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Diastereoselective Diboration of Cyclic Alkenes: Application to the Synthesis of Aristeromycin

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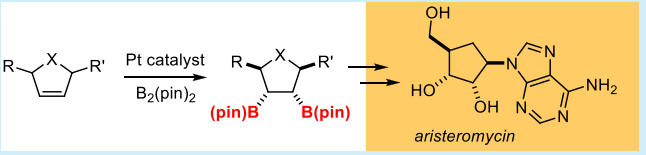
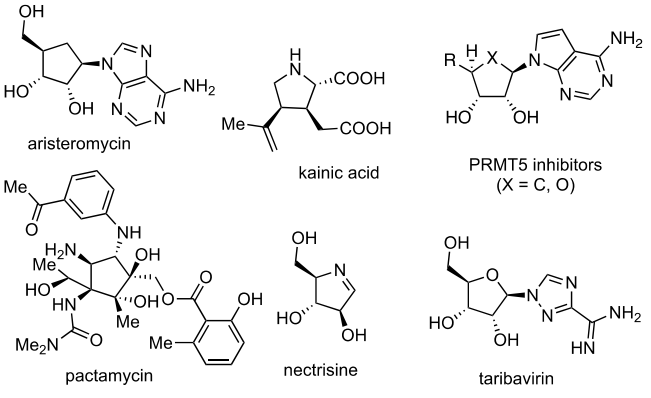
ABSTRACT: The Pt-catalyzed diboration of cyclic alkenes is extended to unsaturated heterocycles and bicyclic compounds and can be accomplished in a diastereoselective fashion. The optimal procedures, substrate scope, and diastereoselectivity were investigated, and examples employing both homogeneous and heterogeneous catalysis were examined. Lastly, application to the construction of the nucleoside analog (±)-aristeromycin was conducted.



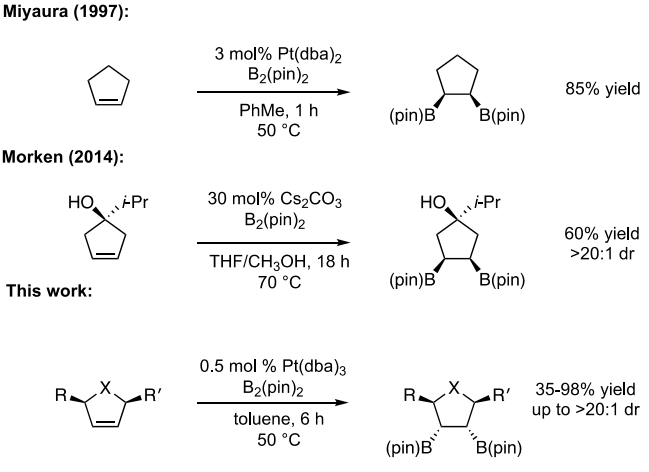
The diboration of alkenes is a synthetically useful strategy for the preparation of important building blocks.[1] Realizing this potential, a number of research groups have advanced enantioselective diboration through the use of both transition-metal catalysis and organocatalysis.[2,3] The investigation of chiral ligands,[4] alkoxides,[5] and diboron reagents[6] has rendered the diboration of terminal alkenes and, to a lesser extent, internal alkenes enantioselective. Two classes of alkenes that have been less explored as substrates for diboration are unsaturated heterocycles and compounds with bicyclic frameworks. Diboration of these compounds would allow for the facile, stereoselective synthesis of a variety of compounds such as nucleoside analogs, a prominent component of many anticancer and antiviral treatment plans,[7] and cyclopentane-, tetrahydrofuran-, and pyrrolidine-containing natural products (Scheme 1).[8] To our knowledge, the diboration of monounsaturated heterocyclic compounds has not been studied, and only a few examples exist of the stereocontrolled diboration of cyclic olefin-containing substrates.[9] In this Letter, we describe the diboration of this class of substrates and examine the utility of this process in chemical synthesis.

Scheme 1. Selected Bioactive Cyclopentane-, Pyrrolidine-, and Tetrahydrofuran-containing Molecules



To enhance the scope and synthetic utility of the diboration of cyclic alkenes without an actively participating directing group, we turned toward a platinum-catalyzed method, envisioning that the previous work of Miyaura and coworkers[2a] could be utilized with unsaturated heterocycles (Scheme 2). We also anticipated that a bis(boryl)platinum intermediate

Scheme 2. Catalytic Diboration of Cyclic Alkenes



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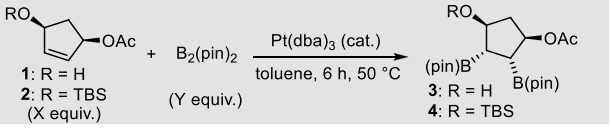
would avoid coordination to the more sterically hindered face of a substituted cycloalkene, allowing for the stereoselective synthesis by syn addition of the diboron reagent to the alkene. In this study, we accomplish this with the use of catalytic $Pt(dba)_3$, incorporating substituted cyclopentane motifs and both substituted and unsubstituted heterocycles into the Pt-catalyzed diboration substrate scope.

