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Studies on Ketene and Its Derivatives. CXII.[1a] Reaction of Ketene with Schiff Bases to give α-Unsubstituted β-Lactams[1b]

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The reaction of ketene with Schiff bases was investigated. Heating of ketene with Schiff bases (**1a**—**l**) without solvent gave α-unsubstituted β-lactams (**2a**—**l**). The reaction of ketene with ethyl *N*-furfurylideneglycinate (**1k**) to give the β-lactam **2k** was carried out at various temperatures, and it was found that the yield of **2k** was not much influenced by the reaction temperature. β-Lactams (**2d**, **f**, **k**, **l**) were treated with 10% aqueous sodium hydroxide in dioxane to give the corresponding carboxylic acids (**4d**, **f**, **k**, **l**) in good yields. Compounds **4d**, **f**, **l** reacted with various amines in the presence of dicyclohexylcarbodiimide (DCC) to give the corresponding amides (**5a**—**c**, **8a**—**d**).

Keywords——ketene; Schiff base; α-unsubstituted β-lactam; carboxylic acid; amide; dicyclohexylcarbodiimide

The [2+2] cycloaddition reaction of ketenes to imines has been widely used for the synthesis of β-lactams.[2] Previous investigations of the reaction have dealt almost exclusively with substituted ketenes to give α-substituted β-lactams. While the reaction of ketene, $CH_2=C=O$, with imines seems to be effective for the synthesis of α-unsubstituted β-lactams, little work has been done in this field because ketene itself seems to be much less reactive than substituted ketenes.[3]

In 1917, Staudinger[4] obtained a β-lactam, 1,4-diphenyl-2-azetidinone (**2a**), by passing ketene gas over *N*-benzylideneailine (**1a**) without solvent at 180°C. Later, employing the same reaction conditions, Pfleger *et al.*[5] reported syntheses of β-lactams from ketene and various Schiff bases derived from aromatic aldehydes and aromatic amines. They also reported that the reaction of ketene with *N*-cinnamylideneaniline (**1g**) did not give the β-lactam, but gave the [2+4] cycloadduct, 2-oxo-1,4-diphenyl-1,2,3,4-tetrahydropyridine (**3**).

In view of the above results, we have investigated the reaction of ketene with Schiff bases, in particular those derived from aliphatic amines. The Schiff bases (**1a**—**l**) used in this reaction were prepared from aldehydes and amines in the usual way. The results are sum-

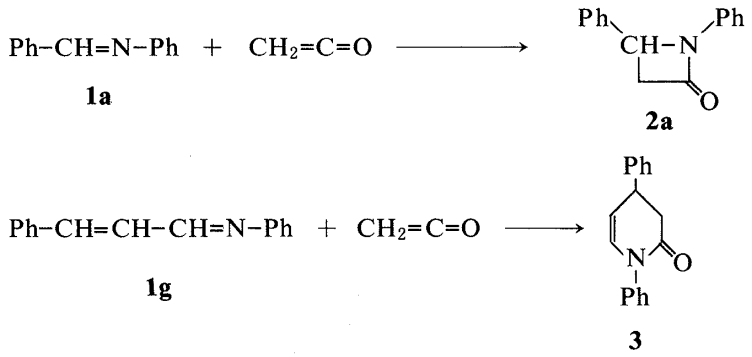


Chart 1

TABLE I. Schiff Bases (**1a**—**l**)

$$R_1\text{-CHO} + H_2N\text{-}R_2 \xrightarrow[\text{or } Et_3N/H_2O]{MgSO_4/\text{benzene}} R_1\text{-CH=N-}R_2$$

