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Synthesis and Crystal Structure Analysis of Monocyclic β-Lactam Derivatives

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Abstract

Single crystals of monocyclic β-lactam derivatives, **I** and **II** were grown by slow evaporation method at room temperature. Crystallographic data set for **I** ($C_{24}H_{23}NO_3$) was collected with D8 Venture Dual Source 100 CMOS diffractometer at 100K using φ and ω scans methods while that for **II** ($C_{32}H_{26}N_2O_2$) was collected with a Nonius Kappa CCD diffractometer. Single crystal X-ray diffraction analysis revealed that both compounds I and II crystallize in monoclinic crystal system with space group $P2_1/c$. The final R-factors for model structures were converged to 3.79 and 5.72%, respectively.

Keywords: β-lactams; Antibiotics; Crystal structure

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Introduction

The four membered β-lactam ring constitutes an important class of heterocyclic compounds as it is a key pharmacophoric feature of the several antibiotic families such as penicillins, cephalosporins, carbapenems and monobactams **(Figure 1)** [1]. Apart from its wide therapeutic potential, β-lactam core is well explored as a versatile synthon for the preparation of a variety of natural products such as α, β- amino acids, amino sugars, alkaloids and toxoids [2]. In view of the large potential applications of β-lactam derivatives and in extension to our efforts to develop diverse biologically active scaffolds [3-5], crystal structure studies of two monocyclic β-lactam derivatives (I and II) are elaborated.

Experimental

General procedure for the synthesis of β-lactams (I and II)

Initially, Imines were synthesized using the previously reported method [3] by the condensation of aryl aldehyde (1.0 mmol) and substituted benzylamine (1.0 mmol) in anhydrous ethanol. These imines (1.0 mmol) were then treated with phenylacetic acid (1.5 mmol), triethylamine (4.0 mmol) and phosphorous oxychloride (1.1 mmol) in toluene (10 ml) at 110°C under inert atmosphere. After overnight refluxing, it was cooled to room temperature and neutralized with saturated sodium bicarbonate solution. The mixture was extracted with ethyl acetate, washed with brine and dried over anhydrous sodium sulphate. After evaporation of the solvent, the residue was purified by column chromatography using silica gel (230-400 mesh) eluted with 30-40% ethyl acetate in hexane. The title compounds were obtained in low to good yield [3]. The structures of the synthesized compounds are given in **Figure 6**.

X-ray structural study of compounds I and II

The structures of β-lactams I and II were unambiguously established by X-ray crystallographic studies. Single crystals of I and II were obtained through the slow evaporation of ethyl acetate: hexane solution. Data sets for compound I ($C_{24}H_{23}NO_3$) were collected with D8 Venture Dual Source 100 CMOS diffractometer at 100K using φ and ω scans methods. No significant loss in intensities was observed during data collection.

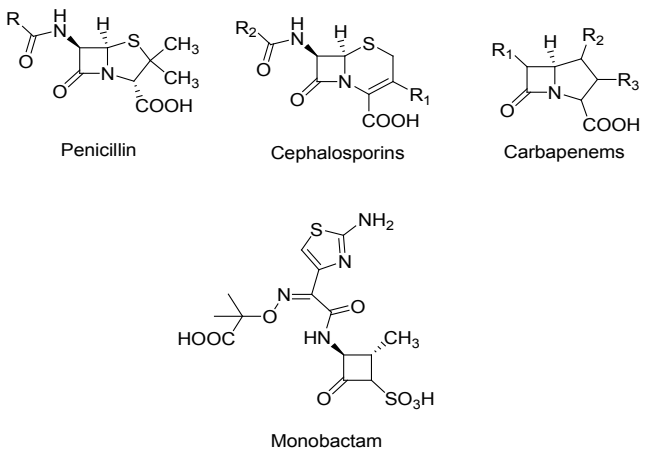

Figure 1 Structure of commonly used β-lactam antibiotics.

A total of 2184 frames were collected within the exposure time of 18.90 hours. The frames were integrated with the Bruker SAINT software package using a wide-frame algorithm resulting in to a total of 27702 reflections. Data were corrected for absorption effects using the multi-scan method (SADABS) [6]. Data collection, reduction and refinement were performed using APEX2 V2014.5-0 and SAINT V8.34A software [7]. The structure was solved and refined using the Bruker SHELXTL Software Package [8]. The data sets for compound II ($C_{32}H_{26}N_2O_2$) were collected with a Nonius Kappa CCD diffractometer. A total of 22382 reflections were collected. Data collection, data reduction, absorption correction, structure solution and refinement were performed using COLLECT [9], Denzo-SMN [10], Denzo [11], SHELXS-97 and SHELXL-97 [12] programs, respectively. The molecular graphics were prepared using XP [13].

