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Gold-Catalyzed Intermolecular, anti-Markovnikov Hydroarylation of Methylenecyclopropanes with Indoles

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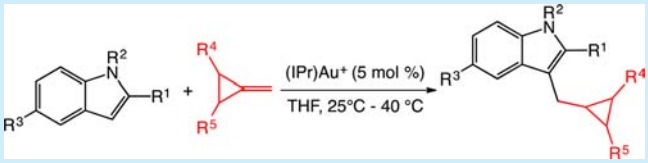
Supporting Information

ABSTRACT: Cationic gold complexes containing an *N*-heterocyclic carbene ligand catalyze the intermolecular anti-Markovnikov hydroarylation of monosubstituted and *cis*- and *trans*-disubstituted methylenecyclopropanes (MCPs) with *N*-alkyl and 1,2-dialkyl indoles to form the corresponding 3-(cyclopropylmethyl)indoles in high regio- and diastereoselectivity and in good to excellent chemical yield.



Cyclopropanes are found in a large number of naturally occurring and biologically active molecules, including existing and potential therapeutics, and find use as mechanistic probes for use in chemical biology and enzymology.[1,2] An important subset of these biologically active cyclopropanes are those containing a (cyclopropylmethyl)aryl or heteroaryl moiety.[3] In addition to classic cyclopropanation strategies, (cyclopropylmethyl)arenes have been synthesized via the catalytic cross-coupling of benzylic halides with cyclopropyl nucleophiles[4] or aryl nucleophiles with cyclopropylmethyl electrophiles.[5] However, direct substitution of an aryl C−H bond with a (cyclopropylmethyl) group has been realized in only one instance.[6]

The anti-Markovnikov hydroarylation of a methylenecyclopropane (MCP)[7] with an arene represents a particularly attractive and atom economical approach to the synthesis of (cyclopropylmethyl)arenes.[8] Although numerous transition-metal-catalyzed reactions of MCPs have been developed,[9] only a small subset of these transformations occur without concomitant rupture of the cyclopropyl ring,[10−12] and of these, only a single example involves hydroarylation.[11] Specifically, Ackermann has described the ruthenium-catalyzed hydroarylation of MCPs with 2-arylpyridines and related arenes under rather forcing conditions (120 °C, 48 h).[11] We envisioned an alternative strategy for the hydroarylation of MCPs involving π-activation of the MCP with the potential to extend the scope of MCP hydroarylation to include electron-rich arenes lacking a directing group. Herein, we report a gold(I)-catalyzed method for the intermolecular, *anti*-Markovnikov hydroarylation of MCPs with indoles and electron-rich arenes that occurs with retention of the cyclopropane ring.

Our approach to the anti-Markovnikov hydroarylation of MCPs was inspired by our recent development of the anti-Markovnikov hydroamination of MCPs with nitrogen heterocycles catalyzed by the cationic gold(I) complex [(**L1**)Au(NCMe)]$^+$ SbF_6^- (**L1** = PCy_2*o*-biphenyl).[12] An initial experiment employing this catalyst system was encouraging, and treatment of a 1:1.5 mixture of 1,2-dimethylindole (**1a**) and 7-methylenebicyclo[4.1.0]heptane (**2a**) with [(**L1**)Au(NCMe)]$^+$ SbF_6^- (5 mol %) in 1,4-dioxane at 80 °C for 16 h led to complete conversion to form a 1:1.3 mixture of the 3-cyclopropylmethylindole **3aa** as a single regio- and diastereoisomer and olefinic, ring-opened byproducts **4** (Table 1, entry 1). Lowering the reaction temperature to 40 °C led to a significant increase in selectivity for **3aa** but with a concomitant increase in reaction time (Table 1, entries 1−3). In an effort to improve catalyst activity, we screened a number of solvents and gold catalysts containing tertiary phosphine ligands, but these permutations led in all cases to erosion of selectivity for **3aa** without a significant increase in activity (Table 1, entries 4−10). Conversely, employment of the gold NHC complex (IPr)AuOTf [IPr = 1,3-bis(2,6-diisopropylphenyl-imidazol-2-ylidene)] led to a dramatic increase in activity, particularly when employed in conjunction with THF as solvent, without a decrease in the selectivity for **3aa** (Table 1, entries 11 and 12). In a preparative scale experiment, treatment of a 1:1.8 mixure of **1a** and **2a** with (IPr)AuOTf in dioxane at 40 °C for 18 h led to isolation of **3aa** in 93% yield as a single (≥25:1) isomer (Table 2).

