

Fresh (fragrant) flowers (150 g) of *R. nasuta* collected from our campus during March were extracted with hot 80% ethanol under reflux and the combined extract concentrated *in vacuo*. The aqueous concentrate was fractionated successively with petrol (60-80°); peroxide-free ether and ethylacetate (EtOAc). The petrol and ether fractions on concentration did not yield any crystalline solid. The residue (0.1 g) from the EtOAc fraction was taken up in minimum of Me₂CO and left in an ice-chest for a few days. The dull yellow solid that separated on recrystallisation (twice) from aq. MeOH gave pale yellow needles, m.p. 186-88° (yield 0.04%), λ_{max} (MeOH) 256, 272, 360 nm, which exhibited bathochromic shifts expected of quercetin-3-O-glycoside with various shift reagents⁴ and appeared deep purple under uv turning yellow with NH₃.

To a solution of the glycoside (0.05 g) in hot aq. MeOH (5 ml, 50%), an equal volume of H₂SO₄ (14%) was added and the mixture refluxed at 100° for 2 hrs. The yellow solid that separated from the aqueous hydrolysate was filtered off. It was recrystallised from Me₂CO, m.p. 313-15° (decomp), pentaacetate, m.p. 193-95° and pentamethylether, m.p. 151-53° and was identified as quercetin on the basis of colour reactions, uv data, R_f and the identity confirmed by co- and mixed m.p. with authentic sample as well as its derivatives. The aqueous filtrate after removal of the above aglycone revealed the presence of glucose and rhamnose on PC. The R_f of the glycoside was also indicative of the presence of two sugar moieties. The glycoside was thus characterised as quercetin-3-O-rhamnoglycoside (rutin) and the identity confirmed by direct comparison with an authentic sample of rutin from *Cleome chelidonii*⁵.

It has been mentioned in the literature that luteolin is a characteristic flavone of the Acanthaceae⁶. The occurrence of chrysoeriol (4'-methoxy-luteolin) and uronides of luteolin and apigenin in this family has also come to light⁷⁻¹⁰. Acanthaceae is well known for unusual flavones¹¹ too. However, there are a few instances of the occurrence of flavonols and their 3-glycosides in this family like kaempferol-3-glucoside and kaempferol-3-sophoroside from *Adhatoda vasica*¹² and kaempferol-3-rungioside from *Rungia repens*¹³. The present report of the isolation of rutin from a plant belonging to the Acanthaceae provides yet another example of the occurrence of a flavonol-3-glycoside in this family which may have biochemical significance.

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References

1. "The Wealth of India, Raw Materials", C. S. I. R., New Delhi, 1972, 9, 6.
2. R. N. CHOPRA, S. L. NAVAR and I. O. CHOPRA, "Glossary of Indian Medicinal Plants", C. S. I. R., New Delhi, 1956, p. 212.
3. K. R. KIRTIKAR and B. D. BASU, "Indian Medicinal Plants", L. M. Basu, Allahabad, 1935, 3, 1904.
4. T. J. MABRY, K. R. MARKHAM and M. B. THOMAS, "The Systematic Identification of Flavonoids", Springer Verlag, New York, 1970, p. 35.
5. S. S. SUBRAMANIAN, A. G. R. NAIR and S. NAGARAJAN, *Curr. Sci.*, 1966, 34, 246.
6. A. G. R. NAIR, S. NAGARAJAN and S. S. SUBRAMANIAN, *Curr. Sci.*, 1965, 34, 79.
7. S. S. SUBRAMANIAN and A. G. R. NAIR, *Indian J. Chem.*, 1966, 4, 461.
8. S. S. SUBRAMANIAN and A. G. R. NAIR, *Curr. Sci.*, 1971, 40, 404.
9. J. B. HARBORNE, *Phytochem.*, 1966, 5, 111.
10. P. BALRAJ and S. NAGARAJAN, Unpubl. results.
11. J. B. HARBORNE, "Comparative Biochemistry of the Flavonoids", Academic Press, London, 1967, p. 225.
12. S. RANGASAMY and T. R. SESHADRI, *Curr. Sci.*, 1971, 40, 84.
13. T. R. SESHADRI and S. VYDESWARAN, *Phytochem.*, 1972, 11, 803.

Chemical Investigation of *Beaumontia grandiflora* Wall

MAHENDRA PAL SHARMA*, S. P. SRIVASTAVA,
V. K. MAHESH, M. P. SALUJA** and J. C. SHARMA***
Department of Chemistry, University of Roorkee,
Roorkee-247 672

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BEAUMONTIA grandiflora Wall (Family-Apocynaceae), a woody climber, is sometimes grown in Indian gardens for its showy white flowers. The seeds have been examined for a number of cardenolides²⁻⁴. Literature survey revealed that no work has been done on its leaves and stem. Therefore, the leaves were taken for the present chemical investigation. In all, seven compounds were isolated as reported in Table 1. Two of these were characterised as normal paraffins, two as aliphatic acids, two as triterpenoid compounds, and one as sterol glucoside. It may be mentioned that all these compounds are being reported for the first time in this plant.

Experimental

The powdered plant material was extracted with ethanol and total extractives successively macerated with petroleum ether (60°-80°) and ethylacetate. The chromatographic separation of pet. ether soluble fraction over deactivated alumina (with 1% aqueous acetic acid) led to the isolation of substances A, B, C, D and F while the ethylacetate soluble fraction led to the isolation of substances E and

** Department of Chemistry, J. V. Jain College, Saharanpur (U. P.).

*** Department of Chemistry, M. S. College, Saharanpur (U. P.).

G along with further quantities of substances D and F.

