Supporting Information for

Expedient Synthesis of Pyrroloquinolinones by Rh-Catalyzed Annulation of *N*-Carbamoyl Indolines with Alkynes through a Directed C-H Functionalization/C-N Cleavage Sequence

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Table of contents **Page No.**

General information

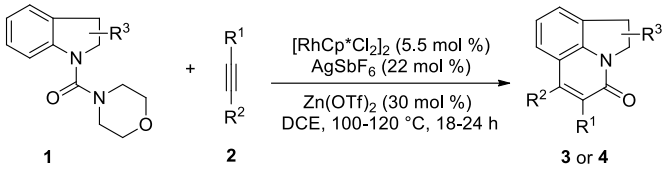
$[RhCp^*Cl_2]_2$, $Zn(OTf)_2$, $Cu(OTf)_2$ and solvents were purchased from commercial suppliers and used as received unless otherwise noted. All reactions were carried out using 4 mL sample vial or standard Schlenk technic. Reactions were monitored through thin layer chromatography [Merck 60 F254 precoated silica gel plate (0.2 mm thickness)]. Subsequent to elution, spots were visualized using UV radiation (254 nm) on Spectroline Model ENF-24061/F 254 nm. Further visualization was possible using basic solution of potassium permanganate. Flash chromatography was performed using Merck silica gel 60 with distilled solvents. HRMS spectra were recorded on a Waters Q–Tof Permier Spectrometer. ^1H NMR and ^{13}C NMR spectra were recorded using Bruker Avance 400 MHz spectrometers. Chemical shifts for ^1H NMR and ^{13}C NMR spectra were recorded using Bruker Avance 400 MHz spectrometers. Chemical shifts for ^1H NMR spectra are reported as δ in units of parts per million (ppm) downfield from $SiMe_4$ (δ 0.0) and relative to the signal of $SiMe_4$ (δ 0.00, singlet). Multiplicities were given as: s (singlet); brs (broad singlet); d (doublet); t (triplet); q (quartet); dd (doublets of doublet); ddd (doublets of doublets of doublet); td (triplet of doublet); m (multiplets); ddt (doublet of doublet of triplet) and etc. Coupling constants are reported as a *J* value in Hz. Carbon nuclear magnetic resonance spectra (^{13}C NMR) are reported as δ in units of parts per million (ppm) downfield from $SiMe_4$ (δ 0.0) and relative to the signal of chloroform-d (δ 77.00, triplet).

Experimental section

Substrate synthesis

Starting materials **1** were synthesized using reported method.[1]

Rhodium-catalyzed annulation of *N*-carbamoyl indolines with alkynes

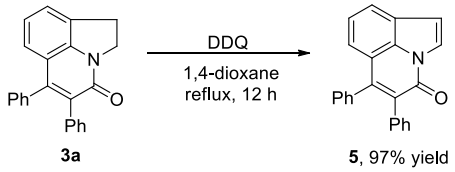


General reaction procedure A: The indoline **1** (0.21 mmol, 1.5 equiv), alkyne (0.14 mmol, 1.0 equiv), $[RhCp^*Cl_2]_2$ (0.0077 mmol, 5.5 mol %), $AgSbF_6$ (0.0308 mmol, 22 mol %), $Zn(OTf)_2$ (0.042 mmol, 30 mol %) and DCE (1.0 mL) were placed in a 4 mL sample vial under N_2. After stirring at 100 ℃ for 18 hours. Removal of the solvent *in vacuo* and purification by column chromatography on silica gel (eluted by hexane/ethyl acetate) afforded the desired product **3** or **4**.

General reaction procedure B: The indoline **1** (0.21 mmol, 1.5 equiv), alkyne (0.14 mmol, 1.0 equiv), $[RhCp^*Cl_2]_2$ (0.0077 mmol, 5.5 mol %), $AgSbF_6$ (0.0308 mmol, 22 mol %), $Zn(OTf)_2$ (0.042 mmol, 30 mol %) and DCE (1.0 mL) were placed in a 4 mL sample vial under N_2. After stirring at 120 ℃ for 24 hours. Removal of the solvent *in vacuo* and purification by column chromatography on silica gel (eluted by hexane/ethyl acetate) afforded the desired product **3** or **4**.

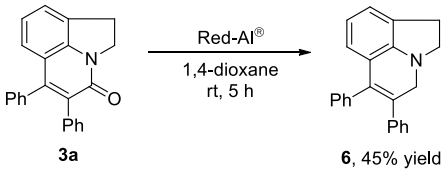
Derivatization of the pyrroloquinolinone products

Reaction procedure C:



Synthetic procedure: **3a** (0.1 mmol) and DDQ (0.2 mmol) were placed in a 10 mL Schlenk tube. 1,4-dioxane (1.0 mL) was added to the reaction vessel. The solution was then refluxed with stirring for 12 h. Removal of the solvent *in vacuo* and purification by column chromatography on silica gel (eluted by hexane/ethyl acetate) afforded the desired product **5** (31.1 mg, 0.097 mmol).

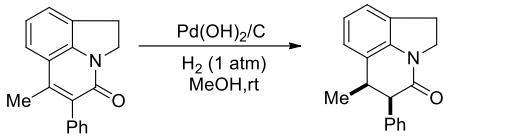
Reaction procedure D:



3a **6**, 45% yield

Synthetic procedure: To a solution of **3a** (0.1 mmol) in 1,4-dioxane (1.0 mL) was added dropwise a solution of Red-Al® in toluene (65% w/w 1.0 mmol, 10.0 equiv) and the mixture was stirred at room temperature for 5 h. Removal of the solvent *in vacuo*. Excess hydride was quenched with water (1.0 mL) and 20% sodium hydroxide solution (0.3 mL). The aqueous mixture was extracted with dichloromethane (5.0 mL × 2) and the combined organic fractions were dried over Na_2SO_4, filtered. Removal of the solvent *in vacuo* and purification by column chromatography on silica gel (eluted by hexane/ethyl acetate) afforded the desired product **6** (13.9 mg, 0.045 mmol).

Reaction procedure E:



3b *cis*-**7**, 52% yield
(40% of **3e** recovery)

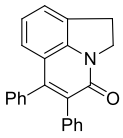
Synthetic procedure: **3b** (0.1 mmol) and $Pd(OH)_2/C$ (10.0 mg) were placed in a 10 mL Schlenk tube. Methanol (1.0 mL) was added. Then the mixture was stirred at room temperature under hydrogen (balloon) overnight. The resultant solution was filtered over celite and washed with ethyl acetate. Removal of the solvent *in vacuo* and purification by column chromatography on silica gel (eluted by hexane/ethyl acetate) afforded *cis*-**7** (13.7 mg, 0.052 mmol).

Reference:

[1] L. Jiao, M. Oestreich, *Org. Lett.* 2013, **15**, 5374.

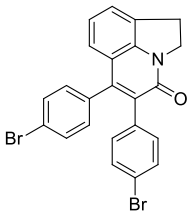
Characterization data of products

5,6-Diphenyl-1H-pyrrolo[3,2,1-*ij*]quinolin-4(2H)-one (3a)



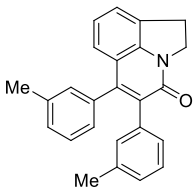
Following the general reaction procedure A, **3a** was obtained as a white solid (33 mg, 0.10 mmol, Yield: 73%); m.p. = 203-205 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (dd, *J* = 6.8, 1.2 Hz, 1H), 7.31 – 7.25 (m, 3H), 7.22 – 7.07 (m, 9H), 4.70 – 4.47 (t, *J* = 8.1 Hz, 2H), 3.52 (t, *J* = 8.1 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.27, 146.91, 141.64, 135.92, 135.45, 133.30, 130.90 × 2, 130.38, 129.72 × 2, 127.92 × 2, 127.49, 127.36 × 2, 126.84, 124.73, 123.63, 122.92, 118.48, 47.27, 27.13; HRMS (ESI): m/z calculated for $[C_{23}H_{18}NO]^+$ [M + H]$^+$: 324.1388, Found: 324.1381; FTIR (NaCl): ν 3053, 2924, 1645, 1637, 1616, 1604, 1338, 1265, 1072 cm^{-1}

5,6-Bis(4-bromophenyl)-1H-pyrrolo[3,2,1-*ij*]quinolin-4(2H)-one (3b)



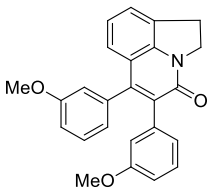
Following the general reaction procedure A, **3b** was obtained as a white solid (47.6 mg, 0.10 mmol, Yield: 71%); m.p. = 266-268 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (d, J = 8.4 Hz, 2H), 7.37 (d, *J* = 6.4 Hz, 1H), 7.33 (d, *J* = 8.4 Hz, 2H), 7.11 – 7.04 (m, 2H), 7.01 (d, *J* = 2.4 Hz, 2H), 6.98 (d, *J* = 2.4 Hz, 2H), 4.53 (t, *J* = 8.1 Hz, 2H), 3.49 (t, *J* = 8.1 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.78, 145.93, 141.73, 134.56, 134.10, 132.60 × 2, 132.20, 131.54× 2, 131.33 × 2, 130.87 × 2, 130.63, 125.25, 123.38, 123.30, 122.12, 121.46, 117.98, 47.41, 27.14; HRMS (ESI): m/z calculated for $[C_{23}H_{16}Br_2NO]^+$ [M + H]$^+$: 479.9599, Found: 479.9597; FTIR (NaCl): ν 3053, 2983, 1639, 1587, 1490, 1419, 1265, 1074, 1010, 894 cm^{-1}

5,6-Di-m-tolyl-1H-pyrrolo[3,2,1-*ij*]quinolin-4(2H)-one (3c)



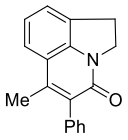
Following the general reaction procedure A, **3c** was obtained as a white solid (33.4 mg, 0.09 mmol, Yield: 68%); m.p. = 134-136 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 (d, *J* = 6.8 Hz, 1H), 7.18 – 7.01 (m, 5H), 6.98 (s, 1H), 6.93 – 6.87 (m, 4H), 4.54 (t, *J* = 8.1 Hz, 2H), 3.48 (t, *J* = 8.1 Hz, 2H), 2.26 (s, 3H), 2.21 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.48, 147.08, 141.58, 137.41, 136.68, 135.88, 135.37, 133.40, 131.55, 130.39, 130.32, 128.19, 127.92, 127.77, 127.66, 127.25, 126.81, 124.64, 123.76, 122.92, 118.67, 47.31, 27.18, 21.33 × 2; HRMS (ESI): m/z calculated for $[C_{25}H_{22}NO]^+$ [M + H]$^+$: 352.1701, Found: 352.1703; FTIR (NaCl): ν 3049, 2920, 2856, 1643, 1616, 1602, 1489, 1404, 1340, 1265, 1190 cm^{-1}

5,6-Bis(3-methoxyphenyl)-1H-pyrrolo[3,2,1-*ij*]quinolin-4(2H)-one (3d)



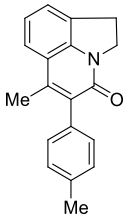
Following the general reaction procedure A, **3d** was obtained as a yellow oil (35.4 mg, 0.09 mmol, Yield: 66%); NMR (400 MHz, $CDCl_3$) δ 7.36 (d, *J* = 7.1 Hz, 1H), 7.21 (t, *J* = 7.9 Hz, 1H), 7.17 (d, *J* = 8.8 Hz, 1H), 7.09 (d, *J* = 7.1 Hz, 1H), 7.07 (d, *J* = 7.1 Hz, 1H), 6.81 – 6.76 (m, 3H), 6.73 – 6.61 (m, 3H), 4.55 (t, *J* = 7.9 Hz, 2H), 3.65 (s, 3H), 3.63 (s, 3H), 3.49 (t, *J* = 7.9 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.21, 159.19, 158.80, 147.12, 141.52, 137.10, 136.64, 132.85, 130.54, 129.04, 128.46, 124.93, 123.73, 123.46, 123.23, 122.14, 118.46, 116.05, 115.15, 113.52, 113.35, 55.17, 55.09, 47.48, 27.15; HRMS (ESI): m/z calculated for $[C_{25}H_{22}NO_3]^+$ [M + H]$^+$: 384.1600, Found: 384.1609; FTIR (NaCl): ν 3061, 2956, 1645, 1614, 1600, 1456, 1317, 1045 cm^{-1}

6-Methyl-5-phenyl-1H-pyrrolo[3,2,1-*ij*]quinolin-4(2H)-one (3e)



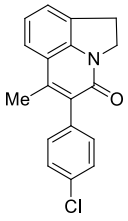
Following the general reaction procedure B, **3e** was obtained as a white solid (27.4 mg, 0.11 mmol, Yield: 75%); m.p. = 152-154 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (d, *J* = 8.1 Hz, 1H), 7.43 (t, *J* = 7.6 Hz, 2H), 7.37 – 7.34 (m, 2H), 7.29 – 7.27 (m, 2H), 7.18 (t, *J* = 7.6 Hz, 1H), 4.52 – 4.38 (t, *J* = 8.1 Hz, 2H), 3.43 (t, *J* = 8.1 Hz, 2H), 2.30 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.26, 142.18, 141.22, 136.28, 133.68, 130.57, 130.21 × 2, 128.11 × 2, 127.40, 124.63, 122.91, 121.56, 118.81, 47.00, 27.14, 16.10; HRMS (ESI): m/z calculated for $[C_{18}H_{16}NO]^+$ [M + H]$^+$: 262.1232, Found: 262.1236; FTIR (NaCl): ν 3053, 2958, 1641, 1598, 1487, 1408, 1346, 1265, 1029 cm^{-1}

6-Methyl-5-(p-tolyl)-1H-pyrrolo[3,2,1-*ij*]quinolin-4(2H)-one (3f)



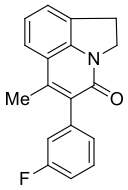
Following the general reaction procedure, **3f** was obtained as a yellow solid (18.1 mg, 0.07 mmol, Yield: 47%); ^{1}H m.p. = 201-203 ℃; NMR (400 MHz, $CDCl_3$) δ 7.49 (d, *J* = 8.4 Hz, 1H), 7.34 (d, *J* = 7.2 Hz, 1H), 7.24 (d, *J* = 8.4 Hz, 2H), 7.21 – 7.14 (m, 3H), 4.51 – 4.39 (t, *J* = 8.1 Hz, 2H), 3.42 (t, *J* = 8.1 Hz, 2H), 2.40 (s, 3H), 2.31 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.40, 141.98, 141.18, 137.02, 133.69, 133.22, 130.52, 130.07 × 2, 128.85 × 2, 124.51, 122.84, 121.53, 118.86, 46.99, 27.15, 21.30, 16.12; HRMS (ESI): m/z calculated for $[C_{19}H_{18}NO]^+$ [M + H]$^+$: 276.1388, Found: 276.1391; FTIR (NaCl): ν 3055, 2980, 1635, 1602, 1508, 1340, 1265, 1112 cm^{-1}

5-(4-Chlorophenyl)-6-methyl-1H-pyrrolo[3,2,1-*ij*]quinolin-4(2H)-one (3g)



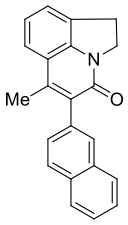
Following the general reaction procedure B, **3g** was obtained as a white solid (25.2 mg, 0.08 mmol, Yield: 61%); m.p. = 214-216 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (d, *J* = 8.1 Hz, 1H), 7.44 – 7.39 (m, 2H), 7.36 (d, *J* = 7.2 Hz, 1H), 7.24 – 7.17 (m, 3H), 4.45 (t, *J* = 8.1 Hz, 2H), 3.44 (t, *J* = 8.1 Hz, 2H), 2.31 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.01, 142.52, 141.25, 134.68, 133.40, 132.43, 131.72 × 2, 130.63, 128.38 × 2, 124.89, 123.06, 121.60, 118.66, 47.04, 27.14, 16.12; HRMS (ESI): m/z calculated for $[C_{18}H_{15}ClNO]^+$ [M + H]$^+$: 296.0842, Found: 296.0843; FTIR (NaCl): ν 2926, 2852, 1635, 1616, 1458, 1344, 1263, 817cm^{-1}

5-(3-Fluorophenyl)-6-methyl-1H-pyrrolo[3,2,1-*ij*]quinolin-4(2H)-one (3h)



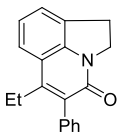
Following the general reaction procedure B, **3h** was obtained as a white solid (19.5 mg, 0.07 mmol, Yield: 50%); m.p. = 145-147 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 (d, *J* = 8.1 Hz, 1H), 7.44 – 7.34 (m, 2H), 7.23 – 7.16 (m, 1H), 7.09 – 6.98 (m, 3H), 4.45 (t, *J* = 8.1 Hz, 2H), 3.44 (t, *J* = 8.1 Hz, 2H), 2.31 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.70 (d, *J* = 246.6.4 Hz), 159.94, 142.69, 141.32, 138.49 (d, *J* = 8.1 Hz), 132.49 (d, *J* = 1.6 Hz), 130.68, 129.66 (d, *J* = 8.4 Hz), 126.06 (d, *J* = 2.9 Hz), 124.97, 123.10, 121.66, 118.63, 117.37 (d, *J* = 21.5 Hz), 114.44 (d, *J* = 21.0 Hz), 47.07, 27.17, 16.09; ^{19}F NMR (376 MHz, $CDCl_3$) δ -113.75; HRMS (ESI): m/z calculated for $[C_{18}H_{15}FNO]^+$ [M + H]$^+$: 280.1138, Found: 280.1135; FTIR (NaCl): ν 2956, 2924, 1637, 1616, 1604, 1583, 1458, 1406, 1186, 719 cm^{-1}

6-Methyl-5-(naphthalen-2-yl)-1H-pyrrolo[3,2,1-*ij*]quinolin-4(2H)-one (3i)



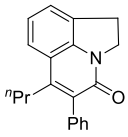
Following the general reaction procedure B, **3i** was obtained as a white solid (34.4 mg, 0.11 mmol, Yield: 79%); m.p. = 140-142 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.4 Hz, 1H), 7.89 – 7.81 (m, 2H), 7.75 (d, *J* = 8.4 Hz, 1H), 7.52 (d, *J* = 8.4 Hz, 1H), 7.50 – 7.46 (m, 2H), 7.43 (dd, *J* = 8.4, 1.6 Hz, 1H), 7.36 (d, *J* = 7.2 Hz, 1H), 7.22 – 7.16 (m, 1H), 4.47 (t, *J* = 8.1 Hz, 2H), 3.44 (t, *J* = 8.1 Hz, 2H), 2.34 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.35, 142.54, 141.28, 133.91, 133.56, 133.31, 132.76, 130.60, 129.28, 128.31, 128.06, 127.65, 127.62, 125.91, 125.81, 124.71, 122.97, 121.60, 118.85, 47.03, 27.16, 16.19; HRMS (ESI): m/z calculated for $[C_{22}H_{18}NO]^+$ $[M + H]^+$: 312.1388, Found: 312.1387; FTIR (NaCl): ν 3035, 2926, 2854, 1639, 1606, 1406, 1256 cm^{-1}

6-Ethyl-5-phenyl-1H-pyrrolo[3,2,1-*ij*]quinolin-4(2H)-one (3j)



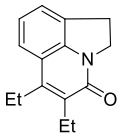
Following the general reaction procedure B, **3j** was obtained as a brown solid (23.9 mg, 0.09 mmol, Yield: 62%); m.p. = 129-131 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 – 7.50 (m, 1H), 7.45 – 7.42 (m, 2H), 7.39 – 7.32 (m, 2H), 7.27 – 7.25 (m, 2H), 7.22 – 7.13 (m, 1H), 4.45 (t, *J* = 8.1 Hz, 2H), 3.43 (t, *J* = 8.1 Hz, 2H), 2.69 (q, *J* = 7.6 Hz, 2H), 1.16 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.51, 148.12, 141.75, 136.36, 133.30, 130.90, 129.76 × 2, 128.23 × 2, 127.37, 124.52, 122.90, 121.61, 117.54, 46.98, 27.12, 22.77, 14.46; HRMS (ESI): m/z calculated for $[C_{19}H_{18}NO]^+$ $[M + H]^+$: 276.1388, Found: 276.1385; FTIR (NaCl): ν 2972, 2931, 1637, 1608, 1456, 1456, 1256 cm^{-1}

5-Phenyl-6-propyl-1H-pyrrolo[3,2,1-*ij*]quinolin-4(2H)-one (3k)



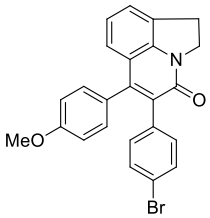
Following the general reaction procedure B, **3k** was obtained as a white solid (23.5 mg, 0.08 mmol, Yield: 58%); m.p. = 185-187 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (d, *J* = 8.1 Hz, 1H), 7.45 – 7.41 (m, 2H), 7.38 – 7.33 (m, 2H), 7.26 – 7.24 (m, 2H), 7.17 (t, *J* = 7.6 Hz, 1H), 4.45 (t, *J* = 8.1 Hz, 2H), 3.42 (t, *J* = 8.1 Hz, 2H), 2.69 – 2.61 (m, 2H), 1.65 – 1.49 (m, 2H), 0.86 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.49, 146.74, 141.65, 136.39, 133.61, 130.85, 129.90 × 2, 128.16 × 2, 127.34, 124.50, 122.84, 121.72, 117.90, 46.97, 31.61, 27.11, 23.41, 14.36; HRMS (ESI): m/z calculated for $[C_{20}H_{20}NO]^+$ $[M + H]^+$: 290.1545, Found: 290.1542; FTIR (NaCl): ν 2962, 2929, 1633, 1614, 1606, 1462, 1404, 1346, 1292 cm^{-1}

5,6-Diethyl-1H-pyrrolo[3,2,1-*ij*]quinolin-4(2H)-one (3l)



Following the general reaction procedure B, **3l** was obtained as a white solid (14.0 mg, 0.06 mmol, Yield: 44%); m.p. = 116-118 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (d, *J* = 8.1 Hz, 1H), 7.30 – 7.24 (m, 1H), 7.13 (t, *J* = 7.4 Hz, 1H), 4.46 – 4.39 (t, *J* = 8.1 Hz, 2H), 3.39 (t, *J* = 8.1 Hz, 2H), 2.89 (q, *J* = 7.6 Hz, 2H), 2.76 (q, *J* = 7.6 Hz, 2H), 1.26 (t, J = 7.6 Hz, 3H), 1.18 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.89, 145.91, 140.91, 133.81, 130.65, 123.64, 122.73, 120.83, 117.87, 46.79, 27.10, 21.56, 20.32, 14.28, 13.92; HRMS (ESI): m/z calculated for $[C_{15}H_{18}NO]^+$ $[M + H]^+$: 228.1388, Found: 228.1391; FTIR (NaCl): ν 2986, 2933, 1637, 1606, 1487, 1265, 1051 cm^{-1}

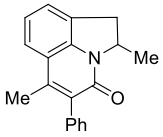
6-(4-Bromophenyl)-5-(4-methoxyphenyl)-1H-pyrrolo[3,2,1-*ij*]quinolin-4(2H)-one (3m)



Following the general reaction procedure A, **3m** was obtained as a yellow solid (19.3 mg, 0.04 mmol, Yield: 32%); m.p. = 219-221 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (d, *J* = 7.3 Hz, 1H), 7.31 (d, *J* = 8.6 Hz, 2H), 7.16 (d, *J* = 7.3 Hz,

