CHAPTER - III

EXPERIMENTAL

Melting points are uncorrected. The petroleum ether used throughout the investigation had b.p. 60°-80°C. The Infra Red spectra were recorded in BECKMAN IR - 20 or Pye Unichem-300 Spectrophotometer. The UV absorption spectra were taken in Shimadzu UV-240 or BECKMAN DU-2 Spectrometer, ^{1}H NMR and ^{13}C NMR spectra were recorded with Bruker WH-400 or Bruker WH-270 Spectrometer using deuterated chloroform solution containing tetramethyl silane as reference. Silica gel used for column chromatography was of 60-120 mesh (B.D.H. and Glaxo). TLC were done on chromatoplates prepared on glass strips with silica gel using benzene-petrol mixture as solvents and the spots were developed in an iodine chamber.

ISOLATION OF LUPEOL - FROM XANTHOXYLUM BUDRUNGA.

About 5 kilogram of air dried finely powdered bark of Xanthoxylum budrunga[33] was extracted with benzene in a Soxhlet extractor for 48 hours. The extract was cooled and the solvent was distilled off. The residual gummy mass was then dissolved in minimum volume of benzene, chromatographed over silica gel and then elution with Petrol-benzene (4:1) mixture ~~that~~ furnished lupeol — (10 gm). Lupeol was further purified by repeated crystallisation from chloroform-methanol mixture, m.p. 215°-16°C,

$[\alpha]_D = -33^\circ$ found to be identical with authentic sample of lupeol (Co-tlc, m.m.p. and Co-IR).

HYDROGENATION OF LUPEOL TO LUPANOL (69)

5.0 gm of lupeol was dissolved in 250 ml of distilled ethyl acetate and 1.6 gm of palladised charcoal (E. Merck) was added. Then it was reduced with hydrogen at atmospheric pressure. The catalyst was then filtered off, the solvent distilled out and the residue on crystallisation from chloroform-methanol mixture gave the fine crystals of lupanol (69) (4.5 gm), m.p. 207°-8°C, $[\alpha]_D = -17.8^\circ$, identical with authentic lupanol (Co-IR, m.m.p.).

DEHYDRATION OF LUPANOL 69 TO 2,3-DEHYDROLUPANE (70)

2.5 gms of lupanol was dissolved in 10 ml of distilled pyridine (Py) and 5 ml of distilled phosphorus oxychloride ($POCl_3$) was added. The mixture was then refluxed over water bath for 4 hours. It was then cooled, diluted with ice cold water cautiously to destroy excess of $POCl_3$ and extracted with ether and washed with water repeatedly till neutral, dried with anhydrous sodium sulphate (Na_2SO_4) and finally a yellowish white gummy mass was formed after distilling off solvent.

The gummy mass was then dissolved in minimum volume of benzene and chromatographed over silica gel (100 gms) column developed with petroleum ether and eluted with solvents as shown in the table below:

TABLE - VI

Eluent	Fraction of 50 ml each	Residue on distillation	M.P.
Petrol	1-5	Oil	
Petrol-Benzene (4:1)	6-12	Solid 1.4 gms	181-2°C

Further elution with more polar solvents did not afford any solid materials. Fractions 6-12 showing the same TLC were combined together and crystallised from $CHCl_3$-MeOH to afford needle shaped crystals of lupan-2, 3-ene (70) m.p. 186-8°C, $[\alpha]_D$ = +13.4°, ν_{max} 730 and 1640 cm^{-1}, produced yellow colour with tetranitro methane (TNM).

OXIDATION OF LUPAN-2(3)-ENE (70) WITH SELENIUM DIOXIDE IN TERTIARY BUTANOL CONTAINING HYDROGEN PEROXIDE.

A solution of lupan-2(3)-ene (1.0 gm) dissolved in t-BuOH (150 ml) containing SeO_2 (0.8 gm) and hydrogen H_2O_2 (2 ml) (30%) was refluxed over water bath for about 40 hours when black selenium metal was separated out indicating the completion of the reaction. The reaction mixture was cooled and then poured into ice cold water when a white solid appeared which was extracted with ether and the ethereal layer was washed three times with 5%

Na_2Co_3 solution followed by water repeatedly till neutral. The ether layer was dried over anhydrous Na_2So_4 and the filtrate on evaporation afforded a gummy mass of about 0.5 gm.

