CHAPTER—III

EXPERIMENTAL

Melting points are uncorrected. Petroleum ether used during the investigation had b.p. 60°—80°. All the optical rotations were determined in $CHCl_3$ at 1—2% solutions. The ^1H NMR spectra were recorded in Varian A—60, T—60, FT—80A and XL—100 NMR spectrometers using $CDCl_3$ containing TMS as interval reference. The IR spectra were taken in Beckman IR—20 spectrophotometer in Nujol mull. UV absorption spectra were taken in Beckman DU—2 spectrophotometer using solvent 95% ethanol. Column chromatography were done on neutral alumina deactivated with 10% aqueous acetic acid (4 ml/100 g of alumina).

SECTION—A:

Oxidation of taraxeryl acetate 76 with NBS in DMSO: Preparation of 15-bromo- β -amyrin acetate 77:

To a solution of taraxeryl acetate (1 gm) in $CHCl_3$ (50 ml) and dimethyl sulfoxide (25 ml), was added N-Bromo succinimide (1 gm) in portions and kept in dark for 14 hours. The reaction mixture was filtered and the filtrate was extracted with chloroform (200 ml), washed with water and was dried over anhydrous sodium sulphate. The solvent was removed when a yellowish solid was obtained. The solid was dissolved in benzene (10 ml) and was poured on a column of alumina (60 gm deactivated with 3.6 ml of 10% aqueous acetic acid) developed with petroleum ether. The following solvents were used for elution:

Table—1

Eluent	Fractions 100 ml each	Residue on evaporation
Petroleum ether	1—10	Solid (900 mg) m.p. 178°-180°
Further elution with more polar solvents did not give any solid material		

Fraction 1—10 (table—1) were combined and the mixture was crystallised from a mixture of chloroform and methanol when crystals of 15-bromo-β-amyrin acetate 77, m.p. 180—182°, $[\alpha]_D$ +47.4° were obtained. It gave a single spot on a chromatoplate (R_f = 0.8) and also showed positive Beilstein test for bromine.

Analysis report:

Found,	C, 70.0%; H, 9.1%
Calculated for $C_{32}H_{51}O_2Br$	C, 70.23%; H, 9.3%

IR(nujol): ν_{max} 1720 and 1250 cm^{-1} Fig.1

NMR : Peaks at 5.3 (m, Vinyl proton), 4.30 (m, $\underline{H}$ CBr), 2.09 ($-OCOC\underline{H}_3$) and 4.53 ($\underline{H}-COCH_3$) ppm. Fig.2

Mass : Peaks at m/e 189, 216, 217, 249, 297, 466 (M —HBr), 547(M^+).

Oxidation of 15-bromo-β-amyrin acetate 77: Preparation of 11-oxo-15-bromo-β-amyrin acetate 79:

To a refluxing solution of 15-bromo-β-amyrin acetate (1 gm) in benzene (40 ml) was added slowly a solution of $Na_2Cr_2O_7$ (1 gm) in glacial AcOH (30 ml) with stirring while maintaining the temp. at 70°C. After the addition was complete the stirring was continued for 24 hours maintaining the temperature at 80°—85°. At the end it was cooled and rectified spirit (5 ml) was added. The solution was concentrated to 1/3rd volume and poured into ice cold water. The ppt was filtered off, washed and crystallised from chloroform-methanol mixture when crystals (0.75 g) having m.p. 240—241° were obtained $[\alpha]_D$ +89°.

Analysis report:

Found, C, 68.24% ; H, 8.63%

Calculated for $C_{32}H_{49}O_3Br$, C, 68.42% ; H, 8.69%

UV λ_{max} 249 nm (ϵ 11,000)

Beilstein test positive.

Attempted dehydrobromination of 11-oxo-15-bromo-β-amyrin acetate 79:

(a) With dimethyl aniline:

Compound (0.5 g) was refluxed with dimethylaniline (75 ml) for six hours. The mixture was cooled, acidified with 6N HCl and then extracted with ether and washed with water to make it acid free. The solution was dried with anhydrous sodium sulphate. Solvent was removed, the residue was then chromatographed in a column of 30 gm alumina. The chromatogram was developed with petroleum ether and the following fractions were collected:

Table—2

Eluent	Fractions 100 ml each	Residue on evaporation
Petroleum ether	1—12	Solid m.p. 229°—230°
Further elution with more polar solvents did not afford any material		

Fractions 1—12 (table—2) were combined. The mass was crystallised from chloroform-methanol mixture when crystals having m.p. 240°—241° were obtained. The compound showed positive Beilstein test. Mixed m.p. with the starting material was also undepressed.

