Experimental
Apigenin derivatives used in these studies are known compounds and were prepared by standard literature procedure. 4'-hydroxy-5,7-dimethoxyflavone have been prepared by debenzylation of 4'-benzyloxy-5,7-dimethoxyflavone with hydrochloric acid in acetic acid. 5-hydroxy-7,4'-dimethoxyflavone have been prepared by demethylation of 5,7,4'-trimethoxyflavone by AlCl₃ in nitrobenzene. All reactions were carried out under nitrogen atmosphere except few of them which are under normal atmospheric conditions. All melting points were recorded on a Kofler microscopical hot stage and are uncorrected. Nuclear magnetic resonance spectra were run in CDCl₃ on a JEOL 4H-60 and 100 MHz instruments using tetramethylsilane as internal standard. Contact shift studies were done with Eu(fod)₃ complex. Mass spectra (MS) were determined on a JEOL-03IG double focusing high resolution mass spectrometer.

Analytical and preparative thin layer chromatography were performed on silica gel (G) (Merck) or silica gel (BDH) using benzene: pyridine: formic acid (36:9:5). Toluene: ethyl formate: formic acid (5:4:1) and benzene: ethyl acetate (22.5:2.5) as the developing solvent systems.
**Reaction of 4'-hydroxy-5,7-dimethoxyflavone with Iron (III) chloride in presence of perchloric acid**

A mixture of 4'-hydroxy-5,7-dimethoxyflavone (2 gm 7 mmol) and ferric chloride (1.6 gm 10 mmol) in (200 ml) acetic acid containing (6 ml) of 70% perchloric acid was refluxed for 5 hrs. on an oil bath maintained at 135°C under nitrogen atmosphere. Reaction mixture was poured into water and the precipitate was filtered, washed and dried. (1.9 gm) of crude product was obtained which was adsorbed on silica gel and added to the column. The column was successively eluted with the mixture of petroleum ether: benzene and benzene:ethyl acetate of different ratios. After the recovery of starting material a chromatographically homogeneous product, WS(160 mg) was obtained.

WS(150 mg), anhydrous potassium carbonate (4 gm), dimethyl sulphate (0.5 ml) and dry acetone (400 ml) was refluxed on a water bath for 6 hrs. It was then filtered and the residue washed several times with hot acetone, the filtrate and washings were combined and evaporated to dryness. The yellow oily mass left behind was treated with petroleum ether and then dissolved in chloroform. The chloroform solution was washed with water dried over anhydrous sodium sulphate and concentrated to give a crude solid. Crude solid gives three products WS-1, WS-2 and WS-3 which was
separated by preparative TLC in benzene:pyridine:formic acid (36:9:5) WS-2 and WS-3 being minor, could not be identified.

WS-1, I-4',II-4',I-5,II-5,I-7,II-7-hexa-O-methyl[I-8-CH₂-II-8] biflavone(homocupressuflavone hexamethylether)(CXXXV)

It was crystallized from chloroform-methanol as yellow crystals m.p. 249°C, C₃₇H₃₂O₁₀ (M⁺ 636).

$^1$H-NMR (CDCl₃); value on δ scale

7.93(d, J=9Hz, 4H, H-2',6',2 ,6 ), 6.99(d, J=9Hz, 4H, H-3',5',3 ,5 ), 6.71(s, 2H, H-3,3 ), 6.33(s, 2H, H-6,6 ), 4.49 (s, 2H, -CH₂-), 3.96(s, 6H, 5,5′-OMe), 3.87(s, 6H, 4',4′-OMe), 3.61 (s, 6H, 7,7′-OMe).

$^1$H-NMR (CDCl₃); value on δ scale, after the addition of Eu(fod)₃

8.10(d, J=9Hz, 4H, H-2',6',2 ,6 ), 7.06(d, J=9Hz, 4H, H-3',5',3 ,5 ), 6.58(s, 2H, H-3,3 ), 10.55(s, 2H, H-6,6 ), 5.40 (s, 2H, -CH₂-), 12.38(s, 6H, 5,5′-OMe), 3.85(s, 6H, 4',4′-OMe), 4.51(s, 6H, 7,7′-OMe).

