



**Chemical Constituents from the Twigs of**  
*Garcinia parvifolia*

**Wanpen Naklue**

**A Thesis Submitted in Partial Fulfillment of the Requirements  
for the Degree of Master of Science in Organic Chemistry**

**Prince of Songkla University**

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**Author**             Miss Wanpen Naklue  
**Major Program**   Organic Chemistry

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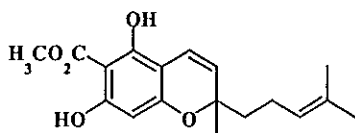
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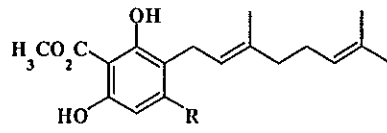
ชื่อวิทยานิพนธ์	องค์ประกอบทางเคมีจากกิ่งชะมวงเล็ก ( <i>Garcinia parvifolia</i> )
ผู้เขียน	นางสาววันเพ็ญ นากลื้อ
สาขาวิชา	เคมีอินทรีย์
ปีการศึกษา	2548

### บทคัดย่อ

ส่วนสกัดหยาบเมธานอลของกิ่งชะมวงเล็ก เมื่อนำมาแยกและทำให้บริสุทธิ์ด้วยวิธีทางโครมาโทกราฟี สามารถแยกสารใหม่ 13 สาร ได้แก่สารประเภท methyl benzoate จำนวน 2 สาร (GP2 และ GP7) สารประเภท benzopyran จำนวน 5 สาร (GP1, GP3, GP4, GP5 และ GP6) สารประเภท depsidone จำนวน 2 สาร (GP12 และ GP14) สารประเภท xanthone จำนวน 3 สาร (GP8, GP9 และ GP11) และสารประเภท benzocyclooctene จำนวน 1 สาร (GP10) รวมทั้งสารที่ทราบโครงสร้างแล้วจำนวน 7 สาร ได้แก่สารประเภท depsidone จำนวน 1 สาร (GP18) สารประเภท xanthone จำนวน 5 สาร (GP15, GP16, GP17, GP19 และ GP20) และสารอนุพันธ์ของ cyclohexenone จำนวน 1 สาร (GP13) โครงสร้างของสารทั้งหมดวิเคราะห์โดยใช้ข้อมูลทางสเปกโทรสโกปี โดยเฉพาะ 1D และ 2D NMR สเปกโทรสโกปี และการเปรียบเทียบข้อมูลกับสารที่มีการรายงานโครงสร้างมาแล้ว

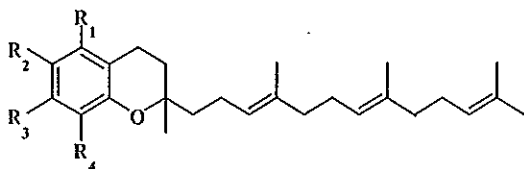


GP1



GP2: R = OCH<sub>3</sub>

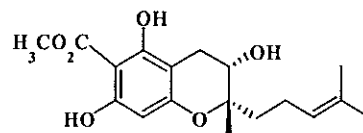
GP7: R = OH



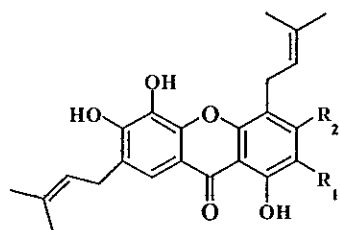
GP3: R<sub>1</sub> = H, R<sub>2</sub> = R<sub>3</sub> = CH<sub>3</sub>, R<sub>4</sub> = OH

GP4: R<sub>1</sub> = R<sub>4</sub> = CH<sub>3</sub>, R<sub>2</sub> = H, R<sub>3</sub> = OH

GP5: R<sub>1</sub> = R<sub>3</sub> = H, R<sub>2</sub> = CH<sub>3</sub>, R<sub>4</sub> = OH

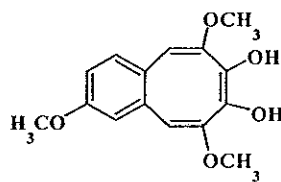


GP6

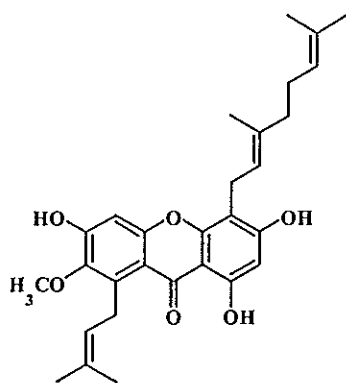


GP8:  $R_1 = H, R_2 = OCH_3$

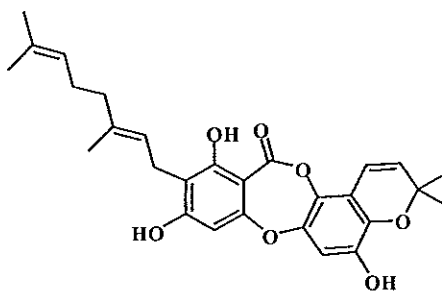
GP9:  $R_1 = \text{prenyl}, R_2 = OH$



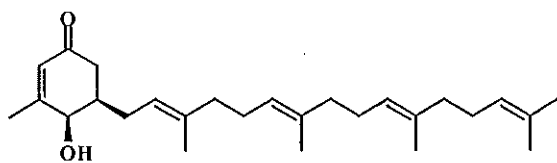
GP10



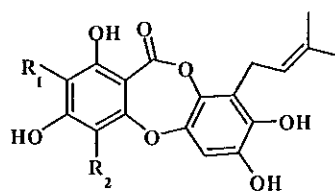
GP11



GP12

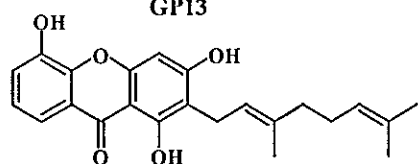


GP13

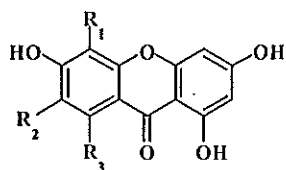


GP14:  $R_1 = H, R_2 = \text{prenyl}$

GP18:  $R_1 = \text{prenyl}, R_2 = H$



GP15



GP16:  $R_1 = H, R_2 = OCH_3, R_3 = \text{prenyl}$

GP17:  $R_1 = H, R_2 = OCH_3, R_3 = \text{prenyl}$

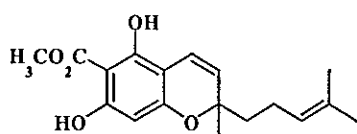
GP19:  $R_1 = OH, R_2 = R_3 = H$

GP20:  $R_1 = R_3 = OH, R_2 = OH$

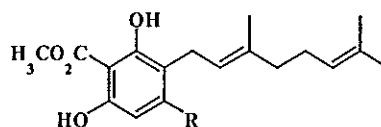
**Thesis Title**            Chemical Constituents from the Twigs of *Garcinia parvifolia*  
**Author**                    Miss Wanpen Naklue  
**Major Program**        Organic Chemistry  
**Academic Year**        2005

### ABSTRACT

The crude methanol extract from the twigs of *Garcinia parvifolia* was separated by chromatographic methods to yield thirteen new compounds: two methyl benzoates (GP2 and GP7), five benzopyrans (GP1, GP3, GP4, GP5 and GP6), two depsidones (GP12 and GP14), three xanthenes (GP8, GP9 and GP11) and one benzocyclooctene (GP10) together with seven known compounds: one depsidone (GP18), five xanthenes (GP15, GP16, GP17, GP19 and GP20) and one cyclohexenone derivative (GP13). The structures were determined by analysis of 1D and 2D NMR spectroscopic data and comparison of the NMR data with those reported in literature.

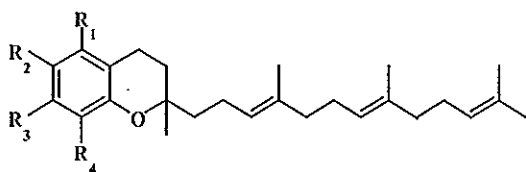


GP1



GP2: R = OCH<sub>3</sub>

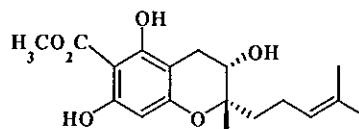
GP7: R = OH



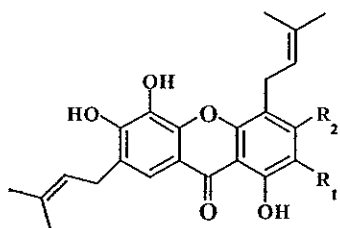
GP3: R<sub>1</sub> = H, R<sub>2</sub> = R<sub>3</sub> = CH<sub>3</sub>, R<sub>4</sub> = OH

GP4: R<sub>1</sub> = R<sub>4</sub> = CH<sub>3</sub>, R<sub>2</sub> = H, R<sub>3</sub> = OH

GP5: R<sub>1</sub> = R<sub>3</sub> = H, R<sub>2</sub> = CH<sub>3</sub>, R<sub>4</sub> = OH

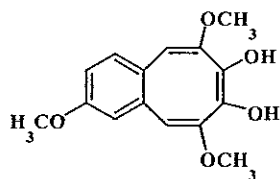


GP6

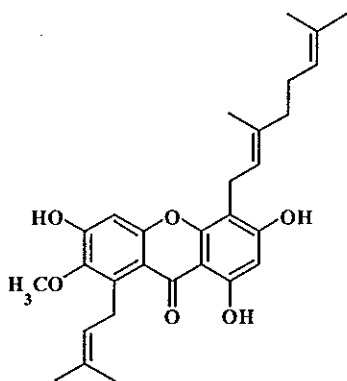


GP8:  $R_1 = H, R_2 = OCH_3$

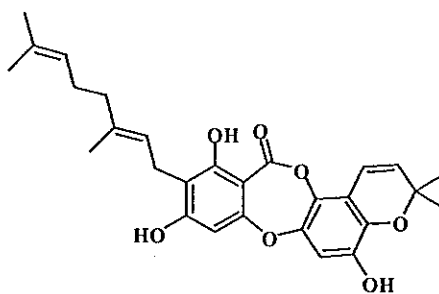
GP9:  $R_1 = \text{isopentenyl}, R_2 = OH$



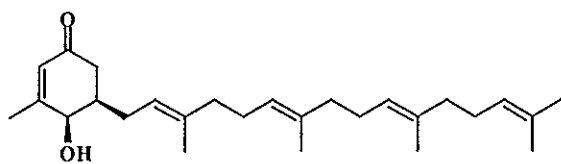
GP10



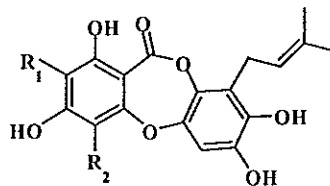
GP11



GP12

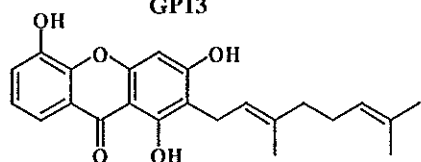


GP13

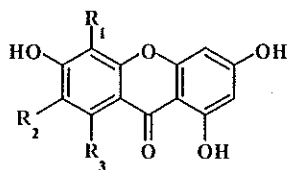


GP14:  $R_1 = H, R_2 = \text{isopentenyl}$

GP18:  $R_1 = \text{isopentenyl}, R_2 = H$



GP15



GP16:  $R_1 = H, R_2 = OCH_3, R_3 = \text{isopentenyl}$

GP17:  $R_1 = H, R_2 = OCH_3, R_3 = \text{isopentenyl}$

GP19:  $R_1 = OH, R_2 = R_3 = H$

GP20:  $R_1 = R_3 = OH, R_2 = OH$

## ACKNOWLEDGEMENT

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Wanpen Naklue

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## LIST OF ABBREVIATIONS AND SYMBOLS

<i>s</i>	=	<i>singlet</i>
<i>d</i>	=	<i>doublet</i>
<i>t</i>	=	<i>triplet</i>
<i>q</i>	=	<i>quartet</i>
<i>m</i>	=	<i>multiplet</i>
<i>brs</i>	=	<i>broad singlet</i>
<i>brd</i>	=	<i>broad doublet</i>
<i>brq</i>	=	<i>broad quartet</i>
<i>dd</i>	=	<i>doublet of doublet</i>
<i>dt</i>	=	<i>doublet of triplet</i>
<i>mt</i>	=	<i>multiplet of triplet</i>
<i>ddd</i>	=	<i>doublet of doublet of doublet</i>
$\delta$	=	chemical shift relative to TMS
<i>J</i>	=	coupling constant
<i>m/z</i>	=	a value of mass divided by charge
$^{\circ}\text{C}$	=	degree celcius
$R_f$	=	retention factor
<i>g</i>	=	gram
<i>ml</i>	=	milliliter
$\text{cm}^{-1}$	=	reciprocal centimeter (wavenumber)
<i>nm</i>	=	nanometer
$\lambda_{max}$	=	maximum wavelength
$\nu$	=	absorption frequency
$\epsilon$	=	Molar extinction coefficient
<i>Hz</i>	=	Hertz

## LIST OF ABBREVIATIONS AND SYMBOLS (Continued)

MHz	=	megaHertz
ppm	=	part per million
$[\alpha]_D$	=	specific rotation
c	=	concentration
UV	=	Ultraviolet
IR	=	Infrared
NMR	=	Nuclear Magnetic Resonance
2D NMR	=	Two Dimensional Nuclear Magnetic Resonance
MS	=	Mass spectroscopy
HMQC	=	Heteronuclear Multiple Quantum Coherence
HMBC	=	Heteronuclear Multiple Bond Correlation
COSY	=	Correlation spectroscopy
DEPT	=	Distortionless Enhancement by Polarization Transfer
NOE	=	Nuclear Overhauser Effect
TLC	=	Thin-Layer Chromatography
DMSO	=	dimethylsulphoxide
MeOH	=	methanol
CD <sub>3</sub> OD	=	deuteromethanol
CDCl <sub>3</sub>	=	deuteriochloroform
ASA	=	anisaldehyde-sulphuric acid in acetic acid solution



# 1. INTRODUCTION

## 1.1 Introduction

*Garcinia parvifolia*, a plant belonging to the Guttiferae family, is widely distributed in the southern part of Thailand, Malaya, Sumatra, Borneo. *G. parvifolia* is a small to medium tree and once reaching 33 m tall, 240 cm girth; rarely a shrub. Inner bark with opaque, yellow exudate. Very similar to *G. cowa*, but leaves abruptly tapering into the tip. Male flowers with slender stalk 4-10 mm long, 7-10 mm wide, petals pale, clear yellow, 4 x 2.5 – 6 x 4 mm, not reflexed; female with calyx 4-6 mm wide. Fruits roundish or elongate, not grooved, occasionally umbonate, stigma and calyx generally deeply sunken, stigma only 1.5 mm wide; seated on persistent petals and sepals. Seeds small, embedded in much pulp. *G. parvifolia* is closely related to *G. nigrolineata* (Whitmore, 1973). In Thailand, *G. parvifolia* has a local name, “Cha maung lek”. (Smitinand, 2001)

## 1.2 Review of Literatures

Plants in the *Garcinia* genus (Guttiferae) are well known to be rich in a variety of compounds: xanthones (Soemiati, 2004; Sherley, 2004; Wahyuni, 2004; Abe, 2004; 2003; Chanmahasathien, 2003a,b; Suksamrarn, 2002; 2003; Chiang, 2003; Ito, 2003b; Rukachaisirikul, 2000a, 2003a,b; 2005c,d; Jantan, 2002; Nilar, 2002; Xu, 2001; Huang, 2001; Thoison, 2000), benzophenones (Merza, 2004; Williams, 2003; Chiang, 2003; Rukachaisirikul, 2003a; Abe, 2004; Ito, 2003a; Lakshmi, 2002; Huang, 2001; Ali, 2000), biflavonoids (Parveen, 2004; Abe, 2004; Thoison, 2000), benzophenone-xanthone dimmers (Kosela, 1999; 2000), chalcones (Ilyas, 2002), flavones (Farombi, 2002), depsidones (Xu, 2000) and triterpenes (Vieira, 2004a,b; Rukachaisirikul, 2003a; 2000b; 2005b; Weng, 2003a,b; Thoison, 2000; Nguyen,

2000). Some of these exhibit a wide range of biological and pharmacological activities, e.g., cytotoxic (Sherley, 2004; Soemiati, 2004; Williams, 2003; Xu, 2000; Thoison, 2000; Kosela, 2000), anti-inflammatory (Weng, 2003a,b), antimicrobial (Kosela, 2000), antifungal (Farombi, 2002; Kosela, 2000), antibacterial (Sherley, 2004; Lakshmi, 2002; Rukachaisirikul, 2000a; 2003b), antimalarial (Hay, 2004a; Kosela, 2000), anti-HIV (Rukachaisirikul, 2003a) and antioxidant (Hay, 2004b; Farombi, 2002) activities.

