

ISOLATION OF BERBERINE

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Berberine is used in medicine as a cholagogue [1] and is the starting material for the preparation of a new drug, and therefore the necessity for its isolation has arisen. For this purpose we have investigated the bark and wood of the roots of *Berberis oblonga*. It was known previously that berberine was isolated from some species of *Berberis* [2-6]. We extracted our raw material by the methods described. However, berberine was obtained in comparatively low yield (Table 1) since in the cited reference the use of two extractions with the preliminary moistening of the raw material with 10% NH₄OH to liberate the tertiary bases was envisaged, which subsequently complicates the isolation of the quaternary bases.

In view of this, we extracted the berberine from the bark and wood of the roots of *Berberis oblonga* with ethanol directly from the raw material without preliminary wetting with ammonia. The concentrated extract was treated with sodium iodide to give berberine iodide [3]. The combined tertiary bases were isolated from the mother solution. The bark of the roots of *Berberis oblonga* yielded 2% of berberine and 0.6% of combined tertiary bases, and the wood yielded 0.8% and 0.2%, respectively.

EXPERIMENTAL

Isolation of Berberine

Extraction. The ground wood of the roots of *Berberis oblonga* (25.0 kg) was extracted with 95% ethanol countercurrently in a battery of extractors. The extract was concentrated in a vacuum evaporator to 40 liters.

Preparation of Technical Berberine Iodide. With stirring, a 50% solution of NaI was added in a thin stream to the concentrated extract. After 1-2 h, the crystals of berberine iodide that had deposited were separated off and were dried in a vacuum-drying cabinet. This gave 650.0 g of technical berberine iodide.

Purification of the Technical Product and Recrystallization. The technical product (650.0 g) was washed twice with acetone, dried in a vacuum-drying cabinet, and dissolved in a minimum amount of boiling methanol (1:100), and the solution was filtered hot and was cooled. The crystals that deposited were

TABLE 1. Comparative Results on the Isolation of Berberine

Extraction	Yield, % on the weight of the air-dry raw material			
	wood of the roots		bark of the roots	
	berberine	sum of the tertiary bases	berberine	sum of the tertiary bases
Acetone-water (1:1); chloroform [2]	not isolated	—	—	—
Chloroform; ethanol [3]	0.24	0.35	1.1	1.2
Ethanol; chloroform (berberine chloride) [4]	0.05	0.1	0.1	0.2
Methanol (with the preliminary moistening of the raw material with 5% HCL)	0.2	—	0.5	—

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separated off and dried in the air. The yield of berberine iodide from the bases of the roots was 200.0 g, mp 262-263°C (from methanol); R_f 0.3 [TLC, fixed layer of silica gel, chloroform-methanol (9:1)].

Extraction of the Tertiary Bases. The residual filtrate was made alkaline to pH 9-10 with 25% NH_4OH and was extracted with chloroform. The extract was concentrated and treated with 10% H_2SO_4 . The sulfuric acid extract of the alkaloids was washed with chloroform, and the washed acid solution was made alkaline with 25% NH_4OH and extracted with chloroform. This gave 60.0 g of combined alkaloids (tertiary bases).

CONCLUSIONS

The conditions for the extraction and isolation from the wood and bark of the roots of Berberis oblonga have been studied.

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