1a—**l**

Compd. No.	R_1	R_2	bp (°C) (mmHg)	Yield (%)	IR ($CHCl_3$) cm^{-1} C=N
1a	Ph	Ph	mp 51—52°C (lit.[7] mp 52°C)	95[a]	1645
1b	Ph	Cyclohexyl	140—145 (18) [lit.[8] 136 (16)]	69[a]	1650
1c	Ph	*tert*-Bu	95—98 (17—18) [lit.[9] 90—92 (11)]	72[a]	1650
1d	Ph	CH_2CO_2Et	65—75 (0.001)	68[b]	1650
1e	Ph	CH_2CO_2-*tert*-Bu	65—70 (0.001)	75[a]	1645
1f	Ph	$CH_2CH_2CO_2Et$	74—76 (0.001)	81[b]	1645
1g	Styryl	Ph	mp 106—108°C (lit.[10] mp 109 °C)	90[a]	1630
1h	Styryl	Cyclohexyl	134—140 (3) [lit.[11] 180—185 (12)]	93[a]	1645
1i	Styryl	*tert*-Bu	95—97 (3)	91[a]	1645
1j	2-Furyl	Ph	mp 58°C (lit.[12] mp 56—57°C)	93[a]	1630
1k	2-Furyl	CH_2CO_2Et	69—75 (0.001)	51[b]	1650
1l	2-Furyl	$CH_2CH_2CO_2Et$	75—85 (0.001)	68[b]	1650

a) $MgSO_4$-benzene. *b*) Et^3N-H_2O.

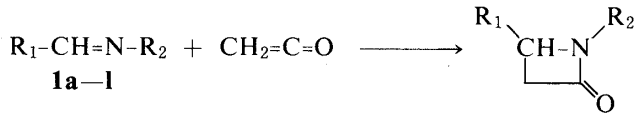
marized in Table I. Reactions were undertaken in the presence of anhydrous magnesium sulfate in benzene, while the reaction of ethyl glycinate and ethyl 3-aminopropionate hydrochlorides was carried out in the presence of triethylamine in water.

First, the reaction of ketene with *N*-benzylideneaniline (**1a**) was investigated. When ketene generated by pyrolysis of acetone[6] was bubbled into a solution of **1a** in acetone at room temperature, the reaction did not proceed and the starting material **1a** was recovered. However, according to the procedure reported by Staudinger,[4] excess ketene was passed over the anil **1a** at 180°C to give colorless needles (**2a**) of mp 155—156°C. The spectral data for **2a** are consistent with the β-lactam structure, 1,4-diphenyl-2-azetidinone. Similar reaction of ketene with *N*-cinnamylideneaniline (**1g**) gave colorless needles (**2g**), whose melting point (82—84°C) agreed approximately with that (mp 87°C) of the product obtained from the same reaction reported by Pfleger *et al.*[5] However, the structure of **2g** was assigned as 4-cinnamyl-1-phenyl-2-azetidinone from the spectral data.

Similarly, β-lactams (**2b**—**f**, **2h**—**l**) were also obtained from the reaction of ketene with Schiff bases (**1b**—**f**, **1h**—**l**). The results are summarized in Table II. The reaction of ketene with Schiff bases having an ester moiety such as **1d**, **f**, **k**,**l** derived from ethyl glycinate or 3-aminopropionate proceeded at *ca.* 100°C. The Schiff base **1e** reacted with ketene even at 50°C to give the β-lactam **2e**. The reaction of ethyl *N*-furfurylideneglycinate (**1k**) with ketene was investigated at various temperatures. The results are summarized in Table III, which shows that the yield of the β-lactam **2k** was not much influenced by the reaction temperature.

Elemental analyses and spectral data for β-lactams (**2a**—**l**) are shown in Table IV. The infrared (IR) spectrum showed the absorption band due to the β-lactam carbonyl group at 1735—1760 cm^{-1}. In the nuclear magnetic resonance (NMR) spectrum, the signals due to the β-lactam ring protons were observed as an ABX type signal. Of the methylene protons at the 3-position, the *cis* proton (H_1) with respect to the 4-substituent is observed at higher field than the *trans* proton (H_2).