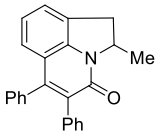
1H), 7.09 (t, *J* = 7.3 Hz, 1H), 7.03 (d, *J* = 8.5 Hz, 2H), 7.01 (d, *J* = 8.6 Hz, 2H), 6.83 (d, *J* = 8.5 Hz, 2H), 4.53 (t, *J* = 7.8 Hz, 2H), 3.81 (s, 3H), 3.48 (t, *J* = 7.8 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.10, 159.09, 147.11, 141.74, 134.73, 132.81 × 2, 132.00, 131.05 × 2, 130.72 × 2, 130.54, 127.80, 125.01, 123.84, 123.14, 121.10, 118.72, 113.71 × 2, 55.21, 47.38, 27.19; HRMS (ESI): m/z calculated for $[C_{24}H_{19}BrNO_2]^+$ $[M + H]^+$: 432.0599, Found: 432.0586; FTIR (NaCl): ν 2958, 2929, 1639, 1604, 1512, 1406, 1290, 1265, 1247, 1176, 1033, 1010 804 cm^{-1}

2,6-Dimethyl-5-phenyl-1H-pyrrolo[3,2,1-*ij*]quinolin-4(2H)-one (4a)



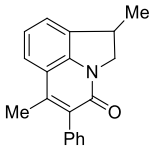
Following the general reaction procedure B, **4a** was obtained as a white solid (20.8 mg, 0.08 mmol, Yield: 54%); m.p. = 137-139 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 (dd, *J* = 8.0, 0.6 Hz, 1H), 7.45 – 7.42 (m, 2H), 7.39 – 7.27 (m, 4H), 7.21 – 7.16 (m, 1H), 5.11 – 5.01 (m, 1H), 3.64 (dd, *J* = 16.7, 9.4 Hz, 1H), 3.00 (dd, *J* = 16.7, 3.8 Hz, 1H), 2.31 (s, 3H), 1.62 (d, J = 6.4 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.19, 142.09, 140.58, 136.34, 134.19, 130.27 × 2, 129.16, 128.10 × 2, 127.35, 124.73, 122.91, 121.59, 118.72, 56.87, 36.32, 20.60, 16.11; HRMS (ESI): m/z calculated for $[C_{19}H_{18}NO]^+$ $[M + H]^+$: 276.1388, Found: 276.1397; FTIR (NaCl): ν 3053, 2960, 2926, 1637, 1616, 1608, 1458, 1404, 1265, 1010 cm^{-1}

2-Methyl-5,6-diphenyl-1H-pyrrolo[3,2,1-*ij*]quinolin-4(2H)-one (4b)



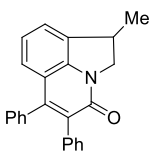
Following the general reaction procedure A, **4b** was obtained as a white solid (29.7 mg, 0.09 mmol, Yield: 63%); m.p. = 235-237 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 – 7.29 (m, 2H), 7.27 – 7.20 (m, 3H), 7.19 – 6.99 (m, 8H), 5.26 – 5.08 (m, 1H), 3.69 (dd, *J* = 16.7, 9.4 Hz, 1H), 3.05 (dd, *J* = 16.7, 3.8 Hz, 1H), 1.69 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.26, 146.89, 141.08, 136.08, 135.57, 133.87, 131.04 × 2, 129.84, 129.82, 129.04, 128.09, 127.89, 127.54, 127.43 × 2, 126.87, 124.89, 123.78, 122.98, 118.47, 57.22, 36.36, 20.62; HRMS (ESI): m/z calculated for $[C_{24}H_{20}NO]^+$ $[M + H]^+$: 338.1545, Found: 338.1546; FTIR (NaCl): ν 2924, 1639, 1618, 1438, 1298, 1265 cm^{-1}

1,6-Dimethyl-5-phenyl-1H-pyrrolo[3,2,1-*ij*]quinolin-4(2H)-one (4c)



Following the general reaction procedure B, **4c** was obtained as a white solid (25.0 mg, 0.09 mmol, Yield: 65%); m.p. = 134-136 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (d, *J* = 8.0 Hz, 1H), 7.45 – 7.42 (m, 2H), 7.37 – 7.33 (m, 2H), 7.31 – 7.18 (m, 3H), 4.62 (dd, *J* = 12.7, 9.4 Hz, 1H), 4.00 (dd, *J* = 12.7, 5.5 Hz, 1H), 3.79 – 3.75 (m, 1H), 2.31 (s, 3H), 1.48 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.23, 142.26, 140.59, 136.31, 135.77, 133.82, 130.19 × 2, 128.13 × 2, 127.41, 123.69, 123.03, 121.77, 118.76, 55.02, 34.73, 20.82, 16.14; HRMS (ESI): m/z calculated for $[C_{19}H_{18}NO]^+$ $[M + H]^+$: 276.1388, Found: 276.1391; FTIR (NaCl): ν 3053, 2964, 2926, 1639, 1616, 1608, 1456, 1265 cm^{-1}

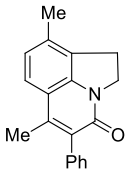
1-Methyl-5,6-diphenyl-1H-pyrrolo[3,2,1-*ij*]quinolin-4(2H)-one (4d)



Following the general reaction procedure A, **4d** was obtained as a white solid (25.5 mg, 0.08 mmol, Yield: 54%); m.p. = 181-183 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 (d, *J* = 6.6 Hz, 1H), 7.29 – 7.24 (m, 3H), 7.19 – 7.05 (m, 9H), 4.71 (dd, *J* = 12.8, 9.4 Hz, 1H), 4.09 (dd, *J* = 12.8, 5.5 Hz, 1H), 3.87 – 3.82 (m, 1H), 1.52 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.27, 147.02, 141.06, 136.00, 135.61, 135.53, 133.49, 130.94 × 2, 129.77 × 2, 127.97 × 2, 127.54, 127.42 × 2, 126.89, 123.88, 123.83, 123.08, 118.47, 55.31, 34.78, 20.83;

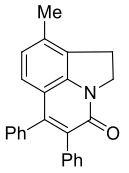
HRMS (ESI): m/z calculated for $[C_{24}H_{20}NO]^+$ [M + H]$^+$: 338.1545, Found: 338.1540; FTIR (NaCl): ν 3055, 2964, 2926, 1641, 1616, 1602, 1456, 1317, 1265, 1024 cm^{-1}

6,9-Dimethyl-5-phenyl-1H-pyrrolo[3,2,1-*ij*]quinolin-4(2H)-one (4e)



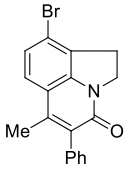
Following the general reaction procedure B, **4e** was obtained as a white solid (28.9 mg, 0.11 mmol, Yield: 75%); m.p. = 168-170 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 – 7.38 (m, 3H), 7.37 – 7.34 (m, 1H), 7.31 – 7.25 (m, 2H), 7.01 (d, *J* = 8.2 Hz, 1H), 4.45 (d, *J* = 8.1 Hz, 2H), 3.32 (d, *J* = 8.1 Hz, 2H), 2.38 (s, 3H), 2.28 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.45, 142.23, 141.02, 136.38, 134.95, 132.44, 130.28 × 2, 128.68, 128.05 × 2, 127.26, 124.60, 121.68, 116.76, 47.06, 26.18, 18.50, 16.04; HRMS (ESI): m/z calculated for $[C_{19}H_{18}NO]^+$ [M + H]$^+$: 276.1388, Found: 276.1382; FTIR (NaCl): ν 3055, 2924, 1645, 1616, 1456, 1340, 1265 cm^{-1}

9-Methyl-5,6-diphenyl-1H-pyrrolo[3,2,1-*ij*]quinolin-4(2H)-one (4f)



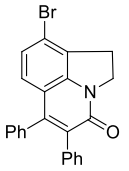
Following the general reaction procedure A, **4f** was obtained as a white solid (31.1 mg, 0.09 mmol, Yield: 66%); m.p. = 205-207 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 – 7.23 (m, 3H), 7.17 – 7.08 (m, 7H), 7.02 (d, *J* = 8.2 Hz, 1H), 6.89 (d, *J* = 8.2 Hz, 1H), 4.54 (t, *J* = 8.1 Hz, 2H), 3.37 (t, *J* = 8.1 Hz, 2H), 2.37 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.48, 146.95, 141.55, 136.14, 135.61, 135.19, 132.14, 131.02 × 2, 129.75 × 2, 128.55, 127.91 × 2, 127.45, 127.36 × 2, 126.75, 124.68, 123.80, 116.47, 47.36, 26.25, 18.53; HRMS (ESI): m/z calculated for $[C_{24}H_{20}NO]^+$ [M + H]$^+$: 338.1545, Found: 338.1543; FTIR (NaCl): ν 3051, 2983, 1635, 1610, 1419, 1338, 1265 cm^{-1}

9-Bromo-6-methyl-5-phenyl-1H-pyrrolo[3,2,1-*ij*]quinolin-4(2H)-one (4g)



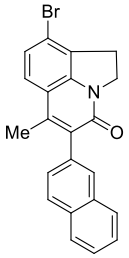
Following the general reaction procedure B, **4g** was obtained as a white solid (23.3 mg, 0.07 mmol, Yield: 49%); m.p. = 212-214 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 – 7.41 (m, 2H), 7.39 – 7.38 (m, 2H), 7.29 (d, *J* = 6.7 Hz, 1H), 7.27 (d, *J* = 6.7 Hz, 2H), 4.47 (d, *J* = 8.0 Hz, 2H), 3.40 (d, *J* = 8.0 Hz, 2H), 2.29 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.33, 141.98, 141.77, 135.90, 133.76, 131.05, 130.11× 2, 128.19 × 2, 127.59, 125.86, 123.50, 119.28, 117.65, 46.63, 28.59, 16.17; HRMS (ESI): m/z calculated for $[C_{18}H_{15}BrNO]^+$ [M + H]$^+$: 340.0337, Found: 340.0335; FTIR (NaCl): ν 3053, 2982, 1639, 1620, 1595, 1394, 1338, 1265, 1010 cm^{-1}

9-Bromo-5,6-diphenyl-1H-pyrrolo[3,2,1-*ij*]quinolin-4(2H)-one (4h)



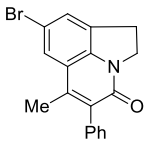
Following the general reaction procedure A, **4h** was obtained as a white solid (32.6 mg, 0.08 mmol, Yield: 58%); m.p. = 173-175 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 – 7.28 (m, 3H), 7.19 – 7.17 (m, 3H), 7.16 – 7.08 (m, 5H), 7.01 (d, *J* = 8.6 Hz, 1H), 4.59 (t, *J* = 8.0 Hz, 2H), 3.46 (t, *J* = 8.0 Hz, 2H); ^{13}C NMR (101 MHz, CDCl3) δ 160.35, 146.68, 142.16, 135.58, 135.13, 133.37, 130.91, 130.84 × 2, 129.67 × 2, 128.10 × 2, 127.76, 127.47 × 2, 127.06, 125.92, 125.58, 119.53, 117.35, 46.91, 28.62.; HRMS (ESI): m/z calculated for $[C_{23}H_{17}BrNO]^+$ [M + H]$^+$: 402.0494, Found: 402.0488; FTIR (NaCl): ν 3053, 2981, 1643, 1616, 1593, 1338, 1265, 1114, 1008 cm^{-1}

9-Bromo-6-methyl-5-(naphthalen-2-yl)-1H-pyrrolo[3,2,1-*ij*]quinolin-4(2H)-one (4i)



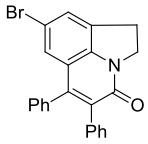
Following the general reaction procedure B, **4i** was obtained as a white solid (20.5 mg, 0.05 mmol, Yield: 48%); m.p. = 204-206 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.4 Hz, 1H), 7.89 – 7.80 (m, 2H), 7.74 (s, 1H), 7.54 – 7.44 (m, 2H), 7.41 – 7.38 (m, 2H), 7.30 (d, *J* = 8.4 Hz, 1H), 4.48 (t, *J* = 8.0 Hz, 2H), 3.39 (t, *J* = 8.0 Hz, 2H), 2.32 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.41, 142.34, 141.80, 133.62, 133.50, 133.27, 132.81, 131.08, 129.25, 128.10, 128.07, 127.72, 127.67, 126.04, 125.92 × 2, 123.52, 119.36, 117.68, 46.65, 28.60, 16.26; HRMS (ESI): m/z calculated for $[C_{22}H_{17}BrNO]^+$ $[M + H]^+$: 390.0494, Found: 390.0490; FTIR (NaCl): ν 3053, 2985, 1635, 1614, 1595, 1421, 1396, 1336, 1265, 896 cm^{-1}

8-Bromo-6-methyl-5-phenyl-1H-pyrrolo[3,2,1-*ij*]quinolin-4(2H)-one (4j)



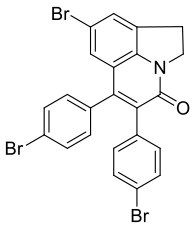
Following the general reaction procedure B, **4j** was obtained as a white solid (32.3 mg, 0.10 mmol, Yield: 68%); m.p. = 174-176 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (s, 1H), 7.46 – 7.42 (m, 3H), 7.38 – 3.37 (m, 1H), 7.27 – 7.25 (m, 2H), 4.45(t, *J* = 8.0 Hz, 2H), 3.42 (t, *J* = 8.0 Hz, 2H), 2.27 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.97, 141.23, 140.14, 135.77, 134.63, 132.62, 130.07 × 2, 128.19 × 2, 127.81, 127.65, 124.31, 119.88, 115.56, 47.20, 26.96, 16.11; HRMS (ESI): m/z calculated for $[C_{18}H_{15}BrNO]^+$ $[M + H]^+$: 340.0337, Found: 340.0340; FTIR (NaCl): ν 3055, 2924, 1643, 1633, 1614, 1483, 1435, 1357, 1263, 856 cm^{-1}

8-Bromo-5,6-diphenyl-1H-pyrrolo[3,2,1-*ij*]quinolin-4(2H)-one (4k)



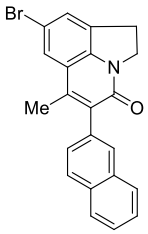
Following the general reaction procedure A, **4k** was obtained as a white solid (42.7 mg, 0.11 mmol, Yield: 76%); m.p. = 220-222 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 – 7.20 (m, 5H), 7.14 – 7.07(m, 7H), 4.54 (d, *J* = 7.9 Hz, 2H), 3.47 (t, *J* = 7.9 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.98, 145.94, 140.62, 135.26, 135.04, 134.34, 132.51, 130.80 × 2, 129.63 × 2, 128.19 × 2, 128.00, 127.85, 127.46 × 2, 127.11, 126.01, 119.56, 115.65, 47.48, 26.96; HRMS (ESI): m/z calculated for $[C_{23}H_{17}BrNO]^+$ $[M + H]^+$: 402.0494, Found: 402.0505; FTIR (NaCl): ν 3053, 2926, 1643, 1618, 1458, 1336, 1265, 1072, 867 cm^{-1}

8-Bromo-5,6-bis(4-bromophenyl)-1H-pyrrolo[3,2,1-*ij*]quinolin-4(2H)-one (4l)



Following the general reaction procedure A, **4l** was obtained as a white solid (47.6 mg, 0.09 mmol, Yield: 61%); m.p. = 280-282 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 – 7.45 (m, 3H), 7.33 (d, *J* = 8.3 Hz, 2H), 7.17 (s, 1H), 6.97 (d, *J* = 8.2 Hz, 2H), 6.97 (d, *J* = 8.2 Hz, 2H), 4.52 (t, *J* = 8.0 Hz, 2H), 3.47 (t, *J* = 8.0 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.42, 144.86, 140.68, 133.88, 133.66, 133.25, 132.69, 132.46 × 2, 131.78 × 2, 131.18 × 2, 130.94 × 2, 128.48, 125.72, 122.48, 121.73, 119.00, 115.97, 47.56, 26.95; HRMS (ESI): m/z calculated for $[C_{23}H_{15}Br_3NO]^+$ $[M + H]^+$: 557.8704, Found: 557.8721; FTIR (NaCl): ν 3053, 2985, 1645, 1616, 1489, 1419, 1265, 1070, 894 cm^{-1}

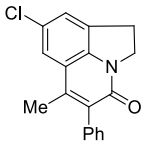
8-Bromo-6-methyl-5-(naphthalen-2-yl)-1H-pyrrolo[3,2,1-*ij*]quinolin-4(2H)-one (4m)



Following the general reaction procedure B, **4m** was obtained as a white solid (32.1 mg, 0.08 mmol, Yield: 59%); m.p. = 189-191 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.4 Hz, 1H), 7.89 – 7.80 (m, 2H), 7.74 (s, 1H), 7.67 (s, 1H), 7.52 – 7.45 (m, 3H), 7.42 – 7.38 (m, 1H), 4.48 (t, *J* = 8.0 Hz, 2H), 3.44 (t, *J* = 8.0 Hz, 2H), 2.31 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.09, 141.57, 140.25, 134.61, 133.41, 133.28, 132.87,

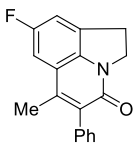
132.67, 129.27, 128.11, 128.06, 127.92, 127.77, 127.70, 126.12, 125.97, 124.38, 119.97, 115.65, 47.25, 27.02, 16.23; HRMS (ESI): m/z calculated for $[C_{22}H_{17}BrNO]^+$ $[M + H]^+$: 390.0494, Found: 390.0479; FTIR (NaCl): ν 3053, 2980, 2856, 1645, 1635, 1487, 1265, 1116, 819 cm^{-1}

8-Chloro-6-methyl-5-phenyl-1H-pyrrolo[3,2,1-*ij*]quinolin-4(2H)-one (4n)



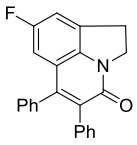
Following the general reaction procedure B, **4n** was obtained as a white solid (22.7 mg, 0.08 mmol, Yield: 55%); m.p. = 257-259 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 (s, 1H), 7.44 – 7.42 (m, 2H), 7.38 – 7.35 (m, 1H), 7.31 (s, 1H), 7.28 – 7.26 (m, 2H), 4.47 (t, *J* = 7.6 Hz, 2H), 3.42 (t, *J* = 7.6 Hz, 2H), 2.27 (s, 3H);^{13}C NMR (101 MHz, $CDCl_3$) δ 160.00, 141.27, 139.82, 135.82, 134.69, 132.31, 130.09 × 2, 128.28, 128.19 × 2, 127.65, 125.20, 121.30, 119.30, 47.26, 27.03, 16.12; HRMS (ESI): m/z calculated for $[C_{18}H_{15}ClNO]^+$ $[M + H]^+$: 296.0842, Found: 296.0839; FTIR (NaCl): ν 3053, 2924, 1639, 1620, 1959, 1483, 1338, 1265, 1010, 869 cm^{-1}

8-Fluoro-6-methyl-5-phenyl-1H-pyrrolo[3,2,1-*ij*]quinolin-4(2H)-one (4o)



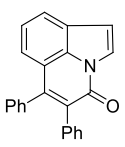
Following the general reaction procedure B, **4o** was obtained as a white solid (16.8 mg, 0.06 mmol, Yield: 43%); m.p. = 166-168 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 – 7.42 (m, 2H), 7.37 (d, *J* = 7.2 Hz, 1H), 7.28 (d, *J* = 1.5 Hz, 1H), 7.26 (d, *J* = 1.5 Hz, 1H), 7.17 (d, *J* = 10.1 Hz, 1H), 7.12 (d, *J* = 8.3 Hz, 1H), 4.48 (t, *J* = 8.0 Hz, 2H), 3.43 (t, *J* = 8.0 Hz, 2H), 2.26 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.87, 159.42 (d, *J* = 241.4 Hz), 141.48, 137.64, 135.96, 134.72, 132.35 (d, *J* = 9.1 Hz), 130.07 × 2, 128.16 × 2, 127.59, 118.73 (d, *J* = 9.8 Hz), 113.65 (d, *J* = 26.8 Hz), 107.0 (d, *J* = 24.9 Hz), 47.31, 27.15 (d, *J* = 1.8 Hz), 16.21; ^{19}F NMR (376 MHz, $CDCl_3$) δ -119.06; HRMS (ESI): m/z calculated for $[C_{18}H_{15}FNO]^+$ $[M + H]^+$: 280.1138, Found: 280.1142; FTIR (NaCl): ν 2956, 2922, 2852, 1645, 1616, 1487, 1396, 1267, 1168, 943 cm^{-1}

8-Fluoro-5,6-diphenyl-1H-pyrrolo[3,2,1-*ij*]quinolin-4(2H)-one (4p)



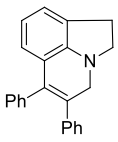
Following the general reaction procedure A, **4p** was obtained as a white solid (29.1 mg, 0.09 mmol, Yield: 61%); m.p. = 202-204 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.23 (m, 3H), 7.21 – 7.03 (m, 8H), 6.79 (dd, *J* = 10.2, 2.0 Hz, 1H), 4.63 – 4.50 (t, *J* = 8.0 Hz, 2H)., 3.48 (t, *J* = 8.0 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.94, 159.37 (d, *J* = 241.4 Hz), 146.27 (d, *J* = 3.4 Hz), 138.17, 135.60, 135.25, 134.42, 132.33(d, *J* = 9.3 Hz), 130.88 × 2, 129.64 × 2, 128.20 × 2, 127.83, 127.50 × 2, 127.12, 118.45 (d, *J* = 9.5 Hz), 113.94 (d, *J* = 27.1 Hz), 108.90 (d, *J* = 25.7 Hz), 47.64, 27.22 (d, *J* = 1.4 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -118.85; HRMS (ESI): m/z calculated for $[C_{23}H_{17}FNO]^+$ $[M + H]^+$: 342.1294, Found: 342.1292; FTIR (NaCl): ν 3053, 2980, 2924, 1645, 1622, 1396, 1265, 1136, 864 cm^{-1}

5,6-Diphenyl-4H-pyrrolo[3,2,1-*ij*]quinolin-4-one (5)



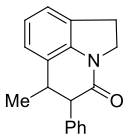
Following the general reaction procedure C, **5** was obtained as a yellow oil (37.4 mg, 0.11 mmol, Yield: 97%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (d, *J* = 3.6 Hz, 1H), 7.85 (d, *J* = 7.3 Hz, 1H), 7.42 – 7.27 (m, 5H), 7.23 – 7.17 (m, 7H), 6.96 (d, *J* = 3.6 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.04, 148.59, 135.38, 135.02, 133.44, 131.66, 131.00 × 2, 130.07 × 2, 127.88 × 2, 127.85, 127.74, 127.62 × 2, 127.27, 124.91, 124.71, 124.01, 123.97, 118.46, 110.84; HRMS (ESI): m/z calculated for $[C_{23}H_{16}NO]^+$ $[M + H]^+$: 322.1232, Found: 322.1236; FTIR (NaCl): ν 3053, 2987, 1666, 1633, 1444, 1384, 1300, 1265 cm^{-1}

5,6-Diphenyl-2,4-dihydro-1H-pyrrolo[3,2,1-*ij*]quinolone (6)



