

THE OCCURRENCE OF TRYPTAMINE AND N-METHYLTRYPTAMINE IN *MIMOSA SOMNIANS*

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While a phytochemical screen of Panamanian plants was being conducted, *Mimosa somnians*, Humb. and Bonpl. ex Willd. (Leguminosae) was found to give a positive test with several alkaloid detection reagents. Although the plant was collected in Panama, it is found in many areas of Central and South America (1). Along with certain other *Mimosa* species, it bears the common name *dormidera* in Central America, indicating that it too is a "sensitive plant" responding visibly to contact or other stimuli. No previous phytochemical investigations of this plant have been reported.

Because of the occurrence of tryptamine derivatives in other *Mimosa* species (2), most notably *Mimosa hostilis* Benth. (3), the plant was tested for indole derivatives by use of tlc and *p*-dimethylaminobenzaldehyde reagent (PDAB). Two constituents were detected which gave a purple chromophore with PDAB and chromatographically corresponded to tryptamine and N-methyltryptamine in four tlc systems.

To confirm the presence of these compounds, the residue from the alkaloid extract was treated with heptafluorobutyrylimidazole (HFBI), and the derivatized mixture was analyzed by gas chromatography-mass spectroscopy (gc-ms) for the presence of heptafluorobutyryl (HFB) derivatives of indolealkylamines. HFBI acylates primary and secondary amines, as well as hydroxyl groups and the indole

nitrogen of indole amines. Single well-defined derivatives are obtained which are separable with gas chromatography (4, 5). The HFB derivatives yield mass spectra containing a low intensity, but detectable, molecular ion as well as other peaks useful in distinguishing between these compounds (4). Direct gc-ms analysis of indolealkylamines has been reported (6-8), but the above method proved to be more sensitive and convenient. Analysis of the HFB derivatives obtained from the alkaloid fraction of *M. somnians* showed the presence of tryptamine (0.026%, dry weight of whole plant) and N-methyltryptamine (0.029%). The retention times and mass spectra were identical to those of derivatized standards.

Since N-oxides of tryptamine derivatives have been reported from the Leguminosae (2), *M. somnians* was screened for these compounds by reducing an alkaloid extract with $\text{TiCl}_3\text{-H}_2\text{SO}_4$ (9) and comparing it chromatographically with an untreated extract. There was no indication that indolealkylamine N-oxides were present in the plant.

Tryptamine and N-methyltryptamine have not been previously isolated from the genus *Mimosa* (2). *M. verrucosa* and *M. hostilis* both contain *N,N*-dimethyltryptamine (2). The latter species has been used by several tribes in eastern Brazil to prepare a potent hallucinogenic drink (10).

EXPERIMENTAL

PLANT MATERIAL.—*Mimosa somnians* was collected near Portobello, Republic of Panama, during the summer of 1976. The plant was identified by Prof. Mireya Correa, Curator of the Herbarium of the University of Panama, where a sample of the collection is deposited. The plant material was air dried (<50°) before storage in airtight containers.

EXTRACTION AND TLC ANALYSIS.—The whole plant, including mature seeds, was ground to a 20-mesh powder in a Wiley mill. Ten grams of the ground material was exhaustively extracted with methanol by percolation. The methanol was removed on a rotary evaporator and the residue extracted with 20 ml of 10% H₂SO₄ by shaking vigorously for 15 min. The filtered extract was made basic (pH 10) with 2 N NaOH and extracted with four 30 ml portions of ether. The water layer was then saturated with NaCl and re-extracted with another portion of ether. The ether extracts were combined and taken to dryness in a rotary evaporator.

The alkaloid extract was screened for the presence of tryptamine derivatives by use of the following silica gel 60F-254 (E.M. Reagents) tlc systems. A: 1-propanol-5% ammonium hydroxide solution (5:2); B: benzene-methanol-5% ammonium hydroxide solution (10:15:2); C: ethyl acetate-methanol-58% ammonium hydroxide solution (80:15:5); D: 1-butanol-glacial acetic acid-water (2:1:1). The developed plates were sprayed in PDAB reagent (2% *p*-dimethylaminobenzaldehyde in conc. HCl-ethanol (1:1) and heated with a heat gun to develop the chromophores. Two compounds were detected which corresponded to tryptamine (R_f: A: 0.44, B: 0.27, C: 0.32, D: 0.57) and *N*-methyltryptamine (0.40, 0.20, 0.29, 0.52). The following compounds were not detected: *N,N*-dimethyltryptamine (A: 0.53, B: 0.46, C: 0.58, D: 0.47), 5-methoxydimethyltryptamine (0.52, 0.44, 0.55, 0.47), 5-methoxytryptamine (0.44, 0.25, 0.30, 0.57), serotonin (0.38, 0.18, 0.18, 0.55) and bufotenine (0.47, 0.36, 0.40, 0.46).

