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Metal-Free Organoselenium-Enabled Radical Relay Azidation-Carbocyclization

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Abstract: An organoselenium-enabled radical relay Azidation-carbocyclization reaction for the preparation of azide-substituted quinoline-2,4-diones was developed. A series of previously unknown structurally diverse azide- substituted quinoline-2,4-diones were obtained under metal-free and room-temperature conditions. The key features of this reaction are a broad substrate scope, amenability to gram-scale synthesis, and easy product derivatization. Preliminary mechanistic studies support participation of an organoselenium species in a radical relay pathway.

Keywords: metal free; radical relay; azidation; carbocyclization; organoselenium

Introduction

The selective installation of azide groups in heterocyclic compounds is a priority research topic in organic synthesis, particularly in pharmaceutical discovery.[1] Given the prevalence of azide motifs, the facile late-stage diversification of azide groups to produce value-added chemicals such as amines, imines, amides, aziridines, and triazoles is important but has not yet been fully explored.[2] The development of sustainable and practical methods for forming C–N_3 bonds in synthetic organic chemistry is therefore still necessary.[3] Nitrogen-containing heterocycles are important,[4] particularly structurally diverse quinoline-2,4-diones, which are widely found in numerous natural products, pharmaceuticals, and agrochemicals.[5] Although some methods have been reported for constructing quinoline-2,4-diones,[6,5c] one-pot multi-bond-forming strategies, including multi-component cascade reactions, are the best choices for attaining atom and step economies and therefore for accessing these complex molecules.[7]

Organoselenium compounds are important in organic synthesis, materials, and catalysis.[8,10d] Among these, $RSeN_3$ compounds are attractive potential candidates for use in organic chemistry. However, they were not isolated or characterized until 2004. Based on the successful structural characterization of ionic selenonium azides, i.e., $[R_3Se]N_3$, Klapötke's group first isolated the stable covalent selenium azide $RSeN_3$ and reported its crystallographic data.[9] As part of our ongoing interest in organoselenium chemistry,[10] we speculated that the interaction between $(PhSe)_2$ and an organic azide, or the intermediate $RSeN_3$ formed in situ, would undergo decomposition to form the stable diselane and the corresponding azide radical, instead of producing dinitrogen. Based on these assumptions, we developed a practical organoselenium-enabled radical relay Azidation-carbocyclization for the preparation of azide- substituted quinoline-2,4-diones (Figure 1). Gram-scale synthesis, Huisgen [3 + 2] cyclization, and selective diversification via reduction show the practicability of the reaction and its potential use in industrial applications.

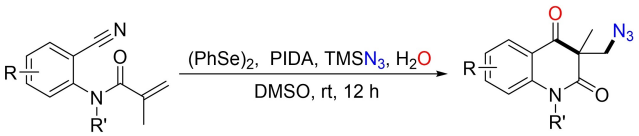


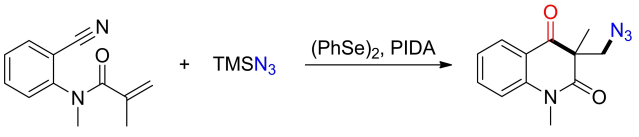
Figure 1. Organic selenium enabled azidation-carbocyclization.

Results and Discussion

Initially, the reaction of *N*-(2-cyanophenyl)-*N*-methylmethacrylamide (**1a**) with azidotrimethylsilane ($TMSN_3$) preactivated by $(PhSe)_2$ and (diacetoxyiodo) benzene (PIDA) was performed in CH_3CN at room temperature. 3-(Azidomethyl)-1,3-dimethylquinoline-2,4(1*H*,3*H*)-dione (**2a**) was obtained in 82% yield after 12 h (Table 1, entry 1). Condition screening showed that the yield of **2a** decreased when the amount of $(PhSe)_2$ or PIDA was decreased, and no **2a** was formed in the absence of $(PhSe)_2$. This indicates that $(PhSe)_2$ is critical for the success of this reaction (Table 1, entries 2–6). The effects of the solvent were then investigated. We found that DMSO, which is less volatile and less toxic than CH_3CN, was the best solvent for this transformation, and its use increased the yield of **2a** to 90%. Other solvents, namely DMF, EtOAc, DCE, 1,4-dioxane, and DCM, gave poorer results (Table 1, entries 7–12). In addition, the reaction yield was temperature sensitive and the yield of **2a** decreased to 74% at 60 °C (Table 1, entry 13). Further condition optimization showed that other diselenides, namely $(4\text{-BrPhSe})_2$ and $(4\text{-MePhSe})_2$, gave decreased yields (Table 1, entries 14 and 15).

