# Diphenyl (2,3-Dihydro-2-thioxo-3-benzoxazolyl)phosphonate: A New, Reactive Condensing Agent for the Synthesis of Amides, Esters, Peptides, and β-Lactams via Condensation

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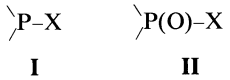
Diphenyl (2,3-dihydro-2-thioxo-3-benzoxazolyl)phosphonate (**1**) prepared from 2-benzoxazolethiol and diphenyl phosphorochloridate was proved to be a very useful condensing agent. A variety of amides, esters, and dipeptides were obtained in excellent yields. Furthermore, this reagent was successfully applied to the synthesis of β-lactams from β-amino acids.

Developing efficient and mild methods for the synthesis of amides and esters by condensation continues to be a significant aspect of organic chemistry. There have been considerable research efforts to develop a new condensing agent for promoting condensations. Most such condensing agents first react with carboxylic acids to give intermediates, such as acid anhydrides, active esters, or amides, which undergo a subsequent nucleophilic attack by amino or hydroxyl groups. Therefore, a primary factor in determining whether or not the condensing agents are effective is their ability to activate carboxylic acids.[1,2]

As part of a program directed toward the synthesis of new condensing agents for the preparation of amides, esters, and polyamides under mild conditions, we have reported on a series of such condensing agents. On examining 3-substituted 1,2-benzoisothiazole 1,1-dioxide,[3] active carbonate,[4] *N,N'*-carbonyldi[2(3*H*)-benzoxazolethione],[5] and 1,2-benzisoxazol-3-yl diphenyl phosphate,[6] we found that the phosphorus-based condensing agent is most useful for the formation of amides, esters and polyamides. Active phosphates react with the carboxylic acids to give the mixed carboxylic–phosphoric anhydrides, which play an important role in protein biosynthesis.

The methods presently available for the direct phosphorylation of carboxylic acids rely on phosphorus(III) and phosphorus(V) reagents of general structures **I** and **II**.

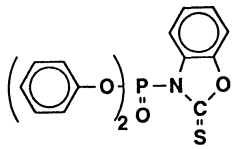


**I** **II**

X: leaving group

where X is a suitable leaving group. The effectiveness of the reagents depends upon the leaving ability of X, which in turns is related to the strength of the acid HX. In this regard, numerous condensing agents have been developed in peptide chemistry. Reagents with chlorine as a leaving group remain the most widely used condensing agents. Since phosphorus(V) reagents with chlorine as a leaving group are often not suitable for use in direct amidation, because of the formation of phosphoramide by the reaction of condensing agents with amines, a number of reactive phosphorus(V) condensing agents have been developed. These include diphenyl phosphorazidate,[7] diethyl phosphorocyanidate,[8] diphenyl succinimido phosphate,[9] and diphenyl 2-oxo-4-oxazolin-3-ylphosphonate.[10]

In a previous paper,[11] we reported that diphenyl (2,3-dihydro-2-thioxo-3-benzoxazolyl)phosphonate (**1**) is a highly efficient and mild condensing agent for the preparation of polyamides. In order to demonstrate the versatility of this condensing agent **1**, it was applied to the condensation of carboxylic acids with nucleophiles.

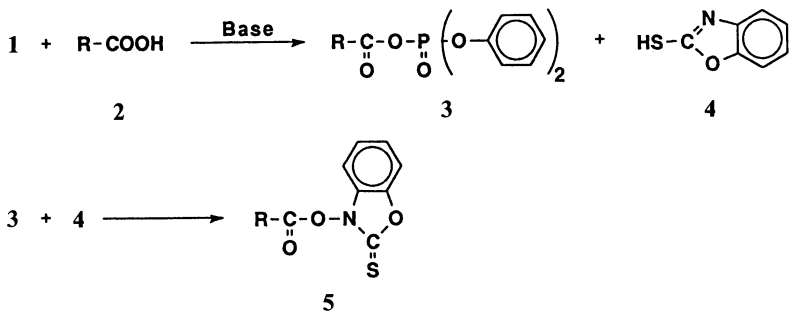


**1**

This article describes a successful synthesis of amides, esters, peptides, and β-lactams by a two-step method and a one-step procedure using condensing agent **1**.

