有机化学 DOI: 10.6023/cjoc202112032 研究论文
Chinese Journal of Organic Chemistry ARTICLE

可见光诱导杂芳烃与脂肪醇的羟烷基化反应

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摘要 发展了一种有效的可见光诱导的杂芳烃与脂肪醇的直接交叉脱氢偶联. 该反应在水溶液中由过硫酸盐引发, 不需要使用化学计量酸来活化杂环. 通过氢原子转移(HAT)从醇瞬时生成的 α-羟烷基自由基与一系列杂芳烃发生 Minisci 反应. 该方法具有无需光催化剂、溶剂绿色环保、反应条件温和、起始材料容易获得和官能团耐受性广泛等优点.
关键词 醇; 交叉脱氢偶联; 杂芳烃; 羟烷基化; 可见光诱导

Visible Light-Induced Hydroxyalkylation of Heteroarenes with Aliphatic Alcohols

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Abstract An efficient visible light-induced direct cross-dehydrogenative coupling of heteroarenes with aliphatic alcohols in aqueous solution at ambient temperature was developed. The reaction is initiated by persulfate salts and does not require the use of stoichiometric acid for activation of the heterocycle. α-Hydroxyalkyl radicals transiently generated from alcohols via hydrogen atom transfer (HAT) undergo Minisci-type reactions with a range of heteroarenes. This protocol was highlighted by photocatalyst-free, green solvent, mild conditions, readily available starting materials, and wide functional group tolerance.
Keywords alcohol; cross-dehydrogenative coupling; heteroarene; hydroxyalkylation; visible light-induction

1 Introduction

The formation of C—C bonds is very significant in the synthesis of natural products and bioactive molecules.[1] Cross-dehydrogenative coupling (CDC) reaction which directly utilizes the C—H bond of substrate to construct C—C bond has emerged as a new way to complex organic synthesis from simple raw materials in recent decades.[2] From a synthetic point of view, direct C—H/C—H cross-coupling is undoubtedly the most step- and atom-economical method.[3] The construction of C—C bonds from sp^3 and sp^2 hybrid carbons by the activation of the C—H bonds has attracted widespread attention as a participant in CDC reaction.[4] However, due to the high bond energy and large pK_a of C(sp^3)—H bond, the direct functionalization of C(sp^3)—H bond has become one of the most challenging areas of modern organic chemistry.[5] Alcohols are ubiquitous raw materials, which are indispensable in organic chemistry and chemical engineering.[6] The most common alcohols are methanol and ethanol, which are often used as excellent solvents.[7] Functionalized alcohol is an important scaffold, which exists in a variety of natural products and pharmaceutical ingredients.[8] These compounds exhibit a wide range of biological activities, including antibacterial, antimalarial and antagonistic.[9] Considering the synthetic value of introducing active alcoholic hydroxyl group, developing C—H functionalization of alcohols is of great significance in the fields of organic synthesis and biopharmaceuticals.[10]

In 2011, Li group[11a] reported palladium-catalyzed coupling of *N*-heterocycles with simple alcohols (Scheme 1, a). At the same time, Wang and co-workers[11b] developed a metal-free C(2)-alkylation of azoles with alcohols and ethers initiated by *tert*-butyl hydroperoxide (TBHP) (Scheme 1, b). Subsequently, di-*tert*-butyl peroxide (DTBP) oxidized CDC reactions of thiophenes, pyridines and chromones with alcohols were developed.[11c-11d] In 2019, Weng *et al.*[9a] reported $K_2S_2O_8$ oxidized CDC reaction of benzothiazoles with alcohols. These reactions are well developed for the hydroxyalkylation of heteroarenes, but they

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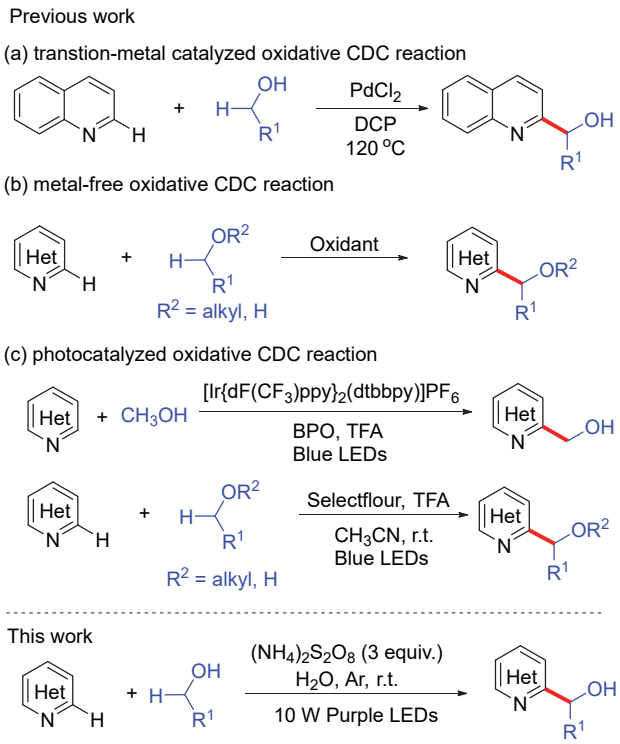
Received December 25, 2021; revised January 18, 2022; published online January 27, 2022.
Project supported by the the Anhui Laboratory of Molecule-Based Materials (No. fzj21005).
安徽省分子基材料实验室(No. fzj21005)资助项目.