Mass Spectrum (75 eV), m/e (rel. int.): 636(66)M⁺, 622(56), C₃₆H₃₀O₁₀⁺; 621(91), C₃₆H₂₉O₁₀⁺; 608(20), C₃₆H₃₂O₉⁺; 606(8),
C_{35}H_{26}O_{10}^+; 591(9), C_{34}H_{23}O_{10}^+; 590(23), C_{35}H_{26}O_{9}^+; 561(5), C_{32}H_{17}O_{10}^+; 318(10), M^{2+}/2; 311(27), C_{18}H_{15}O_{5}^+; 297(10), C_{18}H_{17}O_{4}^+; 193(17), C_{10}H_{9}O_{4}^+; 150(60), C_{7}H_{4}O_{4}^+; 135(100), C_{8}H_{7}O_{2}^+; 132(26), C_{9}H_{8}O_{4}^+.

3,3'-methylene bis(phloroacetophenone di-O-methyl ether)
(CXXXVII)

To a solution of phloroacetophenone 4,6-di-O-methyl ether (5 gm) in alcohol, sulphuric acid (10 gm) and formalin (1.5 ml) was added slowly with gentle stirring and then reaction mixture was stirred vigorously for 15 mts. at 30°C. The reaction mixture was allowed to stand for two hrs. A white solid mass was obtained which was filtered, washed and dried.

The crude product was crystallized from petroleum ether-benzene, white needles were obtained, m.p. 198°C.

3,3'-methylene bis(chalcone tri-O-methylether) (CXXXVIII)

In a mixture of CXXXVII (5 gm) and aniseldehyde (1.5 ml), sodium hydroxide (10 gm) solution in oxygen free water (20 ml) was added dropwise. The reaction mixture was refluxed on a water bath for half an hour after complete addition of sodium hydroxide solution, 2/3 water was added and acidified with hydrochloric acid, yellow precipitate, filtered, washed and dried.
The crude yellow product was crystallized from alcohol as yellow needles, m.p. 219°C.

**Homocupressuflavone hexa-O-methylether (CXXXV)**

A mixture of bis chalcone CXXXVIII (2 gm) sublimed selenium dioxide (2 gm) in isoamyl alcohol (50 ml) was refluxed for 15 hrs. The selenium dioxide was removed, washed with ether and combined solution extracted with 10% sodium hydroxide solution. The precipitate solid was washed and dried.

It was crystallized from chloroform-methanol as yellow crystals, m.p. 253°C, C_{37}H_{32}O_{10} (M^+ 636).

^1H-NMR (CDCl₃); value on δ scale

7.88(d, J=9Hz, 4H, H-2',6',2,6 ), 6.92(d, J=9Hz, 4H, H-3',5',3,5 ), 6.66(s, 2H, H-3,3 ), 6.28(s, 2H, H-6,6 ), 4.43 (s, 2H, -CH₂-), 3.93(s, 6H, 5,5 -OMe), 3.82(s, 6H, 4',4 -OMe), 3.56(s, 6H, 7,7 -OMe).

Mass Spectrum (75 eV), m/e (rel. int.) 636(66)M^+; 622(56), C_{36}H_{30}O_{10}^+; 621(91), C_{36}H_{29}O_{10}^+; 608(20), C_{36}H_{32}O_{9}^+; 606(8), C_{35}H_{26}O_{10}^+; 591(9), C_{34}H_{23}O_{10}^+; 590(23), C_{35}H_{26}O_{9}^+; 561(5), C_{32}H_{17}O_{10}^+; 318(10), M^+2+/2; 311(27), C_{18}H_{15}O_{5}^+; 297(10), C_{18}H_{17}O_{4}^+; 193(17), C_{10}H_{9}O_{4}^+; 150(60), C_{7}H_{2}O_{4}^+; 135(100), C_{8}H_{7}O_{2}^+; 132(25), C_{9}H_{8}O^+. 
Reaction of 5,7,4'-trimethoxyflavone with Iron (III) chloride in presence of perchloric acid

A mixture of 5,7,4'-trimethoxyflavone (1 gm, 3.2 mmol) ferric chloride (0.5 gm, 3.2 mmol) in acetic acid glacial (200 ml) containing 3 ml of 70% perchloric acid was refluxed for 5 hrs. on an oil bath maintained at 135°C under nitrogen atmosphere. The reaction mixture was filtered, washed and dried. (900 mg) of crude product was obtained which was adsorbed on silica gel and added to the column. The column was successively eluted with the mixture of petroleum ether: benzene and benzene:ethyl acetate of different ratio's. After the recovery of small amount of starting material two products HN-5(400 mg) and HN-6(100 mg) was obtained.

