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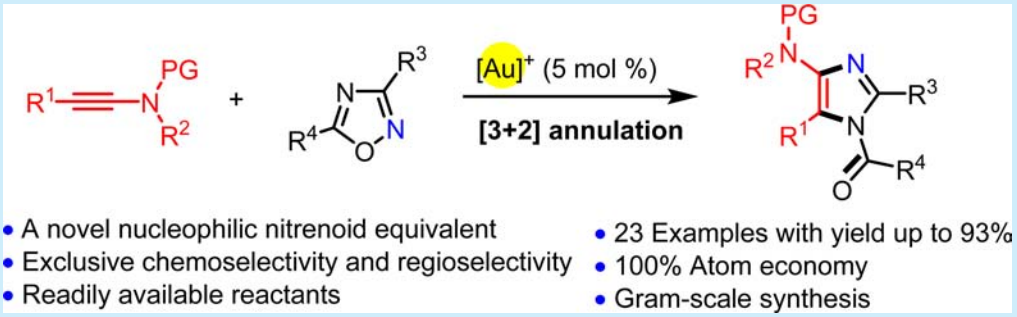
α-Imino Gold Carbenes from 1,2,4-Oxadiazoles: Atom-Economical Access to Fully Substituted 4-Aminoimidazoles

Zhongyi Zeng,[†] Hongming Jin,[†] Jin Xie,[†] Bing Tian,[†] Matthias Rudolph,[†] Frank Rominger,[†] and A. Stephen K. Hashmi*[,†,‡]

[†]Organisch-Chemisches Institut, Ruprecht-Karls-Universität Heidelberg, Im Neuenheimer Feld 270, 69120 Heidelberg, Germany

[‡]Chemistry Department, Faculty of Science, King Abdulaziz University (KAU), 21589 Jeddah, Saudi Arabia

Supporting Information



ABSTRACT: A novel and atom-economical synthesis of fully substituted 4-aminoimidazoles via gold-catalyzed selective [3 + 2] annulation of 1,2,4-oxadiazoles with ynamides is reported. This protocol represents a new strategy to access α-imino gold carbenes, which corresponds to an unprecedented intermolecular transfer of *N*-acylimino nitrenes to ynamides. Moreover, the reaction proceeds with 100% atom economy, exhibits good functional group tolerance, and can be conducted in gram scale.

As one fundamental framework, imidazole motifs frequently exist in a large number of natural products,[1] pharmaceuticals,[2] ionic liquids,[3] and precursors of *N*-heterocyclic carbenes.[4] In this family, fully substituted 4-aminoimidazole derivatives are core structures of some bioactive compounds and also serve as versatile building blocks (Figure 1).[5] However, in contrast with the wealth of imidazole syntheses, few routes can allow direct access to fully substituted 4-aminoimidazoles.[6] Hence, the development of general and practical methods for the construction of highly functionalized 4-aminoimidazole scaffolds is meaningful to synthetic and medicinal chemistry.

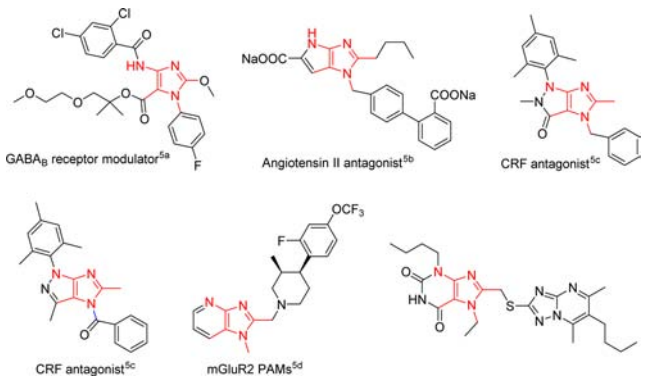


Figure 1. Representative bioactive compounds with fully substituted 4-aminoimidazole frameworks (in red).