The scope with respect to *N*-(2-cyanophenyl)-*N*-methylmethacrylamides **1** is wide. As shown in Table 2, **1** with electron-withdrawing F, Cl, and Br substituents and electron-donating Me and MeO substituents at various positions on the phenyl ring were successfully converted to the desired products **2b**–**2n**. Note that the yield was affected by the electron density of the benzene ring. When 5-Cl- or 8-Cl-substituted substrates were used, the yields decreased to 43% and 16% of **2c** and **2n**, respectively. Multi-substituted substrates reacted smoothly with $TMSN_3$ to provide **2o** and **2p** in 45% and 87% yields, respectively. The heterocyclic substrate pyridineacrylamide (**1q**), was also compatible with this reaction, and delivered **2q** in 91% yield. The use of a benzyl

Table 1. Condition screenings.[a,b]

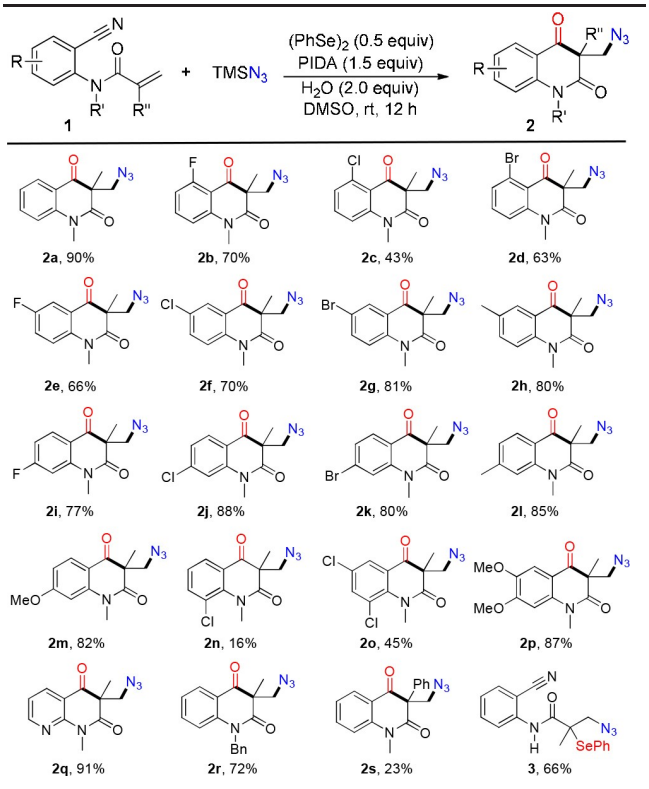


1a 2a

Entry	$(PhSe)_2$ (mol%)	PIDA (equiv.)	Solvent (mL)	Yield (%)[b]
1	50	1.5	CH_3CN	82
2	10	1.5	CH_3CN	21
3	20	1.5	CH_3CN	39
4	none	1.5	CH_3CN	0
5	50	1.0	CH_3CN	68
6	50	0.5	CH_3CN	49
7	50	1.5	DMF	41
8	50	1.5	EtOAc	43
9	50	1.5	DCE	59
10	50	1.5	DMSO	90
11	50	1.5	1,4-dioxane	33
12	50	1.5	DCM	49
13	50	1.5	DMSO	74[c]
14	50	1.5	DMSO	72[d]
15	50	1.5	DMSO	36[e]

[a] Reactions were performed with **1a** (0.3 mmol), $TMSN_3$ (3.0 equiv.), $(PhSe)_2$ (50 mol%), PIDA (1.5 equiv.), and H_2O (2.0 equiv.) in a solvent (2 mL) at room temperature.
[b] Yield of isolated product.
[c] Reaction performed at 60 °C.
[d] $(4\text{-BrPhSe})_2$ (50 mol%) was added instead of $(PhSe)_2$.
[e] $(4\text{-MePhSe})_2$ (50 mol%) was added instead of $(PhSe)_2$.

Table 2. Substrate scope.[a,b]

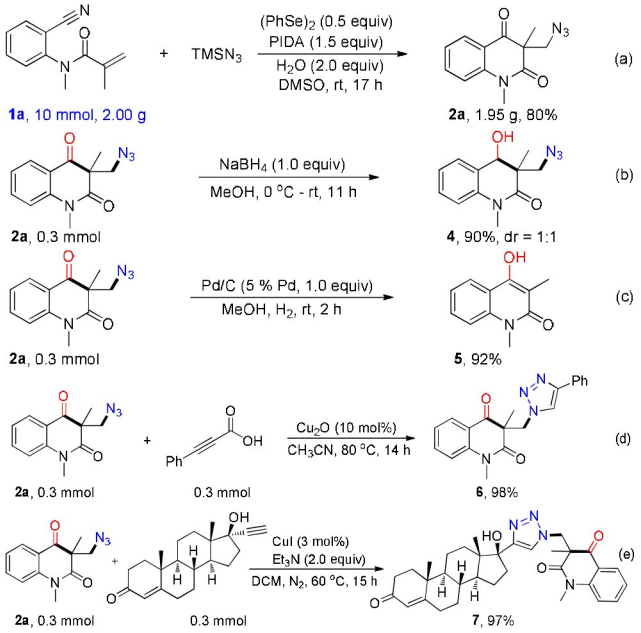


[a] Reaction conditions: **1** (0.3 mmol), $TMSN_3$ (3.0 equiv.), $(PhSe)_2$ (0.5 equiv.), PIDA (1.5 equiv.), and H_2O (2.0 equiv.) in DMSO (2.0 mL) at room temperature for 12 h.
[b] Yield of isolated product.

