

lating this compound, and because future experiments with substituted dibenzoselenophenes are expected to yield substituted biphenyls.

Experimental

Raney Nickel.—The Raney nickel was prepared according to the directions of Pavlic and Adkins.⁶

Deselenization.—A typical experiment follows: In a 3-necked r.b. flask equipped with a mechanical stirrer and reflux condenser, was placed 100 ml. of benzene, 20 ml. of ethanol, 35 g. of Raney nickel and 1.5 g. of dibenzoselenophene oxide. Refluxed in oil-bath for 5 hr. The unreacted solid was filtered and washed with 40 ml. of benzene (nickel burst into flames on drying). Combined solvents were washed twice with 30 ml. of concentrated H₂SO₄ and twice with water. Benzene was removed, and residue recrystallized from ethanol-water; yield 0.67 g. of biphenyl (72%), m.p. 67–68°. One further recrystallization brought m.p. to 71° (reported 70°); no depression on admixture with authentic sample.

Acknowledgment.—It is a pleasure to acknowledge the assistance of Dr. J. R. McCormick who suggested the method some years ago.

(6) A. A. Pavlic and H. Adkins, *THIS JOURNAL*, **68**, 1471 (1946).

(7) R. L. Shriner and R. C. Fuson, "The Systematic Identification of Organic Compounds," Second Edition, John Wiley and Sons, N. Y., 1940, p. 218.

DEPARTMENTS OF CHEMISTRY
ST. JOHN'S UNIVERSITY
POLYTECHNIC INSTITUTE OF BROOKLYN
BROOKLYN 2, NEW YORK

The Isolation of Bufotenine from *Piptadenia peregrina*

BY VERNER L. STROMBERG

RECEIVED NOVEMBER 18, 1953

The inhalation of a narcotic snuff by the natives of Haiti was a common practice at the time of the discovery of the West Indies. This snuff, called cohoba, was used by the necromancers or priests in their ceremonies and was supposed to enable them to communicate with unseen powers.

The ceremonial use of cohoba was described as early as 1496 by Ramon Pane who was with Columbus on his second voyage, but in later years cohoba was confused with tobacco.

The chemistry of this snuff has never been studied although the leguminous shrub *Piptadenia peregrina* is now known to be its source.¹

In the present work there was isolated from the seeds of this plant a crystalline organic base, m.p. 146–147°, with empirical formula C₁₂H₁₆N₂O in 0.94% yield.

The ultraviolet absorption spectrum in 0.1 *N* hydrochloric acid showed a maximum at 277 mμ, a shoulder with a second maximum at 295 mμ and a minimum at 247 mμ. In 0.1 *N* sodium hydroxide the absorption spectrum shows a shift of the second maximum to 322 mμ. This shift is similar to that observed for the vasoconstrictor 5-hydroxytryptamine (serotonin).²

A methiodide, picrate, oxalate and *m*-nitrobenzoate were prepared. The melting points were in good agreement with the literature values for bufotenine

	Bufotenine m.p., °C.	Piptadenia alkaloid m.p., °C.
Base	147 ³	146–147
Red picrate	178 ³	176–177
Methiodide	210 ³	213–214
Oxalate	84–88 ⁴	82–84
<i>m</i> -Nitrobenzoate	258 ³	255–257

The infrared absorption spectrum of the picrate was identical with that of a picrate of a synthetic sample kindly supplied by Dr. M. E. Speeter of the Upjohn Company.

The seeds of *Piptadenia peregrina* evidently constitute an excellent source of bufotenine. The leaves and branches do not give an alkaloid test with Meyer's or silicotungstic acid reagents. The seed pods give only a slight positive test.

Experimental

Isolation of Bufotenine.—*Piptadenia peregrina* seeds, 891 g., secured from Las Mesas, Puerto Rico,⁵ were ground in a Waring blender and extracted twice with 4 l. of 1% ethanolic tartaric acid solution for 2 hours at 55°. The resulting 8 l. of solution was filtered, concentrated to a volume of 1 l. and diluted with 2.5 l. of water. It was acidified with 200 ml. of 2 *N* hydrochloric acid. The solution was filtered and extracted six times with 200-ml. portions of chloroform. The chloroform solution was discarded. The acid solution was neutralized with solid sodium carbonate. This was divided into two parts and each part was extracted seven times with 200-ml. portions of chloroform. The combined chloroform solutions were extracted with 2 *N* hydrochloric acid. This acid solution was made basic with solid sodium carbonate and the base was re-extracted with chloroform. After drying, the solvent was removed to provide 20.95 g. (2.45%) of mixed organic bases.

A crude alkaloid fraction, 10.11 g., was subjected to chromatographic separation on alumina (Merck Reagent). An ethyl acetate fraction contained 0.12 g. of a brown oil. The bufotenine fraction was eluted with 3:1 ethyl acetate-ethanol to give 7.66 g. of material. Several recrystallizations from ethyl acetate gave 4.09 g. (40% of the alkaloid fraction), m.p. 146–147°. Bufotenine represents 0.94% of the *Piptadenia* seed. The material remaining on the column was eluted with ethanol to give 2.31 g. of residue.

(3) H. Wieland, W. Kanz and H. Mittasch, *Ann.*, **513**, 1 (1934).

(4) T. Wieland and W. Motzel, *ibid.*, **581**, 10 (1953). The oxalate as originally prepared by H. Wieland had a melting point of 96.5° and was a monohydrate. The oxalate prepared here is a half-hydrate (*Anal.* Calcd. for 2C₁₂H₁₆N₂O·2C₂H₂O₄·H₂O: C, 55.44; H, 6.31; N, 9.24. Found: C, 55.58; H, 6.41; N, 9.05) and is apparently the same as the material reported by T. Wieland without analytical data.

(5) Through the Agricultural Research Service, Plant Exploration and Introduction, United States Department of Agriculture

LABORATORY OF CHEMISTRY OF NATURAL PRODUCTS
NATIONAL HEART INSTITUTE
NATIONAL INSTITUTES OF HEALTH
BETHESDA, MARYLAND

The Specific Rotation of Isocolchicine¹

BY ROBERT F. RAFFAUF, EDGAR E. BUMBIER AND GLENN E. ULLYOT

RECEIVED NOVEMBER 13, 1953

During the course of our work on the chemistry of colchicine derivatives we observed that the specific rotation of solutions of isocolchicine changed with time. To our knowledge, this is the first recorded instance of this phenomenon in the col-

(1) W. E. Safford, *J. Wash. Acad. Sci.*, **6**, 15, 547 (1916).

(2) V. Erspamer and B. Asero, *Nature*, **169**, 800 (1952).

(1) This investigation was supported (in part) by a grant from the National Cancer Institute of the National Institutes of Health, Department of Health, Education and Welfare.