Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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Supporting Information for

Synthesis of Carbazolones and 3-Acetylindoles via Oxidative C-N Bond Formation through PIFA-Mediated Annulation of 2-Aryl Enaminones

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Supplementary Material

General Information

^{1}H and ^{13}C NMR spectra were recorded on a 400 MHz spectrometer at 25 ^{o}C. Chemical shifts values are given in ppm and referred as the internal standard to TMS: 0.00 ppm. The peak patterns are indicated as follows: s, singlet; d, doublet; t, triplet; q, quadruplet; m, multiplet; br, broad and dd, doublet of doublets. The coupling constants J, are reported in Hertz (Hz). High-resolution mass spectra (HRMS) were obtained on a Q-TOF micro spectroMeter. Melting points were determined with a national micromelting point apparatus without corrections. TLC plates were visualized by exposure to ultraviolet light. 1,2-Dichloroethane were dried by CaH_2 before use, other reagents and solvents were purchased as reagent grade and were used without further purification. Flash column chromatography was performed over silica gel 200-300 m and the eluent was a mixture of ethyl acetate (EA) and petroleum ether (PE), or a mixture of methanol (M) and dichloromethane (D).

General Procedure for the Preparation of 1[1-2]

To a solution of 3-aryl-dione[1] (3.0 mmol) in toluene (30 mL) was added corresponding amine (6.0 mmol). The reaction mixture was stirred at reflux until TLC indicated the total consumption of 3-aryl-pentane-2,4-dione. The mixture was evaporated to partially remove the solvent. EA was used to extract the mixture and the combined organic phase, after dried with anhydrous Na_2SO_4, was evaporated to remove the solvent under vacuum and the residue was purified by flash chromatography to give the desired product **1**.[2]

General Procedure for the Preparation of 2

To a solution of correspond substrate **1** (2.0 mmol) in CH_2Cl_2 (30 mL) was added a solution of PIFA (2.4 mmol) in CH_2Cl_2 (10 mL) dropwise under ice-bath during about 30 min, TLC monitored the process, after the consumption of starting material, to the reaction mixture was added saturated aq. $NaHCO_3$ solution, raised the temperature to rt, the mixture was stirred for 10 min, CH_2Cl_2 was used to extract the mixture and the organic phase, after dried with anhydrous Na_2SO_4, was evaporated to remove the solvent, the residue was purified by flash chromatography to give the desired product **2**.

General Procedure for the Preparation of 3[1-2]

A mixture of 3-phenylpentane2,4-dione[1] (3.0 mmol), corresponding amine (6.0 mmol) in EtOH (30 mL) was stirred at reflux until TLC indicated the total consumption of 3-phenylpentane-2,4-dione. The mixture was evaporated to partially remove the solvent. EA was used to extract the mixture and the combined organic phase, after dried with anhydrous Na_2SO_4, was evaporated to remove the solvent under vacuum. The residue was purified by flash chromatography to give the desired product **3**.[2]

General Procedure for the Preparation of 4

To a solution of corresponding substrate **3** (2.0 mmol) in CH_2Cl_2 (30 mL) was added a solution of PIFA (2.4 mmol) in CH_2Cl_2 (10 mL) dropwise under ice-bath during about 30 min, TLC monitored the process. After the consumption of starting material, saturated aq. $NaHCO_3$ solution was added to the reaction mixture. The reaction mixture was warmed up to rt, stirred for 10 min. CH_2Cl_2 was used to extract the mixture and the organic phase, after dried with anhydrous Na_2SO_4, was evaporated to remove the solvent. The residue was purified by flash chromatography to give the desired product **4**.

^{1}H and ^{13}C NMR Spectra of Substrates 1

3-Amino-2-phenylcyclohex-2-enone (1a)[3] Following the general procedure, **1a** was purified by silica gel column chromatography (EA/PE = 20/80). R_f = 0.45 (M/D = 80/20). Yield: 88%, yellow solid, mp 165-167 ^{o}C. ^{1}H NMR (400MHz, $CDCl_3$) δ 7.32 (t, *J* = 7.6 Hz, 2H, H_{arom}), 7.19 (t, *J* = 7.6 Hz, 1H, H_{arom}), 7.06 (d, *J* = 7.2 Hz, 2H, H_{arom}), 6.64 (br, 1H, NH), 5.67 (br, 1H, NH), 2.47 (t, *J* = 6.4 Hz, 2H, CH_2), 2.22 (t, *J* = 6.4 Hz, 2H, CH_2), 1.88 (dd, *J* = 12.4, 6.4 Hz, 2H, CH_2). HRMS (ESI) *m/z* calcd for $C_{12}H_{13}NNaO^+$ [M + Na^+] 210.1025, found 210.1027.

3-Amino-2-*p*-tolylcyclohex-2-enone (1b) Following the general procedure, **1b** was purified by silica gel column chromatography (EA/PE = 50/50). R_f = 0.32 (EA/PE = 90/10). Yield: 90%, yellow solid, mp 212-214 ^{o}C. ^{1}H NMR (400 MHz, DMSO) δ 7.12 (d, *J* = 7.6 Hz, 2H, H_{arom}), 6.94 (d, *J* = 8.0 Hz, 2H, H_{arom}), 6.59 (br, 1H, NH), 5.61 (br, 1H, NH), 2.46 (t, *J* = 6.0 Hz, 2H, CH_2), 2.29 (s, 3H, CH_3), 2.20 (t, *J* = 6.4 Hz, 2H, CH_2), 1.94-1.79 (m, 2H, CH_2). ^{13}C NMR (100 MHz, DMSO): δ 192.38, 162.31, 135.06, 133.15, 131.43, 129.16, 110.39, 37.25, 29.14, 21.53, 21.27.

HRMS (ESI) *m/z* calcd for $C_{13}H_{15}NNaO^+$ [M + Na^+] 224.1046, found 224.1048.

3-Amino-2-(3,4-dimethoxyphenyl)cyclohex-2-enone (1c) Following the general procedure, **1c** was purified by silica gel column chromatography (EA). R_f = 0.40 (M/D = 5/95). Yield: 43%, yellow solid, mp 203-205 °C. ^{1}H NMR (400 MHz, DMSO) δ 6.90 (d, *J* = 8.0 Hz, 1H, H_{arom}), 6.62-6.53 (m, 2H, H_{arom}), 5.75 (br, 2H, NH_2), 3.74 (s, 3H, CH_3), 3.70 (s, 3H, CH_3), 2.45 (t, *J* = 6.4 Hz, 2H, CH_2), 2.21 (t, *J* = 6.4 Hz, 2H, CH_2), 1.90-1.81 (m, 2H, CH_2). ^{13}C NMR (100 MHz, DMSO): δ 192.14, 162.42, 148.90, 147.45, 128.64, 123.65, 115.33, 112.32, 110.48, 55.94, 55.73, 37.27, 29.13, 21.52. LRMS (ESI) *m/z* calcd for $C_{14}H_{17}NNaO_3^+$ [M + Na^+] 270.1101, found 270.1105.

3-Amino-2-(3-fluorophenyl)cyclohex-2-enone (1d) Following the general procedure, **1d** was purified by silica gel column chromatography (EA/PE = 80/20). R_f = 0.30 (M/D = 10/90). Yield: 57%, white solid, mp 90-91 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (d, *J* = 13.6 Hz, 1H, H_{arom}), 7.31 (t, *J* = 8 Hz, 1H, H_{arom}), 6.93-6.85 (m, 2H, H_{arom}), 6.14 (br, 1H, NH_2), 5.84 (br, 1H, NH_2) 2.45 (t, *J* = 6.0 Hz, 2H, CH_2), 2.38 (t, *J* = 6.0 Hz, 2H, CH_2), 2.04-1.98 (m, 2H, CH_2). ^{13}C NMR (100 MHz, $CDCl_3$): δ 194.51, 163.17 (d, $J_{C\text{-}F}$ = 239.2 Hz), 163.15, 137.05 (d, $J_{C\text{-}F}$ = 7.9 Hz), 130.22 (d, $J_{C\text{-}F}$ = 8.6 Hz), 126.67, 118.10(d, $J_{C\text{-}F}$ = 10.1 Hz), 112.64 (d, $J_{C\text{-}F}$ = 20.7 Hz), 111.4, 36.65, 29.16, 21.17. HRMS (ESI) m/z calcd for $C_{12}H_{12}FNNaO^+$ [M + Na^+] 228.0795, found 228.0795.

3-Amino-2-(3-(trifluoromethyl)phenyl)cyclohex-2-enone (1e) Following the general procedure, **1e** was purified by silica gel column chromatography (EA/PE = 80/20). R_f = 0.40 (EA/PE = 95/5). Yield: 63%, white solid, mp 109-110 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48-7.38 (m, 2H, H_{arom}), 7.37-7.32 (m, 1H, H_{arom}), 7.26-7.22 (m, 1H, H_{arom}), 2.37-2.34 (m, 4H, CH_2), 1.96-1.91 (m, 2H, CH_2). ^{13}C NMR (100 MHz, $CDCl_3$): δ 194.74, 164.52, 135.65, 134.70, 129.18, 128.04, 128.0 124.08 (q, $J_{C\text{-}F}$ = 270.8 Hz), 123.63 (q, $J_{C\text{-}F}$ = 3.6 Hz), 110.35, 36.13, 28.73, 20.97. HRMS (ESI) m/z calcd for $C_{13}H_{12}F_3NNaO^+$ [M + Na^+] 278.0763, found 278.0765.

3-Amino-2-(4-nitrophenyl)cyclohex-2-enone (1f) Following the general procedure, **1f** was purified by silica gel column chromatography (EA/PE = 80/20). R_f = 0.35 (M/D = 5/95). Yield: 37%, yellow solid, mp 127-129 °C. ^{1}H NMR (400 MHz, DMSO) δ 8.16 (d, *J* = 8.8 Hz, 2H, H_{arom}), 7.37 (d, *J* = 8.8 Hz, 2H, H_{arom}), 6.94 (br, 1H, NH), 6.35 (br, 1H, NH), 2.52 (t, *J* = 6.4 Hz, 2H, CH_2),

2.26 (t, J = 6.4 Hz, 2H, CH_2), 1.91-1.85 (m, 2H, CH_2). ^{13}C NMR (100 MHz, DMSO): δ 191.88, 163.71, 145.58, 144.73, 132.85, 123.48, 108.56, 37.09, 29.53, 21.25. HRMS (ESI) m/z calcd for $C_{12}H_{12}N_2NaO_3^+$ [M + Na^+] 255.0740, found 255.0741.

3-Amino-5,5-dimethyl-2-phenylcyclohex-2-enone (1g) Following the general procedure, **1g** was purified by silica gel column chromatography (EA/PE = 80/20). R_f = 0.40 (EA/PE = 95/5). Yield: 73%, white solid, mp 112-114 °C. ^1H NMR (400 MHz, $CDCl_3$) δ 7.34 (t, J = 8.8 Hz, 2H, H_{arom}), 7.22-7.19 (m, 2H, H_{arom}), 7.14-7.12 (m, 1H, H_{arom}), 4.72 (br, 2H, NH_2), 2.30 (s, 4H, CH_2), 1.13 (s, 6H, CH_2). ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.18, 151.21, 136.31, 124.69, 123.19, 122.50, 121.18, 111.92, 111.33, 77.38, 77.06, 76.74, 52.31, 37.34, 35.81, 28.64. HRMS (ESI) m/z calcd for $C_{14}H_{17}NNaO^+$ [M + Na+] 238.1202, found 238.1205.

3-Amino-2-(naphthalen-2-yl)cyclohex-2-enone (1h) Following the general procedure, **1h** was purified by silica gel column chromatography (EA/PE = 95/5). R_f = 0.40 (EA/PE = 95/5). Yield: 69%, white solid, mp 192-193 °C. ^1H NMR (400 MHz, $CDCl_3$) δ 7.80 (d, J = 8.0 Hz, 1H, H_{arom}), 7.78-7.73 (m, 2H, H_{arom}), 7.59 (s, 1H, H_{arom}), 7.43-7.41 (m, 2H, H_{arom}), 7.23 (t, J = 12 Hz, 1H, H_{arom}), 4.72 (br, 2H, NH_2), 2.43 (t, J = 6.0 Hz, 4H, CH_2), 2.02-1.96 (m, 2H, CH_2). ^{13}C NMR (100 MHz, $CDCl_3$): δ 194.74, 161.38, 133.76, 132.49, 132.19, 129.49, 129.19, 128.38, 127.71, 127.60, 125.81, 125.77, 36.74, 29.26, 21.26. HRMS (ESI) m/z calcd for $C_{16}H_{15}NNaO^+$ [M + Na^+] 260.1046, found 260.1047.

3-(Methylamino)-2-phenylcyclohex-2-enone (1i) Following the general procedure, **1i** was purified by silica gel column chromatography (EA/PE = 90/10). R_f= 0.35 (EA/PE = 95/5). Yield: 81%, white solid, mp 92-94 °C. 1H NMR (400 MHz, $CDCl_3$) δ 7.38 (t, J = 7.6 Hz, 2H, H_{arom}), 7.26 (dd, J = 10.4, 4.4 Hz, 1H, H_{arom}), 7.14 (d, J = 7.2 Hz, 2H, H_{arom}), 4.78 (br, 1H, NH), 2.81 (s, 3H, CH_3), 2.56 (t, J = 6.4 Hz, 2H, CH_2), 2.47-2.40 (m, 2H, CH_2), 2.10-2.04 (m, 2H, CH_2). ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.18, 162.59, 134.95, 131.36, 129.02, 126.96, 112.58, 36.55, 29.98, 25.20, 21.15. HRMS (ESI) m/z calcd for $C_{13}H_{15}NNaO^+$ [M + Na^+] 224.1046, found 224.1048.

3-(Methylamino)-2-*p*-tolylcyclohex-2-enone (1j) Following the general procedure, **1j** was purified by silica gel column chromatography (EA/PE = 80/20). R_f = 0.54 (M/D = 5/95). Yield: 93%, white solid, mp 129-130 °C. ^1H NMR (400 MHz, $CDCl_3$) δ 7.20 (d, J = 7.6 Hz, 2H, H_{arom}),

7.03 (d, J = 7.6 Hz, 2H, H_{arom}), 4.76 (br, 1H, NH), 2.82 (d, J = 5.2 Hz, 3H, CH_3), 2.56 (t, J = 6.4 Hz, 2H, CH_2), 2.46-2.41 (m, 2H, CH_2), 2.34 (s, 3H, CH_3), 2.16-2.02 (m, 2H, CH_2). ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.33, 162.44, 136.57, 131.74, 131.15, 129.85, 112.52, 36.60, 29.96, 25.18, 21.28, 21.22. HRMS (ESI) *m/z* calcd for $C_{14}H_{17}NNaO^+$ [M + Na^+] 238.1202, found 238.1207.

6-(Propylamino)-4,5-dihydro-[1,1'-biphenyl]-2(3*H*)-one (1k) Following the general procedure, 1b was recrystallized from EA/PE as a white solid. R_f = 0.56 (M/D = 5/95). Yield: 62%, m.p. 118 ^{o}C. ^{1}H NMR (400MHz, $CDCl_3$) δ 7.39 (t, J = 7.5 Hz, 2H), 7.25 (t, J = 7.4 Hz, 1H), 7.15 (d, J = 7.3 Hz, 2H), 4.76 (br, 1H), 3.07 (dd, J = 12.9, 6.4 Hz, 2H), 2.54 (dd, J = 21.0, 14.9 Hz, 2H), 2.43 (t, J = 6.4 Hz, 2H), 2.12 -1.99 (m, 2H), 1.53-1.37 (m, 2H), 0.84 (t, J = 7.4 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.12, 161.91, 135.06, 131.30, 129.00, 126.90, 112.45, 44.74, 36.67, 25.38, 23.64, 21.30, 11.11. HRMS (ESI) m/z calcd for $C_{15}H_{19}NNaO^+$ [M + Na^+] 252.1359, found 252.1360.

2-Phenyl-3-(phenylamino)cyclohex-2-enone (1l)[4] Following the general procedure, **1l** was purified by silica gel column chromatography (EA/PE = 5/95). R_f = 0.51 (EA/PE = 60/40). Yield: 87%, white solid, mp 127-129 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 (t, J = 7.6 Hz, 2H, H_{arom}), 7.33-7.26 (m, 4H, H_{arom}), 7.18 (t, J = 7.6 Hz, 1H, H_{arom}), 7.01 (d, J = 8.0 Hz, 2H, H_{arom}), 6.46 (s, 1H, H_{arom}), 2.60 (t, J = 6.4 Hz, 2H, CH_2), 2.52 (t, J = 6.4 Hz, 2H, CH_2), 2.07-2.01 (m, 2H, CH_2). LRMS (ESI) m/z calcd for $C_{36}H_{34}N_2NaO_2^+$ [2M + Na^+] 549.2, found 549.2.

3-(4-Methoxyphenylamino)-2-phenylcyclohex-2-enone (1m) Following the general procedure, **1m** was purified by silica gel column chromatography. R_f = 0.40 (EA/PE = 10/90). Yield: 63%, white solid, mp 67-69 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 (t, J = 7.2 Hz, 7.6 Hz, 2H, H_{arom}), 7.29-7.36 (m, 3H, H_{arom}), 7.01 (d, J = 8.0 Hz, 2H, H_{arom}), 6.87 (d, J = 8.0 Hz, 2H, H_{arom}), 6.46 (br, 1H, NH), 3.82 (s, 3H, CH_3),2.66 (t, J = 6.4 Hz, 2H, CH_2), 2.54 (t, J = 6.0 Hz, 2H, CH_2), 2.06-2.02 (m, 2H, CH_2). ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.38, 160.25, 158.01, 134.56, 131.25, 131.00, 129.16, 127.47, 127.27, 114.38, 114.32, 37.07, 26.75, 21.77. HRMS (ESI) m/z calcd for $C_{19}H_{19}NNaO_2^+$ [M + Na^+] 316.1308, found 316.1308.

3-(4-Nitrophenylamino)-2-phenylcyclohex-2-enone (1n) Following the general procedure, **1n** was purified by silica gel column chromatography. R_f = 0.50 (EA/PE = 10/90). Yield: 87%, yellow

solid, mp 132-134 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16 (d, *J* = 8.0 Hz, 2H, H_{arom}), 7.45 (t, *J* = 8.0 Hz, 2H, H_{arom}), 7.35 (t, *J* = 8.0 Hz, 1H, H_{arom}), 7.21 (d, *J* = 8.0 Hz, 2H, H_{arom}), 6.99 (d, *J* = 8.0 Hz, 2H, H_{arom}), 6.64 (br, 1H, NH), 2.80 (t, *J* = 6.0 Hz, 2H, CH_2), 2.59 (t, *J* = 6.0 Hz 2H, CH_2), 2.15 (d, *J* = 4.5 Hz, 2H, CH_2). ^{13}C NMR (100 MHz, DMSO) δ 195.54, 156.08, 147.13, 135.38, 131.36, 131.20, 128.04, 127.58, 126.67, 124.60, 120.45, 37.90, 29.90, 21.40. HRMS (ESI) m/z calcd for $C_{18}H_{16}N_2NaO_3^+$ [M + Na^+] 331.1053, found 331.1057.

^{1}H and ^{13}C NMR spectra of Carbazolones 2

2,3-Dihydro-1*H*-carbazol-4(9*H*)-one (2a)[5] Following the general procedure, **2a** was purified by silica gel column chromatography (EA/PE = 60/40). R_f = 0.50 (EA/PE = 80/20). Yield: 90%, white solid, mp 224-226 °C. ^{1}H NMR (400 MHz, DMSO) δ 7.94 (d, *J* = 6.8 Hz, 1H, H_{arom}), 7.39 (d, *J* = 6.8 Hz, 1H, H_{arom}), 7.26-6.94 (m, 2H, H_{arom}), 2.96 (t, *J* = 6.0 Hz, 2H, CH_2), 2.48-2.36 (m, 2H, CH_2), 2.17-2.06 (m, 2H, CH_2). LRMS (ESI) m/z calcd for $C_{12}H_{11}NNaO^+$ [M + Na^+] 208.1, found 208.1.

7-Methyl-2,3-dihydro-1*H*-carbazol-4(9*H*)-one (2b) Following the general procedure, **2b** was purified by silica gel column chromatography (EA/PE = 60/40). R_f = 0.29 (EA/PE = 60/40). Yield: 90%, white solid, mp>250 °C. ^{1}H NMR (400 MHz, DMSO) δ 7.81 (d, *J* = 8.0 Hz, 1H, H_{arom}), 7.18 (s, 1H, H_{arom}), 6.96 (d, *J* = 7.6 Hz, 1H, H_{arom}), 2.93 (t, *J* = 6.0 Hz, 2H, CH_2), 2.45-2.33 (m, 5H, CH_2, CH_3), 2.11 (dd, *J* = 12.4, 6.2 Hz, 2H, CH_2). ^{13}C NMR (100 MHz, DMSO): δ 193.17, 152.30, 136.74, 132.04, 123.45, 122.76, 120.33, 112.17, 111.90, 38.23, 23.92, 23.17, 21.78. HRMS (ESI) m/z calcd for C13H13NNaO+ [M + Na+] 222.0889, found 222.0892.

6,7-Dimethoxy-2,3-dihydro-1*H*-carbazol-4(9*H*)-one (2c) Following the general procedure, **2c** was purified by silica gel column chromatography (EA/PE = 10/90). R_f = 0.40 (EA/PE = 20/80). Yield: 67%, white solid, mp 230-231 °C. ^{1}H NMR (400MHz, DMSO) δ 11.56 (br, 1H, NH), 7.44 (s, 1H, H_{arom}), 6.94 (s, 1H, H_{arom}), 3.78 (s, 3H, CH_3), 3.76 (s, 3H, CH_3), 2.91 (t, *J* = 6.0 Hz, 2H, CH_2), 2.39 (t, *J* = 6.0 Hz, 2H, CH_2), 2.10 (dd, *J* = 12.0, 5.9 Hz, 2H, CH_2). ^{13}C NMR (100 MHz, DMSO) δ 193.20, 150.87, 147.07, 146.34, 130.39, 117.68, 112.33, 103.17, 96.08, 56.24, 56.20, 38.21, 24.06, 23.23. HRMS (ESI) m/z calcd for $C_{14}H_{16}NO_3^+$ [M + H^+] 246.1125, found 246.1127.

6-Fluoro-2,3-dihydro-1*H*-carbazol-4(9*H*)-one (2d)[6] Following the general procedure, **2d** was

purified by silica gel column chromatography (EA/PE = 10/90). R_f = 0.55 (EA/PE = 20/80). Yield: 65%, white solid, mp>250 ^{o}C. ^{1}H NMR (400 MHz, DMSO) δ 12.52 (br, 1H, NH), 8.24 (s, 1H, H_{arom}), 7.61 (d, *J* = 8.4 Hz, 1H, H_{arom}), 7.49 (d, *J* = 8.4 Hz, 1H, H_{arom}), 3.02 (t, *J* = 6.0 Hz, 2H, CH_2), 2.48 (t, *J* = 6.4 Hz, 2H, CH_2), 2.17-2.14 (m, 2H, CH_2). LRMS (ESI) m/z calcd for $C_{12}H_{10}FNO^+$ [M + H^+] 204.1, found 204.0.

8-Fluoro-2,3-dihydro-1*H*-carbazol-4(9*H*)-one (2d′)[7] Following the general procedure, **2d′** was purified by silica gel column chromatography (PE). R_f = 0.75 (EA/PE = 20/80). Yield: 22%, white solid, mp 224-226 ^{o}C. ^{1}H NMR (400 MHz, DMSO) δ 12.11 (br, 1H, NH), 8.25 (d, *J* = 7.6 Hz, 1H, H_{arom}), 7.51 (d, *J* = 7.6 Hz, 1H, H_{arom}), 7.33 (t, *J* = 7.6, 7.6 Hz, 1H, H_{arom}), 3.04 (t, *J* = 6.4 Hz, 2H, CH_2), 2.48 (t, *J* = 6.8 Hz, 2H, CH_2), 2.15-2.01 (m, 2H, CH_2). ^{13}C NMR (100 MHz, DMSO) δ 193.29, 158.88 (d, J_{C-F} = 233 Hz), 154.28, 132.91, 125.48, 113.14 (d, J_{C-F} = 9.7 Hz), 110.63 (d, J_{C-F} = 25.5 Hz), 105.76, 105.51, 37.23, 23.76, 23.23. LRMS (ESI) m/z calcd for $C_{12}H_{10}FNO^+$ [M + H^+] 204.1, found 204.3.

6-(Trifluoromethyl)-2,3-dihydro-1*H*-carbazol-4(9*H*)-one (2e)[6] Following the general procedure, **2e** was purified by silica gel column chromatography (PE). R_f = 0.60 (EA/PE = 10/90). Yield: 49%, white solid, mp 207-209 ^{o}C. ^{1}H NMR (400 MHz, DMSO) δ 11.96 (br, 1H, NH), 7.60 (m, 1H, H_{arom}), 7.40 (m, 1H, H_{arom}), 7.01 (m, 1H, H_{arom}), 2.96 (t, *J* = 6.0 Hz, 2H, CH_2), 2.43 (t, *J* = 6.0 Hz, 2H, CH_2), 2.13 (m, 2H, CH_2). LRMS (ESI) m/z calcd for $C_{13}H_{10}F_3NO^+$ [M + H^+] 254.1, found 254.1.

