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由 *N*-(2-丙炔基)苯胺和磺酰氯直接合成 3-砜基喹啉

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摘要 3-砜基喹啉广泛存在于多种生物活性分子和合成药物中. 报道了一种温和条件下, 以 *N*-炔丙基苯胺和磺酰氯为原料, 通过一锅法连续肼化/砜化/环化反应高效构建 3-砜基喹啉类化合物的方法. 该方法不仅可以进行克级合成, 还可用于药物分子砜基衍生物的合成.

关键词 3-砜基喹啉; *N*-炔丙基苯胺; 磺酰氯; 一锅法; 环化反应

Direct Synthesis of 3-Sulfonylquinolines from *N*-Propargylanilines with Sulfonyl Chlorides

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Abstract 3-Sulfonylquinolines broadly exist in plenty of bioactive molecules and synthetic pharmaceuticals. Herein, an efficient and facile method for direct synthesizing various 3-sulfonylquinolines through one-pot sequential hydrazination/sulfonylation/cyclization reaction from *N*-propargylanilines and sulfonyl chlorides under mild reaction conditions is reported. This procedure not only can be performed on a gram scale but also be applicable to prepare marketed pharmaceutical derivative.

Keywords 3-sulfonylquinolines; *N*-propargylanilines; sulfonyl chlorides; one pot; cyclization reaction

1 Introduction

N-Heterocyclic sulfones are found in a large variety of naturally occurring products and synthetic pharmaceuticals. They also serve as important building blocks and versatile synthetic intermediates in organic synthetic chemistry.[1] Among various *N*-heterocyclic sulfones, 3-sulfonylquinolines and their analogues are of particular interest, as they have shown a broad spectrum of pharmacological and biological activities.[2] Therefore, considerable effort has been made towards developing novel and practical synthetic strategies for such molecules.

Given the easy availability of *N*-propargylamines,[3] the cascade sulfonylation[4] and cyclization[5] of *N*-propargylanilines was considered as a facile strategy for constructing 3-sulfonylquinoline compounds. As a consequence, the cascade reaction of *N*-propargylanilines with various sulfonating agents (sodium sulfinates,[6] sulfinic acids[7] and sulfonyl hydrazides[8], *etc*.[9]) have been developed by several groups over the past years (Scheme 1a). Although these methodologies are useful, the sulfonating agents have limited commercial availability, and are too expensive for larger-scale operations. They require a pre-functionalization process of sulfonyl chlorides, resulting in the inevitable formation of large amounts of chemical waste during the isolation and purification steps.[10] However, to the best of our knowledge, the direct synthesis of 3-sulfonylquinolines with sulfonyl chlorides as the sulfonylation reagents

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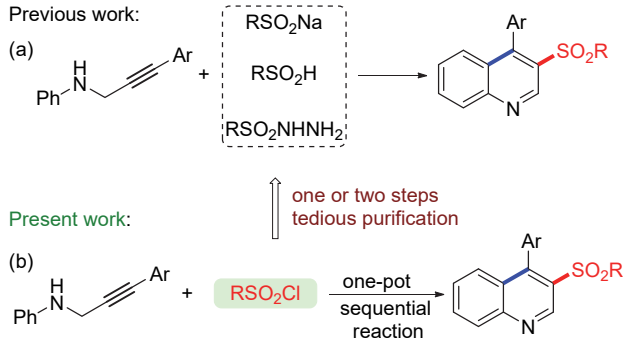
has not been studied, in spite of the fact that this strategy has the advantages of abundance and wide commercial availability of sulfonyl chlorides at competitive prices. Hence, there is a demand for a simple and practical synthetic protocol for various 3-sulfonylquinolines.



Scheme 1 Synthesis of 3-sulfonylquinolines

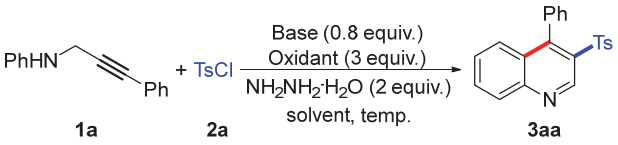
The one-pot sequential reaction enables the employment of abundant and commercially available reagents as the starting materials for the construction of functionalized *N*-heterocycles, which is considered as an atom- and step-economic strategy in current synthetic chemistry.[11] This synthetic strategy not only reduces the production cost and chemical waste (generated during the separation and purification processes) but also increases operational safety.

As a continuation of our interest in green synthesis,[12] a facile and efficient method for the direct synthesis of 3-sulfonylquinolines from *N*-propargylamines and sulfonyl chlorides through one-pot sequential reaction is herein presented (Scheme 1b).

2 Results and discussion

At the outset, the reaction of *N*-(3-phenylprop-2-yn-1-yl)aniline (**1a**) and 4-tosyl chloride (**2a**) was selected as a model reaction for the optimization process. Treatment of **1a** and **2a** with *tert*-butyl hydroperoxide (TBHP) as an oxidant, K_2CO_3 as a base and 1,2-dichloroethane (DCE) as the solvent at 80 ℃ could give the desired 4-phenyl-3-tosylquinoline (**3aa**) in 58% NMR yield. Subsequently, a variety of oxidants were investigated (Table 1, Entries 2～6). However, di-*tert*-butyl peroxyde (DTBP), benzoyl peroxide (BPO), H_2O_2, $PhI(OAc)_2$ and $K_2S_2O_8$ only afforded **3aa** in very low yield. The examinations on the effect of inorganic (Table 1, Entries 7 and 8) and organic bases (Table 1, Entries 9～11) indicated that Cs_2CO_3 was the most suitable base for this transformation, which provided 90% NMR yield of **3aa** (Table 1, Entry 8). After screening of various solvents (Table 1, Entries 12～16), DCM (dichloromethane) was found to be the most appropriate solvent for this reaction. Varying the reaction temperatures did not lead to any further improvement of reaction efficiency (Table 1, Entries 17 and 18). Control experiments suggested that oxidant, base and hydrazine hydrate were essential for the success of the current reaction (Table 1, Entries 19～21). Increasing the amount of Cs_2CO_3 from 0.8 to 1.2 equiv. led to a slight lower yield of **3aa** (Table 1, Entry 22). When the reaction was carried out under nitrogen atmosphere, a low yield of **3aa** was obtained (Table 1, Entry 23).

