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Synthesis of 3-Pyrrolin-2-ones by Rhodium-Catalyzed Transannulation of 1-Sulfonyl-1,2,3-triazole with Ketene Silyl Acetal

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Supporting Information

ABSTRACT: α-Imino rhodium carbenoids generated from 1-sulfonyl 1,2,3-triazole were applied to the 3 + 2 cycloaddition with ketene silyl acetal, offering a novel and straightforward synthesis of biologically interesting compound 3-pyrrolin-2-one with broad substrate scope.

3-Pyrrolin-2-ones, closely related to pyrroles, are not only useful building blocks for the construction of pyrroles or γ-lactam derivative,[1] but also core structures of bioactive natural products and pharmaceuticals (Figure 1).[2] Several methods have been

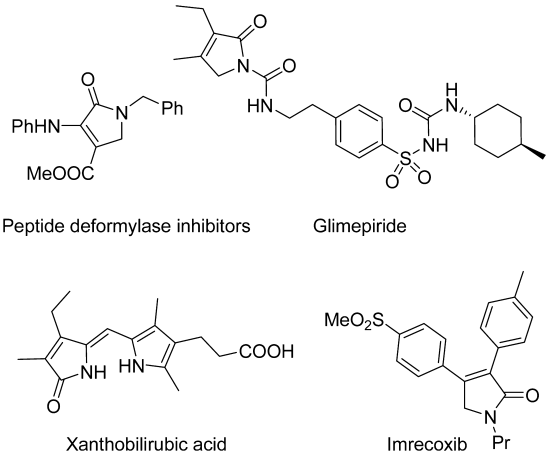

Figure 1. Natural compounds with 3-pyrrolin-2-one skeletons.

reported to access this useful structure, notably reduction of maleimide,[3] oxidation of pyrrole or pyrrole-2-carboxaldehyde,[4] reductive cyclization of β-cyanoesters,[5,2b] condensation of ketoamides, etc.[6] However, each of the above methods has its respective limitations (poor regioselectivity, not easily available starting materials, multiple steps, harsh conditions, etc.). Herein, we report an efficient and versatile process that constructs 3,4-disubstituted 3-pyrrolin-2-ones from easily accessible 1-sulfonyl-1,2,3-triazole and ketene silyl acetals.

In 2007, Gevorgyan and co-workers reported an efficient Rh-catalyzed transannulation of pyridotriazoles with alkynes and nitriles.[7] Substituted pyridotriazoles were used as precursors of rhodium carbenoids and were converted into pyrrolopyridines and imidazopyridines. In 2008, Gevorgyan and Fokin demonstrated that readily available 1-sulfonyl 1,2,3-triazoles[8] can decompose to α-imino carbenoid, which underwent formal 3 + 2 cycloaddition with nitriles to afford imidazoles.[9] Owing to the equilibrium between triazoles and their diazoimine tautomers, no slow-addition techniques or multifold triazoles were needed in the transformation.[10,11] More importantly, due to the high nucleophilic character of the nitrogen atom, α-imino carbene behaves as a 1,3-dipole equivalent to react with diverse unsaturated compounds.[12,13] In view of the prevalence of nitrogen-containing heterocycles in pharmaceuticals and biologically relevant molecules, this method is of great important since it offers new opportunities for construction of functionalized *N*-heterocycles. Very recently, the group of Lee developed an interesting approach for the synthesis of pyrroles.[12q] In the reaction, alkenyl alkyl ethers were employed to trap the α-imino carbenoid, and the products were obtained via the facile elimination of alcohols from dihydropyrroles. We have been interested in carbene chemistry for a long time,[14] and we envisioned that simply changing the position of the double bond, compound **1** can be transformed to compound **2**, which is reasonably broken down into two key synthons **A** and **B** (Scheme 1). We set up a leaving group in synthon **B** in order to reserve the carbonyl group, so the ketene silyl acetal was used as the synthetic equivalent. Although electron-rich olefins acting as the dipolarophiles in this chemistry have been reported,[12n,q,13c] the formation of the amide group in the 3 + 2 cycloaddition is unusual.

