

of the seeds of the same plant upon careful chromatographic separation over Si gel furnished three crystalline compounds, sylvatin, sesamin and diaeudesmin. *Sylvatin*, $C_{24}H_{33}NO_3$ (M^+ 383), mp 114–115° $[\alpha]_D \pm 0$ ($CHCl_3$) [4] exhibited IR and NMR spectra suggestive of its identity which was confirmed by direct comparison (mmp, Co-TLC and mixed IR) with an authentic sample. *Sesamin*, $C_{20}H_{18}O_6$ (M^+ 354), mp 122–124°, $[\alpha]_D + 78.4$ ($CHCl_3$) was identical with an authentic sample (mmp, Co-TLC, mixed IR). *Diaeudesmin*, $C_{22}H_{26}O_6$ (M^+ 386), mp 153°, $[\alpha]_D + 325^\circ$ ($CHCl_3$); λ_{max}^{EtOH} 230 and 280 nm (log ϵ , 4.49, 3.54) exhibited characteristic IR bands at 1600, 1588, 1512, 1235, 1140 and 1085 cm^{-1} for aromatic and cyclic diethers, and the 100 MHz. NMR data [$(CDCl_3)$]: δ 3.04–3.24 m (2H, C-1 and C-5 bridgehead protons), δ 4.90, *d*, *J* 5 (2H, C-2 and C-6 methine protons), δ 3.42–3.60 m, and δ 3.66, 3.78, *dd*, *J* 3 (4H, C-4 and C-8 methylene protons), δ 3.87 and 3.90, *s*, (3H each, two meth-

oxyls), δ 6.90–6.98, *d*, *J* 8 (3H, aromatic protons)] showed it to be symmetrical. A two protons doublet around δ 4.90 together with the appearance of the methylene proton signals below δ 4.00 clearly revealed the presence of two diaxial aryl groups in the compound [5].

Acknowledgements—The authors thank Professor (Mrs.) A. Chatterjee, University of Calcutta, Calcutta, India for sylvatin and sesamin, Dr. N. Dutta, Indian Institute of Experimental Medicine, Calcutta, India for the measurement of optical rotation and Profesor P. Sengupta, University of Kalyani for encouragement. The investigation was supported by a grant-in-aid from Indian Council of Agricultural Research and University Grants Commission, New Delhi, India.

REFERENCES

1. Chatterjee, A. and Dutta, C. P. (1967) *Tetrahedron* **23**, 1769.
2. Atal, C. K. and Dhar, K. L. (1967) *Indian J. Chem.* **5**, 588.
3. Joshi, B. S., Kamat, V. K. and Saksena, A. K. (1968), *Tetrahedron Letters* 2395.
4. Banerjee, A. and Ghosh, P. C. (1973) *Tetrahedron* **29**, 977.
5. Birch, A. J., Macdonald, P. L. and Pelter, A. (1967) *J. Chem. Soc. (C)*, 1968.

* Studies on the genus Piper—III.

Phytochemistry, 1975, Vol. 14, pp. 2091–2092. Pergamon Press. Printed in England.

ACYLATED BETACYANINS OF *PORTULACA OLERACEA*

FILIPPO IMPERATO

Istituto di Chimica Organica dell'Università di Catania, Catania, Italia

(Received 28 February 1975)

Key Word Index—*Portulaca oleracea*; Portulacaceae; betacyanins; ferulic acid derivatives; betanidin and isobetanidin 5-cellobiosides.

Two red-violet pigments (Oleracin I and II) have been found in *Portulaca oleracea* L. [1]. In the present study, these pigments have been reinvestigated in more detail and found to be acylated betacyanins [2–7]. Alkaline hydrolysis of the total betacyanin fraction gave ferulic acid and two new

pigments which were proved to be 5-*O*- β -cellobiosides of betanidin and isobetanidin.

