Chemical Constituents from the Twigs of *Garcinia hombroniana*, the Leaves of *Garcinia prainiana* and the Roots of *Clerodendrum petasites* S. Moore

Saranyoo Klaiklay

A Thesis Submitted in Partial Fulfillment of the Requirements for the Degree of

Master of Science in Organic Chemistry

Prince of Songkla University

2009

Thesis Title	Chemical Constituents from the Twigs of *Garcinia hombroniana*, the Leaves of *Garcinia prainiana* and the Roots of *Clerodendrum petasites* S. Moore
Author	Mr. Saranyoo Klaiklay
Major Program	Organic Chemistry

Major Advisor:

…….
(Dr. Yaowapa Sukpondma)

Co-advisor:

…….
(Prof. Dr. Vatcharin Rukachaisirikul)

Examining Committee:

……...Chairperson
(Dr. Pornsiri Leewanich)

……...……...
(Dr. Yaowapa Sukpondma)

……...……...
(Prof. Dr. Vatcharin Rukachaisirikul)

……...……...
(Asst. Prof. Dr. Kanda Panthong)

The Graduate School, Prince of Songkla University, has approved this thesis as partial fulfillment of the requirements for the Degree of Master of Science in Organic Chemistry.

…….……..
(Assoc. Prof. Dr. Krerkchai Thongnoo)
Dean of Graduate School

ชื่อวิทยานิพนธ์ องค์ประกอบทางเคมีจากกิ่งวา (*Garcinia hombroniana*) ใบจุมพุต (*Garcinia prainiana*) และ รากท้าวยายม่อม (*Clerodendrum petasites* S. Moore)

ผู้เขียน นายศรัณยู ใคลคลาย

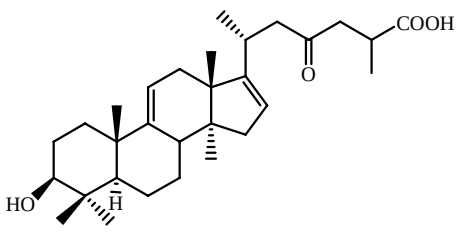
สาขาวิชา เคมีอินทรีย์

ปีการศึกษา 2551

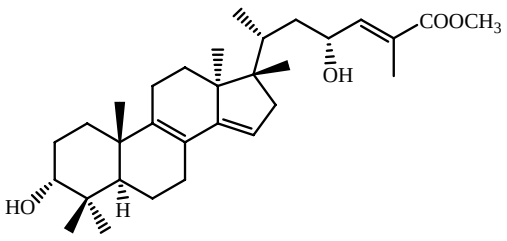
บทคัดย่อ

การศึกษาองค์ประกอบทางเคมีแบ่งเป็น 2 ตอน ตอนแรกเป็นการนำส่วนสกัดหยาบเมทานอลจากกิ่งวา (*Garcinia hombroniana*) และใบจุมพุต (*Garcinia prainiana*) ทำการแยกให้บริสุทธิ์ด้วยวิธีทางโครมาโทรกราฟี สามารถแยกสารใหม่ได้จำนวน 8 สาร เป็นสารประเภท triterpene จำนวน 4 สาร (**SK9 SK11 SK19** และ **SK21**) และสารประเภท xanthone จำนวน 4 สาร (**SK10 SK18 SK20** และ **SK22**) และยังสามารถแยกสารที่มีรายงานโครงสร้างแล้ว จำนวน 11 สาร ซึ่งเป็นสารประเภท triterpene จำนวน 4 สาร (**SK1 SK2 SK3** และ **SK12**) สารประเภท xanthone จำนวน 5 สาร (**SK4 SK5 SK8 SK13** และ **SK16**) สารประเภทอนุพันธ์ของกรดเบนโซอิก จำนวน 2 สาร (**SK7** และ **SK17**) และสารประเภท biflavone จำนวน 1 สาร (**SK6**) จากกิ่งวา ส่วนใบจุมพุต สามารถแยกสารประเภท flavonone glucoside ได้จำนวน 2 สาร (**SK23** และ **SK24**) ตอนที่สองเป็นการนำส่วนสกัดหยาบจากรากท้าวยายม่อม (*Clerodendrum petasites* S. Moore) มาแยกและทำให้บริสุทธิ์ด้วยวิธีทางโครมาโทรกราฟี สามารถแยกสารประเภท flavone จำนวน 2 สาร (**SK14** และ **SK15**)

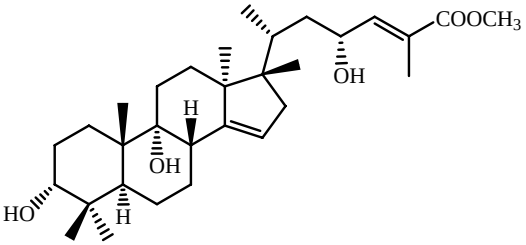
การวิเคราะห์โครงสร้างสารอาศัยข้อมูลทางสเปกโทรสโกปี โดยเฉพาะข้อมูล 1D และ 2D NMR สเปกโทรสโกปี ซึ่งโครงสร้างของ **SK19** วิเคราะห์ในรูปอนุพันธ์อะซิเตท ส่วนสารที่มีการรายงานโครงสร้างแล้ว วิเคราะห์ได้จากการเปรียบเทียบข้อมูล NMR สเปกตรัม และค่าการหมุนระนาบแสง



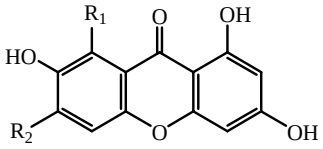
SK1



SK2



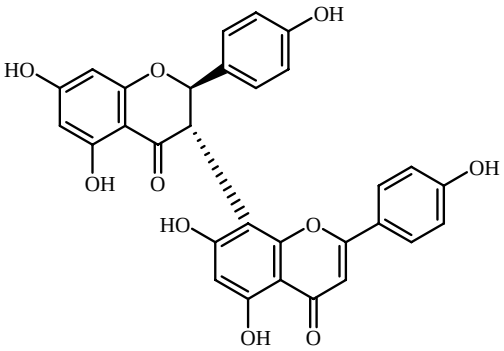
SK3



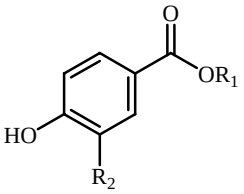
SK4 : R_1 = H R_2 = OH

SK5 : R_1 = H R_2 = H

SK8 : R_1 =  R_2 =OH

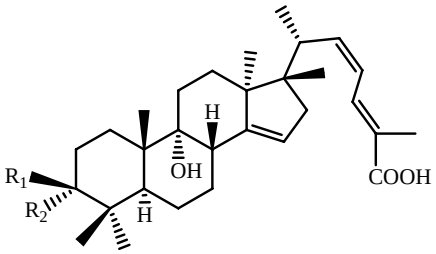


SK6



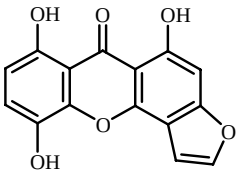
SK7 : R_1 = H, R_2 = H

SK17 : R_1 = CH_3, R_2 = OH

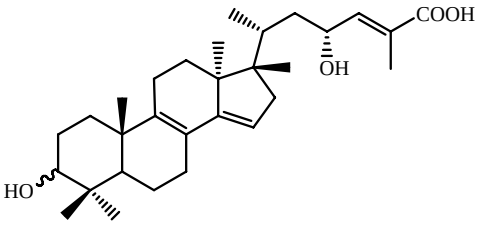


SK9 : R_1 = OH, R_2 = H

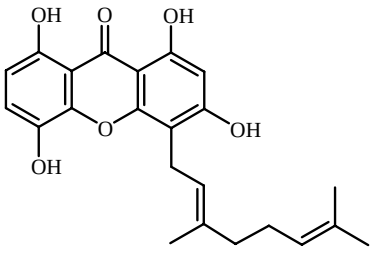
SK12 : R_1 = H, R_2 = OH



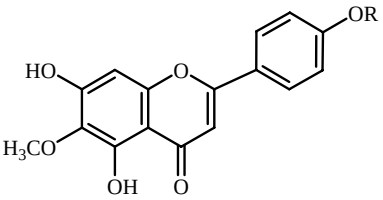
SK10



SK11

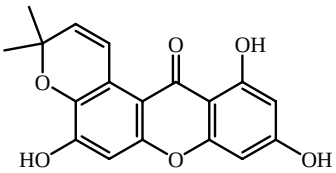


SK13

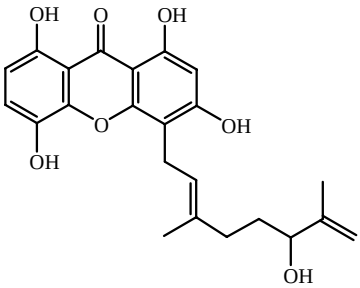


SK14 : R = H

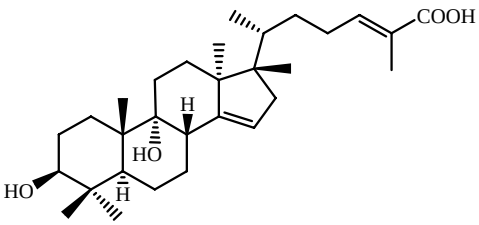
SK15 : R = CH_3



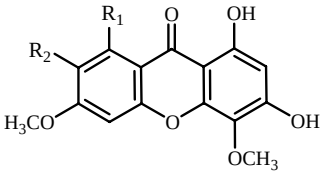
SK16



SK18

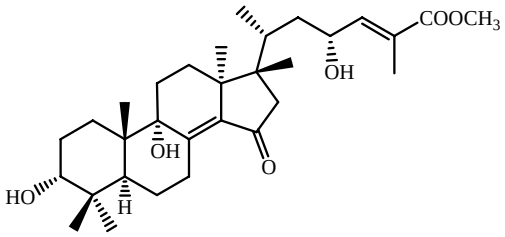


SK19

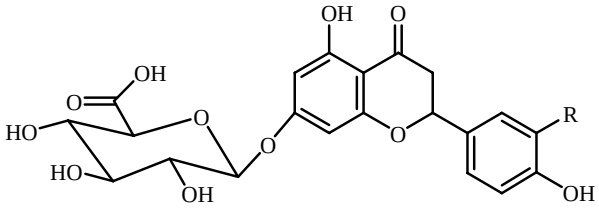


SK20 : R_1 = OH, R_2 = H

SK22 : R_1 = H, R_2 = OH



SK21



SK23 : R = H

SK24 : R = OH

Thesis Title Chemical Constituents from the Twigs of *Garcinia hombroniana***,** the Leaves of *Garcinia prainiana* and the Roots of *Clerodendrum petasites* S. Moore

Auther Mr. Saranyoo Klaiklay

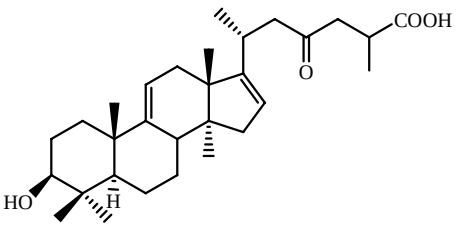
Major Program Organic Chemistry

Academic Year 2008

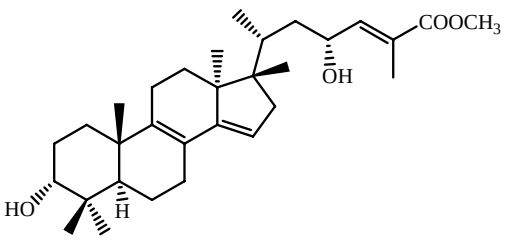
ABSTRACT

Chemical investigation was divided into two parts. The first part involved the chromatographic separation of the crude methanol extracts from the twigs of *Garcinia hombroniana* and the leaves of *Garcinia prainiana*. Eight new compounds: four triterpenes (**SK9**, **SK11**, **SK19** and **SK21**) and four xanthones (**SK10**, **SK18**, **SK20** and **SK22**), together with eleven known compounds: four triterpenes (**SK1**, **SK2**, **SK3** and **SK12**), five xanthones (**SK4**, **SK5**, **SK8**, **SK13** and **SK16**), two benzoic acid derivatives (**SK7** and **SK17**) and one biflavone (**SK6**) were isolated from the twigs of *Garcinia hombroniana* while the leaves of *Garcinia prainiana* yielded two flavonone glucosides (**SK23** and **SK24**). The second part was the investigation of the crude methanol extract from the roots of *Clerodendrum petasites* using various chromatographic techniques. Two known flavones (**SK14** and **SK15**) were obtained.

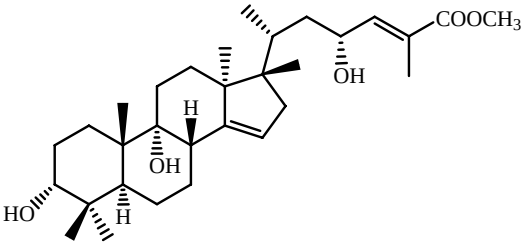
The structures were identified by analysis of UV, IR, 1D and 2D NMR spectroscopic data. Compound **SK19** was identified as its acetate derivative. Known compounds were also identified by comparison of their NMR data and optical rotation with those reported in the literatures.



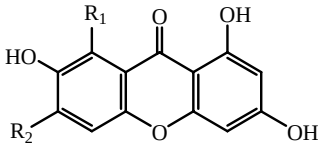
SK1



SK2



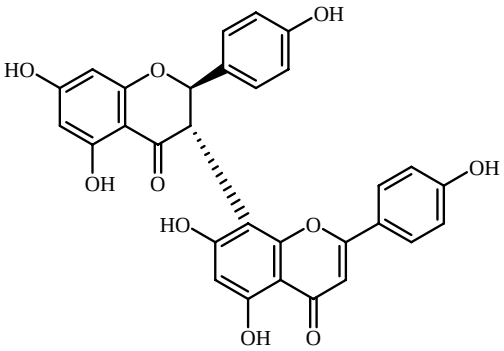
SK3



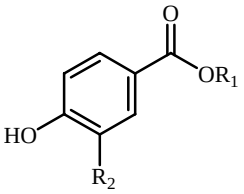
SK4 : R_1 = H R_2 = OH

SK5 : R_1 = H R_2 = H

SK8 : R_1 =  R_2 =OH

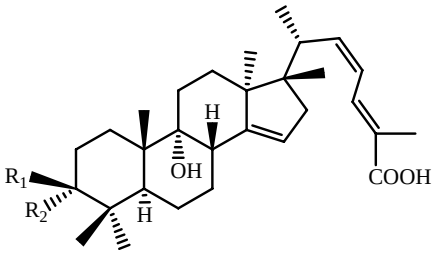


SK6



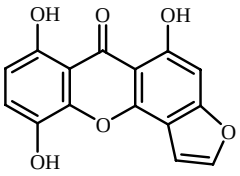
SK7 : R_1 = H, R_2 = H

SK17 : R_1 = CH_3, R_2 = OH

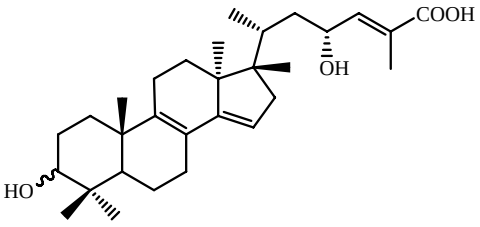


SK9 : R_1 = OH, R_2 = H

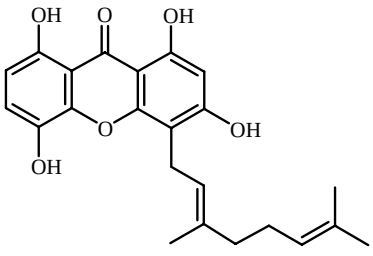
SK12 : R_1 = H, R_2 = OH



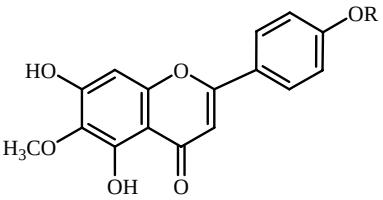
SK10



SK11

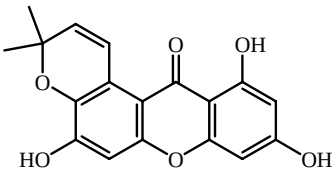


SK13

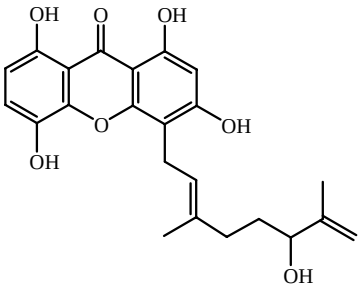


SK14 : R = H

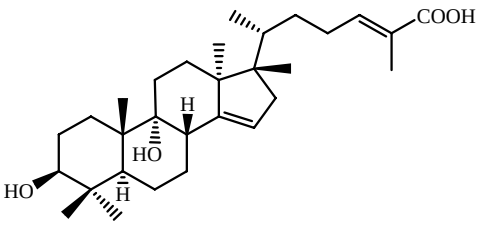
SK15 : R = CH_3



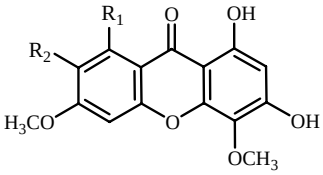
SK16



SK18

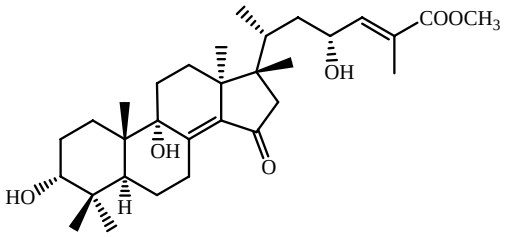


SK19

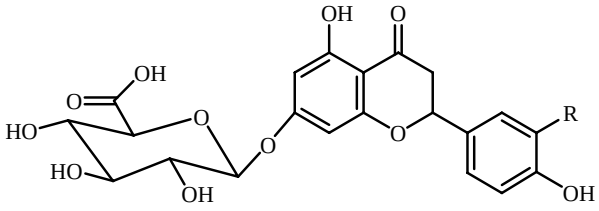


SK20 : R_1 = OH, R_2 = H

SK22 : R_1 = H, R_2 = OH



SK21



SK23 : R = H

SK24 : R = OH

ACKNOWLEDGEMENT

I wish to express my deepest and sincere gratitude to my supervisor, Dr. Yaowapa Sukpondma, for her valuable instruction, expert guidance and excellent suggestion. I would also like to express my appreciation to her for correction of my thesis.

My sincere thanks are expressed to Professor Dr. Vatcharin Rukachaisirikul, my co-advisor for her kindness and valuable advice, to Dr. Pornsiri leewanich and Assistant Professor Dr. Kanda Panthong for the valuable comments.

I would like to extend my appreciation to the staff of the Department of Chemistry, Faculty of Science, Prince of Songkla University, for making this thesis possible.

This work was made possible by a scholarship from Center for Innovation in Chemistry (PERCH-CIC). In addition, I would also like to thank the Graduate School, Prince of Songkla University, for material support.

Finally, none of this would have been possible without love and encouragement of my family and friends. I thank them all for their kindness and valuable advice. Everything will be always kept in my mind.

Saranyoo Klaiklay

THE RELEVANCE OF THE RESEARCH WORK TO THAILAND

Species belonging to the *Garcinia* and *Clerodendrum* genera are well known to be rich in a variety of compounds. Some of compounds showed interesting biological and pharmacological activities such as cytotoxic, antifungal, antioxidant, anti-HIV, antimalaria and antibacterial activities. This research work involved isolation and structural elucidation of compounds isolated from the twigs of *G. hombroniana*, the leaves of *G. prianiana* and the roots of *C. petasites*. Eight new compounds and sixteen known compounds were isolated. The isolated compounds will be further evaluated for antibacterial and anti-HIV activities.

CONTENTS

CONTENTS (Continued)

CONTENTS (Continued)

LIST OF TABLES

LIST OF TABLES (Continued)

LIST OF TABLES (Continued)

LIST OF TABLES (Continued)

LIST OF TABLES (Continued)

LIST OF TABLES (Continued)

LIST OF TABLES (Continued)

LIST OF FIGURES

LIST OF FIGURES (Continued)

LIST OF ABBREVIATIONS AND SYMBOLS

s	=	*singlet*
d	=	*doublet*
t	=	*triplet*
q	=	*quartet*
m	=	*multiplet*
brs	=	*broad singlet*
brd	=	*broad doublet*
dd	=	*doublet of doublet*
dt	=	*doublet of triplet*
dq	=	*doublet of quartet*
ddd	=	*doublet of doublet of doublet*
ddq	=	*doublet of doublet of quartet*
ddm	=	*doublet of doublet of multiplet*
mt	=	*multiplet of triplet*
qd	=	*quartet of doublet*
δ	=	chemical shift relative to TMS
J	=	coupling constant
m/*z*	=	a value of mass divided by charge
°C	=	degree celcius
R_f	=	retention factor
g	=	gram
kg	=	kilogram
mg	=	milligram
μg	=	microgram
ml	=	milliliter

LIST OF ABBREVIATIONS AND SYMBOLS (Continued)

L	=	Liter
cm^{-1}	=	reciprocal centimeter (wave number)
nm	=	nanometer
ppm	=	part per million
λ_{max}	=	maximum wavelength
ν	=	absorption frequencies
$\mathcal{E}$	=	molar extinction coefficient
Hz	=	Hertz
MHz	=	megaHertz
$[\alpha]_D$	=	specific rotation
c	=	concentration
TLC	=	thin-layer chromatography
UV-S	=	Ultraviolet-short wavelength
FT-IR	=	Fourier Transform Infrared
MS	=	Mass Spectroscopy
EIMS	=	Electron Impact Mass Spectroscopy
NMR	=	Nuclear Magnetic Resonance
1D NMR	=	One Dimensional Nuclear Magnetic Resonance
2D NMR	=	Two Dimensional Nuclear Magnetic Resonance
HMQC	=	Heteronuclear Multiple Quantum Coherence
HMBC	=	Heteronuclear Multiple Bond Correlation
DEPT	=	Distortionless Enhancement by Polarization Transfer
NOE	=	Nuclear Overhauser Effect
NOEDIFF	=	NOE Difference Spectroscopy
NOSEY	=	Nuclear Overhauser Enhanced Spectroscopy
COSY	=	Correlation Spectroscopy
TMS	=	tetramethylsilane

LIST OF ABBREVIATIONS AND SYMBOLS (Continued)

$CDCl_3$	=	deuterochloroform
Acetone-d_6	=	hexadeuteroacetone
ASA	=	anisaldehyde-sulphuric acid in acetic acid solution
CD_3OD	=	tetradeuteromethanol
DMSO-d_6	=	hexadeuterodimethylsulphoxide
$CHCl_3$	=	chloroform
CH_2Cl_2	=	dichloromethane
EtOH	=	ethanol
EtOAc	=	ethyl acetate
HCl	=	hydrochloric acid
HCOOH	=	formic acid
H_2O	=	water
MeOH	=	methanol
$NaHCO_3$	=	sodium hydrogen carbonate
NaOH	=	sodium hydroxide
Na_2SO_4	=	sodium sulfate
Petrol	=	Petroleum ether

PART I

CHEMICAL CONSTITUENTS FROM THE TWIGS OF *GARCINIA HOMBRONIANA* AND THE LEAVES OF *GARCINIA PRAINIANA*

CHAPTER 1.1

INTRODUCTION

1.1.1 Introduction

1.1.1.1 *Garcinia hombroniana*

Garcinia hombroniana, a plant belonging to the Guttiferae family, is widely distributed in the southern part of Thailand. *G. hombroniana* is small to medium size tree about 30 to 60 feet high, 180 cm girth. Inner bark with opaque, white or yellow exudates. Leaves: stalk 15-20 mm, stout, irregularly, often finely, transversely striate and drying golden; blade very variable in size, ovate to ovate-oblong, 6.5x3.5-9.5x5.5-15.5x7 cm; broadly tapered to apex; base broadly wedge-shaped or, less usually, rounded, drying warn, chestnut brown, or occasionally blackish brown; leathery; midrib broad, flat, slightly raised on upper surface, strongly raised below, keeled, striate towards base, nerves faint to almost invisible; secondaries fine, parallel, straight, fairly close, 2 mm apart, sometime forking outwards, with equally prominent or fainter intercostals faintly looping and joining to form a weak intramarginal nerve. Flower with 4 sepals and petals, terminal; males in clusters, pedicel 5-10 mm, opening 2.5 cm across staments in a slightly 4-lobed mass surrounding a pistil lode; female solitary, with no staminodes. Fruits globose, to 4 cm across, usually depressed, wall thin, woody, drying brown shiny, amooth, tending to fracture; stigma raised on, and slightly projecting from a distinct apical beak, 1-10 mm long, thin margin wavy or with touching lobes, surface weakly finely papillose; seated on the persistent calyx stout, 1-7 mm. In Thailand, *G. hombroniana* has a local name, ‘‘Waa’’ (Saelim, 2005).

1.1.1.2 *Garcinia prainiana*

Garcinia prainiana, a plant belonging to the Guttiferae family, is widely distributed in the southern part of Thailand. *G. prainiana* is a small tree, 10 m tall crown narrow, dense, with milky latex; branchlets not angled, glabrous. Stipules

absent. Leaves simple, opposite, petioles 3 mm long, stout, blades conriaceous, ovate-oblong, 15-23 by 7-15 cm, the slightly heart-sharped base often clasping the twig, apex acuminate, margins entrie, deep green and glabrous on both surfaces, nerves 12-15 pairs. Flower unisexual, in dense terminal cymes, male and female flowers on the same plant. Male flowers 2.5 cm across, sepals 5, free, the outer 2 smaller than the inner, orbicular, 5 mm long, red with green margin, fleshy, petal 5, free, sur-orbilar, 8 mm long, pink, stamens numerous, filament red, anthers yellow, connate into 5 bundles around a pistillode, pistillode globose, red, with numerous tubercles. Female flowers 3.5 cm across, sepals 5, free, orbicular, 7 mm long, pale green with pink stripe at center, petals 5, free, obovate, 10 mm long, red, when young then creamy white, staminode none, ovary superior, globose, glabrous, pale green, 6 mm. diam., 7- to 8-loculed, ovule 1 in each locule, pink, stima sessile, red, 6-7 mm diam., dome-shaped, margin entire. Fruits a fleshy berry, depressed globose, 2.5-4.5 cm across, with a thin and smooth leathery rind, ripening golden yellow to orange yellow. Seeds 5-8, suborbicular, compressed, 1.3 by 1.0 cm, pale brown, embedded in fleshy orange pulp (Upo, 2005).

1.1.2 Review of Literatures

Chemical constituents from the genus *Garcinia*

Plants in the *Garcinia* genus (Guttiferae) are well known to be rich in a variety of compounds: xanthones (Shadid, 2007; Reutrakul, 2007; Deachathai, 2006; Jung, 2006; Panthong, 2006; Rukachaisirikul, 2006; Suksumrarn, 2006), benzophenones (Kumar, 2007a; Hamed, 2006; Masullo, 2008; Soemiati, 2006), biflavonoids (Lu, 2008, Mbwanbo, 2006), biphenyls (Chen, 2006; Wu, 2008), flavonoids (Okwu, 2007; Hartati, 2007; Shen, 2007b), depsidones (Rukachaisikul, 2006), alkaloids (Fotie, 2007) and triterpenes (Shadid, 2007; Shen, 2006a; Rukachaisirikul, 2005). Some of these compounds showed interesting biological and pharmacological activities such as cytotoxic (Akao, 2008; Kijjao, 2008; Han, 2006a,b; Kumar; 2007b; Suksamrarn, 2006), anti-inflammatory (Castardo, 2008; Chen, 2008; Huang, 2008; Lin, 2006), antimicrobial (Taher, 2008; Kuete, 2007; Okwu, 2007), antifungal (Dharmarate,

2005), antibacterial (Rukachaisirikul, 2008; 2006; 2005; Panthong, 2006; Sukpondma, 2005), anti-HIV (Reutrakul, 2007) and antioxidant (Wu, 2008a; Tarher, 2007; Yu, 2007; Okwu, 2007; Lannang , 2006) activities.

Chemical constituents isolated from *Garcinia* species up to the year 2005 have been reported (Naklue, 2006). The continuing search using SciFinder database revealed additional chemical constituents in the year 2006 up to 2008 which were summarized in **Table 1**.

Table 1 Compounds from the *Garcinia* genus

Scientific name	Investigated parts	Compounds	Structures	References
G. afzelii	stem barks	afzeliixanthone A	**12.3hh**	Kamdem, W., *et al*., 2006
		afzeliixanthone B	**12.2c**	
		β-sitosterol	**10a**	
		stigmasterol	**10b**	
		1,7-dihydroxy-xanthone	**12.1f**	
		1,5-dihydroxy-xanthone	**12.1d**	
		1,3,7-trihydroxy-2-(3-methylbut-2-enyl)xanthone	**12.2l**	
G. benthami	stem barks	salimbenzophe-none	**2t**	Elya, B., *et al*., 2006b
G. brasiliensis	roots	7-epiclusianone	**2x**	Neves, J. S., *et al*., 2007
	fruits	garciniaphenone	**2cc**	Martins, F. T., *et al*., 2008
	seeds	guttiferone A	**2u**	Martins, F. T., *et al*., 2007

Table 1 (continued)

Scientific name	Investigated parts	Compounds	Structures	References
G. brevipedi-cellata	stem barks	brevipsidone scopoletin damnacanthal Pilloin	**6c** **13g** **13j** **7e**	Ngoupayo, J., *et al*., 2007
G. cambogia	fruits fruits rinds	guttiferone I guttiferone J guttiferone K guttiferone M guttiferone N oxyguttiferone K Garcinia lactone	**2p** **2q** **2r** **2n** **2z** **12.6mm** **13f**	Masullo, M., *et al*., 2008 Mahapatra, S., *et al*., 2007
G. cantleyana	leaves and trunk barks	cantleyanone A 7-hydroxyforbe-sione Cantleya none B cantleyanone C cantleyanone D 4-(1,1-dimethyl-prop-2-enyl)-1,3,-5,8-tetrahydroxy-xanthone deoxygaudichau-dione A gaudichaudione H Friedelin	**12.6a** **12.6b** **12.6c** **12.6d** **12.6e** **12.3uu** **12.6bb** **12.6ii** **11b**	Shadid, K. A., *et al*., 2007

Table 1 (continued)

Scientific name	Investigated parts	Compounds	Structures	References
		Garbogiol	**12.3aaaa**	
		macranthol	**13n**	
		glutin-5-en-3β-ol	**11c**	
G. cowa	stems	garccowaside A	**8b**	Shen, J., *et al*., 2007b
		garccowaside B	**8c**	
		garccowaside C	**8d**	
		Quercetin	**7b**	
		2-(3,5-dihydroxy-phenyl)-2,3-dihy-dro-5,7-dihydro-xyflavone	**7g**	
		2-(3,5-dihydroxy-phenyl)-2,3-dihy-dro-3,5,7-trihy-droxyflavone	**7h**	
	fruits and stems	(+)-6-(3,4-dihy-droxybenzoyl)-2,-3,4,4a,8,9,10,11,-12,12a-decahydro-3,3,4a,9,9-penta-methyl-8,10-bis(3-methyl-2-buten-1-yl)-1H-8,11a-me-thano-7H-benzo-[b]cycloocta[e]-pyran-7-13-dione	**2aa**	Shen, J., *et al*., 2007a

Table 1 (continued)

Scientific name	Investigated parts	Compounds	Structures	References
	stems	Cambogin	**2e**	Shen, J., *et al*., 2006c
		1,5,6-trihydroxy-3-methoxy-4-(3-hydroxyl-3-methylbutyl)xanthone	**12.3kk**	
		1,5-dihydroxy-3-methoxy-6',6'-dimethyl-2H-pyrano(2',3':6,7)-4-(3-methylbut-2-enyl)xanthone	**12.3ddd**	
		1,3,5-trihydroxy-6',6'-dimethyl-2H-pyrano(2',3':6,7)-xanthone	**12.3eee**	
		dulxanthone A	**12.3gg**	
		1,5,6-trihydroxy-3,7-dimethoxy-xanthone	**12.4a**	
		1,7-dihydroxy-xanthone	**12.1f**	
		1,3,5-trihydroxy-6-methoxyxanthone	**12.3ii**	
		norathyriol	**12.3cc**	

Table 1 (continued)

Scientific name	Investigated parts	Compounds	Structures	References
	fruits	cowaxanthone A	**12.3w**	Panthong, K., *et al*., 2006
		cowaxanthone B	**12.3e**	
		cowaxanthone C	**12.3nnn**	
		cowaxanthone	**12.3l**	
		cowaxanthone D	**12.3sss**	
		cowaxanthone E	**12.3c**	
		fuscaxanthone C	**12.3f**	
		7-*O*-methyl garcinone E	**12.3d**	
		mangostanin	**12.3www**	
		1,6-dihydroxy-3,-7-dimethoxy-2-(3-methyl-2-butenyl)xanthone	**12.3v**	
		6-*O*-methyl-mangostanin	**12.3yyy**	
		α-mangostin	**12.3a**	
		β-mangostin	**12.3b**	
		cowanol	**12.3m**	
		Cowanin	**12.3y**	
	fruits	β-sitosterol	**10a**	Shen, J., *et al*., 2006a
		daucosterol	**10c**	
		amentoflavone	**4e**	
		cirsiumaldehyde	**13h**	
		p-coumaric acid	**13b**	
		morelloflavone	**4a**	

Table 1 (continued)

Scientific name	Investigated parts	Compounds	Structures	References
G. dulcis	fruits	morelloflavone	**4a**	Hutadilok-Towatana, N., *et al*., 2007
		camboginol	**2d**	
	flowers	Dulcinone	**13o**	Deachathai, M., *et al*., 2006
		dulcisxanthone C	**12.4b**	
		dulcisxanthone D	**12.3ffff**	
		dulcisxanthone E	**12.3pp**	
		dulcisxanthone F	**12.3ttt**	
		rhamnazin	**7a**	
G. eugeniae-folia	stem barks	eugeniaphenone	**2f**	Hartati, S., *et al*., 2008a
	stem barks	enervosanone	**9a**	Taher, M., *et al*., 2007
		Cambogin	**2e**	
		epicatechin	**7j**	
		osajaxanthone	**12.2r**	
		rubraxanthone	**12.3ee**	
		isocowanol	**12.3nn**	
G. gardneriana	leaves	Fukugetin	**4a**	Castardo, J. C., *et al*., 2008
		GB-2a	**4f**	
G. hanburyi	resin and fruits	7-methoxydes-oxymorellin	**12.6q**	Reutrakul, V., *et al*., 2007
		2-isoprenylforbe-sione	**12.6hh**	
		8,8a-epoxymore-llic acid	**12.6x**	

Table 1 (continued)

Scientific name	Investigated parts	Compounds	Structures	References
		desoxymorellin	**12.6o**	
		dihydroisomore-llin	**12.6nn**	
		isomorellin	**12.6j**	
		Gambogic acid	**12.6z**	
		morellic acid	**12.6r**	
		isomorellinol	**12.6l**	
		desoxygamboge-nin	**12.6ee**	
		Hanburin	**12.6gg**	
		forbesione	**12.6jj**	
		Moreollic acid	**12.6oo**	
	dry latex	isogambogenic acid	**12.6cc**	Feng, F., *et al*., 2007
		desoxymorellin	**12.6o**	
		10-methoxy gambogenic acid	**12.6dd**	
		10-methoxy gambogic acid	**12.6g**	
		10-ethoxy gambogic acid	**12.6h**	
		desoxygambo-genin	**12.6ee**	
		Gambogic acid	**12.6z**	
		morellic acid	**12.6r**	

Table 1 (continued)

Scientific name	Investigated parts	Compounds	Structures	References
		2-isoprenylforbe-sone	**12.6hh**	
		Gambogin	**12.6f**	
		Moreollic acid	**12.6oo**	
		gambogellic acid	**12.6w**	
		hanburin	**12.6gg**	
		gambogenic acid	**12.6ff**	
	gamboges	30-hydroxy gambogic acid	**12.6n**	Han, Q.-B., *et al*., 2006b
		epigambogic acid	**12.6v**	
	resin	Gambogic acid	**12.6z**	Han, Q.-B., *et al*., 2006a
		desoxymorellin	**12.6o**	
		isomorellic acid	**12.6k**	
		morellic acid	**12.6r**	
		isogambogic acid	**12.6m**	
		isomorellinol	**12.6l**	
		gambogenic acid	**12.6ff**	
		gambogoic acid A	**12.6kk**	
		gambogoic acid B	**12.6ll**	
		gaudichaudic acid	**12.6aa**	
		isogambogenic acid	**12.6cc**	
		deoxygaudichau-dione A	**12.6bb**	

Table 1 (continued)

Scientific name	Investigated parts	Compounds	Structures	References
G. indica	fruit rinds	Garcinol	**2d**	Huang, M.-T., *et al*., 2008
	fruit rinds	xanthochymol	**2b**	Kumar, S., *et al*., 2007a,b
		isoxanthochymol	**2a**	
G. kola	seeds	naringin-7-rharm-noglucoseside	**8a**	Okwu, D. E., *et al*., 2007
G. lancilimba	stem barks	1,5,6-trihydroxy-6',6'-dimethyl-2H-pyrano(2',3':3,4)-2-(3-methylbut-2-enyl)xanthone	**12.3iii**	Yang, N. Y., *et al*., 2007
		1,6,7-trihydroxy-6',6'-dimethyl-2H-pyrano(2',3':3,2)-4-(3-methylbut-2-enyl)xanthone	**12.3ooo**	
		6-deoxyjacareubin	**12.2p**	
		Xanthone V1	**12.3ppp**	
		Xanthone V1a	**12.3o**	
		dulxanthone B	**12.3p**	
		cudratricusxan-thone E	**12.3q**	
		parvifolixan-thone B	**12.3ff**	
G. linii	roots	(*S*)-3-hydroxygar-cibenzopyran	**5e**	Chen, J.-J., *et al*., 2006

Table 1 (continued)

Scientific name	Investigated parts	Compounds	Structures	References
		garcibiphenyl C garcibiphenyl D garcibiphenyl E	**5a** **5b** **5f**	
G. livingstonei	root barks	*ent*-naringeninyl-(I-3α,II-8)-4'-*O*-methylnaringenin (+)-morellofla-vone (+)-volkensifla-vone 6,11-dihydroxy-3-methyl-3-(4-methyl-3-pent-enyl)xanthone 4-(3',7'-dimethyl-octa-2',6'-dienyl)-1,3,5-trihydroxy-9H-xanthen-9-one Garcilivin A 1,4,5-trihydroxy-3-(3-methyl-2-butenyl)xanthone Garcilivin C	**4c** **4a** **4b** **12.2w** **12.2u** **12.8a** **12.2d** **12.8b**	Mbwambo, Z., *et al*., 2006
G. lucida	stem barks	dihydrochelery-thrine 6-acetonyldihy-drochelerythrine	**1a** **1b**	Fotie, J., *et al*., 2007

Table 1 (continued)

Scientific name	Investigated parts	Compounds	Structures	References
		lucidamine A	**1c**	
		lucidamine B	**1d**	
G. maingayii	stem barks	isoxanthochymol	**2a**	Hartati, S., *et al*., 2007
		camboginol	**2d**	
		stigmasterol	**10b**	
		5,7,2',5'-tetrahy-droxyflavan-3-ol	**7k**	
		griffipavixanthone	**12.8c**	
G. mangostana	fruits	1,2-dihydro-1,8,-10-trihydroxy-2-(2-hydroxypro-pan-2-yl)-9-(3-me-thylbut-2-enyl)-furo[3,2-a]xan-then-11-one	**12.2x**	Chin, Y.-W., *et al*., 2008b
		6-deoxy-7-deme-thylmangostanin	**12.2y**	
		1,3,7-trihydroxy-2,8-di-(3-methyl-but-2-enyl)xan-thone	**12.2n**	
		mangostanin	**12.3www**	
		α-mangostin	**12.3a**	
	pericarps	α-mangostin	**12.3a**	Balunas, M. J., *et al*., 2008

Table 1 (continued)

Scientific name	Investigated parts	Compounds	Structures	References
		1-isomangostin	**12.3eeee**	
		γ-mangostin	**12.3g**	
		8-deoxygartanin	**12.2m**	
		Gartanin	**12.3u**	
		tovophyllin A	**12.3kkk**	
		Garcinone D	**12.3x**	
		mangostinone	**12.2v**	
		Garcinone E	**12.3h**	
		cudraxanthone G	**12.2i**	
		smeathxanthone A	**12.3s**	
		8-hydroxy cudraxanthone G	**12.3t**	
	fruit hulls	γ-mangostin	**12.3g**	Yu, L., *et al*., 2007
		α-mangosin	**12.3a**	
		epicatechin	**7j**	
	pericarps	8-hydroxycudra-xanthone G	**12.3t**	Jung, H.-A., *et al*., 2006
		mangostingone	**12.3aaa**	
		cudraxanthone G	**12.2i**	
		8-deoxygartanin	**12.2m**	
		garcimangosone B	**12.3bbbb**	
		Garcinone D	**12.3x**	
		Garcinone E	**12.3h**	
		Gartanin	**12.3u**	
		1-isomangostin	**12.3eeee**	

Table 1 (continued)

Scientific name	Investigated parts	Compounds	Structures	References
	young fruits	α-mangostin	**12.3a**	Suksamrarn, S., *et al*., 2006
		γ-mangostin	**12.3g**	
		smeathxanthone A	**12.3s**	
		mangostinone	**12.2v**	
		tovophyllin A	**12.3kkk**	
		mangostenone C	**12.3xxx**	
		mangostenone D	**12.3vvv**	
		mangostenone E	**12.3aa**	
		Garcinone C	**12.3bb**	
		Garcinone B	**12.3cccc**	
		demethylcalaba-xanthone	**12.2q**	
		Garcinone D	**12.3x**	
		Garcinone E	**12.3h**	
		11-hydroxy-1-isomangostin	**12.3dddd**	
		mangostinone	**12.2v**	
		mangostanol	**12.3lll**	
		mangostanin	**12.3www**	
		thawaitesixanthone	**12.2aa**	
		8-deoxygartanin	**12.2m**	
		Gartanin	**12.3u**	
		α-mangostin	**12.3a**	
		β-mangostin	**12.3b**	
		γ-mangostin	**12.3g**	

Table 1 (continued)

Scientific name	Investigated parts	Compounds	Structures	References
	stems	Garcinone D	**12.3x**	Ee, G. C. L., *et al*., 2006
		mangosharin	**12.2b**	
		1,6-dihydroxy-3,7-dimethoxy-2-(3-methylbut-2-enyl)xanthone	**12.3v**	
		α-mangostin	**12.3a**	
		β-mangostin	**12.3b**	
		mangostanol	**12.3lll**	
		5,9-dihydroxy-8-methoxy-2,2-dimethyl-7-(3-methylbut-2-enyl)-2H,6H-pyrano-[3,2-b]xanthene-6-one	**12.3ccc**	
	fruit hulls	α-mangostin	**12.3a**	Hu, J., *et al*., 2006
		β-mangostin	**12.3b**	
		γ-mangostin	**12.3g**	
		5,9-dihydroxy-8-methoxy-2,2-dimethyl-7-(3-methylbut-2-enyl)-2H,6H-pyrano-[3,2-b]xanthen-6-one	**12.3ccc**	

Table 1 (continued)

Scientific name	Investigated parts	Compounds	Structures	References
		epicatechin Egonol	**7j** **13m**	
G. merguensis	woods	3,3',4-*O*-trime-thylellagic acid α-mangostin rubraxanthone isocowanol	**13i** **12.3a** **12.3ee** **12.3nn**	Kijjao, A., *et al*., 2008
G. morella	seed coat	morellic acid isomorellic acid Gambogic acid morellin Guttiferic acid 2-methyl-4-[(1*R*,-3a*S*,5*S*,14a*S*)-3a,-4,5,7-tetrahydro-8-hydroxy-3,3,-11,11-tetrame-thyl-13-(3-methyl-2-buten-1-yl)-7,15-dioxo-1,5-methano-1H,3H,-11H-furo[3,4-g]-pyra-no-[3,2-b]-xan-then-1-yl]me-thyl ester	**12.6r** **12.6k** **12.6u** **12.6i** **12.7b** **12.6s**	Rao, D. R., *et al*., 2007

Table 1 (continued)

Scientific name	Investigated parts	Compounds	Structures	References
		2-methyl-4-[(1*R*,-3a*S*,5*S*,14a*S*)-3a,-4,5,7-tetrahydro-8-methoxy-3,3,-11,11-tetramethyl-13-(3-methyl-2-buten-1-yl)-7,15-dioxo-1,5-metha-no-1H,3H,11H-furo[3,4-g]pyra-no[3,2-b]xanthen-1-yl]methyl ether 3a,4,5,7-tetrahy-dro-8-hydroxy-1-[(2*Z*)-4-methoxy-3-methyl-4-oxo-2-buten-1-yl]-3,-3,11,11-tetrame-thyl- 13-(3-me-thyl-2-buten-1-yl)-7-oxoxanthone methyl ester	**12.6t** **12.7a**	
G. multiflora	roots	garcinialone isoxanthochymol	**12.6y** **2a**	Chein, S.-C., *et al*., 2008
G. oblongifolia	stems and leaves	oblongifoliagar-cinine A	**5c**	Wu, X., *et al*., 2008b

Table 1 (continued)

Scientific name	Investigated parts	Compounds	Structures	References
		oblongifoliagar-cinine B	**5d**	
		oblongifoliagar-cinine C	**5g**	
		oblongifoliagar-cinine D	**5h**	
	bark	oblongifolin A	**2h**	Hamed, W., *et al*., 2006
		oblongifolin B	**2j**	
		oblongifolin C	**2k**	
		oblongifolin D	**2i**	
		camboginol	**2d**	
		guttiferone B	**2v**	
G. parvifolia	leaves	parvifoliol B	**13e**	Rukachaisiri-kul, V., *et al*., 2008
		parvifoliol C	**3a**	
		parvifoliol E	**3c**	
		garcidepsidone B	**6b**	
		nigrolineaisofla-vone A	**7i**	
		mangostinone	**12.2v**	
		parvifoliquinone	**13k**	
	roots	parvixanthone A	**12.3z**	Kardono, L. B. S., *et al*., 2006
		rubraxanthone	**12.3ee**	
	twigs	parvifoliol A	**13d**	Rukachaisiri-kul, V., *et al*., 2006

Table 1 (continued)

Scientific name	Investigated parts	Compounds	Structures	References
		parvifoliol B	**13e**	
		parvifoliol C	**3a**	
		parvifoliol D	**3b**	
		parvifoliol E	**3c**	
		parvifoliol F	**3d**	
		parvifoliol G	**3e**	
		parvifolidone A	**6a**	
		parvifolidone B	**6d**	
		parvifolixanthone A	**12.3n**	
		parvifolixanthone B	**12.3ff**	
		parvifolixanthone C	**12.3mm**	
		garcidepsidone B	**6b**	
		mangostinone	**12.2v**	
		rubraxanthone	**12.3ee**	
		dulxanthone D	**12.3dd**	
		(2*E*,6*E*,10*E*)-(+)-4β-hydroxy-3-methyl-5β-(3,7,11,15 tetramethylhexadeca-2,6,10,14-tetraenyl)cyclohex-2-en-1-one	**13l**	

Table 1 (continued)

Scientific name	Investigated parts	Compounds	Structures	References
		1,3,5,6-tetrahy-droxyxanthone	**12.3tt**	
		norathyriol	**12.3cc**	
G. penangiana	leaves	4-(1,1-dimethyl-prop-2-enyl)-1,3,-5,8-tetrahydroxy-xanthone	**12.3uu**	Jabit, M. L., *et al*., 2007
		penangianaxan-thone	**12.3gggg**	
		cudratricusxan-thone H	**12.3uuu**	
		macluraxan-thone C	**12.3oo**	
		gerontoxan-thone C	**12.3zzz**	
G. picrorrhiza	barks	garcinopicoben-zophenone	**2o**	Soemiati, A., *et al*., 2006
		guttiferone F	**2w**	
G. polyantha	wood trunks	polyanxanthone A	**12.2e**	Louh, G. N., *et al*., 2008
		polyanxanthone B	**12.1b**	
		polyanxanthone C	**12.1c**	
		1,3,5-trihydroxy-xanthone	**12.2f**	
		1,5-dihydroxy-xanthone	**12.1d**	
		norathyriol	**12.3cc**	

Table 1 (continued)

Scientific name	Investigated parts	Compounds	Structures	References
		1,6-dihydroxy-5-methoxyxanthone	**12.2o**	
		1,3,5,6-tetrahy-droxyxanthone	**12.3tt**	
	stem bark	3-(3,7-dimethyl-2,6-octadien-1-yl)-1,4,8-trihy-droxyxanthone	**12.2g**	Komguem, J., *et al*., 2006
		Oleanolic acid	**11e**	
		1,5-dimethoxy-xanthone	**12.1a**	
		guttiferone G	**2g**	
		smeathxanthone A	**12.3s**	
		bangangxan-thone A	**12.3fff**	
		bangangxan-thone B	**12.2h**	
		2-hydroxy-1,7-di-methoxyxanthone	**12.2a**	
G. porrecta	stem barks	porxanthone A	**12.3mmm**	Kardono, L. B. S., *et al*., 2006
		porlanosterol	**11g**	
		dulxanthone E	**12.4d**	
		dulxanthone F	**12.4e**	
		dulxanthone G	**12.5a**	
G. rigida	leaves	yahyaxanthone	**12.5b**	Elya, B., *et al*., 2008

Table 1 (continued)

Scientific name	Investigated parts	Compounds	Structures	References
	leaves	musaxanthone asmaxanthone	**12.4c** **12.3ggg**	Elya, B., *et al*., 2006a
G.smeathmahnii	stem barks	bangangxan-thone A guttiferone I cheffouxanthone triacontanylcaf-feate smeathxanthone B smeathxanthone A isoxanthochymol 1,5-dihydroxy-xanthone 1,3,5-trihydroxy-xanthone Friedelin	**12.3fff** **2p** **12.3r** **13c** **12.3bbb** **12.3s** **2a** **12.1d** **12.2f** **11b**	Kuete, V., *et al*., 2007
	root barks	cheffouxanthone guttiferone I isoxanthochymol smeathxanthone A smeathxanthone B triacontanylcaf-feate	**12.3r** **2p** **2a** **12.3s** **12.3bbb** **13c**	Lannang, A. M., *et al*., 2006
G. subelliptica	heartwoods and pericarps	garcinielliptone HF	**9h**	Wu, C.-C., *et al*., 2008a

Table 1 (continued)

Scientific name	Investigated parts	Compounds	Structures	References
		garcinielliptone FC	**2l**	
		garcinielliptone FC tautomer	**2m**	
	green and ripened fruits	(+)-4'''-*O*-methyl-fukugetin	**4d**	Terashima, K., *et al*., 2008
	heartwoods	garcinielliptone HA	**9c**	Lu, Y.-H., *et al*., 2008
		garcinielliptone HB	**9e**	
		garcinielliptone HC	**9f**	
		garcinielliptone HD	**9g**	
		garcinielliptone HE	**9d**	
	fresh fruits	garcinielliptone F	**9b**	Lin, C.-N., *et al*., 2006
		garcinielliptone I	**2y**	
G. tetrandra	stem barks	1,3-dihydroxy-2',2'-dimethyl-pyrano(5',6',5,6)-xanthone	**12.2bb**	Hartati, S., *et al*., 2008b
		cudraxanthone	**12.2z**	
		Lupeol	**11a**	

Table 1 (continued)

Scientific name	Investigated parts	Compounds	Structures	References
		stigmasterol thawaitesixanthone 3-α-hopenol Cambogin camboginol	**10b** **12.2aa** **11f** **2e** **2d**	
G. urophylla	leaves	7-hydroxy-desoxymorellin isocaledonixan-thone D gaudichaudione H 1,7-dihydroxy-3-methoxy-2-(3-methyl-2-buten-yl)xanthone 1,5-dihydroxy-3-methoxy-2-(3-methyl-2-bu-tenyl)xanthone 1,3,7-trihydroxy-2-(3-methyl-2-bu-tenyl)xanthone lupeol	**12.6p** **12.3zz** **12.6ii** **12.2j** **12.2k** **12.2l** **11a**	Mohd Khalid, R., *et al*., 2007
G. vieillardii	stem barks	vieillardiixan-thone B vieillardiixan-thone C	**12.3ww** **12.3xx**	Hay, A.-E., *et al*., 2008

Table 1 (continued)

Scientific name	Investigated parts	Compounds	Structures	References
		pancixanthone A pancixanthone B 1,6-dihydroxy-xanthone pyranojacareubin 5,6-*O*-dimethyl-2-deprenylrhee-diaxanthone clusiachromene C 3-geranyl-2,4,6-trihydroxybenzo-phenone	**12.2s** **12.2t** **12.1e** **12.3hhhh** **12.3iiii** **2s** **2bb**	
G. virgata	stem barks	guttiferone I guttiferone J xanthochymol guttiferone E	**2p** **2q** **2b** **2c**	Merza, J., *et al*., 2006
G. xanthochy-mus	twig barks	1,4,5,6-tetrahy-droxy-7,8-dipre-nylxanthone 1,3,5,6-tetrahy-droxy-4,7,8-tri-prenylxanthone garciniaxanthone E 1,4,6-trihydroxy-5-methoxy-7-(3-methyl-2-buten-1-yl)xanthone	**12.3ll** **12.3vv** **12.3jj** **12.3qq**	Han, Q.-B., *et al*., 2007

Table 1 (continued)

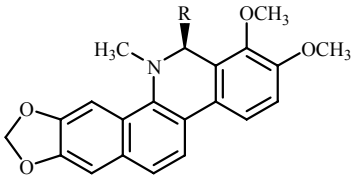
Scientific name	Investigated parts	Compounds	Structures	References
		1,4,5,6-tetrahy-droxy-7-(3-methyl-2-buten-1-yl)-xanthone	**12.3rr**	
		7-(3,7-dimethyl-2,6-octadien-1-yl)-1,2,5,6-tetrahy-droxyxanthone	**12.3yy**	
		6-prenylapigenin	**7f**	
		1,4,5,6-tetrahy-droxy-7,8-dipre-nylxanthone	**12.3ll**	
	barks	1,6-dihydroxy-4,5-dimethoxyxan-thone	**12.3ss**	Zhong, F. F., *et al*., 2007
		1,5,6-trihydroxy-7,8-di-(3-methyl-2-butenyl)-6',6'-di-methylpyrano(2',-3':3,4)xanthone	**12.3hhh**	
G. xipshuan-bannaensis	twigs	bannaxanthone A	**12.3i**	Han, Q.-B., *et al*., 2008
		bannaxanthone B	**12.3j**	
		bannaxanthone C	**12.3k**	
		bannaxanthone D	**12.3qqq**	
		bannaxanthone E	**12.3mmmm**	

Table 1 (continued)

Scientific name	Investigated parts	Compounds	Structures	References
		bannaxanthone F	**12.3nnnnn**	
		bannaxanthone G	**12.3rrr**	
		bannaxanthone H	**12.3kkkk**	
		γ-mangostin	**12.3g**	
		isojacareubin	**12.3jjjj**	
		xanthochymol	**2b**	
		Garcinone C	**12.3bb**	
		Garcinone E	**12.3h**	
		guttiferone E	**2c**	
		allanxanthone C	**12.3llll**	
	fruits	tovophyllin B	**12.3jjj**	Shen, J., *et al*.,
		β-sitosterol	**10a**	2006b
		ursolic acid	**11d**	
		1-stearyl alcohol	**13a**	
		isogarcinol	**2e**	
		Luteolin	**7d**	
		3',5,7-trihydro-xy-4'-methoxy-flavone	**7c**	
		luteolin-7-*O*-glu-curonic acid Me ester	**8f**	
		daucosterol	**10c**	
		Vitexin	**8e**	

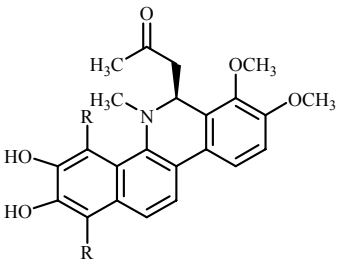
Structures of compounds isolated from plants of the genus *Garcinia*

1. Alkaloids

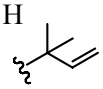


1a : R = H dihydrochelerythrine

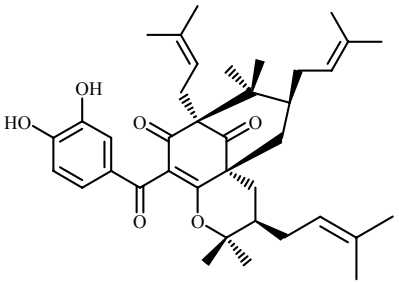
1b : R = CH_2COMe 6-acetonyldihydrochelerythrine



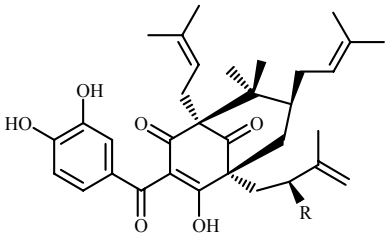
1c : R = H lucidamine A

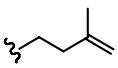
1d : R =  lucidamine B

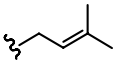
2. Benzophenones

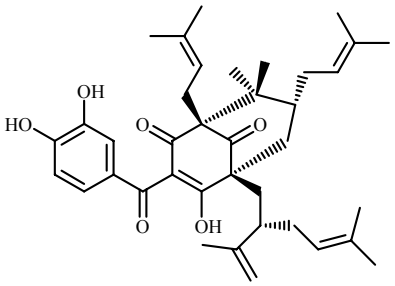


2a : isoxanthochymol

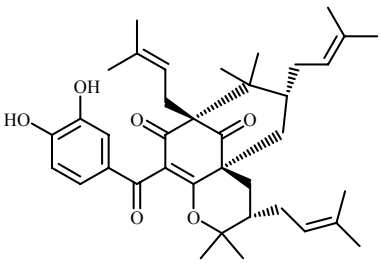


2b : R =  xanthochymol

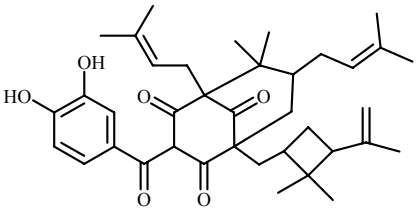
2c : R =  guttiferone E



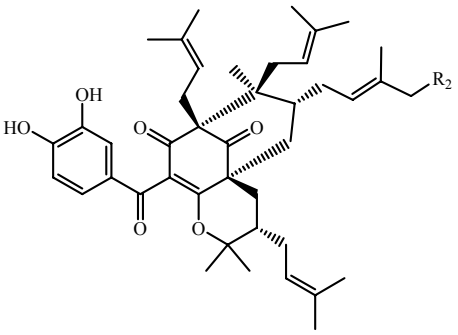
2d : camboginol (garcinol)



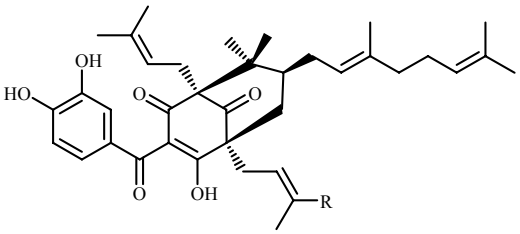
2e : cambogin (isogarcinol)



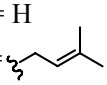
2f : eugeniaphenone

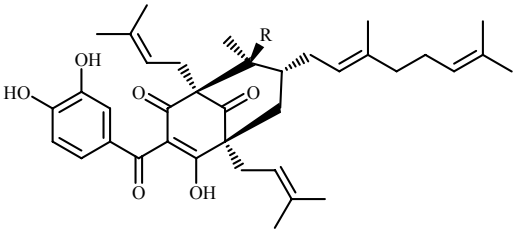


2g : guttiferone G

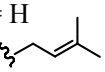


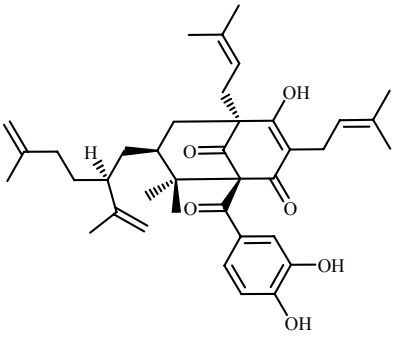
2h : R = H oblongifolin A

2i : R =  oblongifolin D

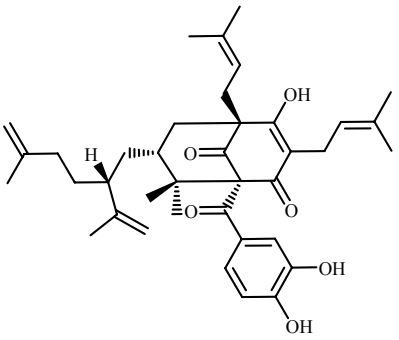


2j : R = H oblongifolin B

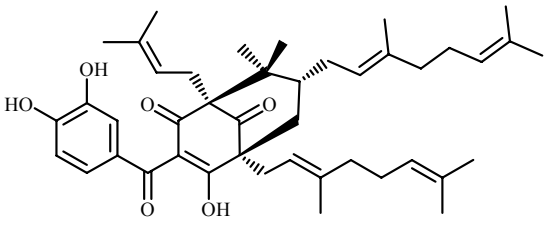
2k : R =  oblongifolin C



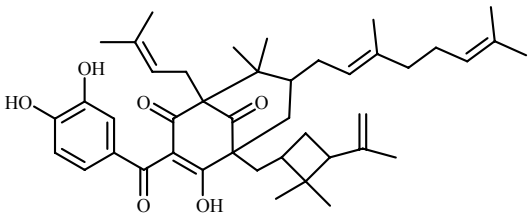
2l : garcinielliptone FC



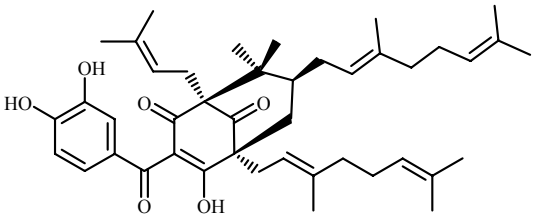
2m : garcinielliptone FC tautomer



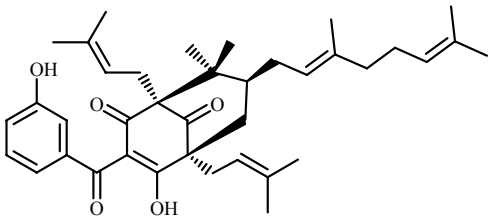
2n : guttiferone M



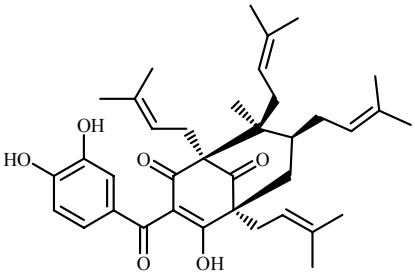
2o : garcinopirobenzophenone



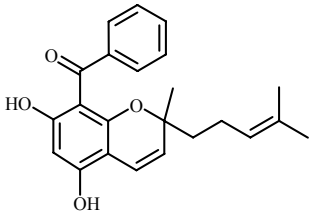
2p : guttiferone I



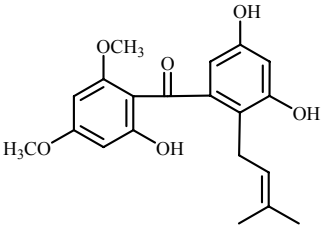
2q : guttiferone J



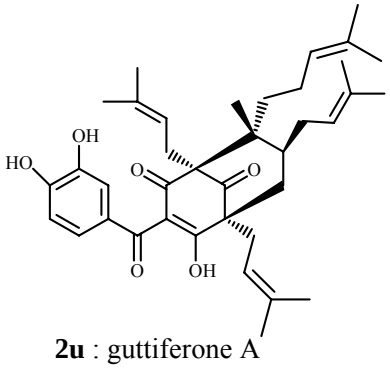
2r : guttiferone K



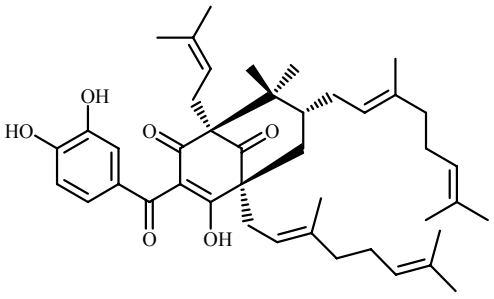
2s : clusiachromene C



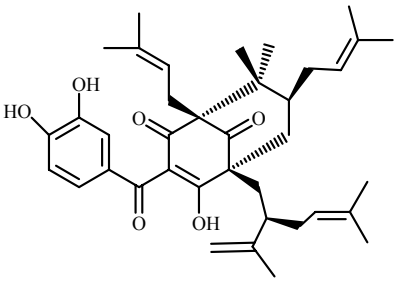
2t : salimbenzophenone



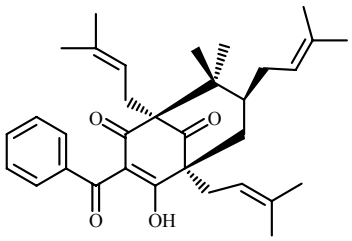
2u : guttiferone A



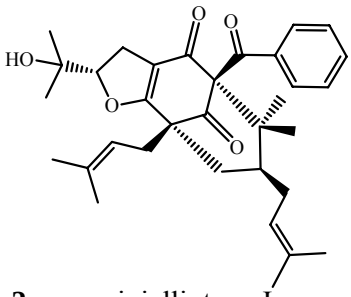
2v : guttiferone B



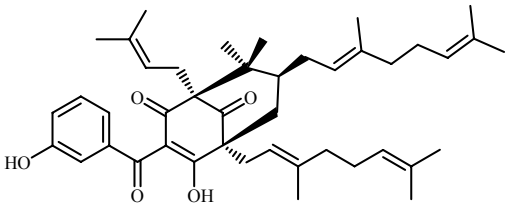
2w : guttiferone F



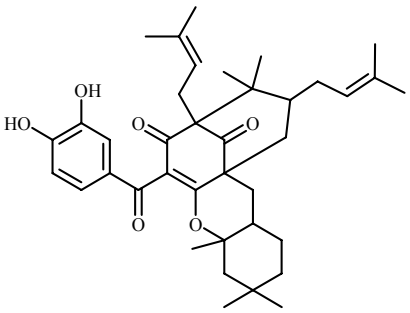
2x : 7-epiclusianone



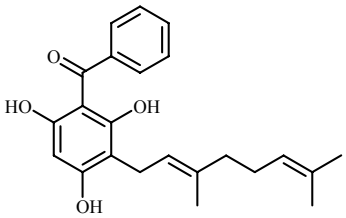
2y : garcinielliptone I



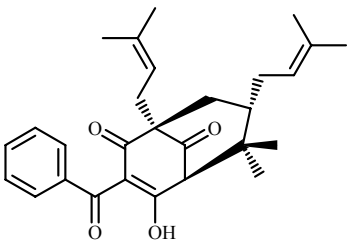
2z : guttiferone N



2aa : (+)-6-(3,4-dihy-droxybenzoyl)-2, 3,4,4a, 8,9,10,11, 12,12a-decahydro- 3,3,4a,9,9-penta-methyl-8,10-bis(3- methyl-8,10-bis(3- methyl-2-buten-1-yl)-1H-8,11a-methano-7H-benzo [b]cycloocta[e]py ran-7-13-dione

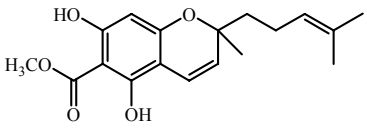


2bb : 3-geranyl-2,4,6-trihydroxybenzophenone

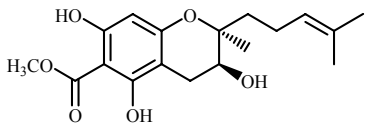


2cc : garciniaphenone

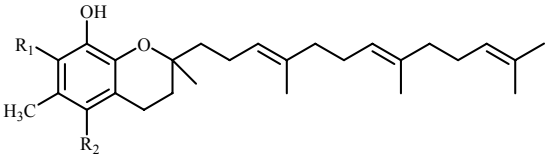
3. Benzopyrans



3a : parvifoliol C

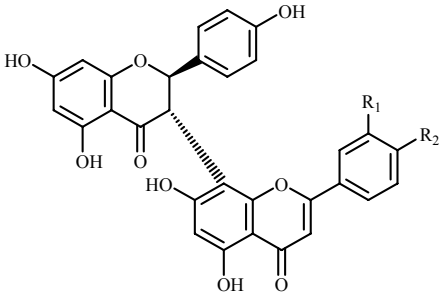


3b : parvifloliol D

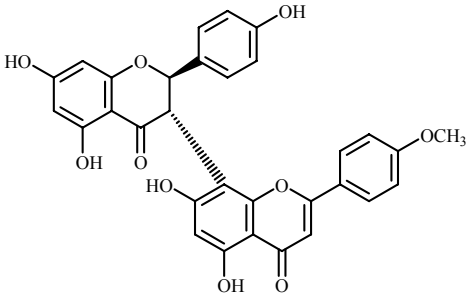


3c : $R_1 = CH_3$, $R_2 = H$ parvifoliol E
3d : $R_1 = H$, $R_2 = H$ parvifoliol F
3e : $R_1 = OH$, $R_2 = CH_3$ parvifoliol G

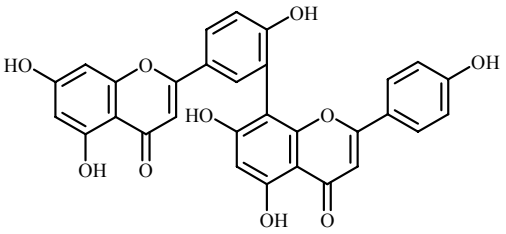
4. Biflavones



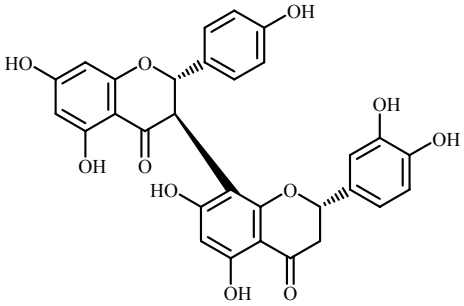
4a : $R_1 = OH$, $R_2 = OH$ (+)-morelloflavone (fukugetin)
4b : $R_1 = H$, $R_2 = OH$ (+)-volkensiflavone
4c : $R_1 = H$, $R_2 = OCH_3$ *ent*-narngeninyl-(I-3α-II-8)-4'-*O*-methylnaringenin



4d : (+)-4'''-*O*-methylfukugetin

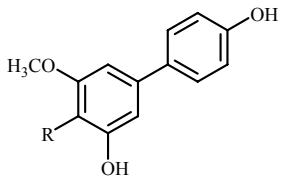


4e : amentoflavone

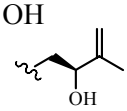


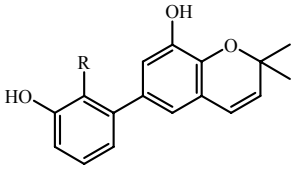
4f : GB-2a

5. Biphenyls

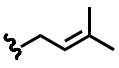


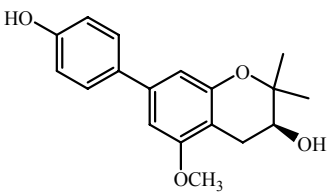
5a : R = OH garcibiphenyl C

5b : R =  garcibiphenyl D

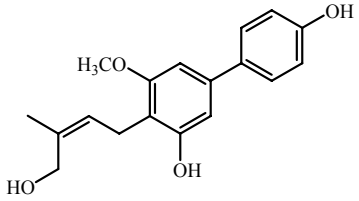


5c : R = H oblongfoliagarcinine A

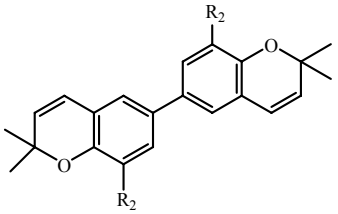
5d : R =  oblongfoliagarcinine B



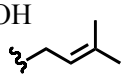
5e : (*S*)-3-hydroxygarcibenzopyran



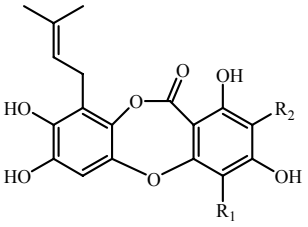
5f : garcibiphenyl E

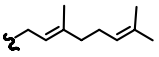


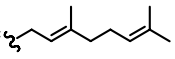
5g : R_1 = H, R_2 = OH oblongfoliagarcinine C

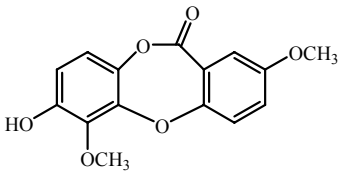
5h : R_1 = OH, R =  oblongfoliagarcinine D

6. Despidones

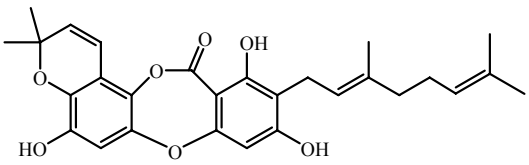


6a : R_1 =  , R_2 = H parvifolidone A

6b : R_1 = H, R_2 =  garcidepidone B

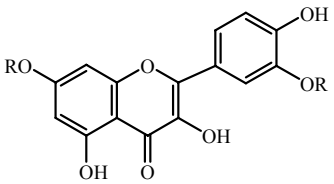


6c : brevipsidone



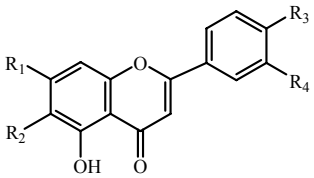
6d : parifolidone B

7. Flavonoids



7a : R = H rhamnazin

7b : R = CH_3 quercetin

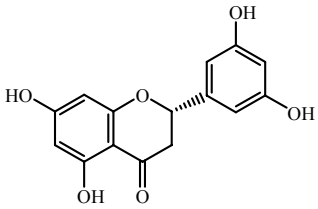


7c : R_1 = OH, R_2 = H, R_3 = OCH_3, R_4 = H 3',5,7-trihydroxy-4'-methoxyflavone

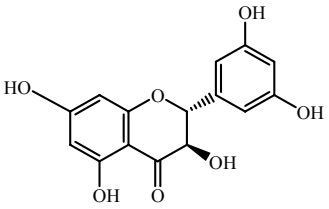
7d : R_1 = OH, R_2 = H, R_3 = OH, R_4 = OH luteotin

7e : R_1 = OCH_3, R_2 = H, R_3 = OCH_3, R_4 = OH pilloin

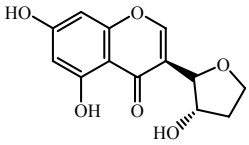
7f : R_1 = OH, R_2 =  , R_3 = OH, R_4 = H 6-prenylapigenin



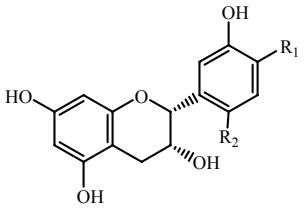
7g : 2-(3,5-dihydroxyphenyl)-2,3-dihydro-5,7-dihydroxyflavone



7h : 2-(3,5-dihydroxyphenyl)-2,3-dihydro-3,5,7-trihydroxyflavone

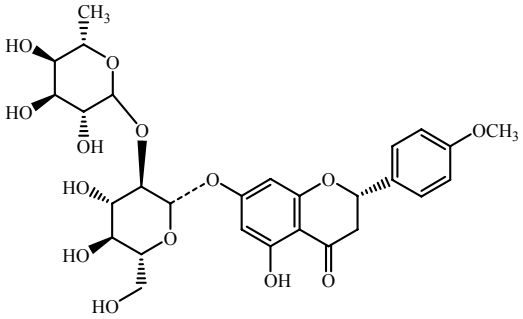


7i : nigrolineaisoflavone A

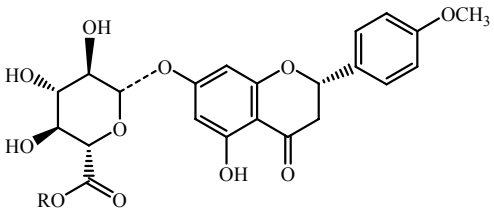


7j : R_1 = OH, R_2 = H epicatechin
7k : R_1 = H, R_2 = OH 5,7,2',5'-tetrahydroxy-flavan-3-ol

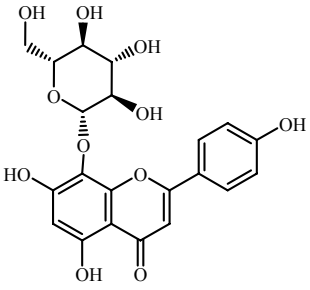
8. Flavone glycosides



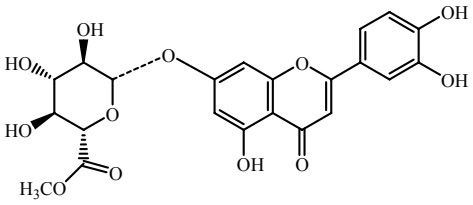
8a : naringin-7-rharmnoglucoseside



8b : R = OCH_2CH_3 garccowaside A
8c : R = *O*-*n*-Bu garccowaside B
8d : R = OCH_3 garccowaside C

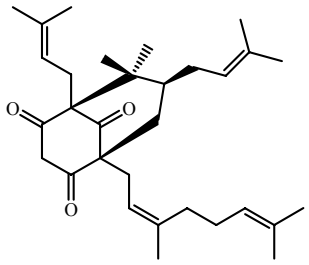


8e : vitexin

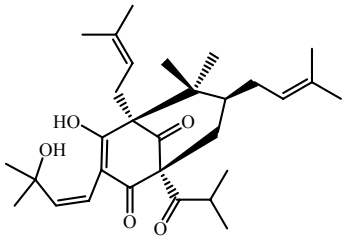


8f : luteolin-7-*O*-glucuronic acid Me ester

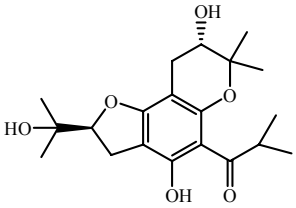
9. Phloroglucinols



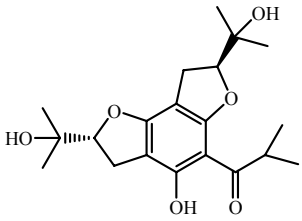
9a : enervosanone



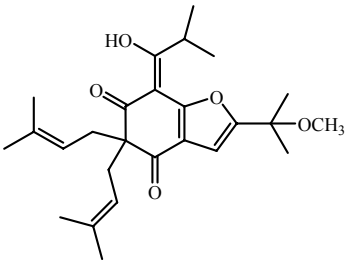
9b : garcinielliptone F



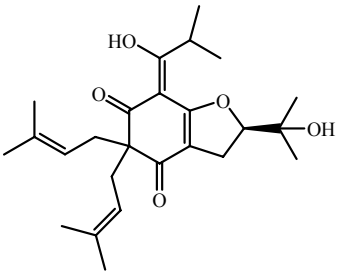
9c : garcinielliptone HA



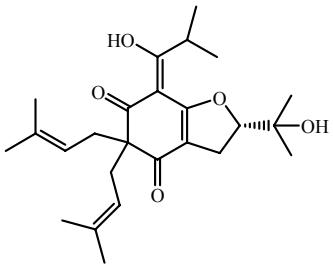
9d : garcinielliptone HE



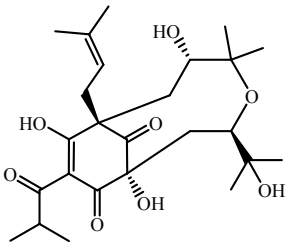
9e : garcinielliptone HB



9f : garcinielliptone HC

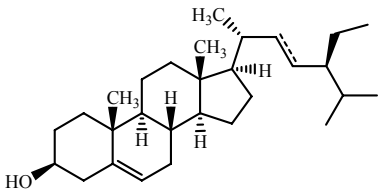


9g : garcinielliptone HD



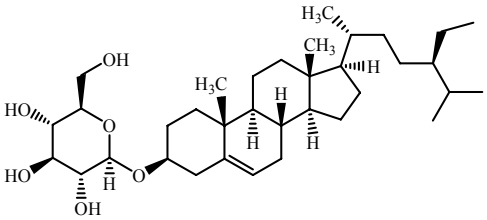
9h : garcinielliptone HF

10. Steroids



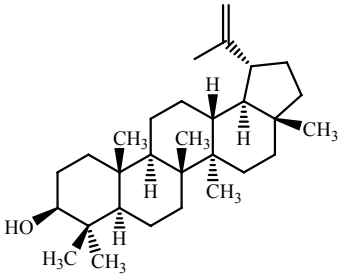
10a : single bond *β*-sitosterol

10b : double bond stigmasterol

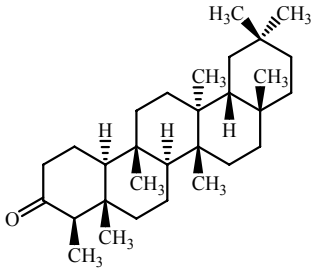


10c : daucosterol

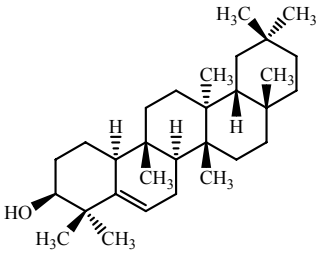
11. Triterpenes



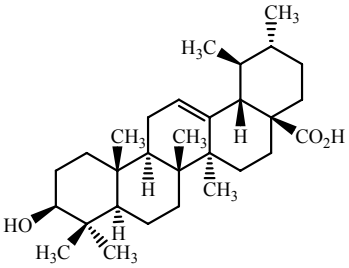
11a : lupeol



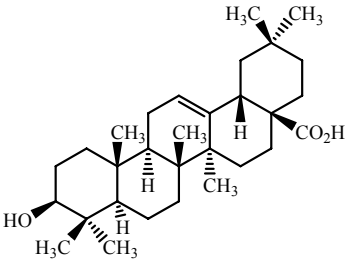
11b : friedelin



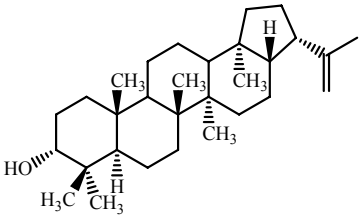
11c : glutin-5-en-3β-ol



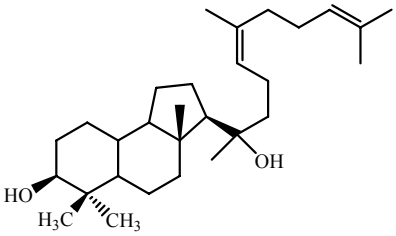
11d : ursolic acid



11e : oleanolic acid



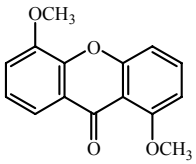
11f : 3-α-hopenol



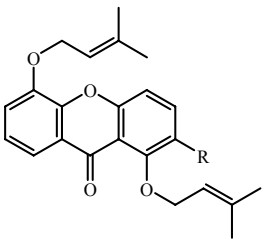
11g : porlanosterol

12. Xanthones

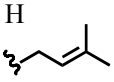
12.1 Dioxygenated xanthones

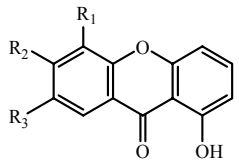


12.1a : 1,5-dimethoxyxanthone



12.1b : R = H polyanxanthone B

12.1c : R =  polyanxanthone C

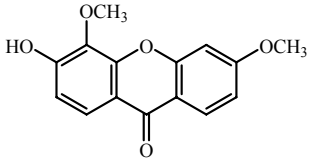


12.1d : R_1 = OH, R_2 = H, R_3 = H 1,5-dihydroxyxanthone

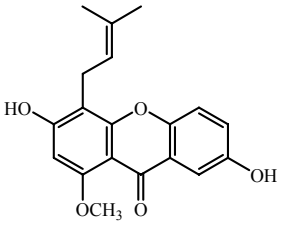
12.1e : R_1 = H, R_2 = OH, R_3 = H 1,6-dihydroxyxanthone

