Electronic Supplementary Material (ESI) for Chemical Communications

Supporting Information

Copper-Catalyzed Aldol-Type Addition of Ketones to Aromatic Nitriles: A Simple Approach to Achieve Enaminones

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1. General Information

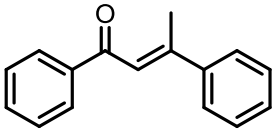
^{1}H NMR and ^{13}C NMR spectra were recorded on either a Varian Inova-400 spectrometer (400 MHz for ^{1}H, 100 MHz for ^{13}C) or a Bruker Avance II-400 spectrometer (400 MHz for ^{1}H, 100 MHz for ^{13}C); $CDCl_3$ and TMS were used as a solvent and an internal standard, respectively. The chemical shifts are reported in ppm downfield (δ) from TMS, and the coupling constants J are given in Hz. The peak patterns are indicated as follows: s, singlet; d, doublet; t, triplet; m, multiplet; q, quartet. IR spectrum was recorded on a NEXUS FT-IR spectrometer. High resolution mass spectrum was recorded on a Q-TOF mass spectrometry. GC analysis was performed on an Agilent Technologies 7820A GC system. The products were separated on a 30 m length by 0.320 mm id, HP-5. Nitrogen was employed as the carrier gas, with a constant column flow of 0.8 mL/min. The injector temperature was held constant at 250 ºC. The GC oven temperature program was as follows: start from 60 ºC, ramp 15 ºC/min to 300 ºC, hold for 5 min. TLC was carried out on SiO_2 (silica gel 60 F_{254}, Merck), and the spots were located with UV light or iodoplatinate reagent. Flash chromatography was carried out on SiO_2 (silica gel 60, 200-300 mesh). Solvents were dried and degassed before use by standard procedures. The starting materials **1a**–**1m** and **2a**–**2g** are commercially available.

2. General Procedure for Aldol-Type Adition Reaction

A solution of copper iodide (CuI, 9.5 mg, 10 mol%) and 2,2'-bipyridine (9.5 mg, 11 mol%) in DMF (1.0 mL) was stirred at room temperature under nitrogen atmosphere for 10 min. And then, aromatic nitrile (**2**, 0.5 mmol), ketone (**1**, 0.6 mmol), and sodium *tert*-butoxide (NaO^tBu, 96.0 mg, 2.0 mmol) were successively added. After the resultant mixture was stirred at 80 °C for 12 h, saturated aqueous ammonium chloride solution (20 mL) was added. The product was extracted with ethyl acetate (25 mL × 3), and the combined organic layers were washed with brine (10 mL × 2), dried over Na_2SO_4. The solvent was removed under reduced pressure, and the residue obtained was purified via silica gel chromatography (eluent: petroleum ether/ethyl acetate = 5:1) to afford enaminones **3**.

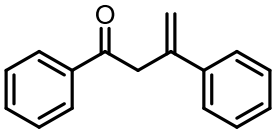
3. Characterization Data

1,3-Diphenylbut-2-en-1-one (4a)[1]



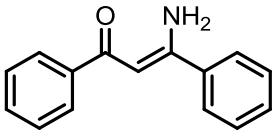
Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.60 (s, 3H), 7.17 (s, 1H), 7.40–7.43 (m, 3H), 7.47 (dd, J = 8.0, 7.2 Hz, 2H), 7.53–7.58 (m, 3H), 7.99 (d, J = 7.2 Hz, 2H).

1,3-Diphenylbut-3-en-1-one (4a′)



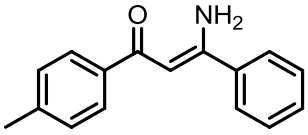
Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.17(s, 2H), 5.18 (s, 1H), 5.60 (s, 1H), 7.25–7.34 (m, 3H), 7.41–7.47 (m, 4H), 7.56 (dd, J = 7.6 , 7.2 Hz, 1H), 7.98 (d, J = 7.2 Hz, 2H).

3-Amino-1,3-diphenylprop-2-en-1-one (**3a**)[2]



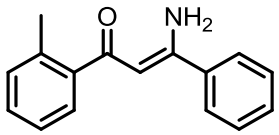
Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.63 (bs, 1H), 6.13 (s, 1H), 7.39–7.47 (m, 6H), 7.61 (d, J = 6.4 Hz, 2H), 7.93 (d, J = 6.4 Hz, 2H), 10.40 (bs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 91.8, 126.4, 127.2, 128.3, 129.0, 130.7, 131.1, 137.6, 140.4, 163.1, 190.1.

3-Amino-3-phenyl-1-p-tolylprop-2-en-1-one (**3b**)



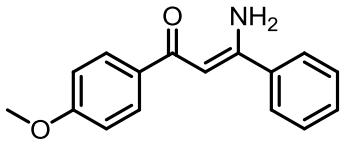
Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.38 (s, 3H), 5.52 (bs, 1H), 6.12 (s, 1H), 7.21–7.23 (m, 2H), 7.42–7.45 (m, 3H), 7.61–7.62 (m, 2H), 7.84–7.86 (m, 2H), 10.37 (bs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.6, 91.8, 126.5, 127.4, 129.1, 130.7, 137.8, 141.5, 162.8, 190.0; IR (neat): 3460, 3353, 2920, 1603, 1485, 1448, 839, 760, 719, 696 cm^{-1}; HRMS (ES) Calcd for $C_{16}H_{15}NO$: 260.1051 $[M+Na]^+$; Found: 260.1053.

3-Amino-3-phenyl-1-o-tolylprop-2-en-1-one (**3c**)



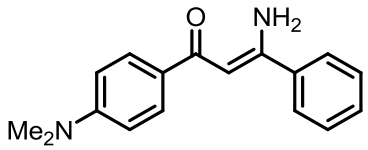
Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.55 (s, 3H), 5.71 (bs, 1H), 5.81 (s, 1H), 7.22–7.33 (m, 3H), 7.43–7.52 (m, 4H), 7.62 (d, J = 7.1 Hz, 2H), 10.33 (bs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.5, 95.8, 125.7, 126.6, 127.6, 129.2, 129.4, 130.9, 131.2, 135.9, 137.3, 142.5, 162.6, 195.2; IR (neat): 3330, 3174, 2924, 2855, 1605, 1523, 846, 758, 748, 699, 652 cm^{-1}; HRMS (ES) Calcd for $C_{16}H_{15}NO$: 260.1051 $[M+Na]^+$; Found: 260.1049.

3-Amino-1-(4-methoxyphenyl)-3-phenylprop-2-en-1-one (**3d**)



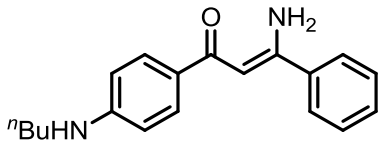
Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.81 (s, 3H), 5.68 (bs, 1H), 6.10 (s, 1H), 6.91 (d, J = 8.4 Hz, 2H), 7.42–7.46 (m, 3H), 7.61 (d, J = 6.8 Hz, 2H), 7.93 (d, J = 8.8 Hz, 2H), 10.33 (bs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.4, 91.5, 113.5, 126.4, 129.0, 129.2, 130.6, 133.1, 137.8, 162.1, 162.5, 189.2; IR (neat): 3460, 3358, 2933, 2837, 1597, 1485, 1413, 844, 768, 726, 696 cm^{-1}; HRMS (ES) Calcd for $C_{16}H_{15}NO_2$: 276.1000 $[M+Na]^+$; Found: 276.0990.

3-Amino-1-(4-(dimethylamino)phenyl)-3-phenylprop-2-en-1-one (**3e**)



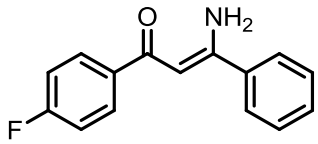
Light yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.00 (s, 3H), 3.01 (s, 3H), 5.32 (bs, 1H), 6.12 (s, 1H), 6.67 (d, J = 8.0 Hz, 2H), 7.43–7.44 (m, 3H), 7.61–7.63 (m, 2H), 7.80 (d, J = 8.4 Hz, 2H), 10.25 (bs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 40.3, 91.6, 111.1, 126.5, 128.1, 129.1, 129.2, 130.4, 138.3, 152.5, 161.5, 189.4; IR (neat): 3455, 3353, 2921, 2809, 1592, 1485, 1443, 1366, 830, 769, 727, 697 cm^{-1}; HRMS (ES) Calcd for $C_{17}H_{18}N_2O$: 289.1317 $[M+Na]^+$; Found: 289.1319.

3-Amino-1-(4-(butylamino)phenyl)-3-(4-chlorophenyl)prop-2-en-1-one (**3f**)



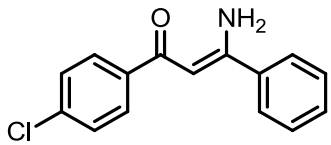
Light yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.95 (t, J = 7.2 Hz, 3H), 1.39–1.46 (m, 2H), 1.53–1.63 (m, 2H), 3.15 (t, J = 6.4 Hz, 2H), 4.06 (bs, 1H), 5.29 (bs, 1H), 6.09 (s, 1H), 6.55–6.57 (m, 2H), 7.44–7.62 (m, 5H), 7.83–7.85 (m, 2H), 10.24 (bs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.1, 20.4, 31.7, 43.4, 91.6, 111.7, 126.5, 129.1, 129.5, 130.5, 138.4, 151.3, 161.5, 189.4; IR(neat): 3404, 3345, 2929, 2869, 1593, 1485, 1329, 835, 791, 769, 725, 696 cm^{-1}; HRMS (ES) Calcd for $C_{19}H_{22}N_2O$: 317.1630 $[M+Na]^+$; Found: 317.1642.

3-Amino-1-(4-fluorophenyl)-3-phenylprop-2-en-1-one (**3g**)



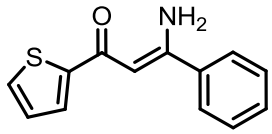
Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.69 (bs, 1H), 6.07 (s, 1H), 7.08 (dd, J = 8.8, 8.4 Hz, 2H), 7.44–7.49 (m, 3H), 7.61 (d, J = 6.8 Hz, 2H), 7.94 (dd, J = 8.0, 6.0 Hz, 2H), 10.37 (bs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 91.5, 115.3 ($^{2}J_{C\text{-}F}$ = 21 Hz), 126.5, 129.2, 129.6 ($^{3}J_{C\text{-}F}$ = 9 Hz), 130.9, 136.7, 137.6, 163.3, 164.7 ($^{1}J_{C\text{-}F}$ = 250 Hz), 188.7; IR (neat): 3465, 3360, 1599, 1485, 850, 766, 723, 697 cm^{-1}; HRMS (ES) Calcd for $C_{15}H_{12}NO$: 264.0801 $[M+Na]^+$; Found: 264.0800.

3-Amino-1-(4-chlorophenyl)-3-phenylprop-2-en-1-one (**3h**)



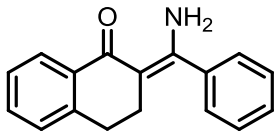
Brown oil.^{1}H NMR (400 MHz, $CDCl_3$) δ 5.73 (bs, 1H), 6.07 (s, 1H), 7.36 (d, J = 8.0 Hz, 2H), 7.42–7.47 (m, 3H), 7.62 (d, J = 8.0 Hz, 2H), 7.86 (d, J = 8.0 Hz, 2H), 10.41 (bs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 91.5, 126.4, 128.6, 128.7, 129.1, 130.9, 137.2, 137.4, 138.7, 163.5, 188.6; IR (neat): 3464, 3357, 1591, 1480, 846, 762, 719, 696 cm^{-1}; HRMS (ES) Calcd for $C_{15}H_{12}ClNO$: 280.0505 $[M+Na]^+$; Found: 280.0501.

3-Amino-3-phenyl-1-(thiophen-2-yl)prop-2-en-1-one (**3i**)



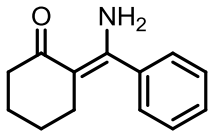
Brown oil. ^{1}H NMR(400 MHz, $CDCl_3$) δ 5.43 (bs, 1H), 6.01 (s, 1H), 7.08–7.10 (m, 1H), 7.45–7.50 (m, 4H), 7.61–7.64 (m, 3H), 10.12 (bs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 91.7, 126.4, 127.9, 128.3, 129.1, 130.7, 130.9, 137.4, 147.2, 162.7, 182.9; IR (neat): 3455, 3356, 1599, 1526, 852, 756, 697 cm^{-1}; HRMS (ES) Calcd for $C_{13}H_{11}NOS$: 252.0459 $[M+Na]^+$; Found: 252.0465.

2-(Amino(phenyl)methylene)-3,4-dihydronaphthalen-1(2H)-one (**3j**)



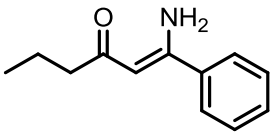
Brown oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.48 (t, J = 6.0 Hz, 2H), 2.73 (t, J = 6.0 Hz, 2H), 5.10 (bs, 1H), 7.13–7.15 (m, 1H), 7.29–7.43 (m, 7H), 8.03–8.05 (m, 1H), 10.72 (bs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 26.4, 29.8, 101.5, 126.7, 126.8, 127.5, 127.7, 128.8, 129.4, 131.4, 136.0, 137.9, 142.0, 161.1, 187.9; IR (neat): 3450, 3346, 2927, 2837, 1591, 1509, 1472, 802, 775, 748, 701 cm^{-1}; HRMS (ES) Calcd for $C_{17}H_{15}NO$: 272.1051 $[M+Na]^+$; Found: 272.1059.

2-(Amino(phenyl)methylene)cyclohexanone (**3k**)



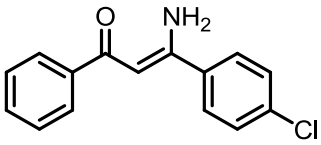
Brown oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.52–1.58 (m, 2H), 1.71–1.77 (m, 2H), 2.16 (t, J = 6.2 Hz, 2H), 2.39 (t, J = 6.8 Hz, 2H), 5.05 (bs, 1H), 7.31–7.33 (m, 2H), 7.37–7.43 (m, 3H), 10.65 (bs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 23.1, 24.2, 27.6, 38.6, 101.7, 127.5, 128.7, 129.1. 137.9, 161.3, 199.5; IR (neat): 3311, 2930, 2859, 1593, 1571, 1468, 1412, 827, 774, 702 cm^{-1}; HRMS (ES) Calcd for $C_{13}H_{15}NO$: 224.1051 $[M+Na]^+$; Found: 224.1047.

1-Amino-1-phenylhex-1-en-3-one (**3l**)



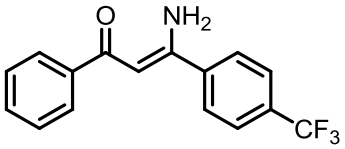
Brown oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.97 (t, J = 7.2 Hz, 3H), 1.64–1.69 (m, 2H), 2.37 (t, J = 7.6 Hz, 2H), 5.24 (bs, 1H), 5.45 (s, 1H), 7.40–7.48 (m, 3H), 7.54–7.56 (m, 2H), 9.95 (bs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 8.9, 14.1, 39.8, 89.6, 121.1, 123.8, 125.3, 132.3, 155.7, 195.4; IR (neat): 3356, 3173, 2930, 2871, 1609, 1529, 1486, 1409, 844, 755, 696 cm^{-1}; HRMS (ES) Calcd for $C_{12}H_{15}NO$: 212.1051 $[M+Na]^+$; Found: 212.1054.

3-Amino-3-(4-chlorophenyl)-1-phenylprop-2-en-1-one (**3n**)



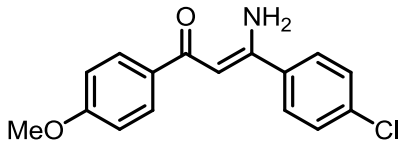
Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.56 (bs, 1H), 6.09 (s, 1H), 7.40–7.49 (m, 5H), 7.56 (d, J = 8.4 Hz, 2H), 7.91–7.93 (m, 2H), 10.36 (bs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 91.9, 127.2, 127.8, 128.4, 129.2, 131.2, 135.9, 136.7, 140.2, 161.8, 190.2; IR (neat): 3317, 3164, 1593, 1526, 845, 783, 750, 642 cm^{-1}; HRMS (ES) Calcd for $C_{15}H_{12}ClNO$: 280.0505 $[M+Na]^+$; Found: 280.0497.

3-Amino-1-phenyl-3-(4-(trifluoromethyl)phenyl)prop-2-en-1-one (**3o**)



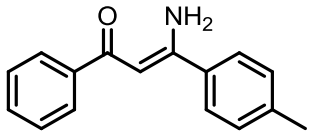
Yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.73 (bs, 1H), 6.10 (s, 1H), 7.40–7.47 (m, 3H), 7.67–7.73 (m, 4H), 7.91 (d, J = 7.5 Hz, 2H), 10.32 (bs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 92.4, 123.8 ($^{1}J_{C\text{-}F}$ = 271 Hz), 126.0, 127.1, 127.3, 128.5, 131.4, 132.4 ($^{2}J_{C\text{-}F}$ = 32 Hz), 140.0, 141.1, 161.5, 190.5; IR (KBr): 3372, 3186, 1603, 1537, 1326, 850, 804, 759, 734, 695 cm^{-1}; HRMS (ES) Calcd for $C_{16}H_{12}F_3NO$: 314.0769 $[M+Na]^+$; Found: 314.0788.

3-Amino-3-(4-chlorophenyl)-1-(4-methoxyphenyl)prop-2-en-1-one (**3p**)



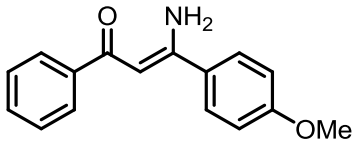
Yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.85 (s, 3H), 5.59 (bs, 1H), 6.06 (s, 1H), 6.93 (d, J = 8.8 Hz, 2H), 7.41 (d, J = 8.4 Hz, 2H), 7.56 (d, J = 8.4 Hz, 2H), 7.91–7.93 (m, 2H), 10.29 (bs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.4, 91.7, 113.6, 127.7, 129.2, 129.3, 132.9, 136.3, 136.6, 160.9, 162.2, 189.3; IR (KBr): 3360, 2932, 2837, 1597, 1557, 1481, 1415, 1387, 841, 783 cm^{-1}; HRMS (ES) Calcd for $C_{16}H_{14}ClNO_2$: 310.0611 $[M+Na]^+$; Found: 310.0621.