(b) With s-collidine:

Bromo compound (0.2 g) was refluxed with s-collidine (15 ml) for 18 hrs. at 180° in an oil bath. The mass was then acidified with 5% H_2SO_4, extracted with ethyl acetate. The organic layer was washed with 5% H_2SO_4 to remove last trace of collidine, washed with water to make organic layer neutral. The solvent was dried over anhydrous sodium sulphate and removed. The residue dissolved in minimum amount of benzene was chromatographed in a column of alumina, and the following fractions were collected:

Table--8

Eluent	Fractions 100 ml each	Residue on evaporation
Petroleum ether	1--10	Solid m.p. 230°-1°
Further elution with more polar solvents did not afford any solid material		

Fractions 1--10 (table--8) were combined and crystallised from chloroform-methanol mixture. The crystals had the m.p. 240--241°. Mixed m.p. with the parent compound and Beilstein test indicated that dehydrobromination did not occur.

Dehydrobromination of 18-bromo- β -amyrin acetate 77 with dimethyl aniline: Preparation of olean-12,18-diene-3 β -yl acetate 81:

18-Bromo- β -amyrin acetate (0.2 g) was refluxed with distilled dimethyl aniline (30 ml) for six hours. The reaction mixture was diluted with water, acidified with 6N hydrochloric acid and was extracted with ether. The ether extract was washed with water till neutral and was dried with sodium sulphate. The solvent was removed when a solid (0.17 g) was obtained. The solid was dissolved in minimum amount of benzene and the solution was poured on a column of alumina (15 g) developed with petroleum ether. The following fractions were collected;

Table—4

Eluent	Fractions 50 ml each	Residue on evaporation
Petroleum ether	1—5	Solid (0.155 g) m.p. 195—197°

Further elution with more polar solvents did not afford any solid material

Fraction 1—5 (table—4) combined and crystallised from chloroform methanol mixture when crystals of 81, m.p. 199°—200°, $[\alpha]_D$ +42° were obtained.

Analysis report:

Found,	C, 81.9%	; H, 10.8%
Calculated for $C_{32}H_{50}O_2$,	C, 82.35%	; H, 10.8%

UV : No absorption in the region 220—300 nm.

IR : ν max 1730, 1240 cm^{-1} (Acetate)
875 cm^{-1} (—CH = CH—)
820 cm^{-1} (trisubstituted double bond)
Fig.3

NMR : 5.2 to 5.6 ppm for three vinyl protons accounting for one trisubstituted and one disubstituted double bond. 2.03 ($OCOC\underline{H}_3$), 4.5 ($\underline{H}$—C—$OCOCH_3$) ppm Fig.4

Oxidation of olean-12,15 dien-3 β yl-acetate 81; preparation of 11-oxo-olean-12,15-dien-3 β -acetate 80:

A solution of sodium dichromate (4 gm) in glacial acetic acid (100 ml) was added slowly during a period of one hour to a vigorously stirred solution of olean-12,15-dien-3 β -acetate (4 gm) in refluxing benzene (50 ml). The mixture was refluxed for 24 hours and then cooled. Excess of dichromate was decomposed with rectified spirit (50 ml). The solution was concentrated to 1/3 rd volume and the contents poured into ice cold water. Then it was extracted with solvent ether. The ethereal layer was washed with water and dried over anhydrous sodium sulphate. The solvent was removed. The residue (4 gm) was dissolved in minimum amount of benzene and was poured on a column of alumina (240 g), developed with petroleum ether. The following solvents were used for elution:

Table—5

Eluent	Fractions 100 ml each	Residue on evaporation
Petroleum ether	1—5	Nil
Petroleum ether: Benzene (4:1)	6—10	Nil
Petroleum ether: Benzene (3:2)	11—18	Solid m.p. 238—240°

Further elution with more polar solvents did not afford any solid material

Fractions 11—18 (table—5) were combined and the mass was crystallised from chloroform-methanol mixture when needle-shaped crystals of 3β-acetoxy olean-12,15-dien-11-one <u>80</u>, m.p. 243°—245° $[\alpha]_D$ +28.8° were obtained.