Chemical constituents isolated from plants of the genus *Garcinia* in 2004 have been reported (Sukpondma, 2005). The continuing search using SciFinder database revealed additional chemical constituents in 2005 which were summarized in Table 1.

**Table 1 Compounds from the genus *Garcinia* in 2005**

Scientific name	Investigated part	Compounds	Structures	References
<i>G. bancana</i>	twigs	[1,1'-biphenyl]-2-(3-methyl-2-butenyl)-3-methoxy-4,4',5,6-tetraol	3a	Rukachaisirikul, <i>et al.</i> , 2005a
		garcinol	1b	
		isogarcinol	1a	
		(-)-mellein	7g	
		8-(OH)-6-(OCH <sub>3</sub> )-3-pentylisocoumarin	7h	
		blumenol C <i>O</i> - $\beta$ -D-glucoside	7c	
	leaves	lupeol	5n	
		stigmasterol	7d	
		quercetin 3- <i>O</i> - $\alpha$ -L-rhamnoside	4b	
		kaemferol 3- <i>O</i> - $\alpha$ -L-rhamnoside	4a	

Table 1 (Continued)

Scientific name	Investigated part	Compounds	Structures	References
<i>G. bracteata</i>	bark	garcibracteaton	6.6d	Thoison, <i>et al.</i> , 2005
		xerophenone C	1i	
		5- <i>O</i> -methylxanthone V <sub>1</sub>	6.3ii	
		nemorosonol	1j	
		10- <i>O</i> -methylmacluraxanthone	6.3bb	
	leaves	neoisobractatin A	6.6b	
		neoisobractatin B	6.6c	
		bracteaxanthone I	6.3y	
		bracteaxanthone II	6.3jj	
		macluraxanthone	6.3aa	
		cudraxanthone R	6.3kk	
		gerontoxanthone I	6.3z	
<i>G. cowa</i>	latex	cowagarcinone A	6.3r	Mahabusarukum, <i>et al.</i> , 2005
		cowagarcinone B	6.3dd	
		cowagarcinone C	6.3cc	
		cowagarcinone D	6.3ee	
		cowagarcinone E	6.3f	
		cowaxanthone	6.3g	
		cowanin	6.3d	
		cowanol	6.3e	
		1,3,6-tri(OH)-7-(OCH <sub>3</sub> )-2,5-bis(3-methyl-2-butenyl)xanthone	6.3q	
		mangostinone	6.2k	
		fuscaxanthone A	6.3rr	

Table 1 (Continued)

Scientific name	Investigated part	Compounds	Structures	References
<i>G. dulcis</i>	green fruits	dulcinoside	4e	Deachathai, <i>et al.</i> , 2005
		dulcisisoflavone	4o	
		dulcixanthone A	6.2o	
		sphaerobioside acetate	4m	
		campoginol	1b	
		octadecanoic acid-2,3-di(OH)propyl ester	7j	
		derriscannoside A	4p	
		1,6-di(OH)-3,7-di(OCH <sub>3</sub> )-2-(3-methyl-2-butenyl)xanthone	6.3yy	
		cowanin	6.3d	
		cowaxanthone	6.3g	
		1,7-di(OH)-3-(OCH <sub>3</sub> )-2-(3-methyl-2-butenyl)-xanthone	6.2q	
		1,5,8-tri(OH)-3-(OCH <sub>3</sub> )-2-(3-methyl-2-butenyl)-xanthone	6.3ss	
		chandalone	4n	
		lupalbigenin	4k	
		BR-xanthone A	6.3mm	
		$\alpha$ -mangostin	6.3h	
		isolupalbigenin	4l	

Table 1 (Continued)

Scientific name	Investigated part	Compounds	Structures	References
		6,8,12-tri(OH)-7-(3-methyl-2-butenyl)-2-methyl-2-(4-methyl-3-pentenyl)pyrano(2',-3':7, 8)xanthone	6.3ee	
		2-(OH)-1,2,3-propanetricarboxylic acid-1,3-dimethylester	7i	
		vitexin	4f	
		morelloflavone	2b	
		clusiaphenone B	1o	
		mangostenol	6.3i	
		cratoxylone	6.3k	
		garcinone D	6.3j	
	ripe fruits	dulcisflavan	4j	
		dulcisxanthone B	6.3c	
		camboginol	1b	
		octadecanoic acid-2,3-di(OH)propyl ester	7j	
		1,5,8-tri(OH)-3-(OCH <sub>3</sub> )-2-(3-methyl-2-butenyl)xanthone	6.3ss	
		BR-xanthone A	6.3mm	
		mangostin	6.3h	
		morelloflavone	2b	
		mangostenol	6.3i	
		isonormangostin	6.3nn	

Table 1 (Continued)

Scientific name	Investigated part	Compounds	Structures	References
		1,6-di(OH)-7-(OCH <sub>3</sub> )-8-(3,7-dimethyl-2,6-octadienyl)-2',2'-dimethylpyrano[3,2-b]xanthen-9-one	6.3rr	
		tovophyllin A	6.3v	
		betulinic acid	5q	
		kaemferol 3-O- $\beta$ -glucopyranosyl-7-O- $\alpha$ -rhamnopyranoside	4c	
		garcinone B	6.3w	
		1,3,6-tri(OH)-7-(OCH <sub>3</sub> )-2,5-bis(3-methyl-2-butenyl)xanthone	6.3q	
		1,6-di(OH)-7-(OCH <sub>3</sub> )-8-(3-methyl-2-butenyl)-2',2'-dimethylchromeno[5',6': 2,3]xanthone	6.3aaa	
		8-desoxygartanin	6.2j	
		gartanin	6.3gg	
		morusignin J	6.3pp	
		apigenin	4h	
		cambogin	1a	
		kaemferol 3,7-di-O- $\alpha$ -L-rhamnopyranoside	4d	
		(-)-epicatechin	4i	

Table 1 (Continued)

Scientific name	Investigated part	Compounds	Structures	References
<i>G. griffithii</i>	stem bark	guttiferone I cambogin 1,7-di(OH)xanthone 1,3,6,7-tetra(OH)- xanthone 1,3,5,6-tetra(OH)- xanthone	1n 1a 6.1c 6.3l 6.3m	Nilar, <i>et al.</i> , 2005
<i>G. hanburyi</i>	fruits	hanburinone isomoreollin B morellin moreollic acid morellic acid	6.5q 6.5t 6.5r 6.5u 6.5s	Sukpondma, <i>et al.</i> , 2005a
<i>G. hombroniana</i>	leaves	garcihombronane B garcihombronane C garcihombronane D garcihombronane E garcihombronane F garcihombronane G garcihombronane H garcihombronane I garcihombronane J methyl (25 <i>R</i> )-3 $\beta$ - (OH)-23-oxo-9,15- lanostadien-26-oate vitexin isovitexin	5d 5e 5k 5l 5f 5g 5h 5i 5j 5m 4f 4g	Rukachaisirikul, <i>et al.</i> , 2005b

Table 1 (Continued)

Scientific name	Investigated part	Compounds	Structures	References
		blumenol C 9-O- $\beta$ -D-apiofuranosyl-(1 $\rightarrow$ 6)- $\beta$ -D-glucopyranoside	7b	
		vomifoliol 9-O- $\beta$ -D-apiofuranosyl-(1 $\rightarrow$ 6)- $\beta$ -D-glucopyranoside	7a	
<i>G. humilis</i>	stem and bark	guttiferone G guttiferone I	1l 1n	Herath, <i>et al.</i> , 2005
<i>G. kola</i>	roots	3'',4',4'',5,5'',7,7''-hepta(OH)-3,8''-biflavanone	2e	Han, <i>et al.</i> , 2005
<i>G. lirii</i>	roots	linixanthone A linixanthone B linixanthone C garcibiphenyl A garcibiphenyl B garcibenzopyran 10-O-methylmacluraxanthone rheediachromenoxanthone globulixanthone D 1,6-di(OH)-5,7-di(OCH <sub>3</sub> )xanthone 1,5-di(OH)xanthone	6.4b 6.2m 6.2c 3d 3e 7f 6.3bb 6.2l 6.2d 6.3uu 6.1b	Chen, <i>et al.</i> , 2005



Table 1 (Continued)

Scientific name	Investigated part	Compounds	Structures	References
		1,5-di(OH)-3-(OCH <sub>3</sub> )-xanthone	6.2e	
		1,6-di(OH)-3,5-di(OCH <sub>3</sub> )-xanthone	6.3tt	
		1,6-di(OH)-3,5,7-tri(OCH <sub>3</sub> )xanthone	6.4a	
		1,6-di(OH)-5-(OCH <sub>3</sub> )-xanthone	6.2f	
		1,6-di(OH)-7-(OCH <sub>3</sub> )-xanthone	6.2g	
		1,7-di(OH)xanthone	6.1c	
		5-(OH)-1-(OCH <sub>3</sub> )-xanthone	6.1a	
		acuparin	3f	
<i>G. mangostana</i>	heartwood	mangoxanthone	6.3ll	Nilar, <i>et al.</i> , 2005
		3',6-di(OH)-2,4,4'-tri(OCH <sub>3</sub> )benzophenone	1p	
		dulxanthone D	6.3b	
		1,3,7-tri(OH)-2-(OCH <sub>3</sub> )xanthone	6.3vv	
		1,3,5-tri(OH)-13,13-dimethyl-2H-pyran[7,6- <i>b</i> ]xanthen-9-one	6.3s	
<i>G. macrophylla</i>	twigs	guttiferone A	1k	Williams, <i>et al.</i> , 2005
		guttiferone G	1l	
		friedelin	5p	

Table 1 (Continued)

Scientific name	Investigated part	Compounds	Structures	References
<i>G. nigrolineata</i>	twigs	nigrolineabenzopyran A	7e	Rukachaisirikul, <i>et al.</i> , 2005d
		nigrolineabiphenyl A	3b	
		nigrolineabiphenyl B	3c	
		nigrolineaxanthone T	6.3o	
		nigrolineaxanthone U	6.3p	
		nigrolineaxanthone V	6.3u	
		nigrolineaxanthone W	6.3xx	
		dulxanthone A	6.3n	
		nigrolineaxanthone A	6.2p	
		1,3,5-tri(OH)- 4-(3-(OH)-3- methylbutyl)xanthone	6.2i	
		forbexanthone	6.3t	
		tovophyllin A	6.3v	
		6-deoxyjacareubin	6.2a	
		morusignin C	6.3oo	
		ananixanthone	6.2b	
		1,5-di(OH)-6',6'- dimethylpyrano[2',3'- :3,2]xanthone	6.2r	
		morusignin I	6.3hh	
		rheediaxanthone A	6.3qq	
		<i>G. polyantha</i>	stem bark	
bangangxanthone B	6.2s			
1,5-di(OH)xanthone	6.1b			
2-(OH)-1,7-di(OCH <sub>3</sub> )- xanthone	6.2n			

Table 1 (Continued)

Scientific name	Investigated part	Compounds	Structures	References
		friedelin	5p	
		oleanolic acid	5o	
		lupeol	5n	
<i>G. scortechinii</i>	fruits	scortechinone A	6.5a	Sukpondma, <i>et al.</i> , 2005b
		scortechinone B	6.5b	
		scortechinone C	6.5c	
		scortechinone D	6.5d	
		scortechinone E	6.5e	
		scortechinone F	6.5f	
		scortechinone H	6.5g	
		scortechinone I	6.5l	
		scortechinone M	6.5i	
		scortechinone L	6.5h	
		scortechinone P	6.5n	
		scortechinone Q	6.5v	
		scortechinone R	6.5k	
		scortechinone S	6.5o	
		scortechinone T	6.5p	
		scortechinone U	6.2t	
		scortechinone V	6.2u	
		scortechinone W	6.2v	
		scortechinone X	6.6a	
		scortechterpene A	5a	
		scortechterpene B	5b	
		(+)-volkensiflavone	2a	
		(+)-morelloflavone	2b	

Table 1 (Continued)

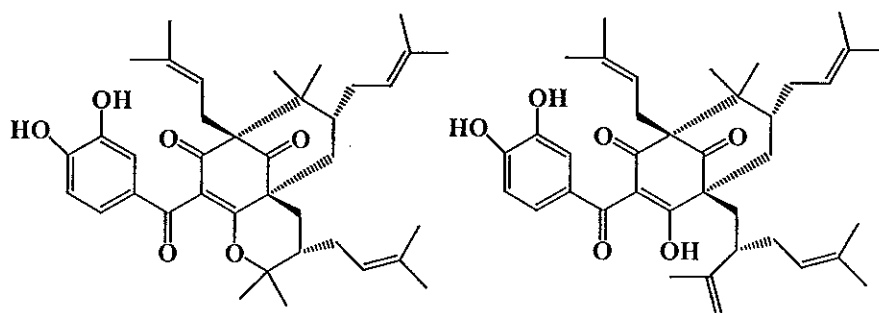
Scientific name	Investigated part	Compounds	Structures	References
	stem bark	germacra-4(15),5 <i>E</i> ,-10(14)-trien-1 $\beta$ -ol scortechinone A scortechinone B scortechinone D scortechinone F scortechinone I scortechinone J scortechinone L scortechinone M scortechinone N scortechinone O scortechinone P 4'',5''-dihydro-1,5-di(OH)-6',6'-dimethylpyrano-(2',3':6,7)-4'',4'',5''-trimethylfurano(2'',3''-:3,4)xanthone	5c 6.5a 6.5b 6.5d 6.5f 6.5l 6.5w 6.5h 6.5i 6.5j 6.5m 6.5n 6.3ww	Rukachaisirikul, <i>et al.</i> , 2005c
<i>G. smeathmannii</i>	stem bark	smeathxanthone A smeathxanthone B 1,3,5-tri(OH)xanthone 1,5-di(OH)xanthone	6.3zz 6.3x 6.2h 6.1b	Komguem, <i>et al.</i> , 2005
<i>G. xanthochymus</i>	fruits	guttiferone H gambogenone aristophenone A xanthochymol guttiferone E	1g 1h 1m 1e 1f	Baggett, <i>et al.</i> , 2005

Table 1 (Continued)

Scientific name	Investigated part	Compounds	Structures	References
		cycloxanthochymol	1d	
		isoxanthochymol	1c	
		alloathyriol	6.3a	
		amentoflavone	2f	
		3,8''-biapigenin	2d	
		(±)-fukugetin	2b	
		(±)-fukugiside	2c	
		(±)-volkensiflavone	2a	

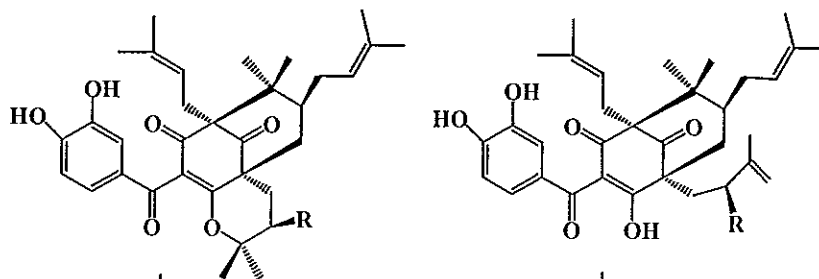
Structures of Compounds Isolated from Plants of the genus *Garcinia*