3-Amino-1-phenyl-3-p-tolylprop-2-en-1-one (**3q**)



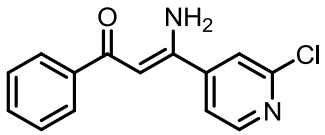
Red oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.40 (s, 3H), 5.54 (bs, 1H), 6.13 (s, 1H), 7.25 (d, J = 7.2 Hz, 2H), 7.42–7.44 (m, 3H), 7.52 (d, J = 7.6 Hz, 2H), 7.94 (d, J = 6.8 Hz, 2H), 10.44 (bs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.5, 91.6, 126.3, 127.3, 128.4, 129.8, 131.0, 134.7, 140.6, 141.2, 163.1, 190.1; IR (neat): 3460, 3357, 2921, 2855, 1598, 1535, 1486, 828, 752, 718, 693 cm^{-1}; HRMS (ES) Calcd for $C_{16}H_{15}NO$: 260.1051 $[M+Na]^+$; Found: 260.1049.

3-Amino-3-(4-methoxyphenyl)-1-phenylprop-2-en-1-one (**3r**)



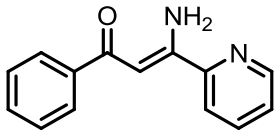
Red oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.83 (s, 3H), 5.54 (bs, 1H), 6.11 (s, 1H), 6.95 (d, J = 8.4 Hz, 2H), 7.40–7.45 (m, 3H), 7.58 (d, J = 8.8 Hz, 2H), 7.93 (d, J = 6.4 Hz, 2H), 10.46 (bs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.6, 91.3, 114.4, 127.3, 127.9, 128.4, 129.7, 131.0, 140.7, 161.8, 162.8, 189.9; IR (neat): 3357, 3165, 2931, 2838, 1607, 1536, 1500, 1439, 1394, 838, 757, 726, 695 cm^{-1}; HRMS (ES) Calcd for $C_{16}H_{15}NO_2$: 276.1000 $[M+Na]^+$; Found: 276.0999.

3-Amino-3-(2-chloropyridin-4-yl)-1-phenylprop-2-en-1-one (**3s**)



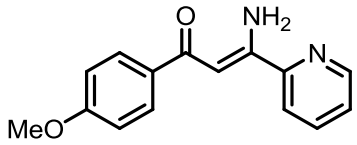
Light yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.71 (bs, 1H), 6.10 (s, 1H), 7.42–7.52 (m, 4H), 7.54 (s, 1H), 7.91 (d, J = 7.2 Hz, 2H), 8.46 (d, J = 4.8 Hz, 1H), 10.14 (bs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 92.8, 119.8, 121.7, 127.3, 128.6, 131.8, 139.6, 148.1, 150.6, 152.5, 158.3, 190.9; IR (neat): 3346, 3172, 1608, 1526, 849, 753, 722, 694 cm^{-1}; HRMS (ES) Calcd for $C_{14}H_{11}ClN_2O$: 281.0458 $[M+Na]^+$; Found: 281.0449.

3-Amino-1-phenyl-3-(pyridin-2-yl)prop-2-en-1-one (**3t**)



Light yellow oil. ^{1}H NMR (400 MHz, CDCl3) δ 6.52 (s, 1H), 7.37–7.50 (m, 5H), 7.80 (ddd, J = 7.7, 7.7, 1.6 Hz, 1H), 7.92 (d, J = 8.0 Hz, 1H), 7.98 (dd, J = 7.8, 1.6 Hz, 2H), 8.67 (d, J = 4.5 Hz, 1H), 10.30 (bs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 89.0, 120.5, 125.3, 127.2, 128,3, 131.0, 137.0, 140.5, 149.1, 151.1, 157.4, 190.5; IR (neat): 3061, 3027, 1643, 1451, 857, 752, 693 cm^{-1}; HRMS (ES) Calcd for $C_{14}H_{12}N_2O$: 224.0950 $[M+Na]^+$; Found: 224.0946.

3-Amino-1-(4-methoxyphenyl)-3-(pyridin-2-yl)prop-2-en-1-one (**3u**)



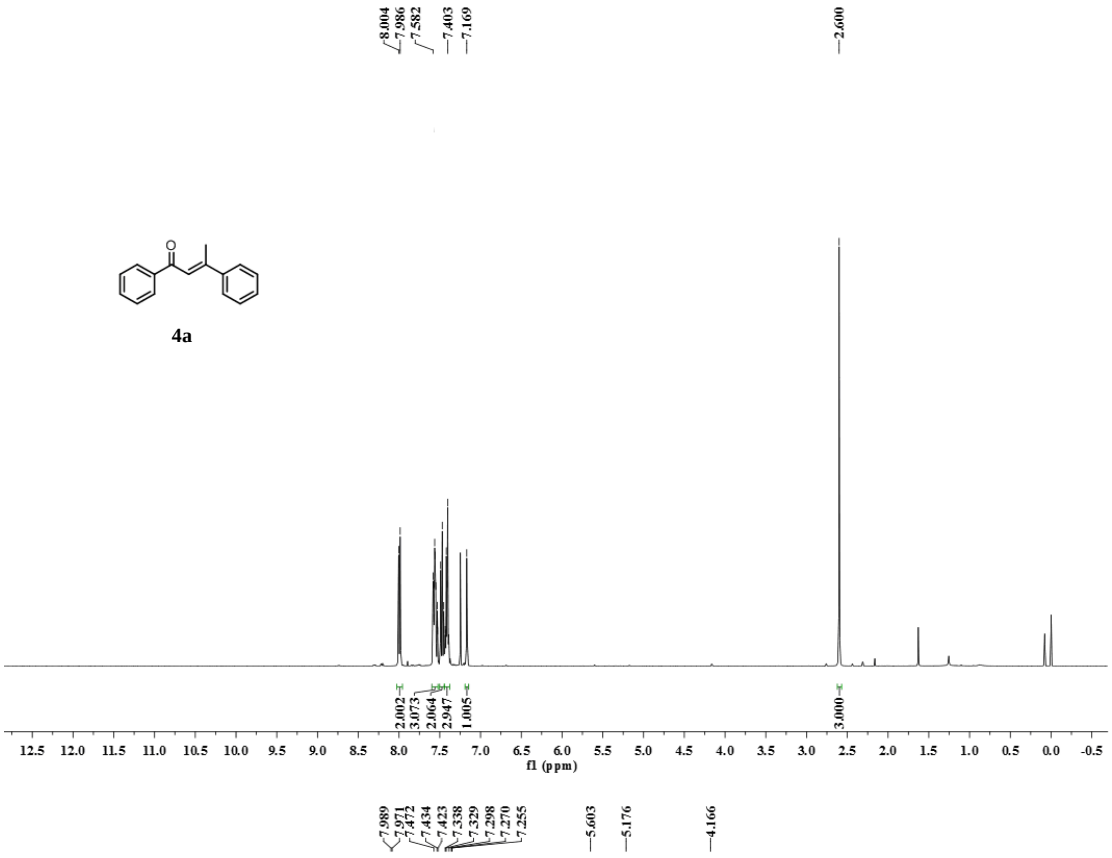
Light yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.82 (s, 3H), 6.48 (s, 1H), 6.93 (d, J = 7.7 Hz, 2H), 7.32–7.36 (m, 2H), 7.73–7.77 (m, 1H), 7.89 (d, J = 7.8 Hz, 1H), 7.98 (d, J = 8.5 Hz, 2H), 8.63 (bs, 1H), 10.22 (bs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.5, 88.8, 113.6, 120.5, 125.2, 129.2, 133.3, 137.0, 149.1, 151.3, 157.0, 162.2, 189.5; IR (neat): 3417, 3240, 2936, 2840, 1620, 1557, 1460, 1441, 1390, 843, 773, 714 cm^{-1}; HRMS (ES) Calcd for $C_{15}H_{14}N_2O_2$: 277.0953 $[M+Na]^+$; Found: 277.0950.

4. References

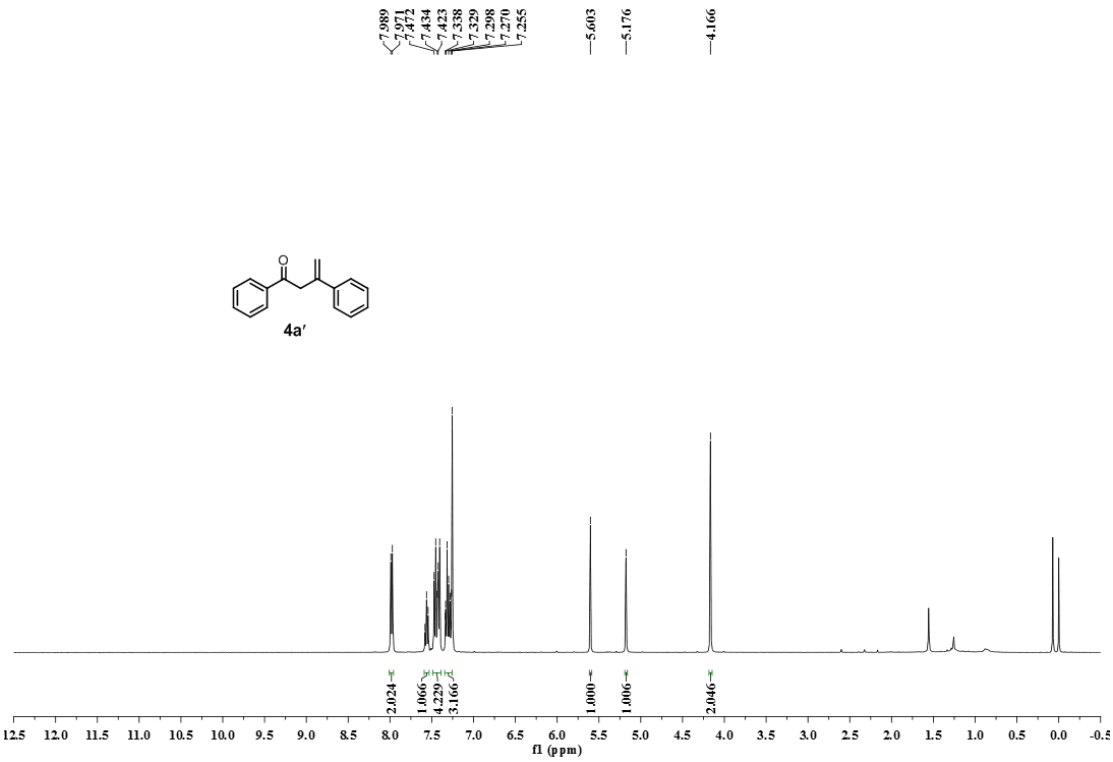
[1] Y. Nishikawa and H. Yamamoto, *J. Am. Chem. Soc.*, 2011, **133**, 8432.

[2] R. F. Klima, A. V. Jadhav, P. N. D. Singh, M. Chang, C. Vanos, J. Sankaranarayanan, M. Vu, N. Ibrahim, E. Ross, S. McCloskey, R. S. Murthy, J. A. Krause, B. S. Ault and A. D. Gudmundsdóttir, *J. Org. Chem.*, 2007, **72**, 6372.

