Chemical Constituents from *Cratoxylum cochinchinense* and *Cratoxylum formosum* ssp. *pruniflorum* and Their Biological Activities

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Thesis Title   Chemical Constituents from *Cratoxylum cochinchinense*

        and *Cratoxylum formosum* ssp. *pruniflorum* and Their

        Biological Activities

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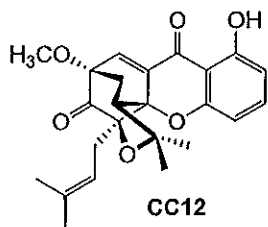|	
ชื่อวิทยานิพนธ์	องค์ประกอบทางเคมีจากต้นติ้วเกลี้ยงและต้นติ้วขนและฤทธิ์ทางชีวภาพ
ผู้เขียน	นายณวงศ์ บุนนาค
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บทคัดย่อ

ส่วนที่ 1   การศึกษาองค์ประกอบทางเคมีจากส่วนยางและผลอ่อนของต้นติ้วเกลี้ยง

การศึกษาองค์ประกอบทางเคมีจากส่วนสกัดหยาบไดคลอโรมีเทนจากส่วนยางของต้นติ้วเกลี้ยง นำไปสู่การแยกพบสารใหม่ในกลุ่มของแซนโทนทั้งหมด 3 สาร (CC1-CC3) และสารที่มีการรายงาน มาแล้วในกลุ่มของแซนโทน 9 สาร (CC4-CC12) สารในกลุ่มไตรเทอร์พีน 1 สาร (CC21) และสารผสม ในกลุ่มสเตอรอยด์ 2 สาร (CC22 และ CC23) จากการแยกสารในส่วนสกัดไดคลอโรมีเทนจากผลอ่อน ของต้นติ้วเกลี้ยงโดยวิธีทางโครมาโทกราฟฟี พบสารใหม่ในกลุ่มของแซนโทน 1 ส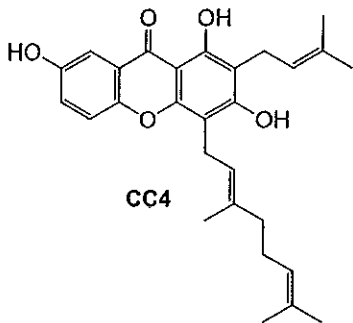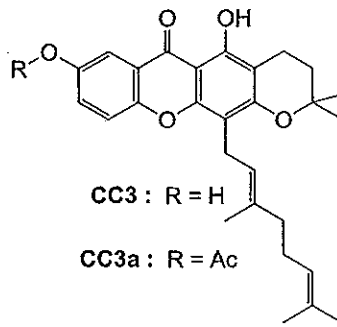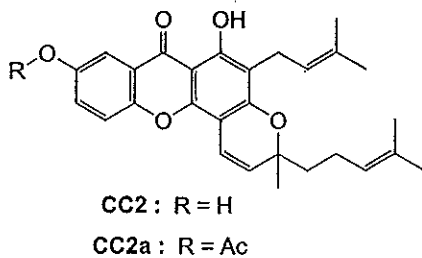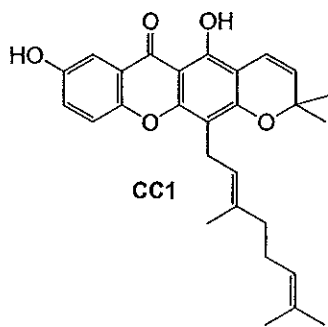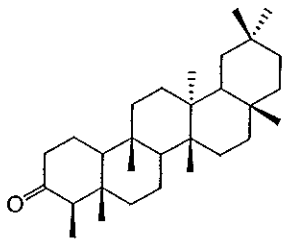าร (CC13) และสารที่มี การรายงานมาแล้วในกลุ่มของแซนโทน 2 สาร นอกจากนั้นสารอนุพันธ์ใหม่ในรูปอะซิเทดทั้งหมด 4 สาร (CC16 และ CC17) และ (CC18 และ CC19) ได้ถูกสังเคราะห์จากสารประกอบ CC14 และ สารประกอบ CC15 ตามลำดับ เพื่อศึกษา และเปรียบเทียบฤทธิ์ยับยั้งการต้านเชื้อแบคทีเรีย และเชื้อรากับ สารตั้งต้นเดิม โครงสร้างสารประกอบทั้งหมดทั้งในส่วนที่แยกจากต้นติ้วเกลี้ยงและที่ถูกสังเคราะห์ขึ้นใหม่ วิเคราะห์โดยใช้วิธีทางสเปกโทรสโกปี เทคนิคการเลี้ยวเบนของรังสีเอกซ์บนผลึกเดี่ยวสนับสนุนโครงสร้าง สารประกอบ CC1 CC6 CC12 อนุพันธุ์ในรูปไดโบรซิเลตของ CC14 (CC20) และอนุพันธ์ในรูป มอนออะซิเทดของ CC15 (CC18)

สารทั้งหมดได้ถูกนำไปทดสอบฤทธิ์ยับยั้งการต้านเชื้อแบคทีเรีย และเชื้อรา จากข้อมูลการทดสอบ ฤทธิ์ทางชีวภาพแสดงให้เห็นว่า สารประกอบ CC4-CC6 และ CC13-CC20 ออกฤทธิ์ยับยั้งการต้านเชื้อ แบคทีเรียโดยเฉพาะการต้านเชื้อแบคทีเรียชนิดซูโดโมแนส ออรูจิโนซา ด้วยค่า MIC เท่ากับ 4.7 μg/mL นอกจากนั้น สารประกอบ CC9 และ CC10 แสดงฤทธิ์ยับยั้งการต้านเชื้อราชนิดแคนดิดา อัลบิแคน ด้วยค่า MIC เท่ากับ 2.4 และ 4.7 μg/mL ตามลำดับ

CC1

CC2 : R = H

CC2a : R = Ac

CC3 : R = H

CC3a : R = Ac

CC4

CC5

CC6

CC7

CC8: R₁ = OCH₃   R₂ = OCH₃

CC9: R₁ = OH   R₂ = OCH₃

CC10: R = CH₃

CC11: R =

CC12

CC20

CC13: R₁ = Ac   R₂ = H

CC14: R₁ = H   R₂ = H

CC16: R₁ = H   R₂ = Ac

CC17: R₁ = Ac   R₂ = Ac

CC15: R₁ = H   R₂ = H

CC18: R₁ = H   R₂ = Ac

CC19: R₁ = Ac   R₂ = Ac
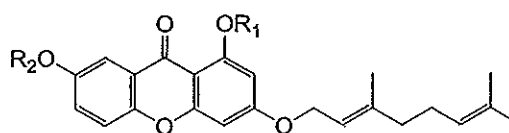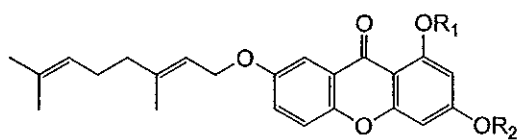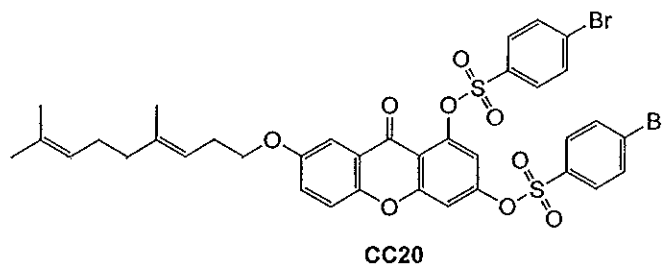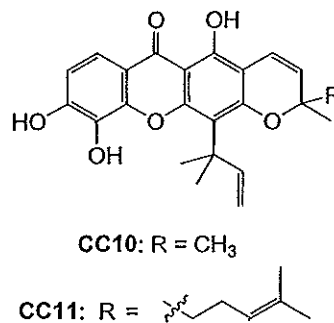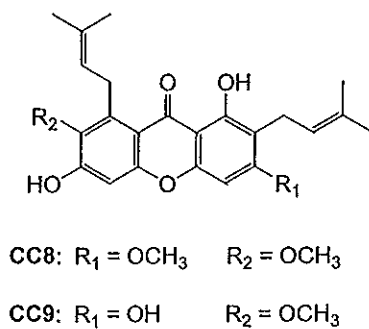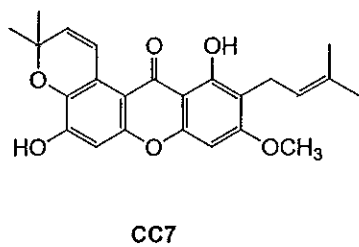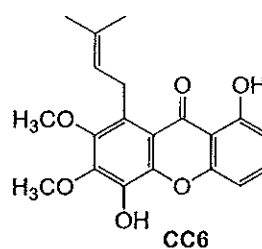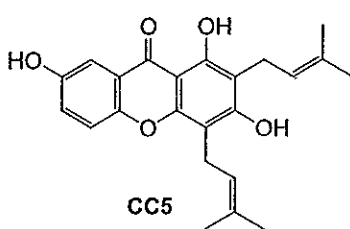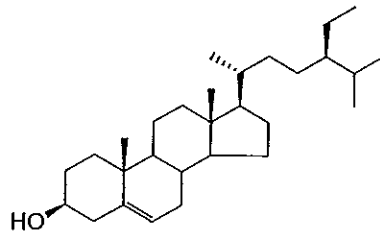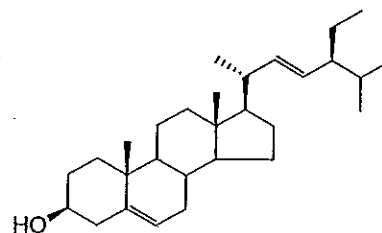
CC21   CC22   CC23

ส่วนที่ 2 *การศึกษาองค์ประกอบทางเคมีจากส่วนรากและผลอ่อนของต้นติ้วขน*

   การแยกสารในส่วนสกัดไดคลอโรมีเทนจากส่วนรากของต้นติ้วขนโดยใช้วิธีทางคอลัมน์โครมาโทกราฟีได้สารใหม่ในกลุ่มของแซนโทน 2 สาร (CP1 และ CP2) รวมทั้งสารที่มีการรายงานมาแล้วในกลุ่มของแซนโทน 15 สาร (CP3-CP17) และสารในกลุ่มแอนทราควิโนน 2 สาร (CP18 และ CP19) จากการแยกสารในส่วนสกัดหยาบไดคลอโรมีเทนจากผลอ่อนของต้นติ้วขน พบสารใหม่ในกลุ่มของแซนโทน 3 สาร (CP20-CP22) สารใหม่ในกลุ่มของแซนโทโนลิกนอยด์ 1 สาร (CP23) และสารที่มีการรายงานมาแล้วอีก 8 สาร ซึ่งเป็นสารในกลุ่มของแซนโทน 3 สาร (CP24-CP26) สารในกลุ่มของฟลาโวนอยด์ 1 สาร (CP27) สารผสมในกลุ่มไตรเทอร์พีน 3 สาร (CP28-CP30) และสารในกลุ่มไตรเทอร์พีน 1 สาร (CP31) โครงสร้างสารประกอบทั้งหมดวิเคราะห์โดยใช้วิธีทางสเปกโทรสโกปีและการวิเคราะห์โครงสร้างด้วยเทคนิคการเลี้ยวเบนของรังสีเอกซ์บนผลึกเดี่ยวสนันสนุนโครงสร้างสารประกอบ CP6 CP11 CP20 CP21 CP25 และ CP26

   สารทั้งหมดได้ถูกนำไปทดสอบฤทธิ์ยับยั้งการต้านเชื้อแบคทีเรีย เชื้อรา และฤทธิ์การต้านการอักเสบโดยวิธีการยับยั้งไนตริกออกไซด์ จากข้อมูลการทดสอบฤทธิ์ทางชีวภาพแสดงให้เห็นว่าสารประกอบ CP26 แสดงฤทธิ์ยับยั้งการต้านเชื้อแบคทีเรียทั้งชนิดแกรมบวก และแกรมลบ ด้วยค่า MIC เท่ากับ 4.7 μg/mL สารประกอบ CP8 แสดงฤทธิ์ยับยั้งการต้านเชื้อแบคทีเรียชนิดบาซิลลัส ซับทิลิส ด้วยค่า MIC เท่ากับ 4.7 $\mu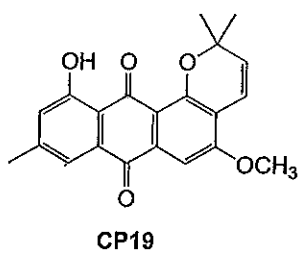$g/mL และทั้งยังออกฤทธิ์ยับยั้งการต้านเชื้อแบคทีเรียชนิดแสตบไพโรคอคคัส ออริอัส และชนิดเอนทีโรคอคคัส เฟคาลิส ด้วยค่า MIC เท่ากับ 9.37 μg/mL นอกจากนั้น สารผสมระหว่างสารประกอบ CC10 และ CP10 ในอัตราส่วน 1 ต่อ 1 แสดงฤทธิ์ยับยั้งการต้านเชื้อแบคทีเรียทั้งชนิด แกรมบวก และ แกรมลบ สารประกอบ CP21 และ CP26 แสดงฤทธิ์ต้านการอักเสบที่ดีโดยการยับยั้ง ไนตริก ออกไซด์ ด้วยค่า IC$_{50}$ เท่ากับ 4.4 μM และ 4.3 μM ตามลำดับ

CP1

CP2

CP3

CP4

CP5

CP6 : R = -CH₃
CP16 : R = -OH

CP7

CP8

CP9

CP10

CP11

CP12 : R = isoprenyl
CP13 : R = ⸙⸙⸙OH
CP14 : R = ⸙⸙⸙OCH₃

CP15

CP17

CP18

CP19

CP20

CP21 : R = H
CP21a : R = CH₃

CP22

CP23

CP24

CP25

CP26

CP27

CP28

CP29 : R$_1$ = CH$_3$ R$_2$ = H
CP30 : R$_1$ = H R$_2$ = CH$_3$

CP31
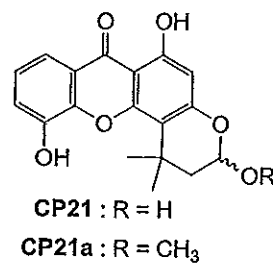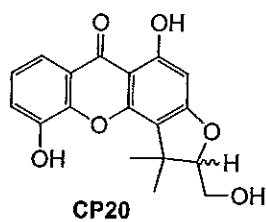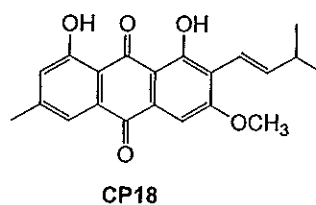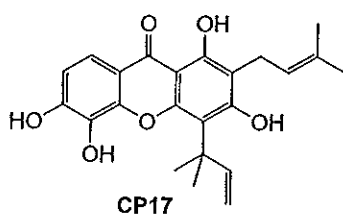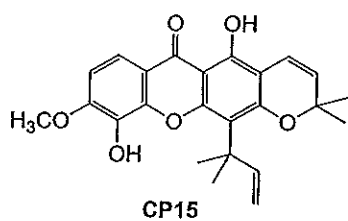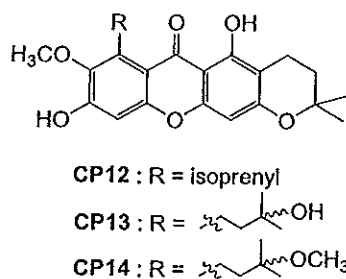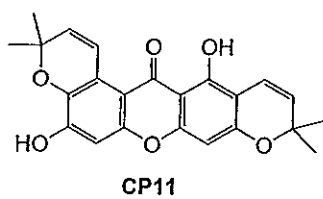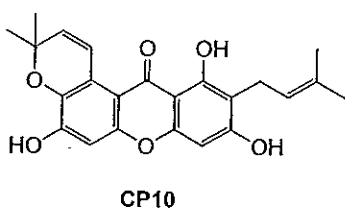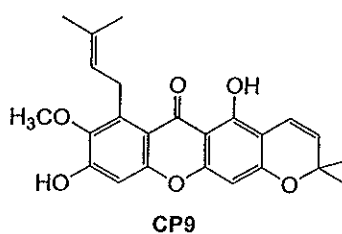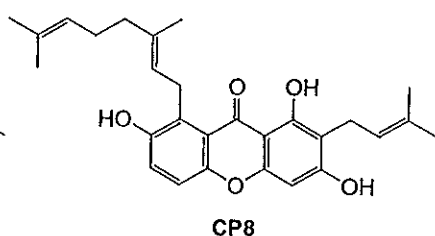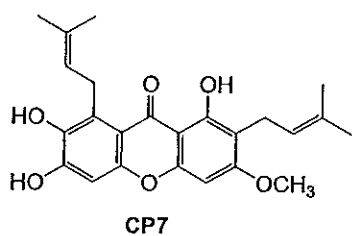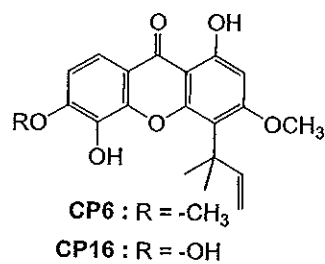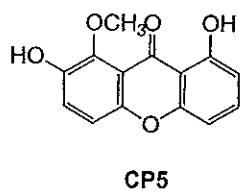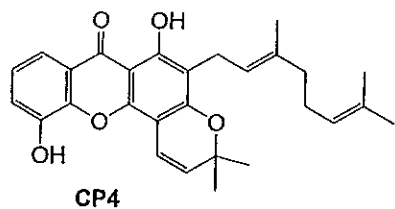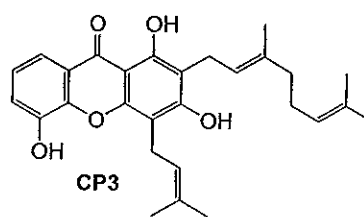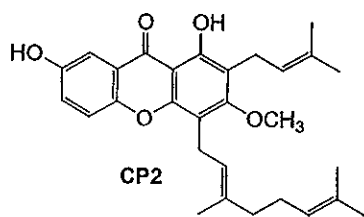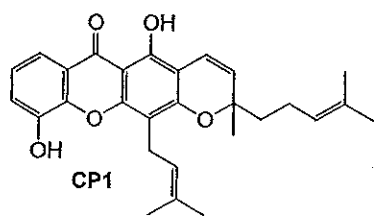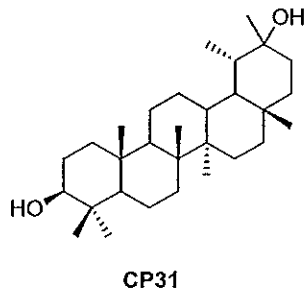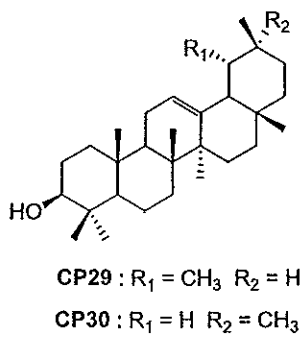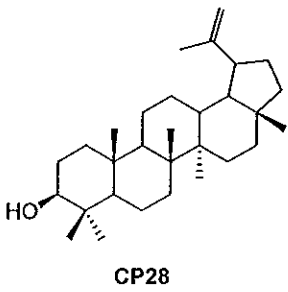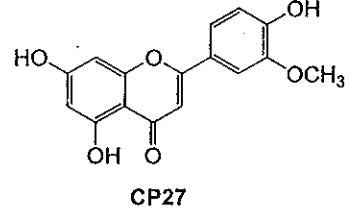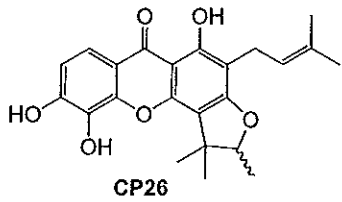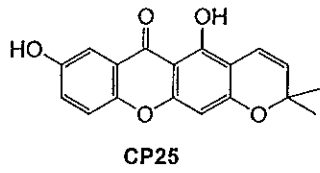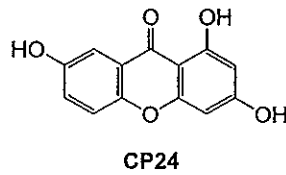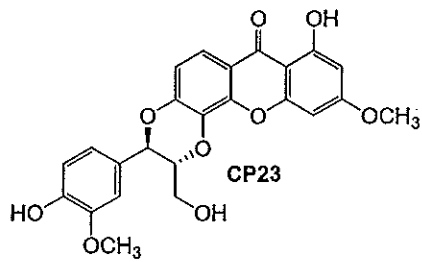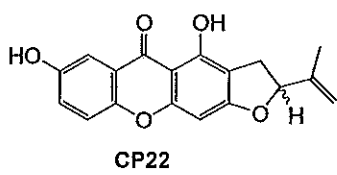
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Author	Mr. Nawong Boonnak
Major Program	Organic Chemistry
Academic Year	2010

ABSTRACT

Part I   Chemical Investigation of the Resin and Green Fruits of Cratoxylum cochinchinense

The chemical investigation of the crude CH_2Cl_2 extract from the resin of *C. cochinchinense* have led to the isolation of three new xanthones (**CC1-CC3**), together with nine known xanthones (**CC4-CC12**), a known triterpene (**CC21**) and a known mixture of two steroids (**CC22** and **CC23**). The CH_2Cl_2 extract of the green fruits of *C. cochinchinense* was subjected to chromatographic separation to give a new xanthone (**CC13**) along with two known xanthones (**CC14** and **CC15**). In addition, four new acetylated derivatives (**CC16** and **CC17**) and (**CC18** and **CC19**) were partially synthesized from compounds **CC14** and **CC15**, respectively to study and compare their antibacterial and antifungal activities with their precursors. The structures of all isolated and partial synthesized compounds were elucidated by spectroscopic methods. Moreover, X-ray diffraction analysis also supported the chemical structures of compounds **CC1, CC6, CC12,** a dibrosylate derivative of **CC14** (**CC20**) and a monoacetate derivative of **CC15** (**CC18**).

All of the compounds were further evaluated for their antibacterial and antifungal activities. Compounds **CC4-CC6** and **CC13-CC20** showed strong antibacterial activity specifically against *Pseudomonas aeruginosa* with MIC value of 4.7 μg/mL. Moreover, compounds **CC9** and **CC10** exhibited strong antifungal acitivity against *Candida albicans* with MIC values of 2.4 and 4.7 μg/mL, respectively.

CC1

CC2 : R = H
CC2a : R = Ac

CC3 : R = H
CC3a : R = Ac

CC4

CC5

CC6

CC7

CC8: R$_1$ = OCH$_3$ R$_2$ = OCH$_3$
CC9: R$_1$ = OH   R$_2$ = OCH$_3$

CC10: R = CH$_3$

CC11: R =

CC12

CC20

CC13: R$_1$ = Ac   R$_2$ = H
CC14: R$_1$ = H   R$_2$ = H
CC16: R$_1$ = H   R$_2$ = Ac
CC17: R$_1$ = Ac   R$_2$ = Ac

CC15: R$_1$ = H   R$_2$ = H
CC18: R$_1$ = H   R$_2$ = Ac
CC19: R$_1$ = Ac   R$_2$ = Ac
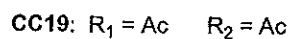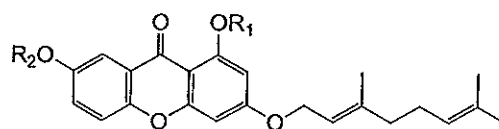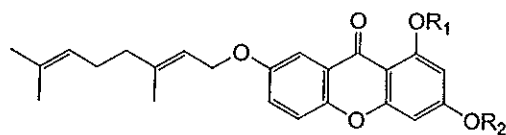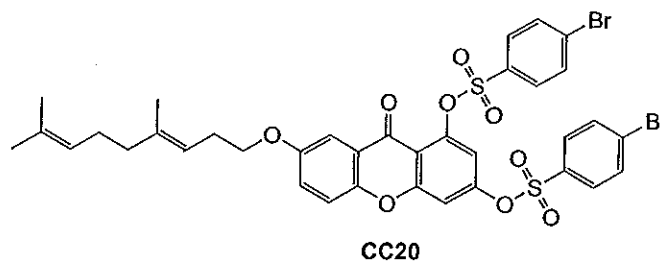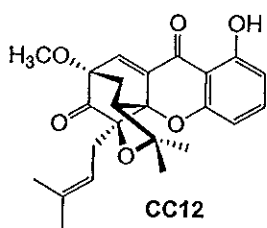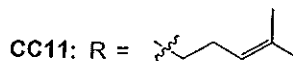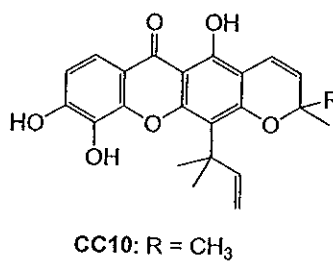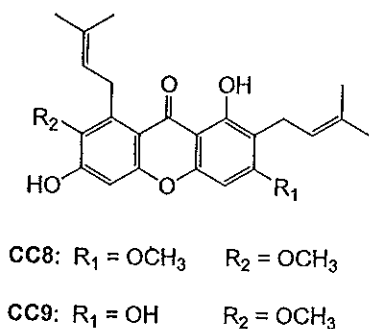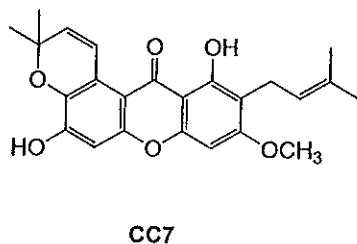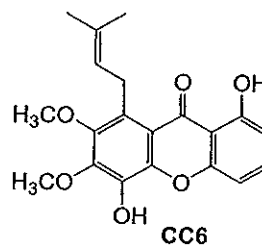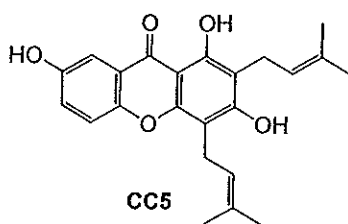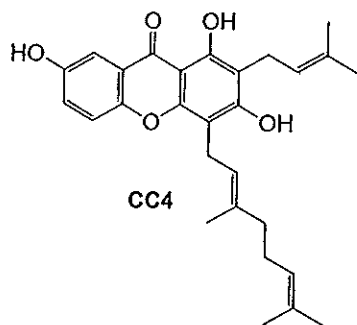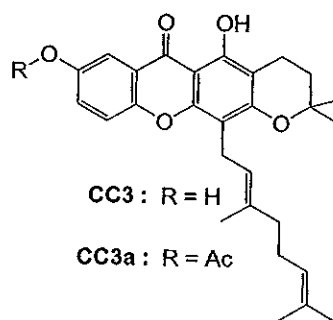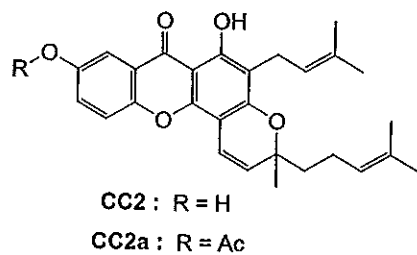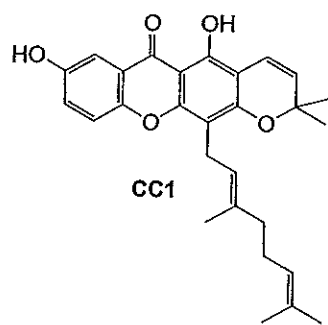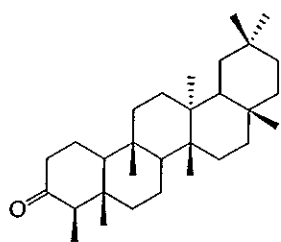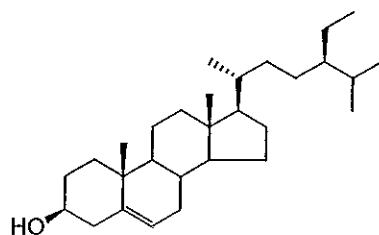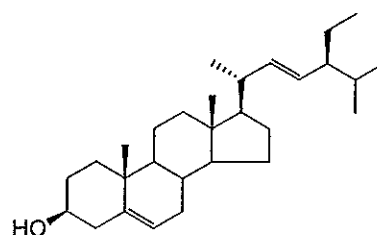
CC21　　　　CC22　　　　CC23

Part II Chemical Investigation of the Roots and Green Fruits of
Cratoxylum formosum ssp. pruniflorum

The column chromatographic separation of the CH_2Cl_2 extract from the roots of C. formosum ssp. pruniflorum afforded two new xanthones (**CP1** and **CP2**) as well as fifteen known xanthones (**CP3-CP17**) and two known anthraquinones (**CP18** and **CP19**). The crude CH_2Cl_2 extract of the green fruits gave three new xanthones (**CP20-CP22**), a new xanthonolignoid (**CP23**) along with eight known compounds, including three xanthones (**CP24-CP26**), a flavonoid (**CP27**), a mixture of three triterpenes (**CP28-CP30**) and a triterpene (**CP31**). Their structures were elucidated by spectroscopic methods. In addition, X-ray diffraction analysis supported the structures of compounds **CP6, CP11, CP20, CP21, CP25** and **CP26**.

Their antibacterial, antifungal and nitric oxide inhibitory activities were also evaluated as well. Compound **CP26** showed strong antibacterial activity against both Gram-positive and Gram-negative bacteria with MIC value of 4.7 μg/mL. Compound **CP8** showed strong activity against Bacillus subtilis with MIC value of 4.7 μg/mL and also showed moderate activity against Staphylococcus aureus and Enterococcus faecalis with MIC value of 9.37 μg/mL. Moreover, a 1:1 mixture of compounds **CC10** and **CP10** showed strong antibacterial activity against all Gram-bacteria tested. In addition, compounds **CP21** and **CP26** showed potent nitric oxide inhibitory with IC_{50} values of 4.4 μM and 4.3 μM, respectively.

CP1

CP2

CP3

CP4

CP5

CP6 : R = -CH₃

CP16 : R = -OH

CP7

CP8

CP9

CP10

CP11

CP12 : R = isoprenyl

CP13 : R =

CP14 : R =

CP15

CP17

CP18

CP19

CP20

CP21 : R = H

CP21a : R = CH₃

CP22

CP23

CP24

CP25

CP26

CP27

CP28

CP29 : R₁ = CH₃ R₂ = H
CP30 : R₁ = H R₂ = CH₃

CP31

ACKNOWLEDGEMENT

THE RELEVANCE OF THE RESEARCH WORK TO THAILAND

This research is a basic research on the evaluation for utilization of Thai medicinal plants as sources of bioactive compounds. Plants in the genus *Cratoxylum* of Guttiferae family have been used as traditional medicine in Thailand for a long time. In this research, the two species in *Cratoxylum* genus, including *C. cochinchinense* and *C. formosum* ssp. *pruniflorum*, were investigated for their chemical constituents in order to search for biologically active compounds. The structures of all isolated compounds from *C. cochichinense* and *C. formosum* ssp. *pruniflorum* were characterized on the basis of spectroscopic and X-ray analyses. Moreover, the isolated compounds were further evaluated for their antibacterial, antifungal and nitric oxide inhibitory activities. The results from this thesis showed that some of the isolated compounds exhibited interesting biological activity, which was useful for natural product chemist and/or pharmacologist who studies the drug design and molecular modeling for further developing and/or enhancing activity of these compounds.

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LIST OF FIGURES (*continued*)

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ABBREVIATIONS AND SYMBOLS

s	=	*singlet*
d	=	*doublet*
t	=	*triplet*
q	=	*quartet*
m	=	*multiplet*
dd	=	*doublet of doublet*
dt	=	*doublet of triplet*
br s	=	*broad singlet*
g	=	Gram
nm	=	Nanometer
mp	=	Melting point
cm^{-1}	=	Reciprocol centimeter (wave number)
δ	=	Chemical shift relative to TMS
J	=	Coupling constant
$[\alpha]_D$	=	Specific rotation
λ_{max}	=	Maximum wavelength
ν	=	Absorption frequencies
ε	=	Molar extinction coefficient
m/z	=	A value of mass divided by charge
°C	=	Degree celcius
MHz	=	Megahertz
ppm	=	Part per million
c	=	Concentration
IR	=	Infrared
UV	=	Ultraviolet-Visible
ESI-TOF MS	=	Electrospray Ionization Time-of-Flight Mass Spectroscopy
CIMS	=	Chemical Impact Mass Spectroscopy
HRCIMS	=	High Resolution Chemical Impact Mass Spectroscopy
EIMS	=	Electron Impact Mass Spectroscopy

ABBREVIATIONS AND SYMBOLS (*continued*)

HREIMS	=	High Resolution Electron Impact Mass Spectroscopy
NMR	=	Nuclear Magnetic Resonance
2D NMR	=	Two Dimensional Nuclear Magnetic Resonance
COSY	=	Correlation Spectroscopy
DEPT	=	Distortionless Enhancement by Polarization Transfer
HMBC	=	Heteronuclear Multiple Bond Correlation
HMQC	=	Heteronuclear Multiple Quantum Coherence
NOE	=	Nuclear Overhauser Effect
NOESY	=	Nuclear Overhauser Effect Correlation Spectroscopy
CC	=	Column Chromatography
QCC	=	Quick Column Chromatography
PLC	=	Preparative Thin Layer Chromatography
DCM	=	Dichloromethane
TMS	=	Tetramethylsilane
$CDCl_3$	=	Deuterochlroform
CD_3OD	=	Deuteromethanol

CHAPTER 1

INTRODUCTION

1.1 Introduction

The bioactive natural products are mostly secondary metabolites, which were produced by living organisms such as plants, microbes and fungi. In some cases, these bioactive secondary metabolites are produced by the organism to help protect itself within its own environmental niche. In other cases, the secondary metabolites also showed several potent biological activities. From these reasons, the herbal plants have been used as natural medicines for treatment of the infectious diseases for a long time. However, the utilization of the whole plants or other crude preparations for therapeutic or experimental reasons can have several drawbacks including:

a) Variation in the amount of the active constituents with geographic areas, from one season to another, with different plant parts and morphology, and with climatic and ecological conditions.

b) Co-occurrence of undesirable compounds causing synergistic, antagonistic, or other undesirable, and possibly unpredictable, modulations of the bioactivity.

c) Changes or losses of bioactivity due to variability in collection, storage, and preparation of the raw materials.

Thus, the isolation of natural products that have biological activity toward organisms other than the sources has several advantages including the following:

a) Pure bioactive compounds can be administered in reproducible, accurate doses with obvious benefits from an experimental or therapeutic aspect.

· b) It can lead to the development of analytical assays for particular compounds or for classes of compounds. This is necessary, for example, in the screening of plants for potential toxicity and for quality control of therapeutic formulations or food for human or animal consumption.

c) It permits the structural determination of bioactive compounds, which may enable the production of synthetic material, incorporation of structural modifications, and rationalization of mechanisms of action. This in turn will lead to reduced dependency on plants, for example, as sources of bioactive compounds and will enable investigations of structure/activity relationships, facilitating the development of new compounds with similar or more desirable bioactivities.

1.2 Chemistry of xanthones

Xanthones or xanthen-9H-ones are phenolic compounds, which are found in some higher plant families, fungi and lichens (Peres *et al.*, 2000; Vieira and Kijjoa, 2005), and they comprise an important class of oxygenated heterocycles. The xanthone nucleus is known as 9-xanthenone or dibenzo-γ-pyrone and it is symmetric (**Figure 1**) (Vieira and Kijjoa, 2005; Pinto *et al.*, 2005; Souza and Pinto 2005; Gales and Damas 2005). The xanthone nucleus is numbered according to a biosynthetic convention with carbons 1–4 being assigned to acetate-derived ring A and carbons 5–8 to the shikimate-derived ring B. The other carbons are indicated as 4a, 4b, 8a, 9, and 9a for structure elucidation purposes (Bennet and Lee 1989).
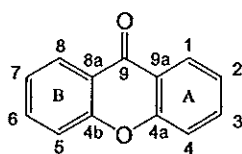
Figure 1 Dibenzo–γ–pyrone or xanthone skeleton

Xanthones is an important class of organic compounds. They have diverse biological properties, many of which are potential leads to pharmaceuticals against diseases such as malaria, HIV-AIDS and various cancers. These pharmacological properties have led to many groups researching the synthesis and biological properties of naturally occurring and synthetic xanthones.

Xanthones was mainly isolated from 20 higher plant families (122 species in 44 genus), 19 fungi species and 3 lichens species, 278 new xanthones were identified between 2000 and 2004 (Vieira and Kijjoa 2005). Currently, approximately 1000 different xanthones have been described (Souza and Pinto 2005). The biological activities of this class of compounds are associated with their tricyclic scaffold but vary depending on the nature and/or position of the different substituents (Souza and Pinto 2005; Bennett and Lee 1989; Mandal *et al.*, 1992).

1.3 Biological activities of xanthones

From many researches, they showed that xanthones have shown several significant biological activities such as antioxidant (Yoshikawa *et al.*, 1994; Fan and Su 1997; William *et al.*, 1995), anti-inflammatory (Yamakuni *et al.*, 2006; Chen *et al.*, 2008), antibacterial (Boonnak *et al.*, 2006), and cytotoxic activities (Kupchan *et al.*, 1980; Geran *et al.*, 1972).

From their high pharmacological properties, it led many researchers to isolate the bioactive xanthones from the higher plants and fungi for a long time.

1.3.1 Antioxidant activity

In 1994, Yoshikawa found that α- and γ-mangostins (1 and 2) (Figure 2), which were isolated from the crude methanol extract from the hull of the *Garcinia mangostin*, showed DPPH radical scavenging activity, antioxidant activity using the ferric thiocyanate method (Yoshikawa *et al.*, 1994; Fan and Su 1997). In 1995, Williams found that α-mangostin decreases the human low density lipoproteins (LDL) oxidation induced by copper or peroxyl radical (William *et al.*, 1995). In this paper, it claimed that α-mangostin is a potent substance for preventing the development of antherosclerosis.

(1) ; R = -CH₃ ; α-mangostin
(2) ; R = -H   ; γ-mangostin
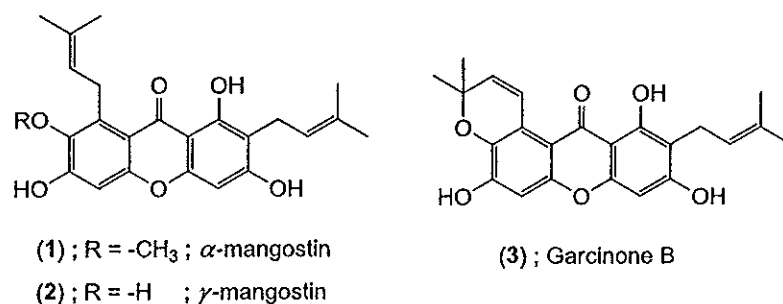
(3) ; Garcinone B

Figure 2 Chemical structures of compounds 1-3

1.3.2 Anti-inflammatory activity

In 2006, Yamakuni found that garcinone B (3) (Figure 2) (10 µM) reduced by 30% the increase of PGE_2 release induced by A23187 in C6 rat glioma cells. Garcinone B (20 µM) also diminished 30% of lypopolisaccharide-induced nuclear factor κB activation. These results suggest that garcinone B may be a pharmacological tool to investigate intracellular signaling pathways involved in inflammation (Yamakuni *et al.*, 2006).

Recently, Chen and co-worker demonstrated that α- and γ-mangostins (Figure 2) significantly exhibited the inhibitory effect on nitric oxide (NO) production in murine macrophage-like RAW264.7 cell lines with the IC_{50} values at 11.1 and 4.5 μM, respectively (Chen *et al.*, 2008). All the above data indicate that the isolated xanthones could be a novel target of anti-inflammatory agents.

1.3.3 Antibacterial activity

In 2006 (Boonnak *et al.*, 2006), Boonnak and co-workers showed that some of the isolated xanthones (**Figure 3**) from the *Cratoxylum formosum* ssp. *pruniflorum* exhibited potent antibacterial activity. From the antibacterial activity results, it suggested that pruniflorone E (**4**) and *α*-mangostin showed potent antibacterial activity against *B. subtilis*, *S. aureus* and *S. faecalis* with MIC values at <1.1 μg/mL, whereas pruniflorone C (**5**) and 3,4-dihydro-jacareubin (**6**) exhibited strong activity against *B. subtilis* and *S. aureus* with MIC value at <1.1 μg/mL. Moreover, gerontoxanthone I (**7**) showed inhibition against two Gram-negative bacteria *S. sonei* and *P. aureginisa* with MIC value at <1.1 μg/mL.

(**4**) ; Pruniflorone E

(**5**) ; Pruniflorone C

(**6**) ; 3,4-dihydrojacareubin

(**7**) ; Gerontoxanthone I
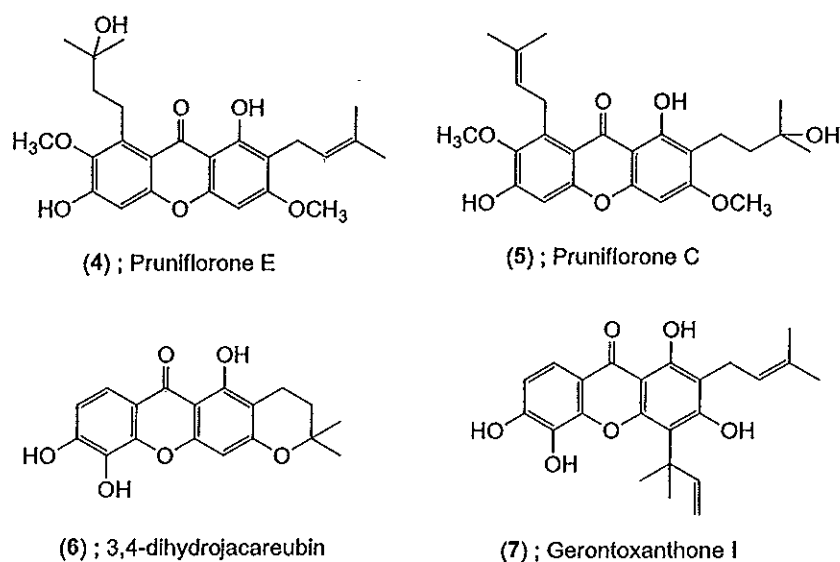
Figure 3 Chemical structures of compounds 4-7

1.3.4 Cytotoxic activity

Kupchan and co-workers showed that psorospermin (**8**), psorofebrin (**9**) and isohydroxyisopsorofebrin (**10**) (**Figure 4**), an angular furanoxanthone, was isolated from the dried roots of *Psorospermum febrifugum* (Kupchan *et al.*, 1980). They exhibited significant activity *in vivo* against P-388 lympphocytic leukemia in mice and *in vitro* against a cell derived from a human epidermoid carcinoma of the nasopharynx (9KB) (Geran *et al.*, 1972).

(8) ; Psorospermin   **(9) ; Psorofebrin**   **(10) ; Isohydroxyisopsorofebrin**
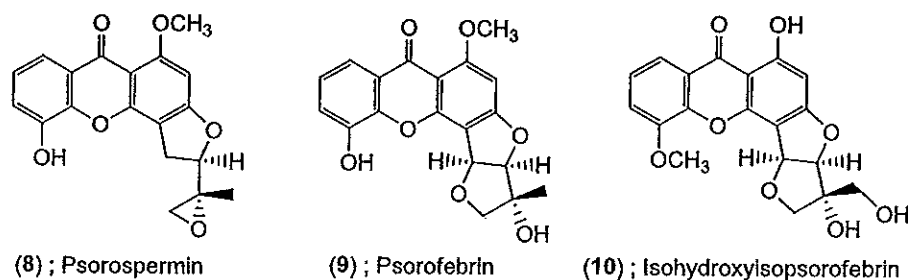
Figure 4 Chemical structures of compounds **8-10**

From theses attractive activity result of psorospermin (**8**), it led Cassady and co-workers to synthesize the analogue of psorospermin (Cassady *et al.*, 1987), their synthesized structures (**11-13**) were illustrated in **Figure 5**. In 1998, Nitiss suggested that psorospermin is believed to be a topoisomerase II poison (Nitiss, J. L. 1998). Topoisomerase II is an enzyme that plays several key roles in DNA metabolism and chromosome structure. It irreversibly binds to the typoisomerase-DNA complex thus having a clinical advantage associated with more potent cytotoxic effects towards cancer cells (Vladu *et al.*, 2000).

(11)   **(12)**   **(13)**
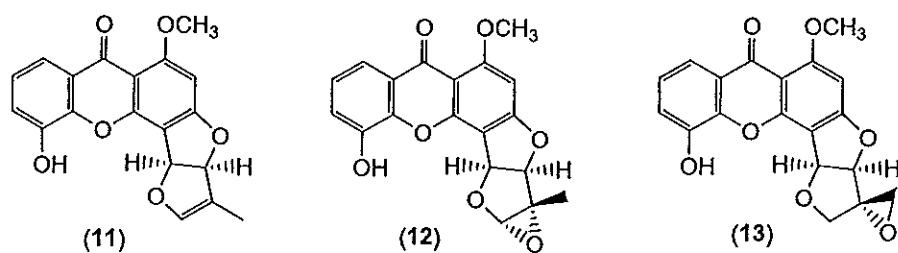
Figure 5 Potential alkylating bisfuranoxanthones **11-13**

1.4 Classification of xanthones

Xanthones have been classified in five categories (Mandal *et al.*, 1992).

a) Simple oxygenated xanthones

b) Xanthone glycosides

c) Prenylated xanthones

d) Xanthonolignoids

e) Miscellaneous

1.4.1 Simple oxygenated xanthones

They can be further subdivided into six groups depending on the degree of oxygenation pattern of the basic skeletons.

(a) Mono-oxygenated xanthones: They are unusual and only a small number of mono-oxygenated. 2-Hydroxyxanthone (**14**) (**Figure 6**) is an example of xanthone in this class (Gottlieb and Stefani 1970).
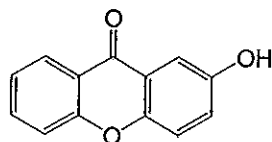
2-hydroxyxanthone (**14**)

Figure 6 The structure of mono-oxygenated xanthone

(b) Di-oxygenated xanthones: 1,5-dihydroxyxanthone (**15**) (**Figure 7**) is a common dioxygenated xanthone, which was firstly isolated from *Calophyllum scriblitifolic* (Jackson *et al.*, 1967).
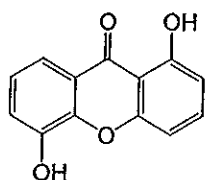
1,5-dihydroxyxanthone (**15**)

Figure 7 The structure of di-oxygenated xanthone

(c) Tri-oxygenated xanthones: They can be more frequently encountered in nature. 1,3,7-trihydroxyxanthone (**16**) (**Figure 8**) was isolated from *Athyrium mesosorum*. (Noro *et al.*, 1984).
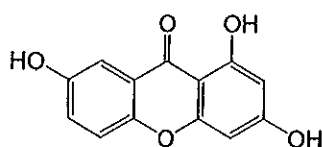
1,3,7-trihydroxyxanthone (**16**)

Figure 8 The structure of tri-oxygenated xanthone

(d) Tetra-oxygenated xanthones: They are more numerous than tri-oxygenated xanthones. 1,3,6,7-tetrahydroxyxanthone (**17**) (**Figure 9**) was isolated from the heart wood of *Garcinia mangostana* (Farnsworth and Bunyapraphatsara 1992).
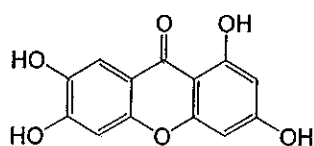
1,3,6,7-tetrahydroxyxanthone (**17**)

Figure 9 The structure of tetra-oxygenated xanthone

(e) Penta-oxygenated xanthones: This class was rarely found in nature. 1,5,6-tri-hydroxy-3,7-dimethoxyxanthone (**18**) (**Figure 10**) was isolated from the *Canscora decussata* (Biwas *et al.*, 1977).
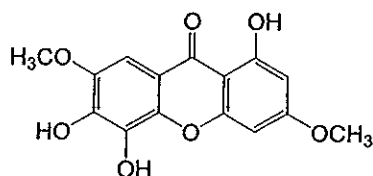
1,5,6-trihydroxy-3,7-dimethoxyxanthone (**18**)

Figure 10 The structure of penta-oxygenated xanthone

(f) Hexa-oxygenated xanthones: They have the highest degree of oxygenation observed so far and only a few compounds were identified. The structure of 1-hydroxy-2,3,4,5,8-pentamethoxyxanthone (**19**) was shown in **Figure 11** as an example of this class (Rodriguez *et al.*, 1995).
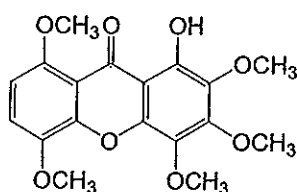
1-hydroxy-2,3,4,5,8-pentamethoxyxanthone (**19**)

Figure 11 The structure of hexa-oxygenated xanthone

1.4.2 Xanthone glycosides

They might be divided into *O*-glycosides and *C*-glycosides according to the nature of the glycosidic linkage.

(a) *O*-Glycoside xanthones: The most of *O*-glycoside xanthones have sugar moiety attached to C-1 and/or C-3 positions of the xanthone nucleus. Since the attachment of sugar moiety at C-1 position might create a strain, it is also possible to observe the glycosyl moiety at any position of the xanthone nucleus such as C-3 position. They are easily hydrolyzed in enzymatic or acid environment (Hostettmann and Wagner 1977). Patuloside A (**20**) (**Figure 12**) was isolated from *H. patulum* (Ishiguro *et al.*, 1999).
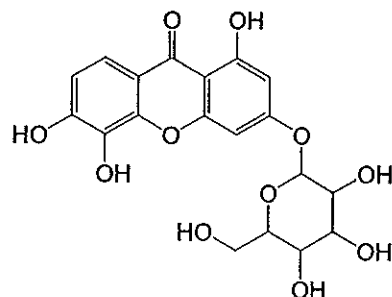
3-*O*-*β*-D-glucopyranosyl-1,5,6-trihydroxyxanthone or patuloside A (**20**)

Figure 12 The structure of *O*-glycoside xanthone

(b) *C*-Glycoside xanthones: They are more resistant to hydrolysis compared with *O*-glycoside xanthones, but their occurrence is very much limited. Mangiferin (**21**) (**Figure 13**) was isolated from the leaves of *Cratoxylum formosum* ssp. *pruniflorum* (Kitanov *et al.*, 1988).
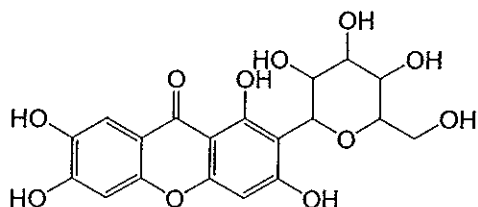
2-*β*-D-glucopyranosyl-1,3,6,7-tetrahydroxyxanthone or mangiferin (**21**)

Figure 13 The structure of *C*-glycoside xanthone

1.4.3 Prenylated xanthones

The plants in the *Guttiferae* family appear to produce a large number of xanthones with prenyl and geranyl substituents. Prenylated and geranylated xanthones (**22** and **23**) (**Figure 14**) were isolated from the bark of Vietnamese *Cratoxylum cochinchinense* (Nguyen and Harrison 1998).
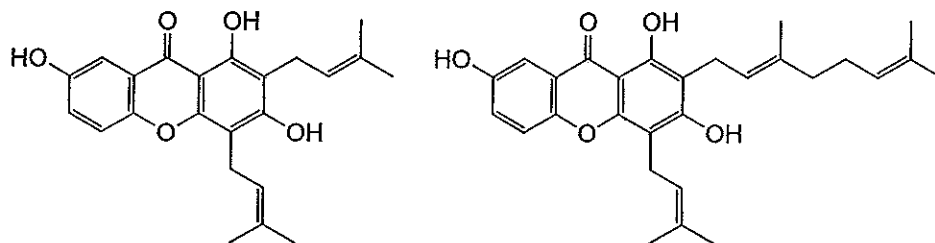
prenylated xanthone (**22**)   geranylated xanthone (**23**)

Figure 14 The structure of prenylated xanthones

1.4.4 Xanthonolignoids

They are a rare group of natural products and principally occur in some genera of the *Guttiferae* family: *Caraipa* (Castelao *et al.*, 1977), *Hypericum* (Cardona *et al.*, 1986) and *Psorospermum* (Abou-Shoer *et al.*, 1989). They are a class of compounds with a phenylpropane (lignoid pattern) skeleton linked to an *ortho*-dihydroxanthone by a dioxane ring, which was formed by radical oxidative coupling (Nielsen and Arends 1978). 5'-demethoxycadensin G (**24**) (**Figure 15**), an oxidative coupling product between 1,3,5,6-tetra-hydroxyxanthone with (*E*)-coniferyl alcohol, was isolated from the bark of *Cratoxylum cochinchinense* (Sia *et al.*, 1995).
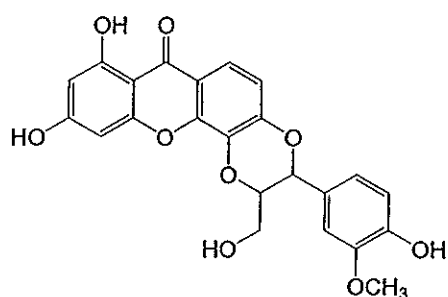
5'-demethoxycadensin G (**24**)

Figure 15 The structure of xanthonolignoid

1.4.5 Miscellaneous xanthones

Besides these groups, some xanthones with unusual substitutions have been isolated from different plant sources including lichens, which could not be classified in the usual manner. 4-chloro-3,8-dihydroxy-6-methoxy-1-methylxanthone (**25**) from *Hypericum ascyron* (Hu *et al.*, 1999) and a sulfonated xanthone (**26**) from *Hypericum sampsonii* (Hong *et al.*, 2004) were shown in **Figure 16** as examples of this group.
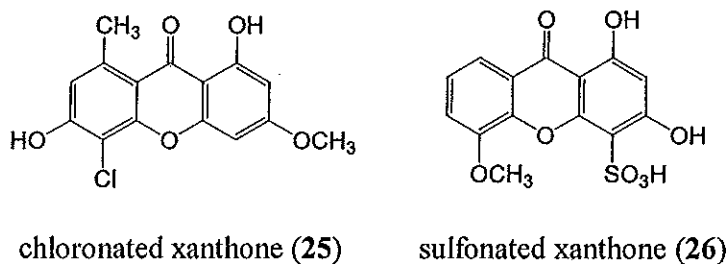
chloronated xanthone (**25**)   sulfonated xanthone (**26**)

Figure 16 The structures of miscellaneous xanthones

1.5 Biosynthesis of xanthones

The biosynthetic pathways to xanthones have been discussed for 40 years. Biosynthetically, the xanthones of higher plants are formed from shikimate and acetate origins. In 1969 (Carpenter *et al.*, 1969), Carpenter and co-worker proposed that phenylalanine (27) was derived from shikimate, then losing two carbon atoms from the side chain and further oxidized to give *m*-hydroxybenzoic acid (28), which further combined with three units of acetate (probably via malonate) to produce the shikimate-acetate intermediate (Figure 17). The benzophenone skeleton was formed in the next step. The xanthone framework was constructed in the final step by an oxidative phenol coupling reaction (Lewis, 1963). In 1971, the important proof of this biosynthesis pathway was performed on *Gentiana lutea* by Gupta and Lewis (Gupta and Lewis 1971).
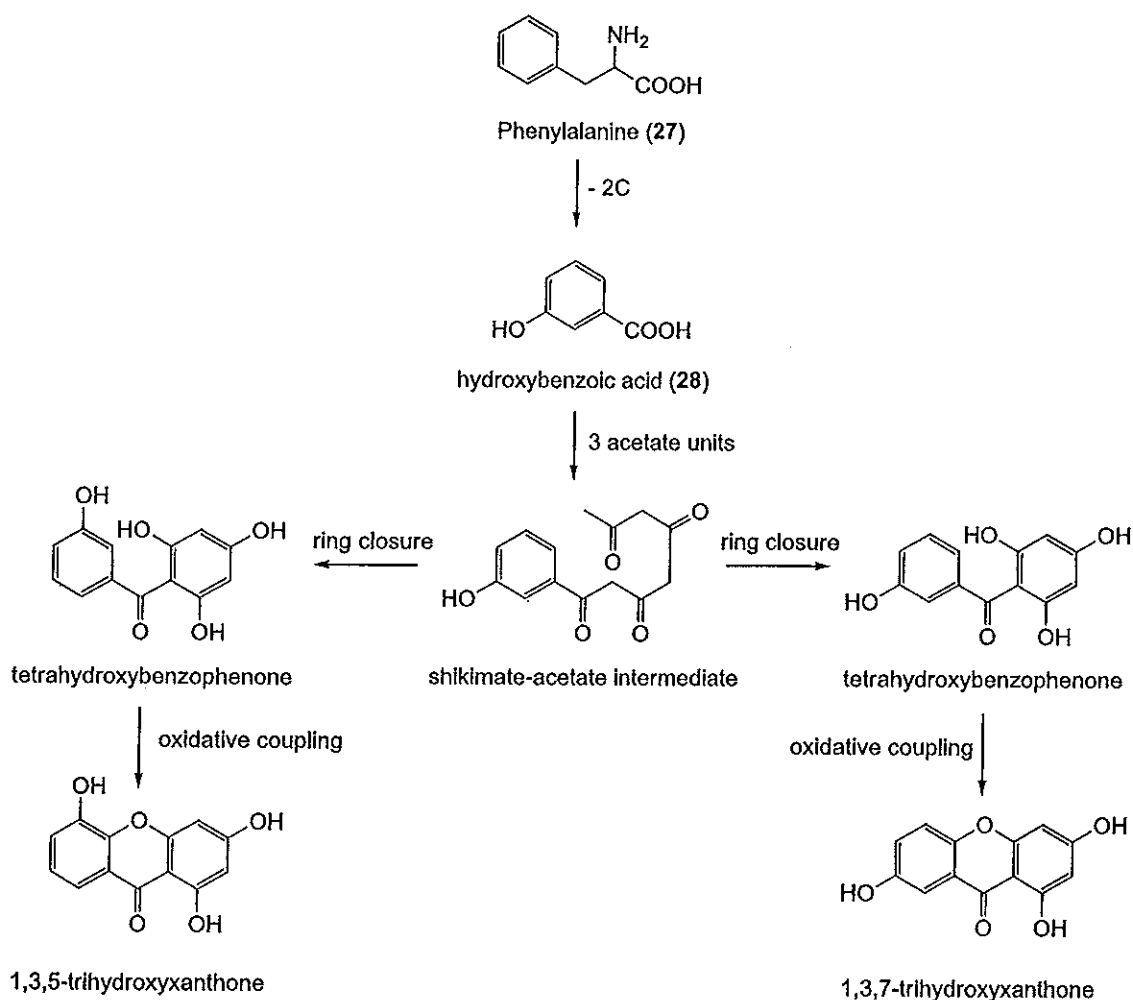
Figure 17 Proposed xanthone biosynthetic pathway by Carpenter

Gottlieb proposed the other biosynthesis xanthone formation (**Figure 18**), which was derived from the 5-dehydroshikimic acid (**29**) as a precursor (Gottlieb, 1968). Firstly, compound **29** condenses with the phenol by acylation reaction resulting in the dione intermediate. The latter will be oxidized to give the aucuparin type products (**30**). The xanthone nucleus was formed in the final step by nucleophilic attack by the ether bridge (route A) to give 5,6,7-trihydroxyxanthone, whereas 6,7,8-trihydroxyxanthone was built through an electrophilic attack by the carbonyl bridge (route B).
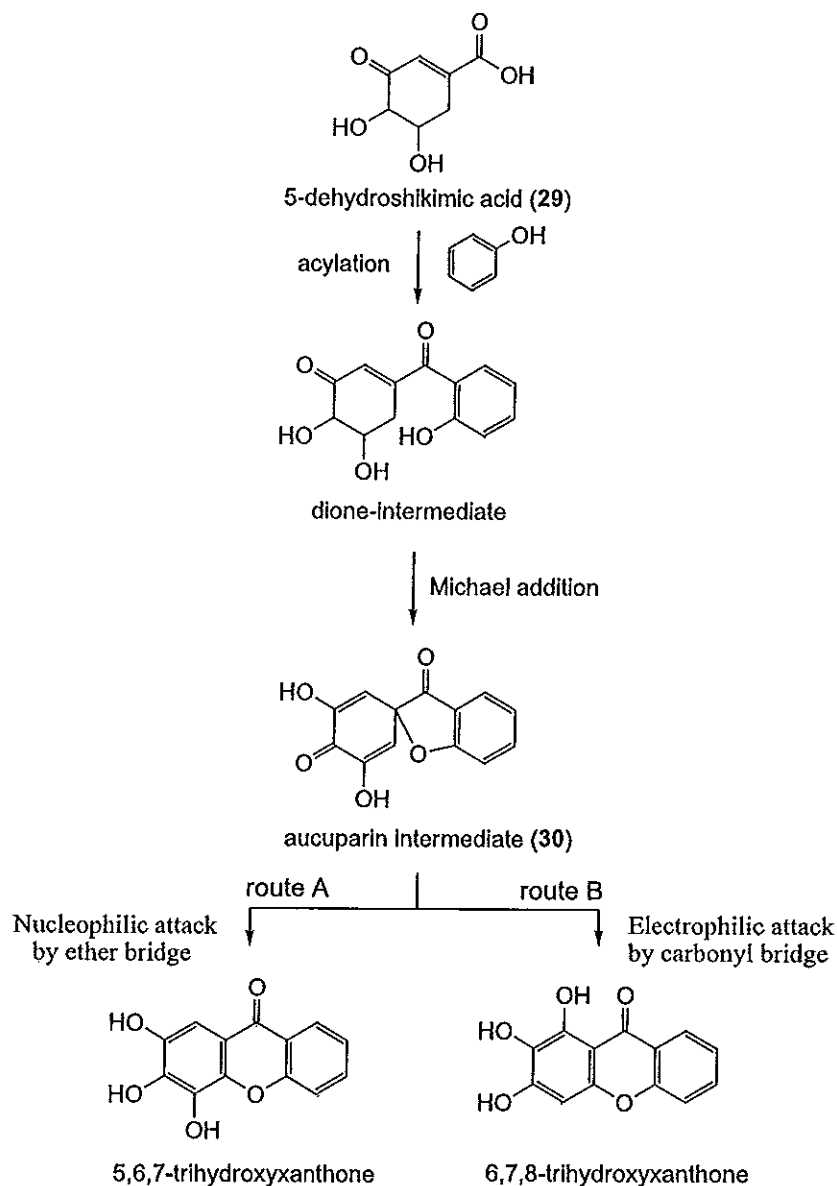
Figure 18 Proposed xanthone biosynthetic routes by Gottlieb

In 1996, Beerhues proposed the different biosynthesis route of xanthone, which was detected from the cultured cells of *Centaurium erythraea* (Beerhues, 1996). Its result showed

that the formation of 2,3',4,6-tetrahydroxybenzophenone was observed in cell-free extracts from cultured cells of *C. erythraea* (**Figure 19**).

A year later, Schimidt and Beerhues also showed the interesting result that the benzophenone-3'-hydroxylase, as a key enzyme for benzophenone formation, was detected in cultured *H. androsaemum* cells (Schimidt and Beerhues 1997). In *C. erythraea* (**Figure 19**), 2,3',4,6-tetrahydoxybenzophenone is converted to 1,3,5-trihydroxyxanthone by xanthone synthase however, in *H. androsaemum* (**Figure 19**), it is cyclized to 1,3,7-trihydoxyxanthone through oxidative phenol coupling reaction. These two isomers are precursors of the majority of higher plant xanthones (Schimidt and Beerhues 1997).
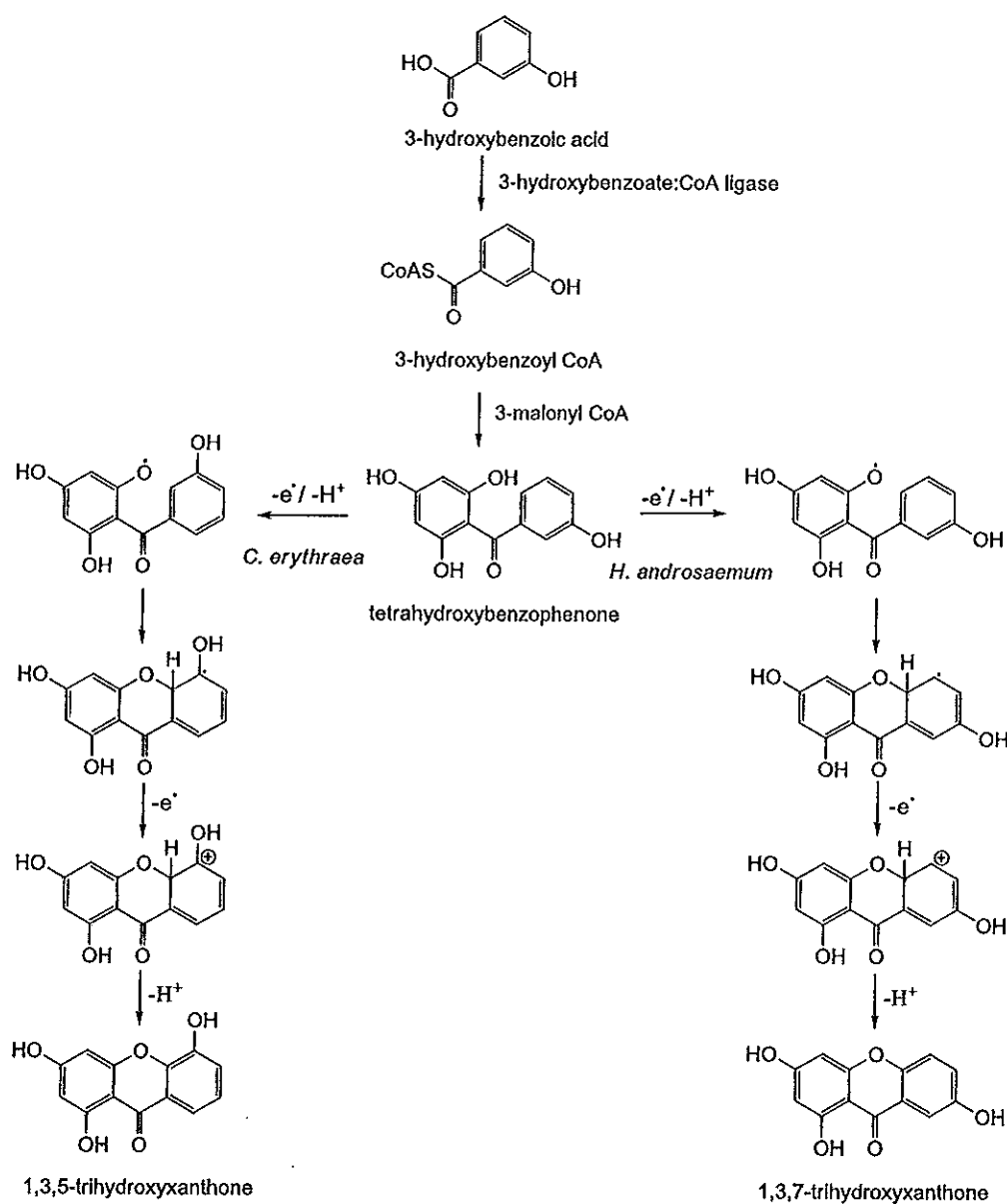
Figure 19 Reaction mechanism of xanthone in cell cultures of *C. erythraea* and *H. androseamum*

In 2001(Kitanov and Nedialkov 2001), Kitanov and Neialkov revealed that the co-occurrence of hypericophenoside (31) and 1,3,7-trihydroxyxanthone (32) were observed in large amount and also found that the benzophenone O-glycoside (31) was easily transformed into 1,3,7-trihydroxyxanthone (32) by acid or enzymatic hydrolysis (Figure 20). This result supports the evidence that 2,4,5',6-tetrahydroxy-benzophenone-2'-O-glycoside (31) is a precursor of 1,3,7-trihydroxyxanthone (32). With these results it can be concluded that some xanthones are formed in vivo by dehydration of 2,2'-dihydroxybenzophenones.

benzophenone O-glycoside (31)

benzophenone intermediate

1,3,7-trihydroxyxanthone (32)
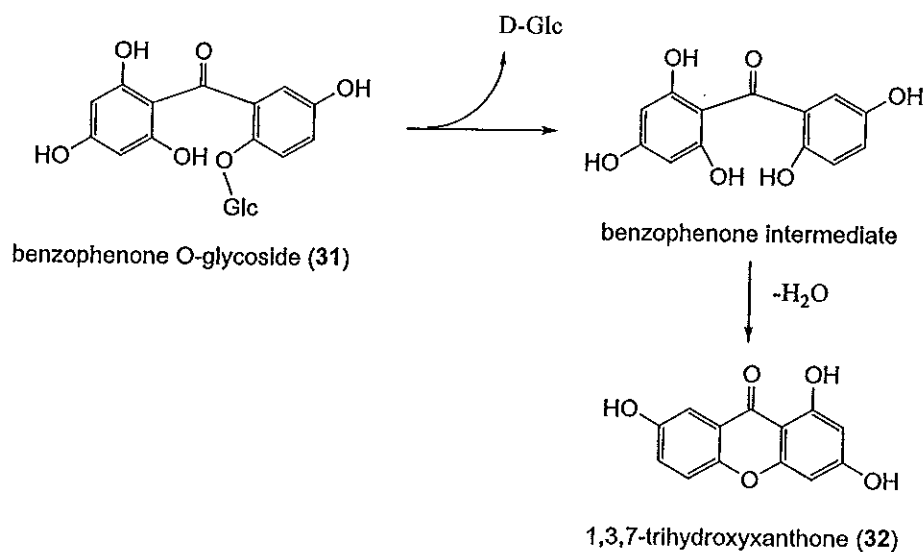
Figure 20 Xanthone formation through dehydration mechanism

1.6 Sources of xanthones

The majority of xanthones are widely distributed in the tropical plants that belong to the *Guttiferae* family, which are contained over 1000 plant species in this family (Bennett and Lee 1989). The *Garcinia* is a well known plants genus, which is mainly produce a 1,3,6,7-tetraoxygenated xanthones as a majority such as mangostin, α-mangostin, β-mangostin and γ-mangostin (Peres *et al.*, 2000). Some earlier articles (Nguyen and Harrison 1998; Phuwapraisirisan *et al.*, 2006), have shown that the *Cratoxylum* plants are new sources to produce the 1,3,7-tri- and 1,3,5,6-tetraoxygenated xanthones, which are different types to those isolated xanthone from the *Garcinia* plants. Some of the isolated xanthones from the *Cratoxylum* plants, also exhibited several interesting biological activities such as antibacterial (Boonsri *et al.*, 2006), antimalarial (Laphookhieo *et al.*, 2006), antioxidant (Mahabusarakam *et al.*, 2006; Phuwapraisirisan *et al.*, 2006) and cytotoxic (Seo *et al.*, 2002) activities. These

interesting results led us to investigate the chemical constituents from the plant in the *Cratoxylum* genus.

1.7 Cratoxylum genus

Cratoxylum belongs to the family Guttiferae, which is distributed in several Southeast Asian countries. Six species of Cratoxylum plants were found in Thailand (Smitinand, T. 2001).

a) *Cratoxylum arboresens*

b) *Cratoxylum cochinchinense*

c) *Cratoxylum maingayi*

d) *Cratoxylum sumatranum ssp neriifolium*

e) *Cratoxylum formosum ssp. formosum*

f) *Cratoxylum formosum ssp. pruniflorum*

The last two species, which are subspecies of *C. formosum*, can be differentiated through the young twigs, leaves, pedicels, and sepals. Those of *C. formosum* ssp. *formosum* are glabrous, whereas *C. formosum* ssp. *pruniflorum* are densely villous (Veesommai and Kavduengtain 2004). Some species of this genus have been used for the treatment of diuretic, stomachic, and tonic effects (Kitanov *et al.*, 1988), as well as for diarrhea and flatulence (Aderson, 1986), and for food poisoning and internal bleeding (Grosvenor *et al.*, 1995). For this study, *C. cochichinese* and *C. formosum* ssp. *pruniflorum* were chosen for detail investigations.

1.7.1 Cratoxylum cochinchinense

"Tui-Kliang" is the local name of *C. cochinchinense*, which is shrub or tree, 1.5-1.8 or 25 m tall. Trunk (**Figure 21**) is with tufted long spines on lower part. Bark is grey-yellow or gray-brown, smooth or finely straight. Twigs are compressed, glabrous and pink when young, interpetiolar scars not always continuous. Petioles are 2-3 mm, glabrous; leaf blades elliptic to oblong or lanceolate, 3-10.5×1-4 cm, apery, both surface glabrous, abaxially gray-green and with pellucid or dark glands, adaxially green, base obtuse to cuneate, apex abruotly acute or acuminate; midvein abaxially elevated, adaxially impressed; lateral vein 8-12 pairs, oblique, free; veins and veinlets reticulate, elevated on both surfaces. Cymes are axially or

extra-axaillary and terminal, 1 or 2 or 3-flowered, pedunculate; peduncles 3-10 mm or longer. Pedicel 2-3 mm. Flowers are 1-1.5 cm in diameter. Sepals are oblong, 5-7×2-5 mm, apex rounded, with dark linear glands on entire surface, accrescent. Petals are deep crimson to pink or pinkish yellow (**Figure 21**), obovate, 5-10×2.5-5 mm, with dark linear gland between veins, without a petal-scale, base cuneat, apex rounde. Stamen fascicles are 4-8 mm, stalk broad to slender. Fasciclodes are oblong to obovate, cucculate, to 3×1-1.5 mm, apex thickened and recurved. Ovary conical, is *ca.* 3 mm, glabrous; styles linear, ca. 2 mm, divaricate from base. Capsule is brown, ellipsoid, 0.8-1.2 cm, glabrous, to 2/3 covered by persistent calyx. Seeds are 5 or 6-8 in each cell (**Figure 21**), obovoid, 6-8×2-3 mm.
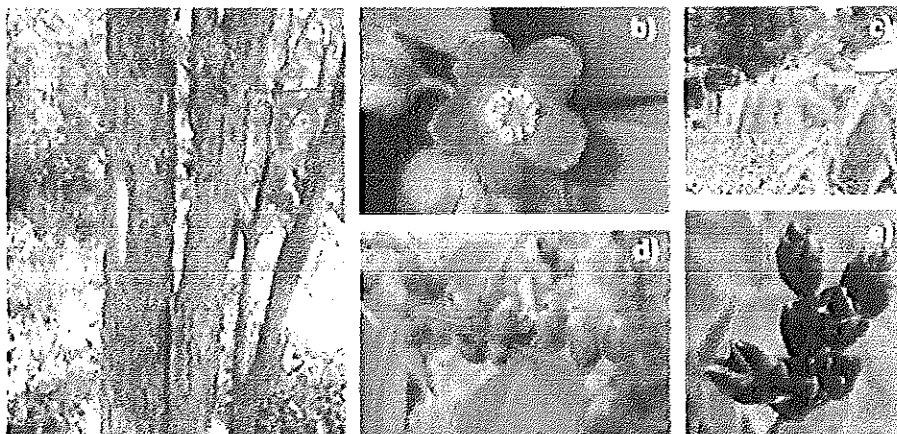
Figure 21 Different parts of *Cratoxylum cochinchinense*
a) Trunk   b) Flower   c) Leaves   d) Green Fruits   e) Ripe Fruits

1.7.2 *Cratoxylum formosum* ssp. *pruniflorum*

The local name of *C. formosum* ssp. *pruniflorum* is "Tui-Khon", which is a shrub or tree (**Figure 22**), deciduous to evergreen, 3-8 m tall. Its barks are brown or dark-grey, and barks and spines are attached on stem or old branch. The resin is red-yellow. Special young twigs, leaves, pedicels and sepals are densely villous. Leaves are simple, opposite, oblong lanceolate, entire, acute, attenuate, 3-5 cm by 6-8 cm. Young leaves are usually red and the mature ones densely villous. Flowers are complete, sepal 5, deep red or scarlet, petal 5, pink or white, 0.3-0.5 by 2-3 cm. Seeds are 5-8 in each cell.
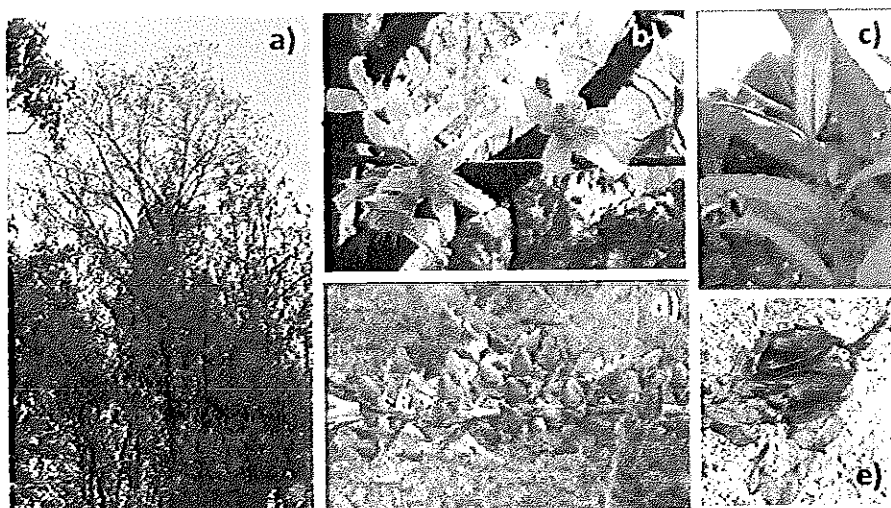
Figure 22 Different parts of *Cratoxylum formosum* ssp. *pruniflorum*

a) Tree b) Flowers c) Leaves d) Green Fruits e) Ripe Fruits

1.8 Reviews of literatures

The literature search has been done by using the SciFinder Scholar database. The chemical constituents isolated from the *Cratoxylum* genus were summarized in **Table 1**.

