Occurrence of Homoserine Betaine in the Hydrolyzate of an Unknown Base Isolated from a Green Alga, Monostroma nitidum*

In a previous paper1), it was reported that the picrate of an unknown base was isolated from the basic fraction (F-2) of green laver which reduces the plasma cholesterol levels in rats. From the hydrolyzate of this base, homoserine betaine was isolated as follows.

The picrate (1 g) was dissolved in a small amount of 50% aqueous ethanol and the solution was run through a column of Dowex 1–X8 (OH- form, 200–400 mesh, 1.0 × 5 cm). Succeedingly, the column was washed with water. The effluent and washings were combined and evaporated to dryness. The residue was dissolved in 5 ml of 6 N HCl and the solution was heated in a sealed glass tube at 150–160°C for 24 hr. The hydrolyzate was filtered with a glass filter and excess HCl was removed by repeated evaporations. The residue was dissolved in water and treated with the column of Dowex 1–X8 as mentioned above. The passing was evaporated and the residue was crystallized twice from 50% aqueous ethanol in colorless prisms (90 mg). M. p. 226–228°C. Anal. Found: C, 51.61; H, 9.48; N, 8.54%. Calcd. for C7H15O3N: C, 52.16; H, 9.38; N, 8.69%. The PMR spectrum (in D2O) showed a large singlet at δ 2.00 (9H) caused by N-trimethyl group, and two multiplets at δ 3.83–3.55 (3H) and δ 2.25–2.00 (2H) which were assignable to the protons of α-methine and γ-methylene groups, and those of β-methylene group of homoserine betaine, respectively. The IR spectrum is shown in Fig. 1. The absorption at 1610 cm⁻¹ indicates the presence of a carboxylate anion. When heated in 6 N HCl, the compound was converted into γ-lactone chloride which showed a characteristic absorption at 1780 cm⁻¹. The formation of γ-lactone supports that the hydroxyl group is attached to the γ-carbon atom as inferred from the PMR spectrum. Judging from the easiness to form lactone, homoserine betaine in the hydrolyzate must have been present as γ-lactone chloride, whose ring usually can be opened by filtering through a column of Dowex 1–X8 (OH⁻).

For comparison, homoserine betaine was synthesized from L-homoserine (6 g) using dimethyl sulfate as reported previously.2) Yield, 2.67 g (22.8 %). M. p. 227–228°C. Anal. Found: C, 51.63; H, 9.56; N, 8.62%. The IR (Fig. 1) and PMR spectra of the isolated and synthesized specimens of homoserine betaine were identical.

Homoserine betaine is an isomer of carnitine3) and atrinine4) isolated from animal tissues. These compounds are possessed of N-trimethyl, carboxyl and hydroxyl groups which form each isomer. Homoserine betaine has not been described in any papers before. Recently, YASUMOTO et al. also isolated it from the ovary of shellfish Callista brevisiphonata and the viscera of turbanshell Turbo argyrostoma (personal communication).

Further studies of the structure of the unknown base will be reported in the next paper.

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References

Fig. 1. Infrared absorption spectra of isolated and synthesized homoserine betaines (in KBr).

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