CHAPTER 4

EXPERIMENTAL

4.1 Instrumentation

Solvents used are AR grade except those used for bulk extractions (distilled) and HPLC methanol 99.8 %. Silica gel used for column chromatography, chromatotron and thin layer chromatography plates were silica gel 60, (230-400 Mesh ASTM) and silica gel 60 UV 254. Aluminium supported silica gel 60 UV 254 plates were also used for TLC.

Melting points were determined in open capillaries using a Gallenkamp melting apparatus and were uncorrected. Ultra-violet light (254 and 365 nm) used when visualised spots on TLC, followed by spraying with the Dragendroff's reagent for alkaloid screening.

For spectroscopic method :

UV Spectra were measured in methanol solution in a UV-Visible Spectrophotometer Model Shimadzu UV-160A.

IR Spectra were obtained on a Perkin Elmer 1600 double beam recording spectrometer (series FT) using chloroform or KBr pellets.

^1H NMR and ^{13}C NMR Spectra were recorded on Jeol GXX 270 MHz apparatus with $CDCl_3$ as solvent and tetramethylsilane as the internal standard.
Mass Spectra were determined on a Shimadzu QP 2000A mass spectrometer and Kratos MS 30.

4.2 Reagents

Mayer's Reagent

Solution A : 1.4 g mercury (II) chloride + 60 ml distilled water.
Solution B : 5.0 g potassium iodide + 10 ml distilled water.

Add solutions A and B together and made up the mixture up to 150 ml with distilled water.

Mayer test - A positive result is indicated by the formation of white percipitate (under acidic conditions).

Dragendroff's Reagent

Solution A : 0.85 g bismuth (III) nitrate + 10 ml glacial acetic acid + 40 ml distilled water.
Solution B : 8.0 g potassium iodide + 20 ml distilled water.
Stock solution : A mixture of equal volumes of solutions A and B.

Spray reagent : The stock solution (20 ml) was diluted in a mixture of 20 ml glacial acetic acid and 60 ml distilled water.

Dragendroff test - A positive result is indicated by the formation of orange spots (alkaloid).

4.3 Plant Materials

The bark of *Fissistigma lanuginosum* (Hk.f et Th) was collected 18 km from Dungun to Bukit Besi, Trengganu in July 1993. The bark of *Polyalthia hookerian king* was collected at Dungun in July 1993.

All these species were identified by the Phytochemical group of Chemistry Department , University Malaya with herbarium species number KL 4274 and KL 4264 respectively.

The botanical description of *Fissistigma lanuginosum* is as follows :

Fissistigma lanuginosum is a member of the tribe Xylopeae and is a climber. The young twigs are rusty-tomentose, tardily becoming glabrous and with numerous lenticels. The leaves are coriaceous, dark green and glossy above, often drying a silvery-grey, oblong or obovate - oblong, acute or less often slightly acuminate, base rounded, edges and midrib above rusty - pubescent, entire lower surface densely rufous-lanate but not shining.

The flowers are 1-4 in cymes, terminal and opposite to a leaf. The pedicels are stout, 1 cm. long, rufous-lanate with 2 amplexicaul, acute and lanate bracts. The sepals are 1-5 cm. long, rufous-lanate like the outer petals.

The petals are coriaceous, oblong-lanceolate, sub-acute, outer 3-3.5 cm. long, thickened at the edges, inner slightly smaller, glabrous or glabrescent, pink, concave at the base and triquetrous above. The stamens are numerous, 2 mm. long, connectives oblique and produced. The ovaries are 3 mm. long, slightly obovoid, curved on the outer side, densly hairy up to 1 mm. and glabrous. The ripe carpels are sub-globose, slightly narrowed to the base, 2 cm. in diameter, densly clustered, sub-sessile or with stalks 5 mm. long. The seeds are 4-6 in 2 rows, shining and dark brown.

The botanical description of *Polyalthia hookerian* is as follows :

Polyalthia hookerian belongs to the tribe Unoneae. It is a tree with 10-25 m. high. The young twigs are tawny-pubescent, later glabrous and rather coarsely striate. The leaves are membranous, shining, obovate-elliptic or oblanceolate, apex acute, base narrow and subcuncate or sometimes rounded, glabrous except for the midrib above and below. The nerves are 10-12 pairs, visible above, prominent beneath, nearly straight and running out of the edge, reticulations rather fine and lax, breadth 4-7.5 cm. petiole 5-7 mm. long and tomentose.

