CHAPTER II

Isolation and Characterization of Coumarins from *Feroniella lucida*

2.1 Extraction and Isolation

Air-dried roots of *Feroniella lucida* were extracted with methanol using Soxhlet apparatus to yield methanolic extract. The extract was subjected to suspend in water and excessively partitioned with dichloromethane and *n*-butanol to yield dichloromethane and butanolic extract, respectively. Dichloromethane extract was suspended in methanol and filtered to remove methanol-insoluble residue. The residue was further purified with column chromatography to yield anisolactone (**1**) and 2″, 3″-epoxyanisolactone (**2**). The methanol layer was concentrated and fractionated with quick column chromatography to yield 2 subfraction, D1 and D2. The subfraction D1 was purified by silica gel column chromatography to yield psoralen (**3**), bergapten (**4**) and isopimpinellin (**5**). The subfraction D2 was further purified by combination of silica gel, flash, size-exclusion and high performance liquid chromatography to yield marmesin (**6**), feroniellin A (**7**), oxypeucedanin hydrate (**8**), feroniellin C (**9**), 2″, 3″-dihydroxyanisolactone (**10**) and feroniellin B (**11**), respectively.

2.2 Structure Elucidation

2.2.1 Feroniellin A

Feroniellin A (**7**) was obtained as pale yellow powder and had molecular formula $C_{21}H_{24}O_7$ as established by HRESIMS. The UV absorbance at 252 and 309 nm suggested the presence of coumarin moiety (Kang *et al*, 2001; Stevenson *et al*, 2003).

The ^{1}H-NMR spectrum showed a pair of doublet protons at δ_H 6.29 (d, J = 9.6 Hz, 1H) and δ_H 8.22 (d, J = 10.0 Hz, 1H) which could be assigned to H-3 and H-4 of an α-pyrone ring system. Moreover, the presence of a pair of furan protons at δ_H 7.60 (d, J = 2.0 Hz, 1H) and δ_H 7.01 (d, J = 1.2 Hz, 1H) and a singlet proton at δ_H 7.17 (s, 1H) indicated that coumarin ring was fused with furan ring at C-6 and C-7, while C-8 was unoccupied by substituted group.

In HMBC spectrum, the correlations of δ_H 4.37 (H-1″a) and δ_H 4.58 (H-1″b) with δ_C 148.6 (C-5) confirmed that the substituted group was connected to C-5 of furanocoumarin unit. The cross peaks of CH_3-9″/C-7″, C-8″; CH_3-10″/C-7″, C-8″ and H-7″/C-9″, C-10″ indicated that the substituted group was contained -$CHC(CH_3)_2OH$ system. In addition, the cross peaks of H-1″a,b/C-2″ ; H-2″/C-1″, C-3″ and CH_3-4″/C-2″, C-3″ and H-7″ / C-3″ suggested that the C-3″ was linked to C-7″ by ether linkage to form tetrahydrofuran ring. According to NMR data, this portion of feroniellin A was similar to those of red seaweeds *Laurencia viridis*, dehydrothyrsiferol (Norte *et al*, 1996). The relative configuration of feroniellin A was accomplished by NOESY data analysis, the cross peaks between H-7″ and CH_3-4″ and CH_3-4″ and H-2″ indicated that they were on the same face of five-membered ring.

Table 2.1 ^{1}H and ^{13}C NMR data for feroniellin A (**7**) in $CDCl_3$

Position	δ_C	δ_H mult (*J* in Hz)	HMBC
2	161.2		
3	112.9	6.29 d (9.6)	C-2, 4a
4	139.3	8.22 d (10.0)	C-2, 5, 8a
4a	107.3		
5	148.6		
6	114.1		
7	158.1		
8	94.7	7.17 s	C-4a, 6, 7, 8a
8a	152.5		
2′	145.1	7.60 d (2.0)	C-6, 7, 3′
3′	104.8	7.01 d (1.6)	C-6, 7, 2′
1″	74.3	a 4.37 dd (8.0, 10.0)	C-5, 2″
		b 4.58 dd (2.8, 10.0)	C-5, 2″
2″	75.9	4.04 dd (2.4, 8.0)	C-1″, 3″
3″	83.9		
4″	27.6	1.24 s	C-2″, 3″, 5″
5″	33.7	α 1.75 m	
		β 2.16 m	C-3″, 6″
6″	26.4	1.91 m	C-5″, 7″
7″	87.6	3.84 m	C-3″, 9″, 10″
8″	70.5		
9″	23.3	1.26 s	C-7″, 8″, 10″
10″	24.0	1.16 s	C-7″, 8″, 9″

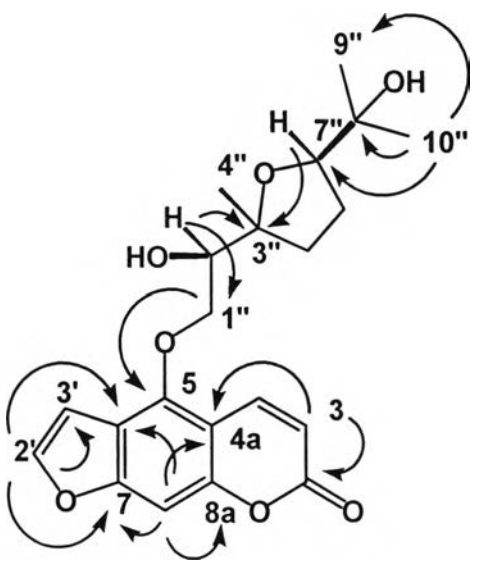


Figure 2.1 Selected HMBC correlations of feroniellin A.

