



**Chemical Constituents from the Roots of *Cratoxylum formosum* and  
*Artocarpus integer* and the Stem of *Thespesia populnea***

**Sompong Boonsri**

**A Thesis Submitted in Partial Fulfillment of the Requirements  
for the Degree of Doctor of Philosophy in Organic Chemistry  
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**Thesis Title**            Chemical Constituents from the Roots of *Cratoxylum formosum* and *Artocarpus integer* and the Stem of *Thespesia populnea*

**Author**                    Mr. Sompong Boonsri

**Major Program**        Organic Chemistry

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**Major Advisor:**

.....  
(Assoc. Prof. Dr. Chatchanok Karalai)

**Examining Committee:**

.....Chairperson  
(Assoc. Prof. Dr. Kan Chantrapromma)

**Co-Advisor:**

.....  
(Assoc. Prof. Chanita Ponglimanont)

.....  
(Assoc. Prof. Dr. Chatchanok Karalai)

.....  
(Assoc. Prof. Chanita Ponglimanont)

.....  
(Assoc. Prof. Dr. Supinya Tewtrakul)

The Graduate School, Prince of Songkla University, has approved this thesis as partial fulfillment of the requirements for the Doctor of Philosophy degree in Organic Chemistry

.....  
(Assoc. Prof. Dr. Kerkchai Tongnoo)  
Dean of Graduate School

ชื่อวิทยานิพนธ์	องค์ประกอบทางเคมีจากรากตี้วขาวและจำปาตะและลำต้นโพทะเล
ผู้เขียน	นายสมพงษ์ บุญศรี
สาขาวิชา	เคมีอินทรีย์
ปีการศึกษา	2552

### บทคัดย่อ

#### ตอน 1 องค์ประกอบทางเคมีจากรากตี้วขาว (*Cratoxylum formosum*)

การศึกษาองค์ประกอบทางเคมีของส่วนสกัดหยาบเฮกเซนจากรากของตี้วขาว สามารถแยกสารประกอบประเภทแซนโทนชนิดใหม่ 3 สาร คือ formoxanthone A (CF1), formoxanthone B (CF2) และ formoxanthone C (CF3) และเป็นสารที่มีการรายงานแล้ว 6 สาร ซึ่งเป็นแซนโทน 3 สาร คือ gerontoxanthone I (CF4), macluraxanthone (CF5) และ xanthone V<sub>1</sub> (CF6) แอนทราควิโนน 3 สาร คือ madagascin (CF7), 3-geranyloxy-6-methyl-1,8-dihydroxyanthraquinone (CF8) และ vismiaquinone (CF9)

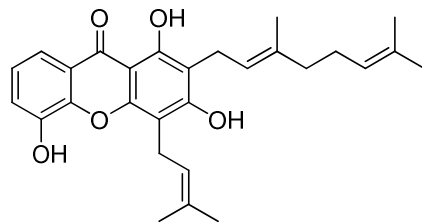
#### ตอน 2 องค์ประกอบทางเคมีจากลำต้นโพทะเล (*Thespesia populnea*)

การศึกษาองค์ประกอบทางเคมีของส่วนสกัดหยาบไดคลอโรมีเทนจากลำต้นของโพทะเล ซึ่งแบ่งเป็นสองส่วน คือ ส่วนกระพี้และแก่น สามารถแยกสารประกอบประเภทคาดีเนนเซสควิเทอร์พีนได้ 19 สาร จากส่วนกระพี้สามารถแยกสารประกอบชนิดใหม่ 2 สาร คือ populene A (TP10) และ populene B (TP11) และเป็นสารประกอบที่มีการรายงานแล้ว 3 สาร คือ mansonone E (TP9), (+)-gossypol (TP18) และ (+)-6, 6'-dimethoxygossypol (TP19) จากส่วนแก่นสามารถแยกสารประกอบประเภทเซสควิเทอร์พีนได้ 17 สาร ซึ่งเป็นสารประกอบชนิดใหม่ 6 สาร คือ populene C (TP12), populene D (TP13), populene E (TP14), populene F (TP15), populene G (TP16) และ populene H (TP17) และเป็นสารประกอบที่มีการรายงานแล้ว 11 สาร คือ 7-hydroxycadalene (TP1), mansonone C (TP2), mansonone G (TP3), mansonone D (TP4), thespesone (TP5), mansonone S (TP6), 7-hydroxy-2,3,5,6-tetrahydro-3,6,9-trimethyl-naphtho[1,8-b,c]pyran-4,8-dione (TP7), mansonone H (TP8), mansonone E (TP9), (+)-gossypol (TP18) และ (+)-6, 6'-dimethoxygossypol (TP19)

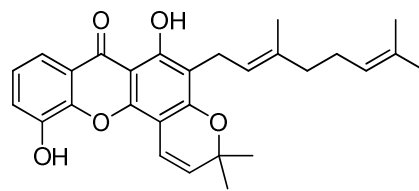
### ตอน 3 องค์ประกอบทางเคมีจากรากจำปาตะ (*Artocarpus integer*)

การศึกษาองค์ประกอบทางเคมีของส่วนสกัดหยาบไดคลอโรมีเทนจากรากของจำปาตะ สามารถแยกสารประกอบประเภทฟลาโวนอยด์ได้ 4 สาร ซึ่งเป็นสารประกอบที่มีการรายงานแล้ว คือ artoindonesianin A (AI1), Artoindonesianin Q (AI2), artoindonesianin S (AI3) และ corylifolin (AI4) โครงสร้างของสารประกอบเหล่านี้วิเคราะห์โดยใช้ข้อมูลทางสเปกโทรสโกปี

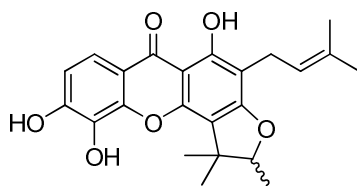
สารประกอบที่แยกได้นำไปทดสอบการออกฤทธิ์ยับยั้งการเจริญของเชื้อแบคทีเรีย และทดสอบความเป็นพิษต่อเซลล์มะเร็ง ซึ่งสารประกอบ mansonone E (TP9) มีความเป็นพิษต่อเซลล์มะเร็งเต้านม (MCF-7) ด้วยค่า  $IC_{50}$  0.05  $\mu\text{g/mL}$  และ (+)-gossypol (TP18) มีความเป็นพิษต่อเซลล์มะเร็งปากมดลูก (HeLa) และ มะเร็งช่องปากและหลอดอาหาร (KB) ด้วยค่า  $IC_{50}$  0.08 และ 0.04  $\mu\text{g/mL}$  ตามลำดับ



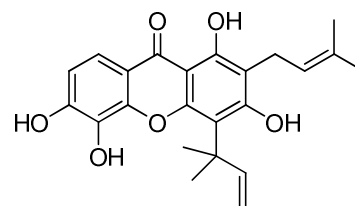
CF1: formoxanthone A



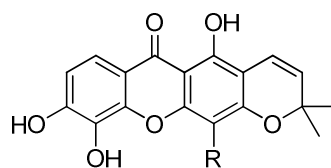
CF2: formoxanthone B

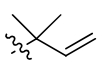


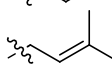
CF3: formoxanthone C

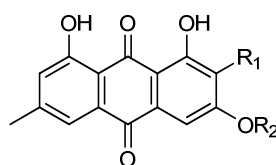


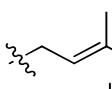
CF4: gerontoxanthone I

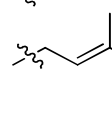


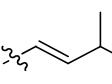
**CF5** : R =  ; macluraxanthone

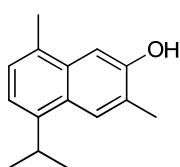
**CF6** : R =  ; xanthone V<sub>1</sub>



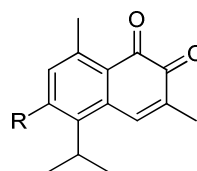
**CF7**: R<sub>1</sub> = H, R<sub>2</sub> =  ; madagascin

**CF8**: R<sub>1</sub> = H, R<sub>2</sub> =  ; 3-geranyloxy-6-methyl-1,8-dihydroxyanthraquinone

**CF9**: R<sub>1</sub> = , R<sub>2</sub> = CH<sub>3</sub> ; vismiaquinone

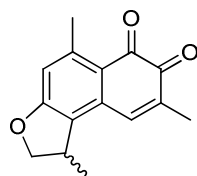


**TP1**: 7-hydroxycadalene

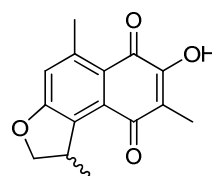


**TP2**: R = H; mansonone C

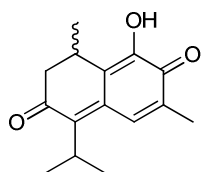
**TP3**: R = OH; mansonone G



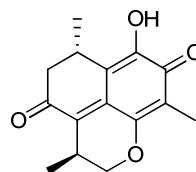
**TP4**: mansonone D



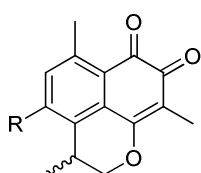
**TP5**: thespesone



**TP6:** mansonone S

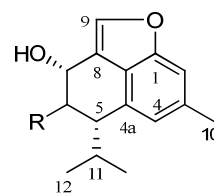


**TP7:** 7-hydroxy-2,3,5,6-tetrahydro-3,6,9-trimethyl-naphtho[1,8-b,c]-pyran-4,8-dione



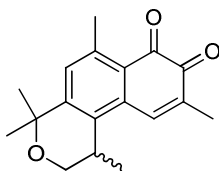
**TP8:** R = OH; mansonone H

**TP9:** R = H; mansonone E

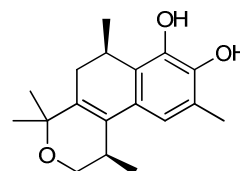


**TP10:** R =  $\beta$ OH; populene A

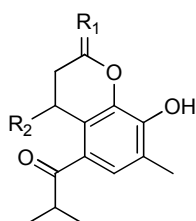
**TP10:** R =  $\alpha$ OH; populene B



**TP12:** populene C

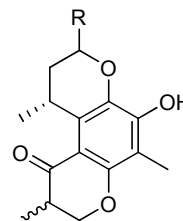


**TP13:** populene D



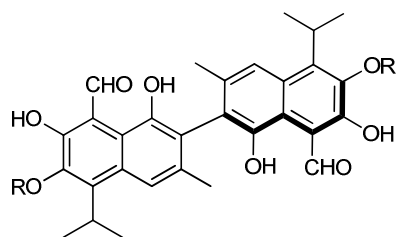
**TP14:** R<sub>1</sub> = O, R<sub>2</sub> = CH<sub>3</sub>; populene E

**TP15:** R<sub>1</sub> =  $\alpha$ OH, R<sub>2</sub> =  $\alpha$ CH<sub>3</sub>; populene F



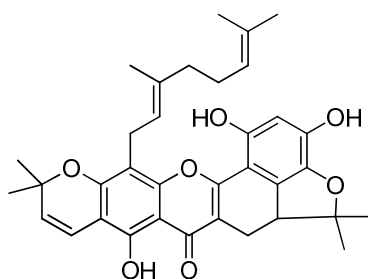
**TP16:** R =  $\alpha$ OH; populene G

**TP17:** R =  $\beta$ OH; populene H

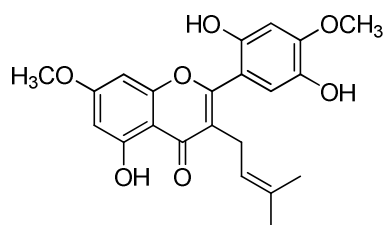


**TP18:** R = H; (+)-gossypol

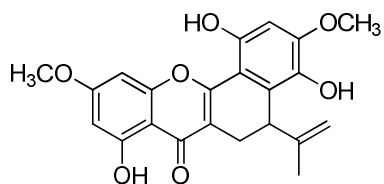
**TP19:** R = CH<sub>3</sub>; (+)-6,6'-dimethoxygossypol



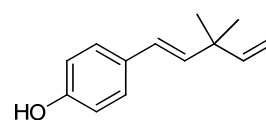
**AI1:** artoindonesianin A



**AI2:** artoindonesianin Q



**AI3:** artoindonesianin S



**AI4:** corylifolin

<b>Thesis Title</b>	Chemical Constituents from the Roots of <i>Cratoxylum formosum</i> and <i>Artocarpus integer</i> and the Stem of <i>Thespesia populnea</i>
<b>Author</b>	Mr. Sompong Boonsri
<b>Major Program</b>	Organic Chemistry

## ABSTRACT

### **Part I Chemical Constituents from the Roots of *Cratoxylum formosum***

Investigation of the chemical constituents of the hexane extract from the roots of *C. formosum* led to the isolation of three new xanthenes: formoxanthone A (**CF1**), formoxanthone B (**CF2**) and formoxanthone C (**CF3**), together with six known compounds: three xanthenes: gerontoxanthone I (**CF4**), macluraxanthone (**CF5**) and xanthone V<sub>1</sub> (**CF6**); three anthraquinones: madagascin (**CF7**), 3-geranyloxy-6-methyl-1,8-dihydroxyanthraquinone (**CF8**) and vismiaquinone (**CF9**).

### **Part II Chemical Constituents from the Stem of *Thespesia populnea***

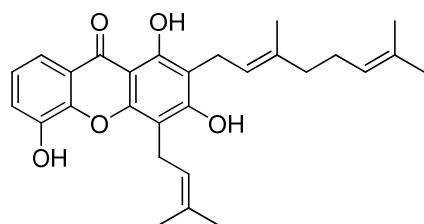
Investigation of the chemical constituents of the dichloromethane extract from the stem of *T. populnea* which was divided to two parts, heartwood and wood, resulted in nineteen cadinan sesquiterpenes. Two new compounds, populene A (**TP10**) and B (**TP11**) along with mansonone E (**TP9**), (+)-gossypol (**TP18**) and (+)-6,6'-dimethoxygossypol (**TP19**) were purified from the wood. Six new compounds, populene C (**TP12**), populene D (**TP13**), populene E (**TP14**), populene F (**TP15**), populene G (**TP16**) and populene H (**TP17**) were obtained from the heartwood, together with eleven known compounds, 7-hydroxycadalene (**TP1**), mansonone C (**TP2**), mansonone G (**TP3**), mansonone D (**TP4**), thespesone (**TP5**), mansonone S (**TP6**), 7-hydroxy-2,3,5,6-tetrahydro-3,6,9-trimethyl-naphtho[1,8-b,c]pyran-4,8-dione (**TP7**), mansonone H (**TP8**), mansonone E (**TP9**), (+)-gossypol (**TP18**) and (+)-6,6'-dimethoxygossypol (**TP19**).



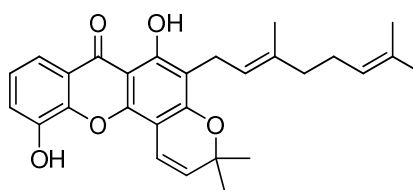
### Part III Chemical Constituents from the Roots of *Artocarpus integer*

The dichloromethane extract of the roots of *Artocarpus integer* yielded four known compounds, artoindonesianin A (**AI1**), artoindonesianin Q (**AI2**), artoindonesianin S (**AI3**) and corylifolin (**AI4**). Their structure were elucidated by spectroscopic method.

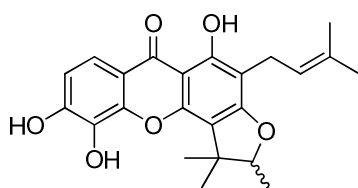
The isolated compounds were evaluated for their antibacterial and cytotoxic activities. Two pure compounds, mansonone E (**TP9**) exhibited potent cytotoxicity against breast cancer cell line (MCF-7) with  $IC_{50}$  value  $0.05 \mu\text{g/mL}$  and (+)-gossypol (**TP18**) exhibited potent cytotoxicity against cervical cancer (HeLa) and oral cavity cancer (KB) cell lines with  $IC_{50}$  values  $0.08$  and  $0.04 \mu\text{g/mL}$ , respectively.



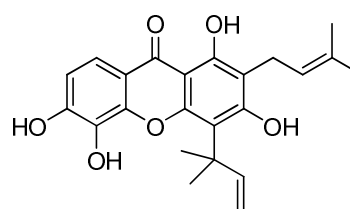
**CF1:** formoxanthone A



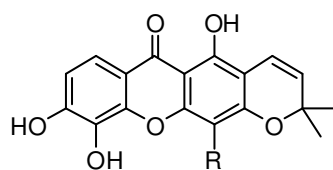
**CF2:** formoxanthone B



**CF3:** formoxanthone C

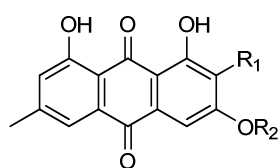


**CF4:** gerontoxanthone I



**CF5:** R = ; macluraxanthone

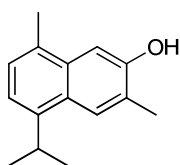
**CF6:** R = ; xanthone V<sub>1</sub>



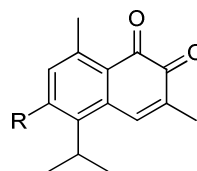
**CF7:** R<sub>1</sub> = H, R<sub>2</sub> = ; madagascin

**CF8:** R<sub>1</sub> = H, R<sub>2</sub> = ; 3-geranyloxy-6-methyl-1,8-dihydroxyanthraquinone

**CF9:** R<sub>1</sub> = , R<sub>2</sub> = CH<sub>3</sub>; vismiaquinone

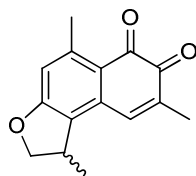


**TP1:** 7-hydroxycadalene

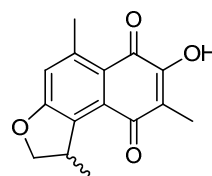


**TP2:** R = H; mansonone C

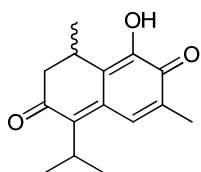
**TP3:** R = OH; mansonone G



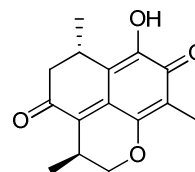
**TP4:** mansonone D



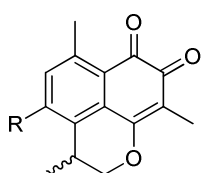
**TP5:** thespesone



**TP6:** mansonone S

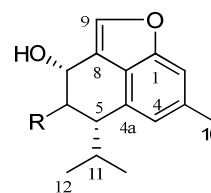


**TP7:** 7-hydroxy-2,3,5,6-tetrahydro-3,6,9-trimethyl-naphtho[1,8-b,c]-pyran-4,8-dione



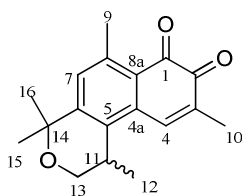
**TP8:** R = OH; mansonone H

**TP9:** R = H; mansonone E

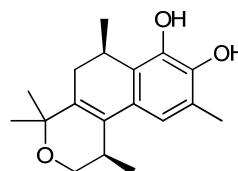


**TP10:** R =  $\beta$ OH; populene A

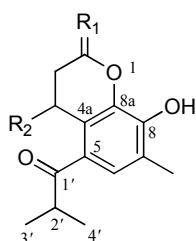
**TP10:** R =  $\alpha$ OH; populene B



**TP12:** populene C

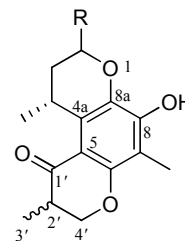


**TP13:** populene D



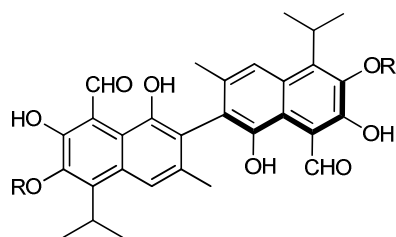
**TP14:** R<sub>1</sub> = O, R<sub>2</sub> = CH<sub>3</sub>; populene E

**TP15:** R<sub>1</sub> =  $\alpha$ OH, R<sub>2</sub> =  $\alpha$ CH<sub>3</sub>; populene F



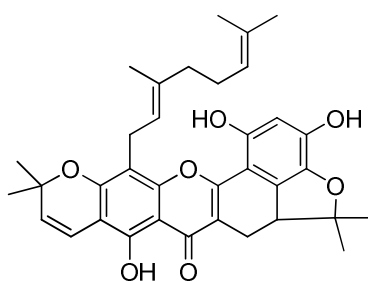
**TP16:** R =  $\alpha$ OH; populene G

**TP17:** R =  $\beta$ OH; populene H

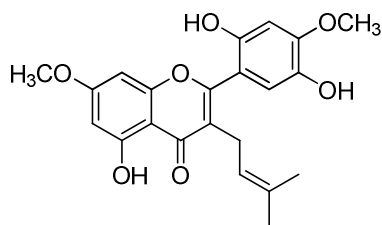


**TP18:** R = H; (+)-gossypol

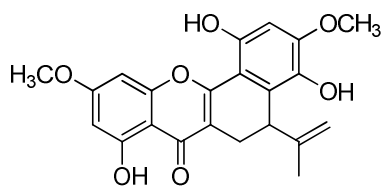
**TP19:** R = Me; (+)-6,6'-dimethoxygossypol



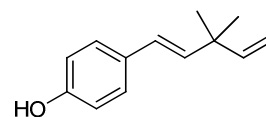
**AI1:** artoindonesianin A



**AI2:** artoindonesianin Q



**AI3:** artoindonesianin S



**AI4:** corylifolin

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## ABBREVIATIONS AND SYMBOLS

<i>s</i>	=	<i>singlet</i>
<i>d</i>	=	<i>doublet</i>
<i>t</i>	=	<i>triplet</i>
<i>m</i>	=	<i>multiplet</i>
<i>sept</i>	=	<i>septet</i>
<i>hept</i>	=	<i>heptet</i>
<i>dd</i>	=	<i>doublet of doublet</i>
<i>dt</i>	=	<i>doublet of triplet</i>
<i>dquint</i>	=	<i>doublet of quintet</i>
<i>tq</i>	=	<i>triplet of quatet</i>
<i>mt</i>	=	<i>multiplet of triplet</i>
<i>br s</i>	=	<i>broad singlet</i>
<i>br d</i>	=	<i>broad doublet</i>
<i>br q</i>	=	<i>broad quatet</i>
<i>br dd</i>	=	<i>broad doublet of doublet</i>
<i>br dq</i>	=	<i>broad doublet of quatet</i>
<i>g</i>	=	<i>gram</i>
<i>kg</i>	=	<i>kilogram</i>
<i>mg</i>	=	<i>milligram</i>
$\mu\text{g}$	=	<i>microgram</i>
<i>mL</i>	=	<i>milliliter</i>
<i>mult.</i>	=	<i>multiplicity</i>
<i>%</i>	=	<i>percent</i>
<i>m.p.</i>	=	<i>melting point</i>
$\text{cm}^{-1}$	=	<i>reciprocal centimeter (wave number)</i>
$\delta$	=	<i>chemical shift relative to TMS</i>
<i>J</i>	=	<i>coupling constant</i>
$[\alpha]_{\text{D}}$	=	<i>specific rotation</i>

## ABBREVIATIONS AND SYMBOLS (Continued)

$\lambda_{\max}$	=	maximum wavelength
$\nu$	=	absorption frequencies
$\epsilon$	=	molar extinction coefficient
$m/z$	=	a value of mass divided by charge
$^{\circ}\text{C}$	=	degree celcius
MHz	=	Megahertz
ppm	=	part per million
$c$	=	concentration
MS	=	Mass Spectroscopy
EIMS	=	Electron Impact Mass Spectrometry
UV	=	Ultraviolet-Visible
IR	=	Infrared
NMR	=	Nuclear Magnetic Resonance
2D NMR	=	Two Dimentional Nuclear Magnetic Resonance
COSY	=	Correlated Spectroscopy
DEPT	=	Distortionless Enhancement by Polarization Transfer
HMBC	=	Heteronuclear Multiple Bond Correlation
HMQC	=	Heteronuclear Multiple Quantum Coherence
NOESY	=	Nuclear Overhauser Effect Spectroscopy
CC	=	Column Chromatography
QCC	=	Quick Column Chromatography
PLC	=	Preparative Thin Layer Chromatography
$\text{CH}_2\text{Cl}_2$	=	dichloromethane
$\text{CHCl}_3$	=	chloroform
EtOAc	=	ethyl acetate
MeOH	=	methanol
TMS	=	tetramethylsilane
Acetone- $d_6$	=	deuteroacetone
DMSO- $d_6$	=	deuterodimethyl sulphoxide

## ABBREVIATIONS AND SYMBOLS (Continued)

$\text{CDCl}_3$	=	deuteriochloroform
$\text{CD}_3\text{OD}$	=	deuteromethanol
$\text{IC}_{50}$	=	50% Inhibition Concentration

# CHAPTER 1.1

## INTRODUCTION

### 1.1.1 Introduction

*Cratoxylum* is a plant belonging to a small genus of the family Guttiferae, which can be found in several Southeast Asian countries, The genus *Cratoxylum* has about 6 species, which are all found in Thailand (Smitinand, 2001): *Cratoxylum aborescens*, *Cratoxylum cochinchinense*, *Cratoxylum maingayi*, *Cratoxylum sumatranum* ssp. *neriifolium*, *Cratoxylum formosum* ssp. *formosum* (Jack) Dyer and *Cratoxylum formosum* (Jack) Dyer ssp. *pruniflorum* (Kurz) Gogel. 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. *pruniflorum* are densely villous, whereas *C. formosum* ssp. *formosum* are glabrous (Veesommai, *et al.*, 2004).

*C. formosum* ssp. *formosum* is a shrub or tree deciduous, 3-6 m tall. Bark exfoliating in flakes. Twigs somewhat compressed. Petiole 5-7 mm, glabrous; leaf blade abaxially greenish, adaxially green, elliptic to oblong, 4-10 × 2-4 mm. Cymes 5-8 flowers, in axils of fallen leaves. Pedicels 3-5 mm. Flowers ca. 1.3 cm in diam. Sepals elliptic or oblong-lanceolate, 5-6 × 2-3 mm, apex obtuse. Petals obovate-oblong, 1.1-1.5 cm, ciliolate and brown-granular on upper half of margin, narrowly clawed at base; petal-scale indistinct, ca 2 mm, base cuneate, apex truncate and denticulate. Ovary narrowly conic, ca. 4 mm, glabrous; styles ca. ca. 3.5 mm. Capsule dark brown, oblong, 0.6-1.5 cm, up to ½ enclosed by persistent calyx. Seeds 6-8 per locule, 3-7 mm.



**Figure 1** Parts of *Cratoxylum formosum* ssp. *formosum*

### 1.1.2 Review of Literatures

Chemical constituents isolated from *Cratoxylum* genus were summarized by Nawong Boonnak in 2006 (Boonnak, 2006). Information from SciFinder Scholar database reported the additional constituents from *Cratoxylum* genus and they could be classified into groups, such as anthraquinones, benzenoids, benzophenones, flavonoids, triterpenes and xanthenes. These compounds are presented in **Table 1**.

**Table 1** Compounds from plants of *Cratoxylum* genus

**a** = Anthraquinones                      **b** = Benzenoids                      **c** = Benzophenones  
**d** = Flavonoids                              **e** = Triterpenes                      **f** = Xanthenes

Scientific name	Investigated Part	Compound	Bibliography
<i>C. aborescens</i>	Leaves+Twigs	3,4-Dihydroxybenzoic acid, <b>1b</b> Betulinic acid, <b>5e</b> Euxanthone, <b>39f</b> 3 $\beta$ -Hydroxylup-20(29)-en-30-oic acid, <b>4e</b> Lup-20(29)-ene-3 $\beta$ ,30-diol, <b>3e</b> Methoxyemodin, <b>8a</b> Friedelin, <b>2e</b> Friedelinol, <b>1e</b> Astilbin, <b>2d</b> Isoastilbin, <b>3d</b> 1,3,8-Trihydroxy-2,4-dimethoxyxanthone, <b>43f</b>	Reutrakul <i>et al.</i> , 2006



Table 1 (Continued)

Scientific name	Investigated Part	Compound	Bibliography
<i>C. aborescens</i>	Leaves+Twigs	1,7-Dihydroxy-2,8-dimethoxyxanthone, <b>57f</b> 1,3,7-Trihydroxy-6-methoxy-4,5-diisoprenylxanthone, <b>40f</b> 3,5,7-Trihydroxy-2-methoxy-1,8-bis(3-methyl-2-buten-1-yl)-9H-xanthone-9-one, <b>34f</b>	Reutrakul <i>et al.</i> , 2006
<i>C. cochinchinense</i>	Fruits	Cochinxanthone A, <b>1f</b> Cochinxanthone B, <b>2f</b> Cochinxanthone C, <b>3f</b> 1,3,7-Trihydroxyxanthone, <b>5f</b> Vismiaquinone C, <b>7a</b> Fuscaxanthone E, <b>6f</b> Cochinchinone G, <b>15f</b>	Laphookhieo <i>et al.</i> , 2008  Laphookhieo <i>et al.</i> , 2008 Laphookhieo <i>et al.</i> , 2009 Laphookhieo <i>et al.</i> , 2008 Mahabusarakam <i>et al.</i> , 2008

Table 1 (Continued)

Scientific name	Investigated Part	Compound	Bibliography
<i>C. cochinchinense</i>	Fruits	7-Geranyloxy-1,3-dihydroxyxanthone, <b>4f</b>  1,8-Dihydroxy-3-methoxy-6-methyl-2-(3-methyl-2-butenyl)anthraquinone, <b>7a</b>	Laphookhieo <i>et al.</i> , 2008 Laphookhieo <i>et al.</i> , 2009 Mahabusarakam <i>et al.</i> , 2008 Mahabusarakam <i>et al.</i> , 2008
	Resin+Fruits	Cochinchinone A, <b>8f</b>  Cochinchinone C, <b>10f</b> Cochinchinone I, <b>16f</b> Cochinchinone J, <b>17f</b> Cochinchinone K, <b>18f</b> Cochinchinone L, <b>19f</b> Dulcisxanthone F, <b>42f</b> 1,3,7-Trihydroxy-2,4-diisoprenylxanthone, <b>7f</b> 7-Geranyloxy-1,3-dihydroxyxanthone, <b>4f</b> Celebixanthone methyl ether, <b>41f</b> $\alpha$ -Mangostin, <b>27f</b> $\beta$ -Mangostin, <b>28f</b> Macluraxanthone, <b>54f</b>	Boonnak <i>et al.</i> , 2009

Table 1 (Continued)

Scientific name	Investigated Part	Compound	Bibliography
<i>C. cochinchinense</i>	Resin+Fruits	Pruniflorone G, <b>51f</b>	Boonnak <i>et al.</i> , 2009
	Roots	5- <i>O</i> -Methylcelebixanthone, <b>20f</b>	Laphookhieo <i>et al.</i> , 2006
		Celebixanthone, <b>21f</b>	Laphookhieo <i>et al.</i> , 2006
		Cochinchinone B, <b>9f</b>	Mahabusarakam <i>et al.</i> , 2006
		Cochinchinone D, <b>11f</b>	Mahabusarakam <i>et al.</i> , 2006
		4-Deprenylbratatin, <b>12f</b>	
		Macluraxanthone, <b>54f</b>	
		Garcinone B, <b>38f</b>	
		Garcinone D, <b>37f</b>	
		Celebixanthone, <b>21f</b>	
1,3,7-Trihydroxy-2,4-di(3-methylbut-2-enyl)xanthone, <b>58f</b>	Laphookhieo <i>et al.</i> , 2006		
Cochinchinone A, <b>8f</b>	Mahabusarakam <i>et al.</i> , 2006		
$\alpha$ -Mangostin, <b>27f</b>			
$\beta$ -Mangostin, <b>28f</b>			
Cochinchinone C, <b>10f</b>			
Cochinchinone E, <b>13f</b>	Mahabusarakam <i>et al.</i> , 2008		
Cochinchinone F, <b>14f</b>			



Table 1 (Continued)

Scientific name	Investigated Part	Compound	Bibliography
<i>C. formosum</i>	Roots	Formoxanthone A, <b>59f</b> Formoxanthone B, <b>60f</b> Formoxanthone C, <b>61f</b> Macluraxanthone, <b>54f</b> Xanthone V <sub>1</sub> , <b>55f</b> Gerontoxanthone I, <b>26f</b> 3-Geranyloxy-6-methyl-1,8-dihydroxyanthraquinone, <b>1a</b> Vismiaquinone, <b>6a</b> Madagascin, <b>3a</b>	Boonsri <i>et al.</i> , 2006
<i>C. formosum</i> subsp. <i>pruniflorum</i>	Bark	Bianthrone J, <b>10a</b>  Bianthrone A <sub>1</sub> , <b>11a</b> Vismiaquinone, <b>6a</b> 11-Hydroxy-5-methoxy-2,2,9-trimethyl-2 <i>H</i> -anthra[1,2- <i>b</i> ]-pyran-7,12-dione, <b>9a</b> 3-Geranyloxy-6-methyl-1,8-dihydroxyanthraquinone, <b>1a</b> Pruniflorone J, <b>2a</b> Madagascin, <b>3a</b>	Boonnak <i>et al.</i> , 2007  Boonnak <i>et al.</i> , 2006, Boonnak <i>et al.</i> , 2007  Boonnak <i>et al.</i> , 2006

**Table 1** (Continued)

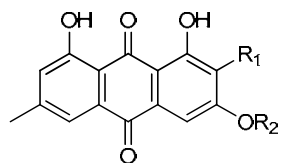
<b>Scientific name</b>	<b>Investigated Part</b>	<b>Compound</b>	<b>Bibliography</b>
<i>C. formosum</i> subsp. <i>pruniflorum</i>	Bark	Physcion, <b>4a</b>  Emodin, <b>5a</b> Formoxanthone B, <b>60f</b> Macluraxanthone, <b>54f</b> Xanthone V <sub>1</sub> , <b>55f</b> Gerontoxanthone I, <b>26f</b> 6-Deoxyjacareubin, <b>56f</b>	Boonnak <i>et al.</i> , 2006
	Roots	Pruniflorone A, <b>44f</b>  Pruniflorone B, <b>45f</b> Pruniflorone C, <b>46f</b> Pruniflorone D, <b>47f</b> Pruniflorone E, <b>48f</b> Pruniflorone F, <b>49f</b> Pruniflorone G, <b>51f</b> Pruniflorone H, <b>52f</b> Pruniflorone I, <b>53f</b> Dulcisxanthone F, <b>42f</b> $\alpha$ -Mangostin, <b>27f</b> $\beta$ -Mangostin, <b>28f</b> 3-Isomangostin, <b>23f</b> Formoxanthone A, <b>59f</b>	Boonnak <i>et al.</i> , 2006

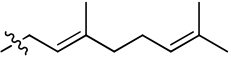
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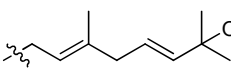
Scientific name	Investigated Part	Compound	Bibliography
<i>C. formosum</i> subsp. <i>pruniflorum</i>	Roots	3,4-Dihydro-5,9-dihydroxy-8-methoxy-7-(3-methoxy-3-methylbutyl)-2,2-dimethyl-2 <i>H</i> ,6 <i>H</i> -pyrano[3,2- <i>b</i> ]-xanthen-6-one, <b>23f</b> 3,4-Dihydro-5,9-dihydroxy - 7-(3-hydroxy-3-methylbutyl)-8-methoxy-2,2-dimethyl-2 <i>H</i> ,6 <i>H</i> -pyrano[3,2- <i>b</i> ]xanthen-6-one, <b>24f</b> Isocudraniaxanthone B, <b>25f</b> 10- <i>O</i> -Methylmacluraxanthone, <b>50f</b>	Boonnak <i>et al.</i> , 2006
<i>C. maingayi</i>	Stem bark	Gerontoxanthone I, <b>26f</b> Macluraxanthone, <b>54f</b> Formoxanthone C, <b>61f</b>	Laphookhieo <i>et al.</i> , 2009
<i>C. sumatranum</i>	Root bark	Sumatranaxanthone A, <b>36f</b>	Buana <i>et al.</i> , 2009

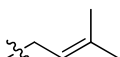
## Structure

### a: Anthraquinones



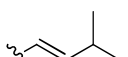
**1a:** R<sub>1</sub> = H, R<sub>2</sub> =  ; 3-Geranyloxy-6-methyl-1,8-dihydroxyanthraquinone

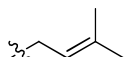
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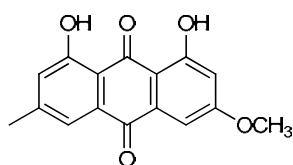
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**4a:** R<sub>1</sub> = H, R<sub>2</sub> = CH<sub>3</sub> ; Physcion

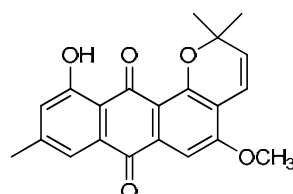
**5a:** R<sub>1</sub> = H, R<sub>2</sub> = H ; Emodin

**6a:** R<sub>1</sub> =  , R<sub>2</sub> = CH<sub>3</sub> ; Vismiaquinone

**7a:** R<sub>1</sub> =  , R<sub>2</sub> = CH<sub>3</sub> ; Vismiaquinone C

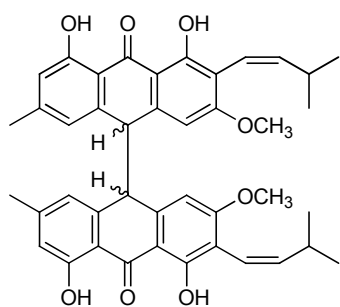
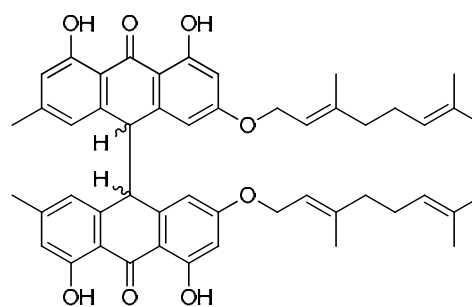
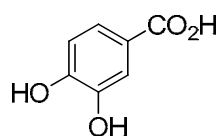
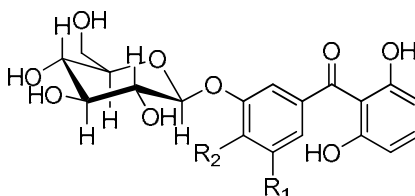


**8a:** Methoxyemodin



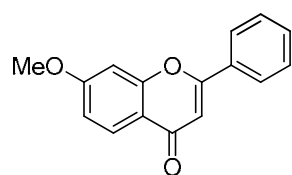
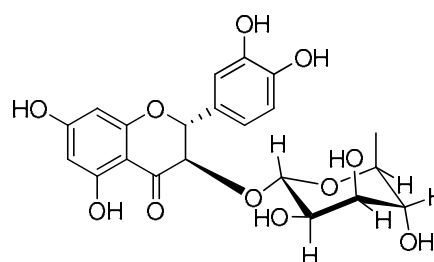
**9a:** 11-Hydroxy-5-methoxy-2,2,9-trimethyl-2H-anthra-[1,2-b]pyran-7,12-dione

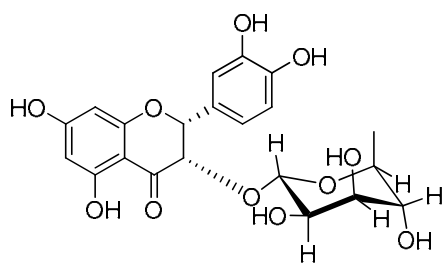
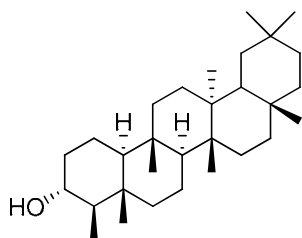
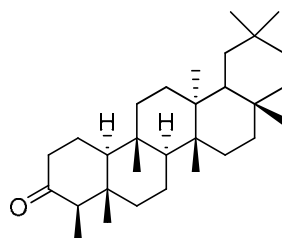
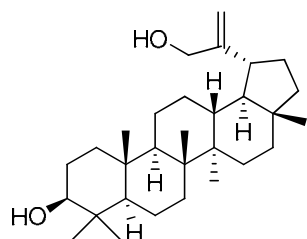
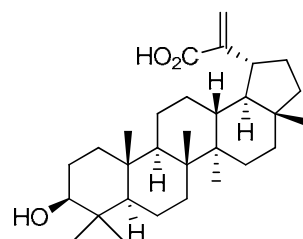
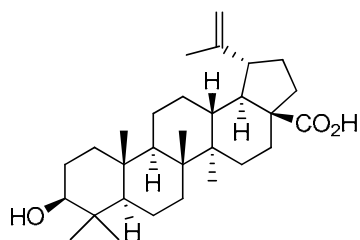


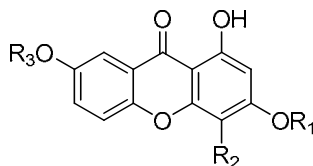
**10a:** Bianthrone J**11a:** Bianthrone A<sub>1</sub>**b: Benzenoids****1b:** 3,4-Dihydroxybenzoic acid**c: Benzophenone**

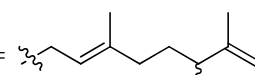
**1c:** R<sub>1</sub> = OH, R<sub>2</sub> = H; 3-O- $\beta$ -D-Glucopyranosyl-2', 5, 6'-trihydroxybenzophenone

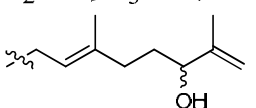
**2c:** R<sub>1</sub> = H, R<sub>2</sub> = OH; 3-O- $\beta$ -D-Glucopyranosyl-2', 4, 6'-trihydroxybenzophenone

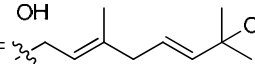
**d: Flavonoids****1d:** Tectochrysin**2d:** Astilbin

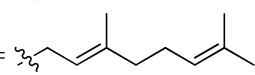
**3d:** Isoastilbin**e: Triterpenes****1e:** Friedelinol**2e:** Friedelin**3e:** Lup-20(29)-ene-3 $\beta$ , 30-diol**4e:** 3 $\beta$ -Hydroxylup-20(29)en-30-oic acid**5e:** Betulenolic acid

**f: Xanthenes**

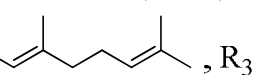
**1f:**  $R_1 = R_2 = H$ ,  $R_3 =$   ; Cochinxanthone A

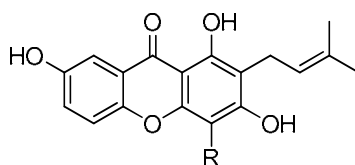
**2f:**  $R_1 =$  ,  $R_2 = H$ ,  $R_3 = H$ ; Cochinxanthone B

**3f:**  $R_1 = R_2 = H$ ,  $R_3 =$   ; Cochinxanthone C

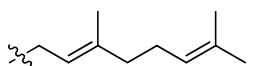
**4f:**  $R_1 = R_2 = H$ ,  $R_3 =$   ; 7-geranyloxy-1,3-dihydroxyxanthone

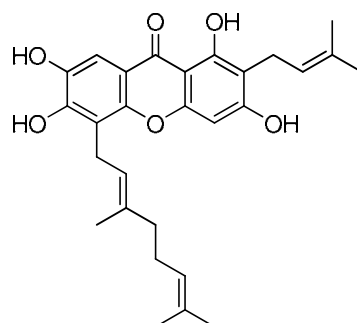
**5f:**  $R_1 = R_2 = R_3 = H$ ; 1,3,7-Trihydroxyxanthone

**6f:**  $R_1 = H$ ,  $R_2 =$  ,  $R_3 = H$ ; Fuscaxanthone

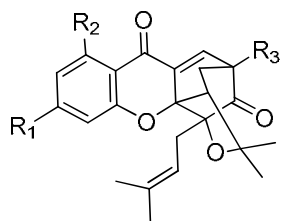


**7f:**  $R =$   ; 1,3,7-Trihydroxy-2,4-diisoprenylxanthone

**8f:**  $R =$   ; Cochinchinone A



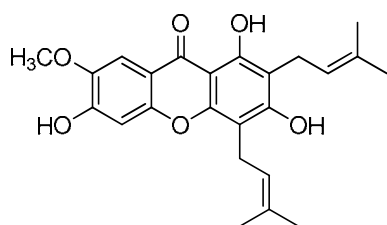
**9f:** Cochinchinone B



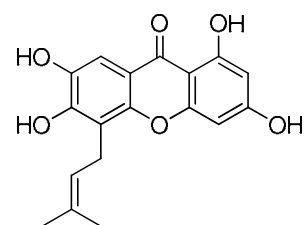
**10f:**  $R_1 = H, R_2 = OH, R_3 = OCH_3$ ; Cochinchinone C

**11f:**  $R_1 = R_2 = OH, R_3 = OCH_3$ ; Cochinchinone D

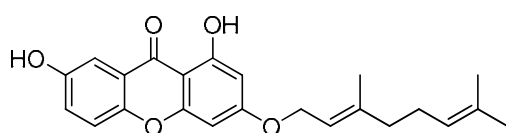
**12f:**  $R_1 = R_2 = OH, R_3 = H$ ; 4-Deprenylbratatin



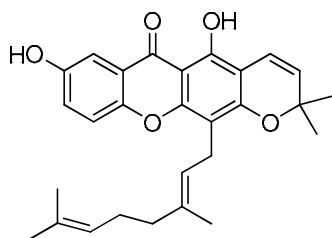
**13f:** Cochinchinone E



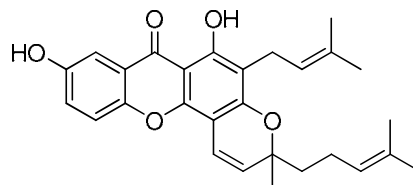
**14f:** Cochochinone F



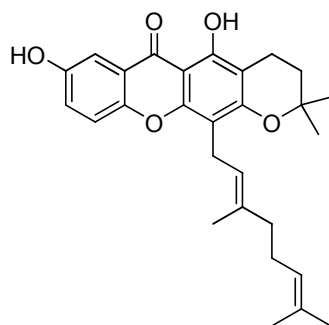
**15f:** Cochochinone G



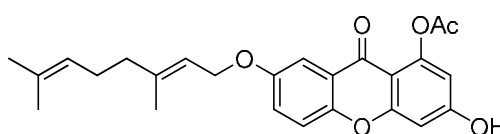
**16f:** Cochinchinone I



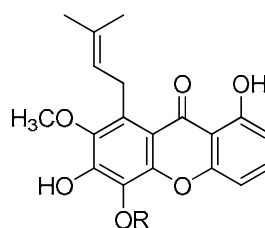
**17f:** Cochinchinone J



**18f:** Cochinchinone K

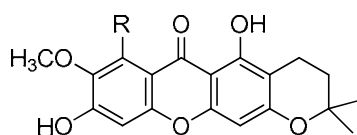


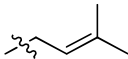
**19f:** Cochinchinone L

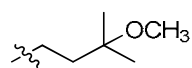


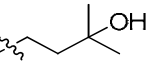
**20f:** R = CH<sub>3</sub>; 5-*O*-Methylcelebixanthone

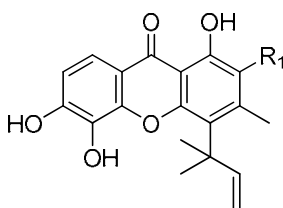
**21f:** R = H; Celebixanthone




**22f:** R =  ; 3-Isomangostin

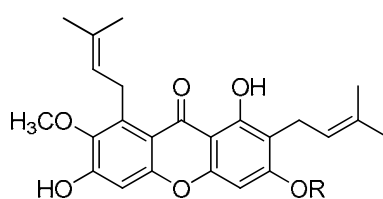
**23f:** R =  ; 3,4-Dihydro-5,9-dihydroxy-8-methoxy-7-(3-methoxy-3-methylbutyl)-2,2-dimethyl-2*H*,6*H*-pyrano-[3,2-*b*]xanthen-6-one

**24f:** R =  ; 3,4-Dihydro-5,9-dihydroxy-7-(3-hydroxy-3-methylbutyl)-8-methoxy-2,2-dimethyl-2*H*,6*H*-pyrano-[3,2-*b*]xanthen-6-one



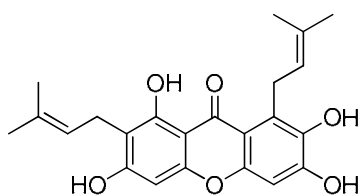
**25f:** R<sub>1</sub> = H, R<sub>2</sub> = CH<sub>3</sub>; Isocudraniaxanthone B

**26f:** R<sub>1</sub> = , R<sub>2</sub> = H; Gerontoxanthone I

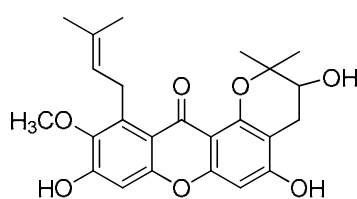


**27f:** R=H;  $\alpha$ -Mangostin

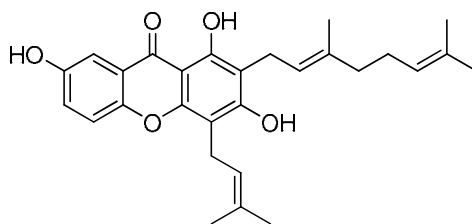
**28f:** R=CH<sub>3</sub>;  $\beta$ -Mangostin



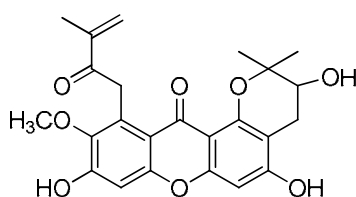
**29f:** Dulcisxanthone B



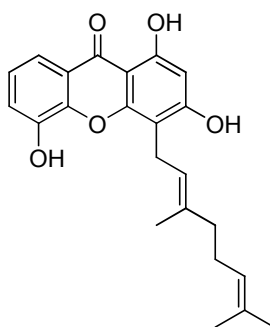
**30f:** (+)-6-Hydroxy-3,7-dimethoxy-8-(3-methylbut-2-enyl)-6,6'-dimethyl-5'-hydroxy-4',5'-dihydropyrano(2',3':1,2)xanthone



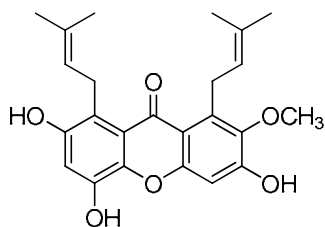
**31f:** 2-Geranyl-1,3,7-trihydroxy-4-(3-methylbut-2-enyl)xanthone



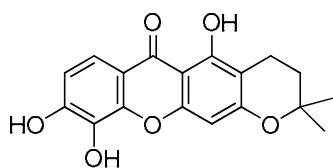
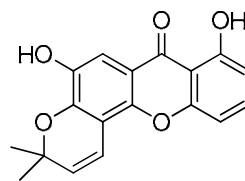
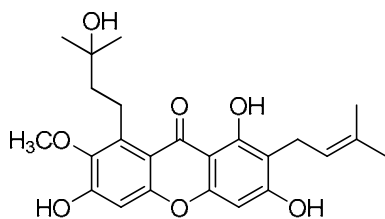
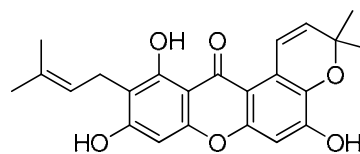
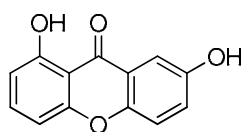
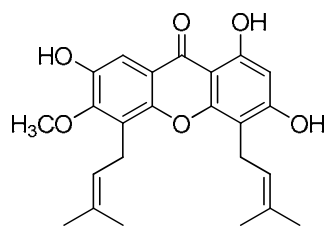
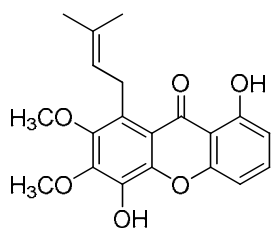
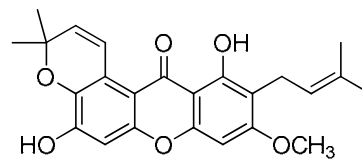
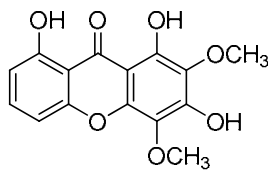
**32f:** (+)-6-Hydroxy-3,7-dimethoxy-8-(2-oxo-3-methylbut-2-enyl)-  
6,6'-dimethyl-5'-hydroxy-4',5'-dihydropyrano(2',3':1,2)xanthone



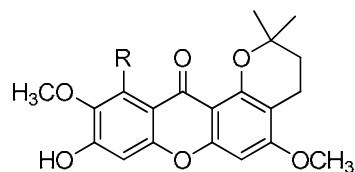
**33f:** 4-(3',7'-Dimethylocta-2',6'-dienyl)-1,3,5-trihydroxy-9H-xanthen-  
9-one



**34f:** 3,5,7-Trihydroxy-2-methoxy-1,8-bis(3-methyl-2-buten-1-yl)-9H-  
xanthen-9-one

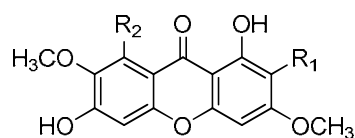
**35f:** 3,4-Dihydrojacareubin**36f:** Sumartranaxanthone A**37f:** Garcinone D**38f:** Garcinone B**39f:** Euxanthone**40f:** 1,3,7-Trihydroxy-6-methoxy-4,5-diisoprenylxanthone**41f:** Celebixanthone methyl ether**42f:** Dulxisxanthone F**43f:** 1,3,8-Trihydroxy-2,4-dimethoxyxanthone





**44f:** R = ; Pruniflorone A

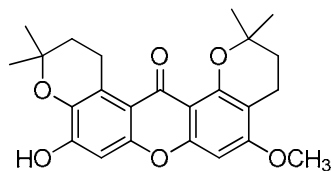
**45f:** R = ; Pruniflorone B



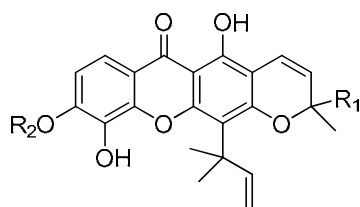
**46f:** R<sub>1</sub> = , R<sub>2</sub> = ; Pruniflorone C

**47f:** R<sub>1</sub> = , R<sub>2</sub> = ; Pruniflorone D

**48f:** R<sub>1</sub> = , R<sub>2</sub> = ; Pruniflorone E



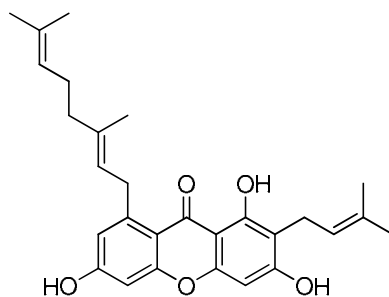
**49f:** Pruniflorone F



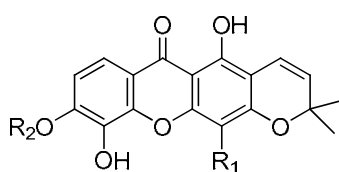
**50f:** R<sub>1</sub> = CH<sub>3</sub>, R<sub>2</sub> = CH<sub>3</sub>; 10-O-Methylmacluraxanthone


**51f:** R<sub>1</sub> = , R<sub>2</sub> = H; Pruniflorone G


**52f:** R<sub>1</sub> = , R<sub>2</sub> = CH<sub>3</sub>; Pruniflorone H



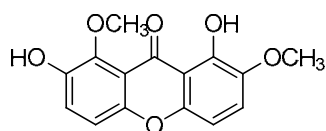
**53f:** Pruniflorone I



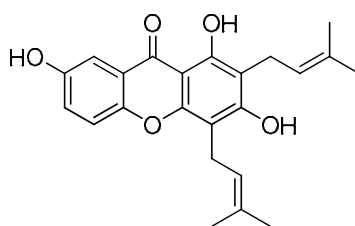
**54f:**  $R_1 =$  ,  $R_2 = H$ ; Macluraxanthone

**55f:**  $R_1 =$  ,  $R_2 = H$ ; Xanthone V<sub>1</sub>

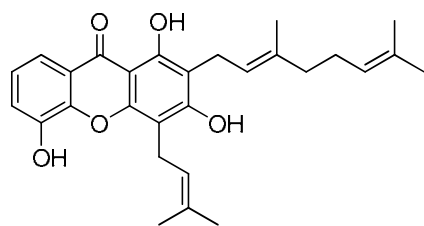
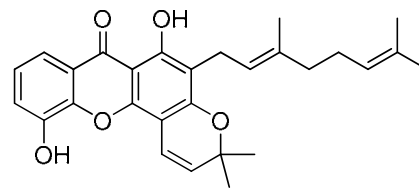
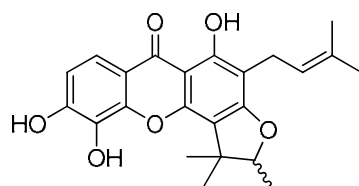
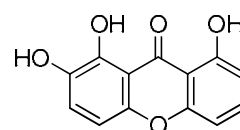
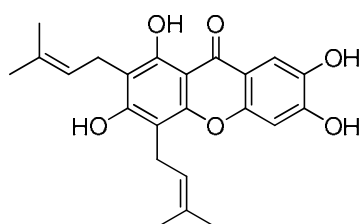
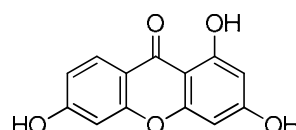
**56f:**  $R_1 = H$ ,  $R_2 = H$ ; 6-Deoxyjacareubin



**57f:** 1,7-Dihydroxy-2,8-dimethoxyxanthone



**58f:** 1,3,7-Trihydroxy-2,4-di(3-methylbut-2-enyl)xanthone

**59f:** Formoxanthone A**60f:** Formoxanthone B**61f:** Formoxanthone C**62f:** 1,2,8-Trihydroxyxanthone**63f:** Cudraticusxanthone**64f:** Norathyriol

### **1.1.3 The objectives**

The goals of this work were to investigate the chemical constituents from the roots of *C. formosum* ssp. *formosum* and to evaluate the antibacterial and cytotoxic activities of the isolated compounds.

## CHAPTER 1.2

### EXPERIMENTAL

#### 1.2.1 Instruments and Chemicals

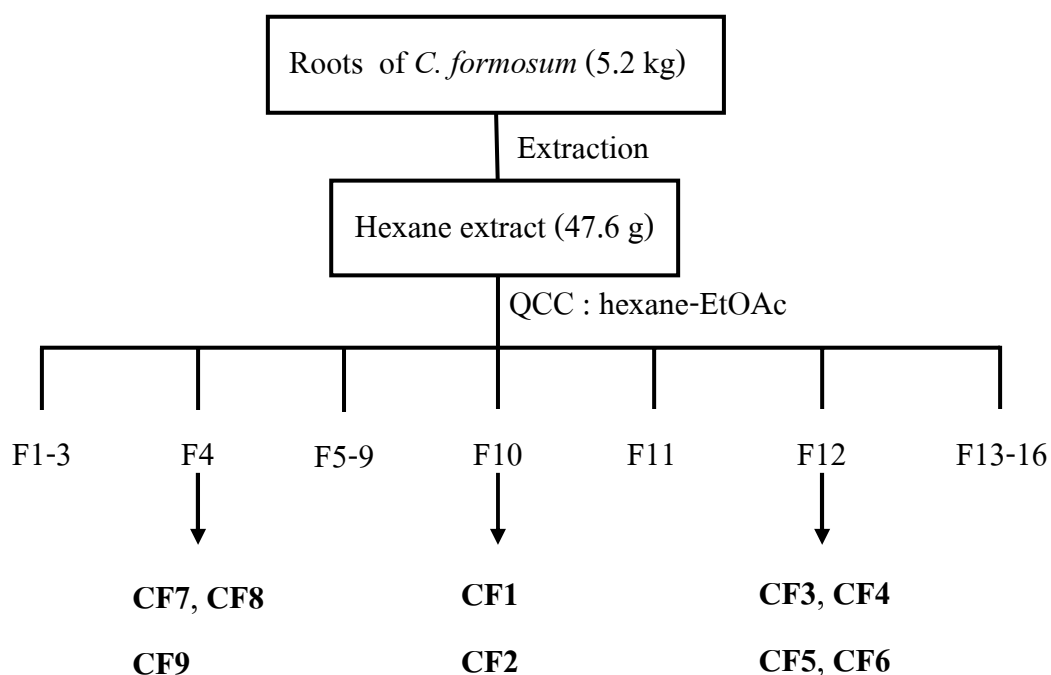
Melting point was recorded in °C on an Electrothermal 9100 melting point apparatus. Ultraviolet (UV) absorption spectra were recorded using a SPECORD S100 spectrophotometer (Analytikjena) and principle bands ( $\lambda_{\max}$ ) were recorded as wavelengths (nm) and  $\log \varepsilon$  in methanol solution. The infrared spectra were recorded using FTS 165 FT-IR Perkin Elmer spectrophotometer. Nuclear Magnetic resonance spectra were recorded using Bruker Avance 300 MHz Bruker FTNMR Ultra Shield<sup>TM</sup>. Spectra were recorded in deuteriochloroform, deuterioacetone and deuteromethanol and were recorded as  $\delta$  value in ppm downfield from TMS (Internal standard  $\delta$  0.00). Optical rotation was measured in MeOH solution at the sodium D line (590 nm) on an AUTOPOL<sup>R</sup> II automatic polarimeter. The EI-MS and HREIMS mass spectra were obtained from a Micromass LCT mass spectrometer. Solvent for extraction and chromatography were distilled at their boiling point ranges prior to use except chloroform was analytical grade reagent. Quick column chromatography (QCC) and column chromatography (CC) were carried out on silica gel 60 F<sub>254</sub> (Merck) and silica gel 100, respectively. Precoated plates of silica gel 60 GF<sub>254</sub> were used for analytical purposes.

#### 1.2.2 Plant Material

The roots of *C. formosum* were collected from Nong Khai Province, Thailand, in March 2004. The plant was identified by Prof. Puangpen Sirirugsa and a voucher specimen (no. PSU 0012676) has been deposited at the Herbarium of Department of Biology, Prince of Songkla University (PSU).

### 1.2.3 Extraction and chemical investigation of the crude hexane extract from the roots of *C. formosum*

Air-dried roots (5.2 kg) were chopped and extracted with hexane (each  $3 \times 15$  L) at room temperature for three days. Evaporation of the solvent under reduced pressure furnished a crude hexane extract (47.6 g).



**Scheme 1** Extraction and isolation of compounds **CF1-CF9** from the root of *C. formosum*

The crude hexane extract was subjected to quick column chromatography on silica gel with solvent mixtures of increasing polarity [hexane to EtOAc-hexane (9:1)] to yield sixteen fractions (1-16). Fraction 12 was chromatographed on silica gel column being eluted with solvents of increasing polarity using hexane and EtOAc, to yield sixteen subfractions (12A-12P). Crystallization of subfraction 12H from an acetone-hexane mixture (1:4) gave **CF5** (43.1 mg) as yellow needles. Subfraction 12K, upon standing overnight gave yellow needles of **CF6** (36.4 mg). Subfraction 12L was further purified by prep. TLC on silica gel, eluting with acetone in  $\text{CH}_2\text{Cl}_2$  (1:99), to yield **CF3** (5.7 mg) and **CF4** (10.6

mg). Fraction 4 was chromatographed on a silica gel column, eluting with solvent mixtures of increasing polarity, (3-10% EtOAc-hexane) to afford twelve subfractions (4A-4L). Subfractions 4A, 4E and 4H were further purified by crystallization from MeOH-CH<sub>2</sub>Cl<sub>2</sub> (1:4) to give **CF7** (9.6 mg), **CF8** (17.1 mg) and **CF9** (6.2 mg). Fraction 10 was subjected to repeated column chromatography over silica gel to afford **CF1** (31.7 mg) and **CF2** (4.6 mg).

**Compound CF1:** Yellow solid ; mp 111-113 °C; UV (MeOH)  $\lambda_{\max}$  (log  $\epsilon$ ): 245 (4.39), 260(sh) (4.29), 319 (4.08), 367 (3.50) nm; IR (neat)  $\nu_{\max}$ : 3373, 2974, 1650 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz) and <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz), see **Table 2**; MS  $m/z$  (rel. int.): 448[M]<sup>+</sup> (7), 363 (40), 341 (46), 323 (87), 281 (86), 269 (100); HREIMS  $m/z$  448.2224 [M]<sup>+</sup> (calcd. for C<sub>28</sub>H<sub>32</sub>O<sub>5</sub>, 448.2250)

**Compound CF2:** Yellow solid; mp 143-146 °C; UV (MeOH)  $\lambda_{\max}$  (log  $\epsilon$ ): 253 (4.15), 269 (4.11), 332 (3.71), 377 (3.18) nm; IR (KBr)  $\nu_{\max}$ : 3426, 1646 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz) and <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz), see **Table 3**; MS  $m/z$  (rel. int.): 446[M]<sup>+</sup> (55), 431 (37), 377 (72), 323 (100), 309 (21), 295 (18); HREIMS  $m/z$  446.2061 [M]<sup>+</sup> (calcd. for C<sub>28</sub>H<sub>30</sub>O<sub>5</sub>, 446.2093)

**Compound CF3:** Yellow solid; mp 152-154 °C;  $[\alpha]_D^{29} = -44^\circ$  (CHCl<sub>3</sub>, c 0.05); UV (MeOH)  $\lambda_{\max}$  (log  $\epsilon$ ): 258 (4.51), 276 (4.44), 392 (3.85) nm; IR (KBr)  $\nu_{\max}$ : 3440, 1646, 1624, 1598 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz) and <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz), see **Table 5**; MS  $m/z$  (rel. int.): 396 [M]<sup>+</sup> (40), 381(43), 353 (30), 341 (100), 325 (26), 311 (15), 285 (14); HREIMS  $m/z$  396.1559 [M]<sup>+</sup> (calcd. for C<sub>23</sub>H<sub>24</sub>O<sub>6</sub>, 396.1573)

**Compound CF4:** Yellow solid; mp 137-139 °C; UV (MeOH)  $\lambda_{\max}$  (log  $\epsilon$ ): 204 (4.26), 253 (4.42), 328 (4.09), 387 (3.92) nm; IR (KBr)  $\nu_{\max}$ : 3380, 1613, 1584 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz) and <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz), see **Table 6**.

**Compound CF5:** Yellow needles; mp 183-184 °C; UV (MeOH)  $\lambda_{\max}$  nm (log  $\epsilon$ ): 241 (4.28), 283 (4.62), 338 (4.25) nm; IR (KBr)  $\nu_{\max}$ : 3447, 1650, 1583  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz) and  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz), see **Table 8**.

**Compound CF6:** Yellow needles; mp 218-219 °C; UV (MeOH)  $\lambda_{\max}$  (log  $\epsilon$ ): 282 (4.80), 337 (4.44) nm; IR (KBr)  $\nu_{\max}$ : 3358, 1646, 1624, 1609  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz) and  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz), see **Table 10**.

**Compound CF7:** Reddish orange solid; mp 135-138 °C; UV (MeOH)  $\lambda_{\max}$  (log  $\epsilon$ ): 226 (4.06), 254 (3.79), 266 (3.78), 288 (3.76), 437 (3.54) nm; IR (KBr)  $\nu_{\max}$ : 3409, 1628, 1609  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz) and  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz), see **Table 14**.

**Compound CF8:** Reddish orange solid; mp 179-18 °C; UV (MeOH)  $\lambda_{\max}$  (log  $\epsilon$ ): 221 (4.30), 253 (4.04), 266 (4.04), 287 (4.02), 438 (3.82) nm; IR (KBr)  $\nu_{\max}$ : 1628, 1609  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz) and  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz), see **Table 16**.

**Compound CF9:** Reddish orange solid; mp 186-188 °C; UV (MeOH)  $\lambda_{\max}$  (log  $\epsilon$ ): 221 (4.48), 263 (4.32), 292 (4.43), 307 (4.33)*sh*, 442 (4.11) nm; IR (KBr)  $\nu_{\max}$ : 1624  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz) and  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz), see **Table 18**.

## 1.2.4 BIOASSAY

### 1.2.4.1 Antibacterial assay

The compounds isolated from *C. formosum* were tested against the microorganisms *Bacillus subtilis* (obtained from Department of Industrial Biotechnology, PSU), *Staphylococcus aureus* (TISTR517) (obtained from Microbial



Resources Center (MIRCEN), Bangkok, Thailand), *Pseudomonas aeruginosa*, *Enterococcus faecalis*, *Shigella sonnei* and *Salmonella typhi*. The last four microorganisms were obtained from Department of Pharmacognosy and Botany, PSU. The antibacterial assay employed was the same as described in Boonsri *et al.* (Boonsri *et al.*, 2006). Vancomycin, which was used as a standard, showed antibacterial activity of 0.078  $\mu\text{g/mL}$ .

#### **1.2.4.2 Cytotoxic assay**

The procedure for the cytotoxic assay was performed by the sulphorhodamine B (SRB) assay as described by Skehan *et al.* (Skehan *et al.*, 1990). In this study, four cancer cell lines obtained from the National Cancer Institute, Bangkok, Thailand, were used: MCF-7 (breast adenocarcinoma), KB (human oral cancer), HeLa (human cervical cancer) and HT-29 (colon cancer). Camptothecin, which was used as a standard, showed cytotoxic activity in the range of 0.2-2.0  $\mu\text{g/mL}$ .

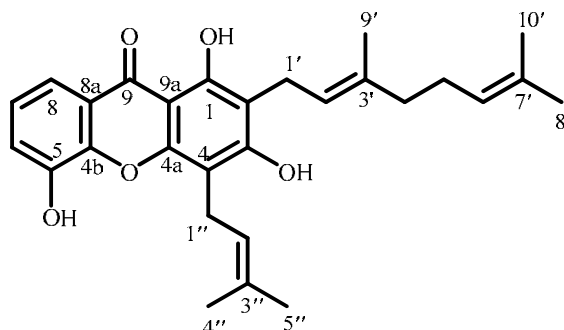
## CHAPTER 1.3

### RESULTS AND DISCUSSION

#### 1.3.1 Structural elucidation of the isolated compounds from the root of *C. formosum*

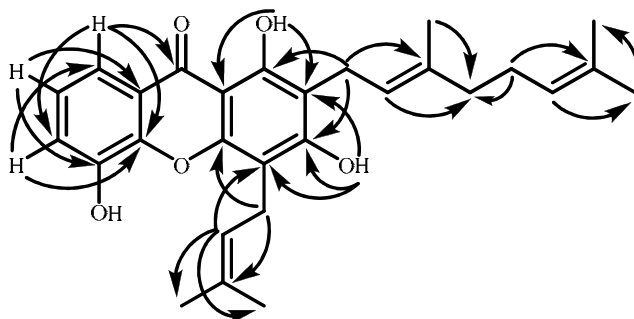
The crude hexane extract from the roots of *C. formosum* was subjected to a succession of chromatographic procedures, including silica gel column chromatography and preparative TLC to afford three new compounds, **CF1-CF3** together with six known compounds **CF4-CF9**. All structures were elucidated using 1D and 2D NMR spectroscopic data and comparison with those reported in the literatures.

### 1.3.1.1 Compound CF1



**CF1** was obtained as a yellow solid. The HREIMS spectrum showed a molecular ion peak at  $m/z$  448.2224, corresponding to  $C_{28}H_{32}O_5$ . The IR spectrum (**Figure 6**) of **1** exhibited strong absorption bands due to hydroxyl ( $3373\text{ cm}^{-1}$ ) and a conjugated carbonyl groups ( $1650\text{ cm}^{-1}$ ). The UV absorption bands (245, 260 $sh$ , 319 and 367 nm) (**Figure 5**) were typical of a xanthone chromophore (Seo *et al.*, 2002; Ito *et al.*, 2003). The  $^{13}\text{C}$  NMR and DEPT spectral data (**Table 2, Figure 8**) disclosed the presence of one carbonyl carbon ( $\delta$  181.1), twelve  $sp^2$  quaternary carbons (five of which were oxygen-bearing) ( $\delta$  103.3, 105.7, 109.0, 120.9, 132.1, 133.1, 140.1, 144.3, 144.5, 152.5, 158.6, 161.0), six  $sp^2$  methines ( $\delta$  116.9, 119.8, 121.1, 122.4, 123.7, 123.8), four  $sp^3$  methylenes ( $\delta$  21.6, 22.0, 26.3, 39.7), and five methyl carbons ( $\delta$  16.3, 17.7, 17.9, 25.6, 25.7). The  $^1\text{H}$  NMR spectrum of **1** (**Table 2, Figure 7**) contained resonances for one chelated [ $\delta$  13.18 (1H, *s*, 1-OH)] and two free hydroxyl groups [ $\delta$  6.59 (1H, *s*, 3-OH) and  $\delta$  5.84, (1H, *s*, 5-OH)]. A 1,2,3-trisubstituted benzene ring was revealed by resonances at  $\delta$  7.75 (1H, *dd*,  $J = 7.8, 1.5$  Hz, H-8), 7.28 (1H, *dd*,  $J = 7.8, 1.5$  Hz, H-6) and 7.21 (1H, *t*,  $J = 7.8$  Hz, H-7). The lowest-field aromatic-proton ( $\delta$  7.75) was assigned to H-8 due to the anisotropic effect of the carbonyl group and this was supported by the HMBC correlations of H-8 to a carbonyl carbon at  $\delta$  181.1 (C-9),  $\delta$  119.8 (C-6) and  $\delta$  144.3 (C-4b), as well as those of H-7 to  $\delta$  144.5 (C-5) and  $\delta$  120.9 (C-8a) and of H-6 to  $\delta$  116.9 (C-8). Furthermore, the  $^1\text{H}$  NMR spectra displayed a geranyl moiety at  $\delta$  1.60 (3H, *s*, H-10'), 1.69 (3H, *s*, H-8'), 1.85 (3H, *s*, H-9'), 2.11 (4H, *m*, H-4', H-5'), 3.49 (2H, *d*,  $J = 7.2$  Hz, H-1'), 5.06

(1H, *m*, H-6') and 5.30 (1H, *m*, H-2'), and a prenyl moiety at  $\delta$ 1.74 (3H, *d*,  $J = 1.2$  Hz, H-4''), 1.86 (3H, *s*, H-5''), 3.53 (2H, *d*,  $J = 6.9$  Hz, H-1'') and 5.25 (1H, *m*, H-2''). In the HMBC spectrum, the chelated hydroxyl proton ( $\delta$ 13.18) showed correlations with C-1 ( $\delta$ 158.6), C-2 ( $\delta$ 109.0) and C-9a ( $\delta$ 103.3), the benzylic allylic methylene protons ( $\delta$ 3.49, H-1') of the geranyl group showed cross peak with C-1 ( $\delta$ 158.6), C-2 ( $\delta$ 109.0) and C-3 ( $\delta$ 161.0) and the allylic methylene protons of the prenyl group at  $\delta$ 3.53 (H-1'') showed the correlations with C-3 ( $\delta$ 161.0) and C-4a ( $\delta$ 152.5), indicating that the geranyl and the prenyl moieties were located at C-2 and C-4, respectively. Therefore, compound **1** was identified as 1,3,5-trihydroxy-2-(3,7-dimethylocta-2,6-dienyl)-4-(3-methylbut-2-enyl)xanthone, a new compound and named as formoxanthone A (Boonsri *et al.*, 2006) which is the isomer of 2-geranyl-1,3,7-trihydroxy-4-(3,3-dimethylallyl)xanthone previously isolated from *C. cochinchinense* (Bennett *et al.*, 1993).



