Supporting Information

Direct Access to Acylated Azobenzenes via Pd-Catalyzed C–H Functionalization and Further Transformation into Indazole Backbone

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1. General considerations

All ^{1}H NMR and ^{13}C NMR spectra were recorded on a 400 MHz Bruker FT-NMR spectrometers (400 MHz or 100 MHz, respectively). All chemical shifts are given as δ value (ppm) with reference to tetramethylsilane (TMS) as an internal standard. The peak patterns are indicated as follows: s, singlet; d, doublet; t, triplet; m, multiplet; q, quartet. The coupling constants, J, are reported in Hertz (Hz). High resolution mass spectroscopy data of the product were collected on a Waters Micromass GCT instrument. Azobenzenes were prepared from arylamines, using CuBr as catalyst (Zhang, C.; Jiao N. *Angew. Chem., Int. Ed.* **2010**, *49*, 6174). The chemicals and solvents were purchased from commercial suppliers either from Aldrich, USA or Shanghai Chemical Company, China. All the solvents were dried and freshly distilled prior to use. Products were purified by flash chromatography on 100–200 mesh silica gels, SiO_2.

2. Typical procedure for the acylation of azobenzenes

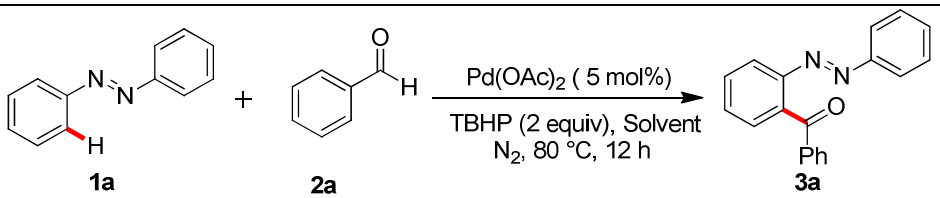
A 10 mL of reaction tube was charged with azobenzene (**1a**, 36.4 mg, 0.20 mmol), benzaldehyde (**2a**, 23.3 mg, 0.22 mmol), $Pd(OAc)_2$ (2.3 mg, 0.01 mmol), TBHP (40μL, 0.40 mmol, dried over 4Å MS prior to use) and anhydrous 1, 2-dichloroethane (1.0 mL) under N_2 atmosphere. The reaction vessel was placed in an oil bath. After the reaction was carried out at 80 ^{o}C for 12 h, it was cooled to room temperature, extracted with EtOAc (2×5 mL). The organic layers were combined, dried over Na_2SO_4, and concentrated to yield the crude product, which was further purified by flash chromatography (silica gel, ethyl acetate/petroleum ether 1:20, v/v), affording the desired product **3a** as a red solid (44.6 mg, 78%).

3. Typical procedure for the synthesis of indazole derivatives

A 5 mL of reaction tube was charged with zinc dust (26.0 mg, 0.40 mmol), NH_4Cl (32.1 mg, 0.60 mmol), **3a** (57.2 mg, 0.20 mmol) and CH_3OH (0.50 mL). The reaction mixture was stirred at room temperature for 5 min. After red color was completely disappeared, the resulting slurry was diluted with water and filtrated. The filter liquor was then extracted with EtOAc (3×5 mL), and dried over $MgSO_4$. Then the solvent was evaporated under reduced pressure and the residue was purified by flash chromatography (silica gel, ethyl acetate/petroleum ether 1:5, v/v), affording the desired product **4a** as a white solid (53.0 mg, 99%).

4. Effect of solvent on the acylation reaction

Table S1 Effect of the solvent on the acylation reaction of azobenzene (**1a)** with benzaldehyde (**2a**)[a]

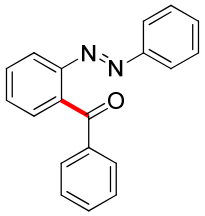


1a **2a** **3a**

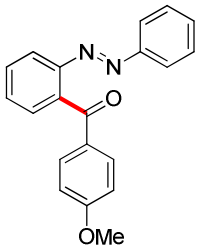
Entry	Solvent	Yield [%][b]
1	benzene	65
2	toluene	72
3	DMF	17
4	DMSO	25
5	DCE	78
6	dioxane	56
7	THF	52
8	diglyme	0

[a] Reaction conditions: azobenzene (**1a**, 0.20 mmol), benzaldehyde (**2a**, 0.22 mmol), $Pd(OAc)_2$(5 mol%), TBHP (0.40 mmol, dried over 4Å MS prior to use), solvent (1.0 mL), 80 °C , sealed tube, N_2, 12 h. [b] Isolated yields.

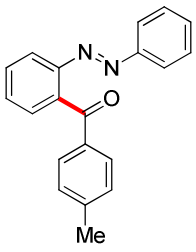
5. Characterization data for the products



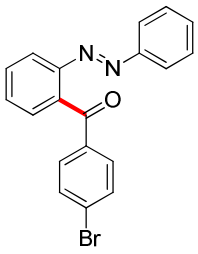
3a:[1] ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.94 (d, *J* = 7.6 Hz, 1H), 7.78–7.76 (m, 2H), 7.67–7.63 (m, 1H), 7.59–7.58 (m, 2H), 7.47–7.42 (m, 3H), 7.36–7.32 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.01, 152.03, 150.36, 138.46, 136.90, 132.68, 131.28, 130.79, 130.74, 129.36, 128.84, 128.74, 128.28, 122.83, 119.99.



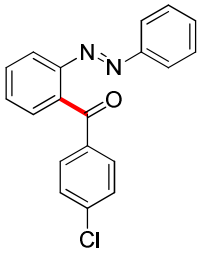
3b: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92 (d, *J* = 8.8 Hz, 1H), 7.76 (dd, *J* = 6.8 Hz, *J* = 2.0 Hz, 2H), 7.62–7.50 (m, 5H), 7.37–7.34 (m, 3H), 6.86 (dd, *J* = 6.8 Hz, *J* = 2.0 Hz, 2H), 3.81 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.1, 163.39, 152.19, 150.18, 137.79, 131.90, 131.47, 131.25, 130.76, 130.40, 128.89, 128.52, 122.94, 119.32, 113.59, 55.43. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{20}H_{17}N_2O_2$: 317.1290, Found: 317.1289.



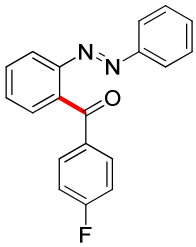
3c: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.93 (d, *J* = 7.6 Hz, 1H), 7.68–7.61 (m, 3H), 7.57–7.55 (m, 2H), 7.49–7.46 (m, 2H), 7.36–7.31 (m, 3H), 7.17 (d, *J* = 7.6 Hz, 2H), 2.35 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.72, 152.13, 150.28, 143.58, 137.44, 135.96, 131.23, 130.74, 130.61, 130.53, 129.64, 129.55, 129.01, 128.84, 128.61, 122.91, 119.65, 30.84. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{20}H_{17}N_2O$: 301.1341, Found: 301.1341.



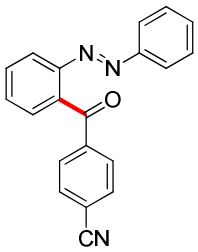
3d: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (d, J = 8.0 Hz, 1H), 7.54–7.52 (m, 3H), 7.47–7.45 (m, 2H), 7.41–7.35(m, 4H), 7.28–7.24 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.85, 151.84, 150.26, 137.24, 136.07, 131.59, 131.49, 130.95, 130.88, 130.74, 128.92, 128.65, 127.77, 122.79, 120.46. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{19}H_{14}BrN_2O$: 365.0290, Found: 365.0290.



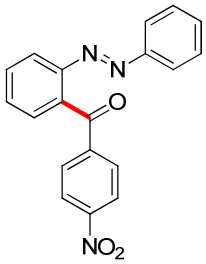
3e: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.96 (d, J = 7.6 Hz, 1H), 7.72–7.66 (m, 3H), 7.59–7.56 (m, 2H), 7.47–7.45(m, 2H), 7.38–7.33 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.78, 151.93, 150.34, 139.14, 136.87, 136.21, 132.71, 131.54, 130.99, 130.93, 130.69, 129.40, 128.98, 128.87, 128.69, 128.67, 128.31, 122.86, 120.52. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{19}H_{14}ClN_2O$: 321.0795, Found: 321.0793.



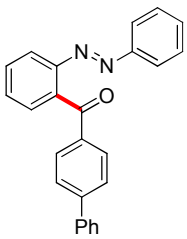
3f: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82 (d, J = 8.4 Hz, 1H), 7.68 (dd, J = 8.8 Hz, J = 5.6 Hz, 2H), 7.53–7.49 (m, 1H), 7.44–7.41 (m, 2H), 7.37–7.35 (m, 2H), 7.24–7.18 (m, 3H), 6.91 (d, J = 8.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.40, 165.35 (d, J_{C-F} = 253.5 Hz), 157.77, 150.08, 136.36, 134.70 (d, J_{C-F} = 3.0 Hz), 131.84 (d, J_{C-F} = 9.1 Hz), 131.42, 130.82 (d, J_{C-F} = 5.4 Hz), 128.86, 128.53, 122.72, 120.17, 115.37 (d, J_{C-F} = 22.0 Hz). HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{19}H_{14}FN_2O$: 305.1090, Found: 305.1090.



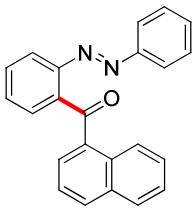
3g: ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.01 (d, *J* = 7.6 Hz, 1H), 7.84 (d, *J* = 8.0 Hz, 2H), 7.75–7.71 (m, 1H), 7.67–7.60 (m, 4H), 7.42–7.34 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.41, 151.60, 150.52, 141.78, 134.68, 132.21, 131.87, 131.61, 131.15, 129.34, 129.04, 128.98, 122.74, 121.67, 117.98, 115.68. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{20}H_{14}N_3O$: 312.1137, Found: 312.1133.



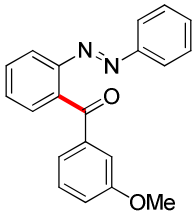
3h: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.95 (d, *J* = 8.0 Hz, 1H), 7.81–7.8 (m, 2H), 7.65–7.63 (m, 1H), 7.59–7.56 (m, 2H), 7.48–7.46 (m, 2H), 7.38–7.32 (m, 3H), 7.06–7.02 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.47, 166.77, 164.24, 151.98, 150.28, 136.55, 132.01, 131.92, 131.49, 130.90, 130.87, 129.39, 128.96, 128.65, 128.31, 122.84, 120.25, 115.57, 115.35. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{19}H_{14}N_3O_3$: 332.1035, Found: 332.1032.



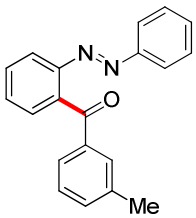
3i: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.96 (d, *J* = 7.6 Hz, 1H), 7.85 (d, *J* = 8.4 Hz, 2H), 7.65–7.60 (m, 1H), 7.59–7.55 (m, 6H), 7.47–7.41 (m, 4H), 7.38–7.30 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.62, 152.11, 150.39, 145.44, 139.98, 137.27, 137.10, 131.32, 130.84, 130.79, 130.03, 128.88, 128.87, 128.79, 128.11, 127.24, 126.99, 122.89, 119.83. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{25}H_{19}N_2O$: 363.1497, Found: 363.1499.



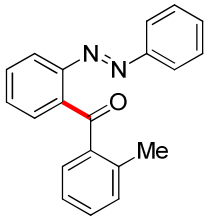
3j: ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.87 (d, *J* = 8.0 Hz, 1H), 7.87–7.79 (m, 4H), 7.67–7.60 (m, 3H), 7.58–7.53 (m, 1H),7.46 (dd, *J* = 7.6 Hz, *J* = 1.6 Hz, 1H), 7.30–7.20 (m, 2H), 7.20–7.07 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.98, 152.03, 150.89, 138.64, 137.10, 133.91, 132.51, 131.43, 131.07, 130.59, 130.45, 130.17, 129.55, 128.59, 128.27, 127.98, 126.41, 126.15, 124.19, 122.86, 118.89. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{23}H_{17}N_2O$: 337.1341, Found: 337.1338.



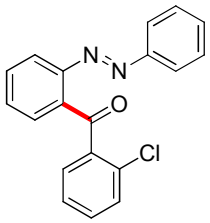
3k: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.94 (d, *J* = 8.0 Hz, 1H), 7.64–7.59 (m, 1H), 7.58–7.56 (m, 2H), 7.49–7.47 (m, 2H), 7.41 (d, *J* = 2.0 Hz, 1H), 7.36–7.34 (m, 3H), 7.24 (d, *J* = 3.6 Hz, 2H), 7.03 (d, *J* = 2.4 Hz, 1H), 3.80 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.84, 159.68, 152.07, 150.44, 139.83, 136.89, 131.32, 130.75, 130.74, 129.31, 128.89, 128.70, 122.90, 122.67, 120.21, 119.38, 113.17, 55.46. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{20}H_{17}N_2O_2$: 317.1290, Found: 317.1292.



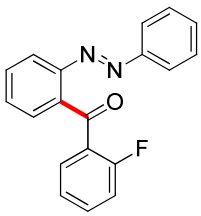
3l: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (d, *J* = 7.6 Hz, 1H), 7.56–7.54 (m, 2H), 7.49 (d, *J* = 3.2 Hz, 3H), 7.44 (d, *J* = 7.2 Hz,, 2H), 7.39–7.37 (m, 3H), 7.28–7.14 (m, 2H), 2.24 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.25, 152.11, 150.41, 138.50, 138.09, 137.21, 133.49, 131.25, 130.72, 130.67, 129.74, 128.84, 128.72, 128.18, 126.88, 122.89, 119.84, 29.67. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{20}H_{17}N_2O$: 301.1341, Found: 301.1348.



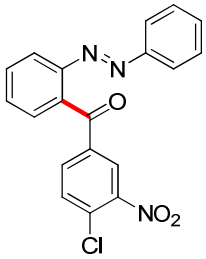
3m: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.79 (d, J = 8.0 Hz, 1H), 7.70–7.68 (m, 1H), 7.64–7.58 (m, 2H), 7.64–7.50 (m, 5H), 7.40–7.35 (m, 2H), 7.32–7.30 (m, 1H), 7.28–7.05 (m, 1H), 2.56 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.98, 152.39, 150.84, 139.10, 138.39, 138.17, 131.49, 131.31, 131.21, 131.18, 130.66, 130.60, 129.42, 128.79, 125.36, 122.89, 118.82, 21.12. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{20}H_{17}N_2O$: 301.1341, Found: 301.1348.



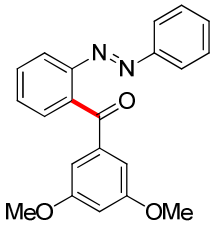
3n: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82 (dd, J = 7.2 Hz, J = 1.6 Hz, 1H), 7.30 (dd, J = 8.0 Hz, J = 1.2 Hz, 1H), 7.67–7.63 (m, 1H), 7.61–7.58 (m, 1H), 7.53 (dd, J = 6.4 Hz, J = 2.0 Hz, 1H), 7.41–7.38 (m, 3H), 7. 35–7.31 (m, 2H), 7.30–7.23 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.77, 152.26, 151.02, 139.85, 137.01, 132.33, 132.15, 131.68, 131.42, 130.76, 130.65, 130.59, 129.97, 128.78, 126.56, 123.15, 117.68. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{19}H_{14}ClN_2O$: 321.0795, Found: 321.0797.



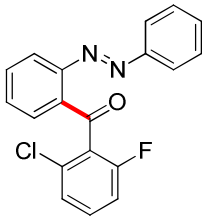
3o: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.86 (dd, J = 7.6 Hz, J = 1.2 Hz 1H), 7.81–7.76 (m, 1H), 7.70–7.56 (m, 3H), 7.42–7.30 (m, 6H), 7.21–7.17 (m, 1H), 6.97–6.92 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.58, 161.03 (d, J_{C-F} = 254.8 Hz), 152.06, 150.47 (d, J_{C-F} = 1.8 Hz), 137.94 (d, J_{C-F} = 1.1 Hz), 133.99, 133.89, 131.48, 131.39, 130.85, 130.66 (d, J_{C-F} = 1.8 Hz), 129.08, 128.86, 128.83, 128.82, 128.69, 128.36, 128.25, 124.06 (d, J_{C-F} = 3.9 Hz), 122.96, 122.85, 120.51, 119.19, 116.42 (d, J_{C-F} = 21.9 Hz). HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{19}H_{14}FN_2O$: 305.1090 , Found: 305.1094.



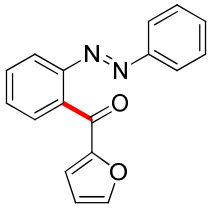
3p: ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.22 (d, J = 2.0 Hz, 1H), 8.01 (dd, J = 6.0 Hz, J = 1.2 Hz, 1H), 7.83 (dd, J = 8.8 Hz, J = 2.4 Hz, 1H), 7.75–7.71 (m, 1H), 7.66–7.62 (m, 1H), 7.58 (dd, J = 7.6 Hz, J = 1.2 Hz, 1H), 7.53 (d, J = 8.4 Hz, 1H), 7.48–7.46 (m, 2H), 7.42–7.36 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.53, 151.58, 150.48, 138.17, 133.95, 132.72, 132.01, 131.83, 131.24, 130.87, 129.14, 128.86, 125.66, 122.80, 122.05. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{19}H_{13}ClN_3O_3$: 366.0645, Found: 366.0644.



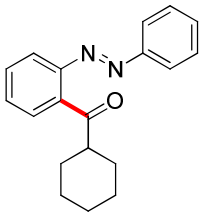
3q: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.94 (d, J = 8.4 Hz, 1H), 7.64–7.60 (m, 2H), 7.59–7.39 (m, 4H), 7.38–7.34 (m, 3H), 7.17 (dd, J = 8.0 Hz, J = 2.0 Hz, 1H),6.74 (d, J = 2.1 Hz, 1H), 3.92 (s, 3H), 3.87 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.68, 153.18, 152.13, 150.26, 148.99, 137.58, 131.52, 131.27, 130.65, 130.38, 128.89, 128.49, 125.52, 122.87, 119.34, 110.78, 109.90, 56.01, 55.97. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{21}H_{19}N_2O_3$: 347.1396, Found: 347.1393.



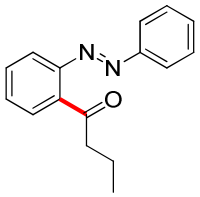
3r: ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.01 (dd, J = 7.2 Hz, J = 1.2 Hz, 1H), 7.69–7.65 (m, 1H), 7.62–7.56 (m, 2H), 7.47–7.34 (m, 5H), 7.17–7.09 (m, 2H), 6.93–6.89 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 191.21, 159.54 (d, J_{C-F} = 250.8 Hz), 152.37, 151.70, 151.69, 135.73, 133.67, 131.86 (d, J_{C-F} = 5.2 Hz), 131.58, 130.78, 130.72, 130.69, 130.62, 130.53, 130.32, 128.77, 125.71 (d, J_{C-F} = 3.4 Hz), 123.29, 116.40, 114.44 (d, J_{C-F} = 11.7 Hz). HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{19}H_{13}ClFN_2O$: 339.0700, Found: 339.0697.



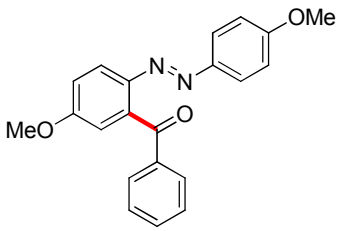
3s: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92 (7.2 Hz, 1H), 7.67–7.61 (m, 4H), 7.59–7.53 (m, 2H), 7.42–7.39 (m, 3H), 6.99 (d, *J* = 2.8 Hz, 1H), 6.47–6.46 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 184.17, 153.39, 152.23, 150.62, 146.73, 136.12, 131.44, 131.18, 130.65, 128.99, 128.76, 122.99, 119.54, 119.27, 112.26. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{17}H_{13}N_2O_2$: 277.0977, Found: 277.0970.



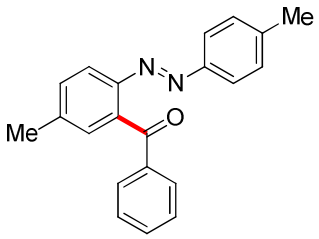
3t: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.89–7.86 (m, 2H), 7.81–7.79 (m, 1H), 7.57–7.46 (m, 6H), 2.96 (q, *J* = 6.8 Hz, 1H), 1.95 (d, *J* = 3.6 Hz, 2H), 1.92–1.73 (m, 2H), 1.62–1.58 (m, 2H), 1.48–1.43 (m, 2H), 1.33–1.16 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 209.34, 152.45, 149.86, 139.01, 131.53, 130.69, 130.59, 129.19, 128.07, 123.15, 118.48, 51.79, 28.69, 25.93, 25.75. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{19}H_{21}N_2O$: 293.1654, Found: 293.1650.



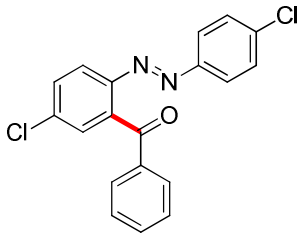
3u: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.89 (dd, *J* = 7.6 Hz, *J* = 2.0 Hz, 2H), 7.79–7.76 (m, 1H), 7.57–7.50 (m, 6H), 2.89 (t, *J* = 7.2 Hz, 2H), 1.76–1.57 (m, 2H), 0.94 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 205.75, 152.42, 150.05, 138.93, 131.61, 131.01, 130.77, 129.22, 127.95, 123.22, 118.55, 46.95, 29.69, 17.92. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{16}H_{17}N_2O$: 253.1341, Found: 253.1339.



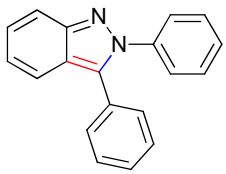
3v: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.93 (d, *J* = 8.8 Hz, 1H), 7.79–7.76 (m, 2H), 7.46–7.44 (m, 1H), 7.38–7.34 (m, 4H), 7.14–7.11 (m, 1H), 7.03 (d, *J* = 6.8 Hz, 1H), 6.79 (dd, *J* = 6.8 Hz, *J* = 1.6 Hz, 2H), 3.89 (s, 3H), 3.79 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.99, 161.79, 161.31, 146.45, 144.55, 138.44, 138.28, 132.60, 129.26, 128.24, 124.39, 121.92, 116.69, 113.96, 112.88, 55.75, 55.43. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{21}H_{19}N_2O_3$: 347.1396, Found: 347.1392.



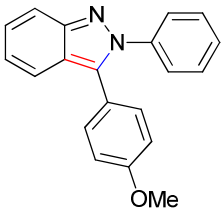
3w: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (d, *J* = 8.4 Hz, 1H), 7.77–7.75 (m, 2H), 7.45–7.41 (m, 2H), 7.37–7.30 (m, 5H), 7.09 (d, *J* = 7.6 Hz, 2H), 2.47 (s, 3H), 2.32 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.40, 150.22, 148.51, 141.63, 141.31, 138.63, 136.73, 132.54, 131.42, 129.48, 129.31, 129.13, 128.22, 122.73, 120.14, 29.67, 21.39. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{21}H_{19}N_2O$: 315.1497, Found: 315.1497.



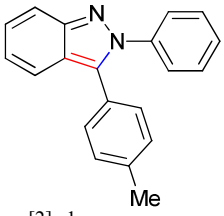
3x: ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.89 (d, *J* = 8.8 Hz, 1H), 7.76 (d, *J* = 7.6 Hz, 2H), 7.60 (dd, *J* = 8.8 Hz, *J* = 2.0 Hz, 1H), 7.56 (d, *J* = 2.4 Hz, 1H), 7.53-7.49 (m, 1H), 7.41–7.34 (m, 4H), 7.30–7.28 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 147.39, 138.42, 135.21, 134.34, 129.52, 129.23, 129.06, 128.83, 128.64, 128.38, 127.14, 126.95, 122.24, 119.30, 119.15. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{19}H_{13}Cl_2N_2O$: 355.0405, Found: 355.0404.



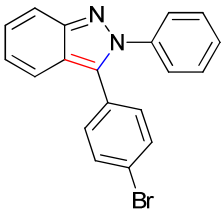
4a[2]**:** ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.86 (d, *J* = 8.8 Hz, 1H), 7.74 (d, *J* = 8.8 Hz, 1H), 7.49–7.47 (m, 2H), 7.45–7.34 (m, 9H), 7.17–7.14 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 148.87, 140.09, 135.23, 129.75, 129.51, 128.82, 128.61, 128.15, 128.09, 126.85, 125.86, 122.38, 121.63, 120.36, 117.62.



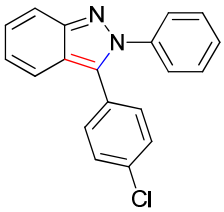
4b[2]**:** ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.78 (d, *J* = 8.8 Hz, 1H), 6.68 (d, *J* = 8.4 Hz, 1H), 7.45-7.42 (m, 2H), 7.38–7.32 (m, 4H), 7.27–7.25 (m, 2H), 7.12–7.07 (m, 1H), 6.91–6.88 (m, 2H), 3.79 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 159.50, 148.87, 140.24, 135.27, 130.85, 128.86, 128.05, 126.84, 125.91, 122.11, 122.06, 121.51, 120.52, 117.58, 114.20, 55.16.



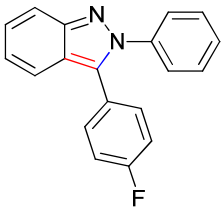
4c[2]**:** ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.79 (d, *J* = 8.8 Hz, 1H), 7.68 (d, *J* = 8.4 Hz, 1H), 7.44–7.42 (m, 2H), 7.36–7.31 (m, 4H), 7.23–7.21 (m, 2H), 7.16–7.15 (m, 2H), 7.11–7.07 (m, 1H), 2.34 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 148.91, 140.26, 138.16, 135.44, 129.42, 129.39, 128.82, 128.04, 126.86, 126.82, 125.91, 122.20, 121.61, 120.52, 117.61, 21.19.



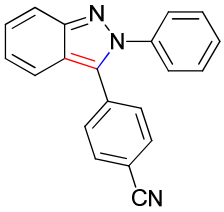
4d[2]**:** ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.79 (d, *J* = 8.8 Hz, 1H), 7.64 (d, *J* = 8.0 Hz, 1H), 7.49–7.47 (m, 2H), 7.42–7.32 (m, 6H), 7.20–7.17 (m, 2H), 7.12 (dd, *J* = 6.8 Hz, *J* = 8.4 Hz,1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 148.94, 139.89, 133.93, 131.94, 130.98, 129.02, 128.75, 128.36, 126.96, 125.89, 122.79, 122.56, 121.59, 119.98, 117.83.



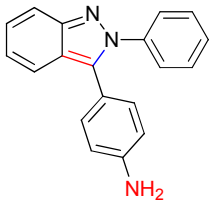
4e[2]**:** ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.80 (d, *J* = 8.8 Hz, 1H), 7.70 (d, *J* = 8.4 Hz, 1H), 7.44–7.35 (m, 8H), 7.30–7.26 (m, 2H), 7.17–7.14 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 148.94, 139.89, 133.93, 131.94, 130.98, 129.02, 128.75, 128.36, 126.96, 125.89, 122.79, 122.56, 121.59, 119.98, 117.83.



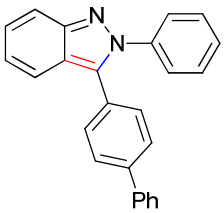
4f: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.83 (d, J = 8.8 Hz, 1H), 7.67 (d, J = 8.0 Hz, 1H), 7.45–7.31 (m, 8H), 7.17–7.07 (m, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 162.45 (d, J_{C-F} = 247.8 Hz), 148.85, 139.94, 134.21, 131.35 (d, J_{C-F} = 8.1 Hz), 128.96, 128.27, 126.94, 125.89, 122.57, 121.62 (d, J_{C-F} = 0.4 Hz), 120.09, 117.73, 115.88 (d, J_{C-F} = 21.6 Hz). HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{19}H_{14}FN_2$: 289.1141, Found: 289.1143.



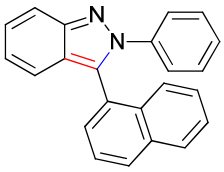
4g[2]**:** ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.83 (d, J = 8.8 Hz, 1H), 7.70–7.66 (m, 3H), 7.47–7.38 (m, 8H), 7.23–7.19 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 149.03, 139.65, 134.40, 132.99, 132.45, 129.94, 129.29, 128.83, 127.23, 125.98, 123.63, 121.84, 119.57, 118.31, 118.13, 111.72.



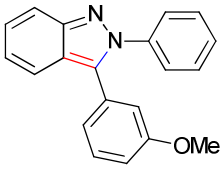
4h: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.79 (d, J = 8.8 Hz, 1H), 7.71 (d, J = 8.8 Hz, 1H), 7.49–7.45 (m, 2H), 7.40–7.33 (m, 4H), 7.14–7.09 (m, 3H), 7.65–7.63 (m, 2H), 3.85 (br, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 149.01, 146.75, 140.56, 136.13, 130.83, 128.94, 128.05, 126.94, 126.06, 121.93, 121.51, 120.93, 119.56, 117.59, 115.09. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{19}H_{16}N_3$: 286.1344, Found: 286.1348.



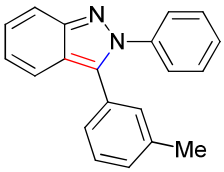
4i: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.88 (d, J = 8.4 Hz, 1H), 7.80 (d, J = 8.8 Hz, 1H), 7.65 (d, J = 8.0 Hz, 4H), 7.54–7.37 (m, 11H), 7.21–7.17 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 148.97, 140.77, 140.19, 139.95, 134.91, 129.87, 128.92, 128.76, 128.65, 128.17, 127.59, 127.23, 126.90, 126.86, 125.95, 122.48, 121.17, 120.42, 117.42. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{25}H_{19}N_2$: 347.1548, Found: 347.1548.



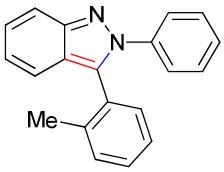
4j: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.96–7.90 (m, 3H), 7.69 (d, J = 8.0 Hz, 1H), 7.51–7.35 (m, 8H), 7.23–7.19 (m, 3H), 7.19–7.06 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 148.79, 140.13, 133.82, 133.59, 131.89, 129.50, 129.37, 128.67, 128.32, 127.80, 127.49, 126.93, 126.70, 126.20, 125.48, 125.18, 124.89, 123.31, 122.18, 120.73, 117.73. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{23}H_{17}N_2$: 321.1392, Found: 321.1388.



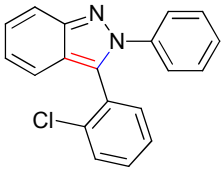
4k: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.80 (d, J = 8.8 Hz, 1H), 7.72 (d, J = 9.2 Hz, 1H), 7.46–7.43 (m, 2H), 7.39–7.33 (m, 4H), 7.29–7.25 (m, 1H), 7.14–7.10 (m, 1H), 6.94–6.87 (m, 3H), 3.66 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 159.55, 148.84, 140.14, 135.15, 130.92, 129.71, 128.87, 128.18, 126.90, 125.91, 122.44, 121.98, 121.59, 120.43, 117.64, 114.86, 114.11, 55.06. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{20}H_{17}N_2O$: 301.1341, Found: 301.1339.



