than that found in higher plants.[9] A comparison of the two extraction methods used with *S. recurvum* shows only slight differences in alkane content due possibly to an increased amount of 'internal alkanes'[10] in the Soxhlet extracted material. We have also noted an increased alkane content (9%) in the sample extracted by dipping as opposed to 2·5% of alkanes in the Soxhlet extracted material.

EXPERIMENTAL

Isolation of the alkanes. Powdered air-dried mosses were exhaustively extracted with light petrol. in a Soxhlet. In addition, whole samples of *S. recurvum* were dipped in light petrol. for 2 × 30 min. The vacuum evaporated residues were chromatographed on 1 mm layers of Kieselgel PF254. After development with light petrol., marker strips were sprayed with Rhodamine B. The least polar band was eluted with $CHCl_3$, co-TLC and film-thin IR revealed the presence of alkanes only.

GLC analysis. The alkane mixtures were analysed on a Perkin–Elmer F11 (FID) using 2·5% SE 301 on Chromosorb G AW.DMCS in glass; nitrogen 43 ml/min. Temperatures (i) identification of alkanes *n*-C_{19} to *n*-C_{25}; 205°, (ii) identification of alkanes *n*-C_{26} to *n*-C_{35}; 230°, (iii) quantitative estimation 180–240° at 10° min. Alkanes were identified by comparison of their retention data with those of standard alkanes and from a plot of Hydrocarbon number V_s Log R_t. The relative amounts of each alkane was determined by peak area measurements.

Acknowledgements—We wish to thank the Pharmaceutical Society of Ireland for financial support and Professor N. R. Farnsworth (University of Illinois) for a gift of reference *n*-alkanes.

[9] K. STRANSKY, M. STREIBL and V. HEROUT, *Coll. Czech. Chem. Commun.* **32**, 3213 (1967).
[10] G. A. HERBIN and P. A. ROBINS, *Phytochem.* **8**, 1985 (1969).

Phytochemistry, 1973, Vol. 12, pp. 214 to 216. Pergamon Press. Printed in England.

ANGIOSPERMAE DICOTYLEDONAE

AMARANTACEAE, ETC.

CRYSTALLINE CHEMICAL COMPONENTS OF SOME VEGETABLE DRUGS

A. K. BATTA and S. RANGASWAMI

Department of Chemistry, University of Delhi, Delhi-7, India

(*Received* 14 *August* 1972. *Accepted* 1 *October* 1972)

Key Word Index—*Achyranthes aspera*; Amarantaceae; *Betula utilis*; Betulaceae; *Quercus infectoria*; Fagaceae; *Punica granatum*; Punicaceae; sterols; triterpenoids.

In the course of our study of the chemistry of vegetable drugs used in Indian medicine from ancient times, we had occasion to reinvestigate the following plant materials in detail. A number of known compounds were isolated, some of them fairly common but also some very rare. The drugs were extracted with solvents of increasing polarity in succession and the solvent-free residues crystallised either directly or after purification by PC on silica gel. The substances were converted into appropriate derivatives and for confirming identity, comparisons were made with authentic samples in all the cases, employing m.p., m.m.p., $[\alpha]_D$, TLC and IR spectra as criteria. Results described in the earlier literature and as obtained by us are mentioned below. Rotations were taken in $CHCl_3$ except where stated otherwise.

I. Seeds of *Achyranthes aspera* Linn. (Amarantaceae) used as emetic and in hydrophobia. *Previous results.* Two oleanolic acid-based saponins called saponin A and saponin B from the fruit[1] and ecdysone from the roots.[2]

Present results. The total saponins were hydrolysed with acid and from the genin fraction oleanolic acid was crystallized out and the mother liquor treated with CH_2N_2 and chromatographed. The following were obtained: (a) Oleanolic acid methyl ester (evidently from the residual genin of the above two saponins). (b) Maslinic acid methyl ester (eluted by $CHCl_3$–MeOH, 49:1), m.p. 216–219°, reaction with $NaIO_4$ and acetonide formation. For further data see under IV *Punica granatum* below.

