CHROMATOGRAPHIC ISOLATION AND CONCENTRATION OF TRITERPENE ALCOHOLS ON COLUMNS WITH POLIKHROM-1 AND POLISORB-4T

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Polytetrafluoroethylene (Polikhrom-1) and a macroporous copolymer of styrene and divinylbenzene (Polisorb-4T) can be used to isolate and concentrate the triterpene fraction from the unsaponifiable part of ethereal extracts of *Betula* leaves.

Using as examples individual triterpene alcohols of the dammarane series it has been found that the sorption affinity of the alcohols rises considerably from Polikhrom-1 to Polisorb-4T. On this basis it has been proposed to use in succession a column with Polikhrom as a separating column and one with Polisorb-4T as a concentrating column.

Procedure. The sorbents (USSR products) with a grain size of 0.25-0.50 mm were freed from dust by sieving and from monomeric products by boiling them three times with ethanol (a new portion each time). They were stored in the form of suspensions in ethanol. Glass columns (10.0 × 0.8 cm) were filled with suspensions of the sorbents in ethanol (layer height 6 cm) and were washed successively with 50% ethanol (10 volumes) and with water (20 volumes). The layer of water in the Polikhrom-1 column was allowed to fall to the surface of the sorbent, and the unsaponifiable fraction (5 mg in 0.3 ml of diethyl ether) was deposited by means of a syringe on the top of the column whereupon it then occupied a 2- to 2.5-cm zone of the sorbent.

The triterpene fraction was eluted first with 90 ml of the eluent ethanol—water (45:55, v/v). The eluate was diluted with water to a 15% concentration of ethanol and was passed through the column with Polisorb-4T at the rate of 1.5-2.0 ml/min. After the complete issuance of the eluate from the column, the triterpene fraction was eluted with 15 ml of diethyl ether, and the eluate was dried over anhydrous sodium sulfate, filtered, and evaporated to dryness.

The columns were regenerated by being washed successively with ethanol, with 50% ethanol, and with water (~6, 6, and 20 volumes) and they were used repeatedly, not less than 20 times.

The conditions given in the procedure were established experimentally both on the total triterpene fraction and on individual compounds with monitoring at all stages by spectrophotometry and TLC and GLC.

It must be mentioned that the solvent system ethanol—water (45:55, v/v) eluted the whole of the triterpene fraction and only an insignificant fraction of the sterols from Polikhrom-1. The sterols and other less polar components of the unsaponifable fraction (higher hydrocarbons, alcohols, carotenoids, etc.), amounting to 80-90% of its weight, remained on the column.

The adsorption filtration of triterpenes from 15% ethanol through Polisorb-4T ensured a high degree of their extraction (0.95-0.98), and 15 ml of diethyl ether proved to be sufficient for their complete desorption.

The use of macroporous sorbents and of polytetrafluoroethylene has advantages over silica gel or alumina in the possibility of the elution of the triterpene fraction from the column first, the absence of residual sorption, and also the non-hazardousness of working with aqueous solutions of ethanol.

The procedure for isolating the concentrating triterpene alcohols has been used to prepare samples before GLC analysis.

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