## 1. Benzophenones



1a: cambogin (isogarcinol)

1b: camboginol (garcinol)

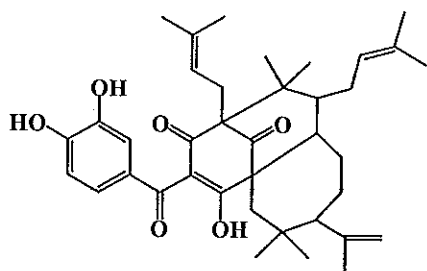


1c: R = : isoxanthochymol

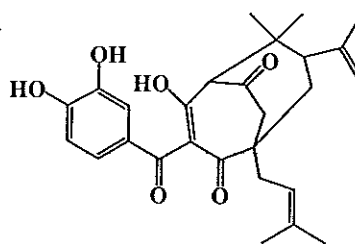
1e: R = : xanthochymol

1d: R = : cycloxanthochymol

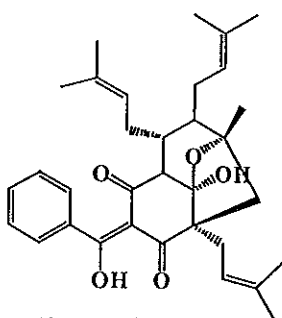
1f: R = : guttiferone E



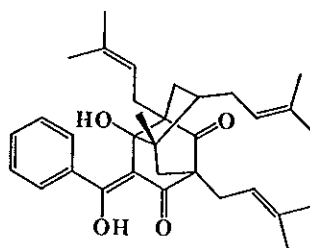
1g: guttiferone H



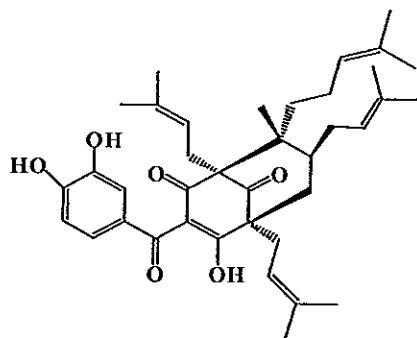
1h: gambogone



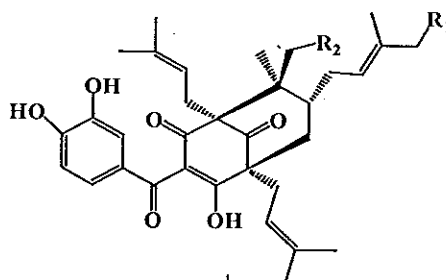

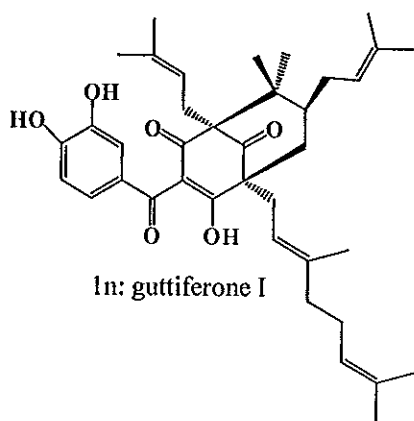
1i: xerophenone C



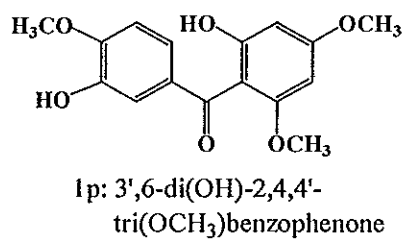
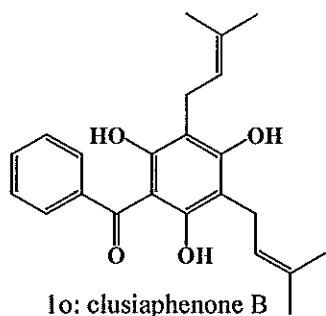
1j: nemorosonol



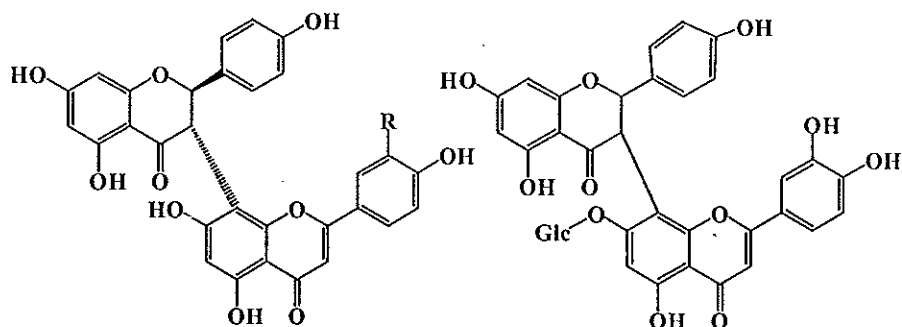
1k: guttiferone A

1l:  $R_1 = R_2 =$   : guttiferone G1m:  $R_1 = R_2 = H$  : aristophenone

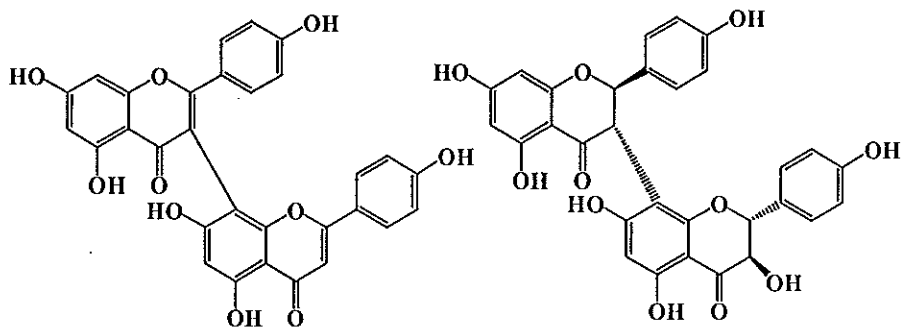
1n: guttiferone I

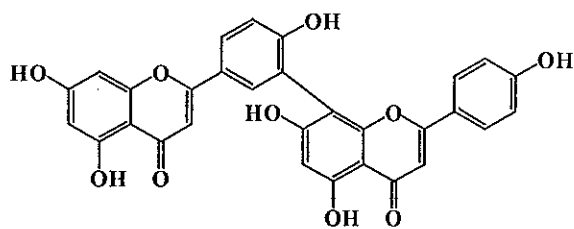


## 2. Biflavanoids



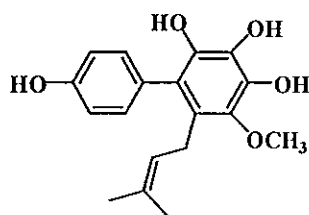
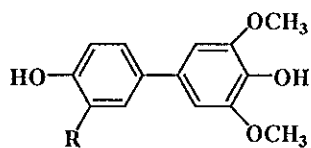
2b: R = OH : morelloflavone (fukugetin)



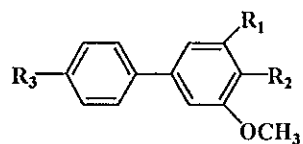


2f: amentoflavone

### 3. Biphenyls

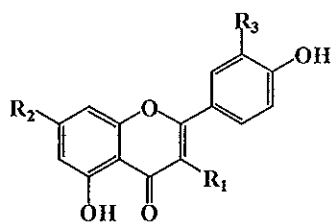
3a: [1,1'-biphenyl]-2-(3-methyl-2-butenyl)-3-(OCH<sub>3</sub>)-4,4',5,6-tetraol

3b: R = OH : nigrolineabiphenyl A

3c: R = OCH<sub>3</sub> : nigrolineabiphenyl B3d: R<sub>1</sub> = R<sub>3</sub> = OH, R<sub>2</sub> = H : garcibiphenyl A3e: R<sub>1</sub> = R<sub>3</sub> = OH, R<sub>2</sub> = CH<sub>2</sub>CH=C(CH<sub>3</sub>)<sub>2</sub> : garcibiphenyl B3f: R<sub>1</sub> = OCH<sub>3</sub>, R<sub>2</sub> = OH, R<sub>3</sub> = H : acuparin



## 4. Flavanoids

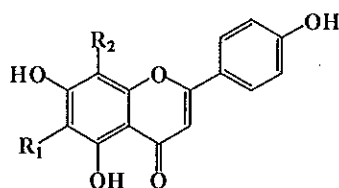


4a:  $R_1 = O\text{-}\alpha\text{-L-rhamnose}$ ,  $R_2 = \text{OH}$ ,  $R_3 = \text{H}$  : kaemferol 3- $O\text{-}\alpha\text{-L-rhamnoside}$

4b:  $R_1 = O\text{-}\alpha\text{-L-rhamnose}$ ,  $R_2 = R_3 = \text{OH}$  : quercetin 3- $O\text{-}\alpha\text{-L-rhamnoside}$

4c:  $R_1 = O\text{-}\beta\text{-D-glucose}$ ,  $R_2 = O\text{-}\alpha\text{-L-rhamnose}$ ,  $R_3 = \text{H}$  : kaemferol 3- $O\text{-}\beta\text{-D-glucopyranosyl-7-}O\text{-}\alpha\text{-L-rhamnopyranoside}$

4d:  $R_1 = R_2 = O\text{-}\alpha\text{-L-rhamnose}$ ,  $R_3 = \text{H}$  : kaemferol 3,7-di- $O\text{-}\alpha\text{-L-rhamnopyranoside}$

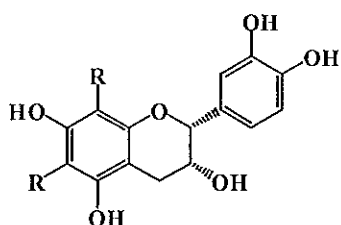


4e:  $R_1 = \beta\text{-D-glucose (6} \rightarrow \text{1)-}\alpha\text{-L-rhamnose}$ ,  $R_2 = \text{H}$  : dulcinoside

4f:  $R_1 = \text{H}$ ,  $R_2 = \beta\text{-D-glucose}$  : vitexin

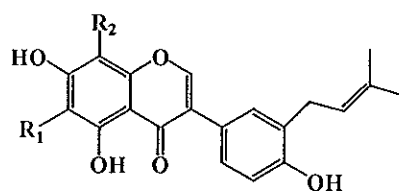
4g:  $R_1 = \beta\text{-D-glucose}$ ,  $R_2 = \text{H}$  : isovitexin

4h:  $R_1 = R_2 = \text{H}$  : apigenin



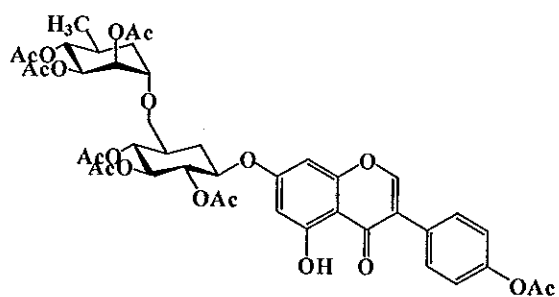
4i:  $R = \text{H}$  : (-)-epicatechin

4j:  $R = \text{OH}$  : dulcisflavan

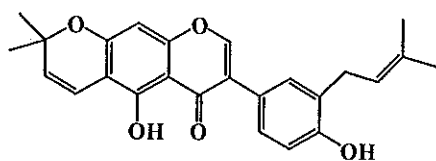


4k:  $R_1 = \text{prenyl}$ ,  $R_2 = \text{H}$  : lupalbigenin

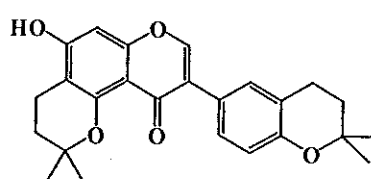
4l:  $R_1 = \text{H}$ ,  $R_2 = \text{prenyl}$  : isolupalbigenin



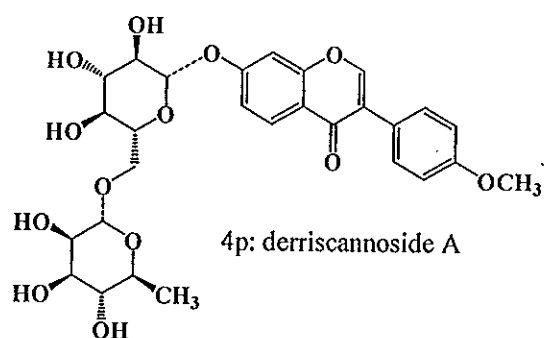
4m: sphaerobioside acetate



4n: chandalone

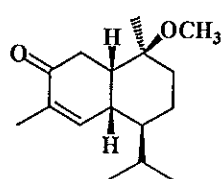


4o: dulcisisoflavone

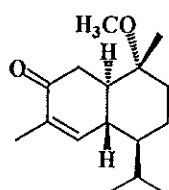


4p: derriscannoside A

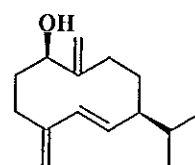
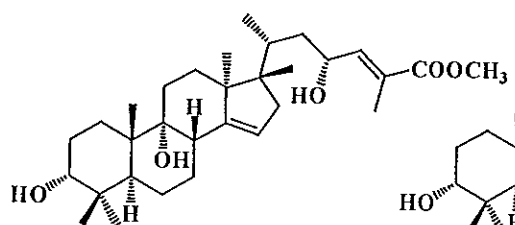
## 5. Terpenoids



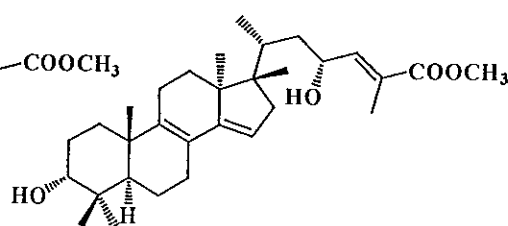
5a: scortechterpene A



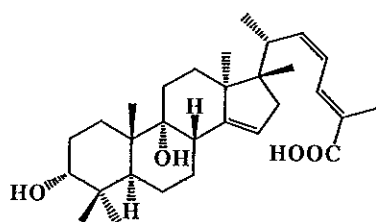
5b: scortechterpene B

5c: germacra-4(15),5E,-  
10(14)-trien-1-ol

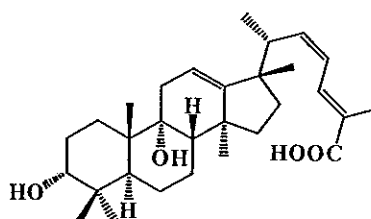
5d: garcihombronane B



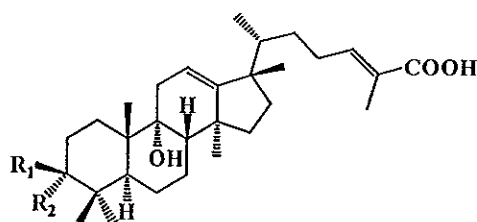
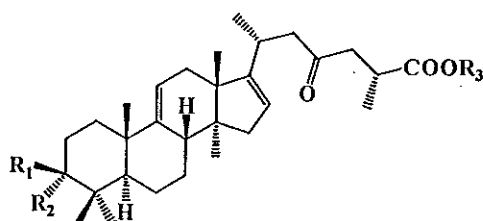
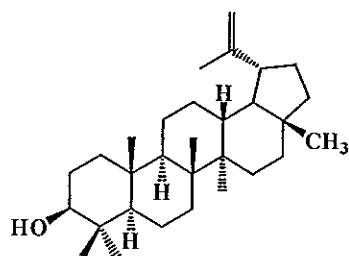
5e: garcihombronane C



