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Enantioselective Pd-catalyzed hydrogenation of tetrasubstituted olefins of cyclic β-(arylsulfonamido)acrylates

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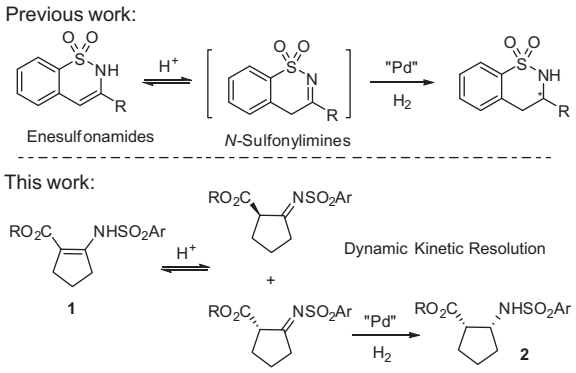
ABSTRACT

Asymmetric hydrogenation of tetrasubstituted olefins of cyclic β-(arylsulfonamido)acrylates produces the corresponding cyclic β-(arylsulfonamido)propionates using $Pd(OCOCF_3)_2$/diphosphine complexes as catalysts in the presence of TFA with up to 96% ee.

Enantiomerically pure β-amino acids and their derivatives are important chiral building blocks for the synthesis of many natural products and biologically active compounds.[1] They are also key structural elements of β-peptides, β-lactams.[2,3] Among them, cyclic β-(arylsulfonamido)propionate acid derivatives drew extreme attention of researchers due to their wide chemical and biological activities.[4] In the past few decades, many methods have been successfully developed to these β-amino acid deriatives.[5] However, despite the importance of β-amino acid derivatives, few researches have been done on the synthesis of chiral cyclic β-(arylsulfonamido) propionates.

According to retrosynthetic analysis of chiral β-(arylsulfonamido)propionates, asymmetric hydrogenation of tetrasubstituted olefins of the corresponding cyclic β-(arylsulfonamido)acrylates is one of the most atom economic and efficient approaches.

Although some examples on the asymmetric hydrogenation of tetrasubstituted unfunctionalized and functionalized olefins have been successfully reported recently,[6] exploring new catalyst system to asymmetric hydrogenation of tetrasubstituted olefins of cyclic β-(arylsulfonamido)acrylates is highly desirable. Recently, the chiral palladium complexes have been successfully applied to asymmetric hydrogenation of imines, ketones, simple indoles and pyrroles by us[7] and other groups.[8] Very recently, we reported the asymmetric hydrogenation of enesulfonamides.[9] The mechanism study showed that the hydrogenation was conducted via Brønsted acid catalyzed tautomerization of enesulfonamides to *N*-sulfonylimine intermediates (Scheme 1). Considering that the cyclic



Scheme 1. Synthesis of chiral cyclic β-amino acids.

β-(arylsulfonamido)acrylates **1** can be readily transformed into the corresponding sulfonylimine intermediates through isomerization in the presence of Brønsted acid (Scheme 1). We envision that the active imine intermediates should be easily hydrogenated with a proper catalytic system. In this Letter, asymmetric hydrogenation of cyclic β-(arylsulfonamido)acrylates is successfully developed using $Pd(OCOCF_3)_2$/DuanPhos complex as catalysts in the presence of TFA with up to 96% ee.

Cyclic β-(arylsulfonamido)acrylates **1** can be readily synthesized from the corresponding cyclic β-ketoester and substituted arylsulfonamides by slightly modified literature procedures in moderate to good yields (Scheme 2).[10]

Our initial study began with **1i** as the model substrate and $Pd(OCOCF_3)_2$/(R,S_p)-JosiPhos as the catalyst on the basis of our

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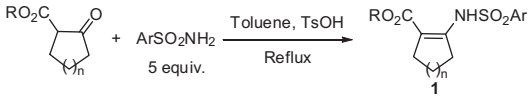
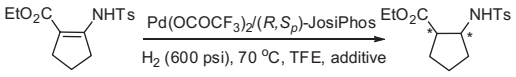


Scheme 2. Synthesis of β-(arylsulfonamido)acrylates **1**.