Table 1 Optimization of reaction conditions[a]



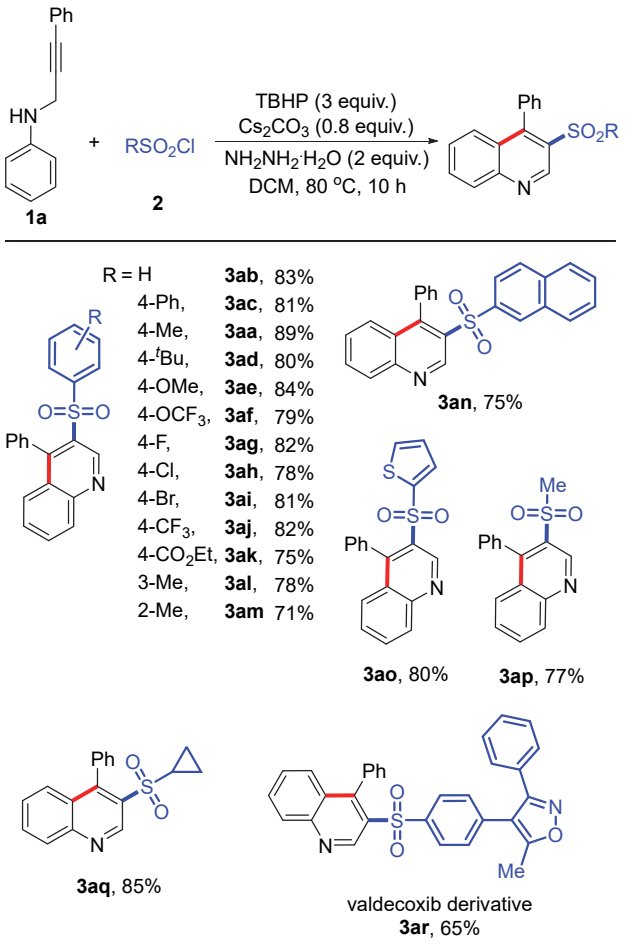
Entry	Oxidant	Base	Solvent	Temp./℃	Yield[b]/%
1	TBHP	K_2CO_3	DCM	80	58
2	DTBP	K_2CO_3	DCM	80	11
3	BPO	K_2CO_3	DCM	80	6
4	H_2O_2	K_2CO_3	DCM	80	Trace
5	$PhI(OAc)_2$	K_2CO_3	DCM	80	8
6	$K_2S_2O_8$	K_2CO_3	DCM	80	14
7	TBHP	Na_2CO_3	DCM	80	44
8	**TBHP**	**Cs_2CO_3**	**DCM**	**80**	**90**
9	TBHP	Et_3N	DCM	80	55
10	TBHP	Pyridine	DCM	80	43
11	TBHP	DABCO	DCM	80	22
12	TBHP	Cs_2CO_3	DCE	80	69
13	TBHP	Cs_2CO_3	MeCN	80	82
14	TBHP	Cs_2CO_3	EtOH	80	9
15	TBHP	Cs_2CO_3	EtOAc	80	45
16	TBHP	Cs_2CO_3	THF	80	35
17	TBHP	Cs_2CO_3	DCM	90	89
18	TBHP	Cs_2CO_3	DCM	70	62
19	—	Cs_2CO_3	DCM	80	—
20	TBHP	—	DCM	80	15
21[c]	TBHP	Cs_2CO_3	DCM	80	—
22[d]	TBHP	Cs_2CO_3	DCM	80	84
22[d]	TBHP	Cs_2CO_3	DCM	80	55

[a] Conditions: **1a** (0.2 mmol), **2a** (0.35 mmol), $NH_2NH_2·H_2O$ (2 equiv.), base (0.8 equiv.), oxidant (3 equiv.), solvent (2 mL), in air, 10 h. [b] Estimated by ^1H NMR using diethyl phthalate as an internal reference. [c] Without $NH_2NH_2·H_2O$. [d] 1.2 equiv. of Cs_2CO_3 was used. [e] Under N_2.

