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STUDY OF THE KINETICS OF THE EXTRACTION OF FLAVONOIDS FROM PLANT RAW MATERIAL

I. EXTRACTION OF RUTIN FROM *Sida hermaphrodita*

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The influence of the concentration of ethanol (50, 60, and 70%, of the degree of grinding of the plant raw material, and of the time of maceration, on the yield of rutin from the epigeal part of *Virginia sida*, family Malvaceae has been studied. It has been established that the highest yield of rutin can be obtained from raw material ground to 2.5-3.0 mm with the use of 70% ethanol and a time of maceration of 12 h. In an investigation of the influence of the concentration of ethanol and the time of steeping on the yield of rutin from raw material ground to 2.5-2.0 mm, regression equations for 50, 60, and 70% ethanolic extractions have been derived.

Questions of the theory and practice of the extraction of raw materials for obtaining medicinal preparations — tinctures and extracts — have been discussed in detail in the literature [1], but the kinetics of the extraction of flavonoids has been little studied [2, 3], although a number of these compounds are used as drugs [4].

We have established by preliminary investigation of some plants of the Malvaceae family that when mucilages are present in the plants the extraction of flavonoids is inhibited. In order to determine the optimum conditions for the extraction of flavonoids we have studied the influence of the degree of grinding of the raw material, the time of steeping, and the concentration of ethanol on the yield of rutin from the epigeal part of *Sida hermaphrodita* Rusby (Virginia sids) in which, according to our results, up to 5% of rutin may be present [5].

Table 1 gives the results on the influence of the degree of grinding of the raw material on the yield of rutin as functions of the time of steeping and of the concentration of ethanol.

TABLE 1. Amounts of Rutin in Extracts as Functions of the Degree of Grinding of the Raw Material, the Time of Steeping, and the Concentration of Ethanol*

Concentration of ethanol, %	Degree of grinding of the raw material, mm	Time of steeping, h				
		1	3	6	12	24
50	1	1,64	1,2	1,62	1,54	1,61
60	1	1,55	1,55	1,50	2,40	1,96
70	1	1,47	1,50	1,55	1,96	2,00
50	2	1,48	1,44	2,16	2,05	1,64
60	2	2,30	2,16	2,22	1,96	1,9
70	2	2,05	2,10	2,22	2,46	2,56
50	3	1,47	1,44	2,16	1,56	2,10
60	3	2,16	2,10	2,16	2,10	2,12
70	3	3,00	2,16	2,10	2,35	2,27
50	7	0,61	1,03	1,34	1,43	1,35
60	7	1,24	1,32	1,44	1,89	1,80
70	7	1,60	1,19	1,42	1,48	1,37

*Sieves Nos. 10, 20, 30, 50, and 70 (GOST [State Standard] 214-57) were used [7].

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TABLE 2. Deviations of the Values of the Amounts of Rutin Extracted Found Experimentally and Theoretically

Time, h	Amount of rutin extracted, %		
	$Y_{practical}$	$Y_{theoretical}$	difference $Y-\hat{Y}$
Using 50% ethanol			
1	1,47	1,47	0
3	1,44	1,44	0
6	2,16	2,17	0,01
Using 60% ethanol			
1	2,16	2,16	0
3	2,10	2,10	0
6	2,16	2,15	0,01
Using 70% ethanol			
1	3,00	3,00	0
3	2,16	2,16	0
6	2,10	2,10	0

It was established that for finely-ground raw materials (1-2 mm) the extraction of rutin took place less intensively, and to extract the maximum amount of rutin it was necessary to macerate the maw material for 12 h. An increase in the time of steeping to 24 h using 50% and 60% ethanol led to a fall in the amount of rutin extracted. When 70% ethanol was used, the yield of rutin from the raw material ground to 3 mm increased in proportion to the time of steeping, but an increase in the size of the ground particles of the raw material to 7 mm led to a retardation of the passage of the rutin into the extract. In all cases, the extraction process took place most intensively in the first few hours, with 70% of the rutin present in the raw material being extracted.