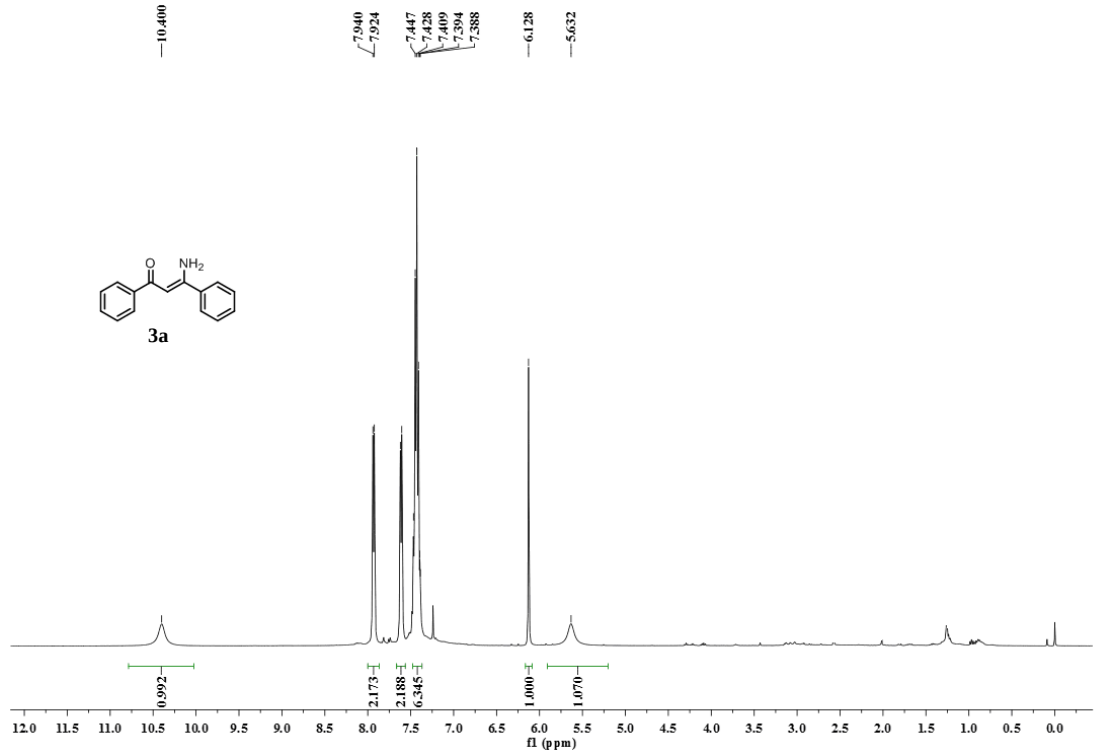
5. ^{1}H and ^{13}C NMR Spectra

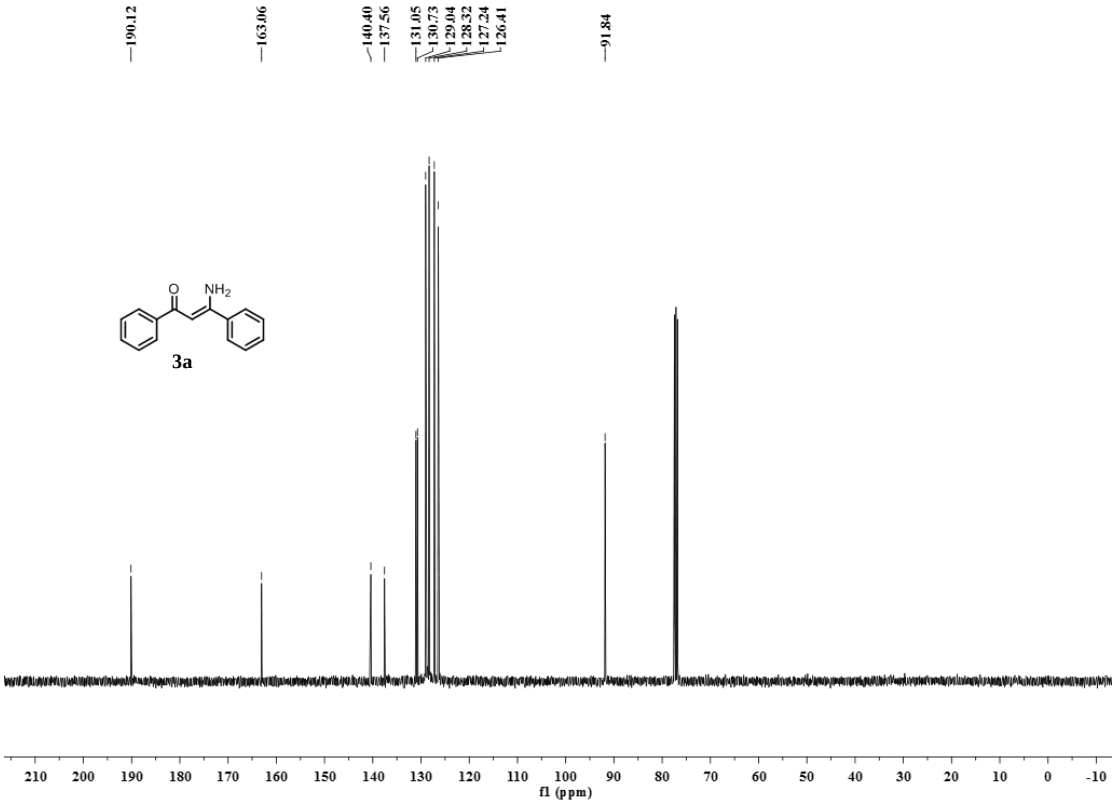


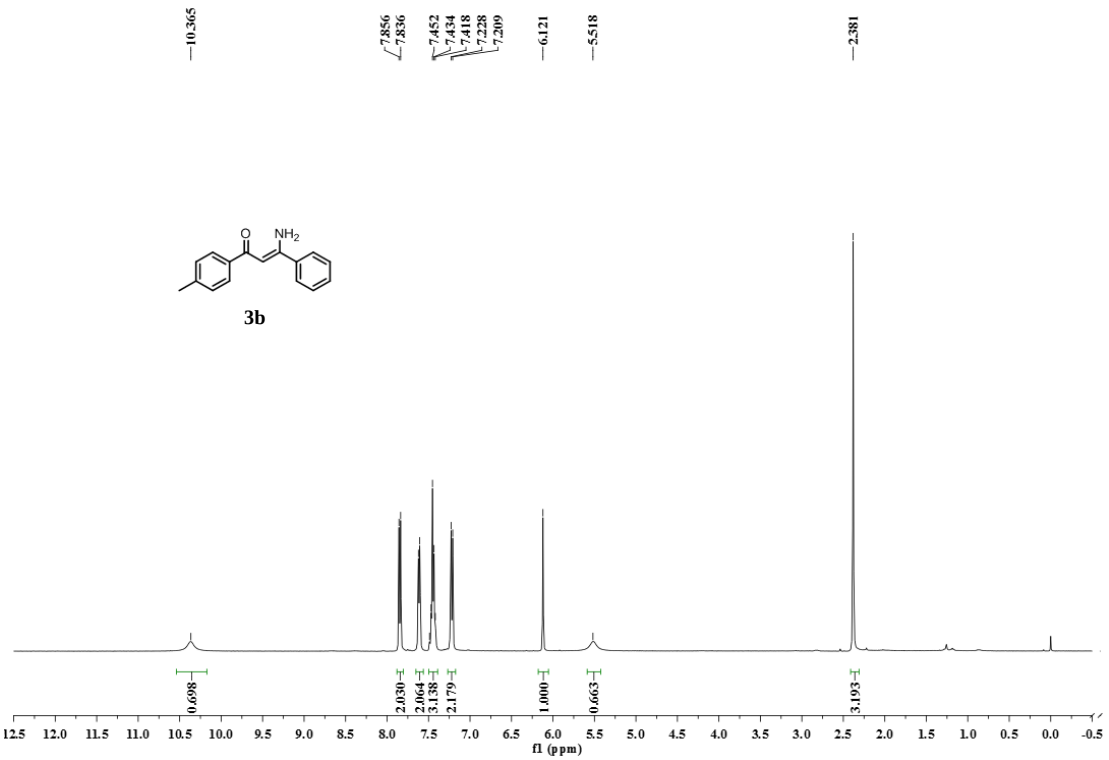
4a



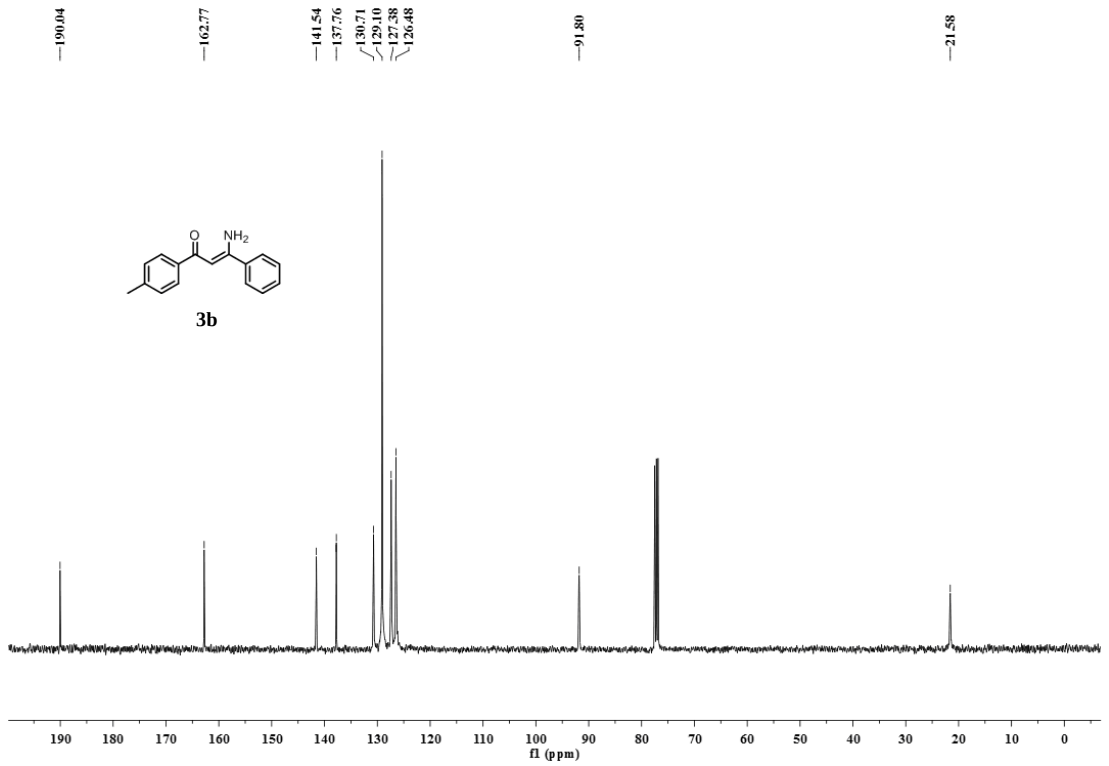
4a′


10.400
7.940
7.924
7.447
7.428
7.409
7.394
7.388
6.128
5.632
O
NH2
3a
0.992
2.173
2.188
6.345
1.000
1.070
f1 (ppm)

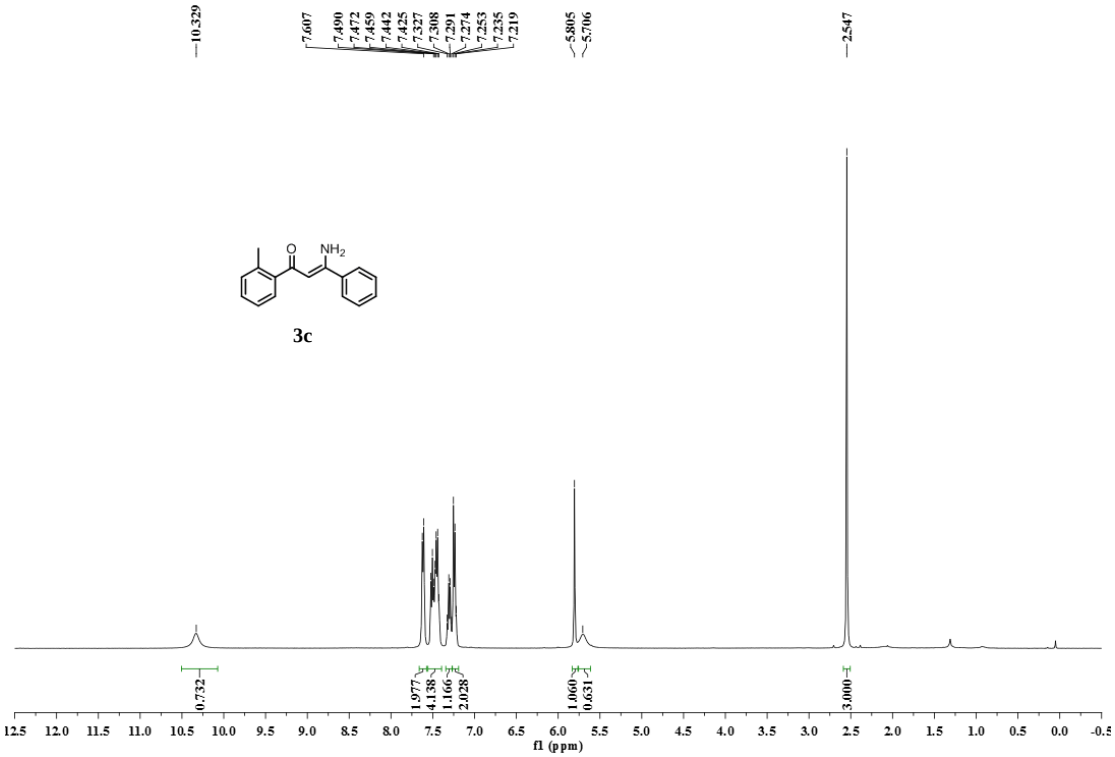

190.12
163.06
140.40
137.56
131.05
130.73
129.04
128.32
127.24
126.41
91.84
O
NH2
3a
f1 (ppm)


10.365
7.856
7.836
7.452
7.434
7.418
7.228
7.209
6.121
5.518
2.381
O
NH2
0.698
2.030
2.064
3.138
2.179
1.000
0.663
3.193
f1 (ppm)

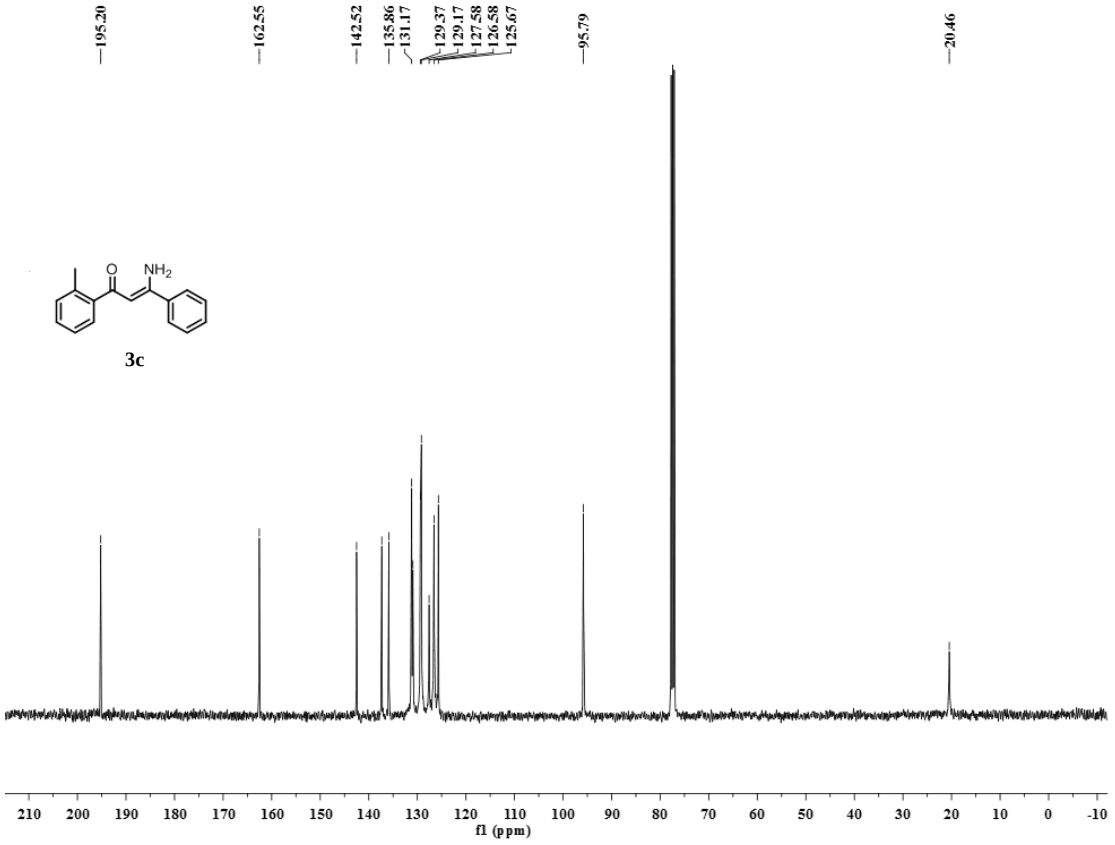
3b


190.04
162.77
141.54
137.76
130.71
129.10
127.38
126.48
91.80
21.58
O
NH2
f1 (ppm)

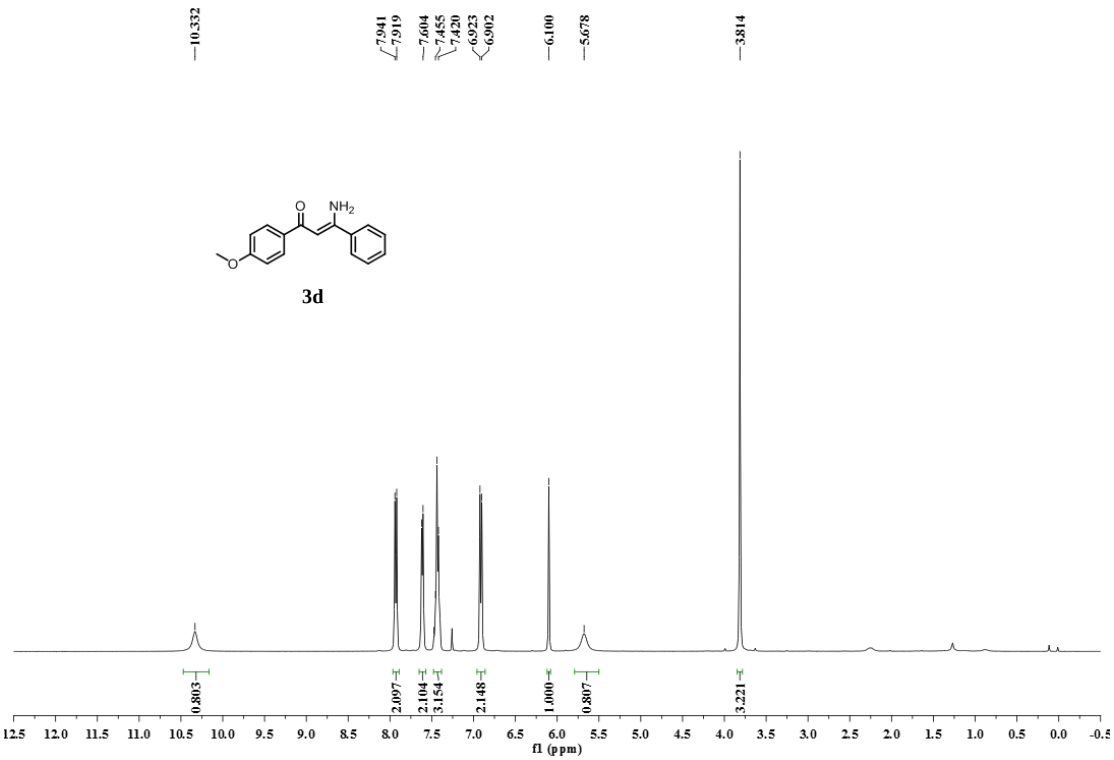
3b


10.329
7.607
7.490
7.472
7.459
7.442
7.425
7.327
7.308
7.291
7.274
7.253
7.235
7.219
5.805
5.706
2.547
O
NH2
0.732
1.977
4.138
1.166
2.028
1.060
0.631
3.000
f1 (ppm)

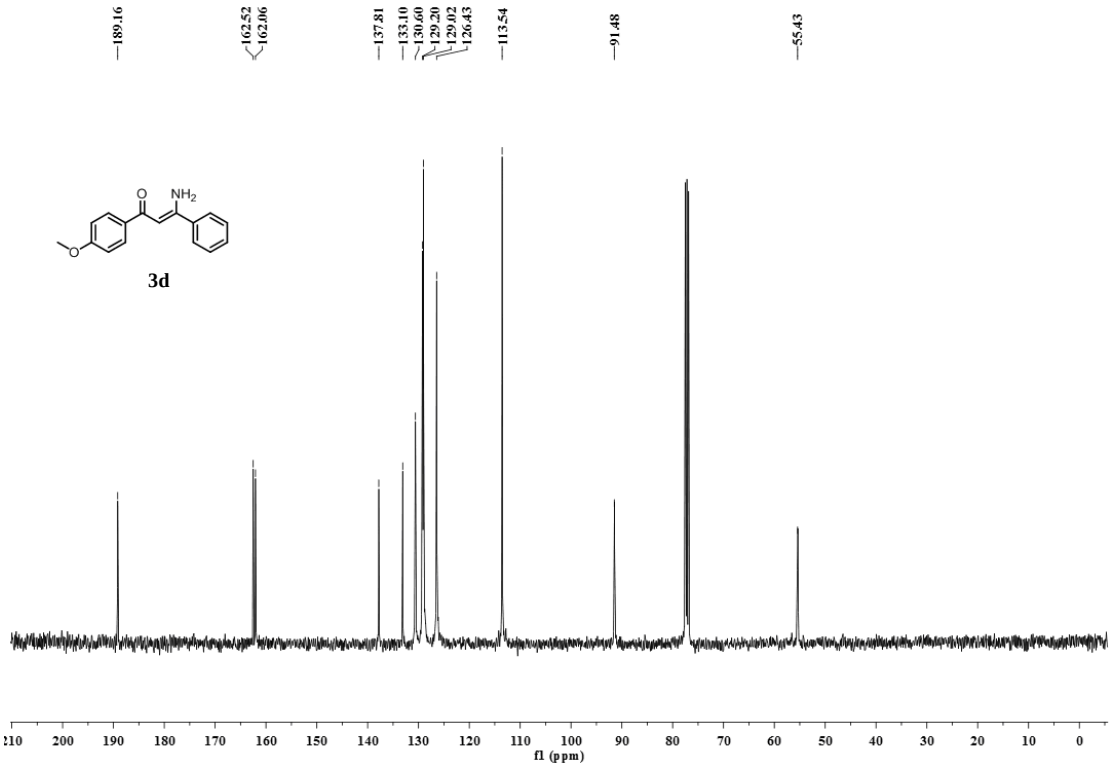
3c


195.20
162.55
142.52
135.86
131.17
129.37
129.17
127.58
126.58
125.67
95.79
20.46
O
NH2
f1 (ppm)

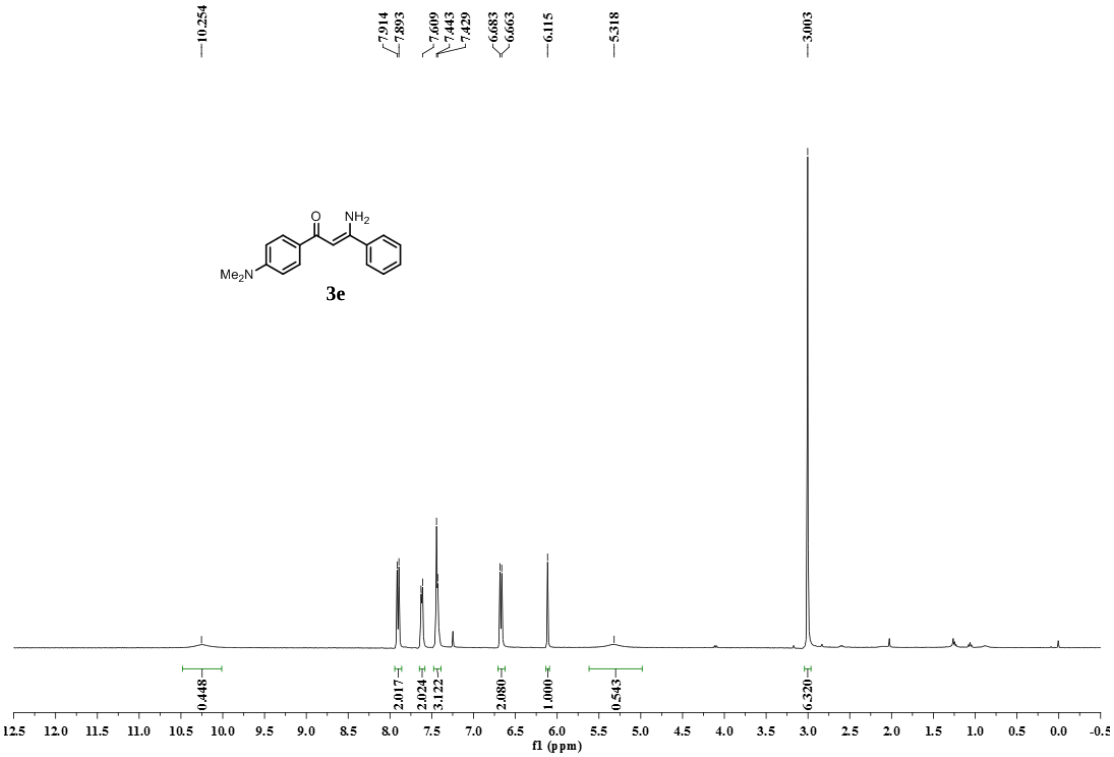
3c


10.332
7.941
7.919
7.604
7.455
7.420
6.923
6.902
6.100
5.678
3.814
0.803
2.097
2.104
3.154
2.148
1.000
0.807
3.221
f1 (ppm)