ISOLATION OF LUPAN-2,3-DIOL (71)

The neutral gum (0.5 gm) obtained as above was dissolved in minimum volume of benzene and chromatographed over silica gel (40 gms). The chromatograph was developed with petrol and eluted with following solvents as shown in table -VII below:

TABLE - VII

Eluent	Fractions of 50 ml each	Residue on distillation
1. Petrol	1-4	Oil
2. Petrol-benzene (3:2)	5-10	Oil
3. Petrol-benzene (1:4)	11-15	Oil
4. Benzene-chloroform (4:1)	16-20	Solid

Further elution with more polar solvents did not furnish any more solid materials. The fraction (16-20) were combined and then attempted crystallisation from $CHCl_3$-MeOH, m.p. 240°-$46^{\circ}C$ that afforded only a semi solid; its IR spectrum (Fig. 36) showed a broad peak at 3540 cm^{-1} indicating the presence of hydroxy group and hence it was acetylated.

ACETYLATION OF FRACTION (16-20) TO LUPAN 2β, 3α DIYL ACETATE (72)

The compound 71 (0.4 gm) was dissolved in 4 ml pyridine and 4 ml of acetic anhydride was added. The mixture was kept over water bath for 4 hours and then poured in ice cold water when a white solid separated out. It was filtered through suction, washed with water and dried. The dried mass was then chromatographed over silica gel column by elution with following solvents as shown in the table - VIII.

TABLE - VIII

Eluent	Fraction of 50 ml each	Residue on distillation
1. Petrol	1-3	Nil
2. Petrol-benzene (4:1)	4-6	Nil
3. Petrol-benzene (3:2)	7-10	White solid (0.35 gm)

Further elution did not afford any solid.

The fractions (7-10) were collected together and crystallised from $CHCl_3$-MeOH mixture giving colourless crystals m.p. 220-21°C, $[\alpha]_D$ = +23.4°, identified as lupan 2β, 3α diyl acetate (72).

ANALYSIS REPORT

Found :	C 77.13; H 10.01%
Calculated for $C_{34}H_{56}O_4$:	C 77.27; H 10.61%
IR (Nujol): ν_{max} (Fig. 36)	1750, 1270 and 1250 cm^{-1} (2 x CH_3-CO-O-)
Mass : m/e (Fig. 37)	528 (M^+, 86), 513(30), 488(43), 468(78), 408(100), 393(88), 365(52.5), 231, 191(86), 187(98), 123(94).
^1H NMR ($CDCl_3$): (δ in ppm) (Fig. 38)	0.77 and 0.84 (dd, 6H, CH_3-CH-CH_3, J = 7 Hz), 0.76, 0.91, 0.95, 0.98, 103 and 1.07 (6s, 18H, 6 x t-CH_3) 2.01 and 2.07 (2s, 6H, 2 x -OCOCH_3) 4.96(dd, 3H, AcO-CH-CH_2, J = 7 & 13 Hz), 5.07 (d, 2H, AcO-CH-CH-OAc, J = 7 Hz).

ISOLATION OF BETULINIC ACID FROM BISCHOFIA JAVANICA

About 5 Kg of air dried finely powdered bark of Bischofia Javanica[25] was extracted with benzene in a Soxhlet extraction for 36 hrs; then after distillation of the solvent gave a gummy mass which was then dissolved in ether. The ethereal solution was treated with aqueous alkali (20% NaOH) and the alkali layer was

separated from ether layer. (The ether layer containing neutral compounds was rejected). The alkali layer on acidification with dil HCl yielded a solid of betulinic acid which was filtered through suction, washed repeatedly with water till neutral and dried properly for further work.

ESTERIFICATION OF BETULINIC ACID

About 5 gms of betulinic acid was dissolved in ether to which an ice cold ethereal solution of diazo-nitroso methane prepared from nitroso methyl urea (8 g). The reaction mixture was kept over night in a freeze. It was acidified with few acetic acid to destroy excess of diazomethane, washed with water till neutral and the solvent was evaporated to give crude methyl ester of betulinic acid. The crude ester was dissolved in minimum volume of benzene and chromatographed over silica gel column with different eluents as shown in tabular form.

