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$I_2/CuCl_2$-Promoted One-Pot Three-Component Synthesis of Aliphatic or Aromatic Substituted 1,2,3-Thiadiazoles

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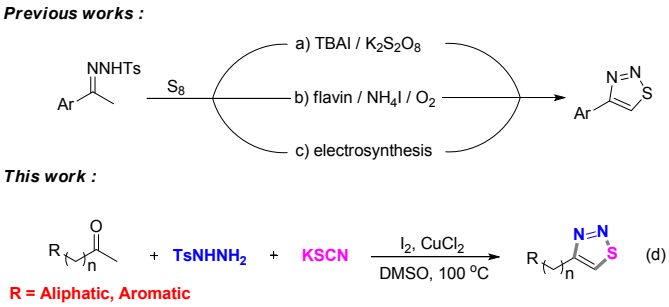
An efficient $I_2/CuCl_2$-promoted one-pot three-component strategy for the construction of 1,2,3-thiadiazoles from aliphatic- or aromatic-substituted methyl ketones, *p*-toluenesulfonyl hydrazide, and potassium thiocyanate has been developed. Simple and commercially available starting materials, a broad substrate scope, and excellent functional group tolerability make this strategy practical for applications. Furthermore, 1,2,3-thiadiazoles synthesis was realized by using potassium thiocyanate as an odorless sulfur source.

1,2,3-Thiadiazoles are important *N*,*S*-heterocyclic moiety, which are ubiquitous in natural products and pharmaceutical molecules.[1] They have widespread applications in medicines and pesticides owing to their various biological activities, including antiviral[2], anticancer[3], and antifungal[4] activities. 1,2,3-Thiadiazoles can also serve as versatile intermediates for the synthesis of various organic compounds.[5]

Owing to their significance, much effort has been made to construct 1,2,3-thiadiazole skeletons. Conventional synthetic protocols, including the Hurd–Mori synthesis[6], Wolff synthesis[7], and Pechmann synthesis,[8] among others[9], have been reported. Nevertheless, these reported protocols generally require the use of highly reactive reagents or pre-functionalized substrates, reaction safety should be considered. Pleasingly, several elegant methodologies using preprepared *N*-tosylhydrazones as substrates have emerged recently, which overcome these known deficiencies to some extent.[10–12] For example, the Cheng's group developed a TBAI-catalyzed reaction between pre-prepared *N*-tosylhydrazones and sulfur to access 1,2,3-thiadiazoles (Scheme 1a).[10] In 2017, Iida and co-workers developed an unusual flavin–iodine-catalyzed reaction to obtain 1,2,3-thiadiazoles (Scheme 1b).[11] More recently, Tang *et al*. reported an admirable metal- and oxidant-free electrochemical method to afford such structures (Scheme 1c).[12] Although hydrazones can be obtained from the dehydration condensation of hydrazines and ketones[13], significant progress in this area is still limited by the use of pre-prepared *N*-tosylhydrazones and poor substrate scope, such as aliphatic analogues being incompatible. Remarkably, many bioactive compounds with 1,2,3-thiadiazole skeleton are aliphatic products.[1g-h,2b-c,4a] Therefore, a more efficient and straightforward strategy to obtain 1,2,3-thiadiazoles that remedies these deficiencies is greatly needed and remains a significant challenge. Herein, we have disclosed an alternative three-component strategy to access 1,2,3-thiadiazoles that avoids using pre-prepared substrates. Notably, aliphatic methyl ketones react smoothly in this reaction to afford the corresponding thiadiazole products (Scheme 1d). Furthermore, this protocol is the first example of cheap and readily available potassium thiocyanate being used as an odorless sulfur source[14] for the efficient construction of 1,2,3-thiadiazole frameworks.



Scheme 1. Previous and present approaches to 1,2,3-thiadiazoles.

First, we selected acetophenone (**1a**), *p*-toluenesulfonyl hydrazide (**2**), and potassium thiocyanate (**3**) as model substrates to evaluate suitable reaction conditions. Pleasingly, in the presence of 1.6 equiv. of I_2 in DMSO at 100 °C for 1 h, target product 4-phenyl-1,2,3-thiadiazole (**4a**) was obtained in 75% yield (Table 1, entry 1). Motivated by this result, we examined the effect of reaction temperature, with 100 °C found to be optimal (Table 1, entries 1–5). Next, the iodine loading was investigated, with 2.0 equiv. of I_2 affording the optimum yield (Table 1, entries 6–9). Furthermore, the reaction yield was not improved by increasing the amount of potassium thiocyanate to 2.0 equiv., while further increasing the amount to 3.0 equiv. afforded a lower yield (Table 1, entries 10–11). Various solvents were screened to further improve the yield, with DMSO found to most suitable for this reaction (Table 1, entries 12–

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15). Additives also significantly affected the reaction results, with $CuCl_2$ found to give the best reaction promotion, affording **4a** in 86% yield (Table 1, entries 16–19). Based on this excellent result, we tested different amounts of $CuCl_2$ additive, with 0.5 equiv. found to be the optimal loading (Table 1, entries 20–21).