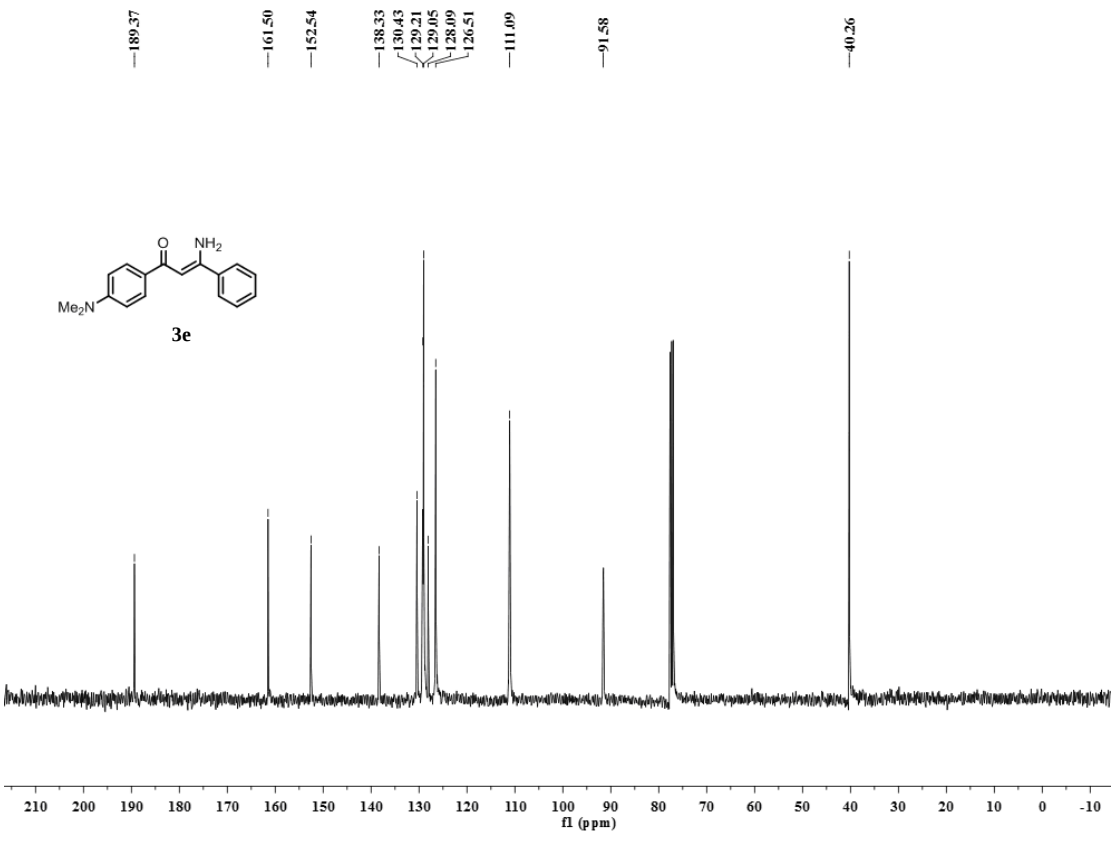
3d


189.16
162.52
162.06
137.81
133.10
130.60
129.20
129.02
126.43
113.54
91.48
55.43
f1 (ppm)

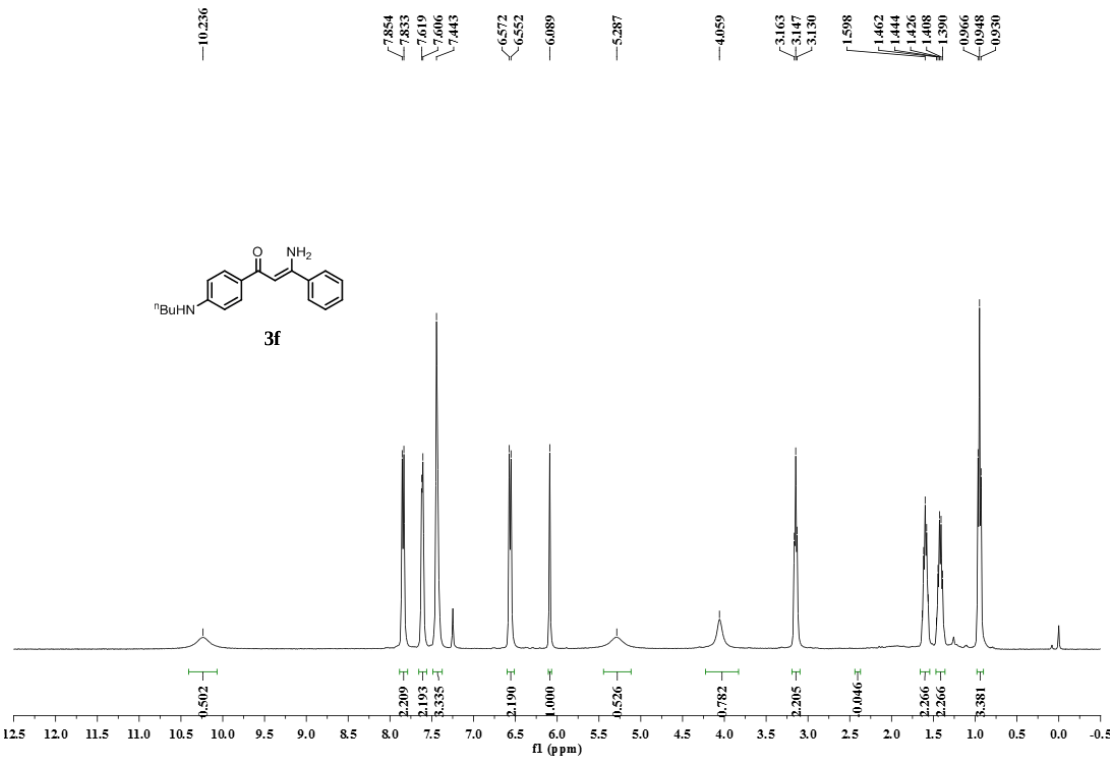
3d


10.254
7.914
7.893
7.609
7.443
7.429
6.683
6.663
6.115
5.318
3.003
O
NH2
Me2N
0.448
2.017
2.024
3.122
2.080
1.000
0.543
6.320
f1 (ppm)

3e


189.37
161.50
152.54
138.33
130.43
129.21
129.05
128.09
126.51
111.09
91.58
40.26
O
NH2
Me2N
f1 (ppm)

3e


10.236
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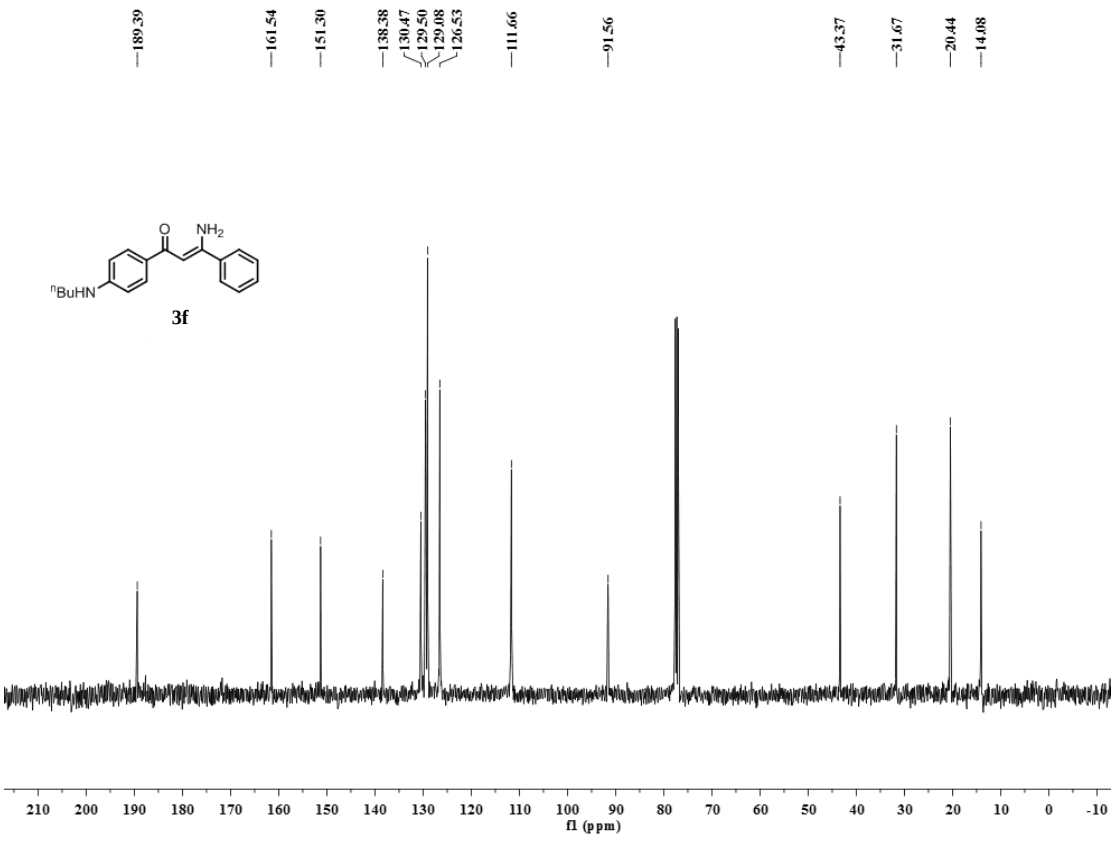
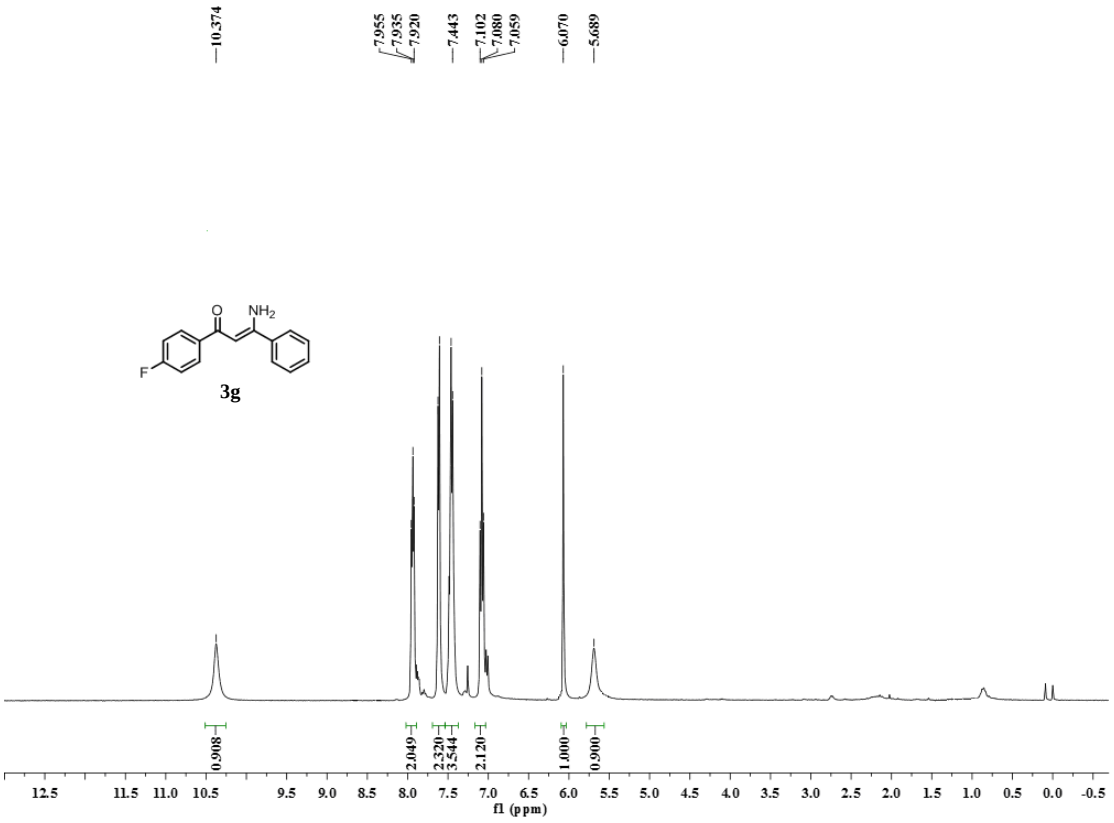
7.833
7.619
7.606
7.443
6.572
6.552
6.089
5.287
4.059
3.163
3.147
3.130
1.598
1.462
1.444
1.426
1.408
1.390
0.966
0.948
0.930
O
NH2
nBuHN
3f
0.502
2.209
2.193
3.335
2.190
1.000
0.526
0.782
2.205
0.046
2.266
2.266
3.381
f1 (ppm)


189.39
161.54
151.30
138.38
130.47
129.50
129.08
126.53
111.66
91.56
43.37
31.67
20.44
14.08
O
NH2
nBuHN
3f
f1 (ppm)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7.935
7.920
7.443
7.102
7.080
7.059
6.070
5.689
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F
3g
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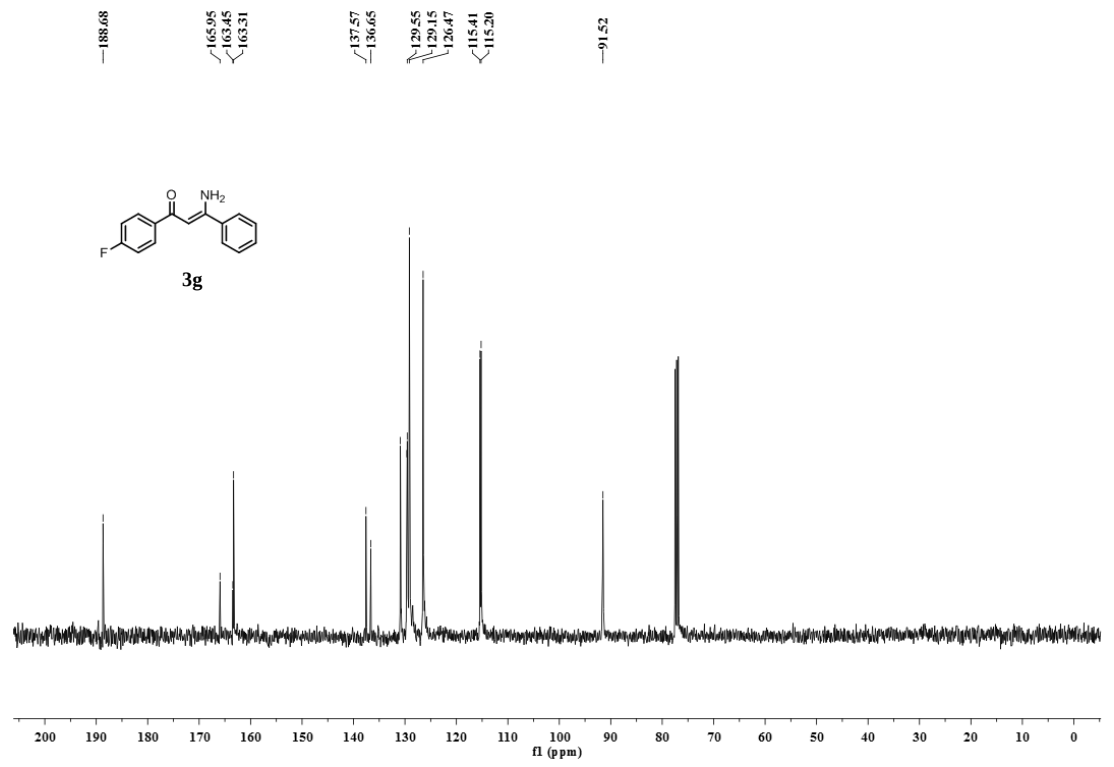
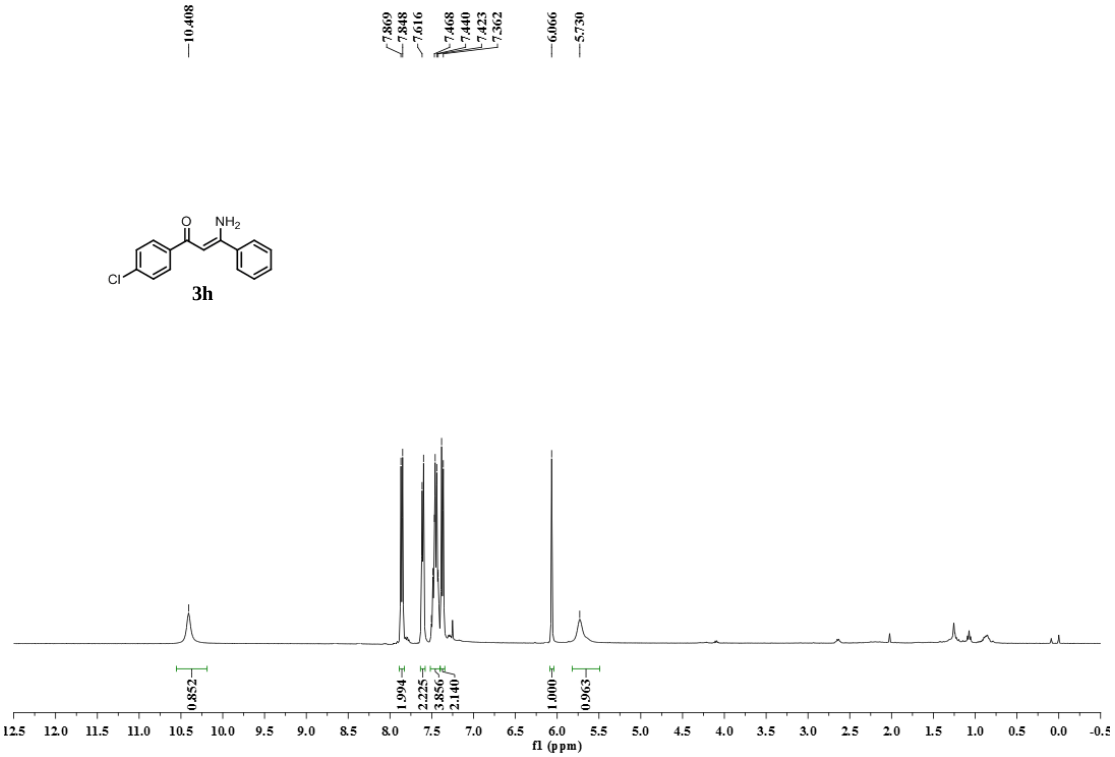
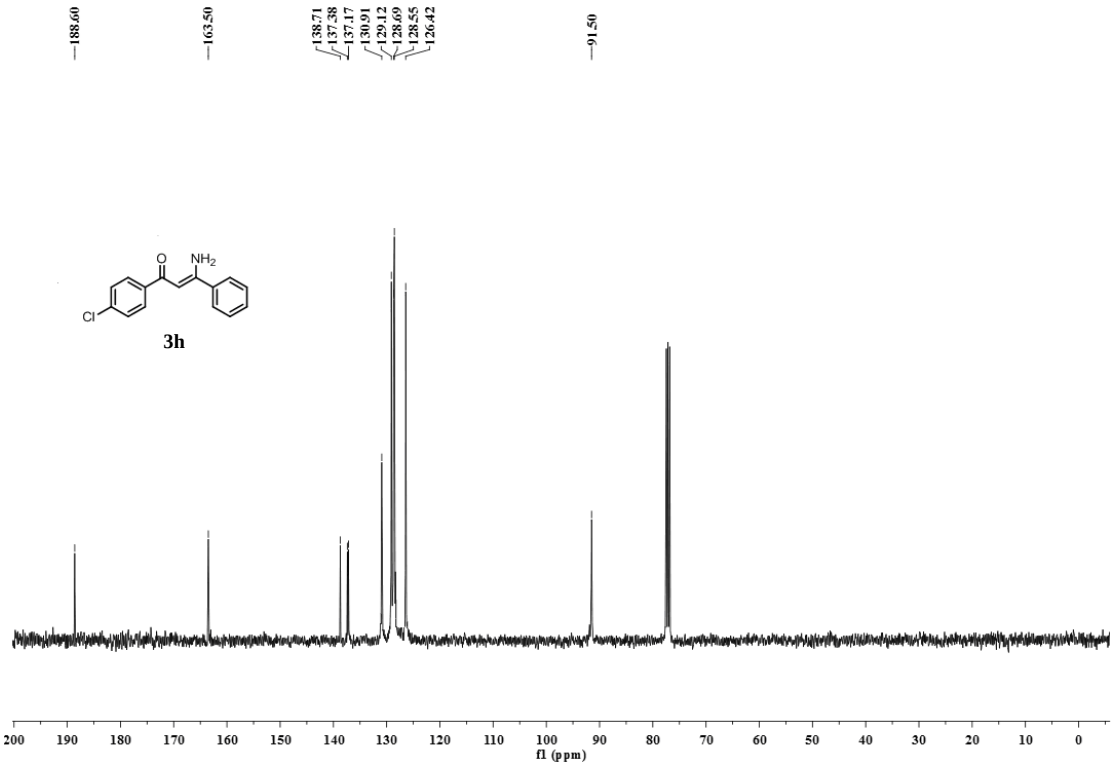
2.220
3.544
2.120
1.000
0.900
fl (ppm)


