

SOLID STATE PHOTOREACTION OF N,N-DIALKYLPIRUVAMIDES:
INCLUSION COMPLEXES WITH DEOXYCHOLIC ACID OR CYCLODEXTRIN

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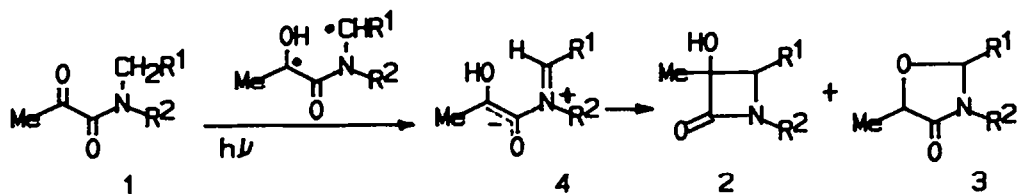
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Abstract - Solid state photolysis of inclusion molecular complexes of N,N-dialkylpyruvamides with deoxycholic acid or cyclodextrin gave the corresponding β -lactams as main products. The chemo-, stereo- and regioselectivities of the solid state reactions were different from those of the reactions in solution. Asymmetric induction due to the chirality of deoxycholic acid or cyclodextrin was observed.

The complexation of guest compounds with unimolecular hosts such as cyclodextrins,¹ crown ethers² or cyclophanes³ in solution has received much attention in relation to enzyme models. There is another type of host-guest complex which exists only in the solid state. These crystalline multimolecular inclusion complexes are known as clathrates, and they are sub-classified as the true clathrate type (Dianin's compound, cyclodextrin), the channel type (deoxycholic acid, urea) and the layer type (graphite).⁴ Recently, solid state photoreactions of the crystalline molecular inclusion complexes have attracted considerable interest. The host compounds used in these reactions are deoxycholic acid (DCA),⁵ urea,⁶ Dianin's compound,^{7,8,9} cyclodextrin,⁹ and other compounds.^{10,11}

DCA is known to form channel type molecular inclusion complexes with a variety of organic compounds, and in these complexes guest molecules are accommodated in continuous canals (channels) running through the crystals of DCA.⁵ The channel has dimensions of $\sim 4 \times 6 \text{ \AA}$ and the length varies with the size of the guest molecule.^{5,9} Lahav, Leiserowitz and their co-workers reported interesting studies on solid state photolysis of molecular complexes of DCA with ketones.⁵ In these reactions, the ketones reacted with DCA to give DCA derivatives. In relation to our previous studies on the photoreactions of N,N-dialkyl α -oxoamides in the crystalline states,¹² we have investigated the solid state photolysis of inclusion complexes of N,N-dialkylpyruvamides (1) with DCA or β -cyclodextrin (CDX), and report here full details of the study.¹³ In these reactions, the host compounds do not react with the guest compounds but instead change the chemo-, stereo- and regioselectivities of the reaction.

Photolysis of the pyruvamides (1) in solution gives two kinds of products, β -lactams (2) and oxazolidin-4-ones (3).¹⁴ The both products are produced from zwitterionic intermediates (4) which are formed by γ -hydrogen abstraction.¹⁴



Preparation of Inclusion Complexes. The DCA complexes were prepared by crystallizing DCA using the pyruvamides (1a-d) as solvents. The CDX complex of 1d was prepared by addition of 1d to a saturated solution of CDX. Meanwhile, 1a-c did not give the corresponding CDX complexes presumably owing to the hydrophilicity of the amides. The molar ratios of DCA to the amides were found to be ca 4:1, whereas that of CDX to 1d was ca 2:1 (see Experimental).

Photolysis of N,N-Symmetrically-disubstituted Pyruvamides (1a, 1b, and 1c). Solid state photolysis of the DCA complexes was carried out in the presence of air at room temperature. The results are summarized in table 1 along with those of the photoreactions in benzene. The structures of the products were determined on basis of elemental analyses and spectral data. The β -lactam (2a) was a hygroscopic liquid and converted to the 3,5-dinitrobenzoate (5a) for analysis. The β -lactam obtained in the photolysis of the complex of 1b was a mixture of two stereoisomers (2b-Z and 2b-E). They could not be separated by chromatography and the yields were calculated from the NMR spectrum of the mixture. The separation was achieved after conversion into the 3,5-dinitrobenzoates (5b-Z and 5b-E). The β -lactam (2b-Z) was isolated in the case of photolysis in highly dry benzene since 2b-E was not formed in this reaction. The stereochemistry of the two β -lactams (2b-Z and 2b-E) was assigned on the basis of the fact that more stable Z-isomers are produced almost exclusively in the photolysis of α -oxoamides in solution^{14,15} (cf. the photoreaction of 1d). The structure of 2c-Z was confirmed by direct comparison with an authentic sample.¹⁹ The formation of 2c-E (the stereoisomer of 2c-Z) in the solid state photolysis of the complex of 1c was suggested by the NMR spectrum of the reaction mixture but the isomer could not be isolated.

