## PREPARATION OF $\epsilon$ -AMINOCAPROIC ACID

## (ACA) AND ITS HYDROCHLORIDE

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 $\varepsilon$ -Aminocaproic acid is used as an effective styptic and for the synthesis of N-benzoyl- $\varepsilon$ -aminocaproic acid, which is the starting material for the preparation of D, L- $\varepsilon$ -benzoyllysine and D, L-lysine.

 $\epsilon$ -Aminocaproic acid is usually prepared by the hydrolysis of  $\epsilon$ -caprolactam in the presence of hydrochloric acid. The solution of its hydrochloride salt is subsequently subjected to demineralization on an anion-exchange resin.

In the present work, data on the electrochemical demineralization of ACA are presented.

## EXPERIMENTAL

The electrodialyzer consists of cathodic and anodic chambers separated by an MA-40 anion-exchange membrane which is converted to the hydroxyl form before using by treatment with a 3% solution of sodium hydroxide, which is changed daily over a period of 3 days, and subsequently washed to neutrality with distilled water. Graphite was used for the anode, while stainless steel was employed for the cathode.

The solution of the hydrochloride of  $\varepsilon$ -aminocaproic acid (ACA) used was obtained from 113.1 g of caprolactam and 100 ml of hydrochloric acid (sp. gr. 1.18) with subsequent addition of 35 ml of hydrochloric acid, evaporation to the original volume, and dilution to a determined concentration with distilled water.



D. I. Mendeleev Moscow Institute of Chemical Engineering. Translated from Khimiko-Farmatsevticheskii Zhurnal, No. 10, pp. 38-39, October, 1968. Original article submitted July 1, 1968. This solution is subjected to electrolysis, during which the chloride ions migrate to the anodic chamber which is continuously filled by the incoming and outgoing distilled water. A solution of free ACA remains in the cathodic chamber. It was found that ACA undergoes virtually no electro-reduction on the cathode, and the only cathode reaction is the evolution of hydrogen.

The differential potentiometric titration curve of the ACA solution (Fig. 1) has three peaks corresponding to the equivalence points of hydrochloric acid, ACA hydrochloride, and ACA. The titration was performed with 0.1 N KOH under continuous stirring.

The time dependence of the electrodialysis on the ACA hydrochloride concn. is presented in Fig. 2. As the ACA hydrochloride concn. approaches 300 g/liter, the dependence deviates somewhat from linearity, i.e., the electrodialysis rate decreases.

Upon increasing the membrane current density to values up to approximately  $1 \text{ A/dm}^2$ , the electrodialysis time is markedly shortened (Fig. 3).

The optimum conditions for demineralizing ACA are as follows (for a two-celled electrodialyzer): current density 1.1-1.5 A/dm<sup>2</sup>, ACA hydrochloride concn. up to 450 g/liter, solution temp. 20°, ACA concn. after dialysis up to 335 g/liter, voltage 5 V (with a distance between electrodes of 17 mm), capacity of  $1 \text{ dm}^2$  of membrane 0.00298 kg of ACA per hour, substance yield ca. 95%, and current yield ca. 96.7%.

The electrical energy expended in the electrolysis is 0.85 kWh for 1 kg of ACA. The percentage of ACA in the product obtained is not less than 98.5%, that of chlorides not more than 0.002%, that of iron not more than 0.003%, that of heavy metals not more than 0.01%, and the ash content is not more than 1%.