are still carried out at relatively high reaction temperature. In recent years, visible light-induced photoredox catalysis has been widely used in green synthetic chemistry due to its high efficiency, mildness, environmental friendliness and low energy consumption.[12] Photocatalysis has also emerged as a powerful tool for C—H bond functionalization through the single-electron transfer (SET) pathway. In 2016, DiRocco group[13] developed iridium complex catalyzed hydroxymethylation of heteroarenes (Scheme 1, c). However, the photocatalysts used such as Ru(II) and Ir(III) complexes are high cost, high toxicity, limited availability and are not suitable for large-scale industrial production.[14] Therefore, how to directly utilize visible light to efficiently drive organic reactions without using additional photocatalysts has become an extremely challenging topic.[15] In 2016, Shah and co-worker[15a] reported a visible light-promoted C—H functionalization of ethers and electron-deficient arenes (Scheme 1, c). Nevertheless, the reaction substrate is limited to ether. Excitingly, Lei *et al.*[9b] reported visible light-induced direct *α*-C—H functionalization of alcohols with (iso)quinolines without the external photocatalyst (Scheme 1, c). Recently, Weng group[15b] developed visible light-induced direct hydroxyalkylation of 2*H*-benzothiazoles with alcohols. However, these reactions were carried out using selectfluor as oxidant in organic solvent in the presence of trifluoro acetic acid (TFA). Therefore, we developed an efficient visible light-induced direct CDC reaction of alcohols with heteroarenes in aqueous media without external photocatalyst (Scheme 1). The reaction is initiated by persulfate salts and does not require the use of stoichiometric acid for activation of the heteroarene.

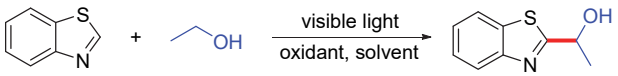


Scheme 1 CDC reaction of alcohols with heteroarenes

2 Results and discussion

Initially, benzothiazole (**1a**) and ethanol (**2a**) were selected to test the hydroxyalkylation reaction irradiated by 10 W purple LEDs under an argon atmosphere at room temperature. Gratifyingly, 32% yield of **3aa** was obtained in the presence of DTBP as oxidant (Table 1, Entry 1). Then, various oxidants were screened, and the results clearly showed that $(NH_4)_2S_2O_8$ is the best choice and the yield can reach 85% (Entries 2～7). Decreasing the amount of $(NH_4)_2S_2O_8$ from 3 equiv. to 1 equiv. resulted in lower yield (Entries 8, 9). Notably, the formation of desired product totally suppressed in the absence of oxidant, illustrating the crucial role of $(NH_4)_2S_2O_8$ (Entry 10). Then, a series of reactions were performed in other solvent or solvent-free conditions, and none gave any advantage (Entries 11～13). It is noteworthy that upon treatment of this reaction with other light sources such as white, blue and green LEDs irradiation resulted in a decline of yields (Entries 14～16). It is worth mentioning that the reaction can also be carried out in the sunlight, although the yield is low (Entry 17). In fact, light irradiation plays a critical role in the reaction, and the reaction in the dark failed to take place at room temperature (Entry 18). Furthermore, when the reaction was conducted in air, the desired product was obtained in 54%

Table 1 Optimization of the reaction conditions[a]



1a **2a** **3aa**

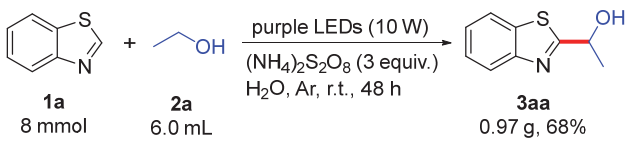
Entry	Oxidant	Light source	Solvent	Yield[b]/%
1	DTBP	Purple LEDs	H_2O	32
2	TBHP	Purple LEDs	H_2O	Trace
3	H_2O_2	Purple LEDs	H_2O	12
4	DDQ	Purple LEDs	H_2O	Trace
5	TBPB	Purple LEDs	H_2O	53
6	$K_2S_2O_8$	Purple LEDs	H_2O	43
7	$(NH_4)_2S_2O_8$	Purple LEDs	H_2O	85
8[c]	$(NH_4)_2S_2O_8$	Purple LEDs	H_2O	61
9[d]	$(NH_4)_2S_2O_8$	Purple LEDs	H_2O	38
10	—	Purple LEDs	H_2O	nr
11	$(NH_4)_2S_2O_8$	Purple LEDs	DMSO	62
12	$(NH_4)_2S_2O_8$	Purple LEDs	CH_3CN	46
13	$(NH_4)_2S_2O_8$	Purple LEDs	—	36
14	$(NH_4)_2S_2O_8$	White LEDs	H_2O	48
15	$(NH_4)_2S_2O_8$	Blue LEDs	H_2O	72
16	$(NH_4)_2S_2O_8$	Green LEDs	H_2O	28
17[e]	$(NH_4)_2S_2O_8$	Sunlight	H_2O	34
18	$(NH_4)_2S_2O_8$	Darkness	H_2O	nr
19[f]	$(NH_4)_2S_2O_8$	Purple LEDs	H_2O	54
20[g]	$(NH_4)_2S_2O_8$	Purple LEDs	H_2O	51
21[h]	$(NH_4)_2S_2O_8$	Purple LEDs	H_2O	93

[a] Reaction conditions: **1a** (0.2 mmol), **2a** (1.0 mL), oxidant (3.0 equiv.), solvent (1.0 mL) at room temperature under light irradiation (10 W) in argon atmosphere for 24 h. [b] Isolated yield. [c] $(NH_4)_2S_2O_8$ (2.0 equiv.). [d] $(NH_4)_2S_2O_8$ (1.0 equiv.). [e] For 10 h. [f] Air atmosphere. [g] For 12 h. [h] For 36 h. nr=no reaction.

yield (Entry 19), which indicated that oxygen molecule in the air may obtund this transformation. The yield decreased significantly when the reaction time was reduced from 24 h to 12 h, while the yield increased by 8% when the reaction time was extended to 36 h (Entries 20 and 21). Thus, by surveying various oxidants, solvents, reaction time and light source, the optimal reaction conditions were set to be 10 W purple LEDs irradiation in the presence of 3 equiv. of $(NH_4)_2S_2O_8$ in water under the atmosphere of argon.