The pedicels are pubescent, about 2.5 cm. long with an amplexicaul bract near base. The sepals are ovate, acute, tomentose outside, glabrous inside and 3-4 mm. long.

The petals are pale yellow, sub-equal, obovate-oblong, obtuse, puberulous except at the base inside, slightly narrowed above base, 2.7-3 cm. long and 1.2 cm. wide at broadest part.

The stamens are 1-1.5 mm. long, numerous and with an orbicular top. The ovaries are oblong, puberulous and stigma obovate with sub-truncate apex. The ripe carpels are red, ovoid-oblong, glabrous, obtuse, 2-2.5 cm. long with stalks 3.5-4 cm. long. The seed is one with longitudinal circumferential groove.

4.4 Extraction of Plant Materials

(Fissistigma Lanuginosum* and *Polyalthia hookerian)

1.0 kg. of dried, grounded bark of the plant were first defatted with petroleum - ether (60-80^{0}C) for 24 hours. The petroleum - ether extracts were first taken up to dryness and then tested for alkaloid and flavonoid (using TLC).

The plant materials were dried up and then soaked with 10 % ammonia and left for overnight. They were then re - extracted with dichloromethane exhaustively by the Soxhlet extractor for about 17 hours followed by methanol. The supernatant obtained was concentrated under reduced pressure to a volume of about 500 ml. and each were examined for their alkaloid contains (using TLC and confirmed by spraying with Dragendroff's reagent).

Flavonoid was found only in petroleum-ether extracts of *Fissistigma lanuginosum*. Both dichloromethane and methanol extracts in both species contain alkaloid.

The dichloromethane extracts were repeatedly extracted with a solution of 5 % hydrochloric acid until Mayer negative. The combined extracts were then basified with concentrated ammonia solution to ca. pH 11 followed by re-extraction with dichloromethane. The dichloromethane extract was washed with distilled water and dried over anhydrous sodium sulphate and evaporated to dryness to give crude alkaloid.

The methanol extracts were evaporated to dryness under reduced pressure and the residue was dissolved in 5 % hydrochloric acid and filtered. The acid extracts were basified with ammonia and re-extracted with dichloromethane. The dichloromethane extracts were evaporated to dryness to give crude alkaloid.

Table 14 : Chemicals content in the bark of the plants

Plant	Chemicals Content	Weight	% Yield
Fissistigma Lanuginosum	flavonoid	18.17 g	1.82
Bark (1.0 kg.)	alkaloid	8.01 g	0.80
Polyalthia hookerian	alkaloid	3.89 g	0.39
Bark (1.0 kg.)			

4.5 Separation and Purification of the Flavonoids.

The petroleum - ether extract from *Fissistigma lanuginosum* was introduced to column chromatography over silica gel with dichloromethane and methanol (100:0, 99:1, 97:3, 95:5, 90:10) as solvents. A total of fractions of 15 ml. each

were collected. The fractions were groups into a series of fractions after monitoring with TLC. The fractions were re - chromatographed which were then separated by preparative TLC followed by recrystallization from dichloromethane to give pure flavonoids.

The flavonoids were not detected in *Polyalthia hookerian.*

4.6 Separation and Purification of the Alkaloids.

The crude alkaloids were subjected to chromatotron or column chromatography over silica gel using dichloromethane and methanol (100:0, 99:1, 98:2, 95:5, 90:10) and finally pure methanol were used as eluants. The fractions collected were groups into a series of fractions after monitoring with TLC. Each series were then treated separately to isolate and purify its alkaloid content by extensive column chromatography. The purity of the alkaloid isolated were controlled by a single spot on TLC using several solvent systems. Some compounds were resolved using HPLC with methanol as a solvent.

The results of the screening are shown in the tables.