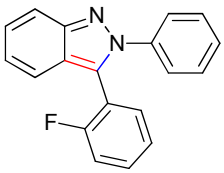
4l: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.85 (d, J = 8.8 Hz, 1H), 7.75 (d, J = 8.0 Hz, 1H), 7.50–7.47 (m, 2H), 7.43–7.37 (m, 4H), 7.30–7.26 (m, 2H), 7.21–7.13 (m, 3H), 2.36 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 148.92, 140.24, 138.36, 135.49, 130.14, 129.75, 129.01, 128.81, 128.53, 128.08, 126.86, 126.78, 125.91, 122.29, 121.68, 120.53, 117.65, 21.31. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{20}H_{17}N_2$: 285.1392, Found: 285.1389.



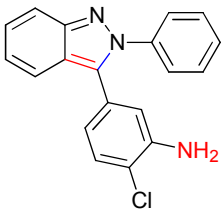
4m[2]**:** ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.82 (dd, J = 9.2 Hz, J = 1.2 Hz, 1H), 7.44–7.41 (m, 3H), 7.36–7.22 (m, 8H), 7.09–7.05 (m, 1H), 1.93 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 148.76, 140.34, 137.69, 135.16, 131.05, 130.56, 129.55, 129.18, 128.79, 127.77, 126.89, 125.96, 124.68, 122.51, 122.02, 120.61, 117.62, 112.19, 19.79.



4n: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.83 (d, J = 8.8 Hz, 1H), 7.48–7.41 (m, 4H), 7.37–7.27 (m, 7H), 7.13–7.09 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 148.69, 140.19, 134.48, 132.60, 132.52, 130.40, 130.10, 129.34, 128.82, 128.04, 126.86, 124.94, 122.64, 122.39, 120.43, 117.77. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{19}H_{14}ClN_2$: 305.0846, Found: 305.0849.

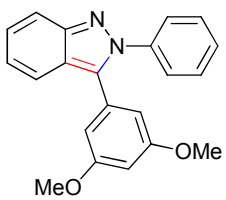


4o: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.86 (d, J = 8.8 Hz, 1H), 7.61 (dd, J = 8.4 Hz, J = 0.8 Hz, 1H), 7.49–7.45 (m, 2H), 7.42–7.34 (m, 6H), 7.22–7.09 (m, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 159.49 (d, J_{C-F} = 249.0 Hz), 148.89, 140.33, 131.79 (d, J_{C-F} = 2.5 Hz), 130.81 (d, J_{C-F} = 7.9 Hz), 129.46, 128.84, 128.15, 126.85, 125.03, 124.35 (d, J = 3.8 Hz), 122.58 (d, J_{C-F} = 6.5 Hz), 120.24 (d, J_{C-F} = 1.6 Hz), 117.78, 116.25 (d, J_{C-F} = 21.4 Hz). HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{19}H_{14}FN_2$: 289.1141, Found: 289.1140.

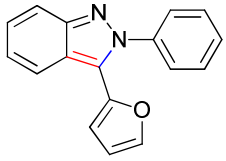


4p: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.78 (d, J = 8.4 Hz, 1H), 7.66 (d, J = 8.0 Hz, 1H), 7.44–7.33 (m, 6H), 7.25–7.23 (m, 1H), 7.13–7.10 (m, 1H), 6.74 (s, 1H), 6.59 (d, J = 8.0 Hz, 1H), 4.11 (br, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 148.87, 143.19, 140.06, 134.57, 129.72, 129.34, 128.96, 128.26, 126.98, 125.81,

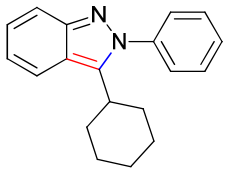
122.50, 121.68, 120.37, 120.17, 119.34, 117.69, 116.42. HRMS (ESI) ([M+H]$^+$) Calcd. for $C_{19}H_{15}ClN_3$: 320.0955, Found: 320.0955.



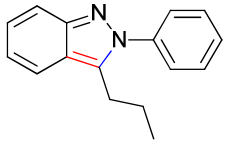
4q: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.78 (d, J = 8.8 Hz, 1H), 7.71 (d, J = 8.8 Hz, 1H), 7.46–7.43 (m, 2H), 7.40–7.32 (m, 4H), 7.13–7.09 (m, 1H), 6.99 (dd, J = 8.0 Hz, J = 2.0 Hz, 1H), 6.89–6.87 (m, 1H), 6.73 (d, J = 2.0 Hz, 1H), 3.88 (s, 3H), 3.62 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 148.99, 148.81, 140.23, 135.24, 128.81, 128.03, 126.78, 125.92, 122.24, 122.15, 121.31, 120.39, 117.58, 112.60, 111.20, 55.70, 55.55. HRMS (ESI) ([M+H]$^+$) Calcd. for $C_{21}H_{19}N_2O_2$: 331.1447, Found: 331.1445.



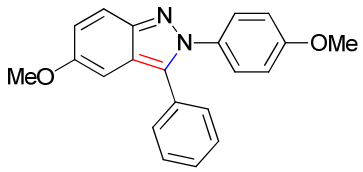
4s: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.03 (d, J = 8.4 Hz, 1H), 7.77 (d, J = 8.8 Hz, 1H), 7.53–7.46 (m, 6H), 7.37–7.34 (m, 1H), 7.19–7.16 (m, 1H), 6.37 (dd, J = 7.6 Hz, J = 2.0 Hz, 1H), 6.04 (d, J = 3.2 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 148.77, 144.73, 142.84, 140.42, 129.14, 129.10, 127.06, 126.97, 126.14, 122.82, 121.15, 120.75, 117.56, 111.30, 109.51. HRMS (ESI) ([M+H]$^+$) Calcd. for $C_{17}H_{13}N_2O$: 261.1028, Found: 2261.1023.



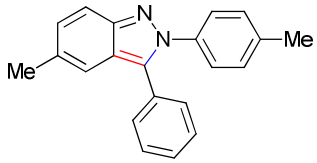
4t: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.86 (d, J = 8.4 Hz, 1H), 7.71 (d, J = 8.8 Hz, 1H), 7.56–7.46 (m, 5H), 7.31–7.27 (m, 1H), 7.06–7.03 (m, 1H), 3.00–2.94 (m, 1H), 2.04–1.91 (m, 4H), 1.87–1.83 (m, 2H), 1.37–1.24 (m, 4H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 148.84, 141.11, 140.18, 129.11, 128.94, 126.41, 126.28, 121.21, 120.49, 119.47, 117.80, 37.26, 32.55, 26.54, 25.84. HRMS (ESI) ([M+H]$^+$) Calcd. for $C_{19}H_{21}N_2$: 277.1705, Found: 277.1708.



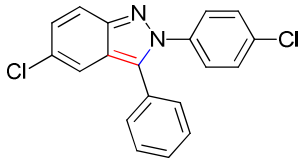
4u: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.74 (d, *J* = 8.8 Hz, 1H), 7.77 (d, *J* = 8.4 Hz,1H), 7.56–7.48 (m, 5H), 7.36–7.31 (m, 1H), 7.10–7.06 (m, 1H), 3.02 (t, *J* = 8.0 Hz, 2H), 1.74–1.65 (m, 2H), 0.92–0.86 (m, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 148.56, 140.09, 136.59, 129.06, 128.77, 126.53, 126.14, 121.10, 120.82, 120.17, 117.53, 114.99, 27.21, 22.69, 13.86. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{16}H_{17}N_2$: 237.1392, Found: 237.1391.



4v[3]**:** ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.70 (d, *J* = 9.2 Hz, 1H), 7.42–7.31 (m, 7H), 7.07 (dd, *J* = 8.8 Hz, *J* = 2.4 Hz, 1H), 6.91–6.86 (m, 3H), 3.83 (s, 3H), 3.82 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 159.13, 155.76, 145.59, 134.16, 133.42, 130.35, 129.50, 128.73, 127.92, 126.97, 121.76, 121.38, 119.05, 114.03, 96.31, 55.43, 55.38.

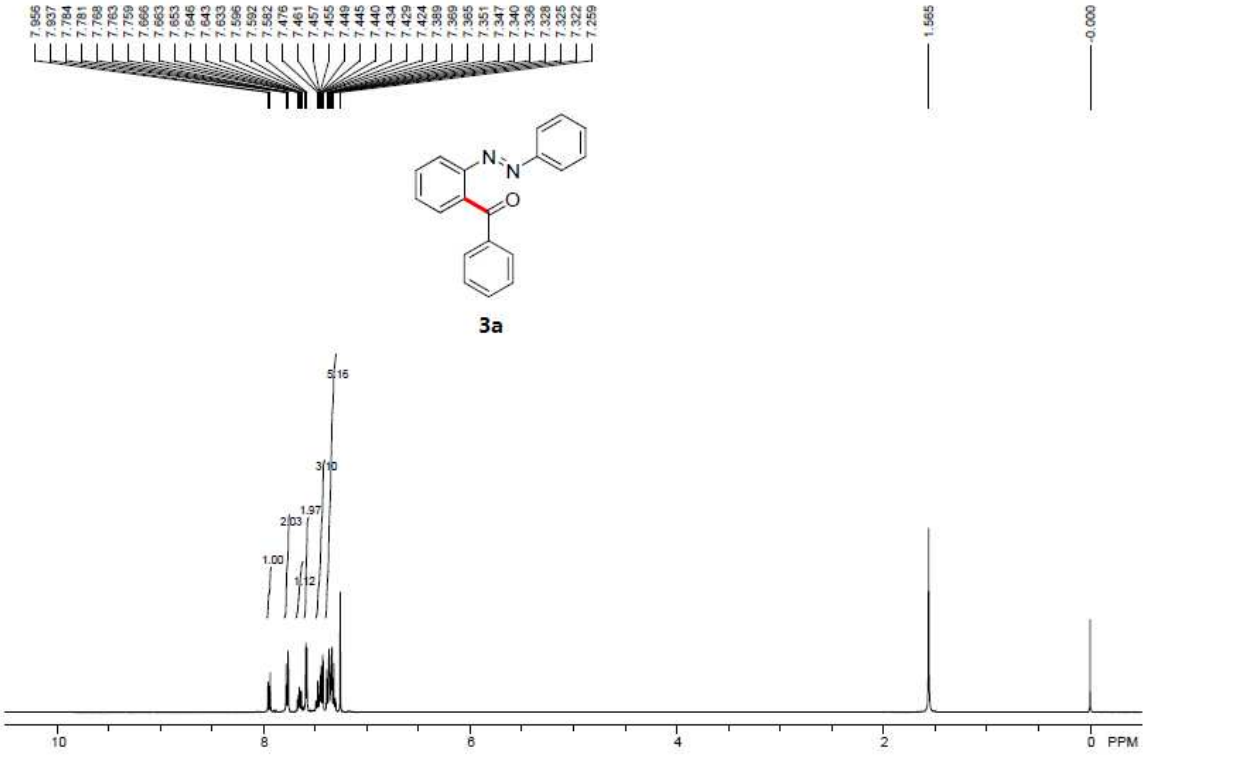


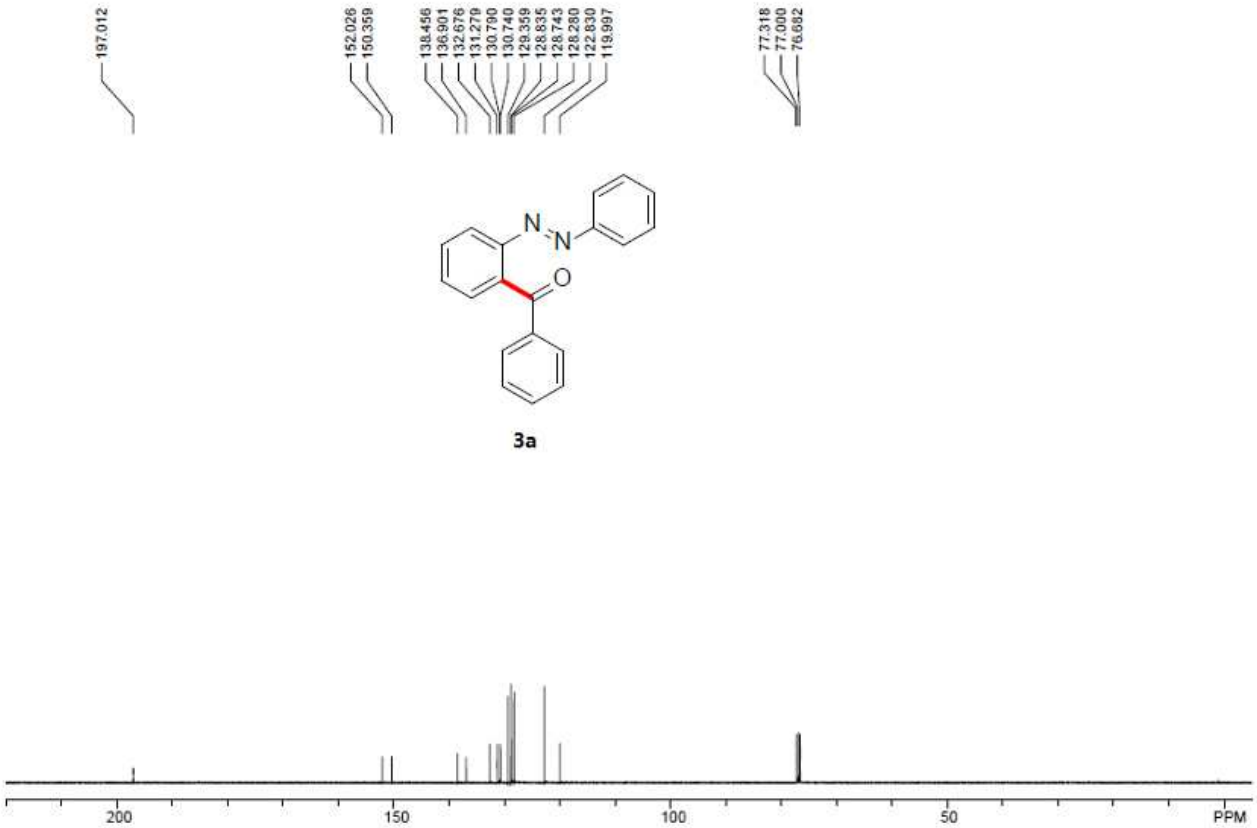
4w: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.76 (d, *J* = 8.8 Hz, 1H), 7.49 (s, 1H), 7.41–7.33 (m, 7H), 7.23 (dd, *J* = 8.8 Hz, *J* = 1.2 Hz, 1H), 7.18 (d, *J* = 8.8 Hz, 2H), 2.46 (s, 3H), 2.39 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 147.77, 137.91, 137.75, 134.22, 131.69, 130.13, 129.67, 129.54, 129.39, 128.56, 127.93, 125.58, 121.83, 118.31, 117.30, 21.72, 20.99. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{21}H_{19}N_2$: 299.1548, Found: 299.1545.



4x: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.72 (d, *J* = 9.6 Hz, 1H), 7.67 (s, 1H), 7.43–7.40 (m, 3H), 7.38–7.35 (m, 4H), 7.34–7.28 (m, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 195.29, 150.23, 148.38, 138.52, 137.86, 137.66, 137.36, 133.15, 130.93, 129.35, 129.24, 128.78, 128.48, 124.13, 121.07. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{19}H_{13}Cl_2N_2$: 339.0456, Found: 339.0455.

6. ^{1}H and ^{13}C NMR spectra of the products



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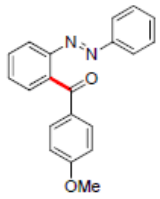
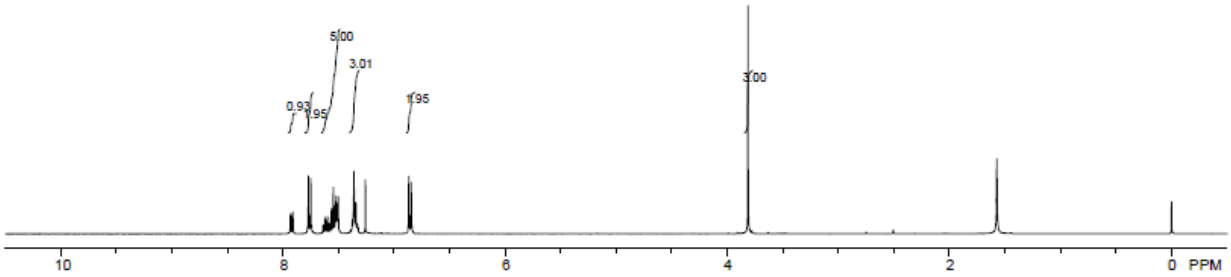
0.000


N
N
O
OMe

3b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3.00
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8
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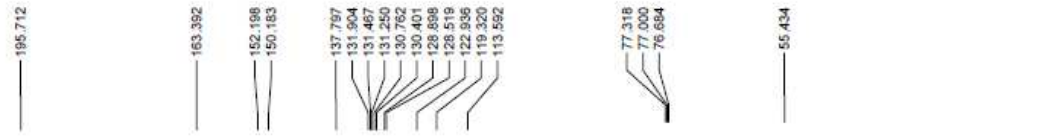
4
2
0 PPM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113.592
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77.000
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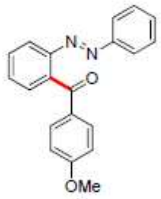
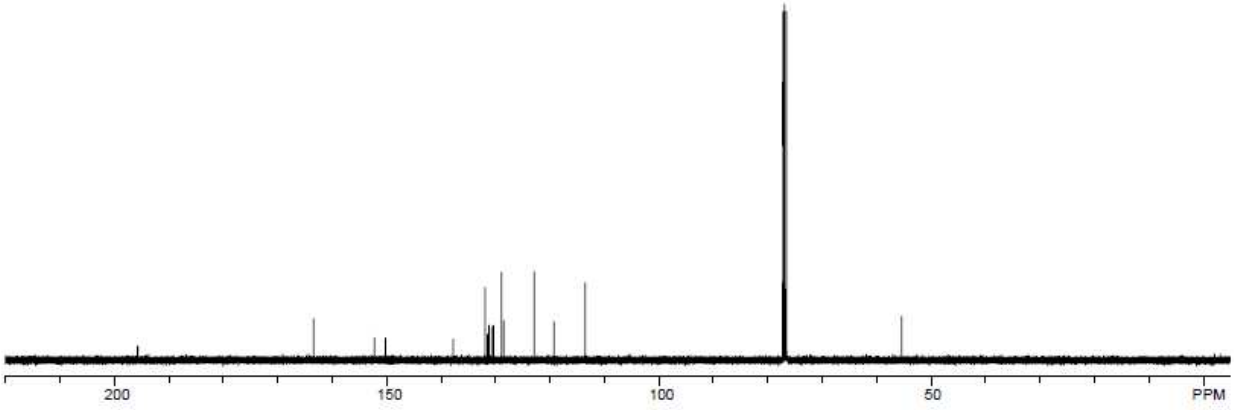
55.434


N
N
O
OMe

3b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100
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PPM


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7.556
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7.482
7.477
7.472
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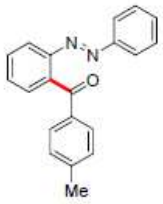
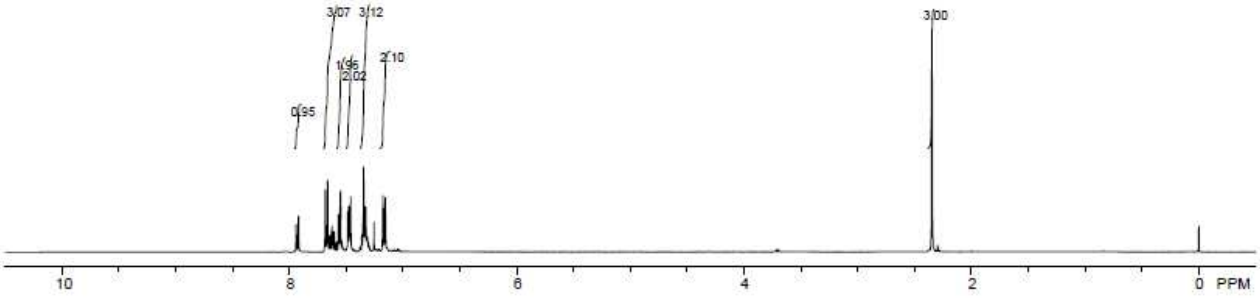

N
N
O
Me

3c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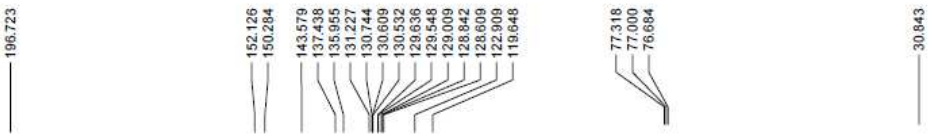
10
8
6
4
2
0
PPM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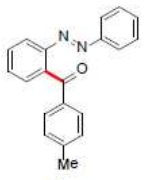
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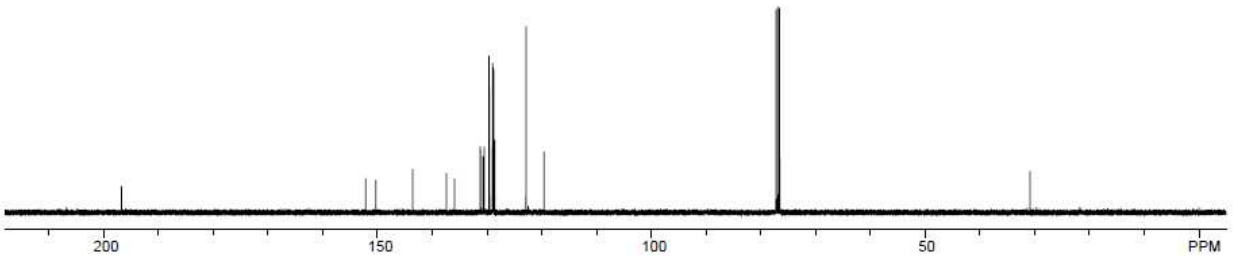
N
O
Me

3c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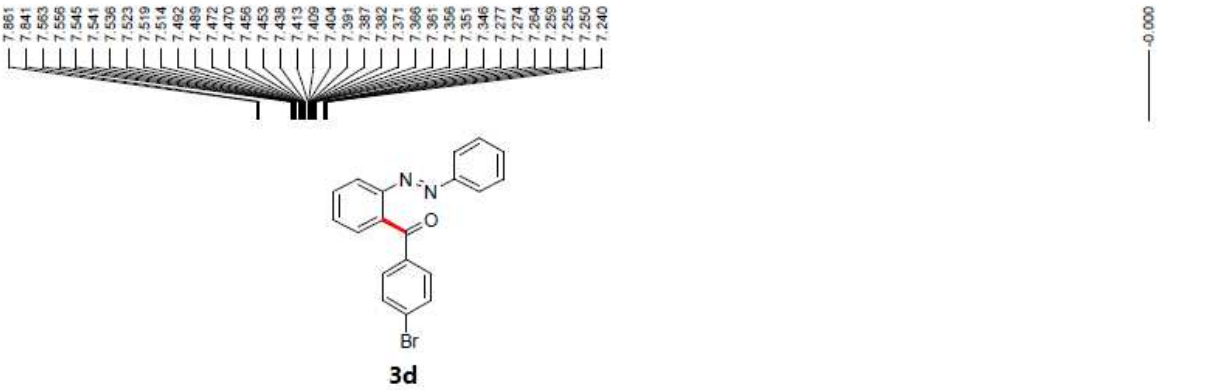
PPM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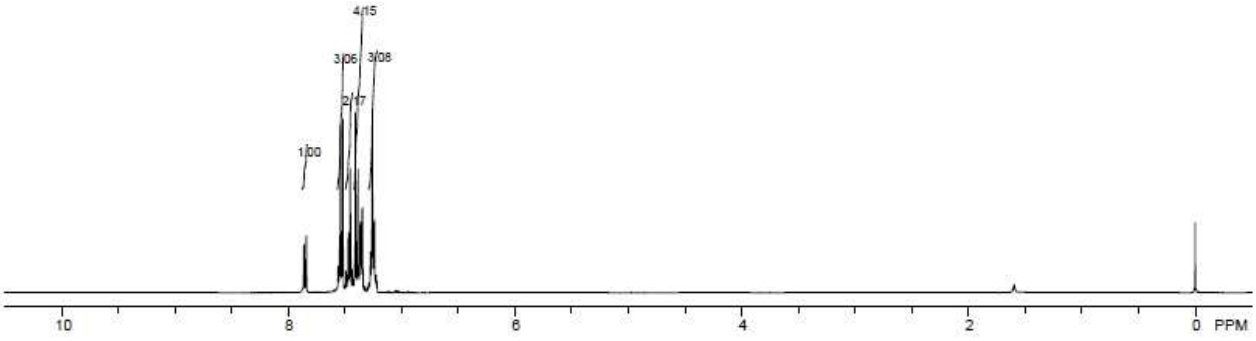
O
Br

3d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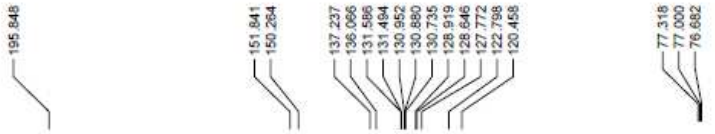
0
PPM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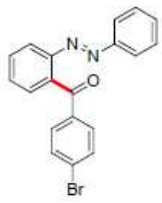
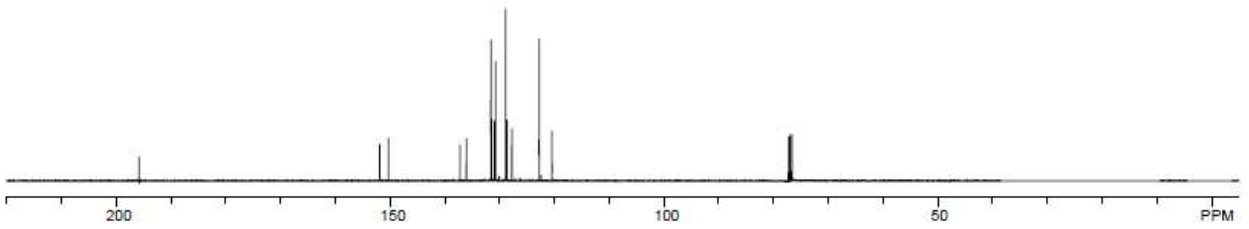
76.682


N
N
O
Br

3d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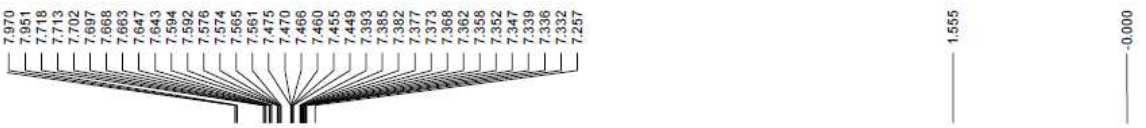
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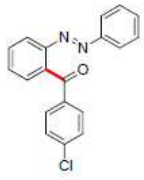
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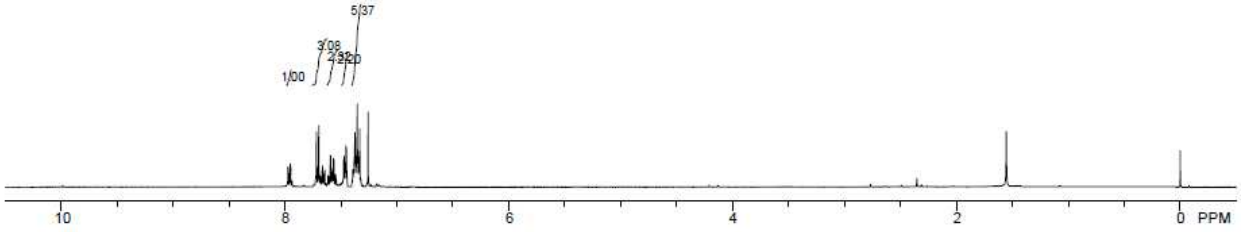
N
O
Cl

3e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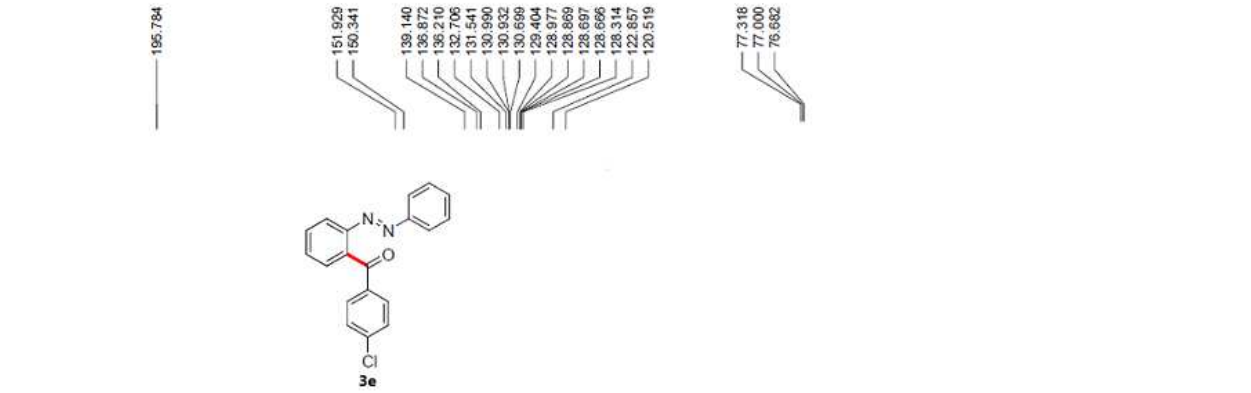
4
2
0
PPM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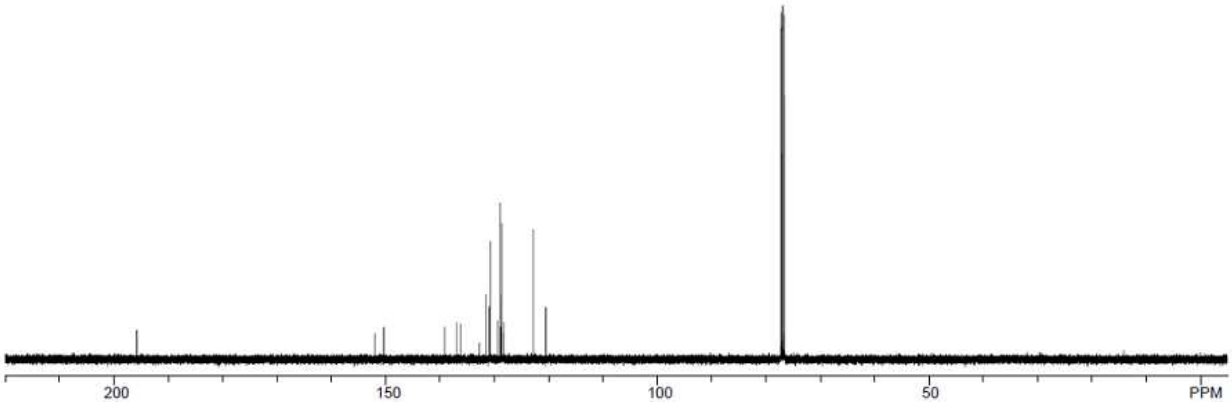
N
N
O
Cl

