ORGANIC LETTERS

2013
Vol. 15, No. 6
1282–1285

Ru-Catalyzed Synthesis of Dihydrofuroquinolines from Azido-cyclopropyl Ketones

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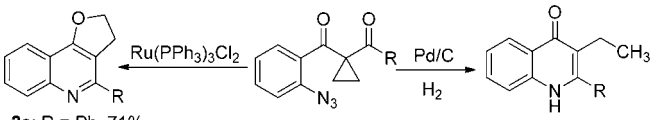
Received January 25, 2013

ABSTRACT



An efficient Ru-catalyzed synthesis of dihydrofuroquinolines from azido-cyclopropyl ketones via the reduction−cyclization−rearrangement process is reported. A plausible reaction mechanism for this process is depicted. Additionally, when the reaction was carried out under H_2 (1 atm) in the presence of Pd/C, 4-quinolones were obtained in excellent yields.

Heterocyclic compounds are worth our attention for their unique biological activities and wide utilities in agriculture, medicine, and material research. Because the class of quinoline derivatives features diverse biological and interesting properties, much attention has been focused toward developing efficient methods for their synthesis since the first discovery of quinoline by Gerhardt in 1842.[1] Recently, via Pd,[2] Rh,[3] Ni,[4] Ru,[5] Au,[6] and other metal catalysts,[7] various methods have been developed for the synthesis of quinolines. However, owing to the difficulty in constructing the pyridine and furan rings in a single step, there are few methods for the synthesis of

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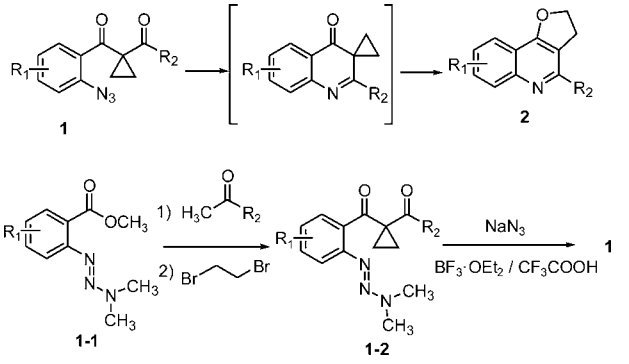
10.1021/ol400223d
Published on Web 02/22/2013

furoquinolines especially the dihydrofuroquinolines which are widely distributed in quinoline alkaloids.[8] Herein, we present a useful single step reaction involving the reduction−cyclization−rearrangement process to new dihydrofuroquinolines starting from azido-cyclopropyl ketones **1**.

Azides[9] and cyclopropyl ketones,[10] as useful building blocks for the construction of nitrogen- or oxygen-containing heterocyclic compounds, have been studied extensively. However, to the best of our knowledge, few studies on the synthetic utility of substrates bearing both azide and cyclopropyl ketone groups have been reported. For the purpose of constructing the dihydrofuroquinoline skeleton, we designed azido-cyclopropyl ketone and anticipated that when the azido group was reduced to an amine, it subsequently condensed with ketone to give the active intermediate which might be converted to the dihydrofuroquinoline **2** (Scheme 1). Starting with the readily available material of triazene **1-1**, the azido-cyclopropyl ketone **1** was easily achieved in three classic and functional group tolerant steps: (1) the Claisen reaction to give triazene-1,3-diketone, (2) the K_2CO_3-mediated cyclopropanation of triazene-1,3-diketone with 1,2-dibromoethane to afford the triazene-cyclopropyl ketone **1-2**, (3) the reaction of triazene-cyclopropyl ketone **1-2** with NaN_3 in the presence of $BF_3{\cdot}OEt_2$/TFA to give azido-cyclopropyl ketone **1** (Scheme 1 and Supporting Information).