Scheme 1. Initial Hypothesis

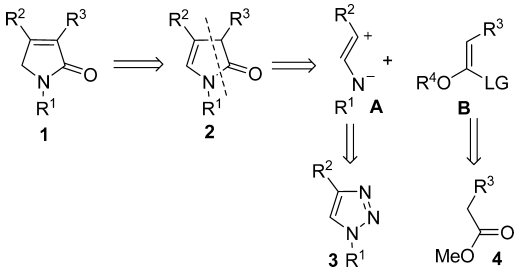

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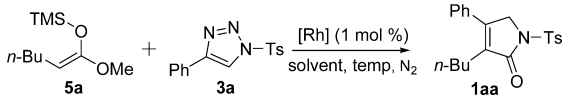
Table 1. Optimization of Reaction Conditions[a]



entry	catalyst	solvent	t (°C)	t (h)	yield (%)[b]
1	$Rh_2(Oct)_4$	DCE	80	9.5	83
2	$Rh_2(OAc)_4$	DCE	80	7.0	93
3	$Rh_2(OOCCF_3)_4$	DCE	80	9.0	0[c]
4	$Rh_2(Piv)_4$	DCE	80	12.0	80
5	$Rh_2(S\text{-}ntv)_4$	DCE	80	9.0	76
6	$Rh_2(S\text{-}nttl)_4$	DCE	80	7.0	24
7	$Rh_2(OAc)_4$	$CHCl_3$	70	11.5	66
8	$Rh_2(OAc)_4$	toluene	80	11.0	43
9	$Rh_2(OAc)_4$	CH_3CN	80	12.0	0[d]
10	$Rh_2(OAc)_4$	DCE	50	29.0	10
11	$Rh_2(OAc)_4$	DCE	60	21.0	19
12	$Rh_2(OAc)_4$	DCE	70	6.0	72
13	$Rh_2(OAc)_4$	DCE	90	11.5	87
14		DCE	80	12.0	0[e]

[a]0.2 mmol of triazole **3a** and 0.36 mmol of ketene silyl acetal **5a** were used; the reaction was carried out in 2 mL of solvent under N_2, DCE = 1,2-dichloroethane. [b]Yield of isolated products. [c]97% of **3a** was recovered. [d]78% of **3a** was recovered. [e]98% of **3a** was recovered.

To test this hypothesis, we began our study using readily available ketene silyl acetal **5a** and triazole **3a**. Gratifyingly, treatment of **3a** with 1.8 equiv of **5a** in the presence of $Rh_2(Oct)_4$ afforded 3-pyrrolin-2-one **1aa** in 83% yield (Table 1, entry 1). Attempts to improve this reaction by screening other catalysts revealed that $Rh_2(OAc)_4$ was a better catalyst (entry 2). When we examined the solvent effects (entries 7–9), we found that DCE gave the best yield of **1aa** (93%). Reaction temperature is a key factor in triazole decomposition, and temperatures higher or lower than 80 °C only led to decreased yield (entries 10–13). Without $Rh_2(OAc)_4$, 98% of the triazole was recovered after 12 h (entry 14).

It has been reported by Tang[11k] that an aryl group on the aryl sulfonyl azide impacted the reactivity of the triazole and the resulting carbene. The scope of the reaction was subsequently examined by first varying the sulfonyl group. As seen from the results compiled in Table 2, the electronic and steric properties have great impact on the yield (entries 1–6). Alkylsulfonyl substituted triazole **3b** and **3g** afforded the corresponding products in moderate yield (entries 1 and 6). Substrates with electron-rich aryl groups at the sulfonyl substituent led to **1ad** and **1ae** with high yield, while **1ac** was obtained in only 55% yield (entry 2). Notably, the reactions maintained satisfactory efficiency with substrates **3h**-**3p** containing a variety of functional groups including methoxy, fluoro, bromo, trifluoromethyl, methoxycarbonyl, and thienyl, the corresponding products **1ah**–**ap** were obtained in 64–91% yields. Finally, we focused our attention on the variation of the ketene silyl acetals. Compounds **5d** and **5e** bearing aryl group also afforded the desired products, albeit in moderate yield (entries 19 and 20). Relatively higher yields were obtained when alkyl ketene silyl acetals were employed (entries 16–18).

When triazole **3a** was treated with dirhodium acetate at 80 °C in the presence of **5b**, 3-pyrrolin-2-one **1ba** was obtained in 66% yield after 16 h (Table 2, entry 16). However, if we quenched the reaction when TLC analysis showed that triazole was just completely consumed (3.6 h), compounds **6** could be isolated in 40% yield (eq 1). On the basis of these experiments, a plausible

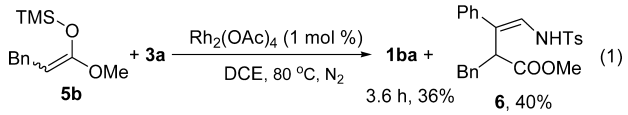
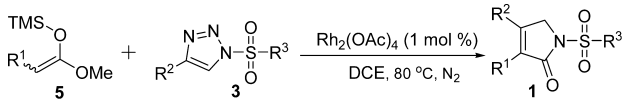


