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Diastereoselective Synthesis of Protected 1,3-Diols by Catalytic Diol Relocation

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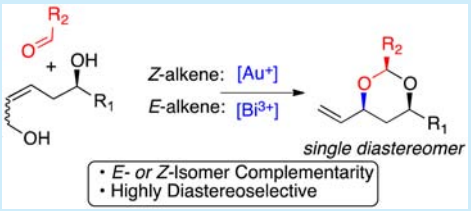
Supporting Information

ABSTRACT: A complementary diastereoselective gold(I) or bismuth(III) catalyzed tandem hemiacetalization/dehydrative cyclization of 1,5-monoallylic diols was developed to access 1,3-dioxolanes and dioxanes. This methodology provides rapid access to protected 1,3-diols under mild conditions with high levels of diastereoselectivity.



The synthesis of 1,3-diols has garnered significant interest from the synthetic community due to the abundance of this motif in natural products[1] as well as their discernible presence in pharmaceutically relevant compounds.[2] In addition to direct incorporation into these molecules, when the hydroxyl groups are used in concert with accompanying functional groups, these compounds are valuable intermediates that can easily be elaborated into more complex architectures such as 3-hydroxy-tetrahydrofurans, which are found in a variety of natural products and bioactive molecules (Figure 1).[3] Many synthetic strategies

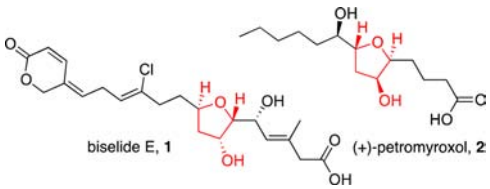
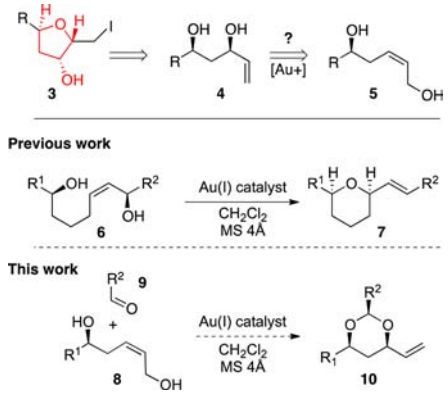


Figure 1. Examples of *syn* 1,3-diol derivatives in natural products.

have been developed to prepare 1,3-diols;[4] however, access to the tetrahydrofurans exemplified in **1** and **2** require an allylic alcohol-containing *syn* 1,3-diol (e.g., **4**) for ring formation that proceeds through iodide **3** by iodoetherification. Transition-metal-catalyzed allylic substitution is a powerful tool for allylic bond formation.[5] In this vein, several nice examples of Pd-catalyzed allylic substitution reactions for the formation of protected allylic 1,3-diols and amino alcohols have recently been reported.[6] These protocols demonstrate the desired reactivity under mild conditions, but the diastereoselectivities are moderate (generally less than 10:1). Zakarian and co-workers overcame the selectivity issue using an exceptional rhenium-catalyzed allylic transposition strategy.[7] To achieve this, their system effects allylic alcohol equilibration and trapping of the thermodynamic *syn* 1,3-diol by transacetalization with $PhCH(OMe)_2$. Related work in this area relies on hemiacetal formation followed by conjugate addition to an electron-deficient olefin.[8] We envisioned that the ideas of olefin addition and allylic transposition could be wed using Au-catalysis[9] to effect substitution of an allylic alcohol (*vide infra*) and provide a complementary approach to the formation of these useful compounds.

In 2008 we reported the gold-catalyzed cyclization of monoallylic diols (**6** → **7**, Scheme 1) and initiated a program

Scheme 1. Au-Catalyzed Acetal Formation Hypothesis



to understand the mechanistic intricacies of this transformation and explore its synthetic utility.[10,11] As a part of this program, we envisioned a strategy for the formation of *syn* 1,3-diols that would take advantage of a transiently tethered nucleophile. In this sequence, reaction of a 1,5-diol **8** with an aldehyde would form the hemiacetal[8] and enable the now pendent nucleophile to undergo a gold-catalyzed dehydrative cyclization to afford protected *syn* 1,3-diols **10** (Scheme 1). Careful selection of catalyst/conditions would be vital as many potential unproductive side reactions could be conceived. Herein we report mild conditions that provide protected 1,3-diols from1,5-diols in high yield with high levels of diastereoselectivity.

