Supporting Information

$CsPbBr_3$ Perovskite Photocatalyst in Chemodivergent Functionalization of *N*-Methylalkanamides using CBr_4

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Content

General Information. All chemicals were obtained from commercial sources. Mainly, all the reactions were carried out under aerobic conditions unless otherwise noted. The reactions were monitored by TLC on aluminium sheets pre-coated with silica gel. Chromatographic purifications of the compounds were performed using silica gel (Mesh 100-200) and ethyl acetate and hexane as eluent.

EXPERIMENTAL SECTION

Characterization and Method. PXRD pattern was collected using Bruker Davinci D8 diffractometer (Cu-Ka radiation; λ=0.15418 nm). TEM images were captured by JEOL (JEM-2100) operating at an accelerating voltage of 200 kV. UV-VIS absorption experiment was carried out with JascoV-730 spectrophotometer. Fluorescence spectroscopy was documented using Edinburgh spectrofluorometer FS5 with SC-25 cuvette holder. Absolute quantum yield was measured using an integrating sphere (SC-30). PL decay measurement was carried out through TCSPC method using Edinburgh Instruments (Model OB-920), decorated with 405 nm laser as the excitation source. ^{1}H and ^{13}C spectra were recorded on Bruker 400 and 700 MHz (BRUKER® ULTRASHIELD) instruments at 25 ºC. The chemical shift value (δ, ppm) was reported to the residual chloroform (7.26 for ^{1}H and 77.16 ppm for ^{13}C). Mass spectra were recorded as ESI-TOF (HRMS). Infrared spectra were recorded on Thermo Scientific (NICOLET iS5) using KBr pellets and described in wavenumber (cm^{-1}). Cyclic voltametric data were investigated on the CorrTest Electrochemical Station (Model: CS310, S/N: 1711458) in dry and oxygen-free DCM: hexane (1:4) solution containing 0.1 M tetrabutylammonium hexafluorophosphate as a supporting electrolyte with a decoration of a glassy carbon electrode, a Ag/AgCl electrode and a platinum wire as the working electrode, reference electrode, and counter electrode, respectively using a scan rate 100 mV/s. Redox potential was referenced

against ferrocene/ferrocenium (Fc/Fc^+). Digital melting point apparatus was used to record the Melting Point of the compound in degree centigrade (°C) and are uncorrected.

Synthesis

$CsPbBr_3$ NCs was synthesized according to the literature procedure.[1] Pre-dried Cs_2CO_3 (195 mg, 0.6 mmol), Octadecene (9 mL), and Oleic acid (1.0 mL) were taken in a two-necked 50 mL round-bottom flask (RB). The reaction mixture was dried under vacuum at 120 °C for 30 min and then transferred to the N_2 atmosphere for 1 h, keeping the temperature same. In another three-necked 25 mL RB, PbO (44 mg, 0.3 mmol), dibromoisocyanauric acid (174 mg, 0.6 mmol) and octadecene (5 mL, pre-dried) were added respectively. The reaction mixture was kept under vacuum for 30 min at elevated temperature (~120 °C) followed by to the N_2 environment. After 10 min, 1.0 mL oleic acid and 1.0 mL oleylamine were injected to the reaction mixture and temperature of the reaction mixture was raised to ~200 °C. Then, cesium-oleate (~0.8 mL) solution (preheated at 100 °C) was swiftly injected into the reaction mixture. Subsequently, the reaction was quenched in an ice bath. After that, 3 mL of methylacetate was added to the mixture and centrifuged for 10 min at 6500 rpm. The supernatant was discarded, and the precipitation was dispersed in hexane and kept in the refrigerator for 30 min. The suspension was again centrifuged for 10 min at 6500 rpm. Finally, the supernatant containing the NCs and precipitation were separated and both were stored for future experiments.

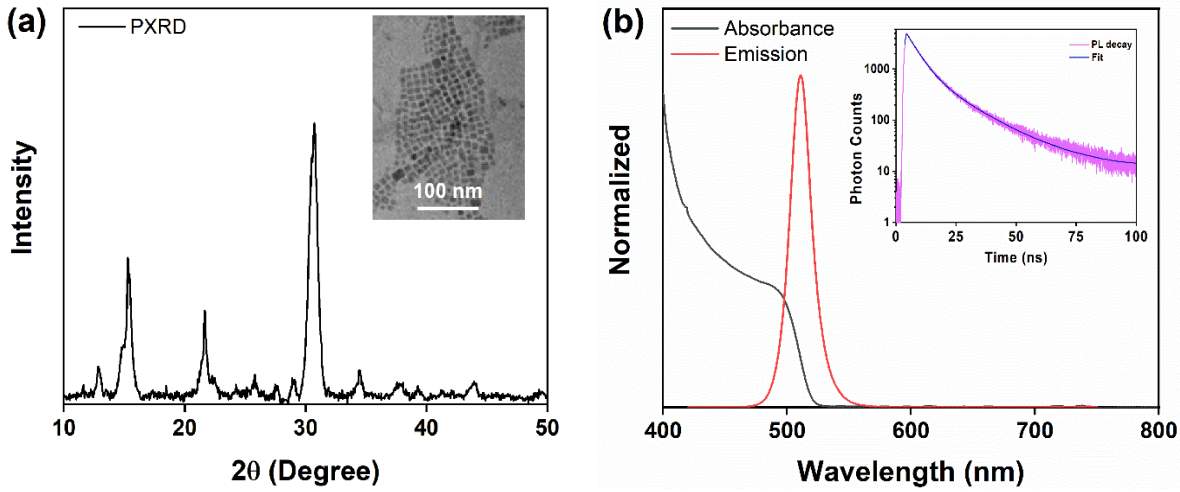


Figure S1. (a) PXRD of $CsPbBr_3$ (Inset TEM), (b) Absorbance and emission spectra of $CsPbBr_3$ (inset PL decay).

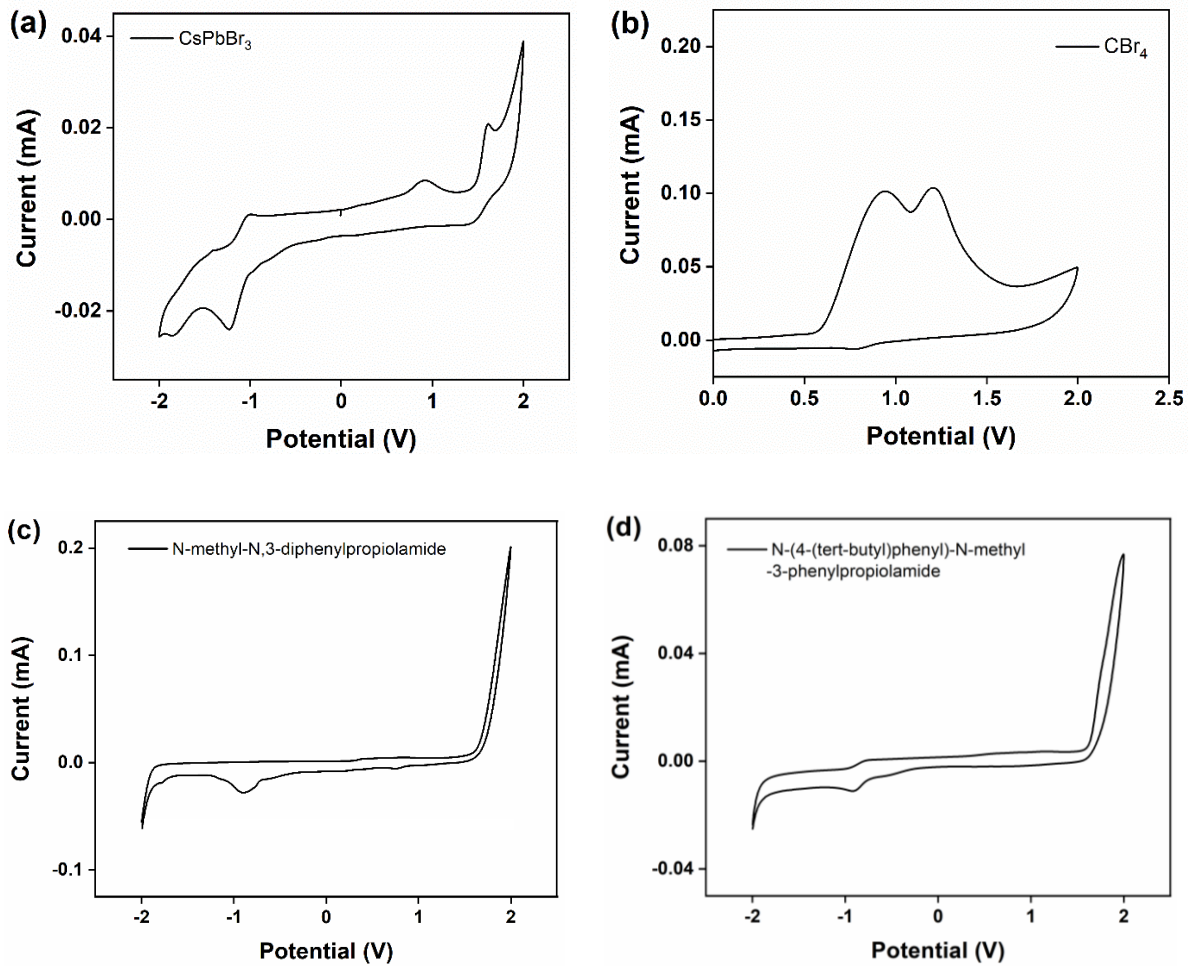
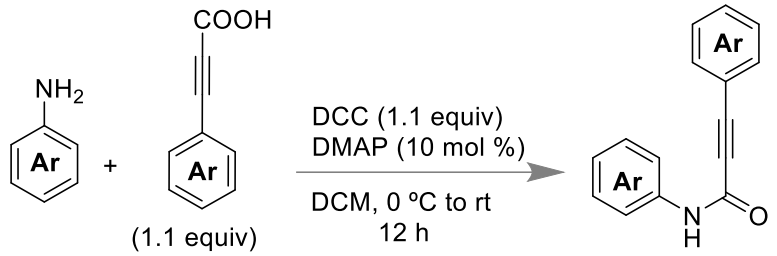
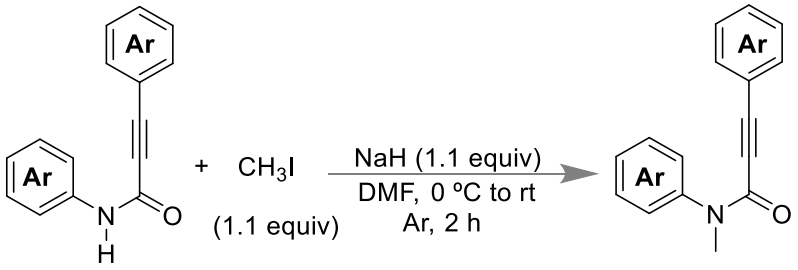


Figure S2. CV diagram of (a) DBIA-$CsPbBr_3$ (b) CBr_4 (c) *N*-methyl-*N*,3-diphenylpropiolamide, (d) N-(4-(*tert*-butyl)phenyl)-N-methyl-3-phenylpropiolamide.



Scheme S1. Synthesis of *N*-methyl-*N*,3-diphenylpropiolamide.

Synthesis of *N*,3-diphenylpropiolamide derivatives. In a 50 mL round-bottomed flask, a solution of 3-phenylpropiolic acid (1.1 mmol, 1.1 equiv, 161 mg) was made by the addition of 15 mL DCM (CH_2Cl_2); followed by the solution was allowed to stir at 0 °C. After that, a mixture of 4-dimethylaminopyridine (0.1 mmol, 0.1 equiv, 131 mg) and dicyclohexylcarbodiimide (1.1 mmol, 1.1 equiv, 227 mg) in 7 mL DCM (CH_2Cl_2) was slowly added to the 3-phenylpropiolic acid solution. Again, a solution of aniline (1.0 mmol, 1.0 equiv, 93 mg) in 8 mL DCM (CH_2Cl_2) was then added to dropwise. Afterward, the reaction mixture was stirred at room temperature for 12 h. Then, the crude mixture was diluted in DCM (CH_2Cl_2) and extracted with brine solution and dried over in Na_2SO_4, and concentrated under rotary-evaporator. Finally, the crude residue was purified by column chromatography to afford the desired *N*,3-diphenylpropiolamide (186 mg, 83%) derivatives.



Scheme S2. Synthesis of *N*,3-diphenylpropiolamide.

Synthesis of *N*-methyl-*N*,3-diphenylpropiolamide. In a 25 mL round-bottomed flask, a solution of *N*,3-diphenylpropiolamide (1.0 mmol, 1.0 equiv, 221 mg) was made by the addition of 3 mL dry DMF, followed by the solution was allowed to stir at 0 °C. After that, NaH (1.2 mmol, 1.2 equiv, 28 mg) was added under argon atmosphere. After that CH_3I (1.2 mmol, 1.2 equiv, 76 µL) was added to reaction mixture. The reaction was proceeded for 2 hours under inert atmosphere. Next, the reaction mixture was extracted with EtOAc and washed with brine solution after 2 h. It was then dried over in Na_2SO_4 and concentrated using a rotary evaporator. Finally, column chromatography was used to separate the crude residue into the required *N*-methyl-*N*,3-diphenylpropiolamide (210 mg, 90%) derivatives.

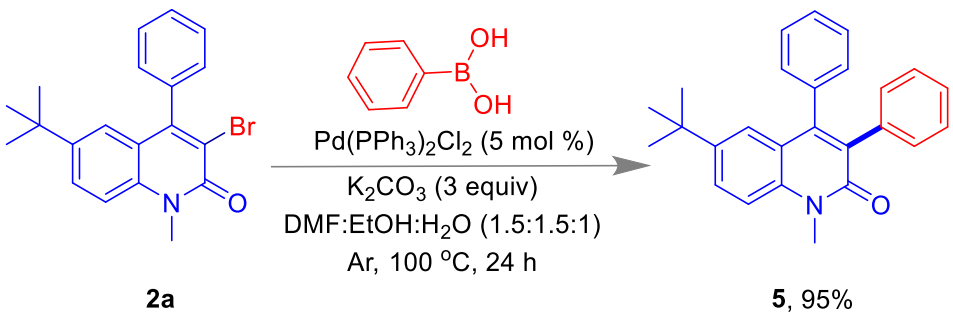
Scheme S3. Synthesis of 3-bromo-6-(*tert*-butyl)-1-methyl-4-phenylquinolin-2(1H)-one.

Preparation of 3-bromo-6-(*tert*-butyl)-1-methyl-4-phenylquinolin-2(1H)-one. In an oven dried quartz tube N-(4-(*tert*-butyl)phenyl)-N-methyl-3-phenylpropiolamide **1a** (1.0 mmol, 1.0 equiv, 279 mg), carbon tetrabromide (0.5 mmol, 0.5 equiv, 166 mg), and $CsPbBr_3$ (3 mol %, 17.39 mg) were dissolved in dry acetonitrile solvent. After that, the reaction mixture was irradiated by Blue LEDs light for 24 h in the presence of an inert atmosphere. The leftover solvent was evaporated when the reaction was finished, and the raw mixture dissolved in DCM (CH_2Cl_2) and extracted with brine solution. The resulting organic solution was concentrated after being dried over anhydrous sodium sulphate, yielding a crude combination that was then further refined using 100-200 mesh silica-gel column chromatography with ethyl acetate and

hexane as the eluent to produce the final, pure product 3-bromo-6-(*tert*-butyl)-1-methyl-4-phenylquinolin-2(1H)-one (334 mg, 90%).

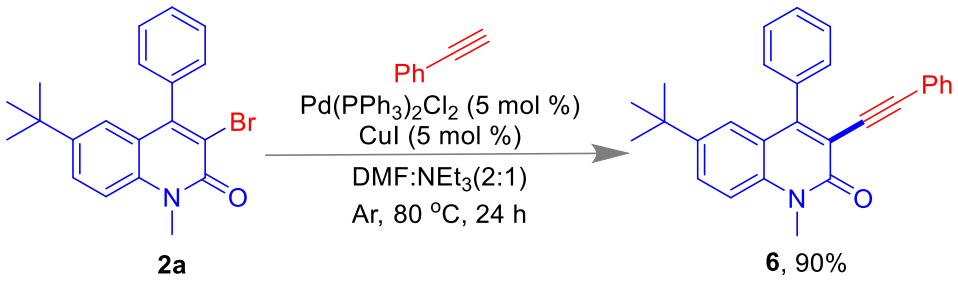
Scheme S4. Synthesis of 3,8-dibromo-1-methyl-4-phenyl-1-azaspiro[4.5]deca-3,6,9-trien-2-one.

Preparation of 3,8-dibromo-1-methyl-4-phenyl-1-azaspiro[4.5]deca-3,6,9-trien-2-one. In an oven dried quartz tube *N*-methyl-*N*,3-diphenylpropiolamide **3a** (1.0 mmol, 1.0 equiv, 235 mg), carbon tetrabromide (1.1 mmol, 1.1 equiv, 365.20 mg), and $CsPbBr_3$ (3 mol %, 17.39 mg) were dissolved in dry acetonitrile solvent. After that, the reaction mixture was irradiated by Blue LEDs light for 24 h in the presence of an inert atmosphere. The solvent was evaporated when the reaction was completed, and the residue was dissolved in DCM (CH_2Cl_2) and extracted with brine solution. The resulting organic solution was concentrated after being dried over anhydrous sodium sulphate, yielding a crude combination that was then further refined using silica-gel column chromatography with ethyl acetate and hexane as the eluent to produce the final, pure product 3,8-dibromo-1-methyl-4-phenyl-1-azaspiro[4.5]deca-3,6,9-trien-2-one (298 mg, 75%).



Scheme S5. Synthesis of 6-(*tert*-butyl)-1-methyl-3,4-diphenylquinolin-2(1*H*)-one.

Preparation of 6-(*tert*-butyl)-1-methyl-3,4-diphenylquinolin-2(1H)-one. To an oven dried Sealed tube holding a magnetic bar was charged with 3-bromo-6-(*tert*-butyl)-1-methyl-4-phenylquinolin-2(1H)-one **2a** (0.081 mmol, 30 mg), phenylboronic acid (0.162 mmol, 20 mg), K_2CO_3 (0.121 mmol, 17 mg), and $Pd(PPh_3)_4$ (0.081 mmol, 10 mg) and DMF: EtOH : NEt_3 (1.5:1.5:1) under inert atmosphere and the reaction mixture was placed into a preheated oil bath at 100 °C for 24 h. After completion the reaction mixture was cooled to room temperature. Then, the crude mixture was diluted in ethyl acetate and extracted with brine solution. The resulting organic solution was dried over anhydrous sodium sulphate and concentrated to obtain a crude mixture which was further purified by silica-gel column chromatography using ethyl acetate and hexane as the eluent to afford the pure product.



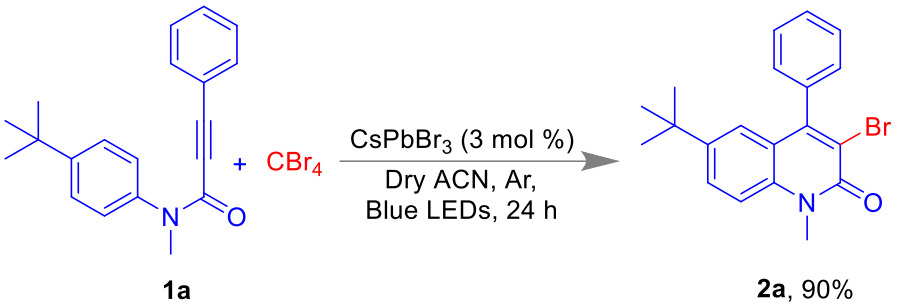
2a **6**, 90%

Scheme S6. Synthesis of 6-(*tert*-butyl)-1-methyl-4-phenyl-3-(phenylethynyl)quinolin-2(1H)-one.

Preparation of 6-(tert-butyl)-1-methyl-4-phenyl-3-(phenylethynyl)quinolin-2(1H)-one. To an oven dried Sealed tube holding a magnetic bar was charged with 3-bromo-6-(*tert*-butyl)-1-methyl-4-phenylquinolin-2(1H)-one **2a** (0.1081 mmol, 40 mg), ethynylbenzene (0.162 mmol, 17 mg), $Pd(PPh_3)_4Cl_2$ (0.005 mmol, 4 mg), CuI (0.021 mmol, 4 mg) and DMF:NEt_3 (2:1) under inert atmosphere and the reaction mixture was placed into a preheated oil bath at 80 °C for 24 h. After completion the reaction mixture was cooled to room temperature. Then, the crude mixture was diluted in ethyl acetate and extracted with brine solution. The resulting organic solution was dried over anhydrous sodium sulphate and concentrated to obtain a crude

mixture which was further purified by silica-gel column chromatography using ethyl acetate and hexane as the eluent to afford the pure product.

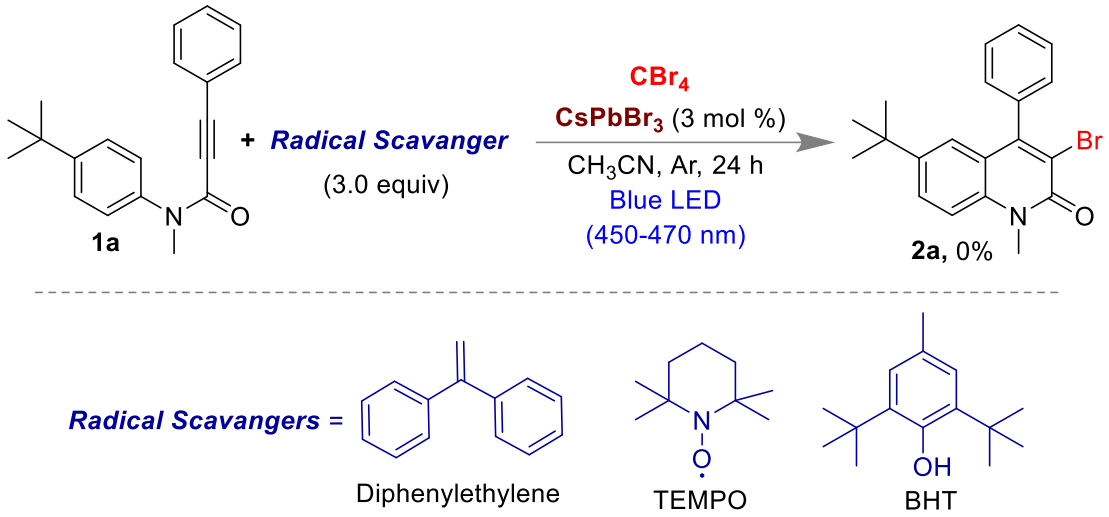
Table S1. Reaction Condition Optimization.[a]



Entry	Catalyst	Br-Source	Solvent	Light Source	Yield(%) [a]
1	_	CBr_4	Dry MeCN	Blue LED	0
2	C_ZIPN	CBr_4	Dry MeCN	Blue LED	30
3	Mes-Acr-$MeClO_4$	CBr_4	Dry MeCN	Blue LED	19
4	$Ru(bpy)_3(PF_6)_2$	CBr_4	Dry MeCN	Blue LED	50
5	Eosin Y	CBr_4	Dry MeCN	Blue LED	0
6	**$CsPbBr_3$**	**CBr_4**	Dry MeCN	**Blue LED**	**90**
7	$CsPbBr_3$	CBr_4	Dry MeCN	Blue LED	49[b]
8	$CsPbBr_3$	CBr_4	MeCN	Blue LED	40[c]
9	$CsPbBr_3$	CBr_4	THF	Blue LED	0
10	$CsPbBr_3$	CBr_4	TFE	Blue LED	0
11	$CsPbBr_3$	CBr_4	DMSO	Blue LED	0
12	$CsPbBr_3$	CBr_4	DMF	Blue LED	0
13	$CsPbBr_3$	CBr_4	Toluene	Blue LED	0
14	$CsPbBr_3$	CBr_4	CCl_4	Blue LED	0
15	$CsPbBr_3$	CBr_3F	Dry MeCN	Blue LED	60
16	$CsPbBr_3$	CH_2Br_2	Dry MeCN	Blue LED	70
17	$CsPbBr_3$	CBr_4	Dry MeCN	Blue LED	0[d]

[a]Reaction Conditions: **1a** (0.215 mmol, 1 equiv), **CBr_4** (0.236 mmol, 0.5 equiv), and $CsPbBr_3$ (0.005 mmol 3 mol %), in dry MeCN under Ar atmosphere for 24 h using Blue LEDs, [b]in dry MeCN under Ar atmosphere for 12 h instead of 24 h using Blue LEDs, [c]in HPLC grade MeCN, [d] under O_2 atmosphere.

Radical trapping experiment with Diphenylethylene/ TEMPO/ BHT. In an oven-dried quartz tube N-(4-(*tert*-butyl)phenyl)-N-methyl-3-phenylpropiolamide **1a** (0.206 mmol, 60 mg), Carbon tetrabromide (CBr_4) (0.236 mmol, 78 mg), and $CsPbBr_3$ (3 mol %, 3 mg) and Diphenylethylene (0.401 mmol, 90 mg) were dissolved in dry acetonitrile (MeCN) solvent. Following, the reaction tube was degassed for 15 min by argon gas. After that, the reaction mixture was irradiated by Blue LEDs light for 24 h in the presence of an argon balloon. The reaction was monitored by TLC. After the reaction time, no desired product was found. The same experiment was carried out using TEMPO (0.412 mmol, 64 mg) and BHT (0.412 mmol, 74 mg). However no successful outcome was achieved.



Scheme S7. Experiemnts using radical scavengers under standard condition.

Light ON-OFF-ON experiment

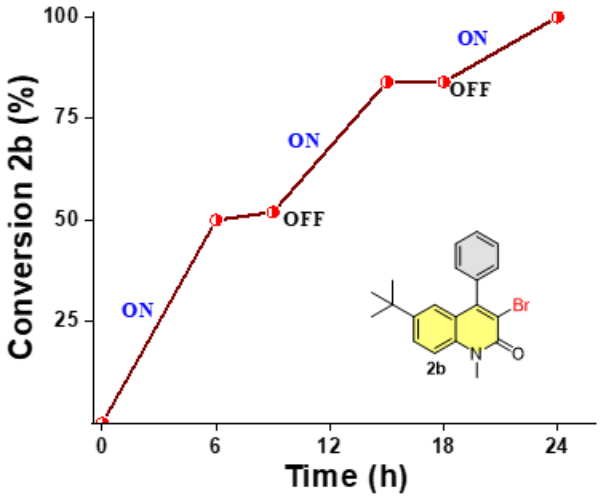


Figure S3. Conversion of **2b** *vs.* time in the presence and absence of light.

In an oven-dried quartz tube N-(4-(*tert*-butyl)phenyl)-N-methyl-3-phenylpropiolamide **1a** (0.206 mmol, 60 mg), Carbon tetrabromide (CBr_4) (0.236 mmol, 78 mg), and $CsPbBr_3$ (3 mol %, 3 mg) were dissolved in dry acetonitrile (MeCN) solvent. After that, the reaction mixture was irradiated by Blue LEDs light for 24 h in the presence of an argon ballon. Successive progress of the reaction was monitored every 6 h and 3 h in the presence of light and absence of light by ^{1}H NMR experiment.