8-(Trifluoromethyl)-2,3-dihydro-1*H*-carbazol-4(9H)-one (2e′) Following the general procedure, **2e′** was purified by silica gel column chromatography (PE). R_f = 0.70 (EA/PE = 10/90). Yield: 41%, white solid, mp 183-185 ^{o}C. ^{1}H NMR (400MHz, DMSO) δ 12.34 (br, 1H, NH), 7.76 (d, *J* = 8.0 Hz, 1H, H_{arom}), 7.11 (m, 1H, H_{arom}), 7.02 (m, 1H, H_{arom}), 2.98 (t, *J* = 6.4 Hz, 2H, CH_2), 2.45 (t, *J* = 6.4 Hz, 2H, CH_2), 2.13 (m, 2H, CH_2). ^{13}C NMR (100 MHz, DMSO) δ 193.62, 154.95, 138.17, 125.78(q, J_{C-F} = 243 Hz), 124.68, 122.92, 122.61, 119.51 (q, J_{C-F} = 3.3 Hz), 117.47 (q, J_{C-F} = 3.5 Hz), 112.87, 38.11, 23.71, 23.16. HRMS (ESI) m/z calcd for $C_{13}H_{11}F_3NO^+$ [M + H^+] 254.0787, found 254.0789.

7-Nitro-2,3-dihydro-1*H*-carbazol-4(9*H*)-one (2f)[8] Following the general procedure, **2f** was

purified by silica gel column chromatography (EA/PE = 50/50). R_f = 0.40 (EA/PE = 70/30). Yield: 79%, yellow solid, mp>250 ^oC. ^1H NMR (400 MHz, DMSO) δ 12.52 (br, 1H, NH), 8.24 (d, *J* = 1.2 Hz, 1H, H_{arom}), 8.30 (d, *J* = 1.4 Hz, 1H, H_{arom}), 8.07 (m, 2H, H_{arom}), 3.06 (t, *J* = 6.0 Hz, 2H, CH_2), 2.48 (t, *J* = 6.4 Hz, 2H, CH_2), 2.16 (m, 2H, CH_2). LRMS (ESI) m/z calcd for $C_{12}H_{10}N_2NaO_3^+$ [M + Na^+] 253.1, found 253.4.

2,2-Dimethyl-2,3-dihydro-1*H*-carbazol-4(9H)-one (2g)[9] Following the general procedure, **2g** was purified by silica gel column chromatography (PE). R_f = 0.70 (EA/PE = 10/90). Yield: 87%, white solid, mp 212-214 ^oC. ^1H NMR (400 MHz, $CDCl_3$) δ 9.82 (br, 1H, NH), δ 8.23 (m, 1H, H_{arom}), 7.41 (m, 2H, H_{arom}), 7.25 (m, 2H, H_{arom}), 2.87 (s, 2H, CH_2), 2.51 (s, 2H, CH_2), 1.17 (s, 6H, CH_3). LRMS (ESI) m/z calcd for $C_{14}H_{15}NNaO^+$ [M + Na^+] 236.1, found 236.5.

8,9,10,11-Tetrahydro-7*H*-benzo[*a*]carbazol-7-one (2h) Following the general procedure, **2h** was purified by silica gel column chromatography (PE). R_f= 0.70 (EA/PE = 20/80). Yield: 84%, white solid, mp 92-94 ^oC. ^1H NMR (400 MHz, DMSO): δ 9.26 (br, 1H, NH), δ 8.36 (d, *J* = 8.8 Hz, 1H, H_{arom}), 8.00 (m, 1H, H_{arom}), 7.70 (d, *J* = 8.8 Hz, 1H, H_{arom}), 7.57 (t, *J* = 8.8 Hz, 1H, H_{arom}), 7.48 (t, *J* = 8.8 Hz, 1H, H_{arom}), 3.12 (t, *J* = 6.4 Hz, 2H, CH_2), 2.67 (t, *J* = 6.4 Hz, 2H, CH_2), 2.33 (m, 2H, CH_2). ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.75, 161.38, 133.76, 132.50, 132.19, 129.50, 129.20, 128.38, 127.72, 127.61, 125.82, 125.76, 112.64, 36.75, 29.27, 21.27. HRMS (ESI) m/z calcd for $C_{16}H_{14}NO^+$ [M + Na^+] 258.1070, found 258.1071.

9-Methyl-2,3-dihydro-1*H*-carbazol-4(9*H*)-one (2i)[10] Following the general procedure, **2i** was purified by silica gel column chromatography (EA/PE = 40/60). R_f = 0.29 (EA/PE = 50/50). Yield: 82%, white solid, mp 205-207 ^oC. ^1H NMR (400MHz, DMSO) δ 8.30-8.22 (m, 1H, H_{arom}), 7.34-7.23 (m, 3H, H_{arom}), 3.72 (s, 3H, CH_3), 2.94 (t, *J* = 6.0 Hz, 2H, CH_2), 2.61-2.57 (m, 2H, CH_2), 2.30-2.23 (m, 2H, CH_2). LRMS (ESI) m/z calcd for $C_{13}H_{13}NNaO^+$ [M + Na^+] 222.1, found 222.3.

7,9-Dimethyl-2,3-dihydro-1*H*-carbazol-4(9*H*)-one (2j) Following the general procedure, **2j** was purified by silica gel column chromatography (EA/PE = 10/90). R_f = 0.54 (EA/PE = 20/80). Yield: 74%, white solid, mp 239-241 ^oC. ^1H NMR (400MHz, $CDCl_3$) δ 8.10 (d, *J* = 8.0 Hz, 1H, H_{arom}), 7.17-7.01 (m, 2H, H_{arom}), 3.63 (s, 3H, CH_3), 2.93-2.84 (m, 2H, CH_2), 2.58-2.51 (m, 2H, CH_2), 2.48 (s, 3H, CH_3), 2.25-2.19 (m, 2H, CH_2). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.65, 151.39, 137.81,

132.88, 124.08, 122.48, 121.29, 112.67, 109.22, 37.84, 29.76, 23.36, 22.19, 21.89. HRMS (ESI) m/z calcd for $C_{14}H_{15}NNaO^+$ [M + Na^+] 236.1046, found 236.1046.

9-Propyl-2,3-dihydro-1*H*-carbazol-4(9H)-one (2k) Following the general procedure, **2k** was purified by silica gel column chromatography (EA/PE = 15/85). R_f = 0.42 (EA/PE = 50/50). Yield: 31%, yellow solid, mp 153-155 ^oC. 1H NMR (400MHz, $CDCl_3$): δ 8.27 (dd, *J* = 6.0, 2.5 Hz, 1H, Harom), 7.35 -7.22 (m, 3H, Harom), 4.07 (t, *J* = 7.3 Hz, 2H, NCH2), 2.94 (t, *J* = 6.2 Hz, 2H, CH_2), 2.66 -2.52 (m, 2H, CH_2), 2.26 (p, *J* = 6.3 Hz, 2H, CH_2), 1.85 (h, *J* = 7.4 Hz, 2H, CH_2), 0.97 (t, *J* = 7.4 Hz, 3H, CH_3). HRMS (ESI) m/z calcd for $C_{15}H_{17}NNaO^+$ [M + Na^+] 250.1202, found 250.1207.

9-Phenyl-2,3-dihydro-1*H*-carbazol-4(9*H*)-one (2l)[10] Following the general procedure, **2l** was purified by silica gel column chromatography (EA/PE = 10/90). R_f = 0.70 (EA/PE = 30/70). Yield: 73%, solid, mp 153-154 ^oC. ^1H NMR (400MHz, $CDCl_3$) δ 8.32 (d, *J* = 7.6 Hz, 2H, H_{arom}), 7.65-7.48 (m, 1H, H_{arom}), 7.40 (d, *J* = 7.6 Hz, 1H, H_{arom}), 7.31 (t, *J* = 7.6 Hz, 1H, H_{arom}), 7.22 (t, *J* = 7.6 Hz, 1H, H_{arom}), 7.16 (d, *J* = 8.0 Hz, 2H, H_{arom}), 2.82 (t, *J* = 6.4 Hz, 2H, CH_2), 2.65 (t, *J* = 6.4 Hz, 2H, CH_2), 2.27-2.17 (m, 2H, CH_2). LRMS (ESI) m/z calcd for $C_{36}H_{30}N_2NaO_2^+$ [2M + Na^+] 545.2, found 545.2.

9-(4-Methoxyphenyl)-2,3-dihydro-1*H*-carbazol-4(9H)-one (2m) Following the general procedure, **2m** was purified by silica gel column chromatography (PE). R_f = 0.60 (EA/PE = 20/80). Yield: 76%, solid, mp 123-125 ^oC. ^1H NMR (400MHz, DMSO) δ 8.32 (d, *J* = 7.6 Hz, 1H, H_{arom}), 7.28-7.32 (m, 3H, H_{arom}), 7.20-7.24 (m, 1H, H_{arom}), 7.08-7.14 (m, 3H, H_{arom}), 3.93 (s, 3H, CH_3), 2.80 (t, *J* = 6.4 Hz, 2H, CH_2), 2.64 (t, *J* = 6.4 Hz, 2H, CH_2), 2.22 (m, 2H, CH_2). ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.31, 159.72, 152.36, 138.46, 128.72, 128.40, 124.63, 123.29, 122.84,121.49, 114.99, 113.27, 110.42, 56.66, 38.10, 23.02, 23.01. HRMS (ESI) m/z calcd for $C_{19}H_{17}NNaO_2^+$ [M + Na^+] 314.1151, found 314.1151.

9-(4-Nitrophenyl)-2,3-dihydro-1*H*-carbazol-4(9H)-one (2n) Following the general procedure, **2n** was purified by silica gel column chromatography (PE). R_f = 0.50 (EA/PE = 20/80). Yield: 70%, yellow solid, mp 166-168 ^oC. ^1H NMR (400MHz, $CDCl_3$) δ 8.46 (d, *J* = 8.0 Hz, 2H, H_{arom}), 8.32 (d, *J* = 8.0 Hz, 1H, H_{arom}), 7.63 (d, J = 8.0 Hz, 2H, H_{arom}), 7.34 (t, *J* = 8.0 Hz, 1H, H_{arom}),

7.26 (t, J = 8.0 Hz, 1H, H_{arom}), 7.21 (d, J = 8.0 Hz, 1H, H_{arom}), 2.87 (t, J = 6.0 Hz, 2H, CH_2), 2.63 (t, J = 6.0 Hz, 2H, CH_2), 2.27-2.21 (m, 2H, CH_2). ^{13}C NMR (100 MHz, DMSO) δ 194.09, 152.71, 147.23, 141.74, 137.24, 128.73, 125.74, 125.06, 124.15, 123.60, 121.16, 113.79, 110.94, 38.14, 23.56, 23.09. HRMS (ESI) m/z calcd for $C_{18}H_{14}N_2NaO_3^+$ [M + Na^+] 329.0897, found 329.0898.

^{1}H and ^{13}C NMR Spectra of Substrates 3

3-Phenyl-4-(propylamino)pent-3-en-2-one (3a) Following the general procedure, **3a** was purified by silica gel column chromatography (PE). R_f = 0.58 (EA/PE = 20/80). Yield: 22%, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.10 (br, 1H, NH), 7.33 (t, J = 7.6 Hz, 2H, H_{arom}), 7.29 -.23 (m, 1H, H_{arom}), 7.15 (d, J = 7.6 Hz, 2H, H_{arom}), 3.26 (dd, J = 12.8, 6.7 Hz, 2H, CH_2), 1.81 (s, 3H, CH_3), 1.74 (s, 3H, CH_3), 1.62 (m, 2H, CH_2), 1.04 (t, J = 7.6 Hz, 3H, CH_3). ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.00, 162.53, 141.17, 132.29, 128.45, 126.41, 109.20, 45.22, 29.03, 23.25, 16.74, 11.56. HRMS (ESI) m/z calcd for $C_{14}H_{19}NNaO^+$ [M + Na^+] 240.1359, found 240.1362.

4-(Benzylamino)-3-phenylpent-3-en-2-one (3b) Following the general procedure, **3b** was purified by silica gel column chromatography (PE). R_f = 0.46 (EA/PE = 20/80). Yield: 58%, yellow oil. ^{1}H NMR (400MHz, $CDCl_3$) δ 12.35 (br, 1H, NH), 7.40-7.23 (m, 8H, H_{arom}), 7.16 (d, J = 7.2 Hz, 2H, H_{arom}), 4.52 (d, J = 5.8 Hz, 2H, CH_2), 1.84 (s, 3H, CH_3), 1.71 (s, 3H, CH_3). ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.00, 162.27, 140.99, 138.06, 132.24, 128.86, 128.51, 127.45, 126.90, 126.54, 110.07, 47.23, 29.27, 16.85. HRMS (ESI) m/z calcd for $C_{18}H_{19}NNaO^+$ [M + Na^+] 288.1359, found 288.1361.

3-Phenyl-4-(phenylamino)pent-3-en-2-one (3c)[3] Following the general procedure, **3c** was purified by silica gel column chromatography (EA/PE = 10/90). R_f= 0.3 (EA/PE = 10/90). Yield: 87%, white solid, mp 105-107 °C. ^{1}H NMR (400MHz, $CDCl_3$) δ 7.43-7.09 (m, 10H, H_{arom}), 1.90 (s, 3H, CH_3), 1.74 (s, 3H, CH_3). LRMS (ESI) m/z calcd for $C_{17}H_{17}NNaO^+$ [M + Na^+] 274.1, found 274.0.

^{1}H and ^{13}C NMR Spectra of 3-Acetylindoles 4

9-Propyl-2,3-dihydro-1*H*-carbazol-4(9*H*)-one (4a) Following the general procedure,**4a** was purified by silica gel column chromatography (EA/PE = 40/60). R_f= 0.42 (EA/PE = 50/50). Yield:

74%, yellow solid, mp 153-155 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.27 (dd, *J* = 6.0, 2.4 Hz, 1H, H_{arom}), 7.35-7.22 (m, 3H, H_{arom}), 4.07 (t, *J* = 7.6 Hz, 2H, CH_2), 2.94 (t, *J* = 6.4 Hz, 2H, CH_2), 2.66-2.52 (m, 2H, CH_2), 2.26 (p, *J* = 6.4 Hz, 2H, CH_2), 1.85 (h, *J* = 7.6 Hz, 2H, CH_2), 0.97 (t, *J* = 7.6 Hz, 3H, CH_3). HRMS (ESI) m/z calcd for C15H17NNaO+ [M + Na^+] 250.1202, found 250.1207.

1-(1-Benzyl-2-methyl-1H-indol-3-yl)ethanone (4b)[11] Following the general procedure, **4b** was purified by silica gel column chromatography (EA/PE = 10/90). R_f = 0.48 (EA/PE = 20/80). Yield: 73%, white solid, mp 108-110 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, *J* = 8.0 Hz, 1H, H_{arom}), 7.29-7.20 (m, 6H, H_{arom}), 6.99 (d, *J* = 7.0 Hz, 2H, H_{arom}), 5.38 (s, 2H, CH_2), 2.74 (s, 3H, CH_3), 2.72 (s, 3H, CH_3). HRMS (ESI) m/z calcd for $C_{18}H_{17}NNaO^+$ [M + Na^+] 286.1202, found 286.1202.

1-(2-Methyl-1-phenyl-1H-indol-3-yl)ethanone (4c) Following the general procedure, **4c** was purified by silica gel column chromatography (EA/PE = 10/90). R_f = 0.51 (EA/PE = 20/80). Yield: 86%, solid, mp 130-132 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (d, *J* = 8.0 Hz, 1H, H_{arom}), 7.65-7.51 (m, 3H, H_{arom}), 7.38-7.24 (m, 3H, H_{arom}), 7.17 (t, *J* = 7.0 Hz, 1H, H_{arom}), 7.03 (d, *J* = 8.0 Hz, 1H, H_{arom}), 2.74 (s, 3H, CH_3), 2.59 (s, 3H, CH_3). ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.88, 145.11, 137.82, 136.25, 129.85, 129.05, 128.32, 126.23, 122.42, 122.40, 120.65, 115.09, 110.88, 31.73, 13.95. HRMS (ESI) m/z calcd for $C_{17}H_{15}NNaO^+$ [M + Na^+] 272.1046, found 272.1047.

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^{1}H and ^{13}C NMR Spectra of 1

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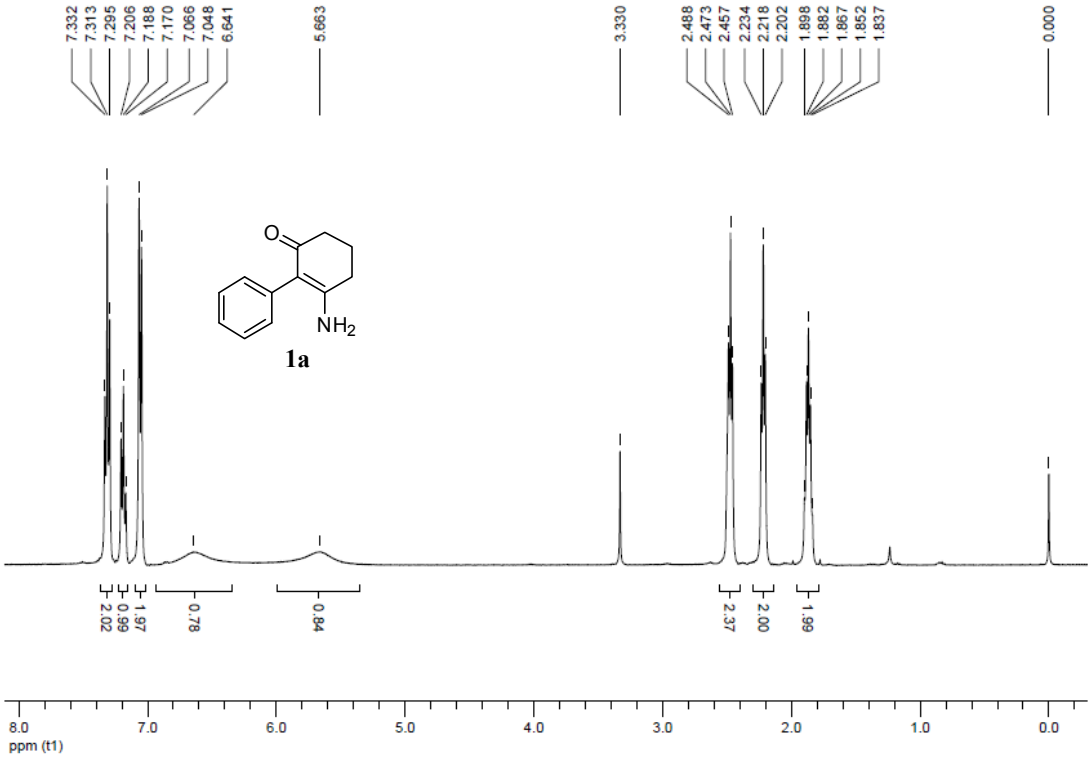

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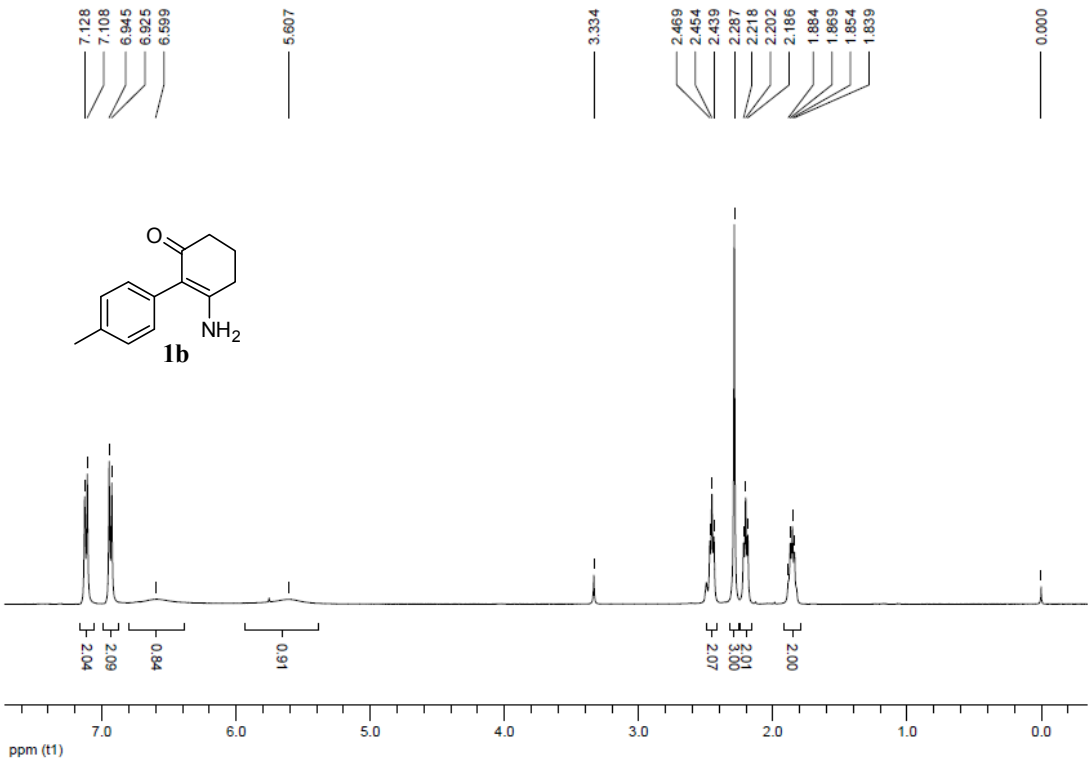

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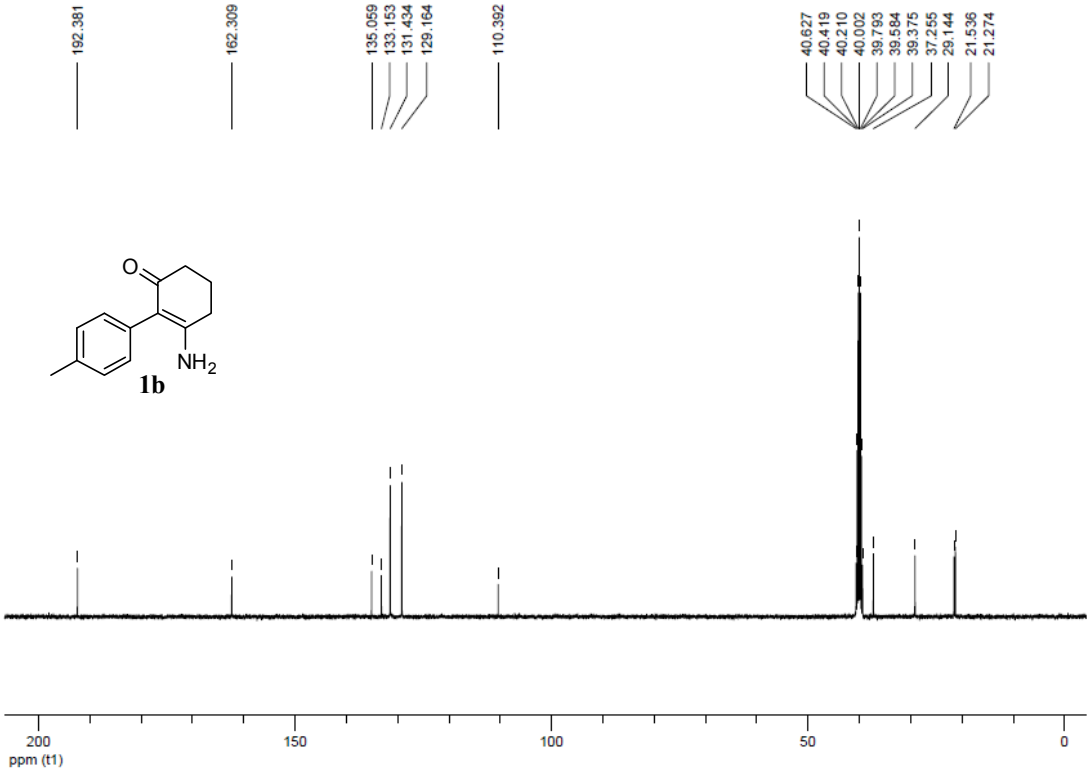

