

Characterization of a Trimethylsilylated Derivative of 2-Pyrrolidone-5-carboxylic Acid

A trimethylsilylate derivative of 2-pyrrolidone-5-carboxylic acid has been prepared and isolated by preparative gas-liquid chromatography. The com-

ound was characterized as trimethylsilyl *N*-trimethylsilyl-2-pyrrolidone-5-carboxylate.

A recent technique for the trimethylsilylation of organic compounds permits the separation of carbohydrates (Sweeley *et al.*, 1963), organic acids (Brunell *et al.*, 1967; Martin and Swinehart, 1968) and amino acids (Mason and Smith, 1966) by gas-liquid chromatography (glc). Lee (1970) has developed a trimethylsilylation procedure to analyze for 2-pyrrolidone-5-carboxylic acid (I), which occurs often in foods. In this communication we wish to report the isolation and characterization of trimethylsilyl *N*-trimethylsilyl-2-pyrrolidone-5-carboxylate (II).

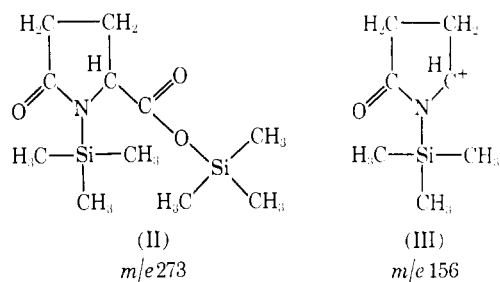
EXPERIMENTAL

One gram of I was dissolved in 100 ml of redistilled and dried pyridine. To this solution was added 20 ml of hexamethyldisilazane followed by 10 ml of trimethylchlorosilane. Since trimethylchlorosilane reacts vigorously with water, care must be used to keep reagents free from moisture. After 1 hr, precipitated salts were removed by centrifugation. The solution was then concentrated *in vacuo* to a thick syrup which was filtered through Whatman No. 52 filter paper to remove silicone polymers of high molecular weight. The derivative was isolated from the filtrate by preparative glc using an Aerograph Model A-700 equipped with 6 ft \times $\frac{1}{4}$ in. i.d. stainless steel column packed with 20% SF-96 on 60 to 80 mesh Chromosorb W and with a variable effluent-splitter. The instrument was operated isothermally at 150° C with inlet and detector temperature at 245° C; helium gas was the carrier gas at 80 ml per min. About 500 mg of II (retention time, 8 min) was collected and was subjected to analysis. *Anal.* Calcd. for $C_8H_{13}O_3N[Si(CH_3)_3]_2$ (II): C, 48.30; H, 8.41; N, 5.12. Found: C, 47.56; H, 8.24; N, 5.17.

RESULTS AND DISCUSSION

The ir spectrum revealed two major absorbance bands at 1700 cm^{-1} and 2950 cm^{-1} assigned to a carbonyl group and a methylene group, respectively. However, there were no bands arising from $>N-H$ or $-OH$ absorption. The mass spectrum, obtained from the combined time of flight mass spectrometer (Bendix Model 12-101A) with a Varian Aerograph glc (Model 1200), showed a molecular ion peak at m/e 273 for the assigned II and a base peak at m/e 156 ($M - 156 =$

117, and m/e for $COOSiMe_3 = 117$) which correspond to the following structures.



Other fragments were shown at m/e 73, 75, 157, 45, 259, and 258. The observed fragmentation of II to yield the decarboxylated product (III) is consistent with result reported (Tham *et al.*, 1968) for the fragmentation of the methyl ester of I. The assignment of structure II is supported by the pmr spectrum (Figure 1). The 100-MHz pmr spectrum yielded

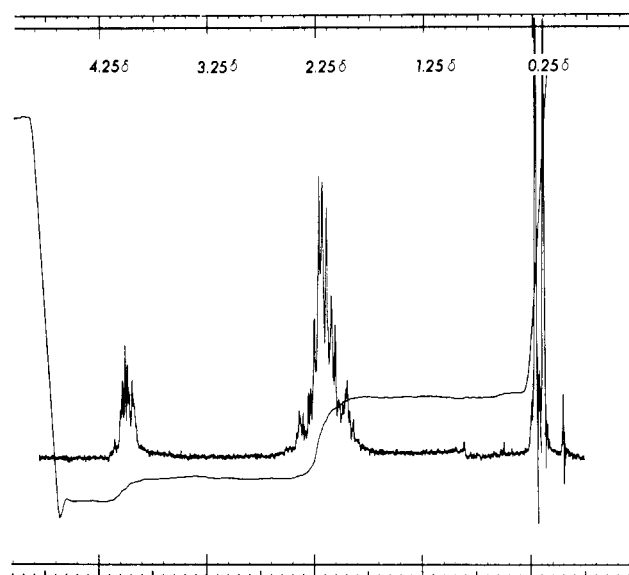


Figure 1. Pmr spectrum of trimethylsilyl *N*-trimethylsilyl 2-pyrrolidone-5-carboxylate

three sets of signals for CH, CH₂, and SiCH₃ which were found at δ 3.95, δ 2.10, and δ 0.15, respectively. The integrated area of these three sets of proton signals were in the ratio 0.94:4.0:17.7. These ratios are consistent with the view that II is ditrimethylsilylated I.

II is very unstable in the atmosphere and decomposes to I. When II was freshly prepared and purified, it was a clear viscous liquid which rapidly decomposed to a white solid. This white solid was introduced into a mass spectrometer equipped with a solid inlet system, and the major ion peak obtained was *m/e* 84 corresponding to decarboxylated I.

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