Unfortunately, the catalyst system optimized for the hydroarylation of **2a** with **1a** proved only modestly effective for the hydroarylation of **2a** with *N*-methylindole (**1b**), forming 3-cyclopropylmethylindole **3ba** with 6:1 selectivity after 48 h at 40 °C (Table 1, entry 13). Fortunately, a second screen of catalysts and reaction conditions (Table 1, entries 14−18) identified the cationic gold benzonitrile complex [(IPr)Au(NCPh)]$^+$ SbF_6^- as an effective catalyst for the hydroarylation of **2a** with **1b** (Table 1, entry 18), and in a preparative-scale experiment, reaction of **1b** with **2a** catalyzed by [(IPr)Au(NCPh)]$^+$ SbF_6^- in THF at 25 °C for 48 h led to isolation of **3ba** in 97% yield as a single (≥25:1) regio- and diastereoisomer (Table 2).

The scope of the anti-Markovnikov hydroarylation of MCPs with indoles catalyzed by either (IPr)AuOTf or [(IPr)Au-

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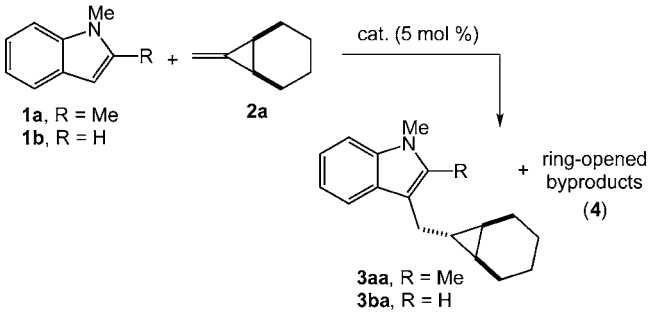
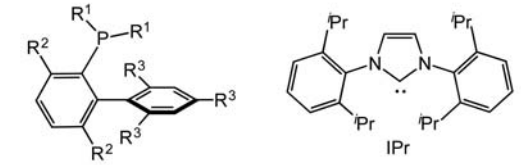
Table 1. Effect of Catalyst, Temperature, and Solvent on the Gold(I)-Catalyzed Hydroarylation of 2a with 1a and 1b[a]