Table 15 : Chromatography Results from Flavonoids Extracts of *Fissistigma Lanuginosum*

Fractions	Solvent system (CH_2Cl_2 : MeOH)	% Weight	Components
A	100 : 0 , 99 : 1	40.5	FL1 FL2 FL3
B	97 : 3 , 95 : 5 90 : 10	19.3	FL4 FL5

Table 16 : Colour of Spots on TLC and % of Weight Obtained in Flavonoid Extracts from *Fissistigma Lanuginosum*

Component	% Rf (98 : 2)	Detection VL	UV	% Yield
FL1	72.4	Or	Be	22.1
FL2	71.8	Or	Be	10.0
FL3	59.7	Y	Be	8.3
FL4	41.0	Y	Be	10.2
FL5	40.6	Y	Be	9.0

Table 17 : Chromatography Results from Alkaloid Extracts of *Fissistigma Lanuginosum*

Fractions	Solvent system (CH_2Cl_2 : MeOH)	% Weight	Components
A	100 : 0 , 99 :1 98:2	30.2	FL6 FL7 FL8 FL9
B	95 : 5 , 90 : 1 80 : 20	25.8	FL10 FL11

Table 18 : Colour of Spots on TLC and % of Weight Obtained of the Components in Alkaloids Extract from *Fissistigma Lanuginosum*

Components	% Rf (95 : 5)	Detection (colour) VL UV DR	% Yield
FL6	93.4	Y Be -	0.19
FL7	86.2	P Be -	1.87
FL8	85.8	P Be -	1.14
FL9	69.5	Y Be -	0.38
FL10	68.1	Y Be Or ,,,	0.71
FL11	51.4	P Be Or ,,,	0.36

Table 19 : Chromatography Results from Alkaloid Extracts of *Polyalthia Hookerian*

Fractions	Solvent system (CH_2Cl_2 : MeOH)	% Weight	Components
A	100 : 0 , 99 : 1	13.8	PH - 1
B	98 : 2 , 95 : 5	16.9	PH - 2 PH - 3

Table 20 : Colour of Spots on TLC and % of Weight Obtained in Alkaloid Extract from *Polyalthia Hookerian*

Component	% Rf (98 : 2)	Detection (colour) VL UV OR	% Yield
PH - 1	69.9	Y Be $Or_{,,,}$	0.22
PH - 2	60.2	Y Be $Or_{,,,}$	0.54
PH - 3	59.7	Y Be $Or_{,,,}$	0.41

4.7 Physical and Spectral Data of Flavonoids and Alkaloids.

4.7.1 Petroleum - ether extract of *Fissistigma lanuginosum*

Compound labelled : FL1 (Pedicin)

This compound crystallized as orange crystals from CH_2C1_2 with melting point 133-135°C (lit. 145°C).

UV : λ_{max} nm (MeOH) : 240 , 315

IR : $\upsilon_{max}cm^{-1}$ ($CHC1_3$) : 1635 , 1570

^1H NMR : δ ppm ($CDC1_3$) : 3.60 (3H,s,6'-OMe) , 3.75 (3H,s,3'-OMe)
4.05 (3H,s,4'-OMe) , 5.30 (1H,s,5'-OH)
6.75 (1H,d,J=15Hz,H-α)
7.26-7.32 (3H,m,H-3,H-4,H-5)
7.45-7.52 (2H,m,H-2,H-6)
7.85 (1H,d,J=15Hz,H-β) , 12.70 (1H,s,2'-OH)

^{13}C NMR : δ ppm ($CDC1_3$) : 61.4 (3'-OMe) , 61.7 (4'-OMe) , 62.4 (6'-OMe)
143.5 (C-6') , 134.7 (C-5') , 147.5 (C-4')
136.3 (C-3') , 152.5 (C-2') , 111.2 (C-1')
130.9 (C-4) , 128.9 (C-3,C-5) , 129.4 (C-2,C-6)
135.5 (C-1) , 144.3 (C-β) , 126.6 (C-α),
193.9 (CO).

MS : m/e (%) : 330 $[M]^+$(92) , 226 (100) , 211 (86)

Compound labelled : FL2 (2',5'-dihydroxy-3',4',6'-trimethoxydihydrochalcone)

This yellowish crystal crystallized from dichloromethane with melting point 86-88°C.

