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Gold(I)-Catalyzed Domino Reaction of Aziridinyl Alkynes

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A novel gold(I)-catalyzed domino transformation of aziridinyl alkynes with arenes to construct 1,2,3,4-tetrahydroisoquinoline and 3,4-dihydroisoquinoline structural motifs, especially sterically congested *syn*-3,4-disubstituted 1,2,3,4-tetrahydroisoquinolines, is described herein. A plausible mechanism that proceeds through a benzylic cation is presented on the basis of deuterium-labeling and control experiments as well as the observed diastereoselectivities.

Gold-catalyzed domino reactions are powerful tools in organic synthesis utilized to access complex molecular frameworks that have drawn increasing attention.[1] One of the most interesting aspects of this field is the introduction of strained small-ring systems as molecular building blocks to initiate such domino reactions, because the relief of ring strain provides a potent thermodynamic driving force. Since the first example of a gold-catalyzed transformation of cyclopropane was reported by Thomas in 1976,[2] the number of gold-catalyzed reactions that use strained small rings, including cyclopropane, cyclopropene, oxirane, aziridine, and so on, has increased rapidly. It is known that aziridine is a versatile building block for the synthesis of many nitrogen-containing biologically active molecules. Among the procedures for the ring opening of aziridines, nucleophilic ring-opening reactions are one of the major routes to form highly functionalized compounds.[3] Moreover, a highly regiospecific $AuCl_3$/AgOTf-catalyzed ring-opening reaction of aziridines by arenes has recently been reported, affording β-arylamines in good yields.[4] Herein, we report a novel gold-catalyzed tandem reaction of aziridinyl alkynes that leads to 1,2,3,4-tetrahydroisoquinoline and 3,4-dihydroisoquinoline structural motifs under mild conditions.[5,6]

Preliminary experiments directed towards optimizing the conditions of the transformation were carried out with aziridinyl alkyne **1a** in the presence of silver or gold salts, as well as combinations of Au and Ag, in toluene (**2a**), at 20 °C (room temperature). We found that, by using a silver salt, $AuCl_3$, $AuCl_3$/AgOTf, $NaAuCl_4{\cdot}2H_2O$, or $HAuCl_4{\cdot}2H_2O$ as the catalyst, the reaction afforded the ring-opened product **3a** in moderate to good yields, but none of the desired product **4a** (Table 1, entries 1–7). Compound **3a** was obtained as the sole product, in 82% yield, in the presence of $HAuCl_4{\cdot}2H_2O$ (5 mol%) after 30 min and in 88% yield in the presence of 20 mol% of this catalyst within 2 min (Table 1, entries 6 and 7, respectively; see the Supporting Information for some other examples). However, the combined catalyst [Au(PPh_3)Cl]/AgOTf (5 mol%) produced **3a** in 35% yield, along with the domino-reaction product **4a** in 44% yield, at 0 °C (Table 1, entry 8). Further optimization of the reaction conditions revealed that the use of [Au(PPh_3)Cl]/$AgSbF_6$ (10 mol%) as the catalyst produced **4a** in 79% yield, as the sole product, at room temperature (20 °C; Table 1, entry 15) and that increasing the reaction temperature to 40 or 60 °C did not further improve the reaction outcome (Table 1, entries 18 and 19).

Under the optimized conditions, it was found that, for a variety of arenes, such as benzene (**2b**), 1,4- and 1,2-xylene (**2c** and **d**), mesitylene (**2e**), 1,2- and 1,4-dimethoxybenzene (**2f** and **g**), and benzo[1,3]dioxole (**2h**), the reaction proceeded smoothly to give the desired 1,2,3,4-tetrahydroisoquinolines **4b–h** in moderate to high yields, as a sole products (Table 2, entries 1–7). By using furan (**2i**) as the electrophile, the reaction afforded **4i** in 44% yield if [Au(PPh_3)Cl]/AgOTf (10 mol%) was used as the catalyst (Table 2, entry 8), because **4i** was formed in low yield in the presence of [Au(PPh_3)Cl]/$AgSbF_6$. The examination of naphthalene (**2j**) and anisole (**2k**) in this domino reaction revealed that the corresponding two regioisomers **4j** (α- and β-**4j**) and **4k** (*ortho*- and *para*-**4k**) were formed in good total yields