188.68
165.95
163.45
163.31
137.57
136.65
129.55
129.15
126.47
115.41
115.20
91.52
O
NH2
F
3g
fl (ppm)


10.408
7.869
7.848
7.616
7.468
7.440
7.423
7.362
6.066
5.730
O
NH2
Cl
0.852
1.994
2.225
3.856
2.140
1.000
0.963
f1 (ppm)

3h


188.60
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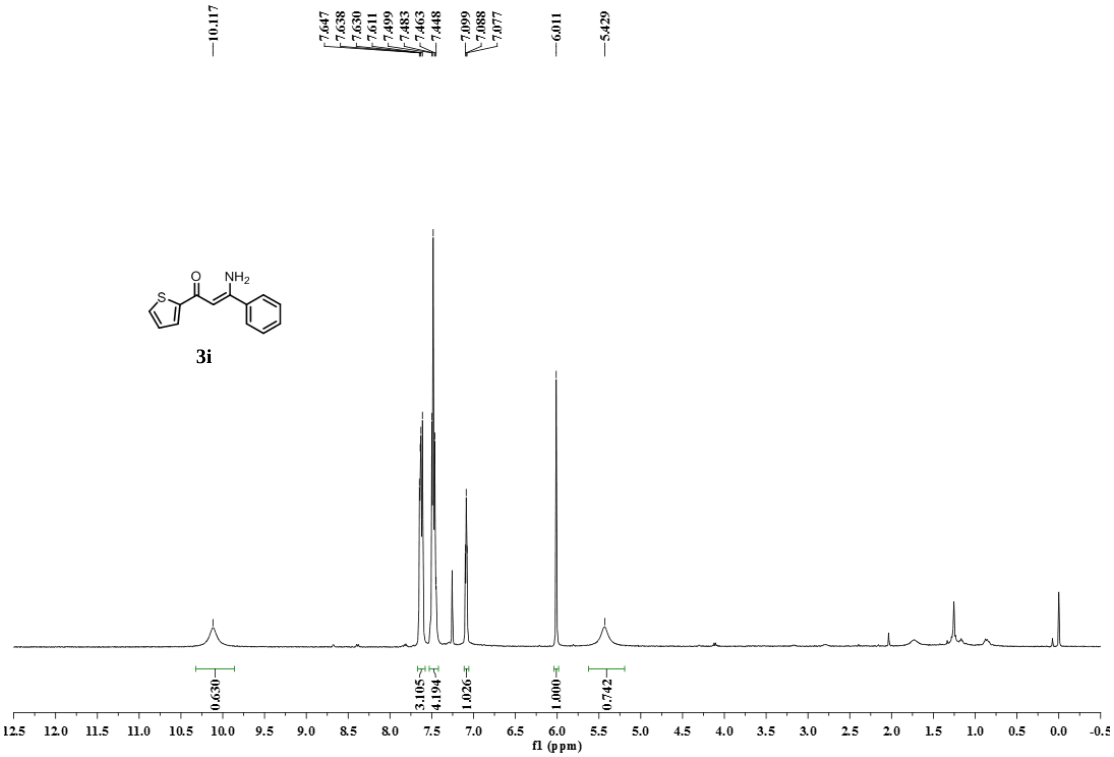
138.71
137.38
137.17
130.91
129.12
128.69
128.55
126.42
91.50
O
NH2
Cl
f1 (ppm)

3h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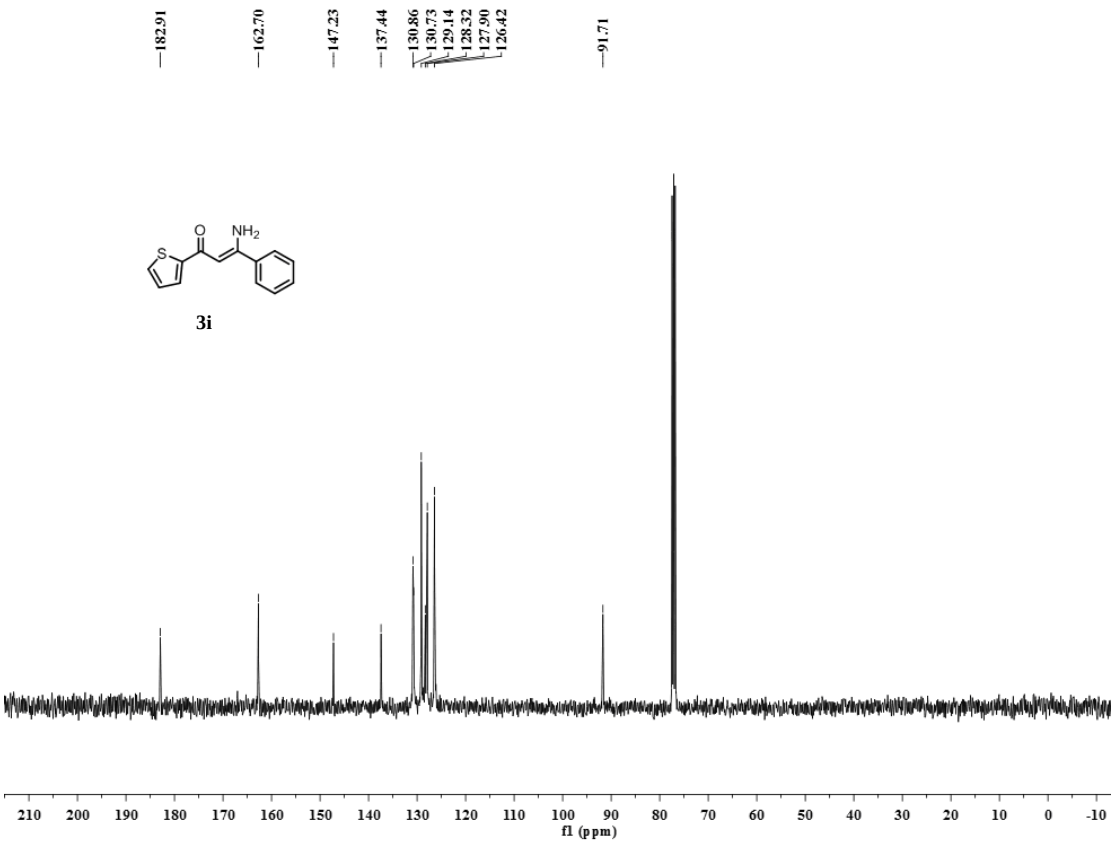
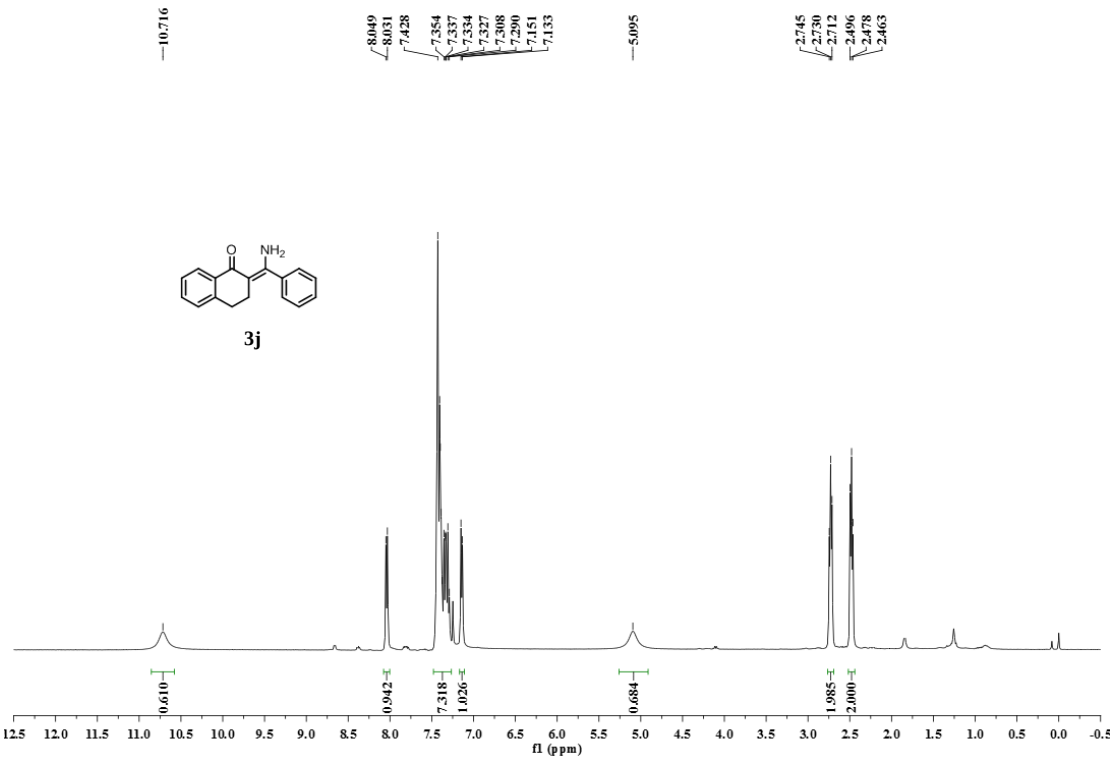
7.630
7.611
7.499
7.483
7.463
7.448
7.099
7.088
7.077
6.011
5.429
O
NH2
S
0.630
3.105
4.194
1.026
1.000
0.742
f1 (ppm)

3i


182.91
162.70
147.23
137.44
130.86
130.73
129.14
128.32
127.90
126.42
91.71
O
NH2
S
f1 (ppm)

3i


10.716
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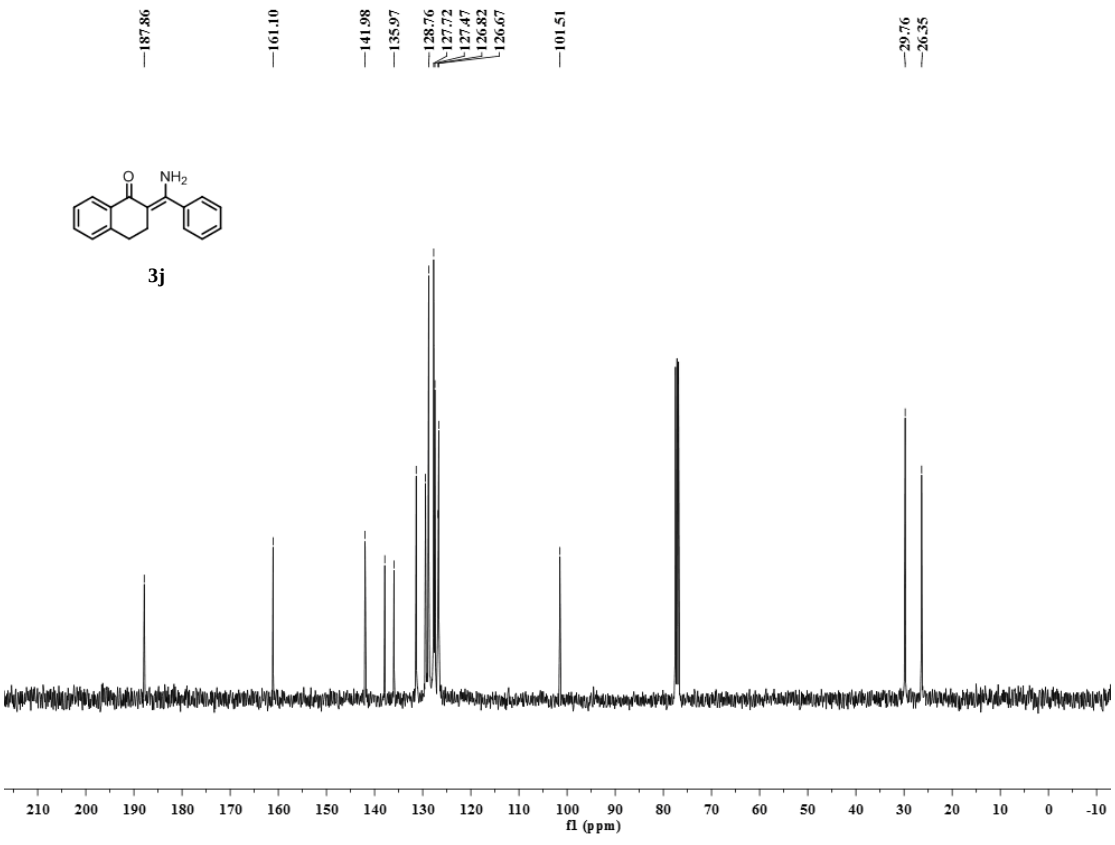
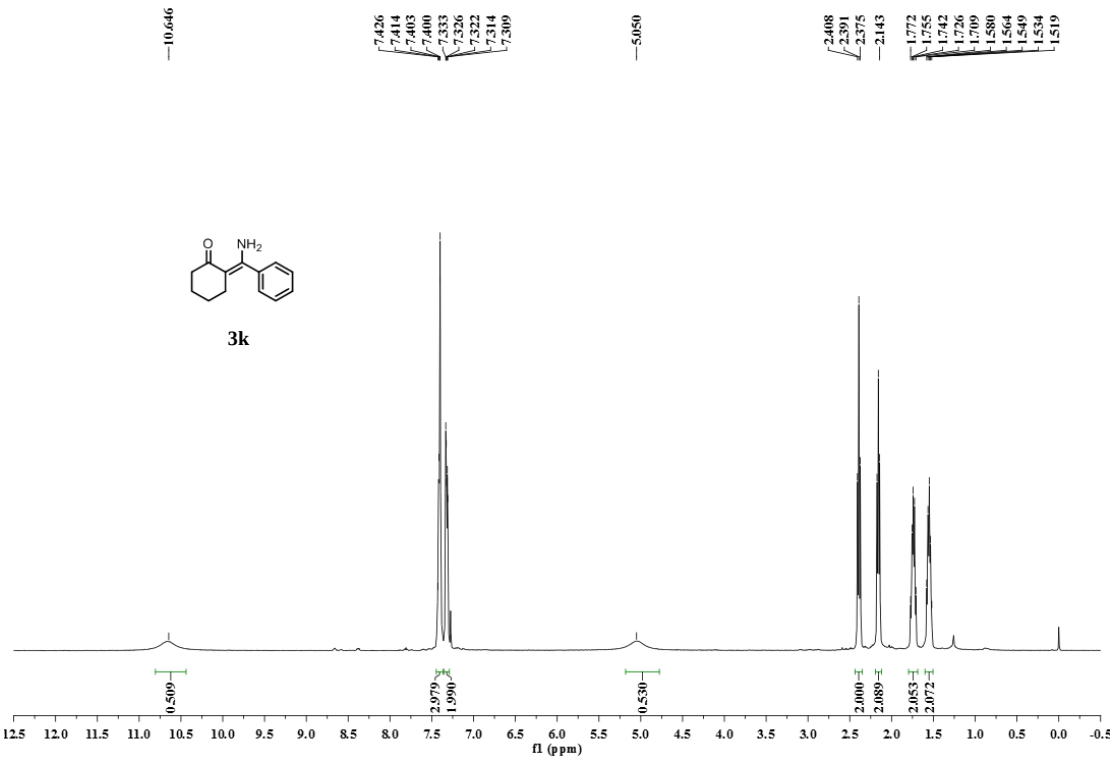
7.354
7.337
7.334
7.327
7.308
7.290
7.151
7.133
5.095
2.745
2.730
2.712
2.496
2.478
2.463
O
NH2
3j
0.610
0.942
7.318
1.026
0.684
1.985
2.000
f1 (ppm)


187.86
161.10
141.98
135.97
128.76
127.72
127.47
126.82
126.67
101.51
29.76
26.35
O
NH2
3j
f1 (ppm)