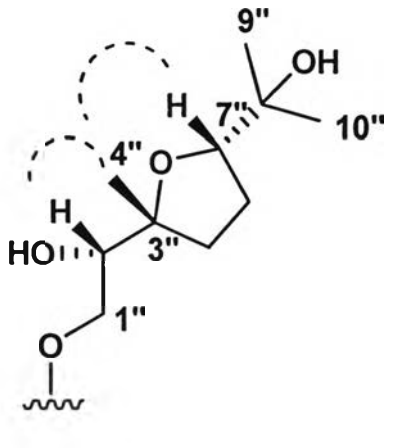
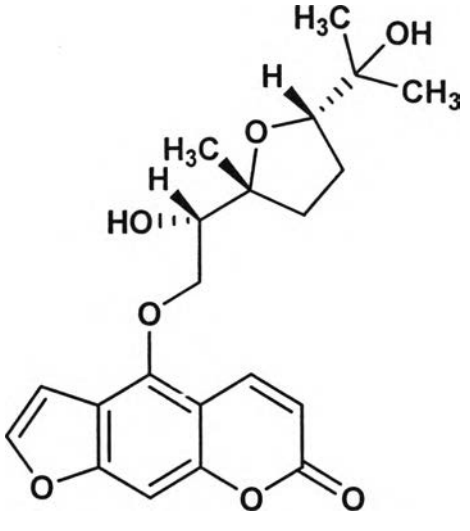


Figure 2.2 Key NOESY correlations of feroniellin A.

From all above data, the complete structure of feroniellin A was depicted as followed.



2.2.2 Feroniellin B

Feroniellin B (**11**) was obtained as dark yellow liquid and had molecular formula $C_{21}H_{24}O_7$ as established by HRESIMS. The UV absorbance at 228, 250 and 309 nm suggested the presence of coumarin moiety (Kang *et al*, 2001; Stevenson *et al*, 2003). The above data indicated that feroniellin B was isomeric of 7. The ^{1}H-NMR spectrum of **11** closely resembled to that of feroniellin A, except for slight difference in signals of oxygenated methines and methylenes, including upfield resonance (**Table 2.2**).

In HMBC spectrum, the correlations of δ_H 4.49 (H-1″a) and δ_H 4.72 (H-1″b) with δ_C 148.4 (C-5) confirmed that the substituted group was connected to C-5 of furanocoumarin unit. The cross peaks of H-1″a/ C-2″, H-2″/ C-7″ and H-7″/C-2″ indicated that C-2″ was linked to C-7″ by ether linkage to form tetrahydropyran ring. Interestingly,

this portion of feroniellin B was similar to that of isodehydrothyrsiferol from red seaweeds *Larencia viridis* (Norte *et al*, 1996). The relative configuration of feroniellin B was accomplished by NOESY data analysis. The cross peaks between H-1″b and H-2″, H-2″ and CH_3-4″, and H-6″β indicated that they were in the β-orientation, while H-1″a, H-6″α and H-7″ were in α-orientation.

Table 2.2 ^{1}H and ^{13}C NMR data for feroniellin B (**11**) in $CDCl_3$.

Position	δ_C	δ_H mult (*J* in Hz)	HMBC
2	161.0		
3	113.1	6.29 d (10.0)	C-2, 4a
4	139.0	8.18 d (9.6)	C-2, 5, 8a
4a	107.1		
5	148.4		
6	113.5		
7	158.1		
8	94.7	7.17 s	C-4a, 6, 7, 8a
8a	152.6		
2′	145.3	7.62 d (2.0)	C-6, 7, 3′
3′	104.6	6.95 d (2.0)	C-6, 7, 2′
1″	69.7	a 4.49 dd (4.4, 10.4)	C-5, 2″
		b 4.72 dd (8.4, 10.4)	C-5, 2″
2″	80.9	4.12 dd (4.4, 8.0)	C-1″, 3″, 5″, 7″
3″	68.0		
4″	25.0	1.24 s	C-2″, 3″, 5″
5″	32.8	α 1.79 m	C-2″, C-3″, C-7″
		β 1.68 m	C-2″, C-3″, C-7″
6″	20.8	α 1.83 m	C-2″, C-3″, C-5″, C-7″, C-8″
		β 1.61 m	C-2″, C-3″, C-7″
7″	76.1	3.44 dd (2.4, 11.2)	C-2″, 5″, C-6″, 8″
8″	71.9		
9″	24.2	1.19 s	C-7″, 8″, 10″
10″	26.3	1.21 s	C-7″, 8″, 9″

