

Alkaloids of *Thalictrum* IV. Isolation of Thalicarpine from *T. dasycarpum* and *T. revolutum*

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A simplified procedure is given for the isolation of thalicarpine from roots of *Thalictrum dasycarpum*. The isolation of thalicarpine from roots of *T. revolutum* is reported for the first time.

IN THE process of extracting alkaloids from the roots of *Thalictrum dasycarpum* L. it was found that alkaloidal material was extracted by petroleum ether while defatting the plant material. Furthermore, it was determined that the amount of alkaloid extracted in this manner was increased when the plant was previously dampened with 20% ammonium hydroxide solution. In view of this result, a 4-year-old petroleum ether extract of *T. revolutum* L. roots was examined and found also to have appreciable alkaloidal content.

The alkaloidal material present in the petroleum ether extracts of both of the above *Thalictrum* species consisted almost entirely of thalicarpine, which has been isolated previously from *T. dasycarpum* by Kupchan *et al.* (1) using a longer and more conventional work-up procedure; thalicarpine has not been reported previously as a constituent of *T. revolutum*. The isolation procedure described in this report provides a rapid and convenient method for the isolation of thalicarpine which the authors feel has considerable practical utility.

EXPERIMENTAL

Plant Material.—*T. dasycarpum* L. was collected by Dr. Ervin M. Herrick, Twinsburg, Ohio. *T. revolutum* L. was collected by Dr. Jack L. Beal along the Scioto River in Delaware County, Ohio. Herbarium specimens of both species are on file.

Isolation of Thalicarpine from *T. dasycarpum*.—Ground root, 2010 Gm. of *T. dasycarpum*, was

dampened with 20% ammonium hydroxide solution, then packed in a percolator and extracted with low-boiling petroleum ether (skellysolve F). The concentrated extract deposited a precipitate which was collected by filtration, and then dissolved in 100 ml. of 5% acetic acid solution. The filtered acidic solution was made alkaline with dilute ammonium hydroxide and then extracted with ether. The ethereal extract, after washing with 5% sodium hydroxide solution, was dried with anhydrous potassium carbonate and then concentrated to a small volume on a steam bath. Colorless crystals, 1.5 Gm., separated which were recrystallized as small prisms, m.p. 129–130°, from a mixed solvent of acetone and ether. This material is apparently a low melting polymorph of thalicarpine. Slow crystallization from a large volume of ether afforded the previously reported (1) high-melting form of thalicarpine as colorless needles, m.p. 159–160°, $[\alpha]_D^{25} + 83^\circ$ (CHCl₃). Its identity as thalicarpine was established by infrared comparison and mixed melting point determination with an authentic sample, m.p. 159–160°.²

Crystallization of the high melting polymorph from a concentrated ether solution afforded the low melting form as small prisms, m.p. 129–130°.

Isolation of Thalicarpine from *T. revolutum*.—A 76.5-Gm. 4-year-old sample of an evaporated petroleum ether extract of *T. revolutum* roots was repeatedly extracted with warm 5% acetic acid solution until the extractive gave a negative test with Valser's test solution. The acid solution was partitioned with ether, and the aqueous phase was made alkaline with diluted ammonia solution and then extracted several times with ether. The ether extract was washed with 5% sodium hydroxide solution and then dried over anhydrous potassium carbonate, filtered, and evaporated to a small volume. Crude thalicarpine, 1.82 Gm., separated on standing; crystallization from acetone-ether afforded small prisms, m.p. 129–130°. Identification of this material was made as with samples from *T. dasycarpum*.

REFERENCES

- (1) Kupchan, S. M., Chakravarti, K. K., and Yokoyama, N., *J. Pharm. Sci.*, **52**, 985 (1963).

² The authors thank Dr. S. Morris Kupchan for the authentic sample of thalicarpine.

Received May 10, 1965, from the College of Pharmacy, The Ohio State University, Columbus.

Accepted for publication July 8, 1965.

This investigation was supported by research grant HE-07502 from the National Institutes of Health, U. S. Public Health Service, Bethesda, Md.

Previous paper: Patil, P. N., *et al.*, *J. Pharm. Sci.*, **54**, 1387 (1965).

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¹ 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.