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Chemical Investigation of Saturated Aliphatic Ester from the Tuber of Dioscorea Bulbifera

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Dioscorea bulbifera (family- Discoreaceae) is found commonly in India. Recent Pharmacological finding indicate that its tubers possess significant activities, rejuvenating and tonic.

Phytochemical investigations of tubers of this plant give saturated aliphastic ester along with other constituent. White compound m.p. 69^0C on the basis of mass fragmentaction patter molecular weight $C_{53}H_{106}O_2$. The compound did not undergo acetylation mor showed any bind in the region above 3200cm^{-1} in IR.

The compoupl did not undergo acetylation nor showed any band in the region above 3200 cm^{-1} in IR spectrum showing the absence of hydroxyl function. Appearance of absorption bands at 1462, 732 and 720 cm^{-1} and the absence of bands in the aromatic region in its IR spectrum, suggested its aliphatic nature. The non reactivity with 2,4 dinitrophenyl hydrazine shows the absence of a carbonyl function. The compound appeared to be an ester as indicated by absorption band at 1732 cm^{-1}. The presence of peaks at 2918 cm-1 and 2848 cm-1 is diagnostic of the streaching vibration of methylene group.

The compound on refluxing with ethanolic alkali for 7 hrs, cleaved into an alcohol and an acid. Alcohol was identified as tetratriacantanol and the acid was identified as nonadecanoic acid by mp, mmp and co—chromatography with their respective authentic samples.

The hydrolysis of ester and thus, its structure can be represented as:

$$CH_3-(CH_2)_{17}-\overset{O}{\overset{\|}{C}}-O-CH_2-(CH_2)_{32}-CH_3 \xrightarrow{10\%\ KOH} CH_3-(CH_2)_{17}-\overset{O}{\overset{\|}{C}}-OH + HO-CH_2-(CH_2)_{32}-CH_3$$

Nonadecanoic acid Tetratriacantanol

The NMR spectrum showed only four types of protons. The presence of a signal at δ4.02 (t,2H) ppm showed the presence of methylene ester functionality. The presence of signal at δ0.84 ppm (6H, C—methyl group) shows that ester contains two methyl groups. The singlet at δ 1.22 ppm (96 H) shows the presence of forty

eight methylene groups. The triplet at δ 2.24 (2H) ppm is due to presence of methylene group adjescent to carbonyl group.

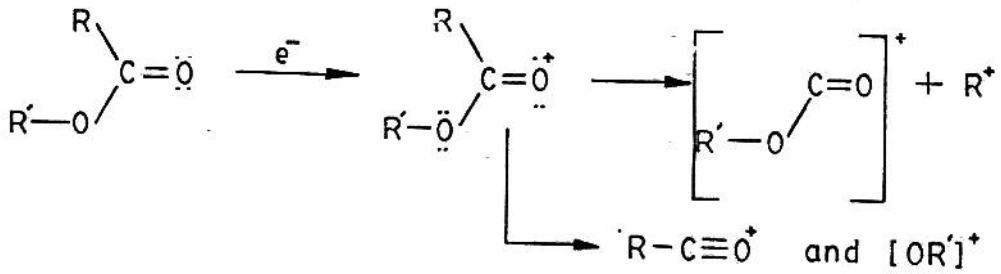
Above facts confirmed that the compound is saturated aliphatic ester having 53 carbon atoms. Thus, the following structure may be assigned to compound.

$$CH_3-(CH_2)_{32}-CH_2-O-\underset{\underset{C}{\|}}{O}-(CH_2)_{17}-CH_3$$

The exact nature and size of alkyl groups attached to the ester was further supported by the study of mass spectrum.