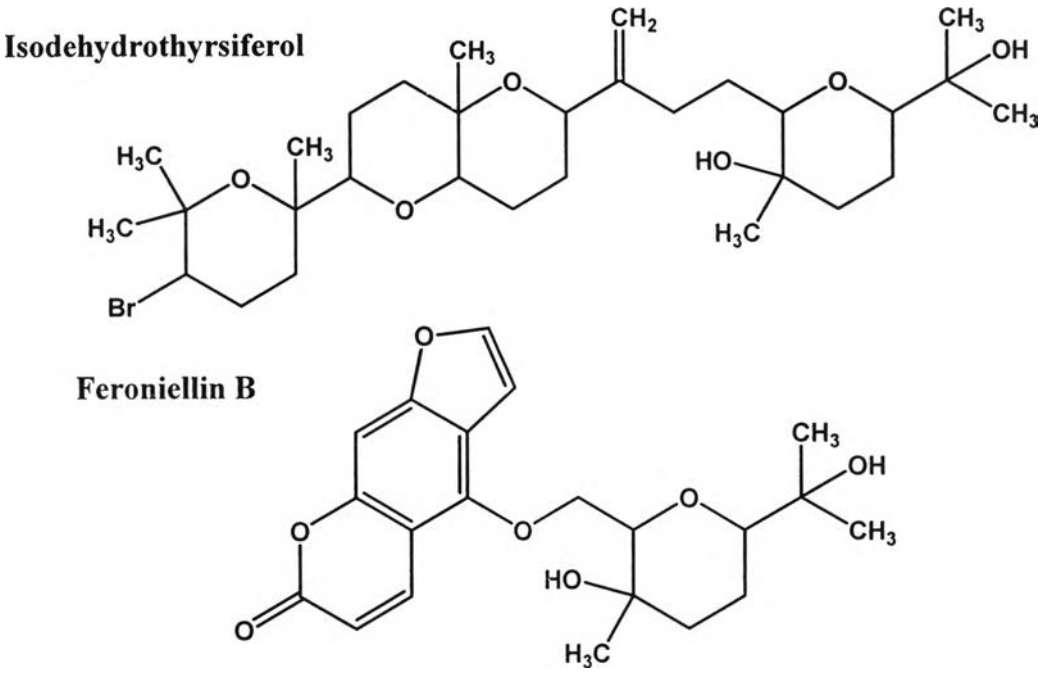


Figure 2.3 Comparison pyran moiety of isodehydrothyrsiferol and feroniellin B.

Table 2.3 Comparison of ^{1}H and ^{13}C NMR data of pyran moiety between isodehydrothyrsiferol and feroniellin B.

Position	Isodehydrothyrsiferol		Feroniellin B	
	δ_C	δ_H	δ_C	δ_H
2"	84.1	3.13 (dd, J = 1.7, 10.7 Hz)	80.9	4.12 (dd, J = 4.4, 8.0 Hz)
3"	70.15	-	68.0	-
4"	20.64	1.17 (s, 3H)	25.0	1.24 (s, 3H)
5"	40.22	1.88 (m)	32.8	1.79 (m)
		1.55 (m)		1.68 (m)
6"	24.99	2.09 (m, 2H)	20.8	1.86 (m)
				1.61 (m)
7"	84.42	3.15 (dd, J = 1.7, 10.7 Hz)	76.1	3.44 (dd, J = 2.4, 11.2 Hz)
8"	72.13	-	71.9	-
9"	24.41	1.15 (s, 3H)	24.2	1.19 (s, 3H)
10"	26.77	1.20 (s, 3H)	26.3	1.21 (s, 3H)

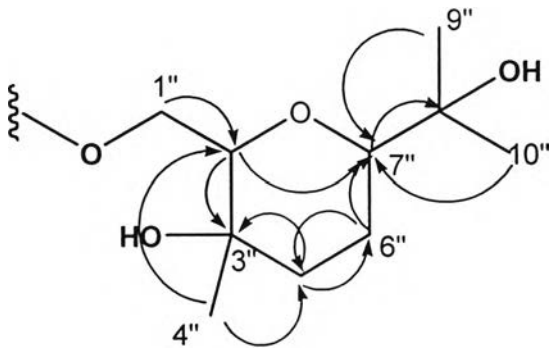


Figure 2.4 Key HMBC correlations of feroniellin B.

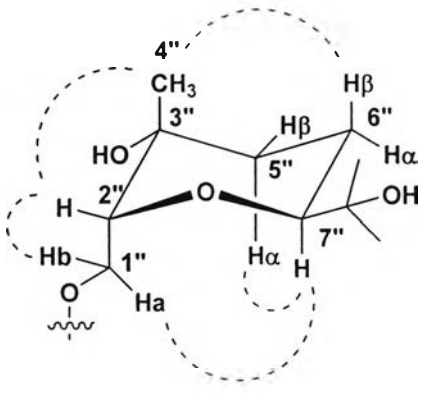
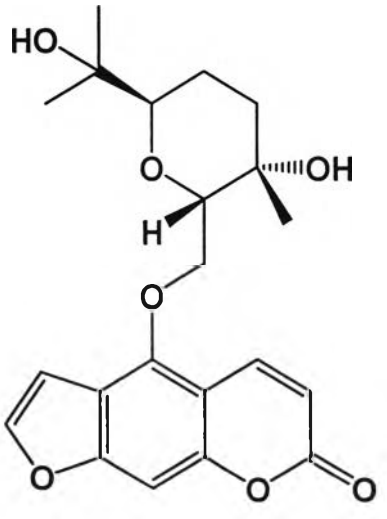


Figure 2.5 Key NOESY correlations observed for pyran moiety of feroniellin B.

From all above data, the complete structure of feroniellin B was described as shown.



2.2.3 Feroniellin C

Feroniellin C (**9**) was obtained as pale yellow powder and had molecular formula $C_{21}H_{24}O_7$ as established by HRESIMS. The UV absorbance at 224, 249 and 309 nm suggested the presence of coumarin moiety (Kang *et al*, 2001; Stevenson *et al*, 2003). The above data indicated that feroniellin C was isomeric of **7** and **11**.

The ^{1}H-NMR spectrum analysis showed that feroniellin C had the same core structure to feroniellin A and feroniellin B but differed in substituted group.

In HMBC spectrum, the correlations of δ_H 4.68 (H-1″a) with δ_C 149.3 (C-5) confirmed that substituted group was connected to C-5 of furanocoumarin unit. The cross peaks of CH_3-9″/C-8″, CH_3-10″/C-8″, H-2″/C-8″ and H-1″/C-2″ indicated that C-2″ was linked to C-8″ to form seven-membered cyclic ether. The relative configuration was accomplished by NOESY data analysis. The cross peaks between CH_3-4″ and H-5″β, H-5″β and H-7″β, and H-7″β and CH_3-9″ indicated that they were on the same face of seven-membered ring.