HN-5(8-chloro-5,7,4'-trimethoxyflavone (CXLIII)

It was crystallized from chloroform methanol as white needles, m.p. 235°C, C_{18}H_{13}O_{5}Cl M^{+}, 346(Cl^{35}), 348 (Cl^{37}).

$^1$H-NMR (CDCl$_3$); value on $\delta$ scale

$8.00(d, 2H, J=9Hz, H-2', 6')$, $7.09(d, 2H, J=9Hz, H-3', 5')$, $6.49(s, 1H, H-6)$, $6.68(s, 1H, H-3)$, $4.05(s, 3H, 5'-Ome)$, $4.00(s, 3H, 4'-Ome)$, $3.96(s, 3H, 7'-Ome)$. 
Mass Spectrum (75 ev), m/e (rel. int.) 346(100)Cl\(^{35}\), M\(^+\); 348(54)Cl\(^{37}\), M\(^+\); 345(72), C\(_{18}\)H\(_{14}\)O\(_5\)Cl\(^+\); 317(64), C\(_{17}\)H\(_{14}\)O\(_4\)Cl\(^+\); 302(27), C\(_{16}\)H\(_{11}\)O\(_4\)Cl\(^+\); 272(10), C\(_{14}\)H\(_9\)O\(_4\)Cl\(^+\); 216(4), C\(_9\)H\(_7\)O\(_4\)Cl\(^+\); 215(2), C\(_9\)H\(_8\)O\(_4\)Cl\(^+\); 214(6), C\(_9\)H\(_7\)O\(_4\)Cl\(^+\); 186(8), C\(_8\)H\(_7\)O\(_3\)Cl\(^+\); 184(25), C\(_8\)H\(_7\)O\(_3\)Cl\(^+\); 151(54), C\(_8\)H\(_7\)O\(_3\); 132(60), C\(_9\)H\(_8\)O\(^+\).

HN-6 (homocupressuflavone hexamethylether) (CXXXV)

HN-6 was crystallized from chloroform-methanol as yellow crystals, m.p. 248-50°C, C\(_{37}\)H\(_{32}\)O\(_{10}\) (M\(^+\) 636).

\(^1\)H-NMR (CDCl\(_3\)); value on \(\delta\) scale

7.93(d, J=9Hz, 4H, H-2', 6', 2, 6 ), 6.99(d, 4H, J=9Hz, H-3', 5', 3, 5 ), 6.71(s, 2H, H-3, 3 ), 6.33(s, 2H, H-6, 6 ), 4.49 (s, 2H, -CH\(_2\)), 3.96(s, 6H, 5, 5 -OMe), 3.87(s, 6H, 4', 4 -OMe), 3.61(s, 6H, 7, 7 -OMe).

Mass Spectrum (75 ev), m/e (rel. int.) 636(66), M\(^+\); 622(56), C\(_{36}\)H\(_{30}\)O\(_{10}\); 621(92), C\(_{36}\)H\(_{29}\)O\(_{10}\); 608(20), C\(_{36}\)H\(_{30}\)O\(_9\); 606(8), C\(_{35}\)H\(_{26}\)O\(_{10}\); 591(9), C\(_{34}\)H\(_{23}\)O\(_{10}\); 590(23), C\(_{35}\)H\(_{26}\)O\(_9\); 561(5), C\(_{32}\)H\(_{17}\)O\(_{10}\); 318(10), M\(^{2+}\)/2; 311(27), C\(_{18}\)H\(_{15}\)O\(_{3}\); 297(10), C\(_{18}\)H\(_{17}\)O\(_4\); 193(17), C\(_{10}\)H\(_9\)O\(_4\); 150(60), C\(_7\)H\(_2\)O\(_4\); 135(100), C\(_8\)H\(_7\)O\(_2\); 132(25), C\(_9\)H\(_8\)O\(^+\).
Reaction of 1,3-dimethoxy benzene with Iron (III) chloride in presence of perchloric acid

A mixture of (10 gm, 9 ml) 1,3-dimethoxy benzene and ferric chloride (11.7 gm) in acetic acid (200 ml) containing perchloric acid (5 ml) were refluxed for 5 hrs. on oil bath maintained at 135°C. The reaction mixture was poured into water and extracted with ethyl acetate, washed with water and dried over anhydrous sodium sulphate, crude product liquid in nature was obtained which was applied to the column. The column was successively eluted with the mixture of petroleum ether and benzene. After the recovery of the major amount of starting material, five chromatographically homogeneous products WS-4, WS-5, WS-6, WS-7, WS-8 were separated by preparative thin layer chromatography in benzene as developing solvent.

