HETEROCYCLES, Vol. 63, No. 10, 2004, pp. 2385 - 2392
Received, 20th July, 2004, Accepted, 19th August, 2004, Published online, 20th August, 2004

ENANTIOSELECTIVE SYNTHESIS OF (*S*)-3-(4-THIAZOLYL)-2-*tert*-BUTOXYCARBONYLAMINOPROPIONIC ACID: A CHIRAL BUILDING BLOCK FOR RENIN INHIBITOR

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Abstract – (*S*)-3-(4-Thiazolyl)-2-*tert*-butoxycarbonylaminopropionic acid (**6**), an important structural constituent of the renin inhibitor, has been synthesized from (*Z*)-3-(4-thiazolyl)-2-benzoylaminoprop-2-enoic acid (**4b**) by enantioselective hydrogenation using the Ru-(*S*)-*p*-tolyl-BINAP complex as the key step, and then followed by acid hydrolysis and *tert*-butoxycarbonylation.

INTRODUCTION

Based on the concept of isosterism in medicinal chemistry, 3-(4-thiazolyl)-L-alanine can be considered as an annular equivalent of L-histidine. Furthermore, (*S*)-3-(4-thiazolyl)-2-*tert*-butoxycarbonylamino-propionic acid (**6**) has become an interesting component in the preparation of a potential renin inhibitory active peptide by its corporation.[1] Several preparations of **6** are known:

a) The synthesis from (*S*)-benzyloxycarbonyl-5-oxo-4-oxazolidinoneacetic acid, which was derived from the commercially available *N*-benzyloxycarbonyl-L-aspartic acid.[2]

b) The enzymatic resolution of the ethyl ester of *N*-acetyl-3-(4-thiazolyl)-DL-alanine by chymotrypsin[2] or acetylase.[3]

c) The synthesis from the methyl ester of *N*-*tert*-butoxycarbonyl-L-aspartic acid.[4]

On the other hand, the enantioselective hydrogenation with the Ru-BINAP complex catalyst is one of the most powerful tools for the synthesis of optically active compounds.[5] In paticular, the enantioselective

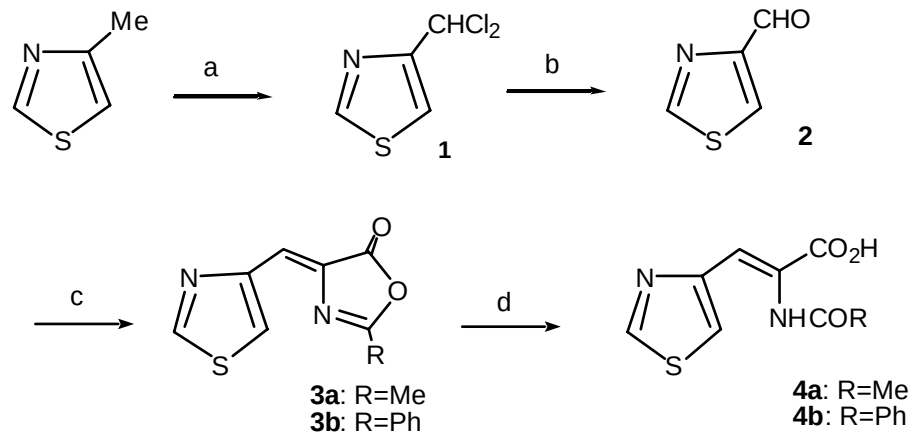
hydrogenation of 3-substituted 2-propenoic acids and 3-phenyl-2-aminoprop-2-enoic acid are well known.[6-9] The enantioselective hydrogenation of a five-membered heterocycle substituted 2-acylaminoprop-2-enoic acid using chiral diphosphinerhodium catalysts has also been reported.[10] However, that of the 3-(4-thiazolyl)-2-acylaminoprop- 2-enoic acids (**4a**) and (**4b**) is unknown.