Table 2.4 ^{1}H and ^{13}C NMR data of feroniellin C (**9**) in $CDCl_3$

Position	δ_C	δ_H mult (*J* in Hz)	HMBC
2	161.5		
3	112.2	6.18 d (9.6)	C-2, 4a
4	139.7	8.16 d (9.6)	C-2, 5, 8a
4a	106.7		
5	149.3		
6	113.4		
7	158.3		
8	93.8	7.02 brs[a]	C-4a, 6, 7, 8a
8a	152.6		
2′	144.6	7.48 d (2.0)	C-6, 7, 3′
3′	105.5	7.02 brs[a]	C-6, 7, 2′
1″	73.7	a 4.21 m	C-5, 2″
		b 4.68 brd (8.4)	C-5
2″	76.1	4.16 m	C-1″, 3″, 8″
3″	74.1		
4″	22.1	1.22 s	C-2″, 3″, 5″
5″	37.9	α 2.04 brt (12.4)	C-3″, C-4″, C-6″, C-7″
		β 1.59 brdd (5.6, 14.0)	C-3″, C-4″, C-6″, C-7″
6″	25.9	α 1.86 m	C-3″, C-5″, C-7″, C-8″
		β 1.76 m	C-3″, C-5″, C-7″, C-8″
7″	76.8	3.72 brd (7.6)	C-5″, 6″, 8″
8″	78.4		
9″	21.5	1.28 s	C-7″, 8″, 10″
10″	28.9	1.14 s	C-7″, 8″, 9″

[a]Signals overlapped

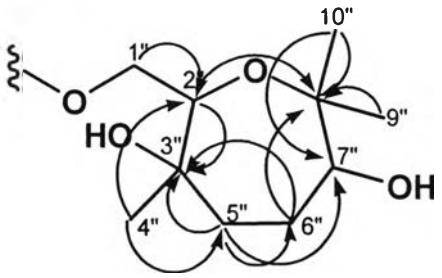


Figure 2.6 Key HMBC correlations of feroniellin C.

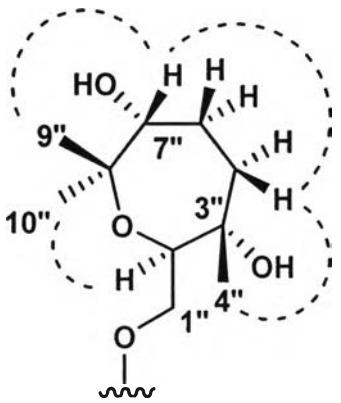
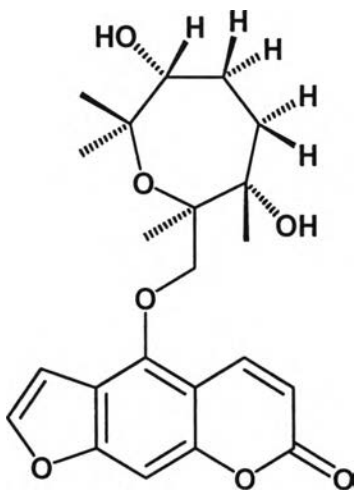


Figure 2.7 Key NOESY correlations observed for feroniellin C.

From all above data, the complete structure of feroniellin C was described as shown.



2.3 Experimental Section

2.3.1 General Experimental Procedure

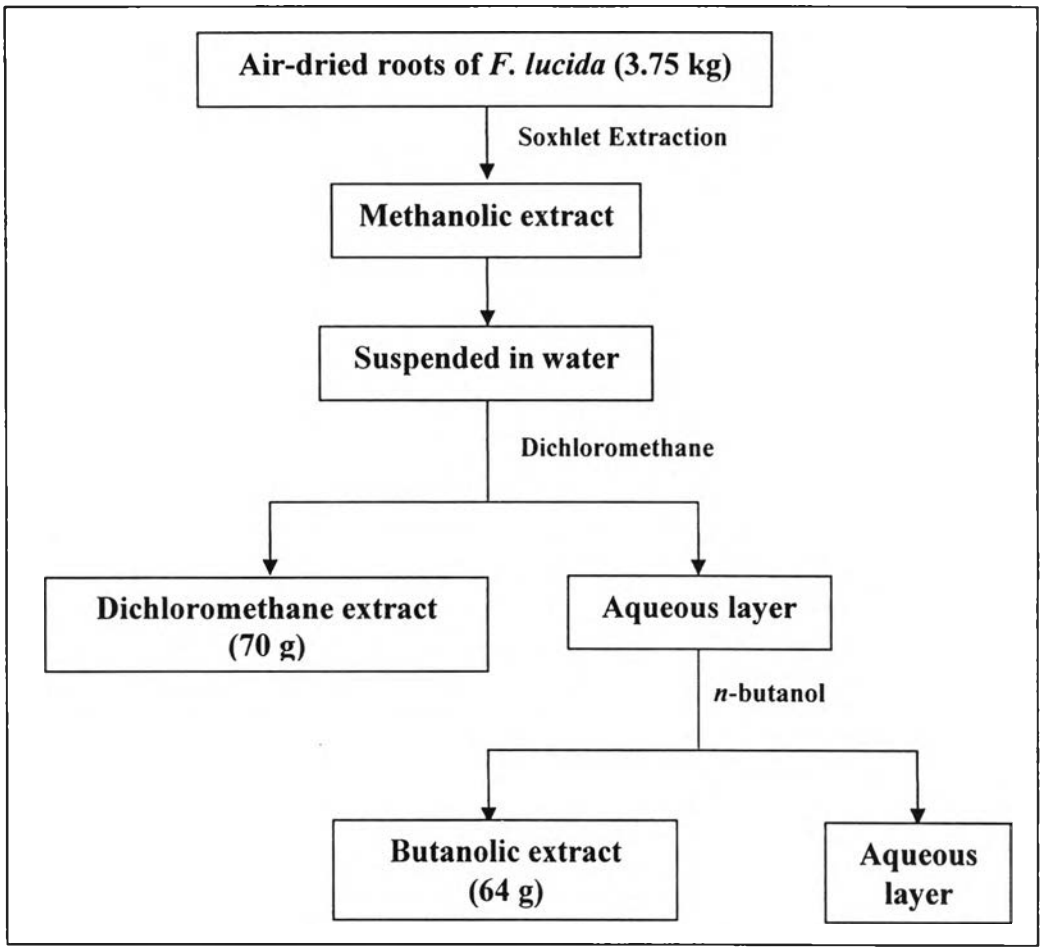
NMR spectra were recorded on a Varian Mercury^{+} 400 NMR Spectrometer operating 400 MHz for ^{1}H and 100 MHz for ^{13}C. The chemical shifts in δ (ppm) were assigned with reference to the signals from residual proton ($\delta_{7.26}$) and carbon ($\delta_{77.0}$) in deuterated chloroform. The UV spectra were recorded on Shimadsu UV-2250 photodiode array spectrophotometer. HRESIMS data were obtained from a Micromass LCt mass spectrometer. Optical rotations were measured on a Jasco P-100 polarimeter. High performance liquid chromatography was accomplished with a Water600 equipped with Waters 2996 photodiode array detector or Water PrepLC equipped with Water 2487 dual wavelength detector. Centrifugal chromatography was carried out on Harrison Research Model 7924T Chromatotron.

