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[3+2] Cyclization of Azidotrimethylsilane with Quinoxalin-2(1*H*)-ones to Synthesize Tetrazolo[1,5-*a*]quinoxalin-4(5*H*)-ones

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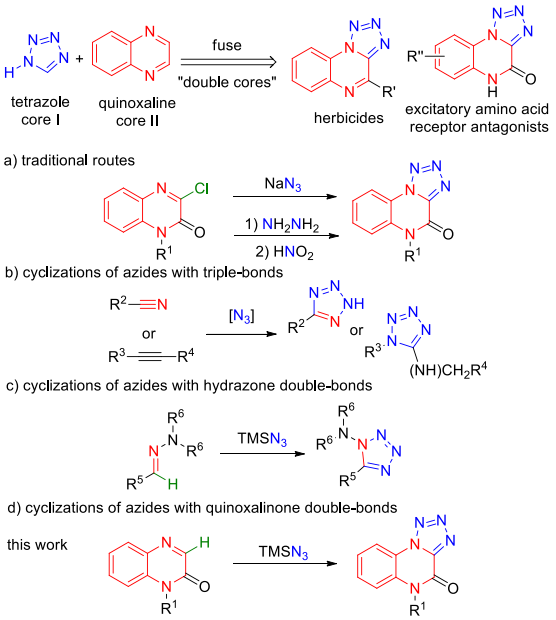
Abstract. A convenient and efficient protocol for the synthesis of thetetrazolo[1,5-*a*]quinoxalin-4(5*H*)-ones *via* copper-catalyzed [3+2] cyclization of azidotrimethylsilane with quinoxalin-2(1*H*)-ones under mild conditions has been disclosed. This practical protocol is compatible with a variety of functional groups and provides an access to functionalized tetrazolo[1,5-*a*]quinoxalin-4(5*H*)-ones from readily available and safe starting materials.

Keywords: Cyclization; Copper-catalyzed; Azidotrimethylsilane; Quinoxalin-2(1*H*)-one; Tetrazole

Multi-nitrogen-containing heterocycles, especially tetrazoles and quinoxalines derivatives, are important scaffolds featured in diverse pharmaceutically and biologically active compounds.[1] Particularly, tetrazolo[1,5-*a*]quinoxalines, the combination of the two well-known tetrazole and quinoxaline moieties into the fused bis-heterocyclic systems have revealed potential application in agrochemistry and medicinal chemistry fields.[2] However, these heterocycles have been rarely reported, due to limitations in straightforward and convenient routes to synthesize them. A traditional procedure for synthesizing tetrazolo[1,5-*a*]quinoxalin-2(1*H*)-ones is the nucleophilic substitution of 3-chloroquinoxalin-2(*1H*)-one with sodium azide.[2a, 2c] Another alternative process is based on the reaction of 3-chloroquinoxalin-2(1*H*)-one with hydrazine, followed by treatment with nitrous acid.[2b, 2d] But both the aforementioned synthetic routes suffered from the requirement of pre-functionalized starting materials, tedious operation procedures, and utilization of explosive and poisonous reagents (Scheme 1, a). Azides, as important nitrogen sources,[3] have been widely used in cyclization reaction with organic nitriles to construct tetrazoles.[4] In 2013, Echavarren group disclosed a novel gold-catalyzed transformation of alkynes to tetrazoles with azidotrimethylsilane ($TMSN_3$).[5] After that Jiao and Shi groups cooperatively reported a remarkable gold-catalyzed nitrogenation of alkynes employing $TMSN_3$ as the nitrogen source for the synthesis of 5-amino-tetrazoles (Scheme 1, b).[6] Subsequently, Zhu group demonstrated an elegant copper-catalyzed cyclization of $TMSN_3$ with aldehyde hydrazones to form 1-amino-tetrazoles (Scheme 1, c).[7] Despite significant achievements have been made, the scope of cyclization of $TMSN_3$ is still limited, and the novel and practical method for synthesis of fused tetrazole quinoxalin-2(1*H*)-ones and analogues from simple and safe starting materials under mild conditions remains rarely.



Scheme 1. Active tetrazolo[1,5-*a*]quinoxalin-4(5*H*)-ones and different strategies for the synthesis of tetrazoles.

