[Chem. Pharm. Bull.
25(1) 166—170 (1977)]

UDC 547.551.44.04 : 547.743.1.04

Stereochemical Studies. XLV.[1] A Novel Regiospecific Ring Opening of optically Active 2-Substituted-1-tosylaziridines

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(Received May 7, 1976)

In the course of our examinations on an applicability of the novel synthetic scheme for optically active pyrrolidines from L-α-amino acids, it was found that the reaction of (*R*) (−)-2-phenyl-1-tosylaziridine ((*R*) (−)-**9**) with diethyl potassiomalonate exclusively afforded (*S*) (+)-pyrrolidine-3-carboxylate ((*S*) (+)-**10**) as a mixture of two diastereoisomers by the attack of malonate anion at the C_2-carbon of the aziridine ring. This result is completely different from those obtained for (*S*)-2-benzyl or isopropyl-1-tosylaziridine ((*S*)-**7a,b**) on which the attack of malonate anion occurred specifically at the unsubstituted C_3-carbon.

These results clearly show that electronic effect of the C_2-substituent is considered as the most important factor which controls regiospecificity of the ring opening of 2-substituted-1-tosylaziridines by malonate anion.

Structual confirmation of (*S*) (+)-**10** was also carried out by converting it into (*S*) (+)-3-phenylpyrrolidine ((*S*) (+)-**17**).

Keywords——malonate synthesis; regiospecific ring opening; optically active 2-alkyl-1-tosylaziridines; reaction mechanism; S_N2 mechanism; benzylic position

In connection with synthetic studies of optically active (*R*)-pyrrolidines ((*R*)-**1a**, **b**) from L-α-amino acids (L-**2a**, **b**) by way of optically active (*R*)-γ-amino acids ((*R*)-**3a**, **b**), it was found that reactions of (*S*)-ditosylates ((*S*)-**4a**, **b**) with diethyl potassiomalonate gave (*R*)-pyrrolidine-3-carboxylates ((*R*)-**5a**, **b**) as diastereoisomeric mixtures and (*R*)-malonates ((*R*)-**6a**, **b**) *via* (*S*)-aziridines ((*S*)-**7a**, **b**).[1]

Formation of (*R*)-**5a**, **b** and (*R*)-**6a**, **b** from (*S*)-**7a**, **b** clearly discloses that the attack of malonate anion to (*S*)-**7a**, **b** occurrs regiospecifically at the unsubstituted C_2-position of aziridine ring.

In order to examine a generality of the above-mentioned pyrrolidine synthesis, commercially available (*R*)(−)-phenylglycine ((*R*)(−)-**8**) was submitted to the exploited synthetic scheme. Contrary to our expectation, it was observed that the C_2-position of (*R*)(−)-2-phenyl-1-tosylaziridine ((*R*)(−)-**9**) was exclusively attacked by malonate anion instead of the C_3-position, giving (*S*)(+)-ethyl (2-oxo-4-phenyl-1-tosyl)pyrrolidine-3-carboxylate ((*S*)(+)-**10**) (*vide infra*) in a high yield.

This report deals with abnormal reaction of (*R*)(−)-**9** with malonate anion, structural elucidation of (*S*)(+)-**10**, and some mechanistic considerations on the reaction of malonate anion with 2-substituted-1-tosylaziridines.

Conversion of (*R*)(−)-**8** into (*R*)(−)-(2-amino-2-phenyl) ethyl alcohol ((*R*)(−)-**11**), $[\alpha]_D^{25}$ −25.8° (methanol), was accomplished *via* (*R*)(−)-ethyl phenylglycinate hydrochloride ((*R*)(−)-**12**),[3] $[\alpha]_D^{20}$ −88.8° (water), by successive esterification with thionyl chloride and ethanol (quantitative yield), and reduction with sodium borohydride (90% yield).[4] Treatment of