Table 1 Chemical Constituents from the *Cratoxylum* genus (1988-2007)

The isolated compounds from the *Cratoxylum* genus were classified in 9 groups.

a) Flavonoids   f) Anthraquinones

b) Xanthones   g) Tocotrienols

c) Caged-xanthones   h) Bicyclic-triterpenoids

d) Bianthrones   i) Triterpenoids

e) Vismones

Bibliography	Part and Plant Name	Isolated Compounds
Kitanov *et al.*, 1988	Leaves of *C. formosum* ssp. *pruniflorum*	quercetin (1a) hyperoside (2a) 1,3,6,7-tetrahydroxyxanthone (6b) mangiferin (60b) isomangiferin (61b)

Table 1 (Continued)

Bibliography	Part and Plant Name	Isolated Compounds
Bennett *et al.*, 1993	Bark of *C. cochinchinense*	2-geranyl-4-(3,3dimethylallyl)-1,3,7-trihydroxyxanthone (**19b**) α-mangostin (**32b**) β-mangostin (**33b**) garcinone D (**34b**) torophyllin A (**35b**) cratoxylone (**36b**) δ-tocotrienol (**1g**) δ-tocotrienol dimer (**2g**) 5-(γ-tocotrienyl)-γ-tocotrienol (**3g**) polypoda-8(26),13,17,21-tetraen-3β-ol (**1h**) friedelin (**5i**)
Sia *et al.*, 1995	Bark of *C. cochinchinense*	1,3,5,6-tetrahydroxyxanthone (**7b**) 11-ydroxy-1-isomangostin (**37b**) cratoxyxanthone (**44b**) 5′-demethoxycadensin G (**62b**)
Ilnuma *et al.*, 1996	Root of *C. formosum* ssp. *formosum*	(-)-epicatechin (**3a**) astilbin (**4a**) 1,7-dihydroxyxanthone (**1b**) 8-methoxy-1,7-dihydroxyxanthone (**2b**) 1,4,7-trihydroxyxanthone (**3b**) 4-methoxy-1,7-dihydroxyxanthone(**4b**) 1,2-dimethoxy-3,8-dihydroxyxanthone (**8b**) 1,8-dimethoxy-2,7-dihydroxanthone (**9b**)

Table 1 (Continued)

Bibliography	Part and Plant Name	Isolated Compounds
Iinuma *et al.*, 1996 (*Continued*)	Root of *C. formosum* ssp. *formosum*	8-methoxy-1,4,7-trihydroxyxanthone (**11b**) 1,2,3,4,8-pentamethoxyxanthone (**12b**) macluraxanthone (**23b**)
Kijjoa *et al.*, 1998	Wood of *C. maingayi*	1,7-dihydroxyxanthone (**1b**) 8-methoxy-1,7-dihydroxyxanthone (**2b**) 4-methoxy-1,7-dihydroxyxanthone (**4b**) 1,2,3,8-tetramethoxy-7-hydroxy-xanthone (**14b**)
Nguyen *et al.*, 1998	Stem bark of *C. cochinchinense*	2-geranyl-4-(3,3dimethylallyl)-1,3,7-trihydroxyxanthone (**19b**) 2,4-di-(3-methyl-but-2-enyl)-1,3,7-tri-hydroxyxanthone (**20b**) 7-geranyloxy-1,3-dihydroxyxanthone (**22b**) *β*-mangostin (**33b**) polypoda-8(26),13,17,21-tetraen-3*β*-ol (**1h**) (13*E*,17*E*)-Polypoda-7,13,17,21-tetra-en-3*β*-ol (**2h**) lupeol (**1i**)
Seo *et al.*, 2002	Twigs, stem barks and leaves of *C. sumatranum*	cratoxyarborenone A-F (**54b-56b, 59b, 38b** and **5b**) vismione B (**1e**) cratoxyarborequinone A-B (**1f-2f**) δ-tocotrienol (**1g**)

Table 1 (Continued)

Bibliography	Part and Plant Name	Isolated Compounds
Pattanaprateeb *et al.*, 2005	Stem bark of C. *arborescenes*	1,7-dihydroxyxanthone (**1b**) 1,3-dihydroxy-6,7-dimethoxy-2,8-di-prenylxanthone (**39b**) fuscaxanthone C (**40b**) 3-geranyloxy-6-methyl-1,8-dihydroxy-anthraquinone (**3f**) 2-geranylemodin (**9f**)
Boonsri *et al.*, 2006	Roots of C. *formosum* ssp. *formosum*	formoxanthone A-C (**15b, 16b** and **27b**) macluraxanthone (**23b**) gerontoxanthone I (**25b**) xanthone V_1 (**28b**) 3-geranyloxy-6-methyl-1,8-dihyroxy-anthraquinone (**3f**) madagascin (**4f**) vismiaquinone (**8f**)
Mahabusarakam *et al.*, 2006	Roots of C. *cochinchinense*	cochinchinone A and B (**21b** and **41b**) 2,4-bis-(3-methyl-2-butenyl)-1,3,7-tri-hydroxyxanthone (**20b**) macluraxanthone (**23b**) α-mangostin (**32b**) β-mangostin (**33b**) garcinone D (**34b**) garcinone B (**42b**) celebixanthone (**57b**) cochinchinone C and D (**1c** and **2c**) caged prenylated xanthone (**3c**)

Table 1 (Continued)

Bibliography	Part and Plant Name	Isolated Compounds
Laphookhieo, *et al.*, 2006	Roots of *C. cochinchinense*	2,4-di(3-methylbut-2-enyl)-1,3,7-tri-hydroxyxanthone (**20b**) cochinchinone A (**21b**) α-mangostin (**32b**) β-mangostin (**33b**) celebixanthone (**57b**) 5-*O*-methylcelebixanthone (**58b**) cochinchinone C (**3c**)
Reutrakul *et al.*, 2006	Leaves and twigs of *C. arborescens*	astilbin (**4a**) isoastilbin (**5a**) eucryphin (**6a**) euxanthone (**1b**) 1,7-dihydroxy-2,8-dimethoxyxanthone (**10b**) 1,3,8-trihydroxy-2,4-dimethoxy-xanthone (**13b**) 1,3,7-trihydroxy-6-methoxy-4,5-di(3-methylbut-2-enyl)xanthone (**38b**) physcion (**5f**) betulinic acid (**2i**) lup-20(29)-ene-3β,30-diol (**3i**) 3β-hydroxylup-20(29)-en-30-oic acid (**4i**) friedelin (**5i**) friedelinol (**6i**)
Boonnak *et al.*, 2006	Root and bark of *C. formosum* ssp. *pruniflorum*	pruniflorone A-I (**45b-50b, 29b, 30b and 18b**) formoxanthone A (**15b**)

Table 1 (Continued)

Bibliography	Part and Plant Name	Isolated Compounds
Boonnak *et al.*, 2006 (*continued*)	Root and bark of *C. formosum* ssp. *pruniflorum*	formoxanthone B (**16b**)
		6-deoxyjacareubin (**17b**)
		macluraxanthone (**23b**)
		10-*O*-methylmacluraxanthone (**24b**)
		gerontoxanthone I (**25b**)
		3,4-dihydrojacareubin (**26b**)
		xanthone V_1 (**28b**)
		α-mangostin (**32b**)
		β-mangostin (**33b**)
		cratoxylumxanthone A (**43b**)
		3-isomangostin (**51b**)
		3,4-dihydro-5,9-dihydroxy-8-methoxy-7-(3-methoxy-3-methylbutyl)-2,2-di-methyl-2H,6H-pyrano-[3,2-b]xanthen-6-one (**52b**)
		3,4-dihydro-5,9-dihydroxy-7-(3-hydro-xy-3-methylbutyl)-8-methoxy-2,2-di-methyl-2H,6H-pyrano[3,2-b]xanthen-6-one (**53b**)
		3-geranyloxy-6-methyl-1,8-dihydroxy-anthraquinone (**3f**)
		madagascin (**4f**)
		physcion (**5f**)
		emodin (**6f**)
		11-hydroxy-5-methoxy-2,2,9-trimethyl-2H-anthra-[1,2-b]pyran-7,12-dione (**7f**)
		vismiaquinone (**8f**)
		friedelin (**5i**)
		friedelinol (**6i**)

Table 1 (Continued)

Bibliography	Part and Plant Name	Isolated Compounds
Phuwapraisirisan *et al.*, 2006	Stems of *C. cochinchinense*	tectochrystin (**7a**) 2-geranyl-4-(3-methylbut-2-enyl)-1,3,7-trihydroxyxanthone (**19b**) *α*-mangostin (**32b**) *β*-mangostin (**33b**) cratoxylumxanthone A (**43b**)
Boonnak *et al.*, 2007	Barks of *C. formosum* ssp. *pruniflorum*	bianthrones J (**1d**) bianthrone A₁ (**2d**) vismone E and D (**2e** and **3e**) 3-geranyloxy-6-methyl-1,8-dihydroxy-anthraquinone (**3f**) 11-hydroxy-5-methoxy-2,2,9-trimethyl-2H-anthra[1,2-b]-pyran-7,12-dione (**7f**) vismiaquinone (**8f**)

1.8.1 Chemical structures of all isolated compounds from Cratoxylum plants

a) Flavonoids
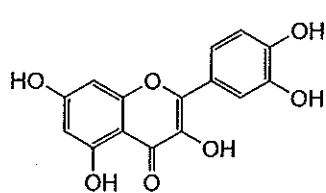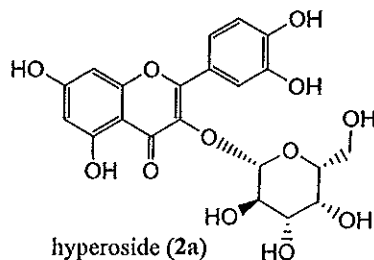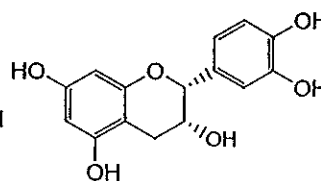
quercetin (1a)   hyperoside (2a)   (-)-epicatechin (3a)
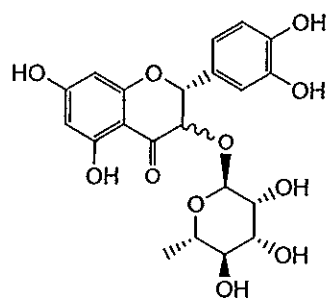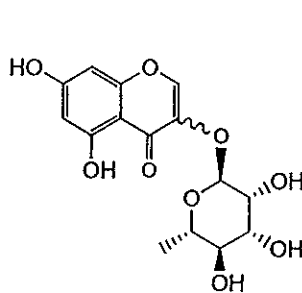
(*3R*)-astibin (4a)   eucryphin (6a)   tectochrystin (7a)

(*3S*)-isoastibin (5a)
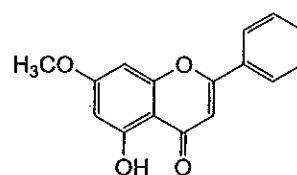
b) Xanthones

- *di-oxygenated xanthone*
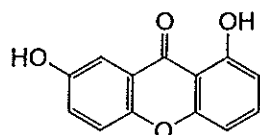
1,7-dihydroxyxanthone (1b)

- *tri-oxygenated xanthone*
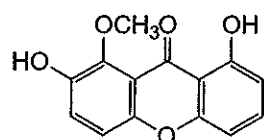
1,7-dihydroxy-8-methoxyxanthone (2b)
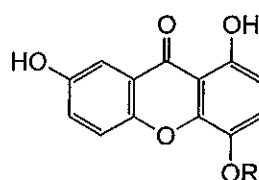
R = H ; 1,4,7-trihydroxyxanthone (3b)

R = CH$_3$; 1,7-dihydroxy-4-methoxyxanthone (4b)

• *tri-oxygenated xanthone (continued)*
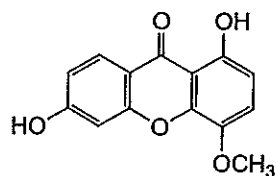
cratoxyarborenone F (5b)

• *tetra-oxygenated xanthone*
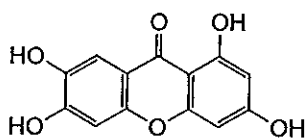
1,3,6,7-tetrahydroxyxanthone (6b)
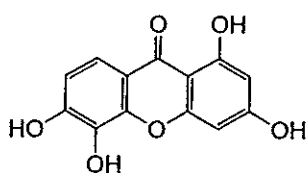
1,3,5,6-tetrahydroxyxanthone (7b)
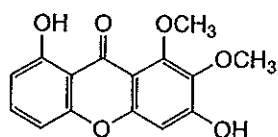
3,8-dihydro-1,2-dimethoxyxanthone (8b)
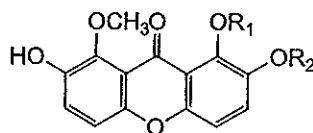
$R_1 = CH_3$ $R_2 = H$
2,7-dihydro-1,8-dimethoxyxanthone (9b)
$R_1 = H$ $R_2 = CH_3$
1,7-dihydro-2,8-dimethoxyxanthone (10b)
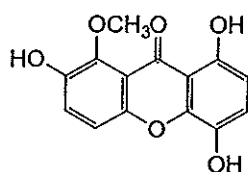
1,4,7-trihydro-8-methoxyxanthone (11b)

• *penta-oxygenated xanthone*
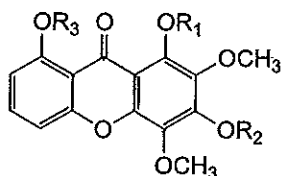
$R_1 = CH_3$ $R_2 = CH_3$ $R_3 = CH_3$
1,2,3,4,8-pentamethoxyxanthone (12b)

$R_1 = H$ $R_2 = H$ $R_3 = H$
1,3,8-trihydroxy-2,4-dimethoxyxanthone (13b)
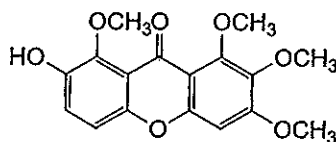
1,2,3,8-tetrahydroxy-7-methoxyxanthone (14b)

- *1,3,5-trioxygenated prenylated xanthone*
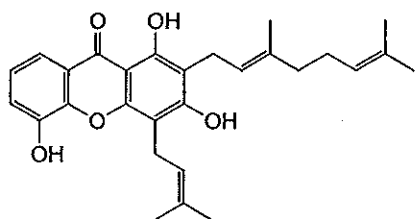
formoxanthone A (15b)
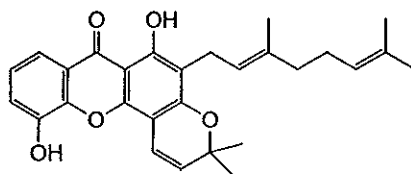
formoxanthone B (16b)
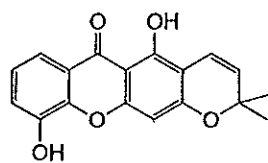
6-deoxyjacareubin (17b)

- *1,3,6-trioxygenated prenylated xanthone*
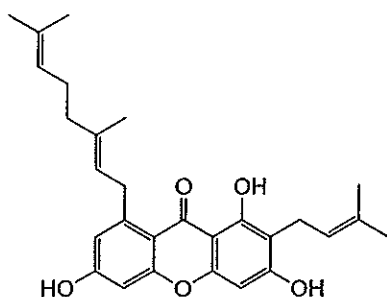
pruniflorone I (18b)

- *1,3,7-trioxygenated prenylated xanthone*
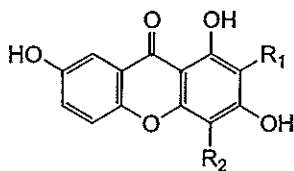
R_1 = geranyl R_2 = isoprenyl
2-geranyl-1,3,7-trihydroxy-4-(3,3-
dimethylallyl)-xanthone (19b)

R_1 = isoprenyl R_2 = isoprenyl
1,3,7-trihydroxy-2,4-(3,3-di-
methylallyl)-xanthone (20b)

R_1 = isoprenyl R_2 = geranyl
cochichinone A (21b)
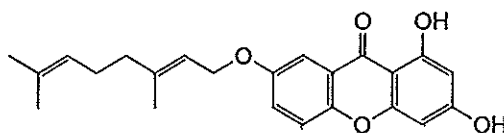
7-geranyloxy-1,3-dihydroxyxanthone (22b)

• *1,3,5,6-tetraoxygenated prenylated xanthone*
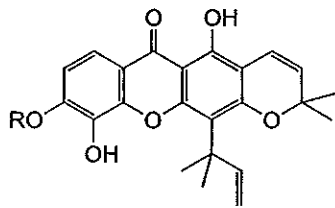
R = H ; macluraxanthone (23b)

R = CH₃ ; 10-*O*-methylmacluraxanthone (24b)
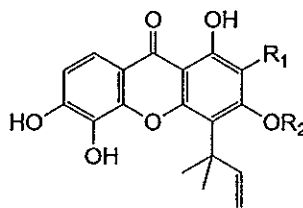
R₁ = isoprenyl R₂ = H ; gerontoxanthone I (25b)

R₁ = H R₂ = H ; 3,4-dihydroxyjacareubin (26b)
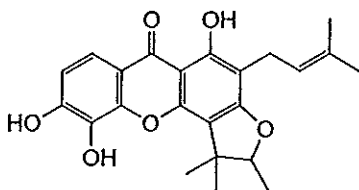
formoxanthone C (27b)
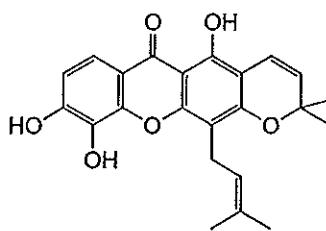
xanthone V₁ (28b)
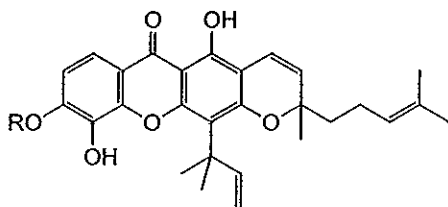
R = H ; pruniflorone G (29b)

R = CH₃ ; pruniflorone H (30b)
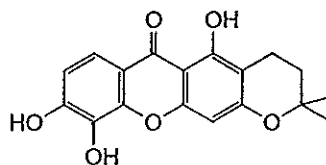
3,4-dihydroxyjacareubin (31b)

• *1,3,6,7-tetraoxygenated prenylated xanthone*
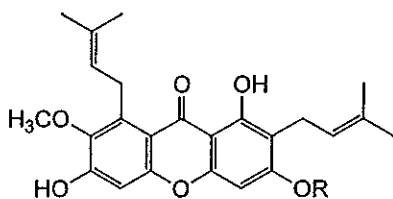
R = H ; α-mangostin (32b)

R = CH₃ ; β-mangostin (33b)
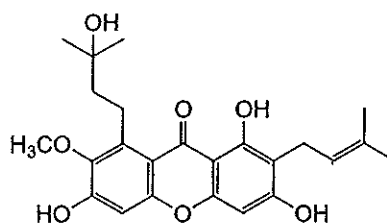
garcinone D (34b)
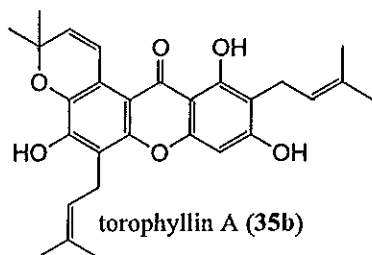
torophyllin A (35b)
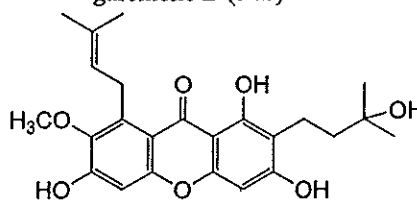
cratoxylone (36b)

● *1,3,6,7-tetraoxygenated prenylated xanthone (continued)*
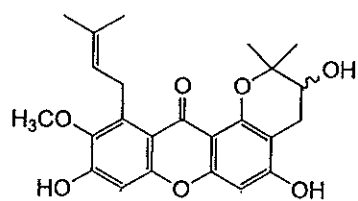
11-hydroxy-1-isomangostin (37b)
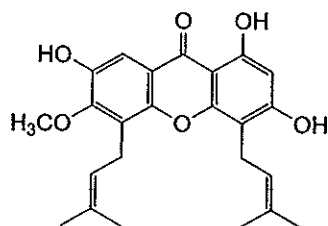
cratoxyarborenone E (38b)
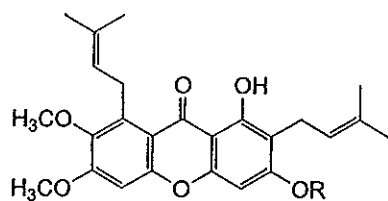
R = H ; 1,3-Dihydroxy-6,7-dime-
thoxy2,8-diprenylxanthone (39b)

R = CH$_3$; fuscaxanthone C (40b)
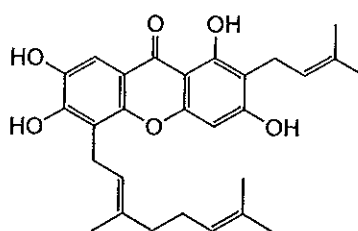
cochichinone B (41b)
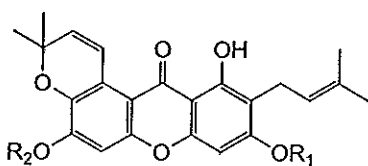
R$_1$ = H   R$_2$ = H ; garcinone B (42b)

R$_1$ = CH$_3$ R$_2$ = H ; cratoxylumxanthone A (43b)
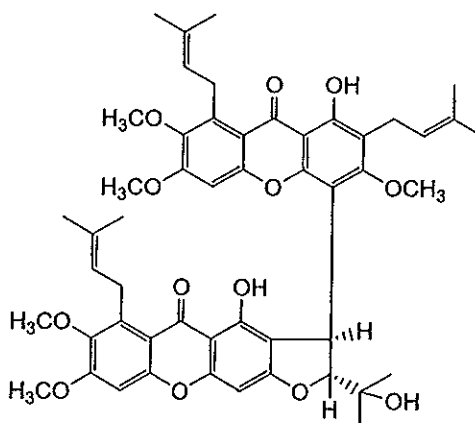
cratoxyxanthone (44b)
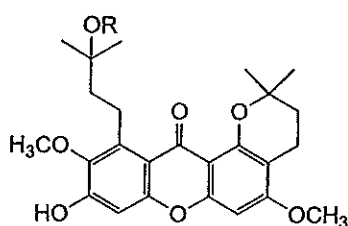
R = H ; pruniflorone A (45b)

R = CH$_3$; pruniflorone B (46b)
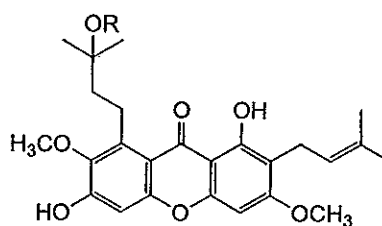
R = H ; pruniflorone C (47b)

R = CH$_3$; pruniflorone D (48b)
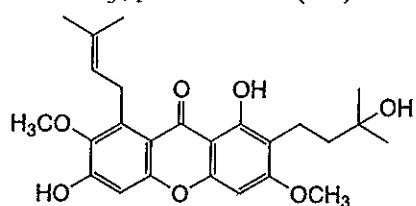
pruniflorone E (49b)
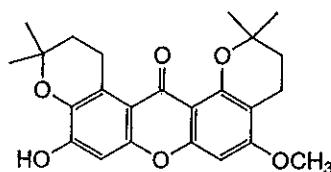
pruniflorone F (50b)

• *1,3,6,7-tetraoxygenated prenylated xanthone (continued)*
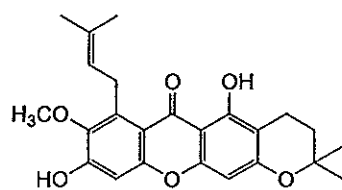
3-isomangostin (51b)
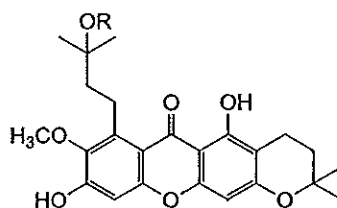
R = H

3,4-dihydro-5,9-dihydroxy-8-methoxy-7-
(3-methoxy-3-methylbutyl)-2,2-dimethyl-
2H,6H-pyrano-[3,2-b]xanthen-6-one (52b)

R = CH₃

3,4-dihydro-5,9-dihydroxy-7-(3-hydroxy-
3-methylbutyl)-8-methoxy-2,2-dimethyl-
2H,6H-pyrano[3,2-b]xanthen-6-one (53b)

• *1,3,5,7-tetraoxygenated prenylated xanthone*
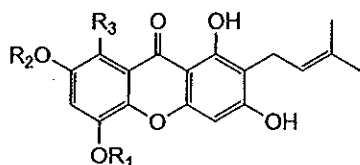
R_1 = H R_2 = H R_3 = geranyl ; cratoxyarborenone A (54b)

R_1 = H R_2 = H R_3 = isoprenyl ; cratoxyarborenone B (55b)

R_1 = CH₃ R_2 = CH₃ R_3 = isoprenyl ; cratoxyarborenone C (56b)

• *1,5,6,7-tetraoxygenated prenylated xanthone*
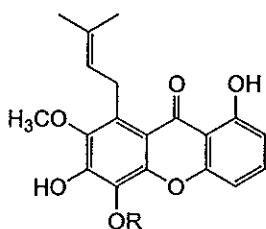
R = H ; celebixanthone (57b)

R = CH₃ ; 5-*O*-methylcelebixanthone (58b)

• *1,3,5,6,7-pentaoxygenated prenylated xanthone*
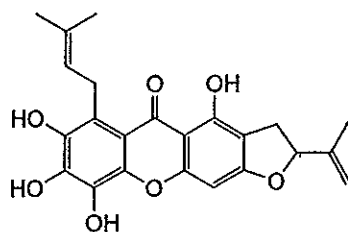
cratoxyarborenone D (59b)

- *C-Glycoside xanthones*
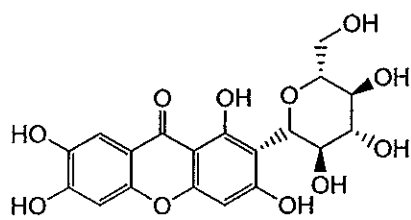
mangiferin (60b)
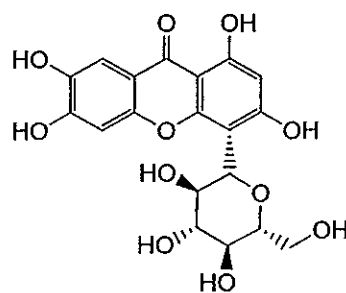
isomangiferin (61b)

- *Xanthonolignoids*
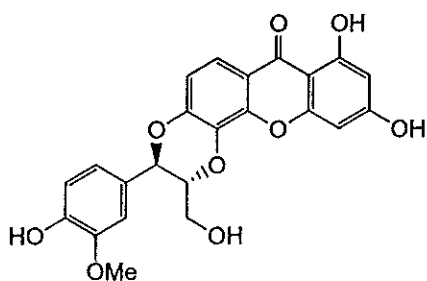
5'-Demethoxycadensin G (62b)

c) *Caged-xanthones*
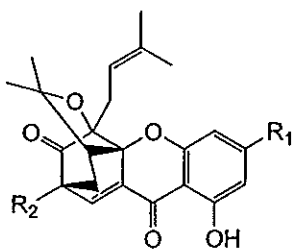
$R_1 = H$ $R_2 = OCH_3$; cochichinone C (1c)

$R_1 = OH$ $R_2 = OCH_3$; cochichinone D (2c)

$R_1 = OH$ $R_2 = H$; caged xanthone (3c)

d) Bianthrones
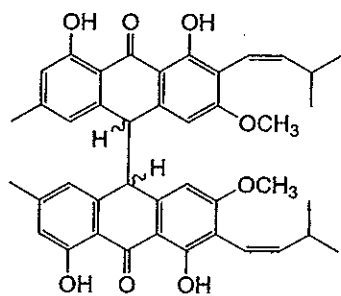
bianthrones J (1d)
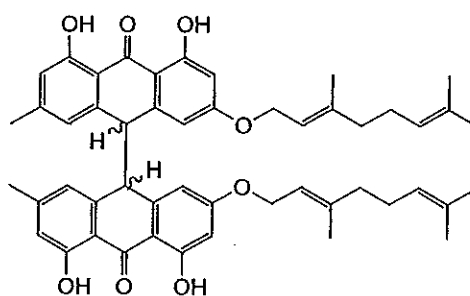
bianthrone A₁ (2d)

e) Vismones
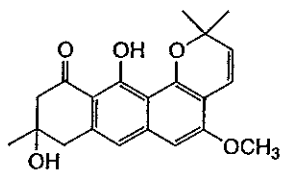
vismone B (1e)
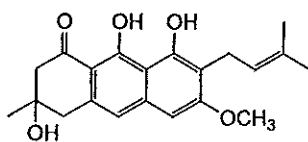
vismone E (2e)
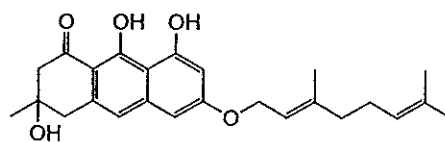
vismone D (3e)

f) Anthraquinones
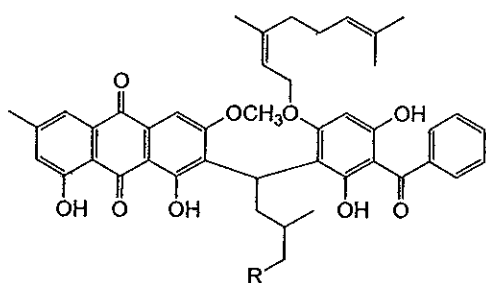
R = H ; cratoxyarborequinone A (1f)
R = isoprenyl ; cratoxyarborequinone A (2f)

R = geranyl
3-geranyloxy-6-methyl-1,8-dihydroxyanthraquinone (3f)
R = isoprenyl ; madagascin (4f)
R = CH₃ ; physcion (5f)
R = H ; emodin (6f)
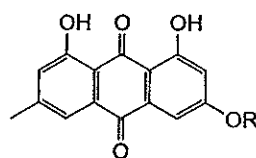
11-hydroxy-5-methoxy-2,2,9-
trimethyl-2H-anthra-[1,2-b]-
pyran-7,12-dione (7f)
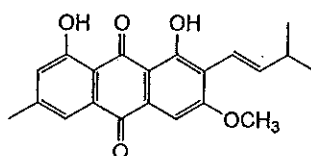
vismiaquinone (8f)
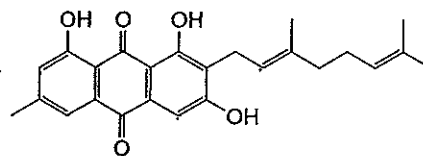
2-geranylemodin (9f)

g) Tocotrienols
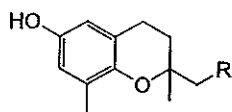
R = farnesyl ; δ-tocotrienol (1g)
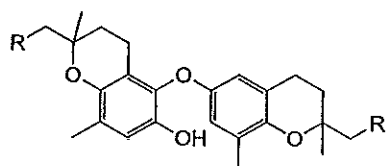
R = farnesyl ; δ-tocotrienol dimer (2g)
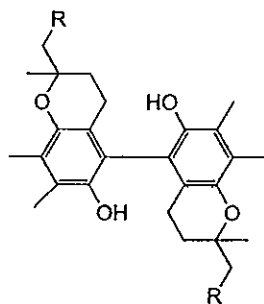
farnesyl =

R = farnesyl ; 5-(γ-tocotrienyl)-γ-tocotrienol (3g)
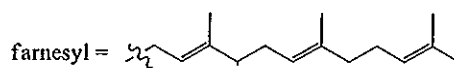
h) Bicyclic triterpenoids
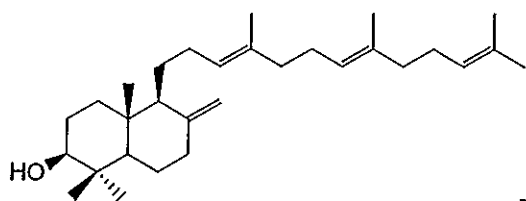
polypoda-8(26),13,17,21-tetraen-3-ol (1h)
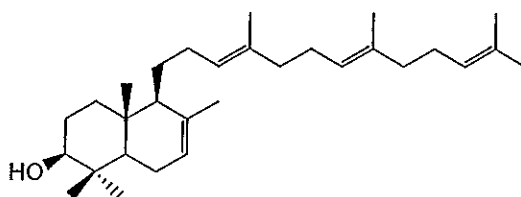
(13E,17E)-polypoda-7,13,17,21-tetraen-3-ol (2h)

i) Triterpenoids
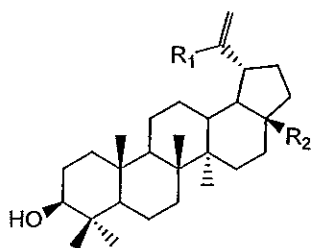
R$_1$ = CH$_3$ R$_2$ = CH$_3$; lupeol (1i)

R$_1$ = CH$_3$ R$_2$ = COOH ; betulinic acid (2i)

R$_1$ = CH$_2$OH R$_2$ = CH$_3$; lup-20(29)-ene-3β,30-diol (3i)

R$_1$ = COOH R$_2$ = CH$_3$; 3β-hydroxylap-20(29)-en-30-oic acid (4i)
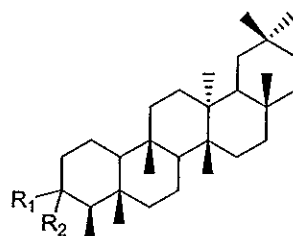
R$_1$ = R$_2$ = O ; friedelin (5i)

R$_1$ = β-OH R$_2$ = H ; friedelinol (6i)

1.9 The objectives of this study

The aims of this work were to investigate the chemical constituents from the green fruits and resin of *Cratoxylum cochinchinense* and also the green fruits and roots of *C. formosum* ssp. *pruniflorum*. All of isolated compounds from *Cratoxylum cochinchinense* and *C. formosum* ssp. *pruniflorum* were evaluated for their antibacterial, antifungal and nitric oxide inhibitory activities.

CHAPTER 2

EXPERIMENTAL

2.1 Instruments and chemicals

Melting points were determined on a Fisher-John melting point apparatus. Optical rotations were measured on a JASCO P-1020 digital polarimeter. UV and IR spectra were recorded on SPECORD S 100 (Analytikjena) and Perkin-Elmer FTS FT-IR spectrophotometer, respectively. The ^{1}H and ^{13}C NMR spectra were recorded on 300, 400 and 500 MHz Bruker FTNMR Ultra Shield^{TM} spectrometers in CD$_3$OD, d_6-acetone, CDCl$_3$ with TMS as the internal standard. Chemical shifts are reported in δ (ppm) and coupling constants (J) are expressed in Hertz. HREI and EI mass spectra were measured on a Kratos MS 25 RFA spectrometer. Crystallographic data were collected at 100.0 (1) K with the Oxford Cryosystem Cobra low-temperature attachment. The data were collected using a Bruker Apex2 CCD diffractometer with a graphite monochromated MoKα radiation at a detector distance of 5 cm and swing angle of –35°. A hemisphere of the reciprocal space was covered by a combination of four sets of exposures using *SMART* program. The collected data were reduced using *SAINT* program, and the empirical absorption corrections were performed using *SADABS* program. The structures were solved by direct methods and refined by least-squares using the *SHELXTL* software package. All non-hydrogen atoms were refined anisotropically, whereas all H atoms were placed in calculated positions with an O-H distance of 0.82 Å and C-H distances in the range 0.93-0.98 Å after checking their positions in the difference map. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atoms for methyl H atoms and $1.2U_{eq}$ for hydroxyl and the other H atoms. The final refinement converged well. Materials for publication were prepared using *SHELXT* and *PLATON*. All the bacteria images were viewed with a JSM-5800LV, JEOL SEM (scanning electron microscope). Quick column chromatography (QCC) and column chromatography (CC) were carried out on silica gel 60 F$_{254}$ (Merck) and silica gel 100 (Merck), respectively.

2.2 Plants material

2.2.1 The resin and green fruits of Cratoxylum cochinchinense

The resin of *C. cochinchinense* was collected in October 2003 at Prince of Songkla University, Hat-Yai campus, whereas the green fruits of *C. cochinchinense* were collected in

October 2007 at Kaun Kha Long District, Satun Province, Southern part of Thailand. Botanical identification was achieved through comparison with a voucher specimen No. SL-1 (PSU) in the herbarium of Department of Biology, Prince of Songkla University, Songkhla, Thailand.

2.2.2 The roots and green fruits of Cratoxylum formosum ssp. pruniflorum

The roots of *C. formosum* ssp. *pruniflorum* were collected in May 2004 from Nong Khai Province, whereas the green fruits of *C. formosum* ssp. *pruniflorum* were collected in August 2008 from Pha Yao Province, northern part of Thailand. Botanical identification was carried out by comparison with a voucher specimen number 0012677 in the herbarium collection of Department of Biology, Faculty of Science, Prince of Songkla University, Thailand.

2.3 Plants extraction

2.3.1 The extraction of the resin of Cratoxylum cochinchinense

The resin of *C. cochinchinense* (87.75 g) was extracted with CH_2Cl_2 (2×2.0 L, for a week) at room temperature and was evaporated under reduced pressure to afford a deep green crude CH_2Cl_2 extract (47.04 g) (see **Scheme 1**).
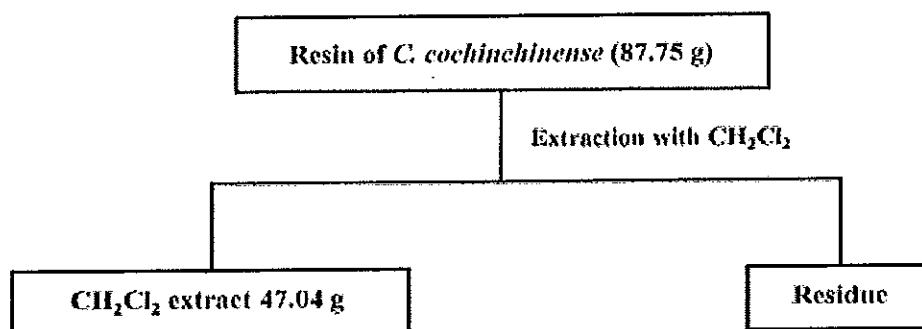
Scheme 1 The extraction of the resin of *C. cochinchinense*

2.3.2 The extraction of the green fruits of Cratoxylum cochinchinense

Air-dried green fruits of *C. cochinchinense* (5.5 kg) were extracted with CH_2Cl_2 (2×20 L, for a week) at room temperature and was evaporated under reduced pressure to afford a deep green crude CH_2Cl_2 extract (40.04 g) (see **Scheme 2**).

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┌─────────────────────────────────────────────────────────┐
│  Air-Dried Green Fruits of C. cochinchinense (5.5 kg)   │
└─────────────────────────────────────────────────────────┘
                            │
                   Extraction with CH₂Cl₂
          ┌─────────────────┴─────────────────┐
┌───────────────────────────┐      ┌───────────────────┐
│  CH₂Cl₂ extract 40.04 g   │      │      Residue      │
└───────────────────────────┘      └───────────────────┘
```
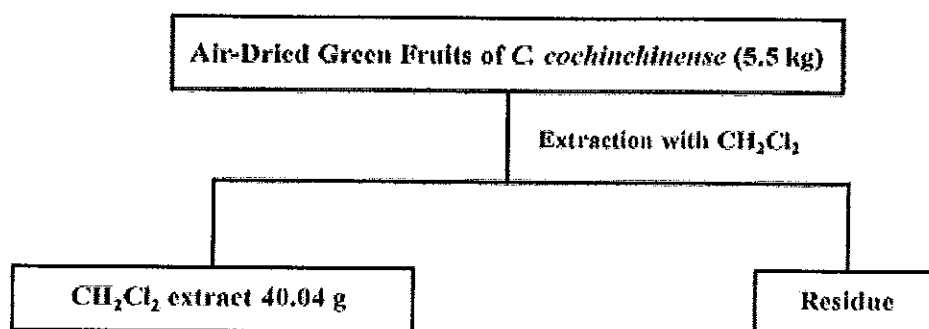
Scheme 2 The extraction of the green fruits of *C. cochinchinense*

2.3.3 The extraction of the roots of Cratoxylum formosum ssp. pruniflorum

The air-dried roots of *C. formosum* ssp. *pruniflorum* (5.0 kg) was extracted with CH_2Cl_2 (2×20 L, for a week) at room temperature and was further evaporated under reduced pressure to afford a deep green crude CH_2Cl_2 extract (58.87 g) (see **Scheme 3**).

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┌─────────────────────────────────────────────────────────┐
│  Air-Dried Roots of C. formosum ssp. formosum (5.0 kg)  │
└─────────────────────────────────────────────────────────┘
                            │
                   Extraction with CH₂Cl₂
          ┌─────────────────┴─────────────────┐
┌───────────────────────────┐      ┌───────────────────┐
│  CH₂Cl₂ extract 58.87 g   │      │      Residue      │
└───────────────────────────┘      └───────────────────┘
```
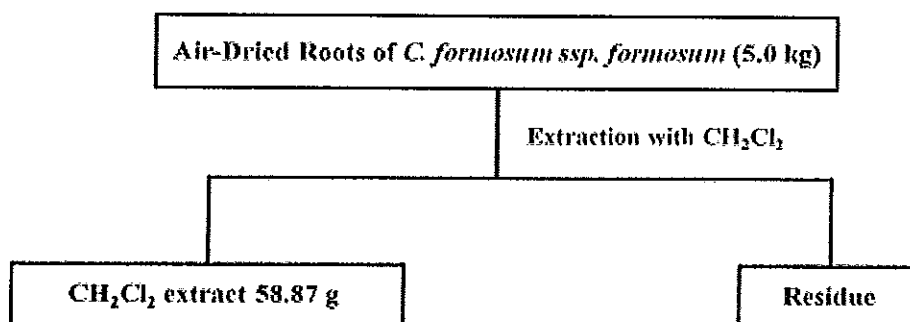
Scheme 3 The extraction of the roots of *C. formosum* ssp. *pruniflorum*

2.3.4 The extraction of the green fruits of Cratoxylum formosum ssp. pruniflorum

The green fruits of *C. formosum* ssp. *pruniflorum* (5.0 kg) was extracted with CH_2Cl_2 and CH_3OH (each 2×20 L, for a week) successively at room temperature and were further evaporated under reduced pressure to afford the crude extracts of CH_2Cl_2 (31.42 g) and CH_3OH (65.74 g) respectively (see **Scheme 4**).
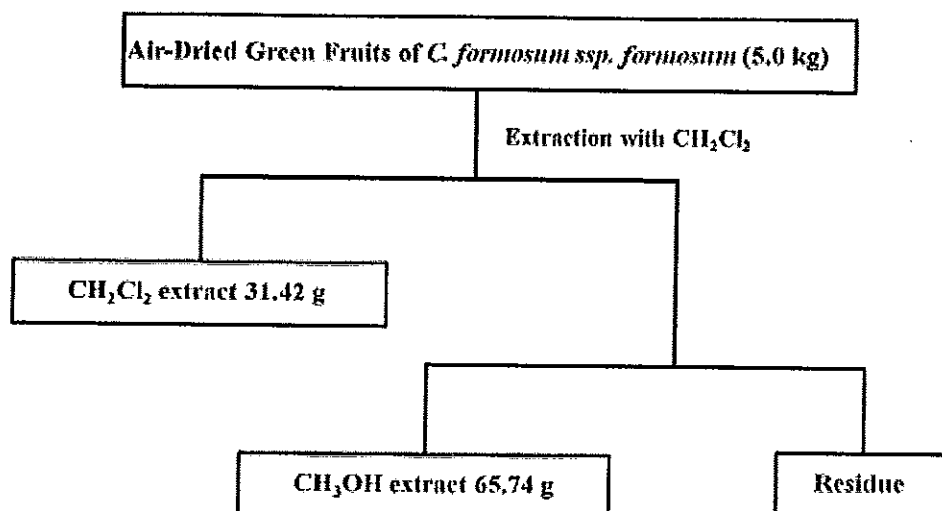
Scheme 4 The extraction of the green fruits of *C. formosum* ssp. *pruniflorum*

2.4 Isolation and chemical investigation

2.4.1 The CH₂Cl₂ extract of the resin of Cratoxylum cochinchinense

The crude CH_2Cl_2 extract (47.04 g) of the resin of *C. cochinchinense* was subjected to QCC (Quick column chromatography) on silica gel (Merck 60 F_{254}) using hexane as a first eluent and then increasing the polarity with acetone to give 16 fractions (FR1-FR16). Fractions FR4 and FR5 were separated by CC eluting with a gradient of acetone-hexane to give 8 subfractions (FR4A-FR4H) and **CC8** (150.2 mg). Subfraction FR4B was further purified by CC and eluted with a gradient of acetone-hexane to give **CC7** (31.4 mg), **CC12** (56.5 mg), **CC21** (24.5 mg) and a mixture of compounds **CC22** and **CC23** (78.5 mg), respectively. Fractions FR6 and FR7 were separated by QCC and eluted with a gradient of CH_2Cl_2-hexane to give 6 subfractions (FR6A-FR6F). Subfraction FR6B was further separated by QCC eluting with a gradient of acetone-hexane to give **CC6** (35.7 mg), **CC8** (83.2 mg) and **CC11** (1.8 mg). Subfraction FR6E was purified by CC on reversed-phase silica gel C-18 eluting with MeOH to give **CC4** (849.4 mg) and **CC5** (1.25 g). Fraction FR8 and FR9 were separated by QCC and eluted with a gradient of CH_2Cl_2-hexane to afford **CC5** (551.3 mg), **CC7** (18.2 mg), **CC8** (116.6 mg) and **CC9** (148.7 mg). Fraction FR10 and FR11 were separated by QCC and eluted with a gradient of acetone-hexane to give 7 subfractions (FR10A-FR10G) and **CC7** (25.9 mg). Subfraction FR10B was further purified by CC on silica gel C-18 and eluted with MeOH to furnish **CC1** (8.5 mg). Subfraction FR10D was separated by QCC eluting with a gradient of acetone-hexane to give 6 subfractions (FR10D1-

FR10D6). Subfraction FR10D2 was further separated by CC and eluted with a gradient of acetone-hexane to give 5 subfractions (FR10D2A-FR10D2E), CC2 (1.8 mg), CC4 (23.1 mg), CC5 (34.2 mg) and an inseparable mixture of CC2 and CC3 (24.2 mg). The mixture was separated by acetylation with Ac$_2$O (0.1 mL) in pyridine (2.0 mL) and stirred over night at the room temperature to give yellow gum which was further purified by CC eluting with 70% CHCl$_3$-hexane to give acetylated derivatives CC2a (5.1 mg) and CC3a (18.9 mg), respectively. Subfraction FR10D2E was purified by CC and eluted with 80% CH$_2$Cl$_2$-hexane to give CC8 (16.7 mg), CC10 (5.0 mg) and CC11 (1.5 mg), respectively (see Scheme 5).

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                    ┌────────────────────────────────┐
                    │  Crude CH₂Cl₂ extract 47.04 g  │
                    └────────────────────────────────┘
                                    │
   ┌──────────┬──────────┬──────────┼──────────┬──────────┬──────────┐
   ↓          ↓          ↓          ↓          ↓          ↓
 FR1-FR3   FR4-FR5    FR6-FR7    FR8-FR9   FR10-FR11  FR12-FR16
               │          │          │          │
               ↓          ↓          ↓          ↓
            CC7  CC21    CC4        CC9        CC1
            CC8  CC22    CC5                   CC2
            CC12 CC23    CC6                   CC3
                         CC11                  CC10
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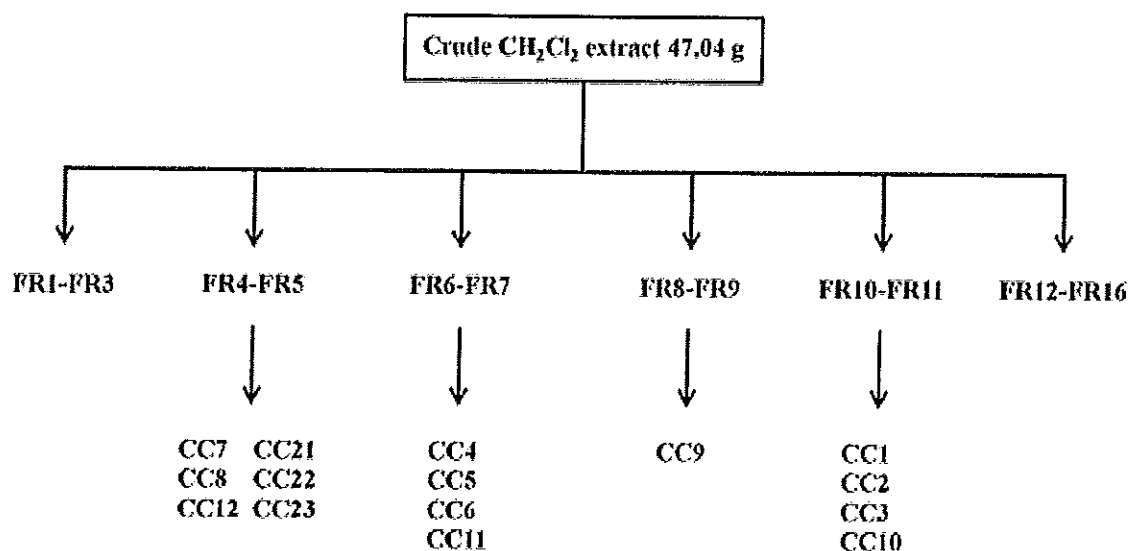
Scheme 5 Isolation of compounds **CC1-CC12** and **CC21-CC23**

Compound CC1: *Cochinchinone I.* Yellow needle single crystals, m.p. 160-162 °C; UV-Vis (CHCl$_3$) λ_{max} (log ε) 261 (4.01), 297 (4.31), 342 (3.69), 393 (3.46) nm; FT-IR (neat) v_{max} 3406, 1650, 1612 cm^{-1}; HRMS m/z 446.2279 for C$_{28}$H$_{30}$O$_5$ (calcd. 446.2093). EIMS m/z (rel. int.): 446 [M]$^+$ (76), , 431 (100), 377 (73), 363 (76), 323 (26), 307 (15), 295 (13), 137 (5), 69 (6). For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR (CDCl$_3$) spectroscopic data see **Table 2.** (Boonnak *et al.*, 2009)

Compound CC2: *Cochinchinone J.* Yellow viscous oil, [α]^{25}$_D$ = -69.8 (*c* 0.08, CHCl$_3$); UV-Vis (CHCl$_3$) λ_{max} (log ε) 243 (3.90), 289 (4.10), 298 (4.13), 320 (3.68), 351 (3.47), 391 (3.33) nm; FT-IR (neat) v_{max} 3397, 1649, 1613 cm^{-1}; HRMS m/z 446.2092 for C$_{28}$H$_{30}$O$_5$ (calcd. 446.2093). EIMS m/z (rel. int.): 446 [M]$^+$ (11), 363 (100), 307 (18), 69 (5). For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR (CDCl$_3$) spectroscopic data see **Table 3.** (Boonnak *et al.*, 2009)

Compound **CC3**: *Cochinchinone K.* Compound **CC3** was isolated as a mono-acetylated form (**CC3a**) from an inseparable mixture with compound **CC2**, and the mixture was thus acetylated with Ac_2O in pyridine. The resulting product was further separated by CC eluting with 70% $CHCl_3$-hexane to give monoacetates **CC3a** (18.9 mg) and **CC2a** (5.1 mg), respectively. The latter was confirmed as an acetylated derivative of **CC2** (**CC2a**) by comparison of its spectral data with those of compound **CC2**. Yellow powder, m.p. 85-87 °C; UV (CHCl₃) λ_{max} (log ε) 243, 271, 303, 326 and 383 nm; IR (neat) v_{max} 3392, 1709, 1648, 1612 cm⁻¹; HRMS *m/z* 490.2355 for $C_{30}H_{34}O_6$ (calcd. 490.2355). EIMS *m/z* (rel. int.): 490 [M]⁺ (53), 473 (53), 419 (44), 405 (31), 365 (100), 323 (52), 311 (41), 267 (51), 69 (24). For ¹H (300 MHz) and ¹³C (75 MHz) NMR (CDCl₃) spectroscopic data see **Table 4**. (Boonnak *et al.*, 2009)

Compound **CC4**: *Cochinchinone A.* Pale-yellow powder, m.p. 119-120 °C; UV-Vis (CHCl₃) λ_{max} (log ε) 232 (4.44), 268 (4.42), 316 (4.04), 384 (3.70) nm; FT-IR (neat) v_{max} 3413, 1641 cm⁻¹; For ¹H (300 MHz) and ¹³C (75 MHz) NMR (CDCl₃) spectroscopic data see **Table 5**. (Mahabussarakam *et al.*, 2006)

Compound **CC5**: *1,3,7-trihydroxy-2,4-diisoprenylaxanthone.* Pale-yellow powder. UV-Vis (CHCl₃) λ_{max} (log ε) 234, 267, 317, 386 nm; FT-IR (neat) v_{max} 3400, 1645 cm⁻¹; For ¹H (300 MHz) and ¹³C (75 MHz) NMR (CDCl₃) spectroscopic data see **Table 6**. (Iinuma *et al.*, 1996; Nguyen and Harrison 1998)

Compound **CC6**: *Celebixanthone methyl ether.* Yellow needle-single crystals, m.p. 172-174 °C; UV-Vis (CHCl₃) λ_{max} (log ε) 243 (3.90), 289 (4.10), 298 (4.13), 320 (3.68), 351 (3.47), 391 (3.33) nm; FT-IR (neat) v_{max} 3397, 1649, 1613 cm⁻¹; For ¹H (300 MHz) and ¹³C (75 MHz) NMR (CDCl₃) spectroscopic data see **Table 7**. (Stout *et al.*, 1963; Dechathai *et al.*, 2006; Boonnak *et al.*, 2007)

Compound **CC7**: *Dulcisxanthone F.* Yellow needle-single, m.p. 213-215 °C; UV-Vis (CHCl₃) λ_{max} (log ε) 244 (4.54), 265 (4.53), 322 (4.43), 331 (4.44) nm; FT-IR (neat) v_{max} 3479, 1675 cm⁻¹; For ¹H (300 MHz) and ¹³C (75 MHz) NMR (CDCl₃) spectroscopic data see **Table 8**. (Dechathai *et al.*, 2006; Boonnak *et al.*, 2006)

Compound **CC8**: *β-Mangostin.* Yellow needle-single crystals, m.p. 172-174 °C; UV-Vis (CHCl₃) λ_{max} (log ε) 243 (3.90), 289 (4.10), 298 (4.13), 320 (3.68), 351 (3.47), 391 (3.33)

nm; FT-IR (neat) v_{max} 3397, 1649, 1613 cm^{-1}; For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR (CDCl$_3$) spectroscopic data see **Table 9**. (Mahabusarakam *et al.*, 1987; Chantrapromma *et al.*, 2006)

Compound CC9: *α-Mangostin*. Deep-yellow powder, m.p. 180-182 °C; UV-Vis (CHCl$_3$) λ$_{max}$ (log ε) 243 (3.90), 289 (4.10), 298 (4.13), 320 (3.68), 351 (3.47), 391 (3.33) nm; FT-IR (neat) v_{max} 3397, 1649, 1613 cm^{-1}; For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR (CDCl$_3$) spectroscopic data see **Table 10**. (Mahabusarakam *et al.*, 1987)

Compound CC10: *Macluraxanthone*. Brown-yellow solid. m.p. 183-184 °C; UV-Vis (CHCl$_3$) λ$_{max}$ (log ε) 240 (4.28), 283 (4.62), 338 (4.25) nm; FT-IR (neat) v_{max} 3446, 1649 cm^{-1}; For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR (CDCl$_3$) spectroscopic data see **Table 11**. (Delle Monache *et al.*, 1981)

Compound CC11: *Pruniflorone G*. Brown powder, m.p. 143-145 °C; $[\alpha]^{27}_D$ = -7.4 (c 0.425, CHCl$_3$); UV-Vis (CHCl$_3$) λ$_{max}$ (log ε) 243 (4.56), 288 (4.81), 335 (4.53) nm; FT-IR (neat) v_{max} 3414, 1649, 1628, 1580 cm^{-1}; For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR (CDCl$_3$) spectroscopic data see **Table 12**. (Boonnak *et al.*, 2006)

Compound CC12: *Cochinchinone C*. Yellow needle crystals, m.p. 158-159 °C; $[\alpha]^{25}_D$ = +125.1 (c 0.14, CHCl$_3$), UV-Vis (CHCl$_3$) λ$_{max}$ (log ε) 262 (3.28), 310 (4.06), 350 (3.82), 400 (3.35) nm; FT-IR (neat) v_{max} 3428, 1746, 1644, 1604 cm^{-1}; For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR (CDCl$_3$) spectroscopic data see **Table 13**. (Mahabussarakam *et al.*, 2006).

Compound CC21: *Friedelin*. White crystal, m.p. 245-247 °C. FT-IR (neat) v_{max} 1715 cm^{-1}; For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR (CDCl$_3$) spectroscopic data see **Table 22**. (Ahad *et al.*, 1991)

Compound CC22 and CC23: a mixture of β-sitosterol (**CC22**) and stigmasterol (**CC23**). White crystal (Thongdeeying, 2005).

2.4.2 The CH₂Cl₂ extract of the green fruits of Cratoxylum cochinchinense

The crude CH₂Cl₂ extract (40.04 g) of the green fruits of *C. cochinchinense* was subjected to QCC on silica gel using hexane as a first eluent and increasing polarity with EtOAc to give 9 fractions (FF1-FF9). Fraction FS5 was purified by CC eluting with pure CHCl₃ to give **CC14** (1.88 g). Fraction FF6 was further separated by CC eluting with pure CHCl₃ to furnish 6 subfractions (FF6A-FF6F), **CC14** (2.10 g) and **CC15** (490.2 mg)., respectively. Subfraction FF6B was further purified by CC eluting with a gradient of acetone-hexane to give **CC13** (53.3 mg) (see **Scheme 6**).
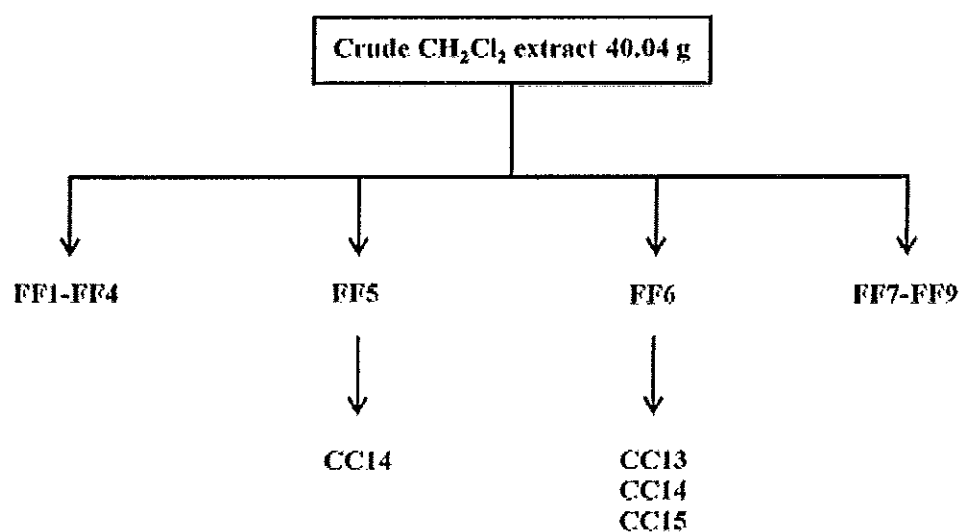
Scheme 6 Isolation of compounds **CC13-CC15**

Compound **CC13**: *Cochinchinone L.* Yellow powder, m.p. 114-116 °C; UV-Vis (CHCl₃) λ_{max} (log ε) 248 (4.59), 273 (4.03), 305 (4.13), 354 (3.80) nm; FT-IR (neat) v_{max} 3237, 1774, 1728, 1628 cm⁻¹; HRMS *m/z* 422.1718 for C₂₅H₂₆O₆ (calcd. 422.1729). EIMS *m/z* (rel. int.): 422 [M]⁺ (1), 286 (40), 244 (100), 187 (4), 81 (9), 69 (28). For ¹H (300 MHz) and ¹³C (75 MHz) NMR (CDCl₃) spectroscopic data see **Table 14**. (Boonnak *et al.*, 2009)

Compound **CC14**: *7-geranyloxy-1,3-dihydroxyxanthone.* Yellow powder, m.p. 138-140 °C; UV-Vis λ_{max} (log ε) 206 (4.25), 236 (4.48), 260 (4.39), 316 (3.89), 364 (3.94) nm; FT-IR (KBr) v_{max} 3162, 1652 cm⁻¹. For ¹H (300 MHz) and ¹³C (75 MHz) NMR (CDCl₃) spectroscopic data see **Table 15**. (Nguyen and Harrison, 1998)

Compound **CC15**: *Cochinchinone G*. Yellow powder, m.p. 147-148 °C; UV-Vis (CHCl$_3$) λ_{max} (log ε) 203 (4.49), 229 (4.30), 259 (4.31), 307 (3.99), 374 (3.63) nm; FT-IR (KBr) v_{max} 3288, 1647 cm^{-1}. For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR (CDCl$_3$) spectroscopic data see **Table 16**. (Mahabusarakam *et al*., 2008)

Compound **CC16**: *Mono-acetylation of CC14*. Compound **CC14** (82.5 mg) was treated with Ac$_2$O (2.5 mL) in pyridine (2.0 mL) and stirred for 6 hr at room temperature. The reaction mixture was diluted with water, extracted with CH$_2$Cl$_2$. The combined organic extract was washed with 10% HCl and then washed with water again. After the organic solvent was removed, the resulting residue was dried over anhydrous Na$_2$SO$_4$. Chromatography over silica gel yielded a pale yellow powder of **16** (80.6 mg). *Compound* **CC16**: *3-Acetoxy-7-geranyloxy-1-hydroxyxanthone*. Yellow powder, m.p. 94-95 °C; UV-Vis (CHCl$_3$) λ_{max} (log ε) 239 (4.40), 262 (4.66), 289 (3.97), 379 (3.93) nm; FT-IR (neat) v_{max} 3429, 1768, 1649, 1612 cm^{-1}; HRMS *m/z* 422.1725 for C$_{25}$H$_{26}$O$_6$ (calcd. 422.1729). EIMS *m/z* (rel. int.): 422 [M]$^+$ (1), 286 (43), 244 (100), 187 (4), 81 (9), 69 (24). For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR (CDCl$_3$) spectroscopic data see **Table 17**. (Boonnak *et al*., 2009)

Compound **CC17**: *Di-acetylation of CC14*. Compound **CC14** (200.5 mg) was treated with Ac$_2$O (6.0 mL) in pyridine (3.0 mL) and stirred overnight at room temperature. Chromatography over silica gel yielded a pale yellow powder of **CC16** (10.6 mg) and **CC17** (177.8 mg), respectively. *Compound* **CC17**: *1,3-Diacetoxy-7-geranyloxyxanthone*. Yellow powder, m.p. 96-97 °C; UV-Vis (CHCl$_3$) λ_{max} (log ε) 253 (4.59), 300 (3.45), 361 (3.86) nm; FT-IR (neat) v_{max} 3429, 1776, 1656, 1624 cm^{-1}; HRMS *m/z* 464.1838 for C$_{27}$H$_{28}$O$_7$ (calcd. 464.1835). EIMS *m/z* (rel. int.): 464 [M]$^+$ (2), 328 (3), 286 (58), 244 (100), 187 (5), 81 (17), 69 (36). For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR (CDCl$_3$) spectroscopic data see **Table 18**. (Boonnak *et al*., 2009)

Compound **CC18**: *Mono-acetylation of CC15*. Compound **CC15** (85.5 mg) was treated with Ac$_2$O (2.5 mL) in pyridine (2.0 mL) and stirred for 6 hr at room temperature. Chromatography over silica gel yielded a pale yellow powder of **CC18** (83.7 mg). *Compound* **CC18**: *7-Acetoxy-3-geranyloxy-1-hydroxyxanthone*. Yellow powder, m.p. 104-106 °C; UV-Vis (CHCl$_3$) λ_{max} (log ε) 243 (4.44), 257 (4.52), 310 (4.29), 358 (3.82) nm; FT-IR (neat) v_{max} 3429, 1758, 1665, 1607 cm^{-1}; HRMS *m/z* 422.1726 for C$_{25}$H$_{26}$O$_6$ (calcd. 422.1729). EIMS *m/z* (rel. int.): 422 [M]$^+$ (5), 286 (16), 244 (100), 187 (2), 81 (16), 69 (56).

For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR (CDCl$_3$) spectroscopic data see **Table 19**. (Boonnak *et al.*, 2009)

Compound CC19: *Di-acetylation of CC15*. Compound **CC15** (190.0 mg) was treated with Ac$_2$O (6.0 mL) in pyridine (3.0 mL) and stirred overnight at room temperature. Chromatography over silica gel yielded a pale yellow powder of **CC18** (8.7 mg) and **CC19** (170.0 mg), respectively. **Compound CC19**: *1,7-Diacetoxy-3-geranyloxyxanthone*. Yellow powder, m.p. 85-87 °C; UV-Vis (CHCl$_3$) λ_{max} (log ε) 246 (4.61), 275 (4.01), 302 (4.28), 334 (3.86) nm; FT-IR (neat) v_{max} 3453, 1770, 1655, 1629 cm^{-1}; HRMS *m/z* 464.1834 for C$_{27}$H$_{28}$O$_7$ (calcd. 464.1835). EIMS *m/z* (rel. int.): 464 [M]$^+$ (4), 328 (4), 286 (32), 244 (100), 187 (2), 81 (24), 69(73). For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR (CDCl$_3$) spectroscopic data see **Table 20**. (Boonnak *et al.*, 2009)

Compound CC20: *Di-brosylation of CC14*. Compound **CC14** (40.0 mg, 105.14 mmol) was stirred overnight at room temperature with *p*-bromobenzenesulfonyl chloride (40.30 mg, 190.2 mmol) and K$_2$CO$_3$ (44.1 mg, 315.4 mmol) in CH$_2$Cl$_2$ (3.0 mL). After the reaction was complete, water (10.0 mL) was added to the reaction mixture. The resulting solution was then extracted with CH$_2$Cl$_2$ (10.0 mL, 3 times). The combined organic extract was dried over anhydrous sodium sulfate and evaporated under reduced pressure to give a crude extract, which was further purified by column chromatography over silica gel eluting with 5% acetone–hexane to yield the dibrosylate **CC20** (75.2 mg). **Compound CC20**: *7-geranyloxy-1,3-dibrosylatedxyxanthone*. Yellow needle crystal, m.p. 106-108 °C. EIMS *m/z* (rel. int.): 816 [M-2]$^+$ (1), 683 (7), 681 (14), 679 (7), 619 (5), 617 (10), 615 (5), 461 (55), 463 (56), 399 (41), 397 (41), 371 (19), 369 (19), 357 (34), 355 (34), 244 (27), 229 (84), 215 (100), 186 (16), 157 (53), 155 (53), 131 (11), 108 (13), 76 (16). For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR (300 MHz, CDCl$_3$+CD$_3$OD) spectroscopic data see **Table 21**. (Boonnak *et al.*, 2009)

2.4.3 The CH$_2$Cl$_2$ extract of the roots of Cratoxylum formosum ssp. pruniflorum

The crude CH$_2$Cl$_2$ extract (58.87 g) of the roots of *C. formosum* spp. *pruniflorum* was subjected to QCC on silica gel using hexane as a first eluent and then increasing the polarity with acetone to give 12 fractions (FR1-FR12). Fraction FR5 was separated by QCC eluting with a gradient of CH$_2$Cl$_2$-hexane to give 9 subfractions (FR5A-FR5I), **CP18** (>350 mg) and

caged prenylated xanthone (>150 mg). Fractions FR6 and FR7 were separated by QCC eluting with a gradient of acetone-hexane to give 10 subfractions (FR6A-FR6J). Subfraction FR6B was separated by QCC and eluted with a gradient of acetone-hexane to give 11 subfractions (FR6B1-FR6B11), **CP15** (1.5 mg) and **CP16** (2.5 mg). Subfractions FR6B2 were separated by CC and eluted with 10% acetone-hexane to give 4 subfractions (FR6B2A-FR6B2D) and **CP3** (25.5 mg). Subfractions FR6B6 and FR6B7 were separated by CC and eluted with 30% CH_2Cl_2-hexane to give 9 subfractions (FR6B6A-FR6B6I), **CP1** (5.7 mg), **CP19** (3.5 mg), caged prenylated xanthone (25.3 mg) and a mixture of β-sitosterol and stigmasterol (>55.4 mg), respectively. Subfraction FR6B6E was further purified by CC on silica gel C-18 and eluted with MeOH to furnish **CP9** (7.0 mg). Subfractions FR6B8 and FR6B9 were separated by CC and eluted with 30% CH_2Cl_2-n-hexane to give 10 subfractions (FR6B8A-FR6B8J), **CP4** (1.5 mg), dulxisxanthone F (23.2 mg), pruniflorone G (3.5 mg) and pruniflorone H (7.5 mg). Subfraction FR6H was further separated by QCC eluting with a gradient of acetone-n-hexane to give 11 subfractions (FR6H1-FR6H11), **CP2** (9.7 mg), cochinchinone A (80.7 mg) and 1,3,7-trihydroxy-2,4-diisoprenylxanthone (150.2 mg). Subfraction FR6H5 was further purified by CC using 10% acetone-n-hexane as a mobile phase to give cochinchinone I (5.6 mg). Subfraction FR6I was separated by QCC eluting with a gradient of acetone-hexane to give 7 subfractions (FR6I1-FR6I7), **CP12** (3.5 mg), **CP13** (5.6 mg) and **CP14** (4.5 mg), respectively. Fractions FR8-FR11 was separated by QCC eluting with 30% EtOAc-n-hexane to give 8 subfractions (FR8A-FR8H). Subfractions FR8E and FR8F were separated by QCC and eluted with 30% EtOAc-n-hexane to obtain 20 subfractions (FR8E1-FR8E20). Subfraction FR8E10-FR8E12 were separated by QCC and eluted with a gradient of CH_2Cl_2-n-hexane to give 12 subfractions (FR8E10A-FR8E10L). Subfraction FR8E10B was further purified by CC and eluted with 5% acetone-n-hexane to give **CP11** (4.5 mg) and **CP17** (5.6 mg). Subfraction FR8E10D was separated by CC eluting with 10% acetone-n-hexane to give 8 subfractions (FR8E10D1- FR8E10D8). Subfraction FR8E10D5 was further purified by CC and eluted with a gradient of CH_2Cl_2-n-hexane to give celebixanthone methyl ether (15.3 mg) and **CP6** (3.5 mg). Subfraction FR8E10E was separated by CC and eluted with a gradient of acetone-n-hexane to give 7 subfractions (FR8E10E1- FR8E10E7) and **CP8** (2.5 mg). Subfraction FR8E10F was separated by CC and eluted with a gradient of acetone-n-hexane to give 8 subfractions (FR8E10F1- FR8E10F8). Subfraction FR8E10F6 was further separated by CC and eluted with 60% $CHCl_3$-n-hexane to give 4 subfractions (FR8E10F6A- FR8E10F6D) and a mixture of macluraxanthone and **CP10**

(35.5 mg) which was further purified by CC on reversed-phase silica gel C-18 eluting with MeOH to give macluraxanthone (21.2 mg) and **CP10** (12.0 mg). Subfraction FR8E8 was separated by CC eluting with acetone-n-hexane to give **CP5** (7.5 mg). Subfraction FR8E9-FR8E11 were separated by CC and eluted with a gradient of acetone-n-hexane to give **CP7** (15.6 mg) (see **Scheme 7**).
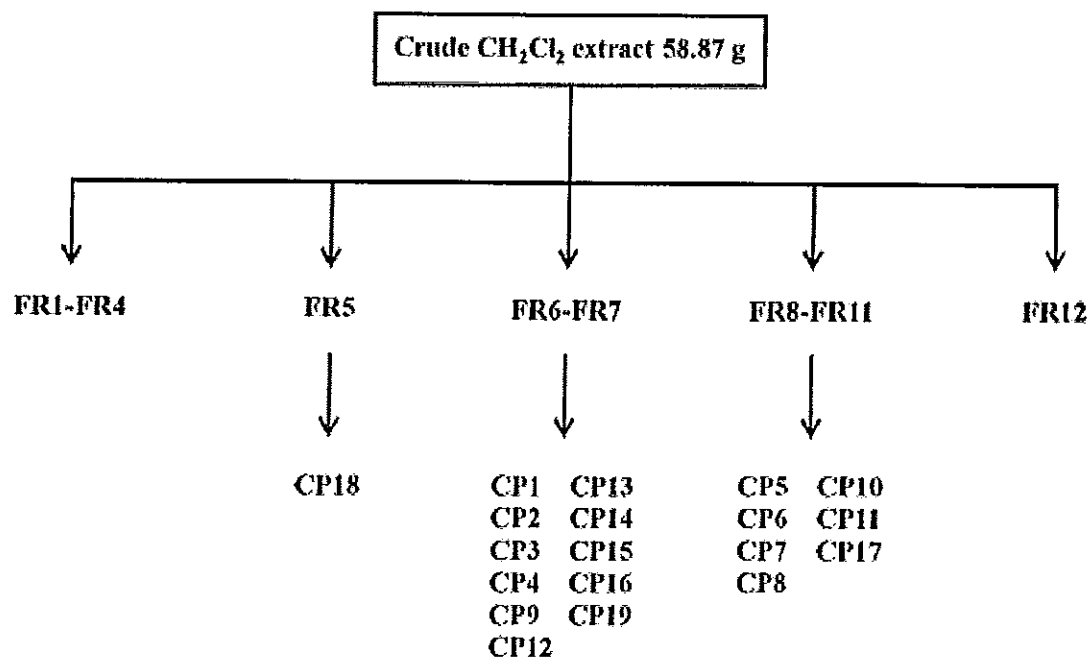
Scheme 7 Isolation of compounds CP1-CP19

Compound **CP1**: *Pruniflorone K.* Yellow viscous oil, $[\alpha]^{28}_D = -18.2$ (c 0.285, CHCl₃); UV-Vis (CHCl₃) λ_{max} (log ε) 245 (4.63), 260 (4.56), 317 (4.37), 367 (3.73) nm; FT-IR (KBr) v_{max} 3338, 1647, 1617 cm^{-1}; HREIMS m/z [M]$^+$ 446.2092 (calcd for $C_{28}H_{30}O_5$: 446.2093). EIMS m/z (rel. int.): 446 [M]$^+$ (13), 363 (100), 295 (8), 149 (14), 83 (8), 69 (13). For ^1H (300 MHz) and ^{13}C (75 MHz) NMR (CDCl₃) spectroscopic data see **Table 24**. (Boonnak *et al.*, 2010)

Compound **CP2**: *Pruniflorone L.* Pale yellow powder, mp 259-260 °C; UV-Vis (CHCl₃) λ_{max} (log ε) 245 (4.05), 268 (4.19), 317 (3.81), 388 (3.39) nm; FT-IR (KBr) v_{max} 3421, 1637 cm^{-1}; HREIMS m/z [M]$^+$ 462.2408 (calcd for $C_{29}H_{34}O_5$: 462.2406). EIMS m/z (rel. int.): 462 [M]$^+$ (100), 419 (83), 407 (74), 393 (29), 337 (100), 323 (16), 305 (23) 369 (22), 137 (10), 69 (16). For ^1H (300 MHz) and ^{13}C (75 MHz) NMR (CDCl₃) spectroscopic data see **Table 25**. (Boonnak *et al.*, 2010)

***Compound* CP3**: *Formoxanthone A.* Yellow powder, mp 111-113 °C; UV-Vis (CHCl₃) λmax (log ε) 245 (4.39), 269 (411), 332 (3.71), 377 (3.18) nm; FT-IR (KBr) νmax 3373, 1650 cm⁻¹. For ¹H (300 MHz) and ¹³C (75 MHz) NMR (CDCl₃) spectroscopic data see **Table 26**. (Boonsri *et al.*, 2006)

***Compound* CP4**: *Formoxanthone B.* Yellow powder, mp 144-146 °C; UV-Vis (CHCl₃) λmax (log ε) 253 (4.15), 260 (4.29), 319 (4.08), 367 (3.50) nm; FT-IR (KBr) νmax 3476, 1646 cm⁻¹. For ¹H (300 MHz) and ¹³C (75 MHz) NMR (CDCl₃) spectroscopic data see **Table 27**. (Boonsri *et al.*, 2006)

***Compound* CP5**: *1,7-dihydroxy-8-methoxyxanthone.* Yellow solid, mp 197-199 °C; UV-Vis (NaOH) λmax 254, 275, 350 nm; FT-IR (KBr) νmax 3330, 1647 cm⁻¹. For ¹H (300 MHz) and ¹³C (75 MHz) NMR (CDCl₃) spectroscopic data see **Table 28**. (Gottilieb *et al.*, 1966; Kijjoa *et al.*, 1997)

***Compound* CP6**: *Vieillardiixanthone B.* Yellow powder, mp 213-215 °C; UV-Vis (CH₃OH) λmax (log ε) 217 (1.87), 253 (2.57), 286 (0.80), 327 (1.34) nm; FT-IR (neat) (CH₂Cl₂) νmax 3304, 1643 cm⁻¹. For ¹H (300 MHz) and ¹³C (75 MHz) NMR (CDCl₃) spectroscopic data see **Table 29**. (Hay *et al.*, 2008; Boonnak *et al.*, 2010)

***Compound* CP7**: *Dulcisxanthone B.* Yellow powder, mp 170-172 °C; UV-Vis (CH₃OH) λmax (log ε) 209 (4.25), 244 (4.50), 261 (4.49), 317 (4.25) 368 (4.04) nm; FT-IR (KBr) νmax 3306, 1642 cm⁻¹. For ¹H (300 MHz) and ¹³C (75 MHz) NMR (CDCl₃) spectroscopic data see **Table 30**. (Dechathai *et al.*, 2005)

***Compound* CP8**: *Cochinxanthone E.* Yellow oil; UV-Vis (CH₃OH) λmax (log ε) 241 (4.20), 265 (4.18), 314 (3.92), 382 (3.43) nm; FT-IR (neat) νmax 3437, 1638 cm⁻¹. For ¹H (300 MHz) and ¹³C (75 MHz) NMR (CDCl₃) spectroscopic data see **Table 31**. (Laphookhieo *et al.*, 2009)

***Compound* CP9**: *5,9-dihydroxy-8-methoxy-2,2-dimethyl-7-(3-methyl-but-2-enyl)-2H,6H-pyrano[3,2b]xanthone.* Yellow powder, mp 156-157 °C. For ¹H (300 MHz) and ¹³C (75 MHz) NMR (CDCl₃) spectroscopic data see **Table 32**. (Sen *et al.*, 1980)

Compound **CP10**: *Garcinone B.* Yellow powder, mp 190-192 °C; UV-Vis (EtOH) λ_{max} (log ε) 247 (4.40), 267 (4.40), 339 (4.10), 390 (4.00) nm; FT-IR (KBr) v_{max} 3480, 1650 cm⁻¹. For ¹H (300 MHz) and ¹³C (75 MHz) NMR (CDCl₃) spectroscopic data see **Table 33**. (Sen *et al.*, 1982)

Compound **CP11**: *Brasilixanthone.* Yellow powder, mp 205-207 °C; UV-Vis (CH₃OH) λ_{max} (log ε) 287 (3.95), 290 (3.94), 310 (3.81), 385 (3.33) nm; FT-IR (KBr) v_{max} 3491, 3355, 1621 cm⁻¹. For ¹H (300 MHz) and ¹³C (75 MHz) NMR (CDCl₃) spectroscopic data see **Table 34**. (Marques *et al.*, 2000; Chantrapromm *et al.*, 2010)

Compound **CP12**: *3-Isomangostin.* Yellow powder, mp 154-155 °C. For ¹H (300 MHz) and ¹³C (75 MHz) NMR (CDCl₃) spectroscopic data see **Table 35**. (Mahabusarakam *et al.*, 1987)

Compound **CP13**: *3,4-Dihydro-5,9-dihydroxy-7-(3-hydroxy-3-methylbutyl)-8-metho-xy-2,2-dimethyl-2H,6H-pyrano[3,2b]xanthone.* Yellow powder; mp 180-182 °C. For ¹H (300 MHz) and ¹³C (75 MHz) NMR (CDCl₃) spectroscopic data see **Table 36**. (Dutta *et al.*, 1987)

Compound **CP14**: *3,4-Dihydro-5,9-dihydroxy-8-methoxy-7-(3-methoxy-3-methyl-butyl)-2,2-dimethyl-2H,6H-pyrano[3,2b]xanthone.* Yellow oil. For ¹H (300 MHz) and ¹³C (75 MHz) NMR (CDCl₃) spectroscopic data see **Table 37**. (Dutta *et al.*, 1987)

Compound **CP15**: *10-O-methylmacluraxanthone.* Yellow solid, mp 157-158 °C; UV-Vis (EtOH) λ_{max} (log ε) 242 (4.31), 281 (4.55), 290 (4.57), 334 (4.27) nm; FT-IR (nujol) v_{max} 3520, 1652 cm⁻¹. For ¹H NMR (300 MHz, CDCl₃) spectroscopic data see **Table 38**. (Gunasekera *et al.*, 1975)

Compound **CP16**: *Isocudraniaxanthone B.* Yellow powder, UV-Vis (CH₃OH) λ_{max} (log ε) 246, 275, 293 (*sh*), 323 (*sh*), 399 nm; FT-IR (KBr) v_{max} 3370, 1650 cm⁻¹. For ¹H NMR (300 MHz, CDCl₃) spectroscopic data see **Table 39**. (Kobayashi *et al.*, 1997)

Compound **CP17**: *Gerontoxanthone I.* Yellow solid, mp 180-181 °C; UV-Vis (CH₃OH) λ_{max} (log ε) 203 (4.26), 253 (4.42), 287 (3.92), 328 (4.09) nm; FT-IR (KBr) v_{max} 3380, 1621 cm⁻¹. For ¹H (300 MHz) and ¹³C (75 MHz) NMR (CDCl₃) spectroscopic data see **Table 40**. (Chang *et al.*, 1989)

Compound **CP18**: *Vismiaquinone A.* Red-orange powder, mp 201-203 °C; UV-Vis (CH$_3$OH) λ_{max} (log ε) 220 (3.59), 278 (3.39), 425 (3.04) nm; FT-IR (KBr) ν_{max} 3425, 1624 cm^{-1}. For ^{1}H (300 MHz) and ^{13}C (75 MHz) NMR (CDCl$_3$) spectroscopic data see **Table 41**. (Goncalves and Mors, 1981)

Compound **CP19**: *11-Hydroxy-5-methoxy-2,2,9-trimethyl-2H-anthra-[1,2b]pyran-7,12-dione.* Orange solid, mp 224-226 °C; UV-Vis (CH$_3$OH) λ_{max} (log ε) 208 (3.05), 224 (3.59), 265 (3.37), 285 (3.39), 424 (3.04) nm; FT-IR (KBr) ν_{max} 3446, 1646 cm^{-1}. For ^{1}H (300 MHz) and ^{13}C (125 MHz) NMR (CDCl$_3$) spectroscopic data see **Table 42**. (Delle Monache *et al.*, 1979)

2.4.4 The CH$_2$Cl$_2$ extract of the green fruits of *Cratoxylum formosum* ssp. *pruniflorum*

The crude CH$_2$Cl$_2$ extract (31.42 g) of the green fruits *C. formosum* spp. *pruniflorum* was further subjected to QCC on silica gel using hexane as a first eluent and then increasing the polarity with acetone to give 14 fractions (FF1-FF14). Fraction FF4 was separated by CC eluting with a gradient of acetone-hexane to give 5 subfractions (FF4A-FF4E), a mixture of β-sitosterol and stigmasterol (>45.6 mg), a mixture of **CP28** (lupeol), **CP29** (α-amyrin) and **CP30** (β-amyrin) (>1.5 g) and **CP31** (3.7 mg), respectively. Fraction FF7 was separated by CC eluting with a gradient of acetone-hexane to give 10 subfractions (FF7A-FF7J). Subfraction FF7G was separated by CC and eluted with 20% acetone-hexane to give 7 subfractions (FF7G1-FF7G7). Subfraction FF7G3 was further purified by CC on reversed-phase silica gel C-18 eluting with MeOH to give **CP25** (1.2 mg). Fraction FF10 was separated by QCC eluting with a gradient of acetone-hexane to give 17 subfractions (FF10A-FF10Q). Subfractions FF10N and FF10O were separated by CC and eluted with a gradient of EtOAc-hexane to give 8 subfractions (FF10N1-FF10N8). Subfractions FF10N1 was further purified by CC on reversed-phase silica gel C-18 eluting with MeOH to give **CP22** (1.2 mg). Subfraction FF10N2 was separated by CC and eluted with CHCl$_3$ to give **CP21** (28.0 mg) and **CP24** (1.5 mg). Subfraction FF10N6 was separated by CC and eluted with CHCl$_3$ to give **CP20** (5.3 mg) and **CP26** (15.2 mg). Fractions FF12 and FF13 were separated by QCC eluting with a gradient of acetone-hexane to give 13 subfractions (FF12A-FF12J). Subfraction FF12H was separated by CC and eluted with a gradient of acetone-hexane to give 10 subfractions (FF12H1-FF12H10). Subfractions FF12H4 and FF12H5 were further purified

by CC eluting with a gradient of EtOAc-hexane to give **CP23** (3.7 mg). Subfraction FF12I was further separated by CC eluting with a gradient of acetone-hexane to give **CP27** (3.0 mg) (see **Scheme 8**).

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                          ┌─────────────────────────────┐
                          │  Crude CH₂Cl₂ extract 31.42 g │
                          └─────────────────────────────┘
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FF1	FF2-FF3	FF4	FF5-FF6	FF7	FF8-FF9	FF10	FF11	FF12-FF13	FF14

| WAX -25 g | | CP28 CP29 CP30 CP31 | | CP25 | | CP20 CP21 CP22 CP24 CP26 | | CP23 CP27 | |
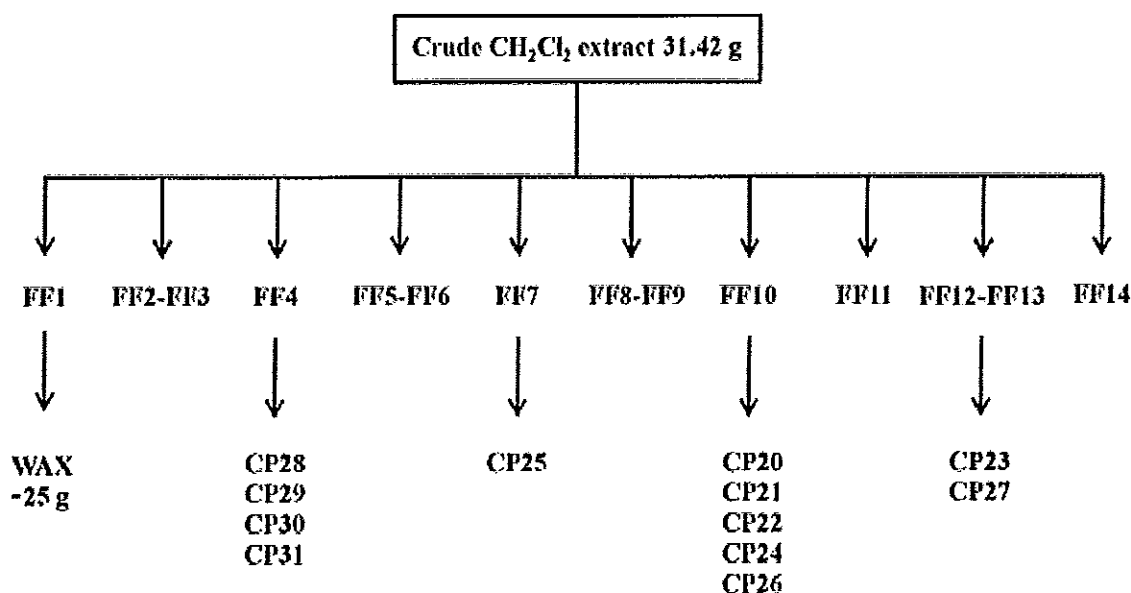
Scheme 8 Isolation of compounds **CP20-CP31**

Compound **CP20**: *Pruniflorone M*. Yellow single crystal, mp 235-237 °C; $[\alpha]^{25}_D$ = +64.6 (c 0.04, CHCl₃); UV-Vis (CHCl₃) λ_{max} (log ε) 246 (4.45), 257 (4.34), 315 (4.14), 357 (3.58) nm; FT-IR (neat) v_{max} 3368, 1648, 1587 cm⁻¹; HRMS m/z 328.0947 for C₁₈H₁₆O₆ (calcd. 328.0947). EIMS m/z (rel. int.): 328 [M]⁺ (38), 313 (93), 283 (100), 255(25), 141 (5). For ¹H (300 MHz) and ¹³C (75 MHz) NMR (d_6-acetone) spectroscopic data see **Table 43**. (Boonnak *et al.*, 2010)

Compound **CP21**: *Pruniflorone N*. Yellow powder, mp 250-252 °C; $[\alpha]^{25}_D$ = +5.2 (c 0.42, acetone); UV-Vis (CHCl₃) λ_{max} (log ε) 246 (4.32), 259 (4.21), 316 (4.02), 356 (3.39) nm; FR-IR (neat) v_{max} 3411, 1651, 1622, 1578 cm⁻¹; HRMS m/z 328.0948 for C₁₈H₁₆O₆ (calcd. 328.0947). EIMS m/z (rel. int.): 328 [M]⁺ (40), 313 (100), 285 (31), 257 (16), 243 (12), 149 (6). For ¹H (300 MHz) and ¹³C (75 MHz) NMR (d_6-acetone) spectroscopic data see **Table 44**. (Boonnak *et al.*, 2010)

Compound **CP21a**: *Hydrolysis of CP21*. A solution of **CP21** (7.8 mg) in 20% HCl–CH₃OH (2.0 mL) was left to stand for 4 days at room temperature. The solution was

evaporated in vacuum to give a residue, which was purified by CC on silica gel and eluted with 25% acetone-hexane to give compounds **CP21** (3.5 mg) and **CP21a** (3.5 mg). Compound **CP21a** was yellow powder. mp 208-210 °C; $[\alpha]^{28}_D$ = +40.8 (c 0.18, acetone). HRMS m/z 342.1091 for $C_{19}H_{18}O_6$ (calcd. 342.1103). EIMS m/z (rel. int.): 342 $[M]^+$ (42), 327 (100), 295 (68), 259 (7), 83 (4). For ^1H (300 MHz) and ^{13}C (75 MHz) NMR (d_6-acetone) spectroscopic data see **Table 45**. (Boonnak *et al.*, 2010)

Compound **CP22**: *Pruniflorone O*. Yellow viscous oil, $[\alpha]^{26}_D$ = +15.1 (c 0.04, acetone); UV-Vis (CHCl$_3$) λ_{max} (log ε) 243 (4.38), 269 (4.07), 280 (3.99), 315 (3.78), 352 (3.61) nm; Ft-IR (neat) v_{max} 3378, 1630 cm^{-1}; HRMS m/z 310.0845 for $C_{18}H_{14}O_5$ (calcd. 310.0841). EIMS m/z (rel. int.): 310 $[M]^+$ (3), 295 (4), 257 (100), 229 (4). For ^1H (300 MHz) and ^{13}C (75 MHz) NMR (d_6-acetone) spectroscopic data see **Table 46**. (Boonnak *et al.*, 2010)

Compound **CP23**: *3-Methoxy-5'-demethoxycadensin G*. Yellow powder, $[\alpha]^{26}_D$ = +53.4 (c 0.06, acetone); UV (CHCl$_3$) λ_{max} (log ε) 253 (4.37), 281 (3.83), 318 (3.97) nm; IR (neat) v_{max} 3431, 1646 cm^{-1}; HRMS m/z 452.1119 for $C_{24}H_{20}O_9$ (calcd. 452.1107). EIMS m/z (rel. int.): 452 $[M]^+$ (100), 434 (2), 420 (13), 393 (12), 315 (18), 285 (17), 274 (35), 245 (23), 180 (73), 162 (15), 137 (66), 124 (40), 119 (13), 101 (13), 77 (5). For ^1H and ^{13}C NMR spectroscopic data, see **Table 47**. (Boonnak *et al.*, 2010)

Compound **CP24**: *1,3,7-Trihydroxyxanthone*. Yellow powder, mp = 318-319 °C. For ^1H NMR spectroscopic data, see **Table 48**. (Noro *et al.*, 1984; Mondal *et al.*, 2006)

Compound **CP25**: *Osajaxanthone*. Yellow powder, mp = 266-268 °C. (Mondal *et al.*, 2006)

Compound **CP26**: *Formoxanthone C*. Yellow solid, mp 152-154 °C; $[\alpha]^{25}_D$ = -44.8 (c 0.05, CHCl$_3$); UV-Vis (CH$_3$OH) λ_{max} (log ε) 258 (4.51), 276 (4.44), 392 (3.85) nm; FT-IR (KBr) v_{max} 3440, 1646, 1624 cm^{-1}; For ^1H (300 MHz) and ^{13}C (75 MHz) NMR (CD$_3$OD+CDCl$_3$) spectroscopic data see **Table 49**. (Boonsri *et al.*, 2006)

Compound **CP27**: *Chrysoeriol*. Pale-yellow solid. For ^1H (300 MHz) and ^{13}C (75 MHz) NMR (d_6-acetone) spectroscopic data see **Table 50**. (Wagner *et al.*, 1976; Nakasuki *et al.*, 2006)

Compound **CP28, CP29** and **CP30**: *a mixture of Lupeol (CP28), α-amyrin (CP29) and β-amyrin (CP30)*. White powder, mp = 266-268 °C. (Oliveira *et al.*, 2002; Laphookhieo S. 2005; Shibuya *et al.*, 2007)

Compound **CP31**: *Taraxastane-3β,20-diol*. White powder, mp 152-154 °C; For ^{1}H (500 MHz) and ^{13}C (125 MHz) NMR (CDCl₃) spectroscopic data see **Table 51**. (Hinge *et al.*, 1966; Anjaneyulu *et al.*, 1985; Akihisa *et al.*, 2004)

2.5 Bioassay

2.5.1 Antibacterial assay

All sufficient quantity compounds isolated from the resin and green fruits of *C. cochinchinense* and the roots and green fruits of *C. formosum* ssp. *pruniflorum* were further tested against both Gram positive and Gram negative bacteria: *Bacillus subtilis, Staphylococcus aureus,* TISTR517, *Enterococcus faecalis* TISTR459, Methicillin-Resistant *Staphylococcus aureus* (MRSA) ATCC43300, Vancomycin-Resistant *Enterococcus faecalis* (VRE) ATCC 51299, *Streptococcus faecalis, Salmonella typhi, Shigella sonnei* and *Pseudomonas aeruginosa*. The microorganisms were obtained from the culture collections, Department of Industrial Biotechnology and Department of Pharmacognosy and Botany, PSU, except for the TISTR and ATCC strains, which were obtained from Microbial Research Center (MIRCEN), Bangkok, Thailand. The antibacterial assay employed was the same as described in Boonsri (Boonsri *et al.*, 2006). Vancomycin, which was used as a standard, showed antibacterial activity against Vancomycin-Resistant *Enterococcus faecalis* (VRE) ATCC 51299 at 75.0 μg/mL.