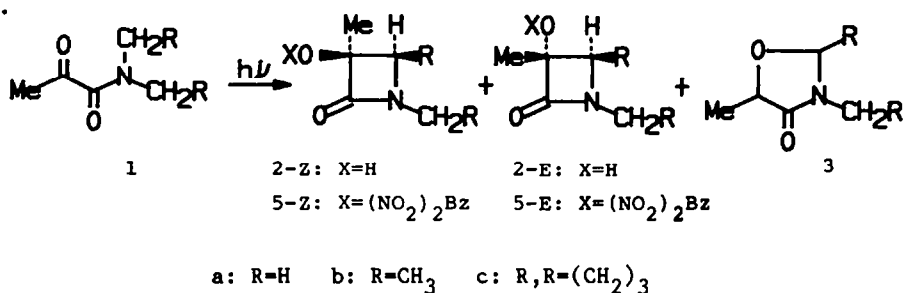
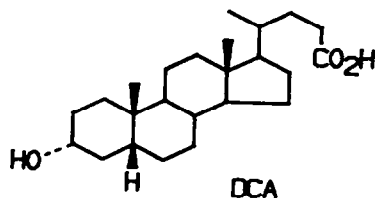


Table 1. Photolysis of 1a, 1b, and 1c.

Compound	Media	Products (%)		
		2-Z	2-E	3
1a	DCA-complex	42		10
	benzene	-		10
1b	DCA-complex	38	36	trace
	benzene	-	-	56
	benzene (dry)	22	-	39
1c	DCA-complex	52	a	15
	benzene ¹⁹	5	-	50

a: not isolated.



Photolysis of *N*-Benzyl-*N*-methylpyruvamide (1d). Photolysis of the inclusion complex of 1d with DCA or CDX was done as with the complexes of 1a-c. The results were shown in Table 2 together with that of the photolysis in benzene. Two rotational isomers, *s*-Z and *s*-E, can exist for 1d, and products formed by methyl hydrogen abstraction and benzylic hydrogen abstraction were obtained in the photolysis. The three isomers, 2d, 2d-Z and 2d-E, could not be separated by chromatography. The 3,5-dinitrobenzoate, 5d-z, was separated from 5d and 5d-E by chromatography, whereas the naphthoate, 6d, was isolated by recrystallization of the mixture of 6d and 6d-E. However, the β -lactam (2d-E) and the derivatives (5d-E and 6d-E) were not completely separated from the isomers even by repeated chromatography or recrystallization. The structure of 2d was assigned on the basis of the NMR spectra of samples containing small amounts of the isomers.