## Results and Discussion

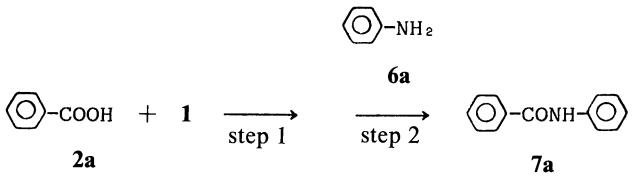
Condensation was investigated by two procedures, a two-step and a one-step procedure. The two-step procedure involves two separate steps: (1) "activation" of the carboxyl component, i.e. generation of the active intermediate from **1** and the carboxylic acid and (2) condensation of this activated carboxyl intermediate with the nucleophile. On the other hand, the one-step procedure consists of adding **1** to a solution of the carboxylic acid and the nucleophile in the presence of a base.



Scheme 1.

**Two-Step Procedure.** The condensing agent **1** reacts rapidly with benzoic acid in the presence of TEA to give 3-benzoylbenzoxazoline-2-thione in excellent yield.[11] The probable reaction pathway is as follows. The condensing agent **1** first reacts with carboxylic acid (**2**) to form carboxylic phosphoric monoanhydride (**3**), a highly activated acylating agent, which reacts rapidly with any available nucleophile 2-benzoxazolethiolate anion (**4**) to give the active amide (**5**) (Scheme 1).

The reactions of active amide **5** with amines also proceed rapidly, affording excellent yields of the corresponding amides.[11] In order to demonstrate the preparative utility of active amide **5** derived from condensing agent **1**, the conversions of carboxylic acids to amides, esters, and dipeptides were carried out using the two-step procedure. To determine the optimum conditions for condensations employing condensing agent **1**, we first studied the synthesis of benzanilide (**7a**) from benzoic acid (**2a**) and aniline (**6a**) (Scheme 2).



Scheme 2.

Table 1 shows the effect of a solvent, the amount of solvent and the amount of **1** in the presence of TEA on condensation. The condensation proceeded in a homogeneous solution, except for acetonitrile, giving excellent yields of **7a**. Two mililiters of NMP were found to be adequate for a reaction on the 1 mmol scale. A 1.1 molar amount of **1** relative to **2a** was sufficient for the reaction. Table 2 lists the effect of the activation time (step 1) and condensation time (step 2). These experiments revealed that the activation of **2a** was very fast and that the condensation came to completion in 5 min at room temperature.

**Synthesis of Amides (7), Esters (8), and Thioesters (9).** The synthesis of amides and esters using condensing agent **1** was carried out in NMP at room temperature (Scheme 3).

The reactions proceeded smoothly to give the corresponding amides **7**, esters **8** and thioesters **9** in excellent

Table 1. Effect of Solvent, the Amount of Solvent and the Amount of **1** on Condensation[a]

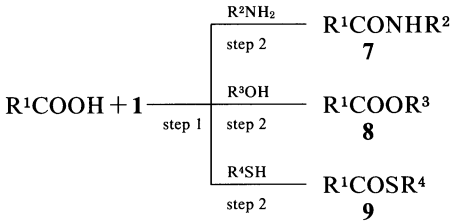
| Ratio of **1**/**2a** | Solvent (ml) | Yield % |
|---|---|---|
| 1.0 | THF (2ml) | 89 |
| 1.0 | $CH_3CN$ (2ml) | 88 |
| 1.0 | DMAc (2ml) | 93 |
| 1.0 | NMP (1ml) | 91 |
| 1.0 | NMP (2ml) | 94 |
| 1.0 | NMP (3ml) | 73 |
| 1.1 | NMP (2ml) | 99 |
| 1.2 | NMP (2ml) | 99 |
| 1.3 | NMP (2ml) | 100 |

a) Reaction was carried out with 1.0 mmol of the reactants using **1** in the presence of TEA (1.0 mmol) in solvent at room temperature. Reaction time: Step 1, 30 min, Step 2, 60 min.