2.3.2 Plant Material

The roots of *F. lucida* were collected in April 2005 from Roi-Et province, Thailand. The specimens (voucher number BCU.O.T. 968) were identified by Associate Professor Dr. Thaweesakdi Boonkerd, Plant of Thailand Research Unit, Department of Botany, Faculty of Science, Chulalongkorn University.

2.3.2 Extraction and Purification

The air-dried roots (3.75 kg) were extracted with methanol using Soxhlet apparatus to yield methanolic extract. The extract was suspended in water and excessively partitioned with dichloromethane and *n*-butanol to yield dichloromethane (70 g) and butanolic (64 g) extracts, respectively. The procedure of extraction is summarized in **Scheme 2.1**.



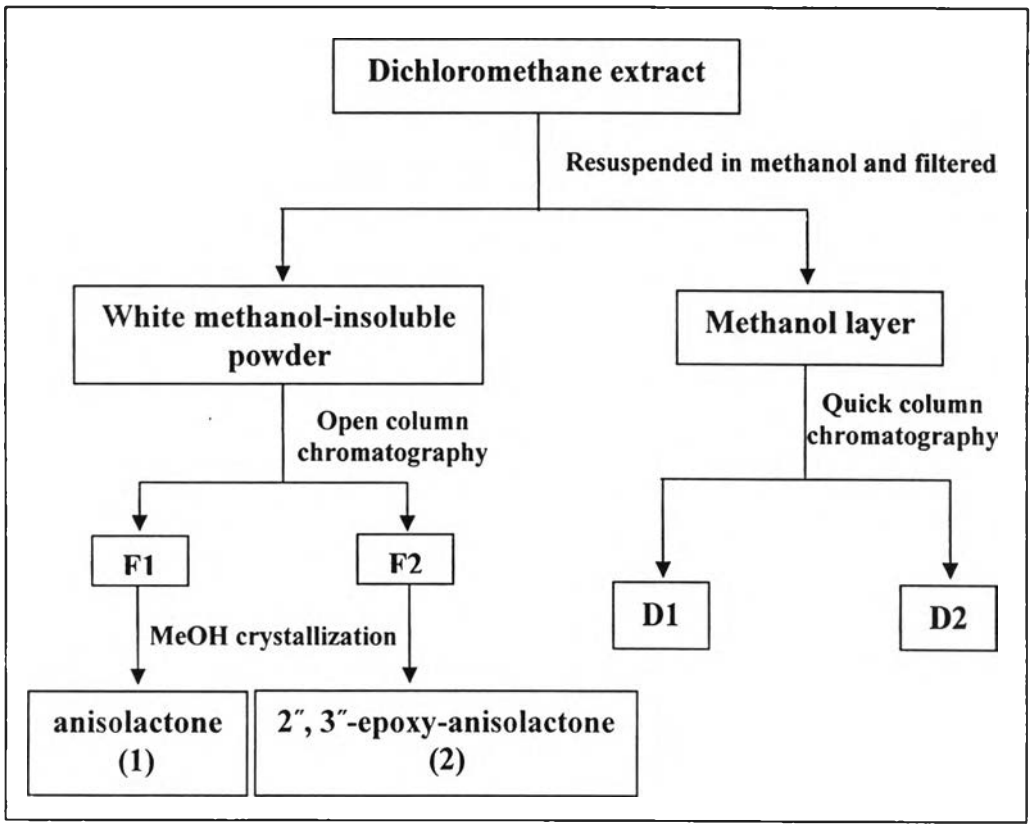
Scheme 2.1 Extraction procedure of *F.lucida* roots.

