

# Alkaloids of *Thalictrum* V. Quaternary Compounds from *T. rugosum* and *T. adiantifolium*

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Magnoflorine and berberine were isolated as iodides from the quaternary alkaloid fraction of the roots of *Thalictrum rugosum* Ait. and *T. minus* var. *adiantifolium* Hort.

THIS PAPER deals with the isolation of magnoflorine and berberine from the roots of *Thalictrum rugosum* Ait. (*T. glaucum* Desf.) and *T. minus* var. *adiantifolium* Hort. (*T. adiantoids* Hort.; *T. adianthifolium* Bess). Both alkaloids were isolated as iodides from the quaternary fraction. Their identity was established by comparison of general chemical properties and infrared spectra with authentic samples.

Studies on additional alkaloid fractions of these plants are being continued by the authors.

## EXPERIMENTAL<sup>1</sup>

**Material.**—Roots of *T. rugosum* were obtained from Wayside Gardens, Mentor, Ohio, while roots of *T. minus* var. *adiantifolium* were obtained from Sunbeam Farm, Westlake, Ohio. In each case a minimum of 100 plants are being cultivated in The Ohio State University College of Pharmacy Medicinal Plant Garden. In addition, herbarium specimens have been made of each.

***T. rugosum*.**—Milled roots, 6.2 Kg., were extracted with methanol in a continuous extractor until a negative test of the extractant was obtained with Valser's reagent. The methanol extract was concentrated almost to dryness, *in vacuo*, and poured while stirring into warm 5% acetic acid. The insoluble material was repeatedly extracted with 5% acetic acid until a negative test was obtained with Valser's reagent. The acidic solution was freed from acidic and neutral substances by extraction with ether, and then made alkaline with ammonium hydroxide solution and extracted exhaustively with ether. The mother liquor was made weakly acidic with dilute hydrochloric acid, and an aqueous saturated solution of ammonium reineckate was

added to precipitate the quaternary alkaloids as reineckates. The alkaloidal reineckate precipitate was dried at room temperature and then dissolved in acetone and the solution filtered. To the filtrate was added slowly an aqueous saturated solution of silver sulfate, thereby changing the alkaloid-reineckate complex into the alkaloid sulfates; the insoluble silver reineckate was removed by filtration. The sulfate solution was concentrated, *in vacuo*, filtered, and to the filtrate was added an aqueous saturated solution of potassium iodide. The resulting precipitate was removed by filtration and then crystallized and recrystallized in methanol to give 0.06 Gm. of colorless prisms, m.p. 252° dec.,  $[\alpha]_D^{20} +203^\circ$  (methanol). The iodide was identified as magnoflorine iodide by comparison of infrared spectra.

Upon concentration of the mother liquor, another crystalline substance was obtained. This substance was crystallized and recrystallized from methanol to obtain 1.88 Gm. of yellow needles, m.p. 262° dec. The infrared absorption spectrum of this compound was in complete agreement with that of berberine iodide.

The mother liquor was concentrated to dryness. The residue was dissolved in 10 ml. of 50% acetic acid solution, and 3 Gm. of powdered zinc was added. The solution was heated for 10 hr. on a steam bath, at which time the reaction solution was made alkaline with ammonium hydroxide solution and extracted with ether. The ether-exhausted solution gave a negative test with Valser's reagent. The ether phase was dried with anhydrous potassium carbonate, filtered, and then evaporated to dryness to give 0.02 Gm. of colorless crystals. They were recrystallized from methanol to obtain colorless prisms, m.p. 170–171°, which had an infrared spectrum identical with that of an authentic sample of tetrahydroberberine, m.p. 170–171°. There was no mixed melting point depression.

***T. minus* var. *adiantifolium*.**—Milled roots, 5.6 Kg., of *T. minus* var. *adiantifolium* were extracted with methanol in the same manner as for *T. rugosum*. The methanol extract was also treated in the same manner employed for *T. rugosum* to isolate magnoflorine iodide, 0.245 Gm., and berberine iodide, 1.424 Gm. The identification of both iodides was carried out by a comparison of their infrared spectra with those of the authentic samples of magnoflorine iodide and berberine iodide.

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<sup>1</sup> Melting points were determined with a Thomas-Hoover melting point apparatus. Infrared spectra were determined in KBr using a Perkin-Elmer Infracord spectrophotometer, model 237.