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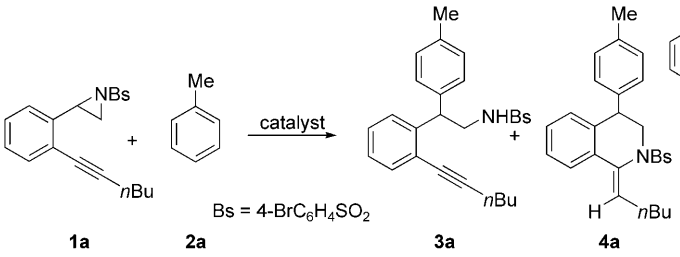
Table 1. Optimization of the reaction conditions.[a]



Entry	Catalyst [(mol%)]	T [°C]	t [min]	Yield [%][b]	
				3a[c]	**4a**[c]
1	AgOTf (5)	RT	120	33	–
2	$AgSbF_6$ (5)	RT	50	75	–
3	$AuCl_3$ (5)	RT	60	67	–
4	$AuCl_3$/AgOTf (5)	RT	30	41	–
5	$NaAuCl_4{\cdot}2H_2O$ (5)	RT	150	37	–
6	$HAuCl_4{\cdot}4H_2O$ (5)	RT	30	82	–
7	$HAuCl_4{\cdot}4H_2O$ (20)	RT	2	88	–
8	[Au(PPh_3)Cl]/AgOTf (5)	0	15	35	44
9	[Au(PPh_3)Cl]/AgOTf (5)	RT	40	16	57
10	[Au(PPh_3)Cl]/$AgBF_4$ (5)	RT	120	54	–
11	[Au(PMe_3)Cl]/$AgBF_4$ (5)	RT	120	23	17
12	[Au(IPr)Cl/]AgOTf (5)[d]	0	60	14	47
13	[Au(IPr)Cl]/$AgBF_4$ (5)[d]	0	70	55	8
14	[Au(PPh_3)Cl]/$AgSbF_6$ (5)	RT	30	–	65
15	[Au(PPh_3)Cl]/$AgSbF_6$ (10)	RT	50	–	79
16	[Au(PPh_3)Cl]/$AgSbF_6$ (10)[e]	RT	1 day	23	23
17	[Au(PPh_3)Cl]/$AgSbF_6$ (5)	60	30	–	66
18	[Au(PPh_3)Cl]/$AgSbF_6$ (10)	40	80	–	76
19	[Au(PPh_3)Cl]/$AgSbF_6$ (10)	60	30	–	78

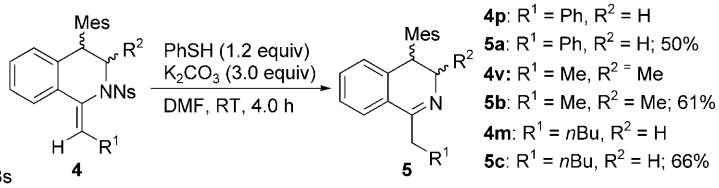
[a] Unless otherwise stated, all reactions were conducted with **1a** (0.200 mmol) with the catalyst in toluene (1.0 mL). [b] Isolated yield based upon aziridine **1a**. [c] The product includes traces of a regioisomer. [d] IPr = 1,3-diisopropylimidazolylidene. [e] The reaction was run in CH_3NO_2 (1.0 mL) with toluene (1.2 equiv).

(Table 2, entries 9 and 10). However, electron-deficient arenes, such as bromobenzene (**2l**) and nitrobenzene (**2m**), are not suitable substrates for this domino reaction, since complex product mixtures were formed under the standard conditions (Table 2, entries 11 and 12).