TABLE - IX

Eluent	Fraction on 50 ml each	Residue on distillation
1. Petrol	1-3	Nil
2. Petrol-benzene (4:1)	4-7	Nil
3. Petrol-benzene (3:2)	8-15	White solid (0.4 gm)

Further elution with more polar solvents did not afford any solid. The fractions (8-15) having same TLC were combined and crystallised from $CHCl_3$-MeOH, m.p. 223°C, $[\alpha]_D$ = +5°; IR : 3520 (-OH), 1735, 1260 ($-COOCH_3$), 1660-870 cm^{-1} ($=CH_2$), which was identical with authentic sample of methyl betulinate (Co, tlc, & m.m.p.).

HYDROGENATION OF METHYL BETULINATE

3.0 gm of methyl betulinate dissolved in distilled ethyl acetate was reduced with hydrogen gas in presence of palladised charcoal catalyst at atmospheric pressure. The catalyst was filtered off, solvent removed and the residue obtained was crystallised from $CHCl_3$-MeOH to give fine crystals of methyl dihydro betulinate 69a, m.p. 236-7°C, IR : 3500 (-OH), 1735, 1250 ($-COOCH_3$) cm^{-1}, identical with authentic sample (Co-tlc & m.m.p.).

DEHYDRATION OF METHYL DIHYDRO BETULINATE (69a)

3.0 gm of methyl dihydro betulinate 1a was dehydrated with pyridine (8 ml) and distilled $POCl_3$ (4 ml). After usual work up, the gummy mass was chromatographed over silica gel. On elution with Petrol-benzene (4:1) a solid was obtained, which was crystallised from $CHCl_3$-MeOH yiedling white crystals of 2,3 dehydro methyl dihydro betulinate (73), m.p. 228-9°C, IR : 1730, 1260 ($-COOCH_3$), 1640, 850 (-CH=CH-), gave yellow colour with TNM, identical with authentic sample (Co-tlc & m.m.p.).

OXIDATION OF 2,3-DEHYDRO METHYL DIHYDRO BETULINATE (73) WITH SeO_2 CONTAINING H_2O_2 IN TERTIARY BUTANOL.

1 gm of the compound 73 was dissolved in 150 ml of t-BuOH and refluxed with a mixture of SeO_2 (0.8 gm) and H_2O_2 (30% 2 ml) for 48 hours. After usual work up, the gummy mass obtained from the neutral ether was chromatographed over silica gel and eluted by different solvents with increasing polarity. A mixture of benzene : chloroform (1:1) afforded a gummy solid which could not be crystallised. The IR of the crude product indicated the presence of hydroxy function hence it was acetylated with Ac_2O-Py mixture.

The alkali layer was acidified with 20% HCl till slightly acidic. It was kept over night which produced no solid material and hence rejected.

ACETYLATION OF THE GUMMY SOLID

The gummy solid (0.6 gm) was acetylated with Ac_2O (10 ml) and Py (10 ml). The solid material obtained on usual work up was chromatographed over silica gel and on elution with benzene-petrol (4:1) mixture yielded a white solid. It was crystallised from $CHCl_3$: MeOH to afford white crystals, identified as 2β, 3α-diacetoxy methyl dihydro betulinate (75) m.p. 209-10°C from spectral studies.

ANALYSIS REPORT

Found :	C 72.8; H 9.10%
Calculated for $C_{35}H_{56}O_6$:	C 73.4; H 9.79%
IR : (Nujol) ν_{max} (Fig. 39)	1730, 1230 cm^{-1} ($-CO-CH_3$) 1720, 1220 cm^{-1} ($-CO-O-CH_3$)
Mass : (m/e (Fig. 40)	572 (M^+), 513, 512, 470, 452, 437, 411, 393, 377, 203, 191, 187 (100%).
^1H NMR ($CDCl_3$) : (δ in ppm) (Fig. 41)	0.74 & 0.85 (dd, 6H, $CH_3-CH-CH_3$) 0.90, 0.95, 0.96 & 1.05 (4s, 15H, 5 x t-CH_3), 2.01 & 2.06 (2s, 6H, 2 x $-O-CO-CH_3$), 3.64 (s, 3H, $-CO-O-CH_3$), 4.76 (dd, $-CH_2-CH-OAc$), 5.06 (d, ACO-CH-CH-oAc).

