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# 碘促进 *N*,*N*-二甲基乙酰胺(DMA)与胺的转酰胺反应

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**摘要** 报道了一种新颖、简便的 *N*,*N*-二甲基乙酰胺(DMA)与胺的转酰胺反应, 用于合成乙酰胺类化合物, 产率适中至优良. 廉价的碘可以有效地促进该反应, 且反应条件简单、官能团耐受性良好、无需预官能团化, 具有一定的实用性, 可广泛用于乙酰胺药物和生物活性分子的合成.

**关键词** 碘; *N*-乙酰化; 转酰胺; *N*,*N*-二甲基乙酰胺; 乙酰胺

# Iodine-Promoted Transamidation of *N*,*N*-Dimethylacetamide (DMA) with Amines

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**Abstract** A novel and convenient transamidation of *N*,*N*-dimethylacetamide (DMA) with amines for the synthesis of acetamides with moderate to excellent yields is reported. Low-cost $I_2$ is used as efficient promoter for this transformation. Furthermore, the reaction has simple conditions, good functional group tolerance, substrates without prefunctionalization, and practicality, which can be widely used in the synthesis of acetamide drugs and biologically active molecules.

**Keywords** iodine; *N*-acylation; transamidation; *N*,*N*-dimethylacetamide; acetamide

## 1 Introduction

Amide bonds are one of the most important functional groups of living organisms and synthetic chemistry.[1] They not only play an essential role in the organization and composition of biological systems but also ubiquitously exist in various synthetic drugs, natural products, peptide compounds and fine chemicals.[2] In the pharmaceutical industry, about 60% of drug synthesis reactions belong to amide bond formation reactions, mainly including synthetic peptides and amide bond coupling.[3] Currently, a quarter of the drug molecules on the market contain amide bonds,[4] such as paracetamol,[5] apremilast,[6] trametinib[7] and lacosamide[8] (Figure 1). The amide bonds in proteins are one of their constituents, and also play a role in nitrogen storage and supply in biotic activities.[9] Therefore, it is of great value to find new protocols and reagents of amidation.

Many efforts have been devoted to the synthesis of acetamides over the past century, and significant progress has been made. Among these methods, the simplest and attractive procedure is the transamidation process of *N*,*N*-dimethylacetamide (DMA) with amines. Several catalysts have been used in transamidation reaction, such as Pd,[10] Ni,[11] Cu,[12] Zn,[13] Fe,[14] Al,[15] Ti,[16] Au,[17] Ru,[18] Zr,[19] Mn,[20] sulfated

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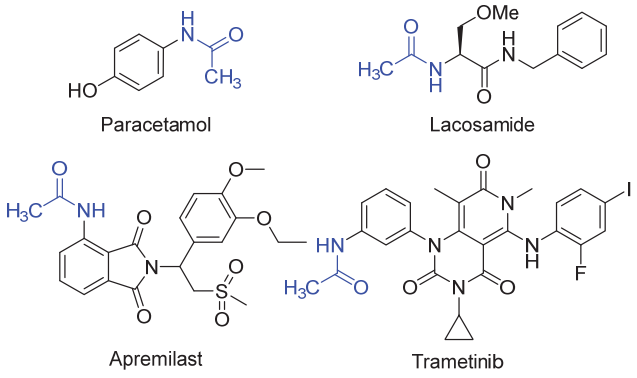


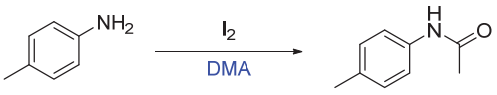

**Figure 1** Acetamides structural units in some drug molecules

tungstate,[21] $H_2SO_4SiO_2$,[22] molybdate sulfuric acid[23], hydroxylamine hydrochloride,[24] *L*-proline, and benzotriazole.[25] In 2017, Jagtap's group[26] developed a $Ni(quin)_2$ catalyst in homogeneous medium for *N*-acetylation of amines in the presence of imidazole (Scheme 1, a). In 2018, they presented a Cu-(II) catalyzed *N*-acetylation of amines in the presence of 1,2,4-triazole (Scheme 1, b).[27] In 2019, Xie's group[3b] reported a protocol using $NH_4I$ as a promoter for the synthesis of amides via *N*-acetylization of amines, however, they only obtained five amides with long reaction time and a maximum yield of 52%. Although non-metallic and metallic catalysis systems have been widely used in amides synthesis, there are still some drawbacks that limit their practical applications, including high production costs, metal-catalyst and sensitive reaction conditions. Therefore, it is imperative to develop a simple, cheap and efficient method to synthesize amide compounds. Here, we report a simple and economical protocol for *N*-acetylation of DMA with amines in the presence of $I_2$, in which $I_2$ acts as a low-cost, efficient, and environmentally friendly promoter.

