New Aryl Derivatives of Quinolinedione and Related Heterocyclic Compounds

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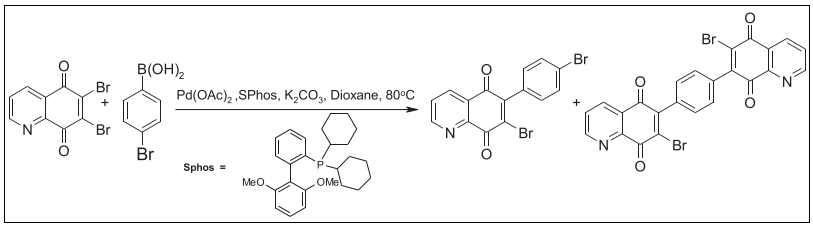

The synthesis of new derivatives of 6,7-dibromoquinoline-5,8-dione and 6,7-dichloroquinoline-5,8-dione via palladium/Sphos-mediated Suzuki–Miyaura cross-coupling reaction is reported. The 6,7-dibromoquinoline-5,8-dione and 6,7-dichloroquinoline-5,8-dione intermediates were prepared in a three-step reaction from 8-hydroxyquinoline. The palladium-catalyzed reactions of 6,7-dibromoquinoline-5,8-dione and 6,7-dichloroquinoline-5,8-dione with a variety of aryl boronic acids provide coupled compounds in high yields. The arylation of 6,7-dibromoquinoline-5,8-dione and 6,7-dichloroquinoline-5,8-dione with 4-bromophenyl boronic acid supplied 6,6′-(1,4-phenylene)bis(7-bromoquinoline-5,8-dione) and (4-(6-(4-(6-chloro-5,8-dihydroquinolin-7-yl)phenyl)-5,8-dihydroquinolin-7-yl)phenyl)boronic acid respectively, in addition to the expected coupled compounds in moderate yields. Also, Pd(0)/PPh_3 allowed the 7-chloro-6-(4-nitrophenyl)quinoline-5,8-dione and 7-chloro-6-phenylquinoline-5,8-dione to be synthesized via Heck reaction. The yields of the synthesized target molecules depend largely on the reaction conditions and the type of ligands employed. Structural assignments of the synthesized compounds were established by spectra and analytical data.

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INTRODUCTION

The surge to employ halogenated quinolines as intermediates in metal-catalyzed C–C bond formation for construction of complex molecules such as natural products, pharmaceuticals, and material science motivated investigating facile reaction protocol for functionalization of structurally related halogenated quinoline-5,8-dione [1–3]. Quinoline-5,8-dione is an active component in various antimalaria, antimicrobial, anagelsic, cardiovascular, anticancer, and anti-inflammatory drugs [4]. The recent increase in microbial infections has generated a renewed interest in search for new drugs [5]. To this end, quinoline-5,8-dione had undergone various synthetic transformations with a view to developing potent antibacterial and antifungal agents [6–10]. Pd-catalyzed reaction on similar system has earlier been reported [11–13]. Also, we have recently reported the synthesis of new quinolinedione derivatives and related heterocyclic compounds, which were obtained by cross-coupling of 6,7-dihaloquinoline-5,8-diones **2**,**3** and chlorophenothiazines/chlorophenoxaines, respectively [14,15].

In continuation of our avid interest in this class of compounds, the synthesis of some new aryl derivatives of halogenatedquinoline-5,8-diones (**2** and **3**) via palladium-mediated Suzuki–Miyuara and Heck cross-coupling reactions is reported.

RESULTS AND DISCUSSION

The intermediates 6,7-dibromoquinoline-5,8-dione and 6,7-dichloroquinoline-5,8-dione were prepared in a three-step reaction starting from 8-hydroxyquinoline **4** (Fig. 1) [16].

The optimization results of the cross-coupling of 6,7-dibromoquinoline-5,8-dione with phenyl boronic acid (Fig. 2) is given in Table 1.

