Organic LETTERS

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Hydrogen-Bond Directed Regioselective Pd-Catalyzed Asymmetric Allylic Alkylation: The Construction of Chiral α-Amino Acids with Vicinal Tertiary and Quaternary Stereocenters

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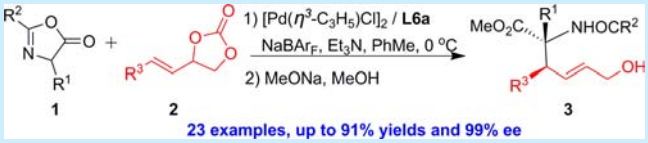
Supporting Information

ABSTRACT: A Pd-catalyzed asymmetric allylic alkylation of azlactones with 4-arylvinyl-1,3-dioxolan-2-ones was developed, providing "branched" chiral α-amino acids with vicinal tertiary and quaternary stereocenters, in high yields and with excellent selectivities. Mechanistic studies revealed that the formation of a hydrogen bond between the Pd-allylic complex and azlactone isomer is responsible for the excellent regioselectivities. This asymmetric alkylation can be carried out on a gram scale without a loss of catalytic efficiency, and the resulting product can be further transformed to a chiral azetidine in two simple steps.

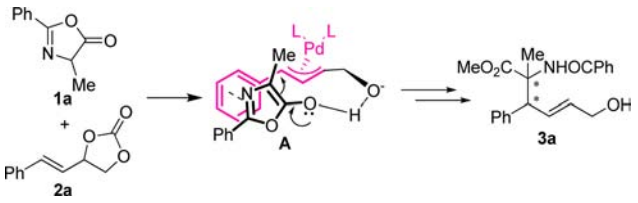


Stereochemically rigid quaternary α-amino acids are essential building blocks for the construction of unnatural peptides and proteins with improved and unusual biological properties, in that their rigid structure increases the stability of proteins and restricts their conformational flexibility.[1] They can also serve as novel drugs and are key intermediates for drug synthesis.[2] Approaches toward the synthesis of quaternary α-amino acids have therefore attracted much attention.[3] However, the synthesis of sterically demanding quaternary α-amino acids bearing vicinal tertiary and quaternary stereocenters remains a neglected area of research.[3]

Due to their convenient synthesis, azlactones have proven to be valuable synthons for the synthesis of stereochemically rigid unnatural amino acids.[4,5] Among them, strategies employing the asymmetric allylic alkylation of azlactones have received less attention than others,[5] although metal-catalyzed allylic alkylations have become powerful and versatile synthetic methods in organic synthesis.[6] Trost reported Pd-catalyzed allylic alkylations of a variety of azlactones with allyl acetates to afford linear products with excellent diastereo- and/or enantioselectivities.[5b] Recently, Jiang reported a Brønsted acid accelerated Pd-catalyzed asymmetric allylic alkylation of azlactones with simple allylic alcohols, providing linear alkylated products in excellent yields and with good enantioselectivities.[5e] However, in the case of the above-mentioned procedures, only linear alkylated products can be obtained when unsymmetrical acyclic allyl substrates are used. Although Mo- and Ir-catalyzed asymmetric allylic alkylations of azlactones were developed by Trost[5c] and Hartwig,[5d] respectively, to give branched products with high regio-, diastereo-, and enantioselectivities, a Pd-catalyzed asymmetric allylic alkylation of azlactones for the preparation of stereochemically rigid branched quaternary products is yet to be realized.[7]

We previously developed a Pd-catalyzed asymmetric allylic amination with a novel allylic substrate, 4-arylvinyl-1,3-dioxolan-2-one, using potassium phthalimide as a nucleophile.[8] The "linear" amination products ("linear" and "branched" products are defined as the nucleophilic group being positioned away from and adjacent to the aromatic ring, respectively) could be obtained exclusively with excellent catalytic results. During our research concerning the effect of hydrogen bonds on allylic substitutions,[9] we envisaged that the allylic alkylation of azlactones could be manipulated to give "branched" unnatural amino acids bearing vicinal tertiary and quaternary stereocenters. Hydrogen-bond formation between the Pd-allylic complex and azlactone isomer could be used to position the substrate for the desired alkylation (Scheme 1). Additionally, a possible π−π interaction between

Scheme 1. Synthesis of "Branched" Amino Acids



the phenyl ring of the Pd-allyl complex and the unsaturated bond/phenyl ring of the azlactone may also contribute to the regioselectivity. The resulting products bearing an additional hydroxyl group will allow for further manipulation.