Selected HMBC correlations of **CF1**

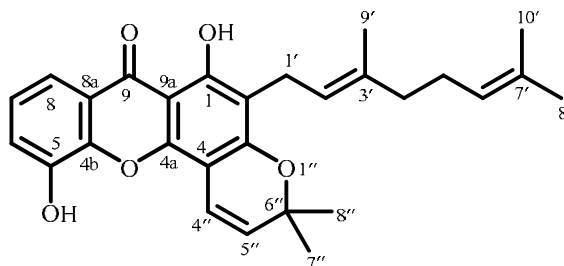
**Table 2**  $^1\text{H}$ ,  $^{13}\text{C}$  NMR, DEPT and HMBC spectral data of **CF1**

Position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	DEPT	HMBC
1		158.6	C	
2		109.0	C	
3		161.0	C	
4		105.7	C	
4a		152.5	C	
4b		144.3	C	
5		144.5	C	
6	7.28 ( <i>dd</i> , 7.8, 1.5)	119.8	CH	5, 8
7	7.21 ( <i>t</i> , 7.8)	123.8	CH	5, 8a
8	7.75 ( <i>dd</i> , 7.8, 1.5)	116.9	CH	4b, 6, 9
8a		120.9	C	
9		181.1	C	
9a		103.3	C	
1'	3.49 ( <i>d</i> , 7.2)	21.6	CH <sub>2</sub>	1, 2, 3, 2', 3'
2'	5.30 ( <i>m</i> )	121.1	CH	2, 1', 4', 9'
3'		140.1	C	
4'	2.11 ( <i>m</i> )	39.7	CH <sub>2</sub>	9'
5'	2.11 ( <i>m</i> )	26.3	CH <sub>2</sub>	3', 7'
6'	5.06 ( <i>m</i> )	123.7	CH	5', 8'
7'		132.1	C	
8'	1.69 ( <i>s</i> )	25.7	CH <sub>3</sub>	6', 7'
9'	1.85 ( <i>s</i> )	16.3	CH <sub>3</sub>	2', 4'
10'	1.60 ( <i>s</i> )	17.7	CH <sub>3</sub>	6', 7'
1''	3.53 ( <i>d</i> , 6.9)	22.0	CH <sub>2</sub>	3, 4, 4a, 2'', 3''
2''	5.25 ( <i>m</i> )	122.4	CH	4, 4''
3''		133.1	C	
4''	1.74 ( <i>d</i> , 1.2)	25.6	CH <sub>3</sub>	2'', 3'', 5''

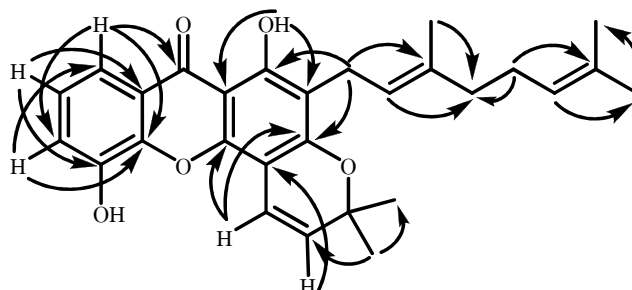
**Table 2** (Continued)

<b>Position</b>	<b><math>\delta_{\text{H}}</math> (<i>mult.</i>, <math>J_{\text{Hz}}</math>)</b>	<b><math>\delta_{\text{C}}</math></b>	<b>DEPT</b>	<b>HMBC</b>
5''	1.86 ( <i>s</i> )	17.9	CH <sub>3</sub>	2'', 3'', 4''
1-OH	13.18 ( <i>s</i> )			1, 2, 9a
3-OH	6.59 ( <i>s</i> )			2, 3, 4
5-OH	5.84 ( <i>s</i> )			4b, 6

### 1.3.1.2 Compound CF2



**CF2**, a yellow solid, gave a HREIMS molecular ion peak at  $m/z$  446.2061 corresponding to a molecular formula  $C_{28}H_{30}O_5$ . The IR (**Figure 9**) and UV spectra of **2** exhibited the same pattern as those of **1**. The  $^1H$  NMR spectrum of **CF2** (**Table 3**, **Figure 9**) was similar to that of **CF1** except for the replacement of the prenyl group in **CF1** with the characteristic signals of a chromene ring, two vinylic protons at  $\delta$  6.79 and 5.64 (each,  $d$ ,  $J = 9.9$  Hz, H-4'', H-5'', respectively) and a methyl signal at  $\delta$  1.49 (6H,  $s$ , Me-7'', Me-8'') (**Table 3**). The dimethylchromene group was connected to ring A at C-3 and C-4 as evidenced by HMBC correlations of the vinylic proton at  $\delta$  6.79 (H-4'') with C-3 ( $\delta$  158.7), C-4 ( $\delta$  100.6) and C-4a ( $\delta$  149.2). Thus, compound **2** was characterized as 1,5-dihydroxy-2-(3,7-dimethylocta-2,6-dienyl)-6'',6''-dimethylpyrano(2'',3'':3,4)xanthone, a new compound and named as formoxanthone B (Boonsri *et al.*, 2006).



Selected HMBC correlations of **CF2**

**Table 3**  $^1\text{H}$ ,  $^{13}\text{C}$  NMR, DEPT and HMBC spectral data of **CF2**

Position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	DEPT	HMBC
1		160.6	C	
2		112.3	C	
3		158.7	C	
4		100.6	C	
4a		149.2	C	
4b		144.1	C	
5		144.3	C	
6	7.30 ( <i>dd</i> , 7.8, 1.5)	120.1	CH	4b, 8
7	7.23 ( <i>t</i> , 7.8)	123.9	CH	5, 8a
8	7.78 ( <i>dd</i> , 7.8, 1.5)	117.2	CH	4b, 6, 9
8a		121.2	C	
9		180.8	C	
9a		103.2	C	
1'	3.37 ( <i>d</i> , 7.5)	21.1	CH <sub>2</sub>	1, 2, 3, 2', 3'
2'	5.25 ( <i>m</i> )	121.7	CH	1', 4', 9'
3'		135.2	C	
4'	2.00 ( <i>m</i> )	39.8	CH <sub>2</sub>	2', 3'
5'	2.05 ( <i>m</i> )	26.7	CH <sub>2</sub>	4', 6', 7'
6'	5.08 ( <i>m</i> )	124.4	CH	8', 10'
7'		131.3	C	
8'	1.64 ( <i>br s</i> )	25.7	CH <sub>3</sub>	6', 7'
9'	1.82 ( <i>s</i> )	16.3	CH <sub>3</sub>	2', 3', 4'
10'	1.57 ( <i>br s</i> )	17.7	CH <sub>3</sub>	6', 7', 8'
1''				
2''				
3''				
4''	6.79 ( <i>d</i> , 9.9)	115.0	CH	3, 4, 4a, 6''
5''	5.64 ( <i>d</i> , 9.9)	127.4	CH	4, 6''



**Table 3** (Continued)

Position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	DEPT	HMBC
6''		78.1	C	
7''	1.49 ( <i>s</i> )	28.2	CH <sub>3</sub>	5'', 6'', 8''
8''	1.49 ( <i>s</i> )	28.2	CH <sub>3</sub>	5'', 6'', 7''
1-OH	13.20 ( <i>s</i> )			1, 2, 3, 9a
5-OH	5.71 ( <i>s</i> )			

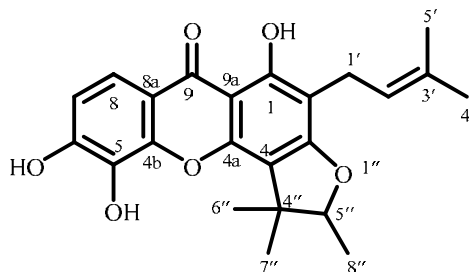
**Table 4** Comparison of <sup>1</sup>H and <sup>13</sup>C NMR spectral data of CF1 and CF2

Position	CF1		CF2	
	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$ (C-Type)	$\delta_{\text{H}}$ , ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$ (C-Type)
1		158.6 (C)		160.6 (C)
2		109.0 (C)		112.3 (C)
3		161.0 (C)		158.7 (C)
4		105.7 (C)		100.6 (C)
4a		152.5 (C)		149.2 (C)
4b		144.3 (C)		144.1 (C)
5		144.5 (C)		144.3 (C)
6	7.28 ( <i>dd</i> , 7.8, 1.5)	119.8 (CH)	7.30 ( <i>dd</i> , 7.8, 1.5)	120.1 (CH)
7	7.21 ( <i>t</i> , 7.8)	123.8 (CH)	7.23 ( <i>t</i> , 7.8)	123.9 (CH)
8	7.75 ( <i>dd</i> , 7.8, 1.5)	116.9 (CH)	7.78 ( <i>dd</i> , 7.8, 1.5)	117.2 (CH)
8a		120.9 (C)		121.2 (C)
1'	3.49 ( <i>d</i> , 7.2)	21.6 (CH <sub>2</sub> )	3.37 ( <i>d</i> , 7.5)	21.1 (CH <sub>2</sub> )
2'	5.30 ( <i>m</i> )	121.1 (CH)	5.25 ( <i>m</i> )	121.7 (CH)
3'		140.1 (C)		135.2 (C)
4'	2.11 ( <i>m</i> )	39.7 (CH <sub>2</sub> )	2.00 ( <i>m</i> )	39.8 (CH <sub>2</sub> )
5'	2.11 ( <i>m</i> )	26.3 (CH <sub>2</sub> )	2.05 ( <i>m</i> )	26.7 (CH <sub>2</sub> )
6'	5.06 ( <i>m</i> )	123.7 (CH)	5.08 ( <i>m</i> )	124.4 (CH)

Table 4 (Continued)

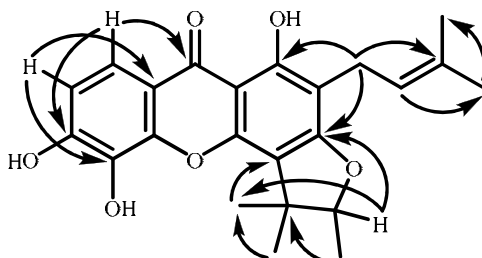
Position	CF1		CF2	
	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$ (C-Type)	$\delta_{\text{H}}$ , ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$ (C-Type)
7'		132.1 (C)		131.3 (C)
8'	1.69 ( <i>s</i> )	25.7 (CH <sub>3</sub> )	1.64 ( <i>br s</i> )	25.7 (CH <sub>3</sub> )
9'	1.85 ( <i>s</i> )	16.3 (CH <sub>3</sub> )	1.82 ( <i>s</i> )	16.3 (CH <sub>3</sub> )
10'	1.60 ( <i>s</i> )	17.7 (CH <sub>3</sub> )	1.57 ( <i>br s</i> )	17.7 (CH <sub>3</sub> )
1''	3.53 ( <i>d</i> , 6.9)	22.0 (CH <sub>2</sub> )		
2''	5.25 ( <i>m</i> )	122.4 (CH)		
3''		133.1 (C)		
4''	1.74 ( <i>d</i> , 1.2)	25.6 (CH <sub>3</sub> )	6.79 ( <i>d</i> , 9.9)	115.0 (CH)
5''	1.86 ( <i>s</i> )	17.9 (CH <sub>3</sub> )	5.64 ( <i>d</i> , 9.9)	127.4 (CH)
6''				78.1 (C)
7''			1.49 ( <i>s</i> )	28.2 (CH <sub>3</sub> )
8''			1.49 ( <i>s</i> )	28.2 (CH <sub>3</sub> )
1-OH	13.18 ( <i>s</i> )		13.20 ( <i>s</i> )	
3-OH	6.59 ( <i>s</i> )			
5-OH	5.84 ( <i>s</i> )		5.71 ( <i>s</i> )	

### 1.3.1.3 Compound CF3



**CF3** was obtained as a yellow solid and the HREIMS spectrum showed a molecular ion peak at  $m/z$  396.1559 consistent with the molecular formula  $C_{23}H_{24}O_6$ . The UV (**Figure 12**) and IR (**Figure 13**) spectra suggested that **3** was also a xanthone derivative (Seo *et al.*, 2002; Ito *et al.*, 2003). The  $^1H$  NMR spectral data of **3** (**Table 5**, **Figure 14**) consisted of one chelated hydroxyl signal at  $\delta$  13.40 and two *ortho*-coupled aromatic signals at  $\delta$  6.92 and 7.74 (1H each, *d*,  $J = 7.7$  Hz, H-7, H-8, respectively). The presence of a prenyl group was evident from the two vinylic methyl signals at  $\delta$  1.69 (3H, *s*, Me-4') and 1.79 (3H, *s*, Me-5'), one methylene doublet at  $\delta$  3.31 (2H, *d*,  $J = 6.9$  Hz, H-1') and a vinylic proton signal at  $\delta$  5.29 (1H, *m*, H-2'). Furthermore, signals of an  $\alpha,\alpha,\beta$ -trimethylfuran ring which comprised of protons resonating at  $\delta$  1.32 (3H, *s*, Me-6''), 1.43 (3H, *d*,  $J = 6.3$  Hz, Me-8''), 1.58 (3H, *s*, Me-7'') and 4.54 (1H, *q*,  $J = 6.3$  Hz, H-5'') were displayed. In the HMBC spectrum, the methylene signal at  $\delta$  3.31 (H-1') showed cross peaks with oxygenated aromatic carbons at  $\delta$  161.3 (C-1) and  $\delta$  164.3 (C-3), indicating that a prenyl group was connected to the C-2 position. In addition, the oxygenated methine proton signal at  $\delta$  4.54 (H-5'') showed a correlation with C-3 ( $\delta$  164.3) and the methyl groups at  $\delta$  1.32 and 1.58 were correlated with C-4 ( $\delta$  112.1). These observations suggested that the furan ring was fused at C-3 and C-4. The relative stereostructure of the trimethylfuran ring was postulated from NOESY cross peaks of the oxygenated methine proton ( $\delta$  4.54, H-5'') with the methyl groups at  $\delta$  1.43 (Me-8'') and 1.58 (Me-7'') and the methyl doublet at  $\delta$  1.43 (Me-8'') with the methyl group at  $\delta$  1.32 (Me-6''). Therefore, compound **3** was identified as 4'',5''-dihydro-1,5,6-trihydroxy-2-(3-methylbut-2-enyl)-

4'',4'',5''-trimethylfurano(2'',3''':3,4)xanthone, a new compound and named as formoxanthone C (Boonsri *et al.*, 2006).



Selected HMBC correlations of **CF3**

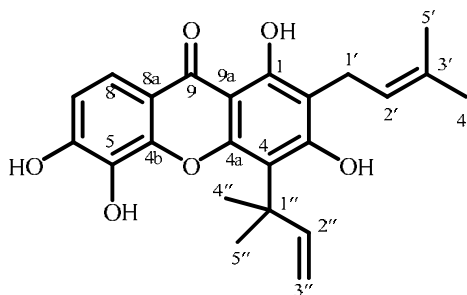
**Table 5**  $^1\text{H}$ ,  $^{13}\text{C}$  NMR, DEPT and HMBC spectral data of **CF3**

Position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	DEPT	HMBC
1		161.3	C	
2		107.3	C	
3		164.3	C	
4		112.1	C	
4a		150.6	C	
4b		145.1	C	
5		130.6	C	
6		149.2	C	
7	6.92 ( <i>d</i> , 7.7)	112.2	CH	5, 6, 8a
8	7.74 ( <i>d</i> , 7.7)	118.3	CH	6, 9
8a		114.6	C	
9		180.1	C	
9a		103.0	C	
1'	3.31 ( <i>d</i> , 6.9)	21.8	CH <sub>2</sub>	1, 2, 3, 2', 3'
2'	5.29 ( <i>m</i> )	121.6	CH	1', 4', 5'
3'		132.2	C	
4'	1.69 ( <i>s</i> )	25.8	CH <sub>3</sub>	2', 3', 5'

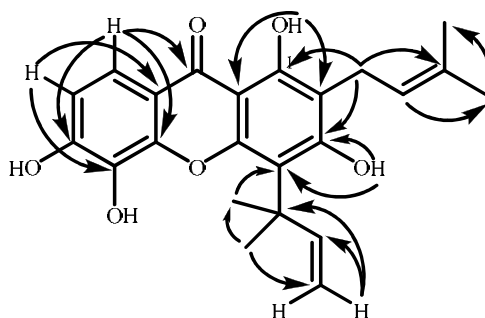
**Table 5** (Continued)

<b>Position</b>	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	<b>DEPT</b>	<b>HMBC</b>
5'	1.79 ( <i>s</i> )	17.8	CH <sub>3</sub>	2', 3', 4'
4''		44.1	C	
5''	4.54 ( <i>q</i> , 6.3)	90.3	CH	3, 4'', 6'', 7''
6''	1.32 ( <i>s</i> )	21.7	CH <sub>3</sub>	4, 4'', 5'', 7''
7''	1.58 ( <i>s</i> )	26.3	CH <sub>3</sub>	4, 4'', 5'', 6''
8''	1.43 ( <i>d</i> , 6.3)	14.4	CH <sub>3</sub>	4'', 5''
1-OH	13.40 ( <i>s</i> )			

### 1.3.1.4 Compound CF4



**CF4** appeared as a yellow solid. The UV (**Figure 16**) and IR (**Figure 17**) spectra closely resembled to those of **CF3**. The  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra (**Table 6**, **Figures 18** and **19**) exhibited signals similar to those of **CF3** except for the appearance of three olefinic protons [ $\delta$  6.88 (1H, *dd*,  $J = 17.7, 10.5$  Hz, H-2'), 5.30 (1H, *dd*,  $J = 17.7, 0.9$  Hz, H-3') and 5.15 (1H, *dd*,  $J = 10.5, 0.9$  Hz, H-3'')] of terminal olefin instead of an oxymethine proton [ $\delta$  4.54 (1H, *q*,  $J = 6.3$  Hz, H-5'')] and one methyl group [ $\delta$  1.43 (3H, *d*,  $J = 6.3$  Hz, Me-8'')] of a furan ring in **CF3**. From the spectroscopic data and comparison with those of gerontoxanthone I (Chang *et al.*, 1989), therefore, **CF4** was determined as gerontoxanthone I.



Selected HMBC correlations of **CF4**

**Table 6**  $^1\text{H}$ ,  $^{13}\text{C}$  NMR, DEPT and HMBC spectral data of **CF4**

Position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	DEPT	HMBC
1		159.0	C	
2		110.1	C	
3		161.4	C	
4		111.2	C	
4a		153.3	C	
4b		144.8	C	
5		131.0	C	
6		149.0	C	
7	6.94 ( <i>d</i> , 8.7)	111.6	CH	5, 6, 8a
8	7.70 ( <i>d</i> , 8.7)	117.2	CH	4b, 6, 9
8a		113.8	C	
9		180.3	C	
9a		103.0	C	
1'	3.47 ( <i>d</i> , 6.9)	21.6	CH <sub>2</sub>	1, 3, 2', 3'
2'	5.24 ( <i>m</i> )	121.2	CH	
3'		136.1	C	
4'	1.79, <i>d</i> , 0.9)	25.9	CH <sub>3</sub>	2', 3', 5'
5'	1.86 ( <i>br s</i> )	18.0	CH <sub>3</sub>	2', 3', 4'
1''		41.6	C	
2''	6.68 ( <i>dd</i> , 17.7, 10.5)	154.9	C	4'', 5''
3''	5.30 ( <i>dd</i> , 17.7, 0.9)	106.1	CH <sub>2</sub>	1'', 2''
	5.15 ( <i>dd</i> , 10.5, 0.9)			
4''	1.69 ( <i>s</i> )	28.0	CH <sub>3</sub>	1'', 2'', 4
5''	1.69 ( <i>s</i> )	28.0	CH <sub>3</sub>	
1-OH	13.60 ( <i>s</i> )			1, 2, 9a
3-OH	6.76 ( <i>s</i> )			3, 4

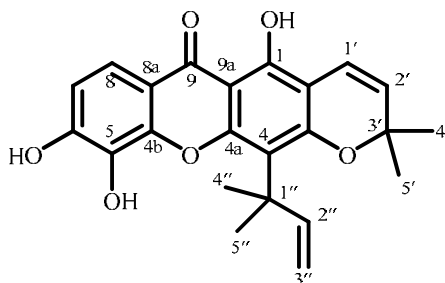
**Table 7** Comparison of  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectral data of **CF4** and gerontoxanthone I

position	CF4		gerontoxanthone I <sup>a</sup>	
	$\delta_{\text{H}}$ (mult., $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	$\delta_{\text{H}}$ (mult., $J_{\text{Hz}}$ )	$\delta_{\text{C}}$
1		159.0		160.4
2		110.1		112.5
3		161.4		161.9
4		111.2		111.9
4a		153.3		155.4
4b		144.8		147.5
5		131.0		134.2
6		149.0		152.1
7	6.94 ( <i>d</i> , 8.7)	111.6	7.01 ( <i>d</i> , 8.8)	113.9
8	7.70 ( <i>d</i> , 8.7)	117.2	7.63 ( <i>d</i> , 8.8)	117.7
8a		113.8		115.2
9		180.3		182.3
9a		103.0		104.1
1'	3.47 ( <i>d</i> , 6.9)	21.6	3.37 ( <i>d</i> , 7.0)	22.8
2'	5.24 ( <i>m</i> )	121.2	5.22 ( <i>m</i> )	123.8
3'		136.1		132.5
4'	1.79, <i>d</i> , 0.9)	25.9	1.66 ( <i>s</i> )	26.3
5'	1.86 ( <i>br s</i> )	18.0	1.66 ( <i>s</i> )	18.4
1''		41.6		42.7
2''	6.68 ( <i>dd</i> , 17.7, 10.5)	154.9	6.60 ( <i>dd</i> , 17.7, 10.4)	151.8
3''	5.30 ( <i>dd</i> , 17.7, 0.9)	106.1	5.47 ( <i>d</i> , 17.7)	112.8
	5.15 ( <i>dd</i> , 10.5, 0.9)		5.35 ( <i>d</i> , 10.4)	
4''	1.69 ( <i>s</i> )	28.0	1.81 ( <i>s</i> )	29.2
5''	1.69 ( <i>s</i> )	28.0	1.81 ( <i>s</i> )	29.2
1-OH	13.60 ( <i>s</i> )		13.86 ( <i>s</i> )	
3-OH	6.76 ( <i>s</i> )			

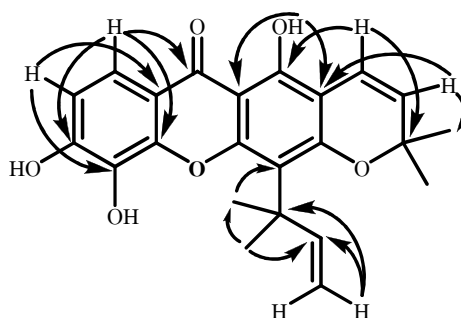
<sup>a</sup> Recorded in  $\text{Me}_2\text{CO}-d_6$



### 1.3.1.5 Compound CF5



**CF5** was obtained as yellow needles. The UV (**Figure 20**) and IR (**Figure 21**) spectra of **CF5** exhibited the same pattern as those of **CF4**. The  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra (**Table 8**, **Figures 22** and **23**) showed signals similar to those of **CF4** except for the replacement of the prenyl group [ $\delta$  3.47 (2H, *d*,  $J = 6.9$  Hz, H-1'), 5.24 (1H, *m*, H-2'), 1.79 (3H, *d*, 0.9 Hz, H-4') and 1.86 (3H, *brs*, H-5')] in **CF4** with the characteristic signals of a chromene ring [ $\delta$  1.52 (6H, *s*, H-4' and H-5'), 5.61 (1H, *d*,  $J = 9.9$  Hz, H-2') and 6.76 (1H, *d*,  $J = 9.9$  Hz, H-1')] in **CF5**. Thus, **CF5** was characterized as macluraxanthone (Iinuma *et al.*, 1994).



Selected HMBC correlations of **CF5**

**Table 8**  $^1\text{H}$ ,  $^{13}\text{C}$  NMR, DEPT and HMBC spectral data of **CF5**

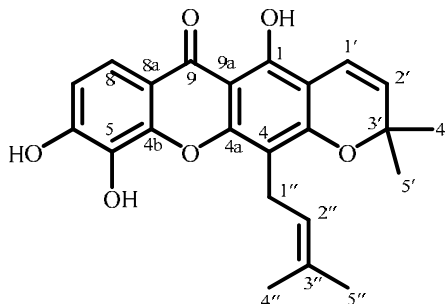
Position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	DEPT	HMBC
1		156.8	C	
2		105.6	C	
3		158.9	C	
4		113.1	C	
4a		154.1	C	
4b		144.5	C	
5		131.1	C	
6		149.0	C	
7	6.94 ( <i>d</i> , 9.0)	112.8	CH	5, 6, 8a
8	7.68 ( <i>d</i> , 9.0)	117.5	CH	6, 9, 4b
8a		113.7	C	
9		180.8	C	
9a		103.0	C	
1'	6.76 ( <i>d</i> , 9.9)	116.1	CH	1, 2, 3, 3'
2'	5.61 ( <i>d</i> , 9.9)	127.2	CH	2, 3', 4', 5'
3'		78.3	C	
4'	1.52 ( <i>s</i> )	27.9	CH <sub>3</sub>	2', 3'
5'	1.52 ( <i>s</i> )	27.9	CH <sub>3</sub>	2', 3'
1''		41.4	C	
2''	6.76 ( <i>dd</i> , 17.7, 10.5)	156.8	CH	1'', 3'', 4'', 5''
3''	5.22 ( <i>dd</i> , 17.7, 1.5) 5.05 ( <i>dd</i> , 10.5, 1.5)	103.3	CH <sub>2</sub>	1'', 2''
4''	1.65 ( <i>s</i> )	28.2	CH <sub>3</sub>	4, 1'', 2''
5''	1.65 ( <i>s</i> )	28.2	CH <sub>3</sub>	4, 1'', 2''
1-OH	13.53 ( <i>s</i> )			1, 2, 9a

**Table 9** Comparison of  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectral data of **CF5** and macluraxanthone<sup>a</sup>

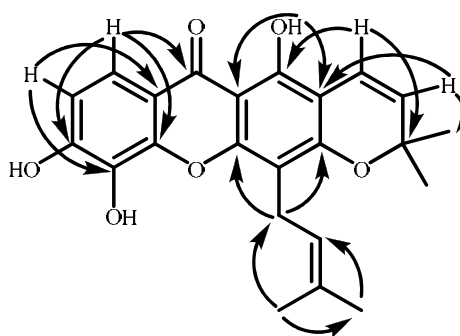
position	CF5		macluraxanthone <sup>a</sup>	
	$\delta_{\text{H}}$ (mult., $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	$\delta_{\text{H}}$ (mult., $J_{\text{Hz}}$ )	$\delta_{\text{C}}$
1		156.8		157.3
2		105.6		105.7
3		158.9		159.6
4		113.1		114.2
4a		154.1		155.9
4b		144.5		146.7
5		131.1		131.1
6		149.0		149.0
7	6.94 ( <i>d</i> , 9.0)	112.8	7.00 ( <i>d</i> , 9.0)	112.8
8	7.68 ( <i>d</i> , 9.0)	117.5	7.60 ( <i>d</i> , 9.0)	117.5
8a		113.7		114.4
9		180.8		180.8
9a		103.0		103.6
1'	6.76 ( <i>d</i> , 9.9)	116.1	6.69 ( <i>d</i> , 10.0)	116.4
2'	5.61 ( <i>d</i> , 9.9)	127.2	5.70 ( <i>d</i> , 10.0)	128.2
3'		78.3		79.0
4'	1.52 ( <i>s</i> )	27.9	1.49 ( <i>s</i> )	28.0
5'	1.52 ( <i>s</i> )	27.9	1.49 ( <i>s</i> )	28.0
1''		41.4		41.8
2''	6.76 ( <i>dd</i> , 17.7, 10.5)	156.8	6.52 ( <i>dd</i> , 17.0, 11.0)	152.9
3''	5.22 ( <i>dd</i> , 17.7, 1.5)	103.3	5.05 ( <i>dd</i> , 17.0, 1.0)	107.2
	5.05 ( <i>dd</i> , 10.5, 1.5)		4.89 ( <i>dd</i> , 11.0, 1.0)	
4''	1.65 ( <i>s</i> )	28.2	1.74 ( <i>s</i> )	29.9
5''	1.65 ( <i>s</i> )	28.2	1.74 ( <i>s</i> )	29.9
1-OH	13.53 ( <i>s</i> )		13.91 ( <i>s</i> )	

<sup>a</sup> Recorded in  $\text{Me}_2\text{CO}-d_6$

### 1.3.1.6 Compound CF6



**CF6** was obtained as yellow needles. The UV (**Figure 24**) and IR (**Figure 25**) spectra closely resembled to those of **CF5**. The  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra (**Table 10**, **Figures 26** and **27**) were similar to those of **CF5** except for the appearance of signals of  $\gamma,\gamma$ -dimethylallyl side chain [ $\delta$  1.75 (3H, *brs*, H-5''), 1.87 (3H, *br s*, H-4''), 3.49 (2H, *d*,  $J = 7.2$  Hz, H-1'') and 5.24 (1H, *mt*,  $J = 7.2$  Hz, H-2'')] in **CF6** instead of  $\alpha,\alpha$ -dimethylallyl group [ $\delta$  1.65 (6H, *s*, H-4'' and H-5''), 5.05 (1H, *dd*,  $J = 10.5, 1.5$  Hz, H-3''), 5.22 (1H, *dd*,  $J = 17.7, 1.5$  Hz, H-3'') and 6.76 (1H, *dd*,  $J = 17.7, 10.5$  Hz, H-2'')] in **CF5**. Therefore, **CF6** was determined as xanthone V<sub>1</sub> (Botta *et al.*, 1986).



Selected HMBC correlations of **CF6**

**Table 10**  $^1\text{H}$ ,  $^{13}\text{C}$  NMR, DEPT and HMBC spectral data of **CF6**

Position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	DEPT	HMBC
1		155.3	C	
2		104.2	C	
3		158.0	C	
4		107.8	C	
4a		154.3	C	
4b		146.5	C	
5		132.3	C	
6		151.2	C	
7	6.95 ( <i>d</i> , 8.7)	112.4	CH	5, 6, 8a
8	7.70 ( <i>d</i> , 8.7)	116.7	CH	4b' 4b, 9
8a		113.8	C	
9		181.2	C	
9a		102.6	C	
1'	6.74 ( <i>d</i> , 9.9)	115.6	CH	1, 2, 3, 3'
2'	5.60 ( <i>d</i> , 9.9)	127.3	CH	2, 3', 4', 5'
3'		77.9	C	
4'	1.48 ( <i>s</i> )	28.0	CH <sub>3</sub>	2', 3', 5'
5'	1.48 ( <i>s</i> )	28.0	CH <sub>3</sub>	2', 3', 4'
1''	3.49 ( <i>d</i> , 7.2)	21.3	CH <sub>2</sub>	3, 4, 4a, 2'', 3''
2''	5.24 ( <i>mt</i> , 7.2)	123.3	CH	
3''		132.3	C	
4''	1.87 ( <i>br s</i> )	25.5	CH <sub>3</sub>	4'', 5''
5''	1.75 ( <i>br s</i> )	17.6	CH <sub>3</sub>	
1-OH	13.20 ( <i>s</i> )			

**Table 11** Comparison of  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectral data of **CF6** and Xanthone V<sub>1</sub>

position	CF6		Xanthone V <sub>1</sub>	
	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$
1		155.3		158.2
2		104.2		104.6
3		158.0		154.5
4		107.8		108.0
4a		154.3		156.3
4b		146.5		146.8
5		132.3		133.0
6		151.2		151.9
7	6.95 ( <i>d</i> , 8.7)	112.4	6.95 ( <i>d</i> , 8.5)	114.3
8	7.70 ( <i>d</i> , 8.7)	116.7	7.60 ( <i>d</i> , 8.5)	117.2
8a		113.8		113.0
9		181.2		181.1
9a		102.6		102.9
1'	6.74 ( <i>d</i> , 9.9)	115.6	6.66 ( <i>d</i> , 10)	115.3
2'	5.60 ( <i>d</i> , 9.9)	127.3	5.66 ( <i>d</i> , 10)	127.1
3'		77.9		79.2
4'	1.48 ( <i>s</i> )	28.0	1.47 ( <i>s</i> )	29.1
5'	1.48 ( <i>s</i> )	28.0	1.47 ( <i>s</i> )	29.1
1''	3.49 ( <i>d</i> , 7.2)	21.3	3.52 ( <i>d</i> , 7)	21.6
2''	5.24 ( <i>mt</i> , 7.2)	123.3	5.30 ( <i>t</i> , 7)	123.3
3''		132.3		131.0
4''	1.87 ( <i>br s</i> )	25.5	1.85 ( <i>br s</i> )	25.7
5''	1.75 ( <i>brs</i> )	17.6	1.65 ( <i>br s</i> )	17.9
1-OH	13.20 ( <i>s</i> )		13.45 ( <i>s</i> )	

**Table 12** Comparison of  $^1\text{H}$  NMR spectral data of CF4-CF6

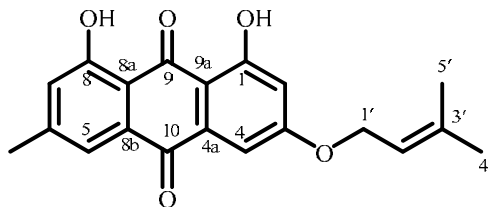
Position	CF4	CF5	CF6
	$\delta_{\text{H}}$ (mult., $J_{\text{Hz}}$ )	$\delta_{\text{H}}$ (mult., $J_{\text{Hz}}$ )	$\delta_{\text{H}}$ (mult., $J_{\text{Hz}}$ )
7	6.94 ( <i>d</i> , 8.7)	6.94 ( <i>d</i> , 9.0)	6.95 ( <i>d</i> , 8.7)
8	7.70 ( <i>d</i> , 8.7)	7.68 ( <i>d</i> , 9.0)	7.70 ( <i>d</i> , 8.7)
1'	3.47 ( <i>d</i> , 6.9)	6.76 ( <i>d</i> , 9.9)	6.74 ( <i>d</i> , 9.9)
2'	5.24 ( <i>m</i> )	5.61 ( <i>d</i> , 9.9)	5.60 ( <i>d</i> , 9.9)
4'	1.79, <i>d</i> , 0.9)	1.52 ( <i>s</i> )	1.48 ( <i>s</i> )
5'	1.86 ( <i>br s</i> )	1.52 ( <i>s</i> )	1.48 ( <i>s</i> )
1''			3.49 ( <i>d</i> , 7.2)
2''	6.68 ( <i>dd</i> , 17.7, 10.5)	6.76 ( <i>dd</i> , 17.7, 10.5)	5.24 ( <i>mt</i> , 7.2)
3''	5.30 ( <i>dd</i> , 17.7, 0.9)	5.22 ( <i>dd</i> , 17.7, 1.5)	
	5.15 ( <i>dd</i> , 10.5, 0.9)	5.05 ( <i>dd</i> , 10.5, 1.5)	
4''	1.69 ( <i>s</i> )	1.65 ( <i>s</i> )	1.87 ( <i>br s</i> )
5''	1.69 ( <i>s</i> )	1.65 ( <i>s</i> )	1.75 ( <i>br s</i> )
1-OH	13.60 ( <i>s</i> )	13.53 ( <i>s</i> )	13.20 ( <i>s</i> )

**Table 13** Comparison of  $^{13}\text{C}$  NMR spectral data of **CF4-CF6**

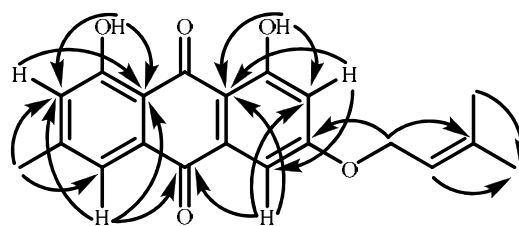
<b>Position</b>	<b>CF4</b>	<b>CF5</b>	<b>CF6</b>
1	159.0	156.8	155.3
2	110.1	105.6	104.2
3	161.4	158.9	158.0
4	111.2	113.1	107.8
4a	153.3	154.1	154.3
4b	144.8	144.5	146.5
5	131.0	131.1	132.3
6	149.0	149.0	151.2
7	111.6	112.8	112.4
8	117.2	117.5	116.7
8a	113.8	113.7	113.8
9	180.3	180.8	181.2
9a	103.0	103.0	102.6
1'	21.6	116.1	115.6
2'	121.2	127.2	127.3
3'	136.1	78.3	77.9
4'	25.9	27.9	28.0
5'	18.0	27.9	28.0
1''	41.6	41.4	21.3
2''	154.9	156.8	123.3
3''	106.1	103.3	132.3
4''	28.0	28.2	25.5
5''	28.0	28.2	17.6



### 1.3.1.7 Compound CF7



**CF7**, a reddish orange solid, the IR spectrum (**Figure 29**) exhibited absorption bands at  $3409\text{ cm}^{-1}$  (hydroxyl),  $1628$  (conjugated carbonyl) and  $1609$  (aromatic ring) and the UV spectrum (**Figure 28**) exhibited  $\lambda_{\text{max}}$  226, 254, 266, 288 and 437 nm suggesting the presence of a quinone structure possibly a hydroxyanthraquinone. The  $^{13}\text{C}$  NMR spectrum (**Table 14, Figure 31**) showed 20 signals, attributable to three methyls, one methylene, five methines and eleven quaternary carbons, as determined by DEPT experiments. The  $^1\text{H}$  NMR (**Table 14, Figure 30**) revealed the presence of two sharp *singlet* signals of chelated hydroxyl groups at  $\delta$  12.28 (1-OH) and 12.11 (8-OH), The signals of two sets of meta-coupled aromatic protons were observed. The first set appeared as doublet signals at  $\delta$  6.66 and 7.35 which was assigned to be H-2 and H-4 by the correlation of H-2 to C-1, C-4 and C-9a and H-4 to C-2, C-3, C-9a and C-10 in HMBC experiment whereas, the second set showed the signals at  $\delta$  7.60 and 7.06. These were proposed for the signals of H-5 and H-7, respectively and was supported by the correlation of H-5 to C-6, C-7, C-8a, C-9 and C-10 and H-7 to C-5, C-8 and C-8a. A *singlet* methyl signal at  $\delta$  2.44 was assigned to be 6-Me according to the correlation to C-5, C-6 and C-7 from the HMBC experiment. Furthermore, The spectrum further showed the signals of a prenyl moiety at  $\delta$  4.64 (1H, *d*,  $J = 6.9$  Hz), 5.48 (1H, *d*,  $J = 6.9$  Hz), 1.82 (3H, *brs*) and 1.79 (3H, *brs*). Since the chemical shift of methylene protons of the prenyl side chain appeared at low field, the prenyl group was attached to oxygen which was assigned at C-3 according to HMBC correlation of the oxymethylene proton H-1' (4.64) to C-3 (165.9). Thus, **CF7** was characterized as madagascin (Nagem and Oliveira 1997).



Selected HMBC correlations of CF7

**Table 14**  $^1\text{H}$ ,  $^{13}\text{C}$  NMR, DEPT and HMBC spectral data of CF7

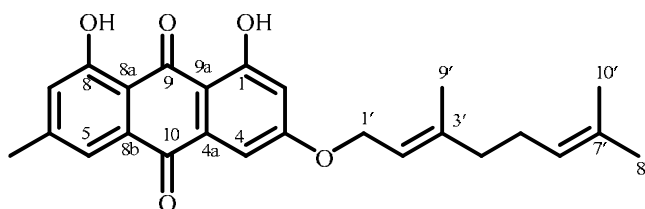
Position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	DEPT	HMBC
1		165.1	C	
2	6.66 ( <i>d</i> , 2.7)	107.5	CH	1, 4, 9a
3		165.9	C	
4	7.35 ( <i>d</i> , 2.7)	108.7	CH	2, 3, 9a, 10
4a		135.2	C	
5	7.60 ( <i>br d</i> , 1.2)	121.2	CH	6, 7, 8a, 9, 10, 6-Me
6		148.3	C	
7	7.06 ( <i>dd</i> , 1.5, 0.9)	124.4	CH	5, 8, 8a, 6-Me
8		162.5	C	
8a		113.7	C	
8b		133.2	C	
9		190.9	C	
9a		110.1	C	
10		182.0	C	
1'	4.64 ( <i>d</i> , 6.9)	65.8	CH <sub>2</sub>	3, 2', 3'
2'	5.48 ( <i>d</i> , 6.9)	118.2	CH	1', 4', 5'
3'		139.7	C	
4'	1.82 ( <i>br s</i> )	25.8	CH <sub>3</sub>	2', 3', 5'
5'	1.79 ( <i>br s</i> )	18.3	CH <sub>3</sub>	2', 3', 4'
6-Me	2.44 ( <i>s</i> )	22.1	CH <sub>3</sub>	5, 6, 7
1-OH	12.28 ( <i>s</i> )			1, 9a
8-OH	12.11 ( <i>s</i> )			7, 8

**Table 15** Comparison of  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectral data of **CF7** and madagascin

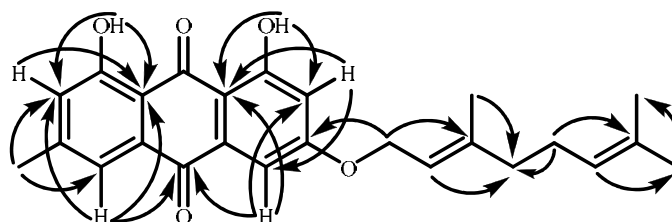
position	CF7		madagascin <sup>a</sup>	
	$\delta_{\text{H}}$ (mult., $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	$\delta_{\text{H}}$ (mult., $J_{\text{Hz}}$ )	$\delta_{\text{C}}$
1		165.1		162.3
2	6.66 ( <i>d</i> , 2.7)	107.5	6.91 ( <i>d</i> , 1.8)	108.6
3		165.9		165.7
4	7.35 ( <i>d</i> , 2.7)	108.7	7.50 ( <i>d</i> , 1.8)	107.4
4a		135.2		135.0
5	7.60 ( <i>brd</i> , 1.2)	121.2	7.23 ( <i>d</i> , 2.0)	121.1
6		148.3		139.7
7	7.06 ( <i>dd</i> , 1.5, 0.9)	124.4	6.56 ( <i>d</i> , 2.0)	124.3
8		162.5		164.9
8a		113.7		110.0
8b		133.2		133.1
9		190.9		190.5
9a		110.1		108.0
10		182.0		182.0
1'	4.64 ( <i>d</i> , 6.9)	65.8	4.54 ( <i>d</i> , 6.4)	65.6
2'	5.48 ( <i>d</i> , 6.9)	118.2	5.40 ( <i>d</i> , 6.7)	118.1
3'		139.7		143.5
4'	1.82 ( <i>br s</i> )	25.8	1.71 ( <i>s</i> )	25.8
5'	1.79 ( <i>br s</i> )	18.3	1.75 ( <i>s</i> )	18.3
6-Me	2.44 ( <i>s</i> )	22.1	2.44 ( <i>s</i> )	22.1

<sup>a</sup> recorded in  $\text{CDCl}_3$

### 1.3.1.8 Compound CF8



**CF8** was isolated as reddish orange solid. The IR (**Figure 33**) and UV (**Figure 32**) spectra exhibited the same patterns as those of **CF7**. The  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra (**Table 16**, **Figures 34** and **35**) were similar to those of **CF7** except for the replacement of the prenyl group in **CF7** with the characteristic signals of a geranyl group in **CF8**. These signals were assigned as follow; two *singlet* signals at  $\delta$  1.61 and 1.68 and one *doublet* signal at  $\delta$  1.78 were of three vinylic methyl groups, a *doublet* signal ( $J = 6.6$  Hz) at  $\delta$  4.66 was assigned for oxymethylene protons  $\text{H}_2\text{-1}'$ , two *multiplet* signals at  $\delta$  2.12 and 2.15 were the signals of two groups of methylene protons  $\text{H}_2\text{-4}'$  and  $\text{H}_2\text{-5}'$ , respectively, a *multiplet* signals ( $J = 6.6$  Hz) at  $\delta$  5.09 and *multiplet of triplet* at  $\delta$  5.47 were the signals of two olefinic methine protons  $\text{H-6}'$  and  $\text{H-2}'$ , respectively. These assignments indicated that **CF8** was 3-geranyloxy-6-methyl-1,8-dihydroxyanthraquinone (Botta, *et al.*, 1983 ).



Selected HMBC correlations of **CF8**

**Table 16**  $^1\text{H}$ ,  $^{13}\text{C}$  NMR, DEPT and HMBC spectral data of **CF8**

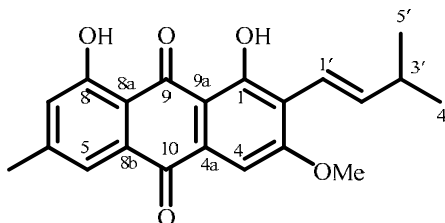
Position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	DEPT	HMBC
1		165.1	C	
2	6.65 ( <i>d</i> , 2.4)	107.5	CH	1, 4, 9a
3		165.9	C	
4	7.33 ( <i>d</i> , 2.4)	108.8	CH	2, 9, 9a, 10
4a		135.1	C	
5	7.58 ( <i>br dd</i> , 1.5, 0.6)	121.2	CH	6-Me, 7, 8a, 9, 10
6		148.3	C	
7	7.05 ( <i>dd</i> , 1.5, 0.6)	124.4	CH	5, 8, 8a, 6-Me
8		162.4	C	
8a		113.7	C	
8b		133.2	C	
9		190.6	C	
9a		110.1	C	
10		181.9	C	
1'	4.66 ( <i>d</i> , 6.6)	65.8	CH <sub>2</sub>	3, 2', 3'
2'	5.47 ( <i>mt</i> , 6.6)	118.0	CH	1', 4', 9'
3'		142.8	C	
4'	2.12 ( <i>m</i> )	39.5	CH <sub>2</sub>	2', 3', 5'
5'	2.15 ( <i>m</i> )	26.2	CH <sub>2</sub>	4', 6', 7'
6'	5.09 ( <i>m</i> )	123.6	CH	5'
7'		132.0	C	
8'	1.61 ( <i>s</i> )	17.7	CH <sub>3</sub>	6', 7', 10'
9'	1.78 ( <i>d</i> , 0.9)	16.8	CH <sub>3</sub>	2', 3', 4'
10'	1.68 ( <i>s</i> )	25.6	CH <sub>3</sub>	6', 7', 8'
6-Me	2.43 ( <i>s</i> )	22.1	CH <sub>3</sub>	5, 6, 7
1-OH	12.25 ( <i>s</i> )			1, 2, 9a
8-OH	12.10 ( <i>s</i> )			7, 8, 8a

**Table 17** Comparison of  $^1\text{H}$  NMR spectral data of **CF8** and 3-geranyloxy-6-methyl-1,8-dihydroxyanthraquinone (**R**)

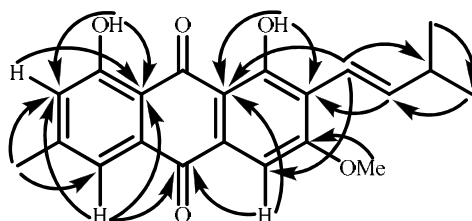
Position	CF8	<b>R</b> <sup>a</sup>
	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )
2	6.65 ( <i>d</i> , 2.4)	6.60 ( <i>d</i> , 2.5)
4	7.33 ( <i>d</i> , 2.4)	7.27 ( <i>d</i> , 2.5)
5	7.58 ( <i>br dd</i> , 1.5, 0.6)	7.50 ( <i>br d</i> , 1.8)
7	7.05 ( <i>dd</i> , 1.5, 0.6)	7.00 ( <i>br d</i> , 1.8)
1'	4.66 ( <i>d</i> , 6.6)	4.60 ( <i>d</i> , 7.0)
2'	5.47 ( <i>mt</i> , 6.6)	5.43 ( <i>t</i> , 7.0)
4'	2.12 ( <i>m</i> )	2.10 ( <i>m</i> )
5'	2.15 ( <i>m</i> )	2.10 ( <i>m</i> )
6'	5.09 ( <i>m</i> )	5.05 ( <i>br s</i> )
8'	1.61 ( <i>s</i> )	1.60 ( <i>s</i> )
9'	1.78 ( <i>d</i> , 0.9)	1.77 ( <i>s</i> )
10'	1.68 ( <i>s</i> )	1.67 ( <i>s</i> )
6-Me	2.43 ( <i>s</i> )	2.40 ( <i>s</i> )
1-OH	12.25 ( <i>s</i> )	12.23 ( <i>s</i> )
8-OH	12.10 ( <i>s</i> )	12.08 ( <i>s</i> )

<sup>a</sup> recorded in  $\text{CDCl}_3$

### 1.3.1.9 Compound CF9



**CF9** was isolated as reddish orange solid. The  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectral data (Table 18, Figures 38 and 39) of **CF9** were comparable to those of **CF7**, except for the presence of *trans*-3-methylbut-1-enyl group at  $\delta$  6.64 (1H, *dd*, 16.2, 0.9 Hz, H-1'), 6.91 (1H, *dd*, 16.2, 7.2 Hz, H-2'), 2.52 (1H, *dsept*, 0.9, 6.9 Hz, H-3'), 1.14, (6H, *d*, 6.9 Hz, H-4' and H-5') in **CF9** instead of the meta-coupled aromatic protons at  $\delta$  6.66 (1H, *d*, 2.7 Hz, H-2) and at  $\delta$  7.35 (1H, *d*, 2.7 Hz, H-4) and signals of a prenyl side chain in **CF7**. The location of *trans*-3-methylbut-1-enyl group was assigned to C-2 by the HMBC correlations from the chelated hydroxyl group at  $\delta$  12.93 (1-OH) to the carbons at  $\delta$  110.5 (C-9a), 120.0 (C-2) and 162.08 (C-1) and the olefinic proton of *trans*-3-methylbut-1-enyl group at  $\delta$  6.64 (H-1') to the carbons at  $\delta$  162.1 (C-1) and 163.0 (C-3). The  $^1\text{H}$  NMR spectrum also showed a *singlet* signal of the methoxyl group at  $\delta$  4.04 (3H, *s*, 3-OMe). The attachment of a methoxyl group was assigned to C-3 by the HMBC correlations of 3-OMe at  $\delta$  4.04 to the carbon at  $\delta$  163.0 (C-3). Therefore, **CF9** was determined as vismiaquinone (Goncalves and Mors, 1981).



Selected HMBC correlations of **CF9**

**Table 18**  $^1\text{H}$ ,  $^{13}\text{C}$  NMR, DEPT and HMBC spectral data of **CF9**

Position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	DEPT	HMBC
1		162.1	C	
2		120.0	C	
3		163.0	C	
4	7.38 (s)	103.4	CH	2, 3, 4a, 9, 9a, 10
4a		132.1	C	
5	7.59 ( <i>d</i> , 1.5)	121.1	CH	6-Me, 7, 8a, 9, 10
6		148.4	C	
7	7.05 ( <i>br d</i> , 0.6)	124.4	CH	5, 8a
8		162.5	C	
8a		113.7	C	
8b		133.2	C	
9		181.8	C	
9a		110.5	C	
10		191.4	C	
1'	6.64 ( <i>dd</i> , 16.2, 0.9)	115.8	CH	1, 3, 2', 3'
2'	6.91 ( <i>dd</i> , 16.2, 7.2)	146.8	CH	2, 3'
3'	2.52 ( <i>dsept</i> , 0.9, 6.9)	33.4	CH	1'
4'	1.14 ( <i>d</i> , 6.9)	22.5	CH <sub>3</sub>	2', 3'
5'	1.14 ( <i>d</i> , 6.9)	22.5	CH <sub>3</sub>	2', 3'
6-Me	2.44 ( <i>s</i> )	22.1	CH <sub>3</sub>	5, 6, 7
1-OH	12.93 ( <i>s</i> )			1, 3, 9a
8-OH	12.08 ( <i>s</i> )			6, 7, 8, 8a
3-OMe	4.04 ( <i>s</i> )	56.3	CH <sub>3</sub>	3



**Table 19** Comparison of  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectral data of **CF9** and vismiaquinone<sup>a</sup>

position	CF9		vismiaquinone <sup>a</sup>	
	$\delta_{\text{H}}$ (mult., $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	$\delta_{\text{H}}$ (mult., $J_{\text{Hz}}$ )	$\delta_{\text{C}}$
1		162.1		161.8
2		120.0		119.8
3		163.0		162.7
4	7.38 (s)	103.4	7.34 (s)	103.1
4a		132.1		131.8
5	7.59 (d, 1.5)	121.1	7.56 (d, 1.5)	120.8
6		148.4		148.1
7	7.05 (br d, 0.6)	124.4	7.03 (s)	124.2
8		162.5		162.2
8a		113.7		113.5
8b		133.2		132.9
9		181.8		181.4
9a		110.5		110.3
10		191.4		191.0
1'	6.64 (dd, 16.2, 0.9)	115.8	6.60 (d, 16.0)	115.7
2'	6.91 (dd, 16.2, 7.2)	146.8	6.95 (dd, 16.0, 6.5)	146.5
3'	2.52 (dsept, 0.9, 6.9)	33.4	2.48 (m)	33.4
4'	1.14 (d, 6.9)	22.5	1.14 (d, 6.5)	22.5
5'	1.14 (d, 6.9)	22.5	1.14 (d, 6.5)	22.5
6-Me	2.44 (s)	22.1	2.42 (s)	22.1
1-OH	12.93 (s)		12.84 (s)	
8-OH	12.08 (s)		12.02 (s)	
3-OMe	4.04 (s)	56.3	4.02 (s)	56.2

<sup>a</sup> recorded in  $\text{CDCl}_3$

**Table 20** Comparison of  $^1\text{H}$  NMR spectral data of CF7-CF9

Position	CF7	CF8	CF9
	$\delta_{\text{H}}$ (mult., J <sub>Hz</sub> )	$\delta_{\text{H}}$ (mult., J <sub>Hz</sub> )	$\delta_{\text{H}}$ (mult., J <sub>Hz</sub> )
2	6.66 ( <i>d</i> , 2.7)	6.65 ( <i>d</i> , 2.4)	
4	7.35 ( <i>d</i> , 2.7)	7.33 ( <i>d</i> , 2.4)	7.38 ( <i>s</i> )
5	7.60 ( <i>br d</i> , 1.2)	7.58 ( <i>brdd</i> , 1.5, 0.6)	7.59 ( <i>d</i> , 1.5)
7	7.06 ( <i>dd</i> , 1.5, 0.9)	7.05 ( <i>dd</i> , 1.5, 0.6)	7.05 ( <i>brd</i> , 0.6)
1'	4.64 ( <i>d</i> , 6.9)	4.66 ( <i>d</i> , 6.6)	6.64 ( <i>dd</i> , 16.2, 0.9)
2'	5.48 ( <i>d</i> , 6.9)	5.47 ( <i>mt</i> , 6.6)	6.91 ( <i>dd</i> , 16.2, 7.2)
3'			2.52 ( <i>dsept</i> , 0.9, 6.9)
4'	1.82 ( <i>br s</i> )	2.12 ( <i>m</i> )	1.14 ( <i>d</i> , 6.9)
5'	1.79 ( <i>br s</i> )	2.15 ( <i>m</i> )	1.14 ( <i>d</i> , 6.9)
6'		5.09 ( <i>m</i> )	
8'		1.61 ( <i>s</i> )	
9'		1.78 ( <i>d</i> , 0.9)	
10'		1.68 ( <i>s</i> )	
6-Me	2.44 ( <i>s</i> )	2.43 ( <i>s</i> )	2.44 ( <i>s</i> )
1-OH	12.28 ( <i>s</i> )	12.25 ( <i>s</i> )	12.93 ( <i>s</i> )
8-OH	12.11 ( <i>s</i> )	12.10 ( <i>s</i> )	12.08 ( <i>s</i> )
3-OMe			4.04 ( <i>s</i> )

**Table 21** Comparison of  $^{13}\text{C}$  NMR spectral data of **CF7-CF9**

<b>Position</b>	<b>CF7</b>	<b>CF8</b>	<b>CF9</b>
1	165.1	165.1	162.1
2	107.5	107.5	120.0
3	165.9	165.9	163.0
4	108.7	108.8	103.4
4a	135.2	135.1	132.1
5	121.2	121.2	121.1
6	148.3	148.3	148.4
7	124.4	124.4	124.4
8	162.5	162.4	162.5
8a	113.7	113.7	113.7
8b	133.2	133.2	133.2
9	190.9	190.6	181.8
9a	110.1	110.1	110.5
10	182.0	181.9	191.4
1'	65.8	65.8	115.8
2'	118.2	118.0	146.8
3'	139.7	142.8	33.4
4'	25.8	39.5	22.5
5'	18.3	26.2	22.5
6'	22.1	123.6	22.1
7'		132.0	
8'		17.7	
9'		16.8	
10'		25.6	
6-Me		22.1	
3-OMe			56.3

### 1.3.2 Biological activities of the isolated compounds from the roots of *C. formosum*

The isolated compounds were evaluated for their antibacterial activities against both Gram-positive (*Bacillus subtilis* and *Staphylococcus aureus*) and Gram-negative (*Streptococcus faecalis*, *Salmonella typhi*, *Shigella sonnei* and *Pseudomonas aeruginosa*) bacteria. Cytotoxicity against MCF-7 (breast adenocarcinoma), HeLa (Human cervical cancer), HT-29 (colon cancer) and KB (human oral cancer) cell lines were also evaluated.

Compounds tested for antibacterial and cytotoxic activities were xanthenes **CF1**, **CF3-CF6** and anthraquinones **CF7-CF9**, whereas compound **CF2** was not tested due to insufficient amount of material. The results of antibacterial activity of the tested compounds were given in **Table 22**. Compound **CF6** exhibited potent antibacterial activity against *B. subtilis*, *S. aureus*, *S. faecalis* and *S. typhi*. Compound **CF4** showed strong inhibition against *S. aureus* and *S. typhi*. The anthraquinones were found to be inactive. For cytotoxicity results as shown in **Table 22**, compound **CF3** was the most cytotoxic against all four cancer cell lines. Compound **CF4** and **CF6** were inactive against the HT-29 and MCF-7 cell lines, respectively, while compounds **CF1**, **CF5**, **CF7**, **CF8** and **CF9** were inactive.

**Table 22** Cytotoxic and antibacterial activities of compounds isolated from *C. formosum*

Compounds	Cytotoxicity against human cancer cell lines, IC <sub>50</sub> (μg/mL)				Antibacterial activity, MIC (μg/mL)				
	MCF-7	HeLa	HT-29	KB	<i>P. aeruginosa</i>	<i>B. subtilis</i>	<i>S. aureus</i>	<i>E. faecalis</i>	<i>S. typhi</i>
<b>CF1</b>	-	-	-	-	-	18.7	37.5	-	-
<b>CF3</b>	4.9	3.7	5.3	3.3	-	4.6	2.3	18.7	4.6
<b>CF4</b>	12.0	5.0	>25.0	4.7	-	2.3	1.1	4.6	1.1
<b>CF5</b>	-	-	-	-	-	4.6	4.6	2.3	9.3
<b>CF6</b>	>25.0	4.7	6.0	2.7	9.3	1.1	1.1	1.1	1.1
<b>CF7</b>	-	-	-	-	-	-	-	-	-
<b>CF8</b>	-	-	-	-	-	-	-	-	-
<b>CF9</b>	-	-	-	-	-	-	-	-	-

- = inactive (> 10 μg/mL)

## CHAPTER 2.1

### INTRODUCTION

#### 2.1.1 Introduction

*Thespesia populnea* (L.) Soland. Ex Coor is a mangrove plant belonging to Malvaceae family. *T. populnea* is widely distributed in Hawaii, California, Florida, Africa, the Caribbean islands and in Asia (Milbrodt *et al.*, 1997). In Thailand, the family Malvaceae comprises 15 genera. In *Thespesia* genus 3 species are found including *T. lampas* (Cav.) Dalzell & Gibson, *T. populnea* (L.) Soland. Ex Coor and *T. populneoilides* (Roxb.) Kostel (Smitinand, 2001).

*T. populnea* has a short, straight or crooked trunk and a dense crown with crowded lower horizontal branches. Flowers are a typical hibiscus shape in appearance: bellshaped, 4–7 cm in length, with five overlapping, broad, rounded petals. Color is pale yellow with a maroon spot at the base of each petal and with star-shaped hairs on outer surface. Flower stalks are 1.3–5 cm. The alternate leaves are glossy green above and paler green below. Leaf blades are heart-shaped, 10–20 cm long, and 6–13 cm broad. Leaf stalks are long, 5–10 cm. Fruits are brittle, dry, woody or papery seed capsules, rounded and flattened, containing five cells and several seeds. The brown or gray capsules, about 2.5–5 cm in diameter. The brown, hairy seeds are about 1 cm long and 0.6 cm broad.



**Figure 2** Parts of *Thespesia populnea*

### 2.1.2 Review of Literatures

Chemical constituents isolated from *Thespesia* genus were summarized in **Table 23**. The literature survey was from SciFinder Scholar database and the chemical constituents could be classified into groups, such as alkanes, flavonoids, sesquiterpenes, steroids and triterpenes.

**Table 23** Compounds from plants of *Thespesia* genus

**a** = Alkanes      **b** = Flavonoids      **c** = Sesquiterpenes  
**d** = Steroids      **e** = Triterpenes

Scientific name	Investigated Part	Compound	Bibliography
<i>T. populnea</i>	Bark	(+)-Gossypol, <b>16c</b>	Waller <i>et al.</i> , 1983
	Flowers	7-Hydroxyisoflavone, <b>1b</b> Tamarixetin-7-O- $\beta$ -glucoside, <b>8b</b> Kaempferol-7-O- $\beta$ -rutinoside, <b>15b</b> $\beta$ -Sitosterol, <b>1d</b> $\beta$ -Sitosterol-3- $\beta$ -D-glucoside, <b>3d</b> Lupeol, <b>1e</b> Nanacosane, <b>11a</b> Lupenone, <b>2e</b> Kaempferol, <b>4b</b> Quercetin, <b>3b</b> Kaempferol-3-O- $\beta$ -glucoside, <b>11b</b> Quercetin-3- O- $\beta$ -glucoside, <b>12b</b>	Seshadri <i>et al.</i> , 1975          Datta <i>et al.</i> , 1973



Table 23 (Continued)

Scientific name	Investigated Part	Compound	Bibliography
<i>T. populnea</i>	Flowers	Kaempferol-5- O- $\beta$ -glucoside, <b>7b</b> Kaempferol-7- O- $\beta$ -glucoside, <b>10b</b> Quercetin-3- O- $\beta$ -rutinoside, <b>14b</b> (+)-Gossypol, <b>13c</b>	Datta et al., 1973
	Fruits	Thespesin, <b>14c</b>	Srivastava <i>et al.</i> , 1963
	Heartwood	Mansonone C, <b>2c</b>  Mansonone D, <b>3c</b> Mansonone E, <b>4c</b> Mansonone F, <b>5c</b>  Mansonone G, <b>6c</b> Mansonone H, <b>7c</b> Mansonone M, <b>8c</b> 7-Hydroxycadalene, <b>1c</b>	Puckhaber <i>et al.</i> , 2004 Milbrodt <i>et al.</i> , 1997 Neelakantan <i>et al.</i> , 1983  Puckhaber <i>et al.</i> , 2004

Table 23 (Continued)

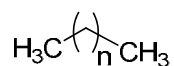
Scientific name	Investigated Part	Compound	Bibliography
<i>T. populnea</i>	Heartwood	Thespesone, <b>9c</b>  Thespesenone, <b>10c</b>  Dehydrooxoperezinone-6-methyl ether, <b>11c</b>  7-Hydroxy-2,3,5,6-tetrahydro-3,6,9-trimethylnaphtho[1,8-b,c]pyran-4,8-dione, <b>12c</b>  Quercetin, <b>3b</b> Calcycopterin, <b>2b</b>	Puckhaber <i>et al.</i> , 2004 Milbrodt <i>et al.</i> , 1997 Neelakantan <i>et al.</i> , 1983 Puckhaber <i>et al.</i> , 2004 Milbrodt <i>et al.</i> , 1997 Puckhaber <i>et al.</i> , 2004 Kasim <i>et al.</i> , 1975
	Leaves	Lupeol, <b>1e</b>  Lupenone, <b>2e</b>  $\beta$ -Sitosterol, <b>1d</b>  $\beta$ -Sitosterol-3-acetate, <b>2d</b>	Goyal <i>et al.</i> , 1989 Goyal <i>et al.</i> , 1987 Goyal <i>et al.</i> , 1985 Goyal <i>et al.</i> , 1987 Goyal <i>et al.</i> , 1985 Goyal <i>et al.</i> , 1989 Goyal <i>et al.</i> , 1987 Goyal <i>et al.</i> , 1985 Goyal <i>et al.</i> , 1989

Table 23 (Continued)

Scientific name	Investigated Part	Compound	Bibliography
<i>T. populnea</i>	Leaves	Lupeol -3-acetate, <b>3e</b> Nanadecane, <b>1a</b> Eicosane, <b>2a</b> Heneicosane, <b>3a</b> Docosane, <b>4a</b> Tricosane, <b>5a</b> Tetracosane, <b>6a</b> Pentacosane, <b>7a</b> Hexacosane, <b>8a</b> Heptacosane, <b>9a</b> Octacosane, <b>10a</b> Nanacosane, <b>11a</b> Triacosane, <b>12a</b> Dotriacontane, <b>13a</b> Hentriacontane, <b>14a</b>	Goyal <i>et al.</i> , 1989 Goyal <i>et al.</i> , 1987

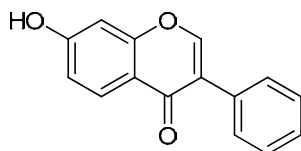
## Structure

### a: Alkanes

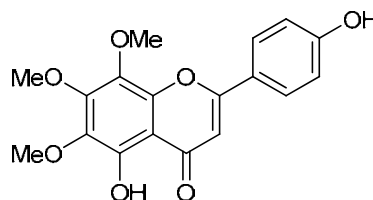


- |                                |                                    |
|--------------------------------|------------------------------------|
| <b>1a:</b> n = 17; Nanadecane  | <b>8a:</b> n = 24; Xexacosane      |
| <b>2a:</b> n = 18; Eicosane    | <b>9a:</b> n = 25; Heptacosane     |
| <b>3a:</b> n = 19; Heneicosane | <b>10a:</b> n = 26; Octacosane     |
| <b>4a:</b> n = 20; Docosane    | <b>11a:</b> n = 27; Nanacosane     |
| <b>5a:</b> n = 21; Tricosane   | <b>12a:</b> n = 28; Triacontane    |
| <b>6a:</b> n = 22; Tetracosane | <b>13a:</b> n = 30; Dotriacontane  |
| <b>7a:</b> n = 23; Pentacosane | <b>14a:</b> n = 31; Hentriacontane |

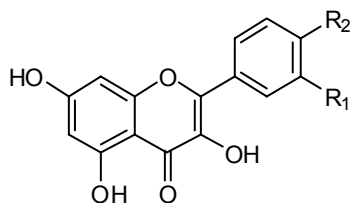
### b: Flavonoids



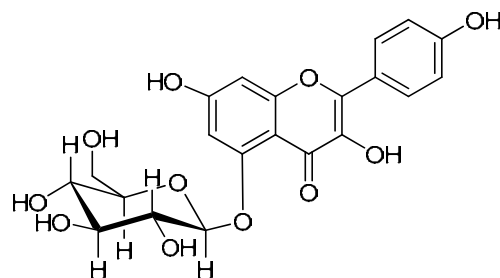
**1b:** 7-Hydroxyisoflavone



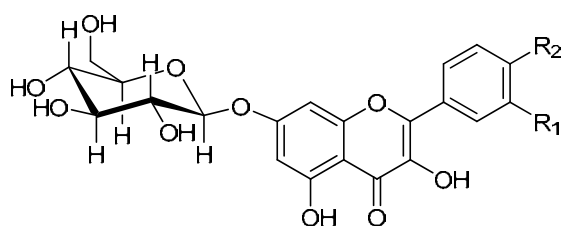
**2b:** Calcyopterin



- 3b:** R<sub>1</sub> = R<sub>2</sub> = OH; Quercetin  
**4b:** R<sub>1</sub> = H, R<sub>2</sub> = OH; Kaempferol  
**5b:** R<sub>1</sub> = OH, R<sub>2</sub> = H; Herbacetin  
**6b:** R<sub>1</sub> = OH, R<sub>2</sub> = OMe; Tamarixetin



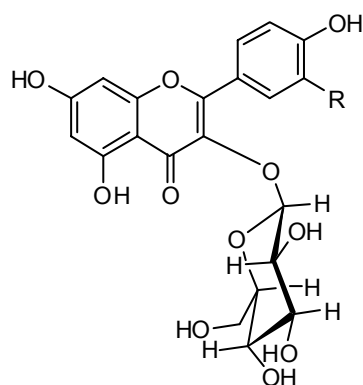
**7b:** Kaempferol-5-O- $\beta$ -glucoside



**8b:**  $R_1 = \text{OH}$ ,  $R_2 = \text{OMe}$ ; Tamarixetin-7-O- $\beta$ -glucoside

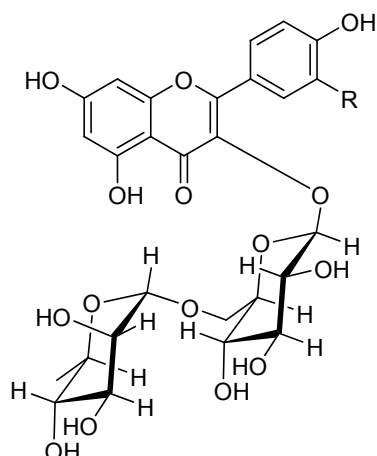
**9b:**  $R_1 = R_2 = \text{OH}$ ; Quercetin-7-O- $\beta$ -glucoside

**10b:**  $R_1 = \text{H}$ ,  $R_2 = \text{OH}$ ; Kaempferol-7-O- $\beta$ -glucoside



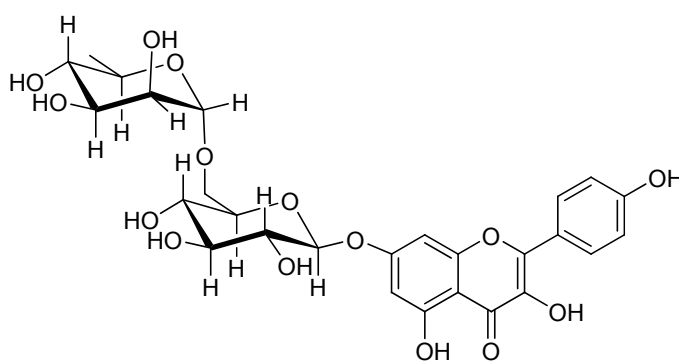
**11b:**  $R = \text{H}$ ; Kaempferol-3-O- $\beta$ -glucoside

**12b:**  $R = \text{OH}$ ; Quercetin-3-O- $\beta$ -glucoside



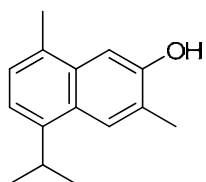
**13b:** R = H; Kaempferol-3-O- $\beta$ -rutinoside

**14b:** R = OH; Quercetin-3-O- $\beta$ -rutinoside

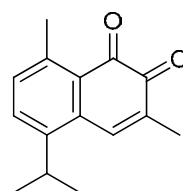


**15b:** Kaempferol -7-O- $\beta$ -rutinoside

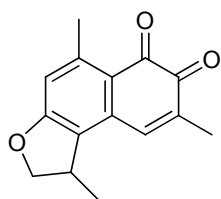
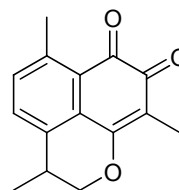
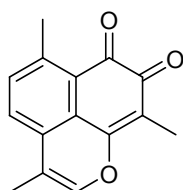
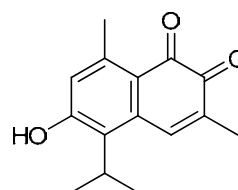
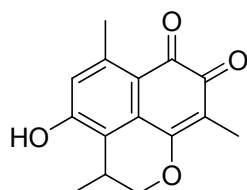
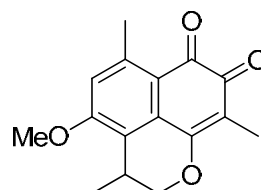
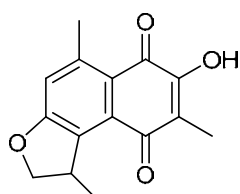
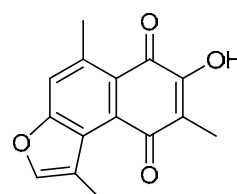
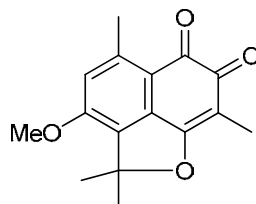
### C: Sesquiterpenes

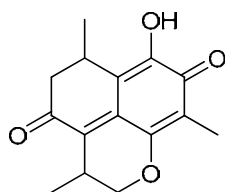


**1c:** 7-Hydroxycadalene

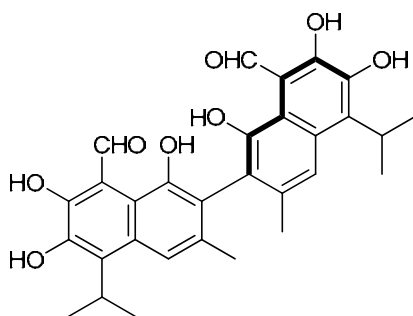


**2c:** Mansonone C

**3c:** Mansonone D**4c:** Mansonone E**5c:** Mansonone F**6c:** Mansonone G**7c:** Mansonone H**8c:** Mansonone M**9c:** Thespesone**10c:** Thespesenone**11c:** Dehydrooxoperezinone-6- methyl ether

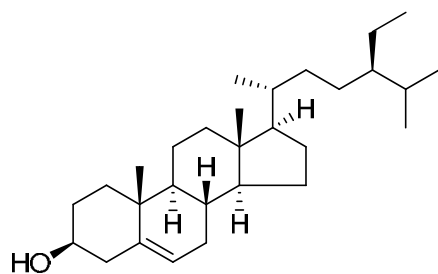


**12c:** 7-Hydroxy-2,3,5,6-tetrahydro-3,6,9-trimethyl-naphtho[1,8-b,c]pyran-4,8-dione

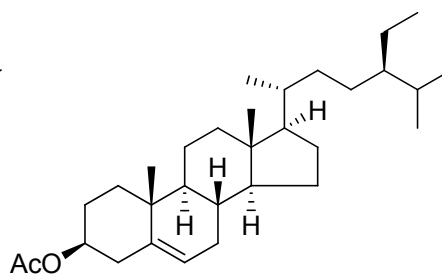


**13c:** (+)-Gossypol

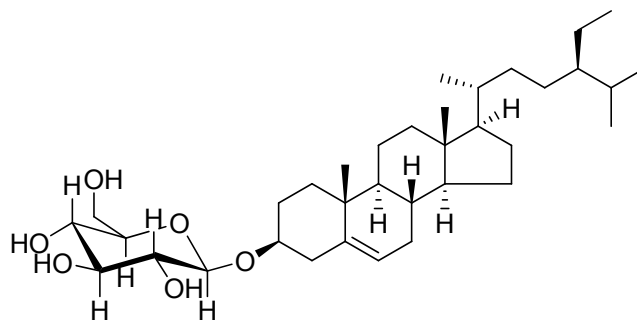
#### D: Steroids



**1d:**  $\beta$ -Sitosterol

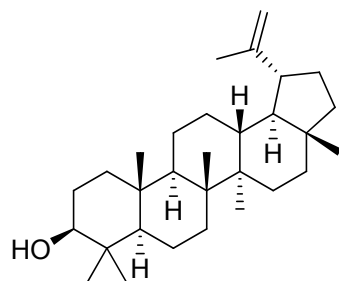
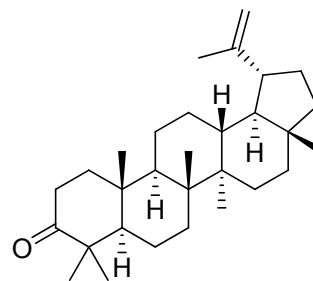
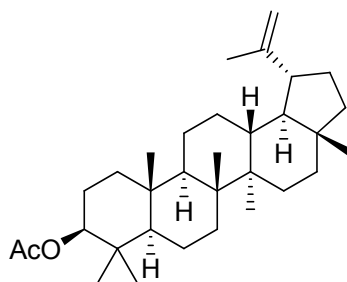


**2d:**  $\beta$ -Sitosterol-3-acetate



**3d:**  $\beta$ -Sitosterol-3- $\beta$ -D-glucoside



**E: Triterpenes****1e:** Lupeol**2e:** Lupenone**3e:** Lupeol-3-acetate

### **2.1.3 The objectives**

The goals of this work were to investigate the chemical constituents from the roots of *T. populnea* and to evaluate the antibacterial and cytotoxic activities of the isolated compounds.

## CHAPTER 2.2

### EXPERIMENTAL

#### 2.2.1 Instruments and Chemicals

Melting point was recorded in °C on an Electrothermal 9100 melting point apparatus. Ultraviolet (UV) absorption spectra were recorded using a SPECORD S100 spectrophotometer (Analytikjena) and principle bands ( $\lambda_{\max}$ ) were recorded as wavelengths (nm) and  $\log \varepsilon$  in methanol solution. The infrared spectra were recorded using FTS 165 FT-IR Perkin Elmer spectrophotometer. Nuclear Magnetic resonance spectra were recorded using Bruker Avance 300 MHz Bruker FTNMR Ultra Shield™. Spectra were recorded in deuteriochloroform, deuterioacetone and deuteromethanol and were recorded as  $\delta$  value in ppm downfield from TMS (Internal standard  $\delta$  0.00). Optical rotation was measured in MeOH solution at the sodium D line (590 nm) on an AUTOPOL<sup>R</sup> II automatic polarimeter. The EI-MS and HREIMS mass spectra were obtained from a Micromass LCT mass spectrometer. Solvent for extraction and chromatography were distilled at their boiling point ranges prior to use except chloroform was analytical grade reagent. Quick column chromatography (QCC) and column chromatography (CC) were carried out on silica gel 60 F<sub>254</sub> (Merck) and silica gel 100, respectively. Precoated plates of silica gel 60 GF<sub>254</sub> were used for analytical purposes.