12.1f : R_1 = H, R_2 = H, R_3 = OH 1,7-dihydroxyxanthone

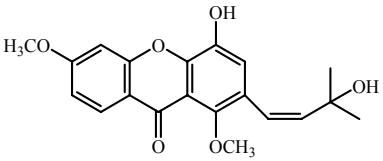
12.2 Trioxygenated xanthones



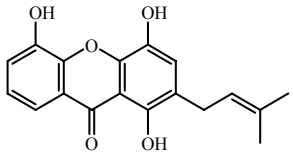
12.2a : 2-hydroxy-1,7-dimethoxyxanthone



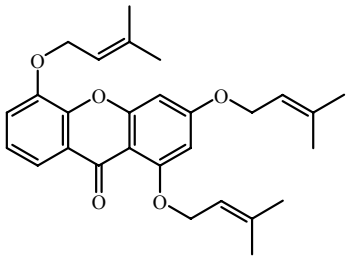
12.2b : mangosharin



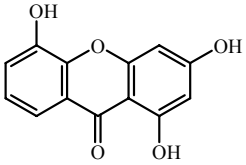
12.2c : afzeliixanthone B



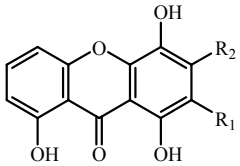
12.2d : 1,4,5-trihydroxy-3-(3-methyl-2-butenyl)xanthone

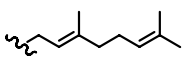


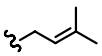
12.2e : polyanxanthone A

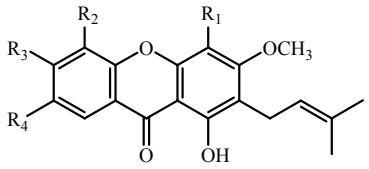


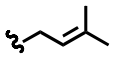
12.2f : 1,3,5-trihydroxyxanthone



12.2g : R_1 = H, R_2 =  3-(3,7-dimethyl-2,6-octadien-1-ly)-1,4,8-trihydroxyxanthone

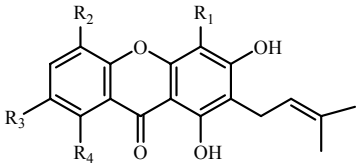
12.2h : R_1 =  , R_2 = H bangangxanthone B



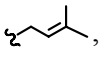
12.2i : R_1 =  , R_2 = OH, R_3 = H, R_4 = H cudraxanthone G

12.2j : R_1 = H , R_2 = H, R_3 = H, R_4 = OH 1,7-dihydroxy-3-methoxy-2-(3-methyl-2-butenyl)xanthone

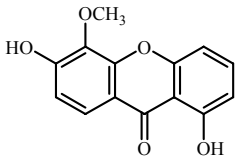
12.2k : R_1 = H , R_2 = OH, R_3 = H, R_4 = H 1,5-dihydroxy-3-methoxy-2-(3-methyl-2-butenyl)xanthone



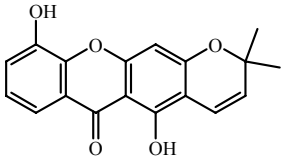
12.2l : R_1 = H, R_2 = H, R_3 = OH, R_4 = H 1,3,7-trihydroxy-2-(3-methylbut-2-enyl)xanthone

12.2m : R_1 =  , R_2 = OH, R_3 = H, R_4 = H 8-deoxygartanin

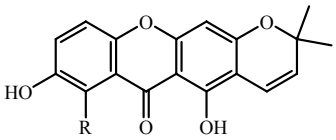
12.2n : R_1 = H, R_2 = H, R_3 = OH, R_4 =  1,3,7-trihydroxy-2,8-di-(3-methylbut-2-enyl)xanthone

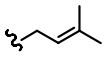


12.2o : 1,6-dihydroxy-5-methoxyxanthone

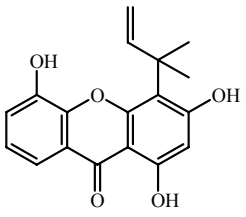


12.2p : 6-deoxyjacareubin

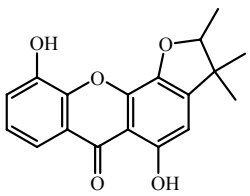


12.2q : R =  demethoxylcalabaxanthone

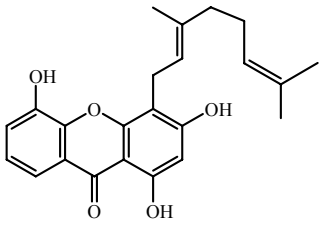
12.2r : R = H osajaxanthone



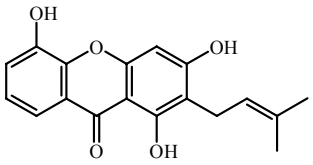
12.2s : pancixanthone A



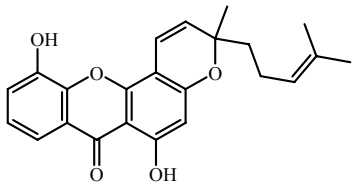
12.2t : pancixanthone B



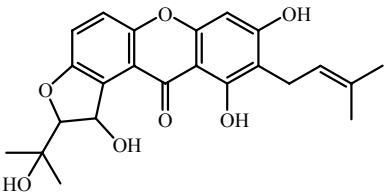
12.2u : 4-(3',7'-dimethylocta-2',6'-dienyl)-1,3,5-trihydroxy-9H-xanthen-9-one



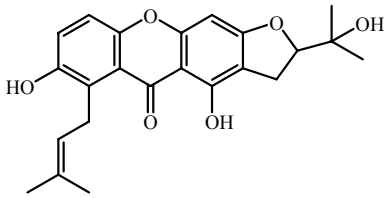
12.2v : mangostinone



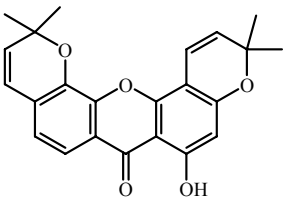
12.2w : 6,11-dihydroxy-3-methyl-3-(4-methyl-3-pentenyl)-xanthone



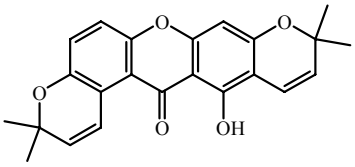
12.2x : 1,2-dihydro-1,8,10,-trihydroxy-2-(2-hydroxy-propan-2-ly)-9-(3-methylbut-2-enyl)furo-[3,2-a]xanthen-11-one



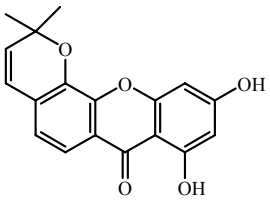
12.2y : 6-deoxy-7-demethylmanostanin



12.2z : cudraxanthone

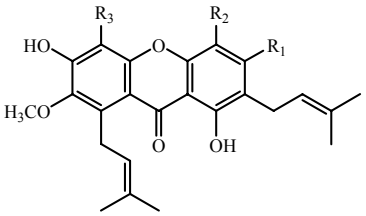


12.2aa : thawaitesixanthone

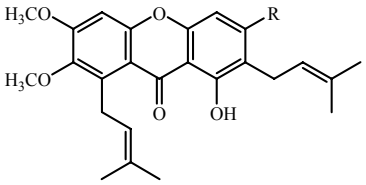


12.2bb : 1,3-dihydroxy-2',2'-dimethyl pyrano-(5',6',5,6)xanthone

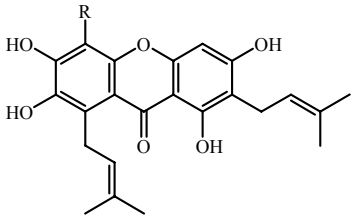
12.3 Tetraoxygenated xanthones



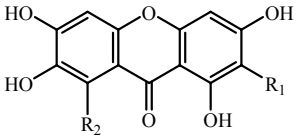
12.3a : R_1 = OH, R_2 = H, R_3 = H *α*-mangostin
12.3b : R_1 = OCH_3, R_2 = H, R_3 = H *β*-mangostin
12.3c : R_1 = OH, R_2 = CHO, R_3 = H cowaxanthone E
12.3d : R_1 = OH, R_2 = H, R_3 =  7-*O*-methyl garcinone E

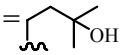
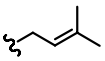


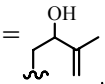
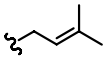
12.3e : R = OH cowaxanthone B
12.3f : R = OCH_3 fuscaxanthone C

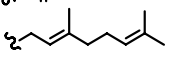


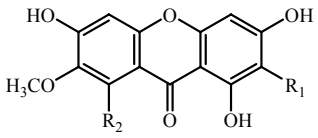
12.3g : R = H *γ*-mangostin
12.3h : R =  garcinone E

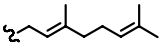


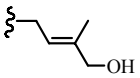
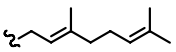
12.3i : R_1 =  , R_2 =  bannaxanthone A

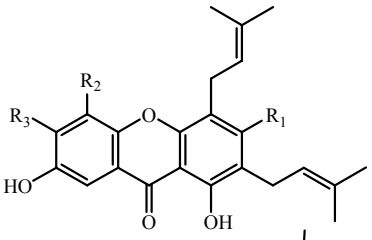
12.3j : R_1 =  , R_2 =  bannaxanthone B

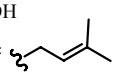
12.3k : R_1 =  , R_2 = H bannaxanthone C



12.3l : R_1 =  , R_2 = H cowaxanthone

12.3m : R_1 =  ,R_2 =  cowanol

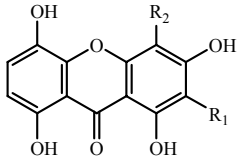


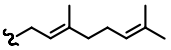
12.3n : R_1 = OH, R_2 = OH, R_3 =  parvifolixanthone A

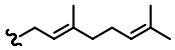
12.3o : R_1 = OH, R_2 = OH, R_3 = H xanthone V1a

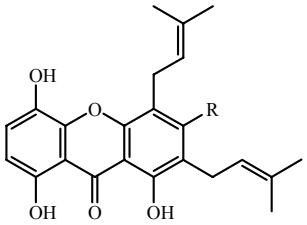
12.3p : R_1 = OCH_3, R_2 = OH, R_3 = H dulxanthone B

12.3q : R_1 = OH, R_2 = H, R_3 = OH cudratricusxanthone E



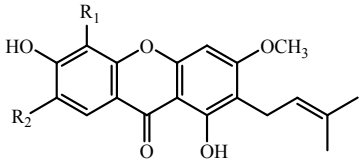
12.3r : R_1 = H, R_2 =  cheffouxanthone

12.3s : R_1 =  , R_2 = H smeathxanthone A



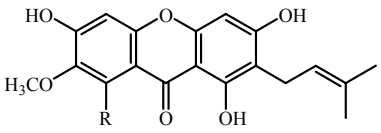
12.3t : R = OCH_3 8-hydroxycudraxanthone G

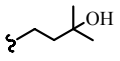
12.3u : R = OH gartanin

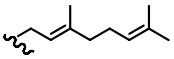


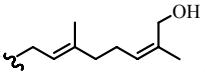
12.3v : R_1 = H, R_2 = OCH_3 1,6-dihydroxy-3,7-dimethoxy-2-(3-methyl-2-butenyl)xanthone

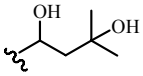
12.3w : R_1 = OCH_3, R_2 = H cowaxanthone A

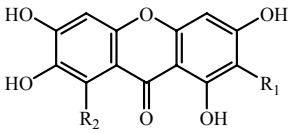


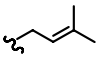
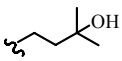
12.3x : R =  garcinone D

12.3y : R =  cowanin

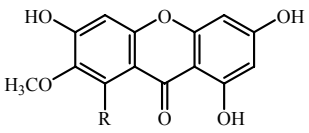
12.3z : R =  parvixanthone A

12.3aa : R =  mangostenone E

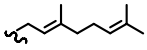


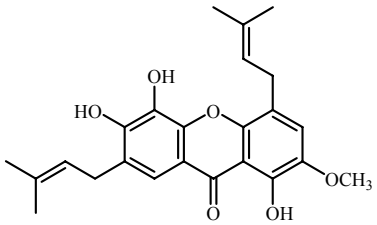
12.3bb : R_1 =  , R_2 =  garcinone C

12.3cc : R_1 = H , R_2 = H norathyriol

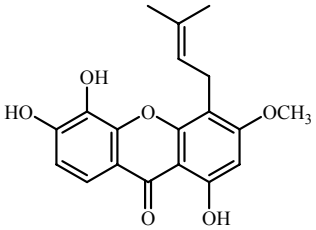


12.3dd : R =  dulxanthone D

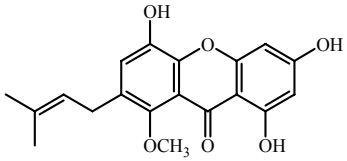
12.3ee : R =  rubraxanthone



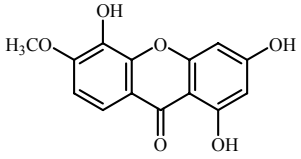
12.3ff : parvifolixanthone B



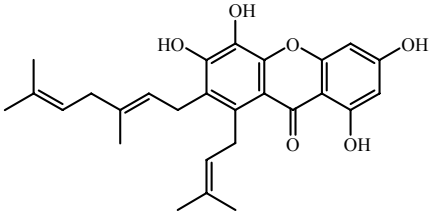
12.3gg : dulxathone A



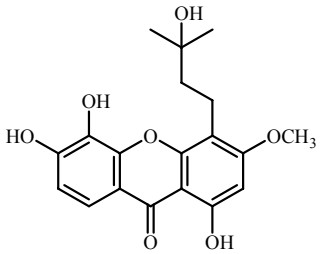
12.3hh : afzeliixanthone A



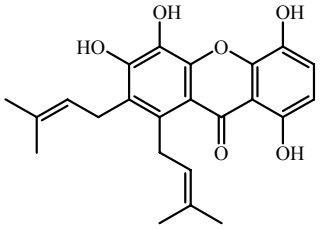
12.3ii : 1,3,5-trihydroxy-6-methoxyxanthone



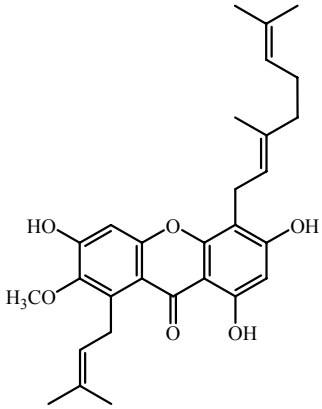
12.3jj : garciniaxanthone E



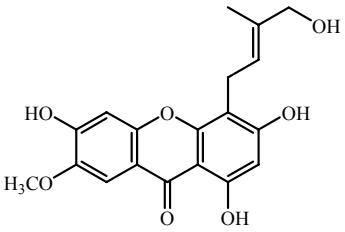
12.3kk : 1,5,6-trihydroxy-3-methoxy-4-(3-hydroxy-3-methylbutyl)xanthone



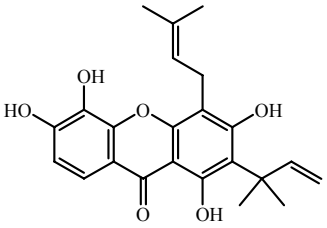
12.3ll : 1,4,5,6-tetrahydroxy-7,8-diprenylxanthone



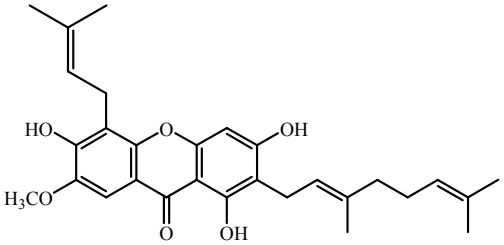
12.3mm : parivifolixanthone C



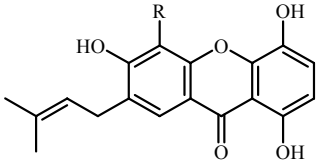
12.3nn : isocowanol



12.3oo : macluraxanthone C

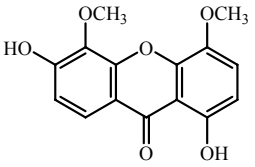


12.3pp : dulcisxanthone E

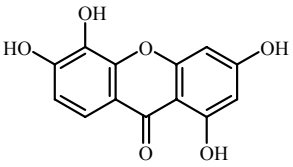


12.3qq : R = OCH_3 1,4,6-trihydroxy-5-methoxy-7-(3-methyl-2-buten-1-yl)xanthone

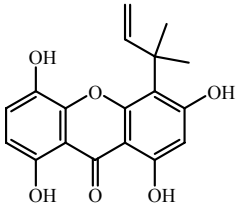
12.3rr : R = OH 1,4,5,6-tetrahydroxy-7-(3-methyl-2-buten-1-yl)xanthone



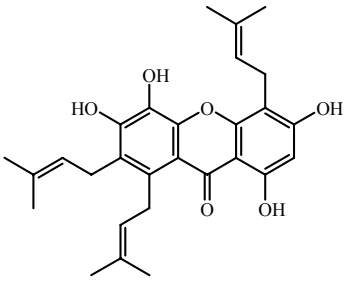
12.3ss : 1,6-dihydroxy-4,5-dimethoxyxanthone



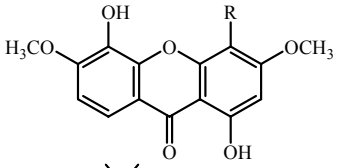
12.3tt : 1,3,5,6-tetrahydroxyxanthone

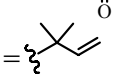


12.3uu : 4-(1,1-dimethylprop-2-enyl)-1,3,5,8-tetrahydroxyxanthone

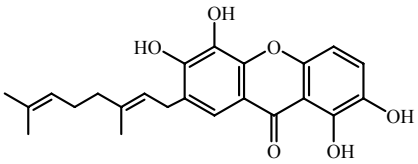


12.3vv : 1,3,5,6-tetrahydroxy-4,7,8-triprenylxanthone

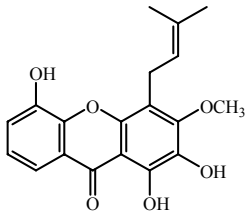


12.3ww : R =  vieillardiixanthone B

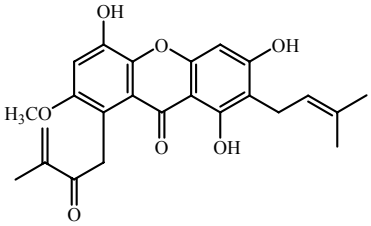
12.3xx : R =  vieillardiixanthone C



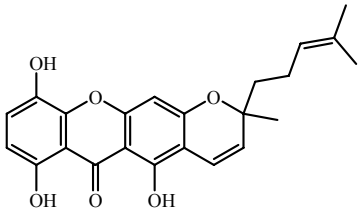
12.3yy : 7-(3,7-dimethyl-2,6-octadien-1-ly)-1,2,5,6-tetrahydroxyxanthone



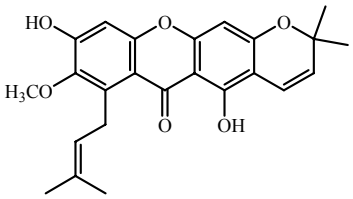
12.3zz : isocaledonixanthone D



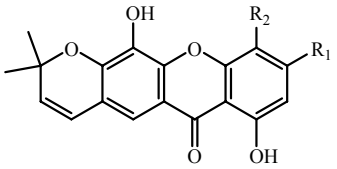
12.3aaa : mangostingone



12.3bbb : smeathxanthone B

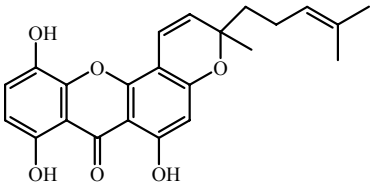


12.3ccc : 5,9-dihydroxy-8-methoxy-2,2-dimethyl-7-(3-methyl-but-2-enyl),2H,6H-pyrano-[3,2-b]-xanthen-6-one

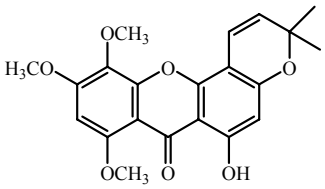


12.3ddd : $R_1 = OCH_3$, R_2 =  1,5-dihydroxy-3-methoxy-6',6'-dimethyl-2H-pyrano(2',3':6,7)-4-(3-methylbut-2-enyl)xanthone

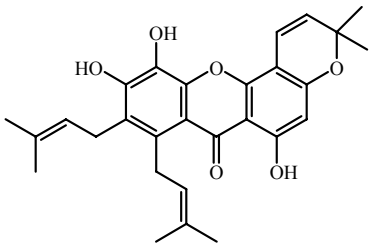
12.3eee : $R_1 = OH$, $R_2 = H$ 1,3,5-trihydroxy-6',6'-dimethyl-2H-pyrano(2',3':6,7)xanthone



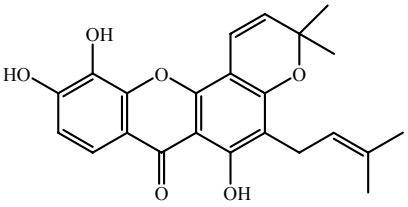
12.3fff : bangangxanthone A



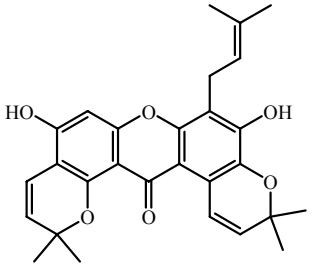
12.3ggg : asmaxanthone



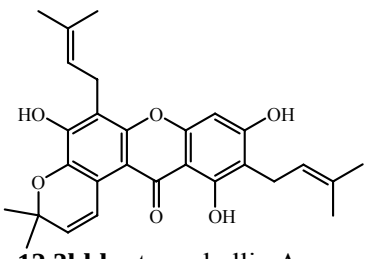
12.3hhh : 1,5,6,-trihydroxy-7-8-di(3-methyl-2-butenyl)-6',6'-dimethyl-pyrano(2',3':3,4)-xanthone



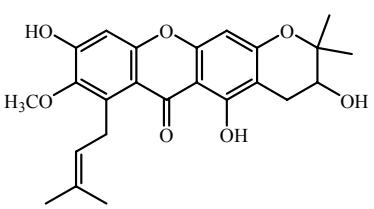
12.3iii : 1,5,6,-trihydroxy-6',6'-dimethyl-2H-pyrano (2',3',:3,4)-2-(3-methylbut-2-enyl)-xanthone



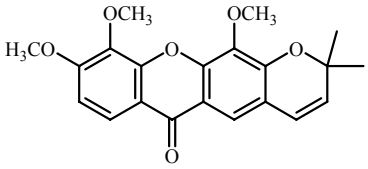
12.3jjj : tovophyllin B



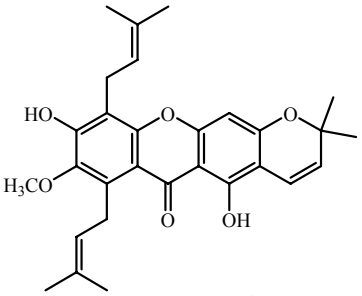
12.3kkk : tovophyllin A



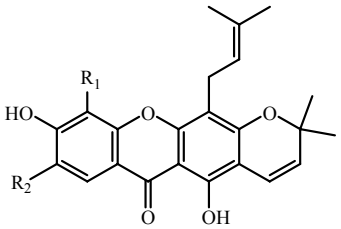
12.3lll : mangostanol



12.3mmm : porxanthone A

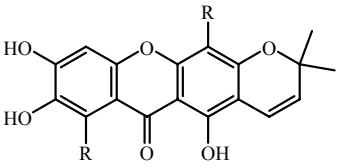


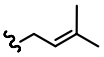
12.3nnn : cowaxanthone C

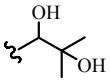


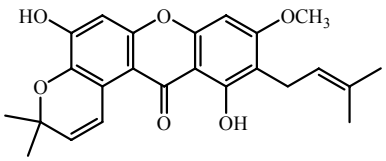
12.3ooo : R_1 = H, R_2 = OH 1,6,7-trihydroxy-6',6'- dimethyl-2H-pyrano-(2',3':3,2)-4-(3-methylbut-2-enyl)xanthone

12.3ppp : R_1 = OH, R_2 = H xanthone V1

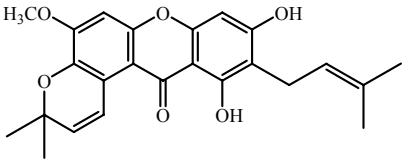


12.3qqq : R =  bannaxanthone D

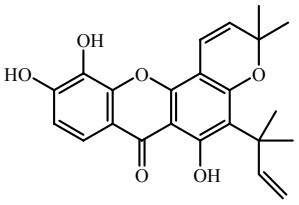
12.3rrr : R =  bannaxanthone G



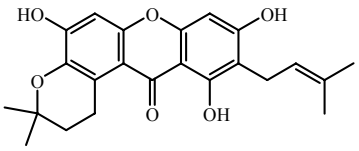
12.3sss : cowaxathone D



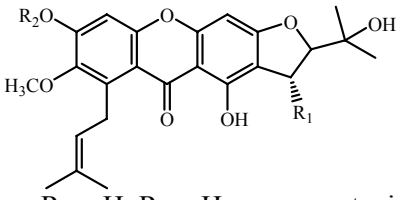
12.3ttt : dulcisxanthone F



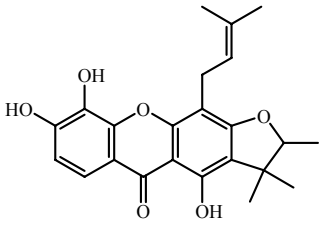
12.3uuu : cudratricusxanthone H



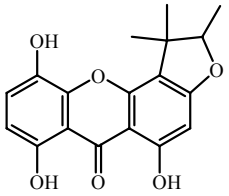
12.3vvv : mangostenone D



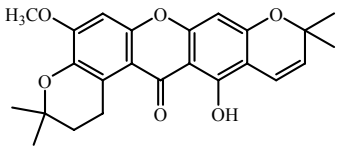
12.3www : R_1 = H, R_2 = H mangostanin
12.3xxx : R_1 = OH, R_2 = H mangostenone C
12.3yyy : R_1 = H, R_2 = CH_3 6-*O*-methyl mangostanin



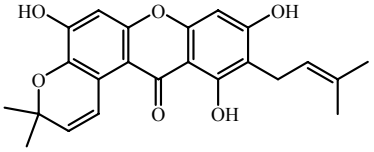
12.3zzz : gerontoxanthone C



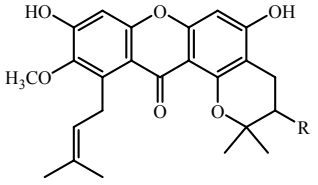
12.3aaaa : garbogiol



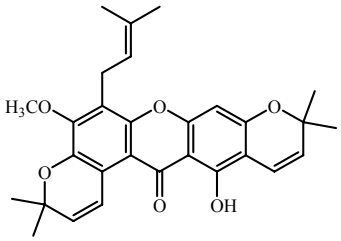
12.3bbbb : garcimangosone B



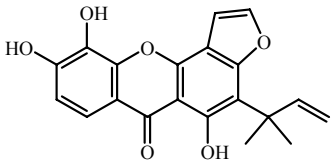
12.3cccc : garcinone B



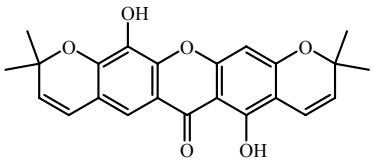
12.3dddd : R = OH 11-hydroxy-1-isomangostin
12.3eeee : R = H 1-isomangostin



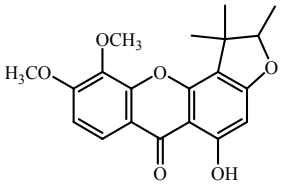
12.3ffff : dulcisxanthone D



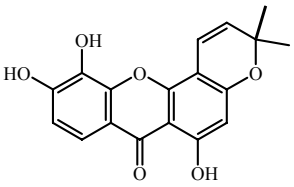
12.3gggg : penangianaxanthone



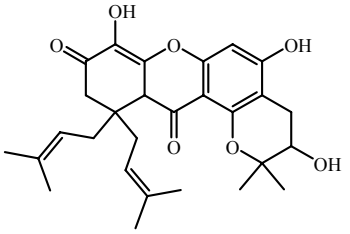
12.3hhhh : pyranojacareubin



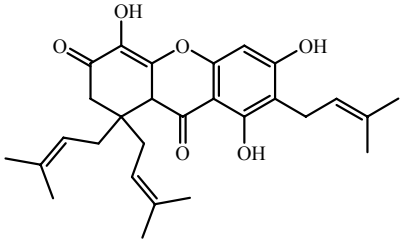
12.3iiii : 5,6-*O*-dimethyl-2-de-prenylrheediaxanthone



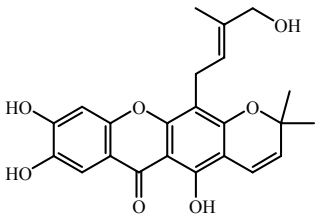
12.3jjjj : isojacareubin



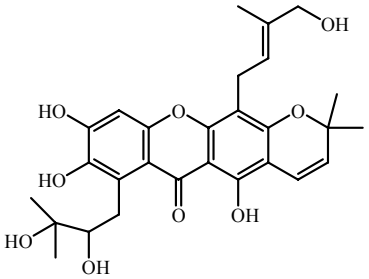
12.3kkkk : bannaxanthone H



12.3llll : allanxanthone C

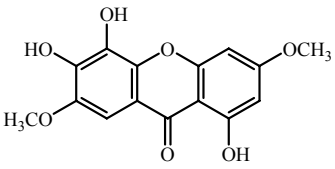


12.3mmmm : bannaxanthone E

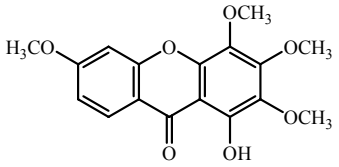


12.3nnnn : bannaxanthone F

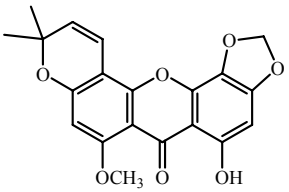
12.4 Pentaoxygenated xanthones



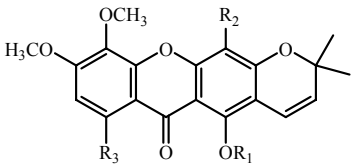
12.4a : 1,5,6-trihydroxy-3,7-dimethoxyxanthone



12.4b : dulcisxanthone C



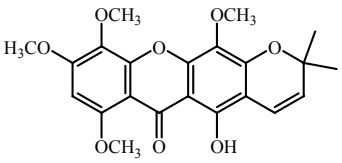
12.4c : musaxathone



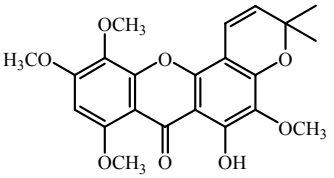
12.4d : $R_1 = CH_3$, $R_2 = OCH_3$, $R_3 = H$ dulxanthone E

12.4e : $R_1 = H$, $R_2 = H$, $R_3 = OCH_3$ dulxanthone F

12.5 Hexaoxygenated xanthones

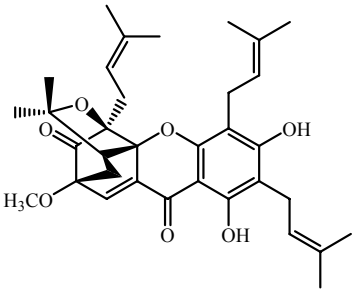


12.5a : dulxanthone G

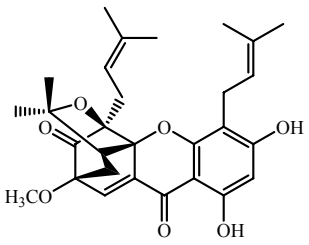


12.5b : yahyaxanthone

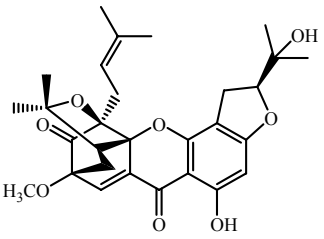
12.6 Caged-polyprenylated xanthones



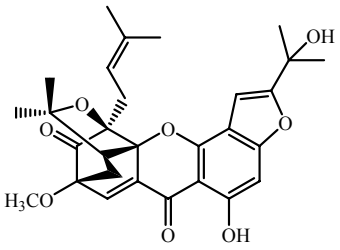
12.6a : cantleyanone A



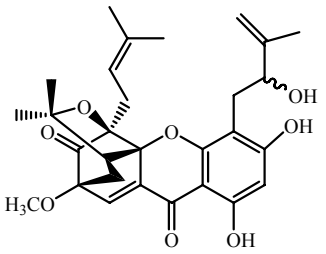
12.6b : 7-hydroxyforbesione



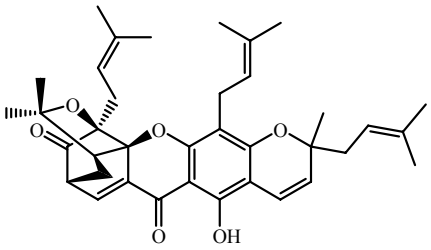
12.6c : cantleyanone B



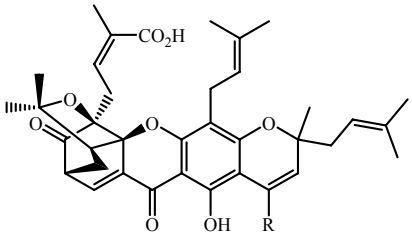
12.6d : cantleyanone C



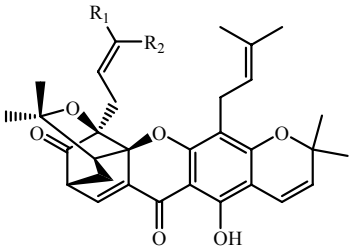
12.6e : cantleyanone D



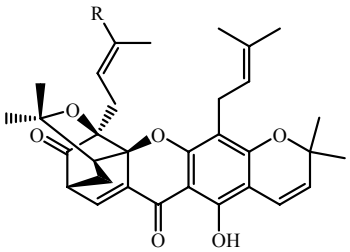
12.6f : gambogin



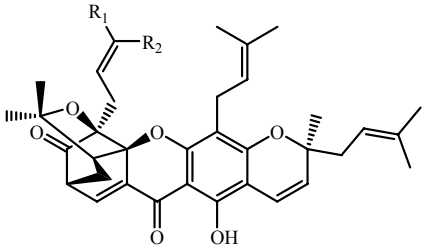
12.6g : R = OCH_3 10-methoxygambogic acid
12.6h : R = OCH_2CH_3 10-ethoxygambogic acid



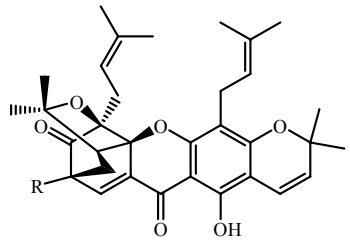
12.6i : R_1 = CH_3, R_2 = CHO morellin
12.6j : R_1 = CHO, R_2 = CH_3 isomorellin



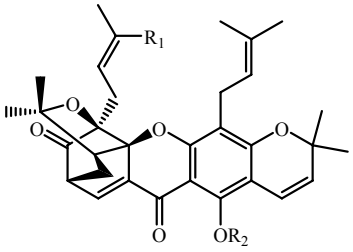
12.6k : R = CO_2 H isomorellic acid
12.6l : R = CH_2OH isomorellinol



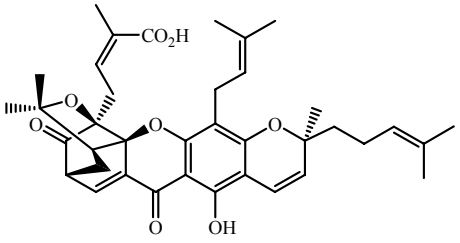
12.6m : R_1 = CO_2H, R_2 = CH_3 isogambogic acid
12.6n : R_1 = CH_2OH, R_2 = CO_2H 30-hydroxygambogic acid



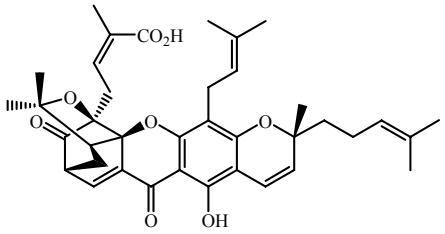
12.6o : R = H desoxymorellin
12.6p : R = OH 7-hydroxydesoxymorellin
12.6q : R = OCH_3 7-methoxydesoxymorellin



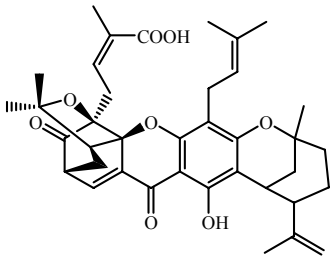
12.6r : $R_1 = CO_2H$, $R_2 = H$ morellic acid
12.6s : $R_1 = CO_2CH_3$, $R_2 = H$ 2-methyl-4-[(1*R*,3a*S*,5*S*,14a*S*)-3a,4,5,7-tetrahydro-8-hydroxy-3,3,11,11-tetramethyl-13-(3-methyl-2-buten-1-yl)-7,15-dioxo-1,5-methano-1H,3H,11H-furo[3,4-g]-pyrano[3,2-b]xanthen-1-yl]methyl ester
12.6t : $R_1 = CO_2CH_3$, $R_2 = CH_3$ 2-methyl-4-[(1*R*,3a*S*,5*S*,14a*S*)-3a,4,5,7-tetrahydro-8-methoxy-3,3,11,11-tetramethyl-13-(3-methyl-2-buten-1-yl)-7,15-dioxo-1,5-methano-1H,3H,11H-furo[3,4-g]-pyrano[3,2-b]xanthen-1-yl]methyl ester



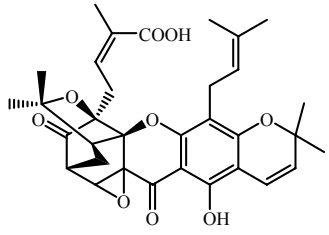
12.6u : gambogic acid



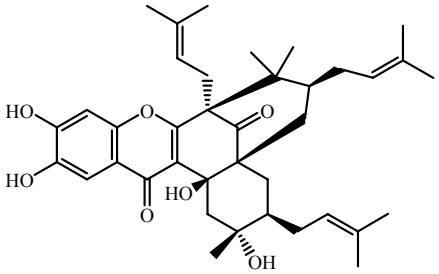
12.6v : epigambogic acid



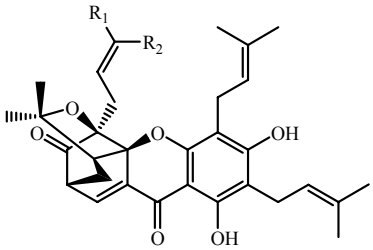
12.6w : gambogellic acid



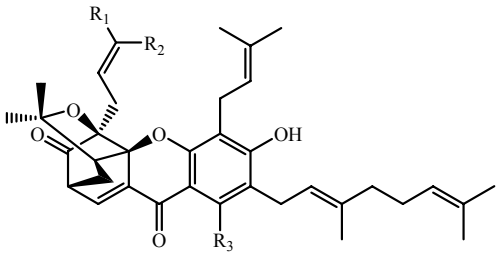
12.6x : 8,8a-epoxymorellic acid



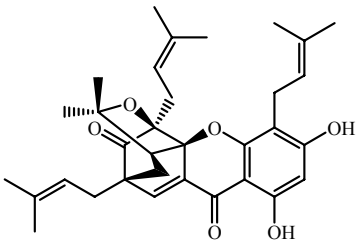
12.6y : garcinialone



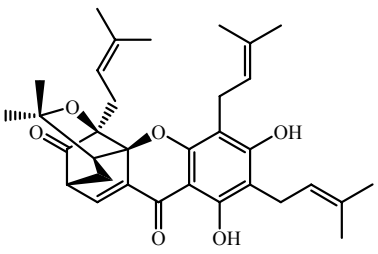
12.6z : $R_1 = CHO$, $R_2 = CH_3$ gambogic acid
12.6aa : $R_1 = CH_3$, $R_2 = CO_2H$ gaudichaudic acid
12.6bb : $R_1 = CH_3$, $R_2 = CH_3$ deoxygaudichaudione A



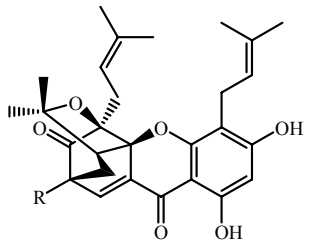
12.6cc : $R_1 = CO_2H$, $R_2 = CH_3$, $R_3 = OH$ isogambogenic acid
12.6dd : $R_1 = CH_3$, $R_2 = CO_2H$, $R_3 = OCH_3$ 10-methoxygambogenic acid
12.6ee : $R_1 = CH_3$, $R_2 = CH_3$, $R_3 = OH$ desoxygambogenin
12.6ff : $R_1 = CH_3$, $R_2 = CO_2H$, $R_3 = OH$ gambogenic acid



12.6gg : hanburin

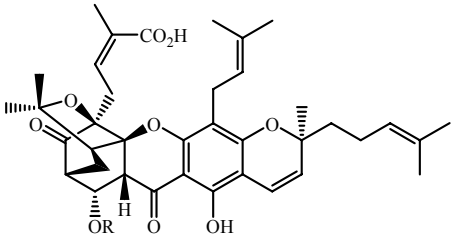


12.6hh : 2-isoprenylforbesione



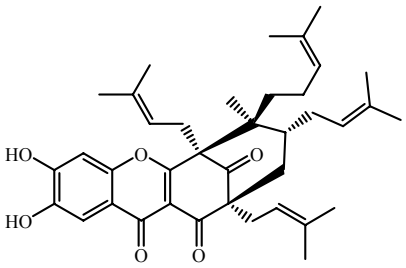
12.6ii : R = OCH_3 gaudichaudione H

12.6jj : R = H forbesione

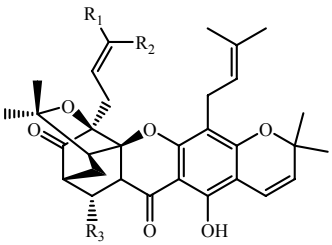


12.6kk : R = CH_3 gambogenic acid A

12.6ll : R = CH_2CH_3 gambogenic acid B



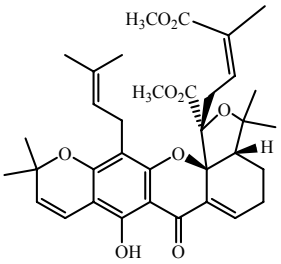
12.6mm : oxyguttiferone K



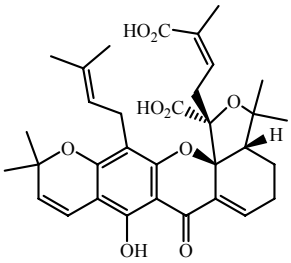
12.6nn : R_1 = CHO, R_2 = CH_3, R_3 = H dihydroisomorellin

12.6oo : R_1 = CH_3, R_2 = CO_2H, R_3 = OCH_3 moreollic acid

12.7 Rearranged xanthones

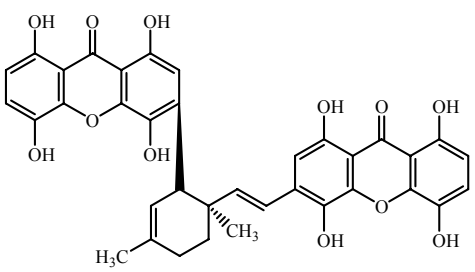


12.7a : 3a,4,5,7-tetrahydro-8-hydroxy-1-[(2*Z*)-4-methoxy-3-methyl-4-oxo-2-buten-1-yl]--3,3,11,11-tetramethyl-13-(3-methyl-2-buten-1-yl)-7-oxoxanthone methyl ester

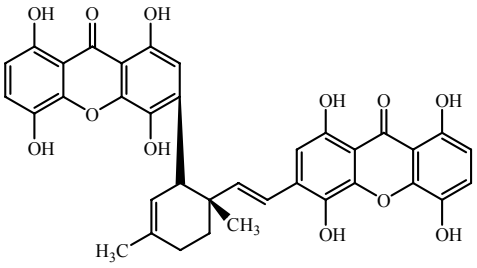


12.7b : guttiferic acid

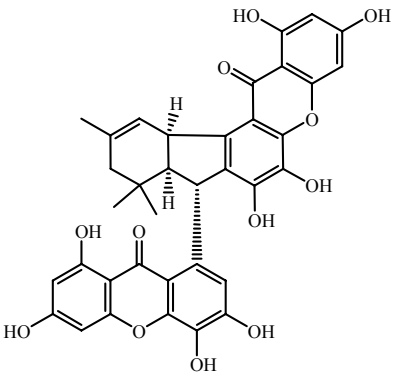
12.8 Bixanthones



12.8a : garcilivin A

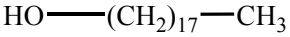


12.8b : garcilivin C

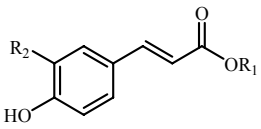


12.8c : griffipavixanthone

13. Miscellaneous

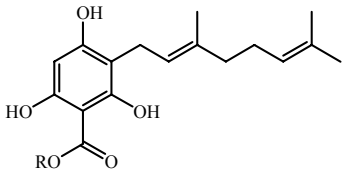


13a : 1-stearyl alcohol



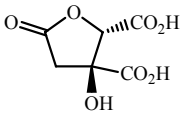
13b : R_1 = H, R_2 = H *p*-coumaric acid

13c : $R_1 = (CH_2)_{29}CH_3$, R_2 = OH triacontanyl caffeate

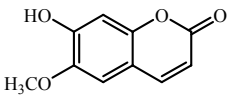


13d : R = CH_3 parvifoliol A

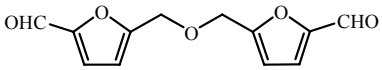
13e : R = H parvifoliol B



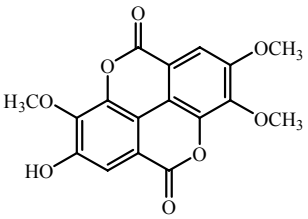
13f : garcinia lactone



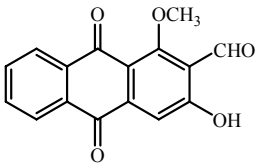
13g : scopoletin



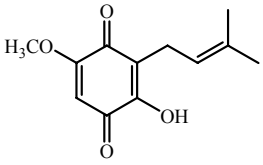
13h : cirsiumaldehyde



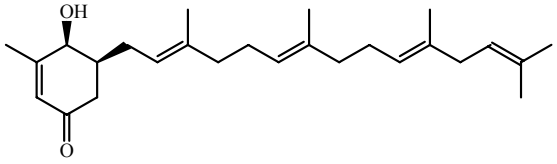
13i : 3,3',4-*O*-trimethylellagic acid



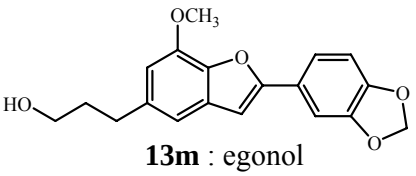
13j : damnacanthal



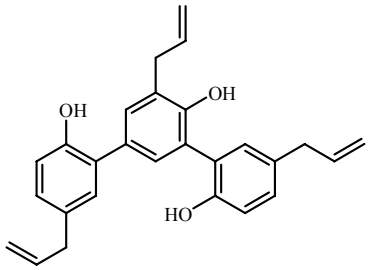
13k : parvifoliquinone



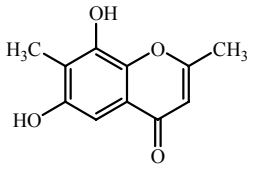
13l : (2*E*,6*E*,10*E*)-(+)-4β-hydroxy-3-methyl-5β-(3,7,-11,15-tetramethylhexadeca-2,6,10,14-tetraenyl)-cyclohex-2-en-1-one



13m : egonol



13n : macranthol



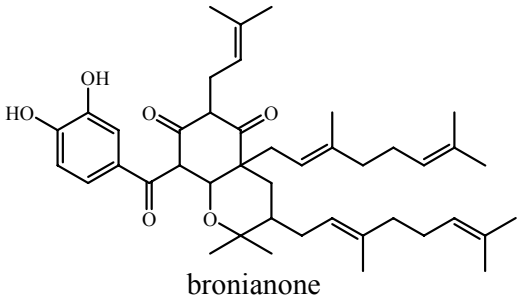
13o : dulcinone

1.1.3 The Objectives

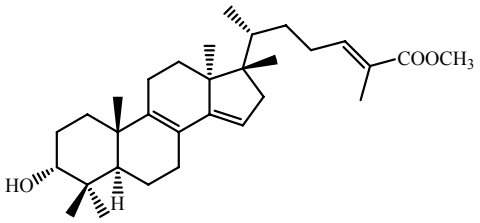
1.1.3.1 *Garcinia hombroniana*

Based on the literature search, phytochemical investigation on the stem woods (Ollis, 1969), pericarp (Rukachaisirikul, 2000) and leaves (Rukachaisirikul, 2005) of *G. hombroniana* resulted in the isolation of triterpenes as a major component. We are interested in investigation of its twigs in order to separate additional chemical constituents. This research involved isolation, purification and structure elucidation of chemical constituents from the twigs of *G. hombroniana* which were collected at Hat Yai campus, Prince of Songkla University.

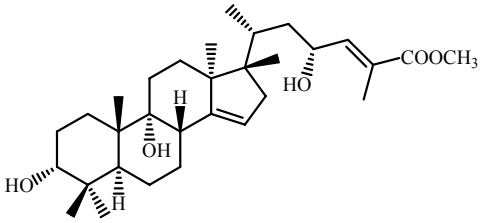
Structures of Compounds Isolated from *Garcinia hombroniana*



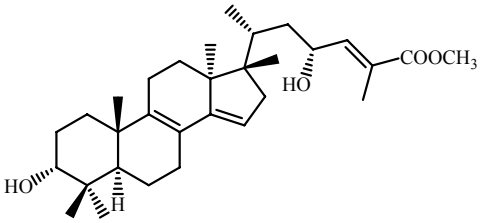
bronianone



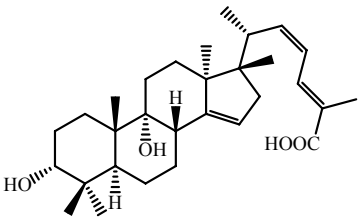
(24*E*)-3α-hydroxy-17,14-friedolanostan-8,14,24-trien-26-oic acid



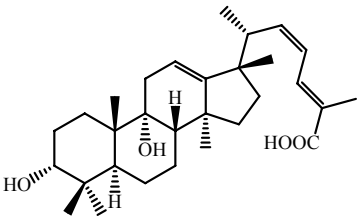
garcihombronane B



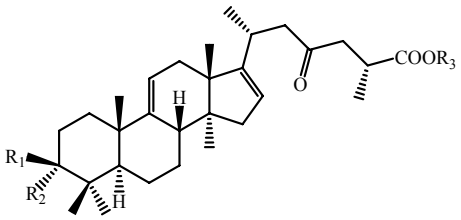
garcihombronane C



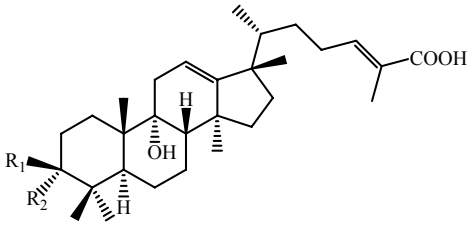
garcihombronane F

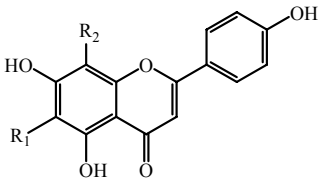


garcihombronane G



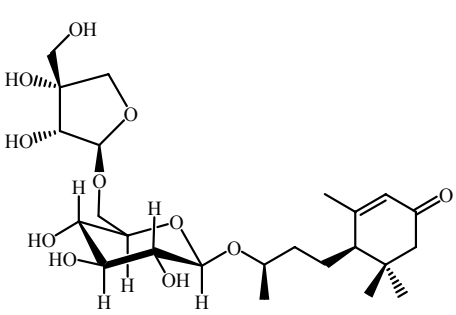
R_1 = H, R_2 = OH, R_3 = CH_3	garcihombronane J
R_1 = OH, R_2 = R_3 = H	garcihombronane D
R_1 = R_3 = H, R_2 = OH	garcihombronane E
R_1 = OH, R_2 = H, R_3 = CH_3	methyl (25*R*)-3β-(OH)-23-oxo-9,15-lanostadien-26-oate



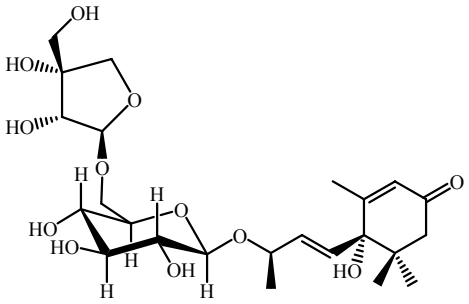


R_1 = H, R_2 = OH	garcihombronane H
R_1 = OH, R_2 = H	garcihombronane I

R_1 = H, R_2 = β-D-glucose	vitexin
R_1 = β-D-glucose, R_2 = H	isovitexin



blumenol C 9-*O*-*β*-D-apiofuranosyl-(1→6)-*β*-D-glucopyranoside



vomifoliol 9-*O*-*β*-D-apiofuranosyl-(1→ 6)-*β*-D-glucopyranoside

1.1.3.2 *Garcinia prainiana*

Based on the literature search, phytochemical investigation on *G. prainiana* has not been reported. This prompted us to investigate its chemical constituents in order to provide additional information of this plant. This research involved isolation, purification and structure elucidation of chemical constituents from the leaves of *G. prainiana* which were collected at Narathiwat Province.

CHAPTER 1.2

EXPERIMENTAL

1.2.1 Chemical and instruments

Melting points were determined on an electrothermal melting point apparatus (Electrothermal 9100) and reported without correction. Infrared spectra (IR) were obtained on a Perkin Elmer Spectrum GX FT-IR system and recorded on wavenumber (cm^{-1}). ^{1}H and ^{13}C-Nuclear magnetic resonance spectra (^{1}H and ^{13}C NMR) were recorded on a FTNMR, Bruker Avance 300 MHz or 500 MHz spectrometers using tetramethylsilane (TMS) as an internal standard. Spectra were recorded as chemical shift parameter (δ) value in ppm down field from TMS (δ 0.00). Ultraviolet spectra (UV) were measured with UV-160A spectrophotometer (SHIMADSU). Principle bands (λ_{max}) were recorded as wavelengths (nm) and log ε in methanol solution. Optical rotations were measured in methanol or chloroform solution with sodium D line (590 nm) on a JASCO P-1020 automatic polarimeter. Quick column chromatography, thin-layer chromatography (TLC) and precoated thin-layer chromatography were performed on silica gel 60 GF_{254} (Merck) or reverse-phase C-18 silica gel. Column chromatography was performed on silica gel (Merck) type 100 (70-230 Mesh ASTM), Sephadex LH-20 or reverse-phase C-18 silica gel. The solvents for extraction and chromatography were distilled at their boiling point ranges prior to use except for petroleum ether (bp. 40-60 °C) and ethyl acetate which were analytical grade reagent.

1.2.2 Plant material

The twigs of *G. hombroniana* were collected at Prince of Songkla University, Hat Yai, Songkhla, Thailand in 2000 while the leaves of *Garcinia prainiana* were

collected at Narathiwat Province, Thailand. The voucher specimens were deposited in the Herbarium of the Department of Biology, Faculty of Science, Prince of Songkla University, Hat Yai, Songkhla, Thailand.

1.2.3 Chemical investigation from the twigs of *G. hombroniana*

1.2.3.1 Isolation and extraction

The twigs of *G. hombroniana* (2.75 kg), cut into small segments, were extracted with MeOH (8 L) over the period of seven days at room temperature for three times. After filtration, the filtrate was evaporated to dryness under reduced pressure to give a crude methanol extract as a dark green gum in 170 g.

1.2.3.2 Chemical investigation of the crude methanol extract of the twigs of *G. hombroniana*

The crude extract was primarily tested for its solubility in various solvents at room temperature. The results were demonstrated in **Table 2**.

Table 2 Solubility of the crude extract in various solvents at room temperature

Solvent	Solubility at room temperature
Petroleum ether	-
Dichloromethane	+ (brown solution mixed with dark green gum)
Ethyl acetate	+ (brown solution mixed with dark green gum)
Acetone	++ (brown solution mixed with dark green gum)
Methanol	+++ (dark brown solution)
Water	++ (pale yellow solution)
10%HCl	++ (yellow solution mixed with dark green gum)
10%NaOH	+++ (dark brown solution)
10%$NaHCO_3$	+++ (dark brown solution)

Symbol meaning: + slightly soluble, ++ moderately soluble, +++ well soluble, - insoluble

The crude methanol extract was well soluble in methanol, 10%NaOH, 10% $NaHCO_3$ but it dissolved slightly in dichloromethane, ethyl acetate and acetone. These indicated that the crude extract contained slightly polar constituents. Chromatogram characteristics on normal phase TLC of the crude methanol extract, using 100%CH_2Cl_2 as a mobile phase, showed eight UV-active spots with the R_f values of 0.23, 0.25, 0.35, 0.46, 0.70, 0.72, 0.73 and 0.74. Further purification by quick column chromatography over silica gel was performed. Elution was conducted initially with pure CH_2Cl_2 and gradually enriched with methanol until pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford seven fractions as shown in **Table 3**.

Table 3 Fractions obtained from the crude methanol extract by quick column chromatography over silica gel

Fraction	Mobile phase	Weight (g)	Physical appearance
A	100%CH_2Cl_2	56.22	Yellow-green gum mixed with white solid
B	0.5%MeOH/CH_2Cl_2	11.57	Yellow-brown gum mixed with white solid
C	2%MeOH/CH_2Cl_2	4.02	Yellow-green gum mixed with white solid
D	5%MeOH/CH_2Cl_2	3.47	Yellow-green gum mixed with yellow-white solid
E	5%MeOH/CH_2Cl_2	0.94	Yellow-green gum
F	7-10%MeOH/CH_2Cl_2	4.04	Yellow-green gum mixed with white solid
G	10%MeOH/CH_2Cl_2-100%MeOH	63.07	Brown-black gum

Fraction A Upon standing at room temperature, a white solid (1.53 g) precipitated. Its chromatogram on normal phase TLC with 60%CH_2Cl_2/Petrol showed one major spot under ASA reagent with the R_f value of 0.33. Its ^1H NMR data indicated the presence of friedelin as a major component.

The filtrate became a yellow green gum (56.7 g) after evaporation to dryness under reduced pressure. Chromatogram characteristics on normal phase TLC with 100%CH_2Cl_2 showed seven UV-active spots with the R_f values of 0.23, 0.35, 0.43, 0.70, 0.72, 0.73 and 0.74. Further separation by column chromatography over Sephadex LH-20 was performed. Elution was conducted with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford seven fractions as shown in **Table 4**.

Table 4 Fractions obtained from the fraction **A** by column chromatography over Sephadex LH-20

Fraction	Weight (g)	Physical appearance
A1	1.23	Green yellow gum
A2	2.67	Green yellow gum
A3	25.70	Green yellow gum
A4	28.45	Yellow gum
A5	0.56	Yellow gum
A6	0.19	Yellow gun
A7	0.11	Brown gum

Fraction A1 Chromatogram characteristics on normal phase TLC with 100%CH_2Cl_2 showed none of well separated spots under UV-S. Its ^1H NMR data indicated the presence of long chain hydrocarbons. Thus, it was not further investigated.

Fraction A2 Chromatogram characteristics on normal phase TLC with 100%CH_2Cl_2 showed two UV-active spots with the R_f values of 0.23 and 0.35 and two brown spots and one purple spot under ASA reagent with the R_f values of 0.42, 0.59 and 0.73, respectively. It was further separated by column chromatography over

silica gel. Elution was conducted initially with pure dichloromethane, gradually enriched with acetone and then with methanol until methanol and finally with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford six fractions as shown in **Table 5**.

Table 5 Fractions obtained from the fraction **A2** by column chromatography over silica gel

Fraction	Mobile phase	Weight (g)	Physical appearance
A2A	100%CH_2Cl_2	0.28	Green yellow gum
A2B	100%CH_2Cl_2	0.56	Green yellow gum
A2C	100%CH_2Cl_2-20%Acetone/CH_2Cl_2	0.25	Green yellow gum
A2D	30-60%Acetone/CH_2Cl_2	0.69	Yellow gum
A2E	80%Acetone/CH_2Cl_2-100%Acetone	0.23	Yellow gum
A2F	1%MeOH/Acetone-100%MeOH	0.65	Brown yellow gum

Fraction A2A Chromatogram characteristics on normal phase TLC with 5%Acetone/CH_2Cl_2 showed none of well separated spots under UV-S. Its ^{1}H NMR data indicated the presence of long chain hydrocarbons. Thus, it was not further investigated.

Fraction A2B Chromatogram characteristics on normal phase TLC with 5%Acetone/CH_2Cl_2 showed three UV-active spots with the R_f values of 0.72, 0.77 and 0.82. Its ^{1}H NMR data indicated the presence of long chain hydrocarbons. Thus, it was not further investigated.

Fraction A2C Chromatogram characteristics on normal phase TLC with 5%Acetone/CH_2Cl_2 showed three UV-active spots with the R_f values of 0.25, 0.40 and 0.62 and two brown spots under ASA reagent with the R_f values of 0.75 and 0.82.

Its ^{1}H NMR data indicated the presence of long chain hydrocarbons. Thus, it was not further investigated.

Fraction A2D Chromatogram characteristics on normal phase TLC with 5%Acetone/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.35 and 0.40 and one purple spot under ASA reagent with the R_f value of 0.62. Its ^{1}H NMR data indicated the presence of **SK2** and **SK3** as major components. Further investigation was then not carried out.

Fraction A2E Chromatogram characteristics on normal phase TLC with 5%Acetone/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.05 and 0.12 and one brown spot under ASA reagent with the R_f value of 0.62. Its ^{1}H NMR data indicated the presence of **SK12** as a major component. Further investigation was then not carried out.

Fraction A2F Chromatogram characteristics on normal phase TLC with 5%Acetone/CH_2Cl_2 showed no definite spot under UV-S. Further investigation was then not carried out.

Fraction A3 Chromatogram characteristics on normal phase TLC with 100%CH_2Cl_2 showed three UV-active spots with the R_f values of 0.04, 0.19 and 0.23 and two brown spots under ASA reagent with the R_f values of 0.42 and 0.59. Further purification by quick column chromatography over silica gel was performed. Elution was conducted initially with pure dichloromethane and gradually enriched with methanol until pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford six fractions as shown in **Table 6**.

Table 6 Fractions obtained from the fraction **A3** by quick column chromatography over silica gel

Fraction	Mobile phase	Weight (g)	Physical appearance
A3A	100%CH_2Cl_2	0.25	Green yellow gum
A3B	1%MeOH/CH_2Cl_2	0.63	Green yellow gum
A3C	2-7%MeOH/CH_2Cl_2	11.23	Green yellow gum

Table 6 (continued)

Fraction	Mobile phase	Weight (g)	Physical appearance
A3D	10-20%MeOH/CH_2Cl_2	1.08	Yellow gum
A3E	20-60%MeOH/CH_2Cl_2	0.41	Yellow gum
A3F	80%MeOH/CH_2Cl_2-100%MeOH	0.26	Brown gum

Fraction A3A Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 (2 runs) showed none of well separated spots under ASA reagent. Its ^{1}H NMR data indicated the presence of long chain hydrocarbons. Thus, it was not further investigated.

Fraction A3B Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 (2 runs) showed three UV-active spots with the R_f values of 0.45, 0.50 and 0.59 and two purple spots under ASA reagent with the R_f values of 0.73 and 0.76. Its ^{1}H NMR data indicated the presence of long chain hydrocarbons. Thus, it was not further investigated.

Fraction A3C Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 (2 runs) showed three UV-active spots with the R_f values of 0.26, 0.35 and 0.42 and one brown spot under ASA reagent with the R_f value of 0.59. Its ^{1}H NMR data indicated the presence of **SK2** and **SK3** as major components. Further investigation was then not carried out.

Fraction A3D Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 (2 runs) showed three UV-active spots with the R_f values of 0.11, 0.19 and 0.26 and one brown spot under ASA reagent with the R_f value of 0.67. Its ^{1}H NMR spectrum showed broad signals. Thus, it was not further studied.

Fraction A3E Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 (2 runs) showed three UV-active spots with the R_f values of 0.04, 0.19 and 0.26. Its ^{1}H NMR spectrum showed broad signals. Thus, it was not further studied.

Fraction A3F Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 (2 runs) showed no definite spot under UV-S and ASA reagent. Thus, it was not further studied.

Fraction A4 Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 showed four UV-active spots with the R_f values of 0.11, 0.16, 0.23 and 0.35 and three brown spots under ASA reagent with the R_f values of 0.45, 0.47 and 0.61. Its ^{1}H NMR data were similar to those of fraction **A3**. Further investigation was then not carried out.

Fraction A5 Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 showed four UV-active spots with the R_f values of 0.07, 0.16, 0.23 and 0.35 and one purple spot under ASA reagent with the R_f value of 0.90. It was further separated by column chromatography over silica gel. Elution was conducted initially with pure dichloromethane, gradually enriched with methanol and finally with pure methanol. Fractions similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford eight fractions as shown in **Table 7**.

Table 7 Fractions obtained from the fraction **A5** by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
A5A	100%CH_2Cl_2	103.7	Yellow gum
A5B	1-2%MeOH/CH_2Cl_2	49.7	Yellow gum
A5C	5%MeOH/CH_2Cl_2	21.1	Yellow gum
A5D	7%MeOH/CH_2Cl_2	118.7	Yellow gum
A5E	7%MeOH/CH_2Cl_2	347.2	Yellow solid
A5F	10-15%MeOH/CH_2Cl_2	55.4	Yellow solid
A5G	20%MeOH/CH_2Cl_2	57.4	Yellow gum
A5H	40%MeOH/CH_2Cl_2-100%MeOH	55.5	Yellow gum

Fraction A5A Chromatogram characteristics on normal phase TLC with 80%CH_2Cl_2/Petrol showed two UV-active spots with the R_f values of 0.52 and 0.66 and two purple spots under ASA reagent with the R_f values of 0.87 and 0.95. Its ^1H NMR data indicated the presence of friedelin as a major component.

Fraction A5B Chromatogram characteristics on normal phase TLC with 100%CH_2Cl_2 showed four UV-active spots with the R_f values of 0.25, 0.35, 0.45 and 0.62. Its ^1H NMR spectrum displayed proton signals in the high field region. Thus, it was not further investigated.

Fraction A5C Chromatogram characteristics on normal phase TLC with 100%CH_2Cl_2 showed three UV-active spots with the R_f values of 0.05, 0.10 and 0.12. It was further separated by column chromatography over silica gel. Elution was conducted with pure dichloromethane. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford four fractions as shown in **Table 8**.

Table 8 Fractions obtained from the fraction **A5C** by column chromatography over silica gel

Fraction	Weight (mg)	Physical appearance
A5C1	3.0	Pale yellow gum
A5C2	4.0	Pale yellow solid
A5C3	7.5	Yellow solid
A5C4	5.2	Yellow gum

Fraction A5C1 Chromatogram characteristics on normal phase TLC with 100%CH_2Cl_2 showed none of well separated spots under UV-S. Its ^1H NMR spectrum displayed proton signals in the high field region. Thus, it was not further investigated.

Fraction A5C2 Chromatogram characteristics on normal phase TLC with 20%EtOAc/Petrol (3 runs) showed two UV-active spots with the R_f values of 0.25 and 0.50. Further purification by precoated TLC with 20%EtOAc/Petrol (7 runs) as a mobile phase afforded two bands.

Band 1 (SK20) was obtained as a yellow gum in 1.0 mg. Chromatogram characteristics on normal phase TLC with 20%EtOAc/Petrol (3 runs) showed one UV-active spot with the R_f value of 0.50.

UVλ_{max}(nm)(MeOH)(log ε)		222 (4.51), 258 (5.71), 278 (4,18), 345 (2.12)
FTIR(neat):υ(cm^{-1})		3443 (OH stretching), 1641 (C=O stretching)
^{1}H NMR(Acetone-d_6)(δ_{ppm})(500 MHz):		12.01 (*s*, 1H), 11.71 (*s*, 1H), 6.63 (*d*, *J* = 2.0 Hz, 1H), 6.36 (*d*, *J* = 2.0 Hz, 1H), 6.32 (*s*, 1H), 3.97 (*s*, 3H), 3.91 (*s*, 3H)
^{13}C NMR(Acetone-d_6)(δ_{ppm})(125 MHz):		184.38, 168.20, 163.72, 159.72, 159.00, 158.53, 150.44, 128.84, 102.55, 102.06, 99.32, 98.36, 93.94 61.82, 56.66
DEPT135° (Acetone-d_6)(δ_{ppm})	CH:	99.32, 98.36, 93.94
	CH_3:	61.82, 56.66
EIMS *m*/*z* (% relative intensity):		304 (57), 289 (100), 261 (60)

Band 2 (SK13) was obtained as a yellow gum in 1.2 mg. Chromatogram characteristics on normal phase TLC with 20%EtOAc/Petrol (3 runs) showed one UV-active spot with the R_f value of 0.25.

UVλ_{max}(nm)(MeOH)(log ε)	221 (2.81), 254 (2.99), 278 (2.41), 346 (1.38)
FTIR(neat):υ(cm^{-1})	3417 (OH stretching), 1661 (C=O stretching)
^{1}H NMR(Acetone-d_6)(δ_{ppm})(500 MHz):	12.03 (*s*, 1H), 11.23 (*s*, 1H), 7.25 (*d*, *J* = 8.5 Hz, 1H), 6.69 (*d*, *J* = 8.5 Hz, 1H) 6.32 (*s*, 1H), 5.27 (*t*, *J* = 7.0 Hz, 1H),

		5.04 (*m*, 1H), 3.56 (*d*, *J* = 7.0 Hz, 2H), 2.11 (*m*, 2H), 2.09 (*m*, 2H), 1.61 (*s*, 3H), 1.86 (*s*, 3H), 1.58 (*s*, 3H),
^{13}C NMR(Acetone-d_6)(δ_{ppm})(125 MHz):		184.79, 162.84, 161.42, 154.24, 154.02, 142.91, 139.23, 135.74, 132.22, 123.60, 123.31, 121.01, 110.15, 107.21, 105.50, 102.79, 99.36, 39.61, 26.36, 25.65, 21.93, 17.72, 16.38
DEPT135° (Acetone-d_6)(δ_{ppm})	CH:	123.60, 123.31, 121.01, 110.15, 99.36
	CH_2:	39.61, 26.36, 21.93
	CH_3:	25.65, 17.72, 16.38

Fraction A5C3 Chromatogram characteristics on normal phase TLC with 100%CH_2Cl_2 (2 runs) showed two UV-active spots with the R_f values of 0.30 and 0.37. Further purification by precoated TLC with 100%CH_2Cl_2 (4 runs) as a mobile phase afforded two bands.

Band 1 was obtained as a yellow solid in 3.2 mg. Chromatogram characteristics on normal phase TLC with 100%CH_2Cl_2 (2 runs) showed one UV-active spot with the R_f value of 0.37. Its ^{1}H NMR data indicated the presence of **SK13** as a major component. Further investigation was then not carried out.

Band 2 was obtained as a yellow gum in 1.0 mg. Chromatogram characteristics on normal phase TLC with 100%CH_2Cl_2 (2 runs) showed two UV-active spots with the R_f values of 0.30 and 0.37. Because of the minute quantity, it was not further investigated.

Fraction A5C4 Chromatogram characteristics on normal phase TLC with 15%Acetone/Petrol (3 runs) showed two UV-active spots with the R_f values of 0.35 and 0.42. Further purification by precoated TLC with 5%Acetone/Petrol (7 runs) as a mobile phase afforded two bands.

Band 1 was obtained as a yellow gum in 1.0 mg. Chromatogram characteristics on normal phase TLC with 15%Acetone/Petrol (3 runs) showed one UV-active spot with the R_f value of 0.42. Its ^{1}H NMR data indicated the presence of **SK13** as a major component. Further investigation was then not carried out.

Band 2 was obtained as a yellow gum in 1.2 mg. Chromatogram characteristics on normal phase TLC with 15%Acetone/Petrol (3 runs) showed two UV-active spots with the R_f values of 0.35 and 0.42. Because of the minute quantity, it was not further investigated.

Fraction A5D Chromatogram characteristics on normal phase TLC with 5%EtOAc/CH_2Cl_2 showed five UV-active spots with the R_f values of 0.02, 0.25, 0.37, 0.42 and 0.52. It was further separated by column chromatography over silica gel. Elution was conducted initially with 5%EtOAc/CH_2Cl_2, gradually enriched with ethyl acetate until pure ethyl acetate then enriched with methanol and finally with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford eight fractions as shown in **Table 9**.

Table 9 Fractions obtained from the fraction **A5D** by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
A5D1	5%EtOAc/CH_2Cl_2	2.4	Yellow gum
A5D2	5%EtOAc/CH_2Cl_2	9.0	Yellow gum
A5D3	5%EtOAc/CH_2Cl_2	20.4	Yellow gum
A5D4	5%EtOAc/CH_2Cl_2	20.5	Yellow gum
A5D5	7%EtOAc/CH_2Cl_2	21.2	Yellow gum
A5D6	10-15%EtOAc/CH_2Cl_2	36.5	Yellow gum
A5D7	20-40%EtOAc/CH_2Cl_2-100%EtOAc	62.9	Yellow gum
A5D8	100%EtOAc-100%MeOH	28.2	Yellow gum

Fraction A5D1 Chromatogram characteristics on normal phase TLC with 5%EtOAc/CH_2Cl_2 (6 runs) showed two brown spots under ASA reagent with the R_f values of 0.12 and 0.45 and two purple spots under ASA reagent with the R_f values of 0.62 and 0.70. Its ^{1}H NMR spectrum displayed proton signals in the high field region. Thus, it was not further investigated.

Fraction A5D2 Chromatogram characteristics on normal phase TLC with 5%EtOAc/Petrol (6 runs) showed three UV-active spots with the R_f values of 0.57, 0.37 and 0.32. Further purification by precoated TLC with 5%EtOAc/Petrol (12 runs) as a mobile phase afforded three bands. They were not further investigated because their chromatograms on normal phase TLC using 5%EtOAc/Petrol (6 runs) showed many spots under UV-S and they were obtained in low quantity. Moreover, their ^1H NMR spectra displayed many compounds.

Fraction A5D3 Chromatogram characteristics on normal phase TLC with 5%EtOAc/CH_2Cl_2 (6 runs) showed three UV-active spots with the R_f values of 0.07, 0.37 and 0.62 and two purple spots under ASA reagent with the R_f values of 0.77 and 0.87. Further separation by column chromatography over Sephadex LH-20 was performed. Elution was conducted with 5%EtOAc/CH_2Cl_2. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford five fractions. They were not further investigated because their chromatograms on normal phase TLC using 5%EtOAc/CH_2Cl_2 (6 runs) showed many spots under ASA reagent and they were obtained in low quantity.

Fraction A5D4 Chromatogram characteristics on normal phase TLC with 5%EtOAc/CH_2Cl_2 showed three UV-active spots with the R_f values of 0.07, 0.12 and 0.20 and one brown spot under ASA reagent with the R_f value of 0.47. Further separation by column chromatography over Sephadex LH-20 was performed. Elution was conducted with 50%MeOH/CH_2Cl_2. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford four fractions. They were not further investigated because their chromatograms on normal phase TLC using 100%CH_2Cl_2 showed many spots under ASA reagent and they were obtained in low quantity.

Fraction A5D5 Chromatogram characteristics on normal phase TLC with 15%EtOAc/Petrol (4 runs) showed three UV-active spots with the R_f values of 0.12, 0.17 and 0.32. Further separation by column chromatography over Sephadex LH-20 was performed. Elution was conducted with 50%MeOH/CH_2Cl_2. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford five fractions. They were not further investigated because

their chromatograms on normal phase TLC using 15%EtOAc/Petrol showed many spots under ASA reagent and they were obtained in low quantity.

Fraction A5D6 Chromatogram characteristics on normal phase TLC with 10%EtOAc/CH_2Cl_2 showed three UV-active spots with the R_f values of 0.37, 0.45 and 0.47. Its ^1H NMR spectrum displayed proton signals in the high field region. Thus, it was not further investigated.

Fraction A5D7 Chromatogram characteristics on normal phase TLC with 10%EtOAc/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.25 and 0.37. Its ^1H NMR spectrum displayed proton signals in the high field region. Thus, it was not further investigated.

Fraction A5D8 Chromatogram characteristics on normal phase TLC with 10%EtOAc/CH_2Cl_2 showed no definite spot under UV-S. It was not further investigated.

Fraction A5E Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 showed three UV-active spots with the R_f values of 0.07, 0.20 and 0.37. It was further separated by column chromatography over silica gel. Elution was conducted initially with pure dichloromethane, gradually enriched with methanol and finally with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford eight fractions as shown in **Table 10**.

Table 10 Fractions obtained from the fraction **A5E** by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
A5E1	100%CH_2Cl_2	15.6	Yellow gum
A5E2	1-3%CH_2Cl_2	50.5	Yellow gum
A5E3	3-7%MeOH/CH_2Cl_2	74.2	Yellow gum
A5E4	7%MeOH/CH_2Cl_2	60.5	Yellow gum
A5E5	10%MeOH/CH_2Cl_2	30.5	Yellow gum
A5E6	12-20%MeOH/CH_2Cl_2	70.6	Yellow gum

Table 10 (continued)

Fraction	Mobile phase	Weight (mg)	Physical appearance
A5E7	20-60%MeOH/CH_2Cl_2	20.8	Yellow gum
A5E8	60%MeOH/CH_2Cl_2-100%MeOH	24.5	Yellow gum

Fraction A5E1 Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 (2 runs) showed three purple spots under ASA reagent with the R_f values of 0.75, 0.87 and 0.95. Its ^{1}H NMR spectrum displayed proton signals in the high field region. Thus, it was not further investigated.

Fraction A5E2 Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 showed four UV-active spots with the R_f values of 0.40, 0.50, 0.55 and 0.65 and two purple spots under ASA reagent with the R_f values of 0.75 and 0.82. Further separation by column chromatography over Sephadex LH-20 was performed. Elution was conducted with 50%MeOH/CH_2Cl_2. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford three fractions as shown in **Table 11**.

Table 11 Fractions obtained from the fraction **A5E2** by column chromatography over Sephadex LH-20

Fraction	Weight (mg)	Physical appearance
A5E2A	10.1	Yellow gum
A5E2B	36.2	Yellow gum
A5E2C	8.1	Yellow gum

Fraction A5E2A Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 showed none of well separated spots under UV-S. Its ^{1}H NMR spectrum displayed proton signals in the high field region. Thus, it was not further investigated.

Fraction A5E2B Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 showed three UV-active spots with the R_f values of 0.45, 0.55 and 0.65 and two purple spots under ASA reagent with the R_f values of 0.75 and 0.82. Its ^{1}H NMR data indicated the presence of **SK2** and **SK3** as major components. Further investigation was then not carried out.

Fraction A5E2C Chromatogram characteristics on normal phase TLC with 0.5%MeOH/CH_2Cl_2 (5 runs) showed two UV-active spots with the R_f values of 0.25 and 0.30. Further purification by precoated TLC was carried out with 0.5%MeOH/CH_2Cl_2 (10 runs) as a mobile phase afforded two bands.

Band 1 (SK17) was obtained as a yellow gum in 3.1 mg. Chromatogram characteristics on normal phase TLC with 0.5%MeOH/CH_2Cl_2 (5 runs) showed one UV-active spot with the R_f value of 0.30.

UVλ_{max}(nm)(MeOH)(log ε)		278 (3.51)
FTIR(neat):υ(cm^{-1})		3338 (OH stretching), 1690 (C=O stretching)
^{1}H NMR($CDCl_3$)(δ_{ppm})(300 MHz):		7.59 (*d*, *J* = 1.8 Hz, 1H), 7.55 (*dd*, *J* = 8.1 and 1.8 Hz, 1H), 6.90 (*d*, *J* = 8.1 Hz, 1H), 3.88 (s, 3H)
^{13}C NMR($CDCl_3$)(δ_{ppm})(75 MHz):		167.00, 148.49, 142.14, 123.90, 123.87, 116.61, 114.88, 52.05
DEPT135° ($CDCl_3$)(δ_{ppm})	CH:	123.87, 116.61, 114.88
	CH_3:	52.05

Band 2 (SK18) was obtained as a yellow gum in 3.2 mg. Chromatogram characteristics on normal phase TLC with 0.5%MeOH/CH_2Cl_2 (5 runs) showed one UV-active spot with the R_f value of 0.25.

$[\alpha]^{26}$	-5.0º (c = 0.04, MeOH)
UVλ_{max}(nm)(MeOH)(log ε)	222 (3.82), 229 (3.14), 250 (2.57), 259 (2.47), 277 (1.68)

FTIR(neat):υ(cm^{-1})		3541 (OH stretching), 1648 (C=O stretching)
^{1}H NMR(Acetone-d_6)(δ_{ppm})(500 MHz):		11.98 (*s*, 1H), 11.30 (*s*, 1H), 7.32 (*d*, *J* = 8.7 Hz, 1H), 6.62 (*d*, *J* = 8.7 Hz, 1H), 6.40 (*s*, 1H), 5.39 (*mt*, *J* = 7.0 Hz, 1H), 4.83 (*brs*, 1H), 4.69 (*brs*, 1H), 3.94 (*t*, *J* = 6.6 Hz, 1H), 3.58 (*d*, *J* = 7.0 Hz, 2H), 2.20 (*m*, 2H), 1.86 (*s*, 3H), 1.64 (*s*, 3H), 1.60 (*m*, 2H)
^{13}C NMR(Acetone-d_6)(δ_{ppm})(125 MHz):		185.86, 165.25, 161.86, 155.78, 154.23, 149.35, 145.20, 138.24, 135.94, 124.81, 123.02, 110.31, 110.07, 108.03, 102.65, 99.05, 75.32, 36.59, 34.56, 22.05, 17.81, 16.48
DEPT135° (Acetone-d_6)(δ_{ppm})	CH:	124.81, 123.02, 110.07, 99.05, 75.32
	CH_2:	110.31, 36.59, 34.56, 22.05
	CH_3:	17.81, 16.48
EIMS *m*/*z* (% relative intensity):		412 (2), 394 (4), 311 (20), 273 (100), 260 (5), 121 (7)

Fraction A5E3 Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 (2 runs) showed two UV-active spots with the R_f values of 0.12 and 0.25. Its ^{1}H NMR data indicated the presence of **SK2** and **SK3** as major constituents. Further investigation was then not carried out.

Fraction A5E4 Chromatogram characteristics on normal phase TLC with 15%Acetone/CH_2Cl_2 showed four UV-active spots with the R_f values of 0.07, 0.11, 0.54 and 0.59 and two purple spots under ASA reagent with the R_f values of 0.60 and 0.75. It was further separated by column chromatography over silica gel. Elution was conducted initially with 10%Acetone/Petrol, gradually enriched with acetone until pure acetone then enriched with methanol and finally with pure methanol. Fractions

with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford six fractions as shown in **Table 12**.

