$ZnCl_2$-Catalyzed Intramolecular Cyclization Reaction of 2-Aminochalcones Using Polymer-Supported Selenium Reagent: Synthesis of 2-Phenyl-4-quinolones and 2-Phenyl-2,3-dihydroquinolin-4(1*H*)-one

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Dedicated to the memory of Prof. Xian Huang

Abstract: A new and efficient method for the synthesis of 2-phenyl-4-quinolones and 2-phenyl-2,3-dihydroquinolin-4(1*H*)-ones is described. The reaction involves $ZnCl_2$-mediated polystyrene-supported selenium-induced intramolecular cyclization of 2-aminochalcones and subsequent traceless or functionalizing cleavage of selenium linker.

Key words: polystyrene-supported selenium reagent, intramolecular cyclization, zinc chloride, 2-phenyl-4-quinolones, 2-phenyl-2, 3-dihydroquinolin-4(1*H*)-one

2-Aryl-4-quinolones and 2-phenyl-2,3-dihydroquinolin-4(1*H*)-ones have been studied as potential treatments for a range of diseases[1] because of their important biological properties, such as antiviral,[1c,d] antiplatelet,[1b] antitumor,[2] and positive cardiac effects.[1a] Various methods have been reported for the synthesis of 2-aryl-4-quinolones and 2-phenyl-2,3-dihydroquinolin-4(1*H*)-ones.[3] Conrad–Limpach and Niementowski reactions[4] generally focus on the condensation of amines and carboxyl derivatives followed by cyclization to produce the desired quinolones. Often the substrate scope of these reactions is limited by the necessity to employ harsh cyclization conditions, including temperature above 200 °C or strong acids such as polyphosphoric acid or Eaton's reagent. Examples on transition-metal-catalyzed syntheses of these compounds,[5] including palladium-catalyzed carbonylation reaction,[6] titanium-mediated reductive coupling reaction,[7] and ruthenium-catalyzed reduction reaction,[8] have also been reported. Huang has reported a solid-phase synthesis of 4(1*H*)-quinolones by the thermal cyclization reaction of polymer-bound substituted aminomethylene cyclic malonic acid esters at 220–240 °C.[9] High reaction temperature limits the application of this method. Therefore, it is still a challenge to explore a mild and efficient solid-phase synthesis of 4-quinolones.

Solid-phase synthesis is becoming an increasingly important tool for the synthetic chemists due to the simple work-up procedure and adaptable to parallel synthesis. Organoselenium resins are ideal linkers and reagents for solid-phase synthesis because organoselenium compounds can be used as synthetic intermediates[10] and Se–C bond can be easily broken by various methods.[11] Furthermore, polymer-supported organic selenides can overcome the shortcomings such as highly malodorous and toxicity of organic selenides. In the past decade, a variety of heterocyclic compounds libraries have been constructed from organoselenium resins by several research groups[12] and ours.[13] In recent years, we have become interested in Lewis acid catalyzed polystyrene-supported selenium-mediated intramolecular cyclization reaction of electron-deficient olefins with heteroatom.[13a] While polystyrene-supported selenium bromide has been successfully applied in the synthesis of heterocyclic compounds by the intramolecular electrophilic cyclization of electron-rich olefins with heteroatom,[12c,d] olefins conjugated with the carbonyl group, and of course the electron-deficient olefins, fail to react with selenium resins.[13a] In order to overcome the preparative obstacle, we envisioned that Lewis acid could dampen the impact of carbonyl group on olefin and realize the addition of organoseleium reagent to olefins. We have reported that $ZnCl_2$ has a high catalytic activity in the polystyrene-supported selenium-induced cyclization of 2-hydroxychalcones.[13a] In continuation of our efforts toward the solid-phase synthesis of heterocyclic compounds, herein we describe an efficient approach for the solid-phase synthesis of 2-aryl-4-quinolones and 2-phenyl-2,3-dihydroquinolin-4(1*H*)-ones. The key transformation of this synthetic process involves $ZnCl_2$-catalyzed intramolecular cyclization reaction of 2-aminochalcones induced by polymer-supported organoselenium reagents and subsequent oxidation and elimination reaction of selenides, and free-radical hydrogenation or allylation reaction of selenides.

