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MORETENOL AND OTHER CONSTITUENTS OF *CELTIS LAEVIGATA*

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Key Word Index—*Celtis laevigata*; Ulmaceae; *n*-alkanes; fatty acids; moretenol; sitosterol; stigmasterol.

Plant. Celtis laevigata Willd. *Source.* Montgomery, Alabama, U.S.A. *Use.* Source of Wood. *Previous work.* Cellulose and α-cellulose[1]. *Present work.* The dried, ground leaves (3·2 kg) were extracted by percolation with EtOH. After removal of the solvent *in vacuo* at 40°, the residue (321 g) was partitioned between 2% HCl and $CHCl_3$ to give basic (6 g) and non-basic (205 g) fractions. The non-basic fraction was fractionated by standard methods into neutral (179 g), acidic (9 g), and phenolic (11 g) fractions.

Neutral fraction. Chromatography over silicic acid and elution with light petrol. gave an alkane fraction which crystallized from EtOAc (15 mg); mp 63–65°; ν_{max}^{KBr} cm^{-1}: 2940, 2870, 1470, 1380, 730 and 720. GLC on a 160 cm column of 0·8% OV-17 on Gas Chrom Q (80–100 mesh) showed the mixture to be composed primarily of C_{25} to C_{35} *n*-alkanes; C_{25} (1%), C_{26} (2), C_{27} (10), C_{28} (11), C_{29} (20), C_{30} (12), C_{31} (22), C_{32} (5), C_{33} (5), C_{34} (1), C_{35} (1). The identity was confirmed by GC–MS. Elution with light petrol–$CHCl_3$ (1:3) gave a fatty acid fraction which crystallized from MeOH (350 mg) mp 77–78°; ν_{max}^{KBr} cm^{-1}: 2940, 2860, 1700, 1460, 1430, 1300, 930, 730, 720 and 680. GLC of the methyl esters on a 160 cm column of 0·8% OV-17 on Gas Chrom Q (80–100 mesh) showed the mixture to be composed of C_{24} (7%), C_{25} (6), C_{26} (29), C_{27} (5), C_{28} (38), C_{29} (3) and C_{30} (12) saturated straight fatty acids. The identity was confirmed by GC–MS.

Elution with light petrol–$CHCl_3$ (1:5) afforded a fraction which was re-chromatographed over silicic acid. Elution with C_6H_6–$CHCl_3$ (3:2) and subsequent preparative TLC on Si gel PF_{254} ($CHCl_3$, R_f 0·3) gave moretenol (60 mg) mp 225–226° (Abs. EtOH) (lit. [2] 236° [$CHCl_3$–MeOH]); $[\alpha]_D^{27}$ + 26·3° ($CHCl_3$, *c* 0·9) (lit. [2] + 27° [*c* 2·3]; λ_{max}^{EtOH} nm (log e): 205 (3·57) (lit. [2] 210 [2·73]); ν_{max}^{KBr} cm^{-1}: 3670, 3350, 3200, 3080, 2940, 2860, 1640, 1440, 1390, 1380, 1045, 990 and 890; MS M^+ *m/e* 426 (38%), 411 (14), 393 (5), 207 (41), 189 (100), 135 (37); $\delta_{60\,MHz}^{CDCl_3}$ 0·68 (Me, 3H, *s*), 0·75 (Me, 3H, *s*), 0·82 (me, 3H, *s*), 0·95 (2 × Me, 6H, *s*), 1·30 (Me, 3H, *d*), 1·60 (Me, 3H, *d*), 3·20 (C<u>H</u>–OH, 1H, *m*) and 4·70 (C = C<u>H</u>$_2$, 2H, *s*). Treatment with Ac_2O–C_5H_5N gave *O*-acetylmoretenol mp 266–268° (Light Petrol.) (Lit. [2] 283–285° [Et_2O–MeOH]); $[\alpha]_D^{27}$ + 24·0° $CHCl_3$, *c* 0·9) (lit. [2] + 24° [*c* 1·6]); ν_{max}^{KBr} cm^{-1}: 3080, 2940, 2860, 1725, 1640, 1440, 1390, 1380, 1250, 1025, 1005, 990, 980, and 890; MS M^+ *m/e* 468 (38%), 453 (9), 408 (12), 393 (12), 249 (8), 203 (14), 189 (100). Direct comparison (mp, mmp, Sp. Rotn., IR, MS) with an authentic sample of *O*-acetylmoretenol confirmed the identity. To our knowledge, this is the second report of the

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natural occurrence of moretenol and the first in the Ulmaceae.

Elution with light petrol–$CHCl_3$ (1:9) gave a sterol mixture which crystallized from $CHCl_3$–MeOH (460 mg); mp 135–137°. ν_{max}^{KBr} cm^{-1}: 3430, 2950, 2930, 2860, 1640, 1460, 1380, 1060, 1050, 1020, 960 and 800. GLC on a 160 cm column of 0·8% OV-17 on Gas Chrom Q (80–100 mesh) showed the mixture to be composed of sitosterol (82%) and stigmasterol (18%). The identity was confirmed by GC–MS.

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COUMARINS FROM *HERACLEUM WALLICHII* AND *H. NEPALENSE*

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Key Word Index—*Heracleum wallichii*; *Heracleum nepalense*; Umbelliferae; coumarins; isobergapten; bergapten; isopimpinellin; sphondin.

Plant Heracleum wallichii DC. (Voucher specimen No. 12725, deposited at Survey and Herbarium Division, R.R.L., Jammu). *Source.* The Himalayas, 10000–12000 ft. *Previous work.* Nil.

Present work. The petrol. (bp 60–80°) extract of the roots on chromatography over SiO_2 gel afforded (a) isobergapten (petrol–C_6H_6 (2:1) eluate), $C_{12}H_8O_4$, mp 220–222°, confirmed by IR and NMR, (b) bergapten (petrol–C_6H_6 (1:1) eluate), $C_{12}H_8O_4$, mp 188–189°, confirmed by IR, NMR and co-TLC with authentic sample and (c) a crystalline fraction (C_6H_6–$CHCl_3$ (2:1) eluate) showing 2 spots on TLC. This mixture on rechromatography over SiO_2 gel furnished (d) isopimpinellin (pale yellow needles), $C_{13}H_{10}O_5$, mp 150–151°, confirmed by IR, NMR and TLC and (e) sphondin, $C_{12}H_8O_4$, mp 191–192°, confirmed by IR, NMR and TLC. R_f values of isobergapten, bergapten, isopimpinellin and sphondin on SiO_2 gel were found to be 0·61, 0·52, 0·31 and 0·25 respectively in cyclohexane–EtOAc (3:1) system.

Plant. Heracleum nepalense D. Don (Voucher specimen No. 13208, deposited at Survey and Herbarium Division, R.R.L., Jammu). *Source.* The Himalayas, 10000–12000 ft. *Previous work.* On seeds [1].

Present work. The petrol (bp 60–80°) extract of the roots on chromatography over SiO_2 gel yielded isobergapten, bergapten, isopimpinellin and sphondin.

Heracleum plants are noted for their rich furanocoumarin content. Furanocoumarins are effective dermal photosensitizing agents [2] and are widely used in the treatment of leucoderma and in various 'Suntan' lotions. The furanocoumarin (total) contents of *Heracleum wallichii* and *H. nepalense* are about 1·2% and 1·5% respectively.

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