2.5.2 Antifungal assay

Candida albicans was obtained from Department of Pharmacognosy and Botany, PSU. The antifungal assay employed was the same as described in Boonsri and co-worker (Boonsri *et al.*, 2006) by using amphotencin B as a positive control.

2.5.3 Nitric oxide inhibitory activity assay

Inhibitory effect on NO production by murine macrophage-like RAW264.7 cells was evaluated using a modified method from that previously reported (Tewtrakul et al., 2009). Briefly, the RAW264.7 cell line (purchased from Cell Lines Services) was cultured in RPMI medium supplemented with 0.1% sodium bicarbonate and 2 mM glutamine, penicillin G (100 units/mL), streptomycin (100 µg/mL) and 10% FCS. The cells were harvested with trypsin-EDTA and diluted to a suspension in a fresh medium. The cells were seeded in 96-well plates with 1×10^5 cells/well and allowed to adhere for 1 h at 37°C in a humidified atmosphere containing 5% CO_2. After that the medium was replaced with a fresh medium containing 50 µg/mL of LPS together with the test samples at various concentrations (3-100 µg/mL for crude extract and 3-100 µM for pure compounds) and was then incubated for 24 h. NO production was determined by measuring the accumulation of nitrite in the culture supernatant using the Griess reagent. Cytotoxicity was determined using the MTT colorimetric method. Briefly, after 24 h incubation with the test samples, MTT solution (10 µL, 5 mg/mL in PBS) was added to the wells. After 4 h incubation, the medium was removed, and isopropanol containing 0.04 M HCl was then added to dissolve the formazan production in the cells. The optical density of the formazan solution was measured with a microplate reader at 570 nm. The test compounds were considered to be cytotoxic when the optical density of the sample-treated group was less than 80% of that in the control (vehicle-treated) group. L-NA, CAPE and indomethacin were used as positive controls. The stock solution of each test sample was dissolved in DMSO, and the solution was added to the medium RPMI (final DMSO is 1%). Inhibition (%) was calculated using the following equation and IC_{50} values were determined graphically (n = 4):

$$\text{Inhibition (\%)} = \frac{A - B}{A - C} \times 100$$

A-C : NO_2^- concentration (µM) [A : LPS (+), sample (-); B : LPS (+), sample(+); C : LPS (-), sample (-)].

CHAPTER 3

RESULTS AND DISCUSSIONS

3.1 Isolated compounds from the resin and green fruits of Cratoxylum cochinchinense

The resin of *C. cochinchinense* (87.75 g) was extracted with CH_2Cl_2 (2×2.0 L, for a week) at room temperature and was evaporated under reduced pressure to afford a deep green crude CH_2Cl_2 extract (47.04 g), which was further subjected to chromatography and/or recrystallization to yield three new xanthones: **CC1-CC3**, (Compound **CC3** was isolated as an acetylated form (**CC3a**) from the inseparable mixture with **CC2**.), together with eight known xanthones: **CC4-CC11**, a known caged-xanthone: **CC12**, a known triterpene: **CC21** and a known mixture of steroids: **CC22-CC23**.

CC1

CC2 : R = H
CC2a : R = Ac

CC3 : R = H
CC3a : R = Ac

CC4

CC5

CC6

CC7

CC8

CC9

CC10

CC11
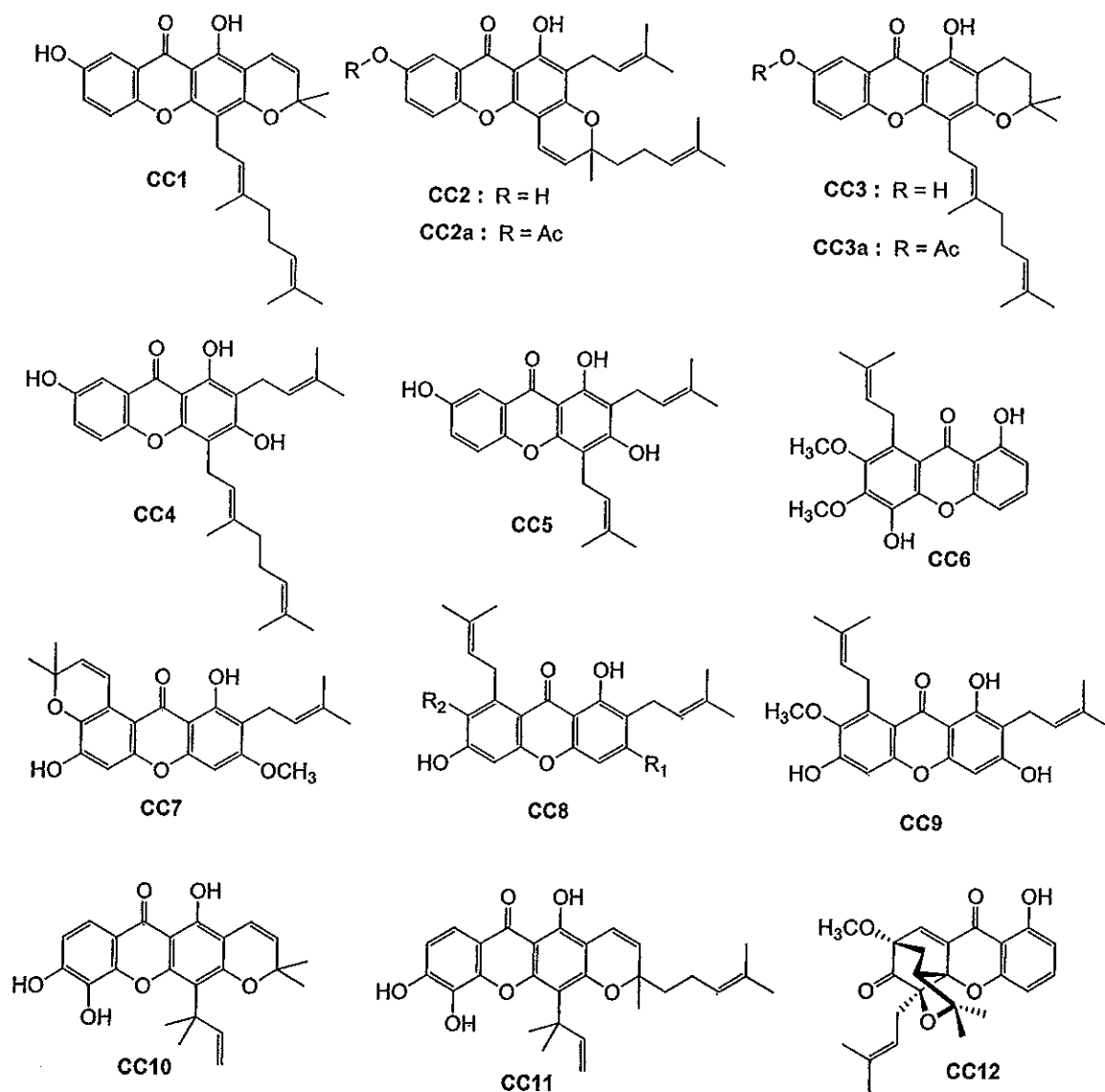
CC12

The structures of **CC1-CC12**
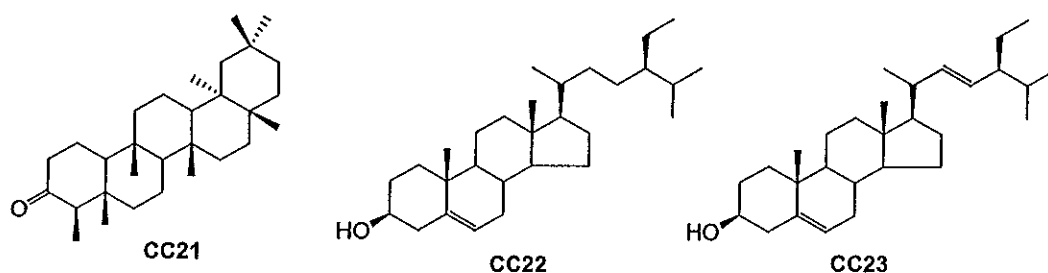
The structures of **CC21-CC23**

Air-dried green fruits of *C. cochinchinense* (5.5 kg) were extracted with CH_2Cl_2 (2×20 L, for a week) at room temperature and was evaporated under reduced pressure to afford a deep green crude CH_2Cl_2 extract (40.04 g), which was further subjected to chromatography and/or recrystallization to yield a new xanthone: **CC13**, along with two known xanthones: **CC14-CC15**. In addition, four new acetylated compounds **CC16-CC19** were derivatized from **CC14** and **CC15**. The structures of **CC1**, **CC6**, **CC18** (monoacetate of **CC15**) and **CC20**, (dibrosylate of **CC14**) were also confirmed by X-ray diffraction analysis.
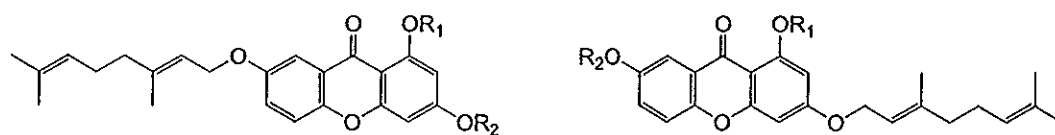
CC13:	R_1 = Ac	R_2 = H	**CC15:**	R_1 = H	R_2 = H
CC14:	R_1 = H	R_2 = H	**CC18:**	R_1 = H	R_2 = Ac
CC16:	R_1 = H	R_2 = Ac	**CC19:**	R_1 = Ac	R_2 = Ac
CC17:	R_1 = Ac	R_2 = Ac			
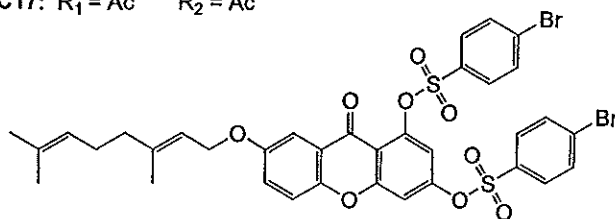
CC20

The structures of **CC13-CC20**

The structures of compounds **CC1-CC23** were completely characterized on the basis of spectroscopic analyses: UV, FT-IR, 1D NMR, 2D NMR, MS and single crystal X-ray structure determination and also comparison of their spectroscopic data with those reported in the literature.

3.1.1 Structural elucidation of compounds CC1-CC23

3.1.1.1 Compound CC1
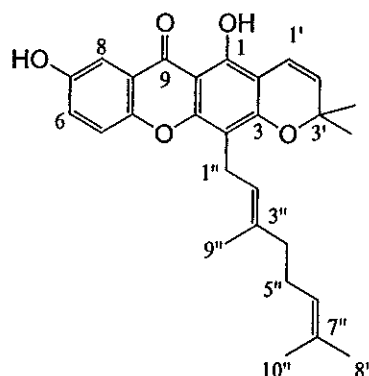
Compound **CC1** was isolated as yellow needle crystals, m.p. 160-162 °C. A molecular ion peak at m/z 446.2279 $[M]^+$ in the HREIMS established the molecular formula of $C_{28}H_{30}O_5$. The UV-Vis spectrum showed absorption bands at 261, 297, 342 and 393 nm (Boonnak *et al.*, 2009), which indicated a typical xanthone chromophore (Seo *et al.*, 2002). The FT-IR spectrum exhibited conjugated carbonyl group at 1650 cm^{-1} and hydroxyl group at 3406 cm^{-1}.

The ^{1}H and ^{13}C NMR spectral data (**Table 2**) of **CC1** were comparable to **CC4** (**Table 5**), which was isolated as a major component from the resin of *C. cochinchinense*. The main difference was observed at C-2 and C-3, where the ^{1}H NMR spectral data of **CC1** showed the signals of a chromene ring at δ 6.74 (d, J = 9.9 Hz, H-1'), 5.60 (d, J = 9.9 Hz, H-2') and 1.48 (s, CH$_3$-4' and CH$_3$-5') instead of an isoprenyl group at C-2 and a free hydroxyl group at C-3 as in **CC4** (**Table 5**). The chromene ring was connected to a xanthone skeleton in a linear fashion whose structure was confirmed by HMBC correlations as shown in **Figure 23**. Finally, the structure of **CC1** was further investigated by single-crystal X-ray diffraction analysis as shown in **Figure 24** (Boonnak *et al.*, 2009). Therefore, compound **CC1** was a new compound, and designated as cochinchinone I (Boonnak *et al.*, 2009).
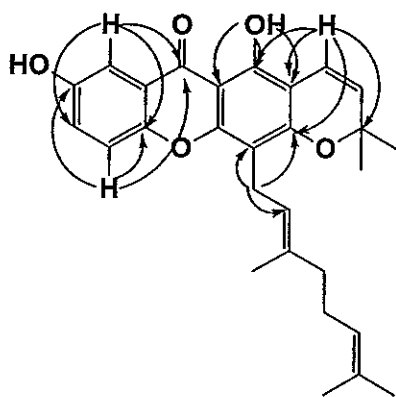
Figure 23. Selected HMBC correlations of **CC1**
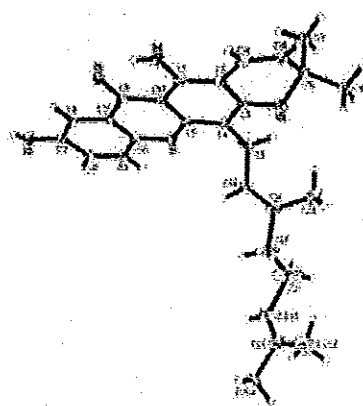
Figure 24. *ORTEP* plot of **CC1**

Table 2 NMR spectroscopic data of **CC1** in CDCl₃

Position	Type of C	$\delta_H{}^a$ (*J* in Herz)	$\delta_C{}^b$	HMBC ($^1H \rightarrow {}^{13}C$)
1-OH	C	12.97, *s*	155.5	C-1, C-2, C-9a
2	C		104.2	
3	C		158.4	
4	C		107.4	
5	CH	7.31, *d*, 9.0	119.0	C-7, C-9, C-4b, C-8a
6	CH	7.23, *dd*, 9.0, 2.4	124.1	C-5, C-7, C-8, C-4b
7	C		150.5	
8	CH	7.56, *d*, 2.4	108.9	C-6, C-7, C-9, C-4b
9	C=O		180.9	
4a	C		154.5	
4b	C		152.4	
8a	C		120.6	
9a	C		103.3	
1'	CH	6.74, *d*, 9.9	115.8	C-1, C-2, C-3, C-3', C-4', C-5'
2'	CH	5.60, *d*, 9.9	127.3	C-2, C-3', C-4', C-5'
3'	C		78.1	
4'	CH₃	1.48, *s*	28.4	C-2', C-3'
5'	CH₃	1.48, *s*	28.4	C-2', C-3'
1''	CH₂	3.46, *d*, 7.5	21.3	C-3, C-4, C-2''
2''	CH	5.22, *br t*, 7.2	122.1	C-4, C-1'', C-4'', C-9''
3''	C		135.0	
4''	CH₂	1.99, *m*	39.7	C-2'', C-3'', C-5''
5''	CH₂	2.04, *m*	26.6	C-3'', C-4'', C-6'', C-7''
6''	CH	5.04, *br t*, 6.6	124.2	C-4'', C-5'', C-8'', C-10''
7''	C		131.3	
8''	CH₃	1.59, *s*	25.6	C-6'', C-7'', C-10''
9''	CH₃	1.86, *s*	16.3	C-2'', C-3''
10''	CH₃	1.53, *s*	17.6	C-6'', C-7'', C-8''

ᵃRecorded in 300 MHz.; ᵇRecorded in 75 MHz.

3.1.1.2 Compound CC2
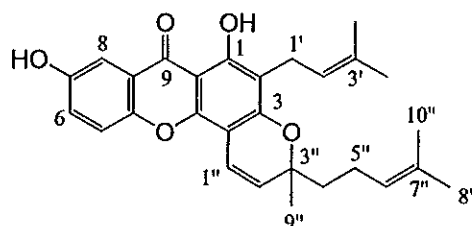
Compound **CC2** was isolated as yellow viscous oil, $[\alpha]^{25}_D$ = -69.8 (c 0.08, CHCl₃). The HREIMS of **CC2** showed a molecular ion peak at m/z 446.2092 $[M]^+$, suggesting the molecular formula $C_{28}H_{30}O_5$. The UV-Vis spectrum showed absorption bands of a xanthone at 243, 289, 298, 320, 351 and 391 nm (Boonnak et al., 2009). The FT-IR spectrum exhibited the hydroxyl group at 3397 cm⁻¹ and conjugated carbonyl group at 1649 cm⁻¹ (Boonnak et al., 2009).

The ¹H and ¹³C NMR data of **CC2** (**Table 3**) were similar to those of **CC4** (**Table 5**), except for the appearance of the signals of a chromene ring bearing a methyl group and six-carbon side-chain of 4-methylpent-3-enyl group which appeared at δ_H 6.88 (d, J = 10.2 Hz, H-1″), 5.54 (d, J = 10.2 Hz, H-2″), 5.10 (br t, J = 7.2 Hz, H-6″), 2.12 (m, H₂-4″ and H₂-5″) 1.89 (m, 1H₂-4″), 1.68 (m, 1H₂-4″), 1.66 (s, CH₃-8″), 1.57 (s, CH₃-10″) and 1.44 (s, CH₃-9″) instead of a geranyl moiety at C-4 as in **CC4**. The loss of 4-methylpent-3-enyl moiety in EI-MS, m/z 363 ($[M]^+$ −83), also supported the proposed structure. Finally, the location of the angular chromene ring was confirmed by HMBC correlations (**Table 3**), in which the methine proton H-1″ (δ 6.88) was correlated with C-3 (δ 158.9), C-4 (δ 100.2), C-4a (δ 150.2) and C-3″ (δ 80.6). The selected HMBC correlations were shown in **Figure 25** for confirmation of this structure. Therefore, compound **CC2** was a new compound, and named as cochinchinone J (Boonnak et al., 2009).
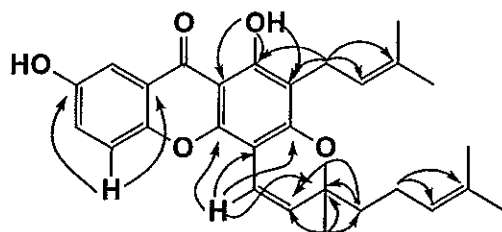
Figure 25. Selected HMBC correlations of **CC2**

Table 3 NMR spectroscopic data of **CC2** in CDCl₃

Position	Type of C	$\delta_H{}^a$ (J in Herz)	$\delta_C{}^b$	HMBC ($^1H \rightarrow {}^{13}C$)
1-OH	C	13.16, s	160.2	C-1, C-2, C-9a
2	C		111.2	
3	C		158.9	
4	C		100.2	
5	CH	7.36, d, 8.7	118.9	C-7, C-8a
6	CH	7.25, m	123.8	C-7, C-4b
7	C		150.3	
8	CH	7.61, br d, 1.8	109.3	C-8
9	C=O		180.5	
4a	C		150.7	
4b	C		152.2	
8a	C		121.1	
9a	C		103.0	
1'	CH₂	3.36, d, 7.2	115.8	C-1, C-2, C-2', C-3'
2'	CH	5.25, br d, 7.2	122.1	C-2'
3'	C		131.5	
4'	CH₃	1.68, s	25.8	C-2', C-3'
5'	CH₃	1.81, s	17.9	C-2', C-3', C-5'
1''	CH	6.88, d, 10.2	115.9	C-3, C-4, C-3'', C-4a
2''	CH	5.54, d, 10.2	125.4	C-4, C-3'', C-4'', C-9''
3''	C		80.6	
4''	CH₂	1.89 (m); 1.68 (m)	41.8	C-2'', C-3''
5''	CH₂	2.12, m	22.8	C-6'', C-7''
6''	CH	5.10, br t, 7.2	123.8	C-6''
7''	C		131.9	
8''	CH₃	1.66, s	25.6	C-6'', C-7'', C-10''
9''	CH₃	1.44, s	27.1	C-2'', C-3'', C-4''
10''	CH₃	1.57, s	17.6	C-6'', C-7'', C-8''

[a] Recorded in 300 MHz.

[b] Recorded in 75 MHz.

3.1.1.3 Compound CC3
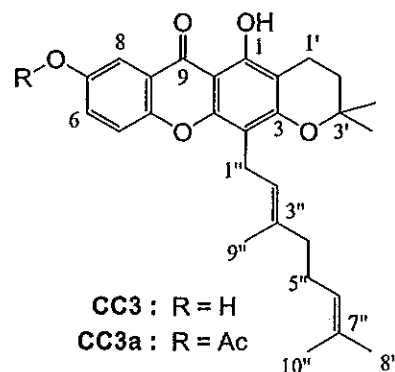
Compound **CC3** was isolated as an inseparable mixture with **CC2**, and the mixture was thus acetylated with Ac_2O in pyridine. The resulting product was further separated by CC eluting with 70% $CHCl_3$-hexane to give monoacetates **CC3a** and **CC2a**. The latter was confirmed as an acetylated derivative of **CC2** by comparison of its spectral data with those of **CC2**.

Compound **CC3a** is a yellow powder, m.p. 85-87 °C. The HREIMS spectrum showed a molecular ion peak at m/z 490.2355 $[M]^+$, corresponding to $C_{30}H_{34}O_6$. The UV spectrum showed absorption bands of a xanthone at 243, 271, 303, 326 and 383 nm (Boonnak *et al.*, 2009), while the IR spectrum exhibited the hydroxyl and conjugated carbonyl functionalities at v_{max} 3392 and 1648 cm^{-1} (Boonnak *et al.*, 2009), respectively.

The ^1H and ^{13}C NMR spectral data of **CC3a** (**Table 4**) were closely related to those of **CC1**, except for the appearance of the signals of a dimethylchromane ring and acetoxy group revealed at δ_H 2.75 (t, J = 6.9 Hz, H_2-1'), 1.81 (t, J = 6.9 Hz, H_2-2'), 1.39 (s, CH_3-4' and CH_3-5') and 2.34 (s, 7-OAc) (**Table 4**) instead of a chromene ring and a hydroxyl group in **CC1**, respectively. The chromane ring was fused to the xanthone nucleus in a linear fashion, which was confirmed by HMBC correlations (**Table 4**), in which the methylene protons H_2-1' at δ_H 2.75 were correlated with C-1 (δ 158.4), C-2 (δ 104.0) and C-3 (δ 159.6), while the H-bonded hydroxyl proton 1-OH (δ 13.02) was correlated with C-1 (δ 158.4), C-2 (δ 104.0) and C-9a (δ 102.4) respectively. Moreover, the selected HMBC correlations were used for confirmation of the structure of **CC3a** as shown in **Figure 26**. Therefore, compound **CC3** was a new compound, and named as cochinchinone K (Boonnak *et al.*, 2009).
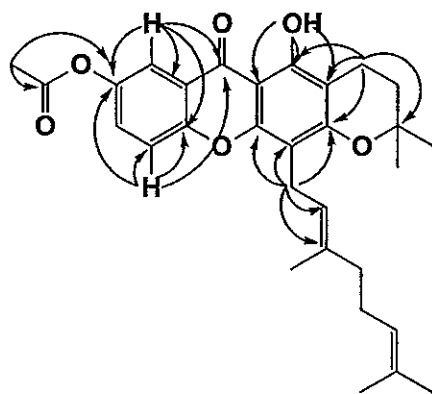
Figure 26. Selected HMBC correlations of **CC3a**

Table 4 NMR spectroscopic data of **CC3a** in CDCl₃

Position	Type of C	$\delta_H{}^a$ (*J* in Herz)	$\delta_C{}^b$	HMBC ($^1H\rightarrow{}^{13}C$)
1-OH	C	13.02, *s*	158.4	C-1, C-2, C-9a
2	C		104.0	
3	C		159.6	
4	C		107.3	
5	CH	7.46, *d*, 8.7	117.8	C-6, C-7, C-9, C-4b
6	CH	7.34, *dd*, 8.7, 2.7	128.6	C-5, C-7, C-4b
7	C		146.3	
8	CH	7.92, *d*, 2.7	118.7	C-7, C-9, C-4b, C-8a
9	C=O		180.3	
4a	C		152.4	
4b	C		153.7	
8a	C		121.2	
9a	C		102.4	
1′	CH₂	2.75, *t*, 6.9	16.2	C-1, C-2, C-3, C-2′, C-3′
2′	CH₂	1.81, *t*, 6.9	31.7	C-1, C-1′, C-3′, C-4′, C-5′
3′	C		76.3	
4′	CH₃	1.39, *s*	26.9	C-2′, C-3′
5′	CH₃	1.39, *s*	26.9	C-2′, C-3′
1″	CH₂	3.48, *d*, 7.5	21.5	C-3, C-4, C-2″, C-3″, C-4a
2″	CH	5.22, *br t*, 6.3	122.3	C-1″, C-3″, C-4″, C-9″
3″	C		134.9	
4″	CH₂	1.96, *m*	39.8	C-2″, C-3″, C-6″
5″	CH₂	2.03, *m*	26.7	C-3″, C-6″, C-7″
6″	CH	5.05, *br t*, 6.6	124.2	C-5″, C-8″, C-10″
7″	C		131.3	
8″	CH₃	1.60, *s*	25.6	C-6″, C-7″, C-10″
9″	CH₃	1.86, *s*	16.3	C-2″, C-3″
10″	CH₃	1.55, *s*	17.7	C-6″, C-7″, C-8″
7-OAc	CH₃	2.34, *s*	21.0	C-7, C-1‴
	C=O		169.4	

ᵃRecorded in 300 MHz.; *ᵇRecorded in 75 MHz.*

3.1.1.4 Compound CC4
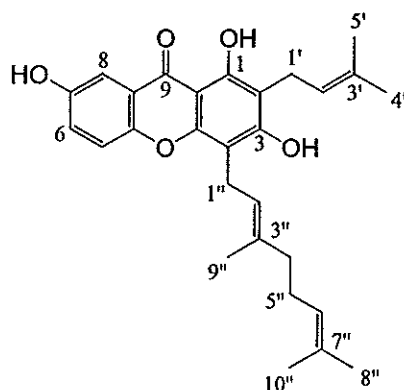
Compound **CC4** was isolated as pale-yellow powder, m.p. 119-120 °C. The UV-Vis spectrum showed absorption bands of a xanthone at 232, 268, 316 and 384 nm (Mahabussarakam *et al.*, 2006). The FT-IR spectrum exhibited the hydroxyl group at 3413 cm^{-1} and conjugated carbonyl group at 1641 cm^{-1} (Mahabussarakam *et al.*, 2006).

The ^{1}H NMR spectrum of **CC4** (**Table 5**) exhibited a chelated hydroxyl proton at δ 12.79 (*s*) and the characteristic signals of ABX trisubstituted benzene at δ 7.44 (*br s*, H-8), 7.07 (*br d*, J = 9.0 Hz, H-6) and 7.04 (*br d*, J = 9.0 Hz, H-5). The presence of an isoprenyl group was suggested by the following ^{1}H NMR spectral data at δ 5.20 (*br t*, J = 6.9 Hz, H-2'), 3.32 (*d*, J = 6.9, H$_2$-1'), 1.75 (*s*, CH$_3$-5') and 1.55 (*s*, CH$_3$-4'). Moreover, the ^{1}H NMR spectrum of **CC4** also showed the characteristic signal of a geranyl side chain at δ 5.16 (*br t*, J = 6.9 Hz, H-2''), 4.96 (*br t*, J = 7.2 Hz, H-6''), 3.39 (*d*, J = 6.9 Hz, H$_2$-1''), 1.99 (*m*, H$_2$-4''), 1.97 (*m*, H$_2$-5''), 1.78 (*s*, CH$_3$-9''), 1.67 (*s*, CH$_3$-8'') and 1.48 (*s*, CH$_3$-10''). The location of an isoprenyl group at C-2 was confirmed by HMBC correlations (**Table 5**) of a chelated hydroxyl group at δ 12.79 to the carbons at δ 103.0 (C-9a), 109.2 (C-2) and 158.2 (C-1), while the methylene protons at δ 3.38 (H$_2$-1') to the carbons at δ 109.2 (C-2), 158.2 (C-1) and 161.1(C-3). Moreover, the attachment of a geranyl side chain at C-4 was connected by using the HMBC correlations of the methylene protons at δ 3.39 (H$_2$-1'') to carbon at δ 104.9 (C-4), 152.9 (C-4a) and 161.1 (C-3). The selected HMBC correlations were shown in **Figure 27** for confirmation of this structure. Therefore, compound **CC4** was assigned as cochinchinone A (Mahabussarakam *et al.*, 2006).
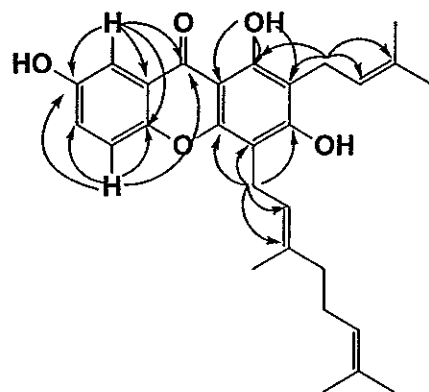
Figure 27. Selected HMBC correlations of **CC4**

Table 5 NMR spectroscopic data of **CC4** in CDCl₃

Position	Type of C	$\delta_H{}^a$ (*J* in Herz)	$\delta_C{}^b$	HMBC (^{1}H→^{13}C)
1-OH	C	12.79, *s*	158.2	C-1, C-2, C-9a
2	C		109.2	
3	C		161.1	
4	C		104.9	
5	CH	7.04, *br d*, 9.0	118.7	C-6, C-7, C-9, C-4b
6	CH	7.07, *br d*, 9.0	124.7	C-5, C-7, C-4b
7	C		150.1	
8	CH	7.44, *br s*	108.7	C-7, C-9, C-4b, C-8a
9	C=O		180.9	
4a	C		152.9	
4b	C		152.6	
8a	C		120.2	
9a	C		103.0	
1′	CH₂	3.32, *d*, 6.9	21.8	C-1, C-2, C-3, C-2′, C-3′
2′	CH	5.20, *br t*, 6.9	121.6	C-1, C-1′, C-3′, C-4′, C-5′
3′	C		134.8	
4′	CH₃	1.55, *s*	25.6	C-2′, C-3′
5′	CH₃	1.75, *s*	17.9	C-2′, C-3′
1″	CH₂	3.39, *d*, 6.9	21.6	C-3, C-4, C-2″, C-3″, C-4a
2″	CH	5.16, *br t*, 6.9	122.7	C-1″, C-3″, C-4″, C-9″
3″	C		137.9	
4″	CH₂	1.99, *m*	39.7	C-2″, C-3″, C-6″
5″	CH₂	1.97, *m*	26.4	C-3″, C-6″, C-7″
6″	CH	4.96, *br t*, 7.2	123.9	C-5″, C-8″, C-10″
7″	C		131.8	
8″	CH₃	1.67, *s*	25.8	C-6″, C-7″, C-10″
9″	CH₃	1.78, *s*	16.2	C-2″, C-3″
10″	CH₃	1.48, *s*	17.6	C-6″, C-7″, C-8″

^{a}Recorded in 300 MHz.; ^{b}Recorded in 75 MHz.

3.1.1.5 Compound CC5
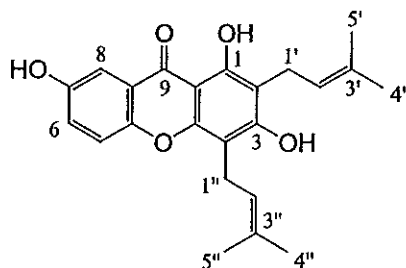
Compound **CC5** was isolated as pale-yellow powder. The UV-Vis spectrum showed absorption bands of a xanthone at 234, 267, 317 and 386 nm (Iinuma *et al.*, 1996). The FT-IR spectrum exhibited the hydroxyl group at 3400 cm^{-1} and conjugated carbonyl group at 1645 cm^{-1} (Iinuma *et al.*, 1996).

The ^{1}H and ^{13}C NMR data of **CC5** (**Table 6**) were similar to those of **CC4** (**Table 5**), except for the appearance of the signals of an isoprenyl side chain which appeared at δ_H 5.25 (*br t*, J = 7.2 Hz, H-2″), 3.47 (*d*, J = 6.9 Hz, H$_2$-1″), 1.87 (*s*, CH$_3$-4″) and 1.74 (*s*, CH$_3$-5″) instead of a geranyl moiety at C-4 as in **CC4**. Finally, the location of the isoprenyl side chain was confirmed by HMBC correlations (**Table 6**) in which the methelyne proton H$_2$-1″ (δ 3.47) was correlated with C-3 (δ 161.1), C-4 (δ 105.2) and C-4a (δ 153.0). The selected HMBC correlation of **CC5** was illustrated in **Figure 28** for confirmation of this structure. Therefore, compound **CC5** was assigned as 1,3,7-trihydroxy-2,4-diisoprenylaxanthone (Iinuma *et al.*, 1996; Nguyen and Harrison 1998).
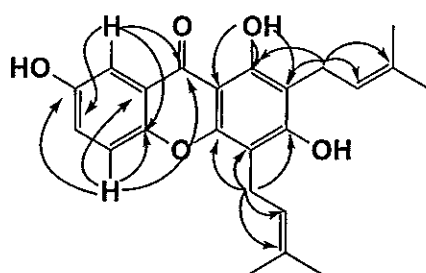
Figure 28. Selected HMBC correlations of **CC5**

Table 6 NMR spectroscopic data of **CC5** in $CDCl_3$

Position	Type of C	$\delta_H{}^a$ (*J* in Herz)	$\delta_C{}^b$	HMBC ($^1H \rightarrow {}^{13}C$)
1-OH	C	12.89, *s*	158.2	C-1, C-2, C-3, C-9a
2	C		108.8	
3	C		161.1	
4	C		105.2	
5	CH	7.16, *d*, 8.7	118.8	C-7, C-9, C-4b, C-8a
6	CH	7.13, *br d*, 8.7	124.2	C-7, C-8, C-4b
7	C		150.2	
8	CH	7.55, *br s*	108.9	C-6, C-9, C-4b
9	C=O		180.9	
4a	C		153.0	
4b	C		152.5	
8a	C		120.3	
9a	C		103.1	
1′	CH_2	3.42, *d*, 6.9	21.6	C-1, C-2, C-3, C-2′, C-3′
2′	CH	5.28, *br t*, 7.2	121.5	C-2, C-1′, C-4′, C-5′
3′	C		135.2	
4′	CH_3	1.84, *s*	17.9	C-2, C-2′, C-3′
5′	CH_3	1.77, *s*	25.8	C-2, C-2′, C-3′
1″	CH_2	3.47, *d*, 6.9	21.8	C-3, C-4, C-2″, C-3″, C-4a
2″	CH	5.25, *br t*, 7.2	121.8	C-4, C-1″, C-4″, C-9″
3″	C		133.9	
4″	CH_3	1.87, *s*	17.9	C-4, C-2″, C-3″
5″	CH_3	1.74, *s*	25.8	C-4, C-2″, C-3″

^{a}Recorded in 300 MHz.

^{b}Recorded in 75 MHz.

3.1.1.6 Compound CC6
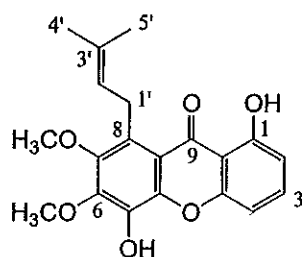
Compound **CC6** was isolated as yellow needle crystals, m.p. 172-174 °C. The UV-Vis spectrum showed absorption bands of a xanthone at 243, 289, 298, 320, 351 and 391 nm (Stout *et al.*, 1963). The FT-IR spectrum exhibited the hydroxyl group at 3397 cm^{-1} and conjugated carbonyl group at 1649 cm^{-1} (Stout *et al.*, 1963).

The ^{1}H NMR spectrum of **CC6** (**Table 7**) showed a chelated hydroxyl proton at δ 13.08 (*s*) and the typical signals of 1,2,3-trisubstituted benzene at δ 7.52 (*t*, J = 8.4 Hz, H-3), 6.92 (*d*, J = 8.4 Hz, H-4) and 6.75 (*d*, J = 8.4 Hz, H-2). The presence of an isoprenyl group was suggested by the following ^{1}H NMR spectral data at δ 5.21 (*br t*, J = 6.6 Hz, H-2′), 4.04 (*d*, J = 6.9, H$_2$-1′), 1.85 (*s*, CH$_3$-4′) and 1.70 (*s*, CH$_3$-5′). Moreover, the ^{1}H NMR spectrum of **CC6** also showed the two signals of methoxyl groups at δ 4.07 (*s*, CH$_3$-6) and 3.82 (*s*, CH$_3$-7). In the HMBC spectrum of **CC6** (**Table 7**), the methylene protons H$_2$-1′ (δ 4.04) was correlated to the aromatic carbons at δ 147.3 (C-7), 128.3 (C-8) and 114.5 (C-8a). It suggested that the isoprenyl side chain could be attached to the carbon at C-8. The location of two methoxyl groups was assigned by using the HMBC correlation, which was shown in **Figure 29**. Structure of **CC6** was also verified by the X-ray diffraction and its structure was illustrated in **Figure 30** (Boonnak *et al.*, 2007). Therefore, compound **CC6** was assigned as *celebixanthone methyl ether* (Stout *et al.*, 1963; Mahabussarakam *et al.*, 2006).
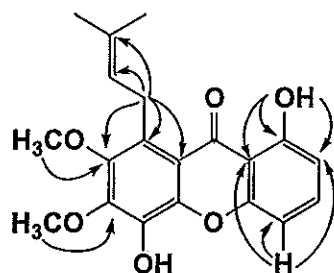
Figure 29. Selected HMBC correlations of CC6
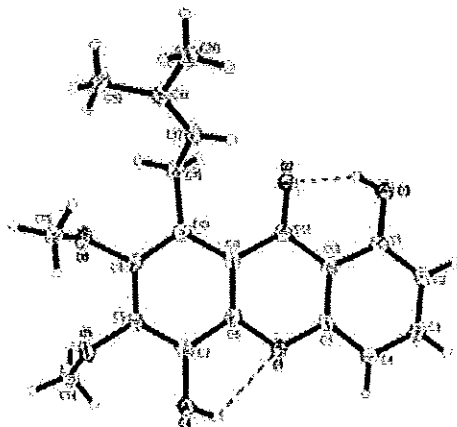
Figure 30. *ORTEP* plot of **CC6**

Table 7 NMR spectroscopic data of **CC6** in $CDCl_3$

Position	Type of C	$\delta_H{}^a$ (*J* in Herz)	$\delta_C{}^b$	HMBC ($^1H \rightarrow {}^{13}C$)
1-OH	C	13.08, *s*	162.1	C-1, C-2, C-3, C-9a
2	CH	6.75, *d*, 8.4	110.6	C-1, C-4, C-4a, C-9a
3	CH	7.52, *t*, 8.4	136.1	C-1, C-4, C-4a
4	CH	6.92, *d*, 8.4	106.2	C-1, C-2, C-4a, C-9a
5	C		135.9	
6	C		145.4	
7	C		147.3	
8	C		128.3	
9	C=O		183.6	
4a	C		155.2	
4b	C		143.4	
8a	C		114.5	
9a	C		109.1	
1'	CH_2	4.04, *d*, 6.6	25.4	C-7, C-8, C-8a, C-2', C-3'
2'	CH	5.21, *br t*, 6.6	123.5	C-8
3'	C		131.6	
4'	CH_3	1.85, *s*	18.1	C-2', C-3'
5'	CH_3	1.70, *s*	25.9	C-2', C-3'
6-OCH$_3$	CH_3	4.07, *s*	61.1	C-6
7-OCH$_3$	CH_3	3.82, *s*	61.1	C-7

^{a}Recorded in 300 MHz.

^{b}Recorded in 75 MHz.

3.1.1.7 Compound CC7
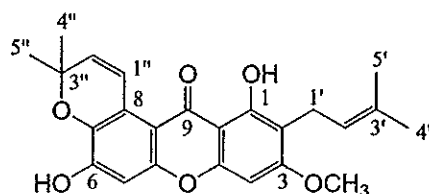
Compound **CC7** was isolated as yellow needle crystals, m.p. 213-215 °C. The UV-Vis spectrum showed typical absorption bands of a xanthone at 244, 265, 322 and 331 nm (Dechathai *et al.*, 2006). The FT-IR spectrum exhibited the hydroxyl group at 3479 cm^{-1} and conjugated carbonyl group at 1675 cm^{-1} (Dechathai *et al.*, 2006).

The ^{1}H NMR spectrum of **CC7** (**Table 8**) showed a chelated hydroxyl proton at δ 13.35 (*s*), two aromatic protons at δ 6.82 (*s*, H-5) and 6.36 (*s*, H-4) and also the characteristic signal of an isoprenyl group at δ 5.23 (*br t*, J = 6.9 Hz, H-2'), 3.35 (*d*, J = 6.6, H$_2$-1'), 1.80 (*s*, CH$_3$-5') and 1.68 (*s*, CH$_3$-4'). Moreover, the ^{1}H NMR spectrum of **CC7** also showed the typical signal of the chromene ring at δ 8.04 (*d*, J = 10.2 Hz, H-1''), 5.82 (*d*, J = 10.2 Hz, H-2'') and 1.50 (*s*, CH$_3$-4'' and CH$_3$-5''). In the HMBC spectrum of **CC7** (**Table 8**), the methylene protons H$_2$-1' (δ 3.35) was correlated to the aromatic carbons at δ 159.6 (C-1), 111.5 (C-2) and 163.1 (C-3), while a chelated hydroxyl group at δ 13.35 was correlated to the carbons at δ 159.6 (C-1), 111.5 (C-2) and 104.6 (C-9a). It suggested that the isoprenyl side chain was fused to the carbon at C-2. The location of the chromene ring was confirmed by using the HMBC correlation (**Table 8**), in which the methine protone H-1'' at δ 8.04 was correlated to the C-7 (δ 159.6), C-8 (δ 159.6) and C-8a (δ 159.6). The selected HMBC correlation of **CC7** was illustrated in **Figure 31** for the structure confirmation. Therefore, compound **CC7** was assigned as dulcisxanthone F (Dechathai *et al.*, 2006; Boonnak *et al.*, 2006).
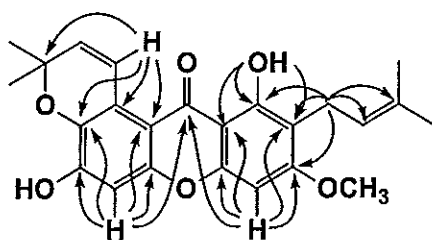
Figure 31. Selected HMBC correlations of CC7

Table 8 NMR spectroscopic data of **CC7** in CDCl₃

Position	Type of C	$\delta_H{}^a$ (*J* in Herz)	$\delta_C{}^b$	HMBC (^{1}H→^{13}C)
1-OH	C	13.35, *s*	159.6	C-1, C-2, C-9a
2	C		111.5	
3	C		163.1	
4	CH	6.36, *s*	88.9	C-2, C-3, C-9, C-4a, C-9a
5	CH	6.82, *s*	102.2	C-6, C-7, C-9, C-4b, C-8a
6	C		150.8	
7	C		137.5	
8	C		120.5	
9	C=O		182.4	
4a	C		155.3	
4b	C		153.3	
8a	C		108.4	
9a	C		104.6	
1'	CH₂	3.35, *d*, 6.9	21.4	C-1, C-2, C-3, C-2', C-3'
2'	CH	5.23, *br t*, 6.9	122.3	-
3'	C		132.0	
4'	CH₃	1.68, *s*	25.8	C-2', C-3', C-5'
5'	CH₃	1.80, *s*	17.8	C-2', C-3', C-4'
1''	CH	8.04, *d*, 10.2	121.0	C-7, C-8, C-8a, C-3''
2''	CH	5.82, *d*, 10.2	132.2	C-8, C-3'', C-4'', C-5''
3''	C		77.2	
4''	CH₃	1.50, *s*	27.4	C-2', C-3'
5''	CH₃	1.50, *s*	27.4	C-2', C-3'
3-OCH₃	CH₃	3.91, *s*	61.1	C-3

*a*Recorded in 300 MHz.

*b*Recorded in 125 MHz.

3.1.1.8 Compound CC8
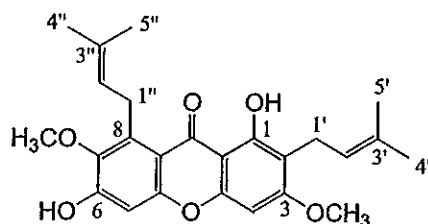
Compound **CC8** was isolated as yellow needle crystals, m.p. 172-174 °C. The UV-Vis spectrum showed typical absorption bands of a xanthone at 243, 289, 298, 320, 351 and 391 nm (Mahabusarakam *et al.*, 1987). The FT-IR spectrum exhibited the hydroxyl group at 3397 cm^{-1} and conjugated carbonyl group at 1649 cm^{-1} (Mahabusarakam *et al.*, 1987).

The ^{1}H and ^{13}C NMR data of **CC8** (**Table 9**) were similar to those of **CC7** (**Table 8**), except for the appearance of the typical signals of an isoprenyl side chain and a methoxyl group which appeared at δ_H 5.26 (*m*, H-2''), 4.09 (*d*, J = 7.2 Hz, H$_2$-1''), 1.83 (*s*, CH$_3$-5'') and 1.68 (*s*, CH$_3$-4'') and 3.81 (*s*, 7-OCH$_3$) instead of a chromene ring as in **CC4**. In the HMBC spectrum of **CC8** (**Table 9**), the methylene protons H$_2$-1'' (δ 4.09) of an isoprenyl side chain was correlated to the aromatic carbons at δ 142.6 (C-7), 137.0 (C-8) and 112.4 (C-8a), while a methoxyl group at δ 3.81 was correlated to C-7 (δ 142.6). It suggested that the isoprenyl side chain and a methoxyl group were connected to the C-8 and C-7, respectively. The selected HMBC correlation of **CC8** was also illustrated in **Figure 32** for confirmation of this structure. Therefore, compound **CC8** was assigned as β-mangostin (Mahabusarakam *et al.*, 1987).
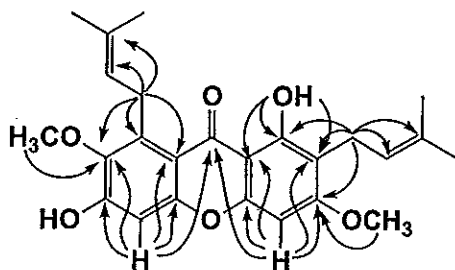
Figure 32. Selected HMBC correlations of **CC8**

Table 9 NMR spectroscopic data of **CC8** in CDCl$_3$

Position	Type of C	$\delta_H{}^a$ (*J* in Herz)	$\delta_C{}^b$	HMBC (^{1}H→^{13}C)
1-OH	C	13.41, *s*	159.8	C-1, C-2, C-9a
2	C		111.5	
3	C		163.5	
4	CH	6.32, *s*	88.8	C-2, C-3, C-9, C-4a, C-9a
5	CH	6.82, *s*	101.5	C-6, C-7, C-9, C-4b, C-8a
6	C		154.4	
7	C		142.6	
8	C		137.0	
9	C=O		181.9	
4a	C		155.7	
4b	C		155.2	
8a	C		112.4	
9a	C		103.8	
1'	CH$_2$	3.35, *d*, 7.2	21.4	C-1, C-2, C-3, C-2', C-3'
2'	CH	5.23, *m*	122.4	C-1', C-3'
3'	C		131.7	
4'	CH$_3$	1.68, *s*	25.8	C-2', C-3'
5'	CH$_3$	1.80, *s*	17.8	C-2', C-3'
1''	CH	4.09, *d*, 7.2	26.6	C-7, C-8, C-8a, C-2'', C-3''
2''	CH$_2$	5.26, *m*	123.2	C-1'', C-3''
3''	C		132.0	
4''	CH$_3$	1.68, *s*	25.8	C-2'', C-3''
5''	CH$_3$	1.83, *s*	18.2	C-2'', C-3''
3-OCH$_3$	CH$_3$	3.90, *s*	62.0	C-3
7-OCH$_3$	CH$_3$	3.81, *s*	55.8	C-7

^{a}Recorded in 300 MHz.

^{b}Recorded in 75 MHz.

3.1.1.9 Compound CC9
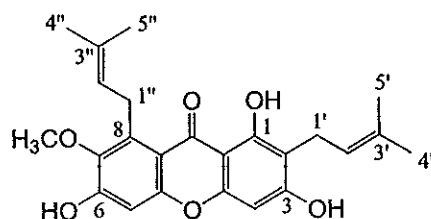
Compound **CC9** was isolated as deep-yellow powder, m.p. 180-182 °C. The UV-Vis spectrum showed typical absorption bands of a xanthone at 243, 289, 298, 320, 351 and 391 nm (Mahabusarakam *et al.*, 1987). The FT-IR spectrum exhibited the hydroxyl group at 3397 cm^{-1} and conjugated carbonyl group at 1649 cm^{-1} (Mahabusarakam *et al.*, 1987).

The ^1H and ^{13}C NMR data of **CC9** (Table 10) were similar to those of **CC8** (Table 9), except for the disappearance of a methoxyl signal in **CC9**. In the HMBC spectrum of **CC9** (Table 10), the methylene protons H_2-1″ (δ 4.09) of an isoprenyl side chain was correlated to the aromatic carbons at δ 142.9 (C-7), 137.3 (C-8) and 111.7 (C-8a), while a methoxyl group at δ 3.81 was correlated to C-7 (δ 142.6). It suggested that the the methoxyl group was connected to the C-7 of the xanthone nucleus. The selected HMBC correlation of **CC9** was also given in **Figure 33** for the structure confirmation. Therefore, compound **CC9** was assigned as α-mangostin (Mahabusarakam *et al.*, 1987).
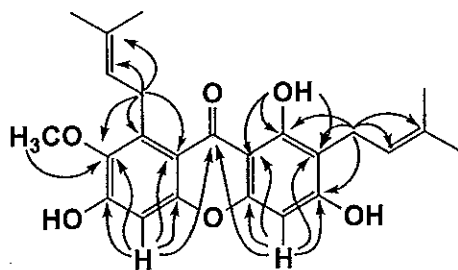
Figure 33. Selected HMBC correlations of CC9

Table 10 NMR spectroscopic data of **CC9** in $CDCl_3$

Position	Type of C	$\delta_H{}^a$ (*J* in Herz)	$\delta_C{}^b$	HMBC ($^1H \rightarrow {}^{13}C$)
1-OH	C	13.77, *s*	160.2	C-1, C-2, C-9a
2	C		109.9	
3	C		161.7	
4	CH	6.29, *s*	92.5	C-2, C-3, C-9, C-4a, C-9a
5	CH	6.82, *s*	101.7	C-6, C-7, C-9, C-4b, C-8a
6	C		155.4	
7	C		142.9	
8	C		137.3	
9	C=O		181.9	
4a	C		154.8	
4b	C		155.5	
8a	C		111.7	
9a	C		103.7	
1'	CH_2	3.45, *d*, 7.2	21.3	C-1, C-2, C-3, C-2', C-3'
2'	CH	5.29, *m*	122.3	-
3'	C		132.3	
4'	CH_3	1.77, *s*	25.7	C-2', C-3'
5'	CH_3	1.84, *s*	17.7	C-2', C-3'
1''	CH_2	4.09, *d*, 6.0	26.3	C-7, C-8, C-8a, C-2'', C-3''
2''	CH	5.26, *m*	123.5	-
3''	C		131.6	
4''	CH_3	1.69, *s*	25.7	C-2'', C-3''
5''	CH_3	1.84, *s*	18.0	C-2'', C-3''
7-OCH_3	CH_3	3.81, *s*	61.2	C-7

^{a}Recorded in 300 MHz.

^{b}Recorded in 75 MHz.

3.1.1.10 Compound CC10
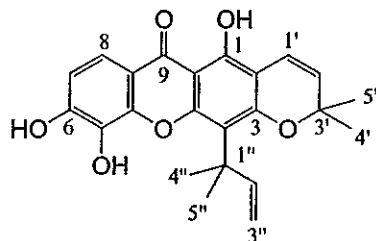
Compound **CC10** was isolated as brown-yellow solid, m.p. 183-184 °C. The UV-Vis spectrum showed typical absorption bands of a xanthone at 240, 283, and 338 nm (Delle Monache *et al.*, 1981). The FT-IR spectrum exhibited the hydroxyl group at 3446 cm^{-1} and conjugated carbonyl group at 1649 cm^{-1} (Delle Monache *et al.*, 1981).

The ^{1}H NMR spectrum of **CC10** (**Table 11**) exhibited a chelated hydroxyl proton at δ 13.53 (*s*), two *ortho*-coupled aromatic protons at δ 7.68 (*d*, J = 9.0 Hz, H-8) and 6.94 (*d*, J = 9.0 Hz, H-7) and the typical signal of a chromene ring at δ 6.76 (*d*, J = 9.9 Hz, H-1'), 5.61 (*d*, J = 9.9, H-2') and 1.52 (*s*, CH$_3$-4' and CH$_3$-5'). The presence of a 1,1-dimethylallyl side chain was suggested by the following ^{1}H NMR spectral data at δ 6.76 (*dd*, J = 17.7, 10.5 Hz, H-2''), 5.22 (*dd*, J = 17.7, 1.5 Hz, 1H-3''), 5.05 (*dd*, J = 10.5, 1.5 Hz, 1H-3'') and 1.65 (*s*, CH$_3$-4'' and CH$_3$-5''). The location of a chromene ring was assigned by using HMBC correlations (**Table 11**) of a chelated hydroxyl group at δ 13.53 to the carbons at δ 103.8 (C-9a), 105.6 (C-2) and 156.8 (C-1) of the methine proton of the chromene ring at δ 6.76 (H-1') to the carbons at δ 105.6 (C-2), 156.8 (C-1) and 158.9 (C-3). It suggested that a chromene ring was fused to the carbon at C-2 and C-3. Moreover, the attachment of a 1,1-dimethylallyl side chain at C-4 was deduced by using the HMBC correlations of the methine proton H-2'' (δ 6.76) to the carbon at C-4 (δ 113.1). The selected HMBC correlations were shown in **Figure 34** for confirmation of this structure. Therefore, compound **CC10** was assigned as macluraxanhone (Delle Monache *et al.*, 1981).
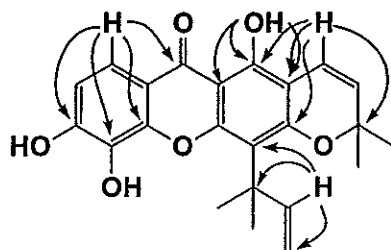
Figure 34. Selected HMBC correlations of **CC10**

Table 11 NMR spectroscopic data of **CC10** in CDCl₃

Position	Type of C	$\delta_H{}^a$ (*J* in Herz)	$\delta_C{}^b$	HMBC ($^1H\rightarrow{}^{13}C$)
1-OH	C	13.53, *s*	156.8	C-1, C-2, C-9a
2	C		105.6	
3	C		158.9	
4	C		113.1	
5	C		131.1	
6	C		149.0	
7	CH	6.94, *d*, 9.0	112.8	C-5, C-6, C-8a
8	CH	7.68, *d*, 9.0	117.5	C-5, C-6, C-9, C-4b
9	C=O		180.8	
4a	C		154.1	
4b	C		144.5	
8a	C		113.7	
9a	C		103.8	
1′	CH	6.76, *d*, 9.9	116.1	C-1, C-2, C-3, C-9a, C-3′
2′	CH	5.61, *d*, 9.9	127.2	C-2, C-3′, C-4′, C-5′
3′	C		78.3	
4′	CH₃	1.52, *s*	27.9	C-2′, C-3′
5′	CH₃	1.52, *s*	27.9	C-2′, C-3′
1″	C		41.4	
2″	CH	6.76, *dd*, 17.7, 10.5	156.8	C-3, C-1″, C-3″, C-4″, C-5″
3″	CH₂	5.22, *dd*, 17.7, 1.5	103.3	C-1″, C-2″
		5.05, *dd*, 10.5, 1.5		C-1″
4″	CH₃	1.65, *s*	28.2	C-4, C-1″, C-2″
5″	CH₃	1.65, *s*	28.2	C-4, C-1″, C-2″
5-OH	-	6.27, *brs*	-	C-5, C-6, C-4b

^{a}Recorded in 300 MHz.

^{b}Recorded in 75 MHz.

3.1.1.11 Compound CC11
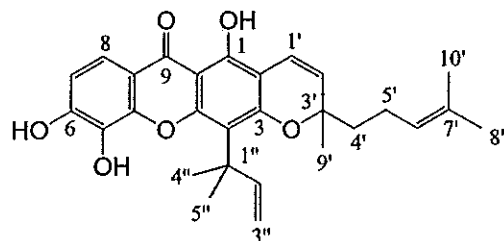
Compound **CC11** was isolated as brown powder, m.p. 143-145 °C and $[\alpha]^{27}_D = -7.4$ (c 0.425, CHCl₃). The UV-Vis spectrum showed typical absorption bands of a xanthone at 243, 288, and 335 nm (Boonnak *et al.*, 2006). The FT-IR spectrum exhibited the hydroxyl group at 3414 cm⁻¹ and conjugated carbonyl group at 1649 cm⁻¹ (Boonnak *et al.*, 2006).

The ¹H and ¹³C NMR data of **CC11** (**Table 12**) were similar to those of **CC10** (**Table 11**), except for the appearance of the signals of a chromene ring bearing a methyl group and six-carbon side-chain of 4-methylpent-3-enyl group which appeared at δ_H 6.81 (d, J = 9.9 Hz, H-1'), 5.57 (d, J = 9. Hz, H-2'), 5.12 (br t, J = 6.9 Hz, H-4'), 2.13 (m, H₂-5'), 1.90 (m, 1H-4'), 1.71 (m, 1H-4'), 1.68 (s, CH₃-8'), 1.59 (s, CH₃-10') and 1.46 (s, CH₃-9') instead of a chromene ring at C-2/C-3 as in **CC10**. The location of the linear chromene ring was confirmed by HMBC correlations (**Table 12**) in which the methine proton H-1' (δ 6.81) was correlated with C-1 (δ 156.8), C-2 (δ 105.2) and C-3 (δ 159.2), while a chelated hydroxyl group at δ 13.53 was also correlated with C-1 (δ 156.8), C-2 (δ 105.2) and C-9a (δ 102.9). The selected HMBC correlations were shown in **Figure 35** for confirmation of this structure. Therefore, compound **CC11** was assigned as pruniflorone G (Boonnak *et al.*, 2006).
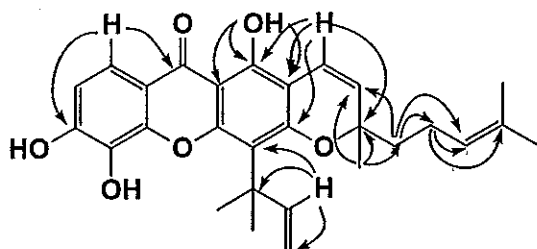
Figure 35. Selected HMBC correlations of **CC11**

Table 12 NMR spectroscopic data of **CC11** in $CDCl_3$

Position	Type of C	$\delta_H{}^a$ (*J* in Herz)	$\delta_C{}^b$	HMBC ($^1H \rightarrow {}^{13}C$)
1-OH	C	13.53, *s*	156.8	C-1, C-2, C-9, C-9a
2	C		105.2	
3	C		159.2	
4	C		112.8	
5	C		131.0	
6	C		149.0	
7	CH	6.94, *d*, 7.8	112.8	-
8	CH	7.67, *d*, 8.4	117.5	C-6, C-9
9	C=O		180.7	
4a	C		154.1	
4b	C		144.5	
8a	C		113.7	
9a	C		102.9	
1'	CH	6.81, *d*, 9.9	116.7	C-1, C-2, C-3, C-3'
2'	CH	5.57, *d*, 9.9	125.6	C-2, C-3', C-4', C-9'
3'	C		81.1	
4'	CH₂	1.90, *m*	41.8	C-2', C-4', C-5', C-6'
		1.71, *m*		-
5'	CH₂	2.13, *m*	23.2	C-6', C-7'
6'	CH	5.12, *brt*, 6.9	123.7	C-5', C-8', C-10'
7'	C		132.1	
8'	CH₃	1.68, *s*	25.7	C-6', C-7'
9'	CH₃	1.46, *s*	26.9	C-2', C-3', C-4'
10'	CH₃	1.59, *s*	17.6	C-6', C-7'
1''	C		41.4	
2''	CH	6.73, *dd*, 17.7, 10.8	156.7	C-4, C-1'', C-4'', C-5''
3''	CH₂	5.21, *dd*, 17.4, 1.2	103.3	C-1'', C-2''
		5.04, *dd*, 10.5, 1.2		C-1''
4''	CH₃	1.65, *s*	28.0	C-4, C-1'', C-2''
5''	CH₃	1.65, *s*	28.4	C-4, C-1'', C-2''

^{a}Recorded in 300 MHz.

^{b}Recorded in 125 MHz.

3.1.1.12 Compound CC12
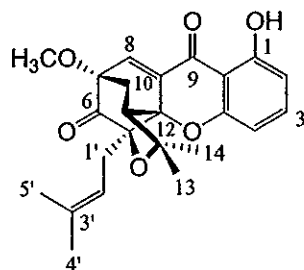
Compound **CC12** was isolated as yellow needle crystal, m.p. 158-159 °C and $[\alpha]^{25}_D$ = +125.1 (c 0.14, $CHCl_3$). The UV-Vis spectrum showed typical absorption bands of a xanthone at 262, 310, 350 and 400 nm (Mahabussarakam *et al.*, 2006). The FT-IR spectrum exhibited the hydroxyl group at 3414 cm^{-1}, conjugated- and unconjugated carbonyl groups at 1649 and 1746 cm^{-1}, respectively. (Mahabussarakam *et al.*, 2006). The ^{13}C NMR spectral data (**Table 13**) confirmed the presence of the unconjugated- and conjugated carbonyl moieties, which appeared at δ 201.1 (C-6) and 180.7 (C-9), respectively.

The ^1H NMR spectrum of **CC12** (**Table 13**) showed a chelated hydroxyl proton at δ 12.10 (s) and the typical signals of 1,2,3-trisubstituted benzene at δ 7.41 (t, J = 8.4 Hz, H-3), 6.55 (dd, J = 8.4, 0.9 Hz, H-2) and 6.52 (dd, J = 8.4, 0.9 Hz, H-4). The ^1H NMR spectrum of **CC12** also showed the appearance of an olefinic proton at δ 7.51 (s, H-8), a methoxy group at δ 3.65 (s, 7-OCH_3), a pair of non-equivalent methylene protons at δ 2.39 ($br\ d$, J = 13.2 Hz, 1H-10) and 1.59 (dd, J = 13.2, 9.9 Hz, 1H-10), a methine proton at δ 2.53 (d, J = 9.6 Hz, H-11) and an isoprenyl side chain at at δ 4.41 ($br\ t$, J = 7.8 Hz, H-2'), 2.64 (d, J = 8.4 Hz, H_2-1'), 1.37 (s, CH_3-4') and 1.01 (s, CH_3-5'). The position of an isoprenyl side chain at C-5 was assigned by using HMBC correlations (**Table 13**) of a methylene proton H-1' at δ 2.64 to the carbons at δ 84.2 (C-5), 88.8 (C-4b) and 201.1 (C-6). In the HMBC spectral data of **CC12** (**Table 13**), a non-equivalent methylene proton at δ 2.39 showed correlations with C-6 (δ 201.1), C-7 (δ 84.9), C-8 (δ 135.3) and C-11 (δ 49.4), whereas a methine proton at δ 2.53 (H-11) showed correlations to the carbons at C-7 (δ 84.9) and C-4b (δ 88.8). It suggested that the methylene and methine carbons at C-10 and C-11 were fused to the carbon at C-7 and C-4b, respectively. The selected HMBC correlations of **CC12** were given in **Figure 36** for confirmation of this structure. Therefore, compound **CC12** was assigned as cochinchinone C (Mahabussarakam *et al.*, 2006).
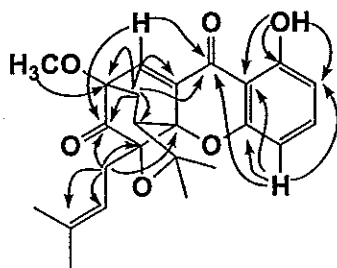
Figure 36. Selected HMBC correlations of **CC12**

Table 13 NMR spectroscopic data of **CC12** in CDCl₃

Position	Type of C	$\delta_H{}^a$ (*J* in Herz)	$\delta_C{}^b$	HMBC (^{1}H→^{13}C)
1-OH	C	12.10, *s*	162.9	C-1, C-2, C-9a
2	CH	6.55, *dd*, 8.4, 0.9	109.5	C-1, C-4, C-9a
3	CH	7.41, *t*, 8.4	138.9	C-1, C-4a
4	CH	6.52, *dd*, 8.1, 0.9	107.4	C-2, C-9, C-4a, C-9a
5	C		84.2	
6	C=O		201.1	
7	C		84.9	
8	CH	7.51, *d*, 1.2	135.3	C-6, C-7, C-9, C-11, C-4b, C-8a
9	C=O		180.7	
4a	C		159.4	
4b	C		88.8	
8a	C		132.1	
9a	C		106.1	
10	CH₂	2.39, *br d*, 13.2	29.7	C-6, C-7, C-8, C-11, C-4b
		1.59, *dd*, 13.2, 9.9		C-6, C-7, C-8
11	CH	2.53, *d*, 9.6	49.4	C-7, C-4b
12	C		83.9	
13	CH₃	1.68, *s*	30.4	C-11, C-12, C-14
14	CH₃	1.33, *s*	29.0	C-11, C-12, C-13
1'	CH₂	2.64, *d*, 8.4	29.2	C-5, C-6, C-4b, C-16, C-17
2'	CH	4.41, *br t*, 7.8	118.4	C-15, C-18, C-19
3'	C		135.7	
4'	CH₃	1.37, *s*	25.5	C-16, C-17, C-19
5'	CH₃	1.01, *s*	16.7	C-16, C-17, C-18
7-OCH₃	CH₃	3.65, *s*	54.1	C-7

^{a}Recorded in 300 MHz.

^{b}Recorded in 75 MHz.

3.1.1.13 Compound CC13
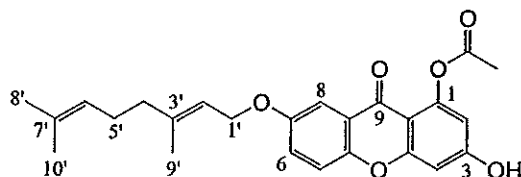
Compound **CC13** was isolated as yellow powder, m.p. 114-116 °C. A molecular ion peak at m/z 422.1718 [M]$^+$ in the HREIMS established the molecular formula of $C_{25}H_{26}O_6$. The UV-Vis spectrum showed absorption bands at 248, 273, 305 and 354 nm (Boonnak *et al.*, 2009), which indicated a typical xanthone chromophore (Seo *et al.*, 2002). The FT-IR spectrum exhibited a hydroxyl group at 3237 cm^{-1}, conjugated- and unconjugated carbonyl groups at 1628 and 1728 cm^{-1}.

The ^{1}H NMR spectrum of **CC13** (**Table 14**) exhibited two *meta*-coupled aromatic protons at δ 6.57 (d, J = 2.1 Hz, H-4) and 6.43 (d, J = 2.1 Hz, H-2), the characteristic signals of 1,2,4-trisubstituted benzene at δ 7.45 (*br s*, H-8), 7.10 (*br d*, J = 9.3 Hz, H-6) and 7.08 (*br d*, J = 9.0 Hz, H-5) and an acetoxyl group at δ 2.38 (*s*, 1-OAc). The presence of an oxygeranyl side chain was suggested by the following ^{1}H NMR spectral data at δ 5.73 (*br t*, J = 6.3 Hz, H-2′), 4.98 (*br t*, J = 6.6 Hz, H-6′), 4.42 (d, J = 6.3 Hz, H$_2$-1′), 1.99 (*m*, H$_2$-4′), 1.98 (*m*, H$_2$-5′), 1.60 (*s*, CH$_3$-8′), 1.57 (*s*, CH$_3$-9′) and 1.50 (*s*, CH$_3$-10′). The location of an oxygeranyl side chain at C-7 was assigned by using HMBC correlations (**Table 14**) of an aromatic proton H-8 at δ 7.45 to the carbons at δ 155.4 (C-7), 125.1 (C-6), 121.9 (C-8a) and 175.7 (C-9), and the methylene protons H$_2$-1′ at δ 4.42 to the carbon at δ 155.4 (C-7). Moreover, the attachment of an acetoxyl group at C-1 was connected by using the HMBC correlations of an aromatic proton H-2 at 6.43 to the carbons at δ 101.3 (C-4), 151.2 (C-1) and 162.6 (C-3), of an acetoxyl protons at δ 2.38 to the carbon at δ 151.2 (C-1). The selected HMBC correlations were shown in **Figure 37** for confirmation of this structure. Therefore, compound **CC13** was a new compound, and named as cochinchinone L (Boonnak *et al.*, 2009).
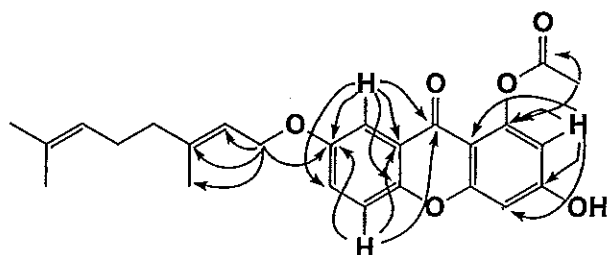
Figure 37. Selected HMBC correlations of **CC13**

Table 14 NMR spectroscopic data for **CC13** in CDCl$_3$

Position	Type of C	$\delta_H{}^a$ (*J* in Herz)	$\delta_C{}^b$	HMBC (^{1}H→^{13}C)
1	C		151.2	
2	CH	6.43, *d*, 2.1	108.3	C-1, C-3, C-4, C-9, C9a
3	C		162.6	
4	CH	6.57, *d*, 2.1	101.3	C-2, C-3, C-9, C-4a, C-9a
5	CH	7.08, *br d*, 9.0	118.8	C-7, C-9, C-8a
6	CH	7.10, *br d*, 9.3	125.1	C-5, C-8, C-4b
7	C		155.4	
8	CH	7.45, *br s*	106.4	C-6, C-7, C-9, C-4b, C-8a
9	C=O		175.7	
4a	C		158.8	
4b	C		150.0	
8a	C		121.9	
9a	C		107.8	
1′	CH$_2$	4.42, *d*, 6.3	65.5	C-7, C-2′, C-3′, C-4′, C-5′, C-9′
2′	CH	5.73, *br t*, 6.3	118.8	C-1′, C-4′, C-9′
3′	C		141.9	
4′	CH$_2$	1.99, *m*	39.5	C-2′, C-3′, C-6′
5′	CH$_2$	1.98, *m*	26.3	C-3′, C-6′, C-7′
6′	CH	4.98, *br t*, 6.6	123.8	C-4′, C-5′, C-8′, C-10′
7′	C		131.8	
8′	CH$_3$	1.60, *s*	25.7	C-6′, C-7′, C-10′
9′	CH$_3$	1.57, *s*	16.7	C-2′, C-3′, C-4′
10′	CH$_3$	1.50, *s*	17.7	C-6′, C-7′, C-8′
1-OAc	CH$_3$	2.38, *s*	21.3	C-1, C-1″
	C=O		170.9	

^{a}Recorded in 300 MHz.

^{b}Recorded in 75 MHz.

3.1.1.14 Compound CC14
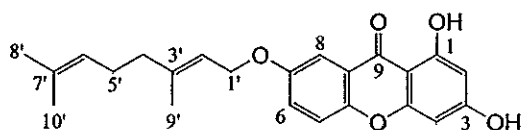
Compound **CC14** was isolated as yellow powder, m.p. 138-140 °C. The UV-Vis spectrum showed a typical xanthone absorption bands at 206, 236, 260, 316 and 364 nm (Nguyen and Harrison, 1998). The FT-IR spectrum exhibited a hydroxyl group at 3162 cm^{-1} and conjugated carbonyl group at 1652 cm^{-1} (Nguyen and Harrison, 1998).

The ^{1}H and ^{13}C NMR data of **CC14** (**Table 15**) were similar to those of **CC13** (**Table 14**), except for the presence of a chelated hydroxyl group at δ 12.85 (s, 1-OH) instead of an acetoxyl group signal at δ 2.38 as in **CC13**. The position of a chelated hydroxyl group at C-1 was confirmed by HMBC correlation (**Table 15**) of an aromatic proton H-2 at δ 6.22 to the carbons at δ 163.8 (C-1), 164.0 (C-3) and 94.4 (C-4), of a chelated hydroxyl group at δ 12.85 to the carbon at δ 163.8 (C-1), 103.3 (C-9a) and 98.5 (C-2). Moreover, the location of an oxygeranyl side chain at C-7 was completely assigned by HMBC correlations as shown in **Figure 38**. Therefore, compound **CC14** was assigned as 7-geranyloxy-1,3-dihydroxy-xanthone (Nguyen and Harrison, 1998).
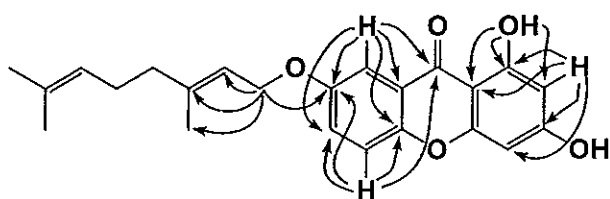
Figure 38. Selected HMBC correlations of CC14

Table 15 NMR spectroscopic data for **CC14** in CDCl₃

Position	Type of C	$\delta_H{}^a$ (*J* in Herz)	$\delta_C{}^b$	HMBC (^{1}H→^{13}C)
1-OH	C	12.85, *s*	163.8	C-1, C-2, C-9, C-9a
2	CH	6.22, *d*, 1.8	98.5	C-1, C-3, C-4
3	C		164.0	
4	CH	6.27, *d*, 1.8	94.4	C-2, C-3, C-9, C-4a, C-9a
5	CH	7.14, *br d*, 9.0	118.9	C-6, C-7, C-9, C-4b
6	CH	7.18, *br d*, 9.6	125.6	C-7, C-8, C-4b
7	C		155.2	
8	CH	7.40, *br d*, 1.5	105.9	C-6, C-7, C-9, C-4b, C-8a
9	C=O		180.5	
4a	C		157.8	
4b	C		150.7	
8a	C		120.4	
9a	C		103.3	
1′	CH₂	4.45, *d*, 6.3	65.6	C-7, C-2′, C-3′, C-4′, C-5′, C-9′
2′	CH	5.39, *br t*, 6.3	118.6	C-1′, C-4′, C-5′, C-9′
3′	C		141.2	
4′	CH₂	2.01, *m*	39.5	C-2′, C-3′, C-6′
5′	CH₂	1.95, *m*	26.3	C-3′, C-6′, C-7′
6′	CH	4.99, *br t*, 5.7	123.8	C-4′, C-5′, C-8′, C-10′
7′	C		131.8	
8′	CH₃	1.57, *s*	26.3	C-6′, C-7′, C-10′
9′	CH₃	1.64, *s*	16.4	C-2′, C-3′, C-4′
10′	CH₃	1.50, *s*	17.7	C-6′, C-7′, C-8′
3-OH		7.86, *br s*		-

^{a}Recorded in 300 MHz.

^{b}Recorded in 75 MHz.

3.1.1.15 Compound CC15
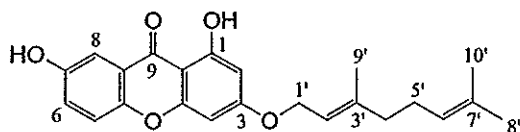
Compound **CC15** was isolated as yellow powder, m.p. 147-148 °C. The UV-Vis spectrum showed a typical xanthone absorption bands at 203, 229, 259, 307 and 374 nm (Mahabusarakam *et al.*, 2008). The FT-IR spectrum exhibited a hydroxyl group at 3288 cm^{-1}, conjugated carbonyl group at 1647 cm^{-1} (Mahabusarakam *et al.*, 2008).

The ^{1}H and ^{13}C NMR data of **CC15** (**Table 16**) were closely similar to those of **CC14** (**Table 15**). The main difference is the position of an oxygeranyl side chain. In HMBC spectral data (**Table 16**) of **CC15**, the methylene proton at δ 4.63 showed correlations with C-3 (δ 166.1), while an aromatic proton H-2 at δ 6.34 showed correlations to the carbons at C-1 (δ 163.2), C-3 (166.1) and C-4 (δ 93.2). It suggested that an oxygeranyl side chain of **CC15** were attached to the carbons at C-3 instead of C-7 as in **CC14**. The selected HMBC correlations were shown in **Figure 39** for confirmation of this structure. Therefore, compound **CC15** was deduced as 3-geranyloxy-1,3-dihydroxyxanthone, a structural isomer of **CC14** (Mahabusarakam *et al.*, 2008).
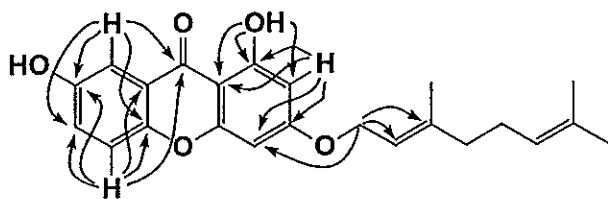
Figure 39. Selected HMBC correlations of **CC15**

Table 16 NMR spectroscopic data of **CC15** in CDCl₃

Position	Type of C	$\delta_H{}^a$ (J in Herz)	$\delta_C{}^b$	HMBC ($^1H \rightarrow {}^{13}C$)
1-OH	C	12.73, s	163.2	C-1, C-2, C-9a
2	CH	6.34, d, 2.4	97.6	C-1, C-3, C-4, C-9a
3	C		166.1	
4	CH	6.40, d, 2.4	93.2	C-2, C-3, C-9, C-4a, C-9a
5	CH	7.30, br d, 9.3	119.0	C-6, C-7, C-9, C-4b, C-8a
6	CH	7.26, br d, 9.3	124.2	C-5, C-7, C-4b
7	C		152.5	
8	CH	7.40, br s	109.0	C-6, C-7, C-9, C-4b
9	C=O		180.6	
4a	C		157.8	
4b	C		150.5	
8a	C		120.9	
9a	C		103.5	
1'	CH₂	4.63, d, 6.6	65.6	C-3, C-2', C-3'
2'	CH	5.50, br t, 6.6	118.4	C-4', C-9'
3'	C		142.4	
4'	CH₂	2.14, m	39.5	C-2', C-3', C-6'
5'	CH₂	2.10, m	26.2	C-3', C-6', C-7'
6'	CH	5.11, br t, 5.7	123.6	-
7'	C		131.9	
8'	CH₃	1.69, s	25.6	C-6', C-7', C-10'
9'	CH₃	1.78, s	16.8	C-2', C-3', C-4'
10'	CH₃	1.63, s	17.7	C-6', C-7', C-8'
7-OH		7.03, br s		-

^{a}Recorded in 300 MHz.

^{b}Recorded in 75 MHz.

3.1.1.16 Compound CC16
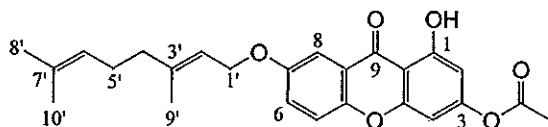
Compound **CC14** (82.5 mg) was treated with Ac$_2$O (2.5 mL) in pyridine (2.0 mL) and stirred for 6 hr at room temperature. The reaction mixture was diluted with water, extracted with CH$_2$Cl$_2$. The combined organic extract was washed with 10% HCl and then washed with water again. After the organic solvent was removed, the resulting residue was dried over anhydrous Na$_2$SO$_4$. Chromatography over silica gel yielded a pale yellow powder of compound **CC16** (80.6 mg).

Compound **CC16** was isolated as yellow powder, m.p. 94-95 °C. A molecular ion peak at m/z 422.1725 [M]$^+$ in the HREIMS established the molecular formula of C$_{25}$H$_{26}$O$_6$. The UV-Vis spectrum showed an absorption bands at 239, 262, 289 and 379 nm. The FT-IR spectrum exhibited a hydroxyl group at 3429 cm^{-1}, conjugated- and unconjugated carbonyl groups at 1652 and 1768 cm^{-1}.

The ^{1}H and ^{13}C NMR data of **CC16** (**Table 17**) were similar to those of **CC14** (**Table 15**), except for the presence of an acetoxyl group at δ 2.23 (s, 3-OAc) instead of a hydroxyl group at C-3 as in **CC14**. The position of an acetoxyl group at C-3 was deduced by using HMBC correlation (**Table 17**) of an aromatic proton H-2 at δ 6.42 to the carbons at δ 162.9 (C-1), 156.8 (C-3), 106.6 (C-9a) and 100.7 (C-4), while an acetoxyl group at δ 2.23 to the carbon at δ 156.8 (C-3). The selected HMBC correlations were also given in **Figure 40** for the structure confirmation. Therefore, compound **CC16** was assigned as 3-acetoxy-7-geranyloxy-1-hydroxyxanthone (Boonnak et al., 2009).
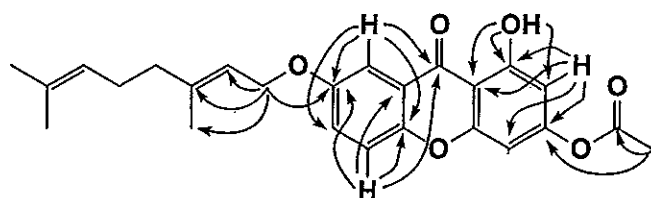
Figure 40. Selected HMBC correlations of CC16

Table 17 NMR spectroscopic data of **CC16** in $CDCl_3$

Position	Type of C	$\delta_H{}^a$ (J in Herz)	$\delta_C{}^b$	HMBC ($^1H\rightarrow{}^{13}C$)
1-OH	C	12.71, s	162.9	C-1, C-2, C-3, C-9, C-4a, C-9a
2	CH	6.42, br d, 2.1	104.0	C-1, C-3, C-4, C-9a
3	C		156.8	
4	CH	6.59, br d, 2.1	100.7	C-3, C-9, C-4a, C-9a
5	CH	7.21, br d, 8.1	119.0	C-7, C-9, C-4b, C-8a
6	CH	7.20, br d, 8.1	126.0	C-7, C-8, C-4b
7	C		155.5	
8	CH	7.44, br s	106.0	C-6, C-7, C-9, C-4b
9	C=O		181.2	
4a	C		156.7	
4b	C		150.8	
8a	C		120.7	
9a	C		106.6	
1'	CH_2	4.51, d, 6.6	65.6	C-7, C-2', C-3', C-9'
2'	CH	5.40, br t, 6.6	118.8	C-1', C-3', C-4', C-9'
3'	C		141.9	
4'	CH_2	2.04, m	39.6	C-2', C-3'
5'	CH_2	2.02, m	26.3	C-4', C-6', C-7'
6'	CH	5.00, br t, 5.7	123.7	C-4', C-5', C-10'
7'	C		131.8	
8'	CH_3	1.58, s	25.6	C-6', C-7', C-10'
9'	CH_3	1.68, s	16.7	C-2', C-3', C-4'
10'	CH_3	1.51, s	17.7	C-6', C-7', C-8'
3-OAc	CH_3	2.23, s	21.2	C-3
	C=O		168.2	

^{a}Recorded in 300 MHz.