First, 2-aminochalcones were synthesized in yield of 80% by reaction of 2-aminoacetophenone with benzaldehyde in the presence of sodium hydroxide at 0 °C.[14] Then polystyrene-supported selenenyl bromide[15] (dark-red resin, Br: 0.99 mmol/g), was treated in turn with $ZnCl_2$ and 2-aminochalcones in dry CH_2Cl_2 to give the corresponding 2,3-dihydro-3-polystyrene-supported selenenyl-4-quinolone (**2**) which underwent further NH-alkylation with alkyl halides under alkaline conditions to give **3**, subsequent selenide oxidation and elimination reactions with H_2O_2 to afford 1-alkyl-4-quinolone **4** in moderate yields (40–48%). The yields of **4** could be increased to 76–89% when 2-alkylamino chalcones **5** were used as reactants

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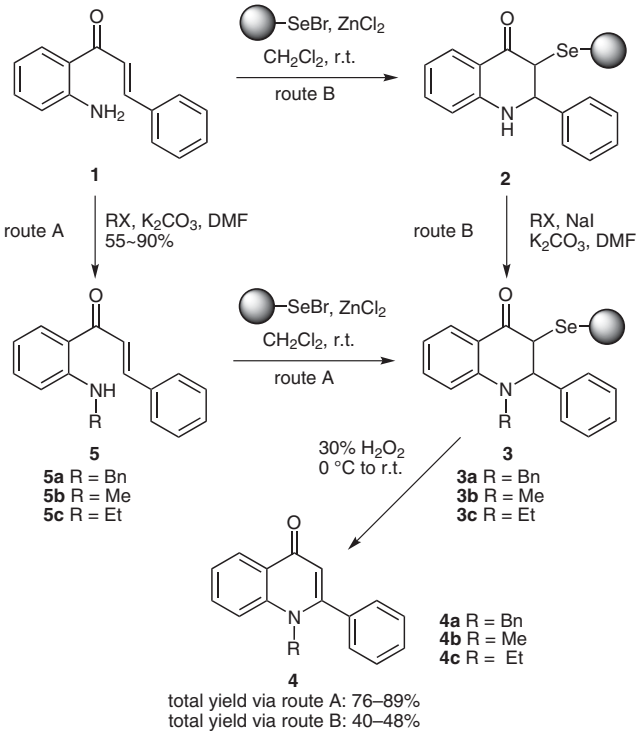
(Scheme 1). This was probably due to the higher nucleophilicity of the alkylamino moiety.

It was observed that the decolorization of polystyrene-supported selenenyl bromide occurred when 5.0 equivalents of compound **1** were used. After being stirred at room temperature for 12 hours, the solid-phase ring-closure reaction was completed, and elemental analysis of resin **2** showed that no Br was present. The reaction was also monitored by FT-IR, which showed a strong peak of the carbonyl absorption at 1638 cm^{-1} in the resulting resin **2**.



Scheme 1

In an effort to optimize the reaction conditions, various cyclization reaction conditions involving 2-benzylamino chalcone (**5a**) and polystyrene-supported selenenyl bromide were explored (Table 1). In the first attempt, 40 mol% of $SnCl_4$, $FeCl_3$, $AlCl_3$, and $ZrCl_4$ were used as catalyst, respectively, product **4a** was obtained in low yields (Table 1, entries 4–7), 82% being the highest yield obtained when using 40 mol% of $ZnCl_2$ as the catalyst at room temperature for 12 hours (Table 1, entry 9).