Results and Discussion

X-ray structural study of compounds I and II

The single X-ray crystallographic study of compounds I and II exhibited the presence of various non-covalent (C-H...O, C-H...π, C-O...π and π-π) interactions which play important roles in molecular recognition, crystal engineering, foldamers and drug development [14]. Diffraction quality crystals of the compounds I and II were obtained from the ethyl acetate: hexane mixture of compounds by slow evaporation at room temperature. Total 27702 and 22382 reflections were collected for compounds I and II, out of which 3566 and 5921 reflections were independent with R_{int} values of 4.65 and 5.70%, respectively. Crystal structure analysis revealed that both compounds I and II crystallize in monoclinic crystal system with space group $P2_{1/}c$. The final R-factors for model structures were converged to 3.79 and 5.72%, respectively. The data collection and structure refinement details are provided in **Table 1**. The bond lengths and bond angles of I and **II** are listed in **Tables 2, 3, 5** and **6**. The relevant hydrogen bond details of **I** and **II** are given in **Tables 4** and **7.** ORTEP diagrams of the compounds **I** and II with ellipsoids drawn at 30% probability level along with their atomic numbering schemes are shown in **Figures 2** and **4**, respectively. Crystal structure analysis of compound I showed that the aromatic rings attached to the β-lactam rings are planer while the four membered β-lactam rings is nearly planer. The absence of puckering of the rings is also indicated through Cremer and Pople analysis [15]. The unit cell of compound II contains four molecules of similar conformations connected through multiple weak intermolecular non-covalent C-H...O interactions, two molecules of which form molecular dimers through C_{26}–H_{26}...O_1 interactions (symmetry position 1-X, -Y, -Z) (**Figure 3).** Various C-H...π (C_{14}-H_{14}...$Cg_{(4)}$, C_{16}-H_{16}...$Cg_{(1)}$, C_{22}-H_{22}...$Cg_{(4)}$, C_{26}-H_{26}...$Cg(_1)$, C_{27}-H_{27}B...$Cg_{(3)}$), C_{28}-H28...Cg(3)) and C-O...π (C1-O1...Cg(1)) interactions [16] are also observed in the packing, with the neighboring molecules at different symmetric positions. Here Cg(1), Cg(2), Cg(3) and Cg(4) represent the centroids of the rings N_1-C_1-C_2-C_3, C_{11}-C_{12}-C_{13}-C_{14}-C_{15}-C_{16}, C_{21}-C_{22}-C_{23}-C_{24}-$_{C25}$-C_{26} and C_{31}-C_{32}-C_{33}-C_{34}-C_{35}-C_{36} respectively. The crystal structure analysis of compound **II** showed that all the benzene rings are planar; four membered β-lactam ring is almost planar, while nine membered indole ring shows deviations from the planarity. It has been observed that the indole ring is puckered to form a twisted boat like structure [17]. Crystal packing of compound II also exhibits the presence of four molecules in unit cell, having similar conformations forming two pairs. The molecules of each pair are connected through bifurcated C-H...O hydrogen bonding interactions (**Figure 5)**, The pairs of molecules are flanked through π...π and C-H...π interactions to form sheet like structure.

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Additional Information

Crystallographic data for the compounds I and II with CCDC1059749, 1059750 have been reported in this article. This data can be obtained free of charge from the Cambridge Crystallographic Data Centre via https://summary.ccdc.cam.ac.uk/structure-summary-form.

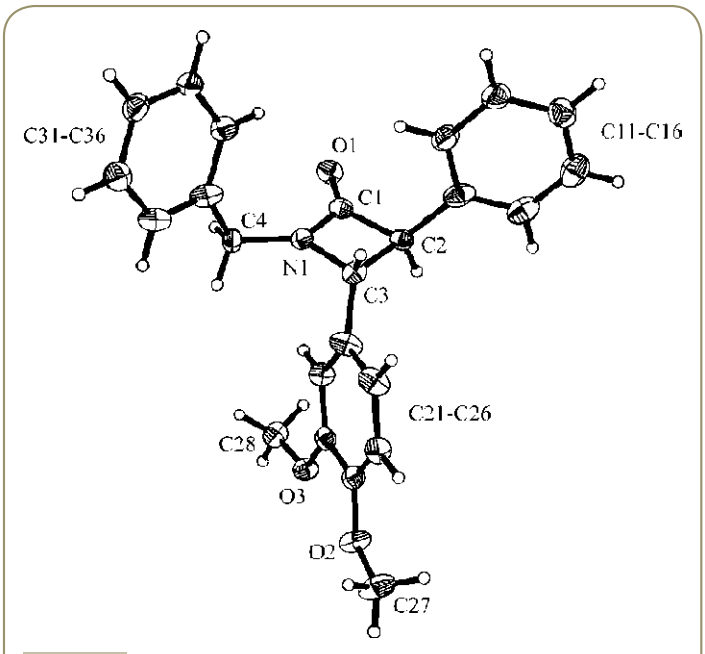

Figure 2 ORTEP diagram of compound **I** with atomic numbering scheme (30% probability level of thermal ellipsoids).