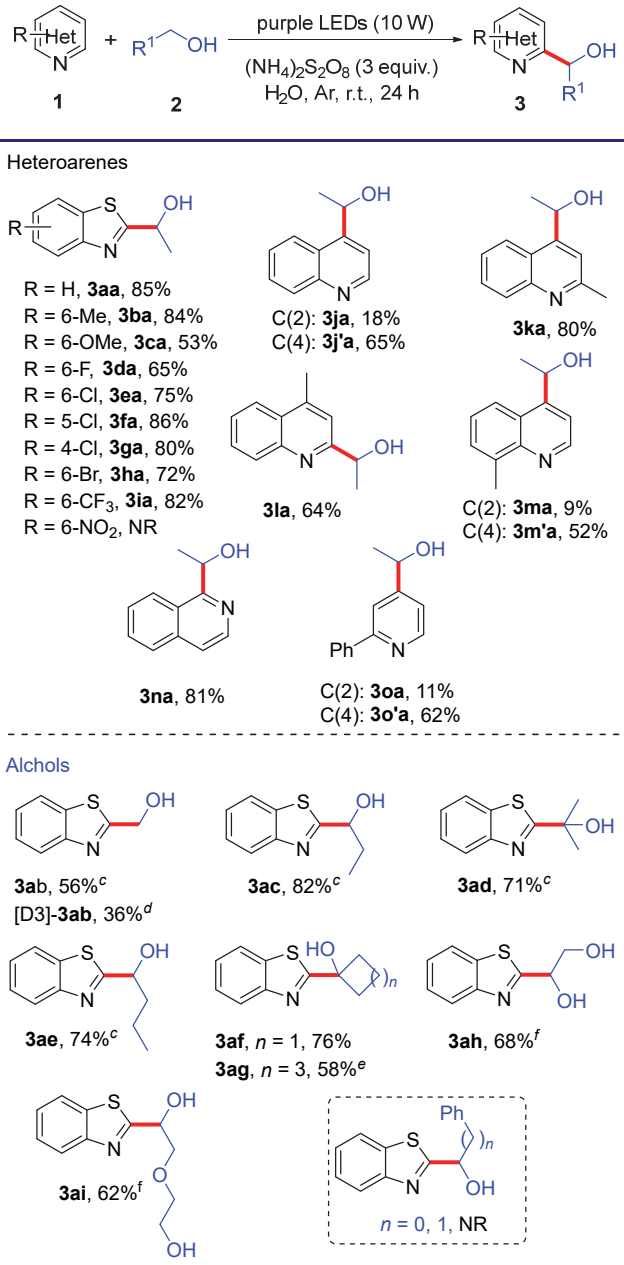
With the optimized reaction conditions in hand, the scope and generality of the heteroarene component in the direct cross-dehydrogenative coupling with ethanol (**2a**) were investigated (Table 2). Good yields (53%～86%) were obtained for benzothiazoles with both electron-donating and electron-withdrawing substituents on the C(6) of aryl ring (**3aa**～**3ia**). This protocol was compatible with a wide range of functional groups, such as methyl, methoxy, fluoro, chloro, bromo and trifluoromethyl groups. However, 6-NO_2-benzothiazole failed to produce the desired product. Benzothiazole with chloro group at different position afforded desired products with good yields. Delightedly, various other nitrogen-containing heterocycles such as quinoline, isoquinoline and 2-phenylpyridine worked well with ethanol (62% ～ 83% yields) (**3ja** ～ **3oa**). Since quinoline and 2-phenylpyridine have two active reaction sites, they all form multiple products (**3ja**, **3j'a**, **3ma**, **3m'a** and **3oa**, **3o'a**). Importantly, no product was afforded while naphthalene was employed, indicating that the presence of the N atom of heteroarenes is crucial for the direct cross-dehydrogenative coupling process. Next, the substrate scope of alcohols with benzothiazole was studied (Table 2). As expected, a series of aliphatic alcohols, such as methanol, ethanol, propanol, 2-propanol, butanol, cyclobutanol and cyclohexanol proceeded smoothly to give the corresponding coupling products (**3ab**～**3ag**) in moderate to good yields. Notably, the reaction of benzothiazole with ethylene glycol also provided the corresponding product (**3ah**) in 68% yield. In addition, when diethylene glycol was used as the starting material, the corresponding coupling product (**3ai**) was obtained in 62% yield, which revealed that hydroxyl carbon was preferred to α-carbon of ether to construct the C—C bond. Unfortunately, when benzyl alcohol or phenyl ethanol was employed as a coupling partner, no desired products were observed.

Additionally, a gram-scale experiment was carried out, and moderate yield of 68% was obtained in the model reaction at 8 mmol scales (Scheme 2). The current visible light induced cross-dehydrogenative coupling reaction provides a practical and facile application in the synthesis of value-added heterocycles.