After identification of the optimal conditions (Table 1, Entry 8), an investigation was carried out into the scope and limitation of this reaction (Table 2). To our delight, a variety of benzenesulfonyl chlorides were well tolerated in this one-pot reaction. The experimental results suggested that the desired products **3** were formed in good to excellent yields whether with neutral (**3ab** and **3ac**), electron-donating (**3aa**, **3ad**～**3af**), weak electron-withdrawing (**3ag**～**3ai**), strong electron-withdrawing group (**3aj** and **3ak**) at the *para*-positions of phenyl ring in bzenesulfonyl chlorides. Reactions of *meta*- and *ortho*-methyl benzenesulfonyl chlorides proceeded smoothly and delivered the target products (**3al** and **3am**) in good yields, indicating that the steric effect of substituents on phenyl rings is negligible. Both naphthalene-2-sulfonyl chloride and thio-

phene-2-sulfonyl chloride participated in the current transformations efficiently and gave the desired products (**3an** and **3ao**) in 75% and 80% yields, respectively. Gratifyingly, aliphatic sulfonyl chlorides, namely methanesulfonyl chloride and cyclopropanesulfonyl chloride were efficiently transformed into the corresponding products (**3ap** and **3aq**) with good yields. Notably, a broad range of functional groups was well tolerated, including alkyl, cyclopropyl, methoxy, trifluoromethoxy, fluoro, chloro, bromo, trifluoromethyl, ester, naphthyl and thienyl group. It is worth mentioning that the one-pot reaction provided a step-economical access to biologically active molecules and their derivatives. Valdecoxib, an inhibitor of cyclooxygenase-2, could be easily converted into the corresponding quinoline-containing valdecoxib **3ar**. However, no product was formed when pyridine-4-sulfonyl chloride was employed in the reaction under the standard conditions.

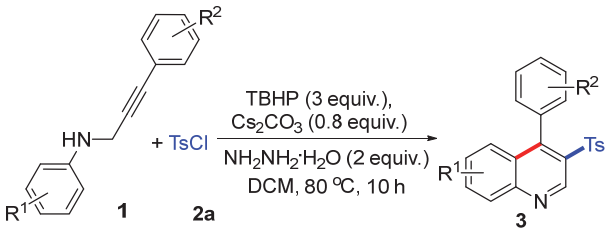
Table 2 Substrate scope of sulfonyl chlorides[a]

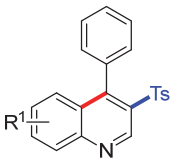


[a] Conditions: **1** (0.2 mmol), **2a** (0.35 mmol), $NH_2NH_2{\cdot}H_2O$ (2 equiv.), Cs_2CO_3 (0.8 equiv.), TBHP (3 equiv.), DCM (2 mL), in air, 10 h.

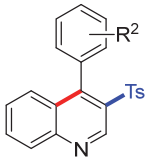
Subsequently, the scope of *N*-propargylanilines was investigated (Table 3). Both the aniline moieties and alkyne moieties substituted with an electron-donating or electron-withdrawing group on the *para*-position of benzene rings were well compatible under the standard reaction conditions, delivering the target products (**3ba**～**3fa**, and **3ia**～**3la**) in good to excellent yields. In addition, steric hindrance on the benzene ring of aniline moieties showed no influence on this transformation (**3ga** and **3ha**). It is noteworthy that halides and methoxy on the benzene rings of product **3** represent versatile handles for further transformations.

Table 3 Substrate scope of *N*-propargylanilines[a]





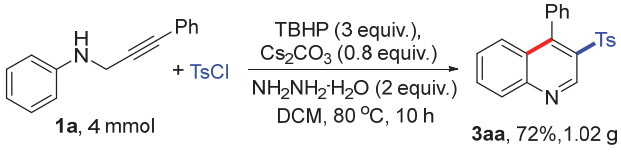
R[1] = 4-Me, **3ab**, 87%
4-F, **3ac**, 86%
4-Cl, **3ad**, 85%
4-Br, **3ae**, 81%
4-Ac, **3af**, 77%
3-Cl, **3ag**, 85%
2-Me, **3ah**, 88%



R[2] = 4-Et, **3ai**, 77%
4-OMe, **3aj**, 72%
4-Br, **3ak**, 86%
4-Ac, **3al**, 84%

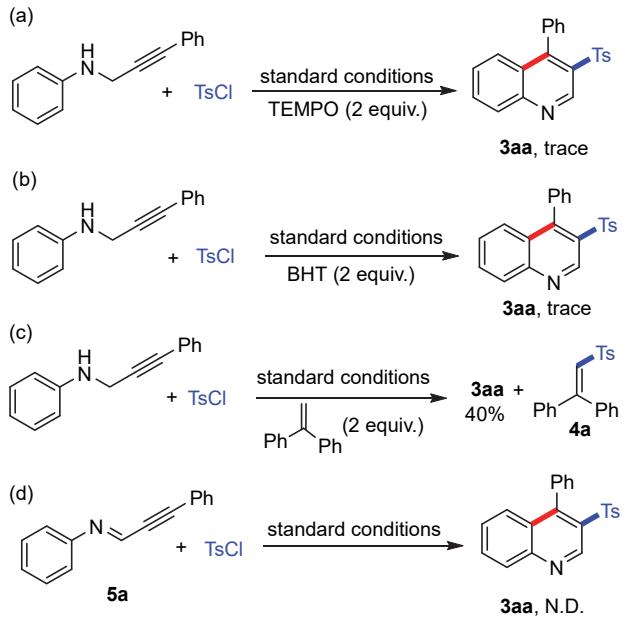
[a] Conditions: **1** (0.2 mmol), **2a** (0.35 mmol), $NH_2NH_2{\cdot}H_2O$ (2 equiv.), Cs_2CO_3 (0.8 equiv.), TBHP (3 equiv.), DCM (2 mL), in air, 10 h.

Furthermore, the present method for synthesizing 4-aryl-3-sulfonyl quinoline was also easily scalable. With the amplification of *N*-(3-phenylprop-2-yn-1-yl)aniline **1a** from 0.2 mmol to 4 mmol, this reaction provided 4-phenyl-3-tosylquinoline **3aa** in 72% yield without further optimization (Scheme 2).