Table 1 Crystal data and structure refinement details for I and II.

Parameters	I	II
Chemical formula	$C_{24}H_{23}NO_3$	$C_{32}H_{26}N_2O_2$
Formula weight	373.43 g/mol	470.55 g/mol
Temperature	100(2) K	223(2) K
Wavelength	1.54178 Å	0.71073 Å
Crystal size	0.108 × 0.189 × 0.327 mm	0.17 × 0.15 × 0.04 mm
Crystal habit	Colourless prism	Colourless prism
Crystal system	monoclinic	monoclinic
Space group	$P2_1/c$	$P2_1/c$
Unit cell dimensions	a = 12.1489(2) Å b = 10.4560(2) Å c = 16.4403(3) Å α =90°	a = 8.5869(1) Å b = 32.0619(5) Å c = 9.0274(2) Å α =90°
Volume	β = 104.6950(10)° γ = 90° 2020.08(6) $Å^3$	β = 99.802(1)° γ = 90° 2449.07(7) $Å^3$
Z	4	4
Density (calculated)	1.228 g/cm^3	1.276 g/cm^3
Absorption coefficient (μ)	0.645 mm^{-1}	0.080 mm^{-1}
F(000) Reflections collected Independent reflections GoF (F^2) Final R indices [I>2σ(I)] R indices (all data)	792 27702 3566 [R_{int}=0.465] 1.036 0.0379 0.0451	992 22382 5971 [R_{in}t=0.057] 1.041 0.0572 0.0796
ϑ range (°)	5.06 –66.59	4.39-28.13
Index Range	-14 ≤ h ≤ 14, -12 ≤ k ≤ 12, -19 ≤ l ≤ 19	0 ≤ h ≤ 11, 0 ≤ k ≤ 42, -11 ≤ l ≤ 11
Refinement Method	Full matrix least squares on F^2	Full matrix least squares on F^2
Data/Restraints/parameters	3566/108/310	5921/0/325
Max. and Min. electron density(e.$Å^{-3}$)	0.180 and -0.199	0.218 and -0.201

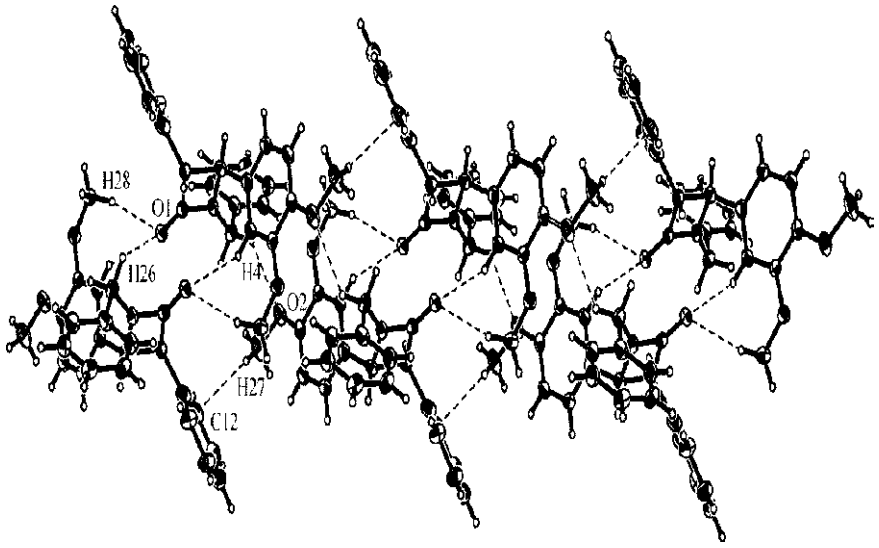

Figure 3 Crystal packing diagram of compound **I** (Hydrogen bonding is shown by dotted lines).

Table 2 Bond lengths (Å) for compound I.