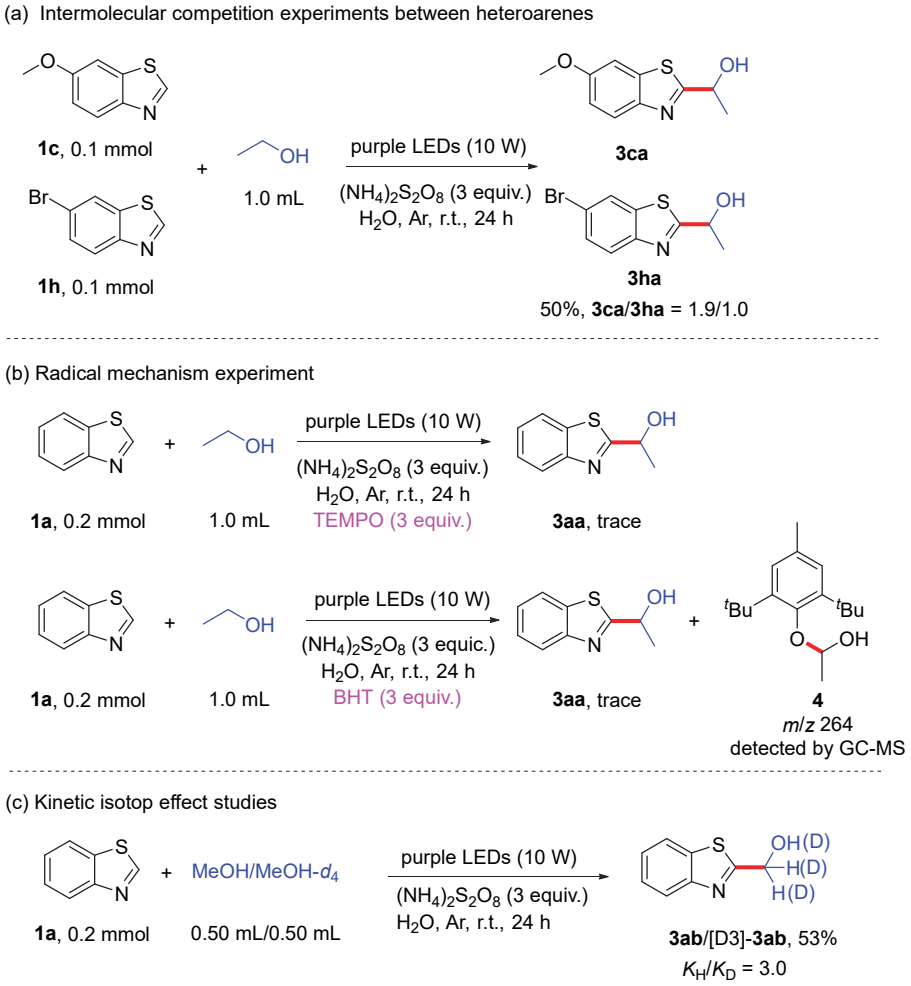
Scheme 2 Gram-scale synthesis

Table 2 Substrate scope of heteroarenes with alcohols



[a] Reaction conditions: **1** (0.2 mmol), **2** (1.0 mL), $(NH_4)_2S_2O_8$ (3 equiv.), H_2O (1.0 mL) at room temperature under purple LEDs (10 W) irradiation in argon atmosphere for 24 h, unless otherwise noted. [b] Isolated yield. [c] For 36 h. [d] Deuteration experiments. [e] For 48 h. [f] For 12 h.

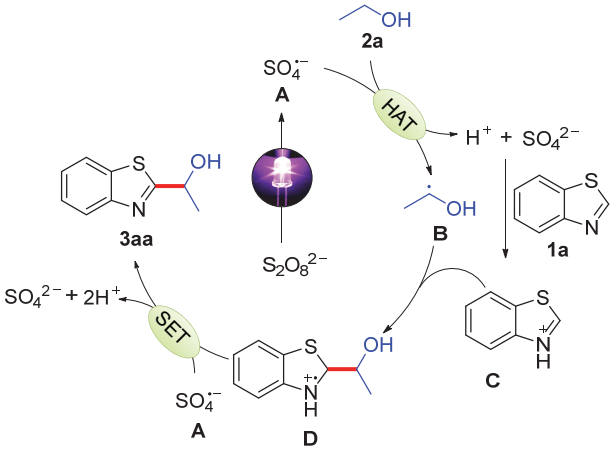
To gain insight into the reaction pathway of the visible light induced hydroxyalkylation reaction, a series of experiments were carried out (Scheme 3). First, intermolecular competition experiments between ethanol and benzothiazoles with electron-donating and electron-withdrawing group (**1c** and **1h**) were performed, and the corresponding products **3ca** and **3ha** were obtained with a ratio of 1.9/1.0 in 50% combined yield (Scheme 3, a), indicating that the electron-rich heteroarenes proved to be inherently more reactive than their electron-deficient counterparts. Second, the desired hydroxyalkylation product was completely inhibited when radical inhibitor 2,2,6,6-tetrame-



Scheme 3 Control experiments

thyl piperidine-*N*-oxide (TEMPO, 3 equiv.) or 2,6-di-*tert*-butyl-4-methylphenol (BHT, 3 equiv.) was added to the reaction system, and the adduct resulting from the sequestration of α-hydroxyl radical by BHT was detected by GC-MS, thus revealing a radical pathway might be involved (Scheme 3, b). Finally, the intermolecular competition reaction between methanol **1b** and [D4]-**1b** with benzothiazole **1a** in one vessel established a kinetic isotopic effect (KIE) of K_H/K_D was 3.0 (Scheme 3, c). The results indicated that the cleavage of α-C(sp^3)—H is the rate determining step for this protocol.

Based on the above experimental results and previous reports,[15a,16] a plausible reaction mechanism for the hydroxyalkylation reaction has been proposed in Scheme 4. Under the irradiation of purple LEDs, $(NH_4)_2S_2O_8$ was converted to sulfate radical anions ($SO_4^{\bullet-}$) (**A**). The generated $SO_4^{\bullet-}$ is capable of abstracting the α-C(sp^3)—H of alcohol to give the α-hydroxyalkyl radical (**B**). Subsequently, the protonated heteroarenes (**C**) can capture the relatively nucleophilic radical and afford the corresponding amine radical cation (**D**). Finally, further oxidation and deprotonation of this radical adduct by another sulfate radical anion would then deliver the product **3aa**.



Scheme 4 A plausible mechanism

3 Conclusions

In summary, an efficient visible light-induced approach to the direct cross-dehydrogenative coupling of α-C(sp^3)—H of alcohols with C(sp^2)—H of heteroarenes was disclosed. This methodology shows a broad scope with regard to both alcohols and heteroarene substrates, providing consistently high yields of α-hydroxyalkylated heteroarenes.