10.646
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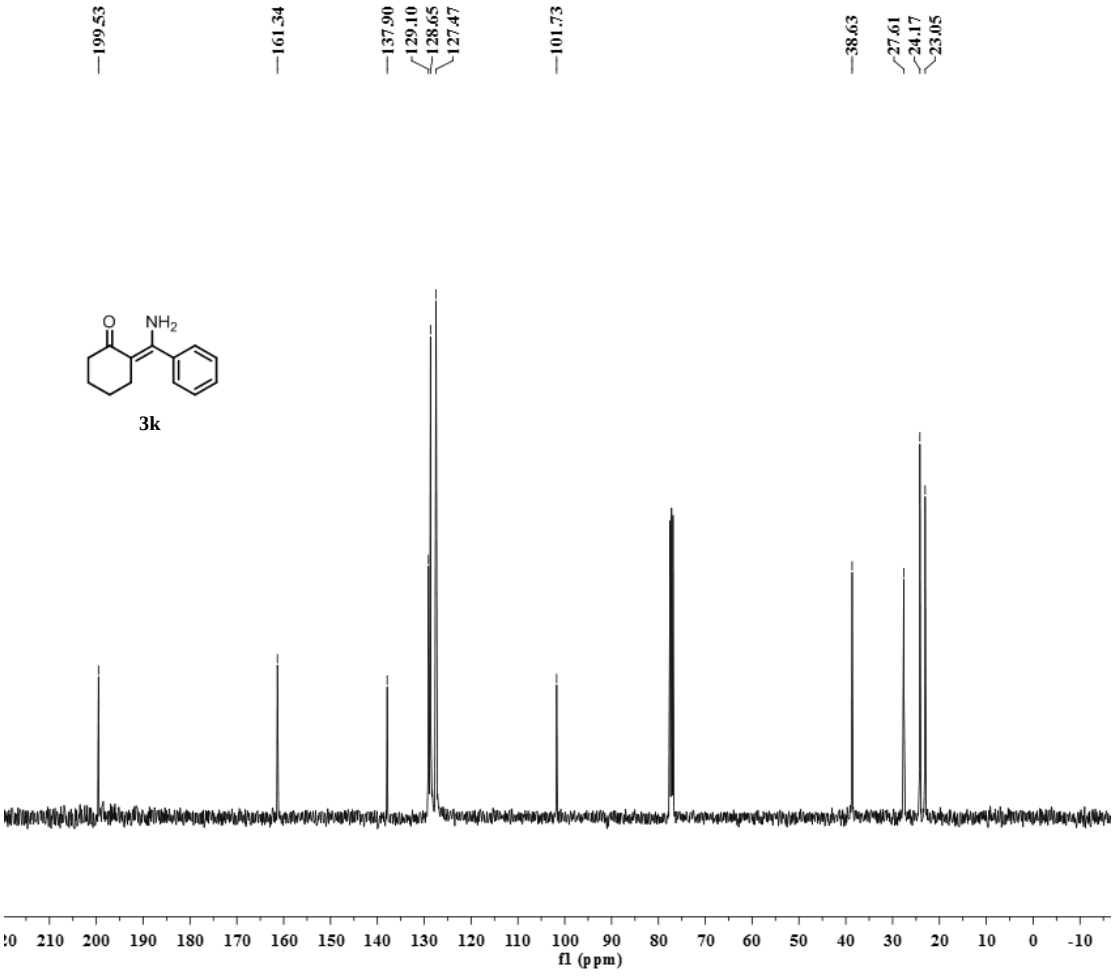
7.403
7.400
7.333
7.326
7.322
7.314
7.309
5.050
2.408
2.391
2.375
2.143
1.772
1.755
1.742
1.726
1.709
1.580
1.564
1.549
1.534
1.519
O
NH2
3k
0.509
2.979
1.990
0.530
2.000
2.089
2.053
2.072
fl (ppm)


199.53
161.34
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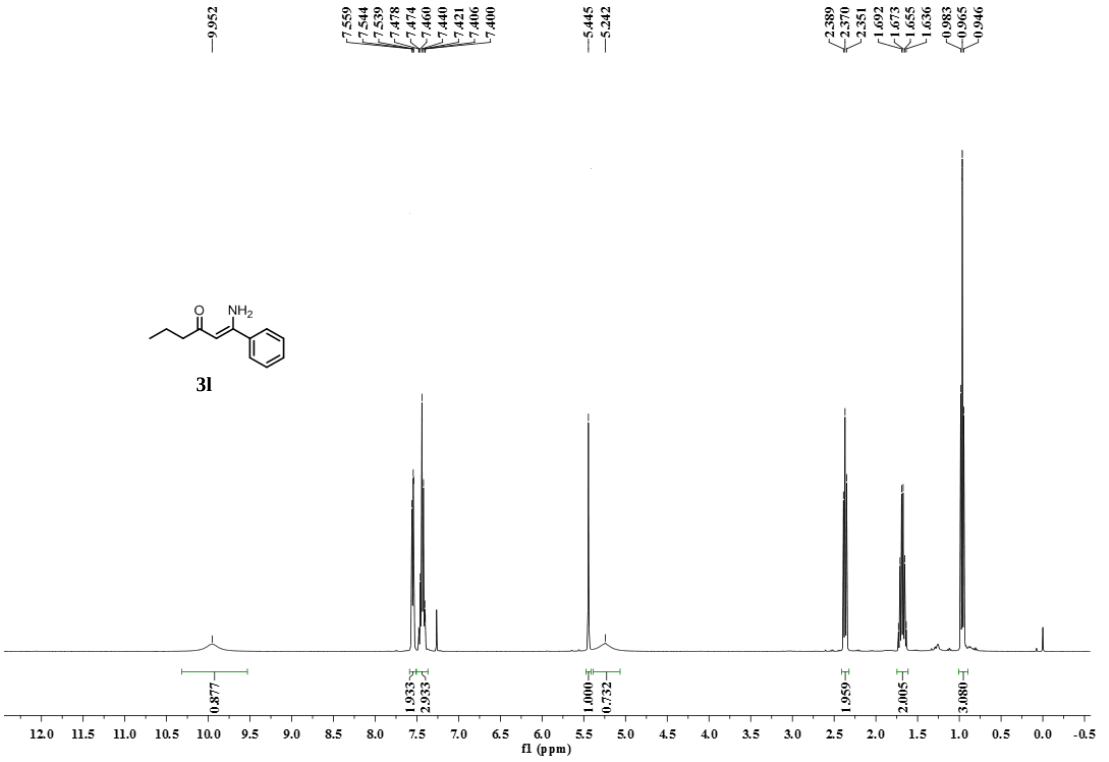
129.10
128.65
127.47
101.73
38.63
27.61
24.17
23.05
O
NH2
3k
fl (ppm)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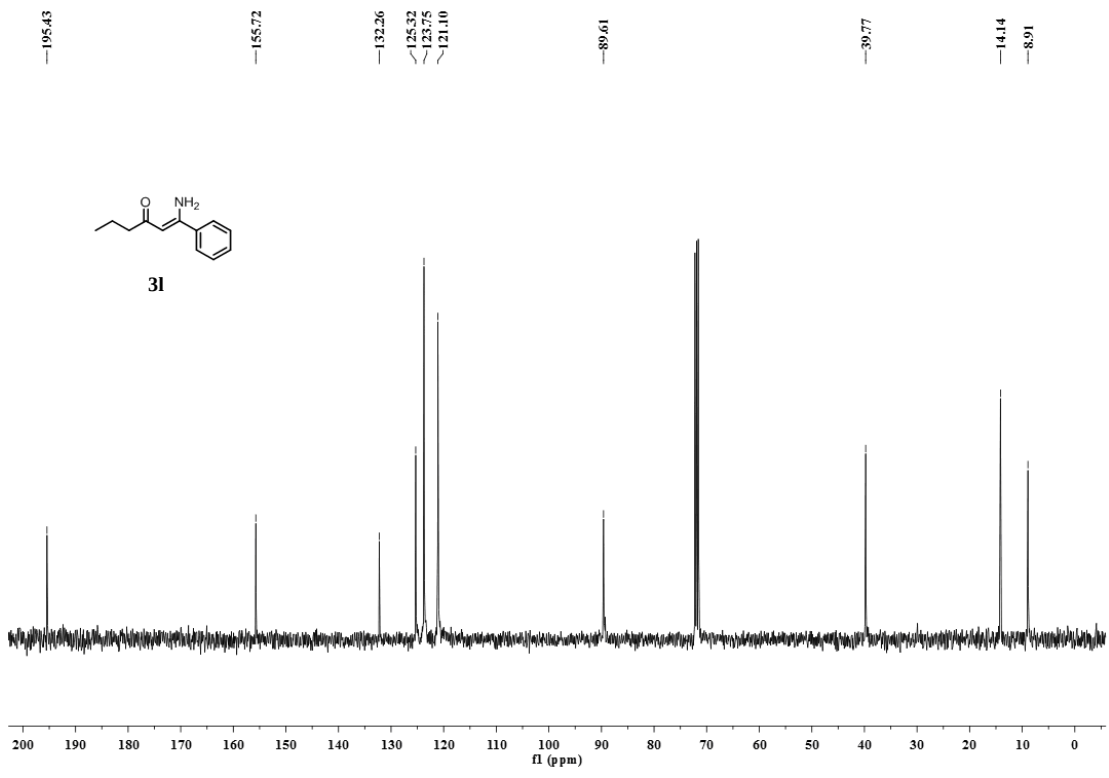
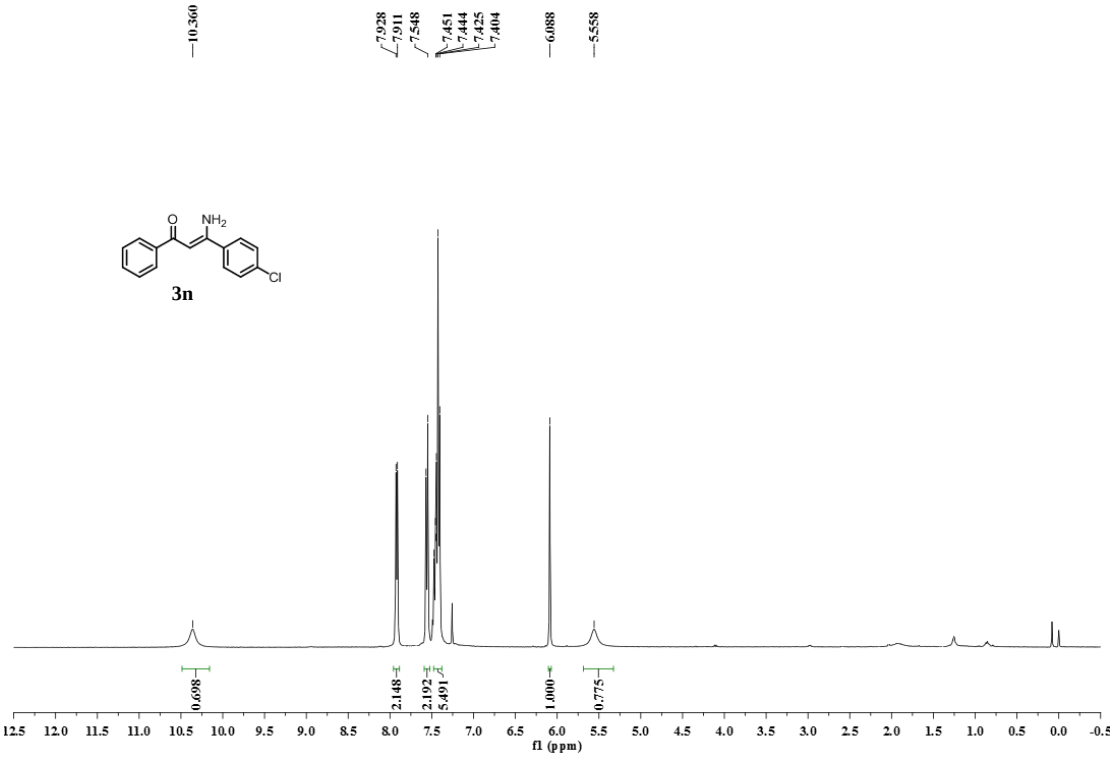
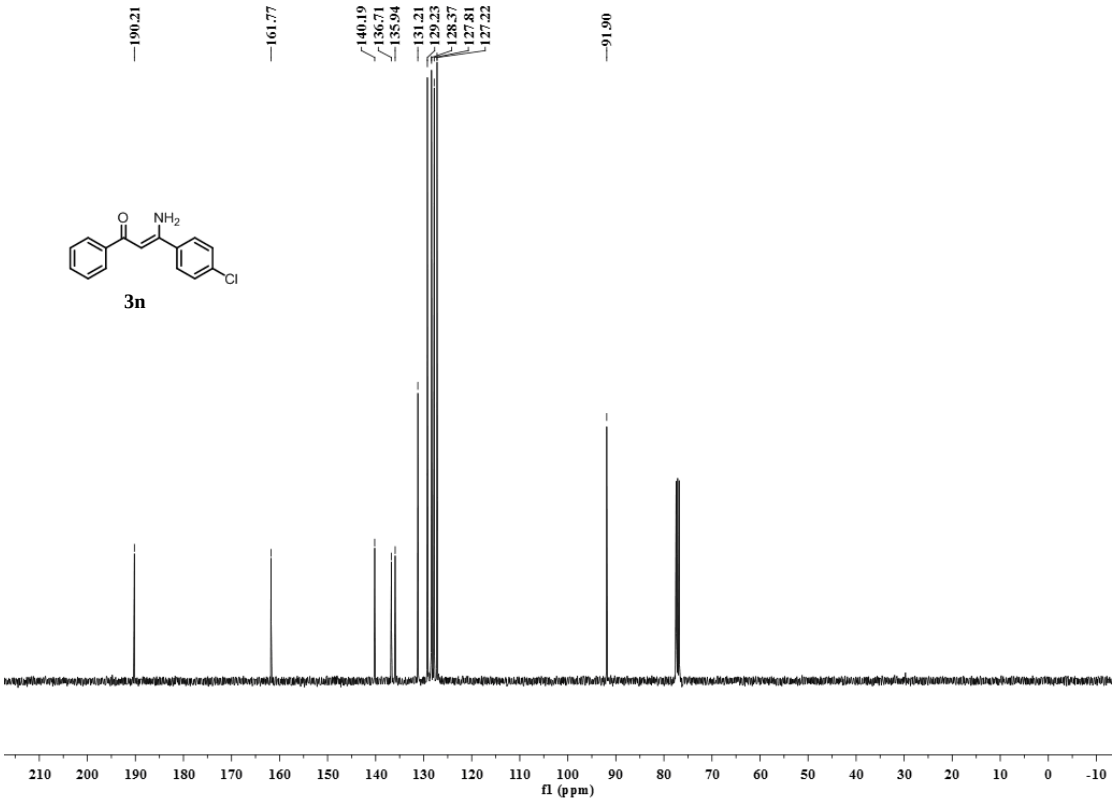
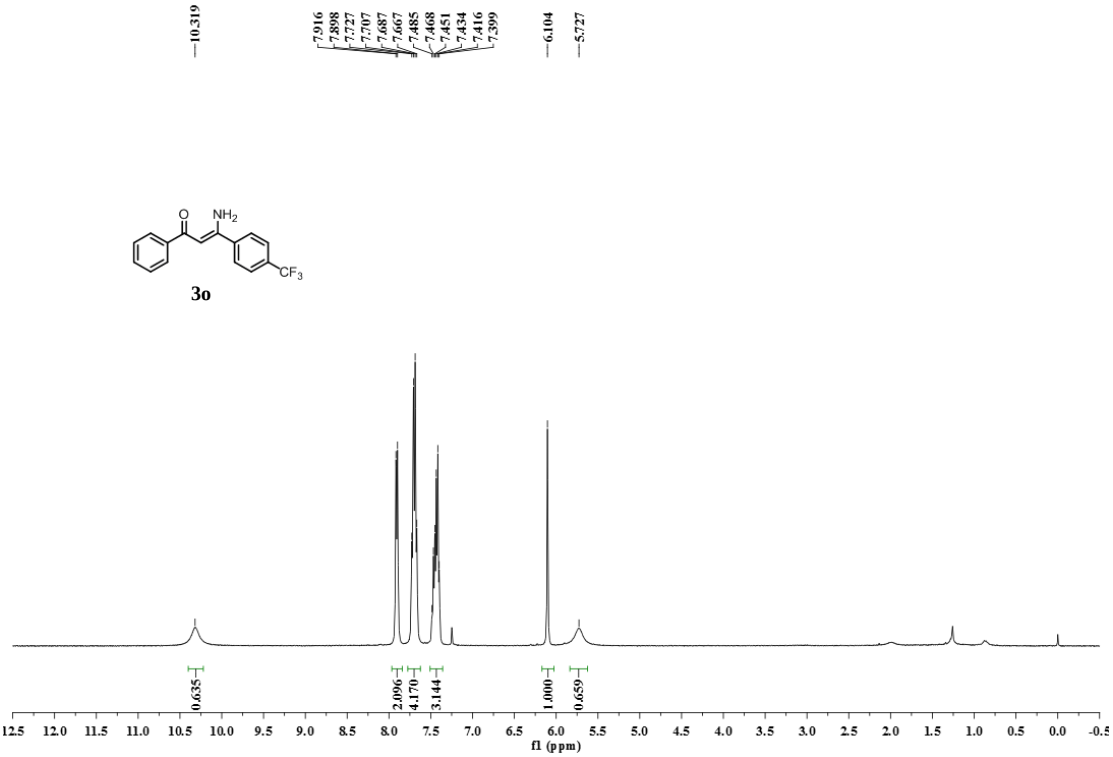
7.544
7.539
7.478
7.474
7.460
7.440
7.421
7.406
7.400
5.445
5.242
2.389
2.370
2.351
1.692
1.673
1.655
1.636
0.983
0.965
0.946
O
NH2
3l
0.877
1.933
2.933
1.000
0.732
1.959
2.005
3.080
f1 (ppm)

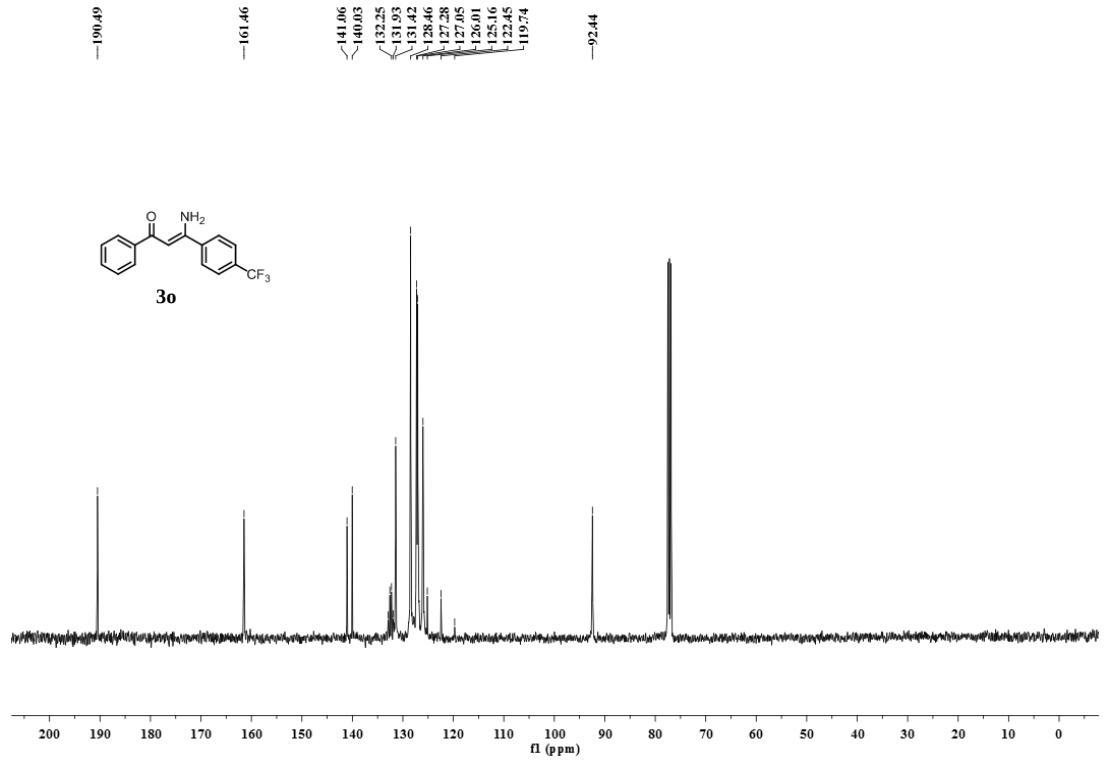

195.43
155.72
132.26
125.32
123.75
121.10
89.61
39.77
14.14
8.91
O
NH2
3l
f1 (ppm)