## 2 Results and discussion

*p*-Toluidine (**1a**) was chosen as a model substrate. The reaction was carried out in the presence of $I_2$ (1 equiv.) at 140 ℃ for 6 h in DMA (1.5 mL) under air atmosphere, and the *N*-(*p*-tolyl)acetamide (**2a**) was successfully obtained in 31% yield as single product (Entry 1). The result encouraged us to optimize the reaction further. The reaction atmosphere was first focused on. The reaction was conducted under oxygen and nitrogen atmospheres, respectively. The results showed that a higher yield (90%) can be obtained in nitrogen than in the other atmosphere (Table 1, Entries 1～3). Subsequently, the effect of the dosage of $I_2$ on the product yield was investigated. Gratifyingly, the yield of **2a** dramatically increased to 90% with 0.5 equiv. of $I_2$ (Table 1, Entries 3～7). Reaction temperature (120, 140, 160 ℃) was also screened and the results indicated that it was a crucial factor, as the yield of **2a** decreased when the reaction was carried out at higher or lower temperature. The results suggest that 140 ℃ was beneficial for the formation of the

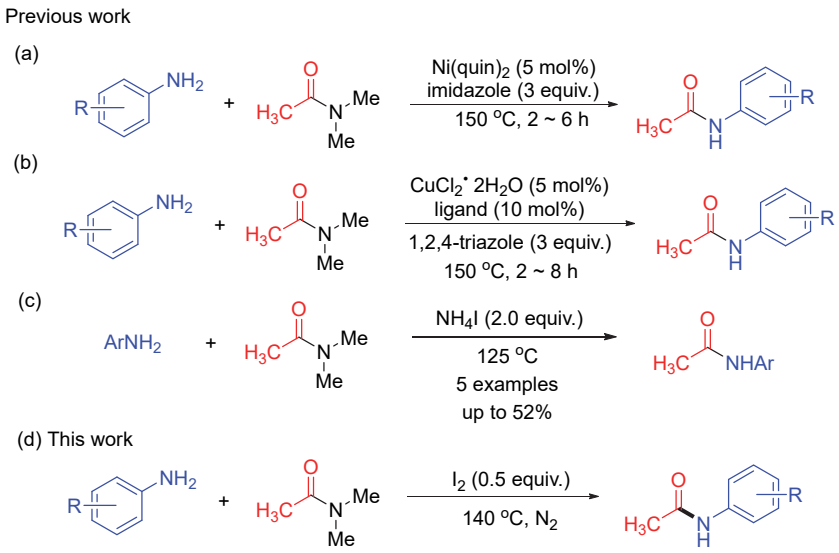
**Table 1** Optimization of the reaction conditions[a]




**1a** **2a**

| Entry | $I_2$/equiv. | Atmosphere | Temp./℃ | Yield[b]/% |
|---|---|---|---|---|
| 1 | 1.0 | Air | 140 | 31 |
| 2 | 1.0 | $O_2$ | 140 | Trace |
| 3 | 1.0 | $N_2$ | 140 | 54 |
| **4** | **0.5** | **$N_2$** | **140** | **90** |
| 5 | 1.5 | $N_2$ | 140 | 40 |
| 6 | 0.2 | $N_2$ | 140 | 36 |
| 7 | 0.1 | $N_2$ | 140 | 30 |
| 8 | 0.5 | $N_2$ | 120 | 61 |
| 9 | 0.5 | $N_2$ | 160 | 68 |
| 10[c] | 0.5 | $N_2$ | 120 | 24 |

[a] Reaction conditions: **1a** (0.2 mmol, 1 equiv), DMA (1.5 mL). [b] Isolated yields. [c] Imidazole (1 equiv) was added as additive.



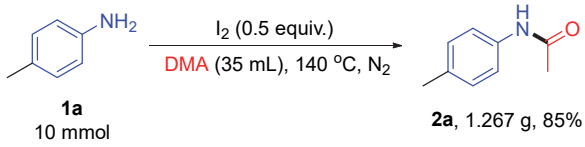

**Scheme 1** Approaches toward acetamides

desired product **2a** (90% yield, Table 1, Entries 4, 8, 9). In addition, imidazole (1 equiv.) as an additive was added in the reaction and a lower yield was given (Entry 10), showing that our method for transamidation did not need imidazole additive. After an extensive screening of various reaction parameters, the optimized reaction conditions for the transamidation of DMA with amines were obtained as follows: *p*-toluidine (0.2 mmol, 1 equiv.) and $I_2$ (0.5 equiv.) in 1.5 mL of DMA at 140 ℃ under nitrogen atmosphere.