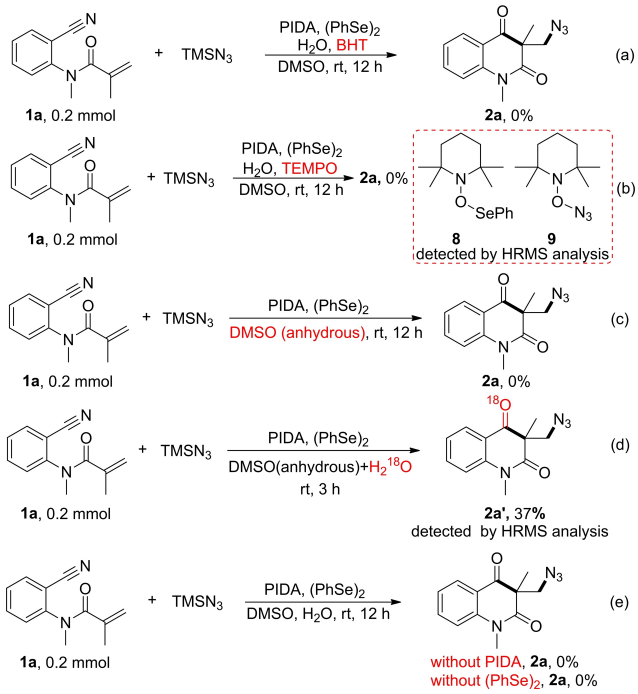
protecting group on the nitrogen atom was also examined and found to be compatible with this transformation; the desired **2 r** was obtained in 72% yield. Substrate **1 s**, which has a phenyl group on the double bond (R″ = Ph), gave **2 s** in 23% yield. This low yield can be attributed to steric effects. The unprotected substrate (R′ = H) was also tested. Only trace amounts of the quinoline-2,4-dione were detected; Azidation-selenation occurred to give the vicinal azide and seleno-substituted product **3** in 66% yield.

The reaction was scaled up to evaluate the synthetic utility of this protocol. When the reaction of **1 a** was performed on a 10 mmol scale (Scheme 1a), **2 a** was obtained in 80% isolated yield. This enabled further synthetic manipulations. Compound **2 a** was selectively reduced to give **4** and **5** in excellent yields. These are attractive as valuable cores for further derivatization (Scheme 1b and 1c).[11] Given the prevalence of azide motifs, further Huisgen [3 + 2] diversifications of the azide group with 3-phenylpropiolic acid and ethisterone were performed with **2 a**. The corresponding triazoles **6** and **7** were obtained in 98% and 97% yields, respectively (Scheme 1d and 1e).

After investigation of the substrate scope and synthetic applications, control experiments were performed to clarify the mechanism (see the Supporting Information for details). The addition of 2,6-di-*tert*-butyl-4-methylphenol (BHT, 2.0 equiv.) and 2,2,6,6-tetramethylpiperidine *N*-oxide (TEMPO, 2.0 equiv.) as radical scavengers under the standard reaction conditions significantly inhibited the reaction; the radical-trapping products **8** and **9** were detected by HRMS (Scheme 2a and 2b). These results indicate that a



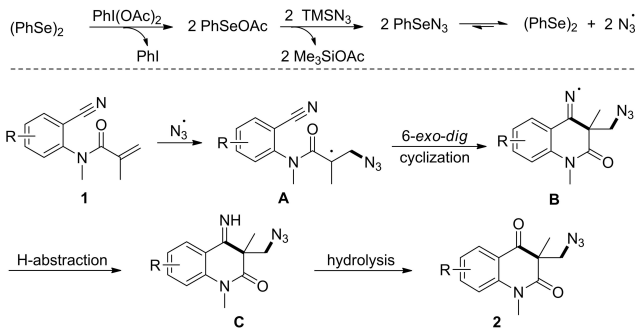
Scheme 1. Application investigation.



Scheme 2. Mechanism study.

radical pathway was involved in these systems. A control experiment showed that in the absence of H_2O the desired **2 a** was not detected (Scheme 2c). An ^{18}O-labeling experiment was then performed. The desired product **2 a′** was obtained in 37% yield after 3 h; the identity of **2 a′** was confirmed by HRMS (Scheme 2d). Further control experiments showed that $(PhSe)_2$ and PIDA are both necessary for this Azidation-carbocyclization reaction (Scheme 2e).

On the basis of the control experiment results and a literature survey,[12,13] a plausible mechanism for the formation of product **2** is proposed, as shown in Scheme 3, First, oxygenolysis between $(PhSe)_2$ and PIDA generates PhSeOAc, which is similar to PhSeX (X = Cl, Br). Then the selenenyl azide $PhSeN_3$ is formed in situ as a suitable precursor for conversion to the corresponding azide radical and diselane. The azide



Scheme 3. Proposed reaction mechanism.

radical then attacks the activated alkenyl moiety in substrate **1** to give radical intermediate **A**. Subsequent rapid intramolecular 6-*exo-dig* cyclization of intermediate **A** yields imine radical **B**, which undergoes hydrogen abstraction to give imine **C**. Finally, imine **C** is hydrolyzed by H_2O to give **2**.

Conclusion

In this work, an organoselenium-enabled radical relay Azidation-carbocyclization was developed and used to construct a series of structurally diverse azide- substituted quinoline-2,4-diones. With the unprotected substrate (R′ = H) radical Azidation-selenation occurred to give vicinal azide and seleno-substituted products. These reactions were conducted at room temperature under metal-free conditions. Ease of scaleup and potential product derivatization make this strategy attractive for the preparation of other valuable molecules. A possible mechanism, which involves radical formation, was proposed on the basis of the results of control experiments. A selenenyl azide ($PhSeN_3$) was proposed as a suitable precursor for conversion to the corresponding azide radical. Further research on the use of this protocol in the synthesis of bioactive molecules and a detailed mechanistic investigation are currently in progress.

