



(I)

EXPERIMENTAL

Two-dimensional chromatographs on Whatman 3MM paper were developed first in TBA (*t*-BuOH-HOAc-H₂O, 3 : 1 : 1) and then in 15% HOAc. The NMR spectra were recorded in CCl₄ and benzene-*d*₆ using tetramethylsilane as an internal standard; UV spectra were recorded using standard procedures.³

Air-dried and ground leaf material of *Parthenium tomentosum* L. (collected 31 miles N. of Oaxaca, Oaxaca, México; Rodríguez and Whiffin No. 48*) was extracted with 85% aq. MeOH and the extract was filtered and concentrated. The aqueous solution which remained was extracted repeatedly with EtOAc, and these extracts were combined and evaporated to dryness. The reddish syrup (700 mg) was chromatographed over polyamide (30 g packed in CHCl₃); the column was eluted with CHCl₃-MeCOEt-MeOH (5 : 1 : 2). The first few fractions contained mostly phenolic acids and the final fractions yielded a mixture of flavonoids which are currently under investigation. The middle fractions afforded 12 mg of the new flavonol quercetagenin 3,3'-dimethyl ether; the material crystallized from benzene-acetone, m.p. (uncorrected) 214–215°; *R_f* (TBA) 61; (HOAc) 19; UV λ_{max} (MeOH): 255sh, 276 348 nm; λ_{max} (NaOMe): 260, 393 nm (slow dec.); λ_{max} (AlCl₃): 276, 295sh, 345, 434 nm; λ_{max} (AlCl₃-HCl): 263, 290, 374 nm; λ_{max} (NaOAc): 263, 365 nm (slow dec.); λ_{max} (NaOAc-H₃BO₃): 270sh, 285, 352 nm. MS measurement showed a parent peak at 346 (C₁₇H₁₄O₈ required 346), a large fragment at 345 (60% intensity of parent ion) and a small fragment at 403 (15% intensity of parent peak) for the loss of the C-3(CH₃CO) group.⁵

Acknowledgements—This investigation was supported by the National Institutes of Health (Grant HD-04488) and The Robert A. Welch (Grant F-130), and National Science (Grants GB-29576X and GB-16411) Foundations.

* The voucher specimen is deposited in the University of Texas at Austin Herbarium.

Key Word Index—*Parthenium tomentosum*; Compositae; quercetagenin 3,3'-dimethyl ether.

Phytochemistry, 1972, Vol. 11, pp. 1508 to 1509. Pergamon Press. Printed in England.

EBENACEAE

STEROIDS AND TRITERPENOIDS OF *DIOSPYROS MONTANA*

G. MISRA, S. K. NIGAM and C. R. MITRA

Utilization Research Laboratory, National Botanic Gardens, Lucknow, India

(Received 19 October 1971)

Plant. *Diospyros montana* Roxb. *Uses.* Medicinal.^{1,2} *Previous work.* Bark.³ On sister species.³⁻⁵

¹ ANON, *The Wealth of India, Raw Materials*, Vol. 3, p. 84, CSIR, New Delhi, India (1952).

² R. N. CHOPRA, S. L. NAYAR and I. C. CHOPRA, *Glossary of Indian Medicinal Plants*, p. 98, CSIR, New Delhi, India (1956).

³ R. S. KAPIL and M. M. DHAR, *J. Sci. Industr. Res.* **20B**, 498 (1961); and references cited therein.

⁴ A. V. B. SANKARAN and G. S. SIDHU, *Phytochem.* **10**, 458 (1971).

⁵ P. S. MISRA, G. MISRA, S. K. NIGAM and C. R. MITRA, *Phytochem.* **10**, 904 (1971); and references cited therein.

Fruit pulp. EtOH concentrate, extrn. with *n*-hexane and Et₂O, chromatography (silica gel): *Viscous yellow liquid*, IR (ester), sapn. with alc. KOH to α -amyrin, C₃₀H₅₀O, m.p., [α]_D, m.m.p.,⁶ co-TLC, IR, acetate, m.p. and *fatty acid mixture* \rightarrow palmitic and stearic acids, co-TLC. *Sitosterol*, C₂₉H₅₀O, m.p., [α]_D, m.m.p.,⁵ IR, co-TLC, acetate, C₃₁H₅₂O₂, m.p., IR. *Betulin*, C₃₀H₅₀O₂, m.p., [α]_D, IR, m.m.p.,⁵ co-TLC, diacetate, C₃₄H₅₄O₄, m.p., [α]_D, m.m.p.,⁵ IR. *Triterpene acid mixture*, methylation (CH₂N₂), chromatography (Al₂O₃): Methyl esters of *ursolic* (yield 0.07% of the pulp), *oleanolic* and *betulinic acids*, C₃₁H₅₀O₃, identified through m.p., m.m.p.,^{5,7,8} [α]_D, IR, MS, their methyl ester acetates, C₃₃H₅₂O₄, m.p., m.m.p., IR. Ursolic and oleanolic acids were further confirmed by LiAlH₄ reduction⁷ of the former to uvaol, C₃₀H₅₀O₂, m.p., IR and SeO₂ oxidation⁸ of the latter to $\Delta^{11,13(18)}$ -diene, UV, respectively.

Seed. Stony hard; extrn. EtOH, *n*-hexane soluble fraction, chromatography: *Betulinic acid*, identified as above. Extrn. with *n*-hexane, insignificant traces of fat.

Studies in the various constituents of the fruit pulp and the seed of *Diospyros montana* and *D. peregrina*,⁵ reveal that the seed-embryos of both the species hardly yield any fat; betulinic acid is the only constituent present. Sitosterol and betulin are the common constituents of the pulps, in addition *D. montana* pulp yields fatty acid esters of α -amyrin and ursolic, oleanolic and betulinic acids and the other species yields hexacosane, hexacosanol, β -D-glucoside of sitosterol, gallic acid and a triterpene ketone.

Acknowledgements—Authors' thanks are due to Messrs. J. Saran, R. K. Mukerji and R. K. Singh for micro-analyses, IR and MS respectively.

⁶ C. R. MITRA and G. MISRA, *Phytochem.* **4**, 345 (1965).

⁷ G. MISRA and C. R. MITRA, *Phytochem.* **7**, 2173 (1968).

⁸ S. K. NIGAM and C. R. MITRA, *Planta Med.* **18**, 44 (1970).

Key Word Index—*Diospyros montana*; Ebenaceae; steroids; triterpenoids; hydrocarbons.

Phytochemistry, 1972, Vol. 11 pp. 1509 to 1510. Pergamon Press. Printed in England.

ERICACEAE

ISOLATION OF ISOPYROSIDE FROM *VACCINIUM VACILLANS*

ALI ASKARI* and LEONARD R. WORTHEN

College of Pharmacy, University of Rhode Island, Kingston, RI 02881, U.S.A.

(Received 13 July 1971, in revised form 17 November 1971)

Plant. *Vaccinium vacillans* Torr. from Rhode Island U.S.A. **Previous work.** Friedrich first reported¹ the presence of pyroside (6'-acetyl-arbutin) in pear leaves (*Pyrus communis* L.) and mountain cranberry leaves (*Vaccinium vitis-idaea* L.).² Haslam *et al.* have confirmed³

* Present address: University of Sulaimani, College of Agriculture, Sulaimani, Iraq.

¹ H. FRIEDRICH, *Pharmazie* **15**, 319 (1960).

² H. FRIEDRICH, *Naturwiss.* **48**, 304 (1961).

³ E. HASLAM, M. O. NAUMANN and G. BRITTON, *J. Chem. Soc.* 5649 (1964).