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$TiCl_4$ mediated facile synthesis of 1,3,4-oxadiazoles and 1,3,4-thiadiazoles

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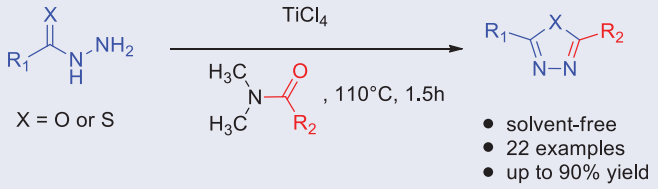
ABSTRACT

An efficient method for the synthesis of 2,5-disubstituted 1,3,4-oxadiazoles and 1,3,4-thiadiazoles has been developed. Various hydrazides or thionyl hydrazides readily react with DMA derivatives in the presence of $TiCl_4$ as a catalyst to afford the desired products. This protocol provides a simple and economical procedure that affords the target products with good yields and wide substrate scope.

GRAPHICAL ABSTRACT



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KEYWORDS
1,3,4-oxadiazoles and 1,3,4-thiadiazoles; synthesis; $TiCl_4$

Introduction

The 1,3,4-oxadiazole and 1,3,4-thiadiazole structural motifs are important heterocycles found in a variety of molecules of critical use. Bioactive compounds featuring these scaffolds, usually exhibit a wide range of pharmacological activities such as antitumor,[1] anti-inflammatory,[2] antimicrobial[3] and anti-HIV[4] in the pharmaceutical sciences. In addition, significant molecules possessing 1,3,4-oxadiazole and 1,3,4-thiadiazole as the core unit are widely applied in organic synthesis as important building blocks.[5] Inspired by the versatile applications of 1,3,4-oxadiazoles and 1,3,4-thiadiazoles, the development of simple and facile synthetic methods for these structures is of great importance.

Currently, the strategies for the synthesis of 2,5-disubstituted 1,3,4-oxadiazoles and 1,3,4-thiadiazoles generally involve the dehydration of diacylhydrazines or oxidative

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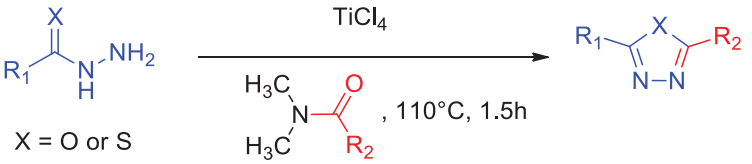


Scheme 1. Synthesis methods for 1,3,4-oxadiazoles and 1,3,4-thiadiazoles.

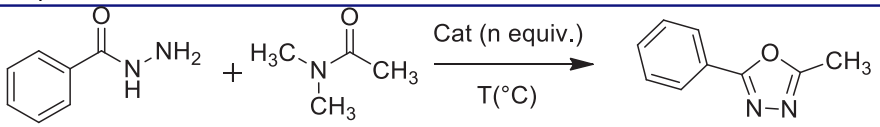
cyclization of hydrazones. Taking 1,3,4-oxadiazoles for example, coupling of hydrazines with carboxylic acids or aryl chlorides has been reported to generate diacylhydrazines, which are converted to the desired 1,3,4-oxadiazoles by cyclization in the presence of various dehydrated reagents including Brønsted acids,[6] phosphorus oxychloride,[7] polyphosphate acid (PPA),[8] Burgess's reagent,[9] H_2SO_4/SiO_2 under MW condition[10] and so on. However, there are one or more limitations to the cyclization strategies presented above, like harsh reaction conditions, extended reaction times and low yields in the most of the cases.[11] Moreover, the acidic cyclization conditions caused by the large amount of strong acids or $POCl_3$ are generally not compatible with diverse functionality. Alternatively, many improvements to the reaction have been made in recent years. Gong[12] and coworkers presented a flexible and elegant protocol for the regioselective synthesis of 1,3,4-oxadiazole and 1,3,4-thiadiazole via reagent-based cyclization of thiosemicarbazide intermediate. On the other hand, hydrazones generated from condensation of an aldehyde and hydrazines have also been proposed as a superior method to afford 1,3,4-oxadiazoles after oxidative cyclization. Typically, the oxidation is carried out using stoichiometric amounts of oxidizing agents such as $KMnO_4$,[13] $Fe(NO_3)_3$/TEMPO,[14] ceric ammonium nitrate[15] or TBHP/I_2.[16] In addition, Dabiri[17] and Bunce[18] reported an improved method to form 1,3,4-oxadiazoles from acyl hydrazides and orthoesters in the presence of $KAl(SO_4)_2 \cdot 12\,H_2O$ or NH_4Cl, respectively. Similarly, the requirement of an expensive catalyst or commercially unavailable starting material and the addition of hazardous or explosive solvents/regents also make these methods unattractive for large scale application. Thus, we became interested in the development of efficient synthesis of 1,3,4-oxadiazoles and 1,3,4-thiadiazoles utilizing a single dehydration reagent for both coupling and cyclodehydration events.

