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Chemical Constituents from the Base Leaves of Caryota Urens (Palmae)

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Abstract: *Triterpenes named as Lupeol, Myricadiol, β-sitosterol along with two carboxylic acids named as Tetracosonid and Ursolic acid, have been isolated from leaves of Caryota Urens. Their structures were elucidated by chemical and physical data (IR, UV, ^{1}H-NMR, and Mass spectra).*
Keywords*: Caryota Urens; triterpenes, carboxylic acids*

I. Introduction

The genus **Caryota** comprises 15 species distributed in the tropical parts of India, Burma, Ceylon, Malaysia and Northern Australia. Out of these, three species are reported in India[1] of which **C. urens** is of economic importance.

Caryota species have been reported for their medicinal properties such as internally nutritious and aphrodisiac and also laxative.[2] Earlier investigations on this plant reported the isolation of Amino acids, sugars, ascorbic acid[3], fatty acids, Kernel lipids[4] and sugarsin.[5]

Medicinal importance and scanty work on this plant accelerated our interest to carryout the comprehensive study of the plant **Caryota urens**. The present discussion deals with the isolation and characterization of following compounds. From base leaves of **Caryota urens**.

1. **Triacontane**
2. **Lupeol**
3. **Myricadiol**
4. **β-sitosterol.**
5. **Tetracosonid**
6. **Ursolic acid**

II. Material and Methods

The defatted base of leaves of **Caryota urens** (2 Kg) procured from fort of A.M.U., Aligarh, India were extracted exhaustively with petroleum ether ($60\text{-}80^0$) and benzene. The base leaves were then extracted with methanol (3 liters x 3) at room temperature and finally on a steam bath.

The petroleum ether and benzene extracts of the base of leaves were divided into neutral (I) and the acidic (ii) parts by treatment with alkali. Chromatographic resolution of the neutral part gave four products **C_Y-1, C_Y-2, C_Y-3, C_Y-4**. The chromatographic resolution of alkali soluble part (ii) over alummina column gave two compounds **C_Y-5** and **C_Y-6**.

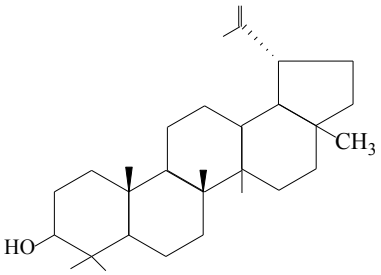
III. Result and Discussion

Compound C_Y-1:

Hexane yielded **C_Y-1** m.p. $62\text{-}67^0$C. It was found to be identical with **triacontane** on the basis of its elemental analysis ($C_{30}H_{62}$) and **infrared** spectrum, υ^{kBr}_{max} 2930 and 2860 cm^{-1} (C-H, saturated), 1460 and 1380 cm^{-1} (C-CH_3) and 720 cm^{-1} $(CH_2)_n$. Finally, it was analysed by **gas liquid chromatography** which indicated **C_Y-1** to be a mixture of n-alkanes of series C_{24}-C_{36}, containing mainly n-tri-triacontane (28.9%), n-nonacosane (21.2%), n-triacontane (2.9%) accompanied by hexacosane, pentacosane and pentatriacontane as minor consit-ituents[6].

Compound C_Y-2*:*

Petroleum ether-benzene (1:4) solution from the neutral part afforded **C_Y-2**, melting point $214\text{-}15^0$C, $[\alpha]^{20}_D$ + 23.64 ($CHCl_3$). It gave positive Liebermann-Burchard[7] and Noller's[8] tests and yellow colour with tetranitromethane. Elemental analysis agreed with the formula $C_{30}H_{50}O$. **Infrared** showed bands at 3360 and



(I)