EPR Experiments. EPR spectra was recorded at 298 K using EPR spectrometer derived at 9.4335 GHz. Typical spectrometer parameters are shown as follows, scan range: 100 G; center field set: 3480.00 G; time constant: 0.16 ms; scan time: 122.88 s; modulation amplitude: 20.0 G; modulation frequency: 100 kHz; receiver gain: 2.00×10^2 ; microwave power: $7.14e^{-001}$ mW; g = 2.00686.

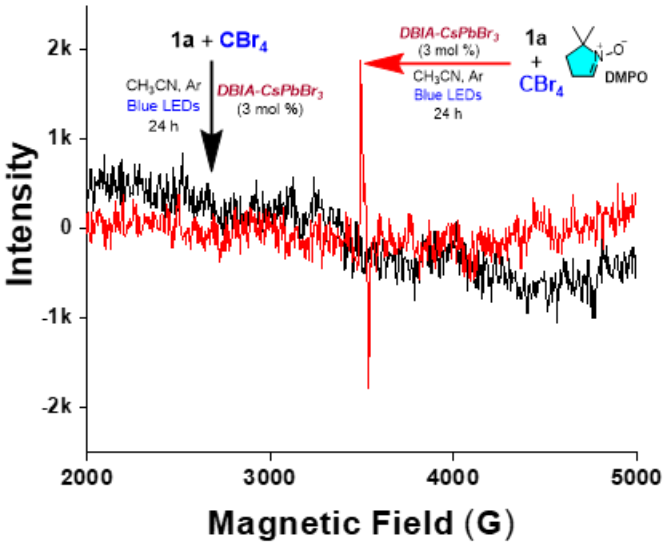


Figure S4. a) EPR experiment under the standard condition.

Spin-trapping experiment in the presence DMPO. A mixture of N-(4-(*tert*-butyl)phenyl)-N-methyl-3-phenylpropiolamide **1a** (0.206 mmol, 60 mg), Carbon tetrabromide (CBr_4) (0.236 mmol, 78 mg), and $CsPbBr_3$ (3 mol %, 3 mg) were dissolved in dry acetonitrile (MeCN) and after degassing the rection mixture for 15 min, the reaction mixture was irradiated by Blue LEDs light for 4 h in the presence of an argon balloon. Afterward, 20 µL 5,5-dimethyl-1-pyrroline-N-oxide (DMPO) solution was quickly poured into EPR tube and 200 µL reaction mixture was appended to analyse EPR. A signal appeared, indicating the presence of an unpaired electron in the reaction pathway.

Photoluminescence Quenching Study. Photoluminescence quenching study of $CsPbBr_3$ was conducted using CBr_4 as quencher. Through Stern-Volmer kinetics, rate of quenching (k_q) was determined using the equation $I_0/I = 1 + k_q\tau$ [*quencher*], where I_0 is the initial PL intensity without the quencher, I is the intensity after addition of the quencher, and τ is the lifetime of the $CsPbBr_3$. Probe sample was prepared by suspending $CsPbBr_3$ NCs in MeCN of

concentration 0.5 mg mL^{-1}. Then 20 μL of the concentrate solution was diluted to make a total volume 2 mL. Quencher (CBr_4) of concentration of 1 mM was added to the probe suspension in an incremental way of 2 μL maintaining the total volume of 2 mL.

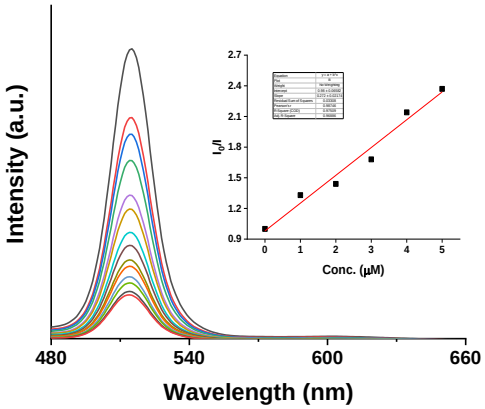


Figure S5. Fluorescence spectra of DBIA-$CsPbBr_3$ upon gradual addition of CBr_4.

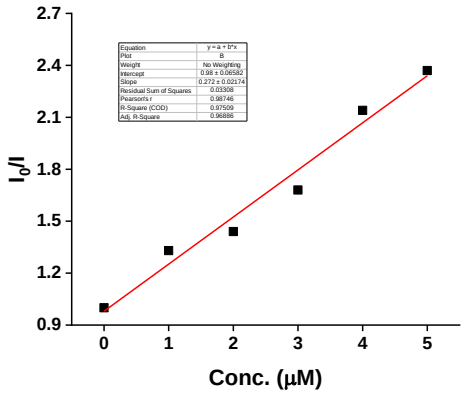


Figure S6. Stern-Volmer plot for CBr_4.

Crystal measurement. Crystals of compound **2** and **4d** were isolated after slow evaporation of $CHCl_3$ and water mixture (1 : 0.5). The crystals data were collected with Bruker SMART D8 goniometer equipped with an APEX CCD detector and with an INCOATEC micro source (Cu-Kα radiation, λ = 0.71073 Å). SAINT+[2] and SADABS[3] were used to integrate the intensities and to correct the absorption respectively The structure was resolved by direct

methods and refined on F^2 with SHELXL-97.[4] ORTEP drawing of the compound **2d** and **4d** show ellipsoid contour at the 50% probability level.

Compound 2d (CCDC 2252573)

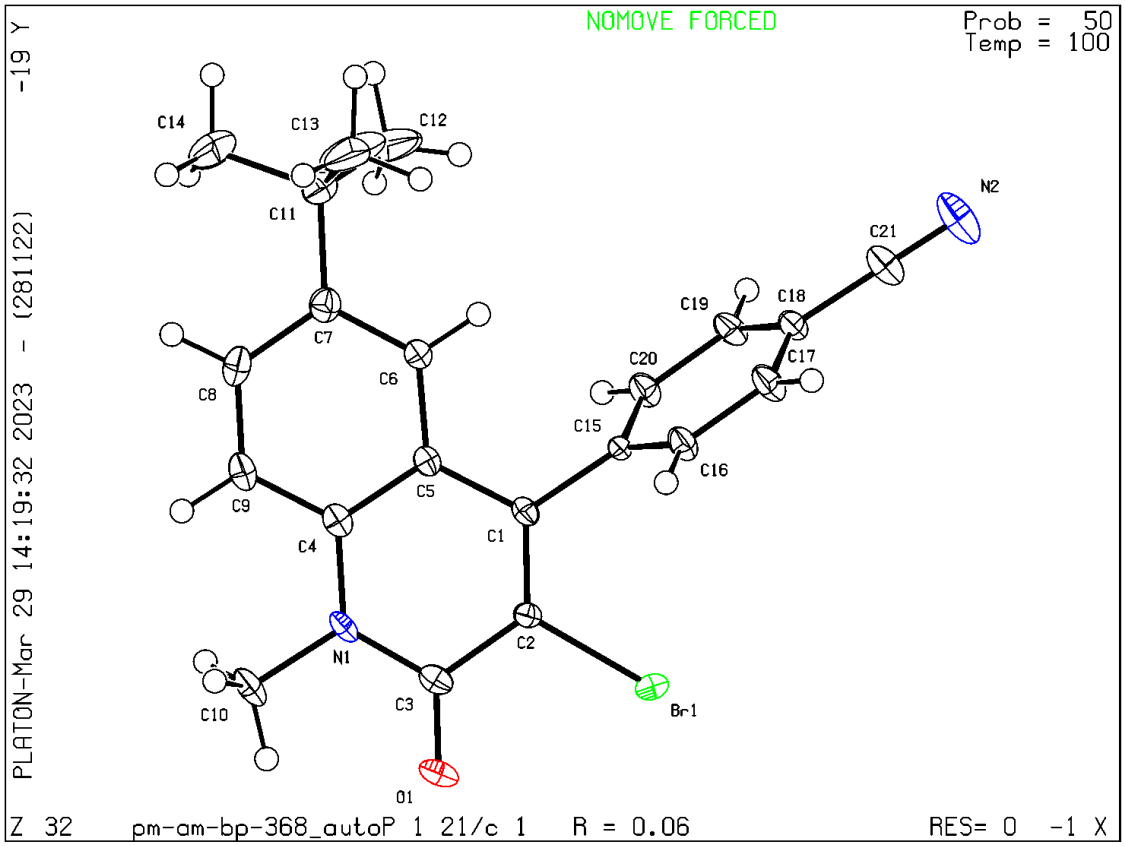


Figure S7. Crystal structure of **2d** (CCDC 2252573). Ellipsoids are drawn at the 50% probability level.

Crystallographic Data for (2d)

Empirical formula	$C_{21}H_{19}BrN_2O$
Formula weight	395.29
Temperature/K	100.00(10)

Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	9.8195(5)
b/Å	31.2383(15)
c/Å	6.9900(4)
α/°	90
β/°	102.685(5)
γ/°	90
Volume/Å3	2091.81(19)
Z	4
ρcalcg/cm^3	1.255
μ/mm^{-1}	1.974
F(000)	808.0
Crystal size/mm^3	0.2 × 0.2 × 0.1
Radiation	Mo Kα (λ = 0.71073)
Reflections collected	24135
Independent reflections	5136 [R_{int} = 0.0704, R_{sigma} = 0.0504]
Goodness-of-fit on F2	1.051
Final R indexes [I>=2σ (I)]	R_1 = 0.0596, wR_2 = 0.1724
Final R indexes [all data]	R_1 = 0.0722, wR_2 = 0.1791
Largest diff. peak/hole / e $Å^{-3}$	2.98/-0.80

Compound 4d (CCDC 2252575)

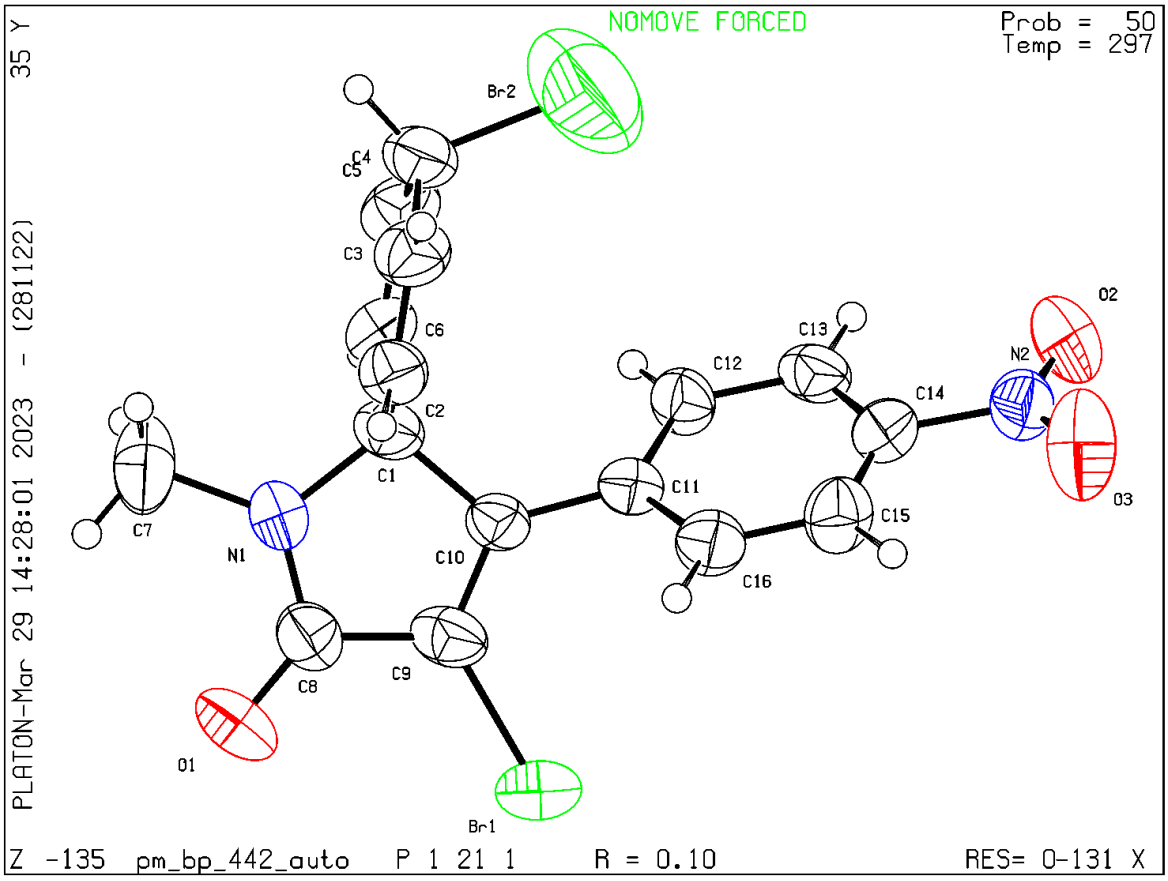


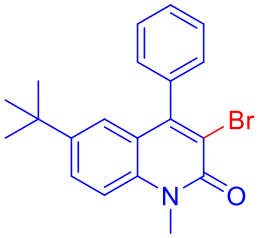
Figure S8. Crystal structure of **4d** (CCDC 2252575). Ellipsoids are drawn at the 50% probability level.

Crystallographic Data for (4d)

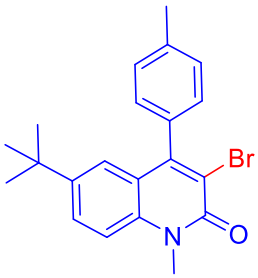
Empirical formula	$C_{16}H_{12}Br_2N_2O_3$
Formula weight	440.10
Temperature/K	297.15
Crystal system	monoclinic

Space group	$P2_1$
a/Å	7.8296(2)
b/Å	7.2484(2)
c/Å	14.2248(3)
α/°	90
β/°	91.931(2)
γ/°	90
Volume/Å3	806.83(3)
Z	2
ρcalcg/cm^3	1.812
μ/mm^{-1}	6.520
F(000)	432.0
Crystal size/mm^3	0.2 × 0.2 × 0.1
Radiation	CuKα (λ = 1.54184)
Reflections collected	9001
Independent reflections	2984 [R_{int} = 0.0394, R_{sigma} = 0.0301]
Goodness-of-fit on F2	1.327
Final R indexes [I>=2σ (I)]	R_1 = 0.0988, wR_2 = 0.2809
Final R indexes [all data]	R_1 = 0.1039, wR_2 = 0.2899
Largest diff. peak/hole / e $Å^{-3}$	1.06/-1.85

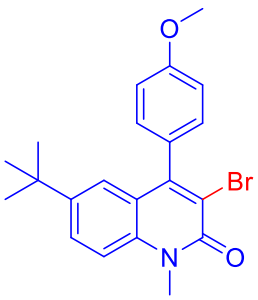
3-Bromo-6-(*tert*-butyl)-1-methyl-4-phenylquinolin-2(1H)-one (2a).[5] R_f = 0.4 (20% ethyl acetate in hexane); white solid mp 170-172 ºC; yield 90% (74 mg); ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.65 (dd, *J* = 8.9, 2.2 Hz, 1H), 7.56-7.50 (m, 3H), 7.37 (d, *J* = 8.9 Hz, 1H), 7.29-7.27 (m, 2H), 7.12 (d, *J* = 2.2 Hz, 1H), 3.87 (s, 3H), 1.19 (s, 9H); ^{13}C{^{1}H} NMR (176 MHz, $CDCl_3$) δ 158.5, 150.8, 145.5, 137.6, 136.9, 128.7×3, 124.6×2, 121.1, 119.0, 114.2, 34.4, 31.3, 31.2.



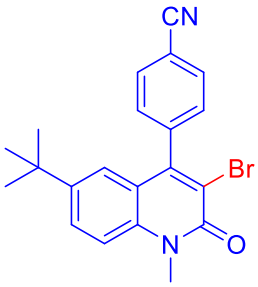
3-Bromo-6-(*tert*-butyl)-1-methyl-4-(p-tolyl)quinolin-2(1H)-one (2b). R_f = 0.4 (20% ethyl acetate in hexane); yellow solid; yield 60% (45 mg); mp 137-140 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (dd, *J* = 8.9, 2.3 Hz, 1H), 7.35 (t, *J* = 8.1 Hz, 3H), 7.18-7.15 (m, 3H), 3.87 (s, 3H), 2.48 (s, 3H), 1.20 (s, 9H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 158.5, 151.0, 145.4, 138.5, 136.9, 134.6, 129.4×2, 128.6, 124.8, 121.2, 119.1, 114.1, 34.4, 31.33, 31.30, 21.6; IR (KBr) ῡ 2957, 2929, 1646, 1071, 808, 640 cm^{-1}; HRMS (ESI/Q-TOF) m/z: $[M + H]^+$ calcd for $C_{21}H_{23}BrNO$ 384.0963; found 384.0934.



3-Bromo-6-(*tert*-butyl)-4-(4-methoxyphenyl)-1-methylquinolin-2(1H)-one (2c). R_f = 0.4 (20% ethyl acetate in hexane); white solid; yield 74% (55 mg); mp 151-153 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (dd, *J* = 8.9, 2.3 Hz, 1H), 7.36 (d, *J* = 8.9 Hz, 1H), 7.22-7.20 (m, 3H), 7.09-7.05 (m, 2H), 3.92 (s, 3H), 3.86 (s, 3H), 1.21 (s, 9H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 159.7, 158.5, 150.6, 145.5, 136.9, 130.2, 129.8, 128.6, 124.8, 121.4, 119.4, 114.1, 114.0, 55.4, 34.4, 31.3×2; IR (KBr) ῡ 2960, 2867, 1645, 1248, 816, 650 cm^{-1}; HRMS (ESI/Q-TOF) m/z: $[M + Na]^+$ calcd for $C_{21}H_{22}BrNO_2Na$ 422.0732; found 422.0756.

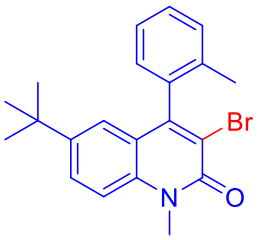


4-(3-Bromo-6-(*tert*-butyl)-1-methyl-2-oxo-1,2-dihydroquinolin-4-yl)benzonitrile (2d). R_f = 0.4 (20% ethyl acetate in hexane); yellow solid; yield 75% (55 mg); mp 170-173 ºC; ^{1}H NMR



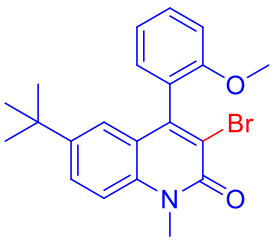
(700 MHz, $CDCl_3$) δ 7.87 (d, *J* = 8.1 Hz, 2H), 7.68 (dd, *J* = 8.9, 2.1 Hz, 1H), 7.43 (d, *J* = 8.1 Hz, 2H), 7.40 (d, *J* = 8.1 Hz, 1H), 6.95 (d, *J* = 2.1 Hz, 1H), 3.88 (s, 3H), 1.20 (s, 9H); $^{13}C\{^1H\}$ NMR (176 MHz, $CDCl_3$) δ 158.1, 148.7, 146.0, 142.2, 137.1, 132.7, 129.8, 129.3, 123.7, 120.2, 119.0, 118.5, 114.5, 112.9, 34.5, 31.4, 31.2; IR (KBr) ῡ 2964, 2929, 1647, 1069, 816, 632 cm^{-1}; HRMS (ESI/Q-TOF) m/z: $[M + H]^+$ calcd for $C_{21}H_{20}BrN_2O$ 395.0759; found 395.0735.

3-Bromo-6-(*tert*-butyl)-1-methyl-4-(o-tolyl)quinolin-2(1H)-one (2e). R_f = 0.4 (20% ethyl



acetate in hexane); white solid; yield 79% (59 mg); mp 135-139 ºC; ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.64 (dd, *J* = 8.8, 2.0 Hz, 1H), 7.42-7.33 (m, 4H), 7.09 (d, *J* = 7.4 Hz, 1H), 6.98 (d, *J* = 2.0 Hz, 1H), 3.88 (s, 3H), 2.05 (s, 3H), 1.17 (s, 9H); $^{13}C\{^1H\}$ NMR (176 MHz, $CDCl_3$) δ 158.5, 150.8, 145.8, 137.0, 136.9, 135.3, 130.3, 128.9, 128.7, 128.3, 126.2, 124.0, 120.6, 119.1, 114.2, 34.0, 31.28, 31.23, 19.4; IR (KBr) ῡ 2958, 2925, 1647, 1067, 815, 623 cm^{-1}; HRMS (ESI/Q-TOF) m/z: $[M + Na]^+$ calcd for $C_{21}H_{22}BrNONa$ 406.0782; found 406.0780.

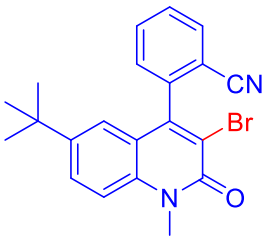
3-Bromo-6-(*tert*-butyl)-4-(2-methoxyphenyl)-1-methylquinolin-2(1H)-one (2f). R_f = 0.4



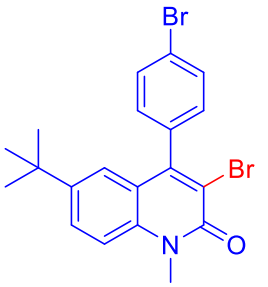
(20% ethyl acetate in hexane); yellow solid; yield 52% (39 mg); mp 152-153 ºC; ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.62 (dd, *J* = 9.0, 1.5 Hz, 1H), 7.51-7.48 (m, 1H), 7.36 (d, *J* = 8.8 Hz, 1H), 7.13-7.07 (m, 4H), 3.86 (s, 3H), 3.73 (s, 3H), 1.19 (s, 9H); $^{13}C\{^1H\}$ NMR (176 MHz, $CDCl_3$) δ 158.6, 156.2, 148.5, 145.4, 136.8, 130.4, 130.1, 128.4, 126.3, 124.2, 120.9, 120.8,

119.8, 114.1, 111.5, 55.8, 34.3, 31.2, 29.8; IR (KBr) ῡ 2957, 2929, 1644, 1026, 818, 756 cm^{-1}; HRMS (ESI/Q-TOF) m/z: $[M + H]^+$ calcd for $C_{21}H_{23}BrNO_2$ 402.0912; found 402.0900.

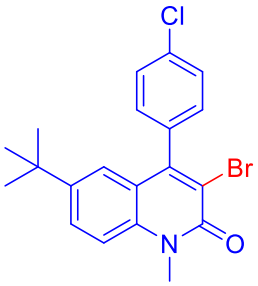
2-(3-Bromo-6-(*tert*-butyl)-1-methyl-2-oxo-1,2-dihydroquinolin-4-yl)benzonitrile (2g). R_f = 0.4 (20% ethyl acetate in hexane); white solid; yield 42% (30 mg); mp 145-148 ºC; ^1H NMR (700 MHz, $CDCl_3$) δ 7.89 (d, *J* = 7.3 Hz, 1H), 7.80 (t, *J* = 7.2 Hz, 1H), 7.68-7.64 (m, 2H), 7.40 (d, *J* = 7.3 Hz, 2H), 6.87 (s, 1H), 3.88 (s, 3H), 1.19 (s, 9H); $^{13}C\{^1H\}$ NMR (176 MHz, $CDCl_3$) δ 158.0, 147.0, 146.0, 141.2, 137.1, 133.4, 133.2, 129.9, 129.4, 129.3, 125.7, 123.3, 120.1, 116.7, 114.7, 112.7, 31.4, 31.2, 27.1; IR (KBr) ῡ 2960, 2870, 1647, 1071, 768, 517 cm^{-1}; HRMS (ESI/Q-TOF) m/z: $[M + H]^+$ calcd for $C_{21}H_{20}BrN_2O$ 395.0759; found 395.0778.



3-Bromo-4-(4-bromophenyl)-6-(*tert*-butyl)-1-methylquinolin-2(1H)-one (2h). R_f = 0.4 (20% ethyl acetate in hexane); white solid; yield 92% (66 mg); mp >180ºC; ^1H NMR (700 MHz, $CDCl_3$) δ 7.69 (d, *J* = 8.2 Hz, 2H), 7.66 (dd, *J* = 5.3, 2.0 Hz, 1H), 7.38 (d, *J* = 8.9 Hz, 1H), 7.17 (d, *J* = 8.2 Hz, 2H), 7.10 (d, *J* = 1.5 Hz, 1H), 3.87 (s, 3H), 1.21 (s, 9H); $^{13}C\{^1H\}$ NMR (176 MHz, $CDCl_3$) δ 158.3, 149.6, 145.8, 137.0, 136.4, 132.1, 130.5, 128.9, 124.3, 123.0, 120.7, 119.2, 114.3, 34.5, 31.4, 31.3; IR (KBr) ῡ 2961, 2918, 1642, 1066, 810, 626 cm^{-1}; HRMS (ESI/Q-TOF) m/z: $[M + H]^+$ calcd for $C_{20}H_{20}Br_2NO$ 447.9911; found 447.9944.

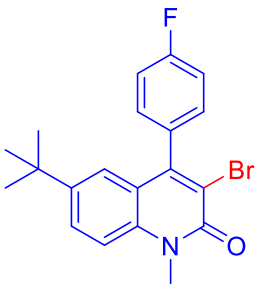


3-Bromo-6-(*tert*-butyl)-4-(4-chlorophenyl)-1-methylquinolin-2(1H)-one (2i). R_f = 0.4 (20%



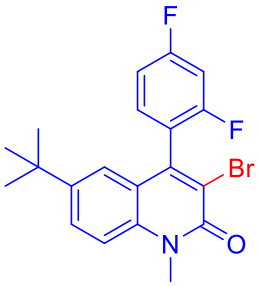
ethyl acetate in hexane); yellow solid; yield 77% (57 mg); mp >180ºC ; ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.66 (dd, *J* = 8.8, 1.9 Hz, 1H), 7.53 (d, *J* = 8.3 Hz, 2H), 7.38 (d, *J* = 8.9 Hz, 1H), 7.23 (d, *J* = 8.3 Hz, 2H), 7.10 (d, *J* = 1.8 Hz, 1H), 3.87 (s, 3H), 1.21 (s, 9H); ^{13}C{^{1}H} NMR (176 MHz, $CDCl_3$) δ 158.3, 149.6, 145.7, 136.9, 135.9, 134.8, 130.2, 129.1, 128.9, 124.2, 120.8, 119.2, 114.3, 34.4, 31.3, 31.2; IR (KBr) ῡ 2959, 2927, 1647, 1089, 813, 631 cm^{-1}; HRMS (ESI/Q-TOF) m/z: $[M + H]^+$ calcd for $C_{20}H_{20}BrClNO$ 404.0417; found 404.0434.