O_2-C2$_4$	1.3658(16)	O_2-C_{27}	1.4312(17)
O_3-C_{25}	1.3659(15)	O_3-C_{28}	1.4233(16)
C_{11}-C_{12}	1.383(2)	C_{11}-C_{16}	1.390(2)
C_{11}-C_{4A}	1.406(8)	C_{11}-C_2	1.565(4)
C_{21}-C_{22}	1.377(2)	C_{21}-C_{26}	1.4060(18)
C_{21}-C_3	1.486(3)	C_{21}-C_{3A}	1.636(7)
C_{31}-C_{36}	1.382(2)	C_{31}-C_{32}	1.396(2)
C_{31}-C_4	1.489(3)	C_{31}-C_{2A}	1.632(7)
N_1-C_1	1.350(4)	N_1-C_4	1.452(3)
N_1-C_3	1.479(3)	C_4-H_{4A}	0.99
C_4-H_{4B}	0.99	C_1-O_1	1.215(5)
C_1-C_2	1.539(5)	C_2-C_3	1.584(5)
C_2-H_2	1.0	C_3-H_3	1.0
N_{1A}-C_{1A}	1.348(9)	N_{1A}-C_{4A}	1.429(8)
N_{1A}-C_{3A}	1.456(9)	C_{4A}-H_{4C}	0.99
C_{4A}-H_{4D}	0.99	C_{1A}-O_{1A}	1.224(8)
C_{1A}-C_{2A}	1.527(9)	C_{2A}-C_{3A}	1.576(10)
C_{2A}-H_{2A}	1.0	C_{3A}-H_{3A}	1.0
C_{12}-C_{13}	1.383(2)	C_{12}-H_{12}	0.95
C_{13}-C_{14}	1.383(2)	C_{13}-H_{13}	0.95
C_{14}-C_{15}	1.383(2)	C_{14}-H_{14}	0.95
C_{15}-C_{16}	1.383(2)	C_{15}-H_{15}	0.95
C_{16}-H_{16}	0.95	C_{22}-C_{23}	1.392(2)
C_{22}-H_{22}	0.95	C_{23}-C_{24}	1.3781(19)
C_{23}-H_{23}	0.95	C_{24}-C_{25}	1.4095(18)
C_{25}-C_{26}	1.3774(18)	C_{26}-H_{26}	0.95
C_{27}-H_{27A}	0.98	C_{27}-H_{27B}	0.98
C_{27}-H_{27C}	0.98	C_{28}-H_{28A}	0.98
C_{28}-H_{28B}	0.98	C_{28}-H_{28C}	0.98
C_{32}-C_{33}	1.3842(19)	C_{32}-$_{H32}$	0.95
$_{c33}$-C_{34}	1.386(2)	C_{33}-H_{33}	0.95
C_{34}-C_{35}	1.381(2)	C_{34}-H_{34}	0.95
C_{35}-C_{36}	1.388(2)	C_{35}-H_{35}	0.95
C_{36}-H_{36}	0.95		

Table 3 Bond angles (°) of compound I.

