

## SUMMARY

The present thesis entitled "Isolation and Characterization of Natural Products", consists of six chapters. The first chapter includes a critical review of the chemistry of coumarins, triterpenic and flavonoidic compounds and high-lighted the recent advances in the analytical techniques applied to the isolation and structure elucidation of natural products.

The chapters second to sixth deal with the isolation and characterization of the naturally occurring compounds from the following five medicinally important plants.

- A. Garcinia munnii, (Guttifereae), (Leaves).
- B. Rhus alata, (Anacardiaceae), (Bark).
- C. Bergenia ligulata, (Saxifragaceae), (Roots).
- D. Ficus lyrata, (Moraceae), (Leaves).
- E. Cassia siamea, (Fabaceae), (Leaves).

### Chapter-II

Following four compounds have been isolated and characterized from

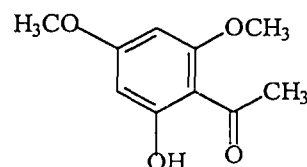
#### Garcinia munnii

##### 1. 2, 4-Dimethoxy-6-hydroxy acetophenone

M.F.  $C_{10}H_{12}O_4$

M.P. 149 °C.

M.W. 196



Crystallized from benzene-chloroform as white shining crystals.

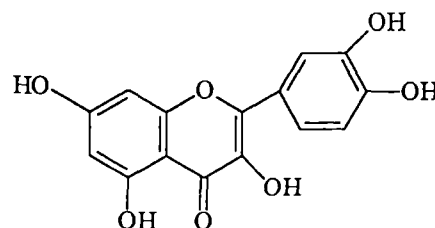
Structure elucidation is based on the basis of chemical reaction, IR, UV,  $^1H$ -NMR,  $^{13}C$ -NMR and MS.

##### 2. Quercetin

M.F.  $C_{15}H_{10}O_7$

M.P. 311-12 °C.

M.W. 302



Crystallized from chloroform-methanol as yellow shining crystals.

$Ac_2O$ /Pyridine (mild)  $\rightarrow$  pentaacetate

M.P. 194-95 °C

Crystallized from ethylacetate as cream coloured crystals.

Me<sub>2</sub>SO<sub>4</sub>/ acetone (mild) → pentamethyl ether

M.P. 151-52 °C

Crystallized from methanol-ethylacetate as colourless needles.

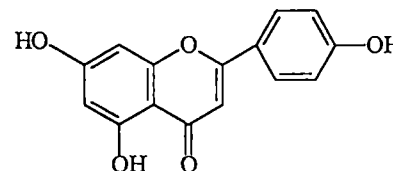
Structure elucidation is based on the basis of chemical reaction and spectral studies, IR, UV, <sup>1</sup>H-NMR and MS.

### 3. 5,7,4'-Trihydroxyflavone (Apigenin)

M.F. C<sub>15</sub>H<sub>10</sub>O<sub>5</sub>

M.P. 352 °C.

M.W. 270



Crystallized from benzene-acetone as yellow crystals.

Ac<sub>2</sub>O/Pyridine (mild) → triacetate

M.P. 183-84 °C

Structure elucidation is based on the basis of chemical reaction and spectral studies, IR, UV, <sup>1</sup>H-NMR and MS.

### 4. I-3, II-3, I-5, II-5, I-7, II-7, I-4', II-4'-Octahydroxy [I-2', II-2'] biflavone.

( new compound)

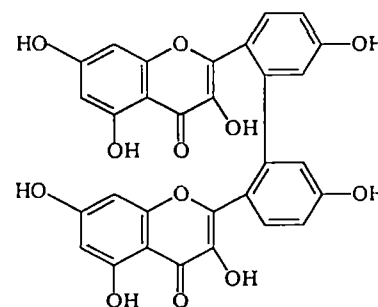
Me<sub>2</sub>SO<sub>4</sub>/ acetone (mild) → octamethyl ether

M.F. C<sub>38</sub>H<sub>34</sub>O<sub>12</sub>

M.P. 189-90 °C.

