Synthesis of Hydrazide-Containing Chroman-2-ones and Dihydroquinolin-2-ones via Photocatalytic Radical Cascade Reaction of Aroylhydrozones

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Table of Contents

1. General information

^{1}H NMR spectra were recorded on 400 MHz spectrophotometers. Chemical shifts are reported in delta (δ (ppm) =) units in parts per million (ppm) relative to the singlet (0 ppm) for tetramethylsilane (TMS). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, dd = doublet of doublets, m = multiplet), coupling constants (Hz) and integration. ^{13}C NMR were recorded on Varian Mercury 400 (100 MHz) with complete proton decoupling spectrophotometers ($CDCl_3$: 77.0 ppm, DMSO-d_6: 39.5 ppm, $(CD_3)CO$: 2.05). HRMS was recorded on Bruker ultrafleXtreme MALDI-TOF/TOF mass spectrometer. UV-vis absorption spectrum was taken using a U-3310 spectrophotometer. Unless otherwise noted, materials were purchased from commercial suppliers and used without further purification. All the solvents were treated according to general methods. Flash column chromatography was performed using 200-300 mesh silica gel.

2. General synthetic procedure and spectral data of substrates 1

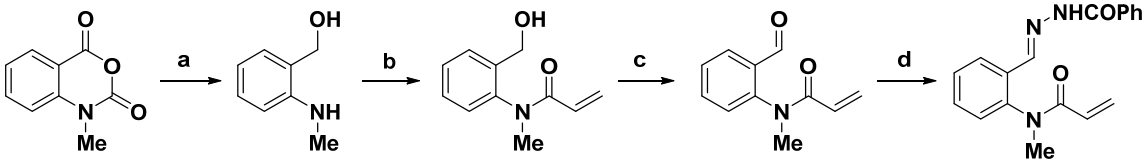
2.1 General procedure of preparation for acyloyl ester 1a-1j

Step 1: To a stirring solution of salicylaldehyde (10 mmol, 1.0 equiv) in 15 mL of a mixture solvent of MeOH/Et_2O (1:1), benzoyl hydrazine (11 mmol, 1.1 equiv) was added. Then this mixture solution was stirred at room temperature for overnight. And then the formed white solid was filtered, washed with a mixture solution (PE/EA = 10:1), dried to afford phenol as a white solid in a quantitative yield.

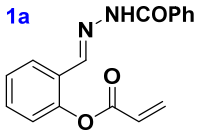
Step 2: To a stirring solution of phenol (10 mmol, 1.0 equiv) in anhydrous dichloromethane (70 mL), was added triethylamine (15 mmol, 1.5 eauiv) in an ice-water bath under Ar atmosphere. And then acryloyl chloride (15 mmol, 1.5 equiv) was added dropwise at the same tempeture. Before the reaction completion, the reaction was monitored by TLC. Solvent was removed by vacuum. The resulting residue was extracted with ethyl acetate (70 mLX3 times) and water (70 mL), washed with brine (100 mL). The organic phase was dried with anhydrous Na_2SO_4 and concentrated. The crude product was purified by column chromatography (PE/ EA (3:1) to EA), followed by recrystallization to give the desired products as a white solid.

2.2 General procedure of preparation for acyloyl amide 1k

This substrate was synthetized in accordance with the known literatures.[1]

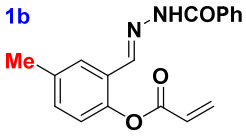


Scheme 1. Reagents and condition: (a) $LiAlH_4$, THF, 91% yield; (b) acyloyl chloride, CH_2Cl_2, $NaHCO_3$, 83% yield; (c) IBX, DMSO, 90% yield; (d) benzoyl hydrazine , MeOH/ Et_2O (1: 1), overnight, 89% yield.



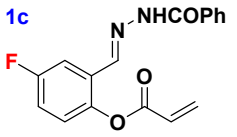
White solid, 1.94 g, 66% yield, M.P: 152.8-154.0.

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 9.66 (s, 1H), 8.34 (s, 1H), 8.09 – 8.08 (m, 1H), 7.88 – 7.81 (m, 2H), 7.50 (t, *J* = 7.3 Hz, 1H), 7.42 – 7.35 (m, 3H), 7.25 – 7.21 (m, 1H), 7.09 (d, *J* = 8.1 Hz, 1H), 6.59 (d, *J* = 17.2 Hz, 1H), 6.34 – 6.27 (m, 1H), 5.99 (d, *J* = 10.4 Hz, 1H). ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm) = 164.2, 163.2, 149.0, 141.8, 134.4, 133.3, 131.9, 131.2, 128.5, 127.7, 127.2, 126.8, 126.6, 126.2, 123.2. HRMS (ESI): m/z $[M - H]^-$ calcd for $C_{17}H_{14}N_2O_3$: 293.0932; found: 293.0934.



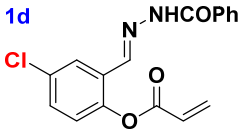
White solid, 1.82 g, 55% yield, M.P: 181.4-183.5.

^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) = 9.62 (s, 1H), 8.29 (s, 1H), 7.95 (s, 1H), 7.85 (s, 2H), 7.53 (t, *J* = 7.4 Hz, 1H), 7.44 (t, *J* = 7.6 Hz, 2H), 7.19 (d, *J* = 8.3 Hz, 1H), 6.99 (d, *J* = 8.3 Hz, 1H), 6.61 (d, *J* = 17.3 Hz, 1H), 6.30 (d, *J* = 15.5 Hz, 1H), 6.01 (d, *J* = 10.5 Hz, 1H), 2.33 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm) = 164.7, 163.6, 147.4, 142.2, 136.3, 134.6, 133.7, 132.3, 132.2, 128.9, 128.1, 127.7, 126.7, 126.5, 123.3, 20.9. HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{18}H_{16}N_2O_3$: 331.1053; found: 331.1046.



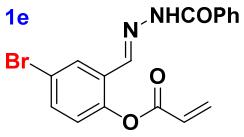
White solid, 1.62 g, 52% yield, M.P: 158.7-161.2.

^{1}H NMR (600 MHz, DMSO-d_6) δ (ppm) = 12.02 (s, 1H), 8.46 (s, 1H), 7.89 (d, *J* = 7.0 Hz, 2H), 7.68 (d, *J* = 8.8 Hz, 1H), 7.60 (t, *J* = 7.0 Hz, 1H), 7.52 (t, *J* = 6.6 Hz, 2H), 7.36 (s, 2H), 6.62 (d, *J* = 17.0 Hz, 1H), 6.49 (dd, *J* = 16.7, 10.6 Hz, 1H), 6.25 (d, *J* = 9.8 Hz, 1H). ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm) = 163.7, 162.8, 160.5, 158.1, 144.6, 140.1, 134.1, 132.6, 131.5, 128.2, 128.2, 128.0, 127.2, 126.5, 124.9, 124.9, 117.6, 117.3, 111.2, 111.0. HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{17}H_{13}N_2O_3F$: 313.0983; found: 313.1002.



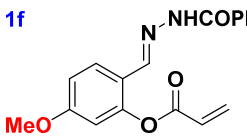
White solid, 2.39 g, 73% yield, M.P: 174.4-176.1.

^{1}H NMR (600 MHz, DMSO-d_6) δ (ppm) = 12.05 (s, 1H), 8.46 (s, 1H), 7.97 (s, 1H), 7.91 (d, *J* = 6.8 Hz, 2H), 7.64 – 7.54 (m, 5H), 7.36 (d, *J* = 8.4 Hz, 1H), 6.64 (d, *J* = 17.2 Hz, 1H), 6.50 (dd, *J* = 17.0, 10.5 Hz, 1H), 6.27 (d, *J* = 10.2 Hz, 1H). ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm) = 163.4, 162.8, 147.0, 139.7, 134.2, 132.6, 131.5, 130.4, 130.1, 128.1, 128.0, 127.2, 126.4, 124.9, 124.7. HRMS (ESI): m/z $[M + K]^+$ calcd for $C_{17}H_{19}O_2Cl$: 329.0705; found: 329.0702.



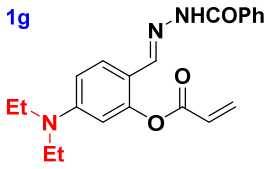
White solid, 1.68 g, 45% yield, M.P: 178.1-180.7.

^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) = 10.33 (s, 1H), 8.43 (s, 1H), 8.16 (s, 1H), 7.86 (d, *J* = 7.5 Hz, 2H), 7.49 – 7.48 (m, 4H), 6.98 (d, *J* = 8.7 Hz, 1H), 6.56 (d, *J* = 17.3 Hz, 1H), 6.29 – 6.15 (m, 1H), 5.94 (d, *J* = 10.5 Hz, 1H). ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm) = 164.3, 163.7, 148.4, 140.5, 135.2, 133.9, 133.5, 132.4, 129.4, 129.0, 128.6, 128.1, 127.4, 126.1, 119.5. HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{17}H_{13}N_2O_3Br$: 373.0182; found: 373.0208.



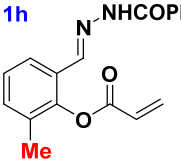
White solid, 1.98 g, 61% yield, M.P: 169.9-171.8.

^{1}H NMR (600 MHz, DMSO-d_6) δ (ppm) = 11.79 (s, 1H), 8.41 (s, 1H), 7.92 (d, J = 8.6 Hz, 1H), 7.88 (d, J = 7.1 Hz, 2H), 7.59 (t, J = 6.6 Hz, 1H), 7.52 (t, J = 7.0 Hz, 2H), 7.00 (d, J = 8.4 Hz, 1H), 6.88 (s, 1H), 6.61 (d, J = 17.2 Hz, 1H), 6.49 (dd, J = 17.4, 10.2 Hz, 1H), 6.24 (d, J = 10.4 Hz, 1H), 3.82 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm) = 163.5, 162.5, 161.0, 149.8, 141.4, 133.8, 133.0, 131.2, 128.0, 127.1, 126.8, 118.8, 112.8, 107.8, 55.3. HRMS (ESI): m/z [M + H]$^+$ calcd for $C_{18}H_{16}N_2O_4$: 325.1183; found: 325.1196.



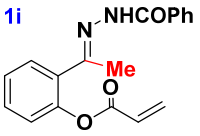
White solid, 1.94 g, 53% yield, M.P: 183.4-185.4.

^{1}H NMR (600 MHz, DMSO-d_6) δ (ppm) = 11.59 (s, 1H), 8.30 (s, 1H), 7.86 (d, J = 7.3 Hz, 2H), 7.77 (d, J = 9.1 Hz, 1H), 7.56 (t, J = 6.8 Hz, 1H), 7.50 (t, J = 7.2 Hz, 2H), 6.69 (d, J = 8.6 Hz, 1H), 6.58 (d, J = 16.9 Hz, 1H), 6.47 (dd, J = 16.8, 10.4 Hz, 1H), 6.40 (s, 1H), 6.19 (d, J = 10.4 Hz, 1H), 3.38 (d, J = 6.7 Hz, 4H), 1.10 (t, J = 6.4 Hz, 6H). ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm) = 163.7, 162.2, 150.4, 149.2, 142.2, 133.2, 131.0, 127.9, 127.1, 127.0, 126.7, 112.2, 109.2, 103.7, 59.3, 43.4, 20.3, 13.6, 11.9. HRMS (ESI): m/z [M + Na]$^+$ calcd for $C_{21}H_{23}N_3O_3$: 388.1632; found: 388.1625.



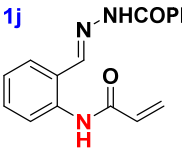
White solid, 1.85 g, 60% yield, M.P: 166.0-167.3.

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 10.63 (s, 1H), 8.37 (s, 1H), 7.83 (d, J = 7.5 Hz, 3H), 7.43 – 7.29 (m, 3H), 7.12 – 6.99 (m, 2H), 6.50 (d, J = 17.1 Hz, 1H), 6.21 (dd, J = 17.2, 10.4 Hz, 1H), 5.87 (d, J = 10.5 Hz, 1H), 2.05 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm) = 163.4, 162.8, 147.4, 141.8, 134.3, 133.1, 132.2, 131.5, 130.6, 128.2, 127.4, 126.6, 126.1, 123.7, 15.6. HRMS (ESI): m/z [M + Na]$^+$ calcd for $C_{18}H_{16}N_2O_3$: 331.1053, found: 331.1050.



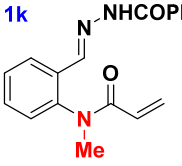
White solid, 1.66 g, 54% yield, M.P: 136.3-137.8.

1H NMR (600 MHz, DMSO-d_6) δ (ppm) = 10.71 (s, 1H), 7.86 (s, 2H), 7.57 (s, 2H), 7.49 (q, J = 8.4 Hz, 3H), 7.36 (d, J = 8.3 Hz, 1H), 7.24 (d, J = 8.1 Hz, 1H), 6.52 (d, J = 17.2 Hz, 1H), 6.40 (s, 1H), 6.14 (d, J = 10.4 Hz, 1H), 2.27 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm) = 164.0, 147.7, 134.2, 134.0, 133.8, 132.8, 131.6, 130.9, 130.0, 129.5, 128.9, 128.3, 128.0, 127.5, 127.1, 126.0, 123.3, 24.4, 17.6. HRMS (ESI): m/z [M + H]$^+$ calcd for $C_{18}H_{16}N_2O_3$: 309.1234; found: 309.1253.



White solid, 1.45 g, 46% yield, M.P: 185.6-188.0.

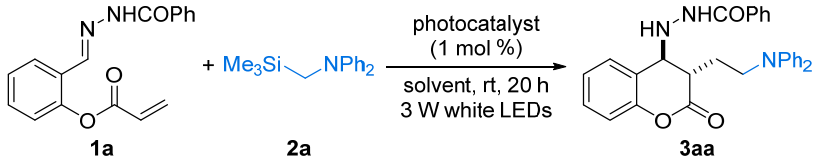
^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 12.07 (s, 1H), 10.18 (s, 1H), 8.74 (d, J = 8.3 Hz, 1H), 8.24 (s, 1H), 7.88 (d, J = 7.4 Hz, 2H), 7.51 (t, J = 7.3 Hz, 1H), 7.42 (t, J = 7.5 Hz, 2H), 7.28 (t, J = 7.8 Hz, 1H), 7.14 (d, J = 7.4 Hz, 1H), 7.01 (t, J = 7.4 Hz, 1H), 6.72 (dd, J = 16.8, 10.2 Hz, 1H), 6.47 (d, J = 16.8 Hz, 1H), 5.79 (d, J = 10.1 Hz, 1H). ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm) = 163.2, 162.8, 148.4, 137.5, 132.1, 131.9, 131.7, 131.6, 130.1, 128.1, 127.4, 127.3, 122.9, 120.1, 119.3. HRMS (ESI): m/z [M + H]$^+$ calcd for $C_{17}H_{15}N_3O_2$: 316.1056; found: 316.1056.



White solid, M.P: 176.7-178.0.

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 11.42 (s, 1H), 8.50 (s, 1H), 8.32 (s, 1H), 7.93 (d, *J* = 7.9 Hz, 2H), 7.50 –7.36 (m, 5H), 7.13 – 7.11 (m, 1H), 6.18 (d, *J* = 16.7 Hz, 1H), 5.89 (dd, *J* = 16.7, 10.3 Hz, 1H), 5.43 (d, *J* = 10.0 Hz, 1H), 3.24 (s, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ (ppm) = 166.0, 164.4, 143.1, 141.5, 132.8, 131.7, 131.2, 128.9, 128.6, 128.2, 127.9, 127.6, 127.4, 127.3, 37.5. HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{18}H_{17}N_3O_2$: 308.1394; found: 308.1406.

3. Condition optimization[a]

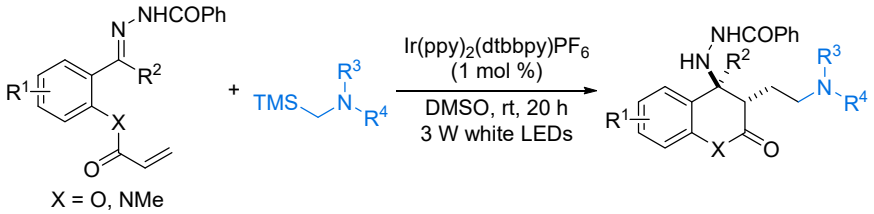


entry	photocatalyst	**1a**/**2a**	solvent	yield[b] (%)
1	$Ir(ppy)_2(dtbbpy)PF_6$	1/1.2	DMF	63
2	$Ru(bpy)_3Cl_2{\cdot}6H_2O$	1/1.2	DMF	58
3	*fac*-$Ir(ppy)_3$	1/1.2	DMF	0
4	$Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$	1/1.2	DMF	55
5	RB	1/1.2	DMF	0
6	$Ir(ppy)_2(dtbbpy)PF_6$	1/1.5	DMF	72
7	$Ir(ppy)_2(dtbbpy)PF_6$	1/1.5	DMSO	76
8	$Ir(ppy)_2(dtbbpy)PF_6$	1/1.5	MeCN	40
9	$Ir(ppy)_2(dtbbpy)PF_6$	1/1.5	DMA	68
10	$Ir(ppy)_2(dtbbpy)PF_6$	1/1.5	THF	44
11	$Ir(ppy)_2(dtbbpy)PF_6$	1/1.5	CH_2Cl_2	13
12[c]	$Ir(ppy)_2(dtbbpy)PF_6$	1/1.5	DMSO	67
13[d]	-	1/1.5	DMSO	0
14[e]	$Ir(ppy)_2(dtbbpy)PF_6$	1/1.5	DMSO	0

[a] Reaction conditions: **1a** (0.20 mmol), **2a** (0.24 or 0.30 mmol), photocatalyst (1 mol %), 2.0 mL of solvent, 20 h, 3 W white LEDs and rt. [b] Isolated yield. [c] 10 equiv of H_2O was added. [d] Without photocatalyst. [e] Without visible light irradiation.

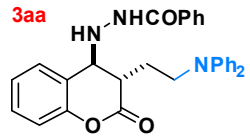
4. Photocatalytic radical cascade reaction

4.1 General procedure and spectral data of the synthesis of 3aa-3kg



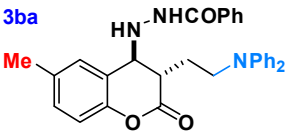
To a dried schlenk tube was treated with **1** (0.20 mmol, 1.0 equiv), **2** (0.30 mmol, 1.5 equiv), photocatalyst (0.002 mmol, 1 mol%) and DMSO (2.0 mL). And then this mixture solution was degassed for 3 times via 'freeze-pump-thaw' procedure. After that, this resulting solution was stirred at a distance of ~5 cm under irradiation by 3 W white LEDs at room temperature for 20 h. The mixture was diluted with ethyl acetate (4 mL), and added saturated $NaHCO_3$ aquous (8 mL), followed by extration with ethyl acetate (10 mLX3 times). The combined organic phase was washed with brine (10 mL), dried with

anhydrous Na_2SO_4 and the solvent was evaporated. The crude product was purified by column chromatography using petroleum ether/ethyl acetate as eluent (5:1 to 3:1), affording the desired product.



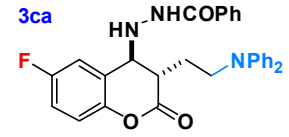
White solid, 72 mg，76% yield, M.P: 185.3-187.1.

^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) = 7.93 (s, 1H), 7.73 (d, *J* = 7.7 Hz, 2H), 7.54 (t, *J* = 7.7 Hz, 1H), 7.43 (t, *J* = 7.7 Hz, 2H), 7.37 (t, *J* = 9.2 Hz, 2H), 7.16 (d, *J* = 15.2 Hz, 5H), 7.08 (d, *J* = 8.0 Hz, 1H), 6.89 (t, *J* = 7.5 Hz, 2H), 6.84 (d, *J* = 8.1 Hz, 4H), 4.88 (br, 1H), 4.28 (s, 1H), 3.79 (t, *J* = 7.6 Hz, 2H), 3.19 (t, *J* = 7.7 Hz, 1H), 1.73 (q, *J* = 7.8 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) = 169.5, 168.3, 151.4, 147.3, 132.2, 132.0, 130.6, 130.2, 129.3, 128.7, 127.0, 124.8, 121.6, 120.8, 119.7, 116.7, 60.0, 49.4, 42.2, 25.8. HRMS (ESI): m/z $[M - H]^-$ calcd for $C_{30}H_{27}N_3O_3$: 476.1980; found: 476.2017.



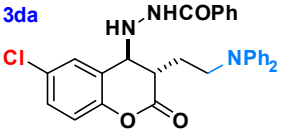
White solid, 70 mg, 71% yield, M.P:184.0-186.0.

^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) = 7.72 (t, *J* = 7.6 Hz, 3H), 7.56 (t, *J* = 7.3 Hz, 1H), 7.46 (t, *J* = 7.5 Hz, 2H), 7.17 (t, *J* = 7.2 Hz, 6H), 6.99 (d, *J* = 8.2 Hz, 1H), 6.90 (t, *J* = 7.2 Hz, 2H), 6.85 (d, *J* = 8.0 Hz, 4H), 4.83 (d, *J* = 4.2 Hz, 1H), 4.23 (s, 1H), 3.80 (t, *J* = 7.1 Hz, 2H), 3.15 (t, *J* = 6.4 Hz, 1H), 2.33 (s, 3H), 1.73 (q, *J* = 7.4 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) = 170.0, 168.4, 149.3, 147.4, 134.6, 132.2, 132.1, 131.1, 130.6, 129.4, 128.8, 127.2, 121.6, 120.9, 119.4, 116.4, 60.3, 49.4, 42.3, 25.8, 20.7. HRMS (ESI): m/z $[M + Na]^+$ calcd for $C_{31}H_{29}N_3O_3$: 514.2101; found: 514.2106.



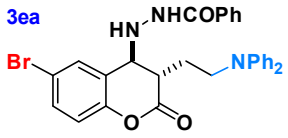
White solid, 61 mg, 62% yield, M.P: 169.9-171.4.

^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) = 7.88 (s, 1H), 7.73 (d, *J* = 7.4 Hz, 2H), 7.55 (t, *J* = 7.2 Hz, 1H), 7.44 (t, *J* = 7.4 Hz, 2H), 7.18 (t, *J* = 7.5 Hz, 4H), 7.10 – 7.05 (m, 3H), 6.91 (t, *J* = 7.2 Hz, 2H), 6.86 (d, *J* = 7.8 Hz, 4H), 4.87 (br, 1H), 4.24 (s, 1H), 3.80 (t, *J* = 7.2 Hz, 2H), 3.18 (t, *J* = 6.5 Hz, 1H), 1.72 (q, *J* = 6.9 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) = 169.2, 168.5, 160.2, 157.8, 147.4 (d, *J* = 2.7 Hz), 147.3, 132.4, 131.9, 129.4, 128.8, 127.1, 121.8, 121.4 (d, *J* = 7.8 Hz), 121.0, 118.1 (d, *J* = 8.2 Hz), 117.03 (dd, *J* = 44.8, 23.6 Hz), 60.0, 49.4, 41.9, 25.8. HRMS (ESI): m/z $[M + Na]^+$ calcd for $C_{30}H_{26}N_3O_3F$: 518.1850; found: 518.1846.



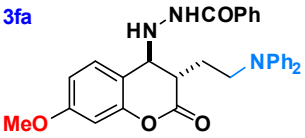
White solid, 72 mg, 70% yield, M.P: 177.9-179.3.

^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) = 7.88 (s, 1H), 7.73 (d, *J* = 7.6 Hz, 2H), 7.55 (t, *J* = 7.5 Hz, 1H), 7.44 (t, *J* = 7.7 Hz, 2H), 7.18 (t, *J* = 7.8 Hz, 4H), 7.10 (d, *J* = 7.6 Hz, 1H), 7.06 (t, *J* = 5.3 Hz, 2H), 6.91 (t, *J* = 7.3 Hz, 2H), 6.86 (d, *J* = 8.0 Hz, 4H), 4.88 (br, 1H), 4.24 (s, 1H), 3.80 (t, *J* = 7.6 Hz, 2H), 3.18 (t, *J* = 7.5 Hz, 1H), 1.72 (q, *J* = 7.7 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) = 169.0, 168.5, 150.0, 147.3, 132.4, 131.9, 130.6, 130.1, 129.8, 129.4, 128.8, 127.1, 121.8, 121.5, 121.0, 118.1, 59.8, 49.3, 41.9, 25.8. HRMS (ESI): m/z $[M + Na]^+$ calcd for $C_{30}H_{26}N_3O_3Cl$: 534.1555; found: 534.1551.