10.360
7.928
7.911
7.548
7.451
7.444
7.425
7.404
6.088
5.558
O
NH2
Cl
3n
0.698
2.148
2.192
5.491
1.000
0.775
f1 (ppm)

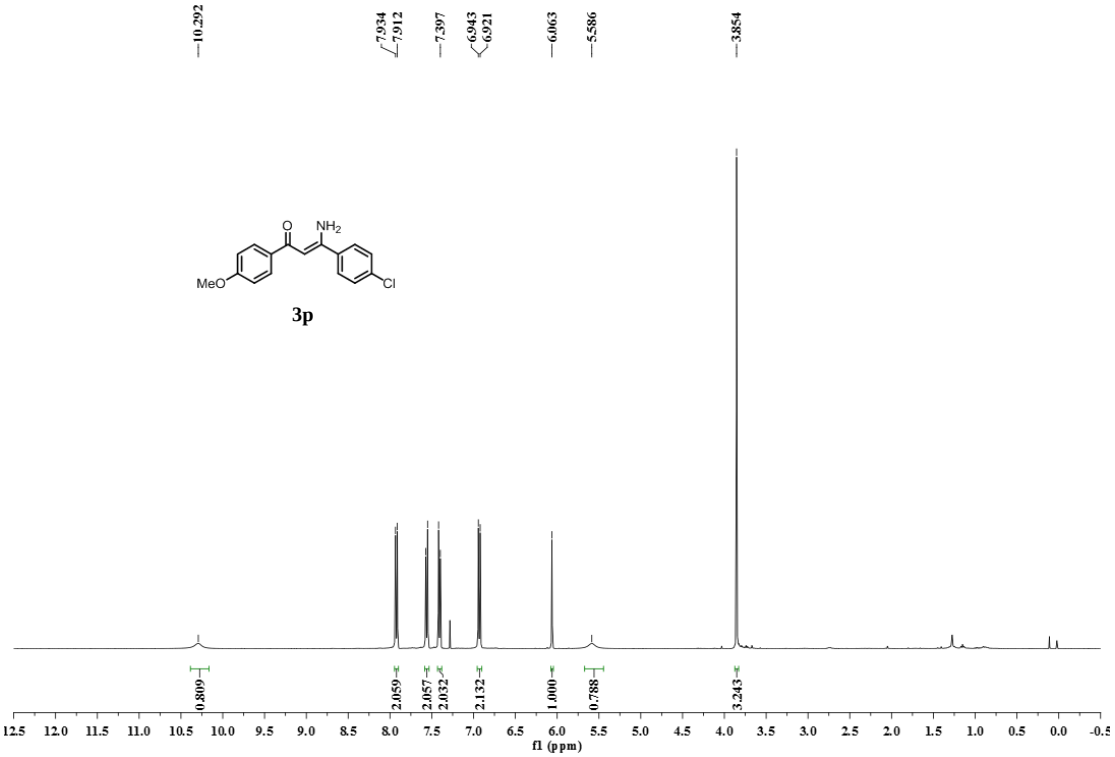

190.21
161.77
140.19
136.71
135.94
131.21
129.23
128.57
127.81
127.22
91.90
O
NH2
Cl
3n
f1 (ppm)


10.319
7.916
7.898
7.727
7.707
7.687
7.667
7.485
7.468
7.451
7.434
7.416
7.399
6.104
5.727
NH2
O
CF3
0.635
2.096
4.170
3.144
1.000
0.659
f1 (ppm)

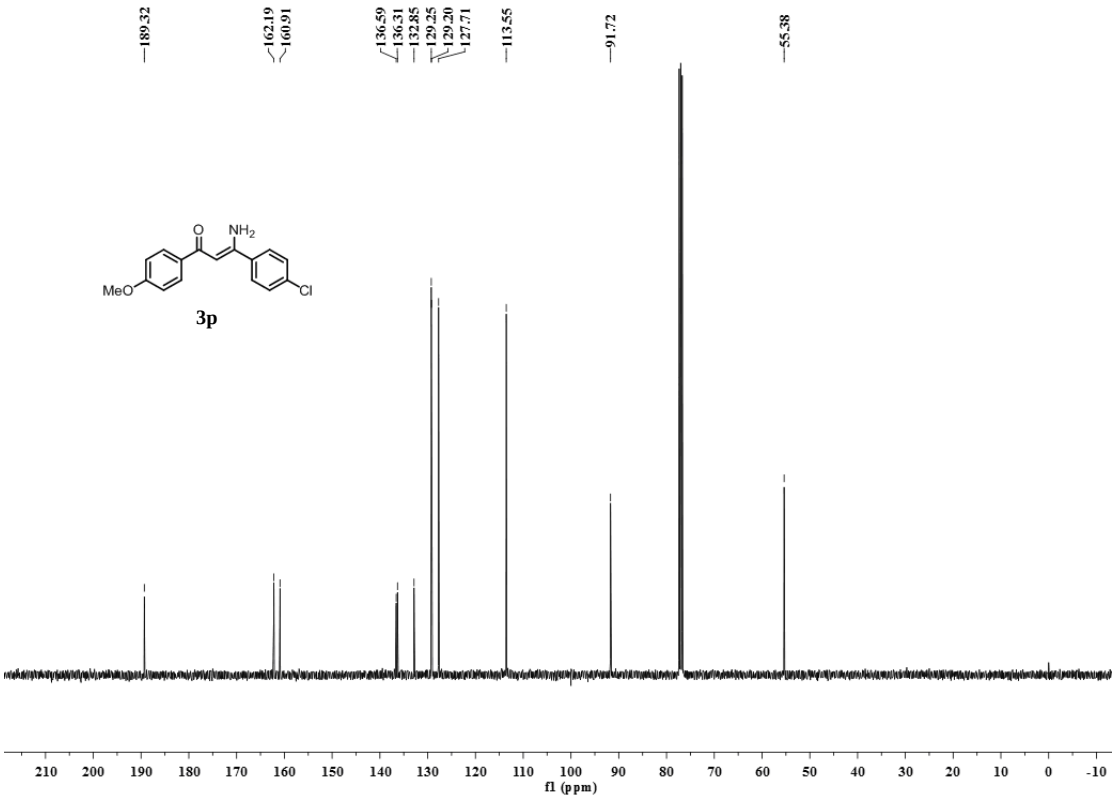
3o


190.49
161.46
141.06
140.03
132.25
131.93
131.42
128.46
127.28
127.05
126.01
125.16
122.45
119.74
92.44
NH2
O
CF3
f1 (ppm)

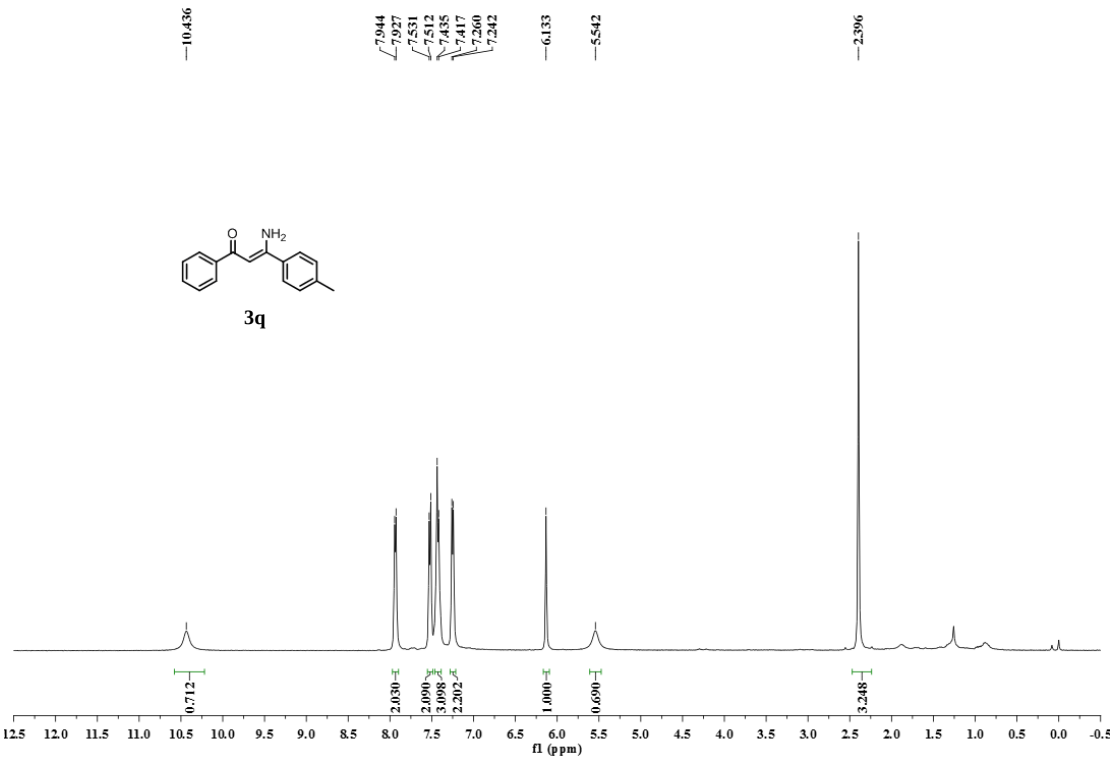
3o

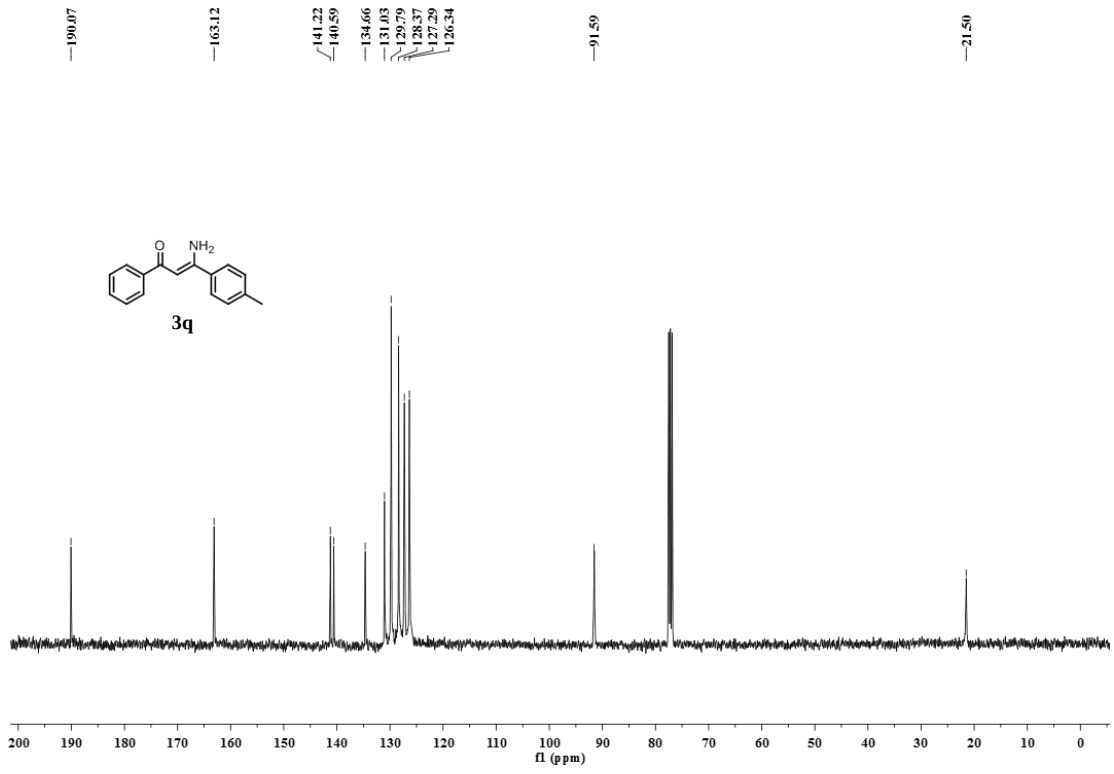

10.292
7.934
7.912
7.397
6.943
6.921
6.063
5.586
3.854
O
NH2
MeO
Cl
0.809
2.059
2.057
2.032
2.132
1.000
0.788
3.243
f1 (ppm)

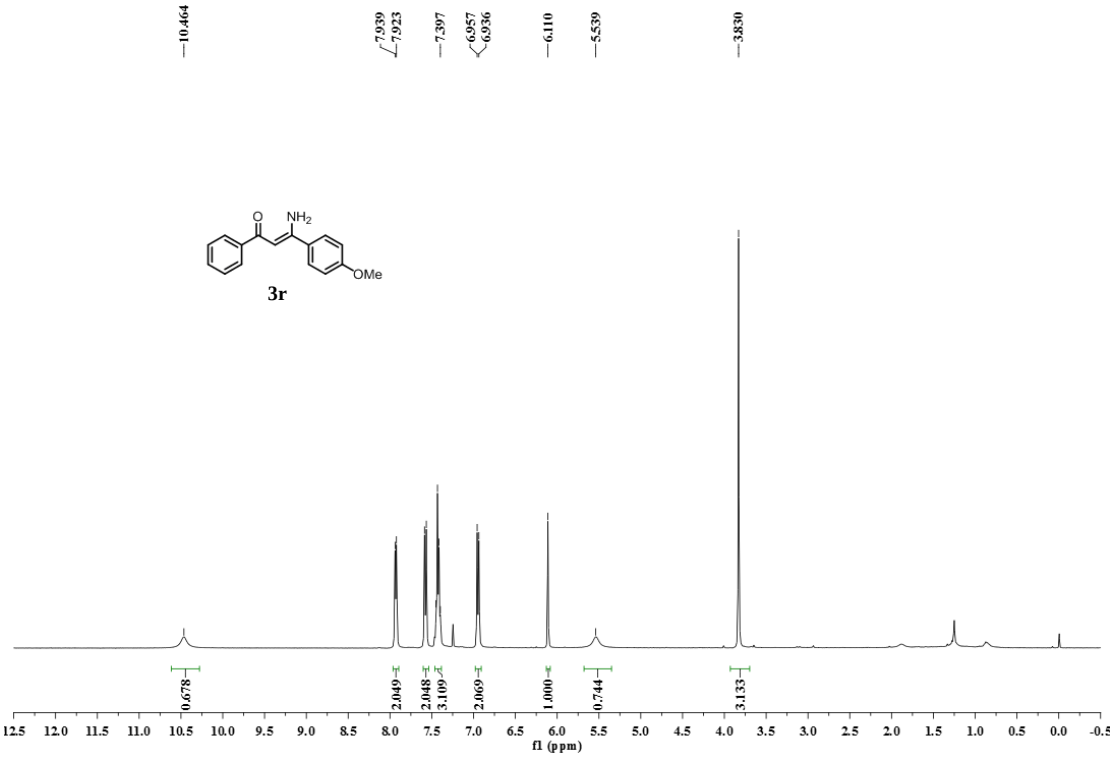
3p

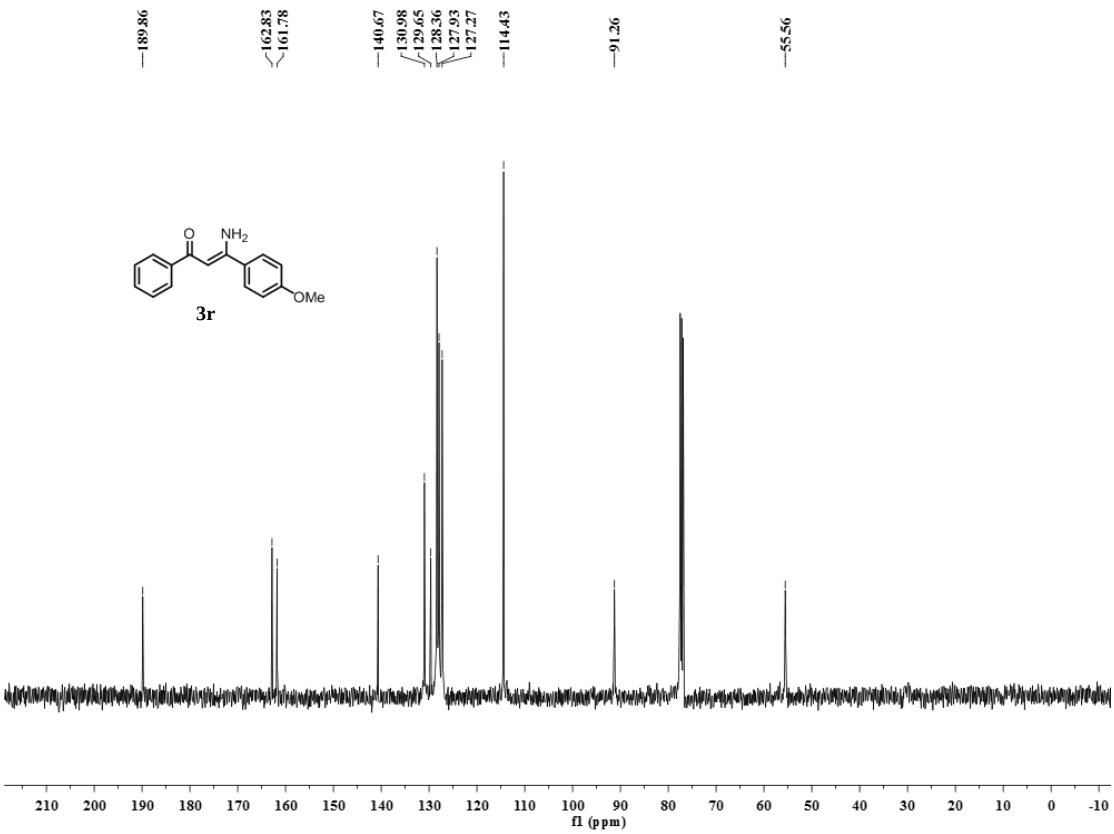

189.32
162.19
160.91
136.59
136.31
132.85
129.25
129.20
127.71
113.55
91.72
55.38
O
NH2
MeO
Cl
f1 (ppm)