UV : λ_{max} nm (MeOH) : 281 , 361

IR : υ_{max} cm^{-1} ($CHCl_3$) : 1650

^1H NMR δ ppm ($CDCl_3$) : 3.88 (3H,s,6'-OMe) , 3.90 (3H,s,3'-Ome)

4.04 (3H,s,4'-OMe) , 3.40 (1H,t,J=7Hz,H-α)

3.05 (1H,t,J=7Hz,H-β) , 5.40 (1H,s,5'-OH)

7.20-7.40 (5H,m,H-2,3,4,5,6) , 12.85 (1H,s,2'-OH)

^{13}C NMR:δ ppm ($CDCl_3$) : 61.0 (6'-OMe) , 60.6 (4'-OMe) , 60.6 (3'-OMe)

146.5 (C-6') , 133.5 (C-5') , 143.0 (C-4') ,

135.7 (C-3'),151.2 (C-2') , 110.0 (C-1') ,

125.7 (C-4) , 141.0 (C-1) , 128.1 (C-3,C-5),

128.1 (C-2,C-6) , 30.2 (C-β), 44.7 (C-α),

205.4 (CO)

MS : m/e (%) : 332 $[M]^+$(100) , 227 (66) , 91 (40)

Compound labelled : FL3 (5,8-dihydroxy-6,7-dimethoxyflavone)

This compound crystallized as yellow crystalfrom dichloromethane with melting point 180-182°C.

UV : λ_{max} nm (MeOH) : 284 , 360

IR : υ_{max} cm^{-1} ($CHCl_3$) : 1658 , 1616 , 1593

^1H NMR : δ ppm ($CDCl_3$) : 3.98 (3H,s,6-OMe) , 4.15 (3H,s,7-OMe) ,

6.70 (1H,s,H-3) , 5.45 (1H,br,s,8-OH),

7.52-7.58 (3H,m,H-3',5',6'),

7.97-7.99 (2H,m,H-2',6'), 12.3 (1H,s,5-OH)

^{13}C NMR: δ ppm ($CDCl_3$) : 61.1 (6-OMe), 61.9 (7-OMe), 132.1 (C-4')

126.6 (C-3',C-5'), 129.2 (C-2',C-6'), 131.5 (C-1')

107.3 (C-10), 139.9 (C-9), 129.7 (C-8),

146.4 (C-7), 136.1 (C-6), 146.0 (C-5),

183.3 (C-4), 107.3 (C-3), 164.3 (C-2)

MS : m/e (%) : 314 $[M]^+$(100), 299 (100), 240 (56), 225 (48)

Compound labelled : FL4 (Fissistin)

This compound was isolated as a yellow gum from dichloromethane.

UV : λ_{max} nm (MeOH) : 288, 367

IR : υ_{max} cm^{-1} ($CHCl_3$) : 3535, 1629

^{1}H NMR : δ ppm ($CDCl_3$) : 1.75 (1H,s,H-12'), 1.80 (1H,s,H-11),

2.12 (1H,m,H-7'), 2.20 (1H,m,H-8'),

2.38 (1H,d,J=8Hz,H-5'), 2.50 (1H,m,H-2'),

3.25 (1H,ddd,J=10,J'=8,J''=8Hz,H-6'),

3.83 (3H,s,3-OMe), 4.03 (3H,s,4-OMe),

4.17 (3H,s,6-OMe), 5.20 (1H,t,J'''=7Hz,H-9'),

4.28 (1H,ddd,J=10Hz,J''''=5Hz,H-1'),

5.20 (1H,br,s,H-3'), 5.20 (1Hs,s,5-OH),

11.85 (1H,s,2-OH),

7.30-7.70 (5H,m,H-2",3",4",5",6")

^{13}C NMR : δ ppm ($CDCl_3$) : 61.3 (6-OMe), 61.8 (4-OMe), 60.9 (3-OMe),

126.2 (C-4"), 127.6 (C-3",C-5"),

128.3 (C-2",C-6") , 144.7 (C-1") , 17.8 (C-12') ,

25.8 (C-11') , 131.7 (C-10') , 124.2 (C-9') ,

26.5 (C-8') , 37.4 (C-7') , 44.0 (C-6') , 37.8 (C-5')

137.5 (C-4') , 119.2 (C-3') , 30.6 (C-2') ,

51.1 (C-1') , 146.3 (C-6) , 133.7 (C-5) ,

142.7 (C-4) , 135.9 (C-3) , 150.5 (C-2) ,

111.7 (C-1) , 210.2 (CO)

MS : m/e (%) : 466 $[M]^+$(92) , 227 (100)

Compound labelled : FL5 (Isofissistin)

This compound was also isolated as a yellow gum from dichloromethane.