Initial experiments were designed to determine the identity of a suitable aldehyde for this transformation with *cis*-1,4-

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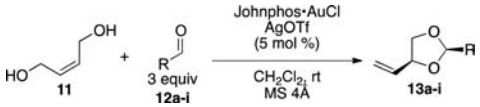
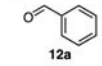
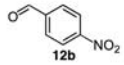
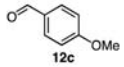
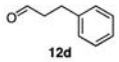
butenediol **11**. This diol is known to undergo direct acetal formation under a variety of protic and Lewis acid conditions[12] and would immediately alert us to this potential problem. By employing JohnPhos·AuCl **I**, a series of aldehydes were screened (Table 1). Gratifyingly, with benzaldehyde (entry 1) a moderate

Table 1. Aldehyde Scope Studies



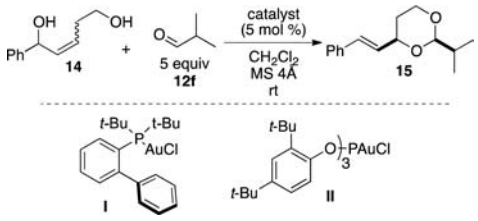
entry	substrate	yield[a]	dr[b]
1	 12a	55[c]	2:1
2	 12b	trace	-
3	 12c	n.r.	-
4	 12d	68[c]	5:1
5	 12e	81[c]	3:1
6	 12f	80	8:1
7	 12g	93[c]	8:1
8	 12h	70	18:1
9	 12i	98	3:2

[a]Isolated yield. [b]Determined by ^{1}H NMR. [c]Purified by reduction of excess aldehyde using $NaBH_4$.

yield of the desired protected 1,2-diol **13a** was observed, albeit with low diastereoselectivity. Although other benzaldehydes performed poorly (entries 2, 3), aliphatic aldehydes (entries 4–8) greatly increased the yield, with α-substituted aldehydes providing increased levels of selectivity (cf. **12h**). Chloral hydrate, **12i**, provided almost quantitative yield of the product, albeit with poor selectivity. With isobutyraldehyde **12f**, a good balance of yield and selectivity was attained. This, coupled with facile removal during purification by evaporation, led to the selection of **12f** as the aldehyde of choice at this stage.

Having established that the desired reactivity could be achieved, the catalyst system was next optimized using **14** as a test substrate (Table 2). Employing Au-catalyst **I** and AgOTf (entry 1) gave the corresponding 1,3-dioxane **15** in moderate yield with good selectivity. The use of a Brønsted acid additive or elevated temperature (entries 2 and 3) did not improve yield or selectivity. However, increasing the concentration had a positive effect on the reactivity, especially when coupled with increased temperature (entry 6), affording the desired product in 85% yield in only 4 h. This suggested that hemiacetal formation may be sluggish with these mildly oxophilic catalysts. A more electron deficient Au-complex may increase the overall reaction rate by slightly increasing the Lewis acidity of the catalyst, thereby facilitating the initial hemiacetalization while retaining the

Table 2. Catalyst Optimization



entry	E/Z	cat.	concn (M)	time (h)	yield (%)[a]	dr[b]
1	Z	**I**/AgOTf	0.2	20	40	23:1
2[c]	Z	**I**/AgOTf	0.2	3.5	36	1:1
3[d]	Z	**I**/AgOTf	0.2	5	40	15:1
4[d]	Z	**I**/AgOTf	0.4	6.5	59	2:1
5	Z	**I**/AgOTf	0.8	20	42	>25:1
6[d]	Z	**I**/AgOTf	0.8	4	85	6:1
7	Z	**II**/$AgSbF_6$	0.8	4.5	91	22:1
8	E	**II**/$AgSbF_6$	0.8	23	83	7:1
9	E	$Bi(OTf)_3$	0.2	0.5	98	>25:1

[a]Isolated yield. [b]Determined by ^{1}H NMR. [c]5 mol % pTSA was added to the reaction. [d]Reaction temperature = 40 °C.

necessary π-acidity required for dehydrative cyclization.[13] Using phosphite Au-complex **II**, **15** was isolated in 91% yield after 4.5 h at rt with excellent selectivity (22:1 dr, entry 7). Unfortunately, under the same conditions *E*-olefins were not suitable, requiring a much longer reaction time and affording the products with low diastereomeric ratio (entry 8). After extensive screening, it was found that *E*-alkenes were viable substrates for the desired transformation when $Bi(OTf)_3$ was used as the catalyst (entry 9).[14] Interestingly, when *Z*-olefins are subjected to the $Bi(OTf)_3$ conditions only direct acetal formation is observed,[12b] demonstrating substrate-based catalyst complementarity.