previous successful hydrogenation of activated enesulfonamides.[9] However, low conversion (57%) and moderate 64% ee value were observed (Table 1, entry 1). To enhance the activity and enantioselectivity, a number of Brønsted acids were tested, the results are summarized in Table 1. When 1 equiv of L-tartaric acid was added, the conversion was improved and with the almost same enantioselectivity (63% ee, Table 1, entry 2). The chirality of the additive had no influence on the hydrogenation (Table 1, entries 2 vs 3). Other Brønsted acids also were screened, giving incomplete conversions but still with moderate enantioselectivities (Table 1, entries 4–6). When strong acid CF_3CO_2H (TFA) was added, full convention and

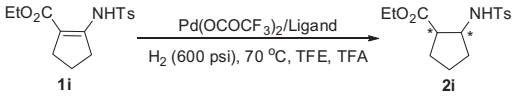
Table 1
The effect of additives on the reactivity and enantioselectivity[a]



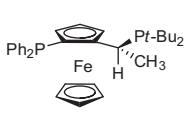
Entry	Additive	Conv[b] (%)	ee[c] (%)
1	—	57	64
2	L-Tartaric acid	70	63
3	D-Tartaric acid	68	63
4	Benzoic acid	80	77
5	4-Nitrobenzoic acid	61	68
6	Phthalic acid	68	76
7	Trifluoro-acetic acid	>95	81
8	2,3,4,5,6-Pentafluorobenzoic acid	28	82
9	2-Hydroxy-3,5-dinitrobenzoic acid	>95	81
10	Morpholine-TFA	27	77

[a] Conditions: 0.125 mmol of **1i**, $Pd(OCOCF_3)_2$ (0.0025 mmol), (R,S_p)-JosPhos (0.003 mmol), H2 (600 psi), additive (100 mol %), 3 mL of TFE, 16 h, 70 °C.
[b] Determined by ^1H NMR on the crude mixture.
[c] Determined by HPLC.

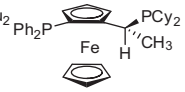
Table 2
Ligand screening for the Pd-catalyzed asymmetric hydrogenation of **1i**[a]



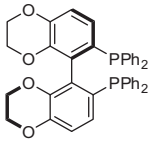
Entry	Ligand	Conv[b] (%)	ee[c] (%)
1	**L1**	>95	47
2	**L2**	>95	81
3	**L3**	64	78
4	**L4**	31	75
5	**L5**	60	68
6	**L6**	29	80
7	**L7**	71	79
8	**L8**	62	58
9	**L9**	>95	77
10	**L10**	71	89
11	**L11**	97[d]	91
12[e]	**L11**	92[d]	90
13[f]	**L11**	95[d]	90
14[g]	**L11**	92[d]	92



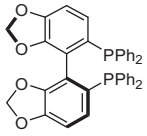
L1: (*R*)-(S)-PPF-P*t*-Bu$_2$



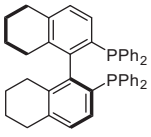
L2: (R,S_p)-JosiPhos



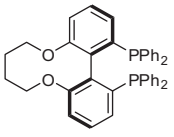
L3: (*R*)-SynPhos



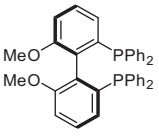
L4: (*S*)-SegPhos



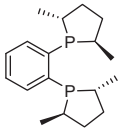
L5: (*S*)-H8-BINAP



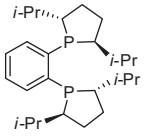
L6: (*R*)-C$_4$-TunePhos



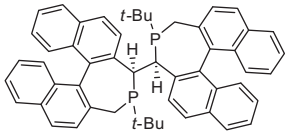
L7: (*S*)-MeO-Biphep



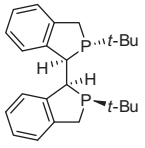
L8: (*R,R*)-Me-DuPhos



L9: (*S,S*)-*i*-Pr-DuPhos



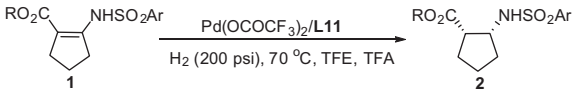
L10: (*S*)-BINAPINE



L11: (1*R*,1'*R*,2*S*,2'*S*)-DuanPhos

[a] The reaction was carried out with **1i** (0.125 mmol), $Pd(OCOCF_3)_2$ (0.0025 mmol), ligand (0.003 mmol), H_2 (600 psi), TFA (100 mol %), and 3 mL TFE under directed condition for 16 h.
[b] Determined by ^1H NMR on the crude mixture.
[c] Determined by HPLC.
[d] Isolated yield.
[e] H_2 (800 psi).
[f] H_2 (400 psi).
[g] H_2 (200 psi).