## 2.2.2 Plant Material

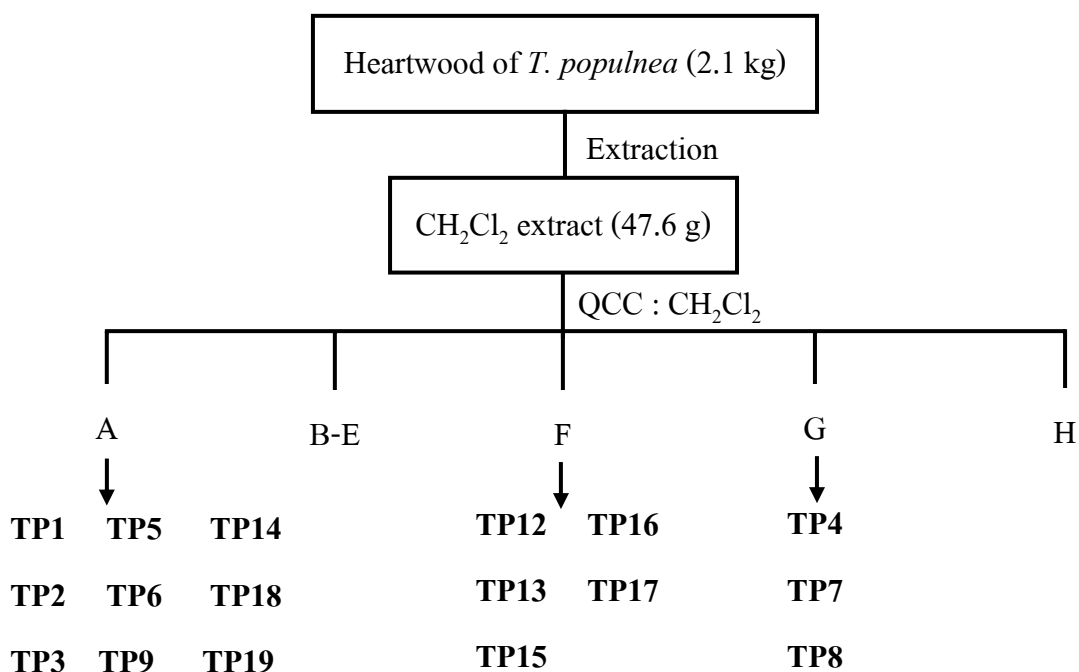
The fresh stem of *T. populnea* was collected from Suratthani Province, Thailand, in 2005. The plant was identified by Prof. Puangpen Sirirugsa and a voucher specimen (no. SB 01-001) has been deposited at the Herbarium of Department of Biology, Prince of Songkla University (PSU).

## 2.2.3 Extraction and chemical investigation from the stem of *T. populnea*

The stem of *T. populnea* was divided to two parts: heartwood and wood.

### 2.2.3.1 Extraction and investigation of the crude dichloromethane extract from the heartwood of *T. populnea*

The air-dried heartwood of *T. populnea* (2.10 kg) was extracted with  $\text{CH}_2\text{Cl}_2$  over a period of 5 days at room temperature. Evaporation of the solvent under reduced pressure furnished a dark residue (37.5 g).

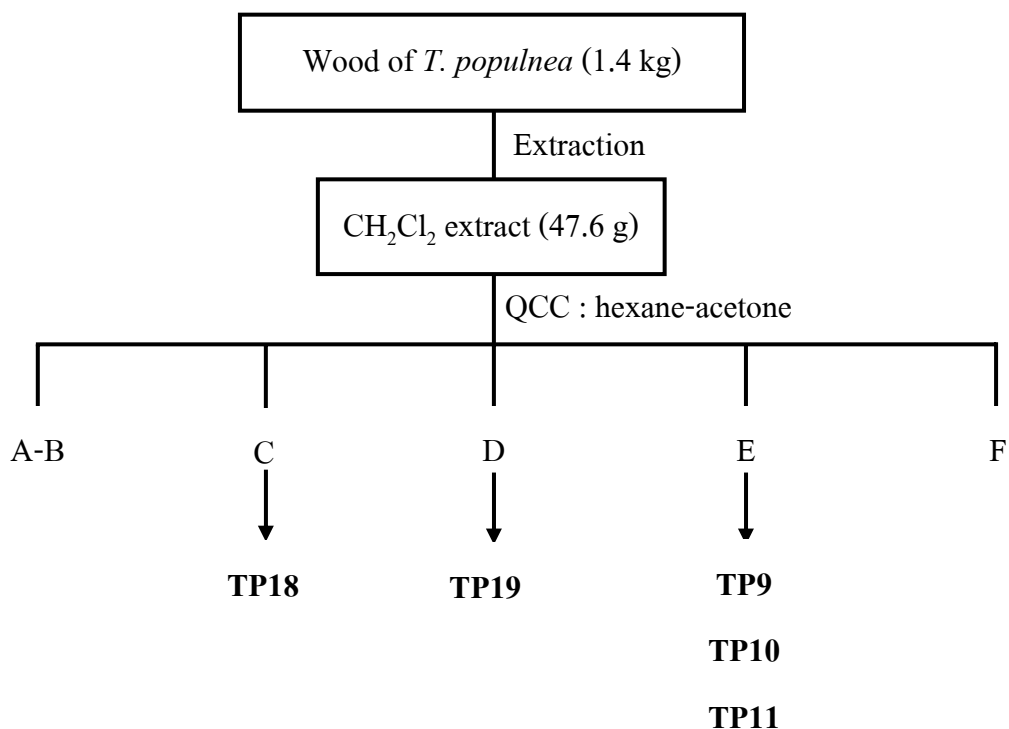


**Scheme 2** Extraction and isolation of compounds **TP1-TP8** and **TP12-TP19** from the heartwood of *T. populnea*

The crude dichloromethane extract was subjected to QCC on silica gel, eluting with  $\text{CH}_2\text{Cl}_2$  and separated into 8 fractions (A-H). Fraction A was purified by QCC using a gradient of hexane-acetone to afford nine subfractions ( $\text{A}_1$ - $\text{A}_9$ ). Subfraction  $\text{A}_2$  and  $\text{A}_3$  were combined and purified by QCC using a gradient of acetone-hexane as a mobile phase to give **TP1** (10.2 mg), **TP2** (2.5 mg) and **TP5** (8.3 mg). Subfraction  $\text{A}_5$  and  $\text{A}_6$  were combined and then purified by QCC with a gradient system of acetone-hexane to afford **TP14** (2.0 mg) and **TP3** (2.0 mg). Subfraction  $\text{A}_7$  and  $\text{A}_8$  were separately purified by QCC using a gradient of  $\text{CH}_2\text{Cl}_2$ -hexane as a mobile phase to yield **TP19** (4.0 mg) from  $\text{A}_7$  and **TP6** (4.5 mg), **TP9** (18.1 mg) and **TP18** (3.3 mg) from  $\text{A}_8$ . Fraction F was separated by QCC with a gradient system of increasing polarity ( $\text{CH}_2\text{Cl}_2$ -hexane) to afford nine subfractions ( $\text{F}_1$ -  $\text{F}_9$ ). Subfraction  $\text{F}_4$  was further purified by QCC using a gradient of  $\text{CH}_2\text{Cl}_2$ - hexane to give **TP12** (10.0 mg) and **TP13** (14.9 mg). Subfraction  $\text{F}_6$  was subjected to QCC using 20% acetone in hexane to afford four subfractions ( $\text{F}_{6\text{A}}$ - $\text{F}_{6\text{D}}$ ). Subfraction  $\text{F}_{6\text{B}}$  was further separated by QCC with a solvent system of 2% acetone- $\text{CHCl}_3$  to afford **TP15** (12.6 mg). Subfraction  $\text{F}_{6\text{C}}$ , upon standing overnight at room temperature gave yellow solid of **TP17** (4.2 mg) and the mother liquor gave **TP16** (4.1 mg). Fraction G was purified by QCC with a gradient of acetone- $\text{CH}_2\text{Cl}_2$  to give five subfractions ( $\text{G}_\text{A}$ - $\text{G}_\text{E}$ ). Subfraction  $\text{G}_\text{A}$  was subjected to precoated TLC using 50%  $\text{CH}_2\text{Cl}_2$ -hexane as a mobile phase (4 runs) to give **TP7** (5.1 mg). Subfraction  $\text{G}_\text{C}$  gave **TP4** (93.0 mg). Fraction H, upon standing overnight at room temperature gave red-brown crystal of **TP8** (30.5 mg).

### **2.2.3.2 Extraction and investigation of the crude dichloromethane extract from the wood of *T. populnea***

The air-dried wood of *T. populnea* (1.40 kg) was extracted with  $\text{CH}_2\text{Cl}_2$  over a period of 5 days at room temperature. Evaporation of the solvent under reduced pressure furnished a dark-green residue (10.2 g) of the  $\text{CH}_2\text{Cl}_2$  extract.



**Scheme 3** Extraction and isolation of compounds **TP9-TP11** and **TP18-TP19** from the wood of *T. populnea*

The CH<sub>2</sub>Cl<sub>2</sub> extract was subjected to QCC on silica gel, and eluted with a gradient of hexane - acetone to give six fractions (A-F). Fraction C was then purified by QCC using a gradient of hexane-acetone to afford **TP18** (22.6 mg). Fraction D, upon standing overnight at room temperature gave **TP19** (20.3 mg). Fraction E was separated by QCC with a gradient system of increasing polarity (acetone-hexane) to afford five subfractions (E<sub>1</sub>- E<sub>5</sub>). Subfraction E<sub>2</sub> was subjected to precoated plates using 50% CH<sub>2</sub>Cl<sub>2</sub>-hexane as a mobile phase (4 runs) to give **TP9** (1.6 mg). Subfraction E<sub>3</sub> was subjected to precoated plates using 3% MeOH-CH<sub>2</sub>Cl<sub>2</sub> as a mobile phase (4 runs) to give **TP10** (2.3 mg) and **TP11** (2.1 mg).

**Compound TP1:** Brown solid; mp 107-109 °C; UV (MeOH)  $\lambda_{\max}$  (log  $\epsilon$ ) 225 (4.81), 235 (4.83), 276 sh (3.87), 286 (3.97), 299 (3.94), 320 (3.60), 334 (3.66) nm; IR (KBr)  $\nu_{\max}$  3328, 2952, 2863, 1623, 1440, 1237 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz) and <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz), see **Table 24**.

**Compound TP2:** Orange solid; mp 123-125 °C; UV (MeOH)  $\lambda_{\max}$  (log  $\epsilon$ ) 213 (4.38), 257 (4.44), 365 sh (3.45), 432 (3.60) nm; IR (KBr)  $\nu_{\max}$ : 1670, 1665, 1381, 1241  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz) and  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz), see **Table 25**.

**Compound TP3:** Orange solid; mp 196-198 °C; UV (MeOH)  $\lambda_{\max}$  (log  $\epsilon$ ) 217 (4.36), 240 (4.15), 273 (4.23), 410 (3.90) nm; IR (neat)  $\nu_{\max}$ : 3328, 1717, 1646, 1254, 1131  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3+\text{CD}_3\text{OD}$ , 300 MHz) and  $^{13}\text{C}$  NMR ( $\text{CDCl}_3+\text{CD}_3\text{OD}$ , 75 MHz), see **Table 27**.

**Compound TP4:** Yellow solid;  $[\alpha]_{\text{D}}^{25}$  -39.0 (*c* 8.25,  $\text{CHCl}_3$ ); mp 159-161 °C; UV (MeOH)  $\lambda_{\max}$  (log  $\epsilon$ ) 219 (4.23), 242 (4.06), 277 (4.06), 404 (3.81) nm; IR (KBr)  $\nu_{\max}$ : 1675, 1550  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz) and  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz), see **Table 29**.

**Compound TP5:** Yellow solid;  $[\alpha]_{\text{D}}^{25}$  -252.8 (*c* 0.09,  $\text{CHCl}_3$ ); mp 135-137 °C; UV (MeOH)  $\lambda_{\max}$  (log  $\epsilon$ ) 213 (4.27), 274 (4.23), 301 (4.15), 358 (3.71) nm; IR (neat)  $\nu_{\max}$ : 3328, 1642, 1597, 1560, 1344, 1243, 1109  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz) and  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz), see **Table 33**.

**Compound TP6:** Yellow solid;  $[\alpha]_{\text{D}}^{25}$  -42.7 (*c* 0.08,  $\text{CHCl}_3$ ); UV (MeOH)  $\lambda_{\max}$  (log  $\epsilon$ ) 214 (4.31), 242 sh (4.05), 273 (4.01), 336 (3.60), 409 (3.62) nm; IR (neat)  $\nu_{\max}$ : 3373, 2955, 2925, 2873, 1709, 1664, 1649, 1254  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz) and  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz), see **Table 35**.

**Compound TP7:** Reddish brown solid;  $[\alpha]_{\text{D}}^{25}$  +326.2 (*c* 0.15,  $\text{CHCl}_3$ ); mp 259-261 °C; UV (MeOH)  $\lambda_{\max}$  (log  $\epsilon$ ) 217 (4.70), 265 sh (4.59), 274 (4.64), 298 (4.40), 364 (4.22), 385 (4.22) nm; IR (KBr)  $\nu_{\max}$ : 3188, 2974, 2923, 1668, 1561, 1266, 1229, 1188  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz) and  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz), see **Table 37**.

**Compound TP8:** Reddish brown solid;  $[\alpha]_D^{25} +736.6$  ( $c$  0.35,  $\text{CHCl}_3$ ); mp 264-266 °C (decomposed); UV (MeOH)  $\lambda_{\text{max}}$  (log  $\epsilon$ ) 218 (4.26), 265 sh (4.23), 274 (4.29), 300 (4.04), 392 (3.92) nm; IR (KBr)  $\nu_{\text{max}}$  3187, 2985, 1668, 1627, 1560, 1265, 1228, 948  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz) and  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz), see **Table 39**.

**Compound TP9:** Reddish brown solid;  $[\alpha]_D^{25} +58.1$  ( $c$  1.27,  $\text{CHCl}_3$ ); mp 104-106 °C; UV (MeOH)  $\lambda_{\text{max}}$  (log  $\epsilon$ ) 218 (4.13), 263(4.14), 432 (3.27) nm; IR (neat)  $\nu_{\text{max}}$  2962, 2925, 1683, 1634, 1616, 1176, 754  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz) and  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz), see **Table 41**.

**Compound TP10:** Yellow gum;  $[\alpha]_D^{25} +57.9$  ( $c$  0.54,  $\text{CHCl}_3$ ); UV (MeOH)  $\lambda_{\text{max}}$  (log  $\epsilon$ ) 216 (4.22), 251 (3.98), 259 (3.91), 279 (3.44), 289 (3.40) nm; IR (Neat)  $\nu_{\text{max}}$  3365, 2959, 2870, 1617, 1591, 758  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz) and  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz), see **Table 45**; EIMS  $m/z$ , 246  $[\text{M}]^+$  (8), 211 (18), 185 (33), 169 (25), 72 (100), 69 (47); HREIMS  $m/z$  246.1262 (calcd for  $\text{C}_{15}\text{H}_{18}\text{O}_3$ , 246.1256).

**Compound TP11:** Yellow gum;  $[\alpha]_D^{25} -63.6$  ( $c$  0.37,  $\text{CHCl}_3$ ); UV (MeOH)  $\lambda_{\text{max}}$  (log  $\epsilon$ ) 213 (4.15), 251 (3.85), 259 (3.80), 278 (3.32), 290 (3.29) nm; IR (Neat)  $\nu_{\text{max}}$  3387, 2959, 2871, 1716, 1524, 754  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz) and  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz), see **Table 46**; EIMS  $m/z$ , 246  $[\text{M}]^+$  (50), 199 (31), 185 (100), 157 (23), 129 (46); HREIMS  $m/z$  246.1255 (calcd for  $\text{C}_{15}\text{H}_{18}\text{O}_3$ , 246.1256).

**Compound TP12:** Orange solid; mp 168-170 °C;  $[\alpha]_D^{25} -46.0$  ( $c$  0.27,  $\text{CHCl}_3$ ); UV (MeOH)  $\lambda_{\text{max}}$  (log  $\epsilon$ ) 213 (4.18), 242 (3.79), 259 (3.98), 380 (3.03) nm; IR (Neat)  $\nu_{\text{max}}$  2974, 2930, 2871, 1757, 1698, 1657  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz) and  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz), see **Table 47**; EIMS  $m/z$ , 286.1556  $[\text{M}+2]^+$  (17), 271 (53), 241 (72), 85 (66), 83 (100); HREIMS  $m/z$  286.1556  $[\text{M}+2]^+$  (calcd for  $\text{C}_{18}\text{H}_{24}\text{O}_3$ , 284.1412).

**Compound TP13:** Brown gum;  $[\alpha]_D^{25} -21.9$  ( $c$  0.75,  $\text{CHCl}_3$ ); UV (MeOH)  $\lambda_{\text{max}}$  (log  $\epsilon$ ) 219 (4.10), 264 (3.92), 277sh (3.81), 366 (2.86) nm; IR (Neat)  $\nu_{\text{max}}$  3417,



2967, 2930, 2863, 1653, 754  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz) and  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz), see **Table 48**; EIMS  $m/z$ , 288  $[\text{M}]^+$  (15), 274 (21), 241 (20), 273 (100); HREIMS  $m/z$  288.1736 (calcd for  $\text{C}_{18}\text{H}_{24}\text{O}_3$ , 288.1725).

**Compound TP14:** Yellow-brown gum;  $[\alpha]_{\text{D}}^{25} +30.1$  ( $c$  0.58,  $\text{CHCl}_3$ ); UV (MeOH)  $\lambda_{\text{max}}$  ( $\log \epsilon$ ) 228 (4.11), 273 (3.86) nm; IR (Neat)  $\nu_{\text{max}}$  3410, 2970, 2925, 2873, 1776, 1675, 1616  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz) and  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz), see **Table 49**; EIMS  $m/z$ , 262  $[\text{M}]^+$  (31), 220 (34), 191 (43), 219 (100); HREIMS  $m/z$  262.1210 (calcd for  $\text{C}_{15}\text{H}_{18}\text{O}_4$ , 262.1205).

**Compound TP15:** Yellow gum;  $[\alpha]_{\text{D}}^{25} +7.5$  ( $c$  0.23,  $\text{CHCl}_3$ ); UV (MeOH)  $\lambda_{\text{max}}$  ( $\log \epsilon$ ) 219 (4.23), 232 (4.14), 281 (3.00) nm; IR (Neat)  $\nu_{\text{max}}$  3417, 2967, 2930, 2871, 1668, 1576  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz) and  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz), see **Table 50**; EIMS  $m/z$ , 264  $[\text{M}]^+$  (27), 221 (100), 203 (22), 193 (26), 179 (44), 177 (25), 151 (20); HREIMS  $m/z$  264.1353 (calcd for  $\text{C}_{15}\text{H}_{20}\text{O}_4$ , 264.1362).

**Compound TP16:** Yellow gum;  $[\alpha]_{\text{D}}^{25} +62.7$  ( $c$  0.07,  $\text{CHCl}_3$ ); UV (MeOH)  $\lambda_{\text{max}}$  ( $\log \epsilon$ ) 214 (4.09), 235 (3.98), 286 (3.95), 339 (3.66) nm; IR (Neat)  $\nu_{\text{max}}$  3424, 2959, 2930, 2871, 1661, 1591, 1429  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz) and  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz), see **Table 51**; EIMS  $m/z$ , 278  $[\text{M}]^+$  (98), 249 (27), 239 (100), 208 (36), 192 (35); HREIMS  $m/z$  278.1196 (calcd for  $\text{C}_{15}\text{H}_{18}\text{O}_5$ , 278.1154).

**Compound TP17:** Yellow gum;  $[\alpha]_{\text{D}}^{25} +43.7$  ( $c$  0.04,  $\text{CHCl}_3$ ); UV (MeOH)  $\lambda_{\text{max}}$  ( $\log \epsilon$ ) 214 (4.06), 237 (3.95), 286 (3.96), 339 (3.60) nm; IR (Neat)  $\nu_{\text{max}}$  3417, 2967, 2930, 2871, 1661, 1587  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz) and  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz), see **Table 52**; EIMS  $m/z$ , 278  $[\text{M}]^+$  (54), 234 (56), 208 (25), 192 (24), 72 (100); HREIMS  $m/z$  278.1159 (calcd for  $\text{C}_{15}\text{H}_{18}\text{O}_5$ , 278.1154).

**Compound TP18:** Yellow solid;  $[\alpha]_{\text{D}}^{25} +417.7$  ( $c$  0.49,  $\text{CHCl}_3$ ); mp 171-173  $^{\circ}\text{C}$ ; UV (MeOH)  $\lambda_{\text{max}}$  ( $\log \epsilon$ ) 237 (4.26), 276 sh (3.90), 290 (3.84), 379 (3.61) nm; IR

(neat)  $\nu_{\max}$  3410, 2959, 2930, 1626, 1314, 754  $\text{cm}^{-1}$ ;  $^1\text{H NMR}$  ( $\text{CDCl}_3$ , 300 MHz) and  $^{13}\text{C NMR}$  ( $\text{CDCl}_3$ , 75 MHz), see **Table 53**.

**Compound TP19:** Yellow solid;  $[\alpha]_{\text{D}}^{25} +246.9$  ( $c$  0.14,  $\text{CHCl}_3$ ); mp 165-167  $^{\circ}\text{C}$ ; UV (MeOH)  $\lambda_{\max}$  (log  $\epsilon$ ) 229 (4.77), 252 sh (4.65), 286 (4.44), 360 (4.00) nm; IR (neat)  $\nu_{\max}$  3373, 2962, 2932, 1608, 1444, 1332, 754  $\text{cm}^{-1}$ ;  $^1\text{H NMR}$  ( $\text{CDCl}_3$ , 300 MHz) and  $^{13}\text{C NMR}$  ( $\text{CDCl}_3$ , 75 MHz), see **Table 55**.

## 2.2.4 BIOASSAY

### 2.2.4.1 Antibacterial assay

The compounds isolated from *T. populnea* were tested against the microorganisms *Bacillus subtilis* (obtained from Department of Industrial Biotechnology, PSU), *Staphylococcus aureus* (TISTR517) (obtained from Microbial Resources Center (MIRCEN), Bangkok, Thailand), *Pseudomonas aeruginosa*, *Enterococcus faecalis*, *Shigella sonnei* and *Salmonella typhi*. The last four microorganisms were obtained from Department of Pharmacognosy and Botany, PSU. The antibacterial assay employed was the same as described in Boonsri *et al.*, (Boonsri *et al.*, 2006). Vancomycin, which was used as a standard, showed antibacterial activity of 0.078  $\mu\text{g}/\text{mL}$ .

### 2.2.4.2 Cytotoxic assay

The procedure for the cytotoxic assay was performed by the sulphorhodamine B (SRB) assay as described by Skehan *et al.*, (Skehan *et al.*, 1990). In this study, four cancer cell lines obtained from the National Cancer Institute, Bangkok, Thailand, were used: MCF-7 (breast adenocarcinoma), KB (human oral cancer), HeLa (human cervical cancer) and HT-29 (colon cancer). Camptothecin, which was used as a standard, showed cytotoxic activity in the range of 0.2-2.0  $\mu\text{g}/\text{mL}$ .

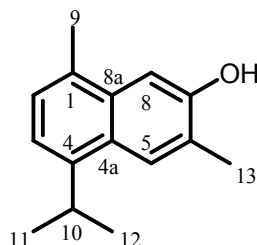
## CHAPTER 2.3

### RESULTS AND DISCUSSION

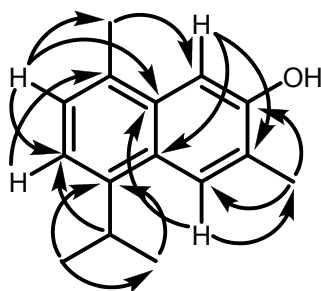
#### 2.3.1 Structural elucidation of the isolated compounds from the wood and the heartwood of *T. populnea*

The CH<sub>2</sub>Cl<sub>2</sub> extracts of the wood and the heartwood of *T. populnea* were subjected to chromatography to give compounds **TP1-TP19**. Two new compounds, **TP10** and **TP11**, along with three known compounds, **TP9**, **TP18** and **TP19**, were purified from the wood. Six new compounds, **TP12-TP17**, were obtained from the dark heartwood, together with eleven known compounds, **TP1-TP9, TP18-TP19**. Their structures were elucidated on the basis of spectroscopic data.

### 2.3.2.1 Compound TP1

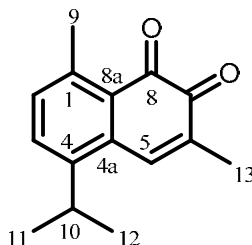


**TP1** was obtained as brown solid. The UV spectrum (**Figure 40**) exhibited the absorption bands at 225, 235, 276, 286, 299, 320 and 344 nm. The IR spectrum (**Figure 41**) indicated the presence of hydroxyl functionality ( $3328\text{ cm}^{-1}$ ). The  $^{13}\text{C}$  NMR and DEPT data showed 15 carbons, ten aromatic carbons, four methyls and one benzylic methine, suggesting a cadalene sesquiterpene (Silva *et al.*, 2006). The  $^1\text{H}$  NMR spectrum of **TP1** (**Table 24**, **Figure 42**) displayed two *ortho*-coupled of aromatic protons at  $\delta$  7.13 (1H, *d*, 7.5 Hz, H-3) and 7.19 (1H, *d*, 7.5 Hz, H-2). Two singlet signals of aromatic protons at  $\delta$  7.89 (*s*, H-5) and 7.25 (*s*, H-8), suggesting that they were *para* to each other. This was confirmed by HMBC correlations of the low-field proton (H-5) with C-13 ( $\delta$  16.8) and C-4 ( $\delta$  142.2) and the upfield proton (H-8) with C-1 ( $\delta$  130.1), C-4a (126.9) and C-6 (125.1). In addition, the presence of two methyl groups [ $\delta$  2.47 (3H, *s*) and 2.56 (3H, *s*)] and one isopropyl moiety [ $\delta$  1.37 (6H, *d*, 6.6 Hz) and 3.67 (1H, *sept*, 6.6 Hz)] was evident by  $^1\text{H}$  and  $^{13}\text{C}$  NMR signals (**Table 24**, **Figure 42** and **43**). The methyl group at  $\delta$  2.47 was placed at C-6 because of its HMBC correlations to C-5 ( $\delta$  125.6), C-6 ( $\delta$  125.1) and C-7 ( $\delta$  152.1) and the methyl at  $\delta$  2.56 was placed at C-1 due to its HMBC correlations to C-2 ( $\delta$  126.2) and C-8a ( $\delta$  133.1). Finally, the isopropyl group was attached at C-4, judging from HMBC correlations of its methine proton at  $\delta$  3.67 (*sept*, 6.6 Hz) with C-3 (119.1), C-4 (142.2) and C-4a (126.9). Thus, **TP1** was identified as 7-hydroxycadalene (Lindgren *et al.*, 1968).

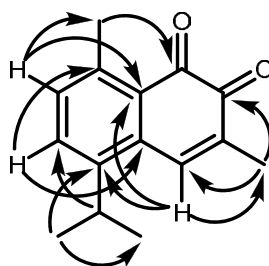
Selected HMBC correlations of **TP1****Table 24**  $^1\text{H}$ ,  $^{13}\text{C}$  NMR, DEPT and HMBC spectral data of **TP1**

position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	DEPT	HMBC
1		130.1	C	
2	7.19 ( <i>d</i> , 7.5)	126.2	CH	3, 4, 8a, 9
3	7.13 ( <i>d</i> , 7.5)	119.1	CH	1, 4a, 9, 10
4		142.2	C	
4a		126.9	C	
5	7.89 ( <i>s</i> )	125.6	CH	13, 4, 8a
6		125.1	C	
7		152.1	C	
8	7.25 ( <i>s</i> )	106.9	CH	1, 4a, 6, 13
8a		133.1	C	
9	2.56 ( <i>s</i> )	19.5	CH <sub>3</sub>	3, 8a
10	3.67 ( <i>sept</i> , 6.6)	28.4	CH	3, 4, 4a
11	1.37 ( <i>d</i> , 6.6)	23.7	CH <sub>3</sub>	4, 10, 12
12	1.37 ( <i>d</i> , 6.6)	23.7	CH <sub>3</sub>	4, 10, 11
13	2.47 ( <i>s</i> )	16.8	CH <sub>3</sub>	5, 6, 7

### 2.3.2.2 Compound TP2



**TP2** was isolated as an orange solid. The IR spectrum (**Figure 45**) exhibited the characteristic absorption of carbonyl groups at 1665 and 1670  $\text{cm}^{-1}$ . The UV spectrum (**Figure 44**) showed absorption maxima at 213, 257, 259 and 380 nm. The  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectral data (**Table 25**, **Figures 46** and **47**) were comparable to those of **TP1**, except for the replacement of an aromatic proton H-8 at  $\delta$  7.25 and a hydroxyl group at C-7 in **TP1** with the carbonyl carbon at  $\delta$  182.0 and 182.8, suggesting an *o*-naphthoquinone cadinane skeleton. This was supported by its color, IR spectrum and UV absorption bands (Zhang *et al.*, 2007). The two carbonyl groups were placed at C-7 ( $\delta$  182.0) and C-8 ( $\delta$  182.8) by  $^3J$  correlations of the methyl group at  $\delta$  2.08 (Me-13) to C7 and  $^4J$  correlation of the methyl group at  $\delta$  2.63 (Me-9) to C-8 in HMBC experiment. Accordingly, **TP2** was identified as mansonone C (Kraus *et al.*, 2006).



Selected HMBC correlations of **TP2**

**Table 25**  $^1\text{H}$ ,  $^{13}\text{C}$  NMR, DEPT and HMBC spectral data of **TP2**

position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	DEPT	HMBC
1		143.0	C	
2	7.19 ( <i>d</i> , 8.1)	134.1	CH	3, 8a, 9
3	7.43 ( <i>d</i> , 8.1)	131.9	CH	1, 2, 4a
4		145.3	C	
4a		132.5	C	
5	7.66 ( <i>br d</i> , 1.5)	138.0	CH	4, 8a, 6, 13
6		135.0	C	
7		182.0	C	
8		182.8	C	
8a		129.3	C	
9	2.63 ( <i>s</i> )	22.8	CH <sub>3</sub>	1, 2, 8, 8a
10	3.39 ( <i>sept</i> , 6.9)	28.3	CH	3, 4, 11, 12
11	1.30 ( <i>d</i> , 6.9)	23.7	CH <sub>3</sub>	4, 10, 12
12	1.30 ( <i>d</i> , 6.9)	23.7	CH <sub>3</sub>	4, 10, 11
13	2.08 ( <i>d</i> , 1.5)	16.0	CH <sub>3</sub>	5, 6, 7

**Table 26** Comparison of  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectral data of **TP2** and mansonone C

position	TP2		mansonone C <sup>a</sup>	
	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$
1		143.0		143.2
2	7.19 ( <i>d</i> , 8.1)	134.1	7.20 ( <i>d</i> , 8.0)	134.3
3	7.43 ( <i>d</i> , 8.1)	131.9	7.44 ( <i>d</i> , 8.0)	132.2
4		145.3		145.5
4a		132.5		132.6
5	7.66 ( <i>br d</i> , 1.5)	138.0	7.66 ( <i>s</i> )	138.2
6		135.0		135.2

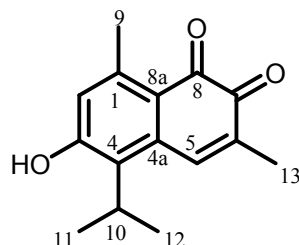
**Table 26** (Continued) mansonone C

position	TP2		mansonone C <sup>a</sup>	
	$\delta_{\text{H}}$ (mult., $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	$\delta_{\text{H}}$ (mult., $J_{\text{Hz}}$ )	$\delta_{\text{C}}$
7		182.0		182.2
8		182.8		182.5
8a		129.3		129.5
9	2.63 ( <i>s</i> )	22.8	2.64 ( <i>s</i> )	23.1
10	3.39 ( <i>sept</i> , 6.9)	28.3	3.43-3.36 ( <i>m</i> )	28.5
11	1.30 ( <i>d</i> , 6.9)	23.7	1.30 ( <i>d</i> , 6.8)	24.0
12	1.30 ( <i>d</i> , 6.9)	23.7	1.30 ( <i>d</i> , 6.8)	24.0
13	2.08 ( <i>d</i> , 1.5)	16.0	2.09 ( <i>s</i> )	16.3

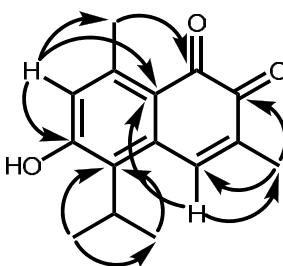
<sup>a</sup> recorded in CDCl<sub>3</sub>



### 2.3.2.3 Compound TP3



**TP3** was isolated as an orange solid. The IR spectrum (**Figure 49**) showed the absorption bands at 1646, 1717 and 3328  $\text{cm}^{-1}$  corresponding to two carbonyl and hydroxyl groups, respectively. The UV spectrum (**Figure 48**) showed the absorption maxima at 217, 240, 273 and 410 nm. The  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra of **TP3** (**Table 27**, **Figures 50** and **51**) were closely resembled to those of **TP2**. In the  $^1\text{H}$  NMR spectrum (**Table 27**), an *ortho*-coupled proton at  $\delta$  7.43 (1H, *d*,  $J = 8.1$  Hz;  $\delta_{\text{c}}$  131.9) as found in **TP2** was missing in **TP3** but the signal due to  $sp^2$  oxyquaternary carbon at  $\delta$  162.2 was instead observed, whose down field signal suggested a connection to a hydroxyl group. The HMBC correlations of an aromatic proton at  $\delta$  6.56 (s, H-2) with C-8a ( $\delta$  122.7), C-4 ( $\delta$  133.2), C-3 ( $\delta$  162.2) and Me-9 ( $\delta$  23.3), supported the location of the hydroxyl group at C-3. Therefore, **TP3** was identified as mansonone G (Letcher *et al.*, 1992 and Puckhaber *et al.*, 2004).



Selected HMBC correlations of **TP3**

**Table 27**  $^1\text{H}$ ,  $^{13}\text{C}$  NMR, DEPT and HMBC spectral data of **TP3**

position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	DEPT	HMBC
1		146.6	C	
2	6.56 ( <i>s</i> )	119.9	CH	2, 3, 8a, 9
3		162.2	C	
4		133.2	C	
4a		134.5	C	
5	7.72 ( <i>s</i> )	139.1	CH	8a, 13
6		135.3	C	
7		182.8	C	
8		180.0	C	
8a		122.7	C	
9	2.58 ( <i>s</i> )	23.3	CH <sub>3</sub>	2, 8a, 8
10	3.58 ( <i>sept</i> , 7.2)	26.8	CH	
11	1.43 ( <i>d</i> , 7.2)	21.2	CH <sub>3</sub>	4, 10, 12
12	1.43 ( <i>d</i> , 7.2)	21.2	CH <sub>3</sub>	4, 10, 11
13	2.07 ( <i>s</i> )	15.9	CH <sub>3</sub>	5, 6, 7

**Table 28** Comparison of  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectral data of **TP3** and mansonone G

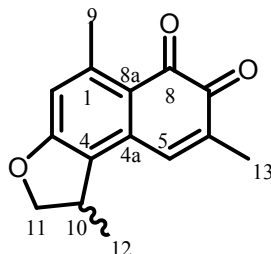
position	TP3		mansonone G	
	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ ) <sup>a</sup>	$\delta_{\text{C}}$ <sup>b</sup>
1		146.6		145.9
2	6.56 ( <i>s</i> )	119.9	6.49 ( <i>s</i> )	120.5
3		162.2		162.6
4		133.2		133.2
4a		134.5		135.8
5	7.72 ( <i>s</i> )	139.1	7.69 ( <i>br s</i> )	138.7
6		135.3		136.8

Table 28 (Continued)

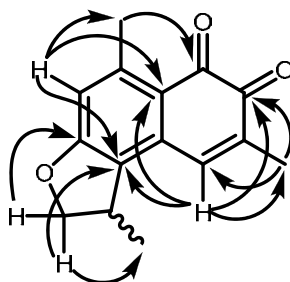
position	TP3		mansonone G	
	$\delta_{\text{H}}$ (mult., $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	$\delta_{\text{H}}$ (mult., $J_{\text{Hz}}$ ) <sup>a</sup>	$\delta_{\text{C}}$ <sup>b</sup>
7		182.8		182.9
8		180.0		180.9
8a		122.7		123.5
9	2.58 (s)	23.3	2.47 (s)	23.2
10	3.58 (sept, 7.2)	26.8	3.48 (sept, 7.0)	27.5
11	1.43 (d, 7.2)	21.2	1.38 (d, 7.0)	21.3
12	1.43 (d, 7.2)	21.2	1.38 (d, 7.0)	21.3
13	2.07 (s)	15.9	1.8 (s)	15.7

<sup>a</sup> recorded in CDCl<sub>3</sub>-CD<sub>3</sub>OD (1:1) <sup>b</sup> recorded in acetone-*d*<sub>6</sub>

### 2.3.2.4 Compound TP4



**TP4** was isolated as an orange solid. The UV (**Figure 52**) and IR spectra (**Figure 53**) showed absorption bands similar to those of **TP3**. The  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectral data (**Table 29**, **Figures 54** and **55**) of **TP4** were closely related to those of **TP3** except that methyl signal at  $\delta$  1.43 (*s*, Me-11) in **TP3** was replaced by oxymethylene protons resonating at  $\delta$  4.27 (*dd*,  $J = 8.7, 2.7$  Hz) and 4.64 (*t*,  $J = 8.7$  Hz) in **TP4**.  $^3J$  HMBC correlations between oxymethylene protons (H<sub>2</sub>-11) and C-3 ( $\delta$  165.3) of aromatic unit established the fusion by ether linkage at C-3. Accordingly, **TP4** was characterized as mansonone D (Puckhaber *et al.*, 2004).



Selected HMBC correlations of **TP4**

**Table 29**  $^1\text{H}$ ,  $^{13}\text{C}$  NMR, DEPT and HMBC spectral data of **TP4**

position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	DEPT	HMBC
1		149.4	C	
2	6.44 ( <i>s</i> )	113.3	CH	3, 4, 8a, 9
3		165.3	C	
4		131.1	C	
4a		132.9	C	
5	7.11 ( <i>s</i> )	137.4	CH	4, 4a, 6, 7, 8a, 13
6		136.6	C	
7		182.5	C	
8		178.7	C	
8a		122.4	C	
9	2.49 ( <i>s</i> )	23.6	CH <sub>3</sub>	1, 2, 8a
10	3.54 ( <i>dq</i> , 2.7, 7.2)	34.5	CH	3
11	4.27 ( <i>dd</i> , 8.7, 2.7) 4.64 ( <i>t</i> , 8.7)	80.0	CH <sub>2</sub>	3, 4, 10, 12
12	1.43 ( <i>d</i> , 7.2)	21.9	CH <sub>3</sub>	4, 10, 11
13	1.94 ( <i>s</i> )	15.7	CH <sub>3</sub>	5, 6, 7

**Table 30** Comparison of  $^{13}\text{C}$  NMR spectral data of **TP4** and mansonone D

Position	TP4	mansonone D <sup>a</sup>
	$\delta_{\text{C}}$	$\delta_{\text{C}}$
1	149.4	149.6
2	113.3	113.4
3	165.3	165.0
4	131.1	130.8
4a	132.9	132.9
5	137.4	137.4

**Table 30** (Continued)

Position	TP4	mansonone D <sup>a</sup>
	$\delta_{\text{C}}$	$\delta_{\text{C}}$
6	136.6	136.7
7	182.5	182.6
8	178.7	178.8
8a	122.4	122.5
9	23.6	23.8
10	34.5	34.6
11	80.0	79.9
12	21.9	22.0
13	15.7	15.8

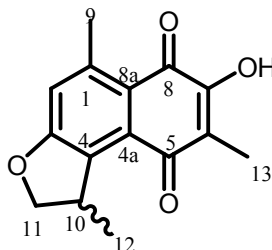
<sup>a</sup> recorded in CDCl<sub>3</sub>**Table 31** Comparison of <sup>1</sup>H NMR spectral data of TP1-TP4

Position	TP1	TP2	TP3	TP4
	$\delta_{\text{H}}$ ( <i>mult.</i> , J <sub>Hz</sub> )	$\delta_{\text{H}}$ ( <i>mult.</i> , J <sub>Hz</sub> )	$\delta_{\text{H}}$ ( <i>mult.</i> , J <sub>Hz</sub> )	$\delta_{\text{H}}$ ( <i>mult.</i> , J <sub>Hz</sub> )
2	7.19 ( <i>d</i> , 7.5)	7.19 ( <i>d</i> , 8.1)	6.56 ( <i>s</i> )	6.44 ( <i>s</i> )
3	7.13 ( <i>d</i> , 7.5)	7.43 ( <i>d</i> , 8.1)		
5	7.89 ( <i>s</i> )	7.66 ( <i>br d</i> , 1.5)	7.72 ( <i>s</i> )	7.11 ( <i>s</i> )
8	7.27 ( <i>s</i> )			
9	2.56 ( <i>s</i> )	2.63 ( <i>s</i> )	2.58 ( <i>s</i> )	2.49 ( <i>s</i> )
10	3.67 ( <i>sept</i> )	3.39 ( <i>sept</i> , 6.9)	3.58 ( <i>sept</i> , 7.2)	3.54 ( <i>dq</i> , 2.7, 7.2)
11	1.37 ( <i>d</i> , 6.6)	1.30 ( <i>d</i> , 6.9)	1.43 ( <i>d</i> , 7.2)	4.27 ( <i>dd</i> , 8.7, 2.7) 4.64 ( <i>t</i> , 8.7)
12	1.37 ( <i>d</i> , 6.6)	1.30 ( <i>d</i> , 6.9)	1.43 ( <i>d</i> , 7.2)	1.43 ( <i>d</i> , 7.2)
13	2.47 ( <i>s</i> )	2.08 ( <i>d</i> , 1.5)	2.07 ( <i>s</i> )	1.94 ( <i>s</i> )

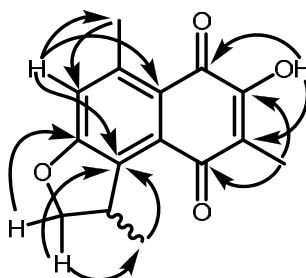
**Table 32** Comparison of  $^{13}\text{C}$  NMR spectral data of TP1-TP4

<b>Position</b>	<b>TP1 <math>\delta_{\text{C}}</math> (C-Type)</b>	<b>TP2 <math>\delta_{\text{C}}</math> (C-Type)</b>	<b>TP3 <math>\delta_{\text{C}}</math> (C-Type)</b>	<b>TP4 <math>\delta_{\text{C}}</math> (C-Type)</b>
1	130.1 (C)	143.0 (C)	134.5 (C)	149.4 (C)
2	126.2 (CH)	134.1 (CH)	119.9 (CH)	113.3 (CH)
3	119.1 (CH)	132.0 (CH)	162.2 (C)	165.3 (C)
4	142.2 (C)	132.5 (C)	133.2 (C)	131.1 (C)
4a	126.9 (C)	145.3 (C)	146.6 (C)	132.9 (C)
5	125.6 (CH)	138.0 (CH)	139.1 (CH)	137.4 (CH)
6	125.1 (C)	135.0 (C)	135.3 (C)	136.6 (C)
7	152.1 (C)	182.0 (C)	182.8 (C)	182.5 (C)
8	106.9 (CH)	182.8(C)	180.0 (C)	178.7 (C)
8a	133.1 (C)	129.3 (C)	122.7 (C)	122.4 (C)
9	19.5 (CH <sub>3</sub> )	22.8 (CH <sub>3</sub> )	23.3 (CH <sub>3</sub> )	23.6 (CH <sub>3</sub> )
10	28.4 (CH)	28.3 (CH)	26.8 (CH)	34.5 (CH)
11	23.7 (CH <sub>3</sub> )	23.7 (CH <sub>3</sub> )	21.2 (CH <sub>3</sub> )	80.0 (CH <sub>2</sub> )
12	23.7 (CH <sub>3</sub> )	23.7 (CH <sub>3</sub> )	21.2 (CH <sub>3</sub> )	21.9 (CH <sub>3</sub> )
13	16.8 (CH <sub>3</sub> )	16.0 (CH <sub>3</sub> )	15.9 (CH <sub>3</sub> )	15.7 (CH <sub>3</sub> )

### 2.3.2.5 Compound TP5



**TP5** was isolated as a yellow solid. IR spectrum (**Figure 57**) exhibited the characteristic absorption of carbonyl groups at 1642 and 1597  $\text{cm}^{-1}$  and hydroxyl group at 3328  $\text{cm}^{-1}$ . The UV spectrum (**Figure 56**) showed absorption maxima at 213, 274, 301 and 358 nm. The  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectral data (**Table 33**, **Figure 58** and **59**) of **TP5** were similar to those of **TP4** except for the absence of a proton at  $\delta$  7.11 (*s*, H-5) in the quinone ring of **TP4**, and the presence of the hydroxyl group at  $\delta$  7.75 whose showed HMBC correlations with carbonyl carbon at  $\delta$  180.6 (C-8),  $\delta$  117.7 (C-6) and  $\delta$  153.8 (C-7), indicating that hydroxyl group was placed at C-7. In addition, the correlation of methyl protons at  $\delta$  2.40 (Me-13) with the carbonyl carbon at  $\delta$  186.3 indicated the location of the second carbonyl carbon at C-5. These data established **TP5** to be *p*-naphthoquinone which was assigned to thespesone (Puckhaber *et al.*, 2004).



Selected HMBC correlations of **TP5**



**Table 33**  $^1\text{H}$ ,  $^{13}\text{C}$  NMR, DEPT and HMBC spectral data of **TP5**

position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	DEPT	HMBC
1	6.82 ( <i>s</i> )	146.0	C	3, 4, 9
2		116.2	CH	
3		165.7	C	
4		134.2	C	
4a		131.2	C	
5		186.3	C	
6		117.7	C	
7		153.8	C	
8		180.6	C	
8a	120.7	C	1, 2, 8a	
9	2.72 ( <i>s</i> )	23.9		CH <sub>3</sub>
10	4.14 ( <i>dquint</i> , 2.4, 6.9)	37.1		CH
11	4.41 ( <i>dd</i> , 4.4, 2.4)	80.5	CH <sub>2</sub>	3, 4, 10, 12
	4.62 ( <i>t</i> , 8.4)			
12	1.29 ( <i>d</i> , 6.9)	19.8	CH <sub>3</sub>	4, 10, 11
13	2.40 ( <i>s</i> )	8.4	CH <sub>3</sub>	5, 6, 7
7-OH	7.75 ( <i>s</i> )			6, 7, 8

**Table 34** Comparison of  $^{13}\text{C}$  NMR spectral data of **TP5** and thespesone

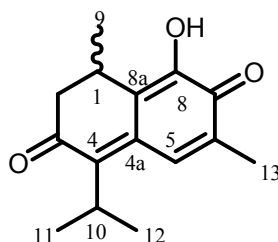
Position	TP5	thespesone <sup>a</sup>
	$\delta_{\text{C}}$	$\delta_{\text{C}}$
1	146.0	146.0
2	116.2	116.2
3	165.7	165.6
4	134.2	134.2
4a	131.2	131.1

**Table 34** (Continued)

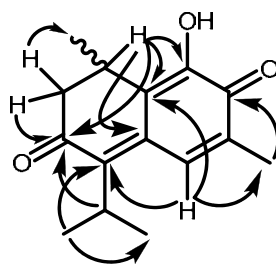
Position	TP5	thespesone <sup>a</sup>
	$\delta_c$	$\delta_c$
5	186.3	186.3
6	117.7	117.7
7	153.8	153.8
8	180.6	180.5
9	23.9	23.9
10	37.1	37.1
11	80.5	80.4
12	19.8	19.7
13	8.4	8.4

<sup>a</sup> recorded in CDCl<sub>3</sub>

### 2.3.2.6 Compound TP6



**TP6** was obtained as a yellow solid. The  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectral data (Table 35, Figures 62 and 63) of **TP6** were comparable to those of **TP3**. The differences were found as the presence of the methylene proton signals at  $\delta$  2.50 (*dd*, 14.7, 1.8 Hz) and 2.81 (*dd*, 14.7, 6.6 Hz);  $\delta_c$  46.1, an olefinic methine proton signal at  $\delta$  7.55 (*d*, 1.5 Hz);  $\delta_c$  132.5 and a methane proton at  $\delta$  3.57 (*dquint*, 1.8, 6.6 Hz);  $\delta_c$  28.1 in **TP6** instead of two aromatic proton signals in **TP3**. Besides the  $^1\text{H}$  NMR signal of Me-9 of **TP6** was shown as a *doublet* at  $\delta$  1.18 (*d*, 6.6 Hz) instead of a *singlet* signal at  $\delta$  2.58 as in **TP3**. In the HMBC experiment a methine proton at  $\delta$  3.57 showed correlations with  $\delta$  200.1 (C-3),  $\delta$  144.7 (C-8),  $\delta$  135.3 (C-4a) and  $\delta$  123.0 (C-8a). A methine proton of an isopropyl group at  $\delta$  3.42 also showed HMBC correlation with  $\delta$  200.1 (C-3), thus supporting a carbonyl carbon of C-3. By comparison of the spectral data of **TP6** with those of mansonone S (Tiew *et al.*, 2003), therefore **TP6** was identified as mansonane S.



Selected HMBC correlations of **TP6**

**Table 35**  $^1\text{H}$ ,  $^{13}\text{C}$  NMR, DEPT and HMBC spectral data of **TP6**

position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	DEPT	HMBC
1	3.57 ( <i>dquint</i> , 1.8, 6.6)	28.1	C	3, 4a, 8, 8a
2	2.50 ( <i>dd</i> , 14.7, 1.8) 2.81 ( <i>dd</i> , 14.7, 6.6)	46.1	CH <sub>2</sub>	1, 3, 4, 8a, 9
3		200.1	C	
4		150.2	C	
4a		135.3	C	
5	7.55 ( <i>d</i> , 1.5)	132.5	CH	4, 4a, 8a, 13
6		136.1	C	
7		180.8	C	
8		144.7	C	
8a		123.0	C	
9	1.18 ( <i>d</i> , 6.6)	20.5	CH <sub>3</sub>	2, 8a
10	3.42 ( <i>hept</i> , 6.9)	28.5	CH	3, 4a, 11, 12
11	1.28 ( <i>d</i> , 6.9)	21.0	CH <sub>3</sub>	4, 10, 12
12	1.38 ( <i>d</i> , 6.9)	22.8	CH <sub>3</sub>	4, 10, 11
13	2.07 ( <i>s</i> )	16.2	CH <sub>3</sub>	5, 6, 7

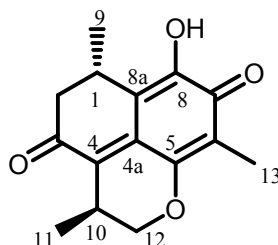
**Table 36** Comparison of  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectral data of **TP6** and mansonone S

position	TP6		mansonone S	
	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$
1	3.57 ( <i>dquint</i> , 1.8, 6.6)	28.1	3.55 ( <i>m</i> )	28.0
2	2.50 ( <i>dd</i> , 14.7, 1.8) 2.81 ( <i>dd</i> , 14.7, 6.6)	46.1	2.50 ( <i>d</i> , 15.0) 2.80 ( <i>dd</i> , 14.7, 6.4)	46.0
3		200.1		200.0
4		150.2		150.2
4a		135.3		135.3

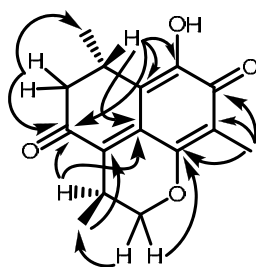
**Table 36** Comparison of  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectral data of **TP6** and mansonone S

position	TP6		mansonone S	
	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$
5	7.55 ( <i>d</i> , 1.5)	132.5	7.55 ( <i>s</i> )	132.4
6		136.1		136.1
7		180.8		180.8
8		144.7		144.6
8a		123.0		123.0
9	1.18 ( <i>d</i> , 6.6)	20.5	1.28 ( <i>d</i> , 7.0)	20.5
10	3.42 ( <i>hept</i> , 6.9)	28.5	3.45 ( <i>m</i> )	28.5
11	1.28 ( <i>d</i> , 6.9)	21.0	1.20 ( <i>d</i> , 7.3)	21.0
12	1.38 ( <i>d</i> , 6.9)	22.8	1.38 ( <i>d</i> , 7.0)	22.7
13	2.07 ( <i>s</i> )	16.2	2.13 ( <i>s</i> )	16.2

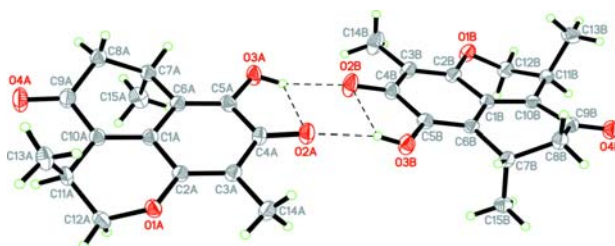
### 2.3.2.7 Compound TP7



**TP7** was isolated as reddish brown solid, which was recrystallized from MeOH- CH<sub>2</sub>Cl<sub>2</sub> (3:7 v/v). Comparison of the <sup>1</sup>H and <sup>13</sup>C NMR data (**Table 37**, **Figures 66** and **67**) suggested that **TP7** is closely related to **TP6**, except for the olefinic proton signal on the quinone ring at  $\delta$  7.55 (*d*, 1.5) and one of methyl proton signal of the isopropyl group at 1.38 (*d*, 6.9) were absent in **TP7**, being replaced instead by oxymethylene proton resonance at  $\delta$  4.09 (1H, *dd*, *J* = 10.8, 3.3 Hz) and 4.22 (1H, *d*, *J* = 10.8 Hz). <sup>3</sup>*J* HMBC correlations between oxymethylene protons (H-12) with C-5 ( $\delta$  157.1) established the fusion by ether linkage at C-5. X-ray structure of **TP7** established its stereochemistry. Therefore, **TP7** was identified as 7-hydroxy-2,3,5,6-tetrahydro-3,6,9-trimethyl-naphtho[1,8-b,c]pyran-4,8-dione (Milbrodt *et al.*, 1997).



Selected HMBC correlations of **TP7**



X-ray structure of **TP7**

**Table 37**  $^1\text{H}$ ,  $^{13}\text{C}$  NMR, DEPT and HMBC spectral data of **TP7**

position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	DEPT	HMBC
1	3.54 ( <i>dquint</i> , 1.8, 6.9)	27.6	CH	3, 4a, 8, 8a
2	2.71 ( <i>dd</i> , 16.2, 6.6) 2.53 ( <i>dd</i> , 16.2, 1.8)	44.6	CH <sub>2</sub>	1, 3, 4, 8a, 9
3		197.1	C	
4		139.5	C	
4a		131.0	C	
5		157.1	C	
6		115.1	C	
7		181.3	C	
8		143.5	C	
8a		115.2	C	
9	1.11 ( <i>d</i> , 6.9)	20.7	CH <sub>3</sub>	1, 2, 8a
10	3.05 ( <i>dq</i> , 3.3, 6.9)	26.5	CH	3, 4a
11	1.09 ( <i>d</i> , 6.9)	16.1	CH <sub>3</sub>	4, 10, 12
12	4.22 ( <i>d</i> , 10.8) 4.09 ( <i>dd</i> , 10.8, 3.3)	71.9	CH <sub>2</sub>	4, 10, 5
13	1.90 ( <i>s</i> )	8.0	CH <sub>3</sub>	5, 6, 7

**Table 38** Comparison of  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectral data of **TP7** and 7-hydroxy-2,3,5,6-tetrahydro-3,6,9-trimethyl-naphtho[1,8-b,c]pyran-4,8-dione (**R**)

position	TP7		R <sup>a</sup>	
	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$
1	3.54 ( <i>dquint</i> , 1.8, 6.9)	27.6	3.61 ( <i>dquint</i> , 1.5, 6.6, 7.1)	27.5
2	2.71 ( <i>dd</i> , 16.2, 6.6) 2.53 ( <i>dd</i> , 16.2, 1.8)	44.6	2.78 ( <i>dd</i> , 16.3, 6.6) 2.60 ( <i>dd</i> , 16.3, 1.5)	44.5
3		197.1		197.1

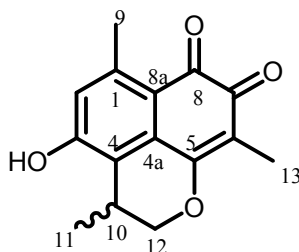
Table 38 (Continued)

position	TP7		R <sup>a</sup>	
	$\delta_{\text{H}}$ (mult., $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	$\delta_{\text{H}}$ (mult., $J_{\text{Hz}}$ )	$\delta_{\text{C}}$
4		139.5		139.4
4a		131.0		131.0
5		157.1		157.3
6		115.1		115.0
7		181.3		181.3
8		143.5		143.6
8a		115.2		115.1
9	1.11 ( <i>d</i> , 6.9)	20.7	1.19 ( <i>d</i> , 7.1)	20.6
10	3.05 ( <i>dq</i> , 3.3, 6.9)	26.5	3.12 ( <i>dq</i> , 3.5, 7.1)	26.4
11	1.09 ( <i>d</i> , 6.9)	16.1	1.16 ( <i>d</i> , 7.1)	16.2
12	4.22 ( <i>d</i> , 10.8)	71.9	4.28 ( <i>dd</i> , 10.5, 1.0)	71.9
	4.09 ( <i>dd</i> , 10.8, 3.3)		4.15 ( <i>dd</i> , 10.5, 3.5)	
13	1.90 ( <i>s</i> )	8.0	1.94 ( <i>s</i> )	8.0

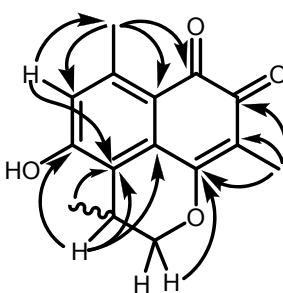
<sup>a</sup> recorded in CDCl<sub>3</sub>



### 2.3.2.8 Compound TP8



**TP8** was isolated as a reddish brown solid. The UV (**Figure 68**) and IR spectra (**Figure 69**) showed absorption bands similar to those of **TP3**. The  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra (**Table 39**, **Figures 70** and **71**) were comparable to those of **TP3** except that a proton H-5 ( $\delta$  7.72) on the quinone ring of **TP3** disappeared and the methyl signal Me-12 ( $\delta$  1.43 *d*, 7.2 Hz) was replaced by oxymethylene protons of **TP8** resonating at  $\delta$  4.41 (*d*,  $J$  = 10.8 Hz) and 4.29 (*dd*,  $J$  = 10.8, 3.3 Hz).  $^3J$  HMBC correlations between oxymethylene protons (H<sub>2</sub>-12) with C-5 ( $\delta$  162.4) of the main skeleton established their fusion by ether linkage at C-5. Therefore, **TP8** was identified as mansonone H (Kim *et al.*, 1996).



Selected HMBC correlations of **TP8**

**Table 39**  $^1\text{H}$ ,  $^{13}\text{C}$  NMR, DEPT and HMBC spectral data of **TP8**

position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	DEPT	HMBC		
1	6.74 ( <i>s</i> )	128.3	C	3, 4, 8, 9		
2		119.5	CH			
3		159.7	C			
4		125.4	C			
4a		128.3	C			
5		162.4	C			
6		115.5	C			
7		181.0	C			
8	2.59 ( <i>s</i> )	180.1	C	1, 2, 8, 8a		
8a		145.6	C			
9		23.0	CH <sub>3</sub>			
10		3.25 ( <i>dq</i> , 3.3, 6.9)	26.1		CH	
11		1.31 ( <i>d</i> , 6.9)	17.2		CH <sub>3</sub>	
12		4.41 ( <i>d</i> , 10.8)	72.0		CH <sub>2</sub>	
		4.29 ( <i>dd</i> , 10.8, 3.3)				
13		1.90 ( <i>s</i> )	7.9		CH <sub>3</sub>	5, 6, 7

**Table 40** Comparison of  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectral data of **TP8** and mansonone H

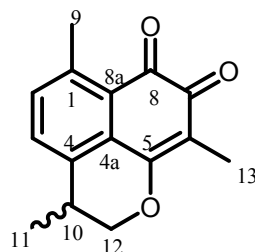
position	TP7		mansonone H <sup>a</sup>	
	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$
1	6.74 ( <i>s</i> )	128.3	6.33 ( <i>s</i> )	148.3
2		119.5		121.8
3		159.7		156.0
4		125.4		118.9
4a		128.3		129.4
5		162.4		165.6

Table 40 (Continued)

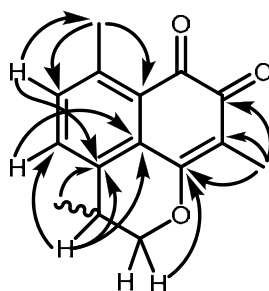
position	TP7		mansonone H <sup>a</sup>	
	$\delta_{\text{H}}$ (mult., J <sub>HZ</sub> )	$\delta_{\text{C}}$	$\delta_{\text{H}}$ (mult., J <sub>HZ</sub> )	$\delta_{\text{C}}$
6		115.5		114.8
7		181.0		180.2
8		180.1		183.2
8a		145.6		129.2
9	2.59 ( <i>s</i> )	23.0	2.48 ( <i>s</i> )	23.8
10	3.25 ( <i>dq</i> , 3.3, 6.9)	26.1	3.21 ( <i>m</i> )	27.5
11	1.31 ( <i>d</i> , 6.9)	17.2	1.24 ( <i>d</i> , 7.3)	17.4
12	4.41 ( <i>d</i> , 10.8)	72.0	4.40 ( <i>br d</i> , 10.3)	73.8
	4.29 ( <i>dd</i> , 10.8, 3.3)		4.28 ( <i>dd</i> , 10.3, 3.5)	
13	1.90 ( <i>s</i> )	7.9	1.85 ( <i>s</i> )	7.9

<sup>a</sup> recorded in CD<sub>3</sub>OD

### 2.3.2.9 Compound TP9



**TP9** was isolated a reddish brown solid. The UV (**Figure 72**) and IR spectra (**Figure 73**) showed absorption bands similar to those of **TP8**. The  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectral data of **TP9** (**Table 41**, **Figures 74** and **75**) and **TP8** (**Table**, **Figure**) showed structural similarity, except for the presence of an aromatic proton at  $\delta$  7.35 ( $d$ ,  $J = 8.1$  Hz);  $\delta_{\text{c}}$  132.6 in **TP9** instead of the hydroxyl group at C-3 ( $\delta_{\text{c}}$  159.7) in **TP8**. This proton was *ortho*-coupled with an aromatic proton H-2 at  $\delta$  7.26 ( $d$ ,  $J = 8.1$  Hz). Thus, **TP9** was assigned as mansonone E (Kim *et al.*, 1996).



Selected HMBC correlations of **TP9**

**Table 41**  $^1\text{H}$ ,  $^{13}\text{C}$  NMR, DEPT and HMBC spectral data of **TP9**

position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	DEPT	HMBC
1		127.4	C	
2	7.26 ( <i>d</i> , 8.1)	134.9	CH	1, 9
3	7.35 ( <i>d</i> , 8.1)	132.6	CH	4, 4a, 10
4		136.9	C	
4a		126.9	C	
5		162.5	C	
6		116.3	C	
7		180.2	C	
8		182.2	C	
8a		142.9	C	
9	2.65 ( <i>s</i> )	22.5	CH <sub>3</sub>	1, 2, 8a
10	3.09 ( <i>m</i> )	31.1	CH	3, 4, 4a
11	1.37 ( <i>d</i> , 7.2)	17.6	CH <sub>3</sub>	4, 10, 12
12	4.41 ( <i>dd</i> , 10.8, 3.9) 4.23 ( <i>dd</i> , 10.8, 5.1)	71.5	CH <sub>2</sub>	4, 5
13	1.96 ( <i>s</i> )	7.8	CH <sub>3</sub>	5, 6, 7

**Table 42** Comparison of  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectral data of **TP9** and mansonone E

position	TP9		mansonone E <sup>a</sup>	
	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$
1		127.4		142.8
2	7.26 ( <i>d</i> , 8.1)	134.9	7.25 ( <i>d</i> , 7.8)	134.9
3	7.35 ( <i>d</i> , 8.1)	132.6	7.35 ( <i>d</i> , 7.8)	132.6
4		136.9		136.9
4a		126.9		126.8
5		162.5		162.4

Table 42 (Continued)

position	TP9		mansonone E <sup>a</sup>	
	$\delta_{\text{H}}$ (mult., $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	$\delta_{\text{H}}$ (mult., $J_{\text{Hz}}$ )	$\delta_{\text{C}}$
6		116.3		116.8
7		180.2		180.2
8		182.2		182.2
8a		142.9		127.3
9	2.65 ( <i>s</i> )	22.5	2.63 ( <i>s</i> )	22.5
10	3.09 ( <i>m</i> )	31.1	3.10 ( <i>m</i> )	31.3
11	1.37 ( <i>d</i> , 7.2)	17.6	1.37 ( <i>d</i> , 6.8)	17.5
12	4.41 ( <i>dd</i> , 10.8, 3.9)	71.5	4.41 ( <i>dd</i> , 10.7, 3.9)	71.4
	4.23 ( <i>dd</i> , 10.8, 5.1)		4.23 ( <i>dd</i> , 10.7, 5.1)	
13	1.96 ( <i>s</i> )	7.8	1.94 ( <i>s</i> )	7.8

<sup>a</sup> recorded in CDCl<sub>3</sub>

**Table 43** Comparison of  $^1\text{H}$  NMR spectral data of TP5-TP9

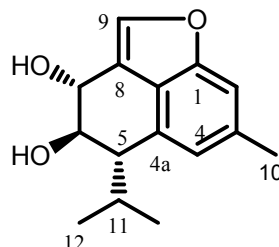
Position	TP5	TP6	TP7	TP8	TP9
	$\delta_{\text{H}}$ (mult., $J_{\text{Hz}}$ )	$\delta_{\text{H}}$ (mult., $J_{\text{Hz}}$ )	$\delta_{\text{H}}$ (mult., $J_{\text{Hz}}$ )	$\delta_{\text{H}}$ (mult., $J_{\text{Hz}}$ )	$\delta_{\text{H}}$ (mult., $J_{\text{Hz}}$ )
1		3.57 ( <i>dquint</i> , 1.8, 6.6)	3.54 ( <i>dquint</i> , 1.8, 6.9)		
2	6.82 ( <i>s</i> )	2.50 ( <i>dd</i> , 14.7, 1.8) 2.81 ( <i>dd</i> , 14.7, 6.6)	2.71 ( <i>dd</i> , 16.2, 6.6) 2.53 ( <i>dd</i> , 16.2, 1.8)	6.74 ( <i>s</i> )	7.26 ( <i>d</i> , 8.1)
3					7.35 ( <i>d</i> , 8.1)
4					
4a					
5		7.55 ( <i>d</i> , 1.5)			
6					
7					
8					
8a					
9	2.72 ( <i>s</i> )	1.18 ( <i>d</i> , 6.6)	1.11 ( <i>d</i> , 6.9)	2.59 ( <i>s</i> )	2.65 ( <i>s</i> )
10	4.14 ( <i>dquint</i> , 2.4, 6.9)	3.42 ( <i>hept</i> , 6.9)	3.05 ( <i>dq</i> , 3.3, 6.9)	3.25 ( <i>dq</i> , 3.3, 6.9)	3.09 ( <i>m</i> )
11	4.41 ( <i>dd</i> , 4.4, 2.4) 4.62 ( <i>t</i> , 8.4)	1.28 ( <i>d</i> , 6.9)	1.09 ( <i>d</i> , 6.9)	1.31 ( <i>d</i> , 6.9)	1.37 ( <i>d</i> , 7.2)
12	1.29 ( <i>d</i> , 6.9)	1.38 ( <i>d</i> , 6.9)	4.22 ( <i>d</i> , 10.8) 4.09 ( <i>dd</i> , 10.8, 3.3)	4.41 ( <i>d</i> , 10.8) 4.29 ( <i>dd</i> , 10.8, 3.3)	4.41 ( <i>dd</i> , 10.8, 3.9) 4.23 ( <i>dd</i> , 10.8, 5.1)
13	2.40 ( <i>s</i> )	2.07 ( <i>s</i> )	1.90 ( <i>s</i> )	1.90 ( <i>s</i> )	1.96 ( <i>s</i> )
7-OH	7.75 ( <i>s</i> )				

**Table 44** Comparison of  $^{13}\text{C}$  NMR spectral data of **TP5-TP9**

Position	TP5	TP6	TP7	TP8	TP9
	$\delta_{\text{c}}$	$\delta_{\text{c}}$	$\delta_{\text{c}}$	$\delta_{\text{c}}$	$\delta_{\text{c}}$
1	146.0	28.1	27.6	128.3	127.4
2	116.2	46.1	44.6	119.5	134.9
3	165.7	200.1	197.1	159.7	132.6
4	134.2	150.2	139.5	125.4	136.9
4a	131.2	135.3	131.0	128.3	126.9
5	186.3	132.5	157.1	162.4	162.5
6	117.7	136.1	115.1	115.5	116.3
7	153.8	180.8	181.3	181.0	180.2
8	180.6	144.7	143.5	180.1	182.2
8a	120.7	123.0	115.2	145.6	142.9
9	23.9	20.5	20.7	23.0	22.5
10	37.1	28.5	26.5	26.1	31.1
11	80.5	21.0	16.1	17.2	17.6
12	19.8	22.8	71.9	72.0	71.5
13	8.4	16.2	8.0	7.9	7.8

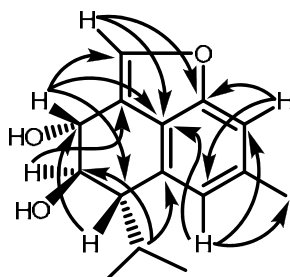


### 2.3.2.10 Compound TP10



**TP10** was obtained as a yellow gum with the molecular formula of  $C_{15}H_{18}O_3$  on the basis of molecular  $[M]^+$  at  $m/z$  246.1262 in the HREIMS (calc. 246.1256). The IR spectrum (**Figure 77**) of **TP10** showed the absorption band of hydroxyl at  $3365\text{ cm}^{-1}$ , while the UV spectrum (**Figure 76**) showed maximum absorptions at 216, 251, 259 (sh), 279 and 289, suggesting a benzofuran chromophore. The  $^1\text{H}$  NMR spectral data of **TP10** (**Table 45**, **Figure 78**) showed the characteristic of cadinane sesquiterpenoid skeleton with a benzofuran moiety. Two aromatic protons resonating at  $\delta$  7.02 (1H, *br s*) and 7.10 (1H, *br s*) were assigned to H-4 and H-2, respectively, whereas a furan proton appearing at  $\delta$  7.50 (*d*,  $J = 0.9$  Hz) was assigned to H-9. Moreover, one methine proton [ $\delta$  3.02 (*dd*,  $J = 7.8, 3.9$  Hz)], two oxymethines [ $\delta$  4.01 (*dd*,  $J = 7.8, 7.8$  Hz) and 4.90 (*dd*,  $J = 7.8, 0.9$  Hz)], one methyl group ( $\delta$  2.48, *s*) and one isopropyl moiety [ $\delta$  1.16 (*d*,  $J = 7.2$  Hz); 1.18 (*d*,  $J = 7.2$  Hz) and 2.58 (*dsept*,  $J = 7.2, 3.9$  Hz)] were also observed. The methyl group at  $\delta$  2.48 was placed at C-3 because of HMBC correlations to C-2 ( $\delta$  109.3) and C-4 ( $\delta$  121.6) and the isopropyl group was placed at C-5 due to HMBC correlations of its methine proton H-11 at  $\delta$  2.58 with C-4a ( $\delta$  131.4), C-5 ( $\delta$  49.8) and C-6 ( $\delta$  75.7). Finally, the two oxymethine protons at  $\delta$  4.01 and 4.90 were assigned to H-6 and H-7, respectively, judging from the allylic coupling (0.9 Hz) of H-9 with H-7 which was in turn coupled to oxymethine proton H-6 (4.01) in the COSY experiment. The relative stereochemistry at C-5, C-6 and C-7 was assigned by NOESY experiment, in which only the isopropyl group showed cross peak with H-6, indicating that H-6 was on the same side as the isopropyl group but opposite to H-5 and H-7. In addition, the pseudotrans-diaxial coupling (7.8 Hz) of H-6 with H-5 and H-7 also supported the NOESY experiment. Therefore, the relative stereostructure at H-5, H-6 and H-7

should be *trans–trans* configuration, **TP10** was a new compound and designated as populene A (Boonsri *et al.*, 2008).

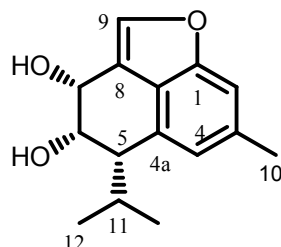


Selected HMBC correlations of **TP10**

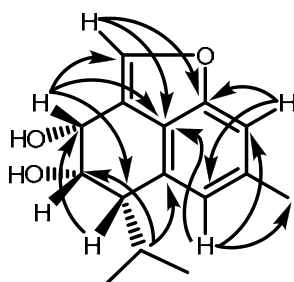
**Table 45**  $^1\text{H}$ ,  $^{13}\text{C}$  NMR, DEPT and HMBC spectral data of **TP10**

position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	DEPT	HMBC
1		153.5	C	
2	7.10 ( <i>br s</i> )	109.3	CH	1, 4, 10
3		135.8	C	
4	7.02 ( <i>br s</i> )	121.6	CH	2, 3, 8a, 10
4a		131.4	C	
5	3.02 ( <i>dd</i> , 7.8, 3.9, $\text{H}_{\beta}$ )	49.8	CH	6, 7
6	4.01 ( <i>dd</i> , 7.8, 7.8, $\text{H}_{\alpha}$ )	75.7	CH	4a, 5, 7, 8, 11
7	4.90 ( <i>dd</i> , 7.8, 0.9, $\text{H}_{\beta}$ )	70.5	CH	5, 6, 8, 8a, 9
8		118.7	C	
8a		123.7	C	
9	7.50 ( <i>d</i> , 0.9)	138.8	CH	1, 8, 8a
10	2.48 ( <i>s</i> )	22.4	$\text{CH}_3$	2, 3, 4
11	2.58 ( <i>m</i> )	27.8	CH	4a, 5, 6, 12, 13
12	1.16 ( <i>d</i> , 7.2)	20.0	$\text{CH}_3$	5, 11, 13
13	1.18 ( <i>d</i> , 7.2)	20.8	$\text{CH}_2$	5, 11, 12

### 2.3.2.11 Compound TP11



**TP11** was a yellow solid, and possessed the same formula as **TP10** by HREIMS ( $m/z$  246.1255  $[M]^+$ ,  $C_{15}H_{18}O_3$ ). The similarity of the mass, IR, UV,  $^1H$  and  $^{13}C$  NMR spectra (**Table 46**) of **TP10** and **TP11** indicated that **TP11** was a diastereomer of **TP10**. The difference was found in the small coupling constant of H-6 ( $\delta$  4.38, *dd*,  $J = 3.3, 3.3$  Hz) in **TP11** as compared to that in **TP10** ( $\delta$  4.01, *t*,  $J = 7.8$  Hz). Moreover, NOESY experiment exhibited cross peaks of H-5 and H-6 and between H-6 and H-7, suggesting their *cis* orientation. Accordingly, **TP11** was a new compound and designated as populene B (Boonsri *et al.*, 2008).