M.W. 682

Crystallized from chloroform-methanol  
as white crystals.



Structure elucidation is based on the basis of chemical reaction and spectral studies, IR, UV, <sup>1</sup>H-NMR, <sup>13</sup>C-NMR and MS.

### Chapter-III

Following nine compounds have been isolated and characterized from

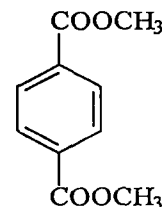
#### Rhus alata

##### 1. Dimethylester of terephthalic acid.

M.F.  $C_{10}H_{10}O_4$

M.P. 138 °C

M.W. 194



Crystallized from chloroform-methanol as white needles.

Structure elucidation is based on the spectral studies, IR, UV,  $^1H$ -NMR and MS.

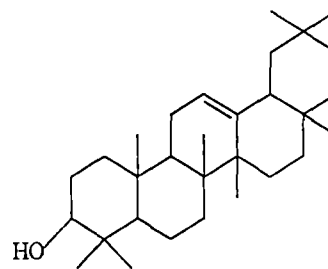
##### 2. $\beta$ -Amyrin

M.F.  $C_{30}H_{50}O$

M.P. 198 °C

M.W. 426

$[\alpha]_D^{19} +88.4^\circ$  ( $CDCl_3$ )



Crystallized from chloroform-methanol as white needles.

$Ac_2O/Pyridine$  (mild)  $\rightarrow$  monoacetate

M.P. 241-42 °C

$[\alpha]_D^{23} +68.9^\circ$  ( $CDCl_3$ )

Crystallized from chloroform-methanol as colourless crystals.

Structure elucidation is based on chemical reaction and spectral studies, IR,  $^1H$ -NMR and MS.

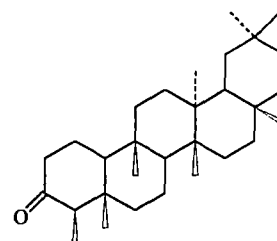
##### 3. Freidelin

M.F.  $C_{30}H_{49}O$

M.P. 262-64 °C

M.W. 426

$[\alpha]_D^{23} -29.4^\circ$  ( $CDCl_3$ )



Crystallized from chloroform-methanol as white needle-shaped crystals.

Structure elucidation is based on the spectral studies, IR and  $^1H$ -NMR and MS.

#### 4. Lupeol

M.F.  $C_{30}H_{50}O$

M.P. 214-15 °C

M.W. 426

$[\alpha]_D^{20} + 23.64^\circ$  ( $CHCl_3$ )

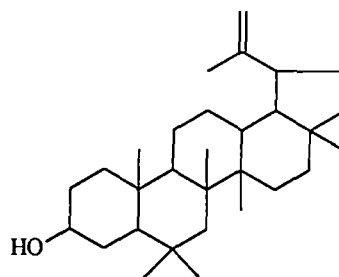
Crystallized from methanol-chloroform.

$Ac_2O/Pyridine$  (mild) → monoacetate

M.P. 218-20 °C

Crystallized from chloroform-methanol as colourless flakes.

Structure elucidation is based on the chemical reaction and spectral studies, IR and  $^1H$ -NMR.



#### 5. $\beta$ -Sitosterol

M.F.  $C_{29}H_{50}O$

M.P. 136-37 °C

M.W. 414

$[\alpha]_D -32.1^\circ$  ( $CDCl_3$ )

Crystallized from chloroform-methanol as white needle-shaped crystals.

$Ac_2O/Pyridine$  (mild) → monoacetate

M.P. 114-15 °C

$[\alpha]_D^{17} -48.5^\circ$  ( $CHCl_3$ )

Crystallized from chloroform-methanol as colourless flakes.

