PREPARATION OF ANTHRAQUINONES ON ION-EXCHANGE RESINS

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We have studied the possibility of using ion-exchange resins to isolate and purify combined anthraquinones. The experiment was performed with the aqueous mother liquor from the production of the preparation "Rhamnil" [1], which contains combined hydroxymethylanthraquinones (HMAs) consisting mainly of emodin (0.14%) and frangulin (0.2%) in a ratio of 2:1.

A study of the sorption of these compounds showed that the best sorbent for HMAs among cation-exchange resins is KU-1, and among anion-exchange resins ÉDÉ-10 P. However, while from the desorbate from KU-1 it is possible to obtain a crystalline powder easily subjected to further processing, the isolation of anthraquinones from the ÉDÉ-10 P desorbate is considerably more difficult. Consequently, we selected the adsorption of HMAs on KU-1 cation-exchange resin by the following procedure.

The mother solutions with the HMAs were filtered through a column containing the KU-1 resin in the H^+ form at the rate of 2 ml/min/cm^2. After saturation, the column was washed with a mixture of 5% caustic soda solution and 2% NH_4OH solution. The desorbate obtained was acidified with sulfuric acid to pH 1 and boiled for 0.5 h. The powder that deposited was filtered off and extracted with boiling benzene or ether. When the extract was concentrated, a red-orange crystalline powder deposited. Thin-layer chromatography on kieselguhr in the toluene–acetone–50% acetic acid (4:1:0.5) system [2] performed in parallel with that of an authentic sample showed that the substance isolated was frangula emodin contaminated with a small amount of polyphenols. The substance was identified by its melting point (254-256° C) and a mixed melting point, and also by its UV and IR spectra. According to chemical analysis, the purity of the sample was about 99.0%, and its yield was 0.6-0.65% (of the weight of the ion-exchange resin in the column). The yield of frangula emodin in the crystalline form was 50-55% calculated on its amount in the initial solution. About 20% of emodin, which did not crystallize, remained in the mother liquor in the form of a contaminated product.

LITERATURE CITED

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