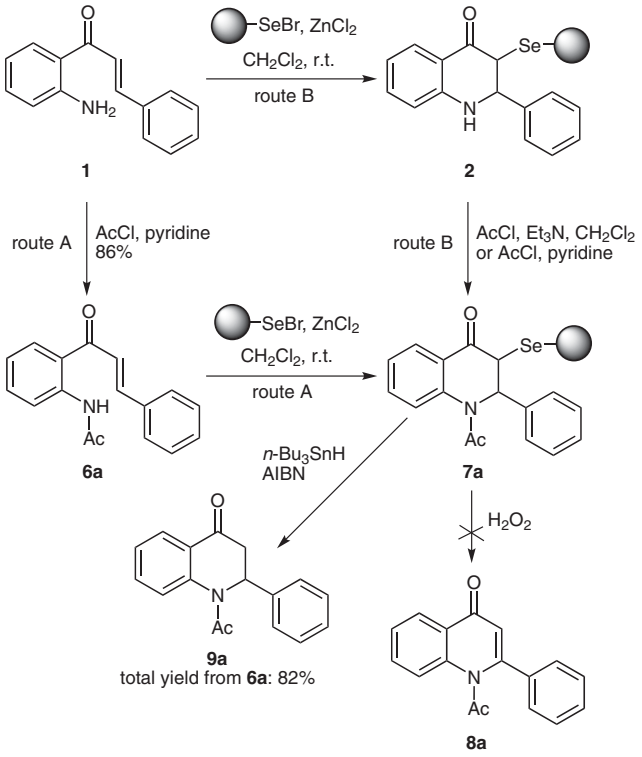
Increasing or decreasing the amount of $ZnCl_2$ or prolonging the reaction time had little effect on the yield of **4a**. $TiCl_4$, $BF_3{\cdot}OEt_2$, and $SnCl_2{\cdot}4H_2O$ has no catalytic activity in the cyclization reaction (Table 1, entries 1–3). As a result, the optimized conditions of the cyclization reaction were 5.0 equivalents of **5**, 40 mol% of $ZnCl_2$, and 1.0 g of polystyrene-supported selenenyl bromide in 15 mL of dry CH_2Cl_2 at room temperature for 12 hours. To test the scope and generality of this protocol, the polystyrene-supported selenium-induced cyclization reactions of a series of substituted 2-amino-chalcones **5** were studied under the

Table 1 Optimization of Solid-Phase Conditions of Cyclization

Entry	Lewis acid (mol%)	Time (h)	Yield of **4a** (%)[a]	Purity of **4a** (%)[b]
1	$TiCl_4$ (40)	12	n.r.	–
2	$BF_3{\cdot}OEt_2$ (40)	12	n.r.	–
3	$SnCl_2{\cdot}4H_2O$ (40)	12	n.r.	–
4	$SnCl_4$ (40)	12	45	>85
5	$AlCl_3$ (40)	12	20	>80
6	$FeCl_3$ (40)	12	28	>80
7	$ZrCl_4$ (40)	12	10	>80
8	$ZnCl_2$ (20)	12	64	>90
9	$ZnCl_2$ (40)	12	82	>95
10	$ZnCl_2$ (60)	12	80	>95
11	$ZnCl_2$ (40)	24	80	>90

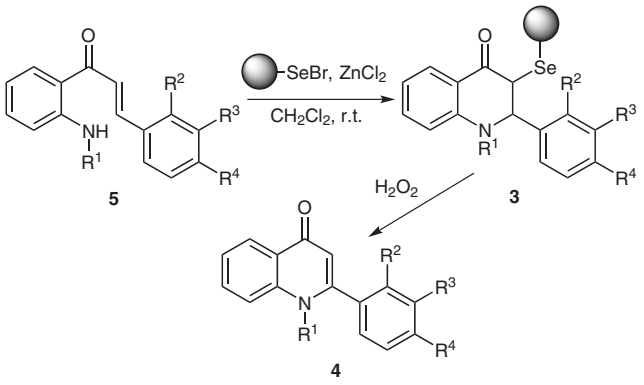
[a] Yields of the crude products based on the loading of selenium bromide resin (Br, 0.99 mmol/g); n.r. = no reaction.
[b] Determined by HPLC analysis.