TABLE II. Reaction of Ketene with Schiff Bases (**1a**—**l**)

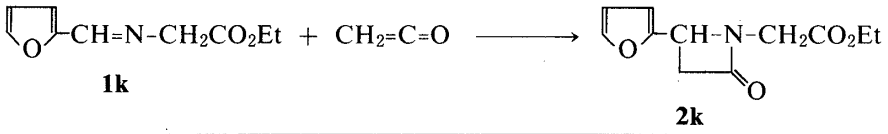


1a—**l** **2a**—**l**

	Schiff base **1**		Reaction temp. (°C)	β—Lactam **2**				
	R_1	R_2		Appearance (Recryst. solvent)	bp (°C) (0.001 mmHg)	mp (°C)	Yield (%)
1a	Ph	Ph	180	**2a** Needles (MeOH)		155—156 (lit.[1] 153—154)	29
1b	Ph	Cyclohexyl	180	**2b** Needles (hexane)		46—48 (lit.[2] 52—53)	35
1c	Ph	*tert*-Bu	150	**2c** Prisms (hexane)		63—65	32
1d	Ph	CH_2CO_2Et	95—100	**2d** Oil	122—128		45
1e	Ph	CH_2CO_2-*tert*-Bu	45—50	**2e** Needles (hexane)		66—68	38
1f	Ph	$CH_2CH_2CO_2Et$	95—100	**2f** Oil	130—135		50
1g	Styryl	Ph	180—200	**2g** Needles (hexane–ether)		82—84 (lit.[3] 87)	52
1h	Styryl	Cyclohexyl	175—180	**2h** Needles (hexane)		75—76	35
1i	Styryl	*tert*-Bu	175—180	**2i** Needles (hexane)		68—69	30
1j	2-Furyl	Ph	100	**2j** Needles (MeOH)		112—113	59
1k	2-Furyl	CH_2CO_2Et	80	**2k** Oil	80—90		50
1l	2-Furyl	$CH_2CH_2CO_2Et$	95—100	**2l** Oil	100—110		55

Eluent : **2a, e, j**; hexane–AcOEt (5 : 1), **2b, h, i**; $CHCl_3$, **2c, d, f, k, l**; benzene–$CHCl_3$ (1 : 1), **2g**; hexane–AcOEt (8 : 1).

TABLE III. Reaction of Ketene with Ethyl *N*-Furfurylideneglycinate (**1k**)



1k **2k**

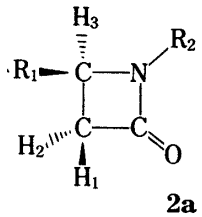
Reaction temp. (°C)	Yield (%)
150	34
120	30
95—100	34
80	50
60	43

Reaction time: 1.5 h.
Ketene gas; 0.09—0.14 mol/h.

Next, the β-lactams (**2d, f, k,l**) were hydrolyzed with 10% aqueous sodium hydroxide in dioxane, and carboxylic acids (**4d, f, k,l**) were obtained in good yields. The results are summarized in Table V.

When compounds **4d, f** were allowed to react with amines in the presence of dicyclohexylcarbodiimide (DCC) in dimethoxyethane, the amides **5a**—**c** were obtained. In this reaction, the adducts of carboxylic acids with DCC, namely urea derivatives (**6, 7**), were also

TABLE IV. Analytical and Spectral Data for Compounds (**2a**—**l**)