With conditions established for substrates of either olefin geometry, the reaction scope was explored (Table 3). In general, the acetals **15a–h** were formed in high yields and selectivities from the corresponding monoallylic diols **14a–h**. Phenyl substituted diols **14a,h** were good substrates for the reaction and gave **15a** and **15h**, respectively, in excellent yield. The electronic nature of the aromatic substituent proved to be important to the transformation. When an electron-donating group was added to the aromatic ring, the reaction pathway was shut down and diol **14b** afforded no desired product. Introduction of an electron-withdrawing group improved the yield, as diol **14c** furnished **15c** in almost quantitative yield. Aliphatic substituents gave excellent yields and selectivities as well, although branching in the aliphatic chain of **14e** led to a lower diastereomeric ratio for the corresponding acetal **15e** (entry 9). Nitrogen-containing substrates were also tolerated as diol **14f** gave **15f** in high yield. 1,3-Dioxolanes were readily formed under Au-catalysis conditions (entries 12, 14), but unfortunately, when the corresponding *E*-olefins **14g** and **14h** were allowed to react under the Bi-catalyzed conditions, **15g** and **15h** were formed in only trace amounts (entries 13, 15).

For the preparation of protected allylic *syn* 1,3-diols **17**, 1,5-diols **16a–f**, containing a primary allylic alcohol leaving group, were prepared. With this substitution pattern, using isobutyraldehyde provided only trace amounts of product, but chloral hydrate **12i** proved to be very effective, exhibiting excellent yield and diastereoselectivity (Table 4). In all cases, only a single diastereomer of the product was observed. Both aliphatic and

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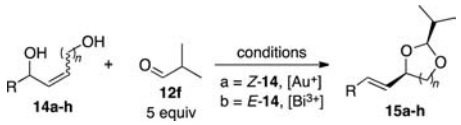
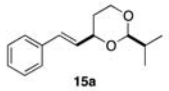
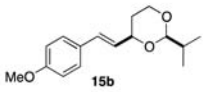
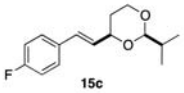
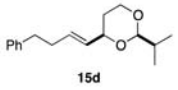
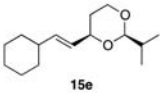
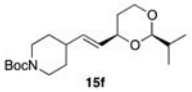
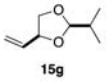
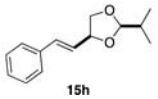
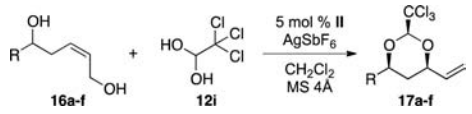
Table 3. Comparison of *E*- and *Z*-Substrates

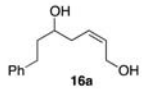
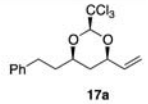
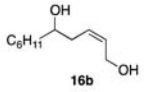
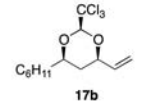
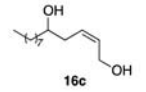
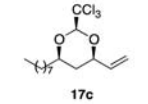
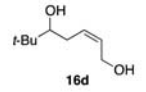
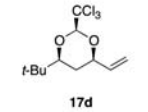
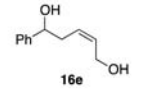
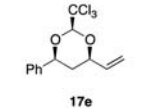
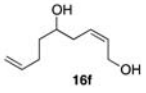
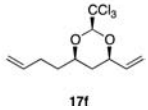


entry	conditions[a]	yield (%)[b]	dr[c]	product
1	a	91	22:1	 15a
2	b	98	>25:1	
3	a	nr	-	 15b
4	b	nr	-	
5	a	98	>25:1	 15c
6	b	80	>25:1	
7	a	83	>25:1	 15d
8	b	95	>25:1	
9	a	81	11:1	 15e
10	b	86	>25:1	
11	a	80	>25:1	 15f
12	a	98	5:1	 15g
13	b	trace	-	
14	a	87	15:1	 15h
15	b	trace	-	

[a]Conditions a: *Z*-**14** = 5 mol % **II**, 5 mol % $AgSbF_6$, CH_2Cl_2 (0.8 M), MS 4 Å, rt; Conditions b: *E*-**14** = 5 mol % $Bi(OTf)_3$ (0.2 M) CH_2Cl_2, MS 4 Å, rt. [b]Isolated yield. [c]Determined by ^1H NMR.