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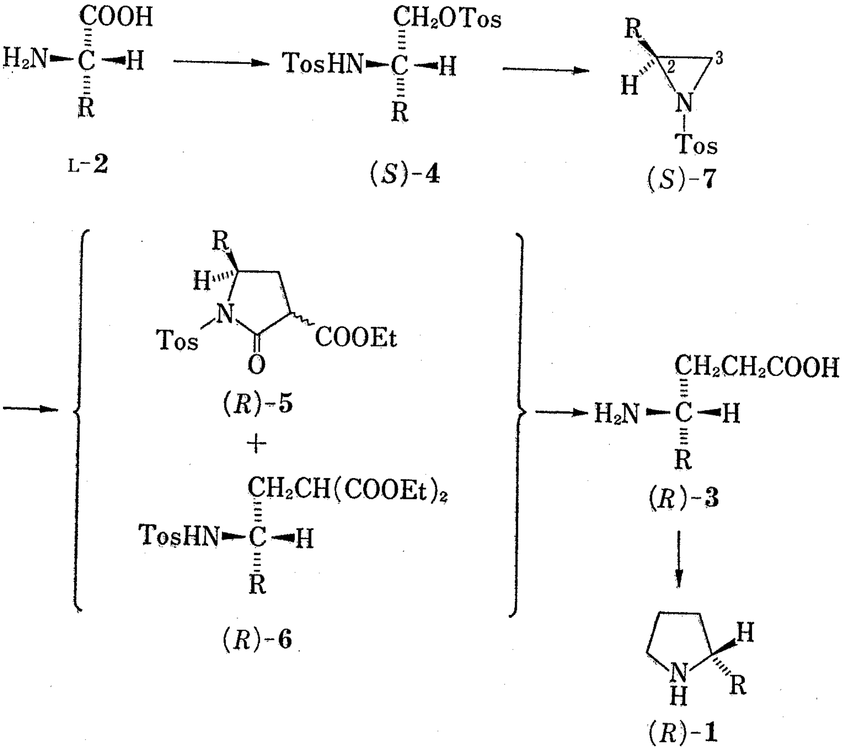
[1] Part XLIV: C.C. Tseng, S. Terashima, and S. Yamada, *Chem. Pharm. Bull.* (Tokyo), 25, 29 (1977).

[2] Location: *Hongo, Bunkyo-ku, Tokyo, 113, Japan.*

[3] H. Herlinger, H. Kleimann, and I. Ugi, *Ann.*, 706, 37 (1967).

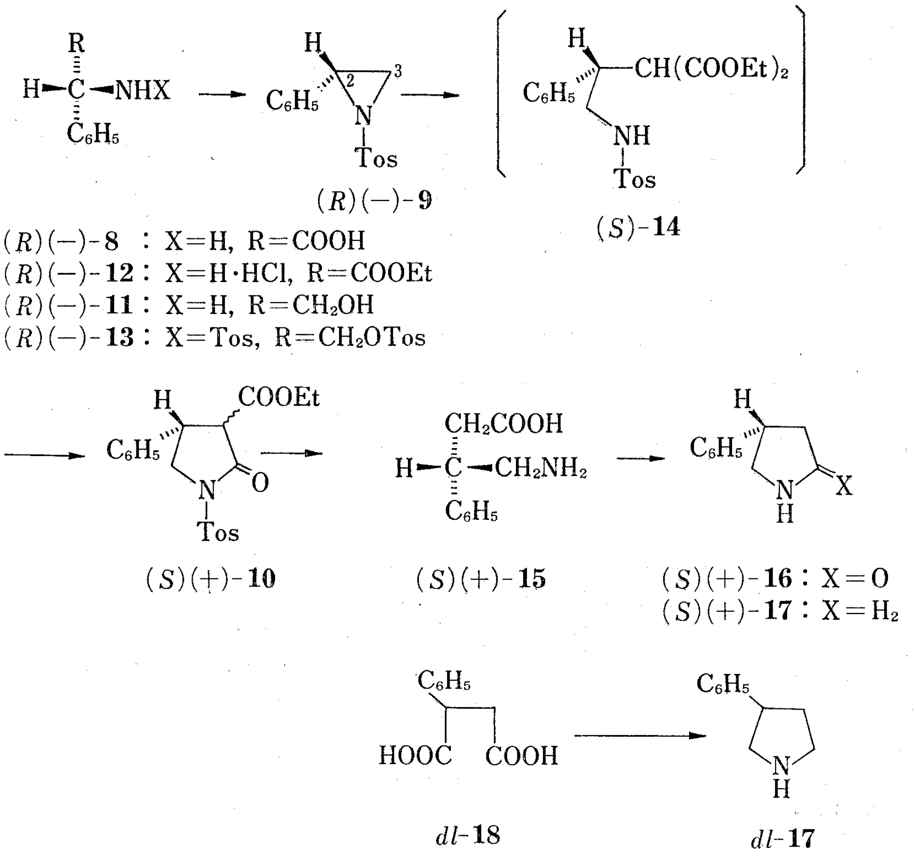
[4] H. Seki, K. Koga, H. Matsuo, S. Ohki, I. Matsuo, and S. Yamada, *Chem. Pharm. Bull.* (Tokyo), 13, 995 (1965).