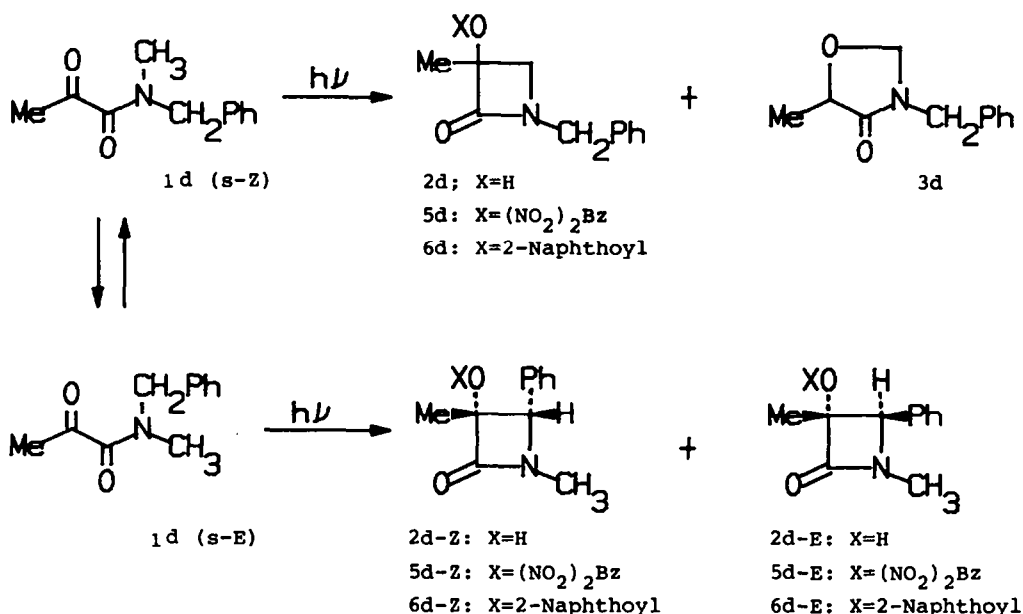


Table 2. Photolysis of *N*-Benzyl-*N*-Methylpyruvamide (1d)

	Methyl H Abst. Products (%)		Benzyl H Abst. Products (%)	
	2d	3d	2d-Z	2d-E
DCA-complex	44	0	19	14
CDX-complex	17	6	12	1
benzene	3	15	33	1

Chemoselectivity. Solid state photolysis of the inclusion complexes of 1a-c with DCA gave the β -lactams (2a-c) in good yields in contrast to the fact that the oxazolidinones (3a-c) were main products in the photolysis in benzene (Table 1). Similarly, the yield of the β -lactam (2d) in the photolysis of the DCA complex is much higher than that in the photolysis in benzene (Table 2). The β -lactams (2) and oxazolidinones (3) are produced from the zwitterionic intermediates (4) as described above (Scheme 1). It is known that protic substances increase the yield of the oxazolidinones and that formation of the β -lactams is favored in aprotic solvents.^{14,15} Since the channels of DCA crystals are hydrophobic,⁹ it is conceivable that the efficient formation of the β -lactams in the photolysis of the inclusion complexes is due to the hydrophobicity of the channels of DCA. In fact, 2b was formed in 22% in the photolysis of 1b in carefully dried benzene (Table 1). However, the yield is still much lower than that in the solid state

photolysis. These facts indicate that the restriction of the molecular motion of the zwitterionic intermediate (4) imposed by DCA molecules plays an important role in the formation of the β -lactams. Molecular models show that the β -lactam (2) is sterically smaller than the oxazolidinones. Therefore, the formation of 2 is presumed to become preferable in the restricted environment. Formation of the oxazolidinone (3d, 6%) in photolysis of the CDX complex of 1d is consistent with this explanation, since the cavity of CDX (7-11 Å diameter)⁹ is larger than the channel of DCA and the restriction of molecular motion in the CDX cavity should be weaker than that in the DCA channel. Turro, Ramamurthy *et al.* recently reported similar channel (or cavity) size effects in the photoreaction of ketones.⁹ The selective formation of β -lactams in the photolysis of some β -oxoamides in the crystalline states¹² is also compatible with the explanation. **Stereoselectivity.** Photolysis of the pyruvamides (1b-d) in solution gave the more stable Z-isomers almost exclusively, whereas the solid state photolysis of the inclusion complexes afforded considerable amounts of the E-isomers (Table 1,2). This result is also explainable in terms of the restriction of the molecular motion, since the sterically more congested E-isomers (2-E) are more compact than 2-Z. The yield of 2d-E in photolysis of the CDX complex of 1d was low as with the photolysis in benzene (Table 2). This fact is explainable by the weak restriction of molecular motion in the wide CDX cavity (*vide supra*). *de Mayo*, *Scaiano et al.* recently reported similar stereoselectivity in type II cyclization of ketones included in urea.⁶

Regioselectivity. In the case of N-benzyl-N-methylpyruvamide (1d), methyl hydrogen abstraction and benzyl hydrogen abstraction take place competitively from the two rotamers, s-Z and s-E isomers, respectively. In the photolysis in benzene, the yield of benzyl-hydrogen-abstraction products (2d-Z and 2d-E) is larger than that of methyl-hydrogen-abstraction products (2d and 3d). The reverse is true in the case of the solid state photolysis of the inclusion complex. Interconversion of the two rotamers takes place rapidly in solution, and the s-Z/s-E ratio was found to be 1.2 in benzene-d₆. The higher efficiency of the benzyl hydrogen abstraction in solution may be attributed to the higher reactivity of benzyl hydrogens toward abstraction than that of methyl hydrogens.¹⁶ However, the reason for the efficient methyl hydrogen abstraction in the solid state photolysis is not clear at present.

