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DOI: <http://dx.doi.org/10.18535/ijetst/v3i06.03>**A New unsaturated Ketone and other constituents from *Dioscorea bulbifera***

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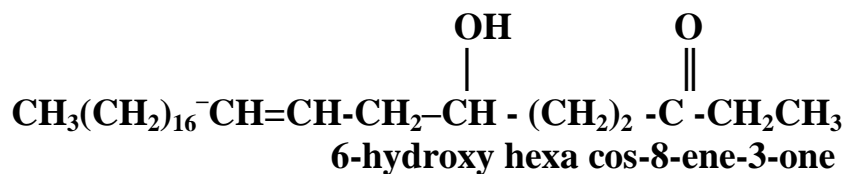
Dioscorea bulbifera (Fam. Dioscoreaceae) is a climber plant with tuberous root is found in tropical part of the world and grows throughout India. Tuber is used by the tribal population of central India as a food particularly in Madhya Pradesh, Chhattisgarh, Jharkhand and Orissa. Plant is anthelmintic, aphrodisiac. Cooling, diuretic, Sweet and tonic. It is also used in the treatment of gonorrhoea, helminthiasis and leprosy². It is used in the treatment of rheumatism arthritis.³ They are also used in heart disease. Phytochemical investigations on this plant species have revealed the isolation of β -sitosterol and aliphatic unsaturated Ketone (Compound I) and Saturated Aliphatic ester both these compounds are new and not reported earlier.

Compound I was isolated as yellowish white compound m.p. 72°C. from the tuberous root on the basis of elemental analysis was found to have molecular formula $C_{26}H_{50}O_2$.

The Compound showed characteristic IR bands as 3413 cm^{-1} for hydroxyl function, at 2971 and 1631 cm^{-1} for decibel band and at 1710 cm^{-1} for presence of carbonyl group⁽⁵⁾. The absorption bands at 2918, 2850, 1463, 720 cm^{-1} and the absence of absorption bands in the aromatic region suggest its aliphatic nature. Compound decolorizes $KMnO_4$ and bromine solution confirms the presence of unsaturation in the compounds. It formed hydrazone, m.p. 207°C with 2,4

dinitrophenyl hydrazine confirming the presence of carbonyl group. Its monoacetate derivative showed peaks at 1740 and 1240 cm^{-1} (acetyl group) in IR spectrum while band at 3413 cm^{-1} (for-OH) in the original compound disappeared, showing presence of only one hydroxyl group in the compound⁽⁶⁾.

The ¹H NMR spectrum exhibited signal at δ 5.2 for methine protons (-CH=CH-), and at δ 2.05 (m, 4H) for four methylene protons adjacent to double bond (-CH₂-CH=CH-CH₂-). Appearance of triplet at δ 0.9 (s, 6H) showed the presence of two methyl groups. A broad singlet at δ 1.30 (brs, 32H) showed the presence of sixteen methylene groups. Multiplet at δ 8.90 (m, 1H) was due to the proton attached to carbon bearing hydroxyl group. The mass spectrum of the compound exhibited a molecular ion peak, M at m/z 394. base peak at m/z 29 corresponding to smaller ion $C_2H_5^+$ and another abundant ion at m/z 57 corresponded to acylium ion $C_2H_5CO^+$. Two other less intense peaks at m/z 337 and m/z 365 corresponded to a larger hydroxy olefinic ion ($C_{23}H_{45}O^+$) and a larger acylium hydroxy olefinic ion ($C_{24}H_{45}O^+$) peaks at m/z 309 and 279 showed the presence of hydroxyl group at C-6. The peaks at m/z 278 and m/z 169 indicated the presence of double bond at C-6. Peak at m/z in due to presence of moiety $CH_3(CH_2)_{15}$. Above discussion compound (1) assigned as



Experimental Section

Melting Points are uncorrected. IR spectra were recorded as (as KBr pellets) on a Perkin-Elmer model 557 spectro photometer. ¹H NMR and ¹³C NMR spectra on model jeol Ax900 at 89.55 MHz and 22.49 MHz respectively using CDCl₃ and DMSO-d₆ as solvents and TMS as an internal standard.