Tos: $SO_2C_6H_4CH_3$-*p*

a) $R=CH_2C_6H_5$; b) $R=CH(CH_3)_2$

Chart 1



Tos: $SO_2C_6H_4CH_3$-*p*

Chart 2

(*R*)(−)-**11** with tosyl chloride (4.0 eq.) in pyridine[1] yielded (*R*)(−)-(2-phenyl-2-tosylamido) ethyl tosylate ((*R*)(−)-**13**), $[\alpha]_D^{19}$ −24.7° (benzene), in a good yield.

Reflux of a mixture containing (*R*)(−)-**13** and diethyl potassiomalonate (3.0 eq.)in anhyd. tetrahydrofuran (THF) for 3 hr gave crude (*S*)(+)-**10**, mp 95—106°, $[\alpha]_D^{20.5}$ +54.7° (benzene) as the sole reaction product in 85% yield based on (*R*)(−)-**13**. Similarly to the cases for (*R*)-**5a**, **b**,[1] the nuclear magnetic resonance (NMR) spectrum of crude (*S*)(+)-**10** exhibited two sets of triplets at 0.70 and 1.10 ppm whose relative intensity was 1 : 19.[5] This spectral feature might be reasonably explained by the assumption that crude (*S*)(+)-**10** contains two diastereoisomers differing the configuration of ester group and that the major diastereoisomer has the ester group being *trans* to the phenyl group. That the ratio of two diastereoisomers is extremely larger than those for (*R*)-**5a**, **b** is considered due to the fact that the α-position of ester group is adjacent to the other asymmetric center. Repeated recrystallizations of crude (*S*)(+)-**10** from a mixture of benzene and hexane afforded pure sample as colorless needles, mp 112—114°, $[\alpha]_D^{20.5}$ +70.2° (benzene). Since the NMR spectrum of pure (*S*)(+)-**10** was the same as that of the crude sample, purified (*S*)(+)-**10** might similarly consist of two diastereoisomers.

On the other hand, when (*R*)(−)-**13** was allowed to react with 1.5 eq. of diethyl potassiomalonate for 5 min, extractive isolation followed by purification with preparative thin-layer chromatography (TLC) gave (*R*)(−)-2-phenyl-1-tosylaziridine ((*R*)(−)-**9**) in 34% yield in addition to crude (*S*)(+)-**10** (30% yield). (*R*)(−)-**9** was identified with the authentic sample, mp 78—79°, $[\alpha]_D^{20.5}$ −66.8° (benzene), independently prepared from (*R*)(−)-**13** in 78% yield by treatment with anhyd. potassium carbonate, by spectral comparisons, and was successfully converted into (*S*)(+)-**10** in 94% yield by the reaction with 2.0 eq. of diethyl potassiomalonate. These results clearly shows that the formation of (*S*)(+)-**10** from (*R*)(−)-**13** proceeds through (*R*)(−)-**9** and (*S*)-diethyl (1-phenyl-2-tosylamidoethyl) malonate ((*S*)-**14**) although isolation of the latter compound from the reaction mixture has not been accomplished, being different from the previous cases.[1]

Two modes such as *S*N1 or *S*N2 mechanism are possible for the ring opening of (*R*)(−)-**9** by malonate anion.[6] However, since the optical integrity of (*R*)(−)-**13** can be transformed into (*S*)(+)-**10** *via* (*R*)(−)-**9** with high retention, it might be reasonable to assume that (*S*)(+)-**10** was produced from (*R*)(−)-**9** preferentially by *S*N2 mechanism. Based on this assumption, the absolute configuration of (+)-**10** was tentively assigned to belong to (*S*)-series.

