A Novel Asymmetric Synthesis of 3-(1*H*-Pyrrol-1-yl)-Substituted *β*-Lactams *via* a Bismuth Nitrate-Catalyzed Reaction

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The reaction of racemic *α*-keto *β*-lactams **5a**–**5c** with the commercially available chiral compound *trans*-4-hydroxy-L-proline (**6**) in the presence of a catalytic amount of $Bi(NO_3)_3 \cdot 5\,H_2O$ in EtOH gave a diastereoisomer mixture of *β*-lactams with a pyrrole ring at C(3) (**7** to **12**). This is the first enantioselective synthesis of optically active *β*-lactams (= azetidin-2-ones) that possess a pyrrolyl residue at C(3), in a single step.

Introduction. – The *β*-lactam (= azeridin-2-one) skeleton is the key structural unit of the most widely used *β*-lactam antibiotics[1]). New *β*-lactam antibiotics are necessary for the treatment of bacterial diseases [2]. The *β*-lactam skeleton has also been recognized as an important starting material by exploiting its strain energy associated with the four-membered ring [3], and many efforts have been made to use enantiomerically pure *β*-lactams as versatile intermediates for organic syntheses [3][4].

Like *β*-lactams, substituted pyrroles are also an important class of compounds with various biological activities[2]). Methods for the synthesis of diversely substituted pyrroles have been described [6]. Conjugate addition reactions [7], transition metal-mediated reactions [8], reductive couplings [9], aza-*Wittig* reactions [10], and other multistep operations [11] have been performed for the preparation of several pyrroles. We disclose the synthesis of optically pure *β*-lactams substituted with a 1*H*-pyrrol-1-yl moiety at C(3) of the azetidine-2-one ring using a novel bismuth nitrate-induced catalytic method. To the best of our knowledge, this is the first report for the synthesis of optically active pyrrolyl-substituted *β*-lactams.

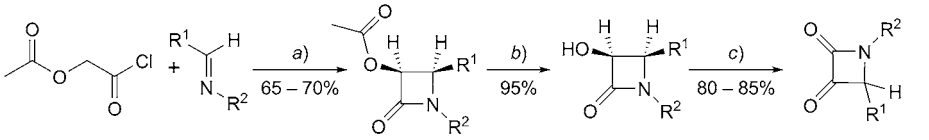
The use of Bi salts in several organic transformations has received much attention. Recently, our group has demonstrated a facile method for the synthesis of substituted pyrroles [12a][3]). In continuation of our endeavor for the synthesis and biological evaluation of novel anticancer *β*-lactams [13], we have developed a new method for the synthesis of novel *β*-lactams that have a pyrrol-1-yl ring at C(3) of the azetidine-2-one ring. Although the chemistry of *β*-lactams is extremely rich, preparation of optically active pyrrolyl-substituted azetidin-2-ones has not been described [14]. Asymmetric synthesis of *β*-lactams has been performed by employing either chiral ketenes derived

[1]) For reviews on *β*-lactam antibiotics, see [1].
[2]) For some biologically active pyrroles, see [5].
[3]) For our other $Bi(NO_3)_3$-catalyzed reactions, see [12b–12k].

from acid precursors or chiral imines (derived from either chiral aldehydes or amines) [15]. Here, we describe a novel method for the synthesis of optically active β-lactams starting from racemic α-keto β-lactam.

Results and Discussion. – The *Staudinger* cycloaddition of 1-(acetoxy)acetyl chloride (**1**) with imines **2** gave 3-acetoxy β-lactams **3** in 65–70% yields, which, on careful hydrolysis with aqueous NaOH in THF, afforded the corresponding 3-hydroxy β-lactams **4** in quantitative yields. Oxidation of the OH group was carried out by a known procedure with P_2O_5 and DMSO [16] to give the desired α-keto β-lactams **5a**–**5c** [16] in 80–85% yield (*Scheme 1*).