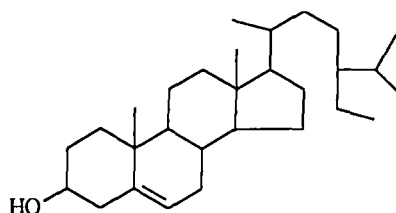
Benzoyl chloride/ Pyridine (mild) → Benzoate.

M.P. 145-46 °C

$[\alpha]_D^{17} -7.52^\circ$

Crystallized from methanol.

Structure elucidation is based on the basis of chemical reactions and spectral studies, IR,  $^1H$ -NMR and MS.



**6. Oleanolic acid**M.F.  $C_{30}H_{48}O_3$ 

M.P. 299-300 °C

Ac<sub>2</sub>O/Pyridine (mild) → monoacetate

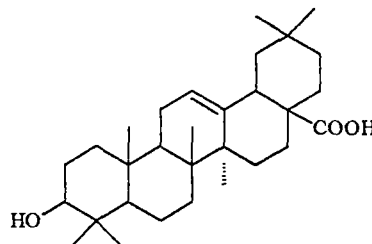
M.P. 258-60 °C.

Solid crystals from methanol.

Diazomethane → monomethylester

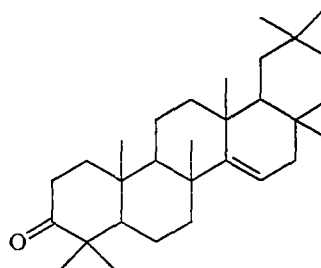
M.P. 220-21 °C.

Crystallized from methanol-chloroform as colourless shining plates.

Structure elucidation is based on the chemical and spectral studies, IR and <sup>1</sup>H-NMR.**7. Taraxerone**M.F.  $C_{30}H_{48}O$ 

M.P. 240 °C

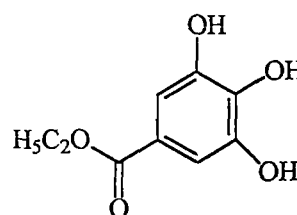
M.W. 424

Crystallized from chloroform-methanol  
as colourless needles.Structure elucidation is based on the spectral studies, IR, <sup>1</sup>H-NMR and MS.**8. Ethyl gallate**M.F.  $C_9H_{10}O_5$ 

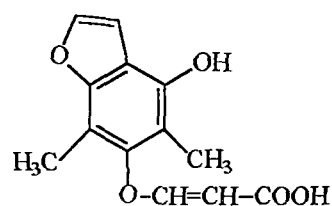
M.P. 155 °C

M.W. 195

Crystallized from methanol as reddish buff needles.

Structure elucidation is based on the spectral studies, <sup>1</sup>H-NMR and MS.**9. (2 E)-3-(4-hydroxy-5, 7-dimethylbenzo[3, 4-b] furan-6-yloxy)-prop-2-enoic acid.****(new compound)**M.F.  $C_{13}H_{12}O_5$ 

M.P. 160-62 °C



M.W. 248

Crystallized from chloroform-methanol as white crystals.

Structure elucidation is based on the spectral studies, IR,  $^1\text{H-NMR}$ ,  $^{13}\text{C-NMR}$  and MS.

### Chapter IV

Following six compounds have been isolated and characterized from

#### Ficus lyrata

##### 1. $\beta$ - Sitosterol-D-glucoside

M.F.  $\text{C}_{35}\text{H}_{60}\text{O}_6$

M.P. 282 °C.

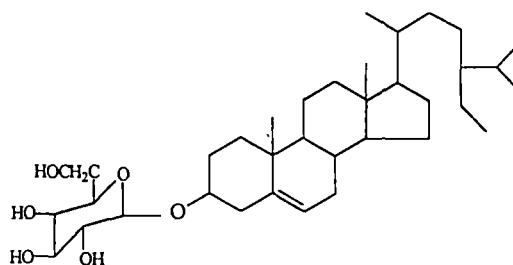
Crystallized from chloroform-methanol as colourless crystals.

