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Direct Annulation of Hydrazides to 1,3,4-Oxadiazoles via Oxidative C(CO)−C(Methyl) Bond Cleavage of Methyl Ketones

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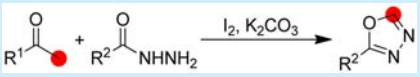
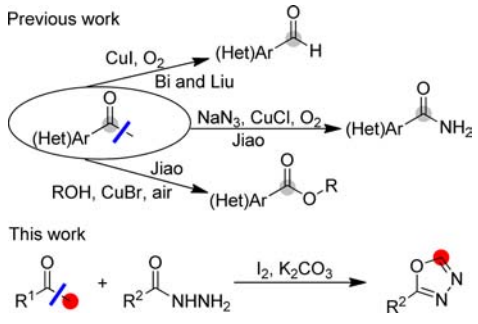
Supporting Information

ABSTRACT: A new strategy for the synthesis of 1,3,4-oxadiazoles was established through direct annulation of hydrazides with methyl ketones. It was found that the use of K_2CO_3 as a base achieves an unexpected and highly efficient C−C bond cleavage. This reaction is proposed to go through oxidative cleavage of C_{sp^3}−H bonds, followed by cyclization and deacylation.



Cleavage of the C−C bond has emerged as a challenging and attractive area which provides new modes of chemical reactivity to synthetic organic chemistry.[1] Traditionally, C−C bond cleavage can be achieved by oxidative cyclometalation of three- or four-membered-ring compounds[2] and directing a metal complex to a particular C−C bond using functional groups.[3] Nevertheless, the direct transformation through the C(CO)−C bond cleavage of ketones is still limited and attracts the continuous attention of chemists.[4] Recently, a few elegant works on oxidative C(CO)−C bond cleavage of methyl ketones have been reported (Scheme 1). The group of Bi and Liu

Scheme 1. Oxidative C(CO)−C Bond Cleavage of Methyl Ketones



described CuI-catalyzed chemoselective oxidation cleavage of methyl ketones to aldehydes, which proceeded via an arylglyoxal intermediate without overoxidization.[5] Jiao developed a Cu-catalyzed oxidative C−N and C−O bond formation through C−C bond cleavage to realize the transformations of ketones to amides[6a] and esters.[6b,c] Despite the significance of these novel reactions, the direct transformation of ketones through C(CO)−C bond cleavage is still a fascinating theme. Herein, we report our progress in the oxidative C(CO)−C bond cleavage of methyl ketones for the convenient construction of the 1,3,4-oxadiazole skeleton.

1,3,4-Oxadiazoles are important five-membered aromatic heterocycles due to their interesting properties in medicinal chemistry[7] and material sciences.[8] Consequently, much attention has been paid to the synthesis of the 1,3,4-oxadiazole skeleton, through either the dehydrative cyclization of 1,2-diacylhydrazines[9] or oxidative cyclization of *N*-acylhydrazones.[10] Recently, some pioneering approaches for their synthesis have been explored.[11] In 2014, Xu reported a Pd-catalyzed oxidative annulation for the synthesis of 2-amino-1,3,4-oxadiazoles through sequential isocyanide insertions into N−H and O−H bonds of hydrazides.[11a] Subsequently, Lindhardt and Skrydstrup et al. demonstrated a three-component approach to 1,3,4-oxadiazoles via a Pd-catalyzed carbonylative assembly of aryl bromides with hydrazides.[11b] Most recently, we disclosed Cu-catalyzed decarboxylative coupling of isatins with hydrazides for the synthesis of 2-(1,3,4-oxadiazol-2-yl)anilines.[11c] However, direct annulation of hydrazides and methyl ketones to construct 1,3,4-oxadiazoles has not been reported to date, most likely due to the inertia of ketones.

Owing to our continuing interest in the construction of heterocycles, we wished to accomplish this goal by oxidative cleavage of C_{sp^3}−H bonds of methyl ketones. In the beginning, *p*-acetylanisole (**1a**) and benzohydrazide (**2a**) were selected as model substrates. To our delight, the reaction was successful in 3.0 equiv of K_2CO_3 over 20 h using 2.5 equiv of I_2 at 100 °C in DMSO. The products were a mixture of the 2-phenyl-1,3,4-oxadiazole (**3a**, 78%) and the (4-methoxyphenyl)(5-phenyl-1,3,4-oxadiazol-2-yl)methanone (**4a**, 21%) (Table 1, entry 1). Having obtained an initial and promising result, we next focused on the optimization of the reaction conditions. After screening a series of bases including K_2CO_3, KOH, *t*-BuOK, $K_2S_2O_8$, K_3PO_4, Cs_2CO_3, $NaHCO_3$, and $NaOCH_3$ and exploring the reaction temperature and time (Supporting Information, Table S1), it was found that using 3.0 equiv of K_3PO_4 afforded **4a** in up to 89% yield (Table 1, entry 5). It was also found that an increased reaction time or temperature caused C−C bond cleavage of **4a** to form **3a**. If the reaction was