Scheme 1

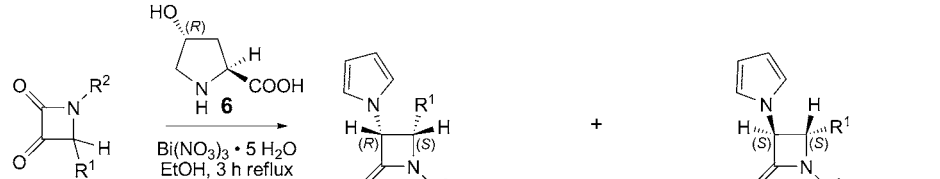


a $R^1 = R^2 = Ph$
b $R^1 = Ph$, $R^2 = 4\text{-MeO–}C_6H_4$
c $R^1 = R^2 = 4\text{-MeO–}C_6H_4$

a) Dry Et_3N, dry CH_2Cl_2, 0°, 15 h. *b*) Aq. NaOH, THF, 0°, 30 min; *c*) P_2O_5, dry DMSO, 24 h [16].

Initially, the racemic α-keto β-lactam **5a** was reacted with *trans*-4-hydroxy-L-proline (**6**) in the presence of a catalytic amount of $Bi(NO_3)_3 \cdot 5\,H_2O$ in EtOH at room temperature. However, no product formation was observed. We then tried the same reaction without $Bi(NO_3)_3 \cdot 5\,H_2O$ using only EtOH, but, no change of the reaction mixture was observed in TLC, and the starting material was recovered. When a solution of **5a** and **6** was refluxed for 3 h in the presence of $Bi(NO_3)_3 \cdot 5\,H_2O$ and EtOH, a dramatic change was observed (*Scheme 2*). The TLC indicated formation of two new products. The ^{1}H-NMR spectra of the crude product revealed the presence of a mixture of two diastereoisomeric β-lactams.

Scheme 2



	R^1	R^2			R^1	R^2
7	Ph	Ph	(3.5:1)	**8**	Ph	Ph
9	Ph	4-MeO–C_6H_4	(4:1)	**10**	Ph	4-MeO–C_6H_4
11	4-MeO–C_6H_4	4-MeO–C_6H_4	(4:1)	**12**	4-MeO–C_6H_4	4-MeO–C_6H_4

The two diastereoisomers were separated by flash column chromatography to obtain pure *cis*- and *trans*-β-lactam **7** and **8**, respectively, in a 3.5 : 1 ratio with a pyrrolyl

substituent at C(3). The stereochemical outcome and structures for **7** and **8** were established by IR and NMR spectroscopy. The major diastereoisomer **7** showed a characteristic absorption band at 1746 cm^{-1} for the β-lactam C=O group. The ^{1}H-NMR spectrum for **7** showed two *doublet*s at 5.42 and 5.76 ppm for H–C(4) and H–C(3) of the β-lactam ring, respectively. The coupling constant of these two *doublet*s was $J =$ 5.5 Hz, which confirmed the *cis*-configuration for **7** [17]. There were two *triplet*s observed at 5.88 and 6.47 ppm for four pyrrole H-atoms. The coupling constant ($J =$ 2.1 Hz) of these two *triplet*s confirmed the presence of the pyrrolyl moiety[4]). The signal at 167.8 ppm in the ^{13}C-NMR spectrum was also characteristic for a β-lactam C=O group.

The minor diastereoisomer **8** also gave rise to a characteristic absorption band at 1745 cm^{-1} in the IR spectrum, and its ^{1}H-NMR spectrum exhibited two *doublet*s at 4.96 and 5.13 ppm for H–C(4) and H–C(3), respectively, of the β-lactam ring. The smaller coupling-constant value ($J = 2.0$ Hz) confirmed the *trans*-configuration for **8**. Additionally, two *triplet*s at 6.25 and 6.76 ppm with a coupling constant J of 2.19 Hz also confirmed the presence of the pyrrole ring. A peak at 167.8 ppm in the ^{13}C-NMR spectrum was an additional evidence for the presence of a β-lactam C=O group in **8**.