2a—**l**

Compd. No.	Formula	Analysis (%) Calcd (Found) C	H	N	IR ($CHCl_3$) cm^{-1} β-Lactam carbonyl	^{1}H-NMR ($CDCl_3$) δ H_1	H_2	H_3
2a	$C_{15}H_{13}NO$	80.69 (80.86	5.87 5.90	6.27 6.24)	1750	2.95 (*J*=3 Hz, *J*=15 Hz)	3.63 (*J*=6 Hz, *J*=15 Hz)	5.01 (*J*=3 Hz, *J*=6 Hz)
2b	$C_{15}H_{19}NO$	78.56 (78.71	8.35 8.17	6.11 6.20)	1740	2.75 (*J*=2 Hz, *J*=15 Hz)	3.31 (*J*=5 Hz, *J*=15 Hz)	4.58 (*J*=2 Hz, *J*=5 Hz)
2c	$C_{13}H_{17}NO$	76.81 (76.81	8.43 8.27	6.89 6.88)	1740	2.63 (*J*=2 Hz, *J*=15 Hz)	3.65 (*J*=6 Hz, *J*=15 Hz)	4.58 (*J*=2 Hz, *J*=6 Hz)
2d	$C_{13}H_{15}NO_3$	66.93 (66.72	6.48 6.52	6.01 5.89)	1758	2.88 (*J*=3 Hz, *J*=15 Hz)	3.50 (*J*=6 Hz, *J*=15 Hz)	4.88 (*J*=3 Hz, *J*=6 Hz)
2e	$C_{15}H_{19}NO_3$	68.94 (69.27	7.33 7.39	5.36 5.23)	1750	2.90 (*J*=2 Hz, *J*=15 Hz)	3.41 (*J*=5 Hz, *J*=15 Hz)	4.70 (*J*=2 Hz, *J*=5 Hz)
2f	$C_{14}H_{17}NO_3$	67.99 (68.06	6.93 7.00	5.66 5.76)	1743	2.80 (*J*=3 Hz, *J*=15 Hz)	3.36 (*J*=5 Hz, *J*=15 Hz)	4.57 (*J*=3 Hz, *J*=5 Hz)
2g	$C_{17}H_{15}NO$	81.90 (81.80	6.06 5.99	5.62 5.81)	1745	2.90 (*J*=3 Hz, *J*=15 Hz)	3.43 (*J*=6 Hz, *J*=15 Hz)	4.67 *a*)
2h	$C_{17}H_{21}NO$	79.96 (79.97	8.29 8.25	5.49 5.34)	1735	2.66 (*J*=2 Hz, *J*=15 Hz)	3.18 (*J*=5 Hz, *J*=15 Hz)	4.24 (*J*=2 Hz, *J*=5 Hz)
2i	$C_{15}H_{19}NO$	78.56 (78.85	8.35 8.34	6.11 5.91)	1740	2.51 (*J*=2 Hz, *J*=15 Hz)	3.09 (*J*=5 Hz, *J*=15 Hz)	4.20 (*J*=2 Hz, *J*=5 Hz)
2j	$C_{13}H_{11}NO_2 \cdot 1/6H_2O$	72.22 (72.54	5.25 5.11	6.48 6.45)	1735	3.16 (*J*=3 Hz, *J*=15 Hz)	3.50 (*J*=5 Hz, *J*=15 Hz)	5.02 (*J*=3 Hz, *J*=5 Hz)
2k	$C_{11}H_{13}NO_4$	59.18 (58.97	5.87 5.98	6.28 6.36)	1740	3.32 (*J*=3 Hz, *J*=15 Hz)	3.43 (*J*=5 Hz, *J*=15 Hz)	4.86 (*J*=3 Hz, *J*=5 Hz)
2l	$C_{12}H_{15}NO_4$	60.75 (60.47	6.37 6.42	5.90 5.87)	1745	3.09 (*J*=3 Hz, *J*=15 Hz)	3.34 (*J*=5 Hz, *J*=15 Hz)	4.64 (*J*=3 Hz, *J*=5 Hz)

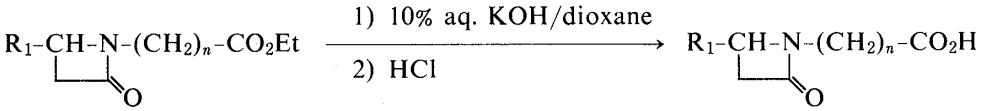
a) Due to the complexity of the spectrum, coupling constants (*J*) were difficult to determine.

obtained as by-products. The results are summarized in Table VI. Similarly; the amides **8a**—**d**, together with the urea **9**, were obtained from the carboxylic acid (**4l**).

Compound **8d** was directly prepared by heating of the ester **2l** in pyrrolidine in 53% yield. The results are summarized in Table VII.

Studies on the antibacterial activity of the β-lactams obtained and work on the synthesis of carbapenam and carbacepham from the β-lactams **2k**, **l** are in progress.

