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An innovative synthesis of tertiary hydroxyl thieno[2,3-*d*] pyrimidinone skeleton: natural-like product from the tandem reaction of *o*-aminothienonitrile and carbonyl compound

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ABSTRACT

A straightforward base accelerant tandem protocol for the synthesis of the tertiary hydroxyl natural-like thieno[2,3-*d*]pyrimidinone skeleton was developed from the cyclocondensation of *o*-aminothienonitrile and carbonyl compound. The reaction process includes PDF conversion and photo-catalytic oxygenation. This synthetic strategy offers an alternative method for regioselective construction of tertiary hydroxylated thieno[2,3-*d*]pyrimidinone architectures with kinetic, thermodynamic control, and six-member ring effect.

Introduction

The thienopyrimidinone nucleus is a basic structural feature which exists in many biologically active, pharmaceutical relevant natural products and synthetic analogues (Fig. 1, red). Substituted thienopyrimidinones have been assigned as privileged structures in drugs because of the remarkable pharmacological activities including anti-inflammation,[1] anti-malarial,[2] anti-plasmodial,[3] and anti-cancer.[4] The traditional synthetic approach toward thieno[2,3-*d*]pyrimidinone employs the condensation of ketones with *o*-aminothiophene dicarboxamide, which was derived by hydrolysis from the corresponding *o*-aminonitriles.[5] However, these synthetic methods suffer from significant limitations such as multistep sequences, time-consuming process, and use of toxic reagents. In recent years, the use of green procedure in one pot in place of traditional synthetic routes has attracted considerable attention.[6] To improve the desired properties,[7] the intentional introduction of a hydroxyl group on NPs or the biologically active natural-like products (NLPs) have been considered as one of the most facile strategies in pharmaceutical research (Fig. 1, blue).[8] Noteworthily, optically active tertiary hydroxyl chirality compound is a structural motif in numerous biological compounds and also serves as fundamental building blocks for various applications in organic synthesis.[9] Embedding an extra tertiary hydroxyl group on the NPs/NLPs could endow their outstanding bioactivity and metabolic stability.[10]

With respect to the valuable tertiary hydroxyl group and bioactivity of thieno[2,3-*d*]pyrimidinone, it was necessary to construct the novel tertiary hydroxyl thieno[2,3-*d*]pyrimidinone skeleton compound. The traditional synthesis of tertiary hydroxyl NLPs is a two step process: first, to prepare the NLPs skeleton compound; and second to modify the tertiary hydroxyl group by microbial epoxidation,[11] in vivo oxidation of secondary hydroxyl series,[12] alkylation of carbonyl compound,[13] reaction with α-furyllithium,[14] asymmetric Michael addition,[8,15] and photo-catalytic oxygenation.[16] To the best of our knowledge, there is rare study on the synthesis of the tertiary hydroxyl NLPs derivatives via a one-pot tandem approach. Inspired by the tertiary hydroxyl thieno[2,3-*d*] pyrimidinone potential bioactivity and continuing our interest in the construction of significant heterocyclic skeletons with *o*-aminonitriles as starting material, herein we are glad to report the discovery of tertiary hydroxyl thieno[2,3-*d*]pyrimidinone derivatives synthesized *via* a one-pot tandem approach of PDF reaction[17] and photo-catalytic oxygenation from *o*-aminothienonitrile and carbonyl compound.