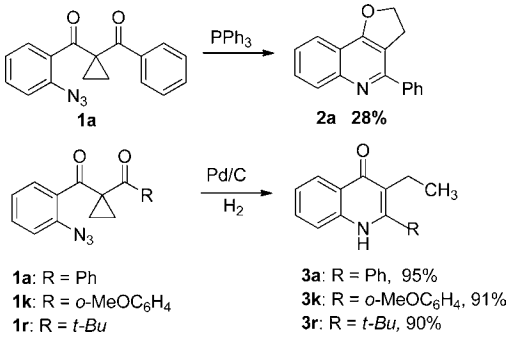
To test our hypothesis, we initially treated the azido-cyclopropyl ketone **1a** with PPh_3 in THF. We indeed achieved the desired product **2a** in 28% yield. Although various conditions, including solvents and reaction temperatures, were tested, the yield could not be increased.

Scheme 1. Our Strategy for the Synthesis of Dihydrofuroquinolines



However, when the reaction was carried out under H_2 (1 atm) in the presence of Pd/C, the 4-quinolone[11] **3a** was obtained instead of dihydrofuroquinoline **2a** in 95% yield (Scheme 2). The substrates of **1k** and **1r** can also be converted to their corresponding 4-quinolone **3k** and **3r** in high yields (90−91%) under H_2 reduction conditions (Scheme 2).

Scheme 2. Reducing Azido-cyclopropyl Ketone **1** with PPh_3 and Pd/C−H_2



To improve the yield of the dihydrofuroquinoline, other reaction conditions were tried. Upon treating azido-cyclopropyl ketone **1a** with $AuPPh_3Cl$ in CH_2Cl_2 at 50 °C, the starting material was recovered in 90% yield, and no trace of the desired quinoline compound was obtained (entry 1, Table 1). Further screening among various metal catalysts revealed that both $Rh_2(O_2CCH_3)_4$ and $InCl_3$ gave full recovery of the starting material (entries 2 and 3). To our delight, heating the substrate **1a** with $FeCl_2$ in CH_2Cl_2 at 50 °C afforded the desired quinoline **2a** in 10% yield (entry 4). When the reaction was carried out using $Ru(PPh_3)_3Cl_2$ as the catalyst in CH_2Cl_2 at 50 °C, the yield increased to 20% (entry 5). To optimize the result, the effect of the solvents was further investigated. Changing the solvent to toluene, DMF, CH_3CN, and EtOH did improve the yield from 20% to 30%, 55%, 64%, and 56%, respectively (entries 6−9). Interestingly, when the reaction was carried out in EtOH, the byproduct **2a′** (35% yield) was obtained with the desired product **2a** (56% yield).

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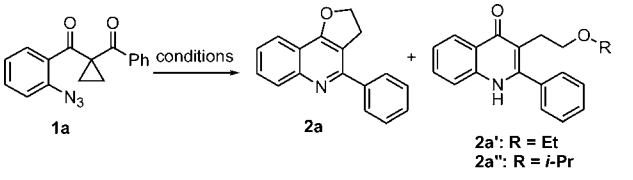
When we changed the solvent to *i*-PrOH, whose nucleophilicity is lower than EtOH, the yield of the desired product **2a** increased to 71%, and the byproduct **2a″** decreased to 11%. Thus, the best result was obtained when the reaction was carried out in *i*-PrOH at 80 °C using $Ru(PPh_3)_3Cl_2$ as the catalyst (entry 10). As expected, when the reaction was carried out in the absence of a catalyst, the starting material **1a** was recovered (entry 11).