TABLE V. Hydrolysis of Esters (**2d, f, k, l**) to Carboxylic Acids (**4d, f, k, l**)

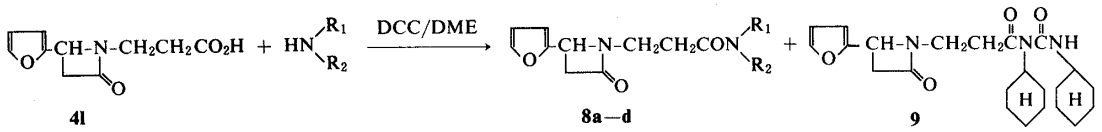


2d, f, k, l **4d, f, k, l**

Compd. No.	R_1	n	Appearance (Recryst. solvent)	Yiled (%)	Formula	Aanlysis (%) Calcd (Found) C	H	N	^{1}H-NMR ($CDCl_3$) δ -COOH
4d	Ph	1	Oil	68	$C_{11}H_{11}NO_3 \cdot 1/3CHCl3$	55.55 (55.55	4.63 4.83	5.72 5.74)	8.13
4f	Ph	2	Prisms (mp 89—91°C)	93	$C_{12}H_{13}NO_3$	65.74 (65.52	5.98 6.08	6.39 6.49)	8.80
4k	2-Furyl	1	Oil	73	$C_9H_9NO_4 \cdot 1/4H_2O$	54.14 (54.31	4.76 4.98	7.02 6.76)	8.63
4l	2-Furyl	2	Oil	77	$C_{10}H_{11}NO_4 \cdot 1/4H_2O$	56.21 (56.41	5.39 5.53	6.59 6.24)	9.50

a) Recrystallized from ether

TABLE VI. Reaction of Carboxylic Acids (**4d, f**) with Amines

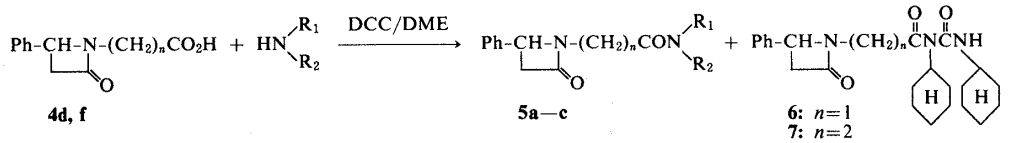


4l **8a—d** **9**

Compd. No.	R_1	R_2	Appearance	Yield (%) (Yield (%) of **9**)	Formula	Analysis (%) Calcd (Found) C	H	N	IR ($CHCl_3$) cm^{-1} -CO-N<	^{1}H-NMR ($CDCl_3$) δ Lactam 4-H
8a	h	Ph	Needles (mp 130—131°C)[a]	62 (30)	$C_{16}H_{16}N_2O_3$	67.59 (67.59	5.67 5.52	9.85 9.61)	1690	4.60 (dd) (*J*=3 Hz, *J*=5 Hz)
8b	H	$CH_2CH_2CO_2Me$	Oil	36 (58)	$C_{14}H_{18}N_2O_5$	57.13 (57.07	6.17 6.26	9.52 9.58)	1670	4.60 (dd) (*J*=3 Hz, *J*=5 Hz)
8c	$CH_2CH_2OCH_2CH_2$		Oil	71 (24)	$C_{14}H_{18}N_2O_4 \cdot 1/4H_2O$	59.47 (59.75	6.55 6.86	9.91 9.95)	1640	4.65 (dd) (*J*=3 Hz, *J*=5 Hz)
8d	$(CH_2)_4$		Oil	11 (64)	$C_{14}H_{18}N_2O_3 \cdot 1/4H_2O$	63.04 (63.17	6.94 7.07	10.51 10.53)	1630	4.72 (dd) (*J*=3 Hz, *J*=5 Hz)

a) Recrystallized from benzene.