Table 12 Fractions obtained from the fraction **A5E4** by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
A5E4A	10-30%Acetone/Petrol	9.0	Colorless gum
A5E4B	40-50%Acetone/Petrol	26.5	Yellow gum
A5E4C	70%Acetone/Petrol	8.3	Colorless gum
A5E4D	80-90%Acetone/Petrol	5.1	Colorless gum
A5E4E	100%Acetone-10%MeOH/Acetone	8.0	Colorless gum
A5E4F	10%MeOH/Acetone-100%MeOH	16.1	Yellow gum

Fraction A5E4A Chromatogram characteristics on normal phase TLC with 15%Acetone/Petrol showed one UV-active spot with the R_f value of 0.33 and long tail under UV-S. Its ^{1}H NMR data indicated the presence of **SK2** as a major component. Further investigation was then not carried out.

Fraction A5E4B Chromatogram characteristics on normal phase TLC with 15%Acetone/Petrol showed one UV-active spot with the R_f value of 0.23 and long tail. Its ^{1}H NMR data indicated the presence of **SK3** as a major component. Further investigation was then not carried out.

Fraction A5E4C Chromatogram characteristics on normal phase TLC with 15%Acetone/Petrol showed two UV-active spots with the R_f values of 0.50 and 0.54 and one brown spot under ASA reagent with the R_f value of 0.59. It was further subjected to acetylation reaction in acetic anhydride (3 ml) in the presence of pyridine (1 ml). The reaction mixture was stirred at room temperature overnight. After working up, the acetate derivative (**A5E4CA**) was obtained as a pale yellow gum (5.1 mg). Chromatogram characteristics on normal phase TLC with 10%Aectone/Petrol showed two UV-active spots with the R_f values of 0.55 and 0.60 and one purple spot under

ASA reagent with the R_f value of 0.63. Because its ^1H NMR data indicated the presence of many compounds, it was not further investigated.

Fraction A5E4D Chromatogram characteristics on normal phase TLC with 15%Acetone/Petrol (11 runs) showed two UV-active spots with the R_f values of 0.50 and 0.52. Further purification by precoated TLC with 15%Acetone/Petrol (22 runs) as a mobile phase afforded a colorless gum in 1.5 mg. Chromatogram characteristics on normal phase TLC with 15%Acetone/Petrol (11 runs) showed two UV-active spots with the R_f values of 0.50 and 0.52. Because its ^1H NMR data indicated the presence of many compounds, it was not further investigated.

Fraction A5E4E Chromatogram characteristics on normal phase TLC with 15%Acetone/Petrol (9 runs) showed two UV-active spots with the R_f values of 0.40 and 0.50 and two purple spots under ASA reagent with the R_f values of 0.42 and 0.52. Further purification by precoated TLC with 15%Acetone/Petrol (18 runs) as a mobile phase afforded two bands. They were not further investigated because their chromatograms on normal phase TLC using 15%Acetone/Petrol (9 runs) showed many spots under UV-S and they were obtained in low quantity. Moreover, their ^1H NMR spectra displayed many compounds.

Fraction A5E4F Chromatogram characteristics on normal phase TLC with 15%Acetone/Petrol showed no definite spot under UV-S. It was not further investigated.

Fraction A5E5 Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 (5 runs) showed two UV-active spots with the R_f values of 0.35 and 0.62. Its ^1H NMR data indicated the presence of **SK3** as a major compound. Further investigation was then not carried out.

Fraction A5E6 Chromatogram characteristics on normal phase TLC with 20%Acetone/Petrol showed five UV-active spots with the R_f values of 0.12, 0.19, 0.29, 0.34 and 0.39. It was further purified by column chromatography over silica gel. Elution was conducted initially with 20%Acetone/Petrol, gradually enriched with acetone until pure acetone then enriched with methanol and finally with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford five fractions as shown in **Table 13**.

Table 13 Fractions obtained from the fraction **A5E6** by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
A5E6A	20%Acetone/Petrol	9.0	Colorless gum
A5E6B	30-60%Acetone/Petrol	17.1	Colorless gum
A5E6C	70-90%Acetone/Petrol-100%Acetone	6.4	Colorless gum
A5E6D	1-10%MeOH/Acetone	8.2	Pale yellow gum
A5E6E	20%MeOH/Acetone-100%MeOH	35.0	Yellow gum

Fraction A5E6A Chromatogram characteristics on normal phase TLC with 20%Acetone/Petrol showed three UV-active spots with the R_f values of 0.04, 0.29 and 0.43 and two purple spots under ASA reagent with the R_f values of 0.75 and 0.85. Its ^{1}H NMR spectrum displayed proton signals in the high field region. Thus, it was not further investigated.

Fraction A5E6B Chromatogram characteristics on normal phase TLC with 20%Acetone/Petrol showed two UV-active spots with the R_f values of 0.29 and 0.34 and one brown spot under ASA reagent with the R_f value of 0.60. Its ^{1}H NMR data indicated the presence of **SK1** as a major component. Further investigation was then not carried out.

Fraction A5E6C Chromatogram characteristics on normal phase TLC with 20%Acetone/Petrol showed four UV-active spots with the R_f values of 0.12, 0.19, 0.34 and 0.39. Because of low quantity, it was not further investigated.

Fraction A5E6D Chromatogram characteristics on normal phase TLC with 15%Acetone/Petrol (10 runs) showed three UV-active spots with the R_f values of 0.12, 0.34 and 0.39. Further purification by precoated TLC with 15%Acetone/Petrol (20 runs) as a mobile phase gave three bands. They were not further investigated because their chromatograms on normal phase TLC using 15%Acetone/Petrol (10 runs) showed many spots under UV-S and they were obtained in low quantity. Moreover, their ^{1}H NMR spectra displayed signals of many compounds.

Fraction A5E6E Chromatogram characteristics on normal phase TLC with 20%Acetone/Petrol showed no definite spot under UV-S and ASA reagent. Thus, it was not further studied.

Fraction A5E7 Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 (5 runs) showed three UV-active spots with the R_f values of 0.35, 0.37 and 0.57. Its ^{1}H NMR spectrum displayed broad signals. Thus, it was not further studied.

Fraction A5E8 Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 (5 runs) showed no definite spot under UV-S and ASA reagent. Thus, it was not further studied.

Fraction A5F Chromatogram characteristics on normal phase TLC with 20%Acetone/Petrol (3 runs) showed four UV-active spots with the R_f values of 0.11, 0.28, 0.33, and 0.38 and one brown spot under ASA reagent with the R_f value of 0.54. It was further separated by column chromatography over silica gel. Elution was conducted initially with 20%Acetone/Petrol, gradually enriched with acetone until pure acetone then enriched with methanol and finally with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford six fractions as shown in **Table 14**.

Table 14 Fractions obtained from the fraction **A5F** by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
A5F1	20-30%Acetone/Petrol	4.3	Yellow gum
A5F2	40-50%Acetone/Petrol	12.3	Yellow gum
A5F3	60%Acetone/Petrol-100%Acetone	8.8	Pale yellow gum
A5F4	1-10%MeOH/Acetone	6.2	Pale yellow gum
A5F5	10-40%MeOH/Acetone	18.2	Pale yellow gum
A5F6	60%MeOH/Acetone-100%MeOH	6.7	Pale yellow gum

Fraction A5F1 Chromatogram characteristics on normal phase TLC with 20%Acetone/Petrol (3 runs) showed none of well separated spots under UV-S. Its ^{1}H NMR spectrum displayed proton signals in the high field region. Thus it was not further investigated.

Fraction A5F2 Chromatogram characteristics on normal phase TLC with 15%Acetone/Petrol (8 runs) showed three UV-active spots with the R_f values of 0.10, 0.20 and 0.35. Further purification by precoated TLC with 15%Acetone/Petrol (17 runs) as a mobile phase afforded three bands. They were not further investigated because their chromatograms on normal phase TLC using 15%Acetone/Petrol (8 runs) showed many spots under UV-S and they were obtained in low quantity. Moreover, their ^{1}H NMR spectra displayed signal of many compounds.

Fraction A5F3 Chromatogram characteristics on normal phase TLC with 20%Acetone/Petrol (3 runs) showed four UV-active spots with the R_f values of 0.11, 0.28, 0.33 and 0.38. Its ^{1}H NMR data indicated the presence of **SK12** as a major component. Further investigation was then not carried out.

Fraction A5F4 Chromatogram characteristics on normal phase TLC with 15%Acetone/Petrol (8 runs) showed one UV-active spot with the R_f value of 0.35 and one brown spot under ASA reagent with the R_f value of 0.38. Further purification by precoated TLC with 15%Acetone/Petrol (17 runs) as a mobile phase afforded a colorless gum in 1.8 mg. Chromatogram characteristics on normal phase TLC with 15%Acetone/Petrol (8 runs) showed one UV-active spot with the R_f value of 0.35. Because its ^{1}H NMR data indicated the presence of many compounds, it was not further investigated.

Fraction A5F5 Chromatogram characteristics on normal phase TLC with 20%Acetone/Petrol (3 runs) showed four UV-active spots with the R_f values of 0.04, 0.11, 0.14 and 0.28. Its ^{1}H NMR spectrum displayed broad signals. Thus, it was not further studied.

Fraction A5F6 Chromatogram characteristics on normal phase TLC with 20%Acetone/Petrol (3 runs) showed no definite spot under UV-S and under ASA reagent. Thus, it was not further studied.

Fraction A5G Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.12 and 0.20.

Its ^{1}H NMR data showed **SK12** as a major component. Further investigation was then not carried out.

Fraction A5H Chromatogram characteristics on normal phase TLC with 15%Acetone/Petrol (2 runs) showed two UV-active spots with the R_f values of 0.19 and 0.28 and two brown spots under ASA reagent with the R_f values of 0.21 and 0.33. It was further separated by column chromatography over silica gel. Elution was conducted initially with 15%Acetone/CH_2Cl_2, gradually enriched with acetone until pure acetone then enriched with methanol and finally with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford four fractions as shown in **Table 15**.

Table 15 Fractions obtained from the fraction **A5H** by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
A5H1	15%Acetone/CH_2Cl_2-100%Acetone	2.4	Yellow gun
A5H2	1%MeOH/Acetone	2.0	Pale yellow gum
A5H3	1-20%MeOH/Acetone	31.9	Yellow gum
A5H4	20%MeOH/Acetone-100%MeOH	14.2	Yellow gum

Fraction A5H1 Chromatogram characteristics on normal phase TLC with 15%Acetone/Petrol (2 runs) showed four purple spots under ASA reagent with the R_f values of 0.21, 0.33, 0.83 and 0.95. Its ^{1}H NMR spectrum displayed proton signals in the high field region. Thus, it was not further investigated.

Fraction A5H2 (**SK9**) Chromatogram characteristics on normal phase TLC with 15%Acetone/Petrol (2 runs) showed one UV-active spot with the R_f value of 0.28.

$[\alpha]_D^{27}$	-176.3º (c = 0.08, MeOH)
UV λ_{max}(nm)(MeOH)(log ε)	257 (2.83)

FTIR(neat):$\upsilon(cm^{-1})$		3404 (OH stretching), 1713 (C=O stretching)
^{1}H NMR($CDCl_3$)(δ_{ppm})(500 MHz):		7.65 (*t*, *J* = 11.5 Hz, 1H), 6.22 (*t*, *J* = 11.5 Hz, 1H), 5.98 (*t*, *J* = 11.0 Hz, 1H), 5.35 (*brs*, 1H), 3.23 (*dd*, *J* = 11.0 and 4.0 Hz, 1H), 3.21 (*m*, 1H), 2.37 (*m*, 1H), 2.28 (*m*, 1H), 2.03 (*m*, 1H), 1.98 (*m*, 1H), 1.97 (*s*, 3H), 1.85 (*m*, 1H), 1.84 (*m*, 1H), 1.69 (*m*, 1H), 1.64 (*m*, 1H), 1.57 (*m*, 1H), 1.53 (*m*, 2H), 1.52 (*m*, 1H), 1.51 (*m*, 1H), 1.50 (*m*, 1H), 1.40 (*m*, 1H), 1.34 (*m*, 1H), 1.08 (*s*, 3H), 0.99 (*s*, 3H), 0.93 (*d*, *J* = 7.0 Hz, 3H), 0.89 (*s*, 3H), 0.79 (*s*, 6H)
^{13}C NMR($CDCl_3$)(δ_{ppm})(125 MHz):		172.10, 153.30, 144.07, 134.97, 126.05, 121.90, 120.24, 78.65, 75.48, 53.76, 49.26, 44.96, 44.19, 42.27, 39.03, 38.77, 36.96, 29.71, 29.56, 28.95, 27.47, 27.12, 24.94, 20.71, 18.81, 17.74, 16.43, 15.63, 15.23, 12.21
DEPT135° ($CDCl_3$)(δ_{ppm})	CH:	144.07, 134.97, 121.90, 120.24, 78.65, 44.96, 39.96, 39.03
	CH_2:	44.19, 29.71, 29.56, 27.47, 27.12, 24.94, 20.71
	CH_3:	28.95, 18.81, 17.74, 16.43, 15.43, 15.63, 15.23, 12.21
EIMS *m*/*z* (% relative intensity):		470 (6), 454 (12), 452 (26), 314 (35), 313 (100), 295 (73), 159 (69)

Fraction A5H3 Chromatogram characteristics on normal phase TLC with Toluene:$CHCl_3$:EtOAc:HCOOH in a ratio of 10:60:30:1 (2 runs) showed two UV-active spots with the R_f values of 0.25 and 0.45 and two brown spots under ASA reagent with the R_f values of 0.20 and 0.50. It (10.0 mg) was further purified by

precoated TLC with Toluene:$CHCl_3$:EtOAc:HCOOH in a ratio of 10:60:30:1 (4 runs) as a mobile phase afforded four bands.

Band 1 was obtained as a pale yellow gum in 2.9 mg. Chromatogram characteristics on normal phase TLC with Toluene:$CHCl_3$:EtOAc:HCOOH in a ratio of 10:60:30:1 (2 runs) showed one brown spot under ASA reagent with the R_f value of 0.50. Because its ^{1}H NMR data indicated the presence of many compounds, it was not further investigated.

Band 2 (SK12) was obtained as a pale yellow gum in 3.0 mg. Chromatogram characteristics on normal phase TLC with Toluene:$CHCl_3$:EtOAc: HCOOH in a ratio of 10:60:30:1 (2 runs) showed one UV-active spot with the R_f value of 0.45.

$[\alpha]_D^{26}$	-150.8º (c = 0.05, MeOH)
UVλ_{max}(nm)(MeOH)(log ε)	266 (3.74)
FTIR(neat):υ(cm^{-1})	3443 (OH stretching), 1681 (C=O stretching)
^{1}H NMR($CDCl_3$)(δ_{ppm})(500 MHz):	7.58 (*d*, *J* = 12.0 Hz, 1H), 6.14 (*d*, *J* = 11.5 Hz, 1H), 5.90 (*d*, *J* = 11.5 Hz, 1H), 5.27 (*brs*, 1H), 3.31 (*brs*, 1H), 3.14 (*dq*, *J* = 14.0 and 7.0 Hz, 1H), 2.30 (*dd*, *J* = 15.0 and 4.5 Hz, 1H), 2.25 (*m*, 1H), 1.94 (*m*, 1H), 1.93 (*m*, 1H), 1.87 (*s*, 3H), 1.85 (*m*, 2H), 1.80 (*m*, 1H), 1.78 (*m*, 2H), 1.56 (*m*, 1H), 1.51 (*m*, 2H), 1.50 (*m*, 1H), 1.38 (*m*, 2H), 1.12 (*m*, 1H), 1.03 (*s*, 3H), 0.90 (*s*, 3H), 0.86 (*d*, *J* = 7.0 Hz, 3H), 0.84 (*s*, 3H), 0.82 (*s*, 3H), 0.78 (*s*, 3H)
^{13}C NMR($CDCl_3$)(δ_{ppm})(125 MHz):	173.20, 153.23, 144.05, 134.94, 126.25, 121.88, 119.89, 76.10, 75.55, 53.72, 49.26, 44.18, 42.30, 39.16, 39.04, 37.52, 36.96, 29.51, 28.43, 27.55, 25.12, 23.55,

22.02, 20.71, 18.82, 17.71, 16.37, 15.63, 12.17

Band 3 was obtained as a pale yellow gum in 2.2 mg. Chromatogram characteristics on normal phase TLC with Toluene:$CHCl_3$:EtOAc:HCOOH in a ratio of 10:60:30:1 (2 runs) showed one UV-active spot with the R_f value of 0.25. Because its ^1H NMR data indicated the presence of many compounds, it was not further investigated.

Band 4 was obtained as a pale yellow gum in 1.4 mg. Chromatogram characteristics on normal phase TLC with Toluene:$CHCl_3$:EtOAc:HCOOH in a ratio of 10:60:30:1 (2 runs) showed one brown spot under ASA reagent with the R_f value of 0.20. It was not further investigated because its ^1H NMR data displayed many compounds.

Fraction A5H4 Chromatogram characteristics on normal phase TLC with 15%Acetonr/Petrol (2 runs) showed two brown spots under ASA reagent with the R_f values of 0.11 and 0.21. Because its ^1H NMR data indicated the presence of many compounds, it was not further studied.

Fraction A6 Chromatogram characteristics on reverse phase TLC with 50% MeOH/H_2O showed five UV-active spots with the R_f values of 0.09, 0.18, 0.23, 0.32 and 0.45. It was further purified by column chromatography over reverse phase C_{18} silica gel. Elution was conducted initially with 50%MeOH/H_2O, gradually enriched with methanol until pure methanol. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford eight fractions as shown in **Table 16**.

Table 16 Fractions obtained from the fraction **A6** by column chromatography over reverse phase C_{18} silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
A6A	50%MeOH/H_2O	9.6	Yellow gum
A6B	60-70%MeOH/H_2O	1.8	Yellow gum
A6C	70%MeOH/H_2O	4.8	Yellow gum

Table 16 (continued)

Fraction	Mobile phase	Weight (mg)	Physical appearance
A6D	70%MeOH/H_2O	18.8	Yellow gum
A6E	80-90%MeOH/H_2O	53.0	Yellow gum
A6F	90%MeOH/H_2O	16.7	Yellow gum
A6F	100%MeOH	70.2	Yellow gum
A6H	100%MeOH	17.8	Yellow gum

Fraction A6A Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 (2 runs) showed two UV-active spots with the R_f values of 0.12 and 0.27. It was not further investigated due to the presence of many compounds in the ^{1}H NMR spectrum.

Fraction A6B Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 (2 runs) showed two UV-active spots with the R_f values of 0.35 and 0.40. It was not further investigated because of the minute quantity.

Fraction A6C Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 (2 runs) showed two UV-active spots with the R_f values of 0.27 and 0.37. Further separation by column chromatography over Sephadex LH-20 was performed. Elution was conducted with 50%MeOH/CH_2Cl_2. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford three fractions as shown in **Table 17**.

Table 17 Fractions obtained from the fraction **A6C** by column chromatography over Sephadex LH-20

Fraction	Weight (mg)	Physical appearance
A6C1	1.1	Pale yellow gum
A6C2	1.2	Pale yellow gum
A6C3	2.1	Pale yellow gum

Fraction A6C1 Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 (2 runs) showed one UV-active spot with the R_f value of 0.35. Because its ^{1}H NMR data indicated the presence of many compounds, it was not further investigated.

Fraction A6C2 (**SK22**) Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 (2 runs) showed one UV-active spot with the R_f value of 0.25.

UVλ_{max}(nm)(MeOH)(log ε)		235 (3.47), 258 (3.91), 310 (1.83), 369 (1.18)
FTIR(neat):υ(cm^{-1})		3343 (OH stretching), 1641 (C=O stretching)
^{1}H NMR(Acetone-d_6)(δ_{ppm})(500 MHz):		12.87 (*s*, 1H), 7.52 (*s*, 1H), 7.19 (*s*, 1H), 6.29 (*s*, 1H), 4.07 (*s*, 3H), 3.91 (*s*, 3H)
^{13}C NMR(Acetone-d_6)(δ_{ppm})(125 MHz):		180.68, 159.44, 158.73, 155.75, 152.39, 150.80, 145.36, 128.47, 114.29, 108.91, 103.17, 100.76, 98.65, 61.71, 57.03
DEPT135° (Acetone-d_6)(δ_{ppm})	CH:	108.91, 100.76, 98.65
	CH_3:	61.71, 57.03
EIMS *m*/*z* (% relative intensity):		304 (60), 289 (100), 261 (59), 259 (32), 231 (29)

Fraction A6C3 Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 (2 runs) showed no definite spot under UV-S. It was not further investigated.

Fraction A6D Chromatogram characteristics on reverse phase TLC with 60% MeOH/H_2O showed three UV-active spots with the R_f values of 0.15, 0.27 and 0.42. It was further purification by column chromatography over reverse phase C_{18} silica gel. Elution was conducted initially with 60%MeOH/H_2O, gradually enriched with methanol until pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford six fractions. They were not further investigated because their chromatograms on normal

phase TLC using 3%MeOH/CH_2Cl_2 showed many spots under ASA reagent and they were obtained in low quantity.

Fraction A6E Chromatogram characteristics on normal phase TLC with 20%EtOAc/Petrol (2 runs) showed five UV-active spots with the R_f values of 0.23, 0.33, 0.38, 0.47 and 0.59. It was further separated by column chromatography over silica gel. Elution was conducted initially with 20%EtOAc/Petrol, gradually enriched with ethyl acetate until pure ethyl acetate. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford eight fractions as shown in **Table 18**.

Table 18 Fractions obtained from the fraction **A6E** by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
A6E1	20%EtOAc/Petrol	4.7	Pale yellow gum
A6E2	30%EtOAc/Petrol	1.5	Pale yellow gum
A6E3	30%EtOAc/Petrol	2.1	Pale yellow gum
A6E4	30-40%EtOAc/Petrol	5.5	Yellow gum
A6E5	40%EtOAc/Petrol	3.5	Yellow gum
A6E6	40-60%EtOAc/Petrol	14.6	Yellow gum
A6E7	80%EtOAc/Petrol	20.0	Yellow gum
A6E8	100%EtOAc	19.5	Yellow gum

Fraction A6E1 Chromatogram characteristics on normal phase TLC with 20%EtOAc/Petrol (2 runs) showed one brown spot under ASA reagent with the R_f value of 0.47. Its ^1H NMR spectrum displayed proton signals in the high field region. Thus, it was not further investigated.

Fraction A6E2 Chromatogram characteristics on normal phase TLC with 20%EtOAc/Petrol (2 runs) showed two brown spots under ASA reagent with the R_f values of 0.47 and 0.52. Because of the minute quantity, it was not further investigated.

Fraction A6E3 Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.38 and 0.45. Because of the minute quantity, it was not further investigated.

Fraction A6E4 Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 (2 runs) showed two UV-active spots with the R_f values of 0.33 and 0.45. Further purification by precoated TLC with 1%MeOH/CH_2Cl_2 (4 runs) as a mobile phase afforded two bands.

Band 1 (SK10) was obtained as a pale yellow gum in 1.5 mg. Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 (2 runs) showed one UV-active spot with the R_f value of 0.45.

UVλ_{max}(nm)(MeOH)(log ε)	249 (3.52), 269 (2.27), 273 (2.04), 329 (1.68)
FTIR(neat):υ(cm^{-1})	3417 (OH stretching), 1676 (C=O stretching)
^{1}H NMR($CDCl_3$)(δ_{ppm})(500 MHz):	12.01 (*s*, 1H), 11.29 (*s*, 1H), 7.63 (*d*, *J* = 2.0 Hz, 1H), 7.35 (*d*, *J* = 9.0 Hz, 1H), 7.05 (*dd*, *J* = 2.0 and 1.0 Hz, 1H), 6.97 (*d*, *J* = 1.0 Hz, 1H), 6.79 (*d*, *J* = 9.0 Hz, 1H)
^{13}C NMR($CDCl_3$)(δ_{ppm})(125 MHz):	185.00, 162.20, 159.45, 154.30, 148.00, 144.72, 144.20, 135.00, 123.95, 110.98, 110.50, 108.00, 103.50, 102.65, 95.44
DEPT135° ($CDCl_3$)(δ_{ppm}) CH:	144.72, 123.95, 110.98, 103.50, 95.44
EIMS *m*/*z* (% relative intensity):	284 (100), 268 (7), 255 (4), 228 (5)

Band 2 (SK16) was obtained as a pale yellow gum in 1.3 mg. Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 (2 runs) showed one UV-active spot with the R_f value of 0.33.

UVλ_{max}(nm)(MeOH)(log ε)	242 (4.00), 261 (3.88), 322 (2.93), 330 (3.03)

FTIR(neat):υ(cm^{-1})		3369 (OH stretching), 1648 (C=O stretching)
^{1}H NMR(Acetone-d_6)(δ_{ppm})(300 MHz):		13.38 (*s*, 1H), 8.03 (*d*, *J* = 10.2 Hz, 1H), 6.82 (*s*, 1H), 6.34 (*d*, *J* = 2.1 Hz, 1H), 6.20 (*d*, *J* = 2.1 Hz, 1H), 5.94 (*d*, *J* = 10.2 Hz, 1H), 1.45 (*s*, 6H)
^{13}C NMR(Acetone-d_6)(δ_{ppm})(75 MHz):		183.12, 165.67, 164.76, 158.22, 154.08, 153.80, 138.93, 133.64, 121.49, 120.95, 108.50, 103.90, 103.50, 98.78, 93.95, 76.77, 27.16
DEPT135° (Acetone-d_6)(δ_{ppm})	CH:	133.64, 121.49, 103.90, 98.78, 93.95
	CH_3:	27.16

Fraction A6E5 Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 (2 runs) showed two UV-active spots with the R_f values of 0.33 and 0.47. Further purification by precoated TLC with 1%MeOH/CH_2Cl_2 (4 runs) as a mobile phase afforded two bands.

Band 1 was obtained as a pale yellow gum in 1.2 mg. Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 (2 runs) showed one UV-active spot with the R_f value of 0.47. Its ^{1}H NMR spectrum showed the absence of aromatic and olefinic protons. Because of the minute quantity, it was not further investigated.

Band 2 was obtained as a pale yellow gum in 1.5 mg. Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 (2 runs) showed one UV-active spot with the R_f value of 0.33. Its ^{1}H NMR data indicated the presence of **SK16** as a major component. Further investigation was then not carried out.

Fraction A6E6 Chromatogram characteristics on normal phase TLC with 20%EtOAc/Petrol (2 runs) showed two UV-active spots with the R_f values of 0.38 and 0.42 and one brown spot under ASA reagent with the R_f value of 0.50. Its ^{1}H NMR spectrum displayed proton signals in the high field region. Thus, it was not further investigated.

Fraction A6E7 Chromatogram characteristics on normal phase TLC with 20%EtOAc/Petrol (2 runs) showed four UV-active spots with the R_f values of 0.11, 0.19, 0.23, 0.42 and 0.47. Its ^{1}H NMR spectrum displayed proton signals in the high field region. Thus, it was not further investigated.

Fraction A6E8 Chromatogram characteristics on normal phase TLC with 20%EtOAc/Petrol (2 runs) showed three UV-active spots with the R_f values of 0.04, 0.11 and 0.35. Its ^{1}H NMR spectrum showed the absence of aromatic and olefinic protons. It was not further investigated.

Fraction A6F Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 (2 runs) showed three UV-active spots with the R_f values of 0.27, 0.52 and 0.72. It was separated into two fractions by dissolving in dichloromethane. The dichloromethane soluble fraction (8.5 mg) was obtained as a green yellow gum. Its ^{1}H NMR spectrum displayed proton signals in the high field region. Therefore, it was not further investigated. The dichloromethane insoluble fraction (8.2 mg) was obtained as a yellow gum. Chromatogram characteristics on normal phase TLC with 100%CH_2Cl_2 (2 runs) showed two UV-active spots with the R_f values of 0.20 and 0.50. Further purification by precoated TLC with 100%CH_2Cl_2 (4 runs) as a mobile phase afforded two bands. They were not further investigated. Because their chromatograms on normal phase TLC using 100%CH_2Cl_2 (2 runs) showed many spots under UV-S and they were obtained in low quantity. Moreover, their ^{1}H NMR spectra displayed many compounds.

Fraction A6G Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 showed four UV-active spots with the R_f values of 0.11, 0.28, 0.38 and 0.76 and two purple spots under ASA reagent with the R_f values of 0.90 and 0.95. It was further separated by column chromatography over silica gel. Elution was conducted initially with pure dichloromethane, gradually enriched with methanol until pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford four fractions as shown in **Table 19**.

Table 19 Fractions obtained from the fraction **A6G** by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
A6G1	100%CH_2Cl_2-3%MeOH/CH_2Cl_2	14.4	Colorless gum
A6G2	5%MeOH/CH_2Cl_2	20.2	Yellow gum
A6G3	5-80%MeOH/CH_2Cl_2	26.0	Yellow gum
A6G4	100%MeOH	9.1	Brown gum

Fraction A6G1 Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 showed three purple spots under ASA reagent with the R_f values of 0.76, 0.90 and 0.95. Its ^{1}H NMR spectrum displayed proton signals in the high field region. Thus, it was not further investigated.

Fraction A6G2 Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 showed four UV-active spots with the R_f values of 0.19, 0.28, 0.38 and 0.76. Its ^{1}H NMR data indicated the presence of **SK2** and **SK3** as major components. Further investigation was then not carried out.

Fraction A6G3 Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.04 and 0.11. Because its ^{1}H NMR data indicated the presence of many compounds, it was not further investigated.

Fraction A6G4 Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 showed none of well separated spots under UV-S and ASA reagent. Thus, it was not further investigated.

Fraction A6H Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 (2 runs) showed no definite spot under UV-S and ASA reagent. It was not further investigated.

Fraction A7 Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 showed no definite spot under UV-S and ASA reagent. Its ^{1}H NMR spectrum showed the absence of aromatic and olefinic protons. It was not further investigated.

Fraction B Upon standing at room temperature, a white solid (1.03 g) precipitated. Its chromatogram on normal phase TLC with 60%CH_2Cl_2/Petrol showed one major purple under ASA reagent with the R_f value of 0.33. Its ^1H NMR data indicated the presence of friedelin as a major component.

The filtrate became a yellow brown gum (10.54 g) after evaporation to dryness under reduced pressure. Chromatogram characteristics on normal phase TLC with 100%CH_2Cl_2 showed five UV-active spots with the R_f values of 0.18, 0.25, 0.46, 0.72 and 0.73. Further separation by column chromatography over Sephadex LH-20 was performed. Elution was conducted with 100%MeOH. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford seven fractions as shown in **Table 20**.

Table 20 Fractions obtained from the fraction **B** by column chromatography over Sephadex LH-20

Fraction	Weight (mg)	Physical appearance
B1	11157.9	Green yellow gum
B2	9398.0	Green yellow gum
B3	526.4	Yellow solid
B4	56.4	Yellow gum
B5	46.1	Yellow gum
B6	22.3	Yellow gum
B7	37.1	Brown yellow gum

Fraction B1 Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 showed none of well separated spots under UV-S. Its ^1H NMR data indicated the presence of long chain hydrocarbons. Thus, it was not further investigated.

Fraction B2 Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 showed three UV-active spots with the R_f values of 0.20, 0.25 and 0.37. It was further separated by column chromatography over silica gel. Elution was conducted initially with pure dichloromethane, gradually enriched with methanol and

finally with pure methanol. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford seven fractions as shown in **Table 21**.

Table 21 Fractions obtained from the fraction **B2** by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
B2A	100%CH_2Cl_2	2160. 8	Green yellow gum
B2B	1%MeOH/CH_2Cl_2	338.0	Yellow solid
B2C	2%MeOH/CH_2Cl_2	1434.7	Green yellow gum
B2D	3-5%MeOH/CH_2Cl_2	4100.4	Green yellow gum
B2E	5%MeOH/CH_2Cl_2	549.3	Yellow gum
B2F	7-40%MeOH/CH_2Cl_2	320.5	Yellow gum
B2G	60%MeOH/CH_2Cl_2-100%MeOH	149.1	Yellow gum

Fraction B2A Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 (5 runs) showed four UV-active spots with the R_f values of 0.07, 0.30, 0.52 and 0.95. Its ^1H NMR data indicated the presence of long chain hydrocarbons. Thus, it was not further investigated.

Fraction B2B Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 (5 runs) showed three UV-active spots with the R_f values of 0.07, 0.37 and 0.57. It was further separated by column chromatography over silica gel. Elution was conducted initially with pure dichloromethane, gradually enriched with methanol and finally with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford seven fractions as shown in **Table 22**.

Table 22 Fractions obtained from the fraction **B2B** by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
B2B1	100%CH_2Cl_2	5.7	Colorless gum
B2B2	1-2%MeOH/CH_2Cl_2	77.3	Yellow gum
B2B3	3-5%MeOH/CH_2Cl_2	23.3	Yellow gum
B2B4	5%MeOH/CH_2Cl_2	25.6	Yellow solid
B2B5	7-15%MeOH/CH_2Cl_2	73.8	Yellow solid
B2B6	20-60%MeOH/CH_2Cl_2	64.8	Yellow gum
B2B7	80%MeOH/CH_2Cl_2-100%MeOH	21.6	Yellow gum

Fraction B2B1 Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 (3 runs) showed one UV-active spot with the R_f value of 0.46 and two brown spots under ASA reagent with the R_f values of 0.12 and 0.18. Its ^{1}H NMR data indicated the presence of long chain hydrocarbons. Thus, it was not further investigated.

Fraction B2B2 Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 (3 runs) showed two UV-active spots with the R_f values of 0.42 and 0.48. Its ^{1}H NMR data indicated the presence of long chain hydrocarbons. Thus, it was not further investigated.

Fraction B2B3 Chromatogram characteristics on normal phase TLC with 20%Acetone/Petrol (2 runs) showed three UV-active spots with the R_f values of 0.10, 0.28 and 0.48. It was further separated by column chromatography over silica gel. Elution was conducted initially with 20%Acetone/Petrol, gradually enriched with acetone and finally with pure acetone then enriched with methanol and finally with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford six fractions as shown in **Table 23**.

Table 23 Fractions obtained from the fraction **B2B3** by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
B2B3A	20%Acetone/Petrol	4.8	Colorless gum
B2B3B	30%Acetone/Petrol	16.1	Colorless gum
B2B3C	40%Acetone/Petrol	4.6	Colorless gum
B2B3D	60%Acetone/Petrol	6.0	Colorless gum
B2B3E	80%Acetone/Petrol-100%Acetone	6.1	Colorless gum
B2B3F	100%Acetone-100%MeOH	9.3	Yellow gum

Fraction B2B3A Chromatogram characteristics on normal phase TLC with 20%Acetone/Petrol (2 runs) showed none of well separated spots under UV-S. Thus, it was not further investigated.

Fraction B2B3B Chromatogram characteristics on normal phase TLC with 20%Acetone/Petrol (2 runs) showed two UV-active spots with the R_f values of 0.25 and 0.37. Its ^{1}H NMR data indicated the presence of **SK2** and **SK3** as major components. Further investigation was then not carried out.

Fraction B2B3C Chromatogram characteristics on normal phase TLC with 20%Acetone/Petrol (2 runs) showed two UV-active spots with the R_f values of 0.20 and 0.32. Because of the low quantity, it was not further investigated.

Fraction B2B3D Chromatogram characteristics on normal phase TLC with 20%Acetone/Petrol (2 runs) showed one UV-active spots with the R_f value of 0.15. Because its ^{1}H NMR data indicated the presence of many compounds, it was not further investigated.

Fraction B2B3E Chromatogram characteristics on normal phase TLC with 40%EtOAc/Petrol (4 runs) showed two UV-active spots with the R_f values of 0.52 and 0.54. Further purification by precoated TLC with 40%EtOAc/Petrol (8 runs) as a mobile phase afforded two bands. They were not further investigated because their chromatograms on normal phase TLC using 40%EtOAc/Petrol showed many spots

under UV-S and they were obtained in low quantity. Moreover, their ^{1}H NMR spectra displayed many compounds.

Fraction B2B3F Chromatogram characteristics on normal phase TLC with 20%Acetone/Petrol (2 runs) none of well separated spots under UV-S. Thus, it was not further investigated.

Fraction B2B4 Chromatogram characteristics on normal phase TLC with 30%Acetone/Petrol showed three UV-active spots with the R_f values of 0.45, 0.52 and 0.55. It was further separated by column chromatography over silica gel. Elution was conducted initially with 30%Acetone/CH_2Cl_2, gradually enriched with acetone and finally with pure acetone then enriched with methanol and finally with pure methanol. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford six fractions. They were not further investigated because their chromatograms on normal phase TLC using 30%Acetone/Petrol showed many spots under ASA reagent and they were obtained in low quantity. Moreover, their ^{1}H NMR spectra displayed proton signals in the high field region.

Fraction B2B5 Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 (3 runs) showed three UV-active spots with the R_f values of 0.09, 0.23 and 0.28. Its ^{1}H NMR data indicated the presence of **SK2** and **SK3** as major components. Further investigation was then not carried out.

Fraction B2B6 Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.23 and 0.35. Its ^{1}H NMR data indicated the presence of **SK3** as a major component. Further investigation was then not carried out.

Fraction B2B7 Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.12 and 0.18. Its ^{1}H NMR spectrum showed broad signals. Thus, it was not further studied.

Fraction B2C Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 showed none of well separated spots under UV-S. It was not further investigated.

Fraction B2D Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 (5 runs) showed two UV-active spots with the R_f values of 0.15

and 0.30. Its ^{1}H NMR data indicated the presence of **SK2** and **SK3** as major components. Further investigation was then not carried out.

Fraction B2E Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 (5 runs) showed two UV-active spots with the R_f values of 0.12 and 0.25. It was further separated by column chromatography over silica gel. Elution was conducted initially with pure dichloromethane, gradually enriched with methanol and finally with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford five fractions as shown in **Table 24**.

Table 24 Fractions obtained from the fraction **B2E** by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
B2E1	100%CH_2Cl_2	40.5	Yellow gum
B2E2	1-7%MeOH/CH_2Cl_2	153.2	Yellow gum
B2E3	7%MeOH/CH_2Cl_2	26.6	Yellow gum
B2E4	7-60%MeOH/CH_2Cl_2	174.4	Yellow gum
B2E5	60%MeOH/CH_2Cl_2-100%MeOH	35.1	Yellow gum

Fraction B2E1 Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.07 and 0.13. Its ^{1}H NMR spectrum displayed proton signals in the high field region. Thus, it was not further investigated.

Fraction B2E2 Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 showed five UV-active spots with the R_f values of 0.20, 0.25, 0.30, 0.35 and 0.40. Its ^{1}H NMR data indicated the presence of **SK2** and **SK3** as major components. Further investigation was then not carried out.

Fraction B2E3 Chromatogram characteristics on normal phase TLC with 30%Acetone/Petrol (2 runs) showed three UV-active spots with the R_f values of 0.37, 0.45 and 0.50 and two brown spots under ASA reagent with the R_f values of 0.20 and

0.30. This fraction was further separated by column chromatography over silica gel. Elution was conducted initially with 30%Acetone/Petrol, gradually enriched with acetone and finally with pure acetone. Fractions the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford five fractions as shown in **Table 25**.

Table 25 Fractions obtained from the fraction **B2E3** by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
B2E3A	30%Acetone/Petrol	1.6	Colorless gum
B2E3B	30%Acetone/Petrol	3.4	Colorless gum
B2E3C	30%Acetone/Petrol	4.7	Colorless gum
B2E3D	60%Acetone/Petrol	4.0	Colorless gum
B2E3E	80%Acetone/Petrol-100%Acetone	5.7	Colorless gum

Fraction B2E3A Chromatogram characteristics on normal phase TLC with 30%Acetone/Petrol (2 runs) showed two purple spots under with the R_f values of 0.30 and 0.45. Because of the minute quantity, it was not further investigated.

Fraction B2E3B Chromatogram characteristics on normal phase TLC with 30%Acetone/Petrol (2 runs) showed two brown spots with the R_f values of 0.20 and 0.30. Because of the minute quantity, it was not further investigated.

Fraction B2E3C Chromatogram characteristics on normal phase TLC with 30%Acetone/Petrol (2 runs) showed one UV-active spot with the R_f value of 0.50 and two brown spots with the R_f values of 0.20 and 0.30. Because of low quantity, it was not further investigated.

Fraction B2E3D (**SK21**) Chromatogram characteristics on normal phase TLC with 25%Acetone/Petrol (10 runs) showed one UV-active spot with the R_f value of 0.48. Further purification by precoated TLC with 25%Acetone/Petrol (18 runs) as a mobile phase gave a colorless gum in 2.3 mg. Chromatogram characteristics on

normal phase TLC with 25%Acetone/Petrol (10 runs) showed one UV-active spot with the R_f value of 0.48.

$[\alpha]_D^{27}$		-62.2º (c = 0.04, MeOH)
UV λ_{max}(nm)(MeOH)(log ε)		258 (2.46)
FTIR(neat):υ(cm^{-1})		3443 (OH stretching), 1698, 1742 (C=O stretching)
^{1}H NMR($CDCl_3$)(δ_{ppm})(500 MHz):		6.70 (*qd*, *J* = 8.0 and 1.0 Hz, 1H), 4.57 (*td*, *J* = 10.5 and 2.0 Hz, 1H), 4.09 (*ddd*, *J* = 15.0, 4.0 and 2.0 Hz, 1H), 3.76 (*s*, 3H), 3.41 (*t*, *J* = 2.5 Hz, 1H), 2.39 (*d*, *J* = 18.5 Hz, 1H), 2.31 (*m*, 1H), 2.26 (*m*, 1H), 2.20 (*m*, 2H), 2.18 (*m*, 1H), 2.09 (*d*, *J* = 18.5 Hz, 1H), 1.94 (*m*, 2H), 1.87 (*d*, *J* = 1.5 Hz, 3H), 1.79 (*m*, 1H), 1.75 (*m*, 1H), 1.70 (*m*, 1H), 1.66 (*m*, 1H), 1.57 (*m*, 1H), 1.32 (*m*, 1H), 1.28 (*m*, 1H), 1.21 (*s*, 3H), 1.10 (*m*, 1H), 1.00 (*s*, 3H), 0.95 (*d*, *J* = 7.0 Hz, 3H), 0.92 (*s*, 3H), 0.87 (*s*, 3H), 0.86 (*s*, 3H)
^{13}C NMR($CDCl_3$)(δ_{ppm})(125 MHz):		207.75, 168.32, 151.65, 143.91, 140.18, 127.52, 75.67, 74.93, 66.61, 52.35, 52.01, 45.99, 44.58, 44.41, 39.66, 39.24, 37.80, 33.36, 32.82, 29.91, 28.75, 25.38, 24.79, 24.05, 22.55, 22.12, 21.11, 17.46, 16.80, 15.40, 12.77
DEPT135° ($CDCl_3$)(δ_{ppm})	CH:	143.91, 75.67, 66.61, 39.66, 33.36
	CH_2:	52.35, 39.24, 32.82, 29.91, 25.38, 24.79, 24.05, 22.12
	CH_3:	52.01, 28.75, 22.55, 21.11, 17.46, 16.80, 15.40, 12.77

EIMS *m*/*z* (% relative intensity): 516 (90), 498 (35), 313 (15), 191 (81), 121 (37)

Fraction B2E3E Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 (2 runs) showed no definite spot under UV-S. Thus, it was not further investigated.

Fraction B2E4 Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 (2 runs) showed four UV-active spots with the R_f values of 0.25, 0.35, 0.37 and 0.42. Its ^{1}H NMR spectrum showed broad signals. Thus, it was not further studied.

Fraction B2E5 Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 (2 runs) showed no definite spot under UV-S and ASA reagent. Thus, it was not further studied.

Fraction B2F Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 (5 runs) showed two UV-active spots with the R_f values of 0.07 and 0.12. Its ^{1}H NMR data indicated the presence of **SK3** as a major component. Further investigation was then not carried out.

Fraction B2G Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 (2 runs) showed no definite spot under UV-S. Further investigation was then not carried out.

Fraction B3 Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.12 and 0.35 and two brown spots under ASA reagent with the R_f values of 0.32 and 0.37. It was separated by column chromatography over Sephadex LH-20. Elution was conducted with 50%MeOH/CH_2Cl_2. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford six fractions as shown in **Table 26**.

Table 26 Fractions obtained from the fraction **B3** by column chromatography over Sephadex LH-20

Fraction	Weight (mg)	Physical appearance
B3A	69.3	Green yellow gum
B3B	215.1	Yellow solid
B3C	213.3	Yellow solid
B3D	104.2	Yellow gum
B3E	32.4	Yellow gum
B3F	40.2	Yellow gum

Fraction B3A Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 (2 runs) showed none of well separated spots under UV-S. Its ^1H NMR spectrum displayed proton signals in the high field region, it was not further investigated.

Fraction B3B Chromatogram characteristics on normal phase TLC with 10%Acetone/CH_2Cl_2 (2 runs) showed four UV-active spots with the R_f values of 0.12, 0.22, 0.32 and 0.37 and one brown spot under ASA reagent with the R_f value of 0.24. It was further separated by column chromatography over silica gel. Elution was conducted initially with 10%Acetone/CH_2Cl_2, gradually enriched with acetone and finally with pure acetone then enriched with methanol and finally with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford eight fractions as shown in **Table 27**.

Table 27 Fractions obtained from the fraction **B3B** by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
B3B1	10%Acetone/CH_2Cl_2	68.1	Yellow gum
B3B2	10%Acetone/CH_2Cl_2	34.2	Yellow solid

Table 27 (continued)

Fraction	Mobile phase	Weight (mg)	Physical appearance
B3B3	20-30%Acetone/CH_2Cl_2	156.3	Yellow solid
B3B4	30%Acetone/CH_2Cl_2	128.2	Yellow solid
B3B5	40%Acetone/CH_2Cl_2	82.3	Yellow solid
B3B6	50-60%Acetone/CH_2Cl_2	72.1	Yellow gum
B3B7	60-80%Acetone/CH_2Cl_2-40%MeOH/Acetone	78.6	Yellow gum
B3B8	60%MeOH/Acetone-100%MeOH	102.0	Yellow gum

Fraction B3B1 Chromatogram characteristics on normal phase TLC with 50%CH_2Cl_2/Petrol showed three UV-active spots with the R_f values of 0.17, 0.27 and 0.25 and two purple spots under ASA reagent with the R_f values of 0.42 and 0.55. It was further separated by column chromatography over silica gel. Elution was conducted initially with 50%CH_2Cl_2/Petrol, gradually enriched with dichloromethane and finally with pure dichloromethane. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford ten fractions. They were not further investigated because their chromatograms on normal phase TLC using 50%CH_2Cl_2/Petrol showed many spots under ASA reagent and they were obtained in low quantity. Moreover, their ^{1}H NMR data indicated the presence of friedelin as a major component.

Fraction B3B2 Chromatogram characteristics on normal phase TLC with 15%Acetone/Petrol showed one UV-active spot with the R_f value of 0.27 and three brown spots under ASA reagent with the R_f values of 0.12, 0.60 and 0.75. Its ^{1}H NMR spectrum displayed proton signal in the high field region, it was not further investigated.

Fraction B3B3 Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.23 and 0.35 and three brown spots under ASA reagent with the R_f values of 0.55, 0.62 and 0.72. It was further separated by column chromatography over silica gel. Elution was

conducted initially with 1%MeOH/CH_2Cl_2, gradually enriched with methanol and finally with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford seven fractions as shown in **Table 28**.

Table 28 Fractions obtained from the fraction **B3B3** by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
B3B3A	1%MeOH/CH_2Cl_2	11.5	Yellow gum
B3B3B	2%MeOH/CH_2Cl_2	30.1	Pale yellow solid
B3B3C	5%MeOH/CH_2Cl_2	10.5	Pale yellow solid
B3B3D	5%MeOH/CH_2Cl_2	62.1	Pale yellow solid
B3B3E	7-15%MeOH/CH_2Cl_2	47.2	Pale yellow solid
B3B3F	20-80%MeOH/CH_2Cl_2	9.1	Pale yellow gum
B3B3G	100%MeOH	15.1	Yellow gum

Fraction B3B3A Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 showed none of well separated spots under UV-S. Its ^{1}H NMR spectrum displayed proton signals in the high field region, it was not further investigated.

Fraction B3B3B Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.42 and 0.50 and one spot under ASA reagent with the R_f value of 0.62. It was further separated by column chromatography over silica gel. Elution was conducted initially with 1%MeOH/CH_2Cl_2, gradually enriched with methanol and finally with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford four fractions as shown in **Table 29**.

Table 29 Fractions obtained from the fraction **B3B3B** by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
B3B3B1	1%MeOH/CH_2Cl_2	4.2	Colorless gum
B3B3B2	1%MeOH/CH_2Cl_2	8.3	Colorless gum
B3B3B3	2%MeOH/CH_2Cl_2	9.1	Yellow gum
B3B3B4	5%MeOH/CH_2Cl_2-100%MeOH	9.1	Yellow gum

Fraction B3B3B1 Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 (2 runs) showed two UV-active spots with the R_f values of 0.50 and 0.52 and two purple spots under ASA reagent with the R_f values of 0.32 and 0.62. Because of the low quantity, it was not further investigated.

Fraction B3B3B2 Chromatogram characteristics on normal phase TLC with 15%Acetone/Petrol (5 runs) showed one UV-active spot with the R_f value of 0.27 and one brown spot under ASA reagent with the R_f value of 0.29. Further purification by precoated TLC with 15%Acetone/Petrol (10 runs) as a mobile phase afforded a colorless gum in 5.7 mg. Chromatogram characteristics on normal phase TLC with 15%Acetone/Petrol (5 runs) showed one UV-active spot with the R_f value of 0.27. Because its ^{1}H NMR data indicated the presence of many compounds, it was not further investigated.

Fraction B3B3B3 Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 (2 runs) showed one UV-active spot with the R_f value of 0.45 and two brown spots under ASA reagent with the R_f values of 0.77 and 0.87. Its ^{1}H NMR spectrum displayed proton signals in the high field region, it was not further investigated.

Fraction B3B3B4 Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 (2 runs) showed none of well separated spots under UV-S. The ^{1}H NMR spectrum indicated the presence of proton signals in the high field region, it was not further investigated.

Fraction B3B3C Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.42 and 0.50

and one brown spot under ASA reagent with the R_f value of 0.62. Its ^1H NMR data were similar to those of fraction **B3B3B**. Thus, it was further investigated.

Fraction B3B3D Chromatogram characteristics on normal phase TLC with 25%Acetone/Petrol showed four UV-active spots with the R_f values of 0.25, 0.35, 0.45 and 0.52. It was further separated by column chromatography over silica gel. Elution was conducted initially with 25%Acetone/Petrol, gradually enriched with acetone and finally with pure acetone then enriched with methanol and finally with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford five fractions as shown in **Table 30**.

Table 30 Fractions obtained from the fraction **B3B3D** by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
B3B3D1	25%Acetone/Petrol	27.0	Colorless gum
B3B3D2	25%Acetone/Petrol	17.1	Colorless gum
B3B3D3	25%Acetone/Petrol	20.2	Colorless gum
B3B3D4	30%Acetone/Petrol-100%Acetone	6.1	Colorless gum
B3B3D5	100%Acetone-100%MeOH	3.5	Yellow gum

Fraction B3B3D1 Chromatogram characteristics on normal phase TLC with 25%Acetone/Petrol showed two UV-active spots with the R_f values of 0.45 and 0.50. Its ^1H NMR data indicated the presence of **SK2** and **SK3** as major components. Further investigation was then not carried out.

Fraction B3B3D2 Chromatogram characteristics on normal phase TLC with 25%Acetone/Petrol showed one UV-active spot with the R_f value of 0.45. Its ^1H NMR data indicated the presence of **SK3** as a major component. Further investigation was then not carried out.

Fraction B3B3D3 Chromatogram characteristics on normal phase TLC with 5%MeOH/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.45 and 0.50.

Its ^{1}H NMR data indicated the presence of **SK2** and **SK3** as major components. Further investigation was then not carried out.

Fraction B3B3D4 Chromatogram characteristics on normal phase TLC with 25%Acetone/Petrol showed three UV-active spots with the R_f values of 0.25, 0.35 and 0.52. Its ^{1}H NMR spectrum showed the absence of aromatic and olefinic protons. Thus, it was not further investigated.

Fraction B3B3D5 Chromatogram characteristics on normal phase TLC with 25%Acetone/Petrol showed no definite spot under UV-S. It was not further investigated.

Fraction B3B3E Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.23 and 0.25. Its ^{1}H NMR data indicated the presence of **SK2** and **SK3** as major components. Further investigation was then not carried out.

Fraction B3B3F Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 showed one UV-active spot with the R_f value of 0.20 and one purple spot with the R_f value of 0.70. Because its ^{1}H NMR data indicated the presence of many compounds, it was not further investigated.

Fraction B3B3G Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.12 and 0.20 and one purple spot under ASA reagent with the R_f value of 0.72. Because its ^{1}H NMR data indicated the presence of many compounds, it was not further investigated.

Fraction B3B4 Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.23 and 0.35 and three brown spots under ASA reagent with the R_f values of 0.55, 0.62 and 0.67. It was further separated by column chromatography over silica gel. Elution was conducted initially with 1%MeOH/CH_2Cl_2, gradually enriched with methanol and finally with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford five fractions as shown in **Table 31**.

Table 31 Fractions obtained from the fraction **B3B4** by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
B3B4A	1-5%MeOH/CH_2Cl_2	48.2	Pale yellow gum
B3B4B	5%MeOH/CH_2Cl_2	49.2	Yellow gum
B3B4C	5-7%MeOH/CH_2Cl_2	17.5	Yellow gum
B3B4D	10-60%MeOH/CH_2Cl_2	17.3	Yellow gum
B3B4E	100%MeOH	10.4	Yellow gum

Fraction B3B4A Chromatogram characteristics on normal phase TLC with 25%Acetone/Petrol showed two UV-active spots with the R_f values of 0.20 and 0.37 and one brown spot under ASA reagent with the R_f value of 0.37. It was further separated by column chromatography over silica gel. Elution was conducted initially with 25%Acetone/Petrol, gradually enriched with acetone and finally with pure acetone then enriched with methanol and finally with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford four fractions as shown in **Table 32**.

Table 32 Fractions obtained from the fraction **B3B4A** by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
B3B4A1	25%Acetone/Petrol	4.1	Colorless gum
B3B4A2	25%Acetone/Petrol	2.0	Colorless gum
B3B4A3	25-60%Acetone/Petrol	21.2	Pale yellow gum
B3B4A4	60%Acetone/Petrol-100%MeOH	18.0	Pale yellow gum

Fraction B3B4A1 Chromatogram characteristics on normal phase TLC with 25%Acetone/Petrol showed none of well separated spots under UV-S. It was not further investigated.

Fraction B3B4A2 Chromatogram characteristics on normal phase TLC with 25%Acetone/Petrol showed two UV-active spots with the R_f values of 0.37 and 0.45. Because of the minute quantity, it was not further investigated.

Fraction B3B4A3 Chromatogram characteristics on normal phase TLC with 25%Acetone/Petrol showed two UV-active spots with the R_f values of 0.20 and 0.25. Its ^{1}H NMR data indicated the presence of **SK2** and **SK3** as major components. Further investigation was then not carried out.

Fraction B3B4A4 Chromatogram characteristics on normal phase TLC with 25%Acetone/Petrol showed none of well separated spots under UV-S. Its ^{1}H NMR spectrum showed the absence of aromatic and olefinic protons. Thus, it was not further investigated.

Fraction B3B4B Chromatogram characteristics on normal phase TLC with 25%Acetone/Petrol showed three UV-active spots with the R_f values of 0.20, 0.25 and 0.35 and one brown spot under ASA reagent with the R_f value of 0.37. It was further separated by column chromatography over silica gel. Elution was conducted initially with 30%Acetone/Petrol, gradually enriched with acetone and finally with pure acetone then enriched with methanol and finally with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford four fractions as shown in **Table 33**.

Table 33 Fractions obtained from the fraction **B3B4B** by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
B3B4B1	30%Acetone/Petrol	9.5	Colorless gum
B3B4B2	30-60%Acetone/Petrol	10.1	Pale yellow gum
B3B4B3	60%Acetone/Petrol	10.5	Pale yellow gum
B3B4B4	80%Acetone/Petrol-100%MeOH	25.0	Pale yellow gum

Fraction B3B4B1 Chromatogram characteristics on normal phase TLC with 20%Acetone/Petrol showed three brown spots under ASA reagent with the R_f values

of 0.37, 0.62 and 0.77. Its ^{1}H NMR spectrum indicated the presence of proton signals in the high field region, it was not further investigated.

Fraction B3B4B2 Chromatogram characteristics on normal phase TLC with 20%Acetone/Petrol showed one UV-active spot with the R_f value of 0.35. Because its ^{1}H NMR data indicated the presence of many compounds, it was not further investigated.

Fraction B3B4B3 Chromatogram characteristics on normal phase TLC with Toluene:$CHCl_3$:EtOAc:HCOOH in a ratio of 40:20:40:1 (2 runs) showed two UV-active spots with the R_f values of 0.37 and 0.45. Further purification by precoated TLC with Toluene:$CHCl_3$:EtOAc:HCOOH in a ratio of 40:20:40:1 (4 runs) as a mobile phase afforded three bands. They were not further investigated because their chromatograms on normal phase TLC using Toluene:$CHCl_3$:EtOAc:HCOOH in a ratio of 40:20:40:1 (8 runs) showed many spots under UV-S and they were obtained in low quantity. Moreover, their ^{1}H NMR spectra displayed many compounds.

Fraction B3B4B4 Chromatogram characteristics on normal phase TLC with 20%Acetone/Petrol showed no definite spot under UV-S. Further investigation was then not carried out.

Fraction B3B4C Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 (3 runs) showed three UV-active spots with the R_f values of 0.25, 0.37 and 0.45. Its ^{1}H NMR data were similar to those of fraction **B3B3B**. Further investigation was then not carried out.

Fraction B3B4D Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 (3 runs) showed two UV-active spots with the R_f values of 0.30 and 0.35 and three brown spots under ASA reagent with the R_f values of 0.20, 0.32 and 0.37. Because its ^{1}H NMR data indicated the presence of many compounds, it was not further investigated.

Fraction B3B4E Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 (3 runs) showed no definite spot under UV-S and ASA reagent. It was not further investigated.

Fraction B3B5 Chromatogram characteristics on normal phase TLC with 30%Acetone/Petrol (2 runs) showed three UV-active spots with the R_f values of 0.12, 0.25 and 0.32 and one brown spot under ASA reagent with the R_f value of 0.37. It was

further separated by column chromatography over silica gel. Elution was conducted initially with 30%Acetone/Petrol, gradually enriched with acetone and finally with pure acetone then enriched with methanol and finally with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford four fractions as shown in **Table 34**.

Table 34 Fractions obtained from the fraction **B3B5** by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
B3B5A	30%Acetone/Petrol	25.1	Yellow solid
B3B5B	30-60%Acetone/Petrol	35.8	Pale yellow solid
B3B5C	60%Acetone/Petrol	8.2	Colorless gum
B3B5D	60%Acetone/Petrol-100%MeOH	16.8	Colorless gum

Fraction B3B5A Chromatogram characteristics on normal phase TLC with 25%Acetone/Petrol (4 runs) showed one UV-active spot with the R_f value of 0.32 and three brown spots under ASA reagent with the R_f values of 0.37, 0.62 and 0.77. It was further separated by column chromatography over silica gel. Elution was conducted initially with 30%Acetone/Petrol, gradually enriched with acetone and finally with pure acetone then enriched with methanol and finally with pure methanol. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford five fractions. They were not further investigated because their chromatograms on normal phase TLC using 30%Acetone/Petrol showed many spots under ASA reagent and they were obtained in low quantity. Moreover, their ^{1}H NMR spectra displayed proton signals in the high field region.

Fraction B3B5B Chromatogram characteristics on normal phase TLC with 25%Acetone/Petrol (4 runs) showed two UV-active spots with the R_f values of 0.32 and 0.37. Its ^{1}H NMR data indicated the presence of **SK3** as a major component. Further investigation was then not carried out.

Fraction B3B5C Chromatogram characteristics on normal phase TLC with 25%Acetone/Petrol (4 runs) showed one UV-active spot with the R_f value of 0.05 and one brown spot under ASA reagent with the R_f value of 0.25. Its ^1H NMR spectrum showed the absence of aromatic and olefinic protons. Thus, it was not further investigated.

Fraction B3B5D Chromatogram characteristics on normal phase TLC with 25%Acetone/Petrol (4 runs) showed none of well separate under UV-S. Its ^1H NMR spectrum showed the absence of aromatic and olefinic protons. Thus, it was not further investigated.

Fraction B3B6 Chromatogram characteristics on normal phase TLC with 15%Acetone/Petrol (4 runs) showed three UV-active spots with the R_f values of 0.12, 0.15 and 0.20 and one brown spot under ASA reagent with the R_f value of 0.60. Its ^1H NMR data were similar to those of fraction **B3C6**. Further investigation was then not carried out.

Fraction B3B7 Chromatogram characteristics on normal phase TLC with 15%Acetone/Petrol (4 runs) showed three UV-active spots with the R_f values of 0.20, 0.22 and 0.37 and one brown spot under ASA reagent with the R_f value of 0.72. Its ^1H NMR data were similar to those of fraction **B3C7**. Further investigation was then not carried out.

Fraction B3B8 Chromatogram characteristics on normal phase TLC with 15%Acetone/Petrol showed no definite spot under UV-S. Further investigation was then not carried out.

Fraction B3C Chromatogram characteristics on normal phase TLC with 10%Acetone/Petrol showed four UV-active spots with the R_f values of 0.20, 0.25, 0.50 and 0.60 and three purple spots under ASA reagent with the R_f values of 0.50, 0.72 and 0.82. It was further separated by column chromatography over silica gel. Elution was conducted initially with 10%Acetone/CH_2Cl_2, gradually enriched with acetone and finally with pure acetone then enriched with methanol and finally with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford seven fractions as shown in **Table 35**.

Table 35 Fractions obtained from the fraction **B3C** by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
B3C1	10%Acetone/CH_2Cl_2	14.0	Yellow gum
B3C2	10%Acetone/CH_2Cl_2	16.8	Yellow solid
B3C3	20-50%Acetone/CH_2Cl_2	109.8	Yellow solid
B3C4	60%Acetone/CH_2Cl_2-100%Acetone	79.1	Yellow gum
B3C5	1-5%MeOH/Acetone	21.9	Yellow gum
B3C6	10%MeOH/Acetone	61.9	Yellow gum
B3C7	10%MeOH/Acetone - 100%MeOH	68.9	Yellow gum

Fraction B3C1 Chromatogram characteristics on normal phase TLC with 80%CH_2Cl_2/Petrol showed three UV-active spots with the R_f values of 0.25, 0.30 and 0.40 and two purple spots under ASA reagent with the R_f values of 0.50 and 0.62. It was further investigated together with fraction **B3B1**.

Fraction B3C2 Chromatogram characteristics on normal phase TLC with 80%CH_2Cl_2/Petrol showed three UV-active spots with the R_f values of 0.12, 0.20 and 0.25 and two purple spots under ASA reagent with the R_f values of 0.52 and 0.62. Its ^{1}H NMR spectrum displayed proton signals in the high field region. Thus, it was not further investigated.

Fraction B3C3 Chromatogram characteristics on normal phase TLC with 5%Acetone/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.20 and 0.25 and one brown spot under ASA reagent with the R_f value of 0.50. Its ^{1}H NMR data indicated the presence of **SK2** and **SK3** as major components. Further investigation was then not carried out.

Fraction B3C4 Chromatogram characteristics on normal phase TLC with 10%Acetone/Petrol showed three UV-active spots with the R_f values of 0.12, 0.15 and 0.20 and one brown spot under ASA reagent with the R_f value of 0.40. It was further subjected to acetylation reaction in acetic anhydride (3 ml) in the presence of pyridine

(1 ml). The reaction mixture was stirred at room temperature overnight. After working up, the acetate derivative (**B3C4A**) was obtained as a pale yellow gum (15.1 mg). Chromatogram characteristics on normal phase TLC with 10%Acetone/Petrol showed three UV-active spots with the R_f values of 0.12, 0.25 and 0.30 and one brown spot under ASA reagent with the R_f value of 0.52. This fraction was further separated by column chromatography over silica gel. Elution was conducted initially with 10%Acetone/Petrol, gradually enriched with acetone and finally with pure acetone then enriched with methanol and finally with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford four fractions as shown in **Table 36**.

Table 36 Fractions obtained from the fraction **B3C4A** by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
B3C4A1	10-30%Acetone/Petrol	42.8	Pale yellow gum
B3C4A2	40%Acetone/Petrol	5.0	Pale yellow gum
B3C4A3	40%Acetone/Petrol	3.2	Colorless gum
B3C4A4	50%Acetone/Petrol-100%MeOH	18.9	Colorless gum

Fraction B3C4A1 Chromatogram characteristics on normal phase TLC with 10%Acetone/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.26 and 0.36 and one brown spot under ASA reagent with the R_f value of 0.56. This fraction was further separated by column chromatography over silica gel. Elution was conducted initially with 10%Acetone/CH_2Cl_2, gradually enriched with acetone and finally with pure acetone. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford five fractions as shown in **Table 37**.

Table 37 Fractions obtained from the fraction **B3C4A1** by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
B3C4A1A	10%Acetone/CH_2Cl_2	7.4	Colorless gum
B3C4A1B	10%Acetone/CH_2Cl_2	9.4	Colorless gum
B3C4A1C	10%Acetone/CH_2Cl_2	8.2	Pale yellow gum
B3C4A1D	10%Acetone/CH_2Cl_2	5.0	Pale yellow gum
B3C4A1E	10%Acetone/CH_2Cl_2-100%Acetone	15.6	Pale yellow gum

Fraction B3C4A1A Chromatogram characteristics on normal phase TLC with 10%Acetone/CH_2Cl_2 (3 runs) showed none of well separated spots under UV-S. Further investigation was then not carried out.

Fraction B3C4A1B Chromatogram characteristics on normal phase TLC with Acetone:Petrol:HCOOH in a ratio of 15:85:1 (6 runs) showed one UV-active spot with the R_f value of 0.25 and one brown spot under ASA reagent with the R_f value of 0.27. Further purification by precoated TLC with Acetone:Petrol:HCOOH in a ratio of 15:85:1 (12 runs) as a mobile phase gave two bands. They were not further investigated because their chromatograms on normal phase TLC using with Acetone:Petrol:HCOOH in a ratio 15:85:1 of (6 runs) showed many spots under UV-S and they were obtained in low quantity.

Fraction B3C4A1C Chromatogram characteristics on normal phase TLC with 10%Acetone/CH_2Cl_2 (3 runs) showed one UV-active spot with the R_f value of 0.36. Because its ^{1}H NMR data indicated the presence of many compounds, it was not further investigated.

Fraction B3C4A1D Chromatogram characteristics on normal phase TLC with 10%Acetone/CH_2Cl_2 (3 runs) showed one UV-active spot with the R_f value of 0.26. Its ^{1}H NMR data were similar to those of **SK12** except it gave methyl protons of acetate group. Thus, it was acetate derivative of **SK12**.

Fraction B3C4A1E Chromatogram characteristics on normal phase TLC with 10%Acetone/CH_2Cl_2 (3 runs) showed no definite spot under UV-S. It was not further investigated.

Fraction B3C4A2 Chromatogram characteristics on normal phase TLC with 10%Acetone/Petrol (2 runs) showed three UV-active spots with the R_f values of 0.12, 0.25 and 0.30 and one purple spot under ASA reagent with the R_f value of 0.95. Because its ^{1}H NMR data indicated the presence of many compounds, it was not further investigated.

Fraction B3C4A3 Chromatogram characteristics on normal phase TLC with 10%Acetone/Petrol (2 runs) showed two UV-active spots with the R_f values of 0.05 and 0.20. Because its ^{1}H NMR data indicated the presence of many compounds, it was not further investigated.

Fraction B3C4A4 Chromatogram characteristics on normal phase TLC with 10%Acetone/Petrol (2 runs) showed none of well separated spots under UV-S. It was not further investigated.

Fraction B3C5 Chromatogram characteristics on normal phase TLC with 20%Acetone/CH_2Cl_2 showed three UV-active spots with the R_f values of 0.37, 0.50 and 0.62 and one brown spot under ASA reagent with the R_f value of 0.72. It was further subjected to acetylation reaction in acetic anhydride (3 ml) in the presence of pyridine (1 ml). The reaction mixture was stirred at room temperature overnight. After working up, the acetate derivative (**B3C5A**) was obtained as a pale yellow gum (24.5 mg). Chromatogram characteristics on normal phase TLC with 20%EtOAc/Petrol showed four UV-active spots with the R_f values of 0.20, 0.30, 0.37 and 0.42. This fraction was further purified by column chromatography over silica gel. Elution was conducted initially with 20%EtOAc/Petrol, gradually enriched with ethyl acetate and finally with pure ethyl acetate then enriched with methanol and finally with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford five fractions as shown in **Table 38**.

Table 38 Fractions obtained from the fraction **B3C5A** by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
B3C5A1	20-40%EtOAc/Petrol	11.1	Colorless gum
B3C5A2	50-70%EtOAc/Petrol	10.8	Pale yellow gum
B3C5A3	70EtOAc/Petrol	6.6	Pale yellow gum
B3C5A4	80%EtOAc/Petrol-100%EtOAc	7.8	Pale yellow gum
B3C5A5	1-20%MeOH/EtOAc	5.9	Pale yellow gum
B3C5A6	40%MeOH/EtOAc-100%MeOH	9.5	Pale yellow gum

Fraction B3C5A1 Chromatogram characteristics on normal phase TLC with 20%EtOAc/Petrol showed none of well separated spots under UV-S. It was not further investigated.

Fraction B3C5A2 Chromatogram characteristics on normal phase TLC with 5%Acetone/Petrol (7 runs) showed two UV-active spots with the R_f values of 0.11 and 0.23 and two brown spots under ASA reagent with the R_f values of 0.09 and 0.25. Further purification by precoated TLC with 5%Acetone/Petrol (16 runs) as a mobile phase afforded three bands.

Band 1 was obtained as a colorless gum in 1.0 mg. Chromatogram characteristics on normal phase TLC with 5%Acetone/Petrol (7 runs) showed one brown spot under ASA reagent with the R_f value of 0.25. Because its ^{1}H NMR data indicated the presence of many compounds, it was not further investigated.

Band 2 was obtained as a colorless gum in 2.4 mg. Chromatogram characteristics on normal phase TLC with 5%Acetone/Petrol (7 runs) showed two UV-active spots with the R_f values of 0.11 and 0.23. Because its ^{1}H NMR data indicated the presence of many compounds, it was not further investigated.

Band 3 (SK19) was obtained as a pale yellow gum in 2.6 mg. Chromatogram characteristics on normal phase TLC with 5%Acetone/Petrol (7 runs) showed one UV-active spot with the R_f value of 0.09.

$[\alpha]_D^{25}$		-76.6º (c = 0.02, MeOH)
UV λ_{max}(nm)(MeOH)(log ε)		264 (3.52)
FTIR(neat):υ(cm^{-1})		3434 (OH stretching), 1669, 1714 (C=O stretching)
^{1}H NMR($CDCl_3$)(δ_{ppm})(500 MHz):		6.90 (*t*, *J* = 4.5 Hz, 1H), 5.35 (*brs*, 1H), 4.49 (*dd*, *J* = 9.5 and 4.0 Hz, 1H), 2.24 (*m*, 1H), 2.18 (*m*, 1H), 2.05 (*s*, 3H), 2.04 (*m*, 1H), 1.95 (*m*, 1H), 1.92 (*m*, 1H), 1.85 (*s*, 3H), 1.78 (*m*, 2H), 1.73 (*m*, 1H), 1.71 (*m*, 1H), 1.67 (*m*, 3H), 1.66 (*m*, 2H), 1.60 (*m*, 1H), 1.50 (*m*, 2H), 1.44 (*m*, 1H), 1.42 (*m*, 1H), 1.39 (*m*, 1H), 1.15 (*s*, 3H), 1.10 (*m*, 1H), 0.92 (*s*, 3H), 0.88 (*s*, 3H), 0.87 (*s*, 3H), 0.85 (*brs*, 3H), 0.76 (*s*, 3H)
^{13}C NMR($CDCl_3$)(δ_{ppm})(125 MHz):		171.20, 170.88, 153.01, 145.15, 126.74, 120.74, 80.63, 75.20, 54.53, 49.13, 45.14, 44.66, 42.10, 39.11, 37.70, 37.59, 31.14, 29.92, 29.05, 28.83, 28.10, 27.46, 25.63, 23.60, 21.26, 20.78, 19.82, 16.52, 16.35, 15.34, 15.15, 12.05
DEPT135° ($CDCl_3$)(δ_{ppm})	CH:	145.15, 120.74, 80.63, 45.14, 39.11, 37.59
	CH_2:	44.66, 31.14, 29.92, 29.05, 28.83, 27.46, 25.63, 23.60, 20.78
	CH_3:	28.10, 21.26, 19.82, 16.52, 16.35, 15.34, 15.15, 12.05
EIMS *m*/*z* (% relative intensity) :		514 (3), 497 (9), 495 (27), 387 (19), 355 (66), 313 (53), 295 (100), 161 (73), 121 (62)

Fraction B3C5A3 Chromatogram characteristics on normal phase TLC with 20%EtOAc/Petrol showed two UV-active spots with the R_f values of 0.37 and 0.42 and two brown spots under ASA reagent with the R_f values of 0.15 and 0.50.

Because its ^{1}H NMR data indicated the presence of many compounds, it was not further investigated.

Fraction B3C5A4 Chromatogram characteristics on normal phase TLC with 20%EtOAc/Petrol showed two UV-active spots with the R_f values of 0.20 and 0.30. Because its ^{1}H NMR data indicated the presence of many compounds, it was not further investigated.

Fraction B3C5A5 Chromatogram characteristics on normal phase TLC with 20%EtOAc/Petrol showed two UV-active spots with the R_f values of 0.07 and 0.20. Because its ^{1}H NMR data indicated the presence of many compounds, it was not further investigated.

Fraction B3C5A6 Chromatogram characteristics on normal phase TLC with 20%EtOAc/Petrol showed no definite spot under UV-S. It was not further investigated.

Fraction B3C6 Chromatogram characteristics on normal phase TLC with 20%Acetone/CH_2Cl_2 showed three UV-active spots with the R_f values of 0.25, 0.50 and 0.62 and one brown spot under ASA reagent with the R_f value of 0.72. Its ^{1}H NMR data were similar to those of fraction **B3C5**. Further investigation was then not carried out.

Fraction B3C7 Chromatogram characteristics on normal phase TLC with 20%Acetone/CH_2Cl_2 showed no definite spot under UV-S and ASA reagent. Further investigation was then not carried out.

Fraction B3D Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 (2 runs) showed six UV-active spots with the R_f values of 0.07, 0.19, 0.24, 0.29, 0.43 and 0.58 and three brown spots under ASA reagent with the R_f values of 0.46, 0.53 and 0.61. Its ^{1}H NMR data were similar to those of fraction **B3C**. Further investigation was then not carried out.

Fraction B3E Chromatogram characteristics on reverse phase TLC with 30% MeOH/H_2O showed three UV-active spots with the R_f values of 0.25, 0.36 and 0.40. It was further purified by column chromatography over reverse phase C_{18} silica gel. Elution was conducted initially with 30%MeOH/H_2O, gradually enriched with methanol until pure methanol. Fractions with similar chromatogram characteristics

were combined and evaporated to dryness under reduced pressure to afford four fractions as shown in **Table 39**.

Table 39 Fractions obtained from the fraction **B3E** by column chromatography over reverse phase C_{18} silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
B3E1	30%MeOH/H_2O	5.2	Brown yellow gum
B3E2	30-40%MeOH/H_2O	14.2	Brown yellow gum
B3E3	40-80%MeOH/H_2O	8.7	Yellow gum
B3E4	100%MeOH	5.2	Yellow gum

Fraction B3E1 Chromatogram characteristics on normal phase TLC with 5%MeOH/CH_2Cl_2 (2 runs) showed three major UV-active spots with the R_f values of 0.26, 0.40 and 0.42. Further separation by column chromatography over Sephadex LH-20 was performed. Elution was conducted with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford seven fractions. No further purification of each fraction was attempted as each fraction was obtained in low quantity.

Fraction B3E2 Chromatogram characteristics on normal phase TLC with 5% MeOH/CH_2Cl_2 (2 runs) showed two major UV-active spots with the R_f values of 0.33 and 0.35. Further separation purified by column chromatography over Sephadex LH-20 was performed. Elution was conducted with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford three fractions. All fractions were obtained in low quantity. They were not further investigated.

Fraction B3E3 Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 (2 runs) showed three UV-active spots with the R_f values of 0.12, 0.19 and 0.28. Because its ^1H NMR data indicated the presence of many compounds, it was not further investigated.

Fraction B3E4 Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 (2 runs) showed no definite spot under UV-S. It was not further investigated.

Fraction B3F Chromatogram characteristics on reverse phase TLC with 30% MeOH/H_2O showed three major UV-active spots with the R_f values of 0.42, 0.45 and 0.64. It was further purified by column chromatography over reverse phase C_{18} silica gel. Elution was conducted initially with 30%MeOH/H_2O, gradually enriched with methanol until pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford five fractions as shown in **Table 40**.

Table 40 Fractions obtained from the fraction **B3F** by column chromatography over reverse phase C_{18} silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
B3F1	30%MeOH/H_2O	7.8	Brown yellow gum
B3F2	30%MeOH/H_2O	8.2	Brown yellow gum
B3F3	40%MeOH/H_2O	5.7	Brown yellow gum
B3F4	40%MeOH/H_2O	3.2	Yellow gum
B3F5	50%MeOH/H_2O-100%MeOH	15.7	Yellow gum

Fraction B3F1 Chromatogram characteristics on normal phase TLC with 5%MeOH/CH_2Cl_2 showed none of well separated spots under UV-S. It was not further investigated.

Fraction B3F2 Chromatogram characteristics on normal phase TLC with 5%MeOH/CH_2Cl_2 showed one UV-active spot with the R_f value of 0.35. Its ^1H NMR data were similar to those of fraction **B3E2**. It was further investigated with fraction **B3E2**.

Fraction B3F3 Chromatogram characteristics on normal phase TLC with 5%MeOH/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.23 and 0.59.

Because its ^{1}H NMR data indicated the presence of many compounds, it was not further investigated.

Fraction B3F4 Chromatogram characteristics on normal phase TLC with 5%MeOH/CH_2Cl_2 showed one UV-active spot with the R_f value of 0.71. Because its ^{1}H NMR data indicated the presence of many compounds, it was not further investigated.

Fraction B3F5 Chromatogram characteristics on normal phase TLC with 5%MeOH/CH_2Cl_2 showed none of well separated spots under UV-S. It was not further investigated.

Fraction B4 Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 showed three UV-active spots with the R_f values of 0.30, 0.40 and 0.57 and two brown spots under ASA reagent with the R_f values of 0.77 and 0.87. Its ^{1}H NMR data indicated the presence of **SK2** and **SK3** as major components. Further investigation was then not carried out.

Fraction B5 Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 showed three UV-active spots with the R_f values of 0.12, 0.17 and 0.40. Its ^{1}H NMR data indicated the presence of **SK4** and **SK8** as major components. Further investigation was then not carried out.

Fraction B6 Chromatogram characteristics on reverse phase TLC with 60% MeOH/H_2O showed four major UV-active spots with the R_f values of 0.22, 0.32, 0.50 and 0.64. It was further purified by column chromatography over reverse phase C_{18} silica gel. Elution was conducted initially with 60%MeOH/H_2O, gradually enriched with methanol until pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford four fractions as shown in **Table 41**.

Table 41 Fractions obtained from the fraction **B6** by column chromatography over reverse phase C_{18} silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
B6A	60%MeOH/H_2O	4.3	Yellow gum
B6B	70%MeOH/H_2O	1.3	Yellow gum

Table 41 (continued)

Fraction	Mobile phase	Weight (mg)	Physical appearance
B6C	70-80%MeOH/H_2O	3.1	Yellow gum
B6D	90%MeOH/H_2O-100%MeOH	10.1	Yellow gum

Fraction B6A Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 (2 runs) showed four UV-active spots with the R_f values of 0.12, 0.14, 0.28 and 0.33. Its ^{1}H NMR data indicated the presence of **SK4** as a major component. Further investigation was then not carried out.

Fraction B6B Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 (2 runs) showed five UV-active spots with the R_f values of 0.19, 0.24, 0.28, 0.33 and 0.35. Because its ^{1}H NMR data indicated the presence of many compounds, it was not further investigated.

Fraction B6C Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 (2 runs) showed one UV-active spot with the R_f value of 0.21. Its ^{1}H NMR data indicated the presence of **SK8** as a major component. Further investigation was then not carried out.

Fraction B6D Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 (2 runs) showed no definite spot under UV-S. It was not further investigated.

Fraction B7 Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 showed no definite spot under UV-S. It was not further investigated.

Fraction C (**SK1**) Upon standing at room temperature, a white solid (0.32 g) precipitated. Its chromatogram on normal phase TLC with 60%CH_2Cl_2/Petrol showed one brown spot under ASA reagent with the R_f value of 0.25.

Melting point (°C)	221-224 °C
$[\alpha]_D^{28}$	+51.5º (c = 0.20, MeOH)
UV λ_{max}(nm)(MeOH)(log ε)	207 (2.87)

FTIR(neat):υ(cm^{-1})	3365 (OH stretching), 1696 (C=O stretching)
^{1}H NMR ($CDCl_3 + CD_3OD$) (δ_{ppm}) (300 MHz):	5.28 (*d*, *J* = 6.0 Hz, 1H), 5.21 (*s*, 1H), 3.21 (*dd*, *J* = 12.0 and 5.0 Hz, 1H), 2.85 (*m*, 1H), 2.80-2.70 (*m*, 1H), 2.67 (*m*, 1H), 2.66-2.61 (*m*, 1H), 2.49 (*m*, 1H), 2.45 (*m*, 1H), 2.39-2.30 (*m*, 2H), 2.07 (*d*, *J* = 14.0 Hz, 1H), 1.80 (*m*, 1H), 1.80-1.60 (*m*, 4H), 1.57-1.29 (*m*, 4H), 1.18 (*d*, *J* = 6.9 Hz, 3H), 1.05 (*s*, 3H), 1.02 (*d*, *J* = 6.6 Hz, 3H), 0.99 (*s*, 3H), 0.89 (*m*, 1H), 0.79 (*s*, 6H), 0.75 (*s*, 1H)
^{13}C NMR ($CDCl_3$+CD_3OD) (δ_{ppm}) (125 MHz):	208.49, 177.65, 155.72, 149.52, 120.40, 114.44, 78.84, 52.53, 50.98, 49.23, 46.66, 46.58, 40.78, 39.95, 39.64, 39.13, 36.16, 34.52, 31.20, 28.21, 28.04, 28.00, 27.69, 21.24, 22.12, 21.05, 19.88, 19.38, 16.97, 15.61

The filtrate became a yellow green gum (3.70 g) after evaporation to dryness under reduced pressure. Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 showed five UV-active spots with the R_f values of 0.24, 0.36, 0.48, 0.51 and 0.60. It was further separated by column chromatography over Sephadex LH-20. Elution was conducted with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford eight fractions as shown in **Table 42**.

Table 42 Fractions obtained from the fraction **C** by column chromatography over Sephadex LH-20

Fraction	Weight (g)	Physical appearance
C1	435.1	Brown solid
C2	2203.6	Pale yellow solid

Table 42 (continued)

Fraction	Weight (g)	Physical appearance
C3	300.9	Pale yellow solid
C4	58.5	Yellow solid
C5	49.6	Yellow solid
C6	11.2	Yellow solid
C7	18.3	Yellow solid
C8	23.4	Brown yellow solid

Fraction C1 Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 (2 runs) showed two UV-active spots with the R_f values of 0.07 and 0.13. Its ^{1}H NMR data indicated the presence of long chain hydrocarbons. Thus, it was not further investigated.

Fraction C2 Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 (2 runs) showed four UV-active spots with the R_f values of 0.13 0.25, 0.46 and 0.56. Its ^{1}H NMR spectrum displayed proton signals in the high field region. Thus, it was not further investigated.

Fraction C3 Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 (2 runs) showed two UV-active spots with the R_f values of 0.13 and 0.38. Its ^{1}H NMR spectrum was similar to that of fraction **D2**. Thus, it was not further investigated.