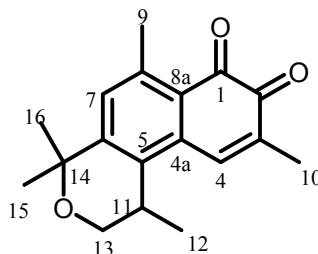


Selected HMBC correlations of **TP11**

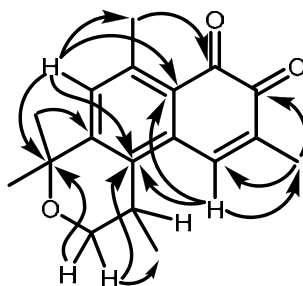
**Table 46**  $^1\text{H}$ ,  $^{13}\text{C}$  NMR, DEPT and HMBC spectral data of **TP11**

position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	DEPT	HMBC
1		153.6	C	
2	7.14 ( <i>br s</i> )	109.8	CH	1, 4, 8a, 10
3		135.7	C	
4	6.91 ( <i>br s</i> )	124.5	CH	2, 3, 8a, 10
4a		129.8	C	
5	2.90 ( <i>dd</i> , 8.7, 3.3, $\text{H}_{\beta}$ )	53.5	CH	4, 4a, 6, 7, 8a, 11, 13
6	4.38 ( <i>dd</i> , 3.3, 3.3, $\text{H}_{\beta}$ )	73.4	CH	4a, 8
7	5.08 ( <i>m</i> , $\text{H}_{\beta}$ )	65.6	CH	8, 9
8		118.2	C	
8a		123.4	C	
9	7.57 ( <i>d</i> , 1.5)	140.9	CH	1, 8
10	2.48 ( <i>s</i> )	22.2	$\text{CH}_3$	2, 3, 4
11	1.63 ( <i>m</i> )	31.0	CH	
12	1.12 ( <i>d</i> , 6.6)	21.3	$\text{CH}_3$	5, 11, 13
13	0.94 ( <i>d</i> , 6.6) <sup>a</sup>	21.6	$\text{CH}_2$	5, 11, 12

### 2.3.2.12 Compound TP12

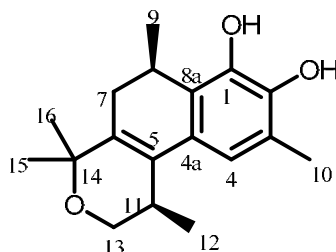


**TP12** was obtained as an orange solid whose molecular formula was determined as  $C_{18}H_{20}O_3$  by HREIMS ( $m/z$  286.1556  $[M+2]^+$ ). The EI mass spectrum was diagnostic, showing the relatively intense  $[M+2]^+$  characteristic ion peak of *ortho*-naphthoquinones which was not displayed by *para*-naphthoquinones (Letcher *et al.*, 1992). The IR spectrum (**Figure 85**) exhibited the characteristic absorption of carbonyl groups at 1757 and 1698  $cm^{-1}$ . The UV spectrum (**Figure 84**) showed absorption maxima at 213, 242, 259 and 380 nm. The  $^1H$  and  $^{13}C$  NMR spectral data (**Table 47**, **Figures 86** and **87**) of **TP12** were comparable to those of mansonone D (**TP4**), which was isolated from the dark heartwood of this plant. The differences between these two compounds were found as the additional isopropyl group, which appeared as two methyl singlet signals at  $\delta$  1.57 and 1.53 in the  $^1H$  NMR spectrum of **TP12**, whose HMBC correlations to oxygenated quaternary carbon at  $\delta$  74.9 (C-14) supported the connection of this group to oxygen. In addition, the correlation of oxymethylene protons at  $\delta$  3.97 and 3.79 (H<sub>2</sub>-13) with C-5 ( $\delta$  135.8) and C-14 ( $\delta$  74.9), of gem-dimethyl with C-6 ( $\delta$  150.1) and of an aromatic proton H-7 ( $\delta$  6.95) with C-14 ( $\delta$  74.9), indicated that a pyran moiety was connected to an aromatic ring at C-5 and C-6. The methine proton on C-11 was deduced to be equatorially oriented from the two small vicinal coupling constants ( $J_{11,13\beta} = 1.2$  Hz and  $J_{11,13\alpha} = 2.4$  Hz). The relative stereostructure of the trimethylpyran ring was postulated from NOESY cross-peaks of a methylene proton H-13 $\beta$  ( $\delta$  3.79) with a methyl group at  $\delta$  1.40 (Me-12) and of H-13 $\alpha$  ( $\delta$  3.97) with a methyl group at  $\delta$  1.53 (Me-15). Therefore, **TP12** was identified as a new compound and designated as populene C (Boonsri *et al.*, 2008).

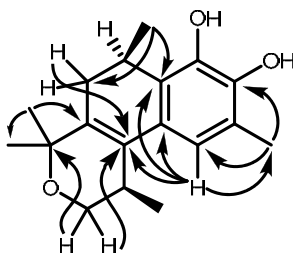
Selected HMBC correlations of **TP12****Table 47**  $^1\text{H}$ ,  $^{13}\text{C}$  NMR, DEPT and HMBC spectral data of **TP12**

position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	DEPT	HMBC
1		181.7	C	
2		181.6	C	
3		135.8	C	
4	7.52 ( <i>d</i> , 1.2)	137.3	CH	2, 4a, 5, 10
4a		128.4	C	
5		135.8	C	
6		150.1	C	
7	6.95 ( <i>s</i> )	131.2	CH	5, 8a, 9, 14
8		142.6	C	
8a		133.1	C	
9	2.62 ( <i>s</i> )	23.0	CH <sub>3</sub>	7, 8, 8a
10	2.09 ( <i>d</i> , 1.2)	16.0	CH <sub>3</sub>	2, 3, 4
11	3.01 ( <i>br q</i> , 6.9, $\text{H}_{\alpha}$ )	29.9	CH	
12	1.40 ( <i>d</i> , 6.9)	21.2	CH <sub>3</sub>	5, 13
13	3.97 ( <i>dd</i> , 11.7, 2.4, $\text{H}_{\alpha}$ ) 3.79 ( <i>dd</i> , 11.7, 1.2, $\text{H}_{\beta}$ )	64.9	CH <sub>2</sub>	5, 11, 12, 14
14		74.9	C	
15	1.53 ( <i>s</i> )	31.3	CH <sub>3</sub>	6, 14, 16
16	1.57 ( <i>s</i> )	27.8	CH <sub>3</sub>	6, 14, 15

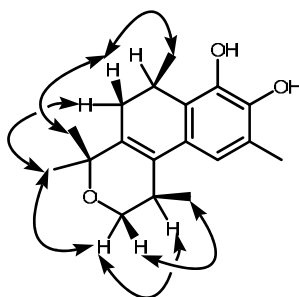
### 2.3.2.13 Compound TP13



**TP13** was a brown gum and its molecular formula was deduced as  $C_{18}H_{24}O_3$  from the HREIMS ( $m/z$  288.1736,  $[M]^+$ ). The IR spectrum (**Figure 89**) exhibited OH absorption at  $3417\text{ cm}^{-1}$ . The structural assignment was initiated by comparison of the NMR spectra of **TP13** with those of **TP12**. In the  $^1\text{H}$  NMR spectrum (**Table 48, Figure 90**), an aromatic proton signal at  $\delta$  6.95 and an aromatic methyl at  $\delta$  2.62 as found in **TP12** were missing in **TP13** and the signals of  $-\text{CH}(\text{CH}_3)\text{CH}_2-$  were instead observed at  $\delta$  1.04 (3H, *d*,  $J = 6.9$  Hz, H-9), 3.19 (1H, *br quint*,  $J = 6.9$  Hz, H-8), 2.00 (1H, *d*,  $J = 15.3$  Hz, H-7) and 2.36 (1H, *dd*,  $J = 15.3, 5.1$  Hz, H-7). This assignment was confirmed by COSY cross-peaks and HMBC correlations of H<sub>2</sub>-7 to C-5 ( $\delta$  128.7), C-6 ( $\delta$  132.2) and C-9 ( $\delta$  17.9) and of H<sub>3</sub>-9 to C-7 ( $\delta$  31.0) and C-8a ( $\delta$  125.2). In addition, the replacement of two carbonyl carbons of the quinone ring at  $\delta$  181.7 (C-1) and 181.6 (C-2) ppm in **TP12** with oxygenated aromatic carbons at  $\delta$  140.3 and  $\delta$  140.9 ppm in **TP13** indicated that **TP13** was a reduced form of **TP12**. The relative stereochemistry of H-8 and H-11 were elucidated by NOESY spectrum as shown in **Figure 3**, which indicated that Me-9 and Me-12 were on the same side of the molecule. Therefore, **TP13** was identified as a new compound and designated as populene D (Boonsri *et al.*, 2008).



Selected HMBC correlations of **TP13**



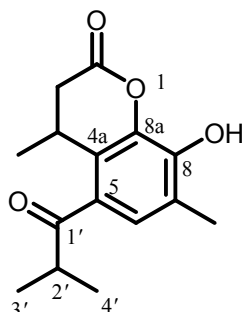
**Figure 3** Populene D with selected NOESY correlations.

**Table 48**  $^1\text{H}$ ,  $^{13}\text{C}$  NMR, DEPT and HMBC spectral data of **TP13**

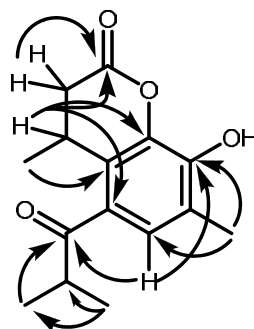
position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	DEPT	HMBC
1		140.3	C	
2		140.9	C	
3		121.0	C	
4	6.65 ( <i>s</i> )	117.0	CH	4a, 5, 10
4a		125.0	C	
5		128.7	C	
6		132.2	C	
7	2.00 ( <i>d</i> , 15.3, $\text{H}_{\beta}$ ) 2.36 ( <i>dd</i> , 15.3, 5.1, $\text{H}_{\alpha}$ )	31.0	$\text{CH}_2$	5, 6, 9
8	3.19 ( <i>br quint</i> , 6.9, $\text{H}_{\alpha}$ )	25.2	CH	
8a		125.2	C	
9	1.04 ( <i>d</i> , 6.9)	17.9	$\text{CH}_3$	7, 8, 8a
10	2.25 ( <i>s</i> )	15.8	$\text{CH}_3$	2, 3, 4
11	2.68 ( <i>m</i> , $\text{H}_{\alpha}$ )	28.4	CH	
12	1.14 ( <i>d</i> , 6.9)	17.6	$\text{CH}_3$	5, 11, 13
13	3.90 ( <i>dd</i> , 11.1, 3.0, $\text{H}_{\alpha}$ ) 3.66 ( <i>dd</i> , 11.1, 2.4, $\text{H}_{\beta}$ )	65.7	$\text{CH}_2$	5, 11, 12, 14
14		75.0	C	
15	1.26 ( <i>s</i> )	23.6	$\text{CH}_3$	6, 14, 16
16	1.41 ( <i>s</i> )	27.6	$\text{CH}_3$	6, 14, 15



### 2.3.2.14 Compound TP14



**TP14** was obtained as a yellow-brown gum. The molecular formula was established as  $C_{15}H_{18}O_4$  on the basis of HREIMS ( $m/z$  262.1210,  $[M]^+$ ). The  $^{13}C$  NMR (**Table 49**) showed the presence of 15 resonances, which corresponded by DEPT analysis to three methines (one  $sp^2$ ), one methylene, four methyls and seven  $sp^2$  quaternary carbons including two carbonyl carbons ( $\delta_C$  167.4 and 205.8). The  $^1H$  NMR (**Table 49**, **Figure 94**) and COSY spectra allowed assignment of signals of a dihydrocoumarin moiety at  $\delta$  1.31 (3H, *d*,  $J = 6.9$  Hz, 4-Me), 2.72 (2H, *d*,  $J = 3.6$  Hz, H<sub>2</sub>-3), 3.88 (1H, *tq*,  $J = 3.6, 6.9$  Hz, H-4), and 7.40 (1H, *s*, H-6). This moiety was also supported by the  $^3J$  HMBC correlations between the methine proton H-4 and aromatic carbons C-5 ( $\delta$  126.2), C-8a ( $\delta$  139.2) and a lactone carbonyl ( $\delta$  167.4). Moreover, the signals of 2-methyl-1-oxopropyl unit [ $\delta$  3.47 (1H, *sept*,  $J = 6.9$ , H-2'), 1.21 (3H, *d*,  $J = 6.9$  Hz, H-3') and 1.14, (3H, *d*,  $J = 6.9$ , H-4')] were also observed in the  $^1H$  NMR spectrum whose HMBC correlation between an aromatic proton H-6 ( $\delta$  7.40) and C-1' ( $\delta$  205.8) supported its connection at C-5 of the dihydrocoumarin moiety. An aromatic methyl at  $\delta$  2.30 was attributed to 7-Me due to its HMBC correlation with C-6 ( $\delta$  127.8), C-7 ( $\delta$  123.5) and C-8 ( $\delta$  145.4). Additionally, a downfield carbon chemical shift of C-8 at  $\delta$  145.4 indicated its connection to a hydroxyl group. Thus, the structure of **TP14** was elucidated to be a new compound and designated as populene E (Boonsri *et al.*, 2008).

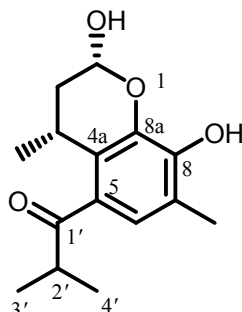


Selected HMBC correlations of TP14

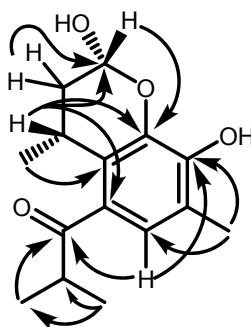
**Table 49**  $^1\text{H}$ ,  $^{13}\text{C}$  NMR, DEPT and HMBC spectral data of TP14

position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	DEPT	HMBC
2		167.4	C	
3	2.72 ( <i>d</i> , 3.6)	36.3	CH <sub>2</sub>	2
4	3.88 ( <i>tq</i> , 3.6, 6.9)	27.5	CH	2, 5, 8a
4a		127.9	C	
5		126.2	C	
6	7.40 ( <i>s</i> )	127.8	CH	4, 5, 8, 8a, 7-Me, 1'
7		123.5	C	
8		145.4	C	
8a		139.2	C	
1'		205.8	C	
2'	3.47 ( <i>sept</i> , 6.9)	37.2	CH	1', 3', 4'
3'	1.21 ( <i>d</i> , 6.9)	19.0	CH <sub>3</sub>	1', 3', 4'
4'	1.14 ( <i>d</i> , 6.9)	19.4	CH <sub>3</sub>	2', 3'
4-Me	1.31 ( <i>d</i> , 6.9)	20.2	CH <sub>3</sub>	3, 4, 4a
7-Me	2.30 ( <i>s</i> )	15.5	CH <sub>3</sub>	6, 7, 8

### 2.3.2.15 Compound TP15



**TP15** was obtained as a yellow gum. The molecular formula was established as  $C_{15}H_{20}O_4$  on the basis of HREIMS ( $m/z$ , 264.1353  $[M]^+$ ). The UV (Figure 96) and IR (Figure 97) spectra were similar to those of **TP14**, but with one carbonyl absorption at  $1668\text{ cm}^{-1}$ . The NMR (Table 50, Figures 98 and 99) data were comparable to those of **TP14**, except for the replacement of a lactone carbonyl ( $\delta$  167.4) in **TP14** with a hemiacetal proton signal of H-2 at  $\delta_H$  5.65 ( $dd$ ,  $J = 9.0, 3.0\text{ Hz}$ ;  $\delta_C$  92.6) in **TP15**. The large coupling constant (13.5 Hz) was the characteristic geminal coupling of the methylene protons; H-3 $\beta$  (2.07,  $td$ ,  $J = 3.0, 13.5\text{ Hz}$ ) and H-3 $\alpha$  (1.87,  $ddd$ ,  $J = 13.5, 9.0, 5.1\text{ Hz}$ ), while the vicinal coupling constant of 9.0 and 5.1 Hz were the pseudotrans-diaxial coupling of H-3 $\alpha$  with H-2 and H-4, respectively. This was also in agreement with the multiplicity of H-3 $\beta$  observed as a triplet of doublet with a large ( $J_{\text{gem}} = 13.5\text{ Hz}$ ) and a small ( $J_{\text{ax-eq}} = 3.0\text{ Hz}$ ) coupling constants, justifying its *syn* relationship to H-2 and H-4. **TP15** was thus identified as a new compound and designated as populene F (Boonsri *et al.*, 2008).

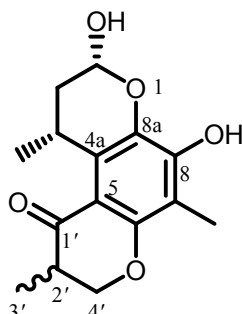


Selected HMBC correlations of **TP15**

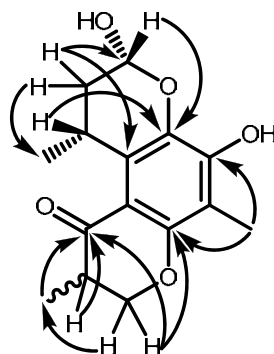
**Table 50**  $^1\text{H}$ ,  $^{13}\text{C}$  NMR, DEPT and HMBC spectral data of **TP15**

position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	DEPT	HMBC
2	5.65 ( <i>dd</i> , 9.0, 3.0, $\text{H}_{\beta}$ )	92.6	CH	3, 4, 8a
3	1.87 ( <i>ddd</i> , 13.5, 9.0, 5.1, $\text{H}_{\alpha}$ ) 2.07 ( <i>td</i> , 3.0, 13.5, $\text{H}_{\beta}$ )	36.6	$\text{CH}_2$	2, 4, 4a, 4-Me
4	3.84 ( <i>m</i> , $\text{H}_{\beta}$ )	26.2	CH	
4a		126.3	C	
5		127.1	C	
6	7.18 ( <i>s</i> )	125.0	CH	4a, 8, 8a, 7-Me, 1'
7		121.0	C	
8		146.2	C	
8a		140.0	C	
1'		207.1	C	
2'	3.45 ( <i>sept</i> , 6.9)	37.4	CH	1', 2', 3', 4'
3'	1.17 ( <i>d</i> , 6.9)	19.1	$\text{CH}_3$	1', 2', 4'
4'	1.15 ( <i>d</i> , 6.9)	19.6	$\text{CH}_3$	1', 2', 3'
4-Me	1.25 ( <i>d</i> , 6.9)	22.3	$\text{CH}_3$	3, 4, 4a
7-Me	2.23 ( <i>s</i> )	15.3	$\text{CH}_3$	6, 7, 8

### 2.3.2.16 Compound TP16



**TP16** was obtained as a yellow gum. The molecular formula of **TP16** was established as  $C_{15}H_{18}O_5$  as determined by HREIMS ( $m/z$  278.1196,  $[M]^+$ ). The  $^1H$  and  $^{13}C$  NMR spectra (**Table 51**, **Figures 102** and **103**) were similar to those of **TP15** except that in **TP16** an aromatic proton H-6 at  $\delta$  7.18 in **TP15** disappeared and a methyl signal Me-4' was replaced by oxymethylene protons resonating at  $\delta$  4.43 (1H, *dd*,  $J = 11.1, 5.1$  Hz, H-4') and 4.03 (1H, *dd*,  $J = 11.1, 11.1$  Hz, H-4') in **TP16**. The  $^3J$  HMBC correlation between oxymethylene protons (H<sub>2</sub>-4') with C-6 ( $\delta$  157.7) of an aromatic moiety established their fusion by an ether linkage at C-6. The stereochemistry of H-2' was deduced to be equatorially oriented from the small coupling constant ( $J_{2',4'_{ax}} = 5.1$  Hz). Thus, **TP16** was concluded to be a new compound and designated as populene G (Boonsri *et al.*, 2008).

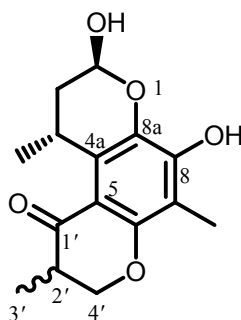


Selected HMBC correlations of **TP16**

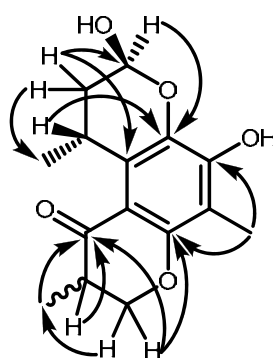
**Table 51**  $^1\text{H}$ ,  $^{13}\text{C}$  NMR, DEPT and HMBC spectral data of **TP16**

position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	DEPT	HMBC
2	5.56 ( <i>dd</i> , 9.9, 2.7, $\text{H}_{\beta}$ )	92.2	CH	3, 8a
3	1.84 ( <i>ddd</i> , 13.5, 9.9, 5.4, $\text{H}_{\alpha}$ ) 2.04 ( <i>td</i> , 2.7, 13.5, $\text{H}_{\beta}$ )	36.8	$\text{CH}_2$	2, 4, 4a, 4-Me
4	4.09 ( <i>m</i> , $\text{H}_{\beta}$ )	27.2	CH	2, 4a, 8a, 4-Me
4a		125.1	C	
5		109.4	C	
6		157.7	C	
7		110.4	C	
8		149.3	C	
8a		134.6	C	
1'		195.3	C	
2'	2.75 ( <i>m</i> )	41.2	CH	1', 3', 4'
3'	1.16 ( <i>d</i> , 6.9)	11.0	$\text{CH}_3$	1', 2', 4'
4'	4.03 ( <i>dd</i> , 11.1, 11.1) 4.43 ( <i>dd</i> , 11.1, 5.1)	71.6	$\text{CH}_2$	1', 2', 3', 6
4-Me	1.28 ( <i>d</i> , 6.9)	22.4	$\text{CH}_3$	3, 4, 4a
7-Me	2.09 ( <i>s</i> )	8.1	$\text{CH}_3$	6, 7, 8

### 2.3.2.17 Compound TP17



**TP17**, isolated as a yellow gum, had the molecular formula  $C_{15}H_{18}O_5$  as determined by HREIMS ( $m/z$  278.1159,  $[M]^+$ ). The similar mass and NMR spectra of **TP16** (Table 51, Figure 102 and 103) and **TP17** (Table 52, Figures 106 and 107) indicated diastereomers. The main spectroscopic differences were the downfield shift of H-2 in **TP17** at  $\delta$  5.81 and the smaller coupling constants ( $dd$ ,  $J = 7.5, 4.5$  Hz) as compared to those of **TP16** at  $\delta$  5.56 ( $dd$ ,  $J = 9.9, 2.7$  Hz). The coupling constant  $J_{2,3}$  of 7.5 and 4.5 Hz indicated  $J_{eq-ax}$  and  $J_{eq-eq}$ , therefore suggesting  $\alpha$ -orientation of H-2. Accordingly, **TP17** was elucidated to be a new compound and designated as populene H (Boonsri *et al.*, 2008).



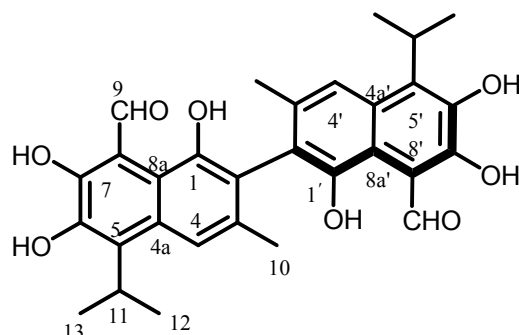
Selected HMBC correlations of **TP17**

**Table 52**  $^1\text{H}$ ,  $^{13}\text{C}$  NMR, DEPT and HMBC spectral data of **TP17**

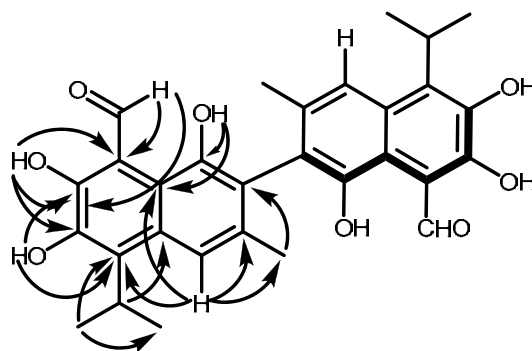
position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	DEPT	HMBC
2	5.81 ( <i>dd</i> , 7.5, 4,5, $\text{H}_\alpha$ )	95.9	CH	8a
3	2.00 ( <i>m</i> )	36.1	$\text{CH}_2$	2, 4, 4a, 4-Me
4	4.10 ( <i>m</i> )	27.1	CH	4-Me
4a		127.2	C	
5		111.4	C	
6		158.7	C	
7		110.6	C	
8		149.9	C	
8a		134.9	C	
1'		196.1	C	
2'	2.75 ( <i>m</i> )	42.1	CH	1', 3', 4'
3'	1.17 ( <i>d</i> , 6.5)	11.8	$\text{CH}_3$	1', 2', 4'
4'	4.05 ( <i>dd</i> , 11.5, 11.5)	72.6	$\text{CH}_2$	1', 2', 3', 6
	4.45 ( <i>dd</i> , 11.5, 5.5)			
4-Me	1.32 ( <i>d</i> , 7.0)	23.0	$\text{CH}_3$	3, 4a
7-Me	2.11 ( <i>s</i> )	9.0	$\text{CH}_3$	6, 7, 8



### 2.3.2.18 Compound TP18



**TP18** was isolated as a yellow solid. The UV spectrum exhibited the absorption bands at 237, 276, 290 and 379 nm. The IR spectrum indicated the presence of hydroxyl functionality ( $3410\text{ cm}^{-1}$ ). The  $^1\text{H}$  NMR spectrum of **TP18** (Table 54, Figures 110 and 111), the low field chemical shift of the aldehyde proton at  $\delta$  10.98 (*s*, H-9) indicated chelation to an *ortho* hydroxyl proton which appeared at  $\delta$  14.50 (*s*, 7-OH). Two hydroxyl groups appearing at  $\delta$  6.19 and 6.81 were located at C-6 and C-1, respectively. An aromatic proton resonating at  $\delta$  7.71 (*s*) was assigned to H-4. Signals of a methyl group at  $\delta$  2.13 (*s*) and an isopropyl moiety [ $\delta$  1.48 (*d*,  $J = 7.2\text{ Hz}$ , 6H) and 3.82 (*m*)] were also observed. The methyl group at  $\delta$  2.13 was placed at C-3 because of HMBC correlations to C-2 ( $\delta$  116.7) and C-3 ( $\delta$  134.0) and the isopropyl group was placed at C-5 due to HMBC correlations of its methine proton H-10 at  $\delta$  3.82 with C-4a ( $\delta$  129.5), C-5 ( $\delta$  134.4) and C-6 ( $\delta$  143.0). Since the  $^{13}\text{C}$  NMR spectrum exhibited only 15 signals and its  $^1\text{H}$  NMR spectrum also showed signals corresponding to a monomer. **TP18** was inferred to be a symmetrical dimer. A quaternary  $\text{sp}^2$  carbon resonating at  $\delta$  116.7 in the  $^{13}\text{C}$  NMR was assigned to C-2. Thus this compound was deduced to be a symmetrical dimer which connected at C-2-C-2'. Therefore, **TP18** was identified as (+)-gossypol (Meyers *et al.*, 1998).

Selected HMBC correlations of **TP18****Table 53**  $^1\text{H}$ ,  $^{13}\text{C}$  NMR, DEPT and HMBC spectral data of **TP18**

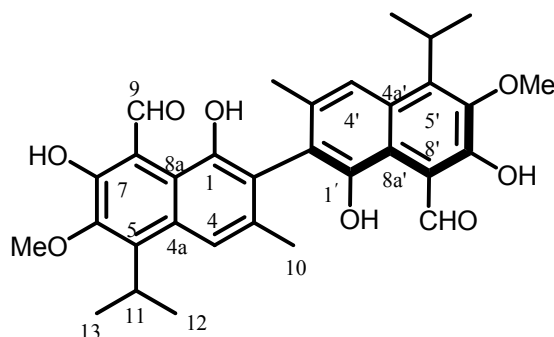
position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	DEPT	HMBC
1		150.7	C	
2		116.7	C	
3		134.0	C	
4	7.71 ( <i>s</i> )	117.8	CH	1, 3, 5, 8a
4a		129.5	C	
5		134.4	C	
6		143.0	C	
7		155.7	C	
8		111.6	C	
8a		114.8	C	
9	10.98 ( <i>s</i> )	199.1	CH	6, 7, 8
10	2.13 ( <i>s</i> )	20.3	$\text{CH}_3$	2, 3
11	3.82 ( <i>m</i> )	27.9	CH	4a, 5, 6, 12, 13
12	1.48 ( <i>d</i> , 7.2)	20.2	$\text{CH}_3$	5, 11, 13
13	1.48 ( <i>d</i> , 7.2)	20.2	$\text{CH}_3$	5, 11, 12
1-OH	6.81 ( <i>s</i> )			1, 2, 8a
6-OH	6.19 ( <i>s</i> )			5, 6, 7
7-OH	14.50 ( <i>s</i> )			6, 7, 8

**Table 54** Comparison of  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectral data of **TP18** and gossypol<sup>a</sup>

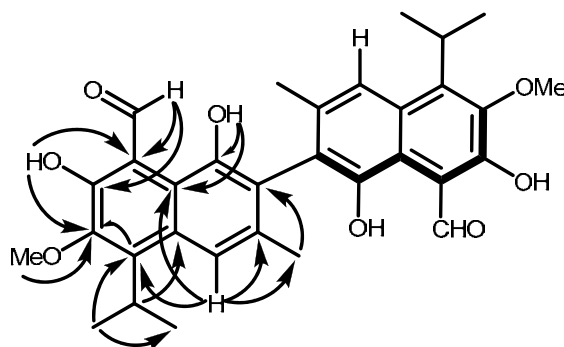
position	TP9		gossypol <sup>a</sup>	
	$\delta_{\text{H}}$ (mult., $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	$\delta_{\text{H}}$ (mult., $J_{\text{Hz}}$ )	$\delta_{\text{C}}$
1		150.7		150.8
2		116.7		116.5
3		134.0		134.0
4	7.71 (s)	117.8	7.77 (s)	118.2
4a		129.5		129.8
5		134.4		134.4
6		143.0		143.4
7		155.7		156.0
8		111.6		111.9
8a		114.8		114.9
9	10.98 (s)	199.1	11.11 (s)	199.5
10	2.13 (s)	20.3	2.14 (s)	20.5
11	3.82 (m)	27.9	3.88 (septet, 6.9)	28.1
12	1.48 (d, 7.2)	20.2	1.54 (d, 7.0)	20.5
13	1.48 (d, 7.2)	20.2	1.54 (d, 7.0)	20.5
1-OH	6.81 (s)		6.39 (s)	
6-OH	6.19 (s)		5.85 (s)	
7-OH	14.50 (s)		15.11 (s)	

<sup>a</sup> recorded in  $\text{CDCl}_3$

### 2.3.2.19 Compound TP19



**TP19** was obtained as a yellow solid. The  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectral data (Table 55, Figures 114 and 115) of **TP19** were similar to those of **TP18** except for the replacement of a hydroxyl proton at  $\delta$  6.19 (*s*) in **TP18** with the methoxyl group at  $\delta$  4.00 whose HMBC correlation with the quaternary carbon at  $\delta$  147.7 (C-6), indicated that the methoxyl group was attached to C-6. Thus, the structure of **TP19** was concluded to be (+)-6, 6'-dimethoxygossypol.



Selected HMBC correlations of **TP19**

**Table 55**  $^1\text{H}$ ,  $^{13}\text{C}$  NMR, DEPT and HMBC spectral data of **TP19**

position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	DEPT	HMBC
1		150.1	C	
2		117.3	C	
3		133.1	C	
4	7.83 ( <i>s</i> )	119.2	CH	3, 4a, 5, 8a
4a		129.4	C	
5		144.5	C	
6		147.7	C	
7		161.1	C	
8		113.3	C	
8a		116.9	C	
9	11.15 ( <i>s</i> )	199.2	CH	6, 7, 8
10	2.16 ( <i>s</i> )	20.3	CH <sub>3</sub>	2, 3, 4
11	4.00 ( <i>m</i> )	27.9	CH	
12	1.56 ( <i>d</i> , 6.9)	21.7	CH <sub>3</sub>	5, 11, 13
13	1.55 ( <i>d</i> , 6.9)	21.7	CH <sub>3</sub>	5, 11, 12
1-OH	6.81 ( <i>s</i> )			1, 4a, 8a, 9
6-OMe	4.00 ( <i>s</i> )		CH <sub>3</sub>	6
7-OH	14.56 ( <i>s</i> )			6, 7, 8

### 2.3.2 Biological activities of the isolated compounds from the roots of *T. populnea*

All of the isolated compounds except for **TP2**, **TP3**, **TP10**, **TP11**, **TP14** and **TP17** for which insufficient materials were available, were evaluated for cytotoxicity against four human cancer cell lines; breast cancer (MCF-7), cervical cancer (HeLa), colon cancer (HT-29) and oral cavity cancer (KB). They were also tested for antibacterial activity against both Gram-positive (*Bacillus subtilis* and *Staphylococcus aureus*) and Gram-negative (*Enterococcus faecalis*, *Salmonella typhi*, *Shigella sonnei* and *Pseudomonas aeruginosa*). The results are summarized in **Table 56**. (+)-Gossypol (**TP18**) exhibited potent cytotoxic activity against HeLa and KB cell lines, with  $IC_{50}$  values 0.08 and 0.04  $\mu\text{g/mL}$ , respectively. Mansonone E (**TP9**) showed good activity against all four cancer cell lines, especially MCF-7 ( $IC_{50}$  0.05  $\mu\text{g/mL}$ ). Populene D (**TP13**) and mansonone D (**TP4**) possessed strong inhibitory activity against HeLa and MCF-7, respectively, whereas populene C (**TP12**) exhibited moderate inhibitory activity against all four cell lines. Antibacterial activity against *B.subtilis* was found for 7-hydroxycadalene (**TP1**). (+)-6,6'-methoxygossypol (**TP19**) was weakly active against *E. faecalis*, *B.subtilis* and *S. aureus*, whereas (+)-gossypol (**TP18**) exhibited moderate activity against *B.subtilis* and *S. aureus*. None of the compounds were active against *S. typhi*, *S. sonnei* or *P. aeruginosa*. Compounds **TP5**, **TP8**, **TP15** and **TP16** showed no cytotoxic or antibacterial activity.

**Table 56** Cytotoxic and antibacterial activities of compounds isolated from *T. populnea*

Compounds	Cytotoxicity against human cancer cell lines, IC <sub>50</sub> (μg/mL)				Antibacterial activity, MIC (μg/mL)		
	MCF-7	HeLa	HT-29	KB	<i>B. subtilis</i>	<i>S. aureus</i>	<i>E. faecalis</i>
<b>TP1</b>	>5	>5	>5	>5	0.59	-	-
<b>TP4</b>	0.80	2.80	>5	4.90	2.34	-	-
<b>TP6</b>	>5	>5	>5	>5	- <sup>b</sup>	-	-
<b>TP7</b>	>5	>5	>5	>5	-	-	-
<b>TP9</b>	0.05	0.55	0.18	0.40	4.69	-	-
<b>TP12</b>	2.35	3.40	2.90	3.00	4.69	-	-
<b>TP13</b>	1.85	0.95	2.37	3.10	4.69	-	-
<b>TP18</b>	NT <sup>a</sup>	0.08	>5	0.04	1.17	1.17	-
<b>TP19</b>	4.00	>5	3.00	>5	2.34	4.69	1.17

<sup>a</sup> NT = not tested. <sup>b</sup> = inactive (> 10 μg/mL)

## CHAPTER 3.1

### INTRODUCTION

#### 3.1.1 Introduction

*Artocarpus integer* (Thunb.) Merr. is a plant belonging to the family Moraceae. This family is distributed in the tropical and subtropical regions of Asia, comprises some 1400 species divided among 60 genera (Hakim *et al.*, 2005). In Thailand only 8 genera are found, from *Artocarpus* genus only 14 species are found (Smitinand 2001).

*A. integer* is a large tree with dense crown, reaching a height of 15 m or more; the cylindrical stem is rounded at the ends; bark grey-brown to dark brown with warty excrescences; blaze pale pink to yellow, exuding a copious milky latex when cut. Leaves obovate to elliptic, 5-25 cm long and 2.5-12 cm wide, with cuneate to round base; margin entire; pointed tip and 6-10 pairs of lateral veins curving forward; leafstalk 1-3 cm long. Fruits cylindrical to almost globose; 20-35 x 10-15 cm; yellowish or brown to orange-green.





**Figure 4** Parts of *Artocarpus integer*

### 3.1.2 Review of Literatures

Chemical constituents isolated from *Artocarpus* genus were summarized in **Table 57**. The literature survey was done from SciFinder Scholar database and the constituents could be classified into groups, such as benzofuran, chalcone, dihydrochalcones, flavonoids, neolignan, stilbenoids, steroids and triterpenoids.

**Table 57** Compounds from plants of *Artocarpus* genus

**a** = Benzofuran      **b** = Chalcone      **c** = Dihydrochalcones  
**d** = Flavonoids      **e** = Neolignan      **f** = Stilbenoids  
**g** = Steroids      **h** = Triterpenoids

Scientific name	Investigated Part	Compound	Bibliography
<i>A. altilis</i>	Bud cover	AC-5-1, <b>1c</b> Cycloaltisin 6, <b>8c</b> Cycloaltisin 7, <b>7d</b>	Patil <i>et al.</i> , 2002
	Leaves	1-(2,4-Dihydroxyphenyl)-3-[8-hydroxy-2-methyl-2-(4-methyl-3-pentenyl)-2H-1-benzopyran-5-yl]-1-propanone, <b>2c</b> 1-(2,4-Dihydroxyphenyl)-3-{4-hydroxy-6,6,9-trimethyl-6a,7,8,10a-atetrahydro-6H-dibenzo[ <i>b,d</i> ]pyran-5-yl}-1-propanone, <b>9c</b> 2-Geranyl-2',3,4,4',-tetrahydroxydihydrochalcone, <b>6c</b>	Wang <i>et al.</i> , 2007

Table 57 (Continued)

Scientific name	Investigated Part	Compound	Bibliography
<i>A. altilis</i>	Leaves	<p>1-(2,4-Dihydroxyphenyl)-3-[3,4-dihydro-3,8-dihydroxy-2-methyl-2-(4-methyl-3-pentenyl)-2H-1-benzopyran-5-yl]-1-propanone, <b>3c</b></p> <p>1-(2,4-Dihydroxyphenyl)-3-[8-hydroxy-2-methyl-2-(3,4-epoxy-4-methyl-1-pentenyl)-2H-1-benzopyran-5-yl]-1-propanone, <b>4c</b></p> <p>1-(2,4-Dihydroxyphenyl)-3-[8-hydroxy-2-methyl-2-(4-hydroxy-4-methyl-2-pentenyl)-2H-1-benzopyran-5-yl]-1-propanone, <b>5c</b></p> <p>2-[6-Hydroxy-3,7-dimetylocta-2(E),7-dienyl]-2',3,4,4'-tetrahydroxydihydrochalcone, <b>7c</b></p> <p>2'-Geranyl-3',4',7-trihydroxyflavanone, <b>8d</b></p> <p>Cycloaltilisin 6, <b>8c</b></p>	Wang <i>et al.</i> , 2007
<i>A. chama</i>	Roots	<p>Artochamin A, <b>52d</b></p> <p>Artochamin B, <b>50d</b></p> <p>Artochamin C, <b>25d</b></p> <p>Artochamin D, <b>26d</b></p>	Wang <i>et al.</i> , 2004

Table 57 (continued)

Scientific name	Investigated Part	Compound	Bibliography
<i>A. chama</i>	Roots	Artochamin E, <b>36d</b> Artocarpin, <b>18d</b> Cycloartocarpin A, <b>58d</b> Cudraflavone A, <b>51d</b> Artonin A, <b>48d</b> Artonin U, <b>14d</b> Cycloartobiloxanthone, <b>46d</b> Artonin E, <b>20d</b> 3',4',5,7-Tetrahydroxy-8-(methylbut-2-enyl)flavone, <b>15d</b>	Wang <i>et al.</i> , 2004
<i>A. champeden</i>	Bark	Cyclochampedol, <b>55d</b>  Cycloeucalenol, <b>1g</b> Glutinol, <b>1h</b> Cycloartenone, <b>2g</b> 24-Methylenecycloartenone, <b>3g</b> $\beta$ -Sitosterol, <b>4g</b>	Achmad <i>et al.</i> , 1996
	Heartwood	Artoindonesianin Q, <b>29d</b> Artoindonesianin R, <b>30d</b> Artoindonesianin S, <b>37d</b> Artoindonesianin T, <b>38d</b> Artoindonesianin U, <b>35d</b> Artoindonesianin V, <b>41d</b> 5'-Hydroxycudraflavone A, <b>53d</b>	Syah <i>et al.</i> , 2002     Syah <i>et al.</i> , 2004

Table 57 (continued)

Scientific name	Investigated Part	Compound	Bibliography
<i>A. champeden</i>	Heartwood	Cyclocommunin, <b>62d</b>  Artonin B, <b>59d</b> Artoindonesianin A-2, <b>56d</b> Artoindonesianin A-3, <b>40d</b> Artonin B, <b>59d</b> Heterophyllin, <b>31d</b> Cudraflavone C, <b>19d</b> Artoindonesianin Q, <b>29d</b> Artoindonesianin R, <b>30d</b> Artoindonesianin T, <b>38d</b>	Syah <i>et al.</i> , 2004          Syah <i>et al.</i> , 2006
	Roots	Artoindonesianin A, <b>43d</b> Artoindonesianin B, <b>11d</b> Artonin A, <b>48d</b>	Hakim <i>et al.</i> , 1999
<i>A. communis</i>	Roots	Artocommunol CA, <b>16d</b> Artocommunol CB, <b>60d</b> Artocommunol CC, <b>61d</b> Artocommunol CD, <b>24d</b> Artocommunol CE, <b>17d</b> Cyclomorusin, <b>54d</b>	Chan <i>et al.</i> , 2003
	Heartwood	3",3"- Dimethylpyrano[3',4']2,4, 2'-trihydroxychalcone, <b>1b</b> Isobacachalcone, <b>2b</b> Morachalcone A, <b>3b</b>	Han <i>et al.</i> , 2006



Table 57 (continued)

Scientific name	Investigated Part	Compound	Bibliography
<i>A. dadah</i>	Twigs	Resveratrol, <b>3f</b> Steppogenin, <b>2d</b> Moracin M, <b>1a</b> Isogemichalcone B, <b>6b</b> Gemichalcone B, <b>5b</b> Norartocarpetin, <b>12d</b> Engelet, <b>3d</b>	Su <i>et al.</i> , 2002  Su <i>et al.</i> , 2002
<i>A. elasticus</i>	Root bark	Artelastoheterol, <b>33d</b> Artelasticinol, <b>28d</b> Cycloartelastoxanthone, <b>45d</b> Artelastoxanthone, <b>39d</b> Cycloartelastoxanthediol, <b>47d</b> Artonin F, <b>49d</b> Cycloartobiloxanthone, <b>46d</b> Cyclomorusin, <b>54d</b>	Ko <i>et al.</i> , 2005
<i>A. fretessi</i>	Bark+Roots	Artoindonesianin X, <b>6a</b> Artoindonesianin Y, <b>5a</b> Mulberrin, <b>13d</b> Norartocarpetin, <b>12d</b> (±)-Catechin, <b>1d</b> (-)-Afzelechin-3- <i>O</i> -rhamnoside, <b>6d</b> Mulberrochromene, <b>21d</b> Artonin A, <b>48d</b>	Soekamto <i>et al.</i> , 2003      Soekamto <i>et al.</i> , 2003

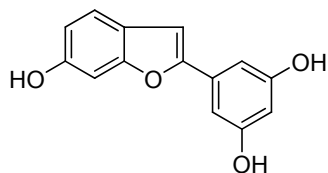
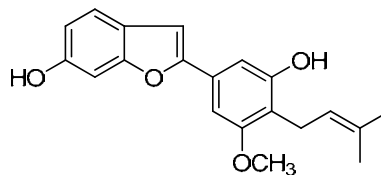
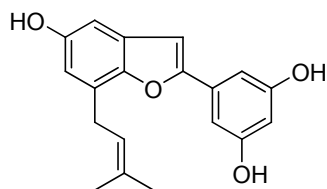
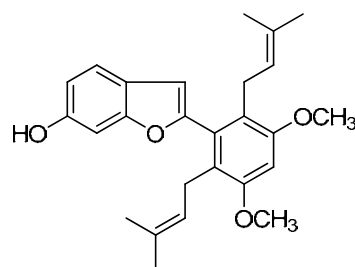
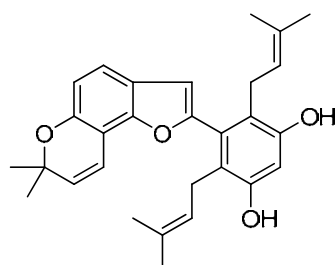
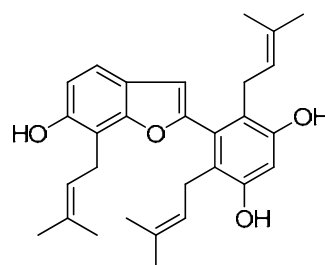
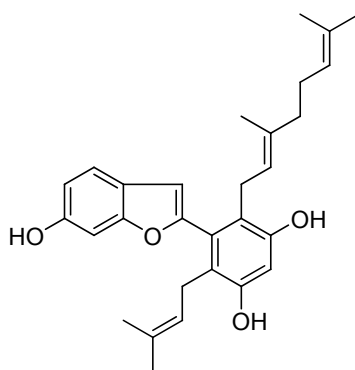
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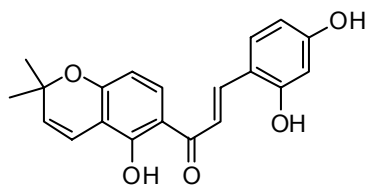
Scientific name	Investigated Part	Compound	Bibliography
<i>A. fretessi</i>	Bark+Roots	(-)-Afzelechin, <b>5d</b>	Soekamto <i>et al.</i> , 2003
<i>A. gomezianus</i>	Bark	Artoindonesianin N, <b>2f</b> Artoindonesianin O, <b>2a</b> Oxyresveratrol, <b>1f</b>	Hakim <i>et al.</i> , 2002
<i>A. lakoocha</i>	Roots	Lakoochin A, <b>4a</b> Lakoochin B, <b>7a</b>	Puntumchai <i>et al.</i> , 2004
<i>A. lanceifolius</i>	Bark	Artoindonesianin P, <b>42d</b> Artobiloxanthone, <b>44d</b> Cycloartobiloxanthone, <b>46d</b>	Hakim <i>et al.</i> , 2002
<i>A. nobilis</i>	Leaves	2',4'-Trihydroxy-3'-geranylchalcone, <b>7b</b> 2',4',4'-Trihydroxy-3'-[6-hydroxy-3,7-dimethyl-2( <i>E</i> ),7-octadienyl]chalcone, <b>8b</b> 2',4',4'-Trihydroxy-3'-[2-hydroxy-7-methyl-3-methylene-6-octaenyl]chalcone, <b>9b</b> 2',3,4,4'-Tetrahydroxy-3'-geanyloxychalcone, <b>10b</b>	Jayasinghe <i>et al.</i> , 2004



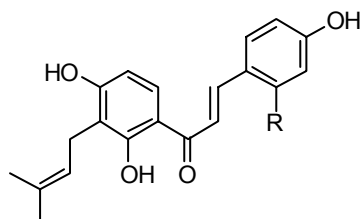
Table 57 (continued)

Scientific name	Investigated Part	Compound	Bibliography
<i>A. nobilis</i>	Leaves	2',3,4,4'-Tetrahydroxy-3'-[6-hydroxy-3,7-dimethyl-2( <i>E</i> ),7-octadienyl]chalcone, <b>11b</b>	Jayasinghe <i>et al.</i> , 2004
	Root bark	Artobioxanthone, <b>44d</b> Artonin E, <b>20d</b> Cycloartobioxanthone, <b>46d</b> Artonin E 2'-methylether, <b>22d</b> Isortonin E 2'-methylether, <b>34d</b> Dihydroisoartonin E 2'-methylether, <b>32d</b> Artonin V 2'-methylether, <b>27d</b>	Jayasinghe <i>et al.</i> , 2008
<i>A. sepicanus</i>	Leaves	Sepicanin A, <b>10d</b>  Afzelechin-3-O- $\alpha$ -L-rhamnopyranoside, <b>6d</b>	Radwan <i>et al.</i> , 2009

**Structure****a: Benzofuran****1a: Moracin M****2a: Artoindonesianin O****3a: 3-(γ,γ-Dimethylpropenyl)moracin M****4a: Lakoochin A****5a: Artoindonesianin Y****6a: Artoindonesianin X****7a: Lakochin B**

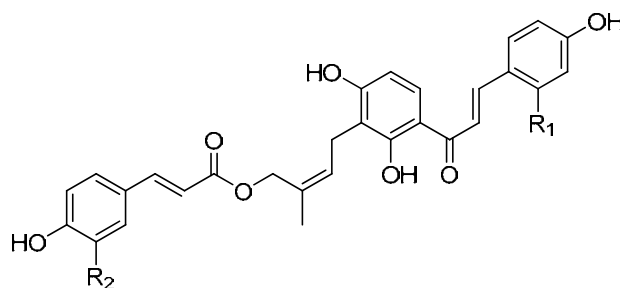
**b: Chalcone**

**1b:** 3'',3''-Dimethylpyrano[3',4']-  
2,4,2'-trihydroxychalcone



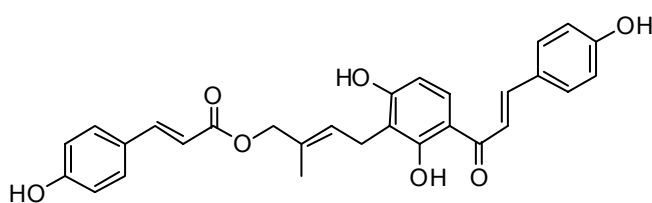
**2b:** R = H: Isobacachalcone

**3b:** R = OH: Morachalcone A

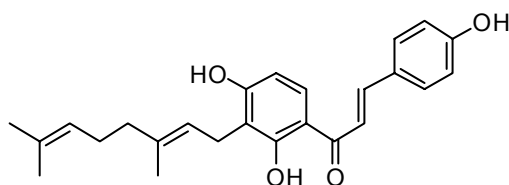


**4b:** R<sub>1</sub> = R<sub>2</sub> = H; Gemichalcone B

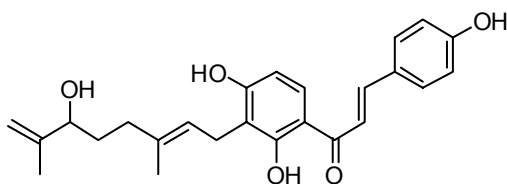
**5b:** R<sub>1</sub> = OH, R<sub>2</sub> = OCH<sub>3</sub>; Gemichalcone C



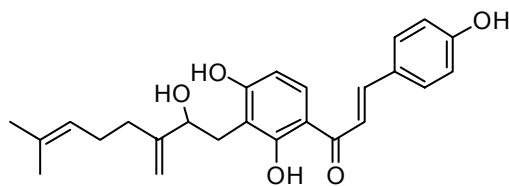
**6b:** Isogemichalcone B



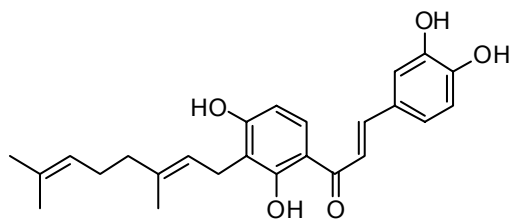
**7b:** 2',4',4'-Trihydroxy-3'-  
geranylchalcone



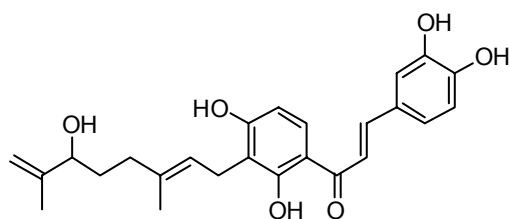
**8b:** 2',4',4'-Trihydroxy-3'-[6-  
hydroxy-3,7-dimethyl-2(*E*),7-  
octadienyl]chalcone



**9b:** 2',4',4'-Trihydroxy-3'-[2-hydroxy-7-methyl-3-methylene-6-octaenyl]chalcone

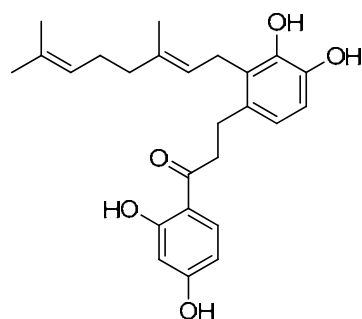


**10b:** 2',3,4,4'-Tetrahydroxy-3'-geanyloxychalcone

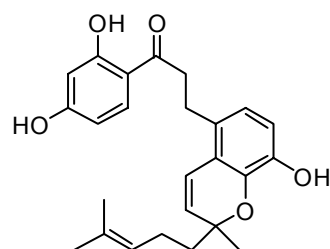


**11b:** 2',3,4,4'-Tetrahydroxy-3'-[6-hydroxy-3,7-dimethyl-2(*E*),7-octadienyl]chalcone

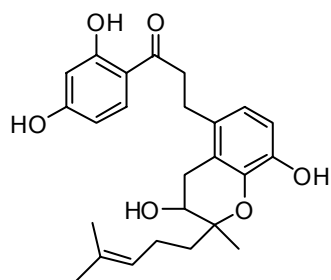
**c : Dihydrochalcone**



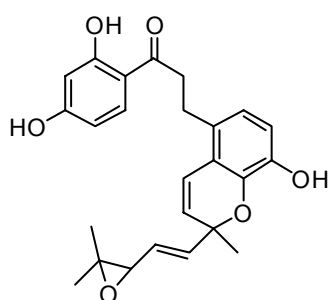
**1c:** AC-5-1



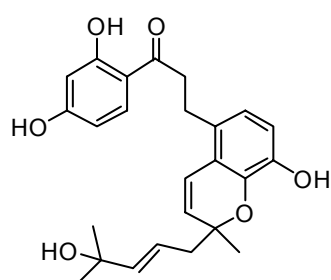
**2c:** 1-(2,4-Dihydroxyphenyl)-3-[8-hydroxy-2-methyl-2-(4-methyl-3-pentenyl)-2H-1-benzopyran-5-yl]-1-propanone



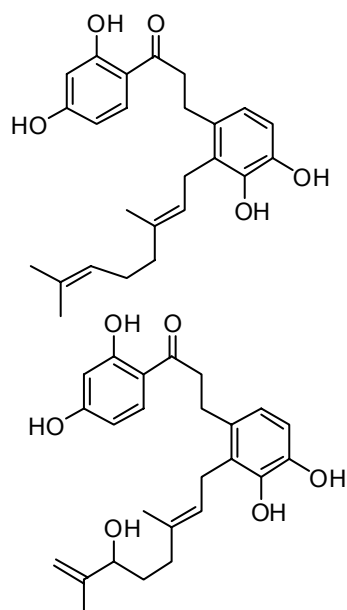
**3c:** 1-(2,4-Dihydroxyphenyl)-3-[3,4-dihydro-3,8-dihydroxy-2-methyl-2-(4-methyl-3-pentenyl)-2H-1-benzopyran-5-yl]-1-propanone



**4c:** 1-(2,4-Dihydroxyphenyl)-3-[8-hydroxy-2-methyl-2-(3,4-epoxy-4-methyl-1-pentenyl)-2H-1-benzopyran-5-yl]-1-propanone

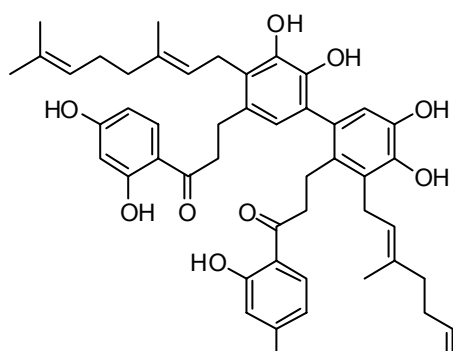
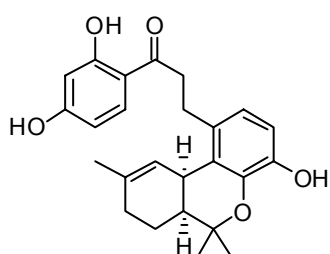
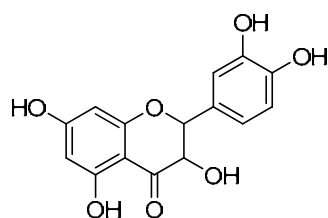
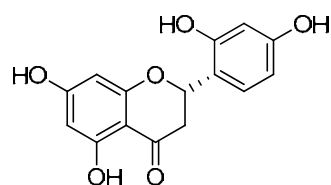
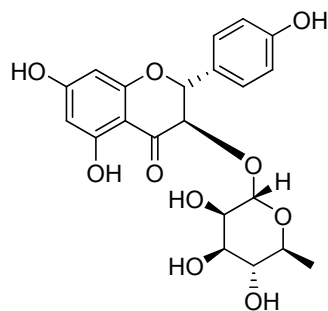
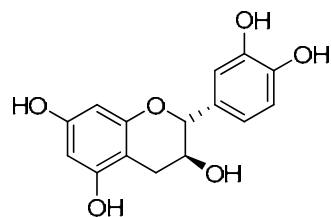


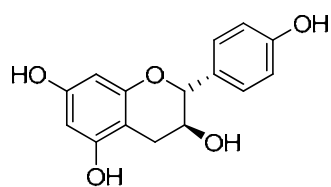
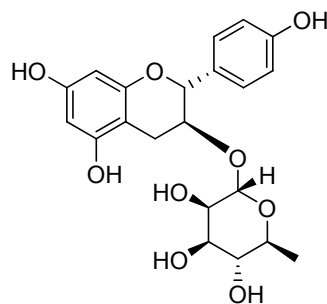
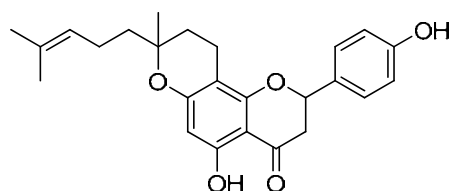
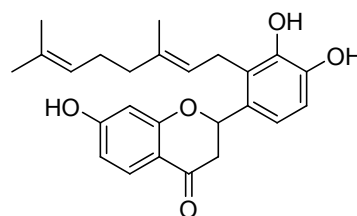
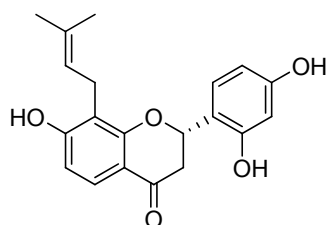
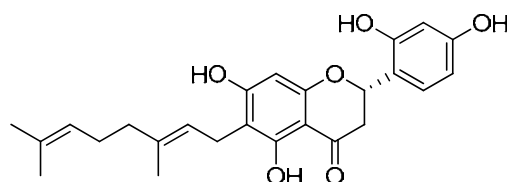
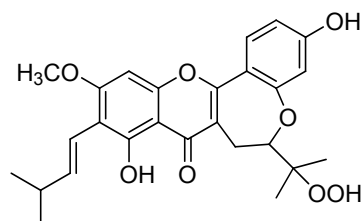
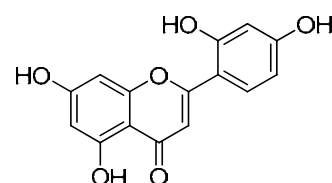
**5c:** 1-(2,4-Dihydroxyphenyl)-3-[8-hydroxy-2-methyl-2-(4-hydroxy-4-methyl-2-pentenyl)-2H-1-benzopyran-5-yl]-1-propanone

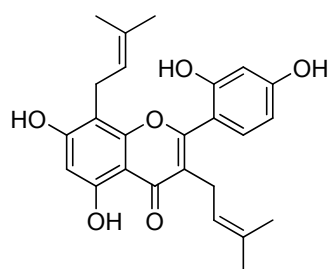
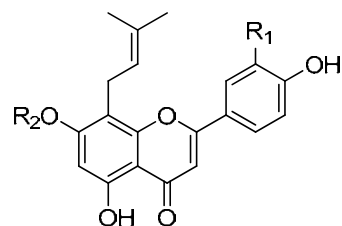


**6c:** 2-Geranyl-2',3,4,4',-tetrahydroxy-dihydrochalcone

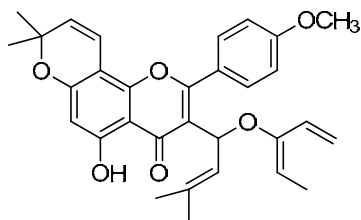
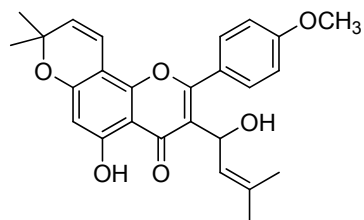
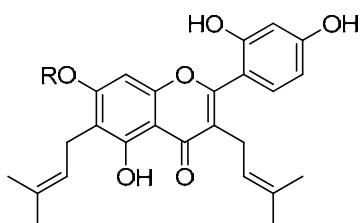
**7c:** 2-[6-Hydroxy-3,7-dimethylocta-2(E),7-dienyl]-2',3,4,4'-tetrahydroxydihydrochalcone

**8c:** Cycloaltisin 6**9c:** 1-(2,4-Dihydroxyphenyl)-3-{4-hydroxy-6,6,9-trimethyl-6a,7,8,10a-tetrahydro-6H-dibenzo[*b,d*]pyran-5-yl}-1-propanone,**d: Flavonoids****1d:** (±)-Catachin**2d:** Steppogenin**3d:** Engelet**4d:** (+)-Epicatechin

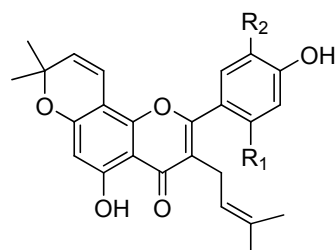
**5d:** (-)-Afzelechin**6d:** Afzelechin-3-O- $\alpha$ -L-rhamnopyranoside**7d:** Cycloaltisin 7**8d:** 2'-Geranyl-3',4',7-trihydroxyflavanone**9d:** (2*S*)-Euchrenone a<sub>7</sub>**10d:** Sepicanin A**11d:** Artoindonesianin B**12d:** Norartocarpetin

**13d:** Mulberrin**14d:**  $R_1 = H, R_2 = CH_3$ ; Artonin U

**15d:**  $R_1 = OH, R_2 = H$ ; 3',4',5,7-Tetrahydroxy-8-(methylbut-2-enyl)flavones

**16d:** Artocommunol CA**17d:** Artocommunol CE**18d:**  $R = CH_3$ ; Artocarpin

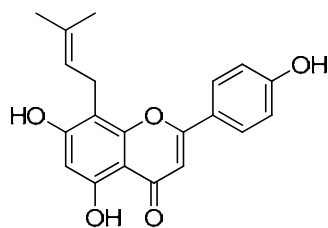
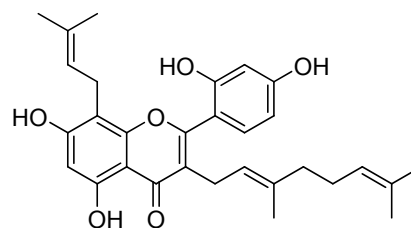
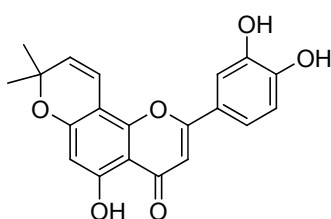
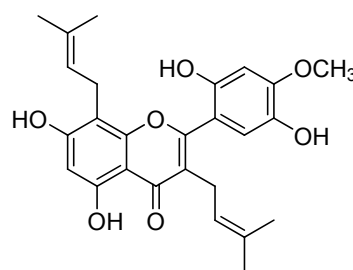
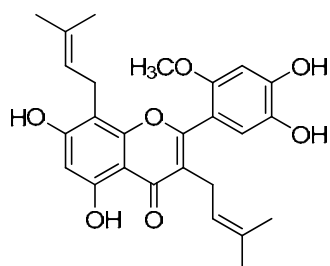
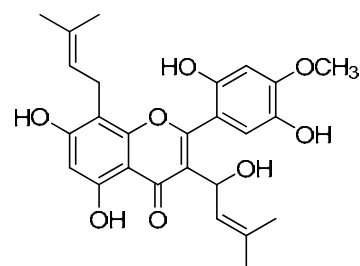
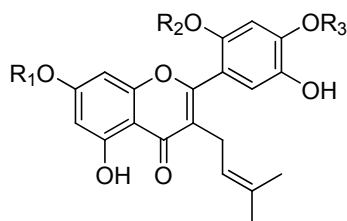
**19d:**  $R = H$ ; Cudraflavone C

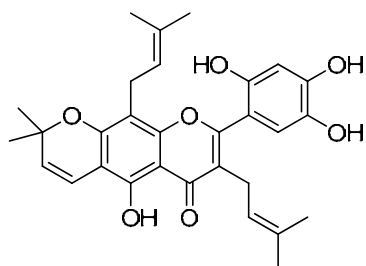
**20d:**  $R_1 = R_2 = OH$ ; Artonin E

**21d:**  $R_1 = OH, R_2 = H$ ; Mulberrochromene

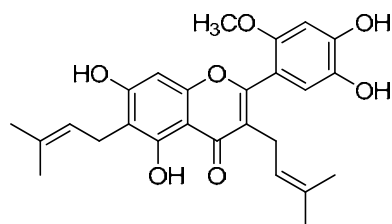
**22d:**  $R_1 = OCH_3, R_2 = OH$ ; Artonin E 2'-methylether



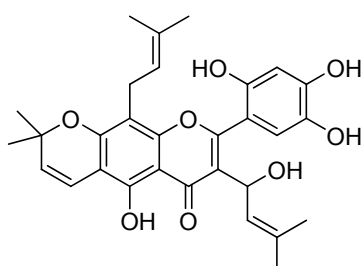
**23d:** Licoflavone C**24d:** Artocommunol CD**25d:** Artochamin C**26d:** Artochamin D**27d:** Artonin V 2'-methyl ether**28d:** Artelastincol**29d:**  $R_1 = R_3 = \text{CH}_3$ ,  $R_2 = \text{H}$ : Artoindonesianin Q**30d:**  $R_1 = \text{H}$ ,  $R_2 = R_3 = \text{CH}_3$ : Artoindonesianin R



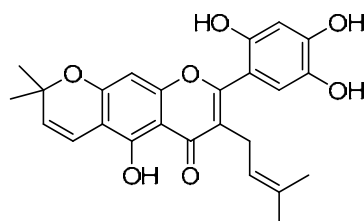
**31d:** Hetrophyllin



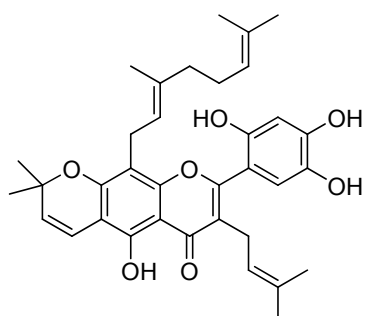
**32d:** Dihydroisoartonin E 2'-methylether



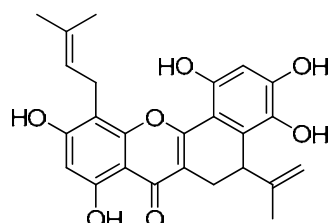
**33d:** Artelastoheterol



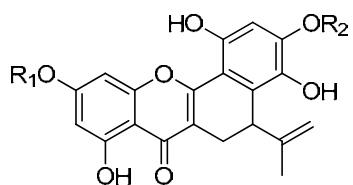
**34d:** Isoartonin E 2'-methylether



**35d:** Artoindonesianin U

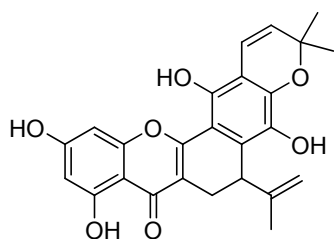
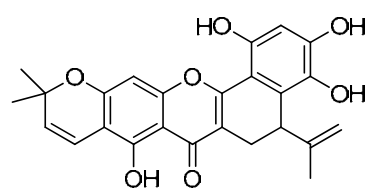
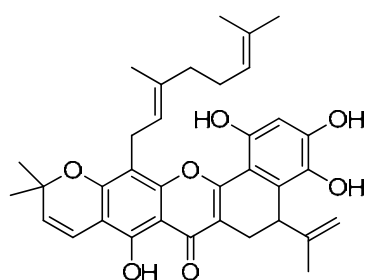
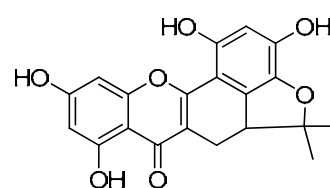
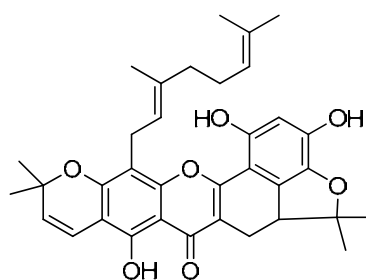
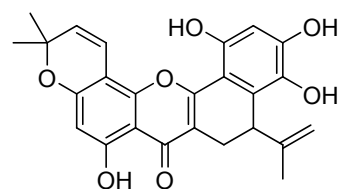
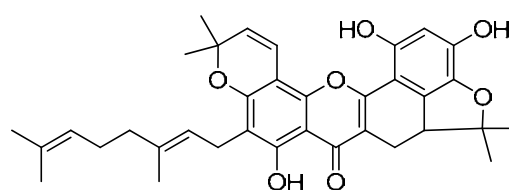
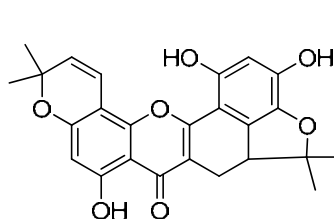
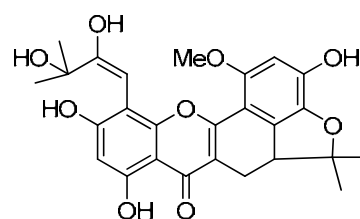


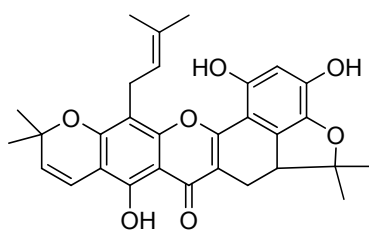
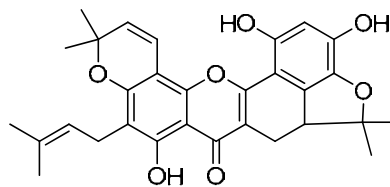
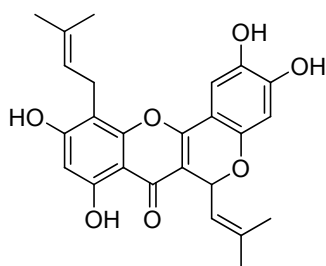
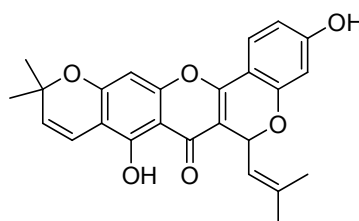
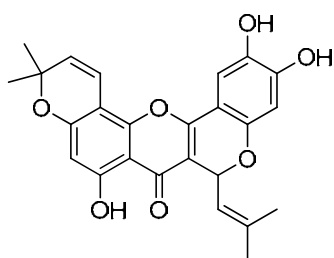
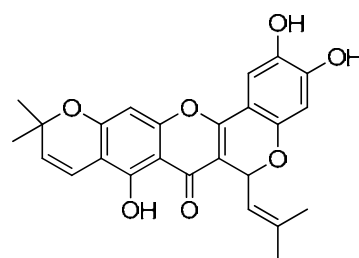
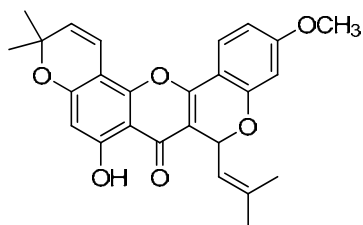
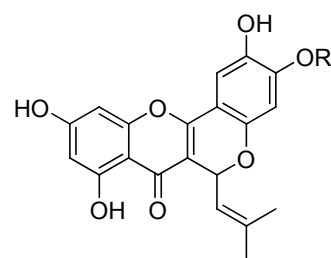
**36d:** Artochamin E

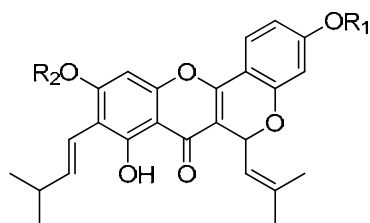


**37d:**  $R_1 = R_2 = \text{CH}_3$ : Artoindonesianin S

**38d:**  $R_1 = \text{H}, R_2 = \text{CH}_3$ : Artoindonesianin T

**39d:** Artelastoxanthone**40d:** Artoindonesianin A-3**41d:** Artoindonesianin V**42d:** Artoindonesianin P**43d:** Artoindonesianin A**44d:** Artibiloxanthone**45d:** Cycloartelastoxanthone**46d:** Cycloartobiloxanthone**47d:** Cycloartelastoxanthendiol

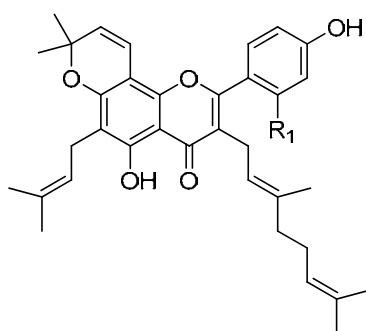
**48d:** Artonin A**49d:** Artonin F**50d:** Artonin B**51d:** (-)-Cudraflavone A**52d:** Artochamin A**53d:** 5'-Hydroxycudraflavone A**54d:** Cycломорусин**55d:** R = H; Cyclochampedol**56d:** R = CH<sub>3</sub>; Artoindonesianin



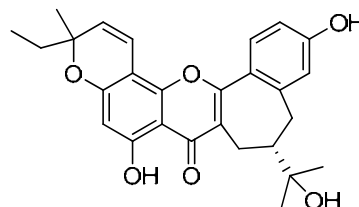
**57d:** R<sub>1</sub> = H, R<sub>2</sub> = CH<sub>3</sub>; (-)-Cycloartocarpin

**58d:** R<sub>1</sub> = CH<sub>3</sub>, R<sub>2</sub> = H; Cycloartocarpin A

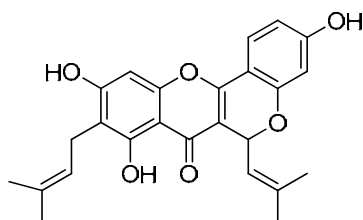
**59d:** R<sub>1</sub> = R<sub>2</sub> = CH<sub>3</sub>; Artonin B



**60d:** Artocommunol CB

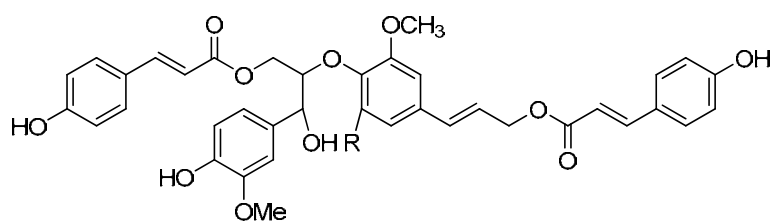


**61d:** Artocommunol CC



**62d:** Cyclocommunin

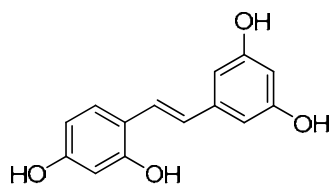
### e: Neolignans



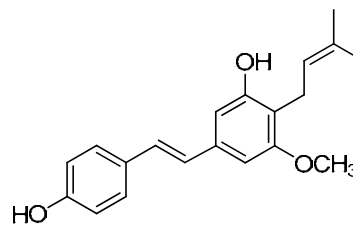
**1e:** R = OCH<sub>3</sub>; Dadahol A

**2e:** R = H; Dadahol B

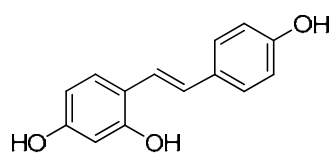
## f: Stilbenoids



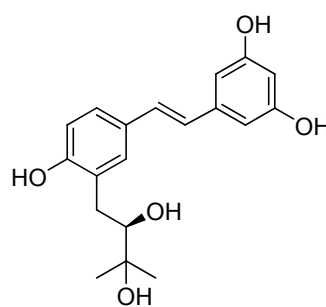
1f: Oxyresveratrol



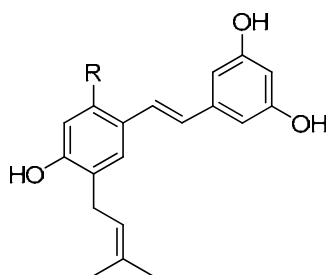
2f: Artoindonesianin N

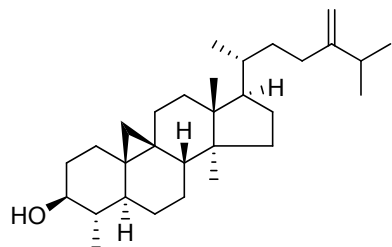
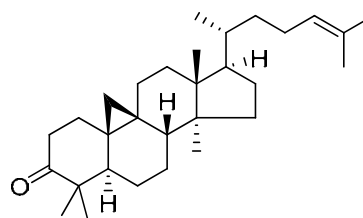
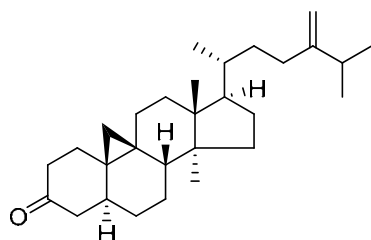
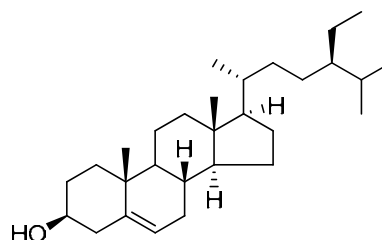
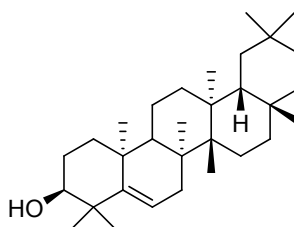


3f: Resveratrol



4f: 3-(2,3-Dihydroxy-3-methylbutyl)resveratrol

5f: R = H; 3-( $\gamma,\gamma$ -Dimethylallyl)resveratrol6f: R = OH; 5-( $\gamma,\gamma$ -Dimethylallyl)oxyresveratrol

**g: Steroids****1g:** Cycloeucalenol**2g:** Cycloartenone**3g:** 24-Methylenecycloartenone**4g:**  $\beta$ -Sitosterol**h: Triterpenoids****1h:** Glutinol**3.1.3 The objectives**

The goals of this work were to investigate the chemical constituents from the roots of *Artocarpus integer* and to evaluate the antibacterial and cytotoxic activities of the isolated compounds.