Initial studies employed the conditions originally established by Miyaura and coworkers[2a] and focused on diboration of a scaffold derived from *cis*-4-acetoxy-2-cyclopenten-1-ol (**1**) to give **3**, a derivative of a known building block for the synthesis of carbocyclic nucleosides and prostaglandins (Table 1).[10]

Table 1. Impact of Reaction Conditions on the Diboration of Functionalized Cyclopentenes



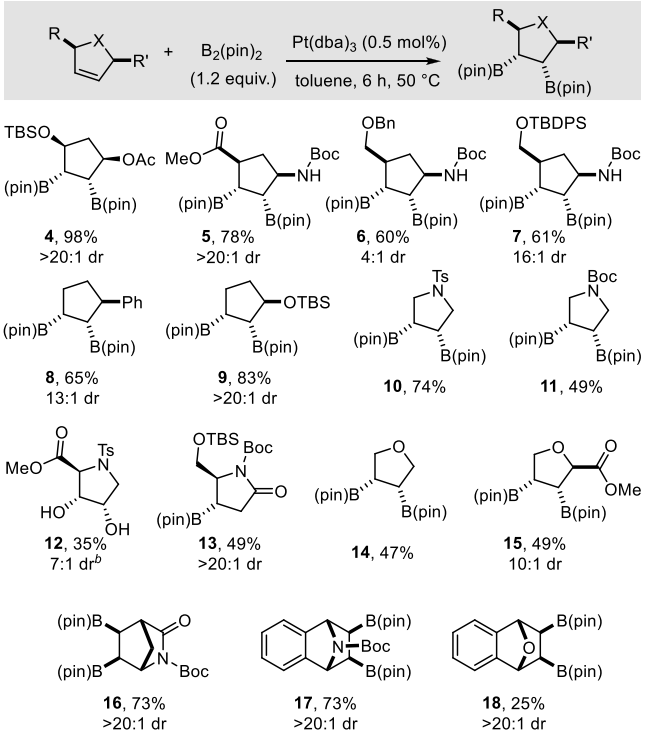
entry	R	equiv X	equiv Y	$Pt(dba)_3$ (%)	% yield
1	H	1.5	1.0	3	<15
2	TBS	1.5	1.0	3	88
3	TBS	1.2	1.0	0.5	82
4	TBS	1.0	1.2	0.5	95
5[a]	TBS	1.0	1.2	0.5	99
6[b]	TBS	1.0	1.2	0.5	98

[a]Reaction conducted in open atmosphere for 18 h. [b]Reaction conducted in open atmosphere for 6 h.

Whereas initial experiments employing the unprotected alcohol **1** resulted in low conversion (entry 1), masking of the hydroxyl as a silyl ether (**2**) allowed for a successful reaction. With a 3 mol % loading of $Pt(dba)_3$ and bis(pinacolato)diboron as the limiting reagent, an 88% isolated yield of the diboration product **4** was obtained, and ^1H NMR analysis revealed the product to arise by a >95% trans addition of the two boron groups relative to the pre-existing substituents (entry 2). It was also found that the catalyst loading could be lowered to 0.5 mol % with minimal reduction in yield (entry 3). When the reaction was conducted with bis(pinacolato)diboron in excess relative to the alkene substrates, the reaction yield was increased, thereby allowing for the more efficient transformation of the precious alkene substrate (entry 4). Shortening the reaction time to 6 h and conducting the reaction without an inert argon atmosphere (entries 5 and 6, under air) were shown to have a minimal impact on the isolated yield, allowing for a glovebox-free operation.