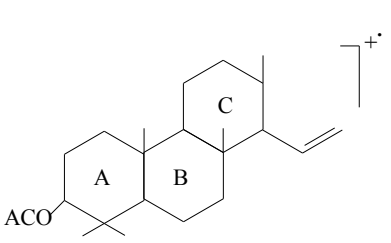
1030 cm^{-1} (OH), 1645 cm^{-1} (C=C) and 1385 cm^{-1} (geminal dimethyl), 885 cm^{-1} (terminal methylene). **Mass** spectrum of the triterpene alcohol gave $M^{+\bullet}$ at m/z 426 (11%) with other principal ions m/z 411 / (M-CH_3) (6%), 207 (34%), 189 (77%), and a base peak at m/z 95. It afforded an acetate m.p. 218-220^{0}C. **Infrared** spectrum of the acetate revealed the presence of terminal methylene, by a band at 875 cm^{-1}. Some other important bands were observed at 1245 cm^{-1} (acetate), 1640 cm^{-1} (C=C) and 1730 cm^{-1} (C=O). **^{1}H-NMR** spectrum gave the signals at δ 0.82, 0.87, 0.94, 1.04 (CH_3 protons), 1.27, 1.41, 1.46, 1.470 (CH_2 protons), 2.03 ($OCOCH_3$) and multiplets at 4.28 and 4.77 (>C CHOAc), 4.59, 4.67 (>C=CH_2). On the basis of the above physico-chemical data of the compound and its derivatives the **C_Y-2** was identified as **lupeol**[9] **(I)**.

Compound C_Y-3:

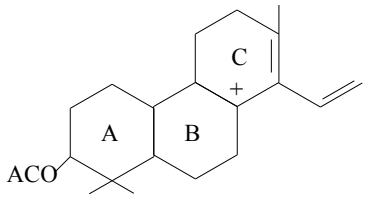
Elution of the column with benzene followed by crystallization from benzene-ethylacetate gave a colourless amorphous compound **(C_Y-3)**, m.p. 259-60^{0}C. It gave positive Liebermann-Burchard test[7] showing it to be a triterpene which was confirmed by stannic chloride test[8]. Its **ir.** Spectrum, (KBr) showed the bands at 3415 (OH), 2940 (unsaturation), 2880, 1470, 1450, 1390, 1380 (charac-teristic of triterpenic skeleton), 1080, 1030 (C-O stretching and O-H in plane deformation of secondary alcohol) and 815 cm^{-1}.

C_Y-3 on **mass** spectrometric analysis showed a molecular ion peak at m/z 442 revealing its molecular formula to be $C_{30}H_{50}O_2$, further substantiated by elemental analysis. The other abundant ion peaks observed were 442 {$M^{+\cdot}$-18 (H_2O)}, 409 ($M^{+\cdot}$-H_2O-CH_3), 339, 302, 287, 271, 245 (100), 220, 203, 202, 189.

The peaks at m/z 302 and 189 distinguished it to be having a Δ^{14} characteristic i.e. taraxerene skeleton. **C_Y-3** on acetylation formed a diacetate, m.p. 245-47^{0}C. Its **mass** spectrum showed the molecular ion peak at m/z 526 ($C_{34}H_{54}O_2$) confirming it to be a diacetate and hence in turn **C_Y-3** to be a diol with one primary alcoholic group and one secondary alcoholic group **(ir)**, other ion peaks observed at m/z 511 ($M^{+\cdot}$-CH_3), 466 ($M^{+\cdot}$-HOAc), 334(a), 329(b), 284 (a-HOAc), 269 (b-HOAc), 262 (c), 202 (c-HOAc) and 189 (100) (d); tallied with those of myricadiol diacetate[10], can be explained as shown in the **(scheme-I)**



(a)



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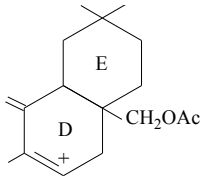
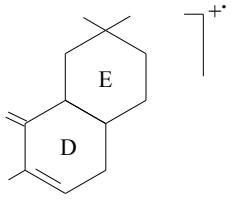
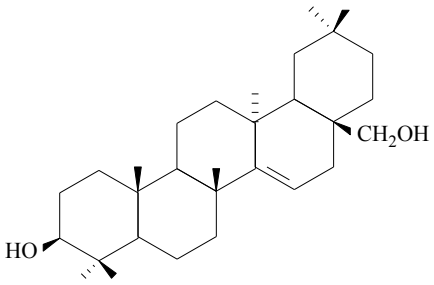


(c)



(d)

On the basis of m.p., acetate, **ir** and **EIMS** studies of **C_Y-3** and its diacetate, the **C_Y-3** was identified as **Myricadiol (II).**



(II)

Compound C_Y-4:

Benzene and chloroform (1:1) eluate afforded another white crystalline **C_Y-4** compound m.p. 159^0C, $[\alpha]^{20}_D - 53.48^0$ ($CHCl_3$). It gave positive Lieber-mann-Burchard test[7] and responded to tetranitromethane colour test. **IR** spectrum showed the band at 3350 and 1050 cm^{-1} (OH) and at 1655 and 840 cm^{-1} (C=C). The **^{1}H-nmr** spectrum indicated signals at δ 0.7, 0.80, 0.88, 1.02 (CH_3 protons), 3.56 (3α-hydroxyl) and at 5.36 (1H, vinyl proton). Spectral data and elemental analysis ($C_{29}H_{48}O$) suggested it to be a **β-sitosterol**. Its acetate m.p. 126^0C gave **ir** bands at 2990, 2880, 1740, 1680, 1470, 1390, 1262 and 970 cm^{-1}. Further derivatisation led to the preparation of benzoate, m.p. 144-45^0C and 3,5-dintro benzoate m.p. 208-12^0C.