The exclusive formation of (*S*)(+)-**10** which results from the attack of malonate anion at the C_2-position of the aziridine ring, differs from the previous cases.[1] This can be explained by the assumption that the electronic effect of phenyl group which assists the creation of positive charge at benzylic position, operates more predominantly than its steric effect because the steric bulkiness of phenyl group of (*R*)(−)-**9** is clearly larger than those of alkyl groups of (*S*)-**7a**, **b** when their conformational energies are taken into consideration.[7]

Aiming to further confirm the structure of (*S*)(+)-**10**, conversion of (*S*)(+)-**10** into (*S*)(+)-3-phenylpyrrolidine ((*S*)(+)-**17**) was attempted by following the synthetic route previously established.[1]

Acidic hydrolysis of (*S*)(+)-**10** with 47% hydrobromic acid gave (*S*)(+)-4-amino-3-phenylbutyric acid ((*S*)(+)-**15**), $[\alpha]_D^{20}$ +12.9° (water), in 61% yield. Dehydration of (*S*)(+)-**15**, followed by reduction with lithium aluminum hydride (LAH), afforded (*S*)(+)-**17**, $[\alpha]_D^{20}$ +22.7° (ethanol), *via* (*S*)(+)-4-phenylpyrrolidine-2-one ((*S*)(+)-**16**), $[\alpha]_D^{20.5}$ +45.6° (benzene). (*S*)(+)-**17**, thus obtained, showed the same spectral and chromatographic properties as those of *dl*-**17**

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5) The methyl signal of tosyl group also appeared as two sets of singlets at 1.70 and 2.38 whose relative intensity was 1 : 19.

6) O.C. Dermer and G.E. Ham, "Ethyleneimine and Other Aziridines," Academic Press, New York, London, 1969, p. 206.

7) J.A. Hirsh, "Topics in Stereochemistry," Vol. I, ed. by N.L. Allinger and E.L. Eliel, Interscience Publisher, New York, London, Sydney, 1967, p. 199.

which was independently prepared from *dl*-phenylsuccinic acid (*dl*-**18**).[8]

Although only few results are now available from this and the previous reports,[1] it might be concluded that the attack of malonate anion to 2-substituted-1-tosylaziridine is controlled by the electronic effect of the C_2-substituent more preferentially than by the steric effect.

Experimental[9]

(*R*) (−)-Ethyl Phenylglycinate Hydrochloride ((*R*) (−)-12)——This was prepared in a quantitative yield by treating (*R*) (−)-**8** with thionyl chloride in ethanol. The sample which was recrystallized from ethanol–ether as colorless prisms, showed mp 203.5—205.5° and $[\alpha]_D^{20}$ −88.8° (c=0.778, H_2O) (lit.,[3] mp 199—200° and $[\alpha]_D^{20}$ −91.8° (c=4.522, H_2O)).

(*R*) (−)-(2-Amino-2-phenyl)ethyl Alcohol ((*R*) (−)-11)——Reduction of (*R*) (−)-**12** (22.0 g, 0.10 mole) with sodium borohydride (15.0 g, 0.40 mole) in 50% aqueous ethanol (500 ml) according to the reported method,[4] gave crude (*R*) (−)-**11**, mp 69.5—71.5°, as pale yellow needles (12.4 g, 90%) after extractive isolation followed by evaporation *in vacuo*. Recrystallizations from ether afforded pure (*R*) (−)-**11** as colorless needles, mp 76—78°, $[\alpha]_D^{25}$ −25.8° (c=6.60, methanol). IR ν_{max}^{Nujol} cm^{-1}: 3340—3100 (OH, NH_2); 1050 (OH). NMR (in $CDCl_3$): 2.71 (3H, s, $O\underline{H}$ and $N\underline{H}_2$), 3.46 (1H, doubled d, J=12, 9 Hz, one of $C\underline{H}_2O$), 3.71 (1H, doubled d, J=12, 5 Hz, one of $C\underline{H}_2O$), 4.00 (1H, doubled d, J=9, 5 Hz, $C\underline{H}N$), 7.32 (5H, s, $C_6\underline{H}_5$). *Anal.* Calcd. for $C_8H_{11}ON$: C, 70.04; H, 8.08; N, 10.21. Found: C, 70.32; H, 8.28; N, 10.29.

