No. dans le répertoire	No. dans le répertoire	No. dans le répertoire	No. dans le répertoire
tchat	16	wahédo	107
tulomé hinna	36	wahihurra	108
		walaïnabi	22
unda kéna	55		
undal hé	69	yaha	109
unga	45	yatheni	06
urré dawa	39	yayabto	48

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Journal of Ethnopharmacology, 14 (1985) 315—318
Elsevier Scientific Publishers Ireland Ltd.

Short Communication

PHYTOCHEMICAL INVESTIGATION OF *TABERNAEMONTANA CRASSA**

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(Accepted August 27, 1985)

Summary

From the stembark of *Tabernaemontana crassa* the alkaloid ibogaine was isolated as the major component. Ibogaine showed activity against the gram-positive *Bacillus subtilis*. Conopharyngine was identified as one of the minor compounds.

Introduction

Tabernaemontana crassa Benth. (Apocynaceae) occurs widely in tropical Africa from Sierra Leone to Uganda in the north and to Angola and Tanzania in the south. It is a bush or small tree which grows particularly in sparse forest or in secondary vegetation from the coast up to an altitude of 2000 m. The species has many synonyms, the most important of which are *T. durissima* Stapf, *T. jollyana* Pierre ex Stapf, *Conopharyngia crassa* (Benth.) Stapf, *C. durissima* Stapf and *Gabunia odoratissima* Stapf (van Beek et al., 1984a). Various parts of this species are widely used in traditional medicine for several different ailments (van Beek et al., 1984a). In a recent antimicrobial screening of 19 *Tabernaemontana* species (van Beek et al., 1984b) the ethanolic stembark extract of *T. crassa* showed significant activity against gram-positive bacteria and weak activity against gram-negative bacteria. This fact led to the present study, the results of which are described below. Previous phytochemical investigations of the stembark

*Part 12 in the series: Pharmacognostical studies of *Tabernaemontana* species. For part 11 see van Beek et al. (1985).

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have yielded the alkaloids *O*-acetyl-polyneuridine, anhydro-vobasindiol, akuammiline, conopharyngine, 19-hydroxy-conopharyngine, 3-oxo-conopharyngine, conopharyngine-hydroxy-indolenine, coronaridine, 3-oxo-coronaridine, 5-oxo-coronaridine, 3-oxo-coronaridine-hydroxy-indolenine, heyneanine, 3-oxo-heyneanine, isovoacangine, voacristine and crassanine (van Beek et al., 1984a).

Materials and methods

The plant material was collected in Cameroon, 3 km from Rocher du Loup by H. Beentje in December 1980. The plant material was identified by Dr. A.J.M. Leeuwenberg and a voucher specimen (H. Beentje 1548) has been deposited at the Laboratory for Plant Systematics, Wageningen, The Netherlands. The ^{1}H-NMR spectrum was recorded at 300 MHz in $CDCl_3$ (Bruker WM 300). The MS was recorded at 70 eV using a direct inlet system (AEI MS 20). The following TLC systems were used in combination with silica gel plates: (A) cyclohexane/$CHCl_3$/Et_2NH (6:3:1); (B) EtOAc/isoPrOH (9:1); (C) toluene/EtOH satd. with NH_3 (19:1) (prior to development the plates were left standing in an atmosphere of NH_3 for 20 min). (D) $CHCl_3$/MeOH (9:1); (E) EtOAc/isoPrOH/26% NH_4OH (45:4:1). After development the TLC plates were sprayed with iodoplatinate reagent, 1% $Ce(SO_4)_2$ in 10% H_2SO_4 or with 0.2 M $FeCl_3$ in 35% $HClO_4$ followed by heating with hot air.

Isolation procedure: The ground stem bark (800 g) was extracted for 15 h with 96% EtOH in a Soxhlet apparatus working under a pressure of 0.2 atm. After cooling at −16°C a precipitate was formed, which was collected (steroids and triterpenes). The remaining solution was evaporated in vacuo to dryness (10 g). Five grams of the extract was partitioned between $CHCl_3$ and 2% HOAc. The aqueous layer was collected and adjusted to pH 10 with NH_4OH and extracted twice with $CHCl_3$. The $CHCl_3$ layer was collected, dried (Na_2SO_4) and evaporated in vacuo (yield 0.97 g tertiary alkaloids, 0.24%). Part (100 mg) of this fraction was separated by means of preparative TLC with systems A and E. The major amorphous product (12 mg) was identified as ibogaine (1) and showed the following physical data: R_f-values and chromogenic reactions (see van Beek et al., 1984c); UV λ_{max} (MeOH) 227 and 297 nm; MS (175°C) *m/z* (rel. int.) 310 (M^+, 70), 309 (27), 295 (12), 225 (39), 149 (46), 136 (100), 135 (78), 122 (48); ^{1}H-NMR: 7.52 (bs. NH), 7.14 (d, J = 8.8 Hz, H12), 6.93 (d, J = 2.5 Hz, H9), 6.76 (dd, J = 8.8 and 2.5 Hz, H11), 3.85 (s, OMe), 3.42—3.38 (m, H5a and H5b), 3.18—3.10 (m, H6a), 3.07 (ddd, J = 9.5, 2.1 and 2.1 Hz, H3a), 2.97 (ddd, J = 9.5, 3 and 3 Hz, H3b), 2.90 (ddd, J = 11.6, 4.2 and 1.5 Hz, H16), 2.85 (bs, H21), 2.65—2.57 (m, H6b), 2.04 (dddd, J = 13.0, 11.6, 2.6 and 2.6 Hz, H17a), 1.84 (bs, H14), 1.81 (m, H15a), 1.65 (m, H17b), 1.63—1.42 (m, H19a, H19b and H20), 1.21 (m, H15b), 0.89 (J = 7.2 Hz, H18).