Experimental Section

General Remarks: All reagents were purchased from commercial sources and used without further purification. ^{1}H NMR, ^{13}C NMR spectra were recorded on a Bruker Ascend™ 400 or a Bruker Ascend™ 600 spectrometer in deuterated solvents containing TMS as an internal reference standard. All high-resolution mass spectra (HRMS) were measured on a mass spectrometer by using electrospray ionization orthogonal acceleration time-of-flight (ESI-OA-TOF), and the purity of all samples used for HRMS (>95%) was confirmed by ^{1}H NMR and ^{13}C NMR spectroscopic analysis. Melting points were measured on a melting point apparatus equipped with a thermometer and were uncorrected. All the reactions were monitored by thin-layer chromatography (TLC) using GF254 silica gel-coated TLC plates. Purification by flash column chromatography was performed over SiO_2 (silica gel 200–300 mesh).

The General Procedure for the Synthesis of 2

To a reaction tube were add acrylamides 1 (0.3 mmol), diphenyl diselenide (50 mol%, 0.15 mmol, 46.8 mg), (diacetoxyiodo) benzene (PIDA) (1.5 equiv., 0.45 mmol, 145 mg), $TMSN_3$ (93%, 3.0 equiv., 0.9 mmol, 127 μL) and H_2O (2.0 equiv.) in DMSO (2 mL). Then the tube was stirred at room temperature for 12 hours until complete consumption of starting material as monitored by TLC analysis. After the completion of the reaction, the mixture was quenched by H_2O (15 mL) and extracted with CH_2Cl_2 (3 × 5 mL). Then the organic solvent was concentrated in vacuo. The residue was purified by flash column chromatography with Ethyl acetate and Petroleum ether as eluent to give 2.

3-(Azidomethyl)-1,3-dimethylquinoline-2,4(1*H*,3*H*)-dione (2 a). Red liquid (66 mg, 90% yield). R_f (Petroleum ether: Ethyl acetate = 3:1): 0.44. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.03 (d, J = 7.8 Hz, 1H), 7.65 (t, J = 7.8 Hz, 1H), 7.19 (t, J = 7.4 Hz, 2H), 3.97 (s, 2H), 3.49 (s, 3H), 1.38 (s, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 195.47, 171.89, 143.19, 136.60, 128.31, 123.40, 119.77, 115.07, 57.11, 55.68, 29.97, 23.21. HRMS (ESI) calcd for $C_{12}H_{12}N_4O_2$ $[M+Na]^+$: 267.0632, found: 267.0631.

3-(Azidomethyl)-5-fluoro-1,3-dimethylquinoline-2,4(1*H*,3*H*)-dione (2 b). Red liquid (55 mg, 70% yield). R_f (Petroleum ether: Ethyl acetate = 3:1): 0.29. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.60–7.56 (m, 1H), 6.99 (d, J = 8.5 Hz, 1H), 6.91–6.88 (m, 1H), 3.97 (d, J = 11.5 Hz, 1H), 3.91 (d, J = 11.5 Hz, 1H), 3.49 (s, 3H), 1.40 (s, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 192.59, 171.27, 162.23 (d, J = 267.1 Hz), 144.17 (d, J = 3.0 Hz), 136.64 (d, J = 11.9 Hz), 111.61 (d, J = 21.2 Hz), 110.85 (d, J = 3.3 Hz), 109.72 (d, J = 9.5 Hz), 57.99, 54.80, 30.79, 22.73. **^{19}F NMR** (565 MHz, $CDCl_3$) δ −119.02. HRMS (ESI) calcd for $C_{12}H_{11}FN_4O_2$ $[M+Na]^+$: 285.0758, found: 285.0754.

3-(Azidomethyl)-5-chloro-1,3-dimethylquinoline-2,4(1*H*,3*H*)-dione (2 c). Red liquid (36 mg, 43% yield). R_f (Petroleum ether: Ethyl acetate = 3:1): 0.31. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.49 (t, J = 8.2 Hz, 1H), 7.24–7.23 (m, 1H), 7.13–1.11 (m, 1H), 3.94 (d, J = 11.7 Hz, 1H), 3.90 (d, J = 11.7 Hz, 1H), 3.48 (s, 3H), 1.39 (s, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 193.30, 170.64, 144.51, 135.62, 134.69, 126.76, 118.18, 113.87, 58.25, 54.19, 30.87, 21.77. HRMS (ESI) calcd for $C_{12}H_{11}ClN_4O_2$ $[M+Na]^+$: 301.0463, found: 301.0460.