Fraction C4 Chromatogram characteristics on reverse phase TLC with 60%MeOH/H_2O showed three UV-active spots with the R_f values of 0.11, 0.22 and 0.42. It was further separated by column chromatography over reverse phase C_{18} silica gel. Elution was conducted initially with 60%MeOH/H_2O, gradually enriched with methanol until pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford five fractions. They were not further investigated because their ^{1}H NMR spectra showed the absence of aromatic and olefinic protons.

Fraction C5 Chromatogram characteristics on reverse phase TLC with 60% MeOH/H_2O showed four major UV-active spots with the R_f values of 0.13, 0.15, 0.43

and 0.49. It was further purified by column chromatography over reverse phase C_{18} silica gel. Elution was conducted initially with 60%MeOH/H_2O, gradually enriched with methanol until pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford eight fractions. They were not further purified because their chromatograms showed many spots under UV-S and they were obtained in low quantity.

Fraction C6 Chromatogram characteristics on reverse phase TLC with 60% MeOH/H_2O showed three UV-active spots with the R_f values of 0.21, 0.25 and 0.31. Further separation by column chromatography over reverse phase C_{18} silica gel was performed. Elution was conducted initially with 60%MeOH/H_2O, gradually enriched with methanol until pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford five fractions as shown in **Table 43**.

Table 43 Fractions obtained from the fraction **C6** by column chromatography over reverse phase C_{18} silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
C6A	60%MeOH/H_2O	1.8	Yellow gum
C6B	70%MeOH/H_2O	4.8	Yellow gum
C6C	80%MeOH/H_2O	1.8	Yellow gum
C6D	80%MeOH/H_2O	1.9	Yellow gum
C6E	100%MeOH	7.5	Yellow gum

Fraction C6A Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 (3 runs) showed one UV-active spot with the R_f value of 0.11. Its ^{1}H NMR spectrum indicated the presence of many compounds, it was not further investigated.

Fraction C6B Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 (3 runs) showed six UV-active spots with the R_f values of 0.16, 0.28, 0.30, 0.34, 0.38 and 0.51. Because of the low quantity, it was not further investigated.

Fraction C6C (SK8) Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 (3 runs) showed one UV-active spot with the R_f value of 0.30.

UVλ_{max}(nm)(MeOH)(log ε)		239 (4.08), 254 (4.13), 3.13 (3.65), 364 (3.65)
FTIR (neat):υ(cm^{-1})		3375 (OH stretching), 1671 (C=O stretching)
^{1}H NMR (Acetone-d_6)(δ_{ppm})(500 MHz):		13.70 (*s*, 1H), 6.83 (*s*, 1H), 6.29 (*brs*, 1H), 6.18 (*brs*, 1H), 5.32 (*mt*, *J* = 7.0 Hz, 1H), 4.18 (*d*, *J* = 7.0 Hz, 2H), 1.83 (*s*, 3H), 1.64 (*s*, 3H)
^{13}C NMR (Acetone-d_6)(δ_{ppm})(125 MHz):		183.11, 165.15, 164.89, 158.01, 153.71, 153.02, 141.98, 131.28, 128.90, 124.49, 111.78, 103.87, 101.25, 98.48, 93.58, 26.36, 26.00, 18.28
DEPT135° (Acetone-d_6)(δ_{ppm})	CH:	124.49, 101.25, 93.58
	CH_2:	26.36
	CH_3:	26.00, 18.28

Fraction C6D Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 (3 runs) showed three UV-active spots with the R_f values of 0.21, 0.32 and 0.62. Because of the minute quantity, it was not further investigated.

Fraction C6E Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 (3 runs) showed no definite spot under UV-S. It was not further investigated.

Fraction C7 Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 (3 runs) showed three UV-active spots with the R_f values of 0.05, 0.18 and 0.28. It was separated by column chromatography over Sephadex LH-20. Elution was conducted with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford four fractions as shown in **Table 44**.

Table 44 Fractions obtained from the fraction **C7** by column chromatography over Sephadex LH-20

Fraction	Weight (mg)	Physical appearance
C7A	5.9	Yellow gum
C7B	3.6	Yellow gum
C7C	5.1	Yellow gum
C7D	1.5	Yellow gum

Fraction C7A Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 (3 runs) showed none of well separated spots under UV-S. It was not further investigated.

Fraction C7B Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 (3 runs) showed three UV-active spots with the R_f values of 0.22, 0.33 and 0.48. Because of the low quantity, it was not further investigated.

Fraction C7C Chromatogram characteristics on normal phase TLC with 5%MeOH/CH_2Cl_2 (3 runs) showed two UV-active spots with the R_f values of 0.34 and 0.41. Further purification by precoated TLC was carried out with 5%MeOH/CH_2Cl_2 (6 runs) as a mobile phase to gave three bands.

Band 1 was obtained as a colorless gum in 1.5 mg. Its chromatogram characteristics on normal phase TLC with 5%MeOH/CH_2Cl_2 (3 runs) showed one UV-active spot with the R_f value of 0.41. Because its ^{1}H NMR spectrum indicated the presence of many compounds, it was not further investigated.

Band 2 was obtained as a pale yellow gum in 2.1 mg. Its chromatogram characteristics on normal phase TLC with 5%MeOH/CH_2Cl_2 (3 runs) showed two UV-active spot with the R_f value of 0.34. It was not further investigated because of the minute quantity.

Band 3 (SK4) was obtained as a yellow solid in 2.3 mg. Its chromatogram characteristics on normal phase TLC with 5%MeOH/CH_2Cl_2 (3 runs) showed one UV-active spot with the R_f value of 0.12.

Melting point (°C) 212-215 °C

UVλ_{max}(nm)(MeOH)(log ε)	235 (5.48), 253 (5.50), 312 (5.25), 362 (5.16)
FTIR(neat):υ(cm^{-1})	3419 (OH stretching), 1655 (C=O stretching)
^{1}H NMR(Acetone-d_6)(δ_{ppm})(500 MHz):	13.23 (*s*, 1H), 7.53 (*s*, 1H), 6.92 (*s*, 1 H), 6.37 (*brs*, 1H), 6.22 (*brs*, 1H)
^{13}C NMR(Acetone-d_6)(δ_{ppm})(125 MHz):	179.59, 164.68, 163.58, 157.99, 153.92, 151.81, 143.48, 112.66, 108.16, 102.51, 102.27, 97.69, 93.50
DEPT135° (Acetone-d_6)(δ_{ppm}) CH:	108.16, 102.51, 97.69, 93.50

Fraction C7D Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 (3 run) showed one UV-active spot with the R_f value of 0.12. Its ^{1}H NMR spectrum was similar to that of **SK4**. It was not further investigated.

Fraction C8 Chromatogram characteristics on normal phase TLC with 2% MeOH/CH_2Cl_2 (2 runs) showed none of well separated spots under UV-S. Its ^{1}H NMR spectrum showed the absence of aromatic and olefinic protons, it was not further investigated.

Fraction D Upon standing at room temperature, a white solid (0.32 g) precipitated. Its chromatogram on normal phase TLC with 60%CH_2Cl_2/Petrol showed one major spot under ASA reagent with the R_f value of 0.25. Its ^{1}H NMR data indicated the presence of **SK1** as a major component.

The filtrate became a yellow green gum (3.70 g) after evaporation to dryness under reduced pressure. Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 showed five UV-active spots with the R_f values of 0.12, 0.14, 0.24, 0.33 and 0.74. It was separated by column chromatography over Sephadex LH-20. Elution was conducted with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford six fractions as shown in **Table 45**.

Table 45 Fractions obtained from the fraction **D** by column chromatography over Sephadex LH-20

Fraction	Weight (mg)	Physical appearance
D1	388.6	Brown-yellow gum
D2	1493.2	Brown-yellow gum
D3	538.8	Yellow gum with yellow solid
D4	481.7	Pale-yellow gum
D5	31.0	Yellow solid
D6	29.2	Pale-yellow solid

Fraction D1 Chromatogram characteristics on normal phase TLC with 5%MeOH/CH_2Cl_2 showed none of well separated spots under UV-S and ASA reagent. Its ^{1}H NMR spectrum displayed proton signals in the high field region, it was not further investigated.

Fraction D2 Chromatogram characteristics on normal phase TLC with 5%MeOH/CH_2Cl_2 showed five UV-active spots with the R_f values of 0.24, 0.42, 0.51, 0.73 and 0.83. This fraction was further separated by column chromatography over silica gel. Elution was conducted initially with pure dichloromethane, gradually enriched with methanol and finally with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford four fractions as shown in **Table 46**.

Table 46 Fractions obtained from the fraction **D2** by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
D2A	100%CH_2Cl_2-0.5%MeOH/CH_2Cl_2	146.8	Dark yellow gum with white solid
D2B	1%MeOH/CH_2Cl_2	67.2	Dark yellow gum with white solid

Table 46 (continued)

Fraction	Mobile phase	Weight (mg)	Physical appearance
D2C	1-2%MeOH/CH_2Cl_2	552.2	Yellow-brown gum
D2D	5%MeOH/CH_2Cl_2-100%MeOH	400.3	Brown gum

Fraction D2A Upon standing at room temperature, a white solid (0.32 g) precipitated. Its chromatogram on normal phase TLC with 60%CH_2Cl_2/Petrol showed one brown spot under ASA reagent with the R_f value of 0.25. Its ^1H NMR data indicated the presence of **SK1** as a major component.

The filtrate became a yellow green gum (127.0 mg) after evaporation to dryness under reduced pressure. Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.30 and 0.50 and long tail. Further separation by column chromatography over silica gel was performed. Elution was conducted initially with 0.5%MeOH/CH_2Cl_2, gradually enriched with methanol and finally with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford eight fractions as shown in **Table 47**.

Table 47 Fractions obtained from the fraction **D2A** by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
D2A-1	0.5%MeOH/CH_2Cl_2	38.5	Colorless gum
D2A-2	0.5%MeOH/CH_2Cl_2	9.0	Colorless gum
D2A-3	1.0-1.5%MeOH/CH_2Cl_2	1.7	Colorless gum
D2A-4	2.0-7.0%MeOH/CH_2Cl_2	3.3	Colorless gum
D2A-5	20%MeOH/CH_2Cl_2	5.1	Colorless gum
D2A-6	15%MeOH/CH_2Cl_2	1.2	Colorless gum
D2A-7	20%MeOH/CH_2Cl_2	98.8	Yellow gum

Table 47 (continued)

Fraction	Mobile phase	Weight (mg)	Physical appearance
D2A-8	40%MeOH/CH_2Cl_2-100%MeOH	15.6	Yellow gum

Fraction D2A-1 Chromatogram characteristics on normal phase TLC with 100%CH_2Cl_2 showed one UV-active spot with the R_f value of 0.61 and three brown spots under ASA reagent with the R_f values of 0.39, 0.49 and 0.73. This fraction was separated by column chromatography over silica gel. Elution was conducted initially with 50%CH_2Cl_2/Petrol, gradually enriched with dichloromethane until pure dichloromethane then enriched with methanol and finally with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford six fractions. They were not further investigated because their chromatograms on normal phase TLC using 80%CH_2Cl_2/Petrol showed many spots under ASA reagent and they were obtained in low quantity. Moreover, their ^{1}H NMR spectra displayed proton signals in the high field region.

Fraction D2A-2 Chromatogram characteristics on normal phase TLC with 100%CH_2Cl_2 showed one spot under ASA reagent with the R_f value of 0.61. Its ^{1}H NMR spectrum displayed proton signals in the high field region. Thus, it was not further investigated.

Fraction D2A-3 Chromatogram characteristics on normal phase TLC with 100%CH_2Cl_2 showed one UV-active spot with the R_f value of 0.44 and two purple spots under ASA reagent with the R_f values of 0.51 and 0.61. Its ^{1}H NMR spectrum displayed proton signals in the high field region. Thus, it was not further investigated.

Fraction D2A-4 Chromatogram characteristics on normal phase TLC with 100%CH_2Cl_2 showed one brown spot under ASA reagent with the R_f value of 0.29. Its ^{1}H NMR spectrum displayed proton signals in the high field region. Thus, it was not further investigated.

Fraction D2A-5 Chromatogram characteristics on normal phase TLC with 100%CH_2Cl_2 showed one UV-active spot with the R_f value of 0.41 and two purple

spots under ASA reagent with the R_f values of 0.29 and 0.63. Its ^{1}H NMR spectrum displayed proton signals in the high field region. Thus, it was not further investigated.

Fraction D2A-6 Chromatogram characteristics on normal phase TLC with 100%CH_2Cl_2 showed one brown spot under ASA reagent with the R_f value of 0.34. Its ^{1}H NMR spectrum displayed proton signals in the high field region. Thus, it was not further investigated.

Fraction D2A-7 Chromatogram characteristics on normal phase TLC with 10%Acetone/CH_2Cl_2 showed three UV-active spots with the R_f values of 0.35, 0.45 and 0.50. It was further separated by column chromatography over silica gel. Elution was conducted initially with 5%Acetone/CH_2Cl_2, gradually enriched with acetone until pure acetone then enriched with methanol and finally with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford seven fractions as shown in **Table 48**.

Table 48 Fractions obtained from the fraction **D2A-7** by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
D2A-7A	5%Acetone/CH_2Cl_2	1.7	Colorless gum
D2A-7B	7%Acetone/CH_2Cl_2	5.0	Colorless gum
D2A-7C	10%Acetone/CH_2Cl_2	2.5	Yellow gum
D2A-7D	10%Acetone/CH_2Cl_2	7.5	Yellow gum
D2A-7E	15-40%Acetone/CH_2Cl_2	13.0	Yellow gum
D2A-7F	60%Acetone/CH_2Cl_2	30.6	Yellow gum
D2A-7G	60%Acetone/CH_2Cl_2-100%MeOH	19.9	Colorless gum

Fraction D2A-7A Chromatogram characteristics on normal phase TLC with 2%Acetone/CH_2Cl_2 showed none of well separated spots under UV-S. Thus, it was not further investigated.

Fraction D2A-7B Chromatogram characteristics on normal phase TLC with 2%Acetone/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.50 and 0.62.

Because its ^{1}H NMR data indicated the presence of many compounds, it was not further investigated.

Fraction D2A-7C Chromatogram characteristics on normal phase TLC with 5%Acetone/CH_2Cl_2 showed one UV-active spot with the R_f value of 0.20 and long tail under UV-S. Thus, it was not further investigated because of the minute quantity.

Fraction D2A-7D (**SK2**) Chromatogram characteristics on normal phase TLC with 2%Acetone/CH_2Cl_2 showed one UV-active spot with the R_f value of 0.30.

$[\alpha]_D^{28}$	-23.3º (c = 0.09, MeOH)
UVλ_{max}(nm)(MeOH)(log ε)	218 (5.02)
FTIR(neat):υ(cm^{-1})	3420 (OH stretching), 1704 (C=O stretching)
^{1}H NMR ($CDCl_3$) (δ_{ppm})(300 MHz):	6.72 (*qd*, *J* = 8.1 and 1.5 Hz, 1H), 5.27 (*brs*, 1H), 4.57 (*ddd*, *J* = 11.0 , 8.4 and 2.4 Hz, 1H), 3.76 (*s*, 3H), 3.45 (*t*, *J* = 2.4 Hz, 1H), 2.35 (*m*, 1H), 2.33 (*m*, 1H), 2.30 (*brd*, *J* = 16.2 Hz, 1H) 2.18 (*m*, 1H), 2.09 (*m*, 1H), 2.03 (*m*, 1H), 1.98 (*m*, 1H), 1.95 (*m*, 1H), 1.87 (*d*, *J* = 1.5 Hz, 3H), 1.74 (*m*, 1H), 1.66 (*m*, 1H), 1.65 (*m*, 1H), 1.62 (*m*, 1H), 1.59 (*m*, 4H), 1.49 (*m*, 1H), 1.12 (*m*, 1H), 1.01 (*s*, 3H), 0.99 (*s*, 3H), 0.94 (*d*, *J* = 6.6 Hz, 3H), 0.90 (*s*, 3H), 0.89 (*s*, 3H), 0.76 (*s*, 3H)
^{13}C NMR($CDCl_3$)(δ_{ppm})(75 MHz):	168.49, 148.79, 144.49, 142.36, 127.09, 122.85, 115.80, 75.85, 66.89, 51.95, 50.02, 48.01, 45.54, 44.75, 39.46, 37.81, 37.60, 33.40, 30.10, 29.22, 27.99, 26.69, 25.58, 22.72, 22.19, 18.95, 18.15, 17.08, 15.65, 15.27, 12.73

Fraction D2A-7E Chromatogram characteristics on normal phase TLC with 5%Acentone/CH_2Cl_2 showed one UV-active spot with the R_f value of 0.30 and two brown spots under ASA reagent with the R_f values of 0.45 and 0.52. Its ^{1}H NMR spectrum was similar to that of **SK2** as a major component. Thus, it was not further investigated.

Fraction D2A-7F (SK3) Chromatogram characteristics on normal phase TLC with 5%Acentone/CH_2Cl_2 showed one UV-active spot with the R_f value of 0.25.

$[\alpha]_D^{29}$	-42.3º (c = 0.41, MeOH)
UVλ_{max}(nm)(MeOH)(log ε)	217 (4.28)
FTIR(neat):υ(cm^{-1})	3420 (OH stretching), 1697 (C=O stretching)
^{1}H NMR($CDCl_3$)(δ_{ppm})(300 MHz):	6.71 (*qd*, *J* = 8.1 and 1.5 Hz, 1H), 5.32 (*brs*, 1H), 4.54 (*t*, *J* = 8.4 Hz, 1H), 3.77 (*s*, 3H), 3.37 (*brs*, 1H), 2.35 (*m*, 1H), 2.28 (*m*, 1H), 2.27 (*m*, 1H), 1.98 (*m*, 1H), 1.95 (*m*, 1H), 1.90 (*m*, 2H), 1.85 (*d*, *J* = 1.2 Hz, 3H), 1.78 (*m*, 2H), 1.69 (*m*, 1H), 1.65 (*m*, 2H), 1.63 (*m*, 1H), 1.52 (*m*, 2H), 1.39 (*m*, 1H), 1.36 (*m*, 1H), 1.23 (*s*, 3H), 1.16 (*m*, 1H), 0.95 (*s*, 3H), 0.91 (*d*, *J* = 6.6 Hz, 3H), 0.90 (*s*, 3H), 0.84 (*s*, 3H), 0.75 (*s*, 3H)
^{13}C NMR($CDCl_3$)(δ_{ppm})(75 MHz):	168.62, 153.60, 144.79, 126.79, 120.34, 76.14, 75.56, 66.68, 53.99, 51.95, 49.07, 44.71, 42.15, 39.15, 39.02, 38.95, 37.51, 32.96, 29.58, 28.97, 28.51, 25.59, 25.09, 23.60, 22.04, 20.78, 19.46, 16.43, 15.34, 15.09, 12.69

Fraction D2A-7G Chromatogram characteristics on normal phase TLC with 5%MeOH/CH_2Cl_2 showed none of well separated spots under UV-S. Thus, it was not further investigated.

Fraction D2A-8 Chromatogram characteristics on normal phase TLC with 5%MeOH/CH_2Cl_2 showed many spots under UV-S. Its ^{1}H NMR spectrum indicated the presence of many compounds. Thus, it was not further investigated.

Fraction D2B Upon standing at room temperature, a white solid (101.2 mg) precipitated. Its chromatogram on normal phase TLC with 60%CH_2Cl_2/Petrol showed one brown spot under ASA reagent with the R_f value of 0.25. Its ^{1}H NMR data indicated the presence of **SK1** as a major component.

The filtrate became a yellow green gum (56.1 mg) after evaporation to dryness under reduced pressure. Chromatogram characteristics on normal phase TLC with 5%MeOH/CH_2Cl_2 showed three UV-active spots with the R_f values of 0.12, 0.30 and 0.40. Its ^{1}H NMR data indicated the presence of **SK2** and **SK3** as major components. Further investigation was then not carried out.

Fraction D2C Chromatogram characteristics on normal phase TLC with 5%MeOH/CH_2Cl_2 showed three UV-active spots with the R_f values of 0.12, 0.30 and 0.40. Its ^{1}H NMR data indicated the presence of **SK2** and **SK3** as major components. Further investigation was then not carried out.

Fraction D2D Chromatogram characteristics on normal phase TLC with 5%MeOH/CH_2Cl_2 showed none of well separated spots under UV-S. Thus, it was not further investigated.

Fraction D3 Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 (2 runs) showed four UV-active spots with the R_f values of 0.13 0.38, 0.42 and 0.51. Its ^{1}H NMR spectrum was similar to that of fraction **D2**. Thus, it was not further investigated.

Fraction D4 Chromatogram characteristics on normal phase TLC with 5%MeOH/CH_2Cl_2 showed four UV-active spots with the R_f values of 0.24, 0.32, 0.47, and 0.56. It was further separated by column chromatography over Sephadex LH-20. Elution was conducted with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford eight fractions as shown in **Table 49**.

Table 49 Fractions obtained from the fraction **D4** by column chromatography over Sephadex LH-20

Fraction	Weight (mg)	Physical appearance
D4A	17.5	Colorless gum
D4B	28.6	Yellow gum
D4C	90.1	Yellow gum
D4D	28.6	Yellow gum
D4E	102.6	Yellow-brown gum
D4F	130.6	Yellow gum
D4G	18.2	Yellow gum
D4H	3.6	Yellow gum

Fraction D4A Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 showed none of well separated spots under UV-S. Further investigation was then not carried out.

Fraction D4B Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 showed four UV-active spots with the R_f values of 0.12, 0.19, 0.38 and 0.40. Its ^{1}H NMR spectrum displayed proton signals in the high field region. Thus, it was not further investigated.

Fraction D4C Chromatogram characteristics on reverse phase TLC with 50% MeOH/H_2O showed three major UV-active spots with the R_f values of 0.11, 0.25 and 0.55. It was further purified by column chromatography over reverse phase C_{18} silica gel. Elution was conducted initially with 50%MeOH/H_2O, gradually enriched with methanol until pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford nine fractions. They were not further investigated because their chromatograms on normal phase TLC using 3%MeOH/CH_2Cl_2 showed many spots under UV-S and they were obtain in low quantity. Moreover, their ^{1}H NMR spectra displayed proton signals in the high field region.

Fraction D4D Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 showed three UV-active spots with the R_f values of 0.07, 0.21 and

0.38. Its ^{1}H NMR spectrum displayed proton signals in the high field region. Thus, it was not further investigated.

Fraction D4E Chromatogram characteristics on reverse phase TLC with 50% MeOH/H_2O showed three major UV-active spots with the R_f values of 0.25, 0.35 and 0.69. This fraction was further purified by column chromatography over reverse phase C_{18} silica gel. Elution was conducted initially with 50%MeOH/H_2O, gradually enriched with methanol until pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford eight fractions. They were not further investigated because their chromatograms on normal phase TLC using 3%MeOH/CH_2Cl_2 showed many spots under UV-S and they were obtained in low quantity. Moreover, their ^{1}H NMR spectra displayed proton signals in the high field region.

Fraction D4F Chromatogram characteristics on reverse phase TLC with 50% MeOH/H_2O showed six UV-active spots with the R_f values of 0.08, 0.13, 0.25, 0.35, 0.55 and 0.69. This fraction was further purification by column chromatography over reverse phase C_{18} silica gel. Elution was conducted initially with 50%MeOH/H_2O, gradually enriched with methanol until pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford six fractions as shown in **Table 50**.

Table 50 Fractions obtained from the fraction **D4F** by column chromatography over reverse phase C_{18} silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
D4F-1	50%MeOH/H_2O	38.9	Colorless gum
D4F-2	60%MeOH/H_2O	10.2	Colorless gum
D4F-3	70%MeOH/H_2O	8.4	Yellow gum
D4F-4	80-90%MeOH/H_2O	33.8	Yellow gum
D4F-5	90%MeOH/H_2O	11.0	Yellow gum
D4F-6	90%MeOH/H_2O-100%MeOH	27.2	Yellow gum

Fraction D4F-1 Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 showed four UV-active spots with the R_f values of 0.08, 0.10, 0.15 and 0.26. Its ^{1}H NMR spectrum showed the absence of aromatic and olefinic protons. Thus, it was not further investigated.

Fraction D4F-2 Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 showed one UV-active spot with the R_f value of 0.21. Its ^{1}H NMR data indicated the presence of **SK5** as a major component. Further investigation was then not carried out.

Fraction D4F-3 Chromatogram characteristics on normal phase TLC with 5%MeOH/CH_2Cl_2 showed three UV-active spots with the R_f values of 0.05, 0.12 and 0.55. Further purification by precoated TLC was carried out with 5%MeOH/CH_2Cl_2 (7 runs) as a mobile phase afforded three bands.

Band 1 (SK5) was obtained as a yellow gum in 2.4 mg. Chromatogram characteristics on normal phase TLC with 5%MeOH/CH_2Cl_2 showed one UV-active spot with the R_f value of 0.55.

UVλ_{max}(nm)(MeOH)(log ε)	228 (3.34), 257 (3.31), 310 (2.95), 374 (2.73)
FTIR(neat):υ(cm^{-1})	3666 (OH stretching), 1696 (C=O stretching)
^{1}H NMR(Acetone-d_6)(δ_{ppm})(500 MHz):	12.98 (*s*, 1H), 10.34 (*brs*, 1H), 9.34 (*s*, 1H), 7.56 (*d*, *J* = 3.0 Hz, 1H), 7.44 (*d*, *J* = 9.0 Hz, 1H), 7.34 (*dd*, *J* = 9.0 and 3.0 Hz, 1H), 6.41 (*d*, *J* = 2.5 Hz, 1H), 6.25 (*d*, *J* = 2.5 Hz, 1H)
^{13}C NMR(Acetone-d_6)(δ_{ppm})(125 MHz):	181.25, 166.57, 164.63, 159.03, 154.99, 150.73, 125.15, 121.88, 119.71, 109.38, 103.47, 98.78, 94.60
DEPT135° (Acetone-d_6)(δ_{ppm}) CH:	125.15, 119.71, 109.38, 98.78, 94.60

Band 2 was obtained as a yellow gum in 1.2 mg. Chromatogram characteristics on normal phase TLC with 5%MeOH/CH_2Cl_2 showed one UV-active

spot with the R_f value of 0.12. Its ^{1}H NMR spectrum indicated the absence of olefinic and aromatic protons. Thus, it was not further studied.

Band 3 (SK6) was obtained as a yellow gum in 2.4 mg. Chromatogram characteristics on normal phase TLC with 5%MeOH/CH_2Cl_2 showed one UV-active spot with the R_f value of 0.05.

$[\alpha]_D^{29}$	+144.5º (c = 0.05, MeOH)
UV λ_{max}(nm)(MeOH)(log ε)	221 (4.04), 288 (3.79), 335 (3.57)
FTIR(neat):υ(cm^{-1})	3420 (OH stretching), 1650 (C=O stretching)
^{1}H NMR(DMSO-d_6)(δ_{ppm})(300 MHz):	13.07 (*s*, 1H), 12.29 (*s*, 1H), 7.94 (*d*, *J* = 8.1 Hz, 2H), 7.09 (*d*, *J* = 7.8 Hz, 2H), 6.93 (*d*, *J* = 8.1 Hz, 2H), 6.78 (*s*, 1H), 6.35 (*d*, *J* = 7.8 Hz, 2H), 6.64 (*s*, 1H), 6.04 (*s*, 1H), 5.94 (*s*, 1H), 5.67 (*d*, *J* = 12.0 Hz, 1H), 4.99 (*d*, *J* = 12.0 Hz, 1H)

Fraction D4F-4 Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 showed three UV-active spots with the R_f values of 0.10, 0.23 and 0.41. Its ^{1}H NMR data indicated the presence of **SK5** as a major component. Further investigation was then not carried out.

Fraction D4F-5 Chromatogram characteristics on reverse phase TLC with 50%MeOH/H_2O showed two major UV-active spots with the R_f values of 0.35 and 0.39. This fraction was further purification by column chromatography over reverse phase C_{18} silica gel. Elution was conducted initially with 60%MeOH/H_2O, gradually pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford four fractions. They were not further investigated because their chromatograms on normal phase TLC using 5%MeOH/CH_2Cl_2 showed many UV-active spots and they were obtained in low quantity. Moreover, their ^{1}H NMR spectra displayed many compounds.

Fraction D4F-6 Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 showed no definite spot under UV-S. It was not further investigated.

Fraction D4G Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 showed one UV-active spot with the R_f value of 0.12. Its ^1H NMR data indicated the presence of **SK4** as a major component. Further investigation was then not carried out.

Fraction D4H Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 showed no definite spot under UV-S. It was not further investigated.

Fraction D5 Chromatogram characteristics on reverse phase TLC with 50% MeOH/H_2O showed two major UV-active spots with the R_f values of 0.66 and 0.84. It was further purified by column chromatography over reverse phase C_{18} silica gel. Elution was conducted initially with 50%MeOH/H_2O, gradually enriched with methanol until pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford four fractions as shown in **Table 51**.

Table 51 Fractions obtained from the fraction **D5** by column chromatography over reverse phase C_{18} silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
D5A	50%MeOH/H_2O	2.6	Yellow gum
D5B	60%MeOH/H_2O	22.5	Yellow solid
D5C	70%MeOH/H_2O	2.4	Yellow gum
D5D	80%MeOH/H_2O-100%MeOH	1.9	Yellow gum

Fraction D5A Chromatogram characteristics on normal phase TLC with 5%MeOH/CH_2Cl_2 showed one UV-active spot with the R_f value of 0.32. Its ^1H NMR spectrum indicated the presence of many compounds, it was not further investigated.

Fraction D5B Chromatogram characteristics on normal phase TLC with 5%MeOH/CH_2Cl_2 showed one UV-active spot with the R_f value of 0.12. Its ^1H NMR spectrum was similar to that of **SK4**, it was not further investigated.

Fraction D5C Chromatogram characteristics on normal phase TLC with 5%MeOH/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.48 and 0.60. Because of the minute quantity, it was not further investigated.

Fraction D5D Chromatogram characteristics on normal phase TLC with 5%MeOH/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.14 and 0.16. Because of the minute quantity, it was not further investigated.

Fraction D6 Chromatogram characteristics on reverse phase TLC with 50% MeOH/H_2O showed two major UV-active spots with the R_f values of 0.66 and 0.70. This fraction was further purification by column chromatography over reverse phase C_{18} silica gel. Elution was conducted initially with 50%MeOH/H_2O, gradually enriched with methanol until pure methanol. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford four fractions. They were not further investigated because their chromatograms on normal phase TLC using 5%MeOH/CH_2Cl_2 showed many spots under UV-S and they were obtained in low quantity. Moreover, their ^1H NMR spectra displayed many compounds.

Fraction E Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 showed four UV-active spots with the R_f values of 0.24, 0.33, 0.36 and 0.50. This fraction was separated by column chromatography over Sephadex LH-20. Elution was conducted with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford nine fractions as shown in **Table 52**.

Table 52 Fractions obtained from the fraction **E** by column chromatography over Sephadex LH-20

Fraction	Weight (mg)	Physical appearance
E1	114.6	Dark brown gum
E2	620.3	Brown yellow gum

Table 52 (continued)

Fraction	Weight (mg)	Physical appearance
E3	11.4	Yellow gum
E4	17.4	Brown red solid
E5	9.7	Yellow gum
E6	14.2	Yellow red gum
E7	13.2	Brown yellow gum
E8	10.0	Brown yellow gum
E9	29.2	Yellow solid

Fraction E1 Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 showed none of well separated spots under UV-S and ASA reagent. Its ^{1}H NMR spectrum indicated the presence of proton signals in the high field region, it was not further investigated.

Fraction E2 Chromatogram characteristics on normal phase TLC with 5%Acetone/CH_2Cl_2 showed four UV-active spots with the R_f values of 0.12, 0.24, 0.36 and 0.51. This fraction was separated by column chromatography over silica gel. Elution was conducted initially with 5%Acetone/CH_2Cl_2, gradually enriched with acetone until pure acetone then enriched with methanol and finally with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford eight fractions as shown in **Table 53**.

Table 53 Fractions obtained from the fraction **E2** by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
E2A	5%Acetone/CH_2Cl_2	6.2	Colorless gum
E2B	7%Acetone/CH_2Cl_2	2.1	Colorless gum
E2C	7-10%Acetone/CH_2Cl_2	3.0	Colorless gum

Table 53 (continued)

Fraction	Mobile phase	Weight (mg)	Physical appearance
E2D	10%Acetone/CH_2Cl_2	12.3	Colorless gum
E2E	15-40%Acetone/CH_2Cl_2	17.3	Colorless gum
E2F	60-80%Acetone/CH_2Cl_2	171.3	Yellow gum
E2G	100%Acetone	47.3	Yellow gum
E2H	100%Acetone-100%MeOH	219.4	Yellow gum

Fraction E2A Chromatogram characteristics on normal phase TLC with 5%Acetone/CH_2Cl_2 showed one UV-active spot with the R_f value of 0.70 and three brown spots under ASA reagent with the R_f values of 0.46, 0.48 and 0.81. Its ^{1}H NMR spectrum indicated the presence of proton signals in the high field region, it was not further investigated.

Fraction E2B Chromatogram characteristics on normal phase TLC with 5%Acetone/CH_2Cl_2 showed one UV-active spot with the R_f value of 0.23 and two purple spots under ASA reagent with the R_f values of 0.37 and 0.41. Its ^{1}H NMR spectrum indicated the presence of proton signals in the high field region, it was not further investigated.

Fraction E2C Chromatogram characteristics on normal phase TLC with 5%Acetone/CH_2Cl_2 showed one UV-active spot with the R_f value of 0.18 and one brown spot under ASA reagent with the R_f value of 0.32. Because of the minute quantity, it was not further investigated.

Fraction E2D Chromatogram characteristics on normal phase TLC with 5%Acetone/CH_2Cl_2 showed one UV-active spot with the R_f value of 0.48. Its ^{1}H NMR data indicated the presence of **SK2** as a major component. Further investigation was then not carried out.

Fraction E2E Chromatogram characteristics on normal phase TLC with 5%Acetone/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.23 and 0.39. Its ^{1}H NMR data indicated the presence of **SK3** as a major component. Further investigation was then not carried out.

Fraction E2F Chromatogram characteristics on normal phase TLC with 10%Acentone/Petrol showed three UV-active spots with the R_f values of 0.10, 0.25 and 0.34. This fraction was separated by column chromatography over silica gel. Elution was conducted initially with 10%Acetone/Petrol, gradually enriched with acetone until pure acetone then enriched with methanol and finally with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford seven fractions as shown in **Table 54**.

Table 54 Fractions obtained from the fraction **E2F** by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
E2F1	10-20%Acetone/Petrol	4.6	Colorless gum
E2F2	20-50%Acetone/Petrol	1.3	Colorless gum
E2F3	50%Acetone/Petrol	1.2	Colorless gum
E2F4	50%Acetone/Petrol	7.2	Colorless gum
E2F5	70%Acetone/Petrol	73.2	Yellow gum
E2F6	70-90%Acetone/Petrol	12.0	Yellow gum
E2F7	100%Acetone-100%MeOH	21.3	Yellow gum

Fraction E2F1 Chromatogram characteristics on normal phase TLC with 10%Acetone/Petrol showed none of well separated spots under UV-S. Thus, it was not further investigated.

Fraction E2F2 Chromatogram characteristics on normal phase TLC with 10%Acetone/Petrol showed one UV-active spot with the R_f value of 0.25 and long tail. Thus, it was not further investigated.

Fraction E2F3 Chromatogram characteristics on normal phase TLC with 10%Acetone/Petrol showed one UV-active spot with the R_f value of 0.12 and long tail. Its ^{1}H NMR data indicated the presence of **SK2** as a major component. Further investigation was then not carried out.

Fraction E2F4 Chromatogram characteristics on normal phase TLC with 10%Acetone/Petrol showed two UV-active spots with the R_f values of 0.25 and 0.32.

Its ^{1}H NMR data indicated the presence of **SK3** as a major component. Further investigation was then not carried out.

Fraction E2F5 Chromatogram characteristics on normal phase TLC with 20%Acetone/Petrol showed three UV-active spots with the R_f values of 0.10, 0.25 and 0.35. This fraction was separated by column chromatography over silica gel. Elution was conducted initially with 10%Acetone/Petrol, gradually enriched with acetone until pure acetone then enriched with methanol and finally with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford seven fractions. They were not further investigated because their chromatograms on normal phase TLC using 20%Acetone/Petrol showed many spots under ASA reagent and they were obtained in low quantity. Moreover, their ^{1}H NMR spectra displayed broad signals.

Fraction E2F6 Chromatogram characteristics on normal phase TLC with 25%Acetone/Petrol showed two UV-active spots with the R_f values of 0.17 and 0.20. Its ^{1}H NMR spectrum showed broad signals. Thus, it was not further studied.

Fraction E3F7 Chromatogram characteristics on normal phase TLC with 25%Acetone/Petrol showed no definite spot under UV-S. Its ^{1}H NMR spectrum showed broad signals. Thus, it was not further studied.

Fraction E2G Chromatogram characteristics on normal phase TLC with 20%Acetone/Petrol showed three UV-active spots with the R_f values of 0.09, 0.25 and 0.55. This fraction was separated by column chromatography over silica gel. Elution was conducted initially with 10%Acetone/Petrol, gradually enriched with acetone until pure acetone then enriched with methanol and finally with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford five fractions as shown in **Table 55**.

Table 55 Fractions obtained from the fraction **E2G** by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
E2G1	10-40%Acetone/Petrol	9.4	Colorless gum
E2G2	50%Acetone/Petrol	5.5	Colorless gum
E2G3	80%Acetone/Petrol	10.1	Yellow gum

Table 55 (continued)

Fraction	Mobile phase	Weight (mg)	Physical appearance
E2G4	80%Acetone/Petrol	21.6	Yellow gum
E2G5	100%Acetone-100%MeOH	24.1	Colorless gum

Fraction E2G1 Chromatogram characteristics on normal phase TLC with 10%Acetone/Petrol showed none of well separated spots under UV-S. Thus, it was not further investigated.

Fraction E2G2 Chromatogram characteristics on normal phase TLC with 10%Acetone/Petrol showed one UV-active spot with the R_f value of 0.44 and four brown spots under ASA reagent with the R_f values of 0.23, 0.25, 0.49 and 0.57. Its ^{1}H NMR spectrum indicated the presence of proton signals in the high field region, it was not further investigated.

Fraction E3G3 Chromatogram characteristics on normal phase TLC with 10%Acetone/Petrol showed one UV-active spot with the R_f value of 0.11 and two brown spots under ASA reagent with the R_f values of 0.35 and 0.39. Therefore, it was subjected to acetylation reaction in acetic anhydride (3 ml) in the presence of pyridine (1 ml). The reaction mixture was stirred at room temperature overnight. After working up, the acetate derivative (**E3G3Ac**) was obtained as a pale yellow gum in 5.1 mg. Its ^{1}H NMR data indicated the presence of many compounds. It was obtained in a minute quantity. Thus, it was not further investigated.

Fraction E2G4 Chromatogram characteristics on normal phase TLC with 20%Acetone/Petrol showed one UV-active spot with the R_f value of 0.13 and two purple spots under ASA reagent with the R_f values of 0.25 and 0.34. This fraction was separated by column chromatography over silica gel. Elution was conducted initially with 10%Acetone/Petrol, gradually enriched with acetone until pure acetone then enriched with methanol and finally with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford four fractions. They were not further investigated because their chromatograms on normal phase TLC using 20%Acetone/Petrol showed many

spots under ASA reagent and they were obtained in low quantity. Moreover, their ^{1}H NMR spectra displayed broad signals.

Fraction E2H Chromatogram characteristics on reverse phase TLC with 50% MeOH/H_2O showed four major UV-active spots with the R_f values of 0.13, 0.25, 0.35 and 0.40. This fraction was further purification by column chromatography over reverse phase C_{18} silica gel. Elution was conducted initially with 50%MeOH/H_2O, gradually enriched with methanol until pure methanol. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford eight fractions. They were not further investigated because their chromatograms on normal phase TLC using 20%Acetone/CH_2Cl_2 appeared many spots under ASA reagent and they were obtained in low quantity. Moreover, their ^{1}H NMR spectra displayed broad signals.

Fraction E3 Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 showed three UV-active spots with the R_f values of 0.07, 0.19 and 0.39. Because of the low quantity, it was not further investigated.

Fraction E4 Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 showed three UV-active spots with the R_f values of 0.07, 0.29 and 0.39. This fraction was separated by column chromatography over Sephadex LH-20. Elution was conducted with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford four fractions as shown in **Table 56**.

Table 56 Fractions obtained from the fraction **E4** by column chromatography over Sephadex LH-20

Fraction	Weight (mg)	Physical appearance
E4A	2.0	Yellow gum
E4B	7.0	Yellow solid
E4C	2.0	Yellow solid
E4D	5.1	Yellow solid

Fraction E4A Chromatogram characteristics on normal phase TLC with 12%Acetone/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.26 and 0.39. Its ^{1}H NMR spectrum showed the absence of aromatic and olefinic protons. Because of the minute quantity, it was not further investigated.

Fraction E4B Chromatogram characteristics on normal phase TLC with Toluene: EtOAc:$CHCl_3$:HCOOH in a ratio of 60:30:10:1 (3 runs) showed two UV-active spots with the R_f values of 0.15 and 0.39. Further purification by precoated TLC with Toluene: EtOAc:$CHCl_3$:HCOOH in a ratio of 60:30:10:1 (7 runs) as a mobile phase afforded two bands.

Band 1 (SK7) was obtained as a yellow gum in 2.6 mg. Chromatogram characteristics on normal phase TLC with Toluene:EtOAc:$CHCl_3$:HCOOH in a ratio of 60:30:10:1 (2 runs) showed one UV-active spot with the R_f value of 0.39.

UVλ_{max}(nm)(MeOH)(log ε)	251 (3.89)
FTIR(neat):υ(cm^{-1})	3442 (OH stretching), 1663 (C=O stretching)
^{1}H NMR($CDCl_3$+CD_3OD)(δ_{ppm})(300 MHz):	7.95 (*d*, *J* = 6.9 Hz, 2H), 6.85 (*d*, *J* = 6.9 Hz, 2H)

Band 2 was obtained as a yellow gum in 2.6 mg. Chromatogram characteristics on normal phase TLC with Toluene:EtOAc:$CHCl_3$:HCOOH in a ratio of 60:30:10:1 (2 runs) showed one UV-active spot with the R_f value of 0.15. It was not further investigated because its ^{1}H NMR spectrum the displayed the absence of aromatic and olefinic protons.

Fraction E4C Chromatogram characteristics on normal phase TLC with 12%Acetone/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.05 and 0.13. Because of the minute quantity, it was not further investigated.

Fraction E4D Chromatogram characteristics on normal phase TLC with 12%Acetone/CH_2Cl_2 showed no definite spot under UV-S. It was not further investigated.

Fraction E5 Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 showed four UV-active spot with the R_f values of 0.07, 0.29, 0.36 and 0.40. Because of low quantity, it was not further investigated.

Fraction E6 Chromatogram characteristics on reverse phase TLC with 50% MeOH/H_2O showed two major UV-active spots with the R_f values of 0.35 and 0.55. This fraction was further purification by column chromatography over reverse phase C_{18} silica gel. Elution was conducted initially with 50%MeOH/H_2O, gradually enriched with methanol until pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford four fractions as shown in **Table 57**.

Table 57 Fractions obtained from the fraction **E6** by column chromatography over reverse phase C_{18} silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
E6A	50%MeOH/H_2O	2.6	Colorless gum
E6B	50%MeOH/H_2O	6.7	Yellow gum
E6C	60-80%MeOH/H_2O	6.2	Yellow gum
E6D	100%MeOH	5.2	Colorless gum

Fraction E6A Chromatogram characteristics on normal phase TLC with 5%Acetone/CH_2Cl_2 showed none of well separated spots under UV-S. Its ^{1}H NMR spectrum showed the absence of aromatic and olefinic protons. Because of the minute quantity, it was not further investigated.

Fraction E6B Chromatogram characteristics on normal phase TLC with 12%Acetone/CH_2Cl_2 showed one UV-active spot with the R_f value 0.14. Its ^{1}H NMR data indicated the presence of many compounds. It was obtained in low quantity. Thus, it was not further investigated.

Fraction E6C Chromatogram characteristics on normal phase TLC with 12%Acetone/CH_2Cl_2 showed three UV-active spots with the R_f values of 0.24, 0.36 and 0.39. Its ^{1}H NMR spectrum displayed the absence of aromatic and olefinic protons. Because of low quantity, it was not further investigated.

Fraction E6D Chromatogram characteristics on normal phase TLC with 5%Acetone/CH_2Cl_2 showed no definite spot under UV-S. It was not further investigated.

Fraction E7 Chromatogram characteristics on reverse phase TLC with 60% MeOH/H_2O showed three major UV-active spots with the R_f values of 0.25, 0.35 and 0.40. This fraction was further purification by column chromatography over reverse phase C_{18} silica gel. Elution was conducted initially with 60%MeOH/H_2O, gradually enriched with methanol until pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford six fractions. They were not further investigated because their chromatograms on normal phase TLC using 2%MeOH/CH_2Cl_2 showed many spots under ASA reagent and they were obtained in low quantity. Moreover, their ^{1}H NMR spectra displayed broad signals.

Fraction E8 Chromatogram characteristics on normal phase TLC with 3%MeOH/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.02 and 0.26. Its ^{1}H NMR spectrum indicated the presence of many compounds, it was not further investigated.

Fraction E9 Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 showed no definite spot under UV-S. Its ^{1}H NMR spectrum indicated the presence of many compounds, it was not further investigated.

Fraction F Upon standing at room temperature, a white solid (0.32 g) precipitated. Its chromatogram on normal phase TLC with 60%CH_2Cl_2/Petrol showed one major brown spot under ASA reagent with the R_f value of 0.25. Its ^{1}H NMR data indicated the presence of **SK1** as a major component.

The filtrate became a yellow green gum (3.33 g) after evaporation to dryness under reduced pressure. Chromatogram characteristics on reverse phase TLC with 50%MeOH/H_2O showed four UV-active spots with the R_f values of 0.07, 0.11, 0.22 and 0.42. It (0.48 g) was further purified by column chromatography over reverse phase C_{18} silica gel. Elution was conducted initially with 50%MeOH/H_2O, gradually enriched with methanol until pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford seven fractions as shown in **Table 58**.

Table 58 Fractions obtained from the fraction **F** by column chromatography over reverse phase C_{18} silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
F1	50%MeOH/H_2O	4.0	Pale yellow gum
F2	50%MeOH/H_2O	275.6	Yellow gum
F3	60-70%MeOH/H_2O	71.6	Brown yellow gum
F4	80%MeOH/H_2O	55.3	Yellow gum
F5	90%MeOH/H_2O	39.9	Yellow gum
F6	90%MeOH/H_2O	43.3	Yellow gum
F7	100%MeOH	76.6	Yellow gum

Fraction F1 Chromatogram characteristics on normal phase TLC with 5%MeOH/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.05 and 0.14. Because of low quantity, it was not further investigated.

Fraction F2 Chromatogram characteristics on normal phase TLC with 5%MeOH/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.12 and 0.17. Its ^{1}H NMR spectrum showed only sugar signals. Thus, it was not further investigated.

Fraction F3 Chromatogram characteristics on normal phase TLC with 5%MeOH/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.12 and 0.21. Its ^{1}H NMR data indicated the presence of **SK6** as a major component. Further investigation was then not carried out.

Fraction F4 Chromatogram characteristics on normal phase TLC with 5%MeOH/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.12 and 0.24 and one brown spot under ASA reagent with the R_f value of 0.85. Its ^{1}H NMR data indicated the presence of **SK1** as a major component. Further investigation was then not carried out.

Fraction F5 Chromatogram characteristics on normal phase TLC with 5%MeOH/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.18 and 0.24 and one brown spot under ASA reagent with the R_f value of 0.85. Its ^{1}H NMR

spectrum showed the absence of aromatic and olefinic protons. Thus, it was not further investigated.

Fraction F6 Chromatogram characteristics on normal phase TLC with 5%MeOH/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.18 and 0.23 and one spot under ASA reagent with the R_f value of 0.45. This fraction was separated by column chromatography over silica gel. Elution was conducted initially with 0.5%MeOH/CH_2Cl_2 enriched with methanol until pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford five fractions as shown in **Table 59**.

Table 59 Fractions obtained from the fraction **F6** by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
F6A	0.5-1.5%MeOH/CH_2Cl_2	2.5	Colorless gum
F6B	3-20%MeOH/CH_2Cl_2	6.8	Colorless gum
F6C	20-40%MeOH/CH_2Cl_2	9.0	Colorless gum
F6D	60%MeOH/CH_2Cl_2	17.0	Yellow gum
F6E	80%MeOH/CH_2Cl_2 - 100%MeOH	7.0	Yellow gum

Fraction F6A Chromatogram characteristics on normal phase TLC with 2%Acetone/CH_2Cl_2 showed two purple spots under ASA reagent with the R_f values of 0.39 and 0.48. Because of low quantity, it was not further investigated.

Fraction F6B Chromatogram characteristics on normal phase TLC with 2%Acetone/CH_2Cl_2 showed three purple spots under ASA reagent with the R_f values of 0.11, 0.39 and 0.48. Because of low quantity, it was not further investigated.

Fraction F6C Chromatogram characteristics on normal phase TLC with 10%Acetone/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.05 and 0.29 and two purple spots under ASA reagent with the R_f values of 0.16 and 0.23. Because of low quantity, it was not further investigated.

Fraction F6D (**SK11**) Chromatogram characteristics on normal phase TLC with Toluene:EtOAc:$CHCl_3$:HCOOH in a ratio of 60:30:10:1 (2 runs) showed one

UV-active spot with the R_f value of 0.33 and one brown spot under ASA reagent with the R_f value of 0.39. Further purification by precoated TLC with Toluene:EtOAc: $CHCl_3$:HCOOH in a ratio of 60:30:10:1 (4 runs) as a mobile phase afforded a yellow gum 2.6 mg. Chromatogram characteristics on normal phase with Toluene:EtOAc: $CHCl_3$: HCOOH in a ratio of 60:30:10:1 (2 runs) showed one UV-active spot with the R_f value of 0.33.

UVλ_{max}(nm)(MeOH)(log ε)	217 (4.28)
FTIR(neat):υ(cm^{-1})	3420 (OH stretching), 1697 (C=O stretching)
^{1}H NMR($CDCl_3$)(δ_{ppm})(500 MHz):	6.84 (*d*, *J* = 8.0 Hz, 1H), 6.82 (*d*, *J* = 8.0 Hz, 1H), 5.28 (*brs*, 1H), 5.27 (*brs*, 1H), 4.59 (*t*, *J* = 8.5 Hz, 2H), 3.45 (*brs*, 1H), 3.25 (*dd*, *J* = 9.0 and 4.5 Hz, 1H), 2.34 (*m*, 2H), 2.28 (*m*, 2H), 2.23 (*m*, 3H), 2.08 (*m*, 4H), 2.02 (*m*, 2H), 1.98 (*dt*, *J* = 16.0 and 3.5 Hz, 2H), 1.87 (*s*, 6H), 1.77 (*m*, 2H), 1.74 (*m*, 2H), 1.67 (*m*, 2H), 1.65 (*m*, 1H), 1.62 (*m*, 1H), 1.58 (*m*, 2H), 1.47 (*m*, 1H), 1.35 (*m*, 3H), 1.34 (*m*, 1H) 1.15 (*m*, 2H), 1.12 (*m*, 1H), 1.03 (*s*, 3H), 1.02 (*s*, 3H), 1.01 (*s*, 6H), 0.99 (s, 3H), 0.95 (*d*, *J* = 6.0 Hz, 6H), 0.89 (*s*, 6H), 0.83 (*s*, 3H), 0.82 (*s*, 3H), 0.76 (*s*, 3H)
^{13}C NMR($CDCl_3$)(δ_{ppm})(125 MHz):	171.78, 171.77, 148.79, 148.63, 146.63, 142.41, 142.09, 126.38, 123.01, 122.58, 116.73, 115.79, 78.93, 75.93, 67.01, 50.58, 50.22, 50.07, 48.02, 45.57, 44.45, 39.29, 38.87, 37.97, 37.81, 37.60, 33.40, 31.74, 30.10, 29.26, 28.64, 28.01, 27.06, 26.70, 25.56, 22.77, 22.74, 22.19, 19.15, 18.97, 18.26, 17.10, 15.65, 15.28, 12.46

DEPT135° ($CDCl_3$)(δ_{ppm})	CH:	148.79, 146.63, 116.73, 115.79, 78.93, 75.93, 67.01, 50.58, 44.45, 33.40
	CH_2:	45.57, 39.29, 31.74, 30.10, 29.26, 28.64, 27.06, 26.70, 25.56, 22.77, 22.74, 18.26
	CH_3:	28.01, 22.19, 19.15, 18.97, 17.10, 15.65, 15.28, 12.46

Fraction F7F Chromatogram characteristics on normal phase TLC with 20%Acetone/CH_2Cl_2 showed one UV-active spot with the R_f value of 0.44 and two purple spots under ASA reagent with the R_f values of 0.51 and 0.61. Its ^{1}H NMR spectrum displayed the absence of aromatic and olefinic protons. Thus, it was not further investigated.

Fraction G Chromatogram characteristics on normal phase TLC with 20%Acetone/CH_2Cl_2 showed three UV-active spots with the R_f values of 0.09, 0.12 and 0.19. Its ^{1}H NMR spectrum showed sugar as major components. Thus, it was not further investigated.

1.2.4 Chemical investigation from the leaves of *G. prainiana*

1.2.4.1 Isolation and extraction

The leaves of *Garcinia prainiana* (0.80 kg), cut into small segments, were extracted with MeOH (4 L) for three times over the period of 3, 7 and 30 days at room temperature. After filtration, the filtrate was evaporated to dryness under reduced pressure to give a crude methanol extract as a dark brown gum in 56.25 g.

1.2.4.2 Chemical investigation of the crude methanol extract of the leaves of *G. prainiana*

The crude methanol extract was primarily tested for its solubility in various solvents at room temperature. The results were demonstrated in **Table 60**.

Table 60 Solubility of the crude extract in various solvents at room temperature

Solvent	Solubility at room temperature
Petroleum ether	-
Dichloromethane	++ (green yellow solution mixed with dark brown gum)
Ethyl acetate	+ (green yellow solution mixed with dark brown gum)
Acetone	+ (green solution mixed with dark brown gum)
Methanol	++++ (brown yellow solution)
Water	+ (brown solution mixed with dark brown gum)
10% HCl	+ (brown yellow solution with dark brown gum)
10% NaOH	+++ (brown solution)
10% $NaHCO_3$	+++ (yellow solution mixed with dark brown gum)

Symbol meaning: + slightly soluble, ++ moderately soluble, +++ well soluble - insoluble

The crude methanol extract was soluble well in methanol, 10%NaOH, and 10%$NaHCO_3$. The solubility results indicated that major components were high polar and acidic compounds. Chromatogram characteristics on normal phase TLC with 20%MeOH/CH_2Cl_2 showed five UV-active spots with the R_f values of 0.23, 0.26, 0.39, 0.41 and 0.89 and showed three spots under ASA reagent with the R_f values of 0.31, 0.36 and 0.83. The crude methanol extract was then separated into two fractions by dissolving in dichloromethane. The dichloromethane soluble fraction (26.40 g) was obtained as a green gum. Its ^{1}H NMR spectrum displayed long chain hydrocarbons. Therefore, it was not further investigated. The dichloromethane insoluble fraction (29.85 g) was obtained as a dark brown gum. This fraction was separated into two fractions by dissolving in methanol. The methanol insoluble fraction (1.93 g) was obtained as a dark brown gum. Its ^{1}H NMR displayed the absence of aromatic and olefinic protons. Therefore, it was not further investigated. The methanol soluble fraction (27.91 g) was obtained a brown gum. Chromatogram characteristics on normal reverse phase TLC of the methanol soluble fraction with 20%MeOH/H_2O showed four UV-active spots with the R_f values of 0.67, 0.83, 0.83 and 0.91. Further purification by Sephadex LH-20 was performed. Elution was conducted with 100%MeOH. Fractions with the similar chromatogram characteristics were combined

and evaporated to dryness under reduced pressure to afford five fractions as shown in **Table 61**.

Table 61 Fractions obtained from the crude methanol extract by column chromatography over Sephadex LH-20

Fraction	Weight (g)	Physical appearance
H1	12.08	Brown gum
H2	10.40	Brown gum
H3	2.51	Brown yellow gum
H4	2.27	Brown yellow gum
H5	0.65	Brown gum

Fraction H1 Chromatogram characteristics on reverse phase TLC with 20% MeOH/H_2O showed no definite spot under UV-S. Its ^{1}H NMR spectrum showed the absence of aromatic and olefinic protons. Thus, it was not further investigated.

Fraction H2 Chromatogram characteristics on reverse phase TLC with 20% MeOH/H_2O showed three UV-active spots with the R_f values of 0.83, 0.87 and 0.91. Its ^{1}H NMR spectrum displayed sugar signals. Thus, it was not further investigated.

Fraction H3 Chromatogram characteristics on reverse phase TLC with 20% MeOH/H_2O showed three UV-active spots with the R_f values of 0.73, 0.83 and 0.91. It was further purified by column chromatography over reverse phase C_{18} silica gel. Elution was conducted initially with 20%MeOH/H_2O, gradually enriched with methanol until pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford eleven fractions as shown in **Table 62**.

Table 62 Fractions obtained from the fraction **H3** by column chromatography over reverse phase C_{18} silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
H3A	20%MeOH/H_2O	526.8	Brown gum
H3B	20%MeOH/H_2O	31.0	Yellow gum

Table 62 (continued)

Fraction	Mobile phase	Weight (mg)	Physical appearance
H3C	30%MeOH/H_2O	9.5	Pale yellow solid
H3D	30%MeOH/H_2O	59.3	Yellow gum
H3E	30%MeOH/H_2O	12.8	Pale yellow solid
H3F	30%MeOH/H_2O	4.8	Pale yellow gum
H3G	40%MeOH/H_2O	156.7	Brown yellow gum
H3H	40-50%MeOH/H_2O	130.2	Brown yellow gum
H3I	50-60%MeOH/H_2O	151.1	Brown yellow gum
H3J	60-80%MeOH/H_2O	230.4	Yellow gum
H3K	100%MeOH/H_2O	609.0	Brown gum

Fraction H3A Chromatogram characteristics on reverse phase TLC with 20% MeOH/H_2O showed two UV-active spots with the R_f values of 0.87 and 0.91. Its ^1H NMR spectrum displayed sugar signals. Thus, it was not further investigated.

Fraction H3B Chromatogram characteristics on reverse phase TLC with 20% MeOH/H_2O showed four UV-active spots with the R_f values of 0.67, 0.80, 0.81 and 0.88. It was further purified by column chromatography over reverse phase C_{18} silica gel. Elution was conducted with 20%MeOH/H_2O. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford three fractions as shown in **Table 63**.

Table 63 Fractions obtained from the fraction **H3B** by column chromatography over reverse phase C_{18} silica gel

Fraction	Weight (mg)	Physical appearance
H3B1	13.9	Pale yellow gum
H3B2	4.3	Pale yellow gum
H3B3	10.5	Pale yellow gum

Fraction H3B1 Chromatogram characteristics on reverse phase TLC with 20%MeOH/H_2O showed one UV-active spot with the R_f value of 0.88. Because the

^{1}H NMR data indicated the presence of many compounds, it was not further investigated.

Fraction H3B2 Chromatogram characteristics on reverse phase TLC with 20%MeOH/H_2O showed two UV-active spots with the R_f values of 0.80 and 0.66. Because the ^{1}H NMR data indicated the presence of many compounds, it was not further investigated.

Fraction H3B3 Chromatogram characteristics on reverse phase TLC with 20%MeOH/H_2O showed one UV-active spot with the R_f value of 0.66. Because the ^{1}H NMR data indicated the presence of many compounds, it was not further investigated.

Fraction H3C (**SK24**) Chromatogram characteristics on reverse phase TLC with 20%MeOH/H_2O showed one UV-active spot with the R_f value of 0.76.

Melting point (°C)		267-269 °C
$[\alpha]_D^{28}$		-42.7° (c = 1.00, MeOH)
UV λ_{max}(nm)(MeOH)(log ε)		339 (2.97), 279 (3.07), 224 (4.10)
FTIR(neat):υ(cm^{-1})		3260 (OH stretching), 1730, 1650 (C=O stretching)
^{1}H NMR (CD_3OD)(δ_{ppm})(500 MHz):		6.93 (*brs*, 1H), 6.78 (*d*, *J* = 8.4 Hz, 2H), 6.23 (*d*, *J* = 2.4 Hz, 1H), 6.19 (*d*, *J* = 2.4 Hz, 1H), 5.33 (*dd*, *J* = 12.9 and 3.3 Hz, 1H), 5.00 (*m*, 1H), 3.78 (*m*, 1H), 3.50 (*m*, 3H), 3.11 (*dd*, *J* = 17.4 and 12.9 Hz, 1H), 2.75 (*dd*, *J* = 17.4 and 3.3 Hz, 1H)
^{13}C NMR (CD_3OD)(δ_{ppm})(125 MHz):		197.17, 182.00, 165.71, 165.65, 163.14, 145.52, 145.10, 130.19, 117.91, 114.91, 113.42, 103.60, 99.75, 96.75, 95.65, 79.25, 76.22, 75.26, 73.06, 72.04, 42.76
DEPT135° (CD_3OD)(δ_{ppm})	CH:	117.91, 114.91, 113.42, 99.75, 96.75, 95.65, 79.25, 76.22, 75.26, 73.06, 72.04
	CH_2:	42.76

Fraction H3D Chromatogram characteristics on reverse phase TLC with 20% MeOH/H_2O showed three UV-active spots with the R_f values of 0.55, 0.67 and 0.77. It was further purified by column chromatography over reverse phase C_{18} silica gel. Elution was conducted initially with 20%MeOH/H_2O, gradually enriched with methanol until pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford five fractions as shown in **Table 64**.

Table 64 Fractions obtained from the fraction **H3D** by column chromatography over reverse phase C_{18} silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
H3D1	20%MeOH/H_2O	10.0	White solid
H3D2	20%MeOH/H_2O	23.7	Yellow gum
H3D3	20%MeOH/H_2O	8.2	Yellow gum
H3D4	30%MeOH/H_2O	4.2	Yellow gum
H3D5	30%MeOH/H_2O-100%MeOH	11.1	Pale yellow gum

Fraction H3D1 Chromatogram characteristics on reverse phase TLC with 50%MeOH/H_2O showed no definite spot under UV-S. Its ^{1}H NMR spectrum showed the absence of aromatic and olefinic protons. Thus, it was not further investigated.

Fraction H3D2 Chromatogram characteristics on reverse phase TLC with 50%MeOH/H_2O showed two UV-active spots with the R_f values of 0.80 and 0.86. Its the ^{1}H NMR data indicated the presence of **SK24** as a major component. It was not further investigated.

Fraction H3D3 Chromatogram characteristics on reverse phase TLC with 50%MeOH/H_2O showed two UV-active spots with the R_f values of 0.76 and 0.80. Further purification by Sephadex LH-20 was performed. Elution was conducted with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford three fractions as shown in **Table 65**.

Table 65 Fractions obtained from **H3D3** by column chromatography over Sephadex LH-20

Fraction	Weight (mg)	Physical appearance
H3D3A	3.5	Pale Yellow gum
H3D3B	1.5	Pale Yellow gum
H3D3C	3.5	Pale yellow gum

Fraction H3D3A Chromatogram characteristics on reverse phase TLC with 30%MeOH/H_2O showed two UV-active spots with the R_f values of 0.50 and 0.68. Because of the minute quantity, it was not further investigated.

Fraction H3D5B Chromatogram characteristics on reverse phase TLC with 30% MeOH/H_2O showed one UV-active spot with the R_f value of 0.68. Its the ^1H NMR data indicated the presence of **SK23** as a major component. It was not further investigated

Fraction H3D3C Chromatogram characteristics on reverse phase TLC with 30%MeOH/H_2O showed three UV-active spots with the R_f values of 0.45, 0.54 and 0.68. Because of the minute quantity, it was not further investigated.

Fraction H3D4 Chromatogram characteristics on reverse phase TLC with 50%MeOH/H_2O showed two UV-active spots with the R_f values of 0.76 and 0.80. The ^1H NMR data indicated the presence of **SK23** as a major component. It was not further investigated.

Fraction H3D5 Chromatogram characteristics on reverse phase TLC with 50%MeOH/H_2O showed one UV-active spot with the R_f value of 0.76. The ^1H NMR data indicated the presence of **SK23** as a major component. It was not further investigated.

Fraction H3E (**SK23**) Chromatogram characteristics on reverse phase TLC with 20% MeOH/H_2O showed one UV-active spot with the R_f value of 0.67.

Melting point (°C)	252-255 °C
$[\alpha]_D^{28}$	-80.9º (c = 0.68, MeOH)
UV λ_{max}(nm)(MeOH)(log ε)	335 (2.11), 282 (2.91), 223 (3.13)

FTIR(neat):υ(cm^{-1})		3220 (OH stretching), 1730, 1644 (C=O stretching)
^{1}H NMR (CD_3OD)(δ_{ppm})(500 MHz):		7.32 (*d*, *J* = 8.4 Hz, 2H), 6.81 (*d*, *J* = 8.4 Hz, 2H), 6.23 (*brs*, 1H), 6.19 (*brs*, 1H), 5.39 (*dd*, *J* = 12.6 and 2.7 Hz, 1H), 5.00 (*d*, *J* = 7.2 Hz, 1H), 3.80 (*m*, 2H), 3.78 (*d*, *J* = 9.0 Hz, 1H), 3.50 (*m*, 3H), 3.17 (*dd*, *J* = 17.1 and 12.6 Hz, 1H), 2.74 (*dd*, *J* = 17.1 and 2.7 Hz, 1H)
^{13}C NMR (CD_3OD)(δ_{ppm})(125 MHz):		197.18, 182.00, 165.73, 163.80, 163.24, 157.64, 129.51, 127.68, 114.95, 103.58, 99.77, 96.78, 95.60, 79.25, 76.23, 75.28, 73.07, 72.04, 42.75
DEPT135° (CD_3OD)(δ_{ppm})	CH:	127.68, 114.95, 99.77, 96.78, 95.60, 79.25, 76.23, 75.28, 73.07, 72.04
	CH_2:	42.75

Therefore, it was subjected to acetylation reaction in acetic anhydride (6 ml) in the presence of pyridine (2 ml). The reaction mixture was stirred at room temperature overnight. After working up, the acetate derivative (**H3EAc**) was obtained as a pale yellow gum (15.1 mg). Chromatogram characteristics on normal phase TLC with 10%MeOH/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.12 and 0.62. Further purification by precoated TLC was carried out with 10%MeOH/CH_2Cl_2 (3 runs) as a mobile phase afforded a colorless gum (1.5 mg). Its chromatogram characteristics on normal phase TLC with 10%MeOH/CH_2Cl_2 (3 runs) showed one UV-active spot with the R_f value of 0.62. Because the ^{1}H NMR spectrum displayed broad signals, it was not further investigated.

Fraction H3F Chromatogram characteristics on normal reverse phase TLC with 20%MeOH/H_2O showed one UV-active spot with the R_f value of 0.67. The ^{1}H NMR data indicated the presence of **SK23** as a major component. It was not further investigated.

Fraction H3G Chromatogram characteristics on reverse phase TLC with 20% MeOH/H_2O showed four UV-active spots with the R_f values of 0.44, 0.67, 0.77 and 0.88. The ^1H NMR data indicated the presence of **SK23** and **SK24** as major components. It was not further investigated.

Fraction H3H Chromatogram characteristics on reverse phase TLC with 20% MeOH/H_2O showed four UV-active spots with the R_f values of 0.17, 0.22, 0.62 and 0.67. It was further purified by column chromatography over reverse phase C_{18} silica gel. Elution was conducted initially with 30%MeOH/H_2O, gradually enriched with methanol until pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford six fractions as shown in **Table 66**.

Table 66 Fractions obtained from the fraction **H3H** by column chromatography over reverse phase C_{18} silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
H3H1	30%MeOH/H_2O	12.4	Yellow gum
H3H2	30%MeOH/H_2O	18.5	Yellow gum
H3H3	30%MeOH/H_2O	17.3	Yellow gum
H3H4	30-50%MeOH/H_2O	31.0	Yellow gum
H3H5	50-70%MeOH/H_2O	24.0	Brown yellow gum
H3H6	70%MeOH/H_2O-100%MeOH	35.8	Brown yellow gum

Fraction H3H1 Chromatogram characteristics on reverse phase TLC with 30%MeOH/H_2O showed one UV-active spot with the R_f value of 0.88. The ^1H NMR data were similar to those of **SK24**. It was not further investigated.

Fraction H3H2 Chromatogram characteristics on reverse phase TLC with 30%MeOH/H_2O showed two UV-active spots with the R_f values of 0.71 and 0.88. The ^1H NMR data indicated the presence of **SK23** and **SK24** as major components. It was not further investigated.

Fraction H3H3 Chromatogram characteristics on reverse phase TLC with 30%MeOH/H_2O showed three UV-active spots with the R_f values of 0.66, 0.71 and 0.88. It was further purified by column chromatography over reverse phase C_{18} silica

gel. Elution was conducted initially with 20%MeOH/H_2O, gradually enriched with methanol until pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford five fractions as shown in **Table 67**.

Table 67 Fractions obtained from the fraction **H3H3** by column chromatography over reverse phase C_{18} silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
H3H3A	20%MeOH/H_2O	4.6	Pale yellow gum
H3H3B	20%MeOH/H_2O	14.1	Pale yellow gum
H3H3C	20%MeOH/H_2O	12.9	Pale yellow gum
H3H3D	20%MeOH/H_2O	6.6	Pale yellow gum
H3H3E	20%MeOH/H_2O-100%MeOH	16.1	Brown yellow gum

Fraction H3H3A Chromatogram characteristics on reverse phase TLC with 20%MeOH/H_2O showed one UV-active spot with the R_f value of 0.80. Its ^1H NMR spectrum displayed sugar signals. It was not further investigated.

Fraction H3H3B Chromatogram characteristics on reverse phase TLC with 20%MeOH/H_2O showed one UV-active spot with the R_f value of 0.77. The ^1H NMR data were similar to those **SK24**. It was not further investigated.

Fraction H3H3C Chromatogram characteristics on reverse phase TLC with 20%MeOH/H_2O showed two UV-active spots with the R_f values of 0.67 and 0.77. The ^1H NMR data were similar to those of **SK23** and **SK24**. It was not further investigated.

Fraction H3H3D Chromatogram characteristics on reverse phase TLC with 20%MeOH/H_2O showed one UV-active spot with the R_f value of 0.50. The ^1H NMR data indicated the presence of **SK23** as a major component. It was not further investigated.

Fraction H3H3E Chromatogram characteristics on normal reverse phase TLC with 20%MeOH/H_2O showed none of well separated spots under UV-S. It was not further investigated.

Fraction H3H4 Chromatogram characteristics on reverse phase TLC with 30%MeOH/H_2O showed two UV-active spots with the R_f values of 0.66 and 0.71. The ^1H NMR data were similar to those of **SK23**. It was not further investigated.

Fraction H3H5 Chromatogram characteristics on reverse phase TLC with 30%MeOH/H_2O showed two UV-active spots with the R_f values of 0.22 and 0.62. The ^1H NMR data displayed morelloflavone as a major component (Salae, 2006). It was not further investigated.

Fraction H3H6 Chromatogram characteristics on reverse phase TLC with 30%MeOH/H_2O showed none of well separated spots under UV-S. It was not further investigated.

Fraction H3I Chromatogram characteristics on normal phase TLC with 20% MeOH/H_2O showed four UV-active spots with the R_f values of 0.17, 0.22, 0.50 and 0.62. It was further purified by column chromatography over reverse phase C_{18} silica gel. Elution was conducted initially with 20%MeOH/H_2O, gradually enriched with methanol until pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford four fractions as shown in **Table 68**.

Table 68 Fractions obtained from the fraction **H3I** by column chromatography over reverse phase C_{18} silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
H3I1	30%MeOH/H_2O	11.8	Pale yellow gum
H3I2	30%MeOH/H_2O	13.3	Pale yellow gum
H3I3	40-60%MeOH/H_2O	68.5	Yellow gum
H3I4	60%MeOH/H_2O-100%MeOH	9.1	Brown yellow gum

Fraction H3I1 Chromatogram characteristics on reverse phase TLC with 40%MeOH/H_2O showed three UV-active spots with the R_f values of 0.71, 0.77 and 0.84. The ^1H NMR data indicated the presence of **SK23** and **SK24** as major components. It was not further investigated.

Fraction H3I2 Chromatogram characteristics on reverse phase TLC with 40%MeOH/H_2O showed one UV-active spot with the R_f value of 0.75. The ^1H NMR data were similar to those of **SK24**. It was not further investigated.

Fraction H3I3 Chromatogram characteristics on reverse phase TLC with 40%MeOH/H_2O showed four UV-active spots with the R_f values of 0.22, 0.33, 0.44 and 0.55. The ^1H NMR spectrum was similar to that of fraction **H3H5**. It was not further investigated.

Fraction H3I4 Chromatogram characteristics on reverse phase TLC with 40%MeOH/H_2O showed none of well separated spots under UV-S. It was not further investigated.

Fraction H3J Chromatogram characteristics on reverse phase TLC with 20% MeOH/H_2O showed three UV-active spots with the R_f values of 0.11, 0.17 and 0.22. The ^1H NMR spectrum was similar to that of fraction **H3H5**. It was not further investigated.

Fraction H3K Chromatogram characteristics on reverse phase TLC with 20% MeOH/H_2O showed none of well separated spots under UV-S. It was not further investigated.

Fraction H4 Chromatogram characteristics on reverse phase TLC with 20% MeOH/H_2O showed four UV-active spots with the R_f values of 0.22, 0.38, 0.45 and 0.73. The ^1H NMR data were similar to those of fraction **H3H5**. It was not further investigated.

Fraction H5 Chromatogram characteristics on reverse phase TLC with 20% MeOH/H_2O showed none of well separated spots under UV-S. It was not further investigated.

CHAPTER 1.3

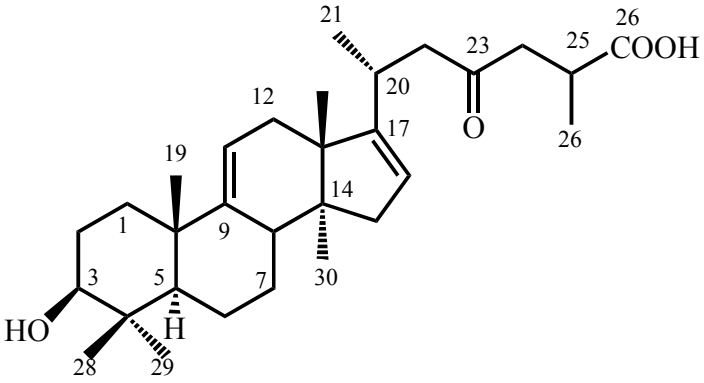
RESULTS AND DISSCUSSION

The crude methanol extract from the twigs of *G. hombroniana* was separated by chromatographic methods to yield eight triterpenes (**SK1**, **SK2**, **SK3**, **SK9**, **SK11**, **SK12**, **SK19** and **SK21**), nine xanthones (**SK4**, **SK5**, **SK8**, **SK10, SK13**, **SK16**, **SK18**, **SK20** and **SK22**), two benzoic acid derivatives (**SK7** and **SK17**) and one biflavone (**SK6**) while that from the leaves of *G. prainiana* afforded two flavonone glucosides (**SK23** and **SK24**). Their structures were determined by analysis of 1D and 2D NMR spectroscopic data and comparison of the NMR data with those reported in the literatures.

1.3.1 Triterpenes

1.3.1.1 Compound SK1

Compound **SK1** was obtained as a white solid, melting at 221-224 °C. Its UV showed an absorption band at λ_{max} 207 nm while its IR spectrum exhibited absorption bands at 3365 and 1696 cm^{-1} due to hydroxyl and carbonyl groups. The ^{1}H and ^{13}C NMR data (**Table 69**) (**Figures 1** and **2**) contained signals of olefinic protons [δ_H 5.28 (*d*, *J* = 6.0 Hz, 1H), and 5.21 (*s*, 1H)], one oxymethine proton (δ_H 3.21, *dd*, *J* = 12.0 and 6.0 Hz, 1H) and seven methyl groups [δ_H 1.18 (*d*, *J* = 6.9 Hz, 3H), 1.05 (*s*, 3H), 1.02 (*d*, *J* = 6.6 Hz, 3H), 0.99 (*s*, 3H), 0.79 (*s*, 6H) and 0.75 (*s*, 3H)]. Comparison of its NMR data, TLC chromatogram and optical rotation ($[\alpha]_D^{28}$ +51.5° (c = 0.20, MeOH)) with those of garcihombronane D ($[\alpha]_D^{29}$+58° (c = 0.34, MeOH)) which was isolated from the pericarps of *G. hombroniana* (Rukachaisirikul, 2000) indicated that **SK1** was garcihombronane D.



(SK1)

Table 69 The NMR data of compound **SK1** and garcihombronane D in $CDCl_3$+CD_3OD

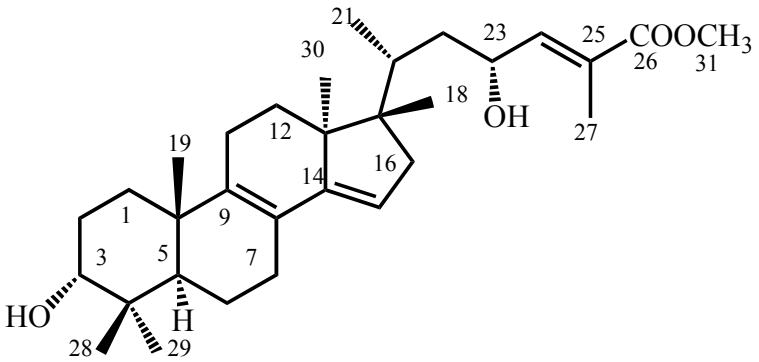
Position	**SK1**		garcihombronane D	
	δ_H (*mult, J* Hz)	δ_C (C-Type)	δ_H (*mult, J* Hz)	δ_C
1	1.57-1.29 (*m*, 2H)	36.16 (CH_2)	1.57-1.28 (*m*, 2H)	36.1
2	1.80-1.60 (*m*, 2H)	27.69 (CH_2)	1.78-1.57 (*m*, 2H)	27.6
3	3.21 (*dd,* 12.0, 5.0, 1H)	78.84 (CH)	3.20 (*dd,* 9.6, 4.0, 1H)	77.4
4	-	39.13 (C)	-	39.0
5	0.89 (*m*, 1H)	52.53 (CH)	0.82 (*dd*, 6.2, 2.0, 1H)	52.4
6	1.80-1.60 (*m*, 1H)	21.05 (CH_2)	1.78-1.28 (*m*, 1H)	21.0
	1.57-1.29 (*m*, 1H)		1.57-1.28 (*m*, 1H)	
7	1.57-1.29 (*m*, 1H)	28.04 (CH)	1.57-1.28 (*m*, 1H)	27.8
8	2.39-2.30 (*m*, 1H)	39.95 (CH)	2.40-2.28 (*m*, 1H)	39.7
9	-	149.52 (C)	-	149.5
10	-	39.64 (C)	-	39.4
11	5.28 (*d*, 6.0, 1H)	114.44 (CH)	5.30 (*d*, 6.4, 1H)	113.9
12	2.39-2.30 (*m*, 1H)	31.20 (CH_2)	2.40-2.28 (*m*, 1H)	31.0
	1.80-1.60 (*m*, 1H)		1.78-1.57 (*m*, 1H)	
13	-	50.98 (C)	-	50.7
14	-	46.66 (C)	-	46.4
15	2.07 (*d*, 14.0, 1H)	40.78 (CH_2)	2.07 (*brd*, 15.2, 1H)	40.5
	1.80 (*m*, 1H)		1.82 (*dd*, 15.2, 3.6, 1H)	
16	5.21 (*s*, 1H)	120.40 (CH)	5.29 (*s*, 1H)	120.1
17	-	155.72 (C)	-	155.4
18	0.75 (*s*, 3H)	19.38 (CH_3)	0.75 (*s*, 3H)	19.2

Table 71 (continued)

Position	**SK1**		garcihombronane D	
	δ_H (*mult, J* Hz)	δ_C (C-Type)	δ_H (*mult, J* Hz)	δ_C
19	1.05 (*s*, 3H)	22.12 (CH_3)	1.04 (*s*, 3H)	22.1
20	2.66-2.61 (*m*, 1H)	28.21 (CH)	2.65-2.62 (*m*, 1H)	27.7
21	1.02 (*d*, 6.6, 3H)	21.24 (CH_3)	1.02 (*d*, 7.0, 3H)	20.9
22	2.67 (*m*, 1H)	49.23 (CH_2)	2.68 (*dd*, 18.0, 6.0, 1H)	49.2
	2.49 (*m*, 1H)		2.49 (*dd*, 18.0, 10.0, 1H)	
23	-	208.49 (C=O)	-	207.8
24	2.85 (*m*, 1H)	46.58 (CH_2)	2.85 (*dd*, 20.0, 8.0, 1H)	46.3
	2.45 (*m*, 1H)		2.46 (*dd*, 20.0, 10.0, 1H)	
25	2.80-2.70 (*m*, 1H)	34.52 (CH)	2.80-2.75 (*m*, 1H)	34.3
26	-	177.65 (C=O)	-	177.1
27	1.18 (*d*, 6.9, 3H)	16.97 (CH_3)	1.16 (*d*, 7.0, 3H)	17.0
28	0.99 (*s*, 3H)	28.00 (CH_3)	0.99 (*s*, 3H)	28.4
29	0.79 (*s*, 3H)	15.61 (CH_3)	0.79 (*s*, 3H)	15.9
30	0.79 (*s*, 3H)	19.88 (CH_3)	0.79 (*s*, 3H)	19.8

1.3.1.2 Compound SK2

Compound **SK2** was obtained as a pale yellow gum. The IR spectrum showed the presence of a hydroxyl group (3420 cm^{-1}) and a carbonyl group of an α,β-unsaturated ester (1704 cm^{-1}). In the UV spectrum, an absorption band at λ_{max} 218 nm indicated that **SK2** had an α,β-unsaturated ester chromorphore. **SK2** was identified as garcihombronane C by direct comparison of its ^1H and ^{13}C NMR (**Table 70**) data and TLC chromotogram with those of garcihombronane C which was obtained from the pericarps of *G. hombroniana* (Rukachairisikul, 2000).