## CHAPTER 3.2

### EXPERIMENTAL

#### 3.2.1 Instruments and Chemicals

Melting point was recorded in °C on an Electrothermal 9100 melting point apparatus. Ultraviolet (UV) absorption spectra were recorded using a SPECORD S100 spectrophotometer (Analytikjena) and principle bands ( $\lambda_{\max}$ ) were recorded as wavelengths (nm) and  $\log \varepsilon$  in methanol solution. The infrared spectra were recorded using FTS 165 FT-IR Perkin Elmer spectrophotometer. Nuclear Magnetic resonance spectra were recorded using Bruker Avance 300 MHz Bruker FTNMR Ultra Shield™. Spectra were recorded in deuteriochloroform, deuterioacetone and deuteromethanol and were recorded as  $\delta$  value in ppm downfield from TMS (Internal standard  $\delta$  0.00). Optical rotation was measured in MeOH solution at the sodium D line (590 nm) on an AUTOPOL<sup>R</sup> II automatic polarimeter. The EI-MS and HREIMS mass spectra were obtained from a Micromass LCT mass spectrometer. Solvents for extraction and chromatography were distilled at their boiling point ranges prior to use. Quick column chromatography (QCC) and column chromatography (CC) were carried out on silica gel 60 F<sub>254</sub> (Merck) and silica gel 100, respectively. Precoated plates of silica gel 60 GF<sub>254</sub> were used for analytical purposes.

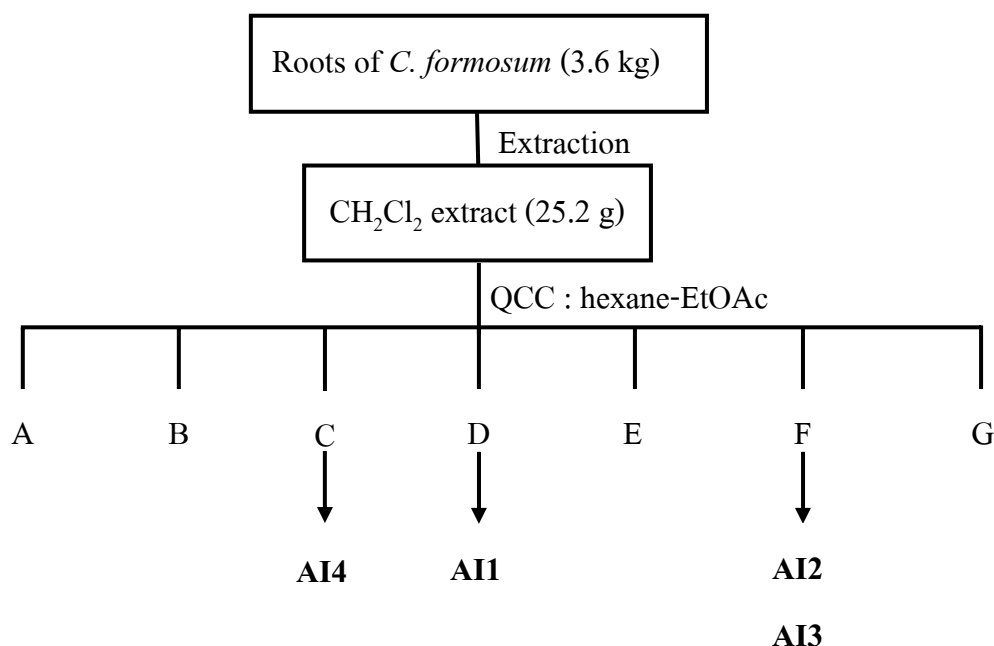
#### 3.2.2 Plant Material

The roots of *A.integer* were collected from Sa Toon Province, Thailand. The plant was identified by Prof. Puangpen Sirirugsa.



### 3.2.3 Extraction and investigation of the crude dichloromethane extract from the roots of *A. integer*

Air-dried roots (3.6 kg) were chopped and extracted with dichloromethane at room temperature for three days. Evaporation of the solvent under reduced pressure furnished a crude dichloromethane extract (25.2 g).



**Scheme 4** Extraction and isolation of compounds **AI1-AI4** from the root of *A. integer*

The crude dichloromethane extract was subjected to quick column chromatography (QCC) on silica gel with solvent mixtures of increasing polarity [hexane to EtOAc] to yield seven fractions (A-G). Fraction C was purified by QCC using a gradient of EtOAc-hexane to afford five subfractions (A<sub>1</sub>-A<sub>5</sub>). Fractions A<sub>3</sub> was further purified by QCC using a gradient of EtOAc-hexane as a mobile phase to give **AI4** (4.4 mg). Fraction D was separated by QCC with a gradient system of increasing EtOAc in hexane to afford seven subfractions (D<sub>1</sub>-D<sub>7</sub>). Subfraction D<sub>3</sub> was further purified by QCC using a gradient of EtOAc-hexane to give **AI1** (7.0 mg). Fraction F was subjected to repeated column chromatography over silica gel to afford **AI2** (30.6 mg) and **AI3** (8.1 mg).

Compound **AI1**: yellow powder; mp 234-236 °C; UV (MeOH)  $\lambda_{\max}$  (log  $\epsilon$ ) 296 (2.93), 386 (2.78) nm; IR (KBr)  $\nu_{\max}$  3368, 1676, 1602, 1515  $\text{cm}^{-1}$ ;  $^1\text{H}$  and  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ ) spectra see **Table 58**.

Compound **AI2**: yellow powder; mp 190-192 °C; UV (MeOH)  $\lambda_{\max}$  (log  $\epsilon$ ) 296 (3.88), 332 (3.79) nm; IR (KBr)  $\nu_{\max}$  3234, 1654, 1611, 1506, 1354  $\text{cm}^{-1}$ ;  $^1\text{H}$  and  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ ) spectra see **Table 60**.

Compound **AI3**: yellow solid; mp 238-239 °C; UV (MeOH)  $\lambda_{\max}$  (log  $\epsilon$ ) 290 (3.75), 377 (4.15) nm; IR (KBr)  $\nu_{\max}$  3449, 1648, 1596, 1492, 1440, 1367  $\text{cm}^{-1}$ ;  $^1\text{H}$  and  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ ) spectra see **Table 62**.

Compound **AI4**: yellow-brown viscous oil; UV (MeOH)  $\lambda_{\max}$  (log  $\epsilon$ ) 290 (3.59) nm; IR (KBr)  $\nu_{\max}$  3200, 1603, 1476, 1148  $\text{cm}^{-1}$ ;  $^1\text{H}$  and  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ ) spectra see **Table 64**.

### 3.2.4 BIOASSAY

#### 3.2.4.1 Antibacteria assay

The isolated compounds from the roots of *A. integer* were 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*, *Pseudomonas aeruginosa*, *Shigella sonnei* and *Salmonella typhi*. 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 *et al.* (Boonsri *et al.*, 2006). Vancomycin, which was used as a standard, showed antibacterial activity of 0.078  $\mu\text{g}/\text{mL}$ .

#### **3.2.4.2 Antifungal assay**

*Candida albicans* was obtained from Department of Pharmacognosy and Botany, PSU. The antifungal employed was the same as described in Boonsri *et al.* (Boonsri *et al.*, 2006). Amphotericin B was used as a standard.

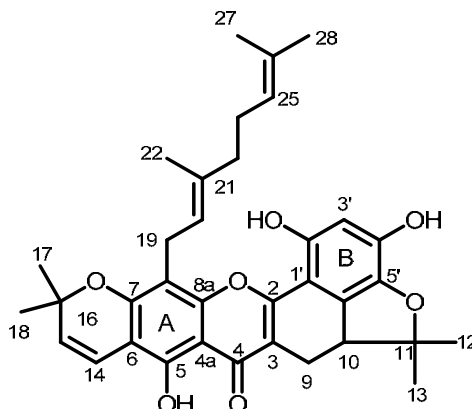
## CHAPTER 3.3

### RESULTS AND DISCUSSION

#### 3.3.1 Structural determination of compounds isolated from the roots of *A. integer*

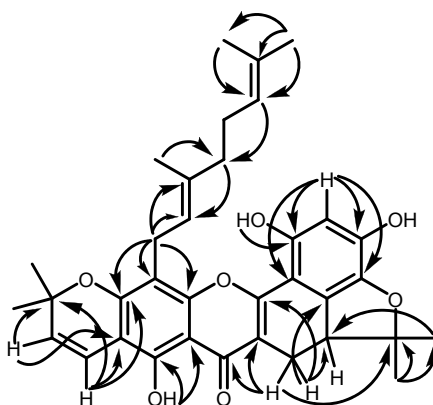
The crude hexane extract from the roots of *A. integer* was subjected to a succession of chromatographic procedures, including silica gel column chromatography and preparative TLC to afford four known compounds, **AI1-AI4**. All structures were elucidated using 1D and 2D NMR spectroscopic data and comparison with those reported in the literatures.

### 3.3.1.1 Compound AI1



**AI1** was isolated as a yellow powder. The IR spectrum (**Figure 117**) of **AI1** exhibited strong absorption bands due to hydroxyl ( $3368\text{ cm}^{-1}$ ) and a conjugated carbonyl groups ( $1676\text{ cm}^{-1}$ ). The UV absorption bands (296 and 386 nm) (**Figure 116**) were typical of a flavone chromophore (Syah *et al.*, 2004). The  $^1\text{H}$  NMR spectrum of **AI1** (**Table 58**, **Figure 118**) contained resonances for one chelated [ $\delta$  13.20 (1H, *s*, 5-OH)] and a free hydroxyl groups [ $\delta$  6.61 (1H, *s*, 3'-OH)]. A  $^1\text{H}$  NMR signal of a 1,2,4,5,6-pentasubstituted benzene ring resonating at  $\delta$  6.26 (1H, *s*) was assigned to H-3' because of its HMBC correlations to C-1' ( $\delta$  103.4), C-2' ( $\delta$  149.8), C-4' ( $\delta$  145.1), C-5' ( $\delta$  138.0). The signals of a geranyl moiety [ $\delta$  1.48 (3H, *s*, H-27), 1.55 (3H, *s*, H-28), 1.74 (3H, *s*, H-22), 1.95 (2H, *m*, H-23), 2.06 (2H, *m*, H-24), 3.37 (2H, *m*, H-19), 4.99 (1H, *m*, H-25) and 5.00 (1H, *m*, H-20)] and a dimethylchromene ring [ $\delta$  6.67 (1H, *d*,  $J = 10.0\text{ Hz}$ , H-14), 5.56 (1H, *d*,  $J = 10.0\text{ Hz}$ , H-14), 1.40 (6H, *s*, H-17 and H-18)] were also observed. The geranyl group was placed at C-8 due to HMBC correlations of a benzylic allylic methylene protons ( $\delta$  3.37, H-19) of the geranyl group which showed cross peak with C-7 ( $\delta$  156.3), C-8 ( $\delta$  106.9) and C-8a ( $\delta$  153.3). The dimethylchromene group was connected to ring A at C-6 and C-7 as evidenced by HMBC correlations of the vinylic proton at  $\delta$  6.67 (H-14) with C-5 ( $\delta$  154.6) and C-7 ( $\delta$  156.3). Furthermore, signals of an isoprenyl group which comprised of protons resonating at  $\delta$  1.28 (3H, *s*, H-12), 1.60 (3H, *s*, H-13), 3.37 (1H, *m*, H-10), 2.37 (1H, *t*,  $J = 15.2\text{ Hz}$ , H-9) and 3.19 (1H, *dd*,  $J = 15.2, 7.2\text{ Hz}$ , H-9)

were displayed. In the HMBC spectrum, the methylene signal at  $\delta$  2.37 (1H, H-9) and 3.19 (1H, H-9) showed cross peaks with carbonyl carbon at  $\delta$  180.8 (C-4), oxygenated aromatic carbons at  $\delta$  159.9 (C-2) and quaternary aromatic carbon  $\delta$  131.2 (C-6') and methyl signals at  $\delta$  1.28 (3H, H-12) and 1.60 (3H, H-13) with methine carbon at  $\delta$  46.3 (C-10), indicating that a prenyl group was connected to the C-3 position and cyclized to form a cyclohexene ring at C-6' of ring B. In addition, the  $sp^3$  oxyquaternary carbon at  $\delta$  94.9 of a prenyl group was observed, whose downfield signal suggested that a connection to oxygen atom and the dihydrobenzofuran was formed. Thus, **AI1** was identified as artoindonesianin A (Hakim *et al.*, 1999).



Selected HMBC Corelation of **AI1**

**Table 58**  $^1\text{H}$ ,  $^{13}\text{C}$  NMR, DEPT and HMBC spectral data of **AI1**

position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	DEPT	HMBC
2		159.9	C	
3		111.9	C	
4		180.8	C	
4a		104.6	C	
5		154.6	C	
6		105.7	C	
7		156.3	C	
8		106.9	C	
8a		153.3	C	
9	3.19 ( <i>dd</i> , 15.2, 7.2) 2.37 ( <i>t</i> , 15.2)	20.0	CH <sub>2</sub>	2, 3, 4, 10, 11, 6'
10	3.37 ( <i>m</i> )	46.3	CH	
11		94.9	C	
12	1.28 ( <i>s</i> )	22.7	CH <sub>3</sub>	10, 11, 13
13	1.60 ( <i>s</i> )	28.1	CH <sub>3</sub>	10, 11, 12
14	6.67 ( <i>d</i> , 10.0)	115.9	CH	5, 7, 16
15	5.56 ( <i>d</i> , 10.0)	128.0	CH	6, 16
16		77.9		
17	1.40 ( <i>s</i> )	28.2	CH <sub>3</sub>	15, 16, 18
18	1.40 ( <i>s</i> )	28.2	CH <sub>3</sub>	15, 16, 17
19	3.37 ( <i>m</i> )	21.3	CH <sub>2</sub>	7, 8, 8a, 20, 21
20	5.00 ( <i>m</i> )	121.0	CH	19, 22, 23
21		138.0	C	
22	1.74 ( <i>s</i> )	16.6	CH <sub>3</sub>	20, 21, 23
23	1.95 ( <i>m</i> )	39.5	CH <sub>2</sub>	20, 21, 24
24	2.06 ( <i>m</i> )	26.4	CH <sub>2</sub>	23
25	4.99 ( <i>m</i> )	124.2	CH	23
26		131.5	C	

**Table 58** (Continued)

position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	DEPT	HMBC
27	1.48 ( <i>s</i> )	17.7	CH <sub>3</sub>	25, 26
28	1.55 ( <i>s</i> )	25.6	CH <sub>3</sub>	25, 26
1'		103.4	C	
2'		149.8	C	
3'	6.26 ( <i>s</i> )	104.6	CH	1', 2', 4', 5'
4'		145.1	C	
5'		138.0	C	
6'		131.2	C	
5-OH	13.20 ( <i>s</i> )			4a, 5,
2'-OH	6.61 ( <i>s</i> )			2', 3'

**Table 59** Comparison of <sup>1</sup>H and <sup>13</sup>C NMR spectral data of **AI1** and artoindonesianin

A

position	<b>AI1</b>		Artoindonesianin A <sup>a</sup>	
	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$
2		159.9		160.7
3		111.9		110.9
4		180.8		180.2
4a		104.6		103.8
5		154.6		153.5
6		105.7		104.5
7		156.3		155.6
8		106.9		107.0
8a		153.3		152.9



Table 59 (Continued)

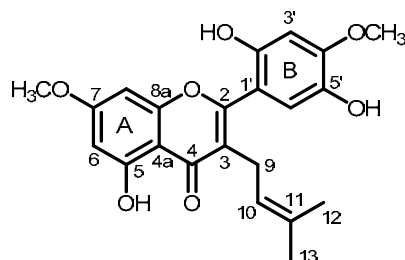
position	AII		Artoindonesianin A <sup>a</sup>	
	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$
9	3.19 ( <i>dd</i> , 15.2, 7.2) 2.37 ( <i>t</i> , 15.2)	20.0	3.10 ( <i>dd</i> , 15.2, 7.1) 2.27 ( <i>t</i> , 15.2)	19.5
10	3.37 ( <i>m</i> )	46.3	3.34 ( <i>dd</i> , 15.2, 7.1)	46.2
11		94.9		92.4
12	1.28 ( <i>s</i> )	22.7	1.23 ( <i>s</i> )	22.6
13	1.60 ( <i>s</i> )	28.1	1.58 ( <i>s</i> )	27.9
14	6.67 ( <i>d</i> , 10.0)	115.9	6.57 ( <i>d</i> , 10.0)	115.1
15	5.56 ( <i>d</i> , 10.0)	128.0	5.74 ( <i>d</i> , 10.0)	128.5
16		77.9		77.5
17	1.40 ( <i>s</i> )	28.2	1.39 ( <i>s</i> )	27.8
18	1.40 ( <i>s</i> )	28.2	1.38 ( <i>s</i> )	27.7
19	3.37 ( <i>m</i> )	21.3	3.55 ( <i>dd</i> , 13.8, 8.0) 3.35 (partly obscured)	20.9
20	5.00 ( <i>m</i> )	121.0	5.26 ( <i>t</i> , 7.0)	122.3
21		138.0		134.1
22	1.74 ( <i>s</i> )	16.6	1.79 ( <i>s</i> )	16.1
23	1.95 ( <i>m</i> )	39.5	1.97 ( <i>m</i> )	39.3
24	2.06 ( <i>m</i> )	26.4	1.88 ( <i>m</i> )	26.1
25	4.99 ( <i>m</i> )	124.2	4.98 ( <i>t</i> , 7.3)	124.1
26		131.5		130.6
27	1.55 ( <i>s</i> )	25.6	1.53 ( <i>s</i> )	25.4
28	1.48 ( <i>s</i> )	17.7	1.45 ( <i>s</i> )	17.5
1'		103.4		103.1
2'		149.8		151.1
3'	6.26 ( <i>s</i> )	104.6	6.28 ( <i>s</i> )	104.0
4'		145.1		140.5
5'		138.0		136.2

**Table 59** (Continued)

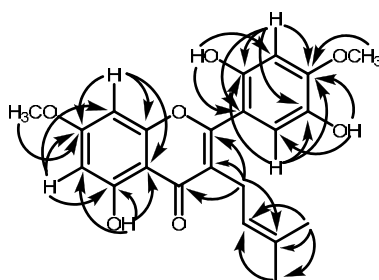
position	<b>AI1</b>		Artoindonesianin A <sup>a</sup>	
	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$
6'		131.2		132.3
5-OH	13.2 ( <i>s</i> )		13.7 ( <i>s</i> )	
2'-OH	6.61 ( <i>s</i> )		9.83 ( <i>s</i> )	

<sup>a</sup> recorded in DMSO-*d*<sub>6</sub>

### 3.3.1.2 Compound AI2



**AI2** was isolated as yellow powder. The UV (**Figure 120**) and IR (**Figure 121**) spectra were similar to those of **AI1**. The  $^1\text{H}$  NMR (**Table 60**, **Figure 122**) of **AI2** disclosed the presence of *meta*-coupled aromatic proton signals at  $\delta$  6.36 and 6.35 (*d*,  $J = 2.4$  Hz) for the proton H-6 and H-8, respectively, two *singlets* at  $\delta$  6.57 and 6.89 were assigned for H-3' and H-6', respectively, of ring B of a flavones which was 1,2,4,5-tetrasubstituted benzene ring. A down field signal at  $\delta$  13.20 indicated a chelated hydroxyl group. A set of signals was assigned to an isoprenyl side chain [ $\delta$  1.67 (*s*), 1.52 (*s*), 3.15 (*d*, 6.8 Hz) and 5.19 (*m*)]. In addition, two *singlets* at  $\delta$  3.84 and 3.94 were attributed to two methoxyl groups at C-7 and C-4, respectively due to the HMBC correlations of the former with the carbon at  $\delta$  165.5 (C-7) and the latter with the carbon at  $\delta$  149.4 (C-4'). Two *broad singlets* resonating at  $\delta$  5.30 and 5.32 were assigned for the additional hydroxyl groups in ring B. The HMBC correlations also showed connectivities between methylene protons at  $\delta$  3.15 (H<sub>2</sub>-9) and the carbons at  $\delta$  157.8 (C-2), 121.4 (C-3) and 182.1 (C-4), confirming the position of the isoprenyl group at C-3. Accordingly, **AI2** was characterized as Artoindonesianin Q (Syah *et al.*, 2002).



Selected HMBC correlations of **AI2**

**Table 60**  $^1\text{H}$ ,  $^{13}\text{C}$  NMR, DEPT and HMBC spectral data of **AI2**

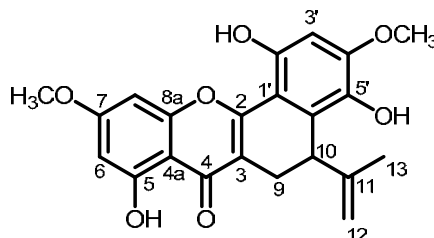
position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	DEPT	HMBC
2		157.8	C	
3		121.4	C	
4		182.1	C	
4a		105.0	C	
5		162.1	C	
6	6.36 ( <i>d</i> , 2.4)	98.1	CH	4a, 5, 7, 8
7		165.5	C	
8	6.35 ( <i>d</i> , 2.4)	92.0	CH	4a, 6, 7, 8a
8a		157.8	C	
9	3.15 ( <i>d</i> , 6.8)	24.4	CH <sub>2</sub>	2, 3, 4, 10, 11
10	5.19 ( <i>br m</i> )	120.6	CH	12, 13
11		133.8	C	
12	1.67 ( <i>s</i> )	25.7	CH <sub>2</sub>	10, 11, 13
13	1.52 ( <i>s</i> )	17.7	CH <sub>3</sub>	10, 11, 12
1'		111.3	C	
2'		147.6	C	
3'	6.57 ( <i>s</i> )	100.4	CH	1', 2', 4', 5'
4'		149.4	C	
5'		139.5	C	
6'	6.89 ( <i>s</i> )	114.8	C	2', 4', 5'
5-OH	12.80 ( <i>s</i> )			4a, 5, 6
7-OMe	3.84 ( <i>s</i> )	55.8	CH <sub>3</sub>	7
2'-OH	5.32 ( <i>s</i> )			3'
4'-OMe	3.94 ( <i>s</i> )	56.1	CH <sub>3</sub>	4'
5'-OH	5.30 ( <i>s</i> )			4', 5', 6'

**Table 61** Comparison of  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectral data of **AI2** and Artoindonesianin Q

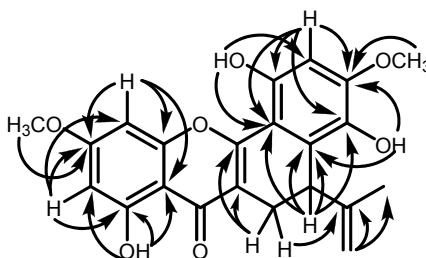
position	AI2		Artoindonesianin Q <sup>a</sup>	
	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$
2		157.8		162.0
3		121.4		121.9
4		182.1		183.1
4a		105.0		105.8
5		162.1		162.9
6	6.36 ( <i>d</i> , 2.4)	98.1	6.29 ( <i>d</i> , 2.3)	98.3
7		165.5		166.4
8	6.35 ( <i>d</i> , 2.4)	92.0	6.45 ( <i>d</i> , 2.3)	92.4
8a		157.8		159.1
9	3.15 ( <i>d</i> , 6.8)	24.4	3.13 ( <i>br d</i> , 7.1)	24.6
10	5.19 ( <i>br m</i> )	120.6	5.13 ( <i>t sept</i> , 7.1, 1.4)	122.4
11		133.8		132.2
12	1.67 ( <i>s</i> )	25.7	1.57 ( <i>s</i> )	25.8
13	1.52 ( <i>s</i> )	17.7	1.45 ( <i>s</i> )	17.6
1'		111.3		112.2
2'		147.6		149.2
3'	6.57 ( <i>s</i> )	100.4	6.67 ( <i>s</i> )	101.4
4'		149.4		151.1
5'		139.5		140.5
6'	6.89 ( <i>s</i> )	114.8	6.84 ( <i>s</i> )	116.5
5-OH	12.80 ( <i>s</i> )		13.10 ( <i>s</i> )	
7-OMe	3.84 ( <i>s</i> )	55.8	3.88 ( <i>s</i> )	56.3
2'-OH	5.32 ( <i>s</i> )		8.29 ( <i>s</i> )	
4'-OMe	3.94 ( <i>s</i> )	56.1	3.87 ( <i>s</i> )	56.2
5'-OH	5.30 ( <i>s</i> )		7.41 ( <i>s</i> )	

<sup>a</sup> recorded in acetone- $d_6$

### 3.3.1.3 Compound AI3



**AI3** was isolated as a yellow solid. The UV (**Figure 124**) and IR (**Figure 125**) spectra were similar to those of **AI2**. The NMR (**Table 62**, **Figures 16** and **127**) data were comparable to those of **AI2**. The differences were shown in ring B and the isoprenyl side chain of **AI2**. In ring B of **AI3**, only a *singlet* aromatic proton was shown at  $\delta$  6.47 (H-3') corresponding to 1,2,4,5,6-pentasubstituted benzene instead of two *singlet* aromatic protons H-3' and H-6' of **AI2**. An isoprenyl group in **AI2** was replaced by the set of signals at  $\delta$  1.82 (s), 2.55 (*dd*,  $J = 16.0, 6.8$  Hz), 3.41 (*dd*,  $J = 16.0, 1.2$  Hz), 4.00 (*br d*,  $J = 6.8$  Hz), 4.30 (*br d*,  $J = 1.2$  Hz) and 4.71 (*br d*,  $J = 1.2$  Hz), assignable to a  $-\text{CH}_2-\text{CH}-\text{C}(\text{CH}_3)=\text{CH}_2$  in **AI3**.  $^3J$  HMBC correlation between a methine proton at  $\delta$  4.00 (H-10) and C-1' (105.1), C-5' (136.4) and C-6' (126.0) of ring B established their fusion at C-10 and C-6' to form dihydrobenzoxanthone-type flavones. Therefore, **AI3** was identified as Artoindonesianin S (Syah *et al.*, 2002).



Selected HMBC correlations of **AI3**

**Table 62**  $^1\text{H}$ ,  $^{13}\text{C}$  NMR, DEPT and HMBC spectral data of **AI3**

position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	DEPT	HMBC
2		159.7	C	
3		111.8	C	
4		180.2	C	
4a		105.0	C	
5		162.3	C	
6	6.39 ( <i>d</i> , 2.4)	98.2	CH	4a, 5, 7, 8
7		165.1	C	
8	6.38 ( <i>d</i> , 2.4)	92.2	CH	4a, 6, 7, 8a
8a		155.7	C	
9	2.55 ( <i>dd</i> , 16.0, 6.8) 3.41 ( <i>dd</i> , 16.0, 1.2)	21.7	CH <sub>2</sub>	2, 3, 4, 10, 11, 6'
10	4.00 ( <i>br d</i> , 6.8)	36.5	CH	3, 9, 11, 12, 1', 5', 6'
11		144.4	C	
12	4.30 ( <i>br d</i> , 1.2) 4.71 ( <i>br d</i> , 1.2)	111.7	CH <sub>2</sub>	10, 11, 13
13	1.82 ( <i>s</i> )	21.7	CH <sub>3</sub>	10, 11, 12
1'		105.1	C	
2'		150.0	C	
3'	6.47 ( <i>s</i> )	99.1	CH	1', 2', 5'
4'		150.8	C	
5'		136.4	C	
6'		126.0	C	
5-OH	13.0 ( <i>s</i> )			4a, 5, 6
7-OMe	3.87 ( <i>s</i> )	55.9	CH <sub>3</sub>	7
2'-OH	7.66 ( <i>s</i> )			1', 2', 3'
4'-OMe	3.95 ( <i>s</i> )	56.2	CH <sub>3</sub>	4'
5'-OH	5.38 ( <i>s</i> )			4', 5', 6'

**Table 63** Comparison of  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectral data of **AI3** and Artoindonesianin S

position	AI3		Artoindonesianin S <sup>a</sup>	
	$\delta_{\text{H}}$ (mult., J <sub>Hz</sub> )	$\delta_{\text{C}}$	$\delta_{\text{H}}$ (mult., J <sub>Hz</sub> )	$\delta_{\text{C}}$
2		159.7		161.4
3		111.8		112.1
4		180.2		181.0
4a		105.0		105.5
5		162.3		162.8
6	6.39 ( <i>d</i> , 2.4)	98.2	6.30 ( <i>d</i> , 2.3)	98.6
7		165.1		166.0
8	6.38 ( <i>d</i> , 2.4)	92.2	6.69 ( <i>d</i> , 2.3)	93.1
8a		155.7		157.5
9	2.55 ( <i>dd</i> , 16.0, 6.8)	21.7	2.45 ( <i>dd</i> , 16.0, 6.6)	22.2
	3.41 ( <i>dd</i> , 16.0, 1.2)		3.40 ( <i>dd</i> , 16.0, 1.7)	
10	4.00 ( <i>br d</i> , 6.8)	36.5	4.17 ( <i>br d</i> , 6.8)	37.6
11		144.4		145.3
12	4.30 ( <i>br d</i> , 1.2)	111.7	4.27 ( <i>br s</i> )	111.7
	4.71 ( <i>br d</i> , 1.2)		4.64 ( <i>br s</i> )	
13	1.82 ( <i>s</i> )	21.7	1.77 ( <i>br m</i> )	21.9
1'		105.1		106.8
2'		150.0		151.0
3'	6.47 ( <i>s</i> )	99.1	6.56 ( <i>s</i> )	100.5
4'		150.8		152.6
5'		136.4		137.6
6'		126.0		127.9
5-OH	13.00 ( <i>s</i> )		13.18 ( <i>s</i> )	
7-OMe	3.87 ( <i>s</i> )	55.9	3.90 ( <i>s</i> )	56.3
2'-OH	7.66 ( <i>s</i> )		7.47 ( <i>s</i> )	

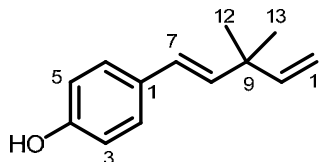


**Table 63** (Continued)

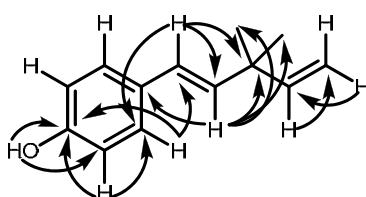
position	AI3		Artoindonesianin S <sup>a</sup>	
	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$
4'-OMe	3.95 ( <i>s</i> )	56.2	3.91 ( <i>s</i> )	56.3
5'-OH	5.38 ( <i>s</i> )		8.20 ( <i>s</i> )	

<sup>a</sup> recorded in <sup>a</sup> recorded in acetone-*d*<sub>6</sub>

### 3.3.1.4 Compound AI4



**AI4** was isolated as yellow-brown viscous oil. The  $^1\text{H}$  NMR spectrum (Table 64, Figures 130 and 131) showed the presence of a *singlet* of two methyls at  $\delta$  1.20 (6H, *s*). Two terminal olefinic protons resonated as *doublet of doublet* at  $\delta$  4.97 (1H, *dd*,  $J = 10.4, 1.2$  Hz) and 5.01 (1H, *dd*,  $J = 17.6, 1.2$  Hz) and an olefinic proton as *doublet of doublet* at  $\delta$  5.90 (1H, *dd*,  $J = 17.6, 10.4$  Hz) could be assigned to an ABC pattern (-CH=CH<sub>2</sub>). Two *doublets* of two olefinic methine protons resonating at  $\delta$  6.06 and 6.26 (each 1H, *d*,  $J = 16.4$  Hz) was assigned to a *trans* double bond. Two *doublets* in a AA'BB' pattern resonating at  $\delta$  6.77 (2H, *d*,  $J = 8.4$  Hz) and 7.25 (2H, *d*,  $J = 8.4$  Hz), were assigned to a *p*-disubstituted benzene ring. The *singlet* at  $\delta$  4.82 (1H, *br s*) could be assigned to a phenolic hydroxyl group which was placed at C-4 because of its HMBC correlations to C-3 ( $\delta$  115.4), C-4 ( $\delta$  154.6) and C-5 ( $\delta$  115.4). Accordingly, **AI4** was characterized as corylifolin (Sun *et al.*, 1998).



Selected HMBC correlations of **AI4**

**Table 64**  $^1\text{H}$ ,  $^{13}\text{C}$  NMR, DEPT and HMBC spectral data of **AI4**

position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	DEPT	HMBC
1		130.8	C	
2	7.25 ( <i>d</i> , 8.4)	127.4	CH	4, 6, 7
3	6.77 ( <i>d</i> , 8.4)	115.4	CH	1, 4, 5
4		154.6	C	
5	6.77 ( <i>d</i> , 8.4)	115.4	CH	1, 4, 5
6	7.25 ( <i>d</i> , 8.4)	127.4	CH	4, 6, 7
7	6.26 ( <i>d</i> , 16.4)	125.6	CH	2, 6, 8, 9
8	6.06 ( <i>d</i> , 16.4)	136.9	CH	1, 7, 9, 12, 13
9		39.3	C	
10	5.90 ( <i>d</i> , 17.6, 10.4)	147.1	CH	8, 9, 12, 13
11	4.97 ( <i>dd</i> , 10.8, 1.2) 5.01 ( <i>dd</i> , 17.6, 1.2)	110.8	CH <sub>2</sub>	9, 10
12	1.20 ( <i>s</i> )	27.0	CH <sub>3</sub>	8, 9, 10
13	1.20 ( <i>s</i> )	27.0	CH <sub>3</sub>	8, 9, 10
4-OH	4.82 ( <i>s</i> )			3, 4, 5

**Table 65** Comparison of  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectral data of **AI4** and corylifolin <sup>a</sup>

position	<b>AI4</b>		<b>corylifolin</b> <sup>a</sup>	
	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$
1		130.8		131.8
2	7.25 ( <i>d</i> , 8.4)	127.4	7.23 ( <i>d</i> , 8.6)	128.0
3	6.77 ( <i>d</i> , 8.4)	115.4	6.76 ( <i>d</i> , 8.6)	115.8
4		154.6		153.1
5	6.77 ( <i>d</i> , 8.4)	115.4	6.76 ( <i>d</i> , 8.6)	115.8
6	7.25 ( <i>d</i> , 8.4)	127.4	7.23 ( <i>d</i> , 8.6)	128.0
7	6.26 ( <i>d</i> , 16.4)	125.6	6.27 ( <i>d</i> , 16.3)	135.6
8	6.06 ( <i>d</i> , 16.4)	136.9	6.08 ( <i>d</i> , 16.3)	127.6
9		39.3		43.0
10	5.90 ( <i>dd</i> , 17.6, 10.4)	147.1	5.90 ( <i>dd</i> , 17.4, 10.7)	147.3
11	4.97 ( <i>dd</i> , 10.8, 1.2)	110.8	5.00 ( <i>m</i> )	111.9
	5.01 ( <i>dd</i> , 17.6, 1.2)			
12	1.20 ( <i>s</i> )	27.0	1.17 ( <i>s</i> )	25.0
13	1.20 ( <i>s</i> )	27.0	1.09 ( <i>s</i> )	23.9
4-OH	4.82 ( <i>s</i> )		9.65 ( <i>br s</i> )	

<sup>a</sup> recorded in acetone- $d_6$

### 3.3.2 Biological activities of the isolated compounds from the roots of *A. integer*

The isolated compounds were evaluated for their antibacterial activity against both Gram-positive: *Bacillus subtilis*, *Staphylococcus aureus* and *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 subjected to antifungal assay against *Candida albicans*. The results are summarized in **Table 66**. Only compound **AI2** exhibited strong activity against Methicillin-Resistant *Staphylococcus aureus* (MRSA).

**Table 66** Antimicrobial activity of compounds isolated from the roots of *A. integer*

Compounds	Antibacterial activity, MIC ( $\mu\text{g}/\text{mL}$ )								Antifungal activity, MIC ( $\mu\text{g}/\text{mL}$ )
	Gram-positive bacteria					Gram-negative bacteria			
	<i>B. subtilis</i>	<i>S. aureus</i>	<i>E. faecalis</i>	MRSA	VRE	<i>S. typhi</i>	<i>S. sonnei</i>	<i>P. aeruginosa</i>	
<b>AI1</b>	>300	>300	>300	>300	>300	>300	>300	>300	>300
<b>AI2</b>	>300	75	>300	4.69	75	>300	>300	300	>300
<b>AI3</b>	37.5	300	300	75	150	>300	>300	>300	300
<b>AI4</b>	37.5	75	>300	37.5	75	>300	>300	>300	>300

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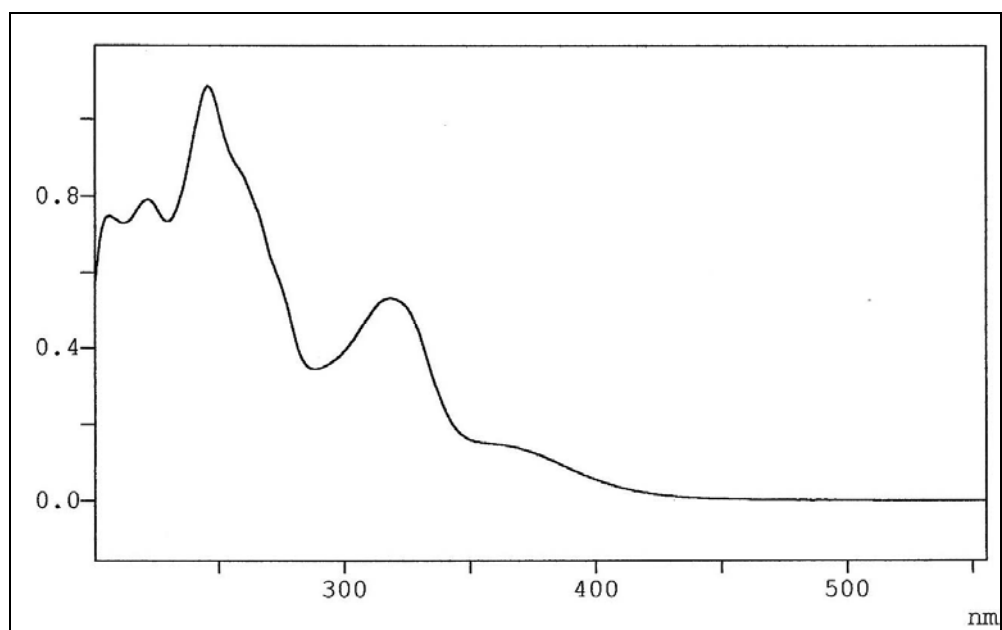
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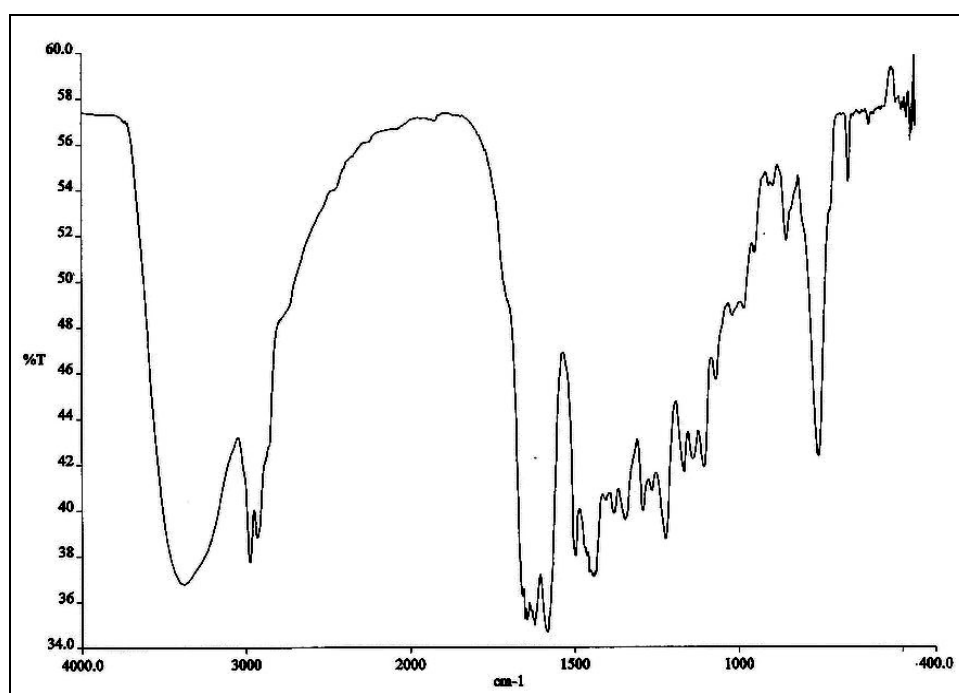
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**APPENDIX**

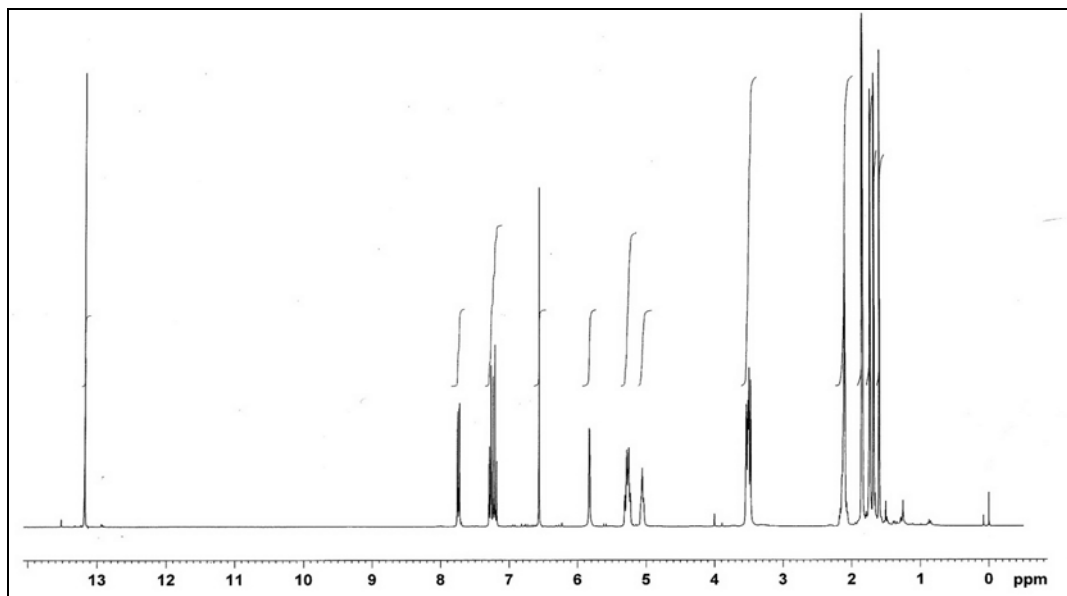


**Figure 5** UV (MeOH) spectrum of compound **CF1**

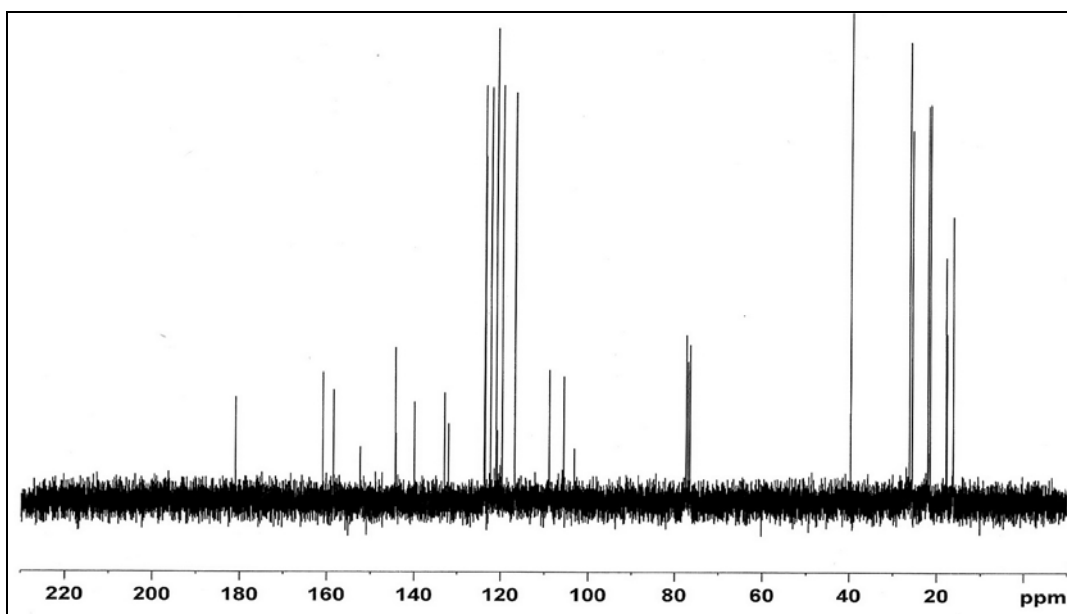


**Figure 6** IR (neat) spectrum of compound **CF1**

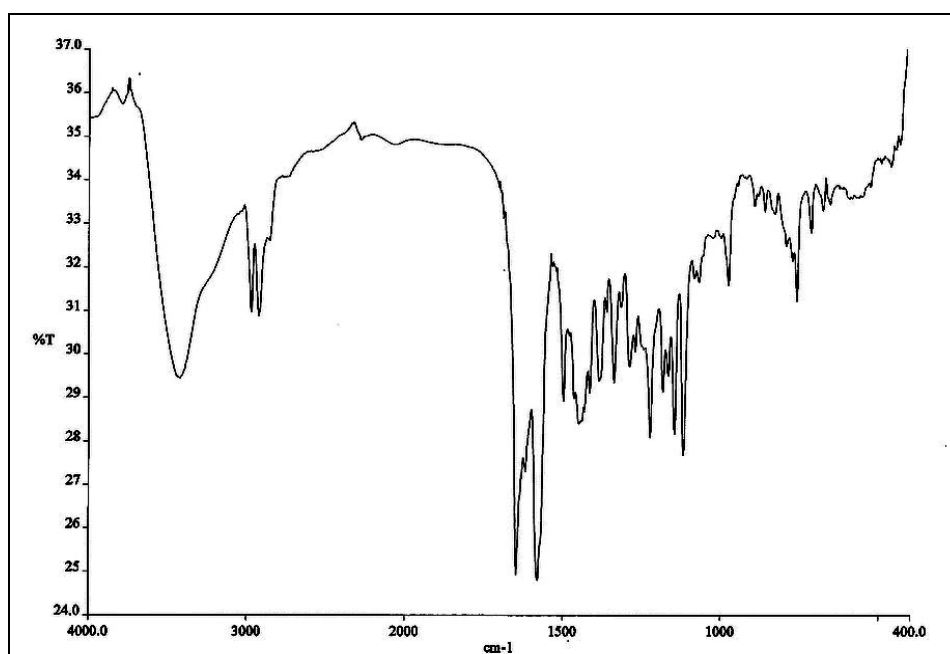




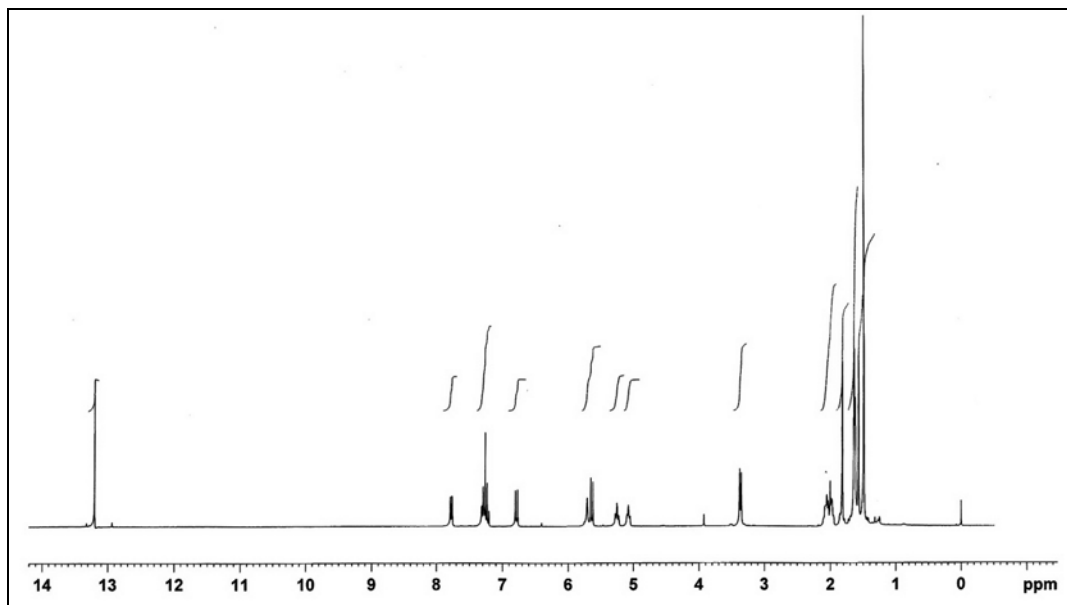
**Figure 7**  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **CF1**



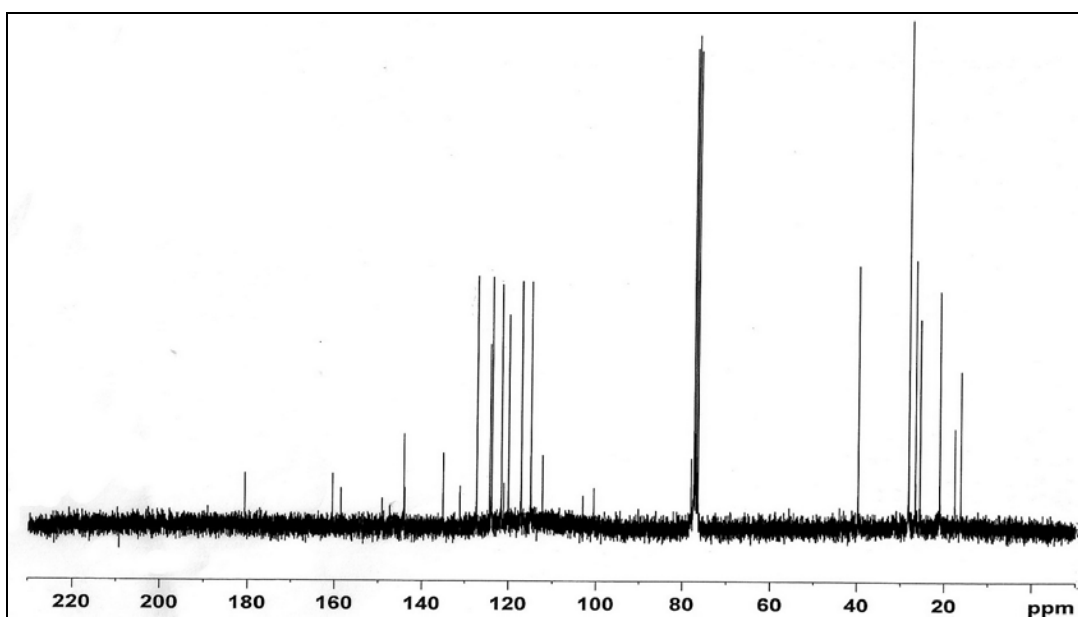
**Figure 8**  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **CF1**



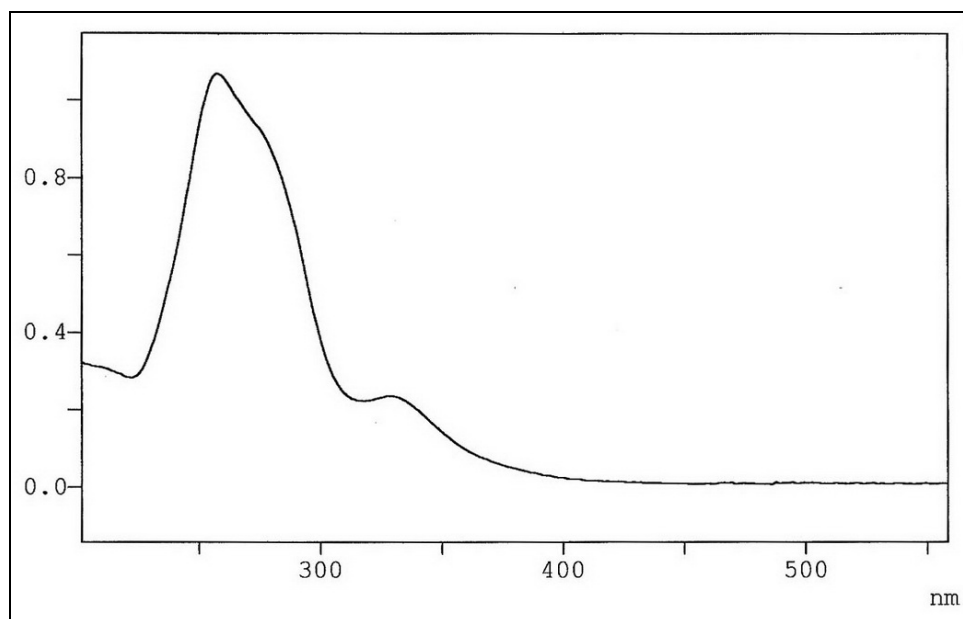
**Figure 9** IR (KBr) spectrum of compound **CF2**



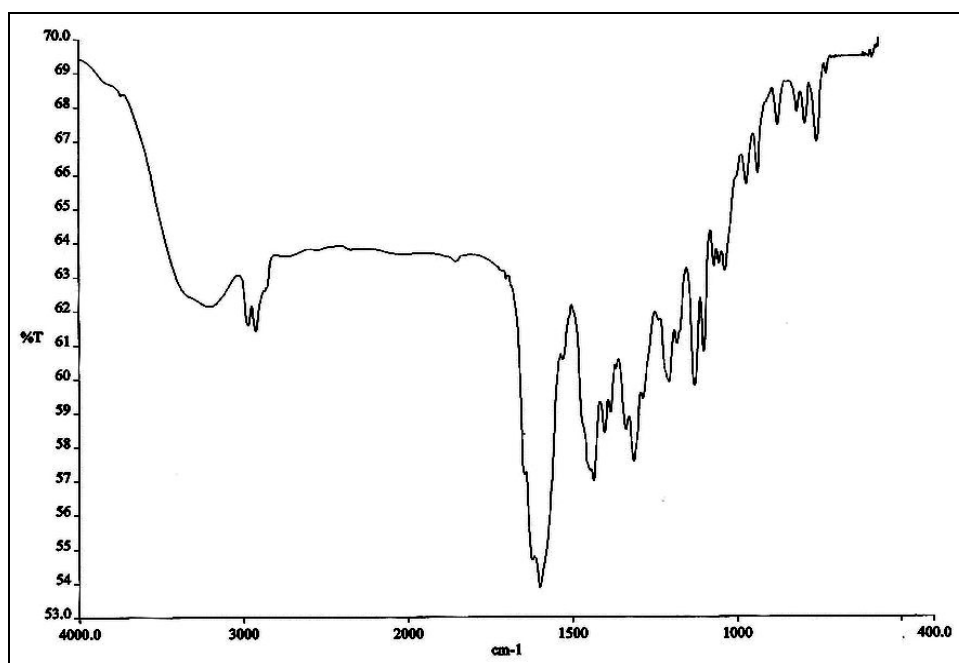
**Figure 10**  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **CF2**



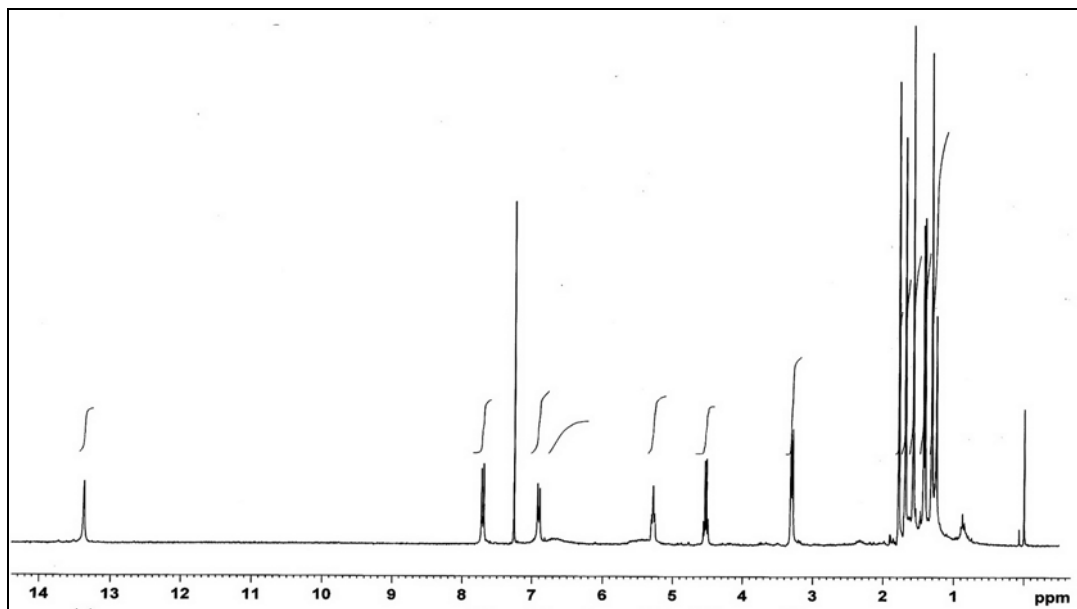
**Figure 11**  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **CF2**



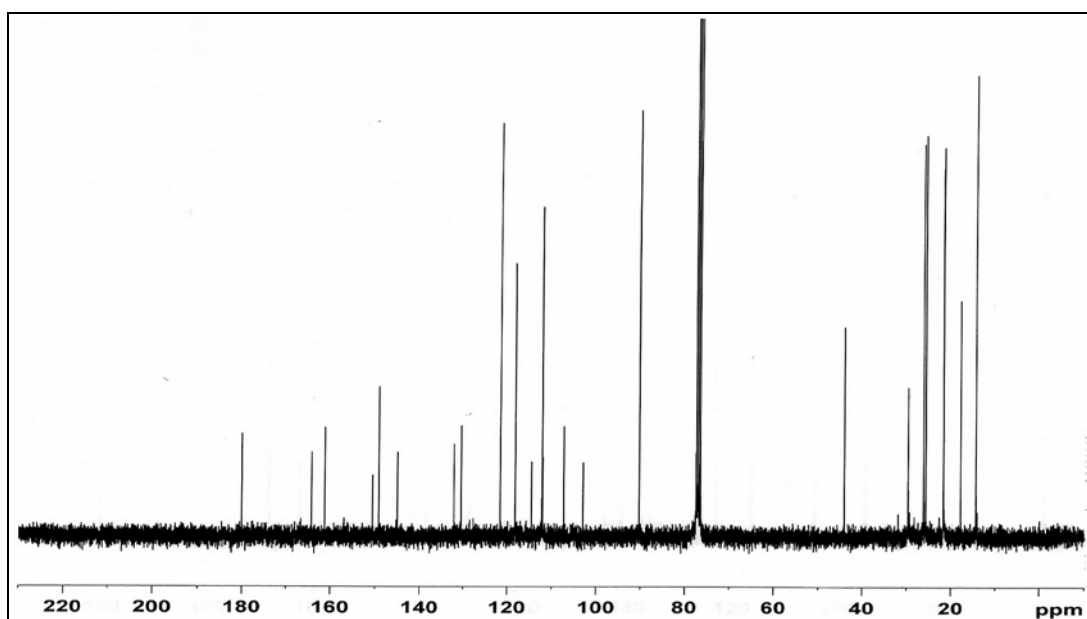
**Figure 12** UV (MeOH) spectrum of compound CF3



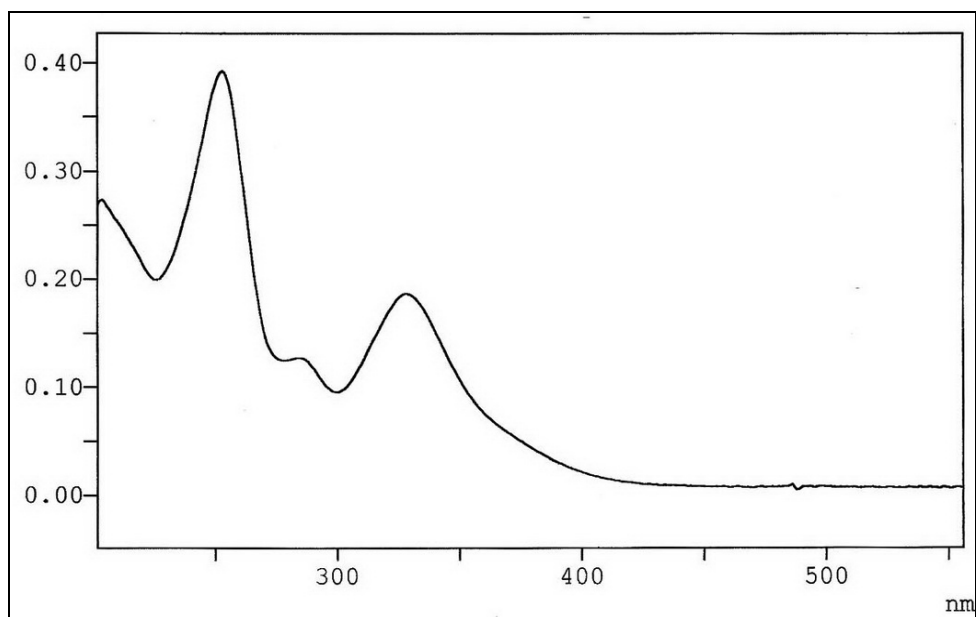
**Figure 13** IR (KBr) spectrum of compound CF3



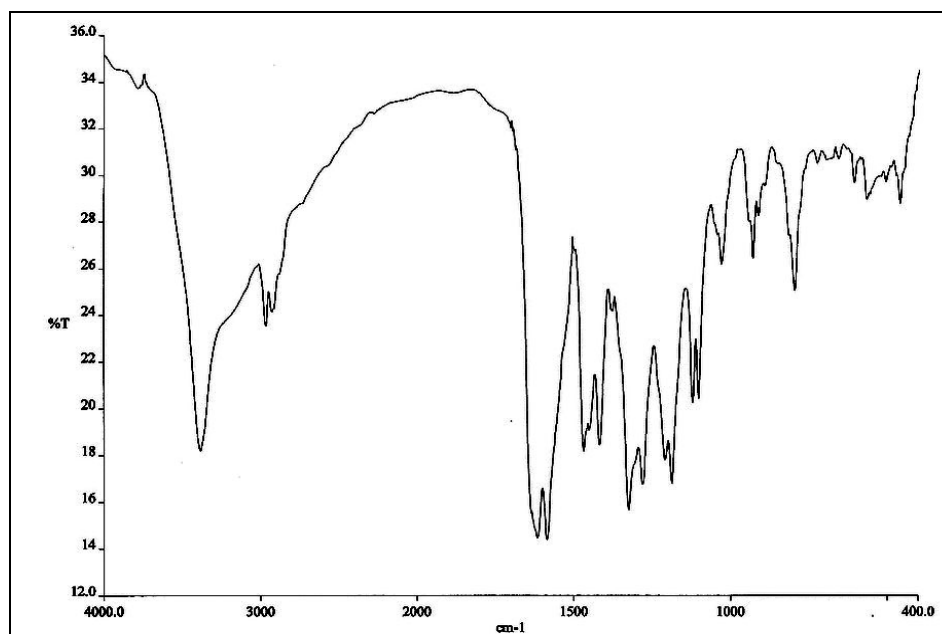
**Figure 14**  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **CF3**



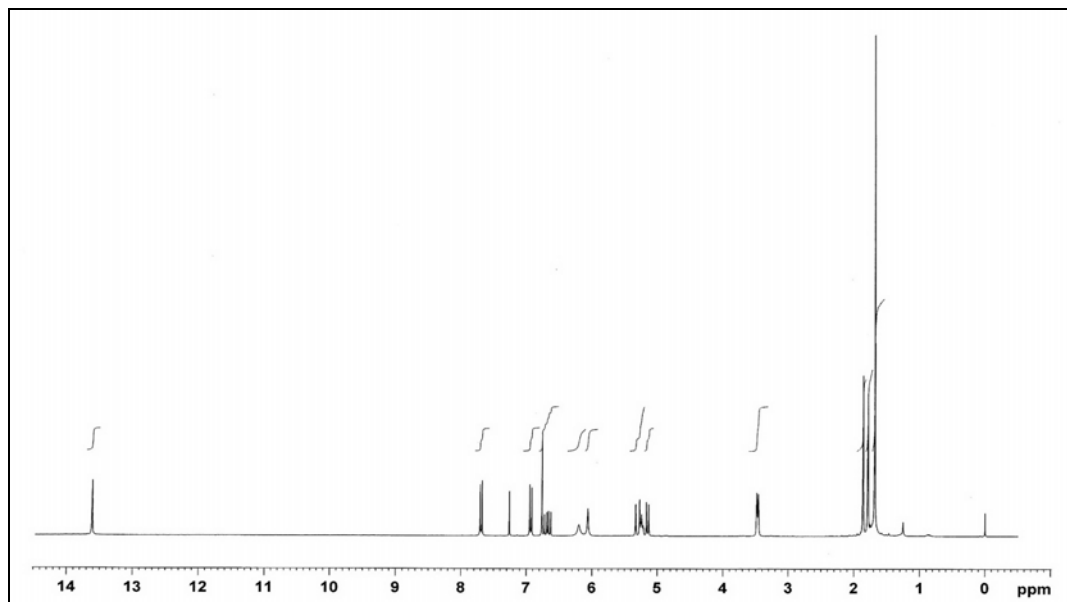
**Figure 15**  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **CF3**



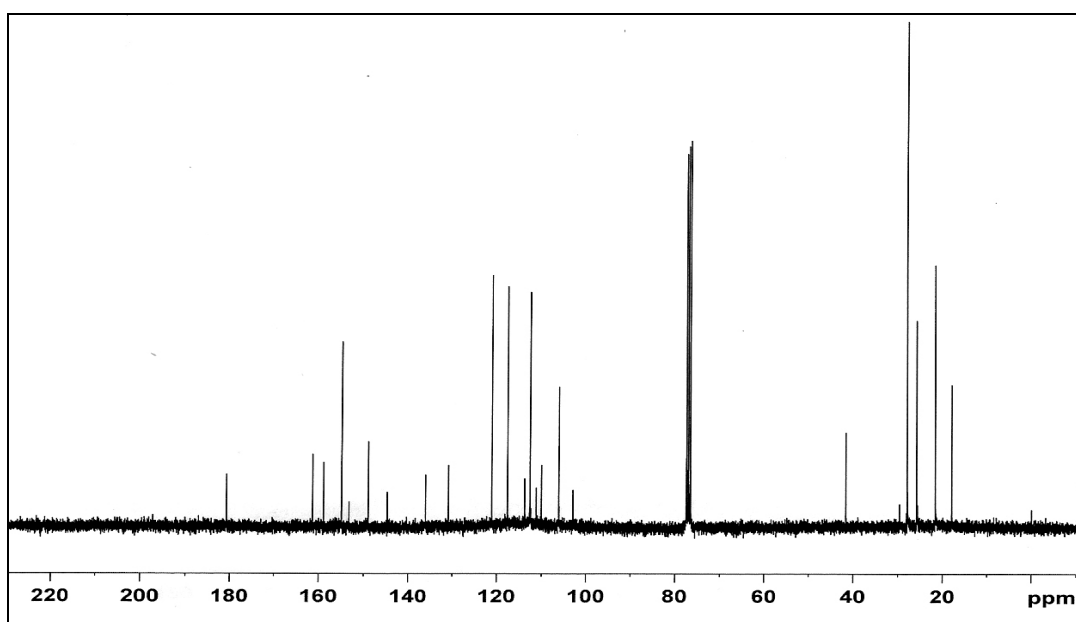
**Figure 16** UV (MeOH) spectrum of compound **CF4**



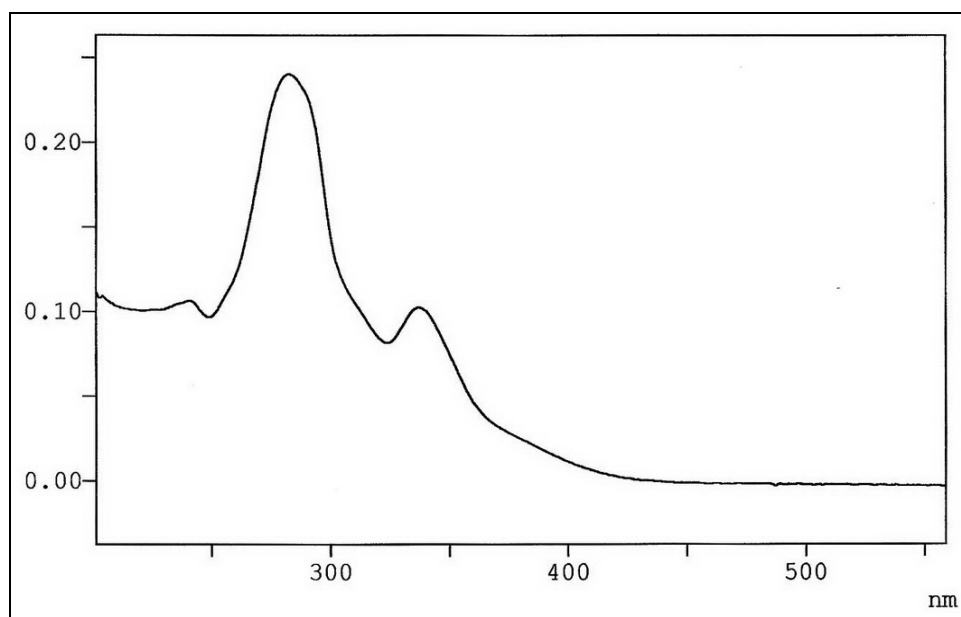
**Figure 17** IR (KBr) spectrum of compound **CF4**



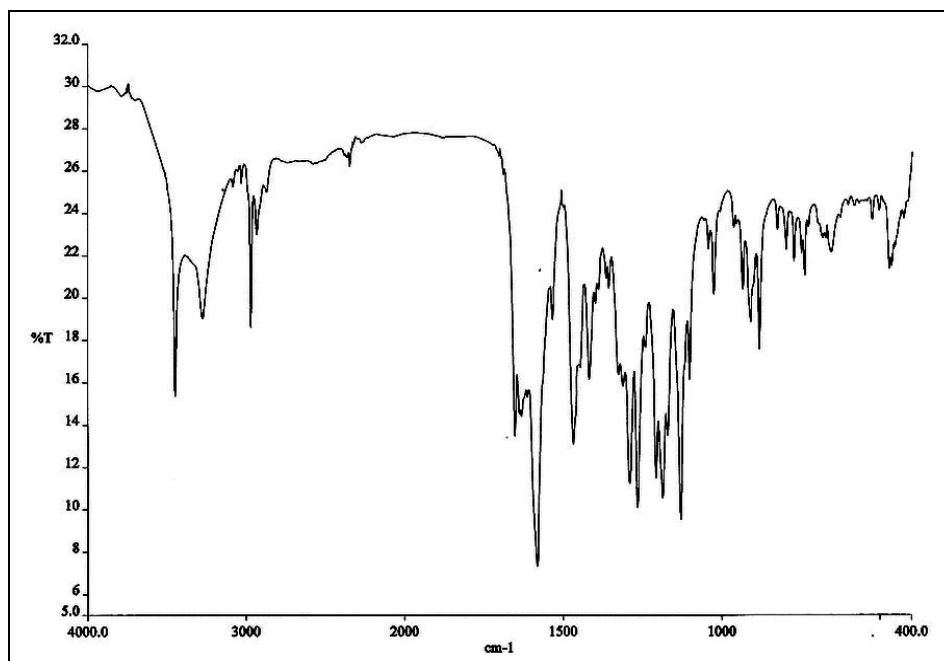
**Figure 18**  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **CF4**



