

PURIFICATION OF THE TOTAL GLYCOSIDES OF ETHANOLIC EXTRACTS OF
Polygonatum stenophyllum ON HYDROPHOBIC SORBENTS BEFORE
QUANTITATIVE ANALYSIS

L. I. Berezhevskaya, Zh. I. Ul'kina,
and L. I. Glebko

UDC 547.918+543.544.4

We have used the macroporous sorbents Polisorb-4T (a copolymer of styrene and divinylbenzene, USSR) and Sepharon SE (a copolymer of styrene and ethylene dimethacrylate, Czechoslovakia), and also the hydrophobic sorbent Polikhrom-1 (polytetrafluoroethylene, USSR) in order to free the glycoside fraction of ethanolic extracts of plants from accompanying compounds. As compared with the re-extraction of the glycoside into n-butanol, the macroporous sorbents, and also polytetrafluoroethylene, provide the possibility of achieving higher quality of purification, which is important in a number of cases.

The sorbents were studied on ethanolic extracts from the roots and leaves of *Polygonatum stenophyllum* Maxim., the glycosidic fraction of which is represented mainly by glycosides of pennogenin (17 α -hydroxydiosgenin) [1].

It was found that the affinity of the glycosides for the sorbent increases in the sequence Polikhrom-1, Polisorb-4T, and Sepharon SE. Thus, the retention times of the least polar glycoside on elution with 40% ethanol at the rate of 2.5 ml/min amounted to 7.2, 15.2, and 18.3 min, respectively. The loading capacity of the sorbents, determined by the frontal deposition of the same amount of extract on the sorbents and elution from the column until the initial material appeared, increased in the same sequence. It amounted to 5.5, 11, and 35.5 mg of dry extract per 1 g of sorbent, respectively.

As we see, the macroporous sorbents have advantageous characteristics as compared with polytetrafluoroethylene, although the latter can also be used successfully for the purposes mentioned. It has also been established that the irreversible sorption of the glycosides on all three sorbents is negligibly small.

In order to avoid large volumes of eluents the glycosides were purified on sorbents only to remove polar impurities, the nonpolar impurities having been removed previously by extracting the dry material with n-hexane or chloroform.

The sorbents, freed from monomers by boiling with ethanol three times and having a grain size of 25-35 μ m, were charged into the columns in the form of suspensions in 96% ethanol and they were then equilibrated with water.

The samples were introduced into the columns by the adsorption filtration of ethanolic extracts previously diluted with water to an ethanolic concentration of 5-7%, at the rate of 1 ml/min. The polar impurities were eluted first with 10% ethanol at the rate of 2.5 ml/min, and the eluate was discarded. The glycosides were eluted with 96% ethanol and the solution was evaporated to dryness. The columns were again equilibrated with water and were re-used not less than 25 times. The necessary dimensions of the columns and the volumes of the eluents were established experimentally with monitoring by TLC.

The method of purification was used before the quantitative determination of the glycosides in terms of their common aglycone with the aid of the color reaction in 75% sulfuric acid [2].

LITERATURE CITED

1. L. I. Strigina, E. V. Kol'chuk, A. I. Kalinovskii, and V. V. Isakov, *Khim. Prir. Soedin.*, 580 (1977).
2. L. I. Glebko, N. N. Syromolotova, L. I. Strigina, and P. G. Gorovoi, *Khim. Prir. Soedin.*, 332 (1982).

Pacific Ocean Institute of Bioorganic Chemistry, Far Eastern Scientific Center, Academy of Sciences of the USSR, Vladivostok. Translated from *Khimiya Prirodnikh Soedinenii*, No. 5, pp. 669-670, September-October, 1984. Original article submitted July 14, 1983.