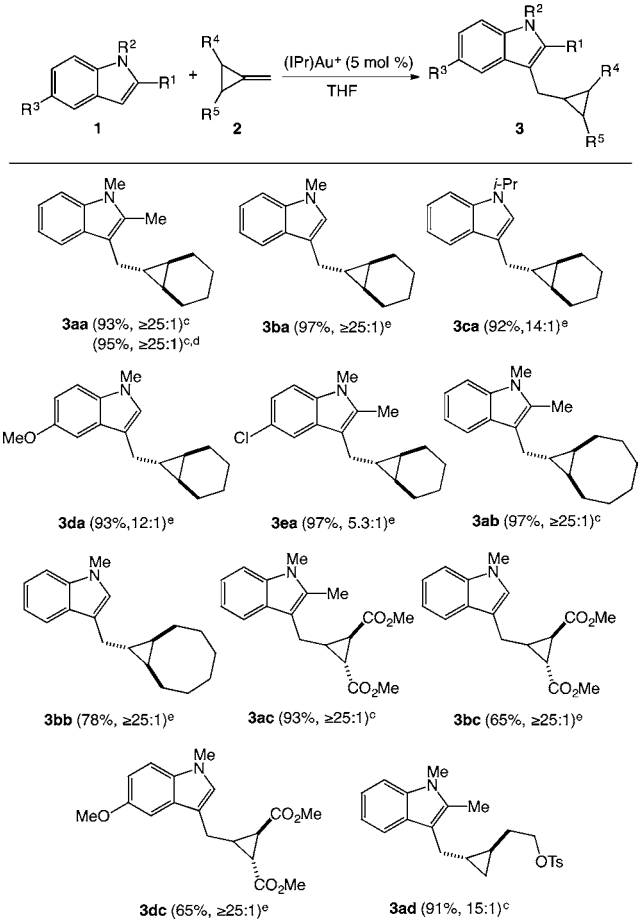


entry	indole	catalyst	solvent	temp/time (°C/h)	conv. (%)	3/4[b]
1	1a	[(L1)Au(NCMe)][SbF_6]	dioxane	80/16	>95	1:1.3
2	1a	[(L1)Au(NCMe)][SbF_6]	dioxane	60/24	>95	4.5:1
3	1a	[(L1)Au(NCMe)][SbF_6]	dioxane	40/72	>95	≥25:1
4	1a	[(L1)Au(NCMe)][SbF_6]	DCM	30/72	>95	5:1
5	1a	[(L1)Au(NCMe)][SbF_6]	PhMe	40/72	>95	6.7:1
6	1a	[(L1)Au(NCMe)][SbF_6]	THF	40/46	>95	5:1
7	1a	[(L2)Au(NCMe)][SbF_6]	dioxane	40/48	66	6:1
8	1a	[(L3)Au(NCMe)][SbF_6]	dioxane	40/72	75	1:5
9	1a	PPh_3AuNTf_2	dioxane	40/72	50	3.7:1
10	1a	$P(^tBu)_3AuNTf_2$	dioxane	40/48	>95	10:1
11	1a	(IPr)AuOTf	dioxane	35/36	>95	≥25:1
12	1a	(IPr)AuOTf	THF	40/16	>95	≥25:1
13	1b	(IPr)AuOTf	THF	40/48	>95	6:1
14	1b	(IPr)AuOTf	THF	25/48	>95	12:1
15	1b	(IPr)AuOTf	diglyme	25/48	<50	5:1
16	1b	$(IPr)AuNTf_2$	THF	25/48	<50	5:1
17	1b	[(IPr)Au(NCPh)][SbF_6]	dioxane	25/48	<50	2:1
18	1b	[(IPr)Au(NCPh)][SbF_6]	THF	25/48	>95	≥25:1



[a]Standard reaction conditions: **1a** (0.15 mmol), **2a** (0.27 mmol), cat. (7.5×10^{-3} mmol), solvent (0.75 mL) for the designated time and temperature. [b]Denotes the ratio of **3** to the sum of all ring-opened products **4** as determined by GC analysis of the crude reaction mixture.

Table 2. Scope of the Gold-Catalyzed Hydroarylation of MCPs with *N*-Alkyl Indoles



[a]Isolated yield. [b]Ratio in parentheses refers to the ratio of 3-(cyclopropylmethyl)indole to ring-opened olefinic byproducts. All 3-(cyclopropylmethyl)indoles were formed as a single (≥25:1) diastereomer. [c](IPr)AuOTf employed as catalyst at 40 °C for 18 h. [d]Reaction conditions: **1a** (2 mmol), **2a** (2.4 mmol), and (IPr)AuOTf (4 mol %) at 40 °C for 32 h. [e][(IPr)Au(NCPh)]$^+$ SbF_6^- employed as catalyst at 25 °C for 48 h.

(NCPh)]$^+$ SbF_6^- is summarized in Table 2. With respect to the MCP, gold(I)-catalyzed hydroarylation was effective for *cis*- and *trans*-1,2-disubstituted and mono(alkyl)substituted MCPs (Table 2). With respect to the indole component, hydroarylation was effective with 1-alkyl- and 1,2-dimethylindoles and tolerated heteroatom substitution at the 5 position of the indole (Table 2).[13] Gold-catalyzed anti-Markovnikov MCP hydroarylation was scalable employing only 1.2 equiv of the MCP component. Specifically, gold-catalyzed reaction of **1a** (2 mmol) and **2a** (2.4 mmol) at 40 °C for 32 h led to isolation of **3aa** in 95% yield as a single regio- and diastereomer (Table 2).

