Isolation of meso-1,3-Diphenyl-1,3-propanediol from a Soft Coral Nephthea Sp.

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ISOLATION OF *meso*-1,3-DIPHENYL-1,3-PROPANEDIOL FROM A SOFT CORAL *NEPHTHEA* SP.

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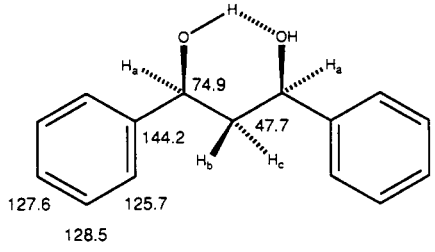
ABSTRACT.—*meso*-1,3-Diphenyl-1,3-propanediol has been isolated from a soft coral *Nephthea* sp. It has been characterized by spectral data and synthesis.

In connection with our chemical investigations on marine species collected from the Indian Ocean, we report here the isolation of *meso*-1,3-diphenyl-1,3-propanediol [**1**].

The ir spectrum of **1** indicated the presence of hydroxyl group(s) and monosubstituted phenyl ring(s). The ^{1}H-nmr spectrum of **1** showed signals at δ 7.20–7.45 (10H, m) for two monosubstituted benzene rings, the presence of which was also supported by the uv spectrum. A signal at δ 3.67 (2H, s, disappeared on D_2O exchange) was assigned to two hydroxyl groups. The signals for the carbinyl protons appeared as two doublets at δ 4.94 (2H, J = 10.1 Hz and 2.8 Hz, H_a) and those for two nonequivalent methylene protons appeared at δ 2.14 (1H, ddd, J_{gem} = 14.6 Hz, J_{vic} = 10.1 Hz and 10.1 Hz, H_b) and δ 1.91 (1H, ddd, J_{gem} = 14.6 Hz, J_{vic} = 2.8 Hz and 2.8 Hz, H_c). The above conclusion was drawn on the basis of chemical shifts, homodecoupling, and PANIC (nmr simulation) experiments. The ^{13}C-nmr spectrum of **1** was also in perfect conformity with the structure assigned. The multiplicities were determined by a DEPT experiment. The carbon shift assignments are given around structure **1**. The mass spectrum indicated a mol wt of 228 daltons and the observed fragments were consistent with the proposed structure. As the compound did not show any optical activity it should have the relative configuration *R*,*S*, which is evident from the ^{1}H coupling pattern observed.

The compound **1** was finally characterized as *meso*-1,3-diphenyl-1,3-propanediol by direct comparison (mixed mp, tlc, and ir) with an authentic sample prepared by following the method of Dale (1). The synthesis of *meso*-1,3-diphenyl-1,3-propanediol was reported by several workers earlier (1–3).

That the compound **1** is not an artifact was confirmed by tlc comparison of the crude extract with the pure compound **1**. The extremely low yield (5 mg from 3 kg of the soft coral) was due to the



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loss incurred during the process of isolation and repeated crystallization from solvents.

This is the first report of the isolation of **1** from a natural source.

EXPERIMENTAL

GENERAL EXPERIMENTAL PROCEDURES.—The ir spectrum (KBr) was recorded on a Shimadzu IR-408 instrument. The uv spectrum (MeOH) was recorded on a Shimadzu UV-160 instrument. ^{1}H-nmr, ^{13}C-nmr, and 2D nmr spectra, with TMS as internal standard, were recorded on a Bruker AM 300L instrument in $CDCl_3$. The mass spectrum was recorded on an AEI MS-30 instrument. For cc and tlc, Si gel (60–120 mesh, Glaxo, India) and Si gel G (Merck, India), respectively, were used. Melting point was determined with an electrothermal apparatus and is uncorrected.

CORAL COLLECTION AND TAXONOMY.—The *Nephthea* sp. was collected from the coastal sea (Bay of Bengal) of Orissa which is about 615 km from Calcutta, in September 1987. This *Nephthea* sp. was characterized by Zoological Survey of India. Calcutta up to the generic level by comparison with type specimens. Classification: Phylum Cnidaria, Class Anthozoa, Order Alcyonacea, Family Nephthyidae. This is an undescribed Indian coral which resembles other *Nephthea* sp. It has a cylindrical stalk, from the lateral surface of which arise the polyp-bearing lobes which also bear smaller outgrowths covered with polyps. It is gray-ruby in color, with central portion lighter than the polyps; the polyps are short and bear some angles with the stalk in the preserved state; the spicules are spiny and warty spindles, straight or curved.

A specimen of the above coral has been deposited with Z.S.I., Calcutta, Registration No. P-3262/1.

ISOLATION AND IDENTIFICATION OF *meso*-1,3-DIPHENYL-1,3-PROPANEDIOL [**1**].—The corals (3 kg) were homogenized with $CHCl_3$-MeOH (2:1) (10 liters), and the extract was concentrated under reduced pressure at room temperature. The viscous residue (9.5 g) was chromatographed over Si gel. The C_6H_6-$CHCl_3$ (7:3) eluate yielded a yellowish oily residue which after preparative tlc [C_6H_6-$CHCl_3$-MeOH (3:1:0.25), 0.35 mm; detection I_2; R_f 0.48] furnished the compound **1**, mp 104° [repeated crystallization from Et_2O (5%)/petroleum ether]; yield 5 mg (0.00017%); $[\alpha]^{20}_D$ 0° (c = 0.26, $CHCl_3$); uv λ max (MeOH) (ε) 218 (1055), 240 (173), 246 (238), 252 (303), 258 (350), 263 (266) nm; ir ν max (KBr) 3440–3340, 1500, 1460, 1408, 1065, 1035, 1023, 935, 754, 700, 665, cm^{-1}, eims *m/z* (rel. int.) $[M]^+$ 228 (55), $[M-H_2O]^+$ 210 (45), $[M-PhCHOH]^+$ 121 (60), $[PhCHO]^+$ 106 (90), $[Ph\text{-}C{\equiv}O]^+$ 105 (100), $[Ph]^+$ 77 (66).

PREPARATION OF *meso*-1,3-DIPHENYL-1,3-PROPANEDIOL.—To an MeOH solution of dibenzoyl methane (0.5 g) was added MeOH solution of $NaBH_4$ (0.6 g) and NaOH (0.004 g). The solution was refluxed for 2 h and worked up following the method of Dale (1): mp 106°; yield 200 mg.

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