UV : λ_{max} nm (MeOH) : 289 , 366

IR : υ_{max} cm^{-1} ($CHCl_3$) : 3528 , 1629

^1H NMR: δ ppm ($CDCl_3$) : 1.63 (1H,s,H-12') , 1.73 (1H,s,H-11') ,

2.14 (1H,m,H-8') , 2.08 (1H,m,H-7') ,

5.18 (1H,t,J"=7Hz,H-9) ,

3.18 (1H,ddd,J=10,J=10,J'"=5Hz,H-6') ,

2.36 (1H,m,H-5') , 5.56 (1H,br,s,H-4') ,

5.65 (1H,br,s,H-3') , 2.36 (1H,m,H-2') ,

4.30 (1H,ddd,J=10,J'=8,J'=8Hz,H-1') ,

7.03-7.28 (5H,m,H-2",3",4",5",6",) ,

3.73 (3H,s,3-OMe) , 3.93 (3H,s,4-OMe) ,

4.08 (3H,s,6-OMe) , 5.28 (3H,s,5-OH) ,

11.90 (1H,s,2-OH)

^{13}C NMR : δ ppm (CDCl$_3$) : 61.4 (6-OMe) , 61.8 (4-OMe) , 60.9 (3-OMe) ,
126.2 (C-4") , 127.6 (C-3",C-5") ,
128.2 (C-2",C-6") , 144.6 (C-1") , 17.9 (C-12') ,
25.8 (C-11') , 131.8 (C-10') , 120.5 (C-9') ,
26.6 (C-8') , 37.6 (C-7') , 43.7 (C-6') ,
34.6 (C-5') , 120.5 (C-4') , 136.5 (C-3') ,
33.5 (C-2') , 51.4 (C-1') , 146.3 (C-6) ,
133.7 (C-5) , 142.7 (C-4) , 136.0 (C-3) ,
150.6 (C-2) , 111.7 (C-1) , 210.2 (CO)

MS : m/e (%) : 466 $[M]^+$(100) , 227 (100)

4.7.2 Alkaloid extract of *Fissistigma Lanuginosum.*

Compound labelled : FL6 (3',4',6'-trimethoxy-2',5'-quino-chalcone)

This compound crystallized as yellow crystals with melting point 112-113^{o}C (lit. 113-114^{o}C).

UV : λ_{max} nm (MeOH) : 298 , 402

IR : υ_{max} cm^{-1} (CHCl$_3$) : 1681 , 1637 , 1600

^{1}H MNR : δ ppm (CDCl$_3$) : 3.95 (3H,s,3'-OMe) , 4.00 (3H,s,4'-OMe) ,
4.08 (3H,s,OMe) , 6.95 (1H,d,J=15Hz,H-α) ,
7.52 (1H,d,J=15Hz,H-β) ,
7.38-7.42 (3H,m,H-3',4',5') ,
7.54-7.60 (2H,m,H-2',6')

^{13}C NMR: δ ppm (CDCl$_3$) : 61.0 (3'-OMe) , 61.0 (4'-OMe) , 61.5 (6'-OMe) ,
182.4 (C-5') , 143.2 (C-4') , 144.6 (C-3') ,

179.2 (C-2'), 121.1 (C-1'), 128.9 (C-3,C-5),

129.2 (C-2,C-6), 131.4 (C-1), 128.1 (C-4),

147.0 (C-β), 128.1 (C-α), 191.3 (CO).

MS : m/e (%) : 328 $[M]^+$(100), 269 (37), 257 (30), 209 (50)

Compound labelled : FL7

This compound was afforded as violet amorphus solid from dichloromethane.