Table 2. Effect of Reaction Time (Step 1 and Step 2) on Condensation[a]

| Reaction time (min) | | Yield % |
|---|---|---|
| Step 1 | Step 2 | |
| 5 | 60 | 92 |
| 10 | 60 | 98 |
| 30 | 60 | 99 |
| 10 | 5 | 97 |
| 10 | 10 | 98 |

a) Reaction was carried out with 1.0 mmol of the reactants using **1** (1.1 mmol) in the presence of TEA (1.0 mmol) in NMP (2 ml) at room temperature.



Scheme 3.

Table 3. Preparation of Amides **7**, Esters **8**, and Thioesters **9**[a]

| Carboxylic acid R-COOH | Amine or Alcohol | Product | Yield % |
|---|---|---|---|
| $C_6H_5CH=CH-$ | Cyclohexylamine | *N*-Cyclohexylcinnamamide | 96 |
| $(CH_3)_3C-$ | Aniline | *N*-Phenyl-2,2-dimethylpropanamide | 56[b] |
| $2,4,6\text{-}(CH_3)_3C_6H_2-$ | Benzylamine | *N*-Benzyl-2,4,6-trimethylbenzamide | 77[b] |
| $C_6H_5-$ | Phenol | Phenyl benzoate | 98 |
| $C_6H_5-$ | *p*-Nitrobenzyl alcohol | *p*-Nitrobenzyl benzoate | 82 |
| $C_6H_5-$ | Benzenethiol | *S*-Phenyl benzothioate | 99 |
| $C_6H_5-$ | Phenylmethanethiol | *S*-Benzyl benzothioate | 93 |
| $C_6H_5-$ | *p*-Aminophenol | 4′-Hydroxybenzanilide | 87 |
| $C_6H_5-$ | 1/2 *p*-Aminophenol | 4′-Benzoyloxybenzanilide | 93 |

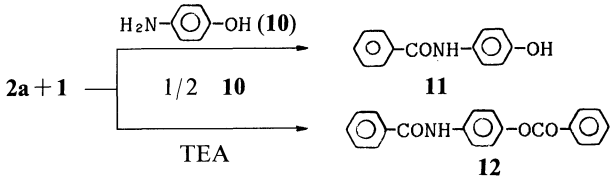
a) Reaction was carried out with 1.0 mmol of the reactants using **1** (1.1 mmol) in the presence of TEA (1.0 mmol) in NMP (2 ml) at room temperature. Reaction time: Amide, Step 1, 10 min, Step 2, 10 min , Ester, Step 1, 10 min, Step 2, 4 h. b) Step 1, 30 min, Step 2, 2 h.

Table 4. Preparation of Dipeptide Esters **15**[a]