Scheme 2 Gram-scale synthesis of **3aa**

To gain insight into the reaction mechanism, a series of control experiments were performed. With two equiv. of radical scavenger (TEMPO or BHT) as the additives, only a trace amount of product **3aa** was observed (Scheme 3a, 3b). The adduct **4a** of 1,1-diphenylethylene with sulfonyl radical was also detected (Scheme 3c). These experimental results suggested that the present reaction proceeded through a free-radical pathway. The treatment of *N*-(3-phenylprop-2-yn-1-ylidene)-aniline (**5a**) with TsCl under the standard could not give **3aa** (Scheme 3d). This result suggested that compound **5a** was not a crucial intermediate in the present reaction.



Scheme 3 Control experiments of mechanism research

Based on the above-mentioned results and previously reported literature,[6a,7a] a possible reaction mechanism for directly constructing 3-sulfonylquinolines from *N*-propargylanilines with sulfonyl chlorides was proposed in Scheme 4. First, the reaction of sulfonyl chloride **2** with hydrazinium hydrate *in situ* generated a sulfonyl hydrazide **A**, which was oxidized to form a sulfonyl radical **B**. The radical **B** was then trapped by the alkynyl group of *N*-propargylaniline **1** providing a radical intermediate **C**, followed by an intramolecular cyclization process to form a radical **D**. The intermediate **D** was further oxidized to generate a cationic intermediate **E**, which underwent deprotonation followed by an oxidative aromatization to yield the terminal product **3**.

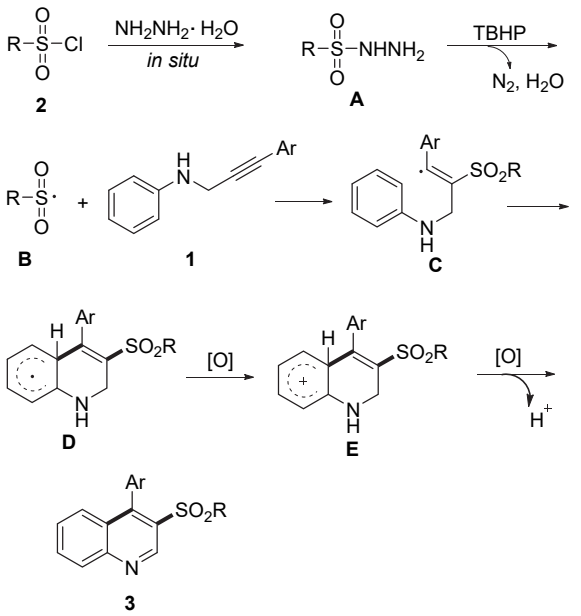
3 Conclusions

In summary, we have developed an efficient and facile protocol for the direct synthesis of 3-sulfonylquinolines from *N*-propargylanilines and sulfonyl chlorides through a one-pot sequential hydrazination/sulfonylation/cyclization reaction. A variety of functionalized 3-sulfonylquinolines could be obtained in good to excellent yields and high regioselectivities with broad functional-group compatibility. In addition, this procedure not only can be performed on a gram scale but also be applicable to prepare marketed pharmaceutical derivative (quinoline-containing valdecoxib). These features make the present method highly attractive in the field of pharmaceutical industry and synthetic chemistry.

4 Experimental section

4.1 General information

All reactions were carried out in anhydrous solvent and



Scheme 4 Proposed reaction mechanism of the sulfonylation reaction

commercially available reagents were used as received unless otherwise stated. Analytical thin layer chromatography (TLC) was performed on precoated aluminium-backed silica gel 60 F_{254} plates (EMD Millipore, 200 μm thickness). TLC plates were visualized with ultraviolet light and treatment with $KMnO_4$ or vanillin stains followed by heating. Flash column chromatography was performed using Tsingtao silica gel (200～300). ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker Avance DRX-500 spectrometers. Chemical shifts (δ) were calibrated using the signal of residual undeuterated solvent as internal reference ($CDCl_3$: δ_H=7.26 and δ_C=77.16).

4.2 General experimental procedure for compounds 3

N-Propargylanilines **1** (0.2 mmol), sulfonyl chloride **2** (0.35 mmol), TBHP (0.6 mmol), Cs_2CO_3 (0.16 mmol), hydrazinium hydrate (0.4 mmol) and DCM (2 mL) were sequentially added to a Schlenk tube at room temperature. The reaction mixture was heated at 80 ℃ under air atmosphere. When the reaction was completed, the reaction mixture was filtrated. The resulting mixture was purified by flash column chromatography using a mixture of petroleum ether and ethyl acetate (V∶V=5∶1) as eluent to give the desired products.

4.3 Larger-scale synthesis of 3aa

1a (4 mmol), **2a** (7 mmol), TBHP (12 mmol), Cs_2CO_3 (3.2 mmol), hydrazinium hydrate (8 mmol) and DCM (20 mL) were sequentially added to a 50 mL Schlenk tube at room temperature. The reaction mixture was heated with stirring under air at 80 ℃ for 8 h. When the reaction was completed, the reaction mixture was filtrated. The resulting mixture was purified by flash column chromatography using a mixture of petroleum ether and ethyl acetate

(V∶V=5∶1) as eluent to give the desired products (1.02 g, 72%).