**Figure 19**  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **CF4**

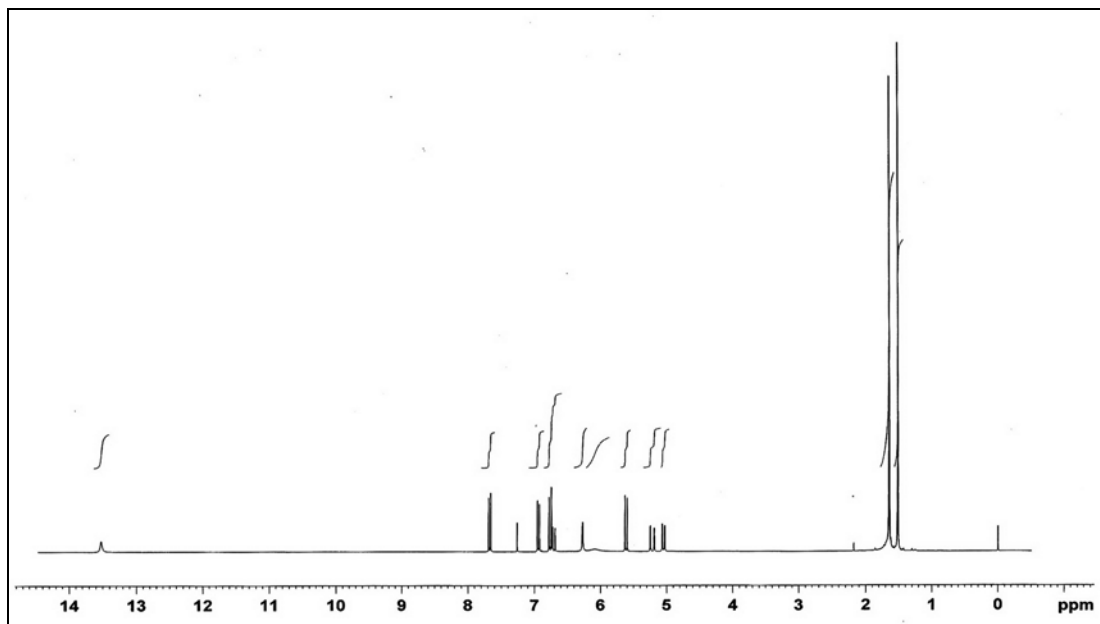


**Figure 20** UV (MeOH) spectrum of compound **CF5**

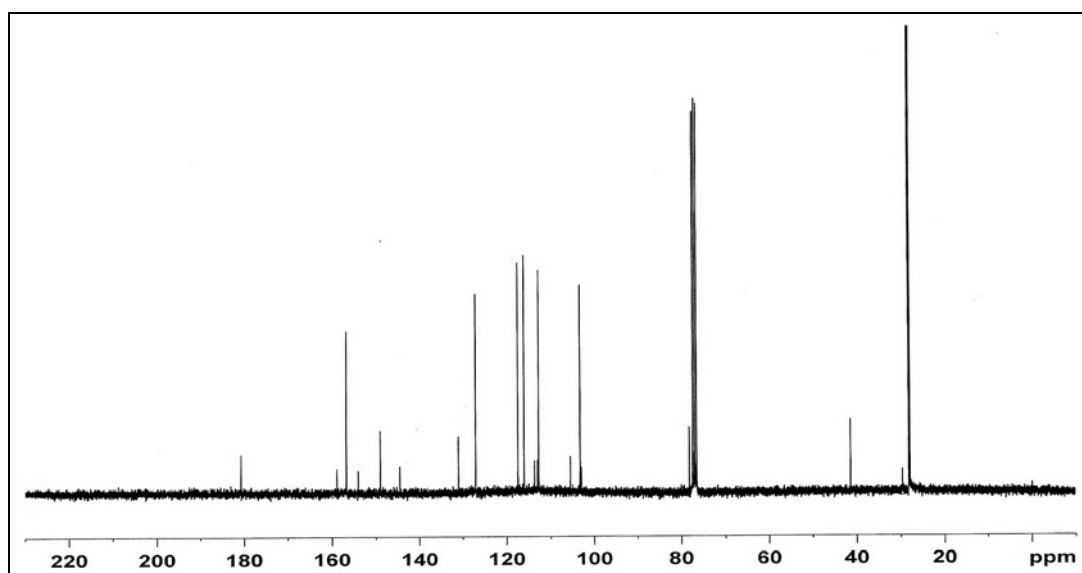


**Figure 21** IR (KBr) spectrum of compound **CF5**

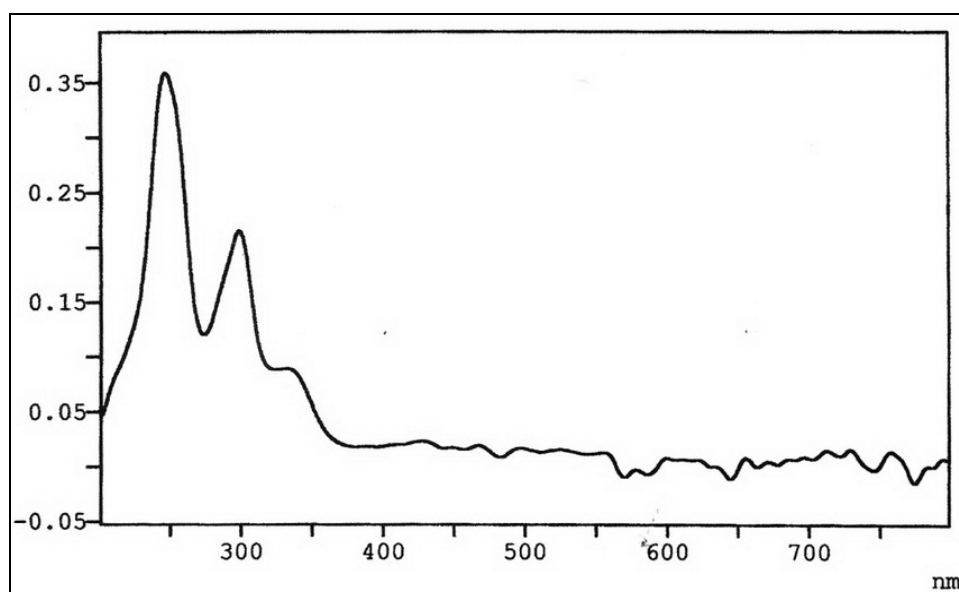




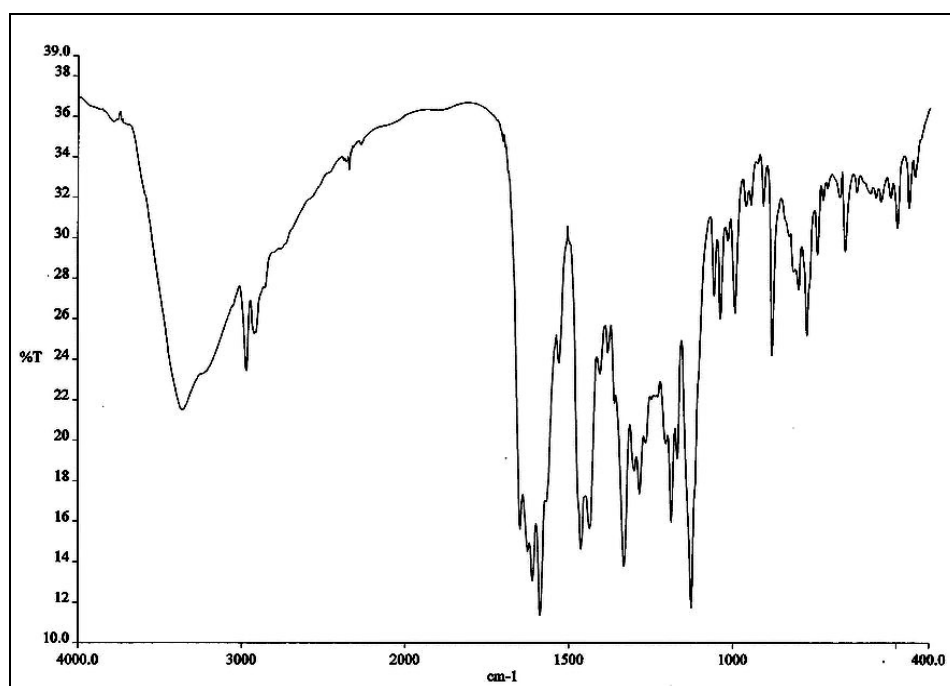
**Figure 22**  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound CF5



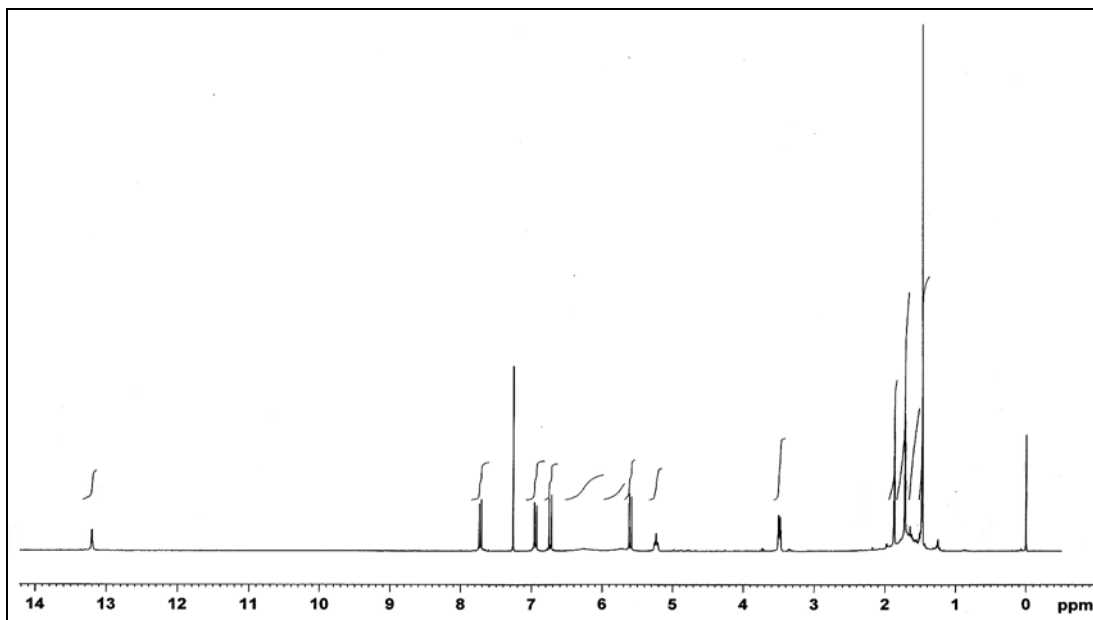
**Figure 23**  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound CF5



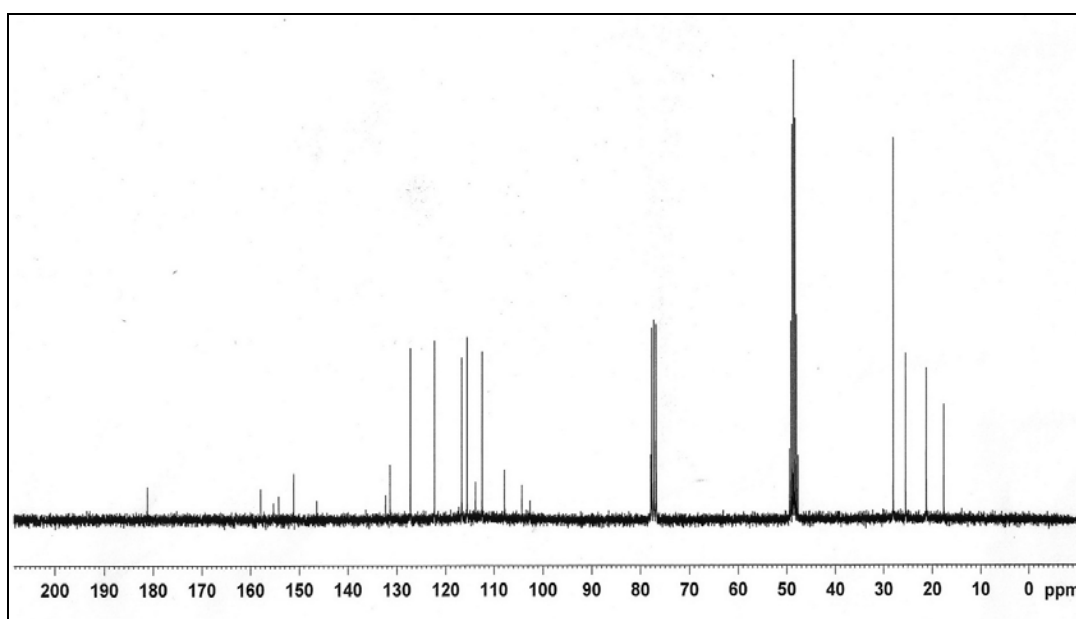
**Figure 24** UV (MeOH) spectrum of compound CF6



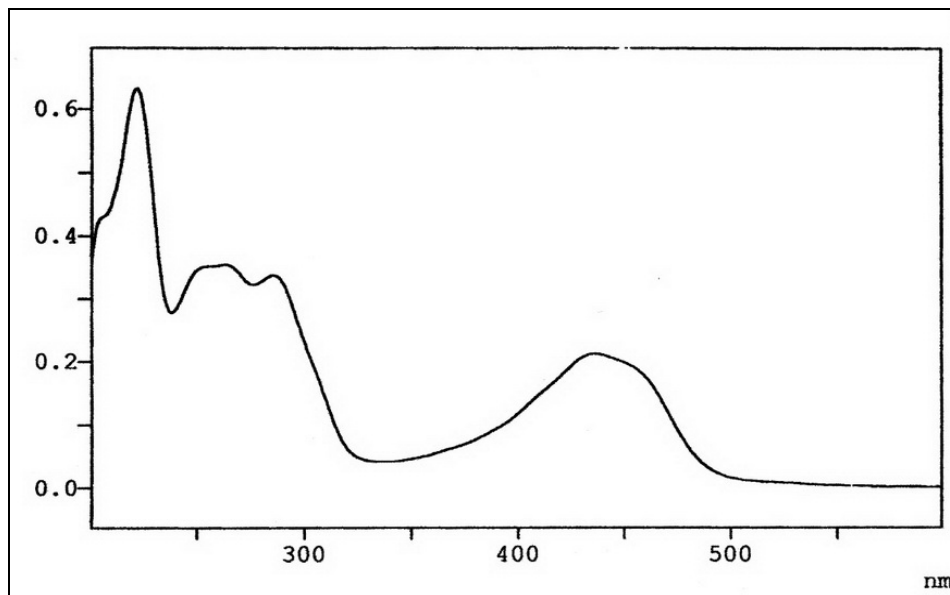
**Figure 25** IR (KBr) spectrum of compound CF6



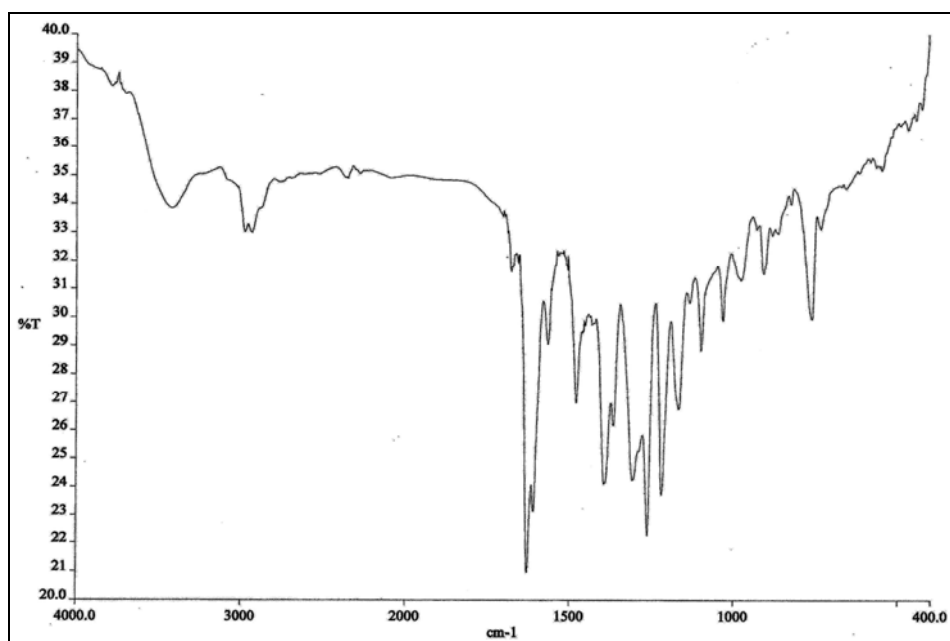
**Figure 26**  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **CF6**



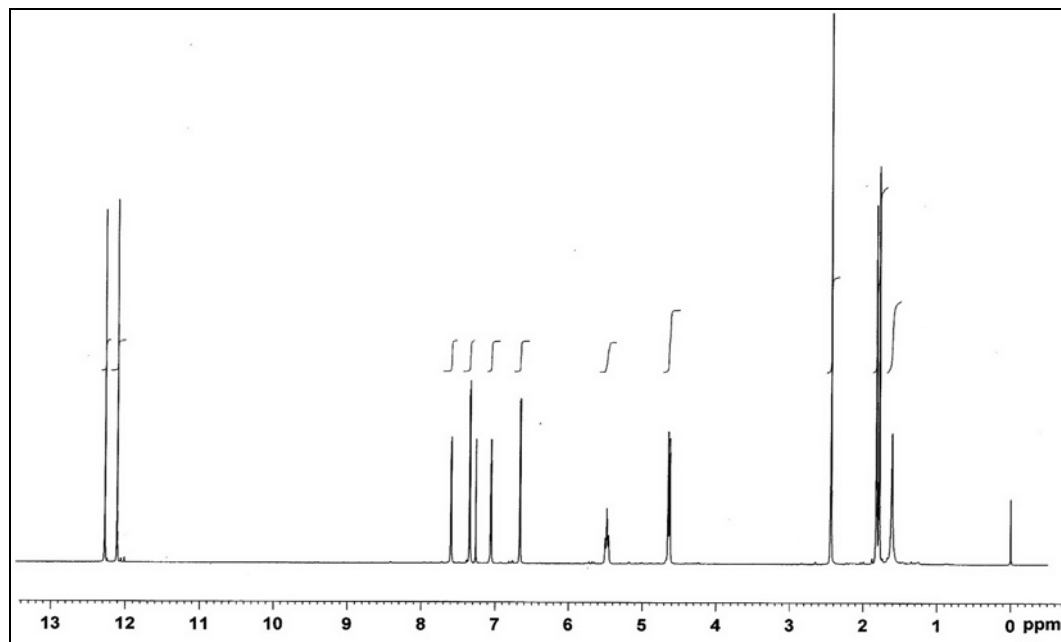
**Figure 27**  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **CF6**



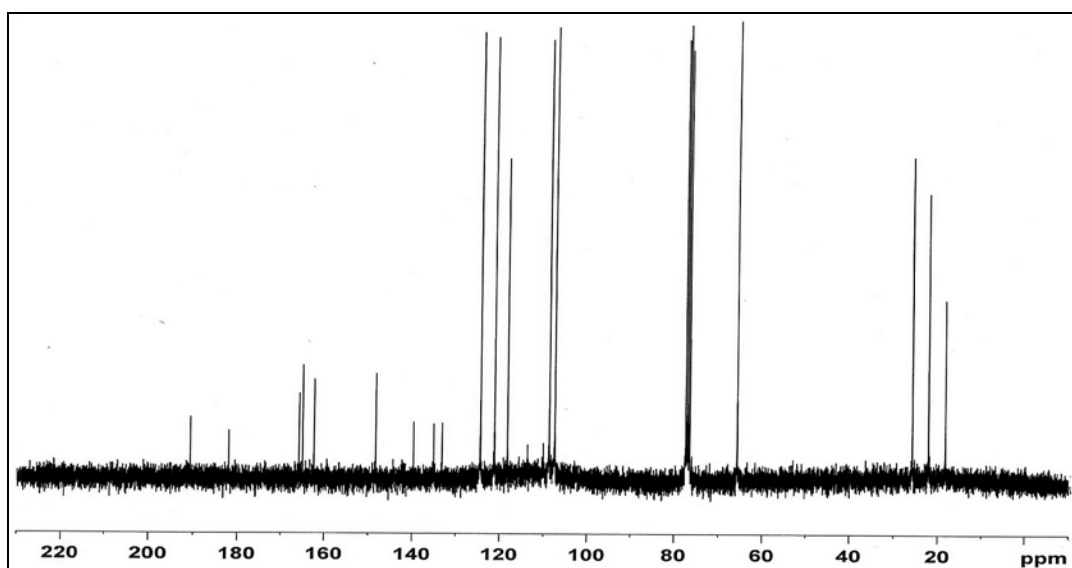
**Figure 28** UV (MeOH) spectrum of compound CF7



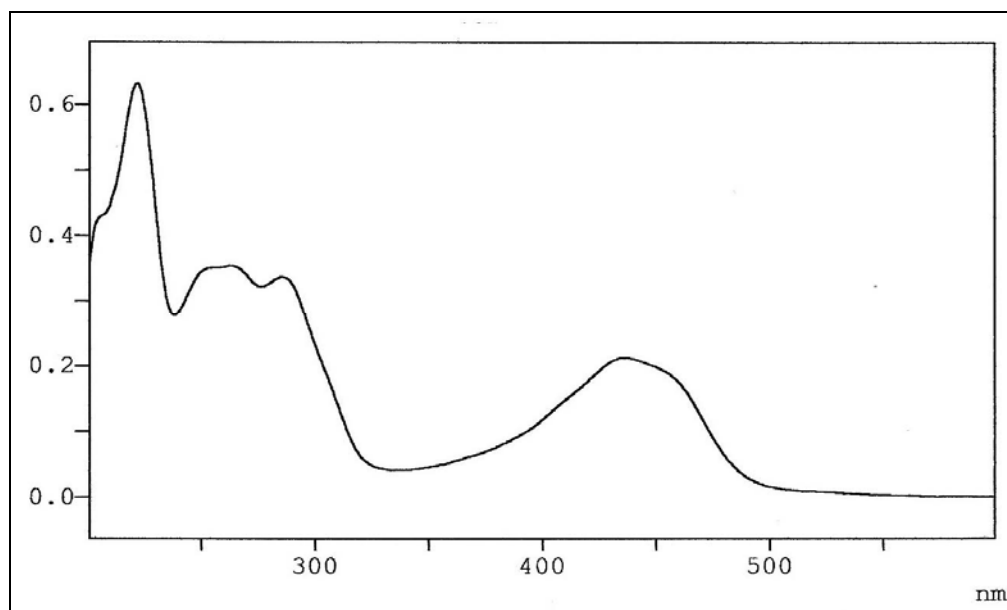
**Figure 29** IR (KBr) spectrum of compound CF7



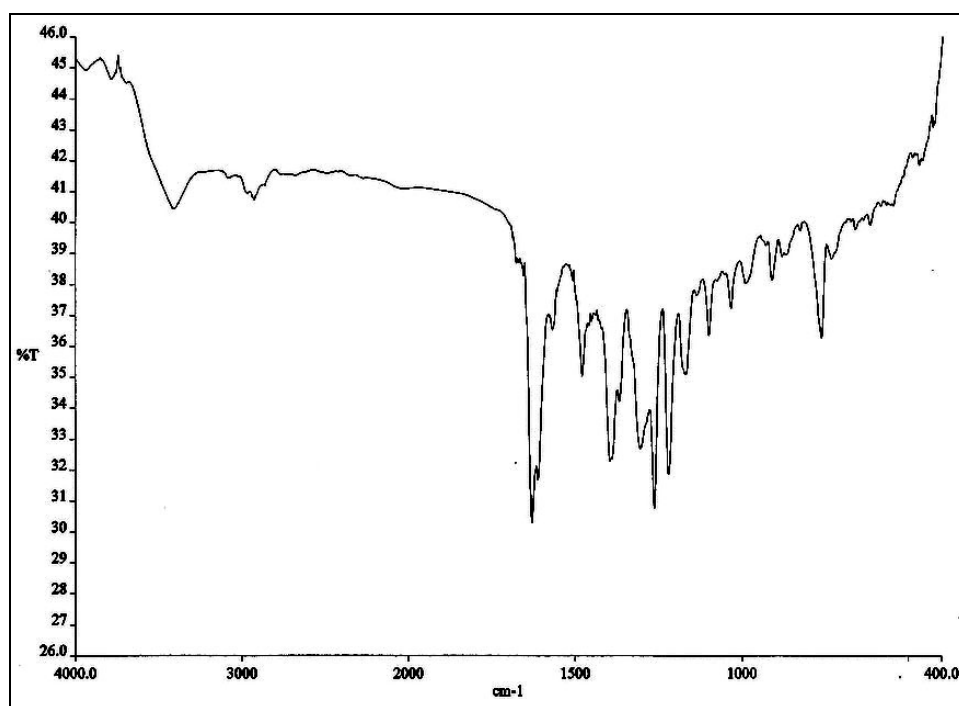
**Figure 30**  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound CF7



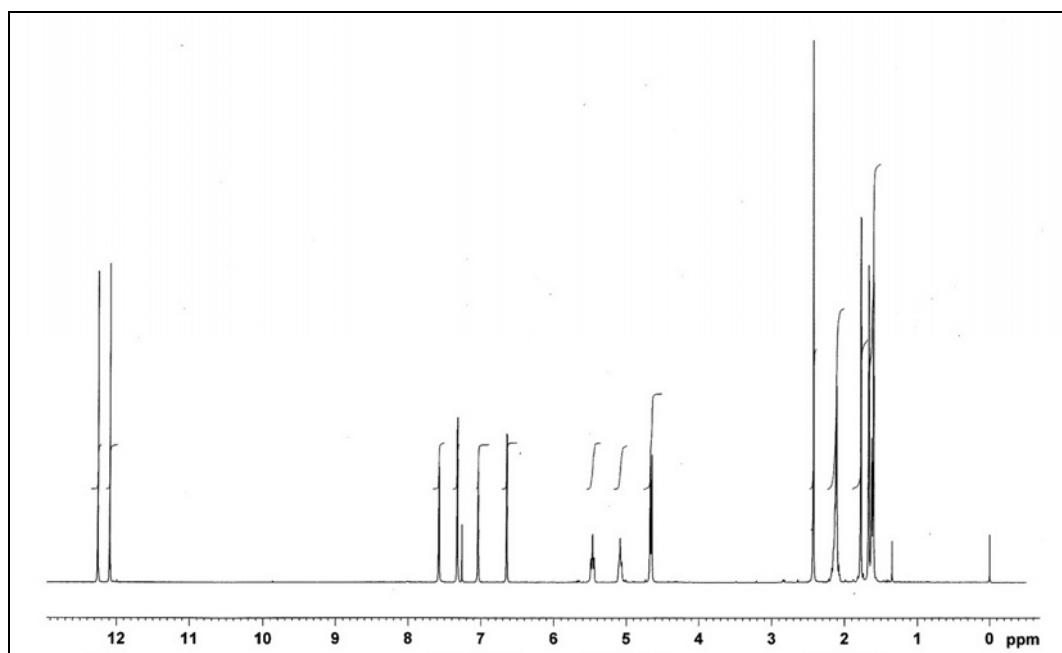
**Figure 31**  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound CF7



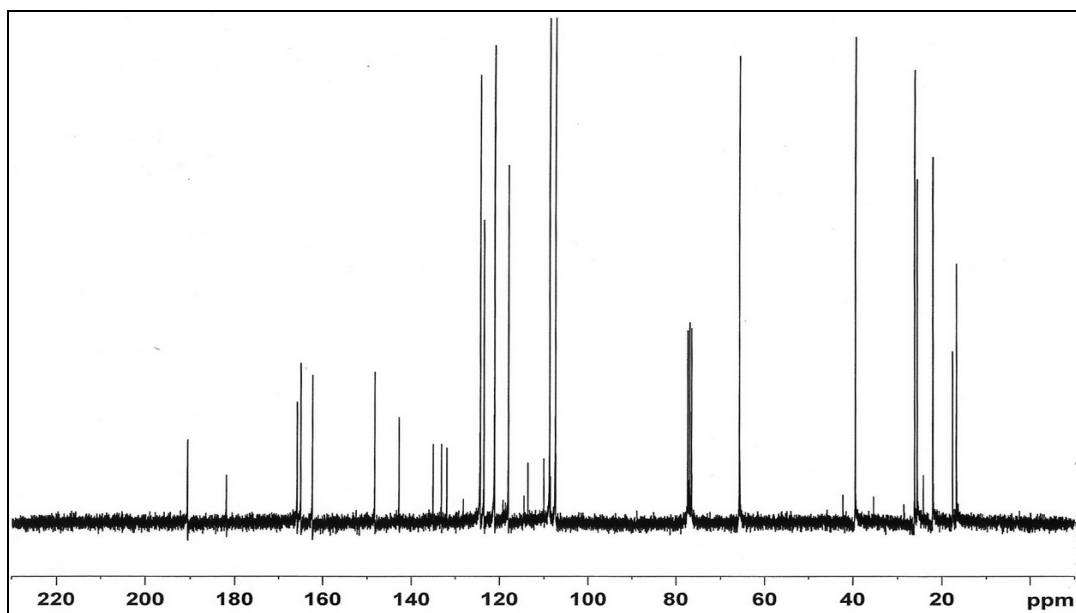
**Figure 32** UV (MeOH) spectrum of compound **CF8**



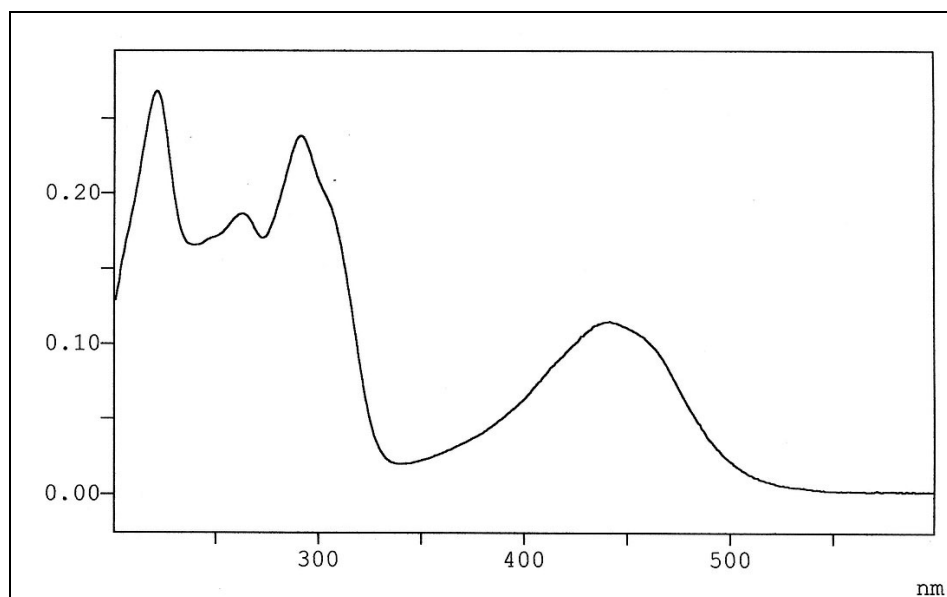
**Figure 33** IR (KBr) spectrum of compound **CF8**



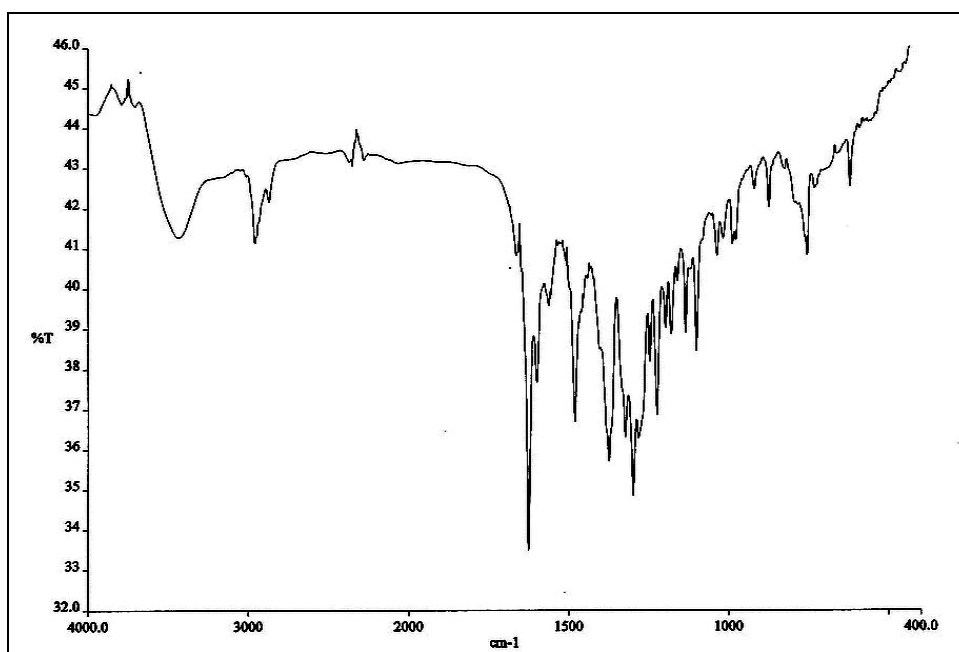
**Figure 34**  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **CF8**



**Figure 35**  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **CF8**

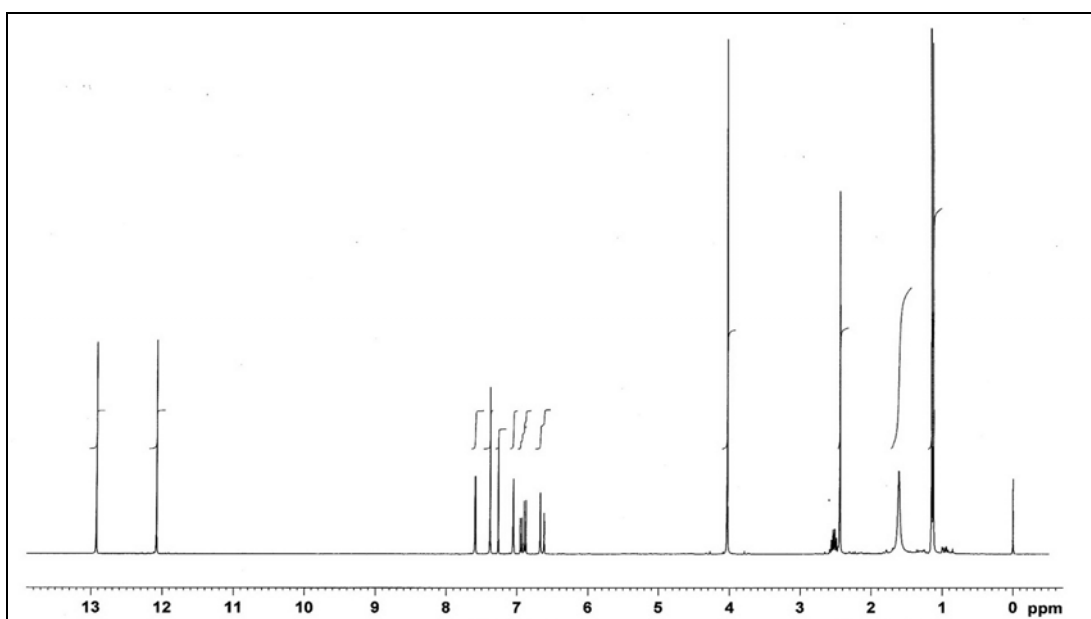


**Figure 36** UV (MeOH) spectrum of compound **CF9**

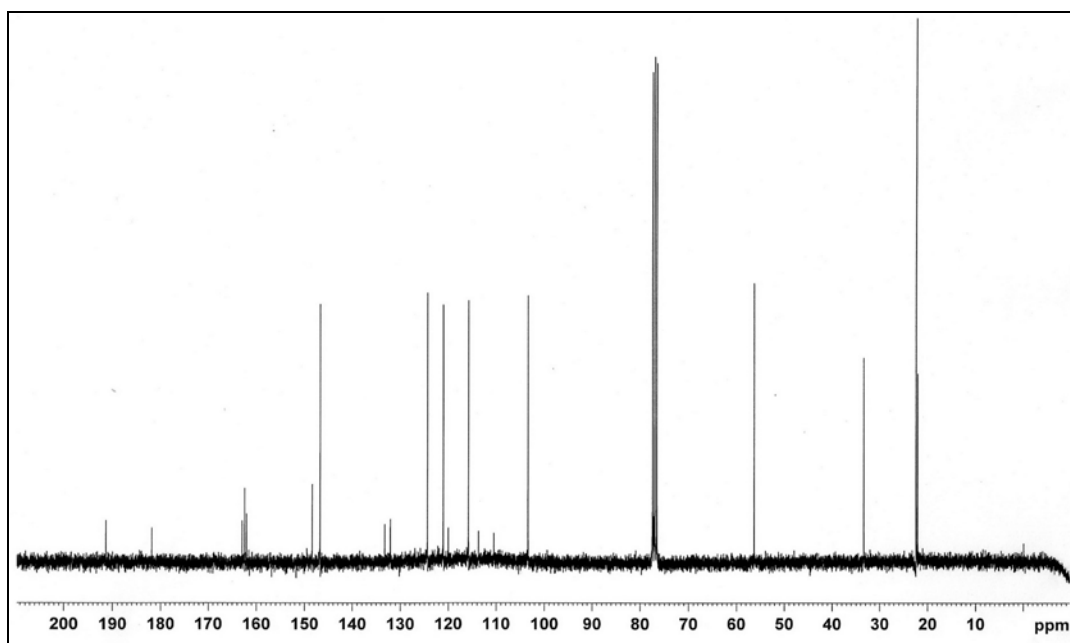


**Figure 37** IR (KBr) spectrum of compound **CF9**

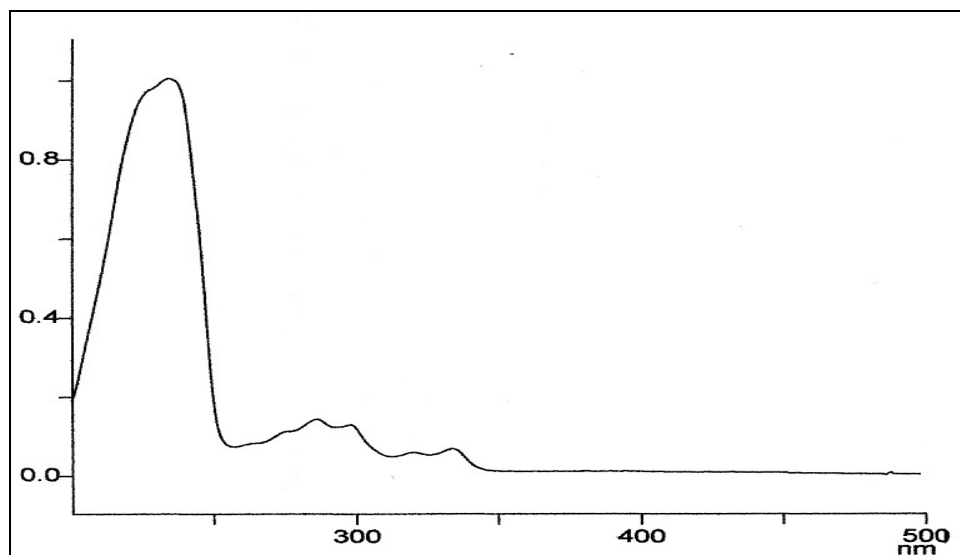




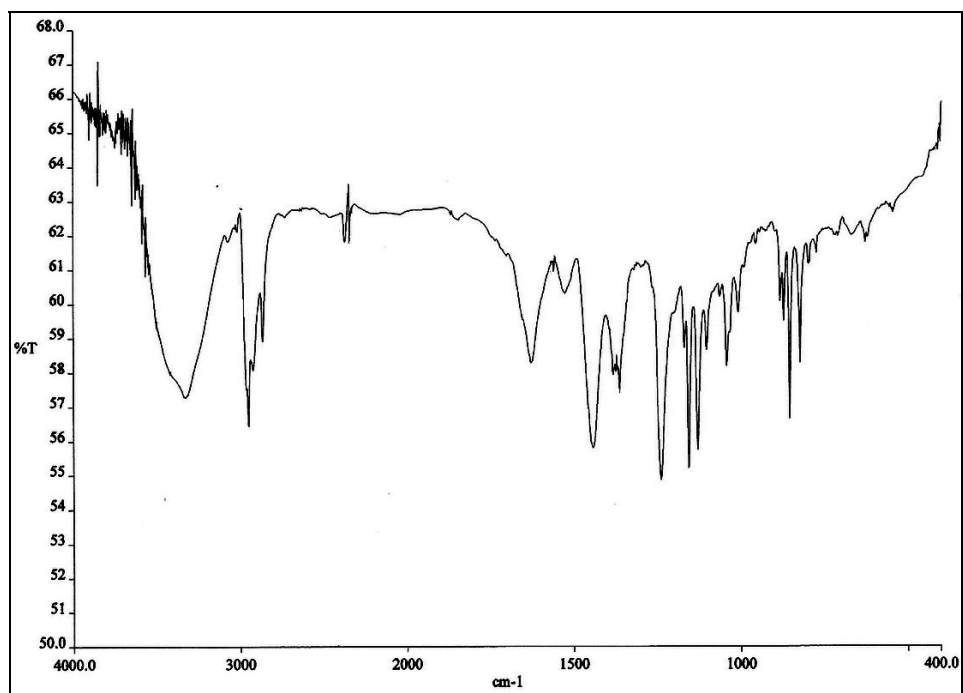
**Figure 38**  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **CF9**



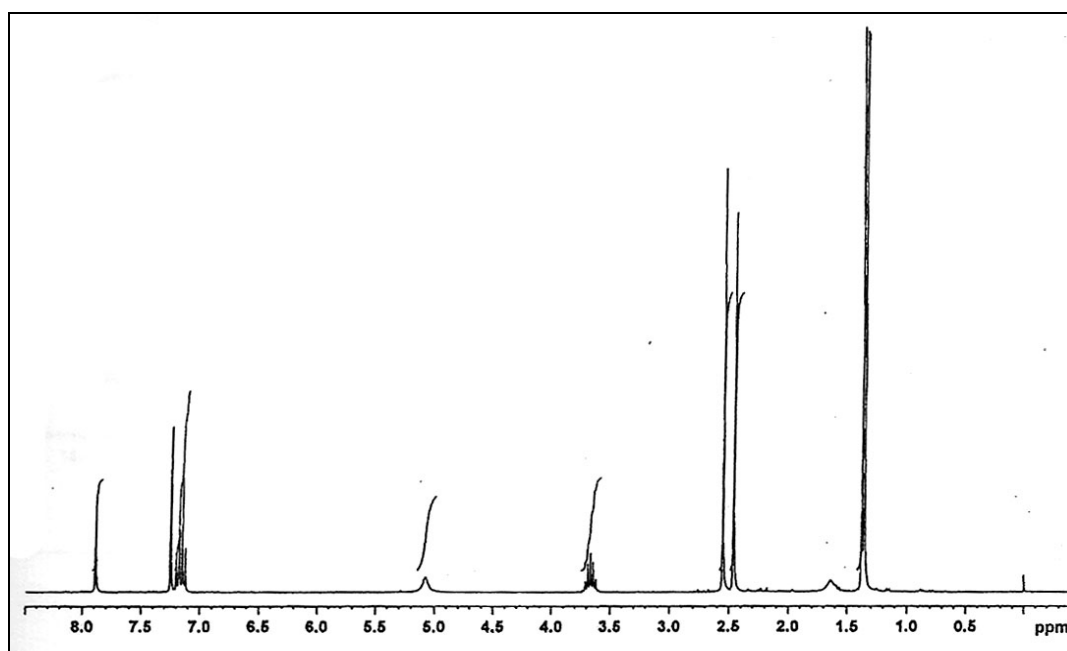
**Figure 39**  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **CF9**



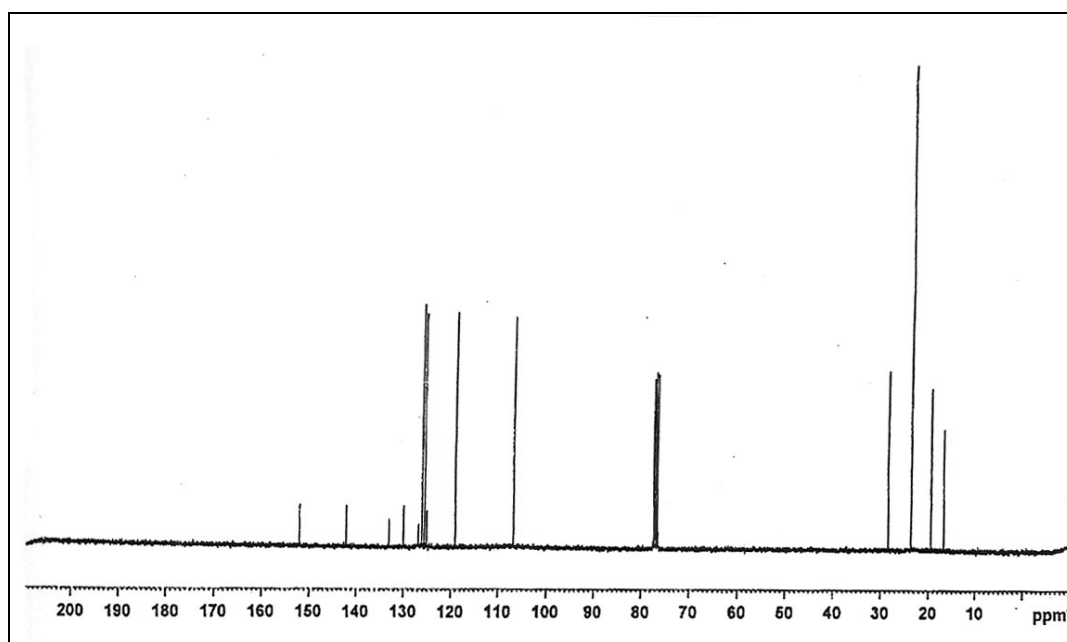
**Figure 40** UV (MeOH) spectrum of compound **TP1**



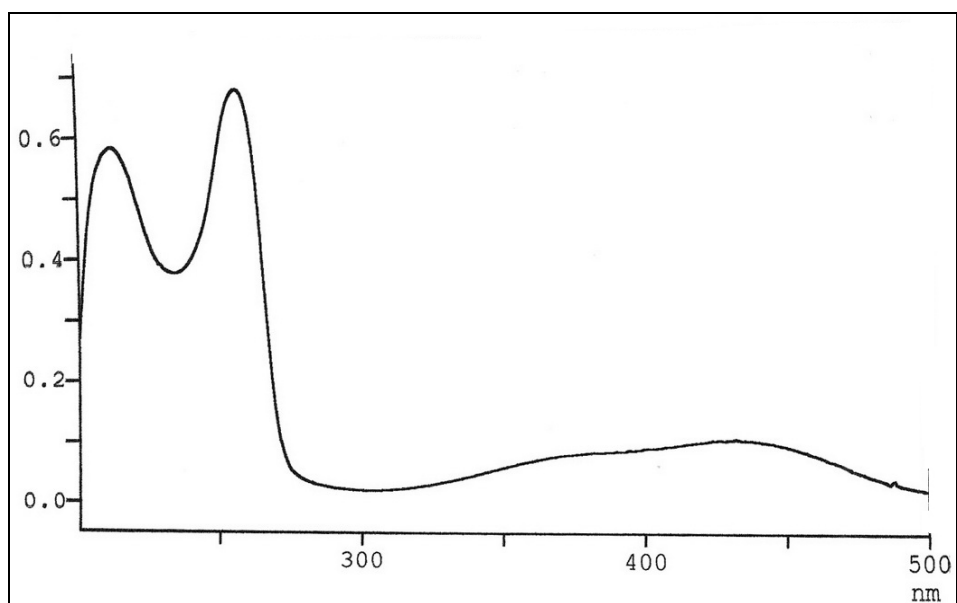
**Figure 41** IR (KBr) spectrum of compound **TP1**



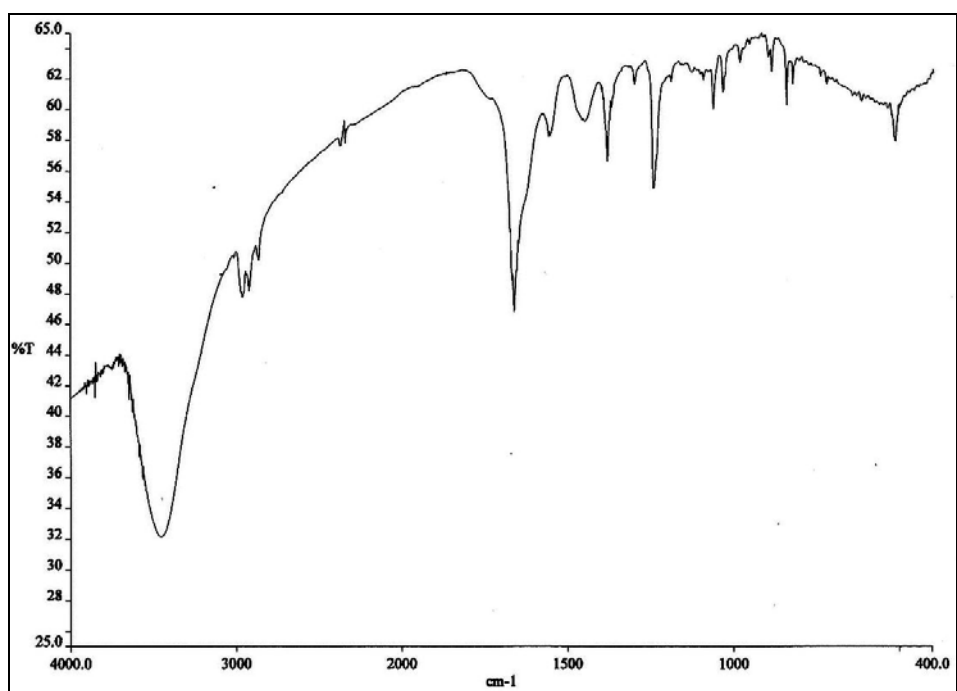
**Figure 42**  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **TP1**



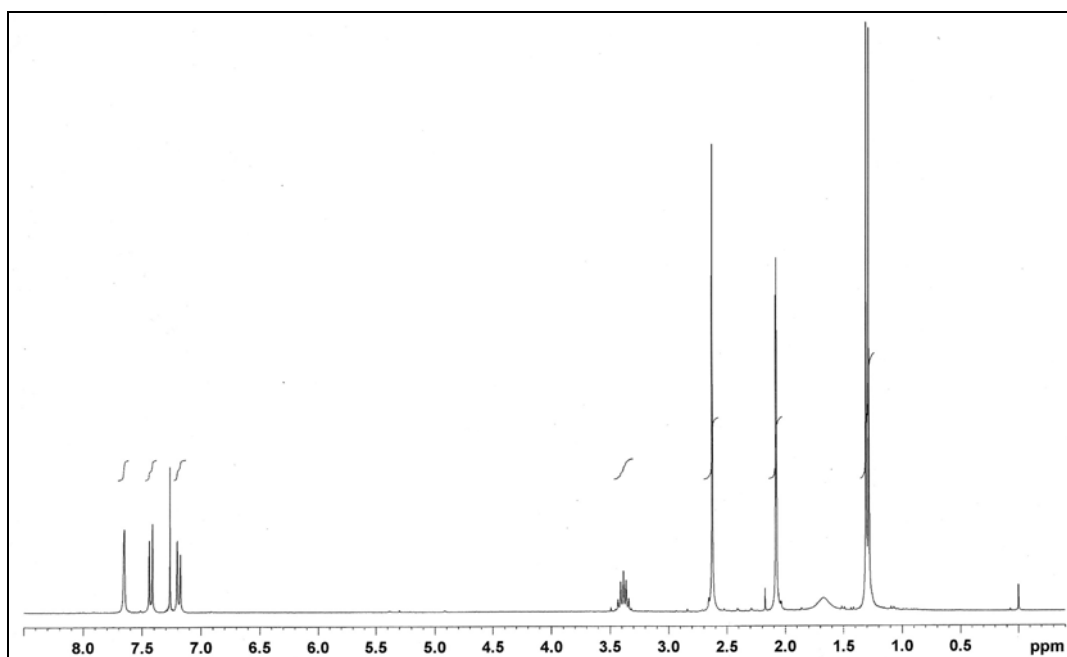
**Figure 43**  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **TP1**



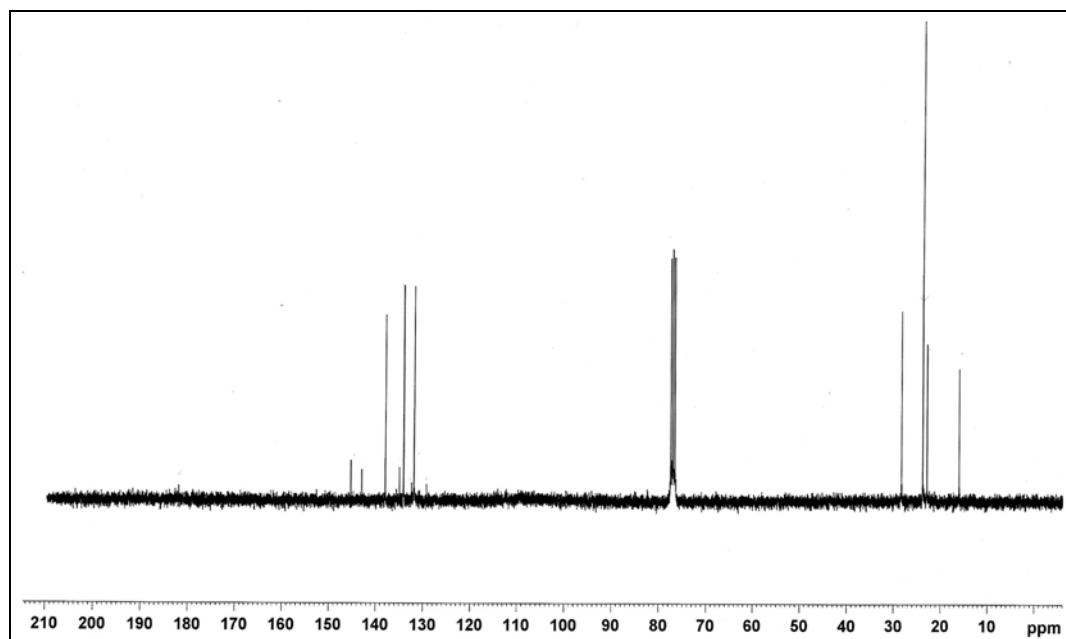
**Figure 44** UV (MeOH) spectrum of compound TP2



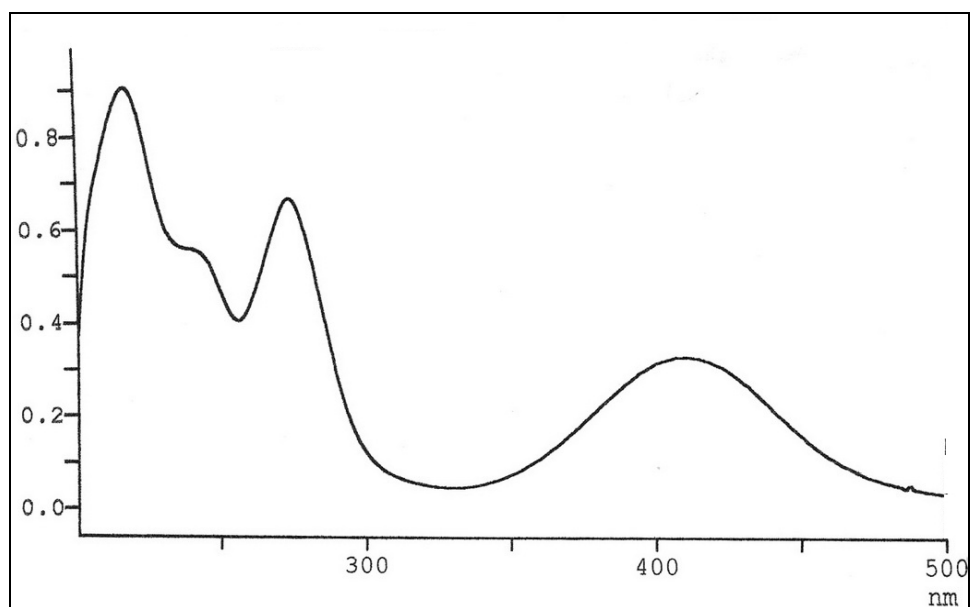
**Figure 45** IR (neat) spectrum of compound TP2



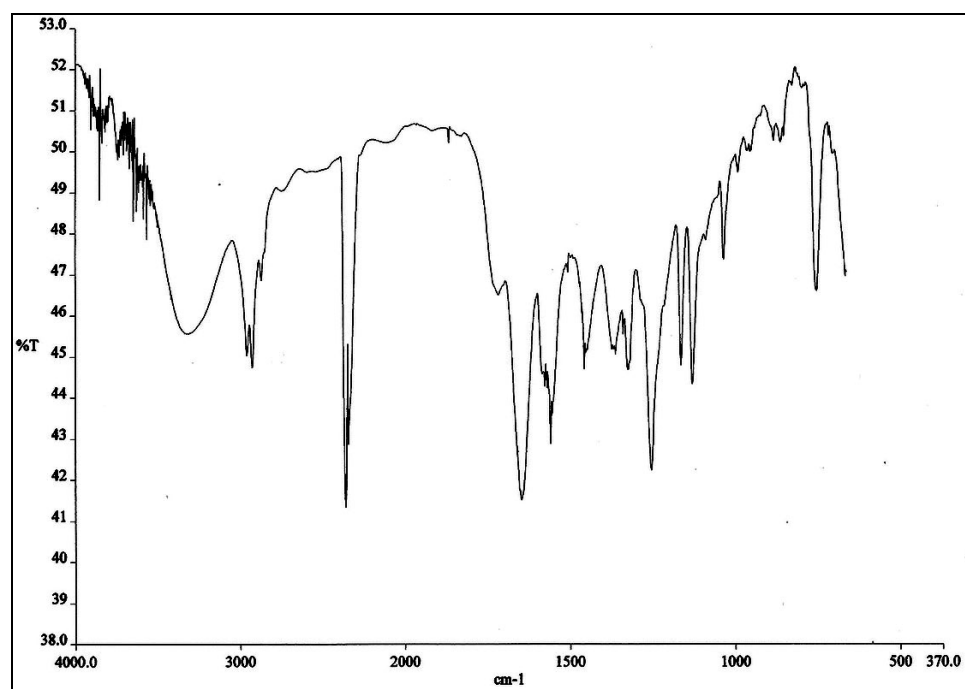
**Figure 46**  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **TP2**



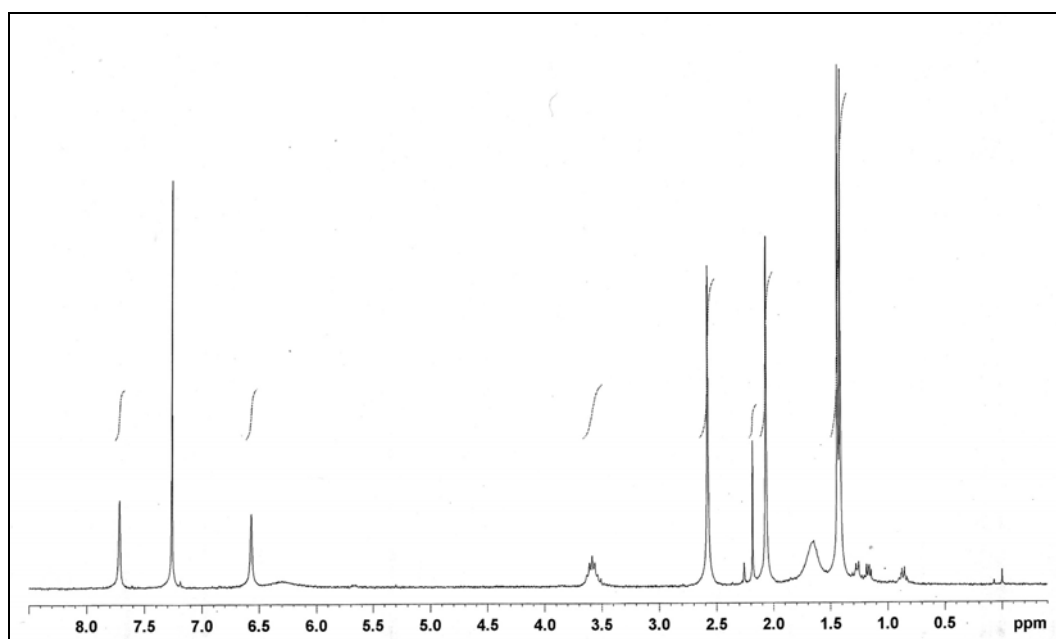
**Figure 47**  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **TP2**



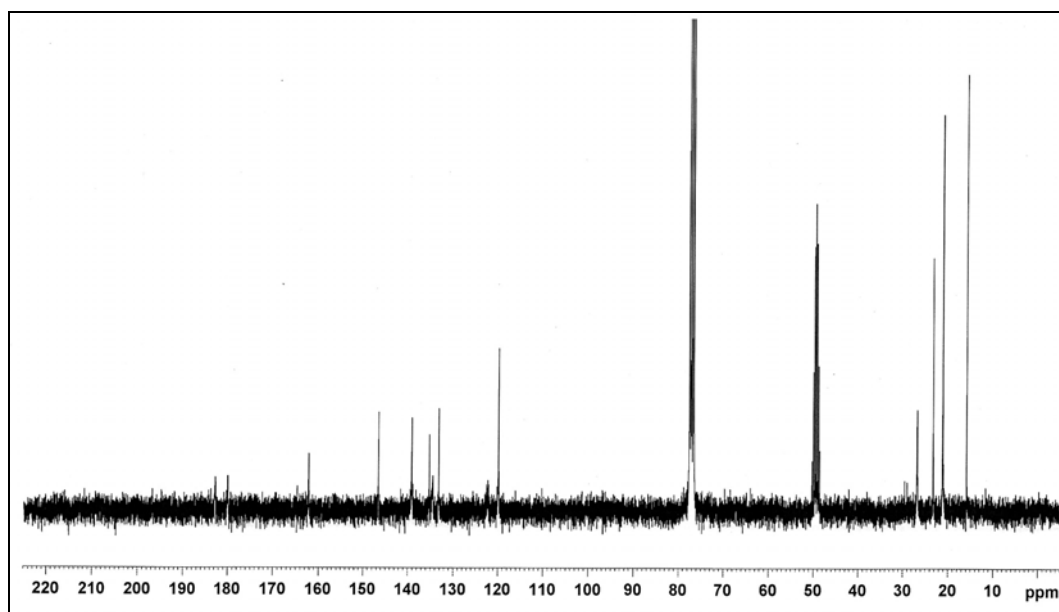
**Figure 48** UV (MeOH) spectrum of compound TP3



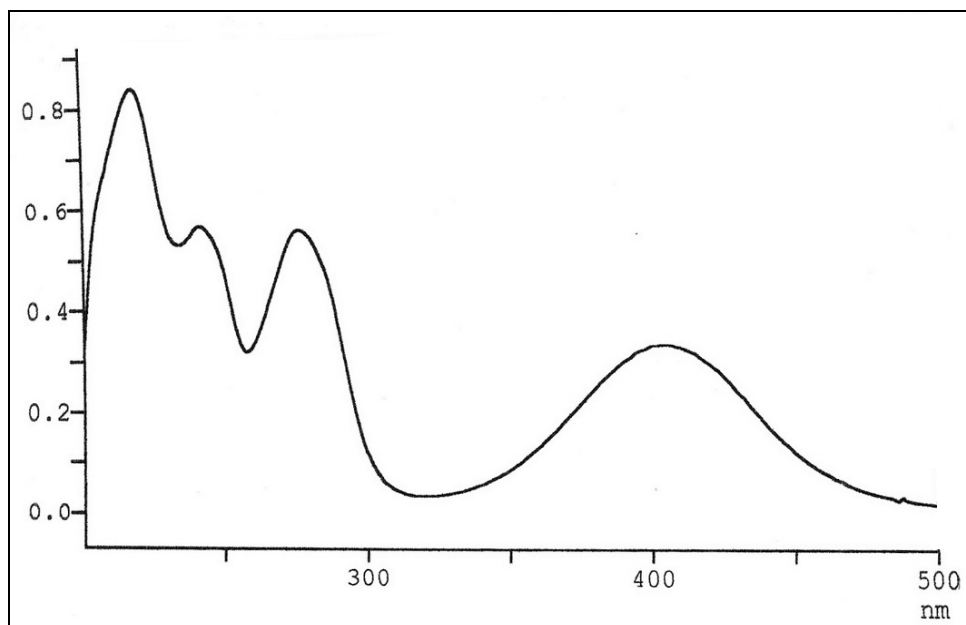
**Figure 49** IR (neat) spectrum of compound TP3



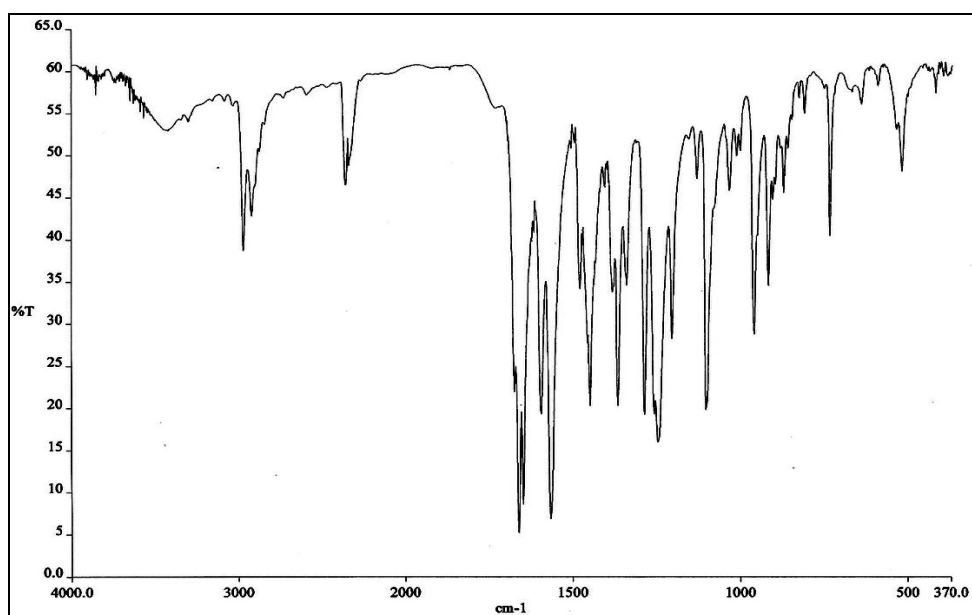
**Figure 50**  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3+\text{CD}_3\text{OD}$ ) spectrum of compound **TP3**



**Figure 51**  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3+\text{CD}_3\text{OD}$ ) spectrum of compound **TP3**

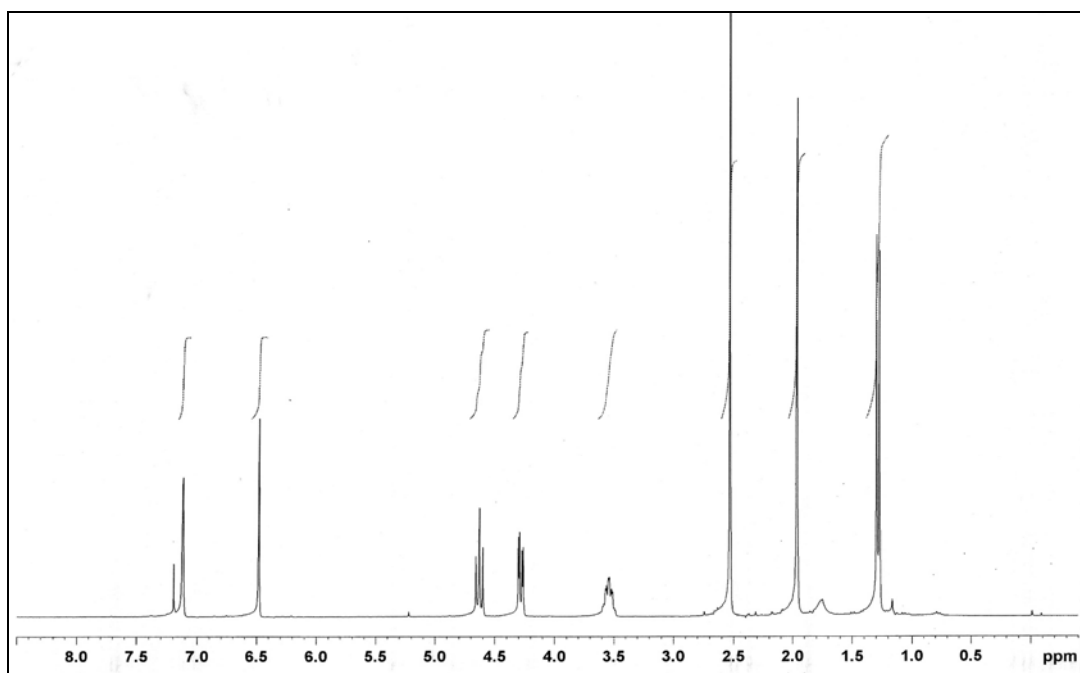


**Figure 52** UV (MeOH) spectrum of compound **TP4**

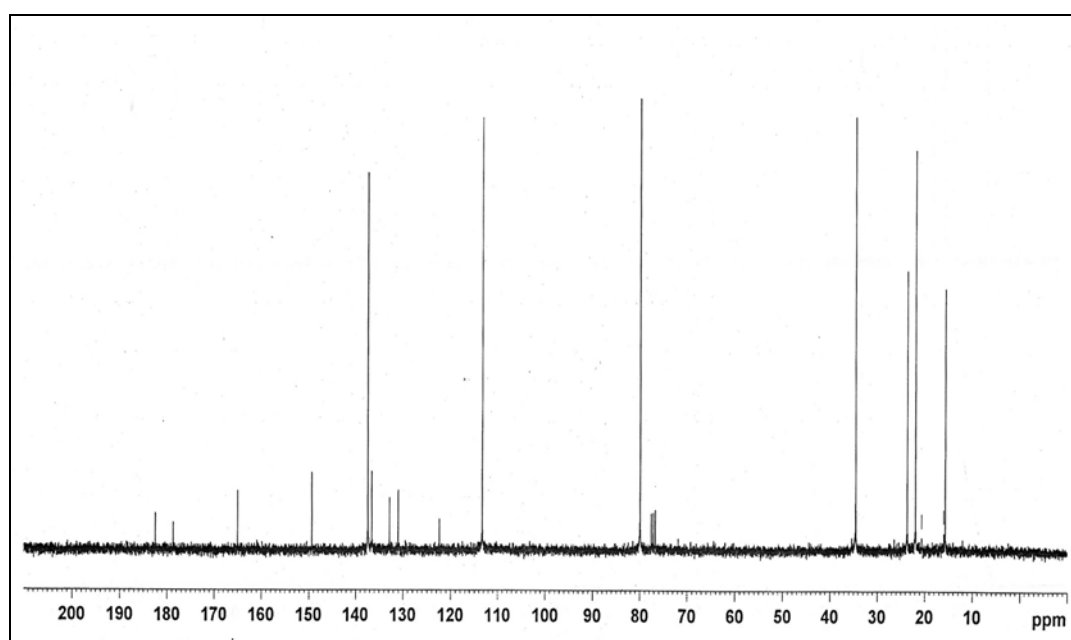


**Figure 53** IR (neat) spectrum of compound **TP4**

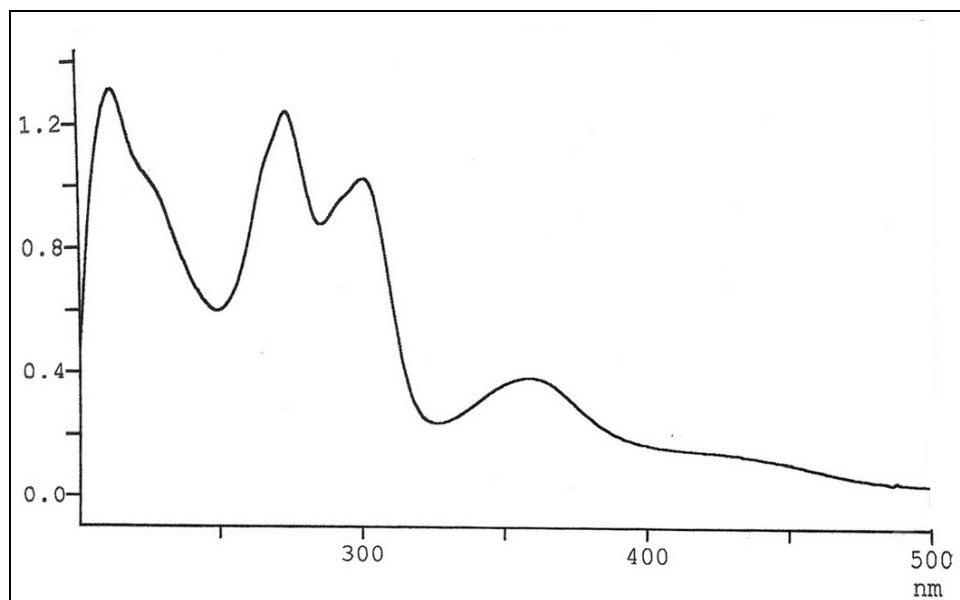




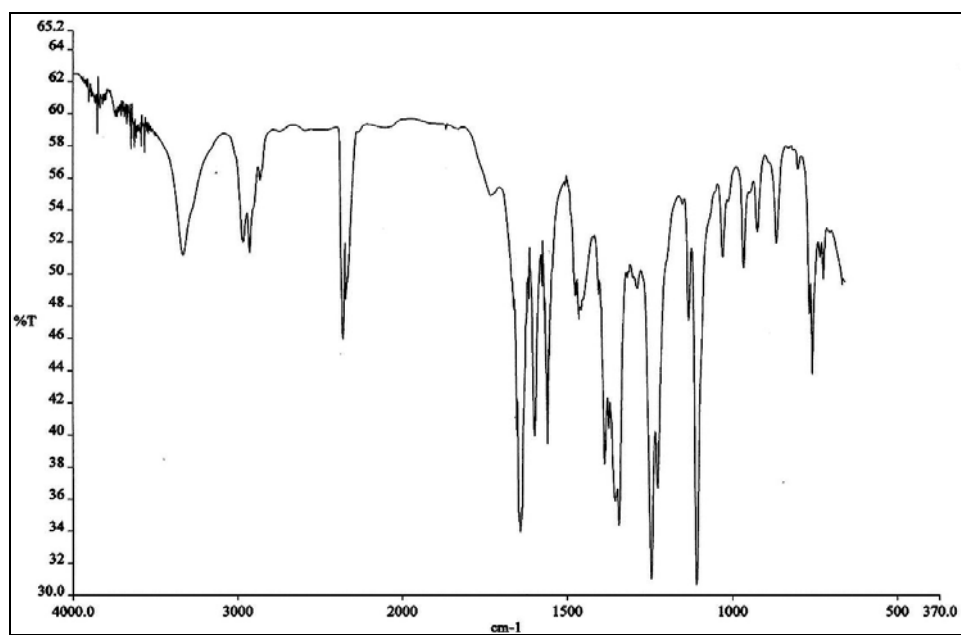
**Figure 54**  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **TP4**



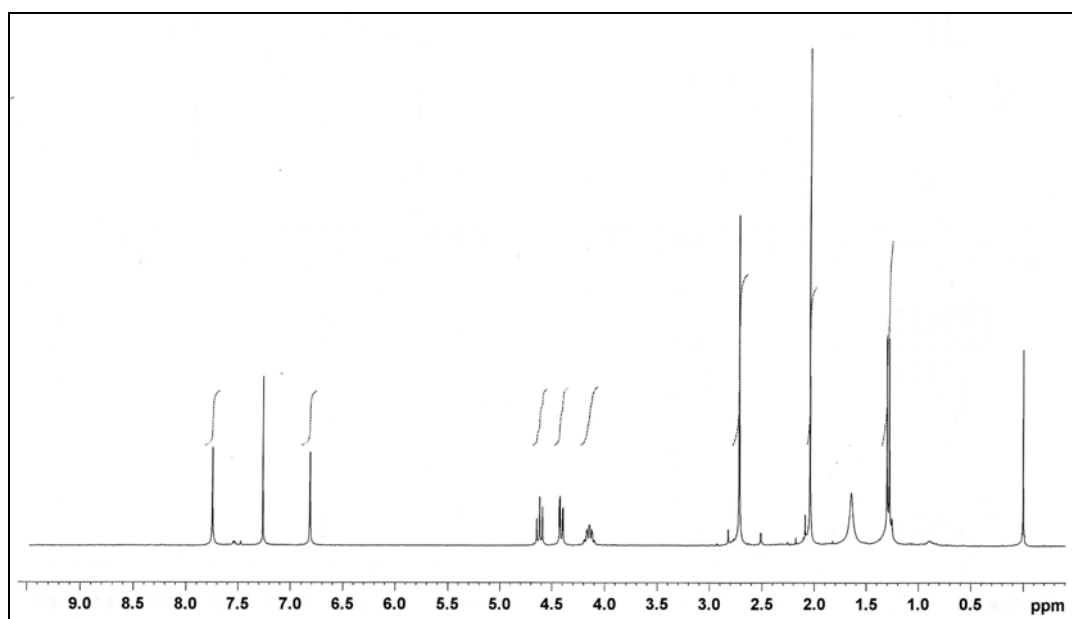
**Figure 55**  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **TP4**



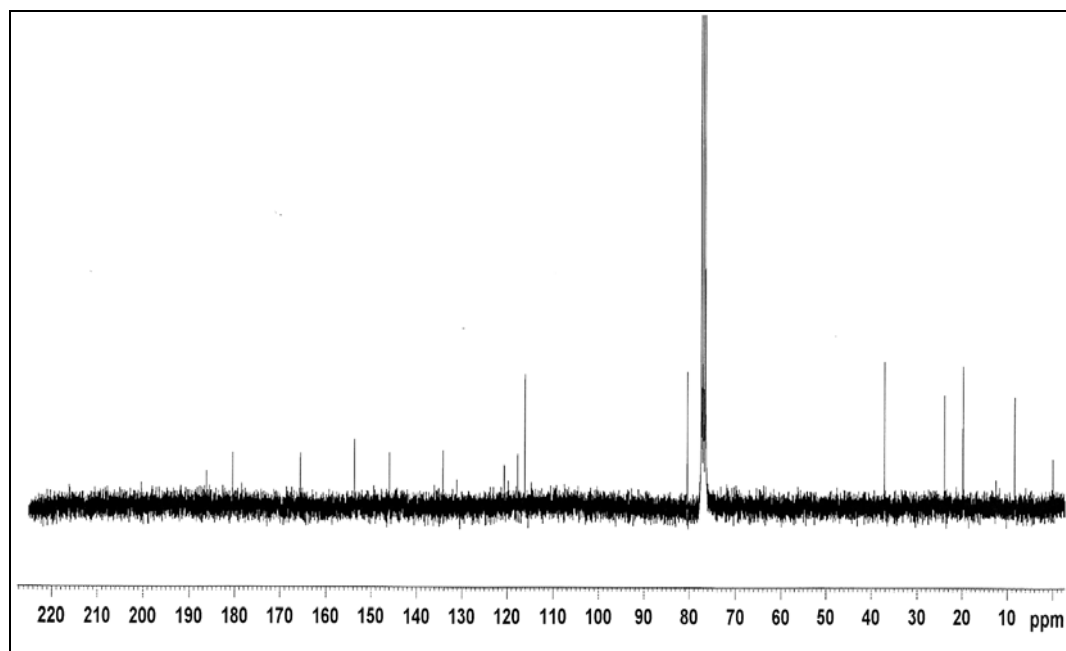
**Figure 56** UV (MeOH) spectrum of compound TP5