For final confirmation **gc-ms (Table-1)** analysis were performed, using a 2.54 x 4 m. I.D. Glass column of 1% Dexil 300 G.C. on 100-120 Diatomic CQ at 260, flow rate 40 ml/min (helium carrier gas) connected through a silicone rubber membrane into an AEIMS-9 **mass** spectrometer. This has been found to consist of the following four components.

Table -1
GLC data of sterols (TMS derivatives) on Dexsil 300 G.C.

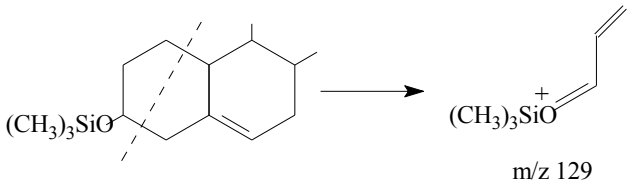
Components	% sterol	RRT**
Cholesterol	0.4	0.60
Campesterol*	11.0	0.81
Stigmasterol*	41.5	0.85
β-sitosterol	47.0	1.0

*Neither **glc** nor **ms** techniques are able to distinguish between sterol C_{24} epimers and these compounds may be either named compounds or its C_{24} epimers.

** Relative retention time (RRT) is expressed by the ratio of retention time for the substance under examination to the retention time of β-sitosterol.

The TMS ether of cholesterol, campesterol, stigmasterol and β-sitosterol gave molecular ions at m/z 458 (24%), 472 (25%), 484 (59%) and at m/z 486 (30%) respectively **(Table-2)**. The characteristic peak at m/z 129 at Δ^5 3β-trimethyl silyloxy steroid for all sterols. The peak at 129 has been identified as the fragment originating from the cleavage of ring-A along with the TMS moiety.

Similarly, the other characteristic fragmentation from Δ^5 3β-trimethyl silyloxy steroid, as reported by Brook,[11] was series of ions from $M^{+\cdot}$-129. These ions were also prominent at m/z 329 (100), 343 (100), 355 (36) and 357 (100) in the **mass** spectra of cholesterol, campesterol, stigmasterol and β-sitosterol trimethyl-silyloxy derivatives respectively.



m/z 129

The structural features which distinguish each of these sterols in the side chain of cholesterol contain C_8H_{17} chain, campesterol has a C_9H_{19} chain, stigmasterol has $C_{10}H_{19}$ chain, due to the presence of double bond at carbon 22, β-sitosterol has a $C_{10}H_{20}$ chain. The peaks at m/z 255, 275 were due ot the loss of TMS and side chain moieties from the parent compounds of cholesterol and campesterol respectively.

Table-2
Mass spectral data of sterols as their trimethylsilyl ether

Mole Formula	High mass species* m/z (A%)		Identity
$C_{27}H_{45}OSiMe_3$	459 (9.8), 458 (24) $M^{+\cdot}$, 443 (12) M^{+}-15, 369 (17), 368 (68) $M^{+\cdot}$-90, 355 (9), 354 (11), 353 (36) $M^{+\cdot}$-90-15, 330 (20), 329 (100) $M^{+\cdot}$-129, 328 (26), 274 (19), 255 (22) $M^{+\cdot}$-S.C., 247 (15).		Cholesterol
$C_{28}H_{47}OSiMe_3$	472 (25) $M^{+\cdot}$, 457 (12) M^{+}-15, 383 (18), 382 (64) $M^{+\cdot}$-90, 368 (11) 367 (34) $M^{+\cdot}$-90-15, 344 (26), 243 (100) $M^{+\cdot}$-129, 342 (15), 389(5), 261(14), 255 (17) $M^{+\cdot}$-BC.S.C., 213 (13).	(or 24epimer)	Campesterol
$C_{29}H_{47}OSiMe_3$	485 (25), 484 (59) $M^{+\cdot}$, 469 (15) $M^{+\cdot}$-15, 395 (24) 394 (68) $M^{+\cdot}$-90, 379 (25) $M^{+\cdot}$-90-13, 355 (36) $M^{+\cdot}$-129, 354 (19.8), 351 (43), 255 (100) $M^{+\cdot}$-90-S.C., 215 (23), 213 (27).	(or 24 epimer)	Stigmasterol
$C_{29}H_{47}OSiMe_3$	488 (11), 486 (30) $M^{+\cdot}$, 471 (13) $M^{+\cdot}$-15, 397 (22), 396 (73) $M^{+\cdot}$-90, 382 (12) 381 (36) $M^{+\cdot}$-90-15, 358 (28), 357 (100), $M^{+\cdot}$-129, 356 (18), 275 (16), 255 (19)$M^{+\cdot}$-90-S.C., 213 (19).		β-sitosterol