| Product | Yield % | Mp[$\theta_m$/°C] Found | Mp[$\theta_m$/°C] Reported | $[\alpha]_D$/(°C, *c*, solv.) Found / Reported |
|---|---|---|---|---|
| Z-Gly-Ala-OBzl | 99 | 79—81 | 77—78 | −13.0 (25, 1.0, AcOEt)<br>−13.3 (20, 0.47, Acetone)[14] |
| Z-Ala-Gly-OEt | 91 | 97—99 | 98—99 | −21.6 (20, 1.0, EtOH)<br>−21.7 (16, 1.9, EtOH)[15] |
| Z-Ala-Ala-OBzl | 86 | 116—118 | 113 | −8.7 (26, 1.0, AcOEt)<br>−3.2 (20, —, $CHCl_3$)[16] |
| Z-Val-Gly-OEt | 99 | 167—168 | 170—171 | −31.0 (28, 1.0, Dioxane)<br>−31.6 (20, 1.2, Dioxane)[3] |
| Z-Val-Ala-OBzl | 98 | 159—162 | 152 | −16.7 (23, 1.5, AcOEt)<br>−13.2 (20, 0.63, AcOEt)[17] |
| Z-Phe-Gly-OEt | 90 | 109—112 | 109—110 | −16.8 (20, 2.0, EtOH)<br>−15.9 (24, 2.0, EtOH)[18] |
| Z-Phe-Leu-OMe | 91 | 109—111 | 110—111 | −23.1 (20, 3.1, MeOH)<br>−24.7 (20, 3.1, MeOH)[19] |
| Z-Ser-Gly-OEt | 80 | 100—101 | 103 | −5.9 (25, 4.1, EtOH)<br>−5.0 (25, 1.0, EtOH)[20] |
| Boc-Phe-Gly-OEt | 90 | 89—91 | 89—90 | −7.2 (20, 2.0, Dioxane)<br>−8.1 (16, 2.0, Dioxane)[21] |

a) Reaction was carried out with 2.0 mmol of the reactants using **1** (2.2 mmol) in the presence of TEA (4.0 mol) in $CH_2Cl_2$ (5 ml) at room temperature. Reaction time: Step 1, 20 min, Step 2, 3 h.

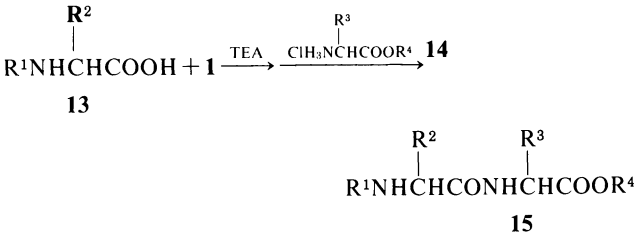
yields. An overall two-fold excess of TEA in alcoholysis was required for the reaction to go to completion. The amines reacted more rapidly than did the alcohols with the active amide **5**. Using this different reactivity, selective *N*-acylation and *N,O*-diacylation of *p*-aminophenol (**10**) were performed either in the presence of equimolar amount of **2a** and TEA or 2 molar amounts of these to **10**, respectively. The corresponding amide 4′-hydroxybenzanilide (**11**) or amide ester, 4′-(benzoyloxy)benzanilide (**12**) was obtained in excellent yields (Scheme 4). These results are summarized in Table 3.




Scheme 4.

**Synthesis of Dipeptides (15).** Peptide synthesis is essentially concerned with the formation of the amide bond. Therefore, the present reaction was applied to the preparation of dipeptides (Scheme 5).

Thus, the N-protected amino acid (**13**) was reacted with




Scheme 5.

Table 5. Preparation of Amides **7**, Esters **8**, and Thioesters **9**[a]

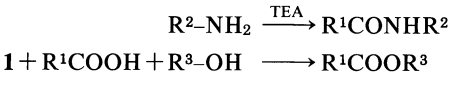
| Carboxylic acid R-COOH | Amine or Alcohol | Product | Yield % |
|---|---|---|---|
| $C_6H_5$– | Benzylamine | *N*-Benzylbenzamide | 90 |
| *n*-$C_5H_{11}$ | Benzylamine | *N*-Benzylhexanamide | 89 |
| $C_6H_5CH$=CH– | Cyclohexylamine | *N*-Cyclohexylcinnamamide | 99 |
| $(CH_3)_3C$– | Aniline | *N*-Phenyl-2,2-dimethylpropanamide | 99[b] |
| 2,4,6-$(CH_3)_3C_6H_2$– | Benzylamine | *N*-Benzyl-2,4,6-trimethylbenzamide | 98[b] |
| $C_6H_5$– | Phenol | Phenyl benzoate | 99 |
| $C_6H_5$– | *p*-Nitrophenol | *p*-Nitrobenzoate | 99 |
| $C_6H_5$– | *p*-Nitrobenzyl alcohol | *p*-Nitrobenzyl benzoate | 82 |
| $C_6H_5$– | Benzenethiol | *S*-Phenyl benzothioate | 99 |
| $C_6H_5$– | Phenylmethanethiol | *S*-Benzyl benzothioate | 94 |
| $C_6H_5$– | *p*-Aminophenol | 4′-Hydroxybenzanilide | 98 |
| $C_6H_5$– | D-Glucosamine | *N*-Benzoyl-D-Glucosamine | 88[c] |