Importantly, the reaction avoids the use of expensive photocatalyst, requires no existence of TFA, operates in aqueous media at ambient temperature, is easy to set up, and is scalable to the gram level. We expect that this approach will proceed to widen an ever growing toolbox of synthetic tools for medicinal chemists.

4 Experimental section

4.1 General information

All starting materials and reagents were commercially available and used directly without further purification. All known products gave satisfactory analytical data by NMR spectra, corresponding to the reported literature values. In addition, unknown compounds were confirmed by HRMS.

Melting points were determined using X-4 micro melting point apparatus and were uncorrected. NMR spectra were recorded at room temperature on a Bruker Avance-400 at 400 MHz with tetramethylsilane (TMS) as an internal standard. Chemical shifts are given relative to TMS. High-resolution mass spectra (HRMS) were recorded on an Agilent 6200 LC/MS TOF mass spectrometer using ESI in positive mode. Thin layer chromatography (TLC) was performed using commercially available 100～400 mesh silica gel plates (GF_{254}).

4.2 Experimental method

To a 10 mL Schlenk tube were added $(NH_4)_2S_2O_8$ (3 equiv., 0.1368 g), heteroarenes (0.2 mmol), alcohols (1.0 mL) and water (equal volume to alcohols) under argon atmosphere. The sealed reaction mixture was placed 5 cm away from 10 W purple LEDs, and stirred (2～48 h) under the irradiation of purple light until the reaction was completed (monitored by TLC). The reaction mixture was diluted with saturated $NaHCO_3$ solution, and extracted with ethyl acetate (20 mL×3). The organic layer was dried over Na_2SO_4. After concentration, the resulting residue was purified by flash chromatography on silica gel to afford the product.

1-(Benzo[*d*]thiazol-2-yl)ethanol (**3aa**): Yellow solid, 30.4 mg, 85% yield. m.p. 56～57 ℃ (Lit.[9a] 55～56 ℃); TLC (petroleum ether/ethyl acetate, *V*∶*V*=2∶1) R_f=0.33; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.98～7.94 (m, 1H), 7.89～7.85 (m, 1H), 7.49～7.43 (m, 1H), 7.40～7.34 (m, 1H), 5.25 (q, *J*=6.4 Hz, 1H), 3.44 (br, 1H), 1.71 (d, *J*=6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 177.2, 152.8, 134.8, 126.1, 125.1, 122.8, 121.9, 68.5, 24.0.

1-(6-Methylbenzo[*d*]thiazol-2-yl)ethanol (**3ba**): Yellow solid, 32.4 mg, 84% yield. m.p. 104～105 ℃; TLC (petroleum ether/ethyl acetate, *V*∶*V*=2∶1) R_f=0.33; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.82 (d, *J*=8.4 Hz, 1H), 7.63 (s, 1H), 7.29～7.20 (m, 1H), 5.22 (q, *J*=6.4 Hz, 1H), 3.46 (br, 1H), 2.46 (s, 3H), 1.68 (d, *J*=6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 176.1, 150.9, 135.1, 134.9, 127.7, 122.3, 121.6, 68.4, 24.0, 21.5; HRMS (ESI) calcd for $C_{10}H_{12}NOS$ $[M+H]^+$ 194.0634, found 194.0636.

1-(6-Methoxybenzo[*d*]thiazol-2-yl)ethanol (**3ca**): Yellow solid, 22.2 mg, 53% yield. m.p. 93～94 ℃ (Lit.[9a] 91～92 ℃); TLC (petroleum ether/ethyl acetate, *V*∶*V*=2∶1) R_f=0.20; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.82 (d, *J*=8.8 Hz, 1H), 7.30 (d, *J*=2.4 Hz, 1H), 7.04 (dd, *J*=8.8, 2.4 Hz, 1H), 5.20 (q, *J*=6.8 Hz, 1H), 3.86 (s, 3H), 3.69 (br, 1H), 1.68 (d, *J*=6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 174.3, 157.6, 147.3, 136.2, 123.3, 115.5, 104.3, 68.4, 55.8, 24.0.

1-(6-Fluorobenzo[*d*]thiazol-2-yl)ethanol (**3da**): Yellow solid, 25.6 mg, 65% yield. m.p. 31～32 ℃; TLC (petroleum ether/ethyl acetate, *V*∶*V*=2∶1) R_f=0. 33; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.87 (dd, *J*=8.8, 4.8 Hz, 1H), 7.52 (dd, *J*=8.0, 2.8 Hz, 1H), 7.22～7.11 (m, 1H), 5.22 (q, *J*=6.4 Hz, 1H), 3.50 (br, 1H), 1.68 (d, *J*=6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 176.9, 160.3 (d, $J_{C\text{-}F}$=244.0 Hz), 149.5, 135.8 (d, $J_{C\text{-}F}$=10.0 Hz), 123.7 (d, $J_{C\text{-}F}$=9.0 Hz), 114.8 (d, $J_{C\text{-}F}$=25.0 Hz), 108.0 (d, $J_{C\text{-}F}$=26.0 Hz), 68.4, 23.9; HRMS (ESI) calcd for C_9H_9FNOS $[M+H]^+$ 198.0383, found 198.0383.

1-(6-Chlorobenzo[*d*]thiazol-2-yl)ethanol (**3ea**): White solid, 32.1 mg, 75% yield. m.p. 145～146 ℃; TLC (petroleum ether/ethyl acetate, *V*∶*V*=2∶1) R_f=0.35; ^{1}H NMR (400 MHz, DMSO-d_6) δ: 8.21 (d, *J*=2.0 Hz, 1H), 7.91 (d, *J*=8.8 Hz, 1H), 7.49 (dd, *J*=8.8, 2.0 Hz, 1H), 6.40 (d, *J*=2.4 Hz, 1H), 5.05 (d, *J*=5.2 Hz, 1H), 1.52 (d, *J*=6.4 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 181.3, 152.5, 136.5, 129.7, 126.9, 124.0, 122.4, 67.5, 24.2; HRMS (ESI) calcd for C_9H_9ClNOS $[M+H]^+$ 214.0088, found 214.0085.