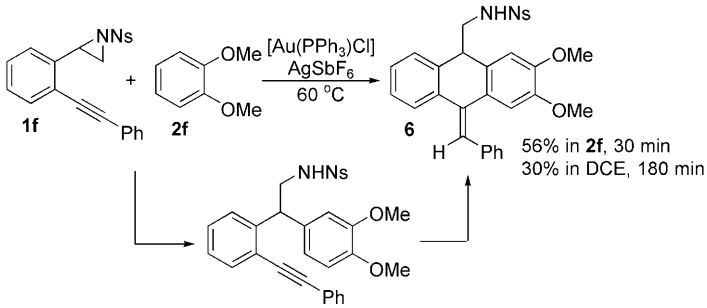
Under the optimized conditions, it was also found that, for a variety of aziridinyl alkynes **1**, the reactions proceeded smoothly with arenes **2e**, **a**, **f**, or **h** to give the corresponding tetrahydroisoquinoline derivatives in moderate to good yields. For the substituted aziridines **1d** and **h**–**k**, the products **4n**, **s**–**v**, and **x** were obtained as *syn*- and *anti*-isomeric mixtures (Table 3, entries 3, 8–11, and 13).[7] By using aziridinyl alkyne **1l** [R^1 = trimethylsilyl (TMS)] as the substrate the reaction afforded **4w** in 79% yield, in which the elimination of the TMS group occurred during the reaction with **2e** (Table 3, entry 12).

The *para*-nitrobenzenesulfonyl group (Ns) can easily be removed from products containing it by treatment with thiophenol in the presence of K_2CO_3. This gives access to the corresponding 3,4-dihydroisoquinolines **5**, in good yields (Scheme 1).[8]

It should be noted that the reaction of **1f** with **2f** gave product **6** rather than the corresponding tetrahydroisoquinoline derivative, presumably due to the steric and electronic properties of **2f** (Scheme 2). The ring-opened intermediate of the reaction of electron-rich arene **2f** with **1f** may more easily undergo nucleophilic attack of the aromatic carbon to the gold(I)-activated alkyne than attack by the nitrogen



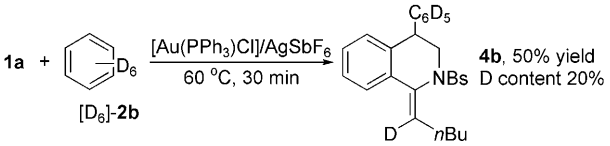
Scheme 1. The removal of the *para*-nitrobenzenesulfonyl group from compounds **4**; Mes = mesityl.



Scheme 2. The gold(I)-catalyzed reaction of aziridine **1f** with arene **2f**.

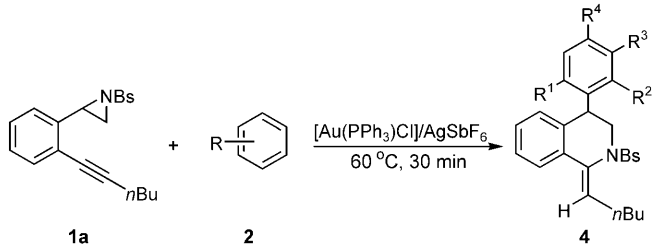
atom in the amine moiety, since this nucleophilic attack is sterically favored and the aromatic ring is very electron-rich in this particular case. Alternatively, an aryl–gold complex of the electron-rich arene with gold(I) may be formed, which may be responsible for this intramolecular hydroarylation reaction, although the formation of an aryl–gold complex is not proven at the present stage.[9]

To verify the reaction pathway, a control experiment was performed using **3a** as the starting material and it was found that **4a** could be obtained in 98% yield by using [Au(PPh_3)Cl]/$AgSbF_6$ as the catalyst, indicating that compounds **3** are intermediates in this domino reaction. Moreover, deuterium labeling experiments were carried out utilizing **1a** as the substrate in [D_6]benzene ([D_6]-**2b**), under the standard conditions. The corresponding product [D]-**4b** was formed in 50% yield with 20% of this product containing D as the olefinic proton ([D_6]-**4b**), supporting the internal hydrogen transfer from the arene to the final product as shown in Scheme 3 (see the Supporting Information for more details).[10]

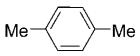
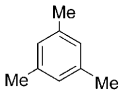
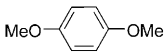
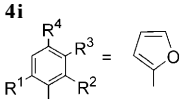
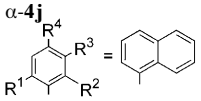
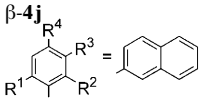
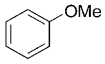
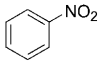


Scheme 3. A deuterium-labeling experiment.