We have studied the enantioselective hydrogenation of **4a** and **4b** in order to develop the synthesis of **6**. We now report the efficient synthesis of **6** from 3-(4-thiazolyl)-2-benzoylaminopropanoic acid (**5b**) *via* the enantioselective hydrogenation of **4b** using Ru-*p*-tolyl-BINAP complex catalyst.[11]

RESULTS AND DISCUSSION

The preparation of (*Z*)-3-(4-thiazolyl)-2-acylaminoprop-2-enoic acids (**4a**) and (**4b**) as substrates for the enantioselective hydrogenation is illustrated in scheme 1. The chlorination of 4-methylthiazole with Cl_2 gas in conc. H_2SO_4 and SO_3 in the presence of a catalytic amount of AIBN at 100°C for 20 h afforded a mixture of 4-dichloromethylthiazole (**1**) and 4-chloromethylthiazole in 45% and 21% yields, respectively.[12]

Scheme 1



Reagents and conditions: a) $Cl_2/SO_3/PCl_3/H_2SO_4$, 100°C, 20 h. b) 10%H_2SO_4, 100°C, 2 h c) $RCONHCH_2CO_2H$, AcONa, rt, 20 min. d) **3a**: 25% aqueous acetone, reflux, 2 h **3b**: NaOH, 100°C, 45 min.

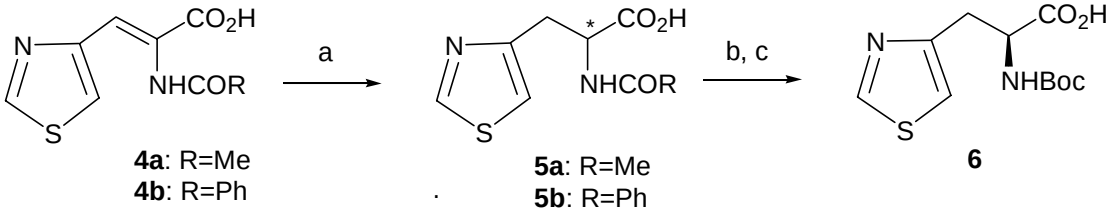
The dichloride (**1**) was then treated with 10% H_2SO_4 at 100°C for 2 h to give 4-thiazolecarbaldehyde (**2**) in 85% yield. Subsequently, **2** was condensed with acetylglycine and hippuric acid to give 4-thiazolylazlactone (**3a**) and (**3b**) in 52% and 84% yields, respectively. The treatment of **3a** with aqueous acetone solution gave **4a** in 68% yield. Alternatively, **3b** in aqueous NaOH solution was refluxed to give **4b** in 85% yield.

We examined the hydrogenation of **4a** and **4b** in the presence of the Ru-(*S*)- or (*R*)-*p*-tolyl-BINAP

complex as a catalyst under several different conditions. The results are summarized in Table 1.

The reaction was carried out in a polar solvent such as MeOH or MeOH/H_2O. It was found that the reaction temperature affected the enantioselectivity of the product. Upon the hydrogenation of **4a** and **4b** at 20°C, the reaction proceeded at a significantly slower rate, therefore a longer reaction time was required to complete the reaction, but the optical yield was in a moderate range (56-77% ee) (Entries 1~4). When the temperature was raised from 20°C to 60°C, the reaction rate accelerated, but the enantioselectivity was lower than the others (Entry 5). The reaction at 35°C gave a result similar to that at 60°C in yield and enantioselectivity (Entry 6). In addition, it is known that the hydrogenation using the Ru-*p*-tolyl-BINAP complex as a catalyst can be carried out in the presence of triethylamine;[6] however, the enantioselective hydrogenation of **4b** could not be effected in the presence of a triethylamine (Entry 7).

Scheme 2



Reagents alnd conditions : a) H_2, 5MPa, Ru_2Cl_4[(*S*)- or (*R*)-*p*-tolyl-BINAP] $_2Et_3N$, MeOH, b) HCl, reflux, 20 h. c) (*t*-Boc$_2$)O, THF rt, 40 h.