The combination of $Pd(OAc)_2$ and PPh_3 gave insignificant % yield of 7-bromo-6-phenylquinoline-5,8-dione **5a** even when reaction was run for 2 days. While the use of $Pd(OAc)_2$/Sphos supplied moderate % yield of same product, the reaction time was relatively long (8 h). Based on earlier work of Buchwald and coworkers [17], the yield of 7-bromo-6-phenylquinoline-5,8-dione was further enhanced by water preactivation of the catalyst system. This was performed by first heating both $Pd(OAc)_2$ (1 mol%), Sphos (3 mol%) in H_2O (4 mol%) at 80°C in 1,4-dioxane within 1 min before the introduction of 6,7-dibromoquinoline-5,8-dione and phenyl boronic acid.

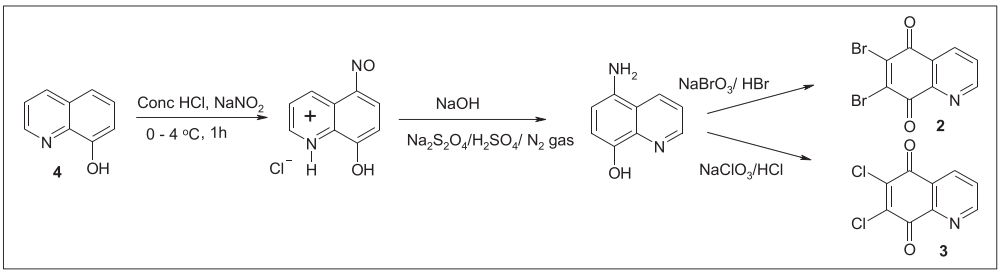


Figure 1. Synthesis of 6,7-dihaloquinoline-5,8-diones.

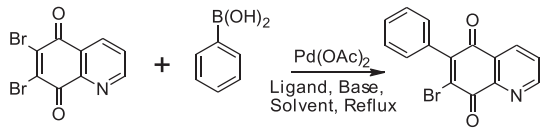


Figure 2. Synthesis 6,7-dibromoquinoline-5,8-dione derivatives via Suzuki-Miyaura cross-coupling.

Table 1

Optimization reaction of 6,7-dibromoquinoline-5,8-dione with phenyl boronic acid.

S/N	Reaction condition[a,b]	Percent yield[f] (%)	Reaction time (h)
1	$Pd(OAc)_2/PPh_3$	trace	48
2	$Pd(OAc)_2$/SPhos	44	8
3	$Pd(OAc)_2$/SPhos[c] (WA[d])	56	2
4	$Pd(OAc)_2$/BHDTBBP[e] (WA[d])	45	3

[a]Reaction condition: $Pd(OAc)_2$ = 5 mol%; ligand = 10 mol%; $Ar(OH)_2$ = 1 eq; 6,7-dibromoquinoline-5,8-dione = 1.5 eq; water = 1 mL; *tert*-BuOH = 2 mL.
[b]In reactions 3 and 4, $Pd(OAc)_2$ and ligand in aqueous *tert*-BuOH were heated to 80°C (water preactivated) within 60 s before addition of dibromoquinoline-5,8-dione and phenyl boronic acid.
[c]2-Dicyclohexylphosphino-2′,6′-dimethoxybiphenyl.
[d]WA = water activation.
[e]1,4-bis(2-hydroxy-3,5-di-*tert*-butylbenzyl)piperizine.
[f]Isolated yields.

The preactivation was visually monitored by color change. Therefore, with water preactivation of catalytic system, the synthesis of 7-bromo-6-phenylquinoline-5,8-dione was achieved in a very short time (2 h) (Table 1).