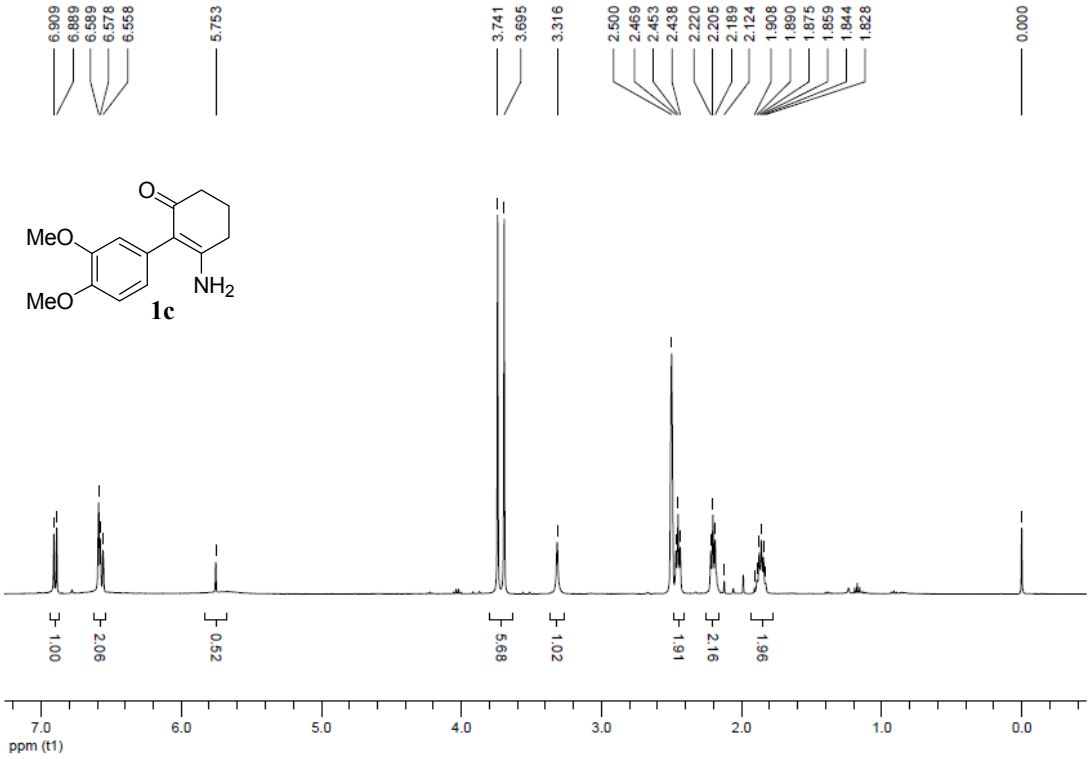

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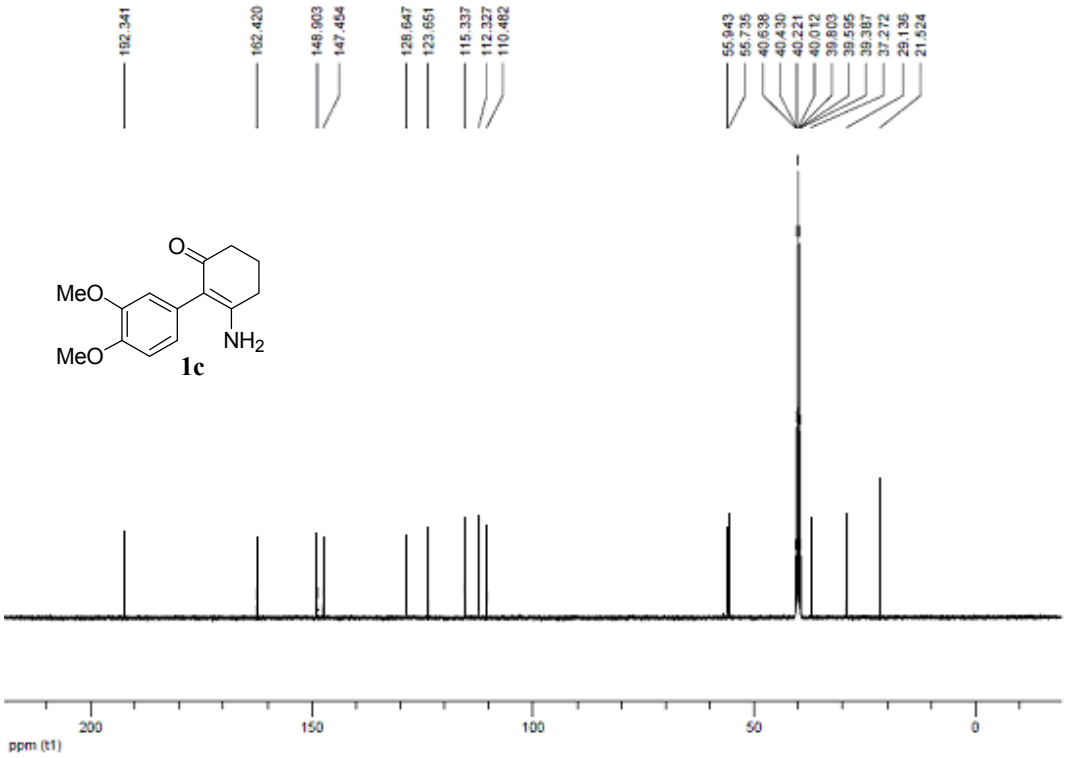

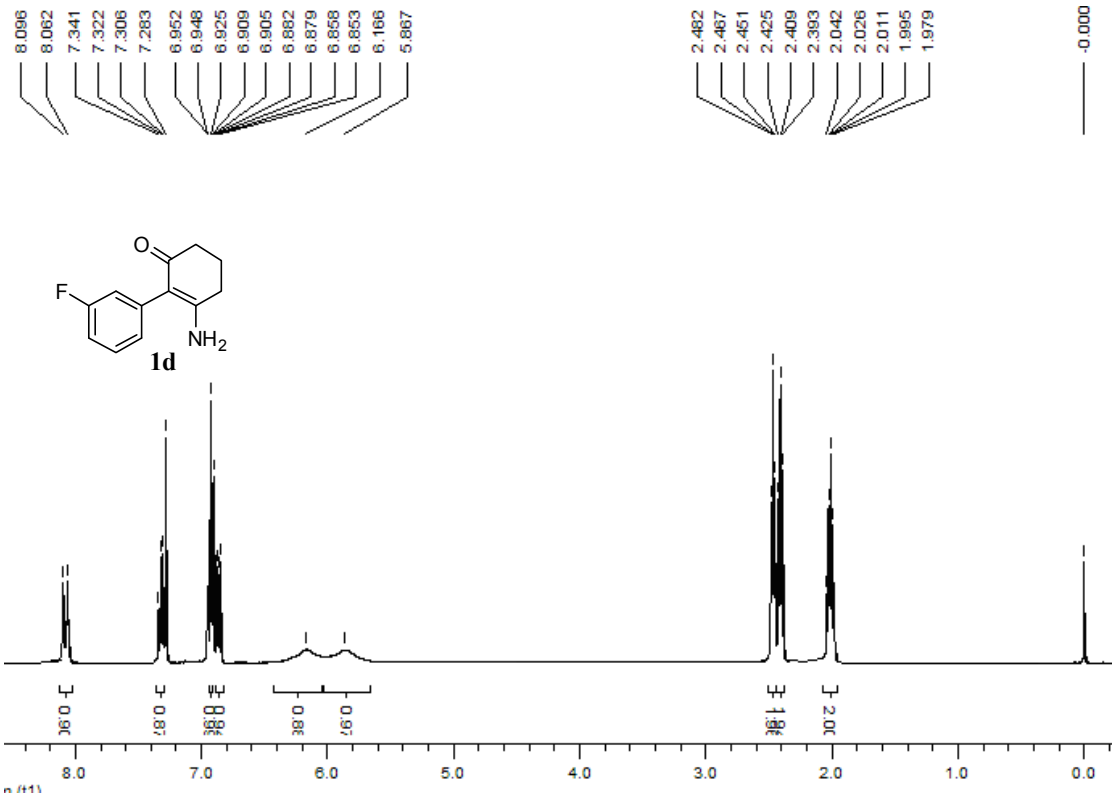

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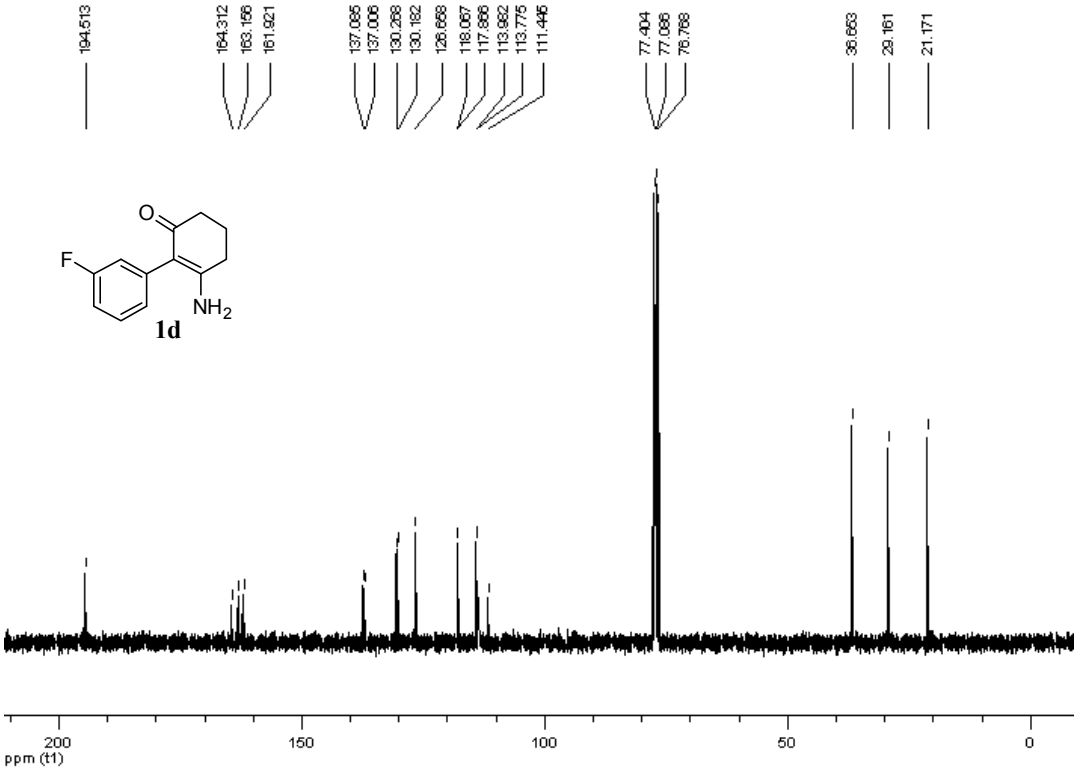

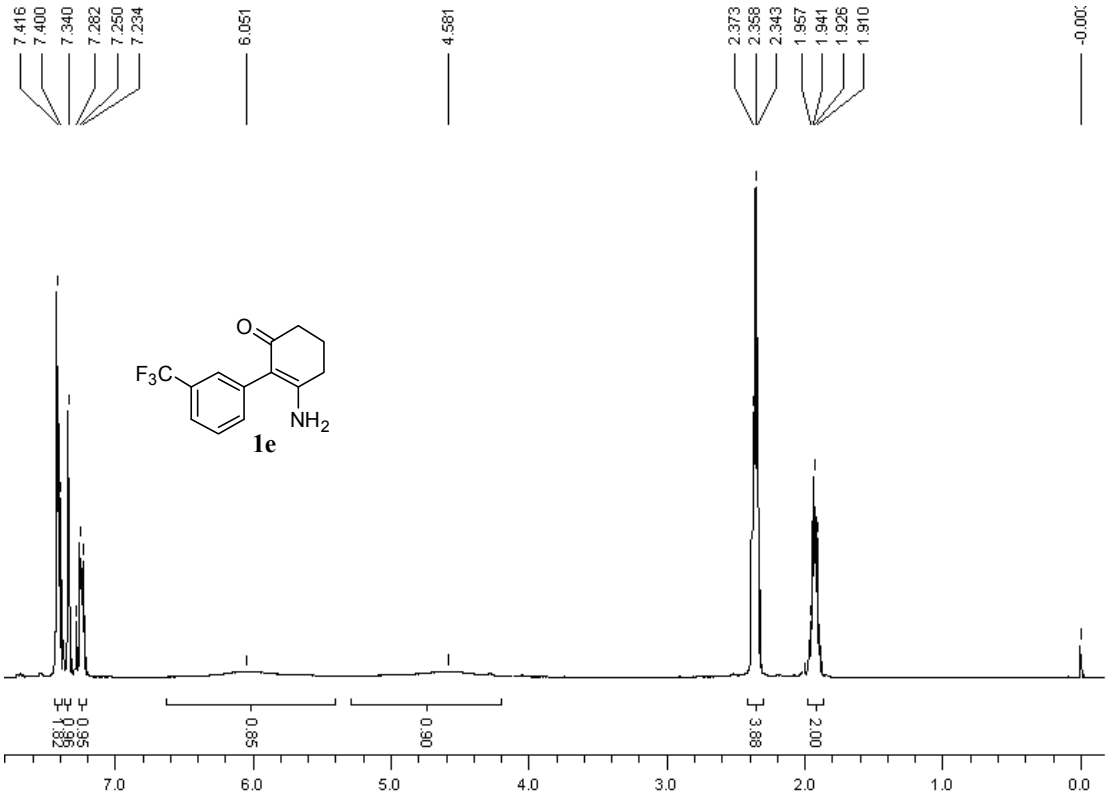

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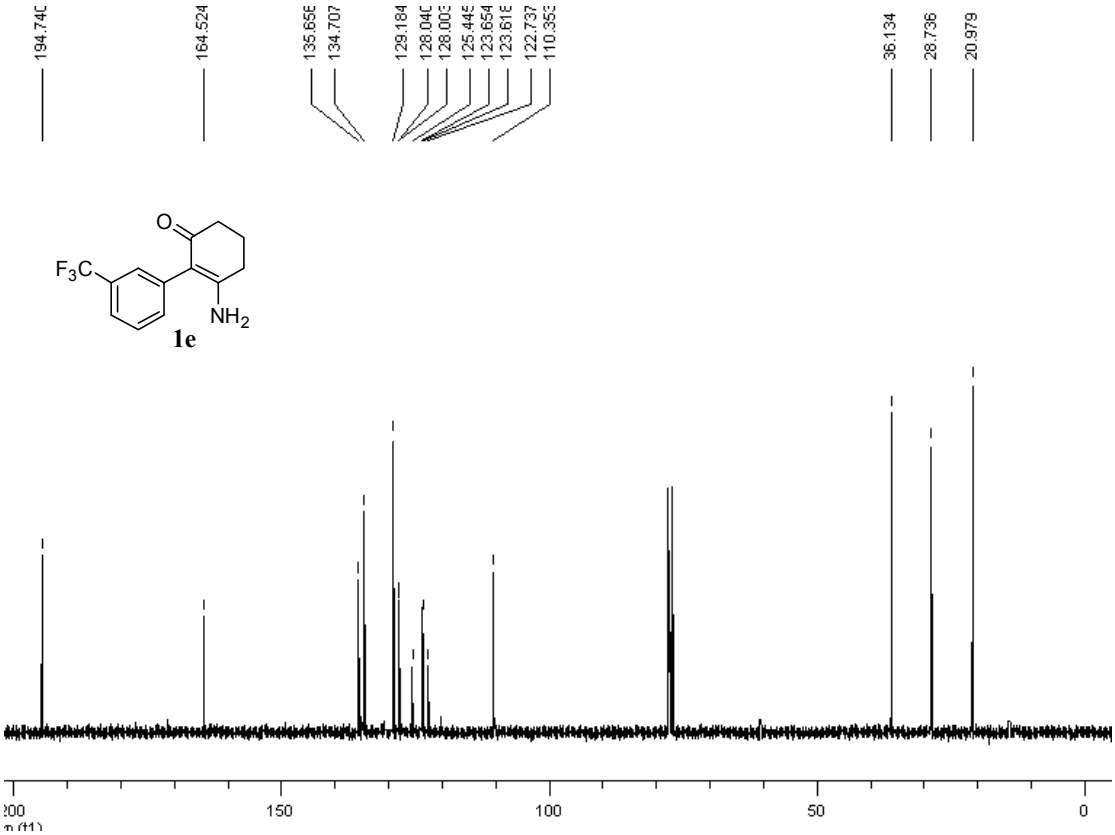

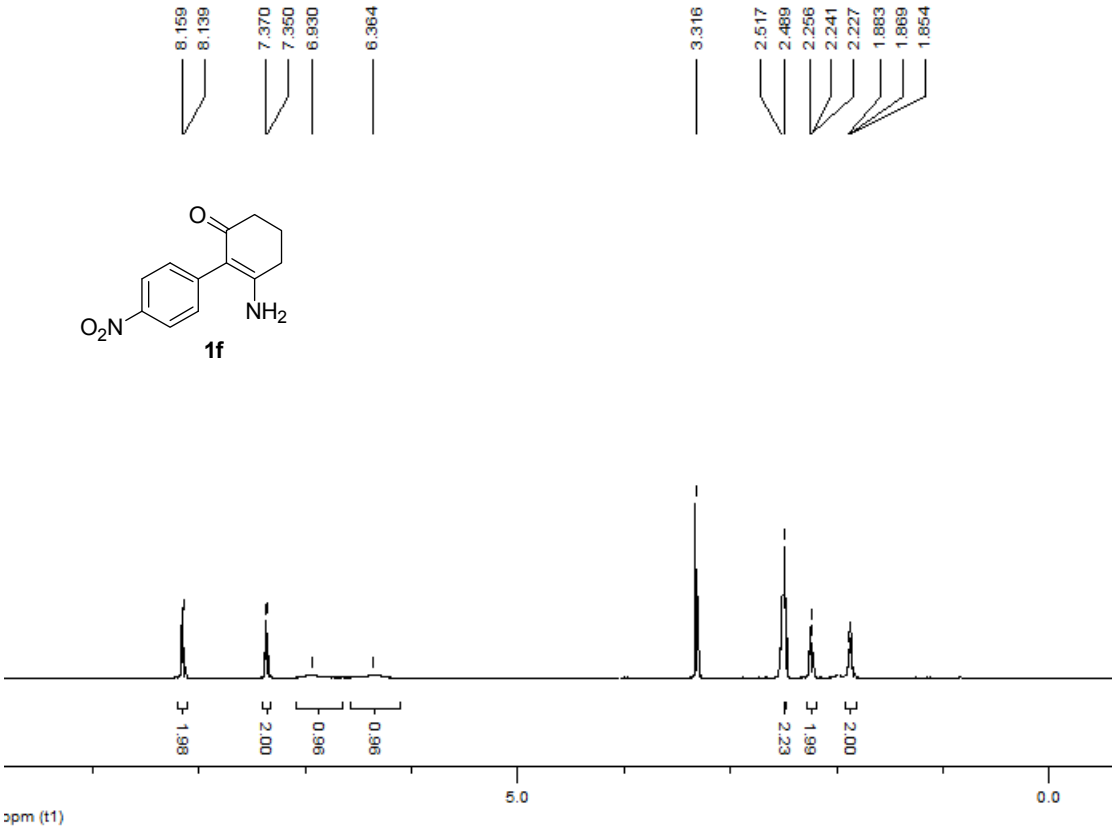

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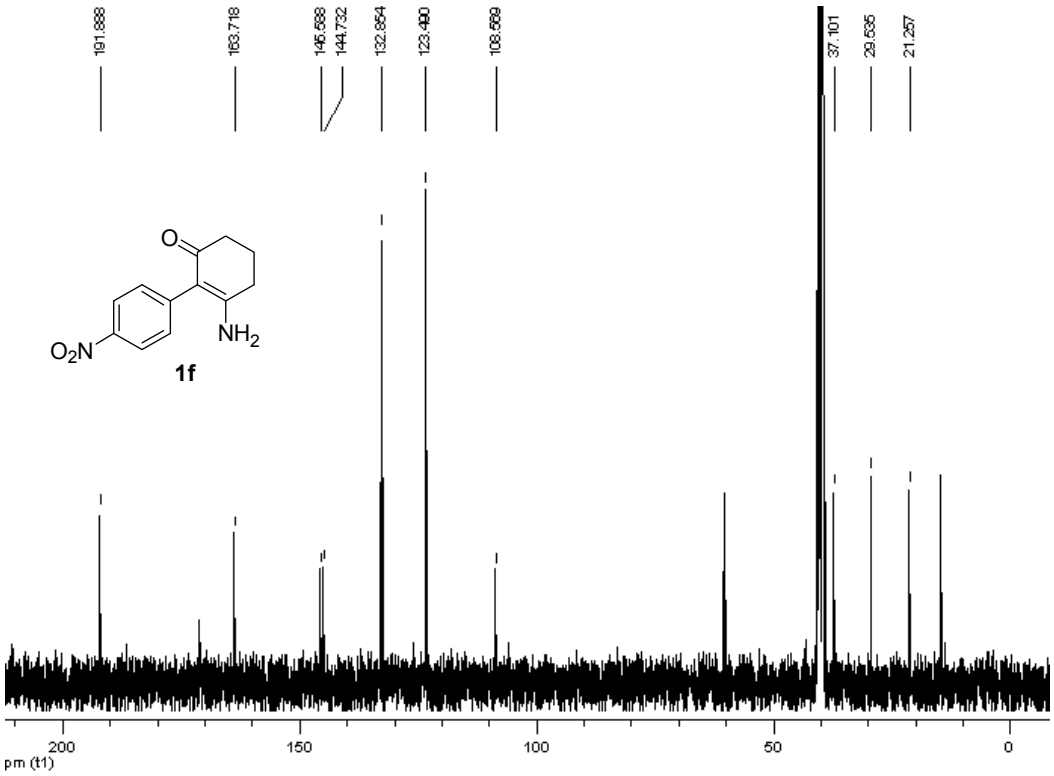

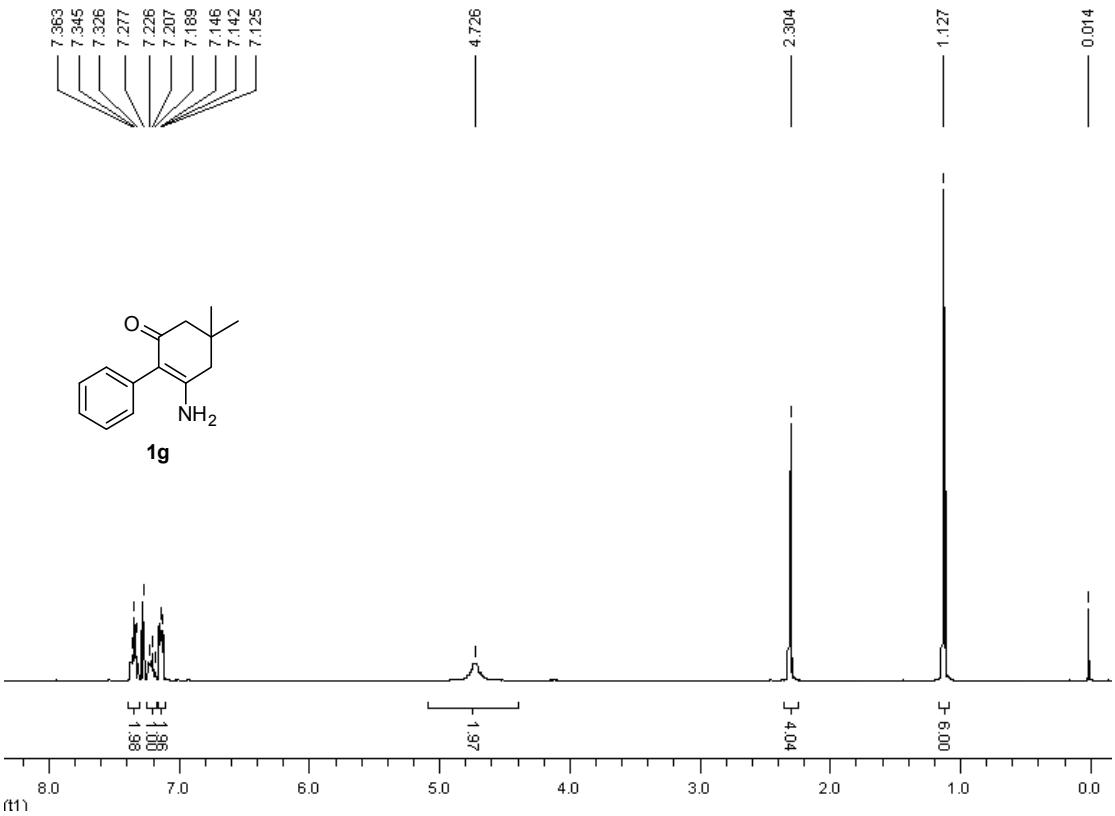

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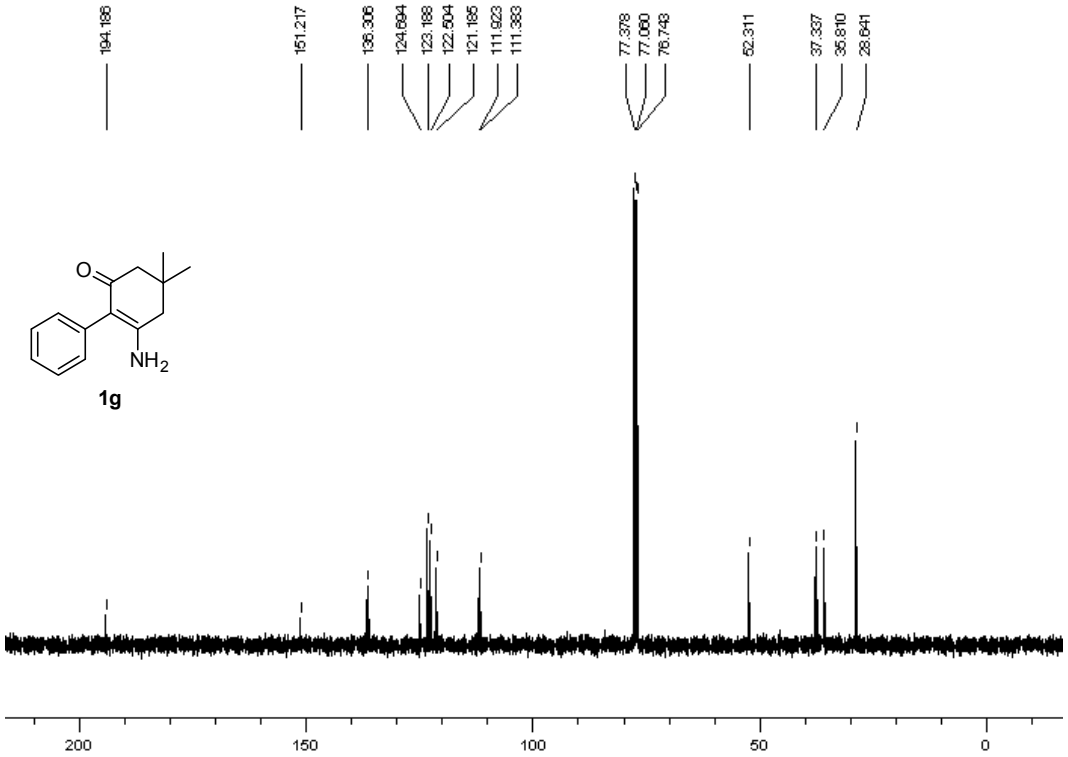

