Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry. This journal is © The Royal Society of Chemistry 2023

## **Supporting Information**

# Photoredox Radical Cascade Reaction of *o*-Vinylaryl Isocyanides with Acyl Chlorides to Access 2,4-Disubstituted Quinolines

Peng-Fei Huang,\* Jia-Le Fu, Jia-Jing Huang, Bi-Quan Xiong, Ke-Wen Tang, Yu Liu\*

Department of Chemistry and Chemical Engineering, Hunan Institute of Science and

Technology, Yueyang 414006, China

12021027@hnist.edu.cn (Peng-Fei Huang) and lyxtmj\_613@163.com (Yu Liu)

### **List of Contents**

1.	General Information	<b>S1</b>
2.	Experimental Section	S1-S11
	2.1 General Procedure for the Synthesis of Substrates	<b>S1-S1</b>
	2.2 Details of Visible-Light Source	S1-S2
	2.3 Control Experiments	S2-S11
	2.4 The Light on/off Experiments	S11-S12
3.	Reference	S12
4.	Spectra	<b>S13-S87</b>

#### 1. General Information

Unless otherwise stated, all commercial reagents were used as received. >98%). Propiophenone, aldehydes (Innochem, o-Phenylenediamine and Phenylhydrazine hydrochloride were used without further treatment. All reagents and solvents were commercially available and used without any further purification unless specified. All solvents were dried and distilled according to standard procedures. Flash column chromatography was performed using silica gel (0.25mm, 300-400 mesh). Analytical thin-layer chromatography was performed using glass plates pre-coated with 0.25mm 300-400 mesh silica gel impregnated with a fluorescent indicator (254 nm). All reactions were carried out with magnetic stirring and in dried glassware. Nuclear magnetic resonance (NMR) spectra are recorded in parts per million from internal tetramethylsilane on the  $\delta$  scale. <sup>1</sup>H NMR, <sup>19</sup>F NMR and <sup>13</sup>C NMR spectra were recorded in CDCl<sub>3</sub> on a Bruker DRX-400 spectrometer operating at 400 MHz, 282 MHz and 100 MHz, respectively. All chemical shift values are quoted in ppm and coupling constants quated in Hz. The solvent peak was used as a reference value, for <sup>1</sup>H NMR: TMS = 0.00 ppm, for <sup>13</sup>C NMR: CDCl<sub>3</sub> = 77.00 ppm. The following abbreviations were used to explain multiplicities: s = singlet, d =doublet, dd = doublet of doublet, t = triplet, td = triplet of doublet, q = quartet, m =multiplet, and br = broad. High-resolution mass spectra (HRMS) were obtained on an Agilent mass spectrometer using ESI-TOF (electrospray ionization-time of flight).

#### 2. Experiment Section

#### 2.1 General Procedure for the Synthesis of Substrates

1-Isocyano-2-(1-phenylvinyl)benzene 1 was synthesized according to the known methods<sup>[1]</sup>.

#### 2.2 Details of Visible-Light Source

ThelightsourceboughtfromSANYI(https://item.taobao.com/item.htm?spm=a1z09.2.0.0.42672e8dv2Chsz&id=35497290577&\_u=j35sh1qt9325), 5 W blue LED light bulb (E27). The wavelength was about

460-470 nm and the wavelength of peak intensity was about 467.5 nm. The picture of the experimental facility was shown as follow:



**Figure S1**. Pictures of Visible-Light Source. Reproduced from [Liu, Y.; Wang, Q.-L.; Chen, Z.; Zhou, Q.; Li, H.; Zhou, C.-S.; Xiong, B.-Q.; Zhang, P.-L.; and Tang, K.-W.; Visible-Light-Catalyzed C–C Bond Difunctionalization of Methylenecyclopropanes with Sulfonyl Chlorides for the Synthesis of 3-Sulfonyl-1,2-dihydronaphthalenes, *J. Org. Chem.* **2019**, *84*, 2829-2839]. Copyright [2019] American Chemical Society