*Only masses between m/z 650 and 205 are recorded as this is the most diagnostic region of the spectrum.

Compound C_Y-5:

The product **C_Y-5**, m.p. 87^0C showed **ir** absorption υ^{kBr}_{max} at 2900, 1705, 1480, 1300, 940, 730 and 720 cm^{-1}, thereby indicating it to be an aliphatic carboxylic acid. Elemental analysis showed the molecular formula to be $C_{24}H_{48}O_2$, further confirmed by molecular ion peak at m/z 368. It gave methyl ester m.p. 58-59^0C. The compound was identified as **tetracosnoic acid**[12].

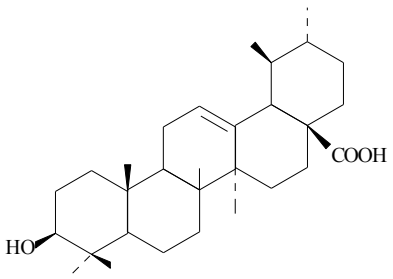
Compound C_Y-6:

C_Y-6 on acetylation gave an acetate (C_Y-6A) m.p. 263-64^0C. The **ir** spectrum of the acetate showed absorptions at υ^{nujol} 1785, 1725, 1265 cm^{-1} charac-teristic of acetyl function.

The **^{1}H-nmr** spectrum of the compound revealed seven methyl groups at δ 0.8 (3H), 0.92 (3H), 1.0 (6H) and 1.1 (3H) and one acetoxyl at δ 2.1. In addition there was a triplet centered at δ 4.46 for a proton α- to the acetoxyl and a signal at δ 5.2 characteristic of the olefinic protons.

On methylation, C_Y-6A gave an acetyl methyl ester m.p. 236-37^0C. Its **^{1}H-nmr** spectrum also showed methyl functions as signlets at δ 0.78 (3H), 0.9 (6H), 0.92 (3H) and 0.98 (3H) and one acetoxyl function as a signlet at δ 2.09. A singlet at δ 3.65 showed the ester methoxyl function. The olefinic proton signal was at δ 5.28 and a triplet for the proton α-to the acetoxyl at δ 4.46.

The acetate (C_Y-6A) on deacetylation gave the **genin** m.p. 268-70^0C. The above physical and spectral data of the genin and its derivatives showed that the compound is mono-hydroxy mono-carboxylic acid. Its identify as **Ursolic acid (III)** was established by comparing the **ir** spectrum of its acetate with an authentic sample of ursolic acid acetate, which was super impossible.



(III)

IV. Conclusion

Extraction and isolation:

Well dried and crushed base of leaves (2 kg) were extracted successively with petroleum ether (60-80^0), benzene and methanol at room temperature and at their boiling points respectively. The petrol and benzene concentrates on TLC examination in petrol-ether as (4:1) solvent system showed at least six major spots, having the same R_f values. As the TLC behaviour of both the petroleum ether and benzene concentrates was the same, these two were combined (35 gm) together for further processing.

Separation into acidic and neutral part:

The dark green viscous mass (35 gm) was taken in ether, treated with aq. solution of potassium hydroxide (15%) and divided into alkali-soluble and alkali insoluble parts. The alkali insoluble part (25 gm) was refluxed with alcoholic potassium hydroxide (30 gm KOH dissolved in 600 ml of 80% ethanol) for half hour. Half of the solvent-was then distilled off and the contents were diluted with water (2 liters) and extracted three times with ether. All the ether extracts were combined together and washed with water, till free of alkali. The ethereal layer was dried over anhydrous sodium sulphate. Sodium sulphate was filtered off and ether was recovered to give the neutral part. (≈ 15 gm).