Interestingly, it was observed that there was no apparent difference in yield of product when reaction was conducted under inert and aerobic conditions. The optimized reaction was employed as a general protocol to synthesized compounds 6,6′-(1,4-phenylene)bis(7-bromoquinoline-5,8-dione) **5b**, 7-bromo-6-(3-nitrophenyl)quinoline-5,8 dione **5c**, 7-bromo-6-phenylquinoline-5,8-dione **5d** (Table 2). Furthermore, a synthesized, non-phosphorus, air and moisture stable 1,4-bis(2-hydroxy-3,5-di-tert-butylbenzyl)piperazine (BHDTBBP) ligand [18] in combination with Pd(OAc)2 under water preactivation and similar reaction conditions, also displayed good activity in the Suzuki-Miyaura cross-coupling of 6,7-dichloroquinoline-5,8-dione. $Pd(OAc)_2$/BHDTBBP allowed the synthesis of 6-(4-bromophenyl)-7-chloroquinoline-5,8-quinone **6a**, (4-(6-(4-(6-chloro-5,8-dihydroquinolin-7-yl)phenyl)-5,8-dihydroquinolin-7-yl) phenyl)boronic acid **6b** and 7-chloro-6-(3-nitrophenyl) quinoline-5,8-quinone **5c** derivatives. The synthesized derivatives were purified by recrystallization from aqueous acetone. Derivatives **5b** and **6b** were isolated by column chromatography as reddish solids in addition to the desired coupled products (**5a and 6a**) from the reaction of 6,7-dibromoquinoline-5,8-dione and 6,7-dichloroquinoline-5,8-dione with 4-bromophenylboronic acid, respectively. The spectral and elemental analyses of **5b** and **6b** agreed with structural assignments. Moreover, their mass spectra furnished their molecular ion fragments (M^+, 100%) with mass/charge ratios of 550.26 and 546.34, respectively.

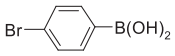
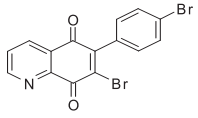
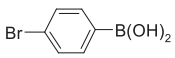
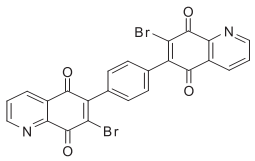
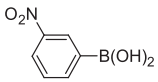
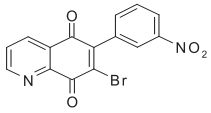
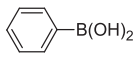
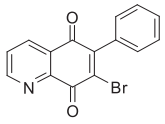
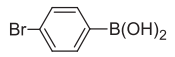
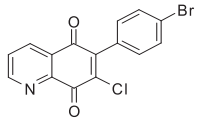
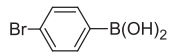
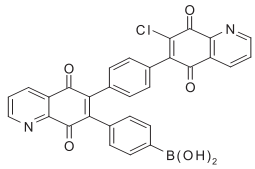
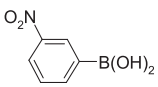
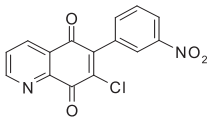
In another development, 7-chloroquinolin-5.8-dione substrate **7**, which was obtained by multistep[19] conversion of 8-hydroxyquinoline **4** (Fig. 3), was coupled with 4-nitroiodobenzene and 4-iodobenzene, respectively, *via* Pd(0)/PPh_3-catalyzed Heck reaction to supply 7-chloro-6-(4-nitrophenyl)quinoline-5,8-dione **8** and 7-chloro-6-phenylquinoline-5,8-dione **9** in moderate yields. The structures of all the synthesized compounds were established by spectral and analytical data.

CONCLUSION

The palladium-catalyzed Suzuki–Miyaura cross-coupling of 6,7-dibromoquinoline-5,8-dione and 6,7-chloroquinoline-5,8-dione, respectively, provides a convenient route for synthesizing their aryl derivatives in good yields. Arylation of 6,7-dibromoquinoline-5,8-dione and 6,7-chloroquinoline-5,8-dione via water-preactivated palladium-catalyzed cross-coupling furnished, 6,6′-(1,4-phenylene)bis(7-bromoquinoline-5,8-dione) and (4-(6-(4-(6-chloro-5,8-dihydroquinolin-7-yl)phenyl)-5,8-dihydroquinolin-7-yl)phenyl)boronic acid, respectively, in

Table 2

Derivatives of 6,7-halogenatedquinoline-5,8-dione.[a,b,c]