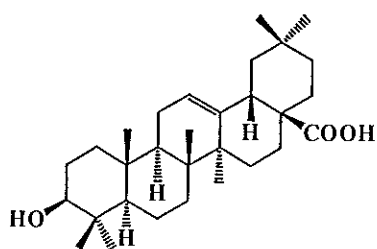
5f: garcihombronane F



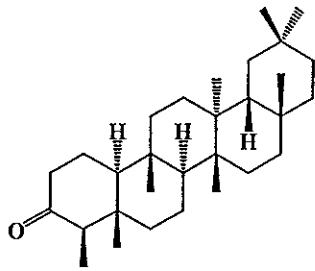
5g: garcihombronane G

5h:  $R_1 = H, R_2 = OH$  : garcihombronane H5i:  $R_1 = OH, R_2 = H$  : garcihombronane I5j:  $R_1 = H, R_2 = OH, R_3 = CH_3$  : garcihombronane J5k:  $R_1 = OH, R_2 = R_3 = H$  : garcihombronane D5l:  $R_1 = R_3 = H, R_2 = OH$  : garcihombronane E5m:  $R_1 = OH, R_2 = H, R_3 = CH_3$  : methyl (25R)-3 $\beta$ -(OH)-23-oxo-9,15-lanostadien-26-oate

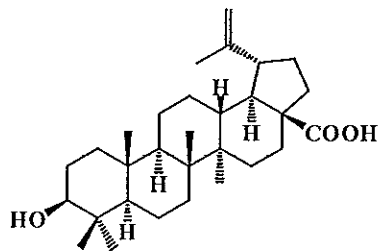
5n: lupeol



5o: oleanolic acid



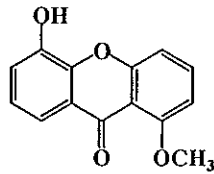
5p: friedelin



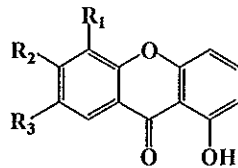
5q: betulinic acid

## 6. Xanthenes

### 6.1 Dioxygenated xanthenes



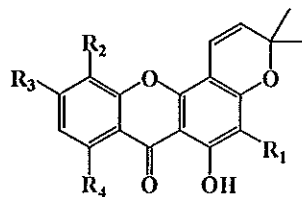
6.1a: 5-(OH)-1-(OCH<sub>3</sub>)xanthone



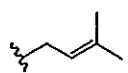
6.1b: R<sub>1</sub> = OH, R<sub>2</sub> = R<sub>3</sub> = H : 1,5-di(OH)xanthone

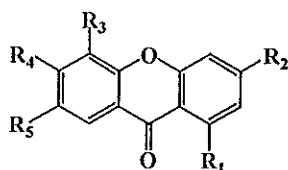
6.1c: R<sub>1</sub> = R<sub>2</sub> = H, R<sub>3</sub> = OH : 1,7-di(OH)xanthone

### 6.2 Trioxxygenated xanthenes



6.2a: R<sub>1</sub> = R<sub>3</sub> = R<sub>4</sub> = H, R<sub>2</sub> = OH : 6-deoxyjacareubin

6.2b: R<sub>1</sub> = , R<sub>2</sub> = OH, R<sub>3</sub> = R<sub>4</sub> = H : ananixanthone



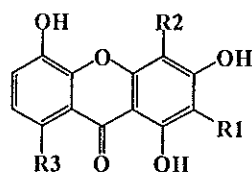
6.2c:  $R_1 = \text{OH}$ ,  $R_2 = \text{H}$ ,  $R_3 = R_4 = \text{OCH}_3$ ,  $R_5 = \text{isopentenyl}$  : linixanthone C

6.2d:  $R_1 = R_4 = \text{OH}$ ,  $R_2 = \text{H}$ ,  $R_3 = \text{OCH}_3$ ,  $R_5 = \text{isopentenyl}$  : globulixanthone D

6.2e:  $R_1 = R_3 = \text{OH}$ ,  $R_2 = \text{OCH}_3$ ,  $R_4 = R_5 = \text{H}$  : 1,5-di(OH)-3-(OCH<sub>3</sub>)xanthone

6.2f:  $R_1 = R_4 = \text{OH}$ ,  $R_2 = R_5 = \text{H}$ ,  $R_3 = \text{OCH}_3$  : 1,6-di(OH)-5-(OCH<sub>3</sub>)xanthone

6.2g:  $R_1 = R_4 = \text{OH}$ ,  $R_2 = R_3 = \text{H}$ ,  $R_5 = \text{OCH}_3$  : 1,6-di(OH)-7-(OCH<sub>3</sub>)xanthone

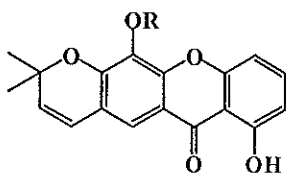


6.2h:  $R_1 = R_2 = R_3 = \text{H}$  : 1,3,5-tri(OH)xanthone

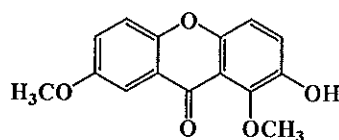
6.2i:  $R_1 = R_3 = \text{H}$ ,  $R_2 = \text{3-hydroxy-3-methylbutyl}$  : 1,3,5-tri(OH)-4-(3-(OH)-3-methylbutyl)xanthone

6.2j:  $R_1 = R_2 = \text{isopentenyl}$ ,  $R_3 = \text{H}$  : 8-desoxygartanin

6.2k:  $R_1 = \text{geranyl}$ ,  $R_2 = R_3 = \text{H}$  : mangostinone

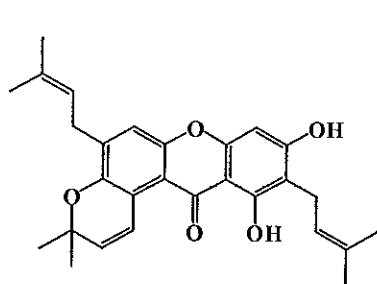


6.2l:  $R = \text{H}$  : rheediachromenoxanthone

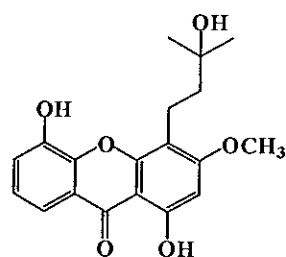


6.2n: 2-(OH)-1,7-di(OCH<sub>3</sub>)xanthone

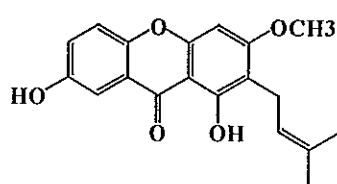
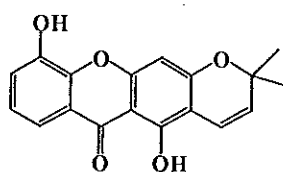
6.2m:  $R = \text{CH}_3$  : linixanthone B



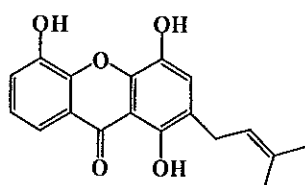
6.2o: dulcisxanthone A



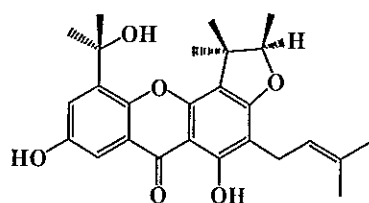
6.2p: nigrolineaxanthone A

6.2q: 1,7-di(OH)-3-(OCH<sub>3</sub>)-2-(3-methyl-2-butenyl)xanthone

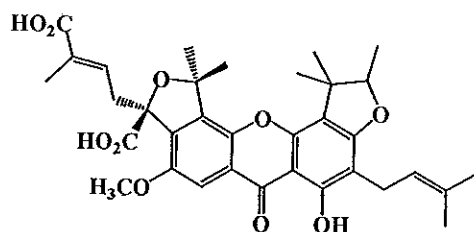
6.2r: 1,5-di(OH)-6',6'-dimethylpyrano[2',3':3,2]xanthone



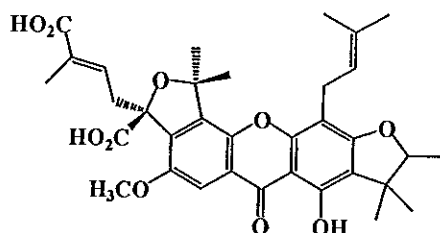
6.2s: bangangxanthone B



6.2t: scortechinone U

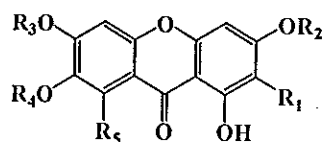


6.2u: scortechinone V






6.2v: scortechinone W


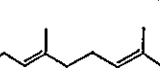
### 6.3 Tetraoxygenated xanthones







6.3a:  $R_1 = R_2 = R_3 = R_5 = H, R_4 = CH_3$  : alloathyriol


6.3b:  $R_1 = R_2 = R_3 = H, R_4 = CH_3, R_5 =$   : dulxanthone D


6.3c:  $R_1 =$  ,  $R_2 = CH_3, R_3 = R_4 = H, R_5 =$   : dulcisxanthone B



6.3d:  $R_1 =$  ,  $R_2 = R_3 = H, R_4 = CH_3, R_5 =$   : cowanin


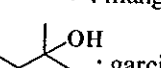
6.3e:  $R_1 =$  ,  $R_2 = R_3 = H, R_4 = CH_3, R_5 =$   : cowanol



6.3f:  $R_1 =$  ,  $R_2 = R_3 = H, R_4 = CH_3, R_5 =$   : cowagarcinone E

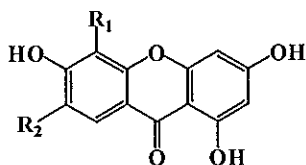
6.3g:  $R_1 =$  ,  $R_2 = R_3 = R_5 = H, R_4 = CH_3$  : cowaxanthone

6.3h:  $R_1 = R_5 =$  ,  $R_2 = R_3 = H, R_4 = CH_3$  :  $\alpha$ -mangostin

6.3i:  $R_1 =$  ,  $R_2 = R_3 = H, R_4 = CH_3, R_5 =$   : mangostenol

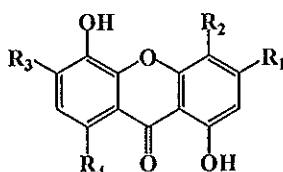
6.3j:  $R_1 =$  ,  $R_2 = R_3 = H, R_4 = CH_3, R_5 =$   : garcinone D

6.3k:  $R_1 =$  ,  $R_2 = R_3 = H, R_4 = CH_3, R_5 =$   : cratoxylone



6.3l:  $R_1 = H, R_2 = OH$  : 1,3,6,7-tetra(OH)xanthone

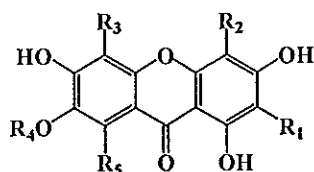
6.3m:  $R_1 = OH, R_2 = H$  : 1,3,5,6-tetra(OH)xanthone



6.3n:  $R_1 = OCH_3, R_2 = \text{3-methyl-2-butenyl}, R_3 = OH, R_4 = H$  : dulxanthone A

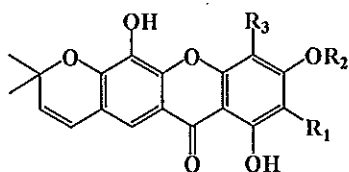
6.3o:  $R_1 = OCH_3, R_2 = \text{3-hydroxy-3-methylbutyl}, R_3 = OH, R_4 = H$  : nigrolineaxanthone T

6.3p:  $R_1 = R_4 = OH, R_2 = \text{3-hydroxy-3-methylbutyl}, R_3 = H$  : nigrolineaxanthone U



6.3q:  $R_1 = \text{3-methyl-2-butenyl}, R_2 = R_5 = H, R_3 = \text{3-methyl-2-butenyl}, R_4 = CH_3$  : 1,3,6-tri(OH)-7-(OCH<sub>3</sub>)-2,5-bis(3-methyl-2-butenyl)xanthone

6.3r:  $R_1 = \text{3-methyl-2-butenyl}, R_2 = H, R_3 = \text{3-methyl-2-butenyl}, R_4 = CH_3, R_5 = \text{3-methyl-2-butenyl}$  : cowagarcinone A

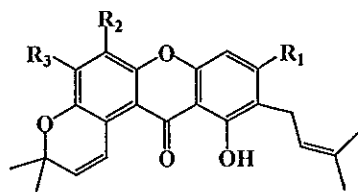


6.3s:  $R_1 = R_2 = R_3 = H$  : 1,3,5-tri(OH)-13,13-dimethyl-2H-pyran[7,6-b]xanthen-9-one

6.3t:  $R_1 = R_3 = H, R_2 = CH_3$  : forbexanthone

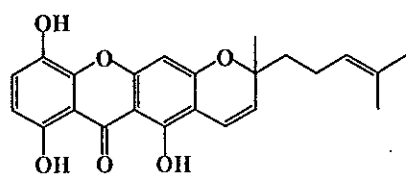
6.3u:  $R_1 = H, R_2 = CH_3, R_3 = \text{3-methyl-2-butenyl}$  : nigrolineaxanthone V



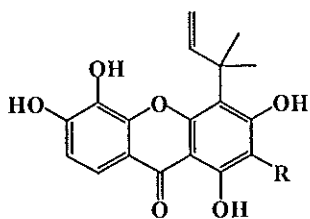


6.3v: R<sub>1</sub> = R<sub>3</sub> = OH, R<sub>2</sub> = : tovophyllin A

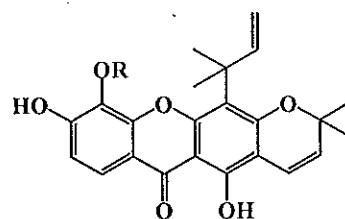
6.3w: R<sub>1</sub> = R<sub>3</sub> = OH, R<sub>2</sub> = H : garcinone B