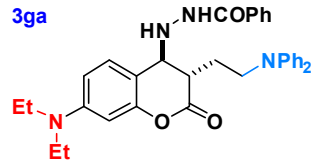
White solid, 67 mg, 60% yield, M.P: 165.4-167.1.

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 7.72 – 7.68 (m, 3H), 7.55 – 7.40 (m, 5H), 7.17 – 7.13 (m, 4H), 6.94 (d, *J* = 8.5 Hz, 1H), 6.89 (t, *J* = 7.3 Hz, 2H), 6.83 (d, *J* = 8.0 Hz, 4H), 4.83 (br, 1H), 4.23 (d, *J* = 2.3 Hz, 1H), 3.78 (t, *J* = 7.5 Hz, 2H), 3.17 (td, *J* = 7.4, 2.3 Hz, 1H), 1.71 (q, *J* = 7.4 Hz, 2H).^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) = 168.9, 168.4, 150.4, 147.2, 133.4, 132.9, 132.3, 131.8, 129.3, 128.7, 127.0, 121.8, 121.7, 120.9, 118.4, 117.1, 59.7, 49.2, 41.8, 25.7. HRMS (ESI): m/z $[M + Na]^+$ calcd for $C_{30}H_{26}N_3O_3Br$: 578.1050; found: 578.1051.



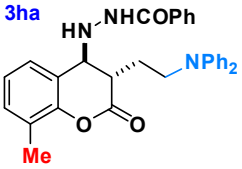
White solid, 61 mg, 60% yield, M.P: 177.9-179.2.

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 7.71 – 7.68 (m, 3H), 7.52 (t, *J* = 7.1 Hz, 1H), 7.42 (t, *J* = 7.4 Hz, 2H), 7.23 (s, 2H), 7.15 (t, *J* = 7.6 Hz, 4H), 6.90 – 6.82 (m, 7H), 6.67 (d, *J* = 8.4 Hz, 1H), 6.61 (s, 1H), 4.21 (s, 1H), 3.79 – 3.77 (m, 5H), 3.13 (t, *J* = 6.8 Hz, 1H), 1.73 (q, *J* = 7.3 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) = 169.2, 168.0, 161.0, 152.1, 147.1, 132.0, 131.9, 130.6, 129.1, 128.5, 126.9, 121.4, 120.7, 111.6, 110.5, 102.3, 59.7, 55.6, 49.5, 42.5, 26.1. HRMS (ESI): m/z $[M - H]^-$ calcd for $C_{31}H_{29}N_3O_4$: 506.2085; found: 506.2122.



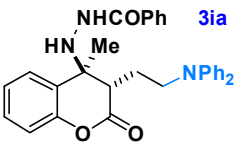
White solid, 62 mg, 57% yield, M.P: 150.1-152.0.

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 7.78 (s, 1H), 7.70 (d, *J* = 7.6 Hz, 2H), 7.49 (t, *J* = 7.3 Hz, 1H), 7.40 (t, *J* = 7.6 Hz, 2H), 7.16 – 7.12 (m, 4H), 6.88 – 6.82 (m, 5H), 6.39 – 6.36 (m, 1H), 6.31 (d, *J* = 2.6 Hz, 1H), 4.79 (s, 1H), 4.14 (s, 1H), 3.78 (t, *J* = 7.7 Hz, 2H), 3.32 (q, *J* = 7.0 Hz, 4H), 3.10 – 3.07 (m, 1H), 1.79 – 1.71 (m, 2H), 1.16 (t, *J* = 7.1 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) = 170.1, 167.8, 152.5, 149.3, 147.1, 132.1, 131.8, 130.5, 129.1, 128.4, 126.9, 121.3, 120.7, 107.4, 105.4, 98.8, 59.8, 49.5, 44.5, 42.8, 26.2, 12.6. HRMS (MALD1): m/z $[M + H]^+$ calcd for $C_{34}H_{36}N_4O_3$: 549.2860, found: 549.2886.



White solid, 75 mg, 76% yield, M.P: 154.1-155.8.

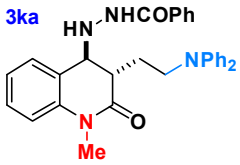
^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 8.00 (s, 1H), 7.77 – 7.66 (m, 2H), 7.49 (t, *J* = 7.4 Hz, 1H), 7.39 (t, *J* = 7.6 Hz, 2H), 7.20 – 7.06 (m, 6H), 7.01 (t, *J* = 7.4 Hz, 1H), 6.91 – 6.74 (m, 6H), 4.85 (s, 1H), 4.25 (d, *J* = 2.3 Hz, 1H), 3.87 – 3.67 (m, 2H), 3.17 (td, *J* = 7.5, 2.3 Hz, 1H), 2.28 (s, 3H), 1.71 (q, *J* = 7.5 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) = 169.7, 168.2, 149.6, 147.3, 132.2, 132.1, 132.0, 129.3, 128.7, 127.7, 127.0, 126.0, 124.3, 121.6, 120.8, 119.4, 60.2, 49.4, 42.1, 25.7, 15.5. HRMS (ESI): m/z $[M + Na]^+$ calcd for $C_{31}H_{29}N_3O_3$: 514.2101, found: 514.2114.



White solid, 60 mg, 61% yield, M.P: 155.3-157.1.

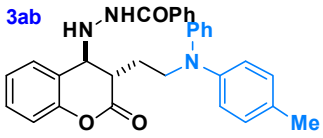
^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) = 7.58 (d, *J* = 7.4 Hz, 2H), 7.49 (t, *J* = 7.2 Hz, 1H), 7.45 (d, *J* = 7.3 Hz, 1H), 7.39 – 7.34 (m, 3H), 7.22 – 7.16 (m, 5H), 7.10 (d, *J* = 7.9 Hz, 1H), 6.98 (d, *J* = 7.7 Hz, 3H), 6.91 (t, *J* = 7.1 Hz, 2H), 5.35 (br, 1H), 3.95 – 3.91 (m, 1H), 3.80 – 3.74 (m, 1H), 2.96 (d, *J* = 10.6 Hz, 1H), 2.07 – 2.02 (m, 1H), 1.76 – 1.74 (m, 1H), 1.39 (s, 3H). ^{13}C NMR

(100 MHz, $CDCl_3$) δ (ppm) = 169.9, 167.6, 150.7, 147.5, 132.1, 131.9, 130.0, 129.3, 128.6, 126.9, 126.1, 125.3, 124.8, 121.5, 121.1, 121.0, 116.9, 61.7, 50.4, 46.4, 23.7, 20.0. HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{31}H_{29}N_3O_3$: 492.2282; found: 492.2282.



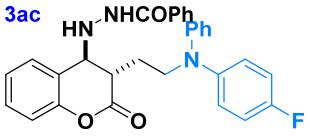
White solid, 81 mg, 83% yield, M.P: 174.3-175.9.

^1H NMR (400 MHz, $CDCl_3$) δ (ppm) = 8.07 (s, 1H), 7.73 – 7.71 (m, 2H), 7.51 – 7.47 (m, 1H), 7.41 – 7.32 (m, 4H), 7.14 – 7.09 (m, 4H), 7.08 – 7.04 (m, 1H), 7.01 – 6.98 (m, 1H), 6.86 – 6.82 (m, 2H), 6.81 – 6.78 (m, 4H), 4.14 (d, *J* = 2.0 Hz, 1H), 3.77 (td, *J* = 7.1, 1.9 Hz, 2H), 3.37 (s, 3H), 2.98 (td, *J* = 7.4, 2.1 Hz, 1H), 1.67 (q, *J* = 7.6 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) = 171.1, 167.5, 147.1, 139.4, 132.1, 131.8, 130.3, 129.6, 129.0, 128.4, 126.9, 123.1, 122.2, 121.1, 120.6, 114.6, 61.0, 49.6, 43.5, 29.5, 26.5. HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{31}H_{30}N_4O_2$: 491.2442; found: 491.2458.



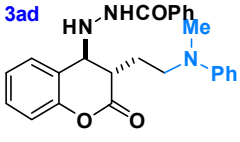
White solid, 75 mg, 76% yield, M.P.: 146.0-147.7.

^1H NMR (600 MHz, $CDCl_3$) δ (ppm) = 8.07 (s, 1H), 7.74 (d, *J* = 7.4 Hz, 2H), 7.52 (t, *J* = 7.2 Hz, 1H), 7.42 – 7.34 (m, 4H), 7.15 – 6.99 (m, 4H), 7.00 (d, *J* = 7.8 Hz, 2H), 6.82 – 6.77 (m, 3H), 6.72 (d, *J* = 7.8 Hz, 2H), 4.27 (s, 1H), 3.74 (t, *J* = 7.3 Hz, 2H), 3.20 (t, *J* = 7.0 Hz, 1H), 2.25 (s, 3H), 1.71 (q, *J* = 7.6, 7.2 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) = 185.2, 169.3, 168.0, 151.2, 147.6, 144.3, 132.4, 132.0, 131.8, 130.4, 130.0, 129.9, 129.0, 128.5, 126.9, 124.6, 123.1, 119.9, 119.7, 118.3, 116.6, 60.1, 49.5, 42.3, 26.0, 20.8. HRMS (MALD1): m/z $[M + H]^+$ calcd for $C_{31}H_{29}N_3O_3$: 492.2287, found: 492.2265.



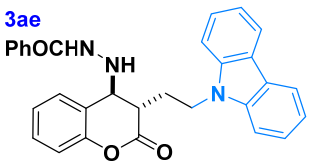
White solid, 75 mg, 76% yield, M.P.: 96.6-98.0.

^1H NMR (400 MHz, $CDCl_3$) δ (ppm) = 7.75 – 7.61 (m, 3H), 7.54 (t, *J* = 7.4 Hz, 1H), 7.44 (t, *J* = 7.6 Hz, 2H), 7.38 – 7.36 (m, 2H), 7.18 –7.07 (m, 4H), 6.90 (d, *J* = 6.6 Hz, 4H), 6.81 (t, *J* = 7.3 Hz, 1H), 6.71 (d, *J* = 8.1 Hz, 2H), 4.87 (br, 1H), 4.29 (s, 1H), 3.74 (t, *J* = 7.5 Hz, 2H), 3.18 (t, *J* = 7.6 Hz, 1H), 1.71 (q, *J* = 7.5 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) = 169.2, 168.0, 159.8, 157.4, 151.2, 147.5, 143.2, 132.1, 131.8, 130.4, 130.0, 129.1, 128.6, 126.9, 125.0, 124.9, 124.7, 120.2, 119.6, 118.2, 116.6, 116.2, 116.0, 60.1, 49.7, 42.3, 26.1. HRMS (ESI): m/z $[M - H]^-$ calcd for $C_{30}H_{26}N_3O_3F$: 494.1885; found: 494.1911.



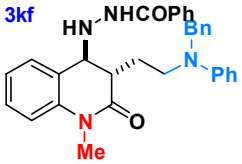
Colourless oil, 45 mg, 54% yield.

^1H NMR (400 MHz, $CDCl_3$) δ (ppm) = 7.91 (s, 1H), 7.70 (d, *J* = 7.4 Hz, 2H), 7.51 (t, *J* = 7.3 Hz, 1H), 7.43 – 7.32 (m, 4H), 7.15 – 7.04 (m, 4H), 6.62 (t, *J* = 7.2 Hz, 1H), 6.57 (d, *J* = 8.1 Hz, 2H), 4.89 (s, 1H), 4.31 (s, 1H), 3.39 (t, *J* = 7.3 Hz, 2H), 3.15 (t, *J* = 6.4 Hz, 1H), 2.83 (s, 3H), 1.71 – 1.58 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) = 169.6, 168.3, 151.4, 132.3, 132.0, 130.5, 130.2, 129.2, 128.8, 127.0, 124.8, 119.8, 116.7, 112.3, 60.0, 50.1, 42.3, 38.5, 25.3. HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{25}H_{25}N_3O_3$: 416.1969; found: 416.1971.



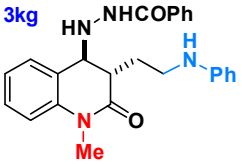
White solid, 60 mg, 63% yield, M.P.: 182.2-183.3.

^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) = 8.02 (d, *J* = 7.7 Hz, 2H), 7.75 (s, 1H), 7.56 (d, *J* = 7.5 Hz, 2H), 7.52 (t, *J* = 7.2 Hz, 1H), 7.41 – 7.34 (m, 8H), 7.18 (t, *J* = 6.2 Hz, 2H), 7.13 (t, *J* = 7.4 Hz, 1H), 7.10 (d, *J* = 8.0 Hz, 1H), 4.47 – 4.42 (m, 1H), 4.38 – 4.34 (m, 1H), 4.22 (s, 1H), 3.25 – 3.22 (m, 1H), 2.04 – 1.98 (m, 1H), 1.90 – 1.84 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) = 169.1, 168.0, 151.0, 139.6, 131.9, 131.7, 130.4, 129.9, 128.4, 126.9, 125.6, 124.7, 122.7, 120.2, 119.4, 118.9, 116.5, 108.2, 60.1, 42.3, 40.2, 27.4. HRMS (ESI): m/z [M + H]$^+$ calcd for $C_{30}H_{25}N_3O_3$: 476.1969; found: 476.1966.



White solid, 97 mg, 96% yield, M.P.: 87.3-89.0.

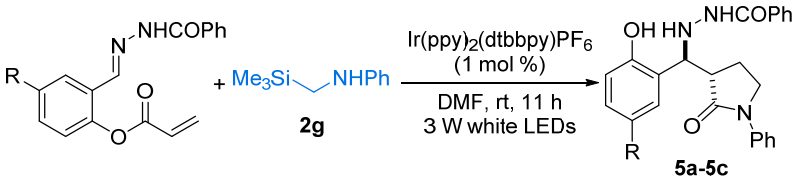
^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 7.81 (d, *J* = 4.5 Hz, 1H), 7.75 – 7.66 (m, 2H), 7.50 (t, *J* = 7.4 Hz, 1H), 7.40 (t, *J* = 7.6 Hz, 2H), 7.35 (t, *J* = 7.6 Hz, 2H), 7.22 – 7.12 (m, 3H), 7.10 – 6.98 (m, 5H), 6.58 (t, *J* = 7.3 Hz, 1H), 6.50 (d, *J* = 8.2 Hz, 2H), 4.81 (d, *J* = 6.2 Hz, 1H), 4.42 (s, 2H), 4.16 (d, *J* = 2.0 Hz, 1H), 3.52 – 3.42 (m, 2H), 3.37 (s, 3H), 2.93 (ddd, *J* = 8.7, 6.6, 2.1 Hz, 1H), 1.66 (ddd, *J* = 17.3, 14.3, 7.8 Hz, 2H). ^{13}C NMR (150 MHz, $CDCl_3$) δ (ppm) = δ 171.4, 167.8, 147.8, 139.6, 138.5, 132.2, 132.0, 130.5, 129.8, 129.1, 128.6, 128.4, 127.1, 126.7, 126.4, 123.3, 122.3, 116.3, 114.8, 112.1, 61.0, 54.4, 48.6, 43.5, 29.4, 26.0. HRMS (ESI): m/z [M + Na]$^+$ calcd for $C_{32}H_{32}N_4O_2$: 527.2417, found: 527.2411.



White solid, 71 mg, 86% yield, M.P.: 139.0-141.9.

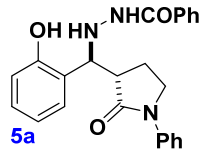
^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 7.77 – 7.63 (m, 3H), 7.52 (t, *J* = 7.3 Hz, 1H), 7.41 (t, *J* = 7.7 Hz, 2H), 7.37 – 7.33 (m, 2H), 7.16 – 7.04 (m, 5H), 6.99 (d, *J* = 8.0 Hz, 2H), 6.85 – 6.74 (m, 3H), 6.72 (d, *J* = 8.1 Hz, 2H), 4.84 (s, 1H), 4.27 (s, 1H), 3.81 – 3.70 (m, 2H), 3.16 (td, *J* = 7.5, 2.3 Hz, 1H), 2.26 (s, 3H), 1.72 (q, *J* = 7.5 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) = 171.3, 167.6, 147.5, 139.4, 132.2, 131.7, 131.7, 130.4, 129.6, 128.9, 128.4, 127.0, 123.2, 122.2, 117.0, 114.7, 112.6, 61.4, 43.5, 41.4, 29.6, 28.6. HRMS (ESI): m/z [M + H]$^+$ calcd for $C_{25}H_{26}N_4O_2$: 415.2129; found: 415.2152.

4.2 General procedure and spectral data of the synthesis of 5a-5c



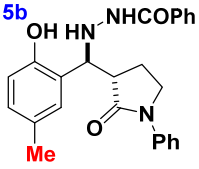
To a dried schlenk tube was treated with **1** (0.20 mmol, 1.0 equiv), **2g** (0.24 mmol, 1.2 equiv), photocatalyst (0.002 mmol, 1 mol%) and DMF (2.0 mL). And then this mixture solution was degassed for 3 times via 'freeze-pump-thaw' procedure. After that, this resulting solution was stirred at a distance of ~5 cm under irradiation by 3 W white LEDs at room temperature for 11 h. The mixture was diluted with ethyl acetate (4 mL), and added saturated $NaHCO_3$ aquous (8 mL), followed by extration with ethyl

acetate (10 mLX3 times). The combined organic phase was washed with brine (10 mL), dried with anhydrous Na_2SO_4 and the solvent was evaporated. The crude product was purified by column chromatography using petroleum ether/ethyl acetate as eluent (3:1 to 1:1), affording the desired product.



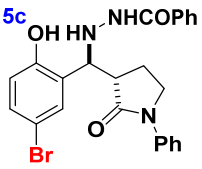
White solid, 72 mg, 90% yield, M.P.: 183.3-184.6.

^{1}H NMR (400 MHz, $(CD_3)_2CO$) δ (ppm) = 9.45 (s, 1H), 9.26 (s, 1H), 7.75 – 7.69 (m, 2H), 7.69 – 7.63 (m, 2H), 7.48 – 7.40 (m, 1H), 7.36 – 7.22 (m, 5H), 7.16 – 7.03 (m, 2H), 6.87 – 6.74 (m, 2H), 5.57 – 5.47 (m, 1H), 5.19 – 5.09 (m, 1H), 3.85 (dt, *J* = 9.5, 7.9 Hz, 1H), 3.77 (td, *J* = 9.1, 3.3 Hz, 1H), 3.15 (td, *J* = 9.4, 3.0 Hz, 1H), 2.79 (s, 1H), 2.70 – 2.57 (m, 1H), 2.34 – 2.20 (m, 1H). ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm) = 173.4, 165.0, 154.4, 139.4, 132.9, 130.8, 128.2, 127.7, 127.5, 127.0, 127.0, 126.4, 123.4, 119.1, 118.4, 114.9, 55.6, 46.6, 46.1, 17.9. HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{24}H_{23}N_3O_3$: 402.1812; found: 402.1828.



White solid, 77 mg, 93% yield, M.P.: 172.5-174.3.

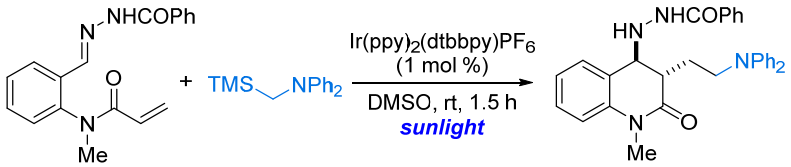
^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) = 9.35 (s, 1H), 8.51 (d, *J* = 6.0 Hz, 1H), 7.76 (d, *J* = 7.7 Hz, 2H), 7.50 (dd, *J* = 13.2, 7.5 Hz, 3H), 7.39 (t, *J* = 7.6 Hz, 2H), 7.34 (t, *J* = 7.7 Hz, 2H), 7.16 (t, *J* = 7.4 Hz, 1H), 7.02 (d, *J* = 8.2 Hz, 1H), 6.90 (s, 1H), 6.77 (d, *J* = 8.2 Hz, 1H), 5.61 (t, *J* = 4.8 Hz, 1H), 4.82 (t, *J* = 4.3 Hz, 1H), 3.59 (td, *J* = 8.9, 5.0 Hz, 1H), 3.25 (dt, *J* = 10.5, 5.7 Hz, 1H), 3.15 (q, *J* = 8.0 Hz, 1H), 2.36 – 2.29 (m, 1H), 2.26 (s, 3H), 2.22 – 2.17 (m, 1H). ^{13}C NMR (150 MHz, $CDCl_3$) δ (ppm) = 174.0, 166.9, 154.4, 138.8, 132.0, 131.9, 130.1, 130.0, 128.7, 128.6, 128.5, 127.0, 125.0, 121.6, 120.7, 117.2, 63.2, 47.3, 46.2, 20.5, 20.4. HRMS (ESI): m/z $[M + Na]^+$ calcd for $C_{25}H_{25}N_3O_3$: 438.1788, found: 438.1794.



White solid, 78 mg, 81% yield, M.P.: 181.9-183.1.

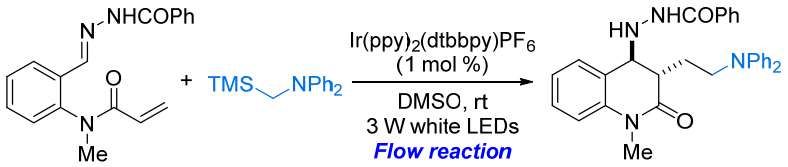
^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm) = 9.94 (s, 1H), 9.83 (d, J = 5.4 Hz, 1H), 7.65 – 7.62 (m, 2H), 7.57 – 7.55 (m, 3H), 7.44 (t, J = 7.4 Hz, 1H), 7.30 (q, J = 7.4 Hz, 4H), 7.21 (dd, J = 8.6, 2.6 Hz, 1H), 7.08 (t, J = 7.4 Hz, 1H), 6.77 (d, J = 8.5 Hz, 1H), 5.65 (t, J = 5.6 Hz, 1H), 5.06 (dd, J = 5.4, 3.1 Hz, 1H), 3.72 (q, J = 5.8, 3.8 Hz, 2H), 3.00 (td, J = 9.4, 3.2 Hz, 1H), 2.38 (dq, J = 12.4, 8.4 Hz, 1H), 2.02 – 1.87 (m, 1H). ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm) = 173.0, 165.2, 153.9, 139.3, 132.9, 130.8, 130.1, 129.9, 129.2, 128.2, 127.8, 126.9, 123.6, 119.3, 116.9, 109.9, 55.5, 46.3, 46.1, 18.2. HRMS (ESI): m/z $[M + Na]^+$ calcd for $C_{24}H_{22}N_3O_3Br$: 502.0737, found: 502.0740.

4.3 General procedure for sunlight irradiation reaction



To a dried schlenk tube was treated with **1k** (0.20 mmol, 1.0 equiv), **2a** (0.30 mmol, 1.5 equiv), photocatalyst (0.002 mmol, 1 mol%) and DMSO (2.0 mL). And then this mixture solution was degassed for 3 times via 'freeze-pump-thaw' procedure. After that, the solution was stirred under irradiation of sunlight directly at room temperature for 1.5 h. The mixture was diluted with ethyl acetate (4 mL), and added saturated $NaHCO_3$ aquous (8 mL), followed by extration with ethyl acetate (10 mL* 3 times). The combined organic phase was washed with brine (10 mL), dried with anhydrous Na_2SO_4 and the solvent was evaporated. The crude product was purified by column chromatography using petroleum ether/ethyl acetate as eluent (5:1 to 3:1), affording 80 mg of a white solid in 82% yield.