WS-4, 2,2',4,4'-tetra-O-methyl diarylmethane (CXLVI)

It was liquid in nature, C_{17}H_{20}O_{4} (M^+ 288).

\(^1H\)-NMR (CDCl\textsubscript{3}); value in \delta scale

- 6.77(d, 2H, H-5,5'), 6.58(d, 2H, H-6,6'), 6.40(m, 2H, H-2,2'), 5.34(s, 2H, -CH\textsubscript{2}-), 3.92(s, 3H, 1'-OMe), 3.88(s, 3H, 2'-OMe), 3.83(s, 3H, 1'-OMe), 3.77(s, 3H, 2'-OMe).
WS-5(1,3-dimethoxy-4-chlorobenzene) (CXLVII)

It was crystallized from petroleum ether-benzene as white needles, m.p. 39-40°C, C₈H₇O₂Cl (M⁺ 172(Cl³⁵), 174(Cl³⁷)).

¹H-NMR (CDCl₃); value on δ scale

7.60(d, 1H, H-5), 6.96(d, 1H, H-6), 6.85(m, 1H, H-2), 3.83(s, 3H, l-OMe), 3.90(s, 3H, 3-OMe).

WS-6(1,3-dimethoxy-4-acetyl benzene) (CXLVIII)

WS-6 was liquid in nature C₁₀H₁₂O₃ (M⁺ 180).

¹H-NMR (CDCl₃); value on δ scale

7.10(d, 1H, H-5), 6.60(d, 1H, H-6), 6.30(m, 1H, H-2), 3.82(s, 3H, l-OMe), 3.73(s, 3H, 3-OMe), 2.57(s, 3H, 4-COCH₃).

WS-7(1-hydroxy-3-methoxybenzene) (CL)

Characterized by authentic sample of resorcinol monomethylether.

WS-8(1-hydroxy-3-methoxy-4-acetyl benzene) (XLIX)

Characterized by authentic sample of resacetophenone monomethy ether.
Reaction of 5-hydroxy-7,4′-dimethoxyflavone with Iron (III) chloride in presence of perchloric acid

A mixture of 5-hydroxy-7,4′-dimethoxyflavone (2 gm 6.71 mmol) and ferric chloride (1.08 gm 6.66 mmol) in 200 ml acetic acid containing (0.4 ml, 6.66 mmol) perchloric acid was refluxed for 5 hrs. on an oil bath maintained at 135°C under nitrogen atmosphere. The reaction mixture was then poured into 1 litre of water and the precipitate was filtered off and washed several times and dried. (1.8 gm) crude product was obtained which was adsorbed on silica gel and added to the column. The column was successively eluted with the mixture of petroleum ether:benzene and benzene:ethyl acetate of different ratio's, two products HN-1(700 mg) and HN-2 (600 mg) was obtained.

HN-1(600 mg), anhydrous potassium carbonate(5 gm), dimethyl sulphate (1.5 ml) and dry acetone 300 ml was refluxed on a water bath for 6 hrs. It was than filtered and the residue washed several times with hot acetone. The filtrate was evaporated to dryness, the yellow oily residue left behind was treated with petroleum ether and than dissolved in chloroform. The chloroform solution was washed with water and dried over anhydrous sodium sulphate and concentrated to give a crude solid. On TLC examination it gives two products HN-1M' and HN-1M which was separated by preparative thin layer chromatography in benzene:pyridine:formic acid (36:9:5). HN-1M'
was identified as 6,8-dichlo-5,7,4'-trimethoxyflavone and HN-1M was identified as 8-chloro-5,7,4'-trimethoxyflavone.

**HN-1M', 6,8-dichloro-5,7,4'-trimethoxyflavone (CLIII)**

It was crystallized from chloroform methanol as white needles, m.p. 260°C, \( C_{18}H_{14}O_5Cl_2 \) \( M^+ \) 381(Cl\(^{35}\)), 383 (Cl\(^{37}\)).

\(^{1}H\)-NMR (CDCl\(_3\)); value on ã scale

7.96(d, 2H, J=9Hz, H-2', 6'), 7.07(d, 2H, J=9Hz, H-3', 5'), 6.68(s, 1H, H-3), 4.03(s, 3H, 5'-OMe), 3.98(s, 3H, 4'-OMe), 3.90 (s, 3H, 7-OMe).