^{b}Recorded in 75 MHz.

3.1.1.17 Compound CC17
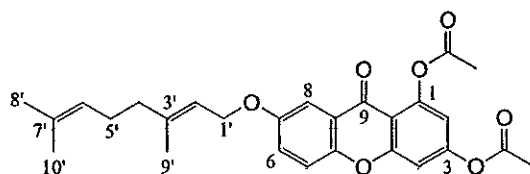
Compound **CC14** (200.5 mg) was treated with Ac_2O (6.0 mL) in pyridine (3.0 mL) and stirred overnight at room temperature. Chromatography over silica gel yielded a pale yellow powder of **CC16** (10.6 mg) and **CC17** (177.8 mg), respectively.

Compound **CC17** was isolated as yellow powder, m.p. 96-97 °C. A molecular ion peak at m/z 464.1838 $[M]^+$ in the HREIMS established the molecular formula of $C_{27}H_{28}O_7$. The UV-Vis spectrum showed an absorption bands at 253, 300 and 361 nm. The FT-IR spectrum exhibited a hydroxyl group at 3429 cm^{-1}, conjugated- and unconjugated carbonyl groups at 1656 and 1776 cm^{-1}.

The ^1H and ^{13}C NMR data of **CC17** (**Table 18**) were similar to those of **CC14** (**Table 15**), except for the presence of two acetoxyl groups at δ 2.47 (s, 1-OAc) and 2.27 (s, 3-OAc) instead of chelated- and hydroxyl groups at C-1 and C-3 as in **CC14**, respectively. The assignment of the location of the acetoxyl groups at δ 2.47 to the carbon at C-1 and δ 2.27 to the carbon at C-3, which were deduced by using HMBC correlation (**Table 18**) of an aromatic proton H-2 at δ 7.16 to the carbons at δ 150.9 (C-1), 154.5 (C-3) and 112.4 (C-4), while the acetoxyl group at δ 2.47 showed correlation to the carbon at δ 150.9 (C-1), whereas an acetoxyl group at δ 2.27 exhibited correlation to the carbon at δ 154.5 (C-3). The selected HMBC correlations were also given in **Figure 41** for the structure confirmation. Therefore, compound **CC17** was assigned as 1,3-diacetoxy-7-geranyloxyxanthone (Boonnak *et al.*, 2009).
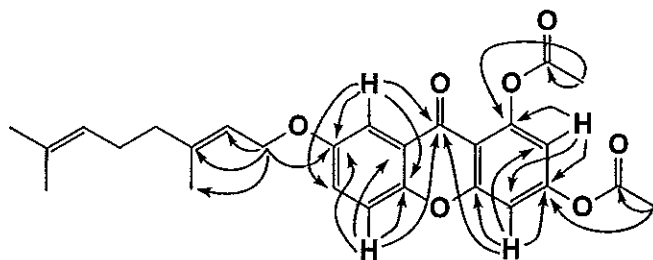
Figure 41. Selected HMBC correlations of **CC17**

Table 18 NMR spectroscopic data of **CC17** in CDCl$_3$

Position	Type of C	$\delta_\text{H}{}^a$ (J in Herz)	$\delta_\text{C}{}^b$	HMBC (^{1}H→^{13}C)
1	C		150.9	
2	CH	7.16, d, 2.1	108.7	C-1, C-3, C-4, C-4a
3	C		154.5	
4	CH	6.80, d, 2.1	112.4	C-1, C-2, C-3, C-4a, C-9a
5	CH	7.23, br d, 9.3	118.9	C-7, C-9, C-4b, C-8a
6	CH	7.28, dd, 9.3, 2.4	125.3	C-7, C-8, C-4b
7	C		155.5	
8	CH	7.44, br d, 2.1	106.6	C-6, C-7, C-9, C-4b, C-8a
9	C=O		174.7	
4a	C		157.7	
4b	C		149.9	
8a	C		122.3	
9a	C		112.2	
1′	CH$_2$	4.56, d, 6.3	65.5	C-7, C-2′, C-3′, C-9′
2′	CH	5.49, br t, 6.3	118.8	C-1′, C-3′, C-4′
3′	C		141.6	
4′	CH$_2$	2.11, m	39.5	C-2′, C-3′, C-5′, C-9′
5′	CH$_2$	2.09, m	26.3	C-3′, C-6′, C-7′
6′	CH	5.09, br t, 6.3	123.8	C-4′, C-5′, C-8′, C-10′
7′	C		131.7	
8′	CH$_3$	1.66, s	25.6	C-6′, C-7′, C-10′
9′	CH$_3$	1.74, s	16.7	C-2′, C-3′, C-4′
10′	CH$_3$	1.60, s	17.7	C-6′, C-7′, C-8′
1-OAc	CH$_3$	2.47, s	21.1	C-1
	C=O		169.2	
3-OAc	CH$_3$	2.27, s	21.0	C-3
	C=O		167.8	

^{a}Recorded in 300 MHz.

^{b}Recorded in 75 MHz.

3.1.1.18 Compound CC18
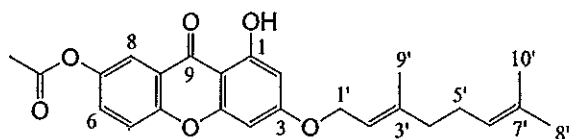
Compound **CC15** (85.5 mg) was treated with Ac_2O (2.5 mL) in pyridine (2.0 mL) and stirred for 6 hr at room temperature. Chromatography over silica gel yielded a pale yellow powder of **CC18** (83.7 mg).

Compound **CC18** was isolated as yellow powder, m.p. 104-106 °C. A molecular ion peak at m/z 422.1726 $[M]^+$ in the HREIMS established the molecular formula of $C_{25}H_{26}O_6$. The UV-Vis spectrum showed an absorption bands at 243, 257, 310 and 358 nm. The FT-IR spectrum exhibited a hydroxyl group at 3429 cm^{-1}, conjugated- and unconjugated carbonyl groups at 1665 and 1758 cm^{-1}.

The ^1H and ^{13}C NMR data of **CC18** (**Table 19**) were similar to those of **CC15** (**Table 16**), except for the presence of an acetoxyl group at δ 2.22 (s, 7-OAc) instead of a hydroxyl group at C-7 as in **CC15**. The position of an acetoxyl group at C-7 of **CC18** was deduced by using HMBC correlation (**Table 19**) of an aromatic proton H-8 at δ 7.76 to the carbons at C-6 (δ 128.9), C-7 (δ 146.5), C-9 (δ 179.8), C-8a (δ 121.1) and C-4a (δ 157.5), of an acetoxyl group at δ 2.22 to the carbons at C-7 (δ 146.5). The selected HMBC correlations were also given in **Figure 42** for confirmation of this structure. Structure of **CC18** was also determined by X-ray diffraction and its structure was illustrated in **Figure 43**. Therefore, compound **CC18** was a new compound, and assigned as 7-acetoxy-3-geranyloxy-1-hydroxyxanthone (Boonnak *et al.*, 2009).
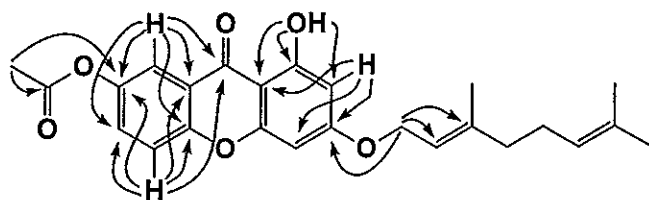
Figure 42. Selected HMBC correlations of **CC18**
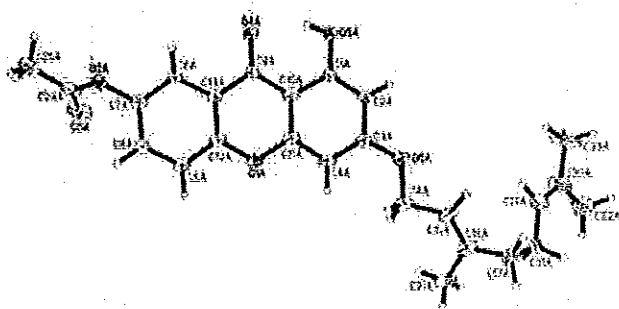
Figure 43. *ORTEP* plot of **CC18**

Table 19 NMR spectroscopic data of **CC18** in CDCl$_3$

Position	Type of C	$\delta_H{}^a$ (*J* in Herz)	$\delta_C{}^b$	HMBC (^{1}H→^{13}C)
1-OH	C	12.55, *s*	163.3	C-1, C-2, C-9, C-9a
2	CH	6.19, *br d*, 2.1	97.8	C-3, C-4, C-9a
3	C		166.2	
4	CH	6.24, *br d*, 2.1	93.4	C-2, C-3, C-4a, C-9a
5	CH	7.25, *d*, 9.3	118.7	C-6, C-7, C-9, C-4b, C-8a
6	CH	7.28, *br d*, 9.0	128.9	C-7, C-8, C-4b
7	C		146.5	
8	CH	7.76, *br s*	117.8	C-6, C-7, C-9, C-4b, C-8a
9	C=O		179.8	
4a	C		157.5	
4b	C		153.4	
8a	C		121.1	
9a	C		103.4	
1'	CH$_2$	4.49, *d*, 6.3	65.6	C-3, C-2', C-3', C-5'
2'	CH	5.37, *br t*, 6.3	118.4	C-1', C-3', C-4', C-9'
3'	C		142.2	
4'	CH$_2$	2.03, *m*	39.5	C-2', C-3', C-5', C-6'
5'	CH$_2$	1.97, *m*	26.2	C-3', C-6', C-7'
6'	CH	4.99, *br t*, 6.9	123.9	C-4', C-5', C-8', C-10'
7'	C		131.9	
8'	CH$_3$	1.58, *s*	25.6	C-6', C-7', C-10'
9'	CH$_3$	1.66, *s*	16.7	C-2', C-3', C-4'
10'	CH$_3$	1.51, *s*	17.7	C-6', C-7', C-8'
7-OAc	CH$_3$	2.22, *s*	20.9	C-7
	C=O		169.2	

^{a}Recorded in 300 MHz.

^{b}Recorded in 75 MHz.

3.1.1.19 Compound CC19
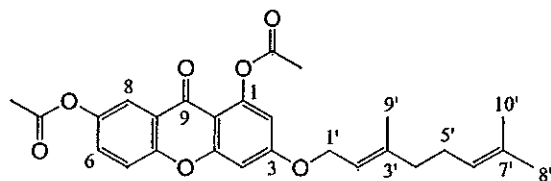
Compound **CC15** (190.0 mg) was treated with Ac_2O (6.0 mL) in pyridine (3.0 mL) and stirred overnight at room temperature. Chromatography over silica gel yielded a pale yellow powder of **CC18** (8.7 mg) and **CC19** (170.0 mg), respectively.

Compound **CC19** was isolated as yellow powder, m.p. 85-87 °C. A molecular ion peak at m/z 464.1834 $[M]^+$ in the HREIMS established the molecular formula of $C_{27}H_{28}O_7$. The UV-Vis spectrum showed an absorption bands at 246, 275, 302 and 334 nm. The FT-IR spectrum exhibited a hydroxyl group at 3453 cm^{-1}, conjugated- and unconjugated carbonyl groups at 1655 and 1770 cm^{-1}.

The ^1H and ^{13}C NMR data of compound **CC19** (**Table 20**) were similar to those of compound **CC15** (**Table 16**), except for the presence of two acetoxyl groups at δ 2.47 (s, 1-OAc) and 2.27 (s, 3-OAc) instead of chelated- and hydroxyl groups at C-1 and C-3, respectively as in compound **CC14**. The position of an acetoxyl group at C-1 (δ 2.40) was deduced by HMBC correlation (**Table 20**) of an aromatic proton H-2 at δ 6.64 to the carbons at δ 163.6 (C-3), 158.7 (C-1), 108.6 (C-9a) and 108.1 (C-4), of an acetoxyl group at δ 2.40 to the carbon at δ 158.7 (C-1). Moreover, an acetoxy proton at δ 2.19 showed correlations with C-7 (δ 146.2), while an aromatic proton H-8 at δ 7.76 showed correlations to the carbons at C-6 (δ 128.3), C-7 (δ 146.2), C-9 (δ 174.1), C-4b (δ 152.7) and C-8a (δ 123.6). It suggested that an acetoxyl group at δ 2.19 was attached to the carbon at C-7. The selected HMBC correlations were also given in **Figure 44** for structure confirmation. Therefore, compound **CC19** was assigned as 1,7-diacetoxy-3-geranyloxyxanthone (Boonnak *et al.*, 2009).
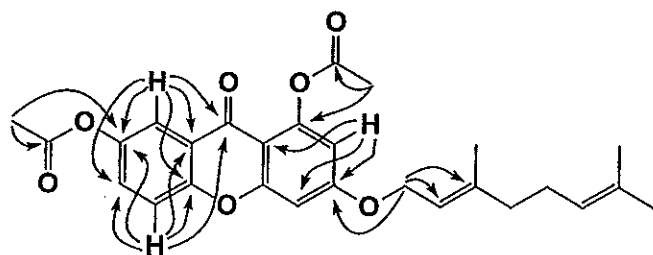
Figure 44. Selected HMBC correlations of **CC19**

Table 20 NMR spectroscopic data of **CC19** in $CDCl_3$

Position	Type of C	$\delta_H{}^a$ (*J* in Herz)	$\delta_C{}^b$	HMBC ($^1H\rightarrow{}^{13}C$)
1	C		158.7	
2	CH	6.64, *d*, 2.1	99.5	C-1, C-3, C-4, C-9, C-9a
3	C		163.6	
4	CH	6.47, *d*, 2.1	108.1	C-2, C-3, C-9, C-4a, C-9a
5	CH	7.24, *d*, 8.1	118.6	C-6, C-7, C-9, C-4b, C-8a
6	CH	7.27, *br d*, 8.1	128.3	C-7, C-8, C-4b
7	C		146.2	
8	CH	7.76, *br d*, 1.5	118.4	C-6, C-7, C-9, C-4b, C-8a
9	C=O		174.1	
4a	C		151.4	
4b	C		152.7	
8a	C		123.6	
9a	C		108.6	
1'	CH_2	4.51, *d*, 6.3	65.8	C-3, C-2', C-3', C-4', C-5'
2'	CH	5.37, *br t*, 6.3	118.0	C-1', C-4', C-9'
3'	C		142.6	
4'	CH_2	2.03, *m*	39.5	C-2', C-3', C-6'
5'	CH_2	2.01, *m*	26.2	C-3', C-6', C-7'
6'	CH	4.99, *br t*, 6.6	123.6	C-4', C-8', C-10'
7'	C		131.9	
8'	CH_3	1.57, *s*	25.7	C-6', C-7', C-10'
9'	CH_3	1.65, *s*	16.7	C-2', C-3', C-4'
10'	CH_3	1.51, *s*	17.7	C-6', C-7', C-8'
1-OAc	CH_3	2.40, *s*	21.2	C-1
	C=O		169.5	
7-OAc	CH_3	2.19, *s*	20.9	C-7
	C=O		169.2	

^{a}Recorded in 300 MHz.

^{b}Recorded in 75 MHz.

3.1.1.20 Compound CC20
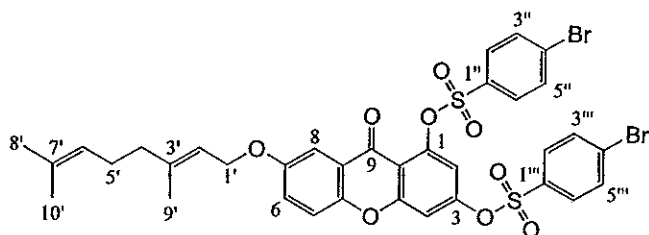
Compound **CC14** (40.0 mg) was stirred overnight at room temperature with
p-bromobenzenesulfonyl chloride (40.30 mg) and K_2CO_3 (44.1 mg) in CH_2Cl_2 (3.0 mL).
After the reaction was complete, water (10.0 mL) was added to the reaction mixture. The
resulting solution was then extracted with CH_2Cl_2 (10.0 mL, 3 times). The combined organic
extract was dried over anhydrous sodium sulfate and evaporated under reduced pressure to
give a crude extract, which was further purified by column chromatography over silica gel
eluting with 5% acetone–hexane to yield the dibrosylate **CC20** (75.2 mg).

Compound **CC20** was isolated as yellow needle crystal, m.p. 106-108 °C. The ^1H and
^{13}C NMR data of **CC20** (**Table 21**) were similar to those of **CC14** (**Table 15**), except for the
presence of two aromatic signals of the *p*-bromobenzenesulfonyl groups at δ 7.81 (*dd*, J =
9.0, 2.1 Hz, H-2″ and H-6″), 7.65 (*dd*, J = 8.7, 2.1 Hz, H-3″, H-5″, H-2‴ and H-6‴) and
7.63 (*dd*, J = 8.7, 2.1 Hz, H-3‴ and H-5‴) instead of chelated- and hydroxyl groups at C-1
and C-3 as in **CC14**. The selected HMBC correlations were also shown in **Figure 45** for
confirmation of this structure. Finally, structure of **CC20** was fully supported by the X-ray
structure as illustrated in **Figure 46** (Boonnak *et al.*, 2009). Therefore, compound **CC20** was
assigned as 7-geranyloxy-1,3-dibrosylatedxyxanthone (Boonnak *et al.*, 2009).
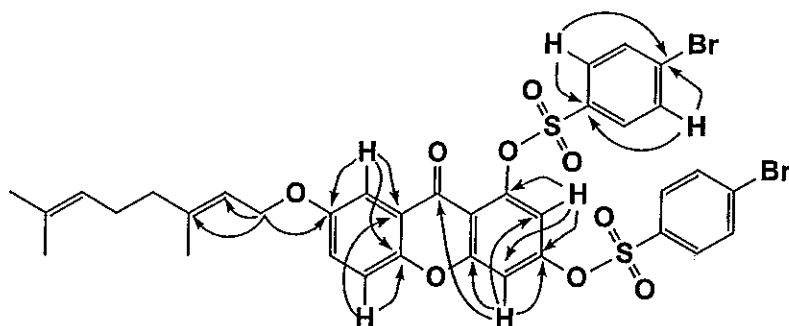
Figure 45. Selected HMBC correlations of **CC20**
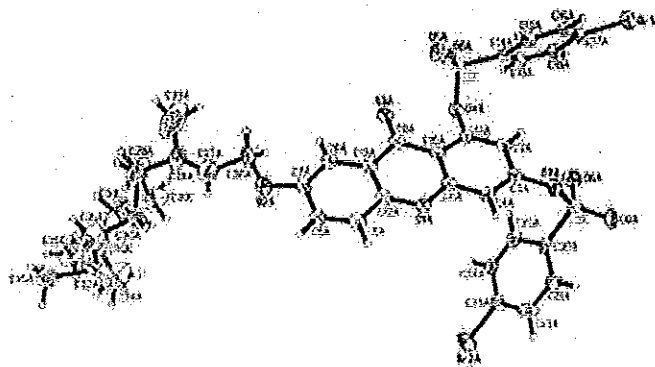
Figure 46. *ORTEP* plot of **CC20**

Table 21 NMR spectroscopic data of **CC20** in CDCl₃

Position	Type of C	$\delta_H{}^a$ (J in Herz)	$\delta_C{}^b$	HMBC (^{1}H→^{13}C)
1	C		157.4	
2	CH	7.32, d, 2.4	111.1	C-1, C-3, C-4, C-4a
3	C		152.0	
4	CH	7.68, d, 2.4	112.8	C-2, C-3, C-4a, C-9a
5	CH	7.26, d, 9.0	118.8	C-4b, C-8a
6	CH	7.19, dd, 9.6, 2.4	124.9	-
7	C		155.9	
8	CH	7.50, dd, 2.4	106.8	C-7, C-4b, C-8a
9	C=O		181.6	
4a	C		148.6	
4b	C		149.7	
8a	C		122.4	
9a	C		114.2	
1'	CH₂	4.56, d, 6.6	65.6	C-7, C-2', C-3'
2'	CH	5.44, br t, 6.6	118.6	C-4', C-9'
3'	C		142.1	
4'	CH₂	2.05, m	39.5	C-2', C-5', C-6'
5'	CH₂	2.07, m	26.2	C-3', C-4', C-7'
6'	CH	5.04, br t, 6.3	123.7	-
7'	C		131.8	
8'	CH₃	1.60, s	25.5	C-6', C-7', C-10'
9'	CH₃	1.71, s	16.6	C-2', C-3', C-4'
10'	CH₃	1.54, s	17.5	C-6', C-7', C-8'
1''	C		134.3	
2''/6''	CH	7.81, dd, 9.0, 2.1	130.3	C-1'', C-4''
3''/5''	CH	7.65, dd, 8.7, 2.1	133.0	C-1'', C-4''
4''	C		130.6	
1'''	C		133.4	
2'''/6'''	CH	7.65, dd, 8.7, 2.1	129.8	C-1''', C-4'''
3'''/5'''	CH	7.63, dd, 8.7, 2.1	132.5	C-1''', C-4'''
4'''	C		130.1	

^{a}Recorded in 300 MHz.; ^{b}Recorded in 75 MHz.

3.1.1.21 Compound CC21
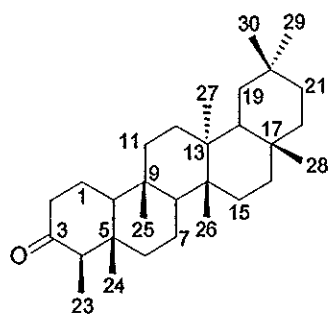
Compound **CC21** was obtained as a white crystal, mp 245-247 °C. The FT-IR spectrum of this compound showed the absorption band at 1715 cm^{-1} (carbonyl group). It gave a purple vanillin-sulfuric acid test indicating a triterpene.

The ^{1}H and ^{13}C NMR spectra **(Table 22)** showed characteristic of friedelan triterpenoids as seven methyl singlets at δ 0.72 (14.7), 0.87 (17.9), 0.95 (35.0), 1.00 (31.8), 1.01 (20.3), 1.05 (18.5), 1.18 (32.1) and one methyl doublet at δ 0.89 (3H, d, 6.3 Hz, H-23). The HMBC experiment **(Table 22)**, in which methyl protons at δ 0.89 (H-23) were correlated with the carbons at δ 42.2 (C-5), 58.2 (C-4) and 213.3 (C-3) confirmed the position of a carbonyl group at C-3. The fully HMBC correlations were also summarized in **Table 22** for confirmation of this structure. Finally, the structure of **CC21** was supported by X-ray structure as shown in **Figure 47**. Therefore, compound **CC21** was assigned as friedelin (Ahad *et al.*, 1991).
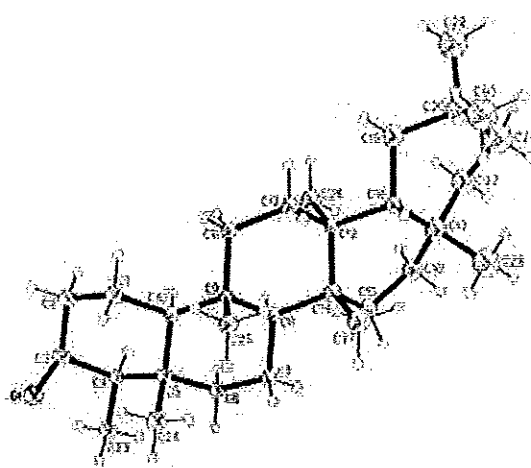
Figure 47. *ORTEP* plot of CC21

Table 22 NMR spectroscopic data of **CC21** in $CDCl_3$

Position	Type of C	$\delta_H{}^a$ (J in Herz)	$\delta_C{}^b$	HMBC ($^1H \rightarrow {}^{13}C$)
1	CH_2	1.64, m, 1.69, m	22.3	-
2	CH_2	2.36, m, 2.23, m	41.5	-
3	C	-	213.3	-
4	CH	2.24, m	58.2	
5	C	-	42.2	-
6	CH_2	2.44, m, 1.78, m	41.3	-
7	CH_2	1.52, m, 1.39, m	18.2	-
8	CH	1.42, m	53.1	-
9	C	-	37.4	-
10	CH_2	1.61, m, 1.43, m	35.6	-
11	CH_2	1.46, m, 1.34, m	30.5	-
12	C	-	39.7	-
13	C	-	38.3	-
14	CH_2	1.51, m, 1.29, m	32.4	-
15	CH_2	1.61, m, 1.36, m	36.0	-
16	C	-	30.0	-
17	CH	1.53, m	42.8	-
18	CH_2	1.64, m, 1.69, m	22.3	-
19	CH_2	1.62, m, 1.49, m	35.3	-
20	C	-	28.2	-
21	CH_2	1.48, m, 0.93, m	39.3	-
22	CH_2	1.50, m, 1.26, m	32.8	-
23	CH_3	0.89, d, 6.3 Hz	6.8	C-3, C-4, C-5
24	CH_3	0.72, s	14.7	C-4, C-5, C-6, C-10
25	CH_3	0.87, s	17.9	C-8, C-9, C-10, C-11
26	CH_3	1.01, s	20.3	C-8, C-13, C-14, C-15
27	CH_3	1.05, s	18.5	C-12, C-13, C-14, C-18
28	CH_3	1.18, s	32.1	C-16, C-17, C-18, C-22
29	CH_3	1.00, s	31.8	C-19, C- 20, C-21
30	CH_3	0.95, s	35.0	C-19, C-20, C-21

[a] Recorded in 300 MHz.

[b] Recorded in 75 MHz.

3.1.1.22 Compounds CC22 and CC23

CC22   CC23
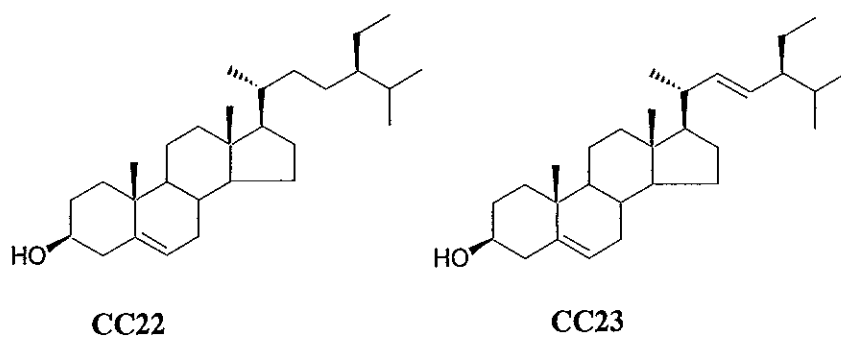
The mixture of compounds **CC22** and **CC23** was obtained as a white crystal. The ^{1}H NMR spectral data showed an oxymethine proton at δ 3.57-3.47 (*m*), three olefinic protons at δ 5.36-5.34 (*d*, 5.1 Hz), 5.16 (*dd*, 8.4, 15.1 Hz) and 5.01 (*dd*, 8.4, 15.1 Hz). The ^{1}H NMR data was corresponding to previous reported data, thus, the mixture was assigned as β-sitosterol (**CC22**) and stigmasterol (**CC23**) (Thongdeeying, 2005).

3.1.2 Biological activities of compounds CC1-CC20

The isolated compounds were evaluated for their antibacterial activities against both Gram-positive bacteria: *Bacillus subtilis*, *Staphylococcus aureus*, *Enterococcus faecalis* TISTR 459, Methicillin-Resistant *Staphylococcus aureus* (MRSA) ATCC 43300, Vancomycin-Resistant *Enterococcus faecalis* (VRE) ATCC 51299 and Gram-negative bacteria: *Salmonella typhi*, *Shigella sonnei* and *Pseudomonas aeruginosa*. All compounds were also submitted to antifungal assay against *Candida albicans*.

Table 23 Antimicrobial activity of compounds CC1-CC20

No	Antibacterial activity								Antifungal activity
	Gram-positive bacteria[a]					Gram-positive bacteria[b]			C. albicans[c]
	BS	SA	EF	MRSA	VRE	ST	SS	PA	
CC1	>300	>300	>300	>300	>300	>300	>300	>300	>300
CC2	300	300	>300	300	300	>300	>300	300	75
CC3	75	>300	300	>300	>300	>300	>300	>300	300
CC4	150	150	150	9.37	150	>150	>150	4.67	75
CC5	150	75	150	37.5	75	>150	>150	4.67	37.5
CC4: CC5[e]	75	150	75	9.37	150	>150	>150	4.67	75
CC6	>150	>150	>150	150	150	>150	>150	4.67	150
CC7	150	300	300	150	150	>300	>300	150	300
CC8	300	75	150	75	150	>300	>300	300	150
CC9	9.37	9.37	9.37	9.37	9.37	>300	>300	18.7	2.34
CC10	18.7	37.5	37.5	37.5	37.5	>300	>300	37.5	4.67
CC11	>300	300	>300	150	150	>300	>300	>300	300
CC12	75	>300	300	150	150	>300	>300	>300	>300
CC13	150	>150	150	37.5	150	>150	>150	4.67	18.7
CC14	150	>150	150	18.7	150	>150	>150	4.67	75
CC15	150	150	150	9.37	150	>150	>150	4.67	37.5
CC14: CC15[e]	75	37.5	37.5	4.67	37.5	>150	>150	4.67	37.5
CC16	150	>150	150	18.7	150	>150	>150	4.67	150
CC17	75	>150	75	37.5	150	>150	>150	4.67	18.7
CC18	>150	>150	150	18.7	150	>150	>150	4.67	150
CC19	>150	>150	>150	37.5	150	>150	>150	4.67	150
CC20	>150	>150	150	300	>300	>150	>150	4.67	>300
STD[d]	37.5	75	>300	150	300	>300	>300	300	300

[a] *Bacillus subtilis*, *Staphylococcus aureus*, *Enterococcus faecalis* TISTR 459, Methicillin-Resistant *Staphylococcus aureus* (MRSA) ATCC 43300, Vancomycin-Resistant *Enterococcus faecalis* (VRE) ATCC 51299.; [b] *Salmonella typhi*, *Shigella sonnei* and *Pseudomonas aeruginosa*.; [c] *Candida albicans*; [d] *1,3,7-trihydroxyxanthone*; [e] a mixture in 1:1 ratio

The results showed that most of the isolated compounds **CC4-CC6** and **CC13-CC15** exhibited strong antibacterial activity specifically against *P. aeruginosa* (**Table 23**) (Boonnak *et al.*, 2009). Interestingly, only compounds **CC9** and **CC10** exhibited strong activity against *Candida albicans*. It is important to note that compound **CC9** also exhibited broad spectrum antimicrobial activity against all Gram positive bacteria. Nearly all compounds which are active against *P. aeruginosa* are the 1,3,7-trihydroxyxanthones (**CC4** and **CC5**) or 1,3,7-trioxygenated xanthones with an oxygeranyl side-chain either at C-3 or C-7 and dihydroxyl groups (**CC14-CC15**) whose indicated structures might contribute to the strong antibacterial activity specifically against *P. aeruginosa*. However, when the free hydroxyl group of the 1,3,7-trihydroxyxanthone was cyclized onto the isoprenyl or geranyl side chain to form a chromane or chromene ring, the antibacterial activity against *P. aeruginosa* decreased drastically as shown in compounds **CC1**, **CC2** and **CC3a**.

In other previous reports, it was suggested that hydroxy xanthones play important roles in biological activity such as antibacterial (Boonnak *et al.*, 2006; Boonsri *et al.*, 2006), α-glucosidase inhibitory (Liu *et al.*, 2006), anti-tumor (Yoshimi *et al.*, 2001; Pedro *et al.*, 2002) and anti-inflammatory effects (Lin *et al.*, 1996). Because compounds **CC14** and **CC15** are the major components obtained from this plant, this prompts us to modify their structures for the structure-activity relationships (SARs). To investigate whether the free hydroxyl group was responsible for antibacterial activity, the acetylation with acetic anhydride in pyridine was therefore applied to **CC14** and **CC15**. Four acetylated geranyloxy xanthone derivatives: 3-acetoxy-7-geranyloxy-1-hydroxyxanthone (**CC16**) and 1,3-diacetoxy-7-geranyloxyxanthone (**CC17**) were obtained from **CC14**, whereas 7-acetoxy-3-geranyloxy-1-hydroxyxanthone (**CC18**) and 1,7-diacetoxy-3-geranyloxyxanthone (**CC19**) were obtained from **CC15** (Boonnak *et al.*, 2009).

The antibacterial activity of acetylated geranyloxy xanthone derivatives **CC16-CC19** and dibrosylate **CC20** were evaluated. All of them showed strong anti-*P. aeruginosa* (**Table 23**). This implied that the free hydroxyl groups should not be responsible for the inhibition of *P. aeruginosa*. For further investigation of the role of an oxygeranyl side-chain, a 1,3,7-trihydroxyxanthone (**CC21**) obtained from the dried fruits of *Cratoxylum cochinchinense* (Laphookhieo *et al.*, 2008), was tested against *P. aeruginosa*. It was inactive against Gram-negative bacteria as shown in **Table 23**. From these results, it can be suggested that the geranyl side-chain is necessary for anti-*P. aeruginosa* activity. Moreover, mixtures of compound **CC4** with compound **CC5**, and compound **CC14** with compound **CC15** were subjected to antimicrobial assay. Interestingly, the mixture of compounds **CC14** and **CC15**

significantly increased antibacterial activity against MRSA compared with the pure forms as indicated by the lower of MIC values shown in **Table 23**. The mixture of compounds **CC4** and **CC5**, on the other hand, did not show any significant differences for antibacterial activity compared to the pure forms as also shown in **Table 23**. Therefore, it may be proposed that the 1,3,7-trihydroxyxanthone with the isoprenyl or geranyl side chain at C-2 and C-4 in (**CC4**) and (**CC5**), respectively and 1,3,7-trioxygenated xanthone with the geranyl side chain at C-3 or C-7 in (**CC13-CC19**) are essential for their antibacterial activity against *P. aeruginosa*. Therefore, 1,3,7-trihydroxyxanthones (**CC4** and **CC5**) and 1,3,7-trioxy-genated xanthone with geranyl side-chain (**CC13-CC19**) should be considered as potent candidates as anti-*P. aeruginosa* (Boonnak *et al.*, 2009).

We further studied the possible mode of action of compounds **CC4** and **CC13** against *P. aeruginosa* by observing the bacteria cell morphology through scanning electron microscopy (SEM) at 3, 6, 9 and 15 h after applying compounds **CC4** and **CC13**. From the SEM results (see **Figures 48** and **49**), it was clearly indicated that the cell morphology of *P. aureginosa*, when treated with compounds **CC4** and **CC13**, started to deform at 3h onward, and at 15h, most cells were completely deformed whose results was correlated to their strong antibacterial activity (**Table 23**). Therefore, it can be suggested that compounds **CC4** and **CC13** may interact with or damage the cell wall of *P. aureginosa* as seen by the formation of pores on the cell wall of *P. aureginosa* (**Figures 48** and **49**) (Boonnak *et al.*, 2009).
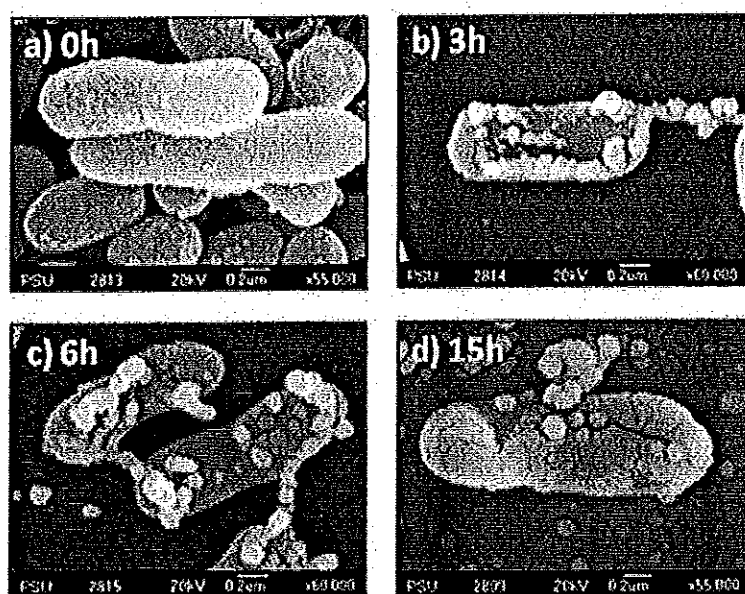
Firgure 48. SEM images of cell morphology of *P. aeruginosa* treated with compound **CC4** at different time.
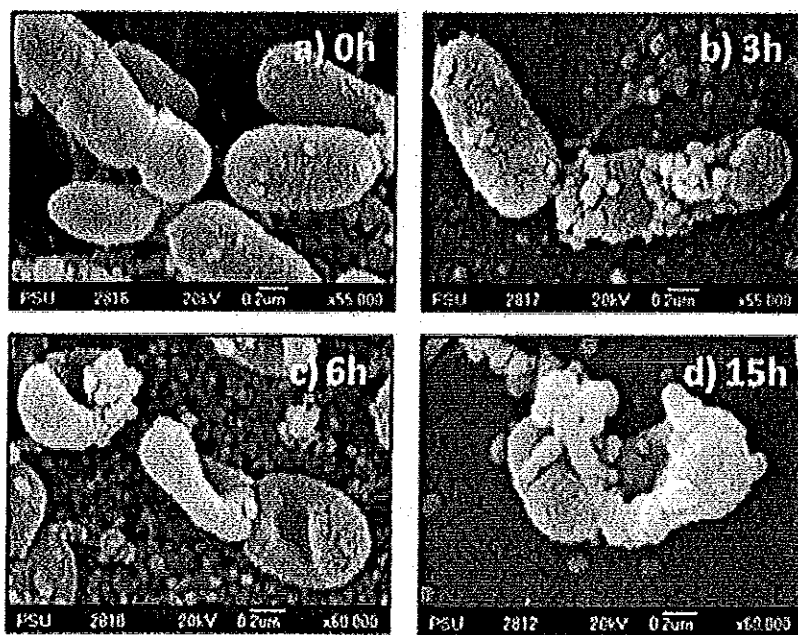
Firgure 49. SEM images of cell morphology of *P. aeruginosa*
treated with compound **CC13** at different time.

3.2 Isolated compounds from the roots and green fruits of Cratoxylum formosum ssp. pruniflorum

The roots of *C. formosum* ssp. *pruniflorum* (5.0 kg) was extracted with CH_2Cl_2 (2×2.0 L, for a week) at room temperature and was evaporated under reduced pressure to afford a deep green crude CH_2Cl_2 extract (58.87 g), which was further subjected to chromatography and/or recrystallization to yield two new xanthones: **CP1** and **CP2**, together with fifteen known xanthones: **CP3-CP17** and two known anthraquinones: **CP18** and **CP19**.
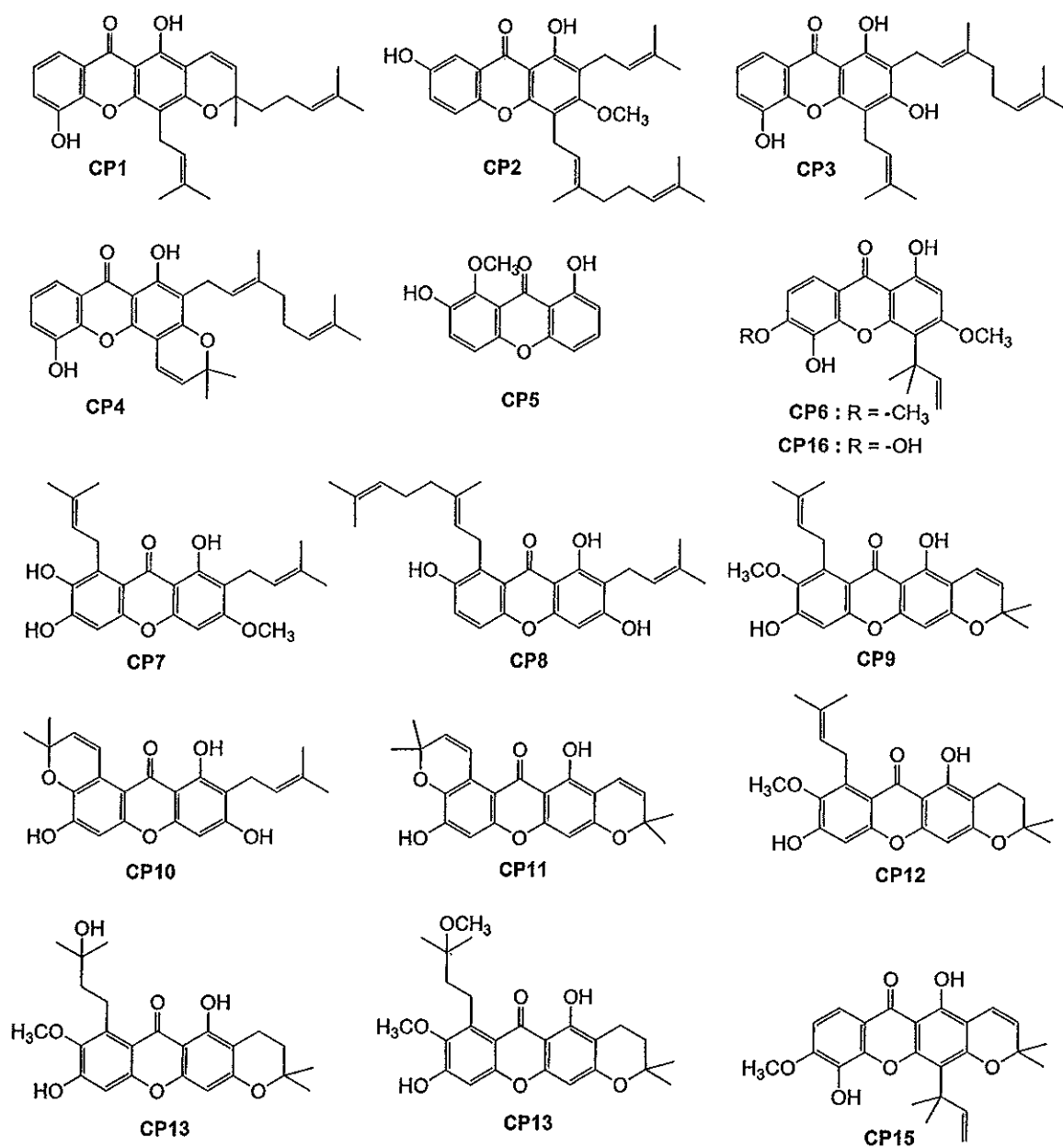
The structures of **CP1-CP16**
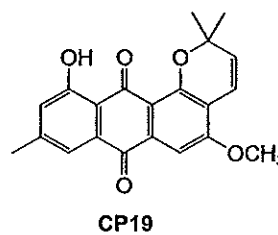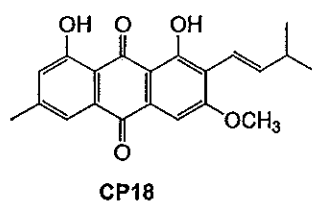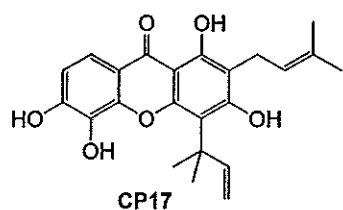
The structures of **CP17-CP19**

Air-dried green fruits of *C. formosum* ssp. *pruniflorum* (5.0 kg) were extracted with CH_2Cl_2 (2×20 L, for a week) at room temperature and was evaporated under reduced pressure to afford a deep green crude CH_2Cl_2 extract (31.42 g), which was further subjected to chromatography and/or recrystallization to yield three new xanthones: **CP20-CP22**, a new xanthonolignoid: **CP23** along with three known xanthones: **CP24-CP26**, a known flavonoid: **CP27** a known mixture of three triterpenes: **CP28-CP30** and a known triterpene: **CP31**.
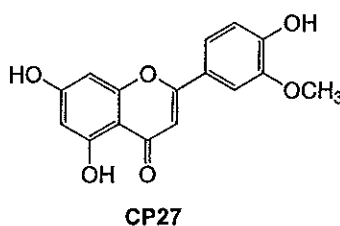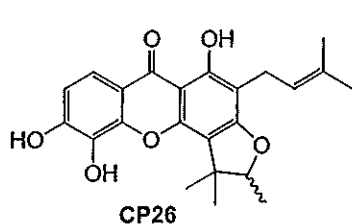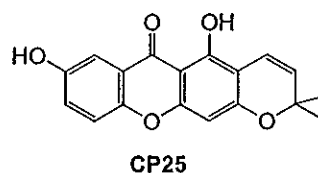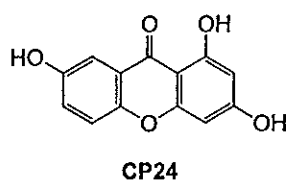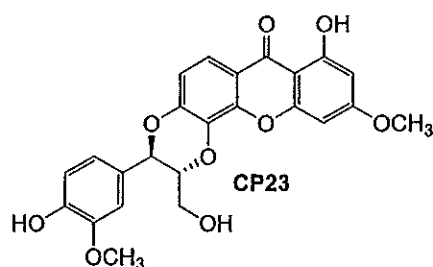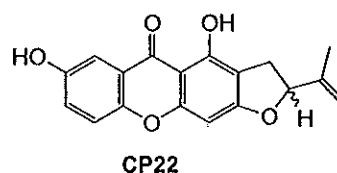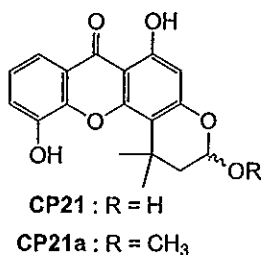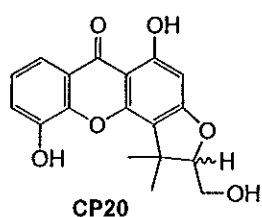
The structures of **CP20-CP27**
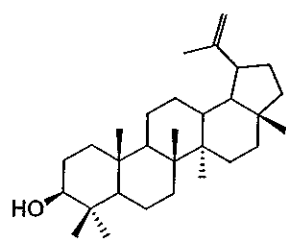
CP28
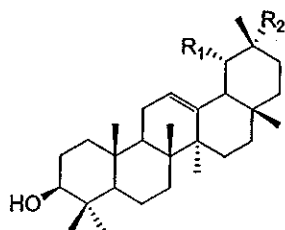
CP29 : R₁ = CH₃ R₂ = H
CP30 : R₁ = H R₂ = CH₃
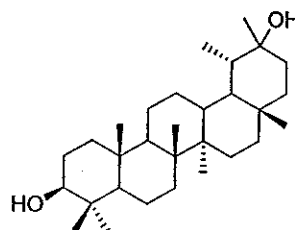
CP31

The structures of **CP28-CP31**

The structures of compounds **CP1-CP31** were completely characterized on the basis of spectroscopic analyses such as UV, FT-IR, 1D NMR, 2D NMR, MS and single crystal X-ray structure determination and also comparison of their spectroscopic data with those reported in the literature.

3.2.1 Structural elucidation of compounds CP1-CP31

3.2.1.1 Compound CP1
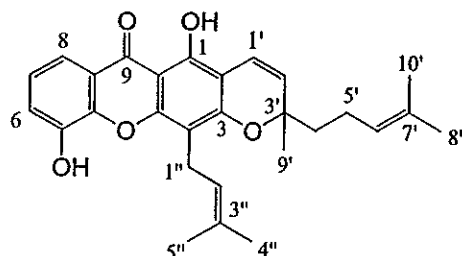
Compound **CP1** was isolated as yellow viscous oil, $[\alpha]^{25}_D = -18.2$ (c 0.285, CHCl$_3$). The HREIMS of **CP1** showed a molecular ion peak at m/z 446.2092 [M]$^+$, suggesting the molecular formula C$_{28}$H$_{30}$O$_5$. The UV-Vis spectrum showed absorption bands of a xanthone at 245, 260, 317 and 391 nm. The FT-IR spectrum exhibited the hydroxyl group at 3338 cm^{-1} and conjugated carbonyl group at 1647 cm^{-1}.

The ^{1}H NMR spectrum of **CP1** (**Table 24**) exhibited a chelated hydroxyl proton at δ 13.08 (s) and the characteristic signals of ABM trisubstituted benzene at δ 7.75 (dd, J = 7.5, 1.8 Hz, H-8), 7.30 (dd, J = 7.8, 1.8 Hz, H-6) and 7.23 (t, J = 7.8 Hz, H-7). The presence of the signals of a chromene ring bearing a methyl group and six-carbon side-chain of 4-methylpent-3-enyl group appeared at δ_H 6.79 (d, J = 10.2 Hz, H-1'), 5.56 (d, J = 10.2 Hz, H-2'), 5.09 (br t, J = 6.9 Hz, H-6'), 2.12 (m, H$_2$-4'), 1.78 (m, H$_2$-5'), 1.68 (s, CH$_3$-8') and 1.44 (s, CH$_3$-9' and CH$_3$-10'). The loss of 4-methylpent-3-enyl moiety in EI-MS, m/z 363 ([M]$^+$ −83), also supported the proposed structure. The location of a chromene ring was assigned by HMBC correlation of chelated hydroxyl group at δ 13.08 to the carbons at δ 103.3 (C-9a), 104.5 (C-2) and 156.1 (C-1), of the methine proton H-1' at δ 6.79 to the carbons at δ 104.5 (C-2), 156.1 (C-1) and 158.5 (C-3). It suggested that the chromene ring was attached to the carbon at C-2 and C-3, respectively. In addition, the location of an isoprenyl group at C-4 was assigned by HMBC correlations (**Table 24**) of the methylene protons at δ 3.50 (H$_2$-1') to the carbons at C-3 (δ 158.5), C-4 (δ 106.7) and C-4a (δ 154.7). The selected HMBC correlations were also shown in **Figure 50** for confirmation of this structure. Therefore, compound **CP1** was a new compound, and designed as pruniflorone K (Boonnak *et al.*, 2010).
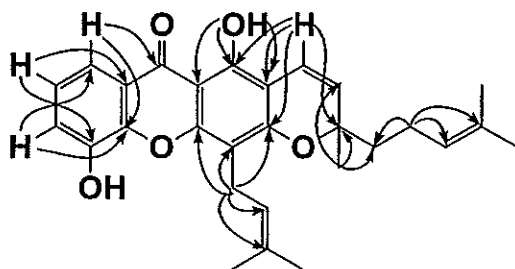
Figure 50. Selected HMBC correlations of **CP1**

Table 24 NMR spectroscopic data of **CP1** in CDCl₃

Position	Type of C	δ_{H}^{a} (*J* in Herz)	δ_{C}^{b}	HMBC (^{1}H→^{13}C)
1-OH	C	13.08, *s*	156.1	C-1, C-2, C-3, C-9a
2	C		104.5	
3	C		158.5	
4	C		106.7	
5-OH	C	5.73, *br s*	144.5	C-5, C-6, C-4b
6	CH	7.30, *dd*, 7.8, 1.8	119.8	C-8, C-4b
7	CH	7.23, *t*, 7.8	123.9	C-5, C-8a
8	CH	7.75, *dd*, 7.5, 1.8	116.8	C-9, C-4b
9	C=O		181.5	
4a	C		154.7	
4b	C		144.5	
8a	C		120.9	
9a	C		103.3	
1'	CH	6.79, *d*, 10.2	116.2	C-1, C-2, C-3, C-3'
2'	CH	5.56, *d*, 10.2	126.2	C-2, C-3', C-4', C-9'
3'	C		80.9	
4'	CH₂	1.78, *m*	41.8	C-2', C-3', C-4', C-5', C-9'
5'	CH₂	2.12, *m*	22.7	C-4', C-6', C-7'
6'	CH	5.09, *br t*, 6.9	123.7	C-5', C-8', C-10'
7'	C		131.9	
8'	CH₃	1.68, *s*	25.6	C-6', C-7'
9'	CH₃	1.45, *s*	27.2	C-2', C-3', C-4'
10'	CH₃	1.45, *s*	17.6	C-6', C-7'
1''	CH₂	3.50, *d*, 6.9	21.7	C-3, C-4, C-4a, C-2'', C-3''
2''	CH	5.23, *br t*, 6.9	122.7	C-1'', C-5''
3''	C		131.7	
4''	CH₃	1.72, *s*	25.5	C-2'', C-3''
5''	CH₃	1.84, *s*	17.9	C-2'', C-3''

^{a}Recorded in 300 MHz.

^{b}Recorded in 75 MHz.

3.2.1.2 Compound CP2
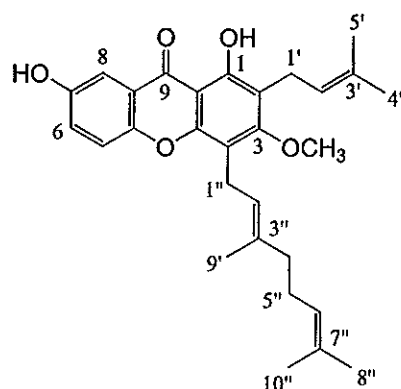
Compound **CP2** was isolated as pale yellow powder, mp 259-260 °C. The HREIMS of **CP2** showed a molecular ion peak at m/z 462.2408 $[M]^+$, suggesting the molecular formula $C_{29}H_{34}O_5$. The UV-Vis spectrum showed absorption bands of a xanthone at 245, 268, 317 and 388 nm. The FT-IR spectrum exhibited the hydroxyl group at 3421 cm^{-1} and conjugated carbonyl group at 1637 cm^{-1}.

The ^{1}H and ^{13}C NMR data of **CP2** (Table 25) were similar to those of **CC4** (Table 5), except for the appearance of a methoxyl group at δ 3.74 (s) instead of a free hydroxyl group at C-3 as in **CC4**. The location of a methoxyl group was assigned by using HMBC correlations (Table 25) in which the methylene protons H_2-1″ (δ 3.34) was correlated with C-1 (δ 158.8), C-2 (δ 116.9) and C-3 (δ 163.8), while a methoxyl group at δ 3.74 was correlated with C-3 (δ 163.8). From this HMBC assignment, it suggested that a methoxyl group at δ 3.74 should be attached to the xanthone nucleus at C-3. Moreover, the selected HMBC correlations of **CP2** were also illustrated in **Figure 51** for confirmation of this structure. Therefore, compound **CP2** was a new compound, and designed as pruniflorone L (Boonnak *et al.*, 2010).
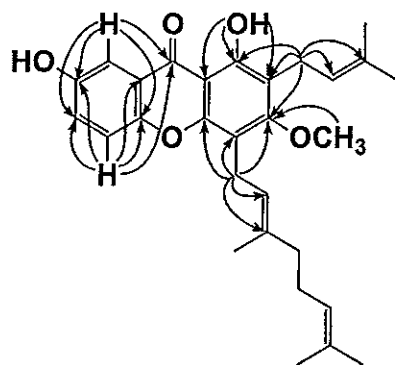
Figure 51. Selected HMBC correlations of **CP2**

Table 25 NMR spectroscopic data of **CP2** in CDCl$_3$

Position	Type of C	$\delta_H{}^a$ (*J* in Herz)	$\delta_C{}^b$	HMBC (^{1}H$\rightarrow$^{13}C)
1-OH	C	12.78, *s*	158.8	C-1, C-2, C-3, C-9, C-9a
2	C		116.9	
3	C		163.8	
4	C		113.1	
5	CH	7.28, *d*, 8.7	118.2	C-6, C-7, C-8, C-9, C-4b, C-8a
6	CH	7.50, *dd*, 8.7, 1.8	124.4	C-7, C-8, C-4b, C-8a
7-OH	C	6.55, *br s*	152.3	-
8	CH	7.54, *d*, 1.8	108.9	C-6, C-7, C-9, C-4b
9	C=O		181.6	
4a	C		153.4	
4b	C		150.7	
8a	C		120.7	
9a	C		105.8	
1'	CH$_2$	3.34, *d*, 6.6	22.6	C-1, C-2, C-3, C-2', C-3'
2'	CH	5.20, *br t*, 6.6	122.6	C-2, C-1', C-4', C-5'
3'	C		131.9	
4'	CH$_3$	1.63, *s*	25.7	C-2', C-3'
5'	CH$_3$	1.74, *s*	17.9	C-2', C-3'
1''	CH$_2$	3.36, *d*, 6.9	22.7	C-3, C-4, C-4a, C-2'', C-3'', C-9
2''	CH	5.15, *br t*, 6.9	122.8	C-4, C-1'', C-4'', C-9''
3''	C		135.3	
4''	CH$_2$	1.94, *m*	39.6	C-2'', C-3'', C-6''
5''	CH$_2$	1.98, *m*	26.6	C-3'', C-6'', C-7''
6''	CH	4.95, *br t*, 6.6	124.1	C-4'', C-5'', C-8'', C-10''
7''	C		131.4	
8''	CH$_3$	1.50, *s*	25.6	C-6'', C-7''
9''	CH$_3$	1.80, *s*	16.3	C-2'', C-3''
10''	CH$_3$	1.46, *s*	17.6	C-6'', C-7''
3-OCH$_3$	CH$_3$	3.74, *s*	61.9	C-3

^{a}Recorded in 300 MHz.

^{b}Recorded in 75 MHz.

3.2.1.3 Compound CP3
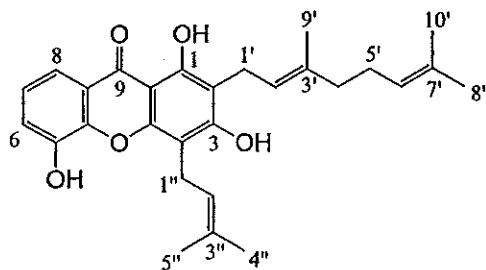
Compound **CP3** was isolated as yellow powder, mp 111-113 °C. The UV-Vis spectrum showed absorption bands of a xanthone at 245, 269, 332 and 377 nm (Boonsri *et al.*, 2006). The FT-IR spectrum exhibited the hydroxyl group at 3373 cm^{-1} and conjugated carbonyl group at 1650 cm^{-1} (Boonsri *et al.*, 2006).

The ^{1}H and ^{13}C NMR data of **CP3** (**Table 26**) were similar to those of **CP1** (**Table 24**), except for the appearance of a geranyl side chain at δ 5.29 (*br t*, J = 7.2 Hz, H-2'), 5.06 (*m*, H-6'), 3.50 (*d*, J = 7.2 Hz, H-1'), 2.11 (*m*, CH$_2$-4' and CH$_2$-5'), 1.85 (*s*, CH$_3$-9'), 1.68 (s, CH$_3$-8') and 1.60 (*s*, CH$_3$-10') 3.74 (*s*) instead of a chromene ring at C-2 as in **CP1**. The location of a geranyl side chain at C-2 was assigned by using HMBC correlations (**Table 26**) in which a chelated hydroxyl group 1-OH at δ 13.20 was correlated with C-1 (δ 158.6), C-2 (δ 108.9) and C-9a (δ 103.3), while the methylene protons H$_2$-1' (δ 3.50) was correlated with C-1 (δ 158.6), C-2 (δ 108.9) and C-3 (δ 161.1). The selected HMBC correlation of **CP3** was illustrated in **Figure 52** for confirmation of this structure. Therefore, compound **CP3** was assigned as Formoxanthone A (Boonsri *et al.*, 2006).
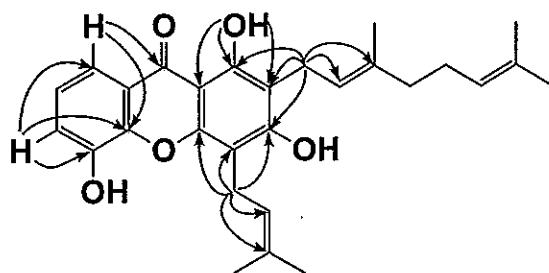
Figure 52. Selected HMBC correlations of **CP3**

Table 26 NMR spectroscopic data of **CP3** in CDCl₃

Position	Type of C	$\delta_H{}^a$ (*J* in Herz)	$\delta_C{}^b$	HMBC ($^1H\rightarrow{}^{13}C$)
1-OH	C	13.20, *s*	158.6	C-1, C-2, C-9a
2	C		108.9	
3-OH	C	6.57, *s*	161.1	C-2, C-3, C-4
4	C		105.7	
5-OH	C	5.86, *br s*	144.5	-
6	CH	7.29, *dd*, 7.8, 0.9	119.8	C-8, C-4b
7	CH	7.22, *t*, 7.8	123.8	C-5, C-8a
8	CH	7.75, *dd*, 7.8, 0.9	116.9	C-9, C-4b
9	C=O		181.1	
4a	C		152.5	
4b	C		144.3	
8a	C		120.9	
9a	C		103.3	
1'	CH₂	3.50, *d*, 7.2	21.6	C-1, C-2, C-3, C-2', C-3'
2'	CH	5.29, *br t*, 7.2	121.1	C-1', C-4', C-9'
3'	C		140.1	
4'	CH₂	2.11, *m*	39.7	C-2', C-3', C-6'
5'	CH₂	2.11, *m*	26.3	C-3', C-7'
6'	CH	5.06, *m*	123.7	-
7'	C		132.2	
8'	CH₃	1.68, s	25.7	C-6', C-7'
9'	CH₃	1.85, *s*	16.3	C-2', C-3'
10'	CH₃	1.60, s	17.7	C-6', C-7'
1''	CH₂	3.54, *d*, 6.9	22.0	C-3, C-4, C-4a, C-2'', C-3''
2''	CH	5.26, *br t*, 6.9	122.4	C-5''
3''	C		133.1	
4''	CH₃	1.74, *s*	25.6	C-2'', C-3''
5''	CH₃	1.86, *s*	17.9	C-2'', C-3''

*a*Recorded in 300 MHz.

*b*Recorded in 75 MHz.

3.2.1.4 Compound CP4
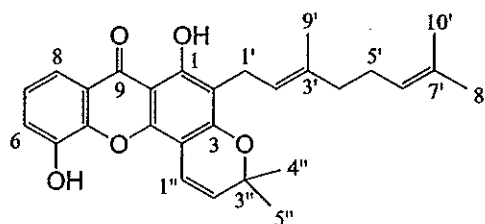
Compound **CP4** was isolated as yellow powder, mp 144-146 °C. The UV-Vis spectrum showed absorption bands of a xanthone at 253, 260, 319 and 367 nm (Boonsri *et al.*, 2006). The FT-IR spectrum exhibited the hydroxyl group at 3476 cm^{-1} and conjugated carbonyl group at 1646 cm^{-1} (Boonsri *et al.*, 2006).

The ^{1}H and ^{13}C NMR data of **CP4** (**Table 27**) were similar to those of **CP3** (**Table 26**), except for the appearance of a chromene ring at δ 6.80 (*d*, J = 9.9 Hz, H-1″), 5.65 (*d*, J = 9.9 Hz, H-2″) and 1.50 (*s*, CH$_3$-4″ and CH$_3$-5″) instead of an isoprenyl side chain at C-4 as in **CP3**. The location of a chromene ring at C-4 was assigned by using HMBC correlations (**Table 27**) of a methine proton H-1″ at δ 6.80 to the carbons at δ 158.7 (C-3), 149.2 (C-4a), 100.7 (C-4), and 78.1 (C-3″). The selected HMBC correlation of **CP4** was illustrated in **Figure 53** for confirmation of this structure. Therefore, compound **CP4** was assigned as Formoxanthone B (Boonsri *et al.*, 2006).
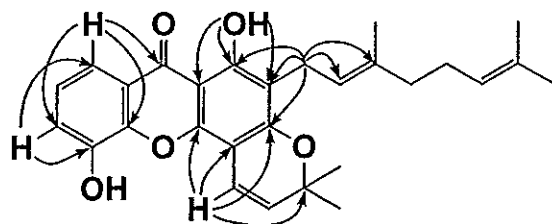
Figure 53. Selected HMBC correlations of **CP4**

Table 27 NMR spectroscopic data of **CP4** in $CDCl_3$

Position	Type of C	$\delta_H{}^a$ (J in Herz)	$\delta_C{}^b$	HMBC ($^1H\rightarrow{}^{13}C$)
1-OH	C	13.20, s	160.6	C-1, C-2, C-9a
2	C		112.3	
3	C		158.7	
4	C		100.7	
5	C		144.3	
6	CH	7.31, dd, 7.8, 1.8	120.1	C-5, C-8
7	CH	7.25, t, 7.8	123.9	C-5, C-8a
8	CH	7.79, dd, 7.8, 1.8	117.2	C-6, C-4b, C-9
9	C=O		180.8	
4a	C		149.2	
4b	C		144.1	
8a	C		121.2	
9a	C		103.2	
1'	CH_2	3.38, d, 7.2	21.1	C-1, C-2, C-3, C-2', C-3'
2'	CH	5.26, br t, 7.2	121.7	C-1', C-4', C-9'
3'	C		135.2	
4'	CH_2	2.02, m	39.8	C-5', C-9'
5'	CH_2	2.02, m	26.7	C-4'
6'	CH	5.09, br t, 7.2	124.4	-
7'	C		131.3	
8'	CH_3	1.64, s	25.7	C-6', C-7', C-10'
9'	CH_3	1.82, s	16.3	C-2', C-3', C-4'
10'	CH_3	1.58, s	17.7	C-6', C-7', C10'
1''	CH	6.80, d, 9.9	114.9	C-3, C-4, C-4a, C-3''
2''	CH	5.65, d, 9.9	127.4	C-4, C-3'', C-4'', C-5''
3''	C		78.1	
4''	CH_3	1.50, s	28.2	C-2'', C-3''
5''	CH_3	1.50, s	28.2	C-2'', C-3''

^{a}Recorded in 300 MHz.

^{b}Recorded in 75 MHz.

3.2.1.5 Compound CP5
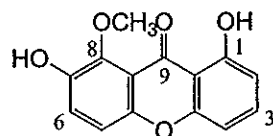
Compound **CP5** was isolated as yellow solid, mp 197-199 °C. The UV-Vis spectrum showed absorption bands of a xanthone at 254, 275 and 350 nm (Gottlieb *et al.*, 1966). The FT-IR spectrum exhibited the hydroxyl group at 3330 cm^{-1} and conjugated carbonyl group at 1647 cm^{-1} (Gottlieb *et al.*, 1966).

The ^{1}H NMR spectrum of **CP5** (**Table 28**) exhibited a chelated hydroxyl proton at δ 12.82 (*s*), a characteristic signals of 1,2,3-trisubstituted benzene at δ 7.49 (*t*, J = 8.4 Hz, H-3), 6.80 (*br d*, J = 8.4 Hz, H-4) and 6.70 (br d, J = 8.4 Hz, H-2), a pair of *ortho*-coupled aromatic protons at δ 7.36 (*dd*, J = 9.3 Hz, H-6) and 7.14 (*d*, J = 9.3 Hz, H-5), and a methoxyl group at δ 3.97 (s, OCH$_3$-8). The position of a methoxyl group at C-8 was assigned by HMBC correlations (**Table 28**) of an aromatic proton H-6 at δ 7.36 to the carbons at δ 150.9 (C-4b), 145.5 (C-7), 144.2 (C-8) and 114.2 (C-5), of the methoxyl protons (OCH$_3$-8) at δ 3.97 to the carbon at δ 144.2 (C-8). The selected HMBC correlations of **CP5** were shown in **Figure 54** for confirmation of this structure. Therefore, compound **CC5** was assigned as 1,7-dihydroxy-8-methoxyxanthone (Gottlieb *et al.*, 1966; Kijjoa *et al.*, 1998).
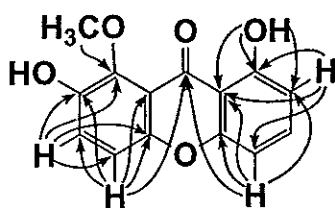
Figure 54. Selected HMBC correlations of **CP5**

Table 28 NMR spectroscopic data of **CP5** in CDCl$_3$

Position	Type of C	$\delta_H{}^a$ (*J* in Herz)	$\delta_C{}^b$	HMBC (^{1}H→^{13}C)
1-OH	C	12.82, *s*	162.0	C-1, C-2, C-3, C-9a
2	CH	6.70, *br d*, 8.4	110.2	C-1, C-4, C-9a
3	CH	7.49, *t*, 8.4	136.6	C-1, C-4a
4	CH	6.80, *br d*, 8.4	106.5	C-2, C-9, C-4a, C-9a
5	CH	7.14, *d*, 9.3	114.2	C-6, C-7, C-9, C-4b, C-8a
6	CH	7.36, *d*, 9.3	123.3	C-5, C-7, C-8, C-4b
7	C		145.5	
8	C		144.2	
9	C=O		182.0	
4a	C		155.8	
4b	C		150.9	
8a	C		114.8	
9a	C		109.0	
8-OCH$_3$	CH$_3$	3.97, *s*	62.8	C-8

^{a}Recorded in 300 MHz.

^{b}Recorded in 75 MHz.

3.2.1.6 Compound CP6
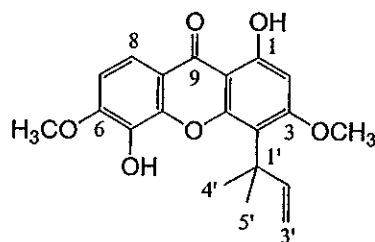
Compound **CP6** was isolated as yellow powder, mp 213-215 °C. The UV-Vis spectrum showed absorption bands of a xanthone at 217, 253, 286 and 327 nm (Hay *et al.*, 2008). The FT-IR spectrum exhibited the hydroxyl group at 3304 cm^{-1} and conjugated carbonyl group at 1643 cm^{-1} (Hay *et al.*, 2008).

The ^{1}H NMR spectrum of **CP6** (**Table 29**) exhibited a chelated hydroxyl proton at δ 12.82 (*s*), an pair of *ortho*-coupled aromatic protons at δ 7.68 (*d*, J = 9.2 Hz, H-8) and 6.90 (*d*, J = 8.8 Hz, H-7), a aromatic proton at δ 6.33 (*s*, H-2) and two methoxyl groups at δ 3.96 (*s*, OCH$_3$-6) and 3.82 (*s*, OCH$_3$-3). Moreover, the presence of a 1,1-dimethylallyl side chain was suggested by the following ^{1}H NMR spectral data at δ 6.58 (*dd*, J = 17.6, 10.4 Hz, H-2'), 5.10 (*d*, J = 17.6 Hz, 1H-3'), 4.97 (*d*, J = 10.4 Hz, 1H-3') and 1.56 (*s*, CH$_3$-4' and CH$_3$-5'). The location of a 1,1-dimethylallyl side chain at C-4 was assigned by using HMBC correlations (**Table 29**) of an aromatic proton H-2 at δ 6.33 to the carbons at C-1 (δ 162.5), C-3 (δ 165.4), C-4 (δ 113.6) and C-9a (δ 103.1), of the methine proton of a 1,1-dimethylallyl side chain at δ 6.58 (H$_2$-1') to the carbon at δ C-4 (δ 113.6). In HMBC spectrum (**Table 29**) of **CP6**, an aromatic proton H-7 at δ 6.90 showed correlations to the carbons at C-5 (δ 133.6), C-6 (δ 151.6) and C-8a (δ 114.2), while the methoxyl group at δ 3.96 also showed correlation to the carbon at C-6 (δ 151.6). It suggested that the methoxyl group at δ 3.96 was attached to the carbon at C-6. In addition, the position of a methoxyl group at C-3 (δ 3.82) was assigned by using HMBC correlation of an aromatic proton H-2 at δ 6.33 to the carbon at C-3 (δ 165.4), while a methoxyl group at δ 3.82 was also shown correlated to the carbon at C-3 (δ 165.4). The selected HMBC correlations of **CP6** were also given in **Figure 55** for confirmation of this structure. Structure of **CP6** was also verified by X-ray diffraction analysis and its structure was illustrated in **Figure 56**. Therefore, compound **CC6** was assigned as vieillardiixanthone B (Hay *et al.*, 2008; Boonnak *et al.*, 2010).
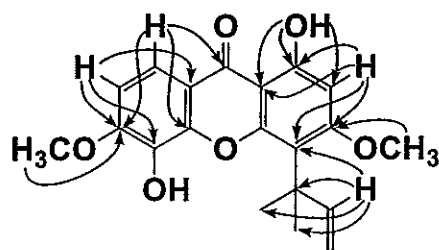
Figure 55. Selected HMBC correlations of **CP6**
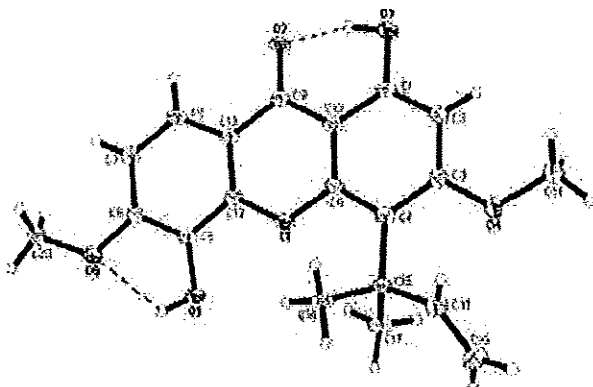
Figure 56. *ORTEP* plot of **CP6**

Table 29 NMR spectroscopic data of **CP6** in CDCl$_3$

Position	Type of C	$\delta_H{}^a$ (*J* in Herz)	$\delta_C{}^b$	HMBC (^{1}H→^{13}C)
1-OH	C	12.82, *s*	162.5	C-1, C-2, C-9a
2	CH	6.33, *s*	95.6	C-1, C-3, C-4, C-9a
3	C		165.4	
4	C		113.6	
5-OH	C	6.18, *br s*	133.6	C-5, C-6, C-4b
6	C		151.6	
7	CH	6.90, *d*, 8.8	108.3	C-5, C-6, C-8a
8	CH	7.68, *d*, 9.2	116.9	C-6, C-9, C-4b
9	C=O		181.1	
4a	C		154.0	
4b	C		144.6	
8a	C		114.2	
9a	C		103.1	
1′	C		41.5	
2′	CH	6.58, *dd*, 17.6, 10.4	155.1	C-4, C-1′, C-4′, C-5′
3′	CH$_2$	5.10, *d*, 17.6	104.5	C-1′, C-2′
		4.97, *d*, 10.4		C-1′
4′	CH$_3$	1.56, *s*	28.2	C-4, C-1′, C-2′
5′	CH$_3$	1.56, *s*	28.1	C-4, C-1′, C-2′
3-OCH$_3$	CH$_3$	3.82, *s*		C-3
6-OCH$_3$	CH$_3$	3.96, *s*	62.8	C-6

^{a}Recorded in 300 MHz.; ^{b}Recorded in 75 MHz.

3.2.1.7 Compound CP7
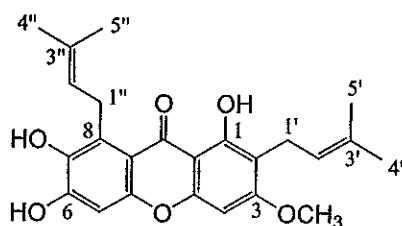
Compound **CP7** was isolated as yellow powder, m.p. 170-172 °C. The UV-Vis spectrum showed typical absorption bands of a xanthone at 209, 244, 261, 317 and 368 nm (Dechathai *et al.*, 2005). The FT-IR spectrum exhibited the hydroxyl group at 3306 cm^{-1} and conjugated carbonyl group at 1642 cm^{-1} (Dechathai *et al.*, 2005).

The ^{1}H and ^{13}C NMR data of **CP7** (**Table 30**) were similar to those of **CC8** (**Table 9**), except for the disappearance of a methoxyl signal in **CP7**. In the HMBC spectrum of **CP7** (**Table 30**), the methylene protons H$_2$-1′ (δ 3.35) of an isoprenyl side chain showed correlations to the aromatic carbons at δ 163.5 (C-3), 159.7 (C-1) and 111.4 (C-2), while a methoxyl group at δ 3.90 was correlated to C-3 (δ 163.5). It was suggested that a methoxyl group was connected to the carbon at C-7 of the xanthone nucleus. The selected HMBC correlation of **CC9** was also given in **Figure 57** for confirmation of this structure. Therefore, compound **CP7** was assigned as dulcisxanthone B (Dechathai *et al.*, 2005).
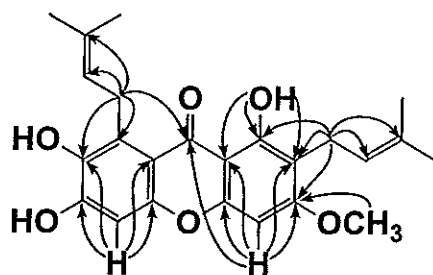
Figure 57. Selected HMBC correlations of **CP7**

Table 30 NMR spectroscopic data of **CP7** in CDCl$_3$

Position	Type of C	$\delta_H{}^a$ (J in Herz)	$\delta_C{}^b$	HMBC (^{1}H→^{13}C)
1-OH	C	13.44, s	159.7	C-1, C-2, C-9a
2	C		111.4	
3	C		163.5	
4	CH	6.32, s	88.8	C-2, C-3, C-9, C-4a, C-9a
5	CH	6.81, s	101.1	C-6, C-7, C-4b, C-8a
6	C		150.7	
7	C		139.6	
8	C		127.4	
9	C=O		182.6	
4a	C		155.3	
4b	C		153.5	
8a	C		111.7	
9a	C		103.9	
1'	CH$_2$	3.35, d, 7.2	21.4	C-1, C-2, C-3, C-2', C-3'
2'	CH	5.23, br t, 7.2	122.4	C-2
3'	C		131.7	
4'	CH$_3$	1.68, s	25.8	C-2', C-3', C-5'
5'	CH$_3$	1.80, s	17.8	C-2', C-3', C-4'
1''	CH$_2$	4.33, d, 6.9	26.0	C-7, C-8, C-8a, C-2'', C-3''
2''	CH	5.31, br t, 6.9	121.5	C-8
3''	C		135.6	
4''	CH$_3$	1.79, s	25.8	C-2'', C-3'', C-5''
5''	CH$_3$	1.89, s	18.1	C-2'', C-3'', C-4''
3-OCH$_3$	CH$_3$	3.90, s	55.8	C-3

^{a}Recorded in 300 MHz.

^{b}Recorded in 75 MHz.

3.2.1.8 Compound CP8
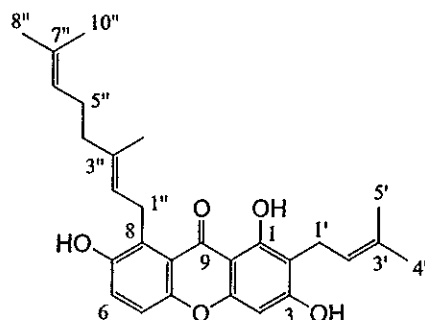
Compound **CP8** was isolated as yellow oil. The UV-Vis spectrum showed typical absorption bands of a xanthone at 241, 265, 314 and 382 nm (Laphookhieo *et al.*, 2009). The FT-IR spectrum exhibited the hydroxyl group at 3437 cm^{-1} and conjugated carbonyl group at 1638 cm^{-1} (Laphookhieo *et al.*, 2009).

The ^1H NMR spectrum of **CP8** (**Table 31**) exhibited a chelated hydroxyl proton at δ 13.44 (*s*), a pair of *ortho*-coupled aromatic protons at δ 7.13 (*s*, H-5) and 7.13 (s, H-6), an aromatic proton at δ 6.24 (*s*, H-4) and an isoprenyl side chain at δ 5.23 (*br t*, J = 7.2 Hz, H-2'), 3.85 (*d*, J = 7.2 Hz, H-1'), 1.78 (*s*, CH_3-5') and 1.70 (s, CH_3-4'). Moreover, the presence of a geranyl side chain was suggested by the following ^1H NMR spectral data at δ 5.20 (*br t*, J = 7.2 Hz, H-2''), 4.97 (*br t*, J = 6.0 Hz, H-6''), 4.25 (*d*, J = 6.9 Hz, H-3''), 2.02 (*m*, CH_2-4'' and CH_2-5''), 1.80 (*s*, CH_3-9''), 1.59 (*s*, CH_3-8'') and 1.51 (*s*, CH_3-10'). The location of an isoprenyl side chain at C-4 was assigned by using HMBC correlations (**Table 31**) of a chelated hydroxyl group 1-OH at δ 13.44 to the carbons at C-1 (δ 169.7), C-2 (δ 108.4) and C-9a (δ 104.1), of the methylene protons H-1'' at δ 3.38 to the carbons at C-1 (δ 160.7), C-2 (δ 108.4) and C-3 (δ 162.2). In HMBC spectral data (**Table 31**) of **CP8**, an aromatic proton H-6 at δ 7.13 showed correlations to the carbons at C-7 (δ 151.3), C-8 (δ 127.1) and C-4b (δ 152.0), while the methylene protons of a geranyl side chain at δ 4.25 also showed correlation to the carbon at C-7 (δ 151.3), C-8 (δ 127.1) and C-8a (δ 118.5). It was suggested that a geranyl side chain was attached to the carbon at C-8. The selected HMBC correlations of **CP8** were also given in **Figure 58** for confirmation of this structure. Therefore, compound **CC8** was assigned as cochinxanthone E (Laphookhieo *et al.*, 2009).
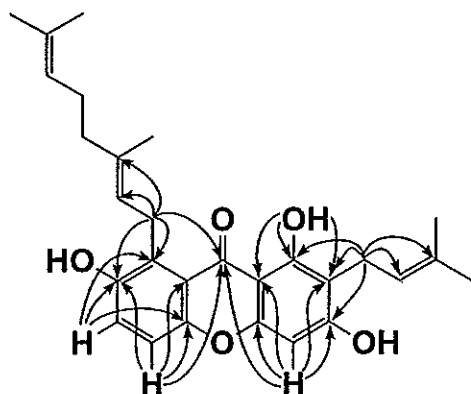
Figure 58. Selected HMBC correlations of **CP8**

Table 31 NMR spectroscopic data of **CP8** in CDCl₃

Position	Type of C	$\delta_H{}^a$ (J in Herz)	$\delta_C{}^b$	HMBC ($^1H{\rightarrow}{}^{13}C$)
1-OH	C	13.44, s	160.7	C-1, C-2, C-9a
2	C		108.4	
3	C		162.2	
4	CH	6.24, s	93.2	C-2, C-3, C-9, C-4a, C-9a
5	CH	7.13, s	116.7	C-7, C-9, C-4b, C-8a
6	CH	7.13, s	123.7	C-7, C-8, C-4b
7	C		151.3	
8	C		127.1	
9	C=O		183.5	
4a	C		155.3	
4b	C		152.0	
8a	C		118.5	
9a	C		104.1	
1'	CH₂	3.45, d, 7.2	21.5	C-1, C-2, C-3, C-2', C-3'
2'	CH	5.23, br t, 7.2	121.4	C-4', C-5'
3'	C		135.7	
4'	CH₃	1.70, s	25.8	C-2', C-3'
5'	CH₃	1.78, s	17.9	C-2', C-3'
1''	CH₂	4.25, d, 7.2	25.7	C-7, C-8, C-8a, C-2'', C-3''
2''	CH	5.20, br t, 7.2	121.4	C-4'', C-9''
3''	C		138.7	
4''	CH₂	2.02, m	39.7	C-9''
5''	CH₂	2.02, m	26.4	C-4''
6''	CH	4.97, br t, 6.0	123.7	-
7''	C		131.9	
8''	CH₃	1.59, s	25.8	C-6'', C-7''
9''	CH₃	1.80, s	16.4	C-2'', C-3'', C-4''
10''	CH₃	1.51, s	17.7	C-6'', C-7''

^{a}Recorded in 300 MHz.; ^{b}Recorded in 75 MHz.

3.2.1.9 Compound CP9
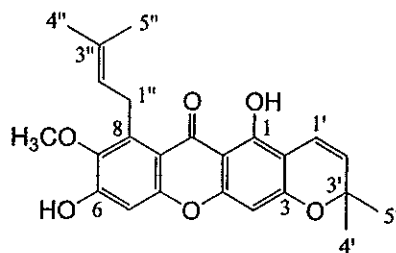
Compound **CP9** was isolated as yellow powder, mp 156-157 °C. The ^{1}H NMR spectrum of **CP9** (**Table 32**) exhibited a chelated hydroxyl proton at δ 13.63 (*s*), two aromatic protons at δ 6.81 (*s*, H-5) and 6.17 (s, H-4), a methoxyl group at δ 3.73 (*s*, OCH$_3$-7) and a chromene ring at δ 6.66 (*d*, J = 9.9 Hz, H-1'), 5.50 (*d*, J = 9.9 Hz, H-2') and 1.39 (*s*, CH$_3$-4' and CH$_3$-5'). Moreover, the presence of an isoprenyl side chain was suggested by the following ^{1}H NMR spectral data at δ 5.19 (*br t*, J = 6.6 Hz, H-2''), 4.01 (*d*, J = 6.3 Hz, H-1''), 1.76 (*s*, CH$_3$-5'') and 1.62 (*s*, CH$_3$-4'').