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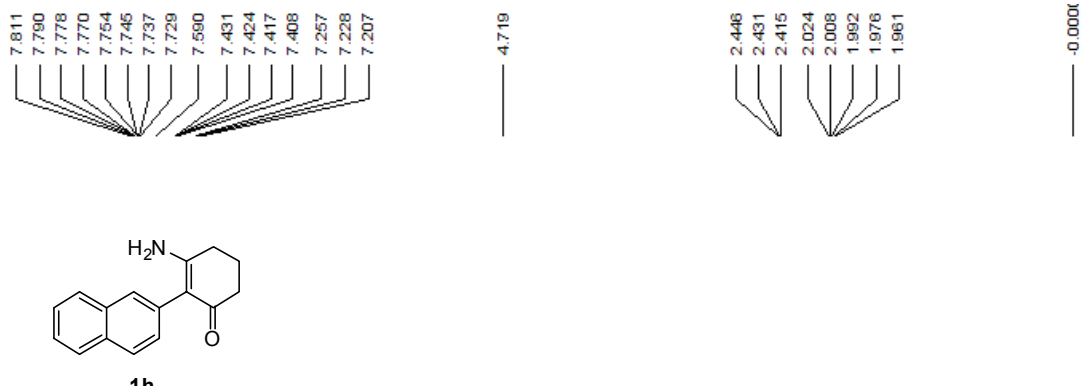

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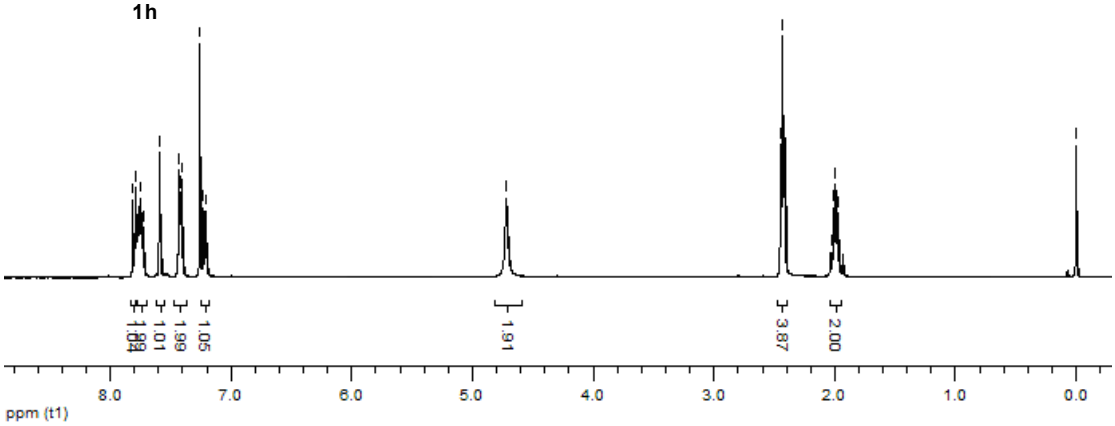

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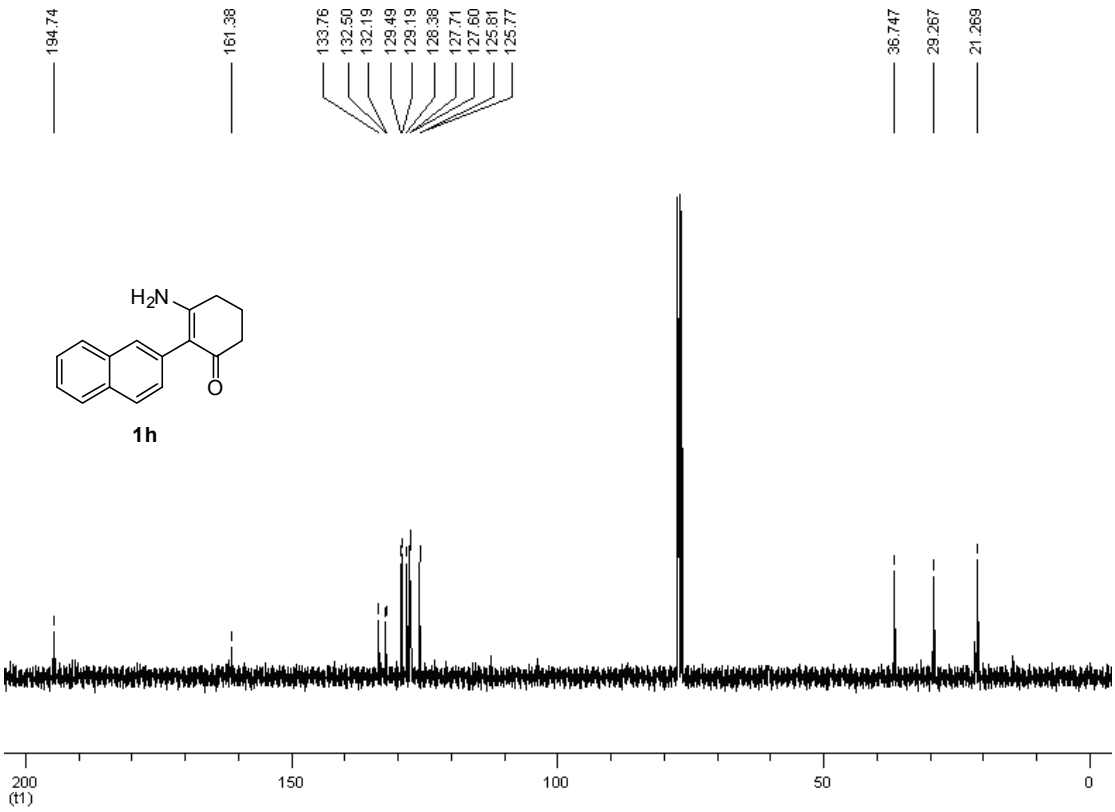

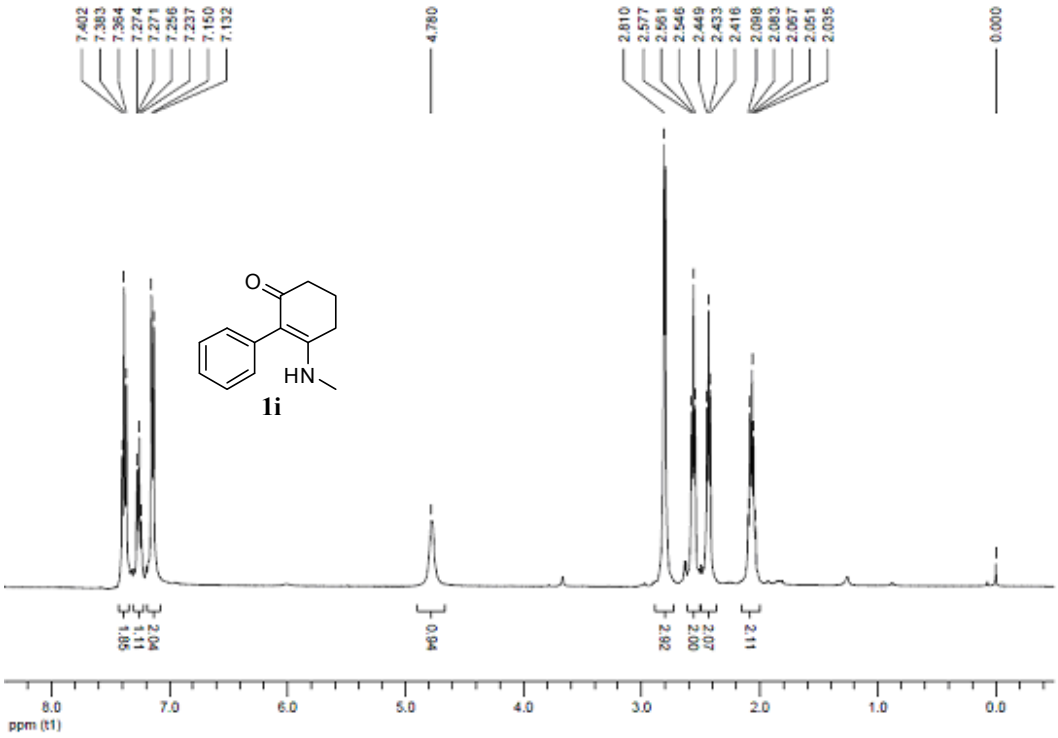

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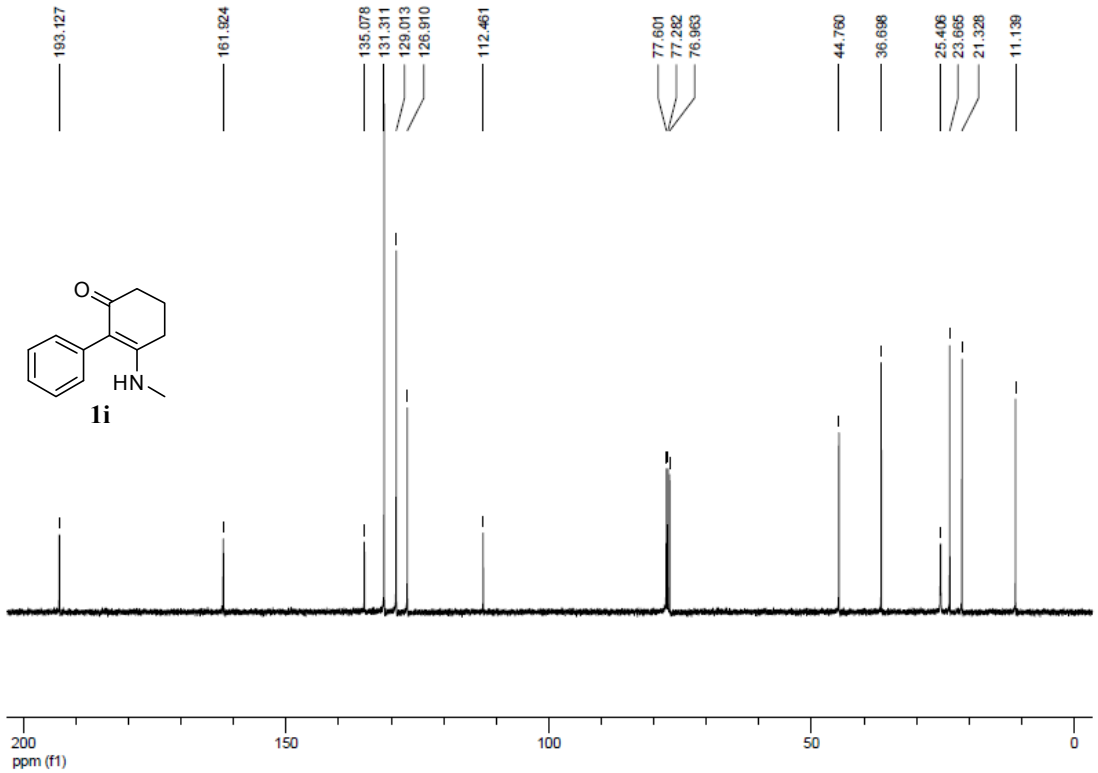

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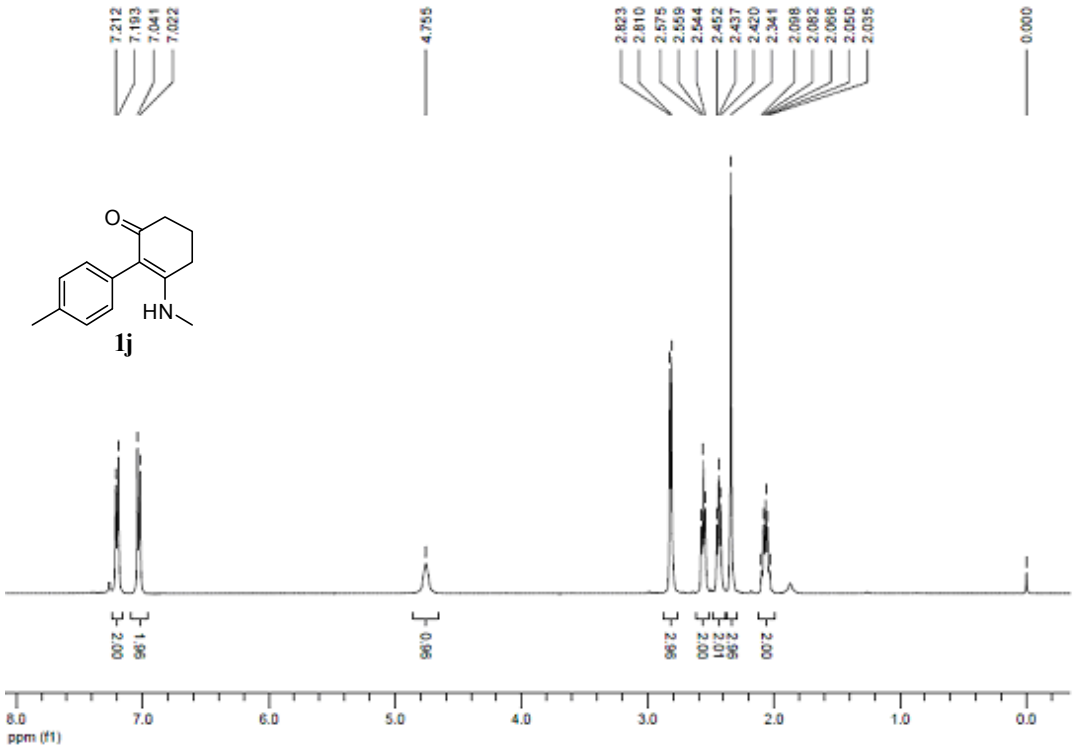

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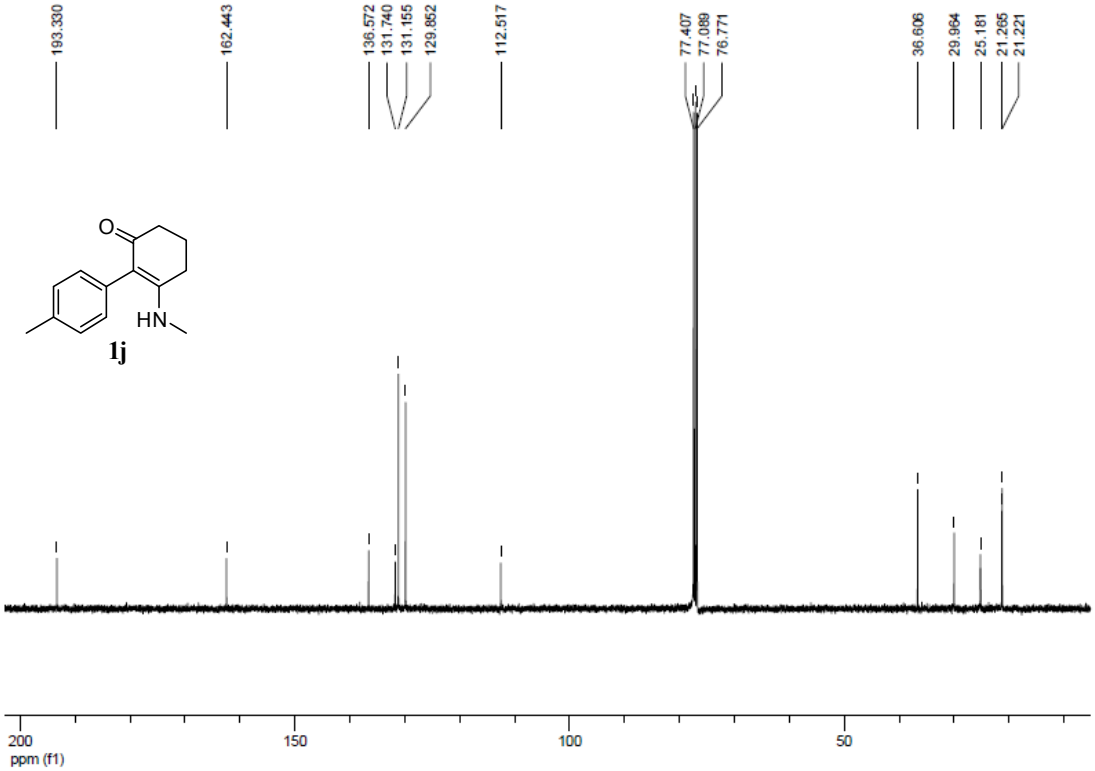

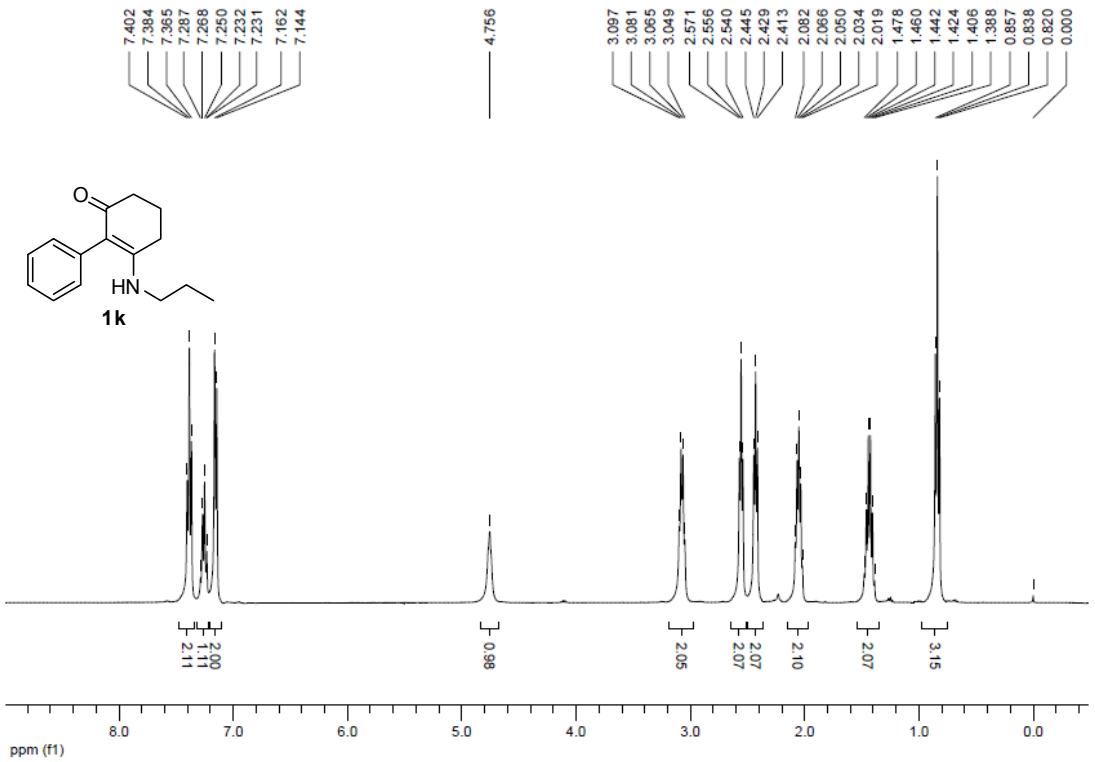

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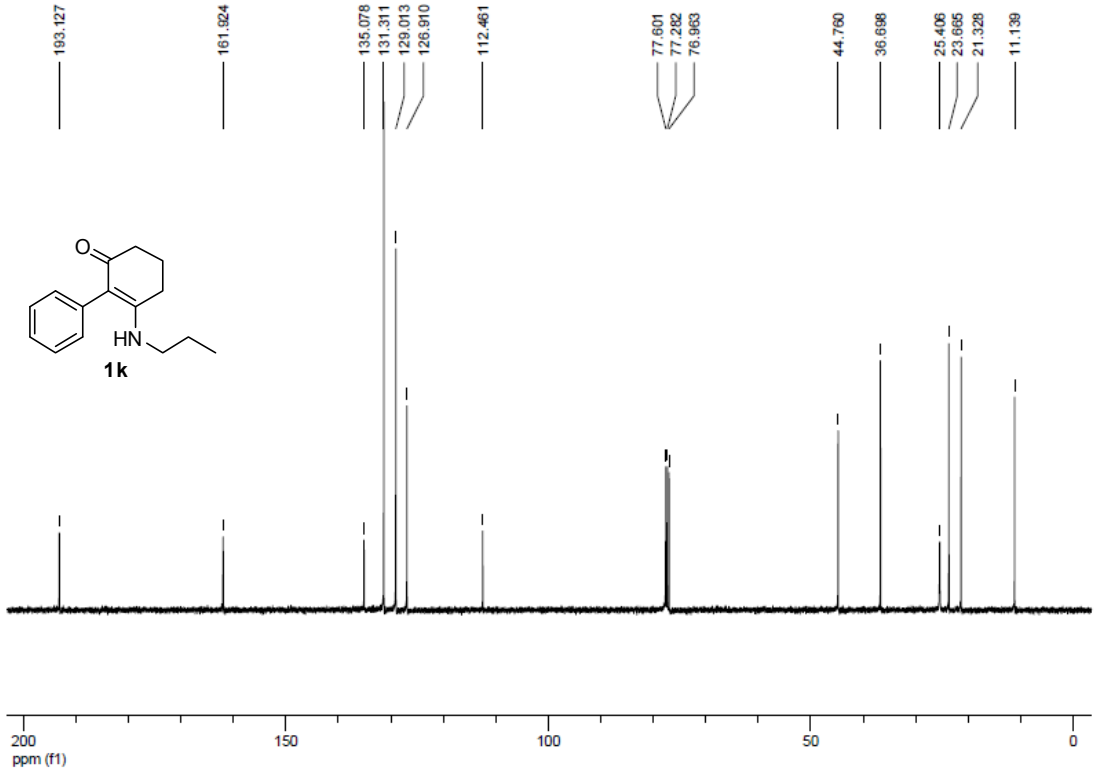

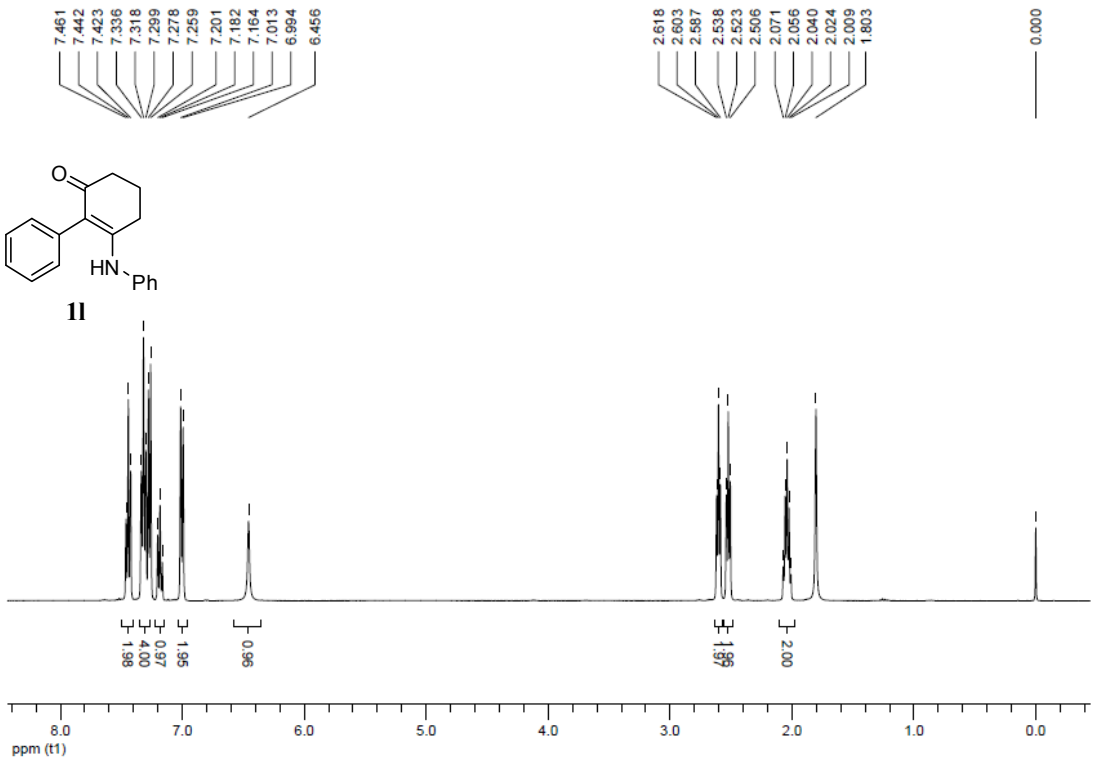

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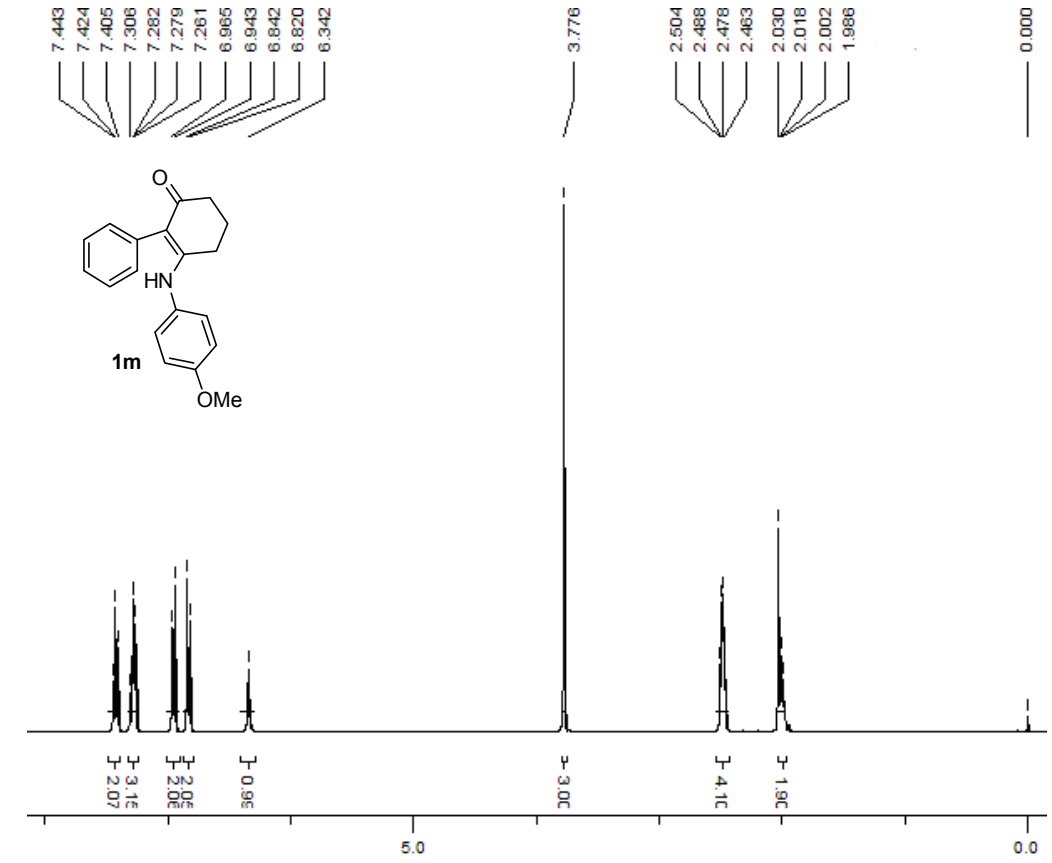