Recently, we and other groups verified that highly reactive α-imino gold carbenes generated in situ from various nucleophilic nitrenoid equivalents, such as azides,[7] *N*-iminopyridium ylides,[8] 2*H*-azirines,[9] and very recently developed isoxazoles,[10] anthranils,[11] triazapentalenes,[12] dioxazoles,[13] and pyrido[1,2-*b*]indazoles,[14] provided a powerful platform to construct an array of structurally diverse aza-heterocycles.[15] Notwithstanding the recent advance attained, the exploration of α-imino gold carbene chemistry with less sensitive nitrene-transfer reagents, especially in an atom-economical and selective manner, is highly desirable.

1,2,4-Oxadiazole is an appealing candidate due to its stability, structural diversity, and simple handling. To date, the further transformations of 1,2,4-oxadiazoles were still surprisingly scarce.[16,17] One representative work is the photoinduced rearrangement of 1,2,4-oxadiazoles to form *N*-acylimino nitrene intermediates which were subsequently trapped by different nucleophiles (Scheme 1a).[17] Inspired by our previous work on gold carbenes,[11,18] we considered the possibility of using 1,2,4-oxadiazoles as formal nitrene equivalents to form α-imino gold carbene intermediates via regioselective addition to gold-activated ynamides. Based on their high electrophilicity, subsequent intramolecular chemoselective trapping completed a [3 + 2] annulation, affording fully substituted 4-amino-imidazoles (Scheme 1b). If successful, this process would open

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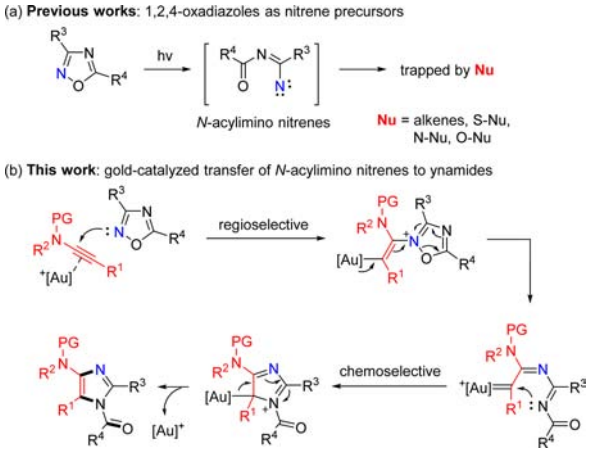
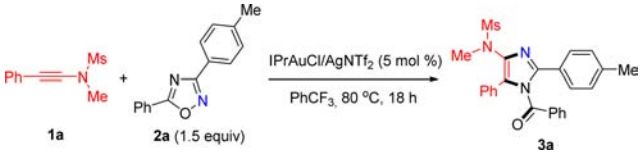
Scheme 1. Previous Works and Our Design



a new window for the reaction pattern of 1,2,4-oxadiazoles and also complement the existing strategies for the synthesis of polysubstituted 4-aminoimidazole derivatives. Herein, we disclose a new route to α-imino gold carbenes from 1,2,4-oxadiazoles and ynamides, enabling the convergent and atom-economical synthesis of fully substituted 4-aminoimidazoles.

To evaluate the feasibility, ynamide **1a** and 1,2,4-oxadiazole **2a** were initially chosen for the model reaction (Table 1).

Table 1. Screening for Optimal Reaction Conditions[a]



entry	deviation from "standard" conditions	yield[b] (%)
1	none	95 (90)
2	no catalyst	nd
3	$AgNTf_2$	trace
4	PPh_3AuNTf_2	52
5	$(2,4\text{-}^tBu_2PhO)_3PAuCl/AgNTf_2$	30
6	$SPhosAuNTf_2$	48
7	$KAuBr_4$	39
8	IPrAuCl/AgOTf	67
9	DCE, instead of $PhCF_3$	72
10	toluene, instead of $PhCF_3$	57
11	60 °C	54

[a]"Standard" conditions: **1a** (0.1 mmol), **2a** (0.15 mmol), IPrAuCl/$AgNTf_2$ (5 mol %) reacted in $PhCF_3$ (0.5 mL) for 18 h at 80 °C. [b]Measured by ^1H NMR with 1,3,5-trimethoxybenzene as the internal standard. Yield of isolated product given in parentheses. nd = not detected.