Analysis report:

Found, C, 79.86% ; H, 9.95%

Calculated for $C_{32}H_{48}O_3$, C, 79.95% ; H, 10.06%

UV : λ_{max} 244 nm (ϵ 11,376)

IR : ν_{max} 1730, 1240 cm^{-1} (Acetate)

1660 cm^{-1} (6 membered ring $\alpha\beta$ unsat. ketones)

890 cm^{-1} (—CH=CH—) Fig.5

NMR : 0.82—1.04 (8, t-Me protons),

2.04 (s, 3H, $-OCOC\underline{H}_3$)

2.25 (dd, 1H, C-18 β -H)

2.42 (s, 1H, C, 9α-H)

2.82 (t, of d, 1H, C-1 β -H)

4.45 (t, 1H $\underline{H}$-C-O-CO-)

5.38 (s, 2H, 2 vinyl H)

5.64 (s, 1H, 1 vinyl H) ppm Fig.6

Lithium aluminium hydride reduction of 11-oxo-olean-12,15-dien-3β yl acetate 80; preparation of olean-12,15-dien-3 11 diol 70 :

A solution of keto acetate 80 (2.5 gm) in dry benzene (30 ml) and dry ether (70 ml) was refluxed with LAH (5 gm) for six hours followed by stirring at room temperature for another twelve hours. Excess LAH was destroyed by adding water dropwise at room temperature. The ethereal layer was separated and the aqueous layer was extracted with ether and the two ether solutions were mixed. The ethereal layer was washed with water and dried over anhydrous sodium sulphate. The solvent was removed and the residue (1.9 gm), of olean-12,15-dien-3,11-diol 70, m.p. 198—200° was obtained. TLC single spot.

IR : ν_{max} 3460, 890, 860, 820, 750 cm^{-1} Fig.7

Treatment of olean-12,15-dien-3,11-diol 70, with hydrogen peroxide in para toluene sulphonic acid; Isolation of two lactones 85 and 86:

To a solution of olean-12,15-dien-3,11-diol (1.9 gm) in methylene chloride (100 ml) was added a solution (85 ml) prepared by mixing para toluene sulphonic acid (5 g) and 30% H_2O_2 (5 ml) in tert-butanol (80 ml). The reaction mixture was stirred slowly for 24 hours and then poured into water. It was then extracted with methylene chloride, washed with water. The methylene chloride was dried over anhydrous sodium sulphate and the solvent was removed under reduced pressure. The residue (1.5 gm) was absorbed in an alumina (80 gm) column. The column was developed with petroleum ether and the following solvents were used for elution:

Table—6

Eluent	Fractions 100 ml each	Residue on evaporation
Petroleum ether	1—4	Trace oil
Petroleum ether:benzene (4:1)	5—8	Nil
Petroleum ether:benzene (3:2)	9—12	Trace oil
Petroleum ether:benzene (2:3)	13—17	Nil
Petroleum ether:benzene (1:4)	18—20	Nil
Benzene	21—40	Solid 220°—30°

Further elution with more polar solvents did not afford any solid material

Fraction 21—40 (table—6) were mixed and found to be a mixture of two compounds. (From TLC, R_f values of 85 = .49; 86 = .46 in EtOAC). The mixture was repeatedly crystallized from chloroform-methanol when 85 (0.3 g) separated as the less soluble part. Further three times crystallization of 85 from chloroform-methanol afforded pure 85, (no increase of m.p. by furthercrystallization), m.p. = 240°—41°.

Analysis report:

Found, C, 78.94% ; H, 9.89%

Calculated for $C_{30}H_{46}O_3$, C, 79.25% ; H, 10.20%

UV : No absorption in the region 220—300 nm

IR : ν_{max} 3520 cm^{-1} (free -OH group),
875 cm^{-1} (—CH=CH—),
1775 cm^{-1} (lactone) Fig.8

Acetylation of C-12-nor olean-15-en-13α-carb→19α-olide-3β-ol 85; Preparation of 3β-acetate of C—12-nor olean-15-en-13α-carb→19α-olide 85a:

The hydroxy lactone (0.1 g) was dissolved in dry pyridine (1 ml) and the solution was treated with acetic anhydride (2 ml). The mixture was then heated on water bath for 4 hours and then poured into ice cold water and then filtered. The residue was washed with water and dried under suction. The product was then crystallised from chloroform methanol mixture when crystals of 85a, $C_{32}H_{48}O_4$, m.p. 215°—16° were obtained.

