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Gold(III)-Catalyzed Chemoselective Annulations of Anthranils with *N*-Allylynamides for the Synthesis of 3-Azabicyclo[3.1.0]hexan-2-imines

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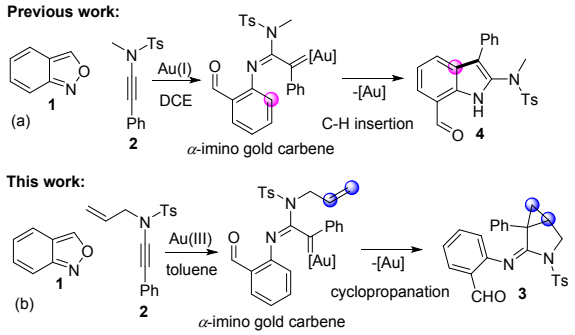
We herein report the gold(III)-catalyzed selective annulation of anthranils with *N*-allylynamides under mild conditions. By trapping the in situ-generated α-imino gold carbenes, 3-azabicyclo[3.1.0]hexan-2-imines were obtained in high synthetic efficiency. The reaction, which can be conducted in gram scale, tolerates electron-rich and electron-deficient anthranils as well as a diverse set of functionalized ynamides (aryl-, alkyl-substituted, terminal).

The cyclopropane core has attracted tremendous attention as it widely exists in natural products and synthetic bioactive agents, and it also can serve as a versatile intermediate in organic synthesis.[1] Among the methods established for preparing cyclopropanes,[2] transition metal-catalyzed reactions of 1,5-enynes[3] are straightforward and efficient. These versatile unsaturated systems can undergo diverse cyclizations to form novel carbocycles and heterocycles. Based on their π acidity, among the applied transition metals, gold catalysts[4] have become a very powerful tool for catalytic cyclizations of 1,5-enynes, by enabling the activation of C–C triple bonds, followed by the intramolecular nucleophilic attack of olefins. This is exemplified by the cycloisomerisation of 1,5-enynes for the synthesis of bicyclo[3.1.0]hexenes.[4b] Recently, the gold-catalyzed synthesis of cyclopropyl indanone derivatives from fused 1,5-enynes through α-oxo gold carbene intermediates were reported.[5] Typically, the gold-activated C–C triple bond of an 1,5-enyne was treated with a carbene precursor (commonly a *N*-oxide) to form a highly reactive gold carbene intermediate, which was rapidly trapped by an alkenyl group to produce a cyclopropane-containing bicycle. This strategy was applied for the enantioselective synthesis of (−)-Nardoaristolone B.[5f] *N*-Allylynamides,[6] as aza-1,5-enynes, were also suitable substrates. Especially, because of the adjacent nitrogen atom, the C–C triple bond is highly activated which facilitates a transition metal-catalyzed oxygen transfer under mild conditions to generate a α-keto metal carbene, which subsequently undergoes an intramolecular cyclopropanation to deliver an 3-azabicyclo[3.1.0]hexan-2-one.[6f-g] Whereas oxidative cyclopropanations of *N*-allylynamides by oxygen donors are well explored, the corresponding cyclopropanations by α-imino gold carbenes, derived from nitrene transfer reagents, remain comparatively underdeveloped.[7] Only our group recently reported an intermolecular gold nitrene transfer from *N*-arylsulfilimines[7b] to ynamides for the synthesis of 3-azabicyclo[3.1.0]hexan-2-imines enabled by α-imino gold carbene intermediates. However, this reaction is limited to sulfilimines bearing strong electron-withdrawing groups (Ms, NO_2, CF_3) on the arene rings, and a general cyclopropanation reaction with excellent functional group tolerance is highly challenging and of great significance.



Scheme 1 Gold-catalyzed divergent annualtion of anthranils with ynamides.