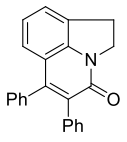
Following the general reaction procedure D, **6** was obtained as a yellow oil (16.7 mg, 0.05 mmol, Yield: 45%); ^{1}H NMR (400 MHz, *d*-Acetone) δ 7.29 – 7.18 (m, 4H), 7.17 – 7.06 (m, 6H), 6.95 (dd, *J* = 7.5, 1.0 Hz, 1H), 6.48 (t, *J* = 7.5 Hz, 1H), 6.40 (d, *J* = 7.5 Hz, 1H), 4.19 (s, 2H), 3.33 (t, *J* = 8.1 Hz, 2H), 3.00 (t, *J* = 8.1 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 149.16, 140.56, 137.47, 134.02, 130.59 × 2, 128.27 × 2, 128.00, 127.97 × 2, 127.75 × 2, 126.96, 126.76, 126.53, 124.05, 123.11, 121.34, 118.79, 55.45, 55.20, 28.87; HRMS (ESI): m/z calculated for $[C_{23}H_{20}N]^+$ $[M + H]^+$: 310.1596, Found: 310.1602; FTIR (NaCl): ν 3054, 2925, 1647, 1446, 1261 cm^{-1}

6-Methyl-5-phenyl-5,6-dihydro-1H-pyrrolo[3,2,1-*ij*]quinolin-4(2H)-one (7)



Following the general reaction procedure E, **7** was obtained as a white solid (16.4 mg, 0.06 mmol, Yield: 52%); m.p. = 158-160 ℃; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.24 – 7.18 (m, 3H), 7.16 – 7.14 (m, 1H), 7.05 – 6.99 (m, 4H), 4.26 – 4.04 (m, 2H), 3.86 (d, *J* = 6.3 Hz, 1H), 3.58 – 3.40 (m, 1H), 3.37 – 3.14 (m, 2H), 1.12 (d, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.44, 140.77, 136.56, 129.06 × 2, 128.47, 128.37 × 2, 127.24, 125.09, 123.83, 123.78, 123.49, 54.26, 45.36, 35.58, 28.00, 14.50; HRMS (ESI): m/z calculated for $[C_{18}H_{18}NO]^+$ $[M + H]^+$: 264.1388, Found: 264.1383; FTIR (NaCl): ν 3030, 2918, 2848, 1668, 1593, 1471, 1394, 1338 cm^{-1}

NMR spectra of products



3a

1198-10-2 CDCl3 BBFO1 400 Apr

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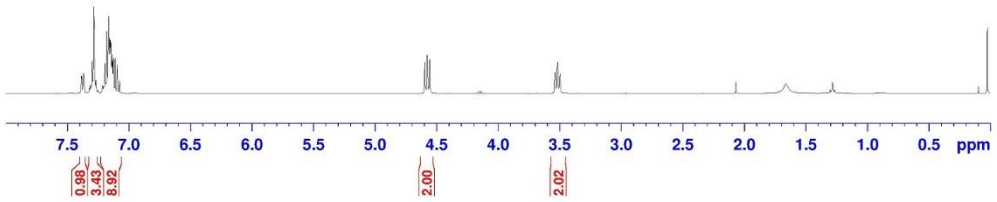
7.212
7.209
7.203
7.196
7.191
7.178
7.162
7.154
7.149
7.144
7.135
7.131
7.125
7.110
7.093
7.073

4.595
4.575
4.554

3.537
3.516
3.496



annula, 1H, CDCl3, 400MHz, AV400

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141.64
135.92
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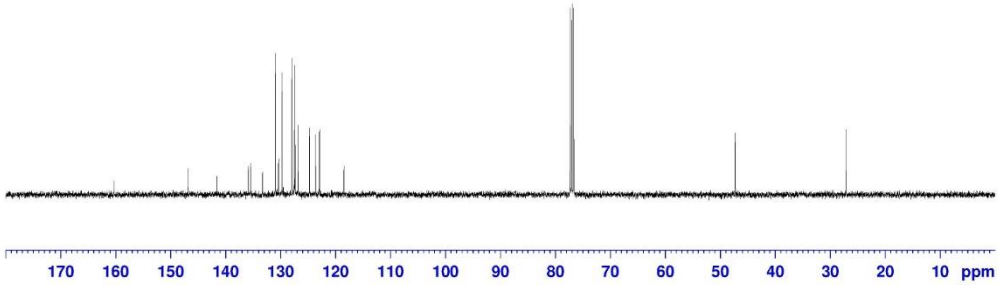
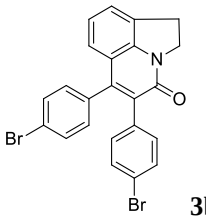
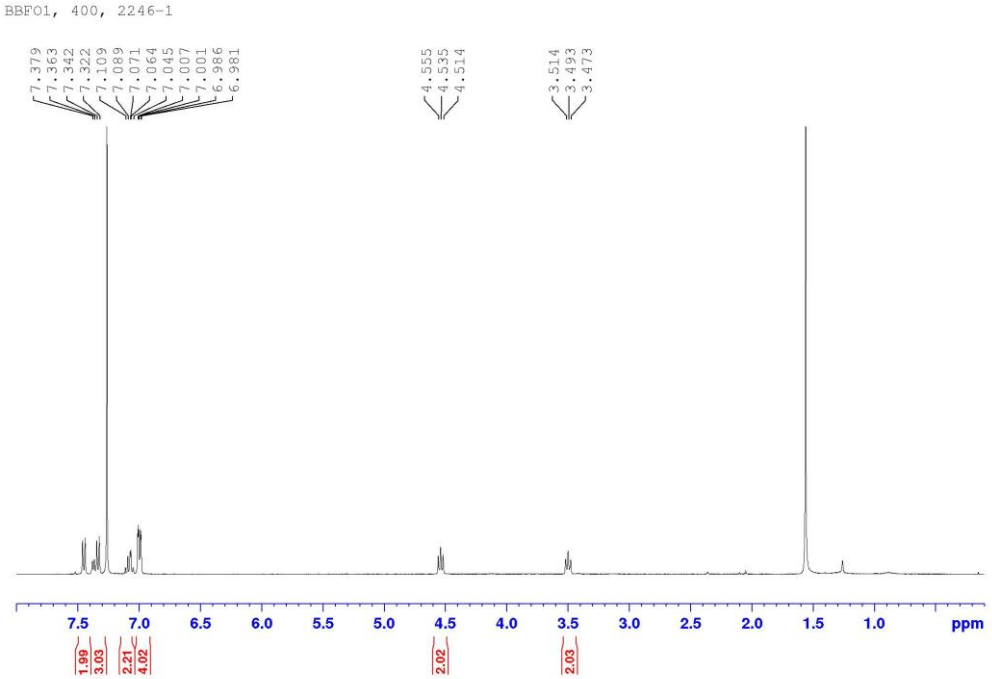
133.00
130.90
130.38
129.72
127.92
127.50
127.37
126.84
124.73
123.63
123.02
118.48
77.29
76.97
76.65
47.27
27.13




N
O
Br
Br

3b


BBFO1, 400, 2246-1
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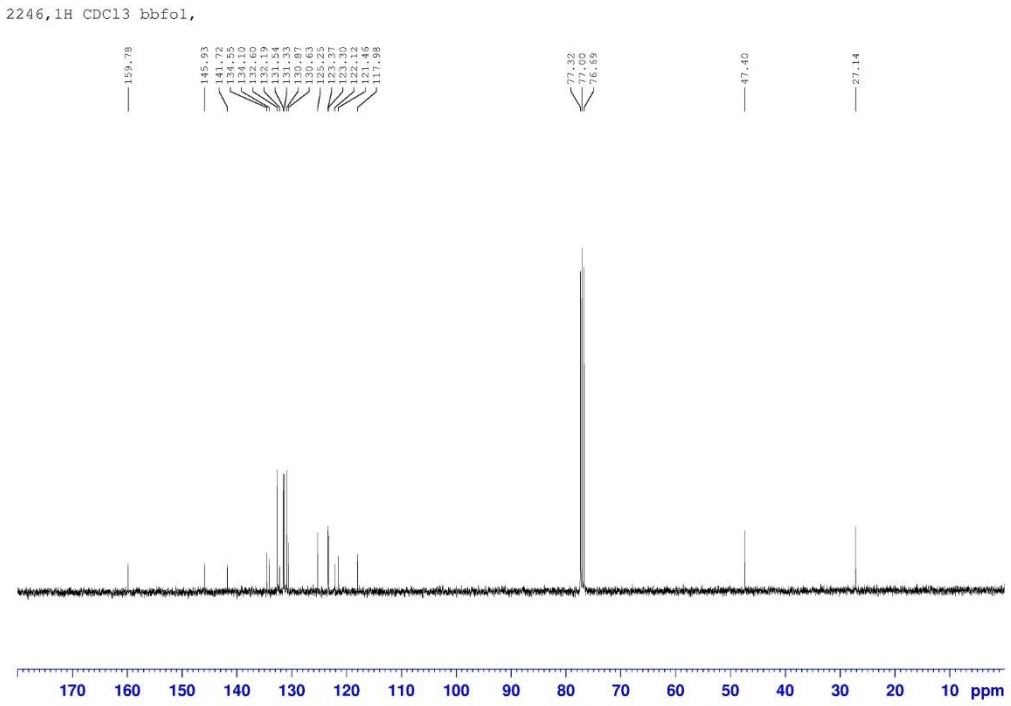
7.342
7.322
7.109
7.089
7.071
7.064
7.045
7.007
7.001
6.986
6.981
4.555
4.535
4.514
3.514
3.493
3.473
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
1.99
3.03
2.21
4.02
2.02
2.03


2246,1H CDCl3 bbfo1,
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145.93
141.72
134.55
134.10
132.60
132.19
131.54
131.33
130.87
130.63
125.25
123.37
123.30
122.12
121.46
117.98
77.32
77.00
76.69
47.40
27.14
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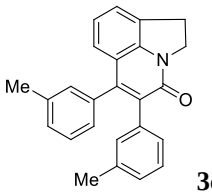
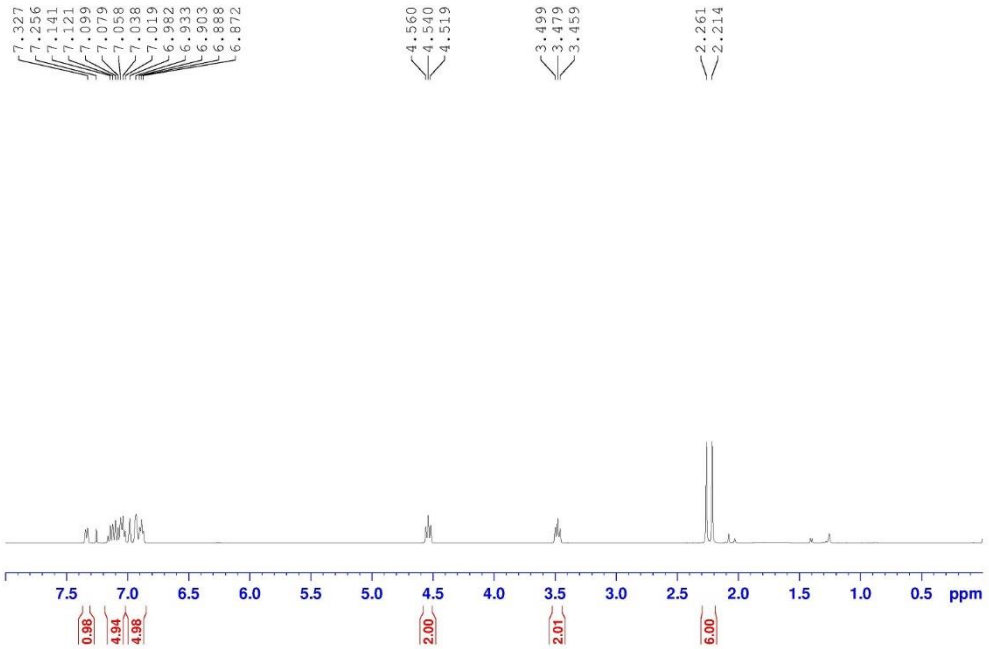
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