II. Bark of *Betula utilis* D. Don. (Betulaceae) used as antiseptic. *Previous results.* Lupeol, betulin, oleanolic acid and its acetate[3] and betulic acid.[4]

Present results. Light petrol. and Et_2O extracts were almost identical, and subsequent extractions with acetone and EtOH yielded only negligible quantities of extractives. Chromatography of the combined light petrol. and Et_2O extracts yielded, besides the above five, the following additional compounds: (a) *Lupenone*, m.p. 170–171°, $[\alpha]_D$ +52·3°, ν_{max}^{KBr} 1710 (*s*), 1659 (*w*), 1460 (*s*), 1380 (*m*), 890 (*m*), 870 (*s*)cm^{-1}. Reference sample was prepared by Jones' oxidation of lupeol. (b) *Methyl betulonate*, m.p. 165–166°, $[\alpha]_D$ +35·6°, ν_{max}^{KBr} 1745 (*s*), 1705 (*s*), 1650 (*m*), 1460 (*s*), 1380 (*m*), 1370 (*m*), 1185 (*m*), 1160 (*s*), 1125 (*m*) 908 (*m*), 879 (*s*) cm^{-1}. Reference sample was obtained by Jones' oxidation of the methyl ester of betulic acid. This is the first instance of its occurrence as natural product. (c) *Sitosterol* (derivative: acetate). (d) *Methyl betulate*, m.p. 222–223°, $[\alpha]_D$ +10·1°, ν_{max}^{KBr} 3600 (*m*), 1740 (*s*), 1650 (*w*), 1470 (*s*), 1380 (*m*), 1200 (*m*), 1170 (*m*), 1140 (*m*), 1050 (*m*), 890 (*m*) cm^{-1}.

III. Galls of *Quercus infectoria* Oliv. (Fagaceae) used as astringent and styptic. *Previous results.* Gallotannins.[5]

Present results. From the light petrol. extract, the following compounds were obtained on chromatography of the diazomethane treated product: (a) sitosterol, (b) methyl betulate, and (c) methyl oleanolate.

IV. Flowers of *Punica granatum* L. (Punicaceae) used as astringent and styptic. *Past results.* Pelargonidin-3,5-diglucoside.[6]

Present results. From light petrol. extract. Sitosterol (derivative: acetate) and ursolic acid (derivatives: acetate and methyl ester). *From $CHCl_3$ extract.* Sitosterol and ursolic acid as major components and the following three as minor components: (a) Maslinic acid (eluted by $CHCl_3$–MeOH, 19:1), m.p. 258–260°, $[\alpha]_D$ +48·4° (py), ν_{max}^{KBr} 3500 (*m*), 1700 (*s*), 1460 (*s*), 1380 (*m*), 1050 (*w*), 870 (*w*), 832 (*w*) cm^{-1}. A clue to its identity came from a close study of the methyl ester (m.p. 217–219°, $[\alpha]_D$ +18·0°), which reacted with $NaIO_4$, yielded an acetonide and gave the following mass peaks (% abundance): 484 (M^+, 7·3), 431 (1·8), 262 (90·7), 249 (11·6), 223 (9·3), 203 (100), 189 (20·4). The reference sample was prepared from oleanolic acid following the method of Caglioti *et al.*[7] (b) Asiatic acid (eluted by $CHCl_3$–MeOH, 93:7), m.p. 300–306°, ν_{max}^{KBr} 3550 (*m*), 1700 (*s*), 1650 (*w*), 1460 (*s*), 1380 (*m*), 1370 (*m*), 1050 (*s*), 870 (*w*), 828 (*w*) cm^{-1}. It also reacted with $NaIO_4$ and yielded an acetonide