$\text{Ac}_2\text{O/Pyridine}$  (mild)  $\square$  tetraacetate

M.P. 166 °C

Crystallized from methanol-chloroform as colourless crystals.

Structure elucidation is based on the basis of chemical reaction and spectral studies, IR,  $^1\text{H-NMR}$  and MS.



##### 2. 4-Methoxychalcone

(new compound)

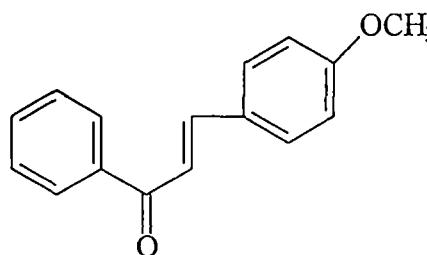
M.F.  $\text{C}_{16}\text{H}_{14}\text{O}_2$

M.P. 59-60 °C.

M.W. 238

Crystallized from chloroform-methanol as light cream crystals.

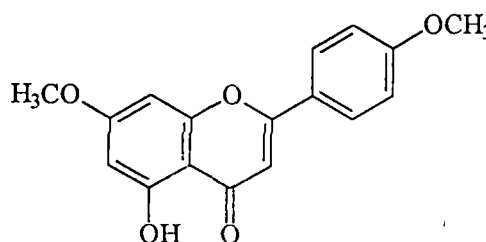
Structure elucidation is based on the basis of IR, UV,  $^1\text{H-NMR}$ , MS and GC-MS.



##### 3. 7, 4'-Dimethoxy apigenin

M.F.  $\text{C}_{17}\text{H}_{14}\text{O}_5$

M.P. 185-87 °C.



M.W. 298

Crystallized from chloroform-methanol as white crystals.

Structure elucidation is based on the basis of IR, UV,  $^1\text{H-NMR}$  and MS.

4. **5, 7, 4'-Trihydroxy-2', 3', 6'-trimethoxyisoflavone**

M.F.  $\text{C}_{18}\text{H}_{16}\text{O}_8$

M.P. 198-200°C.

M.W. 360

Crystallized from chloroform-methanol as pale yellow plates.

$\text{Ac}_2\text{O}$ /Pyridine (mild)  $\rightarrow$  triacetate

M.P. 132 °C

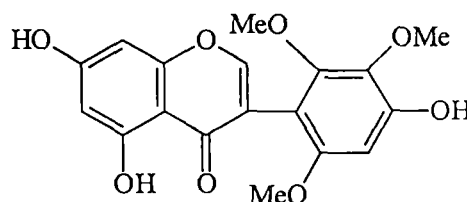
Crystallized from aq. ethanol as colourless needles.

$\text{Me}_2\text{SO}_4$ / acetone (mild)  $\rightarrow$  hexamethyl ether

M.P. 156 °C.

Crystallized from ethylacetate as colourless shining crystals.

Structure elucidation is based on the basis of chemical reaction, IR, UV,  $^1\text{H-NMR}$ ,  $^{13}\text{C-NMR}$  and MS.



5. **Acacetin-7-glucoside**

M.F.  $\text{C}_{22}\text{H}_{22}\text{O}_{10}$

M.P. 255 °C.

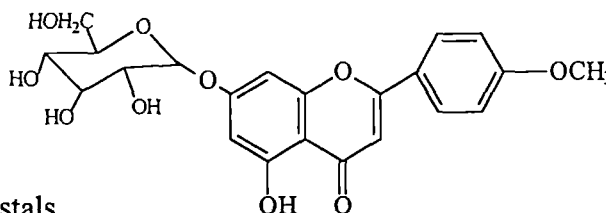
Crystallized from chloroform-methanol as cream coloured crystals.

$\text{Ac}_2\text{O}$ /Pyridine (mild)  $\rightarrow$  pentaacetate.

M.P. 204 °C

Crystallized from chloroform-methanol as colourless needles.