Gratifyingly, employing 5 mol % of IPrAuCl/$AgNTf_2$ as a catalyst at 80 °C can afford the desired product **3a** in 95% NMR yield (entry 1). Control experiments without any catalyst or silver salt alone showed no conversion (entries 2 and 3). Other gold catalysts, PPh_3AuNTf_2, $(2,4\text{-}^tBu_2PhO)_3PAuCl/AgNTf_2$, $SPhosAuNTf_2$, and $KAuBr_4$, were much less efficient, leading to **3a** in lower yields (entries 4–7). Switching the counteranion from NTf_2 to OTf led to a significantly decreased yield (95% versus 67%, entries 1 and 8). In addition, other solvents such as DCE and toluene could not improve the reaction efficiency (entries 9 and 10). Decreasing the reaction temperature from 90 to 60 °C delivered **3a** in moderate yield (entry 11).

Under the optimized reaction conditions, the scope of this novel transformation was investigated (Figure 2). First, with

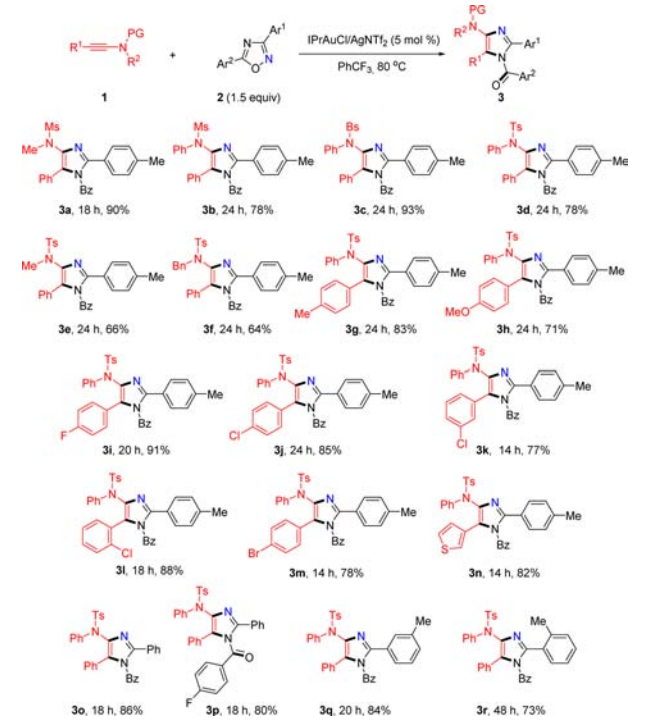


Figure 2. Reaction scope for ynamides and 3,5-diaryl-1,2,4-oxadiazoles. Reaction conditions: **1** (0.2 mmol), **2** (0.3 mmol), IPrAuCl/$AgNTf_2$ (5 mol %), $PhCF_3$ (1 mL), 80 °C. Isolated yields.

1,2,4-oxadiazole **2a** as the reaction partner, diverse ynamides bearing different protecting groups (Ms, Ts and Bs) and substituents on nitrogen tolerated the reaction conditions well, furnishing **3a–f** in satisfying yields. We then examined the influence of the R^1 group on the alkyne terminus. Aryl-substituted ynamides with various substituents on the phenyl ring uniformly afford the desired products **3g–m** in 71–91% yields, regardless of their electronic and positional properties. An array of functional groups, including chloride, bromide, fluoride, ether, and ester, remained intact, offering opportunities for further modification at these positions (**3h–m,p,w**). When thiophene-yl-substituted ynamide was subjected to standard conditions, imidazole **3n** was obtained in high yield. With regard to the scope of 3,5-diaryl-1,2,4-oxadiazoles, it was found that electron-donating, electron-withdrawing, and neutral substituents on the aromatic ring were all compatible (**3e,o–r**).