ISOLATION OF FRIEDELIN (36) FROM THE BARK OF QUERCUS SUBER CORK

5 Kg of finely powered dry cork, the bark of Quercus Suber[26] was extracted with benzene in a Soxhlet apparatus for 48 hours. After removal of the solvent, a white solid separated out. The solid was dissolved in minimum volume of benzene and chromatographed over silica gel. On elution with petrol-benzene (4:1) mixture it gave friedelin, which was crystallised from $CHCl_3$-MeOH mixture to give crystals of friedelin (36), m.p. 262°-3°C, $[\alpha]_D$ = -48.7°; IR : 1715 cm^{-1}.

REDUCTION OF FRIEDELIN (36) WITH SODIUM BOROHYDRIDE IN DIOXIDE.

3.0 gms of friedelin was dissolved in 200 ml of dioxane followed by 150 ml of methanol. Then 4.0 gms of $NaBH_4$ was added to the mixture and it was starred with a magnetic starrer of room temperature for 12 hours. The mixture was then diluted with water and acidified with dil HCl till slightly acidic when a solid separated out which was filtered with suction, washed with water till neutral and dried. It was then crystallised from $CHCl_3$-MeOH producing needle shaped crystals of friedelan 3β-ol (36a), m.p. 268°-9°C, IR : 3540 cm^{-1}, (-OH), identified by comparison with authentic sample (Co-tlc & m.m.p).

DEHYDRATION OF FRIEDELAN 3β-ol (77) BY PHOSPHORUS OXYCHLORIDE -PYRIDINE MIXTURE

2.5 gms of friedelan 3β-ol was dissolved in 10 ml of pyridine and 5 ml of phosphorus oxychloride was added. The mixture was then heated in water bath for 4 hours. After usual work up, the gummy mass obtained was chromatographed. On elution with petrol, a white solid mass (1.3 gm) was obtained, which was repeatedly crystallised from petrol-$CHCl_3$ to furnish white crystals of friedelan-3(4)-ene (76) m.p. 263°C; IR : 1650 and 800 cm^{-1}, gave yellow colour with TNM.

Found :	C 87.60%; H 12.01%
Calculated for $C_{30}H_{50}$:	C 87.73%; H 12.26%

OXIDATION OF FRIEDELAN-3(4)-ENE (76) WITH SELENIUM DIOXIDE IN TERTIARY BUTANOL CONTAINING HYDROGEN PEROXIDE.

A solution of friedel-3(4)-ene (1.0 gm) in t-butanol (150 ml) containing selenium dioxide (0.8 gm) and hydrogen peroxide (30%, 2 ml) was refluxed for 40 hours. After usual work up, the residual gummy mass obtained from neutral part was chromatographed by the following solvents as shown in table - X. The alkali wash on acidification did not afford any solid material hence it was rejected.

TABLE - X

Eluent	Fraction 50 ml each	Residue on distillation
1. Petrol	1-8	Oil
2. Petrol-benzene (4:1)	9-12	Solid (0.15g)
3. Petrol-benzene (4:1)	13-16	Solid (0.12g)
4. Petrol-benzene (4:1)	17-20	Nil
5. Benzene-chloroform (4:1)	21-25	Solid (0.4g)

Further elution did not afford any solid.

Fractions 9-12 were collected together and crystallised from petrol-$CHCl_3$, m.p. 263-4°C which were unreacted compound as identified by the authentic compound. Fractions 13-16 on crystallisation from $CHCl_3$-MeOH afforded needle shaped crystals m.p. 207-8°, $[\alpha]_D$ + 16°, IR : 1715 cm^{-1} identified as lupanone (31) from spectral analysis (^1H NMR & Mass) and comparison with authentic sample of lupanone (m.m.p., Co-tlc and Co-IR).

ANALYSIS REPORT

Found :	C 83.8%; H 12.0%
Calculated for $C_{30}H_{52}O$:	C 84.12%; H 12.14%
IR (Nujol): δ_{max} (Fig. 42)	1715 cm^{-1}
Mass : m/e (Fig. 43)	426 $(M)^+$, 411, 383, 355, 206, 205, 163, 109, 107(100).
^1H NMR ($CDCl_3$): (δ in ppm) (Fig. 44)	0.75 and 0.83 (dd, 6H, CH_3-CH-CH_3) 0.75, 0.98, 0.93, 1.02 and 1.07 (5s, 18H, 6 x $-CH_3$), 1.88 and 2.44 (2m, $-CH_2-CH_2-C=O$).

The fractions 21-25 were combined and crystallised from $CHCl_3$-MeOH, afforded a solid, m.p. 235-6°, $[\alpha]_D$ = +14.1°C, IR : 3340, 3480 cm^{-1} identified as friedelan 3β, 4α-diol (82).