Structure elucidation is based on basis of chemical reaction and spectral studies, IR, UV and  $^1\text{H-NMR}$ .



**6. Acacetin-7-O-neohesperidoside**

M.F.  $C_{28}H_{32}O_{14}$

M.P. 266-68 °C.

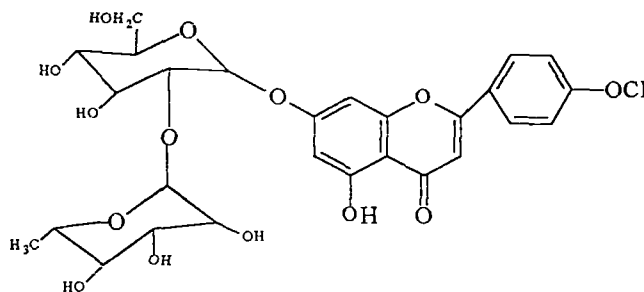
Crystallized from chloroform-methanol as white crystals.

$Ac_2O/Pyridine$  (mild)  $\rightarrow$  heptaacetate.

M.P. 216-20 °C

Crystallized from methanol as light cream crystals.

Structure elucidation is based on the basis of chemical reactions, IR, UV,  $^1H$ -NMR and MS.



**Chapter-V**

Following six compounds have been isolated and characterized from

**Bergenia ligulata**

**1. Lanost-7(8)-en-3 $\beta$ -formyloxy-6 $\beta$ -acetoxy-11 $\beta$ , 15 $\beta$ -diol-26-heptanoxy-27-oic acid.**

M.F.  $C_{40}H_{64}O_{10}$

M.P. 161-62 °C

M.W. 702

Crystallized from chloroform-methanol

$Ac_2O/Pyridine$  (mild)  $\rightarrow$  diacetyl product.

M.P. 110 °C.

M.F.  $C_{44}H_{69}O_1$

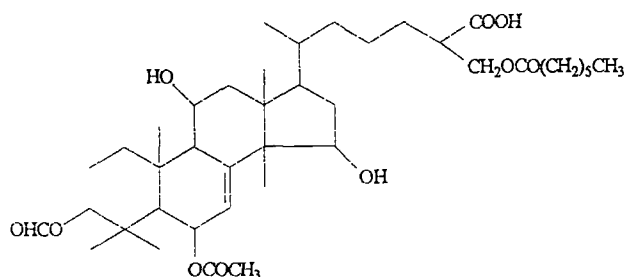
Crystallized from chloroform-methanol as white needles.

$CH_2N_2$  / acetone  $\rightarrow$  monomethylester

M.P. 114-15 °C.

M.F.  $C_{41}H_{66}O_{10}$

Crystallized from chloroform-methanol as colourless needles.





Structure elucidation is based on the basis of chemical and spectral studies, IR, UV,  $^1\text{H-NMR}$  and FABMS.

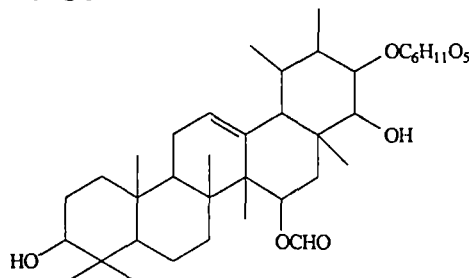
**2. Urs-12-en-3 $\beta$ -22 $\alpha$ -diol-15 $\beta$ -formyl-21 $\beta$ -glycoside**

M.F.  $\text{C}_{37}\text{H}_{10}\text{O}_{10}$

M.P. 190-91  $^\circ\text{C}$ .

M.W. 664

Crystallized from chloroform-methanol as white needles.



$\text{Ac}_2\text{O}$  /Pyridine (mild)  $\rightarrow$  peracetylated product

M.P. 125  $^\circ\text{C}$ .

Crystallized from chloroform-methanol as white needles.