C_{24}-O_2-C_{27}	116.91(11)	C_{25}-O_3-C_{28}	117.15(10)
C_{12}-C_{11}-C_{16}	118.29(14)	C_{12}-C_{11}-C_{4A}	108.2(4)
C_{16}-C_{11}-C_{4A}	131.7(4)	C_{12}-C_{11}-$_{C2}$	123.62(18)
C_{16}-C_{11}-C_2	117.91(18)	C_{22}-C_{21}-C_{26}	119.49(13)
C_{22}-C_{21}-C_3	118.10(16)	C_{26}-C_{21}-C_3	121.58(16)
C_{22}-C_{21}-C_{3A}	124.4(3)	C_{26}-C_{21}-C_{3A}	114.6(3)
C_{36}-C_{31}-C_{32}	119.29(13)	C_{36}-C_{31}-C_4	116.31(18)
C_{32}-C_{31}-C_4	123.90(18)	C_{36}-C_{31}-C_{2A}	126.1(3)
C_{32}-C_{31}-C_{2A}	113.2(3)	C_1-N_1-C_4	131.3(3)
C_1-N_1-C_3	96.1(3)	C_4-N_1-C_3	132.5(3)
N_1-C_4-C_{31}	110.3(2)	N_1-C_4-H_{4A}	109.6
C_{31}-C_4-H_{4A}	109.6	N_1-C_4-H_{4B}	109.6
C_{31}-C_4-H_{4B}	109.6	H_{4A}-C_4-H_{4B}	108.1
O_1-C_1-N_1	131.4(4)	O_1-C_1-C_2	135.8(3)
N_1-C_1-C_2	92.8(3)	C_1-C_2-C_{11}	118.0(2)
C_1-C_2-C_3	84.8(3)	C_{11}-C_2-C_3	121.7(2)
C_1-C_2-H_2	110.0	C_{11}-C_2-H_2	110.0
C_3-C_2-H_2	110.0	N_1-C_3-C_{21}	113.2(2)
N_1-C_3-C_2	86.3(2)	C_{21}-C_{3-C2}	120.9(2)
N_1-C_3-H_3	111.3	$C2_1$-C_3-H_3	111.3
C2-C_3-H_3	111.3	C_{1A}-N_{1A}-C_{4A}	132.7(9)
C_{1A}-N_{1A}-C_{3A}	95.1(5)	C_{4A}-N_{1A}-C_{3A}	131.8(8)
C_{11}-C_{4A}-N_{1A}	107.1(6)	C1$_1$-C_{4A}-H_{4C}	110.3
N_{1A}-C_{4A}-H_{4C}	110.3	C_{11}-C_{4A}-H_{4D}	110.3
N_{1A}-C_{4A}-H_{4D}	110.3	H_{4C}-C_{4A}-H_{4D}	108.6
O_{1A}-C_{1A}-N_{1A}	131.3(10)	O_{1A}-C_{1A}-C_{2A}	135.0(7)
N_{1A}-C_{1A}-C_{2A}	93.6(7)	C_{1A}-C_{2A}-C_{3A}	83.7(6)
C_{1A}-C_{2A}-C_{31}	120.8(5)	C_{3A}-C_{2A}-C_{31}	123.0(5)
C_{1A}-C_{2A}-H_{2A}	109.0	C_{3A}-C_{2A}-H_{2A}	109.0
C_{31}-C_{2A}-H_{2A}	109.0	N_{1A}-C_{3A}-C_{2A}	87.6(6)
N_{1A}-C_{3A}-C_{21}	109.4(5)	C_{2A}-C_{3A}-C_{21}	125.4(5)
N_{1A}-C_{3A}-H_{3A}	110.6	C_{2A}-C_{3A}-H_{3A}	110.6
C_{21}-C_{3A}-H_{3A}	110.6	C_{13}-C_{12}-C_{11}	120.88(14)
C_{13}-C_{12}-H_{12}	119.6	C1$_1$-C_{12}-H_{12}	119.6
C_{14}-C_{13}-C_{12}	120.47(14)	C_{14}-C_{13}-H_{13}	119.8
C_{12}-C_{13}-H_{13}	119.8	C_{13}-C_{14}-C_{15}	119.21(14)
$_{C13}$-C_{14}-H_{14}	120.4	C_{15}-C_{14}-H_{14}	120.4
C_{14}-C_{15}-C_{16}	120.13(14)	C_{14}-C_{15}-H_{15}	119.9
C_{16}-C_{15}-H_{15}	119.9	C_{15}-C_{16}-C_{11}	121.02(14)
C_{15}-C_{16}-H_{16}	119.5	C_{11}-C_{16}-H_{16}	119.5
C_{21}-C_{22}-C_{23}	120.73(13)	C_{21}-C_{22}-H_{22}	119.6
C_{23}-C_{22}-H_{22}	119.6	C_{24}-C_{23}-C_{22}	119.97(12)
C_{24}-C_{23}-H_{23}	120.0	C_{22}-C_{23}-H_{23}	120.0
O_2-C_{24}-C_{23}	125.27(12)	O_2-C_{24}-C_{25}	114.85(11)
C_{23}-C_{24}-C_{25}	119.87(12)	O_3-C_{25}-C_{26}	125.15(11)
$_{O3}$-C_{25}-C_{24}	115.04(11)	C_{26}-C_{25}-C_{24}	119.80(11)
C_{25}-C_{26}-C_{21}	120.12(12)	C_{25}-C_{26}-H_{26}	119.9
C_{21}-C_{26}-H_{26}	119.9	O_2-C2$_7$-H_{27A}	109.5
O_2-C_{27}-H_{27B}	109.5	H_{27A}-$_{C27}$-H_{27B}	109.5
O_{2}-C_{27}-H_{27C}	109.5	H_{27A}-C_{27}-H_{27C}	109.5
H_{27B}-C_{27}-H_{27C}	109.5	O_3-C_{28}-H_{28A}	109.5
O_3-C_{28}-H_{28B}	109.5	H_{28A}-C_{28}-H_{28B}	109.5
O_3-C2$_8$-H2$_{8C}$	109.5	H_{28A}-C_{28}-H_{28C}	109.5
H_{28B}-C_{28}-H_{28C}	109.5	C_{33}-C_{32}-C_{31}	120.05(13)
C_{33}-C_{32}-H_{32}	120.0	C_{31}-C_{32}-H_{32}	120.0
C_{32}-C_{33}-C_{34}	120.10(13)	C_{32}-C_{33}-H_{33}	119.9
C_{34}-C_{33}-H_{33}	119.9	C_{35}-C_{34}-C_{33}	120.12(13)
C_{35}-C_{34}-H_{34}	119.9	C_{33}-C_{34}-H_{34}	119.9
C_{34}-C_{35}-C_{36}	119.72(14)	C_{34}-C_{35}-H_{35}	120.1
C_{36}-C_{35}-H_{35}	120.1	C_{31}-C_{36}-C_{35}	120.72(13)
C_{31}-C_{36}-H_{36}	119.6	C_{35}-C_{36}-$_{H36}$	119.6

Table 4 Hydrogen bond distances (Å) and angles (°) of compound II.

	Donor-H	Acceptor-H	Donor-Acceptor	Angle
C_4-H_{4A}···O_2	0.99	2.50	3.152(4)	123.5
C_{26}-H26···O_1	0.95	2.49	3.175(2)	129.4
C_{27}-H_{27A}···O_{1A}	0.98	2.29	3.258(6)	169.6
C_{28}-H_{28C}···O_1	0.98	2.44	3.342(3)	153.5

Table 5 Bond lengths (Å) of compound II.

