Notes

The preparation of diazopropane and diazobutane for the esterification of amino acids

During some recent work[1,2] on the gas chromatography of amino acids it was found that the esterification of the carboxylic group considerably improves the separation. Esterification can be carried out with diazomethane or by using a homologue of diazomethane. However, the high volatility of certain amino acid methyl esters made it impossible to obtain a quantitative recovery because extensive losses occurred during the evaporation of the samples. This note describes a general method for the esterification of the carboxylic group of amino acids and of their analogues using diazopropane and diazobutane.

For the preparation of these diazo compounds the microsynthesis procedure described by ROGER AND MA[3] was used.

Two ml of 50% aqueous potassium hydroxide were added to 5 ml of diethyl ether in a small flask. N-*n*-Butyl(or propyl)-N'-nitroso-nitroguanidine (1 g) suspended in diethyl ether was added through a small separatory funnel. The reaction was carried out in a water bath maintained at 45°. The diazo compound, passing through a condenser, is collected in ether.

For the esterification the hydrochlorides of the amino acids (5 mg) were dissolved in 4 ml of methanol. The ethereal solution of the diazo compound from the above reaction was added until a yellow colour persisted (about 5 ml). After 5 min the excess of the reagent is easily removed by evaporation under vacuum.

The esterification reaction employing these diazo compounds was found to be simple, rapid and to give excellent yields.

The choice of diazopropane or diazobutane will depend on the degree of volatility required in the esterified compounds.

An example of the utilization of diazopropane for the esterification and gas chromatographic separation of N-acetyl-aspartic acid and other acyl amino acids was recently reported[4].

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