Column chromatography was carried out using silica gel (60-120 mesh) TLC was carried out on silica gel G Chromato Plates.

The above observations clearly indicated that compound is unsaturated aliphatic hydroxy ketone having 26 carbon atoms. The exact nature and size of alkyl groups, position of double bonds, hydroxyl group and carbonyl group was ascertained by the study of mass spectrum and oxidation of compound.

Extraction with Petroleum ether

The air dried grounded bulb of *Dioscorea bulbifera* was extracted with petroleum ether (60-80^o) in soxhlet extractor for 30 hrs. The petroleum ether extract was concentrated under reduced pressure. The Viscous mass was loaded over silica gel 60 column. It was then eluted with solvents Hexane, Benzene and Chloroform in increasing polarity.

In hexane elute, it give compound A m.p. -136^o (100mg). It was found single on TLC and crystallized from hexane as fine and white needles (β-sitosterol).

Further on elution with Hexane, Benzene (8:2v/v) compound m.p. 72^o was obtained and crystallised from Benzene as yellowing white of compound.

Next fraction (c) (150mgs.) mp. 69^o. It was (Compound B) on recrystallized from Hexane.

Compound A recrystallized from benzene soluble in benzene, chloroform. TLC in Hexane: benzene (1:1 V/V) R_f 0.41, IR (KBr) 3413, 2956, 2918,

2850, 1637, 1710, 1473, 1463, 1383, 1089, 874, 803, 730 720 cm, ¹HMR(δPPm), 2.05 (m,4H), 2.24 (m, 4H), 3.40 (m, 1H), 5.2 (t, 2H), J=4Hz. Its Ms (M/z), 394 (Mt), 365, 337, 309, 279, 278, 225, 211, 197, 183, 169, 155, 145, 127, 113, 99, 85, 57, 29. Its monoacetate crystallized from hexane as white powder (m.p. 41^oc) IR (KBr) 2956, 2916, 2850 (C-H) 1740 (C=O), 1710 (C=O), 1637, 1473, 1463, 1383, 1242. Co specking in acetate), 732, 722 cm⁻¹.

EXPERIMENTAL

It was crystallized from hexane.

Solubility	:	Soluble in hexane, benzene, chloroform.
R _f	:	0.41 (T.L.C. on silica gel G plates) Hexane : Benzene (1:1, v/v)
Elemental Analysis	:	Found Calculated for C ₂₀ H ₂₀ O ₂ C : 79.40% C : 79.18% H : 13% H : 12.69%
IR	:	KBr : 3413, 2956, 2918, 2850, 1637, 1710, 1473, max : 1463, 1383, 1089, 874, 803, 730, 720 cm ⁻¹
¹ H NMR	:	(δ . ppm) [CDCl ₃ ,60MHz] 0.9 (6H, t), 1.30 (s, 32H), 2.05 (m, 4H), 2.24 (m, 4H), 3.40 (m, 1H), 5.2 (t, 2H), J = 4Hz
MS : m/z	:	394 (M ⁺), 365, 337, 309, 279, 278, 225, 211, 197, 183, 169, 155, 141, 127, 113, 99, 85, 57, 29.

Acetylation of Compound

Compound (30 mg) dissolved in AC₂O (10 ml) and pyridine (2 ml) was added. The reaction mixture was left for 48 hours at room temperature.. A white product was obtained on pouring the reaction mixture in ice cold water with continuous stirring. It was filtered and washed with water. Then product was crystallised from hexane as white powder (m.p. 41⁰C).

IR
max
KBr : 2956, 2910, 2850 (C-H),
1740 (C=O),
: 1710 (C=O), 1637, 1473,
1463, 1383, 1242
(Co stretching in acetate)
732, 722 cm⁻¹.

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