3-bromo-6-(*tert*-butyl)-4-(4-fluorophenyl)-1-methylquinolin-2(1H)-one (2j). R_f = 0.3 (20%



ethyl acetate in hexane); white solid; yield 41% (30 mg); mp 160-163 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (dd, *J* = 8.9, 2.1 Hz, 1H), 7.38 (d, *J* = 8.9 Hz, 1H), 7.27-7.23 (m, 4H), 7.09 (d, *J* = 2.1 Hz, 1H), 3.87 (s, 3H), 1.20 (s, 9H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 162.7 (d, $^1J_{C\text{-}F}$ = 248.1 Hz), 158.3, 149.8, 145.6, 136.9, 133.4 (d, $^4J_{C\text{-}F}$ = 3.6 Hz), 130.6 (d, $^3J_{C\text{-}F}$ = 8.1 Hz), 128.9, 124.3, 121.0, 119.4, 115.96 (d, $^2J_{C\text{-}F}$ = 21.8 Hz), 114.3, 34.4, 31.3, 31.2; IR (KBr) ῡ 2959, 2897, 1645, 1069, 813 cm^{-1}; HRMS (ESI/Q-TOF) m/z: $[M + H]^+$ calcd for $C_{20}H_{20}BrFNO$ 389.0712; found 389.0695.

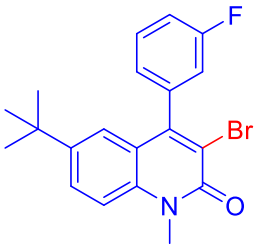
3-Bromo-6-(*tert*-butyl)-4-(2,4-difluorophenyl)-1-methylquinolin-2(1H)-one (2k). R_f = 0.3



(20% ethyl acetate in hexane); yellow solid; yield 40% (30 mg); mp 154-157 ºC; ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.67 (dd, *J* = 8.9, 1.7 Hz, 1H), 7.39 (d, *J* = 8.9 Hz, 1H), 7.06 (m, 4H), 3.87 (s, 3H), 1.22 (s, 9H); ^{13}C{^{1}H} NMR (176 MHz, $CDCl_3$) δ 158.2, 145.2 (d, ^1J = 235.6 Hz), 136.9, 131.74 (d, ^4J = 4.0 Hz), 131.71, 131.6 (d, ^4J = 4.0 Hz), 129.1, 123.5, 121.1-121.3 (m), 120.8, 120.4,

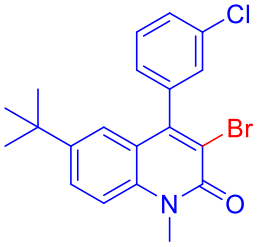
114.5, 112.1 (d, ^4J = 3.5 Hz), 112.0 (d, ^4J = 3.6 Hz), 104.9 (d, ^2J = 25.3 Hz), 104.7 (d, ^2J = 25.3 Hz), 34.4, 31.4, 31.2; IR (KBr) ῡ 2959, 2887, 1646, 1069, 813 cm^{-1}; HRMS (ESI/Q-TOF) m/z: $[M + Na]^+$ calcd for $C_{20}H_{18}BrF_2NONa$ 428.0438; found 428.0456.

3-Bromo-6-(*tert*-butyl)-4-(3-fluorophenyl)-1-methylquinolin-2(1H)-one (2l). R_f = 0.3 (20%



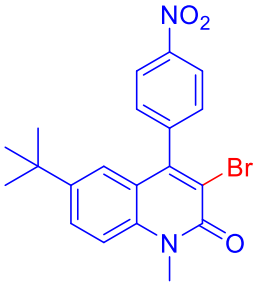
ethyl acetate in hexane); yellow solid; yield 71% (52 mg); mp 145-149 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (dd, *J* = 8.9, 2.3 Hz, 1H), 7.57-7.49 (m, 1H), 7.38 (d, *J* = 8.9 Hz, 1H), 7.26-7.18 (m, 1H), 7.09-6.99 (m, 3H), 3.87 (s, 3H), 1.20 (s, 9H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 162.8 (d, $^1J_{C\text{-}F}$ = 247.7 Hz), 158.3, 149.4 (d, $^4J_{C\text{-}F}$ = 1.7 Hz), 145.7, 139.4 (d, $^3J_{C\text{-}F}$ = 8.0 Hz), 136.9, 130.5 (d, $^3J_{C\text{-}F}$ = 8.3 Hz), 129.0, 124.5 (d, $^4J_{C\text{-}F}$ = 3.2 Hz), 124.2, 120.6, 119.0, 116.1, 115.9 (d, $^4J_{C\text{-}F}$ = 2.6 Hz), 115.6, 114.3, 34.4, 31.4, 31.2; IR (KBr) ῡ 2959, 2924, 1647, 1066, 815 cm^{-1}; HRMS (ESI/Q-TOF) m/z: $[M + Na]^+$ calcd for $C_{20}H_{19}BrFNONa$ 411.0532; found 411.0526.

3-Bromo-6-(*tert*-butyl)-4-(3-chlorophenyl)-1-methylquinolin-2(1H)-one (2m). R_f = 0.4



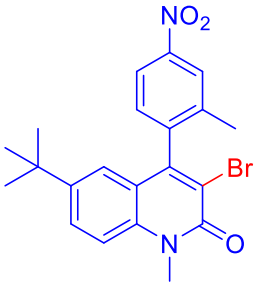
(20% ethyl acetate in hexane); yellow solid; yield 79% (62 mg); mp 115-119 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (dd, *J* = 8.9, 2.0 Hz, 1H), 7.49 (d, *J* = 4.5 Hz, 2H), 7.38 (d, *J* = 8.9 Hz, 1H), 7.29 (s, 1H), 7.19-7.15 (m, 1H), 7.08 (d, *J* = 1.9 Hz, 1H), 3.87 (s, 3H), 1.20 (s, 9H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 158.3, 149.2, 145.7, 139.1, 136.9, 134.7, 130.1, 129.0, 128.98, 128.90, 127.0, 124.1, 120.6, 119.1, 114.3, 34.4, 31.4, 31.2; IR (KBr) ῡ 2958, 2862, 1647, 1068, 793 cm^{-1}; HRMS (ESI/Q-TOF) m/z: $[M + Na]^+$ calcd for $C_{20}H_{19}BrClNONa$ 428.0236; found 428.0237.

3-Bromo-6-(*tert*-butyl)-1-methyl-4-(4-nitrophenyl)quinolin-2(1H)-one (2n). R_f = 0.4 (20%



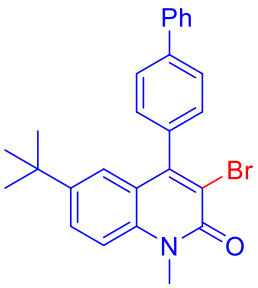
ethyl acetate in hexane); brown solid; yield 30% (22 mg); mp 155-159 ºC; ^{1}H NMR (700 MHz, $CDCl_3$) δ 8.44 (d, *J* = 8.4 Hz, 2H), 7.69 (d, *J* = 8.9 Hz, 1H), 7.50 (d, *J* = 8.4 Hz, 2H), 7.41 (d, *J* = 8.9 Hz, 1H), 6.96 (s, 1H), 3.89 (s, 3H), 1.19 (s, 9H); ^{13}C{^{1}H} NMR (176 MHz, $CDCl_3$) δ 158.0, 148.5, 148.1, 146.1, 144.1, 137.1, 130.1, 129.3, 124.2, 123.6, 120.0, 119.0, 114.6, 34.5, 31.5, 31.2; IR (KBr) ῡ 2957, 2820, 1536, 1071, 828, 598 cm^{-1}; HRMS (ESI/Q-TOF) m/z: [M + H]$^+$ calcd for $C_{20}H_{20}BrN_2O_3$ 416.0657; found 416.0688.

3-Bromo-6-(*tert*-butyl)-1-methyl-4-(2-methyl-4-nitrophenyl)quinolin-2(1H)-one (2o). R_f = 0.4 (20% ethyl acetate in hexane); brown solid; yield 30% (22 mg); mp 154-157 ºC; ^{1}H NMR



(700 MHz, $CDCl_3$) δ 8.28 (s, 1H), 8.24 (d, *J* = 8.3 Hz, 1H), 7.69 (dd, *J* = 8.9, 1.8 Hz, 1H), 7.42 (d, *J* = 8.9 Hz, 1H), 7.31 (d, *J* = 8.3 Hz, 1H), 6.84 (s, 1H), 3.89 (s, 3H), 2.18 (s, 3H), 1.18 (s, 9H); ^{13}C{^{1}H} NMR (176 MHz, $CDCl_3$) δ 158.1, 148.5, 148.2, 146.3, 143.6, 137.9, 137.1, 129.7, 129.3, 125.4, 123.0, 121.7, 119.6, 118.9, 114.7, 34.4, 31.4, 31.2, 19.6; IR (KBr) ῡ 2957, 2820, 1536, 1071, 828, 598 cm^{-1}; HRMS (ESI/Q-TOF) m/z: [M + H]$^+$ calcd for $C_{21}H_{22}BrN_2O_3$ 429.0814; found 429.0825.

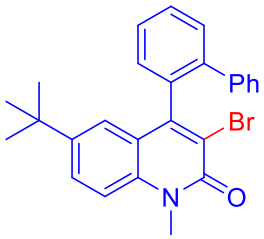
4-([1,1'-Biphenyl]-4-yl)-3-bromo-6-(*tert*-butyl)-1-methylquinolin-2(1H)-one (2p). R_f = 0.4



(20% ethyl acetate in hexane); yellow solid; yield 86% (63 mg); mp >180ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (d, *J* = 8.1 Hz, 2H), 7.73 (d, *J* = 7.8 Hz, 2H), 7.66 (dd, *J* = 8.9, 2.1 Hz, 1H), 7.50 (t, *J* = 7.6 Hz, 2H), 7.42-7.36 (m, 4H), 7.21 (d, *J* = 2.1 Hz, 1H), 3.89 (s, 3H), 1.20 (s, 9H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 158.5, 150.5, 145.6, 141.3, 140.4, 136.9, 136.4, 129.3,

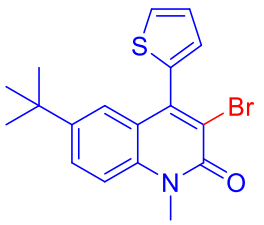
129.0, 128.7, 127.8, 127.2, 124.6, 121.0, 119.0, 114.2, 34.4, 31.3, 31.2; IR (KBr) ῡ 2959, 2878, 1646, 1066, 818, 692 cm^{-1}; HRMS (ESI/Q-TOF) m/z: $[M + H]^+$ calcd for $C_{26}H_{25}BrNO$ 446.1119; found 446.1109.

4-([1,1'-Biphenyl]-2-yl)-3-bromo-6-(*tert*-butyl)-1-methylquinolin-2(1H)-one (2q). R_f = 0.4



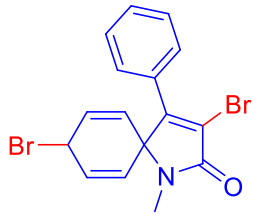
(20% ethyl acetate in hexane); yellow solid; yield 81% (59 mg); mp >180ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (dd, *J* = 18.6, 8.2 Hz, 2H), 7.65-7.62 (m, 2H), 7.53-7.49 (m, 1H), 7.43-7.37 (m, 5H), 6.90 (d, *J* = 2.1 Hz, 1H), 3.94 (s, 3H), 1.04 (s, 9H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 158.5, 149.7, 145.6, 136.9, 135.0, 133.6, 130.5, 129.1, 128.8, 128.6, 126.7, 126.6, 126.4, 125.4, 125.3, 124.7, 121.3, 120.3, 114.2, 34.2, 31.3, 31.0; IR (KBr) ῡ 2958, 2865, 1646, 1065, 802, 630 cm^{-1}; HRMS (ESI/Q-TOF) m/z: $[M + Na]^+$ calcd for $C_{26}H_{24}BrNONa$ 469.0939; found 469.0942.

3-Bromo-6-(*tert*-butyl)-1-methyl-4-(thiophen-2-yl)quinolin-2(1H)-one (2r). R_f = 0.4 (20%



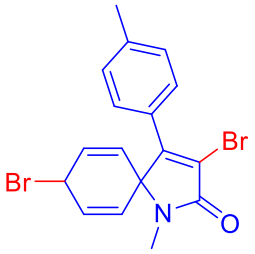
ethyl acetate in hexane); brown solid; yield 66% (50 mg); mp 182-185 ºC; ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.65 (dd, *J* = 8.8, 1.7 Hz, 1H), 7.54-7.53 (m, 1H), 7.37-7.36 (m, 2H), 7.28 (s, 1H), 7.11 (d, *J* = 4.8 Hz, 1H), 3.86 (s, 3H), 1.23 (s, 9H); $^{13}C\{^1H\}$ NMR (176 MHz, $CDCl_3$) δ 158.4, 146.6, 145.6, 136.9, 136.8, 128.8, 128.4, 126.1, 125.1, 124.3, 121.1, 119.6, 114.2, 34.4, 31.3, 31.2; IR (KBr) ῡ 2957, 2880, 1641, 1064, 655 cm^{-1}; HRMS (ESI/Q-TOF) m/z: $[M + H]^+$ calcd for $C_{18}H_{19}BrNOS$ 376.0371; found 376.0379.

3,8-Dibromo-1-methyl-4-phenyl-1-azaspiro[4.5]deca-3,6,9-trien-2-one (**4a**)**.** R_f = 0.3 (20%



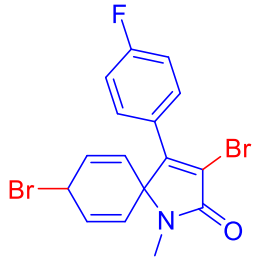
ethyl acetate in hexane); brown solid; yield 75% (73 mg); mp 112-115 ºC; ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.60-7.59 (m, 2H), 7.40-7.38 (m, 3H), 6.43 (dd, *J* = 9.8, 3.6 Hz, 2H), 5.57 (d, *J* = 9.7 Hz, 2H), 5.19 (s, 1H), 2.82 (s, 3H); ^{13}C{^{1}H} NMR (176 MHz, $CDCl_3$) δ 165.8, 154.4, 132.3, 130.9, 130.1, 128.5, 128.3, 128.2, 118.0, 66.5, 38.8, 26.0; IR (KBr) ῡ 2920, 2818, 1020, 870, 700 cm^{-1}; HRMS (ESI/Q-TOF) m/z: $[M + H]^+$ calcd for $C_{16}H_{14}Br_2NO$ 393.9442; found 393.9461.

3,8-Dibromo-1-methyl-4-(p-tolyl)-1-azaspiro[4.5]deca-3,6,9-trien-2-one (**4b**)**.** R_f = 0.3



(20% ethyl acetate in hexane); brown solid; yield 85% (84 mg); mp 118-121 ºC; ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.58 (d, *J* = 8.0 Hz, 2H), 7.22-7.19 (m, 2H), 6.36-6.34 (m, 2H), 5.60-5.59 (m, 2H), 5.02 (s, 1H), 2.82 (s, 3H), 2.36 (s, 3H); ^{13}C{^{1}H} NMR (176 MHz, $CDCl_3$) δ 165.9, 153.7, 140.4, 132.1, 131.8, 129.2, 128.5, 128.3, 117.2, 66.4, 48.5, 25.9, 21.6. IR (KBr) ῡ 2912, 2870, 1070, 880, 650 cm^{-1}; HRMS (ESI/Q-TOF) m/z: $[M + H]^+$ calcd for $C_{17}H_{16}Br_2NO$ 409.9599; found 409.9609.

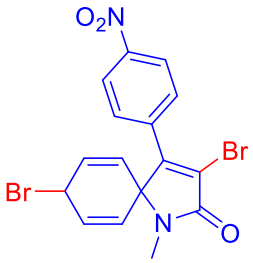
3,8-Dibromo-4-(4-fluorophenyl)-1-methyl-1-azaspiro[4.5]deca-3,6,9-trien-2-one (**4c**)**.** R_f =



0.3 (20% ethyl acetate in hexane); brown solid; yield 66% (65 mg); mp 90-93 ºC; ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.65-7.61 (m, 2H), 7.11-7.06 (m, 2H), 6.36 (dd, *J* = 9.8, 3.5 Hz, 2H), 5.60-5.57 (m, 2H), 5.01 (s, 1H), 2.82 (s, 3H); ^{13}C{^{1}H} NMR (176 MHz, $CDCl_3$) δ 165.7, 163.6 (d, $^1J_{C\text{-}F}$ = 251.2 Hz), 152.8, 132.5, 132.1, 130.6 (d, $^3J_{C\text{-}F}$ = 8.9 Hz), 128.36, 128.28, 126.9 (d, $^4J_{C\text{-}F}$ = 3.8 Hz), 115.7 (d, $^2J_{C\text{-}F}$ = 22.0 Hz), 66.5, 48.2, 26.0; IR (KBr) ῡ 2924, 2852, 1704, 1030, 752,

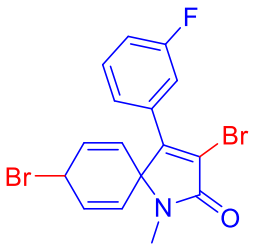
645 cm^{-1}; HRMS (ESI/Q-TOF) m/z: $[M + H]^+$ calcd for $C_{16}H_{13}Br_2FNO$ 411.9348; found 411.9348.

3,8-Dibromo-1-methyl-4-(4-nitrophenyl)-1-azaspiro[4.5]deca-3,6,9-trien-2-one (4d). R_f =



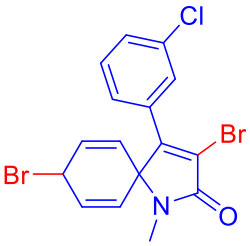
0.4 (20% ethyl acetate in hexane); yellow solid; yield 60% (56 mg); mp 150-154 ºC; ^{1}H NMR (700 MHz, $CDCl_3$) δ 8.25 (d, *J* = 8.6 Hz, 2H), 7.77 (d, *J* = 8.6 Hz, 2H), 6.47-6.38 (m, 2H), 5.60 (dd, *J* = 22.7, 9.6 Hz, 2H), 4.99 (s, 1H), 2.85 (s, 3H); ^{13}C{^{1}H} NMR (176 MHz, $CDCl_3$) δ 165.2, 151.9, 148.6, 137.4, 132.9, 129.8, 128.0, 123.9, 121.0, 66.8, 48.0, 26.4. IR (KBr) ῡ 2919, 2850, 1705, 1034, 694 cm^{-1}; HRMS (ESI/Q-TOF) m/z: $[M + H]^+$ calcd for $C_{16}H_{13}Br_2N_2O_3$ 438.9293; found 438.9279.

3,8-Dibromo-4-(3-fluorophenyl)-1-methyl-1-azaspiro[4.5]deca-3,6,9-trien-2-one (4e). R_f =



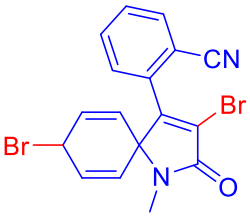
0.3 (20% ethyl acetate in hexane); brown solid; yield 58% (56 mg); mp 108-111 ºC; ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.39-7.34 (m, 3H), 7.10 (t, *J* = 7.5 Hz, 1H), 6.37 (dd, *J* = 9.8, 3.4 Hz, 2H), 5.57 (dd, *J* = 22.3, 9.9 Hz, 2H), 4.99 (s, 1H), 2.82 (s, 3H); ^{13}C{^{1}H} NMR (176 MHz, $CDCl_3$) δ 165.5, 162.5 (d, $^1J_{C\text{-}F}$ = 247.0 Hz), 152.7 (d, $^4J_{C\text{-}F}$ = 2.3 Hz), 132.3, 130.1 (d, $^3J_{C\text{-}F}$ = 8.3 Hz), 128.0, 127.9, 124.3 (d, $^4J_{C\text{-}F}$ = 3.0 Hz), 119.0, 117.0 (d, $^2J_{C\text{-}F}$ = 21.0 Hz), 115.5 (d, $^2J_{C\text{-}F}$ = 23.3 Hz), 66.5, 48.0, 26.0; IR (KBr) ῡ 2919, 2870, 1715, 1034, 672 cm^{-1}; HRMS (ESI/Q-TOF) m/z: $[M + H]^+$ calcd for $C_{16}H_{13}Br_2FNO$ 411.9348; found 411.9349.

3,8-Dibromo-4-(3-chlorophenyl)-1-methyl-1-azaspiro[4.5]deca-3,6,9-trien-2-one (4f). R_f =



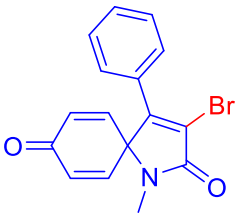
0.3 (20% ethyl acetate in hexane); brown solid; yield 46% (44 mg); mp 125-129 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 (s, 1H), 7.45-7.43 (m, 1H), 7.39-7.32 (m, 2H), 6.46 (dd, *J* = 9.9, 3.4 Hz, 2H), 5.55 (d, *J* = 9.5 Hz, 2H), 5.16 (t, *J* = 3.17 Hz, 1H), 2.83 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.4, 153.0, 134.6, 132.8, 132.6, 130.1, 129.9, 128.1, 127.9, 126.7, 119.1, 66.5, 38.3, 26.1; IR (KBr) ῡ 2927, 1096, 806, 754 cm^{-1}; HRMS (ESI/Q-TOF) m/z: $[M + H]^+$ calcd for $C_{16}H_{13}Br_2ClNO$ 427.9052; found 427.9040.

2-(3,8-Dibromo-1-methyl-2-oxo-1-azaspiro[4.5]deca-3,6,9-trien-4-yl)benzonitrile (4g).



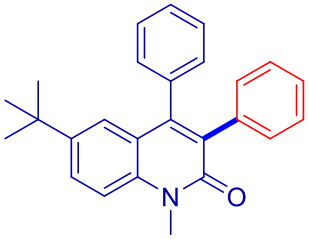
R_f = 0.3 (20% ethyl acetate in hexane); yellow solid; yield 55% (54 mg); mp 125-128 ºC; ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.75 (d, *J* = 7.6 Hz, 1H), 7.62 (t, *J* = 7.8 Hz, 1H), 7.53-7.50 (m, 1H), 7.23 (d, *J* = 7.8 Hz, 1H), 6.30 (dd, *J* = 87.2, 1.5 Hz, 2H), 5.76 (dd, *J* = 89.5, 7.1 Hz, 2H), 4.82 (s, 1H), 2.90 (s, 3H);^{13}C{^{1}H} NMR (176 MHz, $CDCl_3$) δ 164.9, 151.9, 134.6, 133.3, 132.5, 129.9, 129.6, 128.3, 127.8, 122.6, 117.5, 112.9, 68.0, 47.5, 26.7; IR (KBr) ῡ 2922, 2850, 1705, 811, 761 cm^{-1}; HRMS (ESI/Q-TOF) m/z: $[M + H]^+$ calcd for $C_{17}H_{13}Br_2N_2O$ 418.9395; found 418.9402.

3-Bromo-1-methyl-4-phenyl-1-azaspiro[4.5]deca-3,6,9-triene-2,8-dione (4j). R_f = 0.3 (20%

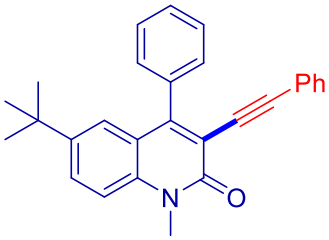


ethyl acetate in hexane); brown solid; yield 70% (52 mg); mp 140-143 ºC; ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.42-7.37 (m, 5H), 6.51 (q, *J* = 10.3 Hz, 4H), 2.94 (s, 3H); ^{13}C{^{1}H} NMR (176 MHz, $CDCl_3$) δ 183.8, 165.9, 151.4, 144.2, 133.6, 130.4, 129.4, 128.9, 127.9, 120.0, 68.4, 26.7; IR (KBr) ῡ 2976, 2912, 1709, 997, 739 cm^{-1}; HRMS (ESI/Q-TOF) m/z: $[M + H]^+$ calcd for $C_{16}H_{13}BrNO_2$ 330.0130; found 330.0125.

6-(*tert*-Butyl)-1-methyl-3,4-diphenylquinolin-2(1H)-one (**5**)**.** R_f = 0.4 (20% ethyl acetate in hexane); brown solid; yield 95% (62 mg); mp 175-179 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (dd, *J* = 8.8, 2.2 Hz, 1H), 7.40 (d, *J* = 8.9 Hz, 1H), 7.31-7.27 (m, 3H), 7.24-7.09 (m, 8H), 3.84 (s, 3H), 1.22 (s, 9H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 161.9, 148.0, 144.8, 137.6, 136.5, 136.2, 132.0, 130.7, 130.0, 128.1, 127.9, 127.6, 127.5, 126.9, 124.7, 121.1, 113.9, 34.4, 31.3, 30.1; IR (KBr) ῡ 2962, 2902, 1638, 1068, 701, 588 cm^{-1}; HRMS (ESI/Q-TOF) m/z: [M + Na]$^+$ calcd for $C_{26}H_{25}NONa$ 390.1834; found 390.1814.



6-(*tert*-Butyl)-1-methyl-4-phenyl-3-(phenylethynyl)quinolin-2(1H)-one (**6**)**.** R_f = 0.4 (20% ethyl acetate in hexane); brown solid; yield 90% (68 mg); mp 165-169 ºC; ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.63 (dd, *J* = 8.8, 2.0 Hz, 1H), 7.57-7.55 (m, 2H), 7.53-7.51 (m, 1H), 7.47-7.46 (m, 2H), 7.37 (d, *J* = 8.8 Hz, 1H), 7.34 (d, *J* = 1.9 Hz, 1H), 7.24-7.19 (m, 5H), 3.84 (s, 3H), 1.23 (s, 9H); ^{13}C{H} NMR (176 MHz, $CDCl_3$) δ 160.9, 153.2, 145.2, 137.4, 136.5, 131.8, 129.6, 128.9, 128.6, 128.35, 128.32, 128.1, 124.6, 123.4, 120.5, 115.8, 114.2, 98.2, 85.9, 34.4, 31.3, 30.2; IR (KBr) ῡ 2959, 2924, 1643, 1079, 815, 693 cm^{-1}; HRMS (ESI/Q-TOF) m/z: [M + Na]$^+$ calcd for $C_{28}H_{25}NONa$ 414.1834; found 414.1815.