Enantioselectivity. Since both DCA and CDX are chiral compounds, asymmetric induction is expected to occur in the photolysis of the inclusion complexes. In fact, the β -lactams produced in the solid state photolysis are optically active. Although the enantiomeric excesses of the products from the small molecules (1a-c) were low, those from the bulky molecule (1d) are relatively high in case of the DCA complexes. The result is reasonable because the effects of the channels should be strong in the case of the large guest molecules. The enantioselectivity in the photolysis in the CDX complex of 1d was lower than that for the DCA complex. This fact indicates again the weak restriction of molecular motion in the CDX complex. Asymmetric synthesis using clathrates has not previously been reported except for the formation of optically active polymers in polymerization of 1,3-dienes included in DCA.¹⁷

Table 3. The Optical Rotations and the Enantiomeric Excesses of the β -Lactams Obtained in Solid State Photolysis of the DCA Complexes

	5a	5b-Z	5b-E	2c	5d-Z	6d	5d-Z (CDX)
e.e. (%)	16	3	9	15	43	36	9
$[\alpha]_D$ (CHCl ₃) (°)	+3.0	+0.6	+5.5	-2.6	+20.8	-11.9	+6.0

Experimental

Yields are isolated yields unless otherwise indicated. IR spectra were recorded on a JASCO IRA-1 infrared spectrometer, and NMR spectra were measured on a JEOL-100 spectrometer. Optical rotations were measured on a UNION Automatic Digital Polarimeter. A Halos 300 W high pressure mercury lamp was used as an irradiation source. Pyruvamides were prepared according to the literature.

Preparation of Inclusion Complexes. Deoxycholic acid (DCA, 5g) was dissolved in the pyruvamide (1, 4-5g) by heating. After cooling, the resulting solid was powdered with an agate mortar and pestle, and washed with ether to remove the free amides. The ratios of DCA to the amides were calculated from the weights of the inclusion complexes and the amounts of the included pyruvamides. The amides were determined by vpc (SE-30, 1m) after dissolving the complexes in methanol using benzoic acid as a standard. The host-guest ratios (DCA/1) were as follows: 1a, 4.2±0.1; 1b, 4.5±0.2; 1c, 3.9±0.2, 1d, 3.8±0.2. The inclusion complex of 1d with β -cyclodextrin was prepared and analyzed according to the literature. The CDX/1d ratio was 2.1±0.1.

General Procedure for Solid State Photolysis of the Inclusion Complexes. The inclusion complex (5 g) was placed on a glass plate and irradiated with a 300 W high pressure mercury lamp for 7-20 days with occasional mixing. The reaction was monitored by TLC. After irradiation, the DCA complex was dissolved in acetone and then DCA was precipitated by addition of hexane. After DCA had been filtered, the filtrate was evaporated and the residue was chromatographed on silica gel. In the case of the CDX complex, the products were extracted with chloroform after dissolving the complex to warm water. The yields of the products were calculated on the basis of the amounts of the inclusion complexes and the host-guest ratios.

1,3-Dimethyl-3-hydroxyazetididin-2-one (2a): bp 75 °C (3 torr)(bath temp.); IR (CHCl₃) 3310 and 1720 cm⁻¹; ¹H-NMR (CDCl₃) δ 1.53 (s, 3H, 3-Me), 1.85 (s, 3H, NMe), 3.25 and 3.38 (ABq, 2H, J=5 Hz, CH₂). Compound 2a is hygroscopic liquid and did not give satisfactory analytical data. Thus, the compound was converted to the corresponding 3,5-dinitrobenzoate (5a).

3,4-Dimethyl-1-ethyl-3-hydroxyazetididin-2-one (2b) was a mixture of two stereoisomers which could not be separated by chromatography (the total yields, 74%), and was converted to the 3,5-dinitrobenzoates (5b-Z and 5b-E). The ratio of the stereoisomers was determined by the NMR spectrum of the mixture of the two dinitrobenzoates.

7-Hydroxy-7-methyl-1-azabicyclo[4.2.0]octan-8-one (2c): mp 137-138 °C (lit., 138-139 °C).