Table 2. Gold(I)-catalyzed reaction of aziridine **1a** with arenes **2b–m**.[a]



1a **2** **4**

Entry	Arene		Product	Yield [%][b]
1		**2b**	**4b**, $R^1=R^2=R^3=R^4=H$	52
2		**2c**	**4c**, $R^1=R^3=Me$, $R^2=R^4=H$	79
3		**2d**	**4d**, $R^1=R^2=H$, $R^3=R^4=Me$	97[c]
4		**2e**	**4e**, $R^1=R^2=R^4=Me$, $R^3=H$	98
5		**2f**	**4f**, $R^1=R^2=H$, $R^3=R^4=OMe$	78, 63[d]
6		**2g**	**4g**, $R^1=R^3=OMe$, $R^1=R^2=H$	71, 55[d]
7		**2h**	**4h**, $R^3=R^4=OCH_2$, $R^2=R^4=H$	69[d]
8		**2i**	**4i** 	44[e]
9		**2j**	α-**4j**  β-**4j** 	60[d] (1.5:1)
10		**2k**	*ortho*-**4k**, $R^1=OMe$, $R^2=R^3=R^4=H$ *para*-**4k**, $R^4=OMe$, $R^1=R^2=R^3=H$	97 (1.7:1)
11		**2l**	–	–[f]
12		**2m**	–	–[f]

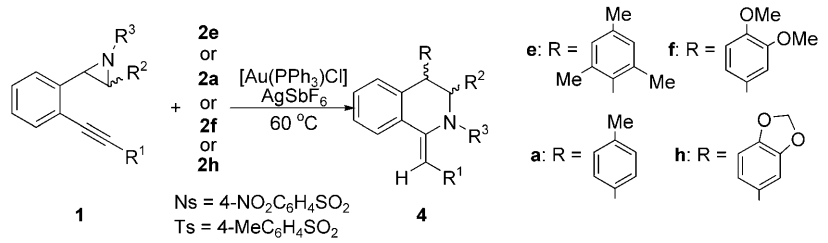
[a] All reactions were conducted with aziridine **1a** (0.200 mmol), $[Au(PPh_3)Cl]/AgSbF_6$ (10 mol%) in **2** as the solvent (1.0 mL) at 60°C for 30 min unless otherwise specified. [b] Isolated yield based upon aziridine **1a**. [c] The product includes traces of a regioisomer. [d] The reaction was run with **2** (2.0 equiv) in dichloroethane (DCE) (1.0 mL) at 60°C for 30 min. [e] $[Au(PPh_3)Cl]/AgOTf$ (10 mol%) was used as the catalyst for 30 min at RT. [f] Complex mixture of products.

According to the stereochemistry of the products **4n**, **s–v**, and **x** (shown in Table 3), *syn*-stereoselectivity is favored in this reaction. This stereochemical outcome can be rationalized by using the Friedel–Crafts reaction mechanism of a benzylic cation, according to the literature.[11] The preferred reaction conformations should, therefore, resemble zwitterionic structures (*R*)-**7** and (*S*)-**7** (Scheme 4).[11a] The preferred direction of attack from ArH should correlate to the preferred conformation and occurs from the bottom face of the plane due to steric hindrance. For the zwitterionic intermediates (*R*)-**7** and (*S*)-**7**, *syn*-**3** is formed preferentially, which is then smoothly transformed into the corresponding final product *syn*-**4**.