$O_{(1)}$-$C_{(1)}$	1.212(2)	$C_{(21)}$-$C_{(22)}$	1.388(2)
$O_{(2)}$-$C_{(13)}$	1.218(2)	$C_{(21)}$-$C_{(26)}$	1.391(2)
$N_{(1)}$-$C_{(1)}$	1.355(2)	$C_{(22)}$-$C_{(23)}$	1.387(2)
$N_{(1)}$-$C_{(4)}$	1.460(2)	$C_{(22)}$-$H_{(22)}$	0.94
$N_{(1)}$-$C_{(3)}$	1.481(2)	$C_{(23)}$-$C_{(24)}$	1.381(3)
$N_{(2)}$-$C_{(13)}$	1.396(2)	$C_{(23)}$-$H_{(23)}$	0.94
$N_{(2)}$-C(6)	1.402(2)	$C_{(24)}$-$C_{(25)}$	1.378(3)
$N_{(2)}$-$C_{(7)}$	1.414(2)	$C_{(24)}$-$H_{(24)}$	0.94
$C_{(1)}$-$C_{(2)}$	1.542(2)	$C_{(25)}$-$C_{(26)}$	1.386(3)
$C_{(2)}$-$C_{(21)}$	1.504(2)	$C_{(25)}$-$H_{(25)}$	0.94
$C_{(2)}$-$C_{(3)}$	1.568(2)	$C_{(26)}$-$H_{(26)}$	0.94
$C_{(2)}$-$H_{(2)}$	0.99	$C_{(31)}$-$C_{(36)}$	1.383(3)
$C_{(3)}$-$C_{(5)}$	1.487(2)	$C_{(31)}$-$C_{(32)}$	1.389(3)
$C_{(3)}$-$H_{(3)}$	0.99	$C_{(32)}$-$C_{(33)}$	1.379(3)
$C_{(4)}$-$C_{(41)}$	1.509(3)	$C_{(32)}$-$H_{(32)}$	0.94
$C_{(4)}$-$H_{(4A)}$	0.98	$C_{(33)}$-$C_{(34)}$	1.370(3)
$C_{(4)}$-$H_{(4B)}$	0.98	$C_{(33)}$-$H_{(33)}$	0.94
$C_{(5)}$-$C_{(6)}$	1.344(2)	$C_{(34)}$-$C_{(35)}$	1.376(3)
$C_{(5)}$-$C_{(12)}$	1.451(2)	$C_{(34)}$-$H_{(34)}$	0.94
$C_{(6)}$-$H_{(6)}$	0.94	$C_{(35)}$-$C_{(36)}$	1.391(3)
$C_{(7)}$-$C_{(8)}$	1.390(3)	$C_{(35)}$-$H_{(35)}$	0.94
$C_{(7)}$-$C_{(12)}$	1.403(2)	$C_{(36)}$-$H_{(36)}$	0.94
$C_{(8)}$-$C_{(9)}$	1.379(3)	$C_{(41)}$-$C_{(42)}$	1.385(3)
$C_{(8)}$-$H_{(8)}$	0.94	$C_{(41)}$-$C_{(46)}$	1.386(3)
$C_{(9)}$-$C_{(10)}$	1.384(3)	$C_{(42)}$-$C_{(43)}$	1.386(3)
$C_{(9)}$-$H_{(9)}$	0.94	$C_{(42)}$-$H_{(42)}$	0.94
$C_{(10)}$-$C_{(11)}$	1.383(3)	$C_{(43)}$-$C_{(44)}$	1.375(3)
$C_{(10)}$-$H_{(10)}$	0.94	$C_{(43)}$-$H_{(43)}$	0.94
$C_{(11)}$-$C_{(12)}$	1.393(2)	$C_{(44)}$-$C_{(45)}$	1.372(3)
$C_{(11)}$-$H_{(11)}$	0.94	$C_{(44)}$-$H_{(44)}$	0.94
$C_{(13)}$-$C_{(14)}$	1.509(3)	$C_{(45)}$-$C_{(46)}$	1.388(3)
$C_{(14)}$-$C_{(31)}$	1.517(3)	$C_{(45)}$-$H_{(45)}$	0.94
$C_{(14)}$-$H_{(14A)}$	0.98	$C_{(46)}$-$H_{(46)}$	0.94
$C_{(14)}$-$H_{(14B)}$	0.98		

Table 6 Bond angles (°) of compound II.