#### **2.3 Control Experiments**

2.3.1 GC-MS Analysis of Raw Reaction Mixture by Using TEMPO as Radical Inhibitor



To a Schlenk tube were added 1-isocyano-2-(1-phenylvinyl)benzene **1a** (0.2 mmol), benzoyl chloride **2a** (6 mmol, 3 equiv),  $Ir(ppy)_3$  (1 mol%),2,6-lutidine (0.8 mmol, 4 equiv.), TEMPO (3 equiv), CH<sub>3</sub>CN (2 mL) at room temperature and 5 W blue LED irradiation for 12 h. The GC-MS analysis of raw reaction mixture showed that only trace amounts of the target product **3aa** was detected. The acyl radical trapping product **4**, which generated from TEMPO capturing the acyl radical, could be detected by GC-Ms analysis.

The GC-MS analysis results of raw reaction mixture The GC spectra of raw reaction mixture



The MS spectra of the peak at 10.53 min



[MS Spec	trum]		60.00	31847	0.38	81.05	944251	11.26
# of Peaks	\$546		61.00	26870.03		82.10	802929	9.58
Raw Spec	trum 10.5	40 (scan :	62.00	72790.09		83.05	6841848	81.60
1309)			62.95	11286	0.13	84.05	901507	10.75
Backgrou	nd No		64.05	64100.08		85.05	72256	0.86
Backgrou	nd Spectru	ım	65.00	69833	0.83	86.05	62159	0.74
Base Peak	m/z 104.9	95 (Inten :	66.05	30959	0.37	87.05	63400.08	
8,384,767)			67.00	416017	4.96	88.05	40669	0.49
Event#	1		68.05	276471	3.30	89.05	35860.04	
m/z Abso	olute Inten	sity	69.00	3041140	36.27	90.05	17420.02	
Rela	tive Intens	ity	70.05	852154	10.16	91.05	50049	0.60
50.00	193502	2.31	71.05	201337	2.40	92.05	12378	0.15
50.95	998132	11.90	72.05	291571	3.48	93.05	53806	0.64
52.05	78758	0.94	73.05	117525	1.40	94.05	56033	0.67
53.00	286834	3.42	74.00	445073	5.31	95.05	152555	1.82
54.05	231648	2.76	75.00	56151	0.67	96.05	293878	3.50
55.00	7926265	94.53	76.05	231665	2.76	97.05	490760	5.85
56.00	3159524	37.68	77.00	6289020	75.01	98.10	409622	4.89
57.05	512937	6.12	78.00	457689	5.46	99.10	36799	0.44
58.00	1073834	12.81	79.00	188367	2.25	100.05	39560	0.47
59.00	49290	0.59	80.05	48237	0.58	100.90	28920.03	