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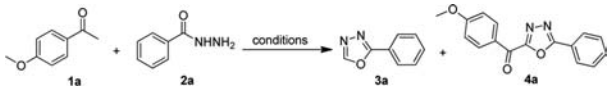
Table 1. Optimization of the Reaction Conditions[a]



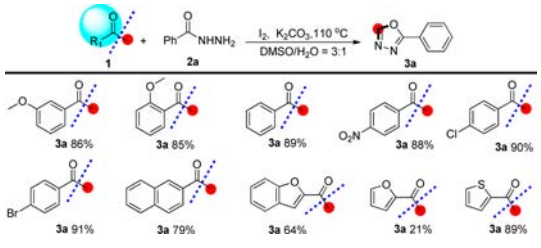
entry	I_2 (equiv)	base (equiv)	solvent	yield**3a**/**4a**[b] (%)
1	2.5	K_2CO_3 (3.0)	DMSO	78/21
2	2.5	KOH (3.0)	DMSO	0/0
3	2.5	*t*-BuOK (3.0)	DMSO	0/<10
4	2.5	$K_2S_2O_8$ (3.0)	DMSO	0/<10
5	**2.5**	**K_3PO_4 (3.0)**	**DMSO**	**<10/89**
6	2.5	Cs_2CO_3 (3.0)	DMSO	75/23
7	2.5	$NaHCO_3$ (3.0)	DMSO	0/54
8	2.5	$NaOCH_3$ (3.0)	DMSO	0/0
9	0.5	K_2CO_3 (3.0)	DMSO	0/0
10	1.0	K_2CO_3 (3.0)	DMSO	0/0
11	1.5	K_2CO_3 (3.0)	DMSO	53/32
12	2.0	K_2CO_3 (3.0)	DMSO	58/41
13	3.0	K_2CO_3 (3.0)	DMSO	15/83
14[c]	2.5	K_2CO_3 (1.0)	DMSO	0/0
15	2.5	K_2CO_3 (2.0)	DMSO	<10/86
16	2.5	K_2CO_3 (4.0)	DMSO	80/17
17	2.5	K_2CO_3 (5.0)	DMSO	85/<10
18	2.5	K_2CO_3 (6.0)	DMSO	89/0
19	2.5	K_2CO_3 (6.0)	DMSO/H_2O (1:1)	<10/0
20	2.5	K_2CO_3 (6.0)	DMSO/H_2O (2:1)	45/0
21	**2.5**	**K_2CO_3 (6.0)**	**DMSO/H_2O (3:1)**	**93/0**

[a]Reaction conditions: **1a** (0.5 mmol), **2a** (0.5 mmol), solvent (2 mL). [b]Isolated yields. [c]*C*-Acylhydrazone was isolated.

carried out using 6.0 equiv of K_2CO_3 at 110 °C for 16 h, **4a** was fully decarboxylated to form **3a** in 89% yield. DMSO/H_2O = 3/1 (v/v) turned out to be more effective solvents for the deacylation process.

With the optimized conditions in hand, the scope of the (het)aryl methyl ketones was investigated and compared (Scheme 2). It was observed that the electronic and substituent of the aryl ring of (het)aryl methyl ketones had a significant influence on the efficiency of the C−C bond cleavage. Obviously, the cleavage of various heteroaryl methyl ketones, including benzofuran, furan, and thiophene fragments, proceeded smoothly to generate the desired product **3a**.

Scheme 2. Scope of Methyl Ketones[a]

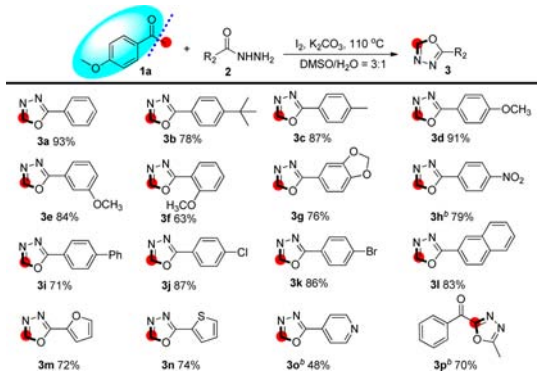


[a]Isolated yield.

However, no products was observed under the standard conditions when propiophenone was used as a substrate.