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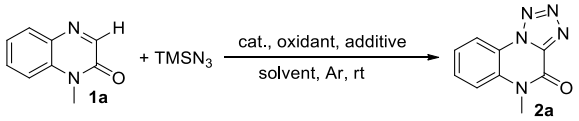
In recent years, the modification of available quinoxalin-2(1*H*)-ones based on direct C_3-H functionalization has drawn significant attention, because this strategy provides an efficient and practical access to 3-functional quinoxalin-2(1*H*)-ones. Several exciting achievements have been made in succession, such as C_3-H amination[8], phosphonation[9], alkylation[10], arylation[11], heterocyclization[12], and acylation[13] of quinoxalin-2(1*H*)-ones. Recently, we have accomplished the original C_3-H trifluoromethylation of quinoxalin-2(1*H*)-ones with CF_3SO_2Na as the trifluoromethyl source under metal-free conditions.[14] However, all of these works focused on C-H functionalization, nevertheless, and other types of reactions including annulation reaction are still scarce and challenging. Hence, we develop the first example for [3+2] cyclization of quinoxalin-2(1*H*)-ones with $TMSN_3$. This reaction employs low-cost copper salt as the catalyst and easily available potassium permanganate as the oxidant. A series of tetrazolo[1,5-*a*]quinoxalin-4(5*H*)-ones with different functional groups are obtained in moderate to excellent yields under mild conditions.

Table 1. Selected reaction conditions optimization.[a)]



Entry	catalyst	oxidant	additive	solvent	yield (%)[b)]
1	$Cu(OAc)_2$	$PhI(OAc)_2$[c)]	-	CH_3CN	N.D.[d)]
2	$Cu(OAc)_2$	$PhI(OAc)_2$[c)]	K_2CO_3	CH_3CN	N.D.
3	$Cu(OAc)_2$	$(NH_4)_2S_2O_8$[c)]	-	CH_3CN	40
4	$Cu(OAc)_2$	TBHP[c), e)]	-	CH_3CN	trace
5	$Cu(OAc)_2$	DTBP[c), f)]	-	CH_3CN	trace
6	$Cu(OAc)_2$	CAN[c), g)]	-	CH_3CN	42
7	$Cu(OAc)_2$	$KMnO_4$[c)]	-	CH_3CN	49
8	$Cu(OAc)_2$	$KMnO_4$	-	CH_3CN	55
9	$Cu(OAc)_2$	$KMnO_4$	CH_3COOH	CH_3CN	43
10	$Cu(OAc)_2$	$KMnO_4$	PivOH[h)]	CH_3CN	74
11	$Cu(OAc)_2$	$KMnO_4$	K_2CO_3	CH_3CN	33
12	$Cu(BF_4)_2$▪$6H_2O$	$KMnO_4$	PivOH	CH_3CN	64
13	Cu_2O	$KMnO_4$	PivOH	CH_3CN	78
14	**$Cu(eh)_2$**[i)]	**$KMnO_4$**	**PivOH**	**CH_3CN**	**81**
15	$Co(OAc)_2$▪$4H_2O$	$KMnO_4$	PivOH	CH_3CN	N.D.
16	$Fe(OAc)_2$	$KMnO_4$	PivOH	CH_3CN	61
17	$Cu(eh)_2$	$KMnO_4$	PivOH	EtOAc	21
18	$Cu(eh)_2$	$KMnO_4$	PivOH	THF	30
19	$Cu(eh)_2$	$KMnO_4$	PivOH	Acetone	34
20[j)]	$Cu(eh)_2$	$KMnO_4$	PivOH	CH_3CN	75

[a)] Unless specifically noted otherwise, reaction conditions: **1a** (0.4 mmol), $TMSN_3$ (3.0 equiv.), catalyst (20 mol %), oxidant (1.5 equiv.), additive (1.0 equiv.) and solvent (4 mL), stirred at room temperature under an argon atmosphere for 10 hours. [b)] Yield of isolated product. [c)] Oxidant (2.0 equiv.). [d)] N. D. = no detected. [e)] TBHP = *tert*-butyl peroxybenzoate (70% solution in H_2O). [f)] DTBP = di-*tert*-butyl peroxide. [g)] CAN = ceric ammonium nitrate. [h)] PivOH = pivalic acid. [i)] $Cu(eh)_2$ = copper bis(2-ethylhexanoate). [j)] under an air atmosphere.