Entry	Ar-X	$Ar(OH)_2$	Product	Reaction time (h)	Yield (%)[d]
1	**2**		 **5a**	1.30	50
2	**2**		 **5b**	1.30	37
3	**2**		 **5c**	1.30	53
4	**2**		 **5d**	2.00	51
5	**3**		 **6a**	2.30	50
6	**3**		 **6b**	2.30	33
7	**3**		 **6c**	2.30	56

[a]Reaction condition: $Pd(OAc)_2$ = 2.5 mol%; Sphos = 10 mol%; $Ar(OH)_2$ = 1 eq; 6,7-dibromoquinoline-5,8-dione = 1.5 eq; water = 1 mL; *tert*-BuOH = 2 mL. The $Pd(OAc)_2$ and ligand in aqueous *tert*-BuOH were heated to 80°C (water preactivated) within 60 s before addition of dibromo or dichloroquinoline-5,8-dione and boronic acid.

[b]For reactions 5 and 6, 1,4-bis(2-hydroxy-3,5-di-*tert*-butylbenzyl)piperizine (5 mol%) was used in place of Sphos.

[c]The reaction of compounds **2** and **3** with 4-bromophenylboronic acid gave product **4a** with **4b** and **5a** with **5b**, which were separated by flash column chromatography by using methanol eluent.

[d]Isolated yields.

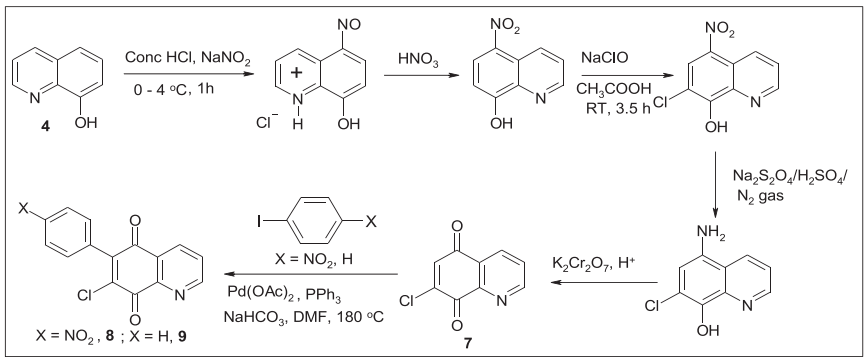


Figure 3. Synthesis of 7-chloro-6-(4-nitrophenyl)quinoline-5,8-dione **6** and 7-chloro-6-phenylquinoline-5,8-dione.

addition to the expected coupled compounds. Also, Pd(0)/PPh_3 allowed the 7-chloro-6-(4-nitrophenyl)quinoline-5,8-dione and 7-chloro-6-phenylquinoline-5,8-dione to be synthesized via Heck reaction. The yields of the synthesized target molecules depend largely on the reaction conditions and the type of ligands employed.

EXPERIMENTAL SECTION

General. All chemicals were purchased from Aldrich Chemical Company, UK, and were used without further purification. Otherwise stated, all compounds were synthesized and characterized in School of Chemistry, Cardiff University, UK. Melting points were determined with Fisher–Johns apparatus and are uncorrected. Ultraviolet and visible spectra were recorded on a Unico-UV2102 PC spectrophotometer by using matched 1 cm quartz cells and methanol solvent. The absorption maxima were recorded in nanometers (nm) and figures in parenthesis are in log ε. The NMR spectra were recorded on Bruker DPX 500 and Oxford 300. Chemical shifts are reported in ppm (δ) scale relative to TMS as internal standard and coupling constants (*J*) were reported in hertz (Hz). The following abbreviations were used: s (singlet); d (doublet); dd (doublet of doublet); t (triplet); m (multiplet). Mass spectrometric data were obtained on a Varian 1200 Quadrupole Mass Spectrometer and Micro-mass Quadro II Spectrometer. Elemental analyses were obtained on Heraeus CHN-O rapid analyzer.