**Mass Spectrum (75 eV), m/e (rel. int.)** 383(5)Cl\(^{37}\), 382(52) Cl\(^{37}\), 381(20)Cl\(^{35}\), 380(100)Cl\(^{35}\) \( M^+ \); 352(19), \( C_{17}H_{13}O_4Cl_2^+ \); 337(10), \( C_{16}H_{10}O_4Cl_2^+ \); 317(5), \( C_{17}H_{14}O_4Cl_2^+ \); 302(5), \( C_{16}H_{11}O_4Cl_2^+ \); 249(9), \( C_9H_6O_4Cl_2^+ \); 250(3), \( C_9H_7O_4Cl_2^+ \); 221(3), \( C_9H_6O_3Cl_2^+ \); 207(9), \( C_7H_4O_3Cl_2^+ \); 132(80), \( C_9H_8O^+ \).

**HN-1M(8-chloro-5,7,4'-trimethoxyflavone)(CLIV)**

It was crystallized from chloroform methanol as white needles, m.p. 234°C, \( C_{18}H_{15}O_5Cl \) \( M^+ \) 346(Cl\(^{35}\)), 348(Cl\(^{37}\)).
$^1$H-NMR (CDCl$_3$); value on $\delta$ scale

7.96 (d, $2H, J=9$Hz, $H-2', 6'$), 7.02 (d, $2H, J=9$Hz, $H-3', 5'$), 6.43 (s, $1H, H-6$), 6.60 (s, $1H, H-3$), 4.00 (s, $3H, 5$-OMe), 3.97 (s, $3H, 4$',-OMe), 3.97 (s, $3H, 7$-OMe).

Mass Spectrum (75 eV), m/e (rel. int.), 346 (100) Cl$^{35}$; 348 (54) Cl$^{37}$ M$^+$; 345 (72), C$^{18}_{18}$$^{14}$O$^5$Cl$^+$; 317 (64), C$^{17}_{17}$$^{14}$O$^4$Cl$^+$; 302 (27), C$^{16}_{16}$$^{11}$O$^4$Cl$^+$; 273 (10), C$^{14}_{14}$H$^5$O$^4$Cl$^+$; 216 (4), C$^9_{9}$H$^7$O$^4$Cl$^+$; 215 (2), C$^9_{9}$H$^8$O$^4$Cl$^+$; 214 (6), C$^9_{9}$H$^7$O$^4$Cl$^+$; 186 (8), C$^8_{8}$H$^7$O$^3$Cl$^+$; 184 (25), C$^8_{8}$H$^7$O$^3$Cl$^+$; 151 (54), C$^8_{8}$H$^7$O$^3$; 132 (60), C$^8_{8}$H$^8$O$^+$.

HN-2 (100 mg), anhydrous potassium carbonate (1 gm), dimethyl sulphate (0.5 ml) and dry acetone 200 ml was refluxed on a water bath for 6 hrs. It was then filtered, washed and evaporated to dryness. Yellow oily mass after treatment with petroleum ether dissolved in chloroform and washed with water and dried over anhydrous sodium sulphate. Crude solid on TLC examination yielded a homogenous product HN-2M which was identified as 6-chloro-5,7,4'-trimethoxyflavone (CLV).

HN-2, 6-chloro-5,7,4'-trimethoxyflavone (CLV)

It was crystallized from chloroform methanol as white needles, m.p. 242°C, C$^{18}_{18}$H$^{14}$O$^5$Cl M$^+$ 346 (Cl$^{35}$), 348 (Cl$^{37}$).
$^1$H-NMR (CDCl$_3$); value on $\delta$ scale

7.86(d, 2H, J=9Hz, 2', 6'), 7.02(d, 2H, J=9Hz, H-3', 5'),
6.84(s, 1H, H-8), 6.60(s, 1H, H-3), 4.00(s, 3H, 5-OMe), 3.97(s, 3H,
4'-OMe), 3.87(s, 3H, 7-OMe).