above conditions. The results are summarized in Table 2. A variety of quinolones **4** containing neutral, electron-rich, and electron-poor aryl substituents could be obtained in good yields. HPLC analysis showed that the purities of the products were >90% in most cases. 1-alkyl substituents were also tolerated. However, the yield of products **4d**, **4e**, and **4f** was low (50–55%) when using 30% H_2O_2 at 0–25 °C as the traceless cleavage reaction conditions of



Scheme 2

Table 2 Solid-Phase Synthesis of 4-Quinolone **4**[17]



Entry	R^1	R^2	R^3	R^4	Product	Yield (%)[a]	Purity (%)[d]
1	Bn	H	H	H	**4a**	89[b]	>95
2	Me	H	H	H	**4b**	77[b]	>95
3	Et	H	H	H	**4c**	76[b]	>95
4	H	H	H	H	**4d**	50[b] 70[c]	– >90
5	H	H	F	H	**4e**	52[b] 72[c]	– >90
6	H	H	OMe	OMe	**4f**	55[b] 70[c]	– >90
7	Me	H	H	OMe	**4g**	79[b]	>95
8	Bn	F	H	Br	**4h**	82[b]	>95
9	Bn	H	H	OMe	**4i**	68[b]	>95
10	Bn	H	F	H	**4j**	77[b]	>95
11	Bn	H	H	CF_3	**4k**	80[b]	>90
12	Bn	Cl	H	Cl	**4l**	75[b]	>85
13	Bn	H	OMe	OBn	**4m**	69[b]	>90
14	Bn	H	H	Cl	**4n**	84[b]	>95
15	Bn	H	H	Br	**4o**	86[b]	>95
16	Bn	H	H		**4p**	73[b]	>85
17	Bn	H	H	OBn	**4q**	83[b]	>90
18	Bn	H	OMe	OMe	**4r**	65[b]	>85

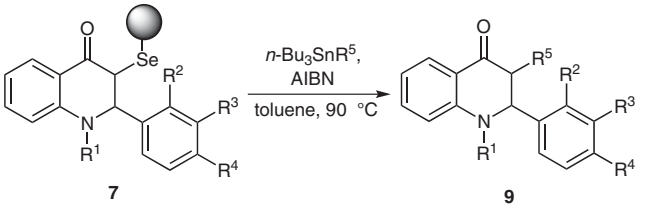
[a] Yields of the crude products based on the loading of selenium bromide resin (Br, 0.99 mmol/g).
[b] The reaction conditions of selenide oxidation and elimination is 30% H_2O_2, THF, 0 °C 1 h, then r.t., 20 min.
[c] Selenide oxidation and elimination reaction involves two steps: (a) 30% H_2O_2, THF, 0 °C, 2 h; (b) CCl_4, 80 °C, 10 min.
[d] Determined by HPLC analysis.

selenium linker. The yield of **4d**, **4e**, and **4f** was significantly improved when selenide was oxidized by 30% H_2O_2 in THF at 0 °C for 2 hours, then the oxidant was washed off, and the selenoxide was eliminated in CCl_4 under heating conditions (Table 2, entries 4–6).

The reaction of 1-(2-acetylaminophenyl)-3-phenylprop-2-en-1-one (**6a**) with polystyrene-supported selenenyl bromide afforded the intramolecular aminoselenation products **7** which could also be obtained from the acetylation of **2** in pyridine or in Et_3N–CH_2Cl_2. However, the reaction of **7a** with hydrogen peroxide did not afford 1-acetyl-2-phenylquilolin-4(1*H*)-one (**8a**). The structure of products **7a** was confirmed by the reduction of **7a** with tributyltin hydride and afforded 1-acetyl-2-phenyl-2,3-dihydroquinolin-4(1*H*)-one (**9a**, Scheme 2).