3e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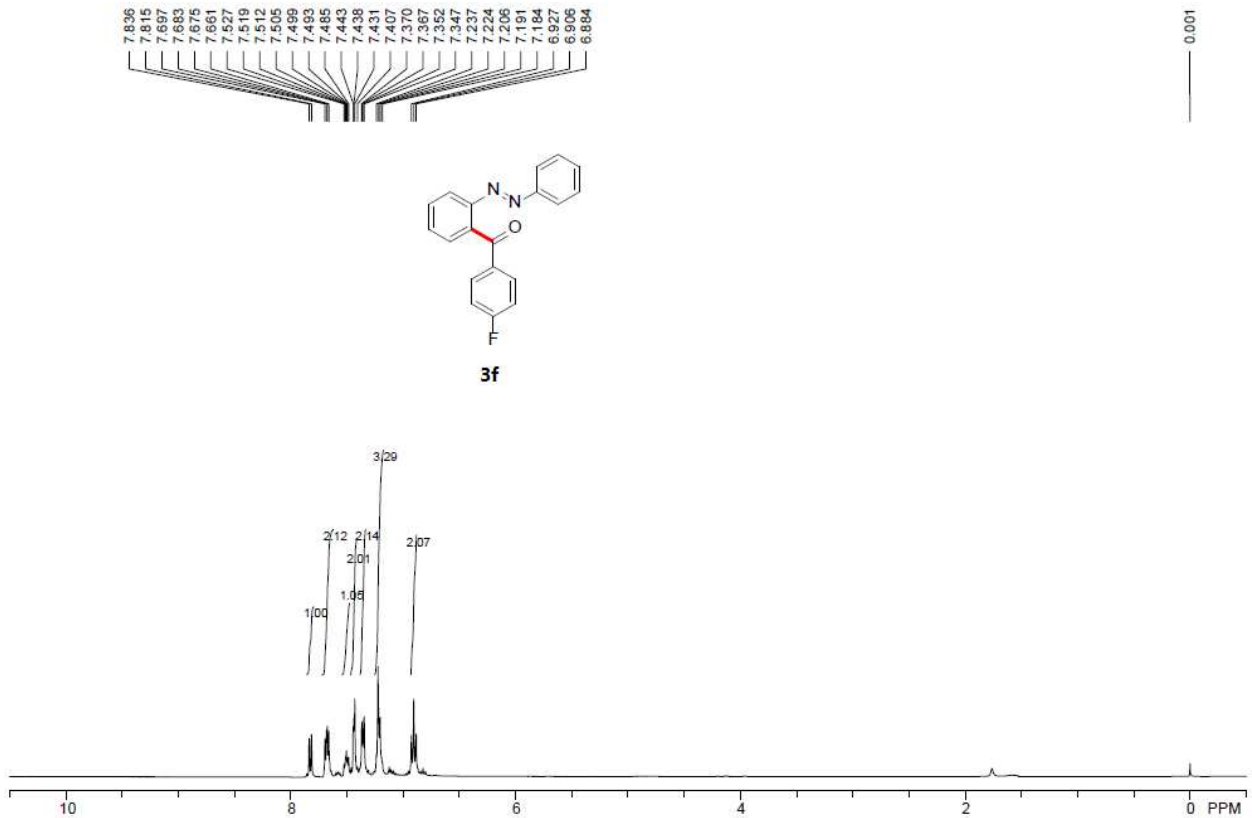
100
50
PPM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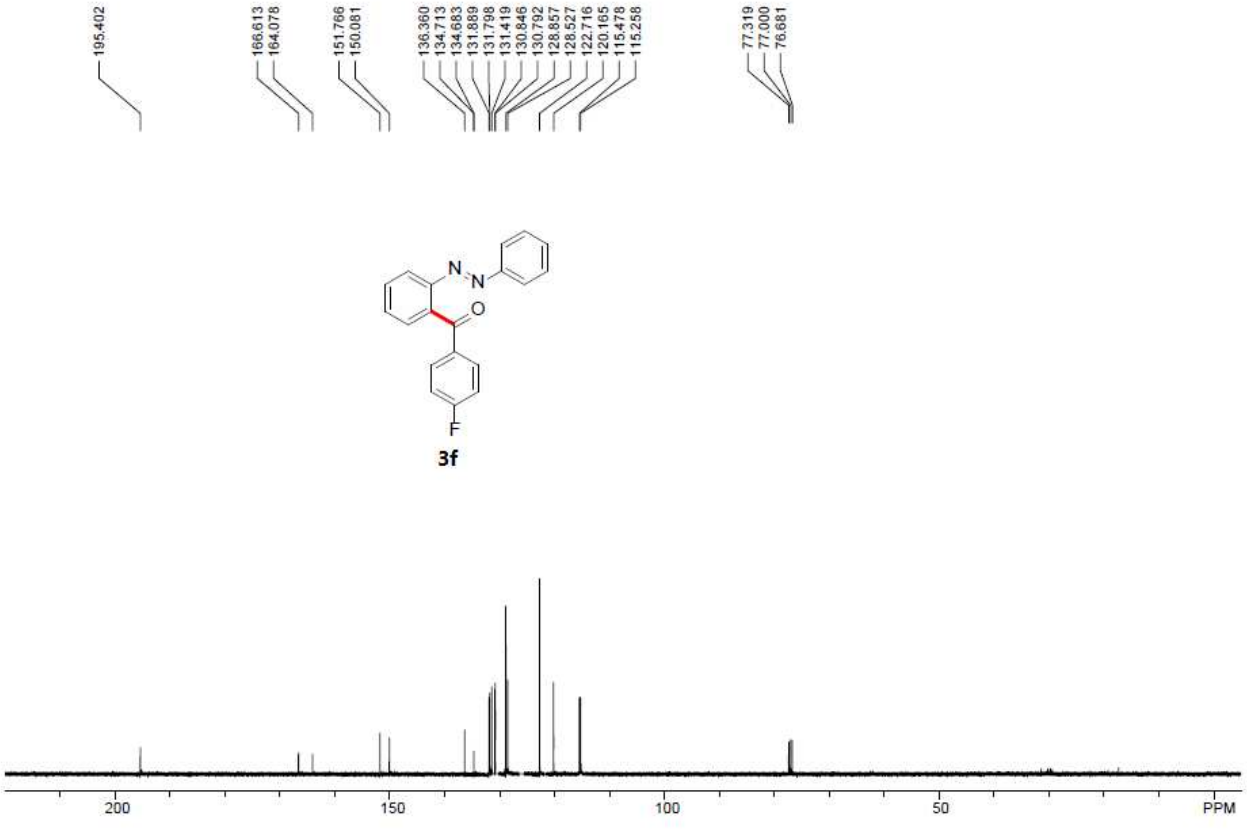
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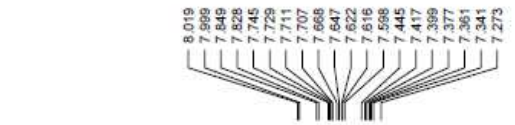
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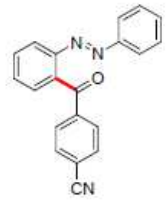
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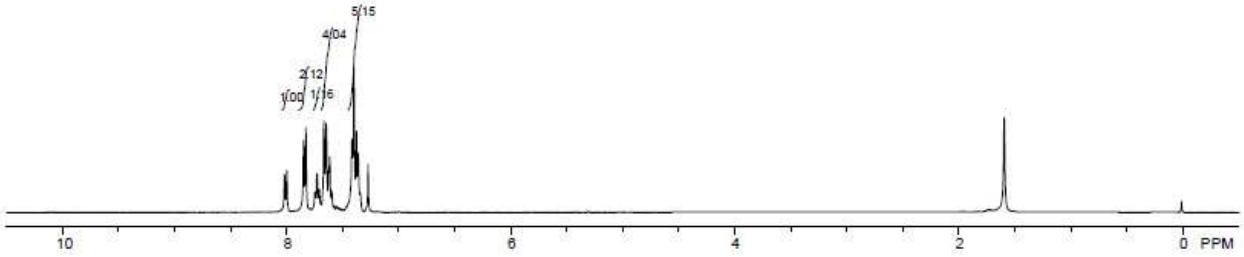
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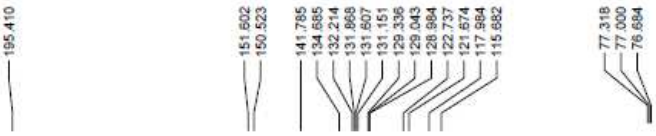
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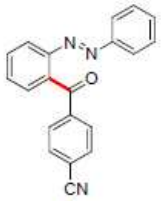
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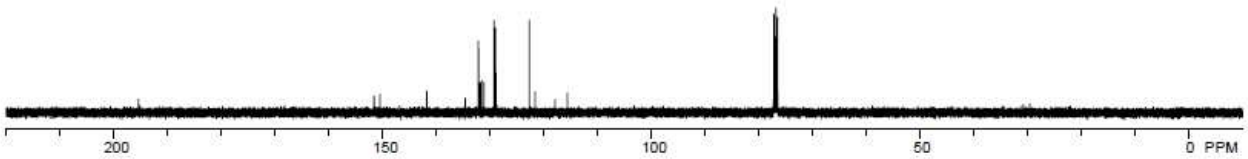
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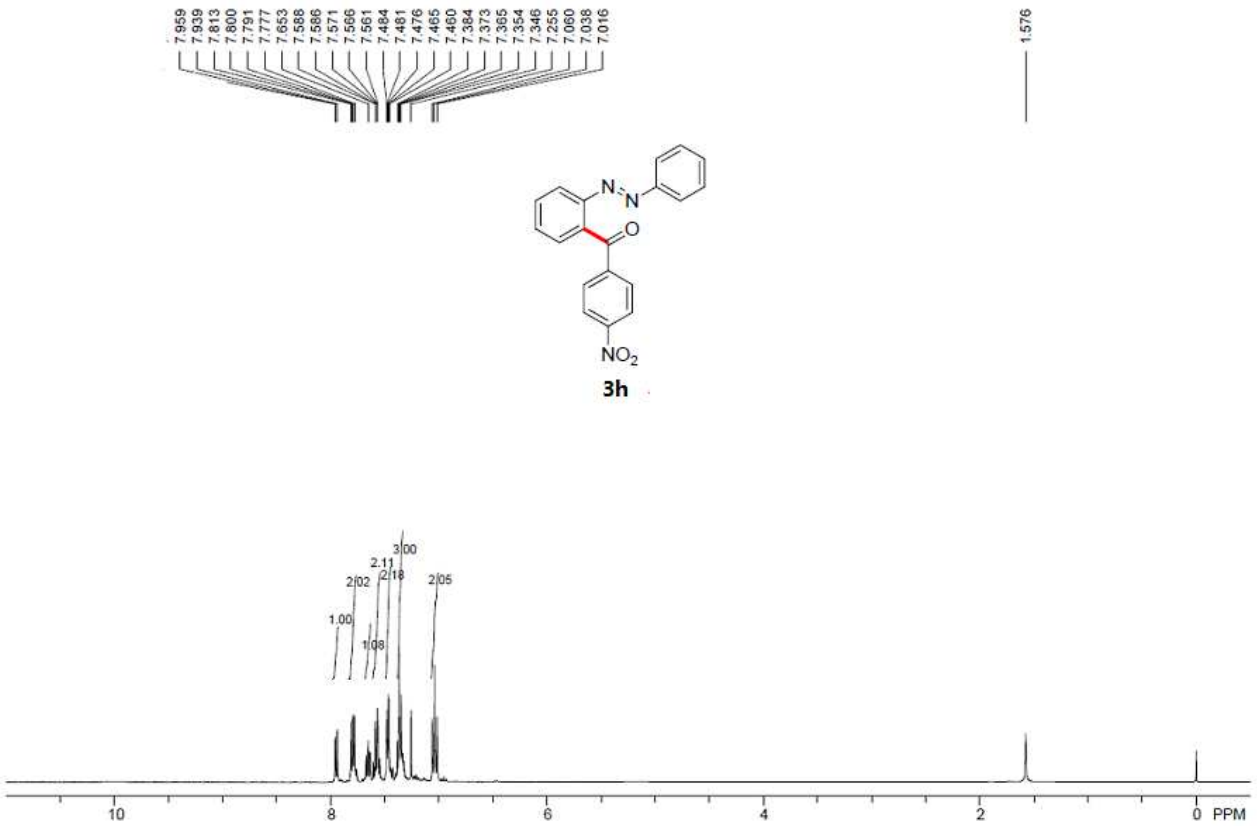
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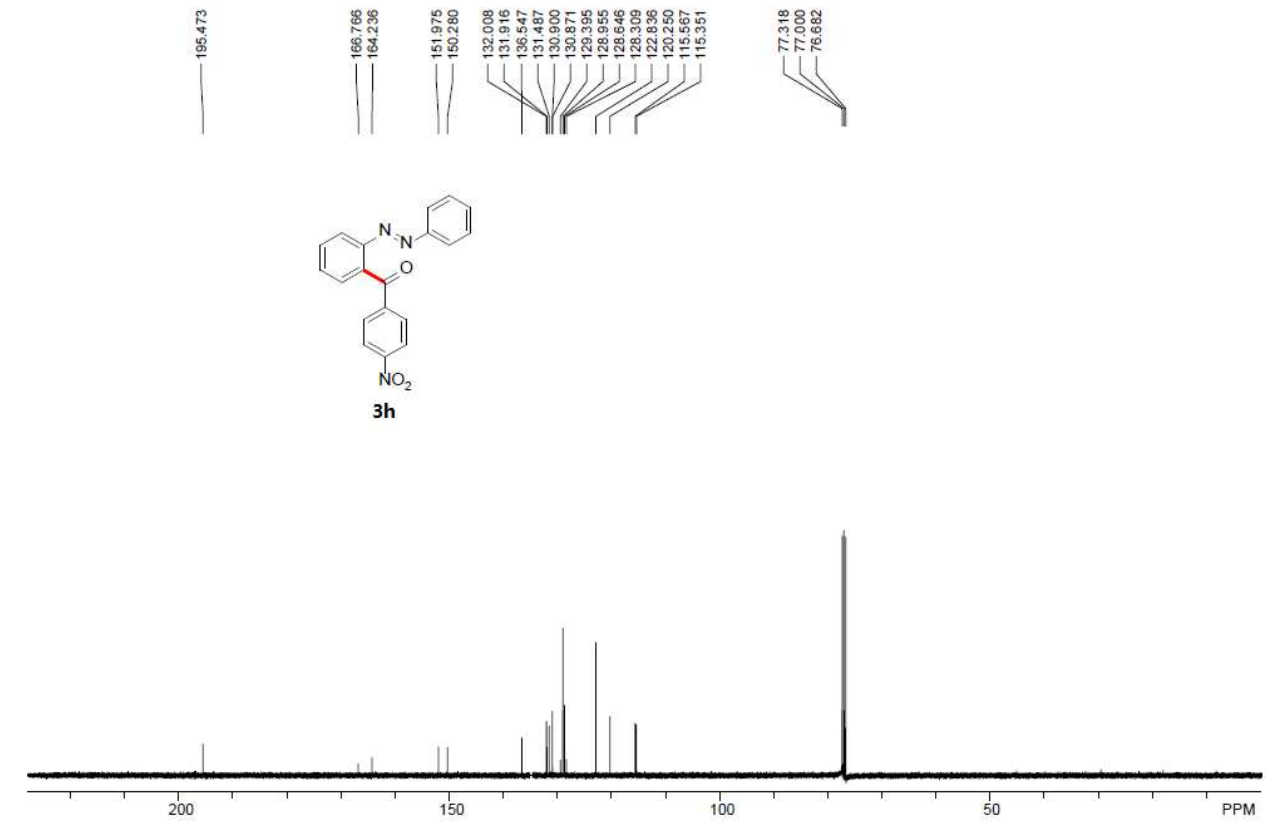
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NO2
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100
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PPM


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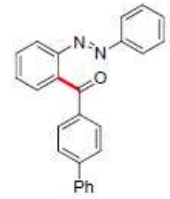
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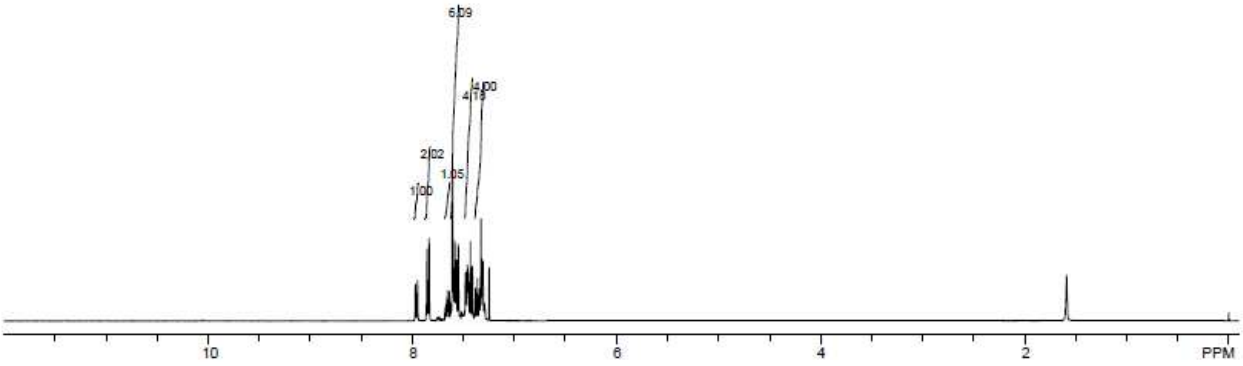
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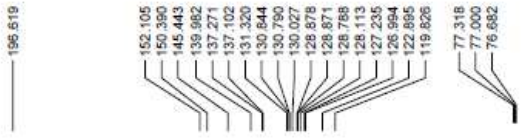
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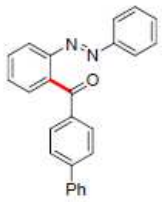
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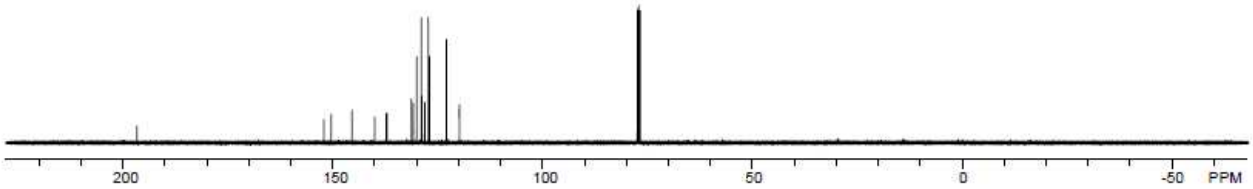
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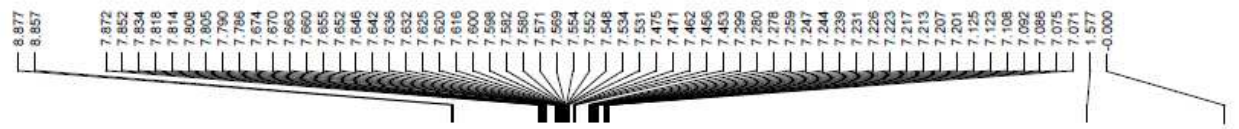
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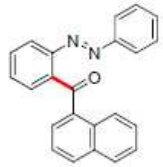

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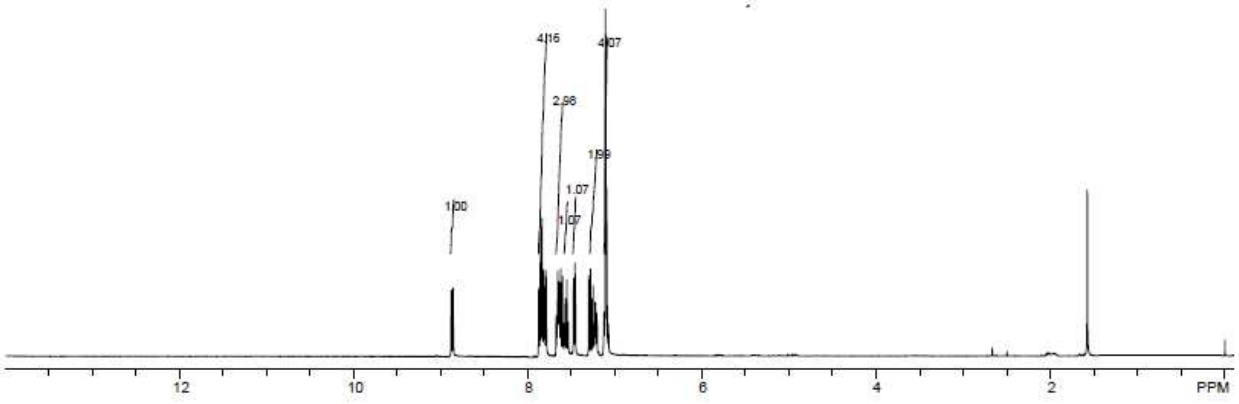
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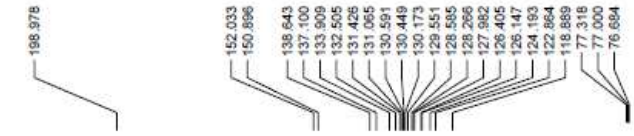
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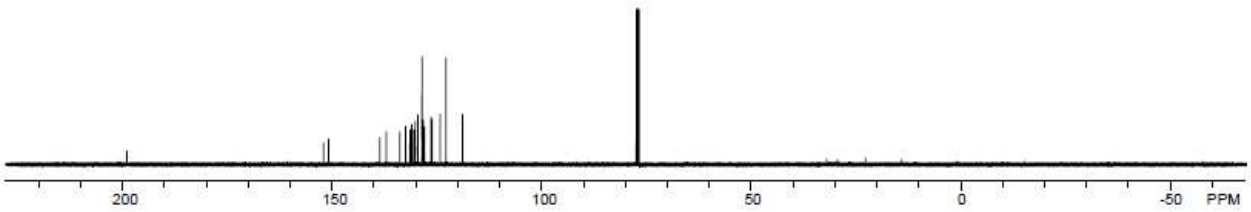
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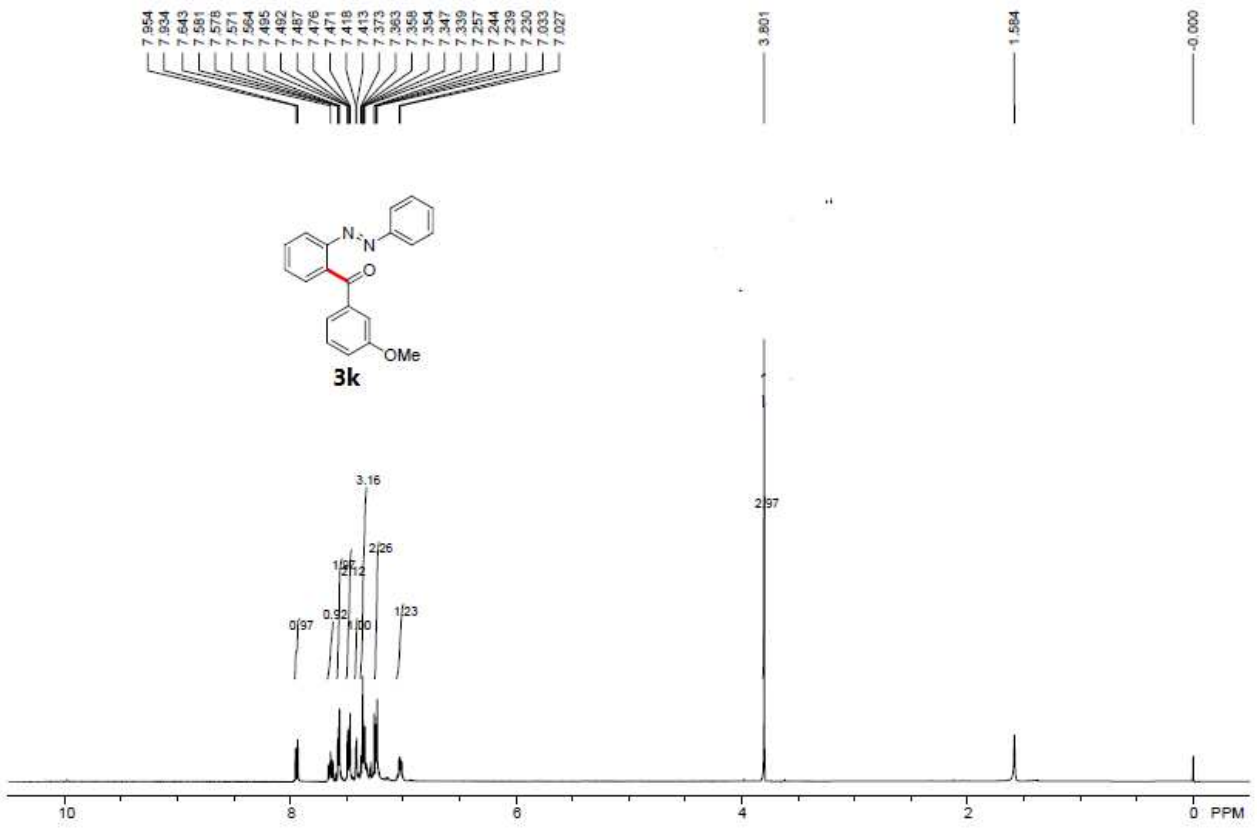
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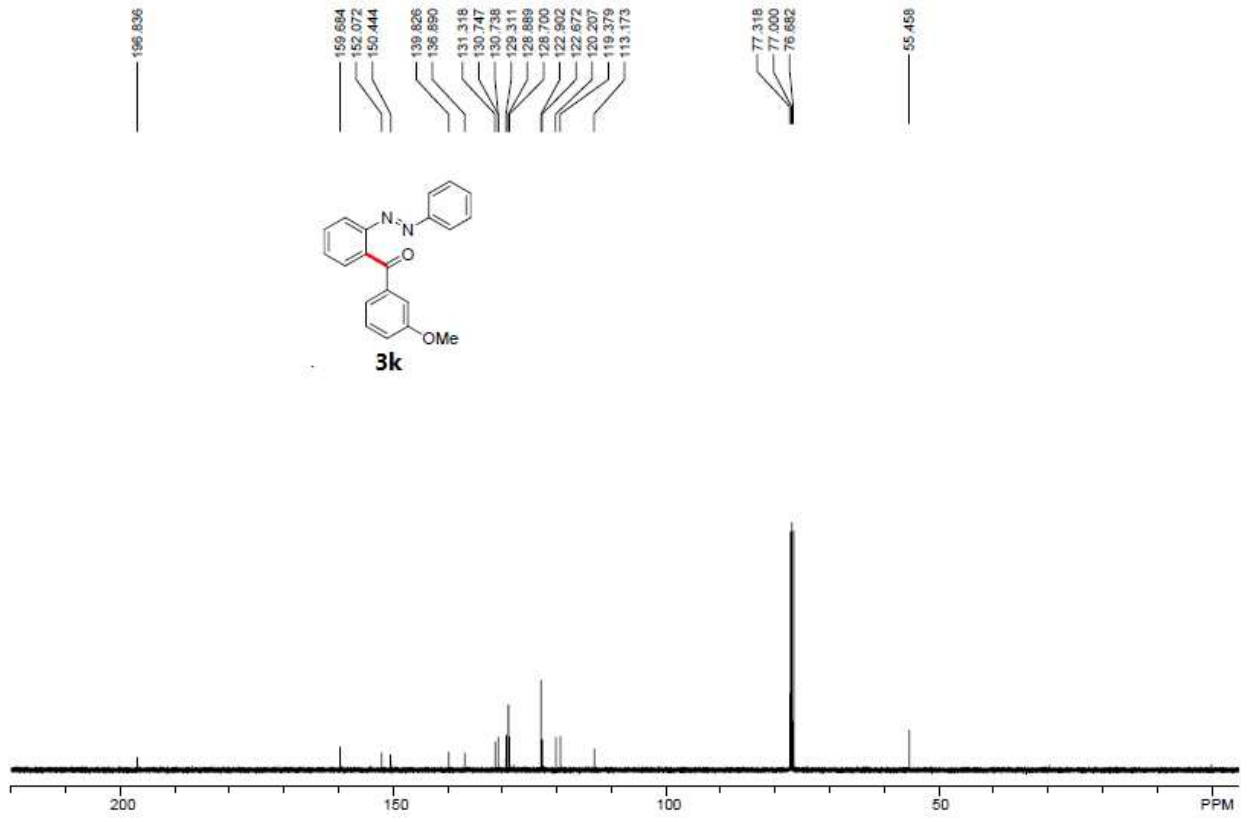
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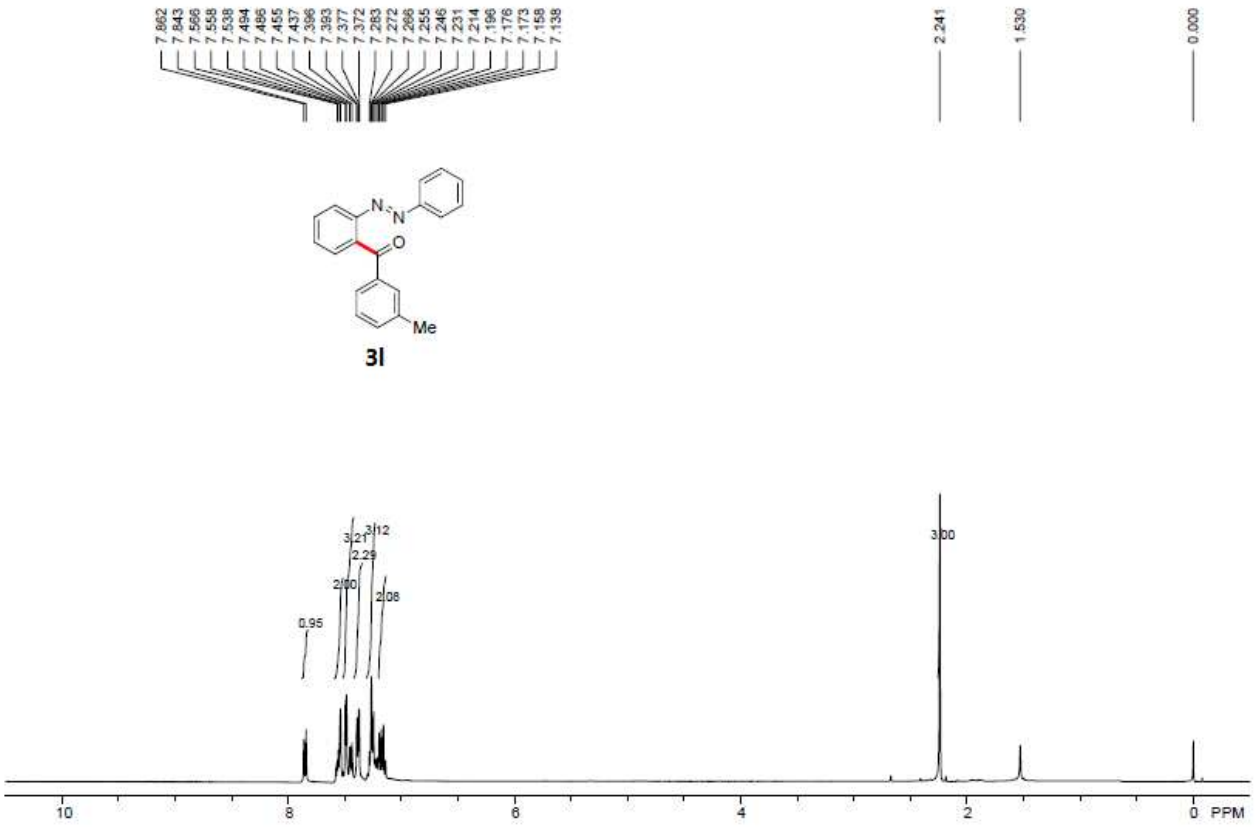
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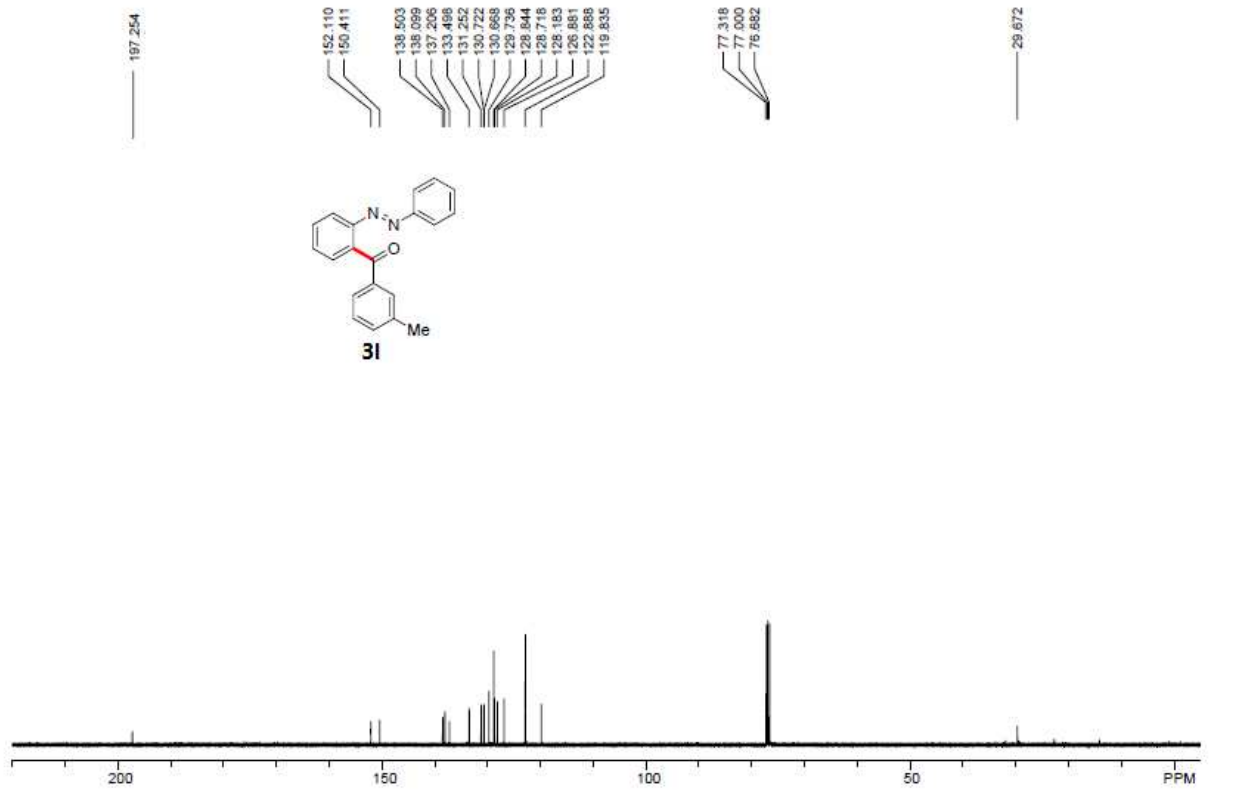
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3l