The aqueous layer was acidified with hydrochloric acid and extracted with ether. The ether extract was washed with water and dried over anhydrous sodium sulphate. The ether was removed, a light yellow solid (≈ 5 gm) was obtained.

Both the hot and cold extracts of methanol showed almost same spots on TLC examination in different solvent systems and therefore were mixed together and concentrated under reduced pressure the resultant mass was refluxed with petrol, benzene, chloroform, ethylacetate respectively and finally with acetone.

The ethylacetate and acetone concentrates on TLC examination in solvent system viz. TEF (5:4:1), BPF (36:9:5) and EtOAc: EtMeCO: AcOH: H_2O (2:3:1:1, 5:3:1) showed two compounds with varying concentration. They were combined and subjected to column chromatography over silica gel column using benzene-ethyacetate (9:1 to 1:1) yield two compounds **C_Y-7** and **C_Y-8** in different fractions. They were separated by repeated column chromatography.

Neutral part:

The neutral part (≈ 15 gm) was subjected to column chromatography over neutral alummina (1 Kg) and eluted with petrol, petrol-benzene and petrol-chloroform in different proportions and monitored by TLC. Following compounds **C_Y-1, C_Y-2, C_Y-3** and **C_Y-4** were isolated from different pools of identical fractions.

Compound C_Y-1:

Elution of the column with petroleum ether ($60\text{-}80^0$) gave a dirty semi-solid mass. This was further purified by column chromatography over silica gel. On elution with hexane and repeated crystallization from carbon tetrachloride and acetone a colourless compound **C_Y-1** m.p. $62\text{-}67^0$C was obtained. This showed an alongated spot on TLC (silica gel/$AgNO_3$, 5%) in petrol-benzene (4:1) solvent system. It was found to be a saturated hydrocarbon, **(ir)** and the elemental analysis, compared with that of triacontane; υ_{max} 2930, 2860 (C-H, saturated), 1460, 1380 (C-CH_3), 720 cm^{-1} $(CH_2)_n$.

Analysed for $C_{30}H_{62}$: Calcd: C, 85.30; H, 14.69%. Found: C, 85.56; H, 14.58%.

For final confirmation it was subjected to **glc** analysis which indicated this product to be a mixture of n-alkanes of the series C_{24}-C_{36} as given below **(Table-6)**. Odd number homologeus predominated as usual.

Table-6

S.No.	n-alkane		Composition %
1.	n-Triacontane	($C_{30}H_{62}$)	36.3
2.	n-Tri-Triacontane	($C_{33}H_{66}$)	28.9
3.	n-Non-acosane	($C_{29}H_{60}$)	21.2
4.	n-Triacontane	($C_{30}H_{62}$)	2.9
5.	n-Heptacosane	($C_{27}H_{56}$)	2.0
6.	n-Hexatriacontane	($C_{36}H_{74}$)	2.0
7.	n-Octacosane	($C_{36}H_{58}$)	2.0
8.	n-Hexacosane	($C_{26}H_{59}$)	very small quantity
9.	n-Pentacosane	($C_{35}H_{72}$)	very small quantity
10.	n-Pentatriacontane	($C_{36}H_{74}$)	very small quantity

Compound C_Y-2:

Further elution of the neutral part with petroleum ether-benzene (1:4) and purification by repeated crystallization from methanol-chloroform gave a crystalline solid **(C_Y-2)**, m.p. $214\text{-}15^0$C, $[\alpha]^{20}_D + 23.64^0$ ($CHCl_3$). It gave positive Liebermann-Burchard and Noller's test and yellow colour with tetranitromethane.

IR, υ^{kBr}_{max} cm^{-1}:

3360, 1030 (OH), 1645 (C=C), 1385 (geminal dimethyl), 885 (terminal methylene)**.**

C_Y-2 was confirmed as **Lupeol** by m.p. and mixed melting point with an authentic sample. Further confirmation of the identity of the compound was obtained by spectral studies and by its derivatisation.

Acetylation of C_Y-2:

The compound (50 mg) was treated with acetic anhydride (2 ml) and pyridine (0.2 ml), allowed to stand overnight at room temperature and then heated on a water both for 6 hours. The solid product obtained, after usual work up, was crystallized from methanol-chloroform mixture as colourless flakes (60 mg), m.p. 218-20^{0}C.

IR, υ^{kBr}_{max} cm^{-1}:

875 (terminal methylene), 1245 (acetate), 1640 (C=C), 1730 (C=O).