(*R*) (−)-(2-Tosylamido-2-phenyl)ethyl Tosylate ((*R*) (−)-13)——The same treatment of (*R*) (−)-**11** (6.87 g, 50 mmole) as that previously reported,[1] gave crude (*R*) (−)-**13** as pale yellow crystals (15.9 g, 77%), mp 102—111°, $[\alpha]_D^{19}$ −25.3° (c=1.15, benzene) after extractive isolation, evaporation, and one recrystallization from benzene–hexane. Repeated recrystallizations from benzene–hexane gave pure (*R*) (−)-**13** as colorless needles, mp 110—112°, $[\alpha]_D^{19}$ −24.7° (c=1.08, benzene). IR ν_{max}^{Nujol} cm^{-1}: 3320 (NH); 1360, 1340, 1180, 1160 (SO_2). NMR (in $CDCl_3$): 2.33, 2.39. (6H, two s, $2\times C_6H_4C\underline{H}_3$), 4.06 (2H, d, J=6 Hz, $C\underline{H}_2O$), 4.50 (1H, doubled t, J=each 6 Hz, $C_6H_5C\underline{H}$), 5.48 (1H, d, J=6 Hz, $N\underline{H}$), 6.78—7.70 (13H, m, aromatic protons). *Anal.* Calcd. for $C_{22}H_{23}O_5NS_2$: C, 59.31; H, 5.20; N, 3.14. Found: C, 59.07; H, 5.01; N, 2.97.

(*R*) (−)-2-Phenyl-1-tosylaziridine ((*R*) (−)-9)——Treatment of (*R*) (−)-**13** (2.5 g, 6.0 mmole) with anhyd. potassium carbonate (0.84 g, 6.0 mmole) in acetone (15 ml) in a similar fashion to that reported,[1] gave crude (*R*) (−)-**9** as a pale brown solid (1.28 g, 78%), mp 74—77°. Two recrystallizations from benzene–hexane gave pure (*R*) (−)-**9** as colorless crystals, mp 78—79°, $[\alpha]_D^{20.5}$ −66.8° (c=1.18, benzene). IR ν_{max}^{Nujol} cm^{-1}: 1320, 1160 (SO_2). NMR (in $CDCl_3$); 2.36 (1H, d, J=4 Hz, one of $C\underline{H}_2$, *cis* to phenyl group[10]), 2.40 (3H, s, $C_6H_4C\underline{H}_3$), 2.93 (1H, d, J=7 Hz, one of $C\underline{H}_2$, *trans* to phenyl group),[10] 3.72 (1H, doubled d, J=7, 4 Hz, $C\underline{H}$), 7.22 (5H, s, $C_6\underline{H}_5$), 7.28 (2H, d, J=9 Hz, aromatic protons *ortho* to CH_3), 7.82 (2H, d, J=9 Hz, aromatic protons *ortho* to SO_2). *Anal.* Calcd. for $C_{15}H_{15}O_2NS$: C, 65.91; H, 5.53; N, 5.12. Found: C, 65.91; H, 5.49; N, 5.25.

(*S*) (+)-Ethyl (2-Oxo-4-phenyl-1-tosyl)pyrrolidine-3-carboxylate ((*S*) (+)-10)——a) (*S*) (+)-**10** from (*R*) (−)-**13**: To a suspension of diethyl potassiomalonate in anhyd. THF prepared from diethyl malonate (480 mg, 3.0 mmole) and potassium *t*-butoxide (340 mg, 3.0 mmole) by the usual manner, was added (*R*) (−)-**13** (414 mg, 1.0 mmole) with stirring, and the whole was heated at reflux for 3.0 hr. After evaporation *in vacuo*, a mixture of benzene (20 ml), satd. NaCl (15 ml), and 10% HCl (5 ml) was added to the residue. The upper organic phase was separated, washed with satd. NaCl (×2), and dried over anhyd. $MgSO_4$. Filtration and evaporation *in vacuo* gave an oily residue (650 mg), which was purified by column chromatography (silica gel, solvent ether: hexane 1: 2), affording crude (*S*) (+)-**10** as a diastereoisomeric mixture (330 mg, 85%), colorless solid, mp 95—106°, $[\alpha]_D^{20.5}$ +54.7° (c=1.03, benzene). IR ν_{max}^{Nujol} cm^{-1}: 1760, 1740 (COOEt and CONH); 1360, 1170 (SO_2). NMR (in $CDCl_3$): 0.70, 1.10 (3H, two t, J=each 7 Hz, the intensity of two triplets was 1: 19, $CH_2C\underline{H}_3$), 1.70, 2.38 (3H, two s, the intensity of two singlet was 1: 19, $C_6H_4C\underline{H}_3$), 3.05—5.00 (6H, m, $NC\underline{H}_2C\underline{H}C\underline{H}COOC\underline{H}_2CH_3$), 6.90—7.45 (7H, m, $C_6\underline{H}_5$+aromatic protons *ortho* to CH_3), 7.87 (2H, d, J=9 Hz, aromatic protons *ortho* to SO_2). Five recrystallizations from benzene–hexane gave pure (*S*) (+)-**10** as colorless needles mp 112—114°, $[\alpha]_D^{20.5}$ +70.2° (c=0.958, benzene).[11] Spectral (IR and NMR) properties of this sample