Table 1. Asymmetric hydrogenation of 4a and 4b

Entry	Substrate	Catalyst[A]	Solvent	Temp. (°C)	Time (h)	Yield (%)	E.e. (%)[C]	Config.
1	**4a**	(+)-A	MeOH	20	120	100	75	(*R*)-**5a**
2	**4a**	(-)-A	MeOH	20	120	100	77	(*S*)-**5a**
3	**4b**	(+)-A	MeOH	20	140	100	60	(*R*)-**5b**
4	**4b**	(-)-A	MeOH	20	140	98	56	(*S*)-**5b**
5	**4b**	(-)-A	MeOH	60	42	100	51	(*S*)-**5b**
6	**4b**	(-)-A	MeOH/H_2O (10/1)	35	120	100	55	(*S*)-**5b**
7	**4b**	(-)-A	MeOH/H_2O (10/1)[B]	35	120	11	—	(*S*)-**5b**

Reaction conditions: H_2 press. 5 MPa. catalyst; 1 mole%,
[A](+)-A; an empirical formula Ru_2Cl_4[(*R*)-(+)-*p*-tolyl-BINAP] $_2Et_3N$, (-)-A; Ru_2Cl_4[(*S*)-(-)-*p*-tolyl-BINAP] $_2Et_3N$.
[B]Entry 7 was carried out in the presence of 1.1 eq. mole triethylamine.
[C]The ee value of **5a** was determined by HPLC after being converted to **6**. The ee value of **5b** was determined by HPLC after being converted to the corresponding methyl ester.

The absolute configurations of **5a** and **5b** were determined by comparison with the optical rotations in the literature[2] after conversion to **6**. The enantiomeric excess of 3-(4-thiazolyl)-2-benzoylaminopropanoic acid (**5b**) was determined by HPLC after converting it to the corresponding methyl ester using diazomethane (Entries 3~7). However, that of 3-(4-thiazolyl)-2-acetylaminopropanoic acid (**5a**) could not

be determined using similar HPLC conditions. The enantiomeric excess of **5a** was determined by HPLC after converting it to **6** (Entries 1 and 2).

Furthermore, the recrystallization of **5b** (Entry 3, 60% ee) using EtOAc/MeOH gave the pure compound **5b** in 40% yield with 100% ee. Attempts to obtain the pure compound (**5a**) under similar recrystallizations were unsuccessful. The target compound (**6**) was derived from **5b** in 57% with 99% ee by the usual method; acid hydrolysis and *tert*-butoxycarbonylation (Scheme 2 and see EXPERIMENTAL).

CONCLUSION

We have described the efficient synthesis of the chiral building block for the renin inhibitor. (*S*)-3-(4-Thiazolyl)-2-*tert*-butoxycarbonylaminopropionic acid (**6**) was synthesized from 3-(4-thiazolyl)-2-benzoylaminopropanoic acid (**5b**) *via* the enantioselective hydrogenation of (*Z*)-3-(4-thiazolyl)-2-benzoylaminoprop-2-enoic acid **4b** using an optically active Ru-(*S*)-*p*-tolyl-BINAP complex catalyst.

EXPERIMENTAL

All reagents and solvents were obtained from commercial sources and used without further purification. Melting points were determined using a Yanagimoto micro melting apparatus and are uncorrected. The IR spectra were run on a JASCO IR-810. The ^{1}H-NMR spectra were recorded in $CDCl_3$ or CD_3OD with TMS as the internal standard using a Bruker AM-400 (400MHz) spectrometer. MS spectra were run on a Hitachi M-80A spectrometer at 70eV. Optical rotations were recorded using a JASCO DIP-4 digital polarimeter. The HPLC was done using a Hitachi L-6000 [column, with a L-4000 UV as the detector, Optical purity; CHIRALCEL OC, 4.6 mm ID x 250 mm; eluent: 2-propanol/*n*-hexane = 35:65, flow rate, 1 mL/min; detector, UV (254 nm)]; chemical purity; [column: Inertsil ODS-2 (250 mm x 4.6 mm); eluent: MeCN/H_2O, 7:3 (pH 2.3; adjusted by using H_3PO_4); flow rate: 0.5 mL/min; detection: UV (254 nm)].