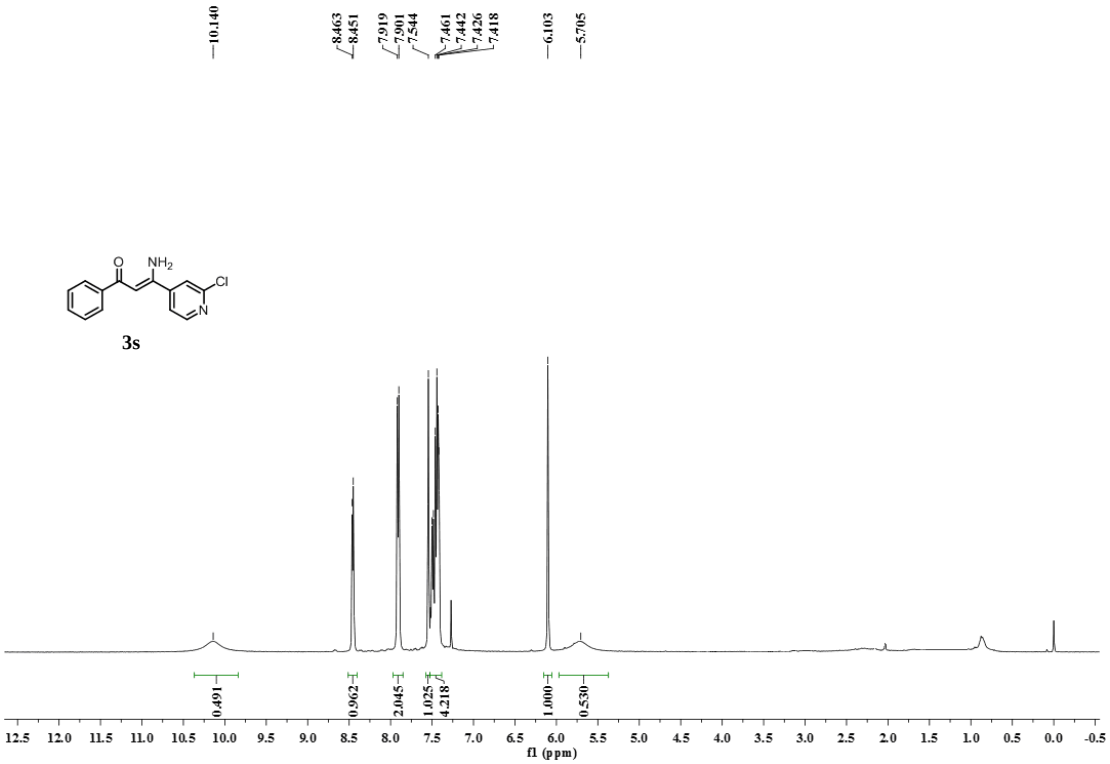
3p

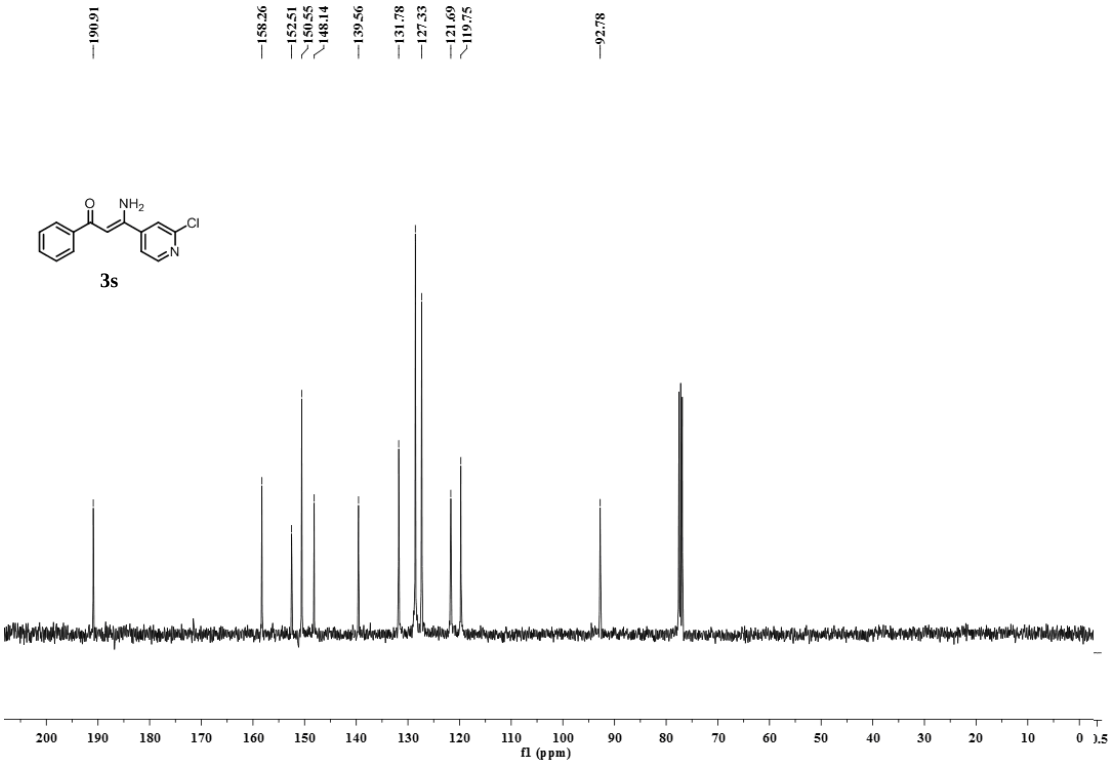

10.436
7.944
7.927
7.531
7.512
7.435
7.417
7.260
7.242
6.133
5.542
2.396
O
NH2
3q
0.712
2.030
2.090
3.098
2.202
1.000
0.690
3.248
f1 (ppm)

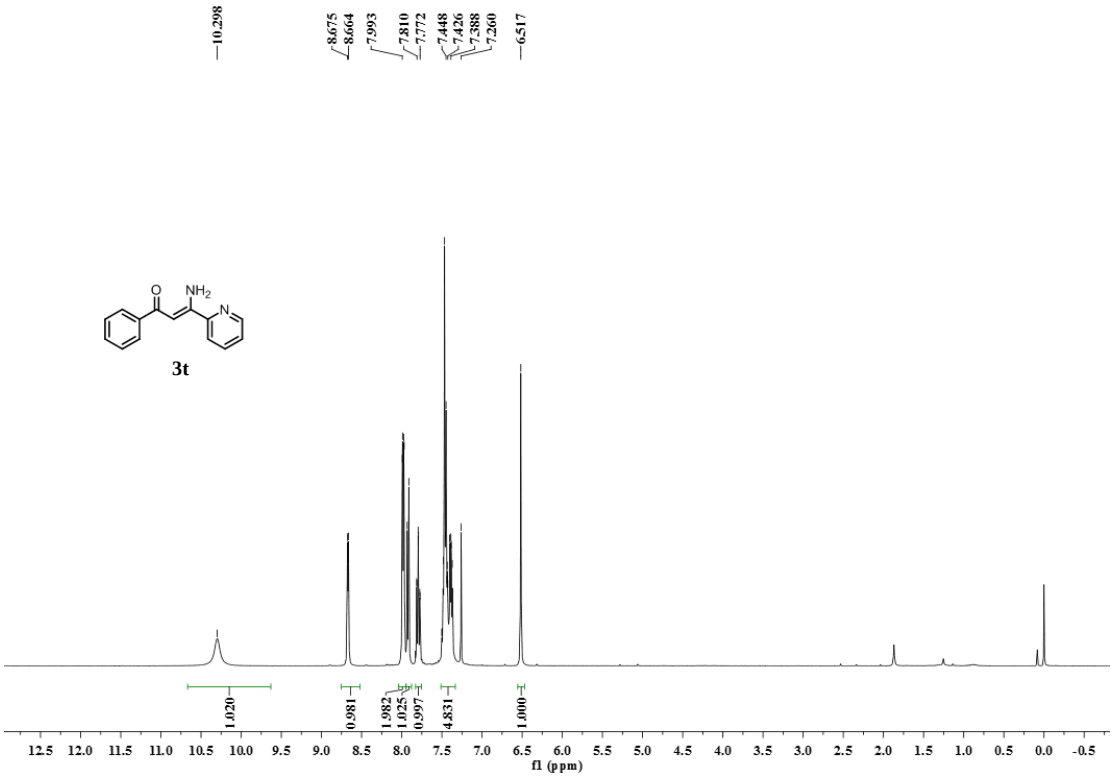

190.07
163.12
141.22
140.59
134.66
131.03
129.79
128.37
127.29
126.34
91.59
21.50
O
NH2
3q
f1 (ppm)

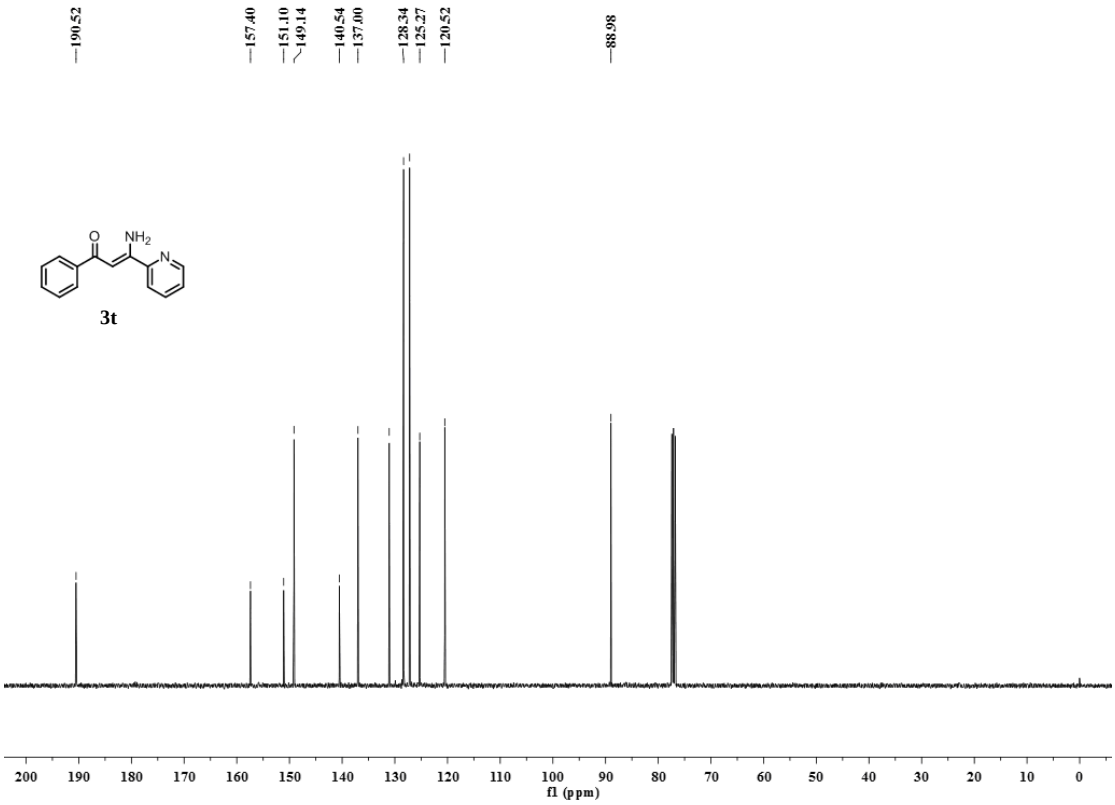

10.464
7.939
7.923
7.397
6.957
6.936
6.110
5.539
3.830
O
NH2
OMe
3r
0.678
2.049
2.048
3.109
2.069
1.000
0.744
3.133
f1 (ppm)

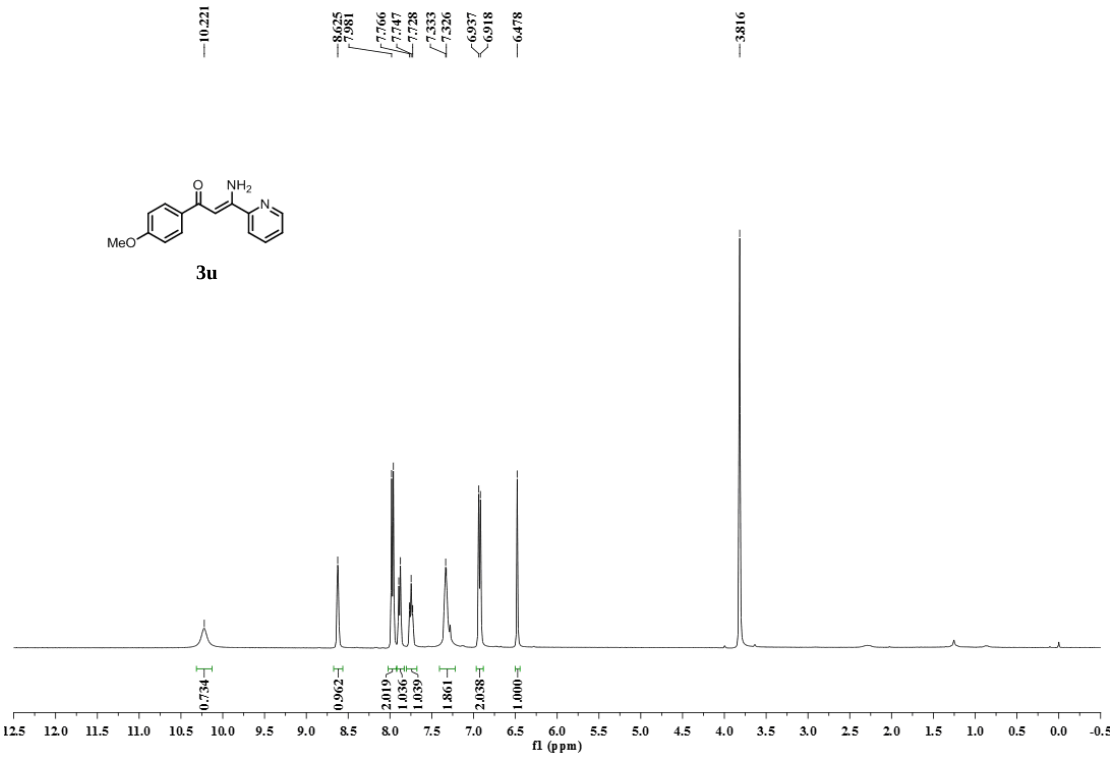

189.86
162.83
161.78
140.67
130.98
129.65
128.36
127.93
127.27
114.43
91.26
55.56
O
NH2
OMe
3r
f1 (ppm)


10.140
8.463
8.451
7.919
7.901
7.544
7.461
7.442
7.426
7.418
6.103
5.705
O
NH2
Cl
N
3s
0.491
0.962
2.045
1.025
4.218
1.000
0.530
f1 (ppm)

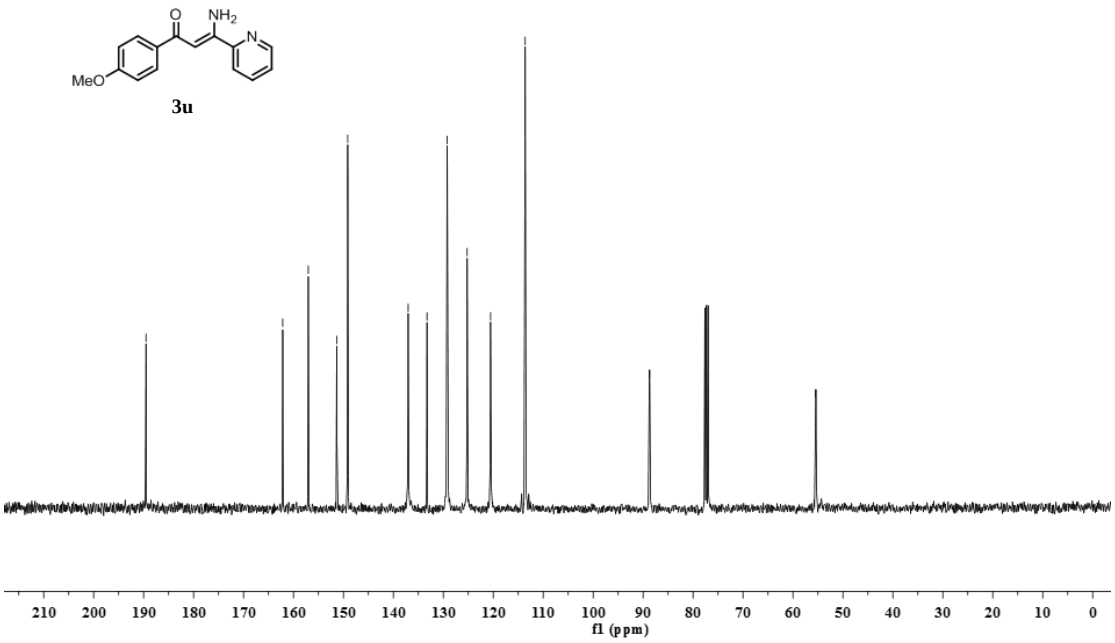

190.91
158.26
152.51
150.55
148.14
139.56
131.78
127.33
121.69
119.75
92.78
O
NH2
Cl
N
3s
f1 (ppm)


10.298
8.675
8.664
7.993
7.810
7.772
7.448
7.426
7.388
7.260
6.517
O
NH2
N
3t
1.020
0.981
1.982
1.025
0.997
4.831
1.000
f1 (ppm)


190.52
157.40
151.10
149.14
140.54
137.00
128.34
125.27
120.52
88.98
O
NH2
N
3t
f1 (ppm)


10.221
8.625
7.981
7.766
7.747
7.728
7.333
7.326
6.937
6.918
6.478
3.816
O
NH2
N
MeO
0.734
0.962
2.019
1.036
1.039
1.861
2.038
1.000
fl (ppm)

3u


189.52
162.16
157.03
151.33
149.14
137.04
133.27
129.22
125.23
120.54
113.61
88.75
55.47
O
NH2
N
MeO
fl (ppm)

3u