On the basis of the aforementioned experiments, a plausible reaction mechanism is outlined in Scheme 5. The initial step is the breaking of the C–N bond in the presence of a Lewis acid, such as $AgSbF_6$, to give the corresponding zwitterionic intermediate **7**.[12] Then, the Friedel–Crafts reaction of a benzylic cation with arene **2** takes place to afford intermediate **8**, which gives intermediate **3** through an intramolecular proton transfer.[13] The gold(I)-catalyzed intramolecular nucleophilic addition of the nitrogen atom to the alkyne takes place via intermediate **9** to produce intermediate **10**, which affords the corresponding final product **4** and eliminates the gold catalyst.

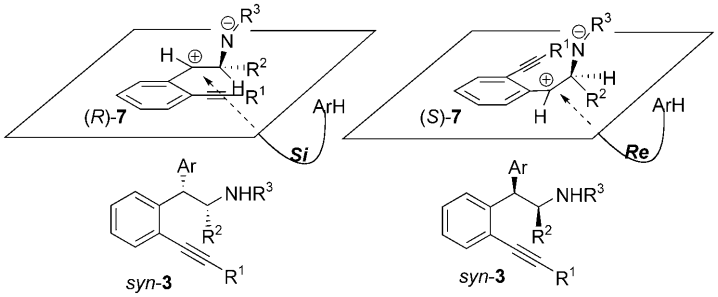
In summary, we have developed a novel gold-catalyzed domino reaction of aziridinyl alkynes with various arenes to give the corresponding 1,2,3,4-tetrahydroisoquinoline and 3,4-dihydroisoquinoline derivatives in moderate to good yields, under mild conditions, with *syn*-stereoselectivities. The reaction mechanism has been discussed and is based upon deuterium-labeling and control experiments, as well as the observed diastereoselectivities. The moderate to high diastereoselectivities can be rationalized by conformational restriction of the corresponding benzylic cation. Further investigations on this kind of domino reaction, staring from chiral aziridines or other highly strained small rings, are ongoing in our laboratory.

Table 3. The gold(I)-catalyzed reaction of aziridines **1b–l** with arenes **2e**, **a**, **f**, or **h**.[a]



Entry	**1** ($R^1/R^2/R^3$) (*trans*/*cis*)[b]	Arene	*t* [min]	Yield [%][c]	d.r[b] (*syn*/*anti*)
1	**1b** (*n*Bu/H/Ts)	**2e**	15	**4l**, 91	–
2	**1c** (*n*Bu/H/Ns)	**2e**	15	**4m**, 97	–
3	**1d** (*n*Bu/Me/Ns) (4.0:1)	**2e**	30	**4n**, 88	3.6:1
4	**1e** (Ph/H/Ts)	**2e**	60	**4o**, 71	–
5	**1f** (Ph/H/Ns)	**2e**	30	**4p**, 88	–
6	**1g** (Ph/H/Bs)	**2e**	30	**4q**, 81	–
7	**1g** (Ph/H/Bs)	**2a**	15	**4r**, 81[d]	–
8	**1h** (Ph/Me/Bs) (9.0:1)	**2e**	60	**4s**, 71	4.2:1
9	**1i** (Ph/nPr/Bs) (4.8:1)	**2e**	120	**4t**, 77	24:1
10	**1j** (Ph/*n*Bu/Bs) (6.5:1)	**2e**	120	**4u**, 82	32:1
11	**1k** (Me/Me/Ns) (4.6:1)	**2e**	150	**4v**, 78	5.6:1
12	**1l** (TMS/H/Bs)	**2e**	240	**4w**,[e] 79, R^1 = H	–
13	**1k** (Me/Me/Ns) (4.6:1)	**2f**	60	**4x**, 42, 35[f]	5.1:1
14	**1f** (Ph/H/Ns)	**2h**	30	**4y**, 85[d,f]	–

[a] All reactions were conducted with aziridine **1** (0.200 mmol), [Au(PPh$_3$)Cl]/AgSbF$_6$ (10 mol%) in mesitylene (1.0 mL) at 60°C unless otherwise specified. [b] Determined by using the ^{1}H NMR spectroscopic data. [c] Isolated yield based on aziridine **1**. [d] The product includes traces of a regioisomer. [e] H_2O (1.0 equiv) was added to the reaction system and product **4w** was 2-(4-bromophenylsulfonyl)-4-mesityl-1-methylene-1,2,3,4-tetrahydroisoquinoline. [f] The reaction was run using **2** (2.0 equiv) in DCE (1.0 mL) at 60°C.