(SK2)

Table 70 The NMR data of compound **SK2** and garcihombronane C in $CDCl_3$

Position	**SK2**		garcihombronane C	
	δ_H (*mult*, *J* Hz)	δ_C (C-Type)	δ_H (*mult*, *J* Hz)	δ_C
1	1.59 (*m*, 2H)	29.22 (CH_2)	1.57 (*m*, 2H)	29.19
2	1.66 (*m*, 1H)	25.58 (CH_2)	1.64 (*m*, 1H)	25.55
	1.98 (*m*, 1H)		1.97 (*m*, 1H)	
3	3.45 (*t*, 2.4, 1H)	75.85 (CH)	3.45 (*t*, 2.5, 1H)	75.83
4	-	37.60 (C)		37.57
5	1.62 (*m*, 1H)	44.75 (CH)	1.60 (*m*, 1H)	44.41
6	1.65 (*m*, 1H)	18.15 (CH_2)	1.65 (*m*, 1H)	18.11
	1.49 (*m*, 1H)		1.49 (*dt*, 11.5, 6.5, 1H)	
7	2.30 (*m*, 1H)	26.69 (CH_2)	2.33 (*m*, 1H)	26.66
	2.35 (*m*, 1H)		2.36 (*m*, 1H)	
8	-	122.85 (C)		122.82
9	-	142.36 (C)		142.37
10	-	37.81 (C)		37.78
11	2.18 (*m*, 1H)	22.72 (CH_2)	2.19 (*m*, 1H)	22.70
	2.09 (*m*, 1H)		2.06 (*m*, 1H)	
12	1.59 (*m*, 2H)	30.10 (CH_2)	1.57 (*m*, 2H)	30.06
13	-	48.01 (C)		47.97
14	-	148.79 (C)		148.76
15	5.27 (*brs*, 1H)	115.80 (CH)	5.27 (*brs*, 1H)	115.78

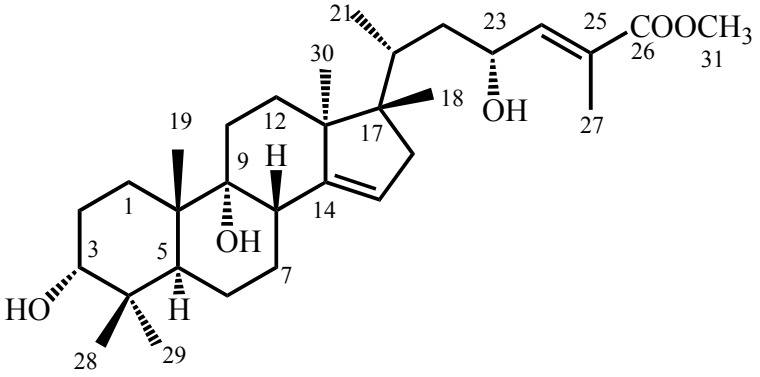
Table 70 (continued)

Position	**SK2**		garcihombronane C	
	δ_H (*mult, J* Hz)	δ_C (C-Type)	δ_H (*mult, J* Hz)	δ_C
16	1.95 (*m*, 1H) 2.30 (*brd*, 16.2, 1H)	45.54 (CH_2)	1.95 (*m*, 1H) 2.33 (*brd*, 15.5, 1H)	45.52
17		50.02 (C)		49.99
18	0.76 (*s*, 3H)	15.65 (CH_3)	0.75 (*s*, 3H)	15.62
19	1.01 (*s*, 3H)	18.95 (CH_3)	1.01 (*s*, 3H)	18.92
20	2.03 (*m*, 1H)	33.40 (CH)	2.02 (*m*, 1H)	33.37
21	0.94 (*d*, 6.6, 3H)	15.27 (CH_3)	0.95 (*d*, 7.0, 3H)	15.25
22	1.74 (*m*, 1H) 1.12 (*m*, 1H)	39.46 (CH_2)	1.74 (*ddd*, 14.0, 11.5, 1.5, 1H) 1.12 (*ddd*, 14.0, 11.5, 2.5, 1H)	39.41
23	4.57 (*ddd*, 11.0, 8.4, 2.4, 1H)	66.89 (CH)	4.57 (*ddd*, 11.5, 8.0, 2.5, 1H)	66.87
24	6.72 (*qd*, 8.1, 1.5, 1H)	144.49 (CH)	6.72 (*qd*, 8.0, 1.5, 1H)	144.42
25	-	127.09 (C)	-	127.06
26	-	168.49 (C=O)	-	168.46
27	1.87 (*d*, 1.5, 3H)	12.73 (CH_3)	1.87 (*d*, 1.5, 3H)	12.72
28	0.89 (*s*, 3H)	22.19 (CH_3)	0.89 (*s*, 3H)	22.17
29	0.99 (*s*, 3H)	27.99 (CH_3)	0.99 (*s*, 3H)	27.98
30	0.90 (*s*, 3H)	17.08 (CH_3)	0.90 (*s*, 3H)	17.06
31	3.76 (*s*, 3H)	51.95 (CH_3)	3.76 (*s*, 3H)	51.94

1.3.1.3 Compound SK3

Compound **SK3** was obtained as a pale yellow gum. Its IR and UV spectral data were similar to those of **SK2**. Their NMR data were also similar except for the fact that two olefinic carbons of a tetrasubstituted double bond were replaced by signals of methine and oxymethine carbons (δ_C 39.15 and 75.56). Comparison of its ^1H, ^{13}C NMR data, TLC chromatogram and optical rotation ($[\alpha]_D^{29}$-42.3° (c = 0.41,

MeOH)) with the previously reported data of garcihombronane B ($[\alpha]_D^{29}$-48° (c = 0.42, MeOH)) (Rukachaisirikul, 2000) indicated that **SK3** was garcihombronane B.



(SK3)

Table 71 The NMR data of compound **SK3** and garcihombronane B in $CDCl_3$

Position	**SK3**		garcihombronane B	
	δ_H (*mult, J* Hz)	δ_C (C-Type)	δ_H (*mult, J* Hz)	δ_C
1	1.90 (*m*, 1H)	23.60 (CH_2)	1.90 (*m*, 1H)	23.54
	1.16 (*m*, 1H)		1.16 (*m*, 1H)	
2	1.63 (*m*, 1H)	25.09 (CH_2)	1.64 (*m*, 1H)	25.05
	1.90 (*m*, 1H)		1.92 (*m*, 1H)	
3	3.37 (*brs*, 1H)	76.14 (CH)	3.39 (*brs*, 1H)	76.09
4	-	37.51 (C)	-	37.48
5	1.98 (*m*, 1H)	38.95 (CH)	1.98 (*m*, 1H)	38.93
6	1.52 (*m*, 1H)	20.78 (CH_2)	1.55 (*m*, 1H)	20.73
	1.36 (*m*, 1H)		1.36 (*m*, 1H)	
7	1.39 (*m*, 1H)	25.59 (CH_2)	1.41 (*m*, 1H)	25.55
	1.95 (*m*, 1H)		1.96 (*m*, 1H)	
8	2.35 (*m*, 1H)	39.15 (CH)	2.35 (*m*, 1H)	39.09
9	-	75.56 (C)	-	75.4
10	-	42.15 (C)	-	42.11
11	1.78 (*m*, 1H)	29.58 (CH_2)	1.80 (*m*, 1H)	29.55
	1.65 (*m*, 1H)		1.66 (*m*, 1H)	

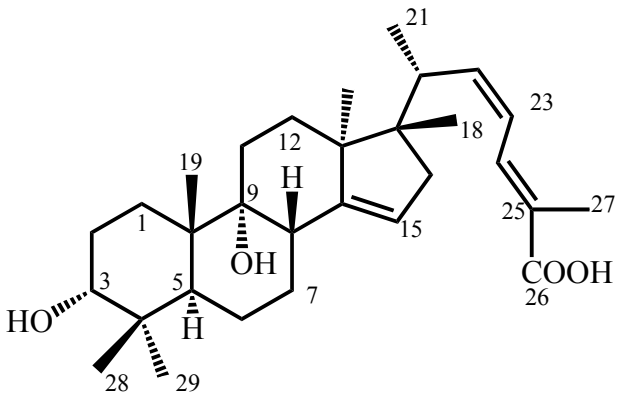
Table 71 (continued)

Position	**SK3**		garcihombronane B	
	δ_H (*mult, J* Hz)	δ_C (C-Type)	δ_H (*mult, J* Hz)	δ_C
12	1.65 (*m*, 1H)	28.97 (CH_2)	1.66 (*m*, 1H)	28.95
	1.52 (*m*, 1H)		1.53 (*m*, 1H)	
13	-	49.07 (C)	-	49.03
14	-	153.60 (C)	-	153.52
15	5.32 (*brs*, 1H)	120.34 (CH)	5.34 (*brs*, 1H)	120.36
16	2.27 (*m*, 1H)	44.71 (CH_2)	2.30 (*m*, 1H)	44.68
	1.78 (*m*, 1H)		1.80 (*m*, 1H)	
17	-	53.99 (C)	-	53.95
18	0.75 (*s*, 3H)	15.34 (CH_3)	0.76 (*s*, 3H)	15.30
19	0.90 (*s*, 3H)	16.43 (CH_3)	0.91 (*s*, 3H)	16.37
20	2.28 (*m*, 1H)	32.96 (CH)	2.26 (*m*, 1H)	32.91
21	0.91 (*d*, 6.6, 3H)	15.09 (CH_3)	0.92 (*d*, 6.5, 3H)	15.05
22	1.69 (*m*, 1H)	39.02 (CH)	1.70 (*m*, 1H)	39.07
23	4.54 (*t*, 8.4, 1H)	66.68 (CH)	4.56 (*ddd*, 10.5, 8.0, 2.0, 1H)	66.69
24	6.71 (*qd*, 8.1, 1.5, 1H)	144.79 (CH)	6.70 (*qd*, 8.0, 1.5, 1H)	144.58
25	-	126.79 (C)	-	126.9
26	-	168.62 (C=O)	-	168.51
27	1.85 (*d*, 1.5, 3H)	12.69 (CH_3)	1.87 (*d*, 1.5, 3H)	12.68
28	0.84 (*s*, 3H)	22.04 (CH_3)	0.85 (*s*, 3H)	21.99
29	0.95 (*s*, 3H)	28.51 (CH_3)	0.96 (*s*, 3H)	28.47
30	1.23 (*s*, 3H)	19.46 (CH_3)	1.24 (*s*, 3H)	19.36
31	3.77 (*s*, 3H)	51.95 (CH_3)	3.75 (*s*, 3H)	51.93

1.3.1.4 Compound SK12

Compound **SK12** was obtained as a colorless gum. The UV spectrum (λ_{max} 266 nm) showed the presence of an α,β-unsaturated carboxylic acid chromophore. Its IR spectrum exhibited absorption bands at 3443 cm^{-1} (a hydroxyl group) and 1681 cm^{-1} (a carbonyl group of carboxylic acid). The ^1H NMR data (**Table 72**) (**Figure 7**)

contained signals of olefinic protons [δ_H, 7.58 (*d*, *J* = 12.0 Hz, 1H), 6.14 (*d*, *J* = 11.5 Hz, 1H), 5.90 (*d*, *J* = 11.5 Hz, 1H) and 5.27 (*brs*, 1H)], one oxymethine proton (δ_H 3.31, *brs*, 1H) and seven methyl groups [δ_H 1.87 (*s*, 3H), 1.03 (*s*, 3H), 0.90 (*s*, 3H), 0.86 (*d*, *J* = 7.0 Hz, 3H), 0.84 (*s*, 3H), 0.82 (*s*, 3H), 0.78 (*s*, 3H)]. Comparison of the ^{1}H, ^{13}C NMR data, TLC chromatogram and optical rotation ($[\alpha]_D^{26}$ -150.8° (c = 0.05, MeOH)) with the previously reported data of garcihombronane F ($[\alpha]_D^{27}$-153.8° (c = 0.03, MeOH)), isolated from the leaves of *G. hombroniana* (Rukachaisirikrul, 2005), suggested that **SK12** was garcihombronane F.



(SK12)

Table 72 The NMR data of compound **SK12** and garcihombronane F in $CDCl_3$

Position	**SK12**		garcihombronane F	
	δ_H (*mult*, *J* Hz)	δ_C (C-Type)	δ_H (*mult*, *J* Hz)	δ_C
1	1.85 (*m*, 1H)	23.55 (CH_2)	1.88 (*m*, 1H)	23.47
	1.12 (*m*, 1H)		1.15 (*m*, 1H)	
2	1.94 (*m*, 1H)	25.12 (CH_2)	α : 2.00 (*m*, 1H)	24.98
	1.85 (*m*, 1H)		β : 1.90 (*m*, 1H)	
3	3.31 (*brs*, 1H)	76.10 (CH)	3.38 (*brs*, 1H)	76.10
4	-	37.52 (C)	-	37.48
5	1.93 (*m*, 1H)	39.04 (CH)	1.97 (*m*, 1H)	38.94

Table 72 (continued)

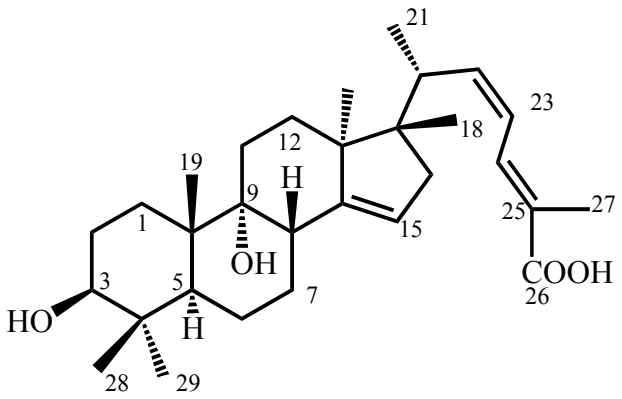
Position	**SK12**		garcihombronane F	
	δ_H (*mult*, *J* Hz)	δ_C (C-Type)	δ_H (*mult*, *J* Hz)	δ_C
6	1.56 (*m*, 1H)	25.12 (CH_2)	α : 1.62 (*m*, 1H)	25.02
	1.38 (*m*, 1H)		β : 1.42 (*m*, 1H)	
7	1.51 (*m*, 1H)	20.71 (CH_2)	α : 1.55 (*m*, 1H)	20.63
	1.38 (*m*, 1H)		β : 1.36 (*m*, 1H)	
8	2.25 (*m*, 1H)	39.16 (CH)	2.31 (*m*, 1H)	39.06
9	-	75.55 (C)	-	75.62
10	-	42.30 (C)	-	42.24
11	1.50 (*m*, 1H)	29.51 (CH_2)	α : 1.54 (*m*, 1H)	29.48
	1.78 (*m*, 1H)		β : 1.83 (*m*, 1H)	
12	1.51 (*m*, 1H)	27.55 (CH_2)	1.55 (*m*, 1H)	27.48
	1.78 (*m*, 1H)		1.37 (*m*, 1H)	
13	-	49.26 (C)	-	49.20
14	-	153.23 (C)	-	153.52
15	5.27 (*brs*, 1H)	119.89 (CH)	5.34 (*brs*, 1H)	119.87
16	2.30 (*dd*, 15.0, 4.5, 1H)	44.18 (CH_2)	α : 2.37 (*dd*, 15, 3.5, 1H)	44.14
	1.80 (*m*, 1H)		β : 1.85 (*m*, 1H)	
17	-	53.72 (C)	-	53.66
18	0.82 (*s*, 3H)	15.63 (CH_3)	0.89 (*s*, 3H)	15.59
19	0.84 (*s*, 3H)	16.37 (CH_3)	0.90 (*s*, 3H)	16.34
20	3.14 (*dq*, 14.0, 7.0, 1H)	36.96 (CH)	3.21 (*dq*, 12.0, 7.5, 1H)	36.89
21	0.86 (*d*, 7.0, 3H)	17.71 (CH_3)	0.93 (*d*, 7.5, 1H)	17.73
22	5.90 (*d*, 11.5, 1H)	144.05 (CH)	5.97 (*t*, 12.0, 1H)	144.09
23	6.14 (*d*, 11.5, 1H)	121.88 (CH)	6.21 (*t*, 12.0, 1H)	121.86
24	7.58 (*d*, 12.0, 1H)	134.94 (CH)	7.66 (*d*, 12.0, 1H)	134.97
25	-	126.25 (C)	-	126.24
26	-	173.20 (C=O)	-	173.12
27	1.87 (*s*, 3H)	12.17 (CH_3)	1.94 (*s*, 3H)	12.13
28	0.78 (*s*, 3H)	22.02 (CH_3)	0.84 (*s*, 3H)	22.00
29	0.90 (*s*, 3H)	28.43 (CH_3)	0.96 (*s*, 3H)	28.44
30	1.03 (*s*, 3H)	18.82 (CH_3)	1.10 (*s*, 3H)	18.69

1.3.1.5 Compound SK9

Compound **SK9** was obtained as a colorless gum. It showed the molecular formula $C_{30}H_{46}O_4$ by EI-MS (**Figure 11**). The IR spectrum showed similar absorption bands to those of **SK12**: a hydroxyl group (3404 cm^{-1}) and a carbonyl group of an α,β-unsaturated carboxylic acid (1713 cm^{-1}). In the UV spectrum, an absorption band at λ_{max} 257 nm indicated that **SK9** had the same chromophore as **SK12**. The ^1H NMR spectrum was also similar to that of **SK12**: two trisubstituted double bonds [δ_H 7.65 (*t*, *J* = 11.5 Hz, 1H), and 5.35 (*brs*, 1H)], one disubstituted double bond [δ_H 6.22 and 5.98 (*t*, *J* = 11.5 Hz, 1H each), one oxymethine proton (δ_H 3.23, *dd*, *J* = 11.0 and 4.0 Hz, 1H) and seven methyl groups [δ_H 1.97 (*s*, 3H), 1.08 (*s*, 3H), 0.99 (*s*, 3H), 0.89 (*s*, 3H), 0.93 (*d*, *J* = 7.0 Hz, 1H), and 0.79 (*s*, 6H)]. The ^{13}C NMR (**Figure 10**) and DEPT experiment showed the same numbers and type of carbons as those of **SK12**. Thus, **SK9** was initially assigned to have the same core structure as **SK12** with one trisubstituted double bond at C-14/C-15 and the same side chain at C-17. This result was confirmed by HMBC data (**Table 73**). The positions of the seven methyl groups were established using the data from HMBC spectrum.

The relative stereochemistry was established based on the following ^1H-^1H NOESY data. In the side chain, H-22 (δ_H 5.38) showed correlation with H-23 (δ_H 6.22) while H-24 did not give a cross peak with Me-27 (δ_H 1.97). Therefore, the configurations of double bonds at C-22/C-23 and C-24/C-25 were *Z*- and *E*-, respectively. Since the oxymethine proton, H-3 (δ_H 3.23), appeared as *doublet* of *doublet*, it was located at an α-position. Me-29 (δ_H 0.99) showed correlations with both axial H-3 and H-5 (δ_H 1.52), suggesting that Me-29 was *cis* to H-3 and H-5. Me-19 gave a cross peak with H-8 (δ_H 2.28), but not H-5, indicating that Me-19 (δ_H 0.89) was *cis* to H-8 and *trans* to H-5. These results also established a *trans*-fused ring. Thus, 9-OH was located at an α-axial position. In addition, Me-30 (δ_H 1.08) did not give cross peaks with H-8 and Me-18 (δ_H 0.79), indicating Me-30 was *trans* to both H-8 and Me-18. Me-18 did not show a correlation with Me-21, suggesting that Me-18 and Me-21 were located at different side of the molecule. Thus, **SK9** had the relative stereochemistry as shown and was H-3α *epimer* of **SK12**. On the basis of these

spectral data, **SK9** was (22*Z*,24*E*)-3β,9α-dihydroxy-17,14-friedolanostan-14,22,-24-trien-26-oic acid, a new naturally occurring 17,14-friedolanostane.



(SK9)

Table 73 The NMR data of compound **SK9** in $CDCl_3$

Position	δ_H (*mult*, *J* Hz)	δ_C (C-Type)	HMBC	NOESY
1	α : 1.53 (*m*, 1H)	29.56 (CH_2)	C-3	-
	β : 1.40 (*m*, 1H)			Me-19
2	α : 1.69 (*m*, 1H)	27.12 (CH_2)	C-3	-
	β : 1.53 (*m*, 1H)			Me-19
3	3.23 (*dd*, 11.0, 4.0, 1H)	78.65 (CH)	C-5, C-28, C-29	Me-29
4	-	38.77 (C)	-	-
5	1.52 (*m*, 1H)	44.96 (CH)	C-3. C-6, C-19	Me-29
6	α : 1.98 (*m*, 1H)	20.71 (CH_2)	C-5, C-8	-
	β : 1.64 (*m*, 1H)			Me-19
7	1.34 (*m*, 1H)	24.94 (CH_2)	C-9	-
	2.03 (*m*, 1H)			
8	2.28 (*m*, 1H)	39.03 (CH)	C-14	Me-19
9	-	75.48 (C)	-	-
10	-	42.27 (C)	-	-
11	α : 1.50 (*m*, 1H)	29.71 (CH_2)	C-13	-
	β : 1.84 (*m*, 1H)			Me-19

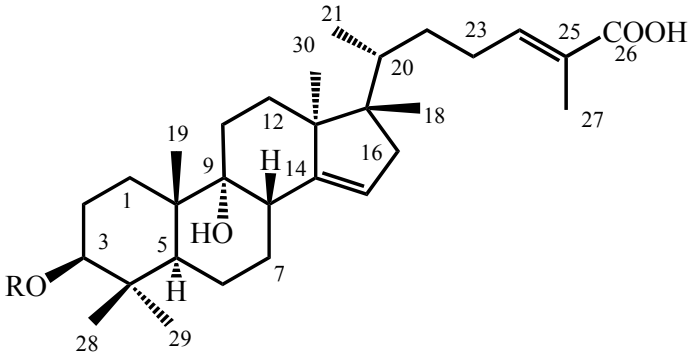
Table 73 (continued)

Position	δ_H (*mult*, *J* Hz)	δ_C (C-Type)	HMBC	NOESY
12	α : 1.51 (*m*, 1H)	27.47 (CH_2)	C-9, C-13, C-17,	Me-30
	β : 1.57 (*m*, 1H)		C-30	-
13	-	49.26 (C)	-	-
14	-	153.30 (C)	-	-
15	5.35 (*brs*, 1H)	120.24 (CH)	C-13, C-16	-
16	2.37 (*m*, 1H)	44.19 (CH_2)	C-20	-
	1.85 (*m*, 1H)			
17	-	53.76 (C)	-	-
18	0.79 (*s*, 3H)	15.23 (CH_3)	C-13, C-17	H-20
19	0.89 (*s*, 3H)	16.43 (CH_3)	C-5, C-9, C-10	H-1β,H-2β,H-6β, H-8, H-11β, Me-28
20	3.21 (*m*, 1H)	36.96 (CH)	C-17, C-22, C-23	-
21	0.93 (*d*, 7.0, 3H)	17.74 (CH_3)	C-13, C-17, C-20	-
22	5.98 (*t*, 11.5, 1H)	144.07 (CH)	C-17, C-20, C-24	H-23
23	6.22 (*t*, 11.5, 1H)	121.90 (CH)	C-20, C-24, C-25	H-22, Me-27
24	7.65 (*t*, 11.5, 1H)	134.97 (CH)	C-25, C-26, C-27	-
25	-	126.05 (C)	-	-
26	-	172.10 (C=O)	-	-
27	1.97 (*s*, 3H)	12.21 (CH_3)	C-24, C-25, C-26	-
28	0.79 (*s*, 3H)	15.63 (CH_3)	C-3, C-4, C-5, C-29	-
29	0.99 (*s*, 3H)	28.95 (CH_3)	C-3, C-4, C-5, C-28	H-3, H-5, H-6α,
30	1.08 (*s*, 3H)	18.81 (CH_3)	C-12, C-13, C-17	H-12α

1.3.1.6 Compound SK19

Compound **SK19** was obtained as a colorless gum and identified as its monoacetated derivative (**SK19-Ac**). **SK19-Ac** showed the molecular formula $C_{32}H_{50}O_5$ by EI-MS, which gave *m/z* 514. The IR spectrum displayed the presence of a hydroxyl group (3434 cm^{-1}), a carbonyl group of an α,β-unsaturated carboxylic acid

(1669 cm^{-1}) and a saturated ester (1714 cm^{-1}). The UV spectrum gave an absorption band at λ_{max} 264 nm, indicating that **SK19-Ac** possessed the α,β-unsaturated carboxylic acid chromophore. The ^1H NMR spectrum was similar to that of **SK9** except that no *trans*-olefinic protons. **SK19-Ac** displayed two sets of nonequivalent methylene protons (δ_H 2.18, 1.10, 1.71 and 2.04) instead of two *trans*-olefinic protons (δ_H 5.98 and 6.22) in **SK9**. These results indicated that **SK19-Ac** contained a -CH(Me)CH_2CH_2CH=C(Me)COOH side chain. It was confirmed by ^1H-^1H COSY and HMBC data as follow. In ^1H-^1H COSY data, the methine H-20 (δ_H 1.92) showed correlation with Me-21 (δ_H 0.85) and the methylene H-22 (δ_H 1.73 and 1.60) which was further coupled with H-23 (δ_H 2.04 and 1.70). In addition, the methylene H-23 showed correlation with olefinic H-24 (δ_H 6.90). The olefinic H-24 showed HMBC correlations with Me-27 (δ_C 12.50) and the carbonyl of carboxylic group (δ_C 171.20). The olefinic proton, H-24 (δ_H 6.90), did not display a cross peak, in the NOESY spectrum, with the vinyl methyl protons, Me-27 (δ_H 1.85), indicating that the configuration of C-24/C-25 double bond in the side chain was *E*. According to the NOESY data, the relative stereochemistry of **SK19-Ac** in the tetracyclic system was identical to that **SK9**. Thus, **SK-19Ac** was the 3-acetoxy derivative of (24*E*)-3β-hydroxy-9α-hydroxy-17,14-friedolanstan-14,24-dien-26-oic acid.



R = H **SK19**

R = $\underset{31}{C}O\underset{32}{C}H_3$ **SK19-Ac**

Table 74 The NMR data of compound **SK19-Ac** in $CDCl_3$

Position	δ_H (*mult*, *J* Hz)	δ_C (C-Type)	HMBC	NOESY
1	α : 1.73 (*m*, 1H)	23.60 (CH_2)	C-3, C-9	H-3
	β : 1.60 (*m*, 1H)			-
2	1.66 (*m*, 1H)	28.83 (CH_2)	-	-
	1.44 (*m*, 1H)			-
3	4.49 (*dd*, 9.5, 4.0, 1H)	80.63 (CH)	C-5	H-1α, H-5, Me-29
4	-	37.70 (C)	-	-
5	1.67 (*m*, 1H)	45.14 (CH)	C-3, C-7, C-28	Me-29
6	α : 1.67 (*m*, 1H)	20.78 (CH_2)	C-7, C-28	Me-29
	β : 1.39 (*m*, 1H)			-
7	α : 1.95 (*m*, 1H)	25.63 (CH_2)	C-6	Me-30
	β : 1.42 (*m*, 1H)			-
8	1.67 (*m*, 1H)	39.11 (CH)	C-11	Me-19
9	-	75.20 (C)	-	-
10	-	42.10 (C)	-	-
11	α : 1.50 (*m*, 1H)	29.92 (CH_2)	C-8, C-9, C-13	-
	β : 1.78 (*m*, 1H)			-
12	1.66 (*m*, 1H)	29.05 (CH_2)	C-8, C-9, C-11	-
	1.50 (*m*, 1H)			-
13	-	49.13 (C)	-	-
14	-	153.01 (C)	-	-
15	5.35 (*brs*, 1H)	120.74 (CH)	C-13, C-16	-
16	2.24 (*m*, 1H)	44.66 (CH_2)	C-14, C-17, C-20	-
	1.78 (*m*, 1H)			-
17	-	54.53 (C)	-	-
18	0.76 (*s*, 3H)	15.34 (CH_3)	C-16, C-17, C-20	H-20
19	0.92 (*s*, 3H)	16.52 (CH_3)	C-2, C-9	H-8, H-11β
20	1.92 (*m*, 1H)	37.59 (CH)	C-17	-
21	0.85 (*brs*, 3H)	15.15 (CH_3)	C-17, C-22	-

Table 74 (continued)

Position	δ_H (*mult*, *J* Hz)	δ_C (C-Type)	HMBC	NOESY
22	2.18 (*m*, 1H)	31.14 (CH_2)	C-17	-
	1.10 (*m*, 1H)			-
23	1.71 (*m*, 1H)	27.46 (CH_2)	-	-
	2.04 (*m*, 1H)			-
24	6.90 (*t*, 4.5, 1H)	145.15 (CH)	C-23, C-26, C-27	-
25	-	126.74 (C)	-	-
26	-	171.20 (C=O)	-	-
27	1.85 (*s*, 3H)	12.05 (CH_3)	C-23, C-25, C-26	-
28	0.87 (*s*, 3H)	16.35 (CH_3)	C-3, C-4, C-5	H-3
29	0.88 (*s*, 3H)	28.10 (CH_3)	C-5, C-28	H-3, H-5
30	1.15 (*s*, 3H)	19.82 (CH_3)	C-14, C-17	H-7α
31	-	170.88 (C=O)	-	-
32	2.05 (*s*, 3H)	21.26 (CH_3)	C-31	-

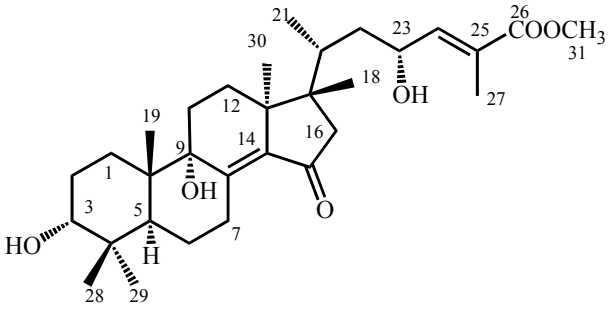
1.3.1.7 Compound SK21

Compound **SK21** was obtained as a colorless gum. It showed the molecular formula $C_{31}H_{48}O_6$. The IR spectrum revealed the presence of a hydroxyl group (3443 cm^{-1}), a carbonyl group of an α,β-unsaturated ester (1698 cm^{-1}) and a carbonyl group of a ketone (1742 cm^{-1}). In ^{13}C NMR spectrum, two carbonyl carbon signals at δ_C 168.32 and 207.75 supported the presence of the α,β-unsaturated ester and the α,β-unsaturated ketone. The UV spectrum with an absorption band at λ_{max} 258 nm indicated that **SK21** contained the α,β-unsaturated ester chromophore. The ^1H NMR spectrum (**Table 75**) (**Figure 15**) showed the presence of five methyl *singlets* (δ_H 1.21, 1.00, 0.92, 0.87 and 0.86, 3H each), two methyl *doublets* [δ_H 0.95 (*d*, *J* = 7.0 Hz, 3H) and δ_H 1.87 (*d*, *J* = 1.5 Hz, 3H)] and one oxymethine proton (δ_H 3.41, *t*, *J* = 2.5 Hz, 1H). These signals were regarded as being due to a tetracyclic triterpene (Rukachaisirikul, 2005), having a hydroxyl group at C-3α-axial position. The signals in the ^{13}C NMR spectrum (**Figure 16**) and DEPT experiment indicated the presence of two carbonyl carbons (δ_C 207.75 and 168.32), eight quaternary carbons (δ_C 151.65,

140.18, 127.52, 74.93, 45.99, 44.58, 44.41 and 37.80), five methine carbons (δ_C 143.91, 5.67, 66.61, 39.66 and 33.36), eight methylene carbons (δ_C 52.35, 39.24, 32.82, 29.91, 25.38, 24.79, 24.05 and 22.12) and seven methyl carbons (δ_C 28.75, 22.55, 21.11, 17.46, 16.80, 15.40 and 12.77). In addition, the ^{1}H NMR spectrum displayed signals at δ_H 2.26 (*m*, 1H), 0.95 (*d*, *J* = 7.0 Hz, 3H), 1.79 (*m*, 1H), 1.10 (*m*, 1H), 4.57 (*td*, *J* = 10.5 and 2.0 Hz, 1H), 6.70 (*dq*, *J* = 8.0 and 1.5 Hz, 1H) and 1.87 (*d*, *J* = 1.5 Hz, 3H). These NMR spectral data indicated that **SK21** contained a -CH(Me)CH_2CH(OH)CH=C(Me)$COOCH_3$ side chain which was the same as **SK2**. It was in agreement with the ^{1}H-^{1}H COSY and HMBC data. In HMBC spectrum, Me-21 [δ_H 0.95 (*d*, *J* = 7.0 Hz, 1H)] gave a cross peak with quaternary carbon, C-17 (δ_C 44.58), indicating the attachment of the side chain at C-17. Moreover, the ^{13}C NMR spectrum displayed the carbon signals of the α,β-unsaturated ketone at δ_C 207.75, 140.18 and 151.65 which was expected to be in the tetracyclic system. The methylene protons ($H_{\alpha,\beta}$-16) showed cross peaks, in the HMBC spectrum, with C-14 (δ_C 140.18), C-15 (δ_C 207.75) and C-17 (δ_C 44.58). The methylene protons (H-7β, δ_H 4.09) gave HMBC correlations with C-13 and C-14. These data suggested that the α,β-unsaturated ketone was located at C-15 and C-8/C-14. This assignment was in accordance with observation of one of the methylene protons at C-7 (δ_C 24.05) at downfield (δ_H 4.09) because of deshielding effect of a carbonyl group (Vieira, 2004). The position of all tertiary methyl groups was established using the data from the HMBC spectrum. A hydroxyl group was located at C-9 (δ_C 74.93) according to the chemical shift value of C-9.

The relative stereochemistry was deduced by NOEDIFF data (**Table 75**). Irradiation of the olefinic proton, H-24 (δ_H 6.69) did not affect signal intensity of the vinylic Me-27 (δ_H 1.87), suggesting that the configuration of double bond at C-24/C-25 of the side chain was *E*. Since the oxymethine H-3 appeared as *triplet* with a small coupling constant, it was assigned at β-equatorial position (Rukachaisirikul, 2005). Irradiation at Me-28 (δ_H 0.87) enhanced the signal intensity of the equatorial H-3 while the signal of H-5 was affected by irradiation of Me-29 (δ_H 1.10). These results implied that the H-3 was *cis* to Me-28 and *trans* to both H-5 and Me-29. Irradiation at Me-19 (δ_H 0.92) did not affect signal intensity of H-5, indicating that H-5 was *trans* to

Me-19. 9-OH was located at α-axial position in order to avoid the steric interaction between Me-19 and this hydroxyl group. The hydroxyl group (9-OH) of other compounds was also at α-axial, supporting above conclusion. Irradiation at Me-18 (δ_H 0.85) did not affect signal intensities of both Me-30 and Me-21, indicating that Me-18 was located at the opposite side of both Me-30 and Me-21. On the basis of these spectral data, **SK21** was methyl (24*E*)-3α,9α,23-trihydroxy-15-oxo-17,14-friedolanostan-8(14),24-dien-26-oate, a new 17,14-friedolanostane.



(SK21)

Table 75 The NMR data of compound **SK21** in $CDCl_3$

Position	δ_H (*mult*, *J* Hz)	δ_C (C-Type)	HMBC	NOE
1	1.32 (*m*, 1H)	24.79 (CH_2)	C-10, Me-19	-
	2.18 (*m*, 1H)			-
2	α : 1.70 (*m*, 1H)	25.38 (CH_2)	C-10	H-3
	β : 1.94 (*m*, 1H)			H-3
3	3.41 (*t*, 2.5, 1H)	75.67 (CH)	C-2, C-5	H-2α, H-2β, Me-28
4	-	37.80 (C)	-	-
5	2.20 (*m*, 1H)	39.66 (CH)	C-4, C-6, C-7, C-10, Me-29	Me-29
6	1.57 (*m*, 1H)	22.12 (CH_2)	C-8, C-10	-
	1.28 (*m*, 1H)			-
7	α : 2.20 (*m*, 1H)	24.05 (CH_2)	C-6, C-9, C-13, C-14	-
	β : 4.09 (*ddd*, 15.0, 4.0, 2.0, 1H)	-		-
8	-	151.65 (C)	-	-
9	-	74.93 (C)	-	-

Table 75 (continued)

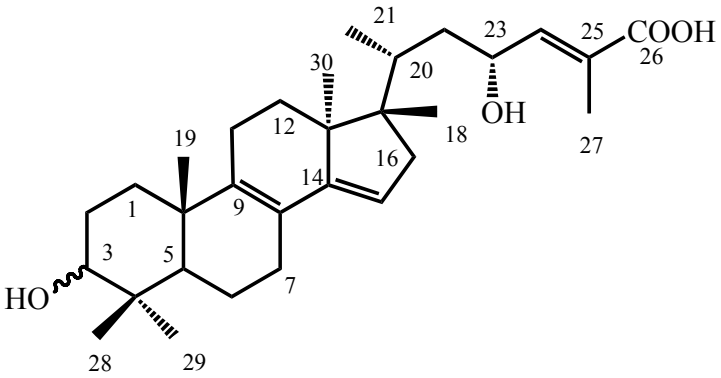
Position	δ_H (*mult, J* Hz)	δ_C (C-Type)	HMBC	NOE
10	-	44.41 (C)	-	-
11	2.31 (*m*, 1H)	29.91 (CH_2)	C-9, C-10, C-12,	-
	1.66 (*m*, 1H)		C-13	-
12	1.75 (*m*, 1H)	32.82 (CH_2)	C-9, C-11, C-13,	-
	1.94 (*m*, 1H)		C-14, C-17, C-30	-
13	-	45.99 (C)	-	-
14	-	140.18 (C)	-	-
15	-	207.75 (C=O)	-	-
16	α : 2.39 (*d*, 18.5, 1H)	52.35(CH_2)	C-13, C-14, C-15,	Me-21, Me-30
	β : 2.09 (*d*, 18.5, 1H)		C-17, C-18, C-20	-
17	-	44.58 (C)	-	-
18	0.86 (*s*, 3H)	16.80 (CH_3)	C-13, C-16, C-17,	H-20
			C-20	
19	0.92 (*s*, 3H)	17.46 (CH_3)	C-1, C-5, C-9,	H-3, H-2β
			C-10	
20	2.26 (*m*, 1H)	33.36 (CH)	C-13, C-16	
21	0.95 (*d*, 7.0, 3H)	15.40 (CH_3)	C-17, C-20,	H-16α
			C-22	
22	1.79 (*m*, 1H)	39.24 (CH_2)	C-17, C-21,	-
			C-23	
	1.10 (*m*, 1H)			-
23	4.57 (*td*, 10.5, 2.0, 1H)	66.61 (CH)	C-20, C-22,	Me-27
			C-24, C-25	
24	6.70 (*dq*, 8.0, 1.5, 1H)	143.91 (CH)	C-22, C-25,	-
			C-26, C-27	
25	-	127.52 (C)	-	-
26	-	168.32 (C=O)	-	-
27	1.87 (*d*, 1.5, 3H)	12.77 (CH_3)	C-24, C-25,	-
			C-26	
28	0.87 (*s*, 3H)	22.55 (CH_3)	C-3, C-4, C-5	H-3, H-6β

Table 75 (continued)

Position	δ_H (*mult, J* Hz)	δ_C (C-Type)	HMBC	NOE
29	1.00 (*s*, 3H)	28.75 (CH_3)	C-3, C-4, C-5	H-3, H-5
30	1.21 (*s*, 3H)	21.11 (CH_3)	C-12, C-13, C-17	H-16α
31	3.76 (*s*, 3H)	52.01 (CH_3)	C-26	

1.3.1.8 Compound SK11

Compound **SK11** was obtained as a colorless gum. The IR spectrum exhibited absorption bands at 3420 (a hydroxyl group) and 1697 (a carbonyl group of an α,β-unsaturated carboxylic acid) cm^{-1}. The UV spectrum with an absorption band at λ_{max} 217 nm indicated that **SK11** contained the α,β-unsaturated carboxylic acid moiety. The ^1H NMR data demonstrated signals of two oxymethine protons at δ_H 3.45 (*brs*, 1H) and 3.25 (*dd*, *J* = 9.0 and 4.5 Hz, 1H). These data implied that **SK11** was a mixture of two C-3 *epimer* triterpenes in a ratio of 1 to 1. In addition, the presence of two sets of carbons in the ^{13}C NMR spectrum supported this conclusion. Comparison of the ^1H and ^{13}C NMR spectra (**Table 76**) (**Figures 18** and **19**) were similar to those of **SK2** except that **SK11** contained no signals of a methoxy group. These results indicated that the -$CH(Me)CH_2CH(OH)CH=C(Me)COOCH_3$ unit of the side chain in **SK2** was replaced by a -$CH(Me)CH_2CH(OH)CH=C(Me)COOH$ moiety in **SK11**. The position of the side chain was located at C-17 by a HMBC correlation of Me-21 (δ_H 0.95) of the side chain with C-17 (δ_C 50.22, 50.07). Thus, **SK11** was assigned to have the same core structure as **SK2** with one tetrasubstituted double bond and one trisubstituted one at C-8/C-9 and C-14/C-15, respectively, according the HMBC correlations of olefinic protons, H-15 (δ_H 5.27, 5.28), with C-8 (δ_C 123.01, 122.58) and C-17 and that of Me-19 (δ_H 1.01) with C-9 (δ_C 148.79, 148.63). The position of all methyl groups was established using HMBC data (**Table 77**). Thus, **SK11** was a mixture of 3α and 3β-(24*E*)-9,23-dihydroxy-17,14-friedolanostan-8,14,24-trien-26-oicacid, two new *epimers* of 17,14-friedolanostane.



(SK11)

Table 76 The NMR data of **SK11** and ***Epimer*-SK11** in $CDCl_3$

Position	**SK11** δ_H (*mult*, *J* Hz)	**SK11** δ_C (C-Type)	***Epimer*-SK11** δ_H (*mult*, *J* Hz)	***Epimer*-SK11** δ_C (C-Type)
1	1.58 (*m*, 2H)	29.26 (CH_2)	1.74 (*m*, 1H)	27.06 (CH_2)
			1.64 (*m*, 1H)	
2	2.02 (*m*, 1H)	25.56 (CH_2)	1.35 (*m*, 2H)	26.70 (CH_2)
	1.67 (*m*, 1H)			
3	3.45 (*brs*, 1H)	75.93 (CH)	3.25 (*dd*, 9.0, 4.5, 1H)	78.93 (CH)
4	-	37.97 (C)	-	37.81 (C)
5	1.62 (*m*, 1H)	44.45 (CH)	1.12 (*m*, 1H)	50.58 (CH)
6	1.74 (*m*, 1H)	18.26 (CH_2)	1.34 (*m*, 1H)	26.70 (CH_2)
	1.47 (*m*, 1H)			
7	1.74 (*m*, 1H)	28.64 (CH_2)	2.02 (*m*, 1H)	25.56 (CH_2)
	1.65 (*m*, 1H)		1.67 (*m*, 1H)	
8	-	123.01 (C)	-	122.58 (C)
9	-	148.79 (C)	-	148.63 (C)
10	-	37.60 (C)	-	38.87 (C)
11	2.08 (*m*, 2H)	22.77 (CH_2)	2.08 (*m*, 2H)	22.74 (CH_2)
12	2.23 (*m*, 1H)	30.10 (CH_2)	2.28 (*m*, 2H)	31.74 (CH_2)
	1.35 (*m*, 1H)			
13	-	48.02 (C)	-	48.02 (C)
14	-	142.41 (C)	-	142.09 (C)

Table 76 (continued)

Position	**SK11** : δ_H (*mult*, *J* Hz)	**SK11** : δ_C (C-Type)	***Epimer*-SK11** : δ_H (*mult*, *J* Hz)	***Epimer*-SK11**: δ_C (C-Type)
15	5.27 (*brs*, 1H)	116.73 (CH)	5.28 (*brs*, 1H)	115.79 (C)
16	1.98 (*dt*, 16.0, 3.5, 1H)	45.57 (CH_2)	1.98 (*dt*, 16.0, 3.5, 1H)	45.57 (CH_2)
	2.34 (*m*, 1H)		2.34 (*m*, 1H)	
17	-	50.22 (C)	-	50.07 (C)
18	0.76 (*s*, 3H)	15.65 (CH_3)	0.82 (*s*, 3H)	15.65 (CH_3)
19	1.01 (*s*, 3H)	19.15 (CH_3)	1.01 (*s*, 3H)	18.97 (CH_3)
20	2.23 (*m*, 1H)	33.40 (CH)	2.23 (*m*, 1H)	33.40 (CH)
21	0.95 (*d*, 6.0, 3H)	15.28 (CH_3)	0.95 (*d*, 6.0, 3H)	15.28 (CH_3)
22	1.77 (*m*, 1H)	39.29 (CH_2)	1.77 (*m*, 1H)	39.29 (CH_2)
	1.15 (*m*, 1H)		1.15 (*m*, 1H)	
23	4.59 (*t*, 8.5, 1H)	67.01 (CH)	4.59 (*t*, 8.5, 1H)	67.01 (CH)
24	6.84 (*d*, 8.0, 1H)	146.63 (CH)	6.82 (*d*, 8.0, 1H)	148.79 (CH)
25	-	126.38 (C)	-	123.01 (C)
26	-	171.78 (C=O)	-	171.77 (C=O)
27	1.87 (*s*, 3H)	12.46 (CH_3)	1.87 (*s*, 3H)	12.46 (CH_3)
28	1.03 (*s*, 3H)	22.19 (CH_3)	0.83 (*s*, 3H)	15.65 (CH_3)
29	0.99 (*s*, 3H)	28.01 (CH_3)	1.02 (*s*, 3H)	28.01 (CH_3)
30	0.89 (*s*, 3H)	17.10 (CH_3)	0.89 (*s*, 3H)	17.10 (CH_3)

Table 77 The HMBC correlations of compound **SK11** and ***Epimer*-SK11**

Proton	HMBC correlations, C_n (δ_C)
H-3	C-4 (37.97, 37.81), C-29 (28.01)
H-5	C-10 (37.60, 38.87), C-28 (22.19, 15.65), C-29 (28.01)
H-15	C-8 (123.01, 122.58), C-13 (48.02), C-17 (50.22, 50.07)
H-16a,b	C-8, C-15 (116.73, 115.79), C-20
Me-19	C-9, C-10 (148.79, 148.63)
H-21	C-17, C-20 (33.40), C-23 (67.01)
H-23	C-22 (39.29), C-27 (12.46)

Table 77 (continued)

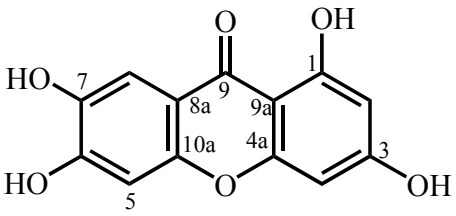
Proton	HMBC correlations, C_n (δ_C)
H-24	C-25 (126.38, 123.01), C-26 (171.78, 171.77)
H-27	C-24 (146.63, 148.79), C-25
Me-28	C-3 (75.93, 78.93)
Me-29	C-3
Me-30	C-15

1.3.2 Xanthones

1.3.2.1 Compound SK4

Compound **SK4** was isolated as a yellow solid, melting at 212-215 °C. Its exhibited UV absorption bands of a xanthone chromophore at λ_{max} 235, 253, 312 and 362 nm while hydroxyl and conjugated carbonyl absorption bands were found at 3419 and 1655 cm^{-1}, respectively, in the IR spectrum. The ^1H NMR spectrum (**Table 78**) (**Figure 20**) contained signals of one chelated hydroxy proton (δ_H 13.23, *s*, 1H), two *singlet* aromatic protons (δ_H 7.53 and 6.92) and two *meta*-coupled aromatic protons [δ_H 6.37 (*s*) and 6.22 (*s*)]. The ^{13}C NMR (**Table 78**) (**Figure 21**) and HMQC data indicated that compound **SK4** consisted of 13 carbons: 9 quarternary and 4 methine carbons. The chelated hydroxy proton, which was located at the *peri*-position to the xanthone carbonyl group, showed HMBC correlations with C-1 (δ_C 163.58), C-2 (δ_C 97.69), C-9 (δ_C 179.59) and C-9a (δ_C 102.27). Two *meta*-coupled aromatic protons (δ_H 6.22 and 6.37) were assigned as H-2 and H-4, respectively, according to a HMQC correlation of H-2/C-2 and HMBC cross peaks of H-2/C-1, C-3 (δ_C 157.99), C-4 (δ_C 93.50) and C-9a as well as those of H-4/C-2, C-3, C-4a (δ_C 164.68) and C-9a. The aromatic proton at δ_H 7.53 was attributed to H-8 on the basis of the chemical shift value and HMBC correlations of H-8/C-6 (δ_C 153.92) and C-10a (δ_C 151.81). The aromatic proton at δ_H 6.92 was then attributed to H-5 and gave ^3J HMBC correlations with C-7 and C-8a (δ_C 112.66). The chemical shift values of C-3, C-6 and C-7 suggested the substituents to be hydroxyl groups. Thus, **SK4** was determined as

norathyriol which was isolated from the twigs of *G. parvifolia* (Rukachaisirikul, 2006).



(SK4)

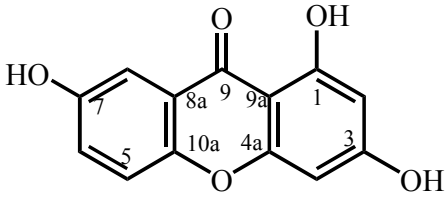
Table 78 The NMR data of compound **SK4** and norathyriol

Position	**SK4** (Acetone-d_6)		HMBC	norathyriol (DMSO-d_6)*	
	δ_H (*mult*, *J* Hz**)**	δ_C (C-Type)		δ_H (*mult*, *J* Hz**)**	δ_C
OH-1	13.23 (*s*, 1H)	163.58 (C)	C-1, C-2, C-9, C-9a	13.26 (*brs*, 1H)	162.5
2	6.22 (*s*, 1H)	97.69 (CH)	C-1, C-3, C-4, C-9a	6.18 (*d*, 1.7, 1H)	97.7
3	-	157.99 (C)	-	-	157.3
4	6.37 (*s*, 1H)	93.50 (CH)	C-2, C-3, C-4a, C-9a	6.34 (*d*, 1.7, 1H)	93.5
4a	-	164.68 (C)	-	-	164.6
5	6.92 (*s*, 1H)	102.51 (CH)	C-6, C-7, C-8a, C-10a	6.85 (*s*, 1H)	102.8
6	-	153.92 (C)	-	-	155.0
7	-	143.48 (C)	-	-	144.0
8	7.53 (*s*, 1H)	108.16 (CH)	C-6, C-7, C-8a, C-10a	7.39 (*s*,1H)	107.6
8a	-	112.66 (C)	-	-	111.3
9	-	179.59 (C=O)	-	-	178.7
9a	-	102.27 (C)	-	-	101.5
10a	-	151.81 (C)	-	-	151.2

* (Noro, 1984)

1.3.2.2 Compound SK5

Compound **SK5** was isolated as a yellow gum. The UV and IR absorption bands were similar to those of **SK4**. The ^{1}H NMR spectrum (**Table 79**) (**Figure 22**) contained signals of one chelated hydroxyl group (δ_H 12.98, *s*, 1H), two *meta*-coupled aromatic protons [δ_H 6.41 and 6.25 (*d*, *J* = 2.5 Hz, 1H, each)], three aromatic protons of a 1,2,4-trisubstituted benzene [δ_H 7.56 (*d*, *J* = 3.0 Hz, 1H), 7.44 (*d*, *J* = 9.0 Hz, 1H) and 7.34, (*dd*, *J* = 9.0 and 3.0 Hz, 1H)]. The ^{1}H NMR data and HMBC correlations on the right-hand ring were similar to those of **SK4**. The aromatic protons of the 1,2,4-trisubstituted benzene at δ_H 7.56, 7.34 and 7.44 were attributed to H-8, H-6 and H-5, respectively, on the basis of the chemical shift value of H-8 and HMBC correlations of H-8/C-6 (δ_C 125.15), C-7 (δ_C 154.99), C-9 (δ_C 181.25) and C-10a (δ_C 150.73), H-6/C-7, C-8 (δ_C 109.38) and C-10a and those of H-5/C-7, C-8a and C-9. Thus, **SK5** was identified as 1,3,7-trihydroxyxanthone which was isolated from the bark of *G. xanthochymus* (Zhong, 2008).



(SK5)

Table 79 The NMR data of compound **SK5** and 1,3,7-trihydroxyxanthone

Position	**SK5** (Acetone-d_6)		HMBC	1,3,7-trihydroxyxanthone (DMSO-d_6)*	
	δ_H (*mult, J* Hz)	δ_C (C-Type)		δ_H (*mult, J* Hz)	δ_C
OH-1	12.98 (*s* ,1H)	164.63 (C)	C-1, C-2, C-9a	12.88 (*s*, 1H)	162.7
2	6.25 (*d*, 2.5, 1H)	98.78 (CH)	C-1, C-3, C-4, C-9a	6.18 (*d*, 1.9, 1H)	98.0
OH-3	10.34 (*brs*, 1H)	166.57 (C)	-	11.04 (*s*, 1H)	163.0
4	6.41 (*d*, 2.5, 1H)	94.60 (CH)	C-2, C-3, C-4a, C-9, C-9a	6.35 (*d*, 2.1, 1H)	93.9
4a	-	159.03 (C)	-	-	154.1

Table 79 (continued)

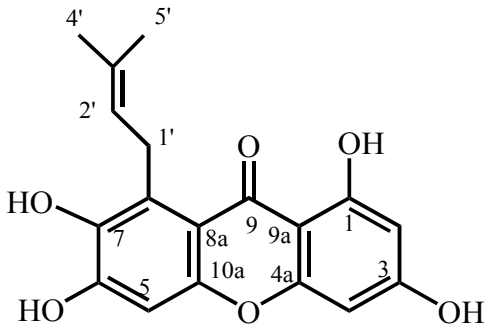
Position	**SK5** (Acetone-d_6)		HMBC	1,3,7-trihydroxyxanthone (DMSO-d_6)*	
	δ_H (*mult, J* Hz)	δ_C (C-Type)		δ_H (*mult, J* Hz)	δ_C
5	7.44 (*d,* 9.0, 1H)	119.71 (CH)	C-7, C-8a, C-9, C-10a	7.45 (*d,* 9.0, 1H)	119.1
6	7.34 (*dd,* 9.0, 3.0, 1H)	125.15 (CH)	C-7, C-8, C10a	7.27 (*dd,* 9.0, 3.0, 1H)	124.6
OH-7	9.34 (*s*, 1H)	154.99 (C)	-	10.00 (*s*, 1H)	149.2
8	7.56 (*d*, 3.0, 1H)	109.38 (CH)	C-6, C-7, C-9, C-10a	7.40 (*d*, 3.0, 1H)	108.2
8a	-	121.88 (C)	-	-	120.6
9	-	181.25 (C=O)	-	-	179.9
9a	-	103.47 (C)	-		102.1
10a	-	150.73 (C)	-	-	157.7

*(Mukulesh, 2006**)**.

1.3.2.3 Compound SK8

Compound **SK8** was isolated as a yellow gum. The UV and IR absorption bands were similar to those of **SK4**. The ^{1}H NMR spectrum (**Table 80**) (**Figure 24**) contained signals of one chelated hydroxyl group (δ_H 13.70, *s*), two *meta*-coupled aromatic protons [δ_H 6.29 and 6.18 (*brs*, 1H each)], one *singlet* aromatic proton (δ_H 6.83, 1H) and one prenyl unit [δ_H 5.32 (*mt*, J = 7.0 Hz, 1H), 4.18 (*d*, J = 7.0 Hz, 2H), 1.84 (*s*, 3H) and 1.64 (*s*, 3H)]. The ^{1}H NMR data and HMBC correlations on the right-hand ring were similar to those of **SK4**, but they were different in the replacement of one aromatic proton with the prenyl group in the left-hand ring. The HMBC correlations between the methylene protons [H_2-1′, (δ_H 4.18)] of the prenyl group and C-7 (δ_C 141.98) and C-8a (δ_C 111.78) and the olefinic proton [H-2′, (δ_H 5.32)] and C-8 (δ_C 128.90) established the attachment of the prenyl unit at C-8. The *singlet* aromatic proton (δ_H 6.83) was located at C-5 (δ_C 101.05) and gave HMBC cross peaks with C-7 and C-8a (δ_C 111.78). Thus, **SK8** was determined as 1,3,6,7-

tetrahydroxy-8-prenylxanthone which was isolated from *Hypericum patunlum* (Ishiguro, 1995).



(SK8)

Table 80 The NMR data of compound **SK8** and 1,3,6,7-tetrahydroxy-8-prenyl xanthone in Acetone-d_6

Position	**SK8**		HMBC	1,3,6,7-tetrahydroxy-8-prenylxanthone	
	δ_H (*mult, J* Hz)	δ_C (C-Type)		δ_H (*mult, J* Hz)	δ_C
OH-1	13.70 (*s*, 1H)	165.15 (C)	C-1, C-2, C-9a	-	164.6
2	6.18 (*brs*, 1H)	98.48 (C)	C-1, C-3, C-4, C-9a	6.17 (*d*, 1.8, 1H)	98.5
3	-	164.89 (C)	-	-	165.0
4	6.29 (*brs*, 1H)	93.58 (CH)	C-2, C-3, C-4a, C-9a,	6.28 (*d*, 1.8, 1H)	93.7
4a	-	158.01 (C)	-	-	158.1
5	6.83 (*s*, 1H)	101.25 (CH)	C-7, C-8a	6.79 (*s*, 1H)	101.2
6	-	153.02 (C)	-	-	154.0
7	-	141.98 (C)	-	-	142.3
8	-	128.90 (C)	-	-	128.3
8a	-	111.78 (C)	-	-	111.4
9	-	183.11 (C=O)	-	-	183.1
9a	-	103.87 (C)	-	-	103.9
10a	-	153.71 (C)	-	-	154.0
1′	4.18 (*d*, 7.0, 2H)	26.36 (CH_2)	C-7, C-8a, C-2′, C-3′, C-4′	4.18 (*d*, 6.7, 2H)	26.4

Table 80 (continued)

Position	SK8		HMBC	1,3,6,7-tetrahydroxy-8-prenylxanthone	
	δ_H (*mult, J* Hz)	δ_C (C-Type)		δ_H (*mult, J* Hz)	δ_C
2′	5.32 (*t*, 7.0, 1H)	124.49 (CH)	C-8, C-4′, C-5′	5.33 (*t*, 6.7, 1H)	124.7
3′	-	131.28 (C)	-	-	131.2
4′	1.64 (*s*, 3H)	26.00 (CH_3)	C-2′, C-3′, C-5′	1.64 (*s*, 3H)	26.0
5′	1.83 (*s*, 3H)	18.28 (CH_3)	C-2′, C-3′, C-4′	1.84 (*s*, 3H)	18.3

1.3.2.4 Compound SK16

Compound **SK16** was obtained as a yellow gum. The UV and IR absorption bands were similar to those of **SK8**, indicating that **SK16** was a xanthone derivative. Its NMR data (**Table 81**) (**Figure 26**) were similar to those of **SK8** which contained one chelated hydroxy proton (δ_H 13.38, *s*, 1H), two *meta*-coupled protons [δ_H 6.34 and 6.20 (*d*, *J* = 2.1 Hz, 1H each)] and one *singlet* aromatic proton (δ_H 6.82, *s*, 1H). The differences in ^{1}H NMR spectrum were signals of a chromene ring [δ_H 8.03 and 5.94 (*d*, *J* = 10.2 Hz, 1H each) and 1.45 (*s*, 3H)] which were replaced by signals of the prenyl group in **SK8**. The appearance of one of *cis*-olefinic protons of a chromene ring at low field (δ_H 8.03, H-1′) indicated that the chromene ring was fused at C-7 (δ_C 138.93) and C-8 (δ_C 120.95). This proton showed cross peak, in the HMBC spectrum, with an oxyaromatic carbon (C-7) while the other olefinic proton (δ_H 5.94, H-2′) gave a cross peak with a quaternary carbon (C-8). These data confirmed that the chromene ring was fused to C-7 and C-8 with an ether linkage at C-7. Thus, **SK16** was assigned as toxyloxanthone B which was isolated from *G. dulcis* (Iinuma, 1996).

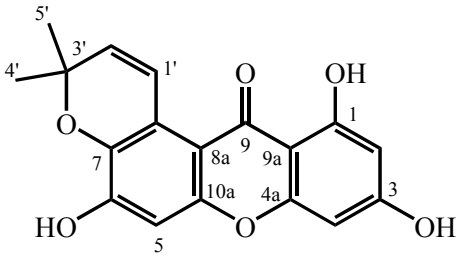


Table 81 The NMR data of compound **SK16** and toxyloxanthone B in Acetone-d_6

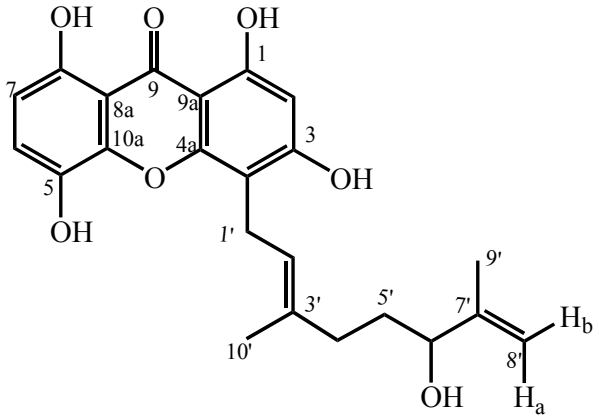
Position	**SK16**		HMBC	toxyloxanthone B*	
	δ_H (*mult, J* Hz)	δ_C (C-type)		δ_H (*mult,J* Hz)	δ_C
OH-1	13.38 (*s*, 1H)	165.67 (C)	C-1, C-2, C-9a	13.36 (*s*, 1H,)	165.6
2	6.20 (*d*, 2.1, 1H)	98.78 (CH)	C-3, C-4, C-9a	6.20 (*d*, 2.4, 1H)	98.8
3	-	164.76 (C)		-	164.8
4	6.34 (*d*, 2.1, 1H)	93.95 (CH)	C-2, C-3, C-9	6.33 (*d*, 1.8, 1H)	94.0
4a	-	154.08 (C)		-	154.1
5	6.82 (*s*, 1H)	103.90 (CH)	C-7, C-9, C-8a, C-10a	6.81 (*s*, 1H)	103.5
6	-	158.22 (C)	-	-	158.3
7	-	138.93 (C)	-	-	138.9
8	-	120.95 (C)	-	-	121.0
8a	-	108.50 (C)	-	-	108.5
9	-	183.12 (C)	-	-	183.1
9a	-	103.50 (C)	-	-	104.0
10a	-	153.80 (C)	-	-	153.8
1′	8.03 (*d*, 10.2, 1H)	121.49 (CH)	C-7, C-3′	8.05 (*d*, 10.4, 1H)	121.5
2′	5.94 (*d*, 10.2, 1H)	133.64 (CH)	C-8, C-1′, C-3′	5.94 (*d*, 10.4, 1H)	133.6
3′	-	76.77 (C)	-	-	76.8
Me-4′, 5′	1.45 (*s*, 6H)	27.16 (CH_3)	C-7, C-3′, Me-4′, 5′	1.46 (*s*, 6H)	27.2

*Ishiguro, 1993

1.3.2.5 Compound SK18

Compound **SK18** was obtained as a pale yellow gum. It showed a molecular ion at *m/z* 412, which was corresponded to the molecular formula $C_{22}H_{22}O_7$. The IR spectrum exhibited absorption bands at 3541 cm^{-1} (a hydroxyl group) and 1698 cm^{-1} (a carbonyl group). The UV spectrum with absorption bands at 222, 229, 250, 259 and 277 nm indicated that **SK18** was a xanthone derivative. Its ^1H NMR spectrum (**Table 82**) (**Figure 28**) showed the signals of two chelated hydroxy protons [δ_H 11.98 and 11.30 (*s*, 1H each)], two *ortho*-coupled aromatic protons [δ_H 7.32 and 6.62 (*d*, *J* = 8.7 Hz, 1H each)] and one *singlet* aromatic proton (δ_H 6.40, *s*, 1H). In addition, it contained signal of a 3,7-dimethyl-6-hydroxyocta-2,7-diene moiety [δ_H 5.39 (*mt*, *J* = 6.5 Hz, 1H), 4.83 (*brs*, 1H), 4.69 (*brs*, 1H), 3.94 (*t*, *J* = 6.6 Hz, 1H), 3.58 (*d*, *J* = 7.0 Hz, 2H), 2.20 (*m*, 2H), 1.86 (*s*, 3H), 1.64 (*s*, 3H) and 1.60 (*m*, 2H)]. The ^{13}C NMR spectrum (**Table 82**) (**Figure 29**) showed twenty three carbons: eleven quaternary carbons (δ_C 185.86, 165.25, 161.86, 155.78, 154.23, 149.35, 145.20, 138.24, 135.94, 108.03 and 102.65), five methine carbons (δ_C 124.81, 123.02, 110.07, 99.05 and 75.36), four methylene carbons (δ_C 110.31, 36.59, 34.61 and 22.05) and two methyl carbons (δ_C 17.81 and 16.48). Two chelated hydroxy protons (δ_H 11.98 and 11.30) were attributed to OH-1 and OH-8, respectively, according to the HMBC correlations of OH-1/C-1 (δ_C 161.86), C-2 (δ_C 99.05) and C-9a (102.65) and those of OH-8/C-7 (δ_C 110.07), C-8 (154.23) and C-8a (108.03). In the HMQC spectrum, the *singlet* aromatic proton (δ_H 6.40) showed correlation with C-2 (δ_C 99.05), suggesting its attachment at C-2. In addition, the *ortho*-aromatic protons were located at C-6 and C-7 by a HMQC correlation of H-7 (δ_H 6.62)/C-7 and HMBC correlations of H-6 (δ_H 7.32)/C-5 (δ_C 138.24), C-8 and C-10a (δ_C 145.20). The methylene protons (H-1′) of the 3,7-dimethyl-6-hydroxyocta-1,7-diene group showed HMBC correlations with C-3 and C-4a (δ_C 155.78), indicating the attachment of this group at C-4. According to the chemical shift values of C-3 and C-5, they contained hydroxyl groups as substituents. The *E*-configulation of side chain was deduced from the NOEDIFF spectrum since irradiation of Me-10′ (δ_H 1.86) did not enhance signal intensity of an olefinic proton, H-2′ (δ_H 5.39). Upon irradiation of Me-9′ (δ_H 1.63), enhancement of

the signal of H-$8_b'$ was observed. Therefore, this methyl group was *cis* to H-$8_b'$. Thus, **SK18** was assigned to have the structure as shown, a new naturally occurring xanthone.



(SK18)

Table 82 The NMR data of compound **SK18** in Acetone-d_6

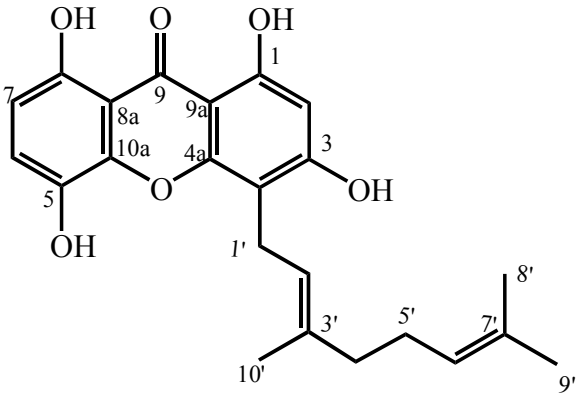
Position	δ_H (*mult, J* Hz)	δ_C (C-type)	HMBC	NOE
OH-1	11.98 (*s*, 1H)	161.86 (C)	C-1,C-2, C-9a	-
2	6.40 (*s*, 1H)	99.05 (CH)	C-3, C-4, C-9a	-
3	-	165.25 (C)	-	-
4	-	108.03 (C)	-	-
4a	-	155.78 (C)	-	-
5	-	138.24 (C)	-	-
6	7.32 (*d*, 8.7, 1H)	124.81 (CH)	C-5, C-8, C-10a	H-7
7	6.62 (*d*, 8.7, 1H)	110.07 (CH)	C-5, C-8, C-8a	H-6
OH-8	11.30 (*s*, 1H)	154.23 (C)	C-7, C-8, C-8a	-
8a	-	108.03 (C)	-	-
9	-	185.86 (C=O)	-	-
9a	-	102.65 (C)	-	-
10a	-	145.20 (C)	-	-
1′	3.58 (*d*, 7.0, 2H)	22.05 (CH_2)	C-3, C-4, C-4a, C-2′, C-3′	H-10′
2′	5.39 (*mt*, 7.0, 1H)	123.02 (CH)	C-4′, C-10′	-
3′	-	135.94 (C)	-	-
4′	2.20 (*m*, 2H)	36.59 (CH_2)	C-2′, C-5′	-

Table 82 (continued)

Position	δ_H (*mult, J* Hz)	δ_C (C-type)	HMBC	NOE
5′	1.60 (*m*, 2H)	34.56 (CH_2)	C-4′	-
6′	3.94 (*t*, 6.6, 1H)	75.32 (CH)	C-4′, C-5′, Me-9′	-
7′	-	149.35 (C)	-	-
8′	a : 4.83 (*brs*, 1H)	110.31 (CH_2)	C-6′, Me-9′	H-6′
	b : 4.69 (*brs*, 1H)			H-9′
Me-9′	1.64 (*s*, 3H)	17.81(CH_3)	C-6′, C-7′, C-8′	H-8_b′
Me-10′	1.86 (*s*, 3H)	16.48 (CH_3)	C-2′, C-3′, C-4′	C-1′

1.3.2.6 Compound SK13

Compound **SK13** was obtained as a yellow gum. The UV and IR absorption bands were similar to those of **SK18**, indicating that **SK13** was a xanthone derivative. Its ^{1}H and ^{13}C NMR (**Figure 31** and **32**) data were similar to those **SK18** except that **SK13** contained none of signals for a 3,7-dimethyl-6-hydroxyocta-2,7-diene substitutent. These signals were replaced by signals for a geranyl group [δ_H 3.56 (*d*, *J* = 7.0 Hz, 2H, H-1′), 5.27 (*t*, *J* = 7.0 Hz, 1H, H-2′), 2.11 (*m*, 2H, H-4′), 2.09 (*m*, 2H, H-5′), 5.04 (*m*, 1H, H-6′), 1.61 (*s*, 3H, H-8′), 1.58 (*s*, 3H, H-9′) and 1.86 (*s*, 3H, H-10′)]. This substituent was assigned to be at C-4 (δ_C 105.50) by HMBC correlations (**Table 83**) of its methylene protons (H_{ab}-1′) with C-3 (δ_C 162.84) and C-4a (δ_C 154.24). The attachment of other substituents was identical to **SK18** by HMBC data. The configuration of the C-2′/C-3′ and C-6′/C-7′ double bonds in geranyl group was deduced from the NOEDIFF data. Irradiation of H-6′ (δ_H 5.04) affected signal intensity of Me-9′, while H-2′ (δ_H 5.04) did not enhance signal intensity of Me-10′. Therefore, the configuration of C-2′/C-3′ double bond was *E*. Thus, **SK13** was assigned as cheffouxanthone which was isolated from root barks of *G. smeathmannii* (Lannang, 2006).



(SK13)

Table 83 The NMR data of compound **SK13** and cheffouxanthone

Position	**SK13** ($CDCl_3$)		HMBC	NOE	cheffouxanthone (Acetone-d_6)	
	δ_H (*mult, J* Hz)	δ_C (C-type)			δ_H (*mult, J* Hz)	δ_C
OH-1	12.03 (*s*, 1H)	161.42 (C)	C-1,C-2, C-9a	-	12.01 (*s*, 1H)	161.4
2	6.32 (*s*, 1H)	99.36 (CH)	C-3, C-4, C-9a	-	6.38 (*s*, 1H)	98.4
3	-	162.84 (C)	-	-	-	164.4
4	-	105.50 (C)	-	-	-	115.7
4a	-	154.24 (C)	-	-	-	144.6
5	-	135.74 (C)	-	-	-	137.7
6	7.25 (*d*, 8.5, 1H)	123.60 (CH)	C-5, C-8, C-10a	H-7	7.32 (*d*, 8.8, 1H)	124.2
7	6.69 (*d*, 8.5, 1H)	110.15 (CH)	C-5, C-8, C-8a	H-6	6.63 (*d*, 8.8, 1H)	109.5
OH-8	11.23 (*s*, 1H)	154.02 (C)	C-7, C-8, C-8a	-	11.30 (*s*, 1H)	153.7
8a	-	107.21 (C)	-	-	-	107.7
9	-	184.79 (C=O)	-	-	-	185.4
9a	-	102.79 (C)	-	-	-	102.2
10a	-	142.91 (C)	-	-	-	155.2

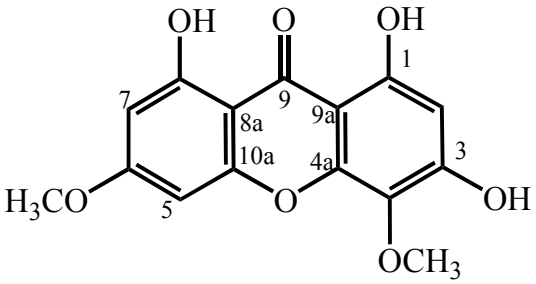
Table 83 (continued)

Position	**SK13** ($CDCl_3$)		HMBC	NOE	cheffouxanthone (Acetone-d_6)	
	δ_H (*mult, J* Hz)	δ_C (C-type)			δ_H (*mult, J* Hz)	δ_C
1′	3.56 (*d*, 7.0, 2H)	21.93 (CH_2)	C-3, C-4, C-4a, C-2′, C-3′	H-2′	3.59 (*d*, 7.2, 2H)	21.5
2′	5.27 (*t*, 7.0, 1H)	121.01 (CH)	C-4, C-4′, C-10′	H-1′, H-4′	5.38 (*d*, 7.2, 1H)	122.6
3′	-	139.23 (C)	-	-	-	135.2
4′	2.11 (*m*, 2H)	39.61 (CH_2)	C-2′, C-6′, C-10′	H-2′	1.98 (*t*, 7.2, 2H)	40.0
5′	2.09 (*m*, 2H)	26.36 (CH_2)	C-3′, C-7′	H-6′	2.02 (*m*, 2H)	26.8
6′	5.04 (*m*, 1H)	123.31 (CH)	C-5′, C-9′	H-5′, Me-9′	5.15 (*m*, 1H)	124.5
7′	-	132.22 (C)				131.1
Me-8′	1.61 (*s*, 3H)	25.65 (CH_3)	C-6′, C-7′, C-9′		1.54 (*s*, 3H)	25.2
Me-9′	1.58 (*s*, 3H)	17.72 (CH_3)	C-6′, C-7′, C-8′	H-6′	1.56 (*s*, 3H)	17.2
Me-10′	1.86 (*s*, 3H)	16.38 (CH_3)	C-2′, C-3′, C-4′		1.86 (*s*, 3H)	15.9

1.3.2.7 Compound SK20

Compound **SK20** was obtained as a pale yellow gum. It showed molecular ion at *m/z* 304, which corresponded to a molecular formula $C_{15}H_{12}O_7$. The IR spectrum exhibited absorption bands at 3443 and 1641 cm^{-1} (a hydroxyl group and a carbonyl group). The UV spectrum with absorption bands at 222, 258, 278 and 345 nm indicated that **SK20** had a xanthone chromophore. The ^1H NMR spectrum (**Table 84**) (**Figure 33**) showed the presence of two chelated hydroxy protons [δ_H 12.01 and 11.71 (1H each)], two *meta*-aromatic protons [δ_H 6.63 and 6.36 (*d*, *J* = 2.0 Hz, 1H

each)], one *singlet* aromatic proton (δ_H 6.32, *s*, 1H) and two sets of methoxy protons [δ_H 3.97 and 3.91 (*s*, 3H each)]. The ^{13}C NMR spectrum (**Table 84**) (**Figure 34**) showed fifteen carbons: ten quaternary carbons (δ_C 184.34, 168.20, 163.72, 159.72, 159.00, 158.53, 150.44, 128.84, 102.55 and 102.06), three methine carbons (δ_C 99.32, 98.36 and 93.94) and two methoxy carbons (δ_C 61.82 and 56.66). The location of all substitutents was established by HMBC data (**Table 84**). Two chelated hydroxy protons at C-1 (δ_C 159.72) and C-8 (δ_C 163.72), *peri*-position of the xanthone carbonyl group, gave ^3J cross peaks of OH-1/C-2 (δ_C 99.32) and C-9a (δ_C 102.55) and OH-8/C-7 (δ_C 98.36) and C-8a (δ_C 102.55). A HMQC correlation of the *singlet* aromatic proton (δ_H 6.32) with C-2 and HMBC correlations between the *singlet* aromatic proton (δ_H 6.32) and C-4 (δ_C 128.84) and C-9a established the attachment of the *singlet* aromatic proton at C-2, *ortho* to the chelated hydroxyl group. Two *meta*-aromatic protons (δ_H 6.63 and 6.36) were attributed to H-5 and H-7, respectively, according to a HMQC correlation of H-7 (δ_H 6.36)/C-7 and the HMBC correlations of H-7/C-6 (δ_C 168.20) and C-5 (δ_C 93.94) and those of H-5/C-7 and C-8a. Two methoxyl groups (δ_H 3.97 and 3.91) were assigned at C-6 and C-4, respectively, by ^3J correlation of OMe-6/C-6 and that of OMe-4/C-4. In the NOEDIFF experiments (**Table 84**), irradiation at OMe-6 enhanced signal intensities of both H-5 and H-7, supporting above assignment. According to the chemical shift value of C-3, the substituent at C-3 was a hydroxyl substituent. Thus, **SK20** had the structure as shown, a new naturally occurring xanthone.



(SK20)

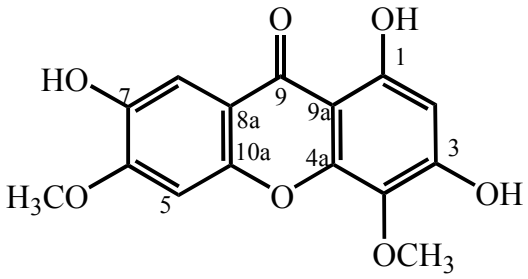
Table 84 The NMR data of compound **SK20** in Acetone-d_6

Position	δ_H (*mult, J* Hz)	δ_C (C-type)	HMBC	NOE
OH-1	11.71 (*s*, 1H)	159.72 (C)	C-1, C-2, C-9a	-
2	6.32 (*s*, 1H)	99.32 (CH)	C-1, C-4, C-9a	-
3	-	159.00 (C)	-	-
4	-	128.84 (C)	-	-
4a	-	150.44 (C)	-	-
5	6.63 (*d*, 2.0, 1H)	93.94 (CH)	C-6, C-7, C-8a , C-10a	OMe-6
6	-	168.20 (C)	-	-
7	6.36 (*d*, 2.0, 1H)	98.36 (CH)	C-5, C-6	OMe-6
OH-8	12.01 (*s*, 1H)	163.72 (C)	C-7, C-8, C-8a	-
8a	-	102.55 (C)	-	-
9	-	184.38 (C=O)	-	-
9a	-	102.06 (C)	-	-
10a	-	158.53 (C)	-	-
OMe-4	3.91 (*s*, 3H)	61.82 (CH_3)	C-4	-
OMe-6	3.97 (*s*, 3H)	56.66 (CH_3)	C-6	H-5, H-7

1.3.2.8 Compound SK22

Compound **SK22** was obtained as a pale yellow gum. It showed molecular ion at *m/z* 304, which corresponded to a molecular formula $C_{15}H_{12}O_7$. The IR and UV spectra were similar to those of **SK20**, indicating that **SK22** had a xanthone derivative. The ^{1}H NMR data of the right-hand ring were similar to those of **SK20**. It showed a chelated hydroxy proton (δ_H 12.87, *s*, 1H), one *singlet* aromatic proton (δ_H 6.29, *s*, 1H) and one methoxy protons (δ_H 3.91, *s*, 3H). The location of these substituents on the right-hand ring of the xanthone nucleus was established by the HMBC data (**Table 85**). In addition, the ^{1}H NMR spectrum exhibited two *para*-aromatic protons [δ_H 7.19 and 7.52 (*s*, 1H each)] and methoxy protons (δ_H 4.07, *s*, 3H). The *para*-aromatic protons were attributed to H-5 and H-8, respectively, according to the ^{1}H chemical shift of H-8 and HMBC correlations of H-5/C-7 (δ_C 145.36) and C-8a (δ_C 114.29) and those of H-8/C-6 (δ_C 155.75) and C-10a (δ_C

152.39). A HMBC correlation between the methoxy protons (δ_H 4.07) and C-6 established the attachment of the methoxyl group at C-6. The NOEDIFF enhancement of methoxy protons, upon irradiation at H-5, confirmed this assignment. According to the chemical shift value of C-7 (δ_C 145.36), C-7 carried a hydroxyl group. Thus, **SK22** had the structure as shown, a new naturally occurring xanthone.



(SK22)

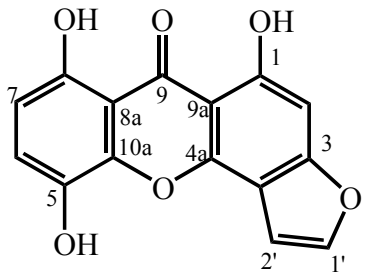
Table 85 The NMR data of compound **SK22** in Acetone-d_6

Position	δ_H (*mult, J* Hz)	δ_C (C-type)	HMBC	NOE
1-OH	12.87 (*s*, 1H)	159.44 (C)	C-1, C-2, C-9a	-
2	6.29 (*s*, 1H)	98.65 (CH)	C-1, C-3, C-4, C-9a	-
3	-	158.73 (C)	-	-
4	-	128.47 (C)	-	-
4a	-	150.80 (C)	-	-
5	7.19 (*s*, 1H)	100.76 (CH)	C-6, C-7, C-9, C-8a, C-10a	OMe-6
6	-	155.75 (C)	-	-
7	-	145.36 (C)	-	-
8	7.52 (*s*, 1H)	108.91 (CH)	C-6, C-7, C-9, C-8a, C-10a	-
8a	-	114.29 (C)	-	-
9	-	180.68 (C=O)	-	-
9a	-	103.17 (C)	-	-
10a	-	152.39 (C)	-	-
OMe-4	3.91 (*s*, 3H)	61.71 (CH_3)	C-4	-
OMe-6	4.07 (*s*, 3H)	57.03 (CH_3)	C-6	H-5

1.3.1.9 Compound SK10

Compound **SK10** was obtained as a pale yellow gum. It showed molecular ion at *m/z* 284, which corresponded to the molecular formula $C_{15}H_8O_6$. The IR spectrum

exhibited absorption bands at 3417 cm^{-1} (a hydroxyl group) and 1676 cm^{-1} (a carbonyl group). The UV spectrum with absorptions bands at 249, 269, 273 and 329 indicated that **SK10** possessed a xanthone chromophore. Its ^{1}H NMR spectrum (**Table 86**) (**Figure 39**) showed signals of two chelated hydroxy protons [δ_H 12.01 and 11.29 (*s*, 1H each)], two *ortho*-couple aromatic protons [δ_H 7.35 and 6.79 (*d*, *J* = 9.0 Hz, 1H each)] and one *double*t aromatic proton (δ_H 6.97, *d*, *J* = 1.0 Hz, 1H). In addition, it contained signals of aromatic protons of a furan ring [δ_H 7.63 (*d*, *J* = 2.0 Hz, 1H) and 7.05 (*dd*, *J* = 2.0 and 1.0 Hz, 1H)] (Inuma, 1996). The ^{13}C NMR spectrum (**Table 86**) (**Figure 40**) showed fifteen carbons: ten quaternary carbons (δ_C 185.0, 162.00, 159.45, 154.30, 148.00, 144.20, 135.00, 110.00, 108.00 and 107.00) and five methine carbons (δ_C 144.72, 123.95, 110.81, 103.56 and 95.44). Two chelated hydroxy protons (δ_H 12.01 and 11.29) were attributed to OH-1 and OH-8, respectively, according to the HMBC correlations of OH-1/C-1 (δ_C 159.45), C-2 (δ_C 99.44) and C-9a (δ_C 102.65) and those of OH-8/C-7 (δ_C 110.98), C-8 (δ_C 154.30) and C-8a (δ_C 108.00). In the HMBC spectrum, the *ortho*-aromatic protons were located at C-6 and C-7 by their HMBC correlations with C-5 (δ_C 135.00), C-8, C-8a and C-10a (δ_C 144.20). In addition, the *singlet* aromatic proton (δ_H 6.97) showed HMBC correlations with C-3 (δ_C 162.20), C-4 (δ_C 110.50) and C-9a, suggesting that this proton was at C-2. The olefinic protons of a furan ring (δ_H 7.63, H-1′ and 7.05, H-2′) gave cross peaks of H-1′/C-3 and C-4 and H-2′/C-3 and C-4a (δ_C 148.00). In NOEDIFF data, irradiation of H-2 did not affect signal intensities of both H-1′ and H-2′. These data implied that the furan ring was fused to C-3 and C-4 with an ether linkage at C-3. Thus, **SK10** had the structure as shown, a new naturally occurring xanthone.