101.95	28270.03		146.05	12000.01		190.00	1126	0.01
102.95	17651	0.21	147.05	25910.03		190.95	512	0.01
103.95	303938	3.62	148.00	999 0.01		191.90	542	0.01
104.95	8384767	100.00	149.00	460 0.01		192.95	1051	0.01
105.90	8382526	99.97	150.05	10470.01		194.00	726	0.01
106.85	1648731	19.66	151.00	978 0.01		194.95	315	0.00
108.10	65708	0.78	152.00	15660.02		196.00	417	0.00
109.10	195321	2.33	153.00	818 0.01		197.00	582	0.01
110.10	141446	1.69	154.05	29600.04		197.95	395	0.00
111.10	46406	0.55	155.15	12528	0.15	198.90	442	0.01
112.10	18025	0.21	156.10	878561	10.48	200.00	886	0.01
113.15	20893	0.25	157.05	92699	1.11	200.95	556	0.01
114.10	239329	2.85	158.05	65610.08		202.00	548	0.01
115.10	19610	0.23	159.05	36280.04		203.00	1162	0.01
116.05	29910.04		159.95	13340.02		204.00	6135	0.07
117.05	38240.05		160.95	780 0.01		204.95	5610	0.07
118.10	23690.03		162.00	16700.02		206.00	1638	0.02
119.05	40980.05		163.05	656 0.01		206.95	2894	0.03
120.05	57700.07		164.00	300 0.00	1	207.95	545	0.01
121.15	25819	0.31	165.00	999 0.01		208.95	392	0.00
122.05	137207	1.64	166.00	377 0.00	1	209.90	225	0.00
123.10	1123894	13.40	167.00	273 0.00	1	211.00	228	0.00
124.10	1390618	16.59	167.95	12670.02		212.00	7257	0.09
125.10	289447	3.45	169.05	812 0.01		213.00	1407	0.02
126.10	420102	5.01	170.00	74780.09		214.00	1038	0.01
127.10	39167	0.47	171.05	23670.03		215.05	569	0.01
128.10	72970.09		172.00	31400.04		216.00	231	0.00
129.05	19760.02		173.05	10880.01		216.95	586	0.01
130.00	14710.02		174.00	574 0.01		218.00	2578	0.03
131.05	20940.02		175.05	371 0.00	1	219.00	1370	0.02
131.95	13760.02		176.05	16740.02		219.90	662	0.01
133.05	16570.02		177.05	98420.12		220.90	431	0.01
134.05	54120.06		178.00	140374	1.67	221.90	250	0.00
135.05	35160.04		179.00	16908	0.20	222.90	132	0.00
136.10	12070	0.14	179.95	25920.03		223.90	111	0.00
137.15	61150.07		180.95	476 0.01		224.90	134	0.00
138.05	66730	0.80	181.95	590 0.01		225.90	302	0.00
139.10	75186	0.90	182.90	177 0.00	1	227.00	175	0.00
140.10	290004	3.46	183.90	294 0.00	1	227.95	1344	0.02
141.10	45326	0.54	184.90	223 0.00	1	229.00	462	0.01
142.05	20412	0.24	186.00	564 0.01		230.00	3876	0.05
143.10	23480.03		187.05	712 0.01		230.95	613	0.01
144.00	871 0.01		188.00	23020.03		231.90	566	0.01
145.00	22020.03		188.95	789 0.01		232.90	241	0.00

233.90	82 0.00	246.05	8377313 99.91	258.00	162 0.00
234.90	178 0.00	247.05	2906641 34.67	259.00	58 0.00
235.90	377 0.00	248.00	299668 3.57	260.15	40900.05
236.90	164 0.00	249.00	23406 0.28	261.05	298960 3.57
237.90	100 0.00	249.95	17540.02	262.05	<b>62278 0.74</b>
238.90	90 0.00	251.00	375 0.00	263.00	77460.09
239.90	70 0.00	252.00	191 0.00	264.05	10580.01
240.90	129 0.00	253.00	241 0.00	265.00	334 0.00
242.05	512 0.01	254.00	137 0.00	266.00	116 0.00
243.05	302 0.00	255.00	97 0.00	266.95	558 0.01
244.15	22930.03	256.00	16810.02	267.90	177 0.00
245.15	260429 3.11	257.00	346 0.00	268.90	226 0.00

#### 2.3.2 GC-MS Analysis of Raw Reaction Mixture by Using BHT as Radical Inhibitor



To a Schlenk tube were added 1-isocyano-2-(1-phenylvinyl)benzene **1a** (0.2 mmol), benzoyl chloride **2a** (6 mmol, 3 equiv),  $Ir(ppy)_3$  (1 mol%),2,6-lutidine (0.8 mmol, 4 equiv.), BHT (3 equiv), CH<sub>3</sub>CN (2 mL) at room temperature and 5 W blue LED irradiation for 12 h. The GC-MS analysis of raw reaction mixture showed that only trace amounts of the target product **3aa** was detected. The acyl radical trapping product 5, which generated from BHT capturing the acyl radical, could be detected by GC-Ms analysis.