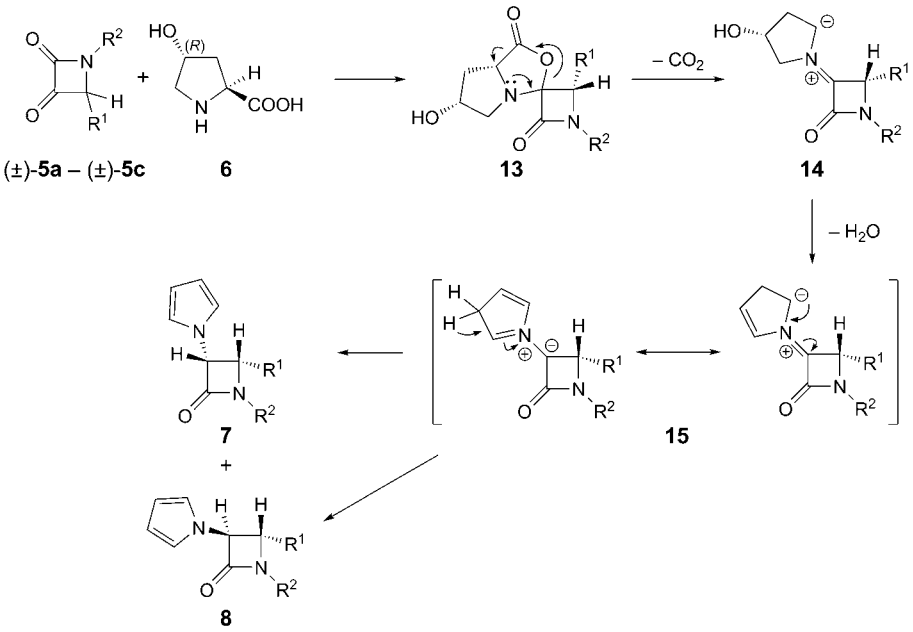
The absolute configurations of β-lactams **7** and **8** were assigned on the basis of their optical rotation data. The optical rotation for **7** was found to be $[\alpha]_D^{20.1} = +10.8$ ($c = 2.0$, $CHCl_3$), and that of compound **8** was $[\alpha]_D^{20.1} = +2.08$ ($c = 1.0$, $CHCl_3$). It has been established that (*S*)-configuration at C(4) of a β-lactam gives a positive optical rotation [19]. The absolute configurations of **7** and **8** were also confirmed by NMR comparison with those of known analogs using optically active shift reagents [19b–19d]. It should be noted that both products were obtained with (*S*)-configuration at C(4), indicating that (*R*)- and (*S*)-**5a** or **13** undergo racemization, and only the (4*S*)-configured **5a** or **13** react with **6** to give the products. Considering the yields and isomeric ratio of the products, we assume that **5b** and **5c** follow similar pathways.

The NH group in **6** is ideally located to undergo a nucleophilic addition reaction with the highly reactive C=O group of the α-keto β-lactams **5a**–**5c** in the presence of $Bi(NO_3)_3 \cdot 5\,H_2O$ to afford **13** *via* a series of unstable intermediates [20] (*Scheme 3*). In the presence of an acid catalyst, the intermediate (probably the amino alcohol) resulting from a nucleophilic reaction reacts with the carboxy group spontaneously to form the cyclic intermediate **13**. The hydroxy-proline derivative **6** reacts with the C=O group of **5** from the opposite side of the *C(4)*-substituent R^1 present in the β-lactam ring, probably because of steric hindrance. This nucleophilic reaction and subsequent ring formation step at C(3) is crucial, since it dictates the absolute configuration at C(4) of as shown in **13**. At reflux temperature, intermediate **13** can form an azomethine ylide **14** *via* decarboxylation, and this ylide then is converted to **15** *via* elimination of H_2O, because of conjugative effects [18]. Proton transfer from **15** may then occur to afford the products **7** and **8** (also **9** to **12**) to gain aromatic character [18]. The *cis*/*trans*-isomer ratios have been confirmed by ^{1}H-NMR. The fragment in the mesomeric structures of **15** involved in the proton transfer is planar, and, as a result, protonation at the anionic center C(3) may occur from the front or the reverse side. The minor *trans*-isomer **8** could also be formed because of thermal isomerization of **7**, since the *trans*-isomer is

[4]) For synthesis of these related compounds, see [18].

thermodynamically more stable. However, this pathway was ruled out, since no isomerization of pure **7** was observed, when it was treated with $Bi(NO_3)\cdot 5\,H_2O$ in the presence of EtOH at reflux temperature for 2 h. We assume that an identical pathway is conceivable with other substrates for the preparation of **9**–**12**.

Scheme 3



Conclusions. – These results indicate the capability of $Bi(NO_3)_3$ as an acid activator in catalyzing several processes. Optically active β-lactams described herein are new[5]). The success of this method would be extremely useful to investigate the reactions between other substituted α-keto β-lactams and hydroxy-proline derivatives.

