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EFFICIENT CONSTRUCTION OF *N*-HETEROCYCLES FROM BENZYLIC ETHERS/ALCOHOLS AND *o*-SUBSTITUTED ANILINES WITHOUT USING ANY CATALYST AND ADDITIVE<sup>§</sup>

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**Abstract** — A novel method for the synthesis of *N*-heterocycles from benzylic ethers/alcohols with *o*-substituted aniline without using any catalyst and additive is developed. This protocol involves C-O bond cleavage of benzylic ethers, *N*-benzylation, and benzylic C-H amidation in one pot, the tandem oxidation-cyclization transformation may open the door for the easy generation of *N*-heterocycles.

*N*-Heterocyclic compounds are key units with physiological significance and pharmaceutical utility.<sup>1</sup> Especially for the quinazolinones core, has shown important biological activities such as anticancer,<sup>2</sup> antibacterial,<sup>3</sup> antidiabetic,<sup>4</sup> antihypertensive,<sup>5</sup> antiinflammatory,<sup>6</sup> antitussive activity.<sup>7</sup> In addition, benzimidazoles and benzothiazoles are also very useful building blocks for the development of pharmaceutical or biological molecules.<sup>8</sup> Generally, there are two methods for the synthesis of *N*-heterocyclic compounds. One is a multi-step process to construct *N*-heterocycles from simple starting materials, however, time consuming, tedious and often low yielding hinder their wide application.<sup>9</sup> The other method is direct cross-coupling of *o*-substituted anilines with aldehydes (ketones) or *in situ* aldehydes generation from alcohols, amines, carboxylic acid (or carboxylic acid derivative).<sup>10-15</sup> Although the second approaches provided more efficient, more straightforward and attractive method to produce *N*-heterocycles, transition metals, hazardous peroxides, and other additives are generally required. A metal-free conditions especially in the drug and pharmaceutical industry are highly desirable, due to transition metal catalysts are toxic and they must be removed from the products. Therefore, the development of a transition metal-free and environment friendly method for the synthesis of *N*-heterocycles using readily available starting materials is highly desirable.

In view of the drawbacks of existing routes and the importance of green chemistry, improvements are focused towards the development of synthetic models which use a "green" oxidant, such as readily available and nontoxic  $O_2$  or air. Herein, we communicate a novel metal-free and acid-free sp<sup>3</sup>C-H bonds oxidation, C-O bond cleavage of benzylic ethers with *o*-substituted anilines to construct *N*-heterocycles (Scheme 1). Compared to conventional methods, this procedure is distinguished by using biofriendly clean dioxygen oxidant without metal catalyst. This new method provides a general and environmentally friendly access to *N*-heterocyclic compounds.



## Scheme 1

We began our study with the reaction of *o*-aminobenzamide **1a** and benzyl methyl ether **2a** for the optimization of the present oxidation-cyclization strategy to construct quinazolin-4(3*H*)-one **3a** and the results were compiled in Table 1. When the reaction mixture was heated in DMF for 12 h at 120 °C, this aerobic oxidation reaction take place to produce the corresponding 2-phenylquinazolin-4(3*H*)-one compound **3a** in 30% isolated yield (Table 1, entry 1). To improve the yield of this reaction, 1,4-dioxane was used as solvent, affording **3a** in 55% yield (Table 1, entry 2). When DMSO was used as solvent, the reaction gave **3a** in 88% isolated yield (Table 1, entry 3). Other solvents such as toluene, benzene, hexane, 1,2-dichloroethane and MeCN were not effective to the present oxidation-cyclization reaction (Table 1, entries 4-8).

Table 1. Optimization of	the reaction conditions	s in the reaction	of 1a with 2a <sup>a</sup>
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Entry	Solvent	Yield of <b>3a</b> <sup>b</sup>	Entry	Solvent	Yield of <b>3a</b> <sup>b</sup>
1	DMF	30	6	hexane	
2	1,4-dioxane	55	7	1,2-dichloroethane	
3	DMSO	88	8	MeCN	
4	toluene		9°	DMSO	
5	benzene		10 <sup>d</sup>	DMSO	89

<sup>a</sup> Reaction conditions: **1a** (0.2 mmol), **2a** (0.6 mmol), sovent (1 mL), O<sub>2</sub> (1 atm) in a sealed glass tube (25 mL) at 120 °C, 12 h. <sup>b</sup> GC yields based on **1a** using dodecane as an internal standard. <sup>c</sup> 100 °C or N<sub>2</sub>. <sup>d</sup> 130 °C.