Initially, we started our exploration with 1-methylquinoxalin-2(1*H*)-one (**1a**) as the model substrate, $TMSN_3$ as a nitrogen source, $Cu(OAc)_2$ as catalyst and CH_3CN as a solvent to optimize the reaction conditions. In the beginning, we chose $PhI(OAc)_2$ as an oxidant based on the optimal conditions for cyclization of $TMSN_3$ with aldehyde hydrazones (Table 1, entries 1-2).[7] Unfortunately, no target product was isolated. This result implied that the cyclization of $TMSN_3$ with quinoxalin-2(1*H*)-ones was clearly different from that with aldehyde hydrazones. Subsequently, we tested almost all oxidants. To our delight, the target product (**2a**) was obtained in 49% yield in the case of $KMnO_4$ as an oxidant (Table 1, entry 7). Other oxidants such as $(NH_4)_2S_2O_8$ and CAN were not as efficient as $KMnO_4$, and organic peroxides including TBHP and DTBP were invalid for this cyclization (Table 1, entries 3–6). Encouraged by these results, we evaluated the loading of $KMnO_4$ and found that 1.5 equiv. of $KMnO_4$ was appropriate for this transformation and the yield of **2a** was promoted from 49% to 55% (Table 1, entries 7-8). Next, various acids or bases were screened as additives, such as CH_3COOH, PivOH, and K_2CO_3. It was found that PivOH was most favorable and the target molecule (**2a**) was obtained in 74% yield (Table 1, entries 9-11). Further screening of different copper catalysts showed that $Cu(eh)_2$ was better than $Cu(OAc)_2$, improving the yield of **2a** distinctly to 81% (Table 1, entries 12-14). Other low-cost metal catalysts, such as $Co(OAc)_2$▪$4H_2O$ and $Fe(OAc)_2$ were inefficient or invalid for this reaction (Table 1, entries 15-16). Finally, the solvents screening revealed that CH_3CN was the most appropriate solvent (Table 1, entries 17-19). Overall, the reaction progressed efficiently under an argon atmosphere in the presence of **1a** (0.4 mmol), $TMSN_3$ (3.0 equiv.), $Cu(eh)_2$ (20 mol %), $KMnO_4$ (1.5 equiv.) and PivOH (1.0 equiv.) in CH_3CN (4.0 mL) at room temperature.

With the optimal reaction conditions in hand, the substrate scope was explored by using an array of quinoxalin-2(1*H*)-one derivatives (Table 2). Initial studies were focused on a series of 1-methylquinoxalin-2(1*H*)-ones. The reactions progressed smoothly and provided the desired products in moderate to good yields (**2a**-**2h**).[15] Next, various *N*-protected quinoxalin-2(1*H*)-ones were also examined. The substrates with ethyl and benzyl protecting groups are more suitable for the transformation than substrates with methyl acetate and *tert*-butyl acetate groups (**2i**-**2l**). It is notable that *N*-SEM protected quinoxalin-2(1*H*)-one was well compatible with this reaction, affording the target product in 71% yield (**2m**). The SEM protecting group was easily removed by treatment with boron trifluoride in dichloromethane.[16] We also tested the quinoxalin-2(1*H*)-ones without a protecting group and found that the target tetrazolo[1,5-*a*]quinoxalin-4(5*H*)-one was gained in moderate yield (**2n**). Considering the case that *N*-ethyl protected quinoxalin-2(1*H*)-one provided a higher yield than *N*-methyl protected quinoxalin-2(1*H*)-one, we chose a variety of 1-ethylquinoxalin-2(1*H*)-ones as substrates