Analysis report:

Found, C, 77.25% ; H, 9.80%

Calculated for $C_{32}H_{48}O_4$, C, 77.33% ; H, 9.74%

UV : No absorption in the region 220°—330° nm

IR : ν_{max} 1720 cm^{-1}, 1250 cm^{-1} ($OCOCH_3$)
1780 cm^{-1} (γ-lactone) Fig.9

Mass : m/e, 496 (M^+), 468 (M^+—CO),
452 (M^+—CO_2), 436 (M^+—AcOH) Fig.15

Isolation of the second lactone 86:

The more soluble fraction was crystallized twice from chloroform-methanol and the purer product was isolated from the filtrate (0.2 g), of m.p. = 253—57°, moleculer formula, $C_{30}H_{46}O_3$.

Analysis report:

Found, C, 79.45% ; H, 9.78%
Calculated for $C_{30}H_{46}O_3$, C, 79.25% ; H, 0.20%

IR(nujol): ν_{max} 3520 cm^{-1} (free OH group),
875 cm^{-1} (—CH=CH—),
1775 cm^{-1} (γ-lactone)

Mass: m/e, 454 (M^+), 436 (M^+—H_2O), 426 (M^+—CO),
410 (M^+—CO_2).

Acetylation of lactone 86; Preparation of 3β-acetate of C-12-nor-olean-18(19)-en-13β-carb→15β-olide 86a:

0.1 gm of the lactone 86 was taken in 1 ml of dry pyridine and 1 ml acetic anhydride was added to it. The mixture was kept on water bath for four hours. Then it was cooled and poured into ice cold water. The ppt. was separated out and filtered. The residue was washed with water and dried over suction. The mass was crystallized from chloroform-methanol mixture which gave the acetate <u>86a</u>, m.p. 228°—229°.

<u>Analysis report:</u>

Found, C, 77.38% ; H, 9.94%

Calculated for $C_{32}H_{48}O_4$, C, 77.30% ; H, 9.74%

<u>IR(nujol):</u> 1720 cm^{-1}, 1250 cm^{-1} (OCOCH$_3$), 1780 cm^{-1} (γ -lactone) Fig.16

<u>Mass</u> : m/e 496 (M^+), 468 (M^+—CO), 452 (M^+—CO_2), 436 (M^+—AcOH).

<u>Lithium aluminium hydride reduction of lactone 86:</u>

200 mg of the lactone <u>86</u> was taken in 50 ml dry ether, 0.4 gm LAH was added. The mixture was refluxed for a period of 4 hours. Excess of LAH was decomposed with a saturated solution of sodium sulphate. The product was extracted with ether. The ethereal solution was washed with water and dried over anhydrous sodium sulphate. The solvent was distilled off and the residue when recrystallized from acetone furnished the triol <u>87</u>, $C_{30}H_{52}O_3$, m.p. 280°—282° (TLC single spot).

Analysis report:

Found, C, 78.42% ; H, 10.84%

Calculated for $C_{30}H_{52}O_3$, C, 78.58% ; H, 10.99%

Acetylation of triol 87; Preparation of the triacetate 87a:

120 mg of the triol 87 was dissolved in 2.0 ml of dry pyridine. To the mixture 2.0 ml of acetic anhydride was added and the reaction mixture was kept on water bath for 12 hours. Then it was cooled and poured into ice cold water when solid separated out. It was filtered, washed with water and dried under suction. It was then crystallized from chloroform methanol to afford the triacetate 87a (0.1 g), $C_{36}H_{58}O_6$, m.p. 190°—193°. It gave single spot in TLC.

