

TABLE 2—1-(PHENYL/METHYLCARBAMOYLOXYMETHYL)-2-METHYL/PHENYL-4-(SUBSTITUTED BENZYLIDENE)-5-IMIDAZOLONES (21-41)

Compd. no.	R	R ¹	R ²	Position of heterocyclic	M.p. °C
21	H	OH ₂	CH ₃	2	108
22	H	OH ₂	OH ₂	3	122
23	H	OH ₂	OH ₂	4	118
24	H	C ₆ H ₅	CH ₃	2	197
25	H	C ₆ H ₅	CH ₃	3	220
26	H	C ₆ H ₅	CH ₃	4	231-2
27	H	C ₆ H ₅	C ₆ H ₅	2	154
28	H	C ₆ H ₅	C ₆ H ₅	3	206-7
29	H	C ₆ H ₅	C ₆ H ₅	4	186
30	p-Cl	CH ₃	C ₆ H ₅	2	111-2
31	p-Cl	CH ₃	C ₆ H ₅	3	193
32	p-Cl	CH ₃	C ₆ H ₅	4	142-3
33	p-Cl	C ₆ H ₅	C ₆ H ₅	2	137-8
34	p-Cl	C ₆ H ₅	C ₆ H ₅	3	177
35	p-Cl	C ₆ H ₅	C ₆ H ₅	4	186
36	p-OCH ₃	C ₆ H ₅	OH ₂	2	140
37	p-OCH ₃	C ₆ H ₅	OH ₂	3	133
38	p-OCH ₃	C ₆ H ₅	OH ₂	4	109
39	p-OCH ₃	C ₆ H ₅	C ₆ H ₅	2	187-9
40	p-OCH ₃	C ₆ H ₅	C ₆ H ₅	3	178-9
41	p-OCH ₃	C ₆ H ₅	C ₆ H ₅	4	158

Compounds no. 16, 20 and 25 recrystallised from benzene and the rest from benzene petroleum ether.

All the compounds gave satisfactory C, H and N analyses.

S. aureus and *E. coli*. Out of the compounds evaluated, 14 and 24 showed inhibition against all the bacteria. Regarding antifungal activity, four compounds, viz. 23, 25, 32 and 38 were effective against *A. terreus*, while only compounds 32 and 41 were effective in inhibiting the growth of *H. sativum*. All the compounds are active at higher concentrations and activity decreases markedly on dilution. Rest of the compounds were not active against any of the three species of fungi.

Acknowledgement

The authors thank the Head, Department of Chemistry, Lucknow University for providing facilities and Dr. S. Sharma of C.D.R.I. for suggestions. One of the authors (A.A.G.) is grateful to C.S.I.R., New Delhi for the award of senior research fellowship.

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Chemical Examination of the Leaves of *Woodfordia fruticosa*

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Manuscript received 28 May 1981, accepted 30 May 1984

WOODFORDIA fruticosa kurz.¹ (syn. *Woodfordia floribunda* Salisb) (Lythraceae) a beautiful much branched shrub with brilliant scarlet flowers, is very common in North India but scarce in Orissa and South India. Leaves of this plant are used for dying and tanning purposes². Besides, the leaves possess antibiotic^{3,4} as well as sedative⁵ properties.

The presence of naphthaquinone⁶, polyphenols⁷ and traces of alkaloid⁸ in the leaves were reported earlier. The present communication deals with the isolation and identification of triterpenoids and sterol in the leaves. The plant material (450 g) collected from Simlipal forest, Orissa, was exhaustively extracted with rectified spirit in a Soxhlet Extractor. The alcoholic extract was concentrated under reduced pressure. The dark coloured semi-solid mass obtained was separated into ether soluble and ether insoluble parts. Ether soluble part was then separated⁹ into acidic and neutral fractions.

The total acid fraction (0.21 g) was chromatographed over silica gel. Elution with CHCl₃: methanol (98:2) yielded a crystalline material showing one major and another minor spots in tlc (silica gel, CHCl₃, visualized with L-B reagent) which were separated by preparative tlc. The major compound on repeated crystallisation from methanol afforded betulinic acid as shining needles (11 mg), m.p. 310-312°. It yielded an acetate as plates, m.p. 291-293°, identical with authentic acetate of betulinic acid⁹ (m.p., m.m.p., ir). On methylation with diazomethane it gave a methyl ester (m.p. and m.m.p. 221-222°).

The minor compound was identified as oleanolic acid by comparative tlc with authentic oleanolic acid and by m.p. (308-310°). Further work could not be done due to paucity of material.

Chloroform: methanol (96:4) eluted another crystalline material (silica gel, CHCl₃: EtOAc 1:1, single purple spot with L-B reagent). It crystallised from methanol as needles (92 mg), m.p. 279-282°, [α]_D+64° (c 0.41 in CHCl₃); acetate m.p. 289-290°, [α]_D+60° (c 0.31 in CHCl₃); methyl ester m.p. 170° identical with authentic methyl ursolate¹⁰ (m.p., m.m.p., ir).