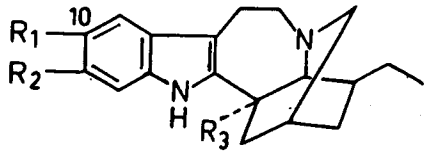

Fig. 1. **1**: R_1 = OMe; R_2 = R_3 = H. **2**: R_1 = R_2 = OMe; R_3 = CO_2Me.

Antimicrobially inactive minor alkaloid: This alkaloid was identified by means of coTLC as conopharyngine (**2**). R_f-values and chromogenic reactions see van Beek et al. (1984c).

Antimicrobially active minor alkaloid: R_f-value in system A 0.43, C 0.58, D 0.09. Colour with the $FeCl_3$ spray reagent: blue before and after heating.

Results and discussion

A preliminary TLC investigation of the crude tertiary alkaloid fraction showed that one major alkaloid, two minor alkaloids and many trace alkaloids were present. Using the agar diffusion method (Verpoorte et al., 1982) as a bioassay it was found that the major alkaloid and one of the minor alkaloids showed activity against *Bacillus subtilis*. The activity of the major alkaloid was in this bioassay comparable with the activity of 3-*R*/*S*-hydroxy-isovoacangine which has a MIC value of 50 μg/ml against *B. subtilis*. The major alkaloid was isolated by means of preparative TLC and identified by means of its spectral data (UV, MS and ^{1}H-NMR) and coTLC as ibogaine (**1**). The antimicrobially active minor alkaloid was present in too low a concentration to allow its isolation. Its R_f-values and chromogenic reactions suggested that it was an alkaloid of the dimeric voacamine type lacking the carbomethoxy group at the 16-position of the iboga half (van Beek et al., 1984c). The other minor alkaloid was identified by means of coTLC and its chromogenic reactions as conopharyngine (**2**). We do not exclude the possibility that some of the trace alkaloids may also contribute to the overall antimicrobial activity of this species. This could not be checked because of the low sensitivity of the biogram method. Ibogaine (**1**) has not previously been isolated from the stembark or any other part from this species.

Acknowledgements

We wish to thank Dr. H. Beentje for collecting the plant material, Dr. A.J.M. Leeuwenberg for the identification and the "Fonds Harald Quintus Bosz" and the "Van Leersum Fonds" for their financial help in collecting the plant material.

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Book Review

Alkaloids: Chemical and Biological Perspectives, Vol. 2, S.W. Pelletier (Ed.) John Wiley & Sons, New York, 1984, xi + 490 pp.

A worthy continuation of the numerous contributions to our knowledge of alkaloids made by Dr. S. William Pelletier of the Institute of Natural Products Research and the University of Georgia, this volume goes a step ahead and gathers together unpublished as well as summaries of hard-to-locate published material not only of purely chemical and biological interest but data indicative of the editor's breadth of interest. It succeeds in convincing the specialist that this field of research must be viewed from the interdisciplinary viewpoint.

The physiological effects and social importance of some of the 5000 known alkaloids are broadly discussed. Yet with this broad approach, the book succeeds admirably in offering a vast amount of highly technical chemical and biological information, a good portion of it new.

The volume treats five aspects of alkaloid study, the work of 12 authors — all highly qualified specialists. Chapter 1 reviews the applications of single-cell X-ray diffraction in alkaloid chemistry. Chapter 2 surveys the imidazole alkaloids. Chapter 3 presents a comprehensive review of quinolizidine alkaloids of the Leguminosae. In Chapter 4, a broad summary of the chemistry and pharmacology of maytansinoid alkaloids is offered. Chapter 5 concerns ^{13}C and proton NMR shift assignments and physical contents of C_{19}-diterpenoid alkaloids.

The two indices, one for subjects and one for organisms, materially increase the availability of the vast amount of material presented.

This is a book which the ethnopharmacologically oriented phytochemist, pharmaceutical scientist or botanist should have at hand. It includes much information so efficiently organized that utility is greatly enhanced and modern concepts are fully summarized.

Richard Evans Schultes