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NMR SPECTRA

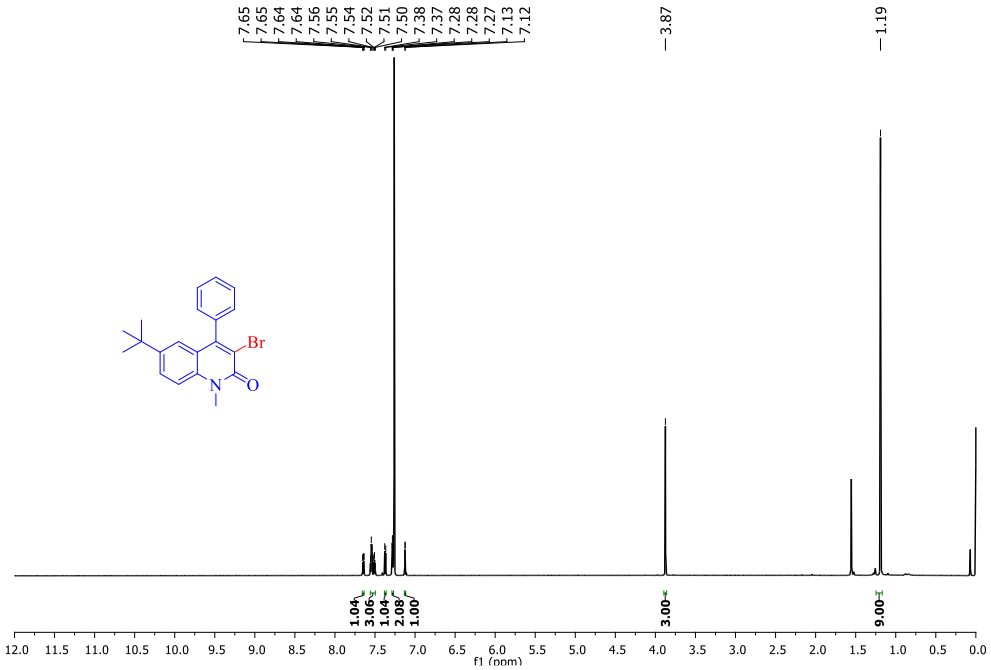


Fig. S9. ^{1}H NMR(700 MHz, $CDCl_3$) spectrum of 3-Bromo-6-(*tert*-butyl)-1-methyl-4-phenylquinolin-2(1H)-one (**2a**)

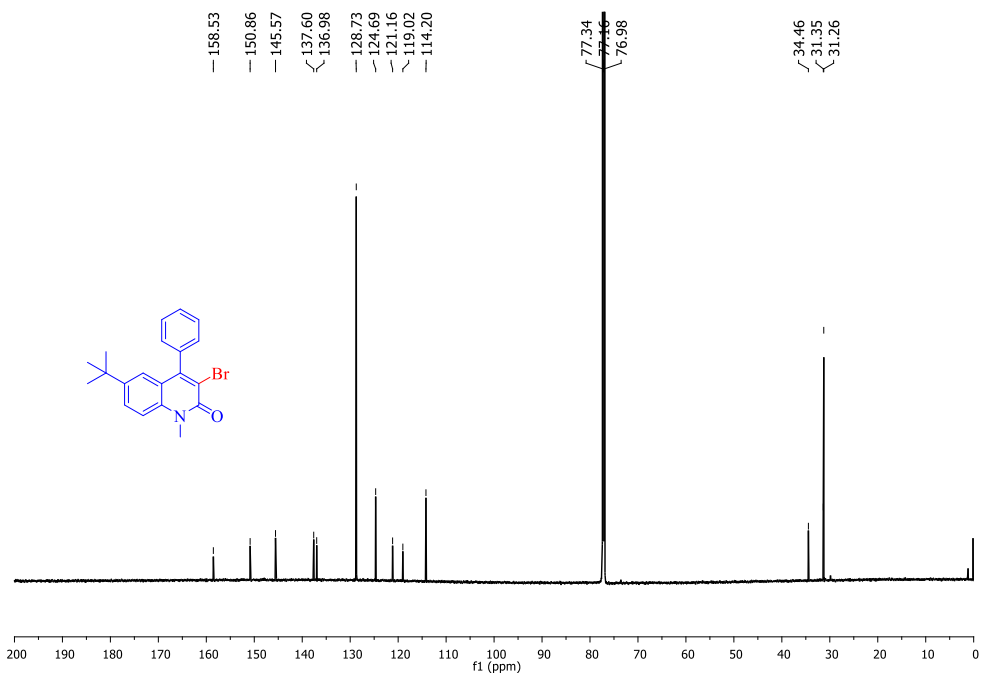


Fig. S10. ^{13}C{^{1}H} NMR (176 MHz, $CDCl_3$) spectrum of 3-Bromo-6-(*tert*-butyl)-1-methyl-4-phenylquinolin-2(1H)-one (**2a**)

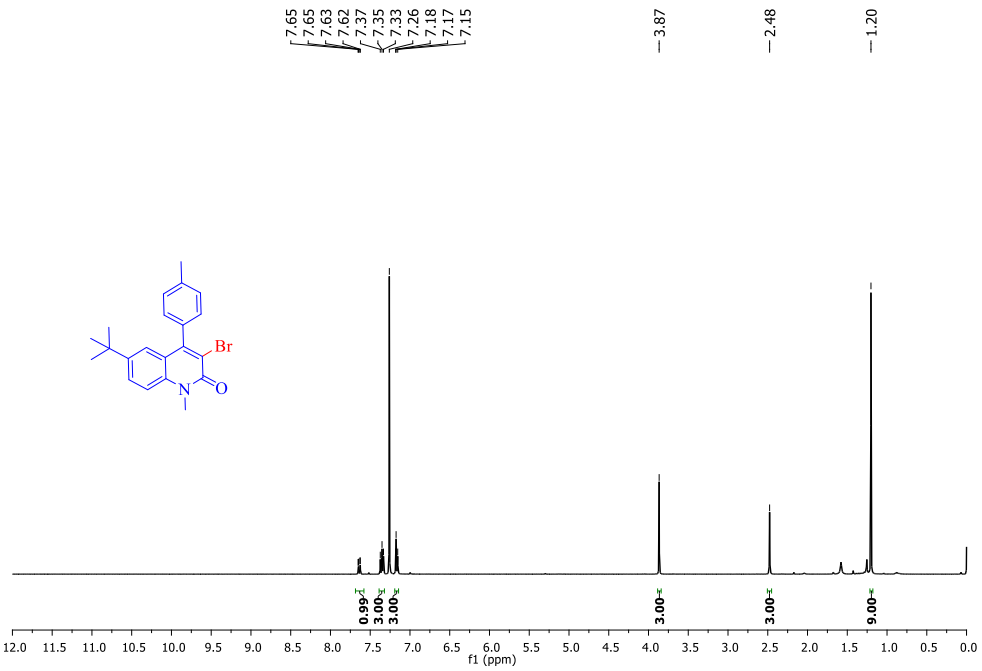


Fig. S11. ^{1}H NMR(400 MHz, $CDCl_3$) spectrum of 3-Bromo-6-(*tert*-butyl)-1-methyl-4-(p-tolyl)quinolin-2(1H)-one (**2b**)

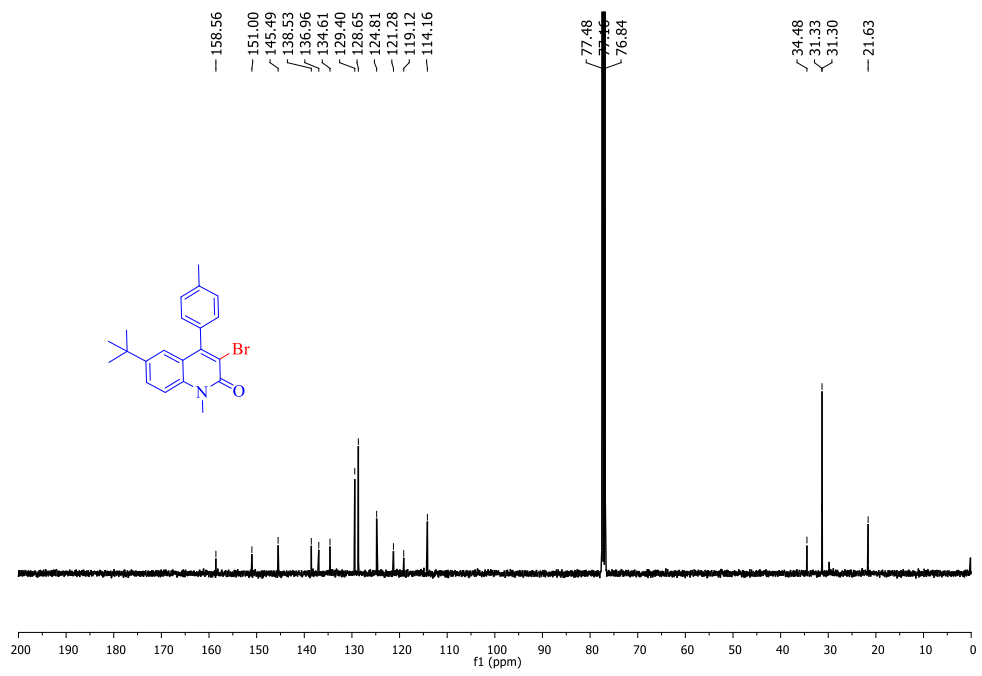


Fig. S12. $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of 3-Bromo-6-(*tert*-butyl)-1-methyl-4-(p-tolyl)quinolin-2(1H)-one (**2b**)

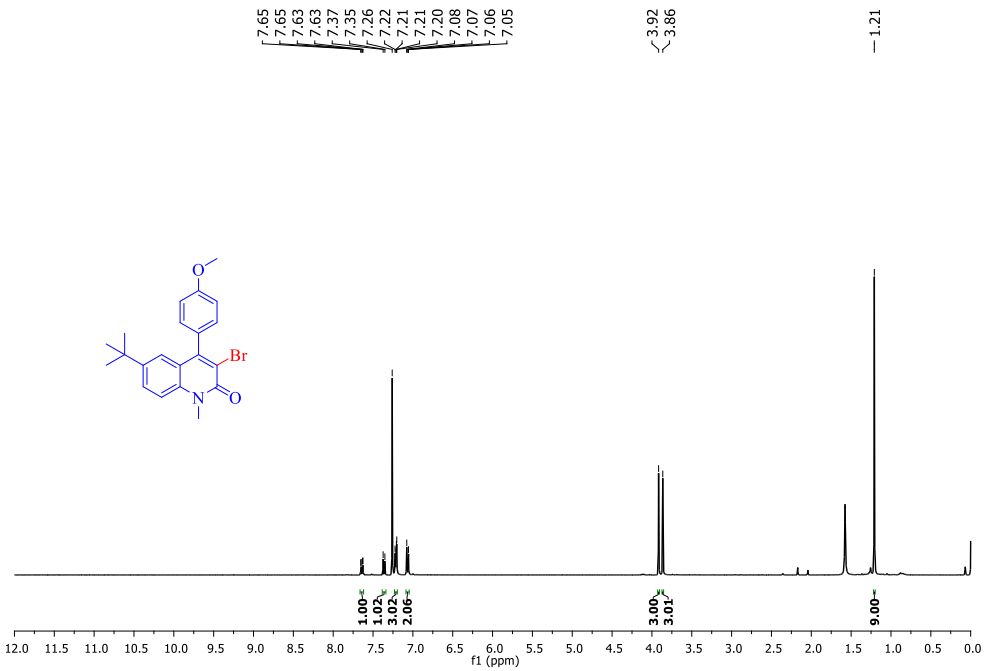


Fig. S13. ^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3-Bromo-6-(*tert*-butyl)-4-(4-methoxyphenyl)-1-methylquinolin-2(1H)-one (**2c**)

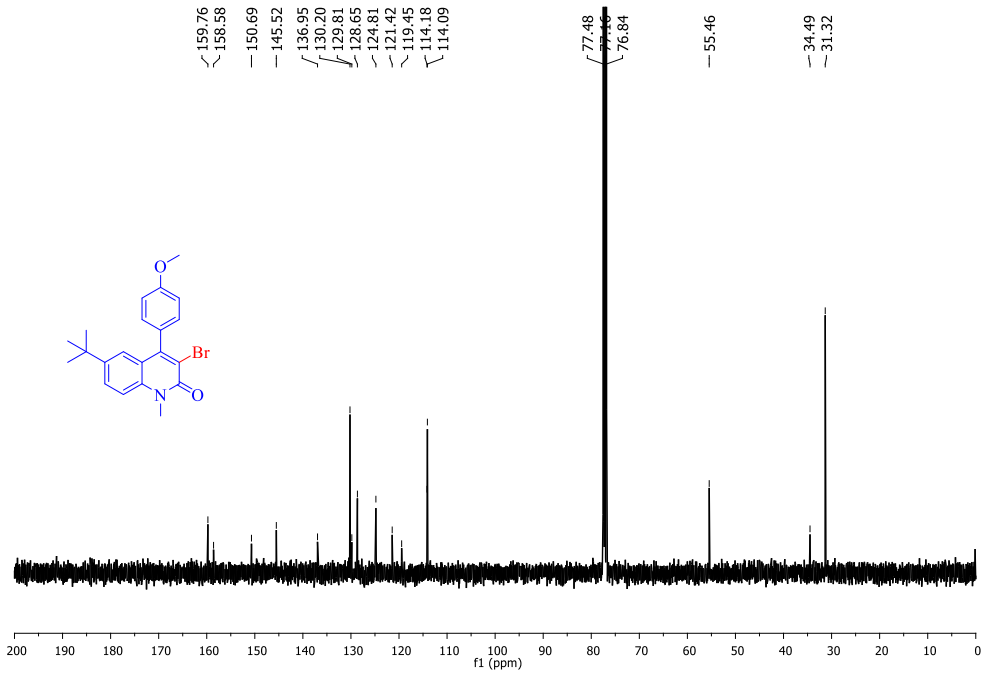


Fig. S14. $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of 3-Bromo-6-(*tert*-butyl)-4-(4-methoxyphenyl)-1-methylquinolin-2(1H)-one (**2c**)

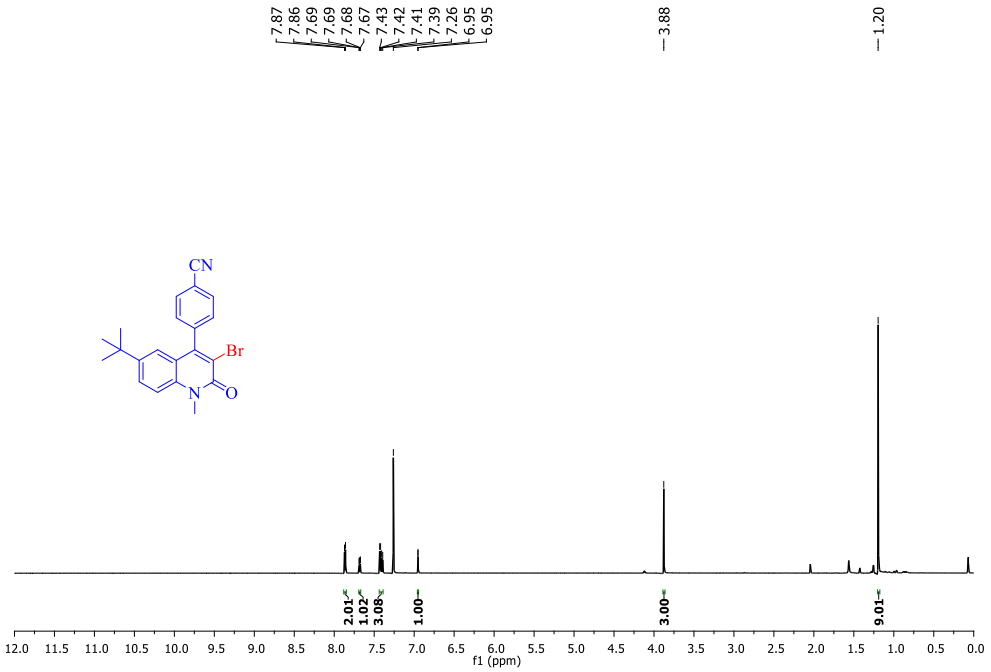


Fig. S15. ^{1}H NMR (700 MHz, $CDCl_3$) spectrum of 4-(3-Bromo-6-(*tert*-butyl)-1-methyl-2-oxo-1,2-dihydroquinolin-4-yl)benzonitrile (**2d**)

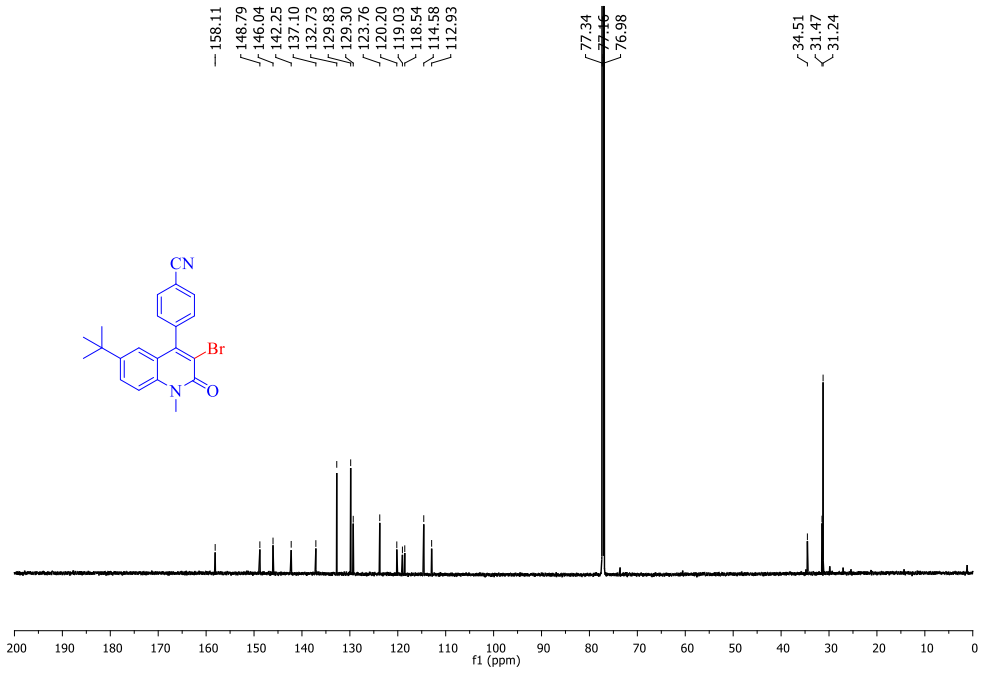


Fig. S16. $^{13}C\{^{1}H\}$ NMR (176 MHz, $CDCl_3$) spectrum of 4-(3-Bromo-6-(*tert*-butyl)-1-methyl-2-oxo-1,2-dihydroquinolin-4-yl)benzonitrile (**2d**)

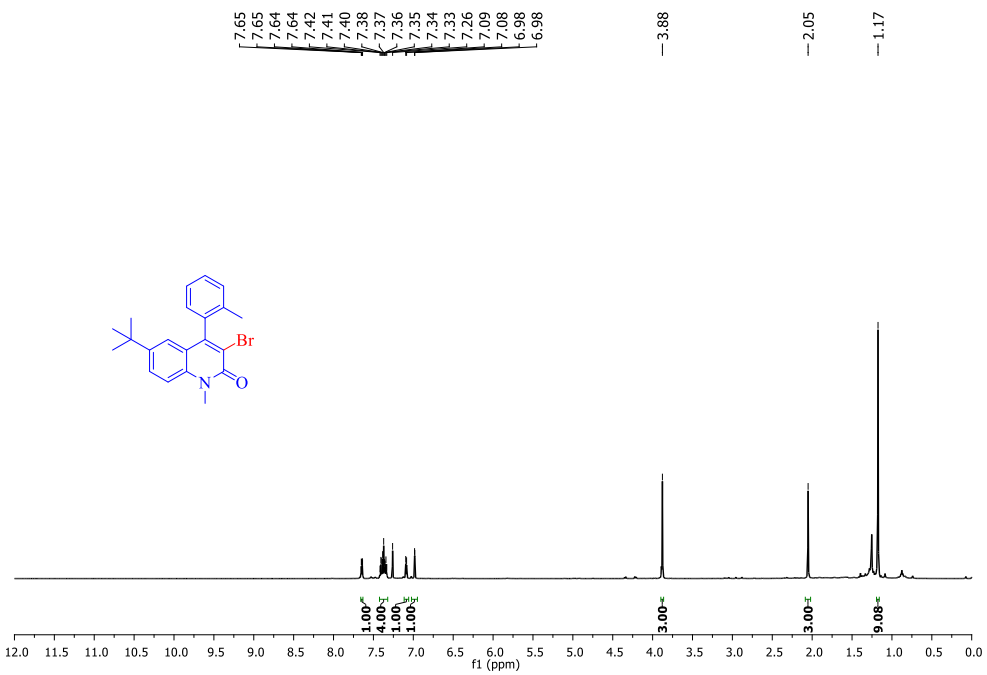


Fig. S17. ^{1}H NMR (700 MHz, $CDCl_3$) spectrum of 3-Bromo-6-(*tert*-butyl)-1-methyl-4-(o-tolyl)quinolin-2(1H)-one (**2e**). (Minor atropisomer could not be separated using column chromatography)

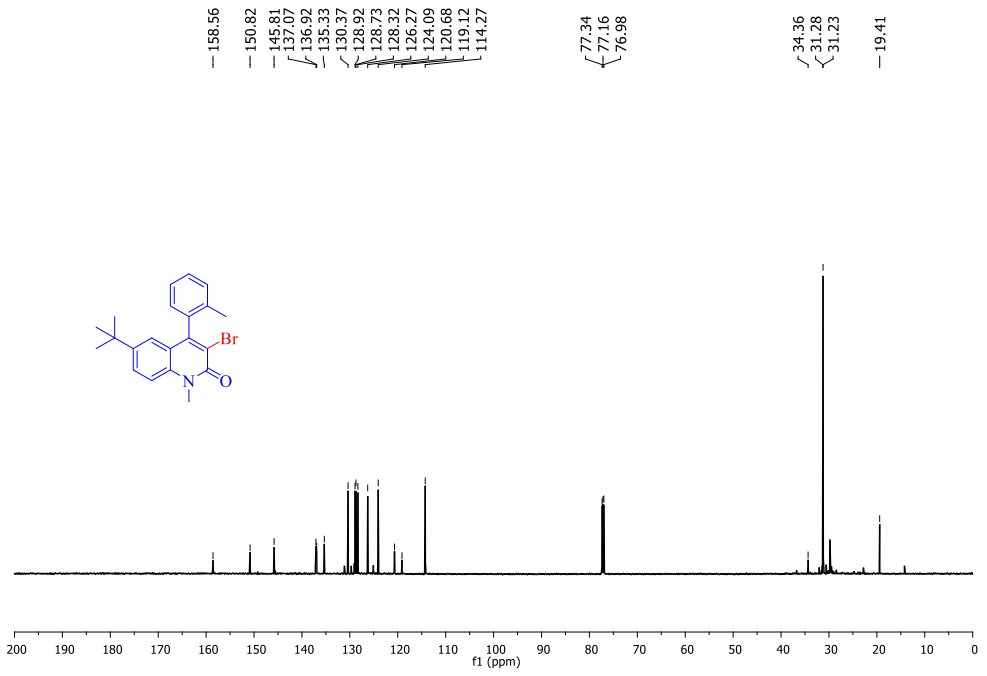


Fig. S18. $^{13}C\{^{1}H\}$ NMR (176 MHz, $CDCl_3$) spectrum of 3-Bromo-6-(*tert*-butyl)-1-methyl-4-(o-tolyl)quinolin-2(1H)-one (**2e**)

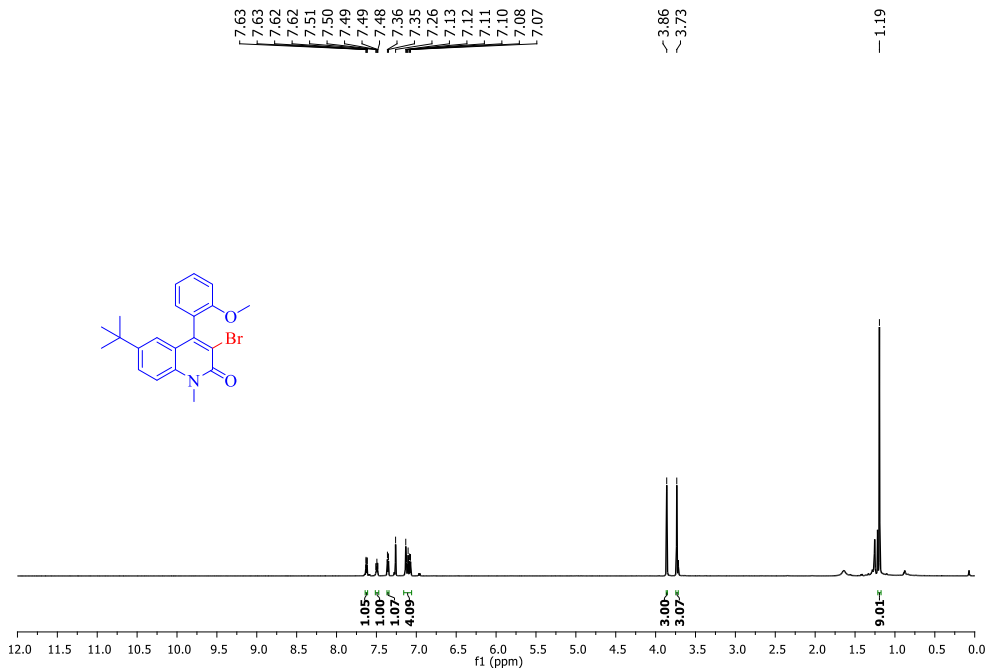


Fig. S19. ^{1}H NMR (700 MHz, $CDCl_3$) spectrum of 3-Bromo-6-(*tert*-butyl)-4-(2-methoxyphenyl)-1-methylquinolin-2(1H)-one (**2f**). (Minor atropisomer could not be separated using column chromatography)

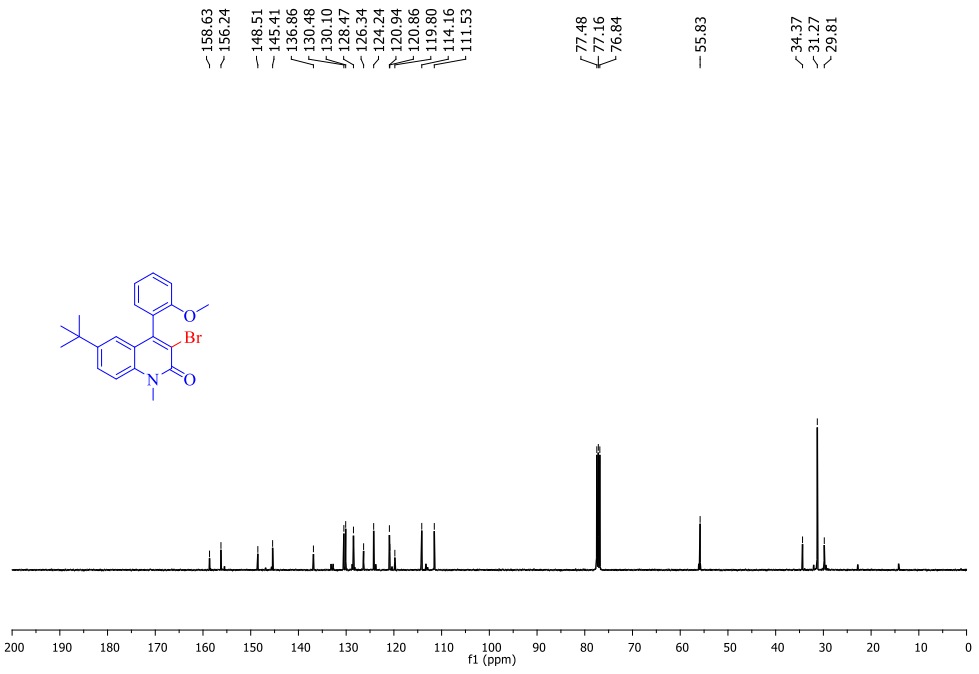


Fig. S20. $^{13}C\{^{1}H\}$ NMR (176 MHz, $CDCl_3$) spectrum of 3-Bromo-6-(*tert*-butyl)-4-(2-methoxyphenyl)-1-methylquinolin-2(1H)-one (**2f**)