The neutral fraction was chromatographed over neutral alumina. Petroleum ether: benzene (1:1) eluted lupeol (100 mg) m.p. 210-211°, [α]_D+22° (c 2.0 in CHCl₃), acetate m.p. 209-211°, [α]_D+44° (c 0.37 in CHCl₃); benzoate m.p. 251-61°. Finally the identity was established as lupeol by comparison with authentic lupeol (m.p., m.m.p., ir). Benzene eluted β-sitosterol (38 mg), m.p. 136-137°; acetate m.p. 128°, [α]_D-35° (c 0.29 in CHCl₃); benzoate m.p. 144°. Identity of β-sitosterol was

established by comparison with an authentic specimen (m.p., m.m.p., superimposable ir).

The residue from benzene : CHCl_3 (9 : 1) eluates afforded betulin crystallises from benzene-methanol (18 mg), m.p. 253-255°; acetate m.p. 220-223°, $[\alpha]_D^{25} +21^\circ$ (c 0.30 in CHCl_3). The latter was identical with authentic betulin acetate¹¹ (m.p., m.m.p., ir).

The leaves of *W. fruticosa* was thus found to contain lupeol (0.023%), β -sitosterol (0.008%), betulin (0.004%), ursolic acid (0.022%), betulinic acid (0.001%) and minute quantity of oleanolic acid.

Acknowledgement

Authors thank Dr. S. K. Jain, Ex-Director, Botanical Survey of India, Howrah for facilities and Dr. S. C. Pakrashi, Deputy Director, Indian Institute of Molecular Biology, Calcutta for instrumental analyses.

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Chemical Investigation of *Viscum articulatum*

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Manuscript received 28 October 1983, revised 12 June 1984,
accepted 21 July 1984

VISCUM articulatum (Loranthaceae) is an epiphyte. No work on the chemical constituents of the plant appears to have been reported in the literature. We now report the result of chemical investigation on this plant.

The plant growing on *Diospyros peregrina* was collected from the district of Hazaribagh, Bihar.

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Three neutral and two acid triterpenes were isolated from the benzene extract of the air-dried and crushed plant material.

The neutral triterpenes were isolated in pure state from the mixture by repeated tlc over silica gel G impregnated with AgNO_3 following the reported method¹. The triterpenes thus obtained had melting points 186°, 211-212° and 258°, respectively. The compound having m.p. 186° has molecular formula $\text{C}_{30}\text{H}_{50}\text{O}$ (M^+426); monoacetate, $\text{C}_{32}\text{H}_{52}\text{O}_2$ (M^+468), m.p. 225°. Its mass spectral peaks at m/e 189, 207 and 218 were very similar to those of α -amyrin. The compound was identified as α -amyrin by comparing its m.p., m.m.p., tlc and glc data with an authentic sample. The compound having m.p. 211-212° has molecular formula $\text{C}_{30}\text{H}_{50}\text{O}$ (M^+426); monoacetate, $\text{C}_{32}\text{H}_{52}\text{O}_2$ (M^+468), m.p. 213-215°. The compound was identified as lupeol by comparison of its m.p., tlc and glc data with an authentic sample. The compound having m.p. 258° has molecular formula $\text{C}_{30}\text{H}_{50}\text{O}_2$ (M^+442). Its mass spectral peaks at m/e 189, 203, 207, 220 and 234 were very similar to those of betulin². The compound was identified as betulin by comparing its m.p., m.m.p., tlc and glc data with an authentic sample.

The two acid triterpenes could be isolated from the mixture by repeated tlc over silica gel G (solvent, CHCl_3 : petroleum ether (b.p. 60-80°) : HOAc = 64 : 33 : 3 v/v). The acids thus obtained had m.p. 304-308° and 314-316°, respectively. The acid, $\text{C}_{30}\text{H}_{48}\text{O}_8$, having m.p. 304-308° formed a monomethyl ester, $\text{C}_{31}\text{H}_{50}\text{O}_8$ (M^+470), m.p. 199-200°, on treatment with ethereal diazomethane. The mass spectrum of the ester showed characteristic peaks at m/e 207 and 262. The acid was identified as oleanolic acid by direct comparison of its m.p., m.m.p., tlc and glc data of its methyl ester with an authentic sample of methyl oleanolate. The second acid, $\text{C}_{30}\text{H}_{48}\text{O}_8$, m.p. 314-316°, formed a monomethyl ester, $\text{C}_{31}\text{H}_{50}\text{O}_8$, m.p. 224-225° (M^+470). The mass spectrum of the methyl ester showed peaks at m/e 189, 202, 203, 207, 220 and 262, very similar to those of methyl betulinate³ and was also supported by the nmr spectrum of the methyl ester. Finally, the acid was identified as betulinic acid by comparison of m.p., m.m.p., tlc and glc data of the methyl ester with an authentic sample.

It should be pointed out that mixture of α -amyrin, lupeol and betulin and also oleanolic acid, ursolic acid and betulinic acid are of common occurrence in the plant kingdom. Separation of individual constituents from above type of mixture even by preparative tlc is difficult and time-consuming. This can, however, be easily achieved by glc as described below.

Glc of some triterpenes: The instrument used was a Pye Unicam, model GCD gas chromatograph equipped with columns (1.8 m \times 3 mm i.d.) containing 3% and 6% SE-30; carrier gas N_2 at 60 ml min^{-1} ; column temp. 260° and 312°; detection and injection temp. 300° and 350°, respectively).