The GC-MS analysis results of raw reaction mixture The GC spectra of raw reaction mixture



The MS spectra of the peak at 13.04 min

3. 0 (x10, 000, 000 TIC (1. 00)	))				时间 13.189	扫描号 1,839 强度	最大 章 -4,787,602	<u>强度: 38,764,982</u> 柱温箱温度293.78
2. 5								
2.0				$\wedge$				
1.0								
0. 5				/ \				
0.0					<u> </u>			
-0. 5	12.70 12.75	12.80 12.85 1	2.90 12.95	13.00 13.0	5 13.10 13.15	13.20 13.25	13.30 13	35 13.40
12.5	105	203 21	9	324		m/z 47.00 斜	a 2时强度	峰: 219/7, 379, 633 0 相对强度 0.00
10. 0	77							
7. 5								
5.0	119	161		325				
	المالا مالي والمالية		253 281		356 401 415	461 489	535 549	578 600
[MS Spec	trum]	150 200	70.95	31020.04	50 <del>1</del> 00	103.05	39503	0.54
# of Peaks	s539		72.05	13990.02		104.05	27761	0.38
Raw Spec	trum 13.0	40 (scan :	72.95	62870.09		105.00	1433650	19.43
1809)			73.95	26780.04		106.00	120100	1.63
Backgrou	nd No		75.05	56090.08		107.05	88718	1.20
Backgrou	nd Spectru	m	76.05	25759	0.35	108.05	10840	0.15
Base Peak	m/z 219.1	0 (Inten :	77.00	791783	10.73	109.05	21635	0.29
7,379,633	)		78.00	74266	1.01	110.10	25910.04	
Event#	1		79.05	90303	1.22	111.05	23120.03	
m/z Abso	olute Intens	sity	80.00	17868	0.24	112.05	541 0.01	
Rela	tive Intens	ity	81.05	43333	0.59	113.00	11660.02	
50.00	11537	0.16	82.05	49590.07		114.05	21100.03	
51.00	84015	1.14	83.00	26233	0.36	115.05	106839	1.45
52.00	10804	0.15	84.00	20260.03		116.05	37959	0.51
53.00	44962	0.61	85.10	13100.02		117.05	76011	1.03
54.05	47680.06		85.95	757 0.01		118.15	16738	0.23
55.00	176780	2.40	87.05	17210.02		119.05	225869	3.06
56.05	13536	0.18	88.05	21090.03		120.10	33698	0.46
57.00	216455	2.93	89.00	13862	0.19	121.05	212005	2.87
58.00	11246	0.15	90.05	59960.08		122.05	21553	0.29
59.00	10884	0.15	91.00	325230	4.41	123.05	22868	0.31
60.05	599 0.01		92.05	31772	0.43	124.10	25560.03	
61.05	309 0.00		93.05	65262	0.88	125.05	11720.02	
61.95	10450.01		94.05	11387	0.15	126.05	28650.04	
63.00	74970.10		95.05	30685	0.42	127.05	35968	0.49
64.05	44790.06		96.05	52290.07		128.05	130389	1.77
65.00	43562	0.59	97.05	68960.09		129.05	133212	1.81
66.00	65480.09		98.00	705 0.01		130.05	47160	0.64
67.00	59321	0.80	99.00	454 0.01		131.05	98909	1.34
68.05	40030.05		100.05	555 0.01		132.10	23439	0.32
69.00	86988	1.18	101.00	42580.06		133.05	188145	2.55
70.00	53560.07		102.00	92460.13		134.05	37000	0.50