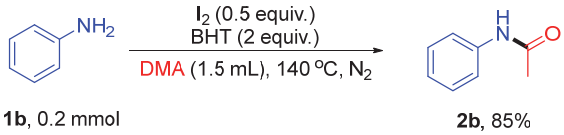
With the optimized reaction conditions in hand, the substrate scope for *N*-acylation of substituted amine derivatives was investigated (Table 2). DMA was chosen as the acetyl donor, and anilines bearing a wide range of functional groups, heterocycle amines were studied. The results demonstrated that the reaction showed highly functional group compatibility. All of them could smoothly react with DMA under the standard reaction conditions and the desired products could be given in excellent yields (Table 2, **2a**～**2p**). Concerning the electronic effects of the substituents on anilines, the anilines bearing either electron-withdrawing (*e.g.*, halogen, $CF_3$, $NO_2$) or electron-donating groups (methyl, methoxy, ethyl) received the corresponding products in good to excellent yields (Table 2, **2a**～**2n**). Moreover, 3-fluoro-4-methylaniline (**1n**) and benzidine (**1o**) transformed smoothly to the desired products in 59% and 70% yields, respectively (Table 2, **2n**～**2o**). In addition, the heterocyclic amine was also compatible and the related products were afforded in 95% yield (Table 2, **2p**). To further verify the application of this reaction in drug synthesis, 4-aminophenol and DMA were carried out under the standard conditions for the synthesis of paracetamol drug. To our delight, the target product paracetamol was obtained in 68% yield (Table 2, **2q**).

To further show the practicality of this method, gram-scale reaction was performed. 10 mmol of *p*-toluidine (**1a**) was chosen under standard reaction conditions. The desired product *N*-(*p*-tolyl)acetamide (**2a**) was obtained in 85% yield without a significant loss (Scheme 2), which verified the practicality and convenience of this strategy.




**Scheme 2** Gram scale reaction

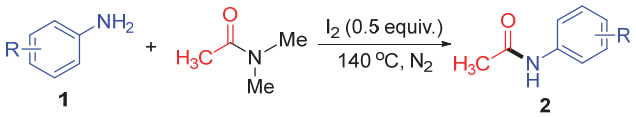
Some control experiments were carried out (Scheme 3) to study the reaction mechanism. The reaction of aniline **1b** was performed in the presence of 2.0 equiv. of butylated hydroxytoluene (BHT) under standard conditions, and no obvious inhibition was observed, which indicated that radical processes were not involved in this transformation.