(SK10)

Table 86 The NMR data of compound **SK10** in $CDCl_3$

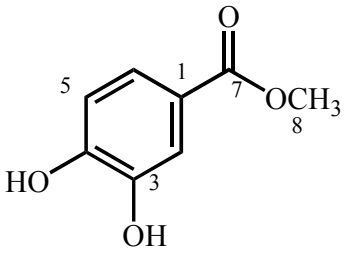
Position	δ_H (*mult, J* Hz)	δ_C (C-type)	HMBC	NOE
OH-1	12.01 (*s*, 1H)	159.45 (C)	C-1, C-2, C-9a	-
2	6.97 (*d*, 1.0, 1H)	95.44 (CH)	C-3, C-4, C-1′, C-9a	-
3	-	162.20 (C)	-	-
4	-	110.50 (C)	-	-
4a	-	148.00 (C)	-	-
5	-	135.00 (C)	-	-
6	6.79 (*d*, 9.0, 1H)	123.95 (CH)	C-5, C-8, C-10a	H-7
7	7.35 (*d*, 9.0, 1H)	110.98 (CH)	C-5, C-8, C-8a	H-6
OH-8	11.29 (*s*, 1H)	154.30 (C)	C-7, C-8, C-8a	-
8a	-	108.00 (C)	-	-
9	-	185.00 (C=O)	-	-
9a	-	102.65 (C)	-	-
10a	-	144.20 (C)	-	-
1′	7.63 (*d*, 2.0, 1H)	144.72 (CH)	C-3, C-4	-
2′	7.05 (*dd*, 2.0, 1.0, 1H)	103.50 (CH)	C-3, C-4, C-4a	-

1.3.3 Benzoic acid derivatives

1.3.3.1 Compound SK17

Compound **SK17** was obtained as a pale yellow gum. The IR spectrum showed absorption bands at 3338 and 1690 cm^{-1} for a hydroxyl group and a carbonyl group, respectively. The UV spectrum exhibited absorption band at λ_{max} 278 nm, indicating that **SK17** possessed an aromatic chromophore. The ^1H NMR spectrum (**Table 87**) (**Figure 42**) showed a presence of a 1,3,4-trisubstituted benzene [δ_H 7.59 (*d*, *J* = 1.8 Hz, 1H), 7.55 (*dd*, *J* = 8.1 and 1.8 Hz, 1H) and 6.90 (*d*, *J* = 8.1 Hz, 1H) and methoxy protons (δ_H 3.88, *s*, 3H). The ^{13}C NMR spectrum (**Table 87**) (**Figure 43**) showed the presence of one carbonyl carbon (δ_C 167.00), three quaternary carbons (δ_C 148.49, 142.14 and 123.90), three methine carbons (δ_C 123.87, 116.61 and 114.88) and one methoxy carbon (δ_C 52.05). The methoxy protons together with its HMBC correlation with the carbon signal at δ_C 167.00 (C-7) indicated the presence of

the methyl ester group. The two aromatic protons at δ_H 7.59 and 7.55 were located at *ortho*-position of an ester carbonyl, on the basis of HMBC correlations between these protons and the carbonyl carbon. The remaining proton was then assigned as H-5. Because no other signals were observed in the ^{1}H NMR spectrum, the substituents at C-3 and C-4 were hydroxyl groups. Thus, **SK17** was determined as protocatechic acid methyl ester which was isolated from fruits of *Euterpe oleracea* (Chin, 2008a).



(SK17)

Table 87 The NMR data of compound **SK17** and protocatechic acid methyl ester

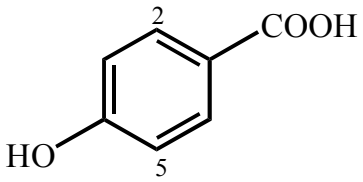
Position	**SK17** ($CDCl_3$)		HMBC	protocatechic acid methyl ester (Acetone-d_6)*	
	δ_H (*mult, J* Hz)	δ_C (C-Type)		δ_H (*mult, J* Hz)	δ_C
1	-	123.90 (C)	-	-	122
2	7.59 (*d*, 1.8, 1H)	116.61 (CH)	C-4, C-6, C-7	7.39 (*d*, 2.0, 1H)	117
3	-	142.14 (C)	-	-	144
4	-	148.49 (C)	-	-	150
5	6.90 (*d*, 8.1, 1H)	114.88 (CH)	C-1, C-3, C-7	6.80 (*d*, 8.3, 1H)	115
6	7.55 (*dd*, 8.1, 1.8, 1H)	123.87 (CH)	C-4, C-5, C-7	7.34 (*dd*, 8.3, 2.0, 1H)	123
7	-	167.00 (C=O)	-	-	166
8	3.88 (*s*, 3H)	52.05 (CH_3)	C-7	3.80 (*s*, 3H)	57

*Miyazawa, 2003

1.3.3.2 Compound SK7

Compound **SK7** was obtained as a yellow gum. Its UV spectrum showed an absorption band at λ_{max} 251 nm while its IR spectrum exhibited absorption bands at 3442 and 1663 cm^{-1} due to a hydroxyl group of carboxylic acid and a conjugated carbonyl group. Its ^{1}H NMR spectrum **(Table 88) (Figure 44)** contained signals for

aromatic protons of a *para*-disubstituted benzene [δ_H 7.95 (*d, J* = 6.9 Hz, 2H) and 6.85 (*d, J* = 6.9 Hz, 2H)]. Comparison of the ^{1}H NMR data suggested that **SK7** was 4-hydroxybenzoic acid which was isolated from fruits of *G. mangostana* (Zadernowski, 2009).



(SK7)

Table 88 The NMR data of compound **SK7** and 4-hydroxybenzoic acid

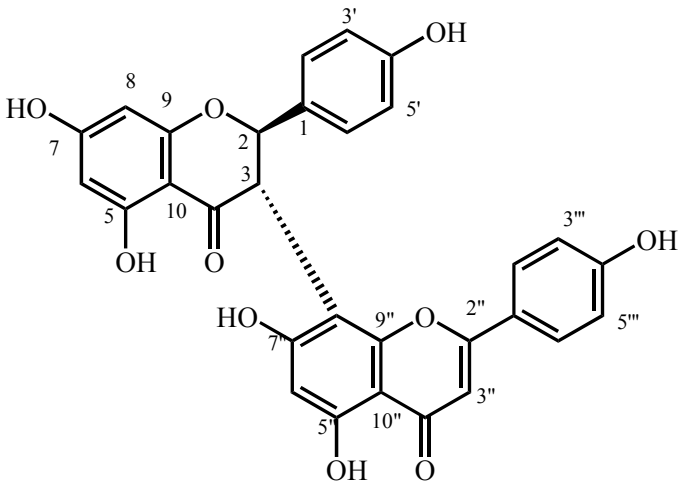
Position	**SK7** ($CDCl_3$+CD_3OD) δ_H (*mult, J* Hz)	4-hydroxybenzoic acid ($CDCl_3$)* δ_H (*mult, J* Hz)
2, 6	7.95 (*d*, 6.9, 2H)	7.96 (*d*, 8.6, 2H)
3, 5	6.85 (*d*, 6.9, 2H)	6.86 (*d*, 8.6, 2H)

*(Hsieh, 2005)

1.3.4 Biflavone

1.3.4.1 Compound SK6

Compound **SK6** was obtained as a yellow gum. Its UV spectrum showed absorption bands at λ_{max} 221, 288 and 335 nm while its IR spectrum exhibited absorption bands at 3420 and 1650 cm^{-1} due to hydroxyl and conjugated carbonyl groups. Its ^{1}H NMR spectrum **(Table 89) (Figure 45)** contained signals of two chelated hydroxy protons [δ_H 13.07 and 12.29)], two *para*-disubstituted benzenes [δ_H 7.09 (*d, J* = 7.8 Hz, 2H), 6.35 (*d, J* = 7.8 Hz, 2H), 7.94 (*d, J* = 8.1 Hz, 2H) and 6.93 (*d, J* = 8.1 Hz, 2H)], a 1,2,3,5-tetrasubstituted benzene [δ_H 6.04 (*s*, 1H) and 5.94 (*s*, 1H)], a *singlet* aromatic proton (δ_H 6.22) and two methine protons [δ_H 5.67 (*d, J* = 12.0 Hz, 1H) and 4.99 (*d, J* = 12.0 Hz, 1H)]. Comparison of the ^{1}H NMR data and the optical rotation of **SK6** ($[\alpha]_D^{29}$ +114.5° (c = 0.05, MeOH)) with those of (+)-volkensiflavone ($[\alpha]_D^{29}$ +133° (c = 0.05, MeOH)), suggested that **SK6** was (+)-volkensiflavone (Sukpondma, 2005).



(SK6)

Table 89 The NMR data of compound **SK6** and (+)-volkensiflavon**e**

Position	**SK6** (DMSO-d_6) δ_H (*mult, J* Hz)	(+)-volkensiflavone (DMSO-d_6) δ_H (*mult, J* Hz)
2	5.67 (*d*, 12.0, 1H)	5.82 (*d*, 12.0, 1H)
3	4.99 (*d*, 12.0, 1H)	5.16 (*d*, 12.0, 1H)
OH-5	12.29 (*s*, 1H)	12.48 (*s*, 1H)
6	6.04 (*s*, 1H)	6.15 (*s*, 1H)
8	5.94 (*s*, 1H)	6.09 (*s*, 1H)
2′,6′	7.09 (*d*, 7.8, 2H)	7.25 (*d*, 8.5, 2H)
3′,5′	6.35 (*d*, 7.8, 2H)	6.49 (*d*, 8.5, 2H)
3″	6.64 (*s*, 1H)	6.80 (*s*, 1H)
OH-5″	13.07 (*s*, 1H)	13.20 (*s*, 1H)
6″	6.22 (*s*, 1H)	6.37 (*s*, 1H)
2‴,6‴	7.94 (*d*, 8.1, 2H)	8.10 (*d*, 9.0, 2H)
3‴, 5‴	6.93 (*d*, 8.1, 2H)	7.09 (*d*, 9.0, 2H)

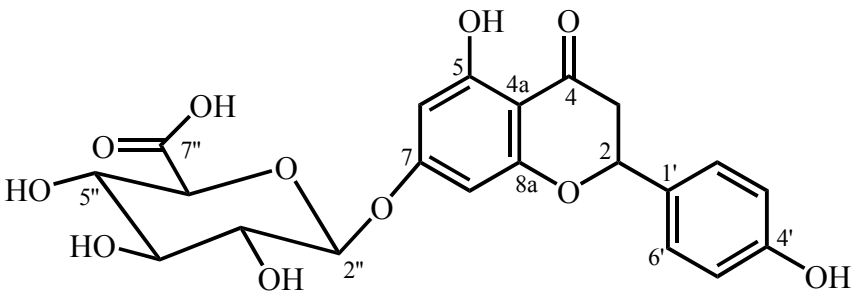
1.3.5 Flavanone glucosides

1.3.5.1 Compound SK23

Compound **SK23** was obtained as a yellow solid, melting at 252-255 °C. The IR spectrum exhibited absorption bands at 3220 cm^{-1} (a hydroxyl of carboxylic acid), 1730 and 1644 cm^{-1} (carbonyl groups). The UV spectrum with absorption bands at 222, 282 and 335 nm indicated that **SK23** had a flavanone chromophore (Cui, 1990).

The ^{1}H NMR spectrum (**Table 90**) (**Figure 46**) contained signals of a flavanone moiety [δ_H 5.39 (*dd*, *J* = 12.6 and 2.7 Hz, 2H), 3.17 (*dd*, *J* = 17.1 and 12.6 Hz, 1H), 2.74 (*dd*, *J* = 17.1 and 12.6 Hz, 1H), 6.23 (*brs*, 1H), 6.19 (*brs*, 1H), 7.32 (*d*, *J* = 8.4 Hz, 2H) and 6.81 (*d*, *J* = 8.4 Hz, 2H)] and signals of glucuronide moiety [δ_H 5.00 (*d*, *J* = 7.2 Hz, 1H), 3.81, (*m*, 1H), 3.80 (*m*, 1H) and 3.78 (*d*, *J* = 9.0 Hz, 1H)]. The presence of the flavanone and glucuronide units was confirmed by ^{1}H-^{1}H COSY and HMBC data (**Table 90**). The ^{13}C NMR spectrum (**Table 90**) (**Figure 47**) consisted of eighteen signals for twenty one carbons, containing two carbonyl (δ_C 197.18 and 182.00), six quaternary carbons (δ_C 165.73, 163.80, 163.24, 157.64, 129.51 and 103.58), ten methine carbons (δ_C 127.68, 114.95, 99.77, 96.78, 95.00, 79.25, 76.23, 75.28, 73.07 and 72.04) and one methylene carbon (δ_C 42.75). The anomeric proton (δ_H 5.00, H-2″) showed HMBC correlation with C-7 (δ_C 163.60), while two methine protons (δ_H 6.23, H-6 and 6.19, H-8) gave a cross peak with C-2″ (δ_C 99.77). These results indicated that glucuronide unit was attached at C-7 of the flavonone unit through an *O*-glycosidic bond.

The relative stereochemistry of the glycuronide moiety was established based on the following NOEDIFF data. The appearance of the anomeric proton as a *doublet* with the large coupling constant (*J* = 7.2 Hz), indicated that it was assigned as a *β*-glucuronide (Cui, 1990). Irradiation of H-3″ (δ_H 3.81) affected signal intensity of H-5″ (δ_H 3.80) while irradiation of H-6″ affected signal intensities of both H-2″ and H-4″. These data confirmed the stereochemistry of the glucuronide moiety. Thus, **SK23** was determined as naringenin 7-*O*-*β*-D-glucuronide (Silberberg, 2006).



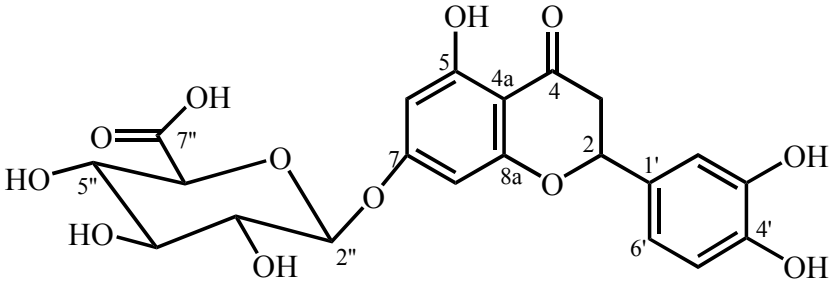
(SK23)

Table 90 The NMR data of compound **SK23** in CD_3OD

Position	δ_H (*mult*, *J* Hz)	δ_C (C-Type)	HMBC	COSY	NOE
2	5.39 (*dd*, 12.6, 2.7, 1H)	79.25 (CH)	C-4, C-1', C-2'	H-3	H-3, H-2', H-6'
3	a: 3.17 (*dd*, 17.1, 12.6, 1H) b: 2.74 (*dd*, 17.1, 2.7, 1H)	42.75 (CH_2)	C-2, C-4, C-4a, C-1'	H-2	H-2
4	-	197.18 (C=O)	-	-	-
4a	-	103.58 (C)	-	-	-
5	-	163.80 (C)	-	-	-
6	6.23 (*brs*, 1H)	95.60 (CH)	C-7, C-8, C-4a, C-2"	-	H-2"
7	-	165.73 (C)	-	-	-
8	6.19 (*brs*, 1H)	96.78 (CH)	C-6, C-7, C-4a, C-2"	-	H-2"
8a	-	163.24 (C)	-	-	-
1'	-	129.51 (C)	-	-	-
2',6'	7.32 (*d*, 8.4, 2H)	127.68 (CH)	C-2, C-3', C-4', C-5'	-	H-3', H-5'
3',5'	6.81 (*d*, 8.4, 2H)	114.95 (CH)	C-1', C-2', C-4', C-6'	-	H-2', H-6'
4'	-	157.64 (C)	-	-	-
2"	5.00 (*d*, 7.2, 1H)	99.77 (CH)	C-7, C-2", C-4"	H-3', H-5'	H-6, H-8
3"	3.80 (*m*, 1H)	76.23 (CH)	C-2", C-3", C-5"	H-2', H-6'	H-5"
4"	3.50 (*m*, 1H)	73.07 (CH)	-	H-3"	-
5"	3.80 (*m*, 1H)	72.04 (CH)	-	-	-
6"	3.78 (*d*, 9.0, 1H)	75.28 (CH)	-	H-3", H-5"	H-2"
7"	-	182.00 (C=O)	-	H-4"	-

1.3.5.2 Compound SK24

Compound **SK24** was obtained as a yellow solid, melting at 267-269 °C. The UV and IR spectra were similar to those of **SK23**, indicating that **SK24** had the same chromophore as **SK23**. The ^{1}H and ^{13}C NMR data (**Table 91**) (**Figures 48** and **49**) were similar to those of **SK23** except that **SK24** contained none of signals for a *para*-disubstituted benzene. These signals were replaced by signals of a 1,2,4-trisubstituted benzene [δ_H 6.93 (*brs*, 1H), 6.78 (*dd*, *J* = 8.4 and 1.8 Hz, 1H) and 6.79 (*d*, *J* = 8.4 Hz, 1H). The location of substituent at C-2 was confirmed by HMBC data (**Table 92)**. Comparison of the optical rotation of **SK24** ($[\alpha]_D^{26}$-42.7° (c = 1.00, MeOH)) with that of the 7-*O*-*β*-glucuronide of eriodictyol ($[\alpha]_D^{30}$-45.2° (c = 1.00, MeOH)), indicated that they had the same relative configuration of glucuronide moeity. Thus, **SK24** was determined as 7-*O*-*β*-glucuronide of eriodictyol which was isolated from *Devallia mariesii* (Cui, 1990).



(SK24)

Table 91 The NMR data of compound **SK24** and 7-*O*-*β*-glucuronide of eriodictyol

Position	**SK25** (CD_3OD)		7-*O*-*β*-glucuronide of eriodictyol (DMSO-d_6)	
	δ_H (*mult, J* Hz)	δ_C (C-Type)	δ_H (*mult, J* Hz)	δ_C
2	5.33 (*dd*, 12.9, 3.3, 1H)	79.25 (CH)	5.28 (*dd*, 12.8, 3.1, 1H)	79.0
3	a: 3.11 (*dd*, 17.4, 12.9, 1H)	42.76 (CH_2)	a: 3.10 (*dd*, 17.0, 12.8, 1H)	42.4
	b: 2.75 (*dd*, 17.4, 3.3, 1H)		b: 2.72 (*dd*, 17.0, 3.1, 1H)	
4	-	197.17 (C=O)	-	197.4
4a	-	103.60 (C)	-	103.6
5	-	165.65 (C)	-	163.1

Table 91 (continued)

Position	**SK25** (CD_3OD)		7-*O*-*β*-glucuronide of eriodictyol (DMSO-d_6)	
	δ_H (*mult, J* Hz)	δ_C (C-Type)	δ_H (*mult, J* Hz)	δ_C
6	6.19 (*d*, 2.4, 1H)	96.75 (CH)	6.16 (*d*, 2.1, 1H)	96.5
7	-	165.71 (C)	-	165.0
8	6.23 (*d*, 2.4, 1H)	95.65 (CH)	6.17 (*d*, 2.1, 1H)	95.5
8a	-	163.14 (C)	-	163.0
1'	-	130.19 (C)	-	129.4
2'	6.93 (*brs*, 1H)	113.42 (CH)	6.92 (*brs*, 1H)	114.6
3'	-	145.10 (C)	-	145.4
4'	-	145.52 (C)	-	146.0
5'	6.79 (*d*, 8.4, 1H)	114.91 (CH)	6.78 (*brs*, 1H)	115.6
6'	6.78 (*dd*, 8.4, 1.8, 1H)	117.91 (CH)	6.78 (*brs*, 1H)	118.3
2"	5.00 (*m*, 1H)	99.75 (CH)	5.07 (*d*, 8.0, 7.5, 1H)	99.1
3"	3.50 (*m*, 1H)	76.22 (CH)	3.51 (*t*, 5.3, 1H)	72.9
4"	3.50 (*m*, 1H)	73.06 (CH)	3.53 (*t*, 8.00, 1H)	75.7
5"	3.50 (*m*, 1H)	72.04 (CH)	3.62 (*dd*, 9.5, 8.0, 1H)	71.4
6"	3.78 (*m*, 1H)	75.26 (CH)	4.05 (*d*, 9.5, 1H)	75.5
7"	-	182.00 (C=O)	-	170.2

Table 92 Major HMBC, ^{1}H-^{1}H COSY and NOEDIFF data of compound **SK24**

Proton	HMBC	COSY	NOE
H-2	C-4, C-1', C-8a	H-3	H-2', H-6'
H-3	C-2, C-4, C-4a, C-1'	H-2	H-2, H-2'
H-6	C-5, C-7, C-8, C-4a	-	H-2"
H-8	C-6, C-7, C-4a	-	H-2"
H-2'	C-2, C-1', C-4', C-6'	H-6'	H-2
H-5'	C-1', C-3'	H-6'	-
H-6'	C-2, C-1', C-2', C-4'	H-2', H-5'	H-2

Table 92 (continued)

Proton	HMBC	COSY	NOE
H-2"	-	H-3"	H-6, H-8, H-4", H-6"
H-3"	C-4", C-6"	-	-
H-4"	C-5", C-6"	-	-
H-5"	C-6"	-	-
H-6"	C-4", C-5"	H-5"	H-2"

PART II

CHEMICAL CONSTITUENTS FROM THE ROOTS OF
CLERODENDRUM PETASITES S. MOORE

CHAPTER 2.1

INTRODUCTION

2.1.1 Introduction

Clerodendrum petasites S. Moore, belongs to the family Verbenaceae. *C. petasites* is erect, shrub or herb, 1-2 m high, dark brown color, and is widely spread over topics long roadside in hill of evergreen forest. Flowers are long tubes with red color, calyx is cup shaped, typically 5 lobes. Leaves whorled with 3-5 per node or opposite, sessile or subsessile, 3-4 inch long. Flowers grow in Aug-Nov. The Thai name is Thao Yaai Mom (วุฒิ, 2540). Leaves are smoked to relieve asthma. Its roots are used as expectorant, antipyretic and antidote against venom and treat insect bites and fever (Upo, 2005).

2.1.2 Review of Literatures

Chemical constituents from the genus *Clerodendrum*

Plant in the genus *Clerodendrum* (verbenaceae) is well known to be rich in variety of compounds, e.g., triterpenes (Jia, 2007; Vu, 2006; Nan, 2006), steroids (Vu, 2006; Shehata, 2001), phenylethanoid glycosides (Li, 2005; Nan, 2005b), diterpenes (Sultana, 2005; Pandey, 2005; Hosny, 2003), flavoniods (Nan, 2005a; Hazekamp, 2001; Rahman, 2000) and iridiod glycosides (Kanchanapoom, 2005), hydrobenzofuran (Yang, 2002). Some of these compounds showed interesting biological and pharmacological activities such as anthelmintic activity (Pal, 2007), antioxidant activity (Chae, 2007; Nyegue, 2007; Hwang, 2007; Le, 2006; Chae, 2006), antifungal activity (Nyegue, 2007; Roy, 1996, 1995), anti-inflammatory (Hwang, 2007; Park, 2007), hepatoprotective activity (Vidya, 2007), antisnake venom activity (Lobo, 2006) and cytotoxic activity (Hosny, 2003).

Chemical constituents isolated from the genus *Clerodendrum* up to the year 2001 have been reported (Boonsri, 2004). The continuing search using SciFinder database revealed additional chemical constituents in the year 2005 up to 2008 which were summarized in **Table 93**.

Table 93 Compounds from the *Clerodendrum* genus

Scientific name	Investigated part	Compounds	Structures	References
C. buchholzii	leaves	benzaldehyde	**7a**	Nyegue, M. A., *et al*., 2005, 2007
		octen-3-ol	**2c**	
C. bungei	-	clerodendronoside	**24p**	Li, Y. *et al*, 2005
		acteoside	**24a**	
		isoacteoside	**24k**	
		cistanoside C	**24b**	
		jionoside C	**24o**	
		leucosceptoside A	**24d**	
		cistanoside D	**24c**	
		campneoside I	**24e**	
		campneoside II	**24f**	
		cistanoside F	**24n**	
	-	*β*-sitosterol	**25c**	Gao, L. *et al*., 2003a
		taraxerol	**27k**	
		glochidone	**27j**	
		glochidonol	**27f**	
		glochidiol	**27e**	
	aerial parts	5-*O*-ethylclero-indicin D	**21a**	Yang, H. *et al.,* 2002
		bungein A	**22a**	
		betulinic acid	**27l**	
		hispidulin	**12c**	

Table 93 (continued)

Scientific name	Investigated part	Compounds	Structures	References
		pentacosane	**2b**	
		clerosterol	**25a**	
		acteoside	**24a**	
		clerosterol 3-*O*-*β*-D-glucopyranoside	**26b**	
		cleroindicin A	**3a**	
		cleroindicin C	**21b**	
		cleroindicin E	**21d**	
		cleroindicin F	**21c**	
		martinoside	**24g**	
C. calamitosum	leaves and stems	phaeophorbide a	**23e**	Cheng, H.-H., *et al*., 2001
		vincristine	**6c**	
		camptothecin	**6a**	
		pheophytin a *O*-allomer	**23a**	
		methyl 10-hydroxy-pheophorbide a	**23b**	
		10-hydroxy pheophorbide a	**23c**	
		13-ethenyl-18-ethyl-7,8-dihydro-3-(methoxycarbonyl)-5-(methoxyoxoacetyl)-2,8,12,17-tetramethylpheophorbide	**23f**	

Table 93 (continued)

Scientific name	Investigated part	Compounds	Structures	References
		methylpheo-phorbide a	**23d**	
		purpurin-7-trime-thyl ester	**23g**	
C. canesens	whole plant	lupeol	**27b**	Jia, L., *et al.*, 2007
		α-amyrin 3-undeca-noate	**27i**	
		lupeol acetate	**27c**	
		lupeol 3-palmitate	**27d**	
		melastomic acid	**27m**	
		β-amyrin acetate	**27h**	
		betulinic acid	**27l**	
C. chinense	aerial part	5-*O*-β-glucopy ranosylharpagide	**18c**	Kanchana-poom, Y., *et al.*, 2005
		harpagide	**18b**	
		melittoside	**18d**	
		monomelittoside	**18a**	
		cornoside	**9b**	
		rengyoxide	**3b**	
		rengyolone	**21e**	
		rengyoside B	**9a**	
C. cyrtophyllum	roots	friedelin	**27a**	Vu, D. H., *et al.*, 2006
		uncinatone	**17a**	
		22-dehydroclero-sterol	**25b**	

Table 3 (continued)

Scientific name	Investigated part	Compounds	Structures	References
C. cyrtophyllum	twigs and leaves	cirsilineol	**12a**	Le, C. N., *et al.,* 2006
		cirsilineol-4'-*O*-*β*-D-glucopyranoside	**13a**	
	leaves	phaeophorbide a	**23e**	Cheng, H.-H., *et al*., 2001
		vincristine	**6c**	
		camptothecin	**6a**	
		pheophytin a *O*-allomer	**23a**	
		methyl 10-hydroxy-pheophorbide a	**23b**	
		10-hydroxy-pheophorbide a	**23c**	
		13-ethenyl-18-ethyl-7,8-dihydro-3-(methoxycarbonyl)-5-(methoxyoxoacetyl)-2,8,12,17-tetramethylpheophorbide	**23f**	
		methylpheophorbide a	**23d**	
		purpurin-7-trimethyl ester	**23g**	
C. fragrans	leaves	*β*-sitosterol	**25c**	Gao, L., *et al.,* 2003b
		clerosterol	**25a**	
		daucosterol	**26a**	
		caffeic acid	**7d**	

Table 93 (continued)

Scientific name	Investigated part	Compounds	Structures	References
		kaempferol	**14a**	
		5,4'-dihydroxy-kaempferol-7-*O*-β-rutinoside	**15a**	
		acteoside	**24a**	
		leucoseceptoside A	**24d**	
C. grayi	leaves	prunasin	**8a**	Miller, R. E.,
		lucumin	**8b**	*et al.*, 2006
C. indicum	-	clerodendrone	**17b**	Ravindranath,
		hispidulin	**12c**	N., *et al.*, 2003
C. inerme	aerial parts	4α-methyl-24β-ethyl-5α-cholesta-14,25-dien-3β-ol	**25f**	Pandey, R., *et al.*, 2007
		24β-ethylcholesta-5,9(11),22*E*-trien-3β-ol	**25e**	
		betulinic acid	**27l**	
	-	lupeol	**27b**	Nan, H.,
		magnificol	**27n**	*et al.*, 2006
		glutinone	**10b**	
		glutinol	**27p**	
		3-*O*-acetyloleanolic aldehyde	**27o**	
		uncinatone	**17a**	
	aerial parts	pentadecanoic acid β-D-glucoside	**5a**	Pandey, R., *et al.*, 2006

Table 93 (continued)

Scientific name	Investigated part	Compounds	Structures	References
		stigmasterol gluco-side	**26a**	
		acacetin	**12e**	
		apigenin	**12d**	
	aerial parts	stigmasterol	**25d**	Nan, H., *et al*.,
		betulinic acid	**27l**	2005a
		acacetin	**12e**	
		syringic acid	**7b**	
		p-methoxybenzoic acid	**7c**	
		apigenin	**12d**	
		daucosterol	**26a**	
	aerial parts	2-(3-methoxy-4-hy-droxylphenyl)ethyl-*O*-2",3"-diacetyl-α-L-rhamnopyrano-syl-(1$\rightarrow$3)-4-*O*-(*E*)-feruloyl-β-D-gluco-pyranoside	**24j**	Nan, H., *et al*., 2005b
		monomelittoside	**18a**	
		melittoside	**18d**	
		inerminoside A1	**18e**	
		verbascoside	**24a**	
		isoverbascoside	**24k**	
		campneoside I	**24e**	

Table 93 (continued)

Scientific name	Investigated part	Compounds	Structures	References
C. inerme	leaves	inerme A	**20c**	Pandey, R.,
		inerme B	**20d**	*et al*., 2005
		14,15-dihydro-15β-methoxy-3-epicary-optin	**20g**	
	aerial parts	4α-methyl-24β-ethyl-5α-cholesta-14,25-dien-3β-ol	**25f**	Pandey, R., *et al*., 2003
		24β-ethylcholesta-5,9(11),22*E*-trien-3β-ol	**25e**	
		11-pentacosanone	**4a**	
		6-nonacosanone	**4b**	
		clerodermic acid	**10c**	
	aerial parts	sammangaoside A	**19a**	Kanchana-
		sammangaoside B	**19b**	poom, T.,
		sammangaoside C	**18f**	*et al*., 2001
		benzyl-*O*-β-D-glu-copyranoside	**16d**	
		salidroside	**16e**	
		melittoside	**18d**	
		monomelittoside	**18a**	
		acteoside	**24a**	
		isoacteoside	**24k**	
		descaffeoylverbas-coside	**16b**	

Table 93 (continued)

Scientific name	Investigated part	Compounds	Structures	References
		leukoceptoside A	**24d**	
		darendoside B	**16c**	
		(*Z*)-3-hexenyl-*β*-glucopyranoside	**5b**	
		leonuriside A	**16j**	
		seguinoside K	**16f**	
		dehydrodiconiferyl-4-*O*-*β*-D-glucopyranoside alcohol	**16h**	
		phenylmethyl 2-*O*-*β*-D-xylopyranosyl-*β*-D-glucopyranoside	**16i**	
		[2*S*-[2*α*,3*β*,5(*E*)]]-[2,3-dihydro-2-(4-hydroxy-3,5-dimethoxyphenyl)-5-(3-hydroxy-1-propenyl-7-methoxy-3-benzofuranyl]methyl-*β*-D-glucopyranoside	**16g**	
C. infortunatum	leaves	daucosterol	**26a**	Pal, D. K., *et al*., 2007
		tetratriacontanol	**2a**	

Table 93 (continued)

Scientific name	Investigated part	Compounds	Structures	References
		melissic acid lupeyl ester	**27g**	
C. myricoides	-	myricoidine	**6b**	Kebenei, J. S., *et al*., 2004
C. petasites	aerial parts aerial parts	arbutin hispidulin	**16a** **12c**	Thongchai, W., *et al*., 2007 Hazekamp, A., *et al*., 2001
C. phlomidis	aerial parts	clerosterol tetratriacontanol	**25a** **2a**	Pandey, R., *et al*., 2008
C. serratum	roots leaves	ursolic acid 5-hydroxyl-10-*O*-cinnamoyloxy-tarennoside 17-aldehydedeyl-oxy-19-β-D-glu-copyranosyloxy-lab-8,13(*E*)-dien-15-ol	**27q** **11a** **11b**	Vidya, S. M., *et al*., 2007 Chen, J.-C., *et al*., 2001
C. splendens	aerial parts	splendensin A splendensin B	**20a** **20b**	Hosny, M., *et al*., 2003
C. splendens	leaves	22-dehydroclero-sterol apigenin	**25b** **12d**	Shehata, A. H., *et al*., 2001

Table 93 (continued)

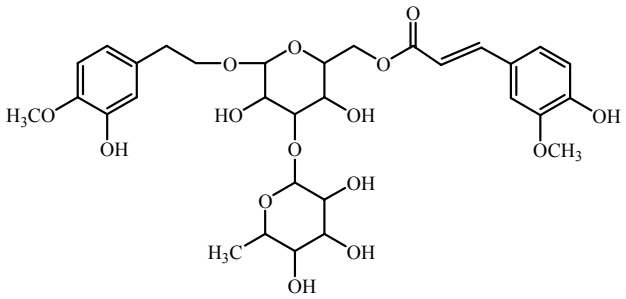
Scientific name	Investigated part	Compounds	Structures	References
		apigenin-7-*O*-glucoside	**13b**	
		3',4',7-trihydroxyflavone-7-*O*-glucoside	**13c**	
C. trichotomum	-	2"-acetylmartynoside	**1b**	Chae, S., *et al*., 2007
		3"-acetylmartynoside	**1c**	
	leaves	acteoside	**24a**	Hwang, W. G., *et al*., 2007
	-	trichotomoside	**24m**	Chae, S., *et al*., 2006
	-	isoacteoside	**24k**	Chae, S., *et al*., 2005
	-	jionoside D	**24i**	Chae, S., *et al*., 2004
	stems	acteoside	**24a**	Kang, D. G., *et al*., 2003
		leucosceptoside A	**24d**	
		martynoside	**24h**	
		isoacteoside	**24k**	
		isomartynoside	**1a**	
	leaves	apigenin-7-*O*-*β*-D-glucuronide	**13d**	Sohn U.-D., *et al*., 2003

Table 93 (continued)

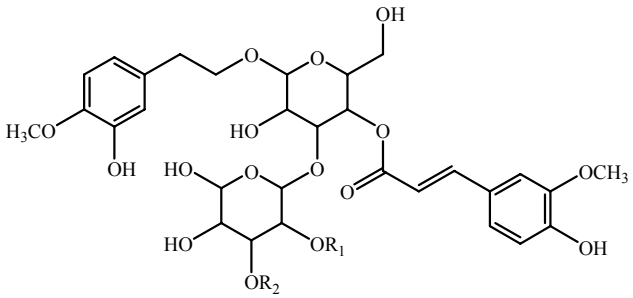
Scientific name	Investigated part	Compounds	Structures	References
	stems	acteoside isoacteoside leucosceptoside A plantainoside C jionoside D martynoside isomartynoside	**24a** **24k** **24d** **24l** **24i** **24h** **1a**	Kim, H. J., *et al*., 2001
C. viscosum	leaves	8-(acetyloxy)-5-[(2*S*,5*R*)-hexahydro-5-hydroxyfuro-[2,3-b]furan-2-yl]-octahydro-5,6-dimethylneoclerodane 8-(acetyloxy)-5-[(2*S*,5*S*)-hexahydro-5-hydroxyfuro-[2,3-b]furan-2-yl] octahydro-5,6-dimethylneoclerodane	**20f** **20e**	Sultana, N., *et al.*, 2005

Structures of Compounds Isolated from Plants of the genus *Clerodendrum*

1. Acetylmartynosides



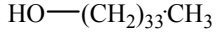
1a : isomartynoside



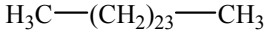
1b : R_1 = Ac, R_2 = H 2"-acetylmartynoside

1c : R_1 = H, R_2 = Ac 3"-acetylmartynoside

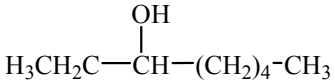
2. Alkanes



2a : tetratriacontanol

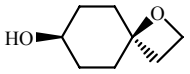


2b : pentacosane

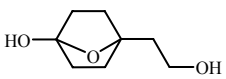


2c : octen-3-ol

3. Alicyclics

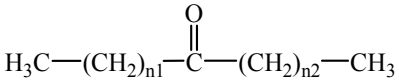


3a : cleroindicin A



3b: rengyoxide

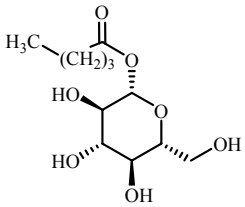
4. Aliphatic ketones



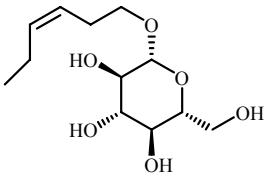
4a : n1 = 9, n2 = 13 11-pentacosanone

4b : n1 = 4, n2 = 22 6-nonacosanone

5. Aliphatic glycosides

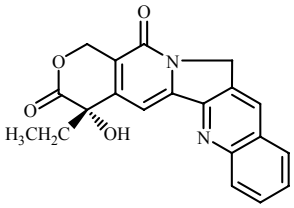


5a : pentadecanoic acid *β*-D-glucoside

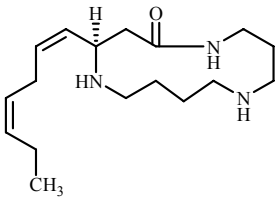


5b : (*Z*)-3-hexenyl-*β*-glucopyranoside

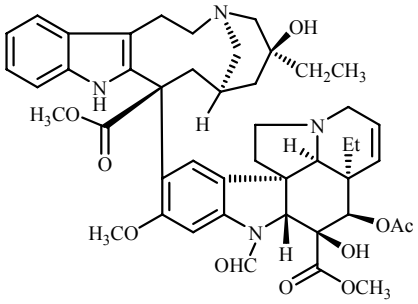
6. Alkaloids



6a : camptothecin

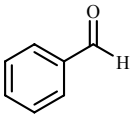


6b : myricoidine

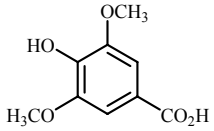


6c : vincristine

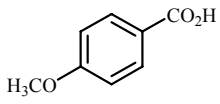
7. Benzenoids



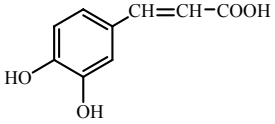
7a : benzaldehyde



7b : syringic acid

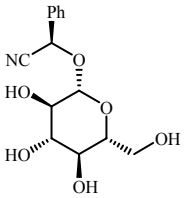


7c : *p*-methoxybenzoic acid

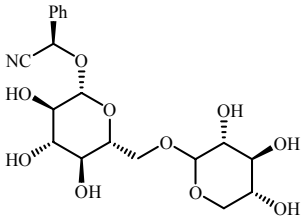


7d : caffeic acid

8. Cyanogenic derivertives

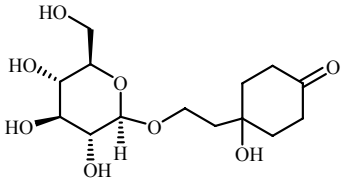


8a : prunasin

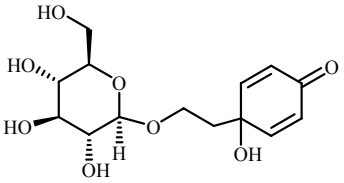


8b : lucumin

9. Cyclohexyl ethanosides

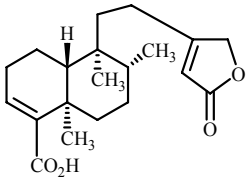


9a : rengyoside B

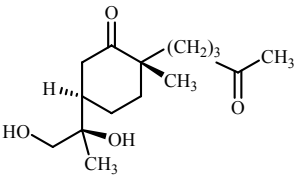


9b : cornoside

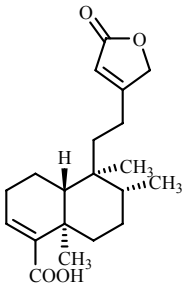
10. Diterpenes



10a : clerodermic acid

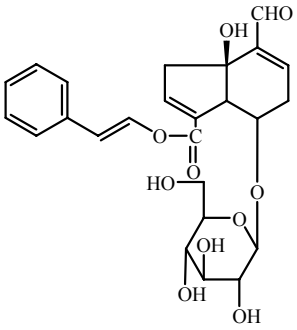


10b : glutinone

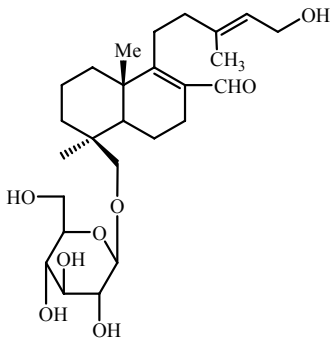


10c : clerodermic acid

11. Diterpene glycosides

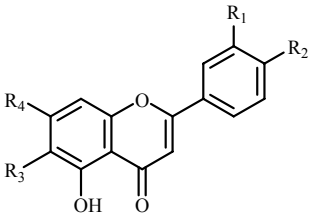


11a : 5-hydroxyl-10-*O*-cinna moyloxy-tarennoside



11b : 17-aldehydedeyloxy-19-*β*-D-glucopy-ranosyl-oxylab-8,13(*E*)-dien-15-iol

12. Flavones



12a : R_1 = OH, R_2 = OCH_3, R_3 = OCH_3, R_4 = OCH_3 cirsilineol

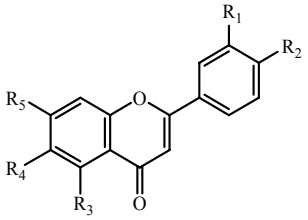
12b : R_1 = H, R_2 = OCH_3,R_3 = OCH_3, R_4 = OH pectolinarigenin

12c : R_1 = H, R_2 = OH, R_3 = OCH_3, R_4 = OH hispidulin

12d : R_1 = H, R_2 = OH, R_3 = H, R_4 = OH apigenin

12e : R_1 = H, R_2 = OCH_3, R_3 = H, R_4 = OH acacetin

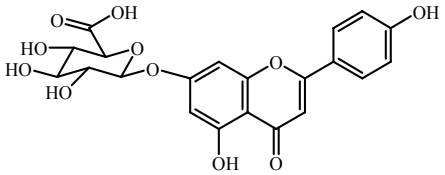
13. Flavone glycosides



13a : R_1 = OCH_3, R_2 = *O*-glu, R_3 = OH, R_4 = OCH_3, R_5 = OCH_3 cirsilineol-4'-*O*-β-D-glucopyranoside

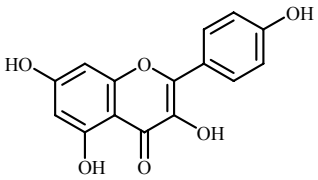
13b : R_1 = H, R_2 = OH, R_3 = OH, R_4 = H, R_5 = *O*-glu apigenin-7-*O*-glucoside

13c : R_1 = OH, R_2 = OH, R_3 = H, R_4 = H, R_5 = *O*-glu 3',4',7-trihydroxy-flavone-7-*O*-glucoside



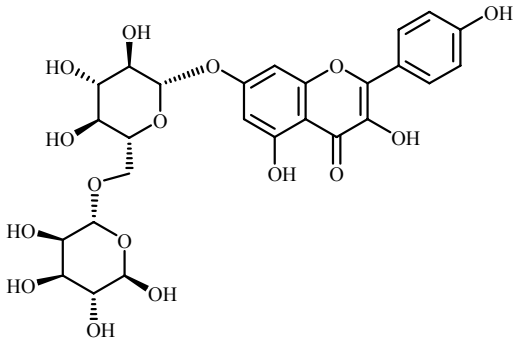
13d : apigenin-7-β-D-glucuronide

14. Flavonol



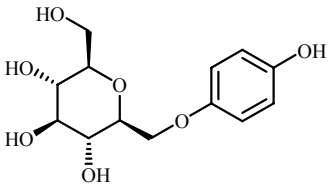
14a : kaempferol

15. Flavonol glycoside

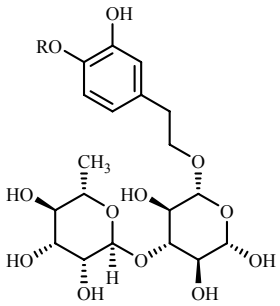


15a : 5,4'-dihydroxy-kaempferol-7-*O*-*β*-rutinoside

16. Glycosides

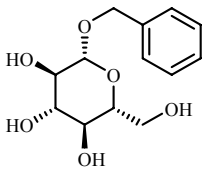


16a : arbutin

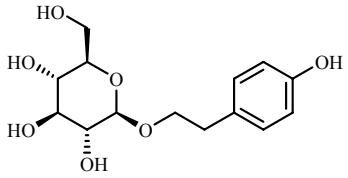


16b : R = H descaffeoylverbascoside

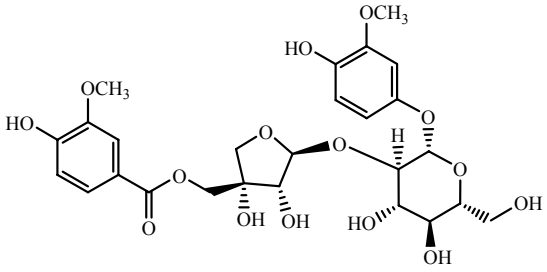
16c : R = CH_3 darendoside B



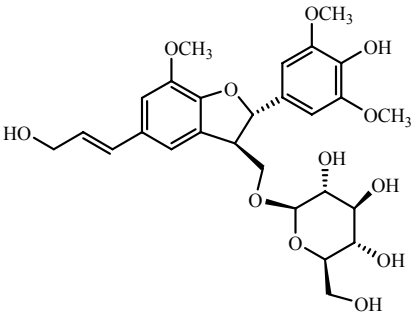
16d : benzyl-*O*-*β*-D-glucopyranoside



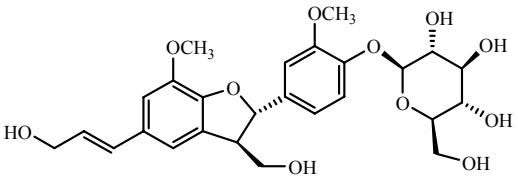
16e : salidroside



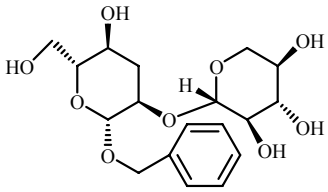
16f : seguinoside K



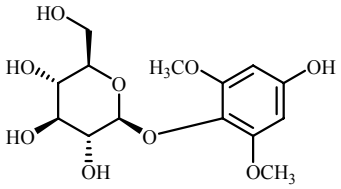
16g : [2*S*-[2α,3β,5(*E*)]]-[2,3-dihydro-2-(4-hydroxy-3,5-dimethoxyphenyl)-5-(3-hydroxy-1-propenyl)-7-methoxy-3-benzofuranyl]methyl, β-D-glucopyranoside



16h : dehydrodiconiferyl 4-*O*-β-D-glucopyranoside alcohol

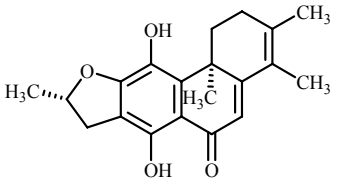


16i : phenylmethyl 2-*O*-β-D-xylopyranosyl-β-D-glucopyranoside

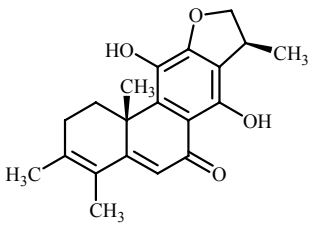


16j : leonuriside A

17. Hydroquinone diterpenes

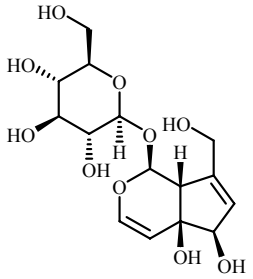


17a : uncinatone

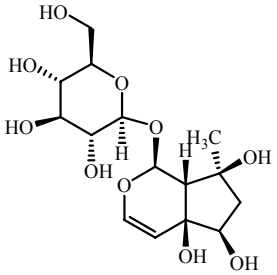


17b : clerodendrone

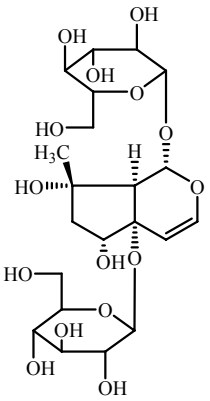
18. Iridoid glycosides



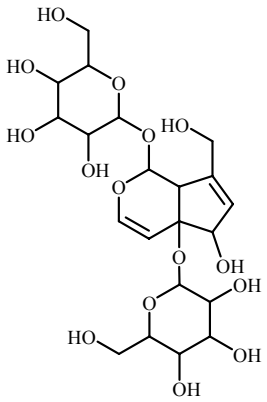
18a : monomelittoside



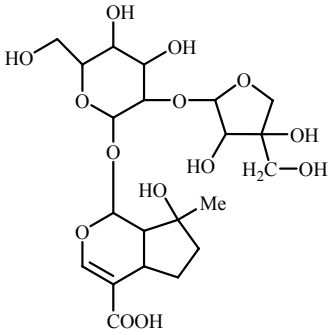
18b : harpagide



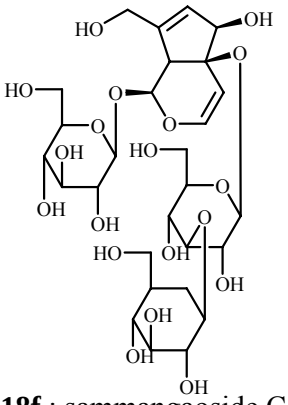
18c : 5-*O*-*β*-glucopyranosylharpagide



18d : melittoside

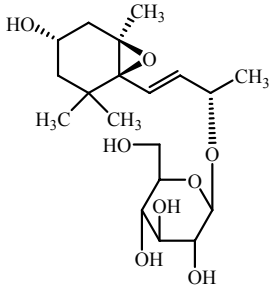


18e : inerminoside A1

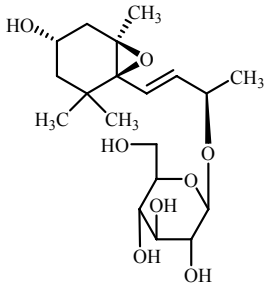


18f : sammangaoside C

19. Megastigmane glycosides

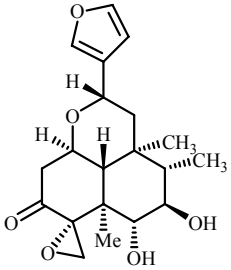


19a : sammangaoside A

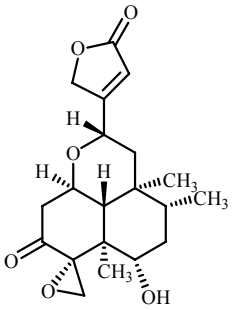


19b : sammangaoside B

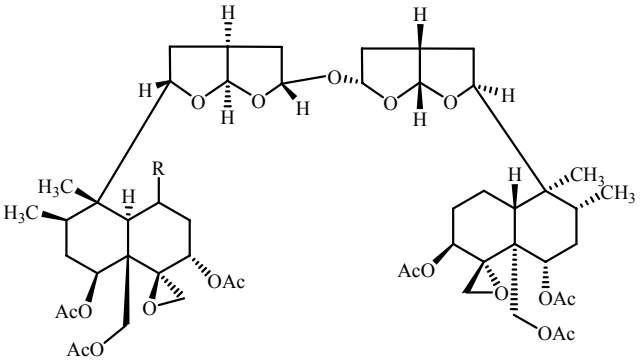
20. Neo-clerodane diterpenes



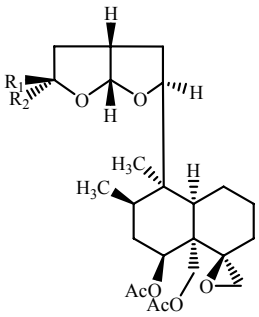
20a : splendensin A



20b : splendensin B

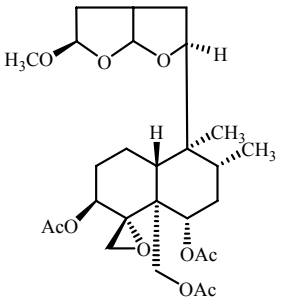


20c : R = H inerme A
20d : R = OCH_3 inerme B



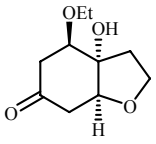
20e : R_1 = OH, R_2 = H 8-(acetyloxy)-5-[(2*S*,5*S*)-hexahydro-5-hydroxyfuro[2,3-b]furan-2-yl]octahydro-5,6-dimethylneoclerodane

20f : R_1 = H, R_2 = OH 8-(acetyloxy)-5-[(2*S*,5*R*)-hexahydro-5-hydroxyfuro[2,3-b]furan-2-yl]octahydro-5,6-dimethylneoclerodane

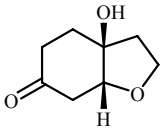


20g :14,15-dihydro-15β-methoxy-3-epicaryoptin

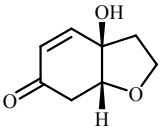
21. Perhydrozofurans



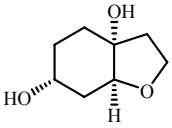
21a : 5-*O*-ethylcleroindicin D



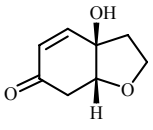
21b : cleroindicin C



21c : cleroindicin F

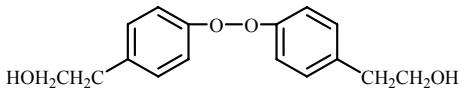


21d : cleroindicin E



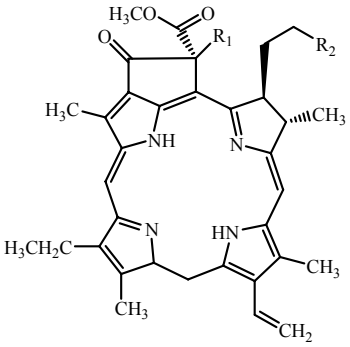
21e : rengyolone

22. Peroxide dimer

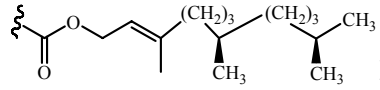


22a : bungein A

23. Peptides



23a : R_1 = OH

R_2 = 

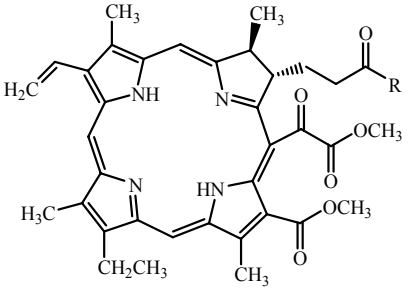
pheophytin a *O*-allomer

23b : R_1 = OH R_2 = $COOCH_3$ methyl 10-hydroxypheophorbide a

23c : R_1 = OH R_2 = COOH 10-hydroxypheophorbide a

23d : R_1 = H R_2 = $COOCH_3$ methyl pheophorbide a

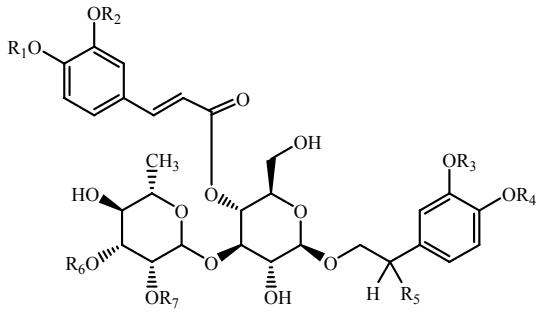
23e : R_1 = H R_2 = COOH phaeophorbide a



23f : R = OH 13-ethenyl-18-ethyl-7,8-dihydro-3-(methoxycarbonyl)-5-(methoxyoxoacetyl)-2,8,12,17-tetramethylpheophorbide

23g : R = OCH_3 purpurin-7-trimethyl ester

24. Phenylethanoid glycosides



24a : $R_1 = R_2 = R_3 = R_4 = R_5 = R_6 = R_7 = H$ acteoside (verbascoside)

24b : $R_1 = R_2 = R_4 = R_5 = R_7 = H$, $R_3 = CH_3$ cistanoside C

24c : $R_1 = R_4 = R_5 = R_6 = R_7 = H$, $R_2 = R_3 = CH_3$ cistanoside D

24d : $R_1 = R_3 = R_4 = R_5 = R_6 = R_7 = H$, $R_2 = CH_3$ leucoseceptoside A

24e : $R_1 = R_2 = R_3 = R_4 = R_6 = R_7 = H$, $R_5 = OCH_3$ campneoside I

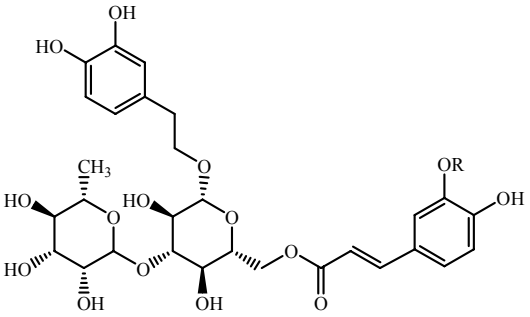
24f : $R_1 = R_2 = R_3 = R_4 = R_6 = R_7 = H$, $R_5 = OH$ campneoside II

24g : $R_1 = R_4 = CH_3$, $R_2 = R_3 = R_5 = R_6 = R_7 = H$ martinoside

24h : $R_1 = R_3 = R_5 = R_6 = R_7 = H$, $R_2 = R_4 = CH_3$ martynoside

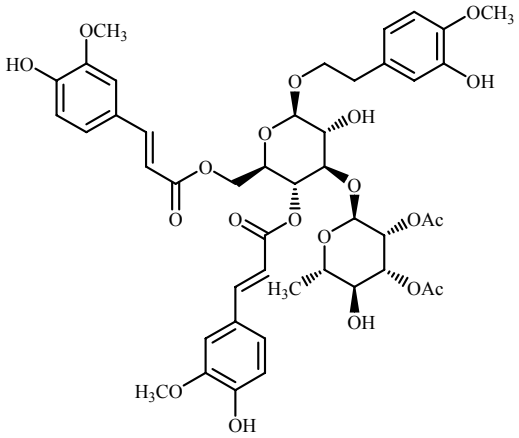
24i : $R_1 = R_2 = R_3 = R_5 = R_6 = R_7 = H$, $R_4 = CH_3$ jionoside D

24j : $R_1 = R_4 = R_5 = H$, $R_2 = R_3 = CH_3$, $R_6 = R_7 = Ac$ 2-(3-methoxy-4-hydroxylphenyl)ethyl-*O*-2",3"-diacetyl-α-L-rhamnopyranosyl-(1-3)-4-*O*-(*E*)-feruloyl-β-D-glucopyranoside

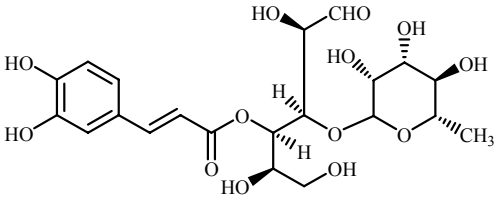


24k : R = H isoacteoside (isoverbascoside)

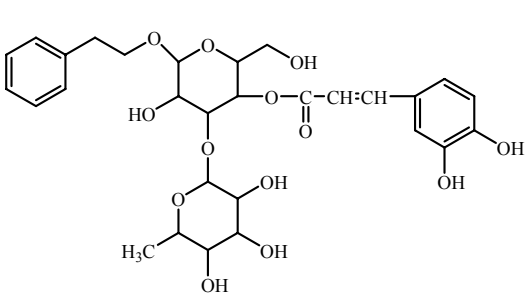
24l : $R = CH_3$ plantainoside C



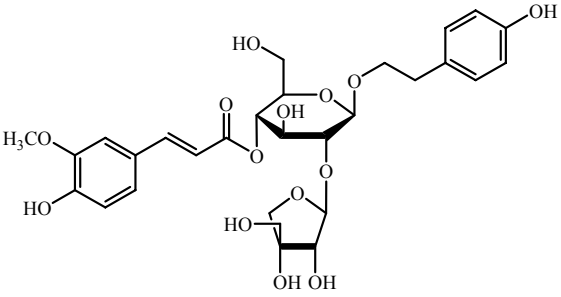
24m : trichotomoside



24n : cistanoside F

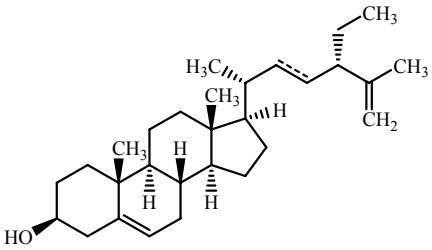


24o : jionoside C



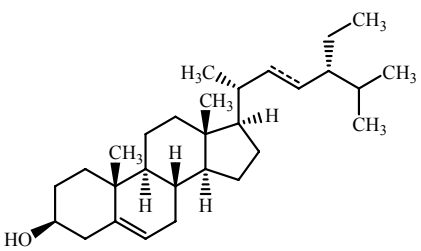
24p : clerodendronnoside

25. Steroids



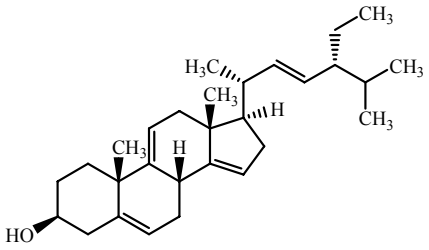
25a : double bond clerosterol

25b : single bond 22-dehydroclerosterol

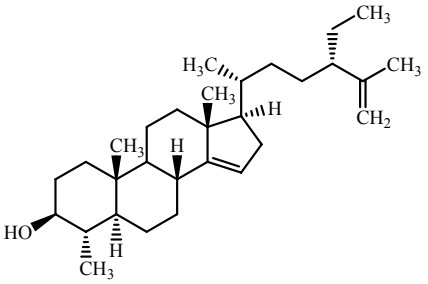


25c : single bond β-sitosterol

25d : double bond stigmasterol

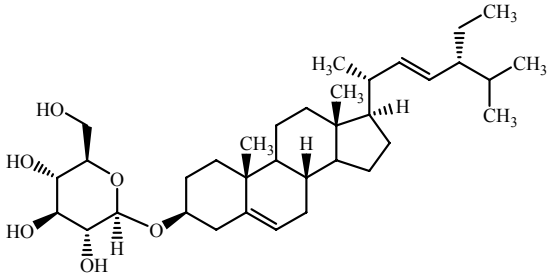


25e : 24β-ethylcholesta-5, 9(11),22*E*-trien-3β-ol

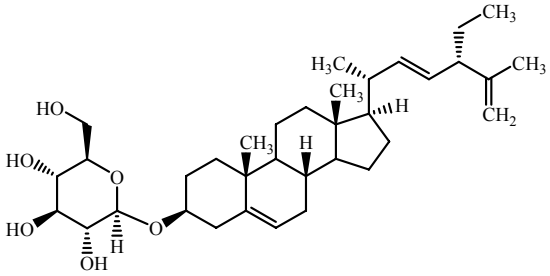


25f : 4α-methyl-24β-ethyl-5α-cholesta-14,25-dien-3β-ol

26. Steroid glycosides

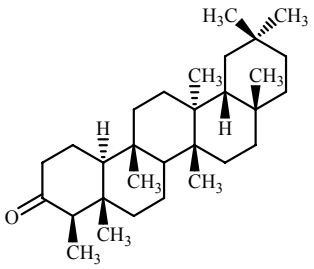


26a : daucosterol (stigmasterol glucoside)

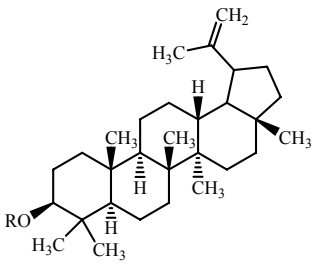


26b : clerosterol 3-*O*-β-D-glucoside

27. Triterpenes

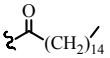


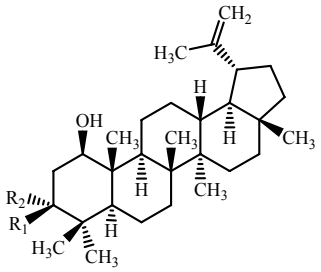
27a : friedelin



27b : R = H lupeol

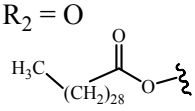
27c : R = Ac lupeol acetate

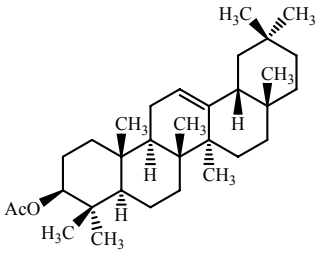
27d : R =  lupeol 3-palmitate



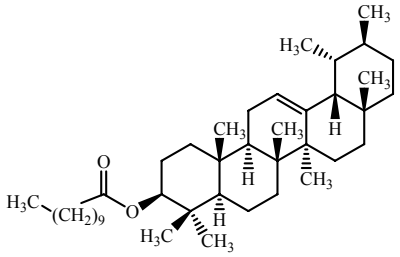
27e : R_1 = H , R_2 = OH glochidiol

27f : $R_1 + R_2$ = O glochidonol

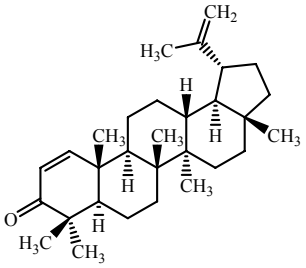
27g : R_1 =  R_2 = H melissic acid lupeyl ester



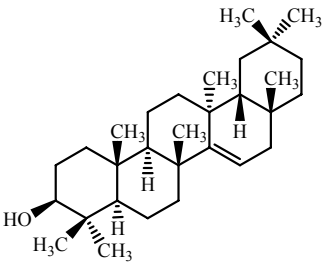
27h : β-amyrin acetate



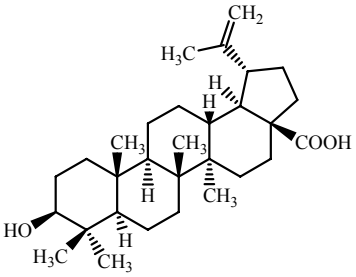
27i : α-amyrin 3-undecanoate



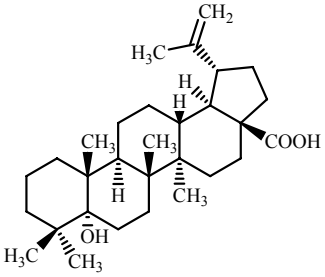
27j : glochidone



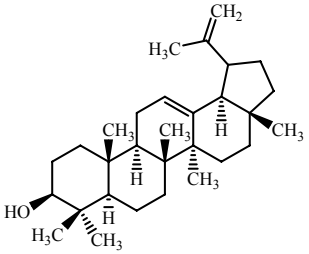
27k : taraxerol



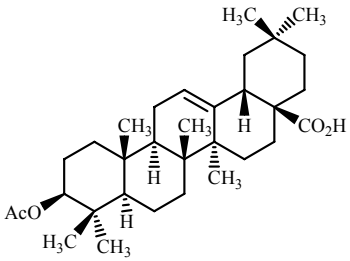
27l : betulinic acid



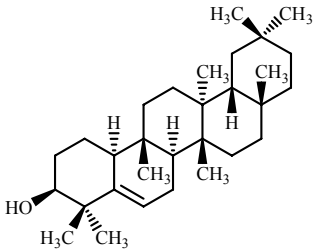
27m : melastomic acid



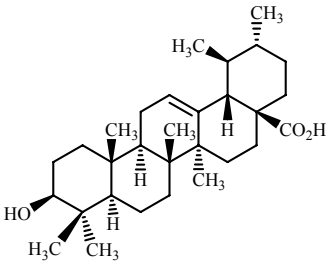
27n : magnificol



27o : 3-*O*-acetyloleanolic aldehyde



27p : glutinol



27q : ursolic acid

2.1.3 The Objectives

Based on the literature search, phytochemical investigation on the aerial part (Hazekamp, 2001) and (Thongchai, 2007) of *C. petasites* resulted in the isolation of flavonoids and glycoside derivatives. We are interested in investigation of its roots in order to separate additional chemical constituents. This research involved isolation, purification and structure elucidation of chemical constituents from the roots of *C. petasites* which were collected at Songkhla province.

CHAPTER 2.2

EXPERIMENTAL

2.2.1 Chemical and instrument

Melting points were determined on an electrothermal melting point apparatus (Electrothermal 9100) and reported without correction. Infrared spectra (IR) were obtained on a Perkin Elmer Spectrum GX FT-IR system and recorded on wavenumber (cm^{-1}). ^{1}H and ^{13}C-Nuclear magnetic resonance spectra (^{1}H and ^{13}C NMR) were recorded on a FTNMR, Bruker Avance 300 MHz or 500 MHz spectrometers using tetramethylsilane (TMS) as an internal standard. Spectra were recorded as chemical shift parameter (δ) value in ppm down field from TMS (δ 0.00). Ultraviolet spectra (UV) were measured with UV-160A spectrophotometer (SHIMADSU). Principle bands (λ_{max}) were recorded as wavelengths (nm) and log ε in methanol solution. Optical rotations were measured in methanol or chloroform solution with sodium D line (590 nm) on a JASCO P-1020 automatic polarimeter. Quick column chromatography, thin-layer chromatography (TLC) and precoated thin-layer chromatography were performed on silica gel 60 GF_{254} (Merck) or reverse-phase C-18 silica gel. Column chromatography was performed on silica gel (Merck) type 100 (70-230 Mesh ASTM), Sephadex LH-20 or reverse-phase C-18 silica gel. The solvents for extraction and chromatography were distilled at their boiling point ranges prior to use except for petroleum ether, chloroform, ethanol and ethyl acetate which were analytical grade reagent.

2.2.2 Plant material

The roots of *Clerodendrum petasites* were collected at Kaorubchang, Maung, Songkhla, Thailand in May in the year 2007.

2.2.3 Chemical investigation from the roots of *C. petasites*

2.2.3.1 Isolation and extraction

The roots of *Clerodendrum petasites* S. Moore (1.20 kg), cut into small segments, were extracted with MeOH (6 L) for three time over the period of 3, 7 and 30 days at room temperature. After filtration, the filtrate was evaporated to dryness under reduced pressure to give a crude methanol extract as a dark brown gum in 46.43 g.

2.2.3.2 Chemical investigation of the crude methanol extract of the roots of *C. petasites*

The crude methanol extract was primarily tested for its solubility in various solvents at room temperature. The results were demonstrated in **Table 94**.

Table 94 Solubility of the crude extract in various solvents at room temperature

Solvent	Solubility at room temperature
Petroleum ether	-
Dichloromethane	+ (pale yellow solution mixed with dark brown gum)
Ethyl acetate	+ (pale yellow solution mixed with dark brown gum)
Acetone	+ (yellow solution mixed with dark brown gum)
Methanol	++ (dark yellow solution mixed with dark brown gum)
Water	++ (brown yellow solution mixed with dark brown gum)
10% HCl	+++ (brown yellow solution)
10% NaOH	+++ (yellow solution mixed with dark brown gum)
10% $NaHCO_3$	+++ (yellow solution mixed with dark brown gum)

Symbol meaning: + slightly soluble, ++ moderately soluble, +++ well soluble
- insoluble

The crude methanol extract was well soluble in methanol, 10% NaOH, 10% HCl, 10% $NaHCO_3$. The solubility results indicated that major components were moderately polar compounds.

Chromatogram characteristics on normal phase TLC with 5%MeOH/CH_2Cl_2 showed five UV-active spots with the R_f values of 0.24, 0.40, 0.42, 0.73 and 0.85 and four purple spots under ASA reagent with the R_f values of 0.19, 0.26, 0.50 and 0.86. Further purification by Sephadex LH-20 was performed. Elution was conducted with pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford five fractions as shown in **Table 95**.

Table 95 Fractions obtained from the crude methanol extract by column chromatography over Sephadex LH-20

Fraction	Weight (g)	Physical appearance
T1	1.52	Brown gum
T2	30.08	Brown gum
T3	9.33	Dark yellow solid
T4	3.09	Dark yellow gum
T5	2.41	Brown gum

Fraction T1 Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 showed no definite spot under UV-S. Its ^{1}H NMR spectrum showed the absence of aromatic and olefinic protons. Therefore, it was not further investigated.

Fraction T2 Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.36 and 0.55 and six purple spots under ASA reagent with the R_f values of 0.12, 0.26, 0.52, 0.64, 0.73 and 0.92. The ^{1}H NMR spectrum displayed sugar signals. Therefore, it was not further investigated.

Fraction T3 Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 showed three UV-active spots with the R_f values of 0.34, 0.40 and 0.52 and two purple spots under ASA reagent with the R_f values of 0.09 and 0.28. This fraction was then separated into two fractions by dissolving in methanol; the methanol soluble fraction **T3M** and the methanol insoluble fraction **T3N**.

Fraction T3M (5.59 g) Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 showed three UV-active spots with the R_f values of 0.34, 0.40 and 0.52 and two purple spots under ASA reagent with the R_f values of 0.09 and 0.28. It was separated by flash column chromatography over silica gel. Elution was conducted initially with pure dichloromethane, gradually enriched with methanol until pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford five fractions as shown in **Table 96**.

Table 96 Fractions obtained from the fraction **T3M** by flash column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
T3MA	100%CH_2Cl_2-1%MeOH/CH_2Cl_2	64.1	Yellow gum
T3MB	2%MeOH/CH_2Cl_2	122.3	Yellow gum
T3MC	5%MeOH/CH_2Cl_2	164.2	Yellow gum mixed with pale yellow solid
T3MD	10-20%MeOH/CH_2Cl_2	37.2	Brown yellow gum
T3ME	40%MeOH/CH_2Cl_2-100%MeOH	5102.4	Brown gum

Fraction T3MA Chromatogram characteristics on normal phase TLC with 80%CH_2Cl_2/Petrol showed three purple spots under ASA reagent with the R_f values of 0.61, 0.71 and 0.80. The ^{1}H NMR data indicated the presence of long chain hydrocarbons. Therefore, it was not further investigated.

Fraction T3MB Chromatogram characteristics on normal phase TLC with 100%CH_2Cl_2 showed two UV-active spots with the R_f values of 0.40 and 0.47 and three purple spots under ASA reagent with the R_f values of 0.19, 0.51 and 0.68. The ^{1}H NMR data indicated the presence of **SK14** as a major component. Further investigation was then not carried out.

Fraction T3MC (**SK14**) Upon standing at room temperature, the yellow solid (15.1 mg) precipitated**.** Its chromatogram on normal phase TLC with 100%CH_2Cl_2 showed one UV-active spot with the R_f value of 0.40.

Melting point (˚C)		219-222
UVλ_{max}(nm)(MeOH)(log ε)		204 (4.30), 224 (3.31), 238 (2.76), 261 (2.14), 281 (2.21)
FTIR(neat):υ(cm^{-1})		3348 (OH stretching), 1668 (C=O stretching)
^{1}H NMR(Acetone-d_6)(δ_{ppm})(300 MHz) :		13.22 (*s*, 1H), 8.03 (*d*, *J* = 9.0 Hz, 2H), 7.12 (*d*, *J* = 9.0 Hz, 2H), 6.69 (*s*, 1H), 6.64 (*s*, 1H), 3.92 (*s*, 3H), 3.88 (*s*, 3H)
^{13}C NMR(Acetone-d_6)(δ_{ppm})(75 MHz) :		183.60, 164.96, 163.78, 157.74, 154.05, 154.00, 132.25, 129.10, 124.42, 115.44, 105.81, 104.06, 94.80, 60.69, 56.01
DEPT135˚(Acetone-d_6)(δ_{ppm})	CH:	129.10, 115.44, 104.06, 94.80
	CH_3:	60.69, 56.01

The filtrate becomes a yellow gum (148.7 mg) after evaporation to dryness under reduced pressure. Chromatogram characteristics on normal phase TLC with 100%CH_2Cl_2 showed two UV-active spots with the R_f values of 0.40 and 0.47 and two purple spots under ASA reagent with the R_f values of 0.51 and 0.68. The ^{1}H NMR data indicated the presence of **SK14** as a major component. Further investigation was then not carried out.

Fraction T3MD Chromatogram characteristics on normal phase TLC with 100%CH_2Cl_2 showed one UV-active spot with the R_f value of 0.47 and four purple spots under ASA reagent with the R_f values of 0.09, 0.19, 0.51 and 0.68. The ^{1}H NMR data indicated the presence of **SK14** as a major component. Further investigation was then not carried out.

Fraction T3ME Chromatogram characteristics on normal phase TLC with 100%CH_2Cl_2 showed one UV-active spot with the R_f value of 0.02 and three purple

spots under ASA reagent with the R_f values of 0.19, 0.24 and 0.63. The ^1H NMR spectrum showed sugar signals. Therefore, it was not further investigated.

Fraction T3N (3.73 g) Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 showed two purple spots under ASA reagent with the R_f values of 0.09 and 0.28. The ^1H NMR spectrum showed sugar signals. Therefore, it was not further investigated.

Fraction T4 Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 showed three UV-active spots with the R_f values of 0.12, 0.40 and 0.52 and three purple spots under ASA reagent with the R_f values of 0.09, 0.28 and 0.81. It was then separated into two fractions by dissolving in methanol; the methanol soluble fraction **T4M** and the methanol insoluble fraction **T4N**.

Fraction T4M (1.38 g) Chromatogram characteristics on reverse phase TLC with 40%MeOH/H_2O showed three major UV-active spots with the R_f values of 0.20, 0.25 and 0.55. It was further purified by column chromatography over reverse phase C_{18} silica gel. Elution was conducted initially with 40%MeOH/H_2O, gradually enriched with methanol until pure methanol. Fractions with similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford seven fractions as shown in **Table 97**.

Table 97 Fractions obtained from the fraction **T4M** by column chromatography over reverse phase C_{18} silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
T4MA	40%MeOH/H_2O	1205.5	Brown gum
T4MB	50-60%MeOH/H_2O	49.1	Brown yellow gum
T4MC	70%MeOH/H_2O	12.4	Yellow gum
T4MD	80%MeOH/H_2O	8.8	Yellow gum
T4ME	80%MeOH/H_2O	43.2	Yellow gum
T4MF	90%MeOH/H_2O	27.6	Yellow gum
T4MG	100%MeOH	47.6	Yellow gum

Fraction T4MA Chromatogram characteristics on normal phase TLC with 4%MeOH/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.12 and 0.22 and four purple spots under ASA reagent with the R_f values of 0.19, 0.51, 0.55 and 0.63. The ^{1}H NMR spectrum showed sugar signals. Therefore, it was not further investigated.

Fraction T4MB Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.12 and 0.48 and two purple spots under ASA reagent with the R_f values of 0.50 and 0.61. The ^{1}H NMR data indicated the presence of **SK15** as a major component. Further investigation was then not carried out.

Fraction T4MC Chromatogram characteristics on normal phase TLC with 30%EtOAc/Petrol (4 runs) showed two UV-active spots with the R_f values of 0.28 and 0.35. Further purification by precoated TLC with 30%EtOAc/Petrol (8 runs) as a mobile phase afforded two bands.

Band 1 (**SK15**) was obtained as a yellow gum in 4.2 mg. Chromatogram characteristics on normal phase TLC with 30%EtOAc/Petrol (4 runs) showed one UV-active spot with the R_f value of 0.35.

UVλ_{max}(nm)(MeOH)(log ε)		214 (2.18), 273 (1.24), 334 (1.25)
FTIR(neat):υ(cm^{-1})		3348 (OH stretching), 1668 (C=O stretching)
^{1}H NMR (Acetone-d_6)(δ_{ppm})(500 MHz) :		13.22 (*s*, 1H), 7.94 (*d*, *J* = 9.0 Hz, 2H), 7.03 (*d*, *J* = 9.0 Hz, 2H), 6.65 (*s*, 1H), 6.64 (*s*, 1H), 3.88 (*s*, 3H)
^{13}C NMR (Acetone-d_6)(δ_{ppm})(125 MHz) :		182.79, 164.34, 161.25, 156.90, 153.16, 153.05, 131.35, 128.35, 122.24, 116.01, 104.78, 102.60, 93.89, 59.76
DEPT135˚ (Acetone-d_6)(δ_{ppm})	CH:	128.35, 116.01, 102.60, 93.89
	CH_3:	59.76

Band 2 was obtained as a yellow gum in 3.2 mg. Chromatogram characteristics on normal phase TLC with 30%EtOAc/Petrol showed two UV-active spots with the R_f values of 0.28 and 0.35 and two purple spots under ASA reagent with the R_f values of 0.04 and 0.61. The ^{1}H NMR spectrum indicated the presence of many compounds. It was not further investigated.

Fraction T4MD Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 showed one UV-active spot with the R_f value of 0.48. The ^{1}H NMR data indicated the presence of **SK14** as a major component. Further investigation was then not carried out.

Fraction T4ME Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.12 and 0.48 and three purple spots under ASA reagent with the R_f values of 0.28, 0.52 and 0.61. The ^{1}H NMR data indicated the presence of **SK14** as a major component. Further investigation was then not carried out.

Fraction T4MF Chromatogram characteristics on normal phase TLC with 1%MeOH/CH_2Cl_2 showed two UV-active spots with the R_f values of 0.42 and 0.48 and two purple spots under ASA reagent with the R_f values of 0.05 and 0.12. The ^{1}H NMR data indicated the presence of **SK14** as a major component. Further investigation was then not carried out.

Fraction T3MG Chromatogram characteristics on normal phase TLC with 80%CH_2Cl_2/Petrol showed three purple spots under ASA reagent with the R_f values of 0.61, 0.71 and 0.80. The ^{1}H NMR data indicated the presence of long chain hydrocarbons. Therefore, it was not further investigated.

Fraction T4N (1.70 g) Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 showed three purple spots under ASA reagent with the R_f values of 0.09, 0.28 and 0.81. The ^{1}H NMR spectrum showed sugar signals. Therefore, it was not further investigated.

Fraction T5 Chromatogram characteristics on normal phase TLC with 2%MeOH/CH_2Cl_2 showed no definite spot under UV-S. Its ^{1}H NMR spectrum showed the absence of aromatic and olefinic protons. Therefore, it was not further investigated.

CHAPTER 2.3

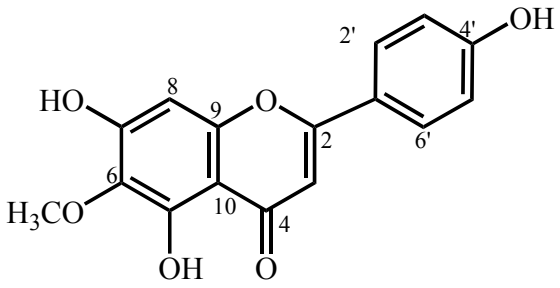
RESULTS AND DISCUSSION

The crude methanol extract from the roots of *C. petasites* was separated by chromatographic methods to yield two flavoniods (**SK14** and **SK15**). The structures were elucidated by analysis of 1D and 2D NMR spectroscopic data and/or comparison of the spectroscopic data, especially ^{1}H and ^{13}C NMR data with those previously reported in the literatures. In addition, the ^{13}C NMR signals were assigned from DEPT, HMQC and HMBC spectra.

2.3.1 Compound SK15

Compound **SK15** was isolated as a yellow gum. It exhibited UV absorption bands of a flavone chromophore at 214, 273 and 334 nm while the hydroxyl and conjugated carbonyl absorption bands were found at 3348 and 1668 cm^{-1}, respectively, in the IR spectrum. The ^{1}H NMR spectrum (**Figure 50**) contained signals of chelated hydroxy proton (δ_H 13.22, *s*, 1H), *para*-disubstituted aromatic protons [δ_H 7.94 and 7.03 (*d, J* = 9.0 Hz, 2H each)], *singlet* aromatic proton (δ_H 6.64, 1H), *singlet* olefinic proton (δ_H 6.65, 1H) and one methoxyl group (δ_H 3.88, 3H). The ^{13}C NMR (**Table 98**) (**Figure 51**) and HMQC data indicated that compound **SK15** consisted of sixteen carbons: nine quarternary, six methine and one methoxy carbons. The location of all substituents was established by HMBC data as follows. The chelated hydroxy proton, δ_H 13.22, which was located at the *peri*-position to the flavone carbonyl group, showed HMBC correlations with C-5 (δ_C 153.16), C-6 (δ_C 131.35) and C-10 (δ_C 104.78). The *singlet* aromatic proton was then attributed to H-8 based on the HMBC correlations of C-6 and C-10. The *singlet* olefinic proton was assigned as H-3 of the flavone moiety and gave cross peaks with C-2 (δ_C 164.34), C-4, C-10 and C-1′(δ_C 122.24). HMBC correlations

between the aromatic protons [H-2′ and H-6′ (δ_H 7.94)] of the *para*-disubstituted benzene and C-2 established the attachment of the *para*-disubstituted benzene at C-2. In addition, the methoxyl group, δ_H 3.88, was located at C-6 on the basis of a HMBC correlation of 6-OCH_3/C-6 (δ_C 131.35). Thus, **SK15** was determined as 6-methoxyscutellarin which was previously isolated from the roots of *C. indicum* (Rahman, 2000).