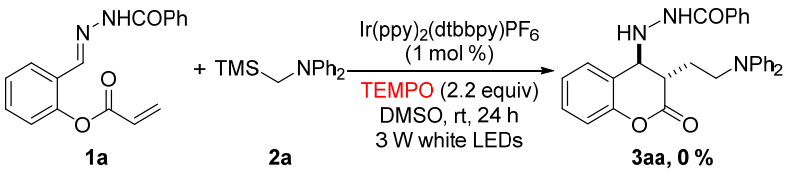
4.4 General procedure for continuing flow reaction



1k **2a** **3ka, 70 min, 83% yield**

To a dried schlenk tube was treated with **1k** (0.20 mmol, 1.0 equiv), **2a** (0.30 mmol, 1.5 equiv), photocatalyst (0.002 mmol, 1 mol%) and DMSO (2.0 mL). And then this mixture solution was degassed for 3 times via 'freeze-pump-thaw' procedure. After that, the resulting mixture is then pumped through the photoreactor at a flow rate (0.12 mL/min) to achieve a residence time of 70 min, and collected in the flask. The mixture was diluted with ethyl acetate (2 mL), and added saturated $NaHCO_3$ aquous (8 mL), followed by extration with ethyl acetate (10 mL* 3 times). The combined organic phase was washed with brine (15 mL), dried with anhydrous Na_2SO_4 and the solvent was evaporated. The crude product was purified by column chromatography using petroleum ether/ethyl acetate as eluent (5:1 to 3:1), affording the desired product 81 mg as a white solid in 83% yield.

4.5 General procedure for control experiment

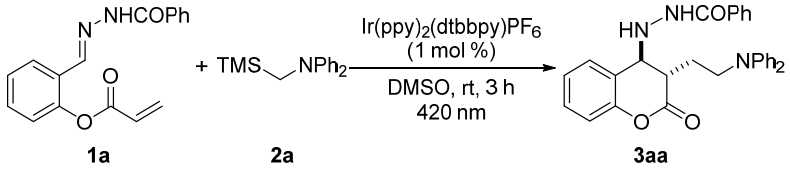


1a **2a** **3aa, 0 %**

To a dried schlenk tube was treated with **1** (0.20 mmol, 1.0 equiv), **2** (0.30 mmol, 1.5 equiv), photocatalyst (0.002 mmol, 1 mol%), 2,2,6,6-tetramethylpiperidine-N-oxyl (TEMPO) (0.44 mmol, 2.2 equiv) and DMSO (2.0 mL). And then this mixture solution was degassed for 3 times via 'freeze-pump-thaw' procedure. After that, this resulting solution was stirred at a distance of ~5 cm under irradiation by 3 W white LEDs at room temperature for 24 h. Monitored by TLC analysis, we found the substrates 1a and 2a were remained and no desired product was detected. So the reaction was completely suppressed, suggesting that this transformation might involve a radical process.

4.6 Mesurement of quantum yield with 1a

Quantum yield measurement was performed in a standard spectro-cell containing a plastic plug which total volume was 4 mL and path-length was 1 cm. The cuvette was filled with the homogeneous solution which was prepared through the model reaction condition (containing substrate **1a** (0.02 mmol,), **2a** (0.30 mmol), DMSO (2.0 mL)， and photocatalyst [Ir] (0.0002 mmol). The quantum efficiency was measured by the irradiation of 420 nm laser light source, and calculated by using the equation: **Φ= Mole number for product/Mole number for absorption of photons**, where the number of reaction molecular was obtained from ^{1}H NMR with triphenylmethane as the internal standard and the number of absorbed photons were calculated from the illumination power and absorbance of the reaction solution. The illumination power was measured using a digital photodiode power meter (Optical Power/Energy Meter/Model 842 PE). The amount of absorbed light was determined from reaction solution absorbance (abs) at the illuminated wavelength. Light intensity was measured before the model reaction solution was irradiated by 420 nm laser light, begining and stopping the photocatalytic reaction, which was 0.93 mW cm^{-2}, and irradiate time was set to 10800 s.



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

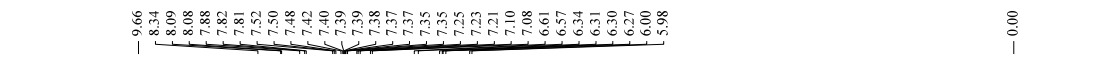
Φ= Mole number for product/Mole number for absorption of photons

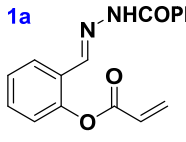
$$\Phi = \frac{n_{3aa} N_A / t}{f P \lambda / h c}$$

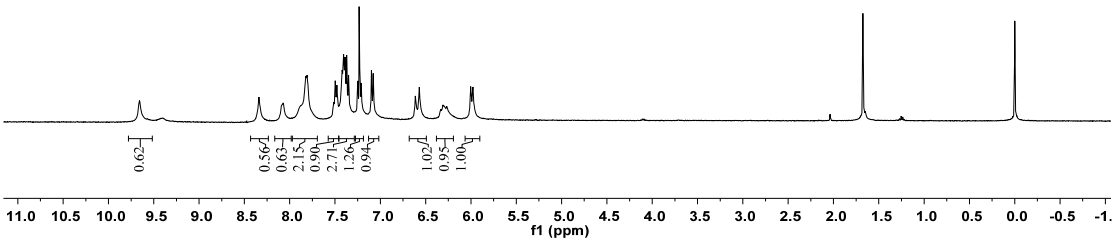
$\mathbf{n_{3aa}}$: the mole number of the product **3aa**, 1.475*10^{-5} mol (7% yield) ; t: reaction time 10800 s; $\mathbf{N_A}$: 6.02×10^{23}/ mol; **f**: 1-10^{-A} (420 nm, A = 3.46); **P**: **P** = **E*****S** (**E**: illumination intensity, **E** = 0.93 mW cm^{-2}; **S**: the area that irradiated **S** = 0.2425 cm^2); **λ**: wavelength (**λ** = 4.2×10^{-7} m); **h**: planck constant (**h** = 6.626×10^{-34} W); **c**: velocity of light (**c** = 3×10^8 m/ s).

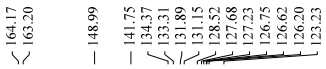
we calculated the quantum yield of the model reaction of **1a** with **2a** to be **0.002**. This result shows that the reaction proceeds through a sequential redox process rather than by a radical chain propagation process.

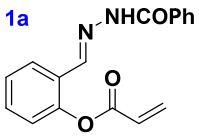
5. ^{1}H and ^{13}C spectra for all new compounds

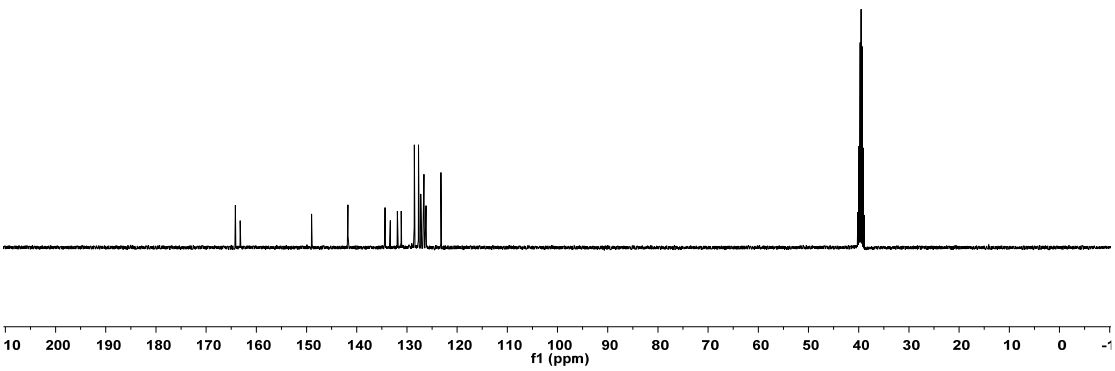


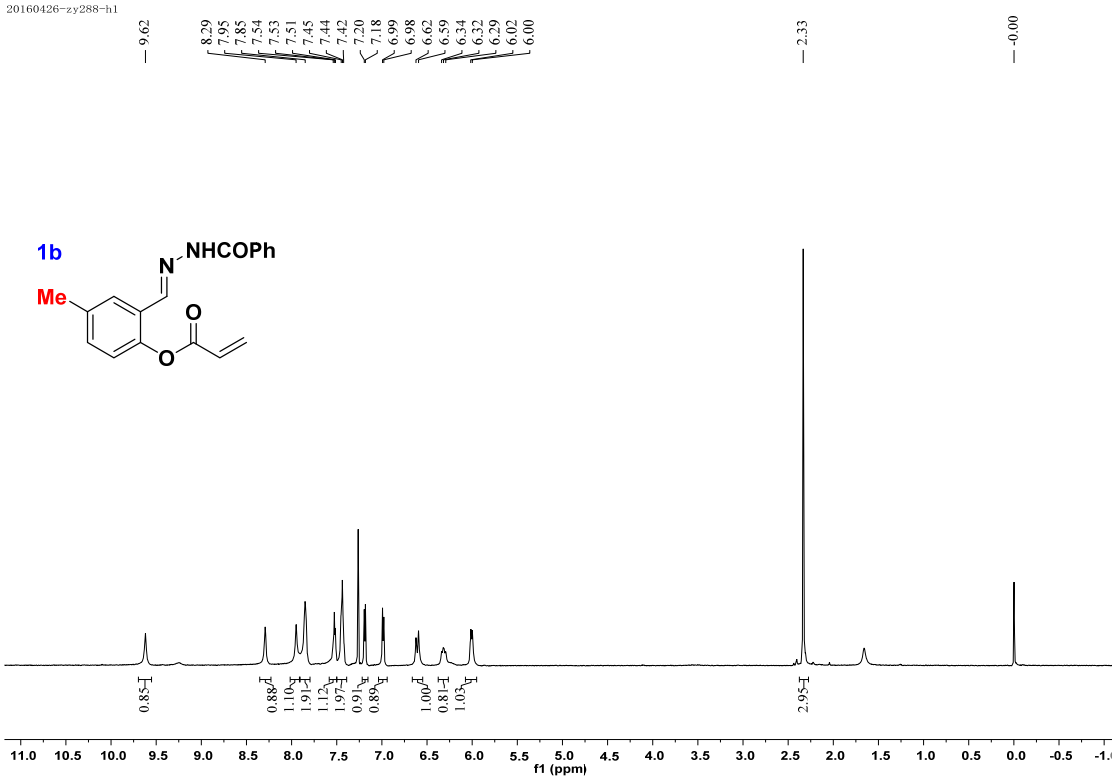


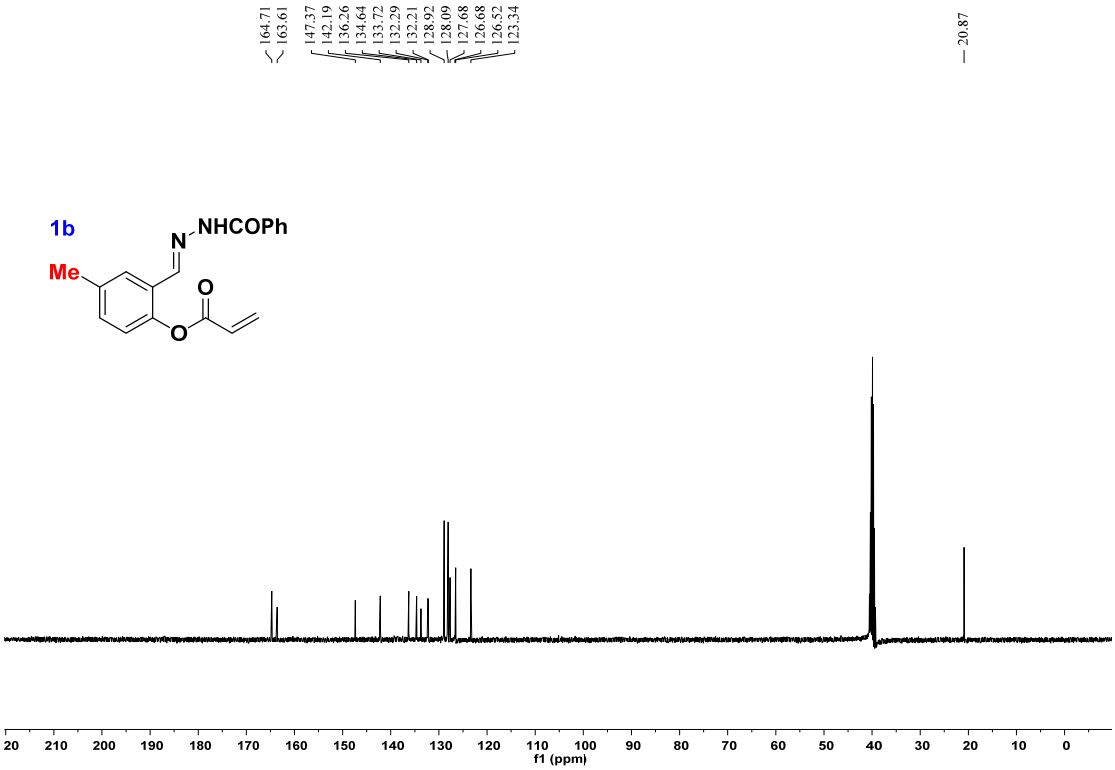


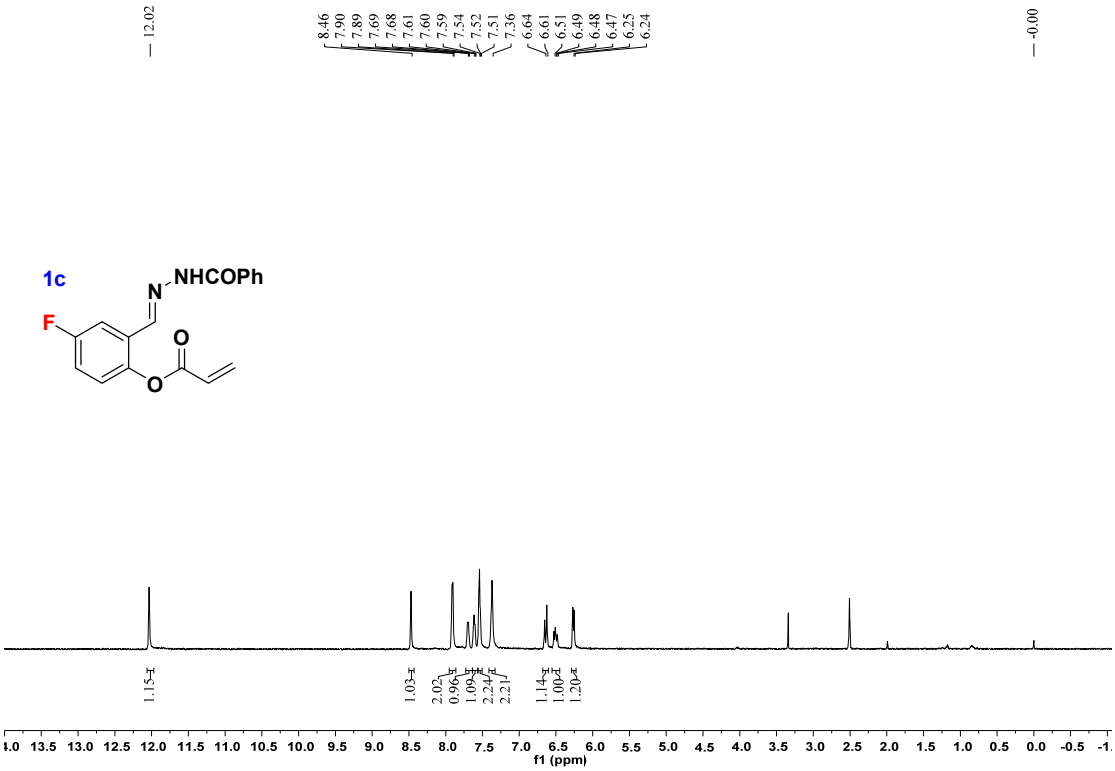


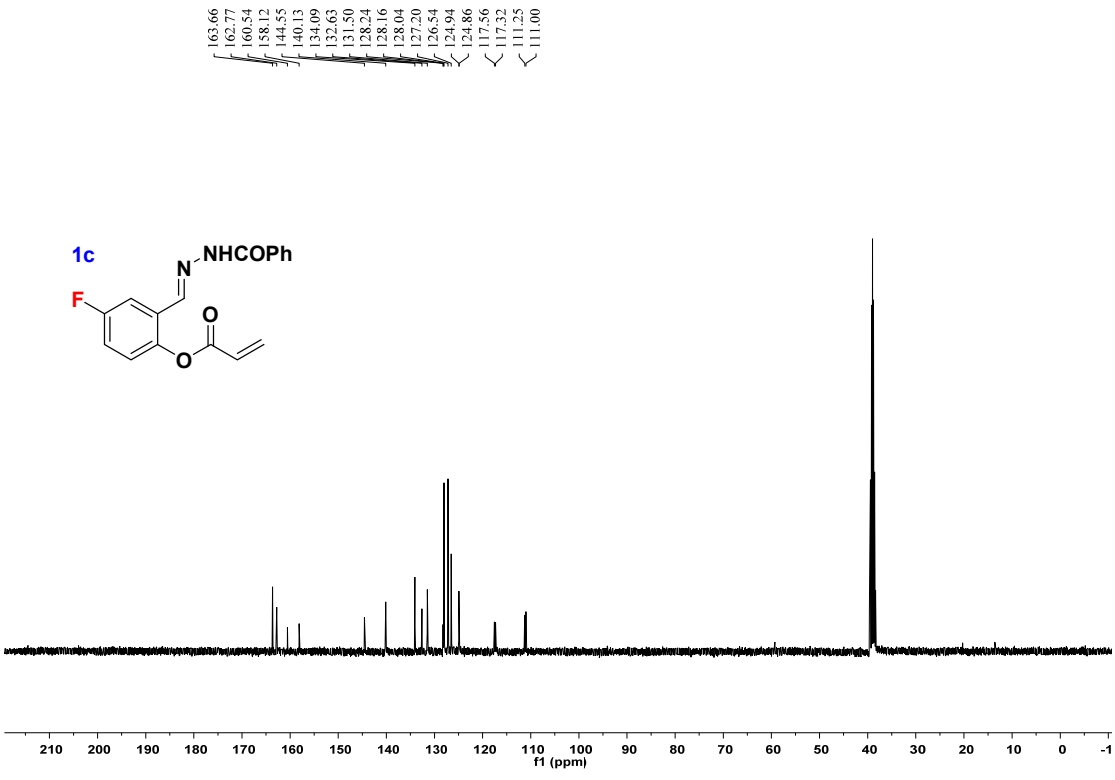


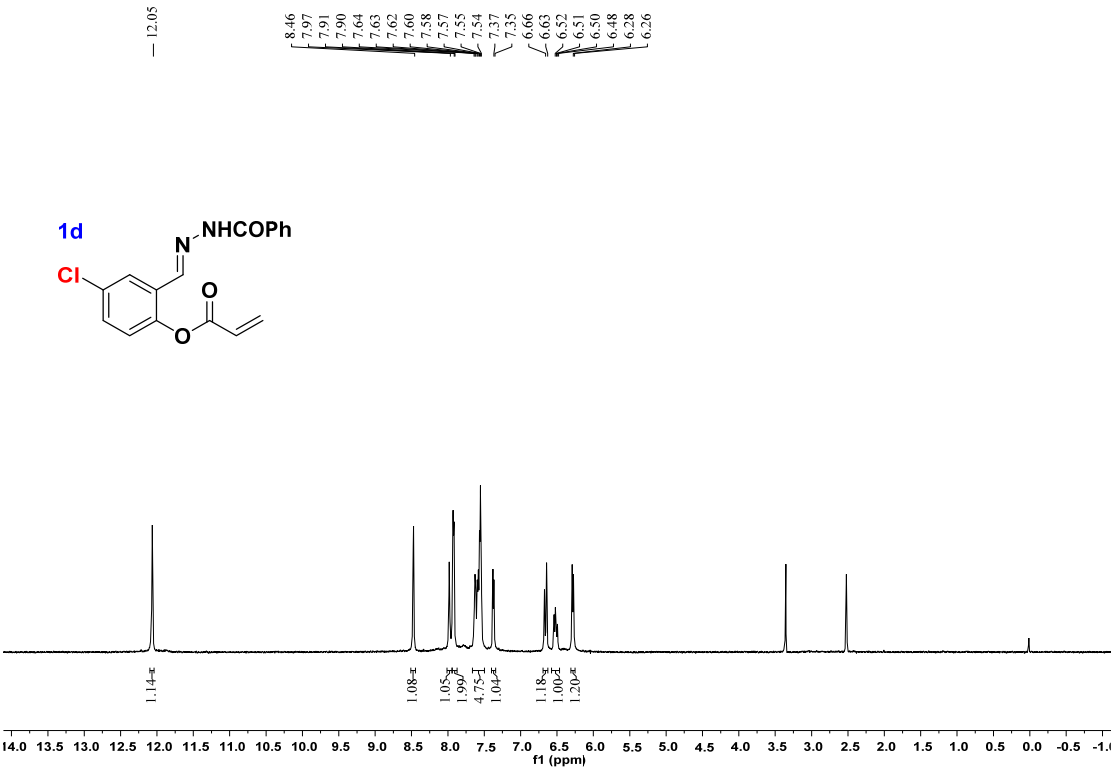



20160426-zy288-h1
9.62
8.29
7.95
7.85
7.54
7.53
7.51
7.45
7.44
7.42
7.20
7.18
6.99
6.98
6.62
6.59
6.34
6.32
6.29
6.02
6.00
2.33
-0.00
1b
NHCOPh
N
Me
O
O
0.85
0.88
1.10
1.91
1.12
1.97
0.91
0.89
1.00
0.81
1.03
2.95
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


164.71
163.61
147.37
142.19
136.26
134.64
133.72
132.29
132.21
128.92
128.09
127.68
126.68
126.52
123.34
20.87
1b
NHCOPh
N
Me
O
O
20 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


12.02
8.46
7.90
7.89
7.69
7.68
7.61
7.60
7.59
7.54
7.52
7.51
7.36
6.64
6.61
6.51
6.49
6.48
6.47
6.25
6.24
-0.00
1c
NHCOPh
F
O
O
1.15
1.03
2.02
0.96
1.09
2.24
2.21
1.14
1.00
1.20
f1 (ppm)


163.66
162.77
160.54
158.12
144.55
140.13
134.09
132.63
131.50
128.24
128.16
128.04
127.20
126.54
124.94
124.86
117.56
117.32
111.25
111.00
1c
NHCOPh
F
O
O
f1 (ppm)