General procedures for Suzuki-Miyaura reaction

Procedure A. SPhos (10 mol%) and $Pd(OAc)_2$ (2.5 mol %) were placed in a 25-mL three-neck round bottom flask. This was followed by addition of water (1 mL) and *tert*-BuOH (2 mL) and the solution boiled for 60 s at 80°C under inert atmosphere. Thereafter, 6,7-dibromo-5,8-quinolin-5,8-dione (0.316 mmol), the boronic acid (0.631 mmol), K_2CO_3 (3 eq.), and *tert*-BuOH (2 mL) were added, and the entire reaction mixture was refluxed with continuous stirring. The reaction progress was monitored by TLC and after completion was cooled and filtered to obtain the crude product. This was further recrystallized from water–acetone to give the desired product. Procedure A was used as a general method for the synthesis of compounds **5a–5d**.

Procedure B. 1,4-Bis(2-hydroxy-3,5-di-*tert*-butylbenzyl) piperazine (5 mol%) and $Pd(OAc)_2$ (5 mol%) were placed in a 25-mL three-neck round bottom flask. This was followed by introduction of water (1 mL) and dioxane (4 mL), and the solution was heated for 60 s at 80°C under inert atmosphere. Thereafter, 6,7-chloroquinoline-5,8-dione (6.60 mmol), boronic acid (7.90 mmol), K_2CO_3 (1.4 eq.), and dioxane (2 mL) were added, and the reaction mixture was heated under reflux with continuous stirring. After the termination of the reaction, it was cooled to room temperature, filtered, and recrystallized from water–acetone to give the desired product. *Procedure B* was used as a general method for the synthesis of compounds **6a–6c**.

7-Bromo-6-(4-bromophenyl)quinoline-5,8-dione (5a). Procedure A was employed to cross-coupled 6,7-dibromoquinoline-5,8-dione (200 mg, 0.63 mmol) with 4-bromophenylboronic acid (250 mg, 1.26 mmol) to obtain the title product (**5a**) in 1.5 h as red solid after purification by flash column chromatography. Yield: 142.1 mg (50% yield); mp 260–262°C. UV-Visible λ_{max} (MeOH): 333 (3.236), 350 (2.868), 421 (3.816), 439 (3.111). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.8 (dd, 1 H, *J* = 1.6, 4.8 Hz), 8.4 (dd, 1 H, *J* = 1.6, 8.0 Hz), 7.7 (dd, 1 H, *J* = 4.8, 7.6 Hz), 7.4 (d, 2H, *J* = 8.5), 7.3 (2H). *Anal.* Calcd. for $C_{15}H_7Br_2NO_2$: C, 45.84; H, 1.80; N, 3.56. Found: C, 45.90; H, 1.76; N, 3.52

6,6'-(1,4-Phenylene)bis(7-bromoquinoline-5,8-dione) (5b). Compound **5b** was isolated as dark red solid by flash column chromatography as second product from the reaction of 6,7-dibromoquinoline-5,8-dione (200 mg, 0.63 mmol) with 4-bromophenylboronic (250 mg, 1.26 mmol). Yield: 147.2 mg (37%); mp 327–329°C. UV-Visible λ_{max} (MeOH): 335 (3.271), 350 (3.123), 441 (3.548), 440 (3.694). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.2 (d, 2H, *J* = 7.5), 7.6 (dd, 2H, *J* = 8.0, 1.0), 7.1 (s, 4H), 7.0 (dd,

2H, $J = 7.0$, 1.0). MS (APCI), *m/z* (% relative intensity): 545.23 (25), 547.26 (51), 550.26 [(100), M^+]. *Anal.* Calcd. for $C_{24}H_{10}Br_2N_2O_4$: C, 52.40; H, 1.83; N, 5.09. Found: C, 52.40; H, 1.52; N, 5.21. Note that compounds **5a** and **5b** were separated by flash chromatography by using methanol as eluent.

