Short Reports

- Hamet, R. and Goutarel, R. (1951) C.R. Acad. Sci. Paris 233, 431.
- Janot, M. M., Goutarel, R. and Prelog, V. (1951) Helv. Chim. Acta 34, 1207.
- Wenkert, E., Chang, C. J., Chawla, H. P. S., Cochran, D. W., Hagaman, E. W., King, J. C. and Orito, K. (1976) J. Am. Chem. Soc. 98, 3645.
- 5. Wenkert, E., Bindra, J. S., Chang, C.-J., Cochran, D. W. and Schell, F. M. (1974) Acc. Chem. Res. 7, 46.
- 6. Lounasmaa, M. and Kan, S. K. (1980) Tetrahedron 36, 1607.
- Main, P., Hull, S. E., Lessinger, L., Germain, G., Declercq, J. P. and Woolfson, M. M. (1970). MULTAN 78. A System of Computer Programmes for the Automatic Solution of the Crystal Structures from X-ray Diffraction Data. Universities of York, U.K. and Luvain, Belgium.
- 8. Sheldrick, G. M., SHELX 76 (1976) A Programme for Crystal Structure Determination, University of Cambridge, U.K.
- 9. Cremer, D. and Pople, J. A. (1975) J. Am. Chem. Soc. 97, 1354.
- 10. Hendrickson, J. B. (1967) J. Am. Chem. Soc. 89, 7036.

Phytochemistry, Vol. 30, No. 7, pp. 2449-2450, 1991 Printed in Great Britain. 0031-9422/91 \$3.00 + 0.00 © 1991 Pergamon Press plc

A PYRIDINE ALKALOID FROM CEROPEGIA JUNCEA

N. A. Adibatti, P. Thirugnanasambantham,* C. Kulothungan, S. Viswanathan, Lalitha Kameswaran,† K. Balakrishna‡ and E. Sukumar‡

Medicinal Chemistry Research Centre, Institute of Pharmacology, Madras Medical College, Madras 600 003, India; ‡ Capt. Srinivasa Murti Drug Research Institute for Ayurveda (CCRAS), Arumbakkam, Madras 600 106, India

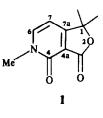
(Received in revised form 3 January 1991)

Key Word Index-Ceropegia juncea; Asclepiadaceae; pyridine-alkaloid; cerpegin.

Abstract—Cerpegin, a new pyridine alkaloid, has been isolated from *Ceropegia juncea* together with lupeol. Based on spectroscopic methods the structure of this alkaloid has been elucidated as 3,4-dioxo-1,1,5-trimethyl-1,3,4,5-tetrahydrofuro-[3,4-c]-pyridine.

INTRODUCTION

Ceropegia juncea Roxb. is reported to be the source of 'Soma', a plant drug of the Ayurvedic system of medicine with a wide variety of uses [1]. The alcoholic extract of the plant was found to possess antipyretic, analgesic, local anaesthetic, antiulcer, mast-cell stabilising, hepato-protective, tranquilising and hypotensive activities in experimental animals. The total alkaloidal fraction exhibited promising tranquilising, hypotensive and local anaesthetic activities and was devoid of side effects as borne out by sub-acute toxicity studies [2]. We deal with the isolation and structural elucidation of a new pyridone derivative, cerpegin, together with the triterpene lupeol from this hitherto phytochemically unexamined plant. Pyridones are relatively rare in nature.



RESULTS AND DISCUSSION

The shade-dried powdered whole plant was successively extracted with *n*-hexane and 90% ethanol. The *n*hexane extract was saponified with 5% alcoholic KOH and the unsaponified portion on column chromatography over silica gel gave lupeol and three minor compounds. The latter could not be characterized due to poor yield.

The 90% ethanol extract was treated with 2% H₂SO₄ and partitioned into basic and non-basic fractions. The chloroform solubles of the basic fraction on column

^{*}Author to whom correspondence should be addressed

[†] Present address: Vice-Chancellor, Dr MGR Medical University, Madras 600 007, India.

chromatography over basic alumina afforded a new alkaloid, designated as cerpegin (1). Compound 1 had IR bands at 1675 and 1750 (cm⁻¹) which can be assigned respectively to the carbonyls of α -pyridone [3] and α,β -unsaturated- γ -lactone systems. The UV spectrum exhibited absorption maxima at 207, 210, 216, 234 and 316 nm showing extended conjugation.