12.05
8.46
7.97
7.91
7.90
7.64
7.63
7.62
7.60
7.58
7.57
7.55
7.54
7.37
7.35
6.66
6.63
6.52
6.51
6.50
6.48
6.28
6.26
1d
NHCOPh
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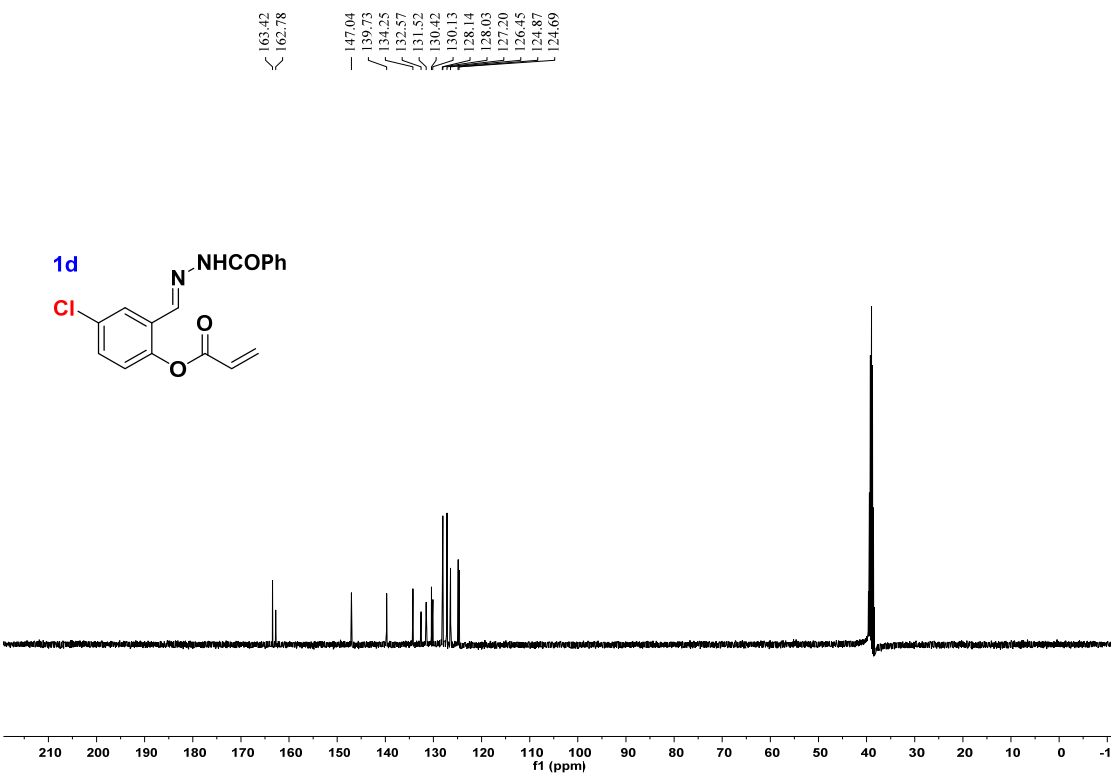
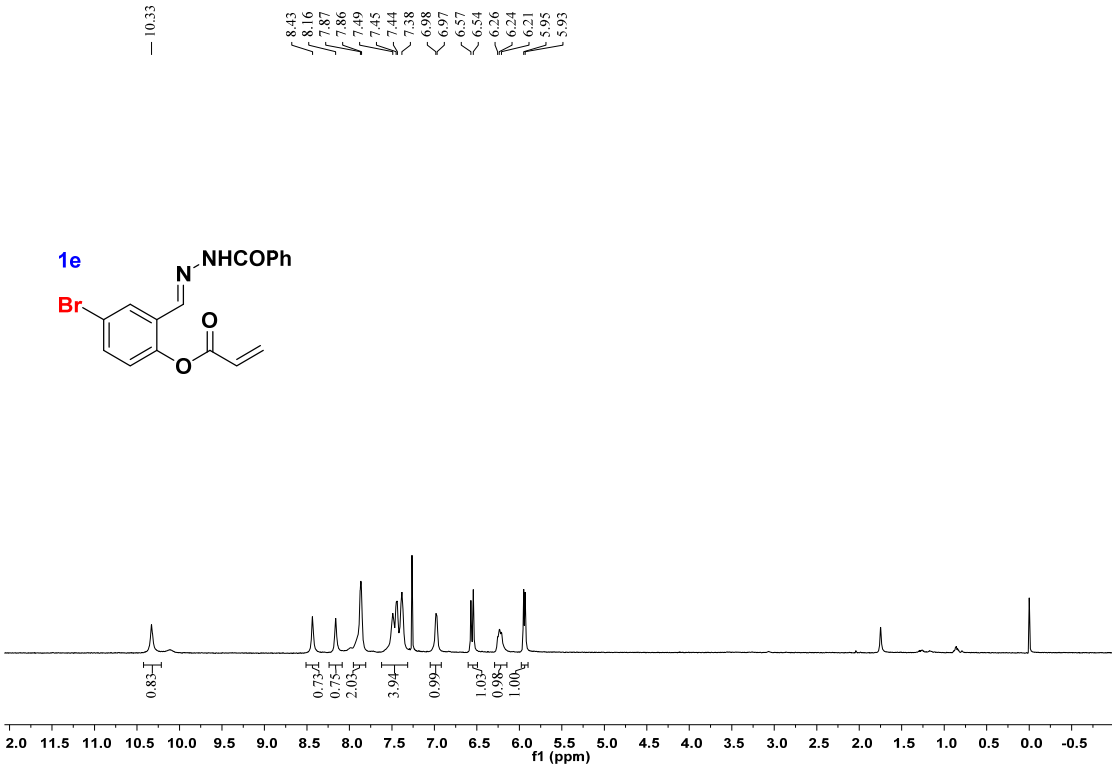
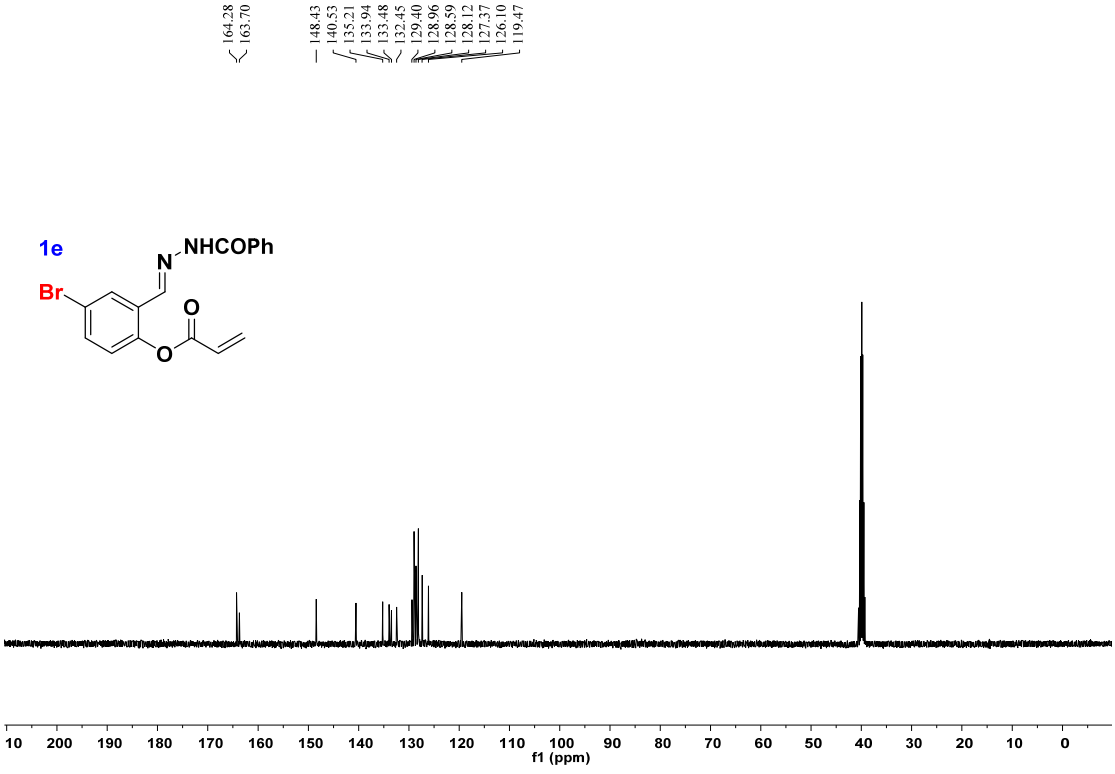
1.04
1.18
1.00
1.20
f1 (ppm)


163.42
162.78
147.04
139.73
134.25
132.57
131.52
130.42
130.13
128.14
128.03
127.20
126.45
124.87
124.69
1d
NHCOPh
N
Cl
O
O
f1 (ppm)


10.33
8.43
8.16
7.87
7.86
7.49
7.45
7.44
7.38
6.98
6.97
6.57
6.54
6.26
6.24
6.21
5.95
5.93
1e
NHCOPh
N
Br
O
O
0.83
0.73
0.75
2.03
3.94
0.99
1.03
0.98
1.00
2.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


164.28
163.70
148.43
140.53
135.21
133.94
133.48
132.45
129.40
128.96
128.59
128.12
127.37
126.10
119.47
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NHCOPh
N
Br
O
O
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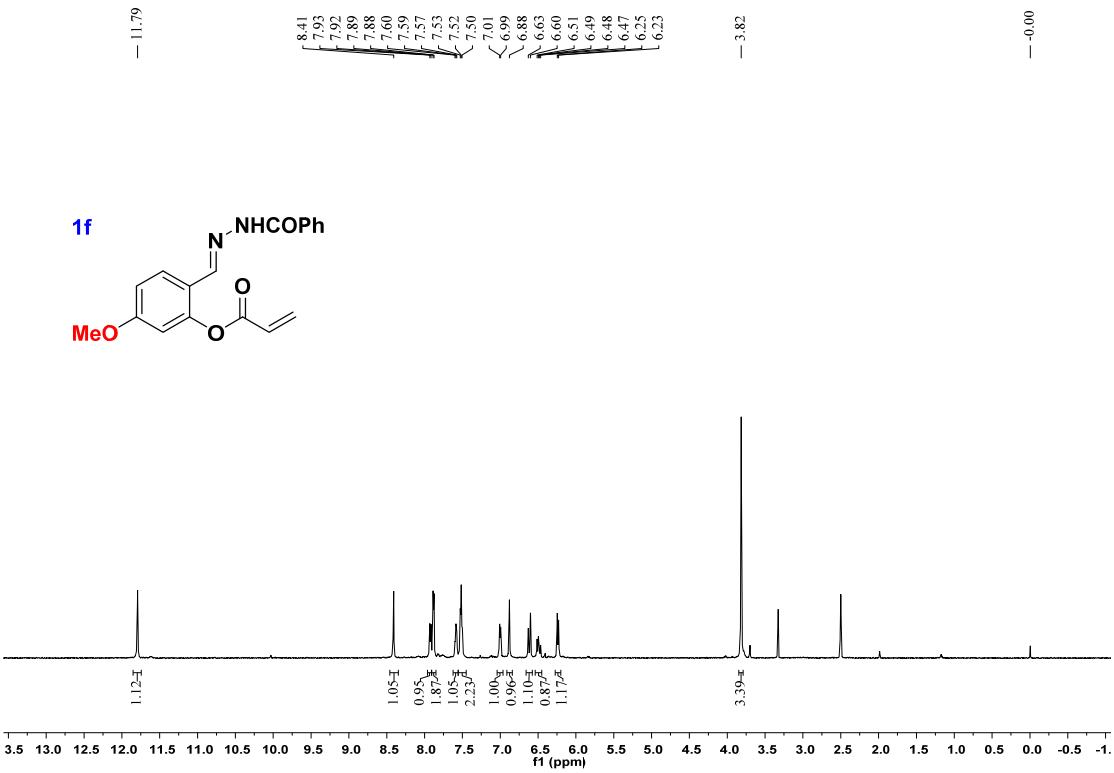
f1 (ppm)


11.79
8.41
7.93
7.92
7.89
7.88
7.60
7.59
7.57
7.53
7.52
7.50
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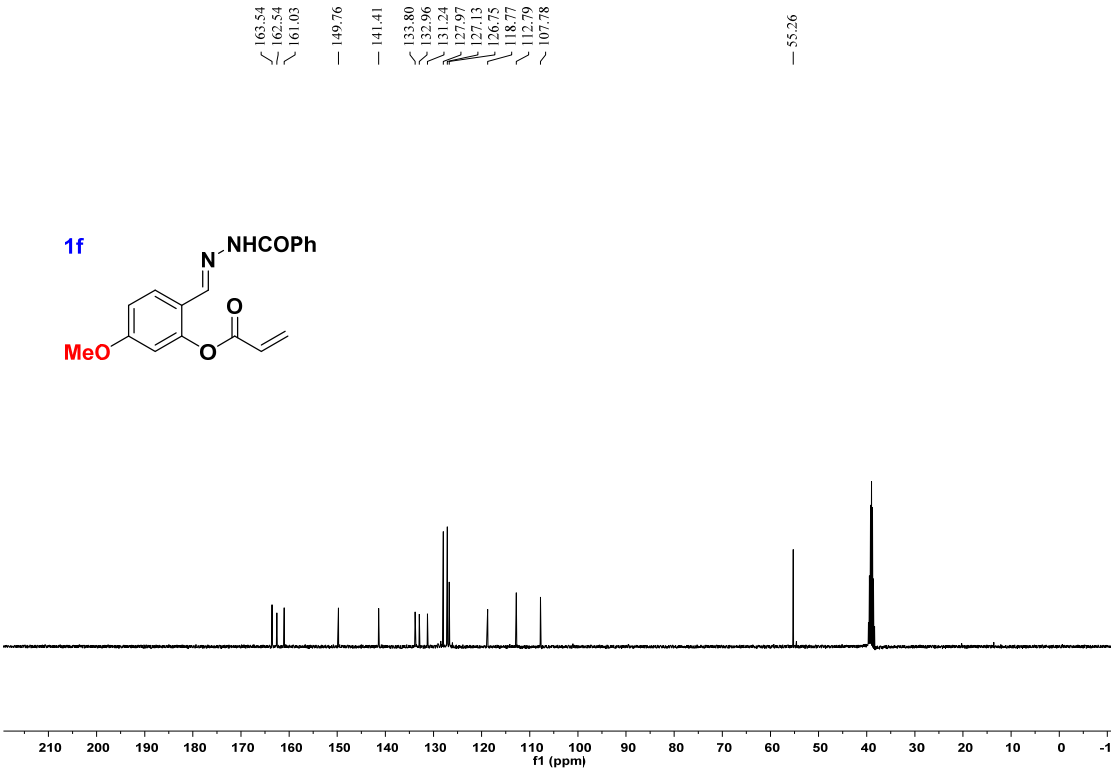
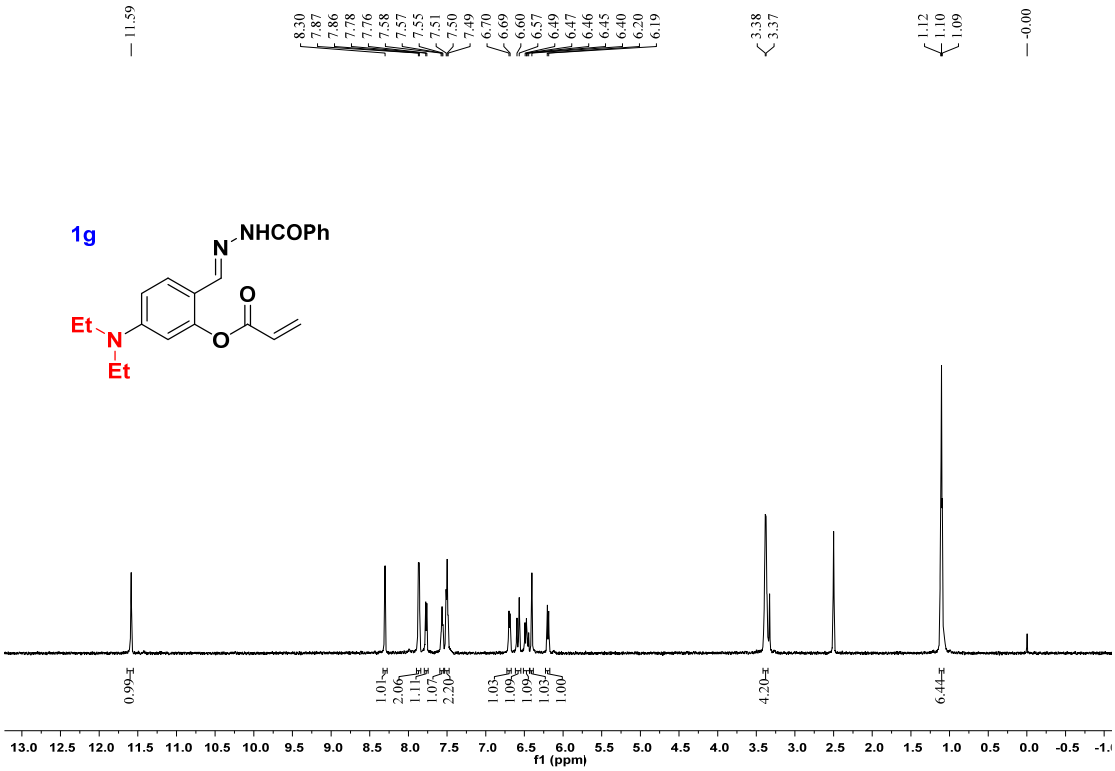
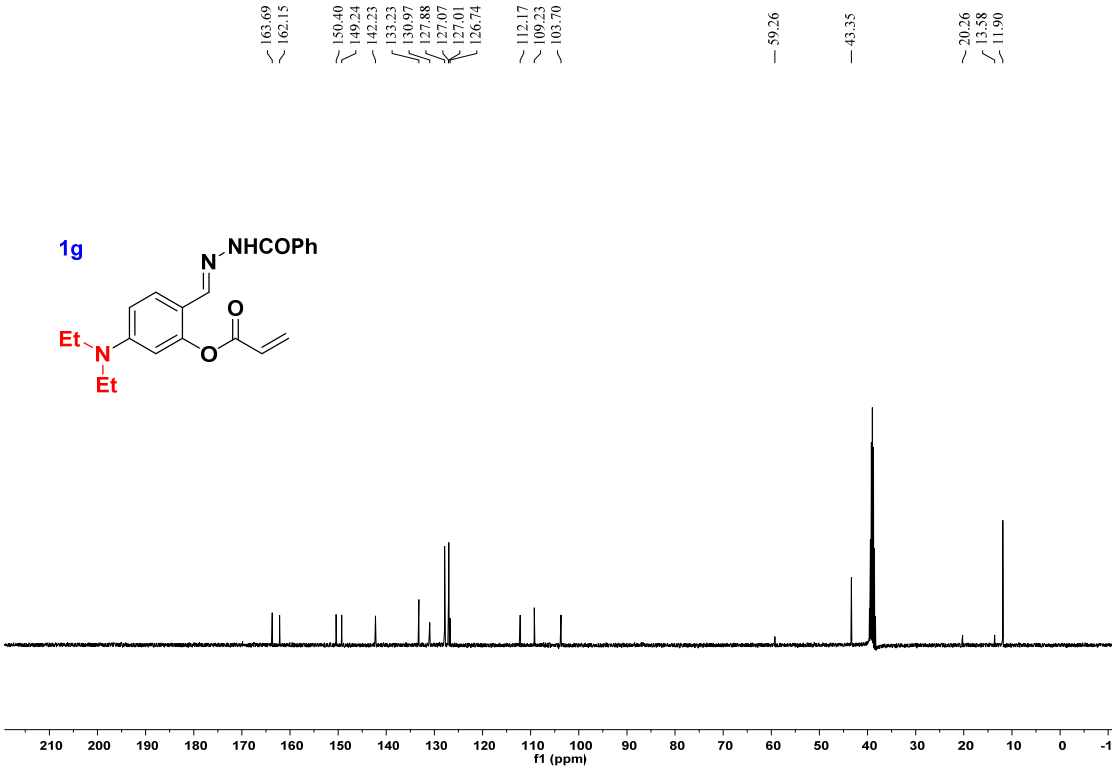
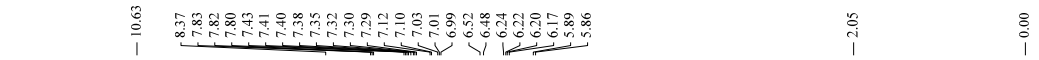
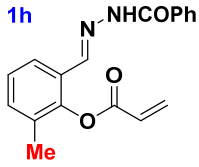
6.99
6.88
6.63
6.60
6.51
6.49
6.48
6.47
6.25
6.23
3.82
-0.00
1f
NHCOPh
N
O
MeO
O
1.12
1.05
0.95
1.87
1.05
2.23
1.00
0.96
1.10
0.87
1.17
3.39
f1 (ppm)

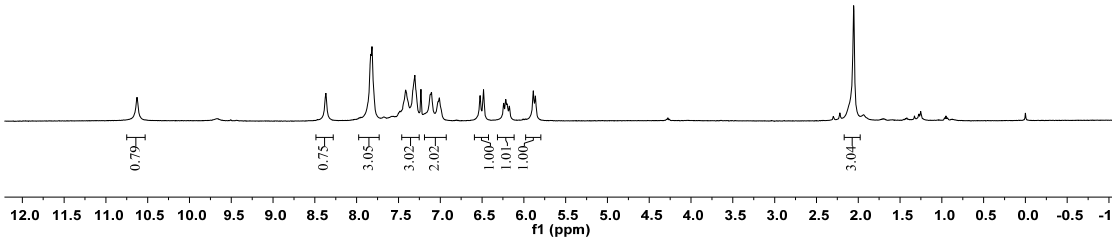

163.54
162.54
161.03
149.76
141.41
133.80
132.96
131.24
127.97
127.13
126.75
118.77
112.79
107.78
55.26
1f
NHCOPh
N
O
MeO
O
f1 (ppm)

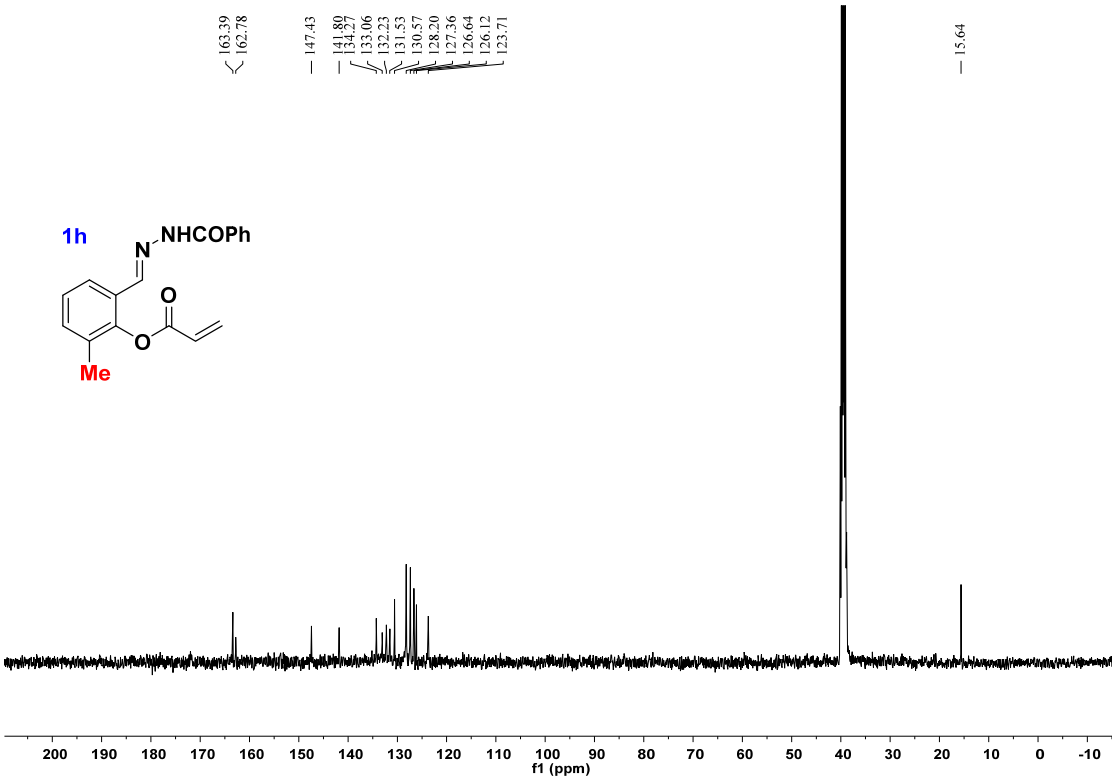

11.59
8.30
7.87
7.86
7.78
7.76
7.58
7.57
7.55
7.51
7.50
7.49
6.70
6.69
6.60
6.57
6.49
6.47
6.46
6.45
6.40
6.20
6.19
3.38
3.37
1.12
1.10
1.09
-0.00
1g
NHCOPh
N
O
Et
N
Et
O
Et
0.99
1.01
2.06
1.11
1.07
2.20
1.03
1.09
1.09
1.03
1.00
4.20
6.44
f1 (ppm)


163.69
162.15
150.40
149.24
142.23
133.23
130.97
127.88
127.07
127.01
126.74
112.17
109.23
103.70
59.26
43.35
20.26
13.58
11.90
1g
NHCOPh
N
O
Et
N
Et
O
f1 (ppm)