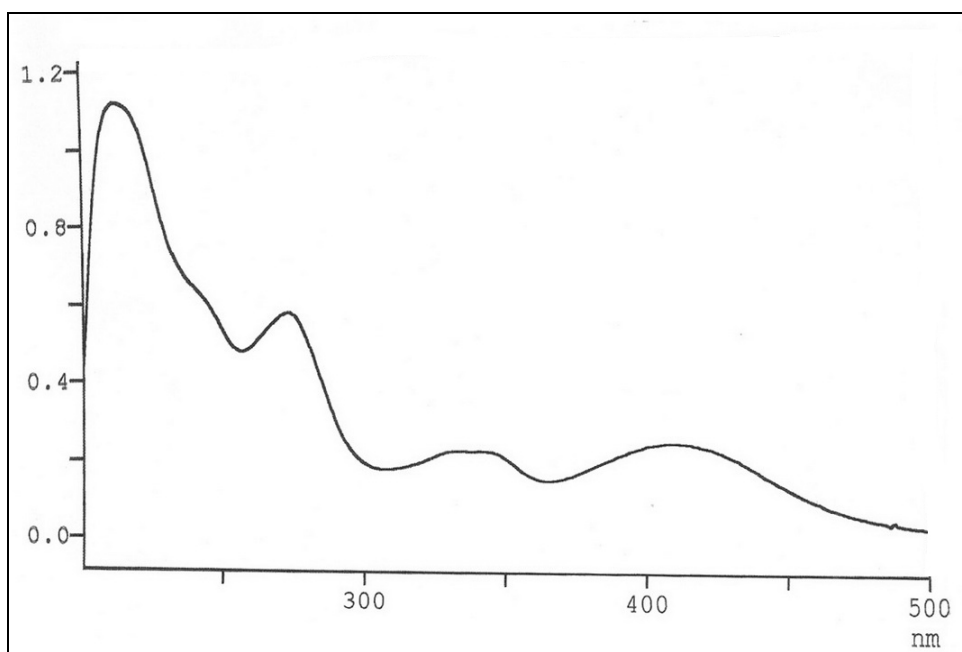
**Figure 57** IR (neat) spectrum of compound TP5



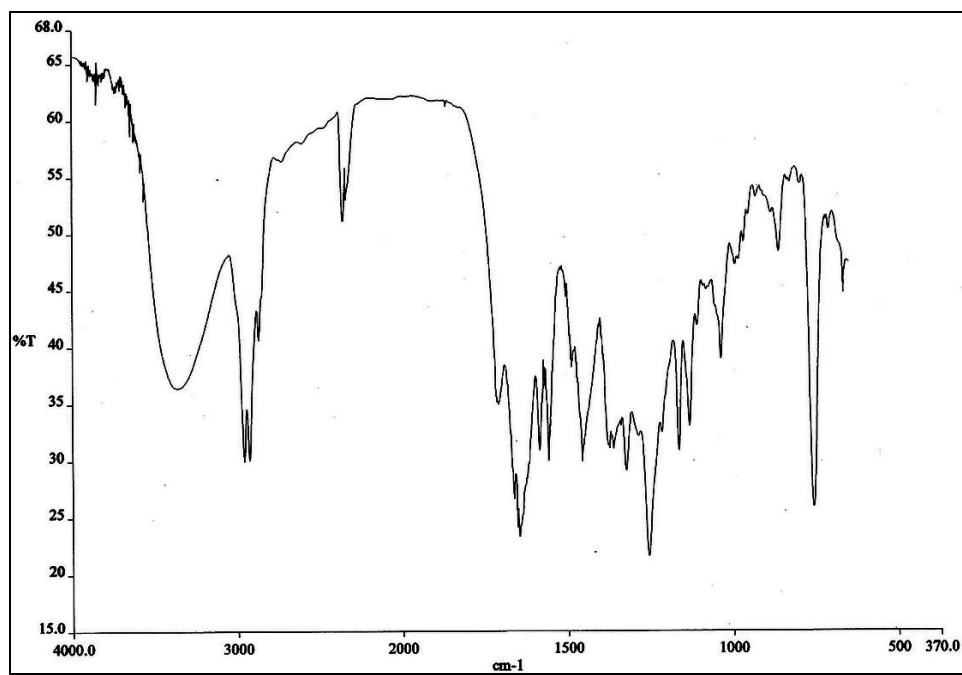
**Figure 58**  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **TP5**



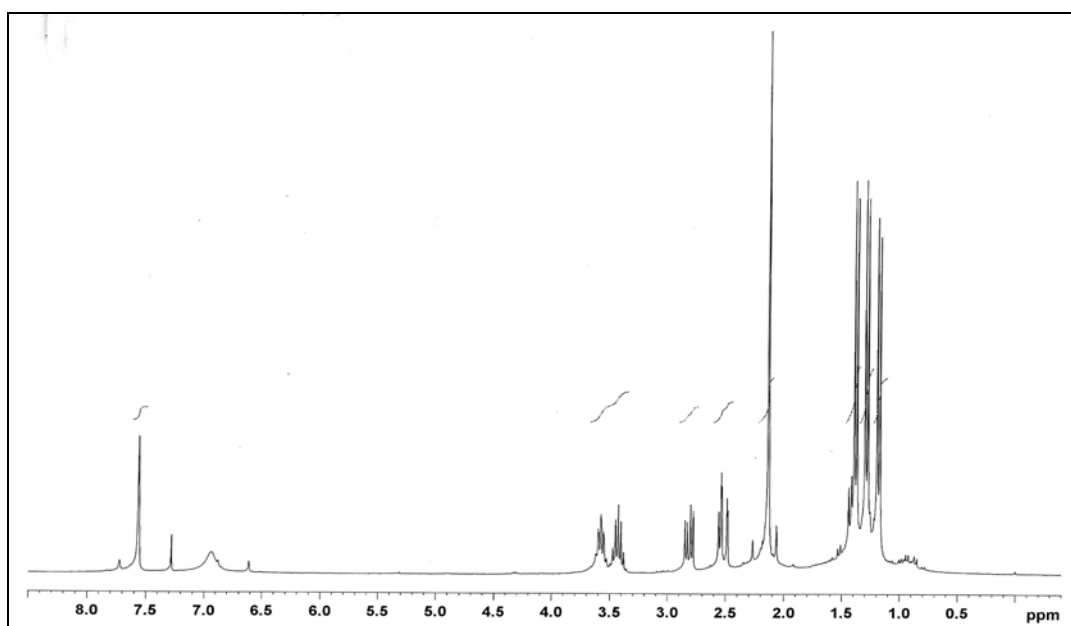
**Figure 59**  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **TP5**



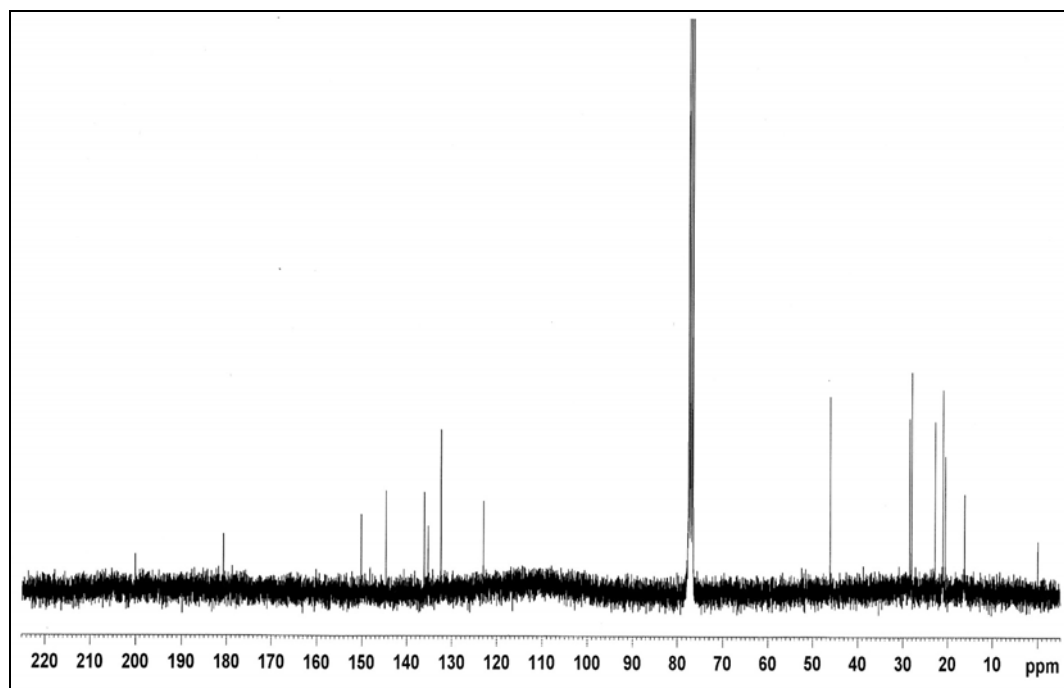
**Figure 60** UV (MeOH) spectrum of compound **TP6**



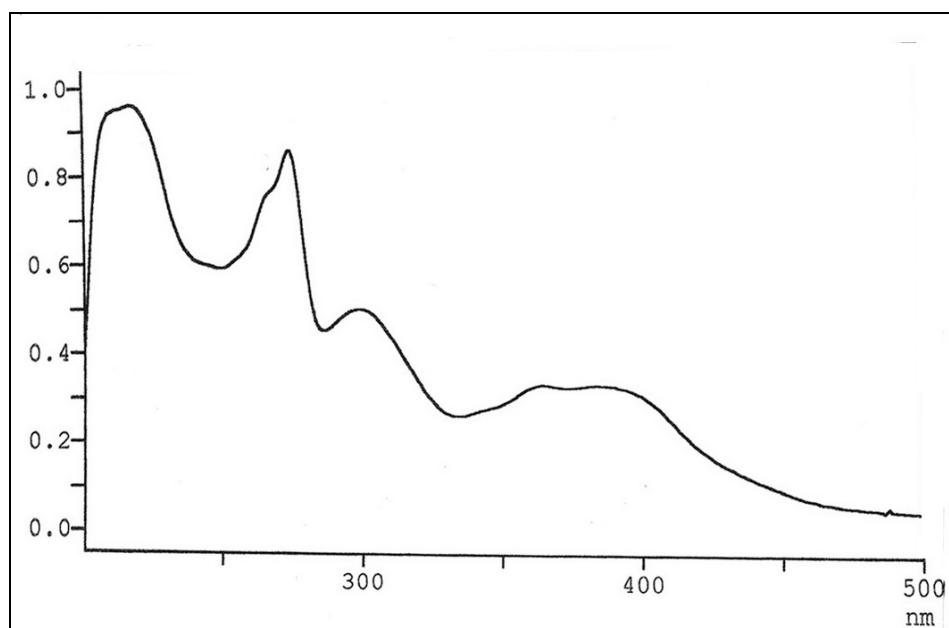
**Figure 61** IR (neat) spectrum of compound **TP6**



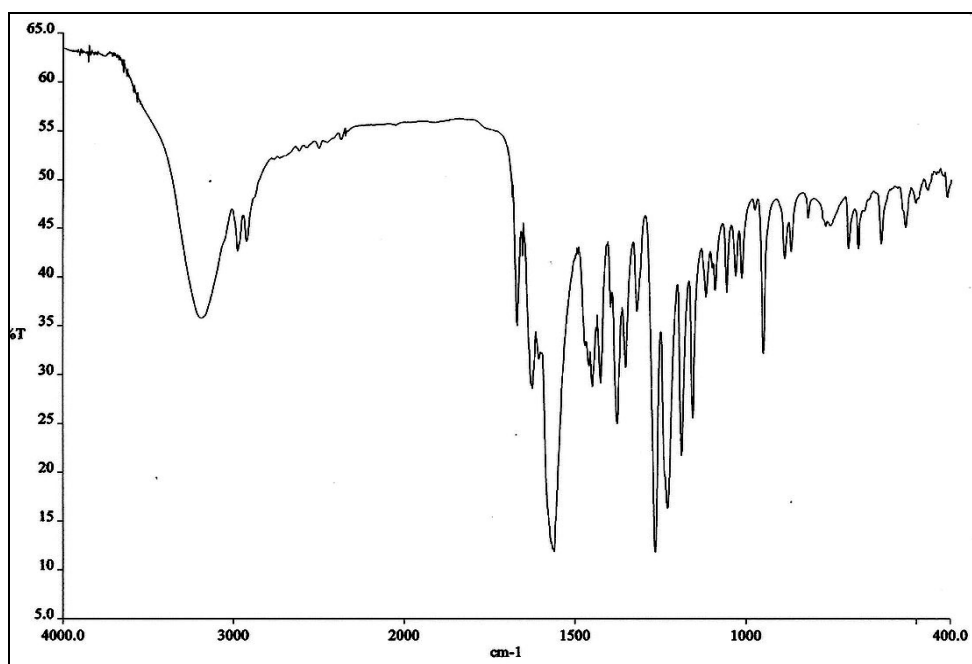
**Figure 62**  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **TP6**



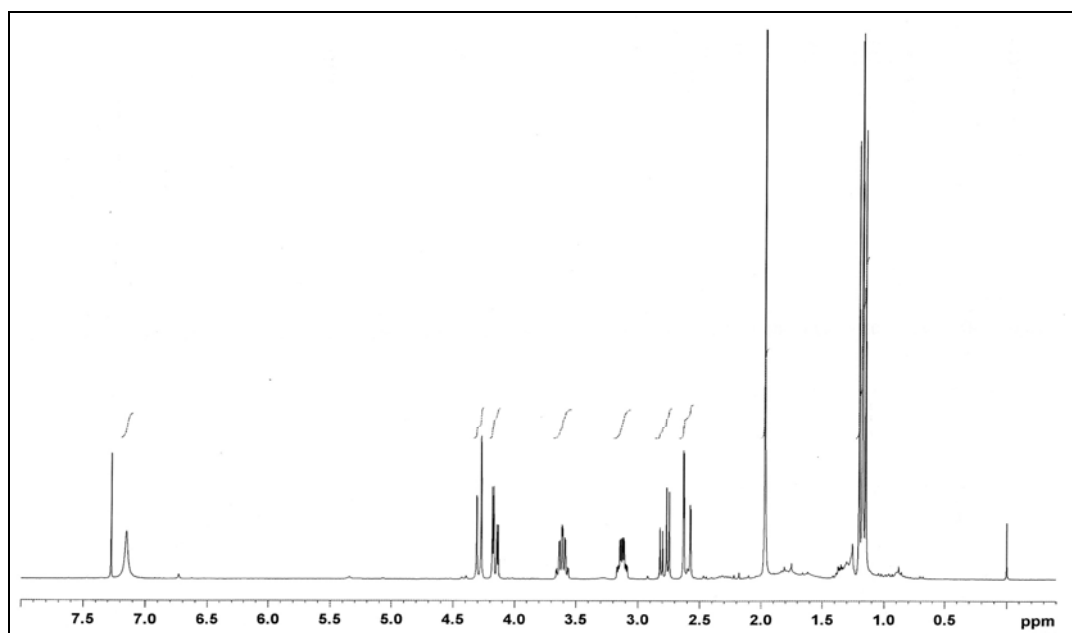
**Figure 63**  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **TP6**



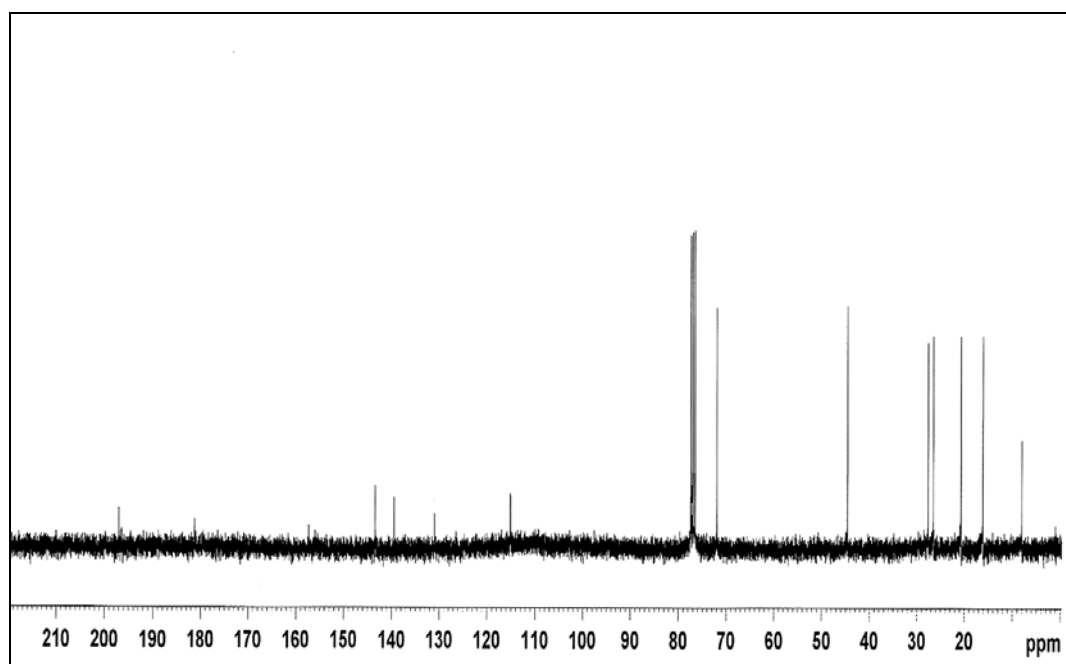
**Figure 64** UV (MeOH) spectrum of compound **TP7**



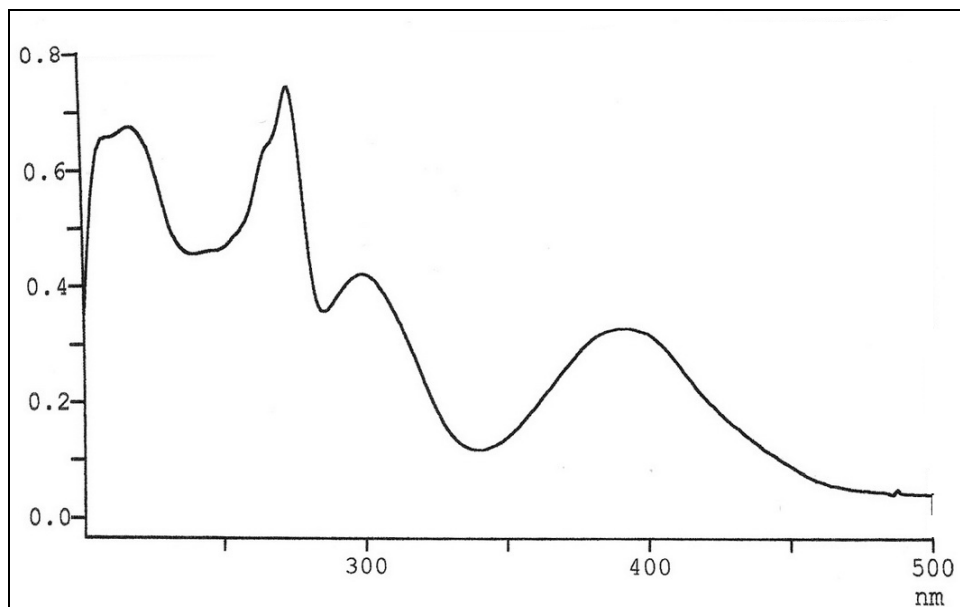
**Figure 65** IR (neat) spectrum of compound **TP7**



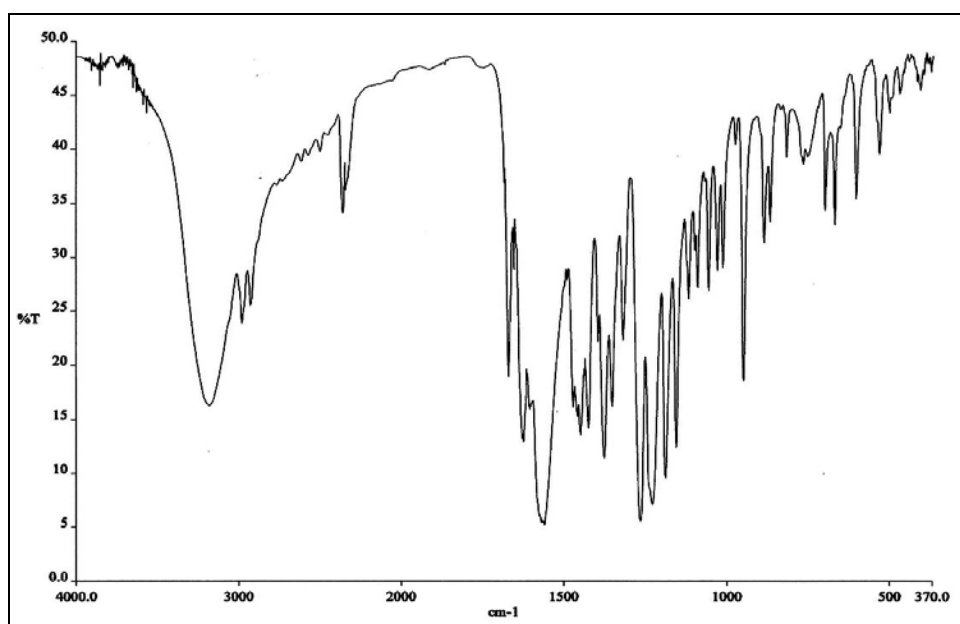
**Figure 66**  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **TP7**



**Figure 67**  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **TP7**

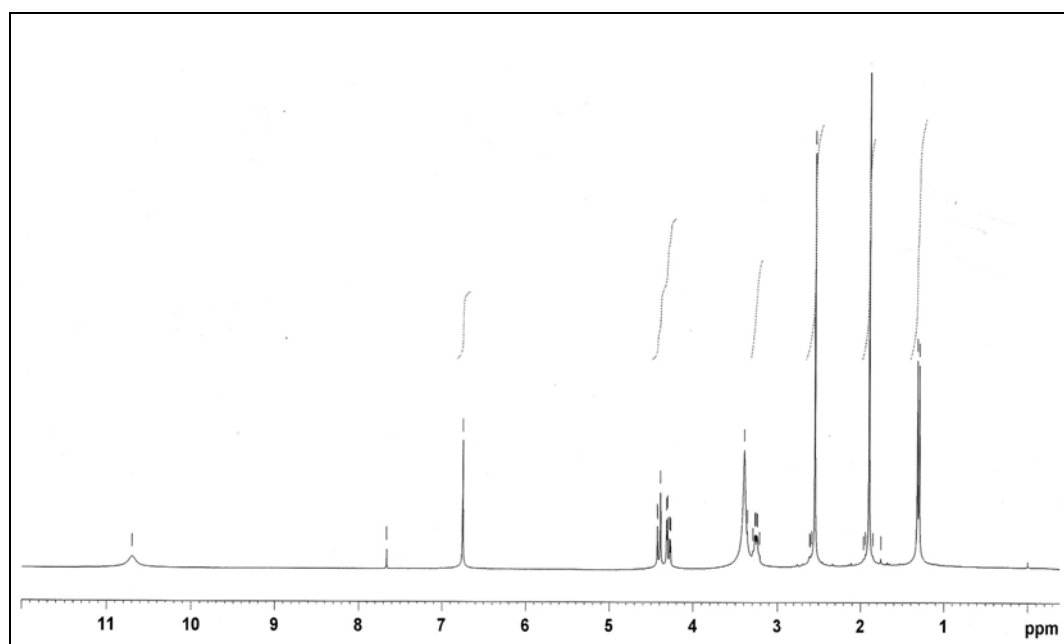


**Figure 68** UV (MeOH) spectrum of compound **TP8**

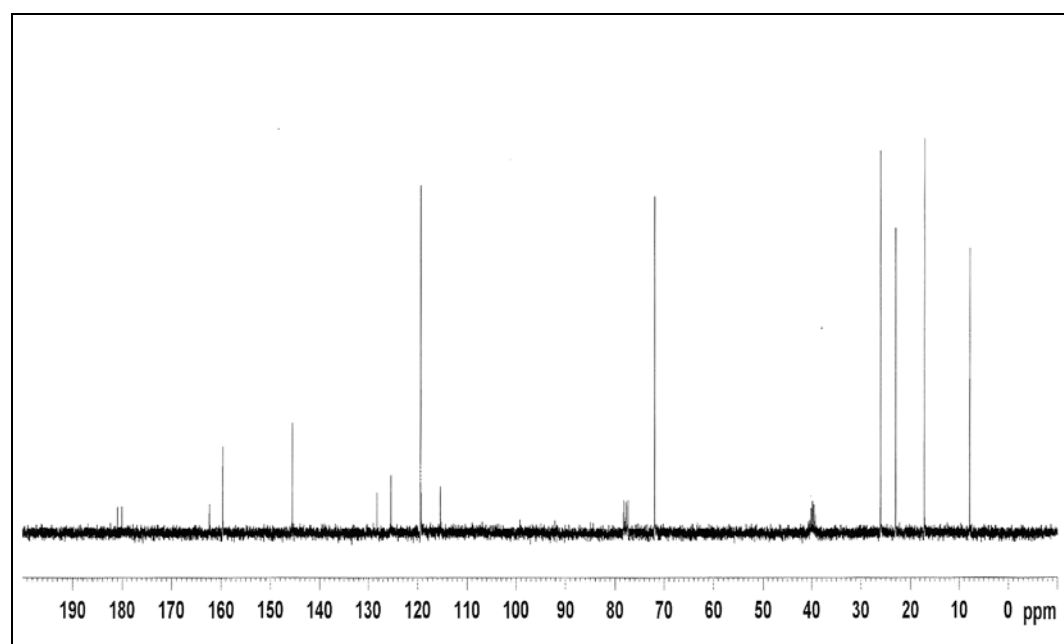


**Figure 69** IR (neat) spectrum of compound **TP8**

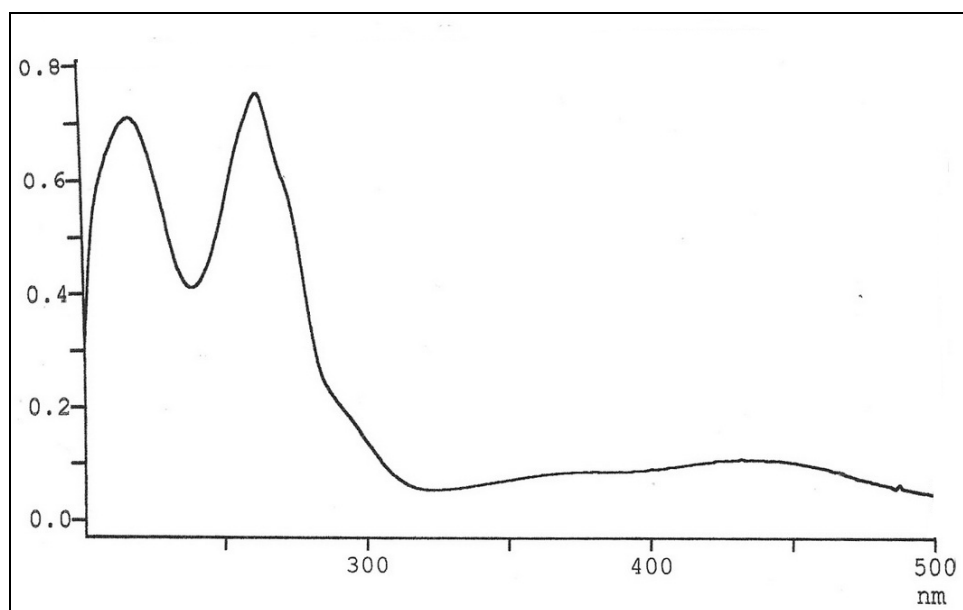




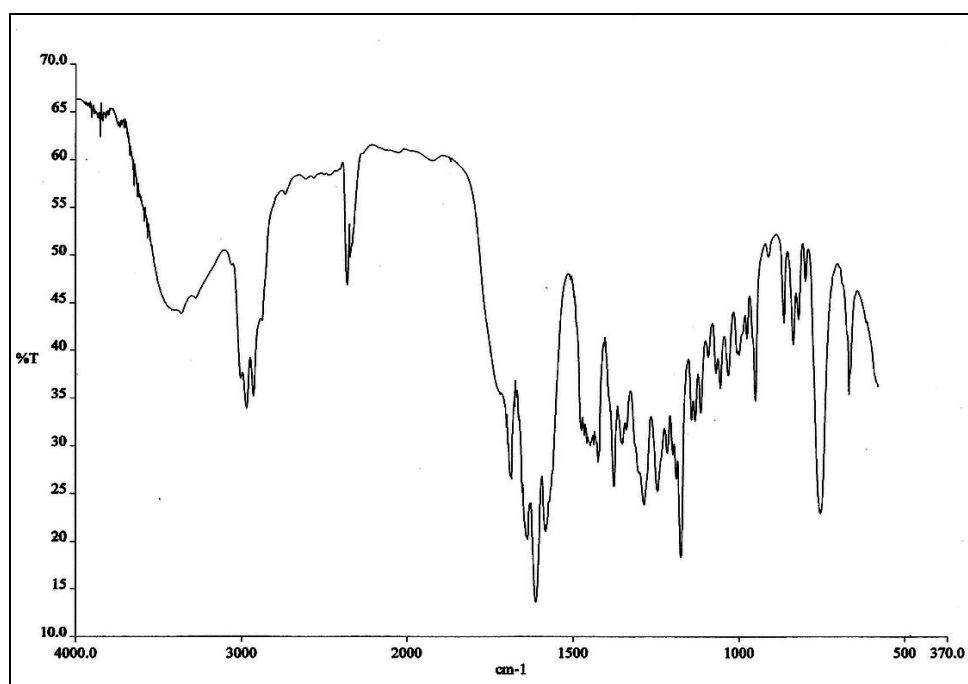
**Figure 70**  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3+\text{DMSO}-d_6$ ) spectrum of compound **TP8**



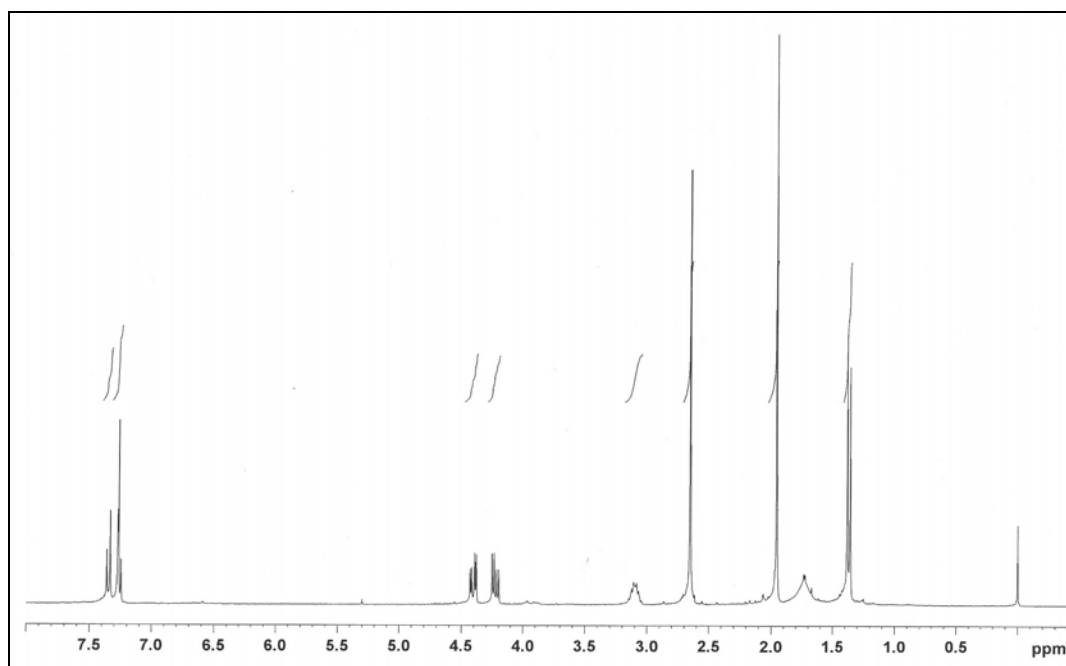
**Figure 71**  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3+\text{DMSO}-d_6$ ) spectrum of compound **TP8**



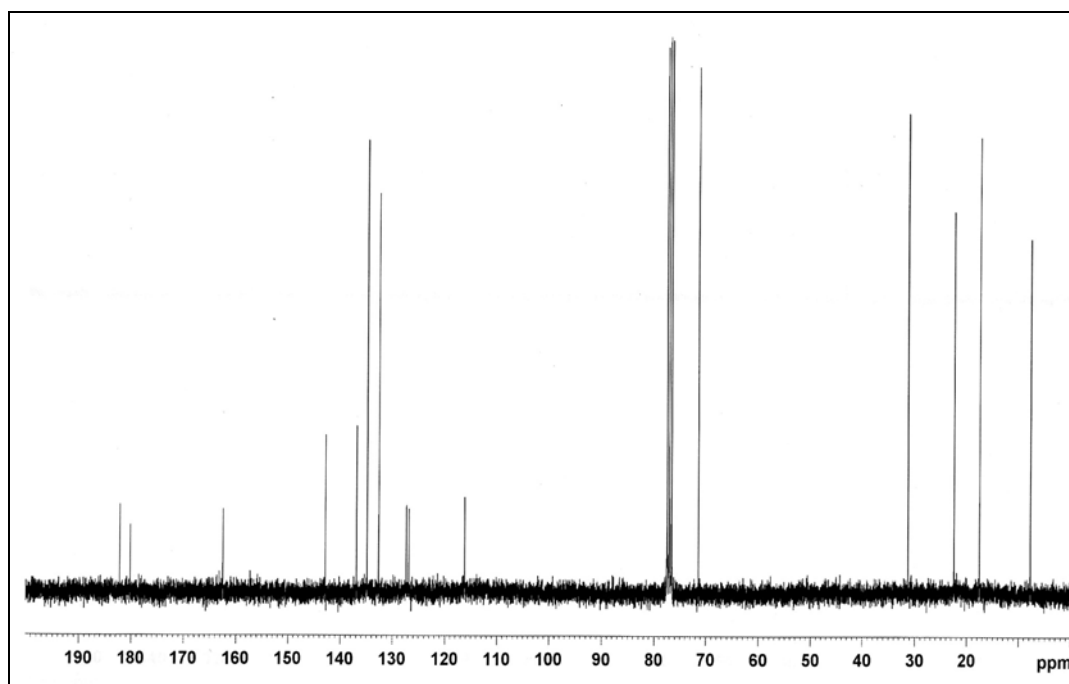
**Figure 72** (MeOH) spectrum of compound **TP9**



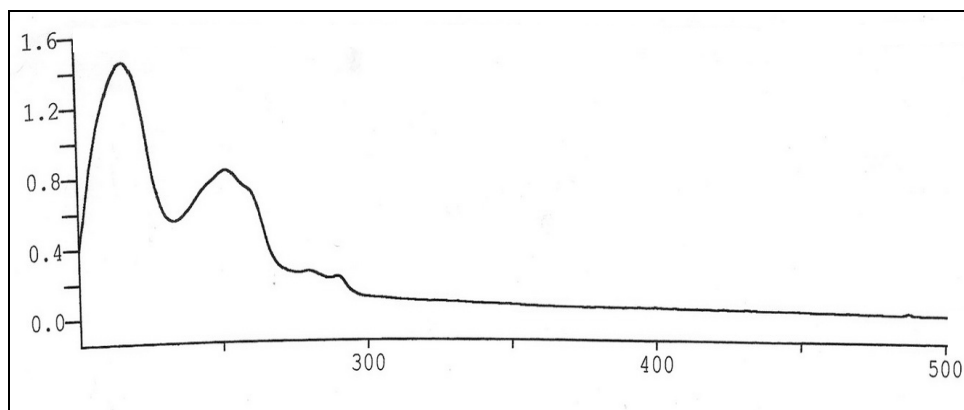
**Figure 73** IR (neat) spectrum of compound **TP9**



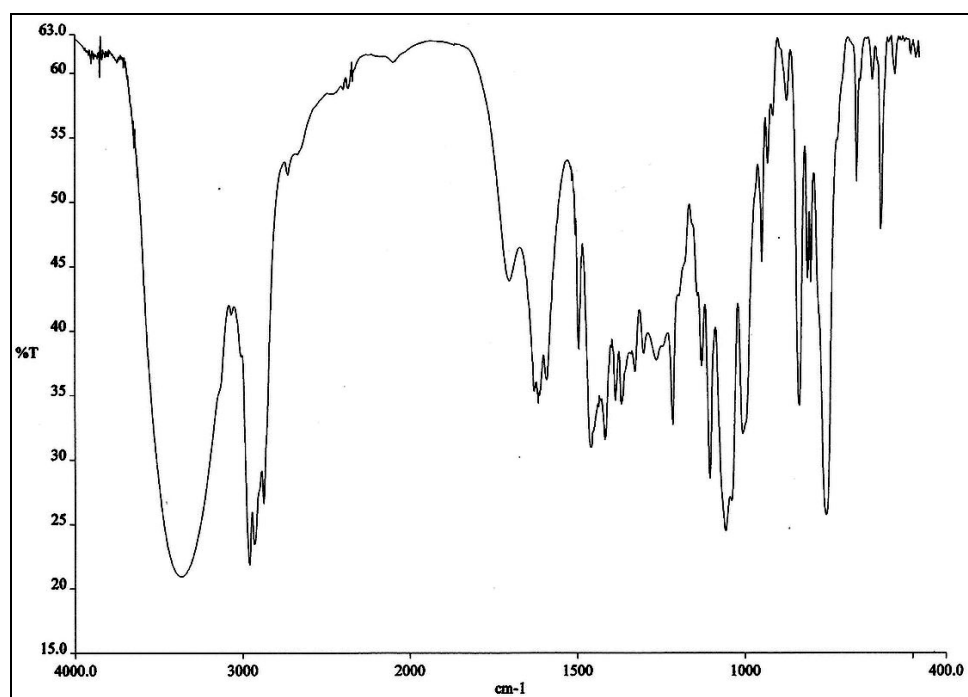
**Figure 74**  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **TP9**



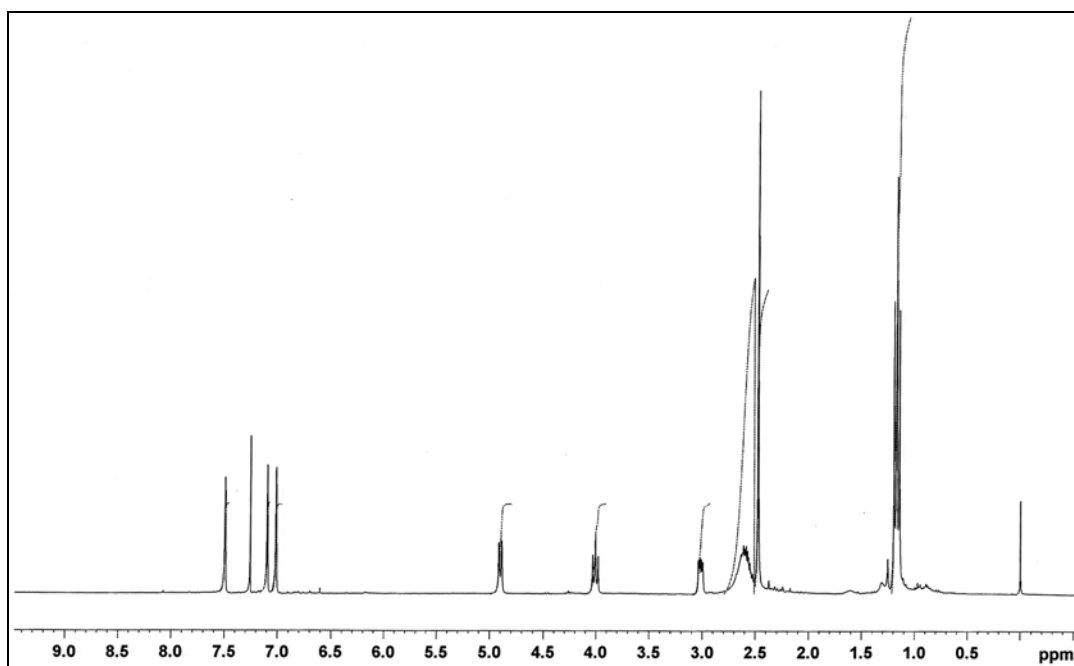
**Figure 75**  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **TP9**



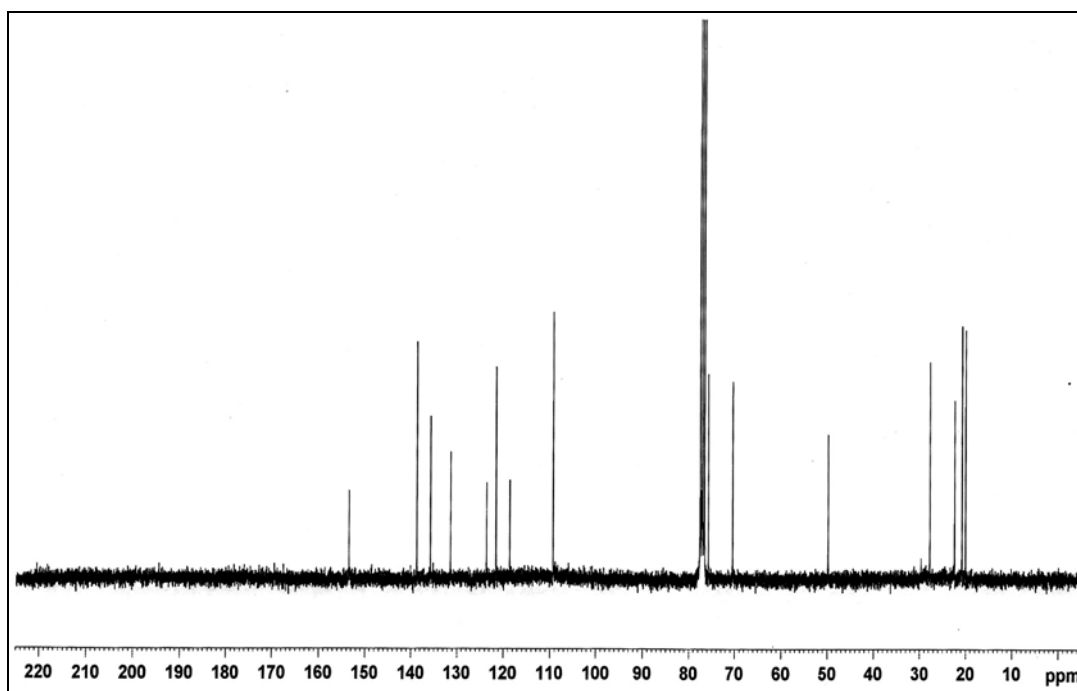
**Figure 76** UV (MeOH) spectrum of compound **TP10**



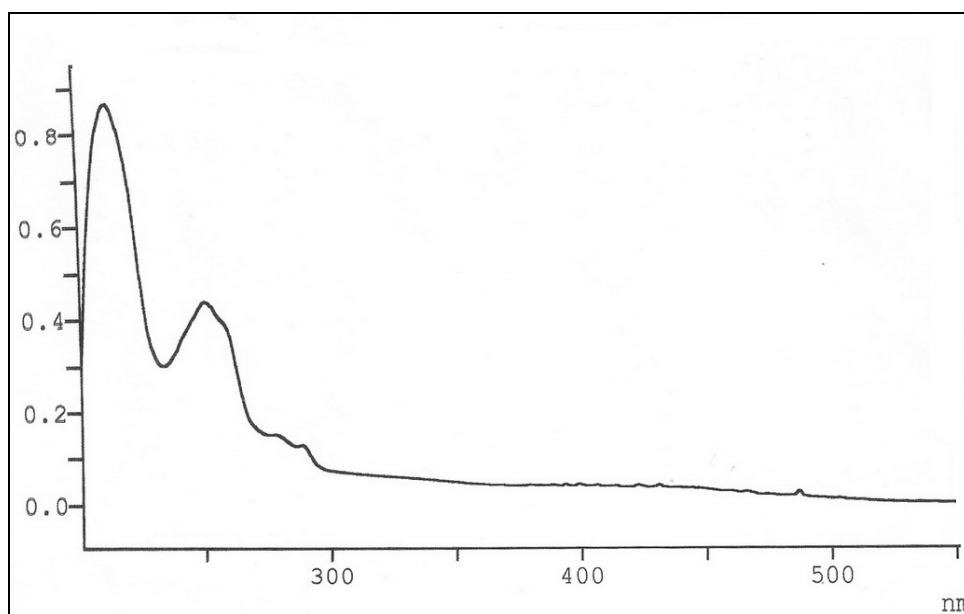
**Figure 77** IR (neat) spectrum of compound **TP10**



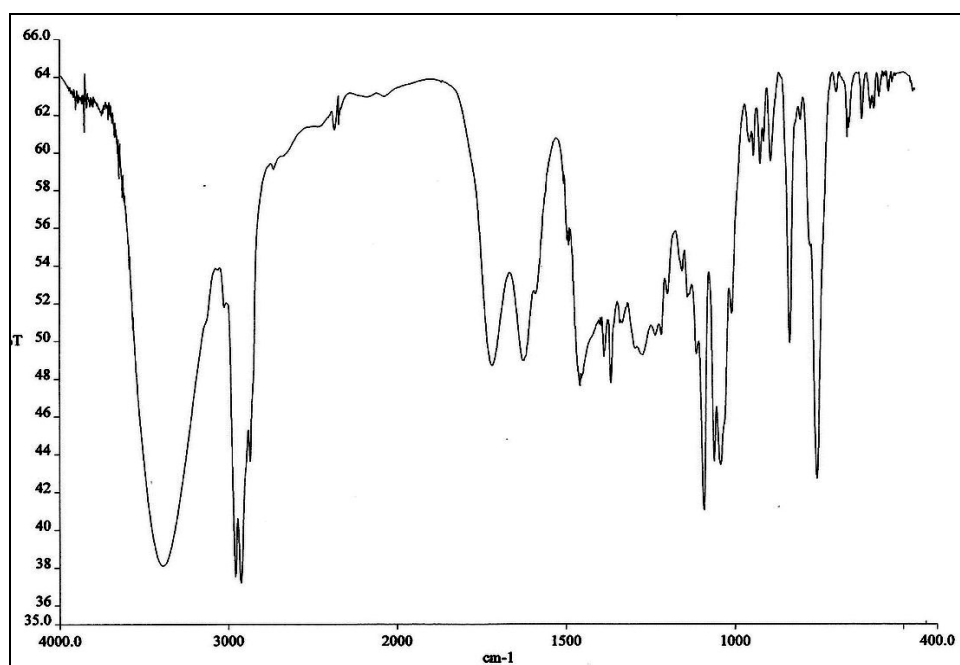
**Figure 78**  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **TP10**



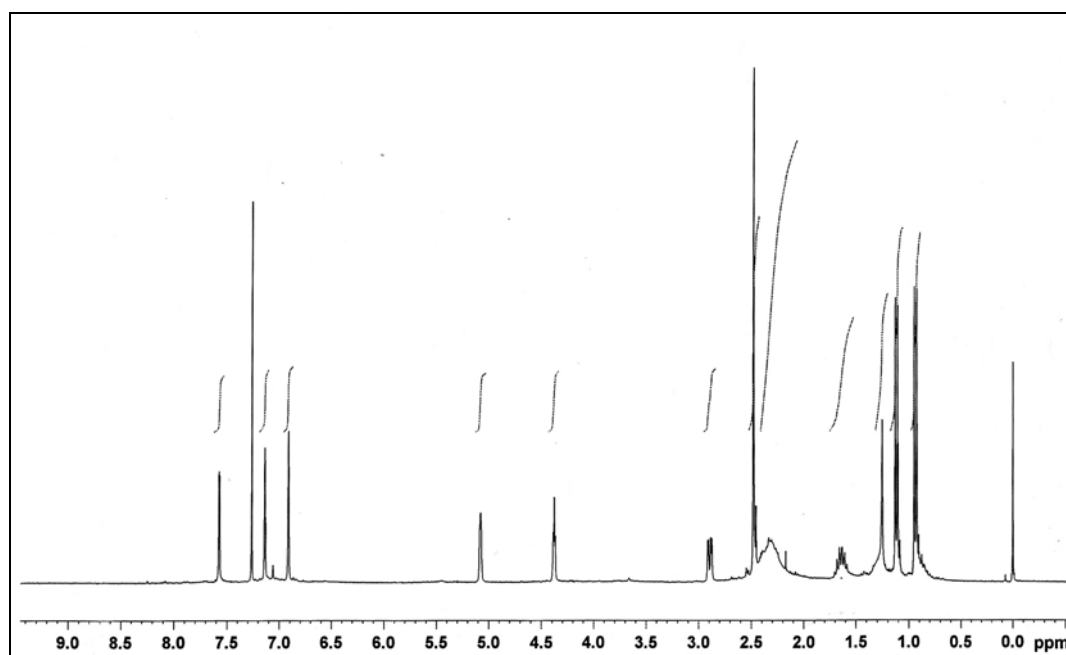
**Figure 79**  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **TP10**



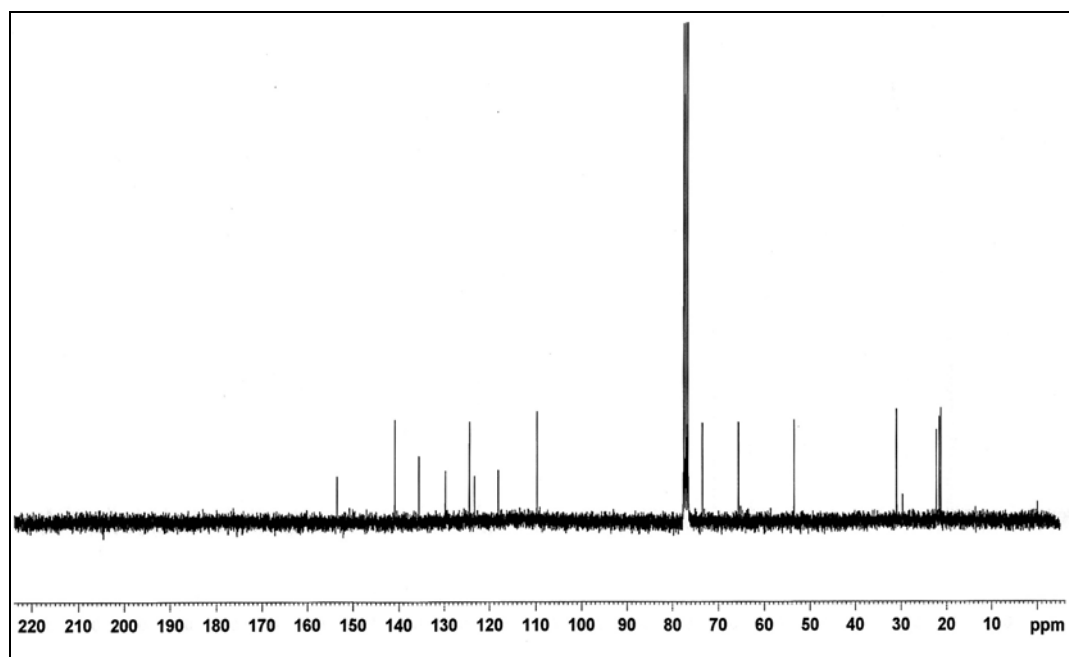
**Figure 80** UV (MeOH) spectrum of compound **TP11**



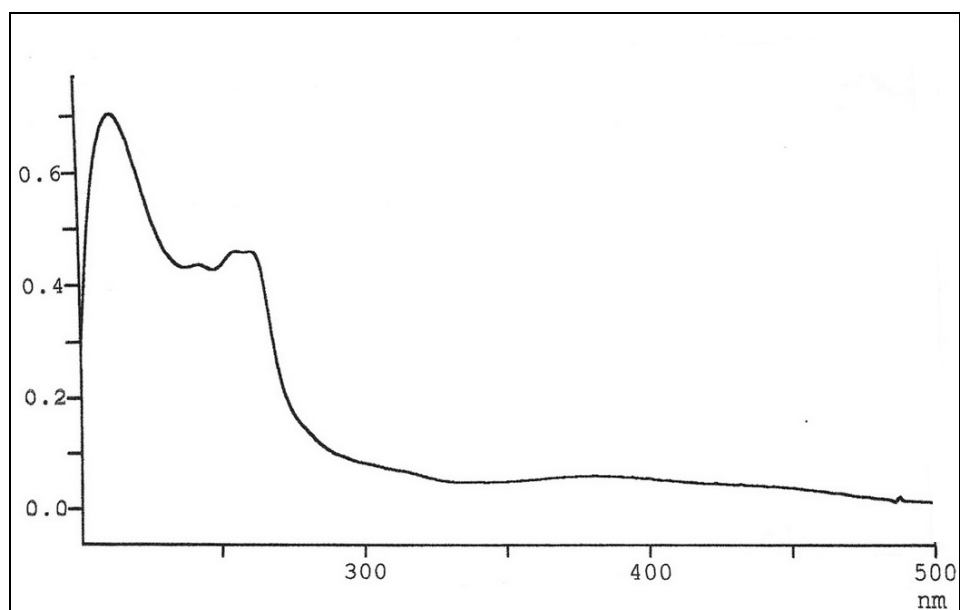
**Figure 81** IR (neat) spectrum of compound **TP11**



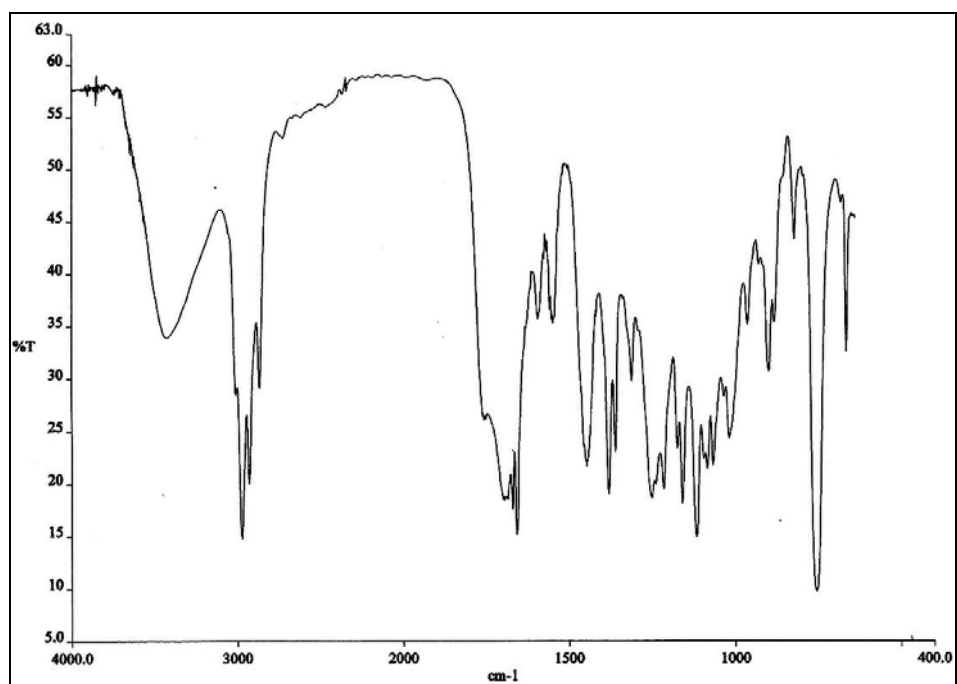
**Figure 82**  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **TP11**



**Figure 83**  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **TP11**

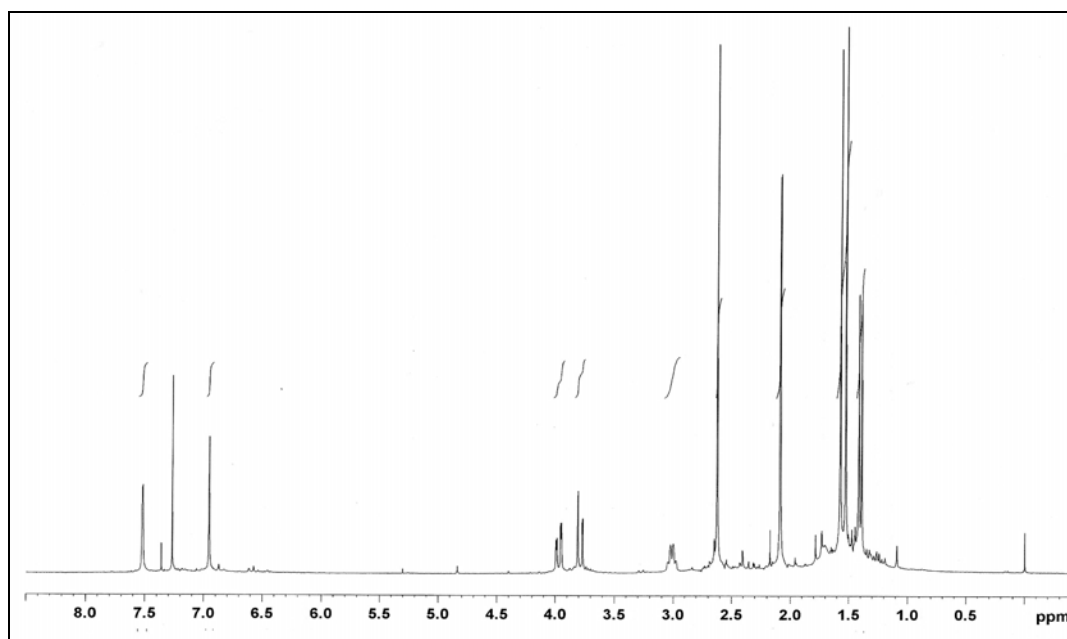


**Figure 84** UV (MeOH) spectrum of compound **TP12**

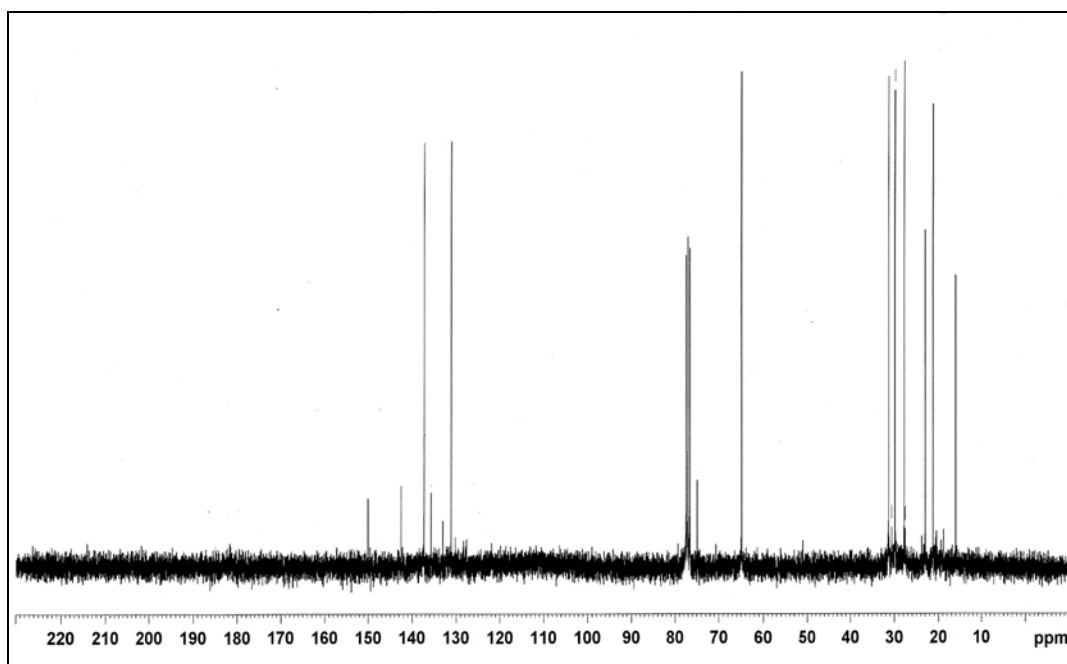


**Figure 85** IR (neat) spectrum of compound **TP12**

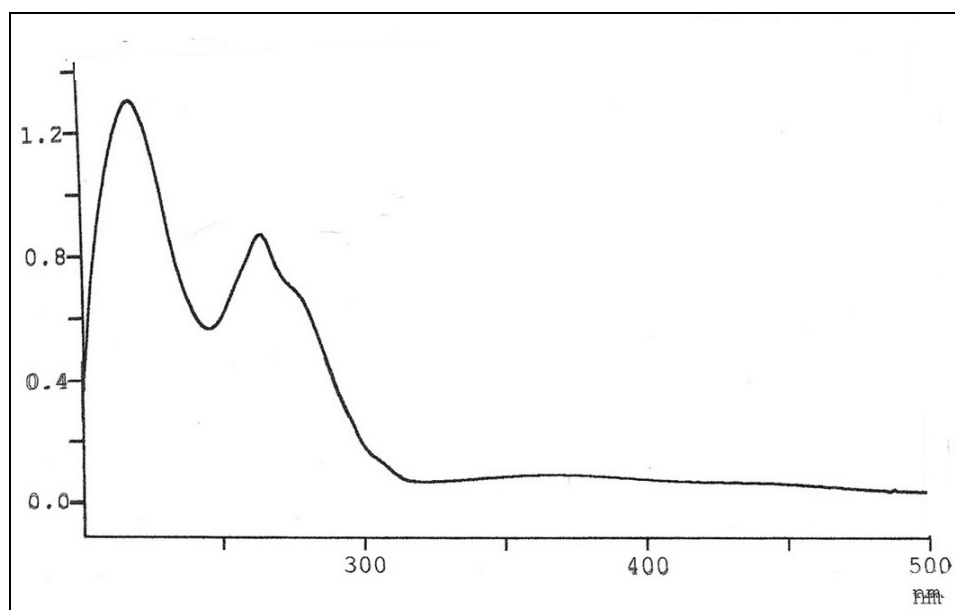




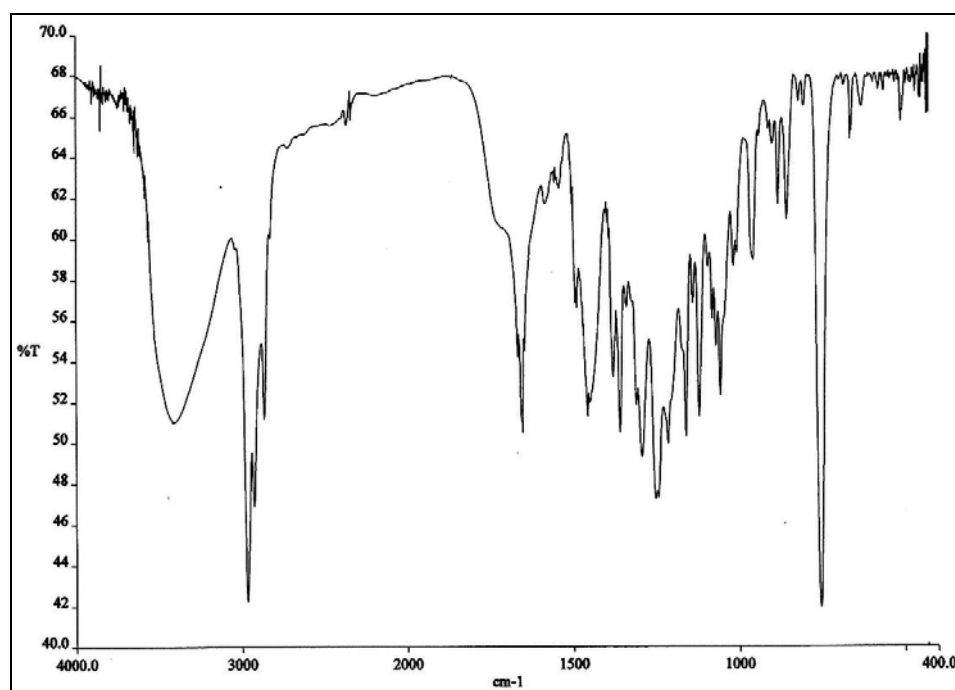
**Figure 86**  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **TP12**



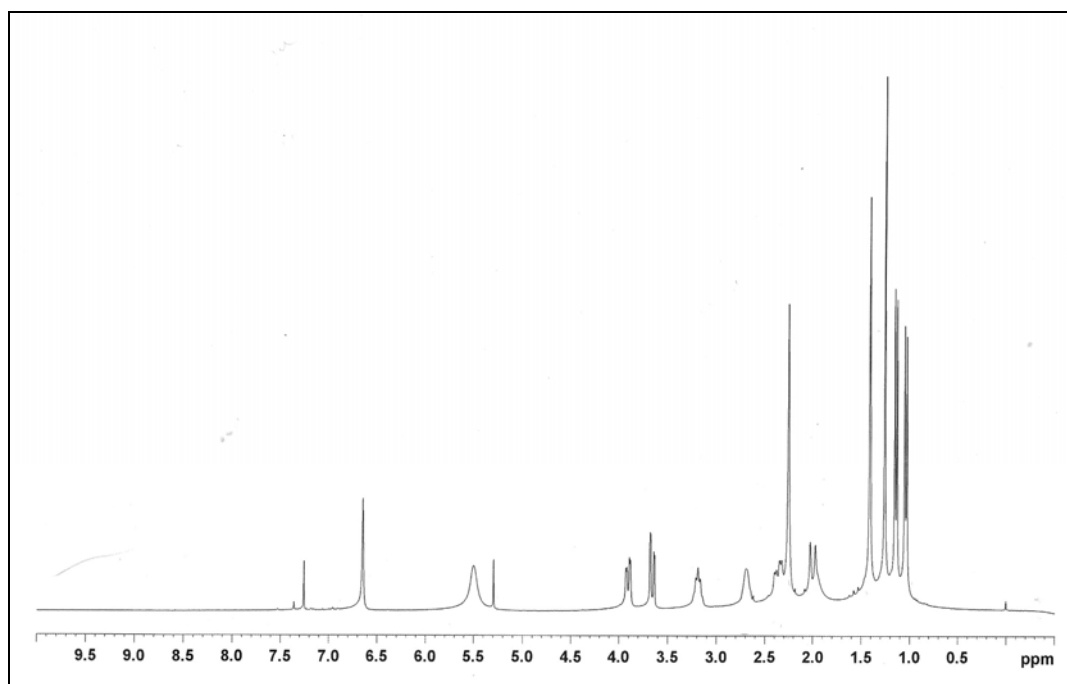
**Figure 87**  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **TP12**



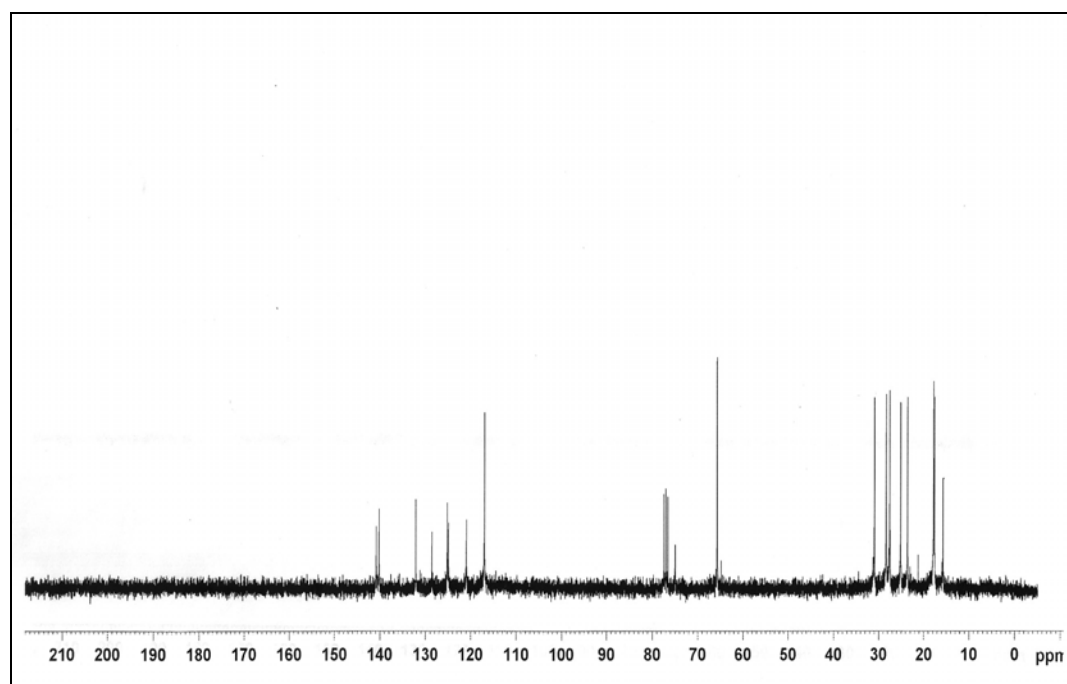
**Figure 88** UV (MeOH) spectrum of compound **TP13**



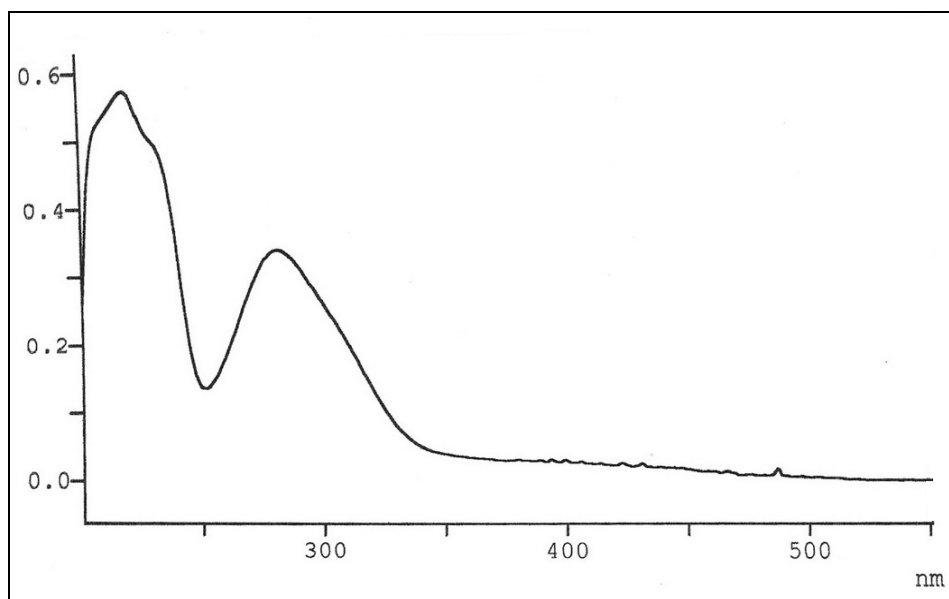
**Figure 89** IR (neat) spectrum of compound **TP13**



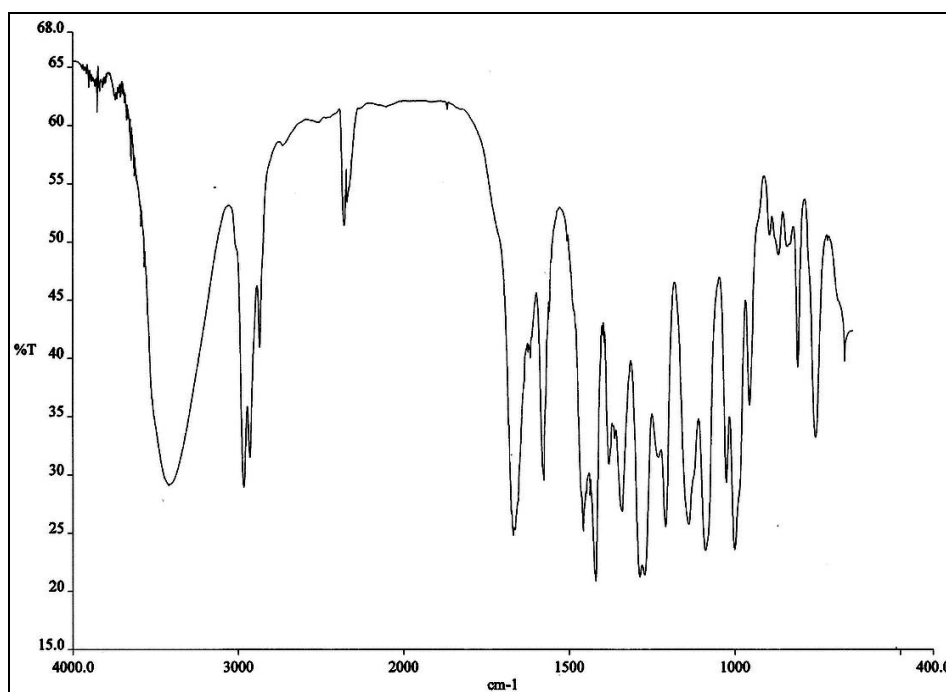
**Figure 90**  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **TP13**



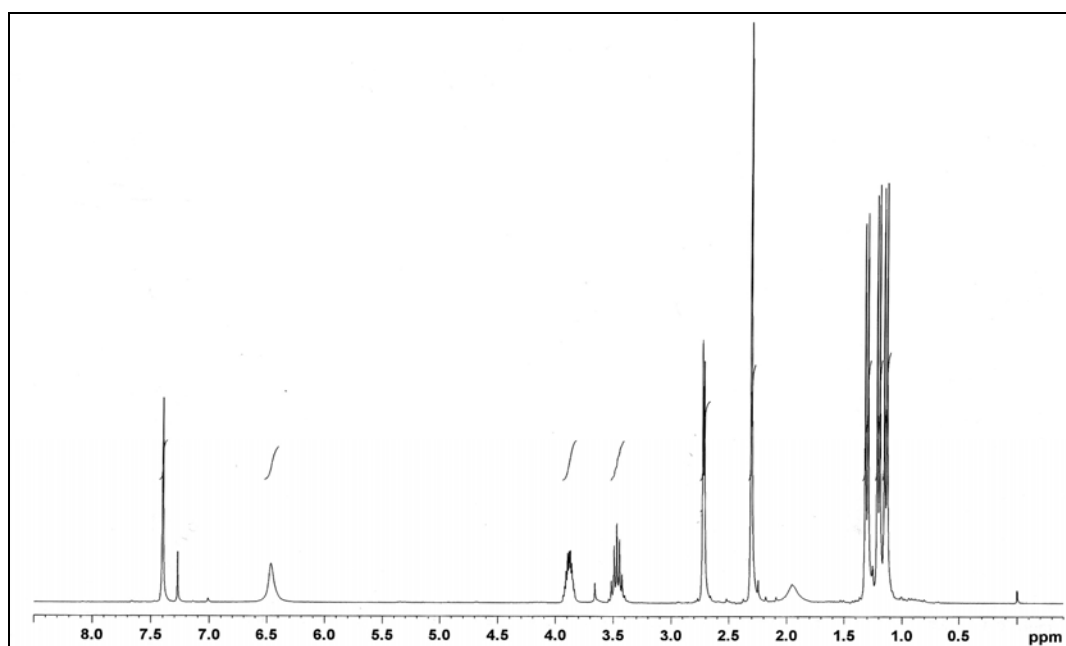
**Figure 91**  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **TP13**



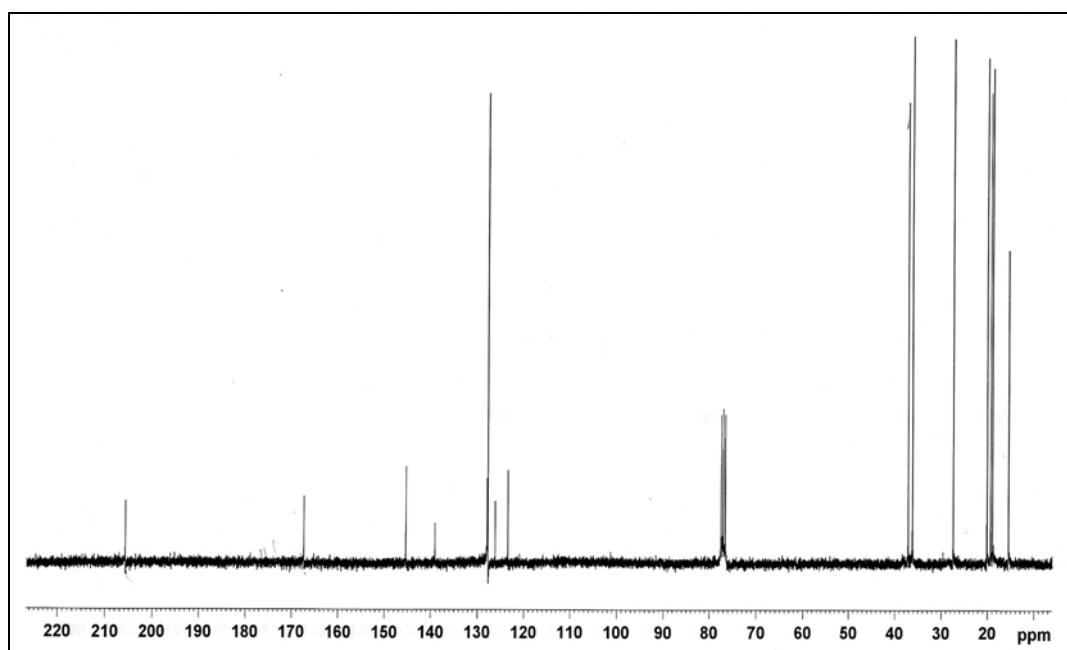
**Figure 92** UV (MeOH) spectrum of compound **TP14**