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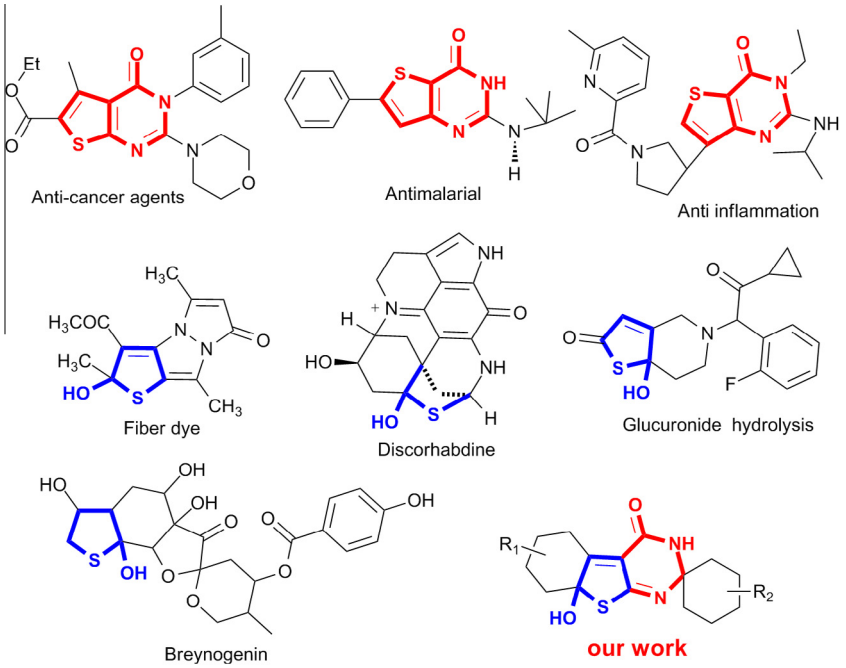
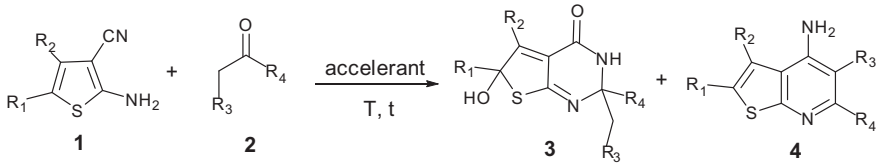
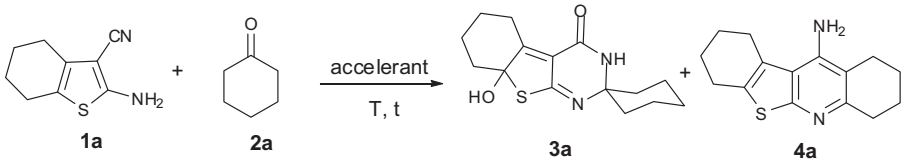


Figure 1. Selected examples of biologically active thienopyrimidinones, tertiary hydroxyl thiophene containing compounds, and our work.



Scheme 1. The divergent conversion from of *o*-aminothienonitrile **1** and ketone **2**.

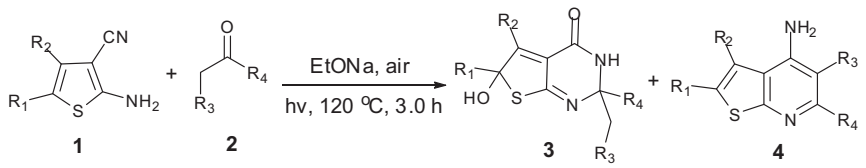
Table 1
Optimization of reaction conditions[a]



Entry	Accelerant(1 equiv)	Solvent	Temp (°C)	Time(h)	Yield[b] (%)	
					3a	**4a**
1	TsOH	**2a**	120	3	—[c]	46.8
2	PPA	**2a**	120	3	—	65.5
3	$AlCl_3$	**2a**	120	3	Trace	65.4
4	$ZnCl_2$	**2a**	120	3	Trace	63.5
5	NaOH	**2a**	120	3	35.0	25.2
6	MeONa	**2a**	120	3	45.9	23.3
7	EtONa	**2a**	120	3	65.7	20.6
8	EtONa	EtOH	120	3	35.5	10.9
9	EtONa	MeCN	120	3	12.7	13.5
10	EtONa	DMF	120	3	15.4	11.6
11	EtONa	Toluene	120	3	12.3	20.6
12	EtONa	**2a**	40	3	18.3	18.4
13	EtONa	**2a**	60	3	27.5	18.0
14	EtONa	**2a**	80	3	35.8	19.5
15	EtONa	**2a**	100	3	58.6	19.1
16	EtONa	**2a**	140	3	48.1	21.8

[a] Reactions conditions: 1a (1 mmol), **2a** (1.5 mmol), and accelerant (1 equiv) in solvent (3.0 mL) with UV lamp irradiation.
[b] Isolated yields.
[c] The target product was not found by TLC.