UV : λ_{max} nm (MeOH) : 300 (sh), 318, 511

IR : υ_{max} cm^{-1} ($CHCl_3$) : 3575, 1670, 1570

^{1}H NMR: δ ppm ($CDCl_3$) : 4.28 (1H,ddd,J=17Hz,J'"=5Hz,H-1'),

2.45 (1H,m,J=17Hz,H-2'),

5.55 (1H,d,J""=1.5Hz,H-3'), 2.02 (1H,m,H-5'),

3.20 (1H,ddd,J=10,J'=8,J'=8Hz,H-6'),

2.02 (1H,m,H-7'), 22.12 (1H,m,H-8'),

1.70 (1H,s,H-12'), 5.25 (1H,t,J'=7Hz,H-9'),

1.62 (1H,s,H-11'),

7.08-7.22 (5H,m,H-2",3",4",5",6"),

3.90 (3H,s,4-OMe),

11.55 (2H,s,3-NH_2), 7.55 (2h,s,6-NH_2)

^{13}C NMR: δ ppm ($CDCl_3$) : 59.7 (4-OMe), 126.0 (C-4"), 127.5 (C-3",C-5"),

128.3 (C-2",C-6"), 145.9 (C-1"), 17.8 (C-12'),

28.8 (C-11'), 132.7 (C-10'), 124.4 (C-9'),

26.6 (C-8'), 37.6 (C-7'), 43.3 (C-6'), 38.2 (C-5')

137.7 (C-4'), 119.5 (C-3'), 30.6 (C-2'),

49.0 (C-1'), 143.0 C-6), 177.2 (C-5),

156.1 (C-4), 131.7 (C-3), 169.7 (C-2),

104.7 (C-1), 204.6 (CO)

MS : m/e (%) : 434 $[M]^{+}$(100), 196 $[M^{+}]$ (100)

Compound labelled : FL8

This violet coloured crystals crystallized from dichloromethane with melting point 226-228°C.

UV : λ_{max}nm (MeOH) : 334, 508

IR : $\upsilon_{max}cm^{-1}$ ($CHCl_3$) : 3575, 1670, 1543

^{1}H NMR : δ ppm ($CDCl_3$) : 3.90 (3H,s,4'-OMe), 7.20-7.35 (3H,m,H-3,4,5)

7.70-7.77 (2H,m,H-2,6), 8.63 (1H,d,J=16Hz,H-β)

8.10 (1H,d,J=16Hz,H-α)

^{13}C NMR : δ ppm ($CDCl_3$) : 59.7 (4'-OMe), 146.3 (C-6'), 179.2 (C-5')

159.1 (C-4'), 133.5 (C-3'), 169.9 (C-2')

105.8 (C-1'), 130.5 (C-4), 129.7 (C-3,5)

129.2 (C-2,6), 136.9 (C-1), 141.2 (C-β)

129.2 (C-α), 189.5 (CO).

MS : m/e (%) : 298 $[M]^{+}$ (100), 221 (68), 103 (100)

Compound labelled : FL9

This compound crystallized as violet amorphous solid with melting points 190-192°C.

UV : $\lambda_{max}nm^{-1}$ (MeOH) : 300 (sh), 328, 512.

IR : $\upsilon_{max}cm^{-1}$ ($CHCl_3$) : 3575 , 1670 , 1612 , 1562.

^1H NMR δppm ($CDCl_3$) : 4.01 (3H,s,4'-OMe) ,

7.42-7.50 (3H,m,H-3,4,5) ,

7.53 (2H,m,H-2,6) ,3.28 (1H,t,J=&Hz,H-β).

^{13}C NMR :δppm ($CDCl_3$) : 59.4 (4'-OMe) , 145.8 (C-6') , 178.6 (C-5') ,

165.5 (C-4') , 136.5 (C-3') , 169.8 (C-2') ,

104.8 (C-1') , 126.3 (C-4) , 129.2 (C-3,5) ,

128.9 (C-2,6) , 145.9 (C-1) , 31.4 (C-β) ,

47.7 (C-α), 200.1 (CO).

ME : m/e (%) : 300 $[M]^+$ (100) , 196 (72) , 91 (60).

Compound labelled : FL10 (Liriodenine)

This yellowish compound had m.p 268-270°C (lit. 275 - 276 ° C).

UV : $\lambda_{max}nm^{-1}$ (MeOH) : 266 , 271 , 314 , 412.

IR : $\upsilon_{max}cm^{-1}$ ($CHCl_3$) : 1664.

^1H NMR :δppm ($CDCl_3$) : 6.35 (2H,s,OCH_2) , 7.14 (1H,s,H-3) ,

7.60-7.76 (2H,m,H-9,10) ,

7.74 (1H,D,J=5.4Hz , H-4) ,

8.54-8.62 (2H,m,H-8,11) ,

8.87 (1H,d,J=5.4Hz,H-5)

MS : m/e (%) : 275 $[M]^+$ (100) , 247 , 189 , 162.