^{1}H-NMR ($CDCl_3$) on δ scale:

0.82, .0.87, 0.94, 1.04, 1.27, 1.41, 1.46, 1.70, 2.03, ($OCOCH_3$), 2.28, 4.77 (>CHOAc).

Analysed for $C_{32}H_{52}O_2$: Calcd.: C, 82.05; H, 11.11%. Found:C. 82.14; H, 11.17%.

Compound C_Y-3:

Elution of the column with benzene, followed by crystallization from benzene-ethylacetate gave a colourless amorphous powder, m.p. 259-60^{0}C. It gave a positive Stannic chloride test showing it to be a triterpene.

IR, υ^{kBr}_{max} cm^{-1}:

3415 (OH), 3060, 2940 (unsaturation), 2880, 1470, 1450, 1390, 1380 (characteristic of triterpenic skeleton), 1080, 1030 (C-O stretching and O-H in plane deformation of secondary alcohol), 815.

Mass, m/z:

$M^{+\cdot}$ 442, 424, 409, 339, 302, 287, 271, 257, 245 (100%), 220, 203, 202, 189.

Acetylation of C_Y-3:

The compound **(C_Y-3),** (100 mg) was treated with acetic anhydride (2.0 ml) and pyridine (1.0 ml) and left overnight at room temperature. After usual work up followed by crystallisation from ethanol colourless needles m.p. 245-47^{0}C, were obtained.

Mass, m/z:

$M^{+\cdot}$ 526, 511, ($M^{+\cdot}$-Me), 466 ($M^{+\cdot}$-HOAc), 344, 329, 284, 269, 262, 202, 189 (100%).

Compound C_Y-4:

Elution of the column with benzene-chloroform (1:1) and purification of the product obtained from the column by repeated crystallization from methanol-chloroform afforded white crystalline solid **(C_Y-4)** m.p. 159-60^{0}C, $[\alpha]^{20}_D$ – 53.48^0. It gave positive Libermann-Burchard test and yellow colour with tetranitro-methane.

Analysed for $C_{29}H_{48}O$: Calcd: C, 84.40; H, 11.72%. Found: C, 84.46; H, 11.91%.

IR, υ^{kBr}_{max} cm^{-1}:

3350, 1050 (OH), 1655 (C=C), 840 (terminal methylen).

^{1}H-NMR, ($CDCl_3$) on δ scale:

0.70, 0.80, 0.88, 1.02 (CH_3 protons), 3.56 (3α-hydroxyl), 5.36 (1H, vinyl proton).

Acetylation of C_Y-4:

The above product (100 mg) was acetylated by the usual method, using acetic anhydride (2 ml) and pyridine (0.4). The acetate was crystallized from methanol-chloroform mixture as colourless flakes m.p. 126^{0}C.

IR, υ^{kBr}_{max} cm^{-1}:

1740 (C=O), 1680 (C=C), 1262 (acetate), 970 (terminal).

Benzoate:

C_Y-4, (50 mg) was dissolved in minimum amount of pyridine and benzoyl chloride (1 ml) was added to it. The mixture was heated over a boiling water bath for 8 hours, cooled to room temperature and poured into crushed ice with stirring. The solid obtained was washed with aqueous solution of potassium hydroxide (2%) followed by excess of water. The solid was crystallized from methanol, m.p. 144-45^{0}C (35 mg).

3,5-Dinitrobenzoate:

C_Y-4, (50 mg) was treated with freshly prepared 3,5-dinitrobenzoyl chloride (60 mg) and pyridine (0.5 ml) and heated over a water bath for 45 mints. After usual work up the crude derivative was crystallized from acetone and methanol m.p. 208-12^{0}C.

Finally **gc-ms** analysis of the sterol (TMS derivative) indicated it to be a mixture of cholestrol ($M^{+\cdot}$ 458), campesterol ($M^{+\cdot}$ 472, m/z 457, 383, 382, 368, 344, 255, 213 etc.), stigmasterol ($M^{+\cdot}$ 484, m/z 469, 395, 394, 379, 355, 354, 255, 215, 213 etc) and β-sitosterol ($M^{+\cdot}$ 486, m/z 471, 397, 396, 382, 357, 275, 255, 213 etc.).

Alkali soluble part:

The yellow acidic part (≈ 5 gm) was dissolved in benzene-ether (8:1, v/v) and chromatographed over silica gel (300 gm). Mainly two products were obtained marked as **C_Y-5** & **C_Y-6**.

