TMSCl-Catalyzed Tandem Reaction of Dihydroisobenzofuran Acetals with Indoles

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Abstract: A TMSCl-catalyzed tandem reaction of dihydroisobenzofuran acetals with indoles has been developed, which could provide an efficient and straightforward access to various tetrahydroisoquinolones in moderate to excellent yields. This process involved the first addition of the indoles to acetals, followed by skeletal rearrangement.

Keywords: organocatalysis; tandem reaction; dihydroisobenzofuran acetals; indoles; tetrahydroisoquinolones

1. Introduction

Isoquinoline frameworks widely exist in many natural bioactive products and synthetic pharmaceutical molecules (Figure 1) [1–5]. Among them, the C1-substituted isoquinolines constitute an important group. For example, the α-phenylisoquinoline solifenacin is an FDA-approved drug for urge incontinence [6]. α-Indolisoquinoline-like IBR2 has a significant inhibitory effect on triple-negative human breast cancer cells [7–10]. Such compounds are typically prepared by metal catalyzed cross-dehydrogenative coupling [11–13]. Recently, we developed an acid catalyzed dearomative arylation strategy for the synthesis of α-indolisoquinolines [14]. We also found that the tandem reaction of dihydroisobenzofuran acetal with indoles could afford tetrahydroisoquinolones [15]. However, such a process was highly substrate dependant, and only 5-OMe dihydroisobenzofuran acetal was suitable for this reaction. Given the potential medicinal value of Cl-indole substituted isoquinoline structures, it is of great significance to synthetise α-indole isoquinolines with structural diversity in a mild, direct, and efficient manner.

Figure 1. Biologically Active Compounds.

$N$-Sulfonyl-1,2,3-triazoles have received considerable attention, since they could serve as precursors of metal-bound imino carbenes or ketenimine intermediates [16–36]. Recently, we found dihydroisobenzofuran acetals, prepared easily from $N$-sulfonyltriazoles [37], are good substrates for tandem reaction, because they could undergo nucleophilic addition and subsequent skeletal
By the catalysis of a phosphoric acid catalyst, the reaction firstly experiences an intermediate A, which can undergo intramolecular Michael or aza-Michael addition to selectively give amino indanones or tetrahydroisoquinolones frameworks. However, the process for the formation of tetrahydroisoquinolones can be challenging, because this type of reaction requires special substrate of 5-OMe dihydroisobenzofuran acetal and gives low to moderate yields, which greatly limit its use. In addition, Yang et al. found that rhodium could promote the reaction of N-sulfonyl triazoles with indoles in tandem manner (Figure 2b) [38]. In this case, the intramolecular Michael addition was realized to give amino indanones, which also demonstrated a challenge for the formation of tetrahydroisoquinolones. Intrigued by the formation of tetrahydroisoquinolones, we tried to optimize this particular process to expand the scope of dihydroisobenzofuran acetals.

2. Results and Discussion

As part of our efforts to develop an efficient and straightforward access to various indole-substituted isoquinolinones, we herein reported an efficient method to synthesize indole-substituted tetrahydroisoquinolones through a TMSCl-catalyzed tandem reaction of dihydroisobenzofuran acetals with indoles.