a) Reaction was carried out with 1.0 mmol of the reactants using **1** (1.1 mmol) in the presence of TEA (1.0 mmol) in NMP (2 ml) at room temperature. Reaction time: Amide, 10 min, Ester, 2 h. b) Reaction time: 2 h. c) Reaction time: 24 h.

a *1*. 1 molar amount of **1** in dichloromethane in the presence of TEA at room temperature. After 10 min, the amino acid ester hydrochloride (**14**) and TEA as an acid acceptor were added. The dipeptide product **15** was isolated in the ordinary manner. The optical purity of the dipeptides was estimated by a comparison of the specific rotation with the reported values. N-protected dipeptide esters **15** were prepared in good yields, virtually without racemization. These results are summarized in Table 4.

**One-Step Procedure.** In this procedure, the active intermediate would be the mixed anhydride, which reacts with the nucleophile to give condensing product. The advantage of this method over the two-step procedure is that more reactive **3** in place of **5** is used in the condensation.

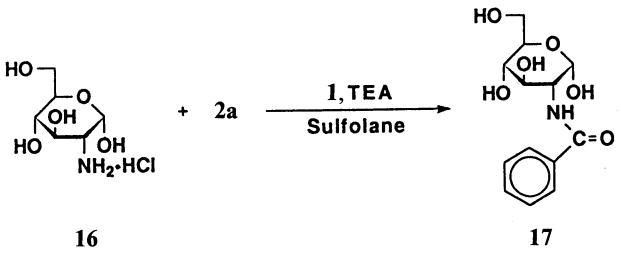
**Synthesis of Amides 7, Esters 8, and Thioesters 9.** Condensing agent **1** was added to a solution of the carboxylic acid, the nucleophiles, and TEA in NMP (Scheme 6).




Scheme 6.

We first carried out the synthesis of **7a** from **2a** and **6a** in the presence of **1** at room temperature. The reaction came to completion in 5 min and gave **7a** quantitatively. On the basis of these findings, several representative amide, ester, and thioester syntheses by a one-step procedure were performed, the results are summarized in Table 5. Condensations proceeded rapidly, giving the corresponding compounds in higher yields than did the two-step procedure.

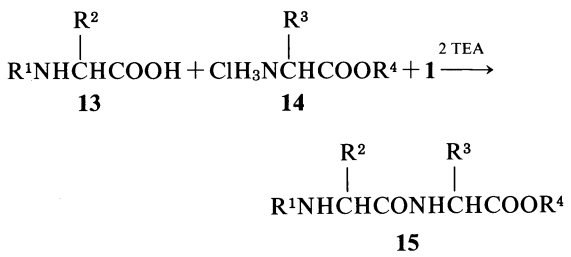
Furthermore, selective *N*-acylation of **10** was performed in quantitative yield. Then, selective *N*-benzoylation of D-(+)-glucosamine·hydrochloride (**16**) was carried out in the presence of TEA at room temperature, giving the desired amide, *N*-benzoyl-D-(+)-glucosamine (**17**) having hydroxyl groups in excellent yield (Scheme 7).




**16** **17**

Scheme 7.