The total betacyanin fraction was isolated by chromatography on Dowex 50 W-X2 resin. Column chromatography on polyamide yielded two red-violet bands (Oleracin I and II) which

had identical spectral and electrophoretic properties (λ_{\max} 548 nm; mobility relative to betanin 0.72 at pH 2.4, 0.80 at pH 4.5). Since Oleracin I gave on acid hydrolysis the diastereoisomeric aglycones betanidin and isobetanidin and Oleracin II only isobetanidin, the former is a betanidin and the latter an isobetanidin derivative. Since Oleracin I treated with aq. citric acid [1] gave a mixture of Oleracin I and II, they are clearly diastereoisomers. On alkali treatment, the mixture yielded ferulic acid and two new diastereoisomeric pigments (DO1 and DO2) which were separated by chromatography on polyamide. These pigments had indistinguishable spectral properties (λ_{\max} 537 nm). DO 1 gave on complete acid hydrolysis a mixture of betanidin and isobetanidin and compound DO2 only isobetanidin; thus DO1 is a betanidin and DO2 the corresponding isobetanidin derivative. Controlled acid hydrolysis of DO1 and DO2 mixture with 10% acetic acid (3.5 hr under reflux) gave 2 sugars, identified as glucose and cellobiose by comparison with authentic materials. When a mixture of Oleracin I and II was methylated with CH_2N_2

followed by alkali fusion, 5-hydroxy-6-methoxyindole-2-carboxylic acid was obtained. Thus cellobiose is bound to the hydroxyl group at position 5 and the phenolic hydroxylic group at position 6 is free. Since controlled acid hydrolysis (1 N HCl; 10 min at 80°) of DO1 + DO2 gave in addition to the products of total hydrolysis, small amounts of betanin and isobetanin, the disaccharide-aglycone linkage is β . Thus DO1 is betanidin 5-*O*-cellobioside and DO 2 is isobetanidin 5-*O*-cellobioside.

Acknowledgement—I thank Prof. M. Piattelli (Catania, Italy) for helpful discussion.

REFERENCES

1. Piattelli, M. and Minale, L. (1964) *Phytochemistry* **3**, 547.
2. Minale, L., Piattelli, M., De Stefano, S., Nicolaus, R. A. (1966) *Phytochemistry* **5**, 1037.
3. Minale, L., Piattelli, M. and De Stefano, S. (1967) *Phytochemistry* **6**, 703.
4. Piattelli, M. and Impellizzeri, G. (1969) *Phytochemistry* **8**, 1595.
5. Piattelli, M. and Imperato, F. (1970) *Phytochemistry* **9**, 455.
6. Piattelli, M. and Imperato, F. (1970) *Phytochemistry* **9**, 2557.
7. Piattelli, M. and Imperato, F. (1971) *Phytochemistry* **10**, 3133.

Phytochemistry, 1975, Vol. 14, pp. 2092-2093, Pergamon Press. Printed in England.

TECLEANONE FROM *DIPHASIA KLAINEANA* AND *TECLEA VERDOORNIANA*

PETER G. WATERMAN

Department of Pharmaceutical Chemistry, University of Strathclyde, Glasgow G1 1XW, Scotland

(Received 17 March 1975)

Key Word Index—*Diphasia klaineana*; *Teclea verdoorniana*; Rutaceae; acridone alkaloid; tecleanone; chemotaxonomy.

Plants. *Diphasia klaineana* Pierre-Enti 490A and Enti 490B; *Teclea verdoorniana* Exell et Mendonca-Enti 390 and Enti R 789. Voucher specimens have been deposited at the herbarium of the Royal Botanic Garden, Edinburgh. *Source.* *D. klaineana* from beside the Awutu-Winneue road and *T. verdoorniana* from the Neung Forest Reserve, Tarkwa, Ghana. *Uses.* Both species are used by the indigenous population as a cure for

various ailments [1]. *Previous work.* On the stem and root barks of *D. klaineana* [2] and *T. verdoorniana* [2-3] and on other species of *Teclea* [4-5].

Present work. The isolation of small quantities of an unidentified alkaloid, designated DK/1, from *D. klaineana* (8 mg) and *T. verdoorniana* (5 mg) has been described previously [2]. From a second, larger, collection of *D. klaineana* root bark (Enti 490B, 1 kg) more of this compound