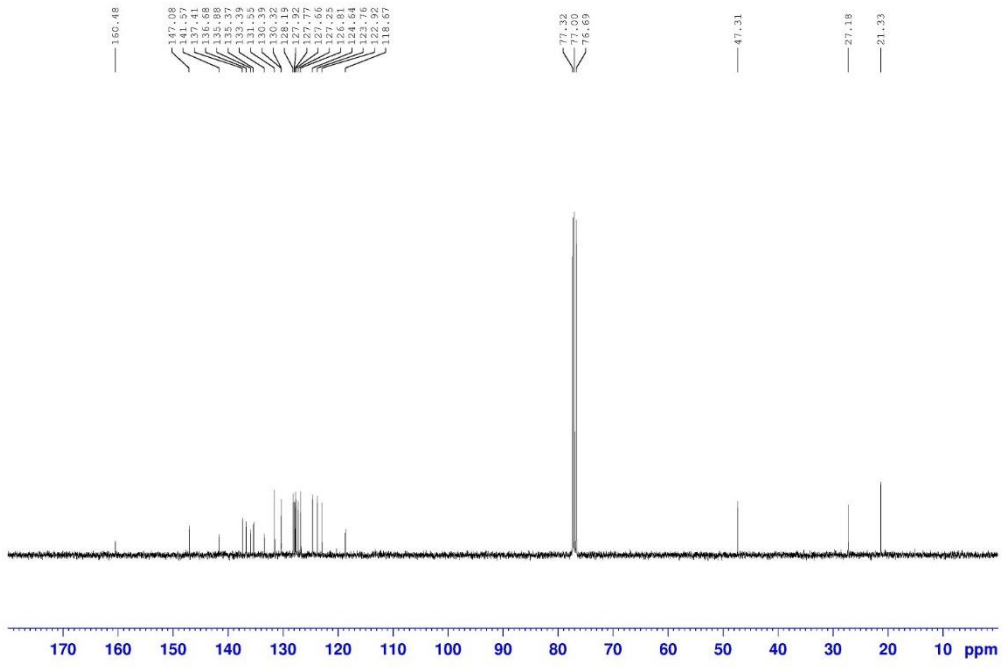


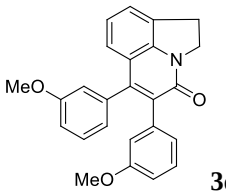
3c

2244-2, AV 400MHz, Nov14



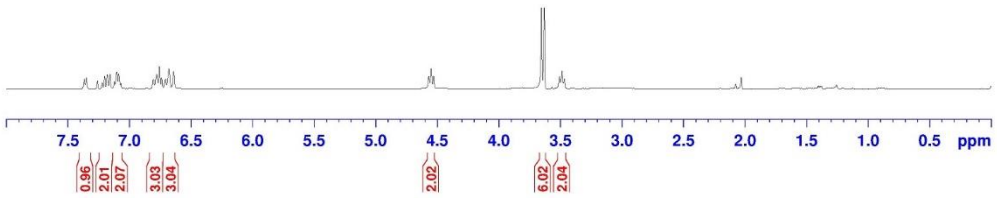
2244-2, AV 400MHz, Nov14



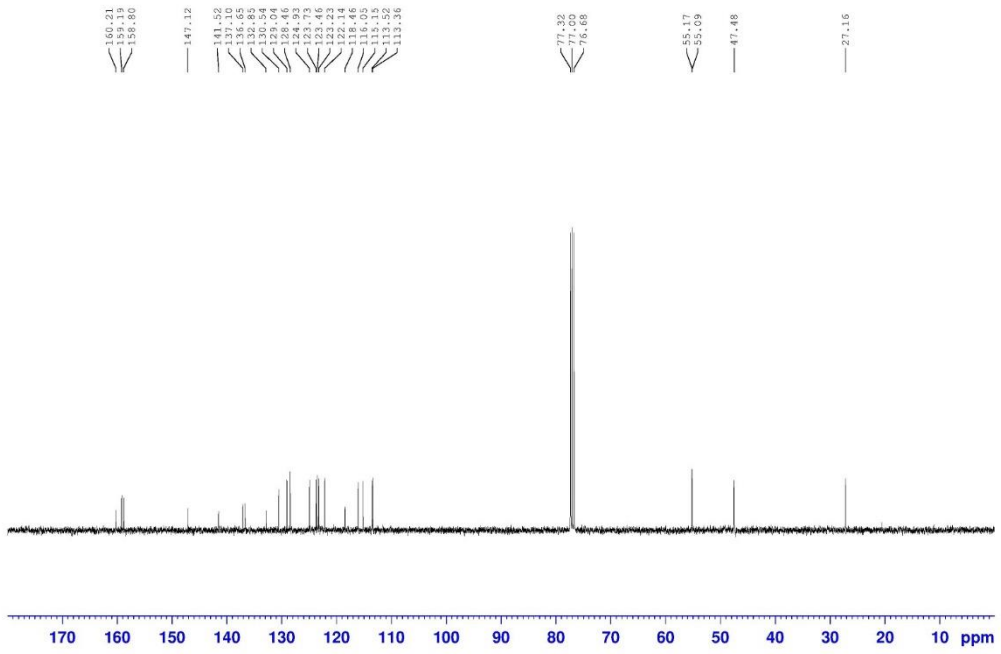


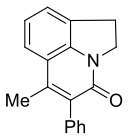
3d

2244-2, AV 400MHz, Nov14



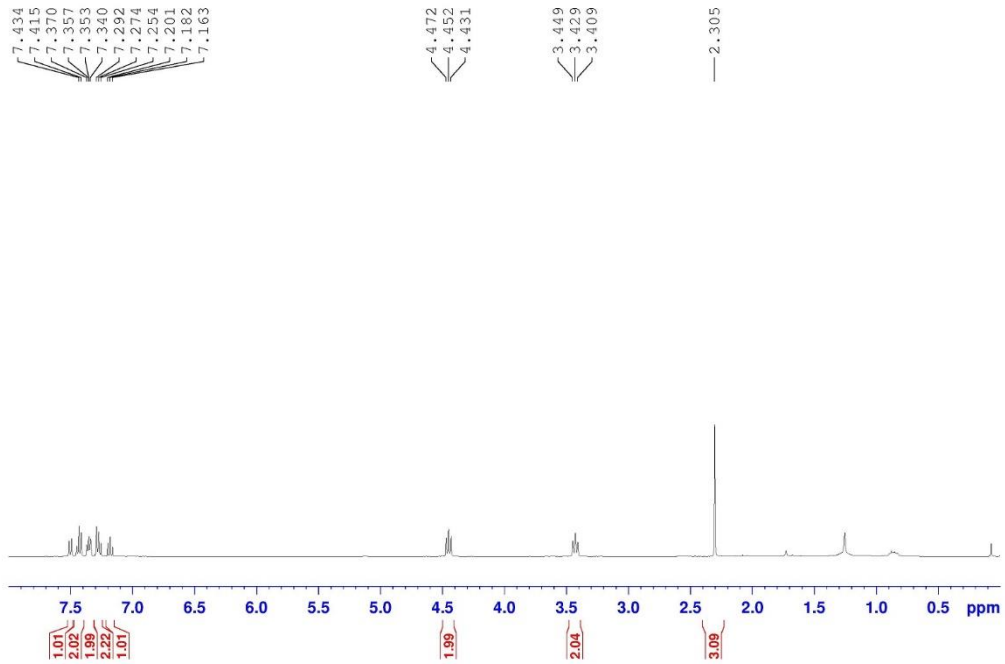
2244-2, AV 400MHz, Nov14



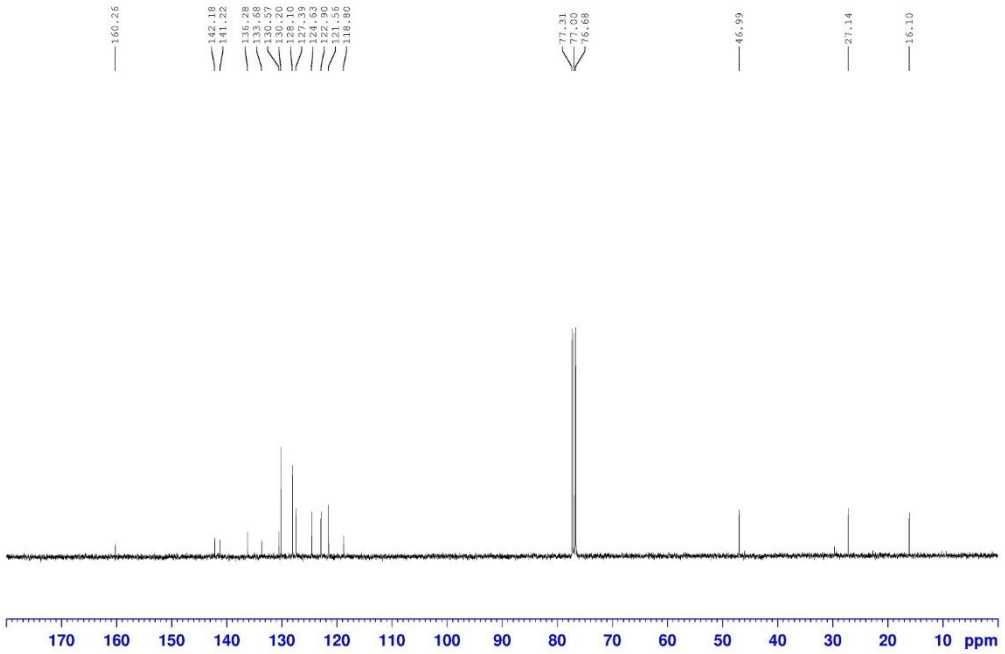


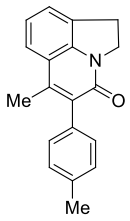
3e

2008-10,BBFO1, MAY 14



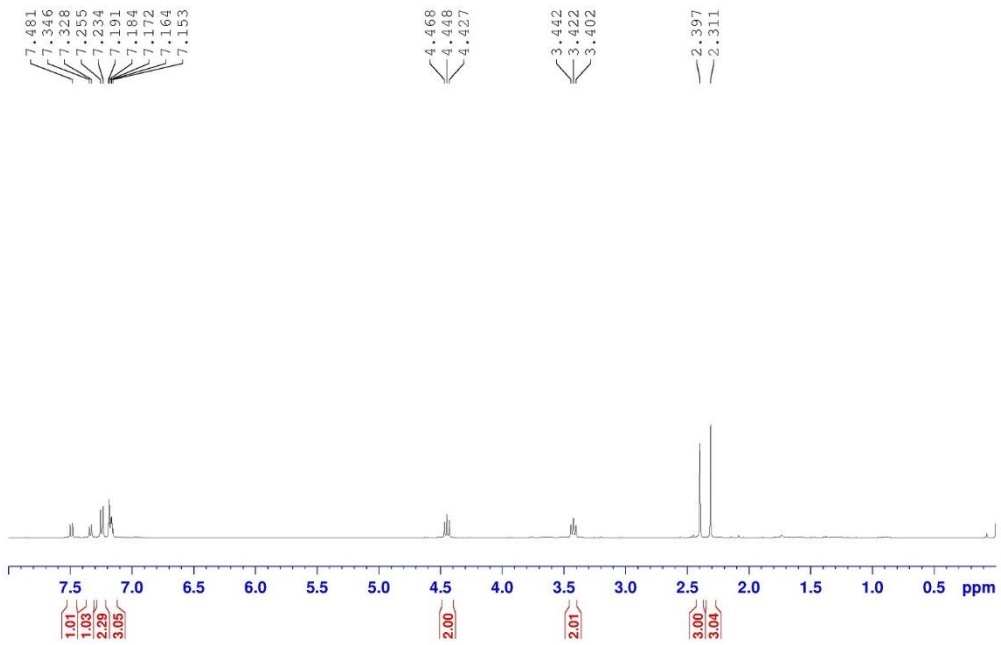
2008-10,BBFO1, MAY 14



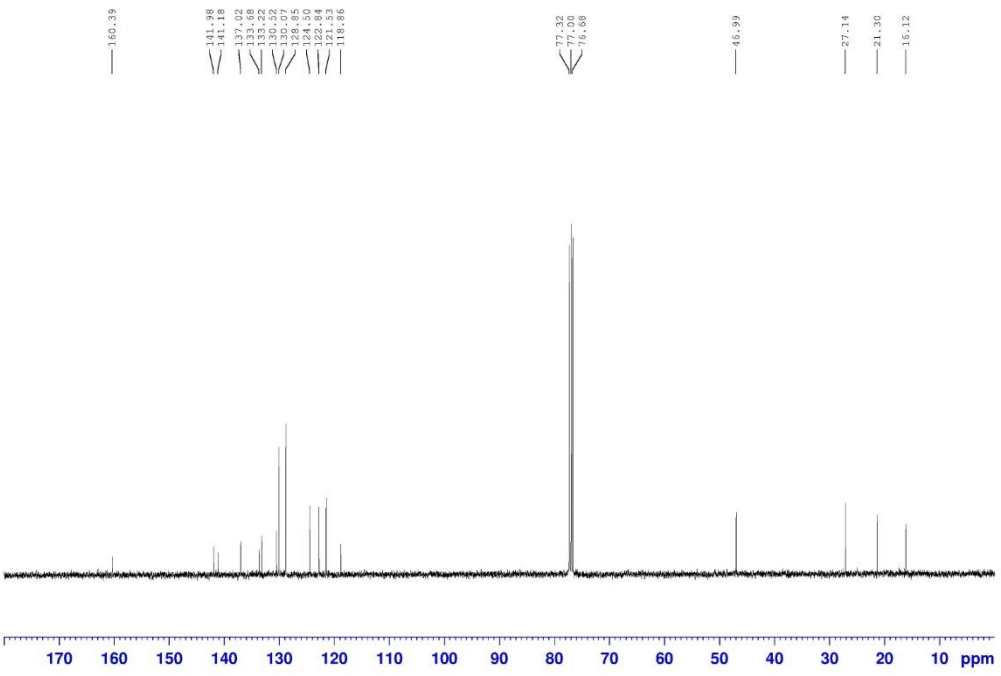


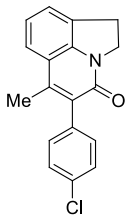
3f

2054-20 BBFO 1



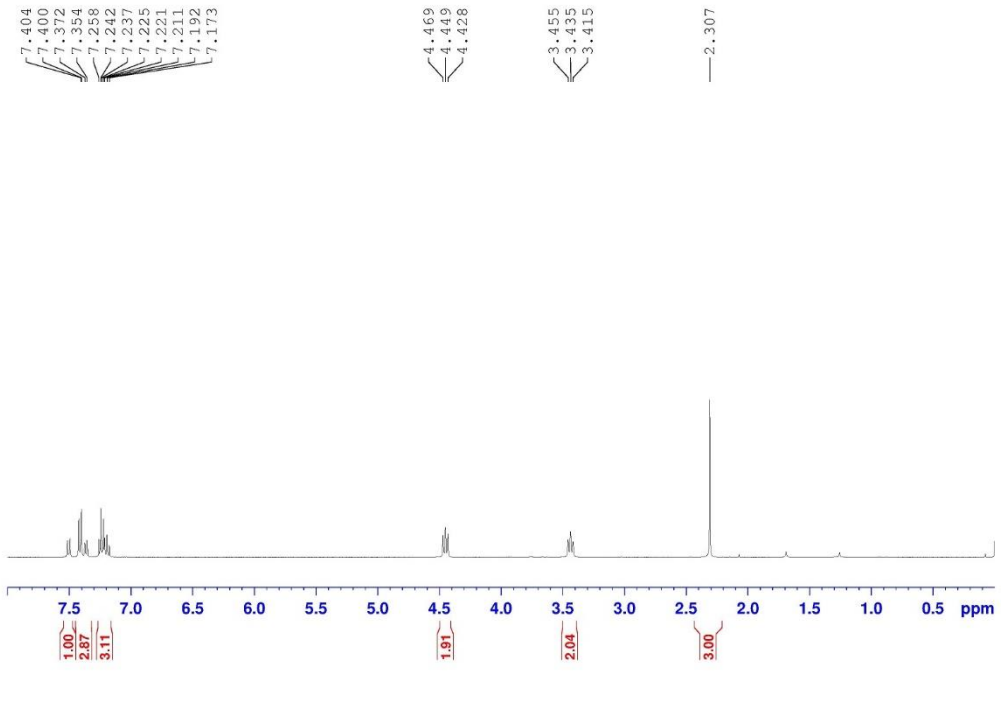
2054-20 BBFO 1



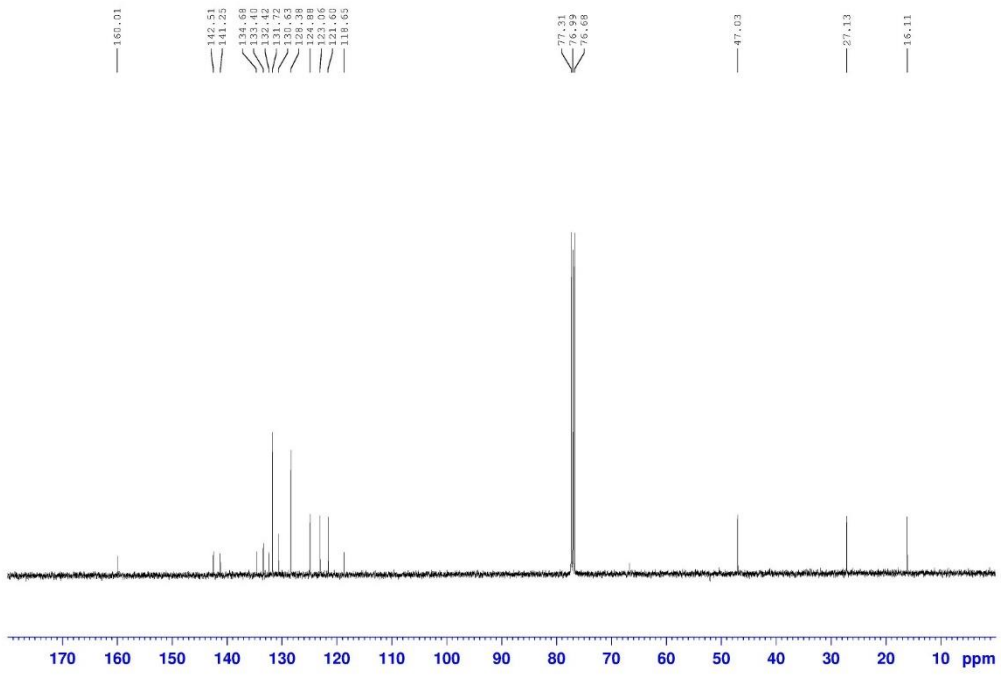


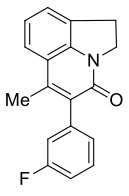
3g

2056-20a BBFO 1

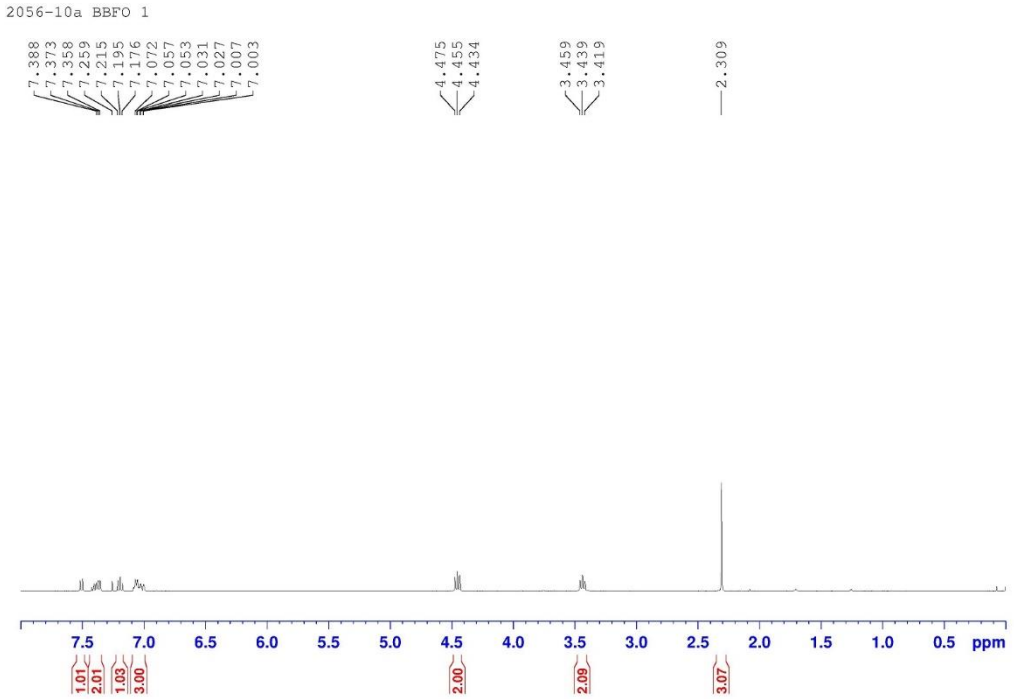


lbsC4-Me-ketone-XH BBFO 1




N
Me
O
F

3h


2056-10a BBFO 1
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7.027
7.007
7.003
4.475
4.455
4.434
3.459
3.439
3.419
2.309
7.5
7.0
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6.0
5.5
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4.0
3.5
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ppm
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1.03
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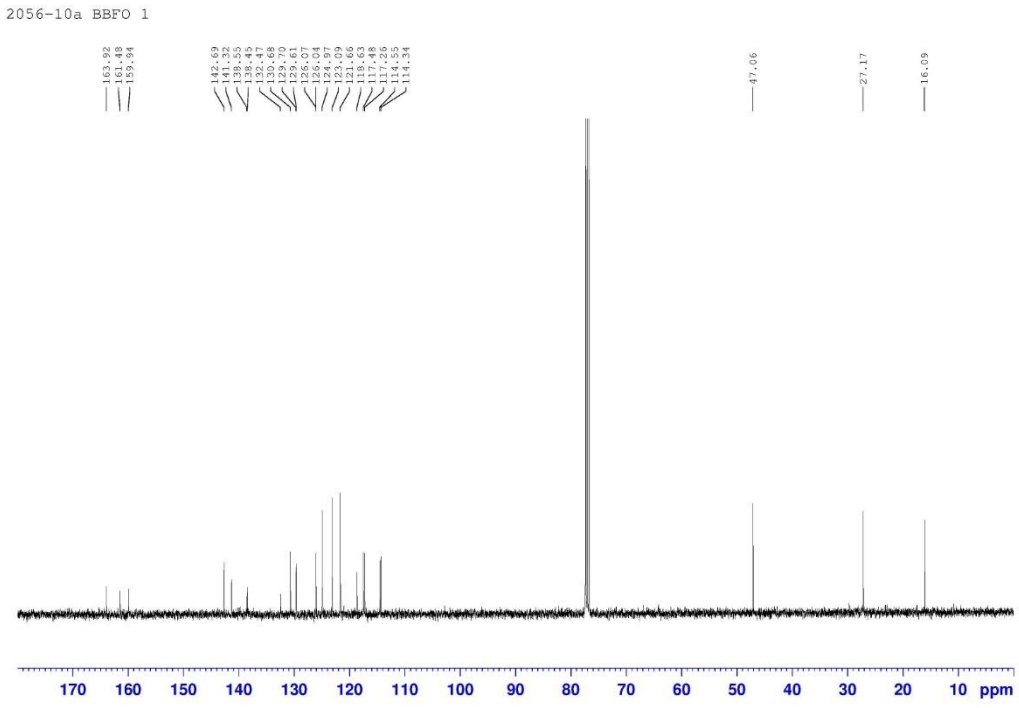
2.09
3.07


2056-10a BBFO 1
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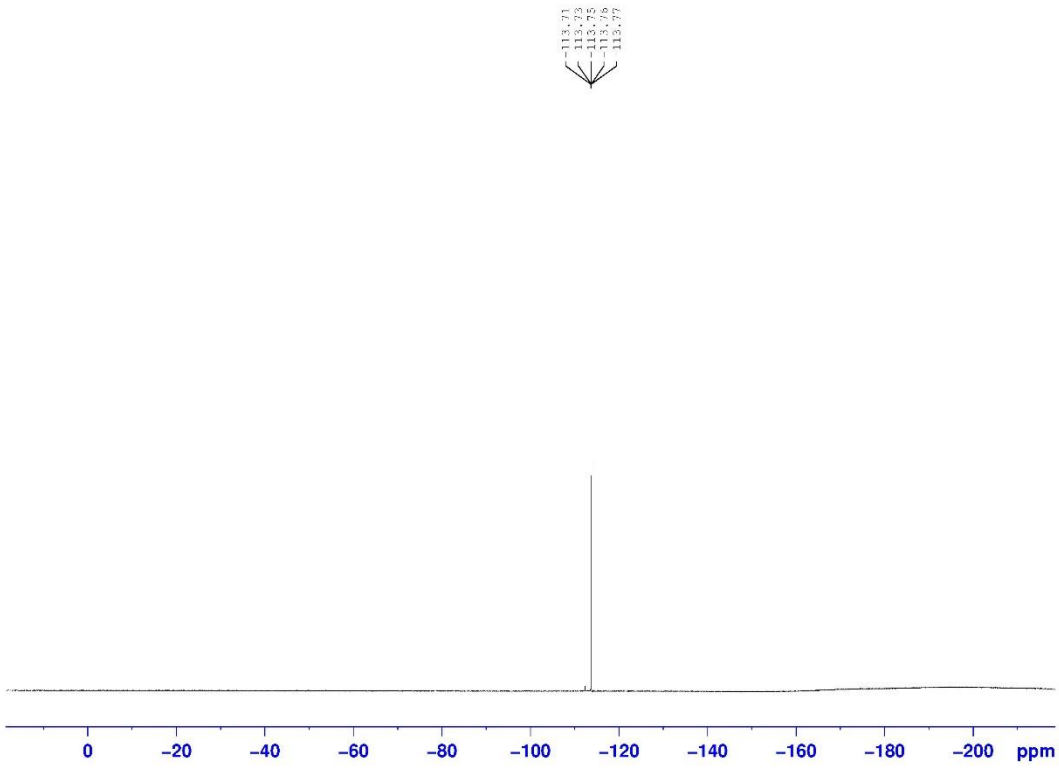
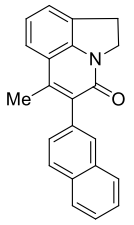
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