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[8] A.G. Chigarev and D.V. Ioffe, *Zh. Org. Khim.*, 3, 85 (1967) [*C.A.*, **66**, 94779*y* (1967)].

[9] All melting and boiling points are uncorrected. IR spectra measurements were carried out using a spectrometer, JASCO-IRA-1 Grating Infrared Spectrometer. NMR spectra were measured with spectrometers, JNM-PS 100 and Hitachi R-24 High Resolution NMR Spectrometers. All signals are expressed by the ppm downfield from internal standards (tetramethylsilane for $CDCl_3$ and sodium trimethylsilylpropanesulfonate for D_2O). Following abbreviations are used: singlet(s); doublet(d); triplet(t); quartet(q); multiplet(m); broad(br). Optical rotations were recorded with YANACO OR-50 Automatic Polarimeter.

[10] L.M. Jackman and S. Sternhell, "Application of Nuclear Magnetic Resonance Spectroscopy in Organic Chemistry," Pergamon Press, Oxford, 1969, p. 272.

[11] This sample was a mixture of two diastereoisomers whose relative ratio determined by the NMR spectrum was 1: 19.

were identical with those of crude (*S*) (+)-**10**. *Anal.* Calcd. for $C_{20}H_{21}O_5NS$: C, 62.00; H, 5.46; N, 3.62. Found: C, 61.88; H, 5.43; N, 3.51.

When the same reaction as that described above was attempted by using 1.5 eq. of diethyl potassiomalonate and the reaction mixture was refluxed for 5 min, extraction followed by separation with preparative TLC (silica gel, solvent ether: hexane 1: 1), gave (*R*) (−)-**9** (94 mg, 34%) as a colorless solid and crude (*S*) (+)-**10** as a diastereoisomeric mixture (115 mg, 30%), pale yellow solid. These compounds were respectively identified with the authentic samples prepared above, by spectral (IR) comparisons.

b) (*S*) (+)-**10** from (*R*) (−)-**9**: To a suspension of diethyl potassiomalonate in anhyd. THF (10 ml) pre pared from diethyl malonate (320 mg, 2.0 mmole) and potassium *t*-butoxide (224 mg, 2.0 mmole) by the usual manner, was added (*R*) (−)-**9** (273 mg, 1.0 mmole) with stirring, and the whole was refluxed for 5 min. Similar extraction to that described in a), followed by evaporation *in vacuo* afforded a pale yellow oil (505 mg) which solidified on standing. Trituration of a part of this solid (301 mg) with hexane gave crude (*S*) (+)-**10** as a diastereoisomeric mixture (218 mg, 94%), colorless powder. IR spectrum of this sample was identical with that of pure (*S*) (+)-**10** obtained in a). Two recrystallizations from benzene–hexane gave pure (*S*) (+)-**10** as colorless needles, mp 110.5—113°.[11] This sample showed no depression on mixed melting point measurement with the authentic sample obtained in a), mp 111—113°.

(*S*) (+)-4-Amino-3-phenylbutyric Acid ((*S*) (+)-15)——A mixture of crude (*S*) (+)-**10** (5.62 g, 0.145 mmole) and 47% hydrobromic acid (40 ml) was heated at reflux, and a volatile material was removed until the temperature of the mixture reached to 110°. After reflux for 11.5 hr, further amount of 47% hydrobromic acid (20 ml) was added to the reaction mixture, and the reflux was continued for additional 5 hr. After cooling, the whole was treated by a similar manner to that previously described,[1] giving crude (*S*) (+)-**15** as a colorless powder (1.58 g, 61%) after evaporation of the aqueous NH_4OH eluate from a column of ion exchanger (Amberlite IR-120, H^+ form). Recrystallization from ethanol–ether–H_2O afforded pure (*S*) (+)-**15** as a hygroscopic colorless powder, mp 213—214°, $[\alpha]_D^{20}$ +12.9° (c=1.11, H_2O). IR ν_{max}^{Nujol} cm^{-1}: 3340, 1665, 1655, 1550 (NH_3^+ and COO^-). NMR (in D_2O): 2.40—3.60 (5H, m, $N^+\underline{CH_2}\underline{CH}\underline{CH_2}COO^-$), 7.42 (5H, s, $C_6\underline{H_5}$). *Anal.* Calcd. for $C_{10}H_{13}O_2N$: C, 67.02; H, 7.31; N, 7.82. Found: C, 66.37; H, 7.27; N, 7.79.