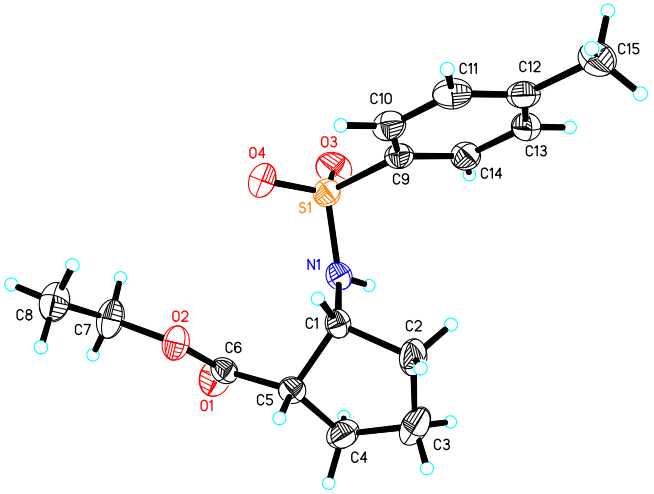
Table 3
Pd-catalyzed asymmetric hydrogenation of **1**[a]



1 2

Entry	Rof **1**	Ar of **1**	Yield[b] (%)	ee[c] (%)
1	Me	C_6H_5	84 (**2a**)	91 (+)
2	Et	C_6H_5	92 (**2b**)	93 (+)
3	*n*-Bu	C_6H_5	93 (**2c**)	90 (+)
4	*n*-Hex	C_6H_5	89 (**2d**)	92 (+)
5	Bn	C_6H_5	93 (**2e**)	89 (+)
6	$C_6H_5CH_2CH_2$	C_6H_5	91 (**2f**)	86 (+)
7	*i*-Pr	C_6H_5	90 (**2g**)	92 (+)
8	Me	4-MeC_6H_4	92 (**2h**)	91 (+)
9	Et	4-MeC_6H_4	92 (**2i**)	92 (1*S*,2*R*)[d]
10	*n*-Bu	4-MeC_6H_4	90 (**2j**)	90 (+)
11	*n*-Hex	4-MeC_6H_4	91 (**2k**)	89 (+)
12	Bn	4-MeC_6H_4	93 (**2l**)	91 (+)
13	$C_6H_5CH_2CH_2$	4-MeC_6H_4	92 (**2m**)	87 (+)
14	*i*-Pr	4-MeC_6H_4	93 (**2n**)	91 (+)
15[e]	Et	4-ClC_6H_4	87 (**2o**)	89 (+)

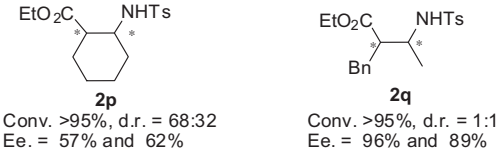
[a] The reaction was carried out with **1** (0.125 mmol), $Pd(OCOCF_3)_2$ (0.0025 mmol), ligand (0.003 mmol), TFA (0.125 mmol), and 3 mL TFE under directed condition for 16 h.
[b] Isolated yield.
[c] ee was determined by HPLC.
[d] The absolute configuration of **2i** was determined by X-ray analysis. Other products' configurations were assigned by analogy to **2i**.
[e] 4 mol % catalyst was used.



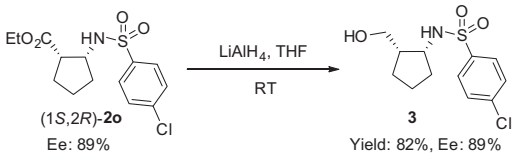
Scheme 3. The absolute configuration of **2i**.

81% ee value were obtained (Table 1, entry 7). Next, some strong Brønsted acids were also tested (Table 1, entries 8 and 9). Morpholine-TFA salt was also tested; only 27% conversion and 77% ee value were obtained. Eventually, TFA was found to be the optimal choice.