Table 2
Synthesis of hydroxylated thiophene derivatives **3**[a]



1 2 3 4

Entry	R_1, R_2	R_3, R_4	Product	Yield[b] (%)	Product	Yield[c] (%)
1	$R_1 + R_2 = (CH_2)_3$	$R_3 + R_4 = (CH_2)_3$	—[d]	—	**4b**	Trace
2	$R_1 + R_2 = (CH_2)_3$	$R_3 + R_4 = (CH_2)_4$	—	Trace	**4c**	33.5
3	$R_1 + R_2 = (CH_2)_3$	$R_3 + R_4 = (CH_2)_5$	—	Trace	**4d**	45.2
4	$R_1 + R_2 = (CH_2)_4$	$R_3 + R_4 = (CH_2)_3$	—	—	**4e**	Trace
5	$R_1 + R_2 = (CH_2)_4$	$R_3 + R_4 = (CH_2)_4$	3a	65.7	**4a**	20.6
6	$R_1 + R_2 = (CH_2)_4$	$R_3 + R_4 = (CH_2)_5$	—	Trace	**4f**	58.9
7	$R_1 + R_2 = (CH_2)_5$	$R_3 + R_4 = (CH_2)_3$	—	—	**4g**	Trace
8	$R_1 + R_2 = (CH_2)_5$	$R_3 + R_4 = (CH_2)_4$	—	Trace	**4h**	45.1
9	$R_1 + R_2 = (CH_2)_5$	$R_3 + R_4 = (CH_2)_5$	—	Trace	**4i**	54.3
10	$R_1 + R_2 = (CH_2)_4$	$R_4 = H$, $R_3 = CH_3$	—	Trace	**4j**	26.3
11	$R_1 + R_2 = (CH_2)_4$	$R_3 = CH_3$,$R_4 = CH_3$	—	Trace	**4k**	22.1

[a] Reactions conditions: 1 (1 mmol) and EtONa (1 equiv) in corresponding carbonyl compounds **2** (3.0 mL) with UV lamp irradiation.
[b] Isolated yields.
[c] Isolated yields or TLC detection yields.
[d] The products **3** were not found by TLC or MS.

Results and discussion

When the reaction of *o*-aminothienonitrile **1** and ketone **2** was carried out at 120 °C in the presence of EtONa (Scheme 1), the expected fused heterocyclic derivatives containing a tertiary hydroxyl moiety of thieno[2,3-*d*]pyrimidinone **3** was obtained. Additionally, a dehydration product, thieno[2,3-*b*]pyridines **4**, was also obtained, with its formation possibly based on a Friedländer condensation.[17]

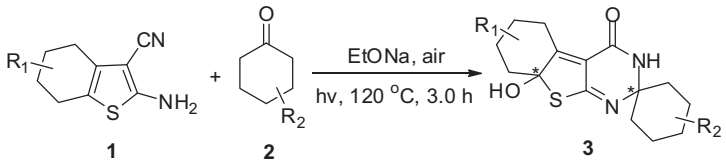
Initially, the reaction of 2-amino-4,5,6,7-tetrahydrobenzo-[*b*] thiophene-3-carbonitrile **1a** and cyclohexanone **2a** was chosen as the model substrate to optimize the conditions (Table 1). Various accelerants such as BrØnsted acids, Lewis acids, and base were evaluated for the reaction and the results showed that accelerant was a key factor to control the reaction route. The data indicated that only a single product **4a** was obtained in the presence of BrØnsted acids of TsOH and PPA (entry 1 and 2), while only a trace amount of product **3a** was observed by thin layer chromatography (TLC) when the Lewis acid of $AlCl_3$ and $ZnCl_2$ were used (entry 3 and 4). The results from Table 1 (entries 5–7) showed slightly decreasing yields of compound **4a** and significantly increasing yields of compound **3a** because of changing the base accelerant from NaOH to NaOMe to NaOEt, and strong base was beneficial to push the reaction forward. Similar reactions were then attempted in different solvents and the results showed that the cyclohexanone itself was the medium of choice for the formation of **3a** (entries 7–11).[18] Additionally, the yield of compound **3** was increased with the rasing of temperature, but 120 °C was the better selection (entries 12–16). It was worth mentioning that the products **3** could be isolated from the reaction mixture by filtration. Therefore, it can be inferred that the preparation of **3a** was a kinetically controlled reaction and the optimum reaction condition was reflux for 3.0 h in the presence of EtONa with cyclohexanone itself as medium with UV lamp irradiation in air.