4.4 Characterization data of products **3**

4-Phenyl-3-tosylquinoline (**3aa**): ^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.72 (s, 1H), 8.15 (dd, J=8.6, 1.2 Hz, 1H), 7.77～7.73 (m, 1H), 7.42～7.35 (m, 2H), 7.28～7.24 (m, 3H), 7.13 (d, J=8.4 Hz, 2H), 6.98 (d, J=8.1 Hz, 2H), 6.92～6.85 (m, 2H), 2.28 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 149.94, 149.63, 147.75, 144.06, 137.97, 132.69, 132.64, 132.21, 130.04, 129.60, 129.27, 128.69, 127.96, 127.86, 127.67, 127.55, 127.46, 21.58. The characterization data are consistent with previous report.[7a]

Phenyl-3-(phenylsulfonyl)quinoline (**3ab**): ^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.82 (s, 1H), 8.22 (dd, J=8.7, 1.3 Hz, 1H), 7.85～7.81 (m, 1H), 7.49～7.44 (m, 3H), 7.35～7.31 (m, 5H), 7.27～7.24 (m, 2H), 6.97～6.91 (m, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 150.07, 149.80, 147.68, 140.88, 133.01, 132.51, 132.39, 132.29, 130.04, 129.68, 128.70, 128.65, 127.91, 127.86, 127.74, 127.50, 127.44. The characterization data are consistent with previous report.[7a]

3-([1,1'-Biphenyl]-4-ylsulfonyl)-4-phenylquinoline (**3ac**): ^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.76 (s, 1H), 8.15 (dd, J=8.5, 1.2 Hz, 1H), 7.77～7.73 (m, 1H), 7.46～7.43 (m, 2H), 7.41～7.35 (m, 6H), 7.34～7.23 (m, 6H), 6.93～6.87 (m, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 149.92, 149.81, 147.67, 145.93, 139.33, 139.16, 132.64, 132.57, 132.29, 130.12, 129.71, 129.10, 128.74, 128.67, 128.42, 127.92, 127.75, 127.51, 127.46, 127.31, 127.25. The characterization data are consistent with previous report.[7a]

3-((4-(*tert*-Butyl)phenyl)sulfonyl)-4-phenylquinoline (**3ad**): ^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.80 (s, 1H), 8.25～8.20 (m, 1H), 7.85～7.80 (m, 1H), 7.47～7.43 (m, 2H), 7.35～7.28 (m, 3H), 7.25 (s, 4H), 6.97～6.93 (m, 2H), 1.29 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 156.98, 149.76, 149.71, 147.69, 137.71, 132.74, 132.65, 132.15, 130.03, 129.67, 128.58, 127.83, 127.72, 127.66, 127.51, 127.42, 125.66, 35.13, 31.03. The characterization data are consistent with previous report.[7a]

3-((4-Mthoxyphenyl)sulfonyl)-4-phenylquinoline (**3ae**): ^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.79 (s, 1H), 8.22 (dd, J=8.5, 1.2 Hz, 1H), 7.84～7.81 (m, 1H), 7.50～7.44 (m, 2H), 7.39～7.32 (m, 3H), 7.25 (t, J=7.2 Hz, 2H), 7.00～6.97 (m, 2H), 6.74～6.71 (m, 2H), 3.81 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 163.27, 149.71, 149.61, 147.75, 132.94, 132.74, 132.43, 132.13, 130.18, 130.09, 129.62, 128.70, 127.83, 127.72, 127.56, 127.43, 113.89, 55.67. The characterization data are consistent with previous report.[7a]

4-Penyl-3-((4-(trifluoromethoxy)phenyl)sulfonyl)quinoline (**3af**): ^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.82 (s, 1H), 8.24 (dd, J=8.5, 1.2 Hz, 1H), 7.87～7.83 (m, 1H), 7.51～7.45 (m, 2H), 7.36～7.32 (m, 5H), 7.07 (dd, J=8.9, 1.1 Hz, 2H), 6.97～6.94 (m, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 152.30, 149.97, 147.34, 139.03, 132.51, 132.41, 132.03, 130.11, 130.06, 129.78, 128.90, 128.07, 127.84, 127.40, 127.35, 120.56, 120.15 (d, J=257.5 Hz). The characterization data are consistent with previous report.[8a]

3-((4-Fluorophenyl)sulfonyl)-4-phenylquinoline (**3ag**): ^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.72 (s, 1H), 8.17～8.12 (m, 1H), 7.78～7.74 (m, 1H), 7.43～7.38 (m, 2H), 7.31～7.22 (m, 5H), 6.91～6.87 (m, 2H), 6.87～6.82 (m, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 165.28 (d, J=255 Hz), 149.92, 149.89, 147.49, 136.84 (d, J=2.5 Hz), 132.50, 132.41, 132.24, 130.74 (d, J=10.0 Hz), 130.11, 129.74, 128.87, 128.01, 127.83, 127.41, 115.92 (d, J=23.8 Hz). The characterization data are consistent with previous report.[7a]

3-((4-Chlorophenyl)sulfonyl)-4-phenylquinoline (**3ah**): ^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.79 (s, 1H), 8.25～8.20 (m, 1H), 7.88～7.82 (m, 1H), 7.51～7.46 (m, 2H), 7.39～7.33 (m, 3H), 7.25～7.20 (m, 4H), 7.00～6.95 (m, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 150.01, 149.91, 147.47, 139.77, 139.29, 132.47, 132.45, 132.07, 130.11, 129.75, 129.41, 129.33, 128.90, 128.04, 127.83, 127.42, 127.39. The characterization data are consistent with previous report.[7a]