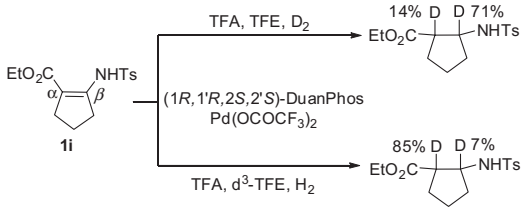
Subsequently, some commercially available bisphosphine ligands were examined under the above optimal conditions (Table 2). Initially, ferrocenyl ligands **L1**, **L2** gave full conversion and moderate enantioselectivity (Table 2, entries 1 and 2). Lower enantioselectivities were also observed with axial chiral ligands (Table 2, entries 3–7) and ligands **L8**, **L9** (Table 2, entries 8 and 9). When (*S*)-BINAPINE was employed, the enantioselectivity was increased to 89% ee (Table 2, entry 10). It is noteworthy that (1*R*,1′*R*,2*S*,2′*S*)-DuanPhos, developed by Zhang group,[11] showed slightly higher enantioselectivity and activity (Table 2, entry 11).



Scheme 4. Asymmetric hydrogenation of **1p** and **1q**.



Scheme 5. Synthesis of drug intermediate **3**.



Scheme 6. Isotopic labeling experiments using D_2 and d^3-TFE.

The hydrogen pressure had no apparent effect on enantioselectivity (Table 2, entries 12 and 13). When the H_2 pressure was lowered to 200 psi, the highest ee value (92%, Table 2, entry 14) was obtained.

Under the optimal reaction conditions, a wide variety of cyclic β-(arylsulfonamido)acrylates **1** were explored to examine the reaction scope. As shown in Table 3, the substrates with steric difference in esters resulted in excellent enantioselectivities (86–93% ee, entries 1–15, Table 3). The phenyl 4-methylphenyl or 4-chlorophenyl sulfonamide group substituted cyclic β-(arylsulfonamido) acrylates **1** can be successfully hydrogenated, giving the corresponding cyclic β-(arylsulfonamido)propionates in 87–92% ee (entries 8–15, Table 3).

The absolute configuration of hydrogenation product **2i** was ambiguously assigned to be (1*S*,2*R*) (Scheme 3) by X-ray crystallographic analysis after recrystallization from CH_2Cl_2/hexane.

In addition to five-membered cyclic β-(arylsulfonamido)acrylates, the hydrogenation of the six-membered cyclic β-(tosylamido) acrylates **1p** and a cyclic ethyl 2-benzyl-3-(tosylamido)but-2-enoate **1q** was also studied. Under the above optimal conditions, moderate to excellent ees but poor diastereoselectivities were obtained (Scheme 4).

To further demonstrate the value of this asymmetric hydrogenation of tetrasubstituted olefins of cyclic β-(arylsulfonamido)acrylates, we focused upon the development of a facile and expeditious route to key the intermediate **3** of the potential drugs to Alzheimer's disease developed by GSK company.[4d] As can be seen in Scheme 5, hydrogenation product **2o** was reduced with $LiAlH_4$ in THF to afford the key intermediate **3** in 82% yield without the loss of the optical purity.

To investigate the process of the reaction, two isotopic labeling experiments were carried out. When **1i** was subjected to hydrogenation with D_2, the deuterium atoms were incorporated to the β-position with 71% and α-position with 14%, respectively (Scheme 6). When the hydrogenation was carried out in d^3-TFE (Scheme 6), ^{1}H NMR analysis of the hydrogenated product showed that the deuterium atoms were taken up to the α-position with 85% incorporation and β-position with 7%, respectively. The result suggested that the

hydrogenation of **1i** was mainly proceeded via *N*-sulfonylimine intermediates in the presence of acids, and the tautomerization process of enesulfamide to *N*-sulfonylimine intermediates was faster than the hydrogenation, which is in fact a dynamic kinetic resolution process.

In conclusion, we have developed an efficient and highly enantioselective Pd-catalyzed hydrogenation of tetrasubstituted olefins of cyclic β-(arylsulfonamido)acrylates with TFE as the solvent in the presence of TFA, giving the chiral cyclic arylsulfonamido substituted β-amino acid derivatives with up to 96% ee. Further exploring the applications of this method in various asymmetric syntheses of biologically active compounds is currently underway.

Acknowledgments

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Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.tetlet.2012.03.035.

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