Compound labelled : FL11 (Lanuginosine)

This yellowish needless crsytallized from dichloromethane melted with decomposition at 303-306^{o}C.

UV : $\lambda_{max}nm^{-1}$ (MeOH) : 247 , 272 , 424

IR : $\upsilon_{max}cm^{-1}$ ($CHCl_3$) : 1665

^1H NMR : δppm ($CDCl_3$) : 3.99 (3H,s,OCH_3) , 6.34 (2H,s,OCH_2O) ,
7.14 (1H,s,H-3) , 7.24 (1H,dd,J 3Hz,H-10) ,
7.75 (1H,d,J=5.8Hz,H-4) ,
8.02 (1H,d,J=3Hz,3-8) ,
8.56 (1H,d,J=9Hz,H-11) ,
8.88 (1H,d,J=5.8Hz,H-5)

MS : m/e (%) : $[M]^+$ (100) , 275 , 247 , 176.

4.7.3 Alkaloid extract of *Polyalthia Hookerian.*

Compound labelled : PH1 (Lysicamine)

This yellowish amorphus solid crystallized from dichlormethane with m.p 206-208^{o}C (lit. 210-211^{o}C).

UV : $\lambda_{max}nm^{-1}$ (MeOH) : 237 , 274 , 305 , 402

IR : $\upsilon_{max}cm^{-1}$ ($CHCl_3$) : 1667

^1H NMR : δppm ($CDCl_3$) : 4.02 (3H,s,OCH_3) , 4.11 (3H,s,OCH_3) ,
7.58-7.81 (2H,m,H-9,10) ,

8.61 (1H,dd,J=8Hz,J'=1Hz,H-8),

9.18 (1H,dd,J"=8Hz,J'"=1Hz,H-11),

8.91 (1H,d,J=5.4Hz,H-4),

7.77 (1H,d,J,5.4Hz,H-5)

MS : m/e (%) : 291 $[M]^+$ (100) , 276 , 248 , 233 , 205 ,177.

Compound labelled : PH2 (Liriodenine).

This compound crystallized as yellow needless crystal.

UV : $\lambda_{max}^{}nm^{-1}$ (MeOH) : 264 , 270 , 316 , 409.

IR : $\upsilon_{max}cm^{-1}$ ($CHCl_3$) : 1665

^1H NMR : δppm ($CDCl_3$) : 6.33 (2H,s,OCH_2O) , 7.14 (1H,s,H-3),

7.58-7.77 (2H,m,H-9,10),

7.72 (1H,d,J=5.4Hz,H-4),

8.54-8.60 (2H,m,H-8,11),

8.88 (1H,d,J=5.4Hz,H-5)

MS : m/e (%) : 275 $[M]^+$ (100)

Compound labelled : PH 3 (Atherospermidine).

This compound afforded as a deep yellow crystal with m.p 280-282°C

(lit. 284-285°C).

UV : $\lambda_{max}nm^{-1}$ (MeOH) : 248 , 280 , 315 (sh) , 435

IR : $\upsilon_{max}cm^{-1}$ ($CHCl_3$) : 1664

^1H NMR : δ ppm ($CDCl_3$) : 4.26 (3H,s,OCH_3) , 6.26 (2H,s,OCH_2O) ,

7.27-7.76 (2H,m,H-9,10) ,

8.08 (1H,d,J=5.4Hz.H-5) ,

8.39-8.51 (2H,m,H-8,11) ,

8.85 (1H,d,J=5.4Hz,H-4)

MS : m/e (%) : 305 $[M]^+$, 290 , 275 , 262 , 260 , 234.

KEY FOR TABLE

Abbreviation

1. Solvent / solvent system

   90 : 10 : CH_2Cl_2 : MeOH

   100 : 0 : CH_2Cl_2

2. Detection

   VL : Visible light

   UV : Ultra violet light

   DR : Dragendorff's reagent

3. Colour

   Y : Yellow

   Be : Blue

   Or : Orange

   P : Purple

4. Intensity of colour

   +++ : Strong

   ++ : Medium

   + : Weak