The reaction scope was next tested using substrate **1a** with different hydrazines under the optimized conditions to build diverse 1,3,4-oxadiazoles (Scheme 3). Aryl hydrazines bearing

Scheme 3. Scope of Hydrazides[a]



[a]Isolated yield. [b]K_2CO_3 (4.0 equiv).

electron-neutral (e.g., 4-H, 4-*t*-Bu, 4-Me), electron-rich (e.g., 4-OMe, 3-OMe, 2-OMe, 3,4-OCH_2O), and electron-deficient (e.g., 4-NO_2, 4-Ph) phenyl rings were converted to the corresponding products in moderate to good yields (63−93%; **3a−i**). Much to our satisfaction, the optimized conditions were mild enough to allow a broad range of halogenated (e.g., 4-Cl, 4-Br) substrates (86−87%; **3j−k**) to be reacted, which allowed for easy further functionalization. 2-Naphthyl hydrazine also provided the expected product (**3l**) in 83% yield. Meanwhile, the optimized conditions could be applied to various heteroaryl hydrazides, including furanyl, thienyl, and pyridyl hydrazines, which gave the corresponding products in moderate to good yields (48−74%; **3m−o**). In addition, an alkyl hydrazide, such as acetohydrazide, was also investigated. However, the undeacylative product (**3p**) was isolated and the desired products was not observed under the standard conditions.

Recently, α-keto-1,3,4-oxadiazoles have been identified as inhibitors of cathepsin K,[12a] human neutrophil elastase (HNE),[12b] fatty acid amide hydrolase (FAAH),[12c] and the 20S proteasome.[12d] However, to the best of our knowledge, there is no report about the effective synthesis of α-keto-1,3,4-oxadiazole derivatives.[13] Interestingly, the above C−C bond cleavage system could be readily switched to produce α-keto-1,3,4-oxadiazoles through slight modification of the reaction conditions. Several (het)aryl methyl ketones and hydrazines were subjected to the procedure, and the corresponding products were obtained in 61−91% yields (Scheme 4).

With the scope of the method established, the reaction of acetophenone-β-^{13}C (0.1 mmol) and **2a** (0.1 mmol) with I_2 (0.25 mmol) in the presence of K_2CO_3 (0.6 mmol) in DMSO-d_6 was monitored by ^{13}C NMR spectroscopy to probe the mechanism of this reaction in greater detail (Figure 1). The results of this study also revealed that phenylglyoxal (**1db′**), *C*-acyl benzoylhydrazone (**5d′**), and α-keto-1,3,4-oxadiazole (**4b′**) were important intermediates in the overall transformation.

A series of control experiments were subsequently carried out to develop a deeper understanding of the reaction mechanism (Scheme 5). The hydrated species (**1dc**) was subjected to the optimized reaction conditions and gave **3a** in

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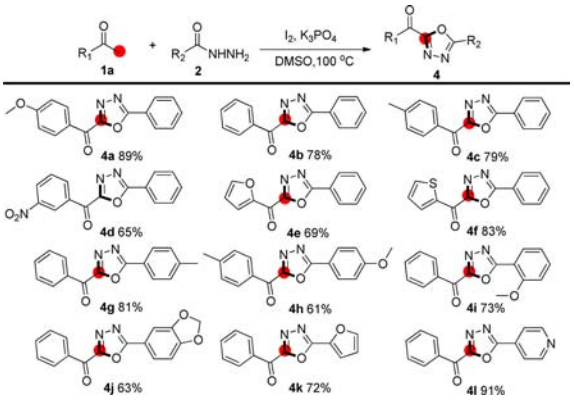
Scheme 4. Scope of Methyl Ketones and Hydrazides[a]



[a]Isolated yield.

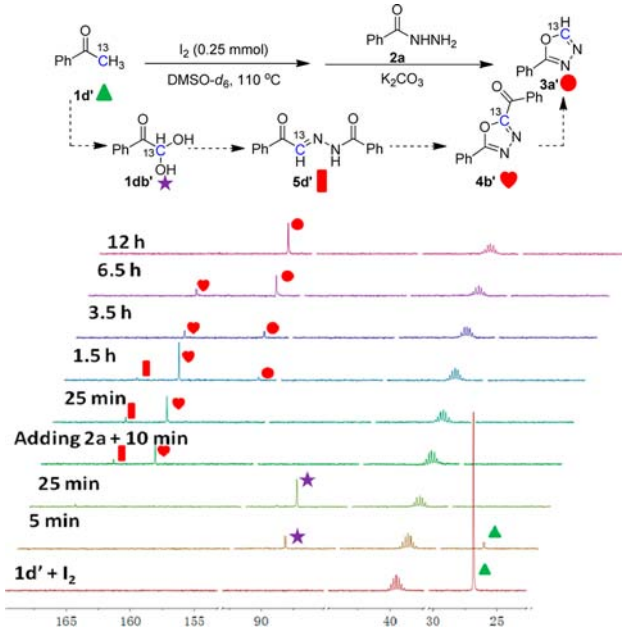
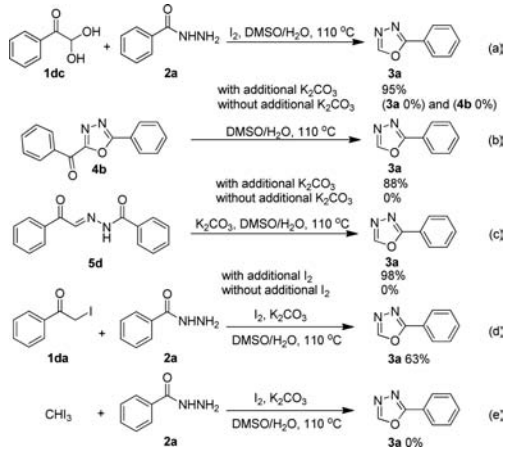