135.10	47825	0.65	179.00	10823	0.15	223.00	72360.10	
136.05	60610.08		180.05	31070.04		224.05	17380.02	
137.10	22600.03		181.00	69860.09		225.00	22553	0.31
138.05	442 0.01		181.95	19930.03		226.00	41240.06	
139.05	44120.06		183.00	38710.05		227.00	948 0.01	
140.05	21110.03		184.00	12170.02		228.00	11270.02	
141.05	52684	0.71	185.05	16282	0.22	229.00	24730.03	
142.05	32458	0.44	186.05	35390.05		230.05	11670.02	
143.05	65399	0.89	187.05	60280	0.82	231.00	45530.06	
144.05	26600	0.36	188.05	47455	0.64	232.05	23010.03	
145.05	135211	1.83	189.05	85991	1.17	233.00	42810.06	
146.05	34667	0.47	190.05	15502	0.21	234.00	40070.05	
147.05	142974	1.94	191.00	11015	0.15	235.05	13015	0.18
148.05	22857	0.31	192.00	53080.07		236.05	30270.04	
149.05	33134	0.45	193.00	53860.07		237.05	48680.07	
150.05	40700.06		194.00	34280.05		238.05	889 0.01	
151.05	30780.04		194.95	48170.07		239.05	24920.03	
152.05	11177	0.15	196.00	964 0.01		239.95	676 0.01	
153.05	17161	0.23	197.00	54280.07		240.95	867 0.01	
154.00	10311	0.14	198.00	13290.02		242.05	457 0.01	
155.05	26523	0.36	199.00	32250.04		243.05	15630.02	
156.05	24986	0.34	200.05	18920.03		244.00	980 0.01	
157.05	27508	0.37	201.05	42890.06		245.00	11610.02	
158.05	11483	0.16	202.15	18283	0.25	246.00	663 0.01	
159.05	47630	0.65	203.05	922112	12.50	247.05	998 0.01	
160.05	28475	0.39	204.05	153855	2.08	248.05	10130.01	
161.05	228527	3.10	205.00	25302	0.34	249.00	47450.06	
162.05	42925	0.58	206.05	54820.07		250.05	21030.03	
163.05	28856	0.39	206.95	21916	0.30	251.00	39030.05	
164.05	44930.06		208.00	56740.08		252.05	14170.02	
165.05	15258	0.21	208.95	14804	0.20	253.05	20404	0.28
166.00	64650.09		210.00	34940.05		254.00	38550.05	
167.05	56160.08		210.95	31710.04		254.95	672 0.01	
168.05	18740.03		212.05	894 0.01		255.90	98 0.00	
169.05	52740.07		213.00	46500.06		257.05	426 0.01	
170.05	24020	0.33	213.95	852 0.01		258.10	823 0.01	
171.05	17574	0.24	215.00	64540.09		258.95	446 0.01	
172.05	63500.09		216.00	36100.05		259.95	584 0.01	
173.05	47940	0.65	217.05	22865	0.31	261.10	455 0.01	
174.05	15807	0.21	218.15	71128	0.96	262.05	379 0.01	
175.05	72316	0.98	219.10	7379633	100.00	263.05	13990.02	
176.05	13521	0.18	220.10	1300937	17.63	264.05	669 0.01	
177.00	47610.06		221.05	129937	1.76	265.00	73160.10	
178.00	12202	0.17	222.05	10466	0.14	266.05	47800.06	

267.05	10349 0.14	289.00	370 0.01	311.10	10316 0.14
268.00	41400.06	290.10	110 0.00	312.05	832 0.01
269.00	18410.02	291.10	21320.03	313.10	158 0.00
270.00	254 0.00	292.15	719 0.01	314.10	65 0.00
271.00	134 0.00	293.10	14258 0.19	315.10	34 0.00
272.00	138 0.00	294.10	34620.05	316.10	60 0.00
273.00	135 0.00	295.10	20860.03	317.10	42 0.00
274.00	79 0.00	296.15	515 0.01	318.10	27 0.00
275.00	398 0.01	297.20	103 0.00	319.10	31 0.00
276.00	218 0.00	298.20	110 0.00	320.10	86 0.00
277.05	542 0.01	299.20	84 0.00	321.10	266 0.00
278.10	186 0.00	300.20	31 0.00	322.10	21500.03
279.05	14150.02	301.20	21 0.00	323.15	89640.12
280.05	862 0.01	302.20	49 0.00	324.10	1120295 15.18
281.05	30839 0.42	303.20	121 0.00	325.10	276106 3.74
282.05	68180.09	304.20	130 0.00	326.10	37550 0.51
283.00	18660.03	305.20	97 0.00	327.00	42120.06
284.00	303 0.00	306.10	239 0.00	327.95	675 0.01
285.00	130 0.00	307.10	29770.04	329.00	140 0.00
286.00	54 0.00	308.15	83870.11	330.00	81 0.00
287.00	90 0.00	309.10	343746 4.66		
288.00	66 0.00	310.10	81161 1.10		

