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PHYCOPSISENONE, A NEW PHENOLIC SECONDARY METABOLITE FROM THE SPONGE *PHYCOPSIS* SP.[1]

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ABSTRACT.—Crotonic acid, phenylacetic acid, 4-hydroxyphenylacetic acid, methyl 4-hydroxyphenylacetate, 4-hydroxybenzaldehyde, 4-isobutyl-α-methylbenzyl alcohol [**1**], and a new phenolic derivative, phycopsisenone [**2**], have been isolated from the sponge *Phycopsis* sp., and characterized by interpretation of spectral data.

Numerous prenylated aromatic compounds have been isolated from marine flora and fauna (1). Particularly large numbers of these compounds have been reported from brown algae (2). For example, a bis-prenylated phenol, 2,4-*bis*-(3-methylbutenyl)-phenol, isolated from the brown algae *Encyothalia cliftonii* (3) and *Penithalia caudata* (4), showed significant feeding deterrence towards the herbivorous sea urchin *Tripneustes esculentus*. Two aromatic sesquiterpenes, curcuphenol and dehydrocurcuphenol, isolated from the sponge *Epipolasis* sp. (5), exhibited strong inhibitory activity against H^+ and K^+ ATPase. In search of biologically active secondary metabolites from marine invertebrates, we investigated a sponge, *Phycopsis* sp., collected from the Tuticorin coast, Tamilnadu, India, during April 1993. A 1:1 CH_2Cl_2-MeOH extract of this organism exhibited antibacterial activity against *E. coli* and *B. subtilis*.

The CH_2Cl_2-MeOH (1:1) extract of the sponge was subjected to gel filtration chromatography followed by Si gel cc, resulting in the isolation of crotonic acid (50 mg), phenylacetic acid (150 mg), 4-hydroxyphenylacetic acid (150 mg), methyl 4-hydroxyphenylacetate (25 mg), 4-hydroxybenzaldehyde (50 mg), 4-isobutyl-α-methylbenzyl alcohol [**1**] (40 mg), and phycopsisenone [**2**] (50 mg).

Compound **1** was obtained as an oil, $[\alpha]_D$ −0.9° (c=1, $CHCl_3$), and identified as 4-isobutyl-α-methylbenzyl alcohol by the study of its ^{1}H-nmr and mass spectral data. Compound **1** is a key intermediate in the synthesis of ibuprofen (6), an antiinflammatory drug. To establish the stereochemistry of the secondary alcohol in **1**, its (*R*)-MTPA ester was prepared (7). The ^{1}H-nmr spectrum of the (*R*)-MTPA-ester of **1** was consistent with the presence of a diastereomeric mixture, composed of the (*S*)- and (*R*)- isomers in a 3:2 ratio. The small negative optical rotation observed for compound **1** compared with that of the synthetic *R*(+)- isomer (8) was attributed to the predominance of the (*S*)- isomer in the isolated sample.

Phycopsisenone [**2**] was obtained as a crystalline solid, which analyzed for $C_{13}H_{16}O_3$ by combustion analysis. A methanolic solution of **2** showed a coloration with $FeCl_3$, implying the phenolic nature of the compound. The ir bands at 3450, 1680, 1640, 1560 cm^{-1} indicated the presence of hydroxyl and α,β-unsaturated carbonyl groups. The ^{1}H-nmr spectrum of compound **2** contained signals for two Me groups attached to an oxygen-bearing carbon at δ 1.3 (6H, s), an uncoupled methylene group at δ 2.8 (2H, s), and a para-disubstituted benzene ring with resonances at δ 6.9 (2H, d, J=7 Hz) and 7.5 (2H, d, J=7 Hz). Furthermore,

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