EXPERIMENTAL

All the melting points are uncorrected.

Friedelin. $C_{30}H_{50}O$. Found: m.p. 255°, C, 84·1; H, 11·66%; M$^+$ 426; 2,4-dinitrophenyl hydrazone $C_{36}H_{54}O_4N_4$, m.p. 297°; N, 9·40; IR and NMR were identical with those of the authentic samples; co-chromatography. Required: m.p. 255–261°; C, 84·5; H, 11·73%; M$^+$ 426; 2,4-dinitrophenyl hydrazone, m.p. 297–299°; N, 9·24%.[2]

Lignoceryl alcohol. $C_{24}H_{50}O$. Found: m.p. 75°; C, 80·96; H, 14·26%; acetate, m.p. 55°; M$^+$ 396; oxidation with CrO_3–AcOH gave lignoceric acid, m.p. 78°. Required: m.p. 77·5°; C, 81·35; H, 14·12% acetate, m.p. 57°; M$^+$ 396; lignoceric acid m.p. 83·5°.

Ferulic acid. $C_{10}H_{10}O_4$. Found: m.p. 171°, λ_{max} 222, 240, 300, 327 nm; C, 61·52; H, 5·38; acetate, m.p. 189°; IR, NMR, UV identical with those of synthetic sample; co-chromatography; mixed m.p. undepressed. Required: m.p. 173°; C, 61·85; H, 5·15; acetate m.p. 196°.

Lignoceryl ferulate. $C_{34}H_{58}O_4$. Found: m.p. 77°; ν_{max} 1725 cm^{-1}; λ_{max} 222, 240, 300, 327 nm; C, 77·26; H, 10·65. Required: C, 76·98; H, 10·94; Saponification with alkali gave an acid m.p. 171·5, λ_{max} 222, 240, 300, 327 nm and an alcohol m.p. 74°, which were identified as ferulic acid and lignoceryl alcohol respectively by comparison of their physical data with those of the authentic samples.

Canophyllal. $C_{30}H_{48}O_2$. Found: m.p. 266°; ν_{max} 2680 (C—H stretching of CHO), 1725, 1715 (C=O), cm^{-1}; NMR (ppm), 9·63 (S,CHO), 1·08, 0·98, 0·95, 0·858, 0·72, 0·686, (S, 6 × CH_3), 0·88 (d, J = 6 c/s CH_3); C, 81·48; H, 11·18%. Required: m.p. 263–265°; C, 81·81; H, 10·91%.[3]

Canophyllol. $C_{30}H_{50}O_2$. Found: m.p. 280°, ν_{max} 1700 cm^{-1}; NMR (ppm) 3·68 (S, CH_2O—), 1·15, 0·93, 0·88, 0·73 (S, 4 × CH_3) 1·0 (S, 2 × CH_3), 0·88 (d, J = 6 c/s, CH_3); C, 80·98, H, 11·24%; IR identical with that of authentic sample; co-chromatography. Required: m.p. 280–282°; C, 81·31; H, 11·38%.[3]

Maslinic acid. $C_{30}H_{48}O_4$. Found: m.p. 272°; ν_{max} 1700 cm^{-1}; C, 75·84; H, 10·44%; acetate, m.p. 237–240°; methyl ester, m.p. 230°, ν_{max} 1725 cm^{-1}; NMR (ppm) 0·716, 0·83, 0·966, 1·2, 1·3 (S, 5 × CH_3), 0·916 (S, 2 × CH_3), 2·98 (d, J = 11 c/s, —CH—O—), 3·63 (S, OCH_3), 3·63 (m, —CH—O—), 5·3 (m, olefinic H), methyl ester acetate, NMR (ppm), 0·7, 1·03, 1·10, 1·23 (S, 4 × CH_3), 0·883, (S, 3 × CH_3), 1·95, 2·016 (2 × 0—$COCH_3$), 3·6 (S, OCH_3), 4·71 (d, J = 11 c/s, CH–OAc), 5·1 (t of d's J = 11, 11, 5 c/s, CHOAc), 5·25 (m, olefinic H). The splitting pattern of the two methine protons (CHOAc) was, therefore, consistent with the disposition of the two hydroxyls as 2α, 3β. Required: m.p. 267–269°, C, 76·27, H, 10·169%; methyl ester, m.p. 227–228°, acetate m.p. 235–239°.[4]

Acknowledgements—The authors are grateful to Mr. J. Saran and his associates for microanalyses and to Prof. T. R. Govindachari for supplying a sample of canophyllol.

[2] J. SIMONSEN and W. C. J. ROSS, *The Terpenes*, Vol. IV, p. 468, The University Press, Cambridge (1957).

[3] T. R. GOVINDACHARI, N. VISHWANATHAN, B. R. PAI, U. A. RAO and M. SRINIVASAN, *Tetrahedron* **23**, 1901 (1967).