Experimental Part

General. Anal. grade chemicals (*Sigma–Aldrich*) were used. Silica gel (SiO_2) was used for column chromatography (CC). Deionized H_2O was used for the preparation of all aq. solns. M.p.: *Fisher Scientific electrochemical Mel-Temp** manual melting-point apparatus (Model *1001*). Optical Rotation: *Rudolph Polarimeter*. FT-IR Spectra: *Bruker IFS 55 Equinox FT-IR* spectrophotometer, as KBr discs. ^{1}H- (300 MHz) and ^{13}C-NMR (75.4 MHz) spectra: at r.t.; *JEOL Eclipse-300* equipment; TMS as internal standard; $CDCl_3$ as solvent.

*Synthesis of 3-(1*H*-Pyrrol-1-yl)-Substituted β-Lactams* **7**–**12**. 3-Keto β-lactam **5a** (100 mg, 0.42 mmol) was suspended in 2 ml of EtOH under N_2 [16]. trans-*4-Hydroxy*-L-*proline* (**6**; 60 mg, 0.46 mmol) and $Bi(NO_3)_3\cdot 5\,H_2O$ (20 mg, 0.04 mmol) were added to the mixture. The mixture was

[5]) Recently, *Tidwell* and co-workers have reported the synthesis of racemic pyrrolyl-substituted β-lactams using 2-(pyrrol-2-yl)acetic acid and imine by cycloaddition; *e.g.*, see [21].

heated to reflux for 3 h. The TLC indicated formation of two new products. Then, the mixture was diluted with H_2O (10 ml) and CH_2Cl_2 (25 ml). The CH_2Cl_2 layer was separated, washed with aq. $NaHCO_3$ soln. (5 ml), and dried (Na_2SO_4). The CH_2Cl_2 layer was evaporated under reduced pressure to give a crude pale-yellow oil (85 mg, 83%). The ^{1}H-NMR spectrum of the crude indicated a mixture of two diastereoisomers: *cis* (78%) and *trans* (22%). The crude mixture was separated chromatographically (5–10% AcOEt/hexane) to afford pure *trans*-isomer **8** as colorless oil, followed by *cis*-isomer **7** as a white solid. The structures of **7** and **8** were confirmed by spectroscopic and anal. data. An identical procedure was applied for the preparation of compounds **9**–**12** (yield of **7**+**8** 219.2 mg (80%); of **9**+**10** 135 mg, and of **11**+**12** 130 mg (85%)).

*(3*R,*4*S*)-1,4-Diphenyl-3-(1*H*-pyrrol-1-yl)azetidin-2-one* (**7**). White solid. R_f (10% AcOEt/hexane) 0.52. M.p. 138–139°. $[\alpha]_D^{20.1} = +10.8$ ($c = 2.0$, $CHCl_3$). IR (KBr): 1387, 1499, 1599,1746, 2365, 2921. ^{1}H-NMR: 5.42 (*d*, $J = 5.5$, H–C(4)); 5.76 (*d*, $J = 5.5$, H–C(3)); 5.88 (*t*, $J = 2.1$, 2 H); 6.47 (*t*, $J = 2.1$, 2 H); 7.13–7.71 (*m*, 10 arom. H). ^{13}C-NMR: 61.1; 68.2; 109.2; 117.6; 119.5; 120.2; 128.7; 129.0; 131.0; 132.5; 137.2; 161.5; 167.8.

*(3*S,*4*S*)-1,4-Diphenyl-3-(1*H*-pyrrol-1-yl)azetidin-2-one* (**8**). Colorless oil. R_f (10% AcOEt/hexane) 0.82. $[\alpha]_D^{20.1} = +2.08$ ($c = 1.0$, $CHCl_3$). IR (KBr): 1242, 1510, 1745, 2360, 2967. ^{1}H-NMR: 4.96 (*d*, $J = 2.0$, H–C(4)); 5.13 (*d*, $J = 2.0$, H–C(3)); 6.25 (*t*, $J = 2.2$, 2 H); 6.76 (*t*, $J = 2.2$, 2 H); 7.25–7.68 (*m*, 10 arom. H). ^{13}C-NMR: 61.0; 68.2; 108.9; 109.5; 119.0; 121.0; 128.7; 129.5; 131.5; 132.5; 140.0; 161.7; 167.8.