Gold-catalyzed MCP hydroarylation was also effective for 1,2,5-trimethylpyrrole (**5**) and azulene (**6**), although selective monoalkylation of the arene was not realized. Rather, treatment of **5** or **6** with excess MCP (2.5 equiv) and a catalytic amount of (IPr)AuOTf led to isolation of the corresponding bis(alkylated) arenes **7** and **8**, respectively, in good yield as a mixture of diastereomers (Scheme 1). Hashmi has similarly documented the strong tendency toward bis(alkylation) in the gold(III)-catalyzed hydroarylation of methylvinylketone with pyrroles.[14]

The high *trans*-selectivity observed for the hydroarylation of *cis*-1,2-disubstituted and monosubstituted MCPs was likewise observed for the gold-catalyzed hydroamination of *cis*-1,2-disubstituted and monosubstituted MCPs with nitrogen heterocycles and is likewise akin to the selective formation of *E*-alkenes in the gold-catalyzed hydroarylation of 1,3-disubstituted and 1,1,3-trisubstituted allenes with electron-rich arenes.[12,15] This outcome is consistent with a mechanism involving outer-sphere addition of the arene to less stable cis π-MCP complex *cis*-**I** followed by protodeauration (Scheme 2).

In summary, we have developed a gold(I)-catalyzed method for the anti-Markovnikov hydroarylation of methylenecyclopropanes (MCPs) with substituted indoles to form 3-(cyclopropylmethyl)indoles in good yield, with excellent

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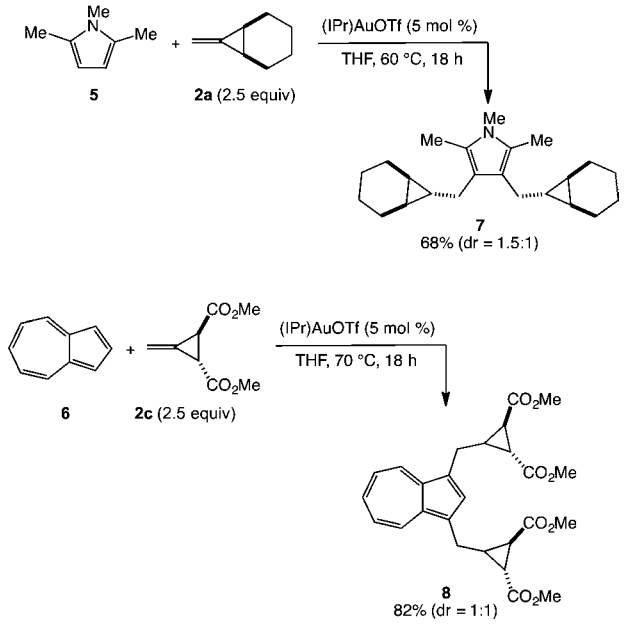
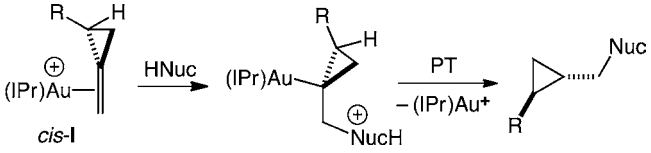
Scheme 1



Scheme 2



regio- and diastereoselectivity, and with preservation of the cyclopropyl functionality.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.6b02431.

Experimental details and characterization data and scans of ^{1}H and ^{13}C NMR spectra for new compounds (PDF)

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Notes

The authors declare no competing financial interest.

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(13) The reaction of 1-benzylindole with **2a** was sluggish and gave poor selectivity for the anti-Markovnikov hydroarylation product while reaction of 5-chloro-1-methylindole with **2a** gave only partial conversion to a complex mixture of products. Additionally, 1,3,5-trimethoxybenzene and 1-methylpyrrole were ineffective for the gold-catalyzed hydroarylation of **2a**.
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