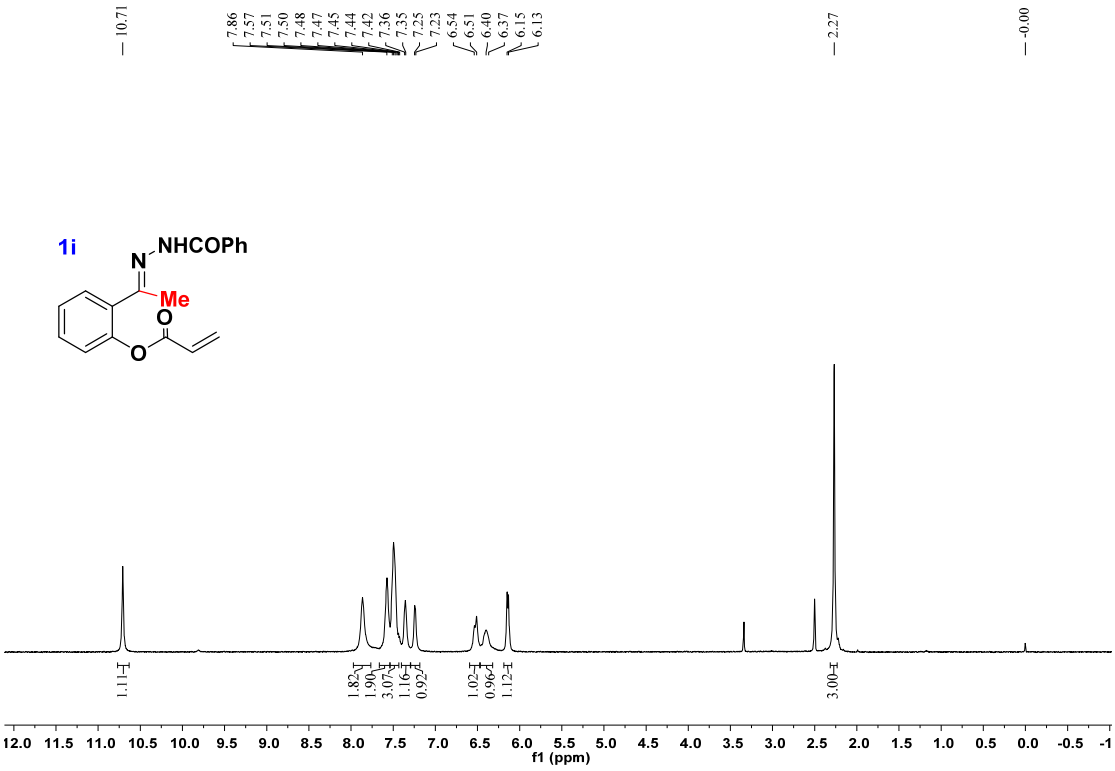

10.63
8.37
7.83
7.82
7.80
7.43
7.41
7.40
7.38
7.35
7.32
7.30
7.29
7.12
7.10
7.03
7.01
6.99
6.52
6.48
6.24
6.22
6.20
6.17
5.89
5.86
2.05
0.00

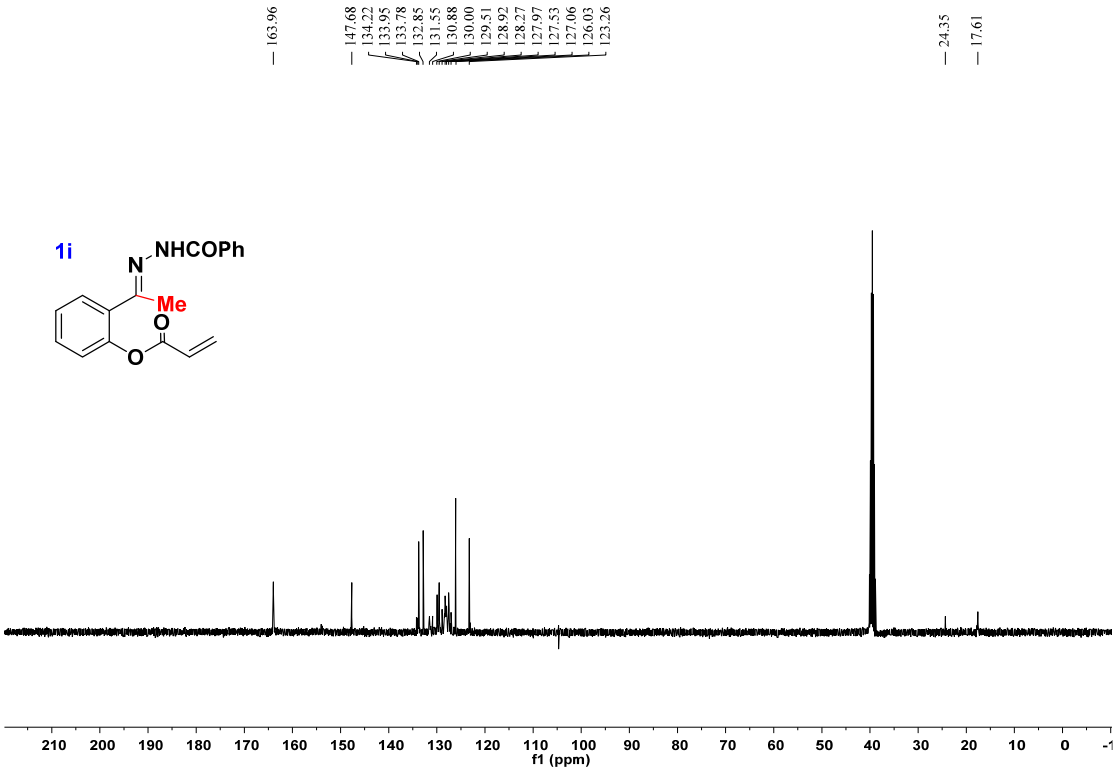

1h
NHCOPh
N
O
O
Me

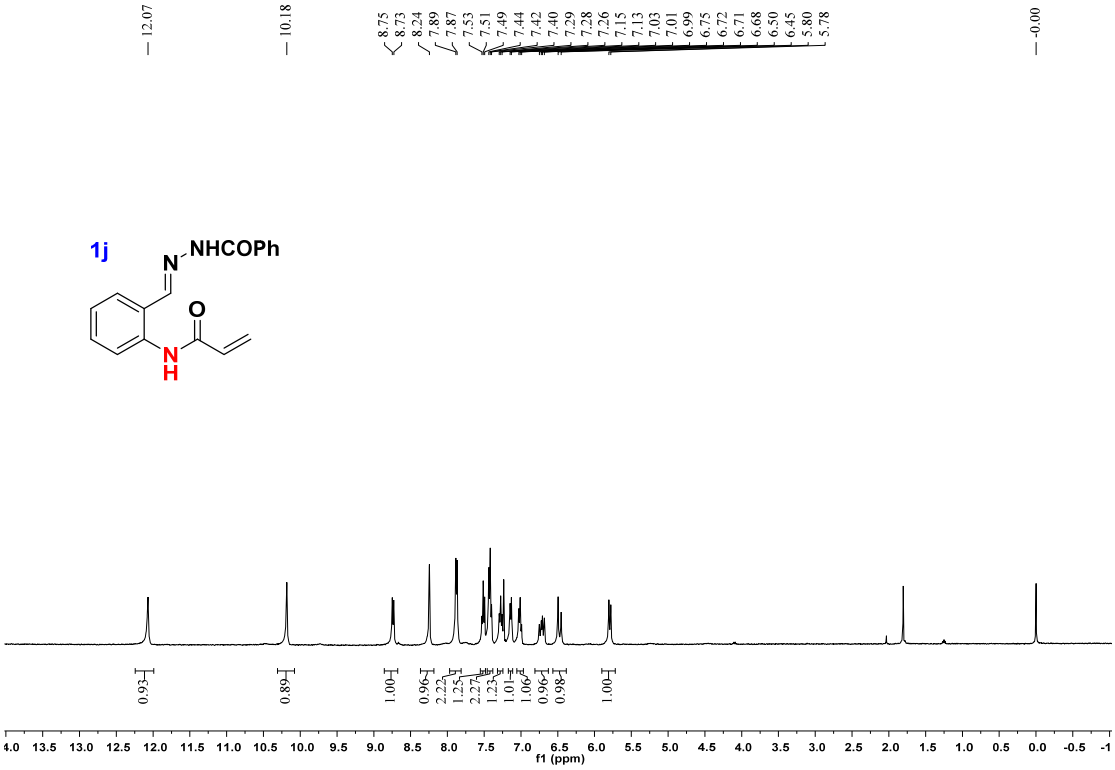

0.79
0.75
3.05
3.02
2.02
1.00
1.01
1.00
3.04
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)

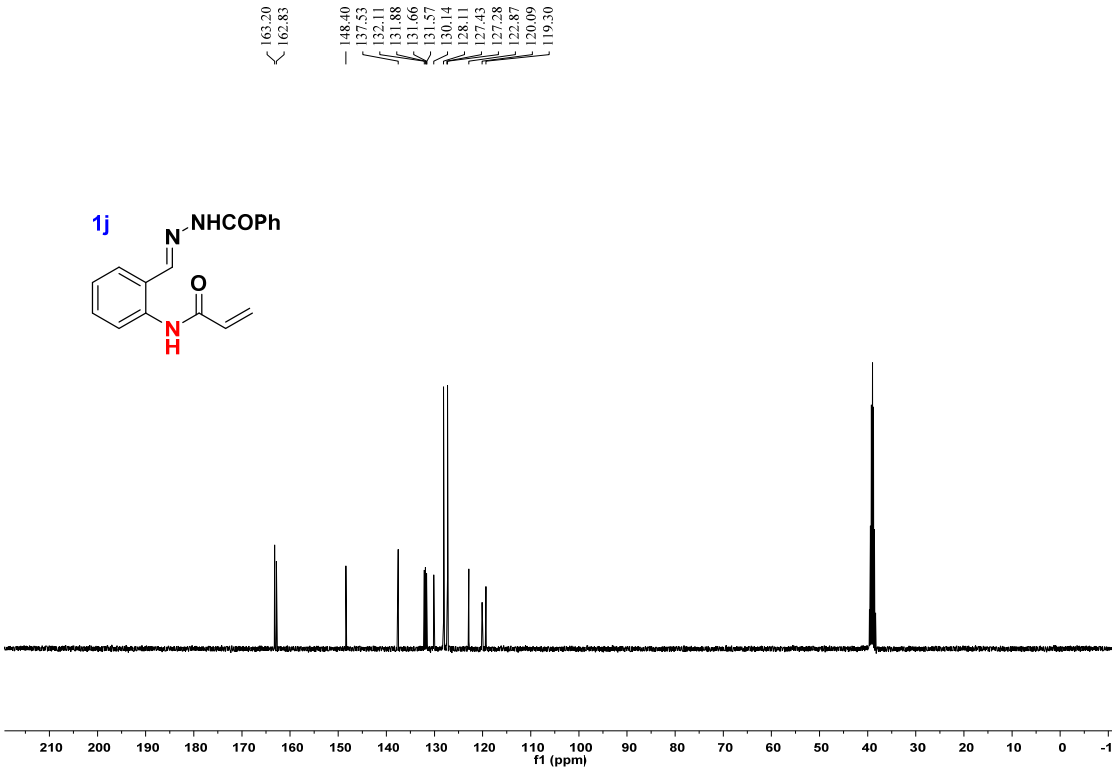

163.39
162.78
147.43
141.80
134.27
133.06
132.23
131.53
130.57
128.20
127.36
126.64
126.12
123.71
15.64
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

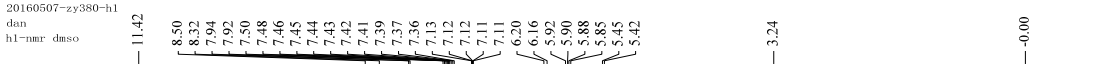
1h
NHCOPh
N
O
O
Me

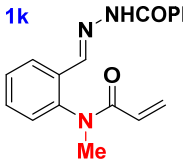

10.71
7.86
7.57
7.51
7.50
7.48
7.47
7.45
7.44
7.42
7.36
7.35
7.25
7.23
6.54
6.51
6.40
6.37
6.15
6.13
2.27
-0.00
1i
NHCOPh
N
Me
O
O
1.11
1.82
1.90
3.07
1.16
0.92
1.02
0.96
1.12
3.00
f1 (ppm)

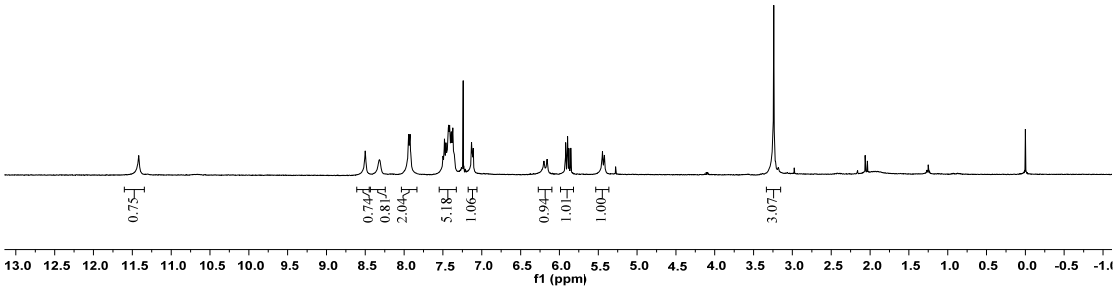

163.96
147.68
134.22
133.95
133.78
132.85
131.55
130.88
130.00
129.51
128.92
128.27
127.97
127.53
127.06
126.03
123.26
24.35
17.61
1i
NHCOPh
N
Me
O
O
f1 (ppm)

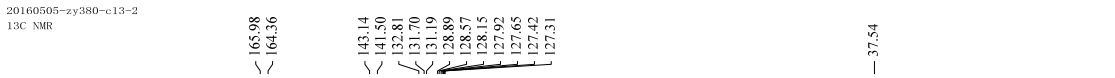

12.07
10.18
8.75
8.73
8.24
7.89
7.87
7.53
7.51
7.49
7.44
7.42
7.40
7.29
7.28
7.26
7.15
7.13
7.03
7.01
6.99
6.75
6.72
6.71
6.68
6.50
6.45
5.80
5.78
0.00
1j
NHCOPh
N
O
NH
0.93
0.89
1.00
0.96
2.22
1.25
2.27
1.23
1.01
1.06
0.96
0.98
1.00
f1 (ppm)

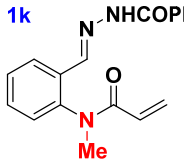

163.20
162.83
148.40
137.53
132.11
131.88
131.66
131.57
130.14
128.11
127.43
127.28
122.87
120.09
119.30
1j
NHCOPh
N
O
NH
f1 (ppm)

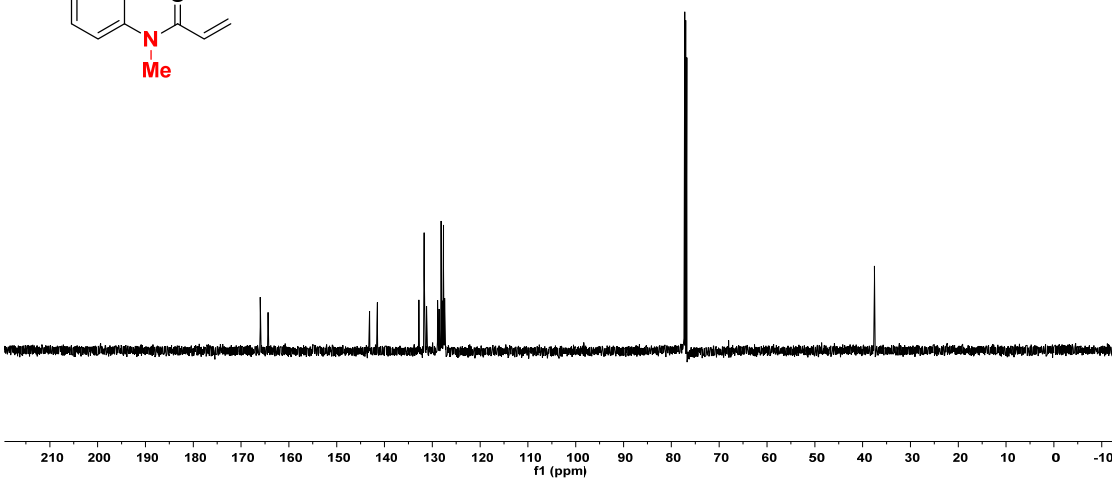

20160507-zy380-h1
dan
h1-nmr dmso
11.42
8.50
8.32
7.94
7.92
7.50
7.48
7.46
7.45
7.44
7.43
7.42
7.41
7.39
7.37
7.36
7.13
7.12
7.12
7.11
7.11
6.20
6.16
5.92
5.90
5.88
5.85
5.45
5.42
3.24
-0.00

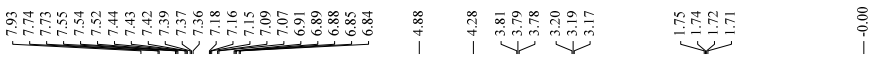

1k
NHCOPh
N
O
N
Me

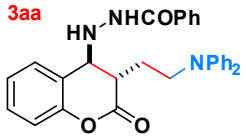

0.75
0.74
0.81
2.04
5.18
1.06
0.94
1.01
1.00
3.07
13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

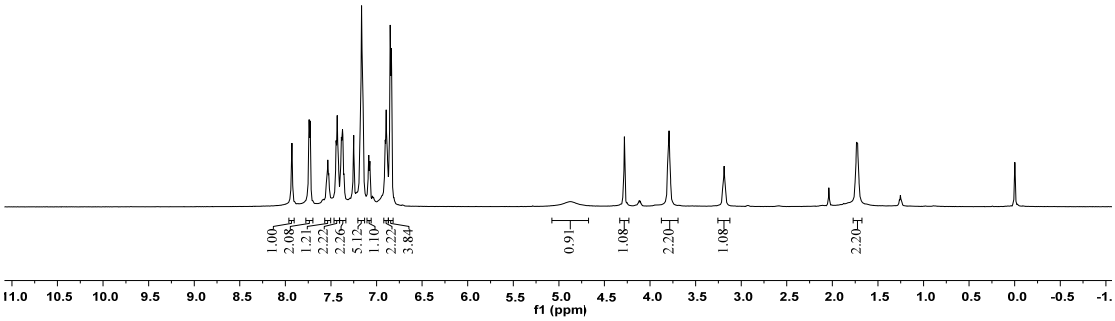

20160505-zy380-c13-2
13C NMR
165.98
164.36
143.14
141.50
132.81
131.70
131.19
128.89
128.57
128.15
127.92
127.65
127.42
127.31
37.54


1k
NHCOPh
N
O
N
Me

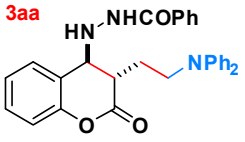

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

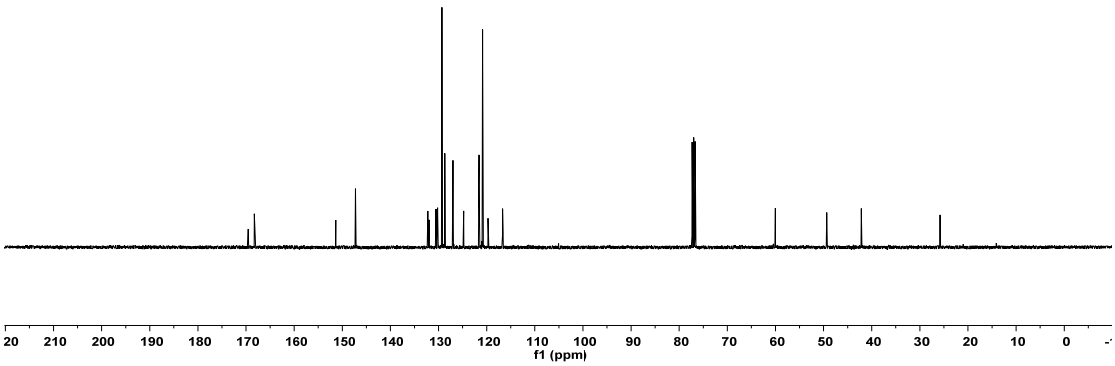

7.93
7.74
7.73
7.55
7.54
7.52
7.44
7.43
7.42
7.39
7.37
7.36
7.18
7.16
7.15
7.09
7.07
6.91
6.89
6.88
6.85
6.84
4.88
4.28
3.81
3.79
3.78
3.20
3.19
3.17
1.75
1.74
1.72
1.71
0.00

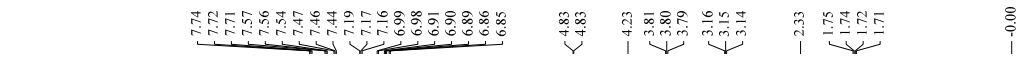

3aa
HN
NHCOPh
NPh2
O
O

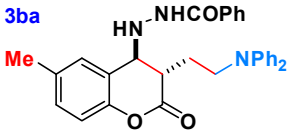

1.00
2.08
1.21
2.22
2.26
5.12
1.10
2.22
3.84
0.91
1.08
2.20
1.08
2.20
f1 (ppm)

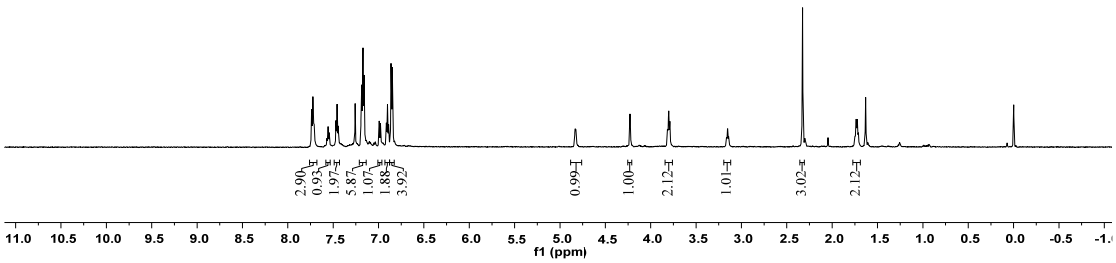

169.53
168.27
151.36
147.26
132.23
131.98
130.55
130.18
129.30
128.70
127.02
124.77
121.58
120.84
119.74
116.70
60.02
49.35
42.17
25.80

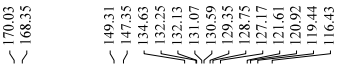

3aa
HN
NHCOPh
NPh2
O
O


f1 (ppm)


7.74
7.72
7.71
7.57
7.56
7.54
7.47
7.46
7.44
7.19
7.17
7.16
6.99
6.98
6.91
6.90
6.89
6.86
6.85
4.83
4.83
4.23
3.81
3.80
3.79
3.16
3.15
3.14
2.33
1.75
1.74
1.72
1.71
-0.00


3ba
HN
NHCOPh
Me
NPh2
O
O


2.90
0.93
1.97
5.87
1.07
1.88
3.92
0.99
1.00
2.12
1.01
3.02
2.12
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


170.03
168.35
149.31
147.35
134.63
132.25
132.13
131.07
130.59
129.35
128.75
127.17
121.61
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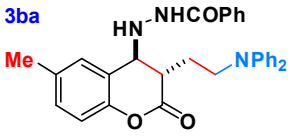
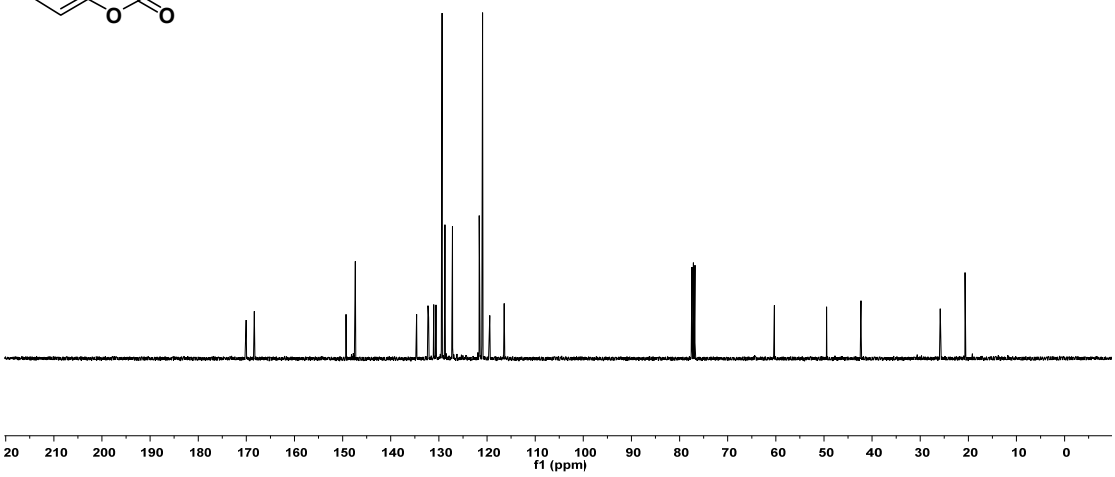
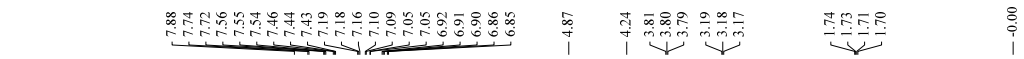
116.43
60.31
49.44
42.30
25.85
20.67