Interestingly, the temperature also plays an important role, and 120  $^{\circ}$ C serves as the suitable temperature. For example, the reaction does not occur at 100  $^{\circ}$ C or N<sub>2</sub> in DMSO (Table 1, entry 9), while further increase of reaction temperature to 130  $^{\circ}$ C leads to no evident improvement on product yield (Table 1, entry 10). Thus, the optimal set of conditions was determined as described in entry 3.

Under these optimized reaction conditions, the scope of substrates that could be used and the versatility of the reaction were investigated. As shown in Table 2, *o*-substituted anilines with substituted benzyl methyl ethers were employed in this reaction to synthesize a variety of *N*-heterocycles derivatives in good yields (Table 2, entries 1-3). In addition to benzyl methyl ether, the symmetric ether such as ether, substituted-benzyl ether also served as efficient substrates under the current reaction conditions and converted to the corresponding substituted quinazolin-4(3*H*)-one in 70%-91% yield (Table 2, entries 4-7). The reaction of *N*-methyl or 5-chloro substituted 2-aminobenzamides with substituted benzyl methyl ethers proceeded smoothly to give the corresponding product in good yield (Table 2, entries 8-9). To our delight, 2-phenylquinazoline has also been efficiently synthesized under the present aerobic oxidation-cyclization strategy (Table 2, entry 10). Using the current strategy, the protocol can also be applied to preparation of the five-membered compounds (Table 2, entries 11-12). The reaction of *o*-phenylenediamine and *o*-aminothiophenol with substituted benzyl methyl ethers provided benzyl methyl ethers provided.

**Table 2.** Metal-free aerobic oxidation of benzylic ethers and o-substituted anilines for the synthesis ofN-heterocycles<sup>a</sup>







<sup>a</sup> Reaction conditions: **1a-1f** (0.2 mmol), **2a-2g** (0.6 mmol), DMSO (1 mL), O<sub>2</sub> (1 atm) in a sealed glass tube (25 mL) at 120 °C, 12 h. <sup>b</sup> Isolated yields. <sup>c</sup> Et<sub>2</sub>O (0.3 mL).

After success in the elaboration of benzyl ether, we moved our attention to investigate a variety of alcohols as coupling partners. To our delight, most alcohols underwent smooth transformation to afford

the corresponding 2-substituted quinazolin-4(3H)-one in excellent yields, and the results are summarized in Table 3. For different substituted benzyl alcohols holding both electron-donating groups and electron-withdrawing groups on the aromatic cycles work well to give the corresponding quinazolines. The electronic properties of the substituents in the aromatic ring had not influence on the reaction rate.





<sup>a</sup> Reaction conditions: **1a** (0.1 mmol), alcohol **2h-2n** (0.6 mmol), DMSO (1 mL), O<sub>2</sub> (1 atm) in a sealed glass tube (25 mL) at 120 °C, 12 h, recharging oxygen after 6 h. <sup>b</sup> Isolated yields.

In conclusion, we have demonstrated an efficient aerobic  $sp^{3}C$ -H bonds oxidation-cyclization strategy to construct *N*-heterocycles from benzylic ethers/alcohols with *o*-substituted anilines. In the present system, metal-free  $sp^{3}C$ -H bond oxidation, C-O bond cleavage and C-N bond formation was achieved in one pot. This tandem reaction provides an environmentally friendly protocol to *N*-heterocyclic compounds with wide substrate scope.

## **EXPERIMENTAL**

## General

All manipulations were carried out under air atmosphere unless otherwise specified. The reactions were monitored by GC and GC-MS. The <sup>1</sup>H NMR and <sup>13</sup>C NMR spectra were obtained from a solution in CDCl<sub>3</sub> or DMSO- $d_6$  with tetramethylsilane (TMS) as internal standard on a Brucker ADVANCE III spectrometer at 400 MHz and 100 MHz, respectively. Flash column chromatography was performed using silica gel 300-400 mesh. *o*-Substituted anilines **1** were purchased from Energy Chemical, Alfa Aesar, Aladdin or Maya Reagent, benzylic ethers/alcohols **2** were purchased from Energy Chemical,.

# General procedure for the synthesis of N-heterocyclic compounds

A 25 mL Schlenk-type tube equipped with a magnetic stir bar was charged with *o*-substituted aniline **1a-1f**. The reaction tube was evacuated and back-filled with  $O_2$ . Under oxygen atmospheres, ethers or alcohols **2a-2n** and DMSO were added at room temperature, then the reaction mixture was stirred at 120 °C for 12 h. The reaction was monitored by TLC. After completion of the reaction, the resulting solution was cooled to room temperature, and neutralized with saturated NaHCO<sub>3</sub> aqueous solution. The product was extracted with EtOAc or CHCl<sub>3</sub>, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated in vacuum. The crude product was purified by flash column chromatography on silica gel to give *N*-heterocyclic compounds **3**.