3-(Azidomethyl)-5-bromo-1,3-dimethylquinoline-2,4(1*H*,3*H*)-dione (2 d). Red liquid (61 mg, 63% yield). R_f (Petroleum ether: Ethyl acetate = 3:1): 0.34. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.45–7.43 (m, 1H), 7.38 (t, J = 8.1 Hz, 1H), 7.16 (d, J = 8.3 Hz, 1H), 3.92 (d, J = 11.7 Hz, 1H), 3.88 (d, J = 11.7 Hz, 1H), 3.46 (s, 3H), 1.37 (s, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 193.60, 170.48, 144.59, 134.86, 130.33, 123.13, 119.37, 114.60, 58.03, 54.13, 30.81, 21.59. HRMS (ESI) calcd for $C_{12}H_{11}BrN_4O_2$ $[M+Na]^+$: 344.9958, found: 344.9955.

3-(Azidomethyl)-6-fluoro-1,3-dimethylquinoline-2,4(1*H*,3*H*)-dione (2 e). Red liquid (52 mg, 66% yield). R_f (Petroleum ether: Ethyl acetate = 3:1): 0.34. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.72–7.70 (m, 1H), 7.39–7.36 (m, 1H), 7.19–7.17 (m, 1H), 4.00–3.95 (m, 2H), 3.50 (s, 3H), 1.39 (s, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 194.76, 171.47, 158.60 (d, J = 245.6 Hz), 139.64, 123.56 (d, J = 23.8 Hz), 120.94 (d, J = 5.9 Hz), 116.93 (d, J = 7.5 Hz), 114.03 (d, J = 23.2 Hz), 57.00, 55.81, 30.26, 23.14. HRMS (ESI) calcd for $C_{12}H_{11}FN_4O_2$ $[M+Na]^+$: 285.0758, found: 285.0755.

3-(Azidomethyl)-6-chloro-1,3-dimethylquinoline-2,4(1*H*,3*H*)-dione (2 f). White soild (58.5 mg, 70% yield). R_f (Petroleum ether: Ethyl acetate = 3:1) 0.46. mp 109–110 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.96 (d, J = 2.6 Hz, 1H), 7.59–7.56 (m, 1H), 7.12 (d, J = 8.9 Hz, 1H), 3.95 (s, 2H), 3.46 (s, 3H), 1.36 (s, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 194.51, 171.58, 141.71, 136.17, 129.27, 127.74, 120.75, 116.71, 57.18, 55.74, 30.16,

23.10. HRMS (ESI) calcd for $C_{12}H_{11}ClN_4O_2$ $[M+Na]^+$: 301.0463, found: 301.0460.

3-(Azidomethyl)-6-bromo-1,3-dimethylquinoline-2,4(1*H*,3*H*)-dione (2 g). White soild (78.5 mg, 81% yield). R_f (Petroleum ether: Ethyl acetate = 3:1): 0.43. mp 106–107 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.12 (d, J = 2.5 Hz, 1H), 7.74–7.72 (m, 1H), 7.08 (d, J = 8.8 Hz, 1H), 3.97 (s, 2H), 3.48 (s, 3H), 1.39 (s, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 194.40, 171.58, 142.17, 139.03, 130.77, 121.05, 116.98, 116.49, 57.21, 55.74, 30.13, 23.11. HRMS (ESI) calcd for $C_{12}H_{11}BrN_4O_2$ $[M+Na]^+$: 344.9958, found: 344.9958.

3-(Azidomethyl)-1,3,6-trimethylquinoline-2,4(1*H*,3*H*)-dione (2 h). Red liquid (62 mg, 80% yield). R_f (Petroleum ether: Ethyl acetate = 3:1): 0.41. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.83 (d, J = 1.8 Hz, 1H), 7.47–7.45 (m, 1H), 7.08 (d, J = 8.4 Hz, 1H), 3.97 (s, 2H), 3.48 (s, 3H), 2.36 (s, 3H), 1.38 (s, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 195.70, 171.77, 141.04, 137.38, 133.20, 128.23, 119.59, 115.04, 56.99, 55.75, 29.94, 23.22, 20.33. HRMS (ESI) calcd for $C_{13}H_{14}N_4O_2$ $[M+Na]^+$: 281.1009, found: 281.1004.

3-(Azidomethyl)-7-fluoro-1,3-dimethylquinoline-2,4(1*H*,3*H*)-dione (2 i). Yellow liquid (60.5 mg, 77% yield). R_f (Petroleum ether: Ethyl acetate = 3:1): 0.51. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.12–8.06 (m, 1H), 6.93–6.87 (m, 2H), 4.01–3.95 (m, 2H), 3.48 (s, 3H), 1.40 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 193.93, 172.09, 167.85 (d, J = 256.8 Hz), 145.55 (d, J = 11.7 Hz), 131.36 (d, J = 11.4 Hz), 116.39 (d, J = 2.3 Hz), 110.83 (d, J = 22.4 Hz), 102.74 (d, J = 27.6 Hz), 56.98, 56.87, 30.14, 23.20. **^{19}F NMR** (565 MHz, $CDCl_3$) δ −98.04. HRMS (ESI) calcd for $C_{12}H_{11}FN_4O_2$ $[M+Na]^+$: 285.0758, found: 285.0753.

3-(Azidomethyl)-7-chloro-1,3-dimethylquinoline-2,4(1*H*,3*H*)-dione (2 j). Yellow liquid (73.5 mg, 88% yield). R_f (Petroleum ether: Ethyl acetate = 3:1): 0.56. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.97 (d, J = 8.3 Hz, 1H), 7.19–7.16 (m, 2H), 3.97 (d, J = 1.2 Hz, 2H), 3.48 (s, 3H), 1.38 (s, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 194.38, 171.95, 144.16, 142.99, 129.73, 123.68, 118.13, 115.41, 57.14, 55.85, 30.11, 23.14. HRMS (ESI) calcd for $C_{12}H_{11}ClN_4O_2$ $[M+Na]^+$: 301.0463, found: 301.0460.