[1] V. HARIHARAN and S. RANGASWAMI, *Phytochem.* **9**, 409 (1970).
[2] A. BANERJI and M. S. CHADHA, *Phytochem.* **9**, 1671 (1970).
[3] V. M. CHARI, T. R. SESHADRI and S. NEELAKANTAN, *Indian. J. Chem.* **6**, 231 (1968).
[4] T. R. SESHADRI and T. N. C. VEDANTHAM, *Phytochem.* **10**, 897 (1971).
[5] E. HASLAM, *Chemistry of Vegetable Tannins*, p. 92, Academic Press, London (1966).
[6] L. PONNIAH and T. R. SESHADRI, *J. Sci. Ind. Res.* **12B**, 605 (1953).
[7] L. CAGLIOTI and G. CAINELLI, *Tetrahedron* **18**, 1061 (1962).

but had an R_f lower than maslinic acid. The reference sample was obtained from *Centella asiatica*.[8] (c) Sitosterol-β-D-glucoside (eluted by $CHCl_3$–MeOH, 23:2), m.p. >300°. Positive Molisch's test. Hydrolysis with acid yielded sitosterol and D-glucose. *From Alcohol extract.* D-Mannitol, m.p. 166° (derivative: acetate), ellagic acid, m.p. >300° (both obtained by direct fractional crystallization) and gallic acid, m.p. 256–258° (obtained by chromatography).

Acknowledgements—The authors thank Professor T. R. Seshadri for his interest and the Central Council for Research in Indian Medicine and Homoeopathy for financial assistance.

[8] P. BOITEAU, A. BUZAS, E. LEDERER and J. POLONSKY, *Bull. Soc. Chim. Biol.* **31**, 46 (1949).

Phytochemistry, 1973, Vol. 12, p. 216. Pergamon Press. Printed in England.

APOCYNACEAE
TRITERPENES OF *PARSONSIA STRAMINEA* LEAVES

K. JEWERS and A. H. MANCHANDA

Tropical Products Institute, 56–62 Gray's Inn Road, London WC1X 8LU

G. BLUNDEN

School of Pharmacy, Portsmouth Polytechnic, Park Road, Portsmouth PO1 2DZ

and

W. J. GRIFFIN

Department of Pharmacy, University of Queensland, Brisbane, Australia

(*Received* 30 *August* 1972. *Accepted* 1 *October* 1972)

Key Word Index—*Parsonsia straminea*; Apocynaceae; ursolic acid; lupeol; α-amyrin.

In a recent paper one of us reported[1] the isolation of two triterpenes from the leaves of *Parsonsia straminea*. We now wish to report on the identity of these compounds.

Compound *A*, a triterpene acid, m.p. 277–279° (M^+, $m/e = 456$) showed in its IR spectrum strong absorptions at 3440, 1690 and 1030 cm^{-1}, indicating the presence of hydroxyl and carboxyl groups. The base peak of the MS occurred at *m/e* 248 which further fragmented to an ion of *m/e* 203, thus suggesting that the compound was either an ursa-12-en or oleana-12-en-28-carboxylic acid.[2] An ion at *m/e* 207 was indicative of the hydroxyl group located at C_3. The NMR spectrum in deuteropyridine[3] indicated that the compound was ursolic acid, and this was supported by the physical constants of the methyl ester and the acetyl derivatives.

The IR and MS of compound *B* suggested that it was a mixture of pentacyclic triterpene alcohols. Separation of the acetylated material on argentized silica gel afforded lupeol acetate and α-amyrin acetate.

EXPERIMENTAL

Separation of compound B. After acetylation with acetic anhydride and pyridine, the product (50 mg) was subjected to preparative TLC on 10% argentized silica gel, using light petrol.–Et_2O (9:1) as developing solvent. The bands were visualized by spraying with 2,7-dichlorofluoresceine and the compounds eluted with Et_2O. This afforded lupeol acetate (18 mg), m.p. 213–217°, and α-amyrin acetate (28 mg), m.p. 220–222°.

[1] W. J. GRIFFIN and J. E. PARKIN, *Planta Med.* **20**, 97 (1971).

[2] H. BUDZIKIEWICZ, C. DJERASSI and D. H. WILLIAMS, *Structure Elucidation of Natural Products by Mass. Spectrometry*, Vol. 2, p. 122, Holden Day, New York (1964).

[3] K. JEWERS and A. H. MANCHANDA, unpublished results.