**Synthesis of Dipeptides (15).** N-protected amino acids **13** were allowed to react with amino acid ester hydrochlorides **14** at room temperature in dichloromethane for 2 h, N-protected dipeptide esters **15** were obtained in excellent to quantitative yields (Table 6) (Scheme 8).




Scheme 8.

**Synthesis of β-Lactam (19).** In order to further demonstrate the preparative utility of our method, it was applied to the synthesis of **19** from β-amino acids

Table 6. Preparation of Dipeptide Esters **15**[a]

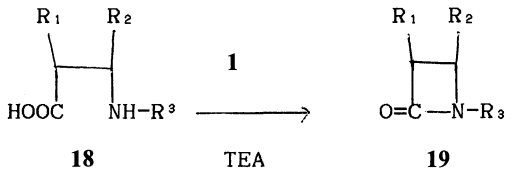
| Product | Yield | Mp[$\theta_m$/°C] | | [$\alpha$]$_D$/(°C, $c$, solv.) Found Reported |
|---|---|---|---|---|
| | % | Found | Reported | |
| Z-Gly-Ala-OBzl | 99 | 79—81 | 77—78 | −13.2 (25, 1.0, AcOEt)<br>−13.3 (20, 0.47, Acetone)[14] |
| Z-Ala-Gly-OEt | 99 | 99—100 | 98—99 | −22.0 (20, 1.0, EtOH)<br>−21.7 (16, 1.9, EtOH)[15] |
| Z-Ala-Ala-OBzl | 99 | 113—114 | 113 | −8.2 (26, 1.0, AcOEt)<br>−3.2 (20, —, $CHCl_3$)[16] |
| Z-Val-Gly-OEt | 99 | 172—174 | 170—171 | −31.3 (28, 1.0, Dioxane)<br>−31.6 (20, 1.2, Dioxane)[3] |
| Z-Val-Ala-OBzl | 99 | 159—160 | 152 | −17.8 (23, 1.0, AcOEt)<br>−13.2 (20, 0.63, AcOEt)[17] |
| Z-Phe-Gly-OEt | 93 | 108—109 | 109—110 | −17.3 (20, 2.0, EtOH)<br>−15.9 (24, 2.0, EtOH)[18] |
| Z-Phe-Leu-OMe | 96 | 109—111 | 110—111 | −24.2 (20, 3.1, MeOH)<br>−24.7 (20, 3.1, MeOH)[19] |
| Z-Ser-Gly-OEt | 86 | 100—101 | 103 | −5.2 (25, 3.0, EtOH)<br>−5.0 (25, 1.0, EtOH)[20] |
| Boc-Phe-Gly-OEt | 90 | 89—91 | 89—90 | −7.4 (20, 2.0, Dioxane)<br>−8.1 (16, 2.0, Dioxane)[21] |
| Z-Tyr-Gly-OEt | 92 | 168—170 | 168—170 | −23.3 (25, 2.0, DMF)<br>−23.0 (25, 5.0, DMF)[22] |

a) Reaction was carried out with 2.0 mmol of the reactants using **1** (2.2 mmol) in the presence of TEA (4.0 mmol) in $CH_2Cl_2$ (5 ml) at room temperature. Reaction time: 2 h.

Table 7. Preparation of β-Lactams **19**[a]

| β-Amino acid | | | β-Lactam |
|---|---|---|---|
| $R^1$ | $R^2$ | $R^3$ | Yield/% |
| $CH_3$– | H– | $PhCH_2$– | 79 |
| $CH_3$– | H– | $(CH_3)_2CHCH_2$– | 77 |
| H– | $CH_3$– | $PhCH_2$– | 84 |
| H– | $CH_3$– | $C_6H_{11}$– | 85 |
| H– | $CH_3$– | $(CH_3)_2CHCH_2$– | 79 |

a) Reaction was carried out with 1.0 mmol of β-amino acids using **1** (1.2 mmol) in the presence of TEA (4.0 mmol) in $CH_2Cl_2$ for 12 h at room temperature.