When the conditions established in Table 1 were applied to a range of alkenes, it was found that a variety of different structures could undergo diboration efficiently and with good selectivity. As depicted in Scheme 3, the reaction could accommodate a variety of five-membered cyclic and bicyclic compounds. For cyclopentene-derived compounds, tetrasubstituted diboration products were formed with yields ranging from 60 to 98% and include functional groups such as silyl ether and acetoxy groups (**4**), esters (**5**, **12**, **15**), a protected hydroxymethyl group (**6**, **7**), and a carbamate. Whereas product **6** was formed with reduced stereoselectivity (4:1 dr) compared with the others, it was discovered that the stereoselectivity in the production of this scaffold could be

Scheme 3. $Pt(dba)_3$-Catalyzed Diboration of Cyclic Alkenes[a]



[a]Yields refer to isolated yield of purified material and are an average of two experiments. Diastereomer ratios were determined by analysis of the crude NMR spectra. [b]This product was oxidized prior to purification because the alkene and the diboron product coelute.

enhanced by replacement of the benzyl ether with a more sterically incumbered silyl ether (**7**). In a further examination of this catalytic system, diboration of unsubstituted, mono- and disubstituted 3-pyrroline derivatives could be accomplished (**10**–**13**) in yields of 35–74% with diastereoselectivity of up to >20:1. Notably, product **13**, originating from an α,β-unsaturated lactam, was formed exclusively as the mono-boronic ester, with the α-boronic ester presumably undergoing protodeborylation during workup or isolation. 2,5-Dihydrofuran substrates were also acceptable reaction partners. Compound **15** containing an ester substituent was formed in useful yield with high diastereoselectivity. These conditions could also be applied to bicyclic substrates, delivering diboron **16** from Vince lactam by diboration of the less hindered face of the bicycle.[11] Bicyclic structures containing nitrogen (**17**) and oxygen (**18**) heteroatoms could also be prepared with high diastereoselectivity, although the yield for **18** was lowered due to the competitive formation of naphthalene. So far, diboration of cyclic enol ethers (i.e., 2,3-dihydrofuran) has been ineffective.

Aspects of the diboration that pertain to practical preparative synthesis utility were examined. First, an experiment was conducted on the multigram scale, with all reagents handled in an open atmosphere and without taking precautions to exclude air and moisture during the course of the reaction. As depicted in Scheme 4a, under these conditions, compound **7** could be delivered in 74% yield on a scale that furnished 2.17 g of the diboration product. In a second set of experiments (Scheme 4b), we probed the utility of a heterogeneous Pt catalyst for the diboration reaction. These catalysts may be recoverable, and

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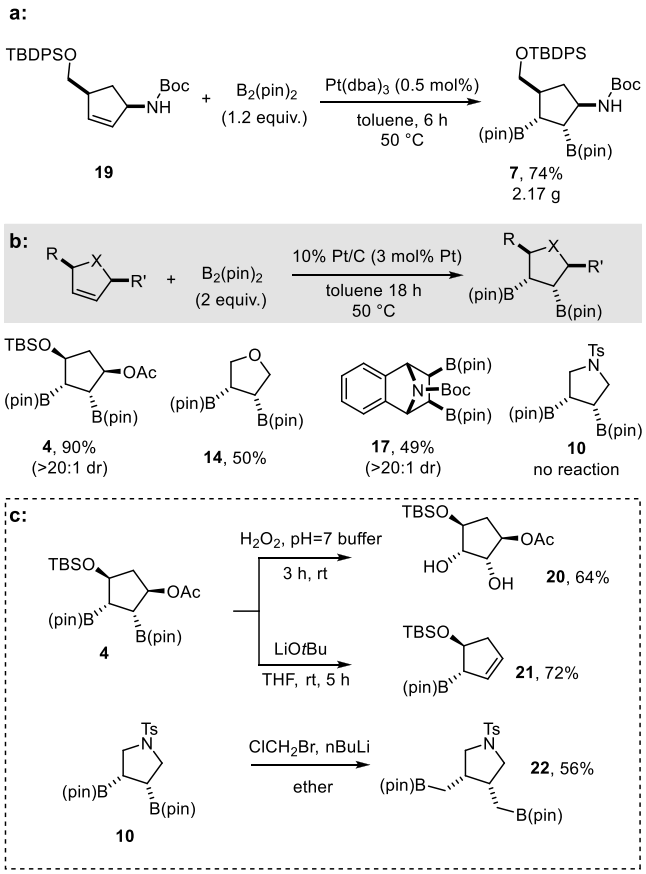
Scheme 4. Practical Aspects of Diastereoselective Alkene Diboration