## 2.3.3 GC-MS Analysis of Raw Reaction Mixture by Using 1,1-diphenylethen as Radical Inhibitor



To a Schlenk tube were added 1-isocyano-2-(1-phenylvinyl)benzene **1a** (0.2 mmol), benzoyl chloride **2a** (6 mmol, 3 equiv),  $Ir(ppy)_3$  (1 mol%),2,6-lutidine (0.8 mmol, 4 equiv.), 1,1-diphenylethene (3 equiv), CH<sub>3</sub>CN (2 mL) at room temperature and 6 W blue LED irradiation for 12 h. The GC-MS analysis of raw reaction mixture showed that only trace amounts of the target product **3aa** was detected. The acyl radical trapping product 6, which generated from 1,1-diphenylethene capturing the acyl radical, could be detected by GC-Ms analysis.

The GC-MS analysis results of raw reaction mixture

The GC spectra of raw reaction mixture



The MS spectra of the peak at 13.47 min

%												基峰: 283/5,	012, 069
100						283				m/z 53.00	绝对强度	8,170 相对强度	0.16
100													
-													
75-													
- 1													
50-													
- 1													
25-		77		178									
1		Ϋ́	105		207								
1	8	<b>j</b> 1	126		239 2	52	207	255 295	415 400	475		0 560 505	
0-4		<u> </u>		#		·····	10 · · · · · · · · · · · · · · · · · · ·	300 300	410 428	***	<u></u>	<u>a 102 - 1080</u>	

[MS Spec	trum]		60.95	10360.0	02		83.05	10980.02	
# of Peaks	s548		62.00	92040.	19		84.00	15020.03	
Raw Spec	trum 13.46	65 (scan :	63.00	37654		0.77	84.95	14730.03	
1894)			63.95	44530.0	09		85.95	74180.15	
Backgrou	nd No		65.00	10623		0.22	87.00	17185	0.35
Backgrou	nd Spectru	m	66.00	11260.0	02		88.05	20781	0.43
Base Peak	cm/z 283.03	5 (Inten :	67.00	506 0.0	01		89.00	39569	0.81
4,881,985	)		68.00	398 0.0	01		89.95	39650.08	
Event#	1		69.00	926 0.0	02		91.00	17742	0.36
m/z Abso	olute Intens	ity	69.95	380 0.0	01		92.00	20020.04	
Rela	tive Intensi	ty	71.05	660 0.0	01		93.55	46480.10	
50.00	41241	0.84	72.05	310 0.0	01		94.50	81480.17	
51.00	261439	5.36	73.05	64160.	13		96.00	28700.06	
52.00	27970	0.57	74.00	23222		0.48	97.05	10420.02	
53.00	67350.14		75.00	47231		0.97	97.95	69890.14	
54.00	362 0.01		76.05	96551		1.98	99.00	79030.16	
55.00	19670.04		77.00	921450	)	18.87	100.05	10771	0.22
55.95	744 0.02		78.00	70839		1.45	101.05	34539	0.71
57.05	926 0.02		79.05	40070.0	08		102.00	63919	1.31
58.00	217 0.00		79.95	422 0.0	01		103.00	28119	0.58
59.00	575 0.01		81.20	21400.0	04		104.05	82420.17	
60.00	182 0.00		82.15	14200.0	03		105.00	707974	14.50