In order to apply this reaction to a library synthesis, a series of *o*-aminothienonitrile **1** and ketone **2** were employed. Astonishingly, as shown in Table 2, only the reaction of 2-amino-4,5,6,7-tetrahydro-benzo[*b*]thiophene-3-carbonitrile **1a** and cyclohexanone **2a** provided the expected tertiary hydroxyl derivative **3a** (entry 5). When the cyclopentanone was tested, no transformation of tertiary hydroxyl derivatives and **4b**, **4e,** and **4g** occurred (entries 1, 4 and 7). Additionally, the products obtained were **4c**, **4d**, **4f**, and **4h–k** but not the corresponding tertiary hydroxyl thieno[2,3-*d*]pyrimidinone when other ketones were employed (entries 2, 3, 6, 8–11). Evidently, only the cyclocondensation of 2-amino-4,5,6,7-tetrahydrobenzo [*b*]thiophene-3-carbonitrile **1a** and cyclohexanone **2a** provided the expected tertiary hydroxyl derivative **3** with good yields perhaps for reasons of steric hindrance and ring tension.

Based on the unique phenomenon, we turned our attention to investigating the scope of substituted 2-amino-4,5,6,7-tetrahydrobenzo[*b*]thiophene-3-carbonitrile **1** and cyclohexanone derivatives **2** for preparing the tertiary hydroxylated thieno[2,3-*d*]pyrimidinone derivatives **3**. The results showed that the reactions were tolerated when **2** bear an electron-donating group (R_2 = 3-Me, 4-Me or 4-Et) (Table 3, entries 2–4 vs 1). Thereafter, the substituents (R_1 = Me) on **1** were investigated and the results showed that the corresponding derivatives **3** were also obtained (Table 3, entries 5 and 9 vs 1). And the reactions were also extended to the interactive variations of R_1 and R_2 (Table 3, entries 6–8 and 10–13). However, the correlation between the products and the molecular structures can currently be described only empirically. Overall, the hydroxylated thieno[2,3-*d*]pyrimidinone derivatives **3** were formed in a one-pot procedure while substituted 2-amino-4,5,6,7-tetrahydrobenzo[*b*]thiophene-3-carbonitrile **1** and cyclohexanone derivatives **2** used as the substrates perhaps for the charge effect, thermodynamic stability of six-member ring, and low strain of cyclohexane[19] collective effect.

The chemical structures of target compounds **3** were fully characterized by IR, ^{1}H NMR, ^{13}C NMR, and HRMS, while product **3a** and **3b** were unequivocally confirmed by X-ray diffraction analysis (Fig. 2). Take crystal data of **3a** as an example, the thieno[2,3-*d*] pyrimidinone skeleton was nearly coplanar while the six-membered rings of C(3)—C(4)—C(5)—C(6)—C(7)—C(8) and C(10)—C(11)—C(12)—C(13)—C(14)—C(15) were present in chair conformations. In addition, the structure confirmed there was a stereogenic center at C(4) with a tertiary hydroxyl group. Furthermore, the C(9)—N(2) (1.3412) and C(1)=N(1) (1.2761) bonds were

Table 3
Synthesis of six-membered hydroxylated fused thiophene derivatives **3**[a]