Titanium tetrachloride ($TiCl_4$) is a typical Lewis acid, and its strong affinity toward oxygenated organic compounds has been widely used in various functional group transformations. Besides, titanium tetrachloride is also a mild and powerful dehydrating agent which is usually used in the synthesis of numerous organic molecules.[19] Recently, our group successfully applied $TiCl_4$ in the convenient synthesis of furan and benzofuran derivatives.[20] Herein in this paper, we would like to describe a facile and general method for the preparation of 1,3,4-oxadiazoles and 1,3,4-thiadiazoles employing $TiCl_4$ as the catalyst, which has not been disclosed in the literature (Scheme 1).

Results and discussion

In our preliminary study, the cyclization of benzohydrazide with *N*,*N*-dimethylacetamide (DMA) was carried out as a model reaction. To our delight, when $TiCl_4$ (2 equiv.) was employed, the reaction afforded the desired product (**2a**) in good isolated yield without any other additives or solvents (Table 1, entry 2). However, replacement of

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



1a **2a**

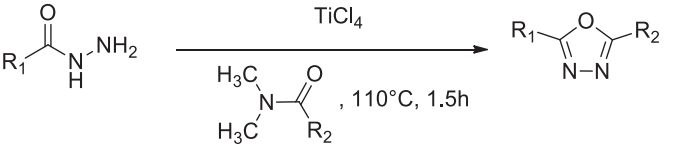
Entry	Catalyst (equiv.)	Temp (°C)	Time (h)	Yield (%)[b]
1	–	110	1.5	NR[c]
2	$TiCl_4$ (2.0)	110	1.5	85
3	H_2SO_4 (2.0)	110	1.5	24
4	Conc. HCl (2.0)	110	1.5	Trace
5	HAc (2.0)	110	1.5	Trace
6	TsOH (2.0)	110	1.5	Trace
7	$ZnCl_2$ (2.0)	110	1.5	NR
8	$FeCl_3$ (2.0)	110	1.5	Trace
9	$Cu(OAc)_2$ (2.0)	110	1.5	NR
10	$CaCl_2$ (2.0)	110	1.5	NR
11	CuBr (2.0)	110	1.5	Trace
12	$TiCl_4$ (0.5)	110	1.5	34
13	$TiCl_4$ (1.0)	110	1.5	68
14	$TiCl_4$ (3.0)	110	1.5	86
15	$TiCl_4$ (2.0)	100	1.5	63
16	$TiCl_4$ (2.0)	90	1.5	40

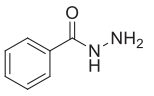
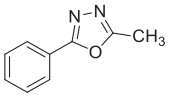
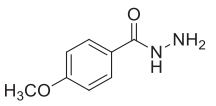
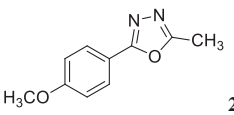
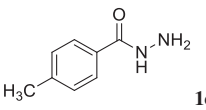
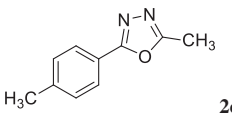
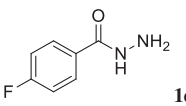
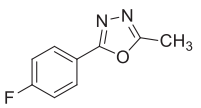
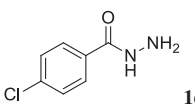
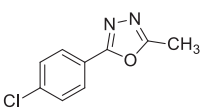
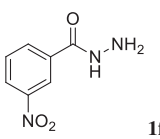
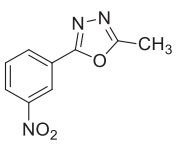
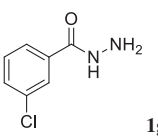
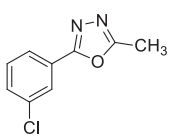
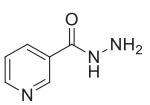
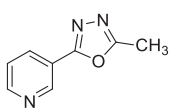
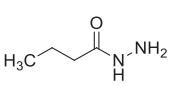
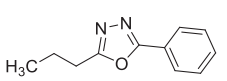
[a]Reactions were carried out with benzohydrazide (0.3 g, 2.2 mmol, 1 equiv.) and DMA (3 mL).
[b]Isolated yield.
[c]No reaction.