4-Thiazolecarbaldehyde (2)

4-Methylthiazole (50 g, 0.5 mol, Aldrich) was dissolved in a mixture of 98% H_2SO_4 (46 g) and concentrated H_2SO_4 containing 25% SO_3 (47.5 g). PCl_3 (1 mL) was added and AIBN (1.5 g) in 98% H_2SO_4 (36 g) was then added to the mixture over a period of 10 h with stirring at 85-90°C. During this time a stream of Cl_2 gas was continually introduced into the reaction mixture. The mixture was then degassed *in vacuo* at 60°C, cooled and quenched by pouring over 300 g of crushed ice. The quenched mixture was then extracted with 3 x 200 mL of benzene. The benzene extracts were combined, washed

with $NaHCO_3$ solution, dried over anhydrous $MgSO_4$, and the benzene evaporated *in vacuo*. The residue was distilled at 53-54°C/0.5 torr to give 4-dichloromethylthiazole (37.5 g, 45%). The benzene extract water layer was treated with conc. NH_4OH until the pH reached 6.0, and the resulting oil extracted into benzene. The benzene extract was dried over anhydrous $MgSO_4$, and the benzene evaporated *in vacuo*. The residue was distilled at 48-49°C/0.7 torr to give 4-chloromethylthiazole (14.0 g, 21%). 4-Dichlorothiazole (24 g, 0.143 mol) in 10% H_2SO_4 (300 mL) was refluxed under N_2 at 125-130°C for 2 h. The mixture was then cooled to rt and a 10% NaOH solution was added until the solution reached pH 6. The mixture was then extracted with 3 x 200 mL of $CHCl_3$. The organic layer was washed with water, dried over anhydrous $MgSO_4$ and concentrated *in vacuo* to give **2** (13.7 g, 85%). mp 65-66°C. lit.,[12] 65-67°C, lit., [13] 59-61°C. ^{1}H NMR ($CDCl_3$): δ 8.32 (d, *J* = 1.9, 1H), 8.99 (d, *J* = 1.9, 1H), 10.12 (s, 1H); IR ν_{max} ($CHCl_3$) 1700, 1585, 1425 cm^{-1}. MS: m/z 113 (62%, M^+), 85 (100), 58 (72), 57 (56), 45 (40), 29 (24).

4-Thiazolylazlactone (3a)

A mixture of **2** (3.2 g, 28 mmol), acetylglycine (3.25 g, 28 mmol), sodium acetate (12.35 g, 0.15 mol) and acetic anhydride (30 mL) were heated for 20 min at 100°C. The reaction products were cooled, filtrated and washed with water. The crude product was recrystallized from acetone to give **3a** (2.8 g, 52%). mp 115-118°C. Anal. Calcd for $C_8H_6N_2O_2S$: C, 49.47; H, 3.11; N, 14.42. Found: C, 49.61; H, 3.09; N, 14.44. ^{1}H NMR ($CDCl_3$): δ 7.53-7.59 (m, 3H), 7.62-7.66 (m, 1H), 8.18-8.21 (m, 2H), 8.83 (d, *J* = 2.1, 1H), 8.90 (d, *J* = 2.1, 1H); IR ν_{max} ($CHCl_3$) 3440, 1710, 1650, 1520 cm^{-1}; MS: m/z 194 (95%, M^+), 166 (19), 148 (3), 124 (100), 97 (38), 60 (14), 43 (87).

4-Thiazolylazlactone (3b)

A mixture of **2** (10.2 g, 90 mmol), hippuric acid (7.4 g, 90 mmol), sodium acetate (36.3 g, 0.43 mol) and acetic anhydride (50 mL) was heated for 20 min at 100°C. The reaction products were cooled, filtrated and washed with water. The crude product was recrystallized from $CHCl_3$ to give **3b** (19.4 g, 84%). mp 174-176°C. Anal. Calcd for $C_{13}H_8N_2O_2S$: C, 60.92; H, 3.15; N, 10.93. Found: C, 61.08; H, 3.09; N, 10.85. ^{1}H NMR ($CDCl_3$): δ 7.53-7.59 (m, 3H), 7.62-7.66 (m, 1H), 8.18-8.21 (m, 2H), 8.83 (d, *J* = 2.1, 1H), 8.90 (d, *J* = 2.1, 1H); IR ν_{max} ($CHCl_3$) 3620, 1800, 1660, 1520 cm^{-1}. MS: m/z 256 (13%, M^+), 105 (100), 77 (56), 69 (10), 51 (6).