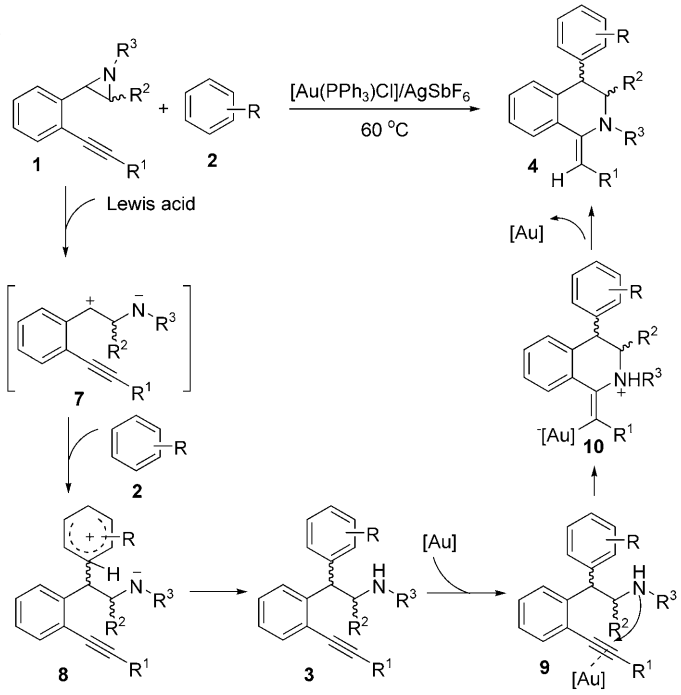
Scheme 4. The diastereoselective attack of an arene (ArH) on the zwitterionic intermediate **7**.

Experimental Section

Further experimental procedures and compound characterization data are available in the Supporting Information. CCDC-756210 (**4r**) and CCDC-763709 (**4t**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

General procedure for the gold-catalyzed domino reaction of aziridinyl alkynes: A solution of [Au(PPh$_3$)Cl] (20.0 μmol, 10.0 mg) and AgSbF$_6$ (20.0 μmol, 7.00 mg) in anhydrous toluene (**2a**; 0.50 mL) was stirred at room temperature for 10 min. Then aziridinyl alkyne **1a** (0.200 mmol, 83.7 mg) in toluene **2a** (0.50 mL) was added into the reaction mixture and stirred at 60°C for 30 min. The reaction was monitored by TLC. When the reaction was complete, the mixture was diluted with EtOAc, the solvent evaporated under reduced pressure and the residue purified by flash column chromatography (silica gel; eluent: EtOAc/petroleum ether 1:20). Compound **4a** (80.0 mg) was isolated as a colorless oil in 78% yield.

General procedure for the removal of the *para*-nitrobenzenesulfonyl group from 4: K_2CO_3 (0.570 mmol, 78.7 mg) and PhSH (0.380 mmol, 39 μL) were added to a solution of **4p** (0.190 mmol, 100 mg) in DMF (2.0 mL). The reaction mixture was stirred at room temperature for 4 h and monitored by TLC. When the reaction was complete, the mixture was quenched with saturated $NaHCO_3$ (10 mL) and extracted with EtOAc (3×10 mL). The organic phase was dried over anhydrous Na_2SO_4, the solvent evaporated under reduced pressure, and the residue purified by flash column chromatography (silica gel; EtOAc/petroleum ether 1:4). Compound **5a** (32.4 mg) was isolated as a colorless oil in 50% yield.



Scheme 5. A plausible reaction mechanism.

Acknowledgements

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Keywords: aziridinyl alkynes · benzylic cations · dihydroisoquinolines · domino reactions · gold · tetrahydroisoquinolines

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