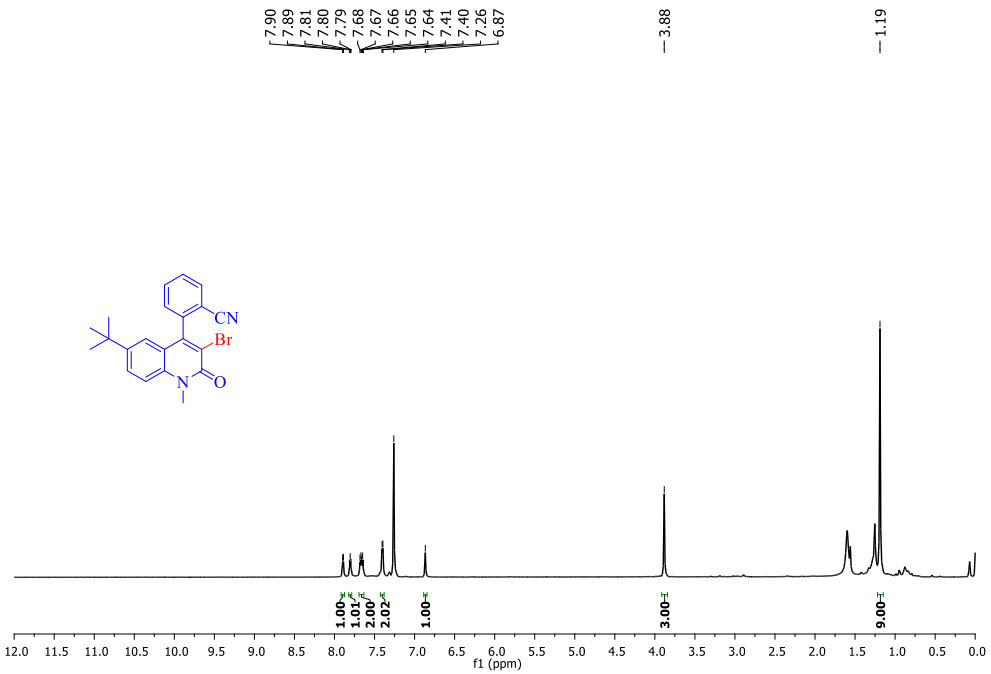


Fig. S21. ^{1}H NMR (700 MHz, $CDCl_3$) spectrum of 2-(3-bromo-6-(*tert*-butyl)-1-methyl-2-oxo-1,2-dihydroquinolin-4-yl)benzonitrile (**2g**). (Minor atropisomer could not be separated using column chromatography)

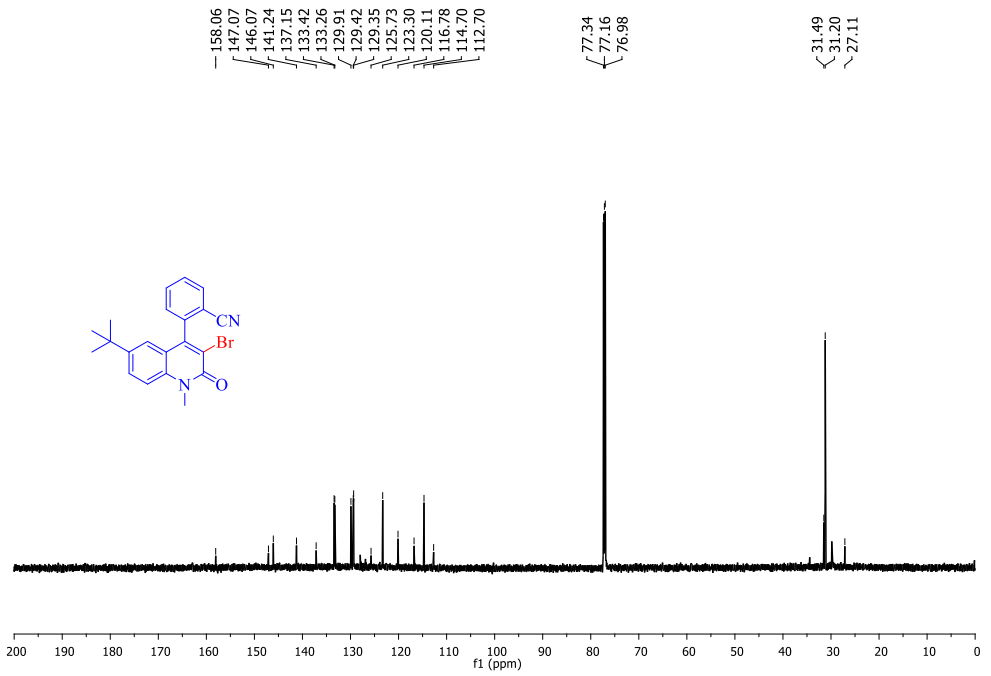


Fig. S22. $^{13}C\{^{1}H\}$ NMR (176 MHz, $CDCl_3$) spectrum of 2-(3-bromo-6-(*tert*-butyl)-1-methyl-2-oxo-1,2-dihydroquinolin-4-yl)benzonitrile (**2g**)

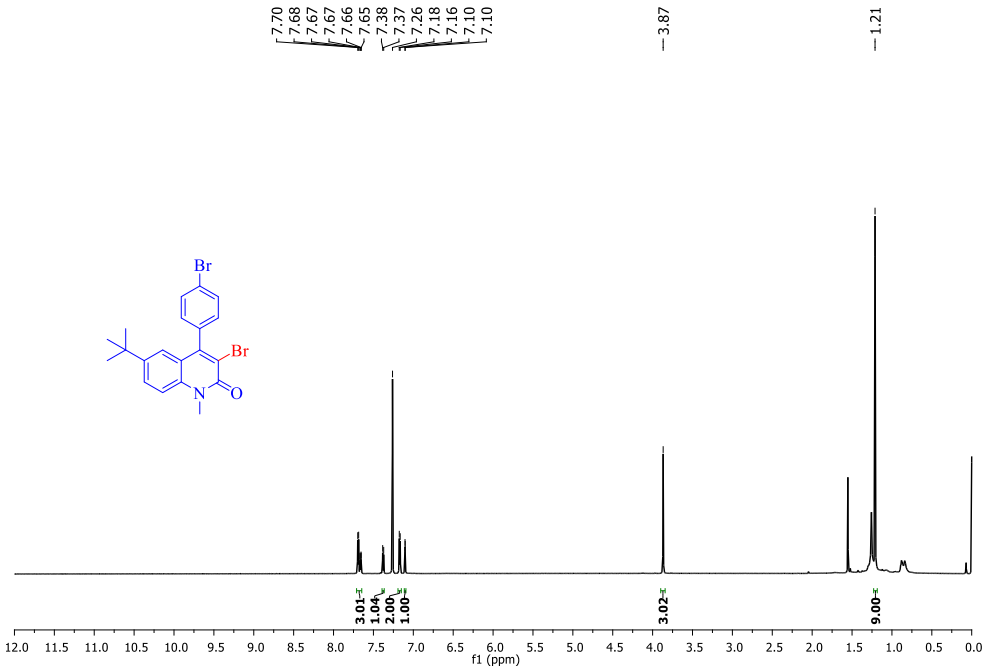


Fig. S23. ^{1}H NMR (700 MHz, $CDCl_3$) spectrum of 3-Bromo-4-(4-bromophenyl)-6-(*tert*-butyl)-1-methylquinolin-2(1H)-one (**2h**)

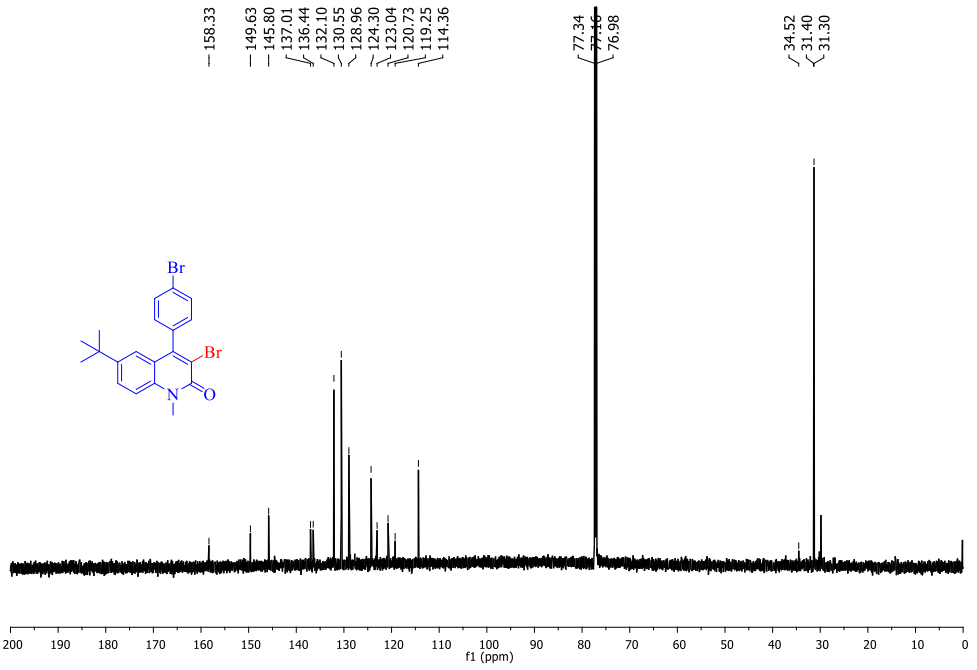


Fig. S24. $^{13}C\{^{1}H\}$ NMR (176 MHz, $CDCl_3$) spectrum of 3-Bromo-4-(4-bromophenyl)-6-(*tert*-butyl)-1-methylquinolin-2(1H)-one (**2h**)

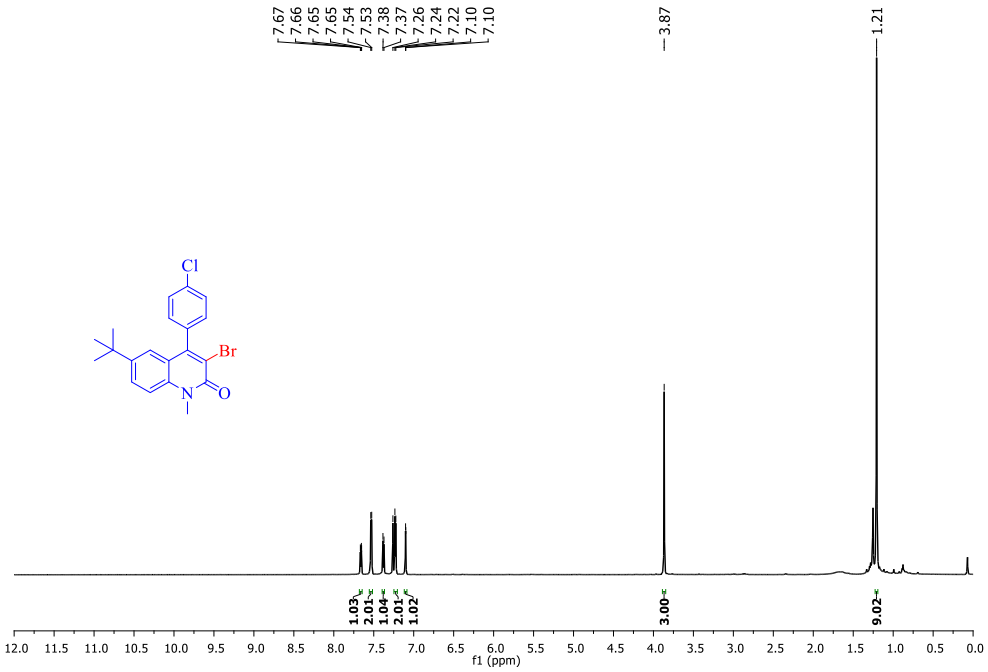


Fig. S25. ^{1}H NMR (700 MHz, $CDCl_3$) spectrum of 3-Bromo-6-(*tert*-butyl)-4-(4-chlorophenyl)-1-methylquinolin-2(1H)-one (**2i**)

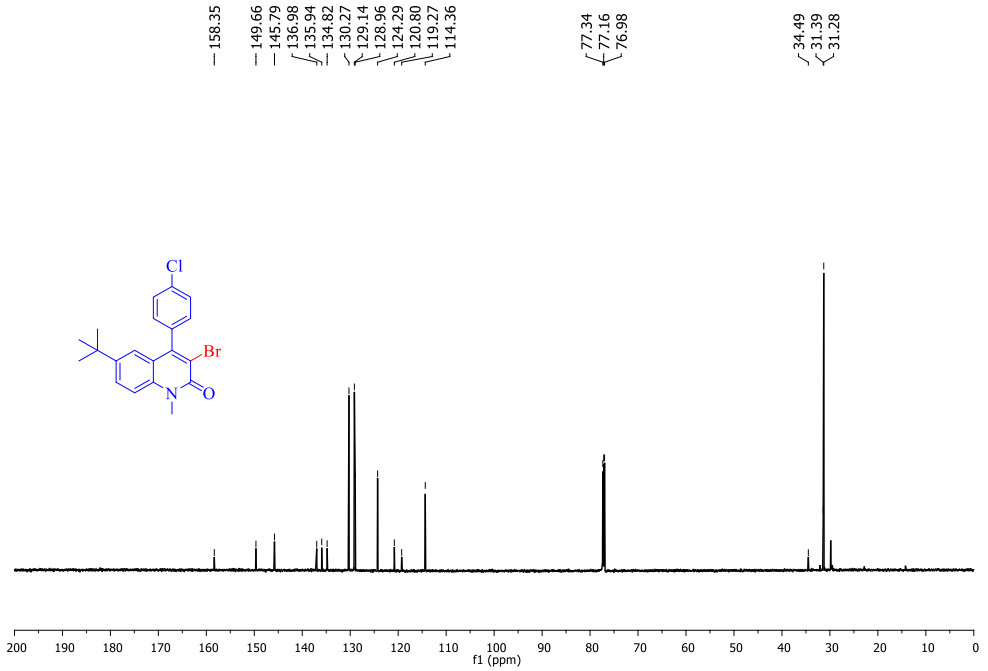


Fig. S26. $^{13}C\{^{1}H\}$ NMR (176 MHz, $CDCl_3$) spectrum of 3-Bromo-6-(*tert*-butyl)-4-(4-chlorophenyl)-1-methylquinolin-2(1H)-one (**2i**)

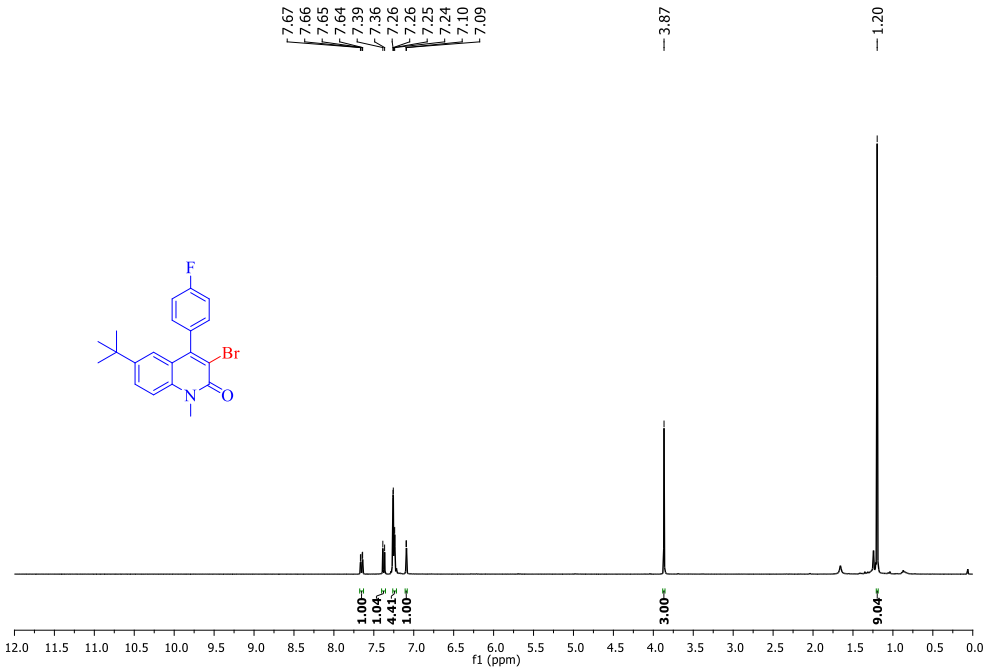


Fig. S27. ^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3-bromo-6-(*tert*-butyl)-4-(4-fluorophenyl)-1-methylquinolin-2(1H)-one (**2j**)

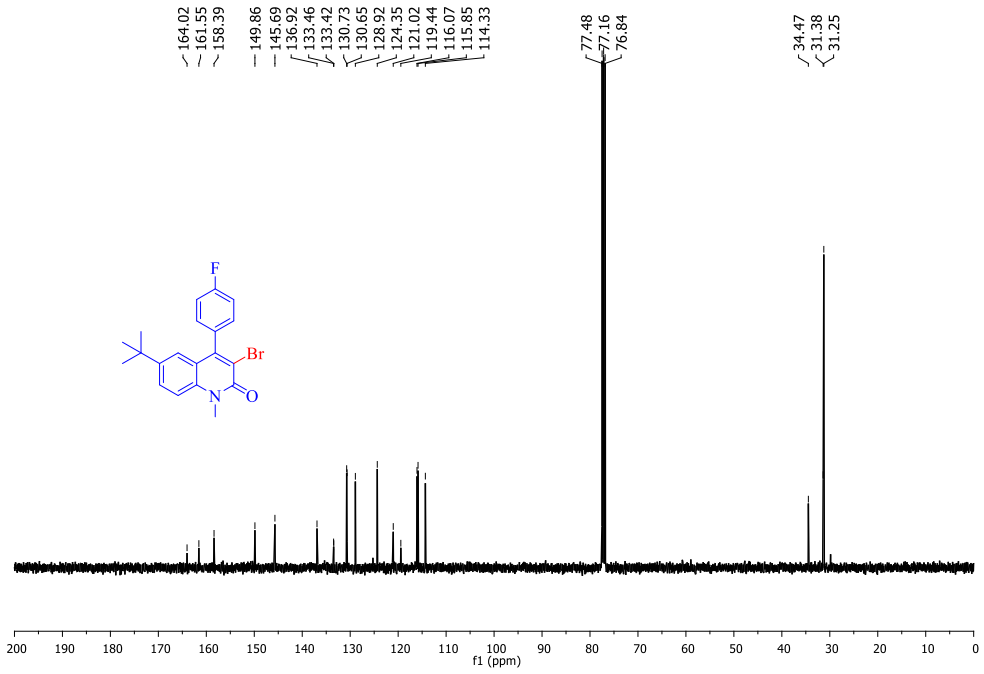


Fig. S28. $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of 3-bromo-6-(*tert*-butyl)-4-(4-fluorophenyl)-1-methylquinolin-2(1H)-one (**2j**)

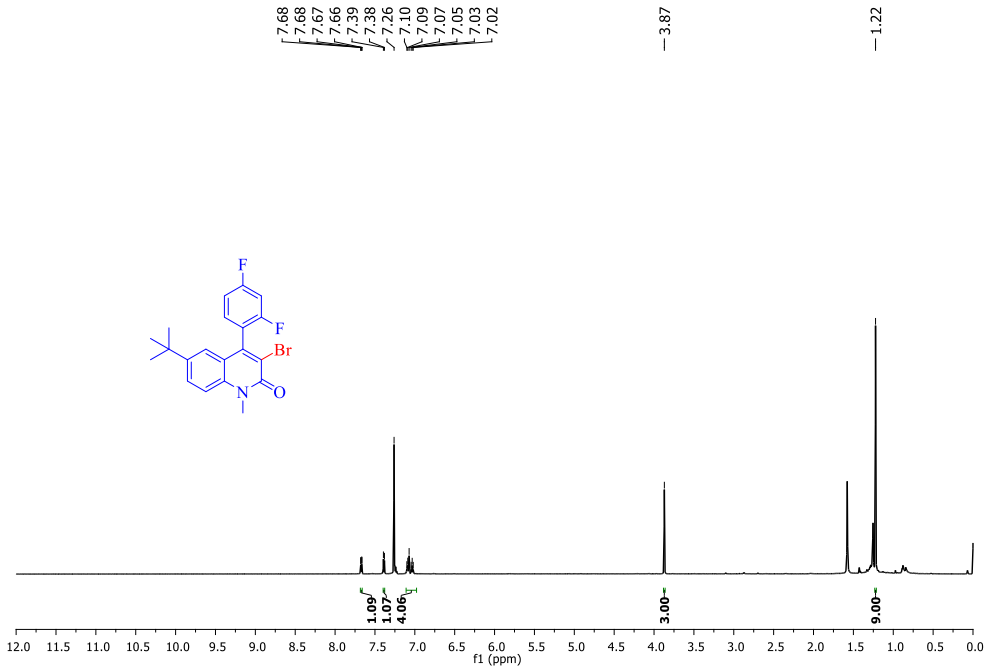


Fig S29. ^{1}H NMR (700 MHz, $CDCl_3$) spectrum of 3-bromo-6-(*tert*-butyl)-4-(2,4-difluorophenyl)-1-methylquinolin-2(1H)-one (**2k**)

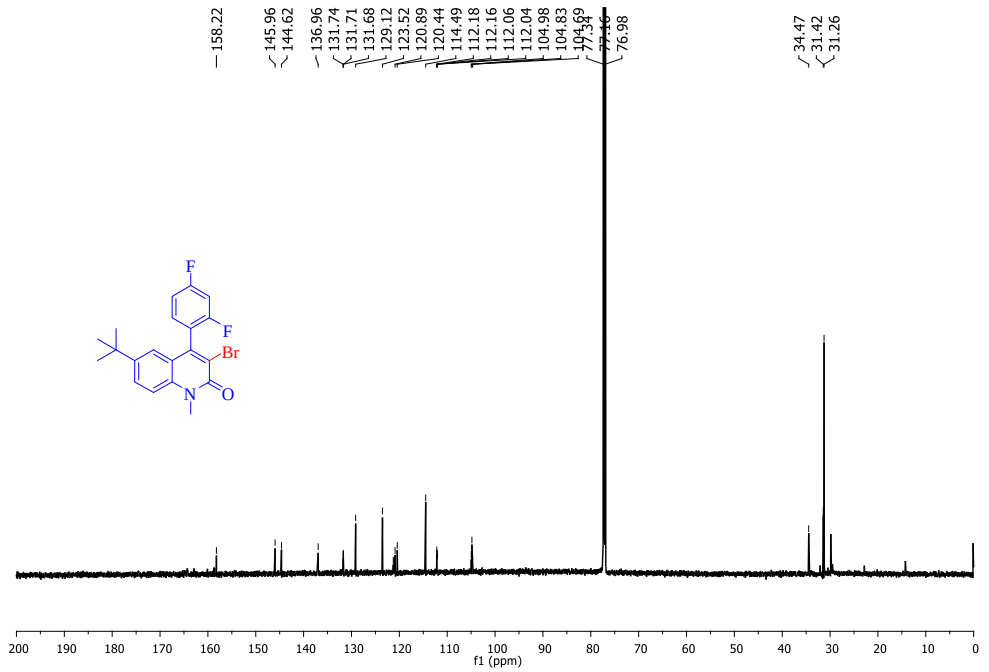


Fig. S30. $^{13}C\{^{1}H\}$ NMR (176 MHz, $CDCl_3$) spectrum of 3-bromo-6-(*tert*-butyl)-4-(2,4-difluorophenyl)-1-methylquinolin-2(1H)-one (**2k**)

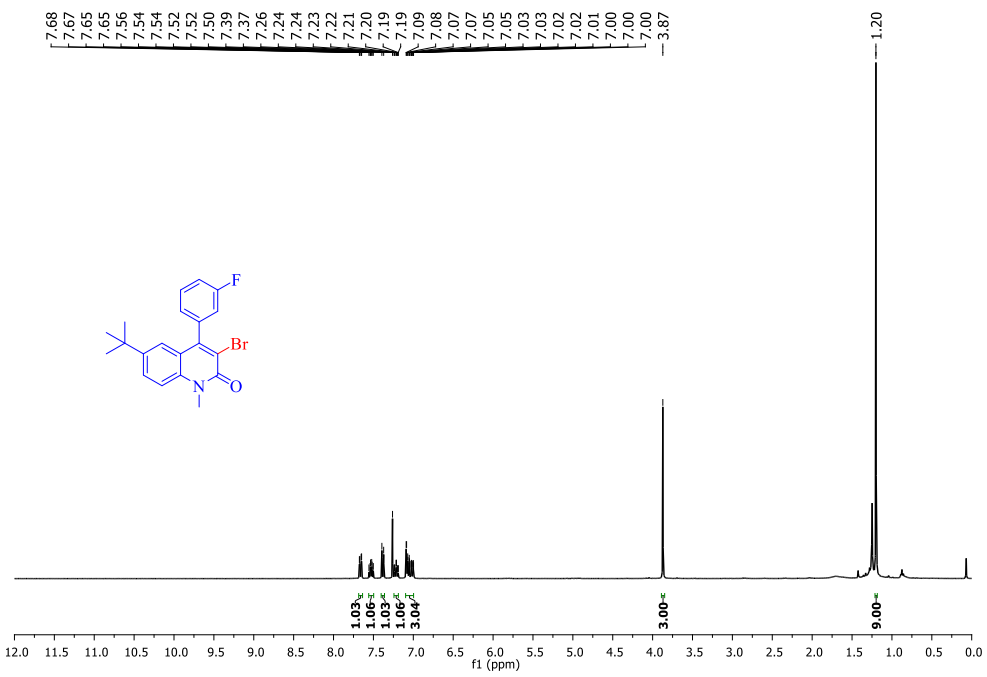


Fig. S31. ^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3-bromo-6-(*tert*-butyl)-4-(3-fluorophenyl)-1-methylquinolin-2(1H)-one (**2l**)