(*Z*)-3-(4-Thiazolyl)-2-acetylaminoprop-2-enoic acid (4a)

The mixture of **3a** (2.5 g, 12.9 mmol) and 25% aqueous acetone (80 mL) was refluxed for 2 h. The reaction mixture was then concentrated *in vacuo*. The residue was extracted with $CHCl_3$ and washed with water. The solvent was removed and the solid residue was recrystallized from MeOH to give **4a** (1.86 g, 68%) as a solid. mp 185-187°C. Anal. Calcd for $C_8H_8N_2O_3S$: C, 45.27; H, 3.80; N, 13.20. Found: C, 45.31; H, 3.78; N, 13.05. ^{1}H NMR (CD_3OD): δ 7.09 (s, 1H), 7.53-7.64 (m, 3H), 7.82 (d, *J* = 1.9, 1H),

7.99-8.02 (m, 2H), 9.11 (d, J = 1.5, 1H); IR ν_{max} ($CHCl_3$) 3440, 1710, 1650, 1520 cm^{-1}; MS: m/z 212 (43%, M^+), 194 (3), 184 (7), 170 (68), 168 (67), 126 (100), 99 (80), 93 (95), 82 (5), 72 (13).

(*Z*)-3-(4-Thiazolyl)-2-benzoylaminoprop-2-enoic acid (4b)

The mixture of **3b** (19 g, 74 mmol) and 0.3% NaOH (780 mL) was stirred at 100°C for 45 min. The reaction mixture was then cooled and filtered. The filtrate was carefully acidified with 10% HCl to give a solid. The solid was washed with acetone and ether. The crude product was recrystallized from a mixture of EtOAc and MeOH to give **4b** (21.7 g, 85%). mp 206-208°C. Anal. Calcd for $C_{13}H_{10}N_2O_3S$: C, 56.92; H, 3.67; N, 10.21. Found: C, 59.88; H, 3.61; N, 10.16. ^{1}H NMR (CD_3OD): δ 7.09 (s, 1H), 7.53-7.64 (m, 3H), 7.82 (d, J = 1.9, 1H), 7.99-8.02 (m, 2H), 9.11(d, J = 1.5, 1H); IR ν_{max} ($CHCl_3$): 3440, 1710, 1650, 1520 cm^{-1}; MS: m/z 275 (80%, M^++1), 257 (27), 231 (23), 230 (27), 153 (14), 122 (6), 105 (100), 77 (17).

(*S*)-3-(4-Thiazolyl)-2-acetylaminopropanoic acid (5a)

Into a 100 mL autoclave were charged **4a** (0.5 g, 2.35 mmol) in MeOH (20 mL) and Ru_2Cl_4[(*S*)-(-)-*p*-tolyl-BINAP]$_2Et_3N$ (20 mg, 0.022 mmol) under an atmosphere of N_2. The atmosphere was then replaced with H_2 at a pressure of 5 MPa. The reaction mixture was stirred for the appropriate time and at the temperature (see Table 1). The reaction solution was concentrated *in vacuo* and the solid residue was recrystallized from $CHCl_3$ to give **5a** (0.45 g, 90%) as a solid. For the measurement of the enantiomeric purity by HPLC, **5a** was converted into **6**. mp 166-170°C, The enantiomeric purity was 77% ee. $[\alpha]_D^{23}$ -8.0^o(*c* 1.0, MeOH). ^{1}H NMR (CD_3OD) δ 3.40 (dd, J = 8.5, 14.5, 1H), 3.52 (dd, J = 4.5, 14.5, 1H), 7.35 (s, 1H), 7.41-7.46 (m, 2H), 7.50-7.55 (m, 1H), 7.76-7.78 (m, 1H), 8.95(d, J = 1.8, 1H); IR ν_{max} ($CHCl_3$): 3410, 3310, 1705, 1630, 1520 cm^{-1}; MS: m/z 214 (11%, M^+), 169 (16), 155 (15), 127 (100), 111 (60), 99 (95), 74 (23), 43 (78). HRMS calcd for $C_8H_{10}N_2O_3S$: 214.2386, Found: 214.2394.