**2-Phenylquinazolin-4(3***H***)-one (3a)**<sup>16</sup>: White solid, mp 236-238 °C; <sup>1</sup>H NMR (400 MHz, DMSO-d): δ 12.59 (s, br, 1H), 8.20 (t, *J* = 8.6 Hz, 3H), 7.84 (d, *J* = 7.2 Hz, 1H), 7.76 (d, *J* = 8.0 Hz, 1H), 7.57 (t, *J* = 9.0 Hz, 4H); <sup>13</sup>C NMR (100 MHz, DMSO-*d*): δ 162.8, 152.8, 149.1, 135.0, 133.2, 131.8, 129.1, 128.2, 127.9, 127.0, 126.3, 121.4. GC-MS: *m*/*z* = 222.

**2-(4-Methoxyphenyl)quinazolin-4(3***H***)-one (3b)**<sup>16</sup>: White solid, mp 248-251 °C; <sup>1</sup>H NMR (400 MHz, DMSO-*d*): δ 12.31 (s, br, 1H), 8.20 (d, *J* = 8.4 Hz, 2H), 8.14 (d, *J* = 7.6 Hz, 1H), 7.83 (t, *J* = 7.4 Hz, 1H), 7.71 (d, *J* = 8.0 Hz, 1H), 7.50 (t, *J* = 7.2 Hz, 1H), 7.10 (d, *J* = 8.4 Hz, 1H), 3.84 (s, 3H); <sup>13</sup>C NMR (100 MHz, DMSO-*d*): δ 162.8, 162.3, 152.3, 149.4, 135.0, 129.9, 127.8, 126.6, 126.3, 125.3, 121.1, 114.5, 55.9. GC-MS: *m*/*z* = 252.

**2-(4-Bromophenyl)quinazolin-4(3***H***)-one (3c)**<sup>16</sup>: white solid, mp 294-296 °C; <sup>1</sup>H NMR (400 MHz, DMSO-*d*):  $\delta$  12.61 (s, br, 1H), 8.13 (t, *J* = 9.4 Hz, 3H), 7.82 (d, *J* = 6.8 Hz, 1H), 7.75 (d, *J* = 7.2 Hz, 3H), 7.53 (d, *J* = 6.4 Hz, 1H); <sup>13</sup>C NMR (100 MHz, DMSO-*d*):  $\delta$  162.7, 152.0, 149.1, 135.2, 132.4, 132.1, 130.3, 128.0, 127.3, 126.4, 125.7, 121.5. GC-MS: *m/z* = 299.

**2-Methylquinazolin-4(3***H***)-one (3d)**<sup>17</sup>: White solid, mp 235-239 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 12.20 (s, br, 1H), 8.26 (d, *J* = 8.0 Hz, 1H), 7.75 (t, *J* = 7.6 Hz, 1H), 7.65 (d, *J* = 8.0 Hz, 1H), 7.45 (t, *J* = 7.6 Hz, 1H), 2.59 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 164.6, 157.7, 153.5, 149.6, 135.1, 127.2, 126.6, 126.4, 120.4, 22.3. GC-MS: *m/z* = 160. **2-(4-Bromophenyl)-3-methylquinazolin-4(3***H***)-one (3e)<sup>18</sup>: white solid, mp >300 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): \delta 8.22 (d,** *J* **= 7.2 Hz, 1H), 7.60-7.66 (m, 4H), 7.41 (d, J = 7.6 Hz, 3H), 3.42 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): \delta 162.5, 155.0, 147.1, 134.4, 134.2, 132.1, 129.8, 127.4, 127.2, 126.6, 124.5, 120.4, 34.2. GC-MS:** *m/z* **= 315.** 

**6-Chloro-2-(4-methoxyphenyl)quinazolin-4(3***H***)-one (<b>3**f)<sup>18</sup>: white solid, mp >300 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): 12.70 (s, br, 1H), 8.08 (s, 1H), 7.80 (d, *J* = 2.8 Hz, 4H), 7.45 (s, 1H), 7.16 (s, 1H), 3.86 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 162.5, 161.8, 152.8, 148.2, 135.1, 130.7, 130.0, 125.3, 124.9, 122.4, 114.5. GC-MS: *m/z* = 286.