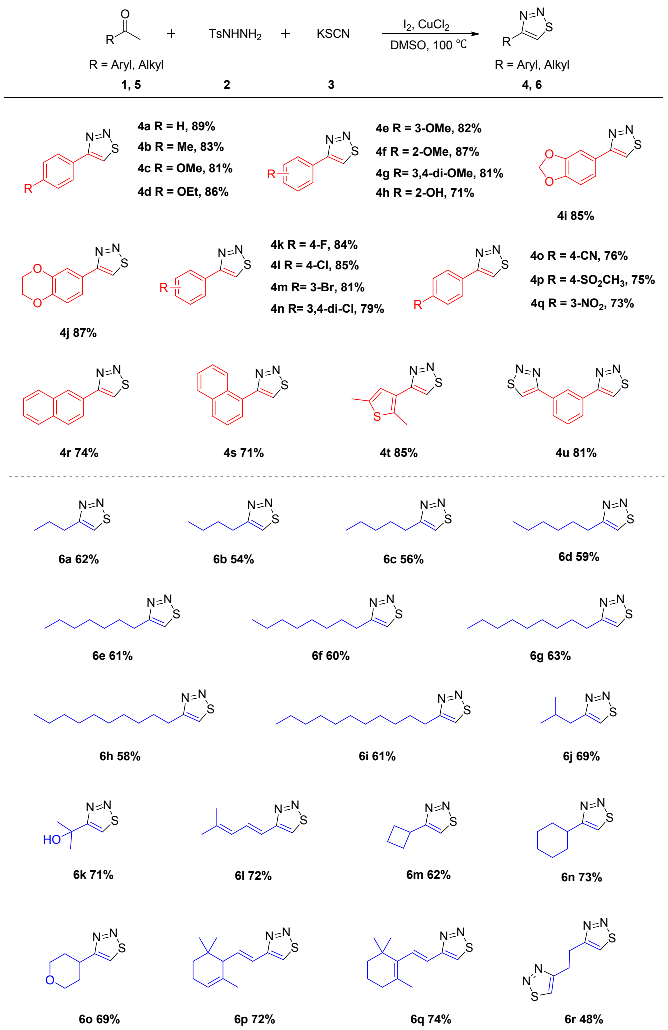
Table 1. Optimization of the Reaction Conditions[a]

1a 2 3 4a

Entry	Temp (°C)	I_2 (eq.)	KSCN (eq.)	Solvent	Additive (1.0 eq.)	Yield[b] (%)
1	100	1.6	1.0	DMSO	-	75
2	rt	1.6	1.0	DMSO	-	trace
3	60	1.6	1.0	DMSO	-	44
4	80	1.6	1.0	DMSO	-	67
5	120	1.6	1.0	DMSO	-	71
6	100	0.4	1.0	DMSO	-	14
7	100	0.8	1.0	DMSO	-	29
8	100	1.2	1.0	DMSO	-	51
9	100	2.0	1.0	DMSO	-	79
10	100	2.0	2.0	DMSO	-	68
11	100	2.0	3.0	DMSO	-	57
12	100	2.0	1.0	DIOX	-	13
13	100	2.0	1.0	DMF	-	trace
14	100	2.0	1.0	NMP	-	NR
15	100	2.0	1.0	DMAC	-	trace
16	100	2.0	1.0	DMSO	$Cu(OAc)_2 \cdot H_2O$	74
17	100	2.0	1.0	DMSO	$CuCl_2$	86
18	100	2.0	1.0	DMSO	$Cu(OTf)_2$	84
19	100	2.0	1.0	DMSO	$CuBr_2$	83
20[c]	100	2.0	1.0	DMSO	$CuCl_2$	81
21[d]	100	2.0	1.0	DMSO	$CuCl_2$	89

[a] Reaction conditions: **1a** (0.5 mmol), **2** (0.5 mmol), **3** (x mmol), additive (0.5 mmol) and I_2 were heated in 2 mL of solvent within 1 h. [b] Isolated yields. [c] $CuCl_2$ (0.1 mmol). [d] $CuCl_2$ (0.25 mmol).