Gold-catalyzed oxygen,[8] nitrene[7b,9] and carbene transfer[10] provides access to functionalized gold carbenes, which are subsequently trapped by different functional groups to produce structural diversity, have experienced a rapid development in the last two decades. Since the first gold-catalyzed annulation between isoxazoles and ynamides was reported by Ye et al.,[9k]

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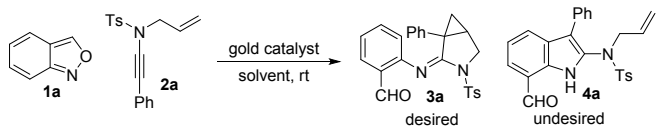
these versatile reagents have been vastly used as gold carbene precursors, allowing the facile syntheses of various aza-heterocycles. Our group described a [3 + 2] C–H annulation of anthranils with ynamides for the synthesis of 7-acylindoles[9l] (Scheme 1a). Inspired by previous research,[8-11] with *N*-allylynamides, we presumed that a bicyclic product **3** might be formed through cyclopropanation of the generated α-imino gold carbene intermediates (Scheme 1b).

With this in mind, our investigation began by employing anthranil **1a** and *N*-allylynamide **2a** as model substrates (Table 1). First, IPrAuCl/$AgNTf_2$ as catalyst delivered indole **4a** and 3-azabicyclo[3.1.0]hexan-2-imine **3a** in 30% and 65% yields at room temperature (Table 1, entry 1). We then sought to selectively synthesize the bicycle **3a** by optimizing the reaction conditions. Phosphine ligands such as PPh_3 and JohnPhos didn't improve the selectivity (Table 1, entries 2-3). AuCN led to indole **4a** as a major product (Table 1, entry 4). A gold(III) catalyst, $PicAuCl_2$, significantly improved the reaction with a high ratio of **3a**/**4a** (Table 1, entry 5). The solvent (DCE, THF, CH_3CN) screening showed that using this procedure with toluene as the solvent provided the highest yield of **3a** (Table 1, entries 6-8). Simple $NaAuCl_4{\cdot}2H_2O$, was highly beneficial for the formation of product **3a** and thus was chosen as the best performer (Table 1, entry 9). Furthermore, in the absence of a gold catalyst, no reaction was observed (Table 1, entry 10).

Table 1 Optimization of the reaction conditions[a,b]

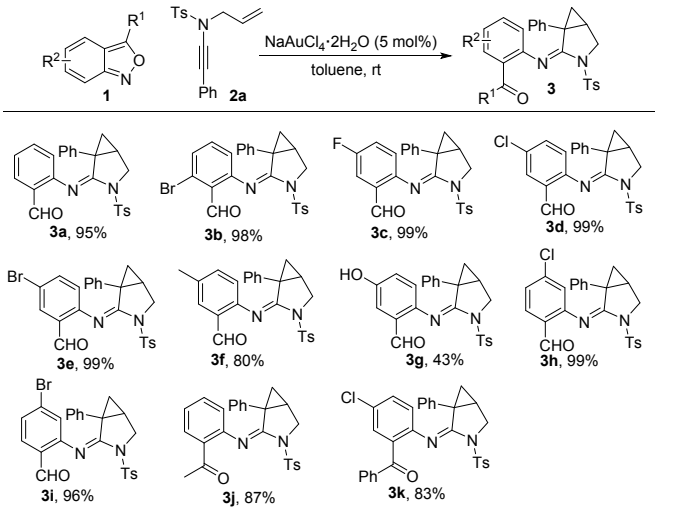


entry	catalyst	solvent	yield of **3a**	yield of **4a**
1	IPrAuCl[c]/$AgNTf_2$	toluene	65%	30%
2	PPh_3AuCl/$AgNTf_2$	toluene	54%	40%
3	JohnPhosAuCl[d]/ $AgNTf_2$	toluene	39%	53%
4	AuCN	toluene	24%	65%
5	$PicAuCl_2$	toluene	91%	6%
6	$PicAuCl_2$	DCE	20%	55%
7	$PicAuCl_2$	THF	82%	13%
8	$PicAuCl_2$	CH_3CN	57%	18%
9	**$NaAuCl_4{\cdot}2H_2O$**	**toluene**	**97%**	**trace**
10	-	toluene	n. d.[e]	n. d.[e]

[a] General reaction conditions: **1a** (0.1 mmol, 1.0 equiv.), **2a** (0.12 mmol, 1.2 equiv.), catalyst (5 mol%), solvent (1.0 mL, 0.1 M). [b] Yields of **3a** and **4a** measured by ^1H NMR with 1,3,5-trimethoxybenzene as the internal standard. [c] IPr = 1,3-bis(2,6-diisopropylphenyl)-1,3-dihydro-2*H*-imidazol-2-ylidene. [d] JohnPhos = 2-(di-*tert*-butylphosphino)biphenyl. [e] n. d.: not detected.