The dichloromethane extract was resuspended in methanol and filtered to separate white methanol-insoluble powder from methanol portion (**Scheme 2.2**). The white methanol-insoluble powder was further purified with silica gel column chromatography using mixed solvents systems of hexane/dichloromethane and dichloromethane/methanol (50% CH_2Cl_2/Hex to 100% CH_2Cl_2 and 1% to 5% MeOH/CH_2Cl_2) to give 2 fractions, F1 and F2. The fraction F1 and F2 were subjected to crystallize in methanol to yield anisolactone (**1,** 0.25 g, 0.36 % w/w; Lakshmi *et al*, 1984) and 2″, 3″-epoxyanisolactone (**2,** 0.06 g, 0.09% w/w; Lakshmi *et al*, 1984).

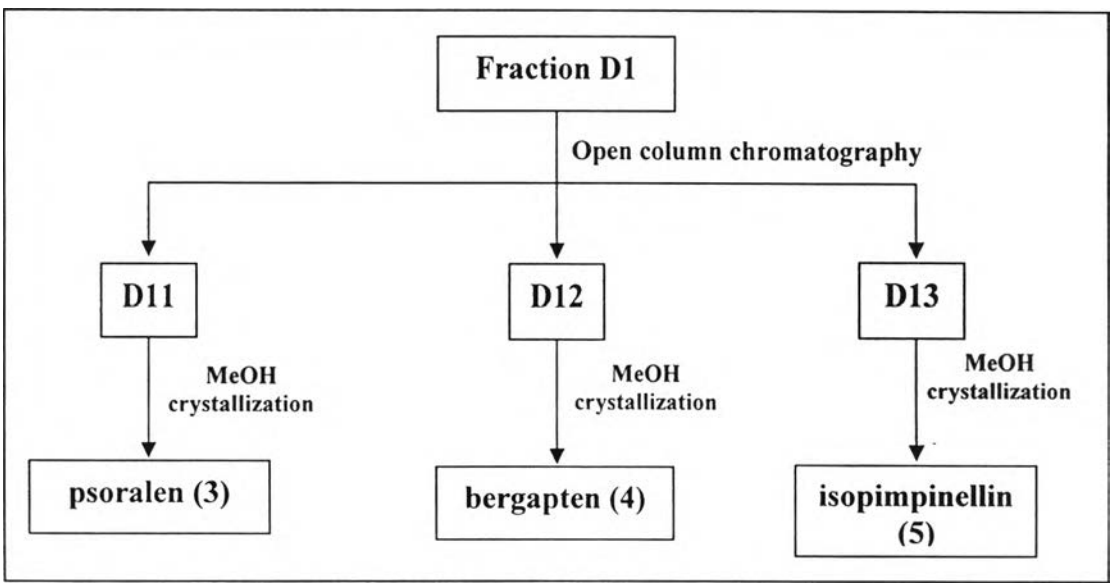
The methanol layer was evaporated under reduced pressure condition and fractionated by quick column chromatography which used two mixed solvent systems, CH_2Cl_2/Hex and MeOH/CH_2Cl_2, to yield 2 subfractions, D1 and D2.

The subfraction D1 was fractionated with silica gel column chromatography using mixed solvents (50% CH_2Cl_2/Hex to 100% CH_2Cl_2 and 5% to 50% EtOAc/CH_2Cl_2) to yield 3 fractions, D11, D12 and D13, respectively. All fractions were crystallized in methanol to yield psoralen (**3,** 0.025g, 0.04% w/w; Jiangning *et al*, 1999), bergapten (**4,**

0.02g, 0.03% w/w; Lui *et al*, 2004) and isopimpinellin (**5**, 0.012g, 0.02% w/w; Lui *et al*, 2004), respectively.



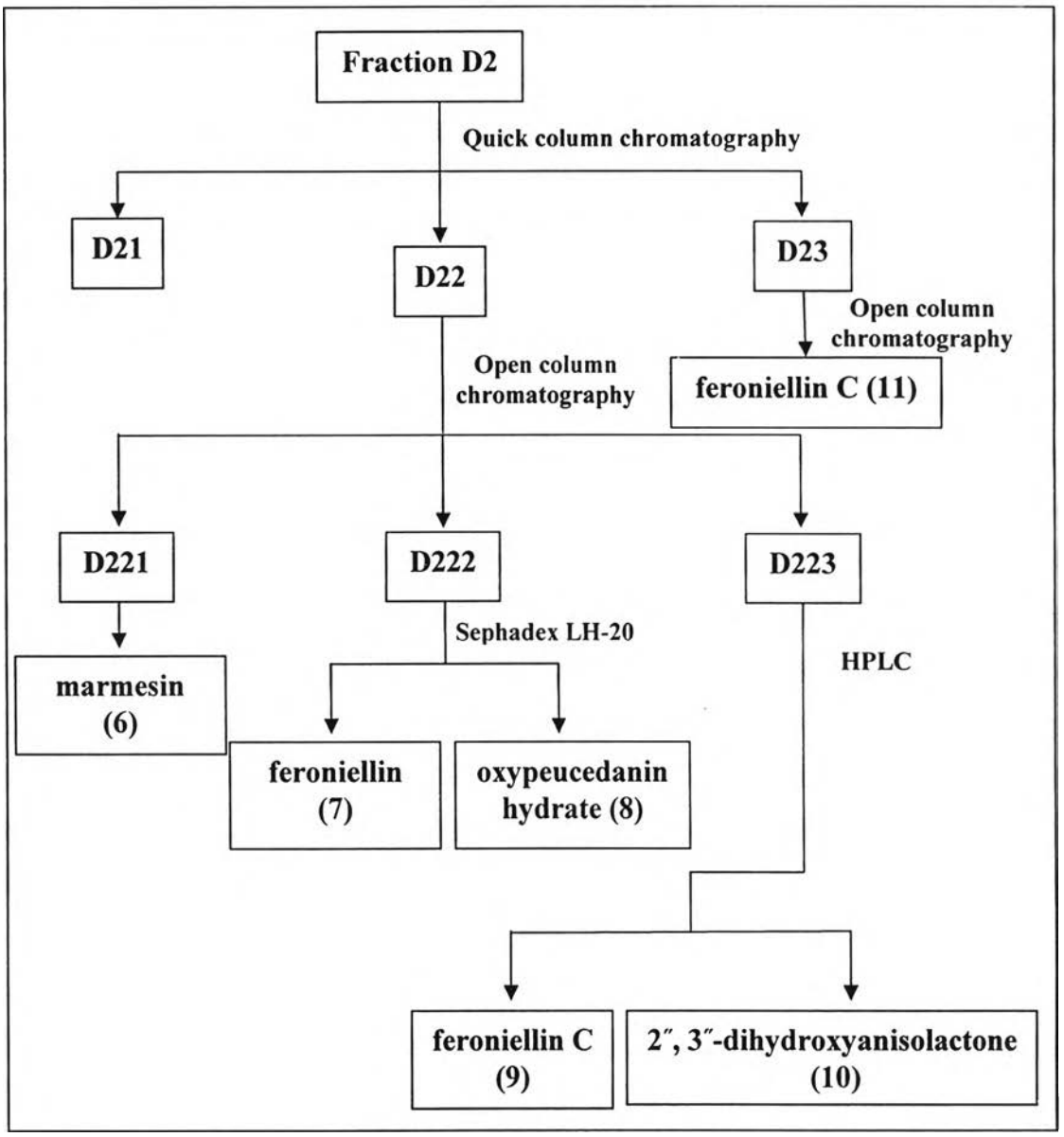
Scheme 2.2 Purification of anisolactone (**1**) and 2″, 3″-epoxyanisolactone (**2**) and fractionation of dichloromethane extract.