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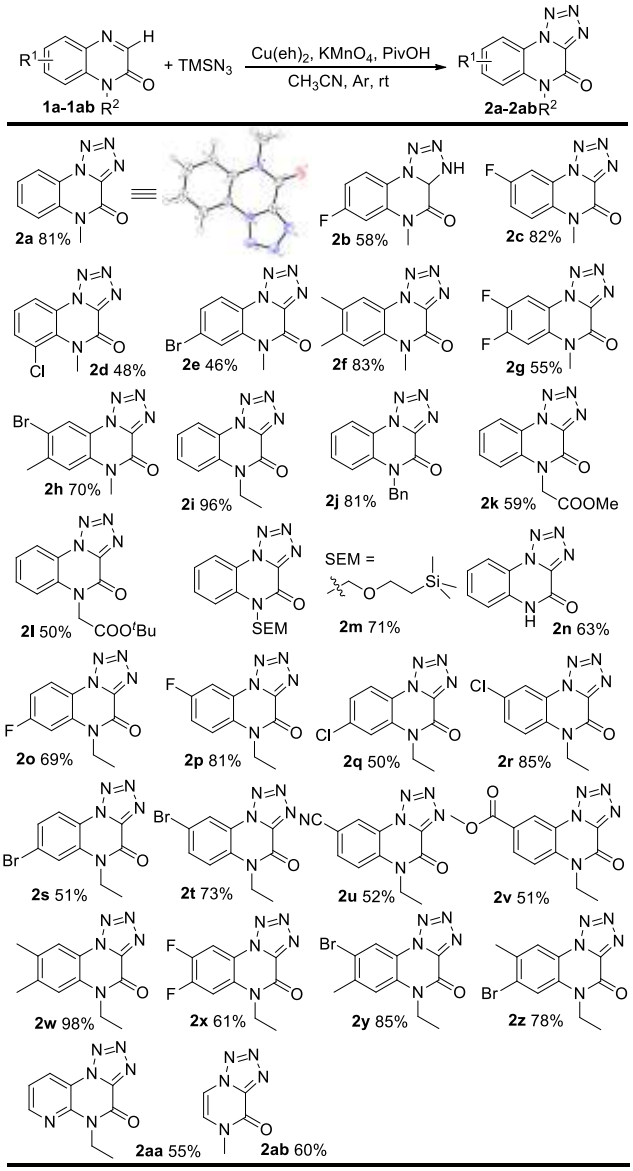
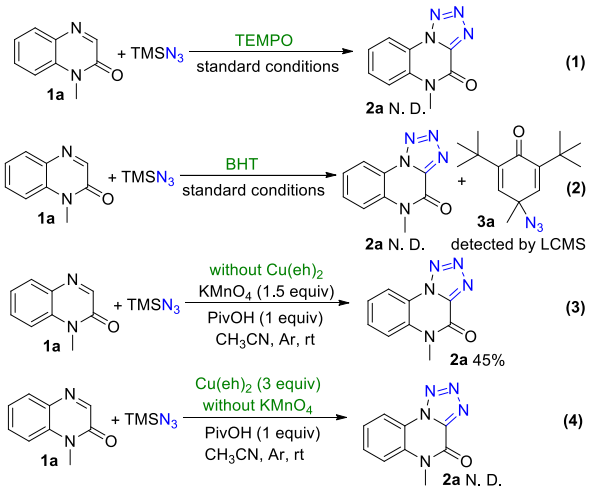
and found that the correspondingly annulate products were obtained in moderate to excellent yields (**2o-2z**). Finally, 4-ethylpyrido[2,3-*b*]pyrazin-3(4*H*)-one and 1-methylpyrazin-2(1*H*)-one were examined as a representative approximate substrates of quinoxalin-2(1*H*)-ones, and the expected products were obtained in moderate yields (**2aa-2ab**).

Table 2. Substrate scope.[a)]



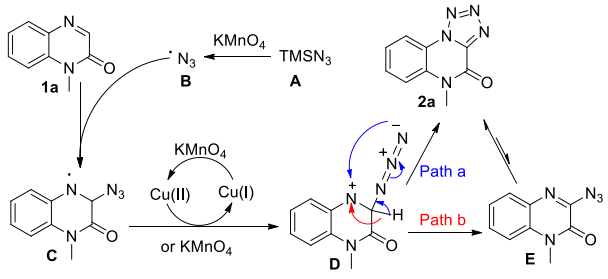
[a)] Unless specifically noted otherwise, reaction conditions: **1a** (0.4 mmol), $TMSN_3$ (3.0 equiv.), $Cu(eh)_2$ (20 mol %), $KMnO_4$ (1.5 equiv.), PivOH (1.0 equiv.) and CH_3CN (4 mL), stirred at room temperature under an argon atmosphere. [b)] Yield of isolated product.