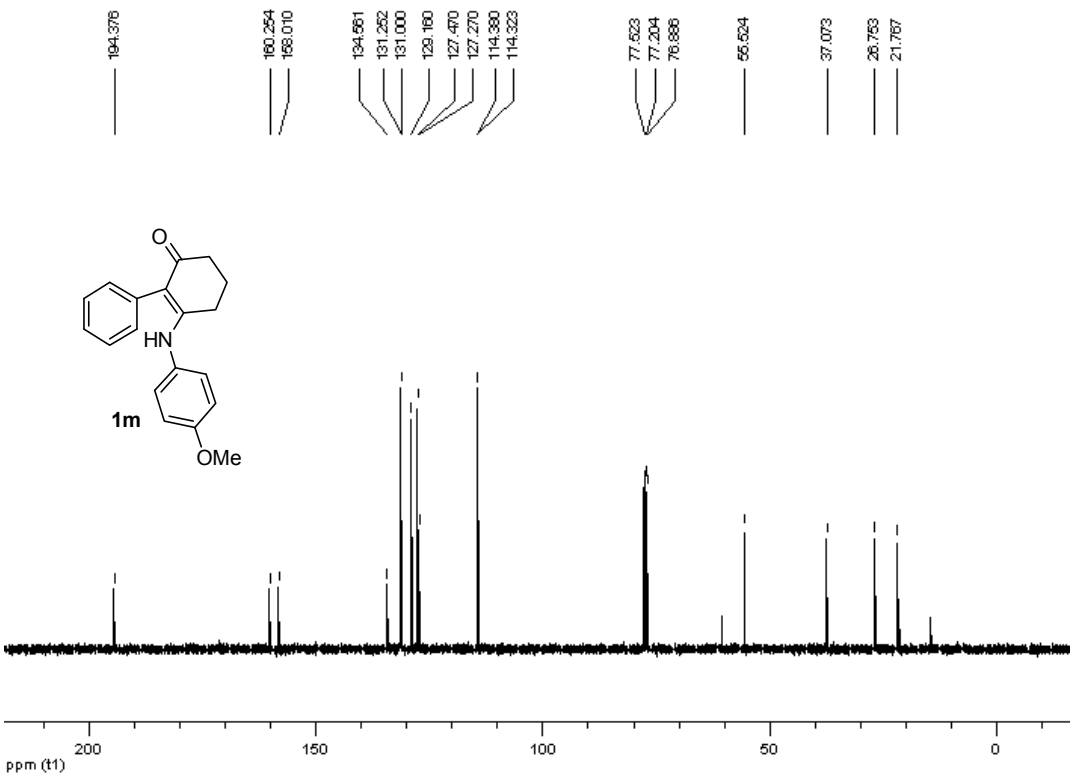

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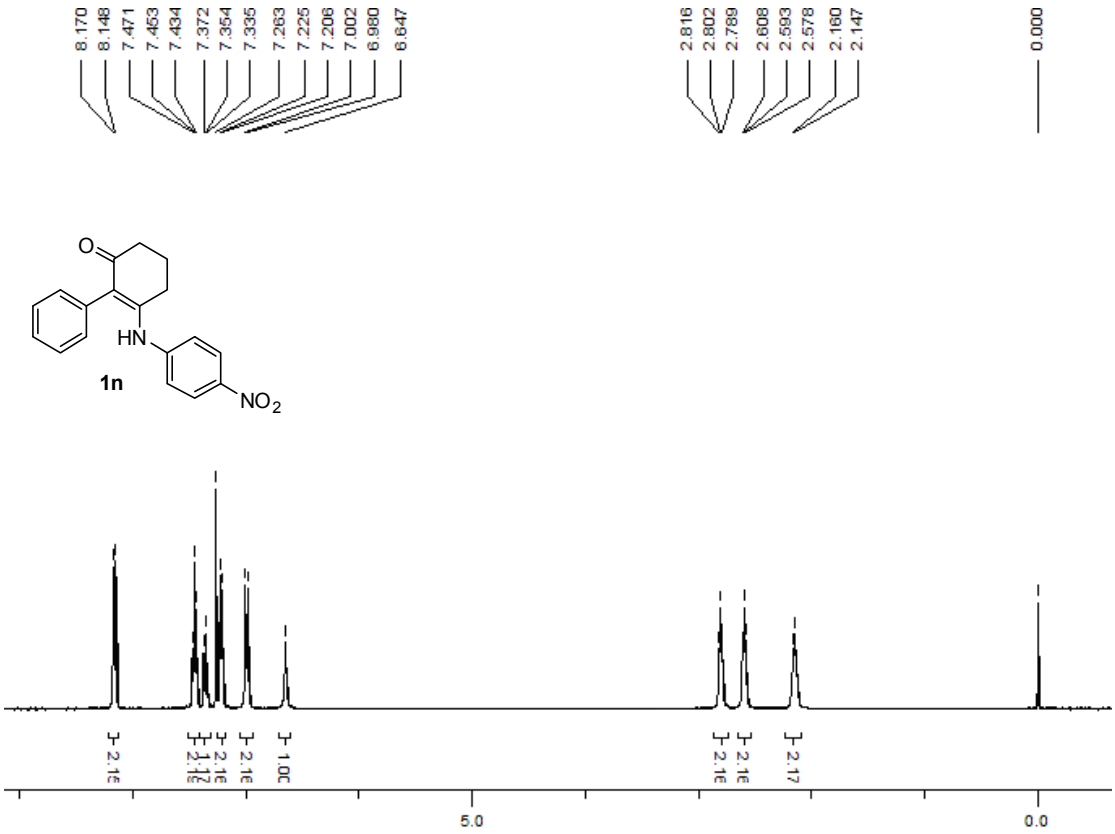

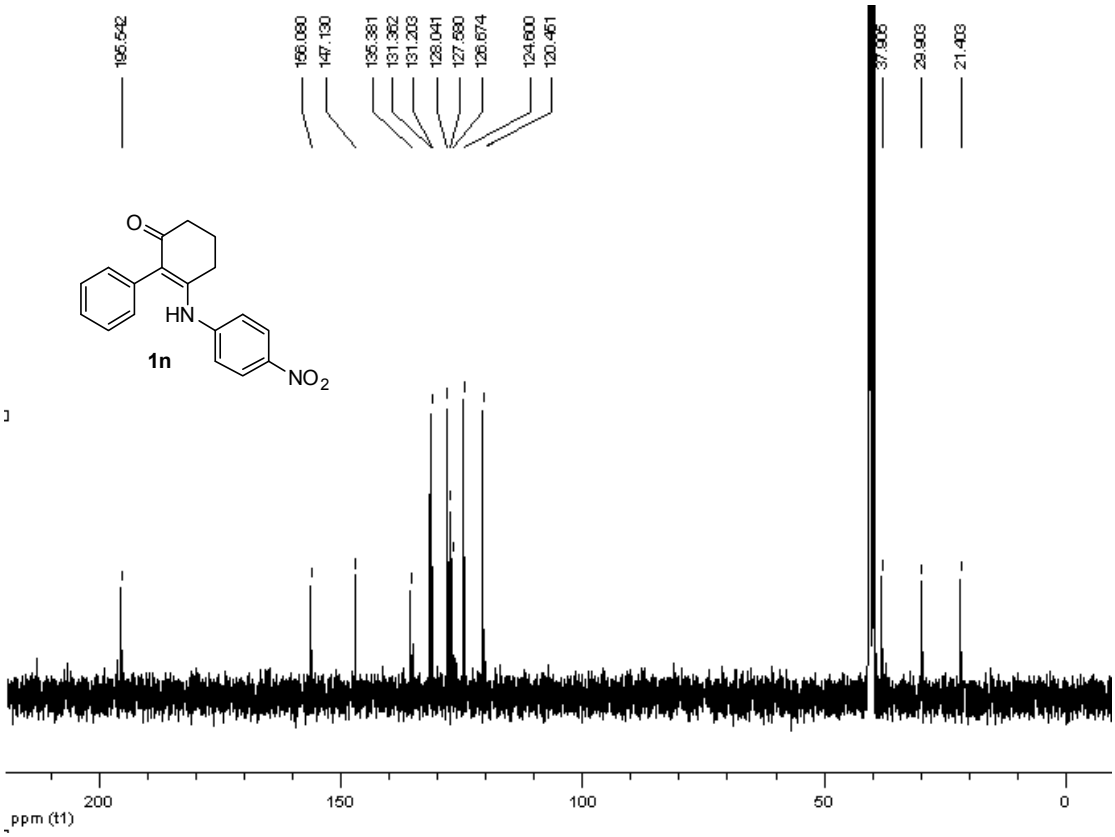

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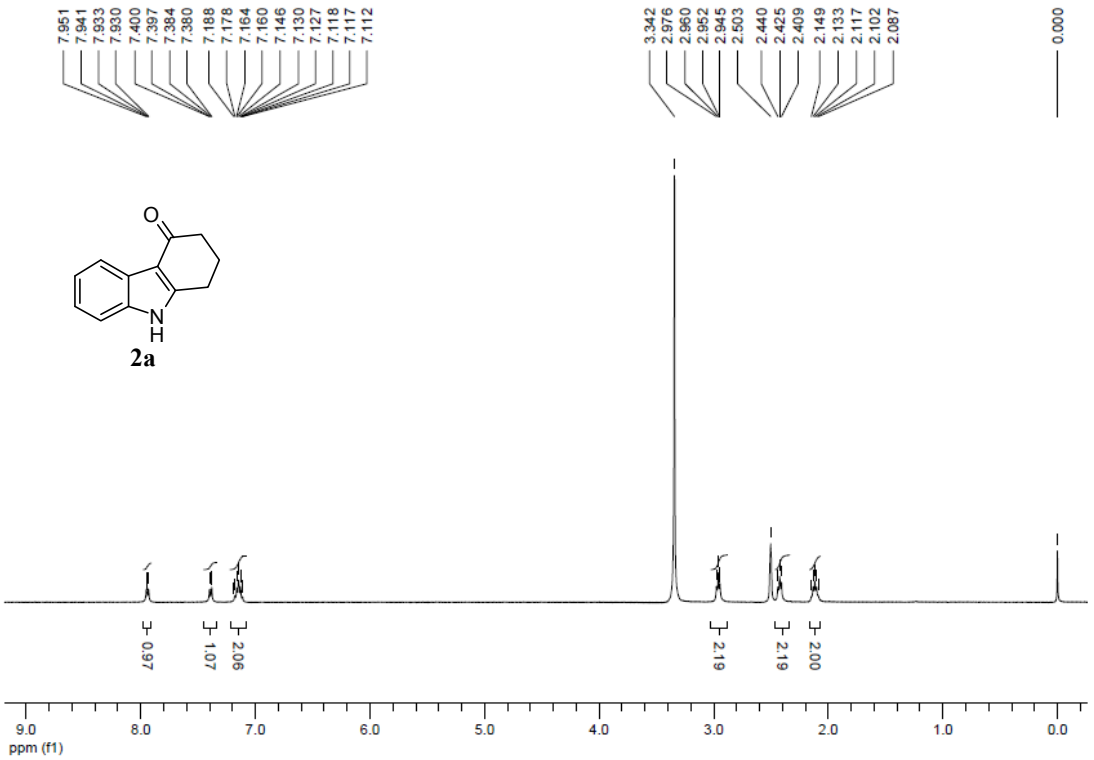
^{1}H and ^{13}C NMR Spectra of Carbazolones 2



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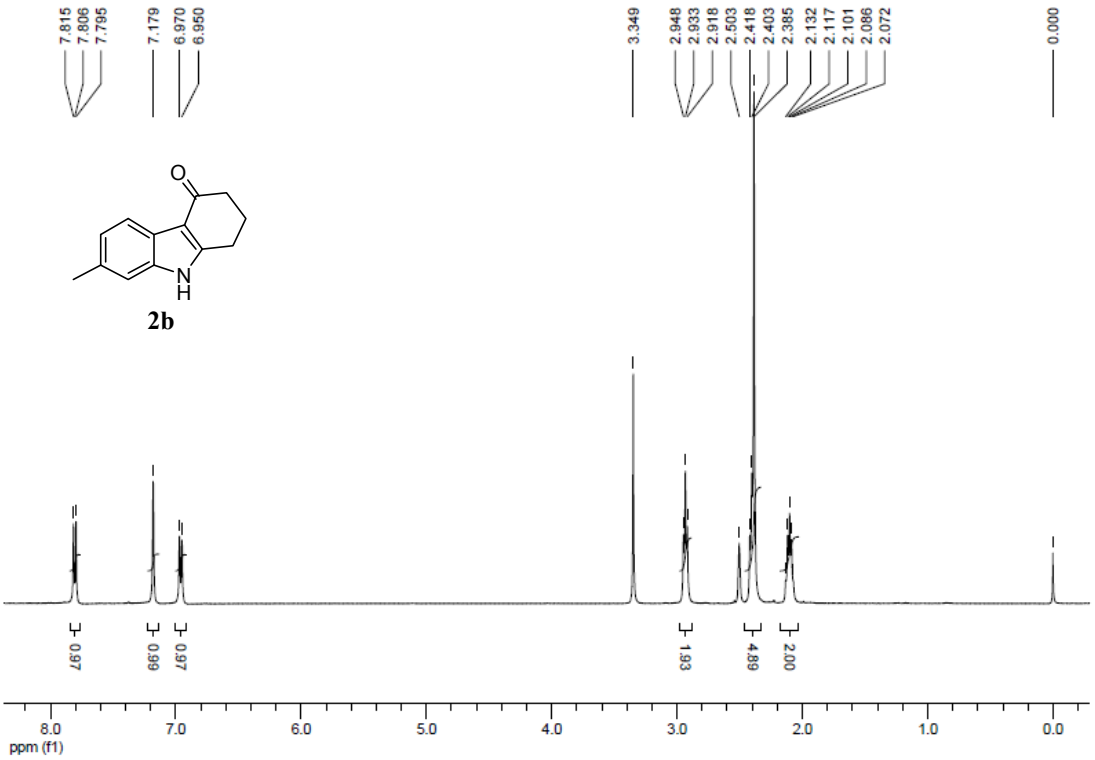

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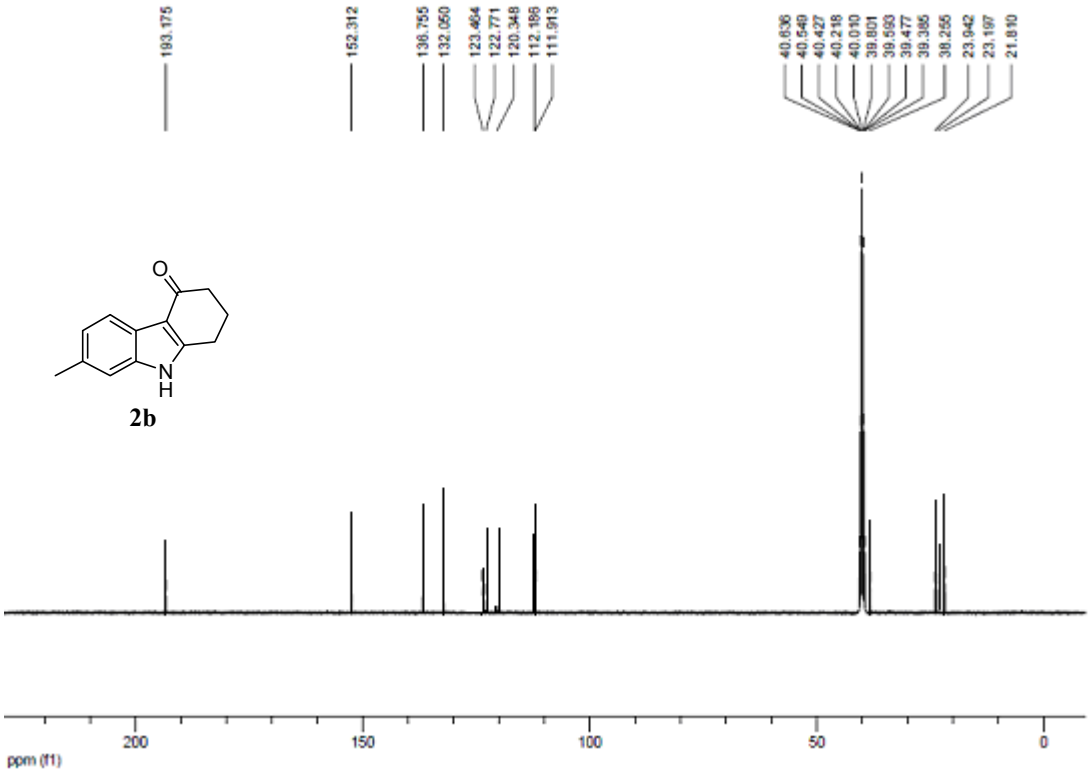

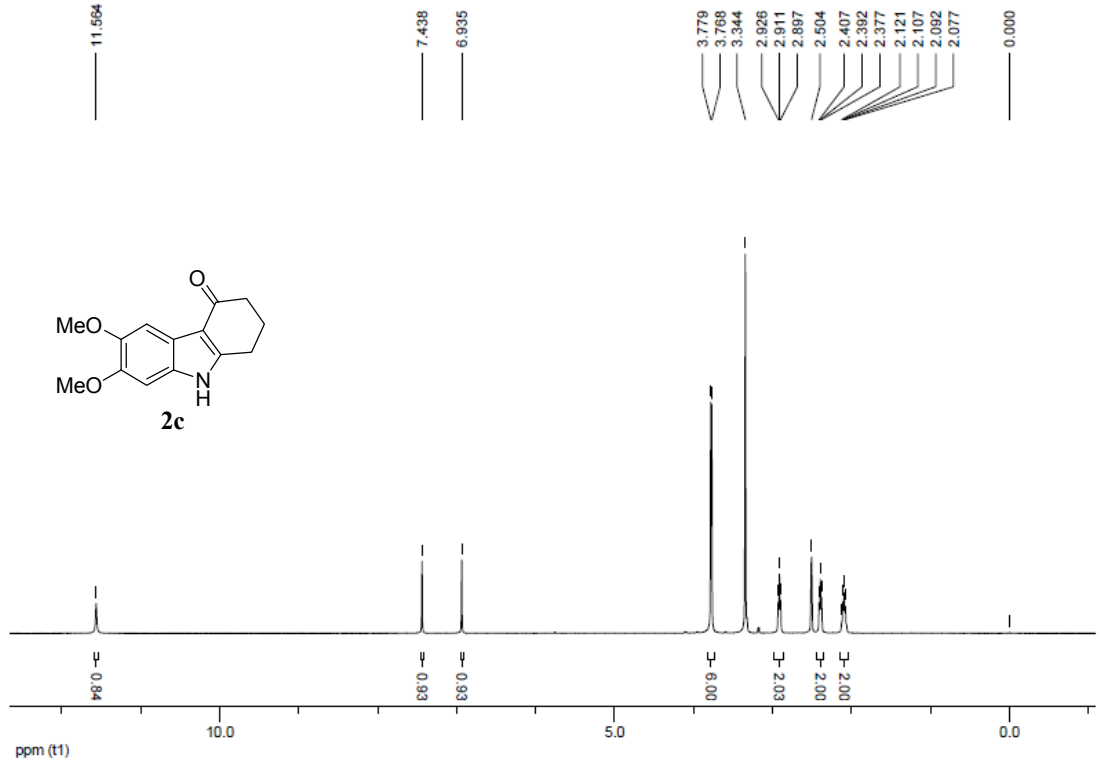

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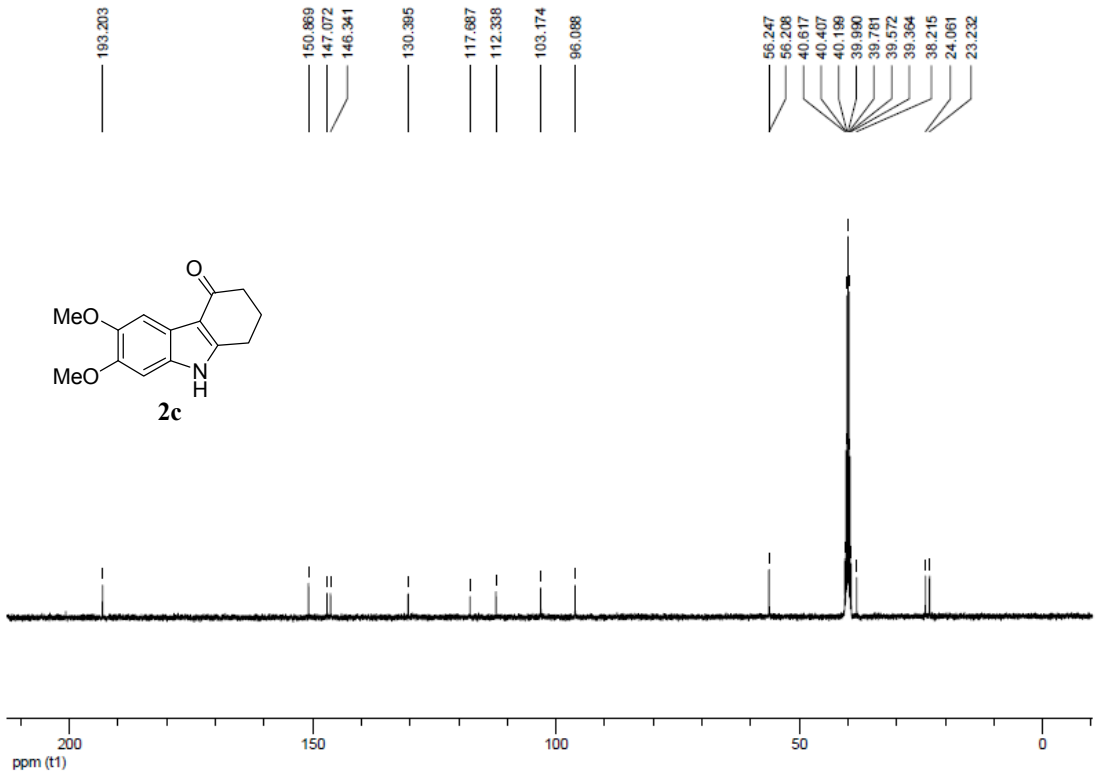

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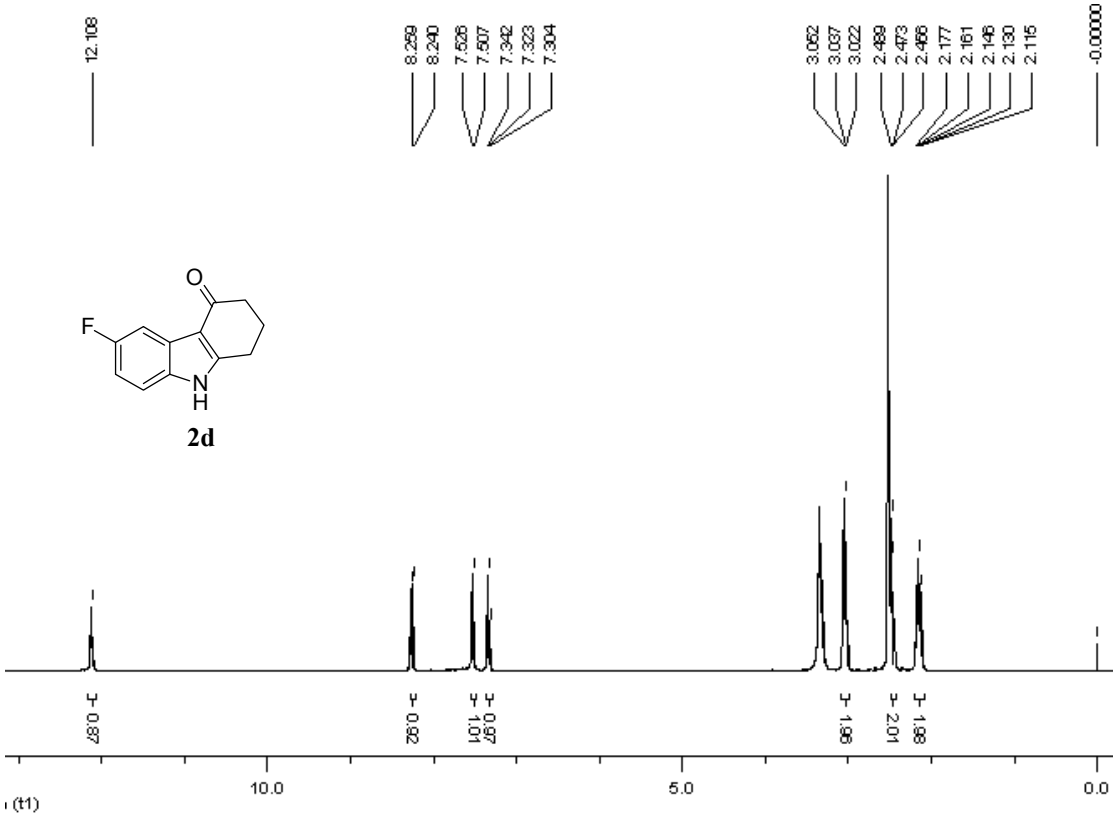