Mass Spectrum (75 eV), m/e (rel. int.), 346(100)C$_{15}^5$; 348(32)
C$_{17}^{37}$ M$^+$; 317(25), C$_{17}^{14}H_4^0Cl^+$; 311(50), C$_{18}^{15}H_5^0^5^+$; 302(14),
C$_{16}^{16}H_4^0^1^4Cl^+$; 272(7), C$_{14}^{14}H_4O^4Cl^+$; 216(4), C$_{9}^{9}H_7^0^4Cl^+$; 214(4),
C$_{9}^{9}H_7^0^4Cl^+$; 186(7), C$_{8}^{8}H_7O^3Cl^+$; 151(46), C$_{8}^{8}H_7^0^3^+$; 132(17),
C$_{9}^{9}H_8^0^+$.

Dried HN-2(100 mg) was heated with pyridine (1.5 ml)
and acetic anhydride (3 ml) on a water bath for 2 hrs. It was
then cooled to room temperature and poured on to crushed ice.
The separated solid was filtered washed with water and dried.
White crystals HN-2A was identified as 5-acetoxy-6-chloro-
7,4'-dimethoxyflavone.

HN-2A, 5-acetoxy-6-chloro-7,4'-dimethoxyflavone (CLVI)

HN-2A was crystallized from chloroform methanol as
white needles, m.p. 250-52°C, C$_{19}^{19}H_{15}^0O_6Cl$ M$^+$ 374(C$_{15}^{37}$), 376
(C$_{17}^{37}$).
$^1$H-NMR (CDCl$_3$); value on δ scale

7.81 (d, 2H, J=9Hz, H-2', 6'), 6.98 (d, 2H, J=9Hz, H-3', 5'),
6.84 (s, 1H, H-8), 6.49 (s, 1H, H-3), 4.00 (s, 3H, 4'-Ome), 3.86 (s, 3H, 7- OMe), 2.47 (s, 3H, 5-OAc).

Mass Spectrum (75 eV), m/e (rel. int.), 374(21)Cl$^{35}$; 376(6)
Cl$^{37}$ M$^+$; 334(98), C$_{17}$H$_{13}$O$_5$Cl$^+$; 332(100), C$_{17}$H$_{13}$O$_5$Cl$^+$; 317(4),
C$_{16}$H$_{10}$O$_5$Cl$^+$; 303(29), C$_{16}$H$_{12}$O$_4$Cl$^+$; 289(27), C$_{15}$H$_{10}$O$_4$Cl$^+$;
243(2), C$_{10}$H$_8$O$_5$Cl$^+$; 200(62.5), C$_8$H$_5$O$_4$Cl$^+$; 172(21), C$_7$H$_5$O$_3$Cl$^+$;
132(83), C$_9$H$_8$O$^+$. 

Reaction of 2'-hydroxy-4',6',4'-trimethoxychalcone with
iron (III) chloride in presence of perchloric acid

A mixture of 2'-hydroxy-4',6',4'-trimethoxychalcone
(5 gm, 16 mmol) and ferric chloride (2.70 gm, 16 mmol) in
acetic acid glacial (200 ml) containing 1.0 ml of 70% perchlo-
ric acid was refluxed for 5 hrs. on an oil bath maintained at
135°C under nitrogen atmosphere. Reaction mixture was poured
into one litre of water and the precipitate was filtered,
washed and dried. (4.5 gm) crude product was obtained which
was refluxed in alcohol on water bath for one hr. Alcoholic
fraction was filtered off. A gummy mass left behind may be
polymerized product.
Alcoholic fraction was concentrated and left for crystallization. 1.5 gm white needle shaped crystals were obtained, which was indentified as 5,7,4'-trimethoxy-naringenin (CLVIII).

5,7,4'-tri-O-methylnaringenin (CLVIII)

It was crystallized from alcohol m.p. 132-35°C, C_{18}H_{18}O_{5} (M^+ 314).

^1H-NMR (CDCl$_3$); value on δ scale

7.00(d, 2H, J=9Hz, H-2',6'), 6.78(d, 2H, J=9Hz, H-3',5'), 6.45(d, 1H, J=2.5Hz, H-8), 6.15(d, 1H, J=2.5Hz, H-6), 4.50(m, 1H, H-2), 3.92(s, 3H, 5-OMe), 3.80(s, 3H, 4'-OMe), 3.71(s, 3H, 7-OMe), 2.98(d, 2H, H-3).

Mass Spectrum (75 eV), m/e (rel. int.), 314(43) M^+; 313(30), C_{18}H_{17}O_{5}^+; 207(7), C_{11}H_{11}O_{4}^+; 181(30), C_{9}H_{9}O_{4}^+; 134(100), C_{9}H_{10}O_{4}^+; 119(13), C_{8}H_{7}O_{4}^+; 107(10), C_{7}H_{7}O_{4}^+; 91(13), C_{7}H_{7}^+.