7-Bromo-6-(3-nitrophenyl)quinoline-5,8-dione (5c). Procedure A was used to coupled 6,7-dibromoquinoline-5,8-dione (100 mg, 0.316 mmol) with 3-nitrophenylboronic acid (110 mg, 0.631 mmol) to supply the title product (**5c**) in 1.5 h as dark reddish solid. Yield: 180.3 mg (53%); mp 360–362°C. UV-Visible λ_{max} (MeOH): 333 (4.654), 350 (3.879), 421 (3.326), 439 (3.267). ^{1}H NMR (400 MHz, DMSO): δ 8.8 (s, 1H), 8.2 (1H), 8.1 (1H), 7.7 (1H), 7.6 (s, 1H), 7.5 (1H), 7.3 (1H). IR ν_{max} cm^{-1}: 1651.14 (C=O); 1392.66 (NO_2). MS (APCI), *m/z* (% relative intensity): 314.06 (10), 315.09 (55), 361.10 [(100), $M^+ + 2$]. *Anal.* Calcd. for $C_{15}H_7BrN_2O_4$: C, 50.17; H, 1.96; N, 7.80. Found: C, 50.20; H, 1.94; N, 7.75.

7-Bromo-6-phenylquinoline-5,8-dione (5d). Procedure A was apply to coupled 6,7-dibromoquinoline-5,8-dione (100 mg, 0.316 mmol) with phenylboronic acid (80 mg, 0.474 mmol) to afford the title product (**5d**) in 2 h as red solid. Yield: 141 mg (51%); mp 316–318°C. UV-Visible λ_{max} (MeOH): 600 (2.536), 620 (2.44), 630 (1.816), 640 (1.341). ^{1}H NMR (400 MHz, DMSO): 8.8 (1H), 7.9 (dd, 1H, $J = 1.2$, 8.0 Hz), 7.5 (2H), 7.4 (dd, 1H, $J = 1.6$, 7.6 Hz), 7.2 (m, 3H). *Anal.* Calcd. for $C_{15}H_8BrNO_2$: C, 57.35; H, 2.57; N, 4.46. Found: C, 57.40; H, 2.71; N, 4.50.

6-(4-Bromophenyl)-7-chloroquinoline-5,8-quinone (6a). Procedure B was employed for the reaction of 6,7-dichloro-5,8-quinolinequinone (1500 mg, 6.60 mmol) with 4-bromophenylboronic acid (1600 mg, 7.90 mmol) to obtain the title product (**6a**) in 2.5 h as red solid after purification by flash column chromatography. Yield: 150 mg (50%); mp 326–328°C. UV-Visible λ_{max} (MeOH): 600 (3.411), 620 (2.732), 630 (3.546), 640 (3.255). ^{1}H NMR (400 MHz, DMSO): 8.9 (dd, 1H, $J = 1.2$, 4.4, Hz), 8.7 (dd, 1H, $J = 1.6$, 8.4 Hz), 8.0 (d, 2H, $J = 7.1$), 7.8 (dd, 1H, $J = 4.8$, 8.4 Hz), 6.8 (2H). *Anal.* Calcd. for $C_{15}H_7BrClNO_2$: C, 51.69; H, 2.02; N, 4.02. Found: C, 51.70; H, 2. 05; N, 4.10.

(4-(6-(4-(6-Chloro-5,8-dihydroquinolin-7-yl)phenyl)-5,8-dihydroquinolin-7-yl)phenyl)boronic acid (6b). Compound **6b** was isolated as dark red solid by flash column chromatography as second product from the reaction of 6,7-dichloroquinoline-5,8-dione (1500 mg, 6.60 mmol) with 4-bromophenylboronic (1600 mg, 7.90 mmol). Yield: 1191 mg (33%); mp 318–320°C. UV-Visible λ_{max} (MeOH): 222 (3.776), 350 (2.443), 427 (3.836), 440 (3.342). ^{1}H NMR (400 MHz, DMSO): 8.2 (d, 2H), 7.9 (2H), 7.5 (m, 2H), 7.3 (4H), 7.2 (m, 4H), 5.8 (s, 2H). MS (APCI), *m/z* (% relative intensity): 545.33 (52), 546.34 [(100), M^+]. *Anal.* Calcd. (found) for $C_{30}H_{24}BClN_2O_4$: C, 65.91; H, 2.95; N, 5.12. Found: C, 65.66; H, 2.60; N, 5.23. Note that compounds **6a** and **6b** were separated by flash chromatography by using methanol as eluent.