3ba
HN
NHCOPh
Me
NPh2
O
O


20 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.88
7.74
7.72
7.56
7.55
7.54
7.46
7.44
7.43
7.19
7.18
7.16
7.10
7.09
7.05
7.05
6.92
6.91
6.90
6.86
6.85
4.87
4.24
3.81
3.80
3.79
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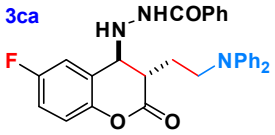
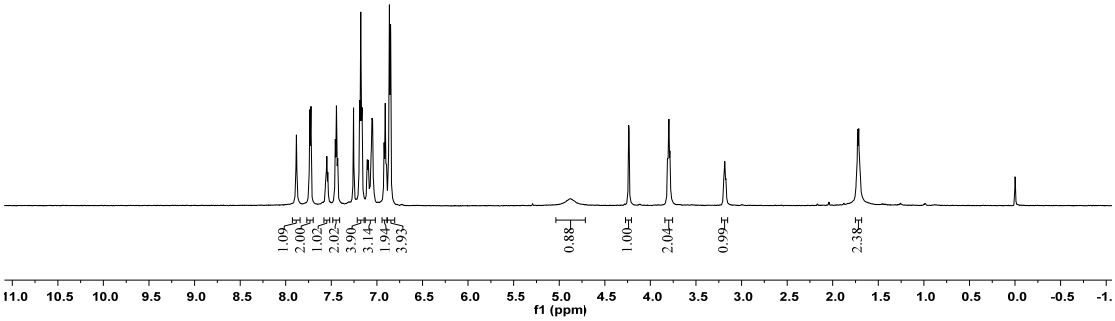
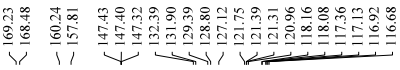
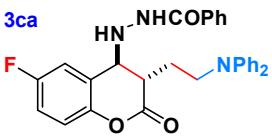
1.74
1.73
1.71
1.70
-0.00

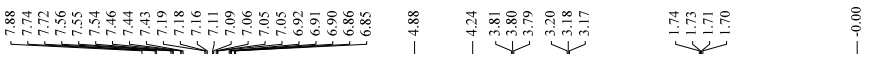

3ca
NHCOPh
HN
F
NPh2
O
O

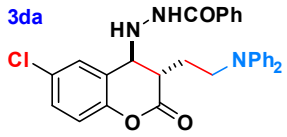

1.09
2.00
1.02
2.02
3.90
3.14
1.94
3.93
0.88
1.00
2.04
0.99
2.38
f1 (ppm)

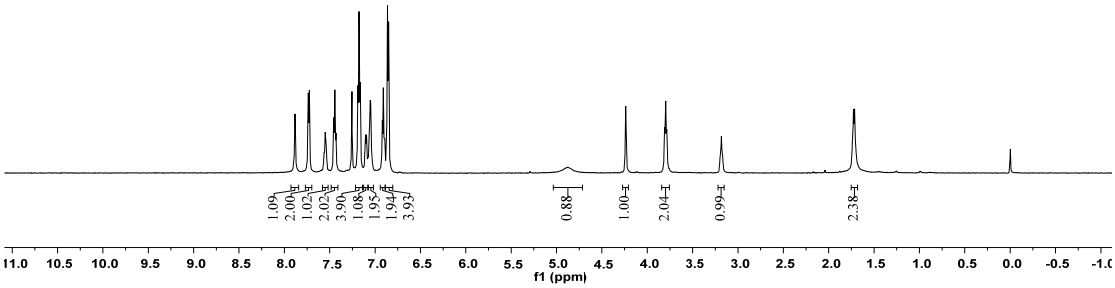

169.23
168.48
160.24
157.81
147.43
147.40
147.32
132.39
131.90
129.39
128.80
127.12
121.75
121.39
121.31
120.96
118.16
118.08
117.36
117.13
116.92
116.68
59.99
49.36
41.87
25.82


3ca
NHCOPh
HN
F
NPh2
O
O

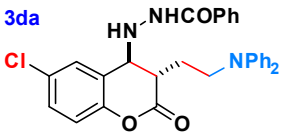
f1 (ppm)


7.88
7.74
7.72
7.56
7.55
7.54
7.46
7.44
7.43
7.19
7.18
7.16
7.11
7.09
7.06
7.05
7.05
6.92
6.91
6.90
6.86
6.85
4.88
4.24
3.81
3.80
3.79
3.20
3.18
3.17
1.74
1.73
1.71
1.70
0.00


3da
HN
NHCOPh
Cl
NPh2
O
O


1.09
2.00
1.02
2.02
3.90
1.08
1.95
1.94
3.93
0.88
1.00
2.04
0.99
2.38
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


168.99
168.50
149.96
147.30
132.40
131.90
130.56
130.06
129.81
129.40
128.81
127.12
121.77
121.46
120.97
118.13
59.85
49.32
41.93
25.82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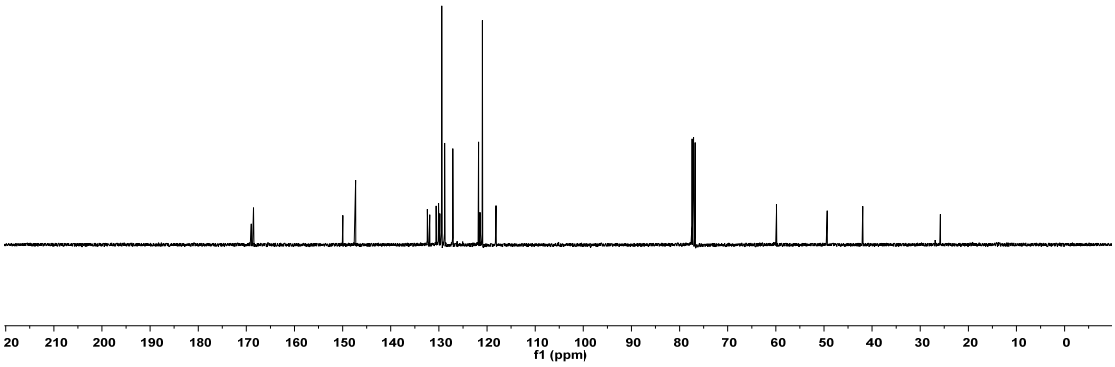
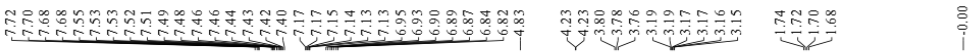
HN
NHCOPh
Cl
NPh2
O
O


220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.72
7.70
7.68
7.68
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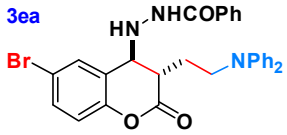
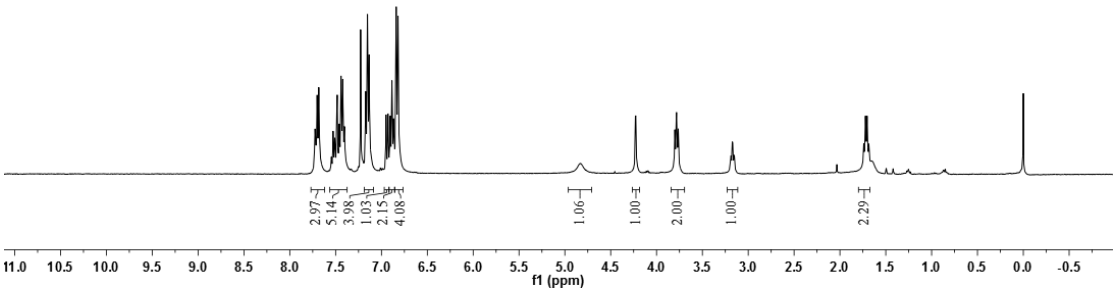
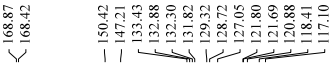
7.53
7.53
7.52
7.51
7.49
7.48
7.46
7.46
7.44
7.43
7.42
7.40
7.17
7.17
7.15
7.14
7.13
7.13
6.95
6.93
6.90
6.89
6.87
6.84
6.82
4.83
4.23
4.23
3.80
3.78
3.76
3.19
3.19
3.17
3.17
3.17
3.16
3.15
1.74
1.72
1.70
1.68
0.00


3ea
HN
NHCOPh
Br
NPh2
O
O


2.97
5.14
3.98
1.03
2.15
4.08
1.06
1.00
2.00
1.00
2.29
f1 (ppm)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168.42
150.42
147.21
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132.88
132.30
131.82
129.32
128.72
127.05
121.80
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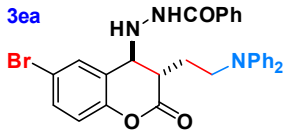
118.41
117.10
59.68
49.23
41.85
25.71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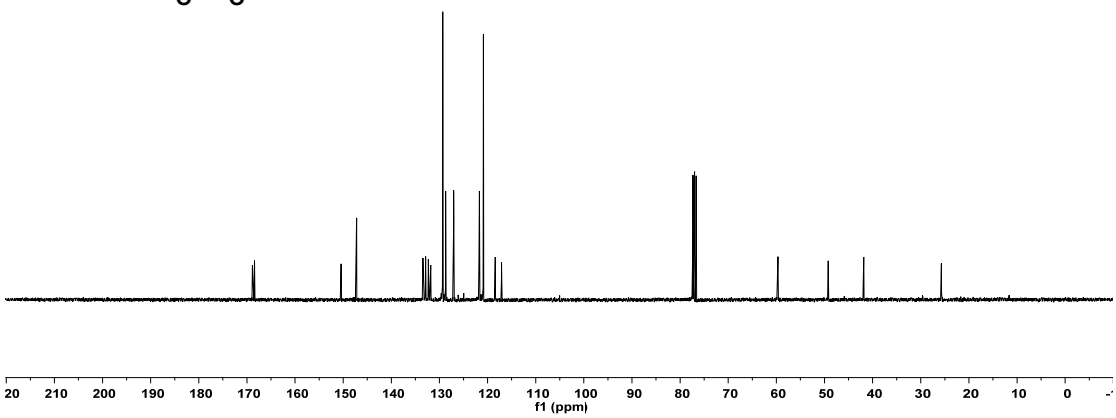
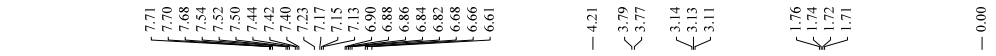
HN
NHCOPh
Br
NPh2
O
O


f1 (ppm)


7.71
7.70
7.68
7.54
7.52
7.50
7.44
7.42
7.40
7.23
7.17
7.15
7.13
6.90
6.88
6.86
6.84
6.82
6.68
6.66
6.61
4.21
3.79
3.77
3.14
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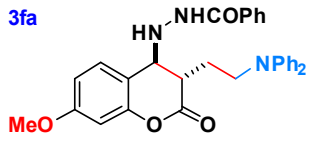
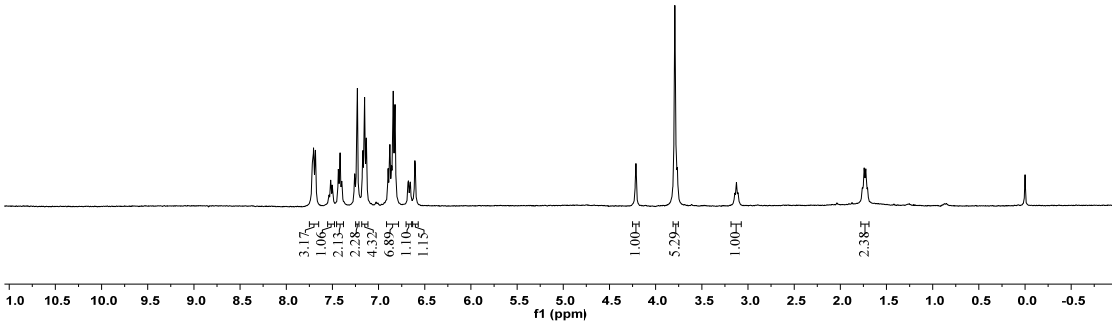
3.11
1.76
1.74
1.72
1.71
0.00


3fa
NHCOPh
HN
NPh2
MeO
O
O


3.17
1.06
2.13
2.28
4.32
6.89
1.10
1.15
1.00
5.29
1.00
2.38
1.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


169.25
167.95
161.03
152.14
147.10
132.01
131.90
130.65
129.14
128.54
126.86
121.44
120.74
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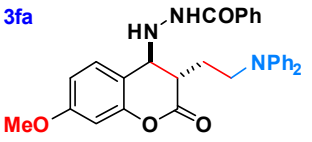
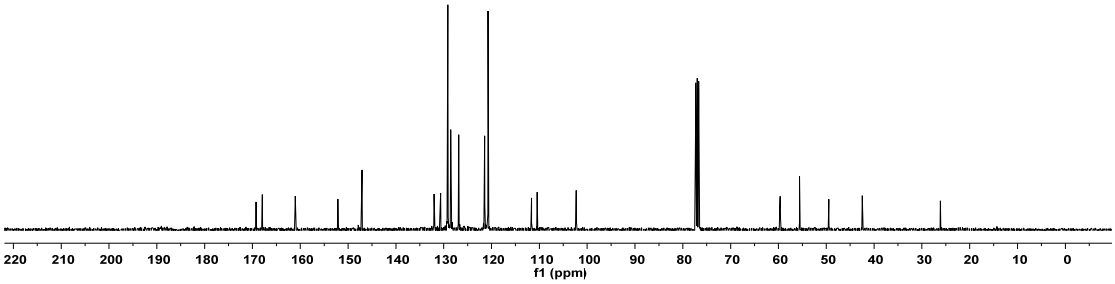
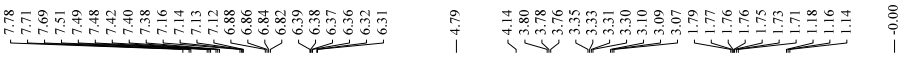
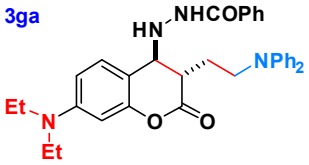
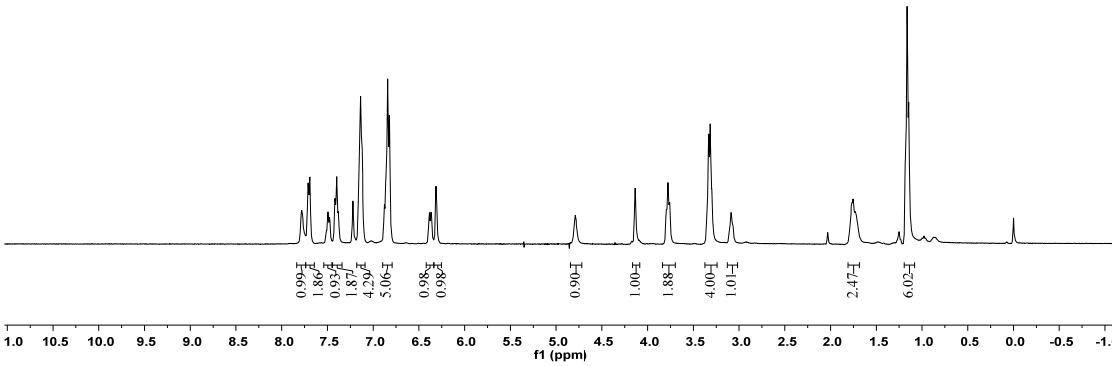
102.28
59.66
55.58
49.46
42.47
26.12

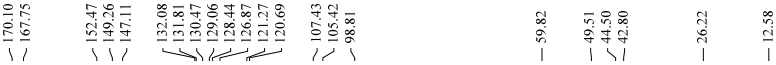

3fa
NHCOPh
HN
NPh2
MeO
O
O

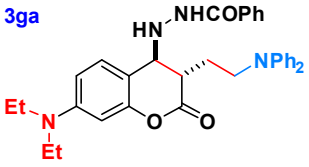

220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.78
7.71
7.69
7.51
7.49
7.48
7.42
7.40
7.38
7.16
7.14
7.13
7.12
6.88
6.86
6.84
6.82
6.39
6.38
6.37
6.36
6.32
6.31
4.79
4.14
3.80
3.78
3.76
3.35
3.33
3.31
3.30
3.10
3.09
3.07
1.79
1.77
1.76
1.76
1.75
1.73
1.71
1.18
1.16
1.14
0.00

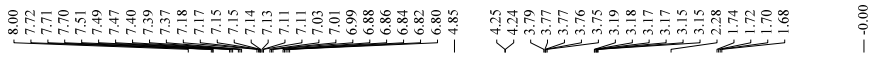

3ga
HN
NHCOPh
NPh2
Et
N
Et
O
O

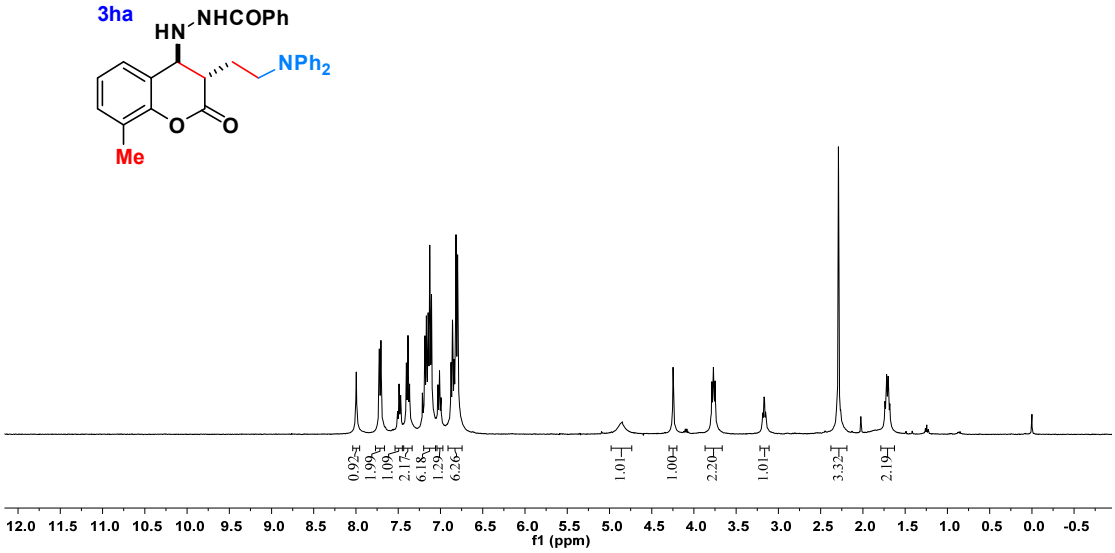

0.99
1.86
0.93
1.87
4.29
5.06
0.98
0.98
0.90
1.00
1.88
4.00
1.01
2.47
6.02
f1 (ppm)


170.10
167.75
152.47
149.26
147.11
132.08
131.81
130.47
129.06
128.44
126.87
121.27
120.69
107.43
105.42
98.81
59.82
49.51
44.50
42.80
26.22
12.58

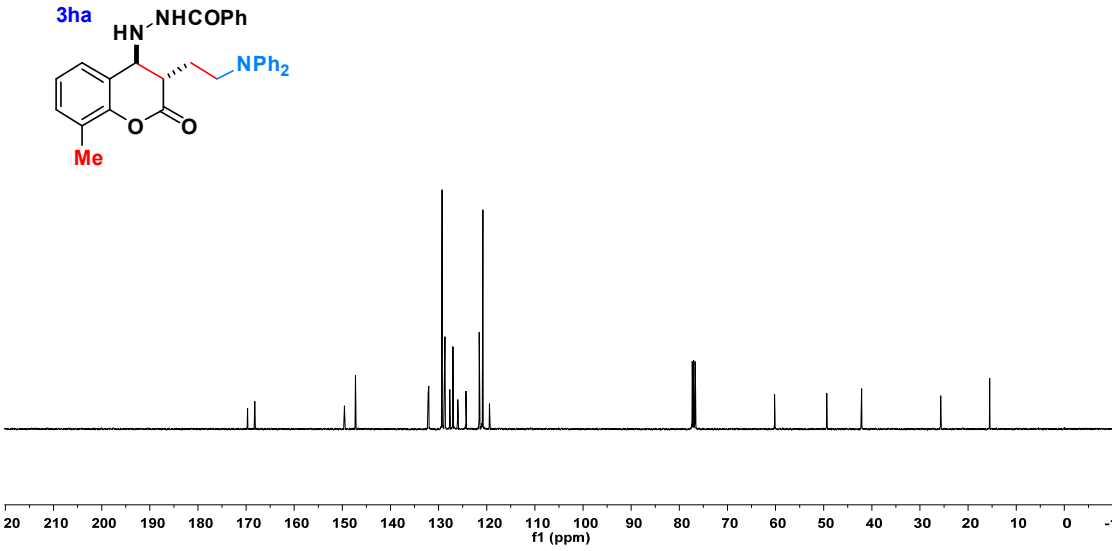

3ga
HN
NHCOPh
NPh2
Et
N
Et
O
O

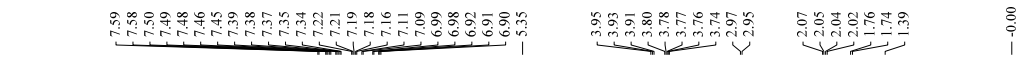
f1 (ppm)


8.00
7.72
7.71
7.70
7.51
7.49
7.47
7.40
7.39
7.37
7.18
7.17
7.15
7.15
7.14
7.13
7.11
7.11
7.03
7.01
6.99
6.88
6.86
6.84
6.82
6.80
4.85
4.25
4.24
3.79
3.77
3.77
3.76
3.75
3.19
3.18
3.17
3.17
3.15
3.15
2.28
1.74
1.72
1.70
1.68
-0.00


3ha
HN
NHCOPh
NPh2
O
O
Me
0.92
1.99
1.09
2.17
6.18
1.29
6.26
1.01
1.00
2.20
1.01
3.32
2.19
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


169.70
168.20
149.56
147.29
132.18
132.08
132.05
129.28
128.69
127.70
127.02
126.03
124.32
121.56
120.82
119.42
60.20
49.37
42.11
25.69
15.50