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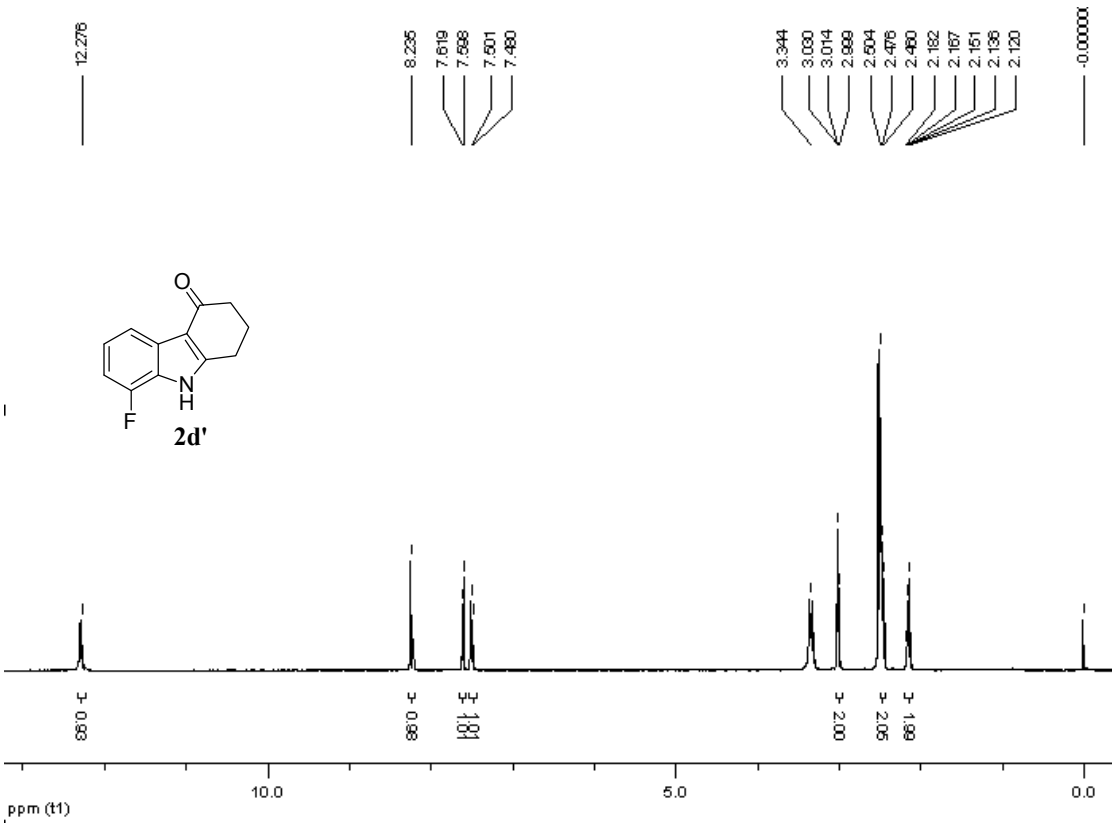

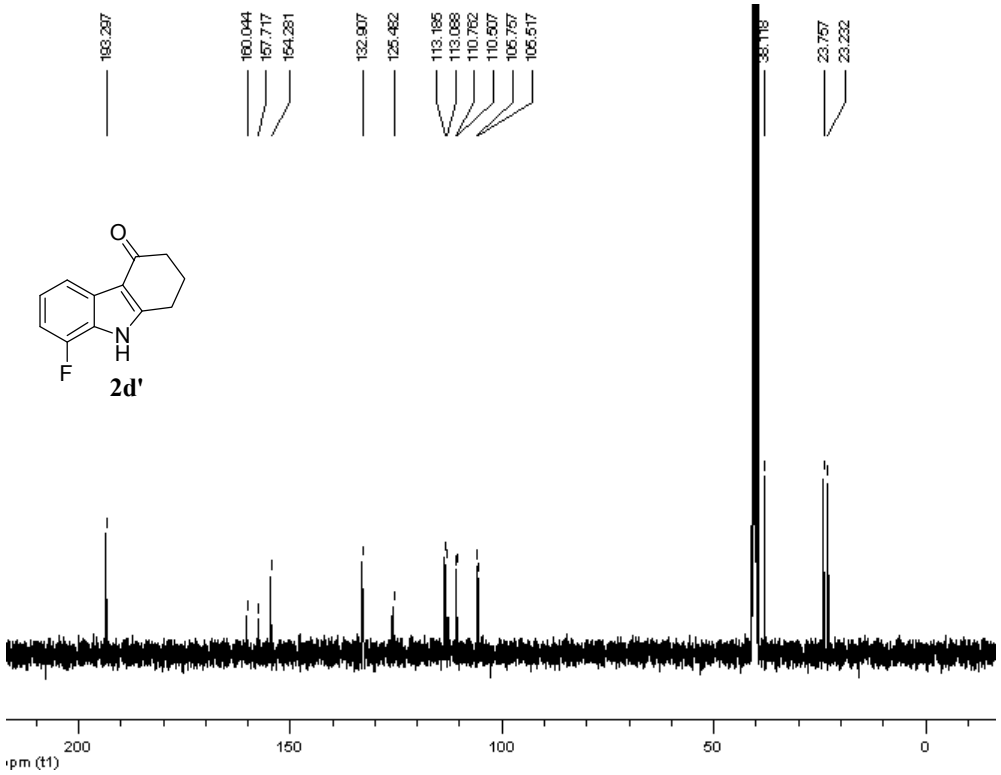

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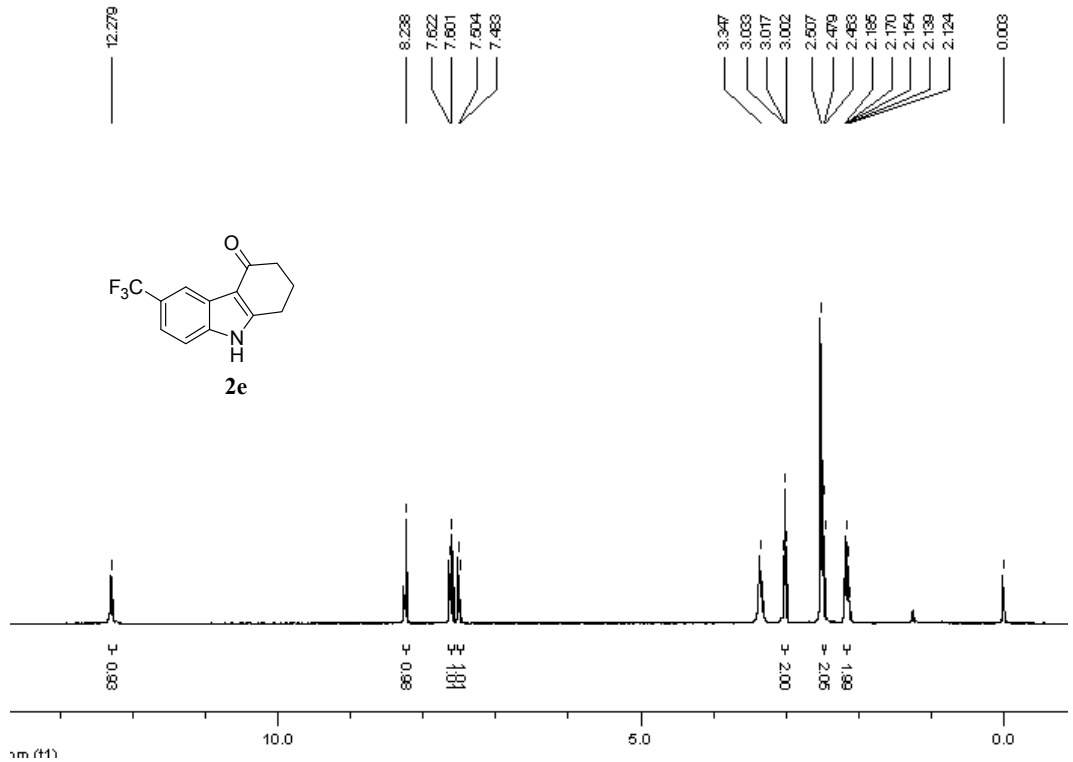

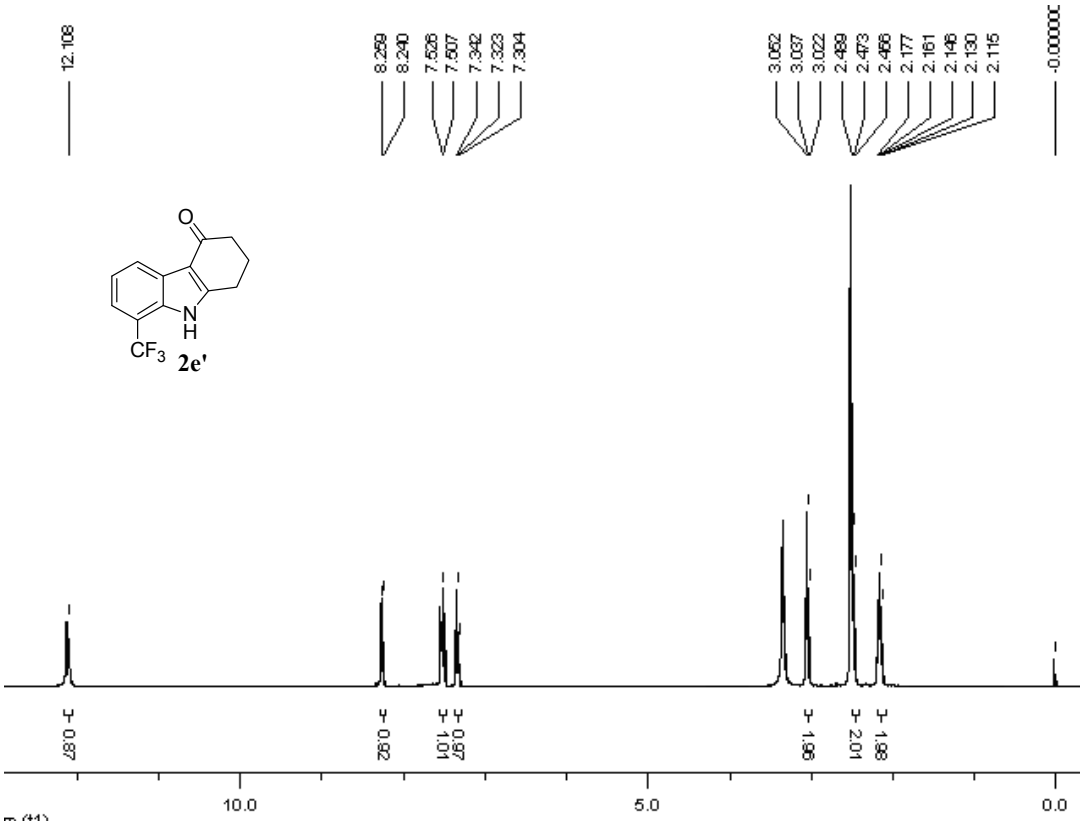

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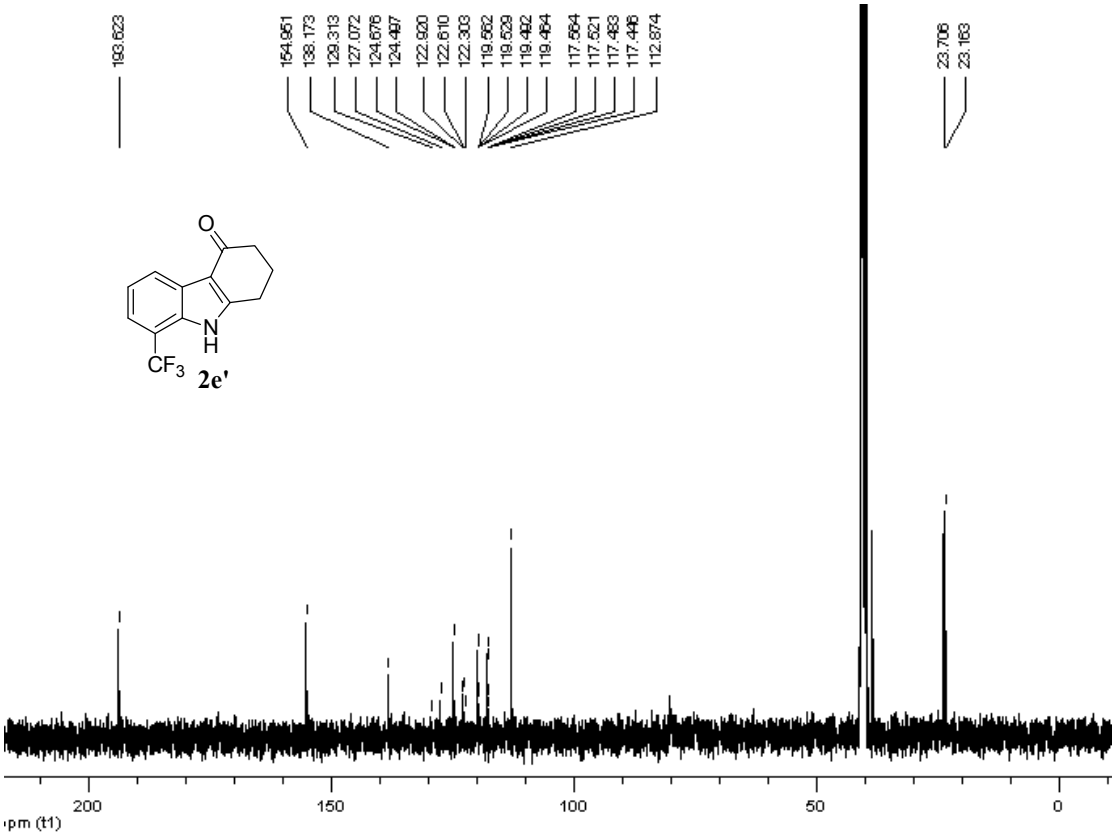

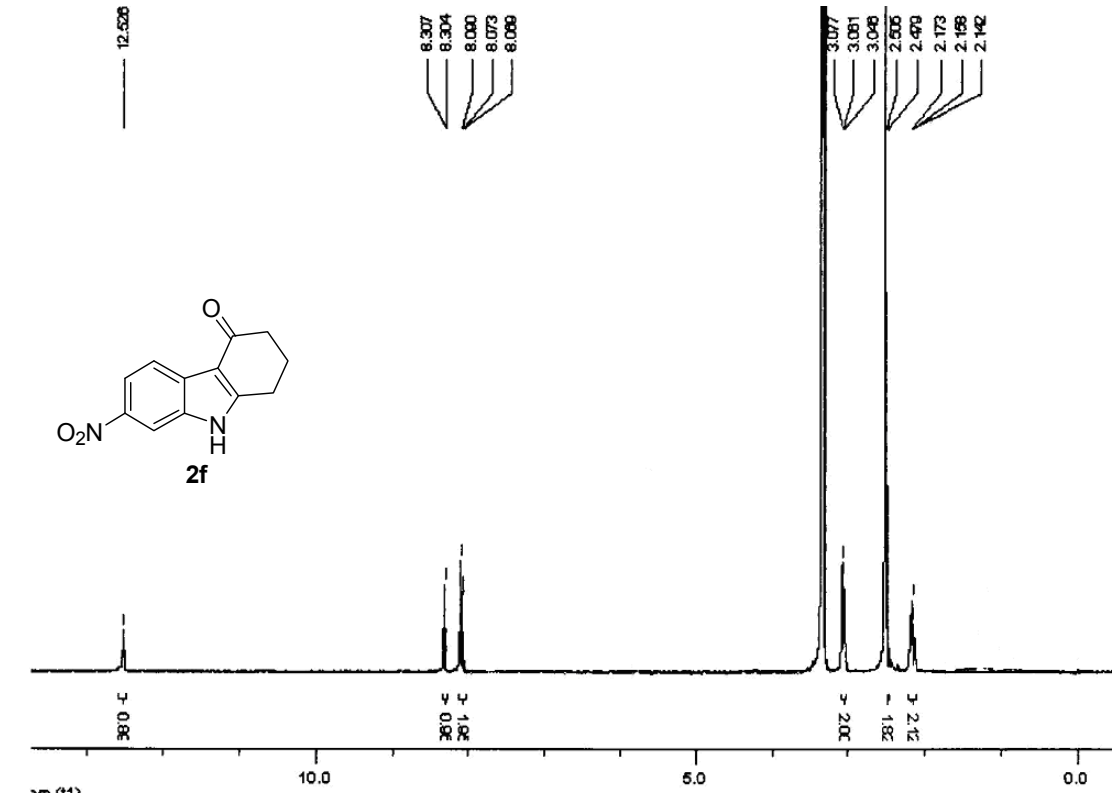

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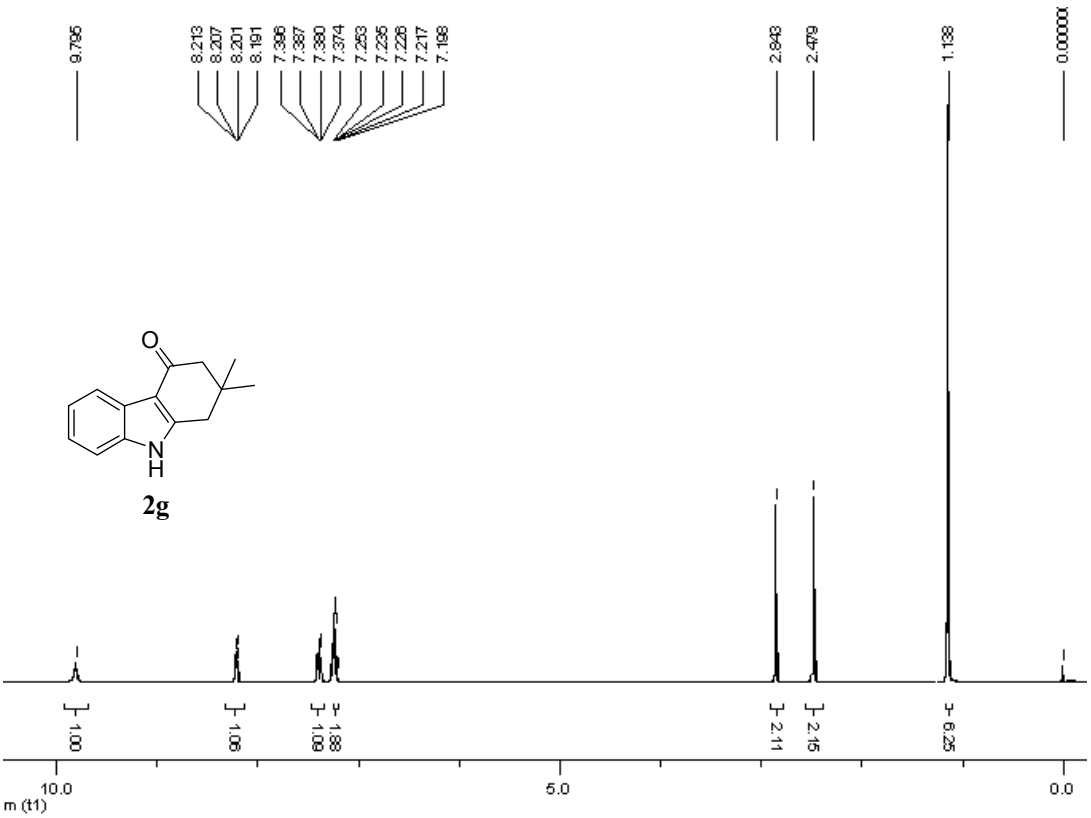

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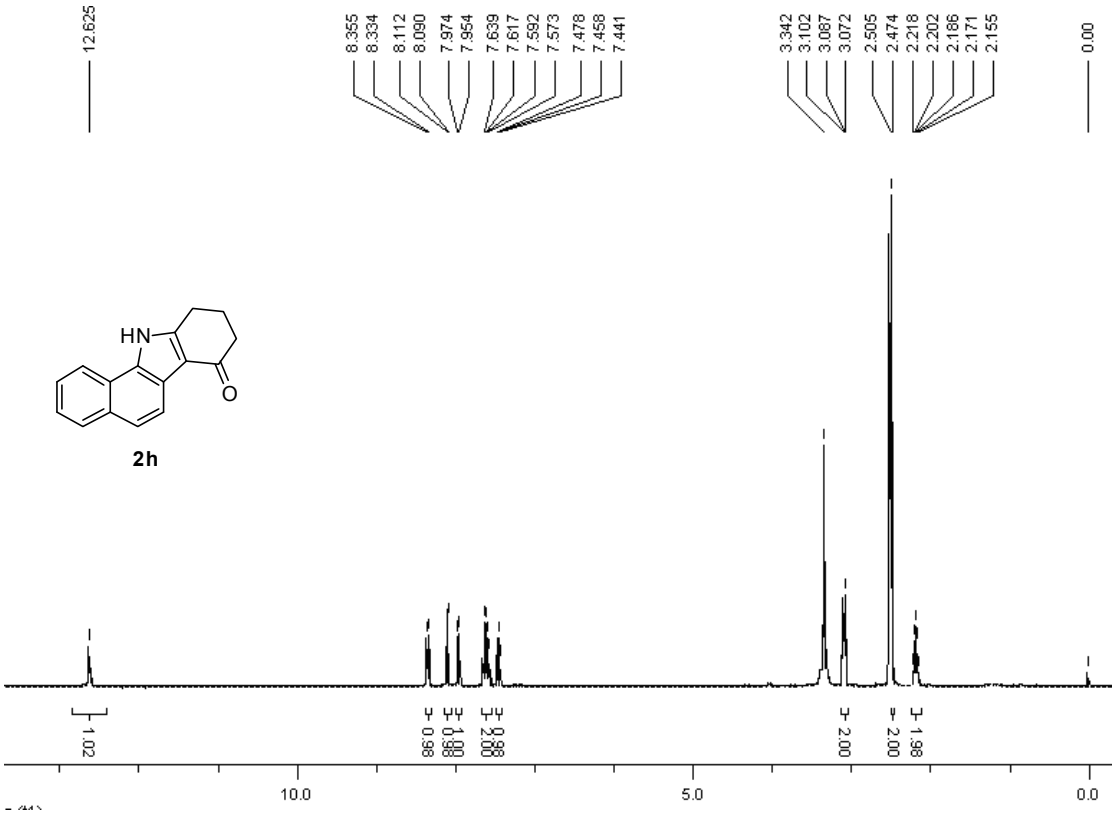

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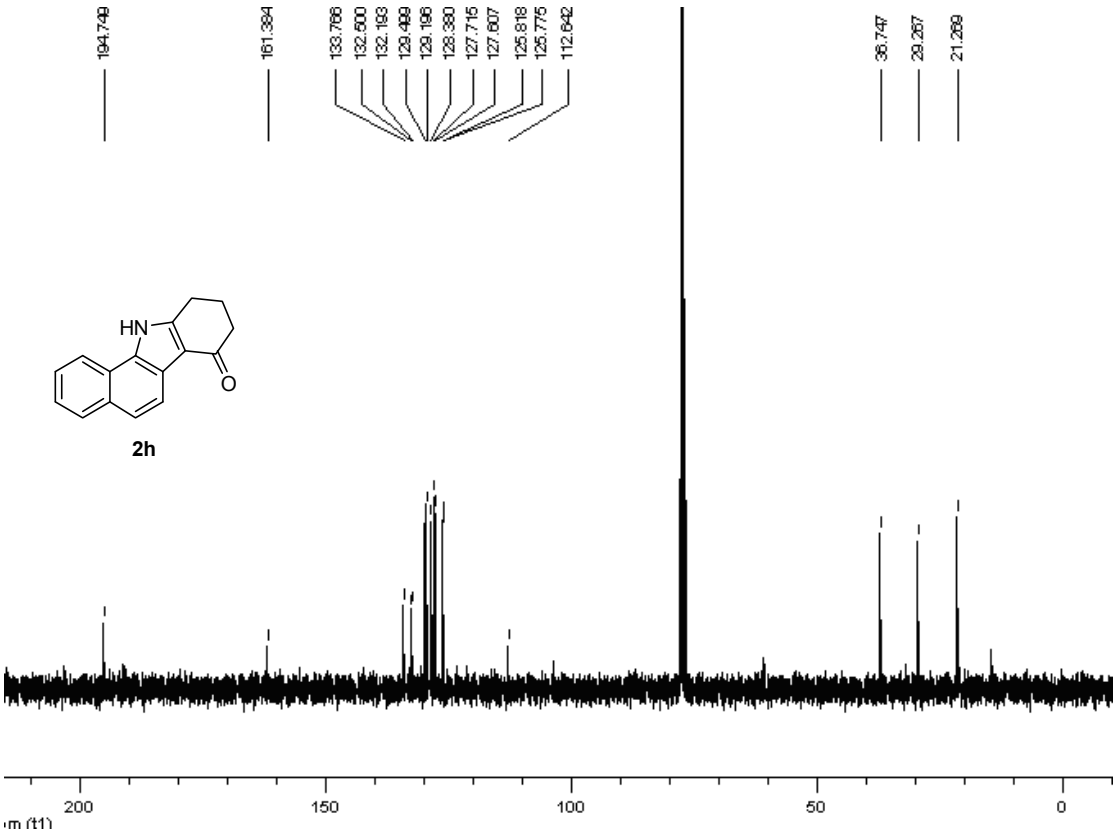