Compound C_Y-5:

On elution with petroleum ether (60-80^0) **C_Y-5**, m.p. 87^{0}C was obtained. This appeared to be a saturated (negative tetranitromethane test) aliphatic acid υ_{max} 2900, 1300, 940 (OH), 1705, 1480 and 730, 720 cm^{-1} $(CH_2)_n$

.

The methyl ester of **C_Y-5** was prepared by treatment with absolute meth-anol. On crystallization with acetone, low melting (58-59^{0}C) crystals were obtained.

Compound C_Y-6:

Further elution of the column with ethylacetate, a colourless product was obtained. It was crystallized from chloroform-methanol as colourless shining needles, m.p. 284-88^{0}C. It appeared to be ursolic acid on the basis of its m.p., m.m.p. and co-TLC with an authentic sample. Its identity as **Ursolic acid** was further supported by derivitisation of the **C_Y-6**.

Acetylation of C_Y-6:

Acetate was prepared by usual method, m.p. 263-64^{0}C. It showed no depression in melting point when mixed with an authentic sample of ursolic acid acetate.

IR, υ^{nujol}_{max} cm^{-1}:

1785, 1725, 1265 **.**

^{1}H-NMR ($CDCl_3$), on δ scale:

0.8 (3H, s), 0.9 (s), 0.92 (3H, s), 1.0 (6H, s), 1.1 (3H, s), 2.1 (s), 4.46 (t), 5.26 (m).

Mass, m/z:

498 ($M^{+\cdot}$), 438, 249, 203, 189.

Acetyl methyl ester:

The above compound was treated with diazomethane. After usual work up followed by crystallization from methanol gave colourless needles m.p. 236-37^{0}C.

^{1}H-NMR ($CDCl_3$) on δ scale:

0.78 (3 H, s), 0.9 (6H, s), 0.92 (3H, s), 0.98 (3H, s), 0.98 (s), 2.09 (s), 3.65 (s), 4.46 (s), 5.28 (m).

Mass, m/z:

512 ($M^{+\cdot}$), 452, 262, 249, 203, 184.