1-(5-Chlorobenzo[*d*]thiazol-2-yl)ethanol (**3fa**): White solid, 36.8 mg, 86% yield. m.p. 111～112 ℃ (Lit.[9a] 109～110 ℃); TLC (petroleum ether/ethyl acetate, *V*∶*V*=2∶1) R_f=0.38; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.90 (d, *J*=2.0 Hz, 1H), 7.75 (d, *J*=8.4 Hz, 1H), 7.32 (dd, *J*=8.4, 2.0 Hz, 1H), 5.23 (q, *J*=6.4 Hz, 1H), 3.46 (br, 1H), 1.69 (d, *J*=6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 179.3, 153.7, 133.1, 132.2, 125.6, 122.7, 122.6, 68.5, 24.0.

1-(4-Chlorobenzo[*d*]thiazol-2-yl)ethanol (**3ga**): Colorless oil, 34.2 mg, 80% yield. TLC (petroleum ether/ethyl acetate, *V*∶*V*=2∶1) R_f=0.40; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.76 (dd, *J*=8.0, 0.8 Hz, 1H), 7.48 (dd, *J*=8.0, 0.8 Hz, 1H), 7.29 (t, *J*=8.0 Hz, 1H), 5.33 (q, *J*=6.4 Hz, 1H), 3.58 (br, 1H), 1.72 (d, *J*=6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 178.4, 150.0, 136.3, 127.6, 126.4, 125.6, 120.4, 68.7, 24.2; HRMS (ESI) calcd for C_9H_9ClNOS $[M+H]^+$ 214.0088, found 214.0087.

1-(6-Bromobenzo[*d*]thiazol-2-yl)ethanol (**3ha**): White solid, 37.2 mg, 72% yield. m.p. 135～136 ℃; TLC (petroleum ether/ethyl acetate, *V*∶*V*=2∶1) R_f=0.34; ^{1}H NMR (400 MHz, DMSO-d_6) δ: 8.34 (d, *J*=2.0 Hz, 1H), 7.86 (d, *J*=8.8 Hz, 1H), 7.61 (dd, *J*=8.8, 2.0 Hz, 1H), 6.39 (br, 1H), 5.04 (q, *J*=6.4 Hz, 1H), 1.52 (d, *J*=6.4 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 181.3, 152.7, 137.0, 129.5, 125.3, 124.4, 117.8, 67.5, 24.2; HRMS (ESI) calcd for C_9H_9BrNOS $[M+H]^+$ 257.9583, found 257.9587.

1-(6-(Trifluoromethyl)benzo[*d*]thiazol-2-yl)ethanol (**3ia**): Yellow solid, 40.5 mg, 82% yield. m.p. 63～64 ℃; TLC (petroleum ether/ethyl acetate, *V*∶*V*=2∶1) R_f=0.41; ^{1}H

NMR (400 MHz, $CDCl_3$) δ: 8.18 (s, 1H), 8.05 (d, J=8.4 Hz, 1H), 7.70 (dd, J=8.4, 1.2 Hz, 1H), 5.28 (q, J=6.8 Hz, 1H), 2.88 (br, 1H), 1.73 (d, J=6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 180.4, 155.0, 135.0, 127.3 (q, J_{C-F}=32.0 Hz), 124.1 (q, J_{C-F}=270.0 Hz), 123.2, 123.2 (q, J_{C-F}=3.0 Hz), 119.6 (q, J_{C-F}=5.0 Hz), 68.7, 24.0; HRMS (ESI) calcd for $C_{10}H_9F_3NOS$ $[M+H]^+$ 248.0351, found 248.0347.

1-(Quinolin-2-yl)ethanol (**3ja**):[11a] Colorless oil, 6.2 mg, 18% yield. TLC (petroleum ether/ethyl acetate, V∶V=2∶1) R_f=0.20; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.11 (d, J=8.4 Hz, 1H), 8.05 (d, J=8.4 Hz, 1H), 7.78 (d, J=8.0 Hz, 1H), 7.71～7.67 (m, 1H), 7.51 (t, J=7.6 Hz, 1H), 7.34 (d, J=8.4 Hz, 1H), 5.12 (br, 1H), 5.04 (q, J=6.8 Hz, 1H), 1.57 (d, J=6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 162.9, 146.4, 137.1, 129.8, 128.8, 127.6, 127.4, 126.4, 118.0, 68.9, 24.1.

1-(Quinolin-4-yl)ethanol (**3j'a**): White solid, 22.5 mg, 65% yield. m.p. 122～123 ℃ (Lit.[17a] 119～121 ℃); TLC (petroleum ether/ethyl acetate, V∶V=1∶1) R_f=0.22; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.71～8.62 (m, 1H), 8.00 (dd, J=28.4, 8.4 Hz, 2H), 7.67～7.59 (m, 1H), 7.55 (d, J=4.4 Hz, 1H), 7.52～7.45 (m, 1H), 5.62 (q, J=6.8 Hz, 1H), 3.93 (br, 1H), 1.61 (d, J=6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 152.2, 150.1, 147.7, 129.8, 129.1, 126.5, 125.4, 123.0, 116.8, 65.8, 24.7.

1-(2-Methylquinolin-4-yl)ethanol (**3ka**): Yellow oil, 29.9 mg, 80% yield. TLC (petroleum ether/ethyl acetate, V∶V=1∶1) R_f=0.22; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.91 (d, J=8.4 Hz, 1H), 7.83 (d, J=8.4 Hz, 1H), 7.58～7.49 (m, 1H), 7.44～7.30 (m, 2H), 5.54 (q, J=6.4 Hz, 1H), 4.97 (br, 1H), 2.49 (s, 3H), 1.55 (d, J=6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 158.9, 152.1, 147.4, 129.0, 128.8, 125.6, 123.6, 122.8, 117.7, 65.5, 25.0, 24.7; HRMS (ESI) calcd for $C_{12}H_{14}NO$ $[M+H]^+$ 188.1070, found 188.1067.