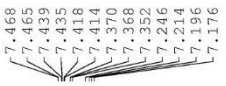
2056-10 BBFO 1

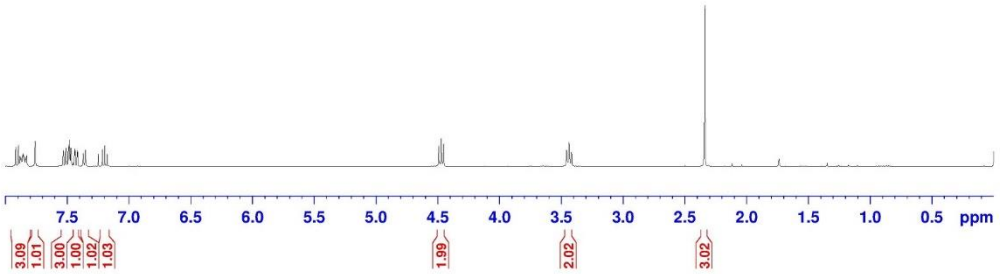




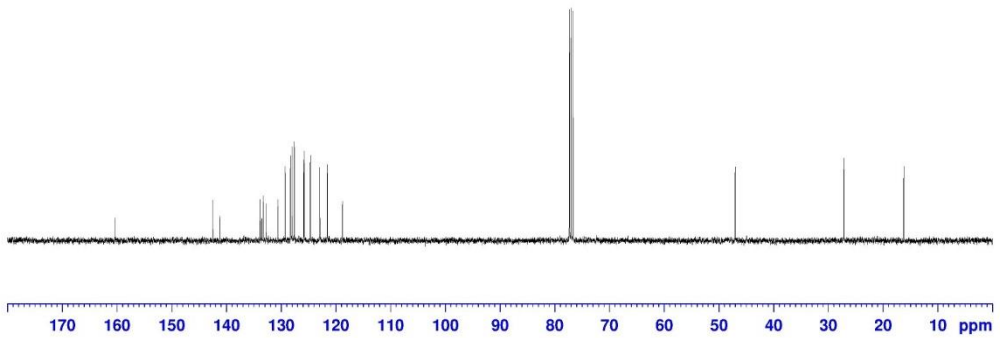
3i

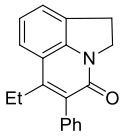
2064-20, 1H, CDCl3, AV 400M, 20140605





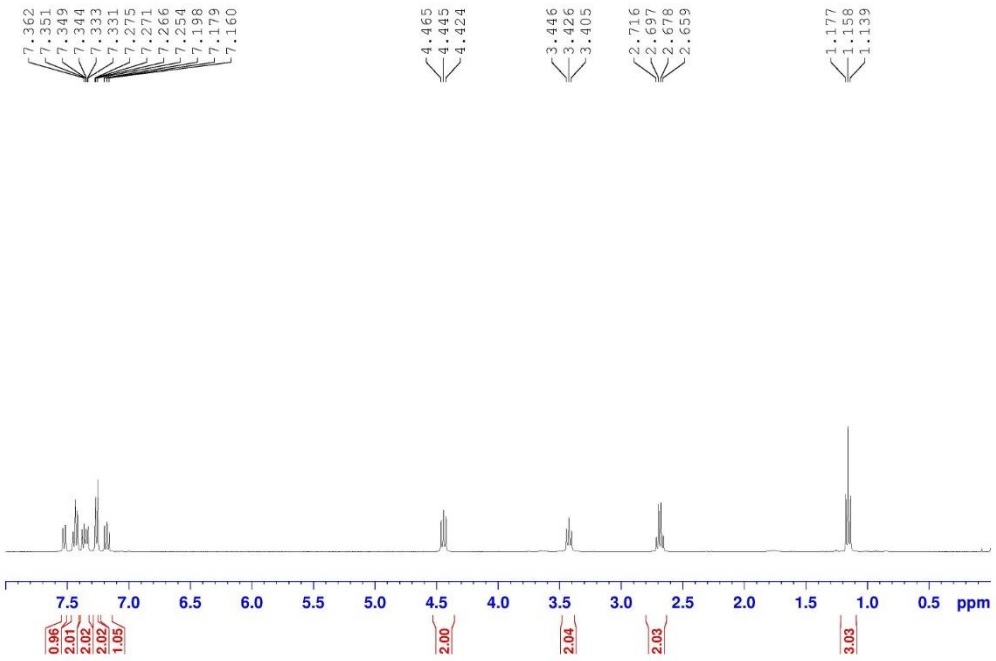
2064-20, 1H, CDCl3, AV 400M, 20140605



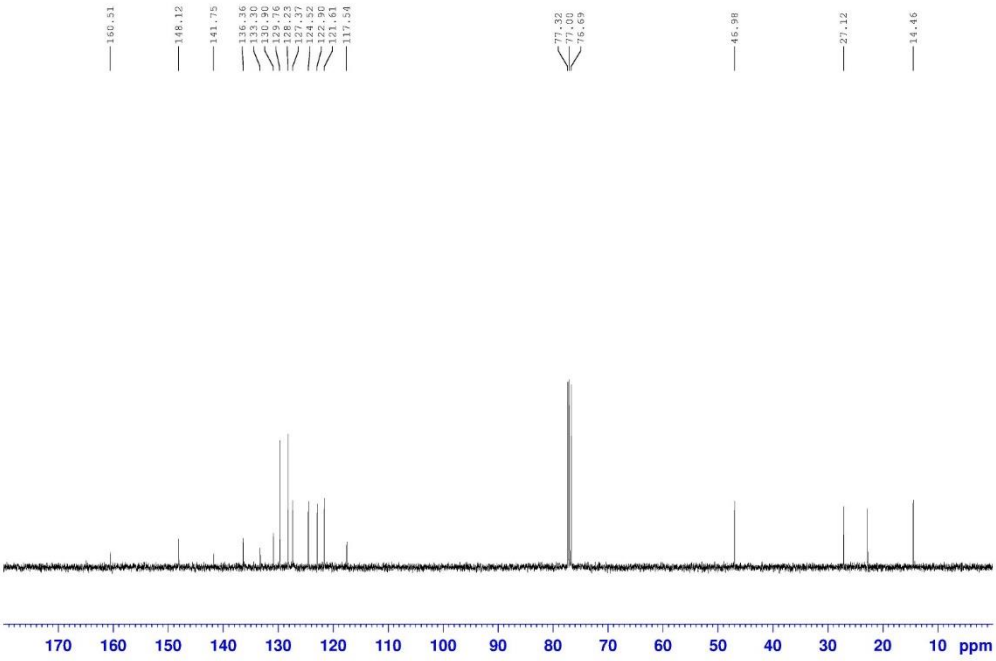


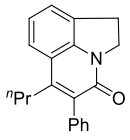
3j

2030-20-1,BBFO 1



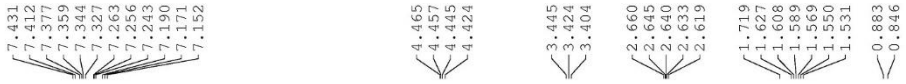
2030-20-1, 1H, CDCl3, AV 400M, 20140605

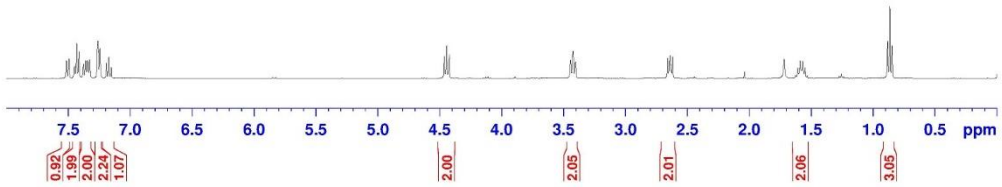




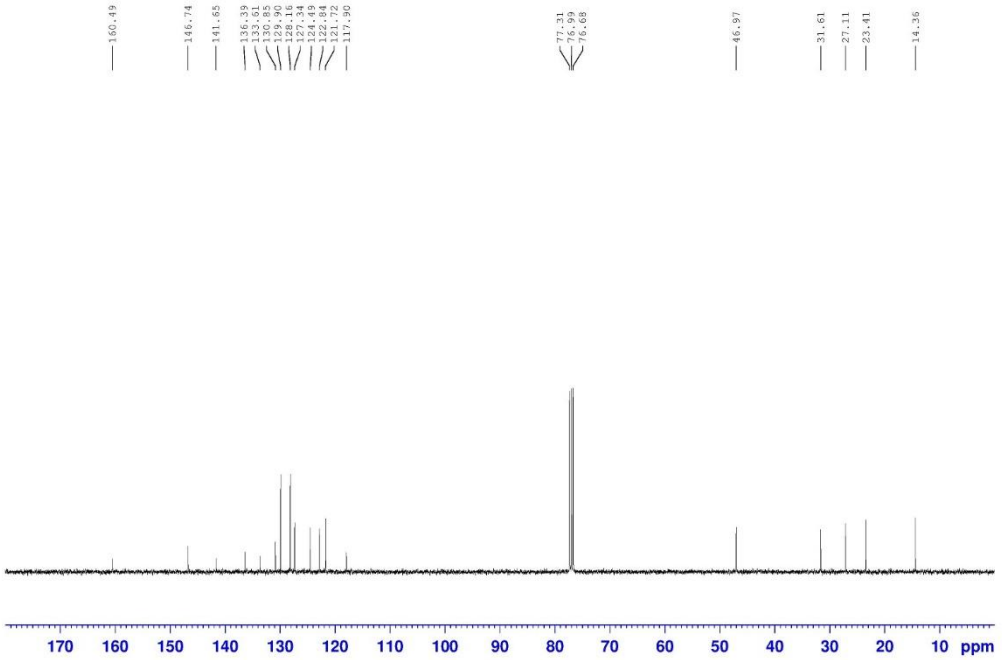
3k

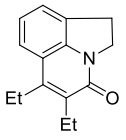
2030-20-2, 1H, CDCl3, AV 400M, 20140605





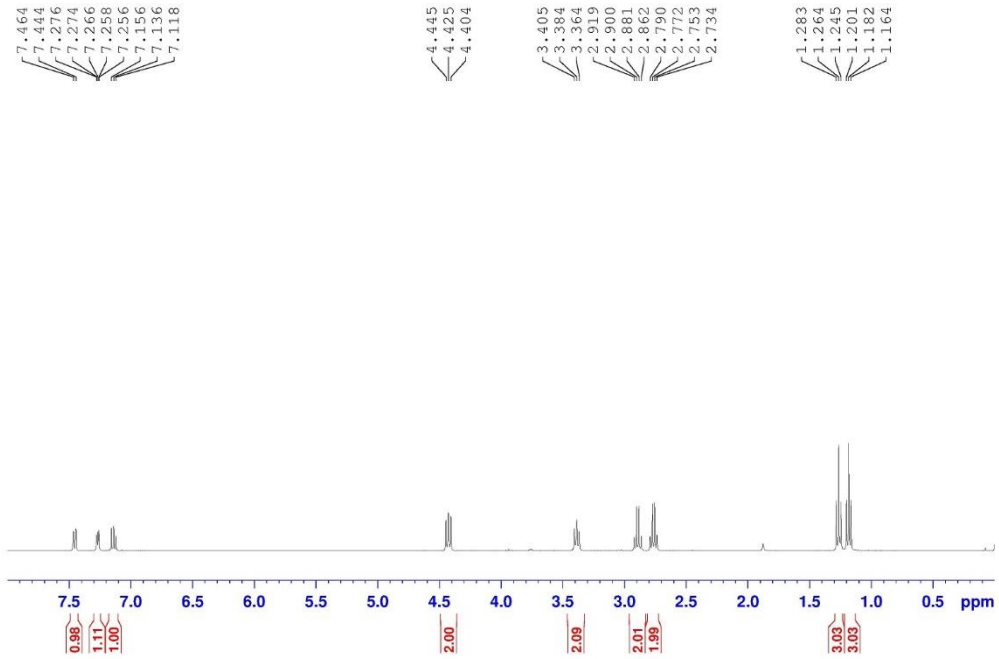
2030-20-2, 1H, CDCl3, AV 400M, 20140605



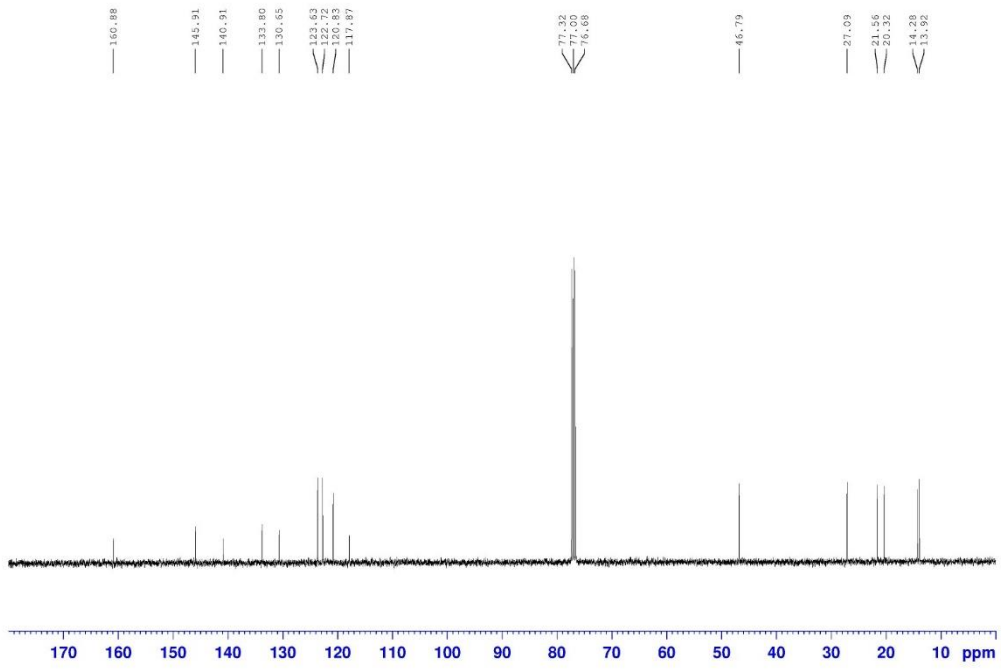


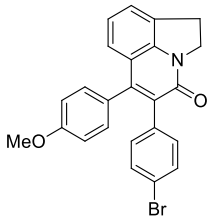
3l

2036-20 BBFO 1

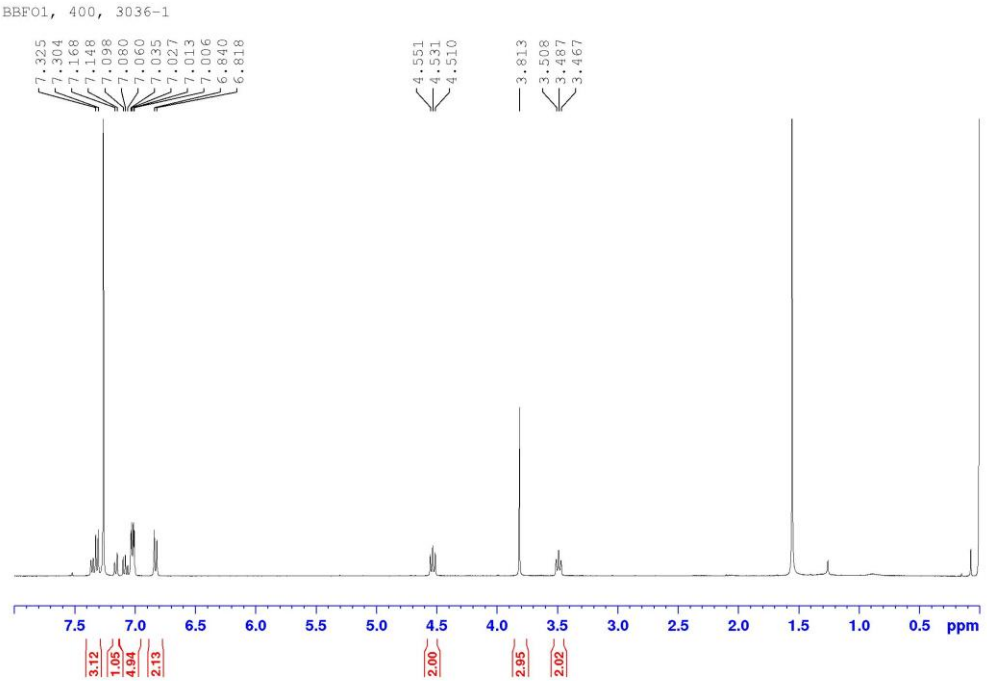


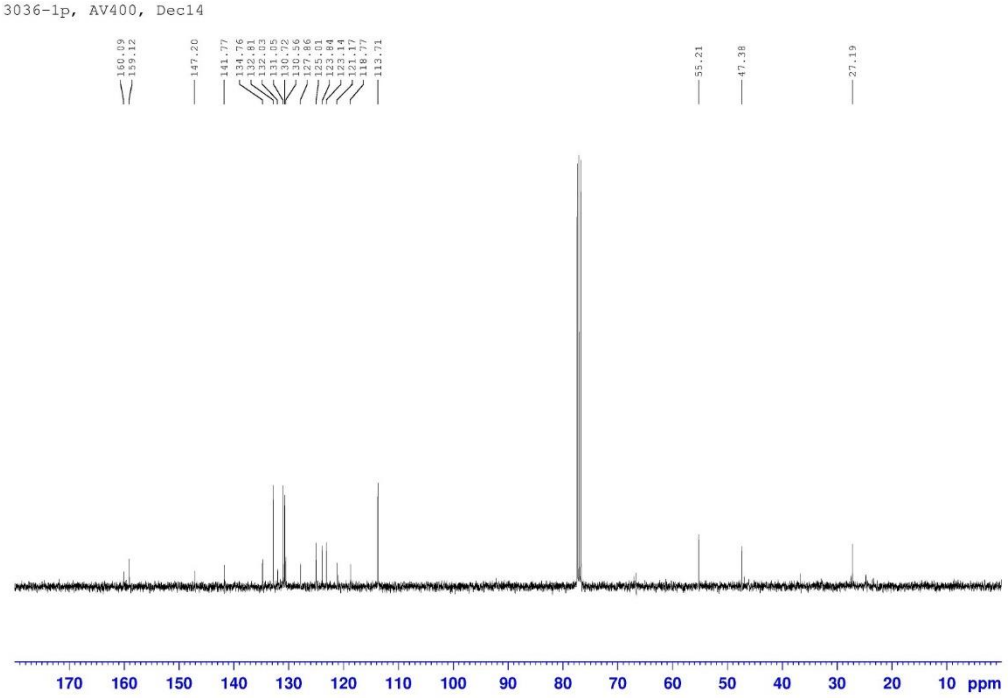
2036-20 BBFO 1

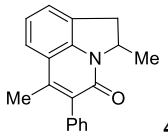



N
O
MeO
Br

3m

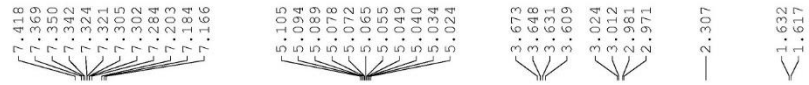

BBFO1, 400, 3036-1
7.325
7.304
7.168
7.148
7.098
7.080
7.060
7.035
7.027
7.013
7.006
6.840
6.818
4.551
4.531
4.510
3.813
3.508
3.487
3.467
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
3.12
1.05
4.64
2.13
2.00
2.95
2.02

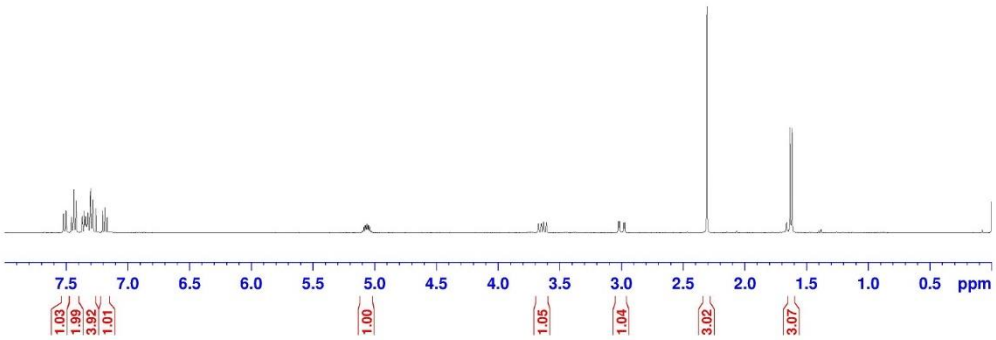

3036-1p, AV400, Dec14
160.09
159.12
147.20
141.77
132.76
132.41
132.03
131.05
130.72
129.56
127.66
125.01
123.84
123.14
121.17
116.77
113.71
55.21
47.38
27.19
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm



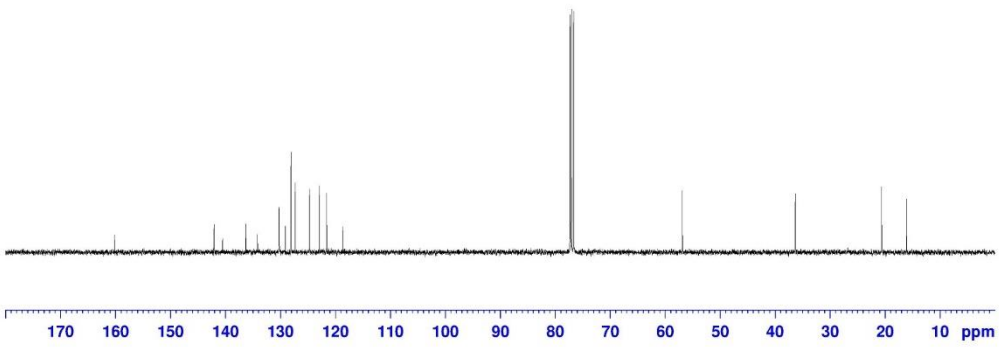
4a

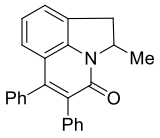
2064-10, 1H, CDCl3, AV 400M, 20140605





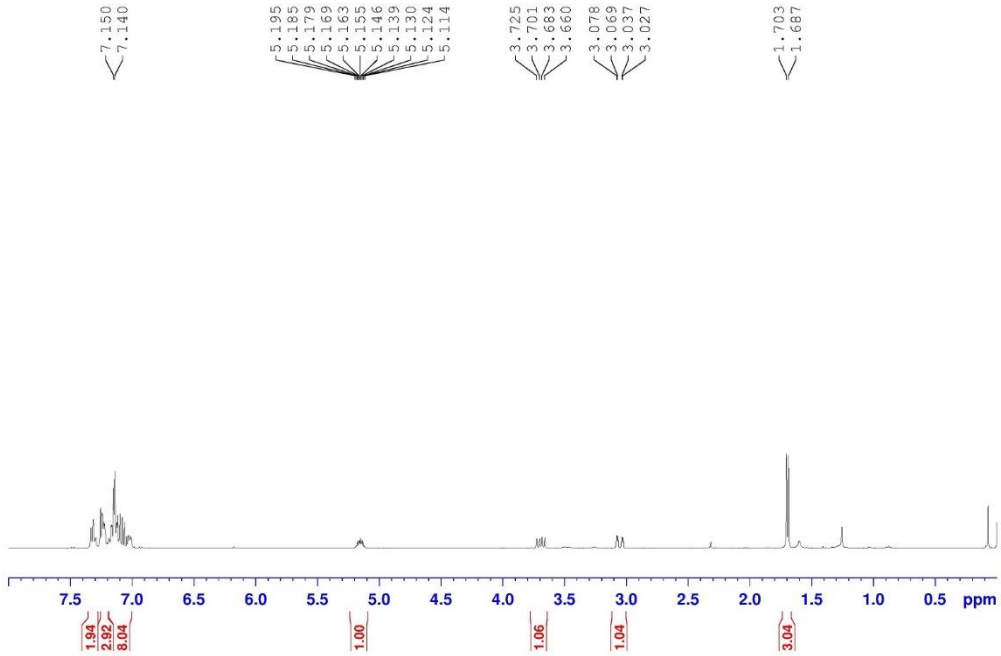
2064-10, 1H, CDCl3, AV 400M, 20140605



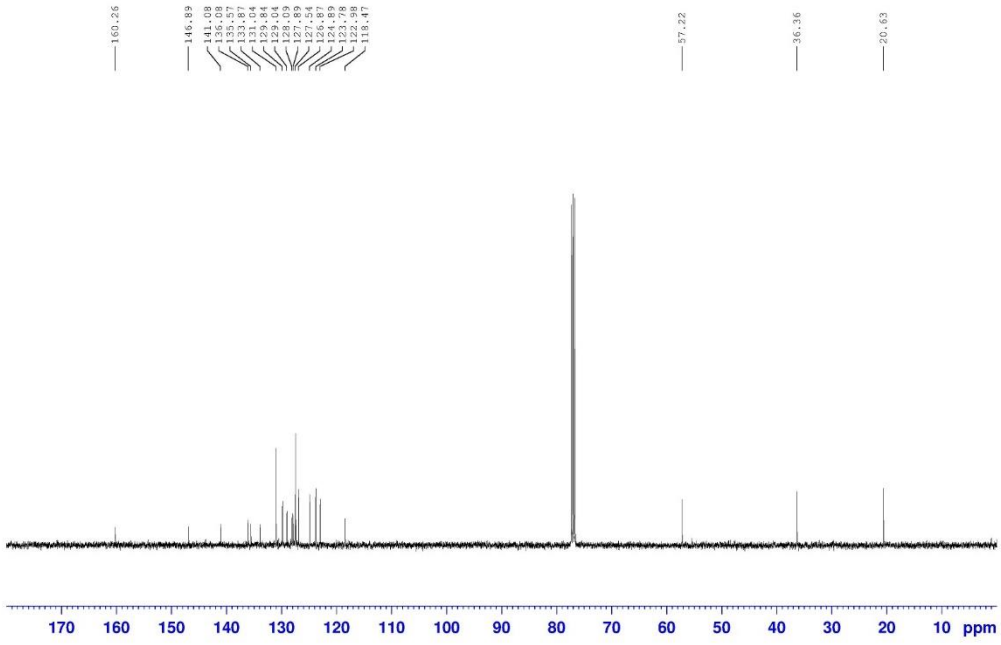


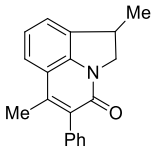
4b

2184-10, AV400, sep 14



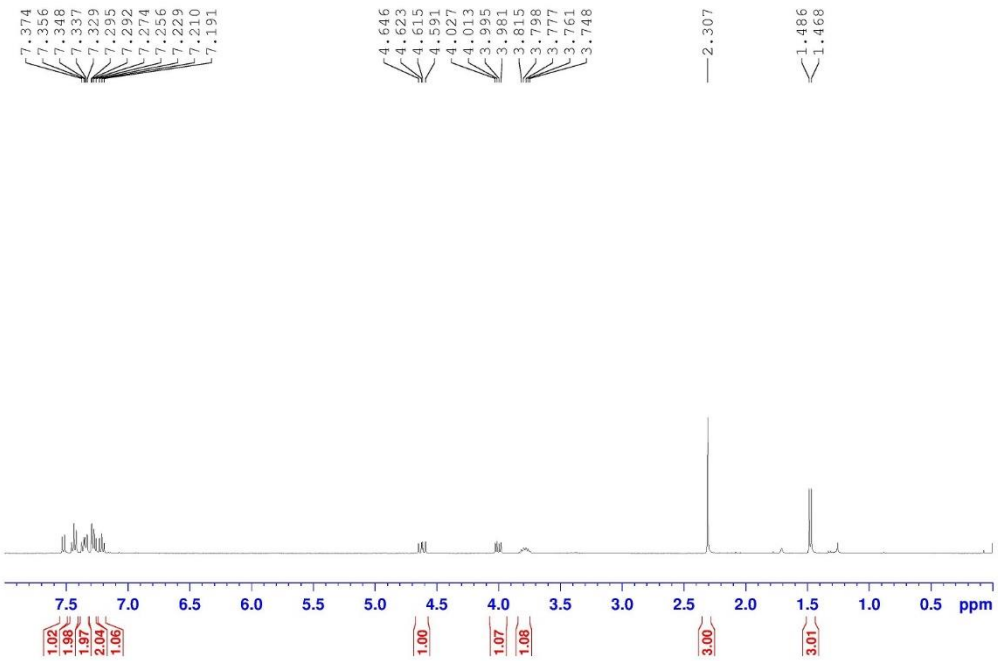
2184-10, AV400, sep 14



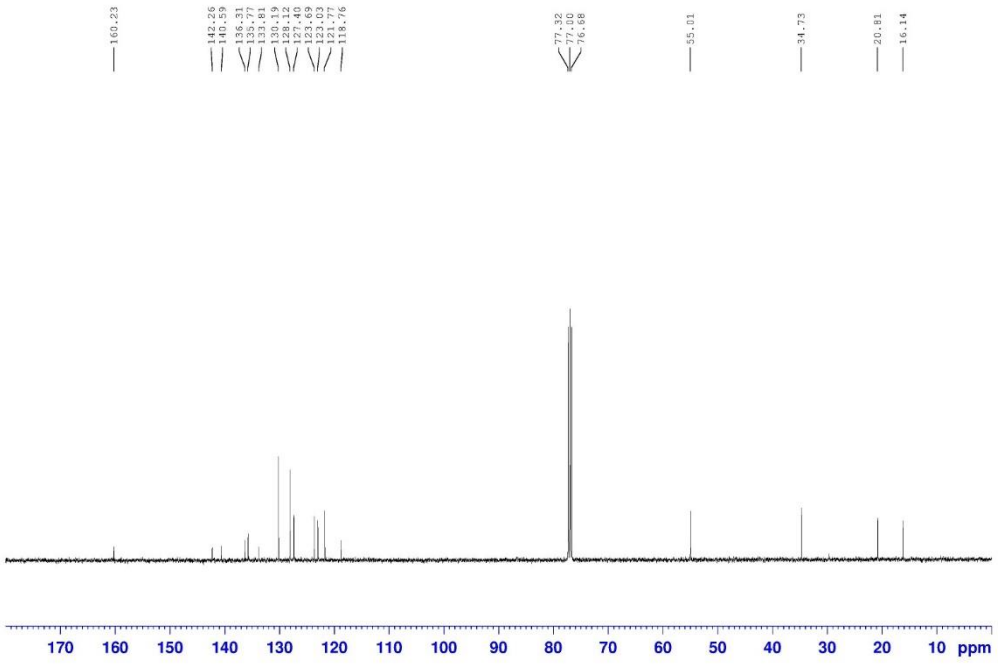


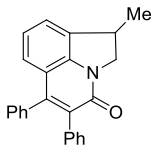
4c

2076-10A, 1H NMR, CDCl3, BBFO-01, Jul 14



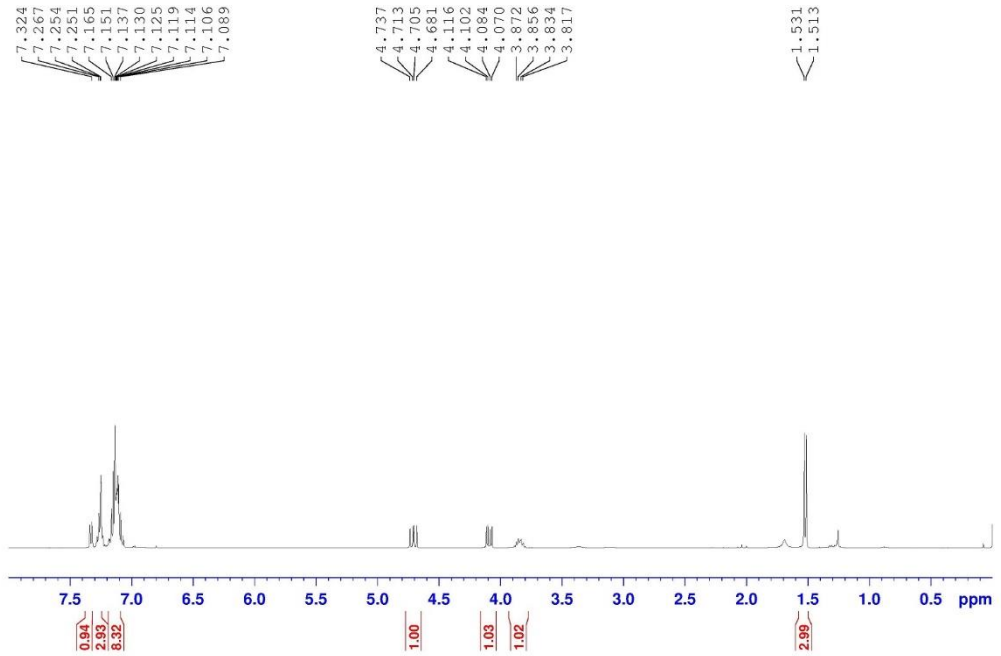
2076-10A, 1H NMR, CDCl3, BBFO-01, Jul 14