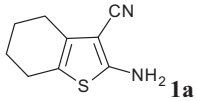
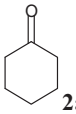
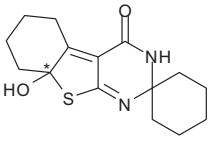
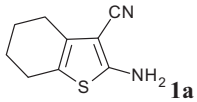
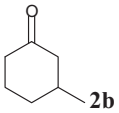
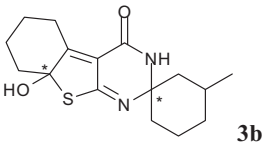
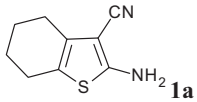
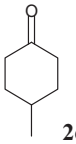
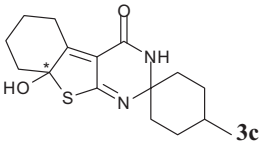
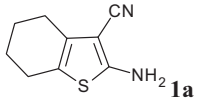
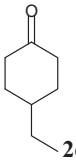
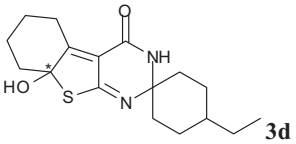
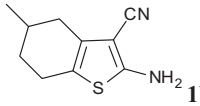
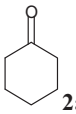
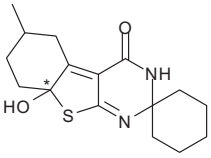
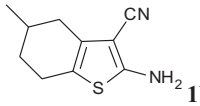
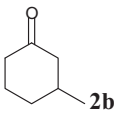
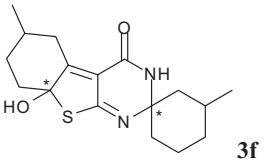
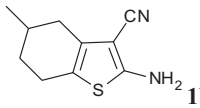
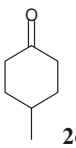
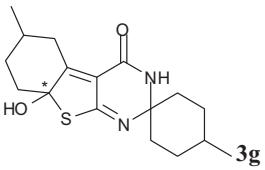
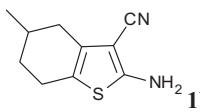
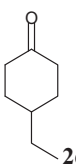
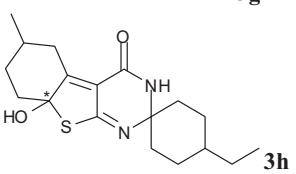
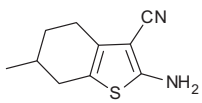
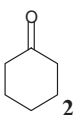
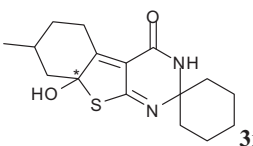
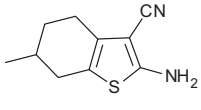
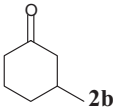
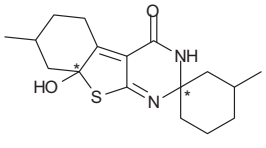
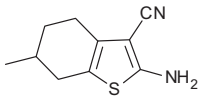
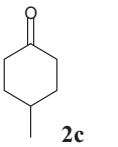
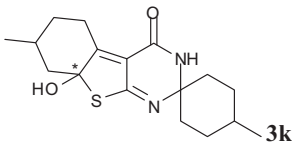
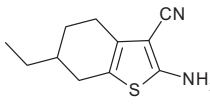
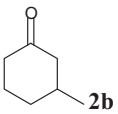
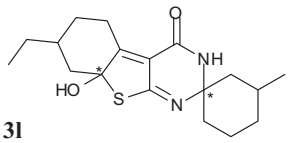
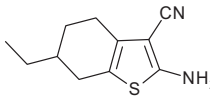
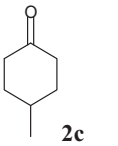
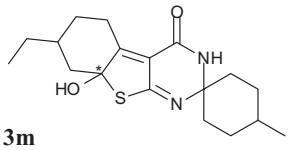
Entry	R_1	R_2	Product	dr[b]	Yield[c] (%)
1	 **1a**	 **2a**	 **3a**	–	65.7
2	 **1a**	 **2b**	 **3b**	>25:1	73.5
3	 **1a**	 **2c**	 **3c**	–	60.2
4	 **1a**	 **2d**	 **3d**	–	80.1
5	 **1b**	 **2a**	 **3e**	–	57.8
6	 **1b**	 **2b**	 **3f**	6:1	66.3
7	 **1b**	 **2c**	 **3g**	–	57.6
8	 **1b**	 **2d**	 **3h**	–	75.2
9	 **1c**	 **2a**	 **3i**	–	71.5