Scheme 2.3 Purification procedure of psoralen (**3**), bergapten (**4**) and isopimpinellin (**5**)

The fraction D2 was fractionated with quick column chromatography using mixed solvents (100% CH_2Cl_2 to 100% EtOAc and 5% to 50% MeOH/EtOAc) to yield 3 fractions, D21, D22 and D23 (**Scheme 2.4**), respectively. The fraction D22 was further fractionated by silica gel column chromatography which was eluted with 100% CH_2Cl_2 to 100% EtOAc and 1% to 10% MeOH/EtOAc to yield 3 fractions, D221, D222 and D223. The fraction D221 was crystallized with hexane-dichloromethane-methanol solvent system to give marmesin (**6**, 0.005g, 0.007% w/w; Hagemeier *et al*, 1999). The fraction D222 was separated by size exclusion chromatography, Sephadex LH-20 as stationary phase, using mixed solvent system of hexane, dichloromethane and methanol with the proportion to 5:5:1 to yield pure form of feroniellin A (**7**, 0.2g, 0.29% w/w) and oxypeucedanin hydrate (**8**, 0.082g, 0.12% w/w; Tesso *et al*, 2005). The fraction D223 was fractionated by Sephadex LH-20 using mixed solvents of hexane, dichloromethane and methanol with the proportion to 5:5:1 to yield mixture of two compounds prior to purify by HPLC with isocratic condition of 20% H_2O/MeOH to yield feroniellin C (**9**, 0.007g, 0.01% w/w) and 2″, 3″-dihydroxyanisolactone (**10**, 0.005g, 0.007% w/w; Lakshmi *et al*, 1985).

The fraction D23 was fractionated by silica gel column chromatography using mixed solvent systems (100% CH_2Cl_2 to 100% EtOAc and 1% to 10% MeOH/EtOAc) to yield pure form of feroniellin B (**11**, 0.06g, 0.09% w/w).



Scheme 2.4 Purification process of marmesin (**6**), feroniellin A (**7**), oxypeucedanin hydrate (**8**), feroniellin C (**9**), 2″, 3″-dihydroxyanisolactone (**10**) and feroniellin B (**11**).

Anisolactone (1) : white fine needle; UV(MeOH)λ_{max} 217, 266 nm; ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 1.79 (3H, s, H-9″), 1.92 (3H, s, H-10″), 2.35 (1H, dd, *J* = 14.4, 8.2 Hz, H-4″a), 2.46 (1H, dd, *J* = 10.4, 5.0 Hz, H-4″b), 4.96 (2H, d, *J* = 6.8 Hz, H-1″), 5.50 (1H, brm, H-5″), 5.67 (1H, t, *J* = 6.4 Hz, H-2″), 6.29 (1H, d, *J* = 9.6 Hz, H-3), 6.95 (1H, d, *J* = 1.2 Hz, H-2′), 6.98 (1H, s, H-6″), 7.17 (1H, s, H-8), 7.61 (1H, d, *J* = 2.0 Hz, H-2′), 8.15 (1H, d, *J* = 10.0 Hz, H-4).

2″, 3″-Epoxyanisolactone (2) : white flake crystal; UV(MeOH)λ_{max} 217, 266 nm; ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 1.49 (3H, s, H-9″), 1.66 (2H, q, *J* = 14.0, 10.0, 4.0 Hz, H-4″), 1.93 (3H, s, H-10″), 3.29 (1H, t, *J* = 8.4 Hz, H-2″), 4.44 (1H, dd, *J* = 11.0, 6.6 Hz, H-1″a), 4.70 (1H, dd, *J* = 11.4, 4.6 Hz, H-1″b), 5.08 (1H, brm, H-5″), 6.32 (1H, d, *J* = 9.6 Hz, H-3), 7.08 (1H, s, H-6″), 7.18 (1H, *s*, H-8), 7.61 (1H, brs, H-2′), 7.62 (1H, d, *J* = 2.0 Hz, H-3′), 8.26 (1H, d, *J* = 9.6 Hz, H-4).

