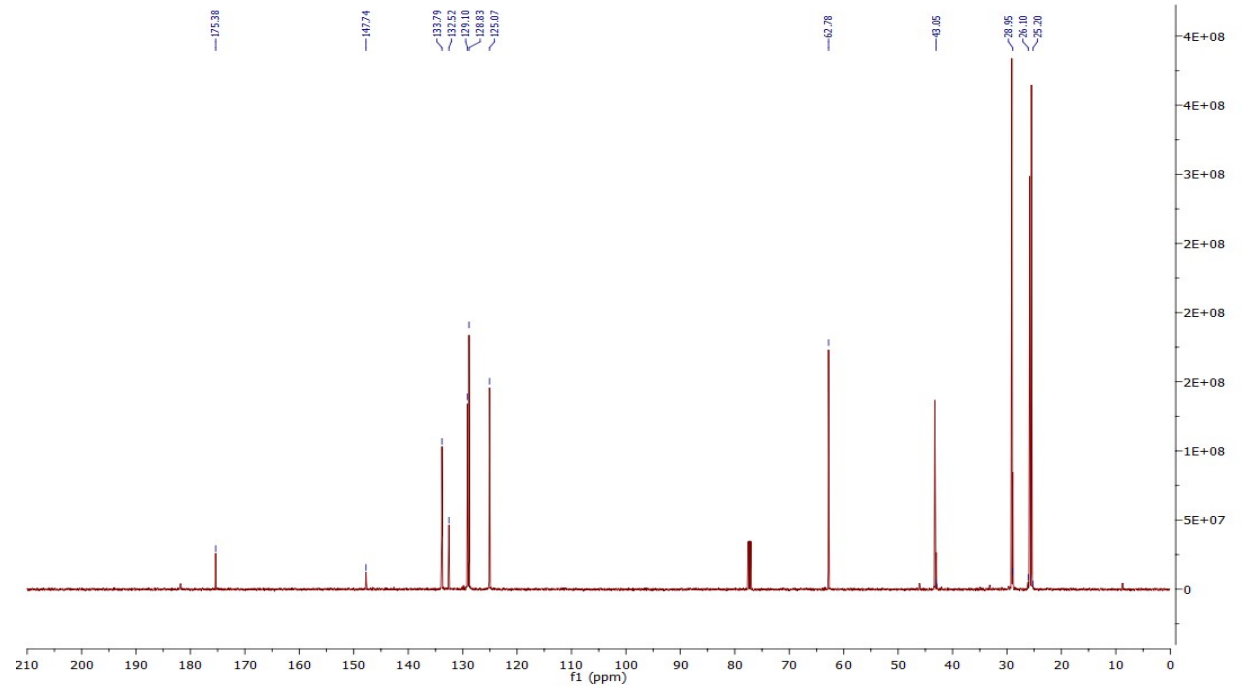


[13]CNMR (6n)

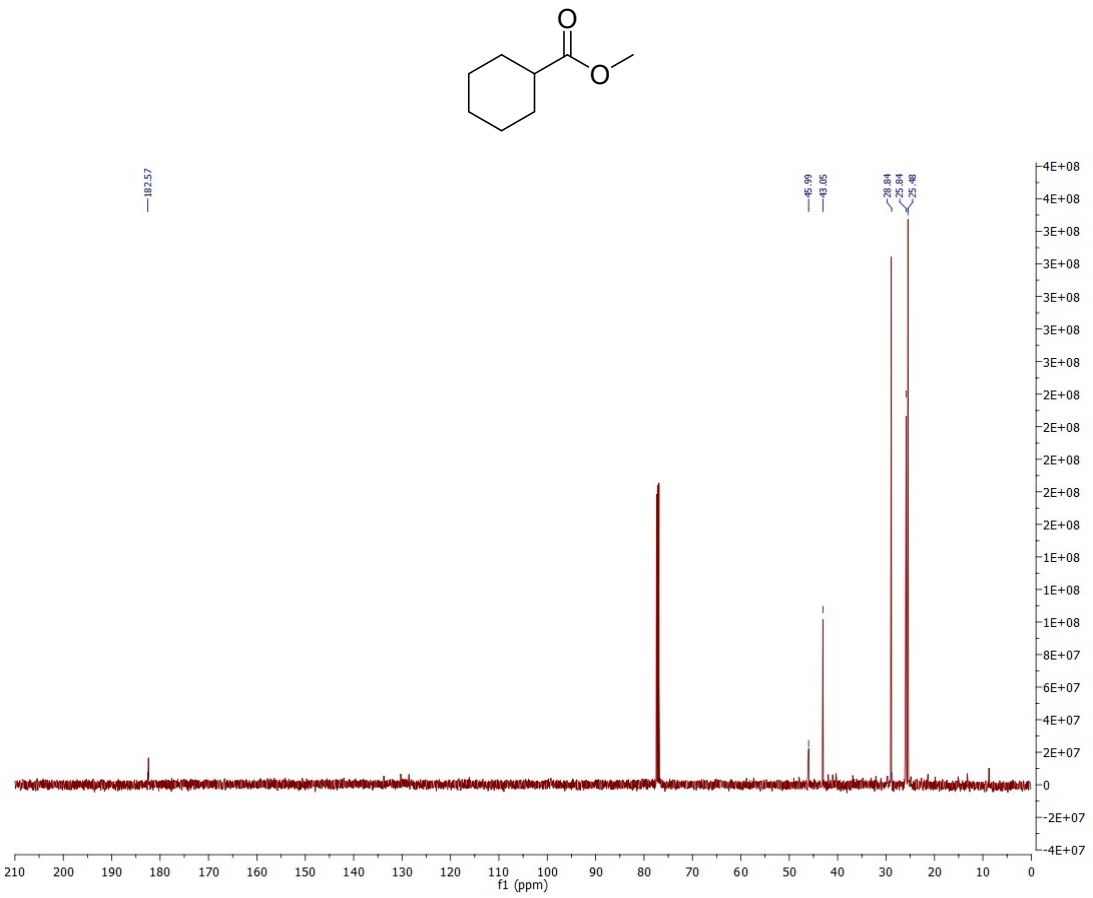




[1]HNMR (6o)



[13]CNMR (6o)



^{13}CNMR (6p)

5) References

1- S. C. Yan, Z. S. Li, Z. G. Zou, *Langmuir* **2009**, *25(17)*, 10397–10401.

2- L. Kong, Y. Dong, P. Jiang, G .Wang, H .Zhang, N. Zhao, *J. Mater. Chem*. *A*. **2016**, *4*, 9998-10007.

3- H. Jensen, J. H. Pedersen, J. E. JØrgensenz, J. Skov Pedersen, K. D. Joensen, S. B. Iversen, E. G. SØgaard, *J. Exp. Nanosci.* 2006, 1, 355–373.