THE ISOLATION OF OLITORISIDE

M. T. Turakhozhaev, M.-R. I. Shamsutdinov, and T. T. Shakirov

UDC 615.45:615.711.5

Olitoriside – a steroid glycoside isolated from the seeds of Corchorus olitorius (potherb jute) [1, 2] – has found use in the treatment of cardiac insufficiency [3]. The methods for its preparation proposed previously were complex and gave comparatively low yields of the substance [1, 2]. We have developed a new method for the isolation of olitoriside increasing its yield to 0.2% of the weight of the seeds [4].

In order to select solvents for the extraction and further purification of olitoriside, we have determined the solubility of the glycosides of the plant in various solvents (Table 1).

It can be seen from Table 1 that to extract olitoriside from the seeds the best solvents are methanol and ethanol. Acetone is the most convenient for the purification of olitoriside from corchoroside.

The glycosides were extracted from the ground and defatted seeds of the plant with ethanol by steeping without heating. After four or five steepings, the glycosides had been extracted almost completely.

As is well known, the seeds of potherb jute contain a considerable amount of free sugars which, on extraction with ethanol, pass into the extract. Acetone was used to remove the sugars from the extract [4]. The best results were obtained by concentrating the combined ethanolic extract to 1/30 of its initial volume and treating the concentrate with a fourfold volume of acetone, whereupon the sugars were precipitated almost completely with comparatively slight losses of glycosides.

To remove hydrophobic substances, the extract after the precipitation of the sugars and the distillation of the acetone from it was treated with ether, whereupon the glycosides precipitated in the form of a dark viscous mass. The fatty oils and resinous and colored substances remained in the ethereal solution. To determine the amount of ether necessary for precipitating the glycosides, we studied the solubility of olitoriside and corchoroside in mixtures of ether and ethanol in various proportions.

When the concentrated extract was treated with a four- to sixfold amount of ether, the losses of olitoriside were insignificant:

Solubility, g/100 ml, 20°C

Ethanol–ether	Olitoriside	Corchoroside
1 : 1	0.27	0.9
1 : 2	0.17	0.4
1 : 4	0.07	0.2
1 : 6	0.03	0.16

To obtain the glycosides from the extract treated with acetone and ether, it was first dissolved in water. From the aqueous solution, the volume of which was 1/10 of the weight of the seeds, first the corchoroside (by means of chloroform) and then the olitoriside were selectively extracted with organic solvents (see Table 1).

The choice of solvent for the extraction of the olitoriside from the aquous solution was made in model experiments. A 2% solution of olitoriside in aqueous methanol (5% methanol) saturated with 10% of common

Institute of the Chemistry of Plant Substances, Academy of Sciences of the Uzbek SSR. Translated from Khimiya Prirodynkh Soedinenii, No. 6, pp. 702-705, November-December, 1970. Original article submitted September 13, 1970.

TABLE 1

Solvent	Solubility, g/100 ml			
	olitoriside		corchoroside	
	20°	bp	20°	bp
Methanol	25,3	84,0	3,6	24,6
Ethanol	7,3	40,0	2,0	8,0
Propanol	5,9	35,0	—	—
Isopropanol	5,6	33,3	1,0	8,0
n-Butanol	5,0	32,0	2,6	15,0
Acetone	0,3	4,5	3,0	8,1
Chloroform	0,04	0,06	0,3	4,8
Ether	—	—	0,07	0,14
Water	1,2	—	Traces	—

TABLE 2

Solvent	Number of extractions	Total consumption of solvent	Yield of olitoriside as a percentage of the amount in the solution
Chloroform–methanol, 2 : 1, 3 : 1	10	500	not extracted
Chloroform–ethanol, 1 : 1	9	450	65
Chloroform–ethanol, 2 : 1	11	550	80
Chloroform–propanol, 1 : 1	8	400	96
Chloroform–isopropanol, 1 : 1	9	450	93
Chloroform–isopropanol, 2 : 1	9	450	89
Chloroform–butanol, 1 : 1	7	350	84,4
Benzene–isopropanol, 1 : 1	10	500	25

salt was extracted with various mixtures of organic solvents (Table 2). The salt was added to decrease the solubility of the glycosides and improve the separation of the phases.