3ha
HN
NHCOPh
NPh2
O
O
Me
20 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.48
7.46
7.45
7.39
7.38
7.37
7.35
7.34
7.22
7.21
7.19
7.18
7.16
7.11
7.09
6.99
6.98
6.92
6.91
6.90
5.35
3.95
3.93
3.91
3.80
3.78
3.77
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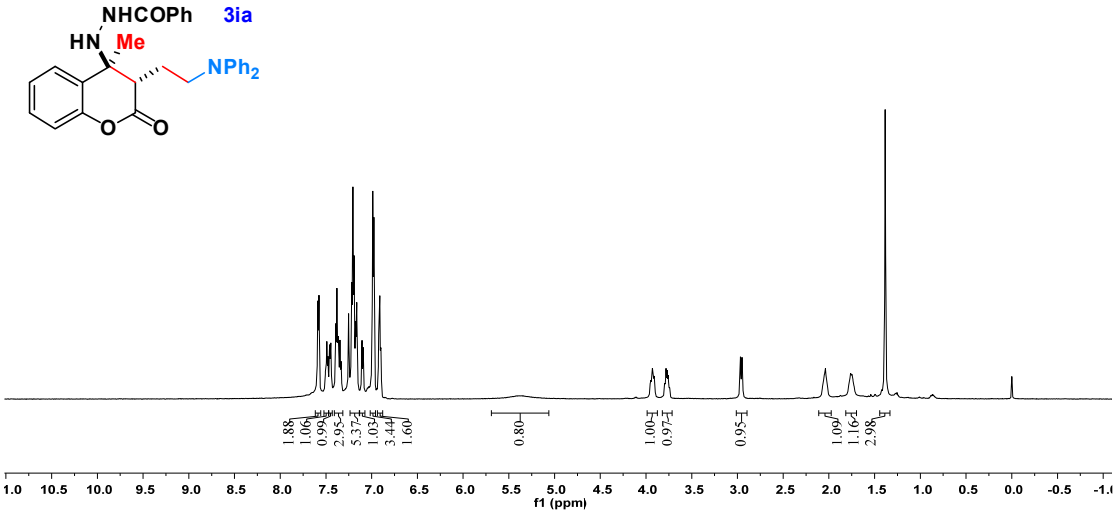
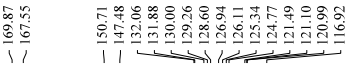
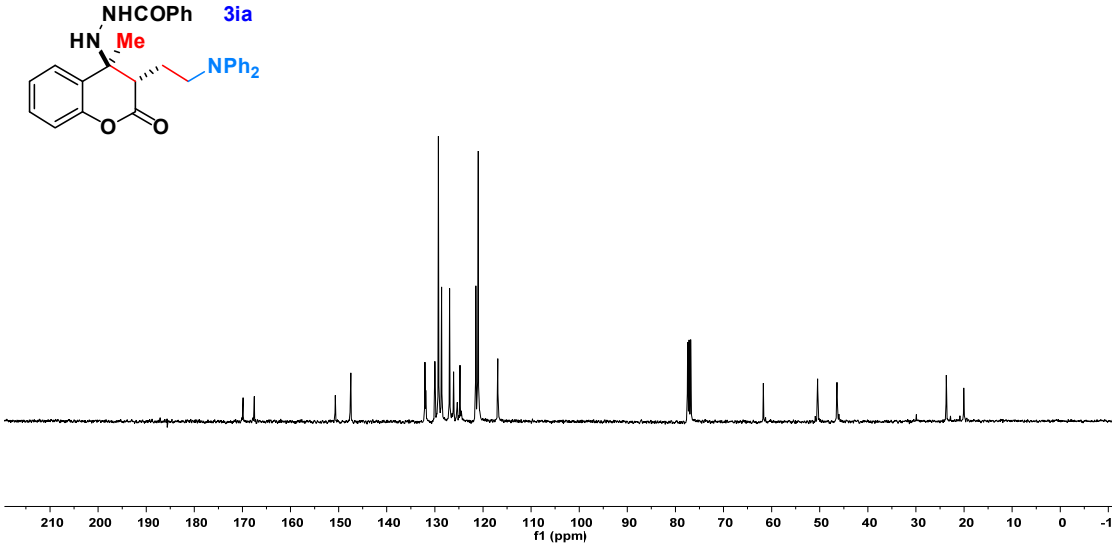
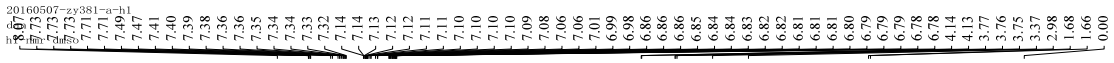
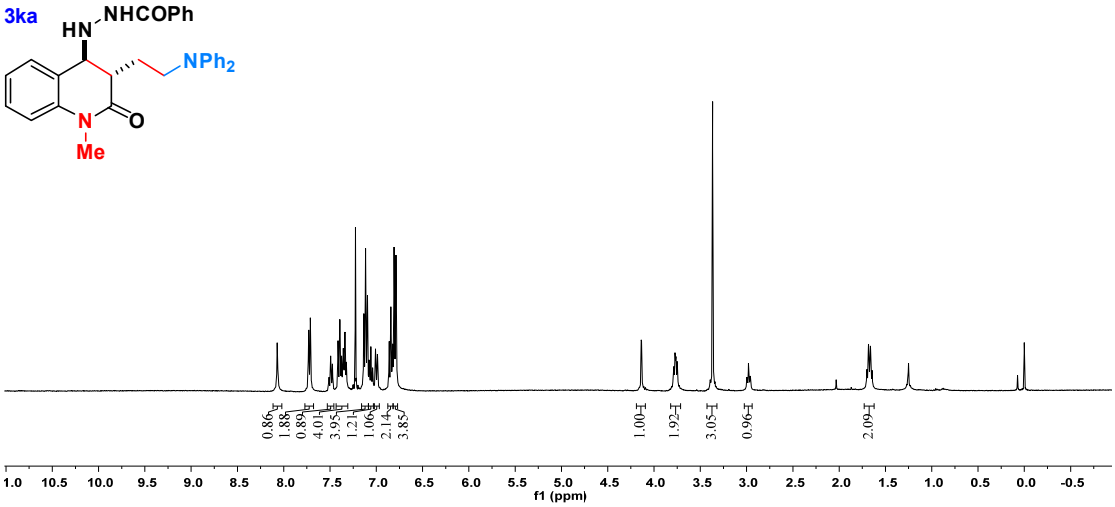
2.04
2.02
1.76
1.74
1.39
0.00

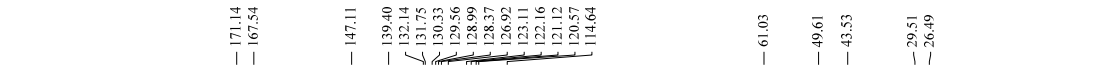

NHCOPh
3ia
HN
Me
NPh2
O
O
1.88
1.06
0.99
2.95
5.37
1.03
3.44
1.60
0.80
1.00
0.97
0.95
1.09
1.16
2.98
f1 (ppm)

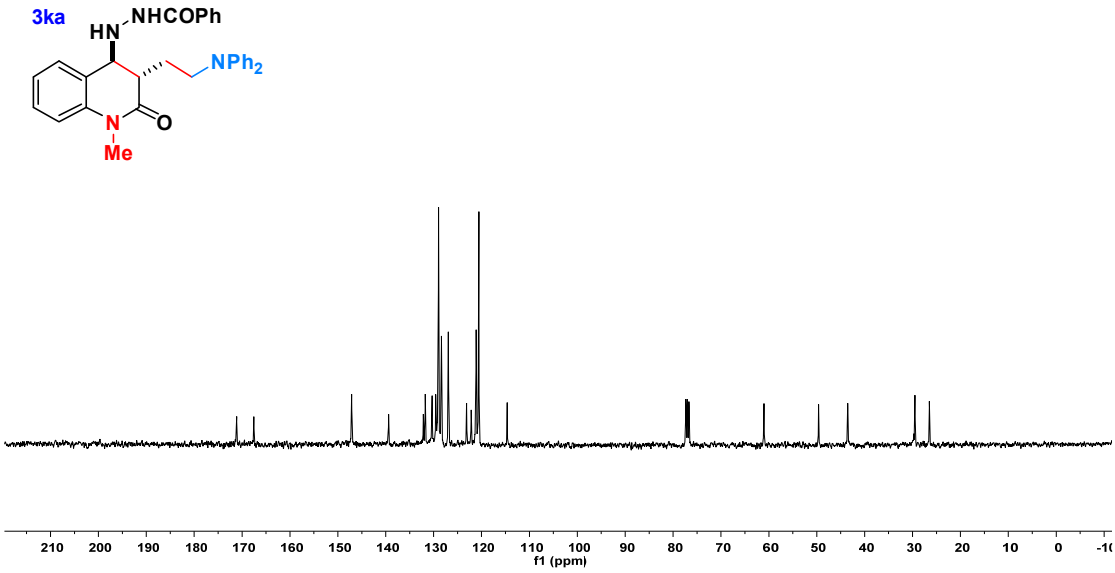

169.87
167.55
150.71
147.48
132.06
131.88
130.00
129.26
128.60
126.94
126.11
125.34
124.77
121.49
121.10
120.99
116.92
61.71
50.43
46.43
23.69
20.05

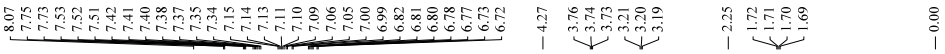

NHCOPh
3ia
HN
Me
NPh2
O
O
f1 (ppm)

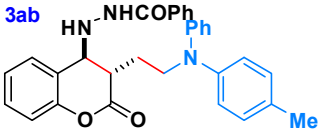


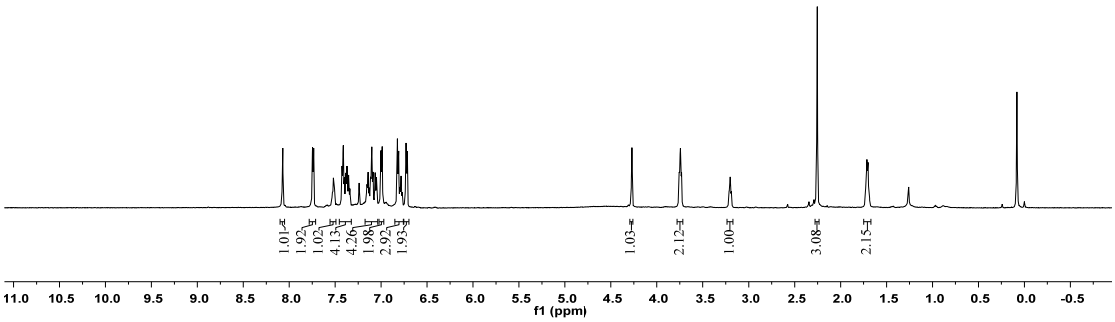

3ka
NHCOPh
HN
NPh2
N
O
Me
0.86
1.88
0.89
4.01
3.95
1.21
1.06
2.14
3.85
1.00
1.92
3.05
0.96
2.09
f1 (ppm)

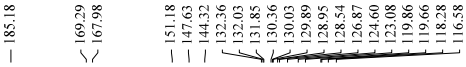



3ka
NHCOPh
HN
NPh2
N
O
Me
f1 (ppm)


8.07
7.75
7.73
7.53
7.52
7.51
7.42
7.41
7.40
7.38
7.37
7.35
7.34
7.15
7.14
7.13
7.11
7.10
7.09
7.06
7.05
7.00
6.99
6.82
6.81
6.80
6.78
6.77
6.73
6.72
4.27
3.76
3.74
3.73
3.21
3.20
3.19
2.25
1.72
1.71
1.70
1.69
0.00


3ab
HN
NHCOPh
Ph
N
O
O
Me


1.01
1.92
1.02
4.13
4.26
1.98
2.92
1.93
1.03
2.12
1.00
3.08
2.15
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


185.18
169.29
167.98
151.18
147.63
144.32
132.36
132.03
131.85
130.36
130.03
129.89
128.95
128.54
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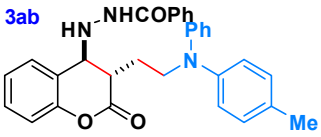
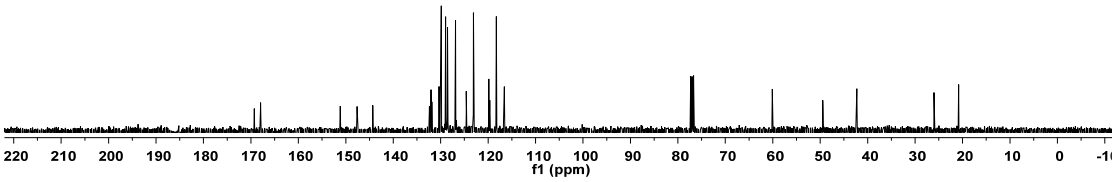
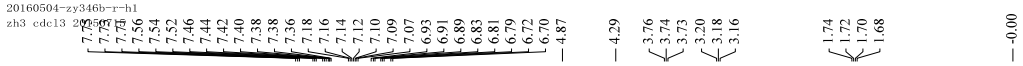
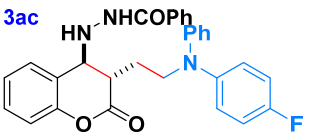
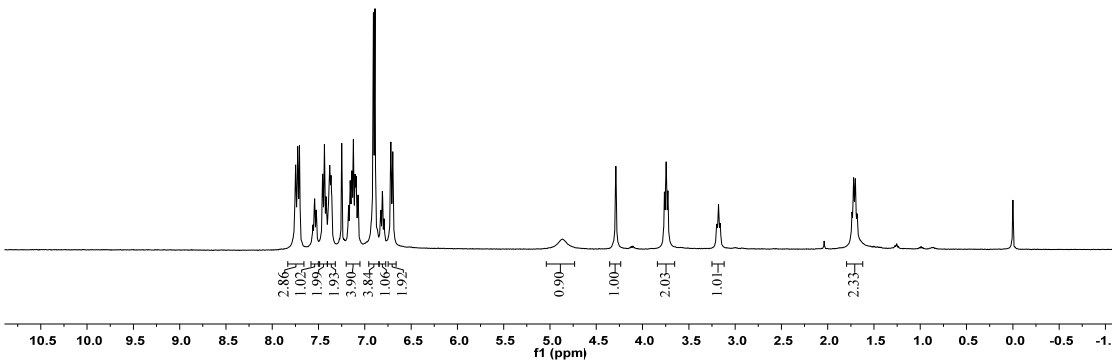
116.58
60.10
49.47
42.28
26.00
20.83

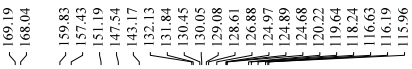

3ab
HN
NHCOPh
Ph
N
O
O
Me

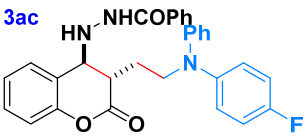

220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

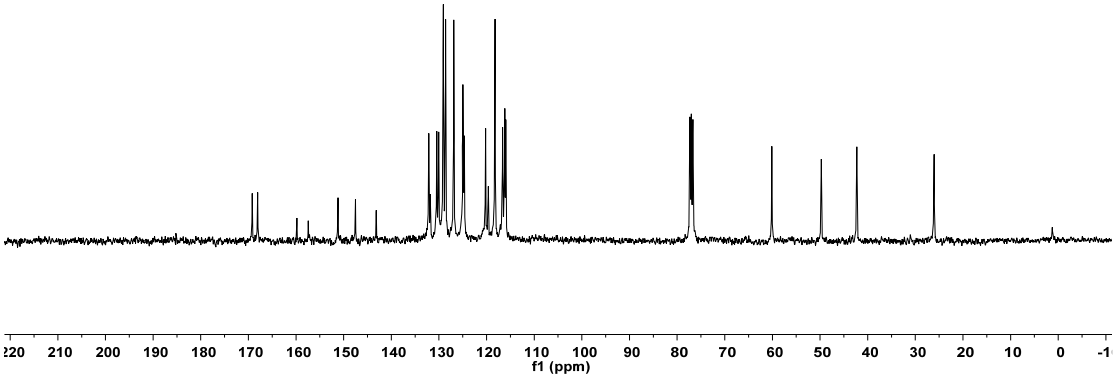

20160504-zy346b-r-h1
zh3 cdcl3 298
7.75
7.74
7.56
7.54
7.52
7.46
7.44
7.42
7.40
7.38
7.36
7.18
7.16
7.14
7.12
7.10
7.09
7.07
6.93
6.91
6.89
6.83
6.81
6.79
6.72
6.70
4.87
4.29
3.76
3.74
3.73
3.20
3.18
3.16
1.74
1.72
1.70
1.68
0.00

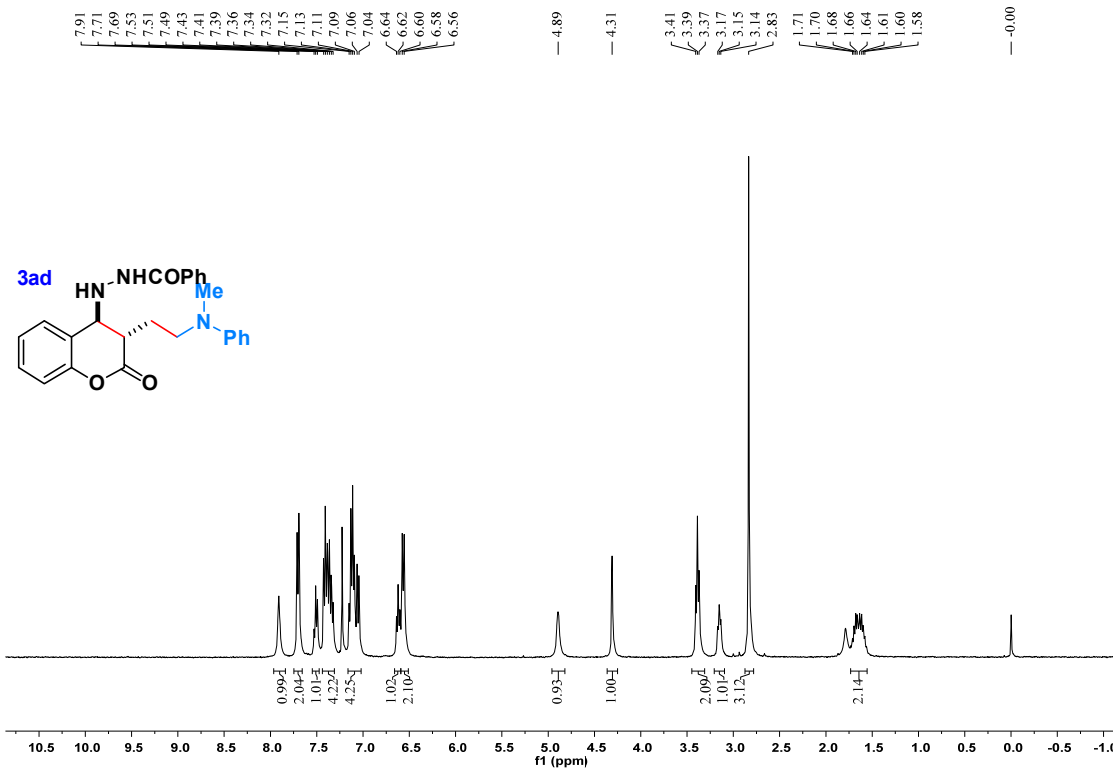

3ac
HN
NHCOPh
Ph
N
O
O
F

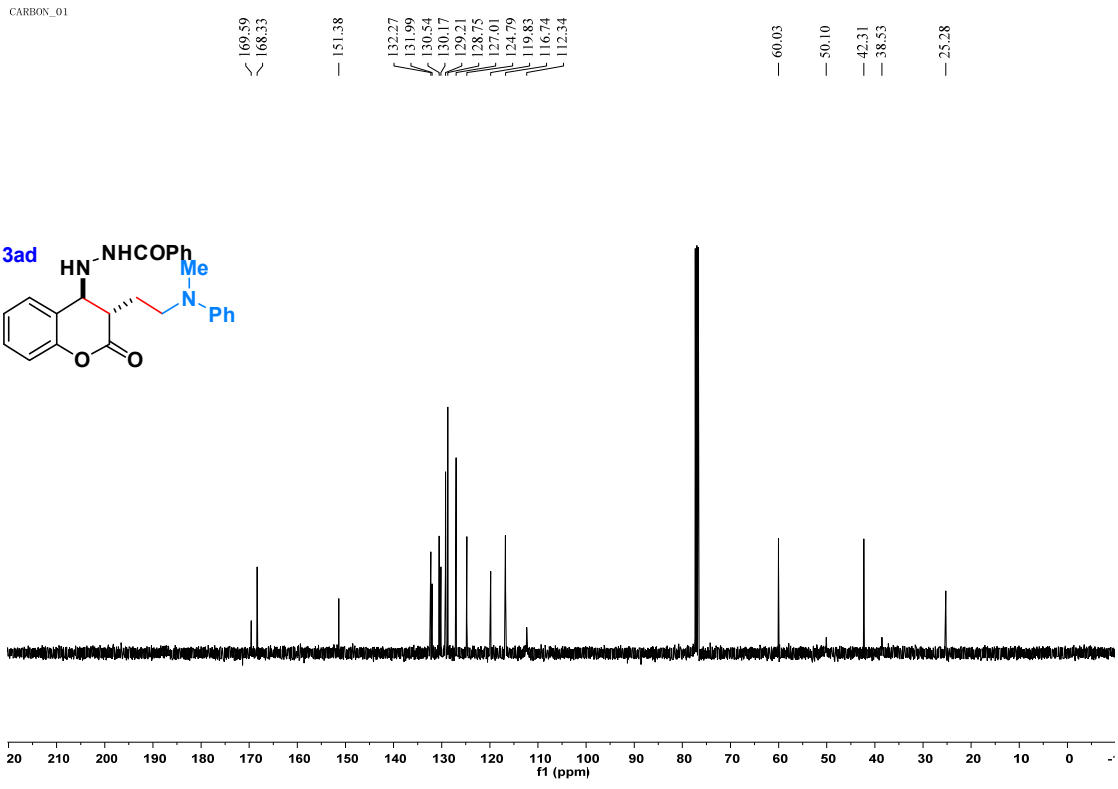

2.86
1.02
1.99
1.93
3.90
3.84
1.06
1.92
0.90
1.00
2.03
1.01
2.33
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

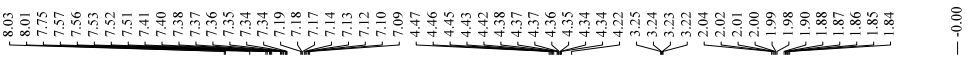

169.19
168.04
159.83
157.43
151.19
147.54
143.17
132.13
131.84
130.45
130.05
129.08
128.61
126.88
124.97
124.89
124.68
120.22
119.64
118.24
116.63
116.19
115.96
60.11
49.74
42.28
26.07

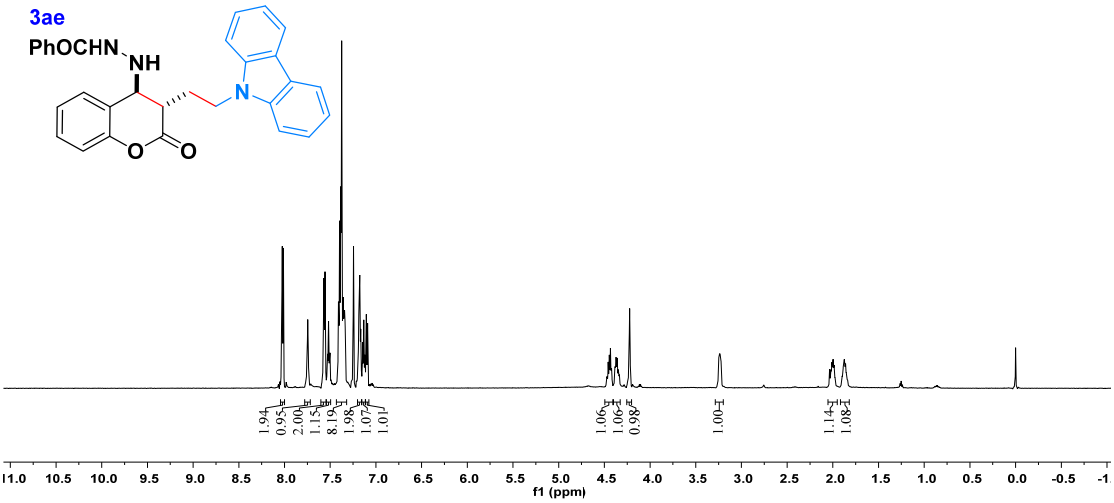

3ac
HN
NHCOPh
Ph
N
O
O
F

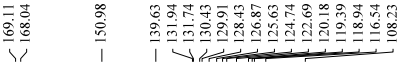

220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

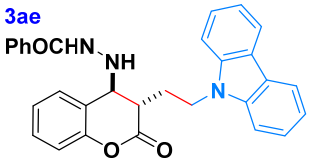

7.91
7.71
7.69
7.53
7.51
7.49
7.43
7.41
7.39
7.36
7.34
7.32
7.15
7.13
7.11
7.09
7.06
7.04
6.64
6.62
6.60
6.58
6.56
4.89
4.31
3.41
3.39
3.37
3.17
3.15
3.14
2.83
1.71
1.70
1.68
1.66
1.64
1.61
1.60
1.58
-0.00
3ad
HN
NHCOPh
Me
N
Ph
O
O
0.99
2.04
1.01
4.22
4.25
1.02
2.10
0.93
1.00
2.09
1.01
3.12
2.14
f1 (ppm)

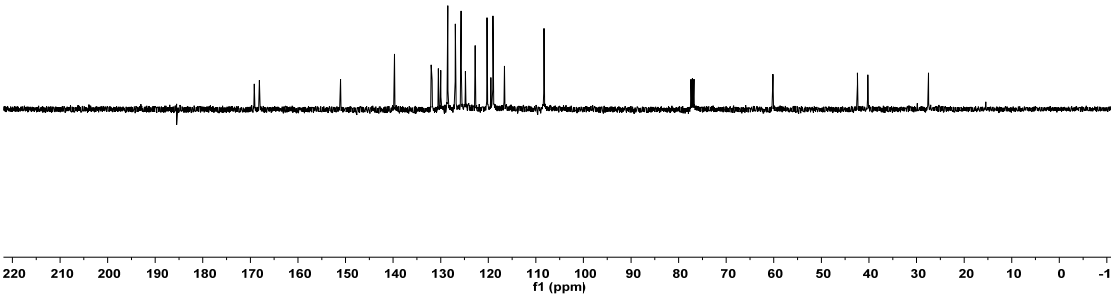

CARBON_01
169.59
168.33
151.38
132.27
131.99
130.54
130.17
129.21
128.75
127.01
124.79
119.83
116.74
112.34
60.03
50.10
42.31
38.53
25.28
3ad
HN
NHCOPh
Me
N
Ph
O
O
f1 (ppm)