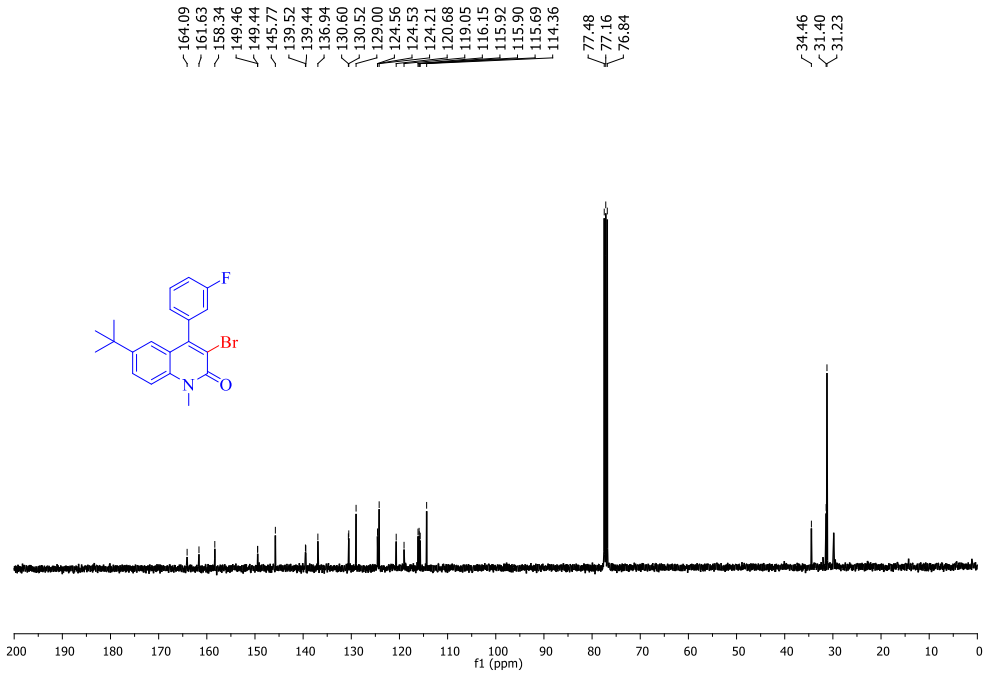


Fig. S32. ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) spectrum of 3-bromo-6-(*tert*-butyl)-4-(3-fluorophenyl)-1-methylquinolin-2(1H)-one (**2l**)

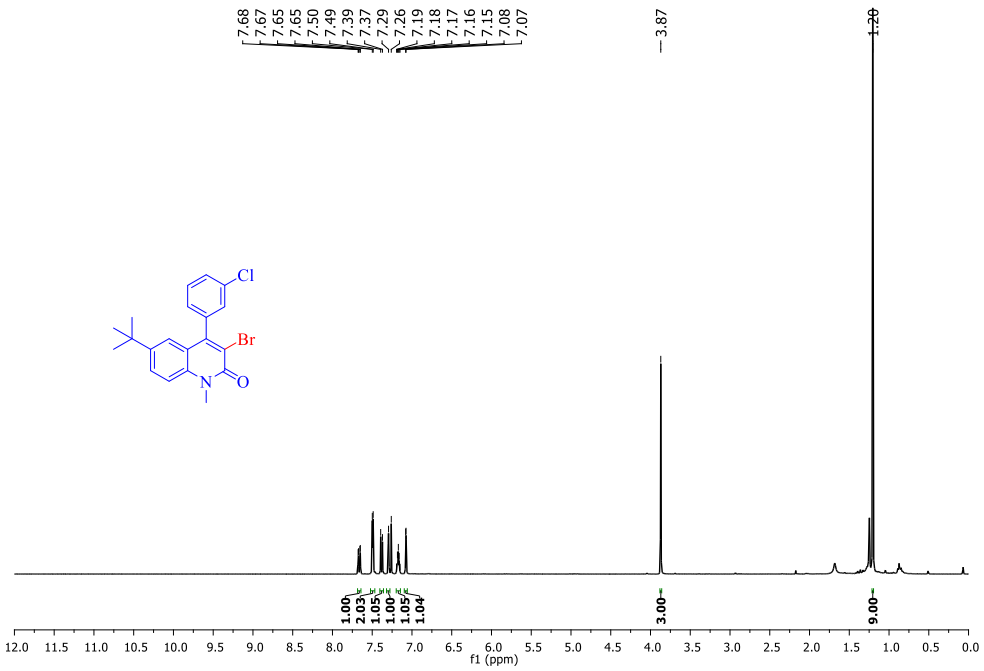


Fig. S33. ^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3-Bromo-6-(*tert*-butyl)-4-(3-chlorophenyl)-1-methylquinolin-2(1H)-one (**2m**)

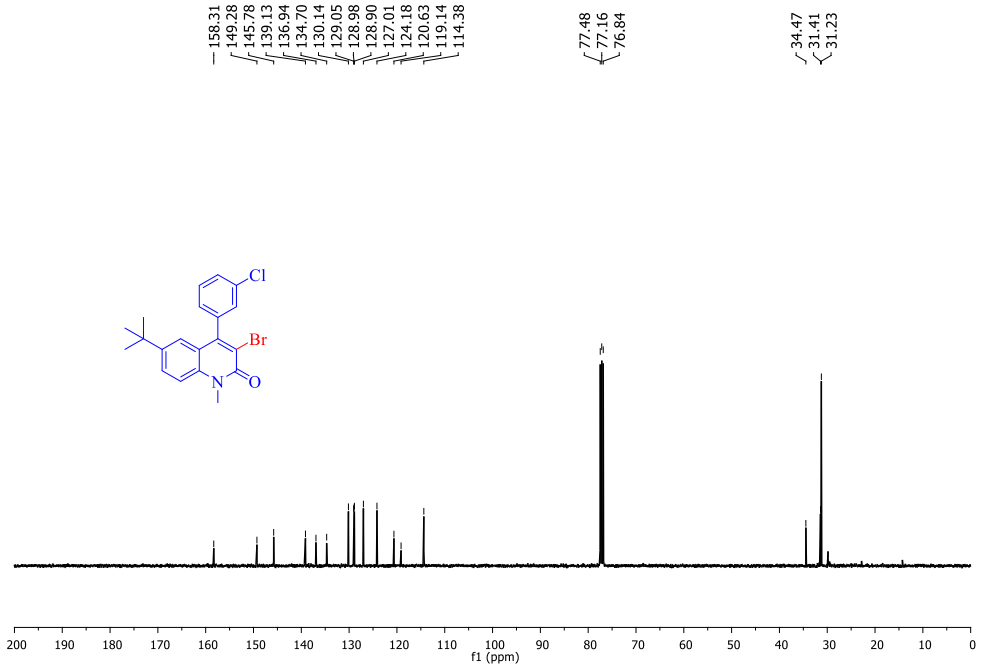


Fig. S34. $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of 3-Bromo-6-(*tert*-butyl)-4-(3-chlorophenyl)-1-methylquinolin-2(1H)-one (**2m**)

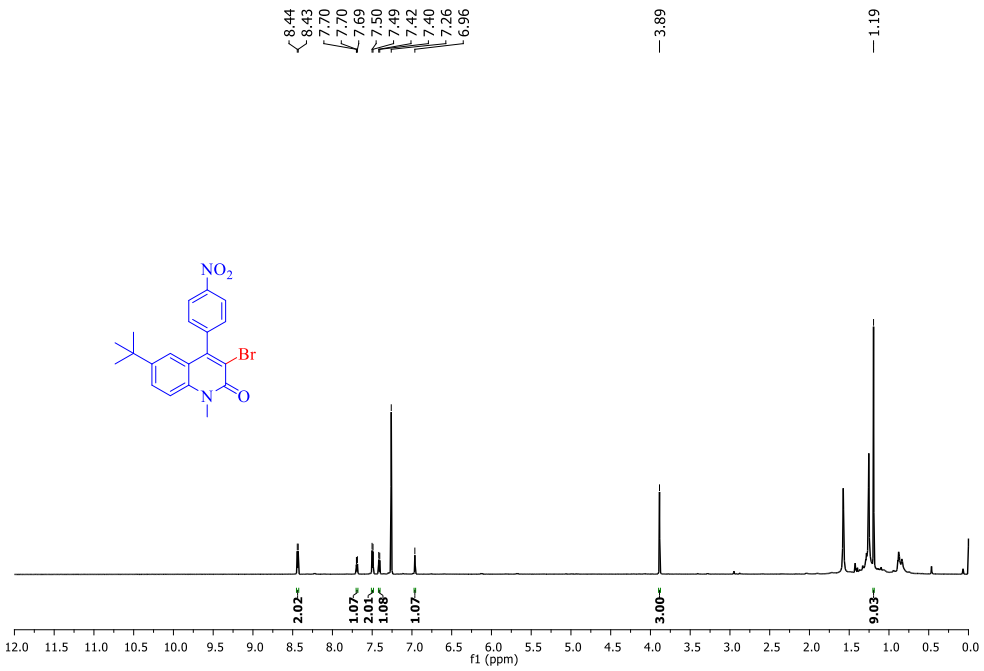


Fig. S35. ^{1}H NMR(700 MHz, $CDCl_3$) spectrum of 3-Bromo-6-(*tert*-butyl)-1-methyl-4-(4-nitrophenyl)quinolin-2(1H)-one (**2n**)

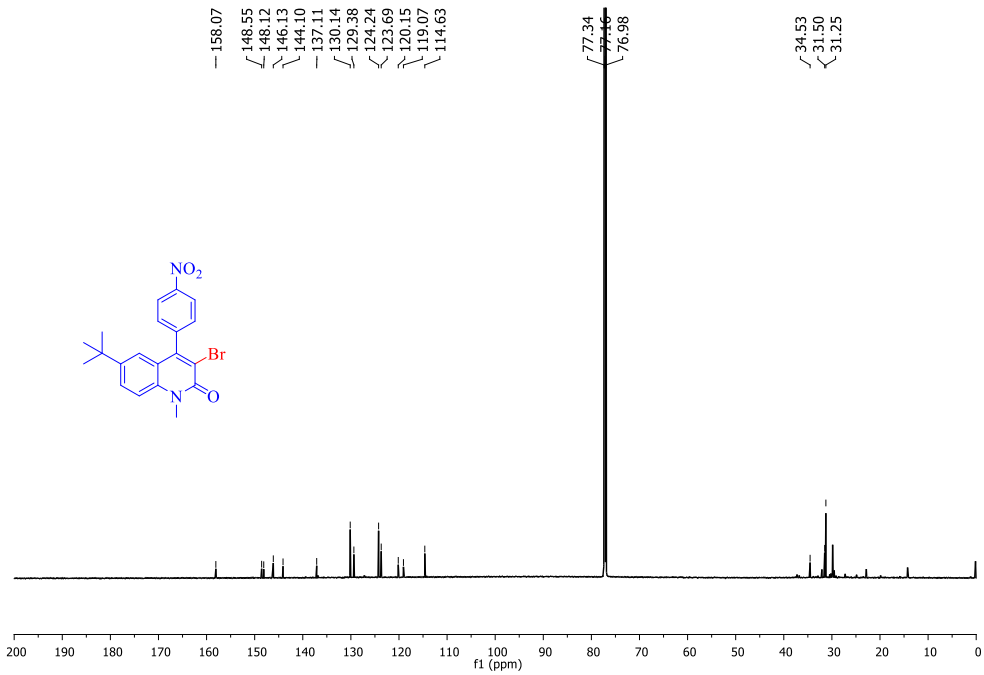


Fig. S36. $^{13}C\{^{1}H\}$ NMR (176 MHz, $CDCl_3$) spectrum of 3-Bromo-6-(*tert*-butyl)-1-methyl-4-(4-nitrophenyl)quinolin-2(1H)-one (**2n**)

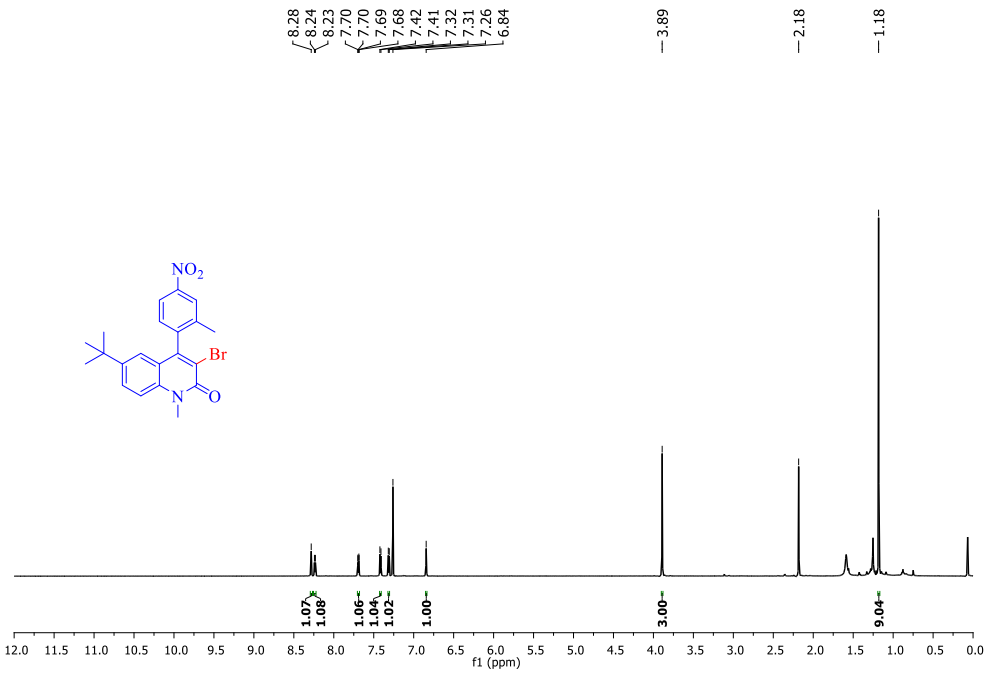


Fig. S37. ^{1}H NMR(700 MHz, $CDCl_3$) spectrum of 3-Bromo-6-(*tert*-butyl)-1-methyl-4-(2-methyl-4-nitrophenyl)quinolin-2(1H)-one (**2o**)

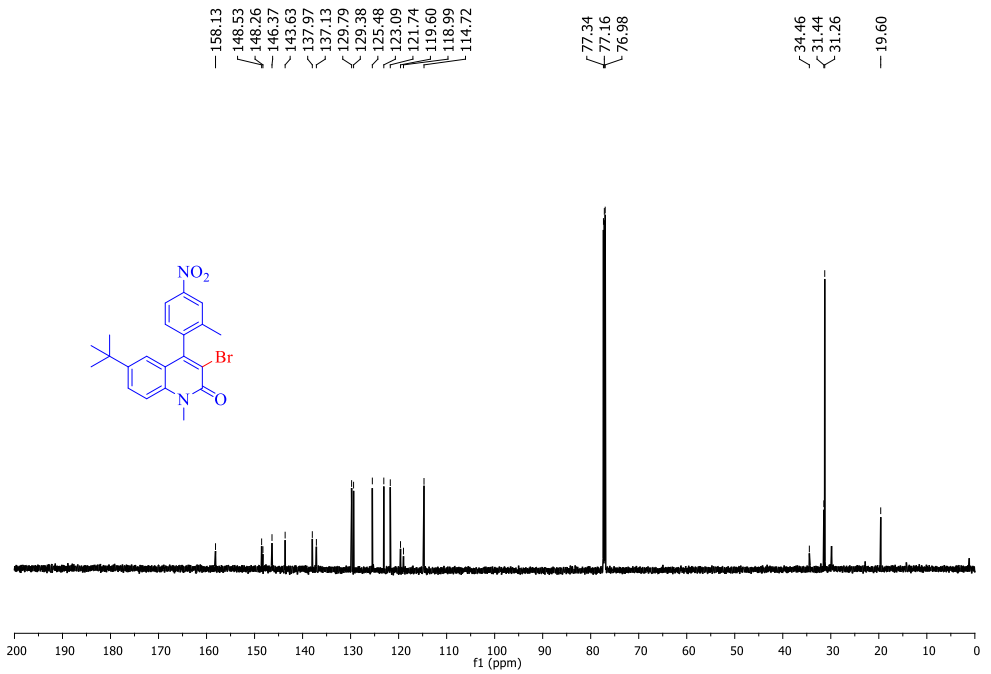


Fig. S38. $^{13}C\{^{1}H\}$ NMR (176 MHz, $CDCl_3$) spectrum of 3-Bromo-6-(*tert*-butyl)-1-methyl-4-(2-methyl-4-nitrophenyl)quinolin-2(1H)-one (**2o**)

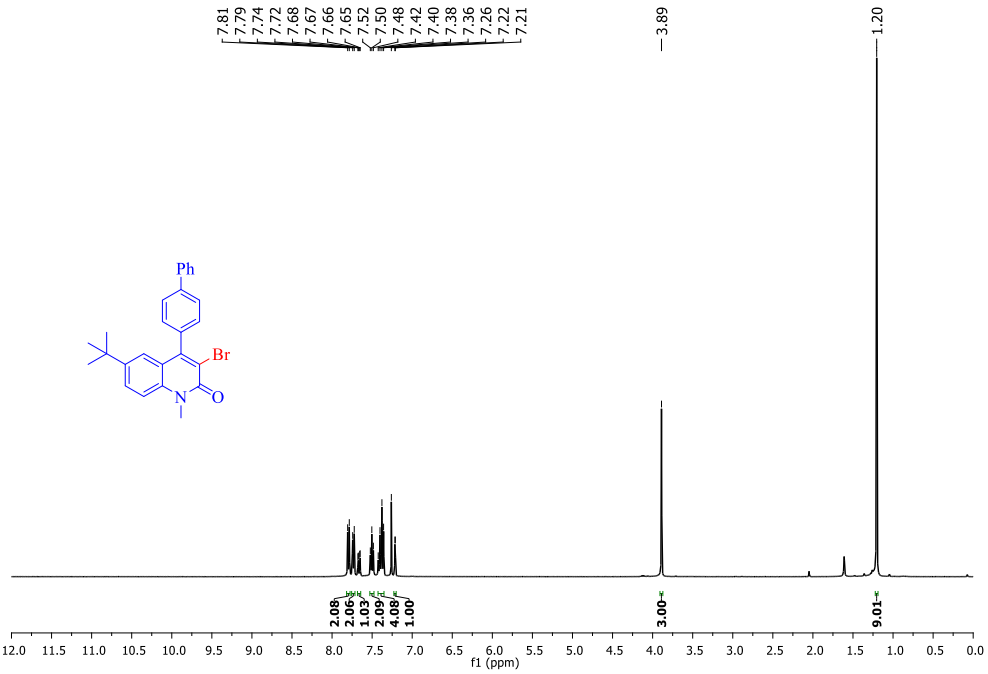


Fig. S39. ^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 4-([1,1'-Biphenyl]-4-yl)-3-bromo-6-(*tert*-butyl)-1-methylquinolin-2(1H)-one (**2p**)

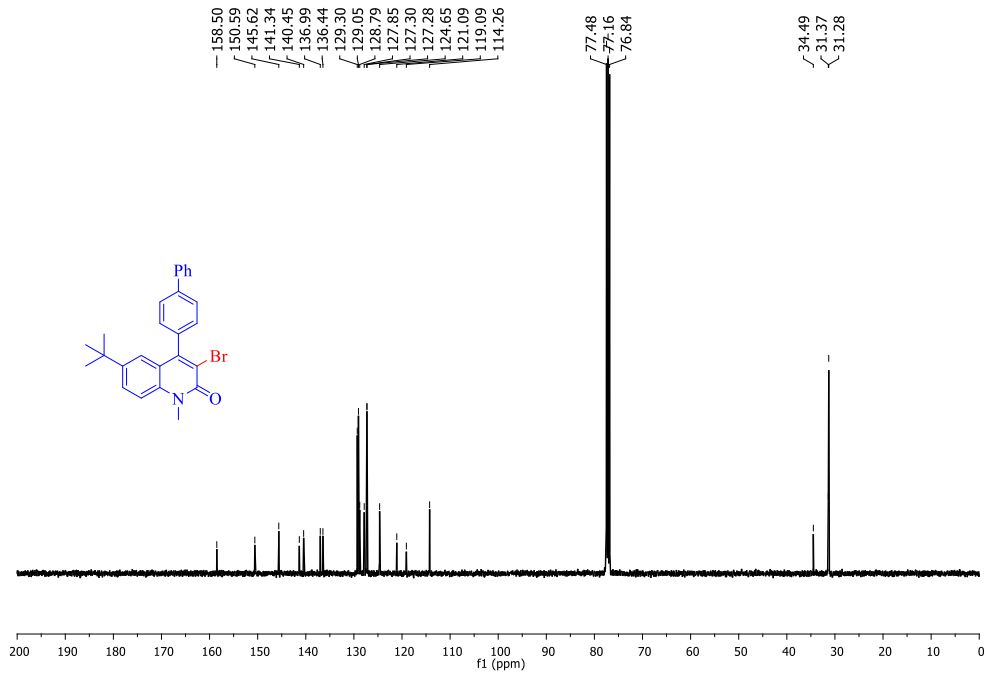


Fig. S40. $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of 4-([1,1'-Biphenyl]-4-yl)-3-bromo-6-(*tert*-butyl)-1-methylquinolin-2(1H)-one (**2p**)

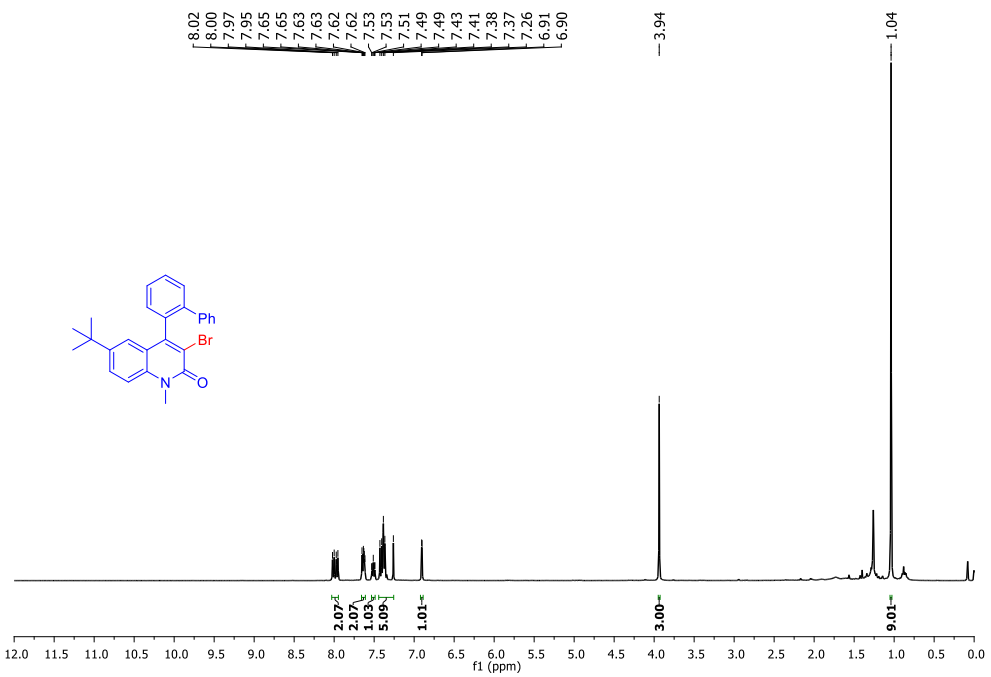


Fig. S41. ^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 4-([1,1'-Biphenyl]-2-yl)-3-bromo-6-(*tert*-butyl)-1-methylquinolin-2(1H)-one (**2q**)

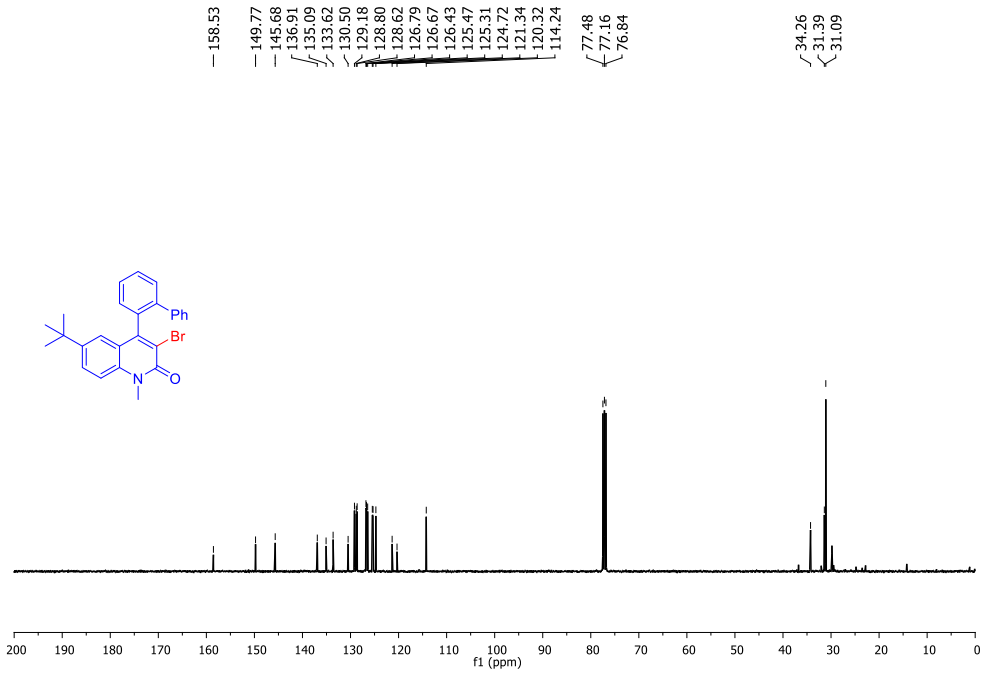


Fig. S42. ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) spectrum of 4-([1,1'-Biphenyl]-2-yl)-3-bromo-6-(*tert*-butyl)-1-methylquinolin-2(1H)-one (**2q**)