(SK15)

Table 98 The NMR data of compound **SK15** and 6-methoxyscutellarin

Position	**SK15** (Acetone-d_6)		HMBC	6-methoxyscutellarin ($CDCl_3$)*	
	δ_H (*mult* , *J* Hz)	δ_C (C-Type)		δ_H (*mult* , *J* Hz)	δ_C
2	-	164.34 (C)	-	-	164.8
3	6.65 (*s*, 1H)	102.60 (CH)	C-2, C-4, C-10, C-1′	6.62 (*s*, 1H)	103.1
4	-	182.79 (C=O)	-	-	183.1
OH-5	13.22 (*s*, 1H)	153.16 (C)	C-5, C-6, C-10	13.22 (*s*, 1H)	153.5
6	-	131.35 (C)	-	-	131.8
7	-	153.05 (C)	-	-	154.2
8	6.64 (*s*, 1H)	93.89 (CH)	C-6, C-9, C-10	6.61 (*s*, 1H)	94.4
9	-	156.90 (C)	-		157.8
10	-	104.78 (C)	-	-	105.3
1′	-	122.24 (C)	-	-	122.7

Table 100 (continued)

Position	**SK15** (Acetone-d_6)		HMBC	6-methoxyscutellarin ($CDCl_3$)*	
	δ_H (*mult* , *J* Hz)	δ_C (C-Type)		δ_H (*mult* , *J* Hz)	δ_C
2′, 6′	7.94 (*d*, 9.0, 2H)	128.35 (CH)	C-2, C-4′	7.92 (*d*, 8.9, 2H)	128.9
3′, 5′	7.03 (*d*, 9.0, 2H)	116.01 (CH)	C-1′, C-4′	7.00 (*d*, 8.9, 2H)	116.4
4’	-	161.25 (C)	-	-	161.7
6-OCH_3	3.88 (*s*, 3H)	59.76 (CH_3)	C-6	3.84 (*s*, 3H)	60.3

*(Lou, *et al*., 2002).

2.3.2 Compound SK14

Compound **SK14** was obtained as a pale yellow solid and decomposed at 219-222 °C. The UV and IR absorption bands were similar to those of **SK15**. The ^{1}H NMR spectrum contained signals of a chelated hydroxy proton (δ_H 13.22, *s*, 1H), *para*-disubstituted aromatic protons, [δ_H 8.03 and 7.12 (*d, J* = 9.0 Hz), 2H each)], *singlet* aromatic proton (δ_H 6.64, 1H), *singlet* olefinic proton (δ_H 6.69, 1H) and two methoxy groups [δ_H 3.88 and 3.92, 3H each)]. The ^{1}H NMR data were similar to those of **SK15** except for an additional signal of the methoxyl group (δ_H 3.92) in **SK14**. The methoxyl group was located at C-4′ (δ_C 163.78) on the basis of a HMBC correlation between the methoxy protons with C-4′ (**Table 99**). The remaining HMBC correlations were similar to those found in **SK15**. Thus, **SK14** was determined as 6,4′-dimethoxyscutellarin which was previously isolated from the roots of *C. indicum* (Rahman, 2000).

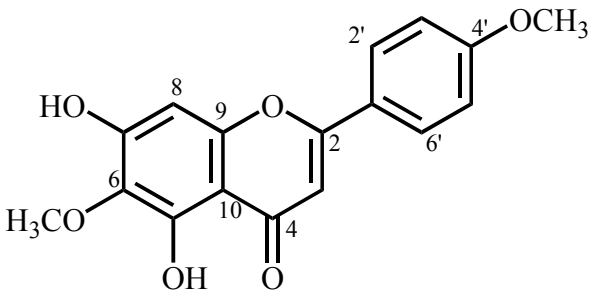


Table 99 The NMR data of compound **SK14** and 6,4′-dimethoxyscutellarin

Position	**SK14** (Acetone-d_6)		HMBC	6,4′-dimethoxyscutellarin ($CDCl_3$)*	
	δ_H (*mult* , *J* Hz)	δ_C (C-Type)		δ_H (*mult* , *J* Hz)	δ_C
2	-	164.96 (C)	-	-	164.0
3	6.69 (*s*, 1H)	104.06 (CH)	C-2, C-4, C-10, C-1′	6.66 (*s*, 1H)	103.1
4	-	183.60 (C=O)	-	-	182.7
OH-5	13.22 (*s*, 1H)	154.05 (C)	C-5, C-6, C-10	13.22 (*s*, 1H)	153.0
6	-	132.25 (C)	-	-	131.8
7	-	154.00 (C)	-	-	153.7
8	6.64 (*s*, 1H)	94.80 (CH)	C-6, C-9, C-10	6.62 (*s*, 1H)	94.4
9	-	157.74 (C)	-	-	157.8
10	-	105.81 (C)	-	-	104.5
1′	-	124.42 (C)	-	-	123.2
2′, 6′	8.03 (*d*, 9.0, 2H)	129.10 (CH)	C-2, C-4′	7.99 (*d*, 8.8, 2H)	128.4
3′, 5′	7.12 (*d*, 9.0, 2H)	115.44 (CH)	C-1′, C-4′	7.10 (*d*, 8.8, 2H)	114.7
4′	-	163.78 (C)	-	-	162.8
6-OCH_3	3.88 (*s*, 3H)	60.69 (CH_3)	C-6	3.84 (*s*, 3H)	60.3
4′-OCH_3	3.92 (*s*, 3H)	56.01 (CH_3)	C-4′	3.89 (*s*, 3H)	55.4

*(Lou, *et al*., 2002).

REFERENCES

วุฒิ วุฒิธรรมเวช, 2540. สารานุกรมสมุนไพร รวมหลักเภสัชกรรมไทย โอ.เอส. พริ้นติ้งเฮ้า, กรุงเทพฯ

Akao, Y., Nakagawa, Y., Iinuma, M. and Nozawa, Y. 2008. Anti-cancer effects of xanthones from pericarps of mangosteen. Int. J. Mol. Sci. 9 (3), 355-370.

Balunas, M. J., Su, B., Brueggemeier, R. W. and Kinghorn, A. D. 2008. Xanthones from the Botanical Dietary Supplement Mangosteen (*Garcinia mangostana*) with Aromatase Inhibitory Activity. J. Nat. Prod. 71 (7), 1161-1166.

Boonsri, S. 2004. Chemical constituents of *Clerodendrum serratum* and *Mesua kunstleri*. Master of Science Thesis, Prince of Songkla University, Songkhla, Thailand.

Castardo, J. C., Prudente, A. S., Ferreira, J., Guimaraes, C. L., Delle Monache, F., Cechinel Filho, V., Otuki, M. F. and Cabrini, D. A. 2008. Anti-inflammatory effects of hydroalcoholic extract and two biflavonoids from *Garcinia gardneriana* leaves in mouse paw oedema. J. Ethnopharmacol. 118 (3), 405-411.

Chae, S., Kang, K. A., Kim, J. S., Kim, H. K., Lee, E. J., Hyun, J. W. and Kang, S. S. 2007. Antioxidant activities of acetylmartynosides from *Clerodendrum trichotomum*. J. Appl. Biol. Chem. 50 (4), 270-274.

Chae, S., Kang, K. A., Kim, J. S., Hyun, J. W. and Kang, S. S. 2006. Trichotomoside: a new antioxidative phenylpropanoid glycoside from *Clerodendron trichotomum*. Chem. Biodivers. 3 (1), 41-48.

Chae, S., Kim, J. S., Kang, K. A., Bu, H. D., Lee, Y., Seo, Y. R., Hyun, J. W. and Kang, S. S. 2005. Antioxidant activity of isoacteoside from *Clerodendron trichotomum.* J. Toxicol. Environ. Health Part A 68 (5), 389-400.

Chae, S., Kim, J. S., Kang, K. A., Bu, H. D., Lee, Y., Hyun, J. W. and Kang, S. S. 2004. Antioxidant activity of jionoside D from *Clerodendron trichotomum.* Biol. Pharm. Bull. 27 (10), 1504-1508.

Chen, L.-G., Yang, L.-L. and Wang, C.-C. 2008. Anti-inflammatory activity of mangostins from *Garcinia mangostana*. Food Chem. Toxicol. 46 (2), 688-693.

Chen, J.-J., Peng, C.-F., Huang, H.-Y. and Chen, I.-S. 2006. Benzopyrans, biphenyls and xanthones from the root of *Garcinia linii* and their activity against Mycobacterium tuberculosis. Planta Med. 72 (5), 473-477.

Chen, J.-C. and Zhu, Q.-X. 2001. Two new terpenoid glucosides from *Clerodendrum serratum*. Pharmazie 56 (3), 270-271.

Cheng, H.-H., Wang, H.-K., Ito, J., Bastow, K. F., Tachibana, Y., Nakanishi, Y., Xu, Z., Luo, T.-Y. and Lee, K.-H. 2001. Cytotoxic pheophorbide-related compounds from *Clerodendrum calamitosum* and *Clerodendrum cyrtophyllum.* J. Nat. Prod. 64 (7), 915-919.

Chien, S.-C., Chyu, C.-F., Chang, I.-S., Chiu, H.-L. and Kuo, Y.-H. 2008. A novel polyprenylated phloroglucinol, garcinialone, from the roots of *Garcinia multiflora*. Tetrahedron Lett. 49 (36), 5276-5278.

Chin, Y.-W., Chai, H.-B., Keller, W. J. and Kinghorn, A. D. 2008a. Lignans and Other Constituents of the Fruits of *Euterpe oleracea* (Acai) with Antioxidant and Cytoprotective Activities. J. Agric. Food. Chem. 56 (17), 7759-7764.

Chin, Y.-W., Jung, H.-A., Chai, H., Keller, W. J. and Kinghorn, A. D. 2008b. Xanthones with quinone reductase-inducing activity from the fruits of *Garcinia mangostana* (Mangosteen). Phytochemistry 69 (3), 754-758.

Cui, C.-B., Tezuka, Y., Kikuchi, T., Nakano, H., Tamaoki, T. and Park, J.-H. 1990. Constituents of Fern, *Davallia mariesii* Moore. I. Isolation and structures of Davallialactone and a New Flavonone Glucuronide. Chem. Pharm. Bull., 38 (12), 3218-3225.

Deachathai, S., Mahabusarakam, W., Phongpaichit, S., Taylor, W. C., Zhang, Y.-J. and Yang, C.-R. 2006. Phenolic compounds from the flowers of *Garcinia dulcis*. Phytochemistry 67 (5), 464-469.

Ee, G. C. L., Daud, S., Taufiq-Yap, Y. H., Ismail, N. H. and Rahmani, M. 2006. Xanthones from *Garcinia mangostana* (Guttiferae). Nat. Prod. Res., Part A: Structure and Synthesis 20 (12), 1067-1073.

Elya, B., He, H. P., Kosela, S., Hanafi, M. and Hao, X. J. 2008. A new cytotoxic xanthone from *Garcinia rigida*. Fitoterapia 79 (3), 182-184.

Elya, B., He, H. P., Kosela, S., Hanafi, M., Hao and X. J. 2006a. Two new xanthons from *Garcinia rigida* leaves. Nat. Prod. Res., Part A. Structure and Synthesis 20 (9), 788-791.

Elya, B., He, H. P., Kosela, S., Hanafi, M. and Hao, X. J. 2006b. A new benzophenone from the stem bark of *Garcinia benthami*. Nat. Prod. Res., Part A. Structure and Synthesis 20 (12), 1059-1062.

Feng, F., Liu, W.-Y., Chen, Y.-S., Guo, Q.-L. and You, Q.-D. 2007. Five novel prenylated xanthones from *Resina Garcinia*. J. Asian Nat. Prod. Res. 9 (8), 735-741.

Fotie, J., Bohle, D. S., Olivier, M., Gomez, M. A. and Nzimiro, S. 2007. Trypanocidal and Antileishmanial Dihydrochelerythrine Derivatives from *Garcinia lucida*. J. Nat. Prod. 70 (10), 1650-1653.

Gao, L., Wei, X. and He, Y. 2003a. Studies on chemical constituents in of *Clerodendrum bungei*. Zhongguo Zhongyao Zazhi 28 (11), 1042-1044.

Gao, L., Wei, X. and He, Y. 2003b. Studies on chemical constituents in leaves of baihuamudan *Clerodendron fragrans*. Zhongguo Zhongyao Zazhi 28 (10), 948-951.

Hamed, W., Brajeul, S., Mahuteau-Betzer, F., Thoison, O., Mons, S., Delpech, B., Nguyen, V. H., Sevenet, T. and Marazano, C. 2006. Oblongifolins A-D, Polyprenylated benzoylphloroglucinol derivatives from *Garcinia oblongifolia*. J. Nat. Prod. 69 (5), 774-777.

Han, Q.-B., Yang, N.-Y., Tian, H.-L., Qiao, C.-F., Song, J.-Z., Chang, D. C., Chen, S.-L., Luo, K. Q. and Xu, H.-X. 2008. Xanthones with growth inhibition against HeLa cells from *Garcinia xipshuanbannaensis*. Phytochemistry 69 (11), 2187-2192.

Han, Q.-B., Qiao, C.-F., Song, J.-Z., Yang, N.-Y., Cao, X.-W.; Peng, Y., Yang, D.-J., Chen, S.-L. and Xu, H.-X. 2007. Cytotoxic prenylated phenolic compounds from the twig bark of *Garcinia xanthochymus*. Chem. Biodivers. 4 (5), 940-946.

Han, Q.-B., Wang, Y.-L., Yang, L., Tso, T.-F., Qiao, C.-F., Song, J.-Z., Xu, L.-J., Chen, S.-L., Yang, D.-J. and Xu, H.-X. 2006a. Cytotoxic polyprenylated xanthones from the resin of *Garcinia hanburyi*. Chem. Pharm. Bull. 54 (2), 265-267.

Han, Q.-B., Yang, L., Wang, Y.-L., Qiao, C.-F., Song, J.-Z., Sun, H.-D. and Xu, H.-X. 2006b. A pair of novel cytotoxic polyprenylated xanthone epimers from gamboges. Chem. Biodivers. 3 (1), 101-105.

Hartati, S., Soemiati, A., Wang, H.-B., Kardono, L. B. S., Hanafi, M., Kosela, S. and Qin, G.-W. 2008a. A novel polyisoprenyl benzophenone derivative from *Garcinia eugeniaefolia*. J. Asian Nat. Prod. Res. 10 (5-6), 509-513.

Hartati, S., Kadono, L. B. S., Kosela, S. and Harrison, L. J. 2008b. A new pyrano xanthone from the stem barks of *Garcinia tetrandra* Pierre. Pak. J. Bio. Sci. 8 (1), 137-142.

Hartati, S., Wang, H.-B., Kardono, L. B. S., Kosela, S. and Qin, G.-W. 2007. Chemical constituents of *Garcinia maingayii*. Zhongguo Tianran Yaowu 5 (4), 272-276.

Hay, A.-E., Merza, J., Landreau, A., Litaudon, M., Pagniez, F., Le Pape, P. and Richomme, P. 2008. Antileishmanial polyphenols from *Garcinia vieillardii*. Fitoterapia 79 (1), 42-46.

Hazekamp, A., Verpoorte, R. and Panthong, A. 2001. Isolation of a bronchodilator flavonoid from the Thai medicinal plant *Clerodendrum petasites*. J. Ethnopharmacol. 78 (1), 45-49.

Hosny, M. 2003. Cytotoxic neoclerodane diterpenoids from *Clerodendrum splendens*. Egyp. J. Biomed. Sci. 11, 285-296.

Hsieh, T.J., Su, C.C., Chen, C.Y., Liou, C.H. and Lu, L.H. 2005. Using experimental studies and theoretical calculations to analyze the molecular mechanism of coumarin, *p*-hydroxybenzoic acid and cinnamic acid. J. Mol. Struc. 741, 193-199.

Hu, J., Chen, J., Zhao, Y., Wang, R., Zheng, Y. and Zhou, J. 2006. Chemical constituents from fruit hulls of *Garcinia mangostana* (Cuttiferae). Yunnan Zhiwu Yanjiu 28 (3), 319-322.

Huang, M.-T., Liu, Y., Badmaev, V. and Ho, C.-T. 2008. Antiinflammatory and anticancer activities of garcinol. ACS Sym. Ser. 987, 293-303.

Hutadilok-Towatana, N., Kongkachuay, S. and Mahabusarakam, W. 2007. Inhibition of human lipoprotein oxidation by morelloflavone and camboginol from *Garcinia dulcis*. Nat. Prod. Res., Part B. 21 (7), 655-662.

Hwang, W. G. and Cho, H. G. 2007. Method for extracting acteoside from *Clerodendron trichotomum* leaf and antioxidant and anti-inflammatory agent containing acteoside. Repub. Korean Kongkae Taeho Kongbo 36pp. August, 1.

Iinuma, M., Ito, T., Tosa, H., Tanaka, T. and Riswan, S. 1996. Five New Xanthones from *Garcinia dulcis*. J. Nat. Prod. 59 (5), 472-475.

Ishiguro, K., Nakajima, M., Fukumoto, H. and Isoi, K. 1995. Co-occurrence of prenylated xanthones and their cyclization products in cell suspension cultures of *Hypericium patulum*. Phytochemistry 38, 867-869.

Ishiguro, K., Nakajima, M., Fukumoto, H. and Isoi, K. 1993. Xanthone in cell suspansion culture of *Hypatum patulum*. Phytochemistry 33, 839-840.

Jabit, M. L., Khalid, R., Abas, F., Shaari, K., Hui, L. S., Stanslas, J. and Lajis, N. H. 2007. Cytotoxic xanthones from *Garcinia penangiana* Pierre. J. Biosci. 62 (11/12), 786-792.

Jia, L. and Min, Z. 2007. Chemical constituents from *Clerodendrum canescens*. Zhongcaoyao 38 (2), 161-163.

Jung, H.-A., Su, B.-N., Keller, W. J., Mehta, R. G. and Kinghorn, A. D. 2006. Antioxidant Xanthones from the Pericarp of *Garcinia mangostana* (Mangosteen). J. Agric. Food. Chem. 54 (6), 2077-2082.

Kamdem, W., Alain, F., Mulholland, D., Wansi, J. D., Mbaze, Luc. M., Powo, R., Mpondo, T. N., Fomum, Z. T., Konig, W. and Nkengfack, A. E. 2006. Afzeliixanthones A and B, 2 new prenylated xanthones from *Garcinia afzelii* ENGL. (Guttiferae). Chem. Pharm. Bull. 54 (4), 448-451.

Kanchanapoom, T., Chumsri, P., Kasai, R., Otsuka, H. and Yamasaki, K. 2005. A new iridoid diglycoside from *Clerodendrum chinense*. J. Asian Nat. Prod. Res. 7 (3), 269-272.

Kanchanapoom, T., Kasai, R., Chumsri, P., Hiraga, Y. and Yamasaki, K. 2001. Megastigmane and iridoid glucosides from *Clerodendrum inerme*. Phytochemistry 58 (2), 333-336.

Kang, D. G., Lee, Y. S., Kim, H. J., Lee, Y. M. and Lee, H. S. 2003. Angiotensin converting enzyme inhibitory phenylpropanoid glycosides from *Clerodendron trichotomum*. J. Ethnopharmacol. 89 (1), 151-154.

Kardono, L. B. S., Hanafi, M., Sherley, G., Kosela, S. and Harrison, L. J. 2006. Bioactive constituents of *Garcinia porrecta* and *G. parvifolia* grown in Indonesia. Pak. J. Biol. Sci. 9 (3), 483-486.

Kebenei, J. S., Ndalut, P. K. and Kiprono, C. P. 2004. Larvicidal activity of myricoidine from *Clerodendrum myricoides*. Bull. Chem. Soc. Ethiop. 18 (2), 225-227.

Kijjoa, A., Gonzalez, M. J., Pinto, M. M., Nascimento, M. S. J., Campos, N., Mondranondra, I-O. Silva, A. M. S., Eaton, G. and Herz, W. 2008.

Cytotoxicity of prenylated xanthones and other constituents from the wood of *Garcinia merguensis*. Planta Med. 74 (8), 864-866.

Kim, H. J., Woo, E.-R., Shin, C.-G., Hwang, D. J., Park, H. and Lee, Y. S. 2001. HIV-1 integrase inhibitory phenylpropanoid glycosides from *Clerodendron trichotomum*. Arch. Pharmacal Res. 24 (6), 618.

Komguem, J., Lannang, A. M., Tangmouo, J. G., Louh, G. N., Ngounou, F. N., Lontsi, D., Choudhary, M. I. and Sondengam, B. L. 2006. Polyanxanthone, a xanthone from the stem bark of *Garcinia polyantha*. Nat. Prod. Commun. 1 (5), 363-365.

Kuete, V., Komguem, J., Beng, V. P., Meli, A. L., Tangmouo, J. G., Etoa, F.-X. and Lontsi, D. 2007. Antimicrobial components of the methanolic extract from the stem bark of *Garcinia smeathmannii* Oliver (Clusiaceae). S. Afr. J. Bot. 73 (3), 347-354.

Kumar, S. and Chattopadhyay, S. K. 2007a. High-performance liquid chromatography and LC-ESI-MS method for the identification and quantification of two biologically active polyisoprenylated benzophenones xanthochymol and isoxanthochymol in different parts of *Garcinia indica*. Biomed. Chromatogr. 21 (2), 139-163.

Kumar, S., Chattopadhyay, S. K., Darokar, M. P., Garg, A. and Khanuja, S. P. S. 2007b. Cytotoxic activities of xanthochymol and isoxanthochymol substantiated by LC-MS/MS. Planta Med. 73 (14), 1452-1456.

Lannang, A. M., Komguem, J., Ngninzeko, N. N., Tangmouo, J. G., Lontsi, D., Ajaz, A., Choudhary, M. I., Sondengam, B. L. and Atta-ur-Rahman. 2006a. Antioxidant benzophenones and xanthones from the root bark of *Garcinia smeathmannii*. Bull. Chem. Soc. Ethiop. 20(2), 247-252.

Le, C. N., Nguyen, V. L., Nguyen, D. H. and Le, M. U. 2006. Chemical constituents and antioxidant activity of flavonoids from *Clerodendron cyrtophyllum* Turcz. Tap Chi Duoc Hoc. 46 (11), 30-33.

Li, Y., Li, J., Li, P. and Tu, P. 2005. Isolation and characterization of phenylethanoid glycosides from *Clerodendron bungei*. Yaoxue Xuebao 40 (8), 722-727.

Lin, C.-N., Wang, J.-P. and Weng, J.-R. 2006. Anti-inflammatory and cure for ageing and Alzheimer's disease based on phloroglucinol derivatives. U.S. Pat. Appl. Publ., 11pp. Setember, 14.

Lobo, R., Punitha, I. S. R., Rajendran, K., Shirwaikar, A. and Shirwaikar, A. 2006. Preliminary study on the antisnake venom activity of alcoholic root extract of *Clerodendrum viscosum* (Vent.) in naja naja venom. Nat. Prod. Sci. 12 (3), 153-156.

Lou, H.-X., Li, G.-Y. and Wang, F.-Q. 2002. A Cytotoxic Diterpenoid and Antifungul Phenolic Compounds from *Frullania Muscicola* Steph. J. Asian Nat. Prod. Res. 4 (2), 87-94.

Louh, G. N., Lannang, A. M., Mbazoa, C. D., Tangmouo, J. G., Komguem, J., Castilho, P., Ngninzeko, F. N., Qamar, N., Lontsi, D., Choudhary, M. I. and Sondengam, B. L. 2008. Polyanxanthone A, B and C, three xanthones from the wood trunk of *Garcinia polyantha* Oliv. Phytochemistry 69 (4), 1013-1017.

Lu, Y.-H., Wei, B.-L., Ko, H.-H. and Lin, C.-N. 2008. DNA strand-scission by phloroglucinols and lignans from heartwood of *Garcinia subelliptica* Merr. and Justicia plants. Phytochemistry 69 (1), 225-233.

Mahapatra, S., Mallik, S. B., Rao, G. V., Reddy, G. C. and Guru Row, T. N. 2007. Garcinia lactone. Acta Crystallogr., Sect. E: Struct. Rep. Online E63 (9), o3869.

Martins, F. T., Camps, I., Doriguetto, A. C., dos Santos, M. H., Ellena, J. and Barbosa, L. C. A. 2008. Crystal structure of garciniaphenone and evidences on the relationship between keto-enol tautomerism and configuration. Helv. Chim. Acta. 91 (7), 1313-1325.

Martins, F. T., Cruz, J. W. Jr., Derogis, P. B. M. C., dos Santos, M. H., Veloso, M. P.; Ellena, J. and Doriguetto, A. C. 2007. Natural polyprenylated benzophenones: keto-enol tautomerism and stereochemistry. J. Braz. Chem. Soc. 18 (8), 1515-1523.

Masullo, M., Bassarello, C., Suzuki, H., Pizza, C. and Piacente, S. 2008. Polyisoprenylated benzophenones and an unusual polyisoprenylated tetracyclic xanthone from the fruits of *Garcinia cambogia*. J. Agric. Food Chem. 56 (13), 5205-5210.

Mbwambo, Z. H., Kapingu, M. C., Moshi, M. J., Machumi, F., Apers, S., Cos, P., Ferreira, D., Marais, J. P. J., Vanden Berghe, D., Maes, L., Vlietinck, A. and Pieters, Luc. 2006. Antiparasitic activity of some xanthones and biflavonoids from the root bark of *Garcinia livingstonei*. J. Nat. Prod. 69 (3), 369-372.

Merza, J., Mallet, S., Litaudon, M., Dumontet, V., Seraphin, D. and Richomme, P. 2006. New cytotoxic guttiferone analogues from *Garcinia virgata* from New Caledonia. Planta Med. 72 (1), 87-89.

Miller, R. E., McConville, M, J. and Woodrow, L. E. 2006. Cyanogenic glycosides from the rare Australian endemic rainforest tree *Clerodendrum grayi* (Lamiaceae). Phytochemistry 67 (1), 43-51.

Miyazawa, M., Oshima, T. and Koshio, K. 2003. Tyrosinase Inhibitor from Black Rice Bran. J. Agric. Food Chem. 51 6553-6956.

Mohd Khalid, R., Jabit, Md. L., Abas, F., Stanslas, J., Shaari, K. and Lajis, N. H. 2007. Cytotoxic xanthones from the leaves of *Garcinia urophylla*. Nat. Prod. Commun. 2 (3), 271-276.

Mondal, M., Puranik, V. G. and Argade, N. P. 2006. Facile synthesis of 1,3,7-trihydroxyhanthone and its regioselective coupling reaction with prenal: simple and efficient access to Osajaxanthone and Nigrolinexanthone F. J. Org. Chem. 71, 4992-4995.

Naklue, W. 2006. Chemical Constituents from the Twigs of *Garcinia parvifolia*, Master of Science Thesis, Prince of Songkla University, Songkhla, Thailand.

Nan, H., Wu, J., Yin, H. and Zhang, C. 2006. Terpene compounds in *Clerodendrum inerme* (L.) Gaertn. Zhongcaoyao 37 (4), 508-509.

Nan, H., Zhang, S. and Wu, J. 2005a. Chemical constituents from *Clerodendrum inerme*. Zhongcaoyao 36 (4), 492-494.

Nan, H., Wu, J. and Zhang, S. 2005b. A new phenylethanoid glycoside from *Clerodendrum inerme*. Pharmazie 60 (10), 798-799.

Neves, J. S., Coelho, L. P., Cordeiro, R. S. B., Veloso, M. P., Rodrigues e Silva, P. M., dos Santos, M. H. and Martins, M. A. 2007. Antianaphylactic properties of 7-epiclusianone, a tetraprenylated benzophenone isolated from *Garcinia brasiliensis*. Planta Med. 73 (7), 644-649.

Ngoupayo, J., Noungoue, D. T., Lenta, B. N., Tabopda, T. K., Khan, S. N., Ngouela, S., Shaiq, M. A. and Tsamo, E. 2007. Brevipsidone, a new depsidone and

other α-glucosidase inhibitors from *Garcinia brevipedicellata* (Clusiaceae). Nat. Prod. Commun. 2 (11), 1141-1144.

Noro, T., Ueno, A., Mizotani, M., Hashimoto, T., Miyase, T., Kuroyangi, M. and Fukushima, S. 1984. Inhibitor of Xanthine Oxidase from *Athyrium mesosorum*. Chem. Pharm. Bull. 32, 4455-4459.

Nyegue, M. A., Belinga-Ndoye, C. F., Zollo, P.-H. A., Agnaniet, H., Menut, C. and Bessiere, J. M. 2005. Aromatic plants of tropical central Africa. Part L. volatile components of *Clerodendrum buchholzii* Gurke from Cameroon. Flavour Frag J. 20 (3), 321-323.

Nyegue, M., Kwanga, S. N., Ndoye, F., Zollo, P.-H. A., Etoa, F.-X., Agnaniet, H. and Menut, C. 2007. Chemical composition, antiradical and antifungal activities of essential oil of fresh leaves of *Clerodendrum buchholzii* (Gurke) from Cameroon. J. Essent. Oil-Bear. Plants. 10 (6), 510-518.

Okwu, D. E. and Morah, F. N. I. 2007. Isolation and characterization of flavanone glycoside 4',5,7-trihydroxy flavanone rhamnoglucose from *Garcinia kola* seeds. J. Appl. Sci. 7 (2), 306-309.

Ollis, W. D., Redmen, B. T., Sutherland, I. O. and Jewers, K., 1969. Constituent of bronianone. J. Chem. Soc. (D). 15, 879-880.

Pal, D. K., Sannigrahi, S. and Dutta, A. 2007. Anthelmintic activity of leaves of *Clerodendrum infortunatum*. Indian J. Nat. Prod., 23 (2), 22-25.

Pandey, R., Kaur, R., Malasoni, R. and Gupta, M. M. 2008. Lupeol ester from *Clerodendrum phlomidis*. Indian J. Chem., Sect B., 47B (3), 470-472.

Pandey, R., Verma, R. K. and Gupta, M. M. 2007. High-performance thin-layer chromatographic method for quantitative determination of 4α-methyl-24β-

ethyl-5α-cholesta-14,25-dien-3β-ol, 24β-ethylcholesta-5,9(11),22*E*-trien-3β-ol and betulinic acid in *Clerodendrum inerme*. J. Sep. Sci. 30 (13), 2086-2091.

Pandey, R., Verma, R. K. and Gupta, M. M. 2006. Pentadecanoic acid β-D-glucoside from *Clerodendrum inerme*. Indian J. Chem., Sect B. 45B (9), 2161-2163.

Pandey, R., Verma, R. K. and Gupta, Madan M. 2005. Neo-clerodane diterpenoids from *Clerodendrum inerme*. Phytochemistry 66 (6), 643-648.

Pandey, R., Verma, R. K., Singh, S. C. and Gupta, M. M. 2003. 4α-Methyl-24β-ethyl-5α-cholesta-14,25-dien-3β-ol and 24β-ethylcholesta-5,9(11), 22*E*-trien-3β-ol, sterols from *Clerodendrum inerme*. Phytochemistry 63 (4), 415-420.

Panthong, K., Pongcharoen, W., Phongpaichit, S. and Taylor, W. C. 2006. Tetraoxygenated xanthones from the fruits of *Garcinia cowa*. Phytochemistry 67 (10), 999-1004.

Park, M.-A. and Kim, H.-J. 2007. Anti-inflammatory constituents isolated from *Clerodendron trichotomum* tunberg leaves (CTL) inhibits pro-inflammatory gene expression in LPS-stimulated RAW 264.7 macrophages by suppressing NF-κB activation. Arch. Pharmacal Res. 30 (6), 755-760.

Rahman, M. A. A., Zafrul Azam, A. T. M. and Gafur, M. A. 2000. *In vivo* Antibacterial Principle of Extract and Two Flavonoids from *Clerodendrum indicum Linn*. Pak. J. Biol. Sci., 3 (10), 1769-1771.

Rao, D. R., Gurudutt, K. N., Mamatha, S. and Rao, L. J. M. 2007. Guttiferic acid, a novel rearrangement product from minor chromenoxanthone pigments of *Garcinia morella* Desr. Magn. Reson. Chem. 45 (7), 578-582.

Ravindranath, N., Ramesh, C., Hara Kishore, K., Murty, U. S. N. and Das, B. 2003. Clerodendrone, a novel hydroquinone diterpenoid from *Clerodendrum indicum*. J. Chem. Res., Synop. 7, 440-441.

Reutrakul, V., Anantachoke, N., Pohmakotra, M., Jaipetch, T., Sophasan, S., Yoosook, C., Kasisit, J., Napaswat, C., Santisuk, T. and Tuchinda, P. 2007. Cytotoxic and anti-HIV-1 caged xanthones from the resin and fruits of *Garcinia hanburyi*. Planta Med. 73 (1), 33-40.

Roy, R., Pandey, V. B., Singh, U. P. and Prithiviraj, B. 1996. Antifungal activity of the flavonoids from *Clerodendron infortunatum* roots. Fitoterapia 67 (5), 473-474.

Roy, R., Singh, U. P. and Pandey, V. B. 1995. Antifungal activity of some naturally occurring flavonoids. Orient. J. Chem. 11 (2), 145-148.

Rukachaisirikul, V., Trisuwan, K., Sukpondma, Y. and Phongpaichit, S. 2008. A new benzoquinone derivative from the leaves of *Garcinia parvifolia*. Arch. Pharmacal Res. 31 (1), 17-20.

Rukachaisirikul, V., Naklue, W., Phongpaichit, S., Towatana, N. H. and Maneenoon, K. 2006. Phloroglucinols, depsidones and xanthones from the twigs of *Garcinia parvifolia*. Tetrahedron 62 (36), 8578-8585.

Rukachaisirikul, V., Saelim, S., Karnsomchoke, P. and Phongpaichit, S. 2005. Friedolanostanes and Lanostanes from the leaves of *Garcinia hombroniana*. J. Nat. Prod. 68, 1222-1225.

Rukachaisirikul, V., Adir, A., Dampawan, P., Taylor, W.C. and Turner, T.C. 2000. Lanostanes and friedolanostanes from the pericarp of *Garcinia hombroniana*. Phytochemistry 55, 183-188.

Salae, S. 2006. Chemical Constituents from the twigs, seeds and fruits of *Garcinia nervosa*, Master of Science Thesis, Prince of Songkla University, Songkhla, Thailand.

Saelim, S. 2005. Chemical Constituents from the leaves of *Garcinia hombroniana*, Master of Science Thesis, Prince of Songkla University, Songkhla, Thailand.

Shadid, K. A., Shaari, K., Abas, F., Israf, D. A., Hamzah, A. S., Syakroni, N., Saha, K. and Lajis, N. H. 2007. Cytotoxic caged-polyprenylated xanthonoids and a xanthone from *Garcinia cantleyana*. Phytochemistry 68 (20), 2537-2544.

Shehata, A. H., Yousif, M. F. and Soliman, G. A. 2001. Phytochemical and pharmacological investigations of *Clerodendron splendens* Don growing in Egypt. Egyp. J. Biomed. Sci. 7, 145-163.

Shen, J. and Yang, J.-S. 2007a. A novel benzophenone from *Garcinia cowa*. Huaxue Xuebao 65 (16), 1675-1678.

Shen, J., Tian, Z. and Yang, J.-S. 2007b. The constituents from the stems of *Garcinia cowa* Roxb. and their cytotoxic activities. Pharmazie 62 (7), 549-551.

Shen, J. and Yang, J. 2006a. Chemical constituents from fruit of *Garcinia cowa*. Zhongguo Yaoxue Zazhi (Beijing, China) 41 (9), 660-661.

Shen, J., Yang, J.-S. and Zhou, S.-X. 2006b. Chemical constituents of fruit of *Garcinia xishuanbannanensis*. Zhongguo Tianran Yaowu 4 (6), 440-443.

Shen, J. and Yang, J.-S. 2006c. Two new xanthones from the stems of *Garcinia cowa*. Chem. Pharm. Bull. 54 (1), 126-128.

Silberberg, M., Gil-Izquierdo, A., Combaret, L., Remesy, C., Scalbert, A. and Morand, C. 2006. Flavanone metabolism in healthy and tumor-bearing rats. Biomed. Pharmacother. 60 (9), 529-535.

Soemiati, A., Kosela, S., Hanafi, M. and Harrison, L. J. 2006. Garcinopicrobenzophenone, a novel polyprenylbenzophenone from the bark of Indonesian *Garcinia picrorrhiza* Miq. ACGC Chem. Res. Commun. 20, 1-5.

Sohn, U.-D., Whang, W.-K., Ham, I.-H, Min, Y-S., Bae, K.-L., Yim, S.-H. and Lee, Y.-P. 2003. Process for preparing apigenin-7-O-*β*-D-glucuronide from *Clerodendron trichotomum* leaves. PCT Int. Appl. 27 pp. December, 4.

Sukpondma, Y., Rukachaisirikul, V. and Phongpaichit, S. 2005. Xanthone and sesquiterpene derivatives from the fruits of *Garcinia scortechinii*. J. Nat. Prod. 68 (7), 1010-1017.

Suksamrarn, S., Komutiban, O., Ratananukul, P., Chimnoi, N., Lartpornmatulee, N. and Suksamrarn, A. 2006. Cytotoxic prenylated xanthones on the young fruit of *Garcinia mangostana*. Chem. Pharm. Bull. 54 (3), 301-305.

Sultana, N., Akanda, S. I., Bhuiyan, R. A., Bagum, S. A., Kazi, M. A. I. and Sharkar, A. M. 2005. A neo-clerodane diterpenoid from *Clerodendrum viscosum leaves* Bangladesh. J. Sci. Ind. Res. 40 (3-4), 337-341.

Taher, M., Idris, M. S. and Arbain, D. 2007. Antimicrobial, antioxidant and cytotoxic activities of *Garcinia eugenifolia* and *Calophyllum enervosum*. Iran. J. Pharm. Ther. 6 (1), 93-98.

Terashima, K., Ishida, T., Furukawa, T., Takaya, Y. and Niwa, M. 2008. Constituents of green and ripened fruit of *Garcinia subelliptica*. Heterocycles 75 (2), 407-413.

Thongchai, W., Liawruangrath, B. and Liawruangrath, S. 2007. High-performance liquid chromatographic determination of arbutin in skin-whitening creams and medicinal plant extracts. J. Cosmet. Sci. 58 (1), 35-44.

Upo, U. 2005. Ethnobotany of Buddhist and Muslin Thais inn the some locations in the lower part of southern Thailand. Doctor Of Philosophy in Biology, Chiang Mai University.

Vidya, S. M., Krishna, V., Manjunatha, B. K., Mankani, K. L., Ahmed, M. and Singh, S. D. J. 2007. Evaluation of hepatoprotective activity of *Clerodendrum serratum* L. Indian J. Exp. Biol., 45 (6), 538-542.

Vieira, L. M. M., Kijjoa, A., Silva, A. M. S., Mondranondra, I.-O., Kengthong, S., Gales, L., Damas, A. M. and Herz, W. 2004. Lanostanes and friedolanostanes from the bark of *Garcinia speciosa*. Phytochemistry 65 (4), 393-398.

Vu, D. H., Ba, T. C., Luu, H. and Pham, G. D. 2006. Chemical study of *Clerodendron cyrtophyllum* Turcz. Part I - chemical constituents of n-hexane extract of the roots. Tap Chi Hoa Hoc, 44 (6), 704-706.

Wu, C.-C., Lu, Y.-H., Wei, B.-L., Yang, S.-C., Won, S.-J. and Lin, C.-N. 2008a. Phloroglucinols with Prooxidant Activity from *Garcinia subelliptica*. J. Nat. Prod. 71(2), 246-250.

Wu, X., Ke, C.-Q., Yang, Y.-P. and Ye, Y. 2008b. New biphenyl constituents from *Garcinia oblongifolia*. Helv. Chim. Acta 91 (5), 938-943.

Yang, N.-Y., Han, Q.-B., Cao, X.-W., Qiao, C.-F., Song, J.-Z., Chen, S.-L., Yang, D.-J., Yiu, H. and Xu, H.-X. 2007. Two new xanthones isolated from the stem bark of *Garcinia lancilimba*. Chem. Pharm. Bull. 55 (6), 950-952.

Yang, H., Hou, A.-J., Mei, S.-X, Sun, H.-D. and Che, C.-T. 2002. Constituents of *Clerodendrum bungei*. J. Asian Nat. Prod. Res., 4 (3), 165-169.

Yu, L., Zhao, M., Yang, B., Zhao, Q. and Jiang, Y. 2007. Phenolics from hull of *Garcinia mangostana* fruit and their antioxidant activities. Food. Chem. 104 (1), 176-181.

Zadernowski, R., Czaplicki, S. and Naczk, M. 2009. Phenolic acid profiles of mangosteen fruits (*Garcinia mangostana*). Food. Chem. 112 (3), 685-689.

Zhong, F. F., Chen, Y., Mei, Z. N. and Yang, G. Z. 2007. Xanthones from the bark of *Garcinia xanthochymus*. Chin. Chem. Lett. 18 (7), 849-851.

Zhong, F.-F., Chen, Y. and Yang, G.-Z. 2008. Chemical constituents from the bark of *Garcinia xanthochymus* and their 1,1-diphenyl-2-picrylhydrazyl (DPPH) radical-scavenging activities. Helv. Chim. Acta. 91(9), 1695-1703.