(*S*) (+)-4-Phenylpyrrolidine-2-one ((*S*) (+)-16)——A toluene solution (40 ml) of (*S*) (+)-**14** (1.33 g, 7.4 mmole) was heated at reflux for 15 hr using a Dean-Stark apparatus to remove the water produced. Filtration and evaporation *in vacuo* gave crude (*S*) (+)-**16** as a pale yellow solid (1.06 g, 88%). Recrystallization from ether–petr. ether afforded pure (*S*) (+)-**16** as colorless plates, mp 101—104°, $[\alpha]_D^{20.5}$ +45.6° (c=0.366, benzene). IR ν_{max}^{Nujol} cm^{-1}: 3200 (NH); 1695 (CONH). NMR (in $CDCl_3$): 2.14—3.08 (2H, m, $\underline{CH_2}CONH$), 3.27—4.00 (3H, m, $C\underline{H}C\underline{H_2}NHCO$), 7.28 (5H, s, $C_6\underline{H_5}$), 7.60 (1H, br s, $N\underline{H}$). *Anal.* Calcd. for $C_{10}H_{11}ON$: C, 74.51; H, 6.88; N, 8.69. Found: C, 74.32; H, 6.87; N, 8.67.

(*S*) (+)-3-Phenylpyrrolidine ((*S*) (+)-17)——A mixture of (*S*) (+)-**16** (3.56 g, 22 mmole) and lithium aluminum hydride (1.04 g, 28 mmole) in anhyd. THF was refluxed with stirring for 26 hr. After cooling, ether (60 ml) was added to the mixture. The metal complex was decomposed by adding 10% NaOH (3 ml), then the whole was heated at reflux for 1.5 hr. After drying over anhyd. K_2CO_3, filtration and evaporation *in vacuo*, followed by fractional distillation, gave pure (*S*) (+)-**17** as a colorless oil (1.98 g, 60%), bp 96—99° (4—5 mmHg), $[\alpha]_D^{20}$ +22.7° (c=2.36, EtOH) and $[\alpha]_D^{20}$ +8.2° (c=1.81, 6N-HCl). IR ν_{max}^{film} cm^{-1}: 3280 (NH); 1603 (aromatic ring). NMR (in $CDCl_3$): 1.83 (1H, s, $N\underline{H}$), 1.46—2.67 (2H, m, $C\underline{H_2}CH_2N$), 2.67—3.73 (5H, m, $C\underline{H_2}NC\underline{H_2}C\underline{H}$), 7.23 (5H, s, $C_6\underline{H_5}$). This oil afforded the picrate as yellow prisms, mp 158—160° (recrystallized from ethanol). *Anal.* Calcd. for $C_{16}H_{16}O_7N_4$: C, 51.06; H, 4.29; N, 14.89. Found: C, 51.20; H, 4.27; N, 14.97.

***dl*-3-Phenylpyrrolidine (*dl*-17)**——This was prepared from *dl*-**18** by way of *dl*-3-phenylpyrrolidine-2,5-dione, mp 83—85° (lit.,[8] mp 89—90°), according to the reported method.[8] *dl*-**17** thus obtained, distilled at bp 106—116° (6—7 mmHg) (lit.,[8] bp 92—94° (2 mmHg)), and showed the identical spectral (IR and NMR) behavior with those of (*S*) (+)-**17** measured in the same state. *dl*-**17** also gave the crystalline picrate, mp 159—161° (recrystallized from ethanol) (lit.,[8] mp 159—161°).

Acknowledgement The authors are indebted to the Central Analysis Room of this Faculty for elemental analyses and spectral data.