Reaction of 2'-hydroxy-4',6',4-trimethoxychalcone with vanadium oxytrichloride (VOCl$_3$)

(1.5 gm) 2'-hydroxy-4',6',4-trimethoxychalcone was dissolved in (100 ml) sodium dried benzene. System was purged with nitrogen and (1 ml) VOCl$_3$ was added. The reaction mixture
was left for over night after stirring for 15 mts. (50 ml) sulphuric acid was added, the acidic layer turned blue. Benzene layer was separated and washed with water and sodium bicarbonate solution and dried over anhydrous sodium sulphate. (1.2 gm) of crude product was obtained which was adsorbed on silica gel and added to the column. The column was successively eluted with the mixture of petroleum ether and benzene of different ratio's. A products WS-19 (800 mg) was obtained.

WS-19, 2'-hydroxy-3'-chloro-4',6',4'-trimethoxychalcone (CLIX)

WS-19 was crystallized from ethyl acetate benzene as yellow needles m.p. 154-56°C, C_{18}H_{17}O_{5}Cl M^+ 348(Cl^{35}), 350(Cl^{37}).

IR(KBr) cm^{-1}

3460(OH broad), 1610(C=O), 1540, 1510, 1440, 1395 (C=C), 1350, 1300, 1270, 1230, 1180, 1130(=C-O-C), 1040 (Ar-Cl), 1070, 990, 830, 740, 690 cm^{-1}.

$^1$H-NMR (CDCl$_3$); value on δ scale

7.75(s,2H,H-α,β), 7.53(d,2H,J=9Hz,H-2,6), 6.93(d, 2H,J=9Hz,H-3,5), 6.00(s,1H,H-5'), 3.99(s,6H,OMe-4',6'), 3.88 (s,3H,OMe-4).
Mass Spectrum (75 eV), m/e (rel. int.), 349(22)C\textsuperscript{35}Cl; 351(6), C\textsubscript{18}H\textsubscript{16}O\textsubscript{5}C\textsuperscript{37}Cl\textsuperscript{+}; 347(60), C\textsubscript{17}H\textsubscript{17}O\textsubscript{4}Cl\textsuperscript{+}; 241(12.5), C\textsubscript{11}H\textsubscript{10}O\textsubscript{4}Cl\textsuperscript{+}; 214(100), C\textsubscript{9}H\textsubscript{7}O\textsubscript{4}Cl\textsuperscript{+}; 186(6), C\textsubscript{8}H\textsubscript{7}O\textsubscript{3}Cl\textsuperscript{+}; 171(17.5), C\textsubscript{7}H\textsubscript{4}O\textsubscript{3}Cl\textsuperscript{+}; 161(25), C\textsubscript{10}H\textsubscript{9}O\textsuperscript{2+}; 134(60), C\textsubscript{9}H\textsubscript{10}O\textsuperscript{+}; 133(15), C\textsubscript{9}H\textsubscript{9}O\textsuperscript{+}; 121(67.5), C\textsubscript{6}H\textsubscript{17}O\textsuperscript{3+}; 107(2.5), C\textsubscript{7}H\textsubscript{7}O\textsuperscript{+}; 91(8), C\textsubscript{7}H\textsubscript{7}.

Reaction of 5,7,4'-trimethoxyflavanone with vanadium oxytrichloride (VOC\textsubscript{3})

(250 mg) 5,7,4'-trimethoxyflavanone was dissolved in (50 ml) sodium dried benzene. System was purged with nitrogen and (0.3 ml) VOC\textsubscript{3} was added. The reaction mixture was left for overnight after stirring for 15 mts. (5.0 ml) sulphuric acid was added, the acidic layer turned blue. Benzene layer was separated and washed with water and sodium bicarbonate solution and dried over anhydrous sodium sulphate. (200 mg) of crude single product was obtained.

WS-20, 8-chloro-5,7,4'-trimethoxyflavanone (CLIX)

It was crystallized from chloroform-methanol as white crystals m.p. 158-60\textdegree C, C\textsubscript{18}H\textsubscript{17}O\textsubscript{5}Cl, 348(C\textsuperscript{35})\textsuperscript{+}, 350 (C\textsuperscript{37})\textsuperscript{+}. 
IR (KBr) cm\(^{-1}\)

1780(C=O), 1600, 1510, 1470, 1430(C=C), 1270, 1220, 1190, 1120(=C-O-C-), 1040(Ar-Cl), 990, 925, 845, 830, 810, 770, 710, 690 cm\(^{-1}\).