$TiCl_4$ with Brønsted acids such as H_2SO_4, conc. HCl, TsOH and HAc under the same condition dramatically decreased the yield (Table 1, entries 3–6), demonstrating that $TiCl_4$ is highly efficient for this reaction. Regarding the effects of different catalysts, a series of Lewis acids were extensively investigated. The screening study indicated that Lewis acids including $ZnCl_2$, $CaCl_2$ and $Cu(OAc)_2$ were ineffective in promoting this reaction, while $FeCl_3$ afforded the desired product in a lower yield along with some unknown impurities (Table 1, entries 7–11). Encouraged by this result, then the stoichiometric of the catalyst on the reaction system was explored. Carrying out the reaction with 0.5 equiv. of $TiCl_4$ afforded the product **2a** in 34% yield (entry 12). A constant increase in the yield of **2a** was observed with the increase in catalyst loading (entries 2 and 13). There was a slight increase in the yield using 3 equiv. of $TiCl_4$ (entry 14), which led us to select 2 equiv. of catalyst for this transformation. Subsequently, upon varying temperatures of the reaction between 110 and 90 °C, the conversion declined, 110 °C was proved to the optimal temperature for the reaction. Finally, the reaction was found to be efficient with 2 equiv. of catalyst in DMA at 110 °C, affording a yield of the product with 85% within 1.5 h.

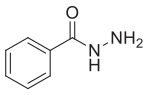
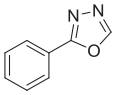
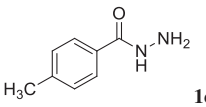
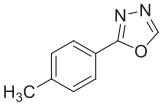
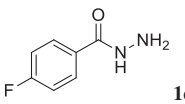
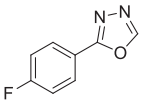
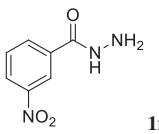
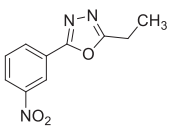
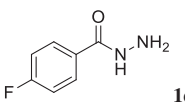
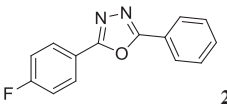
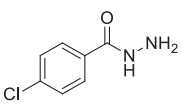
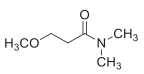
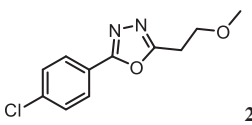
With the optimized conditions in hand, we next set out to investigate the generality of this reaction. The synthesis of 1,3,4-oxadiazoles was demonstrated to be tolerant to various substituted hydrazides, such as benzohydrazides, acetohydrazide and butyrohydrazide, as listed in Table 2. The benzohydrazides bearing electron-donating and -withdrawing groups were both proven to be compatible to this reaction. Benzohydrazides with electron-donating groups, such as CH_3 and OCH_3, generated the corresponding coupling products in better yields than those of electron-withdrawing groups (Table 2,

Table 2. Substrate scope with respect to substituted hydrazides.[a]



Entry	Substrate	Reactant/Solvent	Product	Yield
1	 **1a**	DMA	 **2a**[b]	85%
2	 **1b**	DMA	 **2b**	90%
3	 **1c**	DMA	 **2c**	84%
4	 **1d**	DMA	 **2d**	77%
5	 **1e**	DMA	 **2e**	78%
6	 **1f**	DMA	 **2f**	66%
7	 **1g**	DMA	 **2g**	73%
8	 **1h**	DMA	 **2h**	77%
9	 **1i**	DMBA	 **2i**	80%

(*continued*)

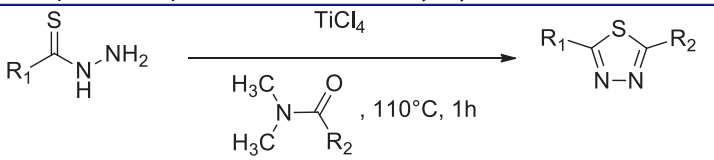
Entry	Substrate	Reactant/Solvent	Product	Yield
10	 **1a**	DMF	 **2j**	82%
11	 **1c**	DMF	 **2k**	79%
12	 **1d**	DMF	 **2l**	77%
13	 **1f**	DMPA	 **2m**	70%
14	 **1d**	DMBA	 **2n**[c]	75%
15	 **1e**		 **2o**	74%