**2-Phenylquinazoline (3g)**<sup>19</sup>: white solid, mp 98-100 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): 9.38 (s, 1H), 8.61 (d, *J* = 7.2 Hz, 2H), 8.03 (d, *J* = 8.4 Hz, 1H), 7.80 (d, *J* = 7.6 Hz, 2H), 7.51 (d, *J* = 7.2 Hz, 4H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): 161.0, 160.5, 150.7, 138.1, 134.1, 130.7, 128.7, 128.6, 127.3, 127.1, 123.6. GC-MS: *m/z* = 206

**2-(4-Methoxyphenyl)-1H-benzo**[*d*]imidazole (3h)<sup>20</sup>: white solid, mp 223-225 °C; <sup>1</sup>H NMR (400 MHz, DMSO-*d*): δ 12.94 (s, br, 1H), 7.77 (d, *J* = 7.2 Hz, 2H), 7.67 (s, 1H), 7.56 (s, *J* = 5.2 Hz, 1H), 7.47 (t, *J* = 7.8 Hz, 1H), 7.22 (s, 2H), 7.06 (d, *J* = 8.0 Hz, 1H), 3.87 (s, 3H); <sup>13</sup>C NMR (100 MHz, DMSO-*d*): δ 160.1, 151.5, 131.9, 130.6, 123.1, 122.2, 119.3, 119.2, 116.3, 111.8. GC-MS: *m/z* = 224.

**2-(4-Bromophenyl)benzo**[*d*]thiazole (3i)<sup>18</sup>: white solid, mp 129-131 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.98 (d, J = 8.0 Hz, 1H), 7.87 (d, J = 8.4 Hz, 2H), 7.81 (J = 8.0 Hz, 1H), 7.54 (d, J = 8.4 Hz, 2H), 7.42 (t, J = 7.6 Hz, 1H), 7.32 (t, J = 7.6 Hz, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  166.7, 154.1, 135.0, 132.5, 132.2, 128.9, 126.5, 125.5, 125.42, 125.39, 123.3, 121.6. GC-MS: m/z = 290.

**2-(4-Nitrophenyl)quinazolin-4(3***H***)-one (3j)**<sup>16</sup>: white solid, mp >300 °C; <sup>1</sup>H NMR (400 MHz, DMSO-*d*):  $\delta$  12.84 (s, br, 1H), 8.40 (d, *J* = 1.6 Hz, 4H), 8.18 (d, *J* = 8.0 Hz, 1H), 7.88 (t, *J* = 7.2 Hz, 1H), 7.80 (d, *J* = 8.0 Hz, 1H), 7.58 (t, *J* = 7.2 Hz, 1H); <sup>13</sup>C NMR (100 MHz, DMSO-*d*):  $\delta$ . 162.6, 151.9, 149.0, 135.1, 132.4, 132.1, 130.3, 127.9, 127.3, 126.4, 125.7, 121.5. GC-MS: *m/z* =267.

**2-(Thiophen-2-yl)quinazolin-4(3***H***)-one (3k)**<sup>16</sup>: white solid, mp 277-278 °C; <sup>1</sup>H NMR (400 MHz, DMSO-*d*): δ 12.65 (s, br, 1H), 8.23 (d, *J* = 3.6 Hz, 1H), 8.12 (d, *J* = 8.0 Hz, 1H), 7.86 (d, *J* = 4.8 Hz, 1H), 7.79 (t, *J* = 7.6 Hz, 1H), 7.65 (d, *J* = 8.4 Hz, 1H), 7.49 (t, *J* = 7.4 Hz, 1H), 7.24 (t, *J* = 4.2 Hz, 1H); <sup>13</sup>C NMR (100 MHz, DMSO-*d*): δ 162.3, 149.0, 148.3, 137.8, 135.2, 132.6, 129.9, 129.0, 127.4, 126.8, 126.5, 121.3. GC-MS: *m/z* = 228.

**2-(Naphthalen-2-yl)quinazolin-4(3***H***)-one (31)<sup>18</sup>**: white solid, mp 213-215 °C; <sup>1</sup>H NMR (400 MHz, DMSO-*d*):  $\delta$  12.70 (s, br, 1H), 8.23 (d, *J* = 8.0 Hz, 1H), 8.18 (d, *J* = 7.2 Hz, 1H), 8.13 (d, *J* = 8.4 Hz, 1H), 8.05 (d, *J* = 7.2 Hz, 1H), 7.87 (t, *J* = 7.2 Hz, 1H), 7.81 (d, *J* = 6.8 Hz, 1H), 7.75 (d, *J* = 8.0 Hz, 1H), 7.66 (t, *J* = 8.8 Hz, 3H); <sup>13</sup>C NMR (100 MHz, DMSO-*d*):  $\delta$  162.4, 154.1, 149.2, 135.0,

133.6, 132.1, 130.8, 130.7, 128.8, 128.1, 127.9, 127.5, 127.2, 126.8, 126.3, 125.6, 125.5, 121.7. GC-MS: *m*/*z* = 272.

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