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RUTACEAE

ISOLATION OF SOME FURANOCOUMARINS FROM *CLAUSENA INDICA* AND IDENTITY OF CHALEPENSIN WITH XYLOTENIN*

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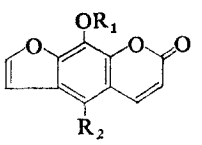
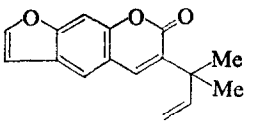
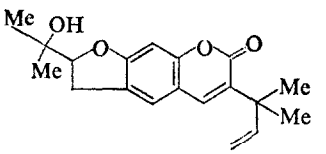
Abstract—From the roots of *Clausena indica* Oliv. the furanocoumarins imperatorin (I), phellopterin (III), chalepensin (IV) and the dihydrofuranocoumarin chalepin (V) have been isolated. Chalepensin has been shown to be identical with xylotenin.

WE HAD reported earlier[1,2] the structural elucidation of two pyranocoumarins clausenin and clausenidin from the roots of *Clausena heptaphylla* Wt. & Arn. The present study has shown the occurrence of the known furanocoumarins imperatorin (I), phellopterin (III), chalepensin (IV) and chalepin (V) in the roots of *Clausena indica* Oliv.





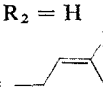
Chalepensin (IV)



Chalepin (V)

Imperatorin I; R_1 =  ; $R_2 = H$

II; $R_1 = R_2 = H$

Phellopterin III; R_1 =  ; $R_2 = OMe$

Imperatorin and phellopterin have been isolated from a number of species of the Rutaceae and Umbelliferae.[3] Chalepensin has been isolated only from *Ruta chalepensis* L.[4] and *R. graveolens* L.[5] Xylotenin, for which the structure (IV) was advanced, was isolated from *Chloroxylon swietenia* DC. (Rutaceae).[6] Comparison of chalepensin with a sample of xylotenin (by mixed m.p., TLC, i.r. and NMR spectral comparison) showed that the two are identical.

* Contribution No. 206 from CIBA Research Centre.

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The physical constants and the spectroscopic data (u.v., i.r. and NMR) of the furanocoumarin (V) isolated from *Clausena indica* were in agreement with those described for chalepin.[4] A direct comparison however could not be made. Heliettin[7] which appears to be the racemic form of chalepin, is identical with (V) in its u.v. and i.r. spectra.

EXPERIMENTAL

Extraction of Plant Material

The dried and powdered roots (20 kg) were extracted with hexane (b.p. 60–70°) (120 l.) and the extract concentrated to give a viscous oil (180 g).

Chromatographic Separation

The oil (160 g) dissolved in hexane (300 ml) was chromatographed on a column of silica gel (0·05–0·2 mm; 1·8 kg). The column was packed with hexane and gradient eluted with hexane, hexane–benzene and benzene–$CHCl_3$. 300 ml fractions were collected and examined by TLC.

(a) Fractions 79–118 (Elution; hexane–benzene 75%) gave chalepensin (IV) (26 g) m.p. 87–88° (TLC; R_f 0·6 in benzene–$CHCl_3$, 1:1) (Found, C, 76·1; H, 5·7 Calc. for $C_{16}H_{14}O_3$, C, 76·6; H, 5·6%), M.W. by m.s. 254. Mixed m.p. with an authentic sample of chalepensin showed no depression. TLC, u.v. and i.r. spectra were superimposable.

(b) Fractions 172–206 (Elution; benzene–$CHCl_3$, 1:1) gave β-sitosterol (1 g) m.p. 138°.

(c) Fractions 209–211 (Elution; benzene–$CHCl_3$ 75%) gave phellopterin (III) m.p. 102°. (TLC; R_f 0·84 in $CHCl_3$–MeOH 2%). It was found to be identical in mixed m.p. TLC and i.r. spectra when compared with an authentic sample.

Phellopterin (20 mg) on hydrolysis with AcOH containing a drop of H_2SO_4 gave a yellow crystalline phenolic compound m.p. 223°, identified as 5-methoxy-8-hydroxypsoralen.[8]

(d) Fractions 215–235 (Elution; benzene–$CHCl_3$ 75%) gave imperatorin (I) m.p. 98–99° (TLC; R_f 0·8 in $CHCl_3$–MeOH 2%). It was found to be identical in all respects when compared with an authentic sample.

Imperatorin (40 mg) on hydrolysis with AcOH containing a drop of H_2SO_4 gave pale yellow crystals (8 mg) m.p. 245–6°, identified as xanthotoxol (II).

(e) Fractions 236–245 (Elution with $CHCl_3$) gave chalepin m.p. 118–9° $[\alpha]_D$ + 28° (TLC, R_f 0·35 in $CHCl_3$–MeOH 2%).

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3-FORMYLINDOLE FROM *MURRAYA EXOTICA**

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OUR TAXONOMIC interest in the genus *Murraya* (Rutaceae)[1–4] prompted an investigation of *M. exotica* L (Syn. *M. paniculata*) from which we reported the isolation and structure proof

* Part XXII in the series "Chemical Taxonomy"; Part XXI S. P. BASAK and D. P. CHAKRABORTY (paper communicated).

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