Table 3 (*continued*)

Entry	R_1	R_2	Product	dr[b]	Yield[c] (%)
10	 **1c**	 **2b**	 **3j**	>25:1	78.3
11	 **1c**	 **2c**	 **3k**	–	72.1
12	 **1d**	 **2b**	 **3l**	>25:1	70.8
13	 **1d**	 **2c**	 **3m**	–	59.6

[a] Reactions conditions: **1** (1 mmol) EtONa (1 equiv) in corresponding ketones **2** (3.0 mL) at 120 °C for 3.0 h with UV lamp irradiation.
[b] Detected by ^{1}H NMR.
[c] Isolated yields.

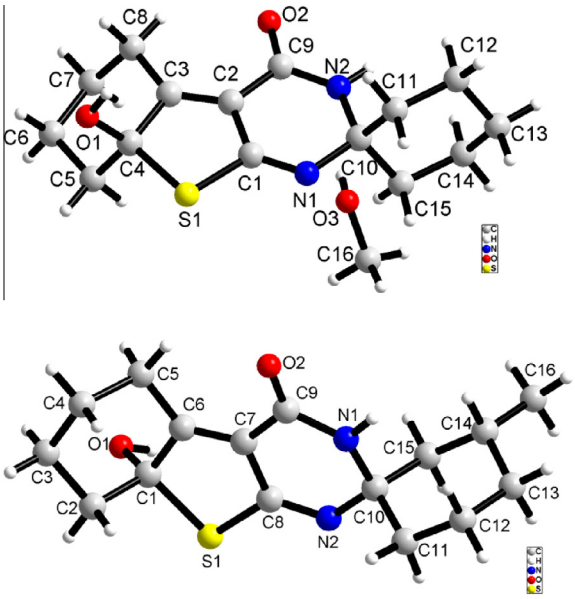
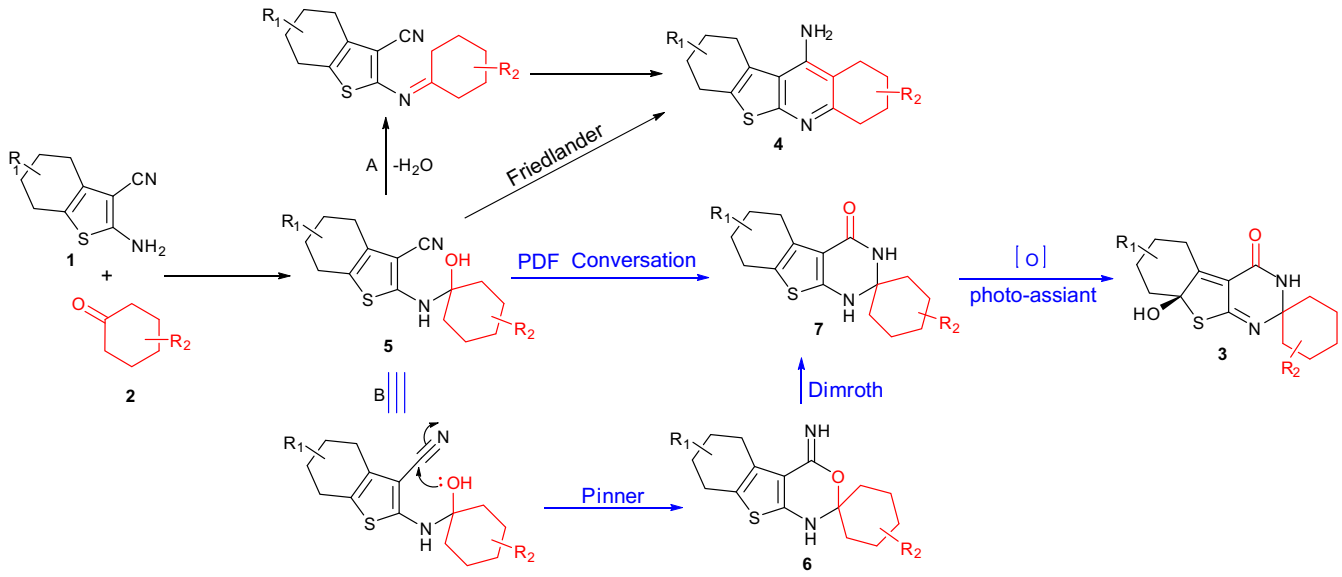