Scheme 9.

(**18**). The cyclization reaction of **18** was conducted in dichloromethane at room temperature with a 1.2 molar amount of the condensing agent **1**. The crude products were purified by column chromatography. The data summarized in Table 7 indicate that the condensing agent is useful for the cyclization of **18** and gave good yields of **19** under mild conditions (Scheme 9).

In summary, our studies indicate that **1** is a very useful reagent for the synthesis of amides, esters, thioesters, dipeptides, and β-lactams. This condensing agent **1** is a crystalline solid having excellent hydrolytic stability. Furthermore, 2-benzoxazolethiol **4**, a reaction product, is easily removed from the reaction products by washing the reaction mixture with 1% aqueous sodium hydrogencarbonate. Compared with the other reagents mentioned in the literature, activaing agent **1** is preferable for the following reasons: (a) simplicity of procedure, (b) excellent yield, (c) excellent hydrolytic stability, and (d) good chemoselectivity.

## Experimental

The melting points were uncorrected. Infrared spectra were obtained in potassium bromide pellets. Benzene, pyridine, triethylamine (TEA), and *N*-methyl-2-pyrrolidinone (NMP) were purified by the usual methods. N-Protected α-amino acids and α-amino acid ester hydrochlorides were prepared by the usual procedures. Diphenyl phosphorochloridate was prepared from phosphoryl trichloride and phenol. *N*-Alkyl-DL-β-amino acids were prepared by the direct addition of amines to the α,β-unsaturated acids.[12,13] The other reagents were used without further purification.

The condensing agent, diphenyl (2,3-dihydro-2-thioxo-3-benzoxazolyl)phosphonate **1** was prepared by the reported procedure.[11]

**Amide 7: General Procedure (Two-Step Procedure).** Condensing agent **1** (0.422 g, 1.1 mmol) was added to a solution of the carboxylic acid (1.0 mmol) and TEA (0.14 ml) in NMP (2 ml) at room temperature. After 10 min, the amine (1.0 mmol) was added. Stirring was continued for 10 min. The mixture was poured into 1% aqueous sodium hydrogencarbonate. The precipitates were collected by filtration, washed with water, and dried.

**Amide Ester 12: Two-Step Procedure.** Condensing agent **1** (1.1 mmol) was added with stirring to a solution of benzoic

acid (1.0 mmol) and TEA (1.0 mmol) in NMP (2 ml) at room temperature. After 10 min, *p*-aminophenol (0.5 mmol) was added to this solution. After 30 min of stirring, TEA (0.5 mmol) was added, and the mixture was stirred for 4 h and worked up as described above.

**Amide 7: General Procedure (One-Step Procedure).** To a solution of the carboxylic acid (1.0 mmol), the amine (1.0 mmol), and TEA (1.0 mmol) in NMP (2 ml) was added **1** (1.1 mmol) at room temperature. The solution was stirred for 10 min and then poured into 1% aqueous sodium hydrogencarbonate. The precipitates were collected by filtration, washed with water, and dried.

**Ester 8 and Thio Ester 9: General Procedure (One-Step Procedure).** A mixture of the carboxylic acid (1.0 mmol), the alcohol (1.0 mmol), or thiol (1. 0 mmol), TEA (2.0 mmol), and **1** (1.1 mmol) in NMP (2 ml) was stirred at room temperature for 2 h. The reaction mixture was worked up as described above.

**Protected Dipeptide Ester 15: General Procedure (One-Step Procedure).** To a solution of the N-protected $\alpha$-amino acid (2.0 mmol), the $\alpha$-amino acid ester hydrochloride (2.0 mmol), and TEA (4.0 mmol) in dichlorormethane (5 ml) was added **1** (2.2 mmol) under nitrogen. The solution was stirred for 2 h at room temperature. After removing of the solvent in vacuo, the residue was dissolved in ethyl acetate, the organic solution was washed successively with 5% aqueous sodium hydrogencarbonate, 1 M hydrochloric acid, and saturated brine. After drying ($MgSO_4$) and evaporation of ethyl acetate, the dipeptide ester was purified by crystallization.