1-(4-Methylquinolin-2-yl)ethanol (**3la**):[17b] Yellow oil, 23.9 mg, 64% yield. TLC (petroleum ether/ethyl acetate, V∶V=12∶1) R_f=0.42; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.06 (d, J=8.4 Hz, 1H), 7.97 (dd, J=8.4, 0.4 Hz, 1H), 7.74～7.66 (m, 1H), 7.60～7.50 (m, 1H), 7.18 (s, 1H), 5.14 (br, 1H), 4.98 (q, J=6.4 Hz, 1H), 2.70 (s, 3H), 1.56 (d, J=6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 162.5, 146.1, 145.3, 129.5, 129.3, 127.5, 126.1, 123.7, 118.6, 68.7, 24.1, 18.9.

1-(8-Methylquinolin-2-yl)ethanol (**3ma**): Colorless oil, 3.4 mg, 9% yield. TLC (petroleum ether/ethyl acetate, V∶V=12∶1) R_f=0.50; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.16 (d, J=8.4 Hz, 1H), 7.68 (d, J=8.4 Hz, 1H), 7.59 (d, J=7.2 Hz, 1H), 7.47～7.43 (m, 1H), 7.34 (d, J=8.4 Hz, 1H), 5.57 (br, 1H), 5.06 (dd, J=12.8, 6.4 Hz, 1H), 2.83 (s, 3H), 1.59 (d, J=6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 161.5, 139.2, 137.7, 136.4, 130.3, 127.4, 126.3, 125.6, 117.7, 68.5, 24.1, 17.9; HRMS (ESI) calcd for $C_{12}H_{14}NO$ $[M+H]^+$ 188.1070, found 188.1067.

1-(8-Methylquinolin-4-yl)ethanol (**3m'a**): Colorless oil, 19.4 mg, 52% yield. TLC (petroleum ether/ethyl acetate, V∶V=2∶1) R_f=0.27; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.77 (d, J=4.4 Hz, 1H), 7.77 (d, J=8.4 Hz, 1H), 7.57～7.46 (m, 2H), 7.38 (dd, J=8.4, 7.2 Hz, 1H), 5.57 (q, J=6.4 Hz, 1H), 3.47 (br, 1H), 2.76 (s, 3H), 1.57 (d, J=6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 151.8, 149.2, 147.0, 137.6, 129.4, 126.2, 125.2, 120.8, 116.4, 66.0, 24.7, 18.8; HRMS (ESI) calcd for $C_{12}H_{14}NO$ $[M+H]^+$ 188.1070, found 188.1074.

1-(Isoquinolin-1-yl)ethanol (**3na**):[9b] White solid, 28.0 mg, 81% yield. m.p. 54 ～ 55 ℃; TLC (petroleum ether/ethyl acetate, V∶V=2∶1) R_f=0.35; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.40 (d, J=5.6 Hz, 1H), 8.00 (d, J=8.4 Hz, 1H), 7.81 (d, J=8.0 Hz, 1H), 7.65 (dd, J=8.0, 7.2 Hz, 1H), 7.60～7.51 (m, 2H), 5.57 (q, J=6.4 Hz, 1H), 5.14 (br, 1H), 1.58 (d, J=6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 162.2, 140.4, 136.4, 130.2, 127.5, 127.3, 124.6, 124.2, 120.5, 66.0, 25.4.

1-(6-Phenylpyridin-2-yl)ethanol (**3oa**):[11c] Colorless oil, 4.4 mg, 11% yield. TLC (petroleum ether/ethyl acetate, V∶V=2∶1) R_f=0.49; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.03 (d, J=7.2 Hz, 2H), 7.77 (t, J=7.6 Hz, 1H), 7.65 (d, J=8.0 Hz, 1H), 7.51～7.42 (m, 3H), 7.21 (d, J=8.0 Hz, 1H), 4.96 (q, J=6.4 Hz, 1H), 4.83 (br, 1H), 1.56 (d, J=6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 162.5, 155.6, 138.6, 137.8, 129.3, 128.8, 127.0, 119.0, 118.3, 68.5, 24.3.

1-(2-Phenylpyridin-4-yl)ethanol (**3o'a**):[17c] White solid, 24.7 mg, 62% yield. m.p. 73～74 ℃; TLC (petroleum ether/ethyl acetate, V∶V=2∶1) R_f=0.25; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.42 (d, J=5.2 Hz, 1H), 7.81 (dd, J=8.0, 1.6 Hz, 2H), 7.58 (s, 1H), 7.45～7.30 (m, 3H), 7.07 (dd, J=5.2, 1.2 Hz, 1H), 4.76 (q, J=6.4 Hz, 1H), 4.67 (br, 1H), 1.40 (d, J=6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 157.5, 156.5, 149.2, 139.1, 129.0, 128.7, 127.1, 119.2, 117.8, 68.6, 25.0.

Benzo[*d*]thiazol-2-ylmethanol (**3ab**): Yellow solid, 18.5 mg, 56% yield. m.p. 95～96 ℃ (Lit.[9a] 94～95 ℃); TLC (petroleum ether/ethyl acetate, V∶V=2∶1) R_f=0.24; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.95 (d, J=8.0 Hz, 1H), 7.85 (d, J=8.0 Hz, 1H), 7.50～7.41 (m, 1H), 7.40～7.32 (m, 1H), 5.07 (s, 2H), 3.89 (br, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 173.0, 152.7, 134.6, 126.2, 125.1, 122.7, 121.9, 62.4.