Mass Spectral Studies of Ester

It is well known that fragmentation peaks observed in mass spectrum of aliphatic esters results from C—C bond clevage, adjescent to carbonyl oxygen atom, where upon the charge remains on the oxygenated fragment as shown below[18,19]. :



The ion $R-C\equiv O^+$ gives an excellent diagnostic peak for on ester, so t he mass fragmentation would have occurred as

The peaks at m/z 153, 139, 125, 111 and 97 is due to loss of hydrogen molecule[22] from the alkyl fragment at m/z 155$[CH_3(CH_2)_{10}]^+$, 141$[CH_3(CH_2)_9]^+$, 127$[CH_3(CH_2)_8]^+$, 113$[CH_3(CH_2)_7]^+$ and 99 $[CH_3(CH_2)_6]^+$.

$$[CH_3(CH_2)_{10}]^+ \xrightarrow{-H_2} [C_{11}H_{21}]^+$$
m/z 155 → m/z 153

$$[C_{10}H_{21}]^+ \xrightarrow{-H_2} [C_{10}H_{19}]^+$$
m/z 141 → m/z139

$$[C_9H_{19}]^+ \xrightarrow{-H_2} [C_9H_{17}]^+$$
m/z 127 → m/z 125

$$[C_8H_{17}]^+ \xrightarrow{-H_2} [C_8H_{15}]^+$$
m/z 113 → m/z 111

$$[C_7H_{15}]^+ \xrightarrow{-H_2} [C_7H_{13}]^+$$
m/z 99 → m/z 97

From the above discussion, it is concluded that ester may be assigned the following structure.

$$CH_3-(CH_2)_{17}-\underset{\underset{C}{\|}}{O}-O-CH_2-(CH_2)_{32}-CH_3$$

EXPERIMENTAL

Found	Calculated for $C_{53}H_{106}0_2$
C = 82.00%	C = 82.17%
H = 13.60%	H = 13.69%

Solubility : Hexane, Benzene, Chloroform.

IR Spectrum

IR ν_{max}^{KBr} : 2918, 2848, 1732, 1462, 1462, 732 and 720 cm^{-1}

NMR Spectrum in $CDCI_3$ (90MHz)

Chemical Shifts (ppm)	**Assignment**
4.02 (t, 2H)	$CH_2-O-\overset{\overset{O}{\|}}{C}-$
2.24 (t, 2H)	$-CH_2-\overset{\overset{O}{\|}}{C}-$
1.22 (s, 96H)	$-CH_2-$
0.84 (s, 6H)	$-CH_3$

Mas spectrum – (70 ev, direct inlet)

774 [M+], 746, 690, 558, 502, 487, 475, 465, 412, 398, 370, 342, 313, 299, 295, 281, 267, 253, 239, 225, 211, 197, 153, 153, 139, 125, 111, 97, 85, 71, 57, 43, 29.

Hydrolysis of Compound A

100 mg of compound was refluxed in 10ml of 7%. alcoholic KOH for 7 hrs. The alcohol was distilled at reduced pressure and contents were poured into 100 ml of distilled water when a white ppt was obtained. The ppt was filtered and crystallized from benzene, m.p. 65°C.

The filtrate was neutralized with dil. HCL and extracted with ether. Extract was washed with distilled water and dried over anhydrous No_2SO_4. On distilling off the ether, the acid was obtained in pure crystallized form, m.p. 66°C.

REFERENCES

1. Chopra, R.N. Nayar SL & Chopra I.C., Glossary of Indian Medical Plants CSIR (New Delhi), 1971.
2. Subhasni, U. Dubey G.P., International Journal of Pharmacy & Life Science, 2013 4 (5).
3. Sharma, N. Vijay Vergia, R., International J. Pharma Bio Sci. 4(2) 581-586, 2013.
4. Chandra Subhash, Sarla, S., International Research Journal of Pharmacy 2012 3(5).
5. Thorat, A. Jadhav, V., International Research Journal of J. Pharmacy 1(1) 77-80, 2010.
6. Ahmad, S., International & Pharnacognosg. I.K. International Publishing House Pvt. Ltd., New Delhi- 2012, 382-386.
7. Kokate C. K. Prohit, A.P. Gokhale, S.B., Pharmacognosy, Nirali Prakashan Pune 2003, 1-62.
8. Prajapati, N.D. Purohit S.S. Sharma, A.K. and Kumar, T.A., Handbook of Medicinal Plants Agrobios Jodhpur 92, 2003.