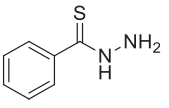
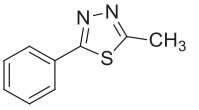
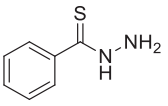
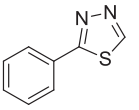
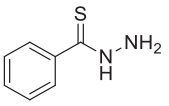
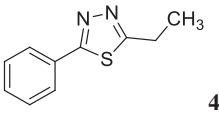
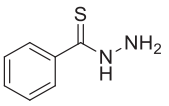
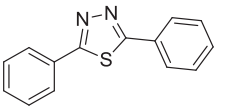
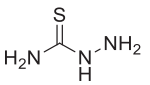
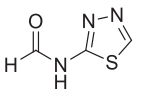
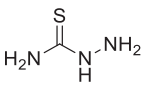
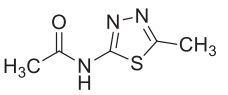
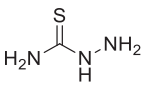
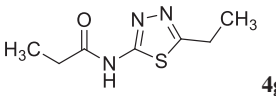
[a]Experiments were performed with substituted hydrazides (0.3 g) and DMA derivates (3 mL) at 110 °C for 1.5 h, isolated yield.
[b]2a can also be obtained from acetohydrazide and DMBA.
[c]Experiments were performed with 4-methylbenzohydrazide (5 mmol) and DMBA (3.7 g, 25 mmol) at 110 °C for 1.5 h, isolated yield.

entries **2b**, **2c** versus **2d**–**2g**), probably owing to the fact that the electron-withdrawing groups disfavor the formation of reaction intermediate. Besides, the results also indicated that the position of the substituents on the benzene moiety had little effect (Table 2, **2e** and **2g**) on the reaction outcome. In addition, butyrohydrazide was well-tolerated in this reaction and generated the product **2i** in 80% yield. It is noteworthy to mention that the present reaction conditions were also suitable for the reaction of heterocyclic substrate and the nicotinohydrazide delivered to the corresponding product **2h** in good yield.

On the other hand, in order to explore the effects of different substituted amides on this reaction, some DMA derivatives were also investigated, including DMF, *N*,*N*-dimethylpropionamide (DMPA) and *N*,*N*-dimethylbenzamide (DMBA). As shown in Table 2, it appears that this method was compatible for a wide range of DMA derivatives. In general, the treatment of **1a** with DMF and DMPA produced the corresponding products in moderate to good yields (Table 2, **2j**–**2m**). Notably, sterically hindered DMBA could also be amenable to the reaction and afforded the corresponding product **2n** in 75% yield. Moreover, for that with long-chain alkyl group amide, the reaction preceded smoothly as well resulted in **2o** with 74% yield.

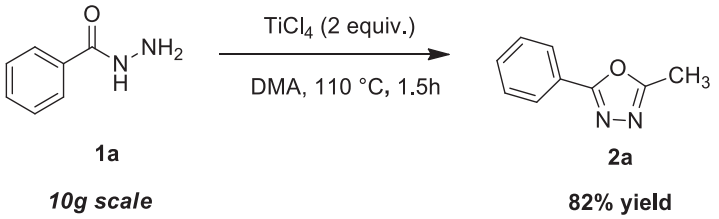
Table 3. Substrate scope with respect to substituted thionyl hydrazides.[a]



Entry	Substrate	Reactant/Solvent	Product	Yield
1	 **3a**	DMA	 **4a**	88%
2	 **3a**	DMF	 **4b**	85%
3	 **3a**	DMPA	 **4c**	80%
4	 **3a**	DMBA	 **4d**	81%
5	 **3b**	DMF	 **4e**	65%
6	 **3b**	DMA	 **4f**	72%
7	 **3b**	DMPA	 **4g**	70%

[a]Experiments were performed with (0.3 g) and DMA (3 mL) at 110 °C for 1.5 h, isolated yield.

Furthermore, the developed reaction was also successfully applied to thionyl hydrazides. The corresponding cyclization products **4a–4d** generated from benzothiohydrazide and DMA, DMF, DMPA or DMBA were successfully obtained under the standard conditions, respectively (Table 3, entries 1–4). However, for the thiosemicarbazide substrate containing a free amino group, no desired product was observed. Instead, *N*-acyl 1,3,4-thiadiazol-2-amines (**4e–4g**, Table 3, entries 5–7) were furnished in slightly lower yields. This may be attributed to the further transamidation of the primary amine after the 1,3,4-thiadiazol-2-amines were generated. Interestingly, to the best of knowledge, there are no methodologies reported for the one-pot synthesis of *N*-acyl 1,3,4-thiadiazol-2-amines.