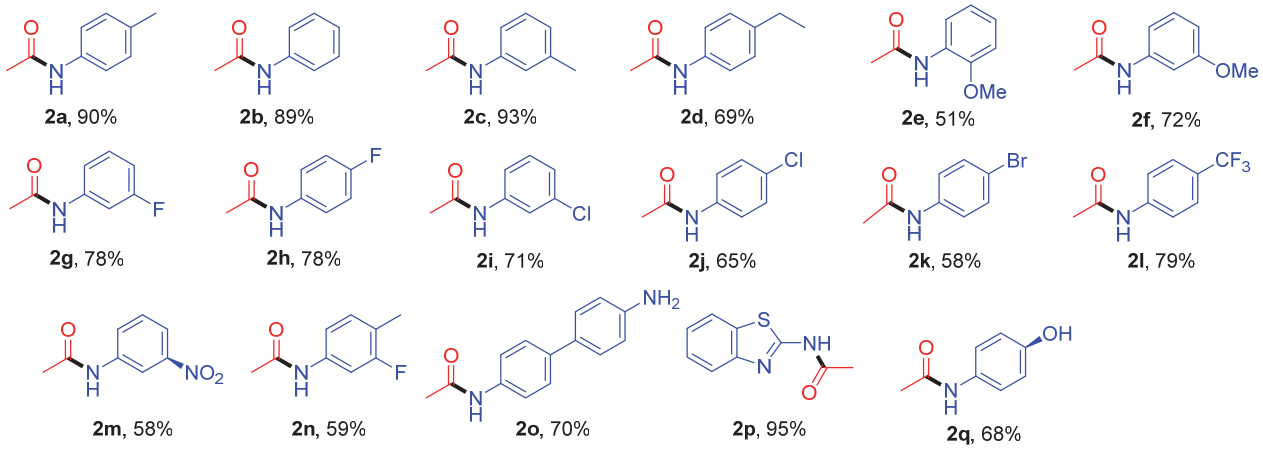



**Scheme 3** Control experiments

According to the above results and the previous literature reports,[28] a plausible reaction pathway is proposed for the synthesis of *N*-acetylation (Scheme 4). Firstly, intermediate **A** is obtained from DMA and $I_2$, followed by a nucleophilic attack of *p*-toluidine **1a** on **A** to form intermediate **B**. Then intermediate **B** transforms into **C** through tautomerization, which converts to the final product **2a** by the elimination of amine and $I^+$.

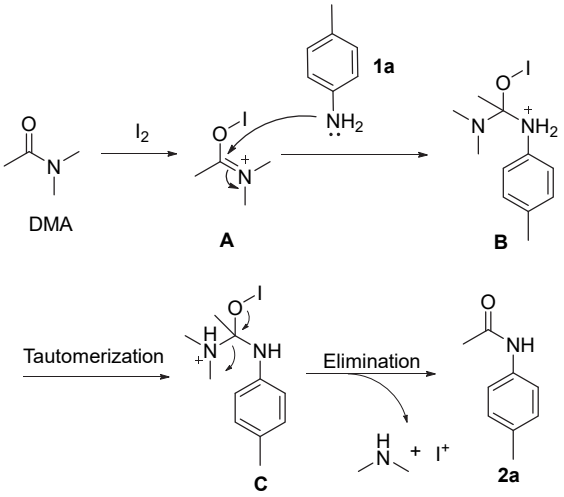
**Table 2** Substrate scope of the $I_2$-promoted transamidation of DMA with amines[a]




[a] Reaction conditions: **1** (0.2 mmol, 1 equiv.) and $I_2$ (0.5 equiv.) in 1.5 mL of DMA at 140 ℃ under nitrogen atmospheres.




**Scheme 4** Proposed mechanism

## 3 Conclusions

In summary, a simple and efficient protocol for the synthesis of acetamides via the transamidation of DMA with amines under iodine-induced promotion is developed. The approach has provided a good functional group tolerance for the synthesis of acetamides in moderate to good yields. The advantages of the present protocol are metal-free, environmental friendliness, mild reaction conditions, and the use of low-cost promoter and substrates without prefunctionalization. This protocol is very practical and promotes the development of synthesis of natural products and drugs.

## 4 Experimental section

### 4.1 General experimental details

$^1$H NMR spectra were recorded on a JNM-ECZ400S (JEOL, Japan) at 400 MHz. Chemical shifts were referenced to DMSO ($\delta$ 2.50) in DMSO as an internal standard. $^{13}$C NMR spectra were obtained at 100 MHz and were calibrated with DMSO ($\delta$ 39.50). Chemical shifts were referenced to tetramethylsilane ($\delta$ 0.00) in $CDCl_3$ as an internal standard. $^{13}$C NMR spectra were obtained at 100 MHz and were calibrated with $CDCl_3$ ($\delta$ 77.00). Products were purified by flash chromatography on 200～300 mesh silica gels. Unless otherwise noted, commercially available reagents and solvents were used without further purification.

### 4.2 General procedure for the synthesis of acetamides

A test tube was charged with amines **1** (0.2 mmol, 1 equiv.), $I_2$ (0.5 equiv.) in DMA (1.5 mL). The reaction tube was evacuated and back-filled with $N_2$ (3 times, balloon). Then the reaction mixture was stirred at 140 ℃ (oil bath temperature) under $N_2$ atmosphere. After cooling down to room temperature, the solvent was extracted with ethyl acetate and washed with brine, followed by drying with $Na_2SO_4$. After the solvent was evaporated *in vacuo*, the residues were purified by column chromatography, eluting with petroleum ether/ethyl acetate ($V$∶$V$=5∶1) to afford pure **2**.

*N*-(*p*-Tolyl)acetamide (**2a**):[27] Yellow solid, m.p. 152～154 ℃; $^1$H NMR (400 MHz, Chloroform-*d*) $\delta$: 7.46 (s, 1H), 7.37 (d, $J$=8.4 Hz, 2H), 7.11 (d, $J$=8.2 Hz, 2H), 2.31 (s, 3H), 2.15 (s, 3H); $^{13}$C NMR (100 MHz, Chloroform-*d*) $\delta$: 168.40, 135.27, 133.90, 129.41, 120.02, 24.46, 20.82.