106.00	59428	1.22	150.00	43596	0.89	193.95	813 0.02	
107.40	14650	0.30	151.00	117575	2.41	194.90	378 0.01	
108.35	33430.07	1	152.00	262798	5.38	195.90	158 0.00	
109.00	11530.02	2	153.00	49727	1.02	196.90	170 0.00	
110.00	22220.05	;	154.00	82380.17	7	198.05	882 0.02	
111.05	38960.08	3	155.05	956 0.02	2	199.00	1223 0.03	
112.05	85290.17	1	156.00	98 0.00	C	199.95	50690.10	
113.05	50630	1.04	157.00	121 0.00	C	200.95	44790.09	
114.00	29902	0.61	158.00	156 0.00	C	202.00	13033	0.27
115.00	25804	0.53	159.00	217 0.00	C	203.00	41450.08	
116.00	37620.08	}	160.00	36 0.00	C	204.05	24280.05	
116.60	11720.02	2	161.00	13020.03	3	205.00	67231	1.38
117.65	17590.04	ļ	162.00	40420.08	8	206.05	50354	1.03
118.65	12493	0.26	163.00	18378	0.38	207.00	710806	14.56
119.65	57577	1.18	164.00	12934	0.26	208.00	114274	2.34
120.55	17877	0.37	165.00	72337	1.48	208.95	12585	0.26
122.05	25688	0.53	166.00	11102	0.23	210.00	11350.02	
122.95	32670.07	,	167.00	35100.07	7	210.95	22550.05	
124.05	27910.06	5	168.00	759 0.02	2	211.95	957 0.02	
125.05	23620	0.48	169.00	150 0.00	0	213.00	79350.16	
126.05	85601	1.75	170.00	50 0.00	0	214.05	29590.06	
127.00	43162	0.88	171.00	79 0.00	0	215.00	25495	0.52
128.00	25183	0.52	172.00	108 0.00	0	216.00	61500.13	
129.00	14359	0.29	173.05	621 0.02	1	216.95	10930.02	
130.45	29570.06	)	174.00	69640.14	4	218.00	986 0.02	
131.45	23772	0.49	175.05	17182	0.35	218.90	524 0.01	
132.35	30975	0.63	176.00	240271	4.92	219.90	169 0.00	
133.35	11923	0.24	177.05	202355	4.14	220.90	13930.03	
134.15	15453	0.32	178.00	1230174	25.20	221.95	856 0.02	
135.15	39870.08	5	179.00	563822	11.55	223.00	10260.02	
136.05	381 0.01		180.00	73338	1.50	224.00	57610.12	
137.00	52920.11		181.00	16476	0.34	224.95	47600.10	
138.00	43620.09	)	182.00	18700.04	4	225.95	26437	0.54
139.05	29705	0.61	183.00	265 0.0	1	227.00	13438	0.28
140.05	98080.20	)	184.00	68 0.00	0	228.00	22042	0.45
141.05	33317	0.68	184.95	428 0.0	1	229.00	11092	0.23
142.05	43672	0.89	186.05	792 0.02	2	230.00	18780.04	
142.95	43790.09	)	187.00	53540.1	1	231.00	930 0.02	
144.00	242 0.00	)	188.00	45750.09	9	232.00	241 0.00	
144.95	405 0.01		189.00	24620	0.50	233.00	170 0.00	
146.00	242 0.00	)	190.05	82970.17	7	233.90	174 0.00	
147.00	13900.03	1	191.00	34575	0.71	234.90	898 0.02	
148.05	462 0.01		192.00	58380.12	2	236.00	742 0.02	
149.05	55850.11		192.95	41150.08	8	236.95	48130.10	