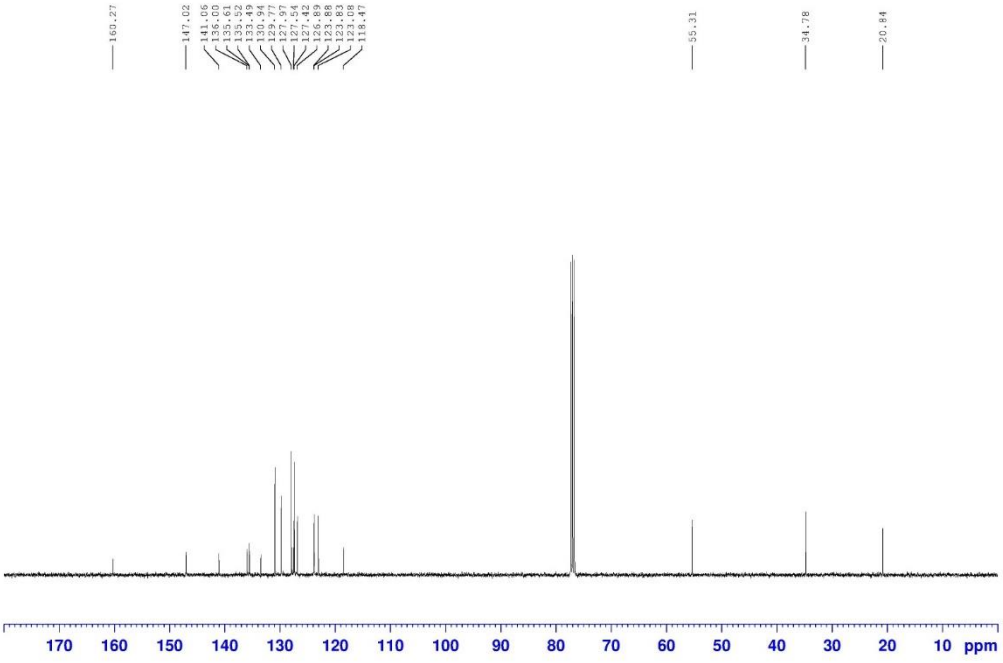


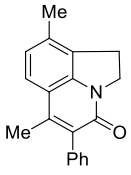
4d

2184-10, AV400, sep 14



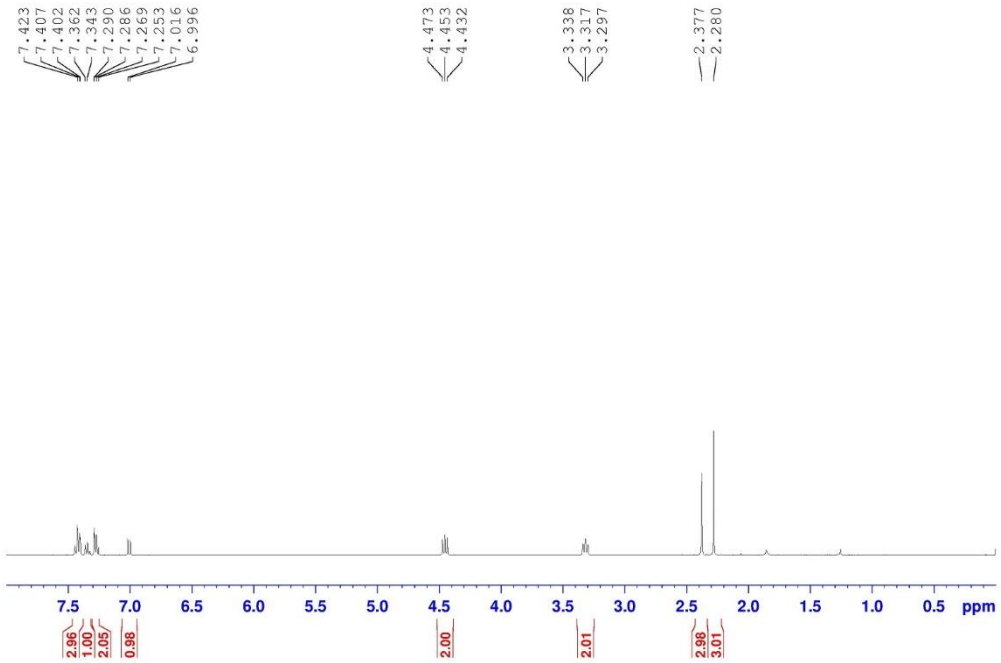
2184-10, AV400, sep 14



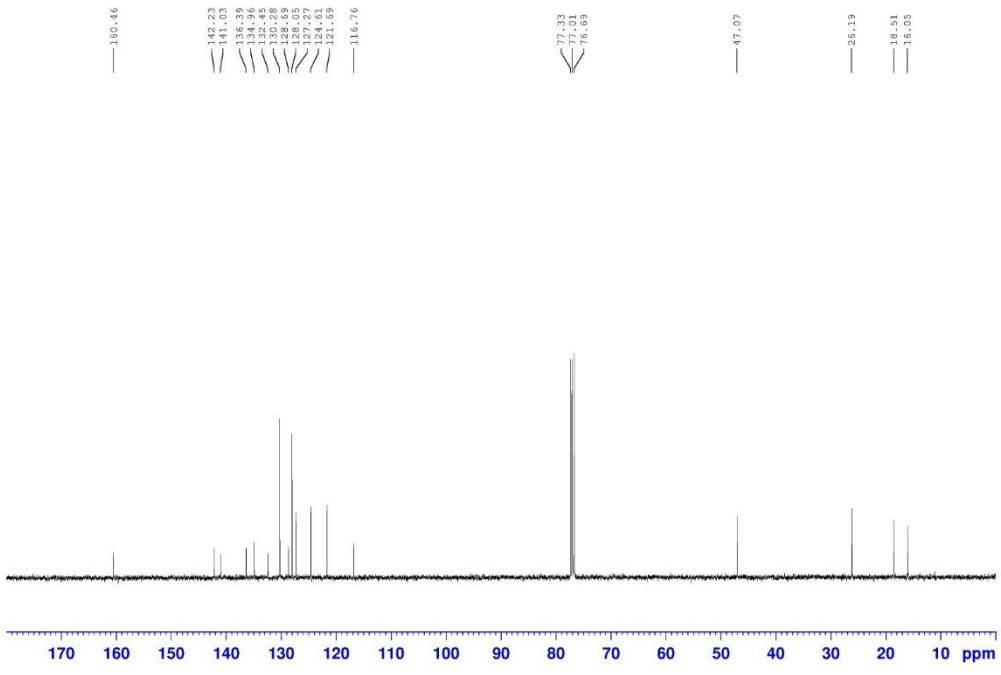


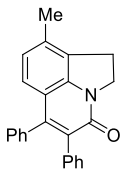
4e

2094-20, 1H NMR, CDCl3, BBFO-01, Jul 14



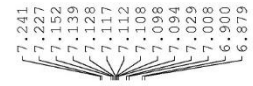
2094-20, 1H NMR, CDCl3, BBFO-01, Jul 14

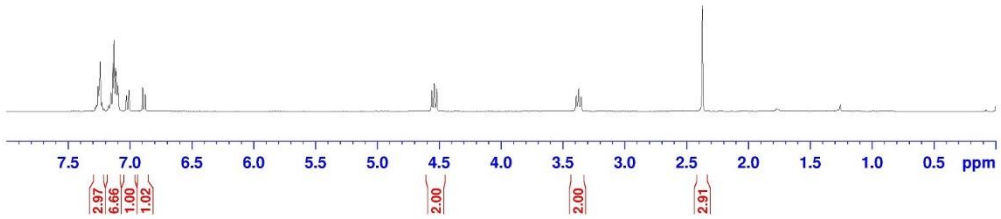




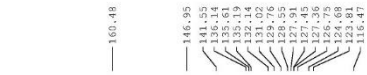
4f

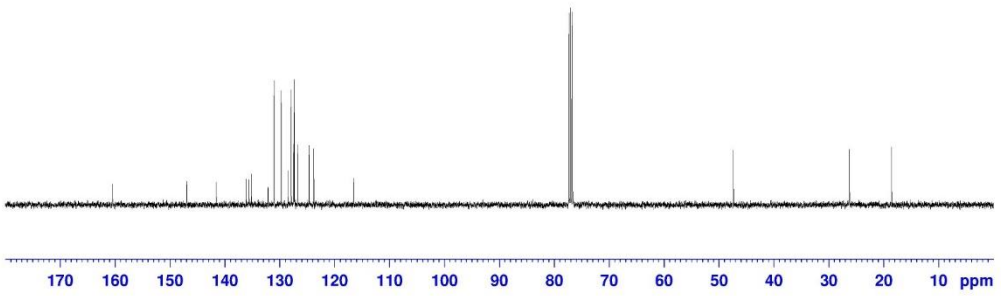
2194-2, AV400, sep 14

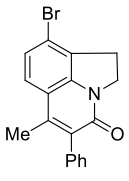




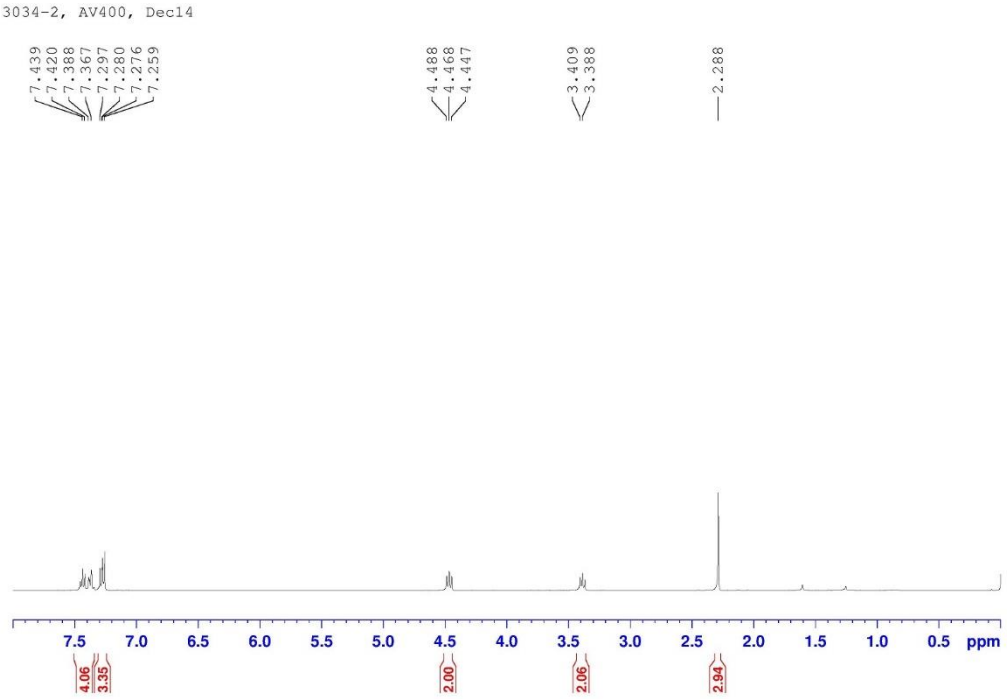
2194-2, AV400, sep 14

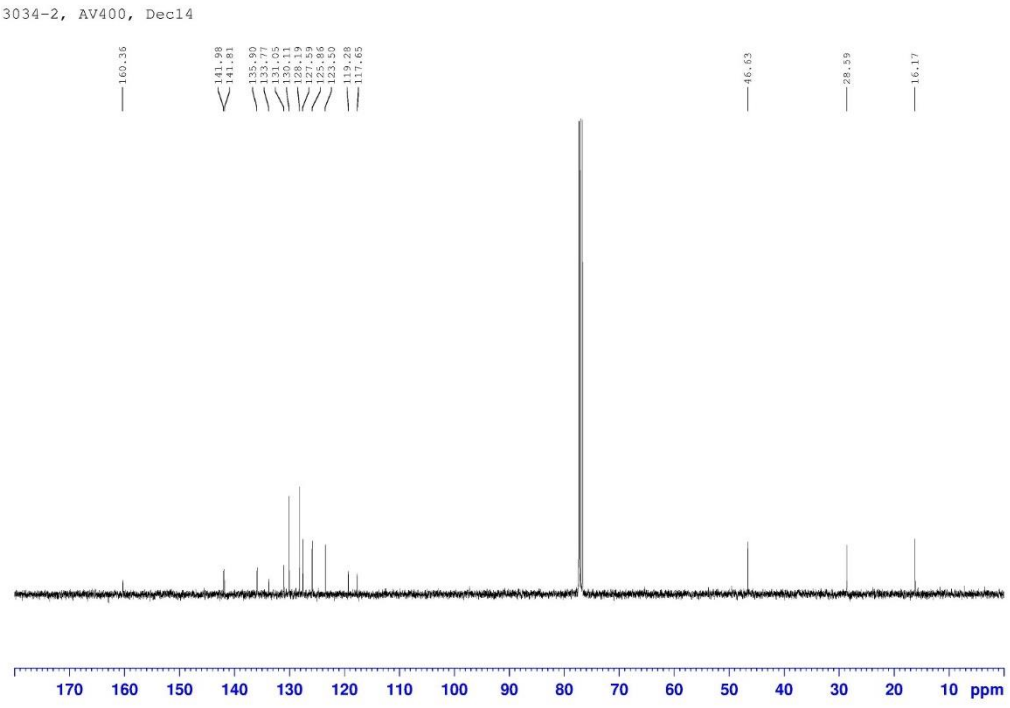


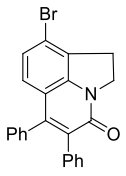



Br
N
O
Me
Ph

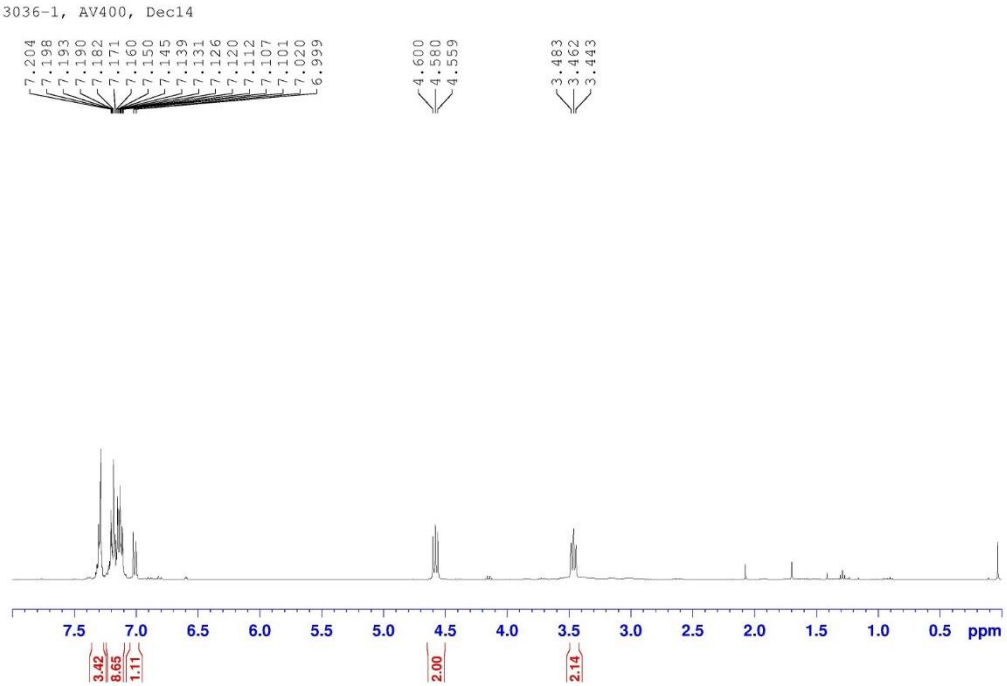
4g

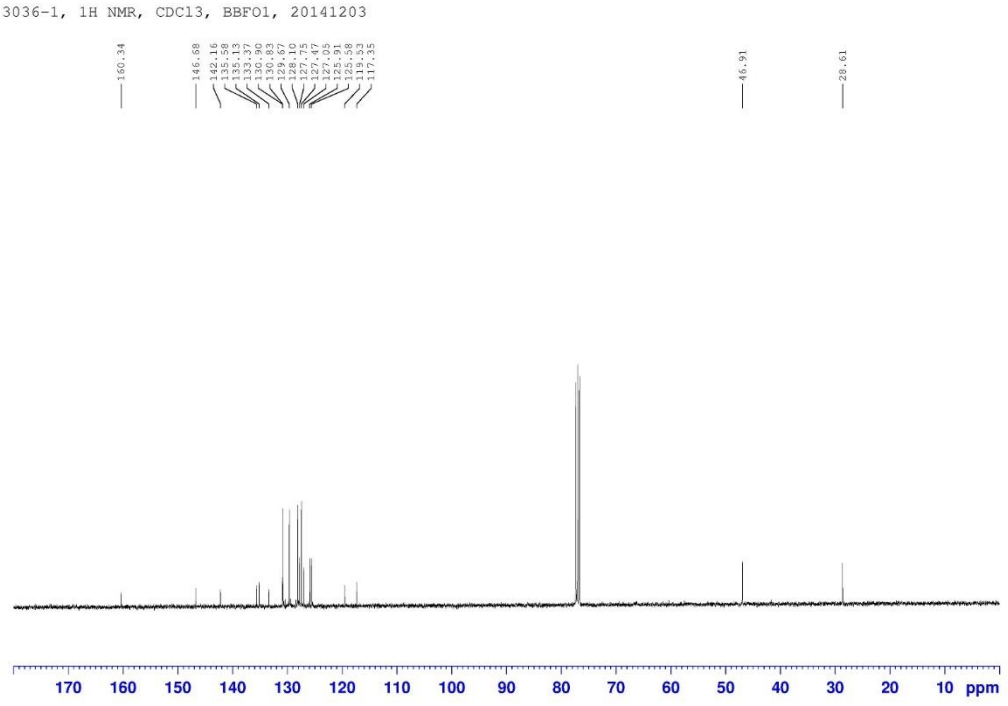

3034-2, AV400, Dec14
7.439
7.420
7.388
7.367
7.297
7.280
7.276
7.259
4.488
4.468
4.447
3.409
3.388
2.288
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
4.06
3.35
2.00
2.06
2.94

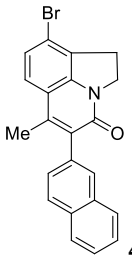

3034-2, AV400, Dec14
160.36
141.98
141.81
135.90
133.77
131.05
130.11
128.19
127.59
125.86
123.50
119.28
117.65
46.63
28.59
16.17
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


Br
N
O
Ph
Ph

4h

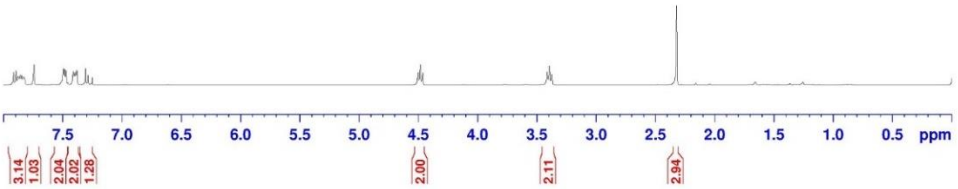

3036-1, AV400, Dec14
7.204
7.198
7.193
7.190
7.182
7.171
7.160
7.150
7.145
7.139
7.131
7.126
7.120
7.112
7.107
7.101
7.020
6.999
4.600
4.580
4.559
3.483
3.462
3.443
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
3.42
8.65
1.11
2.00
2.14


3036-1, 1H NMR, CDCl3, BBFO1, 20141203
160.34
146.68
142.16
135.58
135.13
133.37
130.90
130.83
128.67
128.10
127.75
127.47
127.05
125.91
125.58
119.53
117.35
46.91
28.61
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