Figure 1. Progress of the reaction of **1d′** (0.1 mmol) and **2a** (0.1 mmol) with I_2 (0.25 mmol) in the presence of K_2CO_3 (0.6 mmol) at 110 °C by ^{13}C NMR (150 MHz, DMSO-d_6, 298 ± 0.5 K).

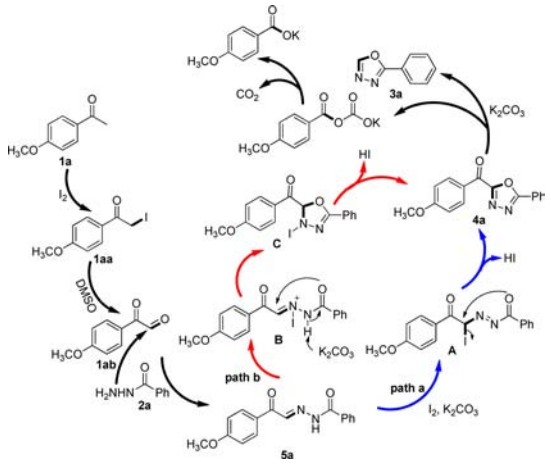
Scheme 5. Control Experiments



95% yield. This result demonstrated that **1dc** was an intermediate in the current reaction. However, both **3a** and **4b** were not observed without K_2CO_3 (Scheme 5a). When **4b** was used as a substrate in the reaction, the desired product **3a** was formed in 88% yield (Scheme 5b). These results suggested K_2CO_3 played an important role in the cyclization/deacylation process. In addition, in the absence of I_2, *C*-acylhydrazone (**5d**)[14] was not converted to the target product (Scheme 5c), indicating that I_2 might be useful for cyclization of *C*-acyl benzoylhydrazone. When the acetophenone substrate was replaced with *α*-iodo acetophenone (**1da**), which was identified as a possible precursor of *α*-ketoaldehyde (**1db**), the desired deacylation product was also obtained in 63% yield (Scheme 5d). We also checked the reaction of the acyl hydrazine with iodoform under the standard conditions in order to eliminate the possibility of annulation of hydrazides with iodoform (Scheme 5e).

On the basis of the results in the current study and previous reports,[15] a possible mechanism has been proposed using **1a** and **2a** as examples (Scheme 6). The initial reaction of I_2 with **1a** results in the formation of the *α*-iodo ketone (**1aa**), which would be converted to **1ab** by a subsequent Kornblum oxidation. The reaction of benzohydrazide (**2a**) with the aldehyde group of **1ab** would then give the *C*-acyl benzoylhydrazone (**5a**), which would react with I_2 to generate the iodide intermediate **A** (path a). This K_2CO_3-promoted oxidative iodination may be responsible for the selective cyclization of **5a**. Consequently, the product **4a** would then be smoothly formed via sequential S_N2'-type cyclization and aromatization reactions of **A**. It is possible that the K_2CO_3-promoted isomerization of **5a** was followed by intramolecular cyclization, and then aromatization (path b). Finally, *α*-keto-1,3,4-oxadiazole would undergo deacylation to provide the product **3a** in the presence of K_2CO_3. In addition, the steric effect of the base might play an important role in the C−C bond cleavage process.

Scheme 6. Possible Mechanism



In summary, we have developed a new type of C(CO)−C(methyl) bond cleavage of (het)aryl methyl ketones for the synthesis of 1,3,4-oxadiazoles via direct annulation of hydrazides. ^{13}C labeling ^{13}C NMR spectroscopy experiments demonstrate that the sp^3 carbon in (het)aryl methyl ketones was used as a one carbon synthon to perform the intermolecular annulation reactions. The generality and high selectivity of the reaction toward C(sp^3)−H bonds together with employing readily available hydrazides as the substrates

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made this method very attractive. Further investigation of the synthetic applications are ongoing in our laboratory and will be reported in due course.

■ ASSOCIATED CONTENT

Supporting Information

General experimental procedure and characterization data of the products. The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.5b01241.

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Notes

The authors declare no competing financial interest.

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