LIGNAN AND AMIDES FROM PIPER SYLVATICUM

J. BANERJI and K. P. DHARA

Department of Pure Chemistry, University College of Science, Calcutta-9, India

(Received 16 February 1974)

Key Word Index—Piper sylvaticum. Piperaceae, Sesamin, Piperine, Piperlongumine.

The roots of Piper sylvaticum Roxb, are widely used as an effective antidote to snake poison in the indigenous system of Indian medicine. Earlier investigations 1-4 on the seeds of this plant resulted in the isolation of a new alkamide sylvatine in addition to 4'-7dimethoxy-5-hydroxy flavone and the extremely unstable N-isobutyl-deca-trans-2-trans-4dienamide. The results prompted us to undertake the detailed chemical investigation of the roots of this plant.

The dried roots (2.5 kg) were extracted with petrol (60–80°) at reflux temperature for 72 hr. The concentrate afforded a cream-coloured solid which was found to be a mixture of three major components from TLC over silica-gel impregnated with 5% AgNO₃ [Solvent system- C_6H_6 : EtOAc (4:1)].

The solid upon chromatography over silica gel (60-100 mesh, Gouri Chemical) afforded in the petrol- C_6H_6 (2:1) eluate the lignan sesamin, $C_{20}H_{18}O_6$ (M⁺ 354), 118–119° m.p., R_f 0.7, in the petrol- C_6H_6 (1:1) fraction the alkamide piperine, $C_{17}H_{19}NO_3$ (M⁺ 285), 127– 129° m.p., R_f 0·32, and in the C_6H_6 eluate another alkamide piperlongumine (Ia⁵ and Ib⁶⁾, $C_{17}H_{19}NO_5$ (M⁺ 317), 121–123° m.p., R_f 0.4. This is the first report of the occurrence of these compounds in this *Piper* species. It may be mentioned here that piperine acts as an analeptic and exerts a stimulant action on the central nervous system (especially on the respiratory centre) in mice and dogs.

All the isolated compounds were identified by direct comparison (m.m.p., IR, co-TLC) with the respective authentic samples isolated earlier in our laboratory. The MS of the isolated compounds were identical with those of authentic samples.

¹ BANERJI, A. and GHOSH, P. C. (1973) Tetrahedron, 29, 977.

² Banerji, A. and Ghosh, P. C. (1972) Abstracts, Annual Chemists' Convention, Allahabad, p. 81.

³ BANERJI, A., GHOSH, P. C. and REJ, R. N. (1974) Experientia 30, 3.

⁴ Banerji, A., Rej, R. N. and Ghosh, P. C. (1973) Abstracts, Annual Chemists' Convention, Calcutta, p. 151.

CHATTERJEE, A. and DUTTA, C. P. (1967) Tetrahedron, 23, 1769.
JOSHI, B. S., KAMAT, V. N. and SAKSENA, A. K. (1968) Tetrahedron Letters 2395.

Voucher specimen No. P.S. (r) of the root has been preserved in our laboratory. This was collected by Home-O-Flora, Calcutta, and identified by botanist Dr. P. C. Dutta, Department of Botany, Calcutta University, Calcutta.

Acknowledgements—Sincere thanks are recorded to Professor (Mrs.) A. Chatterjee. University College of Science. Calcutta, for providing laboratory facilities, and to CCRIMH (India) for allowing one of us (J.B.) to work on this plant.

Phytochemistry, 1974, Vol. 13, p. 2328. Pergamon Press. Printed in England.

PEPTIDE ALKALOIDS FROM ZIZYPHUS MUCRONATA*

RUDOLF TSCHESCHE, MOHAMED ELGAMAL and GERT ECKHARDT

Institut für Organische Chemie und Biochemie der Universtät, 53-Bonn, Germany

and

MICHAEL VON RADLOFF

Chemistry Department, Ahmadu Bello University, Zaria, Nigeria

(Received 29 March 1974)

Key Word Index—Zizyphus mucronata Willd.; Rhamnaceae; cyclic peptide alkaloids; mucronines -A, to -H; abyssenines -A, to -C; isoquinoline alkaloid; (-)N-methylcoclaurine.

Plant. The bark and the leaves of Zizyphus mucronata, collected in Nigeria, January 1971.

Previous work. The eight mucronines, -A to $-H^{1,2}$ have been isolated from the bark of Z. mucronata, collected in Mali. The three abyssenines, -A, -B and $-C^2$ have been found in the bark of Z. abyssinica, collected in Nigeria.

Present work. Extraction and identification of the alkaloids from the bark: the crude alkaloids (0.09%) were obtained by extraction of the powdered bark in the usual manner, and separated to pure components by means of column chromatography and preparative TLC. The compounds were identified by MS, NMR, IR, m.m.p. and co-TLC. In addition to the alkaloids which have been isolated before: abyssenines -A, -B and -C have been identified. Extraction and identification of the alkaloids from the leaves: from the polar fraction of the crude alkaloids (0.06%) mucronines -G, -H and abyssenine -C have been isolated and identified. The polar fraction contains the isoquinoline alkaloid (-)N-methylcoclaurine⁴ which has been identified by physical and chemical methods.

Acknowledgements—We thank the Deutsche Forschungsgemeinschaft for the NMR spectrometer and financial support and the Stiftung Volkswagenwerk for the purchase of the mass spectrometer.

⁴ ARNDT, R. R. (1963) J. Chem. Soc. 2547.

^{*} Part XXIV of the series "Alkaloids from Rhamnaceae". Part XXIII R. TSCHESCHE and E. Ammermann, Chem. Ber. In press.

¹ Tschesche, R., David, S. T., Uhlendorf, J. and Fehlhaber, H. W. (1972) Chem. Ber. 105, 3106; Ann. Chem. 759, 195.

² TSCHESCHE, R., DAVID, S. T., ZERBES, R., ECKHARDT, G. and KAUSSMANN, E. U. (1974) Ann. Chem. In press.

³ Tschesche, R., Welters, R. and Fehlhaber, H. W. (1967) Chem. Ber. 100, 323.