1-(Benzo[*d*]thiazol-2-yl)propan-1-ol (**3ac**): Yellow solid, 31.7 mg, 82% yield. m.p. 104～105 ℃ (Lit.[9a] 102～103 ℃); TLC (petroleum ether/ethyl acetate, V∶V=2∶1) R_f=0.43; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.90 (d, J=8.0 Hz, 1H), 7.79 (dd, J=8.0, 0.4 Hz, 1H), 7.45～7.36 (m, 1H), 7.35～7.27 (m, 1H), 5.02 (dd, J=7.6, 4.8 Hz, 1H), 4.85 (br, 1H), 2.12～1.80 (m, 2H), 1.01 (t, J=7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 177.5, 152.7, 134.6, 126.1, 125.0, 122.6, 121.8, 73.2, 31.1, 9.6.

2-(Benzo[*d*]thiazol-2-yl)propan-2-ol (**3ad**): Yellow solid, 27.4 mg, 71% yield. m.p. 81～82 ℃ (Lit.[9a] 81～82 ℃); TLC (petroleum ether/ethyl acetate, V∶V=2∶1) R_f=0.43; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.97 (d, J=8.0 Hz, 1H), 7.85 (d, J=8.0 Hz, 1H), 7.50～7.40 (m, 1H), 7.38～7.30 (m, 1H), 3.59 (br, 1H), 1.75 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 180.3, 153.1, 135.3, 126.0, 124.9, 122.9, 121.8, 73.6, 30.8.

1-(Benzo[*d*]thiazol-2-yl)butan-1-ol (**3ae**): Yellow solid, 30.6 mg, 74% yield. m.p. 83～84 ℃ (Lit.[9a] 82～83 ℃); TLC (petroleum ether/ethyl acetate, *V*∶*V*=4∶1) R_f=0.26; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.95 (d, *J*=8.0 Hz, 1H), 7.85 (dd, *J*=8.0, 0.4 Hz, 1H), 7.49～7.41 (m, 1H), 7.39～7.30 (m, 1H), 5.09 (dd, *J*=8.0, 4.4 Hz, 1H), 3.72 (br, 1H), 2.10～1.78 (m, 2H), 1.70～1.39 (m, 2H), 0.95 (t, *J*=7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 176.9, 152.7, 134.7, 126.1, 125.0, 122.8, 121.8, 72.1, 40.2, 18.5, 13.8.

1-(Benzo[*d*]thiazol-2-yl)cyclobutanol (**3af**): Yellow oil, 31.2 mg, 76% yield. TLC (petroleum ether/ethyl acetate, *V*∶*V*=4∶1) R_f=0.28; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.99 (d, *J*=8.0 Hz, 1H), 7.85 (d, *J*=7.6 Hz, 1H), 7.49～7.41 (m, 1H), 7.40～7.31 (m, 1H), 4.03 (br, 1H), 2.85～2.67 (m, 2H), 2.62～2.44 (m, 2H), 2.17～1.94 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 177.9, 152.9, 135.3, 126.1, 125.0, 122.9, 121.8, 37.8, 12.9; HRMS (ESI) calcd for $C_{11}H_{12}NOS$ $[M+H]^+$ 206.0634, found 206.0630.

1-(Benzo[*d*]thiazol-2-yl)cyclohexanol (**3ag**):[11b] Yellow solid, 27.0 mg, 58% yield. m.p. 100～101 ℃; TLC (petroleum ether/ethyl acetate, *V*∶*V*=8∶1) R_f=0.20; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.97 (d, *J*=8.0 Hz, 1H), 7.86 (dd, *J*=8.0, 0.4 Hz, 1H), 7.48～7.40 (m, 1H), 7.39～7.30 (m, 1H), 3.13 (br, 1H), 2.15～2.02 (m, 2H), 2.00～1.90 (m, 2H), 1.81～1.67 (m, 5H), 1.45～1.33 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 180.9, 153.2, 135.1, 126.0, 124.8, 122.9, 121.8, 74.8, 38.4, 25.1, 21.7.

1-(Benzo[*d*]thiazol-2-yl)ethane-1,2-diol (**3ah**): Yellow solid, 26.5 mg, 68% yield. m.p. 145～146 ℃ (Lit.[9a] 144～145 ℃); TLC (petroleum ether/ethyl acetate, *V*∶*V*=1∶2) R_f=0.38; ^{1}H NMR (400 MHz, DMSO-d_6) δ: 8.07 (d, *J*=8.0 Hz, 1H), 7.94 (d, *J*=8.0 Hz, 1H), 7.52～7.45 (m, 1H), 7.44～7.35 (m, 1H), 6.40 (d, *J*=5.2 Hz, 1H), 5.00 (t, *J*=6.0 Hz, 1H), 4.97～4.88 (m, 1H), 3.88～3.78 (m, 1H), 3.71～3.62 (m, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 176.8, 153.6, 134.9, 126.4, 125.1, 122.9, 122.6, 73.1, 66.3.

1-(Benzo[d]thiazol-2-yl)-2-(2-hydroxyethoxy)ethanol (**3ai**):[9a] Yellow oil, 29.6 mg, 62% yield. TLC (petroleum ether/ethyl acetate, *V*∶*V*=1∶2) R_f=0.21; ^{1}H NMR (400 MHz, DMSO-d_6) δ: 8.13～8.05 (m, 1H), 8.01～7.93 (m, 1H), 7.54～7.47 (m, 1H), 7.46～7.39 (m, 1H), 5.12 (t, *J*=5.6 Hz, 1H), 4.82 (dd, *J*=6.0, 4.0 Hz, 1H), 4.71 (d, *J*=4.8 Hz, 1H), 3.88～3.80 (m, 1H), 3.79～3.70 (m, 1H), 3.67～3.61 (m, 2H), 3.61～3.55 (m, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 173.4, 153.3, 135.0, 126.5, 125.5, 123.0, 122.8, 81.6, 72.4, 64.9, 60.8.

Supporting Information Deuteration experiments, KIE studies, ^{1}H NMR and ^{13}C NMR spectrum of prepared compounds. The Supporting Information is available free of charge via the Internet at http://sioc-journal.cn.

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