$C_{(1)}$-$N_{(1)}$-$C_{(4)}$	132.81(15)	$C_{(13)}$-$C_{(14)}$-$H_{(14B)}$	109.3
$C_{(1)}$-$N_{(1)}$-$C_{(3)}$	95.63(12)	$C_{(31)}$-$C_{(14)}$-$H_{(14B)}$	109.3
$C_{(4)}$-$N_{(1)}$-$C_{(3)}$	128.71(14)	$H_{(14A)}$-$C_{(14)}$-$H_{(14B)}$	109.3
$C_{(13)}$-$N_{(2)}$-$C_{(6)}$	126.62(16)	$C_{(22)}$-$C_{(21)}$-$C_{(26)}$	108
$C_{(13)}$-$N_{(2)}$-$C_{(7)}$	126.05(16)	$C_{(22)}$-$C_{(21)}$-$C_{(2)}$	119.97(15)
$C_{(6)}$-$N_{(2)}$-$C_{(7)}$	107.33(14)	$C_{(26)}$-$C_{(21)}$-$C_{(2)}$	121.13(15)
$O_{(1)}$-$C_{(1)}$-$N_{(1)}$	132.21(16)	$C_{(23)}$-$C_{(22)}$-$C_{(21)}$	120.45(16)
$O_{(1)}$-$C_{(1)}$-$C_{(2)}$	135.43(15)	$C_{(23)}$-$C_{(22)}$-$H_{(22)}$	119.8
$N_{(1)}$-$C_{(1)}$-$C_{(2)}$	92.36(13)	$C_{(21)}$-$C_{(22)}$-$H_{(22)}$	119.8
$C_{(21)}$-$C_{(2)}$-$C_{(1)}$	117.25(13)	$C_{(24)}$-$C_{(23)}$-$C_{(22)}$	120.29(17)
$C_{(21)}$-$C_{(2)}$-$C_{(3)}$	116.30(13)	$C_{(24)}$-$C_{(23)}$-$H_{(23)}$	119.9
$C_{(1)}$-$C_{(2)}$-$C_{(3)}$	85.06(12)	$C_{(22)}$-$C_{(23)}$-$H_{(23)}$	119.9
$C_{(21)}$-$C_{(2)}$-$H_{(2)}$	111.9	$C_{(25)}$-$C_{(24)}$-$C_{(23)}$	119.59(17)
$C_{(1)}$-$C_{(2)}$-$H_{(2)}$	111.9	$C_{(25)}$-$C_{(24)}$-$H_{(24)}$	120.2
$C_{(3)}$-$C_{(2)}$-$H_{(2)}$	111.9	$C_{(23)}$-$C_{(24)}$-$H_{(24}$	120.2
$N_{(1)}$-$C_{(3)}$-$C_{(5)}$	116.51(13)	$C_{(24)}$-$C_{(25)}$-$C_{(26)}$	120.48(18)
$N_{(1)}$-$C_{(3)}$-$C_{(2)}$	86.72(11)	$C_{(24)}$-$C_{(25)}$-$H_{(25)}$	119.8
$C_{(5)}$-$C_{(3)}$-$C_{(2)}$	118.54(13)	$C_{(26)}$-$C_{(25)}$-$H_{(25)}$	119.8
$N_{(1)}$-$C_{(3)}$-$H_{(3)}$	111	$C_{(25)}$-$C_{(26)}$-$C_{(21)}$	120.33(17)
$C_{(5)}$-$C_{(3)}$-$H_{(3)}$	111	$C_{(25)}$-$C_{(26)}$-$H_{(26)}$	119.8
$C_{(2)}$-$C_{(3)}$-$H_{(3)}$	111	$C_{(21)}$-$C_{(26)}$-$H_{(26)}$	119.8
$N_{(1)}$-$C_{(4)}$-$C_{(41)}$	109.42(14)	$C_{(36)}$-$C_{(31)}$-$C_{(32)}$	117.9(2)
$N_{(1)-C(4)}$-$H_{(4A)}$	109.8	$C_{(36)}$-$C_{(31)}$-$C_{(14)}$	120.19(18)
$C_{(41)}$-$C_{(4)}$-$H_{(4A)}$	109.8	$C_{(32)}$-$C_{(31)}$-$C_{(14)}$	121.89(18)
$N_{(1)}$-$C_{(4)}$-$H_{(4B)}$	109.8	$C_{(33)}$-$C_{(32)}$-$C_{(31)}$	121.5(2)
$C_{(41)}$-$C_{(4)}$-$H_{(4B)}$	109.8	$C_{(33)}$-$C_{(32)}$-$H_{(32)}$	119.2
$H_{(4A)}$-$C_{(4)}$-$H_{(4B)}$	108.2	$C_{(31)}$-$C_{(32)}$-$H_{(32)}$	119.2
$C_{(6)}$-$C_{(5)}$-$C_{(12)}$	107.11(15)	$C_{(34)}$-$C_{(33)}$-$C_{(32)}$	120.0(2)
$C_{(6)}$-$C_{(5)}$-$C_{(3)}$	124.70(15)	$C_{(34)}$-$C_{(33)}$-$H_{(33)}$	120
$C_{(12)}$-$C_{(5)}$-$C_{(3)}$	128.13(15)	$C_{(32)}$-$C_{(33)}$-$H_{(33)}$	120
$C_{(5)}$-$C_{(6)}$-$N_{(2)}$	110.76(15)	$C_{(33)}$-$C_{(34)}$-$C_{(35)}$	119.5(2)
$C_{(5)}$-$C_{(6)}$-$H_{(6)}$	124.6	$C_{(33)}$-$C_{(34)}$-$H_{(34)}$	120.2
$N_{(2)}$-$C_{(6)}$-$H_{(6)}$	124.6	$C_{(35)}$-$C_{(34)}$-$H_{(34)}$	120.2
$C_{(8)}$-$C_{(7)}$-$C_{(12)}$	121.61(17)	$C_{(34)}$-$C_{(35)}$-$C_{(36)}$	120.6(2)
$C_{(8)}$-$C_{(7)}$-$N_{(2)}$	131.07(17)	$C_{(34)}$-$C_{(35)}$-$H_{(35)}$	119.7