Encouraged by these results, we further broaden the scope of this reaction by using a series of 1,2,4-oxadiazoles bearing one alkyl group on the parent ring (Figure 3). However, the use of 5 equiv of 1,2,4-oxadiazoles **2** was necessary to obtain the desired products in acceptable yields, probably due to their reduced nucleophilicity. In this case, the 4-aminoimidazole products were afforded in moderate yields. An ester group was tolerated in this transformation, providing **3w** in 41% yield. To further confirm the 4-aminoimidazole framework, X-ray crystallographic analysis of **3v** was conducted (Figure 3).[19]

To further probe the practicality of this methodology, a gram-scale conversion with a slightly lower catalyst loading (4 mol %) delivered 4-aminoimidazole **3a** in a good yield of 80%

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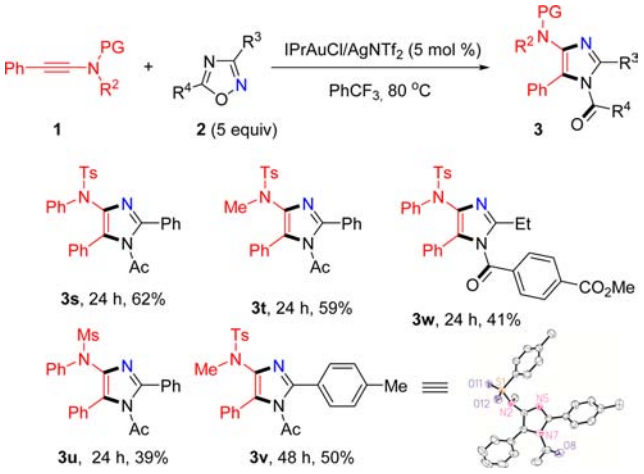
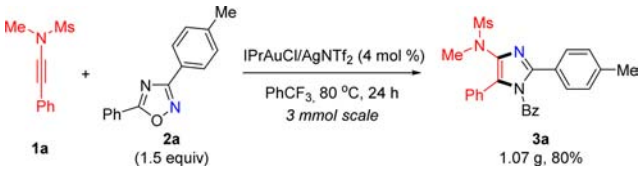


Figure 3. Reaction scope for 3,5-disubstituted 1,2,4-oxadiazoles. Reaction conditions: **1** (0.2 mmol), **2** (1 mmol), IPrAuCl/$AgNTf_2$ (5 mol %), $PhCF_3$ (1 mL), 80 °C. Isolated yields.

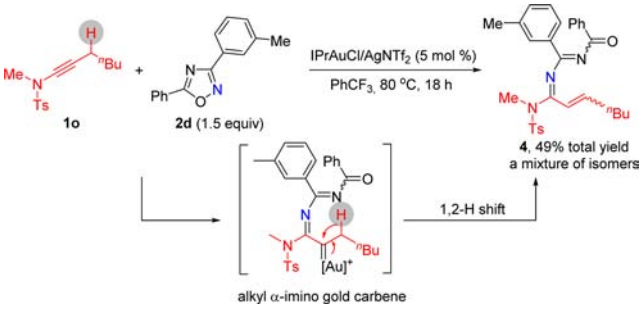
(Scheme 2). Finally, we prepared alkyl-substituted ynamide **1o** and then reacted it with 1,2,4-oxadiazole **2d** under the standard

Scheme 2. Gram-Scale Synthesis



conditions. As expected, α,β-unsaturated imine **4** was isolated in 49% total yield without detection of the desired imidazole (Scheme 3). The successful 1,2-H shift leading to alkenes indicates that the α-imino gold carbenoid intermediate is very likely.[8a,20]

Scheme 3. Reaction of Alkyl-Substituted Ynamide with 1,2,4-Oxadiazole



In conclusion, we have demonstrated that 1,2,4-oxadiazoles could serve as novel nucleophilic nitrenoid equivalents for the generation of α-imino gold carbenes, corresponding to an intermolecular transfer of *N*-acyliminonitrenes to ynamides. This protocol offers a new reaction pattern of 1,2,4-oxadiazoles and opens up a novel and atom-economical strategy for the synthesis of valuable fully substituted 4-aminoimidazoles. The present reaction proceeds with 100% atom economy, displays good functional group compatibility, and can be conducted in gram-scale synthesis. Further applications of 1,2,4-oxadiazoles in other organic syntheses are ongoing in our group.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.7b00001.

Experimental procedures and spectroscopic characterization data (PDF)

X-ray data for **3v** (CIF)

■ AUTHOR INFORMATION

Corresponding Author

*E-mail: hashmi@hashmi.de.

ORCID

A. Stephen K. Hashmi: 0000-0002-6720-8602

Notes

The authors declare no competing financial interest.

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