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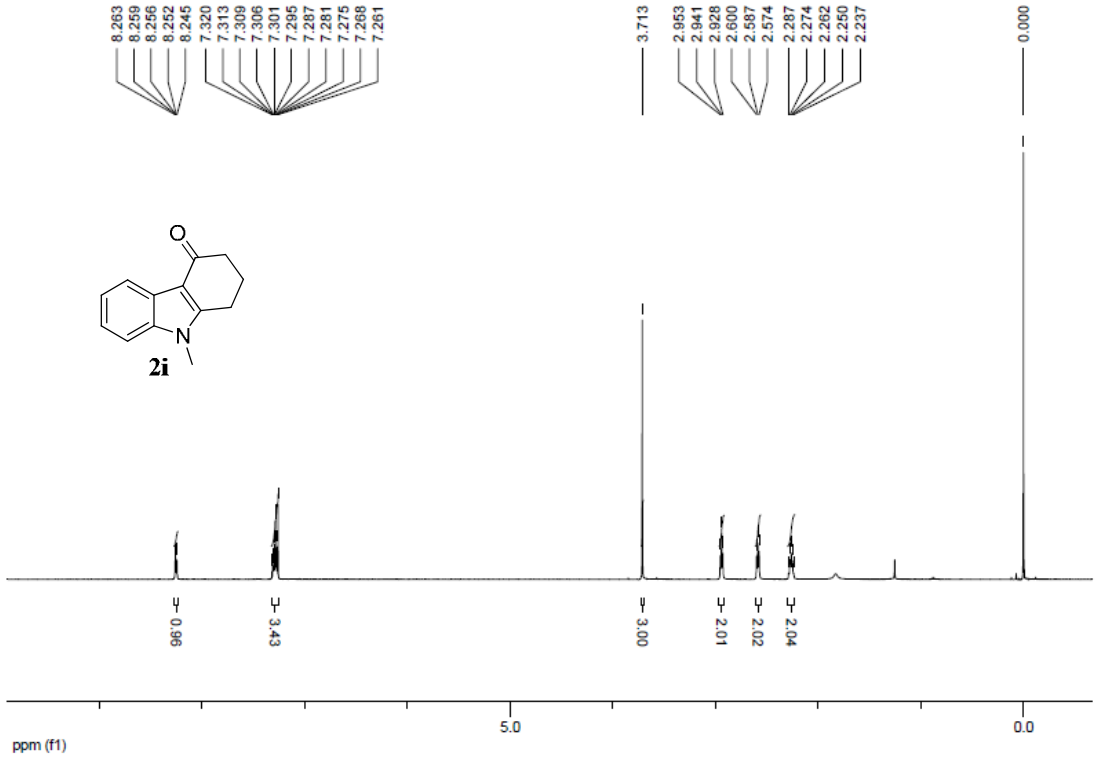

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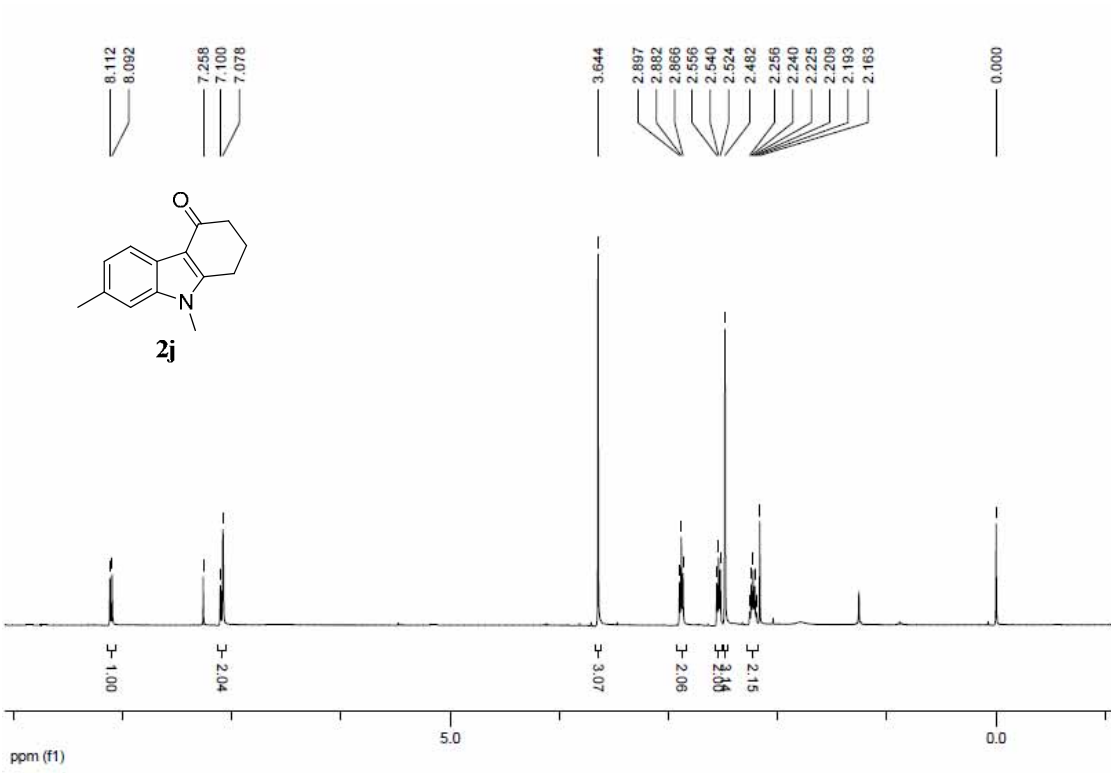

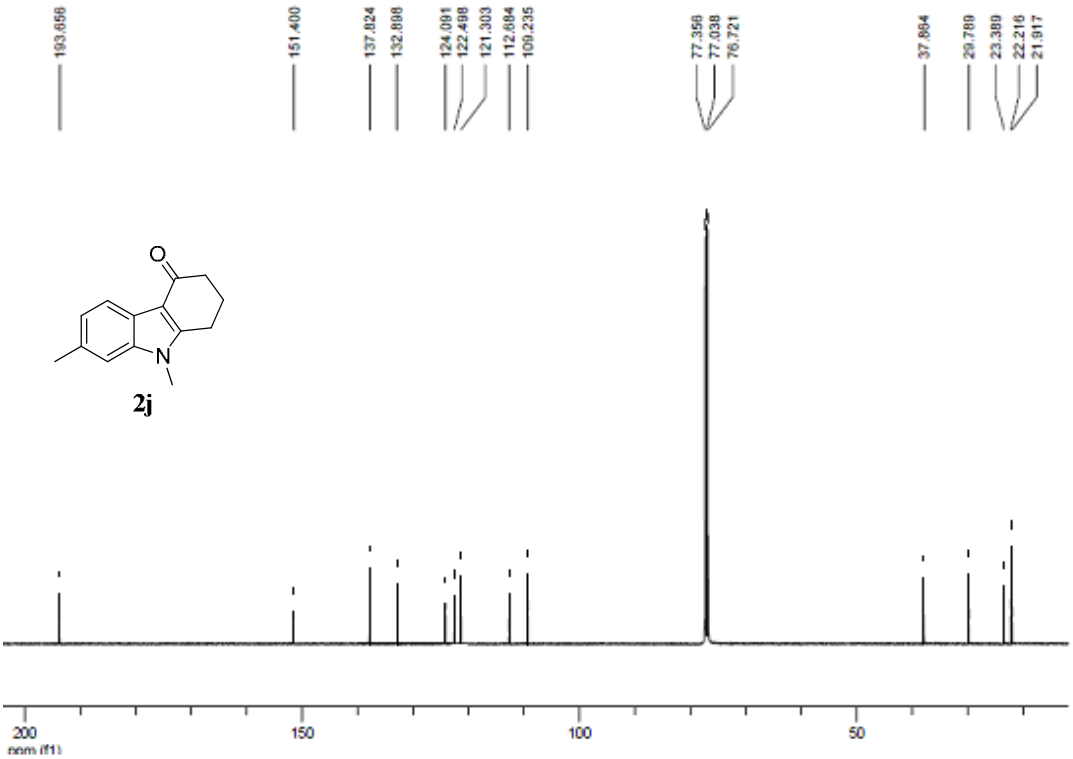

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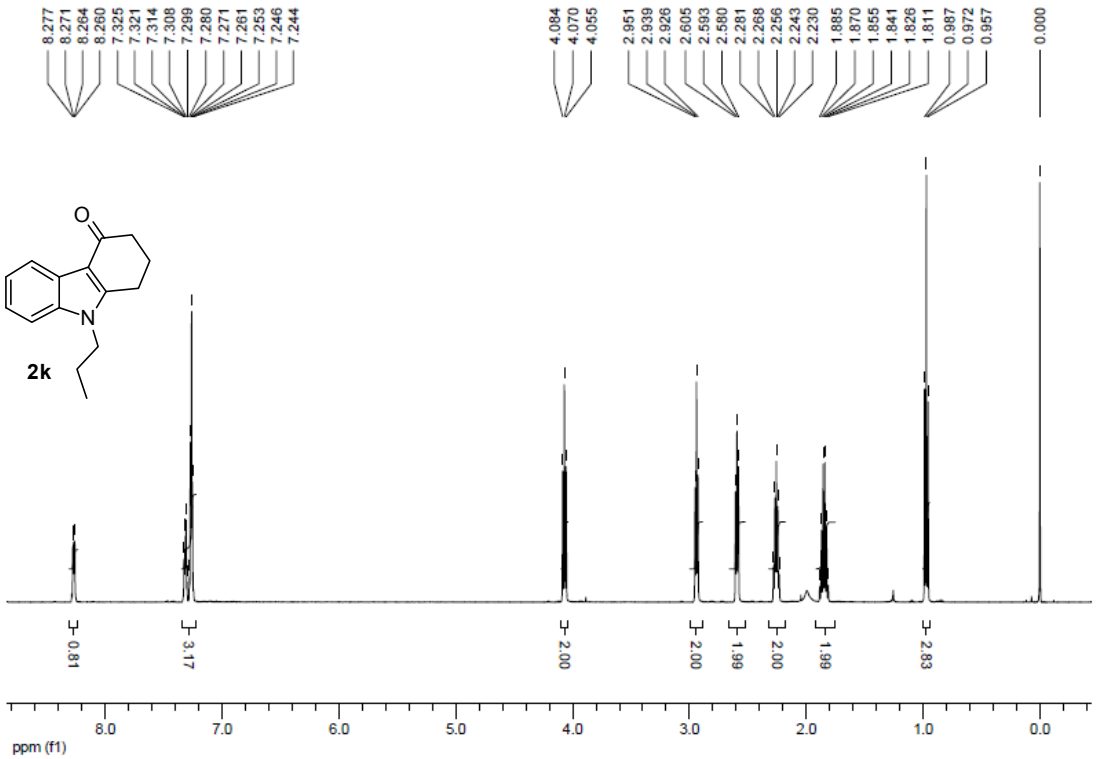

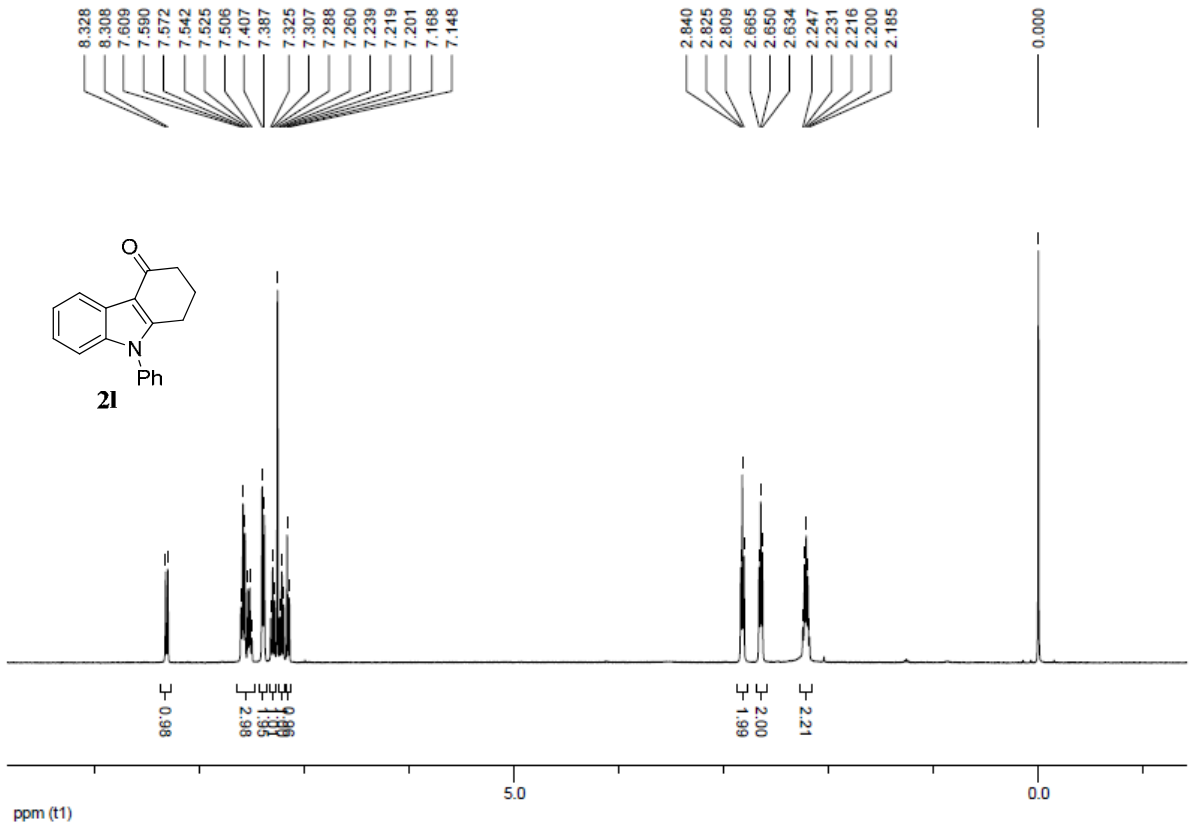

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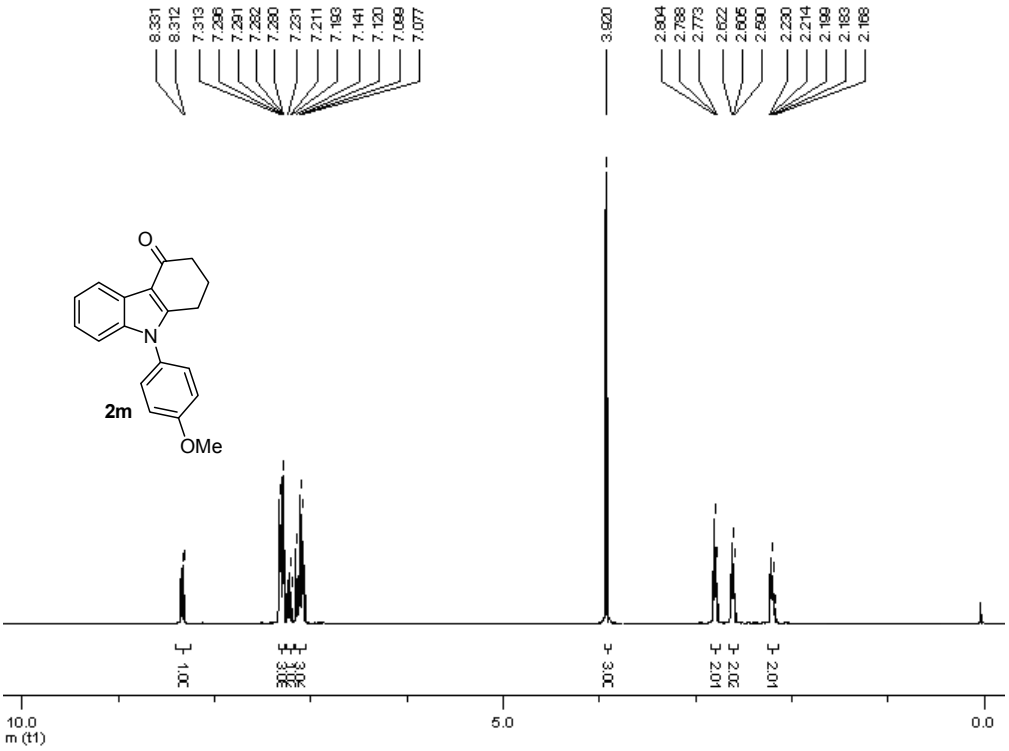

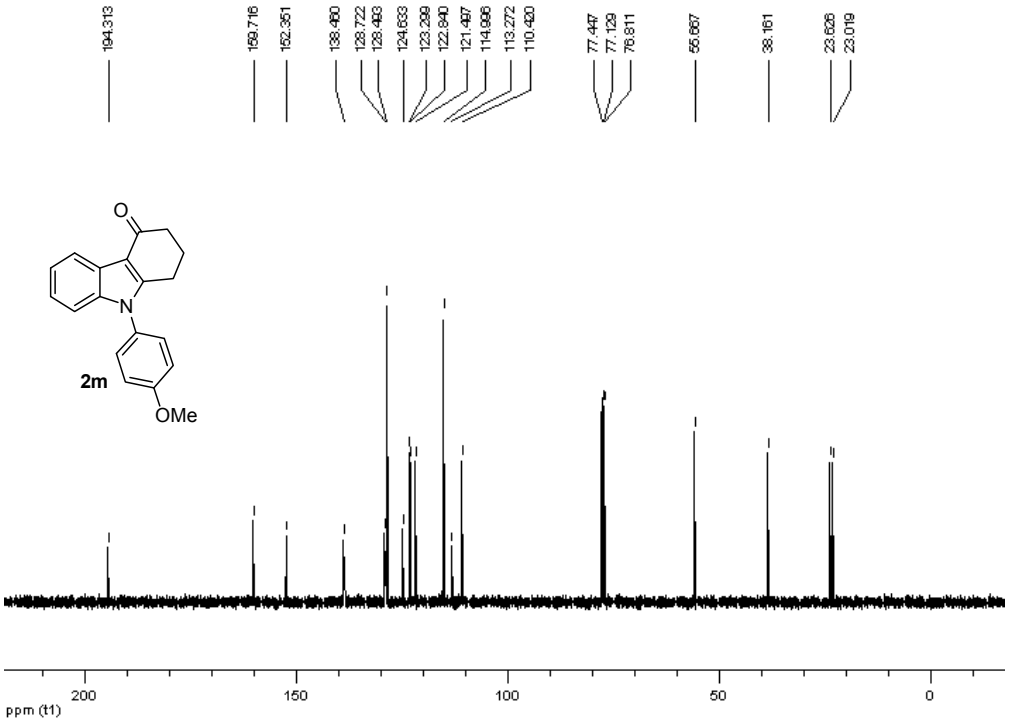

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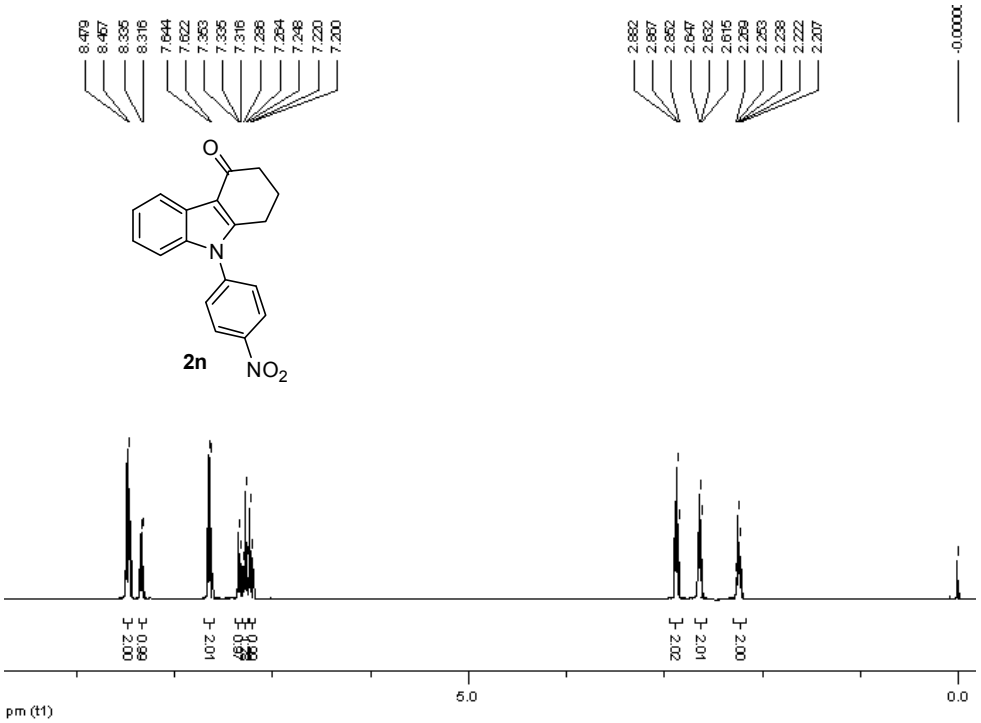