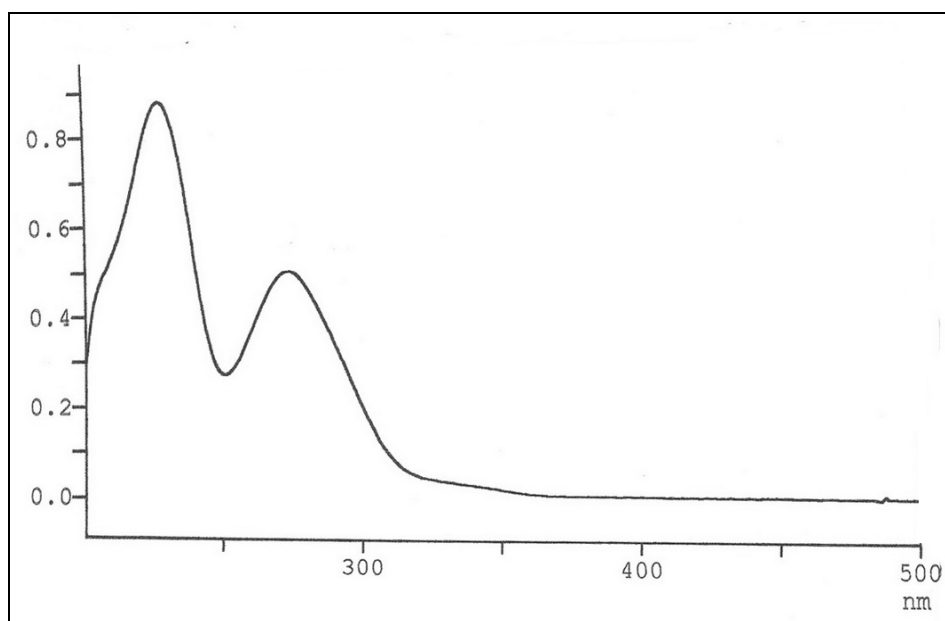
**Figure 93** IR (neat) spectrum of compound **TP14**



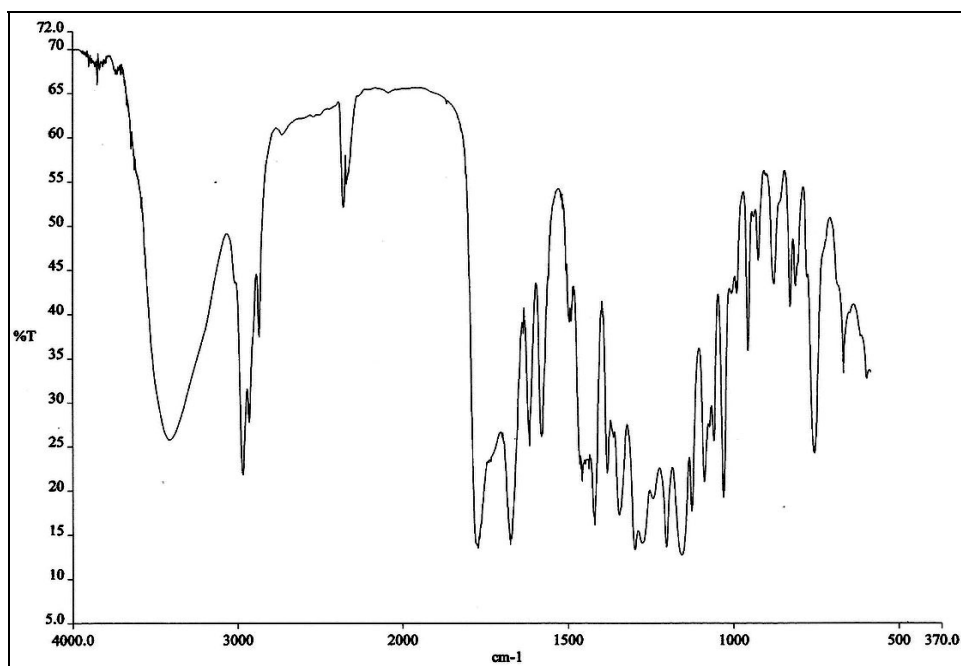
**Figure 94**  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **TP14**



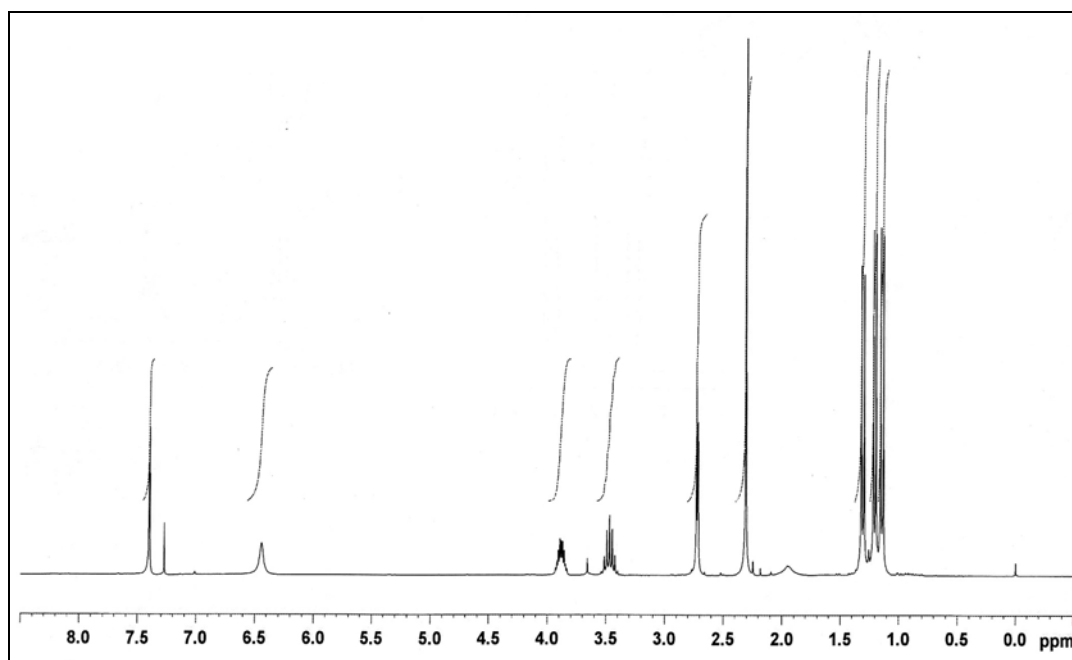
**Figure 95**  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **TP14**



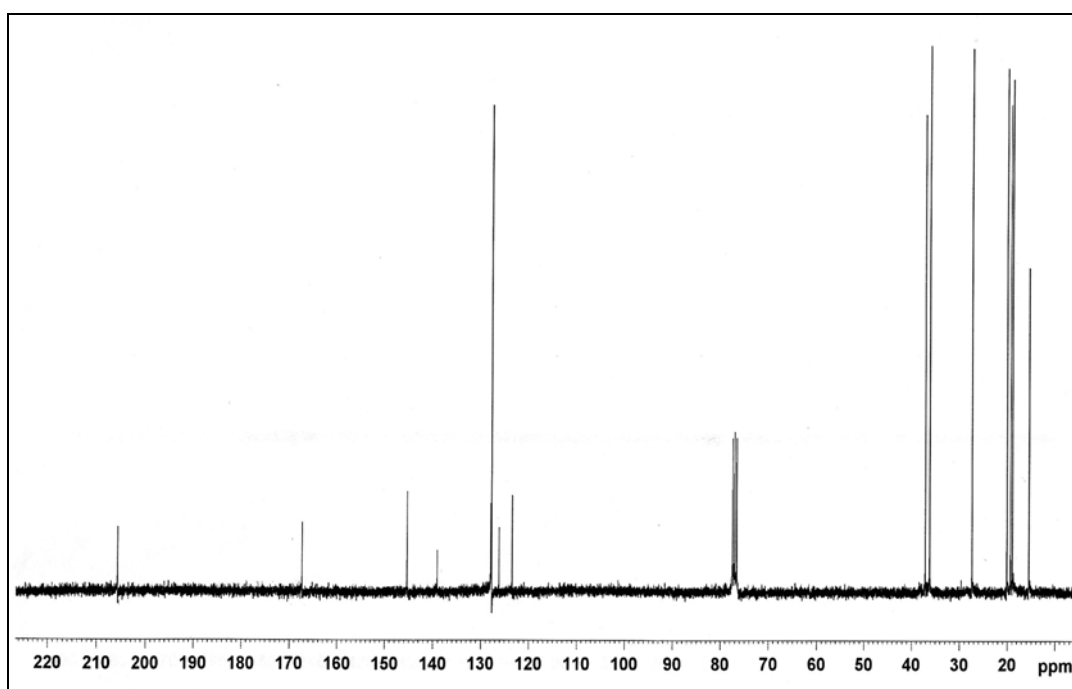
**Figure 96** UV (MeOH) spectrum of compound **TP15**



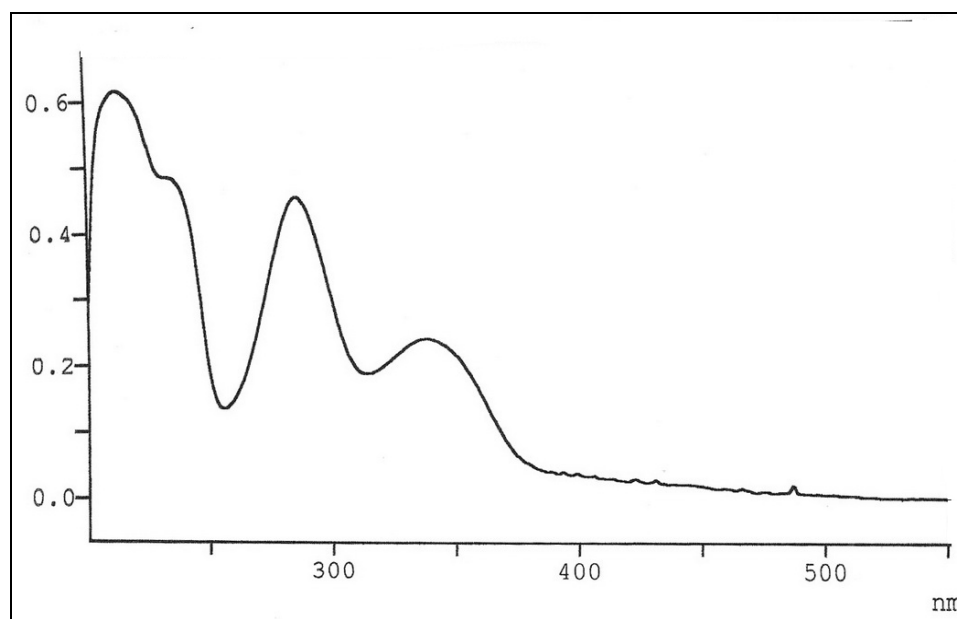
**Figure 97** IR (neat) spectrum of compound **TP15**



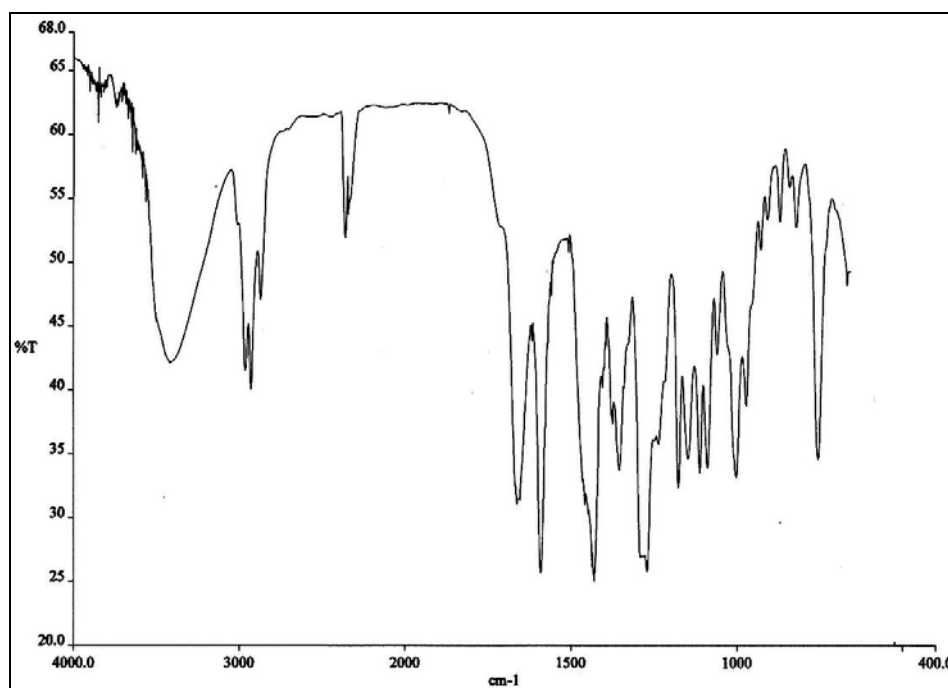
**Figure 98**  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **TP15**



**Figure 99**  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **TP15**

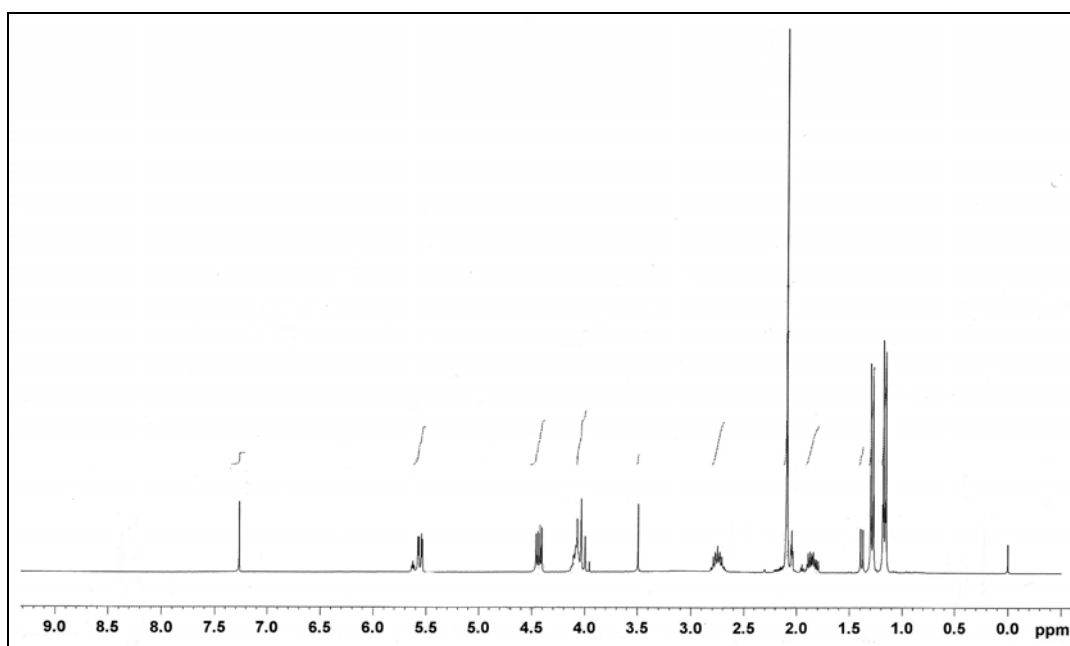


**Figure 100** UV (MeOH) spectrum of compound TP16

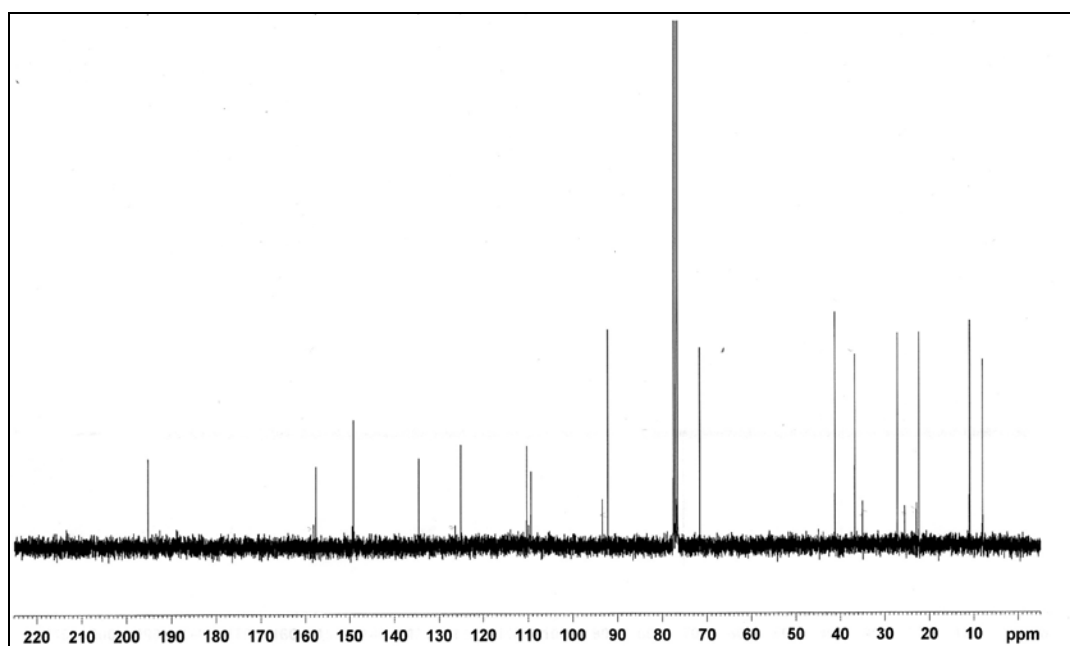


**Figure 101** IR (neat) spectrum of compound TP16

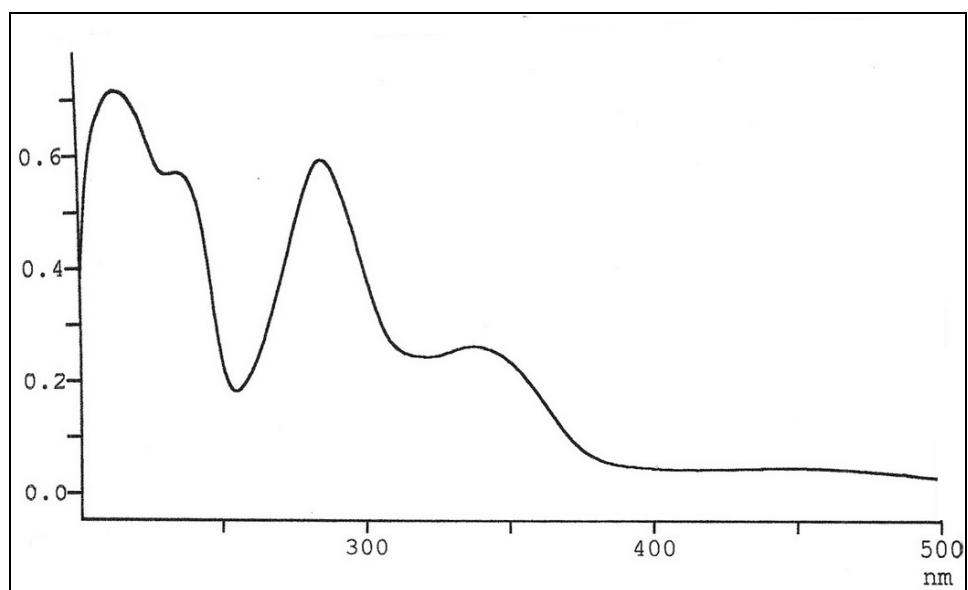




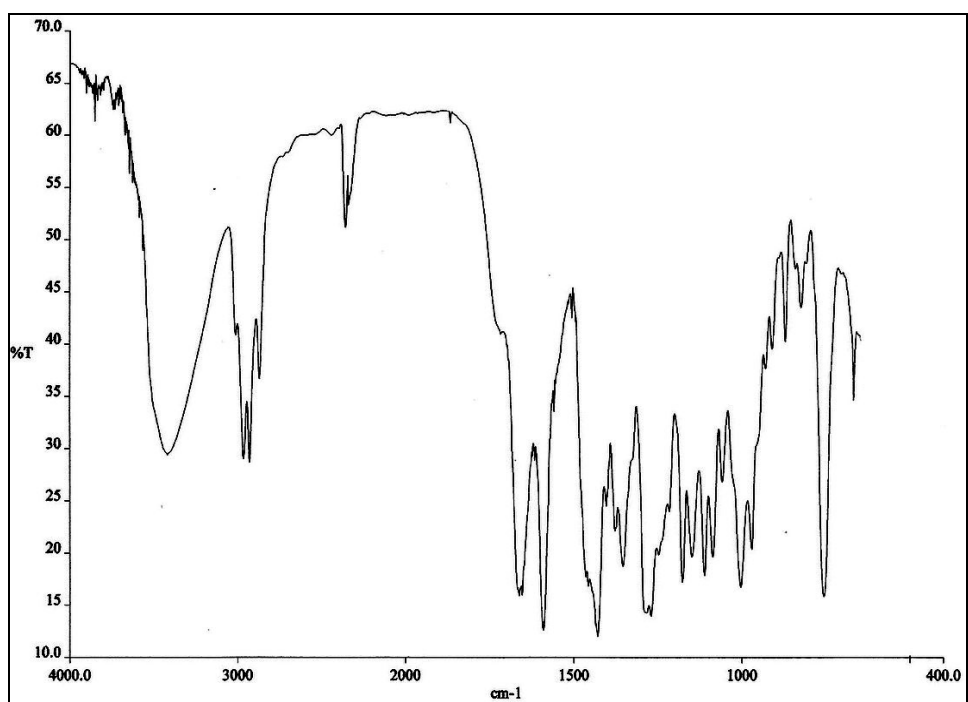
**Figure 102**  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **TP16**



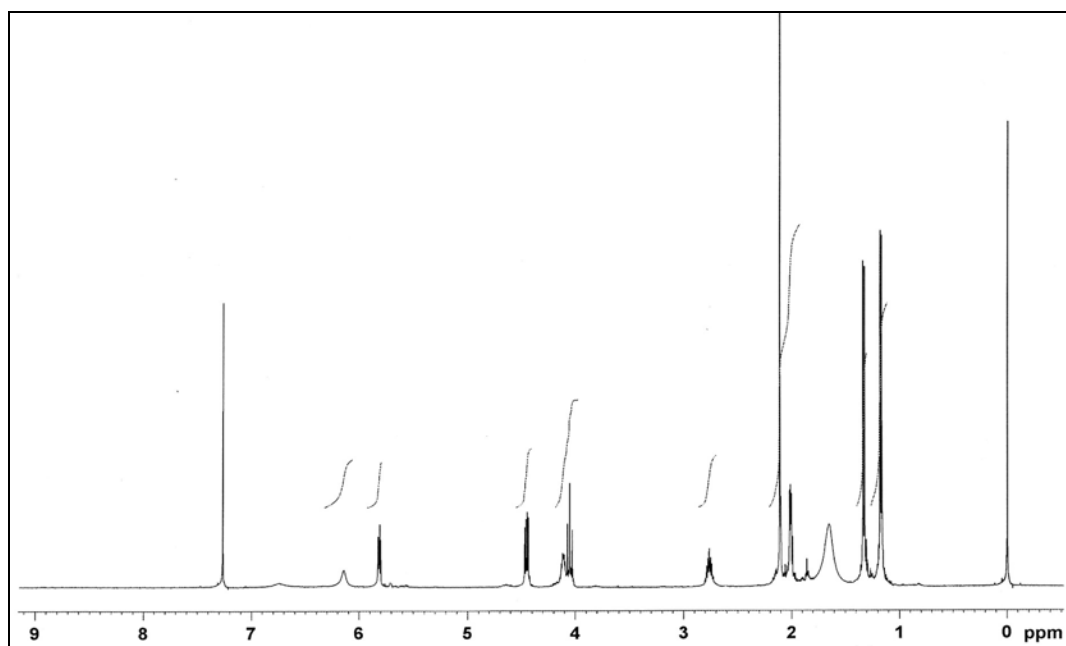
**Figure 103**  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **TP16**



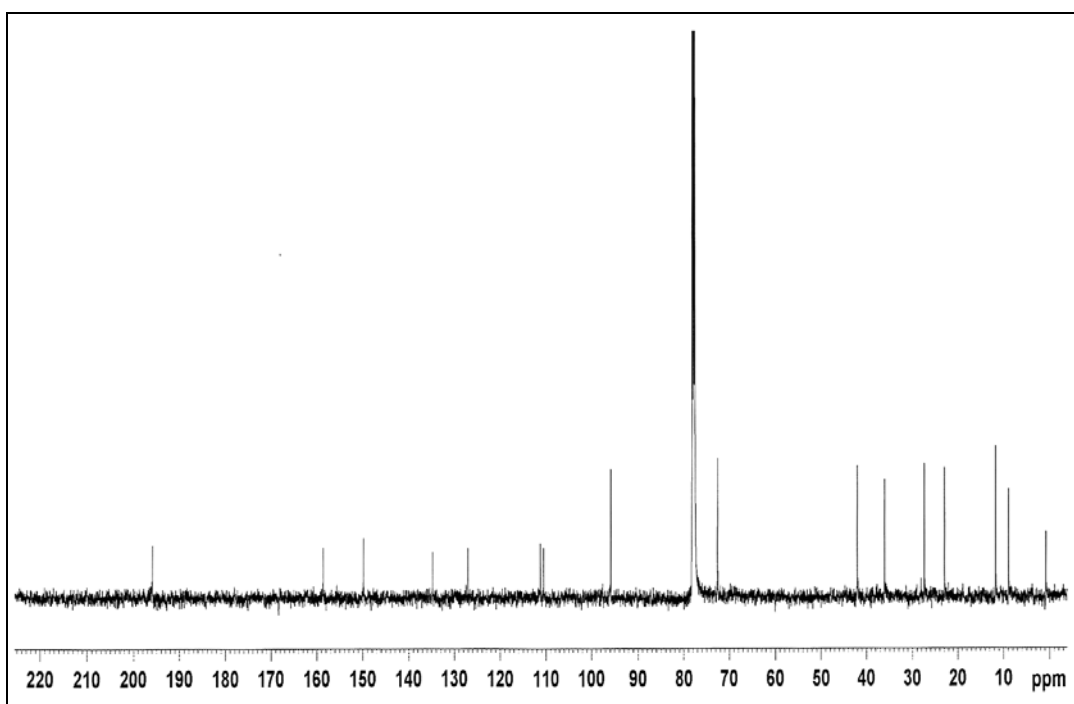
**Figure 104** UV (MeOH) spectrum of compound TP17



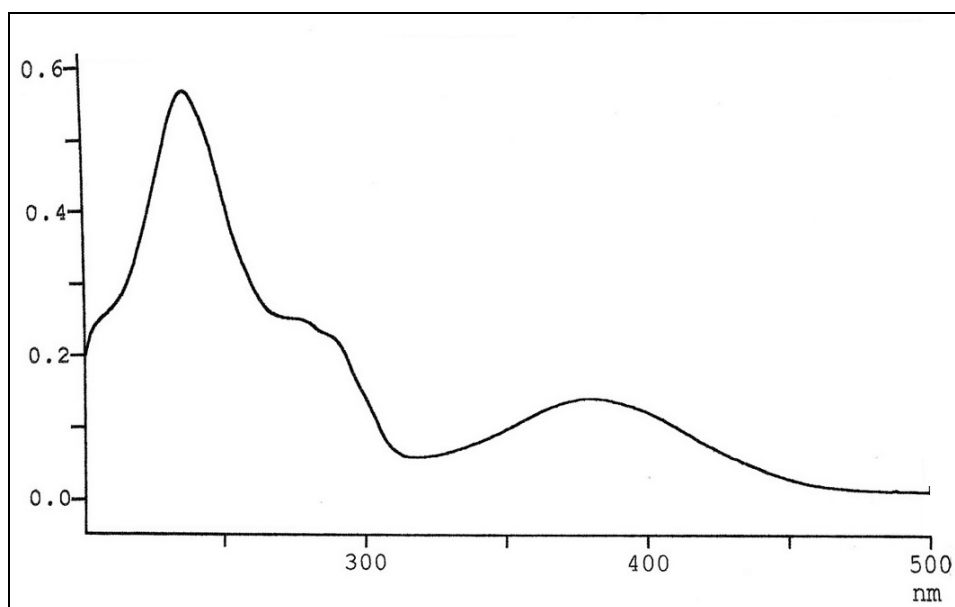
**Figure 105** IR (neat) spectrum of compound TP17



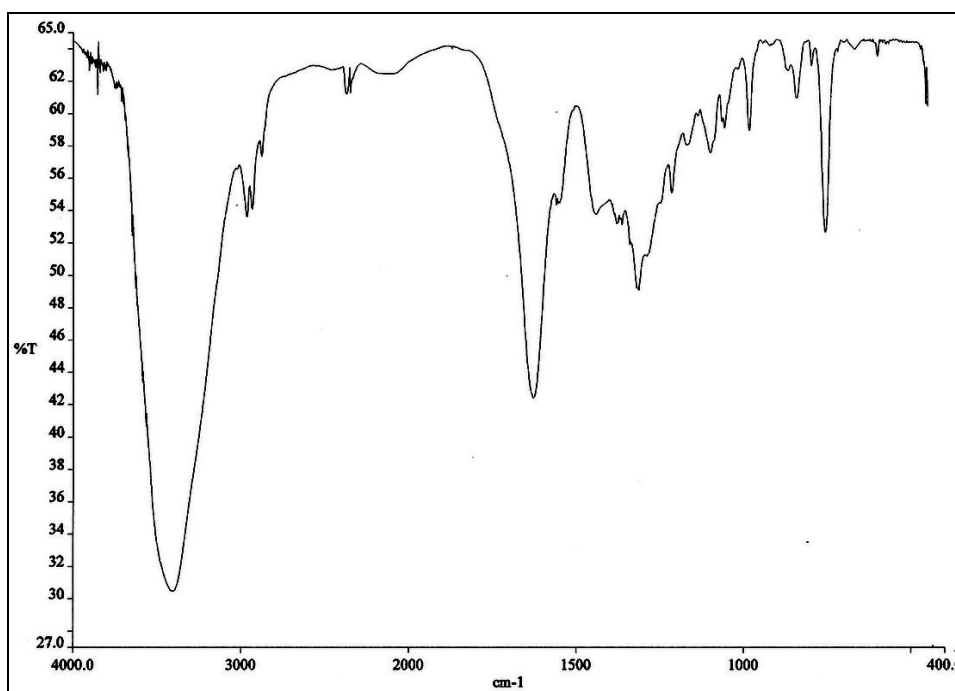
**Figure 106**  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **TP17**



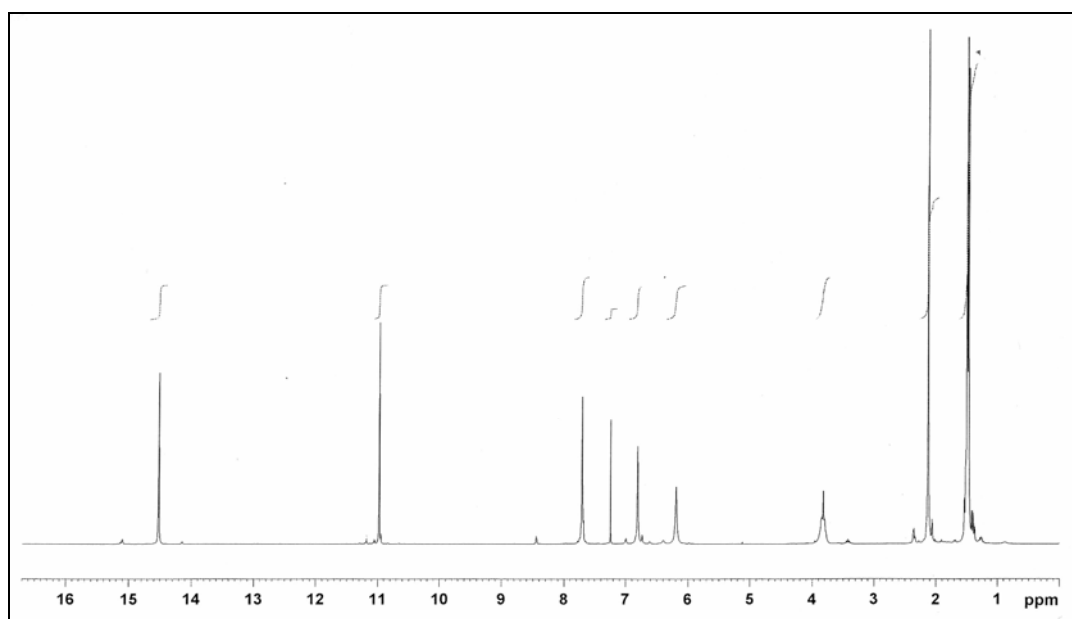
**Figure 107**  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **TP17**



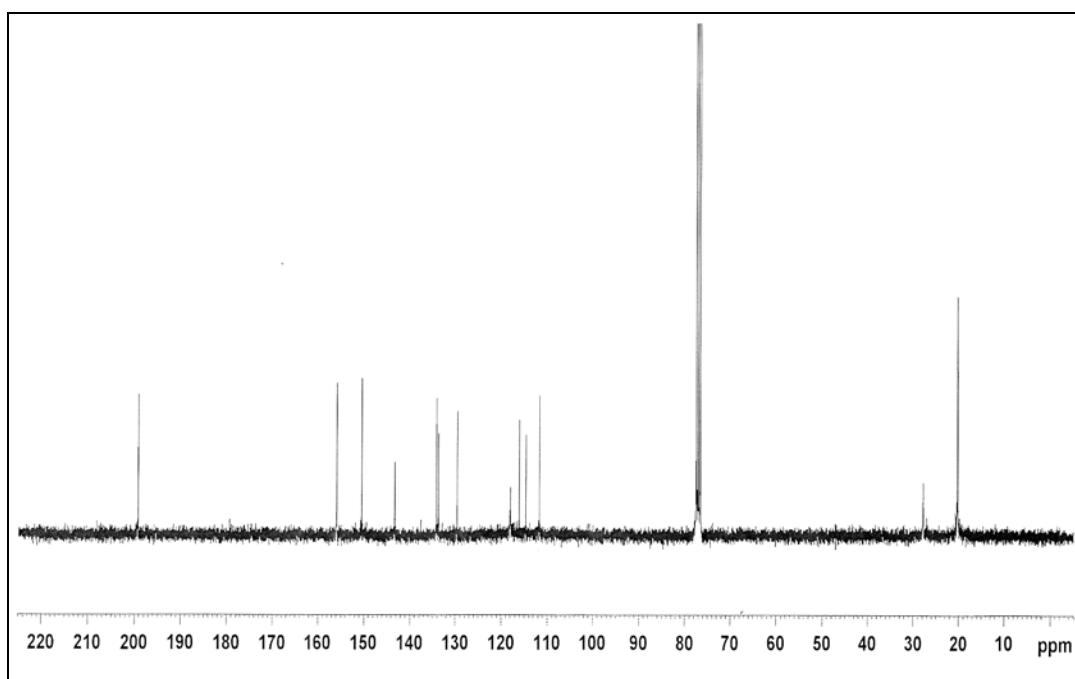
**Figure 108** UV (MeOH) spectrum of compound TP18



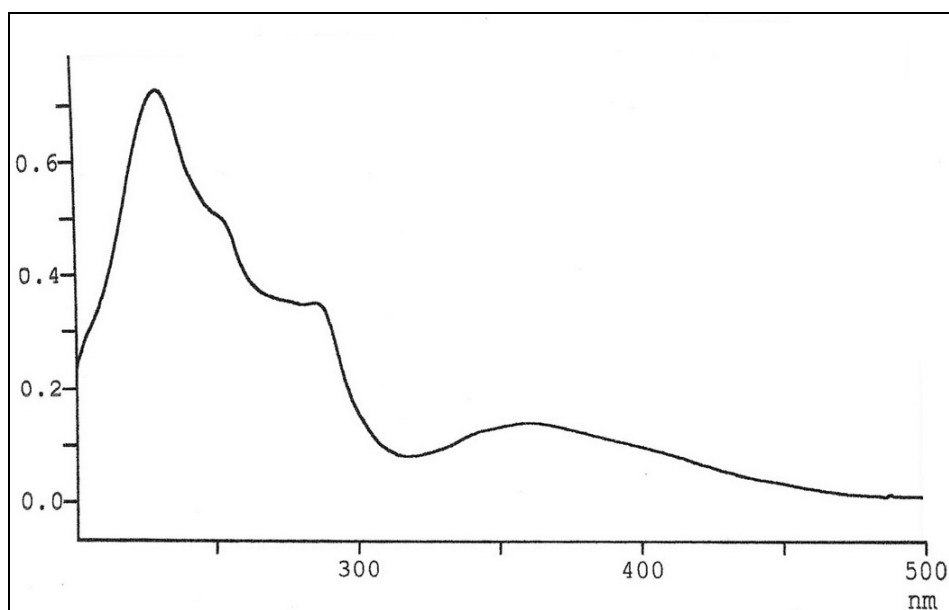
**Figure 109** IR (neat) spectrum of compound TP18



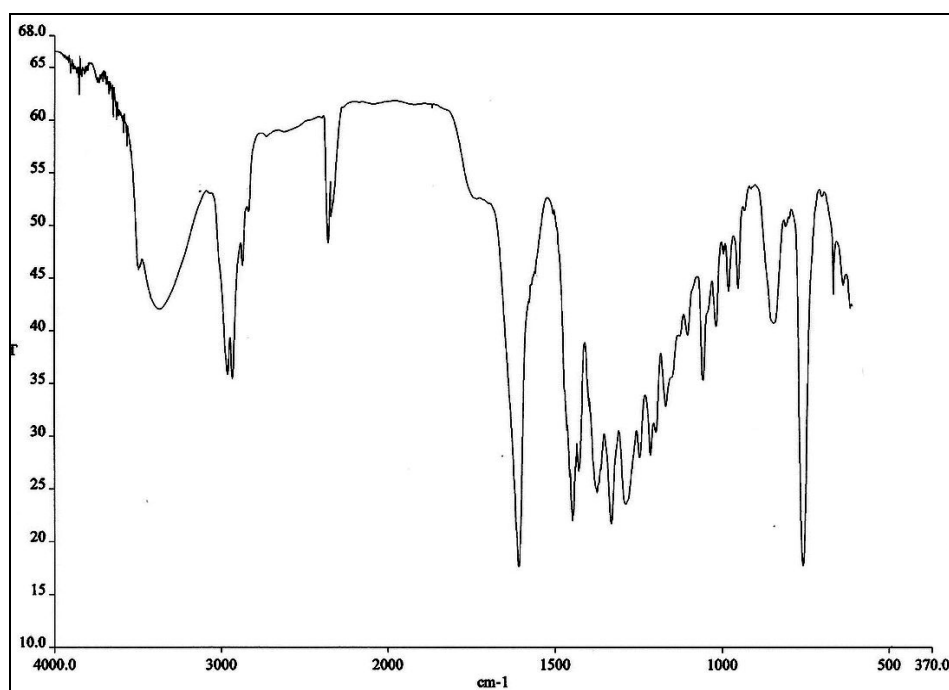
**Figure 110**  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **TP18**



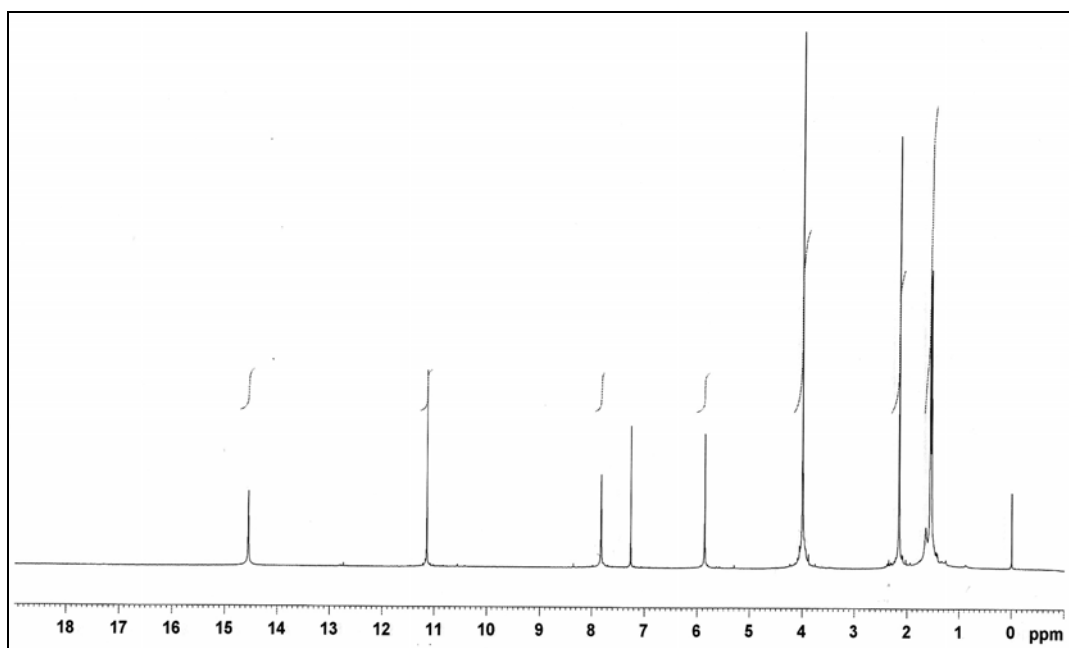
**Figure 111**  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **TP18**



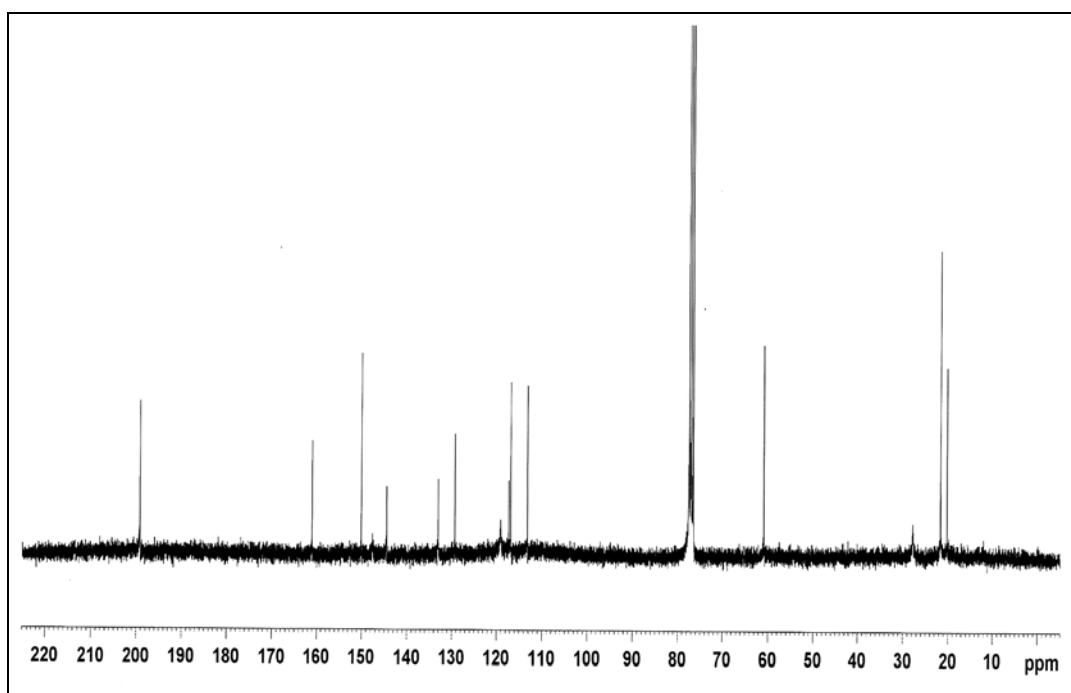
**Figure 112** UV (MeOH) spectrum of compound **TP19**



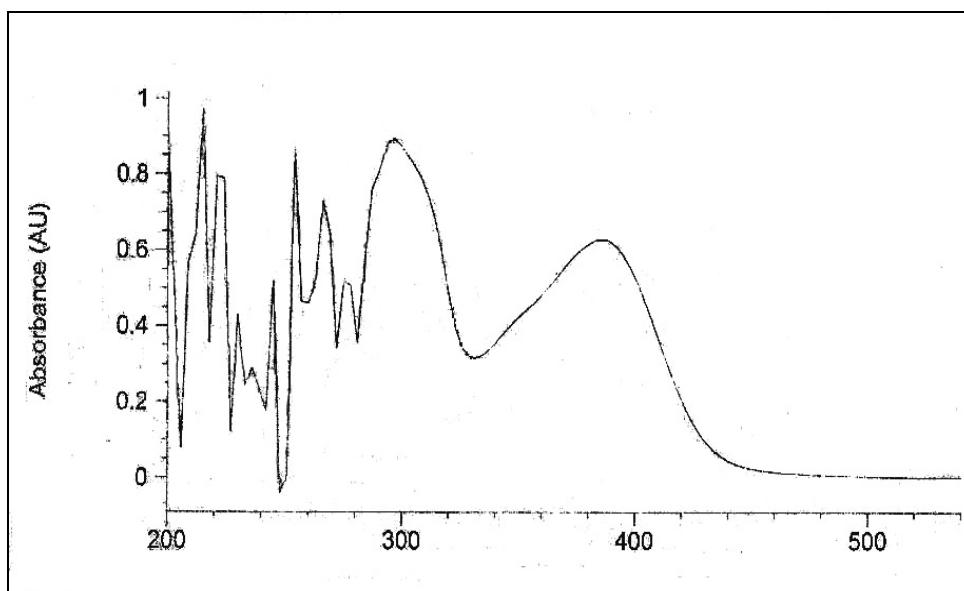
**Figure 113** IR (neat) spectrum of compound **TP19**



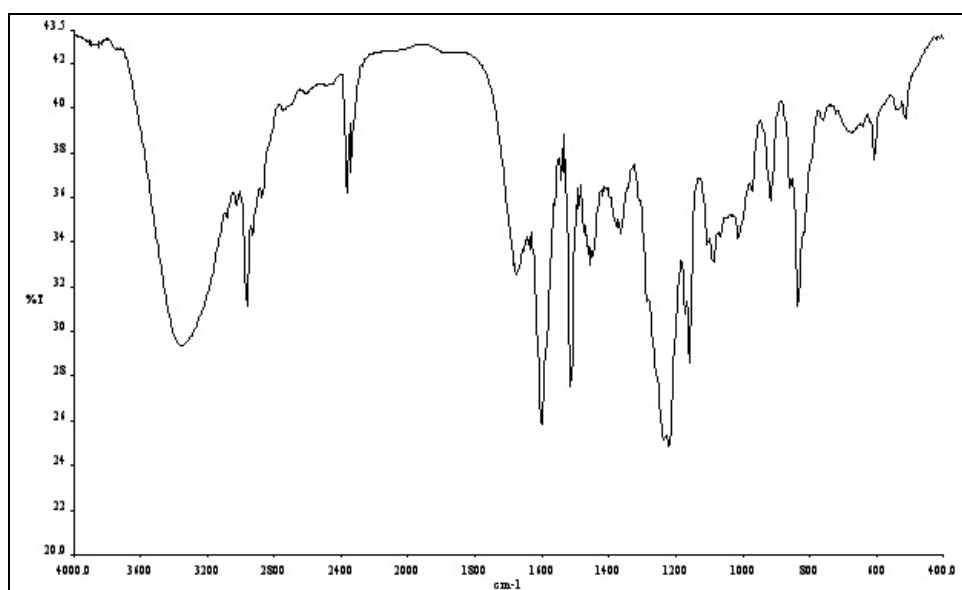
**Figure 114**  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **TP19**



**Figure 115**  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **TP19**

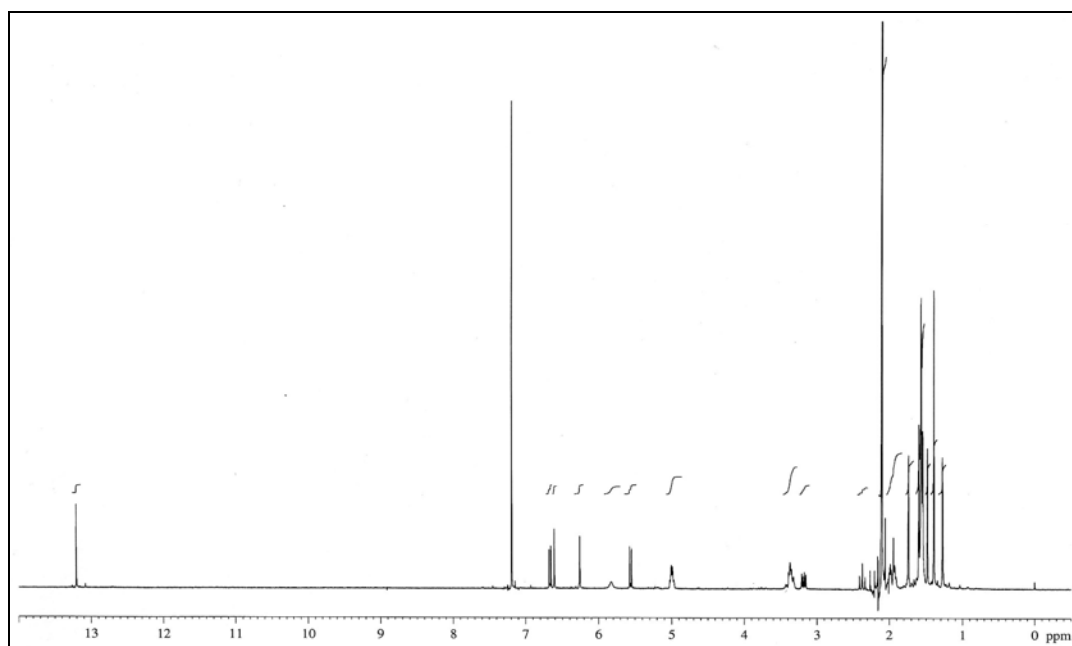


**Figure 116** UV (MeOH) spectrum of compound **AI1**

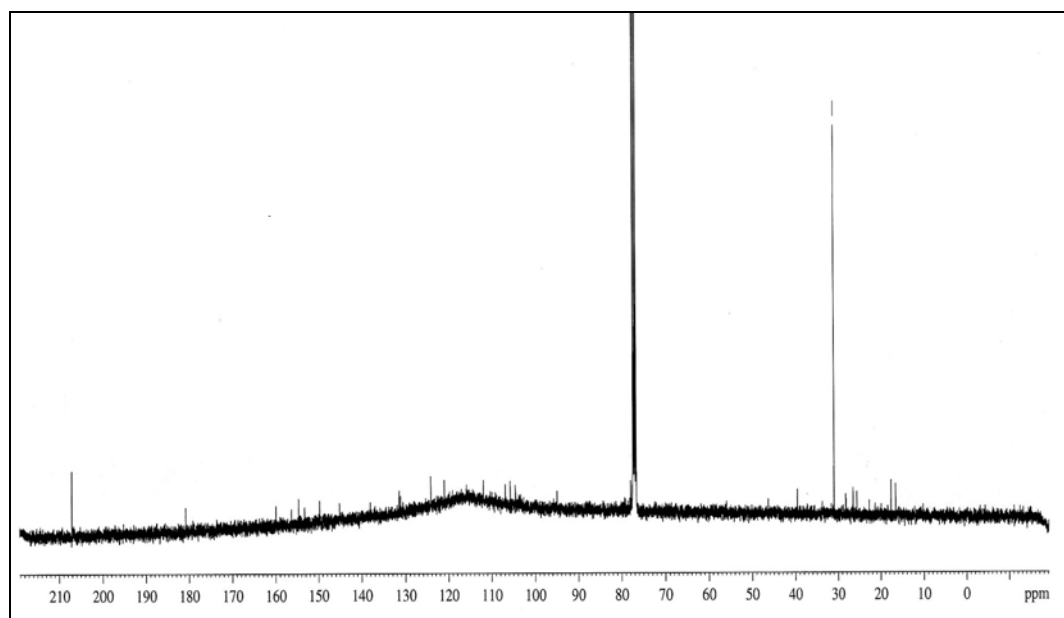


**Figure 117** IR (KBr) spectrum of compound **AI1**

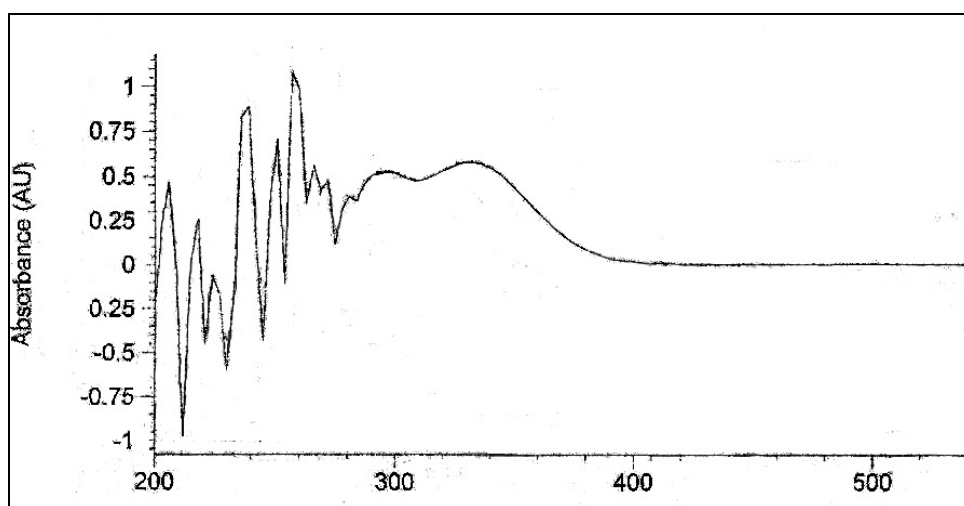




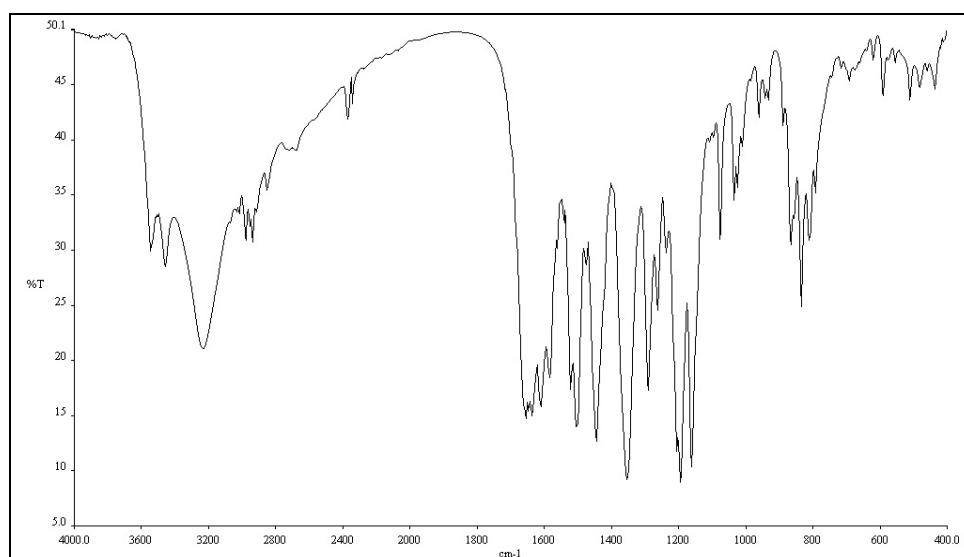
**Figure 118**  $^1\text{H}$  NMR (400 MHz) ( $\text{CDCl}_3$ ) spectrum of compound AI1



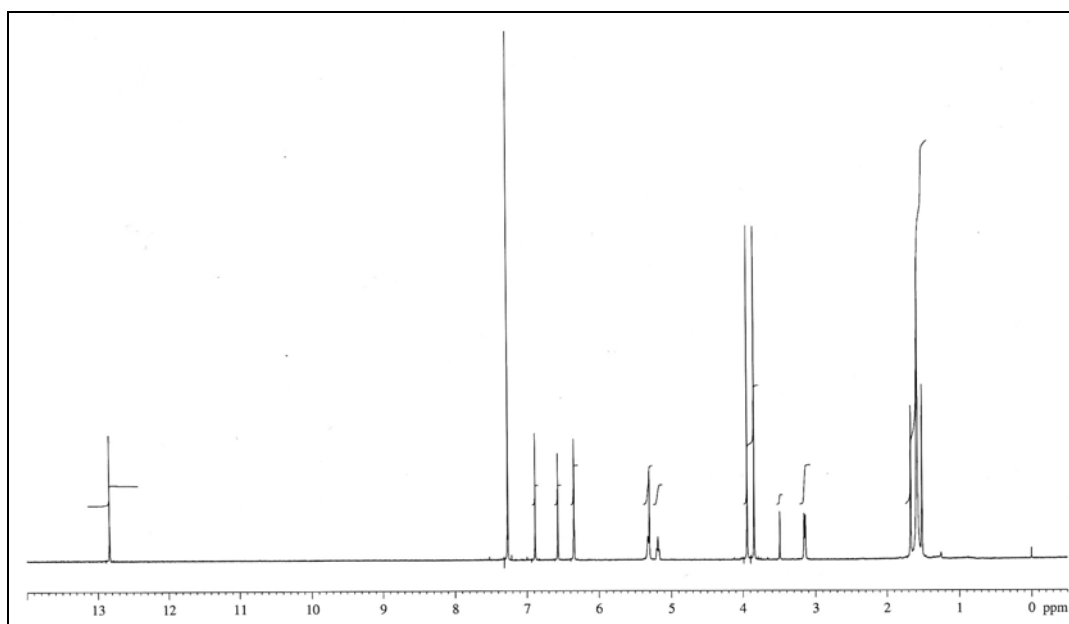
**Figure 119**  $^{13}\text{C}$  NMR (100 MHz) ( $\text{CDCl}_3$ ) spectrum of compound AI1



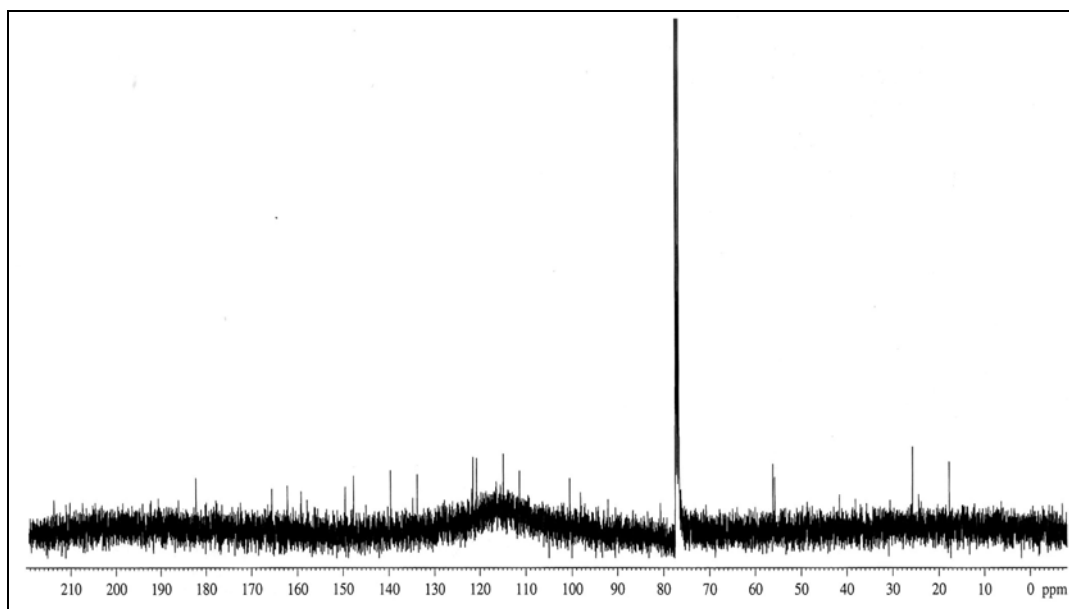
**Figure 120** UV (MeOH) spectrum of compound AI2



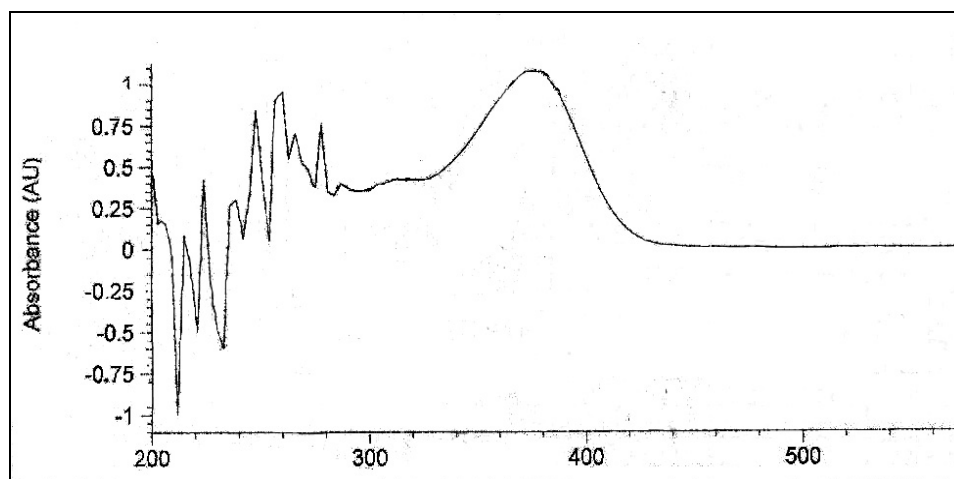
**Figure 121** IR (KBr) spectrum of compound AI2



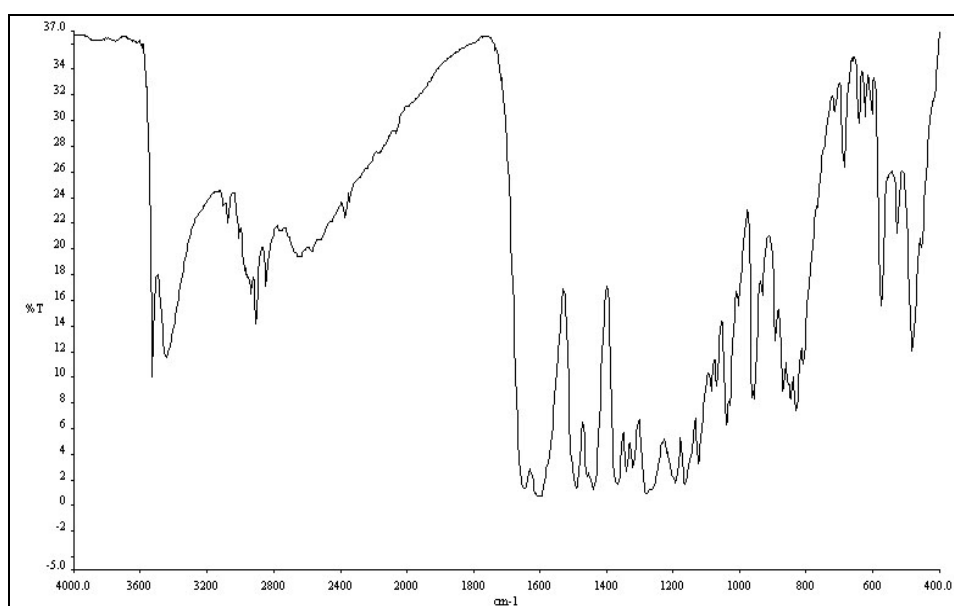
**Figure 122**  $^1\text{H}$  NMR (400 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **AI2**



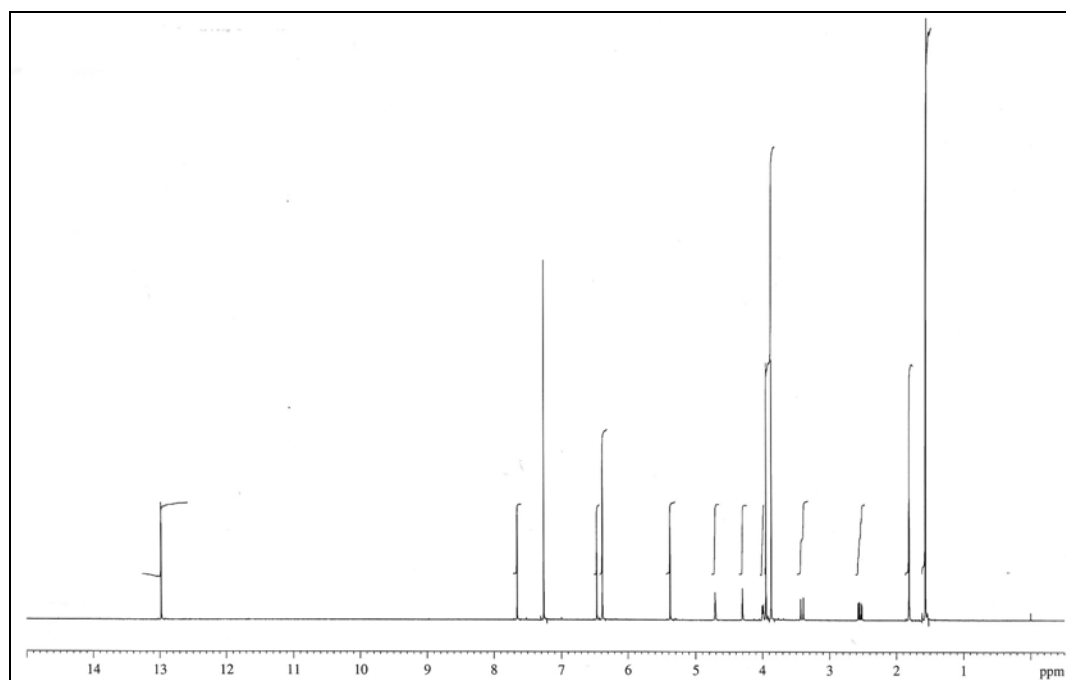
**Figure 123**  $^{13}\text{C}$  NMR (100 MHz) ( $\text{CDCl}_3$ ) spectrum of compound **AI2**



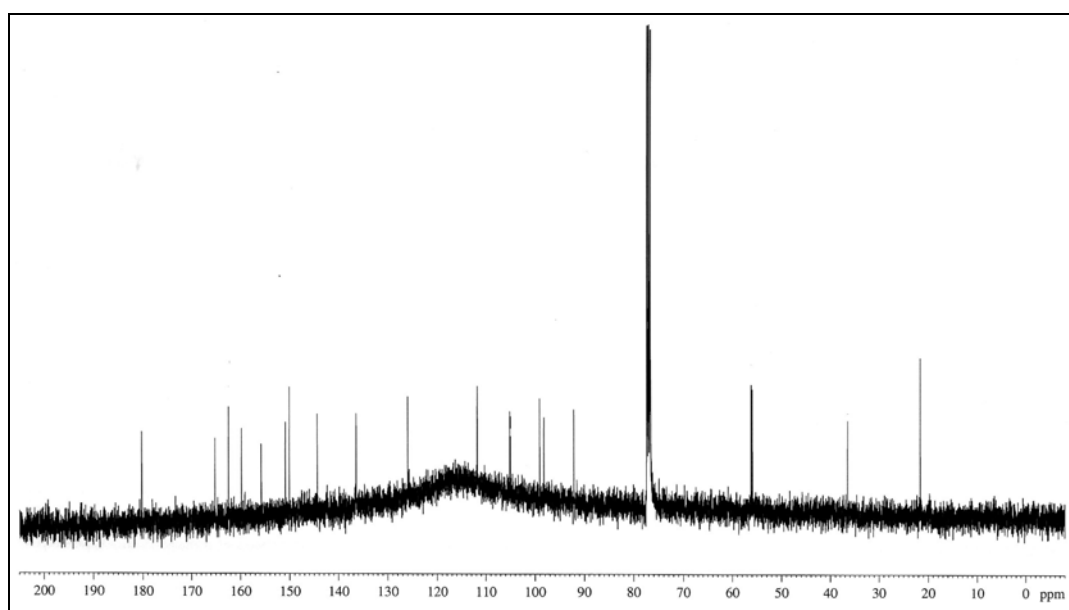
**Figure 124** UV (MeOH) spectrum of compound **AI3**



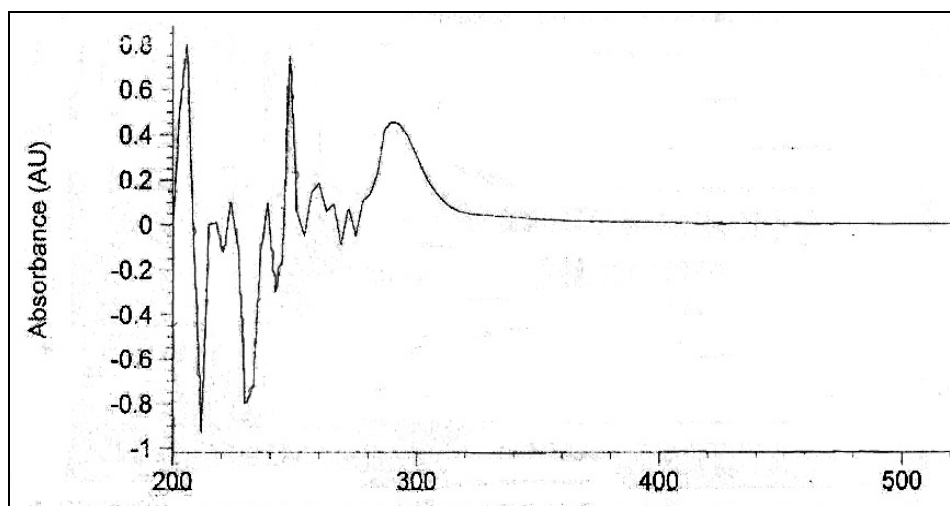
**Figure 125** IR (KBr) spectrum of compound **AI3**



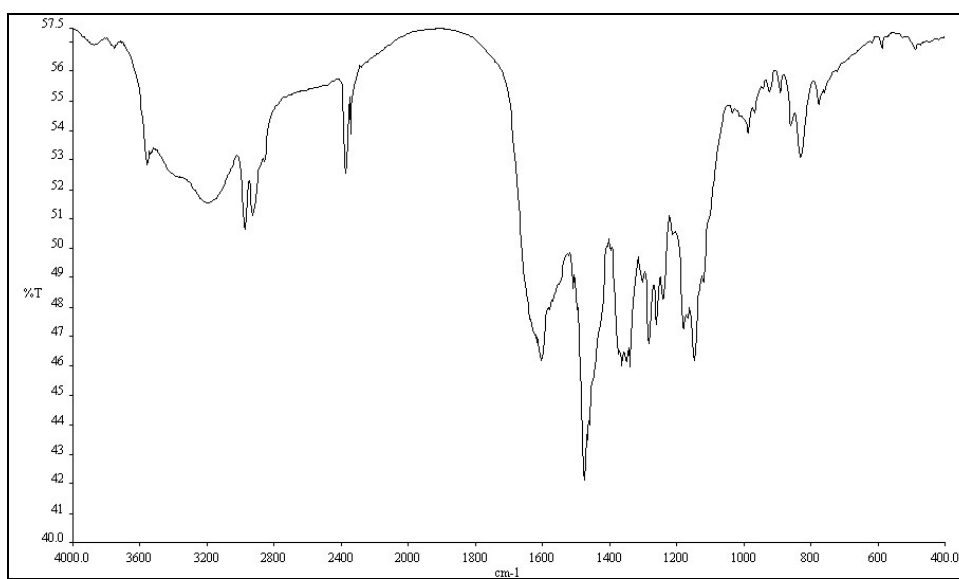
**Figure 126**  $^1\text{H}$  NMR (400 MHz) ( $\text{CDCl}_3$ ) spectrum of compound AI3



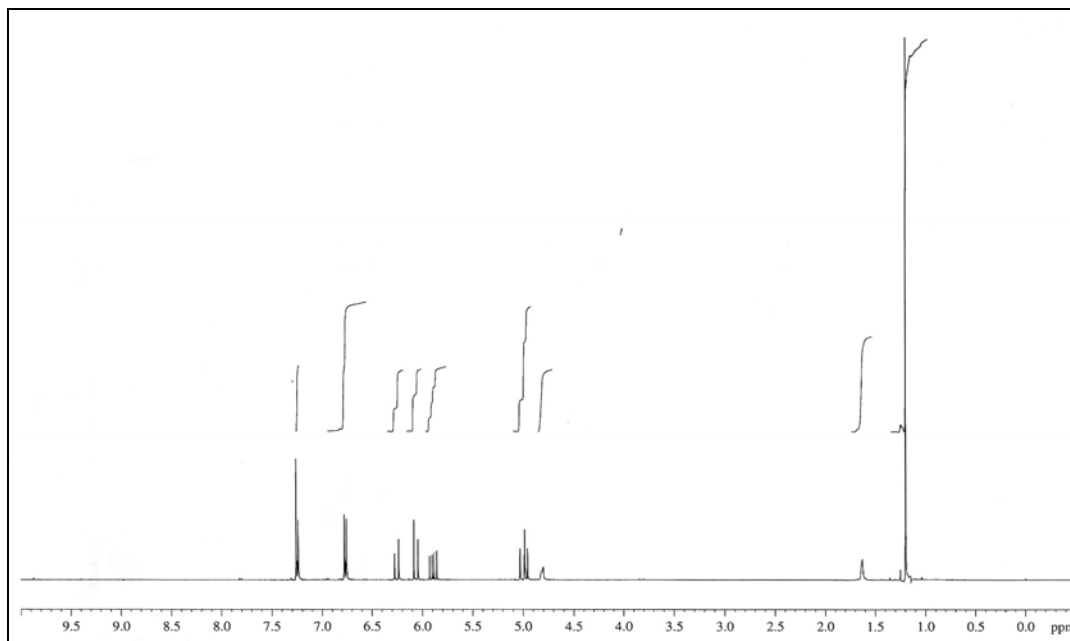
**Figure 127**  $^{13}\text{C}$  NMR (100 MHz) ( $\text{CDCl}_3$ ) spectrum of compound AI3



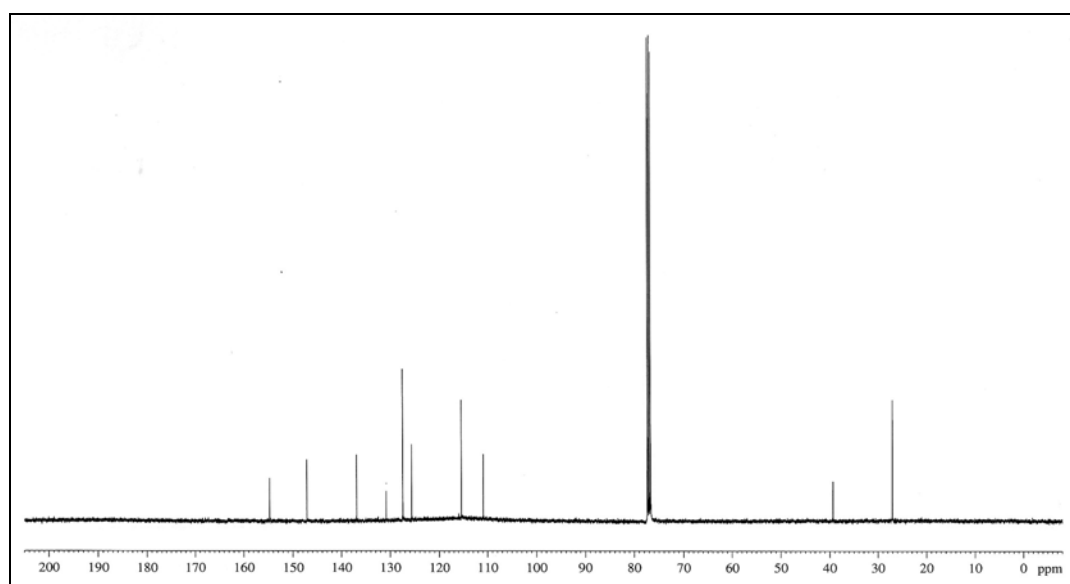
**Figure 128** UV (MeOH) spectrum of compound AI4



**Figure 129** IR (KBr) spectrum of compound AI4



**Figure 130**  $^1\text{H}$  NMR (400 MHz) ( $\text{CDCl}_3$ ) spectrum of compound AI4



**Figure 131**  $^{13}\text{C}$  NMR (100 MHz) ( $\text{CDCl}_3$ ) spectrum of compound AI4

## VITAE

**NAME** Mr. Sompong Boonsri

**Student ID** 4723011

### Educational Attainment

Degree	Name of Institution	Year of Graduate
B. Sc. (Chemistry)	Rajabhat Phuket University	1999
M. Sc. (Organic Chemistry)	Prince of Songkla University	2003

### Scholarship Awards during Enrolment

Scholarship was awarded by the Royal Golden Jubilee Ph. D. Program of the Thailand Research Fund, the Higher Education Development Project: Center for innovation in Chemistry: Postgraduate Education and Research Program in Chemistry (PERCH-CIC), the Commission and Higher Education (CHE-RES-RG), the Directed Basic Research in Medicinal Chemistry (Thailand Research Fund) and the Graduate School, Prince of Songkla University.

### List of Publication and proceedings

#### Publications

- Boonsri, S; Karalai, C.; Ponglimanont, C.; Chantrapromma, S.; Kanjana-opas, A. 2008. Cytotoxic and antibacterial sesquiterpenes from *Thespesia populnea*. *J. Nat. Prod.* 71, 1173-1177.
- Boonsri, S.; Chantrapromma, S.; Fun, H.-K.; Karalai, C. 2007. 1,5,8-Trimethyl-1,2-dihydronaphtho[2,1-b]furan-6,7-dione. *Acta Crystallographica* E63, o4901/1-o4901/10.