Analysis report:

Found, C, 72.96% ; H, 10.52%

Calculated for $C_{36}H_{58}O_6$, C, 73.68% ; H, 9.96%

UV : No UV absorption in the region 220—300 nm

IR : ν_{max} 1740 cm^{-1}, 1250 cm^{-1} (Acetate group)

Fig.17

NMR : 0.5 to 1.5 ppm (tertiary methyl groups) 2.05 ppm ($—OCOCH_3$); 5.26 (vinyl protons)

Fig.18

Mass : m/e 584 (M^+), 524 ($M^+—AcOH$), 464 ($M^+—AcOH$)

Fig.20

SECTION—B:

Isolation of glochidonol from *Glochidon accuminatum*:

10 gms of the benzene extract of *Glochidon accuminatum* was dissolved in minimum amount of benzene and was absorbed in a silica gel column (60 g). The column was eluted with the following solvents:

Table—7

Eluent	Fractions 100 ml each	Residue on evaporation
Petroleum ether	1—4	Oil (3 g)
Petroleum ether:benzene (4:1)	5—8	Solid (0.5 g)
Petroleum ether:benzene (3:2)	9—13	Solid (1 g)
Petroleum ether:benzene (2:3)	14—20	Nil
Petroleum ether:benzene (1:4)	21—30	White solid (0.5 g)

Further elution with more polar solvents did not afford any solid material

Fractions 21—30 (table—7) were mixed and the mixture was crystallized from chloroform-methanol when crystals of m.p. 227° ⎡Lit. m.p. 228°⎦ was identified as Glochidonol 94; IR (nujol); ν_{max} at 3390, 1710, 1449, 1389 and 888 cm^{-1}.

Preparation of lup-3-one-1-ol, 95:

Glochidonol (900 mg), dissolved in ethyl acetate (200 ml) and acetic acid (200 ml), was stirred in presence of Adam's catalyst in an atmosphere of hydrogen till the absorption ceased. The catalyst was removed by filtration and the filtrate was washed with water till neutral. It was dried and the solvent was removed. A solid residue (850 mg) was obtained which after crystallisation from methanol furnished an amorphus solid identified as lup-3-one-1-ol 95.

Dehydration of lupane-3-one-1-ol 95 by acetic anhydride-pyridine; Preparation of lup-1(2)-en-3-one 96:

800 mg of dihydroglochidonol, 95 was taken in 8 ml dry and distilled pyridine and to the reaction mixture 8 cc of acetic anhydride was added and it was kept on water bath for 24 hours. After 24 hours the mixture was cooled and was poured into ice cold water where a solid separated out

which was filtered. The ppt. was washed several times with water and dried under suction. The mass was dissolved in minimum amount of benzene and poured into an alumina column (30 g). The column was eluted with the following solvents:

Table—8

Eluent	Fractions 50 ml each	Residue on evaporation
Petroleum ether	1—15	White solid m.p. 165°—166°
Further elution of more polar solvents did not afford any solid material		

Fractions 1—15 (table—8) were combined and crystallized from chloroform-methanol mixture to afford crystals of m.p. 177—178° (lit. m.p. 177—179°).

Analysis report:

Found,	C, 84.83% ; H, 11.37%
Calculated for $C_{30}H_{48}O$,	C, 84.84% ; H, 11.39%

IR(nujol) : ν_{max} at 1680 cm^{-1} ($\alpha\beta$ unstd. carbonyl)

Fig.21

Lithium aluminium hydride reduction of lup-1(2)-en-3-one 96: Preparation of lup-1(2)-en-3-ol 88:

650 mg of the $\alpha\beta$-unsaturated keto compound 96 dissolved in 500 ml dry ether, 1 gm of LAH was added and the mixture was refluxed for 4 hours. The mixture was cooled and excess LAH was decomposed with saturated solution of sodium sulphate. It was then extracted with ether and washed with water. The washed ether solution was dried with anhydrous sodium sulphate. Removal of ether gave a mass which was dissolved in minimum amount of benzene and poured into a silica gel column (30 gms) developed with petroleum ether. The column was eluted with the following solvents:

Table—9

Eluent	Fraction 50 ml each	Residue on evaporation
Petroleum ether	1—4	Nil
Petroleum ether:benzene (4:1)	5—8	Nil
Petroleum ether:benzene (3:2)	9—20	White solid m.p. 208°—209°

Further elution of more polar solvents did not afford any solid material

The fractions 9—20 (table—9) were mixed and crystallised from chloroform-methanol, when the crystals of m.p. 215° were obtained. TLC single spot.