The ¹H NMR spectrum of 1 indicated the presence of a NMe group (δ 3.50), two tertiary methyls attached to a carbon carrying oxygen (δ 1.54) and two protons of the pyridone ring (δ 6.10 and 7.43, J = 5 Hz). The ¹³C NMR spectrum also supported the presence of the amide and γ -lactone functions (δ 166.24 and 171.92). Based on the above data, the structure of 1 has been elucidated as 3,4-dioxo-1,1,5-trimethyl-1,3,4,5-tetrahydrofuro-[3,4-c]-pyridine.

EXPERIMENTAL

Mp: uncorr. IR: KBr discs. UV: 90% EtOH. ¹HNMR (90 MHz) and ¹³CNMR (100 MHz): CDCl₃ and DMSO- d_6 , TMS as int. standard.

The plant material was collected in the Tirunelveli District, Tamil Nadu, India. An authenticated voucher specimen was deposited in the Pharmacognosy Department of Madras Medical college.

Shade-dried and coarsely powdered whole plant (ca 1 kg) was exhaustively extracted with *n*-hexane and 90% EtOH by a cold percolation method (72 hr). The *n*-hexane extract was saponified with 5% alc. KOH and the unsaponified portion was extracted with Et₂O. The Et₂O extract on CC over silica gel (100-200 mesh) yielded lupeol (250 mg), the identity of which was confirmed by the analysis of its spectroscopic data (IR, ¹H NMR and MS) as well as by comparison with an authentic sample (mmp and co TLC).

Isolation of cerpegin. The 90% EtOH extract was concd in vacuo to a syrupy mass and treated with 2% H_2SO_4 . The aq. acidic extract was cooled and basified with aq. NH₃ to pH 10 and extracted with $CHCl_3$ (3 × 200 ml). The $CHCl_3$ phases were bulked, dried over Na₂SO₄, concd and chromatographed over a column of basic alumina. Elution with CHCl₃-MeOH (9:1) afforded cerpegin, (500 mg), $C_{10}H_{11}NO_3$ ([M]⁺ 193), mp 268-270° (CHCl₃-MeOH) (Found: C, 61.92; H, 5.92; N, 7 59. $C_{10}H_{11}NO_3$ requires: C, 62.18; H, 5.70; N, 7.25. IR ν_{max}^{KBr} cm⁻¹: 1750, 1675, 1600, 1550, 1375, 1300, 1150, 1080, 1040 (w), 950, 900, 880, 810, 680; UV $\lambda_{max}^{90\%}$ EtoII nm: 207, 210, 216, 234, 316; ¹H NMR (90 MHz, CDCl₃): δ 1.54 (6H, s, 2 × Me), 3.50 (3H, s, = NMe), 6.10 and 7.43 (1H each, d, J = 5 Hz, H-7 and H-6); ¹³C NMR (100 MHz, DMSO-d₆): δ 82.10 (C-1), 171.92 (C-3), 109.74 (C-4a), 166.24 (C-4), 147.66 (C-6), 98.24 (C-7), 157.00 (C-7a), 25.36 (2 × Me), 36.75 (N-Me); MS m/z (rel. int.): 193 [M]⁺ $(79.12), 178 [M - Me]^+ (100), 150 [M - Me - CO]^+ (8.45), 136$ (9.80), 108 (12.86), 79 (11.56), 42 (61.46), 28 (29.39), 18 (13.77).

Acknowledgements—The authors thank Mr V. Chelladurai (T. N. Medicinal Plants Corporation, Tirunelveli) for the collection and identification of the plant material and Dr R. Balasubramanian (Department of Organic Chemistry, University of Madras) for elemental analysis.

REFERENCES

- Usman Ali, S. and Narayanaswamy, V. (1970) J. Res. Indian Med. 5, 10.
- 2. Adibatti, N. A. (1985) Ph.D. Thesis, University of Madras.
- Nakanishi, K. (1962) Infrared Absorption Spectroscopy, p. 207. Holden-Day, San Francisco.