Table 2. Reaction Scope[a]

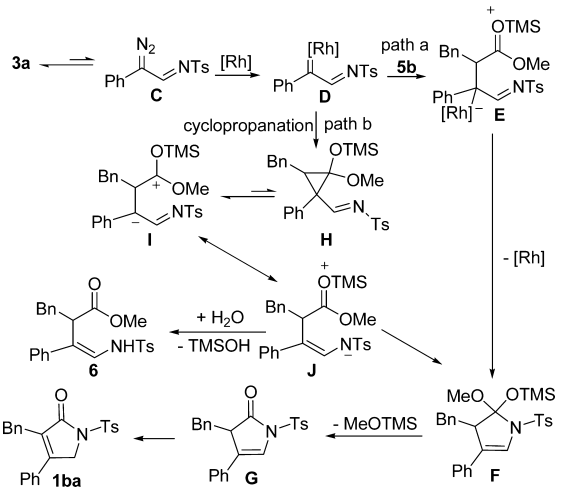


entry	**5** (R^1)	**3** (R^2, R^3)	t (h)	yield (%)[b]
1	**5a** (*n*-Bu)	**3b** (Ph, Me)	23.0	**1ab** (56)
2	**5a** (*n*-Bu)	**3c** (Ph, *p*-BrC_6H_4)	12.0	**1ac** (55)
3	**5a** (*n*-Bu)	**3d** (Ph, *p*-$MeOC_6H_4$)	18.0	**1ad** (78)
4	**5a** (*n*-Bu)	**3e** (Ph, 2-naphthyl)	16.0	**1ae** (85)
5	**5a** (*n*-Bu)	**3f** (Ph, 2,4,6-triisopropylC_6H_2)	20.0	**1af** (61)
6	**5a** (*n*-Bu)	**3g** (Ph, $TMSCH_2CH_2$)	24.0	**1ag** (55)
7	**5a** (*n*-Bu)	**3h** (*p*-MeC_6H_4, *p*-MeC_6H_4)	16.0	**1ah** (68)
8	**5a** (*n*-Bu)	**3i** (*p*-$MeOC_6H_4$, *p*-MeC_6H_4)	9.5	**1ai** (87)
9	**5a** (*n*-Bu)	**3j** (*o*-$MeOC_6H_4$, *p*-MeC_6H_4)	8.0	**1aj** (64)
10	**5a** (*n*-Bu)	**3k** (*m*-FC_6H_4, *p*-MeC_6H_4)	16.0	**1ak** (75)
11	**5a** (*n*-Bu)	**3l** (*p*-FC_6H_4, *p*-MeC_6H_4)	16.5	**1al** (81)
12	**5a** (*n*-Bu)	**3m** (*m*-$CF_3C_6H_4$, *p*-MeC_6H_4)	17.5	**1am** (78)
13	**5a** (*n*-Bu)	**3n** (*p*-BrC_6H_4, *p*-MeC_6H_4)	11.0	**1an** (91)
14	**5a** (*n*-Bu)	**3o** (*p*-$MeOCOC_6H_4$, *p*-MeC_6H_4)	11.5	**1ao** (78)
15	**5a** (*n*-Bu)	**3p** (3-thienyl, *p*-MeC_6H_4)	30.0	**1ap** (72)
16	**5b** (Bn)	**3a** (Ph, *p*-MeC_6H_4)	16.0	**1ba** (66)
17	**5c** (*n*-hexyl)	**3a** (Ph, *p*-MeC_6H_4)	9.0	**1ca** (84)
18	**5c** (*n*-hexyl)	**3n** (*p*-BrC_6H_4, *p*-MeC_6H_4)	8.0	**1cn** (95)
19	**5d** (Ph)	**3a** (Ph, *p*-MeC_6H_4)	17.0	**1da** (52)
20	**5e** (*p*-$MeOC_6H_4$)	**3a** (Ph, *p*-MeC_6H_4)	9.0	**1ea** (58)

[a]In the presence of 0.002 mmol of $Rh_2(OAc)_4$, 0.2 mmol of triazole **3** and 0.36 mmol of **5** were reacted in 2 mL of DCE under N_2. [b]Yield of isolated products, average of two runs.