Having established the optimal reaction conditions, we examined the reaction scope with respect to anthranils. As shown in Table 2, the unsubstituted anthranil **1a** gave product **3a** in 95% yield. 3-Bromoanthranil **1b** is also a successful participant in this cyclopropanation reaction. Benzoisoxazoles **1c**-**e** bearing halogens such as F, Cl, Br were particularly well tolerated to quantitatively produce the desired 3-azabicyclo[3.1.0]hexan-2-imines **3c**-**e**. An electron-sufficient substrate, 5-methylanthranil **1f**, gave the desired product in good yield. As a sensitive electron-donating group, a hydroxyl group remained untouched and the desired hydroxyl-containing bicycle **3g** was prepared in 43% yield. 6-Chloro- and 6-bromoanthranils reacted smoothly with *N*-allylynamide **2a**, furnishing **3h** and **3i** in excellent yield. 3-Substituted anthranils **1j** and **1k** afforded the targets in lower yields.

Table 2 Scope with respect to anthranils[a, b]



[a] Optimized reaction conditions: **1** (0.2 mmol, 1.0 equiv.), **2a** (0.24 mmol, 1.2 equiv.), $NaAuCl_4{\cdot}2H_2O$ (5 mol%, 4.0 mg), toluene (2.0 mL, 0.1 M). [b] Isolated yield of products **3** are shown.

Encouraged by the excellent performance of anthranils, we further investigated the scope by using a variety of *N*-allylynamides (Table 3). Gratifyingly, a broad range of substituted *N*-allylynamides is tolerated. Ynamide **2b** with a bromo substituent gave product **3l** in 99% yield, which is an excellent substrate for further classic cross coupling reactions. Bicycle **3m** was prepared from 3-chlorophenyl ynamide **2c**, the structure of which was further confirmed by X-ray crystal structure analysis.[12] Ynamides **2d**-**f**, bearing electron-donating groups (methyl, ethyl, *tert*-butyl) at 3- or 4-positions of the arene rings, afforded the desired targets **3n**-**p**[12] all in 99% yields. A CF_3-containing ynamide **2g** was also found to be suitable, albeit in 87% yield (**3q**). Other protecting groups including *n*-$PrSO_2$, $BnSO_2$ and the easily removable 4-nitrophenylsulfonyl did not retard the annulation reaction (**3r**-**t**). 2-Methylallyl ynamide **2k** (R^2 = Me) reacted efficiently with 5-bromoanthranil, yielding product **3u** with two vicinal quaternary carbon centers. An internal alkene-derived ynamide **2l** delivered 6-methyl-3-azabicyclo[3.1.0]hexan-2-imine **3v** in 95% yield in a diastereomeric ratio of 6.3:1. Moreover, a terminal ynamide also readily underwent the cyclopropanation pathway (**3w**). While our previous work has demonstrated that the α-imino gold carbene generated from an alkyl ynamide and anthranil will undergo a 1,2-hydride shift to form an unsaturated amidine, this reaction was compatible with a butyl-derived ynamide, delivering the desired product **3x** in 70% yield. This indicates,

that the intramolecular cyclopropanation of the gold carbene is much faster than a competing 1,2-C–H-insertion (Scheme 2).