In HMBC spectral data (**Table 32**) of **CP9**, a chelated hydroxyl group 1-OH at δ 13.63 showed correlations to the carbons at C-1 (δ 157.9), C-2 (δ 104.5), C-9 (δ 182.0) and C-9a (δ 103.7), while the methine proton of a chromene ring at δ 6.66 also showed correlations to the carbons at C-1 (δ 157.9), C-2 (δ 104.5), C-3 (δ 159.8) and C-3' (δ 77.9). It was suggested that a chromene ring was fused to the carbon at C-2 and C-3 in a linear fashion. The location of an isoprenyl side chain at C-8 was assigned by using HMBC correlations (**Table 32**) of the methylene protons H-1'' at δ 4.01 to the carbons at C-7 (δ 142.7), C-8 (δ 137.0), C-8a (δ 112.1), C-2'' (δ 123.2) and C-3'' (δ 132.1), of a methoxyl group at δ 3.73 to the carbon at C-7 (δ 142.7). From this assignment, a methoxyl group could be attached to the carbon at C-7. The selected HMBC correlations of **CP9** were also given in **Figure 59** for confirmation of this structure. Therefore, compound **CP9** was assigned as 5,9-dihydroxy-8-methoxy-2,2-dimethyl-7-(3-methyl-but-2-enyl)-2H,6H-pyrano[3,2b]xanthone (Sen *et al.*, 1980).
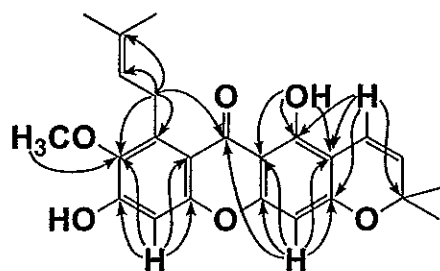
Figure 59. Selected HMBC correlations of CP9

Table 32 NMR spectroscopic data of CP9 in CDCl$_3$

Position	Type of C	$\delta_H{}^a$ (*J* in Herz)	$\delta_C{}^b$	HMBC (^{1}H→^{13}C)
1-OH	C	13.63, *s*	157.9	C-1, C-2, C-9, C-9a
2	C		104.5	
3	C		159.8	
4	CH	6.17, *s*	94.1	C-2, C-3, C-9, C-4a, C-9a
5	CH	6.81, *s*	101.7	C-6, C-7, C-8, C-4b, C-8a
6	C		155.7	
7	C		142.7	
8	C		137.0	
9	C=O		182.0	
4a	C		156.2	
4b	C		154.8	
8a	C		112.1	
9a	C		103.7	
1'	CH	6.66, *d*, 9.9	115.7	C-1, C-2, C-3, C-3'
2'	CH	5.50, *d*, 9.9	127.1	C-2, C-3', C-4', C-5'
3'	C		77.9	
4'	CH$_3$	1.39, *s*	28.3	C-2', C-3'
5'	CH$_3$	1.39, *s*	28.3	C-2', C-3'
1''	CH$_2$	4.01, *d*, 6.3	26.5	C-7, C-8, C-8a, C-2'', C-3''
2''	CH	5.19, *br t*, 6.6	123.2	C-8, C-4'', C-5''
3''	C		132.1	
4''	CH$_3$	1.62, *s*	25.8	C-2'', C-3'', C-5''
5''	CH$_3$	1.76, *s*	18.2	C-2'', C-3'', C-4''
7-OCH$_3$	CH$_3$	3.73, *s*	61.9	C-7

^{a}Recorded in 300 MHz.

^{b}Recorded in 75 MHz.

3.2.1.10 Compound CP10
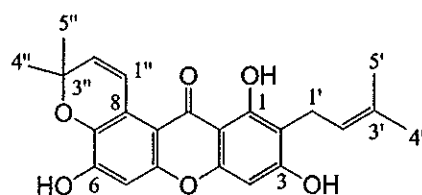
Compound **CP10** was isolated as yellow powder, mp 190-192 The UV-Vis spectrum showed typical absorption bands of a xanthone at 247, 267, 339 and 390 nm (Sen *et al.*, 1982). The FT-IR spectrum exhibited the hydroxyl group at 3480 cm^{-1} and conjugated carbonyl group at 1650 cm^{-1} (Sen *et al.*, 1982).

The ^{1}H NMR spectrum of **CP10** (**Table 33**) exhibited a chelated hydroxyl proton at δ 13.62 (*s*), two aromatic protons at δ 6.74 (*s*, H-5) and 6.25 (s, H-4) and an isoprenyl side chain at δ 5.23 (*br t*, J = 6.6 Hz, H-2′), 3.38 (*d*, J = 6.6 Hz, H-1′), 1.77 (*s*, CH$_3$-5′) and 1.70 (*s*, CH$_3$-4′). Moreover, the presence of a chromene ring was suggested by the following ^{1}H NMR spectral data at δ 7.95 (*d*, J = 10.2 Hz, H-1″), 5.75 (*d*, J = 10.2 Hz, H-2″) and 1.43 (*s*, CH$_3$-4″ and CH$_3$-5″). In HMBC spectral data (**Table 33**) of **CP10**, a chelated hydroxyl group 1-OH at δ 13.62 showed correlations to the carbons at C-1 (δ 160.5), C-2 (δ 108.5) and C-9a (δ 103.8), while the methylene protons of an isoprenyl side chain at δ 3.38 also showed correlations to the carbons at C-2 (δ 108.5), C-3 (δ 161.8), C-2′ (δ 121.5) and C-3′ (δ 135.6). It was suggested that an isoprenyl side chain could be attached to the carbon at C-2. The location of a chromene ring at C-8 was assigned by using HMBC correlations (**Table 33**) of the methine proton H-1″ at δ 7.95 to the carbons at C-7 (δ 136.8), C-8 (δ 119.8), C-8a (δ 108.6) and C-3″ (δ 76.7). The selected HMBC correlations of **CP10** were also given in **Figure 60** for confirmation of this structure. Therefore, compound **CP10** was assigned as garcinone B (Sen *et al.*, 1982).
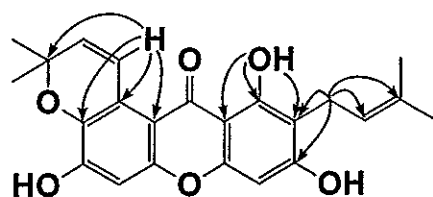
Figure 60. Selected HMBC correlations of **CP10**

Table 33 NMR spectroscopic data of **CP10** in CDCl$_3$

Position	Type of C	$\delta_H{}^a$ (*J* in Herz)	$\delta_C{}^b$	HMBC (^{1}H→^{13}C)
1-OH	C	13.62, *s*	160.5	C-1, C-2, C-9a
2	C		108.5	
3	C		161.8	
4	CH	6.25, *s*	93.4	-
5	CH	6.74, *s*	102.3	-
6	C		153.1	
7	C		136.8	
8	C		119.8	
9	C=O		180.3	
4a	C		155.2	
4b	C		151.7	
8a	C		108.6	
9a	C		103.8	
1'	CH$_2$	3.38, *d*, 6.6	21.4	C-2, C-3, C-2', C-3'
2'	CH	5.23, *br t*, 6.6	121.5 .	C-1', C-4'
3'	C		135.6	
4'	CH$_3$	1.70, *s*	25.8	C-2', C-3', C-5'
5'	CH$_3$	1.77 *s*	17.9	C-1', C-3', C-4'
1''	CH	7.95, *d*, 10.2	121.0	C-7, C-8, C-8a, C-3''
2''	CH	5.75, *d*, 10.2	132.3	C-7, C-8, C-3'', C-4'', C-5''
3''	C		76.7	
4''	CH$_3$	1.43, *s*	27.3	C-2'', C-3''
5''	CH$_3$	1.43, *s*	27.3	C-2'', C-3''

^{a}Recorded in 300 MHz.

^{b}Recorded in 75 MHz.

3.2.1.11 Compound CP11
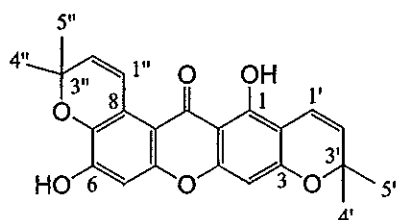
Compound **CP11** was isolated as yellow powder, mp 205-207 °C. The UV-Vis spectrum showed typical absorption bands of a xanthone at 287, 290, 310 and 385 nm (Marques *et al.*, 2000). The FT-IR spectrum exhibited the hydroxyl group at 3355 cm^{-1} and conjugated carbonyl group at 1621 cm^{-1} (Marques *et al.*, 2000).

The ^{1}H and ^{13}C NMR data of **CP11** (**Table 34**) were similar to those of **CP10** (**Table 33**), except for the appearance of a chromene ring at δ 6.65 (*d*, J = 10.2 Hz, H-1'), 5.50 (*d*, J = 10.2 Hz, H-2') and 1.40 (*s*, CH$_3$-4' and CH$_3$-5') instead of an isoprenyl side chain at C-2 as in **CP10**. The location of a chromene ring was assigned by using HMBC correlations (**Table 34**) of a chelated hydroxyl group 1-OH at δ 13.55 to the carbons at C-1 (δ 157.8), C-2 (δ 104.4), C-3 (δ 159.9), C-9 (182.4) and C-9a (δ 103.9), of a methine proton H-1' of a chromene ring at δ 6.65 to the carbons at C-1 (δ 157.8), C-2 (δ 104.4), C-3 (δ 159.9) and C-3' (δ 78.0). From this assignment, it was suggested that a chromene ring could be attached to the carbon at C-2 and C-3 with the xanthone nucleus in a linear fashion. The selected HMBC correlation of **CP11** was also given in **Figure 61** for confirmation of this structure. Structure of **CP11** was further supported by the X-ray structure as illustrated in **Figure 62** (Chantrapromma *et al.*, 2010). Therefore, compound **CP11** was assigned as brasilixanthone (Marques *et al.*, 2000; Chantrapromma *et al.*, 2010).
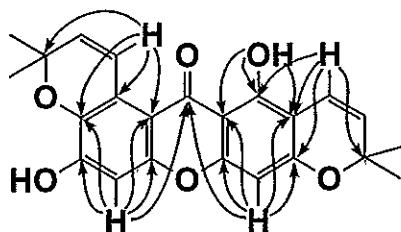
Figure 61. Selected HMBC correlations of **CP11**
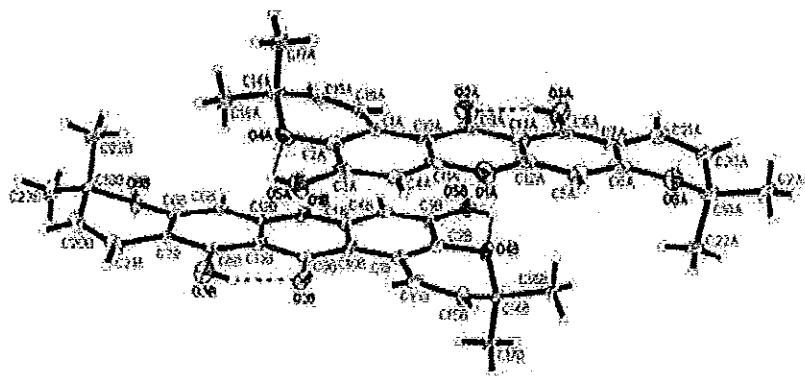
Figure 62. *ORTEP* plot of **CP11**

Table 34 NMR spectroscopic data of **CP11** in CDCl₃

Position	Type of C	$\delta_H{}^a$ (*J* in Herz)	$\delta_C{}^b$	HMBC (^{1}H→^{13}C)
1-OH	C	13.55, *s*	157.8	C-1, C-2, C-3, C-9, C-9a
2	C		104.4	
3	C		159.9	
4	CH	6.19, *s*	94.3	C-2, C-3, C-9, C-4a, C-8a
5	CH	6.75, *s*	102.4	C-6, C-7, C-9, C-4b, C-8a
6	C		153.1	
7	C		136.8	
8	C		119.7	
9	C=O		182.4	
4a	C		156.6	
4b	C		150.9	
8a	C		108.6	
9a	C		103.9	
1'	CH	6.65, *d*, 10.2	115.7	C-1, C-2, C-3, C-3'
2'	CH	5.50, *d*, 10.2	127.2	C-2, C-3', C-4', C-5'
3'	C		78.0	
4'	CH₃	1.40, *s*	28.3	C-1', C-2', C-3'
5'	CH₃	1.40, *s*	28.3	C-1', C-2', C-3'
1''	CH	7.94, *d*, 10.2	120.9	C-7, C-8, C-8a, C-3''
2''	CH	5.75, *d*, 10.2	132.3	C-7, C-8, C-4'', C-5''
3''	C		76.8	
4''	CH₃	1.42, *s*	27.3	C-7, C-1'', C-2'', C-3''
5''	CH₃	1.42, *s*	27.3	C-7, C-1'', C-2'', C-3''

^{a}Recorded in 300 MHz.

^{b}Recorded in 75 MHz.

3.2.1.12 Compound CP12
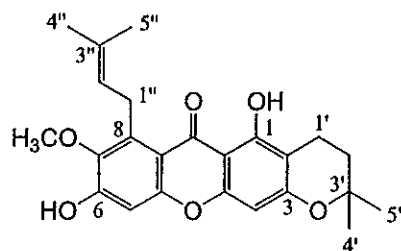
Compound **CP12** was isolated as yellow powder, mp 154-155 °C. The ^{1}H NMR spectrum of **CP12** (**Table 35**) exhibited a chelated hydroxyl proton at δ 13.73 (s), two aromatic protons at δ 6.82 (s, H-5) and 6.23 (s, H-4), a methoxyl group at δ 3.80 (s, OCH$_3$-7) and a chromane ring at δ 2.71 (t, J = 6.6 Hz, H-1'), 1.83 (t, J = 6.6 Hz, H-2') and 1.37 (s, CH$_3$-4' and CH$_3$-5'). Moreover, the presence of an isoprenyl side chain was suggested by the following ^{1}H NMR spectral data at δ 5.27 (br t, J = 6.3 Hz, H-2''), 4.10 (d, J = 6.3 Hz, H-1''), 1.83 (s, CH$_3$-5'') and 1.69 (s, CH$_3$-4'').

In HMBC spectral data (**Table 35**) of **CP12**, a chelated hydroxyl group 1-OH at δ 13.73 showed correlations to the carbons at C-1 (δ 160.6), C-2 (δ 103.8) and C-9a (δ 102.9), while the methylene protons of a chromane ring at δ 2.71 also showed correlations to the carbons at C-1 (δ 160.6), C-2 (δ 103.8), C-3 (δ 160.7), C-2' (δ 31.9) and C-3' (δ 76.0). It was suggested that a chromane ring was fused to the carbon at C-2 and C-3 in a linear fashion. The position of an isoprenyl side chain at C-8 was assigned by using HMBC correlations (**Table 35**) of the methylene protons H-1'' at δ 4.10 to carbon at C-7 (δ 142.4), C-8 (δ 136.9), C-8a (δ 112.1), C-2'' (δ 123.3) and C-3'' (δ 132.2), of a methoxyl group at δ 3.80 to the carbon at C-7 (δ 142.4). From this assignment, a methoxyl group could be attached to the carbon at C-7. The selected HMBC correlations of **CP12** were also given in **Figure 63** for confirmation of this structure. Therefore, compound **CP12** was assigned as 3-isomangostin (Mahabusarakam *et al.*, 1987).
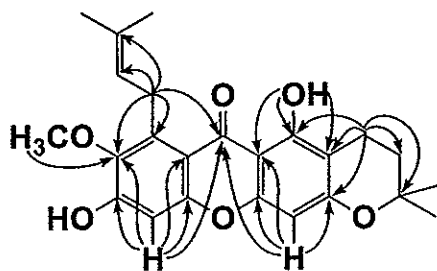
Figure 63. Selected HMBC correlations of **CP12**

Table 35 NMR spectroscopic data of **CP12** in CDCl₃

Position	Type of C	$\delta_H{}^a$ (*J* in Herz)	$\delta_C{}^b$	HMBC (^{1}H→^{13}C)
1-OH	C	13.73, s	160.6	C-1, C-2, C-9a
2	C		103.8	
3	C		160.7	
4	CH	6.23, s	94.0	C-3, C-9, C-4a, C-8a
5	CH	6.82, s	101.6	C-6, C-7, C-9, C-4b, C-8a
6	C		155.9	
7	C		142.4	
8	C		136.9	
9	C=O		182.0	
4a	C		152.5	
4b	C		154.7	
8a	C		112.1	
9a	C		102.9	
1′	CH₂	2.71, t, 6.6	16.1	C-1, C-2, C-3, C-2′, C-3′
2′	CH₂	1.83, t, 6.6	31.9	C-2, C-1′, C-3′
3′	C		76.0	
4′	CH₃	1.37, s	26.7	C-4′, C-5′
5′	CH₃	1.37, s	26.7	C-4′, C-5′
1″	CH₂	4.10, d, 6.3	26.5	C-7, C-8, C-8a, C-2″, C-3″
2″	CH	5.27, br t, 6.3	123.3	-
3″	C		132.2	
4″	CH₃	1.69, s	25.8	C-2″, C-3″, C-5″
5″	CH₃	1.83, s	18.2	C-2″, C-3″, C-4″
7-OCH₃	CH₃	3.80, s	62.0	C-7

^{a}Recorded in 300 MHz.

^{b}Recorded in 75 MHz.

3.2.1.13 Compound CP13
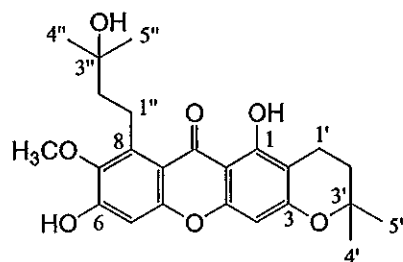
Compound **CP13** was isolated as yellow powder, mp 180-182 °C. The ^{1}H NMR spectrum of **CP13** (**Table 36**) were closely similar to those **CP12**, except for the appearance of a 3-hydroxyl-3-methylbutyl side chain at δ 3.42 (*br t*, J = 8.1 Hz, H-1″), 1.79 (*br* t, J = 8.1 Hz, H-2″) and 1.33 (*s*, CH$_3$-4″ and CH$_3$-5″) instead of an isoprenyl side chain at C-8 as in **CP12**.

In HMBC spectral data (**Table 36**) of **CP13**, the methylene protons of a chromane ring at δ 2.70 showed correlations to the carbons at C-1 (δ 160.5), C-2 (δ 103.9), C-3 (δ 160.9), C-2' (δ 31.9) and C-3' (δ 76.1). It was suggested that a chromane ring was fused to the carbon at C-2 and C-3 in a linear fashion. The position of a 3-hydroxyl-3-methylbutyl side chain at C-8 was assigned by using HMBC correlations (**Table 36**) of the methylene protons H-1″ at δ 3.42 to the carbons at C-7 (δ 142.5), C-8 (δ 138.4) and C-3″ (δ 70.8), of a methoxyl group at δ 3.86 to the carbon at C-7 (δ 142.5). From this assignment, it was also implied that a methoxyl group could be attached to the carbon at C-7. The selected HMBC correlations of **CP13** were also given in **Figure 64** for confirmation of this structure. Therefore, compound **CP13** was assigned as 3,4-Dihydro-5,9-dihydroxy-7-(3-hydroxy-3-methylbutyl)-8-methoxy-2,2-dimethyl-2H,6H-pyrano[3,2b]xanthone. (Dutta *et al.*, 1987).

Figure 64. Selected HMBC correlations of **CP13**

Table 36 NMR spectroscopic data of **CP13** in $CDCl_3$

Position	Type of C	$\delta_H{}^a$ (J in Herz)	$\delta_C{}^b$	HMBC ($^1H \rightarrow {}^{13}C$)
1-OH	C	13.60, s	160.5	-
2	C		103.9	
3	C		160.9	
4	CH	6.22, s	94.1	C-3, C-9, C-4a
5	CH	6.83, s	101.8	C-6, C-7, C-9, C-4b, C-8a
6	C		154.7	
7	C		142.5	
8	C		138.4	
9	C=O		182.0	
4a	C		154.8	
4b	C		156.1	
8a	C		111.8	
9a	C		102.8	
1'	CH_2	2.70, t, 6.9	16.1	C-1, C-2, C-3, C-2', C-3'
2'	CH_2	1.83, t, 6.6	31.9	C-2, C-3', C-4', C-5'
3'	C		76.1	
4'	CH_3	1.37, s	26.8	C-2', C-3'
5'	CH_3	1.37, s	26.8	C-2', C-3'
1''	CH_2	3.42, br t, 8.1	22.1	C-7, C-8, C-3''
2''	CH_2	1.79, br t, 8.1	44.4	C-8, C-1'', C-3'', C-4'', C-5''
3''	C		70.8	
4''	CH_3	1.33, s	29.2	C-2'', C-3''
5''	CH_3	1.33, s	29.2	C-2'', C-3''
7-OCH_3	CH_3	3.86, s	62.2	C-7

[a]Recorded in 300 MHz.

[b]Recorded in 75 MHz.

3.2.1.14 Compound CP14
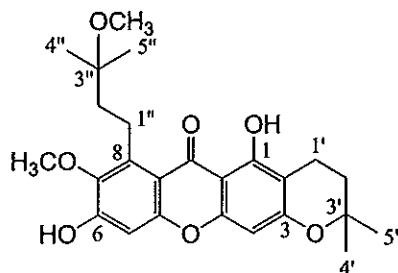
Compound **CP14** was isolated as yellow oil. The ^1H NMR spectrum of **CP14** (**Table 37**) were closely similar to those of **CP12**, except for the appearance of a 3-methoxyl-3-methylbutyl side chain at δ 3.39 (*br t*, J = 8.1 Hz, H-1″), 3.32 (*s*, OCH₃-3″), 1.75 (*br* t, J = 8.1 Hz, H-2″) and 1.30 (*s*, CH₃-4″ and CH₃-5″) instead of an isoprenyl side chain at C-8 as in **CP12**.

In HMBC spectral data (**Table 37**) of **CP13**, the methylene protons of a chromane ring at δ 2.71 showed correlations to the carbons at C-1 (δ 160.7), C-2 (δ 103.7), C-3 (δ 160.7), C-2′ (δ 31.9) and C-3′ (δ 76.0). It was suggested that a chromane ring was fused to the carbons at C-2 and C-3 in a linear fashion. The position of a 3-methoxyl-3-methylbutyl side chain at C-8 was assigned by using HMBC correlations (**Table 37**) of the methylene protons H-1″ at δ 3.39 to the carbons at C-7 (δ 142.4), C-8 (δ 138.7), C-8a (δ 111.9) and C-3″ (δ 74.9), of a methoxyl group at δ 3.86 to the carbon at C-7 (δ 142.4). From this assignment, it was also implied that a methoxyl group at δ 3.86 should be attached to the carbon at C-7. Moreover, the methoxyl group of a 3-methoxyl-3-methylbutyl side chain at δ 3.32 was confirmed by HMBC correlations (**Table 37**) of the methylene protons at δ 1.75 to the carbons at C-8 (δ 138.7), C-1″ (δ 22.1) and C-3″ (δ 74.9), while a methoxyl group at δ 3.32 showed correlations to the carbon at C-3″ (δ 74.9). From the above assignment, the presence of a 3-methoxyl-3-methylbutyl side chain was confirmed. The selected HMBC correlations of **CP14** were given in **Figure 65** for confirmation of this structure. Therefore, compound **CP14** was assigned as 3,4-dihydro-5,9-dihydroxy-8-methoxy-7-(3-methoxy-3-methyl-butyl)-2,2-dimethyl-2H,6H-pyrano[3,2b]xanthone (Dutta *et al.*, 1987).
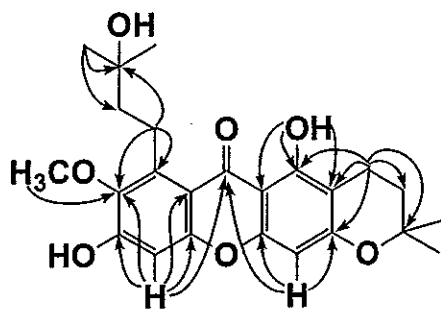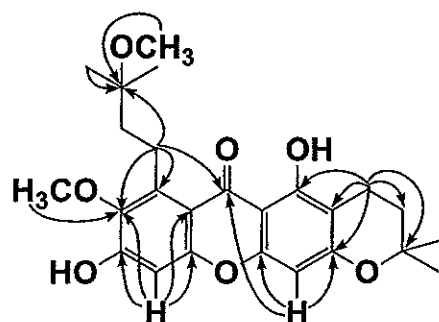
Figure 65. Selected HMBC correlations of **CP14**

Table 37 NMR spectroscopic data of **CP14** in CDCl₃

Position	Type of C	$\delta_H{}^a$ (*J* in Herz)	$\delta_C{}^b$	HMBC (^{1}H→^{13}C)
1-OH	C	13.90, *s*	160.7	-
2	C		103.7	
3	C		160.7	
4	CH	6.23, *s*	93.9	C-3, C-4a, C-9a
5	CH	6.81, *s*	101.5	C-6, C-7, C-4b, C-8a
6	C		156.0	
7	C		142.4	
8	C		138.7	
9	C=O		182.0	
4a	C		154.5	
4b	C		154.7	
8a	C		111.9	
9a	C		102.8	
1′	CH₂	2.71, *t*, 6.9	16.1	C-1, C-2, C-3, C-2′, C-3′
2′	CH₂	1.84, *t*, 6.9	31.9	C-2, C-1′, C-3′, C-4′, C-5′
3′	C		76.0	
4′	CH₃	1.37, *s*	26.8	C-3′
5′	CH₃	1.37, *s*	26.8	C-3′
1″	CH₂	3.39, *br t*, 8.1	22.1	C-7, C-8, C-8a, C-2″
2″	CH₂	1.75, *br t*, 8.1	39.8	C-8, C-1″, C-3″, C-4″, C-5″
3″	C		74.9	
4″	CH₃	1.30, *s*	25.2	C-3″
5″	CH₃	1.30, *s*	25.2	C-3″
3″-OCH₃	CH₃	3.32, *s*	49.2	C-3″
7-OCH₃	CH₃	3.86, *s*	62.1	C-7

^{a}Recorded in 300 MHz.

^{b}Recorded in 75 MHz.

3.2.1.15 Compound CP15
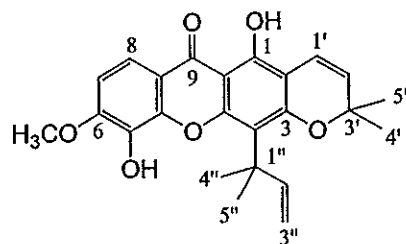
Compound **CP15** was isolated as yellow oil, mp 157-158 °C. The UV-Vis spectrum showed typical absorption bands of a xanthone at 242, 281, 290 and 334 nm (Gunasekera *et al.*, 1975). The FT-IR spectrum exhibited the hydroxyl group at 3520 cm^{-1} and conjugated carbonyl group at 1652 cm^{-1} (Gunasekera *et al.*, 1975).
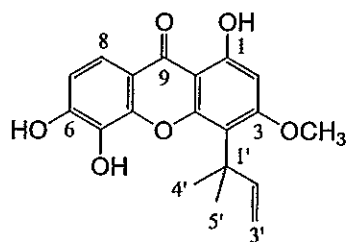
The ^{1}H NMR spectrum of **CP15** (**Table 38**) were closely similar to those of **CC10**, (**Table 11**) except for the appearance of a methoxyl group at δ 3.98 (*s*, 6-OCH$_3$) instead of a free hydroxyl group at C-6 as in **CC10**. The ^{1}H NMR spectrum of **CP15** (**Table 38**) showed a chelated hydroxyl proton at δ 13.52 (*s*), a pair of *ortho*-coupled aromatic protons at δ 7.74 (*d*, J = 8.7 Hz, H-8) and 6.97 (*d*, J = 8.7 Hz, H-7) and the typical signals of a chromene ring at δ 6.77 (*d*, J = 9.9 Hz, H-1'), 5.61 (*d*, J = 9.9 Hz, H-2') and 1.50 (*s*, CH$_3$-4' and CH$_3$-5'). Moreover, the ^{1}H NMR spectrum of **CP15** also showed the typical signal of a 1,1-dimethylallyl side chain at δ 6.66 (*dd*, J = 17.7, 10.8 Hz, H-2''), 5.18 (*br d*, J = 17.7 Hz, 1H$_2$-3''), 5.04 (*br d*, J = 10.8 Hz, 1H$_2$-3'') and 1.66 (*s*, CH$_3$-4'' and CH$_3$-5''). The NMR data of compounds **CP15** and **CC10** were given in **Table 38** for the structural comparison. Therefore, compound **CP15** was assigned as 10-*O*-methyl-macluraxanthone (Gunasekera *et al.*, 1975).

Table 38 NMR spectroscopic data of **CP15** in CDCl₃

Position	CP15		CC10		
	Type of C	$\delta_H{}^a$ (*J* in Herz)	**Type of C**	$\delta_H{}^a$ (*J* in Herz)	$\delta_C{}^b$
1-OH	C	13.52, *s*	C	13.53, *s*	156.8
2	C		C		105.6
3	C		C		158.9
4	C		C		113.1
5	C		C		131.1
6	C		C		149.0
7	CH	6.97, *d*, 8.7	CH	6.94, *d*, 9.0	112.8
8	CH	7.74, *d*, 8.7	CH	7.68, *d*, 9.0	117.5
9	C=O		C=O		180.8
4a	C		C		154.1
4b	C		C		144.5
8a	C		C		113.7
9a	C		C		103.8
1'	CH	6.77, *d*, 9.9	CH	6.76, *d*, 9.9	116.1
2'	CH	5.61, *d*, 9.9	CH	5.61, *d*, 9.9	127.2
3'	C		C		78.3
4'	CH₃	1.51, *s*	CH₃	1.52, *s*	27.9
5'	CH₃	1.51, *s*	CH₃	1.52, *s*	27.9
1''	C		C		41.4
2''	CH	6.66, *dd*, 17.7, 10.8	CH	6.76, *dd*, 17.7, 10.5	156.8
3''	CH₂	5.18, *br d*, 17.7 5.04, *br d*, 10.8	CH₂	5.22, *dd*, 17.7, 1.5 5.05, *dd*, 10.5, 1.5	103.3
4''	CH₃	1.66, *s*	CH₃	1.65, *s*	28.2
5''	CH₃	1.66, *s*	CH₃	1.65, *s*	28.2
6-OCH₃	CH₃	3.98, *s*	-	-	-

[a] Recorded in 300 MHz.

[b] Recorded in 75 MHz.

3.2.1.16 Compound CP16

Compound **CP16** was isolated as yellow powder. The UV-Vis spectrum showed typical absorption bands of a xanthone at 246, 275, 293, 323 and 399 nm (Kobayashi *et al.*, 1997). The FT-IR spectrum exhibited the hydroxyl group at 3370 cm^{-1} and conjugated carbonyl group at 1650 cm^{-1} (Kobayashi *et al.*, 1997).

The ^{1}H NMR spectrum of **CP16** (**Table 39**) were closely similar to those of **CP6**, (**Table 29**) except for the appearance of a free hydroxyl group instead of a methoxyl group at C-6 as in **CP6**. The ^{1}H NMR spectrum of **CP16** (**Table 39**) showed a chelated hydroxyl proton at δ 13.38 (*s*), a pair of *ortho*-coupled aromatic protons at δ 7.69 (*d*, J = 8.7 Hz, H-8) and 6.95 (*d*, J = 8.4 Hz, H-7), a methoxyl group at δ 3.90 (*s*, 3-OCH$_3$) and the typical signal of a 1,1-dimethylallyl side chain at δ 7.73 (*dd*, J = 17.4, 10.8 Hz, H-2''), 5.21 (*br d*, J = 17.4 Hz, 1H$_2$-3''), 5.04 (*br d*, J = 10.8 Hz, 1H$_2$-3'') and 1.58 (*s*, CH$_3$-4'' and CH$_3$-5''). Moreover, the position of a methoxyl group (δ 3.90) at C-3 was confirmed by NOESY cross-peak between a methoxyl group at δ_H 3.90 with an aromatic proton H-2 at δ_H 6.40 as shown in **Figure 66**. In addition, the NMR data of compounds **CP6** and **CP16** were also given in **Table 39** for the structural comparison. Therefore, compound **CP16** was assigned as isocudraniaxanthone B (Kobayashi *et al.*, 1997).
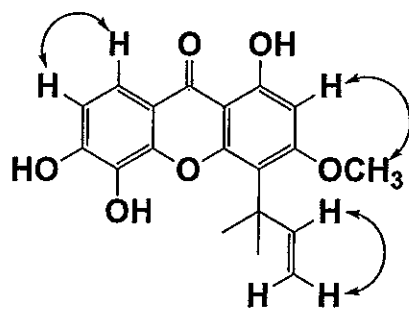

Figure 66. NOESY correlations of **CP16**

Table 39 NMR spectroscopic data of **CP16** in CDCl₃

Position	CP16			CP6	
	Type of C	$\delta_H{}^a$ (*J* in Herz)	NOESY	$\delta_H{}^a$ (*J* in Herz)	$\delta_C{}^b$
1-OH	C	13.38, *s*		12.82, *s*	162.5
2	CH	6.40, *s*	3-OCH₃	6.33,	95.6
3	C				165.4
4	C				113.6
5-OH	C			6.18, *br s*	133.6
6	C				151.6
7	CH	6.95, *d*, 8.4	H-8	6.90, *d*, 8.8	108.3
8	CH	7.69, *d*, 8.7	H-7	7.68, *d*, 9.2	116.9
9	C=O				181.1
4a	C				154.0
4b	C				144.6
8a	C				114.2
9a	C				103.1
1′	C				41.5
2′	CH	7.73, *dd*, 17.4, 10.8	H-3′	6.58, *dd*, 17.6, 10.4	155.1
3′	CH₂	5.21, *br d*, 17.4	H-2′	5.10, *d*, 17.6	104.5
		5.04, *br d*, 10.5		4.97, *d*, 10.4	
4′	CH₃	1.58, *s*		1.56, *s*	28.2
5′	CH₃	1.58, *s*		1.56, *s*	28.1
3-OCH₃	CH₃	3.90, *s*	H-2	3.82, *s*	
6-OCH₃	CH₃	-		3.96, *s*	62.8

*a*Recorded in 300 MHz.

*b*Recorded in 75 MHz.

3.2.1.17 Compound CP17
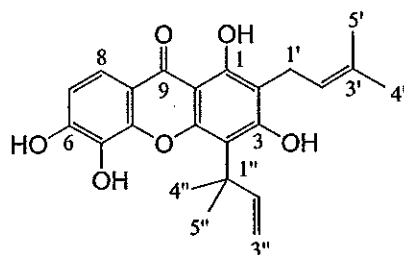
Compound **CP17** was isolated as yellow oil, mp 180-181 °C. The UV-Vis spectrum showed typical absorption bands of a xanthone at 203, 253, 287 and 328 nm (Chang *et al.*, 1989). The FT-IR spectrum exhibited the hydroxyl group at 3380 cm^{-1} and conjugated carbonyl group at 1621 cm^{-1} (Chang *et al.*, 1989).

The ^{1}H NMR spectrum of **CP17** (**Table 40**) were closely similar to those of **CC10**, (**Table 11**) except for the appearance of an isoprenyl side chain at C-2 instead of a chromene ring as in **CC10**. The ^{1}H NMR spectrum of **CP17** (**Table 40**) exhibited a chelated hydroxyl proton at δ 13.61 (*s*), a pair of *ortho*-coupled aromatic protons at δ 7.70 (*d*, J = 8.7 Hz, H-8) and 6.94 (*d*, J = 8.7 Hz, H-7), an isoprenyl side chain at δ 5.24 (*br t*, J = 6.9 Hz, H-2'), 3.47 (*d*, J = 6.9 Hz, H-1'), 1.86 (*br s*, CH3-5') and 1.79 (*s*, CH3-4'). Moreover, the presence of a 1,1-dimethylallyl side chain was suggested by the following ^{1}H NMR spectral data at δ 6.68 (*dd*, J = 17.7, 10.5 Hz, H-2''), 5.30 (*dd*, J = 17.7, 0.9 Hz, 1H2-3''), 5.15 (*dd*, J = 10.5, 0.9 Hz, 1H2-3'') and 1.69 (*s*, CH3-4'' and CH3-5''). In HMBC spectral data (**Table 40**) of **CP17**, a chelated hydroxyl group 1-OH at δ 13.61 showed correlations to the carbons at C-1 (δ 158.9), C-2 (δ 110.2) and C-9a (δ 103.0), while the methylene protons of an isoprenyl side chain at δ 3.47 also showed correlations to the carbons at C-1 (δ 158.9), C-2 (δ 110.2), C-3 (δ 161.4), C-2' (δ 121.2) and C-3' (δ 135.9). It was suggested that an isoprenyl side chain should be attached to the carbon at C-2. The location of a 1,1-dimethylallyl side chain at C-4 was assigned by using HMBC correlations (**Table 40**) of the methyl protons CH3-4'' at δ 1.69 to the carbons at C-4 (δ 111.1) and C-2'' (δ 154.6). The selected HMBC correlations of **CP17** were also given in **Figure 67** for confirmation of this structure. Therefore, compound **CP17** was assigned as gerontoxanthone I (Chang *et al.*, 1989).
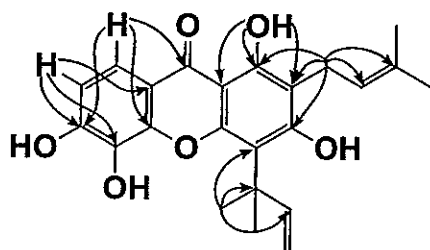
Figure 67. Selected HMBC correlations of **CP17**

Table 40 NMR spectroscopic data of **CP17** in CDCl₃

Position	Type of C	$\delta_H{}^a$ (J in Herz)	$\delta_C{}^b$	HMBC (^{1}H→^{13}C)
1-OH	C	13.61, s	158.9	C-1, C-2, C-9a
2	C		110.2	
3	C		161.4	
4	C		111.1	
5	C		131.0	
6	C		149.0	
7	CH	6.94, d, .87	112.6	C-5, C-6, C-8a
8	CH	7.70, d, 8.7	117.6	C-6, C-9, C-4b
9	C		180.8	
4a	C		153.3	
4b	C		144.8	
8a	C		113.8	
9a	C		103.0	
1′	CH₂	3.47, d, 6.9	21.6	C-1, C-2, C-3, C-2′, C-3′
2′	CH	5.24, br t, 6.9	121.2	C-1′, C-4′, C-5′
3′	C		135.9	
4′	CH₃	1.79, s	25.9	C-2′, C-3′
5′	CH₃	1.86, s	17.9	C-2′, C-3′
1″	C		41.6	
2″	CH	6.68, dd, 17.7, 10.5	154.6	C-1″, C-4″, C-5″
3″	CH₂	5.30, dd, 17.7, 0.9	106.6	C-1″, C-2″
		5.15, dd, 10.5, 0.9		-
4″	CH₃		28.0	C-4, C-1″, C-2″
5″	CH₃	1.69, s	28.0	C-4, C-1″, C-2″

^{a}Recorded in 300 MHz.

^{b}Recorded in 75 MHz.

3.2.1.18 Compound CP18
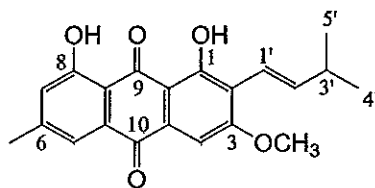
Compound **CP18** was isolated as red-orange powder, mp 201-203 °C. The UV-Vis spectrum showed the absorption bands at 220, 278 and 425 nm (Goncalves and Mors, 1981), characteristic of a conjugated quinine system, which was further confirmed by the presence of FT-IR absorption indicating the presence of hydroxyl (3425 cm^{-1}) and chelated carbonyl (1624 cm^{-1}) group (Goncalves and Mors, 1981).

The ^{1}H NMR spectral data of **CP18** (**Table 41**) showed two chelated hydroxyl groups at δ 12.84 (s) and 12.02 (s), which were assigned to the carbons at C-1 and C-8 from HMBC experiment (**Table 41**). Moreover, The ^{1}H NMR spectral data (**Table 41**) of **CP18** also exhibited a singlet aromatic proton at δ 7.40 (s, H-4), a *meta*-coupled aromatic protons at δ 7.61 (br s, H-5) and 7.07 (br s, H-7), a methoxyl group at δ 4.05 (s, 3-OCH$_3$), an aromatic methyl protons at δ_H 2.45 (s, CH$_3$-6) and a typical signal of a *trans*-3,3-dimethylprop-1-enyl group at δ 6.92 (dd, J = 16.2, 7.2 Hz, H-2'), 6.66 (dd, J = 16.2, 1.2 Hz, H-1'), 2.50 (m, H-3'), 1.14 (d, J = 6.9 Hz, CH$_3$-4' and CH$_3$-5'). The location of a *trans*-3,3-dimethylprop-1-enyl group was assigned to C-2 by HMBC correlations (**Table 41**) of chelated hydroxyl group at δ 12.84 to the carbons at C-1 (δ 162.5) C-2 (δ 120.2), C-9a (δ 110.5), and C-9 (δ 191.4), of an olefinic proton of *trans*-3,3-dimethylprop-1-enyl group H-1'at δ 6.66 to the carbons at C-1 (δ 162.5), C-2 (δ 120.0) and C-3 (δ 163.0). The attachment of a methoxyl group at C-3 was assigned by using HMBC correlations of an olefinic proton H-1' at δ 6.66 to the carbons at C-2 (δ 120.0), C-1 (C-2 (δ 162.5) and C-3 (δ 163.0), of a methoxyl group at δ 4.05 to the carbon at C-3 (δ 163.0). The selected HMBC correlations of **CP18** were also given in **Figure 68** for confirmation of this structure. Therefore, compound **CP18** was assigned as vismiaquinone A (Goncalves and Mors 1981).
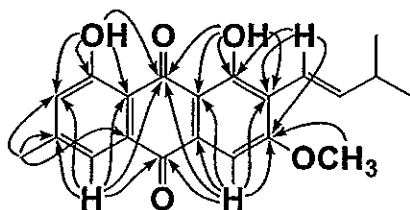
Figure 68. Selected HMBC correlations of **CP18**

Table 41 NMR spectroscopic data of **CP18** in $CDCl_3$

Position	Type of C	$\delta_H{}^a$ (*J* in Herz)	$\delta_C{}^b$	HMBC ($^1H \rightarrow {}^{13}C$)
1-OH	C	12.84, *s*	162.5	C-1, C-2, C-9, C-9a
2	C		120.0	
3	C		163.0	
4	CH	7.40, *s*	103.4	C-2, C-3, C-9, C-10, C-4a, C-9a
5	CH	7.61, *br s*	121.1	C-6, C-7, C-8, C-9, C-10, C-8a
6	C		148.4	
7	CH	7.07, *br s*	124.4	C-5, C-8, 6-CH₃
8-OH	C	12.02, *s*	162.1	C-7, C-8, C-9, 6-CH₃, C-8a
9	C=O		191.4	
10	C=O		181.9	
4a	C		132.1	
4b	C		133.2	
8a	C		113.7	
9a	C		110.5	
1'	CH	6.66, *dd*, 16.2, 1.2	115.8	C-1, C-2, C-3
2'	CH	6.92, *dd*, 16.2, 7.2	146.8	C-2
3'	CH	2.50, *m*	33.4	C-1', C-2', C-4', C-5'
4'	CH₃	1.14, *d*, 6.9	22.5	C-1', C-3'
5'	CH₃	1.14, *d*, 6.9	22.5	C-1', C-3'
3-OCH₃	CH₃	4.05, *s*	56.3	C-3
6-CH₃	CH₃	2.45, *s*	22.2	C-6, C-7, C-4b

aRecorded in 300 MHz.

bRecorded in 75 MHz.

3.2.1.19 Compound CP19
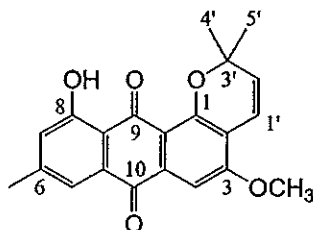
Compound **CP19** was isolated as orange solid, mp 224-226 °C. The UV-Vis spectrum showed the absorption bands at 208, 224, 265, 285 and 424 nm (Goncalves and Mors, 1981), characteristic of a conjugated quinine system, which was further confirmed by the presence of FT-IR absorption indicating the presence of hydroxyl (3446 cm⁻¹) and chelated carbonyl (1646 cm⁻¹) group (Goncalves and Mors, 1981).

The ^{1}H and ^{13}C NMR spectral data of **CP19** (**Table 42**) were similar to those of **CP18** (**Table 41**), except for the presence of the signal of the chromane ring at δ_H 6.73 (d, J = 10.2 Hz, H-1′), 5.84 (d, J = 10.2 Hz, H-2′) and 1.57 (s, CH₃-4′ and CH₃-5′) in **CP19** instead of chelated hydroxyl and *trans*-3,3-dimethylprop-1-enyl groups at δ_H 12.28 (s, 1-OH) and 6.92 (dd, J = 16.2, 6.9 Hz, H-2′), 6.66 (dd, J = 16.2, 1.2 Hz, H-1′), 2.50 (m, H-3′) and 1.14 (d, J = 6.9 Hz, CH₃-4′ and CH₃-5′). The position of a chromane ring on the xanthone nucleus of **CP19** was confirmed by HMBC correlations from an *olefinic* proton of a chromane ring H-1′ at δ_H 6.73 to the carbons at C-1 (δ 156.3), C-3 (δ 158.8) and C-3′ (δ 77.8). The selected HMBC correlations were shown in **Figure 69** for confirmation of this structure. Therefore, compound **CP19** was identified as 11-hydroxy-5-methoxy-2,2,9-trimethyl-2*H*-anthra-[1,2-b]pyran-7,12-dione (Delle Monache *et al.*, 1979).
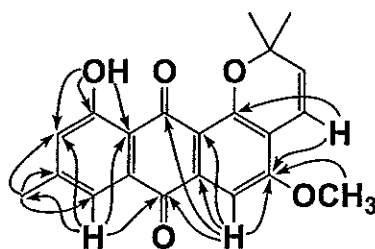
Figure 69. Selected HMBC correlations of CP19

Table 42 NMR spectroscopic data of **CP19** in $CDCl_3$

Position	Type of C	$\delta_H{}^a$ (J in Herz)	$\delta_C{}^b$	HMBC ($^1H \rightarrow {}^{13}C$)
1	C		156.3	
2	C		114.9	
3	C		158.8	
4	CH	7.43, s	102.8	C-3, C-9, C-10, C-4a, C-9a
5	CH	7.56, dd, 1.5, 0.6	119.8	C-7, C-10, C-8a, 6-CH₃
6	C		146.7	
7	CH	7.67, dd, 1.8, 0.9	124.5	C-5, C-8, C-8a, 6-CH₃
8-OH	C	13.18, s	162.6	C-7, C-8, C-8a, 6-CH₃
9	C=O		187.2	
10	C=O		182.7	
4a	C		135.4	
4b	C		132.6	
8a	C		115.4	
9a	C		116.3	
1′	CH	6.73, d, 10.2	116.1	C-1, C-3
2′	CH	5.84, d, 10.2	132.2	C-1′, C-3′, C-4′, C-5′
3′	C		77.8	
4′	CH₃	1.57, s	27.9	C-2′, C-3′
5′	CH₃	1.57, s	27.9	C-2′, C-3′
3-OCH₃	CH₃	4.03, s	56.2	C-3
6-CH₃	CH₃	2.42, s	22.0	C-5, C-6, C-7

^{a}Recorded in 300 MHz.

^{b}Recorded in 75 MHz.

3.2.1.20 Compound CP20
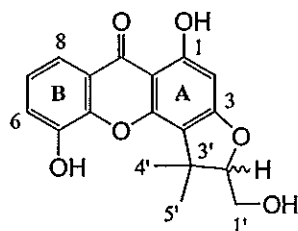
Compound **CP20** was isolated as yellow needle crystal, mp 235-237 °C, $[\alpha]^{25}_D$ = +64.6 (c 0.04, CHCl$_3$). The HREIMS of **CP20** showed a molecular ion peak at m/z 328.0947 [M]$^+$, suggesting the molecular formula $C_{18}H_{16}O_6$. The UV-Vis spectrum showed absorption bands of a xanthone at 246, 257, 315 and 357 nm (Boonnak et al., 2010). The FT-IR spectrum exhibited the hydroxyl group at 3368 cm^{-1} and conjugated carbonyl group at 1648 cm^{-1} (Boonnak et al., 2010).

The ^{1}H and ^{13}C NMR spectral data of **CP20** were summarized in **Table 43**. The ^{1}H NMR spectrum of **CP20** showed a chelated hydroxyl proton at δ 13.27 (s), an aromatic proton in ring A at δ 6.21 (s, H-2) and the characteristic peaks of 1,2,3-trisubstituted benzene ring in ring B at δ 7.68 (dd, J = 7.8, 1.8 Hz, H-8), 7.39 (dd, J = 7.8, 1.8 Hz, H-6) and 7.26 (t, J = 7.8 Hz, H-7). Moreover, the ^{1}H NMR spectrum of **1** also showed the characteristic signal of a 2'-hydroxymethyl-3',3'-dimethyldihydrofuran ring at δ 4.54 (t, J = 5.7 Hz, H-2'), 3.93 (d, J = 5.7 Hz, H$_2$-1'), 1.71 (s, CH$_3$-4') and 1.48 (s, CH$_3$-5'). The connection of the furan ring to ring A at C-3 and C-4 in an angular orientation was indicated from the following HMBC correlations (**Table 43**). The methyl protons CH$_3$-4' (δ 1.71) and CH$_3$-5' (δ 1.48) showed ^3J correlation with C-4 (δ 113.1), whereas H-2' at δ 4.54 showed correlations with C-3 (δ 166.4), C-4 (δ 113.1), C-1' (δ 60.4), C-3' (δ 43.1), C-4' (δ 26.2) and C-5' (δ 20.2). The selected HMBC correlations were shown in **Figure 70** for confirmation of this structure. Structure of **CP20** was further confirmed by the X-ray structure as illustrated in **Figure 71**. Therefore, compound **CP20** was a new compound and characterized as 2'-hydroxymethyl-3',3'-dimethyldihydrofuran-1,5-dihydroxy-xanthon, designated as pruniflorone M (Boonnak et al., 2010).
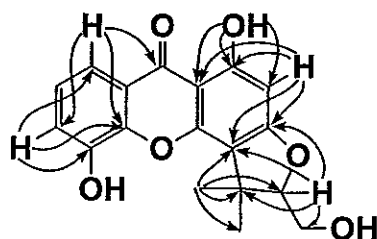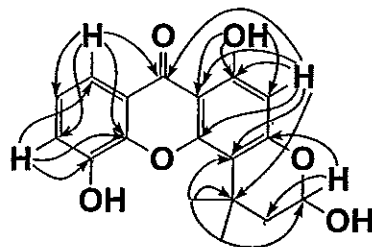
**Figure 70.** Selected HMBC correlations of CP20
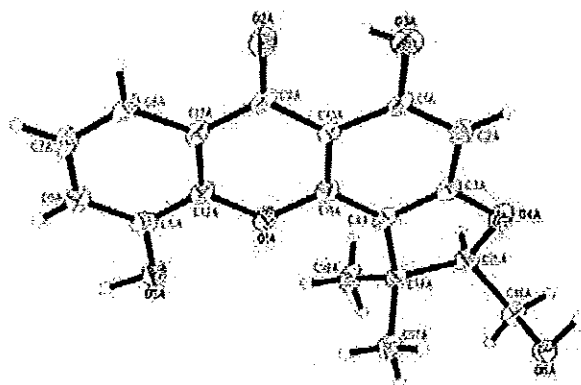
Figure 71. *ORTEP* plot of CP20

Table 43 NMR spectroscopic data of CP20 in d_6-acetone

Position	Type of C	$\delta_H{}^a$ (*J* in Herz)	$\delta_C{}^b$	HMBC (^{1}H→^{13}C)
1-OH	C	13.27, *s*	164.4	C-1, C-2, C-3, C-9a
2	CH	6.21, *s*	93.2	C-1, C-3, C-4, C-4a, C-9a
3	C		166.4	
4	C		113.1	
5	C		146.3	
6	CH	7.39, *dd*, 7.8, 1.8	120.4	C-5, C-8, C-4b
7	CH	7.26, *t*, 7.8	123.9	C-5, C-8, C-4b, C-8a
8	CH	7.68, *dd*, 7.8, 1.5	115.4	C-6, C-9, C-4b
9	C=O		180.7	
4a	C		152.6	
4b	C		145.2	
8a	C		121.5	
9a	C		103.4	
1'	CH$_2$	3.93, *d*, 5.7	60.4	C-2', C-3'
2'	CH	4.54, *t*, 5.7	94.8	C-3, C-4, C-1', C-3', C-4', C-5'
3'	C		43.1	
4'	CH$_3$	1.48, *s*	20.2	C-4, C-1', C-2', C-3', C-5'
5'	CH$_3$	1.71, *s*	26.3	C-4, C-2', C-3', C-4'

*a*Recorded in 300 MHz.

*b*Recorded in 75 MHz.

3.2.1.21 Compound CP21
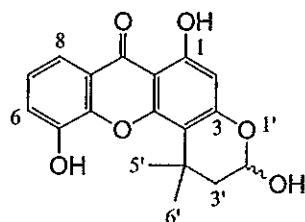
Compound **CP21** was isolated as yellow needle crystal, mp 250-252 °C, $[\alpha]^{25}_D = +5.2$ (c 0.42, acetone). The HREIMS of **CP21** showed a molecular ion peak at m/z 328.0948 $[M]^+$, suggesting the molecular formula $C_{18}H_{16}O_6$. The UV-Vis spectrum showed absorption bands of a xanthone at 246, 259, 316 and 356 nm. The FT-IR spectrum exhibited the hydroxyl group at 3411 cm^{-1} and conjugated carbonyl group at 1651 cm^{-1}.

The ^{1}H NMR spectral data of **CP21** (**Table 44**) showed similarity with those of **CP20**, except for the appearance of 2'-hydroxy-4',4'-dimethylpyran ring at δ 5.39 (dd, J = 7.8, 2.1 Hz, H-2'), 1.87 (dd, J = 13.8, 2.1 Hz, 1H$_2$-3'), 1.78 (dd, J = 13.8, 7.8 Hz, 1H$_2$-3'), 1.59 (s, 5'-CH$_3$) and 1.48 (s, 6'-CH$_3$) in **CP21** instead of 2'-hydroxymethyl-3',3'-dimethyldihydrofuran ring as in **CP20**. The pyran ring was connected to C-3 and C-4 in an angular orientation as indicated from the following HMBC correlations (**Table 44**). The signal of an oxymethine H-2' at δ 5.39 showed correlations with C-3 (δ 160.6), C-3' (δ 45.9) and C-4' (δ 31.9), whereas H$_2$-3' at δ 1.87 and 1.78, CH$_3$-5' (δ 1.59) and CH$_3$-6' (δ 1.48) showed correlations with C-4 (δ 109.6). The selected HMBC correlations were shown in **Figure 72** for confirmation of this structure. Finally, structure of **CP21** was further confirmed by the X-ray structure as illustrated in **Figure 73** (Boonnak et al., 2010). Therefore, compound **CP21** was a new compound and characterized as 2'-hydroxy-4',4'-dimethylpyran-1,5-dihydroxy-xanthone, designated as pruniflorone N (Boonnak et al., 2010).

Figure 72. Selected HMBC correlations of **CP21**
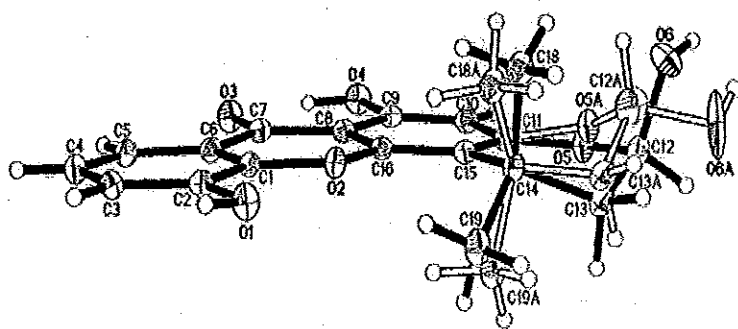
Figure 73. *ORTEP* plot of **CP21**

Table 44 NMR spectroscopic data of **CP21** in d_6-acetone

Position	Type of C	$\delta_H{}^a$ (*J* in Herz)	$\delta_C{}^b$	HMBC (^{1}H→^{13}C)
1-OH	C	12.82, *s*	161.1	C-1, C-2, C-3, C-9a
2	CH	6.03, *s*	99.0	C-1, C-4, C-9, C-4a, C-9a, C-4′
3	C		160.7	
4	C		109.6	
5	C		146.8	
6	CH	7.26, *br d*, 7.5	119.9	C-5, C-8, C-4b
7	CH	7.10, *t*, 7.8	123.9	C-5, C-6, C-8, C-4b, C-8a
8	CH	7.49, *d*, 8.1	114.8	C-5, C-6, C-7, C-9, C-4b
9	C=O		181.2	
4a	C		155.7	
4b	C		145.3	
8a	C		121.2	
9a	C		104.1	
1′	-	-	-	-
2′	CH	5.39, *dd*, 7.8, 2.1	93.1	C-3, C-3′, C-4′
3′	CH$_2$	1.87, *dd*, 13.8, 2.1	45.9	C-4, C-2′, C-4′, C-5′, C-6′
		1.78, *dd*, 13.8, 7.8		C-4, C-2′, C-4′
4′	C		31.9	
5′	CH$_3$	1.59, *s*	28.1	C-4, C-2′, C-4′, C-6′
6′	CH$_3$	1.48, *s*	28.0	C-4, C-4′, C-6′

^{a}Recorded in 300 MHz.

^{b}Recorded in 75 MHz.

3.2.1.22 Compound CP21a
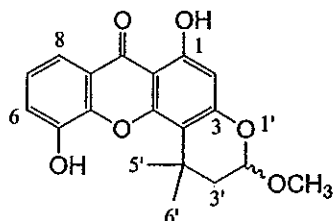
A solution of **CP21** (7.8 mg) in 20% HCl–CH₃OH (2.0 mL) was left to stand for 4 days at room temperature. The solution was evaporated in vacuum to give a residue, which was purified by CC on silica gel and eluted with 25% acetone-hexane to give compounds **CP21** (3.5 mg) and **CP21a** (3.5 mg).

Compound **CC21a** was isolated as yellow powder, m.p. 208-210 °C, $[\alpha]^{28}_{D} = +40.8$ (c 0.18, acetone). A molecular ion peak at m/z 342.1091 $[M]^{+}$ in the HREIMS established the molecular formula of $C_{19}H_{18}O_{6}$. The ¹H and ¹³C NMR data of **CP21a** (**Table 45**) were similar to those of **CP21** (**Table 44**), except for the presence of a methoxyl group at δ 3.41 (s, 2′-OCH₃) instead of a hydroxyl group at C-2′ as in **CP21**. The position of a methoxyl group at C-2′ was assigned by using HMBC correlation (**Table 45**) of a methylene protons H-3′ at δ 1.89 to the carbon at δ 110.0 (C-4), 99.6 (C-2′), 31.1 (C-4′), 28.4 (CH₃-6′) and 28.0 (CH₃-5′), of a methoxyl group at C-2′ at δ 3.41 to the carbon at δ 99.6 (C-2′). The selected HMBC correlations were also given in **Figure 74** for the structure confirmation. Therefore, compound **CP21a** was assigned as 2′-methoxypruniflorone N (Boonnak *et al.*, 2009).
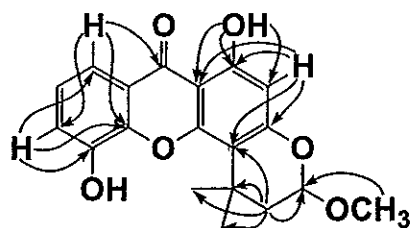
Figure 74. Selected HMBC correlations of **CP21a**

Table 45 NMR spectroscopic data of **CP21a** in d_6-acetone

Position	Type of C	$\delta_H{}^a$ (*J* in Herz)	$\delta_C{}^b$	HMBC (^{1}H→^{13}C)
1-OH	C	12.84, *s*	161.2	C-1, C-2, C-9a
2	CH	6.12, *s*	99.2	C-1, C-3, C-4, C-9a
3	C		159.6	
4	C		110.0	
5	C		146.5	
6	CH	7.28, *dd*, 7.8, 1.2	120.0	C-5, C-8, C-4b
7	CH	7.14, *t*, 7.8	124.0	C-5, C-8a
8	CH	7.55, *d*, 7.8, 1.5	115.1	C-6, C-9, C-4b
9	C=O		181.3	
4a	C		155.7	
4b	C		145.4	
8a	C		121.3	
9a	C		104.4	
1'	-	-	-	-
2'	CH	5.09, *dd*, 6.0, 3.0	99.6	C-3, C-3', C-4', 2'-OCH₃
3'	CH₂	1.89, *dd*, 13.8, 6.3	43.8	C-4, C-2', C-4', C-5', C-6'
		1.82, *dd*, 13.8, 2.7		C-4, C-2', C-4', C-5', C-6'
4'	C		31.1	
5'	CH₃	1.59, *s*	28.0	C-4, C-3', C-4', C-6'
6'	CH₃	1.51, *s*	28.4	C-4, C-3', C-4', C-5'
2'-OCH₃	CH₃	3.41, *s*	55.6	C-2'

^{a}Recorded in 300 MHz.

^{b}Recorded in 75 MHz.

3.2.1.23 Compound CP22
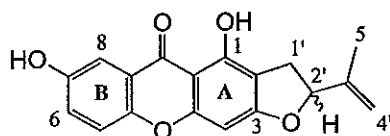
Compound **CP22** was isolated as yellow viscous oil, $[\alpha]^{26}_D = +15.1$ (c 0.04, acetone). The HREIMS of **CP22** showed a molecular ion peak at m/z 310.0845 $[M]^+$, suggesting the molecular formula $C_{18}H_{14}O_5$. The UV-Vis spectrum showed absorption bands of a xanthone at 243, 269, 280, 315 and 352 nm (Boonnak et $al.$, 2010). The FT-IR spectrum exhibited the hydroxyl group at 3378 cm^{-1} and conjugated carbonyl group at 1630 cm^{-1} (Boonnak et $al.$, 2010).

The ^{1}H NMR spectrum of **CP22** (**Table 46**) showed a chelated hydroxyl proton at δ 13.42 (s), an aromatic proton in ring A at δ 6.44 (s, H-4) and the characteristic signals of ABX trisubstituted benzene in ring B at δ 7.57 (d, J = 3.0 Hz, H-8), 7.42 (d, J = 8.7 Hz, H-5) and 7.33 (dd, J = 8.7, 3.0 Hz, H-6). Moreover, the ^{1}H NMR spectrum of **CP22** (**Table 46**) also showed the typical signal of a dihydrofuran ring with an isopropenyl side chain at δ 4.89 (brs, 1H$_2$-4′), 4.72 (brs, 1H$_2$-4′), 4.42 (m, H-2′), 3.05 (dd, J = 14.1, 2.4 Hz, H-1′), 2.92 (dd, J = 14.1, 7.5 Hz, H-1′) and 1.83 (brs, CH$_3$-5′). A dihydrofuran ring was connected to C-2 and C-3 of ring A in a linear fashion as indicated by HMBC correlations (**Table 46**). The nonequivalent methylene protons H$_2$-1′ (δ 3.05 and 2.92) showed correlations with C-1 (δ 160.9), C-2 (δ 108.3), C-3 (δ 165.6) and C-3′ (δ 147.6), whereas an oxymethine H-2′ (δ 4.42) showed correlations with C-2 (δ 108.3) and C-4′ (δ 109.4). The selected HMBC correlations were also given in **Figure 75** for the structure confirmation. Therefore, compound **CP22** was a new compound and characterized as 2′-isopropenyldihydrofuran-1,7-dihydroxyxanthone, designated as pruniflorone O (Boonnak et $al.$, 2010).
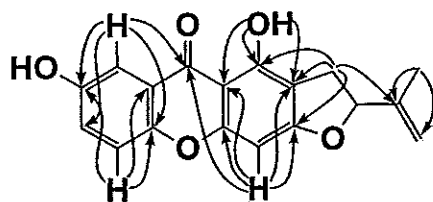
Figure 75. Selected HMBC correlations of **CP22**

Table 46 NMR spectroscopic data of **CP22** in d_6-acetone

Position	Type of C	$\delta_H{}^a$ (*J* in Herz)	$\delta_C{}^b$	HMBC (^{1}H$\rightarrow$^{13}C)
1-OH	C	13.42, *s*	160.9	C-1, C-2, C-9a
2	C		108.3	
3	C		165.6	
4	CH	6.44, *s*	94.1	C-2, C-3, C-9, C-4a, C-9a
5	CH	7.42, *d*, 8.7	118.7	C-7, C-4b, C-8a
6	CH	7.33, *dd*, 8.7, 3.0	124.1	C-7, C-4b
7	C		154.1	
8	CH	7.57, *d*, 3.0	108.4	C-6, C-7, C-9, C-4b
9	C=O		180.2	
4a	C		156.4	
4b	C		149.7	
8a	C		121.0	
9a	C		103.0	
1′	CH$_2$	3.05, *dd*, 14.1, 2.4	29.0	C-1, C-2, C-3, C-3′
		2.92, *dd*, 14.1, 7.5		C-1, C-2, C-3, C-3′
2′	CH	4.42, *m*	75.3	C-2, C-4′
3′	C		147.6	
4′	CH$_2$	4.89, *br s*	109.4	C-3′, C-5′
		4.72, *br s*		C-3′, C-5′
5′	CH$_3$	1.83, *s*	17.3	C-3′, C-4′

^{a}Recorded in 300 MHz.

^{b}Recorded in 75 MHz.

3.2.1.24 Compound CP23
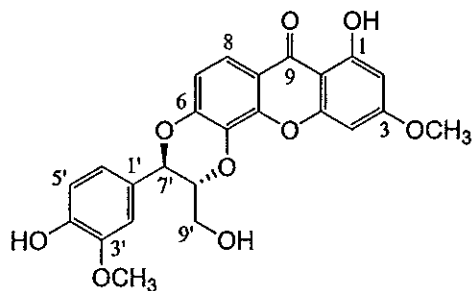
Compound **CP23** was isolated as yellow powder, $[\alpha]^{26}_D = +53.4$ (c 0.06, acetone). The HREIMS of **CP23** showed a molecular ion peak at m/z 452.1119 $[M]^+$, suggesting the molecular formula $C_{24}H_{20}O_9$. The UV-Vis spectrum showed absorption bands of a xanthone at 253, 281, and 318 nm. The FT-IR spectrum exhibited the hydroxyl group at 3431 cm^{-1} and conjugated carbonyl group at 1646 cm^{-1}.

The UV spectrum of **CP23** showed similar absorption bands (253, 281 and 318 nm) to those of 5'-demethoxycadensin G, while the IR spectrum exhibited the hydroxyl and conjugated carbonyl functionalities at ν_{max} 3431 and 1646 cm^{-1} respectively. The ^{1}H and ^{13}C NMR spectral data of compound **CP23** (**Table 47**) were closely related to those of 5'-demethoxycadensin G previously isolated from *C. cochinchinense*. The difference was shown as an additional methoxyl group (δ_H 3.83; δ_C 56.0) in compound **CP23** which was assigned at C-3 due to its HMBC correlation (**Table 47**) to δ 166.7 (C-3). The selected HMBC correlations were also given in **Figure 76** for the structure confirmation. Therefore, compound **CP23** was a new compound and characterized as 3-methoxy-5'-demethoxy-cadensin G (Boonnak *et al.*, 2010)
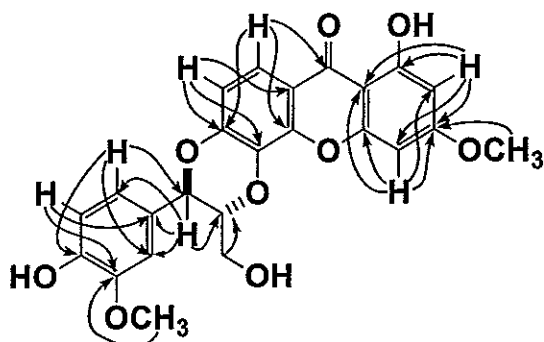
Figure 76. Selected HMBC correlations of **CP23**

Table 47 NMR spectroscopic data of **CP23** in d_6-acetone

Position	Type of C	$\delta_H{}^a$ (J in Herz)	$\delta_C{}^b$	HMBC (^{1}H→^{13}C)
1-OH	C	-	163.3	-
2	CH	6.28, d, 2.4	97.5	C-1, C-3, C-4, C-9a
3	C		166.7	
4	CH	6.46, d, 2.4	93.2	C-2, C-3, C-4a, C-9a
5	C		131.9	
6	C		146.5	
7	CH	6.92, d, 8.8	114.2	C-5, C-6, C-8a
8	CH	7.67, d, 8.8	117.8	C-6, C-8, C-9, C-4b
9	C=O		180.6	
4a	C		157.9	
4b	C		149.5	
8a	C		115.3	
9a	C		103.6	
1'	C		127.1	
2'	CH	6.89, d, 1.6	110.5	C-4', C-6', C-7'
3'	C		147.9	
4'	C		147.2	
5'	CH	6.86, d, 8.8	115.5	C-1', C-3'
6'	CH	6.89, dd, 8.8, 1.6	120.9	C-2', C-4', C-7'
7'	CH	5.06, d, 8.0	77.2	C-1', C-2', C-6', C-8'
8'	CH	4.07, ddd, 8.0, 3.6, 2.8	79.0	-
9'	CH$_2$	3.89, m	61.0	C-8'
		3.54, dd, 12.8, 3.6		-
3-OCH$_3$	CH$_3$	3.83, s	56.0	C-3
3'-OCH$_3$	CH	3.85, s	56.2	C-3'

^{a}Recorded in 400 MHz.

^{b}Recorded in 100 MHz.

3.2.1.25 Compound CP24
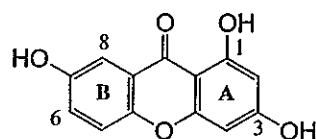
Compound **CP24** was isolated as yellow powder, mp 318-319 °C. The ^{1}H NMR spectrum of **CP24** (**Table 48**) showed a chelated hydroxyl proton at δ 13.92 (*s*), a pair of *meta*-coupled aromatic protons in ring A at δ 6.37 (*d*, J = 2.1 Hz, H-4) and 6.25 (*d*, J = 2.1 Hz, H-2) and a characteristic signals of ABX trisubstituted benzene in ring B at δ 7.51 (*d, J* = 2.7 Hz, H-8), 7.33 (*d, J* = 9.0 Hz, H-5) and 7.25 (*dd, J* = 9.0, 2.7 Hz, H-6). In addition, the NMR data of compounds **CP24** and 1,3,7-trihydroxyxanthone were also given in **Table 48** for the structural comparison. Therefore, compound **CP24** was assigned as 1,3,7-tri-hydroxyxanthone (Noro *et al.*, 1984; Mondal *et al.*, 2006).

Table 48 ^{1}H NMR spectroscopic data of CP24 in CD$_3$OD+CDCl$_3$

Position	CP24		1,3,7-trihydroxyxanthone[b]	
	Type of C	$\delta_a{}^a$ (*J* in Herz)	$\delta_C{}^c$	$\delta_H{}^d$ (*J* in Herz)
1-OH	C	12.92, *s*	162.7	12.88, *s*
2	CH	6.25, *d*, 2.1	98.0	6.18, *d*, 2.1
3	C		163.0	
4	CH	6.37, *d*, 2.1	93.9	6.35, *d*, 1.9
5	CH	7.33, *d*, 9.0	119.1	7.45, *d*, 9.0
6	CH	7.25, *dd*, 9.0, 2.7	124.6	7.27, *dd*, 9.0, 2.9
7	C		154.1	
8	CH	7.51, *d*, 2.7	108.2	7.40, *d*, 2.7
9	C=O		179.9	
4a	C		157.7	
4b	C		149.2	
8a	C		120.6	
9a	C		102.1	

^{a} Recorded at 300 MHz in CDCl$_3$+CD$_3$OD

^{b} It was previously reported by Mondal *et al.*, 2006.

^{c} Recorded at 200 MHz in DMSO

^{d} Recorded at 50 MHz in DMSO

3.2.1.26 Compound CP25
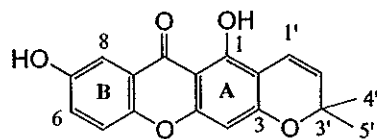
Compound CP25 was isolated as yellow solid, mp 266-268 °C, which was further recrystallized from CHCl$_3$ to yield yellow needle single crystals. The structure of CP25 was confirmed by single-crystal X-ray diffraction analysis. It revealed that a structure of CP25 was a xanthone containing a chromene unit in a linear fashion (Figure 77) Therefore, compound CP25 was assigned as osajaxanthone (Mondal et al., 2006).
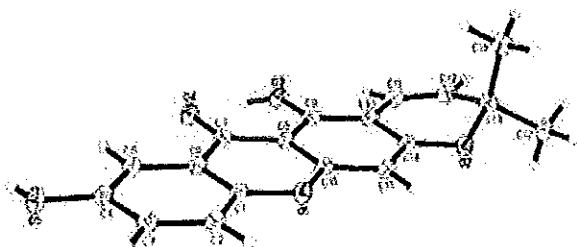
Figure 77. ORTEP plot of CP25

3.2.1.27 Compound CP26
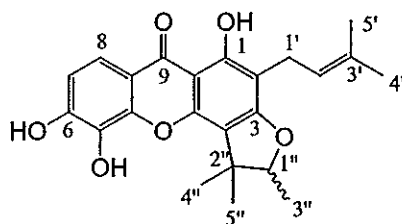
Compound **CP26** was isolated as yellow solid, mp 152-154 °C, $[\alpha]^{25}_D = -44.8$ (c 0.05, CHCl$_3$). The UV-Vis spectrum showed absorption bands of a xanthone at 258, 276 and 392 nm (Boonsri *et al.*, 2006). The FT-IR spectrum exhibited the hydroxyl group at 3440 cm^{-1} and conjugated carbonyl group at 1646 cm^{-1} (Boonsri *et al.*, 2006).

The ^{1}H NMR spectrum of **CP26** (**Table 49**) were closely similar to those of **CP17**, (**Table 40**) except for the appearance of a α,α,β-trimethylfuran ring at C-3/C-4 in **CP26** instead of a free hydroxyl group and 1,1-dimethylallyl side chain as in **CP17**. The ^{1}H NMR spectrum of **CP26** (**Table 49**) exhibited a chelated hydroxyl proton at δ 13.40 (s), a pair of *ortho*-coupled aromatic protons at δ 7.63 (d, J = 8.1 Hz, H-8) and 6.85 (br s, H-7), an isoprenyl side chain at δ 5.28 (br t, J = 7.2 Hz, H-2′), 3.29 (d, J = 7.2 Hz, H-1′), 1.77 (s, CH$_3$-5′) and 1.68 (s, CH$_3$-4′). Moreover, the presence of a α,α,β-trimethylfuran ring was suggested by the following ^{1}H NMR spectral data at δ 4.52 (q, J = 6.6 Hz, H-1″), 1.59 (s, CH$_3$-5″), 1.41 (d, J = 6.6 Hz, CH$_3$-3″) and 1.31 (s, CH$_3$-4″). In HMBC spectral data (**Table 49**) of **CP26**, a chelated hydroxyl group 1-OH at δ 13.40 showed correlations to the carbons at C-1 (δ 164.5), C-2 (δ 110.6) and C-9a (δ 106.8), while the methylene protons of an isoprenyl side chain at δ 3.29 also showed correlations to the carbons at C-1 (δ 164.5), C-2 (δ 110.6), C-3 (δ 168.0), C-2′ (δ 125.6), C-3′ (δ 135.9) and C-5′ (δ 21.6). It suggested that an isoprenyl side chain should be attached to the carbon at C-2. The location of a α,α,β-trimethylfuran ring at C-3/ C-4 was assigned by using HMBC correlations (**Table 49**) of the methyl protons CH$_3$-4″ at δ 1.31 to the carbons at C-4 (δ 115.9), C-1″ (δ 44.4) and C-2″ (δ 47.9). The selected HMBC correlations of **CP26** were also given in **Figure 78** for confirmation of this structure. Finally, the structure of **CP26** was further supported by the X-ray structure as illustrated in **Figure 79**. Therefore, compound **CP26** was assigned as formoxanthone C (Boonsri *et al.*, 2006).
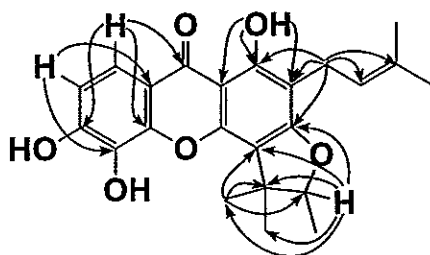
Figure 78. Selected HMBC correlations of **CP26**
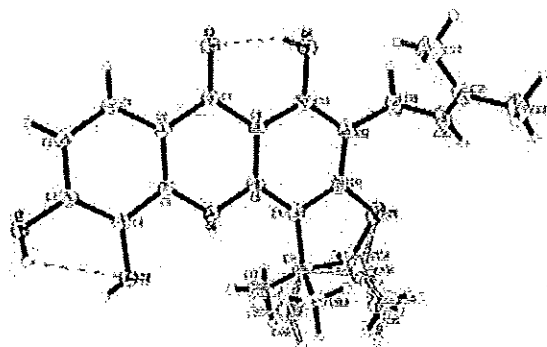
Figure 79. *ORTEP* plot of **CP26**

Table 49 NMR spectroscopic data of **CP26** in $CD_3OD+CDCl_3$

Position	Type of C	$\delta_H{}^a$ (*J* in Herz)	$\delta_C{}^b$	HMBC ($^1H\rightarrow{}^{13}C$)
1-OH	C	13.40, *s*	164.5	C-1, C-2, C-9a
2	C		110.6	
3	C		168.0	
4	C		115.9	
5	C		135.9	
6	C		149.7	
7	CH	6.85, *br s*	115.9	C-5, C-8a
8	CH	7.63, *d*, 8.1	121.1	C-6, C-9, C-4b
9	C		184.5	
4a	C		154.9	
4b	C		154.1	
8a	C		118.2	
9a	C		106.8	
1′	CH_2	3.29, *d*, 7.2	25.6	C-1, C-2, C-3, C-2′, C-3′, C-5′
2′	CH	5.28, *br t*, 7.2	125.6	C-2, C-1′, C-4′, C-5′
3′	C		135.9	
4′	CH_3	1.68, *s*	29.6	C-2′, C-3′
5′	CH_3	1.77, *s*	21.6	C-2′, C-3′
1″	CH	4.52, *q*, 6.6	44.4	C-3, C-4, C-2″, C-4″, C-5″
2″	C		47.9	
3″	CH_3	1.41, *d*, 6.6	18.2	C-1″, C-2″
4″	CH_3	1.31, *s*	25.1	C-4, C-1″, C-2″
5″	CH_3	1.59, *s*	29.6	C-4, C-1″, C-2″

^{a}Recorded in 300 MHz.; ^{b}Recorded in 75 MHz.

3.2.1.28 Compound CP27
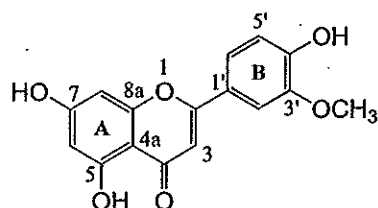
Compound **CP27** was isolated as a pale-yellow solid. The ^{1}H NMR spectrum of **CP27** showed typical signals of a flavone type. The ^{1}H NMR spectrum of **CP27** (**Table 50**) exhibited a chelated hydroxyl proton at δ 13.08 (s, 5-OH), a pair of *meta*-coupled aromatic protons in ring A at δ 6.56 (d, J = 1.8 Hz, H-8) and 6.26 (d, J = 1.8 Hz, H-6), an olefinic proton at δ 6.71 (s, H-3) and a methoxyl group at δ 4.00 (s, OCH$_3$-3'). Moreover, the presence of ABX trisubstituted benzene of ring B was suggested by the following ^{1}H NMR spectral data at δ 7.64 (brs, H-2'), 7.02 (d, J = 8.4, 2.1 Hz, H-5') and 7.62 (dd, J = 8.4, 2.1 Hz, H-6'). The structure of **CP27** was assigned by HMBC correlations, which was shown in **Figure 80**. Therefore, compound **CP27** was assigned as chrysoeriol (Wagner *et al.*, 1976; Nakasuki *et al.*, 2000).
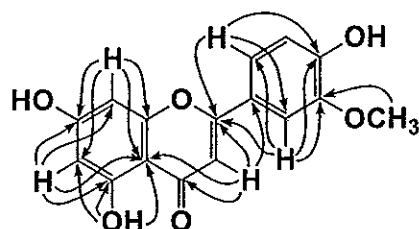
Figure 80. Selected HMBC correlations of **CP27**

Table 50 NMR spectroscopic data of **CP27** in d_6-acetone

Position	Type of C	$\delta_H{}^a$ (*J* in Herz)	$\delta_C{}^b$	HMBC (^{1}H→^{13}C)
1	-		-	
2	C		164.2	
3	CH	6.71, *s*	103.5	C-2, C-4, C-4a, C-1'
4	C=O		182.2	
5-OH	C	13.08, *s*	162.4	C-5, C-6, C-7, C-4a
6	CH	6.26, *d*, 1.8	98.9	
7	C		164.2	C-5, C-7, C-8, C-4b
8	CH	6.56, *d*, 1.8	93.9	C-6, C-7, C-4b, C-8a
4a	C		104.4	
8a	C		157.9	
1'	C		122.7	
2'	CH	7.64, *brs*	109.7	C-2, C-3', C-4', C-6'
3'	C		148.1	
4'	C		150.7	
5'	CH	7.02, *d*, 8.4	115.5	C-1', C-3', C-4'
6'	CH	7.62, *dd*, 8.4, 2.1	120.5	C-2, C-2', C-4'
3'-OCH₃	CH₃	4.00, *s*	55.7	C-3'

^{a}Recorded in 300 MHz.

^{b}Recorded in 75 MHz.

3.2.1.29 Compounds CP28, CP29 and CP30

CP28

CP29 : R₁ = CH₃ R₂ = H

CP30 : R₁ = H R₂ = CH₃
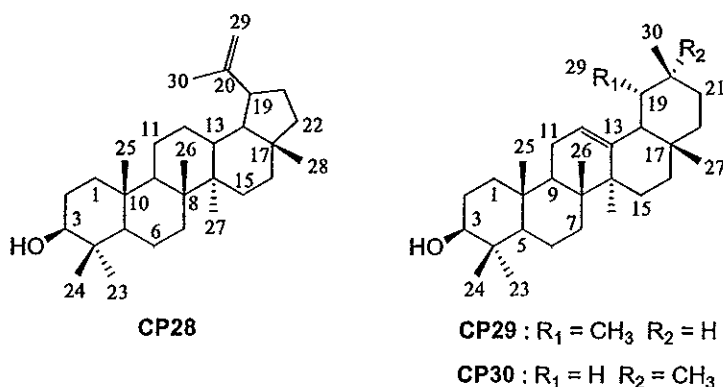
A mixture of compounds **CP28**, **CP29** and **CP30** was isolated as white solid and a major component from the green fruits of *C. formosum* ssp. *pruniflorum*. It gave a purple vanillin-sulfuric acid test indicating a triterpene. By comparing the spectrum of this mixture **(CP28-CP30)** to those previous report (Oliveira *et al.*, 2002), it suggested that this mixture **(CP28, CP29** and **CP30)** should be a mixture of lupeol, α-amyrin and β-amyrin. We found some useful information from the Japanese research group (Shibuya *et al.*, 2007), which explained the origin of some triterpeniods mixture that lupeol, α-amyrin and β-amyrin could be derived from the same precursor. It is possible to find some triterpene mixture in the plants. The characterization of a three mixtures of triterpenes was simplified by the assignment of the carbon atoms in the ^{13}C NMR spectrum. As the chemical shift of a sp^2 carbon atom is very characteristic for each triterpenoid skeleton, ^{13}C NMR spectroscopy has been very frequently employed for the structural analysis of triterpene mixtures. Thus, it appeared in the ^{13}C NMR spectrum signals at δ_C 79.0 ppm relative to C-3 position of the three constituents; δ_C 150.9 and 109.3 ppm corresponding to olefinic carbons at C-20 and C-29 of the lupane skeleton; δ_C 121.8 and 145.1 ppm corresponding to olefinic carbons at C-12 and C-13 of the oleanane skeleton and δ_C 124.3 and 139.3 ppm corresponding to olefinic carbons C-12 and C-13 of the ursane skeleton (Berrondo *et al.*, 2003). This method can be used for rapid characterization of this mixture. From previous investigation of our research group in 2005 (Laphookhieo, S. 2005), Laphookhieo was successful in separating the pure compound from these mixtures by performing some structural modification (Acetylation) and followed by HPLC chromatographic separation technique to obtain lupeol, α-amyrin and β-amyrin as acetate derivatives. From the above result, it pointed out that our mixtures **(CP28-CP30)** was a three-compound mixture of lupeol **(CP28)**, α-amyrin **(CP29)** and β-amyrin **(CP30)**.

3.2.1.30 Compounds CP31
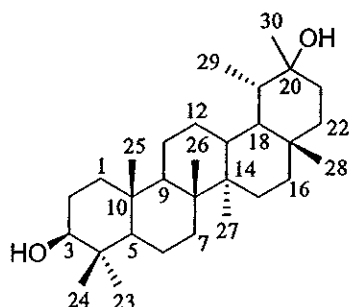
Compound **CP31** was obtained as a white powder, mp 152-154 °C. It gave a purple vanillin-sulfuric acid test indicating a triterpene.