3-((4-Bomophenyl)sulfonyl)-4-phenylquinoline (**3ai**): ^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.79 (s, 1H), 8.23 (d, J=8.5 Hz, 1H), 7.87～7.84 (m, 1H), 7.51～7.46 (m, 2H), 7.41～7.32 (m, 5H), 7.18～7.13 (m, 2H), 6.97 (dd, J=7.2, 1.2 Hz, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 150.03, 149.91, 147.46, 139.82, 132.48, 132.44, 132.03, 131.90, 130.10, 129.74, 129.38, 128.91, 128.35, 128.05, 127.83, 127.43, 127.40. The characterization data are consistent with previous report.[7a]

4-Penyl-3-((4-(trifluoromethyl)phenyl)sulfonyl)quinoline (**3aj**): ^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.74 (s, 1H), 8.16 (dd, J=8.6, 1.2 Hz, 1H), 7.80～7.77 (m, 1H), 7.44～7.34 (m, 6H), 7.27～7.22 (m, 3H), 6.88～6.83 (m, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 150.19, 150.03, 147.31, 144.24, 134.57 (q, J=33 Hz), 132.66, 132.30, 131.68, 130.10, 129.78, 128.96, 128.38, 128.16, 127.86, 127.42, 127.31, 125.68 (q, J=3.33 Hz), 123.07 (q, J=272.5 Hz). The characterization data are consistent with previous report.[7a]

Ethyl 4-((4-phenylquinolin-3-yl)sulfonyl)benzoate (**3ak**): ^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.74 (s, 1H), 8.16 (d, J=8.5 Hz, 1H), 7.83 (d, J=8.6 Hz, 2H), 7.79～7.75 (m, 1H), 7.42～7.39 (m, 2H), 7.32～7.29 (m, 2H), 7.28～7.23 (m, 3H), 6.86 (dd, J=8.2, 1.3 Hz, 2H), 4.32 (q, J=7.1 Hz, 2H), 1.32 (t, J=7.1 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 165.01, 150.28, 149.96, 147.54, 144.58, 134.37, 132.54, 132.32, 131.81, 130.08, 129.76, 129.67, 128.94, 128.06, 127.87, 127.82, 127.44, 127.39, 61.79, 14.23. The characterization data are consistent with previous report.[8b]

4-Penyl-3-(*m*-tolylsulfonyl)quinoline (**3al**): ^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.82 (s, 1H), 8.26～8.21 (m, 1H), 7.85-7.80 (m, 1H), 7.49～7.44 (m, 2H), 7.36～7.32 (m, 3H), 7.27～7.24 (m, 1H), 7.18～7.14 (m, 2H), 7.06 (s, 1H), 6.97～6.93 (m, 2H), 2.23 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 149.80, 149.45, 147.37, 140.27, 138.52, 133.68, 132.36, 132.21, 132.05, 129.86, 129.37, 128.38, 128.35, 128.19, 127.66, 127.37, 127.30, 127.21, 124.79,

20.87. The characterization data are consistent with previous report.[7a]

4-Penyl-3-(*o*-tolylsulfonyl)quinoline (**3am**): ^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.84 (s, 1H), 8.24 (dd, J=8.6, 1.2 Hz, 1H), 7.86～7.82 (m, 1H), 7.49～7.45 (m, 1H), 7.36～7.29 (m, 3H), 7.20 (t, J=7.8 Hz, 2H), 7.15 (dd, J=7.9, 1.4 Hz, 1H), 7.08 (d, J=7.5 Hz, 1H), 6.94～6.89 (m, 1H), 6.88～6.85 (m, 2H), 2.26 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 149.69, 149.66, 148.03, 138.60, 136.72, 133.17, 132.23, 132.19, 132.16, 131.88, 129.76, 129.69, 129.65, 128.56, 127.94, 127.64, 127.41, 127.36, 126.03, 20.00. The characterization data are consistent with previous report.[7a]

3-(Nphthalen-2-ylsulfonyl)-4-phenylquinoline (**3an**): ^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.89 (s, 1H), 8.26～8.21 (m, 1H), 7.84～7.80 (m, 2H), 7.76～7.70 (m, 2H), 7.70～7.66 (m, 1H), 7.64～7.60 (m, 1H), 7.58～7.55 (m, 1H), 7.46～7.41 (m, 2H), 7.41～7.36 (m, 1H), 7.31 (dd, J=8.6, 1.4 Hz, 1H), 7.21～7.16 (m, 2H), 6.88 (dd, J=8.1, 1.3 Hz, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 150.17, 149.82, 147.80, 137.34, 134.86, 132.38, 132.29, 132.23, 131.72, 130.22, 130.01, 129.68, 129.54, 129.19, 129.00, 128.84, 127.87, 127.80, 127.60, 127.50, 127.42, 127.40, 122.44. The characterization data are consistent with previous report.[7a]

4-Penyl-3-(thiophen-2-ylsulfonyl)quinoline (**3ao**): ^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.66 (s, 1H), 8.15 (d, J=8.4 Hz, 1H), 7.79～7.75 (m, 1H), 7.47 (dd, J=4.9, 1.4 Hz, 1H), 7.43～7.39 (m, 2H), 7.34 (dd, J=8.3, 7.0 Hz, 2H), 7.30 (dd, J=8.5, 1.4 Hz, 1H), 7.05～7.00 (m, 2H), 6.91 (dd, J=3.9, 1.4 Hz, 1H), 6.78 (dd, J=4.9, 3.8 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 149.93, 149.77, 147.51, 142.06, 134.54, 134.24, 132.92, 132.67, 132.35, 129.90, 129.71, 129.38, 128.83, 127.97, 127.81, 127.59, 127.49, 127.24. The characterization data are consistent with previous report.[7a]