It follows from Table 2 that the best extractant for the olitoriside from an aqueous solution is a mixture of chloroform and propanol or isopropanol in a ratio of 1 : 1.

To determine satisfactory conditions for the liquid-liquid extraction, a 2% aqueous solution of olitoriside containing 5% of methanol was extracted with a mixture of chloroform and propanol with and without the addition of various amounts of salt.

The addition of up to 10% of common salt in the liquid-liquid extraction considerably decreased the consumption of organic solvent and reduced the loss of olitoriside

NaCl content	Number of extractions	Total consumption of solvents, liters	Yield of olitoriside as a percentage of the amount initially present in the solution
None	12	600	87
5%	9	450	91
10%	8	400	96

EXPERIMENTAL

The Defatting of the Seeds. An admixture of 100 kg of the ground seeds of potherb jute and 2 kg of wood shavings was charged into an extractor and defatted with gasoline by the steeping method. The gasoline residues were eliminated from the defatted seeds by means of vacuum.

Extraction of the Glycosides. An 80-kg sample of the defatted seeds was extracted with 95% ethanol by steeping. Four extracts were made, which were combined and concentrated in a vacuum steam apparatus to 10 liters (distillation temperature not exceeding 45°C, vacuum of 600-700 mm Hg).

Precipitation of the Sugars. In a mixer with the stirrer operating, 40 liters of acetone was added in a thin stream to the concentrated extract. The acetonic solution was separated from the precipitated sugars by decantation and concentrated to 5-6 liters.

Precipitation of the Glycosides from the Extract. In a mixer with the stirrer operating, 25 liters of ether was added in a thin stream to the concentrated extract. The ether-insoluble glycosides and the hydrophilic impurities precipitated in the form of a viscous mass. The hydrophobic impurities passed into the ethereal solution. The precipitated mass was washed with 5 liters of ether and dried.

Extraction of the Corchloroside. The precipitate freed from ether was first dissolved in 10 liters of water in an apparatus with a stirrer. Then 1 kg of common salt and 3 liters of chloroform were added to the aqueous solution. The mixture was stirred for 10 min and, after settling, the lower chloroform layer was decanted off. This operation was repeated three times.

Extraction of Olitoriside. After the extraction of the corchoroside, the olitoriside was isolated from the aqueous solution with a mixture of chloroform and isopropanol in a ratio of 1 : 1. The olitoriside was extracted with 1-liter portions of the ethanol–chloroform mixture nine times. There was no olitoriside in

the first extract. The remaining eight extracts were combined and concentrated until the first crystals of the glycoside appeared (temperature of distillation not above 45°C, vacuum of 600-700 mm Hg).

Crystallization of the Olitoriside. The concentrated extract (2-3 liters) was treated with an equal amount of water and left to crystallize. After 3 days, the crystals that had deposited were separated off and washed with a small amount of ice water.

Recrystallization of the Olitoriside. The crude crystals were dissolved in the minimum amount of hot acetone, and the solution was filtered through a hot filter and left to crystallize. After 5-6 h, the crystals that had deposited were filtered off with suction and dried in the air. The yield of olitoriside was 200 g or 0.2% of the weight of the seeds.

SUMMARY

1. The solubility of the glycosides of potherb jute in various solvents has been studied.

2. The conditions for the purification of the extract from sugars and hydrophobic substances and of the extraction of olitoriside from aqueous solutions with organic solvents have been investigated.

3. A method of obtaining olitoriside, which gives a higher yield of the material, is proposed.

LITERATURE CITED

1. N. K. Abubakirov, V. A. Maslennikova, and M. B. Gorovits, DAN UzSSR, 6, 23, 1957.
2. N. K. Abubakirov, V. A. Maslennikova, and M. B. Gorovits, ZhOKh, 28, 2279, 1958.
3. A. D. Turova, Seventh All-Union Conference of Pharmacologists (Subjects of Lectures) [in Russian], Khar'kov, 1958, p. 47.
4. M. T. Turakhozhaev, M.-R. I. Shamsutdinov, T. T. Shakirov, N. K. Abubakirov, and V. A. Maslennikova, USSR Patent No. 253,295; Byull. Izobr., No. 30, 88, 1969.