The ^{1}H and ^{13}C NMR spectra (**Table 51**) of **CP31** showed characteristic of taraxastane type triterpenoids as six methyl singlets at δ_H 0.90 (δ_C 28.0, CH$_3$-23), 0.69 (δ_C 15.4, CH$_3$-24), 0.77 (δ_C 16.3, CH$_3$-25), 0.98 (δ_C 16.2, CH$_3$-26), 0.88 (δ_C 14.8, CH$_3$-27) and 0.76 (δ_C 28.0, CH$_3$-28) and one methyl doublet at δ_H 0.90 (δ_C 17.9; d, J = 6.0 Hz, CH$_3$-29) (Hinge *et al.*, 1966). The low field shift of one methyl signal at δ_H 1.11 (δ_C 28.0, CH$_3$-30) suggested that it was due to a methyl on a carbon bearing a tertiary hydroxyl (Anjaneyulu *et al.*, 1985). The ^{1}H NMR spectrum of **CP31** also showed the typical signals of an oxymethine proton at δ3.13 (δ_C 79.1; d, J = 11.5, 5.0 Hz, H-3). The structure of **CP31** was successfully assigned by HMBC correlations as shown in **Figure 81**. Therefore, compound **CP31** was assigned as taraxastane-3β,20-diol (Hinge *et al.*, 1966; Anjaneyulu *et al.*, 1985; Akihisa *et al.*, 2004).
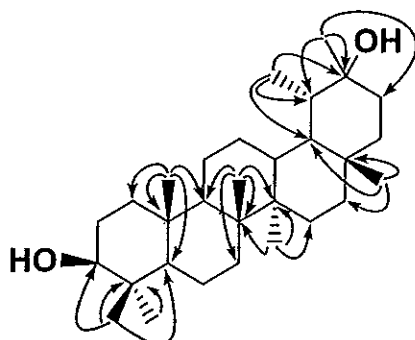
Figure 81. Selected HMBC correlations of **CP31**

Table 51 NMR spectroscopic data of **CP31** in $CDCl_3$

Position	Type of C	$\delta_H{}^a$ (*J* in Herz)	$\delta_C{}^b$	HMBC ($^1H \rightarrow {}^{13}C$)
1	CH_2	1.62, *m* ; 0.85, *m*	38.7	-
2	CH_2	2.73, *m* ; 1.51, *m*	27.4	-
3	CH	3.13, *dd*, 11.5, 5.0	79.1	C-1, C-23, C-24
4	C		37.8	
5	CH	0.62, *m*	55.1	-
6	CH_2	1.50, *m* ; 1.30, *m*	18.3	-
7	CH_2	1.35, *m* ; 1.39, *m*	34.5	-
8	C		41.4	
9	CH	1.25, *m*	48.6	-
10	C		36.9	
11	CH_2	1.44, *m* ; 1.26, *m*	21.6	-
12	CH_2	1.71, *m* ; 1.19, *m*	29.3	-
13	CH	1.71, *m*	38.9	-
14	C		43.2	
15	CH_2	1.69, *m* ; 0.90, *m*	26.6	-
16	CH_2	1.32, *m* ; 1.17, *m*	38.2	-
17	C		35.1	
18	CH	1.30, *m*	47.5	-
19	CH	1.51, *m*	38.8	-
20	C		73.6	
21	CH_2	1.70, *m* ; 1.47, *m*	35.5	-
22	CH_2	1.28, *m* ; 1.28, *m*	37.8	-
23	CH_3	0.90, *s*	28.0	C-4
24	CH_3	0.69, *s*	15.4	C-3, C-4, C-5
25	CH_3	0.77, *s*	16.3	C-1, C-5, C-9, C-10
26	CH_3	0.98, *s*	16.2	C-7, C-8, C-9, C-14
27	CH_3	0.88, *s*	14.8	C-8, C-14, C-15
28	CH_3	0.76, *s*	17.8	C-16, C-17, C-18
29	CH_3	0.90, *d*, 6.0	17.9	C-18, C-19, C-20
30	CH_3	1.11, *s*	30.3	C-19, C-20, C-21

[a]Recorded in 500 MHz.

[b]Recorded in 125 MHz.

3.2.2 Biological activities of compounds CP1-CP31

Only stable compounds of sufficient quantity were evaluated for their antibacterial activities against both Gram-positive bacteria: *Bacillus subtilis*, *Staphylococcus aureus*, *Enterococcus faecalis* TISTR 459, Methicillin-Resistant *Staphylococcus aureus* (MRSA) ATCC 43300, Vancomycin-Resistant *Enterococcus faecalis* (VRE) ATCC 51299 and Gram-negative bacteria: *Salmonella typhi*, *Shigella sonnei* and *Pseudomonas aeruginosa*. All compounds were also submitted to antifungal assay against *Candida albicans*.

Table 52 Antimicrobial activity of compounds CP1-CP3, CP5-CP14, CP17, CP18, CP21, CP21a, CP23, CP26 and CP27

No	Antibacterial activity								Antifungal activity
	Gram-positive bacteria[a]					Gram-positive bacteria[b]			C. albicans[c]
	BS	SA	EF	MRSA	VRE	ST	SS	PA	
CP1	300	>300	>300	>300	>300	>300	>300	>300	>300
CP2	>300	>300	>300	>300	>300	>300	>300	>300	>300
CP3	18.7	37.5	-	NT[d]	NT[d]	-	-	-	NT[d]
CP5	37.5	37.5	300	75	300	>300	>300	300	300
CP6	300	300	300	300	300	>300	>300	>300	>300
CP7	18.7	18.7	300	9.37	9.37	18.7	>300	300	>300
CP8	4.67	9.37	9.37	18.7	75	>300	>300	300	300
CP9	18.7	18.7	75	37.5	75	>300	>300	300	300
CP10	75	75	75	150	75	>300	>300	>300	>300
CP10: CC10[e]	4.67	2.34	9.37	2.34	4.67	>300	>300	>300	300
CP11	300	300	>300	300	300	>300	>300	>300	>300
CP12	-	-	-	NT[d]	NT[d]	-	-	-	NT[d]
CP13	9.37	<1.1	-	NT[d]	NT[d]	-	-	-	NT[d]
CP14	-	-	-	NT[d]	NT[d]	-	-	-	NT[d]
CP17	<1.1	<1.1	4.67	NT[d]	NT[d]	37.5	<1.1	<1.1	NT[d]
CP18	-	-	-	NT[d]	NT[d]	-	-	-	NT[d]
CP20	>300	>300	>300	>300	>300	>300	>300	>300	>300
CP21	300	>300	>300	9.37	300	150	>300	>300	>300
CP21a	18.7	300	300	37.5	75	300	300	37.5	300
CP23	>300	>300	>300	>300	>300	>300	>300	>300	>300
CP26	4.67	9.37	4.67	4.67	4.67	4.67	>300	37.5	>300
CP27	>300	>300	>300	37.5	37.5	300	>300	>300	>300

[a] *Bacillus subtilis*, *Staphylococcus aureus*, *Enterococcus faecalis* TISTR 459, Methicillin-Resistant *Staphylococcus aureus* (MRSA) ATCC 43300, Vancomycin-Resistant *Enterococcus faecalis* (VRE) ATCC 51299.; [b] *Salmonella typhi*, *Shigella sonnei* and *Pseudomonas aeruginosa*.; [c] *Candida albicans*; [d] NT = not tested; [e] a mixture in 1:1 ratio

From the antibacterial results in the 3.1.2 section (**Table 23**), it showed that 1,3,7-trihydroxyxanthone with the isoprenyl or geranyl side chain at C-2 and C-4 is essential for its antibacterial activity against *P. aeruginosa*, we can further added that the hydroxyl group at C-3 is also essential for the activity as compared pruniflorone L (**CP2**, MIC > 300 µg/mL) with cochinchinone A (**CC3**, MIC 4.7 µg/mL) (**Table 52**). During the chromatographic separation, we have observed that compounds **CP10** and **CC10** came as a mixture. Therefore, these two compounds were tested as a mixture for anti-microbial activity whose result was good. This prompted us to purify the mixture of which was successfully separated by reversed phase RP-18 CC eluting with MeOH. We then tested each of the compounds **CP10**, **CC10** and their 1:1 mixture. Interestingly, the 1:1 mixture of compounds **CP10** and **CC10** significantly increased the antibacterial activities against *B. subtilis*, *S. aureus*, *E. faecalis*, MRSA and VRE compared with the pure forms (**CP10** and **CC10**) (**Table 52**). From this result, it may be possible that a 1:1 mixture of compounds **CP10** and **CC10** showed synergistic effect for antibacterial activity against all Gram-positive bacteria tested.

Moreover, compounds **CP17** and **CP26** exhibited strong antibacterial activity against both Gram-positive and Gram-negative (*S. typhi*) bacteria with MIC value of <1.1 and 4.67 µg/mL, whereas compound **CP21** showed moderate antibacterial activity specifically against MRSA with MIC value of 9.37 µg/mL (**Table 52**). The methylated product **CP21a** was not significant for antibacterial activity but showed better activity against *B. subtilis* when compared with compound **CP21**.

Only isolated compounds from the green fruits of *C. formosum* spp. *pruniflorum* with sufficient amount were further evaluated for their nitric oxide inhibitory activity using RAW264.7 cells (Boonnak *et al.*, 2010). As shown in **Table 53**, compounds **CP21** and **CP26** possessed potent NO inhibitory activity against lipopolysaccharide (LPS)-induced nitric oxide release with IC_{50} values of 4.4 and 4.3 µM, respectively better than that of the positive control, indomethacin, which is a non-steroidal anti-inflammatory drug (IC_{50} = 20.1 µM). Compound **CP20** (IC_{50} = 20.6 µM) exhibited moderate NO inhibitory activity when compared with that of compound **CP21** (IC_{50} = 4.4 µM), whereas compound **CP23** was inactive (IC_{50} >100 µM). From the result, it might be proposed that the pyran ring of 1,3,5-trioxygenatedxanthone (**CP21**) was essential for NO inhibitory activity than the furan ring (**CP20**), whereas catechol unit of compound **CP26** might play an important role for NO inhibition. Moreover, compound **CP21** has a part of hemiacetal moiety in its pyran ring, which would be possible to open to an aldehyde side chain in acidic condition. The experiment has been set up in 20% HCl-CH_3OH. It was found that an aldehyde product was

not observed, instead the methylated product **CP21a** was formed. Compound **CP21a** was further tested for NO inhibition which showed less NO inhibitory activity than **CP21** with an IC_{50} value of 9.5 µM. From this result, it could be suggested that the methoxyl group might decrease the NO inhibitory activity (Boonnak *et al.*, 2010).

Table 53 Nitric oxide inhibitory activity of compounds **CP20**, **CP21**, **CP21a**, **CP23** and **CP26**

No	% Inhibition of various concentrations [µM]					IC_{50} [µM]
	0	3	10	30	100	
CP20	0.0±4.5	15.5±0.9[**]	29.6±0.9[**]	44.0±1.6[**]	95.3±0.6[a**]	20.6
CP21	0.0±4.5	34.8±1.4[*]	72.7±1.1[**]	93.1±1.7[**]	99.5±0.6[a**]	4.4
CP21a	0.0±2.4	15.7±5.3	44.4±1.9[**]	91.0±1.0[**]	101.3±3.0[a**]	9.5
CP23	0.0±5.4	–	21.7±1.0[*]	23.5±2.0[**]	47.4±3.1[**]	>100
CP26	0.0±5.4	30.1±1.6[**]	94.3±0.7[a**]	102.1±2.4[a**]	102.9±0.7[a**]	4.3
Indomethacin	0.0±4.2	16.6±2.9	32.7±2.6[**]	53.4±3.0[**]	85.6±1.8[**]	20.1
L-NA	0.0±5.6	15.3±2.8	21.4±2.5	35.6±2.1[**]	73.2±3.5[**]	59.0
CAPE	0.0±5.6	35.2±3.0[*]	70.3±2.7[**]	97.6±2.4[a**]	99.5±2.7[a**]	5.0

[a] Cytotoxic effect was observed.
[b] Each value represents mean ± S.E.M. of four determinations.
Statistical significance, $*p<0.05$, $**p<0.01$