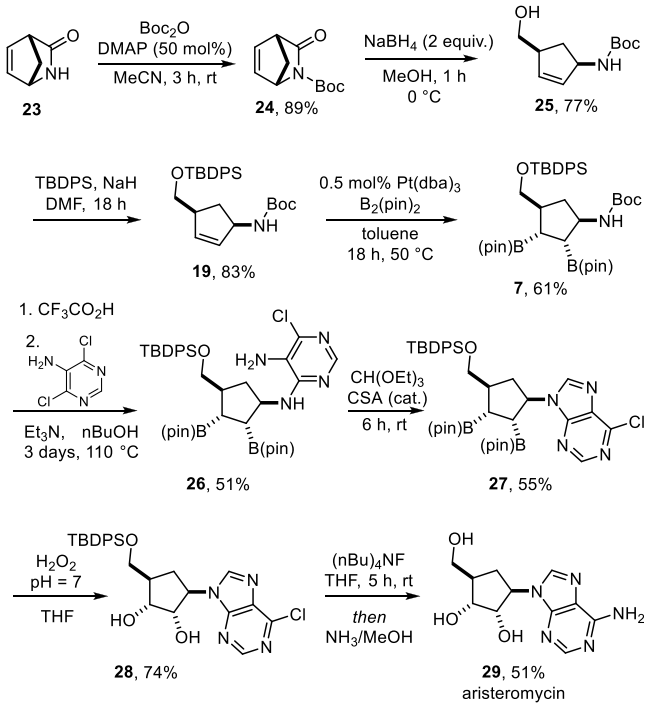


their use allows a simpler purification of the reaction product.[12] With substituted cyclopentene **2** as the substrate, it was found that 10% Pt/C was an effective catalyst. With 3 mol % overall platinum loading, diboron **4** was produced in 90% yield. Whereas tetrahydrofuran (**14**) and bicyclic substrates (**17**) proceeded with similar or slightly decreased yields, reactions involving 3-pyrroline scaffold (**10**) proved to be unsuccessful. Whereas this is not the first diboration of cyclic alkenes under heterogeneous conditions,[13] it is the first stereoselective diboration of its kind. Lastly, as might be expected, the diboron compounds undergo oxidation in the presence of hydrogen peroxide (**20**, Scheme 4c), but with a sufficiently labile leaving group β relative to the boronic ester, the substrate can be prompted to undergo elimination and provide useful new building blocks (i.e., **21**, Scheme 4c). Lastly, subjection of the intermediate to chloromethyllithium provides homologation product **22** in good yield.

To examine the utility of the cyclic alkene diboration products in chemical synthesis, attention was directed toward the synthesis of pharmacologically active nucleoside analogs, in particular, the compound aristeromycin, a known analogue of adenosine.[14] Aristeromycin is naturally occurring and exhibits similar biological properties to adenosine, but as a carbocyclic nucleoside analog, it has increased stability to hydrolysis.[15] A method frequently employed to install the cis diol of aristeromycin is catalytic dihydroxylation, a process that employs Os complexes that can pose a challenge when used on the large scale due to toxicological concerns.[16] We envisioned that replacing dihydroxylation with catalytic diboration followed by oxidation would avoid the use of osmium tetroxide.

As depicted in Scheme 5, Vince lactam (**23**) was protected with a Boc group (**24**), then subjected to reductive ring

Scheme 5. Application of Diboration to the Construction of Aristeromycin



opening and the primary alcohol protected as a TBDPS ether (**19**). Diboration furnished the intermediate **7** that was previously described. This compound was then subjected to TFA-promoted amine deprotection, followed by nucleophilic aromatic substitution with 4,6-dichloropyrimidin-5-amine to afford compound **26** in 51% yield (Scheme 5). Acid-promoted condensation provided **27** in 55% yield. Oxidation of the bis(boronic) ester moiety installed the syn-diol motif **28** in 74% yield. A final one-pot deprotection and aminolysis revealed aristeromycin (**29**) in 51% yield, concluding an eight-step process with 3.7% yield and by a sequence completed without the need for rigorous nitrogen inerting.

In conclusion, the platinum-catalyzed diboration of alkenes can be employed to synthesize a variety of bis(boronic) esters containing carbocyclic, heterocyclic, and bicyclic motifs. This reaction was also feasible in larger scale processes and with the use of a heterogeneous catalyst. Further functionalization could be employed to synthesize nucleoside analogs. We anticipate this reaction to be a greener and safer alternative to current processes toward the synthesis of these targets of interest.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acs.orglett.1c00353.

Procedures, characterization, and crystallographic (compound 16) and spectral data (PDF)

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Accession Codes

CCDC 2068901 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.

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Notes

The authors declare the following competing financial interest(s): C.A., A.R.D., and R.A.S. are employees and stockholders of Pfizer, Inc.

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