ANALYSIS REPORT

Found :	C 80.21%; H 11.10%
Calculated for $C_{30}H_{52}O_2$:	C 81.08%; H 11.71%
IR (Nujol) ν_{max} (Fig. 45)	3340 and 3480 cm^{-1}, (-OH)
Mass : m/e (Fig. 46)	444 $(M)^+$, 429, 426, 411, 341, 273, 218, 208, 163 and 161.
^1H NMR ($CDCl_3$) (δ in ppm) (Fig. 47)	0.88, 0.97, 0.99, 1.00, 1.02, 1.12, 1.17 and 1.24 (8s, 24H, 8x $-CH_3$) 3.56 (AB_q; $-CH-CH_2-$, J = 3 & 7 Hz).

The compound (82) on acetylation with Ac_2O-Py mixture furnished the mono acetate (82a) m.p. 245-6°C, $[\alpha]_D$ = +24°, IR : 3500 (-OH), 1720, 1280 cm^{-1} ($-OCOCH_3$). Mass : 486 $(M)^+$; ^1H NMR : (δ) 0.9-1.3 (8s, 24H, 8x t-CH_3); 2.1 (s, 3H, $-OCOCH_3$), 4.75 (t, 1H, J = 3 Hz).

OXIDATION OF FRIEDELAN 3β, 4α-DIOL (82) BY JONES REAGENT

0.2 gm of friedelan 3β, 4α-diol (82) was dissolved in acetone (100ml) and Jones reagent was added dropwise with shaking until a faint orange colour persisted. The mixture was kept at room temperature for 1 hour, diluted with water and extracted with ether. The ether layer was washed thoroughly with water, dried with anhydrous Na_2SO_4 and the ether on evaporation gave a residue which was chromatographed and on elution with petrol-

benzene (2:3) mixture, a solid mass was obtained that was crystallised from $CHCl_3$-MeOH to give white crystals, m.p. 252-3°C; IR : 1715 cm^{-1} (C = O) and 3470 cm^{-1} (-OH), identified as friedelan 3-oxo 4 α-ol (83) from spectral analysis.

ANALYSIS REPORT

IR (Nujol) : ν_{max} (Fig. 48)	3470 (-OH), 1715 cm^{-1} (C=O)
Mass : m/e (Fig. 49)	442 (M^+), 436, 407, 365, 281, 267, 239, 225, 211, 146, 85.
^1H NMR ($CDCl_3$) (δ in ppm) (Fig. 50)	0.80, 0.87, 0.95, 1.00, 1.05, 1.16, 1.17 (7S, 3H each and 6H, 24H, 8-Me), 2.105 (dd, J = 3 Hz and J = 8 Hz), 2.23 and 2.96 (2m).

DEHYDRATION OF FRIEDELAN-3-OXO 4 α-ol (83)

0.2 gm of friedelan 3-oxo 4 α-ol (83) was dissolved in Py (4 ml) and $POCl_3$ (1 ml) was added. The mixture was then heated over water bath for 4 hours. After usual work up, the gummy mass was chromatographed and on elution with petrol, a solid mass was obtained which was crystallised from petrol, m.p. 208-9°C; UV (Fig. 51) : 220 nm for (CH_2 = CH-C=O), IR : 1690, 1550 and 850 cm^{-1} identified as friedelan 4(24)-en-3-one (80).

ISOLATION OF FRIEDELAN 3-OXO-27 ⟶ 15 α OLIDE (82) (Odolactone) FROM THE BARK OF GYNOCARDIA ODORATA.

About 5 Kg of dry finely powdered bark of Gynocardia Odorata[32] was extracted with benzene in a Soxhlet extractor for 48 hours. After the removal of solvent, the gummy mass was dissolved in minimum volume of benzene and chromatographed over silica gel. Elution with petrol-benzene (4:1) afforded a keto lactone, odolactone[32]. It was crystallised from $CHCl_3$-MeOH; m.p. $>320^{\circ}C$, $[\alpha]_D = -47.06^{\circ}$ that was found identical with odolactone.

Found :	C 79.0%; H 10.13%
Calculated for $C_{30}H_{46}O_3$:	C 79.30%; H 10.13%
IR (Nujol)	1700, 1750 cm^{-1}.