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APPENDIX
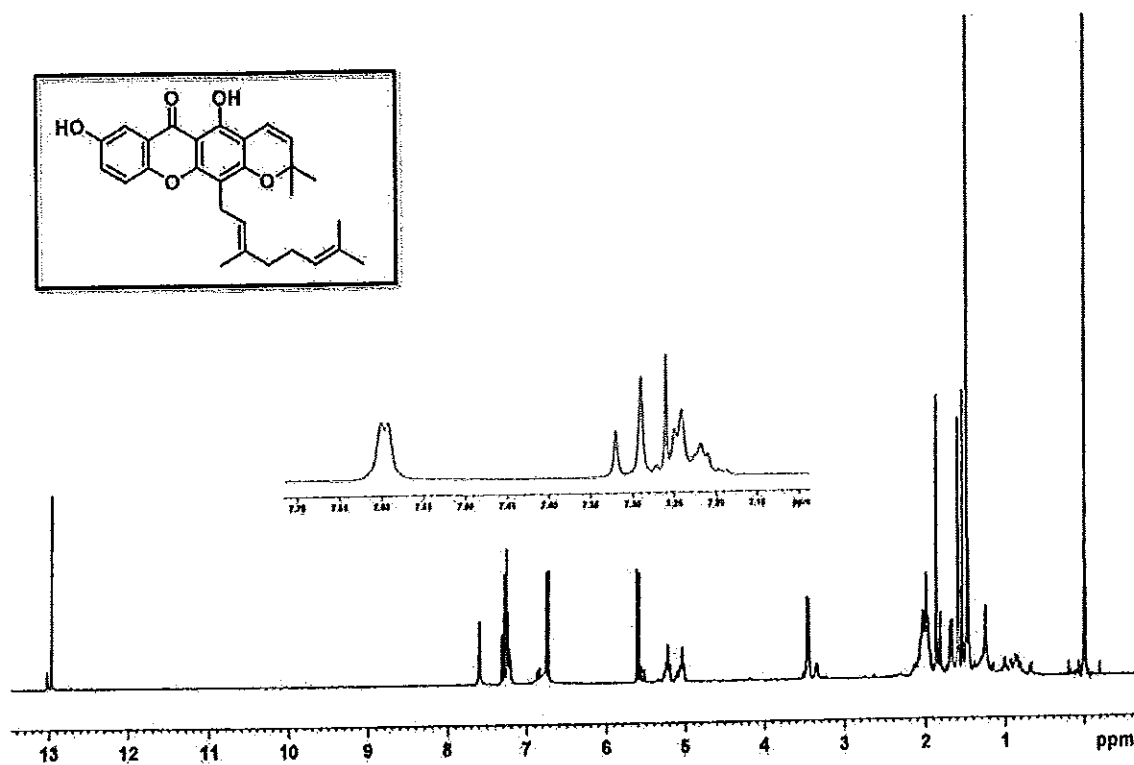
Figure 82 ^{1}H NMR (300 MHz, CDCl$_3$) spectrum of **CC1**
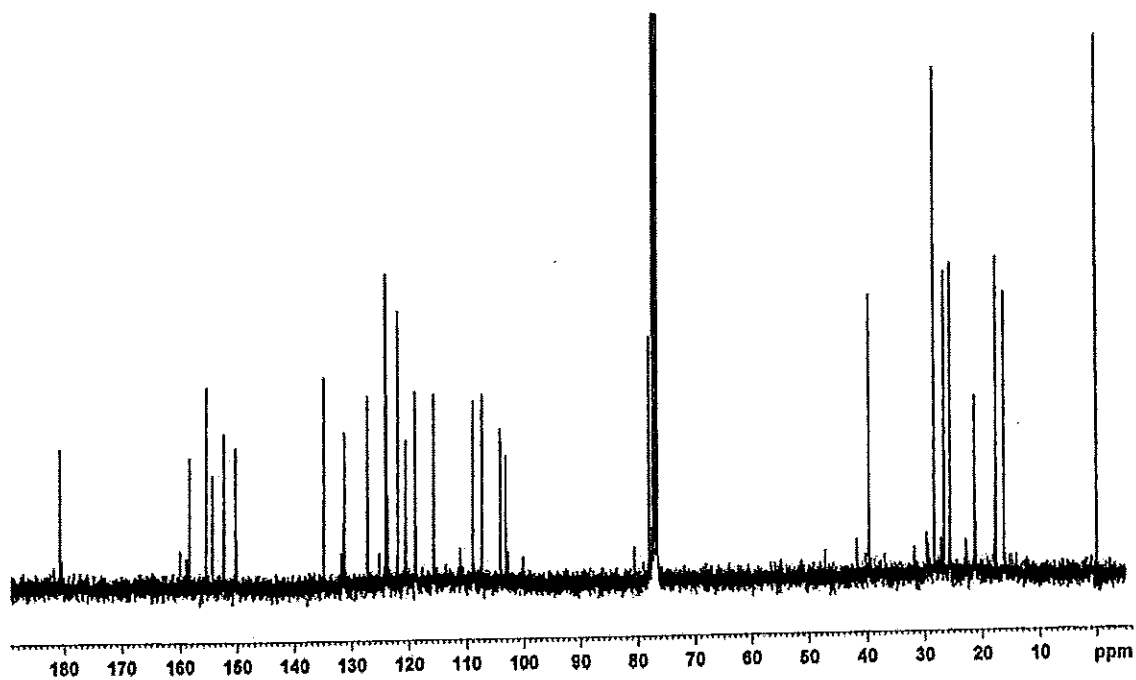
Figure 83 ^{13}C NMR (75 MHz, CDCl$_3$) spectrum of **CC1**
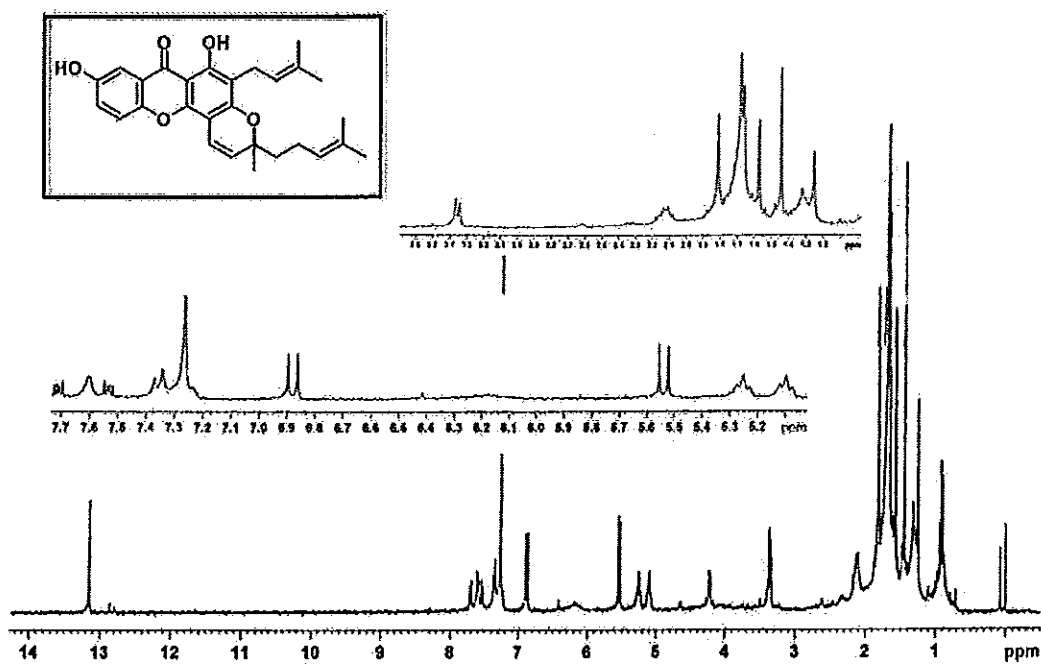
Figure 84 ^{1}H NMR (300 MHz, CDCl$_3$) spectrum of **CC2**
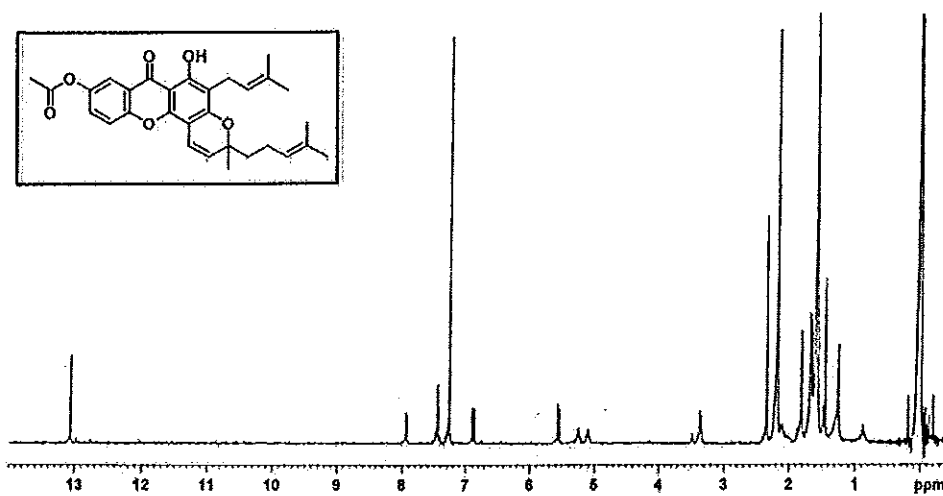
Figure 85 ^{1}H NMR (300 MHz, CDCl$_3$) spectrum of **CC2a**
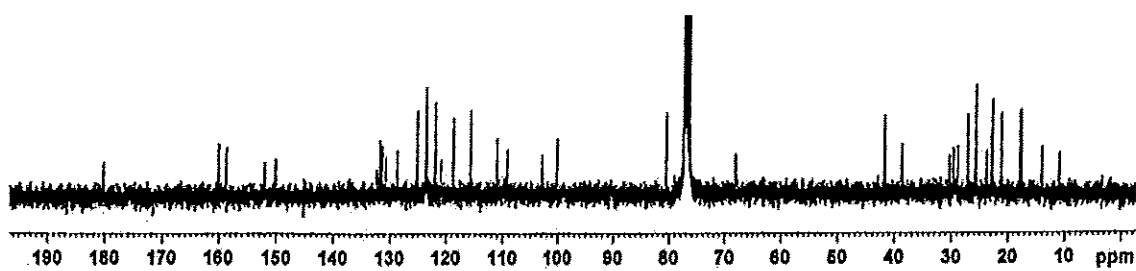
Figure 86 ^{13}C NMR (75 MHz, CDCl$_3$) spectrum of **CC2**
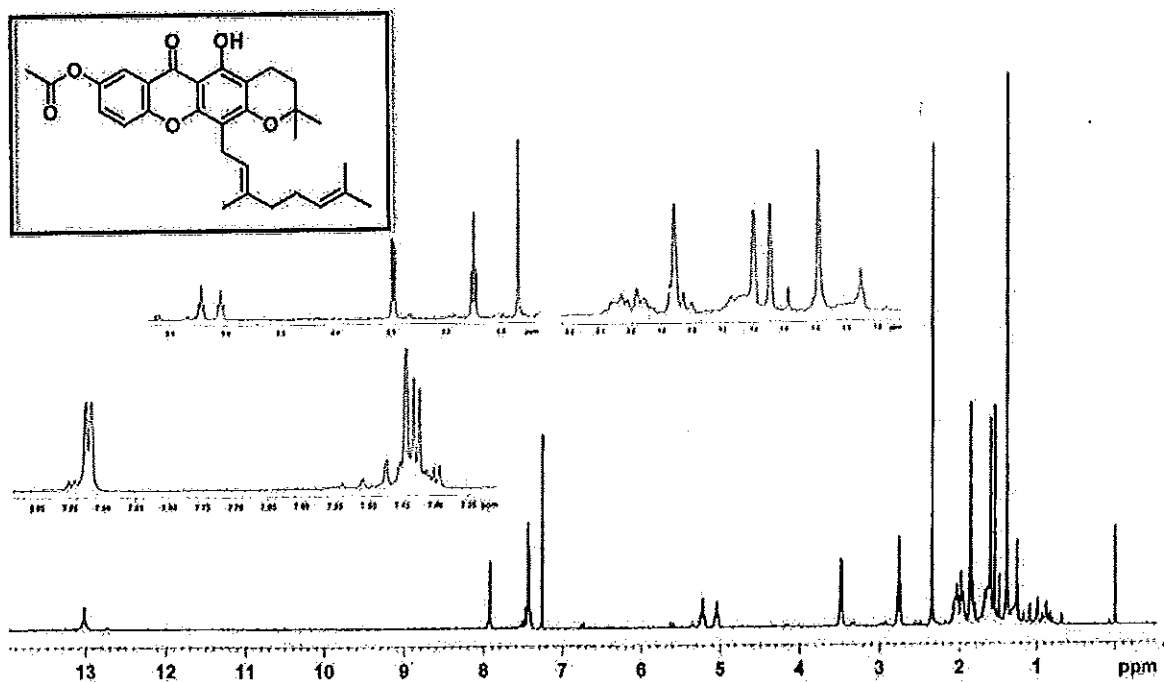
Figure 87 ^{1}H NMR (300 MHz, CDCl$_3$) spectrum of **CC3a**
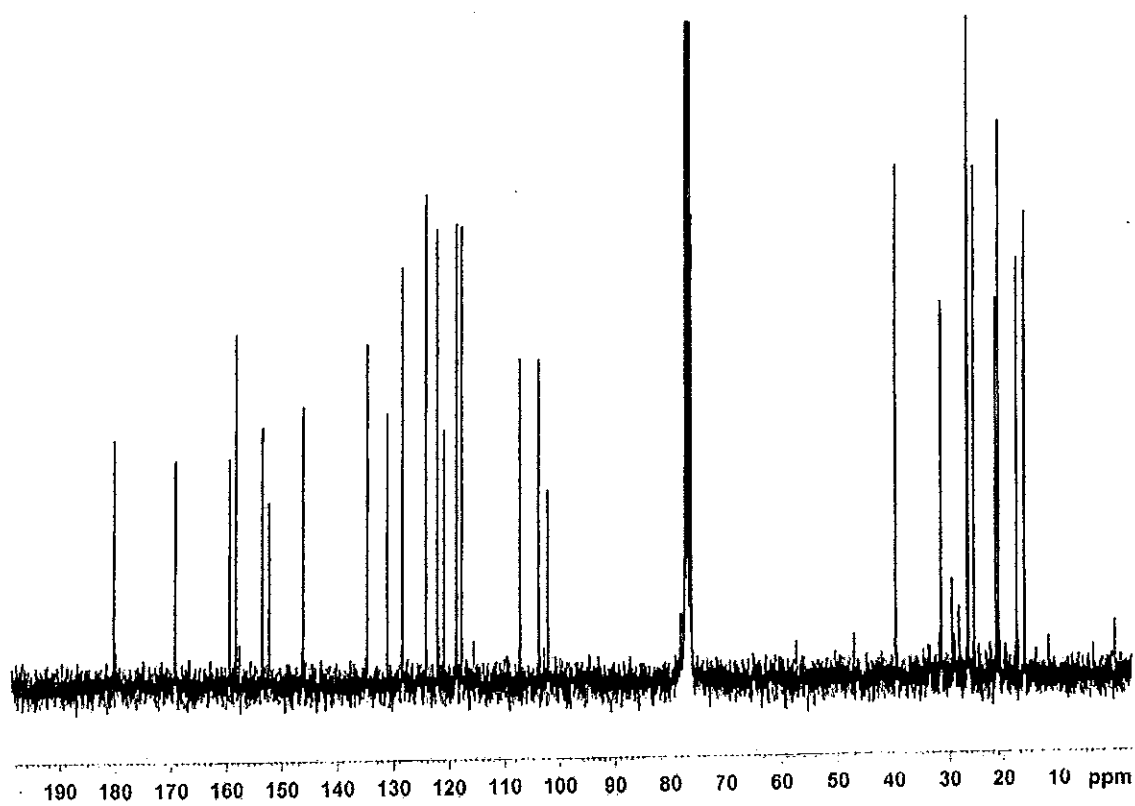
Figure 88 ^{13}C NMR (75 MHz, CDCl$_3$) spectrum of **CC3a**
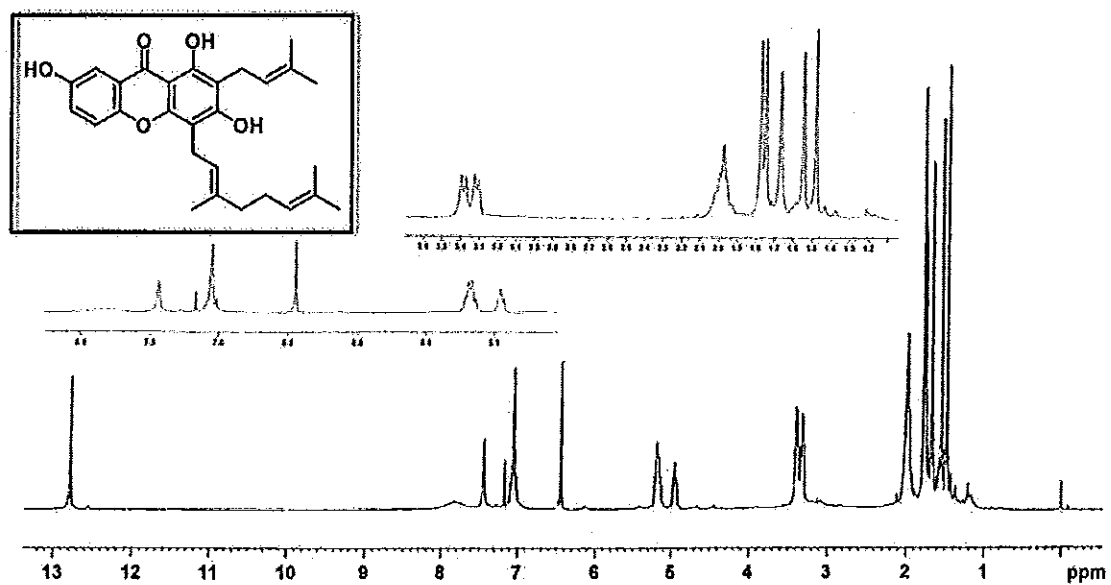
Figure 89 ^{1}H NMR (300 MHz, CDCl$_3$) spectrum of **CC4**
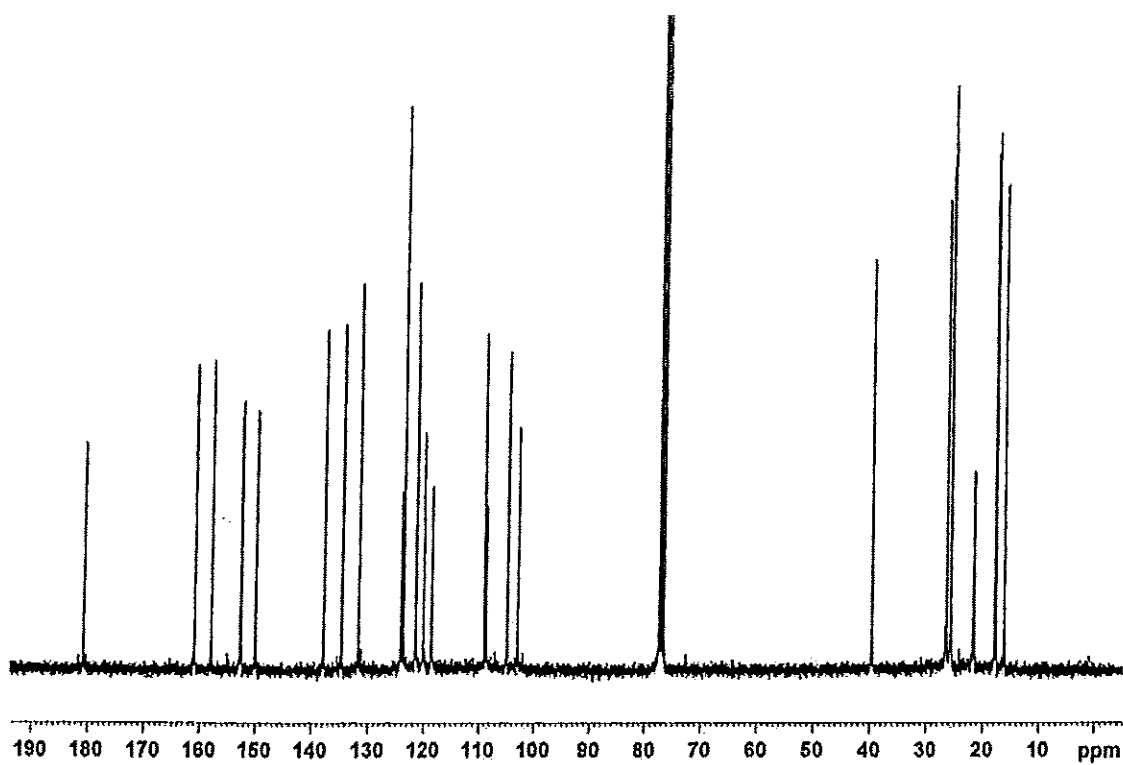
Figure 90 ^{13}C NMR (75 MHz, CDCl$_3$) spectrum of **CC4**
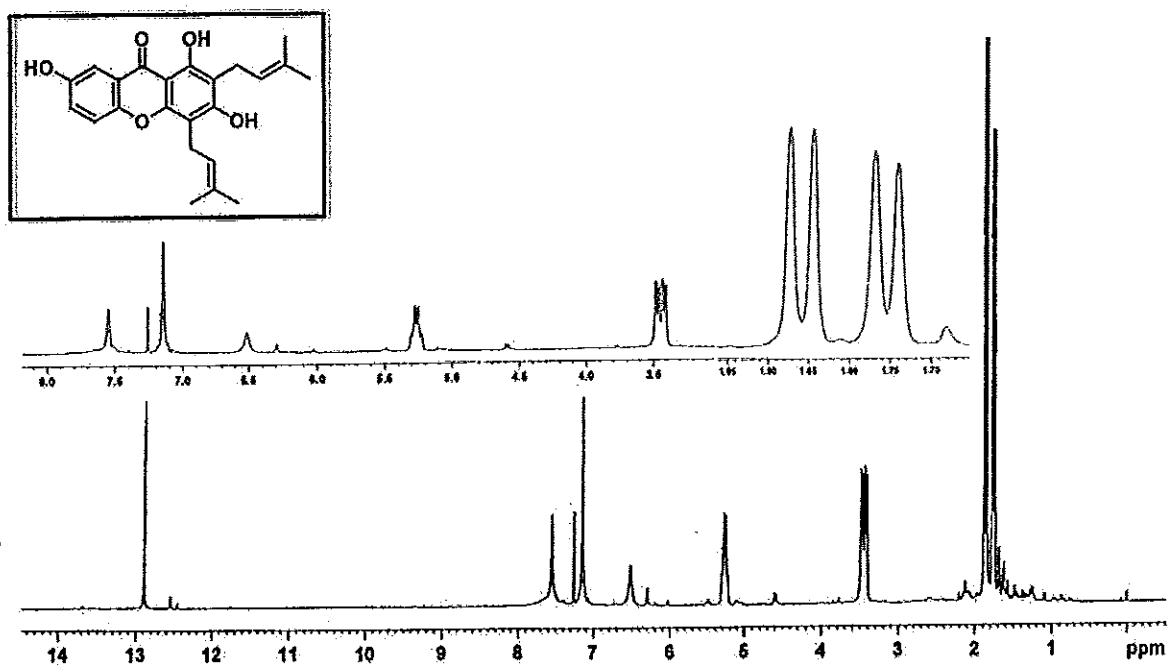
Figure 91 ¹H NMR (300 MHz, CDCl₃) spectrum of **CC5**
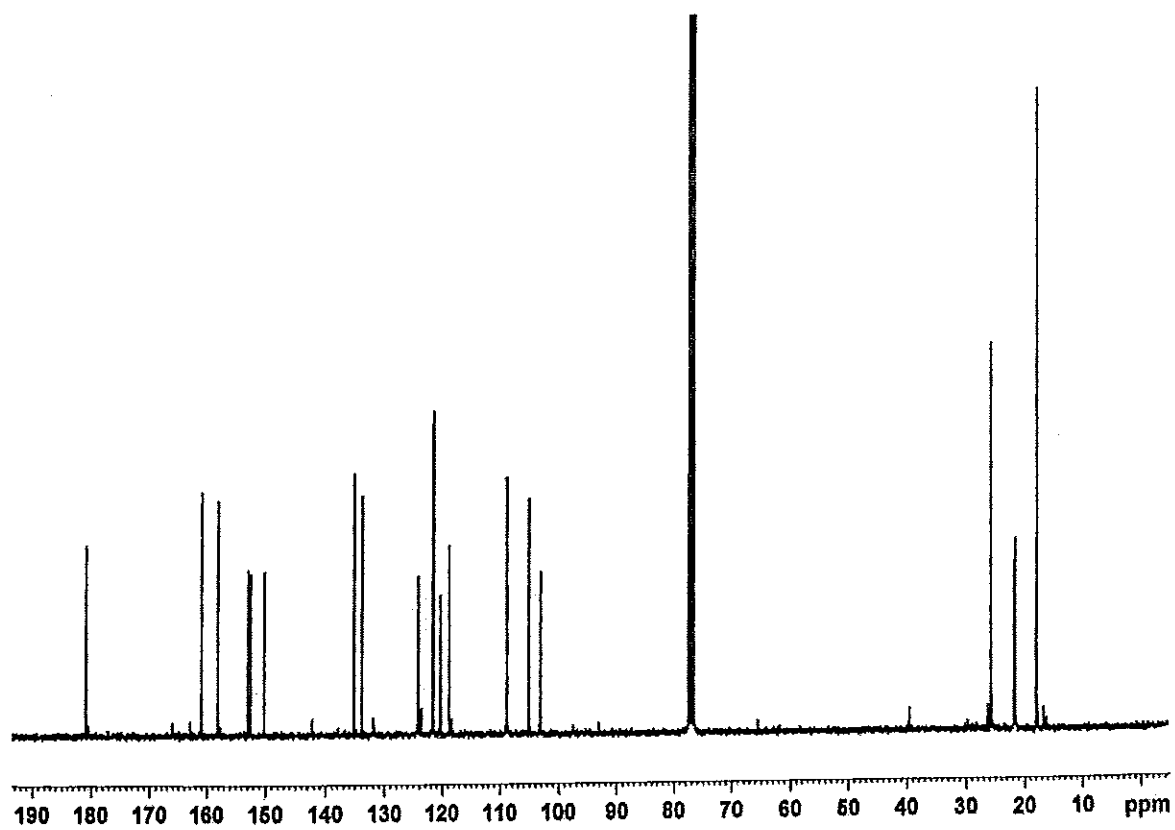
Figure 92 ¹³C NMR (75 MHz, CDCl₃) spectrum of **CC5**
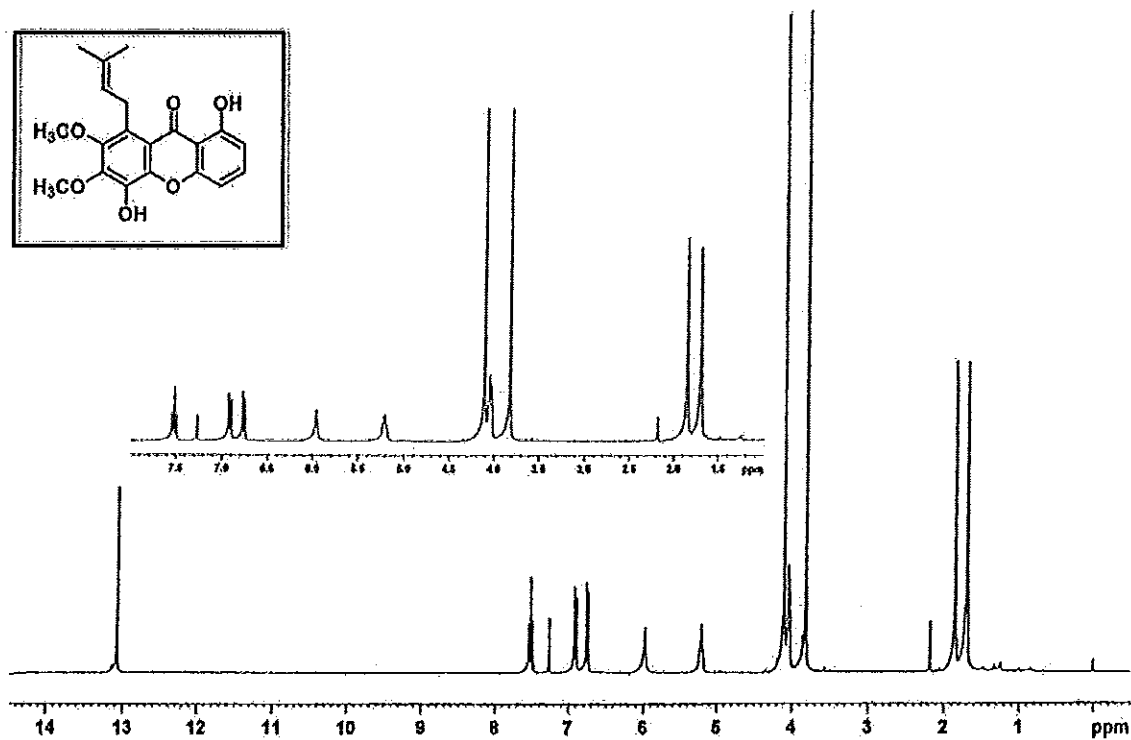
Figure 93 ^{1}H NMR (300 MHz, CDCl$_3$) spectrum of **CC6**
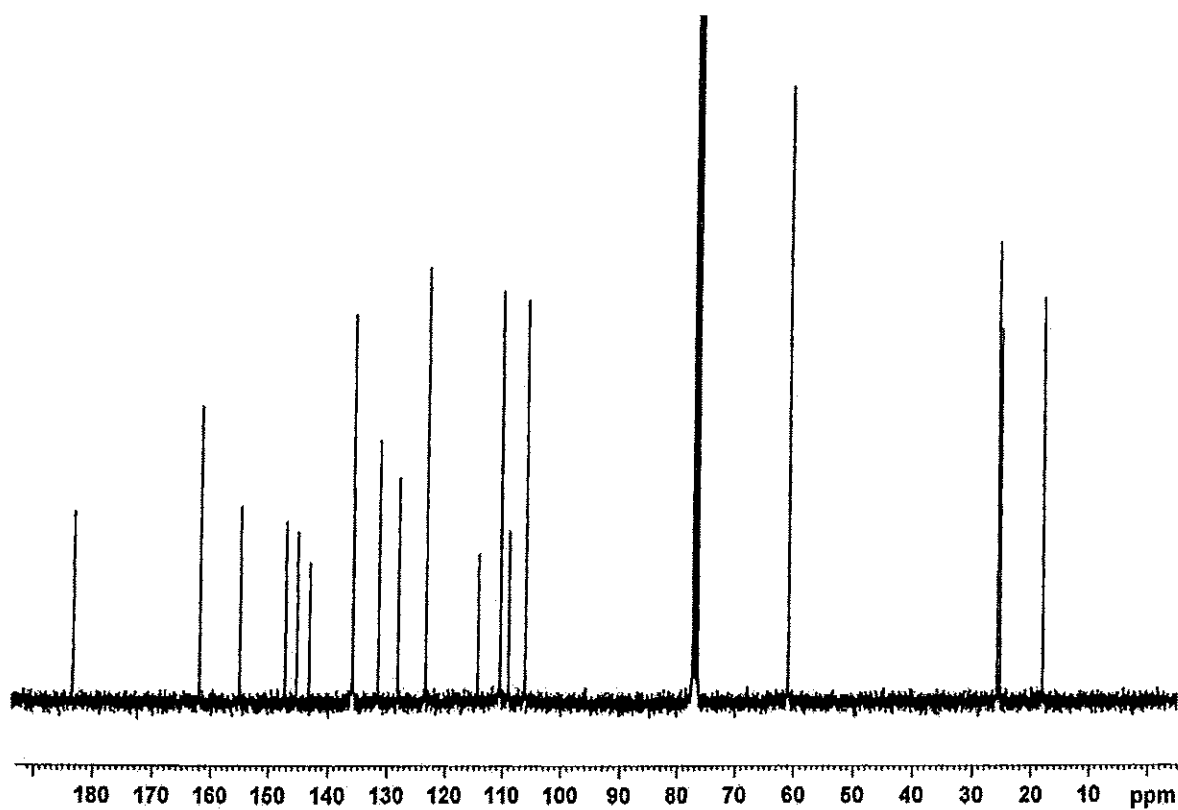
Figure 94 ^{13}C NMR (75 MHz, CDCl$_3$) spectrum of **CC6**
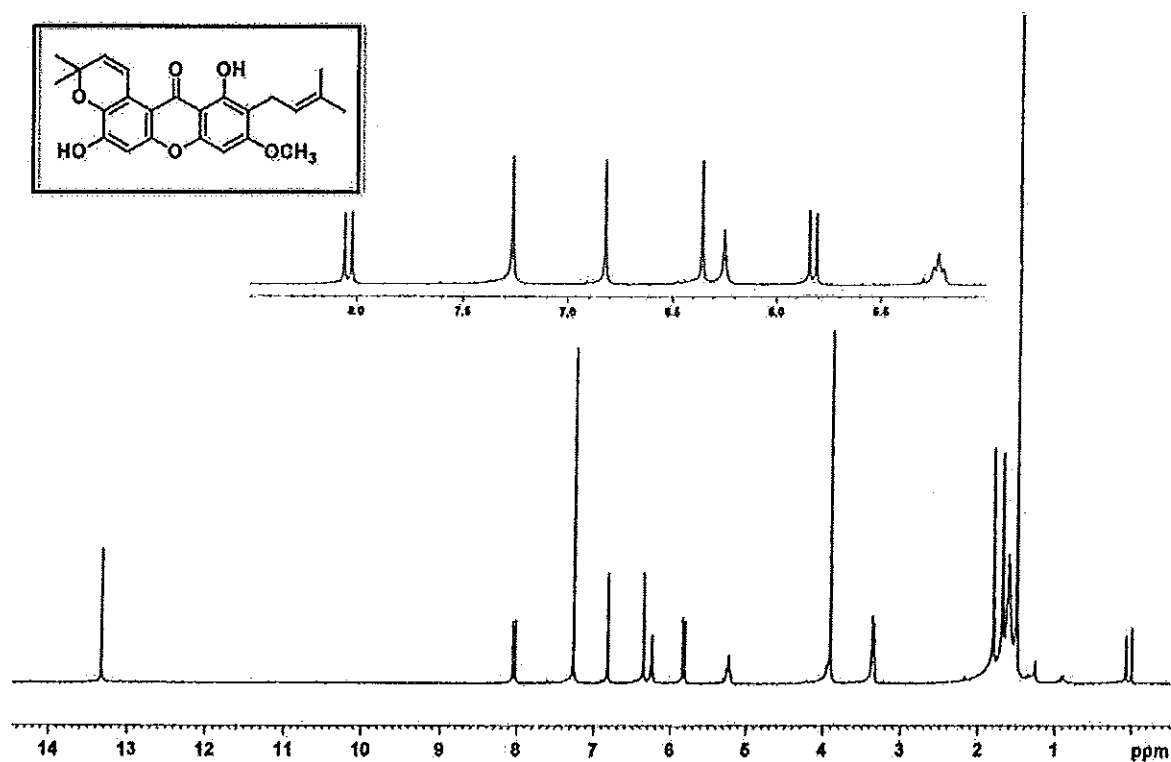
Figure 95 ^{1}H NMR (300 MHz, CDCl$_3$) spectrum of **CC7**
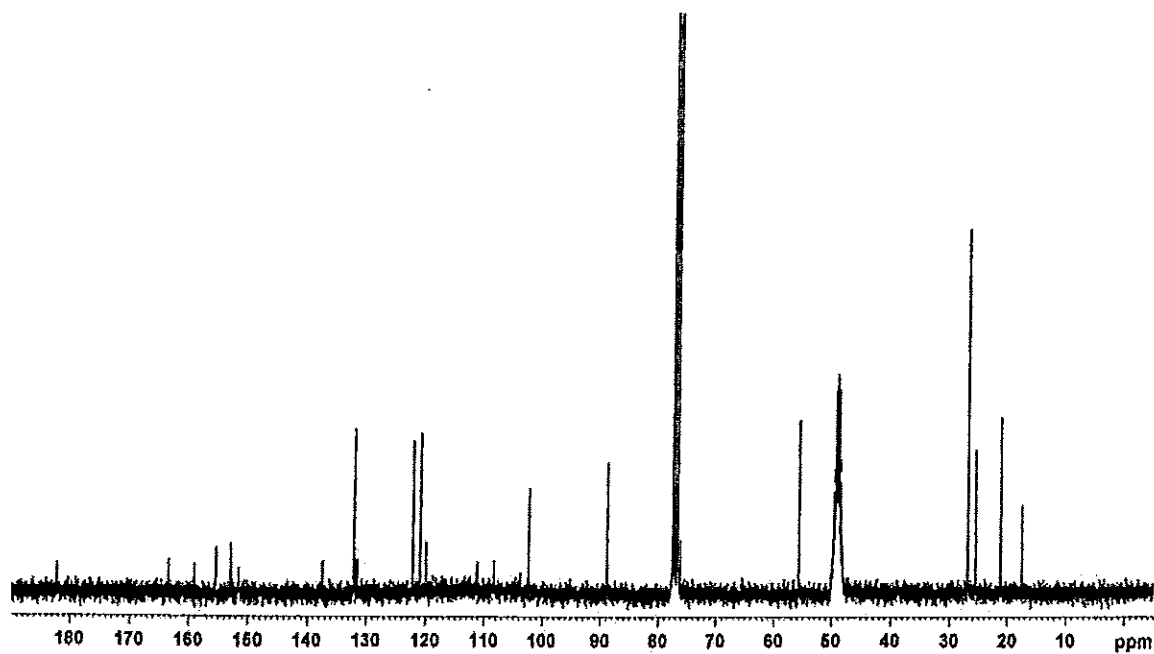
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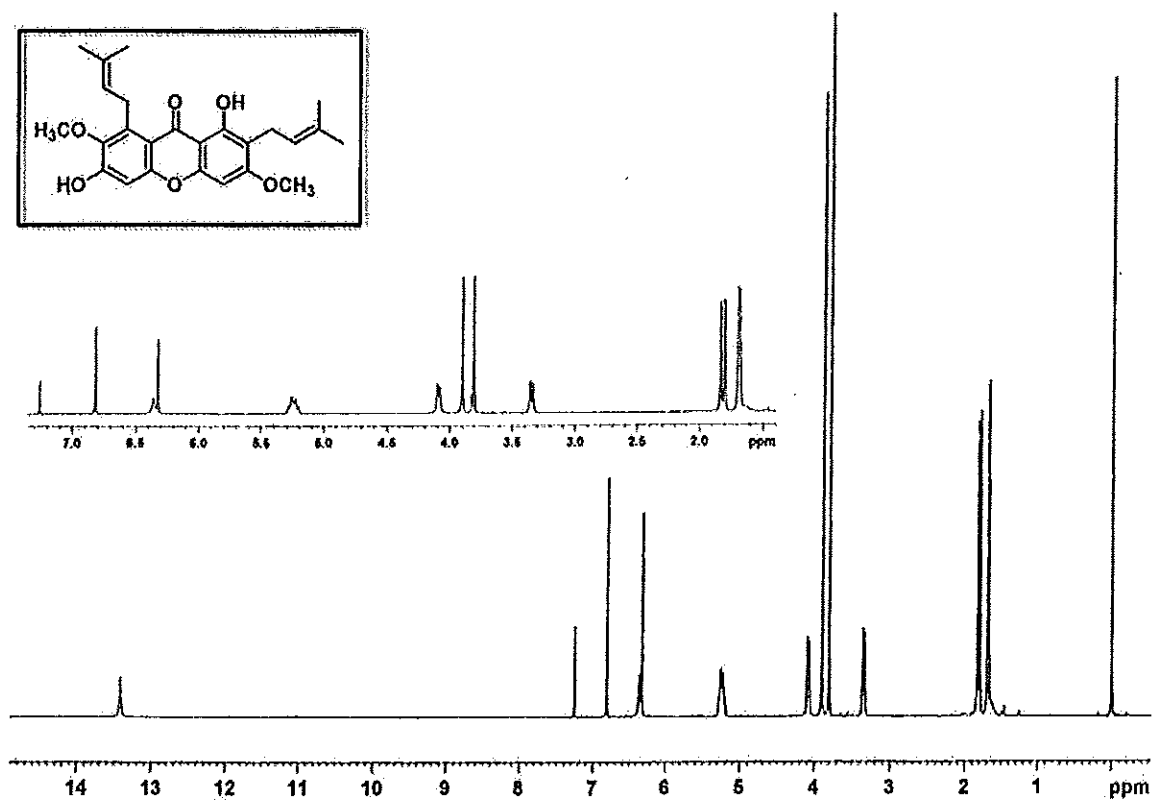
Figure 97 ¹H NMR (300 MHz, CDCl₃) spectrum of **CC8**
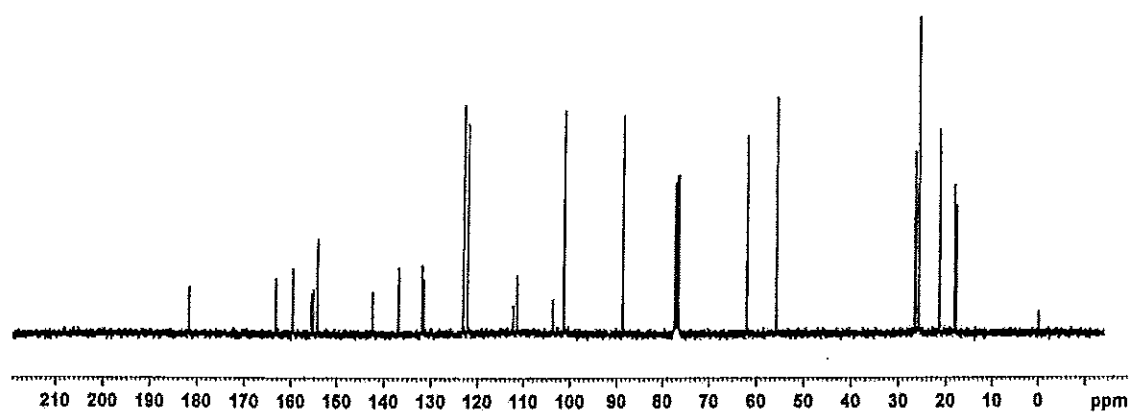
Figure 98 ¹³C NMR (75 MHz, CDCl₃) spectrum of **CC8**
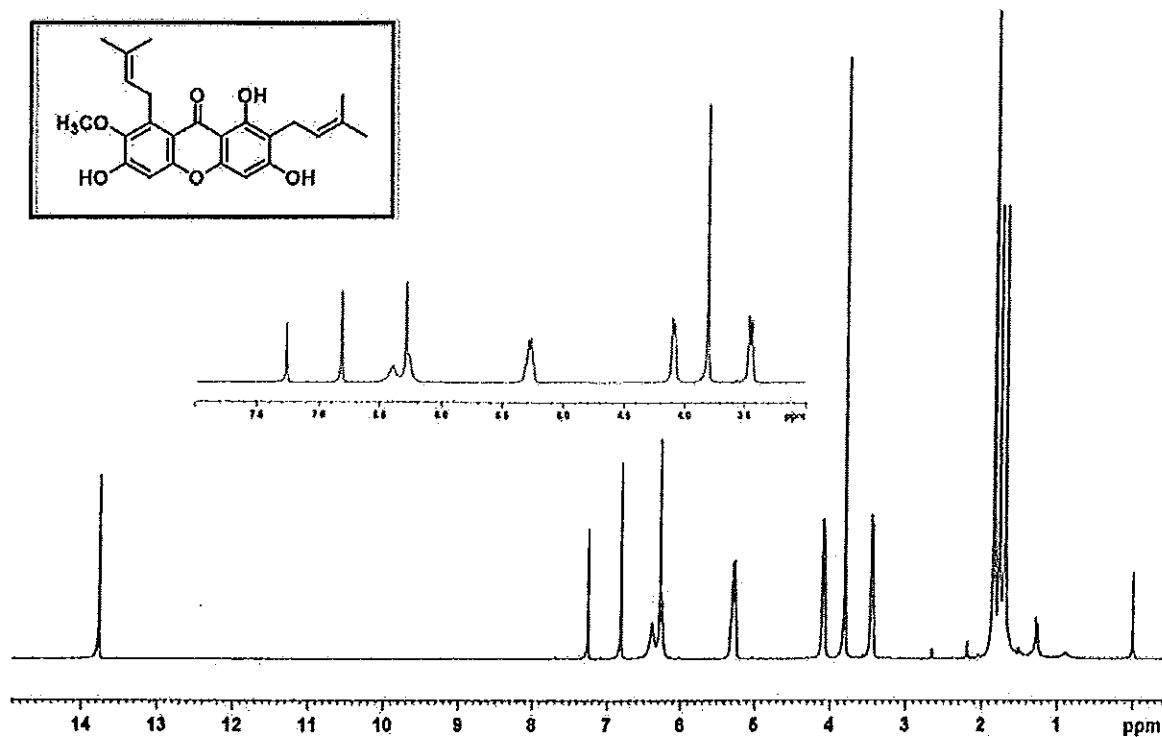
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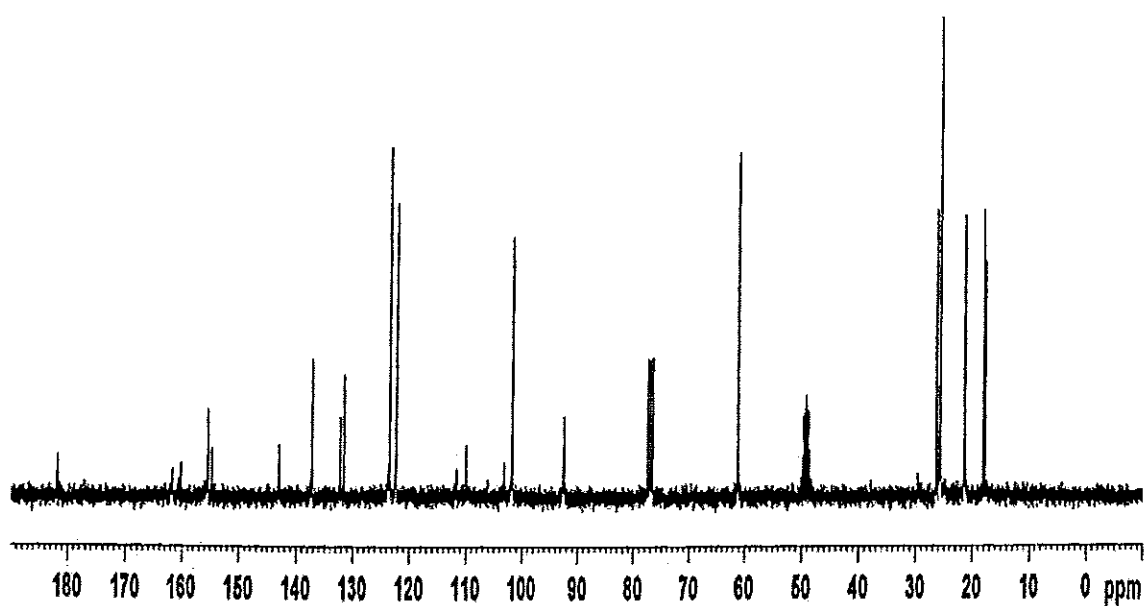
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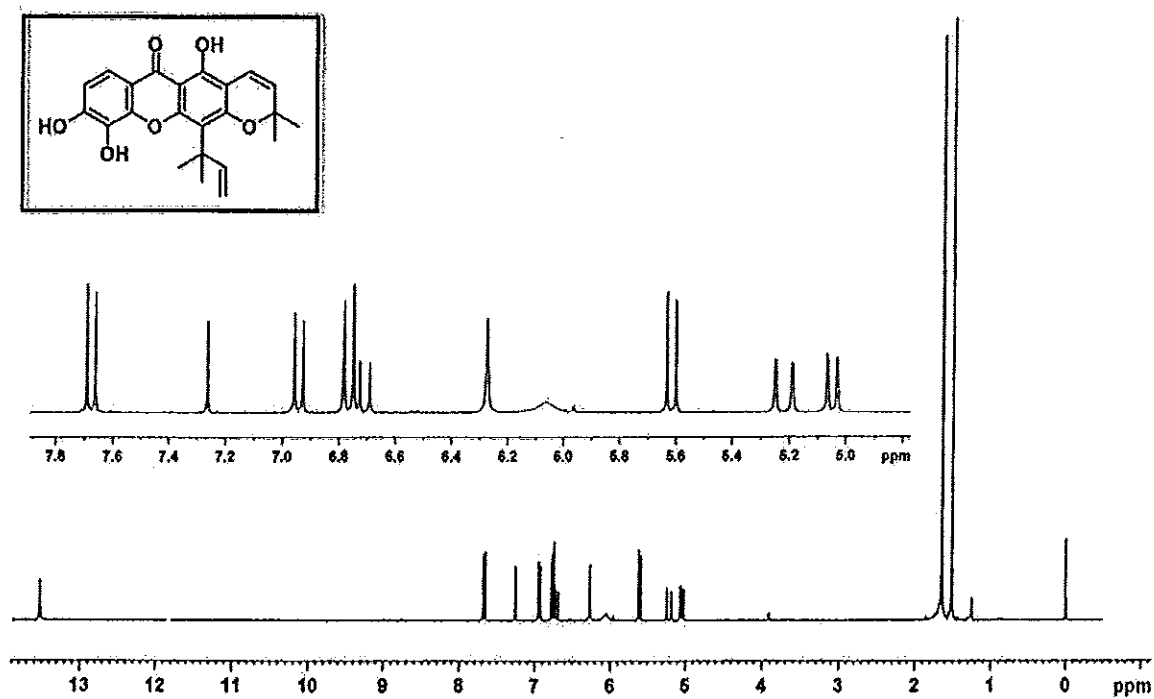
Figure 101 ^{1}H NMR (300 MHz, CDCl$_3$) spectrum of **CC10**
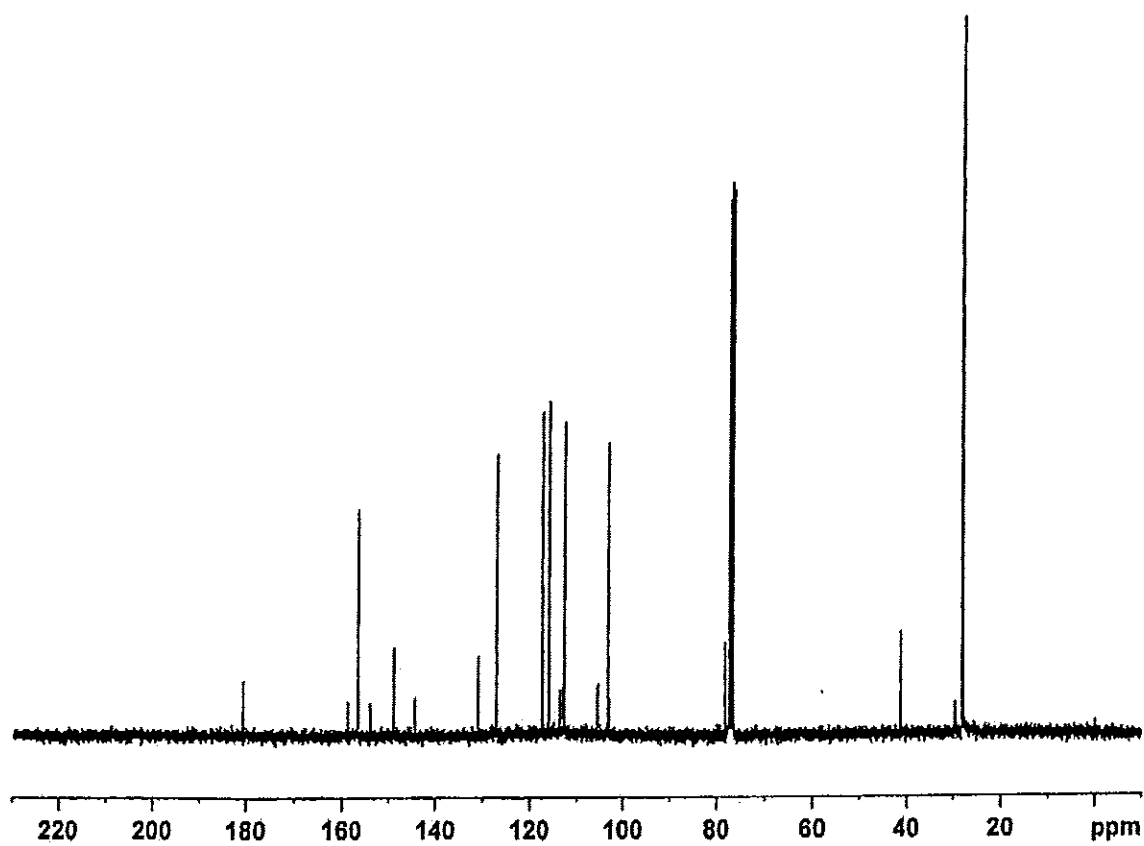
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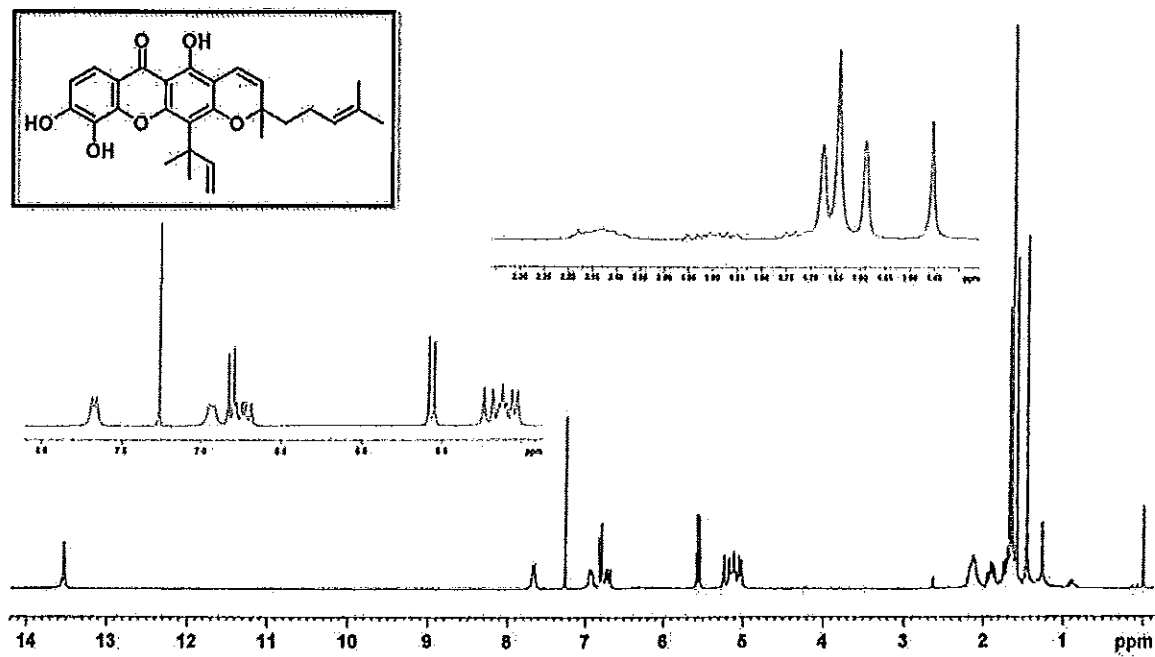
Figure 103 ^{1}H NMR (300 MHz, CDCl$_3$) spectrum of **CC11**
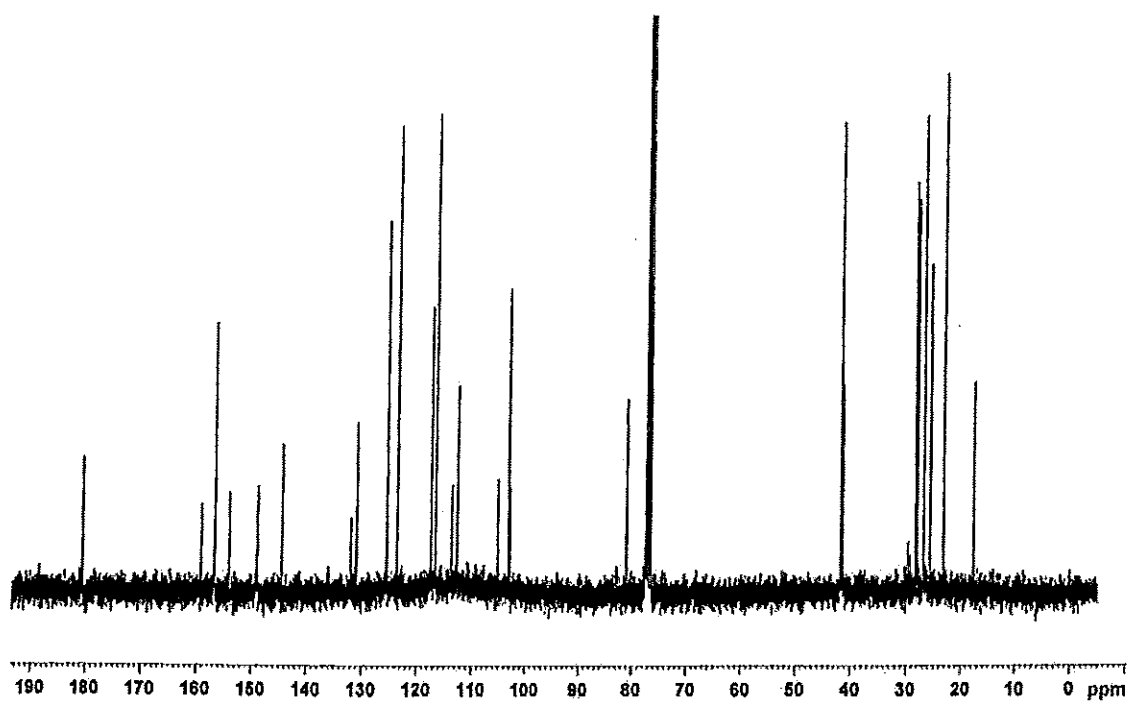
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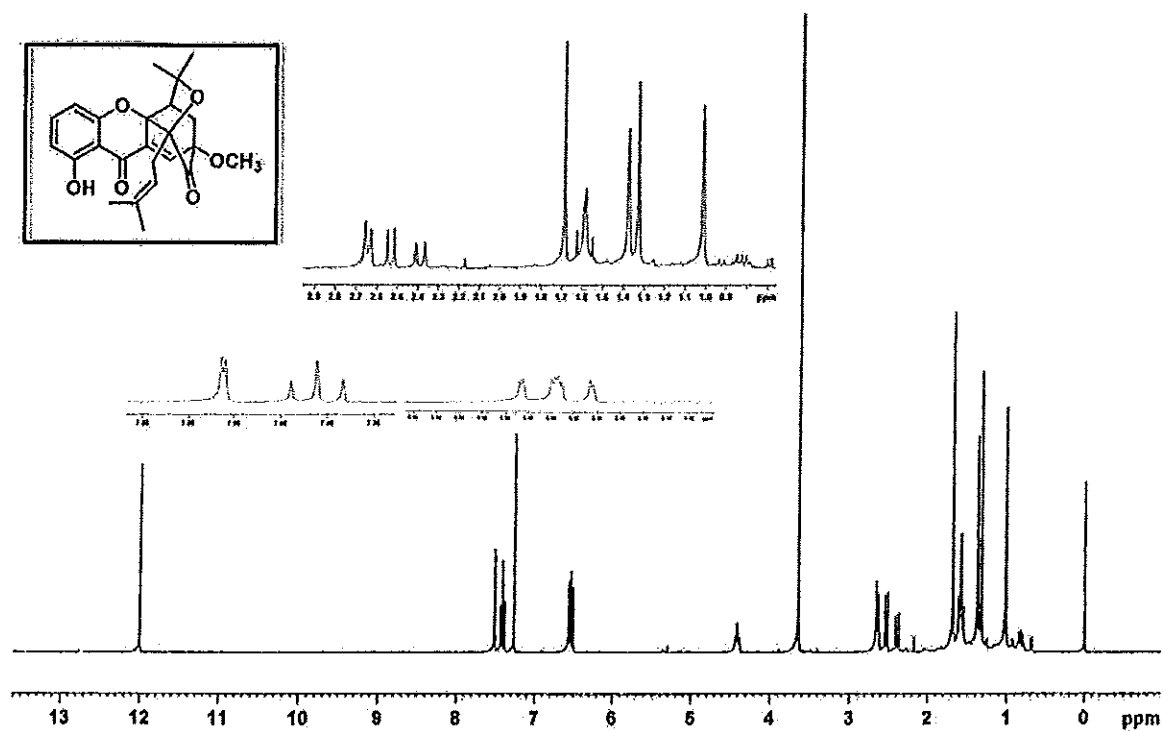
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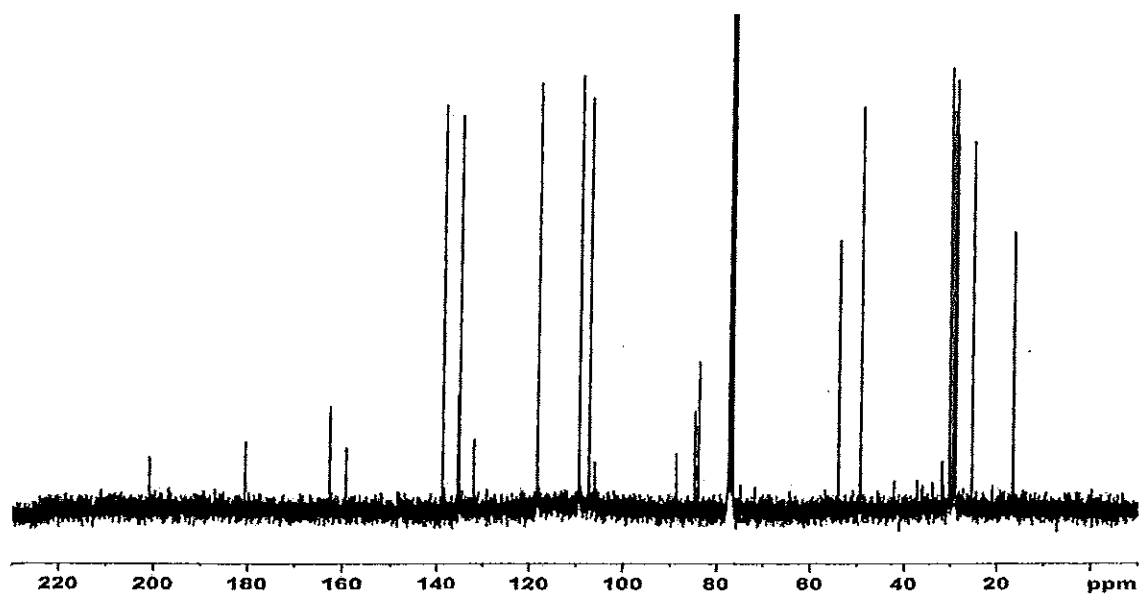
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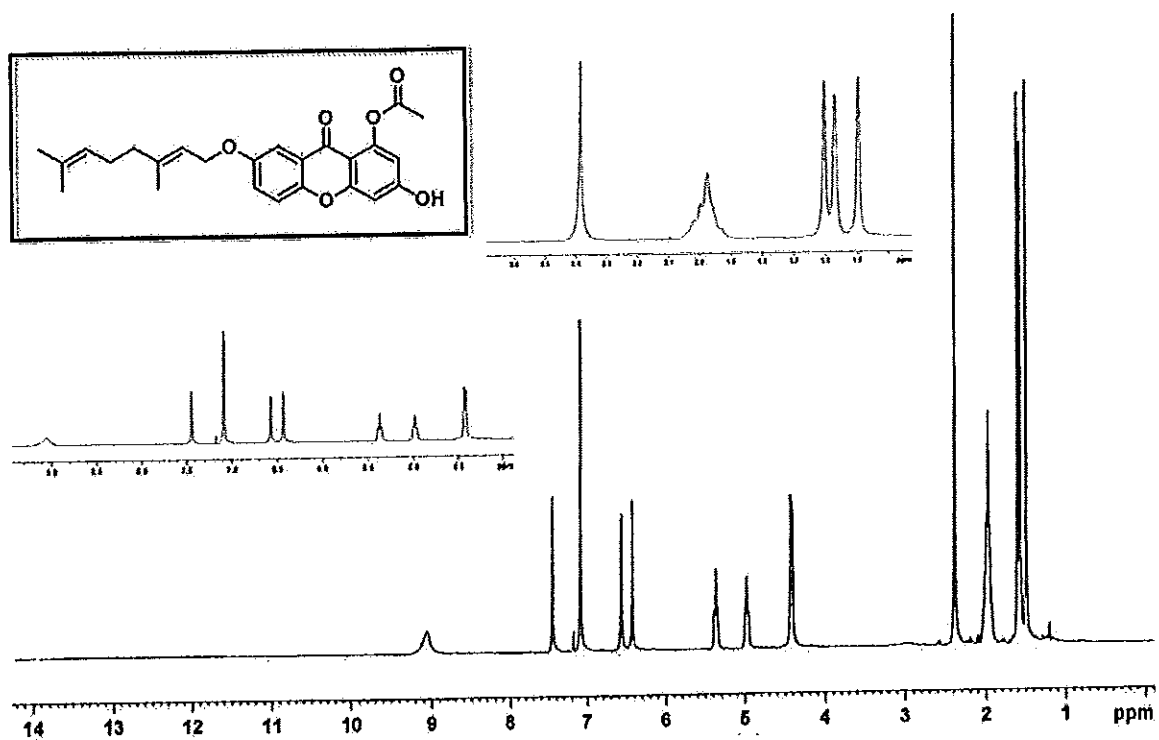
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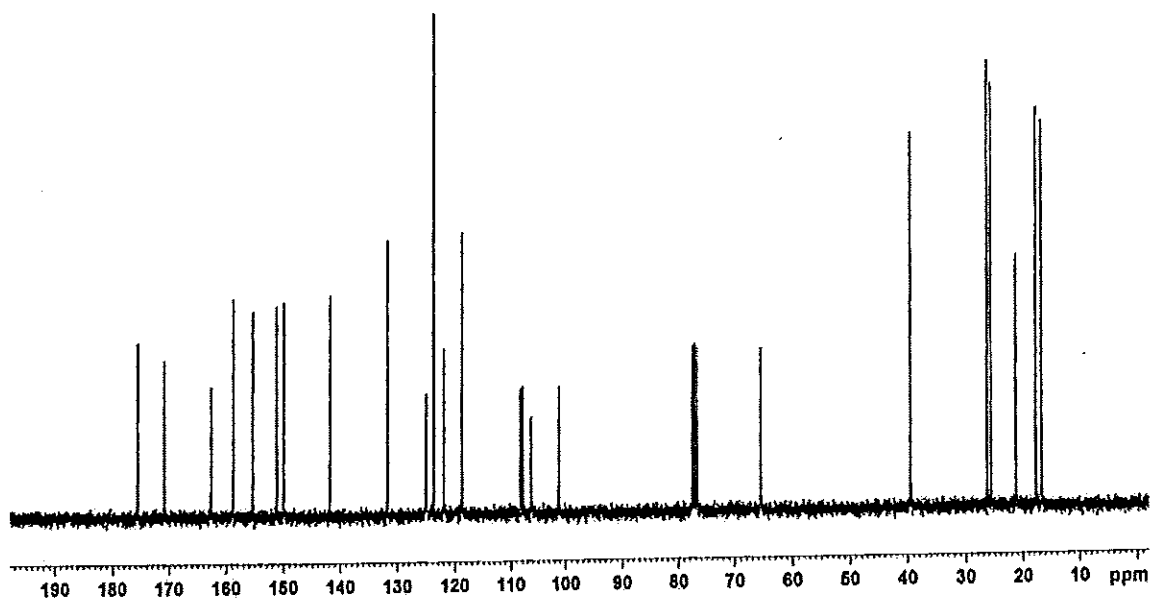
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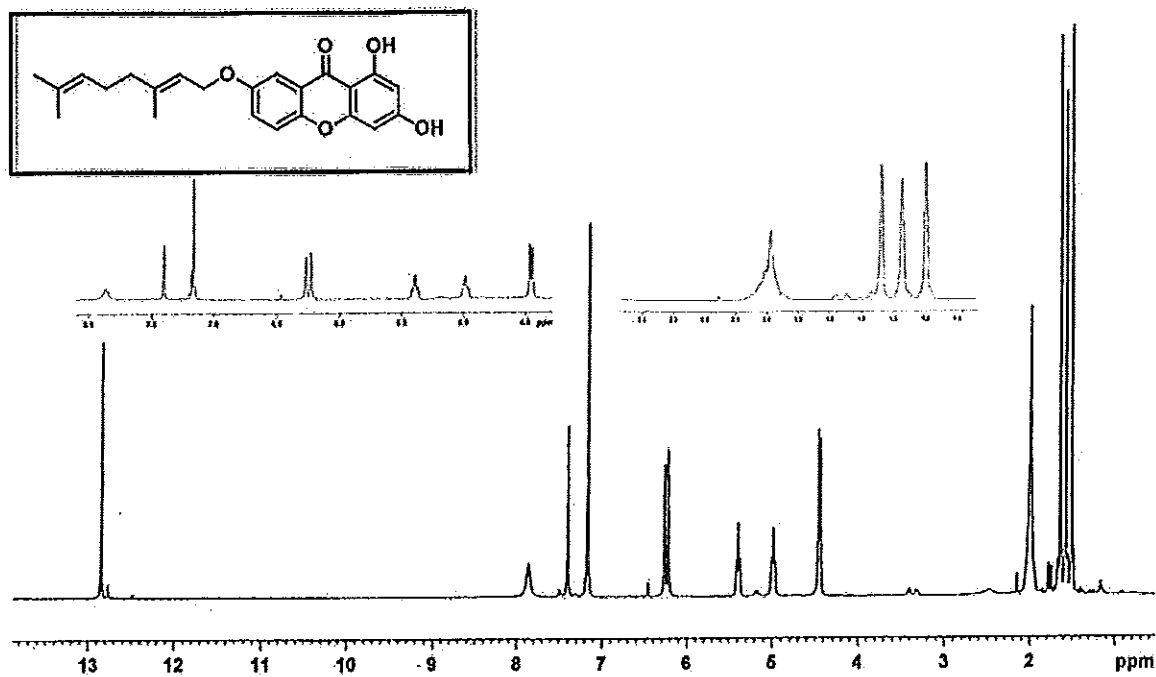
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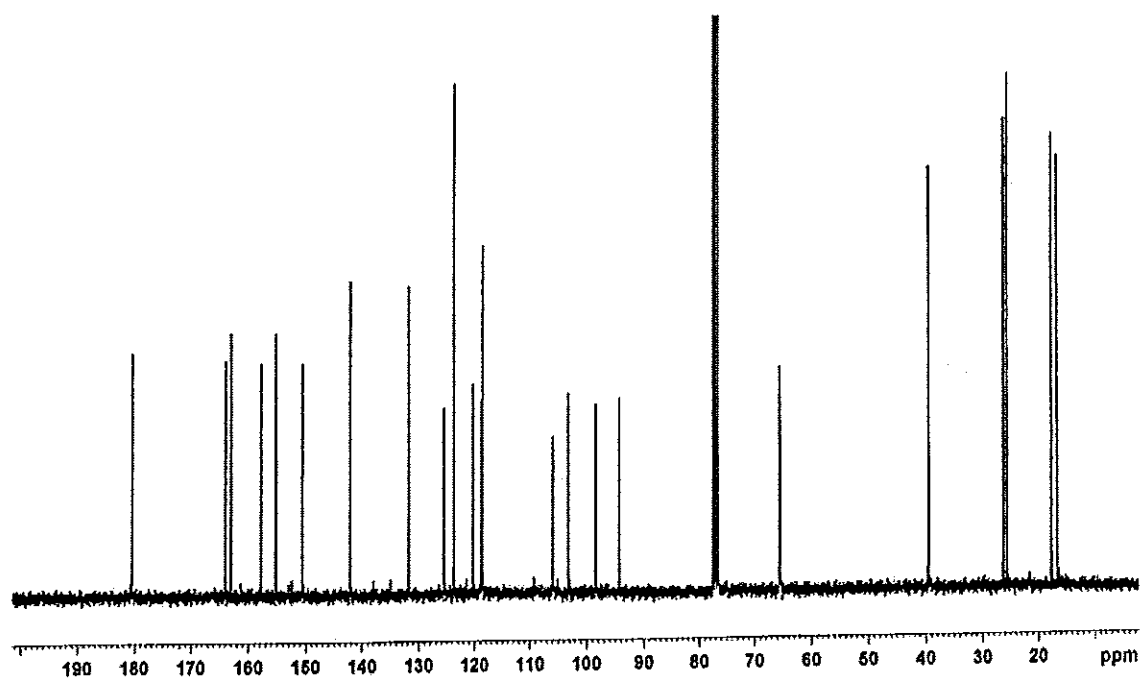
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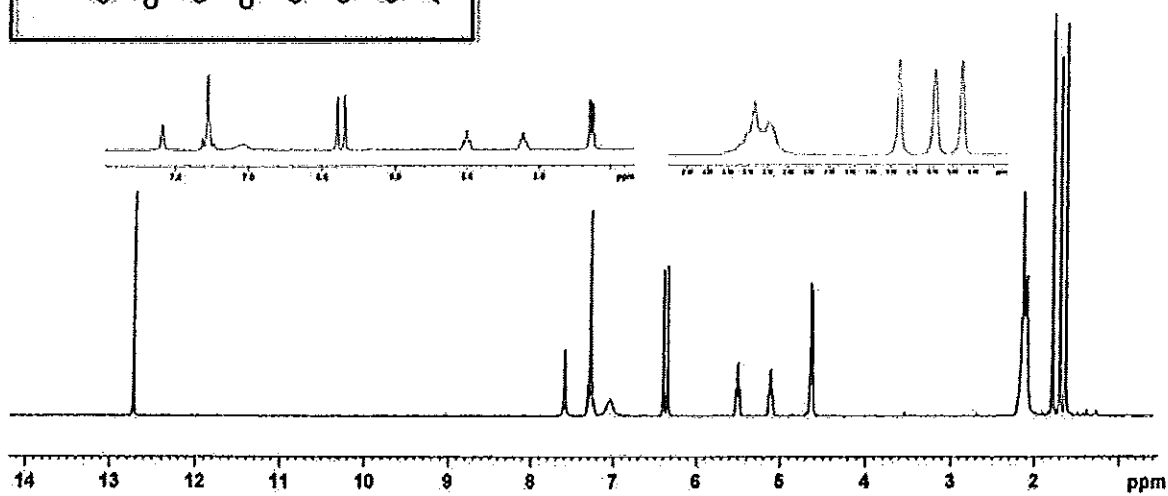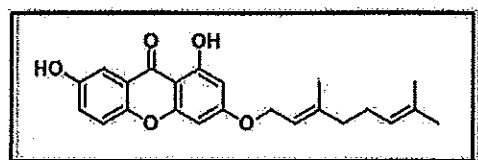
Figure 111 ^{1}H NMR (300 MHz, CDCl$_3$) spectrum of **CC15**
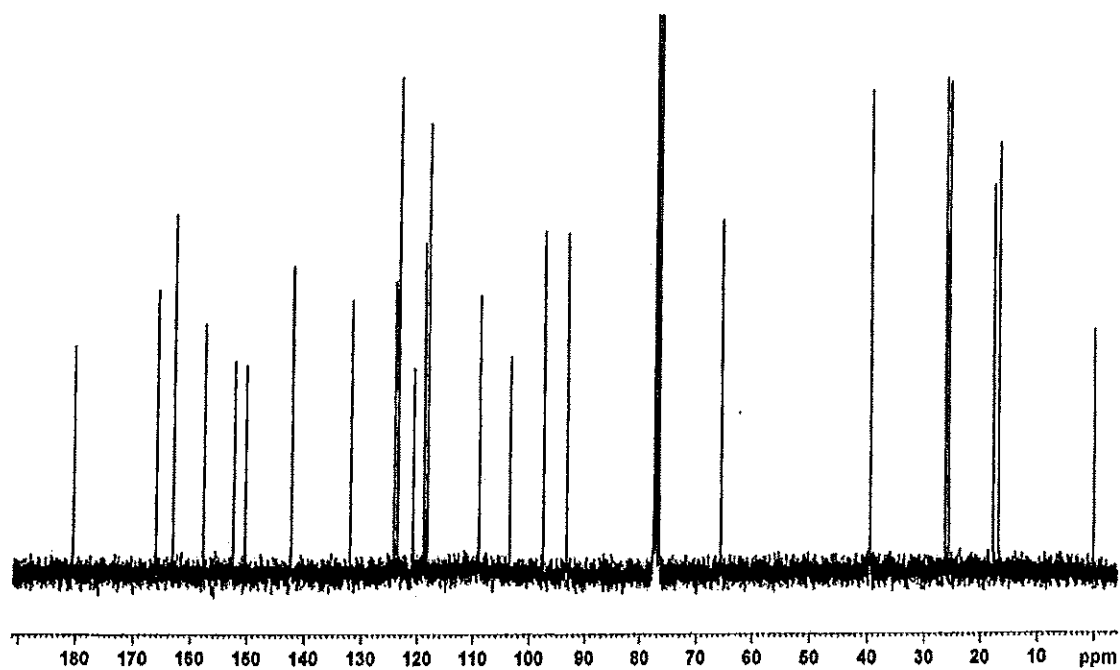
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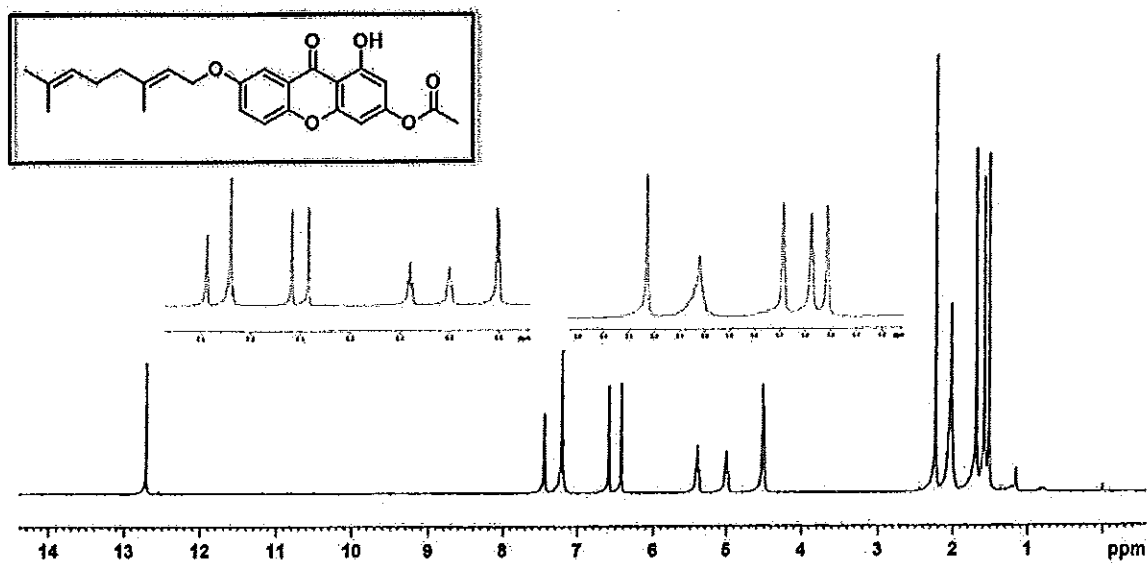
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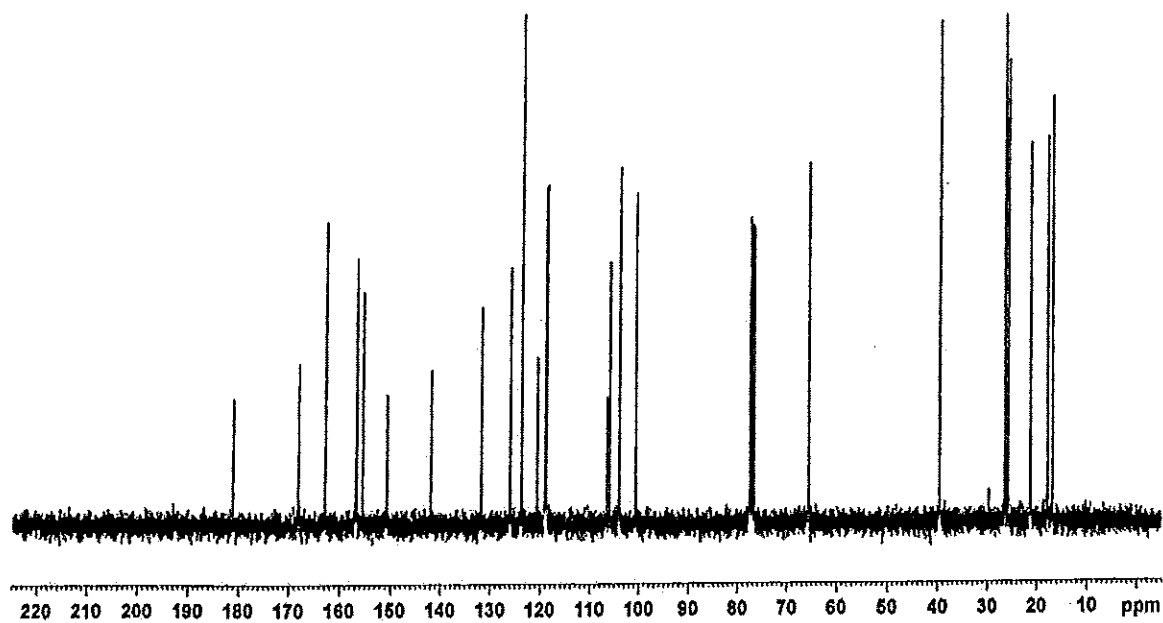
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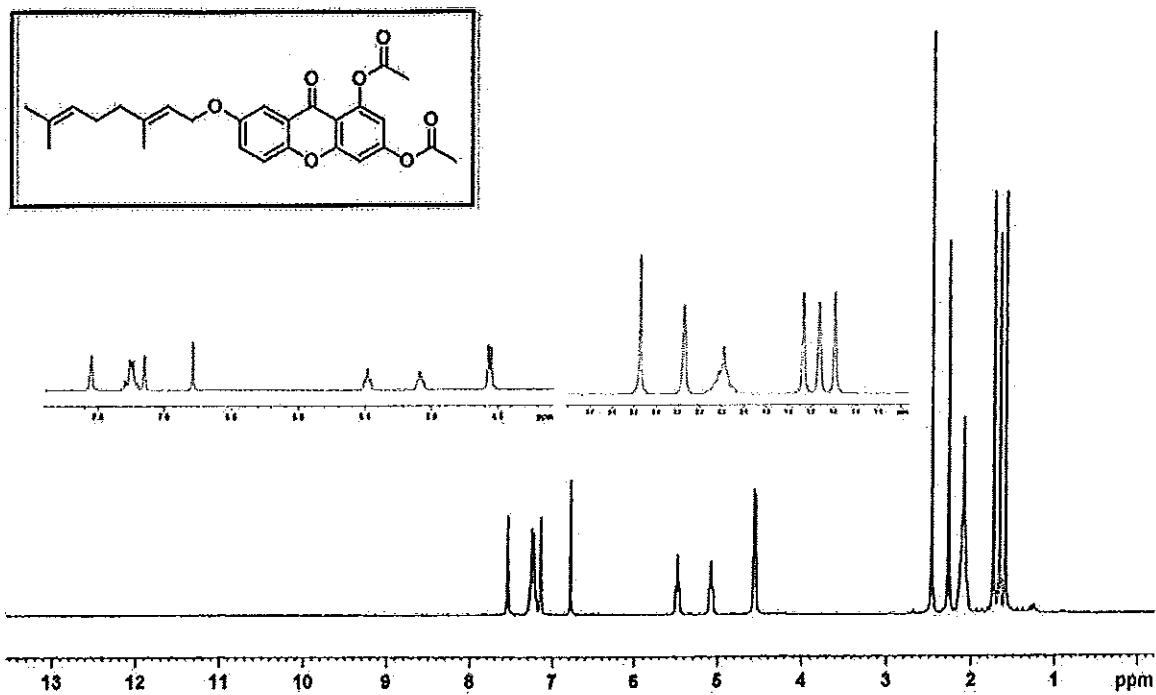
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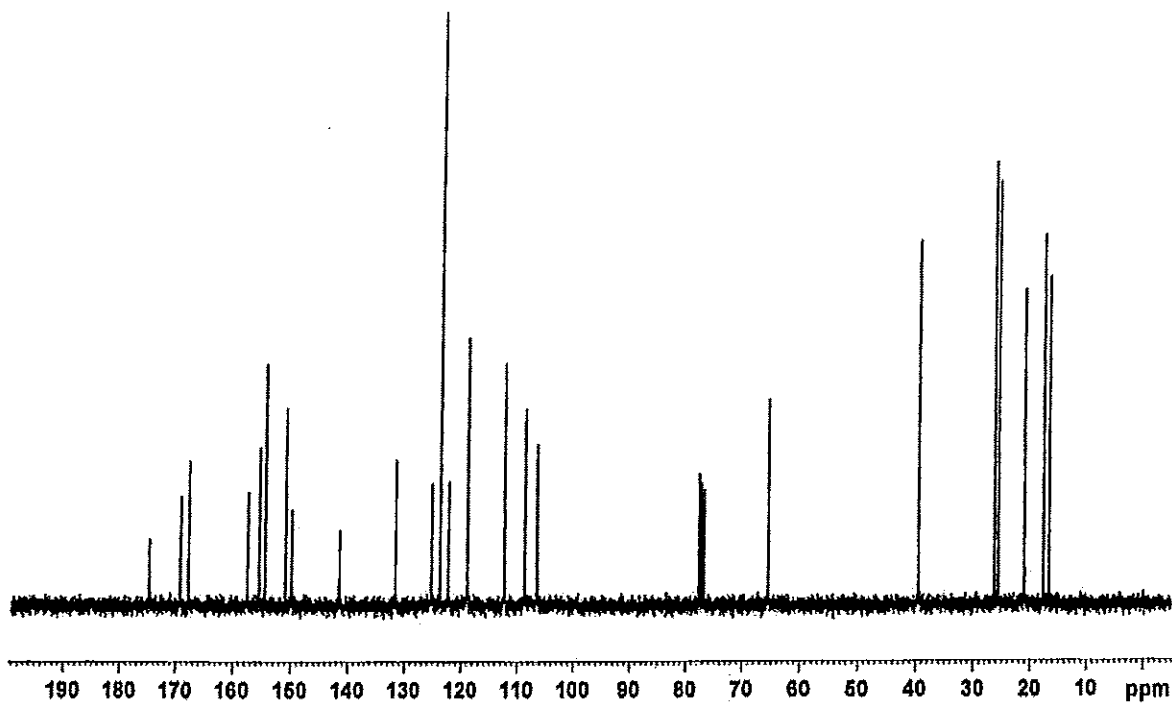
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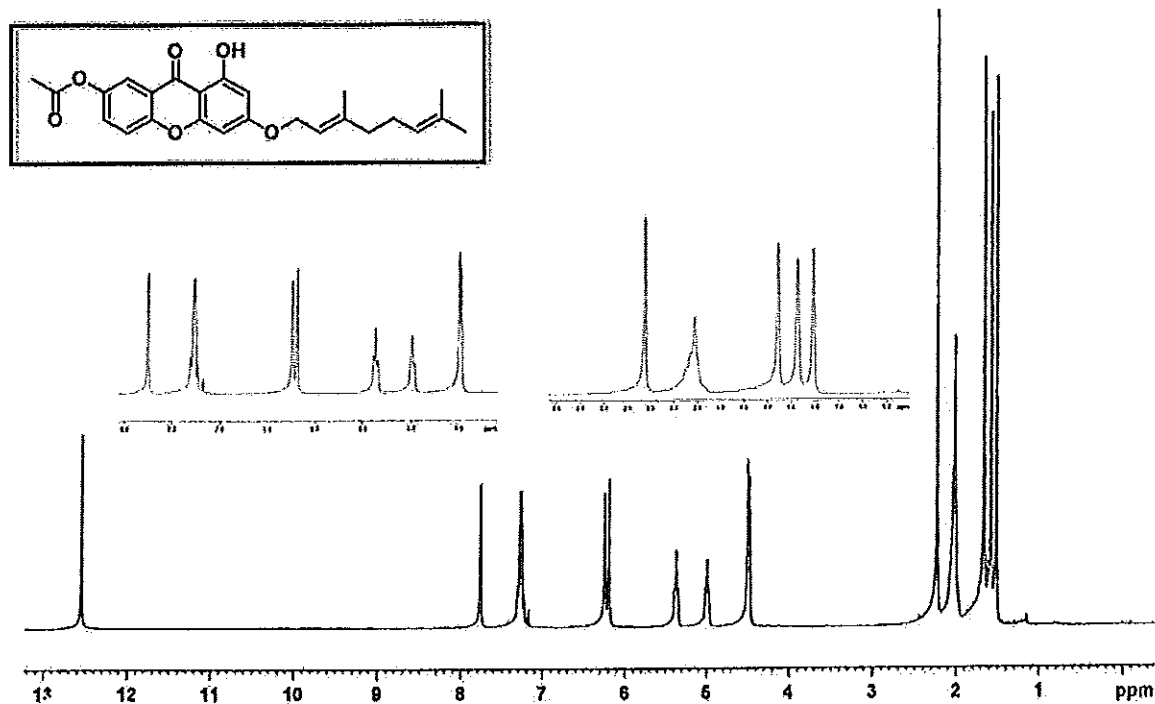
Figure 117 ^{1}H NMR (300 MHz, CDCl$_3$) spectrum of **CC18**
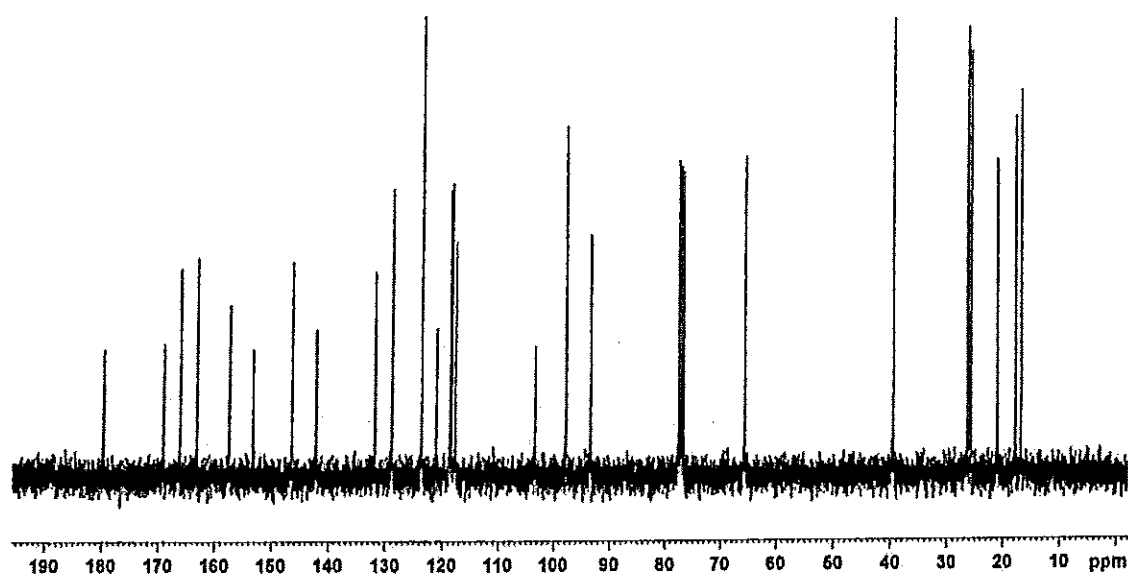
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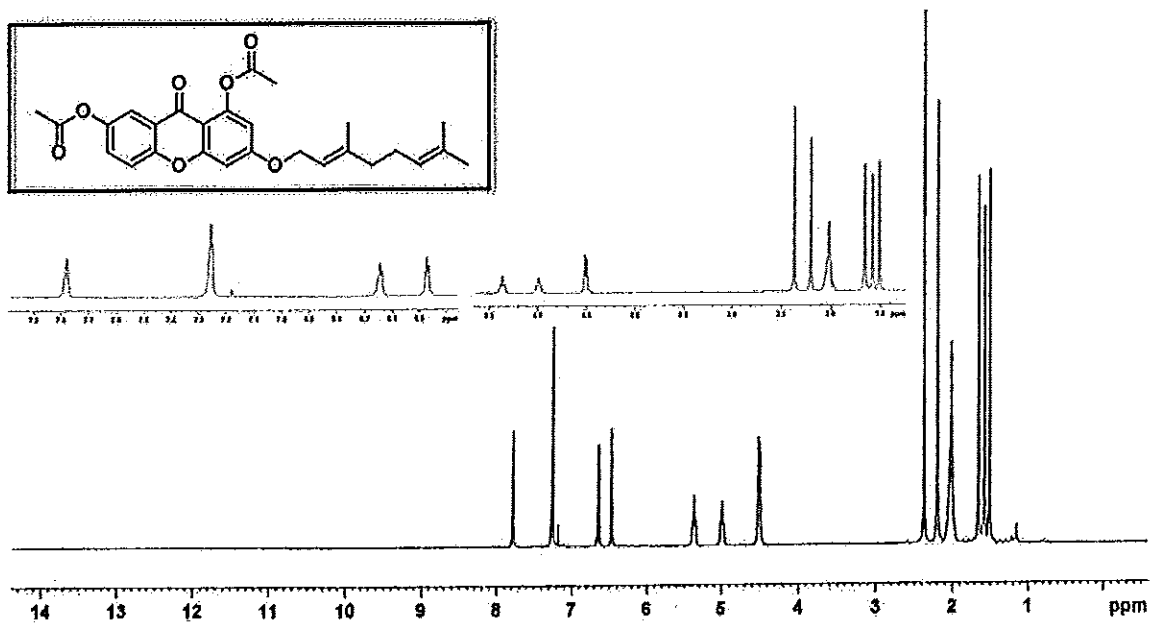
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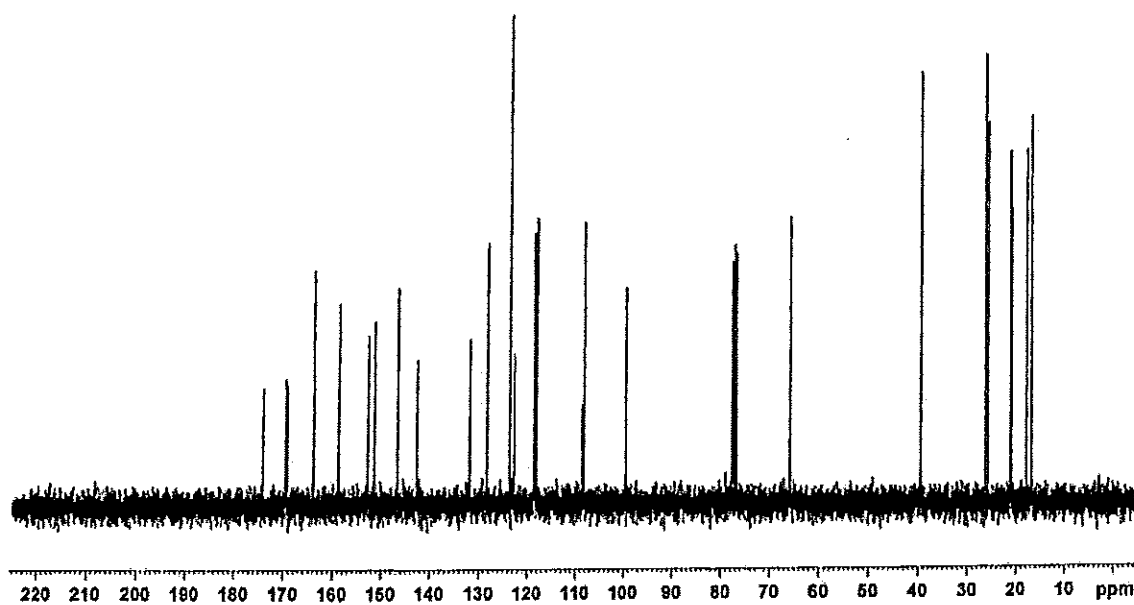
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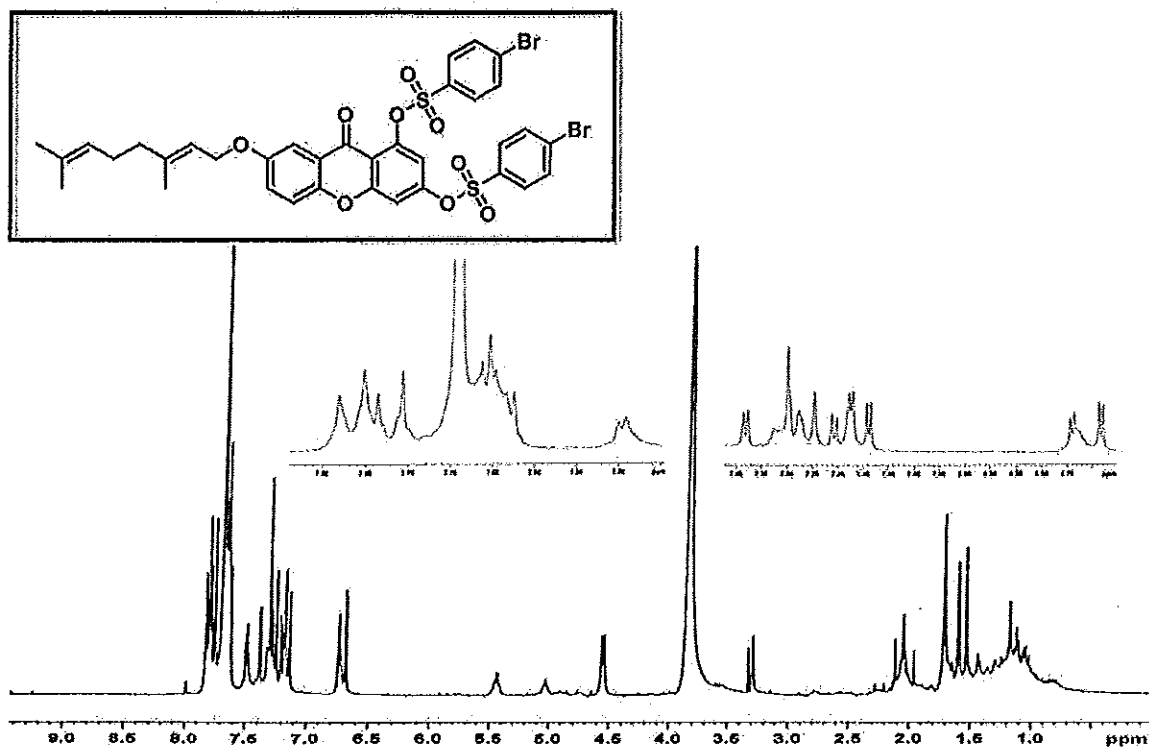
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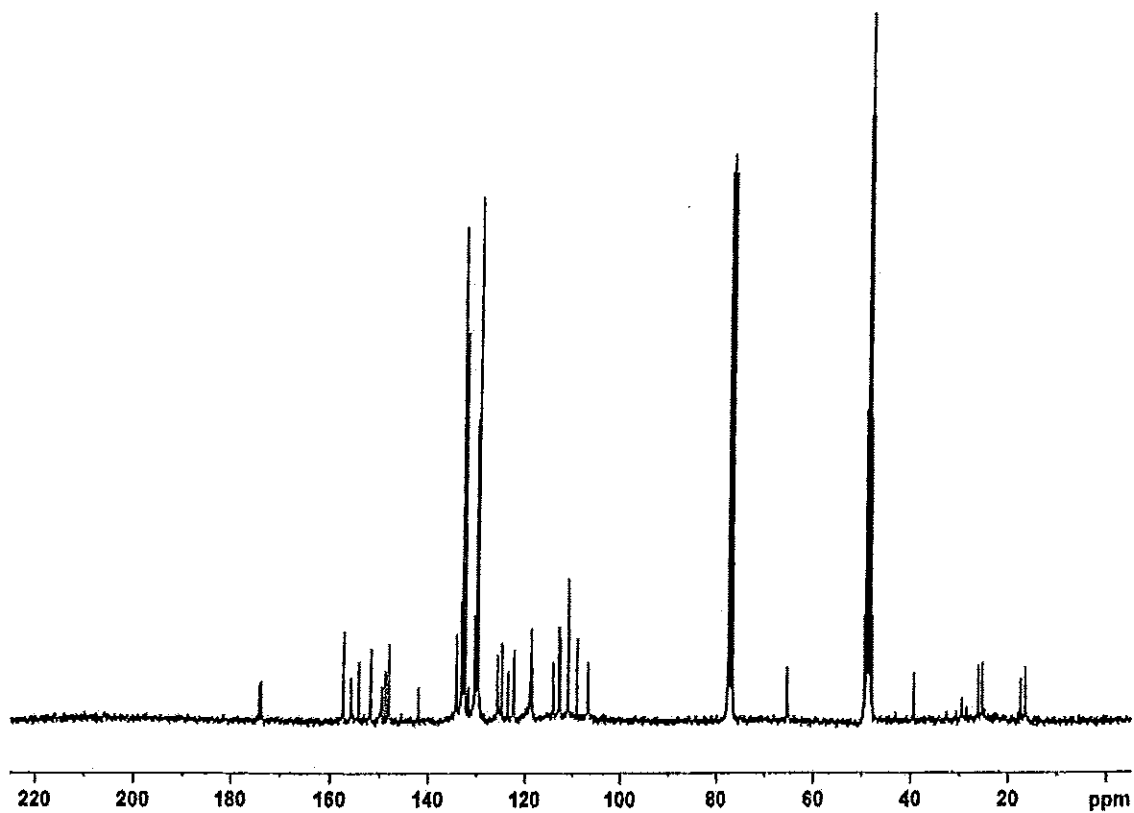
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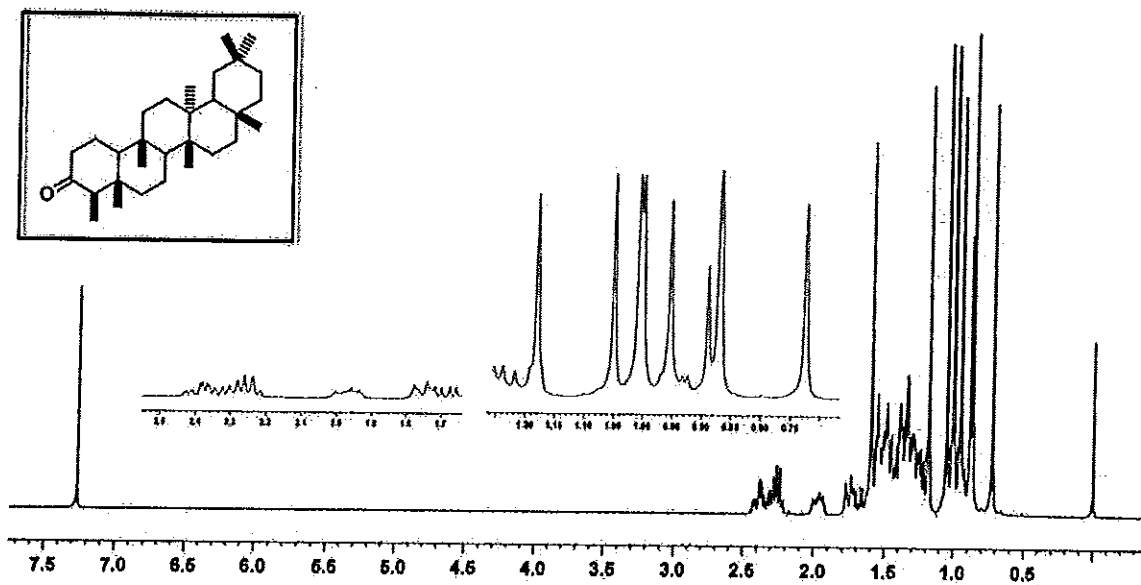
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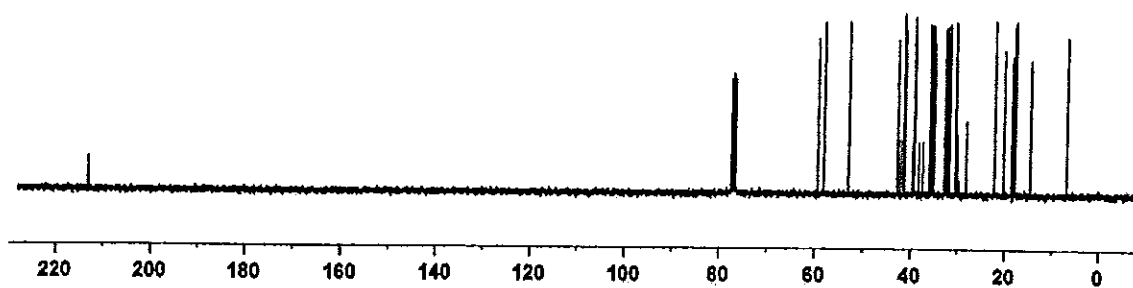
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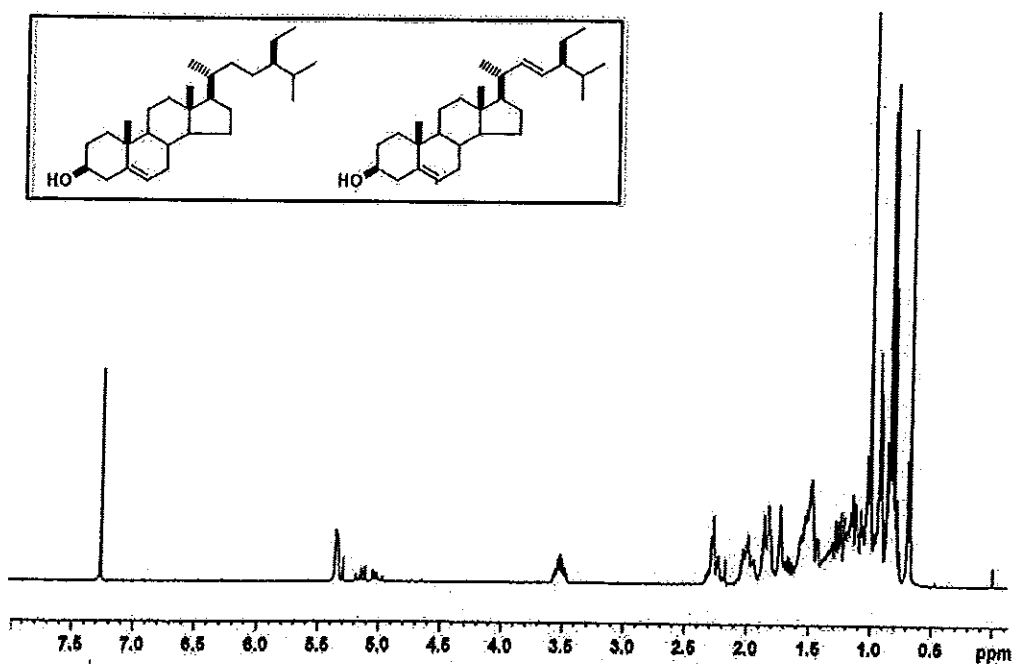
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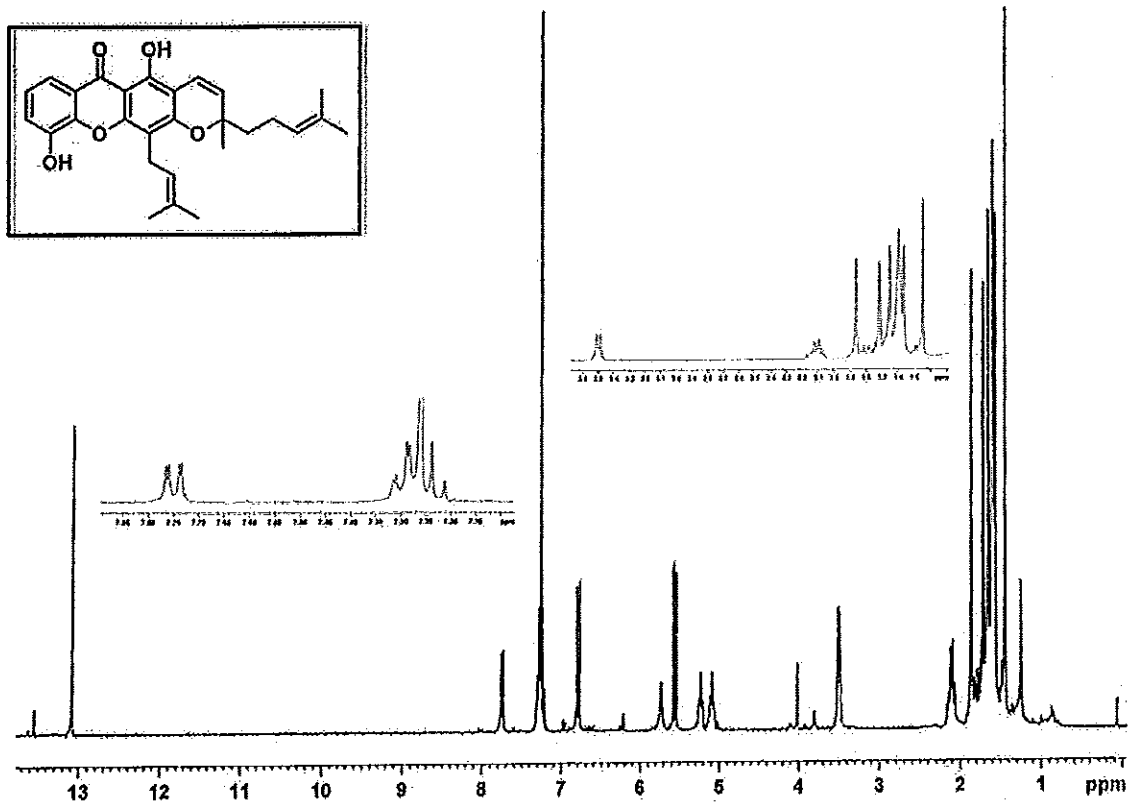
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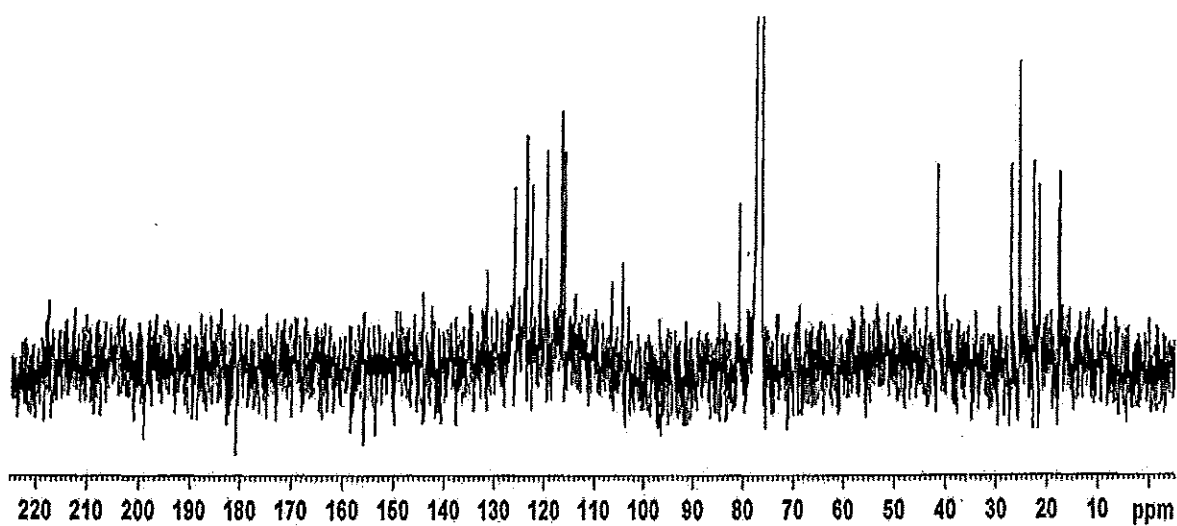
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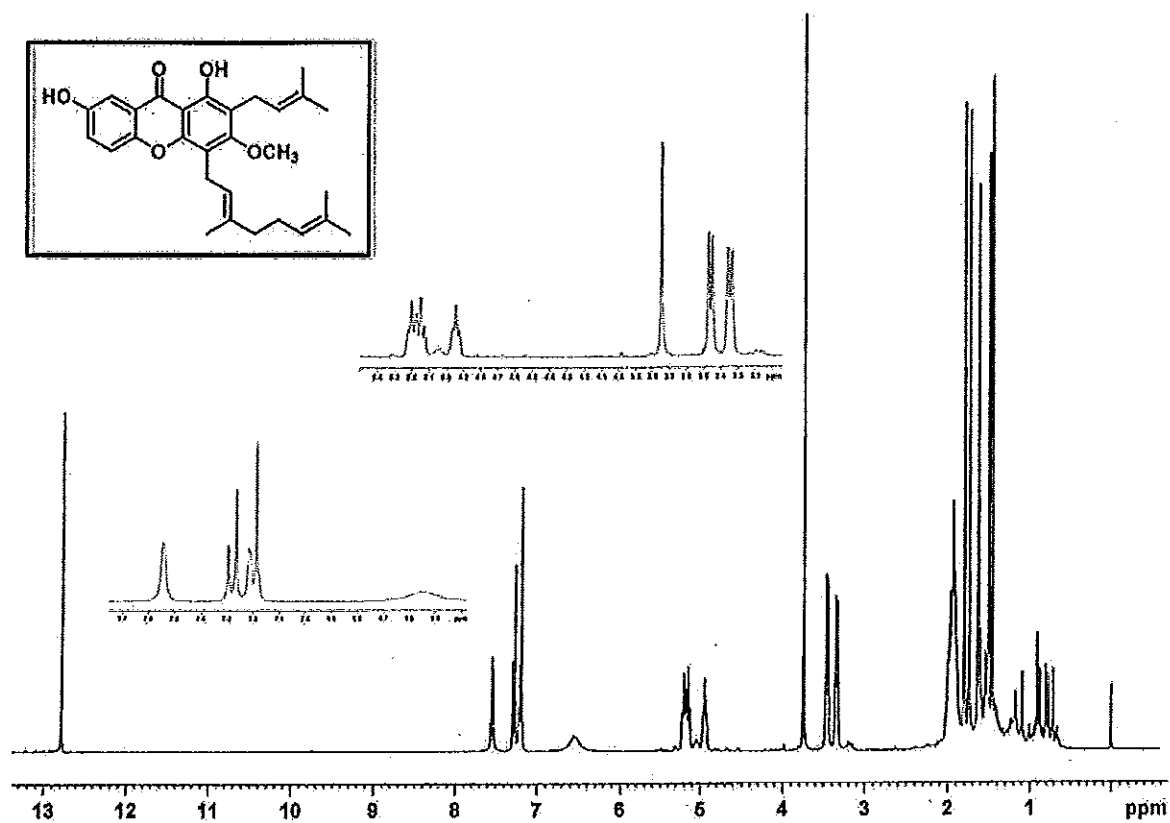
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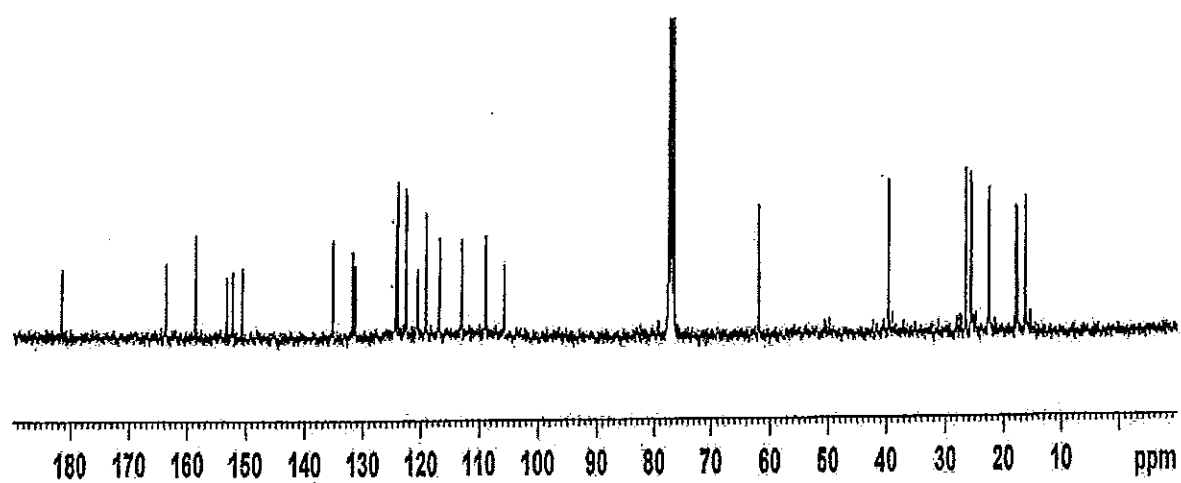
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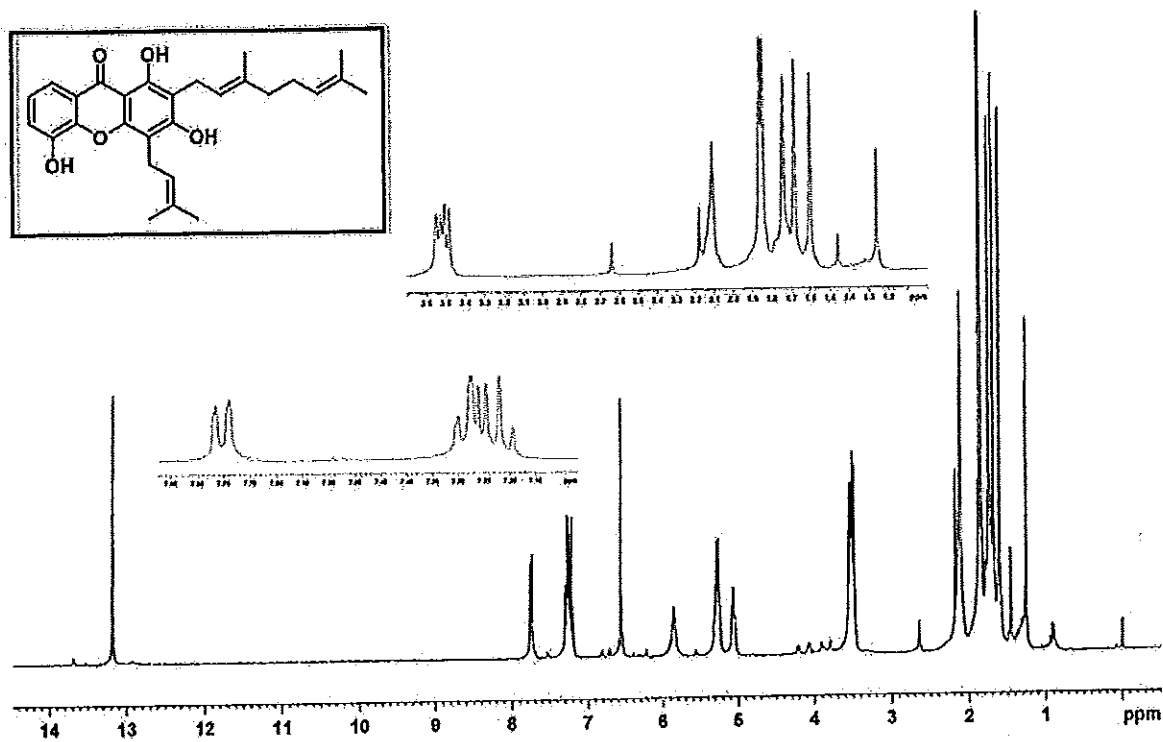
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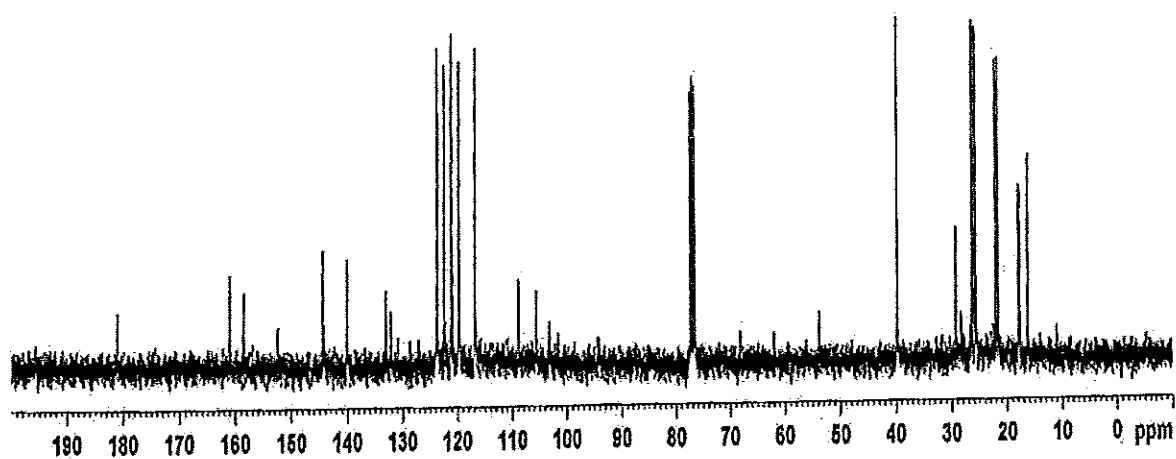
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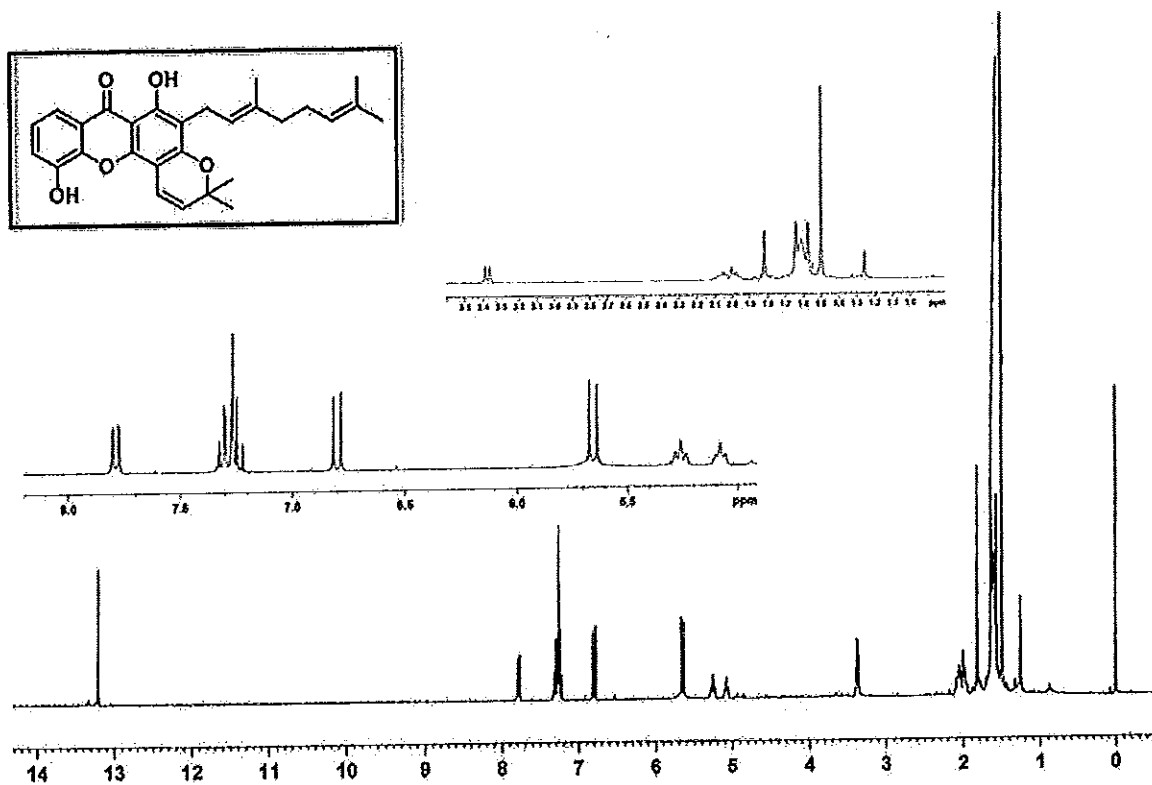
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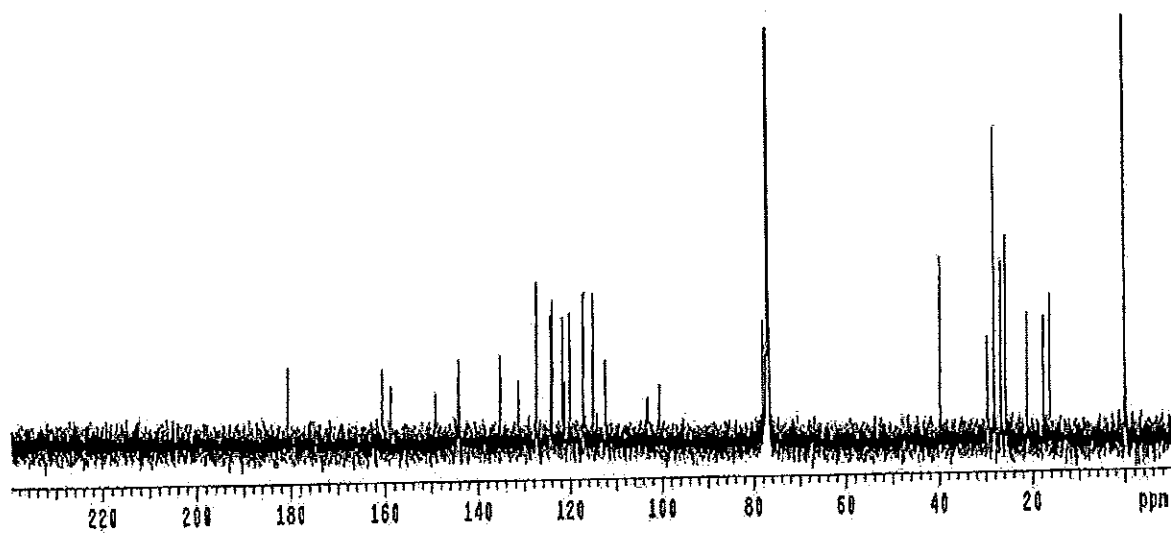
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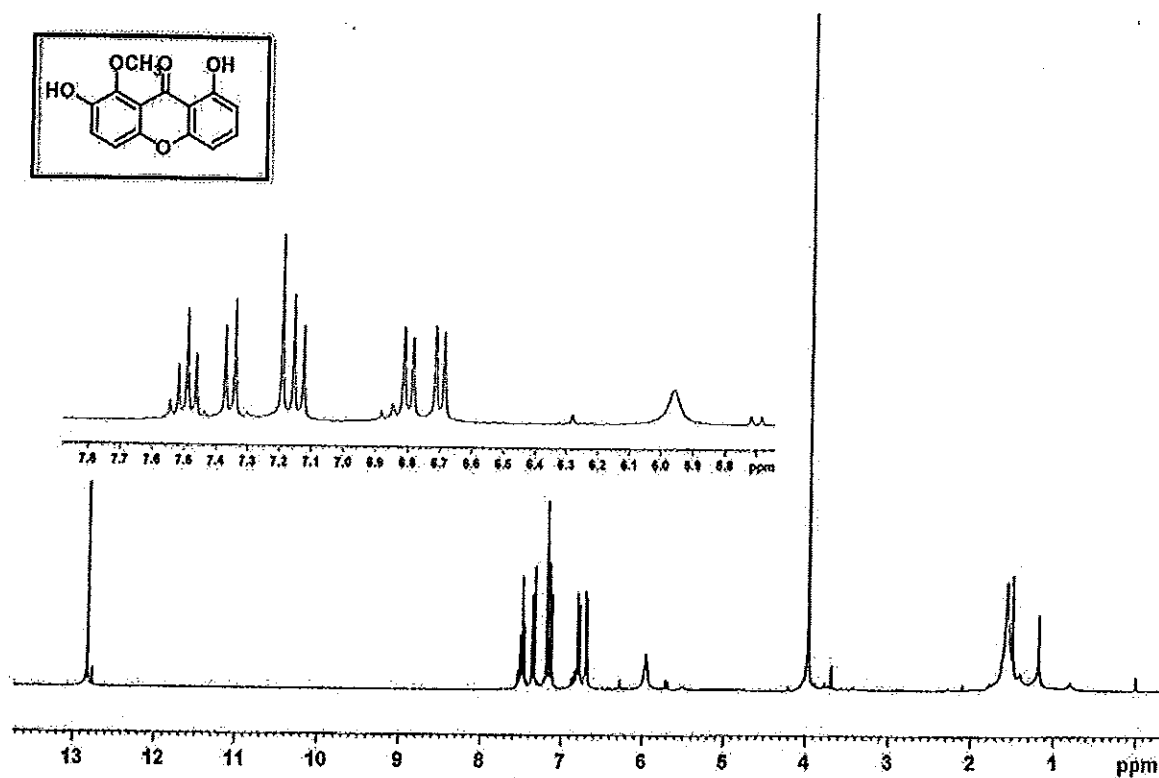
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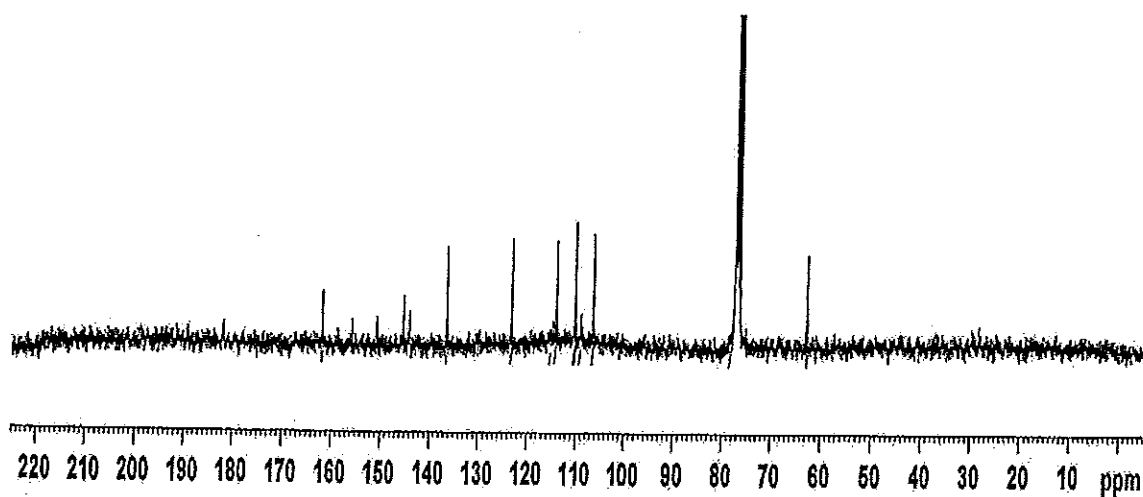
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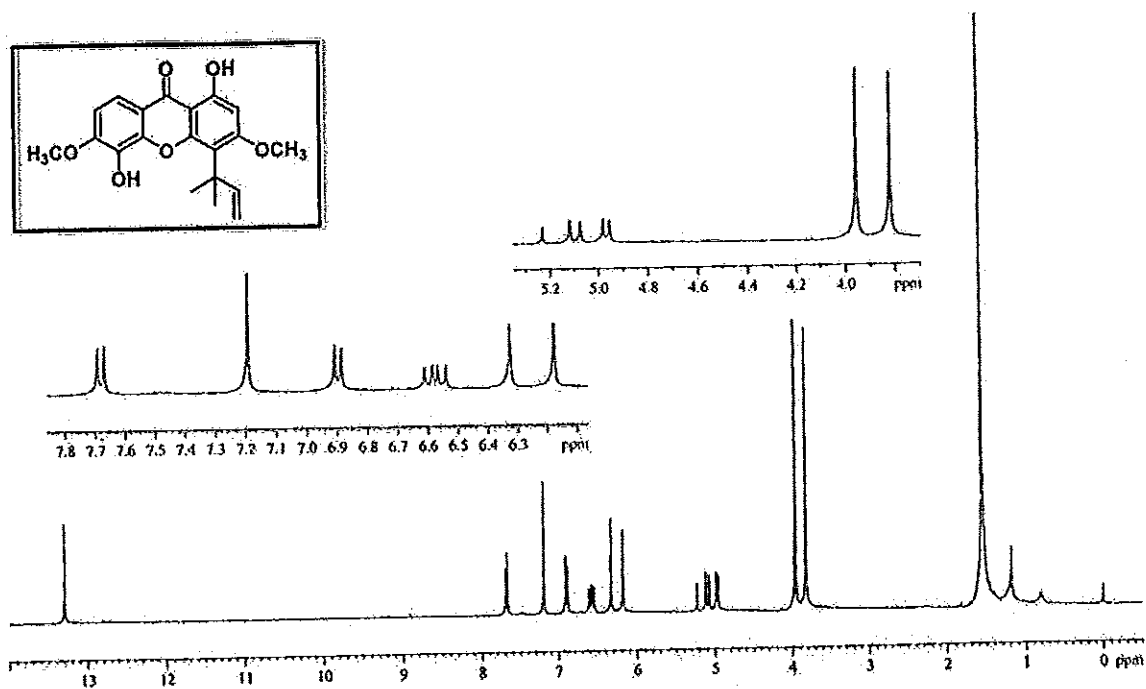
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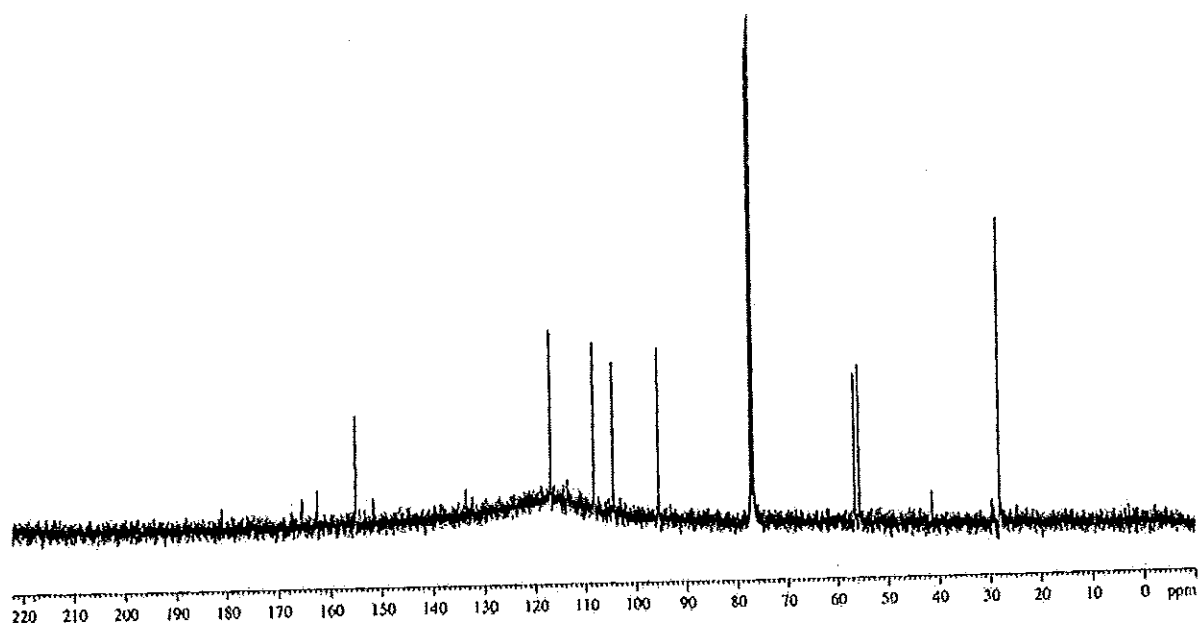
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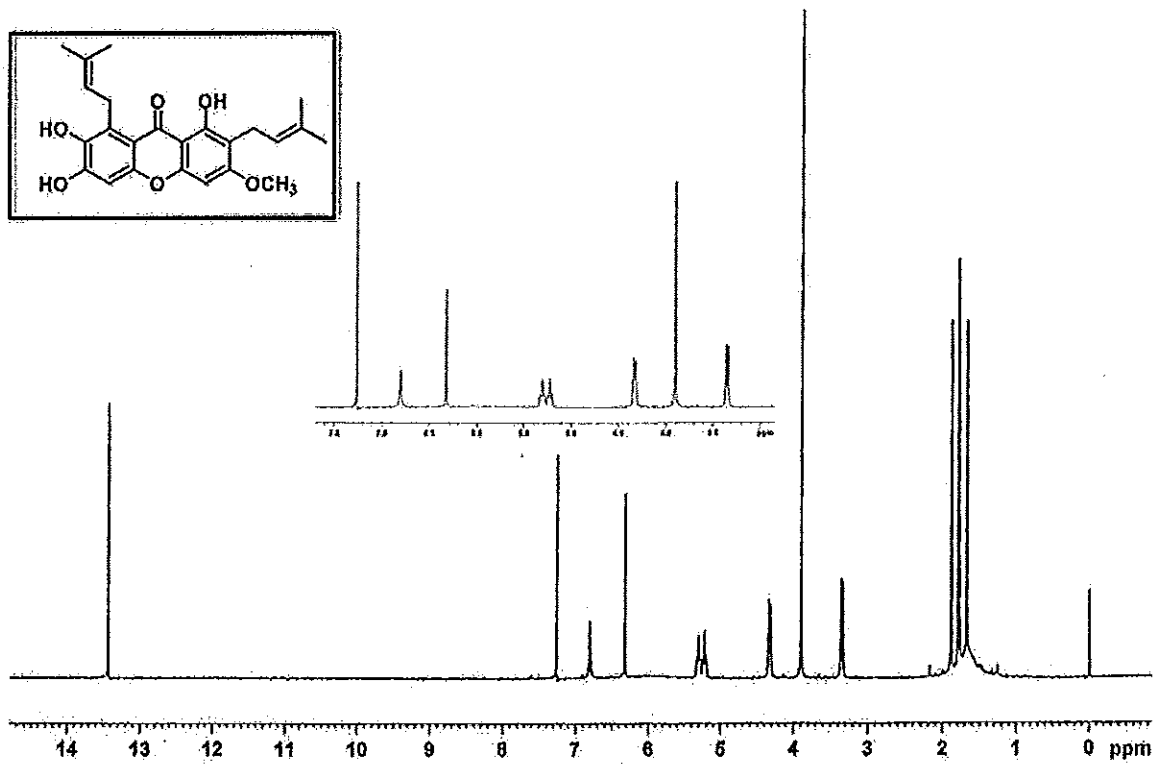
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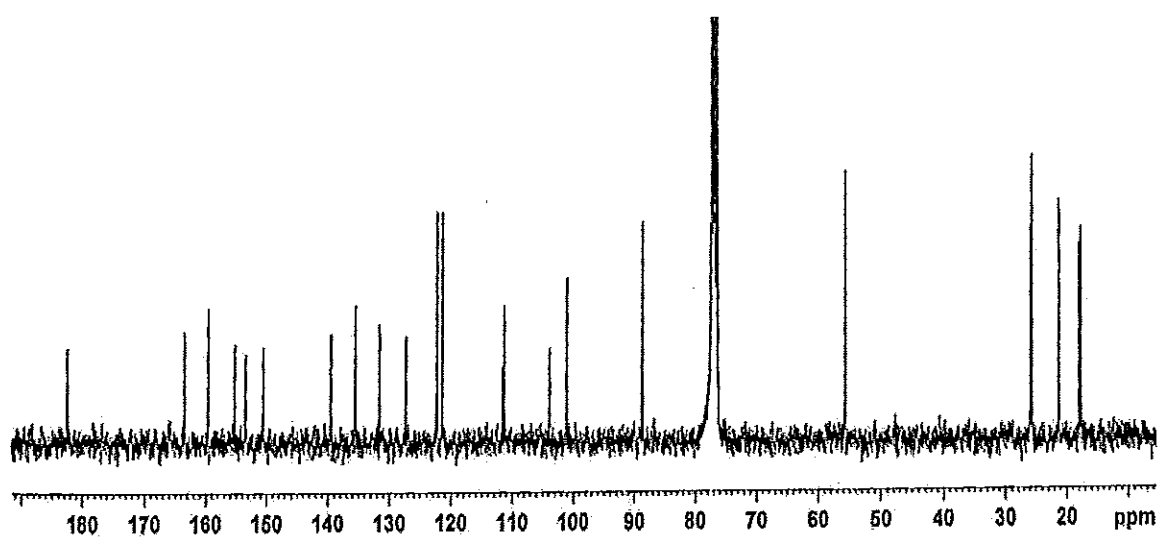
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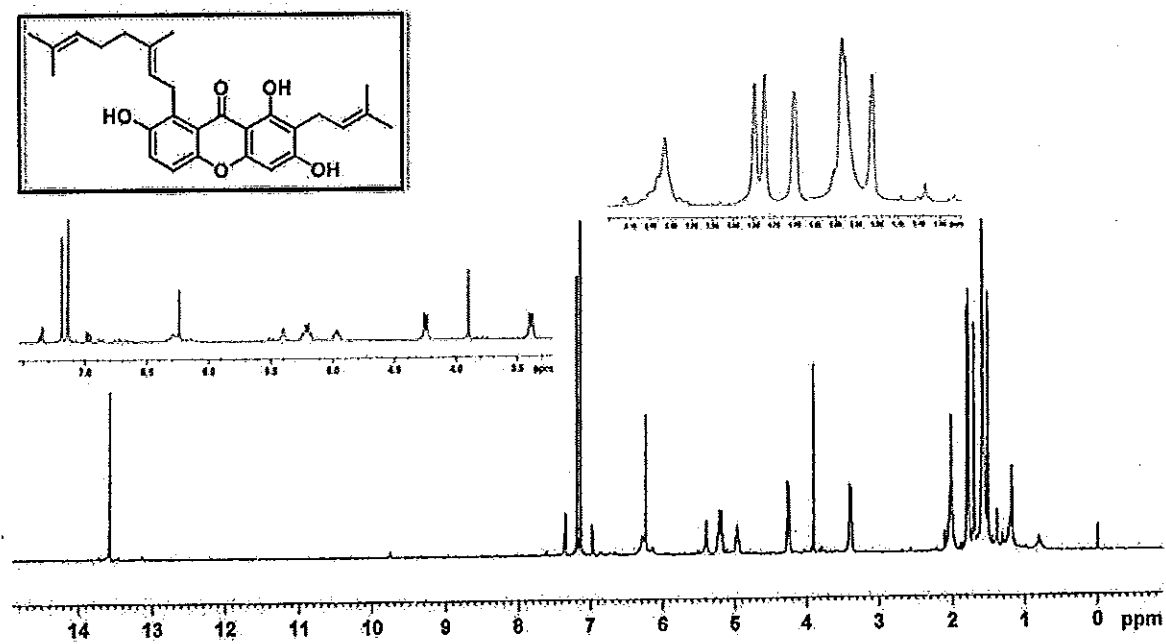
Figure 140 ^{1}H NMR (300 MHz, CDCl$_3$) spectrum of **CP8**
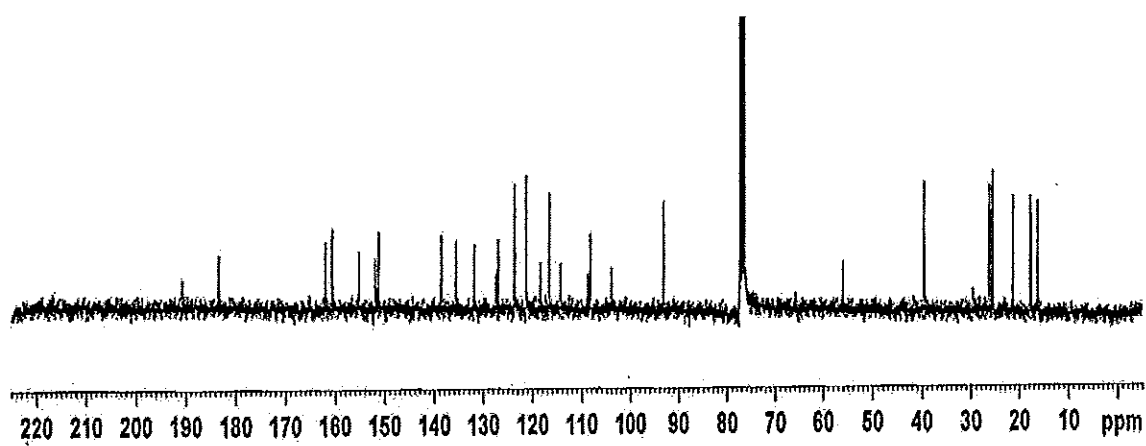
Figure 141 ^{13}C NMR (75 MHz, CDCl$_3$) spectrum of **CP8**
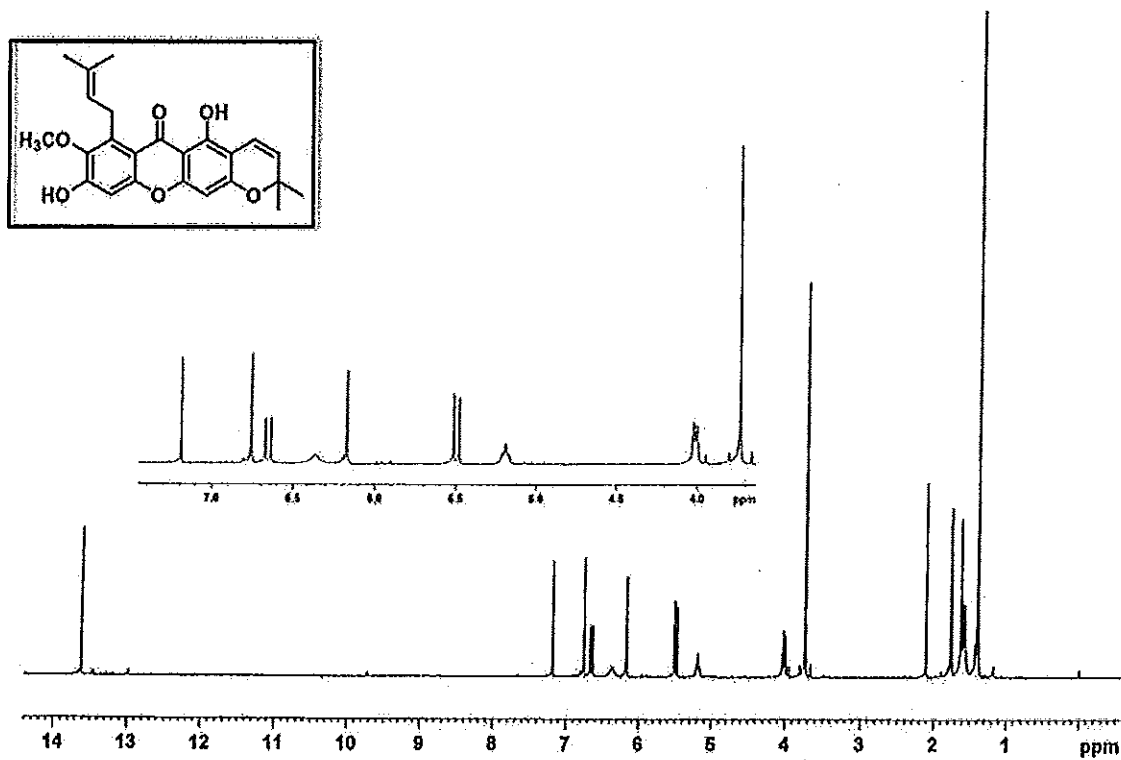
Figure 142 ^{1}H NMR (300 MHz, CDCl$_3$) spectrum of CP9
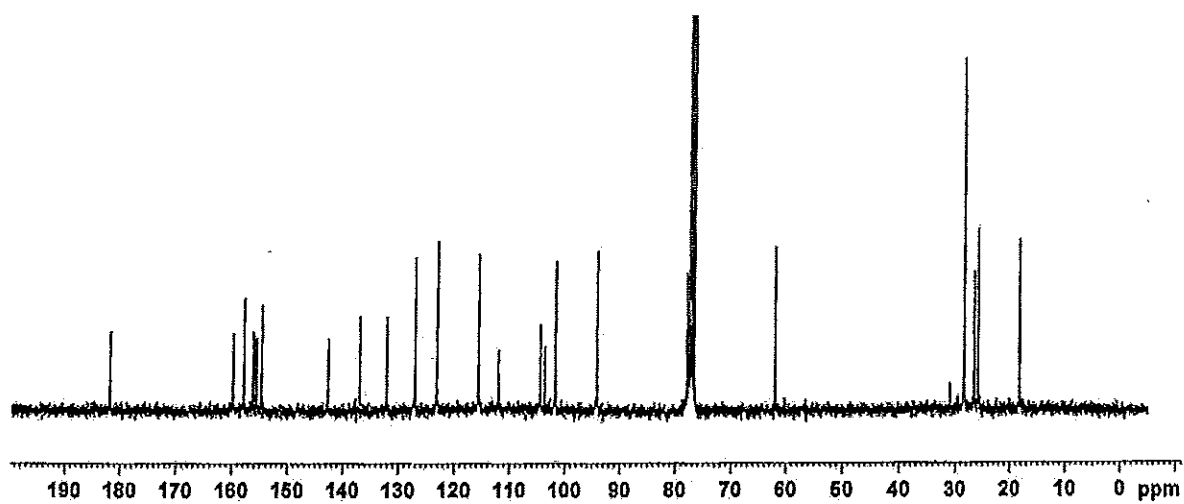
Figure 143 ^{13}C NMR (75 MHz, CDCl$_3$) spectrum of CP9
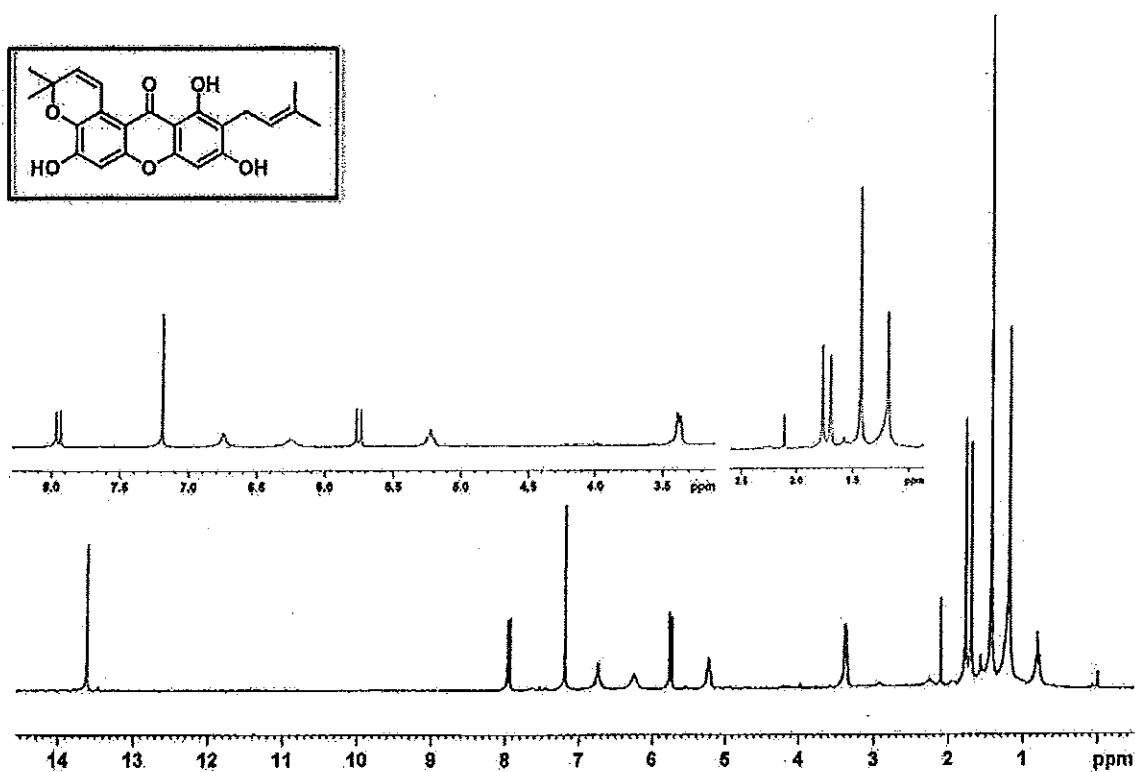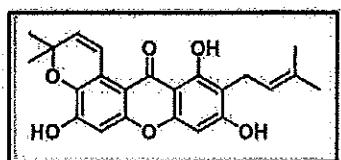
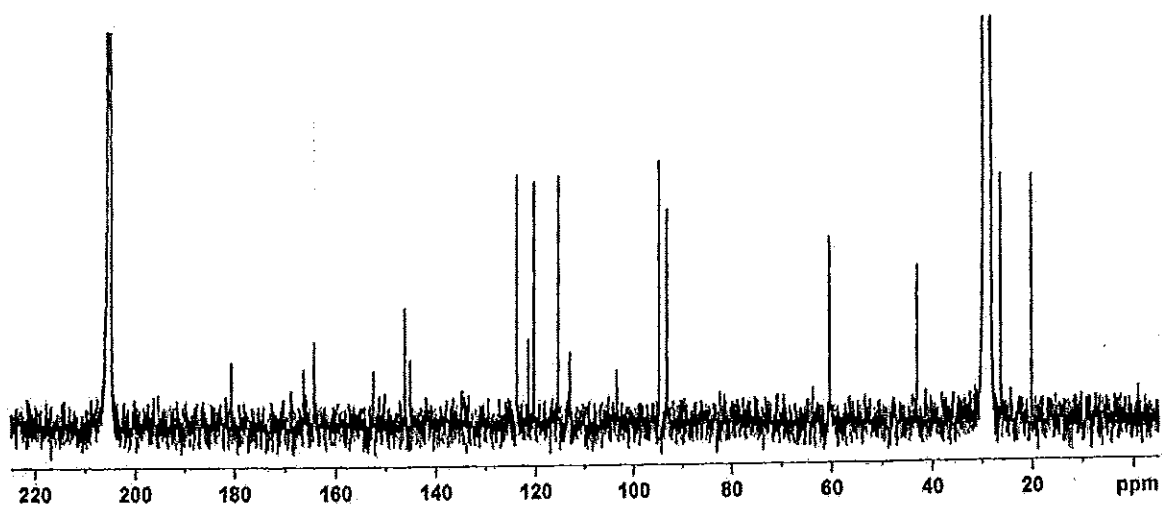**Figure 144** ^{1}H NMR (300 MHz, CDCl$_3$) spectrum of **CP10**
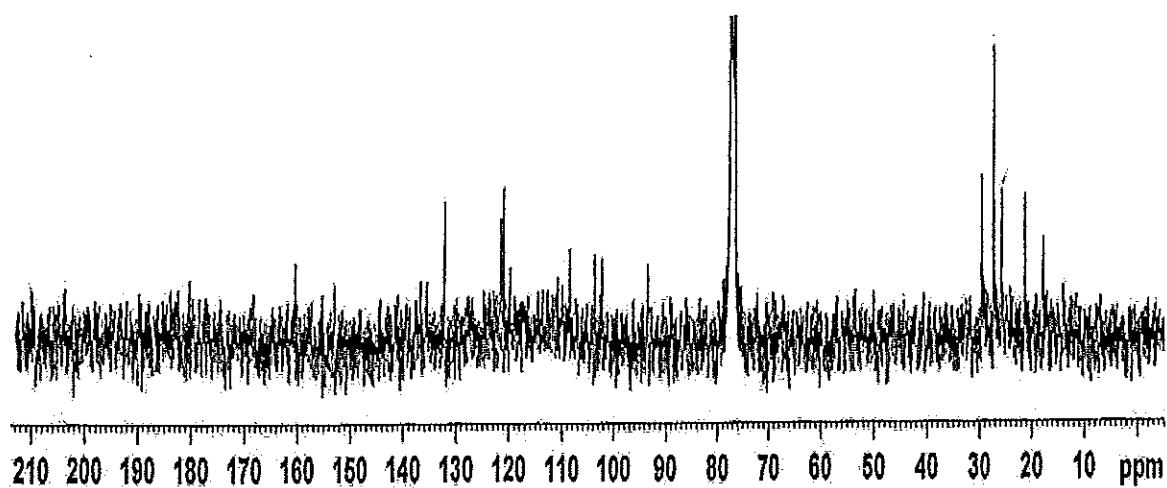
Figure 145 ^{13}C NMR (75 MHz, CDCl$_3$) spectrum of **CP10**
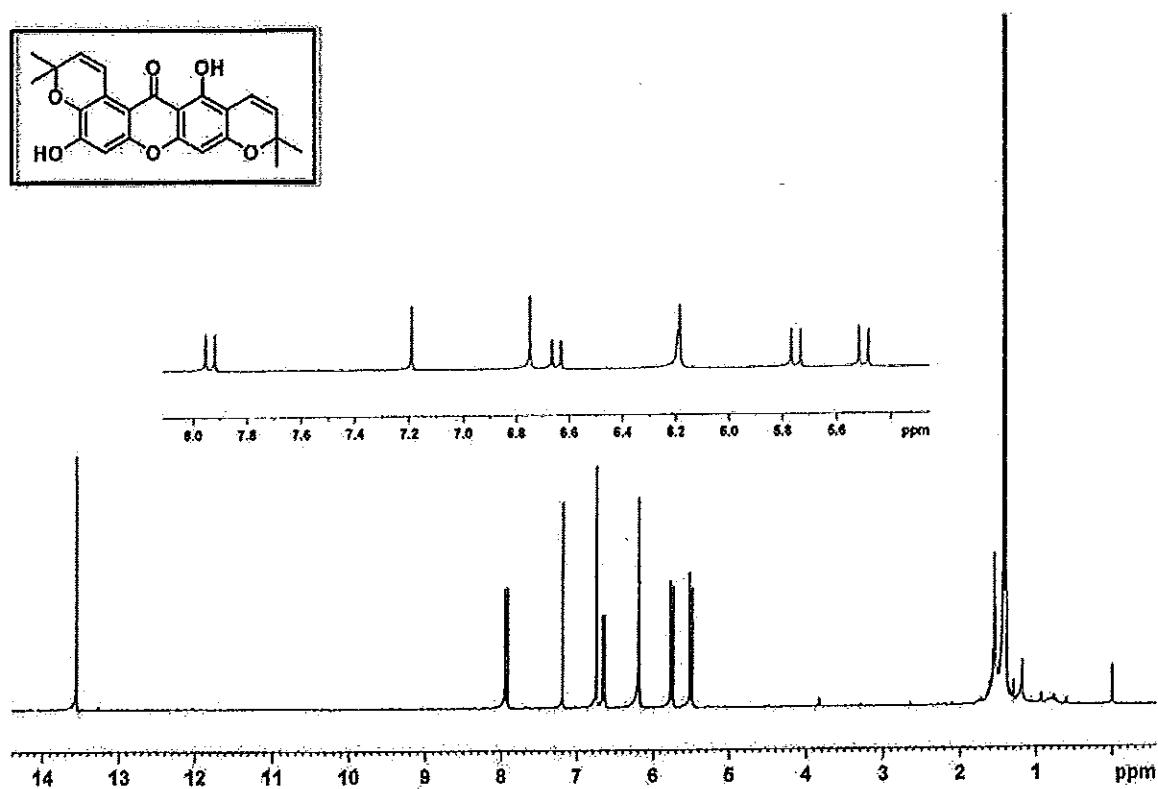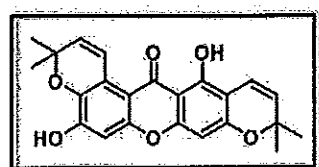
Figure 146 ^{1}H NMR (300 MHz, CDCl$_3$) spectrum of **CP11**
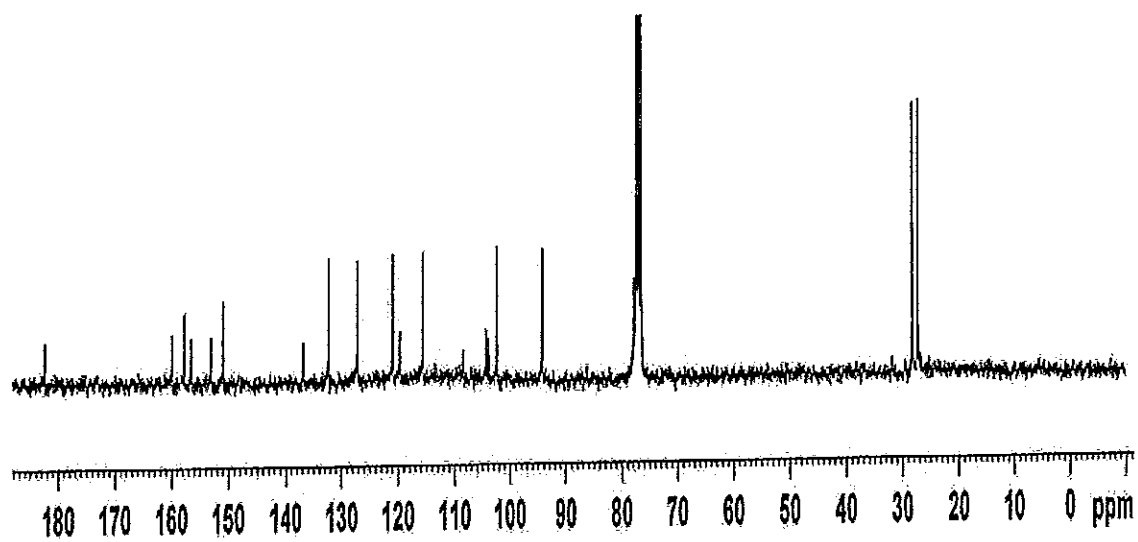
Figure 147 ^{13}C NMR (75 MHz, CDCl$_3$) spectrum of **CP11**
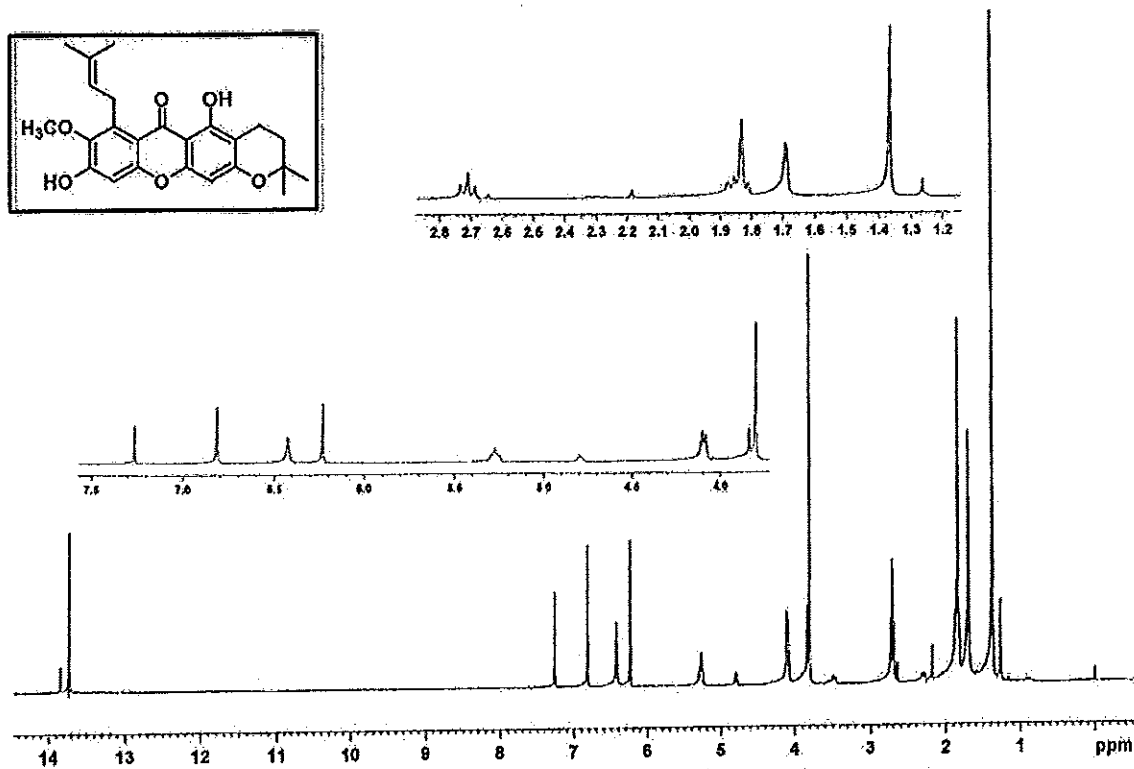
Figure 148 ^{1}H NMR (300 MHz, CDCl$_3$) spectrum of **CP12**
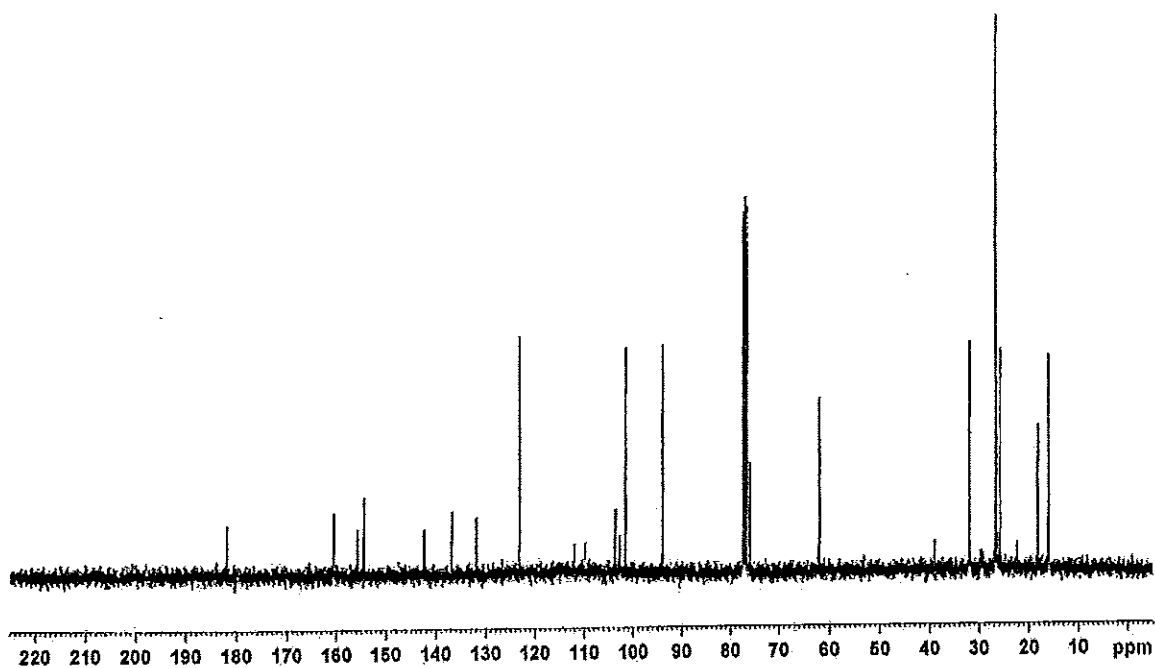
Figure 149 ^{13}C NMR (75 MHz, CDCl$_3$) spectrum of **CP12**
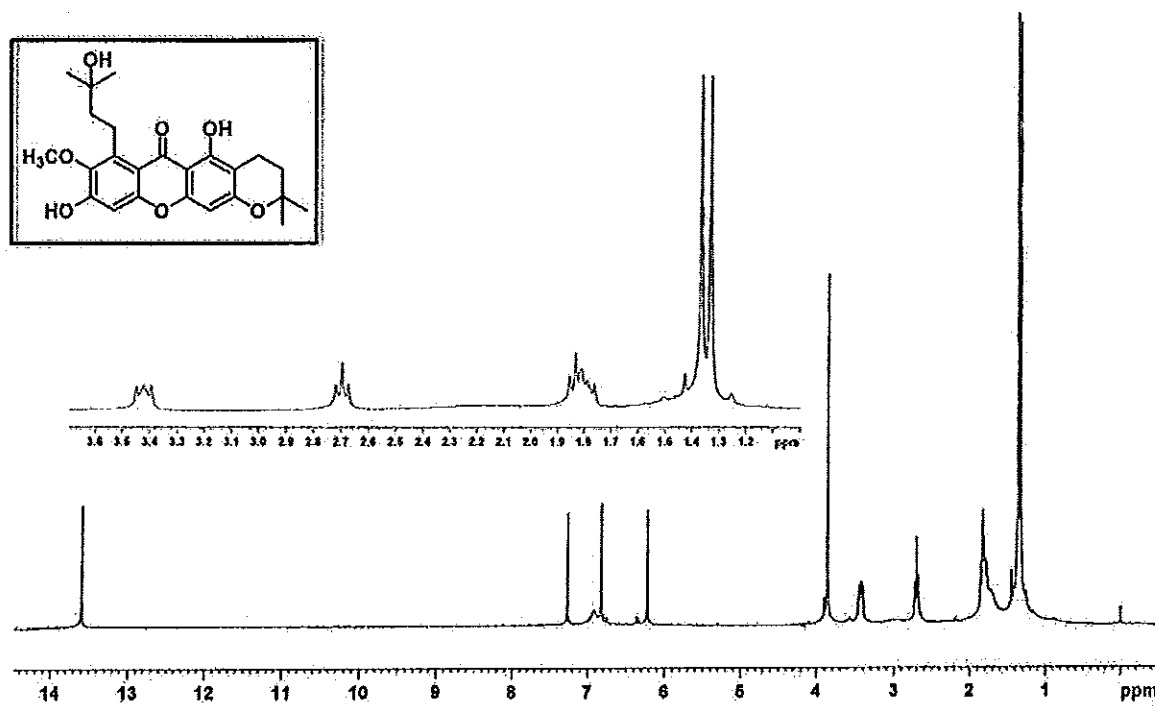
Figure 150 ^{1}H NMR (300 MHz, CDCl$_3$) spectrum of **CP13**
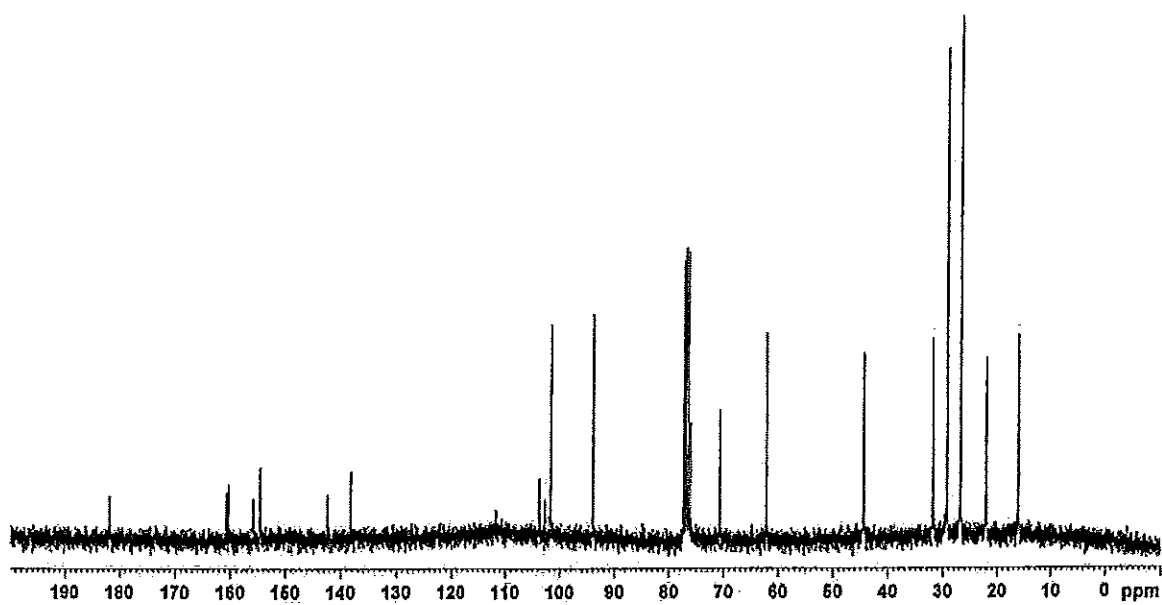
Figure 151 ^{13}C NMR (75 MHz, CDCl$_3$) spectrum of **CP13**
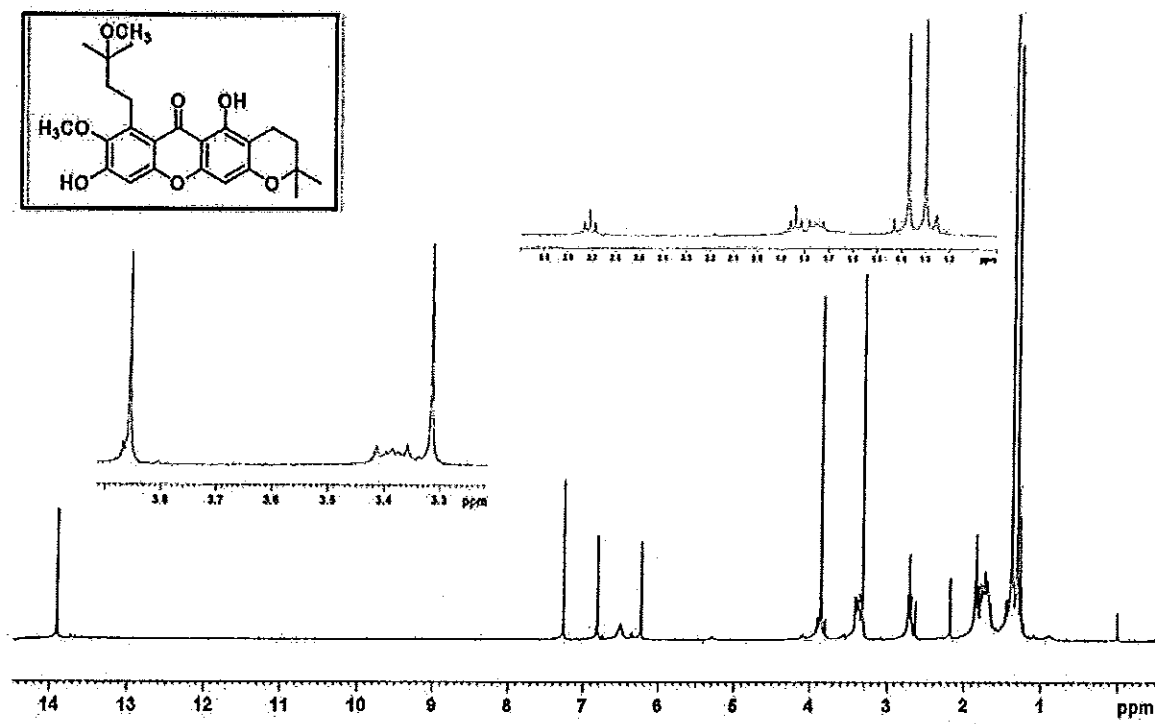
Figure 152 ^{1}H NMR (300 MHz, CDCl$_3$) spectrum of **CP14**
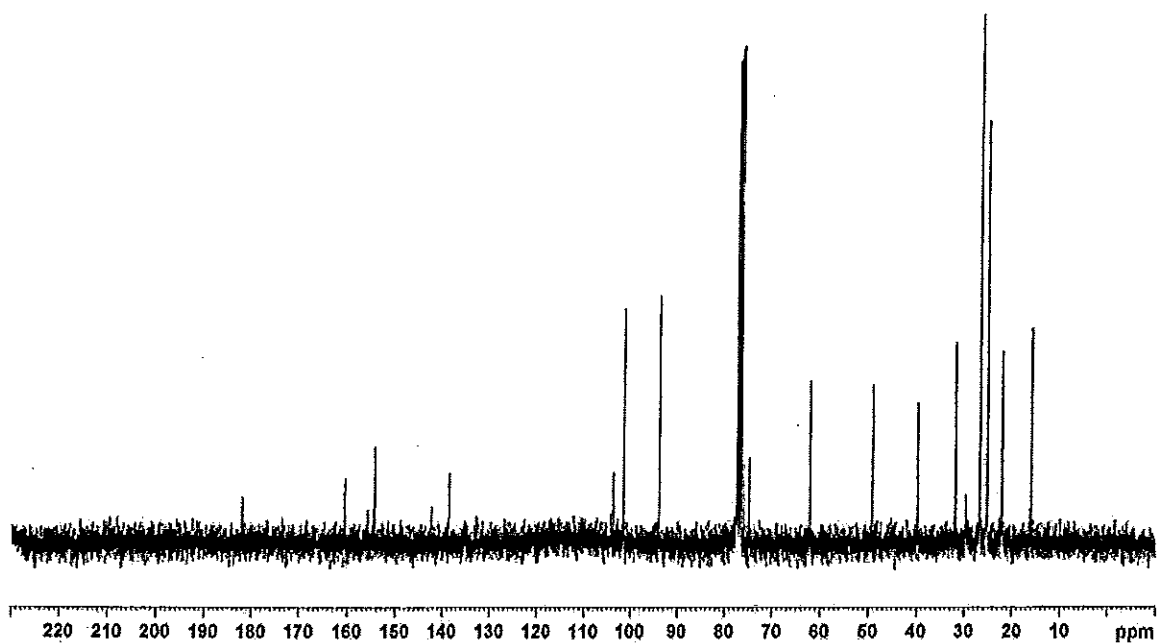
Figure 153 ^{13}C NMR (75 MHz, CDCl$_3$) spectrum of **CP14**
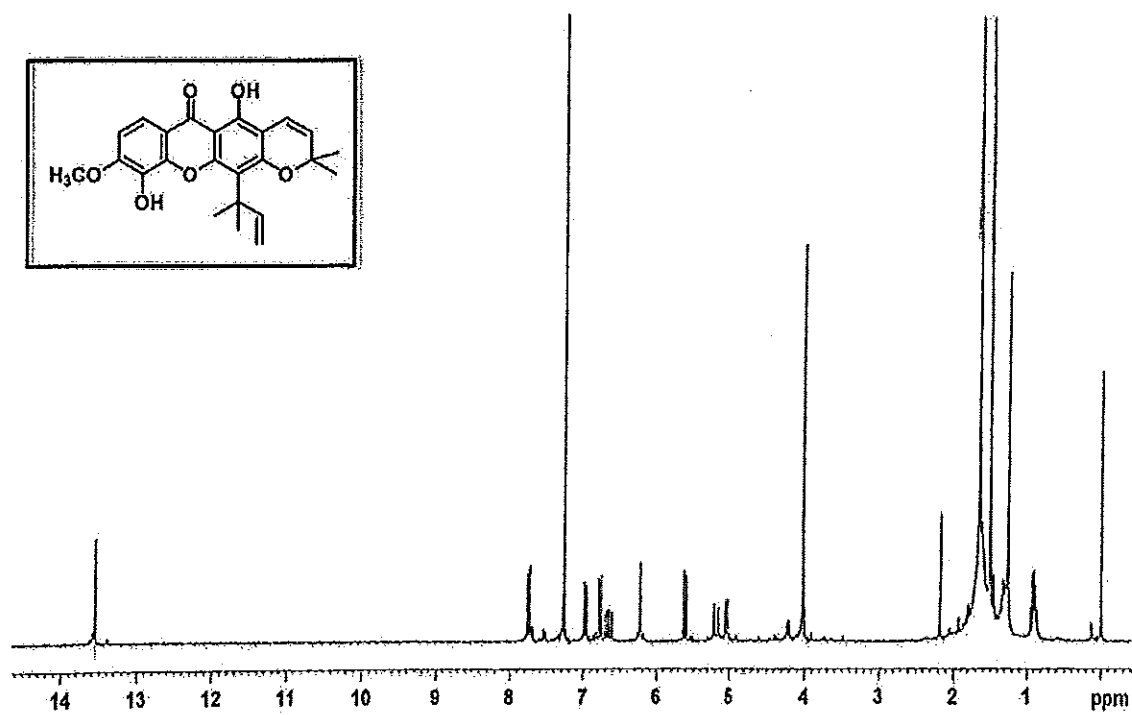
Figure 154 ^{1}H NMR (300 MHz, CDCl$_3$) spectrum of **CP15**
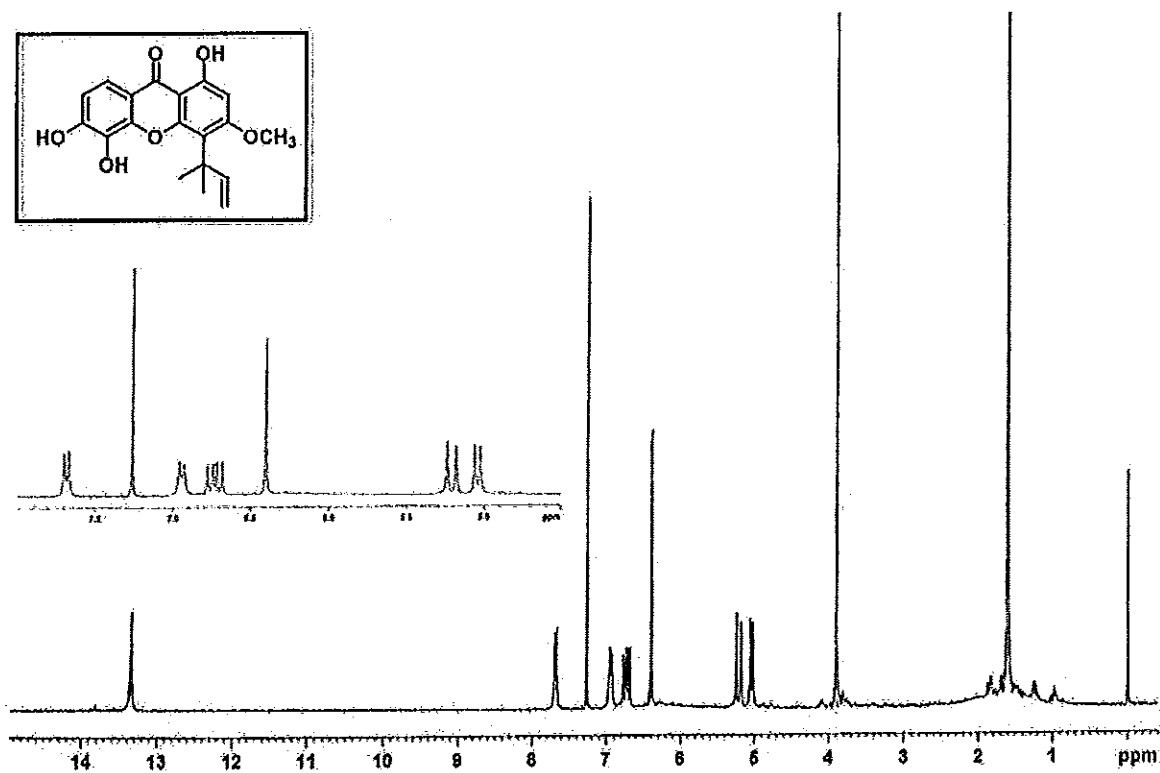
Figure 155 ^{1}H NMR (300 MHz, CDCl$_3$) spectrum of **CP16**
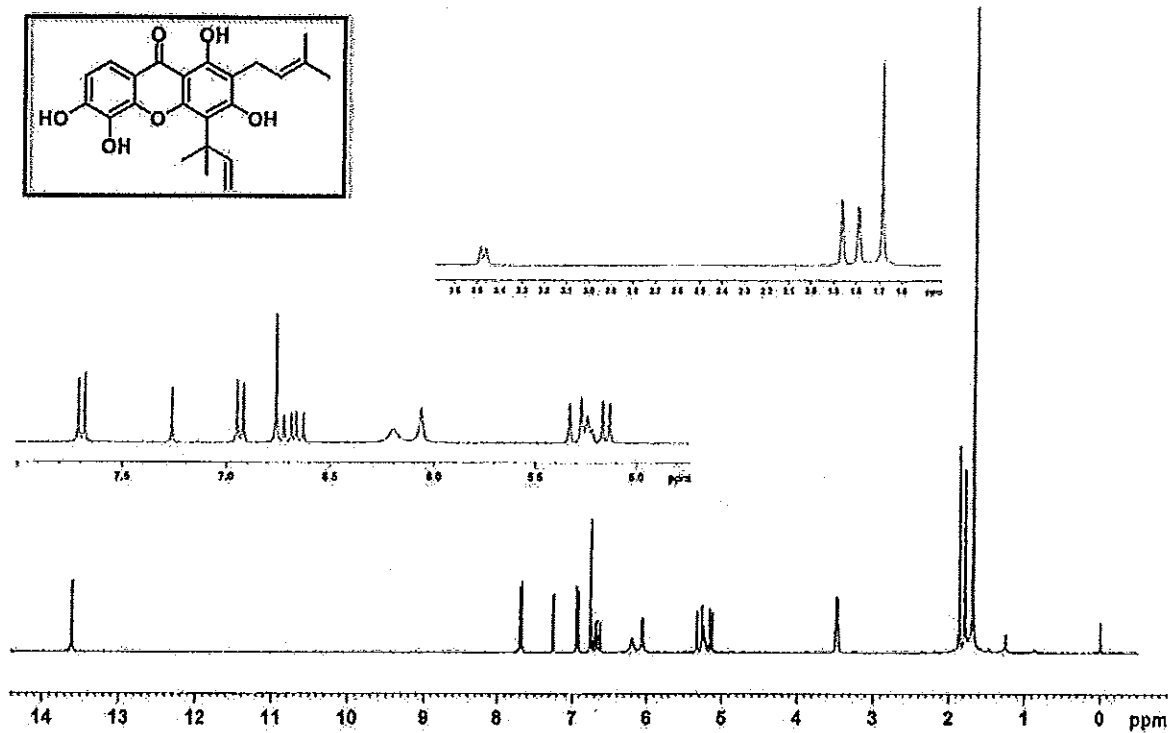
Figure 156 ^{1}H NMR (300 MHz, CDCl$_3$) spectrum of **CP17**
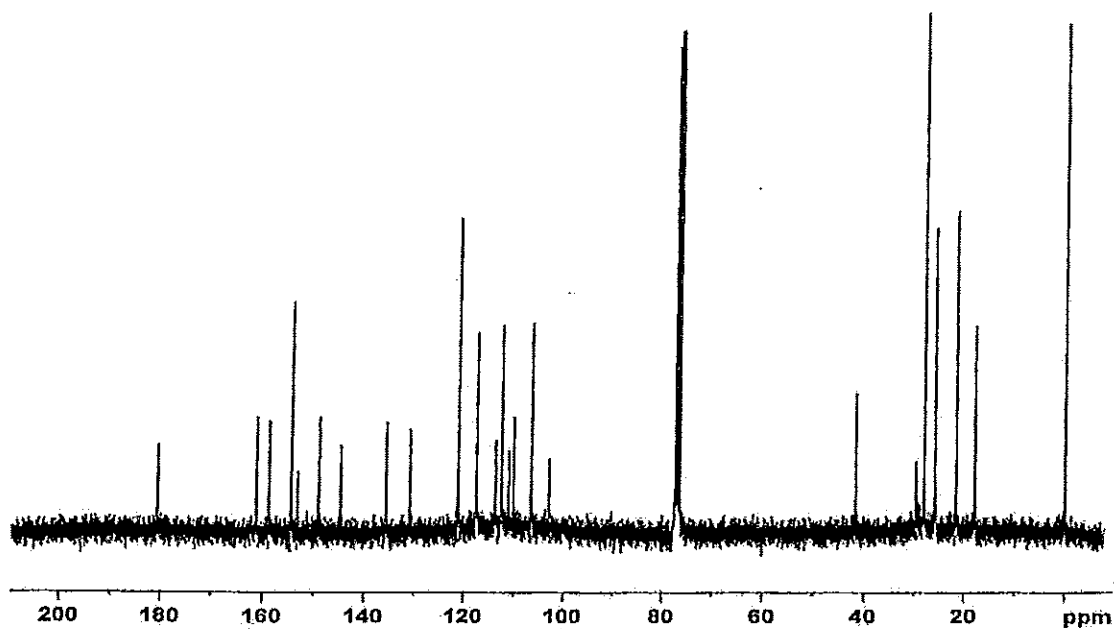
Figure 157 ^{13}C NMR (75 MHz, CDCl$_3$) spectrum of **CP17**
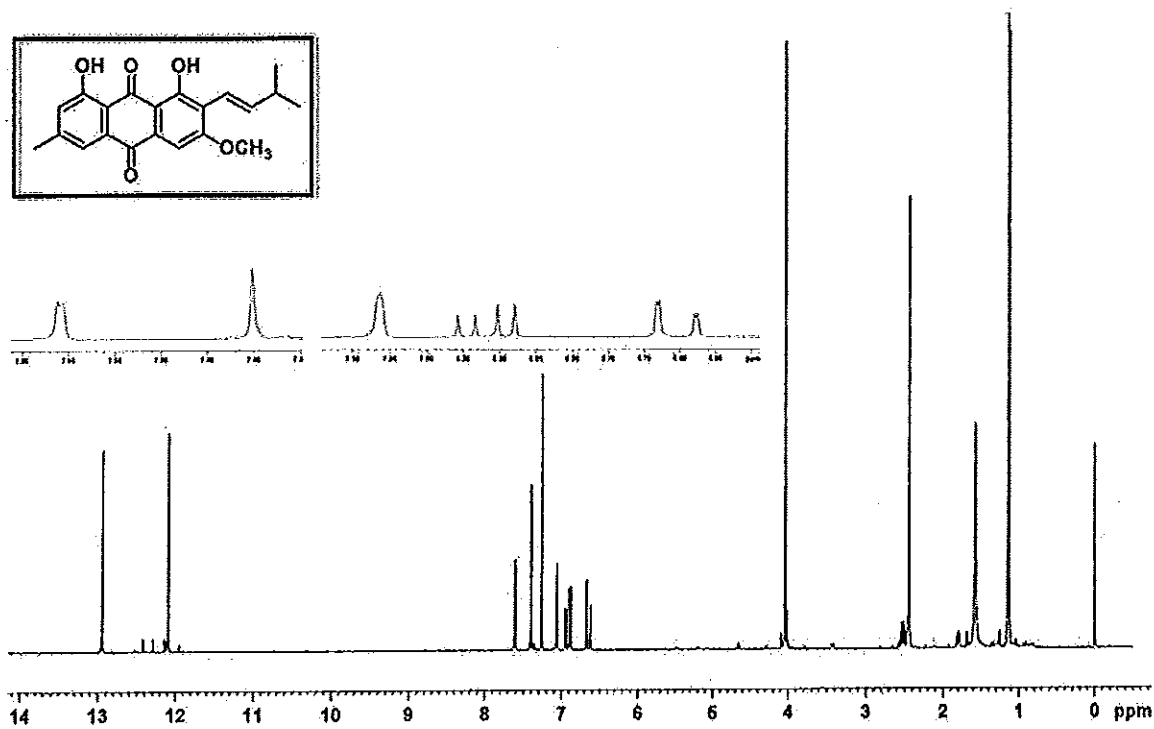
Figure 158 ^{1}H NMR (300 MHz, CDCl$_3$) spectrum of **CP18**
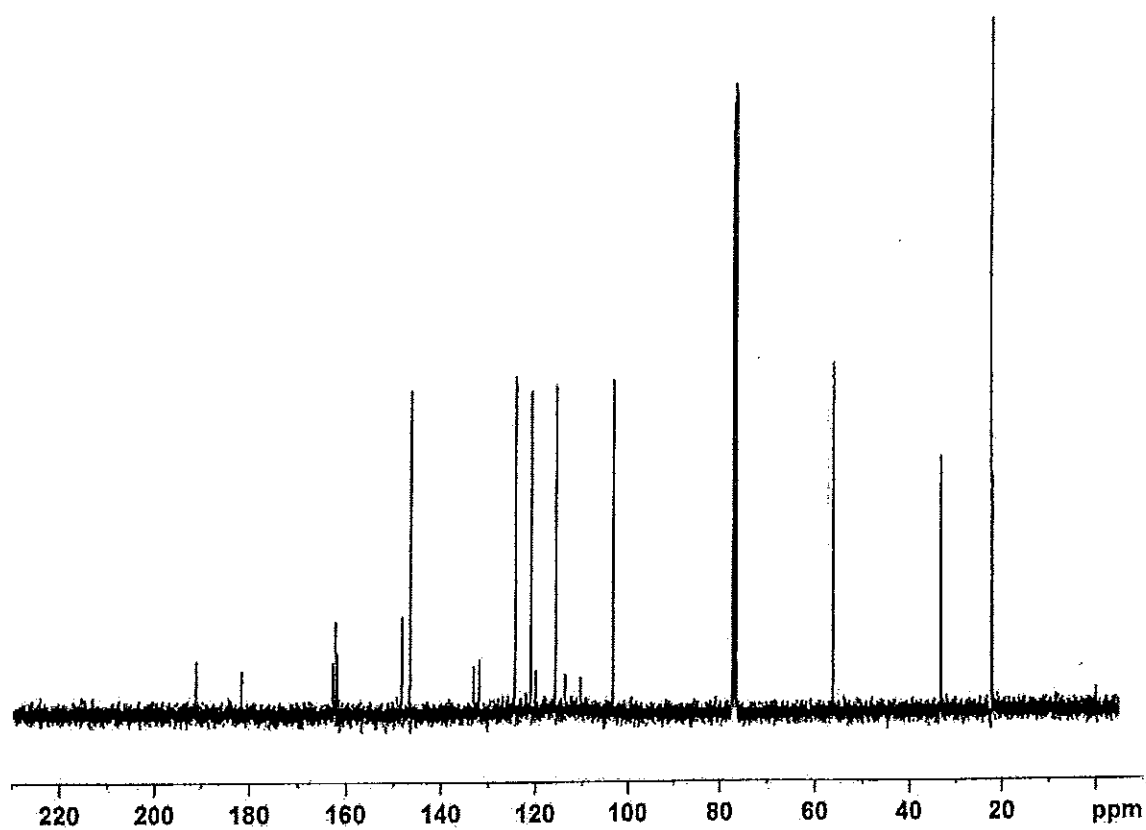
Figure 159 ^{13}C NMR (75 MHz, CDCl$_3$) spectrum of **CP18**
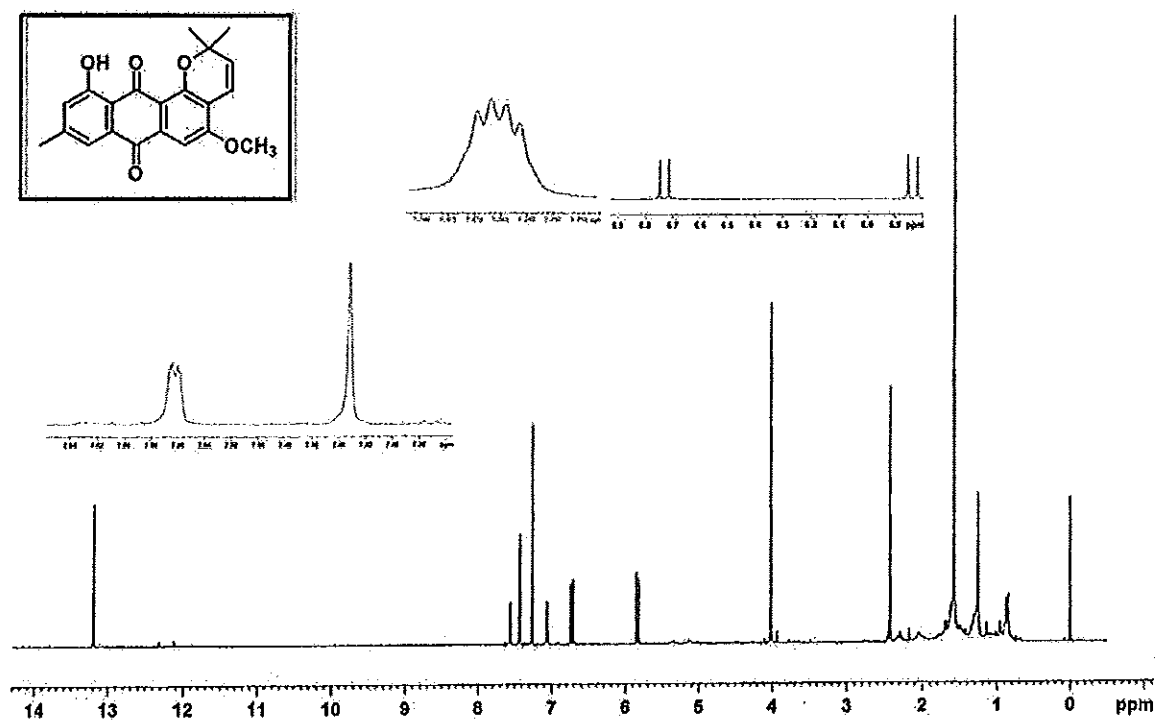
Figure 160 ^{1}H NMR (300 MHz, CDCl$_3$) spectrum of **CP19**
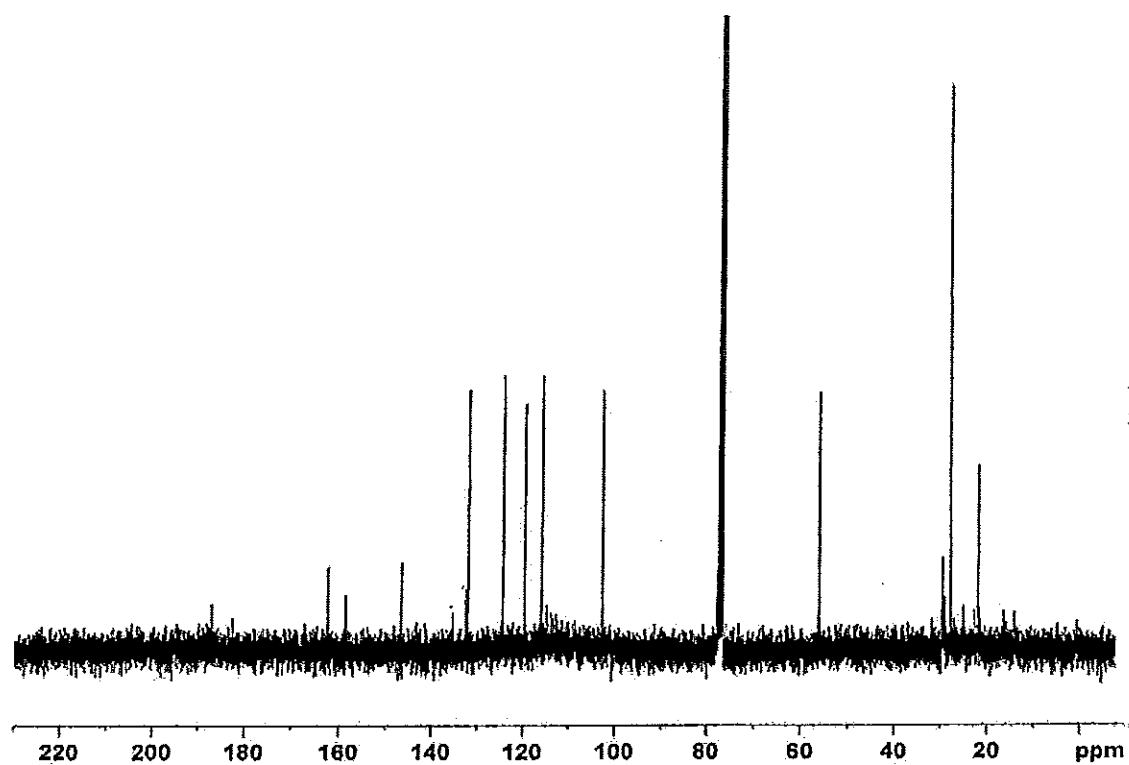
Figure 161 ^{13}C NMR (75 MHz, CDCl$_3$) spectrum of **CP19**
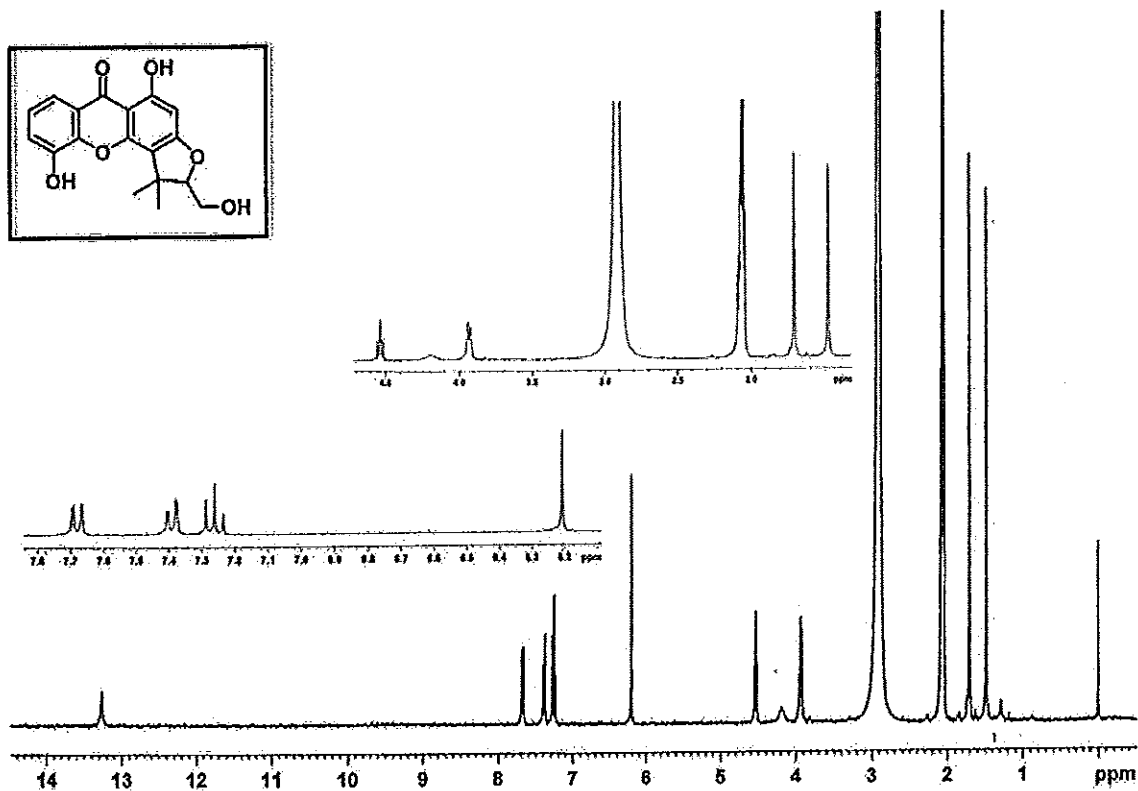
Figure 162 ^{1}H NMR (300 MHz, d_6-acetone) spectrum of **CP20**