Thus, the Pd-catalyzed asymmetric allylic alkylation of azlactones (**1a**) with 4-styryl-1,3-dioxolan-2-one (**2a**) was investigated using a catalyst system consisting of $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$ with **L1**−**6** (Figure 1). Planar chiral metallenyl P,N-ligands were first used, which have proven to be efficient ligands in several Pd-catalyzed asymmetric substitution reactions.[10] The

Received: October 3, 2015
Published: November 17, 2015

DOI: 10.1021/acs.orglett.5b02868
Org. Lett. 2015, 17, 5768−5771

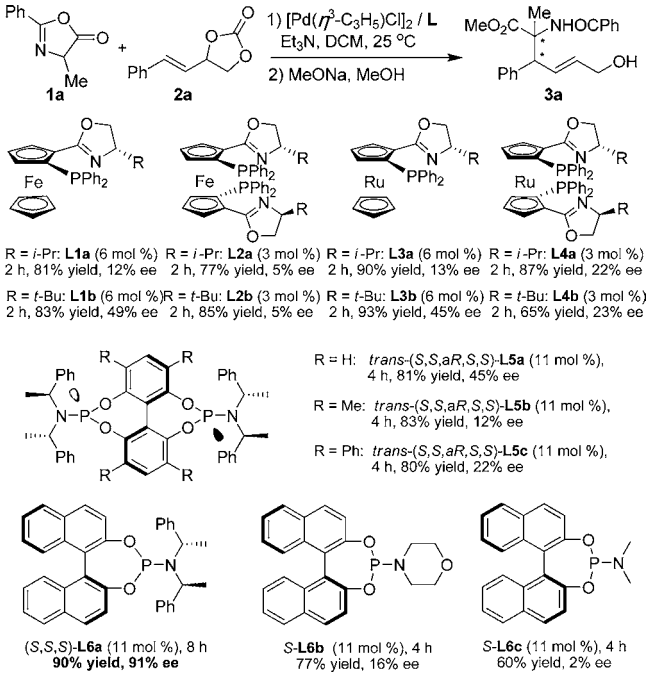


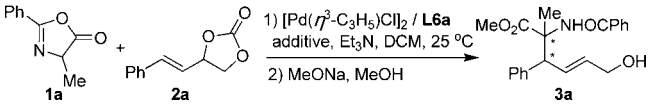
Figure 1. Ligand screening. Reactions of **1a** (0.12 mmol) and **2a** (0.10 mmol) were catalyzed by a catalytic system of $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$ (2.5 mol %) and **L** (3−11 mol %) in the presence of Et_3N (1.0 equiv) and 4 Å MS (20 mg) in DCM (2.0 mL) under a N_2 atmosphere at 25 °C. After the solvent was removed in vacuo, MeONa (1.5 equiv in 2.0 mL MeOH) was added and the mixture was stirred for another 12 h. All reactions afforded >99:1 dr determined by ^1H NMR analysis of the crude reaction mixtures. The yields were isolated ones; ee's were determined by HPLC analysis.

reaction proceeded smoothly providing the anticipated "branched" alkylation product with good yields. However, enantioselectivities were low (less than 50% ee, **L1**−**L4**). Then, axially chiral O,O,P,N-ligands **L5** were used in the reaction. **L5a** with the least steric hindrance on the biphenyl skeleton delivered the best results; however, the desired product was only obtained with 45% ee. To examine the effect the ligand skeleton had on the reaction, **L6a** bearing the same central chirality but different axial chirality to **L5a** was used as a chiral ligand. To our delight, the reaction proceeded smoothly in high yield and with excellent enantioselectivity (91% ee). Further altering of the central chiral group resulted in reduced enantioselectivities (**L6b** and **L6c**).