REDUCTION OF ODOLACTONE (82) BY $NaBH_4$ IN DIOXANE METHANOL

3.0 gm of odolactone was reduced with $NaBH_4$ (6 gm) in dioxane (200 ml) and methanol (150 ml). After usual work up, the gummy mass was dissolved in minimum volume of benzene and then chromatographed. Elution with petrol-benzene (1:4) gave a solid material which was crystallised from $CHCl_3$-MeOH to give epi-odolactone (3 β-hydroxy friedelan 27 ⟶ 15 α olide (82a), m.p. $>320^{\circ}C$, $[\alpha]_D = -2.48^{\circ}$. IR (Nujol) : ν_{max} 3520 (-OH) and 1740 cm^{-1} (γ-lactone).

Found : C 78.90%; H 10.52%

Calculated for $C_{30}H_{48}O_3$: C 78.95%; H 10.53%

Further elution with petrol-benzene (1:1) afforded another solid, which was crystallised from $CHCl_3$-MeOH to give pure compound identified as odolactone (friedelan 3 -hydroxy 27 15 olide 16a, m.p. 320°C, $[\alpha]_D$ = -12.14°; IR : 3480 cm^{-1} (-OH) and 1758 cm^{-1} (δ-lactone) by comparison with authentic sample (Co-tlc of C-IR).

DEHYDRATION OF 3β-HYDROXY FRIEDELAN 27 $\rightarrow$ 15 α OLIDE (82a) (EPI-ODOLACTONE)

2.0 gm of epi-odolactone was dissolved in pyridine (10 ml) and 4 ml of $POCl_3$ was added. The mixture was then heated for 4 hours over water bath and after usual work up the gummy mass obtained was chromatographed over silica gel. Elution with petrol-benzene (4:1) furnished a solid which was crystallised from $CHCl_3$-MeOH, m.p. $>$ 300°C; IR : 800 cm^{-1} for (-CH = CH-), gave yellow colour with TNM, identified as 3(4)-dehydro-friedelan 27 $\rightarrow$ 15 olide (81) from ^{1}H NMR and Mass spectra.

OXIDATION OF 3(4)-DEHYDRO ODOLACTONE (81) WITH SELENIUM DIOXIDE IN t-BuOH CONTAINING HYDROGEN PEROXIDE.

1.0 gm of 3(4) dehydro odolactone 14 was dissolved in t-Butanol and 0.8 gm of SeO_2 was added followed by H_2O_2 (30%,

4 ml). The mixture was refluxed for 40 hours and after usual work up a gummy mass was obtained from the neutral ethereal layer. Chromatography over silica gel and elution of the column with the following solvents as shown in table - XI. The alkali layer was acidified and no solid material was separated and hence rejected.

TABLE - XI

Eluent	Fraction 50 ml each	Residue on distillation
1. Petrol	1-5	Oil
2. Petrol-benzene (4:1)	6-10	Solid (0.1 gm)
3. Petrol-benzene (1:1)	11-15	Nil
4. Benzene (1:1)	21-28	Solid (0.45 gm)
5. Benzene-chloroform	16-20	Nil

Further elution did not afford any solid fractions (6-10) were collected and crystallised from $CHCl_3$-MeOH, which was found to be unreacted compound by comparison (Co-tlc & m.m.p) with the authentic sample.

Fractions (21-28) were combined and on repeated crystallisation from ethyl acetate-petrol, m.p. 270-71°C identified as friedelan 3β, 4α-dihydroxy 27 $\longrightarrow$ 15 olide (83) from spectral studies.

ANALYSIS REPORT

Found :	C 76.01%; H 10.24%
Calculated for $C_{30}H_{40}O_4$:	C 76.27%; H 10.16%
IR (Nujol) : ν_{max} (Fig. 52)	3500 and 3440 cm^{-1} (-OH)
Mass : m/e (Fig. 53)	472 (M, 22%), 436, 386, 385 (100), 123.
^1H NMR : ($CDCl_3$) (δ in ppm) (Fig. 54)	0.86, 0.94, 0.96, 0.99, 1.02, 1.05 and 1.21 (7S, 21H, 7x t-CH_3), 2.02 (t, 1H, J = 3 & 13.5 Hz), 3.54 (t, 1H, J = 3 Hz, 3α H), 4.34 (t, 1H, J = 3 Hz, 15β-H).
^{13}C NMR (Fig. 55)	See Table V

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