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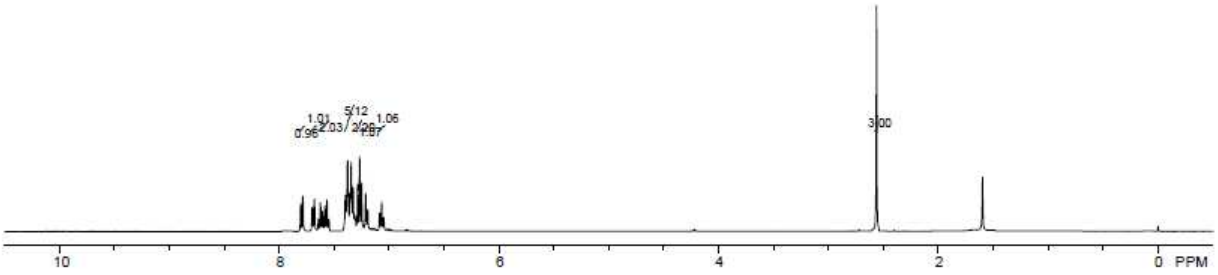
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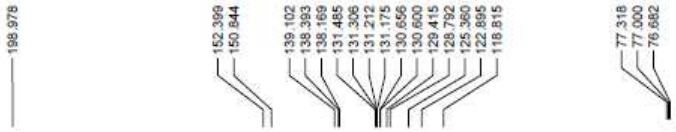
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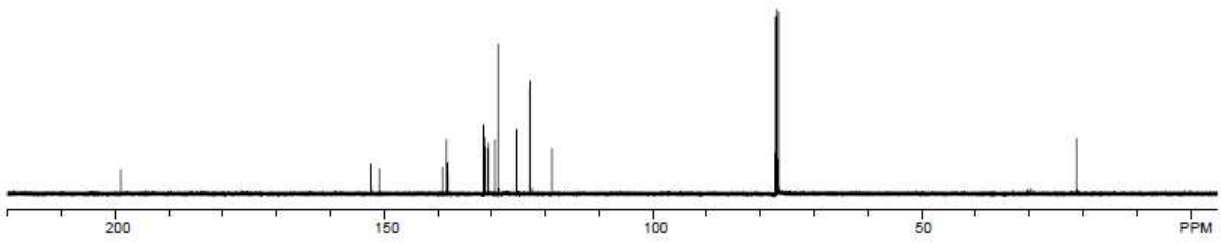
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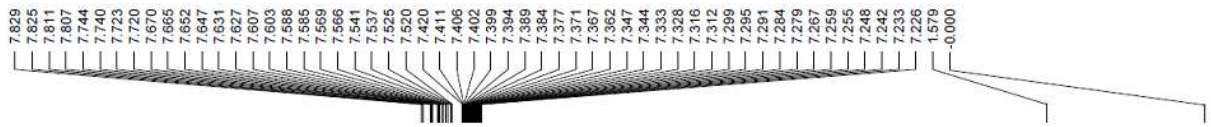
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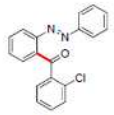

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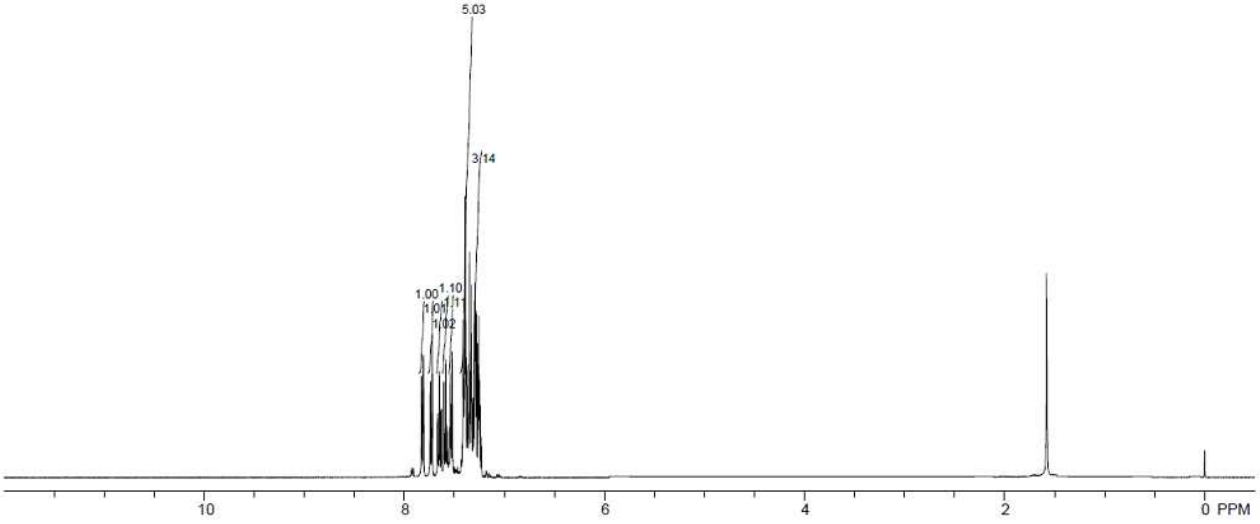
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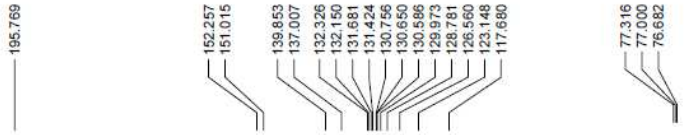
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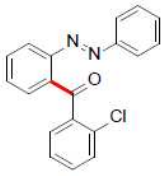

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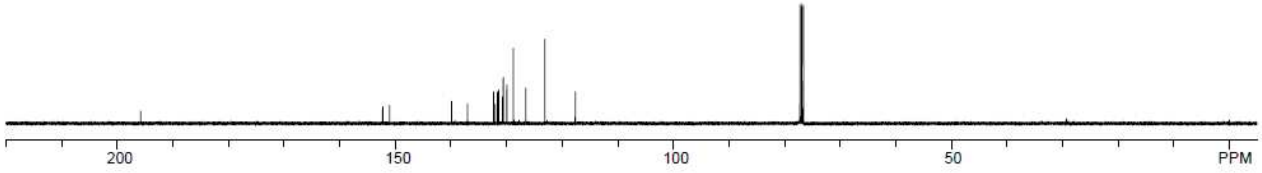
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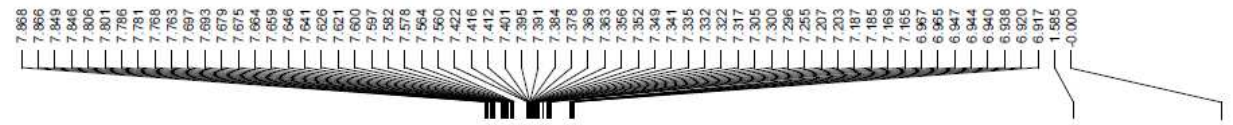
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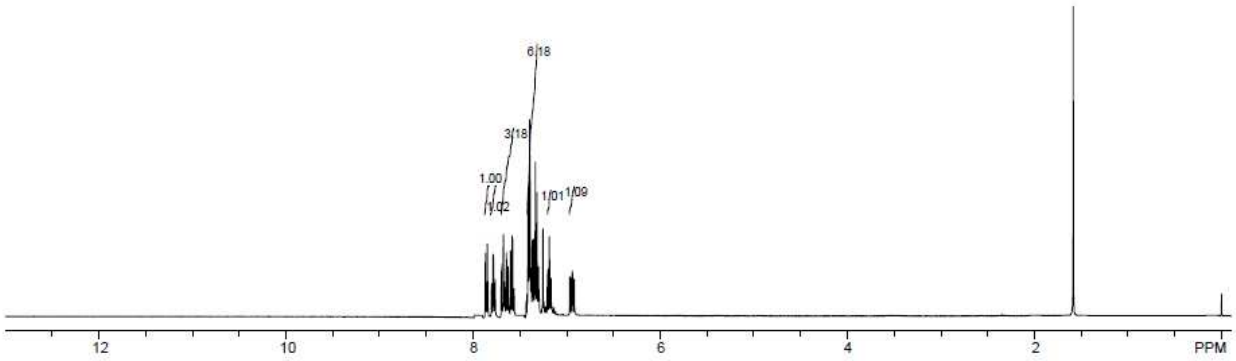
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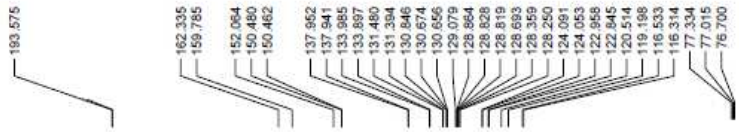
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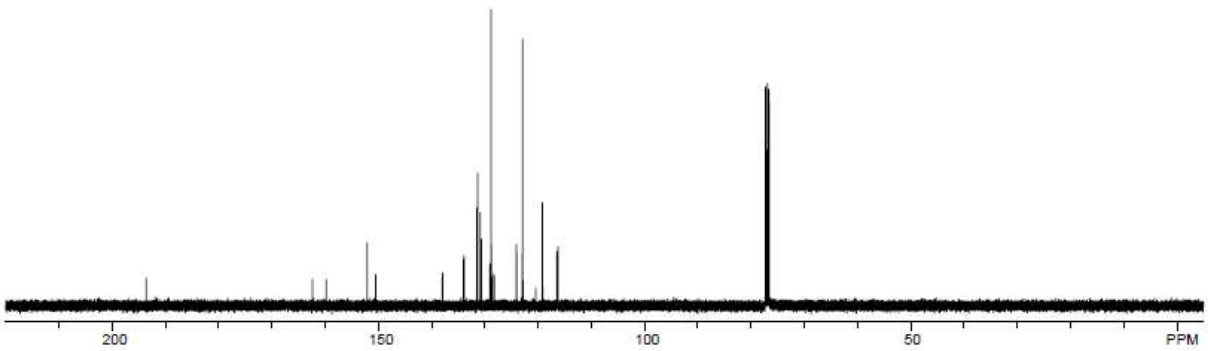
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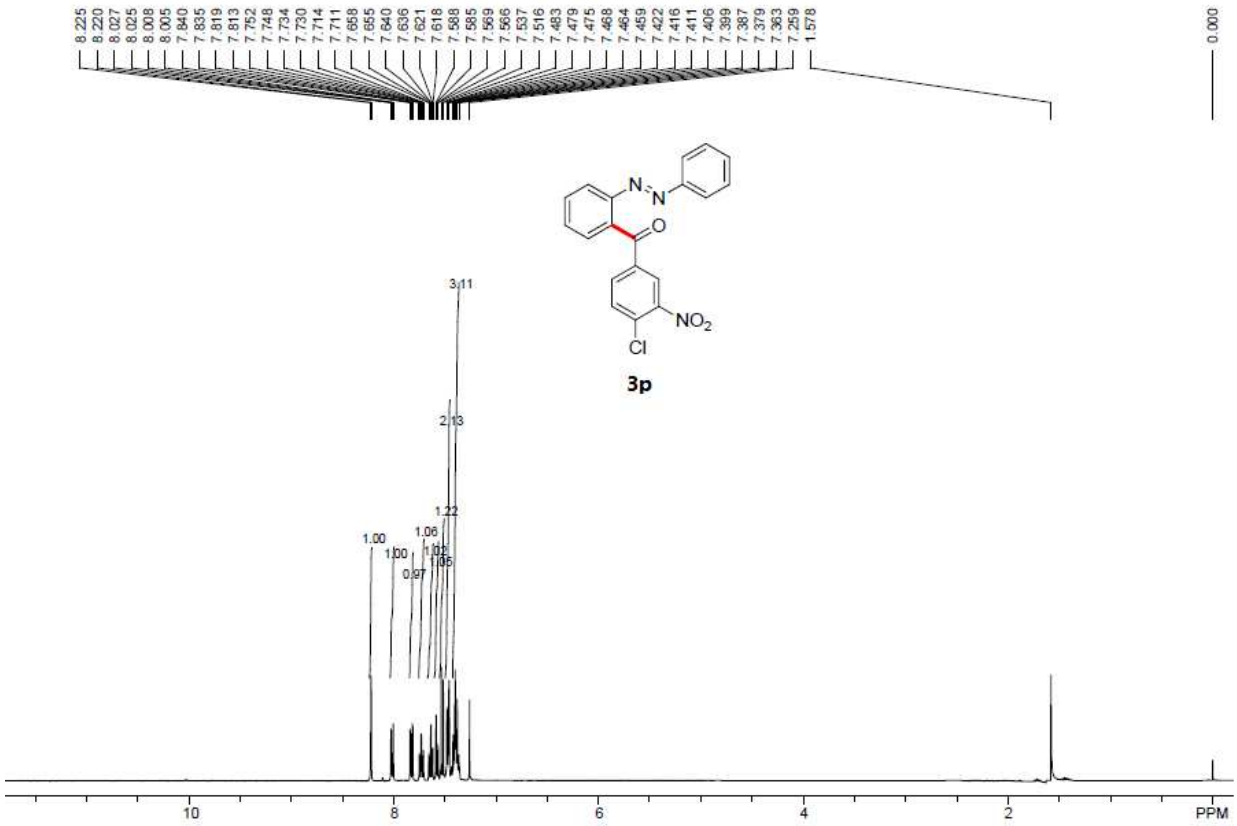
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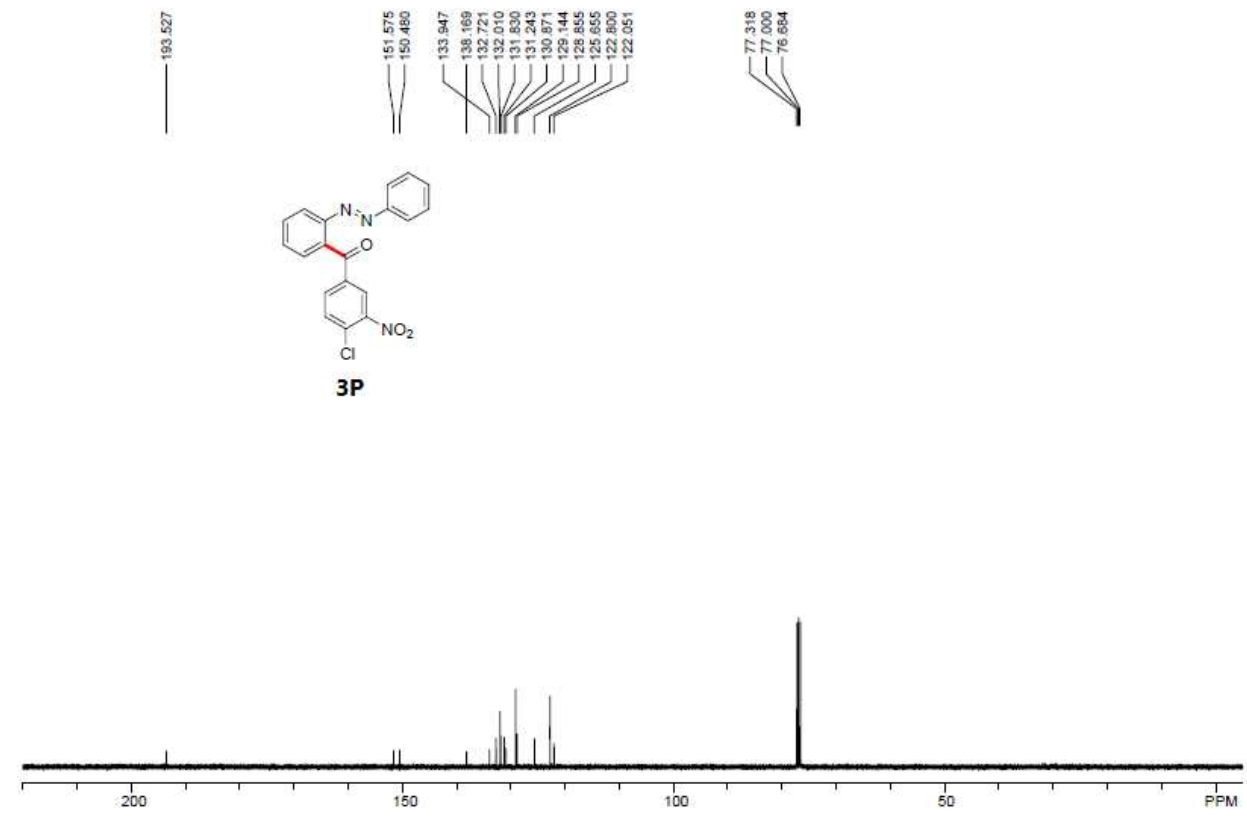
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100
50
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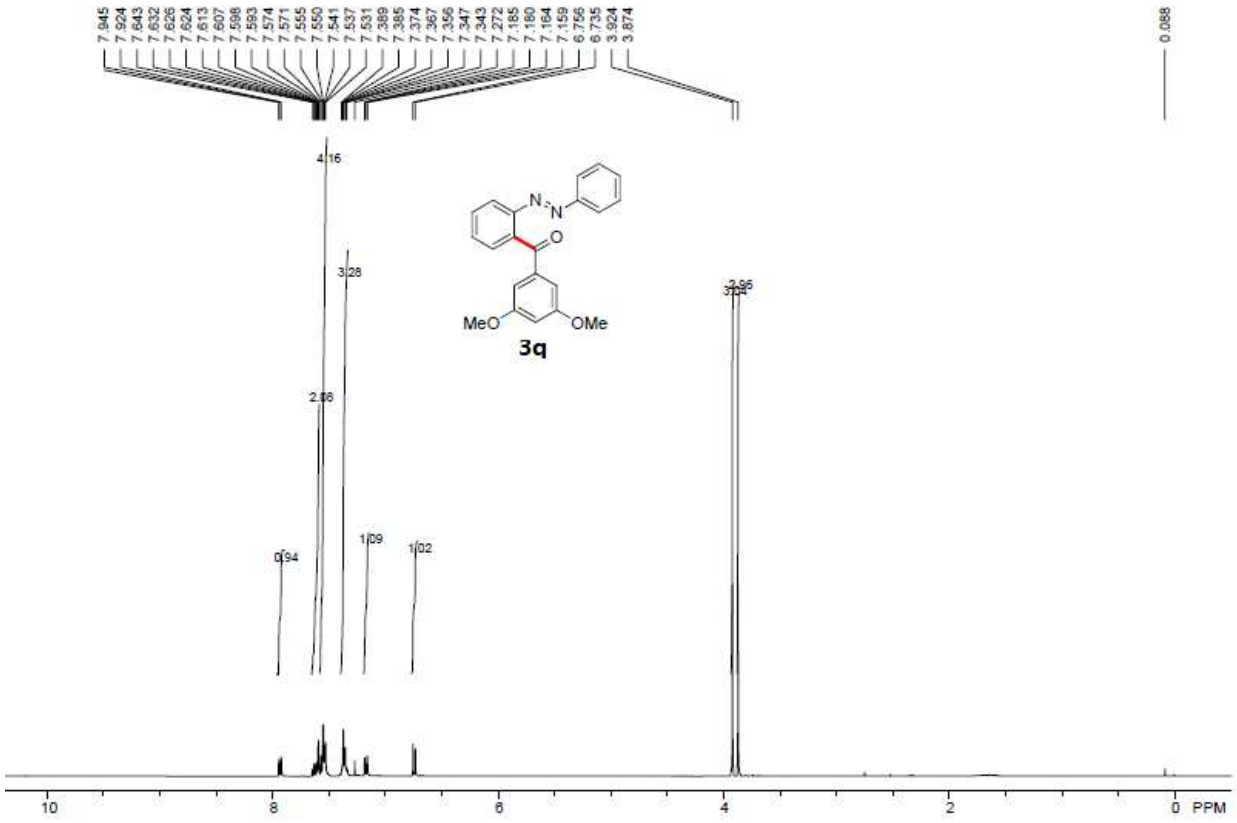
3P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7.550
7.541
7.537
7.531
7.389
7.385
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7.367
7.356
7.347
7.343
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7.180
7.164
7.159
6.756
6.735
3.924
3.874
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3q
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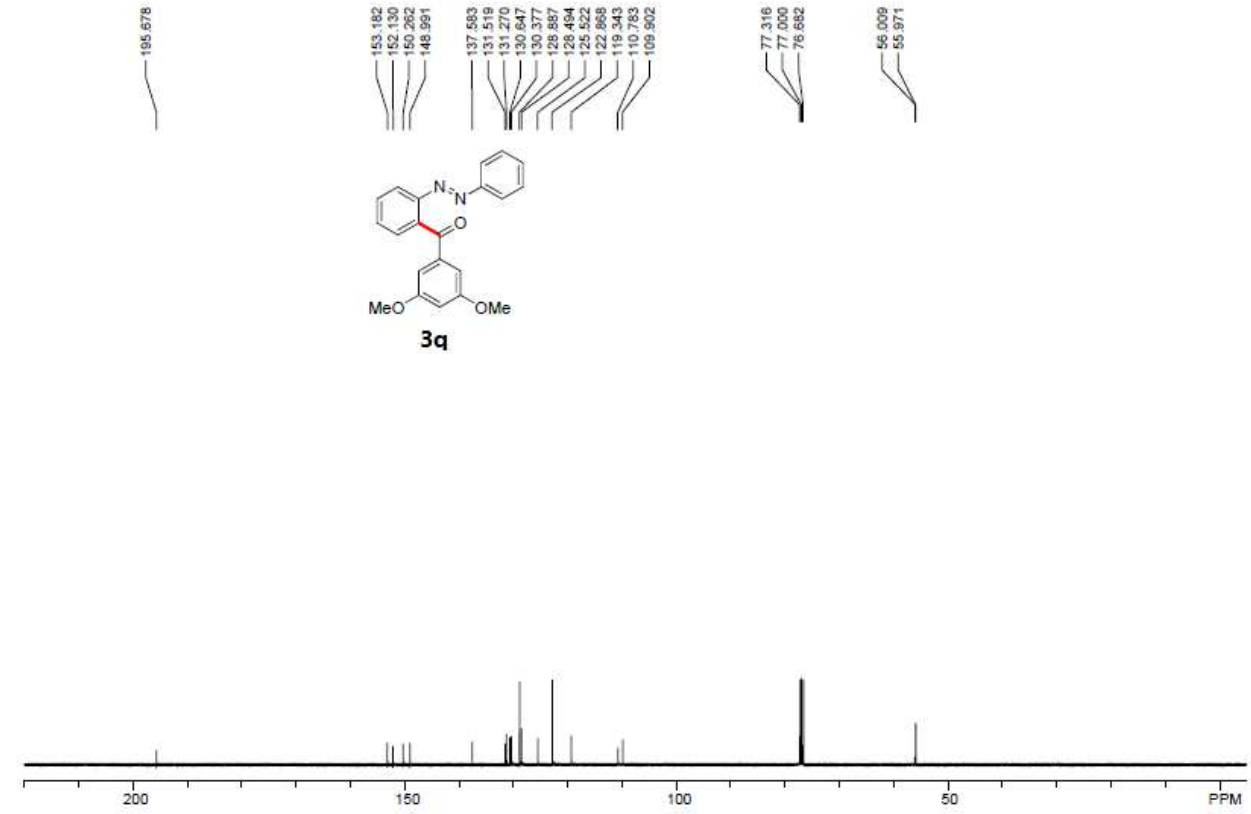
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131.270
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OMe
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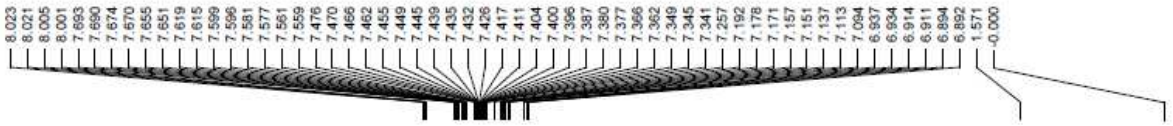
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7.599
7.596
7.581
7.577
7.561
7.559
7.476
7.470
7.466
7.462
7.455
7.449
7.445
7.439
7.435
7.432
7.426
7.417
7.411
7.404
7.400
7.396
7.387
7.380
7.377
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7.362
7.349
7.345
7.341
7.257
7.192
7.178
7.171
7.157
7.151
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6.892
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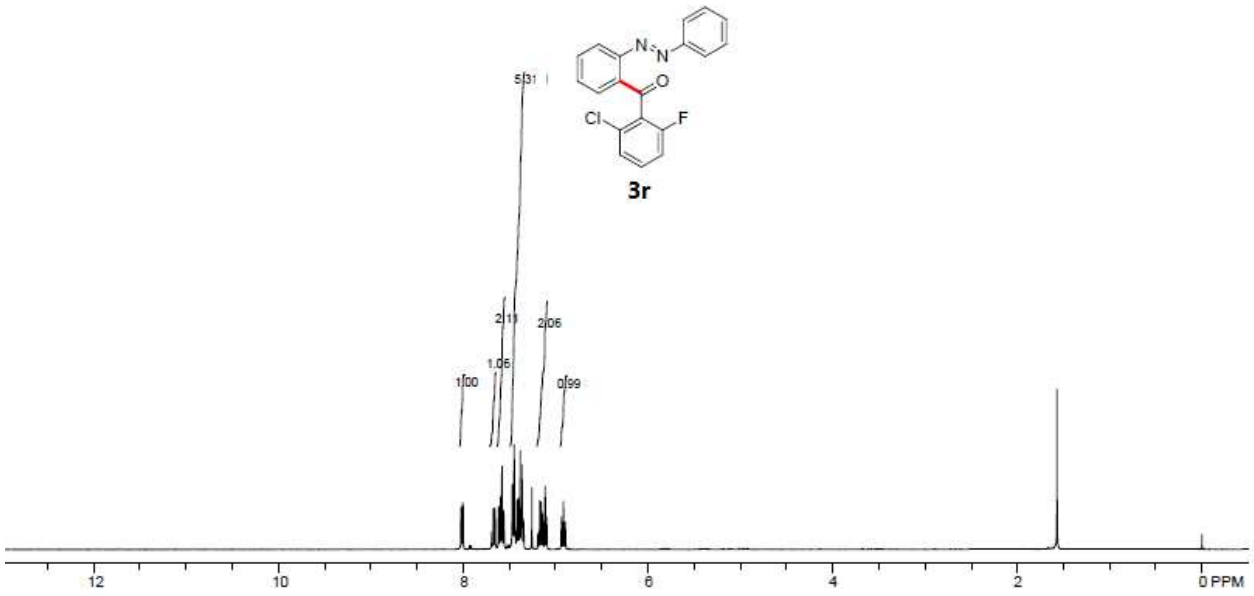
-0.000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10
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4
2
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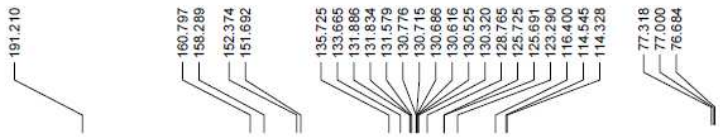
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131.886
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131.579
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130.715
130.686
130.616
130.525
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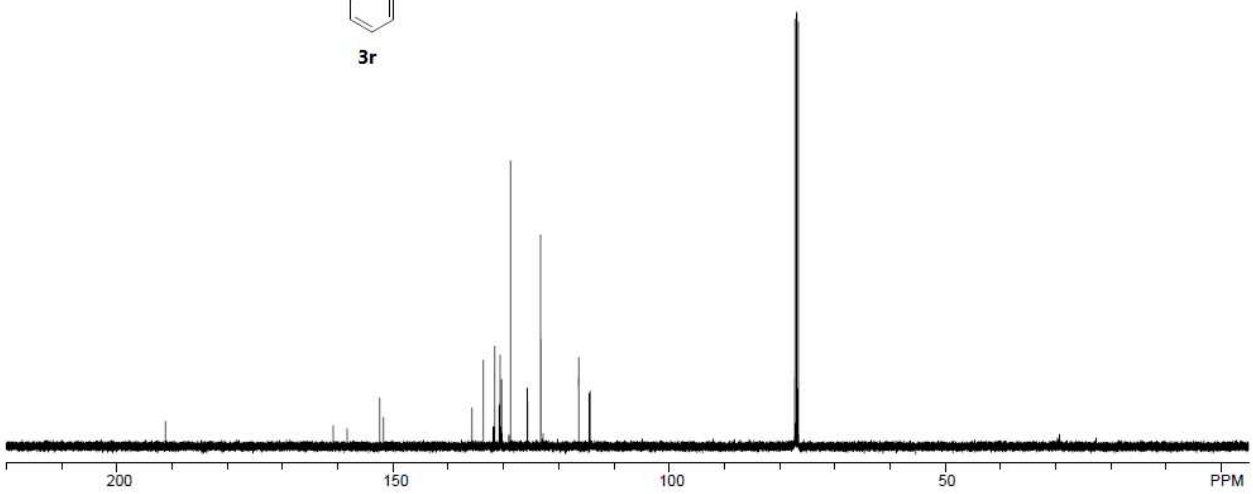
77.000
76.684