3-(Azidomethyl)-7-bromo-1,3-dimethylquinoline-2,4(1*H*,3*H*)-dione (2 k). Yellow liquid (77.5 mg, 80% yield). R_f (Petroleum ether: Ethyl acetate = 3:1): 0.55. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.88 (d, J = 8.3 Hz, 1H), 7.36 (d, J = 1.6 Hz, 1H), 7.34–7.32 (m, 1H), 3.96 (s, 2H), 3.48 (s, 3H), 1.38 (s, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 194.59, 171.90, 144.02, 134.08, 131.76, 129.63, 127.21, 126.65, 118.48, 118.35, 57.18, 55.83, 30.12, 23.12. HRMS (ESI) calcd for $C_{12}H_{11}BrN_4O_2$ $[M+Na]^+$: 344.9958, found: 344.9954.

3-(Azidomethyl)-1,3,7-trimethylquinoline-2,4(1*H*,3*H*)-dione (2 l). Yellow liquid (65.8 mg, 85% yield). R_f (Petroleum ether: Ethyl acetate = 3:1): 0.50. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.94–7.92 (m, 1H), 7.01 (d, J = 7.9 Hz, 1H), 6.98 (s, 1H), 3.99–3.94 (m, 2H), 3.49 (d, J = 1.2 Hz, 3H), 2.46 (s, 3H), 1.38 (s, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 195.02, 172.22, 148.16, 143.27, 128.40, 124.44, 117.64, 115.52, 56.86, 55.86, 29.91, 23.28, 22.48. HRMS (ESI) calcd for $C_{13}H_{14}N_4O_2$ $[M+Na]^+$: 281.1009, found: 281.1001.

3-(Azidomethyl)-7-methoxy-1,3-dimethylquinoline-2,4(1*H*,3*H*)-dione (2 m). Red liquid (67.5 mg, 82% yield). R_f (Petroleum ether: Ethyl acetate = 3:1): 0.33. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.01 (d, J = 8.7 Hz, 1H), 6.72–6.70 (m, 1H), 6.62 (d, J = 2.2 Hz, 1H), 3.97 (d, J = 11.3 Hz, 1H), 3.93 (d, J = 11.3 Hz, 1H), 3.91 (s, 3H), 3.46 (s, 3H), 1.37 (s, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 193.75, 172.49, 166.37, 145.20, 130.88, 113.67, 108.54, 101.11, 56.49, 56.05, 55.85, 29.91, 23.37. HRMS (ESI) calcd for $C_{13}H_{14}N_4O_3$ $[M+Na]^+$: 267.0710, found: 297.0710.

3-(Azidomethyl)-8-chloro-1,3-dimethylquinoline-2,4(1*H*,3*H*)-dione (2 n). White soild (13 mg, 16% yield). R_f (Petroleum ether: Ethyl acetate = 3:1): 0.50. mp 80–81 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.84–7.82 (m, 1H), 7.66–7.64 (m, 1H), 7.18 (t, J = 7.8 Hz, 1H), 3.92 (d, J = 11.8 Hz, 1H), 3.87 (d, J = 11.8 Hz, 1H), 3.62 (s, 3H), 1.37 (s, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 195.30, 171.94, 141.72, 138.60, 126.53, 125.27, 124.78, 122.87, 57.76, 54.05, 37.48, 22.05. HRMS (ESI) calcd for $C_{12}H_{11}ClN_4O_2$ $[M+Na]^+$: 301.0463, found: 301.0462.

3-(Azidomethyl)-6,8-dichloro-1,3-dimethylquinoline-2,4(1*H*,3*H*)-dione (2 o). Colourless liquid (42 mg, 45% yield). R_f (Petroleum ether: Ethyl acetate = 5:1): 0.48. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.79 (d, J = 2.5 Hz, 1H), 7.64 (d, J = 2.5 Hz, 1H), 3.92 (d, J = 11.8 Hz, 1H), 3.86 (d, J = 11.8 Hz, 1H), 3.61 (s, 3H), 1.37 (s, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 194.30, 171.65, 140.43, 137.66, 130.43, 127.29, 126.27, 125.22, 123.68, 57.79, 54.04, 37.42, 22.02. HRMS (ESI) calcd for $C_{12}H_{10}Cl_2N_4O_2$ $[M+Na]^+$: 335.0073, found: 334.0070.

3-(Azidomethyl)-6,7-dimethoxy-1,3-dimethylquinoline-2,4(1*H*,3*H*)-dione (2 p). Yellow solid (79.4 mg, 87% yield). R_f (Petroleum ether: Ethyl acetate = 3:1): 0.11. mp 138–139 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.46 (s, 1H), 6.61 (s, 1H), 4.00 (s, 3H), 3.96 (d, J = 11.3 Hz, 1H), 3.93 (d, J = 11.3 Hz, 1H), 3.90 (s, 3H), 3.49 (s, 3H), 1.37 (s, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 192.95, 171.43, 155.11, 144.45, 138.53, 111.40, 107.88, 97.26, 55.37, 55.32, 55.27, 55.23, 28.96, 22.54. HRMS (ESI) calcd for $C_{14}H_{16}N_4O_4$ $[M+Na]^+$: 327.1064, found: 327.1069.

