Leafless twigs. Disintegrated material was repeatedly extracted with hot 70% MeOH. The MeOH-free extract was subjected to ion exchange on a Dowex 1 × 1 column, followed by elution with a 5% K_2SO_4-solution as previously described.[2] Myrosinase-catalyzed hydrolysis of the purified glucosinolate fraction, performed in the usual fashion,[2] gave an isothiocyanate fraction which, on GLC, exhibited a major and a minor peak, indistinguishable, in retention times, from methyl and 2-propyl isothiocyanates, respectively. Faint traces of a third constituent, possessing a retention time identical with that of 2-butyl isothiocyanate, were discernable. On treatment with NH_3 in MeOH, the isothiocyanate fraction was converted into a mixture of thioureas, which after separation on a silica gel column, with an EtOH-gradient in $CHCl_3$ as the eluent, afforded crystalline specimens of 1-methyl and 1-(2-propyl)-thiourea, indistinguishable (m.m.p., IR and MS) from authentic specimens. Methylglucosinolate appears to be a widely occurring, thus far family-specific constituent of Capparidaceae[1] (see Ref. 3). In contrast, 2-propyl- and 2-butyl-glucosinolate, not infrequently encountered in Cruciferae, have been recorded only in a few members of Capparidaceae,[1] and, in these cases, to the exclusion of methylglucosinolate. The present finding constitutes the first case of co-occurrence of glucosinolates with methyl, 2-propyl, and, possibly, 2-butyl side chains.

[2] A. KJÆR and A. SCHUSTER, *Phytochem.* **10**, 3155 (1971).
[3] R. GMELIN and A. KJÆR, *Phytochem.* **9**, 569 (1970).

Phytochemistry, 1973, Vol. 12, pp. 726 to 727. Pergamon Press. Printed in England.

ISOLATION OF OBLIQUIN FROM *PHAENOCOMA PROLIFERA*

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Key Word Index—*Phaenocoma prolifera*; Compositae; coumarin; obliquin.

The roots of *Phaenocoma prolifera* (L) D.Don. (tribe, Inuleae) contain obliquin (I) previously isolated from *Ptaeroxylon obliquium* (Sapidinaceae).[1] The structure of I has been established by direct comparison of the spectroscopic data and by synthesis starting with aesculetin. Partial acetylation (II) and alkylation with 1,4-dibromo-2-methyl-2-butene yields the ether III, its structure being elucidated by comparing its NMR date with those of IV, obtained by hydrolysis. III, on reaction with KOH yields racemic I.

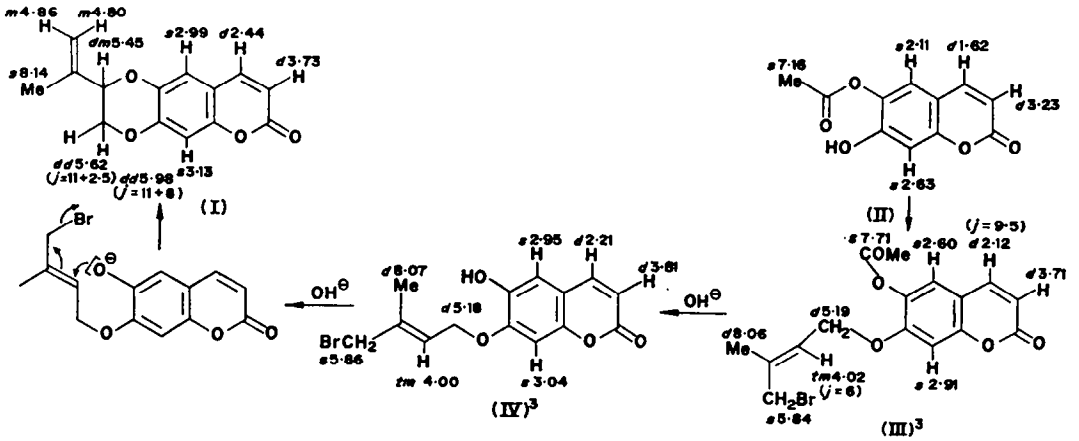
The occurrence of such a special coumarin in different families may be of chemotaxonomical importance.

EXPERIMENTAL

Isolation of I. 200 g fresh, ground material of *Phaenocoma prolifera* (L) D.Don. was extracted with Et_2O. The extract was purified by TLC (Et_2O–light petrol., 1:3) yielding 10 mg I, identical with authentic material.

[1] F. M. DEAN and B. PARTON, *J. Chem. Soc.* 526 (1969).

Synthesis of I. 1·0 g aesculetin in acetone was stirred with 1 g powdered KOH and 1·2 equiv. acetyl chloride for 1 hr at 50°. The reaction product, on crystallization from MeOH, yielded colourless crystals (II), m.p. 182° (yield 60%). UV. λ_{max} 345, 298·5, 257 nm (ε 10 700, 5700, 4800) 1·0 g (II) in acetone was stirred with 3 g powdered KOH and 3 g 1·4-dibromo-2-methyl-2-butene for 12 hr. After filtration, the



residue on chromatography yielded III, crystals from $CHCl_3$–Et_2O–light petrol., m.p. 123°, yield 70%. 60 mg III was warmed at 50° with 60 mg of powdered KOH in 15 ccm DMSO. The reaction product, on chromatography with Et_2O, yielded I, colourless crystals from $CHCl_3$–light petrol., m.p. 145°. IR, UV and NMR spectra identical with those of the natural product (Calc. for $(C_{14}H_{12}O_4$ (244·2): C, 68·85; H, 4·95. Found: C, 68·93, H, 4·94%).

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[2] NMR in DMSO-d_6, τ-values.
[3] NMR in acetone-d_6.

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NOREUGENIN FROM *RHODODENDRON COLLETTIANUM*

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Key Word Index—*Rhododendron collettianum*; Ericaceae; noreugenin.

Plant. Rhododendron collettianum (Aitch and Hemsel).[1] North West Frontier Province, Pakistan. *Uses.* medicinal.[2] *Previous Work.* On sister species.[3] *Present Work.* Dried aerial

[1] Voucher specimen No. 2765 (PES) deposited at P.C.S.I.R. Herbarium, Peshawar.
[2] R. N. CHOPRA, S. L. NAYAR and I. C. CHOPRA, *Glossary of Indian Medicinal Plants*, p. 213, CSIR, New Delhi (1956).
[3] J. B. HARBORNE and C. A. WILLIAMS, *Phytochem.* **10**, 2727 (1971).