Table 1. Optimization of Reaction Condition[a,b]



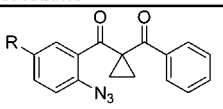
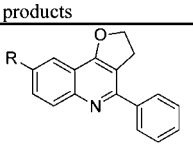
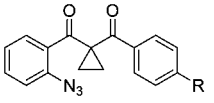
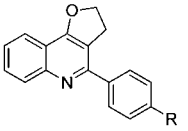
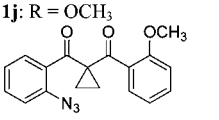
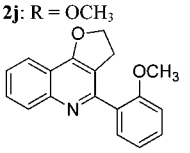
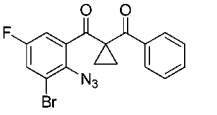
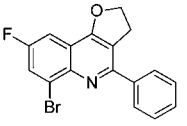
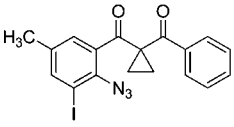
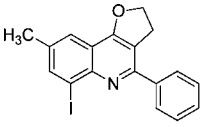
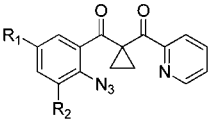
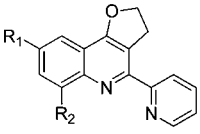
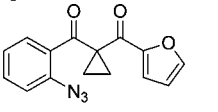
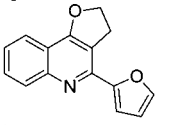
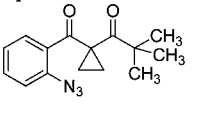
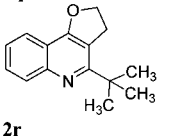
Solvent: CH_3CH_2OH, **2a**: 56%; **2a'**: 35%
i-PrOH, **2a**: 71%; **2a''**: 11%

entry	cat.	solvent	temp	yield **2a** (%)
1	$AuPPh_3Cl$	CH_2Cl_2	50	0
2	$Rh_2(O_2CCH_3)_4$	CH_2Cl_2	50	0
3	$InCl_3$	CH_2Cl_2	50	0
4	$FeCl_2$	CH_2Cl_2	50	10
5	$Ru(PPh_3)_3Cl_2$	CH_2Cl_2	50	20
6	$Ru(PPh_3)_3Cl_2$	toluene	80	30
7	$Ru(PPh_3)_3Cl_2$	DMF	80	55
8	$Ru(PPh_3)_3Cl_2$	CH_3CN	80	64
9	$Ru(PPh_3)_3Cl_2$	EtOH	80	56[c]
10	$Ru(PPh_3)_3Cl_2$	*i*-PrOH	80	71[d]
11	no	*i*-PrOH	80	0

[a] All reactions were carried out with azido-cyclopropyl ketone **1a** (1.0 mmol) and catalyst (0.05 mmol, 5 mol %) in solvent (4 mL) for 36 h. [b] Yield of isolated product after flash column chromatography. [c] When the reaction was carried out in EtOH, the byproduct **2a′** (35% yield) was obtained with the desired product **2a** (56% yield). [d] When we used *i*-PrOH as the solvent, the yield of the desired product **2a** increased to 71% and the byproduct **2a″** decreased to 11%.

With the optimized conditions in hand, the scope of this reaction was further investigated, and the results are summarized in Table 2. The transformation was sensitive to the electronic nature of the aryl azide substituent: the yield of quinoline decreased as stronger electron-withdrawing groups (entries 1–4, Table 2) were directed to the aryl azide motif. The electronic identity of the phenyl acetone aryl group also influenced the yield of the transformation. As the electron-withdrawing ability in the phenyl acetone motif weakened, a decrease in yield was observed (entries 5–8). But when it came to a strong electron-donating group, such as OCH_3, the yield increased to 86% (entry 9). Owing to the steric hindrance of the *o*-OCH_3 (entry 10), the *o*-OCH_3-substituted substrate **1k** gave the desired product **2k** in a lower yield (66%) compared to the *p*-OCH_3-substituted substrate **1j**. Sterically hindered groups in the aryl azide motif, such as *o*-bromo and *o*-iodo, can also be converted to their corresponding quinolines in good yields (74–84%, entries 11 and 12). Heteroaryl motifs, such as pyridyl and furanyl groups, were successfully incorporated into the

Table 2. Scope of Synthesis of Functionalized Quinoline Derivatives from Azido-cyclopropyl Ketones[a,b]

entry	substrates	products	yield (%)
			
