EXPERIMENTAL

PLANT MATERIAL.—The roots of *R. undulata* were collected April 8, 1976, at Hennopspride near Pretoria. Voucher specimen (no. 882) is deposited in the Botanical Research Institute, Pretoria.

EXTRACTION AND FRACTIONATION.—Air-dried, milled, roots of *R. undulata* (3.5 kg) were successively extracted with C_6H_6 (235 g extract), EtOAc (83 g extract) and MeOH (279 g extract) at room temperature for 48 h. After removal of the solvents, the crude extracts were fractionated separately over silica gel (Kieselgel 60, 70-230 mesh; Merck). Elution was conducted with mixtures of petroleum ether, EtOAc, and MeOH of increasing polarity. Fractions with corresponding Rf values on the tlc [petroleum ether-EtOAc (1:1)] were combined into three groups. Of the groups obtained, group 2 was found to exhibit antiinflammatory activity.

ISOLATION OF APIGENIN DIMETHYLETHER.—The active group was chromatographed over silica gel and elution with C_6H_6 gave the title compound that crystallized from EtOAc as fine yellow needles (1.1 g; 0.18% of total extract), mp 171°-172° [Lit (7) mp 170°-171°]; ir ν max (KBr) 3450, 1665, 1605, 1510, 1338, 1310, 1270, 1215, 1190, 1185, 1160, 1022, 1012, 830, 815, and 760 cm^{-1}; ^{1}H-nmr ($CDCl_3$) δ 12.67 (1H, s, disappears on deuteration, OH), 7.83 (2H, dd, J=2.5 Hz and J=8.5 Hz, H-2′, 6′), 6.99 (2H, dd, J=2.5 Hz and J=8.5 Hz, H-3′, 5′), 6.55 (1H, s, H-3), 6.46 (1H, d, J=2.5 Hz, H-8), 6.33 (1H, d, J=2.5 Hz, H-6), 3.8 (6H, s, 2×OMe); ms *m/z* (%) 398 M+ (100).

IDENTIFICATION OF APIGENIN DIMETHYLETHER.—The physical data of apigenin dimethylether are in agreement with those reported in the literature (7,8).

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FIVE COUMARINS AND A CARBAZOLE ALKALOID FROM THE ROOT BARK OF *CLAUSENA HARMANDIANA*

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Clausena harmandiana Pierre (Rutaceae) is a reputed folk medicine, decoctions of the roots being used as a stomachic and antipyretic. The root bark of this species has yielded five known coumarins and a carbazole alkaloid, which are reported for the first time from this species.

EXPERIMENTAL

GENERAL EXPERIMENTAL PROCEDURES.—Spectral data were obtained with the following instruments: a Perkin-Elmer 283 grating infrared spectrophotometer; a JEOL FX90Q (90 MHz nmr spectrometer); a Shimadzu UV-180 spectrophotometer; and a JEOL DX 300 mass spectrometer.

PLANT MATERIAL.—Root bark was collected in the Kalsinth province in the northeast of Thailand in April 1982. Voucher herbarium specimens of the plant were identified and deposited at the Botany Section, Botany and Weed Science Division, Department of Agriculture, Ministry of Agriculture and Cooperatives, Bangken, Bangkok, Thailand.

EXTRACTION, ISOLATION, AND IDENTIFICATION.—The pulverized, dried root bark (2.8 kg) was extracted by refluxing with 20 liters of hexane for 17 h. The hexane extract was decanted and evaporated on a rotary evaporatory to give a gummy residue (56 g). Analytical tlc (silica gel, petroleum ether-Et_2O, 10:2) allowed several compounds to be detected, which gave color reactions indicative of coumarins and/or alkaloids.

A portion of gummy residue (5 g) was chromatographed on a 2.5×37 cm column containing 60 g of silica gel G 60 (230-400 mesh). The column was initially eluted with petroleum ether-Et_2O (10:2), and 43 fractions (F_1-F_{43}), of 25 ml each, were collected; elution with Et_2O produced fractions F_{44}-F_{54} of the same volume. After tlc analysis, the fractions which contained major amounts of a single compound were combined and concentrated to dryness in vacuo. Crystallization of the purified compounds was effected using Et_2O and MeOH.

The crystallized compounds were identified by spectral data (uv, ir, ^{1}H-nmr, and eims) and by comparisons with authentic samples. Fractions F_{12}-F_{17} yielded heptaphylline (yellow crystals, 39.6 mg) 0.016% yield, mp 171-172° (1); F_{18}-F_{20} yielded clausarin (71.7 mg) 0.029%, mp 198-202° (2); F_{22}-H_{29} yielded dentatin (600 mg) 0.24%, mp 95° (3); F_{39}-F_{41} yielded osthol (150 mg) 0.061%, mp 78-81° (4); F_{46} yielded xanthoxyletin (300 mg) 0.12% yield, mp 132-124° (5), and F_{47}-F_{48} yielded nordentatin (56 mg) 0.023%, mp 183-186° (3). *Clausena excavata* Burm. f. (*Clausena lunulata* Hayata) has recently yielded some of the same compounds (6). The spectral data are available upon request to the senior author.

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ISOLATION OF SOLASODINE FROM THE FRUITS OF *SOLANUM ASPERUM* AND *SOLANUM PALUDOSUM*

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Solasodine, an important starting material for the partial synthesis of many useful steroidal hormones, has been encountered in many species of *Solanum* (1). We wish to report here the isolation of solasodine from the unripe fruits of *Solanum asperum* Vahl var. *angustifolium* and *Solanum paludosum* Moric, two of the yet uninvestigated species growing abundantly in the coastal plain of northeastern Brazil. While the total crude glycoalkaloid fraction from the fruits of *S. paludosum* upon hydrolysis furnished pure solasodine in 0.67% yield, that from *S. asperum* was found to be a mixture of several alkaloids, with solasodine as the major constituent (0.24%).

EXPERIMENTAL

PLANT MATERIAL.—The fruits of *S. asperum* were collected in January 1983, from an area 50 km due west from João Pessoa and the fruits of *S. paludosum* were collected from the campus of the Universidade