6.3x: smeathxanthone B



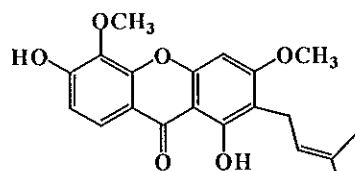
6.3y: R = : bracteaxanthone I



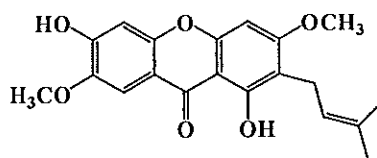
6.3aa: R = H : macluraxanthone

6.3z: R = : gerontoxanthone I

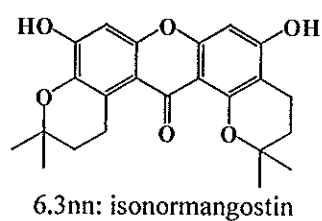
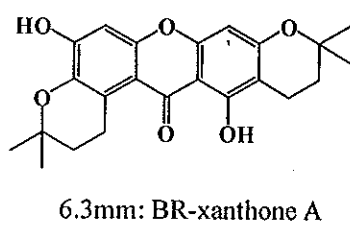
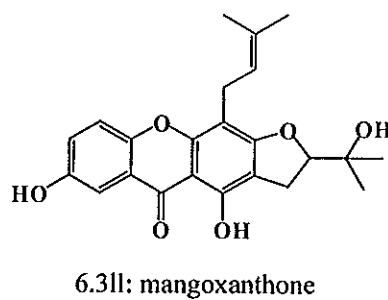
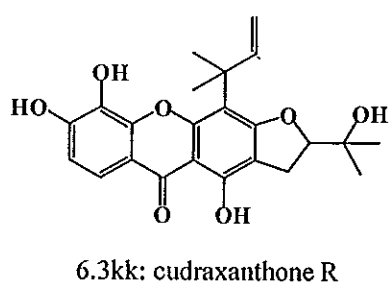
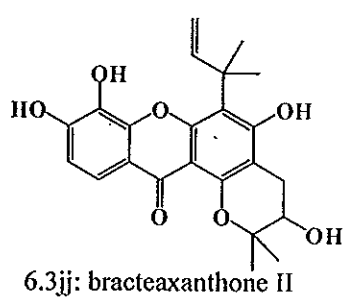
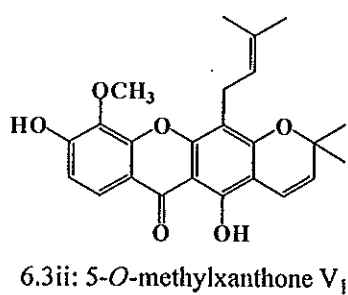
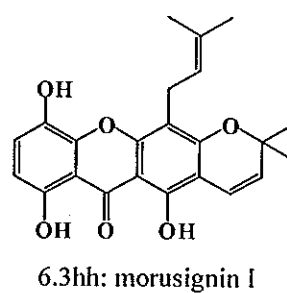
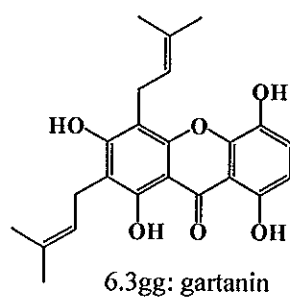
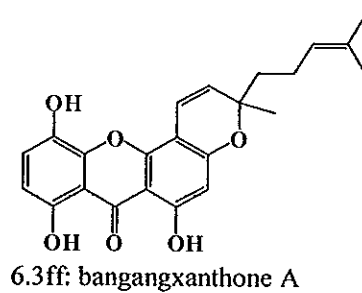
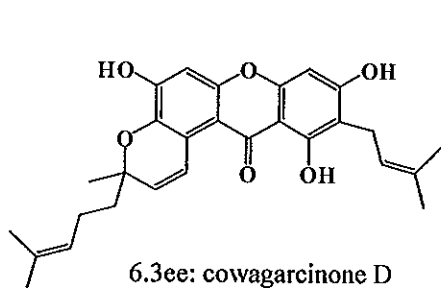
6.3bb: R = CH<sub>3</sub> : 10-*O*-methyl-macluraxanthone

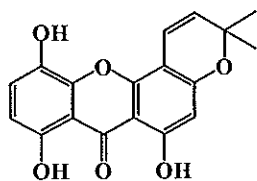


6.3cc: cowagarcinone C

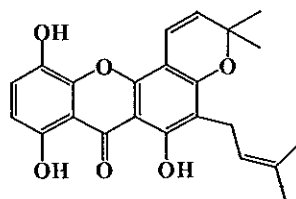


6.3dd: cowagarcinone B

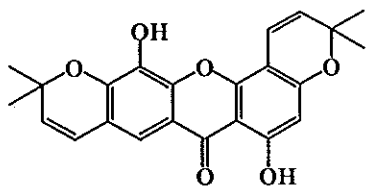




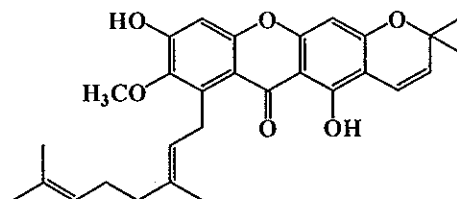
6.3oo: morusignin C



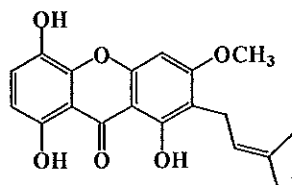
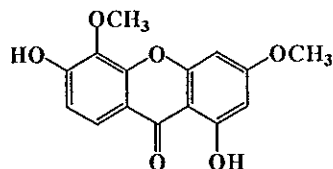
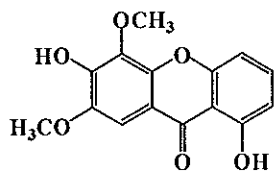
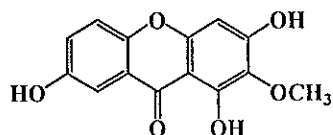
6.3pp: morusignin J

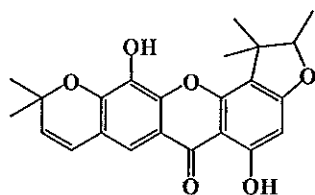


6.3qq: rheedioxanthone A

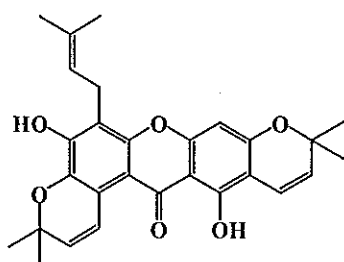


6.3rr: fuscaxanthone A

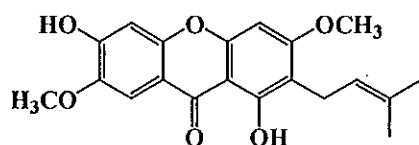
6.3ss: 1,5,8-tri(OH)-3-(OCH<sub>3</sub>)-2-(3-methyl-2-butenyl)xanthone6.3tt: 1,6-di(OH)-3,5-di(OCH<sub>3</sub>)xanthone6.3uu: 1,6-di(OH)-5,7-di(OCH<sub>3</sub>)xanthone6.3vv: 1,3,7-tri(OH)-2-(OCH<sub>3</sub>)xanthone



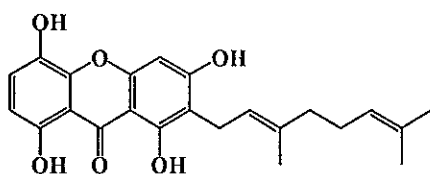
6.3ww: 4'',5''-dihydro-1,5-di(OH)-6',6'-dimethylpyrano(2',3':6,7)-4'',4'',5''-trimethylfurano(2'',3'':3,4)xanthone



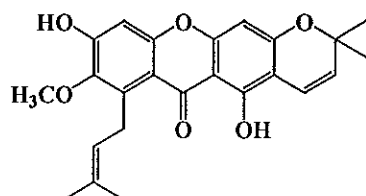
6.3xx: nigrolineaxanthone W



6.3yy: 1,6-di(OH)-3,7-di(OCH<sub>3</sub>)-2-(3-methyl-2-butenyl)xanthone

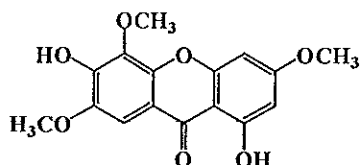


6.3zz: smeathxanthone A

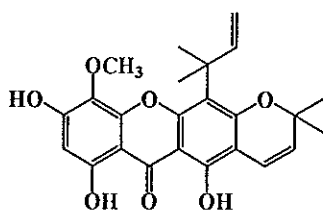


6.3aaa: 1,6-di(OH)-7-(OCH<sub>3</sub>)-8-(3-methyl-2-butenyl)-2',2'-dimethylchromeno [5',6': 2,3]xanthone

#### 6.4 Pentaoxygenated xanthenes

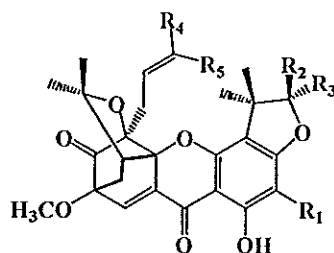











6.4a: 1,6-di(OH)-3,5,7-tri(OCH<sub>3</sub>)xanthone

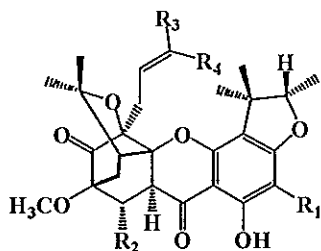


6.4b: linixanthone A

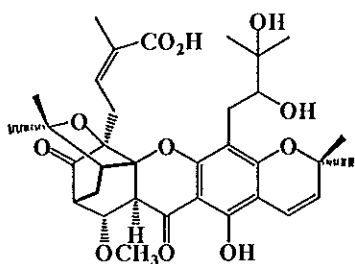
#### 6.5 Caged-polyprenylated xanthenes



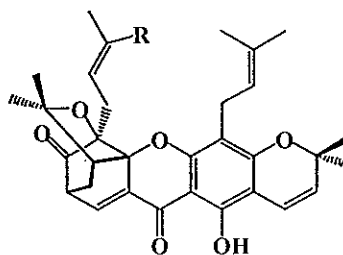
- 6.5a:  $R_1 =$  ,  $R_2 = R_4 = R_5 = \text{CH}_3$ ,  $R_3 = \text{H}$  : scortechinone A
- 6.5b:  $R_1 =$  ,  $R_2 = \text{H}$ ,  $R_3 = R_4 = \text{CH}_3$ ,  $R_5 = \text{CO}_2\text{H}$  : scortechinone B
- 6.5c:  $R_1 =$  ,  $R_2 = \text{H}$ ,  $R_3 = R_4 = \text{CH}_3$ ,  $R_5 = \text{CO}_2\text{H}$  : scortechinone C
- 6.5d:  $R_1 = R_3 = \text{H}$ ,  $R_2 = R_4 = R_5 = \text{CH}_3$  : scortechinone D
- 6.5e:  $R_1 = R_2 = \text{H}$ ,  $R_3 = R_4 = R_5 = \text{CH}_3$  : scortechinone E
- 6.5f:  $R_1 =$  ,  $R_2 = \text{H}$ ,  $R_3 = R_5 = \text{CH}_3$ ,  $R_4 = \text{CO}_2\text{H}$  : scortechinone F
- 6.5g:  $R_1 =$  ,  $R_2 = \text{H}$ ,  $R_3 = R_5 = \text{CH}_3$ ,  $R_4 = \text{CHO}$  : scortechinone H
- 6.5h:  $R_1 =$  ,  $R_2 = \text{H}$ ,  $R_3 = R_4 = R_5 = \text{CH}_3$  : scortechinone L
- 6.5i:  $R_1 =$  ,  $R_2 = \text{H}$ ,  $R_3 = R_4 = \text{CH}_3$ ,  $R_5 = \text{CO}_2\text{H}$  : scortechinone M
- 6.5j:  $R_1 =$  ,  $R_2 = \text{H}$ ,  $R_3 = R_4 = \text{CH}_3$ ,  $R_5 = \text{CO}_2\text{H}$  : scortechinone N
- 6.5k:  $R_1 =$  ,  $R_2 = R_4 = \text{CH}_3$ ,  $R_3 = \text{H}$ ,  $R_5 = \text{CO}_2\text{H}$  : scortechinone R



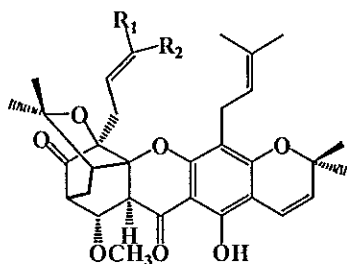
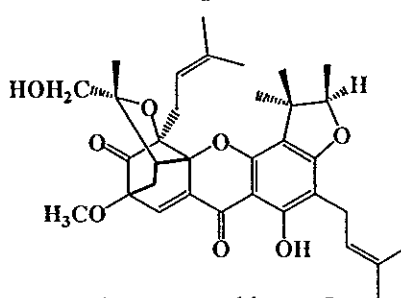
- 6.5l:  $R_1 = \text{CH}_2\text{CH}(\text{CH}_3)\text{CH}_2\text{CH}_2\text{CH}_3$ ,  $R_2 = \text{OCH}_3$ ,  $R_3 = \text{CH}_3$ ,  $R_4 = \text{CO}_2\text{H}$  : scortechinone I  
 6.5m:  $R_1 = \text{CH}_2\text{CH}(\text{CH}_3)\text{CH}_2\text{CH}_2\text{CH}_2\text{OH}$ ,  $R_2 = \text{OCH}_3$ ,  $R_3 = \text{CH}_3$ ,  $R_4 = \text{CO}_2\text{H}$  : scortechinone O  
 6.5n:  $R_1 = \text{CH}_2\text{CH}(\text{CH}_3)\text{CH}_2\text{CH}_2\text{CH}_2\text{OH}$ ,  $R_2 = \text{OH}$ ,  $R_3 = \text{CH}_3$ ,  $R_4 = \text{CO}_2\text{H}$  : scortechinone P  
 6.5o:  $R_1 = \text{CH}_2\text{CH}(\text{CH}_3)\text{CH}_2\text{CH}_2\text{CH}_2\text{OH}$ ,  $R_2 = \text{OCH}_3$ ,  $R_3 = \text{CH}_3$ ,  $R_4 = \text{CO}_2\text{H}$  : scortechinone S  
 6.5p:  $R_1 = \text{CH}_2\text{CH}(\text{CH}_3)\text{CH}_2\text{CH}_2\text{CH}_2\text{OH}$ ,  $R_2 = \text{OCH}_3$ ,  $R_3 = \text{CHO}$ ,  $R_4 = \text{CH}_3$  : scortechinone T



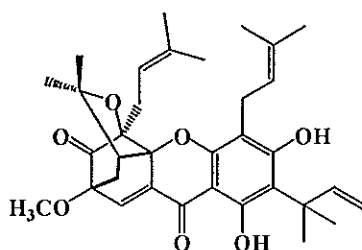
6.5q: hanburinone



6.5r: R = CHO: morellin

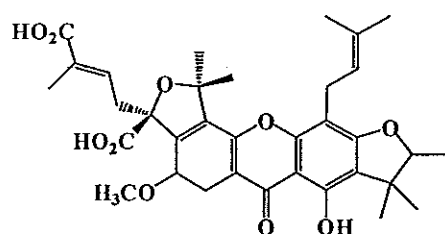
6.5s: R = CO<sub>2</sub>H : morellic acid6.5t:  $R_1 = \text{CHO}$ ,  $R_2 = \text{CH}_3$  : isomorellin B

6.5v: scortechinone Q

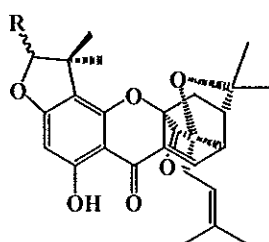
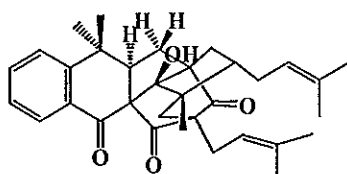
6.5u:  $R_1 = \text{CH}_3$ ,  $R_2 = \text{CO}_2\text{H}$  : moreollic acid

6.5w: scortechinone J

## 6.6 Rearranged xanthenes

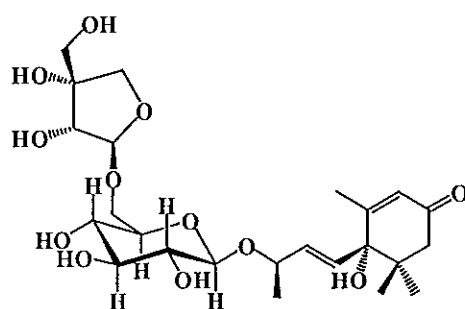


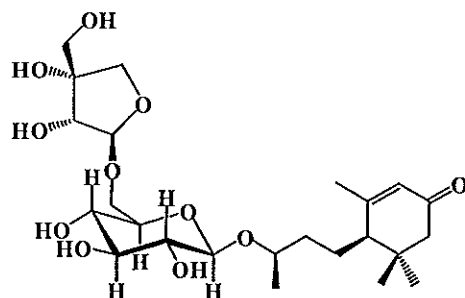
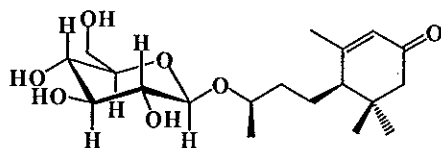
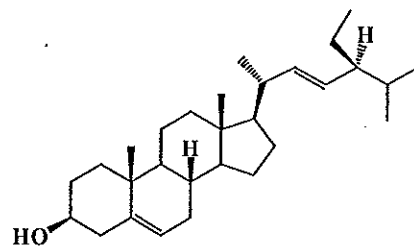
6.6a: scortechinone X