Structure elucidation is based on chemical and spectral studies, IR, UV,  $^1\text{H-NMR}$  and FABMS.

**3. 9, 11-Seco-lanost-20 (22)-en-3 $\beta$ -formyl-18-phenoxyoate-12 $\beta$ -D-glucose**

M.F.  $\text{C}_{43}\text{H}_{66}\text{O}_{10}$

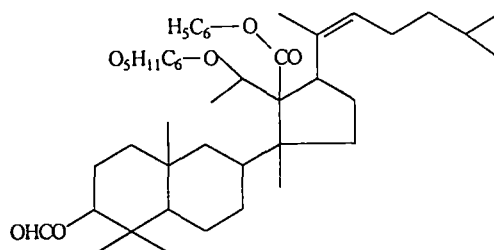
M.P. 210-11  $^\circ\text{C}$ .

M.W. 742

Solid crystals from methanol.

Structure elucidation is based on

Spectral studies, IR, UV,  $^1\text{H-NMR}$  and FABMS.



**4. Sumatrol**

M.F.  $\text{C}_{23}\text{H}_{20}\text{O}_5$  (OMe) $_2$

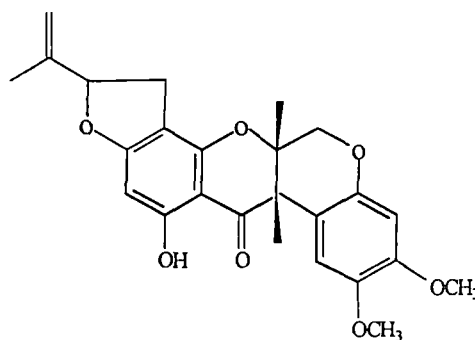
M.P. 190-92  $^\circ\text{C}$ .

Crystallized from acetone as colourless needles.

Hydroxylamine/ HCl (mild)  $\rightarrow$  Oxime.

M.P. 245-46  $^\circ\text{C}$ .

Crystallized from alcohol as colourless needles.



Structure elucidation is based on chemical and spectral studies, IR, UV and  $^1\text{H-NMR}$ .

**5. 5'-Methylepidictyol-3'-O- $\beta$ -D-galactopyranosyl (1  $\rightarrow$  4)- $\alpha$ -L-rhamnopyranose.**

M.F.  $\text{C}_{28}\text{H}_{34}\text{O}_{15}$

M.P.  $>250\text{ }^\circ\text{C}$ .

Crystallized from ethanol

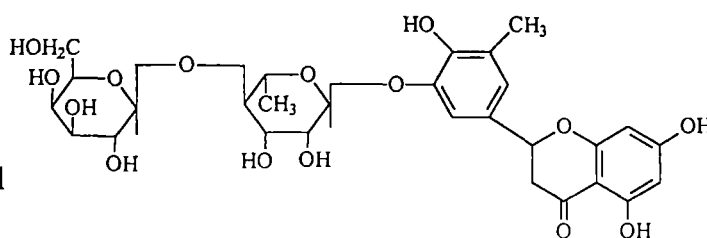
as pale yellow needles.

$\text{Ac}_2\text{O}$  /Pyridine (mild) $\rightarrow$  nonaacetate

M.P.  $85-86\text{ }^\circ\text{C}$

Crystallized from chloroform-methanol as colourless needles.

Structure elucidation is based on the basis of chemical and spectral studies, IR, UV,  $^1\text{H-NMR}$  and MS.



**6. Bergenin**

M.F.  $\text{C}_{13}\text{H}_{15}\text{O}_9$

M.P.  $237-38\text{ }^\circ\text{C}$ .

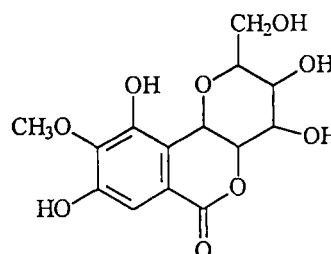
Crystallized from methanol as white crystals.