Analysis report:

Found,	C, 84.43% ; H, 11.80%
Calculated for $C_{30}H_{50}O$,	C, 84.44% ; H, 11.81%

UV : No UV absorption in the region 220—300 nm

IR(nujol): ν_{max} 3450 cm^{-1} (—OH group), 880 cm^{-1} (double bond) Fig.22

NMR : 0.7—1.01 (8 methyl groups), 3.25 (m, H-C-OH), 4.58 (m, 1H vinyl proton), 4.69 (d, 1H vinyl proton) ppm. Fig.23

Treatment of lup-1(2)-en-3-ol 88, with hydrogen peroxide in acid medium:

To 0.5 gm of lup-1(2)-en-3-ol 88 in 25 ml of methylene chloride, was added a mixture of 1.5 ml of 30% H_2O_2 and 20 ml of tertiary butyl alcohol. Then 1.0 gm of p-toluene sulphonic acid was added to the reaction mixture at room temperature. The solution was stirred slowly for 24 hours and then poured into water. The mixture was extracted with methylene chloride and the methylene chloride solution was washed with water. The organic layer was dried over anhydrous sodium sulphate and then evaporated when a solid residue (0.45 gm) was obtained. The residue was dissolved in minimum amount of benzene, poured into a column of silica gel (30 gms). The column was developed with petroleum ether and eluted with following solvents:

Table—10

Eluent	Fraction 100 ml each	Residue on evaporation
Petroleum ether	1—5	Trace oil
Petroleum ether:benzene (4:1)	6—10	Nil
Petroleum ether:benzene (3:2)	11—15	Nil
Petroleum ether:benzene (2:3)	16—20	Nil
Petroleum ether:benzene (1:4)	21—26	Nil
Benzene	27—30	Nil
Benzene:ether (4:1)	31—40	White solid m.p. 270—80°

Further elution with more polar solvents did not afford any solid material

The solid obtained in the fractions 31—40 (table—10) were mixed and the mixture was crystallised from methanol when the crystals of 97 had m.p. 280°—281°.

Analysis report:

Found, C, 78.54% ; H, 10.98%

Required for $C_{30}H_{50}O_3$, C, 78.55% ; H, 10.98%

IR(nujol): ν_{max} 3420 cm^{-1} (—OH group) 1695 cm^{-1}(—COOH group)

Fig.24

Esterification of the above hydrogen peroxide reaction product 27:

200 mg compound was dissolved in ether (30 ml), added a solution of diazomethane in ether prepared from nitrosomethyl urea (200 mg) and was kept over-night. On the following day excess of diazomethane was destroyed with acetic acid. The ethereal solution was washed with water, 10% sodium bicarbonate solution and again with water till neutral and was then dried with anhydrous sodium sulphate. Evaporation of solvent gave a solid (175 mg) which was poured into a column of silica gel (30 gm) and was eluted with following solvents:

Table—11

Eluent	Fraction 100 ml each	Residue on evaporation
Petroleum ether	1—4	Nil
Petroleum ether:benzene (4:1)	5—8	Nil
Petroleum ether:benzene (3:2)	9—15	White solid m.p. 245°—251°

Further elution with more polar solvents did not afford any solid material

Fraction 9—15 (table—11) were mixed and it was crystallised from chloroform-methanol when the crystals of 98, having m.p. 265°—266° were obtained.

Analysis report:

Found,	C, 78.77% ; H, 11.09%
Calculated for $C_{31}H_{52}O_3$,	C, 78.75% ; H, 11.09%

IR(nujol): ν_{max} 1735 cm^{-1}, 3560 cm^{-1} Fig.25

NMR : δ 0.8—1.01 (for 8 methyl groups), 2.75 (H-C-COOCH$_3$), 3.2 (H-C-OH), 3.65 (-COOCH$_3$) Fig.26

Mass : m/e 473 (M$^+$), 456, 454, 439, 411, 395, 251, 235, 205, 191 Fig.27

Acetylation of the above hydroxy ester 98; Preparation of ester acetate 99:

100 mg of the ester 98 was dissolved in 1 ml of pyridine. This was treated with 1 ml of acetic anhydride and the mixture was heated on a water bath for 4 hours. The reaction mixture was then cooled and poured into ice cooled water when a solid was separated out. It was filtered and was washed with water and dried under suction. The compound was crystallised from chloroform-methanol mixture when crystals of m.p. 250°—251° were obtained. TLC single spot.

<u>Analysis report:</u>

Found, C, 77.58% ; H, 9.85%

Calculated for $C_{33}H_{50}O_4$, C, 77.6% ; H, 9.86%

<u>IR(nujol):</u> ν_{max} at 1710 cm^{-1} (broad for ester & acetoxy)

1240 cm^{-1} (acetoxy) Fig.27

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