The optimized reaction conditions were then applied to various aromatic methyl ketone substrates (Scheme 2). As shown in Scheme 2, aryl methyl ketones bearing electron-rich (4-Me, 4-OMe, 4-OEt, etc), electron-deficient (4-CN, 4-SO_2Me, and 3-NO_2), and halogenated (4-F, 4-Cl, 3-Br, etc) groups reacted smoothly with *p*-toluenesulfonyl hydrazide (**2**) and potassium thiocyanate (**3**) under the optimum reaction conditions to afford the corresponding products in good yields (71%–89%, **4a**–**4q**). Notably, 2-naphthyl methyl ketone and 1-naphthyl methyl ketone were also tolerated in this transformation, giving corresponding products **4r** and **4s** in 74% and 71% yields, respectively. Furthermore, this protocol exhibited commendable compatibility with heteroaryl ketones, such as 1-(2,5-dimethylthiophen-3-yl)ethanone (**1t**), with the corresponding product obtained 85% yield. Pleasingly, when 1,1'-(1,3-phenylene)diethanone (**1u**) bearing two acetyl groups was used as substrate, corresponding product **4u** was obtained in 81% yield. To examine the generality of this reaction, we also tested aliphatic methyl ketones as substrates. Surprisingly, various aliphatic methyl ketones (**5**) reacted smoothly with *p*-toluenesulfonyl hydrazide (**2**) and potassium thiocyanate (**3**) in this reaction. Accordingly, we next investigated the scope of aliphatic methyl ketone substrates (Scheme 2). To our delight, various chained aliphatic methyl ketones, including long or short, and straight or branched structures, afforded corresponding compounds **6a**–**6l** in 54%–72% yields. Furthermore, cyclic aliphatic methyl ketones also reacted smoothly to give **6m**–**6o** in 62%–73% yields. Pleasingly, α- and β-ionone also delivered corresponding 1,2,3-thiadiazoles **6p** and **6q** in 72% and 74% yields, respectively. Hexane-2,5-dione (**5r**) bearing two acetyl groups was also suitable substrate for this transformation, affording 48% product yield. To our disappointment, α-substituted aryl ketones are not compatible with this reaction (see Supporting Information).

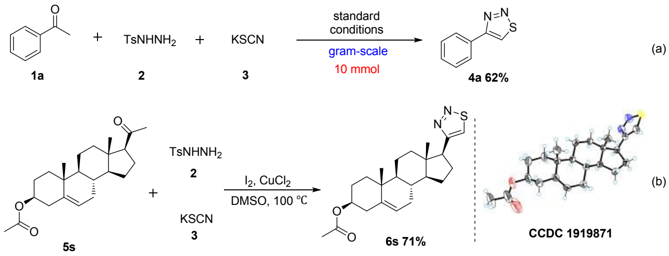


Scheme 2. Scope of methyl ketones. Reaction conditions: 1.0 mmol scale. Isolated yields.

To further demonstrate the synthetic practicability of this strategy, a 10-mmol scale synthesis of 4-phenyl-1,2,3-thiadiazole (**4a**) was conducted, with the product obtained in 62% yield (Scheme 3a). Furthermore, the reaction was also applicable to natural product pregnenolone acetate (**5s**), affording the corresponding 1,2,3-thiadiazole product **6s** in 71% yield (Scheme 3b). The structure of **6s** was unambiguously determined by X-ray crystallographic analysis (see Supporting Information).

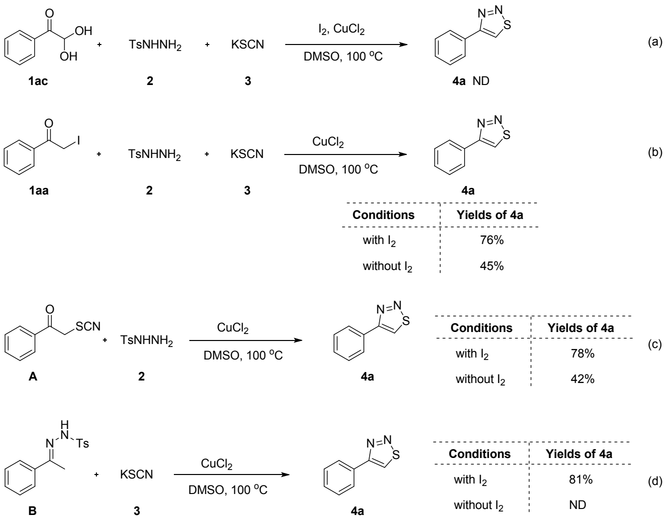
To further understand the reaction mechanism of this three-component reaction process, several control experiments were conducted. First, based on our previous work,[15] we reacted hydrated phenylglyoxal (**1ac**) as substrate with *p*-toluenesulfonyl