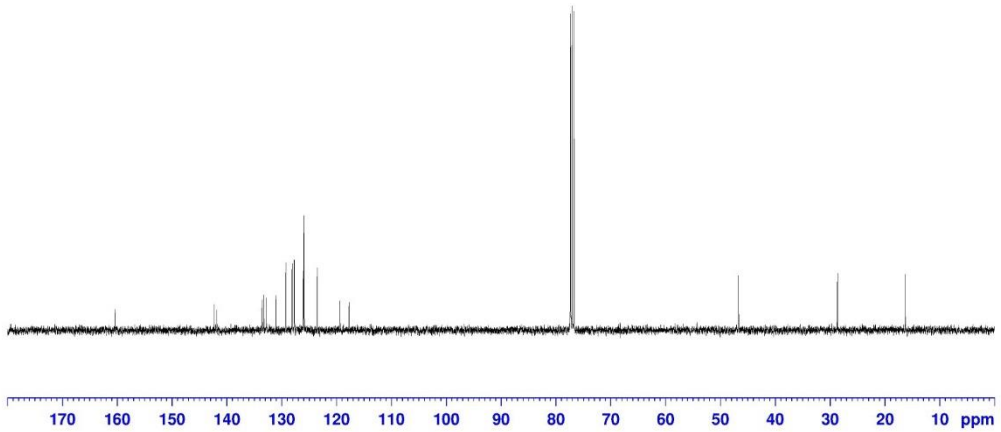


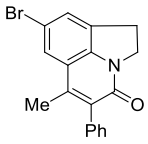
4i

3034-3, AV400, Dec14



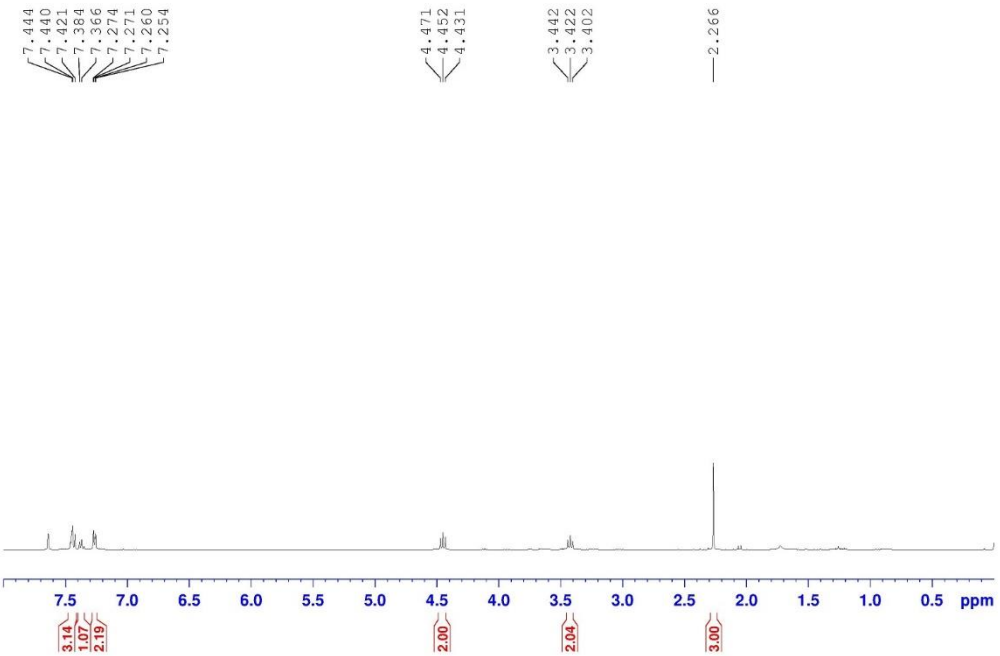
3034-3, AV400, Dec14



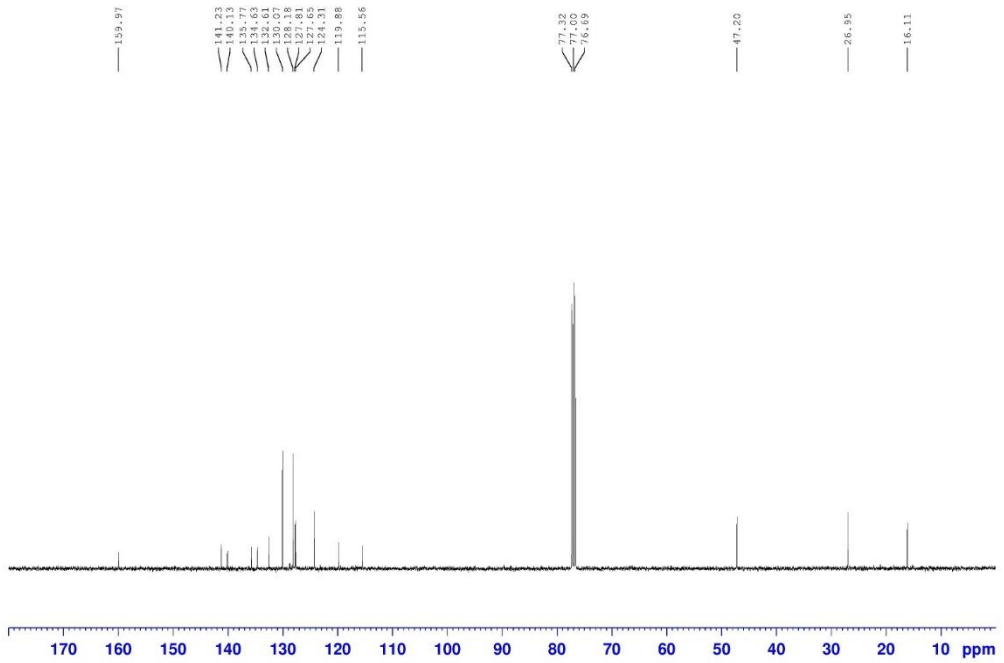


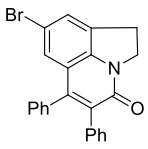
4j

2072-10a, 1H NMR, CDCl3, BBFO-01, Jul 14

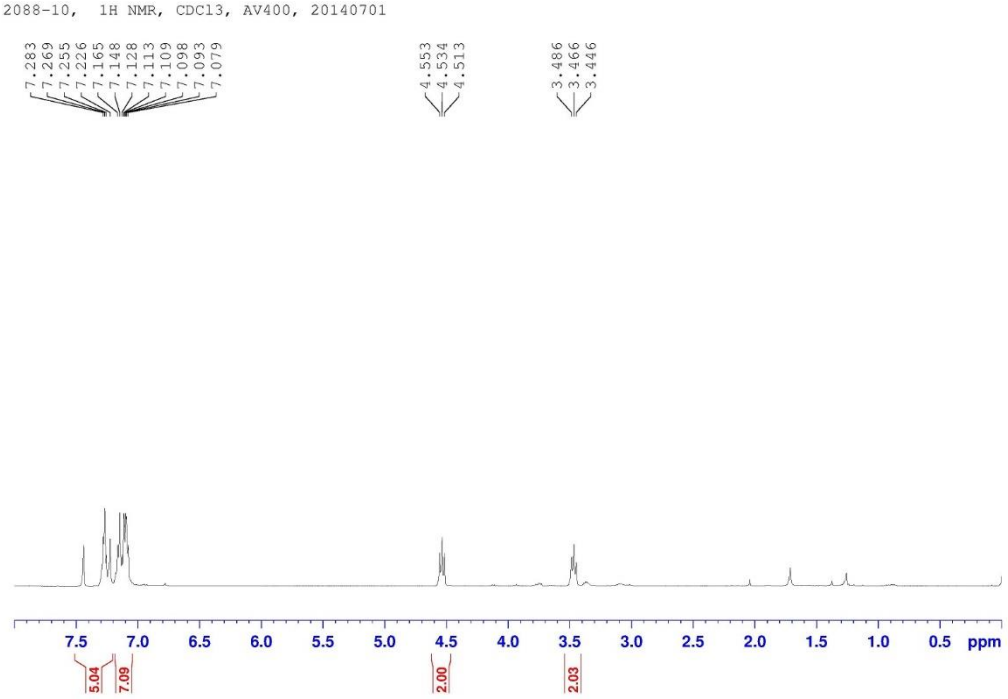


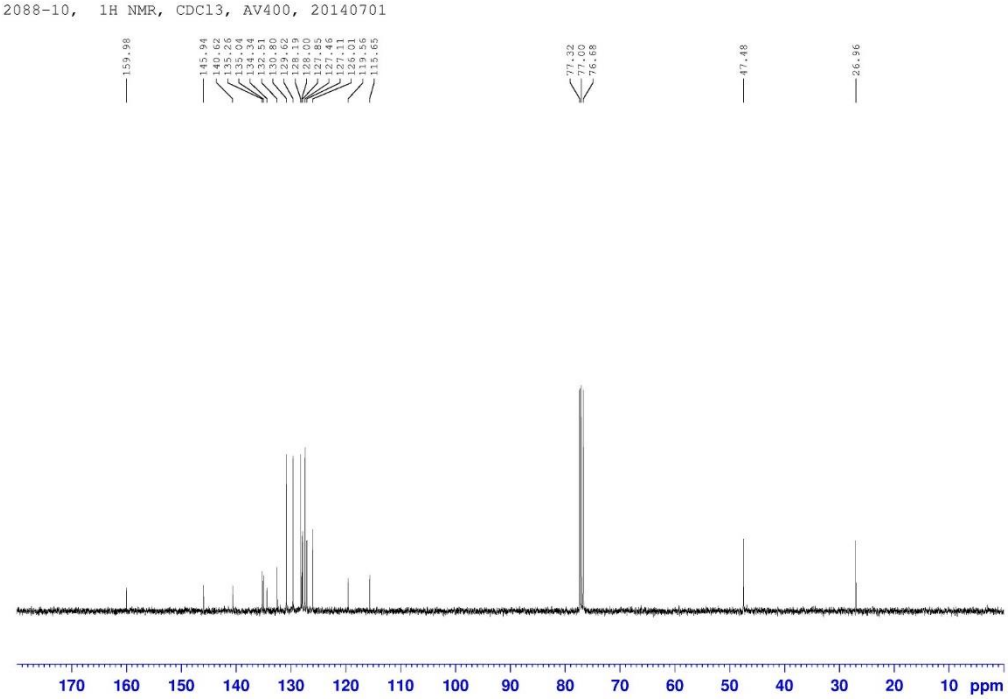
2072-10a, 1H NMR, CDCl3, BBFO-01, Jul 14

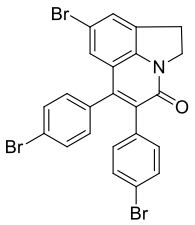



Br
N
O
Ph
Ph

4k

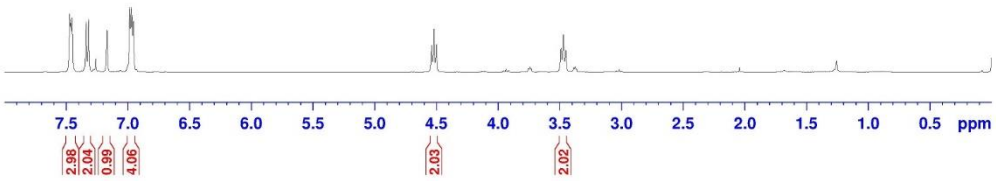

2088-10, 1H NMR, CDCl3, AV400, 20140701
7.283
7.269
7.255
7.226
7.165
7.148
7.128
7.113
7.109
7.098
7.093
7.079
4.553
4.534
4.513
3.486
3.466
3.446
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
5.04
7.09
2.00
2.03


2088-10, 1H NMR, CDCl3, AV400, 20140701
159.98
145.94
140.62
135.26
133.64
133.34
132.51
130.80
129.62
128.53
128.00
127.85
127.46
127.11
126.51
119.56
115.65
77.32
77.00
76.68
47.48
26.96
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

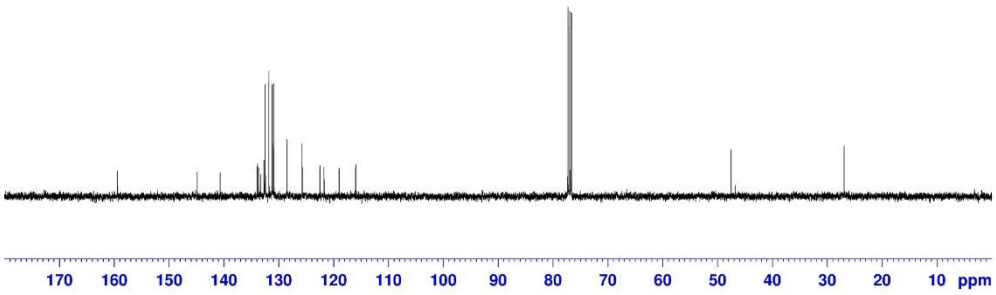


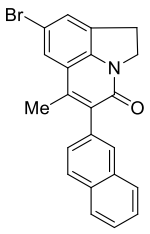
4l

2090-10, 1H NMR, CDCl3, AV400, 20140701

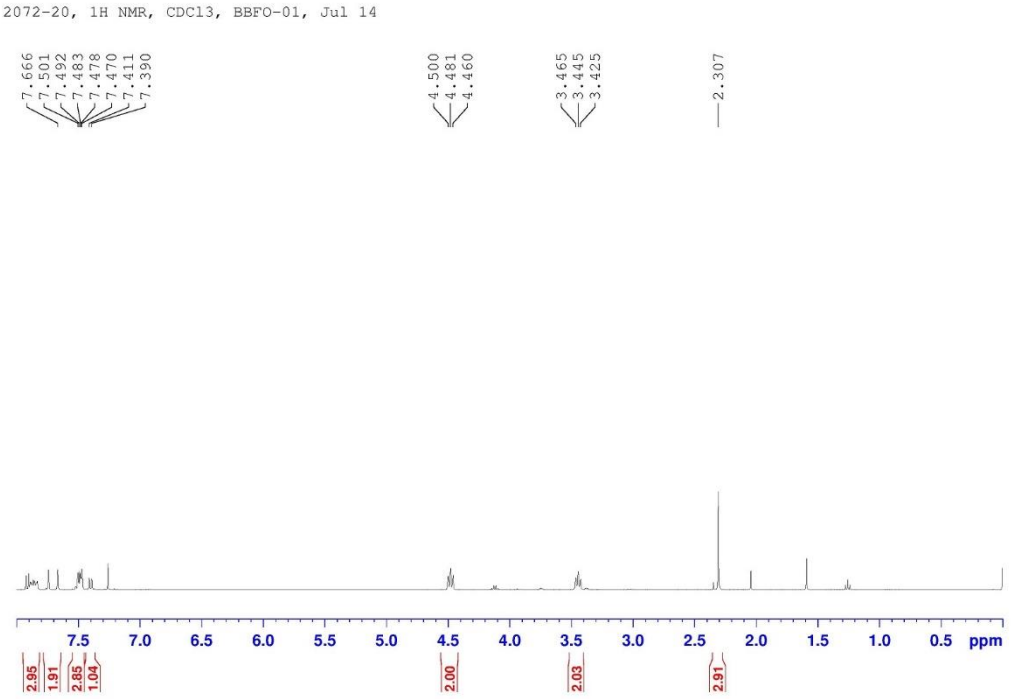


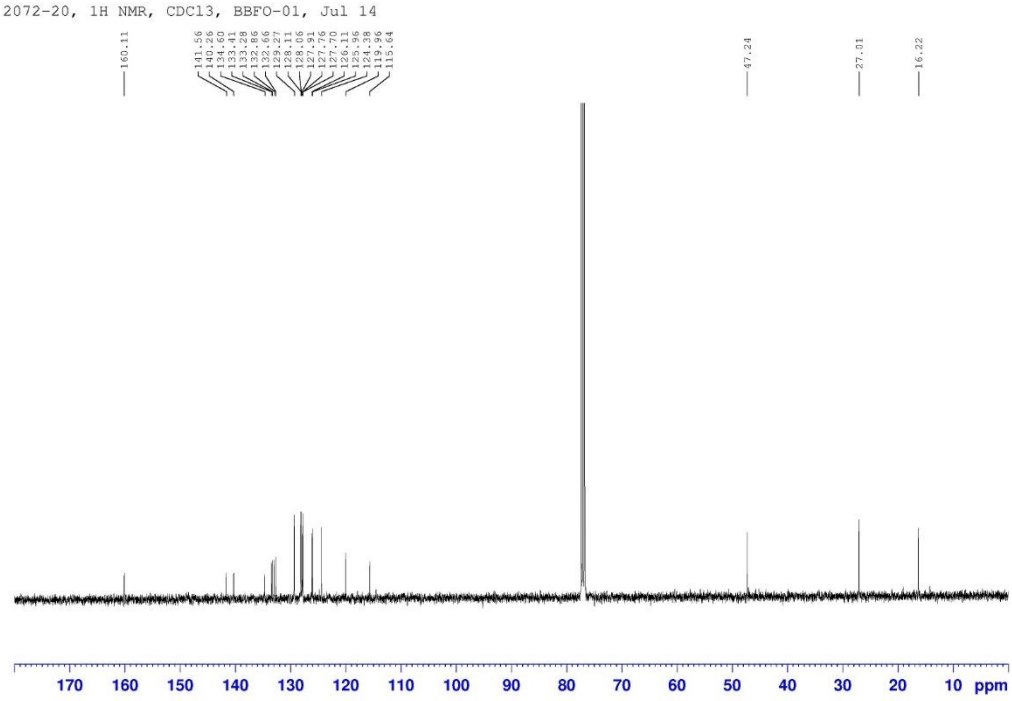
2090-10, 1H NMR, CDCl3, AV400, 20140701

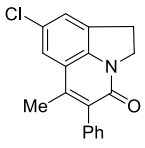



Br
N
Me
O

4m

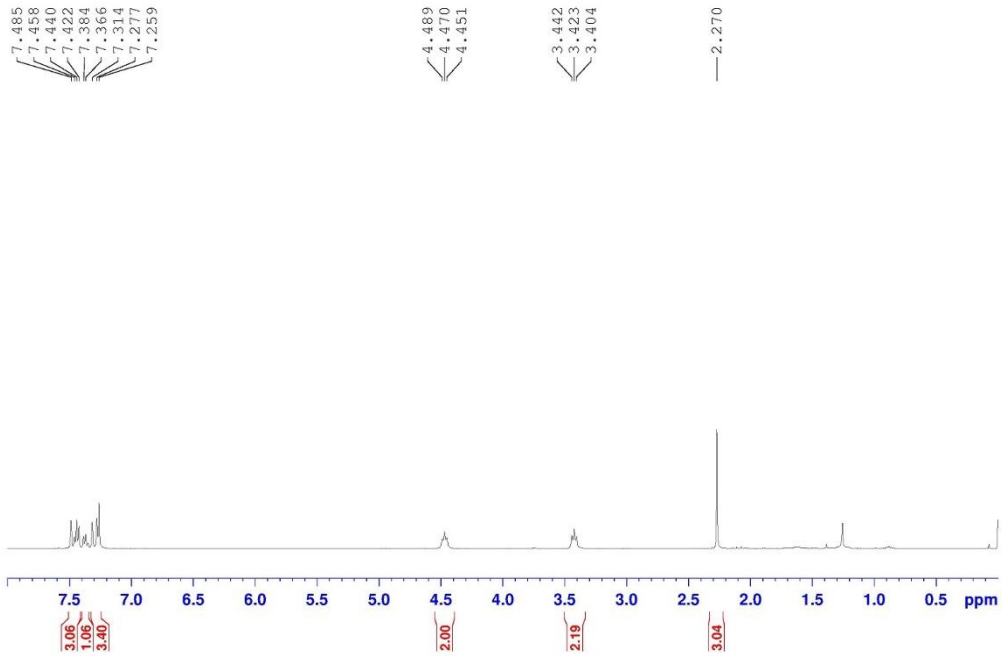

2072-20, 1H NMR, CDCl3, BBFO-01, Jul 14
7.666
7.501
7.492
7.483
7.478
7.470
7.411
7.390
4.500
4.481
4.460
3.465
3.445
3.425
2.307
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.95
1.91
2.85
1.04
2.00
2.03
2.91


2072-20, 1H NMR, CDCl3, BBFO-01, Jul 14
160.11
141.56
140.26
134.60
133.41
133.28
132.86
132.66
129.27
128.11
128.06
127.91
127.76
127.70
126.11
125.96
124.38
119.96
115.64
47.24
27.01
16.22
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

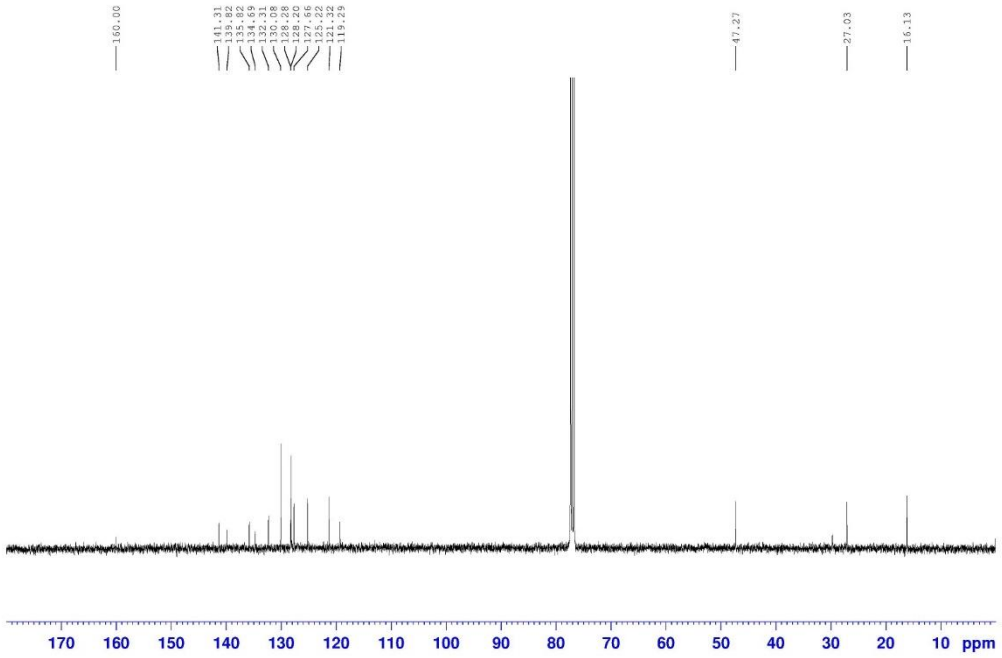


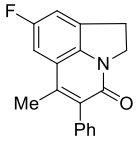
4n

2086-20, 1H NMR, CDCl3, BBFO-01, Jul 14

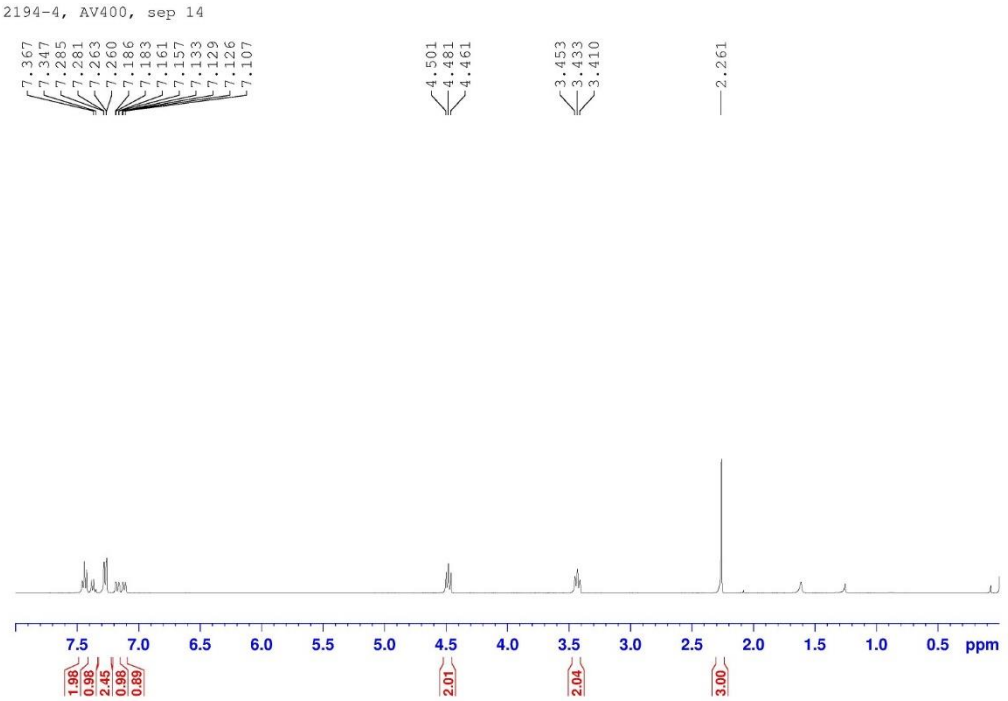


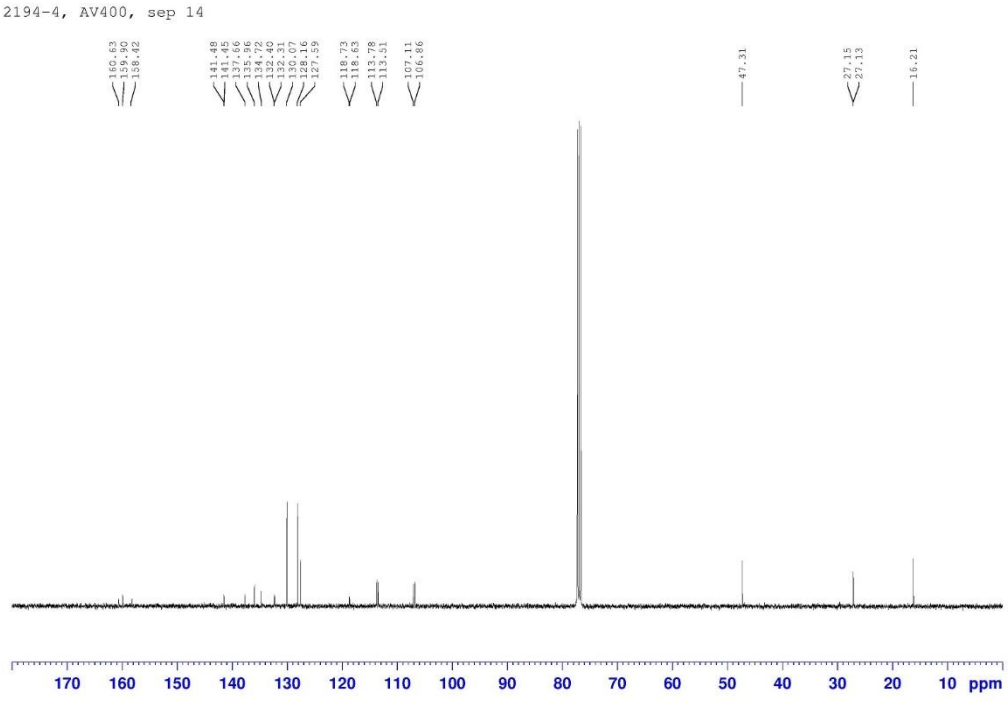
2086-20, AV400, Dec14



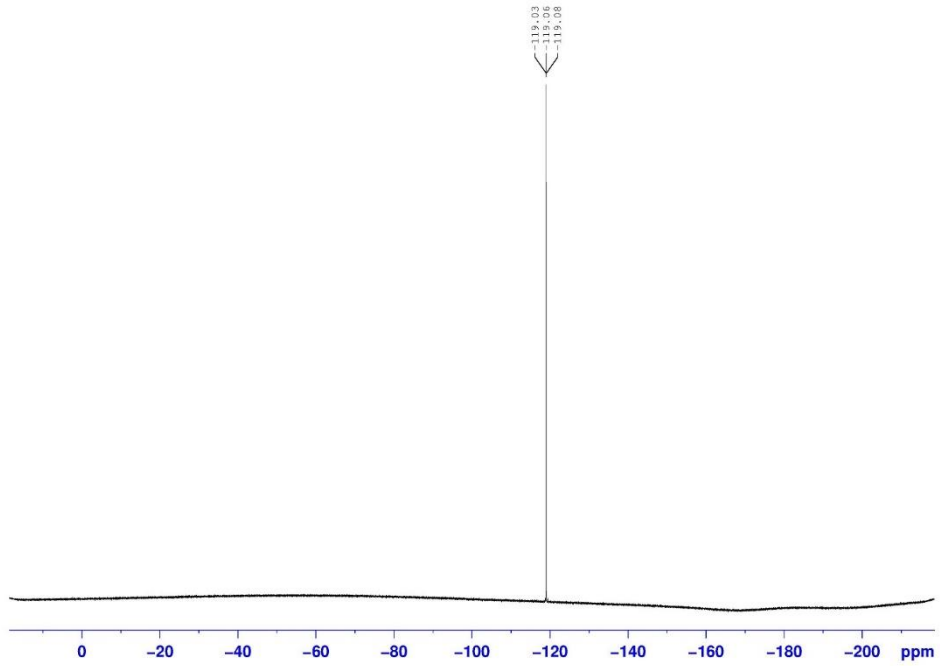

F
N
Me
O
Ph

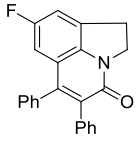
4o


2194-4, AV400, sep 14
7.367
7.347
7.285
7.281
7.263
7.260
7.186
7.183
7.161
7.157
7.133
7.129
7.126
7.107
4.501
4.481
4.461
3.453
3.433
3.410
2.261
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
ppm
1.98
0.98
2.45
0.98
0.89
2.01
2.04
3.00