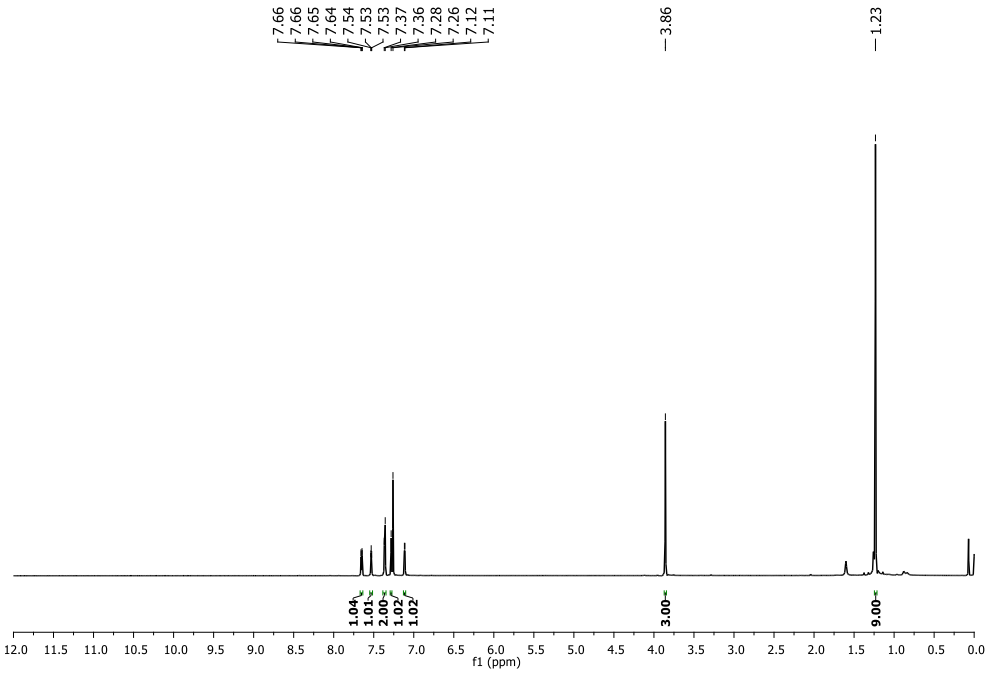


Fig. S43. ^{1}H NMR (700 MHz, $CDCl_3$) spectrum of 3-Bromo-6-(*tert*-butyl)-1-methyl-4-(thiophen-2-yl)quinolin-2(1H)-one (**2r**)

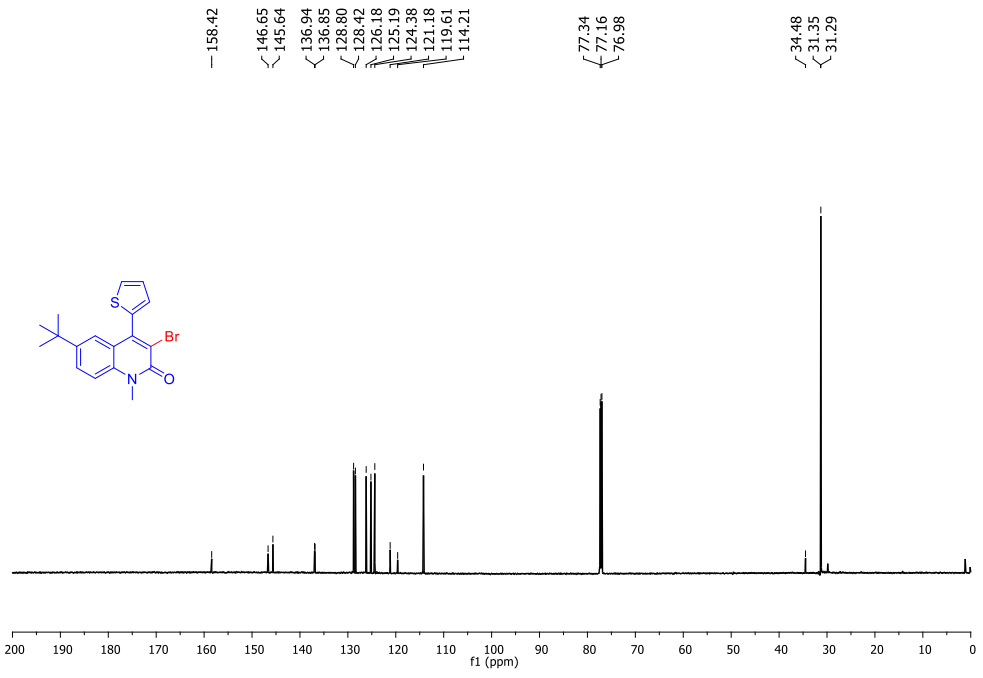


Fig. S44. $^{13}C\{^{1}H\}$ NMR (176 MHz, $CDCl_3$) spectrum of 3-Bromo-6-(*tert*-butyl)-1-methyl-4-(thiophen-2-yl)quinolin-2(1H)-one (**2r**)

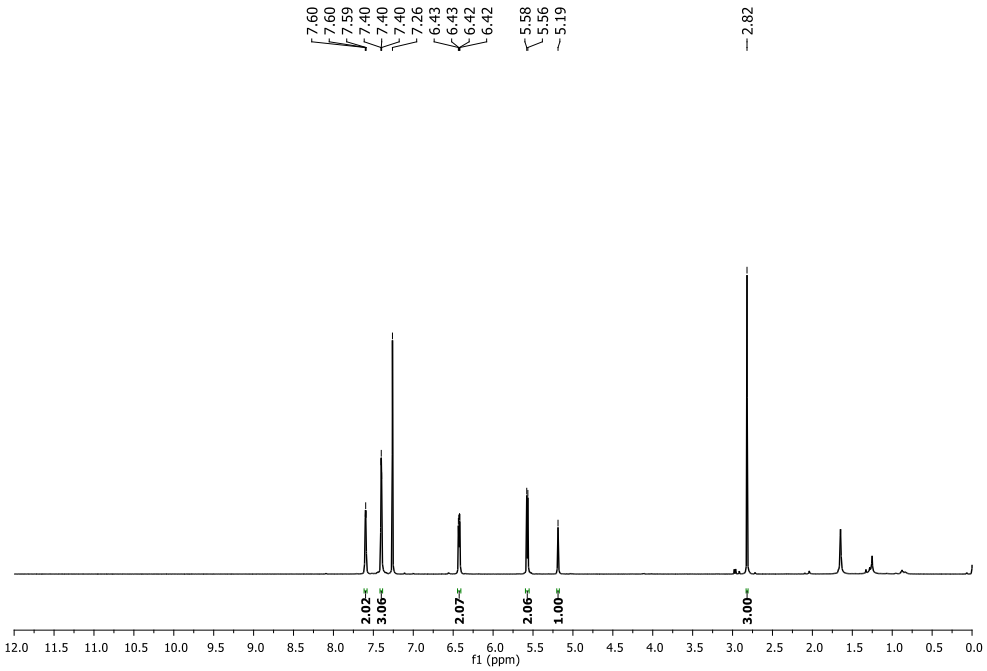


Fig. S45. ^{1}H NMR (700 MHz, $CDCl_3$) spectrum of 3,8-dibromo-1-methyl-4-phenyl-1-azaspiro[4.5]deca-3,6,9-trien-2-one (**4a**)

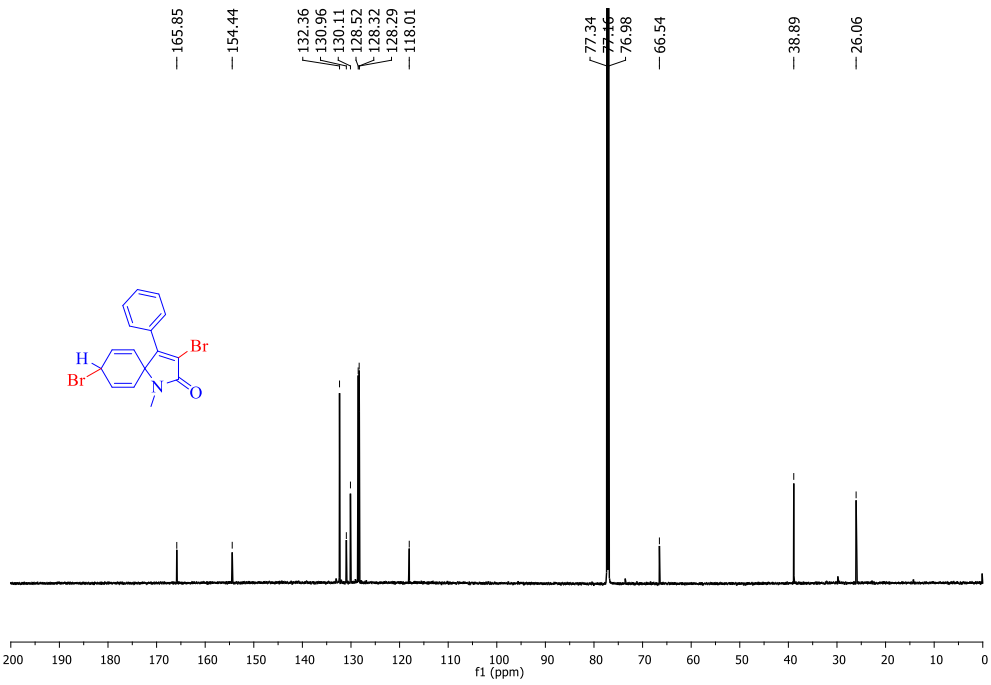


Fig. S46. $^{13}C\{^{1}H\}$ NMR (176 MHz, $CDCl_3$) spectrum of 3,8-dibromo-1-methyl-4-phenyl-1-azaspiro[4.5]deca-3,6,9-trien-2-one (**4a**)

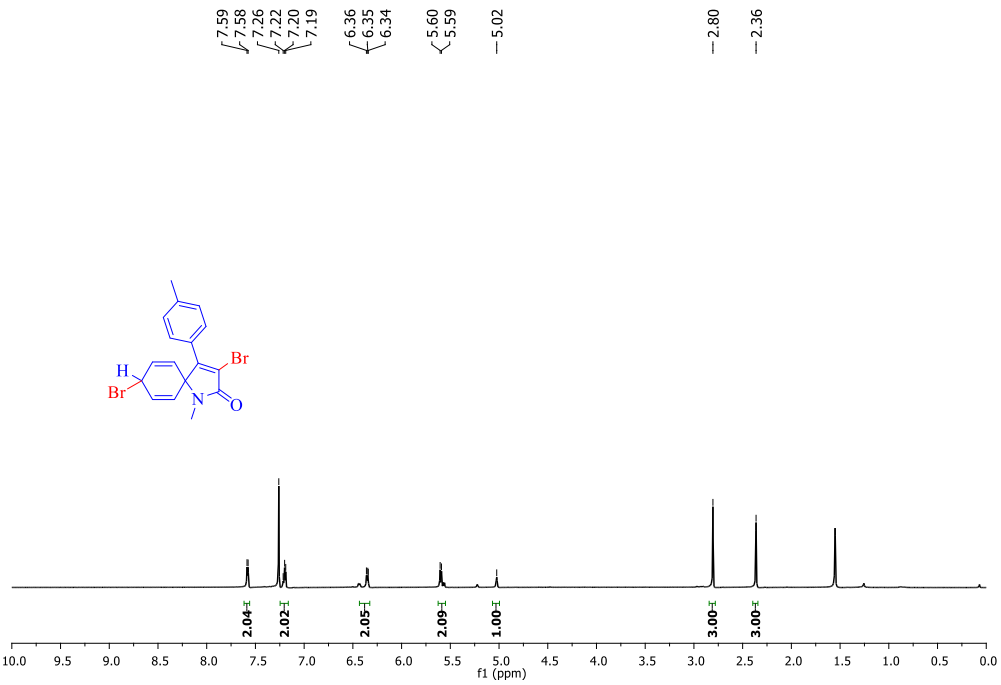


Fig. S47. ^{1}H NMR (700 MHz, $CDCl_3$) spectrum of 3,8-dibromo-1-methyl-4-(p-tolyl)-1-azaspiro[4.5]deca-3,6,9-trien-2-one (**4b**). (Minor diastereomer could not be separated using column chromatography)

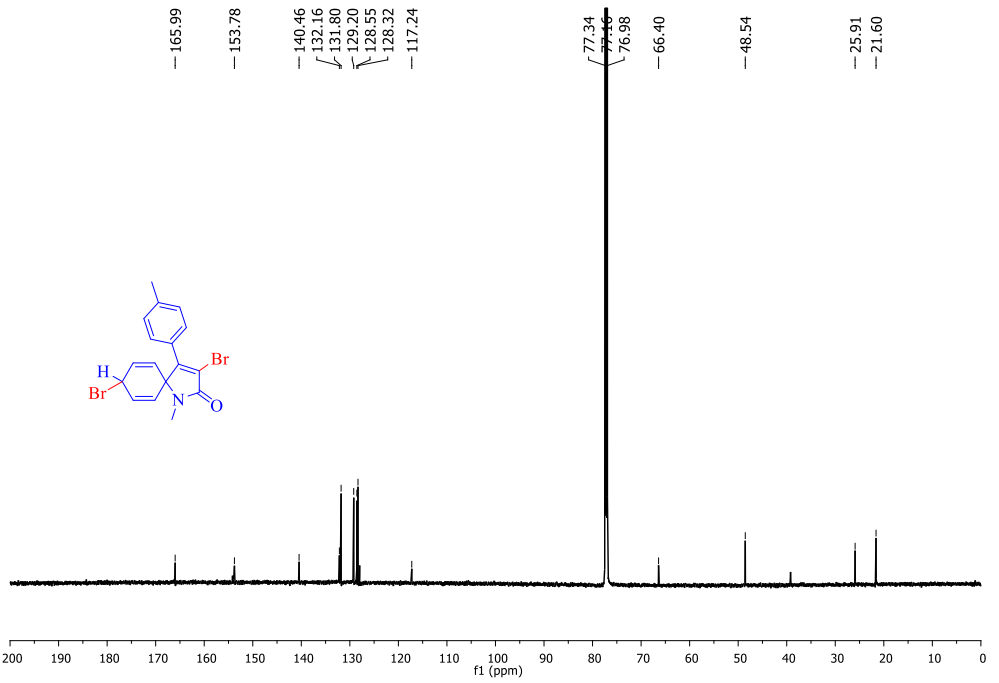


Fig. S48. $^{13}C\{^{1}H\}$ NMR (176 MHz, $CDCl_3$) spectrum of 3,8-dibromo-1-methyl-4-(p-tolyl)-1-azaspiro[4.5]deca-3,6,9-trien-2-one (**4b**)

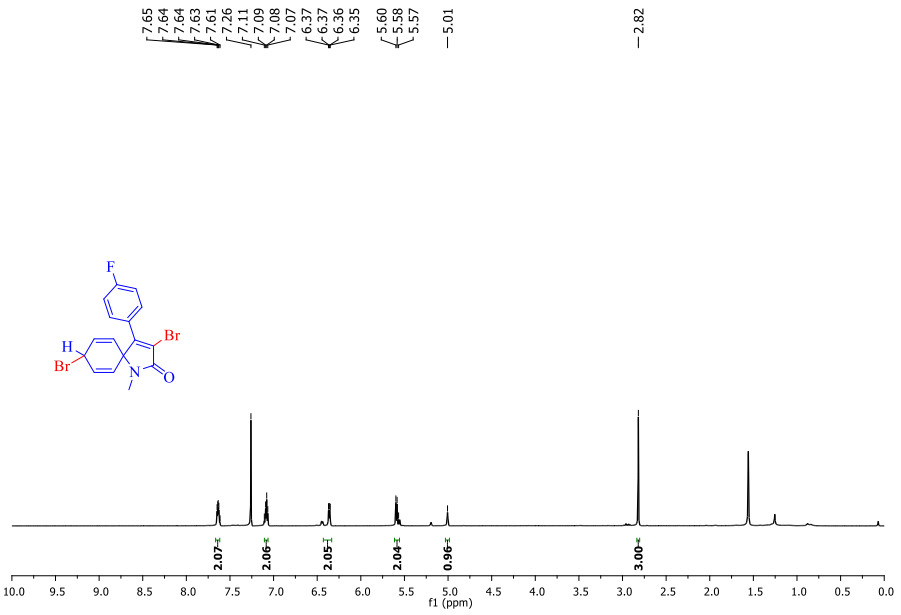


Fig. S49. ^{1}H NMR(700 MHz, $CDCl_3$) spectrum of 3,8-dibromo-4-(4-fluorophenyl)-1-methyl-1-azaspiro[4.5]deca-3,6,9-trien-2-one (**4c**). (Minor diastereomer could not be separated using column chromatography)

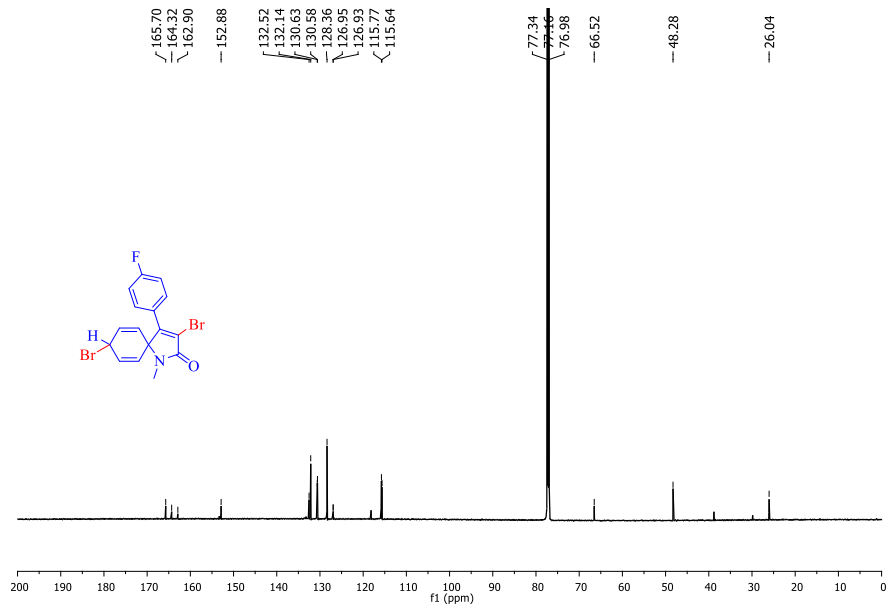


Fig. S50. $^{13}C\{^{1}H\}$ NMR (176 MHz, $CDCl_3$) spectrum of 3,8-dibromo-4-(4-fluorophenyl)-1-methyl-1-azaspiro[4.5]deca-3,6,9-trien-2-one (**4c**).

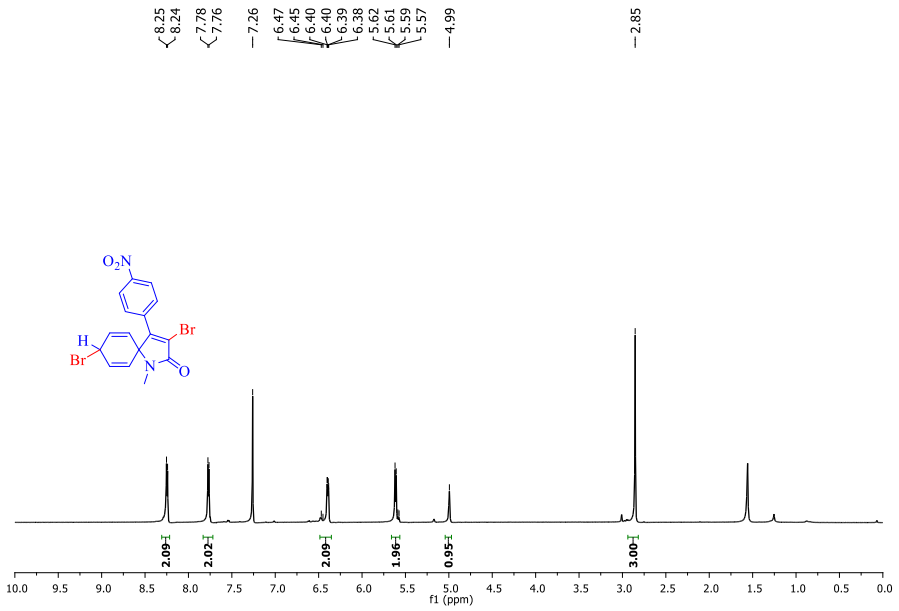


Fig. S51. ^{1}H NMR (700 MHz, $CDCl_3$) spectrum of 3,8-dibromo-1-methyl-4-(4-nitrophenyl)-1-azaspiro[4.5]deca-3,6,9-trien-2-one (**4d**). (Minor diastereomer could not be separated using column chromatography)

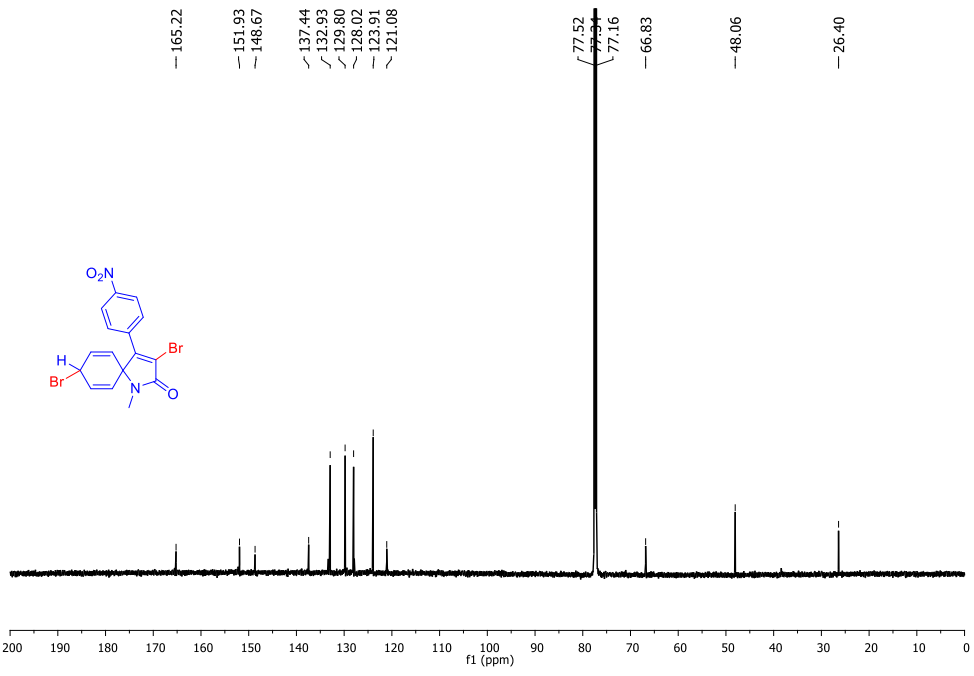


Fig. S52. $^{13}C\{^{1}H\}$ NMR (176 MHz, $CDCl_3$) spectrum of 3,8-dibromo-1-methyl-4-(4-nitrophenyl)-1-azaspiro[4.5]deca-3,6,9-trien-2-one (**4d**).

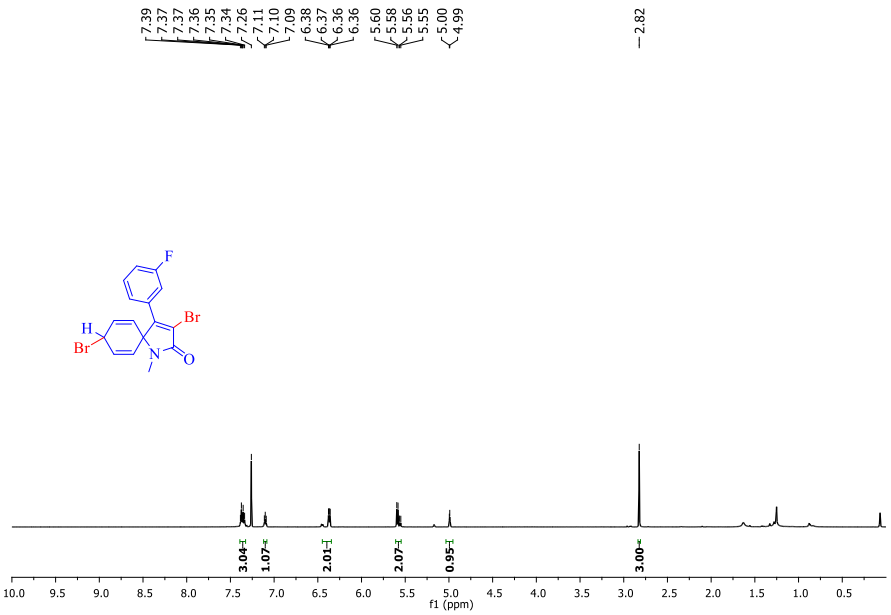


Fig. S53. ^{1}H NMR(700 MHz, $CDCl_3$) spectrum of 3,8-dibromo-4-(3-fluorophenyl)-1-methyl-1-azaspiro[4.5]deca-3,6,9-trien-2-one (**4e**). (Minor diastereomer could not be separated using column chromatography)

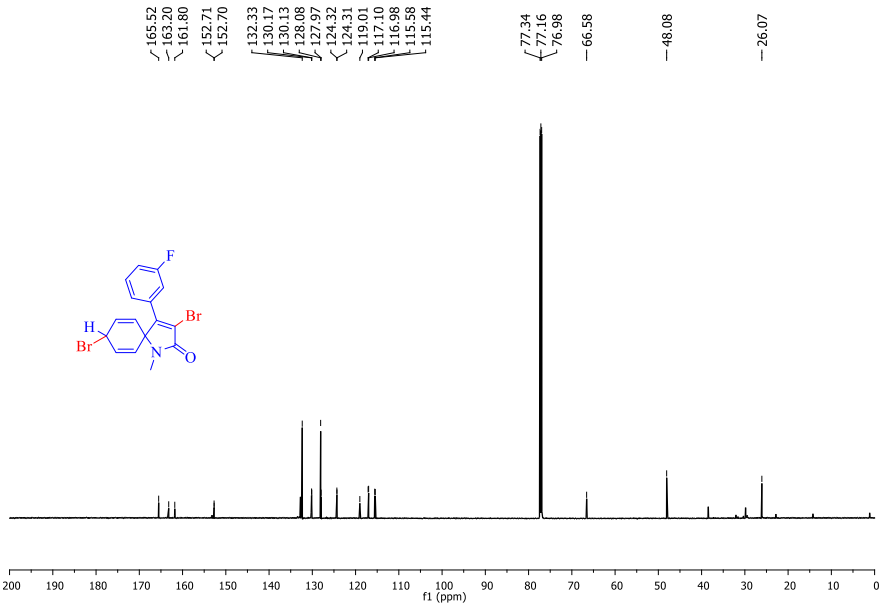


Fig. S54. ^{13}C{^{1}H} NMR (176 MHz, $CDCl_3$) spectrum of 3,8-dibromo-4-(3-fluorophenyl)-1-methyl-1-azaspiro[4.5]deca-3,6,9-trien-2-one (**4e**)