Figure 2. Related Research and This Work. (a) Switchable Skeletal Rearrangement of Dihydroisobenzofuran Acetals with Indoles. (b) Rhodium-catalyzed tandem reaction of N-sulfonyl triazoles with indoles.
with indoles (see Supplementary Materials). According to our previous study, phosphoric acid could promote the reaction of 5-OMe dihydroisobenzofuran acetal with indoles to give indole-substituted tetrahydroisoquinolones. We wondered whether the use of an appropriate acid catalyst could promote this reaction with broader substrate scope. To explore this feasibility, we conducted the reaction of isobenzofuran acetal 1a and commercially available indole 2a by using several acids in CHCl₃ at 0 °C for 2 h. When CF₃CO₂H was employed as an acid catalyst, the reaction could proceed successfully to give the desired dihydroisoquinolinone product 3a in 34% yield (Table 1, entry 1), while no reaction occurred using AcOH (Table 1, entry 2). The structure of 3a was characterized by X-ray diffraction (Figure 3) [39]. Metal Lewis acid such as AlCl₃ could also catalyze this reaction, and the yield was improved to 58% (Table 1, entry 4). Further screening showed the use of trimethylchlorosilane could improve the yield to 60% (Table 1, entry 6). Meanwhile, the loading of indole 2a also played an important role on the yield. The results showed the yield could be increased from 60% to 76% by increasing the ratio of 2a:1a to 2.0 equiv (Table 1, entry 9). Further screening revealed that increasing the reaction temperature to room temperature resulted in an obvious increase in the yield to 84% (Table 1, entry 10). Next, we tested some other solvents with the indole loading of 1.2 equiv at room temperature. The best yield of 92% was obtained when dichloromethane was used (Table 1, entry 11), and no better results were obtained for other solvents such as dichloroethane, toluene, ethyl ether, methyl tert-butyl ether, and tetrahydrofuran (Table 1, entries 12–16). Finally, the reaction was conducted in the presence of 50 mol% TMSCl, with the 2.0 equiv of indole in dichloromethane at room temperature for 2 h.

**Table 1.** Optimization of the Reaction for the Synthesis of Indole-substituted Dihydroisoquinolinone.

| Entry | 1a (eq.) | 2a (eq.) | Acid (50 mol%) | T(°C) | Solvent | Yield (%) b |
|-------|----------|----------|---------------|-------|---------|-------------|
| 1     | 1.0      | 1.2      | CF₃CO₂H       | 0     | CHCl₃   | 34          |
| 2     | 1.0      | 1.2      | AcOH          | 0     | CHCl₃   | -           |
| 3     | 1.0      | 1.2      | ZnCl₂         | 0     | CHCl₃   | 39          |
| 4     | 1.0      | 1.2      | AlCl₃         | 0     | CHCl₃   | 58          |
| 5     | 1.0      | 1.2      | BF₃·Et₂O     | 0     | CHCl₃   | 24          |
| 6     | 1.0      | 1.2      | TMSCl         | 0     | CHCl₃   | 60          |
| 7     | 1.0      | 1.2      | TESOTf        | 0     | CHCl₃   | <10         |
| 8     | 1.2      | 1.0      | TMSCl         | 0     | CHCl₃   | 39          |
| 9     | 1.0      | 2.0      | TMSCl         | rt    | CHCl₃   | 76          |
| 10    | 1.0      | 2.0      | TMSCl         | rt    | CH₂Cl₂  | 84          |
| 11    | 1.0      | 2.0      | TMSCl         | rt    | DCE     | 78          |
| 12    | 1.0      | 2.0      | TMSCl         | rt    | toluene | 77          |
| 13    | 1.0      | 2.0      | TMSCl         | rt    | Et₂O    | 51          |
| 14    | 1.0      | 2.0      | TMSCl         | rt    | MTBE    | 64          |
| 15    | 1.0      | 2.0      | TMSCl         | rt    | THF     | 50          |
| 16    | 1.0      | 2.0      | TMSCl         | rt    | CHCl₃   |             |

a All the reactions were carried out in 0.05 M solvent with an acid catalyst. b Isolated yields.
Figure 3. X-ray structure of 3a.

Under the optimal reaction conditions, we next examined the generality and limitation of this tandem reaction (Table 2). A representative spread of indoles 2 worked well to afford the corresponding adducts 3. Both the electron-withdrawing group and electron-donating group on the different position at indoles could give the corresponding products in satisfactory yields (Table 2, entries 1–14). The 4-Methylindole could provide the desired product in 68% yield (Table 2, entry 2). Indoles with methyl, methoxy, benzyloxy, fluoro, chloro, and bromo substituents at C5 and C6 positions also provide satisfactory yields (71–86%) (Table 2, entries 3–11). C7-methyl, methoxy, and fluoro substituted indoles afford the products in 69–88% yields (Table 2, entries 12–14). The next was focused on different dihydroisobenzofuran acetals. Benzofuran acetals 1 with electron-donating substituents (-Me, -OMe) gave corresponding product in a 63–72% yield (Table 2, entries 18 and 19), while benzofuran acetals with electron-withdrawing substituents (-F, -Cl) led to lower yields (52–58%) (Table 2, entries 15–17 and 20).