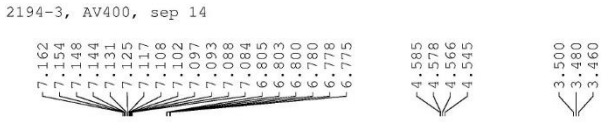

2194-4, AV400, sep 14
160.63
159.90
158.42
141.48
141.45
137.46
135.96
134.72
132.40
132.31
130.07
128.16
127.59
118.73
118.63
113.78
113.51
107.11
106.86
47.31
27.15
27.13
16.21
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

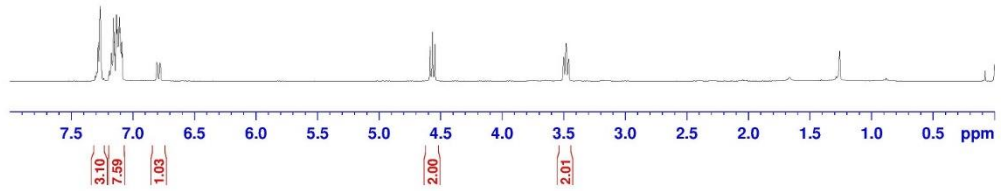
2194-4, BBFO1

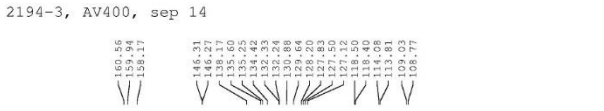


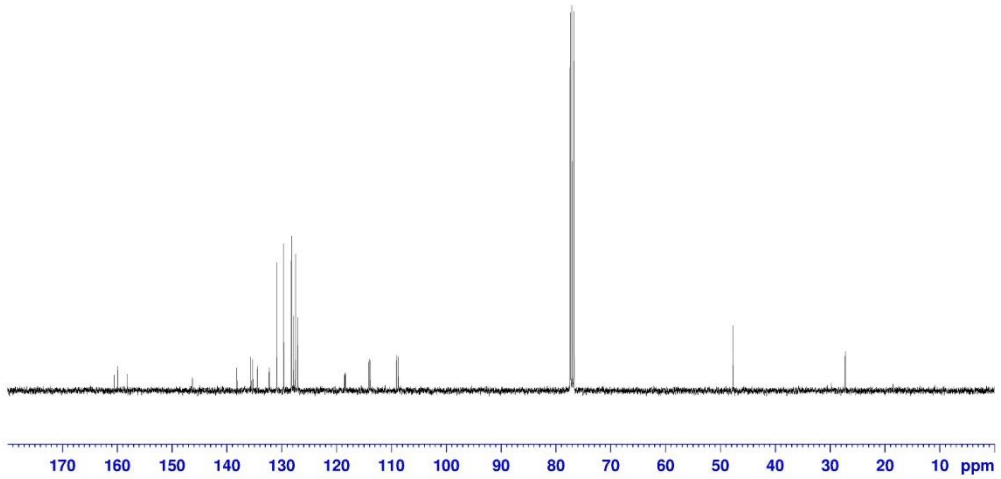

F
N
Ph
O
Ph

4p

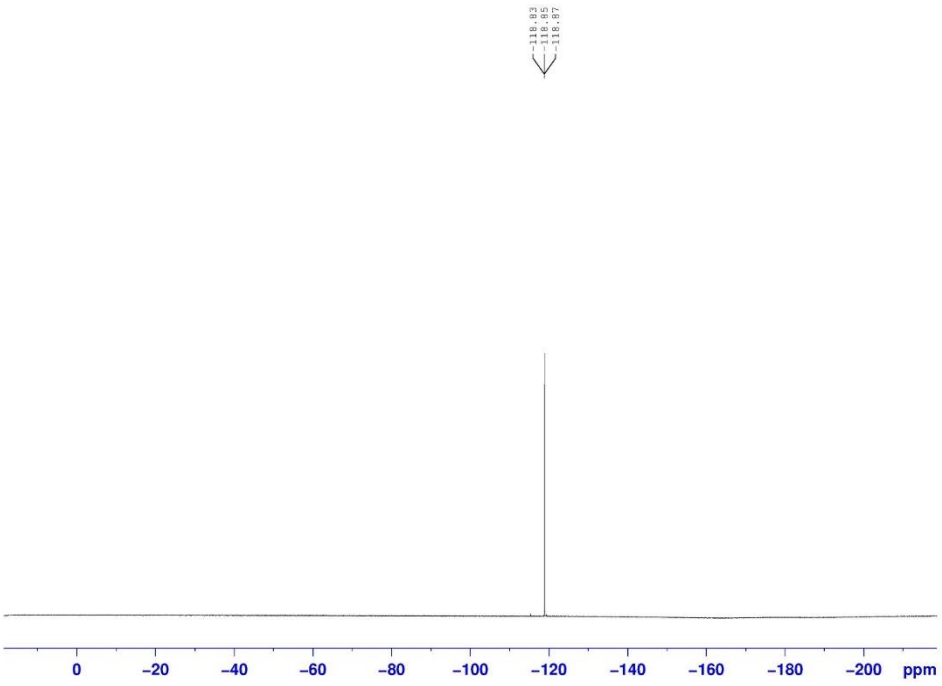

2194-3, AV400, sep 14
7.162
7.154
7.148
7.144
7.131
7.125
7.117
7.108
7.102
7.097
7.093
7.088
7.084
6.805
6.803
6.800
6.780
6.778
6.775
4.585
4.578
4.566
4.545
3.500
3.480
3.460

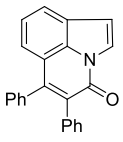

7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
3.10
7.59
1.03
2.00
2.01


2194-3, AV400, sep 14
160.56
154.44
150.17
146.31
146.27
138.17
135.60
133.55
133.42
132.33
132.24
130.88
129.64
128.00
127.83
127.50
127.12
118.50
118.40
114.08
113.81
109.03
108.77
47.64
27.21
27.20


170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

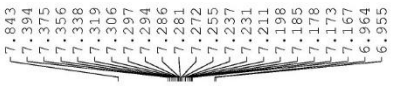
2194-3

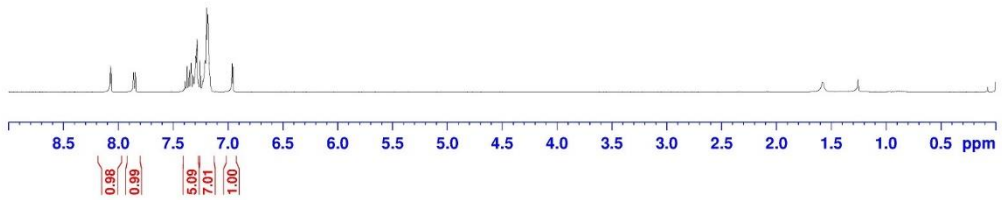




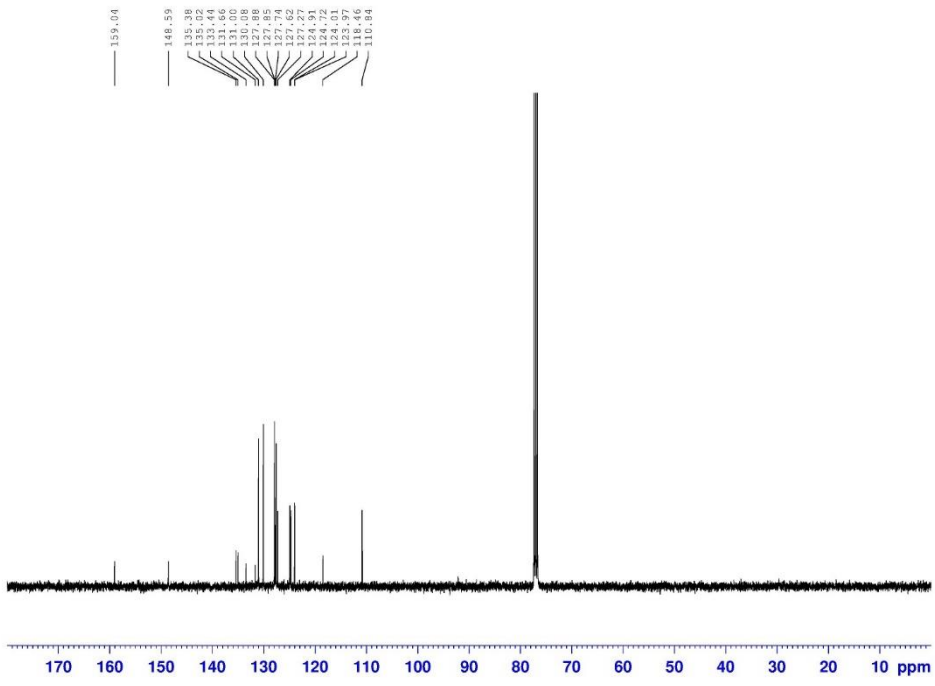
5

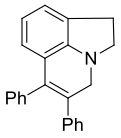
2254, AV 400MHz, Nov14



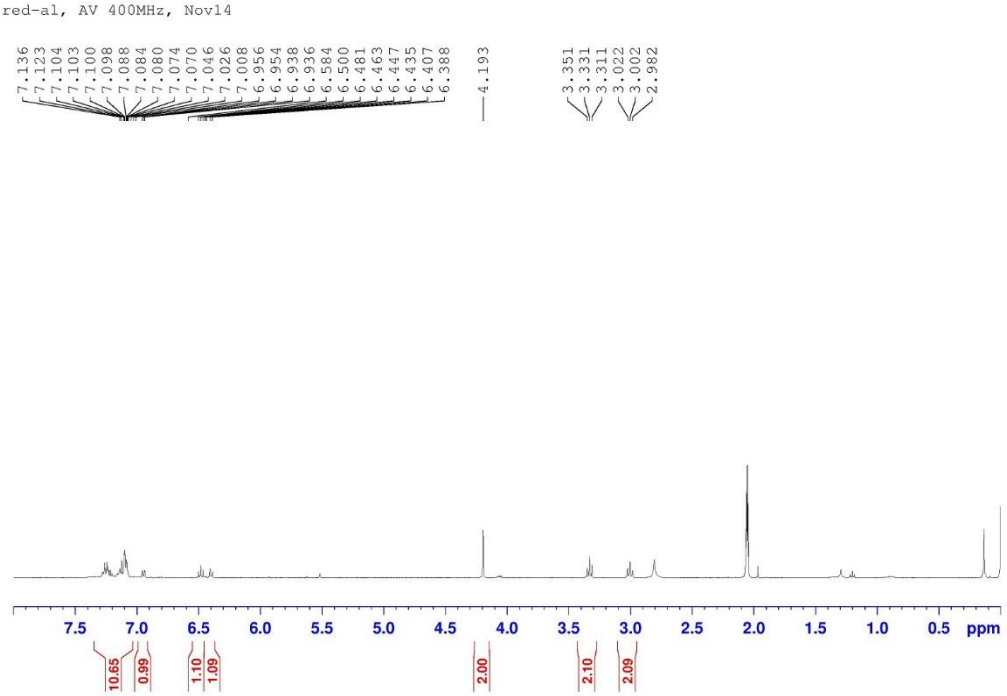


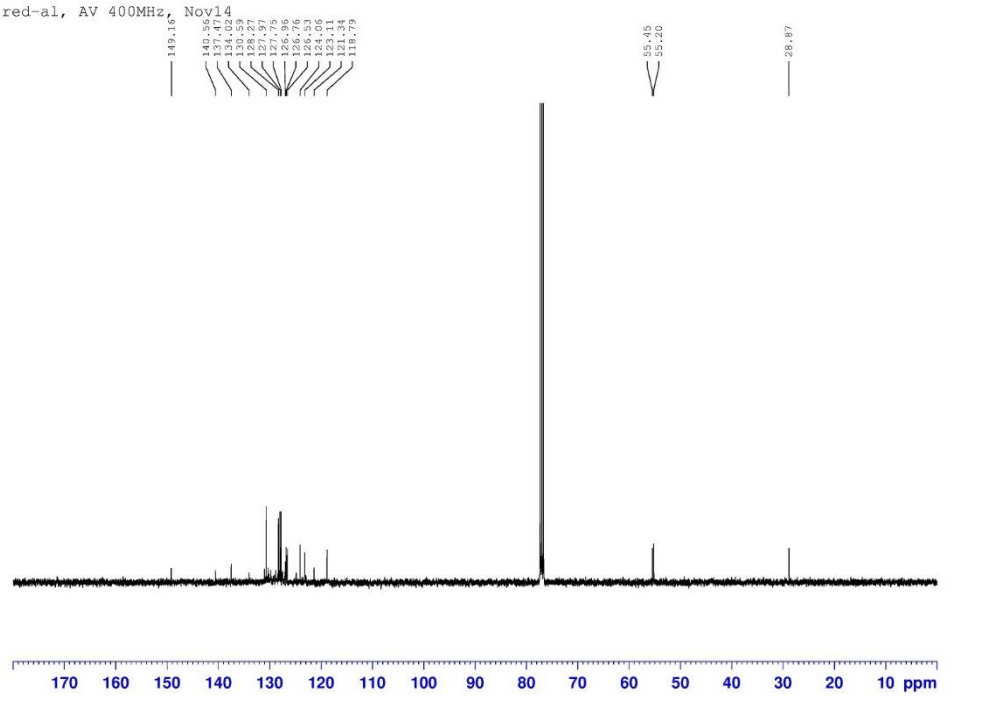
2254, AV 400MHz, Nov14

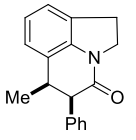



N
Ph
Ph

6

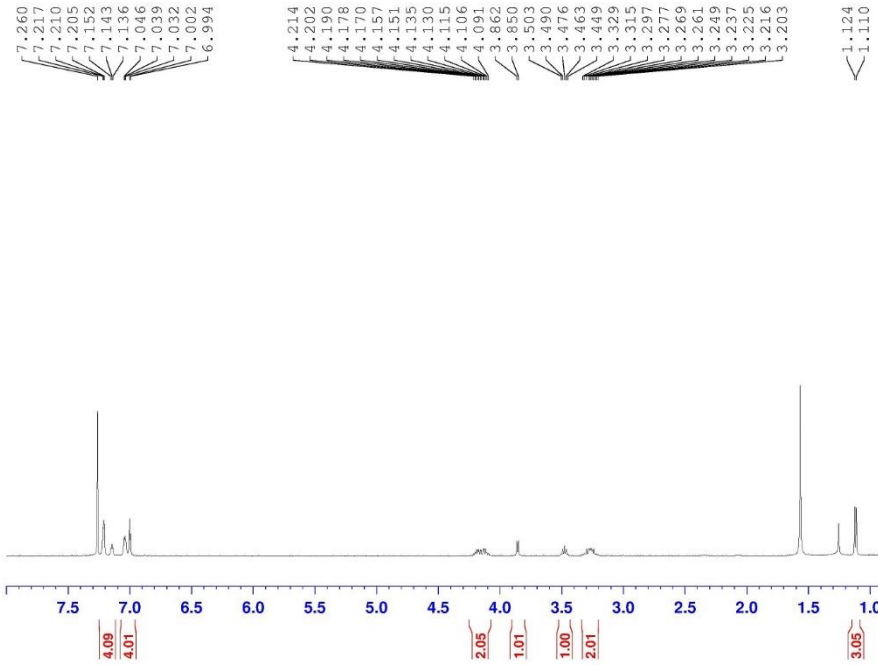

red-al, AV 400MHz, Nov14
7.136
7.123
7.104
7.103
7.100
7.098
7.088
7.084
7.080
7.074
7.070
7.046
7.026
7.008
6.956
6.954
6.938
6.936
6.584
6.500
6.481
6.463
6.447
6.435
6.407
6.388
4.193
3.351
3.331
3.311
3.022
3.002
2.982
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
10.65
0.99
1.10
1.09
2.00
2.10
2.09


red-al, AV 400MHz, Nov14
149.16
140.56
137.47
134.02
130.59
128.77
127.97
127.75
126.96
126.76
126.53
124.06
123.11
121.34
118.79
55.45
55.20
28.87
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

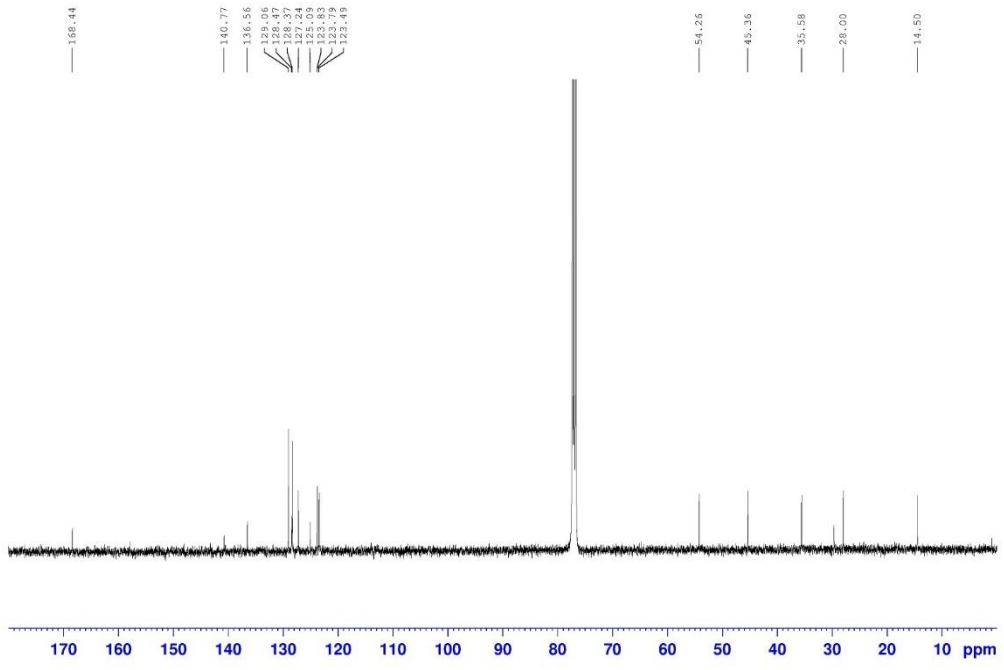


7

H2, AV500, Nov14



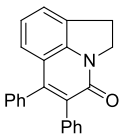
h2, AV 400MHz, Nov14

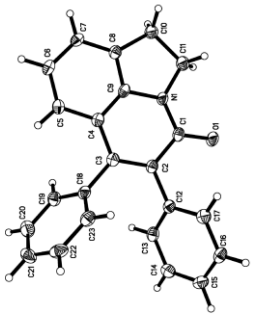


X-ray Data

X-Ray Structure for 3a

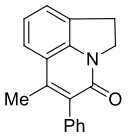
Cambridge Crystallographic Data Centre Deposition Number: 1042776

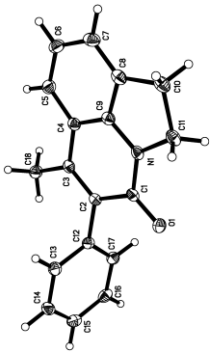




X-Ray Structure for 3e

Cambridge Crystallographic Data Centre Deposition Number: 1042775

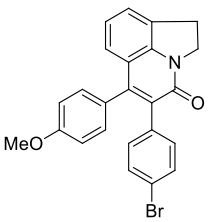


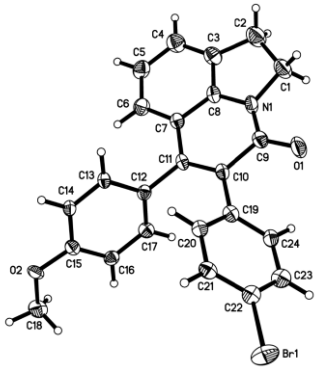


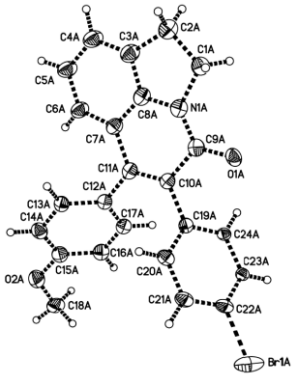
X-Ray Structure for 3m

Cambridge Crystallographic Data Centre Deposition Number: 1042777

The whole molecule is disordered about a non-crystallographic 2-fold axis and the ratio of site occupancy was freely refined to 0.798/0.202. Restraints and constraint (eadp c18 c18a, sadi o2 c14 o2 c16, rigu, simu 0.02, same Br1 > O2) were used in the refinement to restrain the two components having similar molecular geometries and atomic displacement parameters.







major minor