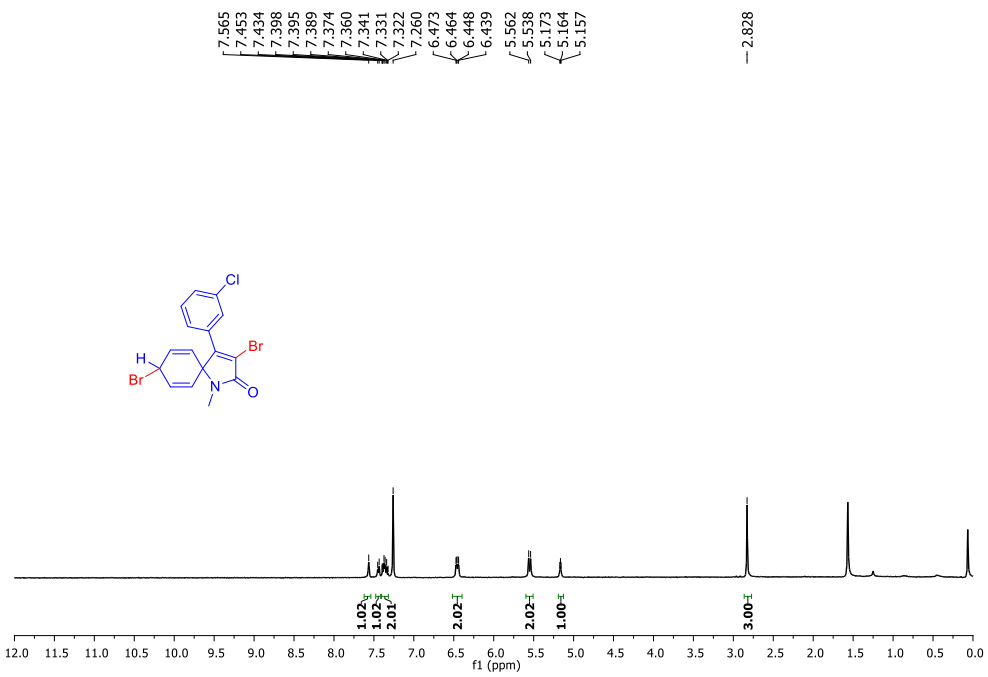


Fig. S55. ^{1}H NMR (700 MHz, $CDCl_3$) spectrum of 3,8-dibromo-4-(3-chlorophenyl)-1-methyl-1-azaspiro[4.5]deca-3,6,9-trien-2-one (**4f**)

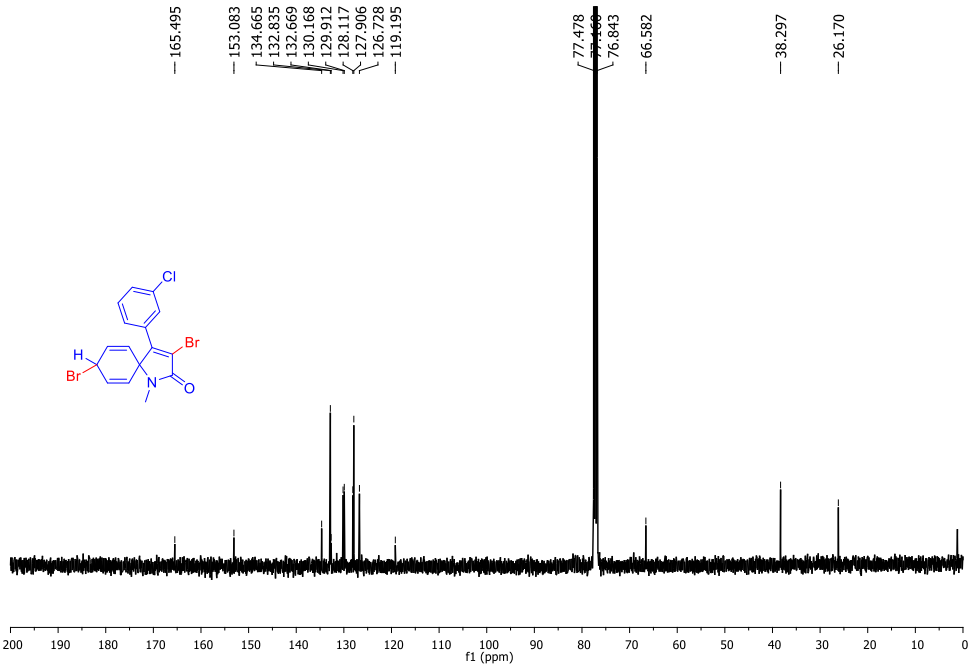


Fig. S56. $^{13}C\{^{1}H\}$ NMR (176 MHz, $CDCl_3$) spectrum of 3,8-dibromo-4-(3-chlorophenyl)-1-methyl-1-azaspiro[4.5]deca-3,6,9-trien-2-one (**4f**)

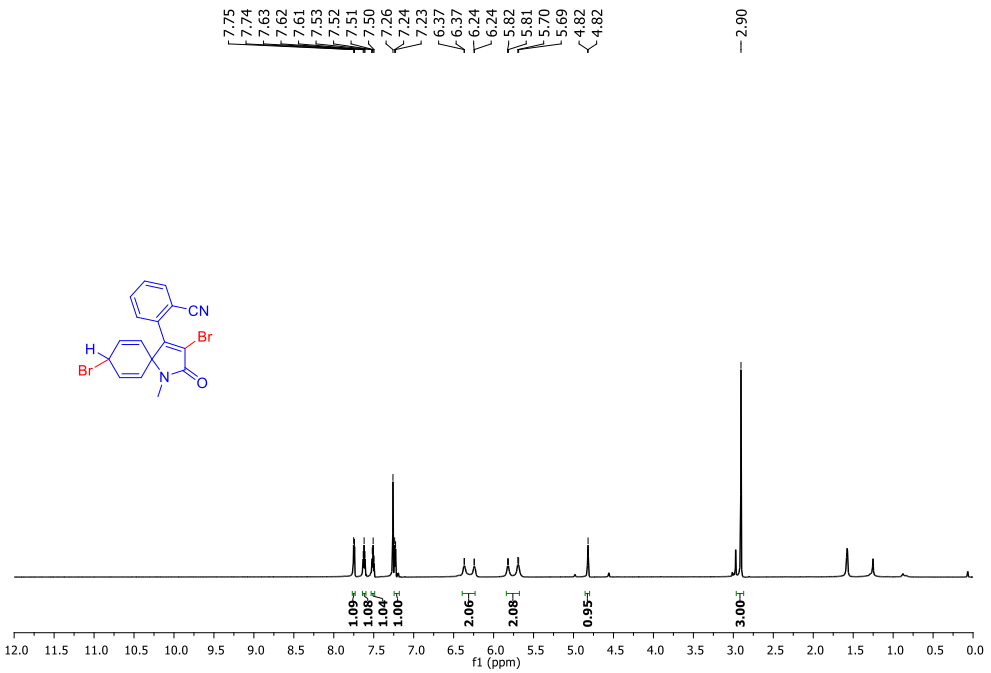


Fig. S57. ^{1}H NMR (700 MHz, $CDCl_3$) spectrum of 2-(3,8-dibromo-1-methyl-2-oxo-1-azaspiro[4.5]deca-3,6,9-trien-4-yl)benzonitrile (**4g**). (Minor diastereomer could not be separated using column chromatography)

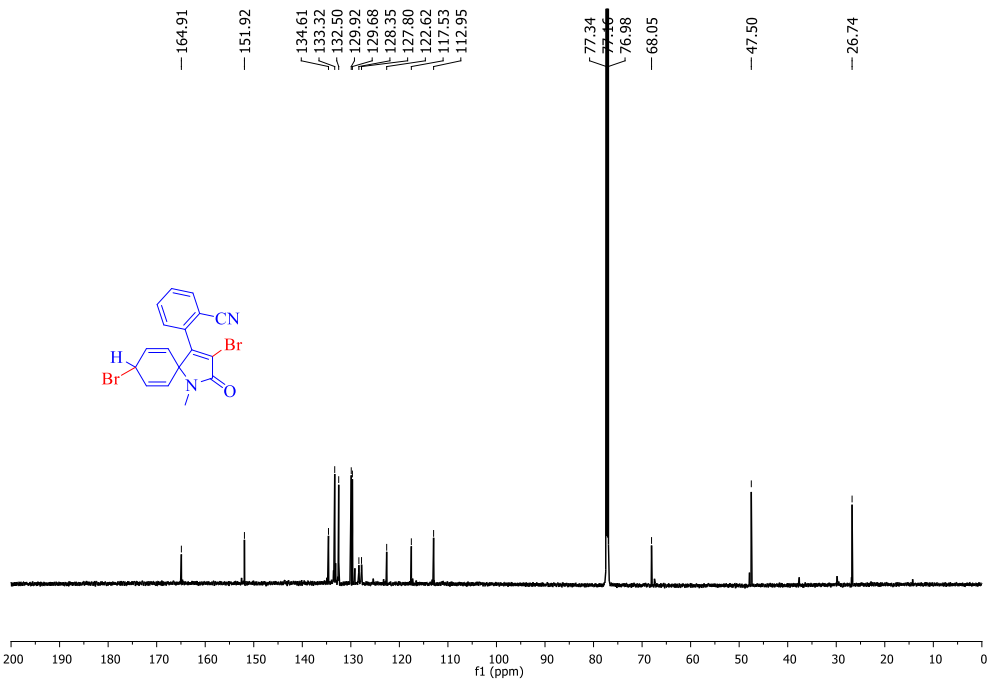


Fig. S58. $^{13}C\{^{1}H\}$ NMR (176 MHz, $CDCl_3$) spectrum of 2-(3,8-dibromo-1-methyl-2-oxo-1-azaspiro[4.5]deca-3,6,9-trien-4-yl)benzonitrile (**4g**)

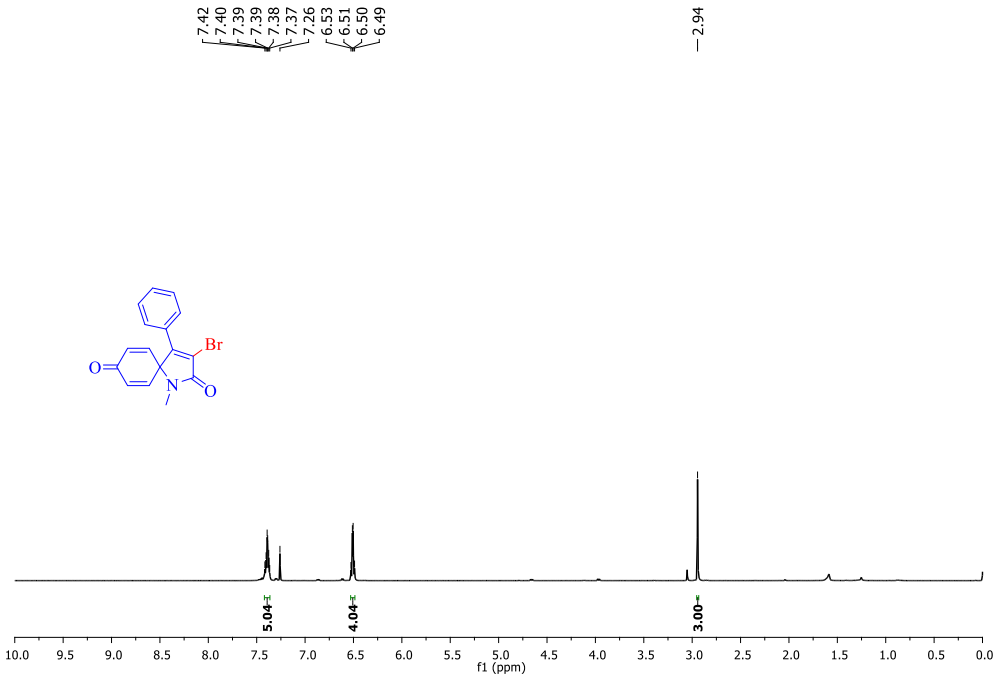


Fig. S59. ^{1}H NMR (700 MHz, $CDCl_3$) spectrum of 3-bromo-1-methyl-4-phenyl-1-azaspiro[4.5]deca-3,6,9-triene-2,8-dione (**4j**)

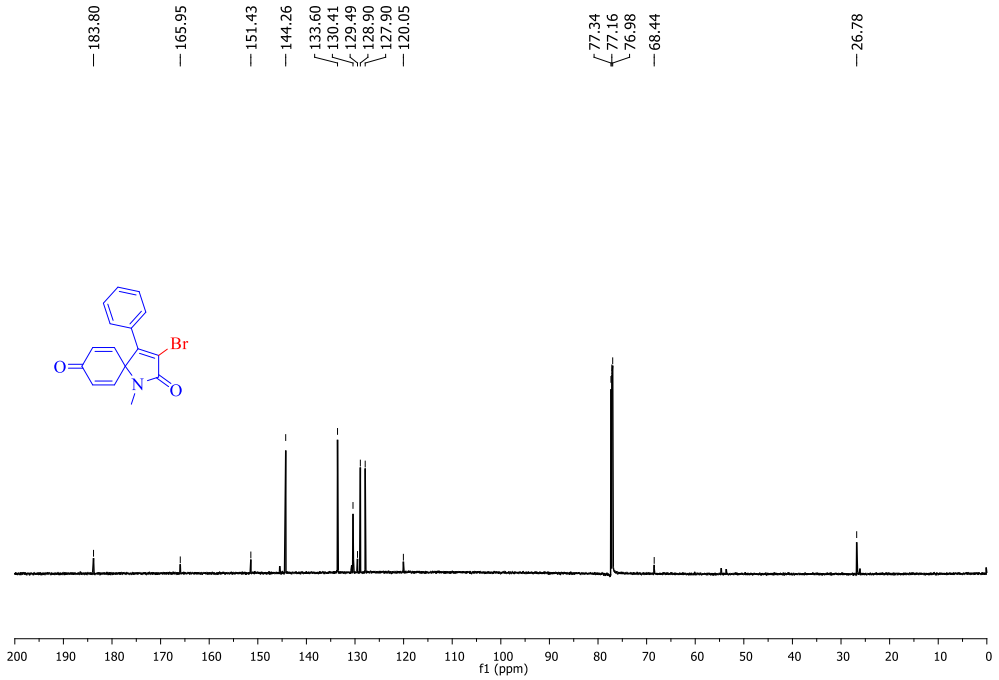


Fig. S60. $^{13}C\{^{1}H\}$ NMR (176 MHz, $CDCl_3$) spectrum of 3-bromo-1-methyl-4-phenyl-1-azaspiro[4.5]deca-3,6,9-triene-2,8-dione (**4j**)

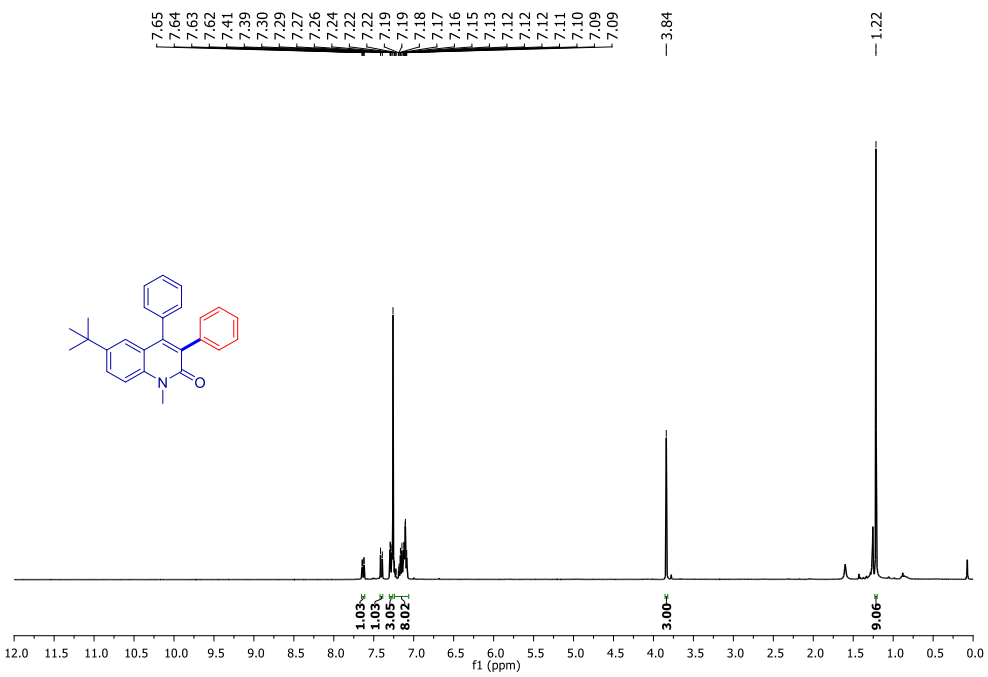


Fig. S61. ^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 6-(*tert*-butyl)-1-methyl-3,4-diphenylquinolin-2(1H)-one (**5**).

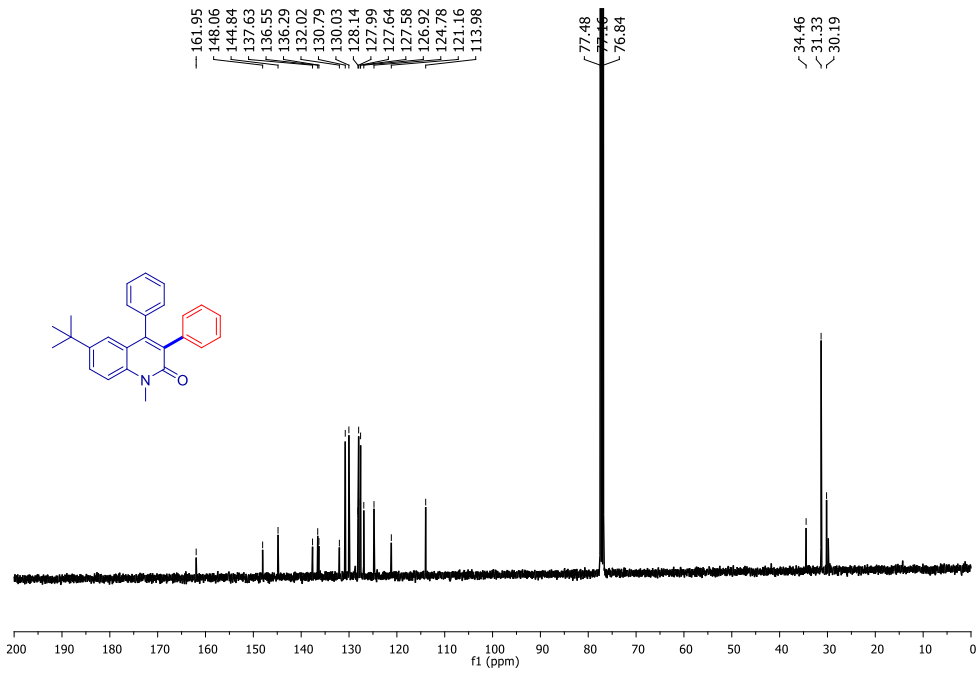


Fig. S62. $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of 6-(*tert*-butyl)-1-methyl-3,4-diphenylquinolin-2(1H)-one (**5**)

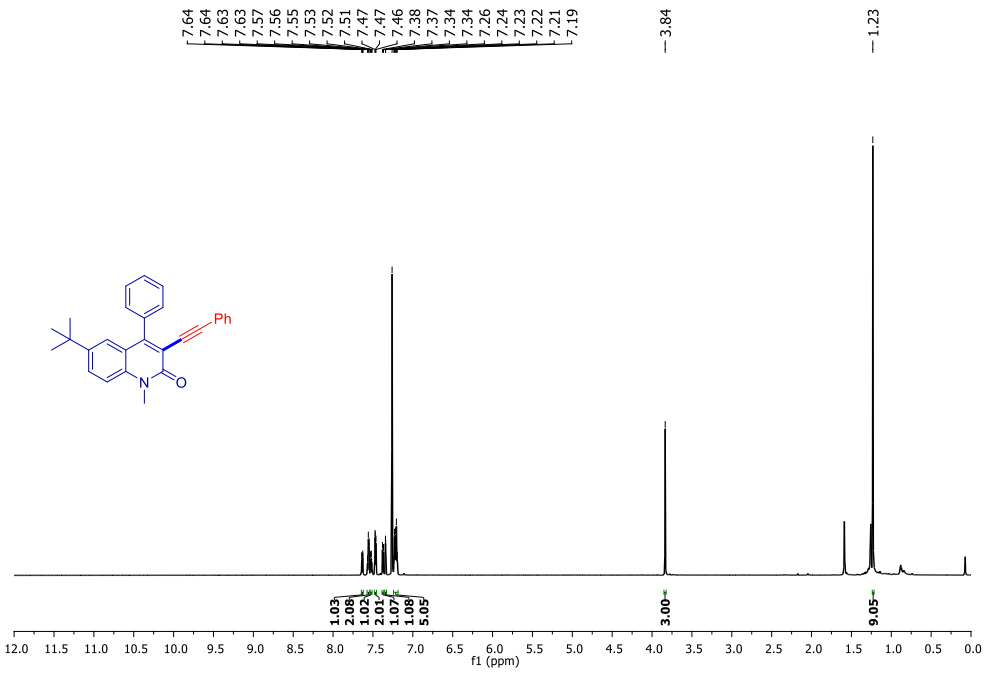


Fig. S63. ^{1}H NMR(700 MHz, $CDCl_3$) spectrum of 6-(*tert*-butyl)-1-methyl-4-phenyl-3-(phenylethynyl)quinolin-2(1H)-one (**6**)

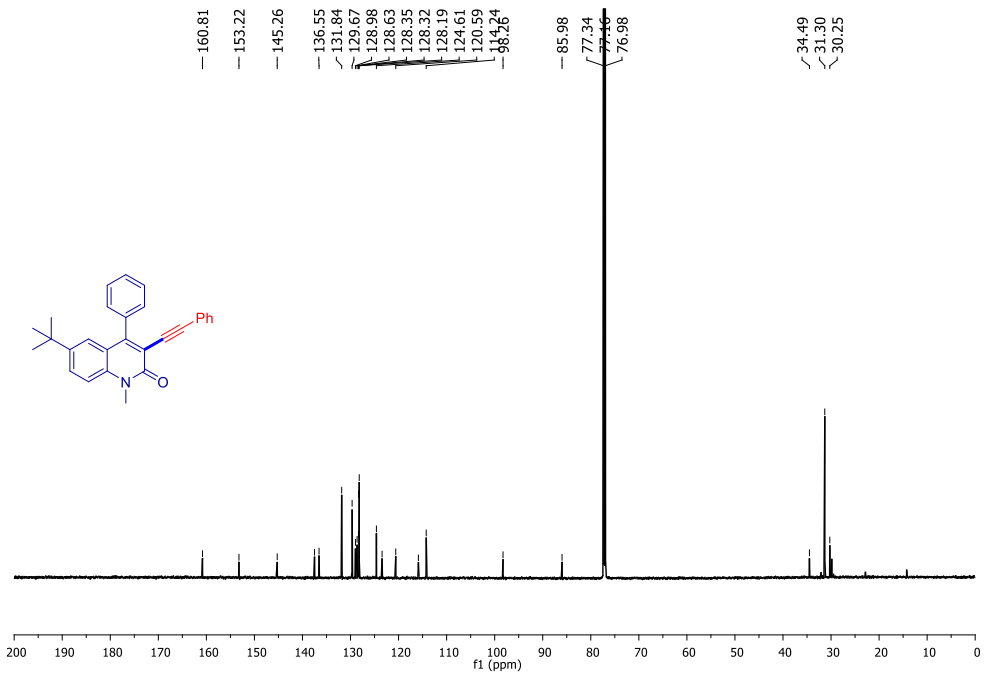
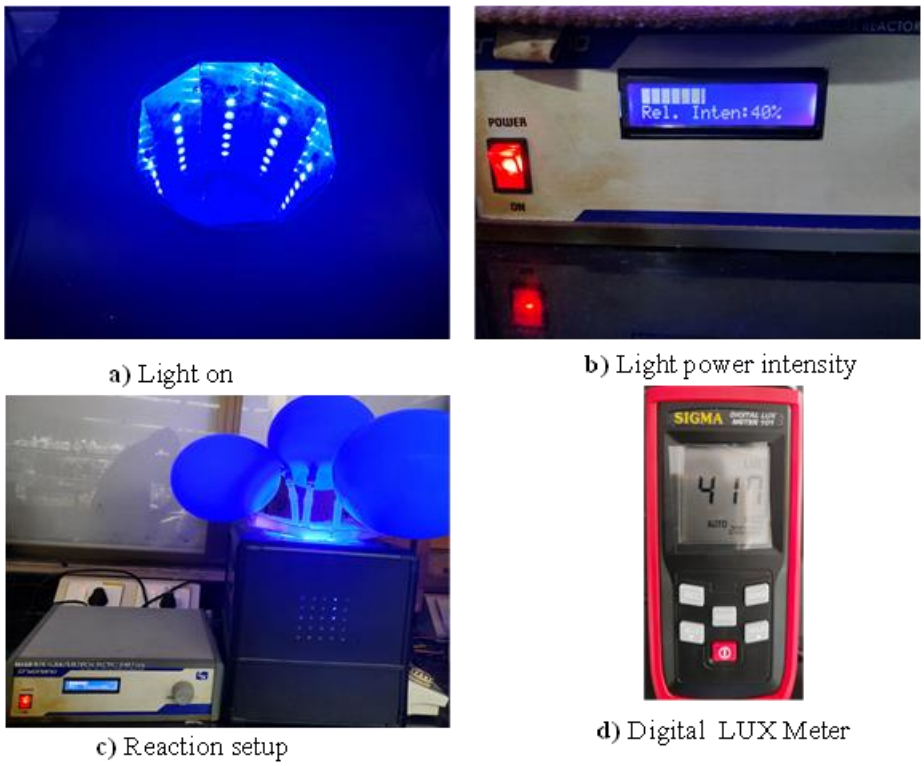


Fig. S64. $^{13}C\{^{1}H\}$ NMR (176 MHz, $CDCl_3$) spectrum of 6-(*tert*-butyl)-1-methyl-4-phenyl-3-(phenylethynyl)quinolin-2(1H)-one (**6**)

Photoreactor. This photoreactor used obtained from commercial source (Model No.-LED Photochemical reactor CN302, CRYOANO VL- PHOTON). Quartz tubes (LUZCHEM) The intensity of the blue LED is (417 × 100) lx (measured by Sigma-Digital Lux Meter 101, Model: 20036176). Distance between quartz tube and light source was approximately 4.2 cm and no filter was used.



a) Light on

b) Light power intensity

c) Reaction setup

d) Digital LUX Meter

Fig. S65. The photoreactor used for the present study.

CRYONANO Labs LED Photochemical Reactor - CNPHOTON 101

The CN-Photon LED Photochemical Reactor from CRYONANO Labs is a compact desktop instrument for conducting research in areas of Photo-biology, Inorganic, Organometallic and Organic Photochemistry (e.g., Drug-DNA Interaction) etc. It has a ventilated illumination chamber with tunable high intensity LEDs and fully automatic operation with countdown timer for setting the reaction time and switching it off automatically. The intensity of light can also be automatically controlled using inbuilt microprocessors.

The reactor includes a controller in a separate housing for light intensity control and automation with display. It also comes with a carousel for liquid samples.

Main Features of the reactor are:

- High flux per LED
- Blue led - 2100 lumens
- White led - 10000 lumens
- Good color uniformity
- Industry best moisture sensitivity level
- JEDEC Level 1
- Low Voltage DC operated
- Instant light (less than 100ns)
- No UV Component
- Dimensions: Internal : 5.5” Diameter, 7” height, Anodized aluminium enclosure
- Power Rating: 220 V AC, 50 Hz, 2Amp