Figure 163 ^{13}C NMR (75 MHz, d_6-acetone) spectrum of **CP20**
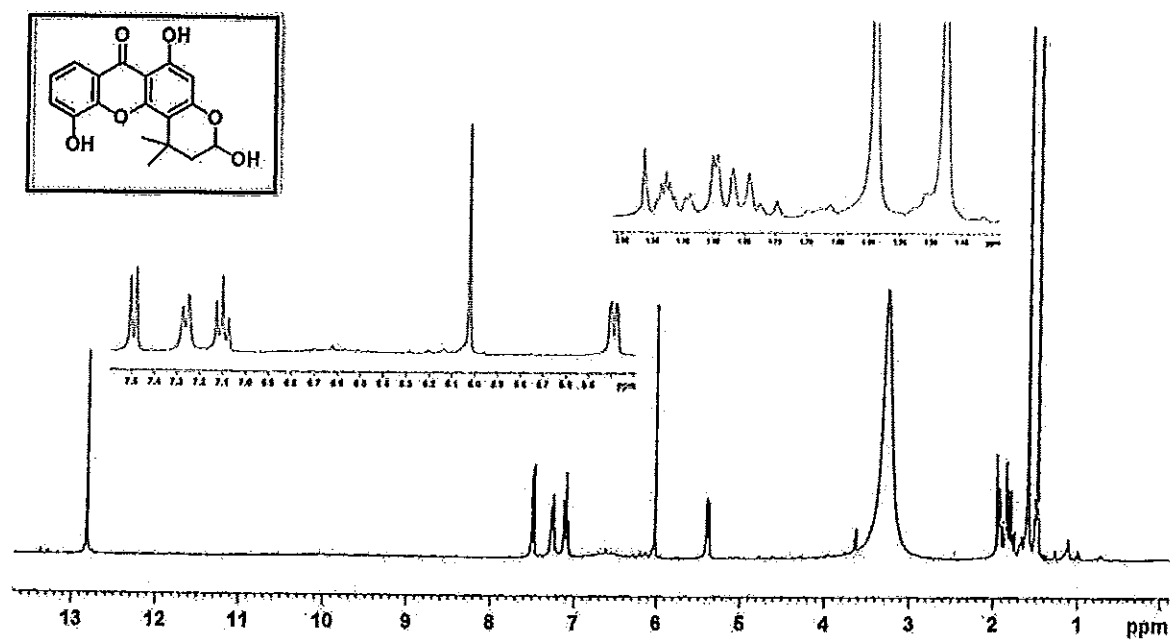
Figure 164 ^{1}H NMR (300 MHz, d_6-acetone) spectrum of **CP21**
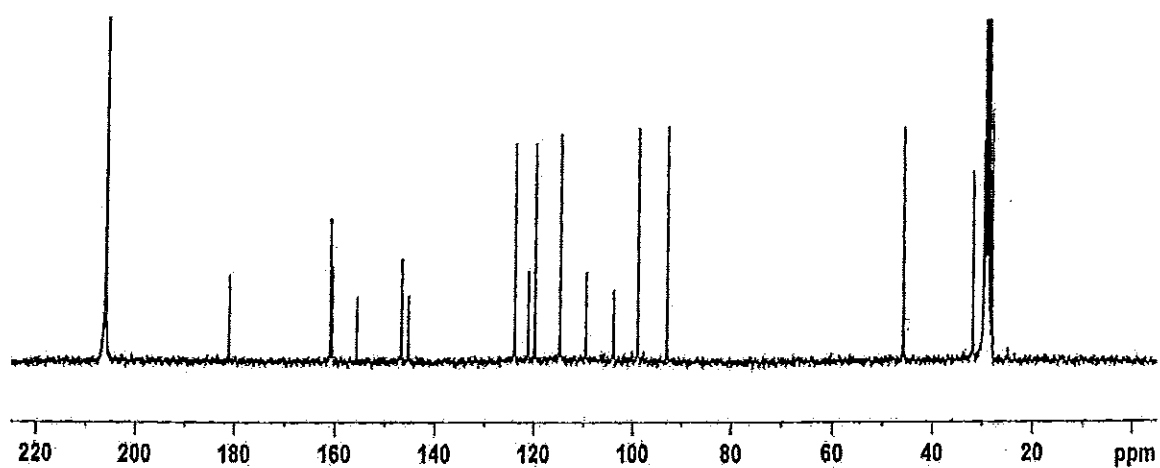
Figure 165 ^{13}C NMR (75 MHz, d_6-acetone) spectrum of **CP21**
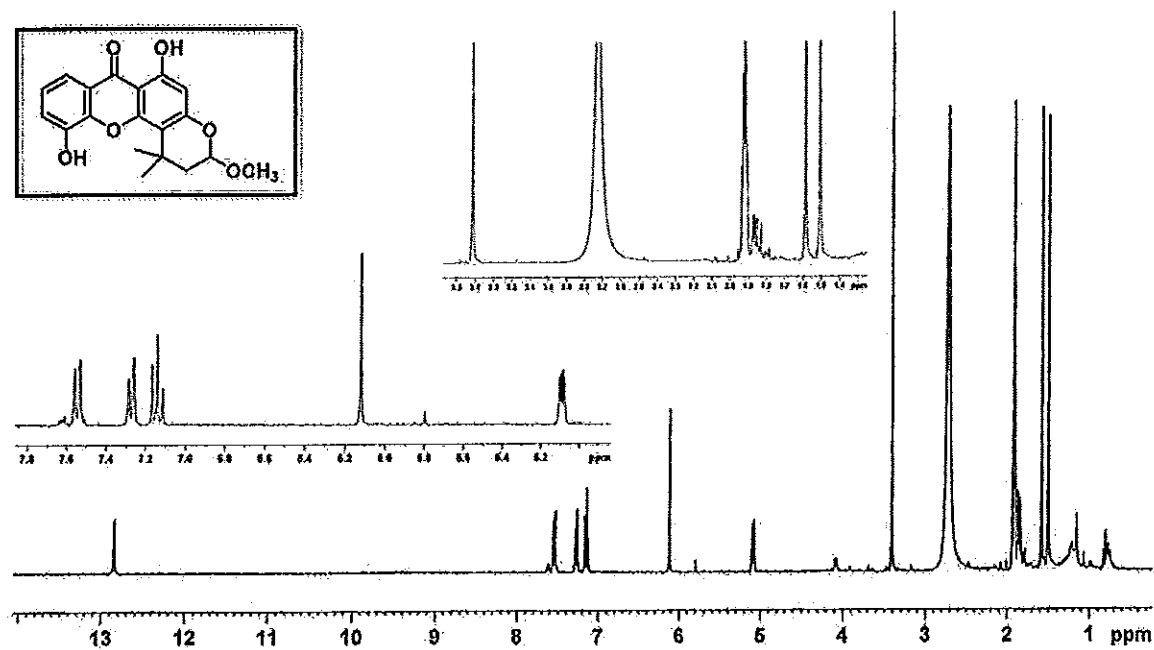
Figure 166 ^{1}H NMR (300 MHz, d_6-acetone) spectrum of **CP21a**
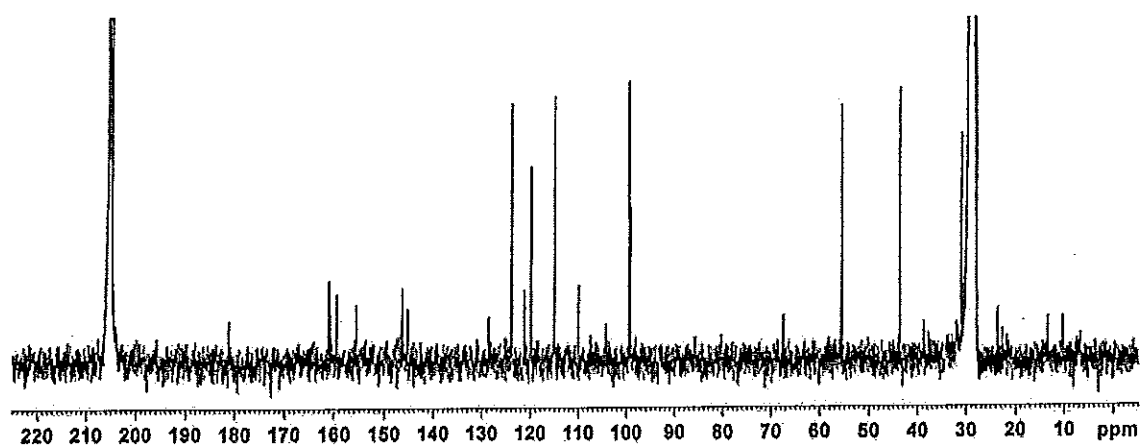
Figure 167 ^{13}C NMR (75 MHz, d_6-acetone) spectrum of **CP21a**
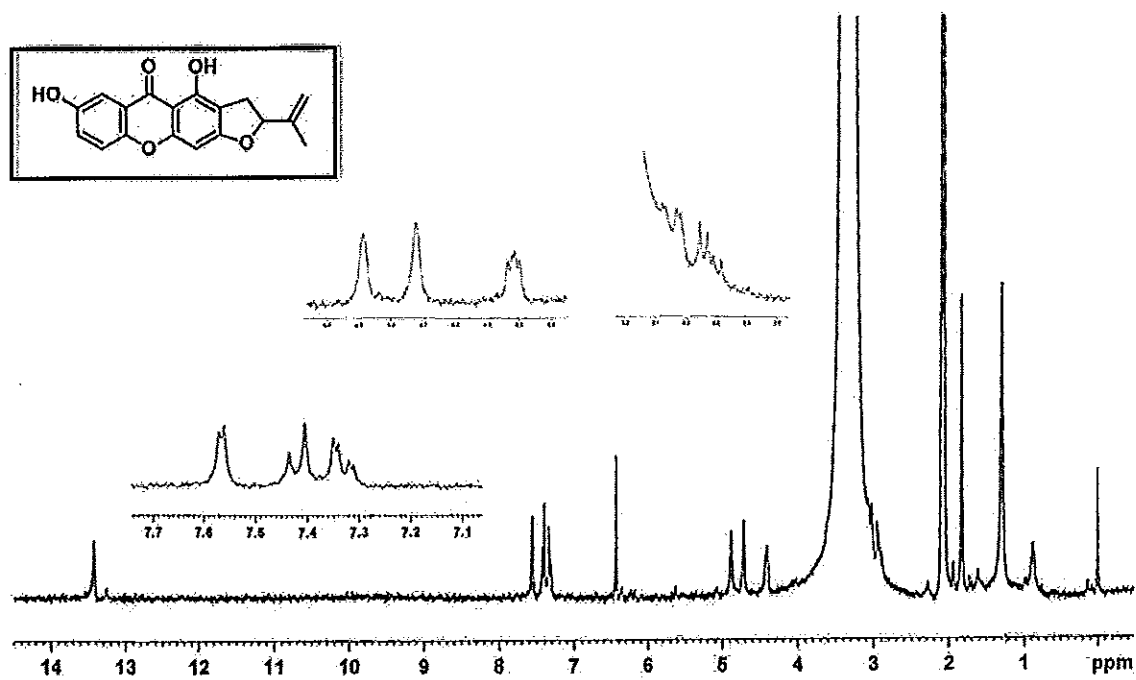
Figure 168 ^{1}H NMR (300 MHz, d_6-acetone) spectrum of **CP22**
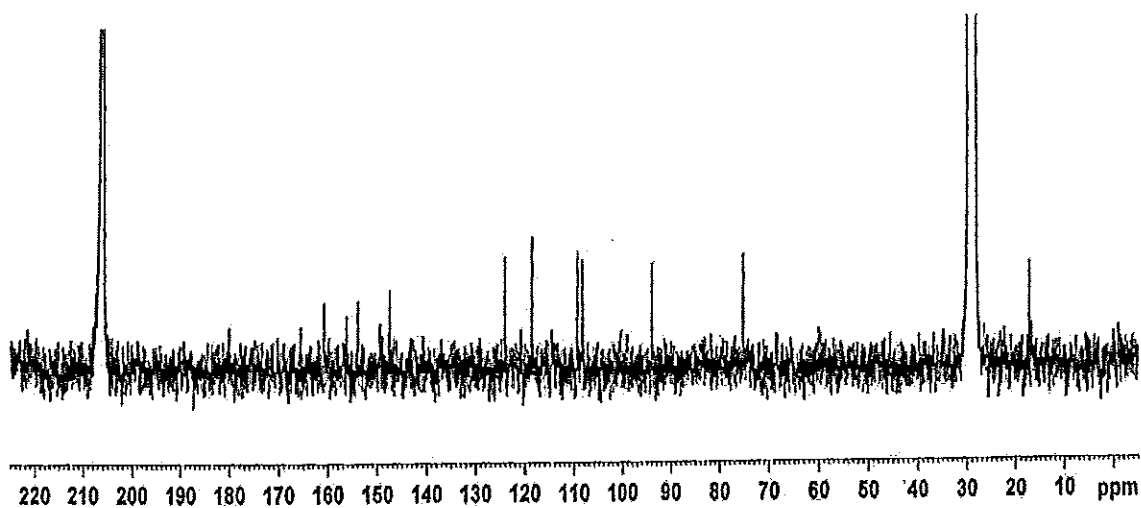
Figure 169 ^{13}C NMR (75 MHz, d_6-acetone) spectrum of **CP22**
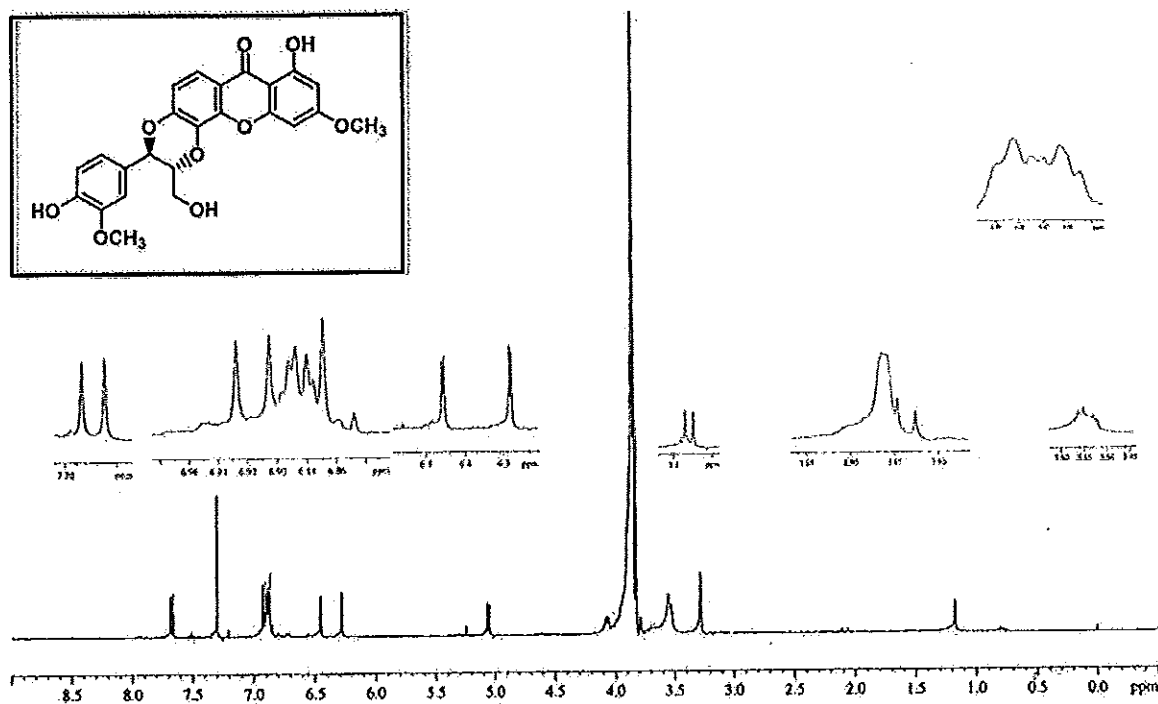
Figure 170 ^{1}H NMR (400 MHz, CD$_3$OD+CDCl$_3$) spectrum of **CP23**
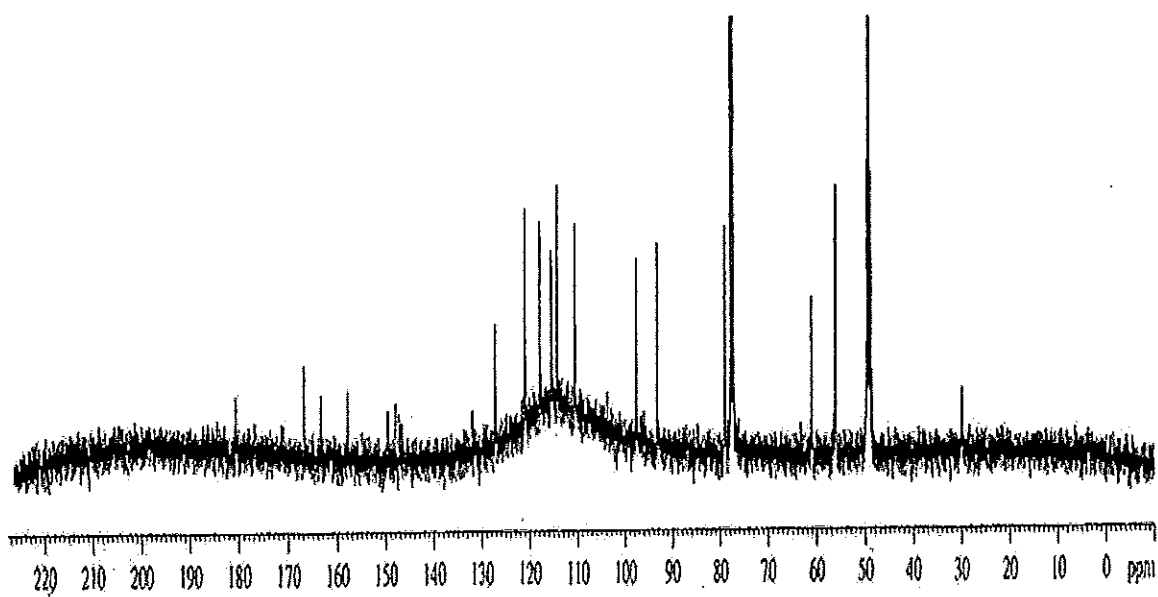
Figure 171 ^{13}C NMR (100 MHz, CD$_3$OD+CDCl$_3$) spectrum of **CP23**
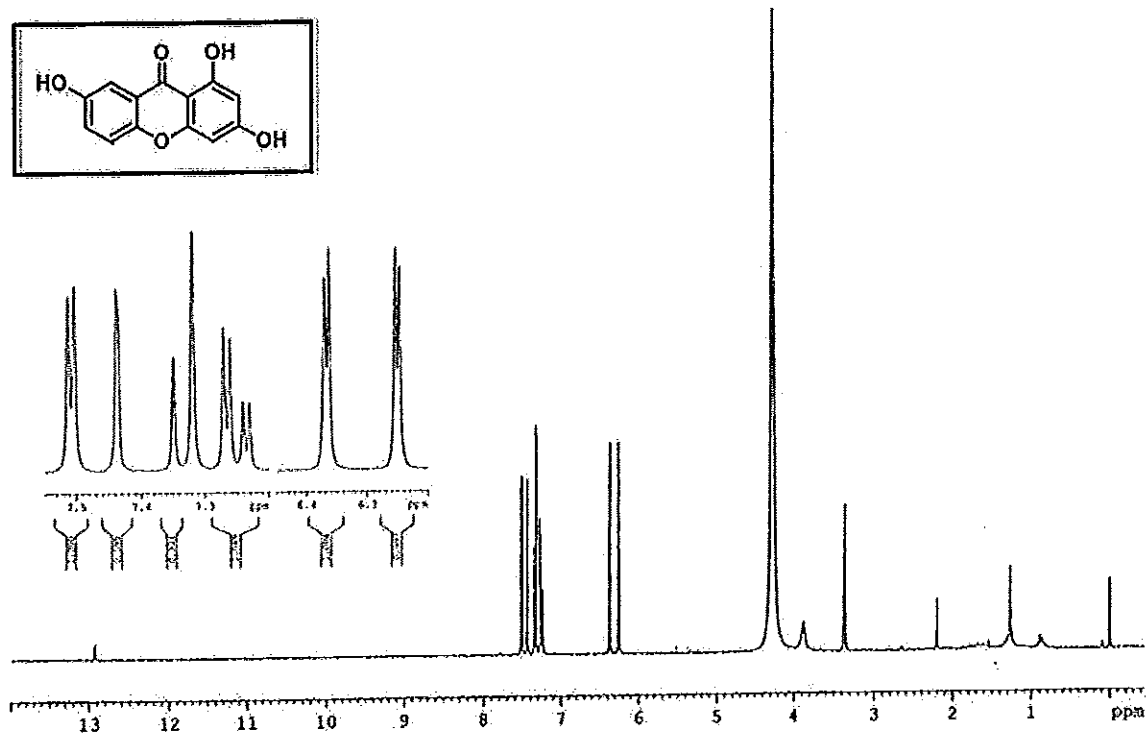
Figure 172 ^{1}H NMR (300 MHz, CD$_3$OD+CDCl$_3$) spectrum of **CP24**
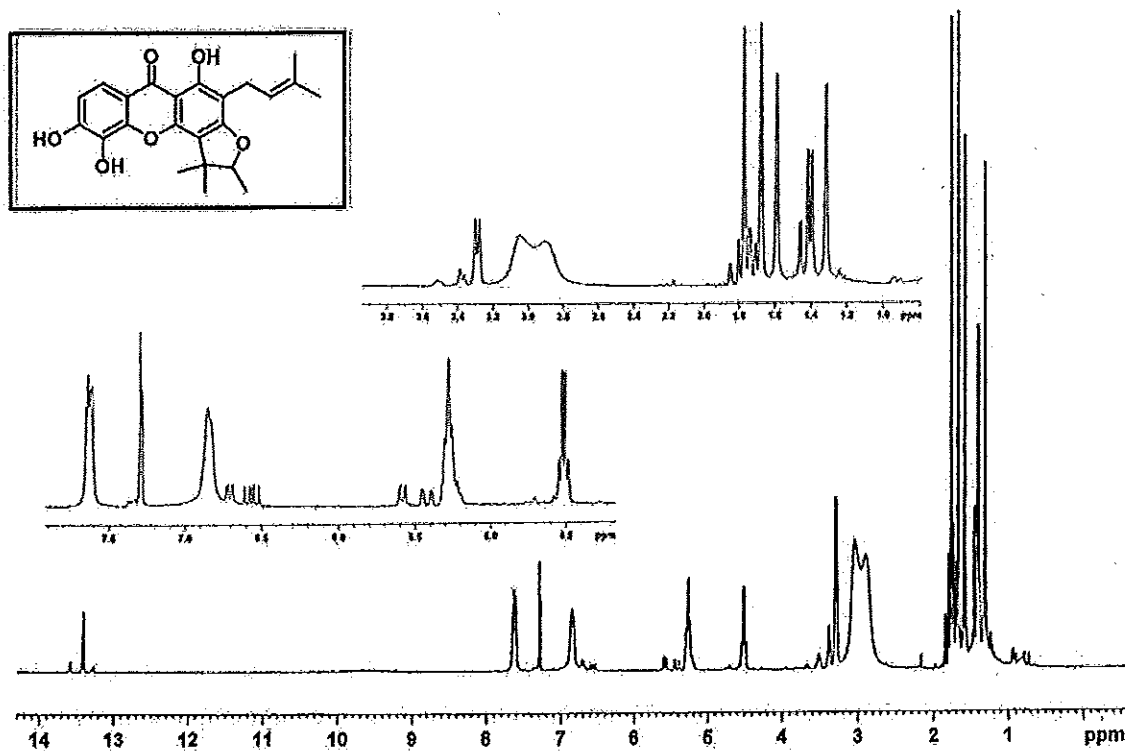
Figure 173 ^{1}H NMR (300 MHz, CD$_3$OD+CDCl$_3$) spectrum of **CP26**
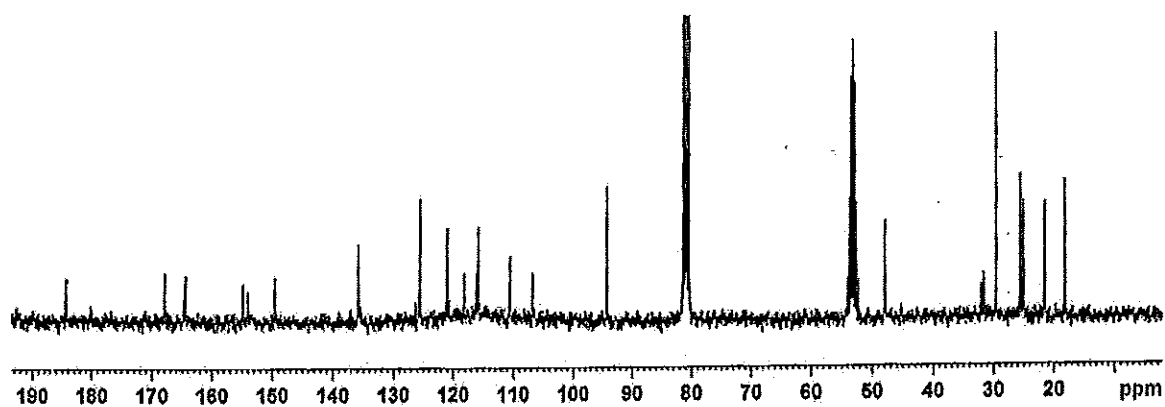
Figure 174 ^{13}C NMR (75 MHz, CD$_3$OD+CDCl$_3$) spectrum of **CP26**
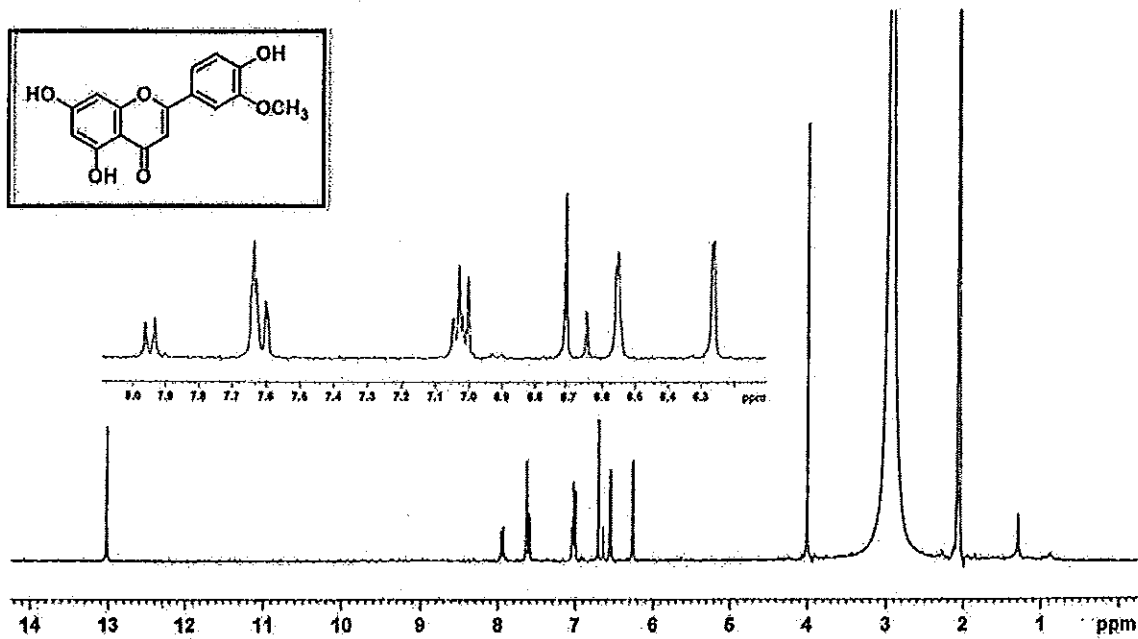
Figure 175 ^{1}H NMR (300 MHz, d_6-acetone) spectrum of **CP27**
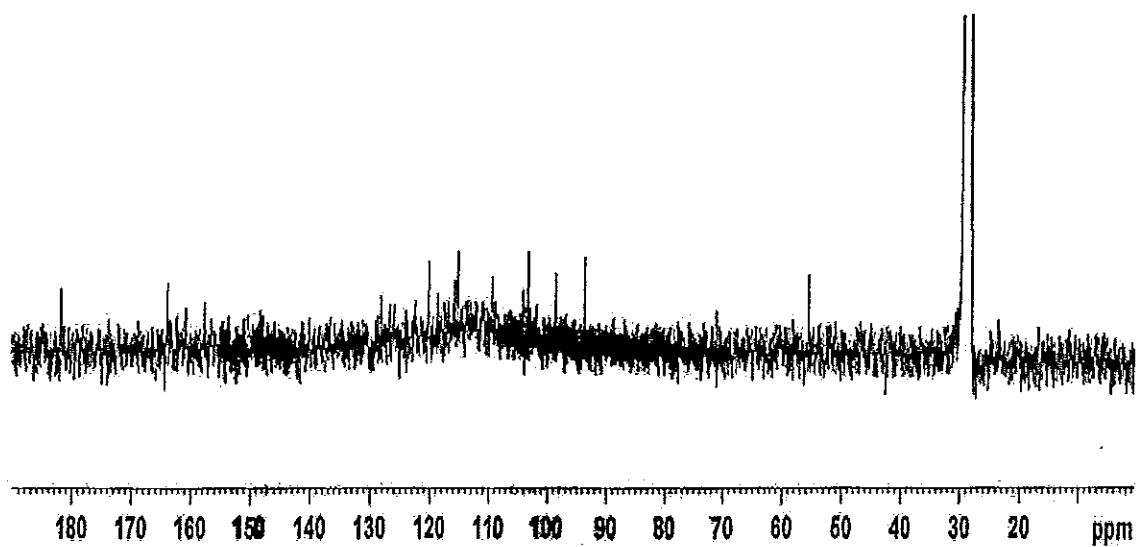
Figure 176 ^{13}C NMR (75 MHz, d_6-acetone) spectrum of **CP27**
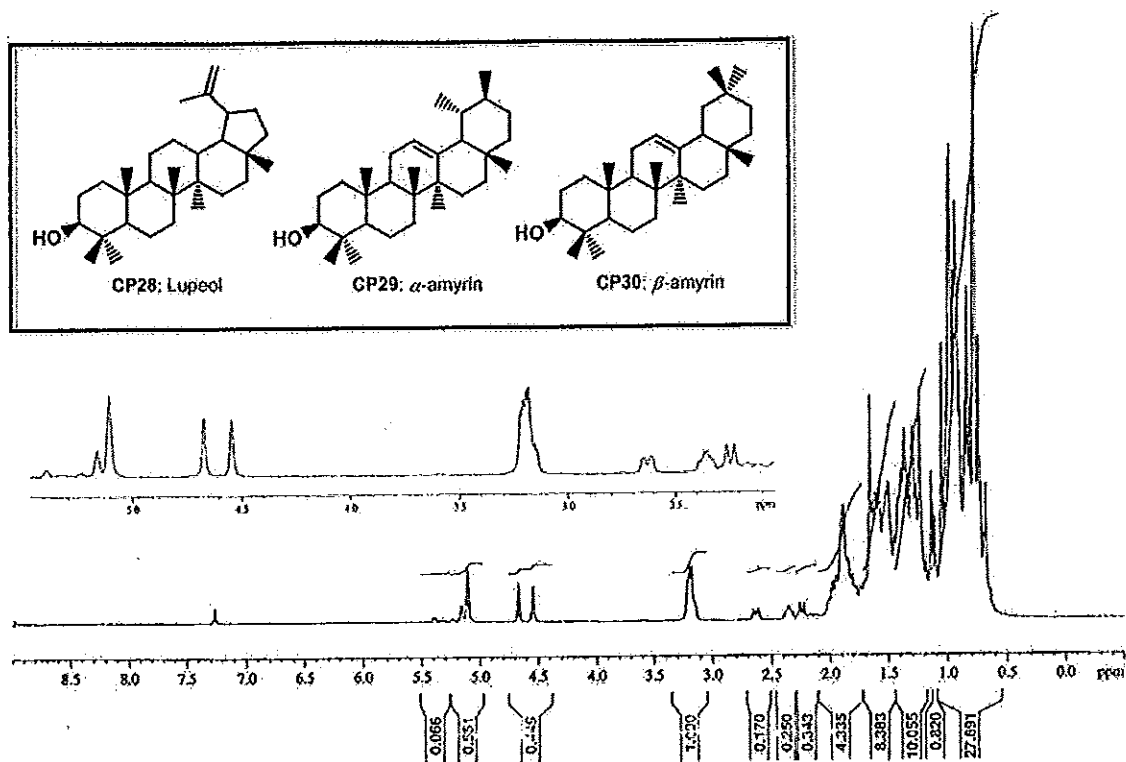
Figure 177 ^{1}H NMR (400 MHz, CDCl$_3$) spectrum of **CP28**, **CP29** and **CP30**
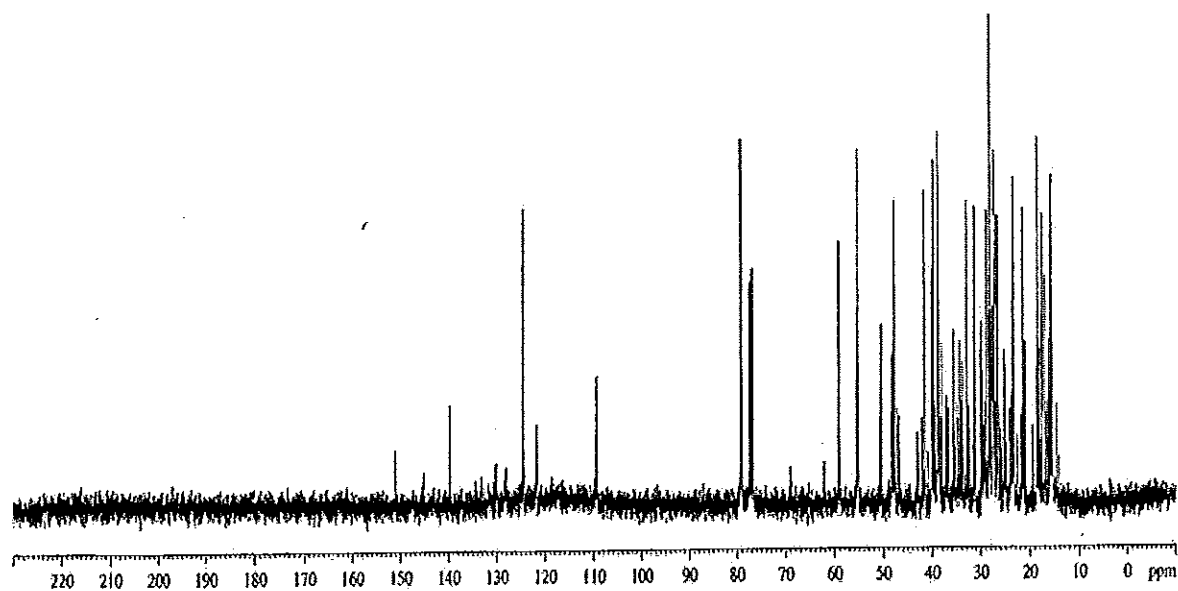
Figure 178 ^{13}C NMR (100 MHz, CDCl$_3$) spectrum of **CP28**, **CP29** and **CP30**
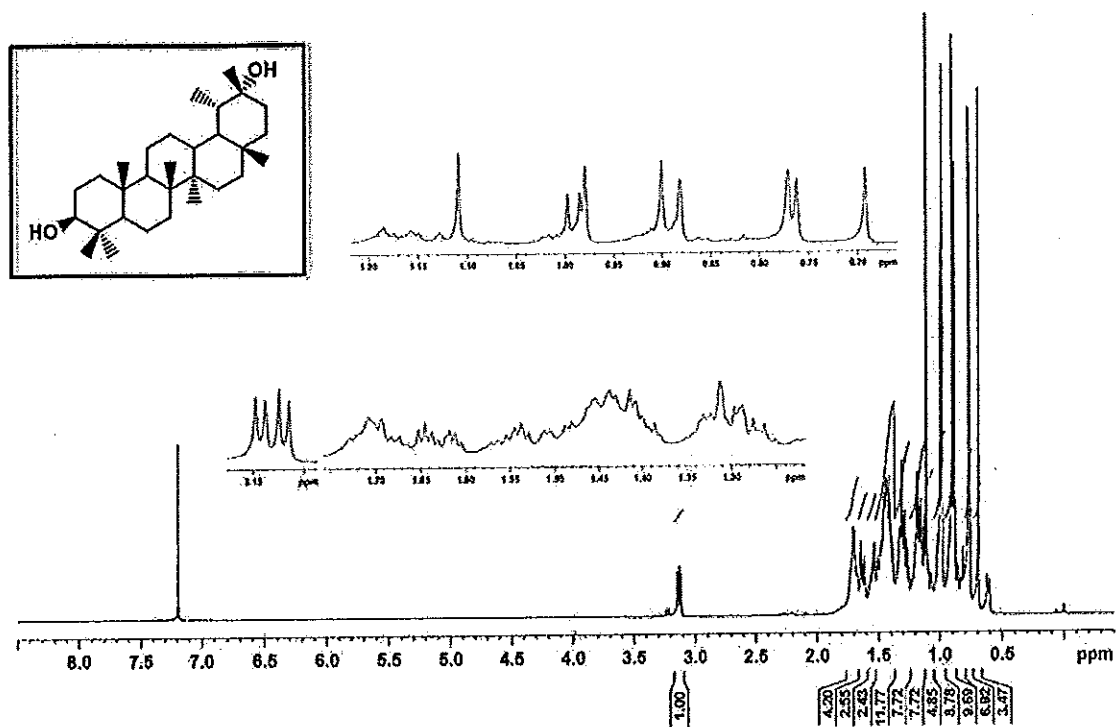
Figure 179 ^{1}H NMR (500 MHz, CDCl$_3$) spectrum of **CP31**
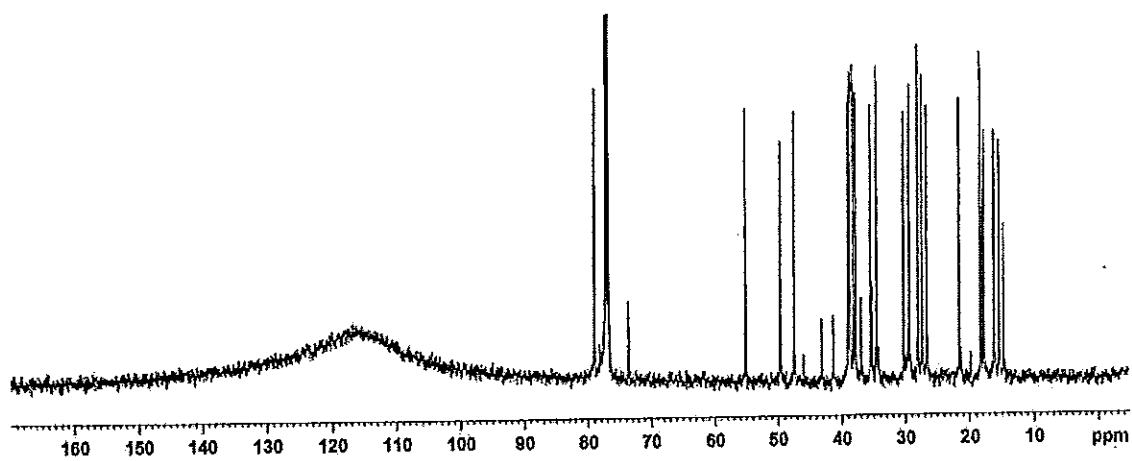

Figure 180 ^{13}C NMR (125 MHz, CDCl$_3$) spectrum of **CP31**

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Scholarship Awards during Enrollment

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- The Development and Promotion of Science and Technology Talents Project (DPST)

- The Center for Innovation in Chemistry (PERCH-CIC), commission on Higher Education, Ministry of Education

- The Thailand Research Fund

- The Graduate School, Prince of Songkla University

- The Koshinocorporation Group (Japan)

- Crystal Materials Research Unit, Department of Chemistry, Prince of Songkla University

List of Publications and Proceedings

Publications

1. Boonnak, N.; Chantrapromma, S.; Fun, H.-K.; Karalai, C. 2007. "4,8-dihydroxy-2,3-dimethoxy-1-(3-methylbut-2-enyl)-9H-xanthen-9-one" *Acta Cryst.* E63, o4903-o4904.

2. Boonnak, N.; Chantrapromma, S.; Fun, H.-K.; Karalai, C. 2010. "Vieillardiixanthone B" *Acta. Cryst.* E66, o817-o818.

3. Boonnak, N.; Karalai, C.; Chantrapromma, S.; Ponglimanont, C.; Kanjana-Opas, A.; Chantrapromma, K.; Shigeru, K. 2010. "Chromene and Prenylated Xanthones

from the Roots of *Cratoxylum formosum* ssp. *pruniflorum*" *Chem. Pharm. Bull.* 58, 386-389.

4. **Boonnak, N.**; Karalai, C.; Chantrapromma, S.; Ponglimanont, C.; Fun, H.-K.; Kanjana-Opas, A.; Chantrapromma, K.; Kato, S. 2009. "Anti-*Pseudomonas aeruginosa* Xanthones from the Resin and Green Fruits of *Cratoxylum cochinchinense*" *Tetrahedron* 65, 3003-3013.

5. **Boonnak, N.**; Khamthip, A.; Karalai, C.; Chantrapromma, S.; Ponglimanont, C.; Kanjana-Opas, A.; Tewtrakul, S.; Chantrapromma, K.; Fun, H.-K.; Kato, S. 2010. "Nitric Oxide Inhibitory Activity of Xanthones from the Green Fruits of *Cratoxylum formosum* ssp. *pruniflorum*" *Aust. J. Chem.* 63, 1550-1556.

6. Chantrapromma, S.; **Boonnak, N.**; Fun, H.-K.; Karalai, C.; Chantrapromma, K. 2010. "Brasilixanthone" *Acta. Cryst.* E66, o2066-o2067.

Proceedings

1. **Boonnak, N.**; Karalai, C.; Chantrapromma, S.; Ponglimanont, C.; Fun, H.-K.; Kanjana-Opas, A.; Chantrapromma, K.; Kato, S. 2009. "Chemical Constituents from resin and green fruits of the *Cratoxylum cochinchinense*" The 6th PERCH-CIC Congress VI, Jomtien Palm Beach Resort Pattaya, Chonburi. 3-6 May 2009. (Poster presentation)