3r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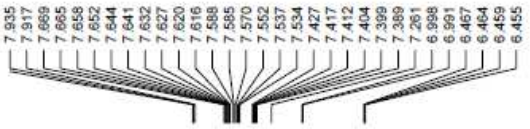
PPM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7.641
7.632
7.627
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7.616
7.588
7.585
7.570
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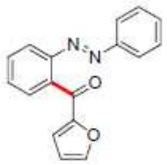

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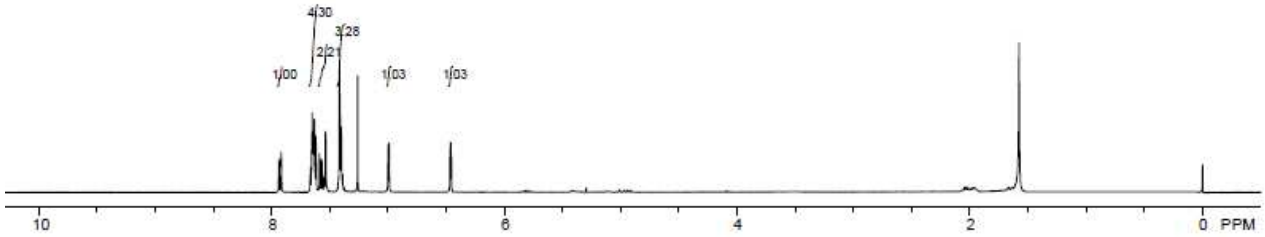
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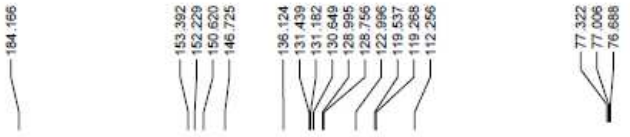
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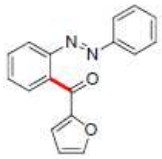
76.688



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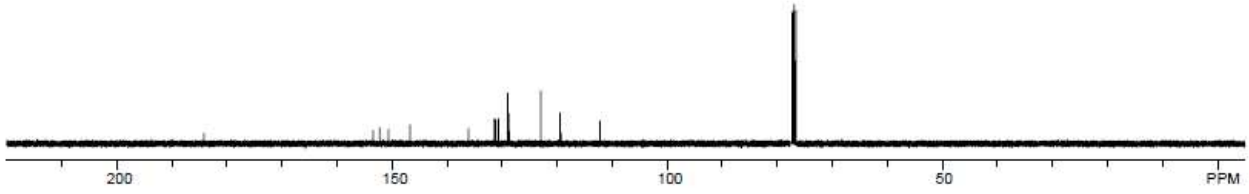
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PPM


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7.503
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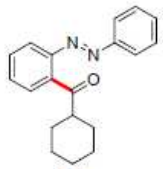

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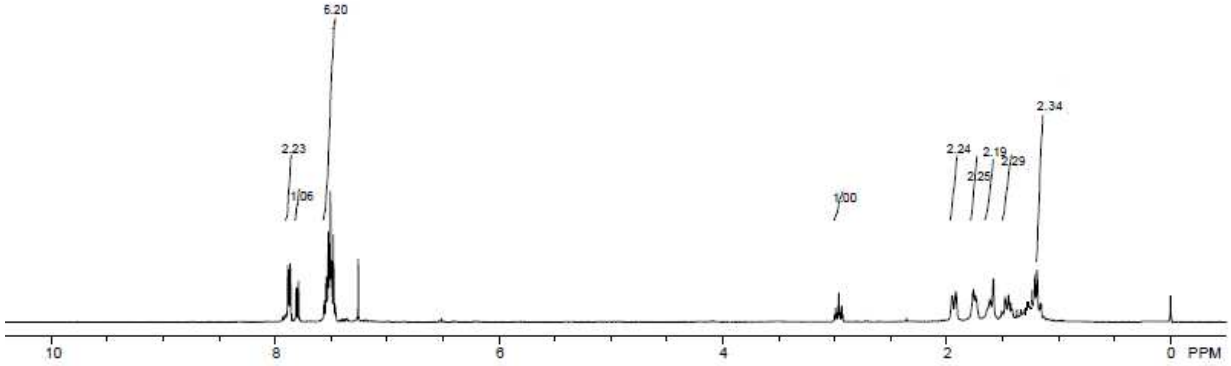
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10
8
6
4
2
0 PPM


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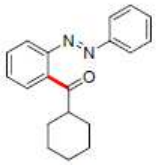
25.752



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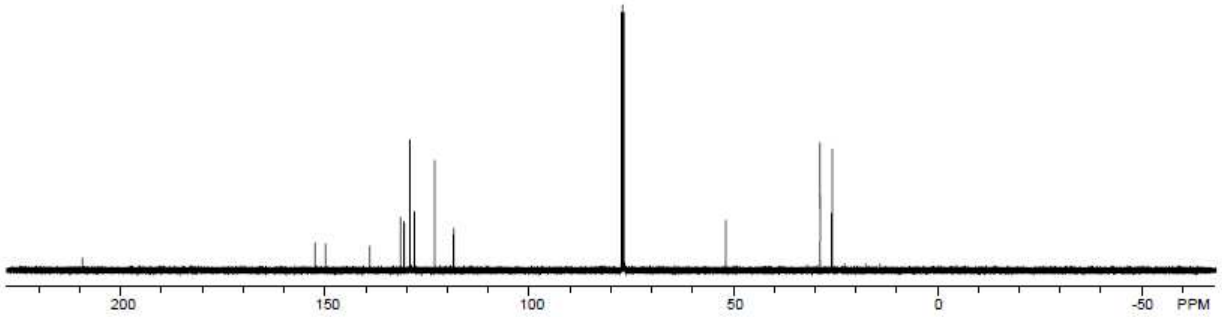
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100
50
0
-50 PPM


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7.528
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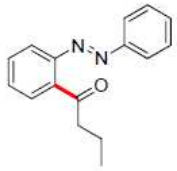

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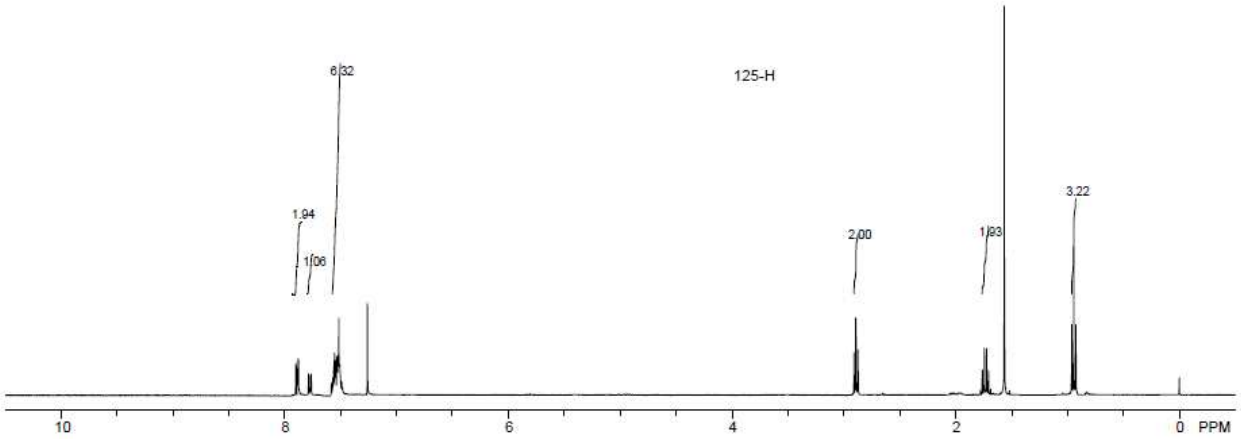
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10
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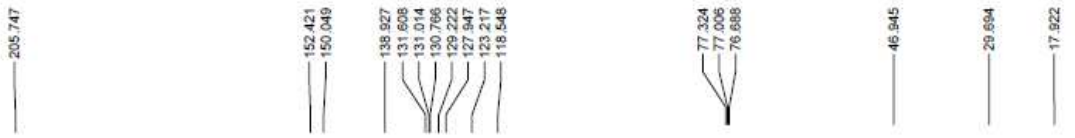
4
2
0
PPM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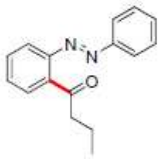
29.694
17.922



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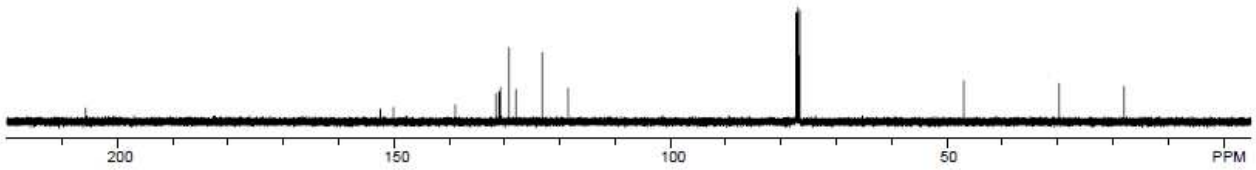
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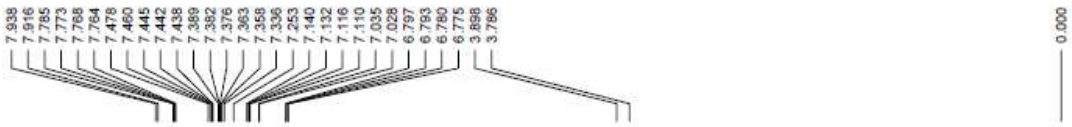
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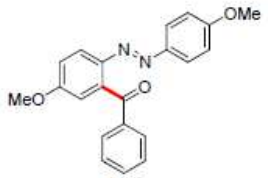
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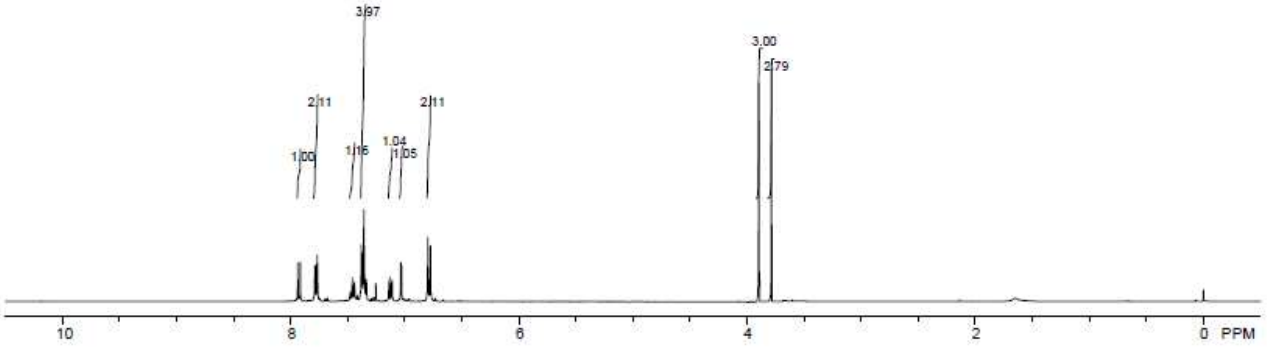
3v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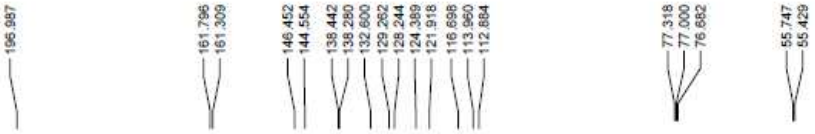
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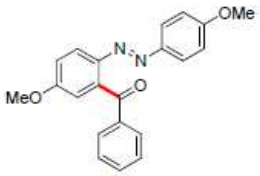
55.429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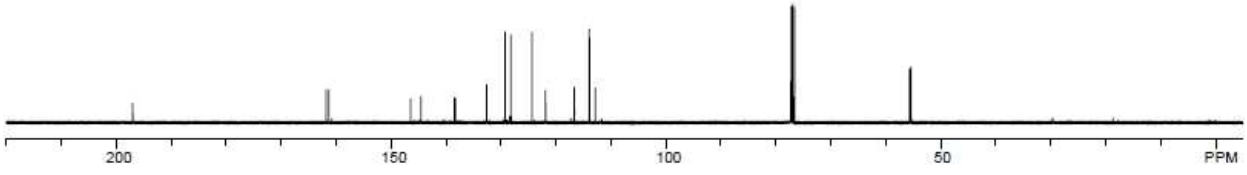
3v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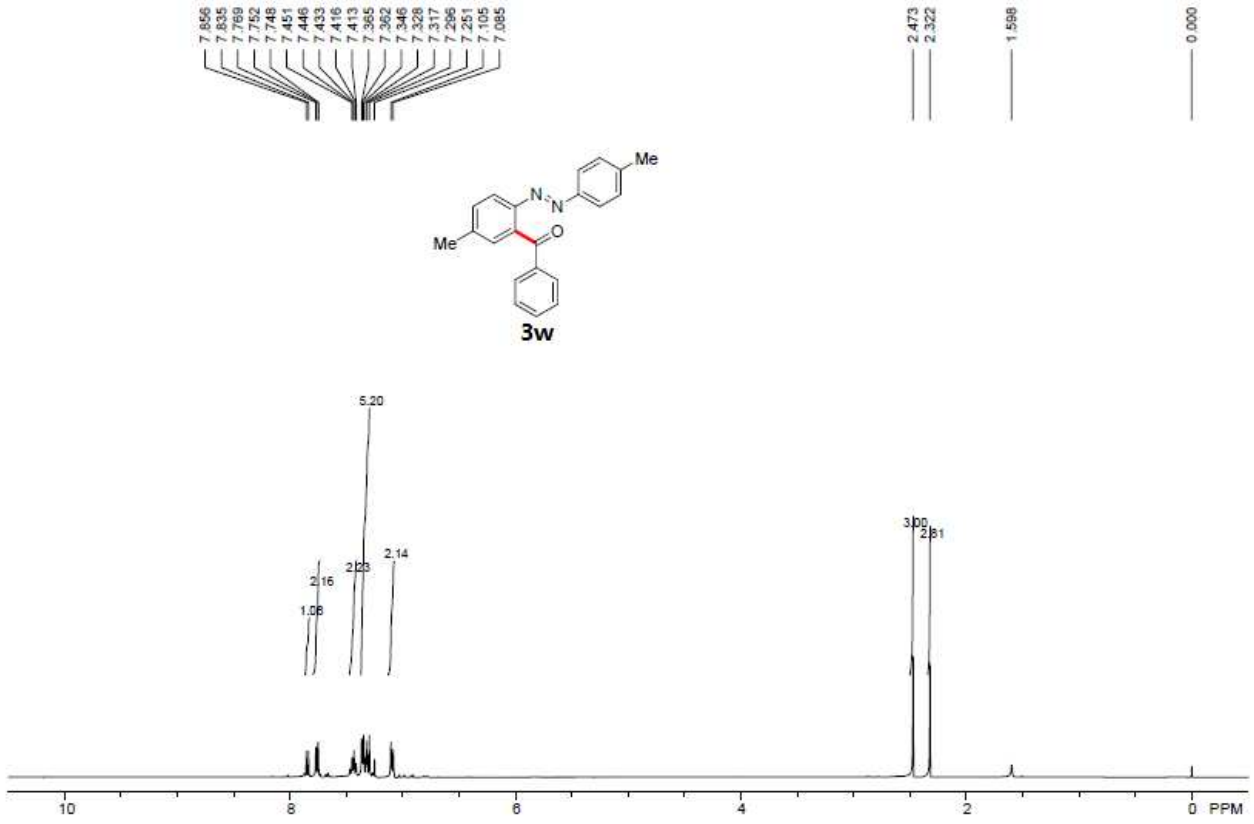
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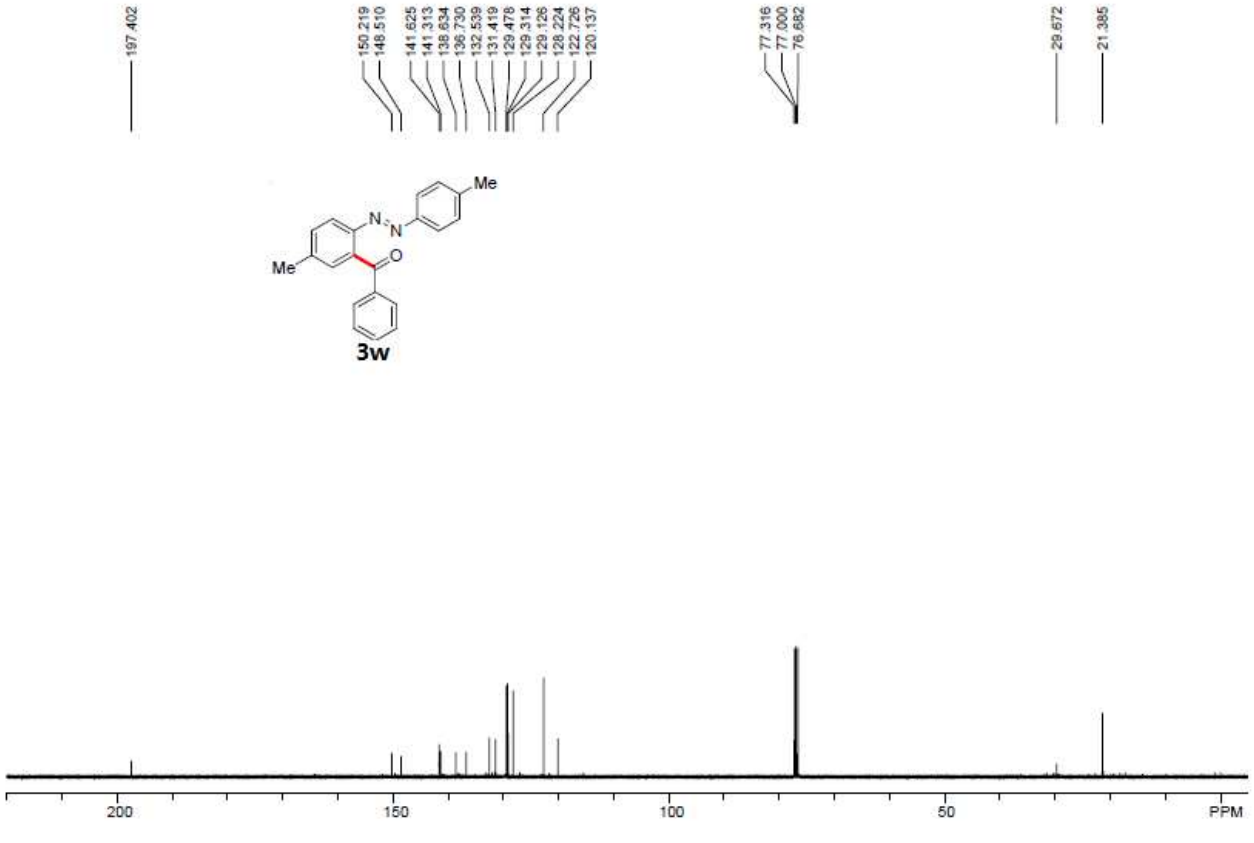
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120.137
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29.672
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Me
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Me
O
3w
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150
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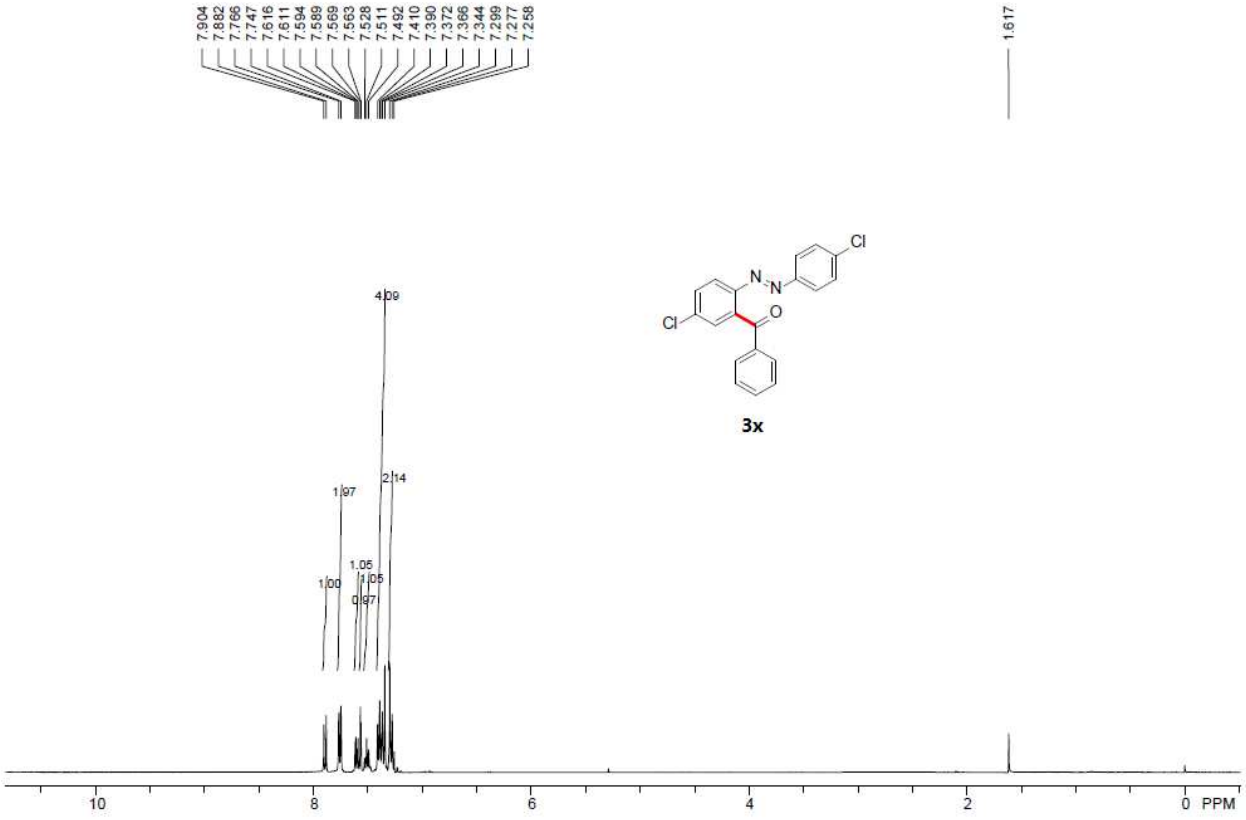
50
PPM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7.344
7.299
7.277
7.258
1.617
4.09
2.14
1.97
1.05
1.05
1.00
0.97
Cl
N
N
O
Cl
3x
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8
6
4
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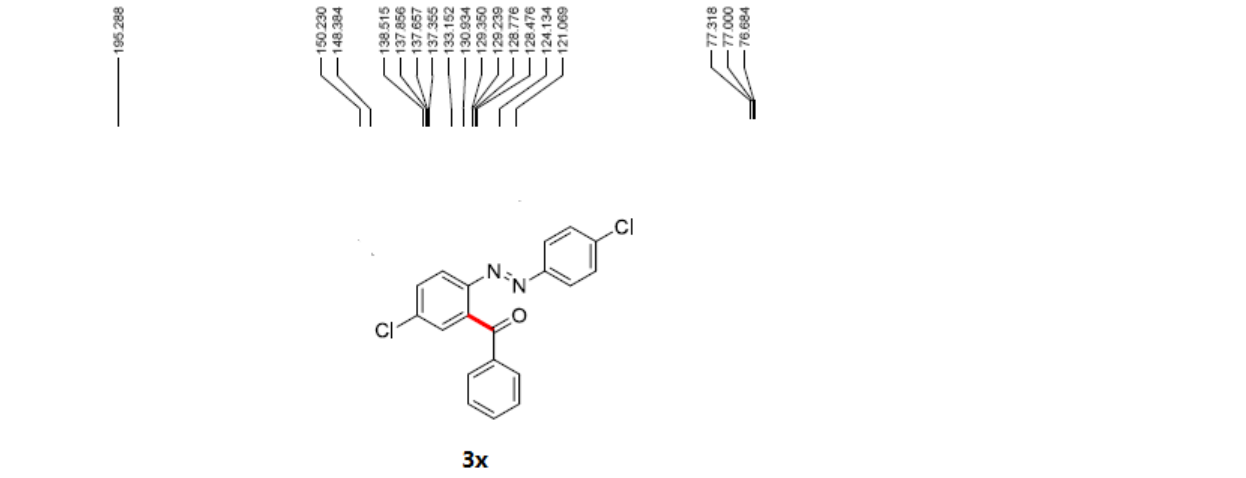
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N
O
Cl
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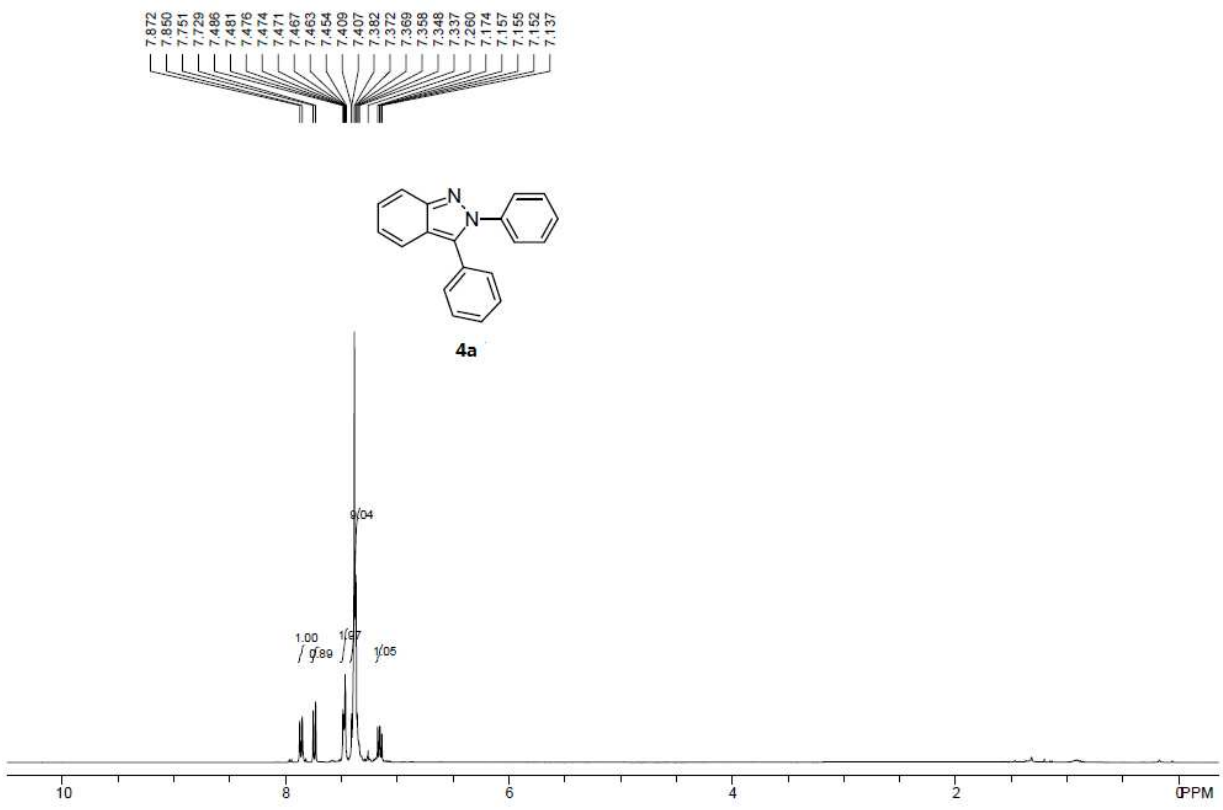
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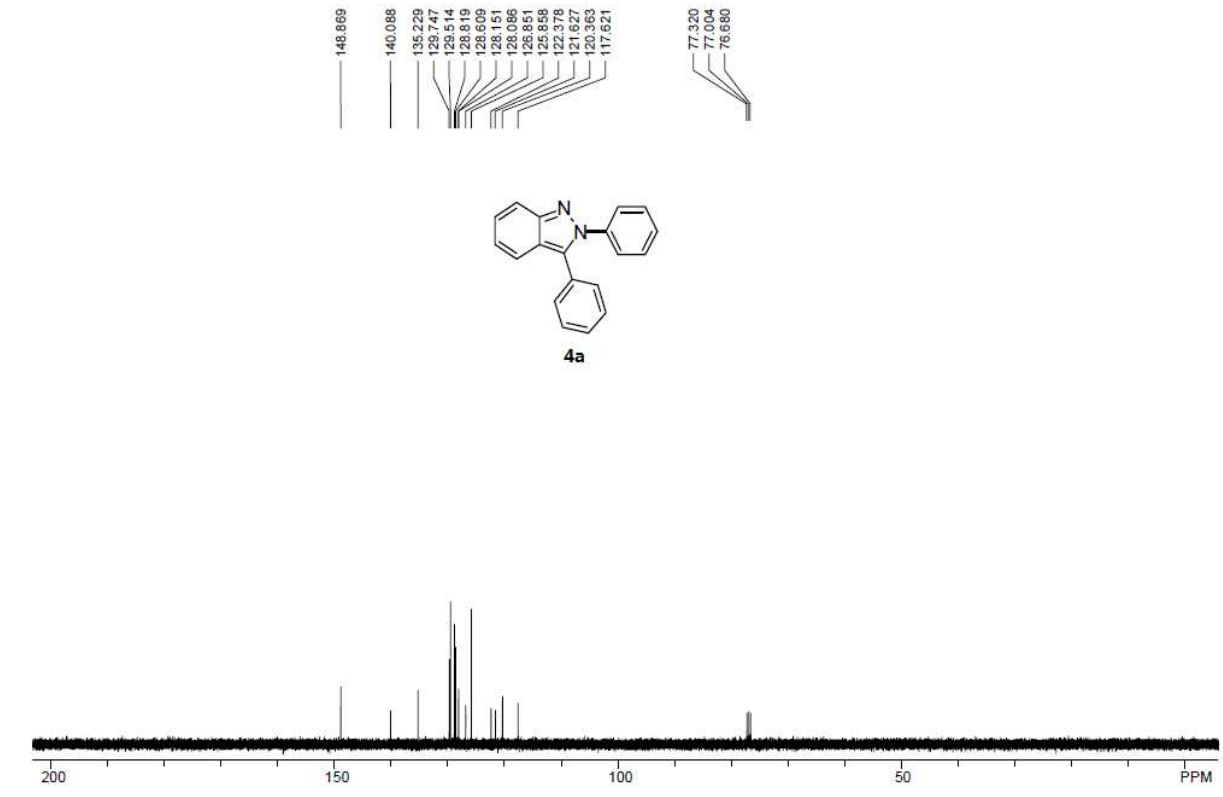
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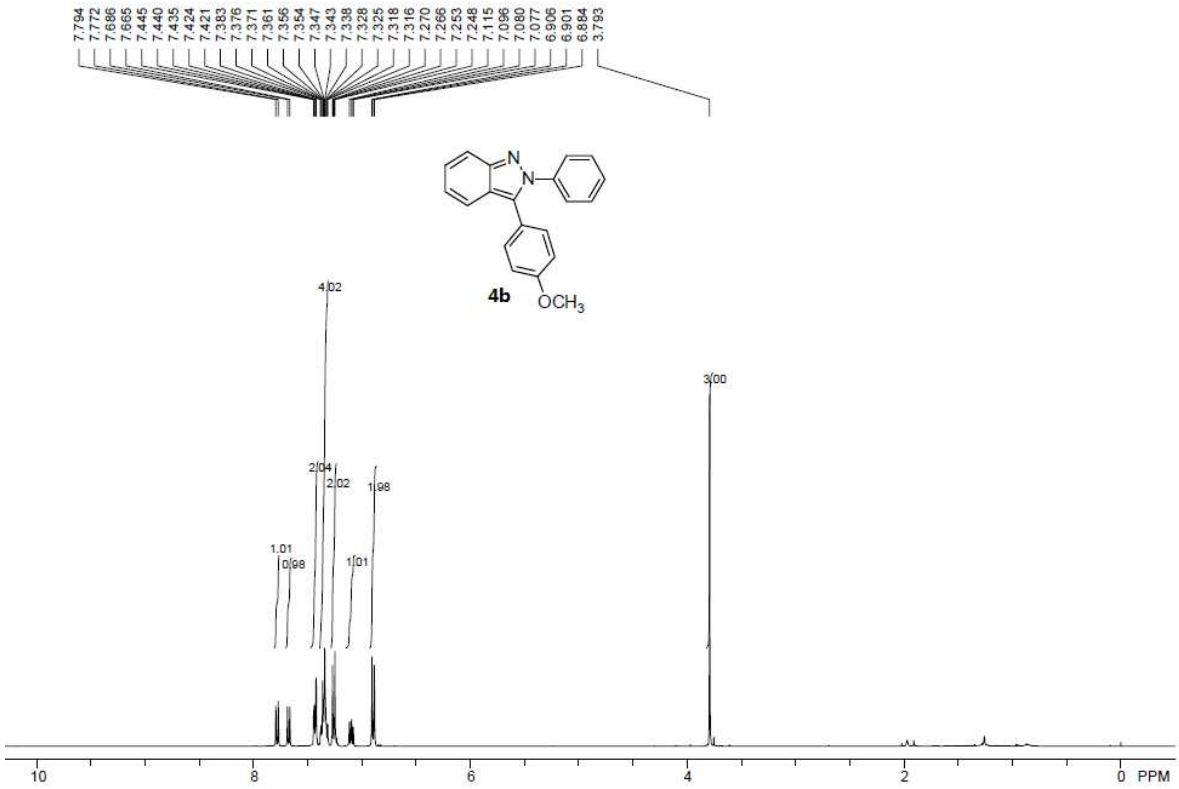
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OCH3
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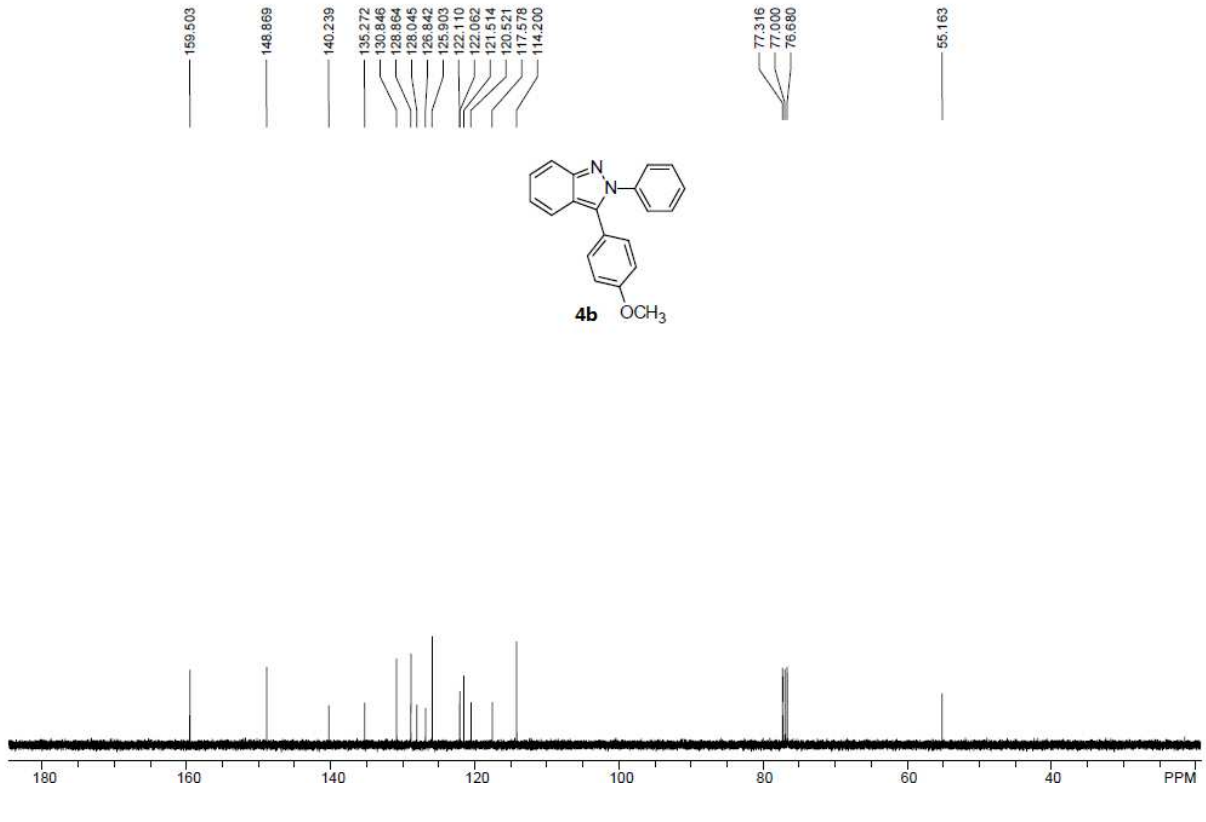
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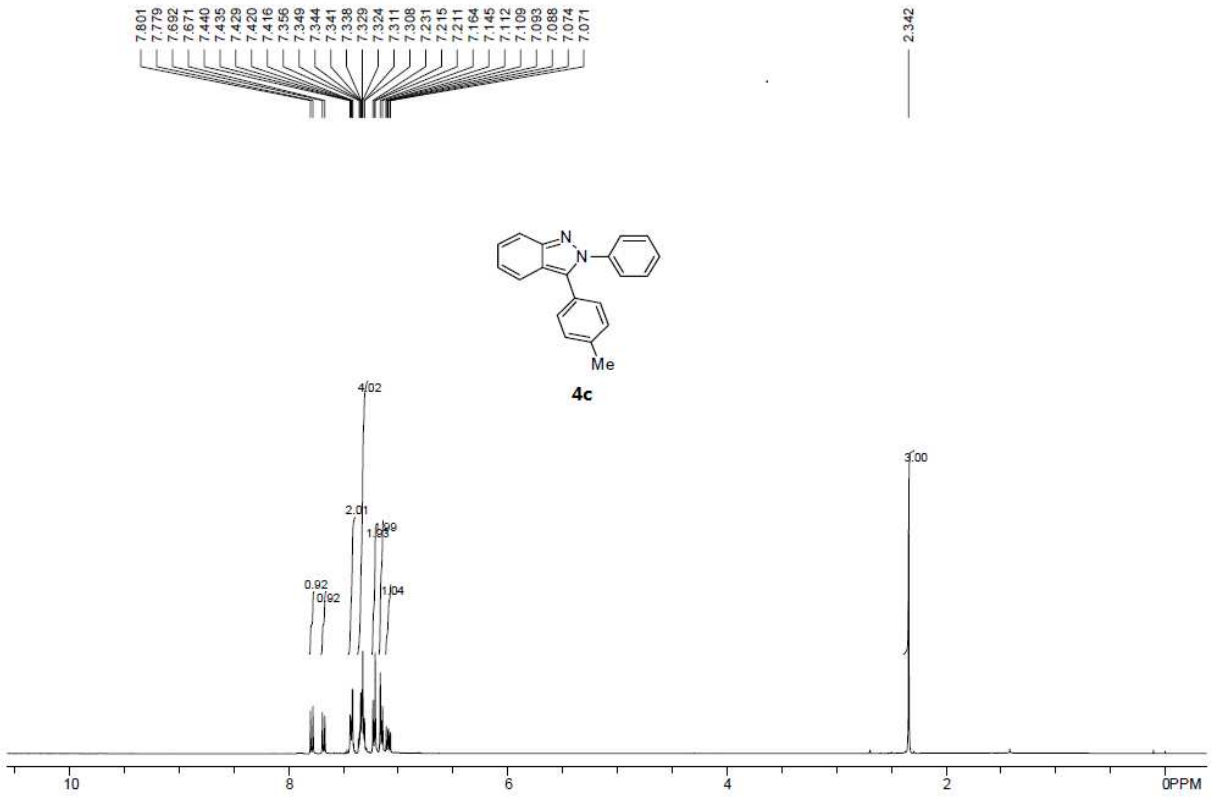
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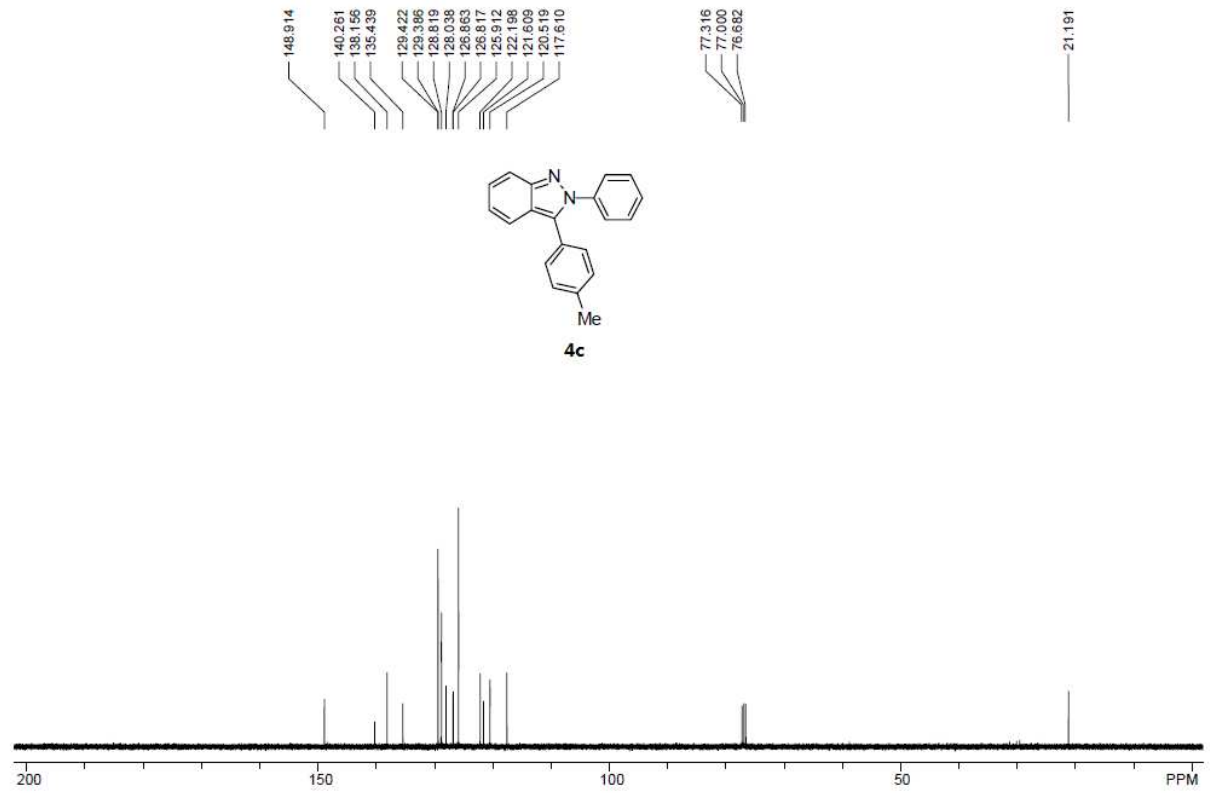
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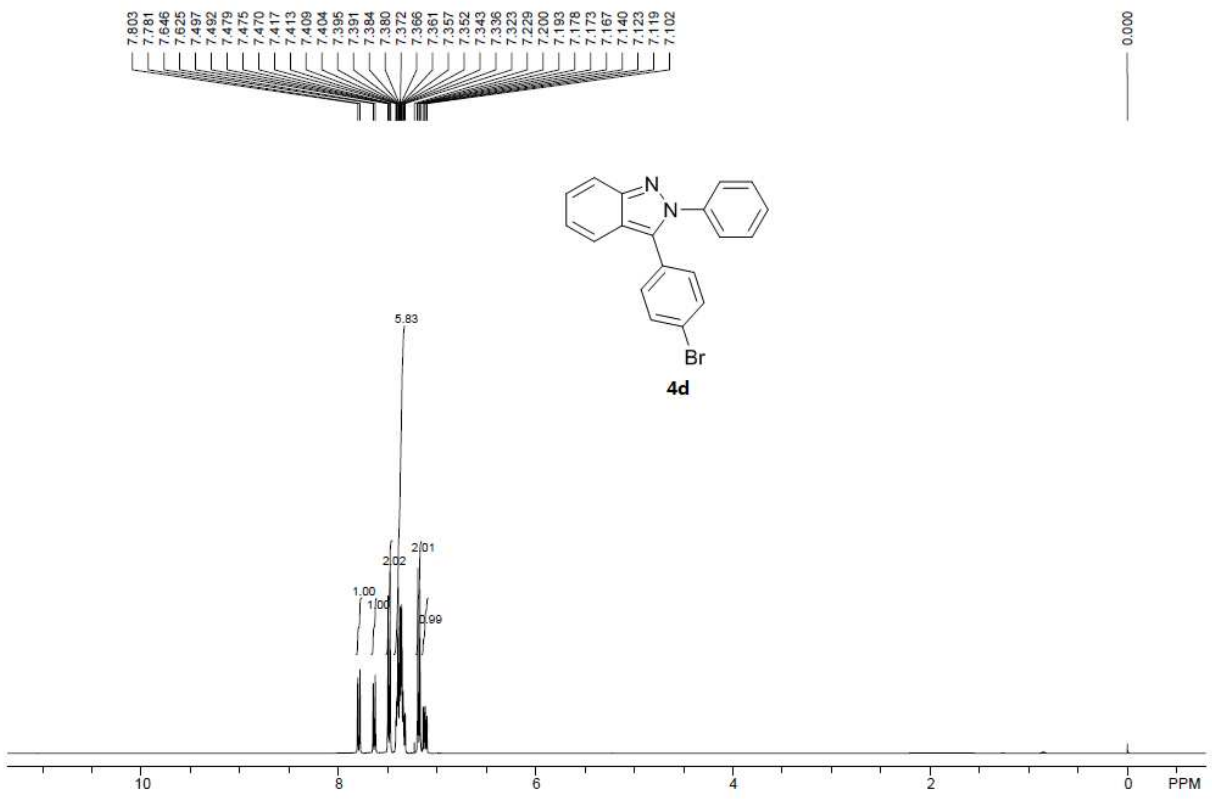
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7.391
7.384
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7.173
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7.119
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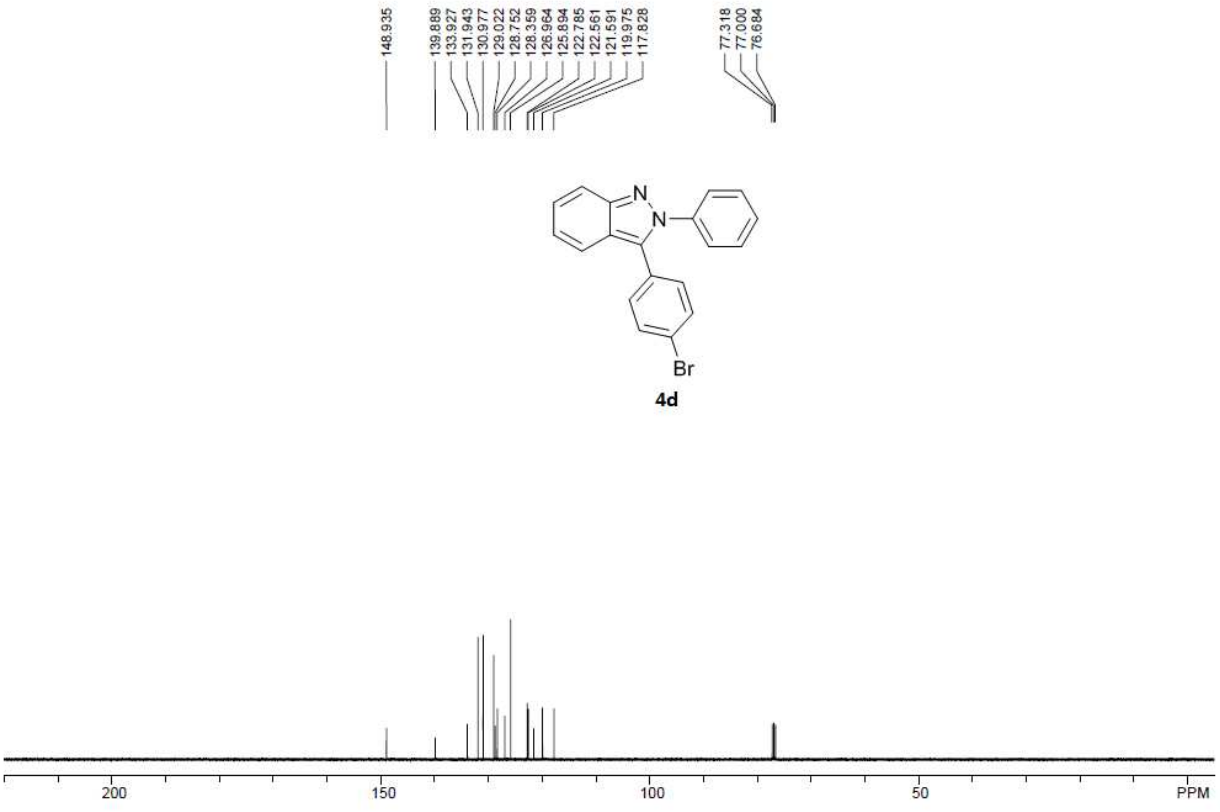
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122.561
121.591
119.975
117.828
77.318
77.000
76.684
N
N
Br
4d
200
150
100
50
PPM