The results of the experiments (see Table 1) show that the highest yield of rutin is obtained when the raw material is extracted with 70% ethanol at a degree of grinding of the raw material of 3 mm.

The mathematical treatment of the experimental results was carried out by the following scheme. For each type of raw material graphs were plotted of the dependence of the yield of rutin on the time of steeping. A suitable relation was selected for this curve. In this way, we found the regression equations

$$Y = \frac{t}{at^2 + bt + c}, \quad (1)$$

where Y is the yield of rutin, %, and t is the time of steeping, h.

On substituting in this equation vlaues of the time t of 1, 3, and 6 h, and the percentage yields of rutin Y corresponding to them we obtained a system of equations by the solution of which the values of the coefficients a, b, and c were found.

The regression equations for 50, 60, and 70% ethanolic extractions at a degree of comminution of the raw material of 3 mm had the following form:

$$Y = \frac{t}{-0.0926\,t^2 + 1.0687\,t - 0.2954}, \quad (2)$$

$$Y = \frac{t}{-0.0064\,t^2 + 0.5089\,t - 0.0385}, \quad (3)$$

$$Y = \frac{t}{-0.0080\,t^2 + 0.5612\,t - 0.2199}. \quad (4)$$

By using these equations it is possible to calculate the theoretical amount of rutin extracted. By giving definite values of t we found the theoretical values of the amounts of rutin and compared them with the experimental results (Table 2).

EXPERIMENTAL

The raw material (1.4 g) with the appropriate degree of grinding was extracted with 50, 60, and 70% ethanol for from 1 to 24 h at room temperature, the extract was filtered into a 50-ml measuring flask, and the volume of liquid was made up to the mark with the appropriate concentration of ethanol. The amount of rutin in the extract was determined spectrophotometri-

cally. For this purpose, 1 ml of the extract obtained was mixed in a 50-ml measuring flask with 4 ml of a 0.3% solution of caustic soda, the volume of the solution was made up to 50 ml with water, and the optical density was determined on a SF-4A spectrophotometer at 410 nm in a cell with a layer thickness of 30 mm. In the case of a high optical density, the solution was diluted with water. A standard solution of rutin was prepared by dissolving 0.025 g of rutin in a 100-ml measuring flask in 96% ethanol. The solution obtained (10 ml) was transferred to a 50-ml measuring flask, 4 mole of 0.03% aqueous caustic soda was added, and the solution was made up to the mark and then the optical density of the resulting solution was determined at 410 nm in cell with a layer thickness of 30 mm.

CONCLUSIONS

It has been established that the greatest amount of rutin can be extracted from the eipgeal part of the plant by using 70% ethanol and a degree of grinding of the raw material material of 3 mm. Regression equations have been derived which permit the time necessary for extracting a given amount of rutin to be determined theoretically.

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ARTENOLIDE — A NEW DISESQUITERPENOID FROM *Artemisia absinthium*

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A new sesquiterpene lactone, artenolide — $C_{30}H_{40}O_8$, mp 163-174°C (ethanol) — has been isolated from the epigeal part of *Artemesia absinthium* L. On the basis of an analysis of spectral characteristics (IR, mass, and ^1H and ^{13}C NMR spectra) of the lactone itself and of a transformation product, a diguaiane structure is suggested for artenolide.

The disesquiterpenoids absinthin [1], anabasin [2], and absintholide [3] have been isolated from *Artemesia absinthium* (common wormwood), family Asteraceae (Compositeae). In the present paper we give information on the isolation and a proof of the structure of a new disesquiterpenoid from common wormwood growing in the Tashkent province — artenolide (I) with the composition $C_{30}H_{40}O_8$, mp 163-174°C (ethanol).

The mass spectrum of (I) did not contain the peak of the molecular ion, but there was a peak of an ion with m/z 510 (M — 18). A broad band at 3350-3500 cm^{-1} in the IR spectrum of (I) showed the presence of hydroxy groups, while bands at 1752-1768 cm^{-1} were due to γ-

*Deceased.

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