[4] L. CAGLIOTI, G. CAINELLI and F. MINUTILLI, *Gazz. Chim. Ital.* **91**, 1387 (1961).

Phytochemistry, 1971, Vol. 10, pp. 2832 to 2833. Pergamon Press. Printed in England.

HUMIRIACEAE

AN ISOCOUMARIN FROM THE BARK OF *SACOGLOTTIS GABONENSIS**

A. U. OGAN

Department of Biochemistry, Division of Biological Sciences,
University of Nigeria, Nsukka, Nigeria

(*Received* 15 *March* 1971)

Sacoglottis gabonensis bark is widely used in West Africa as an additive to palm-wine, the major alcoholic beverage drunk by people in the tropical forest regions of West Africa.[1]

* Part VI of the series "Studies on West African Medicinal Plants". For Part V see A. U. OGAN, *Phytochem.* **10**, 2823 (1971).

[1] J. M. DALZIEL, *The Useful Plants of West Tropical Africa*, p. 134, Crown Agents, London (1937).

It is generally believed that the bark retards the tendency of the palm-wine to sour during fermentation. Its use is particularly important therefore when the fermentation period has to be purposely prolonged—as is the case when the intention is to distil the palm-wine for the production of home-made gin.

The chemical study of *S. gabonensis* bark seemed worthwhile on the grounds that the active principle might prove to be a specific inhibitor for the alcohol dehydrogenase system of yeast. It was hoped that the discovery of this principle might also prove useful in connection with local efforts towards the industrialization of palm-wine production and the large-scale manufacture of refined alcohol from it.

Soxhlet extraction of air-dried and powdered bark first with light petroleum (60°–80°) and then with chloroform yielded traces of oils and gums respectively, neither of which contained any alkaloids. Further extraction with methanol also gave no alkaloids. The residue from methanol was digested with hot water and filtered free from a water-insoluble purple dye (attempts to obtain the dye in crystalline form proved unsuccessful). The filtrate was passed through a column of activated charcoal and the column washed exhaustively first with water to remove inorganic salts and then with methanol. Evaporation of the methanol left a white residue which recrystallized from methanol–ethanol mixture as clusters of white, pyramidal crystals (0·05%), m.p. 145° $[\alpha]_D^{21}$ $-39{\cdot}8°$ (C 2·4 in methanol).

The substance gave a violet colour reaction with ferric chloride reagent and a slow positive reaction with Tollen's reagent. Its molecular weight, as determined by accurate mass measurement (by Dr. John Wilson of the Organic Chemistry Department of the University of Manchester, using an A.E.I. Mass Spectrometer model MS9), was $C_{14}H_{16}O_9$. Zeisel determination indicated the presence of one oxymethyl group. The substance had IR absorption max. (in KBr disc) at 3350, 3200, 1700, 1610 cm^{-1} and a series of six bands at 1040–1140 cm^{-1} with the strongest max. at 1090 cm^{-1}. It had UV absorption max. in methanol 225 nm (ϵ 14,610) as well as a symmetrical max. 275 nm (ϵ 6700) with a broad shoulder at about 310 nm (ϵ 3980). When a drop of alkali was added to the cell the UV max. at 275 nm underwent a marked bathochromic shift to give a broad plateau at 295–350 nm (ϵ 2245). When the UV spectrum was repeated with a few crystals of $NaBH_4$ added to the cell the max. at 275 nm gradually disappeared completely—indicating that it was due to an α,β-unsaturated carbonyl group.

The above chemical and spectral evidence suggest that the compound is an iso-coumarin, namely the rare *C*-glucopyranosyl compound bergenin (i.e. the lactone of 2β-D-glucopyranosyl-4-*O*-methylgallic acid[2,3]). This was supported by nuclear magnetic resonance and mass spectral evidence, and by the finding that, like bergenin, the substance gave a penta-acetoxy derivative m.p. 205°–207°, which had IR absorption max. 1776, 1739 cm^{-1} and UV absorption max. 256, 210 nm (ϵ 16,400 and 14,830 respectively). Of some interest with respect to the IR spectra is the unusually low δ-lactone carbonyl frequency of the parent compound, which was not the case with the penta-acetate. Anomalously low lactone carbonyl frequencies feature in the IR spectra of some iso-coumarins;[2] and the features observed here for the *Sacoglottis* compound are identical with findings of Hay and Haynes for bergenin and its penta-acetate.[2]

The effect of the *Sacoglottis* compound on yeast enzymes is currently under study.

---

[2] J. E. HAY and L. J. HAYNES, *J. Chem. Soc.* 2231 (1958).

[3] HIRAYA SHIMADA, TOKUNOSUKE SAWADA and SHINZO FUKUDA, *J. Pharm. Soc. Japan* **72**, 578 (1952); *Chem. Abs.* **46**, 8811 (1952).