1	**1b**: R = Ph	**2b**: R = Ph	75
2	**1c**: R = I	**2c**: R = I	77
3	**1d**: R = Cl	**2d**: R = Cl	61
4	**1e**: R = CN	**2e**: R = CN	53
			
5	**1f**: R = F	**2f**: R = F	76
6	**1g**: R = Cl	**2g**: R = Cl	74
7	**1h**: R = Br	**2h**: R = Br	60
8	**1i**: R = I	**2i**: R = I	62
9	**1j**: R = OCH_3	**2j**: R = OCH_3	86
			
10	**1k**	**2k**	66
			
11	**1l**	**2l**	84
			
12	**1m**	**2m**	74
			
13[c]	**1n**: $R_1 = R_2 = H$	**2n**: $R_1 = R_2 = H$	50
14[c]	**1o**: R_1 = Br, R_2 = H	**2o**: R_1 = Br, R_2 = H	61
15[c]	**1p**: $R_1 = CH_3$, R_2 = I	**2p**: $R_1 = CH_3$, R_2 = I	57
			
16[c]	**1q**	**2q**	71
			
17	**1r**	**2r**	78

[a] All reactions were carried out with azido-cyclopropyl ketone **1** (1.0 mmol) and $Ru(PPh_3)_3Cl_2$ (0.05 mmol, 5 mol %) in *i*-PrOH (4 mL) at 80 °C for 36 h. [b] Yield of isolated product after flash column chromatography. [c] The reaction was carried out at 120 °C.

reduction–cyclization–rearrangement products, as shown in the formation of products **2n**–**q**. When a pyridine unit was introduced to the substrate, the pyridyl-quinoline compounds, which are widely used as bidentate nitrogen ligands, were obtained in moderate yields of 50–61%

(entries 13–15). In the case of the furoaryl azide substrate, the furoquinoline **2q** was obtained in 71% yield (entry 16). Additionally, it was worth mentioning that upon treating substrate **1r**, which bears an aliphatic *t*-Bu group under the optimized conditions, the corresponding quinoline **2r** was obtained in good yield (78%, entry 17).

The structure of **2j** was confirmed unambiguously by X-ray diffraction analysis, which was in accordance with ^{1}H NMR, ^{13}C NMR, and HRMS spectra (Figure 1).

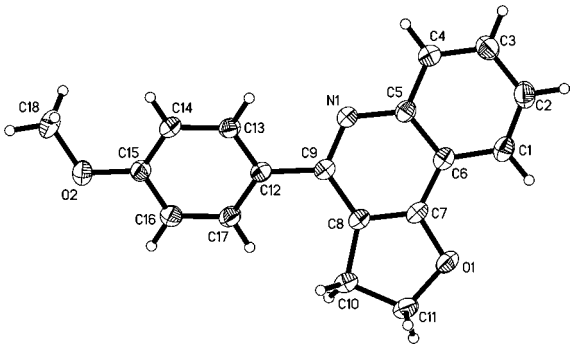


Figure 1. X-ray crystal structure of compound **2j**.

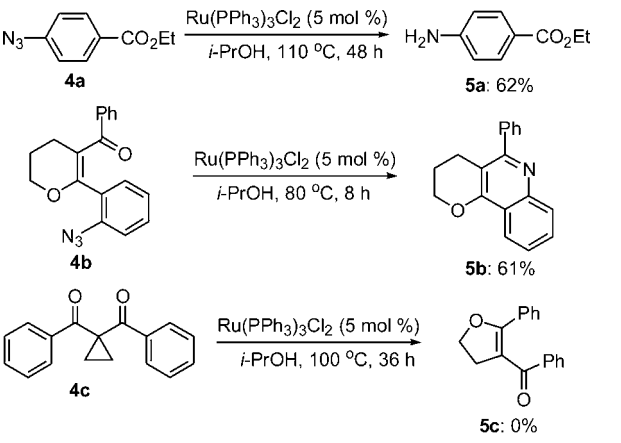
Finally, to unveil the reaction mechanism, some control reactions were carried out. When the reaction of 4-azido-benzoic acid ethyl ester **4a** was carried out in the presence of $Ru(PPh_3)_3Cl_2$ (5 mol %) at 110 °C, the 4-amino-benzoic acid ethyl ester **5a** was obtained in 62% yield.[12] When azide **4b**, bearing an *o*-substituted ketone, was heated under similar conditions, the reduction–cyclization product **5b** was formed in 61% yield. In the case of the substrate of cyclopropyl ketone **4c**, the rearrangement product **5c** was not formed under the heating reaction conditions in the presence of $Ru(PPh_3)_3Cl_2$ (5 mol %). These results indicate that the formation of the aromatic system of quinoline facilitates the rearrangement (Scheme 3).