*N*-Phenylacetamide (**2b**):[27] White solid, m.p. 109～111 ℃; $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 9.93 (s, 1H), 7.57 (dd, $J$=8.7, 1.1 Hz, 2H), 7.31～7.25 (m, 2H), 7.04～6.98 (m, 1H), 2.03 (s, 3H); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta$: 168.30, 139.36, 128.70, 122.98, 118.95, 24.04.

*N*-(*m*-Tolyl)acetamide (**2c**):[1g] Yellow solid, m.p. 62～64 ℃; $^1$H NMR (400 MHz, Chloroform-*d*) $\delta$: 7.32 (d, $J$=36.5 Hz, 3H), 7.23～7.12 (m, 1H), 6.93 (d, $J$=6.3 Hz, 1H), 2.34 (s, 3H), 2.18 (s, 3H); $^{13}$C NMR (100 MHz, Chloroform-*d*) $\delta$: 168.39, 138.88, 137.73, 128.77, 125.10, 120.52, 116.92, 24.61, 21.46.

*N*-(4-Ethylphenyl)acetamide (**2d**):[29] Yellow oil. $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 9.84 (s, 1H), 7.46 (d, $J$=8.4 Hz, 2H), 7.11 (d, $J$=8.4 Hz, 2H), 2.54 (t, $J$=7.6 Hz, 2H), 2.01 (s, 3H), 1.14 (t, $J$=7.4 Hz, 3H); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta$: 168.04, 138.31, 137.07, 127.88, 119.05, 27.60, 23.97, 15.78.

*N*-(2-Methoxyphenyl)acetamide (**2e**):[26] White solid, m.p. 128～130 ℃; $^1$H NMR (400 MHz, Chloroform-*d*) $\delta$: 8.36 (d, $J$=8.0 Hz, 1H), 7.78 (s, 1H), 7.04 (t, $J$=7.7 Hz, 1H), 6.96 (t, $J$=7.7 Hz, 1H), 6.88 (d, $J$=8.0 Hz, 1H), 3.88 (s, 3H), 2.21 (s, 3H); $^{13}$C NMR (100 MHz, Chloroform-*d*) $\delta$: 168.22, 147.56, 127.62, 123.56, 121.06, 119.69, 109.77, 55.59, 24.95.

*N*-(3-Methoxyphenyl)acetamide (**2f**):[30] White solid, m.p. 102～104 ℃; $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 9.92 (s, 1H), 7.28 (t, $J$=2.2 Hz, 1H), 7.18 (t, $J$=8.1 Hz, 1H), 7.10 (d, $J$=7.8 Hz, 1H), 6.62～6.57 (m, 1H), 3.71 (s, 3H), 2.02 (s, 3H); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta$: 168.37, 159.49, 140.53, 129.49, 111.26, 108.31, 104.81, 54.93, 24.11.

*N*-(3-Fluorophenyl)acetamide (**2g**):[27] Yellow oil. $^1$H NMR (400 MHz, Chloroform-*d*) $\delta$: 7.47 (s, 2H), 7.31～7.22 (m, 1H), 7.14 (s, 1H), 6.92～6.64 (m, 1H), 2.19 (d, $J$=5.3 Hz, 3H); $^{13}$C NMR (100 MHz, Chloroform-*d*) $\delta$: 168.47, 162.94 (d, $J$=244 Hz), 139.28, 130.03 (d, $J$=9 Hz), 114.87, 110.97 (d, $J$=21 Hz), 107.25 (d, $J$=26 Hz), 24.62.

*N*-(4-Fluorophenyl)acetamide (**2h**):[29] White oil. $^1$H NMR (400 MHz, Chloroform-*d*) $\delta$: 7.52 (s, 1H), 7.48～7.41 (m, 2H), 7.00 (t, $J$=8.2 Hz, 2H), 2.16 (s, 3H); $^{13}$C NMR (100 MHz, Chloroform-*d*) $\delta$: 168.46, 159.33 (d, $J$=243 Hz), 133.82, 121.79 (d, $J$=8 Hz), 115.57 (d, $J$=22 Hz), 24.35.