\(^1\)H-NMR (CDCl\(_3\)); value on \(\delta\) scale

7.05(d,2H,J=9Hz,H-2',6'), 6.77(d,2H,J=9Hz,H-3',5'), 6.38(s,1H,H-6), 4.55(m,1H,H-2), 3.95(s,3H,5-OMe), 3.85(s,3H,4'-OMe), 3.80(s,3H,7-OMe), 2.99(d,2H,H-3).

Mass Spectrum (75 eV), m/e (rel. int.), 348(100)Cl\(^{35}\); 350(33)Cl\(^{37}\) M\(^+\); 320(47), C\(_{17}\)H\(_{17}\)O\(_4\)Cl\(^+\); 305(61), C\(_{16}\)H\(_{14}\)O\(_4\)Cl\(^+\); 290(8), C\(_{15}\)H\(_{11}\)O\(_4\)Cl\(^+\); 275(55), C\(_{14}\)H\(_{8}\)O\(_4\)Cl\(^+\); 241(11), C\(_{11}\)H\(_{10}\)O\(_4\)Cl\(^+\); 214(11), C\(_9\)H\(_7\)O\(_4\)Cl\(^+\); 135(22), C\(_9\)H\(_11\)O\(^+\); 134(19), C\(_9\)H\(_10\)O\(^+\); 121(44), C\(_6\)H\(_1\)O\(^+\); 119(5), C\(_8\)H\(_7\)O\(^+\); 91(11), C\(_7\)H\(^+\).

Reaction of 5,7,4'-trimethoxyflavone with vanadium oxytrichloride (VOCl\(_3\))

(500 mg) 5,7,4'-trimethoxyflavone was dissolved in 250 ml dry benzene. System was purged with nitrogen and (0.5 ml) vanadium oxytrichloride was added. The reaction mixture was stirred for 15 mts. and then left it overnight. (10 ml) sulphuric acid was added, the acidic layer turned blue. Benzene layer was separated, washed with water.
and sodium bicarbonate solution and dried over anhydrous sodium sulphate. (400 mg) of crude product was obtained which was adsorbed on silica gel and added to the column. The column was successively eluted with the mixture of benzene:ethyl acetate of different ratio's. After the recovery of small amount of starting material a single product WS-21 was obtained.

**WS-21, 8-chloro-5,7,4'-trimethoxyflavone (CXLIII)**

It was crystallized from chloroform methanol as white crystals, m.p. 240°C, \(C_{18}H_{16}O_5Cl\), \(M^+ 346(Cl^{35})\), 348 (\(Cl^{37}\)).

**\(\text{IR (KBr)} \text{ cm}^{-1}\)**

1620 (C=O), 1600, 1510, 1470 (C=C), 1350, 1250, 1220, 1180, 1120 (=C-O-C), 1040 (Ar-Cl), 990, 925, 845, 830, 810, 710 cm\(^{-1}\).

**\(\text{\(^1\text{H-NMR}} \text{ (CDCl}_3\))\); value on \(\delta\) scale**

8.00 (d, 2H, J=9Hz, H-2', 6'), 7.10 (d, 2H, J=9Hz, H-3', 5'), 6.65 (s, 1H, H-3), 6.49 (s, 1H, H-6), 4.05 (s, 6H, 5, 4'-OME), 3.96 (s, 3H, 7-OME).

**Mass Spectrum (75 eV), m/e (rel. int.), 346(100)Cl^{35}; 348(54) Cl^{37} M^+; 345(72), C_{18}H_{14}O_5Cl^+; 317(64), C_{17}H_{14}O_4Cl^+; 302(27),**
\[ C_{16}H_{11}O_4Cl^+; \ 273(10), \ C_{14}H_{5}O_4Cl^+; \ 216(4), \ C_{9}H_7O_4Cl^+; \ 215(2), \ C_{9}H_8O_4Cl^+; \ 214(6), \ C_{9}H_7O_4Cl^+; \ 186(8), \ C_{8}H_9O_3Cl^+; \ 184(25), \ C_{8}H_7O_3Cl^+; \ 151(54), \ C_{8}H_7O_3^+; \ 132(60), \ C_{9}H_8O^+. \]