6.6b: R =  $\text{—CH}_3$  : neoisobractatin A6.6c: R =  $\text{—CH}_2\text{OH}$  : neoisobractatin B

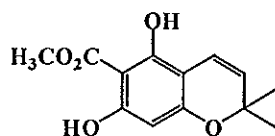
6.6d: garcibracteatone

## 7. Miscellaneous

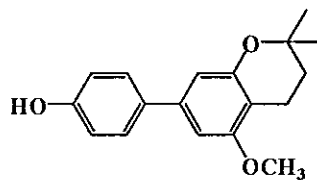
7a: vomifoliol 9-*O*- $\beta$ -D-apiofuranosyl-(1 $\rightarrow$ 6)- $\beta$ -D-glucopyranoside

7b: blumenol C 9-*O*- $\beta$ -D-apiofuranosyl-(1 $\rightarrow$ 6)- $\beta$ -D-glucopyranoside7c: blumenol C *O*- $\beta$ -D-glucoside

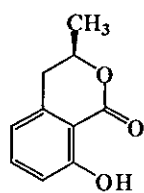
7d: stigmasterol



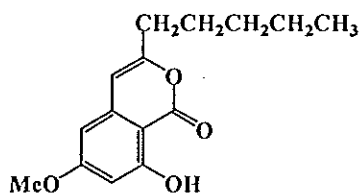
7e: nigrolineabenzopyran A



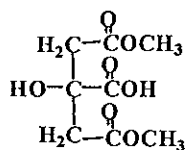
7f: garcibenzopyran



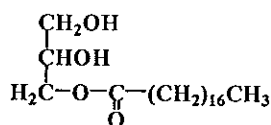
7g: (-)-mellein

7h: 8-(OH)-6-(OCH<sub>3</sub>)-3-pentylisocoumarin





7i: 2-(OH)-1,2,3-propanetricarboxylic acid-1,3-dimethylester

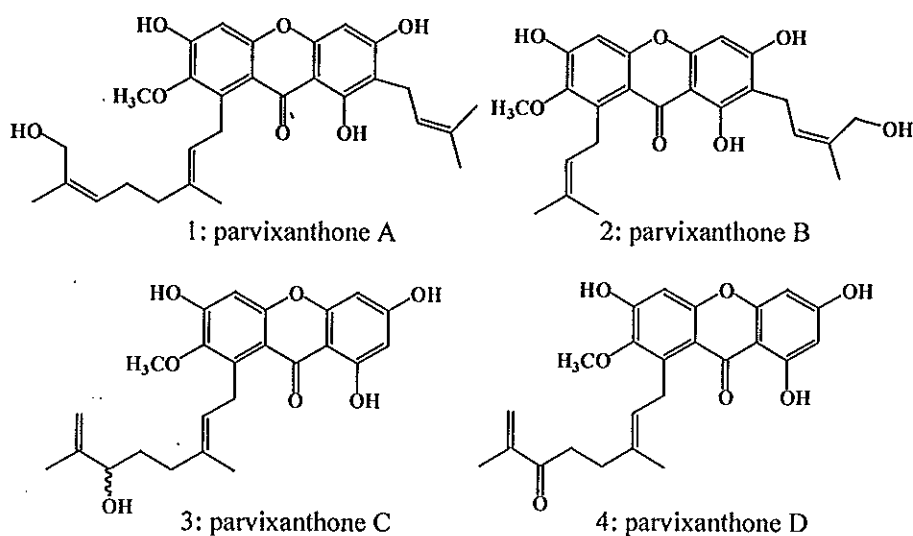


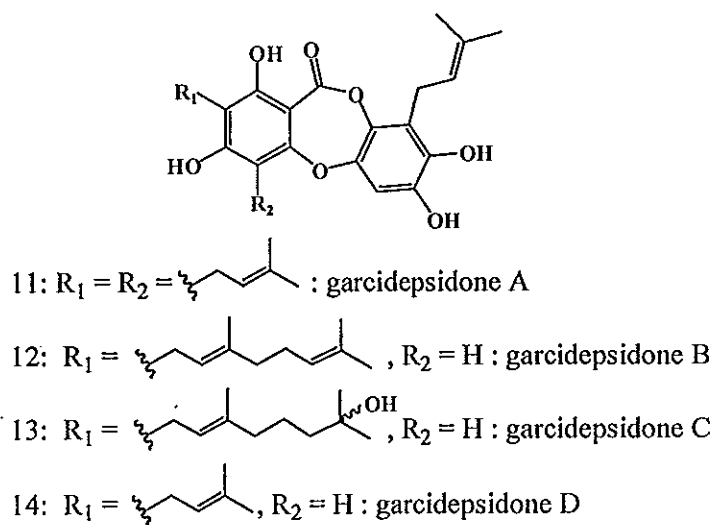
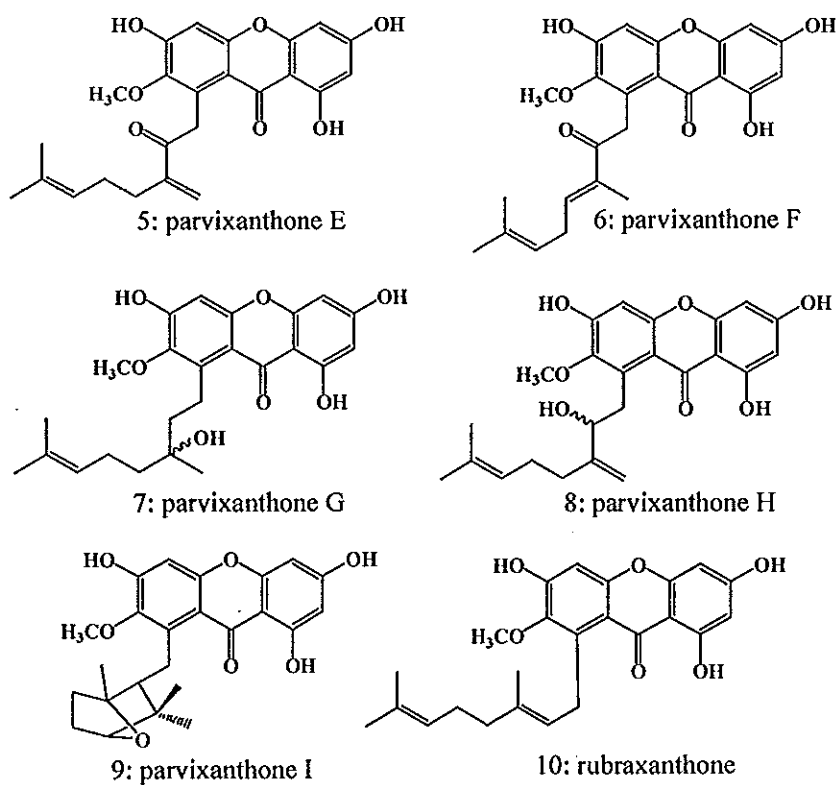
7j: octadecanoic acid-2,3-di(OH) propyl ester

### 1.3 The Objective

Based on the literature search, phytochemical investigation on the latex (Patthalung, 1988), leaves (Xu, 2000) and bark (Xu, 2001) of *G. parvifolia* resulted in the isolation of ten xanthenes and four depsidones which exhibited strong cytotoxic activity (Xu, 2000). We are interested in investigation of its twigs in order to separate additional chemical constituents. This research involved isolation, purification and structure elucidation of chemical constituents from the twigs of *G. parvifolia* which were collected at Trang province.

### Structures of Compounds Isolated from *Garcinia parvifolia*





## 2. EXPERIMENTAL

### 2.1 Chemicals and instruments

Melting points were determined on an electrothermal melting point apparatus (Electrothermal 9100) and reported without correction. Infrared spectra (IR) were obtained on a FTS165 FT-IR spectrometer or a Perkin Elmer Spectrum GX FT-IR system and recorded on wavenumber ( $\text{cm}^{-1}$ ).  $^1\text{H}$  and  $^{13}\text{C}$ -Nuclear magnetic resonance spectra ( $^1\text{H}$  and  $^{13}\text{C}$  NMR) were recorded on a FTNMR, Bruker Avance 300 MHz or a Varian UNITY INOVA 500 MHz spectrometer using tetramethylsilane (TMS) as an internal standard. Spectra were recorded as chemical shift parameter ( $\delta$ ) value in ppm down field from TMS ( $\delta 0.00$ ). Ultraviolet spectra (UV) were measured with an UV-160A spectrophotometer (SHIMADSU). Principle bands ( $\lambda_{\text{max}}$ ) were recorded as wavelengths (nm) and  $\log \varepsilon$  in MeOH solution. Optical rotation was measured in MeOH solution with sodium D line (590 nm) on an AUTOPOL<sup>®</sup> II automatic polarimeter. Quick column chromatography, thin-layer chromatography (TLC) and precoated thin-layer chromatography were performed on silica gel 60 GF<sub>254</sub> (Merck) or reverse phase C-18 silica gel. Column chromatography was performed on silica gel (Merck) type 100 (70-230 Mesh ASTM) or reverse phase C-18 silica gel. The solvents for extraction and chromatography were distilled at their boiling point ranges prior to use except for petroleum ether (bp. 40-60°C) and ethyl acetate which were analytical grade reagent.

### 2.2 Plant material

The twigs of *G. parvifolia* were collected at Trang Province, Thailand. A voucher specimen is deposited in the Herbarium of the Department of Biology, Faculty of Science, Prince of Songkla University, Hat Yai, Songkhla, Thailand.

### 2.3 Isolation and extraction

The twigs of *G. parvifolia* (2.4 kg), cut into small segments, were extracted with MeOH (12 L) for three times. After filtration, the filtrate was evaporated to dryness under reduced pressure to give a crude MeOH extract as a dark brown gum in 102.5 g.

### 2.4 Chemical investigation of the crude MeOH extract

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

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

Solvent	Solubility at room temperature
Petroleum ether	-
Chloroform	+ (pale yellow solution)
Ethyl acetate	++ (dark brown solution)
Acetone	++ (red solid in dark brown solution)
MeOH	+++ (dark brown solution)
Water	+ (pale yellow solution)
10% HCl	-
10% NaOH	+++ (dark brown solution)
10% NaHCO <sub>3</sub>	++ (dark brown solution)

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

The solubility results indicated that the crude extract contained acidic and polar constituents. Chromatogram characteristics on normal phase TLC of the crude extract, using 100%CHCl<sub>3</sub> as a mobile phase, showed seven UV-active spots with the R<sub>f</sub> values of 0.70, 0.61, 0.54, 0.45, 0.36, 0.21 and 0.16.

Chromatogram characteristics on normal phase TLC of the crude extract, using 100%CHCl<sub>3</sub> as a mobile phase, showed clear resolution. Further purification by quick column chromatography over silica gel was performed. Elution was conducted initially with 100%hexane and gradually enriched with chloroform and MeOH until 100%MeOH. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford eleven subfractions, as shown in Table 3.

**Table 3** Subfractions obtained from the crude extract by quick column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
A	100%Hexane-20%CHCl <sub>3</sub> /Hexane	264.4	Yellow liquid
B	20%CHCl <sub>3</sub> /Hexane	299.6	Yellow-brown gum
C	50%CHCl <sub>3</sub> /Hexane	283.9	Yellow-brown gum
D	70%CHCl <sub>3</sub> /Hexane-100%CHCl <sub>3</sub>	1794.8	Red-brown gum
E	0.2-0.7%MeOH/CHCl <sub>3</sub>	1059.0	Red-brown gum
F	1%MeOH/CHCl <sub>3</sub>	272.4	Brown-black gum
G	3%MeOH/CHCl <sub>3</sub>	3203.6	Brown-black gum
H	5%MeOH/CHCl <sub>3</sub>	604.2	Brown-black gum
I	10%MeOH/CHCl <sub>3</sub>	3052.3	Brown-black gum
J	20-40%MeOH/CHCl <sub>3</sub>	1643.4	Brown-black gum
K	40-100%MeOH/CHCl <sub>3</sub>	3301.9	Brown-black gum

**Fraction A** Chromatogram characteristics on normal phase TLC with 100%CHCl<sub>3</sub> showed no definite spot under UV and ASA reagent. Further investigation was then not carried out.

**Fraction B** Chromatogram characteristics on normal phase TLC with 100%CHCl<sub>3</sub> showed three UV-active spots with the R<sub>f</sub> values of 0.75, 0.70 and 0.61. This fraction was separated by column chromatography over silica gel. Elution was conducted initially with 20%CHCl<sub>3</sub>/Petrol, gradually enriched with CHCl<sub>3</sub> and finally with 100%CHCl<sub>3</sub>. Fractions with the similar chromatogram characteristics were combined

and evaporated to dryness under reduced pressure to afford seven subfractions, as shown in Table 4.

**Table 4 Subfractions obtained from the fraction B by column chromatography over silica gel**

Fraction	Mobile phase	Weight (mg)	Physical appearance
BA	20%CHCl <sub>3</sub> /Petrol	18.6	Yellow gum
BB	20%CHCl <sub>3</sub> /Petrol	25.4	Yellow gum
BC	20%CHCl <sub>3</sub> /Petrol	17.4	Yellow gum
BD	20%CHCl <sub>3</sub> /Petrol	13.0	Yellow-brown gum
BE	20-30%CHCl <sub>3</sub> /Petrol	14.6	Yellow-brown gum
BF	40%CHCl <sub>3</sub> /Petrol	14.9	Yellow-brown gum
BG	40-100%CHCl <sub>3</sub> /Petrol	82.3	Yellow-brown gum

**Fraction BA** Chromatogram characteristics on normal phase TLC with 40%CHCl<sub>3</sub>/Petrol showed no definite spot under UV and ASA reagent. Further investigation was then not carried out.

**Fraction BB** Chromatogram characteristics on normal phase TLC with 40%CHCl<sub>3</sub>/Petrol showed three UV-active spots with the R<sub>f</sub> values of 0.52, 0.44 and 0.38. This fraction was separated by column chromatography over silica gel. Elution was conducted initially with 2%EtOAc/Petrol, gradually enriched with EtOAc and finally with 45%EtOAc/Petrol. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford three subfractions, as shown in Table 5.

**Table 5 Subfractions obtained from the fraction BB by column chromatography over silica gel**

Fraction	Mobile phase	Weight (mg)	Physical appearance
BB1	2%EtOAc/Petrol	17.3	Yellow gum
BB2	2%EtOAc/Petrol	6.0	Yellow gum
BB3	2-45%EtOAc/Petrol	0.7	Yellow-brown gum

**Fraction BB1** Chromatogram characteristics on normal phase TLC with 2%EtOAc/Petrol showed three UV-active spots with the  $R_f$  values of 0.77, 0.63 and 0.44. Because the  $^1\text{H}$  NMR data indicated the presence of many compounds, it was not further investigated.

**Fraction BB2** Chromatogram characteristics on normal phase TLC with 2%EtOAc/Petrol showed one UV-active spot with the  $R_f$  value of 0.38. It was named as GP1.

$[\alpha]_D^{29}$	-36.43 (c = 0.06, MeOH)
UV $\lambda_{\text{max}}$ (nm)(MeOH)(log $\epsilon$ )	227 (3.17), 254 (4.39), 262 (4.49), 278 (3.25), 333 (2.82)
FTIR(neat): $\nu$ ( $\text{cm}^{-1}$ )	3431, 1668
$^1\text{H}$ NMR( $\text{CDCl}_3$ )( $\delta_{\text{ppm}}$ )(300 MHz) :	6.65 ( <i>dd</i> , $J = 10.0$ and $0.6$ Hz, 1H), 5.96 ( <i>brs</i> , 1H), 5.41 ( <i>d</i> , $J = 10.0$ Hz, 1H), 5.08 ( <i>mt</i> , $J = 7.0$ Hz, 1H), 4.03 ( <i>s</i> , 3H), 2.06 ( <i>m</i> , 2H), 1.69 ( <i>m</i> , 2H), 1.66 ( <i>s</i> , 3H), 1.57 ( <i>s</i> , 3H), 1.39 ( <i>s</i> , 3H)
$^{13}\text{C}$ NMR( $\text{CDCl}_3$ )( $\delta_{\text{ppm}}$ ) (75 MHz):	169.79, 161.00, 131.83, 124.71, 123.87, 116.43, 102.13, 96.54, 93.37, 80.22, 52.46, 41.65, 27.12, 25.65, 22.61, 17.61
DEPT 90°	CH : 124.71, 123.87, 116.43, 96.54
DEPT 135°	CH <sub>2</sub> : 41.65, 22.61 CH <sub>3</sub> : 52.46, 27.12, 25.65, 17.61
EIMS ( $m/z$ ) (% rel. int.)	318 (15), 271 (9), 235 (97), 203 (100)

**Fraction BB3** Chromatogram characteristics on normal phase TLC with 2%EtOAc/Petrol showed none of well-separated spots under UV and ASA reagent. Because of low quantity, it was not further investigated.