(*S*)-3-(4-Thiazolyl)-2-benzoylaminopropanoic acid (5b)

Into a 100 mL autoclave were charged **4b** (1.0 g, 3.6 mmol) in MeOH (20 mL) and Ru_2Cl_4[(*S*)-(-)-*p*-tolyl-BINAP]$_2Et_3N$ (20 mg, 0.022 mmol) under an atmosphere of N_2. The atmosphere was then replaced with H_2 at a pressure of 5 MPa. The reaction mixture was stirred for the appropriate time and at the temperature (see Table 1). The reaction solution was concentrated *in vacuo* and the solid residue was recrystallized from a mixture of EtOAc and MeOH to give **5b** (0.41 g, 40%). For the measurement of the enantiomeric purity by HPLC, **5b** were converted into the methyl ester of **5b** using diazomethane. The enantiomeric purity was 100% ee. [(*S*)-tr 21.7 min, (*R*)-tr 27.4 min]. mp 198-200°C, $[\alpha]_D^{23}$ -22.0^o (*c* 1.0, MeOH). ^{1}H NMR (CD_3OD): δ 3.40 (dd, J = 8.5, 14.5, 1H), 3.52 (dd, J = 4.5, 14.5, 1H), 7.35 (s, 1H), 7.41-7.46 (m, 2H) 7.50-7.55 (m, 1H), 7.76-7.78 (m, 1H), 8.95 (d, J = 1.8, 1H); IR ν_{max} ($CHCl_3$): 3410, 3310, 1705, 1630, 1520 cm^{-1}. MS: m/z 276 (6%, M^+), 232 (6), 171 (5), 155 (6), 127 (36), 111 (24), 105 (100), 99 (36), 77 (30). HRMS calcd for $C_{13}H_{12}N_2O_3S$: 276.3094, Found: 276.3098.

(*S*)-3-(4-Thiazolyl)-2-*tert*-butoxycarbonylaminopropanoic acid (6)

5b (0.30 g, 1.4 mmol) and 6 N HCl (25 mL) were refluxed for 20 h and then cooled to rt. The reaction mixture was concentrated *in vacuo*. The residue was washed with toluene to remove the benzoic acid. The crude product (0.24 g) was dissolved in THF (25 mL) and adjusted to pH 9 with 3N NaOH solution. Di-*tert*-butyl dicarbonate (0.23 g, 1.05 mmol) in THF (5 mL) was added to the mixture and stirred for 40 h at rt. The solvent was removed *in vacuo* and the residue was adjusted to pH 2 with 3 N HCl, and then extracted with EtOAc (50 mL), washed with brine and dried using anhydrous $MgSO_4$. The solvent was concentrated *in vacuo* and the solid residue was recrystallized from acetone to give **6** (0.18 g, 57%) as a solid. The enantiomeric purity was 99% ee. [(*S*)- tr 7.8 min., (*R*)- tr 9.6 min]. mp 122-123°C, lit.,[2] mp 122-123°C, $[\alpha]_D^{24}$ +11.0^o (*c* 1.0, MeOH). $[\alpha]_D^{24}$ +125.9^o (*c* 1.0, $CHCl_3$) lit.,[2] $[\alpha]_D$ +124.9 (*c* 1.04, $CHCl_3$), ^{1}H NMR ($CDCl_3$): δ 1.46 (s, 9H), 3.31-3.39 (m, 1H), 3.49-3.55 (m , 1H), 4.5 (m, 1H), 5.61-5.65 (m, 1H), 7.18 (s, 1H), 8.89-8.91 (m, 1H); IR ν_{max} ($CHCl_3$): 3430, 1710, 1505 cm^{-1}; MS: m/z 273 (9%, M^++1), 259 (4), 257 (4), 217 (90), 199 (100), 173 (28), 153 (6), 127 (50), 111 (3), 99 (23), 71 (18).

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