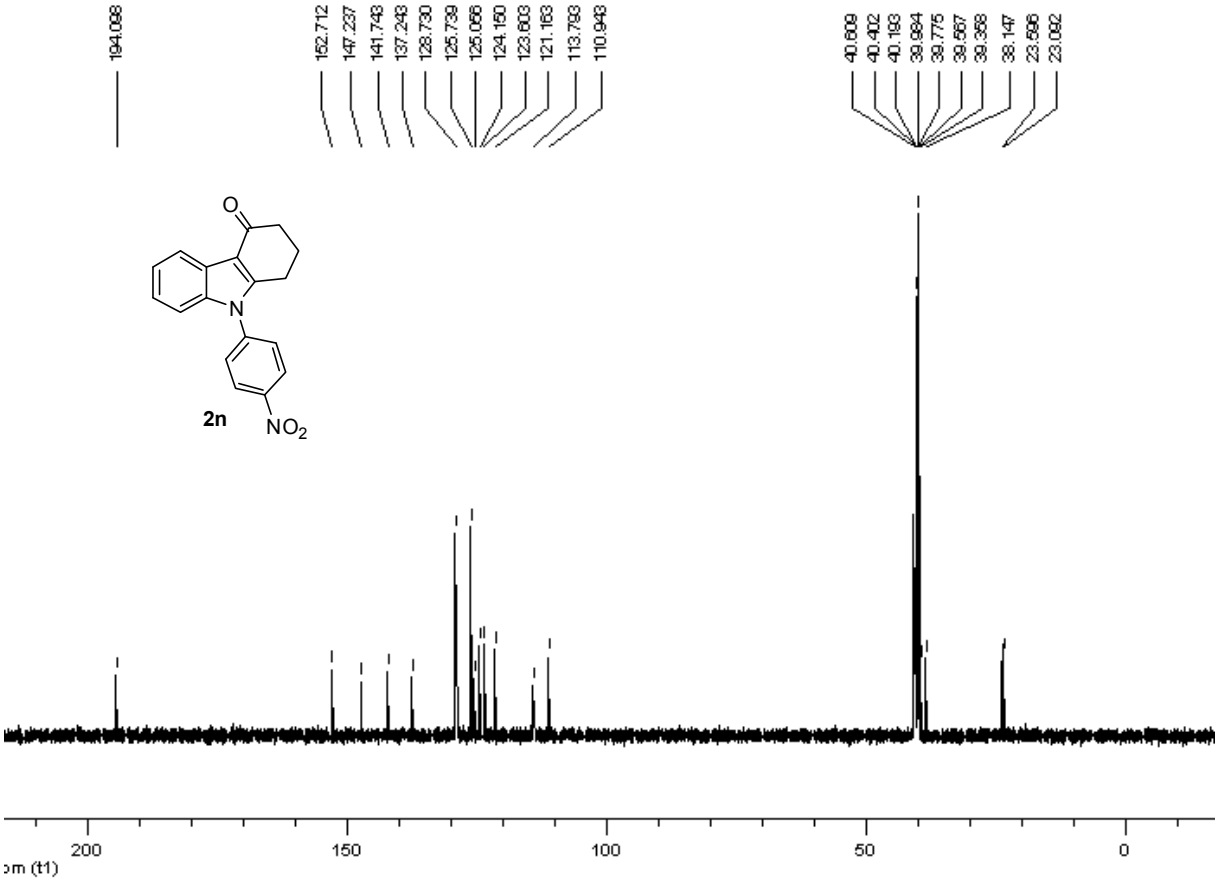

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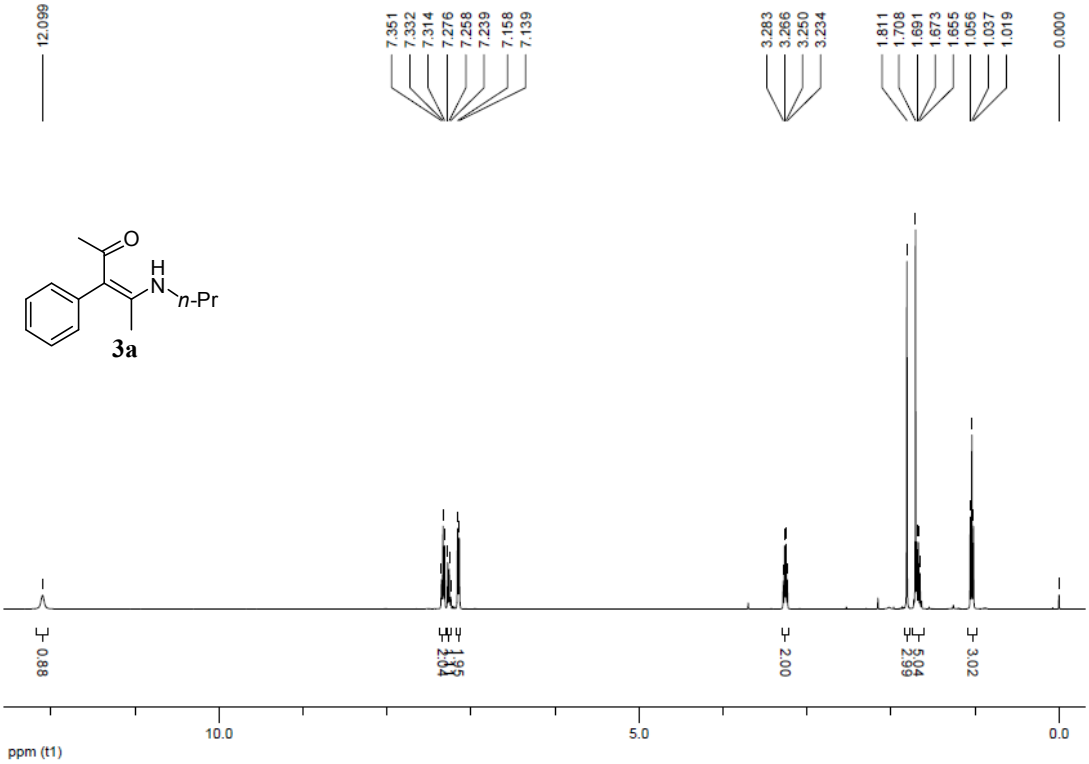
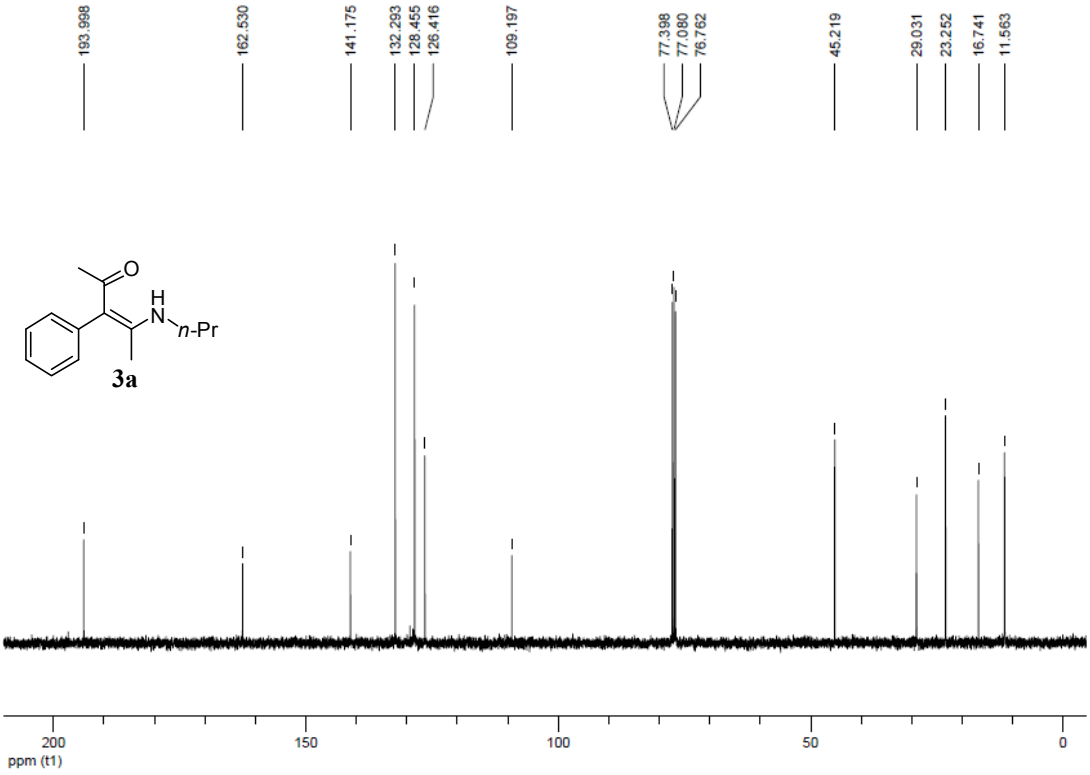
^{1}H and ^{13}C NMR Spectra of 3





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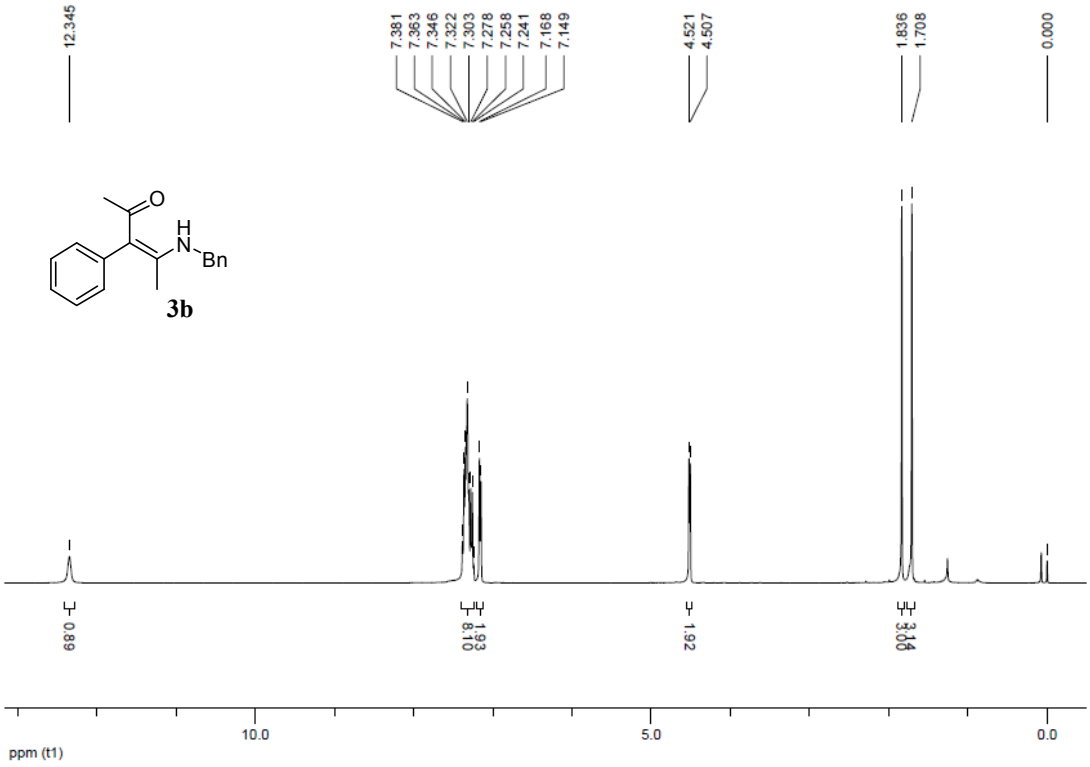

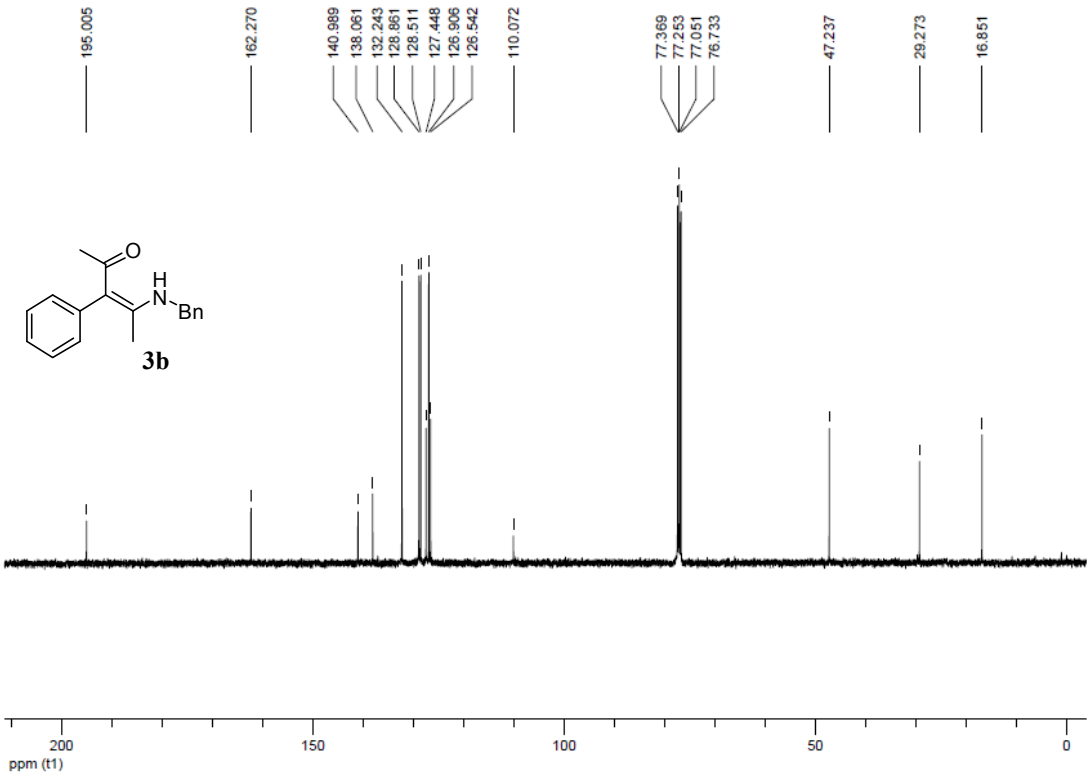

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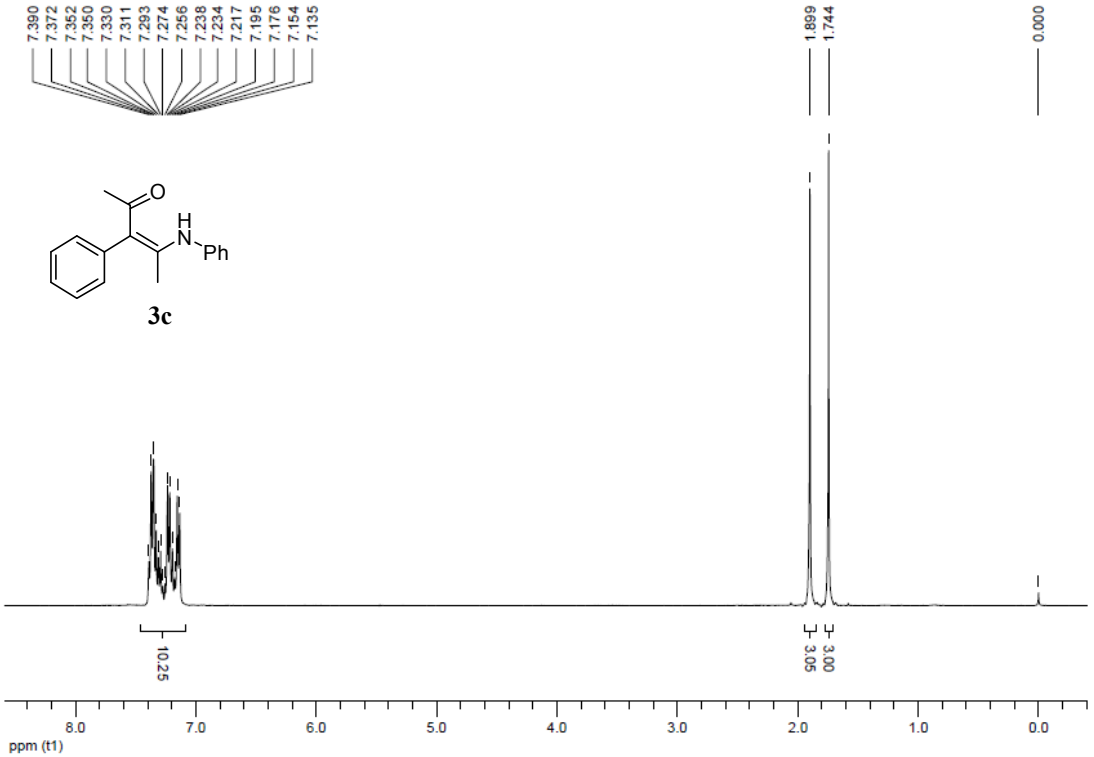
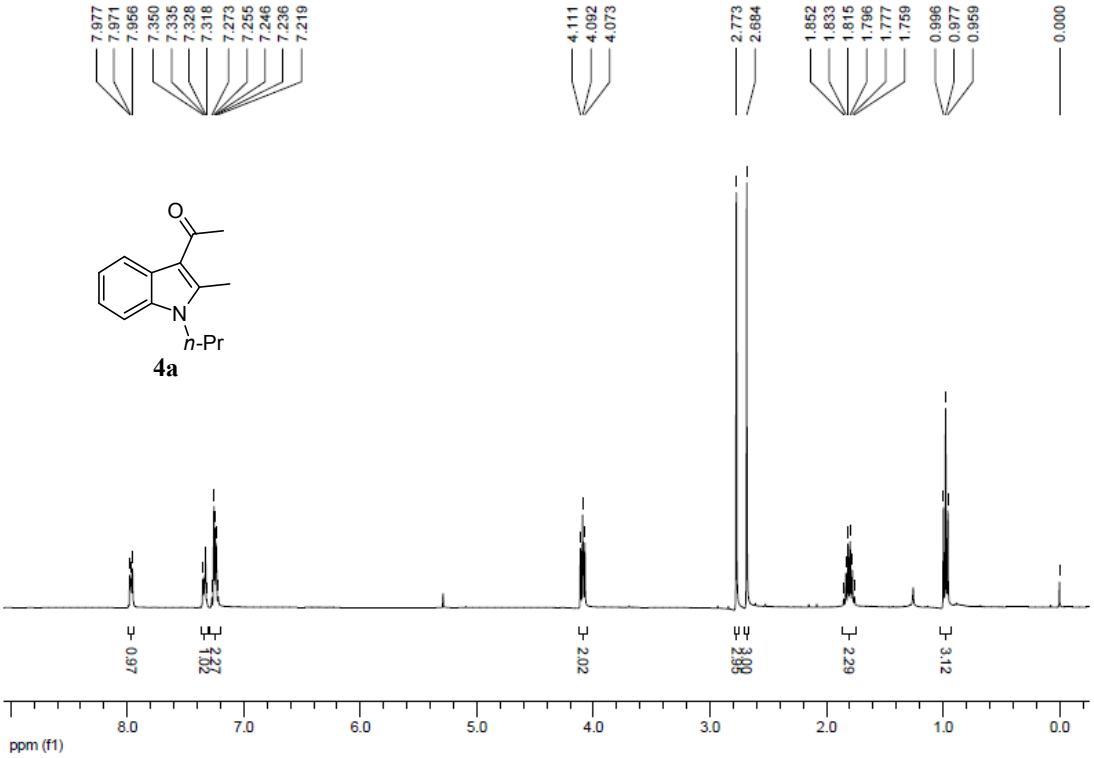


3c

^{1}H and ^{13}C NMR Spectra of 4



4a

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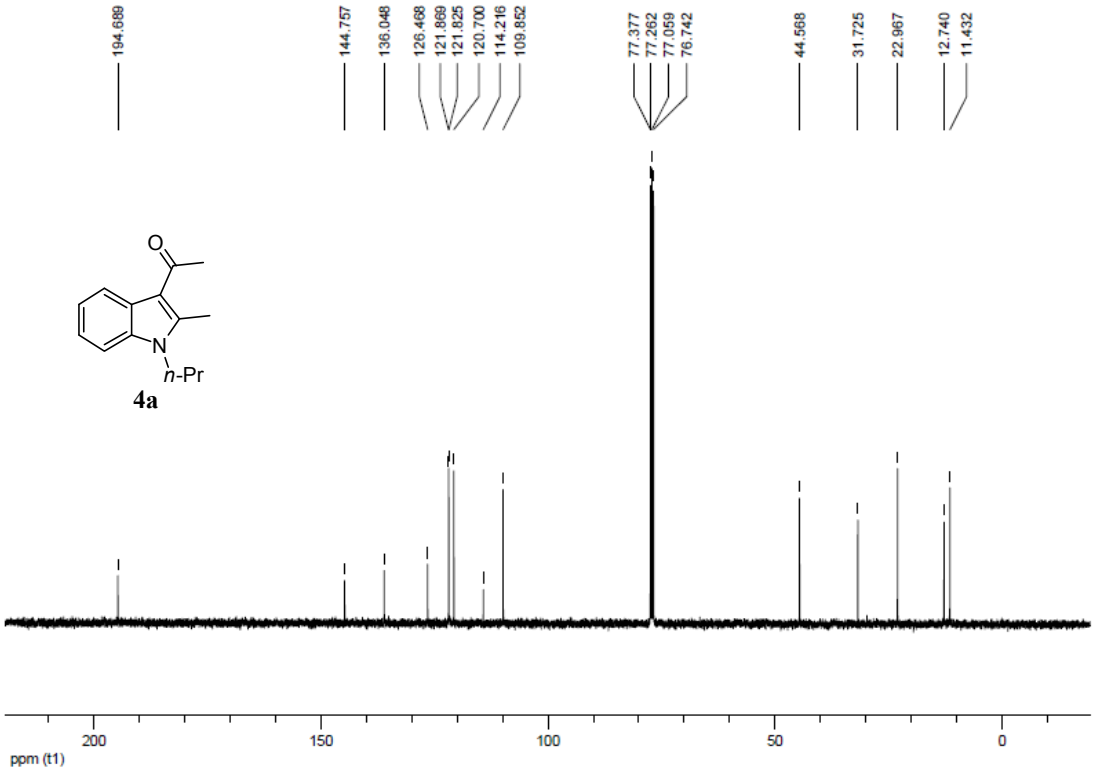

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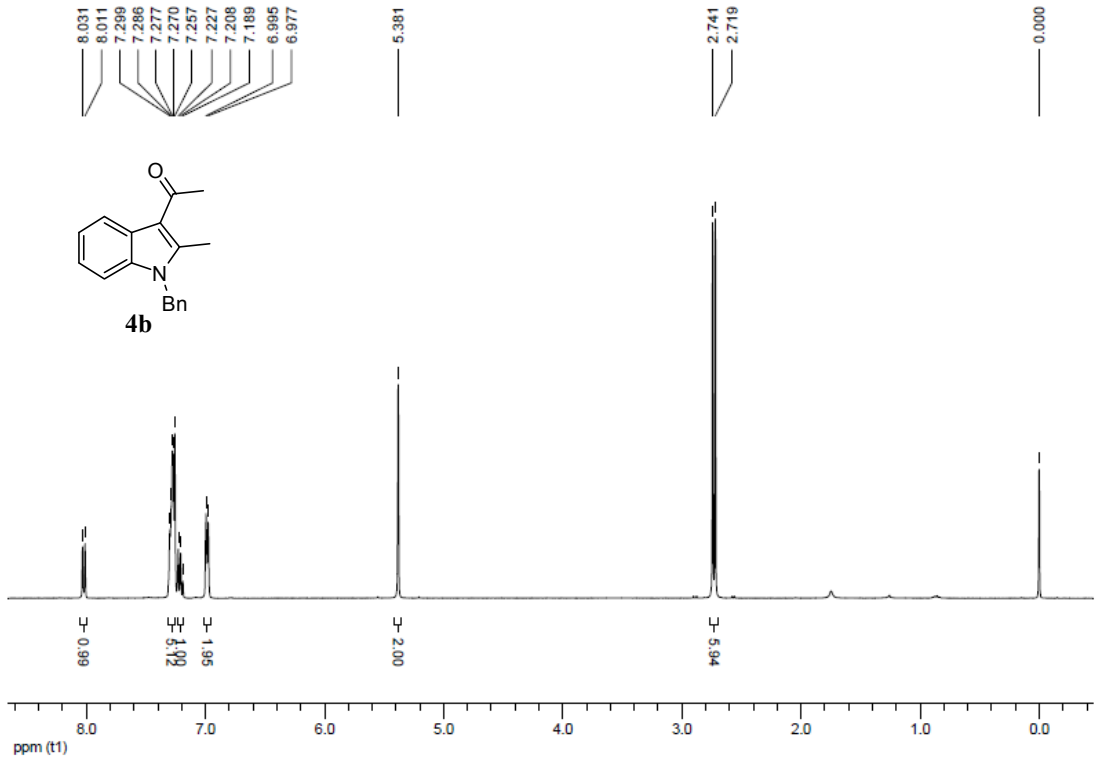

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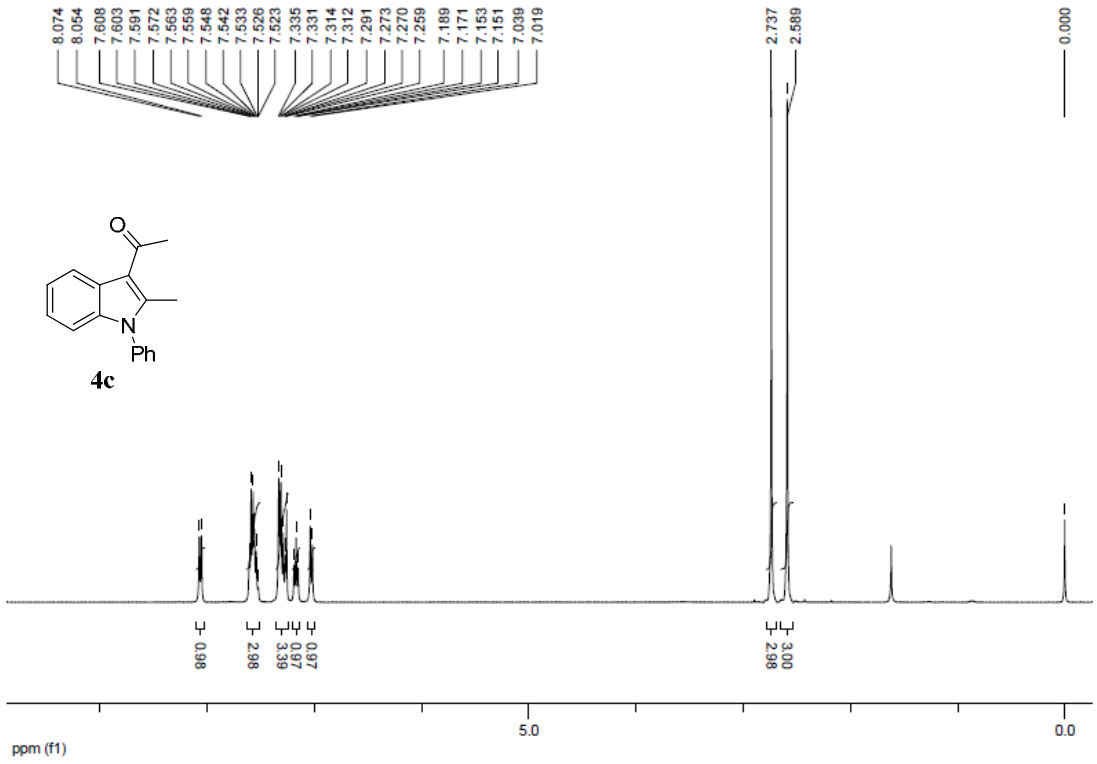

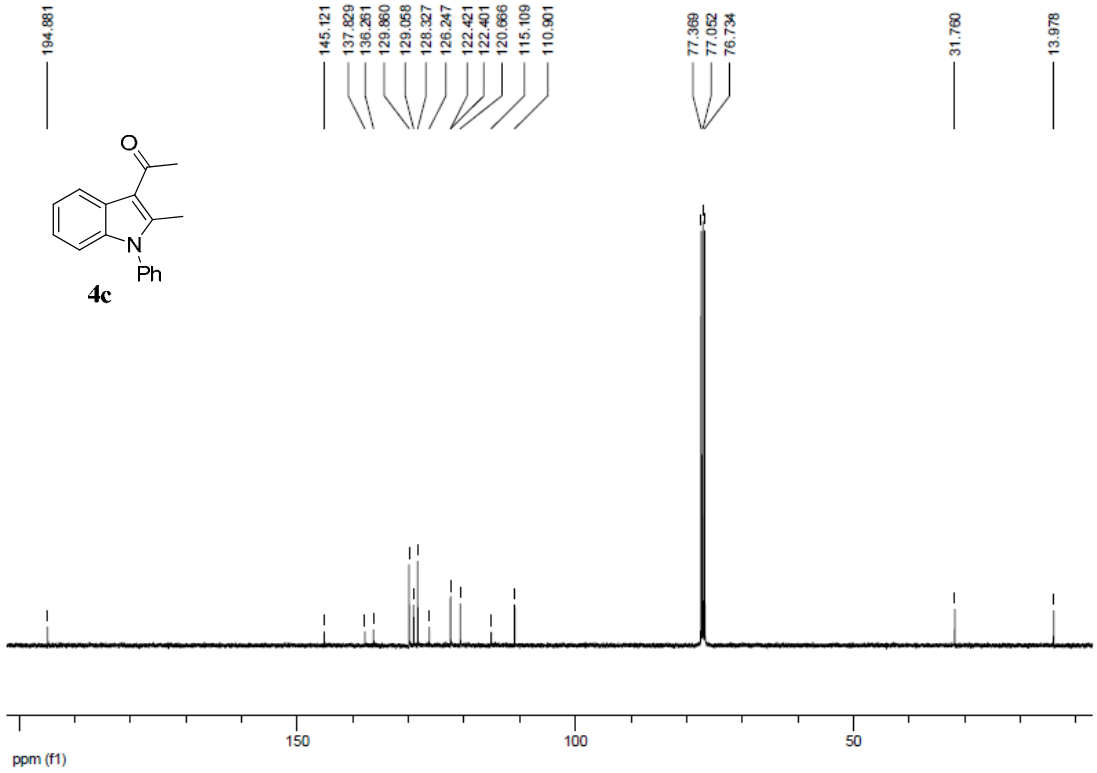

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