Scheme 2. Gram scale synthesis of 2-methyl-5-phenyl-1,3,4-oxadiazole.

Considering the synthetic usefulness of this reaction, a scale-up experiment was conducted. To our delight, when the reaction was scaled up to 73 mmol (**1a**, 10 Gram scale), the desired product **2a** was isolated in 82% yield via the optimized reaction conditions (Scheme 2). This reaction was thus demonstrated to be effective and scalable.

Conclusion

In summary, we have developed a convenient approach to synthesize 2,5-disubstituted 1,3,4-oxadiazoles and 1,3,4-thiadiazoles from hydrazides and DMA derivatives utilizing $TiCl_4$ as the dehydrating and cyclization agent. This novel procedure is efficient in yielding 1,3,4-oxadiazoles and 1,3,4-thiadiazoles, and thus have potential application to the preparation of such bioactive compounds.

Experimental

Hydrazides, thionyl hydrazides and other reagents were commercially available and obtained from Adamas, tansoole or Macklin, and were used without further purification unless otherwise noted. Melting points were determined on X-4 microscopic melting point apparatus and were uncorrected. ^{1}H NMR and 13C HNMR spectra were obtained using a Bruker-600 MHz spectrometer with DMSO-d_6 or $CDCl_3$ as solutions and TMS as the internal standard. Chemical shifts are reported in ppm and coupling constants (J) in Hz. HRMS spectra were obtained with an Aglient 6210 Triple Quad LC–MS instrument. All reactions were monitored by TLC with GF254 silica gel coated plates (petroleum ether/ethyl acetate = 3:1). Flash column chromatography was carried out using 200–300 mesh silica gel. The identity of the known compounds was established by the comparison of their ^{1}H and 13C NMR peaks with the authentic values.

General procedure for the synthesis of 1,3,4-oxadiazole derivatives (2a–2o)

To a 10 mL three-necked round bottle was charged with benzoylhydrazine (0.30 g, 2.2 mmol), $TiCl_4$ (0.83 g, 4.4 mmol) and DMA (3 mL, 32.1 mmol). The resulting solution was warmed to 110 °C and stirred at this temperature for 1.5 h. When the reaction was completed, 50 mL water was added and the resulting mixture was extracted with 50 mL ethyl acetate three times. The combined organic layer was successively washed with H_2O (50 mL) and then brine (50 mL), then dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel with petroleum ether and ethyl acetate to give the title products. 2-

Methyl-5-phenyl-1,3,4-oxadiazole (**2a**) white solid. Yield, 85%. M.p. 65–67 °C; ^{1}H NMR (600 MHz, DMSO-d_6) (δ, ppm): 8.02–7.93 (m, 2H), 7.45–7.35 (m, 3H), 2.59 (s, 3H).; 13C NMR (150 MHz, DMSO-d_6) (δ, ppm): 164.4, 164.4, 132.2, 129.8, 126.7, 124.0, 11.1. HRMS (ESI): m/z $[M+Na]^+$ Calcd for $C_9H_8N_2ONa$: 183.0529; Found: 183.0524.

General procedure for the synthesis of 1,3,4-thiadiazole derivatives (4a–4g)

To a 10 mL three-necked round bottle was charged with benzothiohydrazide (0.30 g, 2.0 mmol), $TiCl_4$ (0.75 g, 4.0 mmol) and DMA (3 mL, 32.1 mmol). The resulting solution was warmed to 110 °C and stirred at this temperature for 1.5 h. When the reaction was completed, 50 mL water was added and the resulting mixture was extracted with 50 mL ethyl acetate three times. The combined organic layer was successively washed with H_2O (50 mL) and then brine (50 mL), then dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel with petroleum ether and ethyl acetate to give the title products. 2-Methyl-5-phenyl-1,3,4-thiadiazole (**4a**). White solid (88%); M.p. 100–102 °C; ^{1}H NMR (600 MHz, $CDCl_3$) (δ, ppm): 7.97–7.86 (m, 2H), 7.52–7.40 (m, 3H), 2.78 (s, 3H); 13C NMR (150 MHz, $CDCl_3$) (δ, ppm): 168.9, 164.9, 131.0, 130.1, 129.1, 127.9, 15.9. HRMS (ESI): m/z $[M+H]^+$ Calcd for $C_9H_9N_2S$: 177.0481; Found: 177.0483.

Spectral data for the synthesized compounds can be found via the 'Supplementary Content'.

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