Furthermore, 1-acetyl-3-allyl-2-phenyl-2,3-dihydroquinolin-4(1*H*)-one (**9b**) was obtained by using allyltributylstannane as the cleavage reagent of selenium linker. Therefore, a series of substituted 2-phenyl-3-polystyrene-supported seleno-2,3-dihydroquinolin-4(1*H*)-ones **7** were

Table 3 Solid-Phase Synthesis of 4-Keton-2-phenyl-1,2,3,4-tetrahydroquinoline **9**[18]

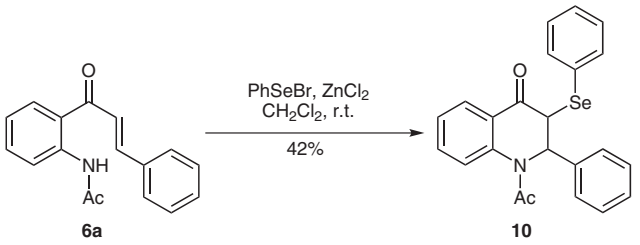


Entry	R^1	R^2	R^3	R^4	R^5	Product	Yield (%)[a]	Purity (%)[b]
1	Ac	H	H	H	H	**9a**	82	>95
2	Ac	H	H	H	Allyl	**9b**	79	>90
3	Ac	H	OMe	H	H	**9c**	78	>90
4	Ac	H	H	OMe	H	**9d**	85	>95
5	Ac	H	OMe	H	Allyl	**9e**	70	>90
6	Ac	H	H	OMe	Allyl	**9f**	73	>85
7	Ac	H	H	F	H	**9g**	80	>90
8	Ac	H	H	F	Allyl	**9h**	68	>85
9	Ac	H	H	Br	H	**9i**	83	>90
10	Ac	H	Br	H	H	**9j**	75	>85
11	Ac	H	H	Br	Allyl	**9k**	69	>80
12	Ac	H	Br	H	Allyl	**9l**	74	>85
13	Ac	H	H	CF_3	H	**9m**	65	>80
14	Ac	H	H	CF_3	Allyl	**9n**	70	>85
15	Ac	Br	H	H	H	**9o**	55	>80
16	Ac	Br	H	H	Allyl	**9p**	50	>80
17	H	H	H	H	H	**9q**	68	>80

[a] Yields of the crude products based on the loading of selenium bromide resin (Br, 0.99 mmol/g) via route A.
[b] Determined by HPLC analysis.

synthesized, followed by free-radical hydrogenation and allylation reaction with tributyltin hydride and allyltributylstannane in the presence of 2,2-azobis(2-methylpropionitrile) (AIBN) in toluene at 90 °C to afford 2-phenyl-2,3-dihydroquinolin-4(1*H*)-ones **9** in good yields and purities. Substituents on benzene ring had little impact on the free-radical cleavage reaction (Table 3).

We have studied the reaction of 1-(2-acetylaminophenyl)-3-phenyl-2-propen-1-one (**6a**) (1.0 equiv) and benzeneselenenyl bromide[16] (1.0 equiv) catalyzed by 40 mol% anhydrous $ZnCl_2$ in CH_2Cl_2 at room temperature. 1-Acetyl-2-phenyl-3-(phenylselenyl)-2,3-dihydroquinolin-4(1*H*)one (**10**) was obtained in isolated yield of 42% (Scheme 3).



Scheme 3

However, in the solid-phase reaction, excess **6a** was used, and the yield of the product was significantly improved. Furthermore, after the reaction, only polymer-supported intermediate **7a** was obtained after washing with various solvents, and all other byproducts and remaining **6a** were washed off. The separation process was very simple.

In conclusion, a new methodology for the solid-phase synthesis of 1-alkyl-2-phenyl-4-quinolones and 2-phenyl-2,3-dihydroquinolin-4(1*H*)-ones has been established. The highlight of the synthetic strategy is that organoselenium resin is capable of loading substrates through electrophilic cyclization reactions. The target products were obtained in good yields and with high purities by the traceless or functionalizing cleavage of selenium linker. Furthermore, the easy workup procedure and mild reaction conditions provide an approach that is well suited for building the parallel libraries upon the basis of further transformation of 4-quinolones and 2,3-dihydroquinolin-4(1*H*)-ones. Further modifications of resin **3** are under way.

Supporting Information for this article is available online at http://www.thieme-connect.com/ejournals/toc/synlett.

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(16) Benzeneselenenyl bromide was prepared by the reaction of diphenyldiselenide (1.0 equiv) and bromine (1.0 equiv) in CH_2Cl_2 at r.t. for 1 hour.