1-Benzyl-3-hydroxy-3-methylazetididin-2-one (2d) and **1,3-dimethyl-3-hydroxy-2-phenylazetididin-2-one (2d-Z and 2d-E)** could not be separated (the total yield, 77%). These lactams were converted to the 3,5-dinitrobenzoates or 2-naphthoates for separation. The ratio of the stereoisomers were determined by the NMR spectrum of the mixture of the three dinitrobenzoates.

General Procedure for 3,5-Dinitrobenzoylation of the β -Lactam

(2). To a solution of the β -lactam (200 mg) in benzene (20 ml) was added dropwise a solution of 3,5-dinitrobenzoylchloride (500 mg) in benzene (10 ml), and then 4,4-dimethylaminopyridine (300 mg) was added to the solution. The solution was refluxed for 6 h and diluted with benzene. The solution was washed with diluted hydrochloric acid (3 N) and then with aqueous sodium hydrogen carbonate. After the solution had been dried and evaporated, the residue was chromatographed on silica gel.

1,3-Dimethyl-3-(3,5-dinitrobenzoyloxy)azetididin-2-one (5a): mp 120-122 °C; IR (CHCl₃) 1760, 1735, and 1545 cm⁻¹; NMR (CDCl₃) δ 1.84 (s, 3H, 3-Me), 2.95 (s, 3H, NMe), 3.54 and 3.80 (ABq, 2H, J=6 Hz, CH₂), 9.1-9.5 (m, 3H, Ar). Anal. Calcd for C₁₂H₁₁N₃O₆: C, 46.60; H, 3.58; N, 13.58. Found: C, 46.30; H, 3.54; N, 13.32.

1,2,3-Dimethyl-1-ethyl-3-(3,5-dinitrobenzoyloxy)azetididin-2-one (5b-Z): mp 111-112 °C; IR (CHCl₃) 1755, 1735, and 1545 cm⁻¹; NMR (CDCl₃) δ 1.26 (t, 3H, J=7 Hz, CH₂Me), 1.49 (d, 3H, J=6 Hz, 2-Me), 1.71 (s, 3H, 3-Me), 3.0-3.7 (m, 2H, CH₂), 4.07 (q, 1H, J=6 Hz, CH), 9.1-9.3 (m, 3H, Ar). Anal. Calcd for C₁₄H₁₅N₃O₇: C, 49.85; H, 4.48; N, 12.45. Found: C, 50.00; H, 4.37; N, 12.40.

1,2,3-Dimethyl-1-ethyl-3-(3,5-dinitrobenzoyloxy)azetididin-2-one (5b-E): mp 143-144 °C; IR (CHCl₃) 1755, 1735, and 1545 cm⁻¹; NMR (CDCl₃) δ 1.26 (t, 3H, J=7 Hz, CH₂Me), 1.36 (d, 3H, J=6 Hz, 2-Me), 1.91 (s, 3H, 3-Me), 3.0-3.7 (m, 2H, CH₂), 3.96 (q, 1H, J=6 Hz, CH), 9.0-9.4 (m, 3H, Ar). Anal. Calcd for C₁₄H₁₅N₃O₇: C, 49.85; H, 4.48; N, 12.45. Found: C, 49.54; H, 4.48; N, 12.21.

1,3-Dimethyl-*r*,3-(3,5-dinitrobenzoyloxy)-*c*,2-phenylazetididin-2-one (5d-Z): mp 198-199 °C; IR (CHCl₃) 1765, 1740, and 1545 cm⁻¹; NMR (CDCl₃) δ 2.07 (s, 3H, 3-Me), 2.92 (s, 3H, NMe), 4.81 (s, 1H, CH), 7.2-7.4 (m, 5H, Ar), 8.6-8.7 (m, 2H, Ar), 9.1-9.2 (m, 1H, Ar). Anal. Calcd for C₁₈H₁₅N₃O₇: C, 56.10; H, 3.92; N, 10.90. Found: C, 55.80; H, 3.75; N, 10.68.

1,3-Dimethyl-*r*,3-(3,5-dinitrobenzoyloxy)-*t*,2-phenylazetididin-2-one (5d-E) was not completely separated from 5d: NMR (CDCl₃) δ 1.38 (s, 3H, 3-Me), 2.99 (s, 3H, NMe), 5.08 (s, 1H, CH), 7.1-7.6 (m, 5H, Ar), 9.2-9.3 (m, 3H, Ar).