$\text{Ac}_2\text{O}$  /Pyridine (mild) $\rightarrow$  pentaacetate product.

M.P.  $207-08\text{ }^\circ\text{C}$ .

Crystallized from methanol as needle-shaped crystals.

Structure elucidation is based on the basis of IR and UV.



**Chapter-VI**

Following five compounds have been isolated and characterized from

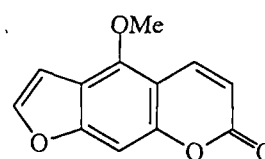
**Cassia alata**

**1. Bergapten**

M.F.  $\text{C}_{12}\text{H}_8\text{O}_4$

M.P.  $190-91\text{ }^\circ\text{C}$ .

M.W. 216



Crystallized from chloroform-methanol as light yellow crystals.

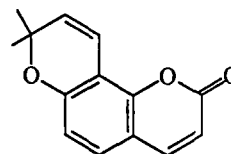
Structure elucidation is made on the basis of IR, UV,  $^1\text{H-NMR}$  and MS.

## 2. Seslin

M.F.  $\text{C}_{14}\text{H}_{12}\text{O}_3$

M.P. 119-20 °C.

M.W. 228



Crystallized from chloroform-methanol as light yellow crystals.

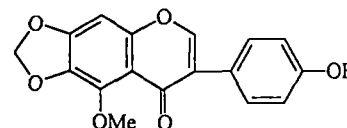
Structure elucidation is based on the basis of IR, UV,  $^1\text{H-NMR}$  and MS.

## 3. 5-Methoxy-4'-hydroxy-6,7-methylenedioxyisoflavone

M.F.  $\text{C}_{17}\text{H}_{12}\text{O}_6$

M.P. 258-60 °C.

M.W. 312



Crystallized from chloroform-methanol as white crystals.

Ac<sub>2</sub>O/Pyridine (mild) → monoacetate

M.F.  $\text{C}_{19}\text{H}_{14}\text{O}_7$

M.P. 189 °C

Crystallized from aq. ethanol as colourless needles.

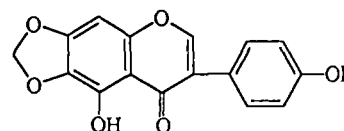
Structure elucidation is based on the basis of IR, UV,  $^1\text{H-NMR}$  and MS.

## 4. 5, 4'-Dihydroxy-6, 7-methylenedioxyisoflavone

M.F.  $\text{C}_{16}\text{H}_{10}\text{O}_6$

M.P. 236-37 °C.

M.W. 298



Crystallized from chloroform-methanol as cream coloured crystals.

Ac<sub>2</sub>O/Pyridine (mild) → diacetate

M.F.  $\text{C}_{20}\text{H}_{14}\text{O}_8$

M.P. 187 °C

Crystallized from ethanol as colourless needles.

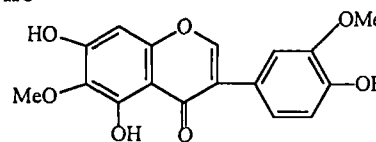
Structure elucidation is based on the basis of chemical reaction and spectral studies, IR, UV,  $^1\text{H-NMR}$  and MS.

**5. 5, 7, 4'-Trihydroxy-6, 3'-dimethoxyisoflavone**

M.F.  $\text{C}_{17}\text{H}_{14}\text{O}_7$

M.P. 255-56 °C.

M.W. 330



Crystallized from chloroform-methanol as cream coloured crystals.

$\text{Ac}_2\text{O/Pyridine}$  (mild)  $\rightarrow$  triacetate

M.F.  $\text{C}_{23}\text{H}_{20}\text{O}_{10}$

M.P. 135 °C

Crystallized from ethanol as colourless needles.

Structure elucidation is based on the basis of chemical reaction and spectral studies, IR, UV,  $^1\text{H-NMR}$  and MS.