*N*-(3-Chlorophenyl)acetamide (**2i**):[1g] Yellow oil. $^1$H NMR (400 MHz, Chloroform-*d*) $\delta$: 7.74 (s, 1H), 7.64 (d, $J$=2.1 Hz, 1H), 7.35 (d, $J$=8.2 Hz, 1H), 7.22 (t, $J$=8.0 Hz, 1H), 7.07 (d, $J$=7.9 Hz, 1H), 2.18 (s, 3H); $^{13}$C NMR (100 MHz, Chloroform-*d*) $\delta$: 168.72, 138.98, 134.52, 129.92, 124.30, 119.95, 117.79, 24.52.

*N*-(4-Chlorophenyl)acetamide (**2j**):[1g] White oil. $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 10.08 (s, 1H), 7.60 (d, $J$=

8.8 Hz, 2H), 7.33 (d, *J*=8.9 Hz, 2H), 2.04 (s, 3H); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta$: 168.48, 138.30, 128.61, 126.49, 120.46, 24.02.

*N*-(4-Bromophenyl)acetamide (**2k**):[27] Yellow solid, m.p. 164～166 ℃; $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 10.07 (s, 1H), 7.55 (d, *J*=8.0 Hz, 2H), 7.46 (d, *J*=8.3 Hz, 2H), 2.03 (s, 3H); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta$: 168.52, 138.71, 131.52, 120.85, 114.50, 24.06.

*N*-(4-(Trifluoromethyl)phenyl)acetamide (**2l**):[29] White oil. $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 10.31 (s, 1H), 7.78 (d, *J*=8.5 Hz, 2H), 7.65 (d, *J*=8.5 Hz, 2H), 2.08 (s, 3H); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta$: 169.05, 142.88, 126.08 (q, *J*=4 Hz), 124.45 (q, *J*=269 Hz), 122.99 (q, *J*=32 Hz), 118.76, 24.16.

*N*-(3-Nitrophenyl)acetamide (**2m**):[27] Yellow solid, m.p. 156～158 ℃; $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 10.44 (s, 1H), 8.62 (s, 1H), 7.91～7.85 (m, 2H), 7.59 (t, *J*=8.2 Hz, 1H), 2.09 (s, 3H); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta$: 169.14, 147.96, 140.42, 130.19, 124.88, 117.58, 112.97, 24.09.

*N*-(3-Fluoro-4-methylphenyl)acetamide (**2n**):[31] Red solid, m.p. 112～114 ℃; $^1$H NMR (400 MHz, Chloroform-*d*) $\delta$: 7.60～7.30 (m, 2H), 7.15～6.97 (m, 2H), 2.20 (d, *J*=22.8 Hz, 6H); $^{13}$C NMR (100 MHz, Chloroform-*d*) $\delta$: 168.38, 161.04 (d, *J*=242 Hz), 136.83, 131.25 (d, *J*=6 Hz), 120.48 (d, *J*=17 Hz), 114.87, 107.20 (d, *J*=27 Hz), 24.52, 14.07.

*N*-(4'-Amino-[1,1'-biphenyl]-4-yl)acetamide (**2o**):[32] Yellow solid, m.p. 200～202 ℃; $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 9.93 (s, 1H), 7.57 (d, *J*=8.7 Hz, 2H), 7.45 (d, *J*=8.7 Hz, 2H), 7.32 (d, *J*=8.6 Hz, 2H), 6.63 (d, *J*=8.5 Hz, 2H), 5.32 (s, 2H), 2.04 (s, 3H); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta$: 168.12, 147.67, 137.31, 135.41, 127.40, 126.77, 125.48, 119.32, 114.41, 24.03.

*N*-(Benzo[d]thiazol-2-yl)acetamide (**2p**):[33] White solid. m.p. 188～190 ℃; $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 12.36 (s, 1H), 7.95 (d, *J*=7.2 Hz, 1H), 7.73 (d, *J*=7.9 Hz, 1H), 7.44～7.38 (m, 1H), 7.31～7.26 (m, 1H), 2.20 (s, 3H); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta$: 169.46, 157.98, 148.55, 131.44, 126.09, 123.47, 121.71, 120.51, 22.81.

*N*-(4-Hydroxyphenyl)acetamide (**2q**):[28a] White solid, m.p. 168～170 ℃;$^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 9.66 (s, 1H), 9.15 (s, 1H), 7.33 (d, *J*=8.9 Hz, 2H), 6.66 (d, *J*=8.8 Hz, 2H), 1.97 (s, 3H); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta$: 167.54, 153.13, 131.06, 120.79, 115.01, 23.78.

**Supporting Information** $^1$H NMR and $^{13}$C NMR spectra of all products. The Supporting Information is available free of charge via the Internet at http://sioc-journal.cn.

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