TABLE 1—SUBSTANCES ISOLATED FROM *B. grandiflora*

| Sl. No. | Compounds | Molecular formula | m.p. °C | Yield† percentage |
|---------|--|---|----------|-------------------|
| 1. | Substance A (<i>n</i> -Hentriacontane) | C ₃₁ H ₆₄ | 64 | 0.141 |
| 2. | Substance B (<i>n</i> -Triacontane) | C ₃₂ H ₆₆ | 66 | 0.026 |
| 3. | Substance C (Palmitic acid) | C ₁₆ H ₃₂ O ₂ | 61 | 0.023 |
| 4. | Substance D (β -sitosterol) | C ₂₇ H ₅₀ O | 135 | 0.050 |
| 5. | Substance E (Linoleic acid) | C ₁₈ H ₃₂ O ₂ | 202(BP)* | 0.032 |
| 6. | Substance F (Ursolic acid) | C ₃₀ H ₄₈ O ₂ | 283 | 0.108 |
| 7. | Substance G (β -sitosterol- β -D-glucopyranoside) | C ₅₃ H ₁₀₀ O ₆ | 301(d) | 0.031 |

† Based on the weight of dry plant material

* Yellowish viscous liquid

Characterisation of the substances A-G was based on physical data (uv, ir, nmr and mass spectra) and formation of acetyl and methyl ester derivatives, wherever possible.

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References

1. The Wealth of India, Raw Materials, C. S. I. R., New Delhi, 1950, 1, 166.
2. A. F. KRASSE, E. K. WEISS and I. REICHSTEIN, *Pharm. Acta Helva.*, 1964, 39, 168.
3. A. F. KRASSE and E. K. WEISS, *Helv. Chim. Acta*, 1963, 46, 1691.
4. N. G. BISSERT, *Ann. begr.*, 1957, 2, 193.

Occurrence of 2,7-Dihydroxy-3,4,6-trimethoxy-9,10-dihydrophenanthrene in *Coelogyne ovalis*, A High Altitude Himalayan Orchid: Application of C-13 NMR Spectroscopy in Structure Elucidation

P. L. MAJUMDER* and (MISS) SWAPNA LAHA

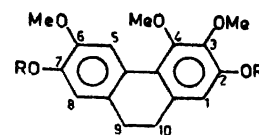
Department of Chemistry, University College of Science, Calcutta-700 009

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THE isolation of physiologically active alkaloids like dendrobine and a number of its structural analogues from the orchid *Dendrobium nobile*^{1,2} prompted us to undertake systematic chemical investigation of a series of high altitude Himalayan orchids. In this communication we report the

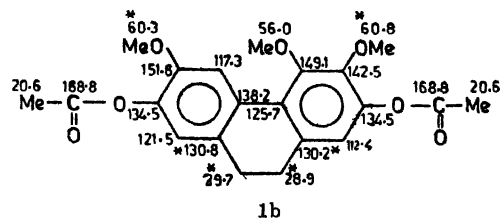
characterisation of the title compound (1a) isolated from one of these orchids, *Coelogyne ovalis*.

Air-dried powdered whole plant of *C. ovalis* (1 kg) was exhaustively extracted with chloroform in a soxhlet apparatus. The concentrated chloroform extract was chromatographed over silica gel. The petrol-ethylacetate (5:1) eluate afforded a crude phenolic mass which was taken in ether and extracted with 1N NaOH solution. The aqueous alkaline solution was acidified with HCl in the cold and the liberated solid was extracted with ether, dried and the solvent removed. The residue on repeated chromatography over silica gel gave a pure compound, C₁₇H₁₈O₅ (M⁺302), m.p. 109°, crystallised from petrol-benzene mixture. With Ac₂O and pyridine it formed a diacetyl derivative, C₂₁H₂₂O₇ (M⁺386), m.p. 107°. The physical constants and the spectral data (uv, ir, pmr and mass) of the coelogyne-phenol and its diacetate compare excellently with those reported³ for 1a isolated from the heart-wood of *Combretum psidioides* and its diacetyl derivative (1b) respectively. Since a direct comparison was not possible due to non-availability of authentic samples of 1a and 1b, further evidence was sought to confirm the structure



1a : R=H, 1b : R=Ac

of the coelogyne-phenol. The compound was heated in a sealed tube filled with nitrogen, with D₂O/KOBU⁴ for 70 hr. The ¹H-nmr spectrum of the product was identical with that of the parent compound except that the integrated areas of the aromatic proton signals at δ 6.55 (H-1) and 6.70 (H-8) were reduced to about 65% and 50% respectively, contrary to complete disappearance of these signals as reported³ earlier for 1a. The MS of the reaction mixture showed molecular ions at m/e 302 and 304. This observation is in conformity with the structure 1a for coelogyne-phenol.



Further confirmation of the structure of the compound was made by ¹³C-nmr spectral analysis of its diacetyl derivative. The carbon shifts and the degree of protonation of the carbons were determined by the proton-noise decoupled (NDC) and the single frequency off-resonance (SFORD) spectra, respectively of 1b. Excepting C-3, C-4 and C-5, the carbon shifts (the carbon shifts were measured using the relationship ⁵TMS = ⁵CDCl₃ + 76.9 ppm