3-(Mthylsulfonyl)-4-phenylquinoline (**3ap**): ^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.51 (s, 1H), 8.16 (d, J=8.5 Hz, 1H), 7.81～7.77 (m, 1H), 7.54～7.46 (m, 4H), 7.42～7.34 (m, 3H), 2.70 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 149.89, 149.63, 147.48, 132.98, 132.44, 131.69, 130.07, 129.78, 129.51, 128.29, 128.15, 127.69, 127.24, 44.55. The characterization data are consistent with previous report.[8b]

3-((4-Mthoxyphenyl)sulfonyl)-4-phenylquinoline (**3aq**): ^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.50 (s, 1H), 8.26～8.21 (m, 1H), 7.88～7.84 (m, 1H), 7.59～7.52 (m, 4H), 7.50～7.43 (m, 3H), 2.05 (tt, J=8.0, 4.8 Hz, 1H), 1.18～1.13 (m, 2H), 0.89～0.83 (m, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 149.65, 149.42, 147.69, 133.50, 132.18, 132.05, 130.20, 129.72, 129.31, 128.04, 127.98, 127.71, 127.50, 32.94, 6.32. The characterization data are consistent with previous report.[8b]

5-Mthyl-3-phenyl-4-(4-((4-phenylquinolin-3-yl)-sulfonyl)-phenyl)isoxazole (**3ar**): ^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.74 (s, 1H), 8.16 (d, J=8.5 Hz, 1H), 7.82～7.74 (m, 1H), 7.42 (t, J=7.7 Hz, 1H), 7.34～7.25 (m, 6H), 7.24～7.18 (m, 5H), 6.99 (d, J=8.0 Hz, 2H), 6.92 (d, J=6.7 Hz, 2H), 2.37 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 166.26, 159.96, 148.99, 148.82, 146.55, 138.75, 134.63, 131.45, 131.42, 131.00, 129.08, 128.76, 128.74, 128.69, 127.85, 127.65, 127.38, 127.31, 127.25, 127.03, 126.70, 126.39, 120.92, 113.36, 10.68. HRMS (ESI) calcd for $C_{31}H_{22}N_2O_3S$ $[M+H]^+$ 503.1424, found 503.1427.

6-Mthyl-4-phenyl-3-tosylquinoline (**3ba**): ^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.63 (s, 1H), 8.10 (d, J=9.2 Hz, 1H), 7.49～7.43 (m, 2H), 7.34 (t, J=7.7 Hz, 2H), 7.21 (d, J=8.4 Hz, 2H), 7.05 (d, J=7.7 Hz, 2H), 6.96 (d, J=6.9 Hz, 2H), 6.51 (d, J=2.8 Hz, 1H), 3.61 (s, 3H), 2.35 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 158.61, 148.05, 146.09, 145.35, 143.96, 138.08, 132.95, 132.89, 131.03, 129.93, 129.23, 128.85, 128.63, 127.93, 127.78, 124.83, 104.80, 55.35, 21.56. The characterization data are consistent with previous report.[7a]

6-Fuoro-4-phenyl-3-tosylquinoline (**3ca**): ^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.66 (s, 1H), 8.14 (dd, J=9.2, 5.4 Hz, 1H), 7.53～7.48 (m, 1H), 7.42～7.37 (m, 1H), 7.27 (t, J=7.8 Hz, 2H), 7.12 (d, J=8.3 Hz, 2H), 6.98 (d, J=8.0 Hz, 2H), 6.90～6.81 (m, 3H), 2.27 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 161.10 (d, J=248.7 Hz), 149.22 (d, J=6.25 Hz), 147.11 (d, J=2.50 Hz), 146.83, 144.26, 137.69, 133.47, 132.24, 132.22 (d, J=10.00 Hz), 129.88, 129.32, 128.95, 128.72 (d, J=9.80 Hz), 127.99, 127.89, 122.51 (d, J=25.02 Hz), 110.74 (d, J=23.75 Hz), 21.57. The characterization data are consistent with previous report.[7a]

6-Cloro-4-phenyl-3-tosylquinoline (**3da**): ^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.68 (s, 1H), 8.07 (d, J=9.0 Hz, 1H), 7.68～7.64 (m, 1H), 7.40 (t, J=7.5 Hz, 1H), 7.27 (t, J=7.8 Hz, 2H), 7.20 (d, J=2.3 Hz, 1H), 7.11 (d, J=8.4 Hz, 2H), 6.97 (d, J=8.1 Hz, 2H), 6.86 (dd, J=8.2, 1.3 Hz, 2H), 2.27 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 149.04, 148.05, 147.94, 144.30, 137.63, 134.08, 133.09, 131.94, 131.22, 129.98, 129.96, 129.34, 129.02, 127.98, 127.90, 126.03, 21.58. The characterization data are consistent with previous report.[7a]

6-Bomo-4-phenyl-3-tosylquinoline (**3ea**): ^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.69 (s, 1H), 7.98 (d, J=8.9 Hz, 1H), 7.78 (dd, J=9.0, 2.2 Hz, 1H), 7.42～7.35 (m, 2H), 7.30～7.24 (m, 2H), 7.10 (d, J=8.4 Hz, 2H), 7.00～6.95 (m, 2H), 6.89～6.82 (m, 2H), 2.26 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 148.90, 148.29, 148.08, 144.29, 137.62, 135.62, 133.58, 131.89, 131.31, 129.97, 129.34, 129.03, 128.79, 127.96, 127.90, 122.31, 21.59. The characterization data are consistent with previous report.[7a]