Psoralen (3) : white needle crystal; UV(MeOH)λ_{max} 216, 247, 275 nm; ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 6.38 (1H, d, *J* = 9.6 Hz, H-3), 6.83 (1H, d, *J* = 1.2 Hz, H-2′), 7.48 (1H, s, H-8), 7.69 (1H, s, H-5), 7.70 (1H, d, *J*= 2.4 Hz, H-3′), 7.80 (1H, d, *J* = 9.6 Hz, H-4).

Bergapten (4) : white needle crystal; UV(MeOH)λ_{max} 221, 267 nm; ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 4.27 (3H, s, -OCH_3-5), 6.28 (1H, d, *J* = 9.6 Hz, H-3), 7.02 (1H, d, *J* = 1.6 Hz, H-2′), 7.14 (1H, s, H-8), 7.63 (1H, d, *J* = 2.0 Hz, H-3′), 8.12 (1H, d, *J* = 9.6 Hz, H-4).

Isopimpinellin (5) : green needle crystal; UV(MeOH)λ_{max} 217, 268 nm; ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 4.16 (6H, s, 2 × -OCH_3-5, 8), 6.29 (1H, d, *J* = 9.6 Hz, H-3), 7.02 (1H, d, *J* = 1.6 Hz, H-2′), 7.63 (1H, d, *J* = 2.0 Hz, H-3′), 8.12 (1H, d, *J* = 10.0 Hz, H-4).

Marmesin (6) : colorless flake crystal; UV(MeOH)λ_{max} 254, 309 nm; ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 1.23 (3H, s, -CH_3-1″), 1.37 (3H, s, -CH_3-1″), 3.21 (2H, m, H-3′), 4.74 (1H, t, *J* = 8.8 Hz, H-2′), 6.22 (1H, d, *J* = 9.6 Hz, H-3), 6.75 (1H, s, H-8), 7.22 (1H, s, H-5), 7.60 (1H, d, *J* = 9.6 Hz, H-4).

Feroniellin A (**7**) : : pale yellow amorphous crystal; UV(MeOH)λ_{max} 228, 249, 310 nm; $[\alpha]^{23}_{D}$ +7.11° (*c* 0.38, CH_2Cl_2); ^{1}H-NMR ($CDCl_3$, 400 MHz) see **Table 2.1**; ^{13}C-NMR ($CDCl_3$, 100 MHz) See **Table 2.1**; HREIMS *m/z* 388.2716 (*calcd* for $C_{21}H_{24}O_7$, 388.2716).

Oxypeucedanin hydrate (**8**) : colorless flake crystal; UV(MeOH)λ_{max} 222, 249, 310 nm; ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 1.31 (3H, s, -CH_3-4″), 1.36 (1H, s, -CH_3-5″), 3.91 (1H, dd, *J* = 7.8, 2.8 Hz, H-1″b), 4.45 (1H, t, *J* = 8.8 Hz, H-2″), 4.54 (1H, dd, *J* = 9.8, 3.0 Hz, H-1″a), 6.29 (1H, d, *J* = 10.0 Hz, H-3), 6.99 (1H, d, *J* = 1.2 Hz, H-2′) 7.16 (1H, s, H-8), 7.61 (1H, d, *J* = 2.4 Hz, H-3′), 8.17 (1H, d, *J* = 9.6 Hz, H-4).

Feroniellin C (**9**) : pale yellow amorphous crystal; UV(MeOH)λ_{max} 224, 249, 309 nm; $[\alpha]^{23}_{D}$ +10.58° (*c* 0.1, CH_2Cl_2); ^{1}H-NMR ($CDCl_3$, 400 MHz) See **Table 2.2**; ^{13}C-NMR ($CDCl_3$, 100 MHz) See **Table 2.2**; HREIMS *m/z* 388.2716 (*calcd* for $C_{21}H_{24}O_7$, 388.2716).

2″, 3″-dihydroxyanisolactone (**10**): colorless needle crystal; UV(MeOH)λ_{max} 224, 249, 310 nm; ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 1.35 (3H, s, H-10″), 1.90 (3H, s, H-9″), 2.44 (2H, brm, H-4″), 4.06 (1H, brd, *J* = 6 Hz, H-1″b), 4.56 (1H, dd, *J* = 9.6, 1.2 Hz, H-1″a), 5.22 (1H, *brm*, H-5″), 5.32 (1H, *t*, *J* = 9.0 Hz, H-2″), 6.18 (1H, d, *J* = 9.6 Hz, H-3), 6.91 (1H, brs, H-2′), 7.03 (1H, s, H-8), 7.06 (1H, brs, H-6″), 7.54 (1H, d, *J* = 2.0 Hz, H-3′), 8.08 (1H, d, *J* = 10 Hz, H-4).

Feroniellin B (**11**) : dark yellow liquid; UV(MeOH)λ_{max} 228, 250, 309 nm; $[\alpha]^{23}_{D}$ +21.74° (*c* 0.92, CH_2Cl_2); ^{1}H-NMR ($CDCl_3$, 400 MHz) See **Table 2.3**; ^{13}C-NMR ($CDCl_3$, 100 MHz) See **Table 2.3**; HREIMS *m/z* 388.2716 (*calcd* for $C_{21}H_{24}O_7$, 388.2716).