**$\beta$-Lactam 19: General Procedure.** Condensing agent **1** (1.2 mmol) was added to a mixture of the *N*-alkyl-DL-$\beta$-amino acid (1.0 mmol) and TEA (0.28 mL, 2.0 mmol) in dichloromethane (10 ml). The mixture was stirred at room temperature for 12 h, followed by the usual workup. The organic layer was dried ($MgSO_4$). After evaporaing of the solvent, the crude product was purified by column chromatography on silica gel by using 1 : 1 ethyl acetate–hexane as the eluent.

The crude obtained products (amides, esters, thioesters, dipeptide esters, and $\beta$-lactams) were virtually pure (IR, $^1$H NMR spectra).

The authors are indebted to Makoto Morishima and Sadao Kato for their techinical assistance and Takeyoshi Takahashi for performing the elemental analyses.

## References

1) M. A. Ogliaruso and J. F. Wolfe, "The Chemistry of Acid Derivatives," ed by S. Patai, John Wiley and Sons, New York (1979), Part I, p. 474.
2) "The Peptides," ed by E. Gross and J. Meienhofer, Academic Press, New York (1979), Vol. 1.
3) M. Ueda, H, Kawaharasaki, and Y. Imai, *Synthesis,* **1982**, 933.
4) M. Ueda, H, Oikawa, N. Kawaharasaki, and Y. Imai, *Bull. Chem. Soc. Jpn.,* **56**, 2485 (1983).
5) M. Ueda, N, Kawaharasaki, and Y. Imai, *Bull. Chem. Soc. Jpn.,* **57**, 85 (1984).
6) M. Ueda and H. Oikawa, *J. Org. Chem.,* **50**, 760 (1985).
7) T. Shioiri, K. Yokoyama, and S. Yamada, *J. Am. Chem. Soc.,* **94**, 6203 (1972).
8) T. Shioiri, K. Yokoyama, Y. Kasai, and S. Yamada, *Tetrahedron,* **32**, 3211 (1976).
9) H. Ogura, S. Nagai, and K. Takeda, *Tetrahedron Lett.,* **1980**, 1467.
10) T. Kunieda, Y. Abe, and M. Hirobe, *Chem. Lett.,* **1981**, 1427.
11) M. Ueda, A. Kameyama, and K. Hashimoto, *Macromolecules,* **21**, 19 (1988).
12) A. Zilkha and J. Rivlin, *J. Org. Chem.,* **23**, 95 (1958).
13) A. Zilkha, E. S. Rachman, and J. Rivlin, *J. Org. Chem.,* **26**, 376 (1961).
14) J. Gante, *Angew. Chem.,* **77**, 813 (1965).
15) R. Paul and G. W. Anderson, *J. Am. Chem. Soc.,* **82**, 4596 (1960).
16) G. Losse and H. Wedige, *Justus Liebigs Ann. Chem.,* **678**, 148 (1964).
17) U. Schmidt, M. Krorner, and U. Beutler, *Synthesis,* **1988**, 475.
18) R. W. Yong, K. H. Wood, R. J. Joyce, and G. W. Anderson, *J. Am. Chem. Soc.,* **78**, 2126 (1956).
19) S. Yamada and Y. Takeuchi, *Tetrahedron Lett.,* **1971**, 3595.
20) B. Castrd, J. R. Dormoy, G. Evin, and C. Selve, *Tetrahedron Lett.,* **1975**, 1219.
21) E. Schroeder and H. Gibian, *Justus Liebigs Ann. Chem.,* **673**, 176 (1964).
22) Y. Watanabe, N. Morimoto, K. Kamekawa, and T. Mukaiyama, *Chem. Lett.,* **1981**, 65.