The effect of different additives on the reaction was therefore investigated using **L6a** as a chiral ligand (Table 1). In the absence of an additive, the reaction went to completion within 8 h, providing the desired product with 90% yield and 91% ee (entry 1). When $AgSbF_6$ was added, a similar yield was obtained but a sharp reduction in enantioselectivity was observed (entries 1 and 2). $AgBF_4$, AgOTf, and KOAc were also examined. The reactions proceeded smoothly with excellent enantioselectivities but only moderate yields, even after 24 h (entries 3−5). To our delight, similar catalytic behavior but increased reaction activity was observed when $NaBAr_F$ was used as an additive (entry 6). This may be due to the improved catalytic activity caused by the larger, noncoordinating BAR_F counterion.[11]

Solvent also had a significant effect on the reaction (Table 2). 1,2-Dichloroethane gave a similar ee but lower reaction activity than that of DCM (entries 1 and 2). Ether solvents such as THF, DME, and Et_2O gave excellent enantioselectivities but only

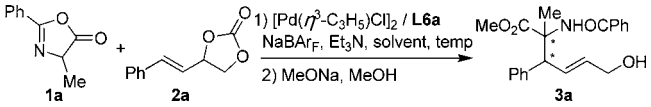
Table 1. Additive Screening[a]



entry	additive	t (h)	yield (%)[b]	ee (%)[c]
1	–	8	90	91
2	$AgSbF_6$	10	87	77
3	$AgBF_4$	24	71	90
4	AgOTf	24	71	90
5	KOAc	24	66	92
6	$NaBAr_F$	4	92	90

[a]The procedure and determination of the dr's were similar to those described in Table 1, except that **L6a** was used as a chiral ligand in the presence of different additives. All reactions afforded >99:1 dr. [b]Isolated yields. [c]Determined by HPLC analysis.

Table 2. Solvent and Temperature Screening[a]



entry	solvent	temp (°C)	t (h)	yield (%)[b]	ee (%)[c]
1	DCM	25	4	92	90
2	DCE	25	12	77	90
3	THF	25	12	71	91
4	DME	25	12	67	97
5	Et_2O	25	12	68	95
6	MeCN	25	12	37	75
7	PhMe	25	5	92	93
8	PhMe	0	20	89	96
9	PhMe	−25	72	75	96
10	PhMe	50	2	59	81

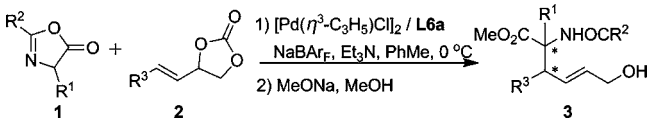
[a]The procedure and determination of the dr's were similar to those described in Table 1, except that **L6a** was used as a chiral ligand in the presence of $NaBAr_F$ in different solvents at a certain temperature. All reactions afforded >99:1 dr. [b]Isolated yields. [c]Determined by HPLC analysis.

moderate yields (entries 3−5). Poor yield and moderate enantioselectivity were observed when MeCN was used as a solvent (entry 6). PhMe was tested to be the best solvent in the reaction (entry 7). The effect of temperature on the reaction was then examined in PhMe (entries 8−10), with a temperature of 0 °C providing the best result. Thus, subsequent reactions were catalyzed by a catalytic system consisting of $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$ and **L6a** using Et_3N as a base in the presence of $NaBAr_F$ and 4 Å MS in PhMe at 0 °C.

The substrate scope was then investigated (Table 3). First, the effect the steric hindrance of the R^1 group of **1** had on the reaction was examined. It was shown that the ee of the desired product decreased when the steric hindrance of R^1 increased (entries 1−3). Electron-donating and -withdrawing R^2 substituents provided similar catalytic behaviors (entries 4 and 5). When substrates bearing electron-donating Me and MeO groups on the phenyl rings were used, good to excellent yields and enantioselectivities were obtained (entries 6−11). Substrates bearing an *ortho*-substituent on the phenyl ring provided the best enantioselectivities but lower yields (entries 6 and 9). Replacing the electron-donating substituent with an electron-withdrawing group, such as a F atom, had no adverse effect on the reaction with up to 99% ee being obtained for a substrate bearing a F atom

DOI: 10.1021/acs.orglett.5b02868
Org. Lett. 2015, 17, 5768−5771

Table 3. Scope of Substrate[a]