3-(Azidomethyl)-1,3-dimethyl-1,8-naphthyridine-2,4(1*H*,3*H*)-dione (2 q). Yellow soild (67 mg, 91% yield). R_f (Petroleum ether: Ethyl acetate = 3:1) 0.45. mp 56–57 °C. **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.65–8.64 (m, 1H), 8.30–8.28 (m, 1H), 7.18–7.15 (m, 1H), 4.02 (d, J = 11.4 Hz, 1H), 3.98 (d, J = 11.4 Hz, 1H), 3.63 (s, 3H), 1.43 (s, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 195.11, 172.52, 154.71, 154.26, 136.76, 119.10, 115.02, 57.28, 56.01, 28.87, 23.07. HRMS (ESI) calcd for $C_{11}H_{11}N_5O_4$ $[M+Na]^+$: 268.0805, found: 268.0808.

3-(Azidomethyl)-1-benzyl-3-methylquinoline-2,4(1*H*,3*H*)-dione (2 r). White soild (69 mg, 72% yield). R_f (Petroleum ether: Ethyl acetate = 3:1): 0.54. mp 155–156 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.06–8.04 (m, 1H), 7.51–7.48 (m, 1H), 7.34 (t, J = 7.5 Hz, 2H), 7.26 (t, J = 8.6 Hz, 3H), 7.15 (t, J = 7.5 Hz, 1H), 7.06 (d, J = 8.4 Hz, 1H), 5.39–5.27 (m, 2H), 4.07 (s, 2H), 1.50 (s, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 195.38, 172.36, 142.46, 136.51, 135.67, 129.06, 128.43, 127.55, 126.26, 123.50, 120.01, 116.02, 57.17, 55.93, 46.31, 23.32. HRMS (ESI) calcd for $C_{18}H_{16}N_4O_2$ $[M+Na]^+$: 343.1163, found: 343.1164.

3-(Azidomethyl)-1-methyl-3-phenylquinoline-2,4(1*H*,3*H*)-dione (2 s). Red liquid (21 mg, 23% yield). R_f (Petroleum ether:

Ethyl acetate = 3:1): 0.50. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.01–7.99 (m, 1H), 7.59–7.56 (m, 1H), 7.30–7.27 (m, 3H), 7.23–7.22 (m, 2H), 7.16–7.13 (m, 1H), 7.11 (d, J = 8.3 Hz, 1H), 4.26 (d, J = 11.6 Hz, 1H), 4.16 (d, J = 11.6 Hz, 1H), 3.58 (s, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 193.16, 169.55, 142.63, 136.20, 134.01, 129.33, 128.84, 128.26, 126.71, 123.47, 121.37, 115.03, 66.95, 54.96, 30.28. HRMS (ESI) calcd for $C_{17}H_{14}N_4O_2$ $[M+Na]^+$: 329.1009, found: 329.1005.

3-Azido-*N*-(2-cyanophenyl)-2-methyl-2-(phenylselanyl) propanamide (3). Yellow soild (76 mg, 66% yield). R_f (Petroleum ether: Ethyl acetate = 5:1): 0.36. mp 65–66 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.75 (s, 1H), 8.31 (d, J = 8.4 Hz, 1H), 7.62–7.57 (m, 4H), 7.42–7.38 (m, 1H), 7.32–7.29 (m, 2H), 7.21–7.18 (m, 1H), 3.92 (d, J = 12.6 Hz, 1H), 3.69 (d, J = 12.6 Hz, 1H), 1.70 (s, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 170.65, 140.34, 137.58, 134.20, 132.28, 129.98, 129.44, 125.32, 124.35, 120.95, 116.26, 102.26, 58.12, 51.31, 22.54. HRMS (ESI) calcd for $C_{17}H_{15}N_5OSe$ $[M+Na]^+$: 408.0334, found: 408.0331.

3-(Azidomethyl)-4-hydroxy-1,3-dimethyl-3,4-dihydroquinolin-2(1*H*)-one (4). Yellow liquid (33.2 mg, 45% yield). R_f (Petroleum ether: Ethyl acetate = 1:1): 0.33. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.53–7.51 (m, 1H), 7.33–7.31 (m, 1H), 7.17–7.14 (m, 1H), 6.99 (d, J = 7.8 Hz, 1H), 5.10 (d, J = 5.9 Hz, 1H), 3.95 (d, J = 12.1 Hz, 1H), 3.47 (d, J = 12.1 Hz, 1H), 3.36 (s, 3H), 2.79 (d, J = 6.1 Hz, 1H), 0.94 (s, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 171.65, 137.29, 128.56, 127.57, 125.47, 123.73, 114.16, 67.93, 54.15, 47.82, 30.13, 14.04. HRMS (ESI) calcd for $C_{12}H_{14}N_4O_2$ $[M+Na]^+$: 269.1009, found: 269.1008.