hydrazide (**2**) and potassium thiocyanate (**3**) under the optimized conditions for 1 h, with no target product 4-phenyl-1,2,3-thiadiazole



Scheme 3. Gram-scale Experiments and Further Conversion of Pregnenolone Acetate.

(**4a**) detected (Scheme 4a). This result demonstrated that **1ac** was not the intermediate in this reaction. Furthermore, α-iodoacetophenone (**1aa**) and 1-phenyl-2-thiocyanatoethanone (**A**) were reacted as substrates under the optimized conditions to obtain the corresponding target compound in 76% and 78% yields, respectively. However, in the absence of iodine, the yield of **4a** decreased obviously (Scheme 4b and 4c). These results showed that **1aa** and **A** were pivotal intermediates in this process, and that iodine also played a significant role. Furthermore, **4a** was also obtained in 81% yield when using acetophenone tosylhydrazone (**B**) as substrate. Meanwhile, no target product formation occurred in the absence of iodine, proving that **B** was a pivotal intermediate in the reaction and further confirming the importance of the iodination process in the reaction (Scheme 4d).



Conditions (b)	Yields of 4a
with I_2	76%
without I_2	45%

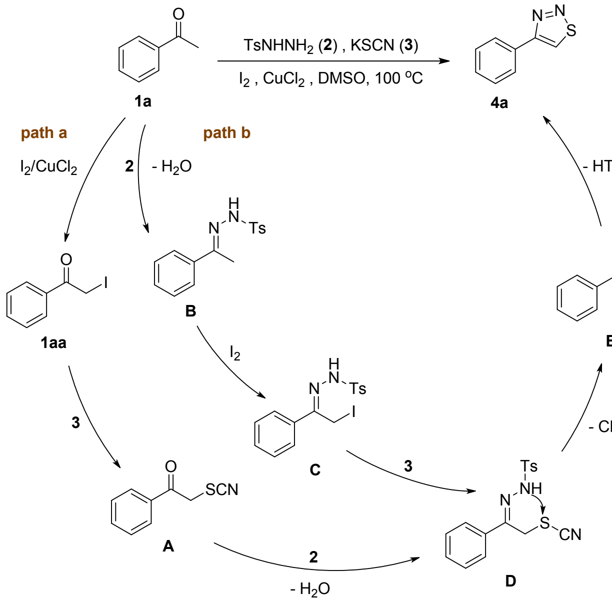
Conditions (c)	Yields of 4a
with I_2	78%
without I_2	42%

Conditions (d)	Yields of 4a
with I_2	81%
without I_2	ND

Scheme 4. Control Experiments

From the above control experiments, we proposed a possible reaction process, as shown in Scheme 5. First, acetophenone **1a** transforms into intermediate **D** via two possible reaction paths. In path a, acetophenone **1a** is first iodinated and then reacts with KSCN (**3**) to give intermediate **A**. Next, **A** and *p*-toluenesulfonyl hydrazide (**2**) undergo dehydration condensation to give intermediate **D**. In path b, **1a** first undergoes dehydration condensation with **2** to obtain intermediate **B**, followed by iodination and then react with **3** to obtain intermediate **D**. Intermediate **D** further undergoes an intramolecular nucleophilic substitution process to give intermediate **E**. Finally, HTs elimination delivers 4-phenyl-1,2,3-thiadiazole **4a**.

In conclusion, we have developed an efficient three-component strategy for preparing 1,2,3-thiadiazoles from methyl ketones, *p*-toluenesulfonyl hydrazide, and potassium thiocyanate that avoids using highly reactive or pre-prepared substrates. This strategy has excellent substrate compatibility, especially for aliphatic substrates, and enriches the substrate diversity of 1,2,3-thiadiazoles synthesis. Further studies toward other applications of this strategy are currently underway in our laboratory.



Scheme 5. Proposed mechanism

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Conflicts of interest

There are no conflicts to declare.

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