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7.681
7.660
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7.420
7.415
7.407
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7.388
7.382
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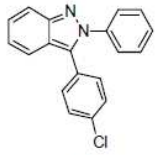
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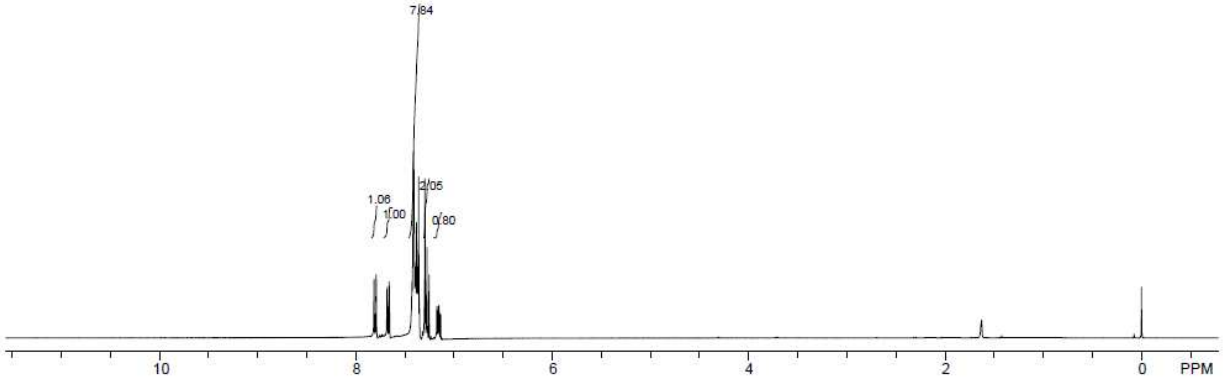
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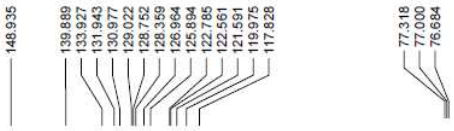
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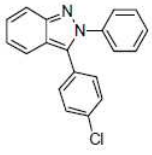
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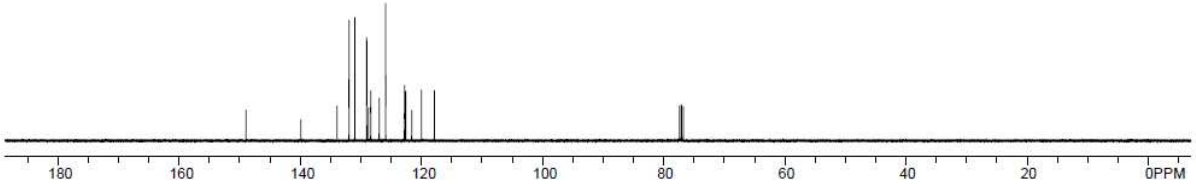
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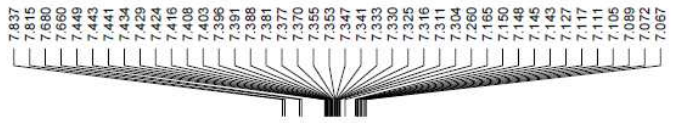
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7.403
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7.388
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7.355
7.353
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7.330
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7.316
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7.304
7.260
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7.067


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N
F

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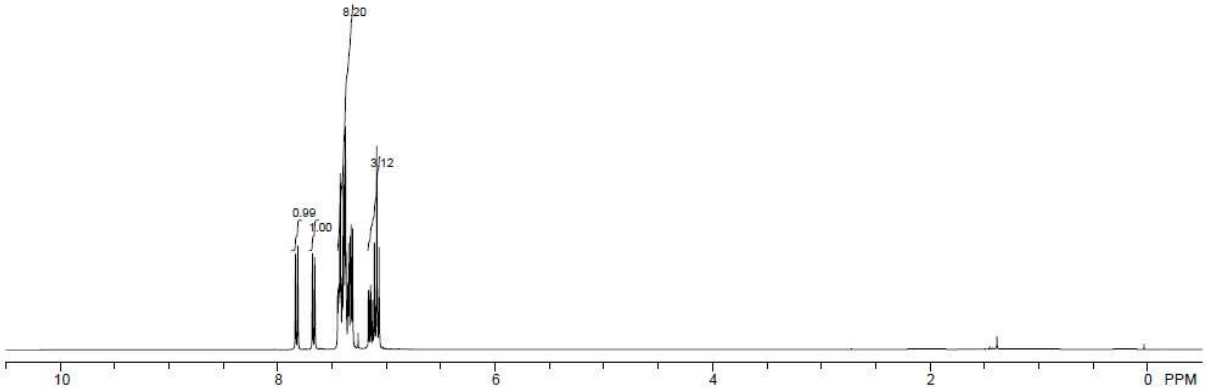
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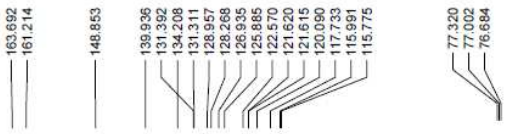
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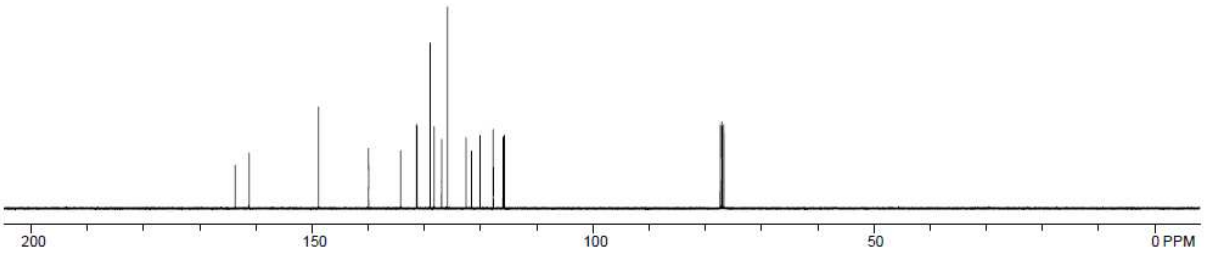
N
F

4f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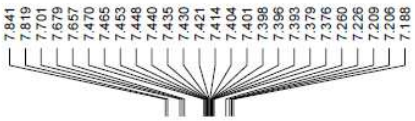
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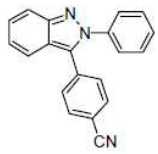
7.188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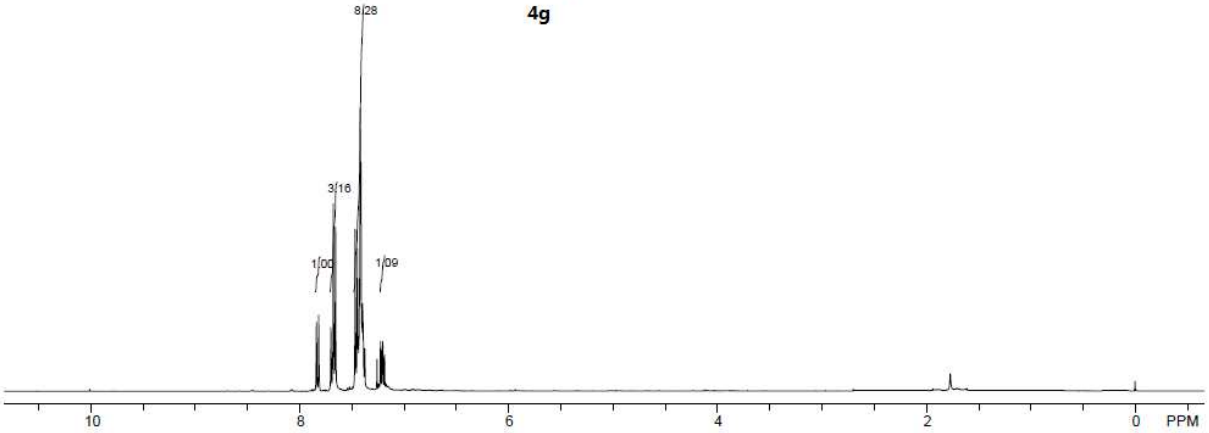
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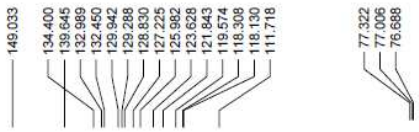
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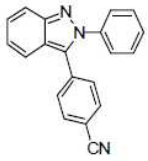
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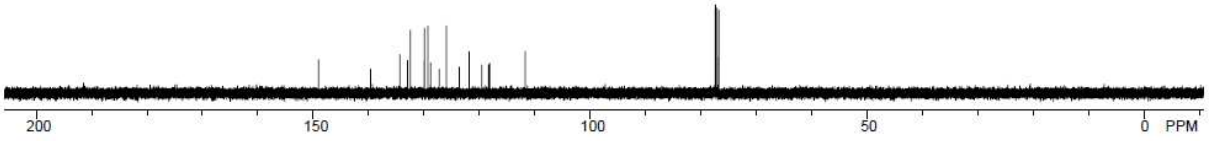
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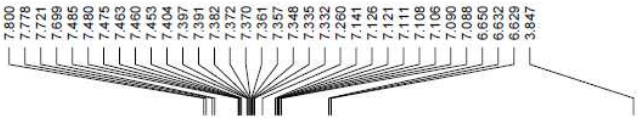
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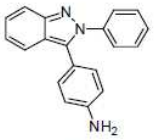
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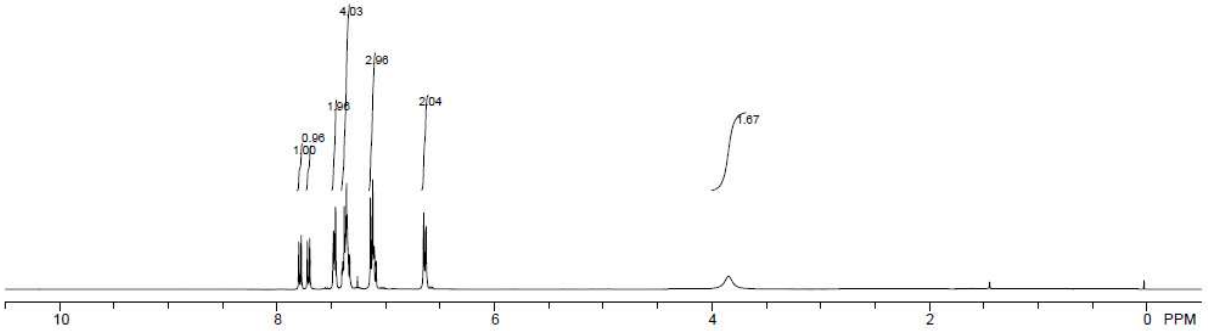
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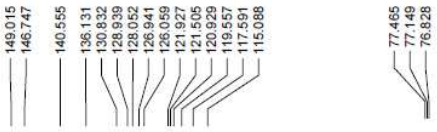
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117.591
115.088