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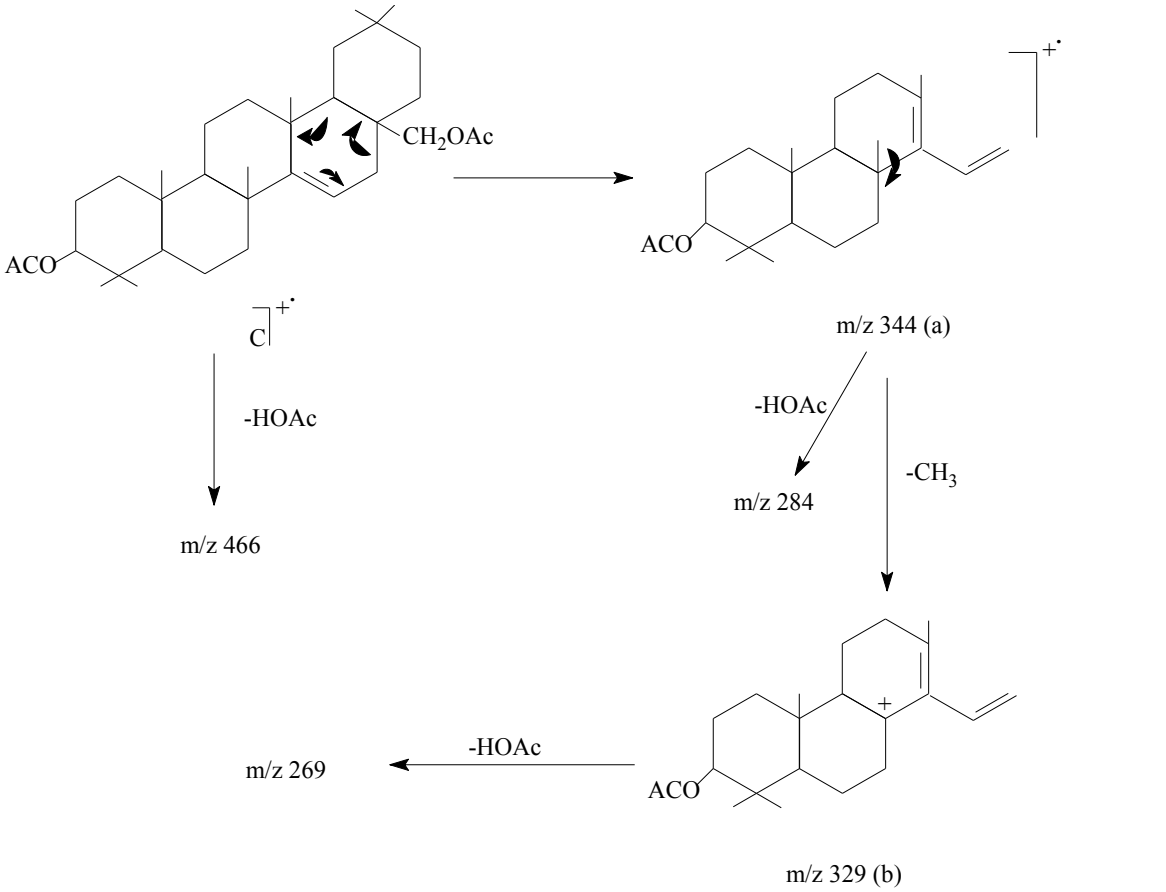
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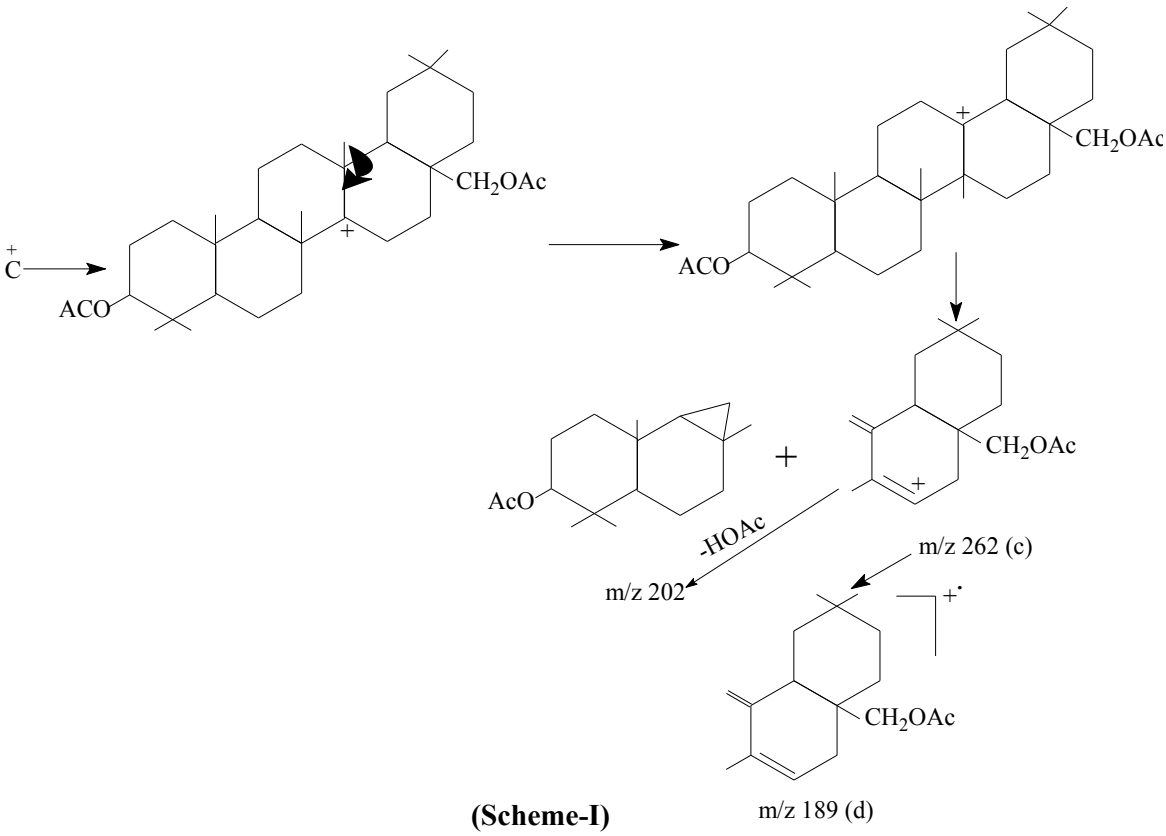
ACO
m/z 344 (a)
-HOAc
-CH3
m/z 284
m/z 466
m/z 269
m/z 329 (b)


CH2OAc
ACO
AcO
-HOAc
m/z 262 (c)
m/z 202
m/z 189 (d)

(Scheme-I)