According to our previous work [15], indole could attack dihydroisobenzofuran acetals to form the intermediate indol-dihydroisobenzofuran, which subsequently underwent an intramolecular (aza)-Michael addition to give amino indanones or tetrahydroisoquinolones. To further understand the regioselectivity of intramolecular (aza)-Michael addition under TMSCl, we next performed the DFT calculations (Figure 4). To make the calculation easier, HCl was used as a catalyst instead of TMSCl. The detailed formation process of intermediate A could be found in SI. Starting from intermediate A, the reaction could proceed through two routes. By path a, the enol intermediate B could be obtained via TS$_{A-B}$, while ketone intermediate C obtained via TS$_{A-C}$ by path b. Furthermore, intermediate B could undergo the Michael addition to form amino indanone D via TS$_{B-D}$, while intermediate C could provide tetrahydroisoquinolones E via TS$_{C-E}$. The energy for the formation of TS$_{A-B}$ ($\Delta G = 13.0$ kcal/mol) is higher than the formation of TS$_{A-C}$ ($\Delta G = 8.4$ kcal/mol). Therefore, the intermediate A was more likely to form tetrahydroisoquinolones E via path b.
Table 2. Scope for the Synthesis of 3a.

| Entry | R^1 | R^2 | 3 | Yield (%)^b |
|-------|-----|-----|---|-------------|
| 1     | H   | H   | 3a | 92          |
| 2     | 4-Me| H   | 3b | 68          |
| 3     | 5-Me| H   | 3c | 83          |
| 4     | 5-OBn| H  | 3d | 71          |
| 5     | 5-OMe| H | 3e | 77          |
| 6     | 5-F | H   | 3f | 75          |
| 7     | 5-Cl| H   | 3g | 82          |
| 8     | 6-Cl| H   | 3h | 78          |
| 9     | 6-F | H   | 3i | 77          |
| 10    | 6-Br| H   | 3j | 86          |
| 11    | 6-Me| H   | 3k | 78          |
| 12    | 7-OMe| H | 3l | 69          |
| 13    | 7-Me| H   | 3m | 88          |
| 14    | 7-F | H   | 3n | 77          |
| 15    | H   | 4-F | 3o | 54          |
| 16    | H   | 5-Cl| 3p | 57          |
| 17    | H   | 5-F | 3q | 58          |
| 18    | H   | 5-OMe| 3r | 72          |
| 19    | H   | 6-Me| 3s | 63          |
| 20    | H   | 6-F | 3t | 52          |

^a^ All reactions were performed with 1 (0.05 mmol), 2 (0.10 mmol), TMSCl (0.025mmol), and 4 Å MS (50 mg) in 1.0 mL of CH2Cl2 at room temperature for 2 h. ^b^ isolated yields.

Figure 4. Detailed Reaction Mechanism by DFT Calculations. A, B and C were the possible intermediates, D and E were the possible products.
3. Materials and Methods

In an ordinary vial, TMSCl (0.05 mmol, 50 mol%) was added to a solution of isobenzofuran acetal 1 (0.10 mmol, 1.0 equiv), indole 2 (0.20 mmol, 2.0 equiv), and 4 Å MS (100 mg) in dry CH$_2$Cl$_2$ (2 mL). After stirring for 2 h at room temperature, the solvent was removed under vacuum and residue was purified by flash column chromatography (petroleum ether/AcOEt 6:1) to give the pure desired products 3 as a whole solid.

4. Conclusions

In conclusion, we have developed a TMSCl-catalyzed tandem reaction of dihydroisobenzofuran acetals with indoles to easily access various indole-substituted tetrahydroisoquinolones in good yields. This strategy overcomes the limitation of substituents on the reaction under a simple and mild condition. A plausible reaction mechanistic was performed by DFT calculations, which indicated that it was easier to afford tetrahydroisoquinolones than amino indanone under TMSCl.

Supplementary Materials: The General Methods, Characterization Data, Computational Details, $^1$H and $^{13}$C NMR Spectra are available online at http://www.mdpi.com/2073-4344/10/4/392/s1.

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