**Fraction BC** Chromatogram characteristics on normal phase TLC with 40% $\text{CHCl}_3$ /Petrol showed four UV-active spots with the  $R_f$  values of 0.52, 0.44, 0.38 and 0.29. This fraction was separated by column chromatography over silica gel. Elution was conducted initially with 2%EtOAc/Petrol, gradually enriched with EtOAc and finally with 30%EtOAc/Petrol. Fractions with the similar chromatogram

characteristics were combined and evaporated to dryness under reduced pressure to afford four subfractions, as shown in Table 6.

**Table 6** Subfractions obtained from the fraction BC by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
BC1	2%EtOAc/Petrol	1.4	Yellow gum
BC2	2%EtOAc/Petrol	8.3	Yellow gum
BC3	2%EtOAc/Petrol	3.3	Yellow-brown gum
BC4	2-30%EtOAc/Petrol	3.6	Yellow-brown gum

**Fraction BC1** Chromatogram characteristics on normal phase TLC with 2%EtOAc/Petrol showed no definite spot under UV and ASA reagent. Further investigation was then not carried out.

**Fraction BC2** Chromatogram characteristics on normal phase TLC with 2%EtOAc/Petrol showed two UV-active spots with the  $R_f$  values of 0.54 and 0.42. Because the  $^1\text{H}$  NMR data indicated the presence of many compounds, it was not further investigated.

**Fraction BC3** Chromatogram characteristics on normal phase TLC with 2%EtOAc/Petrol showed one UV-active spot with the  $R_f$  value of 0.38. The  $^1\text{H}$  NMR data indicated the presence of GP1 as a major component. Further investigation was then not carried out.

**Fraction BC4** Chromatogram characteristics on normal phase TLC with 2%EtOAc/Petrol showed one UV-active spot with the  $R_f$  value of 0.23. It was named as GP2.

UV  $\lambda_{\text{max}}$ (nm)(MeOH)(log  $\epsilon$ )                    223 (4.36), 268 (4.15), 317 (3.40)

FTIR(neat):  $\nu$  ( $\text{cm}^{-1}$ )                                3433, 1662

$^1\text{H}$  NMR( $\text{CDCl}_3$ )( $\delta_{\text{ppm}}$ )(300 MHz) : 6.07 (*s*, 1H), 5.16 (*mt*,  $J = 6.9$  Hz, 1H), 5.07 (*mt*,  $J = 6.9$  Hz, 1H), 4.03 (*s*, 3H), 3.83 (*s*, 3H), 3.26 (*d*,  $J = 6.9$  Hz, 2H), 2.00 (*m*, 4H), 1.76 (*s*, 3H), 1.64 (*s*, 3H), 1.58 (*s*, 3H)



$^{13}\text{C}$ NMR( $\text{CDCl}_3$ )( $\delta_{\text{ppm}}$ )(75 MHz):	169.97, 164.15, 160.80, 158.60, 134.82, 131.37, 124.48, 122.53, 109.00, 93.50, 91.57, 55.62, 52.41, 39.79, 26.75, 25.67, 21.46, 17.66, 16.04
DEPT 90°	CH : 124.48, 122.53, 91.57
DEPT 135°	CH <sub>2</sub> : 39.79, 26.75, 21.46 CH <sub>3</sub> : 55.62, 52.41, 25.67, 17.66, 16.04
EIMS ( $m/z$ ) (% rel. int.)	334 (4), 264 (8), 233 (46), 219 (21), 211 (69), 179 (100)

**Fraction BD** Chromatogram characteristics on normal phase TLC with 40% $\text{CHCl}_3$ /Petrol showed four UV-active spots with the  $R_f$  values of 0.52, 0.44, 0.38 and 0.28. The  $^1\text{H}$  NMR data indicated the presence of many compounds. Therefore, it was not further investigated.

**Fraction BE** Chromatogram characteristics on normal phase TLC with 40% $\text{CHCl}_3$ /Petrol showed one UV-active spot with the  $R_f$  value of 0.36. The  $^1\text{H}$  NMR data indicated the presence of many compounds. Thus, it was not further investigated.

**Fraction BF** Chromatogram characteristics on normal phase TLC with 40% $\text{CHCl}_3$ /Petrol showed three UV-active spots with the  $R_f$  values of 0.38, 0.25 and 0.11. The  $^1\text{H}$  NMR data indicated the presence of many compounds. Therefore, it was not further investigated.

**Fraction BG** Chromatogram characteristics on normal phase TLC with 40% $\text{CHCl}_3$ /Petrol showed none of well-separated spots under UV and ASA reagent. It was not further investigated.

**Fraction C** Chromatogram characteristics on normal phase TLC with 40% $\text{CHCl}_3$ /Petrol showed four UV-active spots with the  $R_f$  values of 0.61, 0.54, 0.50 and 0.45. This fraction was separated by column chromatography over silica gel. Elution was conducted initially with 3%EtOAc/Petrol, gradually enriched with EtOAc and finally with 50%EtOAc/Petrol. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford six subfractions, as shown in Table 7.

**Table 7** Subfractions obtained from the fraction C by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
CA	3%EtOAc/Petrol	18.3	Yellow gum
CB	3%EtOAc/Petrol	20.2	Yellow gum
CC	3%EtOAc/Petrol	12.4	Yellow-brown gum
CD	3-10%EtOAc/Petrol	16.3	Yellow-brown gum
CE	10-50%EtOAc/Petrol	46.2	Yellow-brown gum
CF	20-50%EtOAc/Petrol	92.1	Yellow-brown gum

**Fraction CA** Chromatogram characteristics on normal phase TLC with 3%EtOAc/Petrol showed two UV-active spots with the  $R_f$  values of 0.48 and 0.41. The  $^1\text{H}$  NMR data indicated the presence of GP3 as a major component. Further investigation was then not carried out.

**Fraction CB** Chromatogram characteristics on normal phase TLC with 3%EtOAc/Petrol showed one UV-active spot with the  $R_f$  value of 0.41. The  $^1\text{H}$  NMR data indicated the presence of GP4 as a major component. Further investigation was then not carried out.

**Fraction CC** Chromatogram characteristics on normal phase TLC with 3%EtOAc/Petrol showed two UV-active spots with the  $R_f$  values of 0.41 and 0.30. This fraction was separated by column chromatography over silica gel. Elution was conducted initially with 2%EtOAc/Petrol, gradually enriched with EtOAc and finally with 30%EtOAc/Petrol. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford four subfractions, as shown in Table 8.

**Table 8 Subfractions obtained from the fraction CC by column chromatography over silica gel**

Fraction	Mobile phase	Weight (mg)	Physical appearance
CC1	2%EtOAc/Petrol	0.3	Yellow gum
CC2	2%EtOAc/Petrol	2.4	Yellow gum
CC3	2%EtOAc/Petrol	9.5	Yellow-brown gum
CC4	2-30%EtOAc/Petrol	9.5	Yellow-brown gum

**Fraction CC1** Chromatogram characteristics on normal phase TLC with 3%EtOAc/Petrol showed no definite spot under UV and ASA reagent. Further investigation was then not carried out.

**Fraction CC2** Chromatogram characteristics on normal phase TLC with 3%EtOAc/Petrol showed one UV-active spot with the  $R_f$  value of 0.41. Because the  $^1\text{H}$  NMR data indicated the presence of many compounds, it was not further investigated.

**Fraction CC3** Chromatogram characteristics on normal phase TLC with 3%EtOAc/Petrol showed one UV-active spot with the  $R_f$  value of 0.41. The  $^1\text{H}$  NMR data indicated the presence of GP4 as a major component. Further investigation was then not carried out.

**Fraction CC4** Chromatogram characteristics on normal phase TLC with 3%EtOAc/Petrol showed none of well-separated spots under UV and ASA reagent. It was not further investigated.

**Fraction CD** Chromatogram characteristics on normal phase TLC with 3%EtOAc/Petrol showed three UV-active spots with the  $R_f$  values of 0.41, 0.30 and 0.25. Because the  $^1\text{H}$  NMR data indicated the presence of many compounds, it was not further investigated.

**Fraction CE** Chromatogram characteristics on normal phase TLC with 3%EtOAc/Petrol showed two UV-active spots with the  $R_f$  values of 0.25 and 0.19. This fraction was separated by column chromatography over silica gel. Elution was conducted initially with 15%CHCl<sub>3</sub>/Petrol, gradually enriched with CHCl<sub>3</sub> and finally with 25%CHCl<sub>3</sub>/Petrol. Fractions with the similar chromatogram characteristics were

combined and evaporated to dryness under reduced pressure to afford three subfractions, as shown in Table 9.

**Table 9** Subfractions obtained from the fraction CE by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
CE1	15%CHCl <sub>3</sub> /Petrol	20.3	Yellow gum
CE2	15-25%CHCl <sub>3</sub> /Petrol	14.5	Yellow gum
CE3	25%CHCl <sub>3</sub> /Petrol	14.3	Yellow-brown gum

**Fraction CE1** Chromatogram characteristics on normal phase TLC with 3%EtOAc/Petrol showed no definite spot under UV and ASA reagent. Further investigation was then not carried out.

**Fraction CE2** Chromatogram characteristics on normal phase TLC with 3%EtOAc/Petrol showed two UV-active spots with the R<sub>f</sub> values of 0.25 and 0.19. Because the <sup>1</sup>H NMR data indicated the presence of many compounds, it was not further investigated.

**Fraction CE3** Chromatogram characteristics on normal phase TLC with 3%EtOAc/Petrol showed none of well-separated spots under UV and ASA reagent. It was not further investigated.

**Fraction CF** Chromatogram characteristics on normal phase TLC with 3%EtOAc/Petrol showed none of well-separated spots under UV and ASA reagent. It was not further investigated.

**Fraction D** Chromatogram characteristics on normal phase TLC with 100%CHCl<sub>3</sub> showed six UV-active spots with the R<sub>f</sub> values of 0.71, 0.70, 0.61, 0.50, 0.45 and 0.32. This fraction was separated by column chromatography over Sephadex LH 20. Elution was conducted with 100%MeOH. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford five subfractions, as shown in Table 10.

**Table 10** Subfractions obtained from the fraction D by column chromatography over Sephadex LH 20

Fraction	Weight (mg)	Physical appearance
DA	4.0	Yellow gum
DB	1492.4	Yellow gum
DC	66.2	Yellow-brown gum
DD	15.3	Yellow-brown gum
DE	4.7	Yellow-brown gum

**Fraction DA** Chromatogram characteristics on normal phase TLC with 50%CHCl<sub>3</sub>/Petrol showed no definite spot under UV and ASA reagent. Further investigation was then not carried out.

**Fraction DB** Chromatogram characteristics on normal phase TLC with 50%CHCl<sub>3</sub>/Petrol showed four UV-active spots with the R<sub>f</sub> values of 0.65, 0.44, 0.23 and 0.15. This fraction was separated by column chromatography over silica gel. Elution was conducted initially with 40%CHCl<sub>3</sub>/Petrol, gradually enriched with CHCl<sub>3</sub> and finally with 100%CHCl<sub>3</sub>. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford six subfractions, as shown in Table 11.

**Table 11** Subfractions obtained from the fraction DB by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
DB1	40%CHCl <sub>3</sub> /Petrol	43.7	Yellow gum
DB2	40%CHCl <sub>3</sub> /Petrol	72.4	Yellow gum
DB3	40%CHCl <sub>3</sub> /Petrol	12.3	Yellow gum
DB4	50%CHCl <sub>3</sub> /Petrol	70.3	Yellow-brown gum
DB5	60-80%CHCl <sub>3</sub> /Petrol	294.3	Yellow-brown gum
DB6	100%CHCl <sub>3</sub>	277.3	Yellow-brown gum

**Fraction DB1** Chromatogram characteristics on normal phase TLC with 40%CHCl<sub>3</sub>/Petrol showed none of well-separated spots under UV and ASA

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

**Fraction DB2** Chromatogram characteristics on normal phase TLC with 40%CHCl<sub>3</sub>/Petrol showed two UV-active spots with the R<sub>f</sub> values of 0.58 and 0.35. The  $^1\text{H}$  NMR data indicated the presence of GP4 as a major component. Further investigation was then not carried out.

**Fraction DB3** Chromatogram characteristics on normal phase TLC with 40%CHCl<sub>3</sub>/Petrol showed two UV-active spots with the R<sub>f</sub> values of 0.49 and 0.29. Because the  $^1\text{H}$  NMR data indicated the presence of many compounds, it was not further investigated.

**Fraction DB4** Chromatogram characteristics on normal phase TLC with 40%CHCl<sub>3</sub>/Petrol showed three UV-active spots with the R<sub>f</sub> values of 0.40, 0.35 and 0.20. This fraction was separated by flash column chromatography over silica gel. Elution was conducted with 4%Acetone/Petrol. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford five subfractions, as shown in Table 12.

**Table 12** Subfractions obtained from the fraction DB4 by flash column chromatography over silica gel

Fraction	Weight (mg)	Physical appearance
DB41	2.2	Yellow gum
DB42	8.5	Colorless gum
DB43	4.8	Yellow gum
DB44	20.1	Yellow gum
DB45	12.0	Yellow-brown gum

**Fraction DB41** Chromatogram characteristics on normal phase TLC with 2%Acetone/Petrol showed no definite spot under UV and ASA reagent. Further investigation was then not carried out.

**Fraction DB42** Chromatogram characteristics on normal phase TLC with 2%Acetone/Petrol showed one UV-active spot with the R<sub>f</sub> value of 0.27. It was named as GP4.

$[\alpha]_D^{29}$	+53.02 (c = 0.16, MeOH)
UV $\lambda_{\max}$ (nm)(MeOH)(log $\epsilon$ )	207 (4.24), 226 (3.11), 301 (3.10)
FTIR(neat): $\nu$ (cm <sup>-1</sup> )	3431
<sup>1</sup> H NMR(CDCl <sub>3</sub> )( $\delta_{\text{ppm}}$ )(300 MHz) :	5.14 ( <i>m</i> , 1H), 5.10 ( <i>m</i> , 2H), 4.44 ( <i>brs</i> , 1H), 2.32 ( <i>m</i> , 1H), 2.20 ( <i>s</i> , 3H), 2.19 ( <i>s</i> , 3H), 2.11 ( <i>m</i> , 3H), 2.07 ( <i>m</i> , 4H), 2.00 ( <i>m</i> , 4H), 1.68 ( <i>m</i> , 2H), 1.68 ( <i>s</i> , 3H), 1.60 ( <i>brs</i> , 3H), 1.59 ( <i>m</i> , 2H), 1.59 ( <i>s</i> , 6H), 1.26 ( <i>s</i> , 3H)
<sup>13</sup> C NMR(CDCl <sub>3</sub> )( $\delta_{\text{ppm}}$ ) (75 MHz):	145.86, 144.72, 135.20, 134.97, 131.24, 126.89, 124.39, 124.24, 124.18, 122.22, 117.28, 115.17, 74.96, 39.82, 39.72, 31.27, 26.76, 26.65, 25.69, 23.83, 22.20, 20.62, 17.68, 16.00, 15.85, 12.25, 11.98
DEPT 90°	CH : 124.39, 124.24, 124.18
DEPT 135°	CH <sub>2</sub> : 39.82, 39.72, 31.27, 26.76, 26.65, 22.20, 20.62 CH <sub>3</sub> : 25.69, 23.83, 17.68, 16.00, 15.85, 12.25, 11.98
EIMS ( <i>m/z</i> ) (% rel. int.)	426 (3), 410 (15), 322 (7), 191 (10), 151 (30), 135 (13), 123 (16), 109 (21), 97 (25), 95 (27), 83 (28), 81 (42), 69 (100), 57 (53)

**Fraction DB43** Chromatogram characteristics on normal phase

TLC with 2%Acetone/Petrol showed three UV-active spots with the  $R_f$  values of 0.27, 0.17 and 0.12. Because the <sup>1</sup>H NMR data indicated the presence of many compounds, it was not further investigated.