7-Chloro-6-(3-nitrophenyl)quinoline-5,8-quinone (6c). Procedure B was employed to couple 6,7-dichloroquinoline-5,8-dione (130 mg, 0.570 mmol) with 3-nitrophenylboronic acid (140 mg, 0.86 mmol) to supply the title product (**6c**) in 2 h as reddish solid. Yield: 100 mg (56%); mp 355–357°C. UV-Visible λ_{max} (MeOH): 600 (0.265), 620 (0.144), 630 (0.112), 640 (0.077). ^{1}H NMR (400 MHz, DMSO): 8.8 (dd, 1H, $J = 1.2$, 4.4 Hz), 8.7 (dd, 1H, $J = 1.6$, 4.8 Hz), 8.6 (s, 1H), 7.8 (2H), 7.7 (dd, 1H, $J = 3.2$, 7.6 Hz), 7.6 (1H). IR ν_{max} cm^{-1}: 1678.14 (C=O); 1535.40 (NO_2). *Anal.* Calcd. for $C_{15}H_7Cl_2NO_4$: C, 57.25; H, 2.24; N, 8.90. Found: C, 57.29; H, 2.32; N, 8.82.

General procedure for Heck reaction. Iodobenzene (0.003 mol) and 7-chloroquinoline-5,8-dione substrate (0.004 mol), $Pd(OAc)_2$ (3 mol%), PPh_3 (5 mol%), and $NaHCO_3$ (330 mg) were added to a 50-mL round bottom flask containing 4 mL of DMF, and the reaction mixture was refluxed at 180°C. After the reaction completion as indicated by TLC, it was cooled to room temperature. The crude product was extracted with hot toluene (3 × 10 mL). The combined organic extract was dried with sodium sulfate and concentrated in vacuum. The crude product was purified by flash column chromatography on silica gel employing petroleum ether/ethyl acetate.

Synthesis of 7-chloro-6-(4-nitrophenyl)quinoline-5,8-dione (8). The general Heck procedure was used to cross-coupled 4-iodonitrobenzene with 7-chloroquinolin-5.8-dione to obtain titled compound as deep green solid in 3 h. Yield, 620 mg (57%). mp 251–253°C. UV-Visible λ_{max} (MeOH): 450 (3.736), 470 (2.144), 550 (2.812), 500 (2.511). ^{1}H NMR (400 MHz, DMSO): 9.0 (1H), 8.4 (1H), 8.2 (2H), 7.7 (1H), 7.1 (2H). IR ν_{max} cm^{-1}: 1740.00 (C=O); 1373.40 (NO_2). *Anal.* Calcd. for $C_{15}H_7ClN_2O_4$: C, 57.25; H, 2.24; N, 8.90. Found: C, 57.33); H, 2.43; N, 8.78.

Synthesis of 7-chloro-6-phenylquinoline-5,8-dione (9). The general Heck procedure was used to cross-coupled iodobenzene with 7-chloroquinolin-5.8-dione to obtain titled compound as yellowish green solid in 3 h. Yield, 460 mg (63%). mp 288–290°C. UV-Visible λ_{max} (MeOH): 520 (3.665), 490 (3.736), 450 (2.144), 420 (2.511). ^{1}H NMR (400 MHz, DMSO): 8.8 (1H), 8.7 (1H), 8.3 (2H), 8.2 (2H), 7.7 (m, 1H), 7.6 (m, 1H). IR ν_{max} cm^{-1}: 1738.00 (C=O). *Anal.* Calcd. for $C_{15}H_8ClNO_2$: C, 66.81; H, 2.99; N, 5.19. Found: C, 66.85; H, 2.81; N, 5.27.

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