Figure 2. X-ray structures of **3a** (up) and **3b** (down).

remarkably shorter than the normal bond C—N (1.47) and C=N (1.38), which may be ascribed to the thieno[2,3-*d*]pyrimidinone fused-ring as a big π-conjugated system. Here, the C(9)—N(2) (1.3412) and C(1)=N(1) (1.2761) bonds were also remarkably shorter than the corresponding bonds (C—N (1.4293) and C=N (1.3016)) of the thieno[2,3-*d*]pyrimidinone skeleton without the hydroxylated group substitution which are prepared by the traditional method of aza-Wittig reaction.[20] Maybe the shorter bonds are attributed to the tertiary hydroxyl group electron-donating inductive effect. The further support was provided by the ^{1}H NMR titration experiments of **3l** at D_2O-DMSO-d_6 mixtures. Well resolved proton signals consistent with the chemical formulation, shifts, and splitting patterns were observed in the DMSO-d_6 solution. However, the proton signal of active hydrogens of both hydroxyl group and imino group disappeared when the hydrogen proton exchanged between D_2O with OH and NH groups (Fig. S1).

One possible mechanistic hypothesis was proposed and depicted in Scheme 2. Firstly, intermediate **5** was produced by the addition of the amino group of **1** onto the carbonyl group of cyclohexanone **2**; Then the Pinner reaction (path B) through attack by the hydroxyl group on the nitrile and subsequent Dimroth rearrangement happened with the dehydration reaction (path A). The pathways are controlled by the collective effect of kinetics, thermodynamics, and six-membered ring effect. The initial dehydration reaction route resulted in the formation of thieno[2,3-*b*] pyridines **4** via Friedländer reaction whereas the Pinner reaction way afforded the thieno[2,3-*d*]oxazine **6**. Subsequently **6** rearranged to the **7** (Dimroth rearrangement) (PDF conversion: a transformation from Pinner to Dimroth rearrangement in the Friedländer conditions[17]). Afterward, product **7** slowly translated to the hydroxylated target materials **3** via photo-oxygenation.[16,21]

In order to study the proposed mechanism and explain the thermodynamical favored product **3**, we tried to separate the intermediate thieno[2,3-*d*]pyrimidinone **7**. Fortunately, **7d** was detected by TLC and isolated by thin-layer chromatography. The structure of **7d** was further confirmed by IR, ^{1}H NMR, ^{13}C NMR, and HRMS. To our delight, a slow transformation of **7d** to its hydroxylated derivative **3d** occurred when a solution of **7d** in DMSO-d_6 in a nuclear magnetic resonance tube was irradiated with a 380 nm UV lamp or heated (Fig. 3). Contrasting the ^{1}H NMR spectra between **7d** and **3d** revealed that once the transformation occurred, the chemical shifts of H(a)—N(2) moved to high field region and, in contrast, H(b)—N(1) moved to low field relative to the new hydroxyl H(c)—O(1) signal. The conversion was further confirmed by the appearance of peaks at *m*/*z* 305.16844 and 321.16355 assignable to **7d** and **3d** in the ESI-HRMS spectrum



Scheme 2. Proposed mechanism for the new conversion.