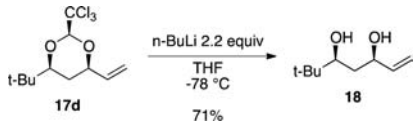
Table 4. Relocation of 1,5-Allylic Diols



entry[a]	substrate	yield (%)[b]	dr[c]	product
1	 16a	87	>25:1	 17a
2	 16b	89	>25:1	 17b
3	 16c	82	>25:1	 17c
4	 16d	83	>25:1	 17d
5	 16e	55	>25:1	 17e
6	 16f	40	>25:1	 17f

[a]Conditions a: 5 mol % **II**, 5 mol % $AgSbF_6$, CH_2Cl_2 (0.8 M), 1 equiv of **12i**, MS 4 Å, rt. [b]Isolated yield. [c]Determined by ^1H NMR.

Scheme 2. Deprotection of Trichloromethyl Acetals

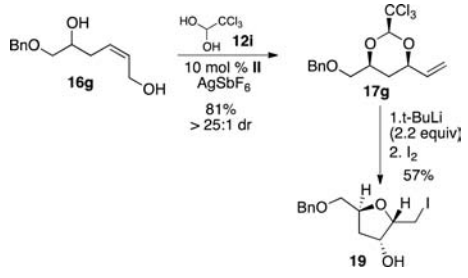


aromatic substituents were well tolerated, although the presence of an additional alkene in the substrate led to reduced yield. The relative stereochemistry of the protected *syn* 1,3-diols was assigned by ^1H NMR analysis in **17a** where NOE enhancement of the methine proton signal was observed upon irradiation of the acetal proton.[15] The stereochemistry of the remaining compounds was assigned by analogy.

To utilize this method, the free 1,3-diols would need to be revealed. Although several sets of reductive/hydrolytic conditions for the deprotection of trichloromethyl acetals have been reported,[16] we set out to investigate whether this group could be cleaved simply by lithiation followed by an aqueous quench, which we felt could provide a useful alternative in certain settings (Scheme 2). Gratifyingly, when acetal **17d** was treated with 2.2 equiv of *n*-butyllithium at low temperatures, the corresponding diol **18** was isolated in 71% yield.

To further demonstrate the synthetic utility of this transformation we envisioned a three-step synthetic sequence to generate tetrahydrofuran **19**. As shown above, this tetrahydrofuran motif bearing a 2,5-*trans* relationship is found in natural products. Additionally, a multitude of further manipulations to the free alcohol, the Bn-protected alcohol, or the alkyl iodide could be used for further functionalization if this sequence could be realized. In the event, Au-catalyzed cyclization of diol **16g** afforded the protected *syn* 1,3-diol **17g** in good yield as a single diastereomer (Scheme 3). Deprotection followed by iodoetherification of the resulting diol[17] then successfully afforded **19** in a straightforward manner. Further deployment of this reaction sequence is underway in our laboratories and will be reported in due course.

Scheme 3. Transformation of *syn* 1,3-Diols to 2,5-*trans*-Tetrahydrofurans



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In summary, a novel hemiacetalization/dehydrative cyclization sequence employing monoallylic diols has been developed. This transformation allows for the highly efficient formation of 1,3-dioxolanes and dioxanes with excellent levels of diastereoselectivity. Although Au-catalysis conditions do not work for *E*-monoallylic diols, a set of complementary Bi-catalyzed conditions has been demonstrated. Employing monoallylic diols **16** allows for the formation of protected 1,3-diols products bearing a terminal alkene, enabling a multitude of further transformations. Furthermore, deprotection of the acetal products provides facile access to the corresponding *syn* 1,3-diols under mild conditions en route to 2,5-*trans*-tetrahydrofurans.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.5b02725.

Experimental procedures and spectral data for all new compounds (PDF)

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Author Contributions

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Notes

The authors declare no competing financial interest.

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