1-(4-Penyl-3-tosylquinolin-6-yl)ethan-1-one (**3fa**): ^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.80 (s, 1H), 8.27 (dd, J=8.8, 1.9 Hz, 1H), 8.20 (d, J=8.9 Hz, 1H), 7.85 (d, J=1.9 Hz, 1H), 7.47～7.42 (m, 1H), 7.31 (t, J=7.8 Hz, 2H), 7.14 (d, J=8.4 Hz, 2H), 7.02～6.98 (m, 2H), 6.95～6.86 (m, 2H), 2.37 (s, 3H), 2.29 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 196.82, 151.23, 149.94, 144.35, 142.42, 137.60, 136.83, 135.89, 131.91, 130.36, 130.18, 130.01, 129.95, 129.36, 129.17, 129.12, 128.01, 127.86, 26.46, 21.58. The characterization data are consistent with previous report.[8b]

7-Cloro-4-phenyl-3-tosylquinoline (**3ga**): ^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.78 (s, 1H), 8.13～8.09 (m, 1H), 7.62

(dd, J=8.5, 7.5 Hz, 1H), 7.48 (dd, J=7.5, 1.3 Hz, 1H), 7.31～7.28 (m, 1H), 7.13～7.07 (m, 2H), 7.04 (d, J=8.4 Hz, 2H), 6.97～6.92 (m, 2H), 6.87～6.79 (m, 2H), 2.27 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 151.32, 149.87, 148.15, 144.04, 137.84, 134.82, 133.43, 132.69, 131.84, 131.61, 130.71, 129.94, 129.29, 128.61, 127.69, 127.00, 124.34, 21.57. The characterization data are consistent with previous report.[7a]

8-Mthyl-4-phenyl-3-tosylquinoline (**3ha**): ^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.72 (s, 1H), 7.58 (d, J=6.9 Hz, 1H), 7.40～7.35 (m, 1H), 7.28～7.23 (m, 3H), 7.14 (d, J=8.4 Hz, 2H), 7.11～7.07 (m, 1H), 7.00～6.94 (m, 2H), 6.89～6.85 (m, 2H), 2.80 (s, 3H), 2.27 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 150.03, 148.57, 146.51, 143.98, 138.10, 137.52, 133.07, 132.43, 132.35, 130.07, 129.24, 128.55, 127.97, 127.60, 127.58, 127.55, 125.44, 21.56, 18.25. The characterization data are consistent with previous report.[7a]

4-(4-Ehylphenyl)-3-tosylquinoline (**3ia**): ^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.64 (s, 1H), 8.06 (d, J=8.6 Hz, 1H), 7.61 (dd, J=8.7, 1.9 Hz, 1H), 7.41～7.35 (m, 1H), 7.29～7.23 (m, 2H), 7.12 (d, J=8.3 Hz, 2H), 7.00～6.94 (m, 3H), 6.87 (dd, J=8.1, 1.4 Hz, 2H), 2.56 (q, J=7.6 Hz, 2H), 2.26 (s, 3H), 1.05 (t, J=7.6 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 149.31, 148.53, 146.87, 144.31, 143.95, 138.11, 133.43, 132.78, 132.62, 130.06, 129.44, 129.22, 128.59, 127.91, 127.64, 127.58, 124.84, 28.98, 21.55, 15.28. The characterization data are consistent with previous report.[8b]

4-(4-Mthoxyphenyl)-3-tosylquinoline (**3ja**): ^{1}H NMR (500 MHz, CDCl3) δ: 9.69 (s, 1H), 8.13 (d, J=8.2 Hz, 1H), 7.75～7.71 (m, 1H), 7.40-7.36 (m, 1H), 7.34～7.30 (m, 1H), 7.15 (d, J=8.3 Hz, 2H), 7.01～6.96 (m, 2H), 6.83～6.76 (m, 4H), 3.83 (s, 3H), 2.27 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 160.02, 150.19, 149.53, 147.69, 144.02, 138.01, 133.12, 132.19, 131.43, 129.50, 129.19, 127.99, 127.91, 127.82, 127.48, 124.59, 113.16, 55.43, 21.56. The characterization data are consistent with previous report.[7a]

4-(4-Bomophenyl)-3-tosylquinoline (**3ka**): ^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.70 (s, 1H), 8.15 (d, J=8.5 Hz, 1H), 7.78～7.74 (m, 1H), 7.44～7.38 (m, 3H), 7.27～7.23 (m, 1H), 7.19～7.15 (m, 2H), 7.03 (d, J=8.1 Hz, 2H), 6.77 (d, J=8.4 Hz, 2H), 2.30 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 149.64, 148.44, 147.61, 144.44, 137.87, 132.86, 132.38, 131.65, 131.59, 130.93, 129.77, 129.39, 128.15, 127.90, 127.09, 123.29, 21.62. The characterization data are consistent with previous report.[7a]

1-(4-(3-Tsylquinolin-4-yl)phenyl)ethan-1-one (**3la**): ^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.65 (s, 1H), 8.13 (d, J=8.5 Hz, 1H), 7.87 (d, J=8.2 Hz, 2H), 7.77～7.72 (m, 1H), 7.41～7.36 (m, 1H), 7.20～7.15 (m, 3H), 7.05～6.97 (m, 4H), 2.62 (s, 3H), 2.27 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 197.49, 149.59, 148.43, 147.71, 144.56, 137.91, 137.73, 137.09, 132.48, 132.41, 130.34, 129.80, 129.45, 128.21, 127.88, 127.52, 127.03, 126.87, 26.78, 21.58. The characterization data are consistent with previous report.[7a]

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