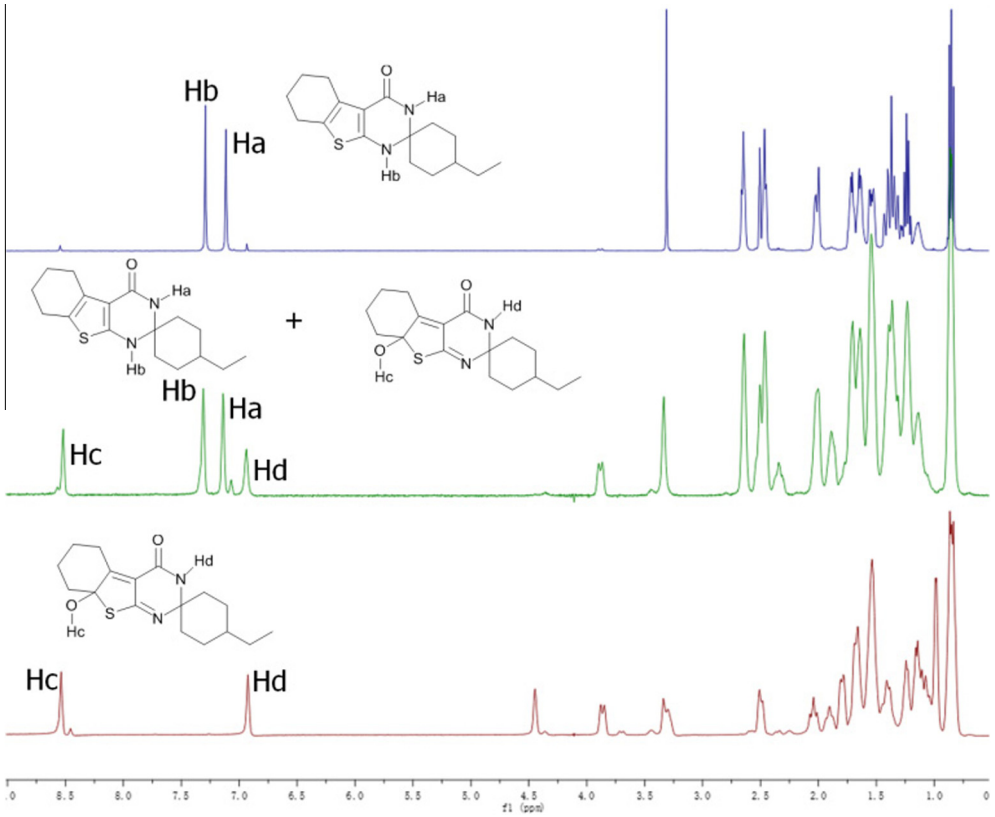


Figure 3. Part of the ^{1}H NMR spectra (DMSO-d_6) of transformation of **7d** to **3d**.

respectively (Fig. S2).[22] Furthermore, the product **3d** could be precipitated from the reaction solution via a slowly photo-catalytic oxygenation with 380 nm UV lamp irradiation. The process afforded a simple and clean isolation of the product by filtration and recrystallization without column chromatography. Furthermore, the control reactions in dark or without O_2 were run, and the product **3a** could not be captured. In the photo-catalytic oxygenation, singlet oxygen was the oxidative agent generated by the application of the oxygen.[16,23]

Conclusions

In summary, we have successfully developed a novel, efficient tandem procedure for the synthesis of a tertiary hydroxyl natural-like thieno[2,3-*d*]pyrimidinone skeleton products **3** from *o*-amino-thienonitrile **1** and carbonyl compound **2** in the presence of the $NaOCH_2CH_3$ with UV lamp irradiation. The tandem process includes PDF conversion and photo-oxygenation. The reaction was shown to have attractive features, including environmentally

friendly conditions, simple, clean isolated processing and large scale production.

Acknowledgments

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Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.tetlet.2016.04.088.

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