entry	R^1	R^2	R^3	3	t (h)	yield (%)[b]	ee (%)[c]
1	Me	C_6H_5	C_6H_5	**3a**	20	89	96
2	Et	C_6H_5	C_6H_5	**3b**	20	87	92
3	Bn	C_6H_5	C_6H_5	**3c**	20	90	90
4	Me	*m*-FC_6H_4	C_6H_5	**3d**	24	90	94
5	Me	*m*-MeC_6H_4	C_6H_5	**3e**	18	91	94
6	Me	C_6H_5	*o*-MeC_6H_4	**3f**	20	85	94
7	Me	C_6H_5	*m*-MeC_6H_4	**3g**	16	90	92
8	Me	C_6H_5	*p*-MeC_6H_4	**3h**	16	91	79
9	Me	C_6H_5	*o*-$MeOC_6H_4$	**3i**	16	75	89
10	Me	C_6H_5	*m*-$MeOC_6H_4$	**3j**	12	84	84
11	Me	C_6H_5	*p*-$MeOC_6H_4$	**3k**	12	89	79
12	Me	C_6H_5	*o*-FC_6H_4	**3l**	26	79	96
13	Me	C_6H_5	*m*-FC_6H_4	**3m**	24	88	97
14	Me	C_6H_5	*p*-FC_6H_4	**3n**	24	90	99
15	Me	C_6H_5	*p*-$CF_3C_6H_4$	**3o**	24	87	99
16	Me	C_6H_5	*p*-$NO_2C_6H_4$	**3p**	24	86	95
17	Me	C_6H_5	*p*-ClC_6H_4	**3q**	24	90	92
18	Me	C_6H_5	*p*-BrC_6H_4	**3r**	24	90	94
19	Me	C_6H_5	1-Nap	**3s**	20	87	91
20	Me	C_6H_5	2-Nap	**3t**	20	91	93
21	Me	C_6H_5	Furyl	**3u**	20	82	67

[a]The procedure and determination of the dr's were similar to those described in Table 1, except that **L6a** was used as a chiral ligand in the presence of $NaBAr_F$ in PhMe at 0 °C. All reactions afforded >99:1 dr. [b]Isolated yields. [c]Determined by HPLC analysis.

at the *para*-position of the phenyl ring (entries 12−14). Several substrates with an electron-withdrawing group at the *para*-position of the phenyl ring were examined, and both high yields and excellent enantioselectivities were obtained (entries 15−18). When the phenyl ring was replaced by naphthalene, high yields and excellent enantioselectivities were also obtained (entries 19 and 20). The phenyl ring could also be replaced by a furan group, with the desired product being prepared in high yield but lower enantioselectivity (entry 21).

Crystals suitable for X-ray analysis were obtained via recrystallization of **3a** from a mixed solvent system of dichloromethane and *n*-hexane. The ORTEP diagram of **3a** showed that the absolute configuration of the alkylated product could be assigned with an (*S*,*R*)-configuration (Figure 2).[12]

The formation of the above-mentioned "branched" allylic alkylated products suggested our hypothesis that a hydrogen-bond and/or a $\pi-\pi$ interaction may have an important effect on

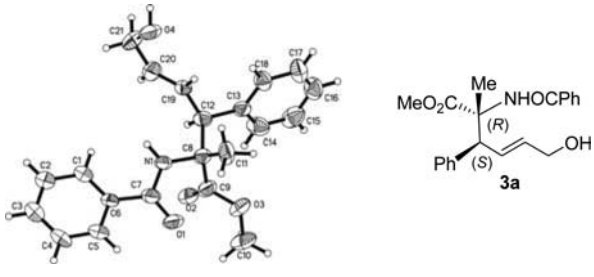
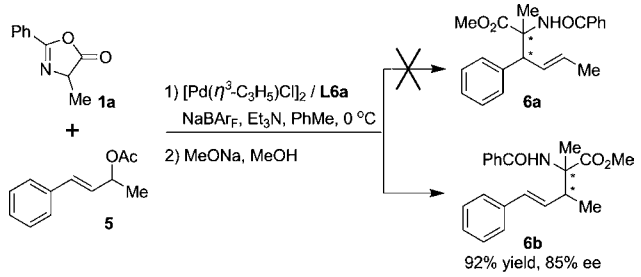


Figure 2. ORTEP diagram of **3a**.

regioselectivity of the reaction to be correct (Scheme 1, **A**). To further validate our original hypothesis, the Pd-catalyzed asymmetric allylic alkylation of **1a** with (*E*)-4-phenylbut-3-en-2-yl acetate (**5**) was carried out in order to avoid hydrogen-bond formation (Scheme 2). Under the above-mentioned optimal

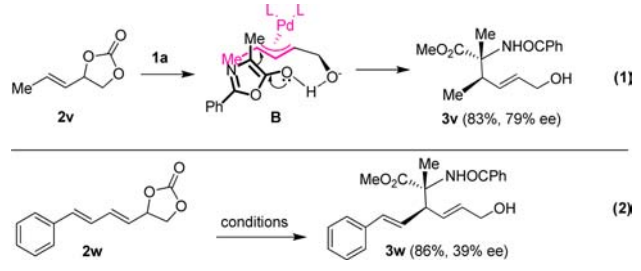
Scheme 2. Alkylation with 5 as an Allylic Substrate



reaction conditions, the reaction proceeded smoothly to afford **6b** in 92% yield and 85% ee after 16 h. However, product **6a** was not obtained. This result suggested that a hydrogen bond is required for the regioselective formation of "branched" products.