White solid (33.2 mg, 45% yield). R_f (Petroleum ether: Ethyl acetate = 1:1): 0.25. mp 84–85 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.40–7.36 (m, 1H), 7.32–7.31 (m, 1H), 7.11–7.08 (m, 1H), 7.03 (d, J = 8.1 Hz, 1H), 4.51 (s, 1H), 3.94 (d, J = 12.3 Hz, 1H), 3.70 (d, J = 12.3 Hz, 1H), 3.36 (s, 3H), 2.41 (s, 1H), 1.03 (s, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 171.82, 138.79, 129.91, 129.04, 125.40, 123.46, 114.85, 72.04, 54.21, 46.55, 29.67, 18.76. HRMS (ESI) calcd for $C_{12}H_{14}N_4O_2$ $[M+Na]^+$: 269.1009, found: 269.1007.

4-Hydroxy-1,3-dimethylquinolin-2(1*H*)-one (5). White solid (52 mg, 92% yield). R_f (Petroleum ether: Ethyl acetate = 1:1): 0.19. mp 212–213 °C. **^{1}H NMR** (600 MHz, DMSO) δ 10.09 (s, 1H), 7.97 (d, J = 7.9 Hz, 1H), 7.57 (t, J = 7.8 Hz, 1H), 7.47 (d, J = 8.3 Hz, 1H), 7.24 (t, J = 7.5 Hz, 1H), 3.59 (s, 3H), 2.05 (s, 3H). **^{13}C NMR** (150 MHz, DMSO) δ 163.65, 156.37, 138.46, 130.71, 123.25, 121.87, 116.57, 114.75, 106.82, 29.71, 10.61. HRMS (ESI) calcd for $C_{11}H_{11}NO_2$ $[M+Na]^+$: 212.0682, found: 212.0683.

1,3-Dimethyl-3-((4-phenyl-1*H*-1,2,3-triazol-1-yl)methyl) quinoline-2,4(1*H*,3*H*)-dione (6). White solid (102 mg, 98% yield). R_f (Petroleum ether: Ethyl acetate = 1:1): 0.40. mp 151–152 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.03–8.02 (m, 1H), 7.80 (s, 1H), 7.71 (d, J = 7.7 Hz, 2H), 7.61–7.56 (m, 1H), 7.34 (t, J = 7.6 Hz, 2H), 7.28–7.24 (m, 1H), 7.16–7.12 (m, 2H), 5.04 (s, 2H), 3.44 (s, 3H), 1.55 (s, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 194.24, 171.63, 147.72, 143.10, 136.49, 130.47, 128.75, 128.51, 128.08, 125.64, 123.30, 120.72, 119.43, 115.21, 56.29, 53.28, 30.00, 24.27. HRMS (ESI) calcd for $C_{20}H_{18}N_4O_2$ $[M+Na]^+$: 369.1322, found: 369.1317.

3-((5-((8R,9S,10R,13S,14S,17S)-17-Hydroxy-10,13-dimethyl-3-oxo-2,3,6,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1*H*-cyclopenta[a]phenanthren-17-yl)-1*H*-1,2,3-triazol-1-yl) methyl)-1,3-dimethylquinoline-2,4(1*H*,3*H*)-dione (7). White solid (162 mg, 97% yield). R_f (Ethyl acetate): 0.35. mp 239–240 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.05–7.99 (m, 1H), 7.64–7.62 (m, 1H), 7.39 (d, J = 3.2 Hz, 1H), 7.18–7.16 (m, 2H), 5.70 (d, J = 6.2 Hz, 1H), 5.03–4.96 (m, 2H), 3.46 (d, J = 5.8 Hz, 3H), 2.73 (d, J = 30.3 Hz, 1H), 2.43–2.20 (m, 5H), 2.03 (d, J = 4.4 Hz, 1H), 1.95–1.93 (m,1H), 1.86–1.63 (m, 4H), 1.56 (d, J = 5.8 Hz, 3H), 1.44–1.31 (m, 5H), 1.15 (d, J = 4.1 Hz, 3H), 0.99 (d, J = 3.7 Hz, 3H), 0.66–0.59 (m, 1H), 0.33–0.22 (m, 1H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 199.54, 194.25, 194.13, 171.59, 171.49, 171.29, 153.46, 153.40, 143.13, 143.09, 136.55, 136.51, 128.55, 128.52, 123.79, 123.33, 123.32, 122.18, 119.37, 115.15, 115.14, 81.96, 81.89, 56.35, 56.29, 53.30, 53.29, 53.09, 53.06, 48.75, 48.73, 46.74, 46.73, 38.56, 37.62, 37.56, 36.20, 36.19, 35.61, 35.59, 33.94, 32.81, 32.51, 32.50, 31.49, 30.00, 24.33, 24.30, 23.63, 23.57, 20.54, 20.51, 17.38, 17.36, 14.16. HRMS (ESI) calcd for $C_{33}H_{40}N_4O_4$ $[M+Na]^+$: 579.2942, found: 579.2940.

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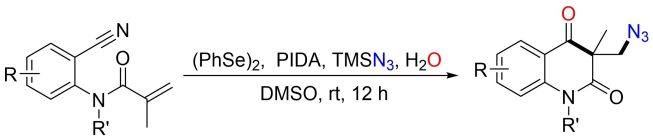
RESEARCH ARTICLE

Metal-Free Organoselenium-Enabled Radical Relay Azidation-Carbocyclization



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