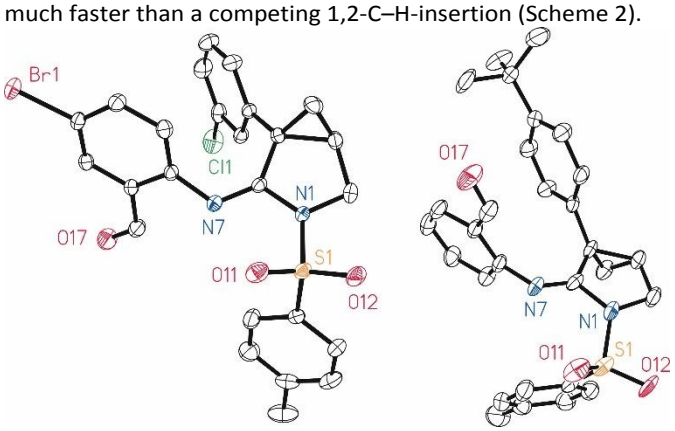
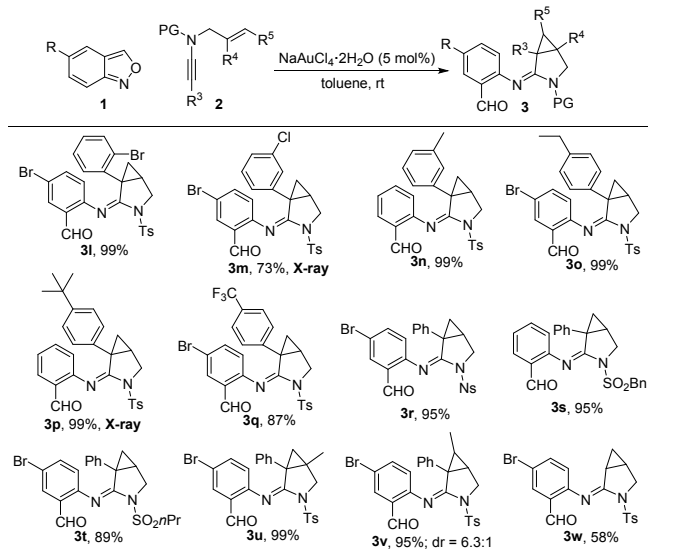
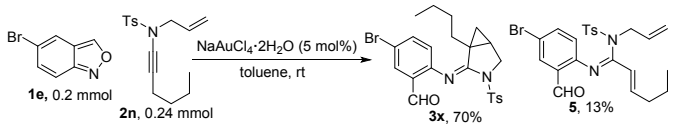


Figure 1 Solid state molecular structures of **3m** (left) and **3p** (right).

Table 3 Scope with respect to ynamides[a, b]

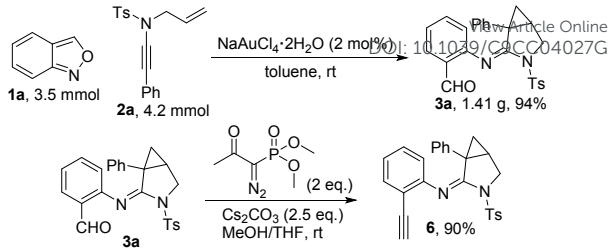


[a] Optimized reaction conditions: **1** (0.2 mmol, 1.0 equiv.), **2** (0.24 mmol, 1.2 equiv.), $NaAuCl_4{\cdot}2H_2O$ (5 mol%, 4.0 mg), toluene (2.0 mL, 0.1 M). [b] Isolated yields of product **3**.



Scheme 2 Test of an alkyl ynamide.

An efficient scale-up is possible, which was exemplified by the excellent-yield of a 3.5-mmol scale annulation of anthranil **1a** with ynamide **2a** (Scheme 3). 1.4 g of **3a** were obtained, **3a** was further converted to an alkynyl product **6** through Seyferth–Gilbert homologation.



Scheme 3 Gram-scale synthesis and an important transformation.

In conclusion, we developed a gold(III)-catalyzed selective annulation of anthranils and *N*-allylynamides for the synthesis of 3-azabicyclo[3.1.0]hexan-2-imines. This efficiently scalable reaction is associated with good functional group tolerance, great efficiency, easy synthesis of starting materials, mild conditions and 100% atom-economy. This reaction has significant potential for total synthesis, further applications of the obtained products will extend the significance of this methodology.

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Conflicts of interest

There are no conflicts to declare.

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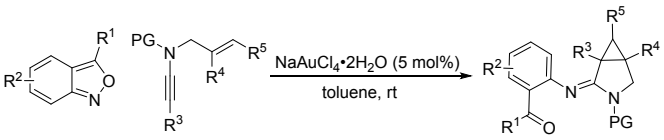

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