1-Benzyl-3-(3,5-dinitrobenzoyloxy)-3-methylazetididin-2-one (5d) was not completely separated from 5d-E: NMR (CDCl₃) δ 1.85 (s, 3H, 3-Me), 3.43 and 3.77 (ABq, 2H, J=6 Hz, NCH₂), 4.52 (s, 2H, benzyl), 7.1-7.6 (m, 5H, Ar), 9.2-9.3 (m, 3H, Ar).

Naphthoylation of the β -Lactam (2d, 2d-Z, 2d-E). Naphthoylation of the

mixture of 2d, 2d-Z and 2d-E was carried out as in the case of 3,5-dinitrobenzoylation using 2-naphthoyl chloride in place of 3,5-dinitrobenzoyl chloride. The naphthoate, 6d-Z and 6d-E, were not separated from the isomers.

1-Benzyl-3-methyl-3-(2-naphthoxy)azetid-2-one (6d) was isolated from the isomers (6d-Z and 6d-E) by recrystallization from hexane-chloroform: mp 160-162 °C; IR (CHCl₃) 1755 and 1715 cm⁻¹; NMR (CDCl₃) δ 1.82 (s, 3H, Me), 3.38 and 3.71 (ABq, 2H, J=8 Hz, NCH₂), 4.40 and 4.58 (ABq, 2H, J=15 Hz, benzyl), 7.3-8.6 (m, 12H, Ar). Anal. Calcd for C₂₂H₁₉NO₃: C, 76.50, H, 5.54; N, 4.05. Found: C, 76.09; H, 5.50; N, 4.03.

Photolysis of N-Benzyl-N-methylpyruvamide (1d) in Benzene. A solution of 1d (700 mg) in benzene (50 ml) was irradiated with a high pressure mercury lamp for 4 h. The products were isolated by flash chromatography on silica gel.

3-Benzyl-5-methyloxazolidin-4-one (3d) was not completely purified because of the instability: IR (CHCl₃) 1700 cm⁻¹; NMR (CDCl₃) δ 1.42 (d, 3H, J=7 Hz, Me), 4.36 (q, 1H, J=7 Hz, CH), 4.48 (s, 2H, benzyl), 4.84 (s, 2H, NCH₂O), 7.30 (s, 5H, Ph).

1,3-Dimethyl-r,3-hydroxy-c,2-phenylazetid-2-one (2d-Z) was separated from small amounts of concomitant isomers by recrystallization from hexane-chloroform: 173-174 °C; IR (CHCl₃) 3340 and 1750 cm⁻¹; NMR (CDCl₃) δ 1.61 (s, 3H, 3-Me), 2.82 (s, 3H, NMe), 4.49 (s, 1H, CH), 7.1-7.7 (m, 5H, Ph). Anal. Calcd for C₁₁H₁₃NO₂: C, 69.09; H, 6.85; N, 7.32. Found: C, 68.96; H, 6.87; N, 7.27.

Photolysis of the amides (1a, 1b, 1c, 1g) in benzene has been reported, and spectral data of the products (3a, 3b, 2c, and 3c) are described in these papers.

Photolysis of N,N-Diethylpyruvamide (1b) in highly dry benzene. A solution of the amide (400 mg) in dry benzene (60 ml) was placed in a Pyrex tube and 5 g of molecular sieves (4 A) was added. The tube was sealed, allowed to stand overnight, and then irradiated with a high pressure mercury lamp for 4 h. After removal of the solvent the residue was chromatographed on silica gel.

r,2,t,3-Dimethyl-1-ethyl-3-hydroxyazetid-2-one (2b-Z): mp 73-74 °C; IR (CHCl₃) 3380 and 1725 cm⁻¹; NMR (CDCl₃) δ 1.16 (t, 3H, J=7 Hz, CH₂Me), 1.22 (d, 3H, J=7 Hz, 2-Me), 1.35 (s, 3H, 3-Me), 2.8-3.6 (m, 2H, CH₂), 3.67 (q, 1H, J=7 Hz, CH), 5.22 (br s, 1H, OH). Anal. Calcd for C₇H₁₃NO₂: C, 58.71; H, 9.15; N, 9.78. Found: C, 58.84; H, 9.31; N, 9.62.

Measurement of Enantiomeric Excesses of the β-Lactams. The enantiomeric excesses were measured by NMR spectroscopy using a chiral shift reagent, Eu(tfc)₃; tfc=tris[3-(trifluoromethylhydroxymethylene)-(-)-camphorato]. The ratios of the amounts of the enantiomers were calculated from the areas obtained by integrations of the signals of the 3-methyl groups.

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