In order to determine whether a possible $\pi-\pi$ interaction contributed to the regioselectivity of the reaction (Scheme 1, **A**), we first carried out the reaction with **2v** as an allylic substrate (Scheme 3, eq 1). The phenyl ring was replaced by a Me group to

Scheme 3. Alkylation with 2v as an Allylic Substrate

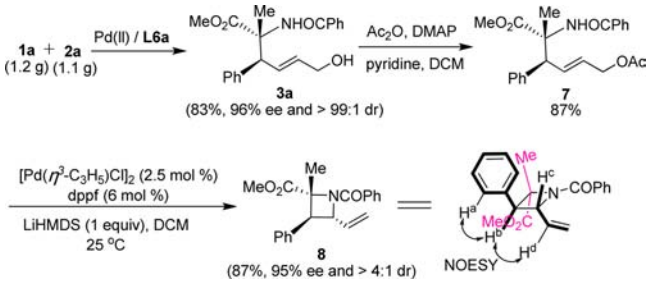


avoid possible $\pi-\pi$ interactions (**B**). The reaction proceeded smoothly giving the product **3v** in 83% yield and 79% ee. Additionally, substrate **2w** bearing an extended π-system was examined, and its corresponding product **3w** was obtained in 86% yield and 39% ee (Scheme 3, eq 2). These results suggest that a possible $\pi-\pi$ interaction between the phenyl ring of the Pd-allylic complex and the unsaturated bond or phenyl ring of the azlactone in **A** is not responsible for the regioselectivity of the reaction.

To prove the usefulness of this new synthetic methodology, the reaction between **1a** and **2a** was performed on a gram scale for 36 h (Scheme 4). The desired product **3a** was obtained in good yield (1.7 g, 83%) with excellent enantioselectivity (96% ee). After acetylation of **3a** with Ac_2O to give the allylic substrate **7**, the chiral azetidine **8** was then obtained from **7** in high yield and with excellent enantioselectivity via an additional Pd-catalyzed allylic amination. According to NOESY experiments, the two hydrogen atoms of the four-membered ring in **8** are of opposite configuration; a strong NOESY correlation is observed between H_b and H_a/H_d, but only a weak NOESY correlation is observed between H_b/H_c.[13] Azetidines are found in drug leads and bioactive natural products and are attracting increasing research interest.[14]

DOI: 10.1021/acs.orglett.5b02868
Org. Lett. 2015, 17, 5768−5771

Scheme 4. Gram Scale Synthesis and Transformation



In summary, we have developed a Pd-catalyzed asymmetric allylic alkylation of azlactones with 4-arylvinyl-1,3-dioxolan-2-ones to give "branched" chiral alkylated α-amino acid products with vicinal tertiary and quaternary stereocenters. The products can be obtained in high yields and with excellent regio-, diastero-, and enantioselectivities. Mechanistic studies suggested that the excellent regioselectivity of our alkylation is determined by a hydrogen bond formed between the Pd-allylic complex and azlactone isomer. Additionally, the alkylation can be performed on a gram scale without a loss of enantioselectivity, and the resulting product can be further transformed to a chiral azetidine in two simple steps.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.5b02868.

Experimental section and copies of NMR and HPLC spectra (PDF)

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Author Contributions

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Notes

The authors declare no competing financial interest.

■ ACKNOWLEDGMENTS

This work was partially supported by the National Natural Science Foundation of China (Nos. 21172143, 21172145, 21372152, and 21232004), Science and Technology Commission of Shanghai Municipality (No. 14XD1402300), and Nippon Chemical Industrial Co., Ltd. We gratefully acknowledge Dr. Masashi Sugiya and Professor Tsuneo Imamoto of Nippon Chemical Industrial Co., Ltd. for helpful discussions and the Instrumental Analysis Center of SJTU for characterization.

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(12) CCDC 1052610 contains 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.

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■ NOTE ADDED AFTER ASAP PUBLICATION

The toc/abs graphic was corrected on November 19, 2015.

DOI: 10.1021/acs.orglett.5b02868
Org. Lett. 2015, 17, 5768–5771