

ISOLATION OF ECDYSTERONE FROM THE INFLORESCENCES OF

Rhaponticum integrifolium

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A method has been developed for isolating ecdysterone from the inflorescences of *Rh. integrifolium*. The yield is 0.15% on the weight of the raw material.

Ecdysterone — a molting hormone — has been isolated previously from inflorescences of *Rhaponticum integrifolium* C. Winkl. [1]. It has been found possible to use ecdysterone as a drug with an anabolic action [2-4].

A study of its insecticidal properties has shown that in concentrations of from 0.25 to 1% ecdysterone lowers the reductive capacity of the spider mite by 50%, and causes a mortality of the cotton aphid of from 39 to 98%, depending on the concentration (0.01-1%). The number of caterpillars of the cutworm moth, a pest of the cotton fields, hatching out was reduced by ecdysterone by 81-87% in comparison with a control [5].

The possibility has been investigated of using ecdysterone for the intensification of silk production [6], without excluding the possibility of using it as a hormone preparation for increasing the productivity of animal husbandry [7].

As a rule, the plants in which ecdysterone is found contain it in minute amounts. The finding of new sources of plant raw material rich in ecdysterone will enable this insect-molting hormone to be obtained in sufficient amounts for practical use in the national economy.

One such source is *Rhaponticum integrifolium*, the flower heads of which contain about 0.5-0.6% of ecdysterone [8].

We have made a search for an economic method of obtaining ecdysterone from the flower heads of this plant.

The raw material was extracted with methanol and the extract was evaporated, diluted with water, and treated with chloroform to eliminate fatty oils and tanning and resinous substances. The ecdysones were extracted from the purified aqueous methanolic solution with a mixture of chloroform and isopropanol, the sugars and other hydrophilic impurities remaining in the aqueous methanolic solution. The extract so obtained was evaporated to dryness, and the residue was freed from pigments on alumina with elution by chloroform-methanol. The dry residue obtained after the evaporation of the eluates was twice recrystallized from methanol-ethyl acetate.

The yield of ecdysterone with mp 238-240°C was 0.15% on the weight of the raw material.

EXPERIMENTAL

Extraction of the Raw Material. The flower heads of *Rhaponticum integrifolium* (containing 0.46% on the weight of the raw material of ecdysterone as determined by a chromatographic method [8]) collected in Osh province of the Kirghiz SSR in June, 1977 (20 kg) was crushed on a roller mill and extracted with methanol five times in a 200-liter extractor by the steeping method with a liquor ratio of the process of 1:26. The extract was evaporat-

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ed to a volume of 4 liters and was diluted with 4 liters of water. The aqueous methanolic solution (8 liters) was treated with chloroform (5 × 5 liters) and the ecdysones were extracted from the purified aqueous methanolic solution with chloroform-isopropanol (1:1) (5 × 4 liters). The combined chloroform-isopropanol extract was evaporated to the state of a syrupy mass.

Isolation of Ecdysterone. The concentrated extract was dissolved in 1500 ml of a methanol-chloroform (1:2) and passed through a column filled with 2.1 kg of alumina (Brockman activity grade II), the diameter of the column being 100 mm and the height of the layer of sorbent 240 mm. The eluate obtained (6 liters) was evaporated to dryness (67.5 g). The residue was crystallized from 500 ml of methanol-ethyl acetate (1:9), and after a day the crystals that had deposited (33 g containing 89% of ecdysterone) were removed and recrystallized from 540 ml of the same mixture. The crystals were separated by filtration, washed with 50 ml of ethyl acetate, and dried at 100°C. This gave 31 g of ecdysterone (0.15% on the weight of the raw material) with a purity of 96% having mp 238-240°C.

SUMMARY

A method has been developed for isolating ecdysterone from the inflorescences of *Rhapon-ticum integrifolium* C. Winkl. (family Compositae) with a yield of 0.15% on the weight of the raw material.

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