APPENDIX

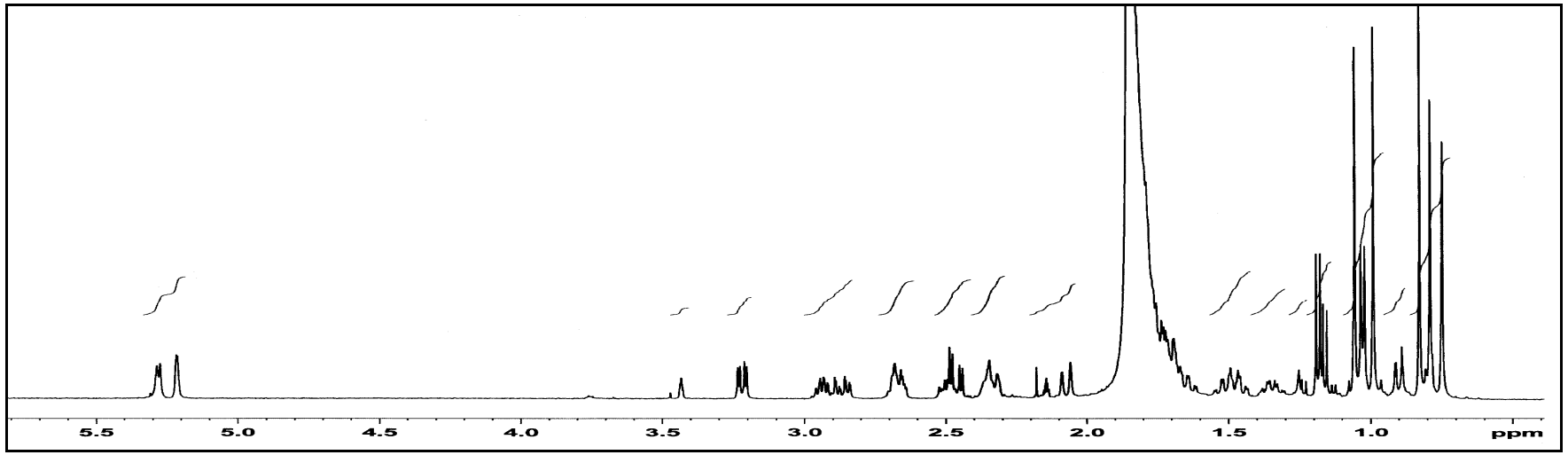


Figure 1 ^{1}H NMR (300 MHz) ($CDCl_3$+CD_3OD) spectrum of compound **SK1**

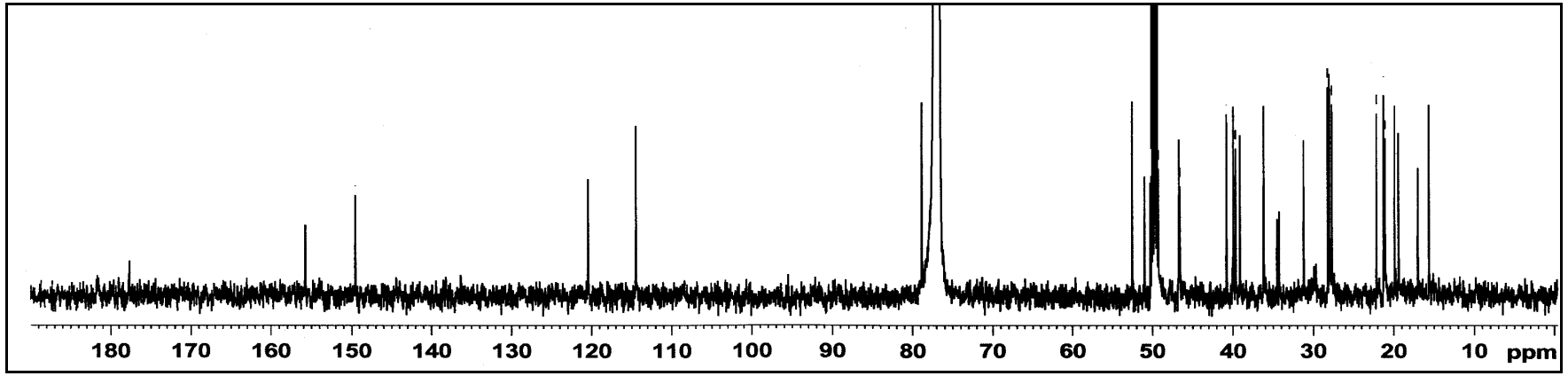


Figure 2 ^{13}C NMR (125 MHz) ($CDCl_3$+CD_3OD) spectrum of compound **SK1**

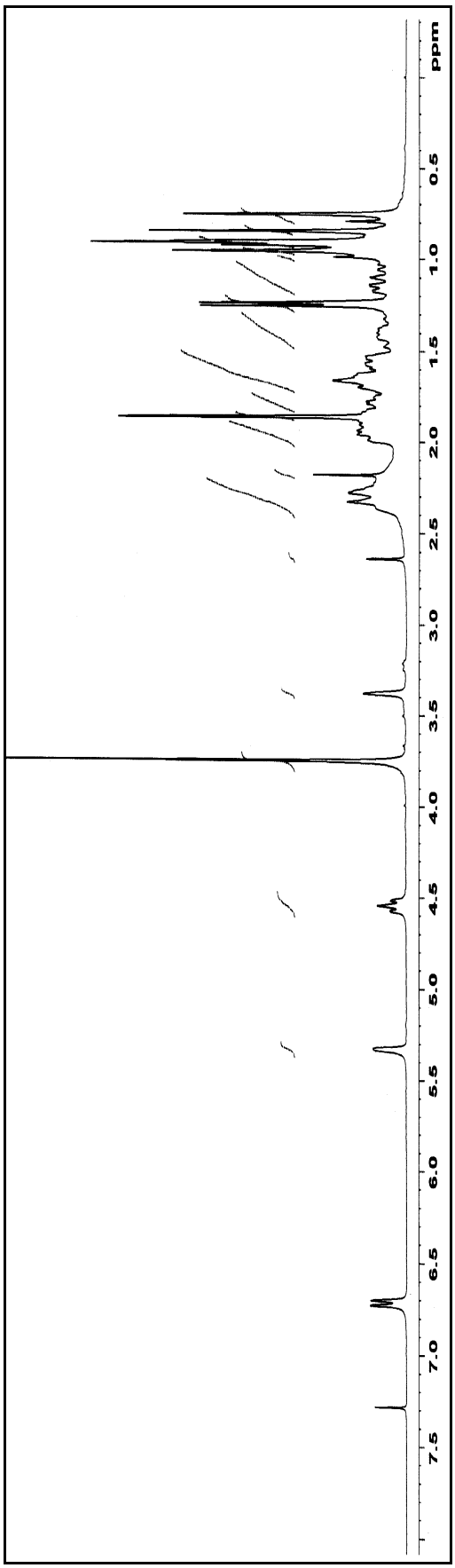


Figure 3 ^{1}H NMR (300 MHz) ($CDCl_3$) spectrum of compound **SK2**

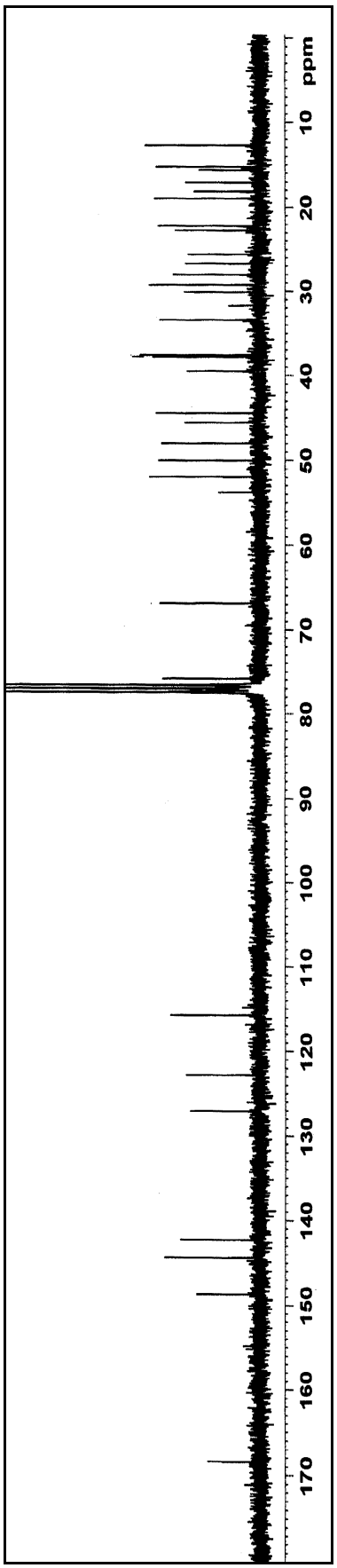


Figure 4 ^{13}C NMR (75 MHz) ($CDCl_3$) spectrum of compound **SK2**

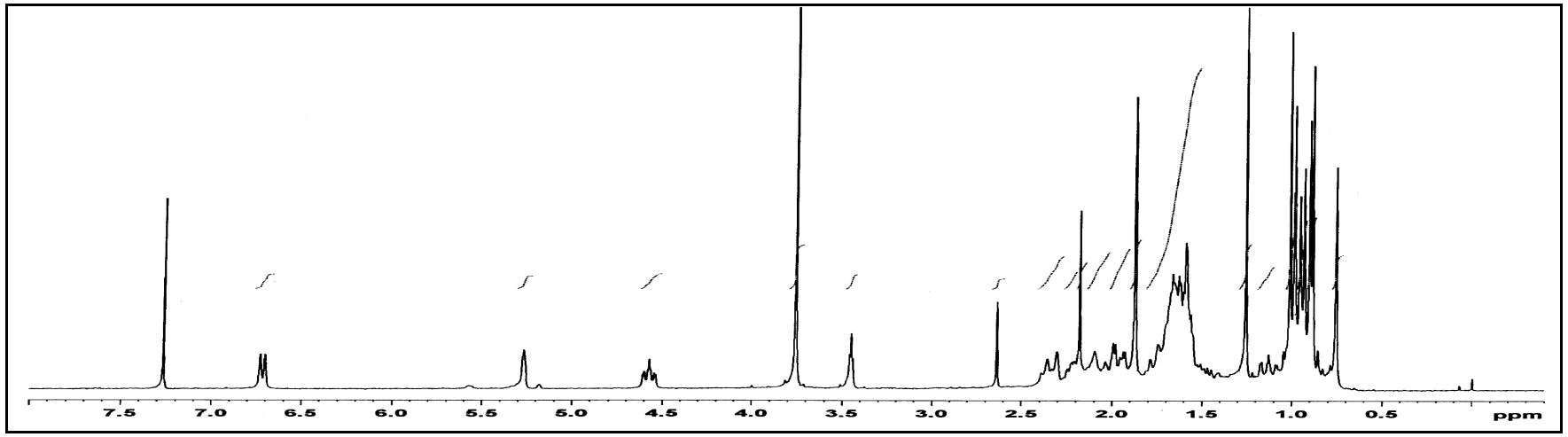


Figure 5 ^{1}H NMR (300 MHz) ($CDCl_3$) spectrum of compound **SK3**

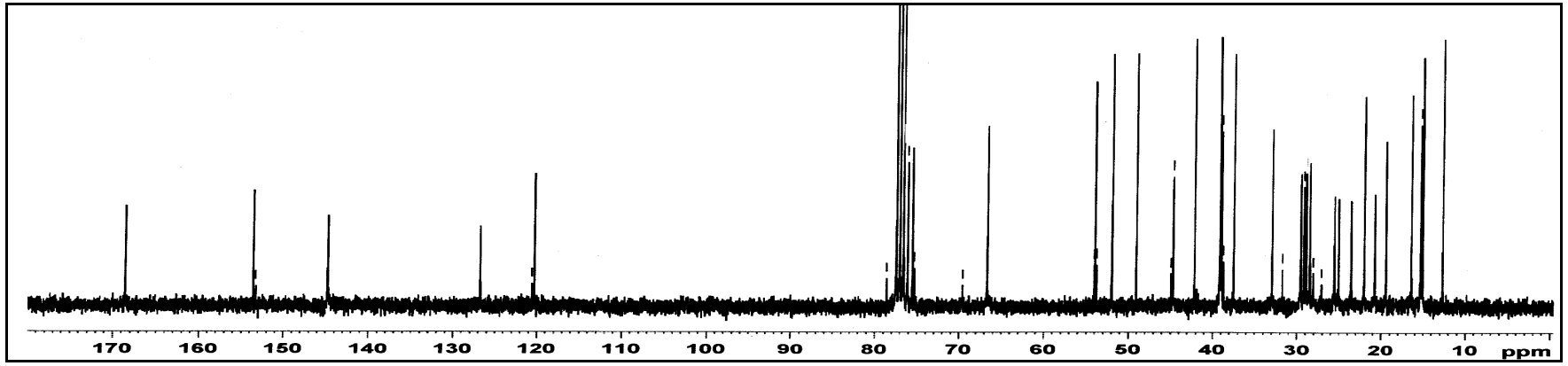


Figure 6 ^{13}C NMR (75 MHz) ($CDCl_3$) spectrum of compound **SK3**

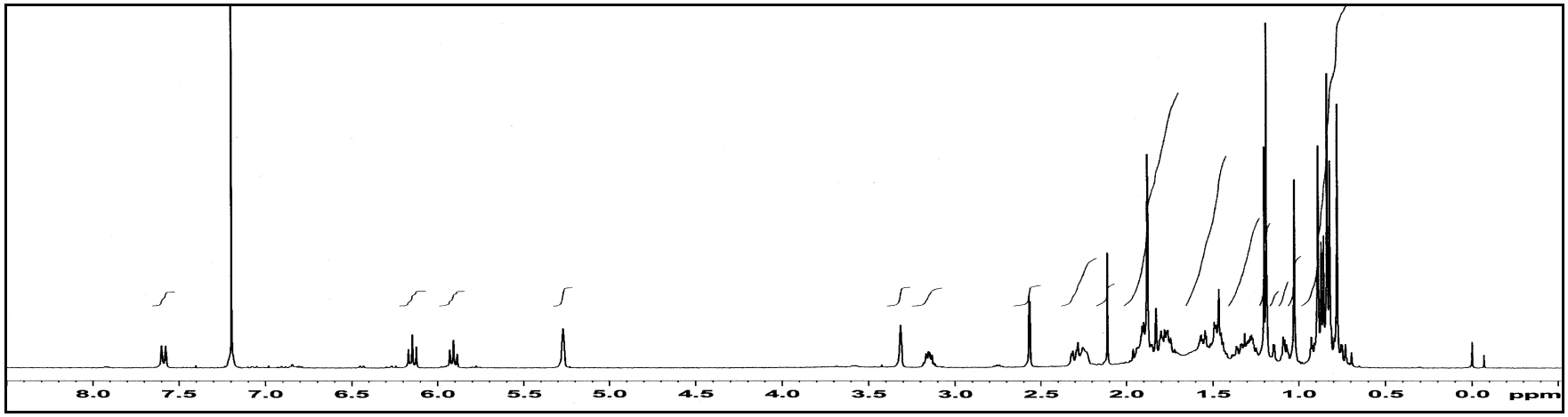


Figure 7 ^{1}H NMR (500 MHz) ($CDCl_3$) spectrum of compound **SK12**

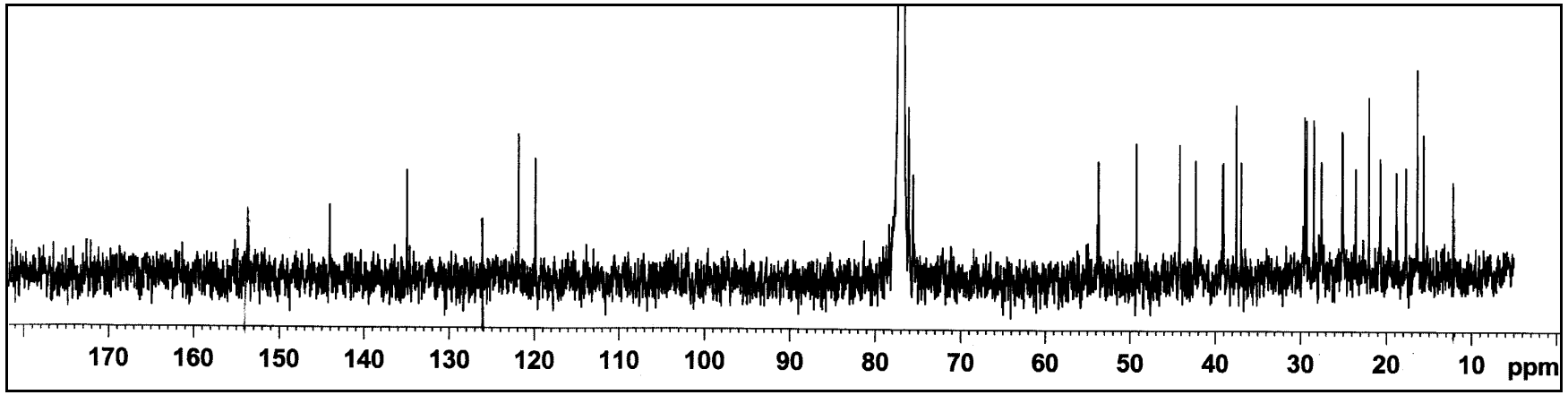


Figure 8 ^{13}C NMR (125 MHz) ($CDCl_3$) spectrum of compound **SK12**

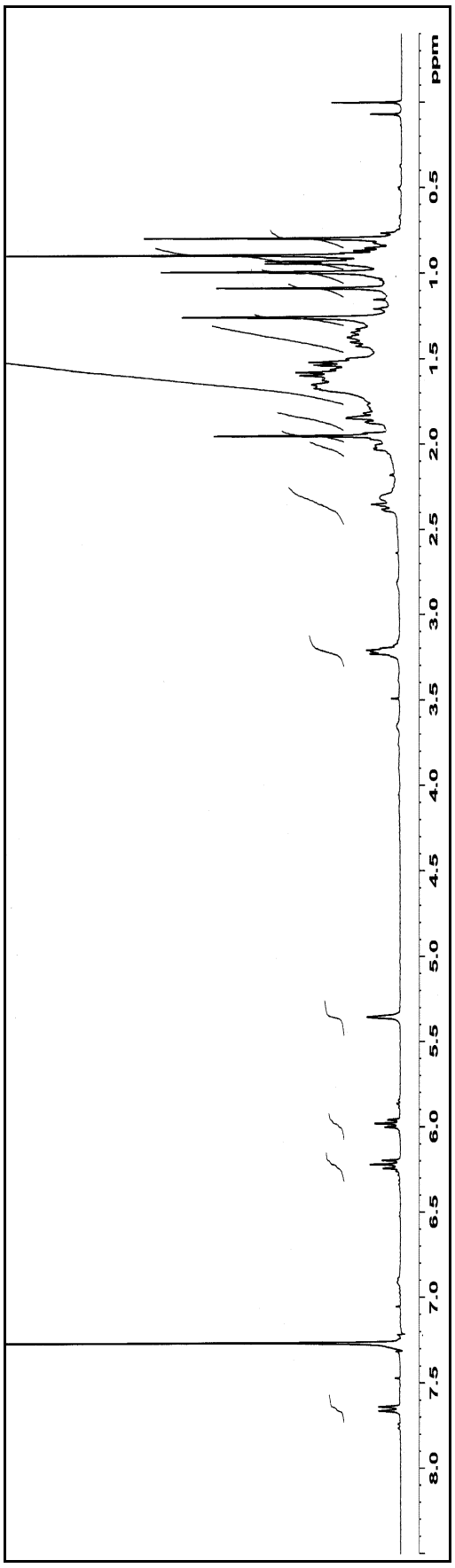


Figure 9 ^{1}H NMR (500 MHz) ($CDCl_3$) spectrum of compound **SK9**

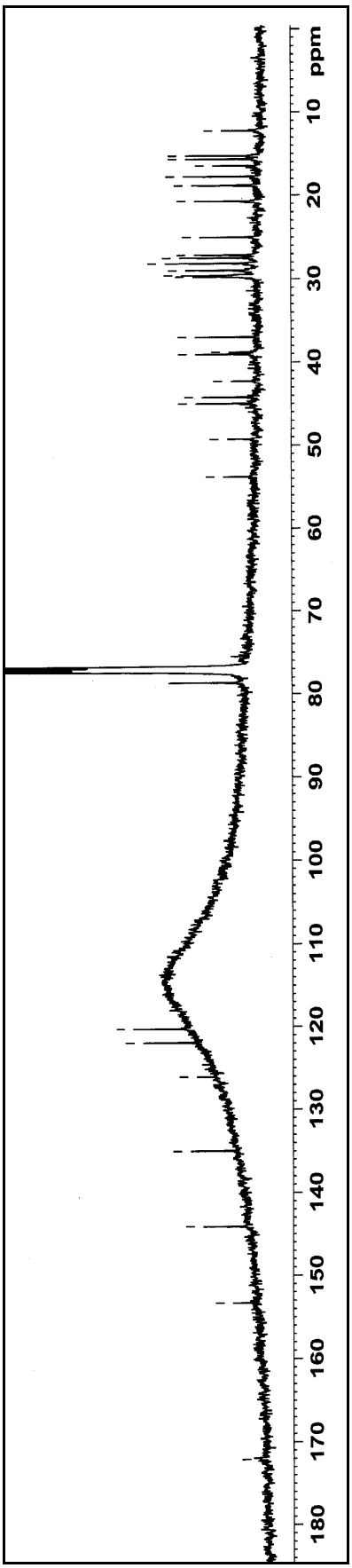


Figure 10 ^{13}C NMR (125 MHz) ($CDCl_3$) spectrum of compound **SK9**

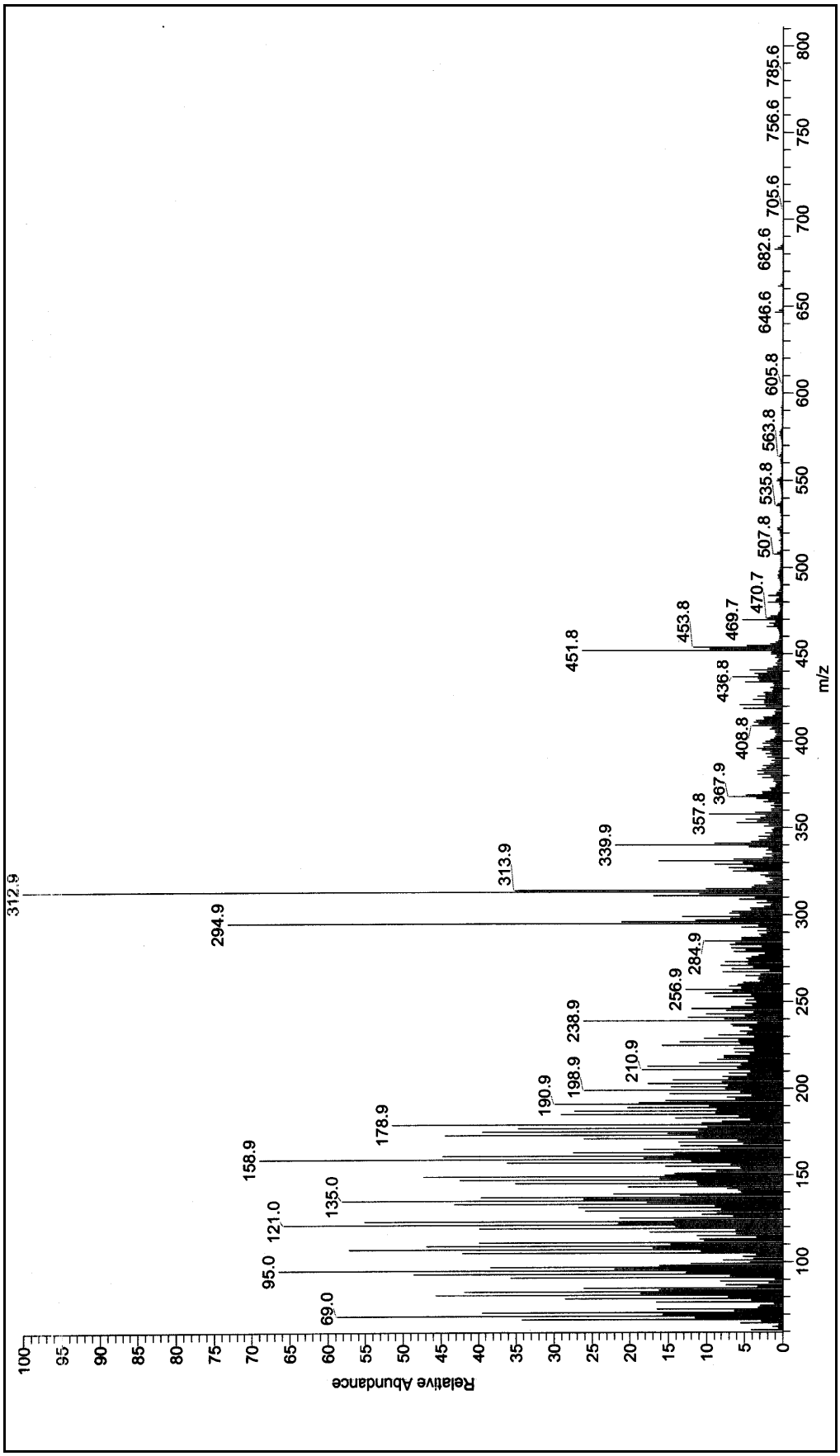


Figure 11 Mass spectrum of compound **SK9**

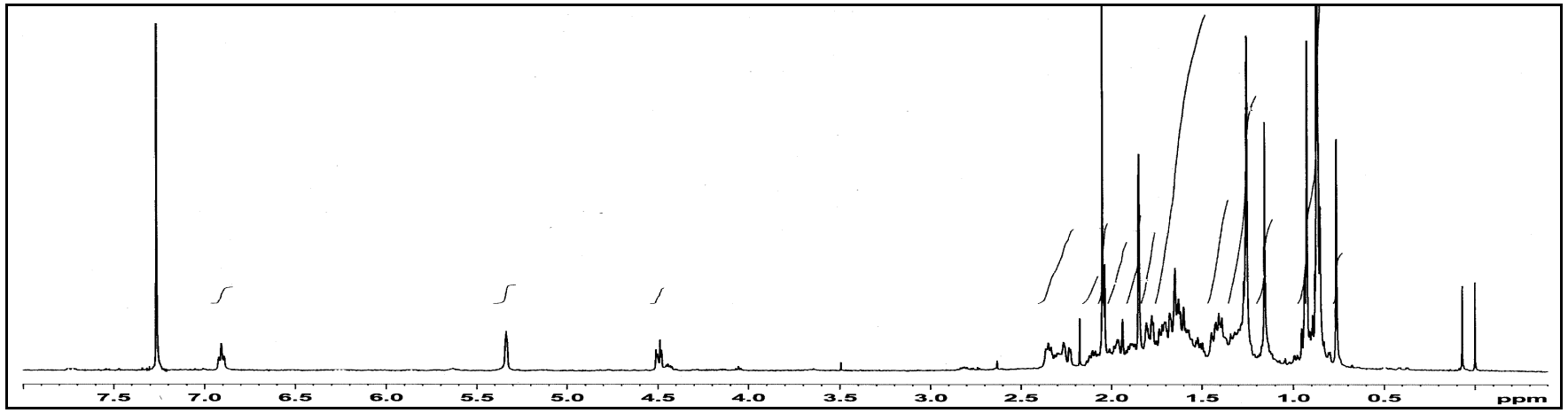


Figure 12 ^{1}H NMR (500 MHz) ($CDCl_3$) spectrum of compound **SK19**

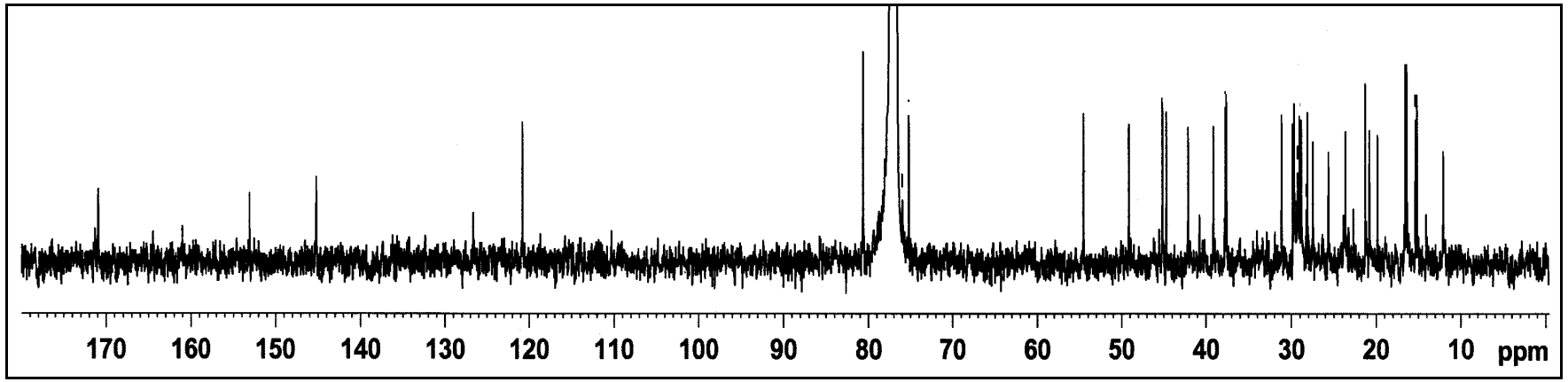


Figure 13 ^{13}C NMR (125 MHz) ($CDCl_3$) spectrum of compound **SK19**

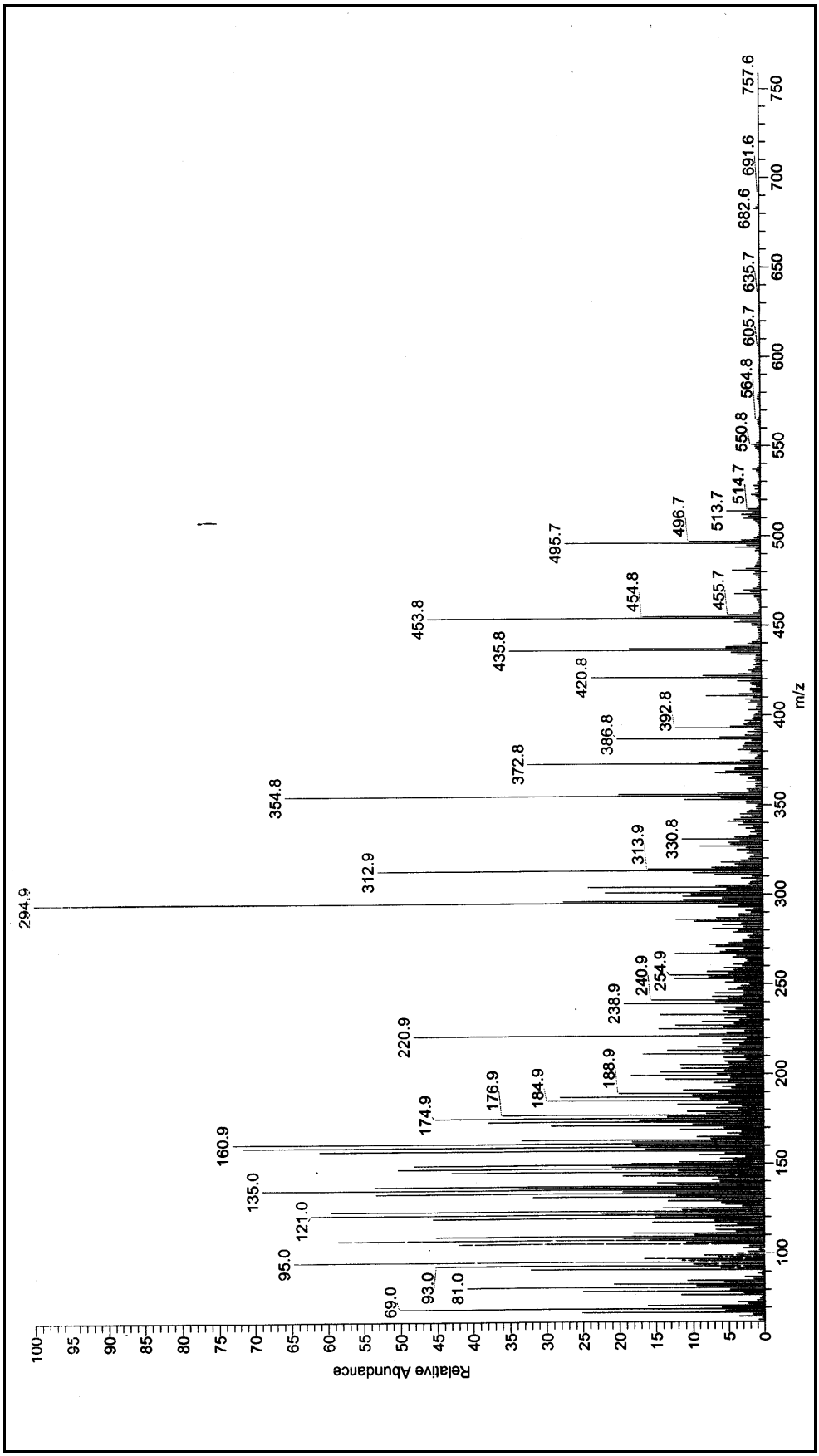


Figure 14 Mass spectrum of compound **SK19**

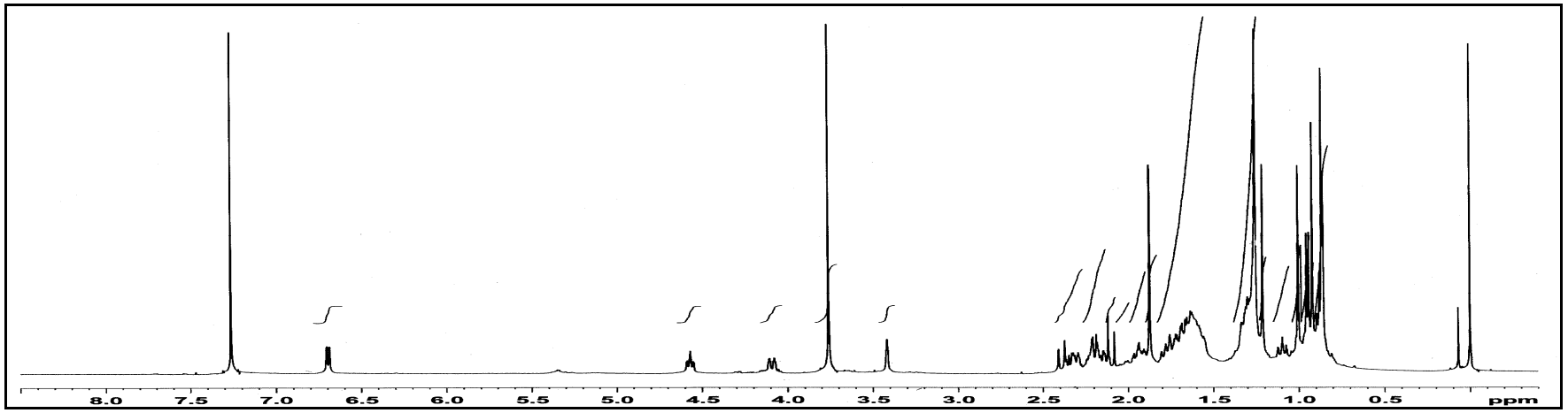


Figure 15 ^{1}H NMR (500 MHz) ($CDCl_3$) spectrum of compound **SK21**

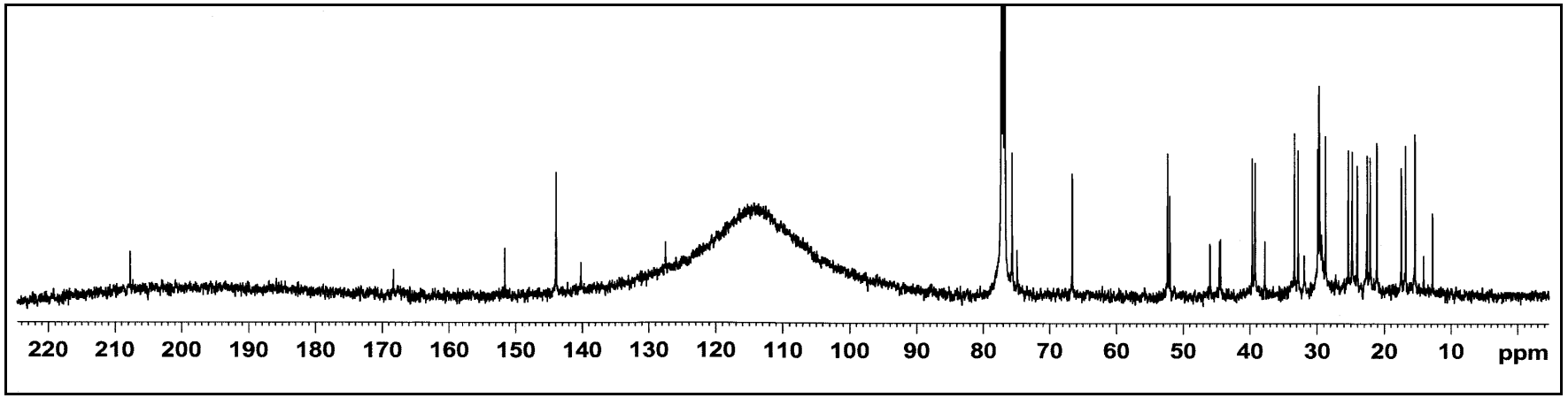


Figure 16 ^{13}C NMR (125 MHz) ($CDCl_3$) spectrum of compound **SK21**

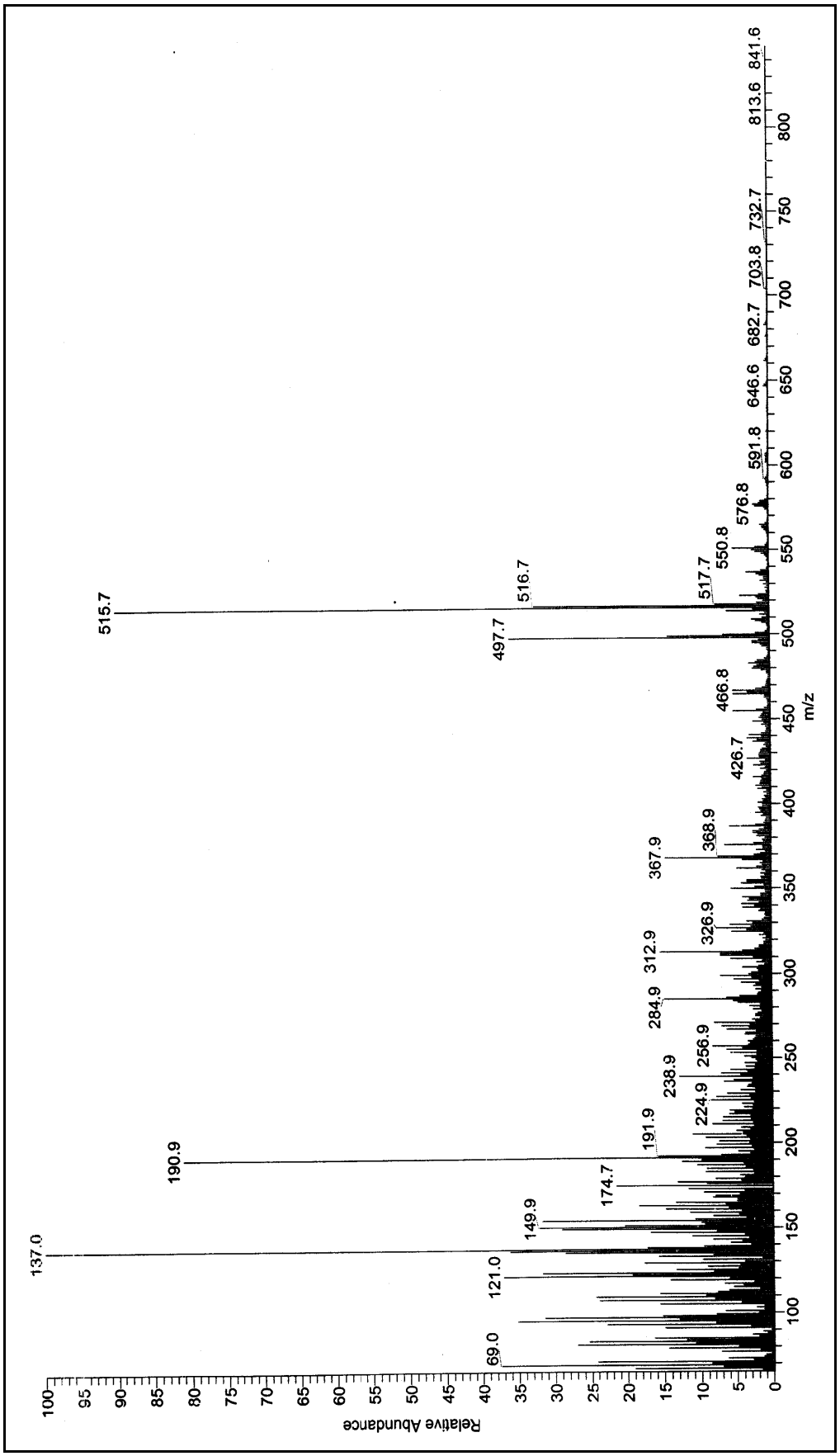


Figure 17 Mass spectrum of compound **SK21**

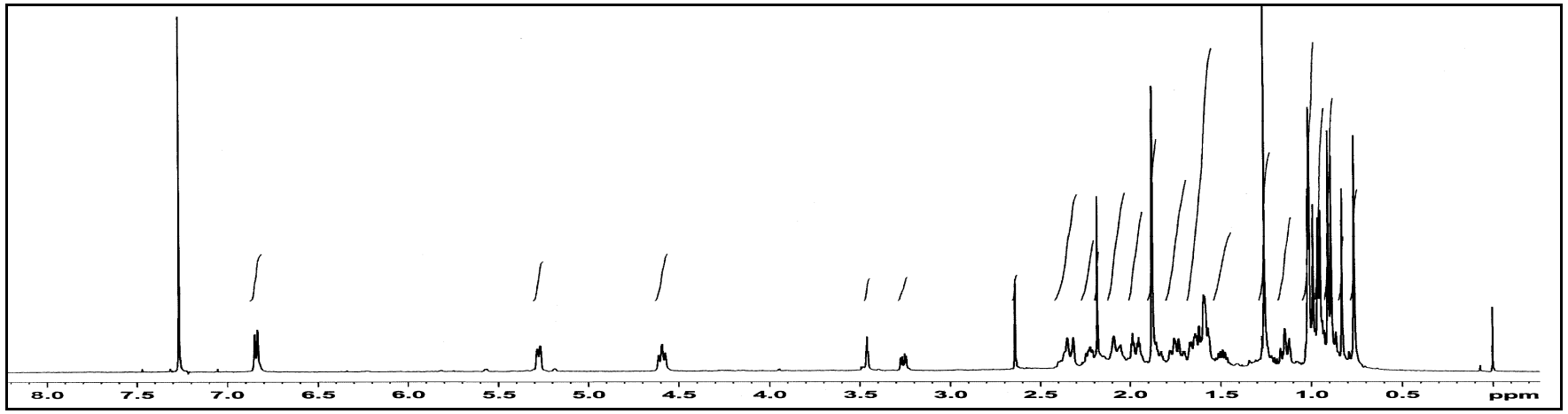


Figure 18 ^{1}H NMR (500 MHz) ($CDCl_3$) spectrum of compound **SK11**

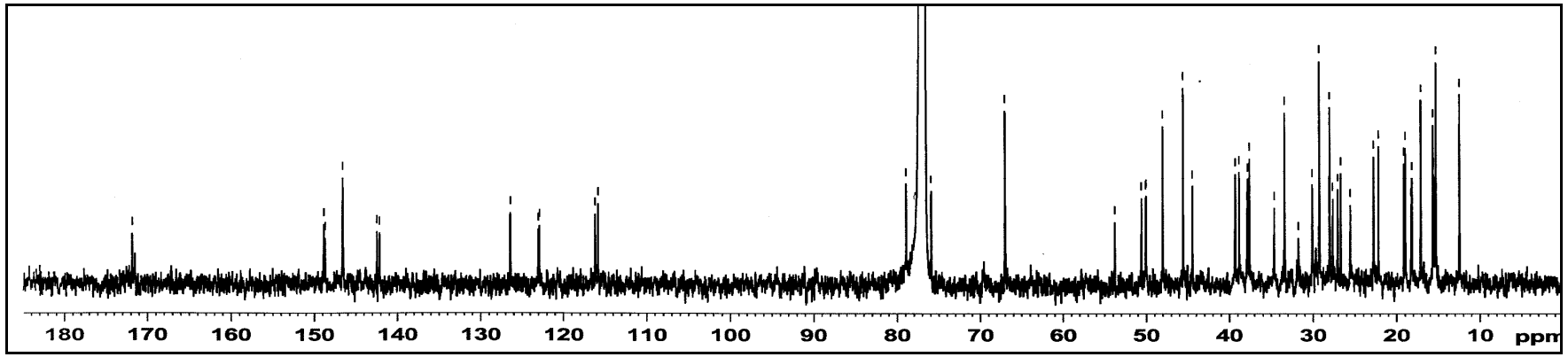


Figure 19 ^{13}C NMR (125 MHz) ($CDCl_3$) spectrum of compound **SK11**

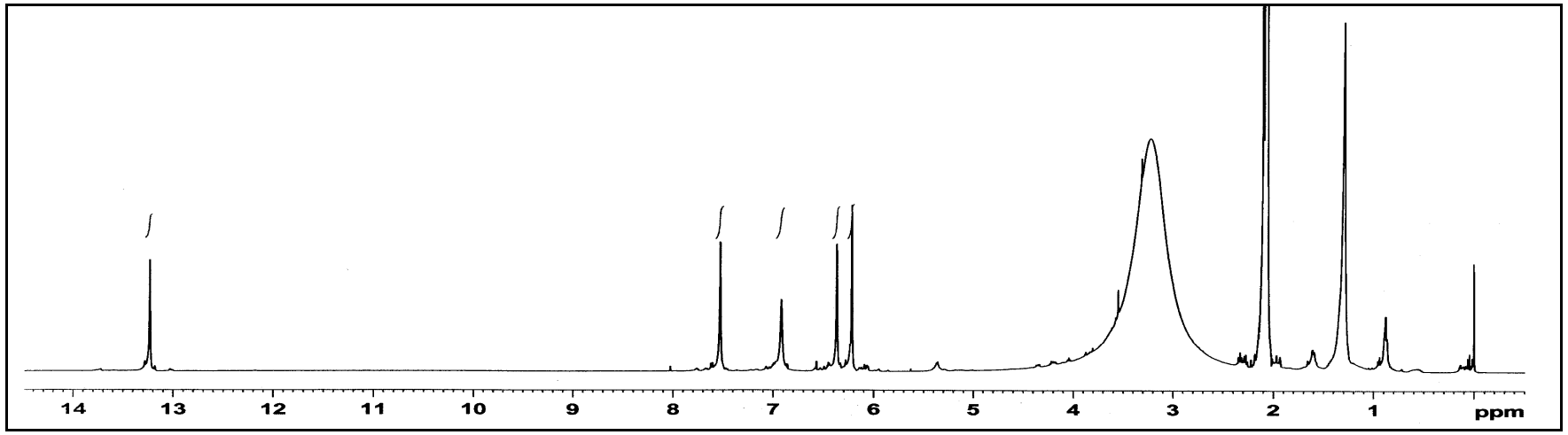


Figure 20 ^{1}H NMR (500 MHz) (Acetone-d_6) spectrum of compound **SK4**

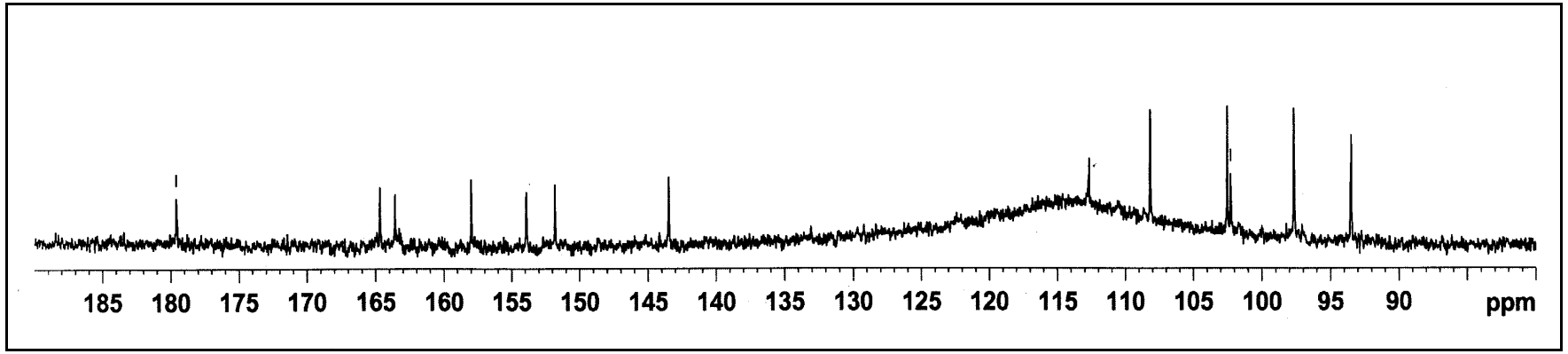


Figure 21 ^{13}C NMR (125 MHz) (Acetone-d_6) spectrum of compound **SK4**

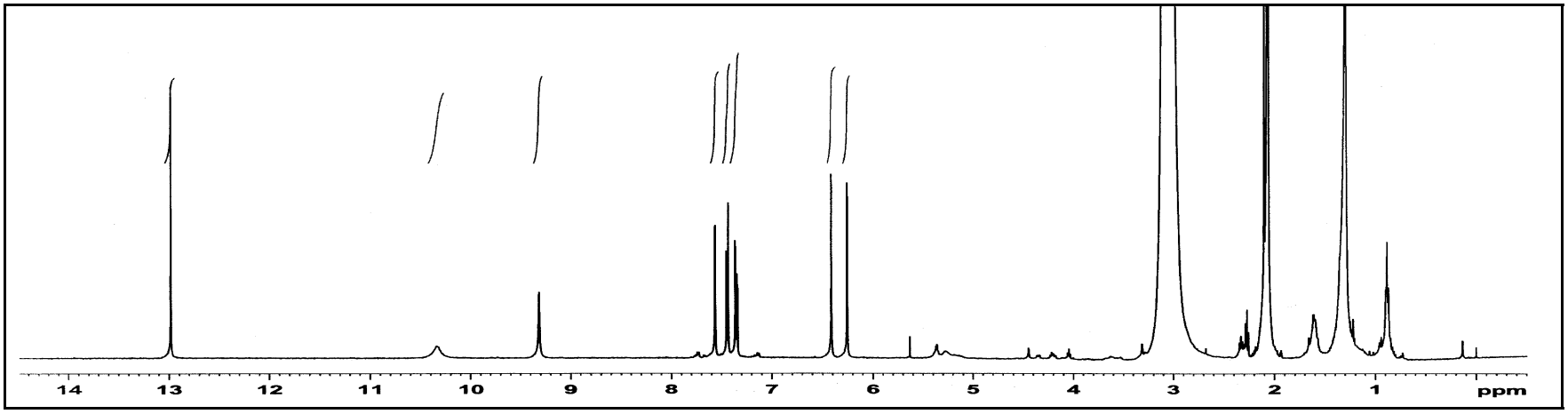


Figure 22 ^{1}H NMR (500 MHz) (Acetone-d_6) spectrum of compound **SK5**

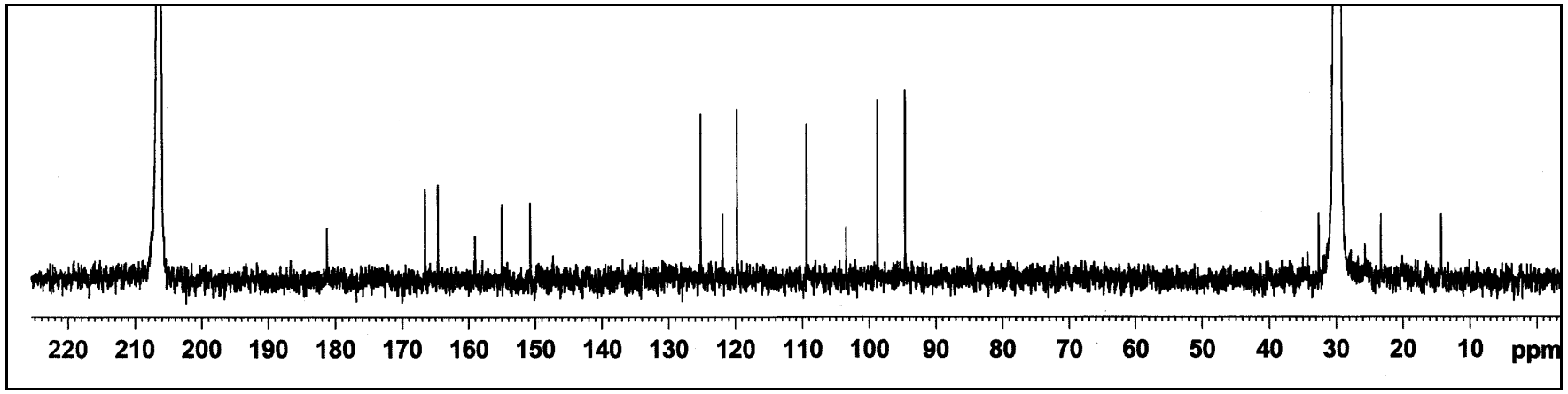


Figure 23 ^{13}C NMR (125 MHz) (Acetone-d_6) spectrum of compound **SK5**

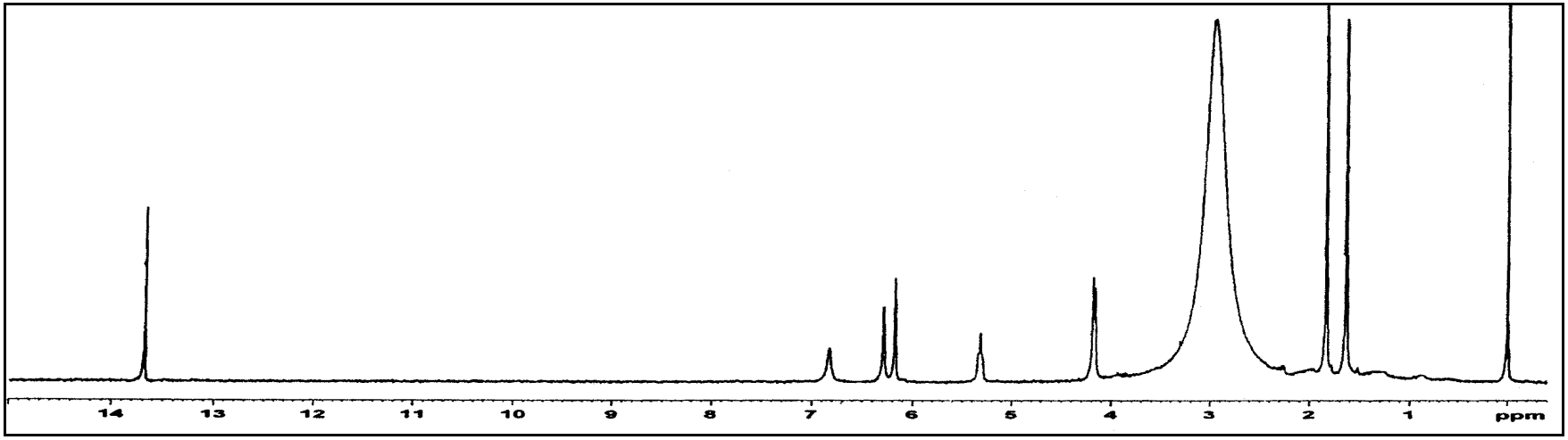


Figure 24 ^{1}H NMR (500 MHz) (Acetone-d_6) spectrum of compound **SK8**

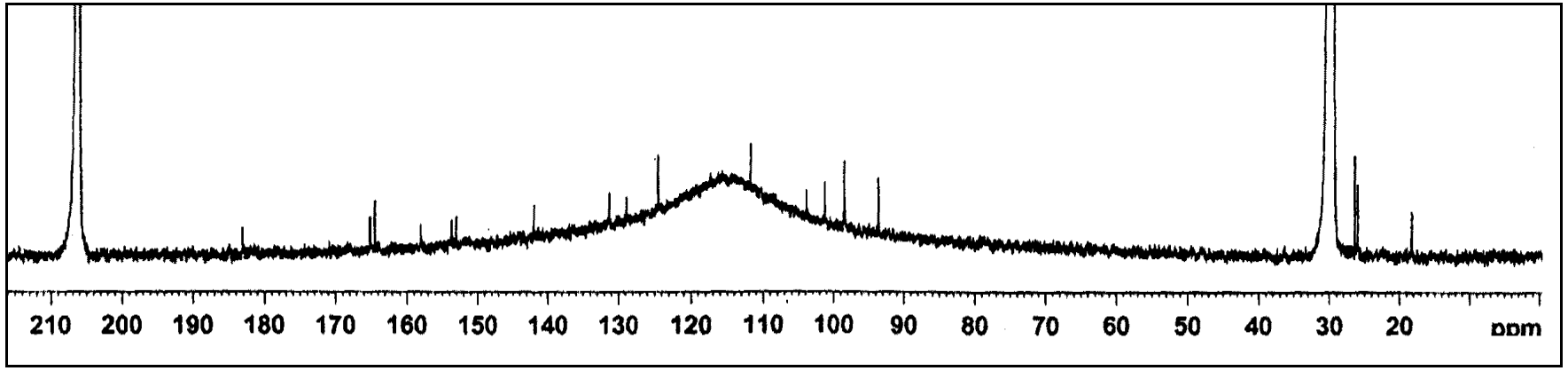


Figure 25 ^{13}C NMR (125 MHz) (Acetone-d_6) spectrum of compound **SK8**

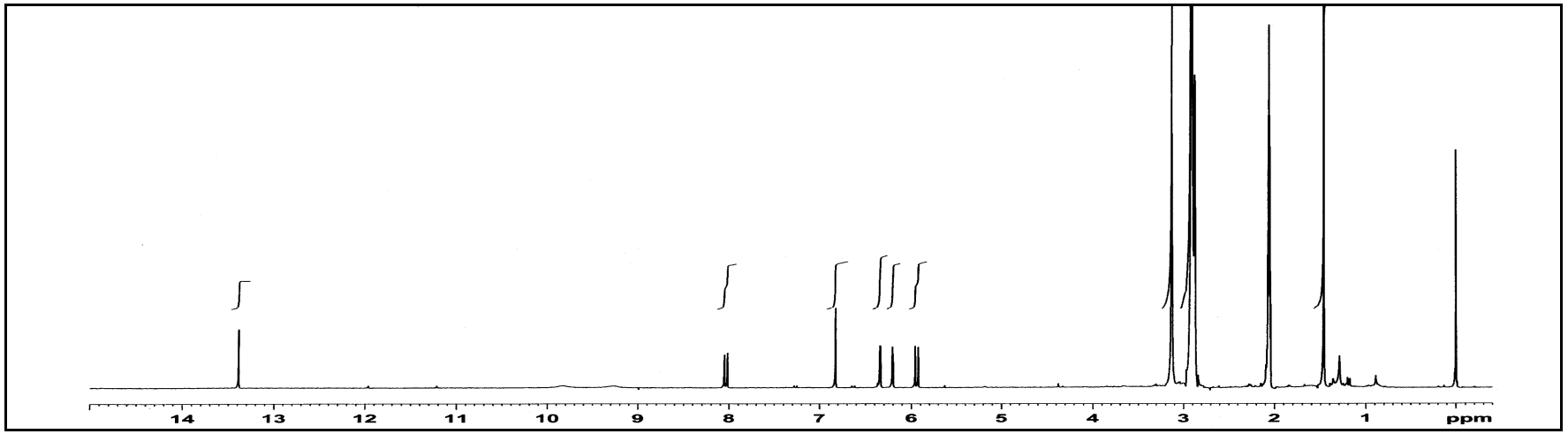


Figure 26 ^{1}H NMR (300 MHz) ($CDCl_3$) spectrum of compound **SK16**

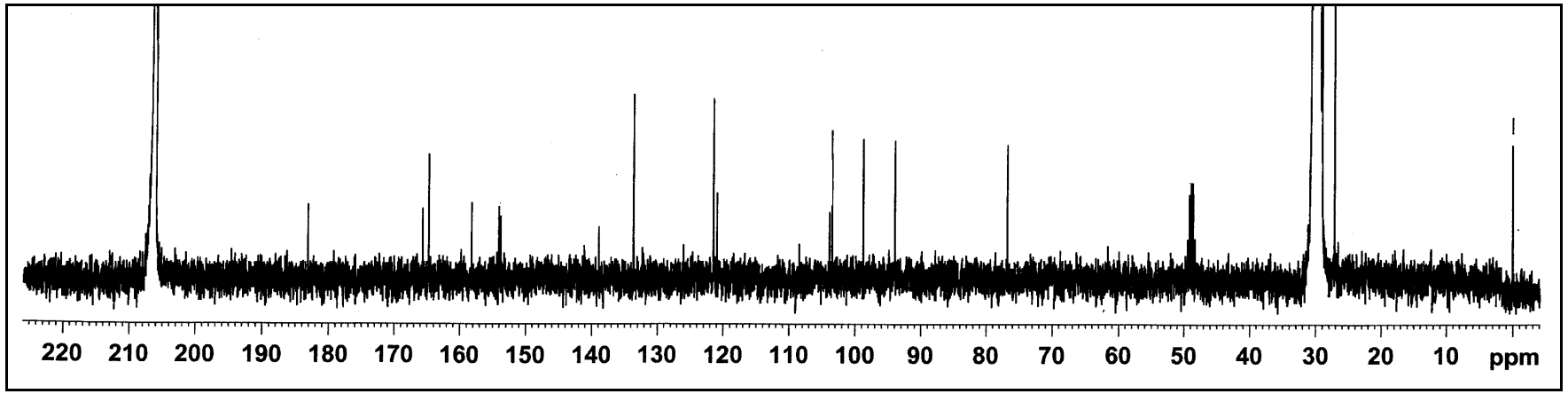


Figure 27 ^{13}C NMR (75 MHz) ($CDCl_3$) spectrum of compound **SK16**

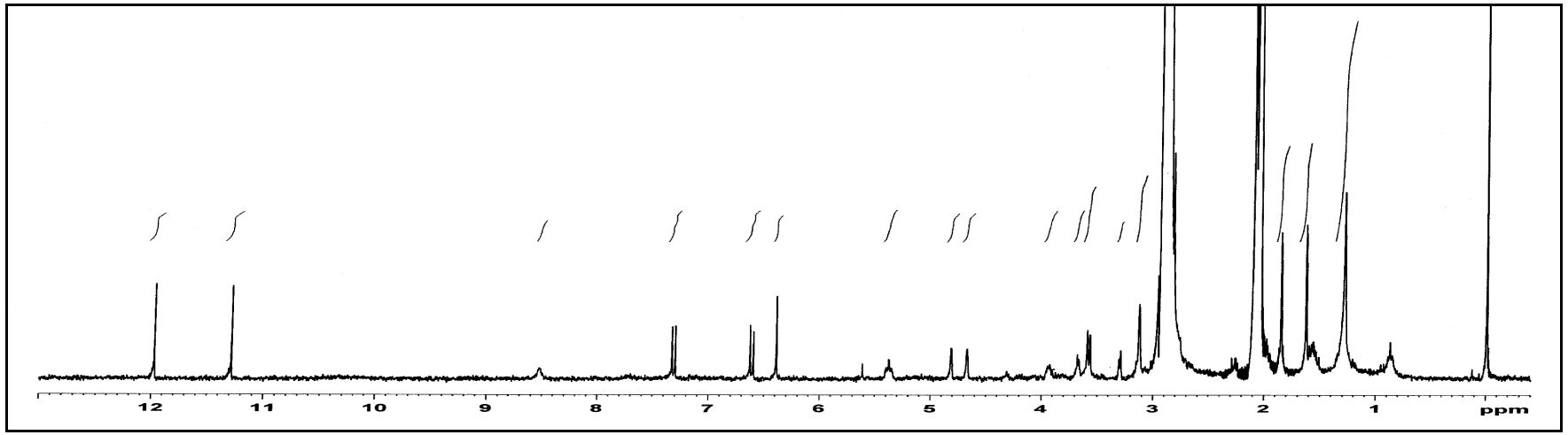


Figure 28 ^{1}H NMR (500 MHz) (Acetone-d_6) spectrum of compound **SK18**

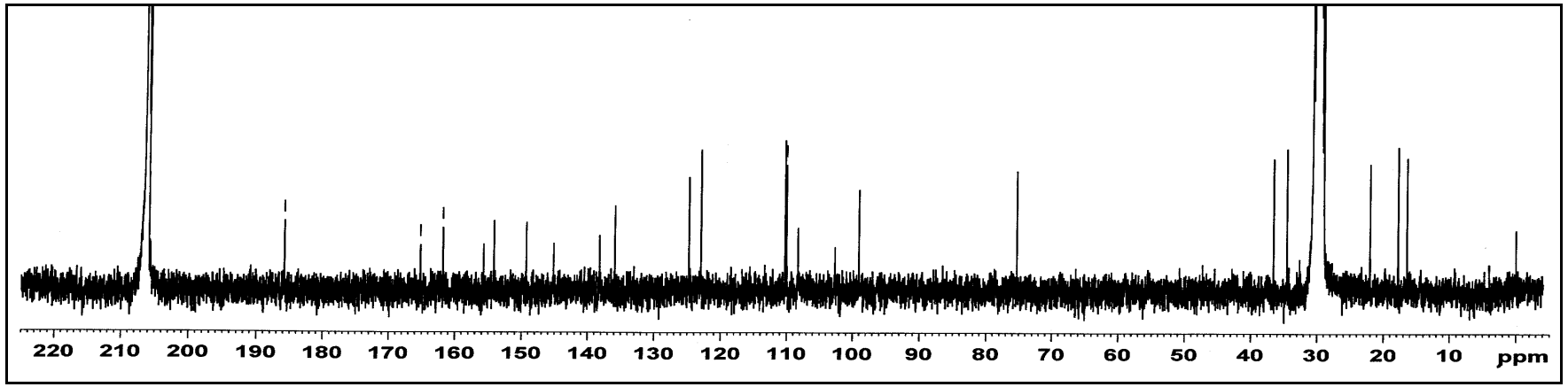


Figure 29 ^{13}C NMR (125 MHz) (Acetone-d_6) spectrum of compound **SK18**

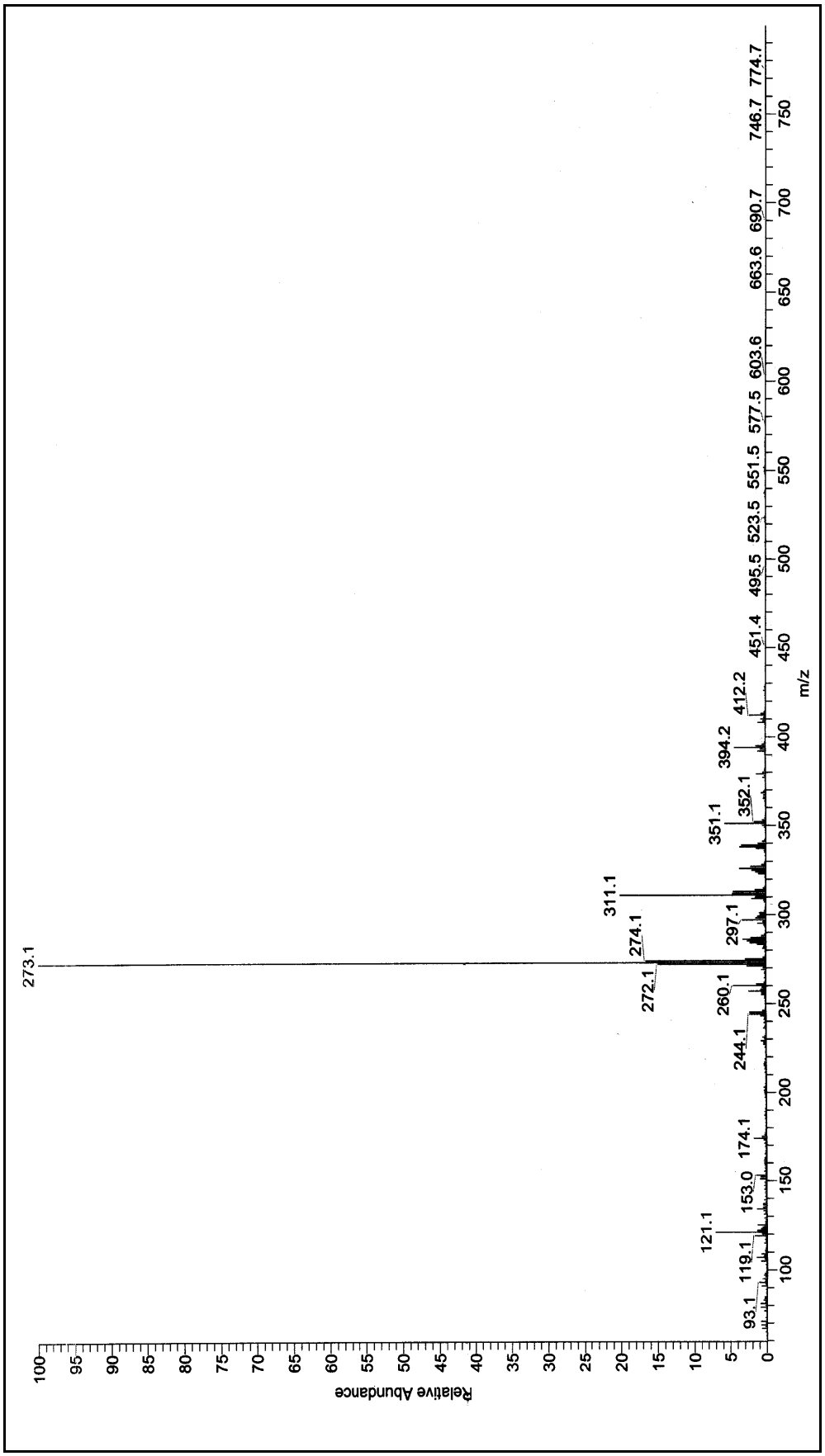


Figure 30 Mass spectrum of compound **SK18**

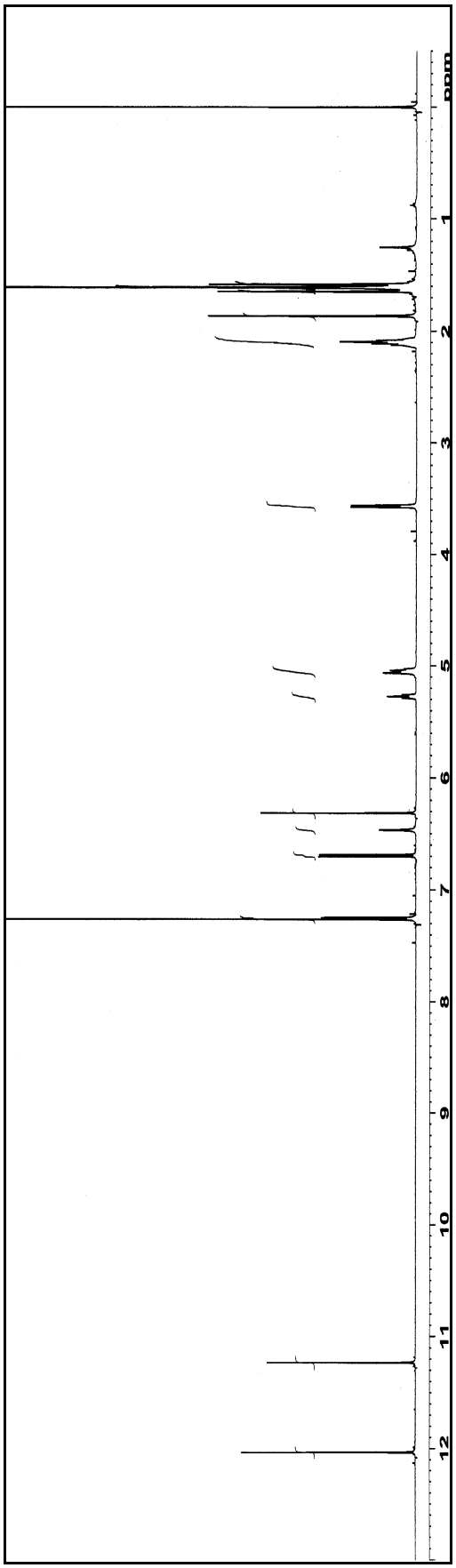


Figure 31 ^{1}H NMR (500 MHz) ($CDCl_3$) spectrum of compound **SK13**

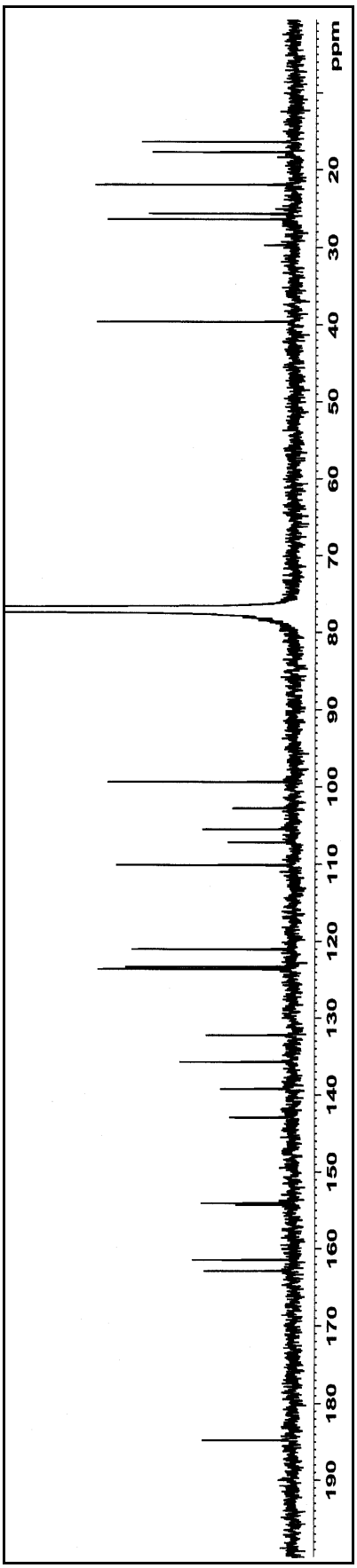


Figure 32 ^{13}C NMR (125 MHz) ($CDCl_3$) spectrum of compound **SK13**

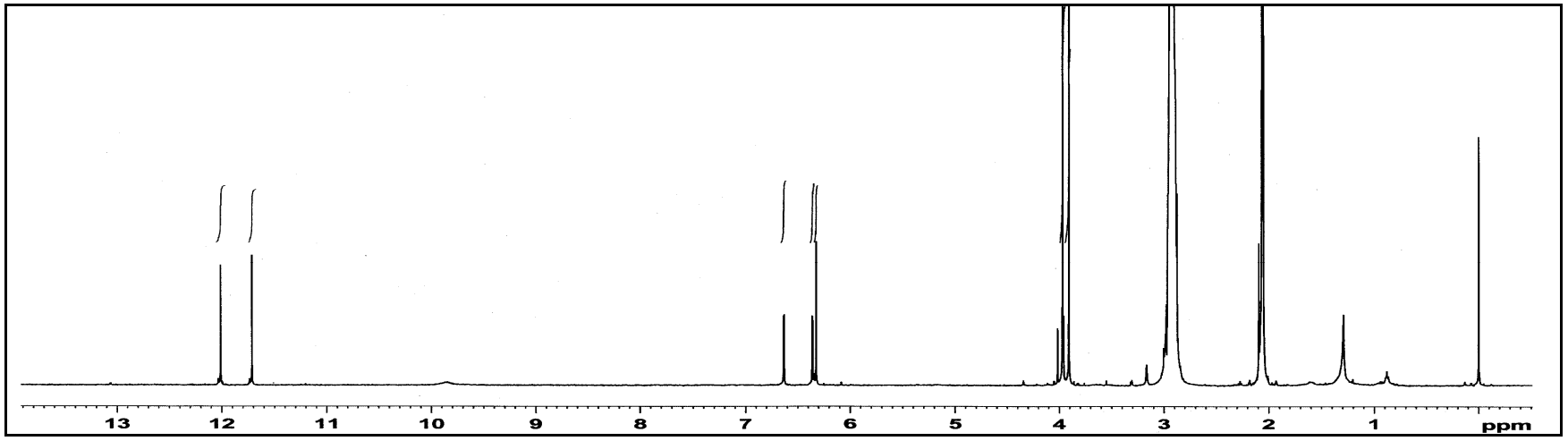


Figure 33 ^{1}H NMR (500 MHz) (Acetone-d_6) spectrum of compound **SK20**

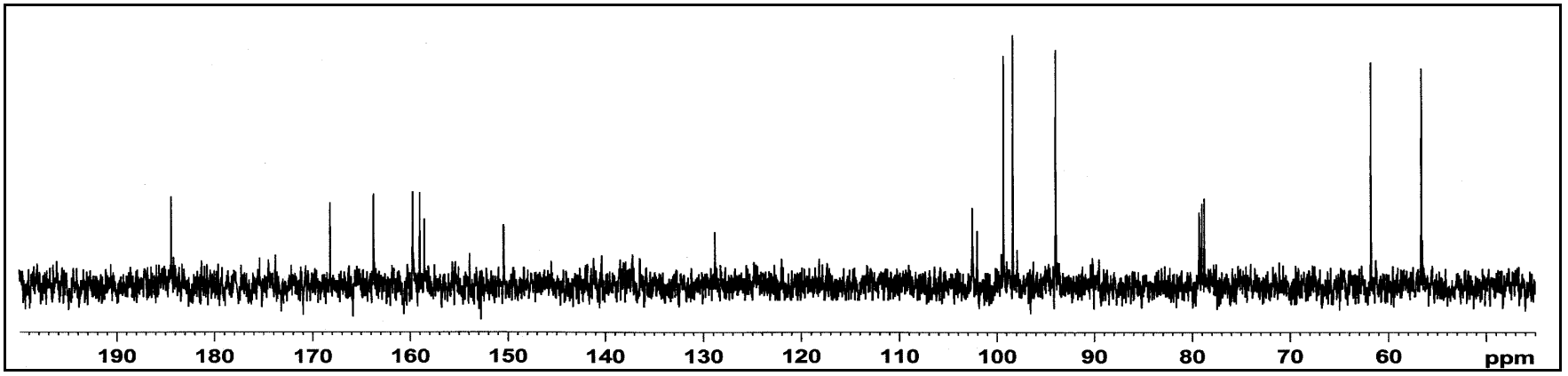


Figure 34 ^{13}C NMR (125 MHz) (Acetone-d_6) spectrum of compound **SK20**

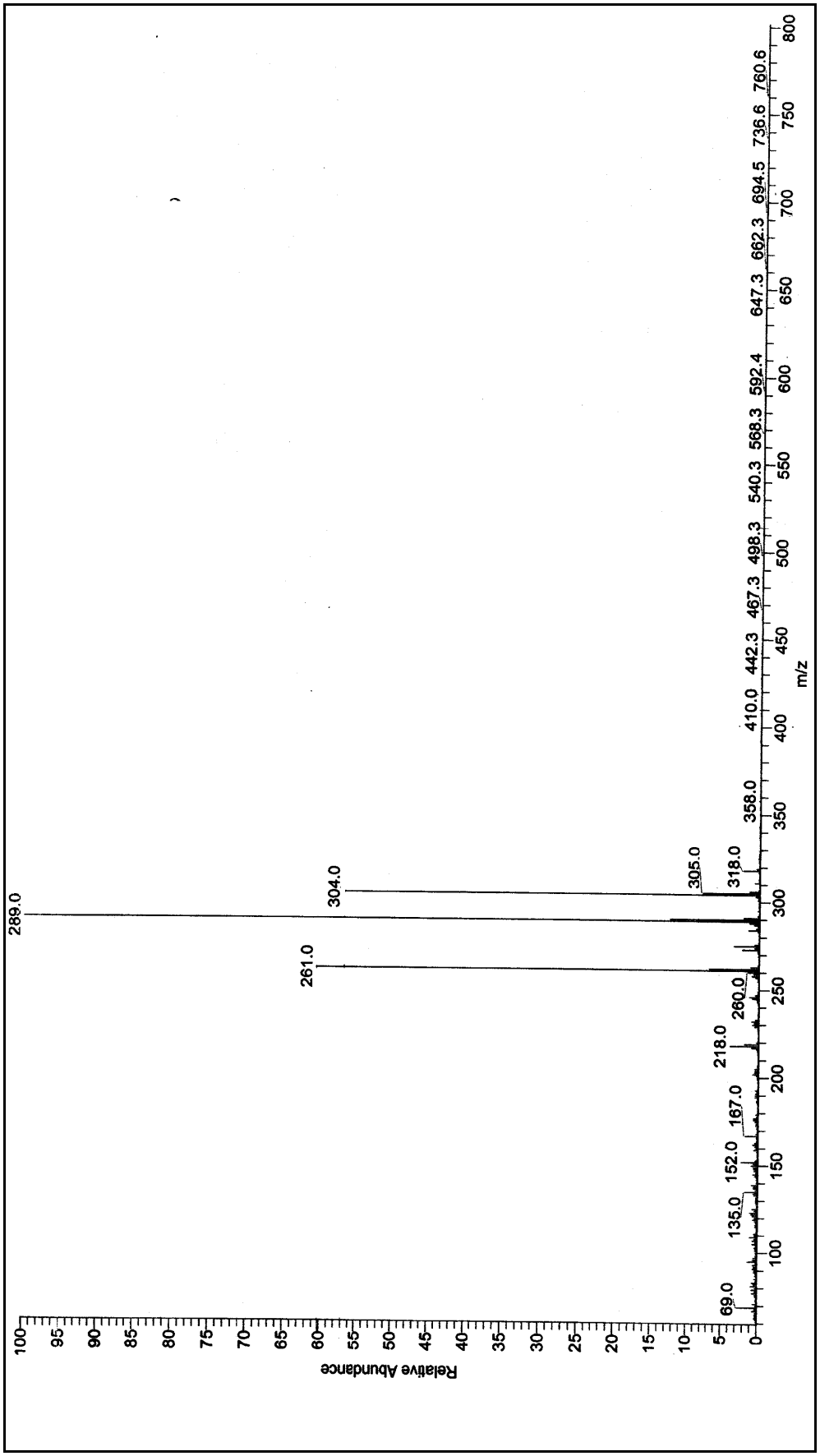


Figure 35 Mass spectrum of compound **SK20**

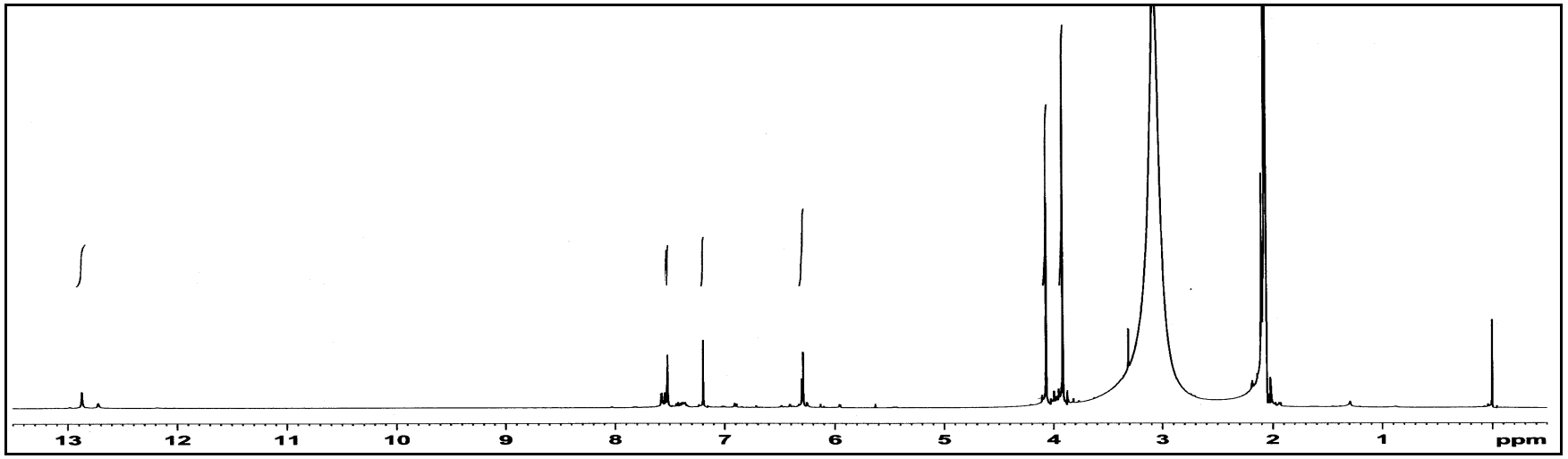


Figure 36 ^{1}H NMR (500 MHz) (Acetone-d_6) spectrum of compound **SK22**

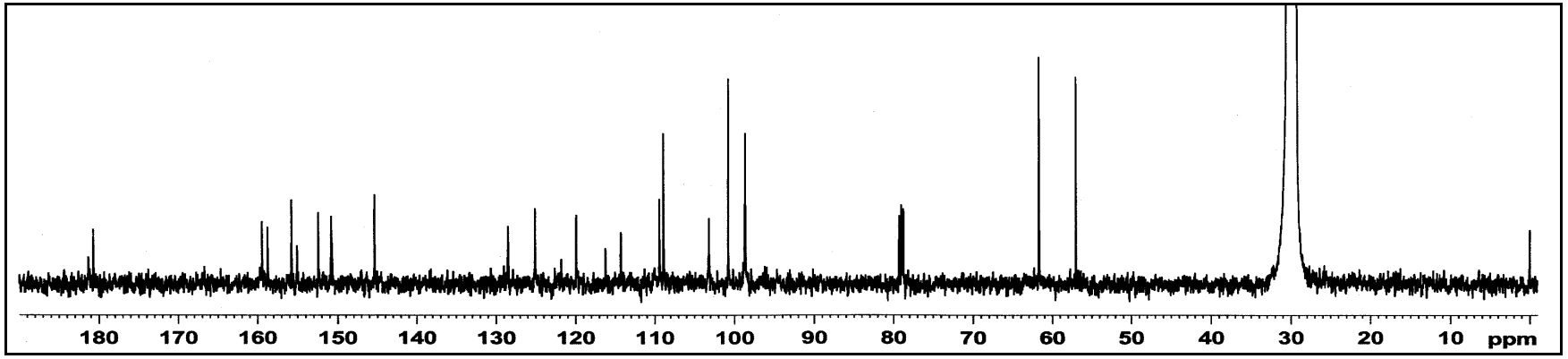


Figure 37 ^{13}C NMR (125 MHz) (Acetone-d_6) spectrum of compound **SK22**

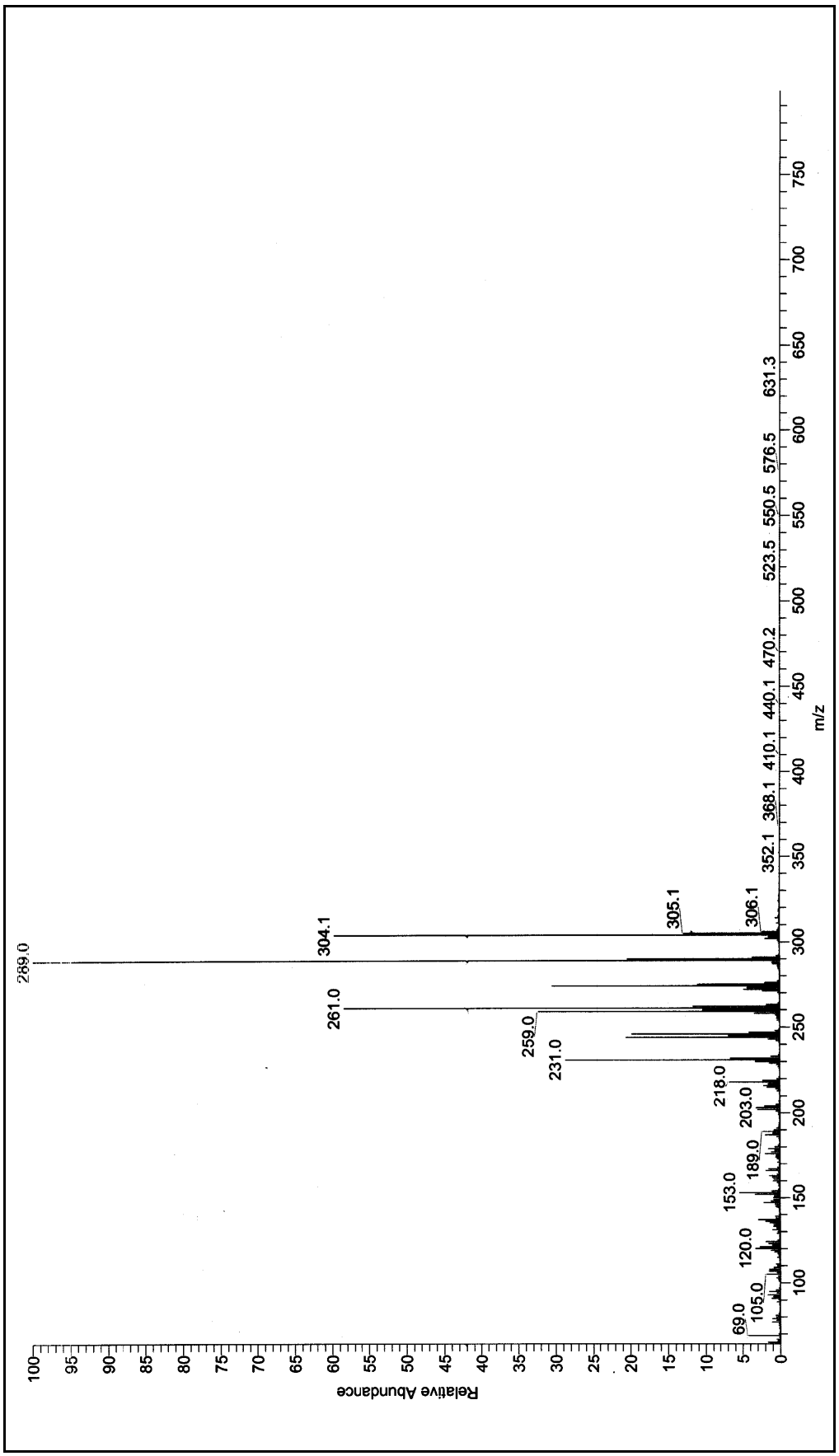


Figure 38 Mass spectrum of compound **SK22**

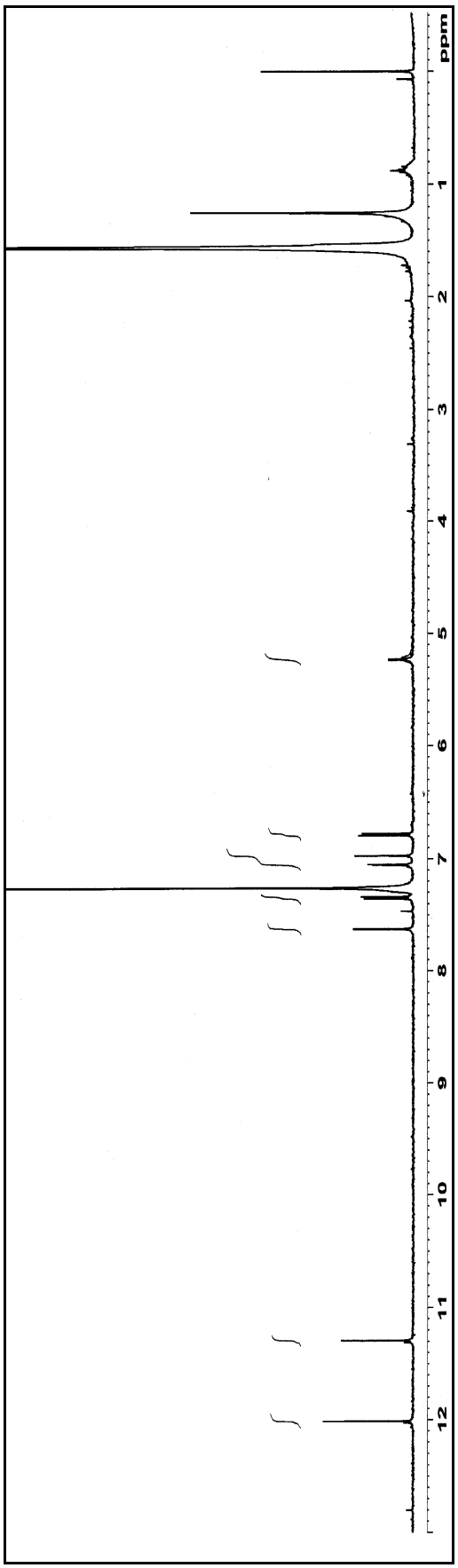


Figure 39 ^{1}H NMR (500 MHz) ($CDCl_3$) spectrum of compound **SK10**

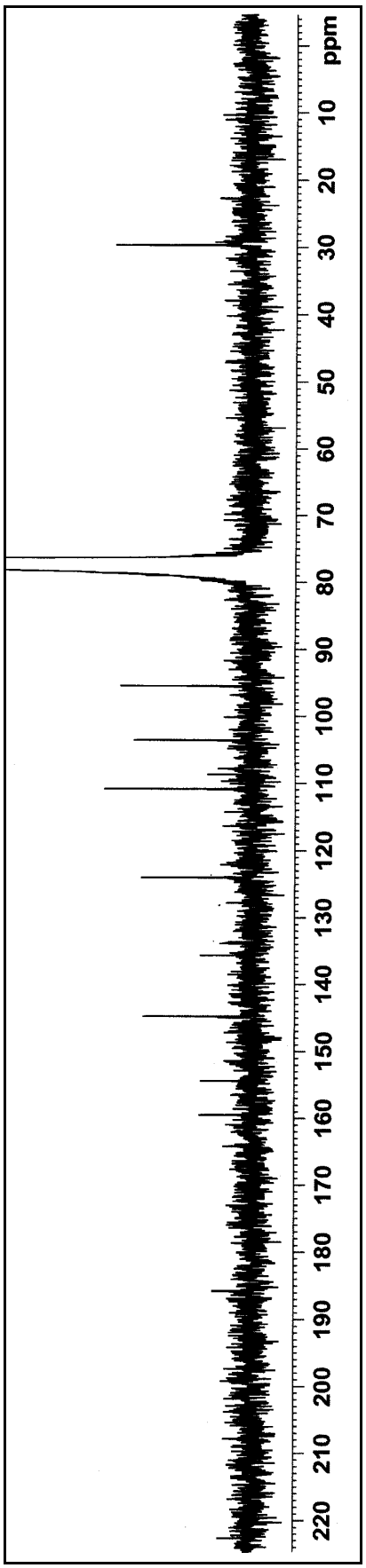


Figure 40 ^{13}C NMR (125 MHz) ($CDCl_3$) spectrum of compound **SK10**

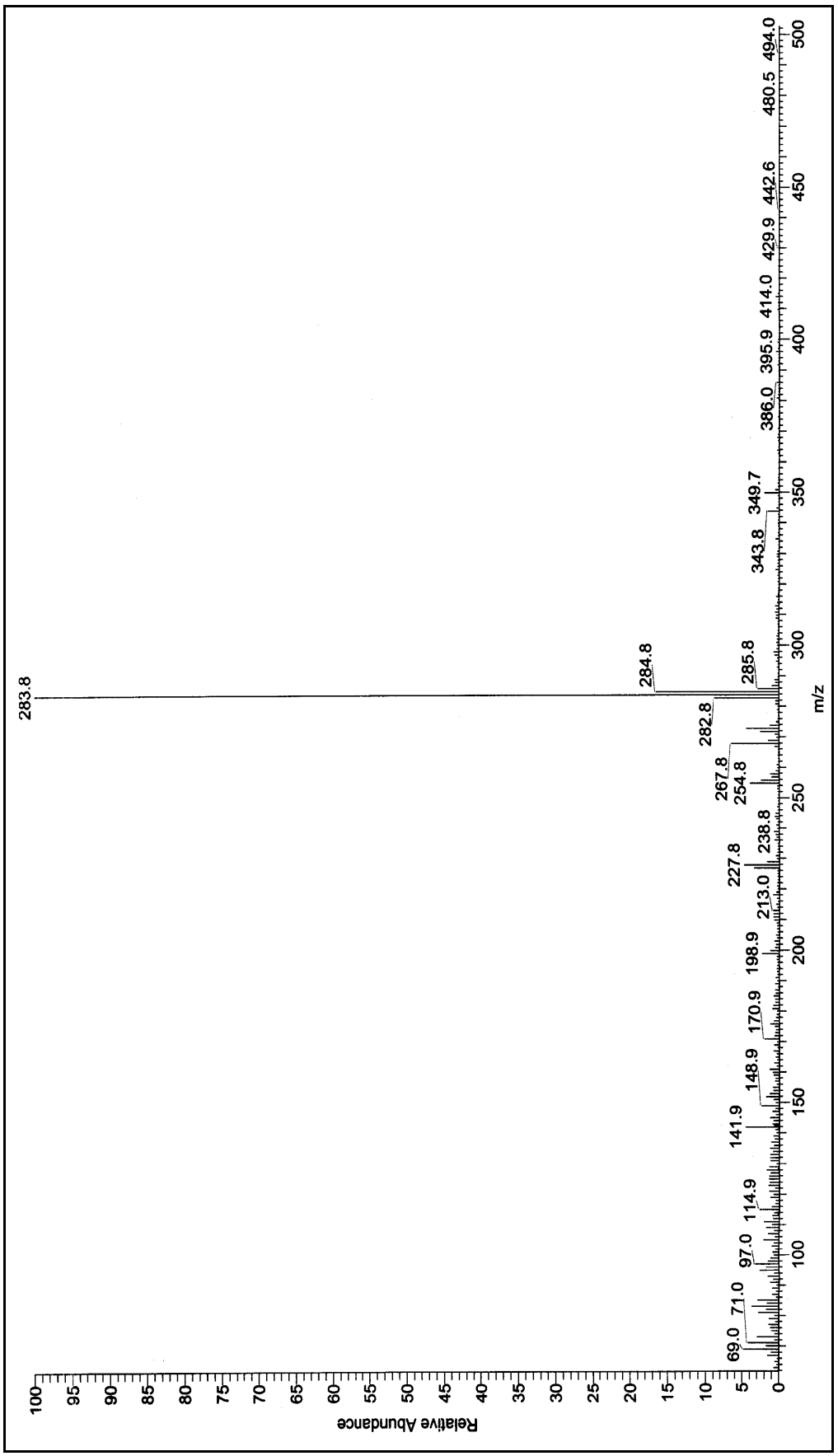


Figure 41 Mass spectrum of compound **SK10**

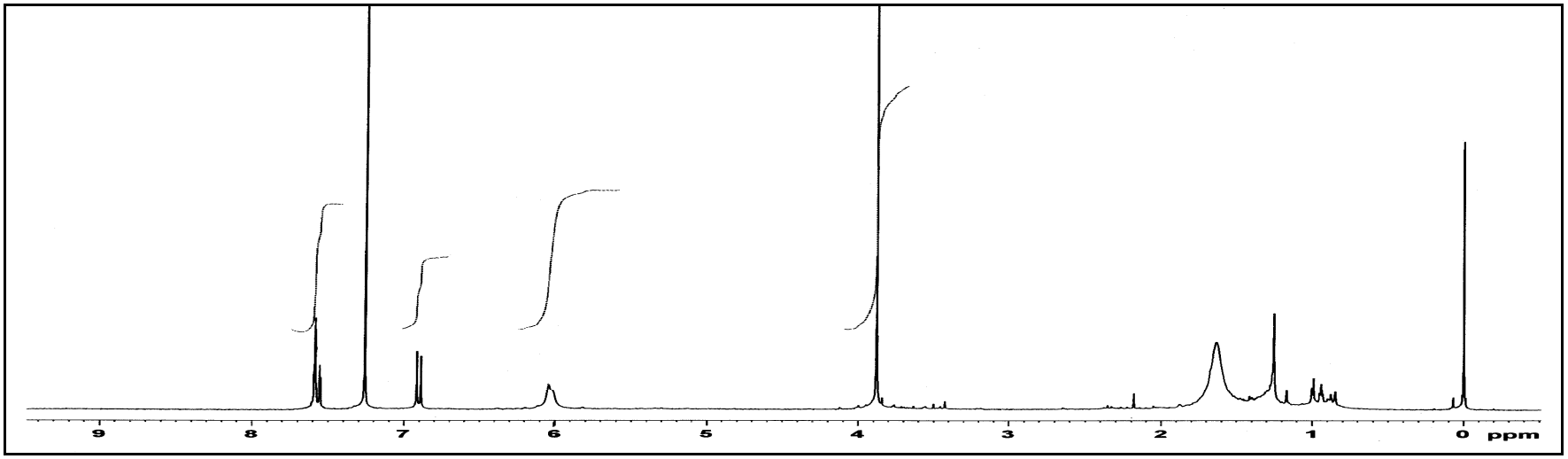


Figure 42 ^{1}H NMR (300 MHz) ($CDCl_3$) spectrum of compound **SK17**

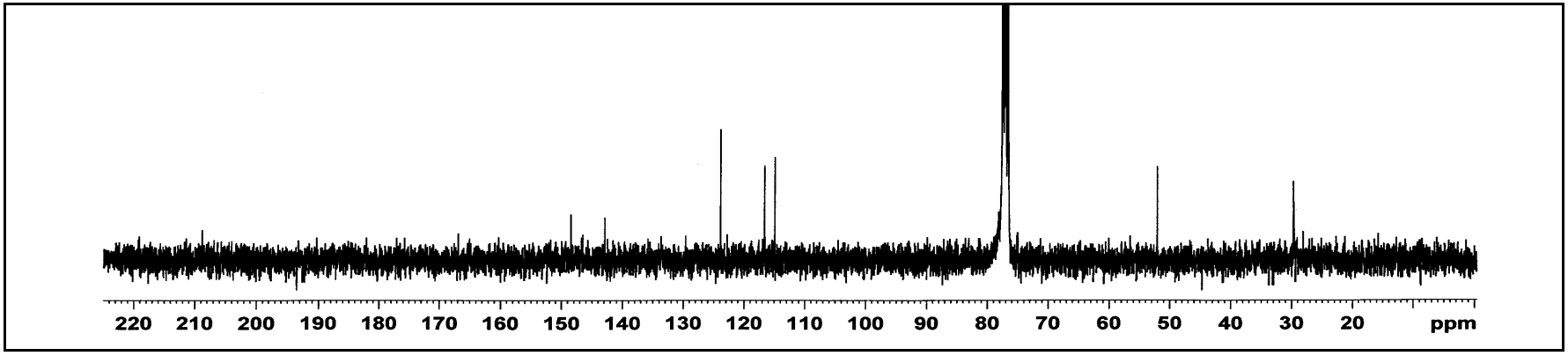


Figure 43 ^{13}C NMR (75 MHz) ($CDCl_3$) spectrum of compound **SK17**

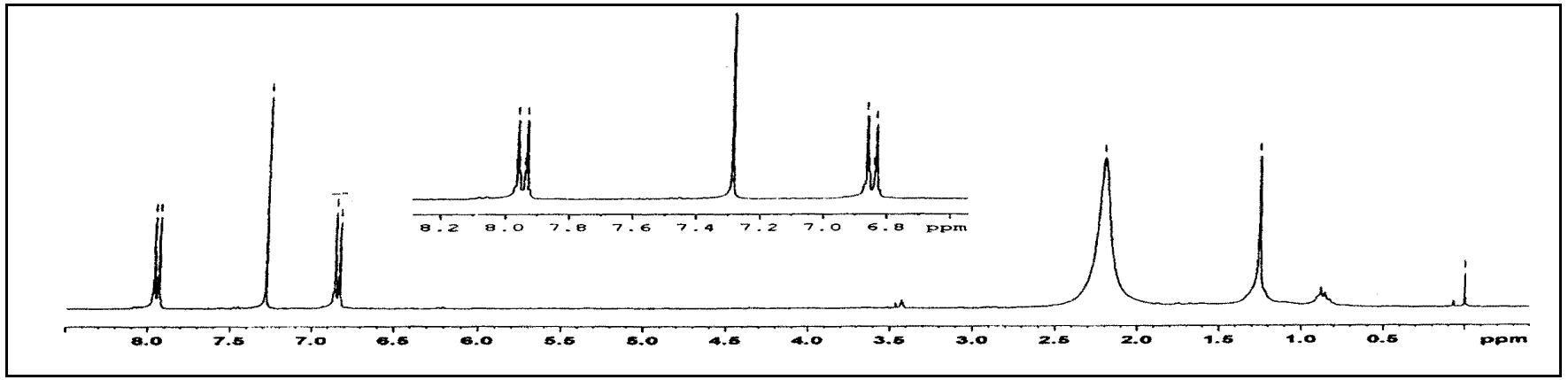


Figure 44 ^{1}H NMR (300 MHz) ($CDCl_3$+CD_3OD) spectrum of compound **SK7**

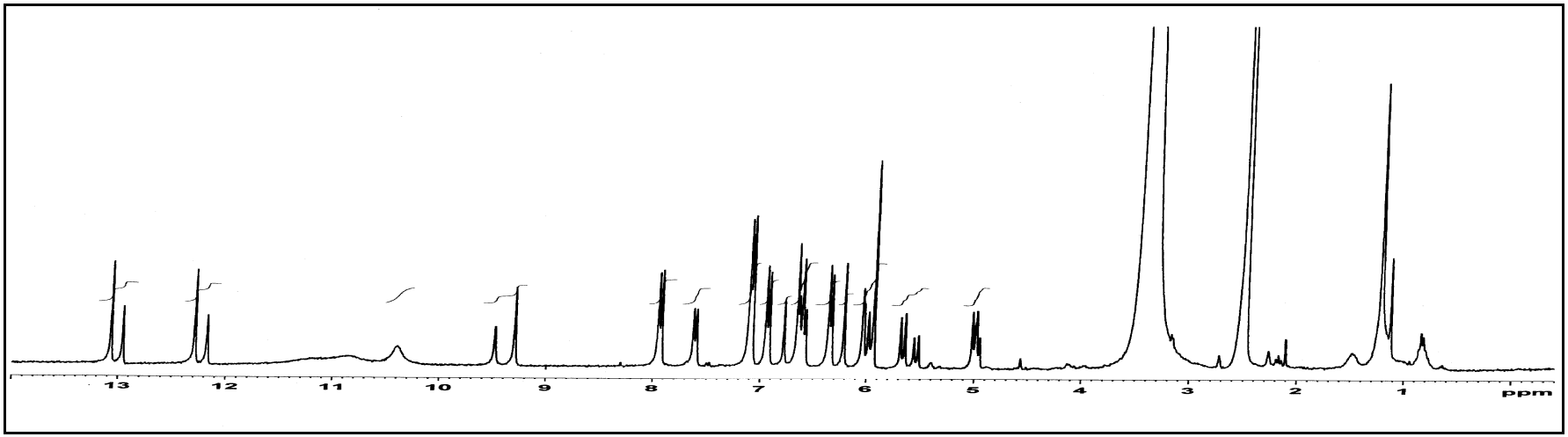


Figure 45 ^{1}H NMR (300 MHz) (DMSO-d_6) spectrum of compound **SK6**

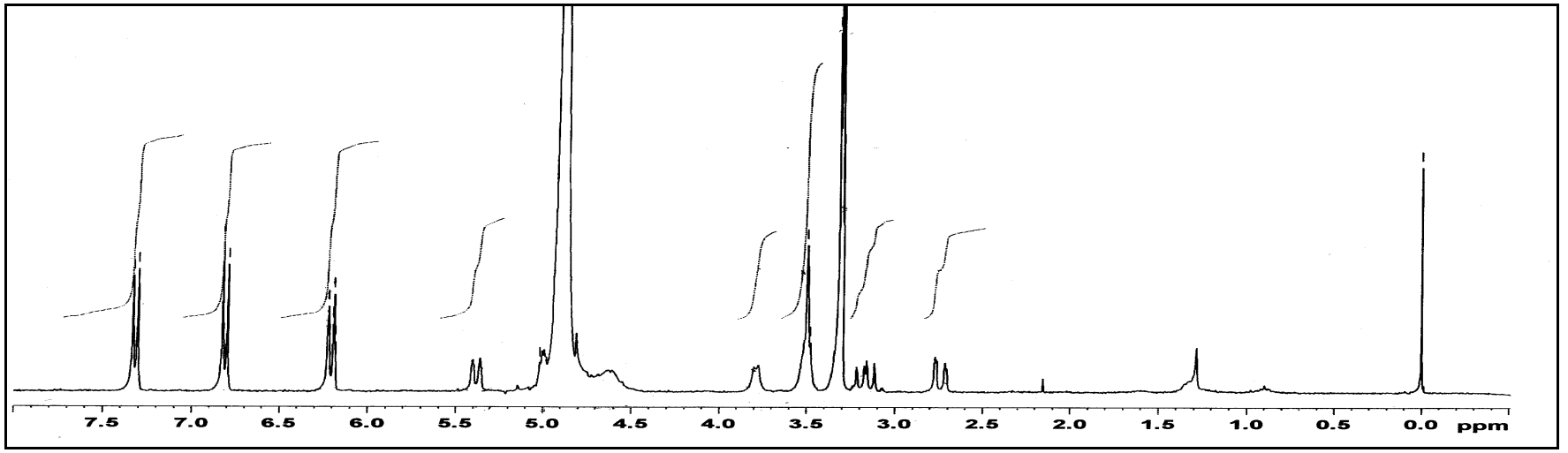


Figure 46 ^{1}H NMR (300 MHz) (CD_3OD) spectrum of compound **SK23**

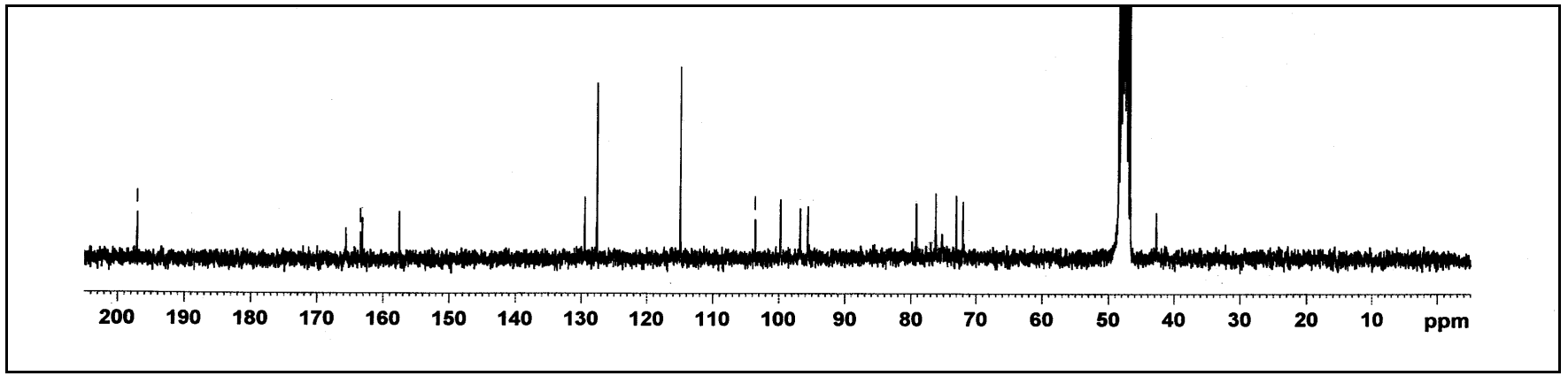


Figure 47 ^{13}C NMR (75 MHz) (CD_3OD) spectrum of compound **SK23**

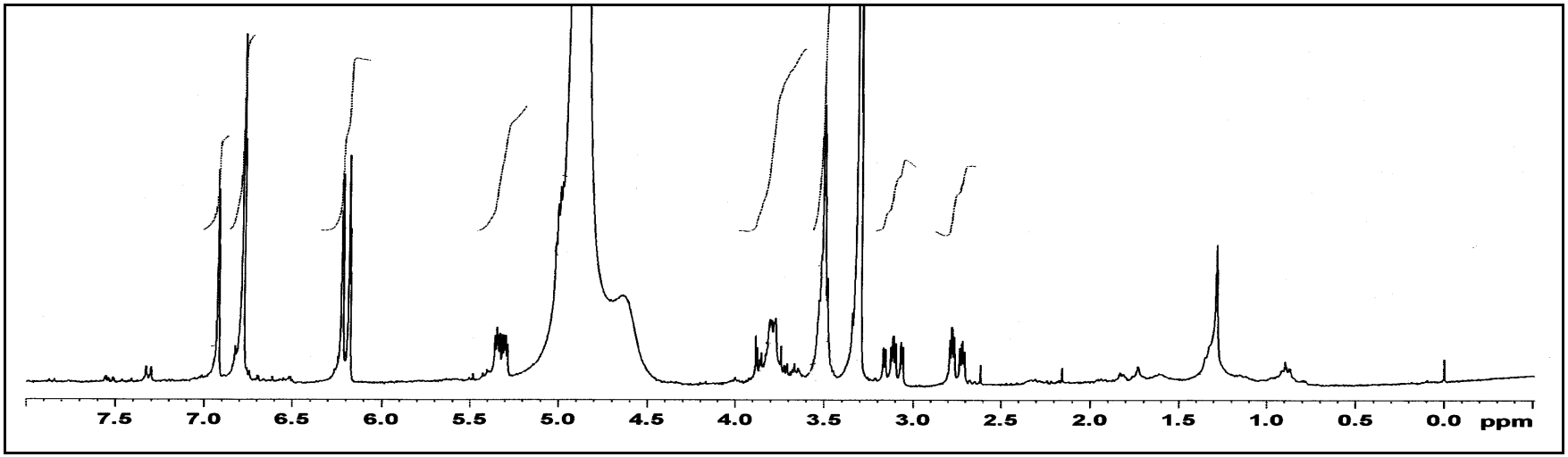


Figure 48 ^{1}H NMR (300 MHz) (CD_3OD) spectrum of compound **SK24**

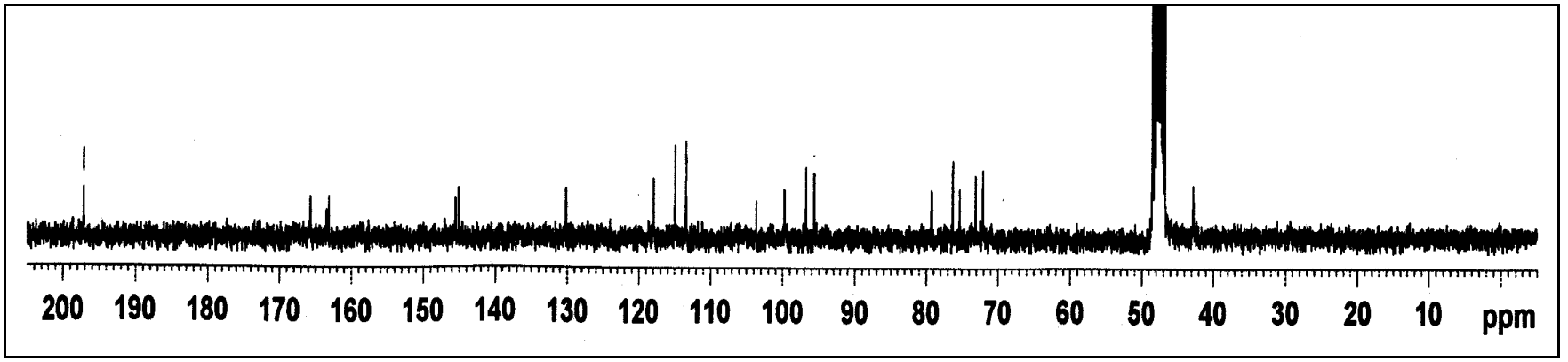


Figure 49 ^{13}C NMR (75 MHz) (CD_3OD) spectrum of compound **SK24**

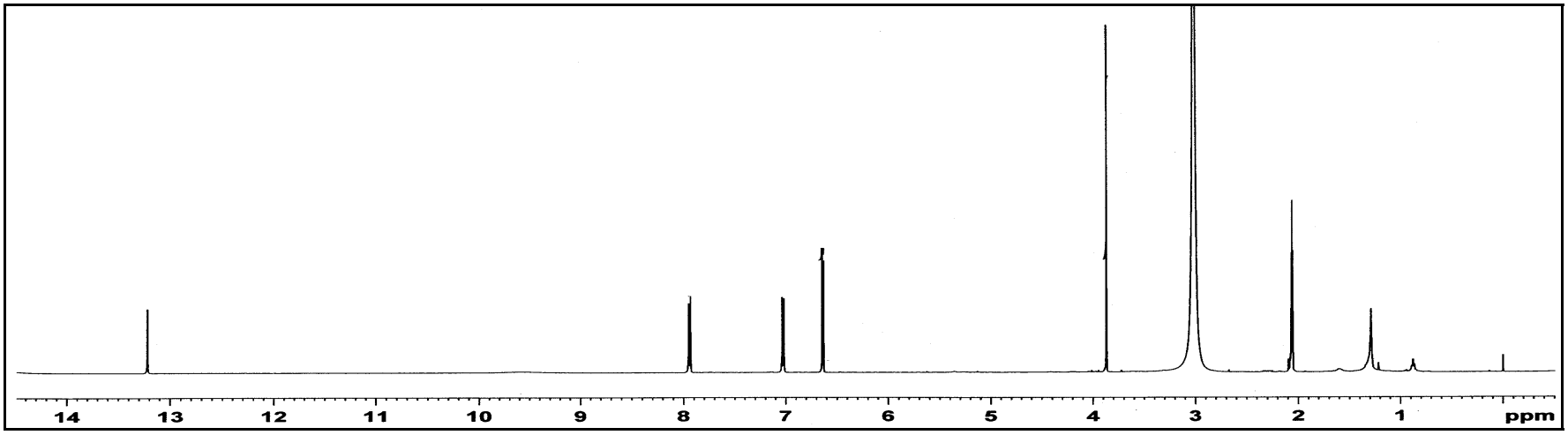


Figure 50 ^{1}H NMR (500 MHz) (Acetone-d_6) spectrum of compound **SK15**

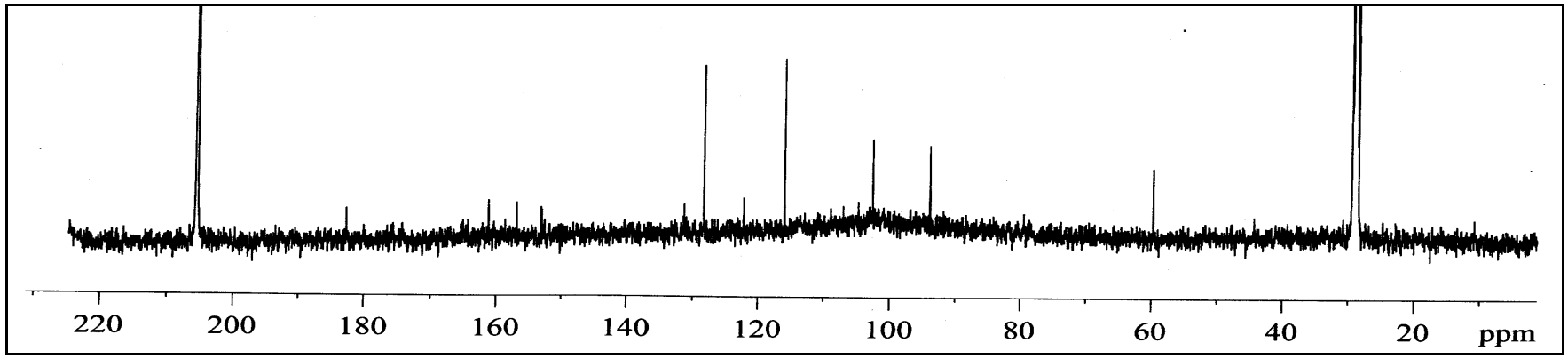


Figure 51 ^{13}C NMR (125 MHz) (Acetone-d_6) spectrum of compound **SK15**

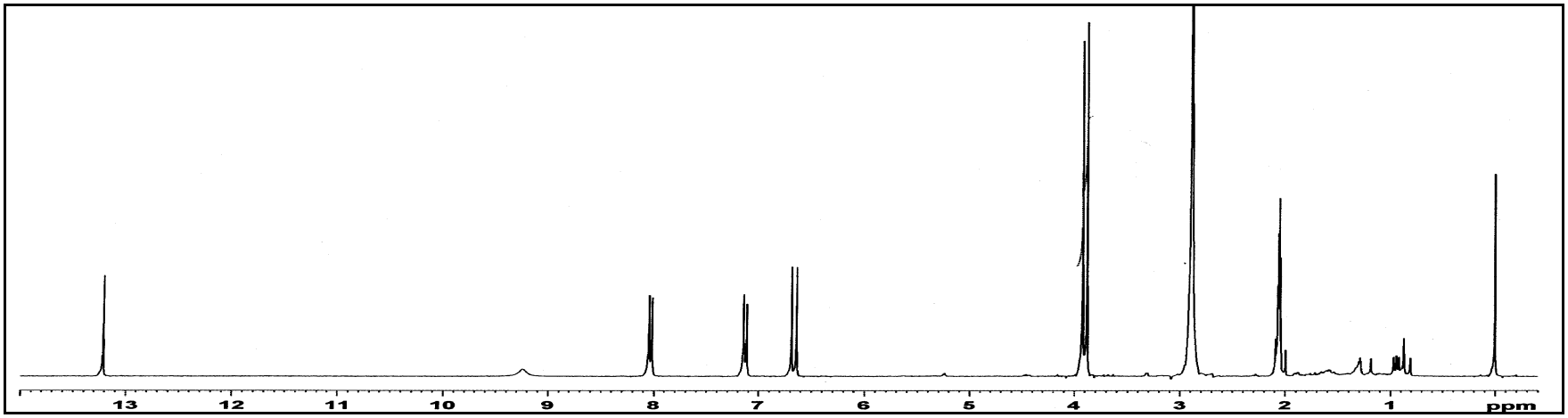


Figure 52 ^{1}H NMR (300 MHz) (Acetone-d_6) spectrum of compound **SK14**

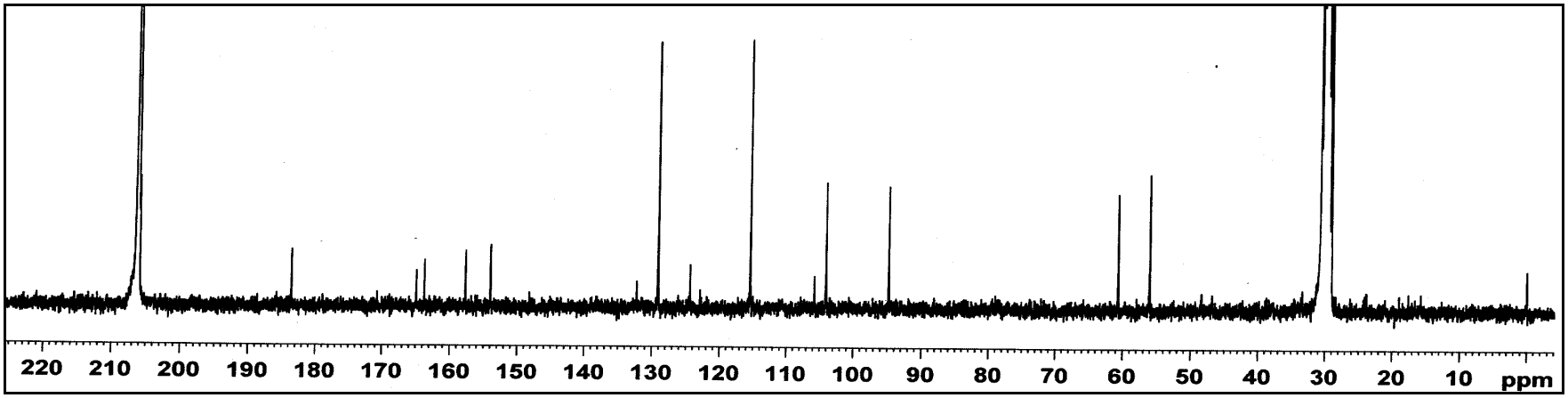


Figure 53 ^{13}C NMR (75 MHz) (Acetone-d_6) spectrum of compound **SK14**

VITAE

Name Mr. Saranyoo Klaiklay

Student ID 5010220132

Educational Attainment

Degree	**Name of Institution**	**Year of Graduation**
B.Sc (2^{nd} Hons.) (Chemistry)	Prince of Songkla University	2007

Scholarship Awards during Enrolment

The Center for Innovation in Chemistry (PERCH-CIC)

List of Publication and Proceedings

Proceeding

1. Klaiklay, S., Sukpondma, Y. and Rukachaisirikul, V. 2007. Chemical constituents from the twigs of *Garcinia hombroniana*. Proceeding of the 33^{rd} Congress on Science and Technology of Thailand. Walailak University, October 18-20, 2007. pp. 171.
2. Klaiklay, S., Sukpondma, Y. and Rukachaisirikul, V. 2008. Chemical constituents from the twigs of *Garcinia hombroniana*. Proceeding of the 6^{th} Regional IMT-GT Uninet Conference. The Gurney Resort Hotel & Residences Penang, Malaysia, August 28-30, 2008. pp. 534-535.
3. Klaiklay, S., Sukpondma, Y. and Rukachaisirikul, V. 2008. Flavonoids from the roots of *Clerodendrum petasites* S. Moore. Proceeding of the 10^{th} the National Graduate Research Conference. Sukhothai Thammathirat Open University, September 11-12, 2008. pp. 237.
4. Klaiklay, S., Sukpondma, Y. and Rukachaisirikul, V. 2007. Chemical constituents from the twigs of *Garcinia hombroniana*. Proceeding of the international congress for innovation in chemistry (PERCH-CIC Congress VI). Jomtein Palm Beach Resort Pattaya, Chonburi, May 3-6, 2009. pp. 236.