GC-MS ANALYSIS OF EXTRACT.—HFB derivatives were prepared by a modification of the method developed by Vessman, *et al.* (4). Heptafluorobutyrylimidazole (Pierce Chemical Company) (0.2 ml) was added to 1-2 mg of the indole amine free base and the solution heated at 85° for one hour. After cooling, 2 ml of *n*-hexane was added to the reaction vial and the mixture was shaken for two min. Five to 10 μ l of the hexane solution was then assayed by gas chromatography. The column (4 mm x 3 ft glass) was filled with chromosorb WHP coated with 3.8% UC W982. Gas flows were as follows: helium 60 ml/min., air 300 ml/min. and hydrogen 40 ml/min. The injector and flame ionization detector were at 300°,

while the column was temperature programmed from 120° to 200° at 4°/min. The retention times for the HFB derivatives prepared were found to be: *N,N*-dimethyltryptamine (10.50 min.), bufotenine (14.50), tryptamine (14.75), *N*-methyltryptamine (16.25) and serotonin (19.25). Retention times for SE-30 and OV-16 columns have also been reported (4, 5).

The alkaloid fraction obtained from 10 g of *M. somnians* was derivatized by the same procedure and analyzed by gc-ms. Peaks corresponding to HFB-tryptamine and HFB-*N*-methyltryptamine were detected.

The mass spectrum of HFB-tryptamine showed that the derivative contained two HFB groups and had a M⁺ at *m/e* 552 (1%). Major ions were detected at *m/e* 339 (50%), 326 (50%), 169 (40%), 142 (40%), 129 (100%), 115 (30%), 102 (35%) and 69 (85%). HFB-*N*-methyltryptamine also contained two HFB groups and exhibited the following mass spectrum: *m/e* 566 (1%, M⁺), 340 (40%), 326 (40%), 240 (45%), 169 (60%), 142 (45%), 129 (100%), 115 (55%), 102 (60%), 69 (95%). The HFB derivative of *N,N*-dimethyltryptamine showed the presence of one HFB group and had the following mass spectrum: *m/e* 382 (1%, M⁺), 169 (10%), 143 (10%), 129 (25%), 115 (20%), 102 (15%), 69 (35%), and 58 (100%). The mass spectra of HFB-serotonin and 5-methoxydimethyltryptamine have been reported by Vessman *et al.* (4). The mass spectra of the HFB derivatives detected in the derivatized *M. somnians* alkaloid extract were identical to those obtained from HFB-tryptamine and HFB-*N*-methyltryptamine standards.

QUANTITATIVE ANALYSIS.—Standard curves for peak height versus concentration were prepared for the HFB derivatives of tryptamine, *N*-methyltryptamine and *N,N*-dimethyltryptamine. This relationship was linear for all three derivatives over the concentration range studied (0.5 to 5 mg derivatized). All three curves were essentially identical. The efficiency of the extraction and derivatization procedure was determined by the addition of 2 mg of *N,N*-dimethyltryptamine to 10 g of ground *M. somnians* prior to extraction. The efficiency was found to be 25.5%. The concentration of tryptamine and *N*-methyltryptamine in the dried whole plant material was found to be 0.026% and 0.029% respectively.

ACKNOWLEDGMENTS

The authors wish to thank Professor Mireya Correa for her assistance in identifying the plant material, and to Mr. Rudolfo Mendoza for collecting it. This investigation was supported in part by the Regional Program of Scientific and Technological Development of the Organization of American States.

Received 2 August 1978.

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BOOK REVIEW

Separation and Purification, 3rd Edition, edited by E. S. Perry and A. Weissberger, Eastman Kodak Company, Rochester, N.Y. Wiley-Interscience, John Wiley and Sons, Inc., 605 Third Ave., New York, N.Y. 1978. xi+438 pp. 16 x 23.5 cm. \$30.00.

Techniques in Chemistry, edited by Arnold Weissberger, is a standard series which undoubtedly has a place in most technical libraries. Volume 12 of this series, *Separation and Purification*, edited by E. S. Perry and Arnold Weissberg, is now in its third edition. There are seven chapters in this book, only a few of which in the reviewer's opinion will be of basic interest to the readers and contributors of the Journal of Natural Products. Chapter I, purity; Concept and Reality, is a short discussion and is interesting because of the fact that many of the purity concepts are not generally known. Chapter II, Solvent Selection for Separation Processes, gives some useful information on the physical properties of a variety of solvents and solvent pairs which can be of some general use in natural products. The types of problems discussed, for the most part, are those met in industry and are less likely to be encountered during the isolation of a complex mixture of natural products. Chapter III is the longest chapter in the book, comprising about 120 pages, and deals with liquid-liquid extraction largely from the viewpoint of chemical engineers. The author of the chapter, E. C. Scheibel, has been an expert in the area for many years. There is an exhaustive theoretical and practical discussion of the Craig countercurrent distribution, a methodology initiated and practiced by the late Lyman Craig. The technique, as Scheibel states in his introduction, was useful some years ago but has been largely superseded by high pressure liquid chromatography and probably is now only of historical interest. Those readers with access to the second edition of *Separation and Purification* in this series can probably obtain all the necessary information from the older edition. Chapter IV is a useful chapter on ion exchange chromatography. The properties and uses of many ion exchangers are discussed in detail, and there is a particularly useful discussion dealing with the uses of ion exchange resins in liquid chromatography and high pressure liquid chromatography. Chapter V presents an excellent and informative account of affinity chromatography. The last two chapters, VI and VII, on centrifugation and filter media, respectively, are treated from the viewpoint of a chemical engineer and will probably be of limited interest to the readers of this journal.

Although *Separation and Purification*, along with the entire Weissberger series, belongs in every major technical library, the reviewer cannot recommend its purchase by the individual non-specialist because of the presence of so much old and highly specialized material.

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