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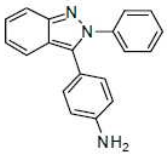
77.149
76.828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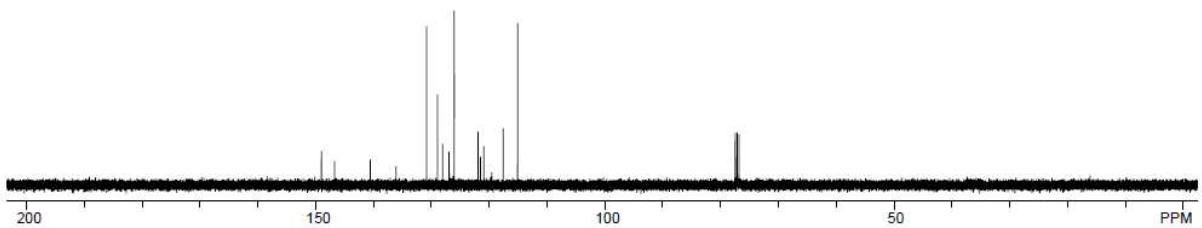
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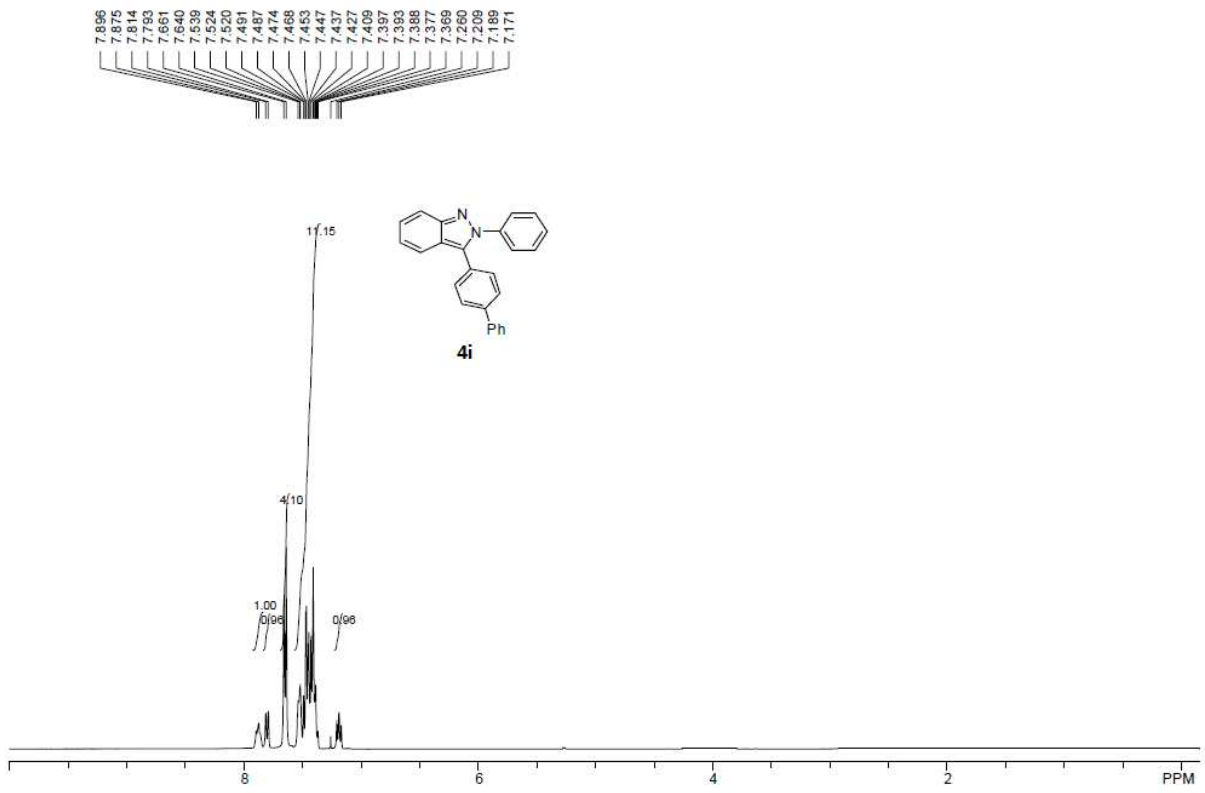
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Ph
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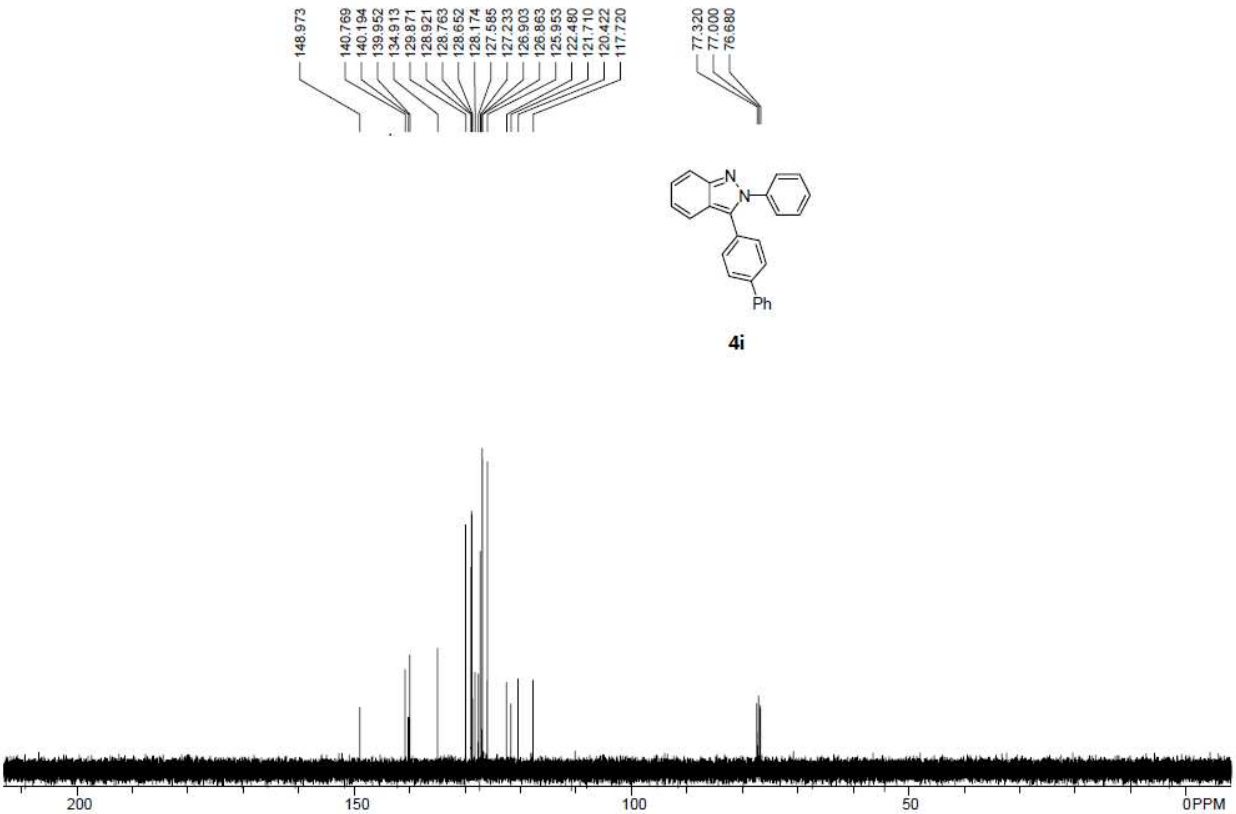
4
2
PPM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127.233
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126.863
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77.000
76.680
Ph
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100
50
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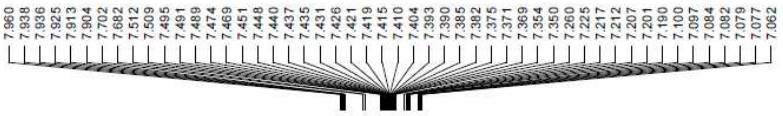
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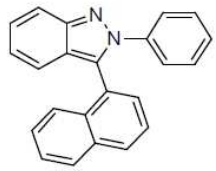

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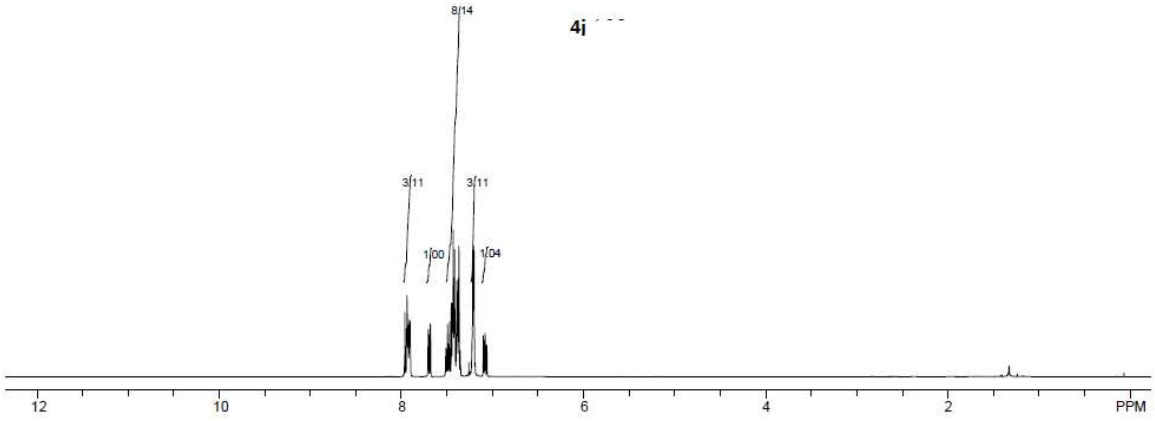
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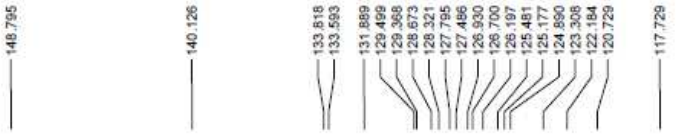
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128.673
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125.481
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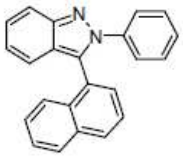
117.729



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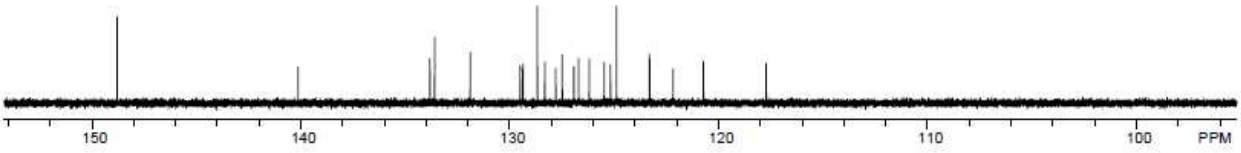
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110
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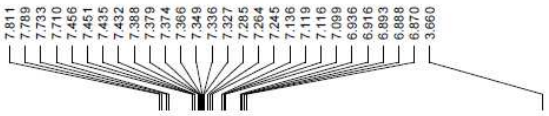
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7.388
7.379
7.374
7.366
7.349
7.336
7.327
7.285
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7.245
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7.119
7.116
7.099
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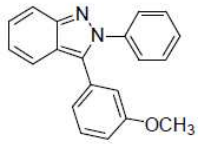
6.888
6.870
3.660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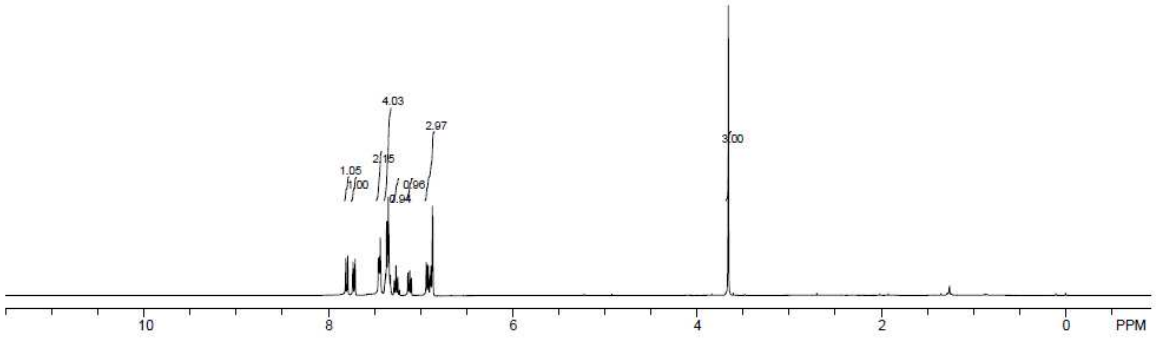
4k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1.00
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0.94
10
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6
4
2
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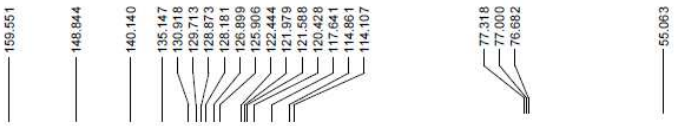
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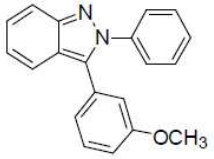
55.063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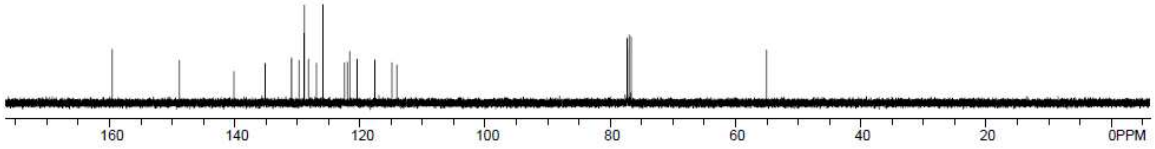
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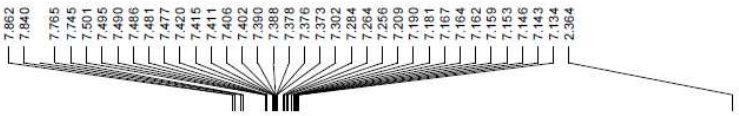
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7.490
7.486
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7.477
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7.411
7.406
7.402
7.390
7.388
7.378
7.376
7.373
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7.284
7.264
7.256
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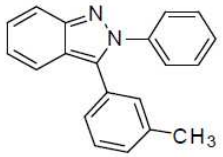
2.364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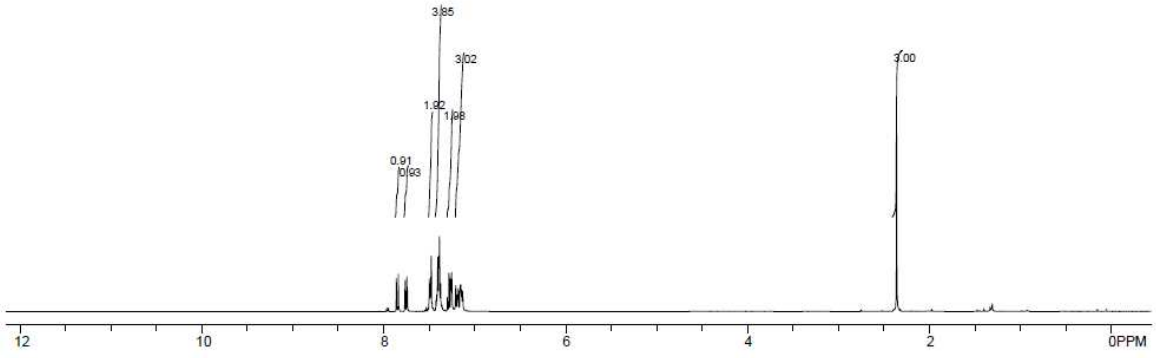
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12
10
8
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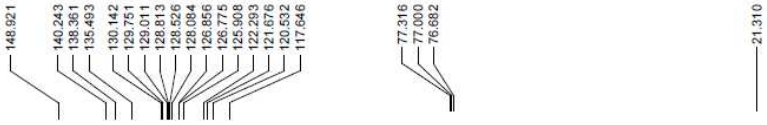
4
2
0PPM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121.676
120.532
117.646
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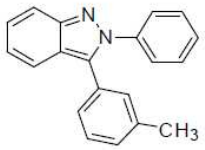
77.000
76.682
21.310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N
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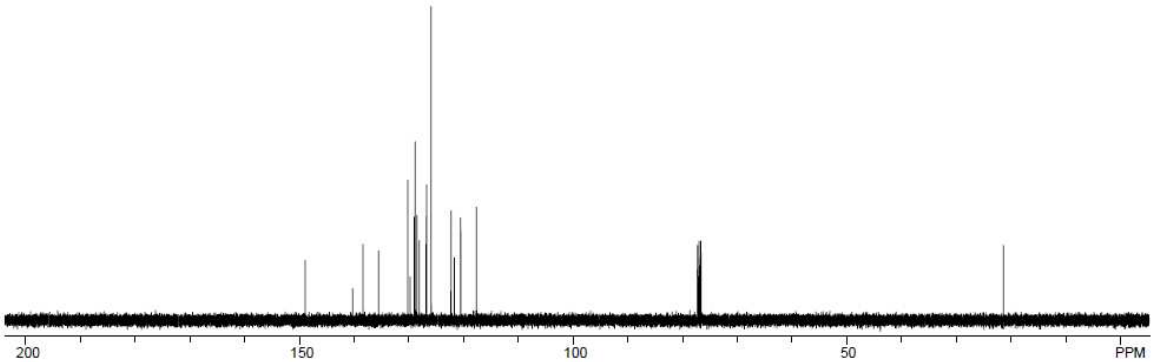
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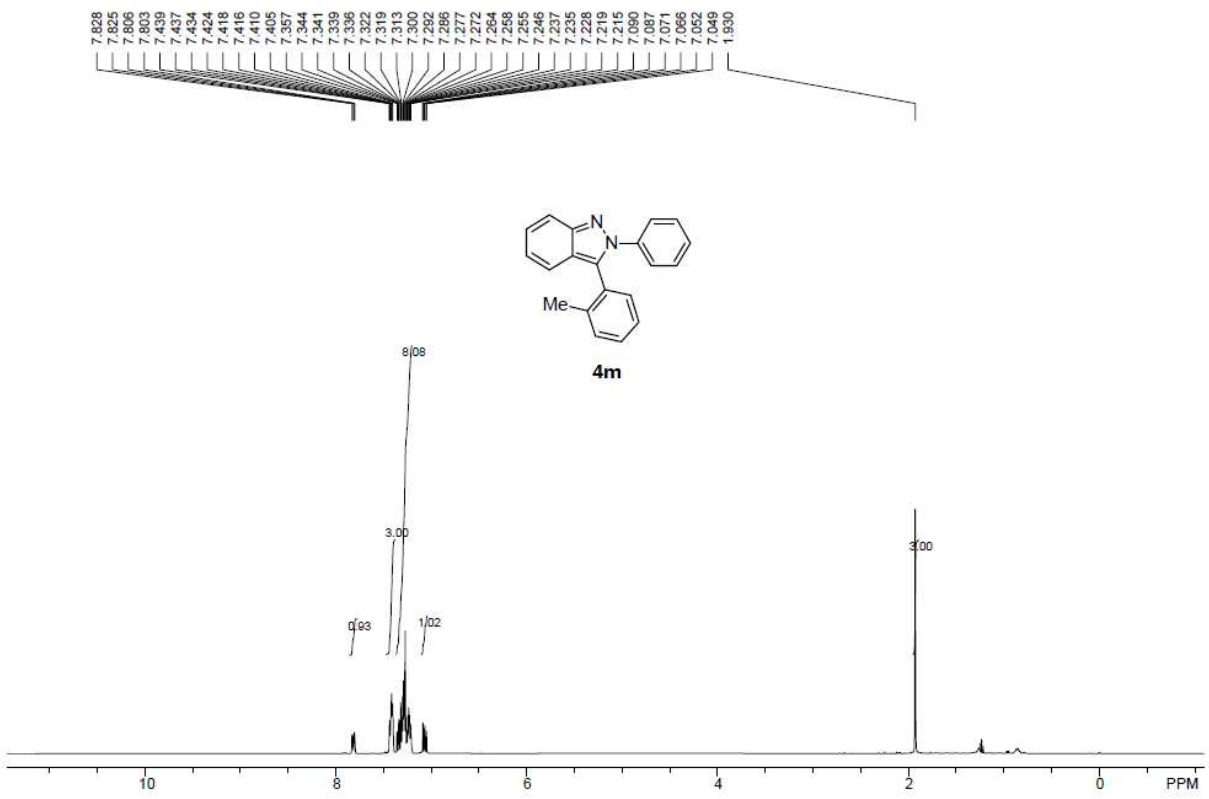
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7.357
7.344
7.341
7.339
7.336
7.322
7.319
7.313
7.300
7.292
7.286
7.277
7.272
7.264
7.258
7.255
7.246
7.237
7.235
7.228
7.219
7.215
7.090
7.087
7.071
7.066
7.052
7.049
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3.00
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2
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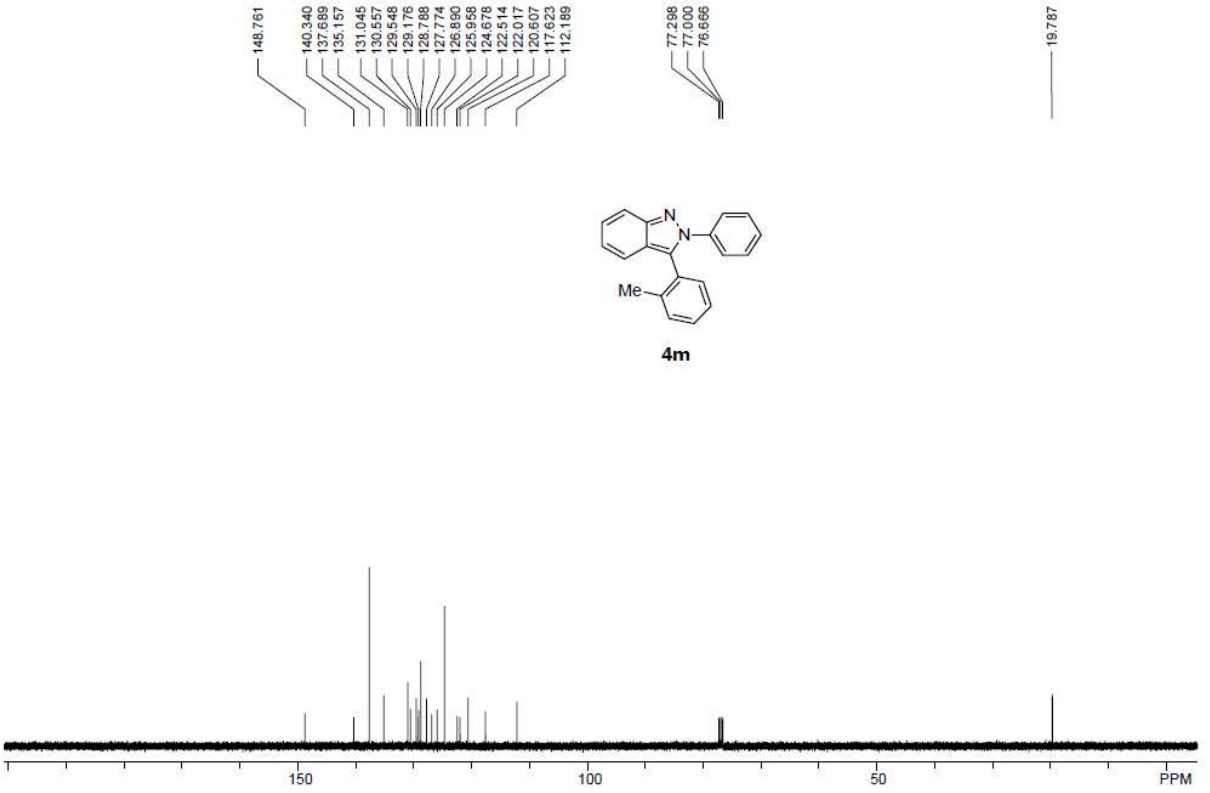
PPM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122.514
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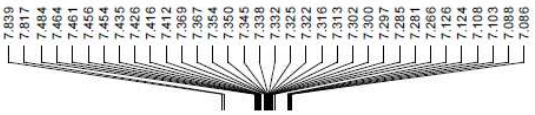
19.787
Me
4m
150
100
50
PPM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7.367
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7.350
7.345
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7.332
7.325
7.322
7.316
7.313
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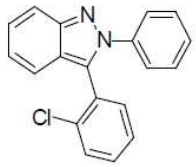
7.088
7.086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Cl

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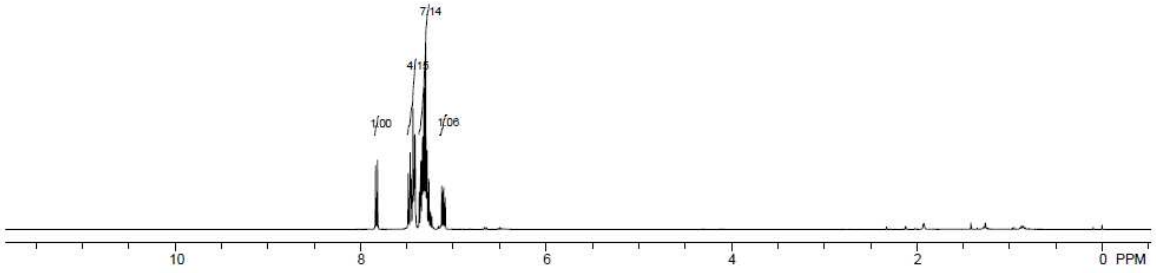
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10
8
6
4
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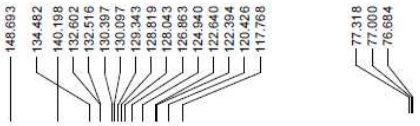
0 PPM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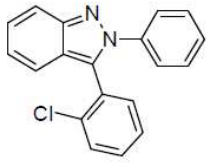
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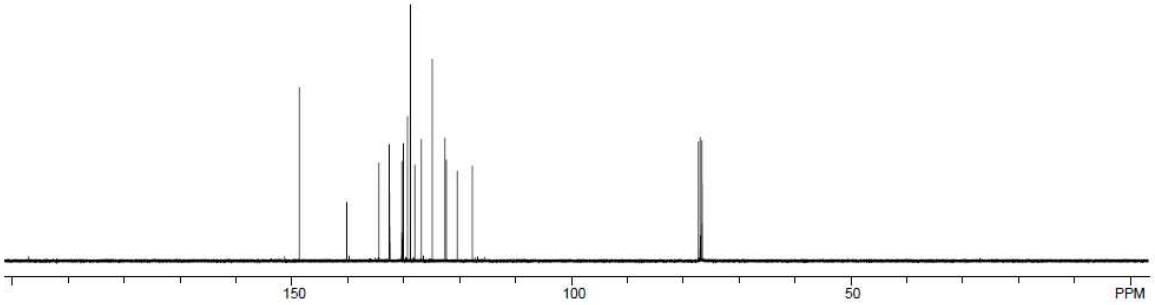
N
Cl

4n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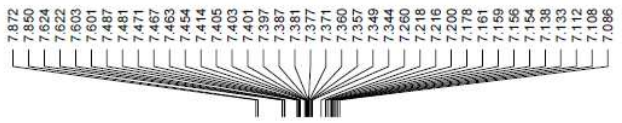
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7.405
7.403
7.401
7.397
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7.377
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7.357
7.349
7.344
7.260
7.218
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7.138
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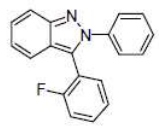
7.108
7.086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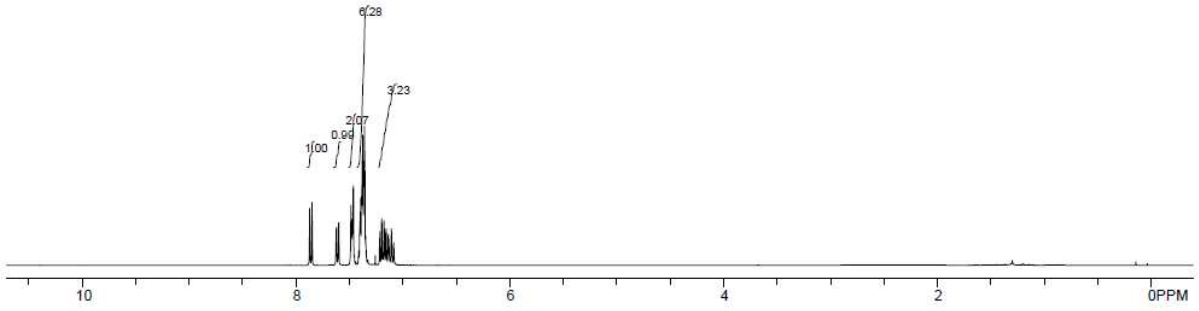
N
F