TABLE VII. Reaction of Carboxylic Acid (**4l**) with Amines



4d, f **5a—c** **6**: n=1 **7**: n=2

Compd. No.	R_1	R_2	n	Appearance	Yield (%) (Yield (%) of **6** or **7**)	Formula	Analysis (%) Calcd (Found) C	H	N	IR ($CHCl_3$) cm^{-1} -CO-N<	^{1}H-NMR ($CDCl_3$) δ Lactam 4-H
5a	H	Ph	1	Needles (mp 138—139 C)[a]	98	$C_{17}H_{16}N_2O_2$	72.84 (73.06	5.75 5.65	9.99 10.09)	1690	4.92 (dd) (*J*=2 Hz, *J*=5 Hz)
5b	H	$CH_2CH_2CO_2Me$	1	Oil	78 (6: 14 %)	$C_{15}H_{18}N_2O_4$	62.05 (61.82	6.25 6.50	9.65 9.49)	1680	4.78 (dd) (*J*=2 Hz, *J*=5 Hz)
5c	H	$CH_2CH_2CO_2Me$	2	Oil	56 (7: 42%)	$C_{16}H_{20}N_2O_4 \cdot 1/6H_2O$	62.54 (62.79	6.62 6.84	9.12 9.09)	1670	4.58 (dd) (*J*=2 Hz, *J*=5 Hz)

a) Recrystallized from benzene–ether (1:1).

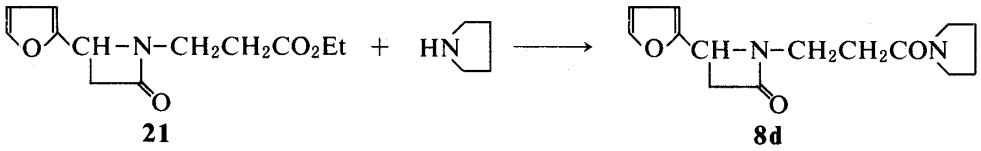


Chart 2

Experimental

Melting points and boiling points are uncorrected. IR spectra were taken on a JASCO A-101 spectrometer. ^{1}H-NMR spectra were recorded on a JEOL JNM-PMX 60 machine with tetramethylsilane as an internal standard.

General Procedure for the Synthesis of Schiff Bases (1a—l)——a) A solution of an amine (0.036 mol) in dry benzene (10 ml) was added dropwise to a mixture of an aldehyde (0.03 mol) and anhydrous magnesium sulfate (3.6 g, 0.03 mol) in dry benzene (10 ml) with stirring and ice-cooling. The mixture was stirred at room temperature for 30 min. Magnesium sulfate was filtered off, and the filtrate was concentrated to give a residue, which was purified by recrystallization or distillation under reduced pressure to afford the product.

b) Triethylamine (3.6 g, 0.036 mol) was added dropwise to a mixture of an aldehyde (0.03 mol) and ethyl glycinate hydrochloride (or ethyl 3-aminopropionate hydrochloride) (0.036 mol) in H_2O (15 ml) with stirring and ice-cooling. The mixture was stirred at room temperature for 20 min. The reaction mixture was extracted with ether. The ethereal solution was dried over anhydrous sodium sulfate. Evaporation of the ether gave an oily residue, which was distilled *in vacuo* to afford the product.

Melting and boiling points, yields, and IR spectral data are shown in Table I. Elemental analyses of new Schiff bases (**1d, e, f, i, k, l**) are given below.

Compd. No.	Formula	Analysis (%) Calcd (Found)		
		C	H	N
1d	$C_{11}H_{13}NO_2$	69.69	6.85	7.33
		(69.49	6.64	7.34)
1e	$C_{13}H_{17}NO_2$	71.15	7.75	6.38
		(70.88	7.54	6.19)
1f	$C_{12}H_{15}NO_2$	70.22	7.37	6.82
		(69.92	7.38	6.92)
1i	$C_{13}H_{17}N$	83.37	9.15	7.48
		(82.89	9.12	7.66)
1k	$C_9H_{11}NO_3 \cdot 1/5H_2O$	58.44	6.17	7.58
		(58.46	6.24	7.47)
1l	$C_{10}H_{13}NO_3$	61.52	6.71	7.18
		(61.23	6.63	7.22)

