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The separation of N- α -trifluoroacetyl-DL-lysine from potassium trifluoroacetate by liquid chromatography

When N- α -trifluoroacetyl-DL-lysine (N- α -TFA-lys) is prepared according to WEYGAND AND GEIGER¹, a major difficulty is the separation of the expected derivative from the salts.

We have isolated pure N- α -TFA-lys by means of liquid chromatography on a silica gel column. The eluting solvent was a mixture of methanol, 2-propanol and cyclohexane in the ratio 4:4:2 (Fig. 1.).

The absence of free lysine and of N- ϵ -TFA-lys was confirmed by high-voltage electrophoresis.

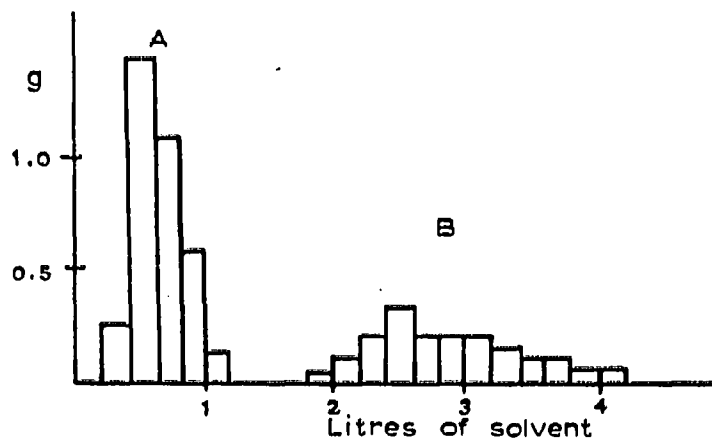


Fig. 1. Weights of material eluted from the column *versus* the volume of eluant. A = Potassium trifluoroacetate; B = N- α -TFA-lys.

Experimental

The synthesis of N- α -TFA-lys was performed according to WEYGAND AND GEIGER¹ with the following changes. Two moles of trifluoroacetic anhydride were used per mole of lysine; at the end of the reaction the excess acid was neutralized with potassium hydrogen carbonate. The yield was almost quantitative.

The silica gel column. A 175-g amount of silica gel (Merck) (particle diameter 0.2–0.5 mm) was used to fill a glass column of 3 cm I.D. to a height of 55 cm. The solvent flow-rate was maintained at 12 ml/min.

The products to be separated. The column was loaded with 1.7 g of N- α -TFA-lys and 3.3 g of potassium trifluoroacetate dissolved in 10 ml of the solvent. This solution had previously been kept at room temperature to avoid partial precipitation.

High-voltage electrophoresis was performed on Whatman 3 MM paper at pH 1.9 and at 60 V/cm for 1 h. The migration rate of ϵ -dinitrophenyllysine was used as the reference. The ratio of the migration rates, N- α -TFA-lys/N- ϵ -DNP-lys, was 1.73 and the ratio N- ϵ -TFA-lys/N- ϵ -DNP-lys was 1.33.

We have also identified the N- α -TFA-lys by its N- ϵ -phthalimide derivative. The elemental analysis was: calculated, C 51.6, H 4.03 %; found, C 51.73, H 4.15 %.

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