TERPENIC CONSTITUENTS FROM *ICACINA SENEGALENSIS*

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Icacina senegalensis A. Juss. (Icacinaceae) is a shrub endemic to Casamance (Senegal); the tuber is exceptionally large. The use of *I. senegalensis* in popular medicine has been described (1,2); during periods of famine the tuber has been used as a starch source but has been found to be toxic in some cases (2). No report on the chemical constituents of *I. senegalensis* has been published until now, but previous work carried out on other *Icacina* spp. has resulted in the isolation of five diterpene lactones related to the pimarane skeleton, three of them being alkaloids (3-6).

Standard procedures for the isolation of alkaloids afforded no alkaloids from either leaves or tuber. Using hplc, gc, tlc, and uv spectrometry, it was possible to identify the pimarane lactones icacinol (5) and icacenone (6) in a purified Me_2CO/EtOH extract of the tuber. Among the polar constituents of the same extract, two steryl glucosides, sitosterol 3-*O*-β-D-glucopyranoside and stigmasterol 3-*O*-β-D-glucopyranoside were isolated and identified by ^{1}H-nmr spectroscopy and by gc after hydrolysis.

EXPERIMENTAL

PLANT MATERIAL.—The plant was collected around Kaolach (Senegal) in May 1985, and identified at the Botanical Institute of the University of Dakkar. A voucher specimen is preserved at this Institute.

ISOLATION AND IDENTIFICATION.—The dried, powdered tuber was first defatted by hexane, then extracted by Me_2CO-EtOH (1:1); the crude extract (8 g) was then fractionated on a short column of silica gel using $CHCl_3$ containing increasing amounts of MeOH and H_2O as solvents; further purification of the fractions eluted with $CHCl_3$-MeOH-H_2O (97.5:2.5:0.5) was performed by preparative tlc (silica gel, $CHCl_3$-MeOH-H_2O 46:5:0.5). Icacenone (0.08% yield) was identified by uv spectroscopy, by tlc on silica gel (solvent: $CHCl_3$-MeOH-H_2O, 46:5:0.5) and on octadecyl silica gel (solvent: EtOH-H_2O, 65:35), and by hplc on a RP-18 column (Lichrosorb 5μ, solvent: MeOH-H_2O, 3:7 at a flow rate of 1 ml/min, T_R=7 min) using an authentic sample as standard (6). Icacinol (0.03% yield) was detected by tlc (solvent: $CHCl_3$-MeOH-H_2O, 46:5:0.5; specific detection by spraying a 5% H_2SO_4 solution in EtOH and heating at 120° for 5 min which gave a bright yellow fluorescent spot with icacinol) and by gc (1 m glass column filled with chromosorb coated with 3%SE-30 and maintained at 230°, He flow rate 30 ml/min, Fl detector, T_R=6 min) using a sample as standard (5).

Finally, from the polar fraction eluted from the silica gel column with $CHCl_3$-MeOH-H_2O (95:5:1), a mixture of two steryl glucosides was recovered; it was further purified by preparative tlc on silica gel (solvent: $CHCl_3$-MeOH-H_2O, 45:5:0.5) (yield 0.1%). After acid hydrolysis, a mixture of β-sitosterol (55%) and stigmasterol (45%) was identified by gc (t_R) which allowed also the identification of glucopyranose as its trimethylsilylderivative. ^{1}H-nmr spectra of the mixture of the two peracetylated glucosides recorded in $CDCl_3$, showed a doublet at δ 4.60 (J=7.9 Hz) confirming that the sugar was β-linked to the aglycones and other signals which supported the gc identification of the aglycones.

Full details of the isolation and identification of the compounds are available on request to the authors.

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