mechanism to rationalize the formation of **1ba** is proposed. The equilibrium is intercepted by $Rh_2(OAc)_4$ to afford highly electrophilic α-imino rhodium carbenoid **D** (Scheme 2), which

Scheme 2. Proposed Mechanism



is attacked by **5b** to give rise to intermediate **E** (path a). Subsequent intramolecular nucleophilic addition of the nitrogen of the imino group would result in the formation of heterocyclic compound **F**, which undergoes elimination of MeOTMS and migration of carbon carbon double bond to afford 3-pyrrolin-2-one **1ba**. In another pathway, cyclopropane **H** is formed and

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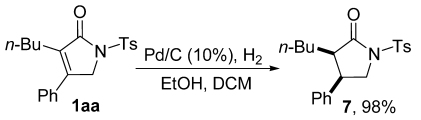
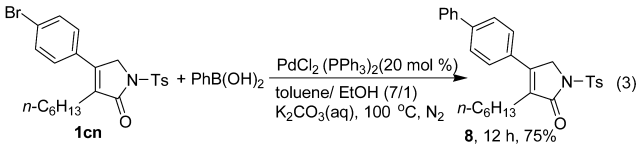
collapses into stabilized zwitterionic intermediate **I** or **J**, which also leads to the formation of **F** by intramolecular nucleophilic addition. In the NMR studies, cyclopropane **H** was not observed and compound **F** was detected as a mixture of diastereoisomers (see the Supporting Information for more details). However, since we can isolate compound **6** in 40% yield (eq 1), cyclopropane **H** should be one of the key intermediates in this reaction. We believe the reason we can not detect **H** is that this donor−acceptor cyclopropane is not stable under the reaction conditions, compound **F** is obtained by rearrangement and compound **6** is formed via reaction with water. Thus, both of these two pathways are possible.

Notably, the corresponding γ-lactam **7** could be easily obtained by hydrogenation of compound **1aa** with 10% Pd/C under 6 atm of H_2 (eq 2). Suzuki coupling of **1cn** and $PhB(OH)_2$



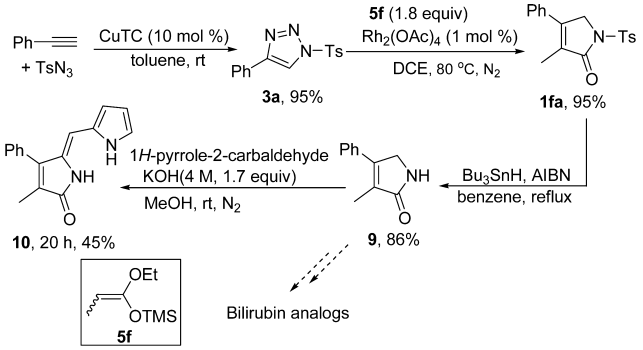
(2)



(3)

afforded compound **8** in 75% yield (eq 3). Finally, the transformation was used in the synthesis of a key intermediate (compound **9**, Scheme 3) of bilirubin analogue. Bilirubin is a bile

Scheme 3. Synthesis of 3-Methyl-4-phenyl-1*H*-pyrrol-2(5*H*)-one



pigment and a powerful antioxidant, and its aromatic congener is bioactive too.[15] In the literature, Barton−Zard reaction between (*p*-toluenesulfonyl)methyl isocyanide and 2-nitro-1-phenylpropanol acetate led to the formation of 4-methyl-3-phenyl-2-(*p*-toluenesulfonyl)pyrrole, which underwent bromination, hydrolysis, and reduction to afford the key compound **9**, and the overall yield based on the isocyanide was 42%.[15] Starting from commercially available phenylacetylene, compound **9** can be synthesized in 78% yield over three steps. In the presence of KOH, **9** can react with pyrrole-2-carbaldehyde to afford compound **10**.

In summary, a novel and convenient method to synthesize 3,4-disubstituted 3-pyrrolin-2-one using 1-sulfonyl 1,2,3-triazole and ketene silyl acetal has been developed. As a new example to demonstrate the synthetic potential of 1-sulfonyl 1,2,3-triazole as α-imino carbene precursor, a broad range of functional groups were well tolerated and the operating process is quite simple. Moreover, since the triazole and the ketene silyl acetal deliver each of the two substituents of the products, this strategy offers much synthetic flexibility in comparison with the traditional methods.

■ ASSOCIATED CONTENT

Supporting Information

Experimental procedures, characterization data, and NMR spectra for new compounds. This material is available free of charge via the Internet at http://pubs.acs.org.

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Notes

The authors declare no competing financial interest.

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