On the basis of the above-mentioned results, a plausible reaction mechanism for this process is depicted in Scheme 4. Initially, the azide group was reduced to an amine in the presence of $Ru(PPh_3)_3Cl_2$ in *i*-PrOH under heating conditions, which subsequently condensed with ketone to provide the intermediate **6**. As a cyclopropyl ketone, a well-known precursor to dihydrofuran, intermediate **6** was further converted to the rearrangement product dihydrofuroquinoline **2** via [1, 3] transposition. Meanwhile, attack by the solvent *i*-PrOH on intermediate **6** led to a ring-opening reaction, generating the byproduct **2″**. Also upon hydrogenation of the cyclopropyl motif of the intermediate **6** in the presence of Pd/C–H_2, the 4-quinolone type product **3** was obtained (Scheme 4).

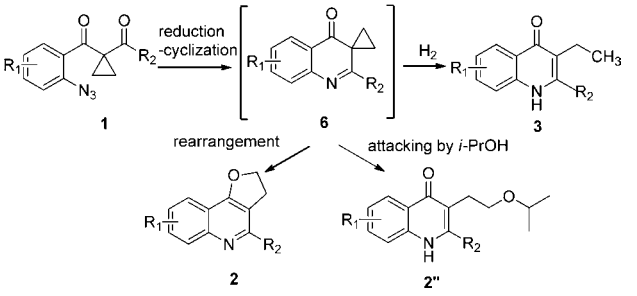
In summary, we have developed a Ru-catalyzed approach to the regioselective synthesis of highly substituted

(12) The reduction of azides to amines is an important transformation in organic synthesis. Recently, David R. Liu contributed a biomolecule-compatible, Ru(II)-catalyzed azide reduction reaction induced by visible light. Chen, Y.; Kamlet, A. S.; Steinman, J. B.; Liu, D. R. *Nat. Chem.* **2011**, *3*, 146.

Scheme 3. Investigation of the Reaction Mechanism



Scheme 4. A Plausible Reaction Mechanism



dihydrofuroquinolines in good to excellent yields from azido-cyclopropyl ketones. These reactions are simple and have been carried out under mild conditions. Mechanism investigations indicate that this reaction may involve the reduction–cyclization–rearrangement process, and the formation of the aromatic system may promote the rearrangement process. Additionaly, the 4-quinolones were obtained in excellent yields when the reaction was carried out under Pd/C–H_2 reduction conditions. Further investigations of the reaction mechanism and synthetic applications are underway.

Acknowledgment. We are thankful for the financial support from the National Nature Science Foundation of China (21272003) and the support from Zhejiang Provincial Top Key Academic Discipline of Applied Chemistry and Eco-Dyeing & Finishing, and Engineering Research Center for Eco-Dyeing & Finishing of Textiles (1206821-Y, Zhejiang Sci-Tech University).

Supporting Information Available. Detailed experimental procedures, characterization data, copies of ^{1}H and ^{13}C NMR spectra for all products, and CIF data of **2j** are provided. This material is available free of charge via the Internet at http://pubs.acs.org.

The authors declare no competing financial interest.