In order to elucidate the preliminary mechanism of this transformation, several control experiments were carried out and demonstrated in Scheme 2. It was found that the reaction was completely suppressed in the presence of 3.0 equivalent of 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) as a radical inhibitor (Equation 1). Besides, when 2,6-di-*tert*-butyl-*p*-cresol (BHT) as anther radical inhibitor was added under standard conditions, the N_3-trapped compound **3a** was detected by LCMS (Equation 2). These results suggested that a radical progress might be involved in the transformation. On the other hand, the effects of a copper catalyst and $KMnO_4$ oxidant on the reaction were also investigated. The target molecule **2a** was obtained in only 45% yield under standard conditions in the absence of a copper catalyst (Equation 3). However, no desired product was detected, when 3.0 equivalents of $Cu(eh)_2$ were used under standard conditions in the absence of $KMnO_4$ (Equation 4). These results indicated that $KMnO_4$ played a crucial role in the transformation and $Cu(eh)_2$ facilitated this transformation.



Scheme 2. Control experiments.

According to the above control experiments and previous reports[7], a presumptive mechanism is proposed in Scheme 3. Initially, $TMSN_3$ reacts with $KMnO_4$ to form the azide radical **B**.[17] Subsequently, the generated **B** adds to the C=N bond of **1a** to afford the aminyl radical intermediate **C**. Next, aminyl radical **C** is oxidized by Cu(II) or $KMnO_4$ to provide the aminyl cation **D** by single-electron oxidation.[18a-c] Thereafter, aminyl cation **D** undergoes intramolecular cyclization to give the expected product **2a** (Path a). Alternatively, intermediate **D** is likely to produce 3-azido-1-methylquinoxalin-2(1*H*)-one **E** *via* β-H elimination and intermediate product **E** tends to transform into final product **2a** (Path b).[18d-e]



Scheme 3. Presumptive reaction mechanism.

To conclude, we have successfully accomplished an original method for synthesis of functionalized tetrazolo[1,5-*a*]quinoxalin-4(5*H*)-ones based on [3+2] cyclization of azidotrimethylsilane with quinoxalin-2(1*H*)-ones. This protocol utilizes cheap $Cu(eh)_2$ as a catalyst and safe $TMSN_3$ as azide source, as well as tolerates a wide range of functional groups under mild reaction conditions. Therefore, this method provides a practical approach to prepare pharmaceutically and biologically interesting tetrazolo[1,5-*a*]quinoxalin-4(5*H*)-ones in moderate to excellent yields.

Experimental Section

General procedure for cyclization of quinoxalin-2(1*H*)-ones

An oven-dried Schlenk tube was charged with quinoxalin-2(1*H*)-one derivatives (**1a-1ab**) (0.4 mmol), $KMnO_4$ (1.5 equiv., 0.6 mmol), $Cu(eh)_2$ (0.2 equiv., 0.08 mmol), PivOH (1 equiv., 0.4 mmol) and a magnetic stirring bar, and then was purged with argon for three times. Anhydrous CH_3CN (4 ml) and $TMSN_3$ (3 equiv., 1.2 mmol) were added in turn *via* respective syringes, and the mixture was stirred at room temperature in water bath under argon atmosphere until the substrate was consumed (monitored by TLC, about 10 hours). The mixture was added with CH_2Cl_2 (20 ml) and saturated aqueous Na_2CO_3 (20ml). The organic layer was isolated and the remaining aqueous phase was further extracted with CH_2Cl_2 (20 mL × 2). The combined organic phases were washed with saturated brine (20 mL). The organic layers were dried over Na_2SO_4, filtered. Solvents were evaporated under reduced pressure and the resulting residue was purified by column chromatography on neutral aluminum oxide (petroleum ether/ethyl acetate) to afford the corresponding cyclization products (**2a-2ab)**.

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[3+2] Cyclization of Azidotrimethylsilane with Quinoxalin-2(1*H*)-ones to Synthesize Tetrazolo[1,5-*a*]quinoxalin-4(5*H*)-ones

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