(17) **General Procedure for Obtaining 4-Quinolone 4 via Route A**

An oven-dried 50 mL round-bottomed flask was charged with a suspension of the swollen polystyrene-supported selenenyl bromide (Br: 0.99 mmol/g) resin (1.0 g) in dry CH_2Cl_2 (20 mL). $ZnCl_2$ (40 mol%) was added. After stirring for 0.5 hour at r.t., substituted 2-aminochalcone **5** (5.0 mmol) was added, and the reaction was stirred for another 12 hours. The resin **3** was collected by filtration, washed with H_2O (4 × 20 mL), THF–H_2O (v/v = 3:1; 2 × 20 mL), THF (2 × 15 mL), MeOH (2 × 15 mL) and CH_2Cl_2 (2 × 15 mL) and dried in vacuo. To a flask containing the suspension of the swollen resin **3** in THF (20 mL) was added 30% H_2O_2 aq (1.0 mL), and the mixture was stirred for 1 hour at 0 °C, followed by 20 min at r.t. After the reaction, the mixture was filtered, and the resin was washed with CH_2Cl_2 (2 × 20 mL). The filtrate was washed with H_2O (2 × 10 mL), dried over $MgSO_4$, and evaporated to dryness in vacuo.

1-Benzyl-2-phenyl-4-quinolone (4a)

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.53 (1 H, d, *J* = 7.8 Hz), 7.56 (1 H, m), 7.45–7.26 (10 H, m), 6.98 (2 H, d, *J* = 7.3 Hz), 6.56 (1 H, s), 5.33 (2 H, s). ^{13}C NMR (125 MHz, $CDCl_3$): δ = 176.81, 155.64, 140.94, 135.98, 135.21, 132.57, 129.74, 128.98, 128.64, 128.01, 127.64, 126.65, 126.49, 125.34, 125.22, 124.09, 117.30, 112.59, 52.36. IR (KBr): ν_{max} = 1608, 1486, 763 cm^{-1}. HRMS: *m/z* calcd for $C_{22}H_{18}NO$ $[M + H]^+$: 312.1388; found: 312.1385.

(18) **General Procedure for the Preparation of 2-Phenyl-2,3-dihydroquinolin-4 (1*H*)-one (9)**

An oven-dried schlenk tube was charged with a suspension of the swollen resin **7** (0.5 g) in dry toluene (10 mL) under nitrogen atmosphere, and tributyltin hydride (0.291 g, 1.0 mmol) or allyltributylstannane (0.331 g, 1.0 mmol) and 2,2′-azobisisobutyronitrile (AIBN, 0.082 g, 0.5 mmol) were added after which the reaction mixture was heated to 90 °C for 2 hours. After cooling, the suspension was poured into a fritted funnel, and the resin was washed with CH_2Cl_2 (2 × 10 mL). The filtrate was then concentrated and 10% HCl (5 mL) was added, and the resulting solution was washed with hexanes (3 × 10 mL). The aqueous phase was then neutralized with 10% NaOH and extracted with Et_2O (2 × 10 mL). The combined organic layers were washed with brine (10 mL), dried over $MgSO_4$, and concentrated to afford the product **9**.

1-Acetyl-2-phenyl-2,3-dihydroquinolin-4 (1*H*)-one (9a)

^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.43 (3 H, s), 3.22–3.28 (1 H, m), 3.35–3.40 (1 H, m), 6.47 (1 H, br), 7.15–7.22 (7 H, m), 7.44–7.48 (1 H, m), 7.93 (1 H, d, *J* = 7.6 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 193.1, 170.0, 141.7, 137.9, 134.3, 128.5, 127.5, 127.2, 126.7, 126.0, 125.4, 125.0, 54.6, 42.5, 23.3. IR (film): ν_{max} = 1693, 1682, 1663, 1601, 1461, 694 cm^{-1}. HRMS: *m/z* calcd for $C_{17}H_{15}NO_2$: 265.1103; found: 265.1100.