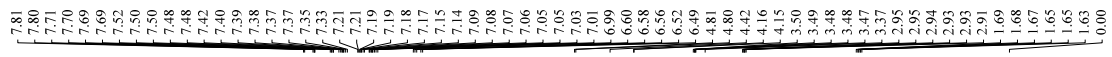

8.03
8.01
7.75
7.57
7.56
7.53
7.52
7.51
7.41
7.40
7.38
7.37
7.36
7.35
7.34
7.34
7.19
7.18
7.17
7.14
7.13
7.12
7.10
7.09
4.47
4.46
4.45
4.43
4.42
4.38
4.37
4.37
4.36
4.35
4.34
4.34
4.22
3.25
3.24
3.23
3.22
2.04
2.02
2.01
2.00
1.99
1.98
1.90
1.88
1.87
1.86
1.85
1.84
-0.00

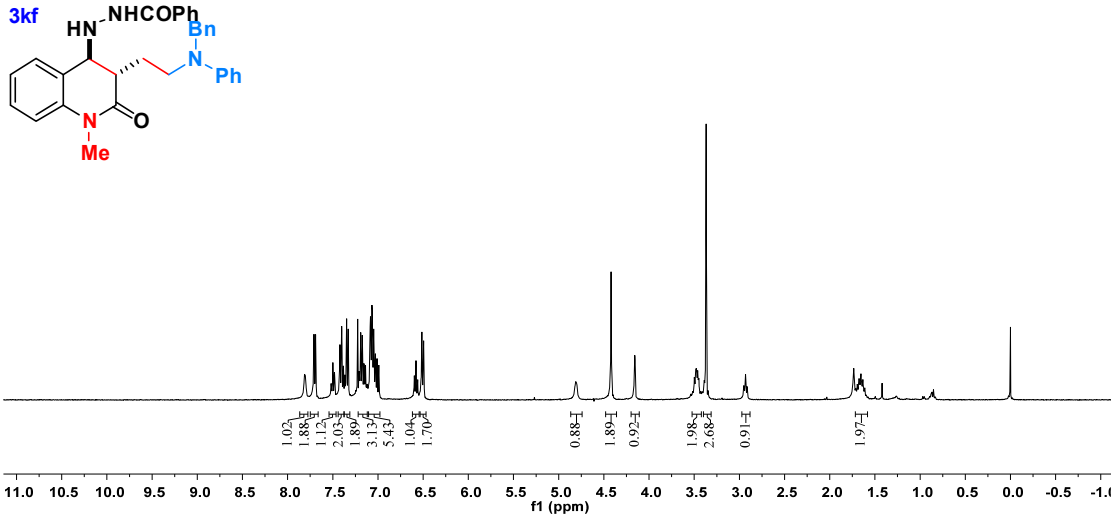

3ae
PhOCHN
NH
N
O
O
1.94
0.95
2.00
1.15
8.19
1.98
1.07
1.01
1.06
1.06
0.98
1.00
1.14
1.08
f1 (ppm)

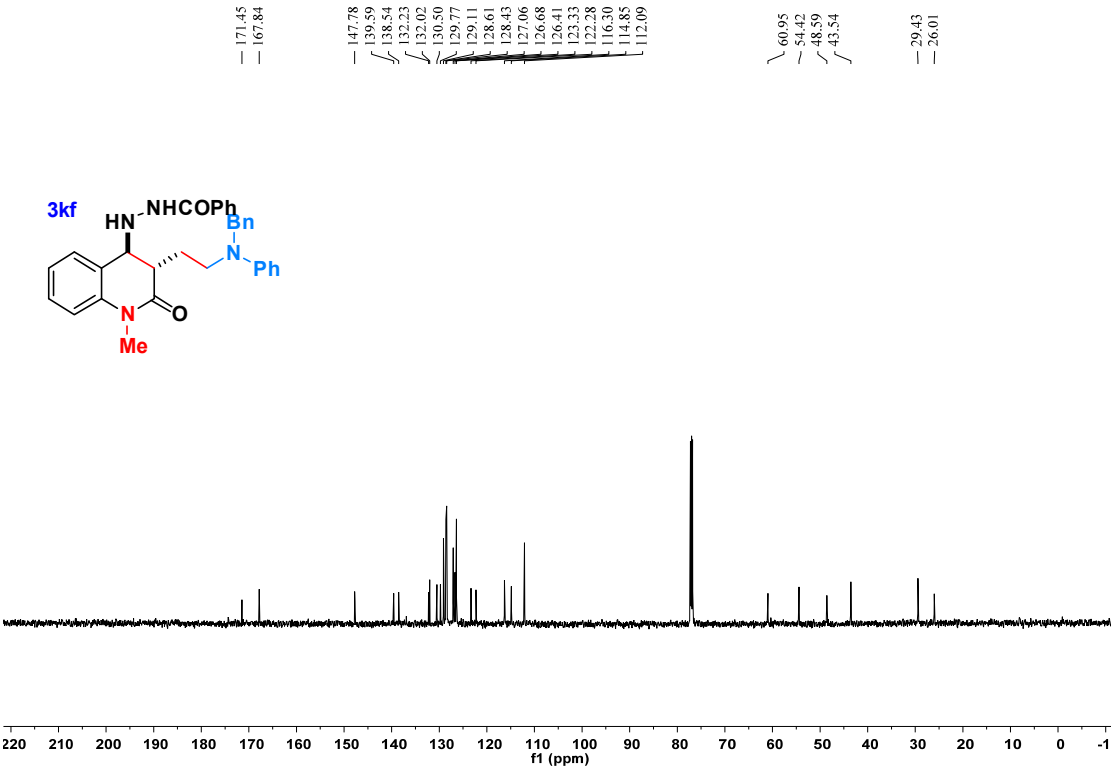

169.11
168.04
150.98
139.63
131.94
131.74
130.43
129.91
128.43
126.87
125.63
124.74
122.69
120.18
119.39
118.94
116.54
108.23
60.11
42.34
40.17
27.42

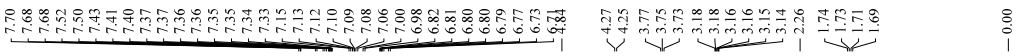

3ae
PhOCHN
NH
N
O
O

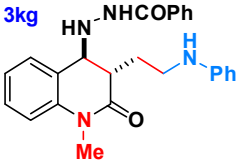

f1 (ppm)

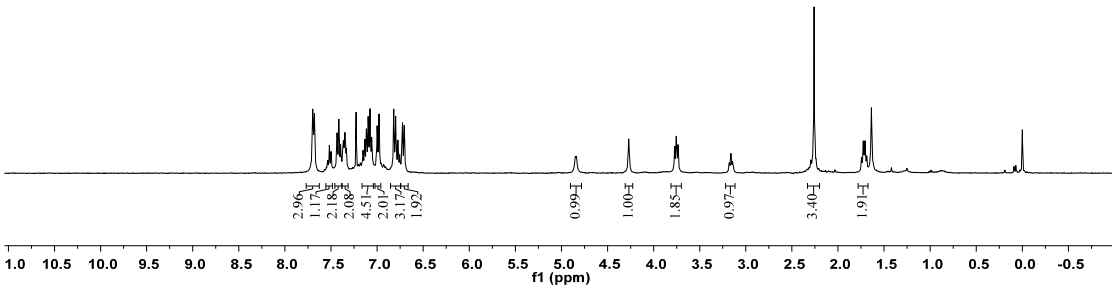


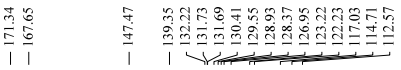

3kf
HN
NHCOPh
Bn
N
Ph
N
O
Me
f1 (ppm)

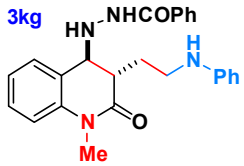

3kf
HN
NHCOPh
Bn
N
Ph
N
O
Me
f1 (ppm)

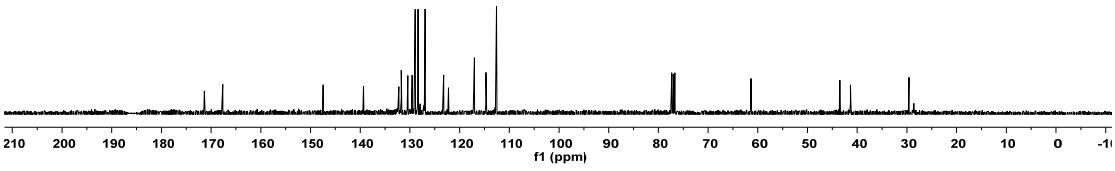


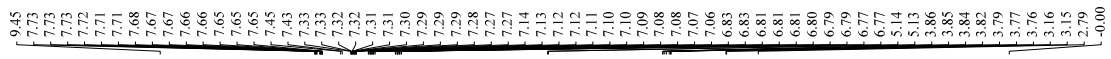

3kg
NHCOPh
HN
H
N
Ph
N
Me
O

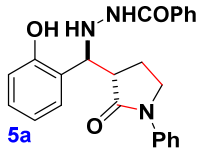

2.96
1.17
2.18
2.08
4.51
2.01
3.17
1.92
0.99
1.00
1.85
0.97
3.40
1.91
1.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

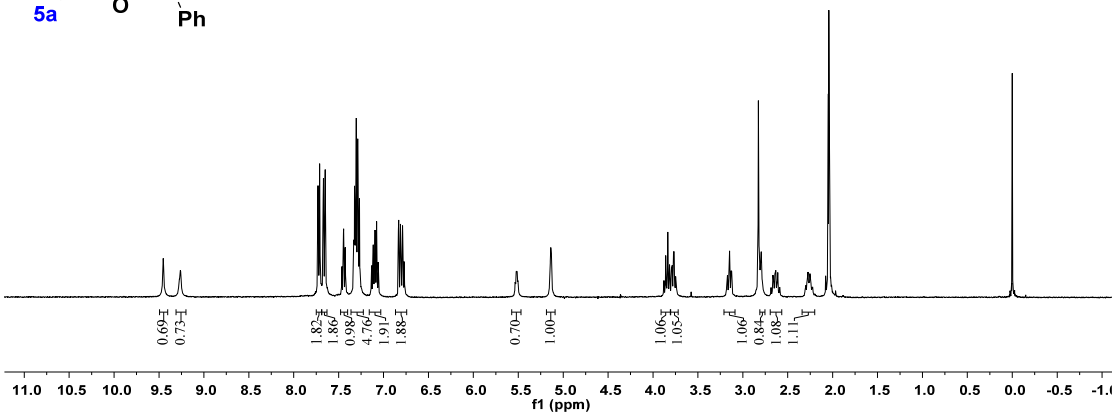


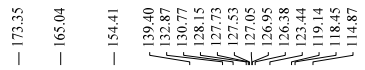

3kg
NHCOPh
HN
H
N
Ph
N
Me
O

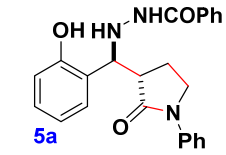

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

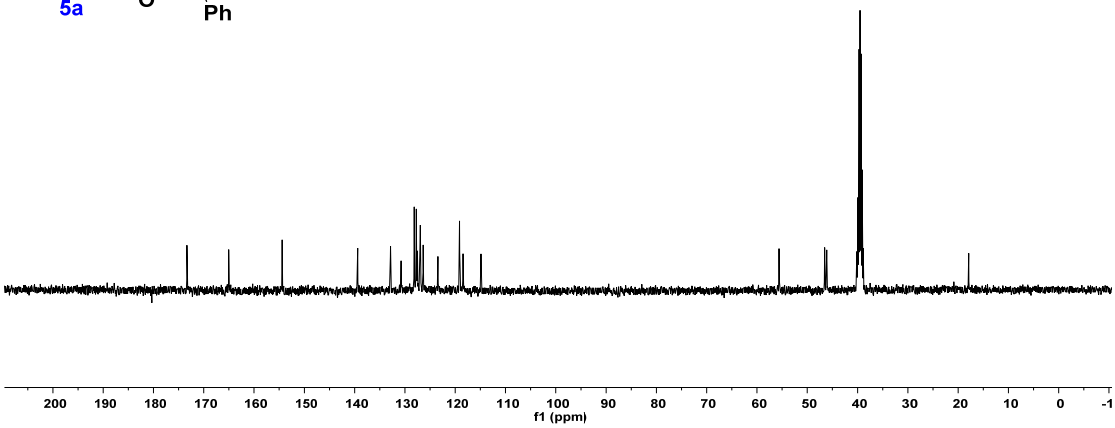


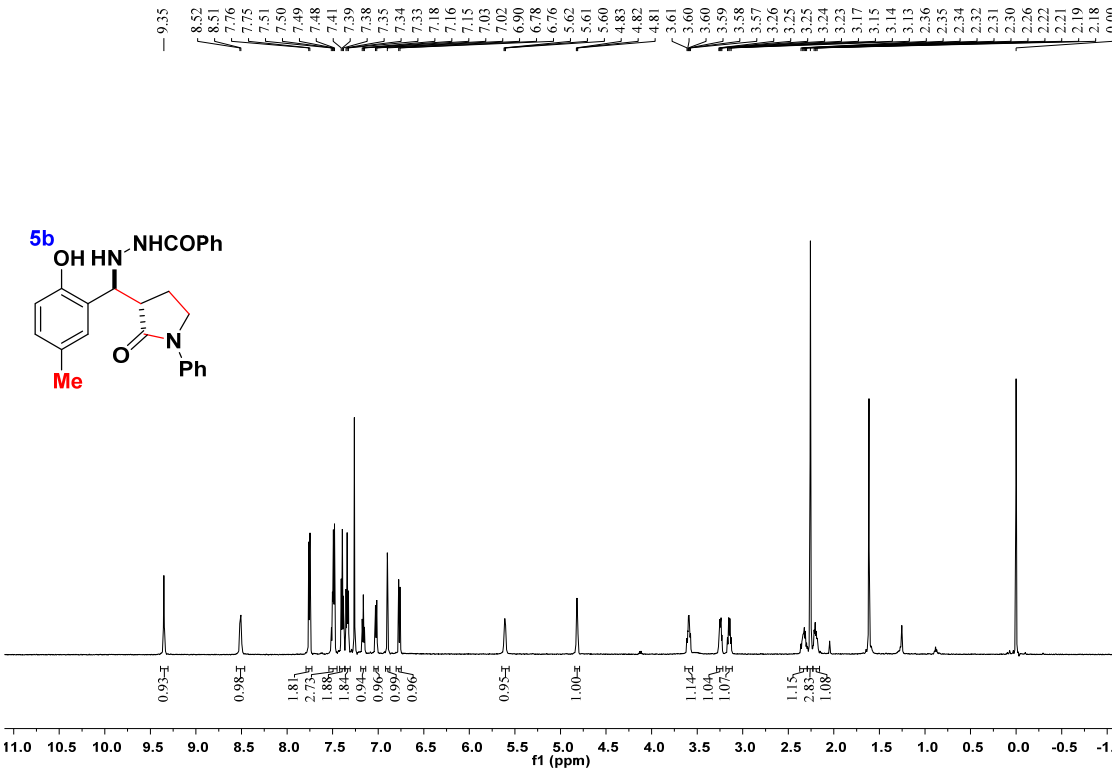

OH HN
NHCOPh
O
N
Ph
5a

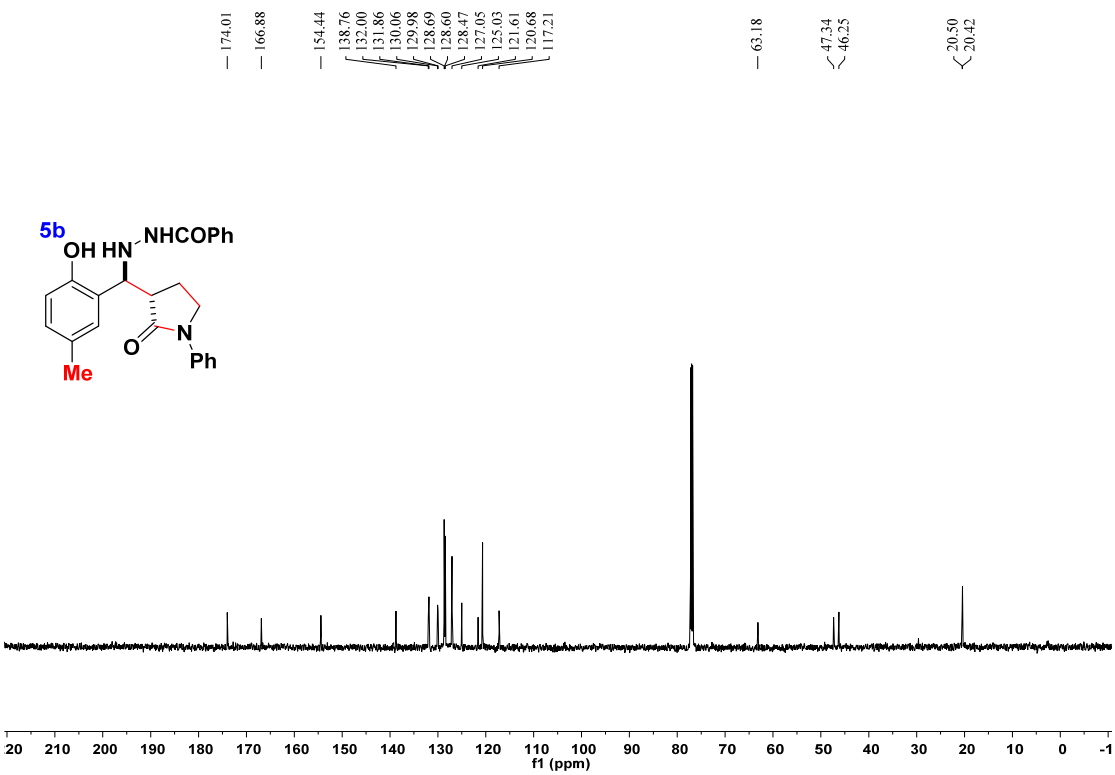

0.69
0.73
1.82
1.86
0.98
4.76
1.91
1.88
0.70
1.00
1.06
1.05
1.06
0.84
1.08
1.11
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

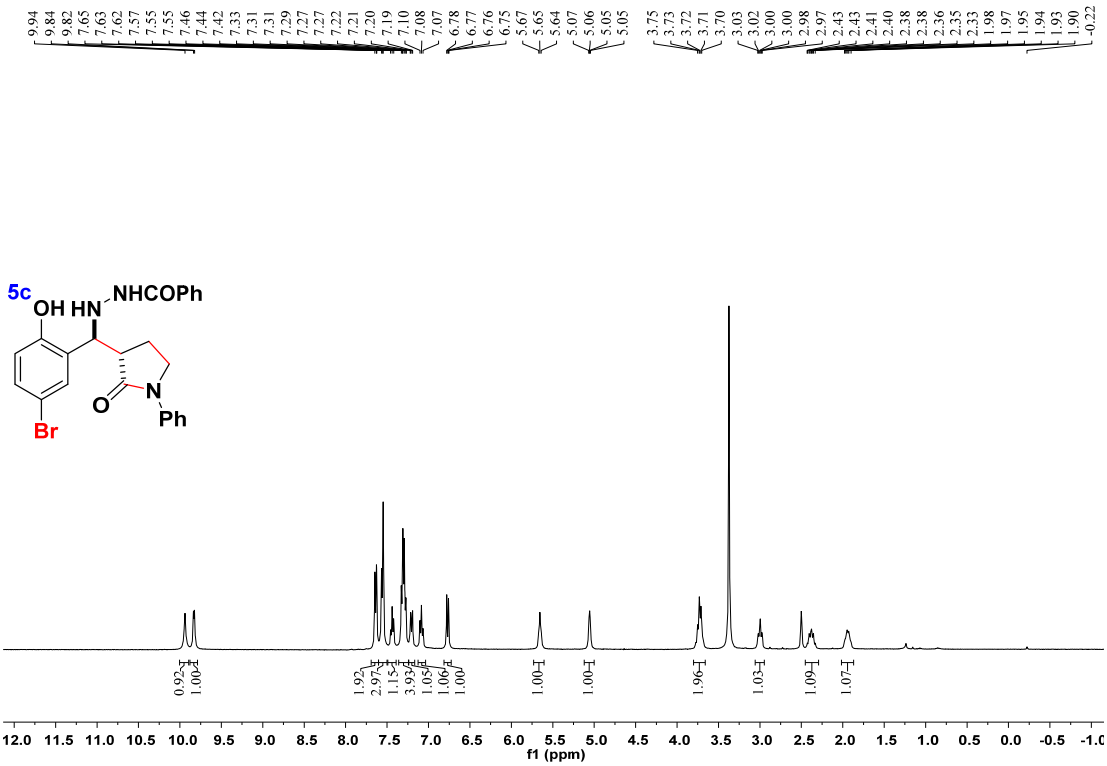


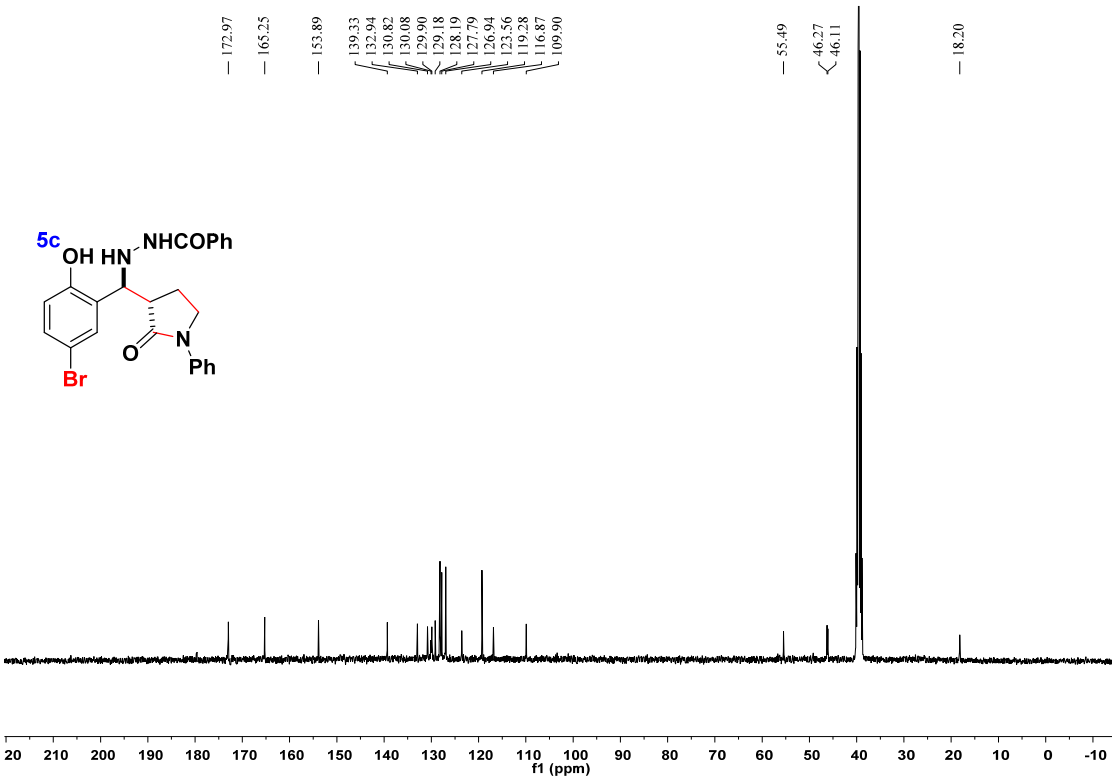

OH HN
NHCOPh
O
N
Ph
5a


200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


5b
OH HN
NHCOPh
O
N
Ph
Me
f1 (ppm)


5b
OH HN
NHCOPh
O
N
Ph
Me
f1 (ppm)


5c
OH HN
NHCOPh
O
N
Ph
Br
f1 (ppm)


5c
OH HN
NHCOPh
O
N
Ph
Br
f1 (ppm)