238.05	37850.08		258.90	254 0.01		280.05	15200.03	
239.00	107818	2.21	260.00	281 0.01		281.00	25447	0.52
240.00	70938	1.45	261.00	14060.03	;	282.05	31295	0.64
241.00	64513	1.32	262.05	877 0.02		283.05	4881985	100.00
242.00	13153	0.27	263.00	12967	0.27	284.05	2393937	49.04
243.00	23610.05		264.00	51780.11		285.00	432991	8.87
243.95	848 0.02		265.00	48985	1.00	286.00	48350	0.99
244.90	284 0.01		266.00	15177	0.31	287.05	33390.07	
245.90	132 0.00		267.05	13878	0.28	288.00	412 0.01	
247.00	199 0.00		268.00	11591	0.24	289.00	138 0.00	
248.00	17960.04		269.00	42950.09	)	290.00	73 0.00	
248.95	25930.05		270.05	925 0.02	2	291.00	92 0.00	
250.00	19766	0.40	271.00	162 0.00	)	292.00	70 0.00	
251.05	91470.19		272.00	78 0.00	)	293.00	241 0.00	
252.00	89367	1.83	273.00	132 0.00	)	294.00	122 0.00	
253.00	83564	1.71	274.00	87 0.00	)	295.00	494 0.01	
254.00	39302	0.81	275.00	170 0.00	)	296.00	345 0.01	
255.00	77166	1.58	276.00	153 0.00	)	297.00	215 0.00	
256.00	37142	0.76	277.00	194 0.00	)	298.00	39 0.00	
257.00	93960.19		278.05	296 0.01				
257.95	11490.02		279.00	14330.03				

## 2.4 The Light on/off Experiments



Figure S2 The Light on/off Experiments

Time/h	0	4 (blue)	8 (dark)	12 (blue)	16 (dark)	20 (blue)
Yield/%	0	35.7	35.7	62.3	62.3	80.0

The above depicted reaction was performed according to the general protocol established. The reaction was irradiated with 5 W blue LEDs for 4 hour and then stirred in the dark for 4 hour. This procedure was repeated for 20 hours, and the yield of the product was determined by <sup>1</sup>H NMR with dibromomethane as an internal standard at each point the light was turned off or on. The results are shown in the graph above. This result shows that constant light irradiation is needed to progress the reaction.

#### 3. References

[1] J. Wang, H. Liu, Y. Liu, W. Hao, Y. Yang, Y. Sun and X. Xu, Org. Chem. Front., 2022, 9, 6484-6489.

#### 4. Spectra



#### Phenyl(4-phenylquinolin-2-yl)methanone(3aa)





#### (4-Phenylquinolin-2-yl)(p-tolyl)methanone(3ab)



S16









(4-Methoxyphenyl)(4-phenylquinolin-2-yl)methanone(3ad)

















#### 4-(4-Phenylquinoline-2-carbonyl)benzonitrile (3ah)





(4-Phenylquinolin-2-yl)(p-tolyl)methanone (3ai)







(3-Methoxyphenyl)(4-phenylquinolin-2-yl)methanone(3aj)


















## (3-Bromophenyl)(4-phenylquinolin-2-yl)methanone (3am)



























## (3,5-Dichlorophenyl)(4-phenylquinolin-2-yl)methanone(3ar)





(4-Phenylquinolin-2-yl)(thiophen-2-yl)methanone(3as)























(6-Methoxy-4-phenylquinolin-2-yl)(phenyl)methanone(3fa)





(6-Fluoro-4-phenylquinolin-2-yl)(phenyl)methanone (3ga)







(6-Chloro-4-phenylquinolin-2-yl)(phenyl)methanone (3ha)














## (8-Methyl-4-phenylquinolin-2-yl)(phenyl)methanone (3ka)









S76





(4-(4-Chlorophenyl)quinolin-2-yl)(phenyl)methanone (3ma)



S79



## (4-(4-Bromophenyl)quinolin-2-yl)(phenyl)methanone (3na)









Phenyl(4-(o-tolyl)quinolin-2-yl)methanone (3pa)





## 1,3,3-Triphenylprop-2-en-1-one (6)