General Procedure for the Reaction of Ketene with Schiff Bases (1a—l)——According to the procedure reported by Hansford,[6] ketene gas was generated by pyrolysis of acetone using No. 26 guage chromel A-wire, and was determined by titration with 0.1 N sodium hydroxide solution. The yield of ketene was 0.09—0.14 mol/h. An excess of ketene was passed over a Schiff base (**1a—l**) (0.01—0.02 mol) without solvent under heating for 1.5 h. The reaction mixture was purified by column chromatography on silica gel to give the corresponding β-lactam (**2a—l**). The results are summarized in Tables II—IV.

General Procedure for the Hydrolysis of Esters (2d, f, k, l)——A solution of an ester (**2d, f, k,l**) (0.01 mol) in dioxane (10 ml) was added dropwise to a mixture of 10% aqueous potassium hydroxide (20 ml) and dioxane (20 ml) with stirring and ice-cooling. After being stirred at room temperature for 10 min, the reaction mixture was acidified with 10% hydrochloric acid. The resulting mixture was concentrated *in vacuo*. The residue was dissolved in chloroform. The chloroform solution was washed with water, and dried over anhydrous sodium sulfate. Evaporation of the solvent gave the residue, which was purified by column chromatography on silica gel with chloroform to give the corresponding carboxylic acid (**4d, f, k, l**). The results are summarized in Table V.

General Procedure for Reactions of Carboxylic Acids (4d, f, l) with Amines——A solution of an amine (0.002 mol) in dry dimethoxyethane (DME) (5 ml) was added to a solution of a carboxylic acid (**4d, f, l**) (0.002

mol) in dry DME (5 ml). A solution of dicyclohexylcarbodiimide (DCC) (0.0024 mol) in dry DME (5 ml) was added dropwise to the mixture with stirring and ice-cooling. Stirring was continued with ice-cooling for 30 min, then at room temperature for 3 h. The precipitated urea was filtered off, and the filtrate was concentrated *in vacuo* to give a residue, which was purified by column chromatography on silica gel with chloroform–ethyl acetate (6:1) to give the corresponding adduct (**6**, **7**, **9**). Subsequent elution with chloroform–ethyl acetate (1:6) gave the amide (**5a**—**c**, **8a**—**d**). The results are summarized in Tables VI and VII.

Compound **6**: mp 160—161°C (benzene). IR ($CHCl_3$): 1740, 1700, 1660 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 4.88 (1H, dd, J=3 and 5 Hz, lactam 4-H). *Anal.* Calcd for $C_{24}H_{33}N_3O_3 \cdot H_2O$: C, 67.73; H, 8.16; N, 9.79. Found: C, 67.50; H, 8.05; N, 10.07.

Compound **7**: foam. IR ($CHCl_3$): 1730, 1695, 1650 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 4.55 (1H, dd, J=2 and 5 Hz, lactam 4-H). *Anal.* Calcd for $C_{25}H_{35}N_3O_3 \cdot 1/2H_2O$: C, 69.12; H, 8.29; N, 9.68. Found: C, 69.11; H, 8.15; N, 9.94.

Compound **9**: mp 96—98°C (benzene). IR ($CHCl_3$): 1735, 1695, 1650 cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ: 4.63 (1H, dd, J=3 and 5 Hz, lactam 4-H). *Anal.* Calcd for $C_{23}H_{33}N_3O_4 \cdot 3/4H_2O$: C, 64.41; H, 7.65; N, 9.80. Found: C, 64.21; H, 7.51; N, 9.55.

Reaction of the Ester 2l with Pyrrolidine———A solution of **2l** (0.55 g, 0.0023 mol) in pyrrolidine (4 ml) was refluxed for 2.5 h. After removal of excess pyrrolidine *in vacuo*, the residue was subjected to column chromatography on silica gel. Elution with chloroform–ethyl acetate (1:6) gave compound **8d**, 0.32 g (53%).

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