4o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4
2
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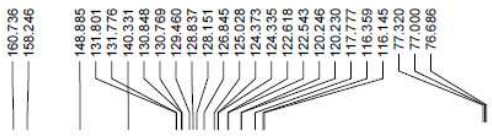
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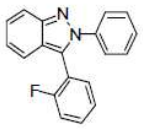
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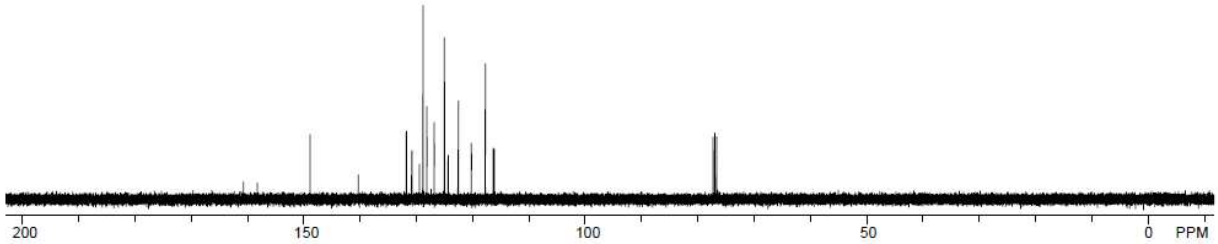
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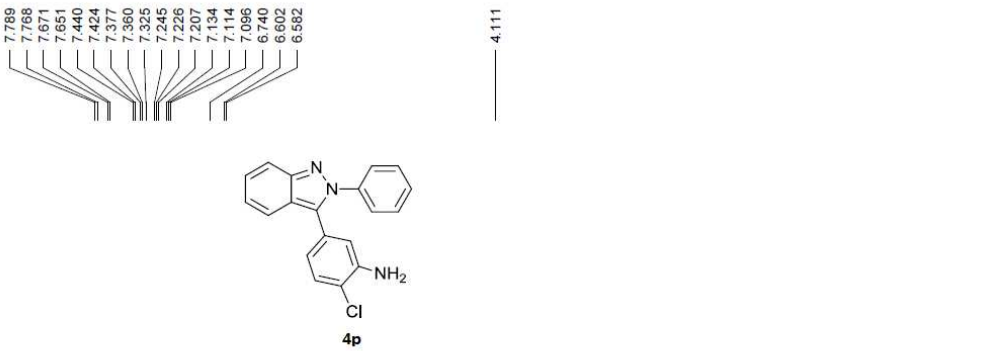
PPM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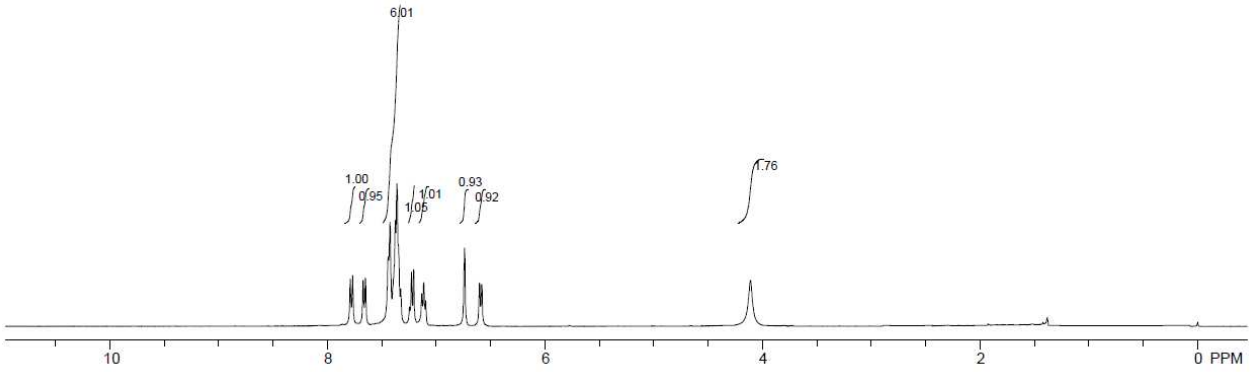
4p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4
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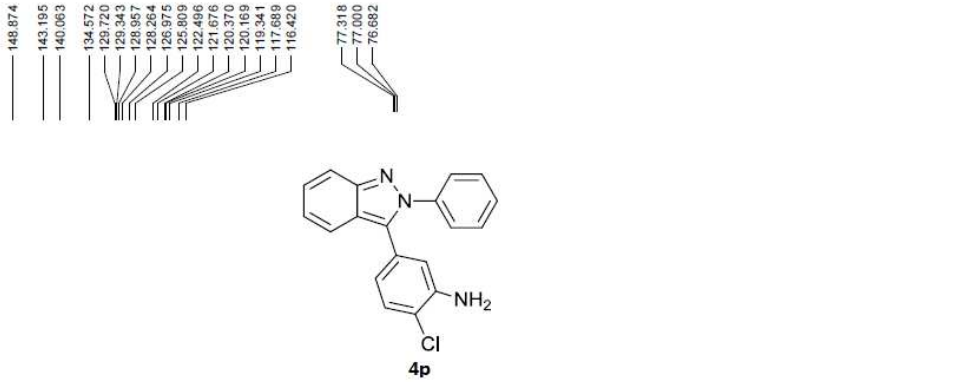
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NH2
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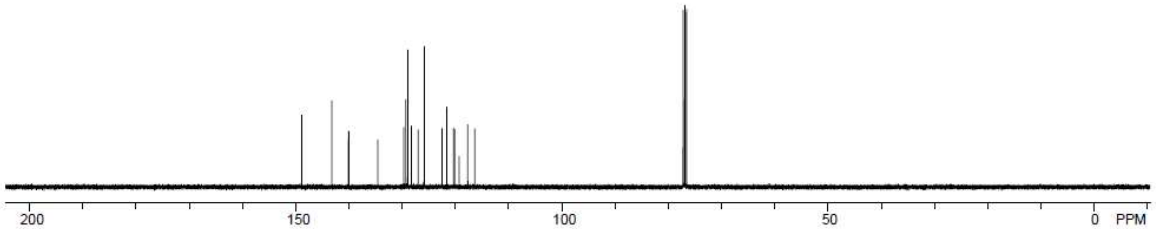
4p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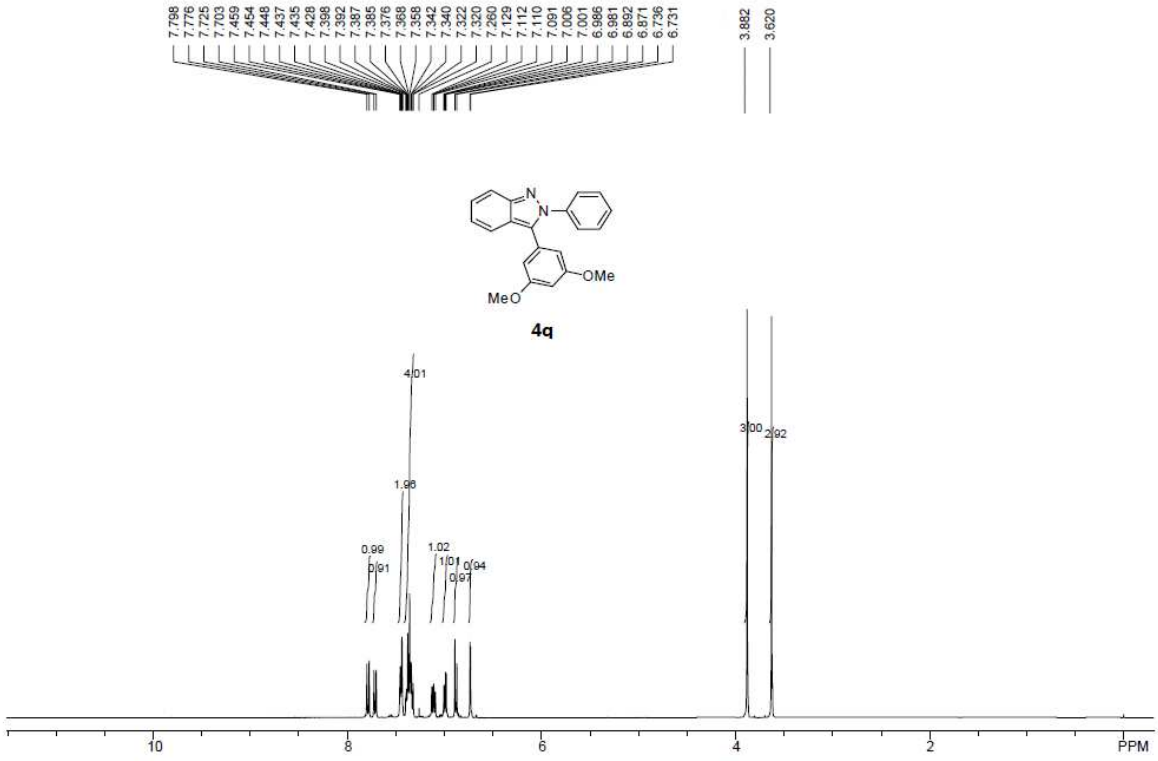
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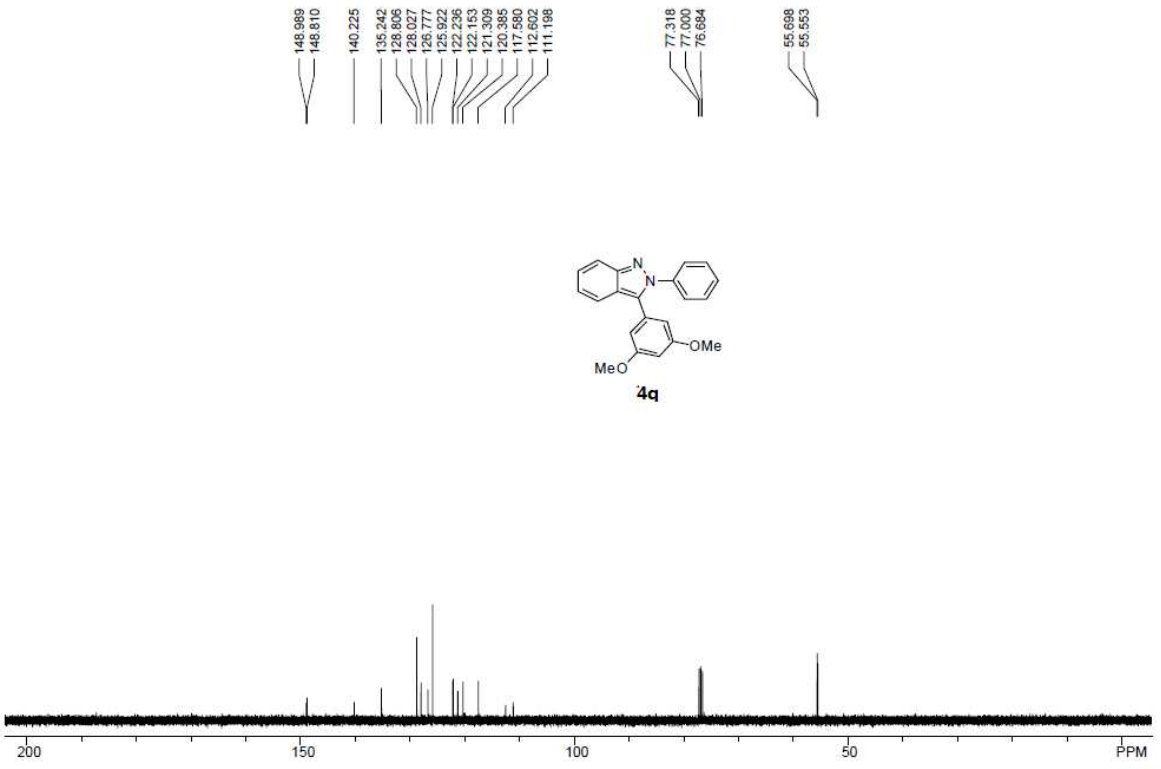
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MeO
4q
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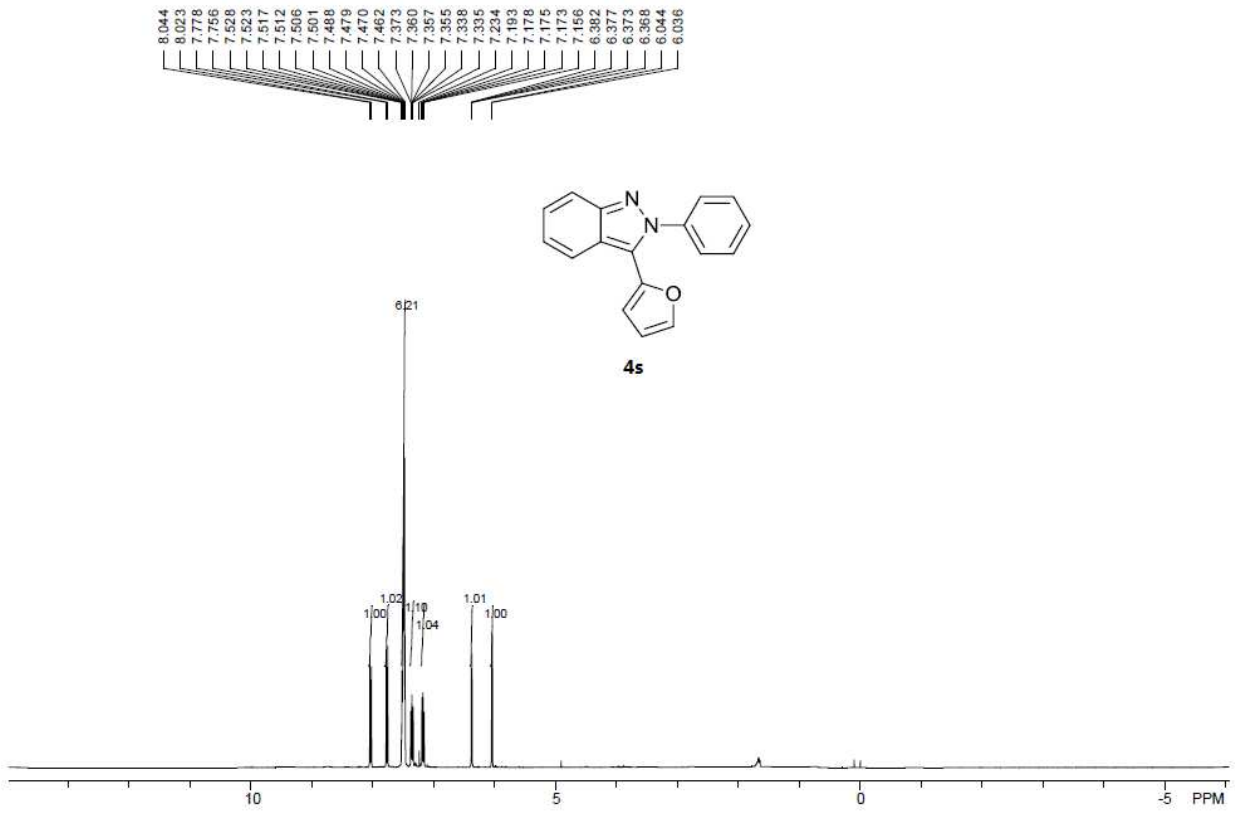
150
100
50
PPM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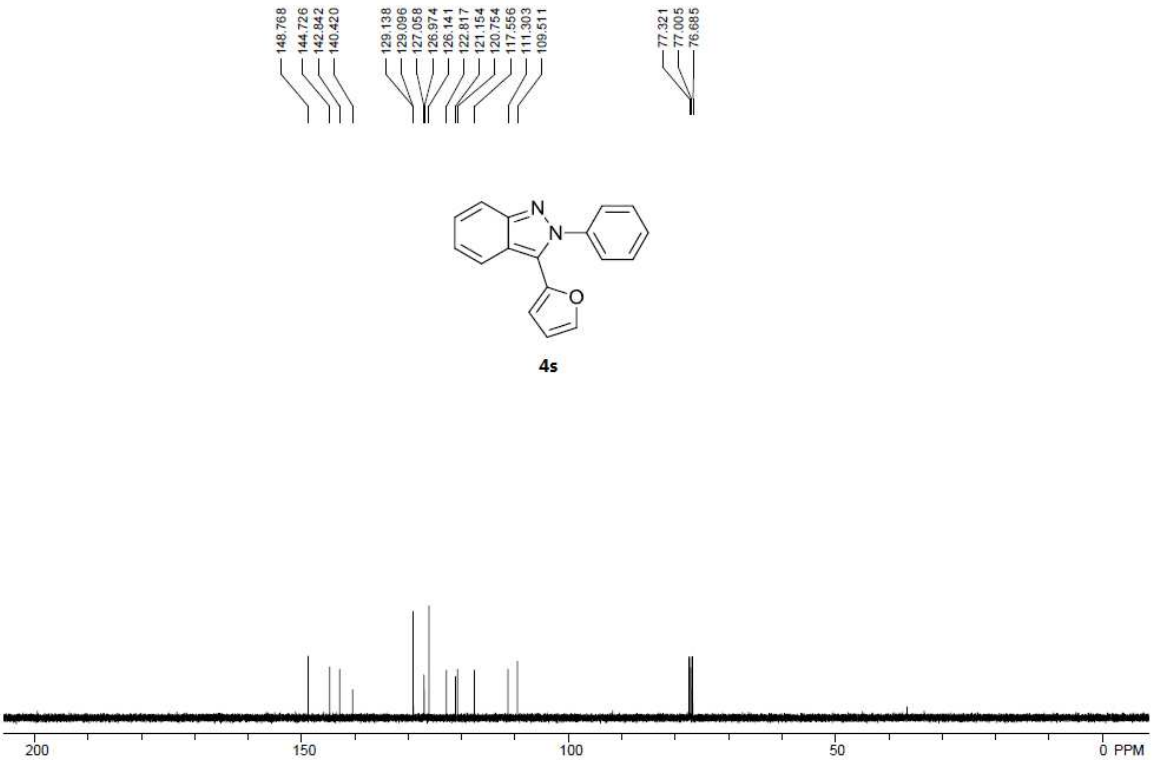
4s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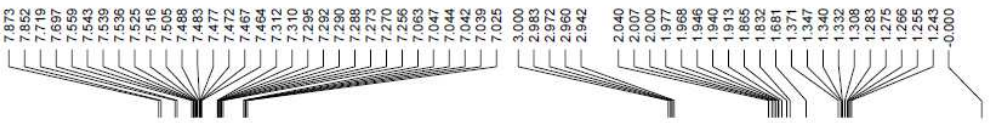
4s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7.039
7.025
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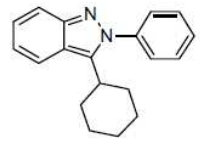

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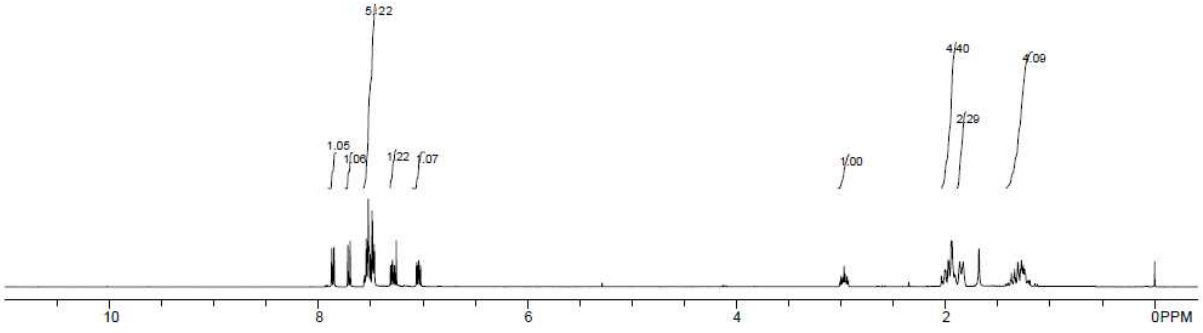
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1.07
1.00
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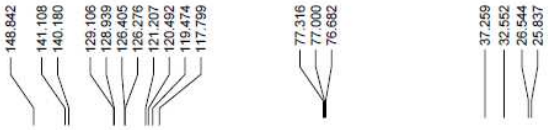
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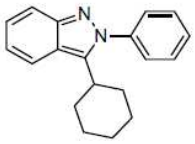
76.682
37.259
32.552
26.544
25.837



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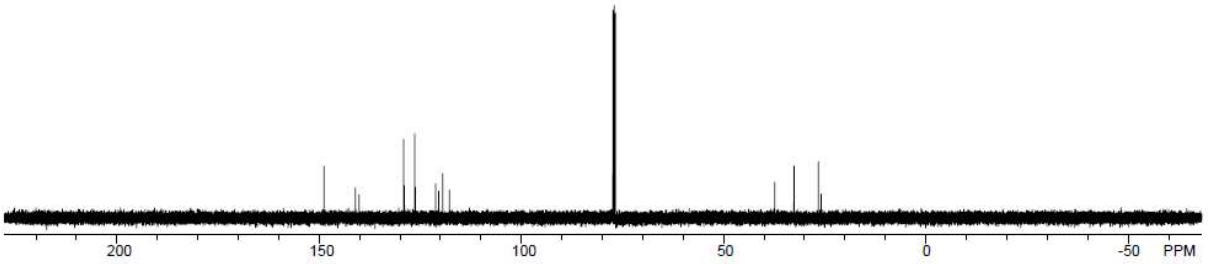
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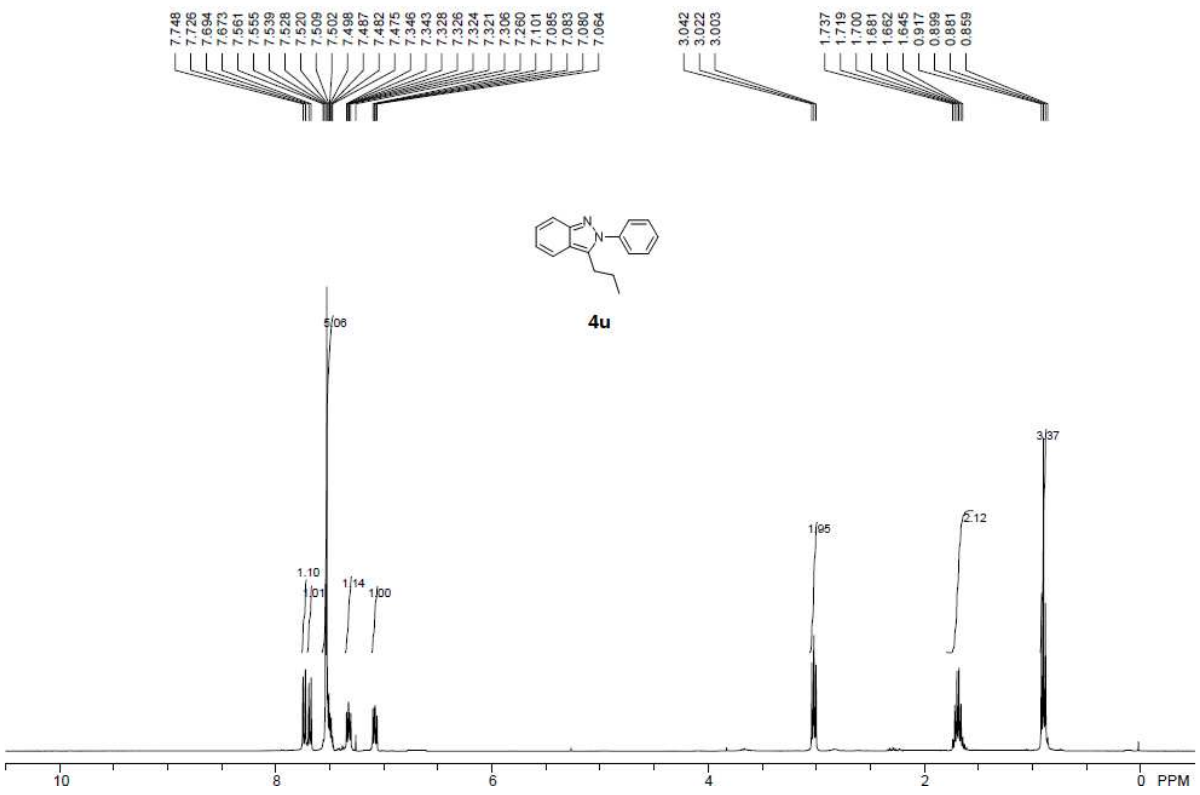
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7.482
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7.326
7.324
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7.101
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7.080
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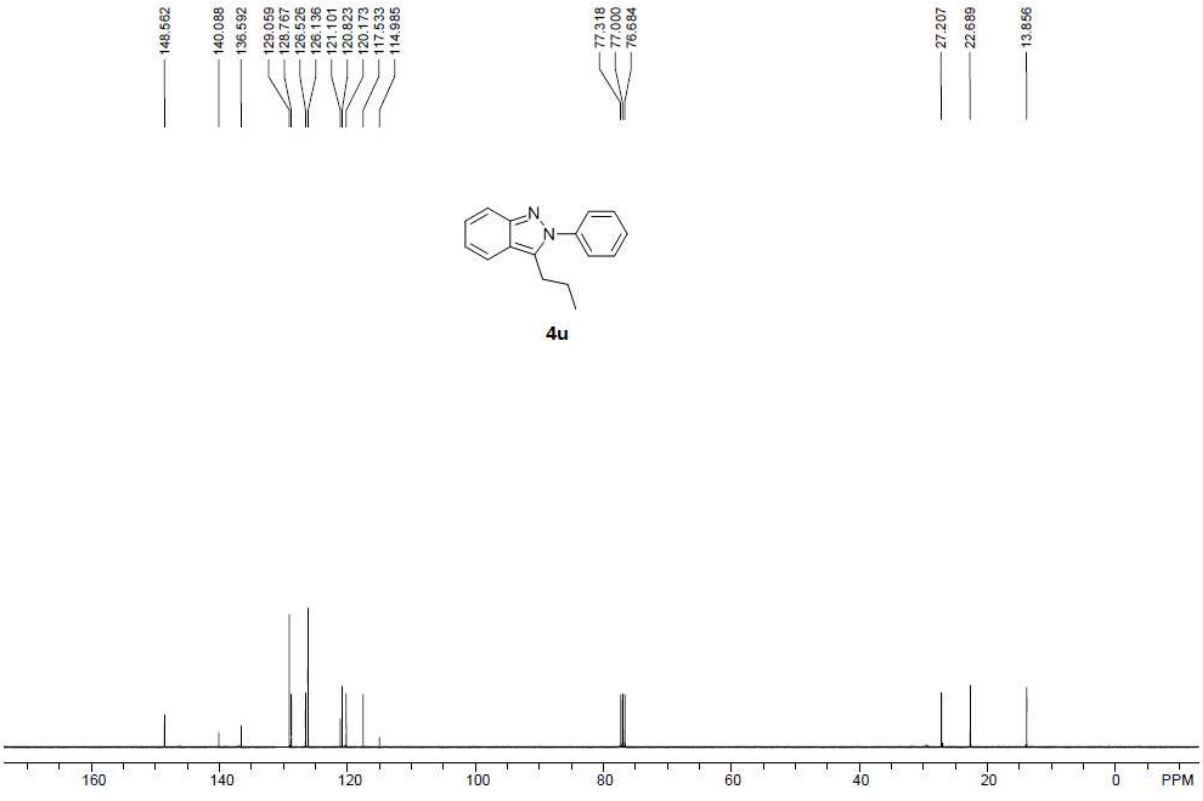
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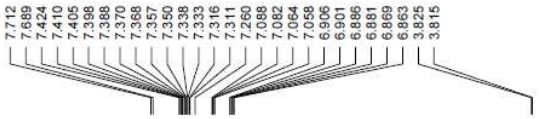
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7.333
7.316
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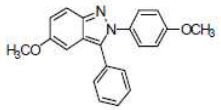
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N
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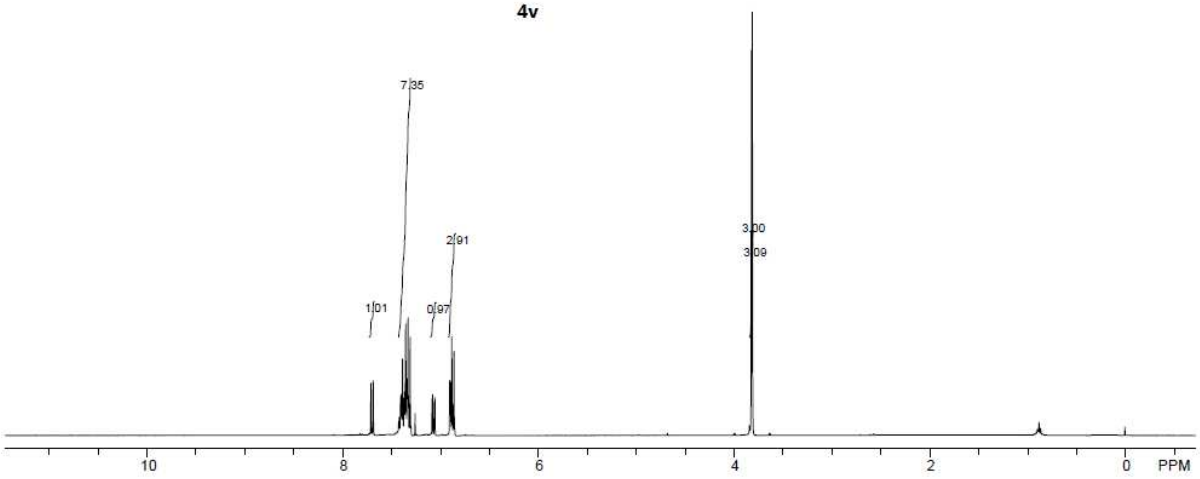
H3CO

4v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6
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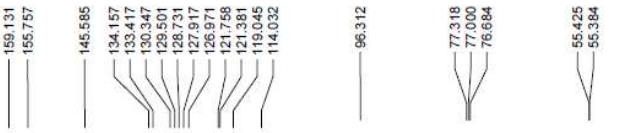
PPM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77.000
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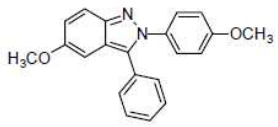
55.384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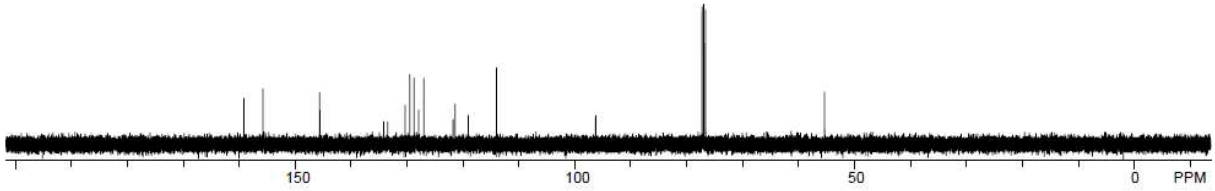
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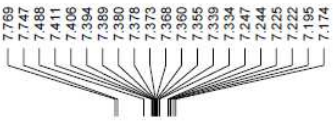
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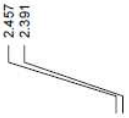
7.222
7.195
7.174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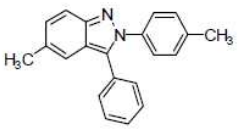
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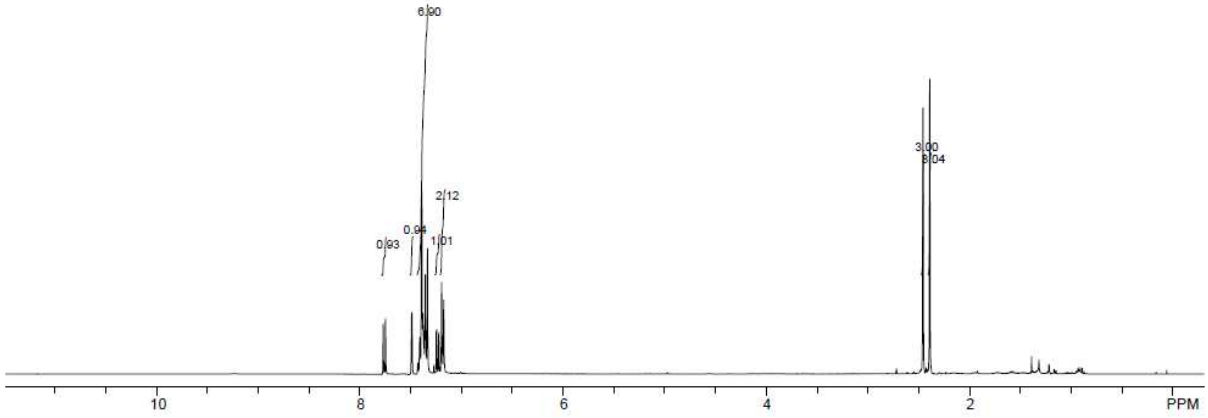
N
CH3
H3C

4w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8
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4
2
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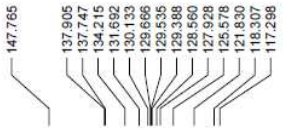
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117.298


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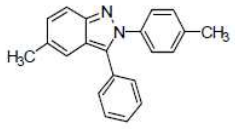
76.680


21.721
20.996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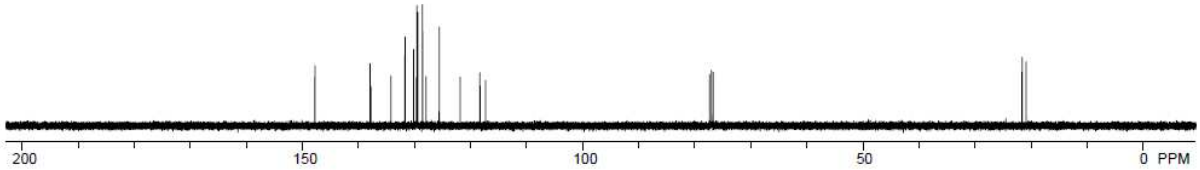
H3C

4w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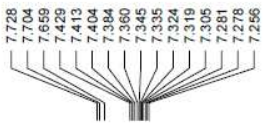
100
50
0 PPM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7.305
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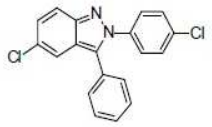
7.256


1.596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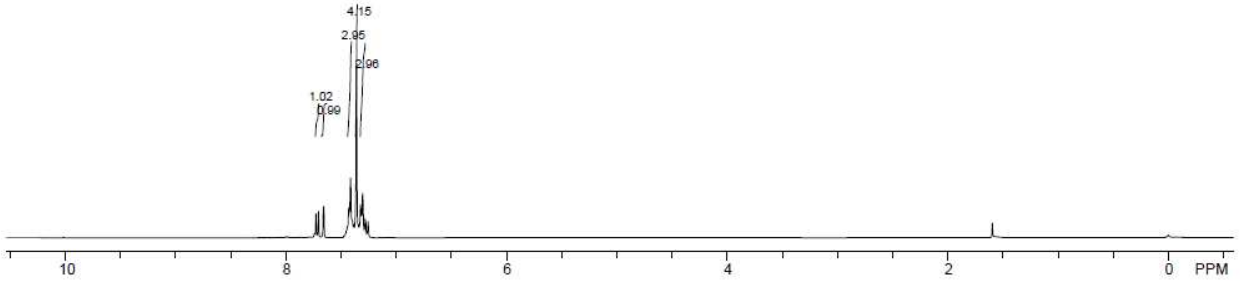
N
Cl
Cl

4x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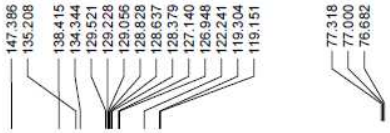
2
0
PPM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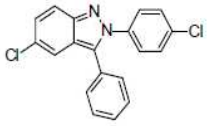
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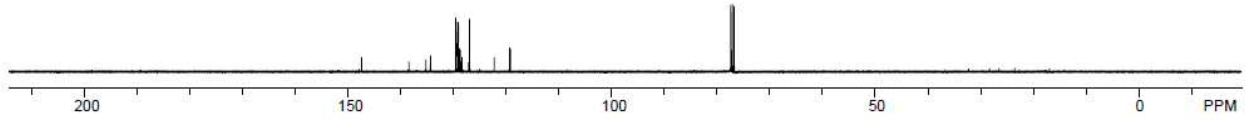
4x


200
150
100
50
0
PPM

7. References

[1] S. Atmaram, A. R. Forrester, M. Gill, R. J. Napier, R. H. Thomson, *Acta Chem. Scand., Ser. B* **1982**, *36b*, 641–645.

[2] S. A. Ohnmacht, A. J. Culshaw, M. F. Greaney, *Org. Lett.* **2010**, *12*, 224–226.

[3] M. D. Angelis, S. Fabio, K. A. Carlson, B. S. Katzenellenbogen, J. A. Katzenellenbogen, *J. Med. Chem.* **2005**, *48*, 1132–1144.