*(3*R,*4*S*)-1,4-Bis(4-methoxyphenyl)-3-(1*H*-pyrrol-1-yl)azetidin-2-one* (**11**). White solid. R_f (10% AcOEt/hexane) 0.48. M.p. 142–143°. $[\alpha]_D^{20.1} = +10.11$ ($c = 1.3$, $CHCl_3$). IR (KBr): 1247, 1513, 1748, 2400, 2999. ^{1}H-NMR: 3.70, 3.76 (2*s*, 2 MeO); 5.32 (*d*, $J = 5.2$, H–C(4)); 5.69 (*d*, $J = 5.2$, H–C(3)); 5.90 (*t*, $J = 2.1$, 2 H); 6.48 (*t*, $J = 2.1$, 2 H); 6.67–7.36 (*m*, 8 arom. H). ^{13}C-NMR: 54.8; 55.3; 61.0; 68.0; 109.1; 113.6; 114.2; 114.7; 118.8; 119.0; 119.8; 120.6; 124.4; 127.8; 128.2; 130.9; 156.6; 159.8; 161.3; 167.9.

*(3*S,*4*S*)-1,4-Bis(4-methoxyphenyl)-3-(1*H*-pyrrol-1-yl)azetidin-2-one* (**12**). Colorless oil. R_f (10% AcOEt/hexane) 0.75. $[\alpha]_D^{20.1} = +19.09$ ($c = 0.3$, $CHCl_3$). IR (KBr): 1244, 1391, 1510, 1746, 2890. ^{1}H-NMR: 3.74, 3.80 (2*s*, 2 MeO); 4.88 (*d*, $J = 2.0$, H–C(4)); 5.06 (*d*, $J = 2.0$, H–C(3)); 6.24 (*t*, $J = 2.1$, 2 H); 6.74 (*t*, $J = 2.1$, 2 H); 6.77–7.71 (*m*, 8 arom. H). ^{13}C-NMR: 55.2; 56.0; 60.8; 68.5; 109.7; 113.5; 114.6; 114.8; 118.5; 119.3; 119.8; 120.0; 125.4; 127.3; 128.8; 130.9; 132.5; 156.0; 159.8; 160.0; 167.9.

*(3*R,*4*S*)-1-(4-Methoxyphenyl)-4-phenyl-3-(1*H*-pyrrol-1-yl)azetidin-2-one* (**9**). White solid. M.p. 158–159°. R_f (10% AcOEt/hexane) 0.44. $[\alpha]_D^{20.1} = +10.0$ ($c = 2.0$, $CHCl_3$). IR (KBr): 1171, 1242, 1296, 1389, 1453, 1745, 2360, 2967. ^{1}H-NMR: 3.76 (*s*, MeO); 5.38 (*d*, $J = 5.2$, H–C(4)); 5.74 (*d*, $J = 5.2$, H–C(3)); 5.87 (*t*, $J = 1.9$, 2 H); 6.46 (*t*, $J = 1.9$, 2 H); 7.12–7.71 (*m*, 9 arom. H). ^{13}C-NMR: 55.7; 62.2; 67.8; 108.8; 109.2; 113.6; 114.3; 114.8; 118.8; 120.6; 126.9; 128.2; 128.4; 132.6; 156.7; 161.2; 167.8.

*(3*S,*4*S*)-1-(4-Methoxyphenyl)-4-phenyl-3-(1*H*-pyrrol-1-yl)azetidin-2-one* (**10**). Colorless oil. R_f (10% AcOEt/hexane) 0.77. $[\alpha]_D^{20.1} = +3.75$ ($c = 0.33$, $CHCl_3$). IR (KBr): 1240, 1389, 1510, 1745, 2340, 2967. ^{1}H-NMR: 3.76 (*s*, MeO); 4.93 (*d*, $J = 1.9$, H–C(4)); 5.10 (*d*, $J = 1.9$, H–C(3)); 6.25 (*t*, $J = 2.1$, 2 H); 6.76 (*t*, $J = 2.1$, 2 H); 6.82–7.68 (*m*, 9 arom. H). ^{13}C-NMR: 53.5; 65.6; 70.4; 109.6; 113.4; 119.4; 126.5; 128.0; 128.5; 131.9; 156.6; 161.7; 166.08.

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