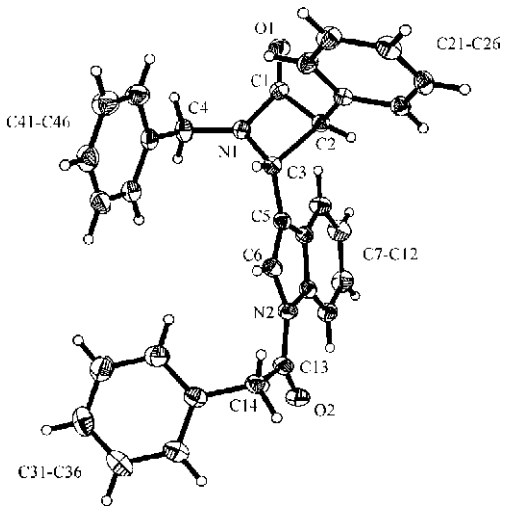

Figure 4 ORTEP diagram of compound **II** with atomic numbering scheme (30% probability level of thermal ellipsoids).

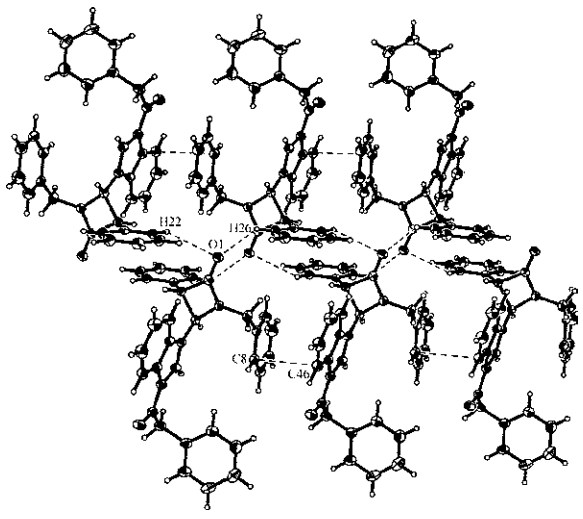

Figure 5 Crystal packing diagram of compound **II** (Hydrogen bonding is shown by dotted lines).

Table 7 Hydrogen coordinates (x 10^4) isotropic displacement parameters ($Å^2$ x 10^3) of compound II.

Atom	x	y	z	U(eq)
H(2)	5368	489	3862	38
H(3)	7827	1091	4762	39
H(4A)	8877	1067	1293	54
H(4B)	9879	662	1821	54
H(6)	6523	1794	4471	44
H(8)	1972	1895	403	56
H(9)	1215	1320	-1117	66
H(10)	2593	701	-804	65
H(11)	4782	631	1094	54
H(14A)	5656	2374	4928	62
H(14B)	4418	2743	4591	62
H(22)	4546	378	6226	43
H(23)	5050	223	8775	51
H(24)	7635	169	10036	54
H(25)	9713	252	8727	56
H(26)	9226	403	6177	50
H(32)	7750	2348	3071	80
H(33)	9540	2747	2054	85
H(34)	9251	3464	1907	74
H(35)	7167	3779	2799	76
H(36)	5335	3378	3789	63
H(42)	11646	673	4362	62
H(43)	13237	1072	6161	76
H(44)	12942	1789	6210	69
H(45)	11097	2112	4408	64
H(46)	9536	1719	2564	58

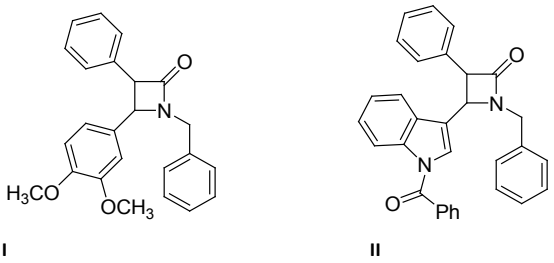

Figure 6 The structures of the synthesized β-lactams.

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