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ISOLATION OF ECDYSTERONE FROM THE ROOTS OF *Rhaponticum carthamoides*

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The solubility of ecdysterone in individual solvents and mixtures has been studied. A method has been developed for isolating ecdysterone from the roots of *Rhaponticum carthamoides*. Extraction from the raw material was carried out with methanol. The catty oils and tanning and resinous substances were eliminated from the concentrated and water-diluted extract by treatment with chloroform. The combined ecdysteroids were extracted from the purified aqueous solution with chloroform—isopropanol (1:1). They were freed from pigments by chromatography on alumina (Brockmann activity grade II) with elution by methanol—chloroform (1:2). The product was recrystallized from methanol—ethyl acetate (1:9), giving 0.05% (on the weight of the raw material) of ecdysterone.

The perennial herbaceous plant *Rhaponticum carthamoides* (Willd.) Iljin. (maral root, safflower leuzea, safflower rhubarb, safflower centaury) growing in the Asiatic part of the USSR and also in the Mongolian People's Republic [1] has been used from ancient times in folk medicine but its introduction into cultivation as a medicinal and fodder plant began only recently [2-4]. At the present time, galenical preparations with a tonic and stimulating action are being made from the roots and rhizomes of *Rh. carthamoides* [5]. An extract of the roots is one of the components of the beverage "Sayany."

The main biologically active substance of the roots of *Rh. carthamoides* — ecdysterone — was discovered in the Institute of the Chemistry of Plant Substances of the Academy of Sciences of the Uzbek SSR by N. K. Abubakirov et al. [6]. The reserves of the wild and cultivated *Rh. carthamoides* make it a promising raw material for the production of ecdysterone. We have investigated the processes of isolating ecdysterone from the roots and rhizomes of this plant, which we obtained from the factory for the primary treatment of medicinal raw material. To select the optimum solvent for the various stages of the isolation and purification of ecdysterone we investigated its solubility in several solvents and mixtures of them:

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Solvent, %	g/100 ml	Solvent	g/100 ml
Acetone 100	0.193	Methanol	7.5
95	0.565	Water	0.19
90	0.737	Chloroform–methanol,	
85	1.13	4:1	1.0
80	1.18	2:1	3.21
Ethanol 95	2.8	Water-saturated n-	
90	2.94	butanol	4.1
80	4.61	Water-saturated ethyl	
70	6.10	acetate	0.059

The roots of the *Rh. carthamoides* were extracted with methanol. The extract was freed from ballast substances and pigments by treatment with chloroform and by chromatography on alumina. The best result was obtained on elution by chloroform–methanol (2:1). The ratio of the total material to sorbent was from 1:5 to 1:15. The optimum sorption of impurities was achieved at ratios of 1:10-1:12. The methanol extraction of the roots was carried out on large-scale laboratory apparatus (see Experimental part), and 0.05% of ecdysterone on the weight of the raw material was isolated.

EXPERIMENTAL

Extraction of the Raw Material. The roots and rhizomes of *Rhaponticum carthamoides* (50 kg) were extracted with methanol in a 200-liter extractor by the steeping method with a solid:liquid ratio of 1:12 five times. The extract was evaporated to a volume of 8 liters and it was then diluted with water to 15 liters and was treated with chloroform (5 × 10 liters). The ecdysteroids were extracted from the purified aqueous methanolic solution with a mixture of chloroform and isopropanol (1:1) (5 × 15 liters). The combined chloroform-isopropanolic extract was evaporated to the state of the syrupy mass.

Isolation of Ecdysterone. The concentrated extract was dissolved in 3 liters of methanol–chloroform (1:2) and passed through a column charged with 5 kg of alumina (Brockmann activity grade II), the diameter of the column being 100 mm and the height of the layer of sorbent 500 mm. The eluate obtained (10 liters) was evaporated to dryness and the residue was crystallized from 600 ml of methanol–ethyl acetate (1:9). After a day, the crystals that had deposited (28.5 g) were separated off and were recrystallized from 600 ml of the same mixture. The crystals were separated off, washed with 70 ml of ethyl acetate, and dried at 100°C. This gave 24.8 g of ecdysterone (0.05% on the weight of the raw material).

SUMMARY

1. The solubility of ecdysterone in several individual solvents and mixtures of them has been studied.

2. A method has been developed for isolating ecdysterone from the roots and rhizomes of *Rhaponticum carthamoides* with a yield of 0.05% on the weight of the raw material.

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