**Fraction DB44** Chromatogram characteristics on normal

phase TLC with 2%Acetone/Petrol showed one UV-active spot with the  $R_f$  value of 0.12. It was named as GP3.

$[\alpha]_D^{29}$	-3.21 (c = 0.27, MeOH)
UV $\lambda_{\max}$ (nm)(MeOH)(log $\epsilon$ )	208 (4.23), 227 (3.14), 298 (3.09)
FTIR(neat): $\nu$ (cm <sup>-1</sup> )	3420

$^1\text{H}$ NMR( $\text{CDCl}_3$ )( $\delta_{\text{ppm}}$ )(300 MHz) :	6.36 ( <i>brs</i> , 1H), 5.12 ( <i>m</i> , 3H), 4.35 ( <i>brs</i> , 1H), 2.67 ( <i>t</i> , $J = 6.6$ Hz, 2H), 2.13 ( <i>s</i> , 6H), 2.12 ( <i>m</i> , 2H), 2.07 ( <i>m</i> , 6H), 1.96 ( <i>m</i> , 2H), 1.75 ( <i>m</i> , 2H), 1.68 ( <i>s</i> , 3H), 1.66 ( <i>m</i> , 1H), 1.61 ( <i>s</i> , 3H), 1.60 ( <i>s</i> , 3H), 1.58 ( <i>s</i> , 3H), 1.54 ( <i>m</i> , 1H), 1.26 ( <i>s</i> , 3H)
$^{13}\text{C}$ NMR( $\text{CDCl}_3$ )( $\delta_{\text{ppm}}$ ) (75 MHz):	146.28, 145.70, 135.09, 134.96, 131.25, 125.82, 124.42, 124.36, 124.21, 121.65, 118.24, 112.16, 75.23, 39.80, 39.72, 31.44, 26.77, 26.61, 25.69, 24.01, 22.29, 17.68, 16.00, 15.89, 11.90, 11.85
DEPT 90°	CH : 124.42, 124.36, 124.21, 112.16
DEPT 135°	CH <sub>2</sub> : 39.80, 39.72, 31.44, 26.77, 26.61, 22.29 CH <sub>3</sub> : 25.69, 24.01, 17.68, 16.00, 15.89, 11.90, 11.85
EIMS ( $m/z$ ) (% rel. int.)	410 (56), 206 (9), 191 (17), 151 (100), 69 (53)

**Fraction DB45** Chromatogram characteristics on normal phase TLC with 2%Acetone/Petrol showed none of well-separated spots under UV and ASA reagent. It was not further investigated.

**Fraction DB5** Chromatogram characteristics on normal phase TLC with 40%CHCl<sub>3</sub>/Petrol showed none of well-separated spots under UV and ASA reagent. It was not further investigated.

**Fraction DB6** Chromatogram characteristics on normal phase TLC with 40%CHCl<sub>3</sub>/Petrol showed no definite spot under UV and ASA reagent. Further investigation was then not carried out.

**Fraction DC** Chromatogram characteristics on normal phase TLC with 50%CHCl<sub>3</sub>/Petrol showed one UV-active spot with the  $R_f$  value of 0.15. Because the  $^1\text{H}$  NMR data indicated the presence of many compounds, it was not further investigated.

**Fraction DD** Chromatogram characteristics on normal phase TLC with 50%CHCl<sub>3</sub>/Petrol showed two UV-active spots with the  $R_f$  values of 0.15 and 0.12. Because the  $^1\text{H}$  NMR data indicated the presence of many compounds, it was not further investigated.



**Fraction DE** Chromatogram characteristics on normal phase TLC with 50%CHCl<sub>3</sub>/Petrol showed none of well-separated spots under UV and ASA reagent. Because of low quantity, it was not further investigated.

**Fraction E** Chromatogram characteristics on normal phase TLC with 100%CHCl<sub>3</sub> showed six UV-active spots with the R<sub>f</sub> values of 0.71, 0.70, 0.54, 0.45, 0.36 and 0.26. This fraction was separated by column chromatography over Sephadex LH 20. Elution was conducted with 100%MeOH. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford six subfractions, as shown in Table 13.

**Table 13 Subfractions obtained from the fraction E by column chromatography over Sephadex LH 20**

Fraction	Weight (mg)	Physical appearance
EA	4.7	Yellow gum
EB	170.2	Yellow gum
EC	390.6	Yellow gum
ED	210.7	Yellow-brown gum
EE	3.0	Yellow-brown gum
EF	3.1	Yellow-brown gum

**Fraction EA** Chromatogram characteristics on normal phase TLC with 50%CHCl<sub>3</sub>/Petrol showed no definite spot under UV and ASA reagent. Further investigation was then not carried out.

**Fraction EB** Chromatogram characteristics on normal phase TLC with 50%CHCl<sub>3</sub>/Petrol showed three UV-active spots with the R<sub>f</sub> values of 0.25, 0.15 and 0.09. Because the <sup>1</sup>H NMR data indicated the presence of many compounds, it was not further investigated.

**Fraction EC** Chromatogram characteristics on normal phase TLC with 50%CHCl<sub>3</sub>/Petrol showed two UV-active spots with the R<sub>f</sub> values of 0.21 and 0.15. This fraction was separated by column chromatography over silica gel. Elution was conducted initially with 2%EtOAc/Petrol, gradually enriched with EtOAc and finally

with 100%EtOAc. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford five subfractions, as shown in Table 14.

**Table 14** Subfractions obtained from the fraction EC by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
EC1	2%EtOAc/Petrol	9.3	Yellow gum
EC2	2-4%EtOAc/Petrol	96.9	Yellow gum
EC3	4-10%EtOAc/Petrol	107.1	Yellow gum
EC4	10-15%EtOAc/Petrol	73.3	Yellow-brown gum
EC5	15-100%EtOAc/Petrol	27.4	Yellow-brown gum

**Fraction EC1** Chromatogram characteristics on normal phase TLC with 5%EtOAc/Petrol showed no definite spot under UV and ASA reagent. Further investigation was then not carried out.

**Fraction EC2** Chromatogram characteristics on normal phase TLC with 5%EtOAc/Petrol showed three UV-active spots with the  $R_f$  values of 0.77, 0.59 and 0.31. This fraction was separated by column chromatography over silica gel. Elution was conducted with 30%CHCl<sub>3</sub>/Petrol. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford three subfractions, as shown in Table 15.

**Table 15** Subfractions obtained from the fraction EC2 by column chromatography over silica gel

Fraction	Weight (mg)	Physical appearance
EC21	3.5	Yellow gum
EC22	12.9	Yellow gum
EC23	43.2	Yellow gum

**Fraction EC21** Chromatogram characteristics on normal phase TLC with 40%CHCl<sub>3</sub>/Petrol showed no definite spot under UV and ASA reagent. Further investigation was then not carried out.

**Fraction EC22** Chromatogram characteristics on normal phase TLC with 40%CHCl<sub>3</sub>/Petrol showed one major UV-active spot with the same R<sub>f</sub> value as GP4. Further investigation was then not carried out.

**Fraction EC23** Chromatogram characteristics on normal phase TLC with 40%CHCl<sub>3</sub>/Petrol showed one major UV-active spot with the same R<sub>f</sub> value as GP3. Further investigation was then not carried out.

**Fraction EC3** Chromatogram characteristics on normal phase TLC with 5%EtOAc/Petrol showed two UV-active spots with the R<sub>f</sub> values of 0.31 and 0.24. This fraction was separated by column chromatography over silica gel. Elution was conducted with 30%CHCl<sub>3</sub>/Petrol. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford two subfractions, as shown in Table 16.

**Table 16 Subfractions obtained from the fraction EC3 by column chromatography over silica gel**

Fraction	Weight (mg)	Physical appearance
EC31	41.3	Yellow gum
EC32	22.4	Colorless gum

**Fraction EC31** Chromatogram characteristics on normal phase TLC with 40%CHCl<sub>3</sub>/Petrol showed no definite spot under UV and ASA reagent. Further investigation was then not carried out.

**Fraction EC32** Chromatogram characteristics on normal phase TLC with 40%CHCl<sub>3</sub>/Petrol showed one UV-active spot with the R<sub>f</sub> value of 0.30. It was named as GP5.

$[\alpha]_D^{28}$  +26.24 (c = 0.31, MeOH)  
 UV  $\lambda_{\max}$ (nm)(MeOH)(log  $\epsilon$ ) 206 (4.17), 223 (3.21), 297 (2.41)

FTIR(neat): $\nu$ ( $\text{cm}^{-1}$ )	3387
$^1\text{H}$ NMR( $\text{CDCl}_3$ )( $\delta_{\text{ppm}}$ )(300 MHz) :	6.47 ( <i>d</i> , $J = 2.7$ Hz, 1H), 6.37 ( <i>d</i> , $J = 2.7$ Hz, 1H), 5.11 ( <i>m</i> , 3H), 2.69 ( <i>t</i> , $J = 6.6$ Hz, 2H), 2.12 ( <i>m</i> , 2H), 2.12 ( <i>s</i> , 3H), 2.07 ( <i>m</i> , 2H), 2.01 ( <i>m</i> , 2H), 1.97 ( <i>m</i> , 4H), 1.76 ( <i>m</i> , 2H), 1.69 ( <i>m</i> , 1H), 1.68 ( <i>s</i> , 3H), 1.60 ( <i>s</i> , 3H), 1.59 ( <i>s</i> , 3H), 1.58 ( <i>s</i> , 3H), 1.53 ( <i>m</i> , 1H), 1.26 ( <i>s</i> , 3H)
$^{13}\text{C}$ NMR( $\text{CDCl}_3$ )( $\delta_{\text{ppm}}$ ) (75 MHz):	147.75, 145.99, 135.13, 134.97, 131.25, 127.36, 124.42, 124.31, 121.25, 115.66, 112.61, 75.34, 39.71, 39.69, 31.38, 26.77, 26.60, 25.69, 24.03, 22.49, 22.18, 17.68, 16.04, 16.00, 15.87
DEPT 90°	CH : 124.42, 124.31, 115.66, 112.61
DEPT 135°	CH <sub>2</sub> : 39.71, 39.69, 31.38, 26.77, 26.60, 22.49, 22.18 CH <sub>3</sub> : 25.69, 24.03, 17.68, 16.04, 16.00, 15.87
EIMS ( $m/z$ ) (% rel. int.)	396 (100), 177 (16), 137 (19), 83 (20), 81 (23), 69 (32)

**Fraction EC4** Chromatogram characteristics on normal phase TLC with 5%EtOAc/Petrol showed two UV-active spots with the  $R_f$  values of 0.15 and 0.13. Because the  $^1\text{H}$  NMR data indicated the presence of many compounds, it was not further investigated.

**Fraction EC5** Chromatogram characteristics on normal phase TLC with 5%EtOAc/Petrol showed none of well-separated spots under UV and ASA reagent. It was not further investigated.

**Fraction ED** Chromatogram characteristics on normal phase TLC with 50% $\text{CHCl}_3$ /Petrol showed three UV-active spots with the  $R_f$  values of 0.38, 0.29 and 0.19. This fraction was separated by column chromatography over Sephadex LH 20. Elution was conducted with 100%MeOH. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford four subfractions, as shown in Table 17.

**Table 17 Subfractions obtained from the fraction ED by column chromatography over Sephadex LH 20**

Fraction	Weight (mg)	Physical appearance
ED1	38.6	Yellow gum
ED2	18.3	Yellow gum
ED3	12.9	Yellow gum
ED4	34.3	Yellow-brown gum

**Fraction ED1** Chromatogram characteristics on normal phase TLC with 40%CHCl<sub>3</sub>/Petrol showed one major UV-active spot with the same R<sub>f</sub> value as GP5. Further investigation was then not carried out.

**Fraction ED2** Chromatogram characteristics on normal phase TLC with 40%CHCl<sub>3</sub>/Petrol showed two UV-active spots with the R<sub>f</sub> values of 0.30 and 0.17. Further purification was performed by precoated TLC, using 3%EtOAc/Petrol as a mobile phase (9 runs), to afford two bands.

**Band 1** was obtained as a yellow gum in 3.0 mg. Chromatogram characteristics on normal phase TLC with 3%EtOAc/Petrol showed one UV-active spot with the R<sub>f</sub> value of 0.30. The <sup>1</sup>H NMR data indicated the presence of many compounds. Because of low quantity, it was not further investigated.

**Band 2** was obtained as a yellow gum in 4.3 mg. Chromatogram characteristics on normal phase TLC with 3%EtOAc/Petrol showed one UV-active spot with the R<sub>f</sub> value of 0.17. It was named as GP6.

[α] <sub>D</sub> <sup>28</sup>	-21.15 (c = 0.07, MeOH)
UV λ <sub>max</sub> (nm)(MeOH)(log ε)	232 (4.16), 272 (4.37), 311 (2.32)
FTIR(neat):ν (cm <sup>-1</sup> )	3430, 1662
<sup>1</sup> H NMR(CDCl <sub>3</sub> )(δ <sub>ppm</sub> )(300 MHz) :	6.00 ( <i>s</i> , 1H), 5.12 ( <i>mt</i> , <i>J</i> = 7.2 Hz, 1H), 4.73 ( <i>t</i> , <i>J</i> = 8.7 Hz, 1H), 4.03 ( <i>s</i> , 3H), 3.03 ( <i>d</i> , <i>J</i> = 8.7 Hz, 2H), 2.11 ( <i>m</i> , 2H), 1.68 ( <i>s</i> , 3H), 1.63 ( <i>s</i> , 3H), 1.60 ( <i>m</i> , 2H), 1.29 ( <i>s</i> , 3H)

$^{13}\text{C}$ NMR( $\text{CDCl}_3$ )( $\delta_{\text{ppm}}$ ) (75 MHz):	169.82, 167.06, 132.20, 124.03, 105.08, 93.00, 91.01, 90.83, 73.70, 52.36, 36.68, 26.65, 25.66, 22.72, 21.93, 17.65
DEPT 90°	CH : 124.03, 91.01, 90.83
DEPT 135°	CH <sub>2</sub> : 36.68, 26.65, 21.93 CH <sub>3</sub> : 52.36, 25.66, 22.72, 17.65
EIMS ( $m/z$ ) (% rel. int.)	336 (43), 279 (15), 251 (19), 210 (61), 209 (43), 178 (36), 177 (38), 167 (24), 149 (100), 109 (22), 97 (20), 83 (21), 69 (32), 57 (34)

**Fraction ED3** Chromatogram characteristics on normal phase TLC with 40%CHCl<sub>3</sub>/Petrol showed one major UV-active spot with the same R<sub>f</sub> value as GP7. Further investigation was then not carried out.

**Fraction ED4** Chromatogram characteristics on normal phase TLC with 40%CHCl<sub>3</sub>/Petrol showed none of well-separated spots under UV and ASA reagent. It was not further investigated.

**Fraction EE** Chromatogram characteristics on normal phase TLC with 50%CHCl<sub>3</sub>/Petrol showed one UV-active spot with the R<sub>f</sub> value of 0.11. It was named as GP7.

UV $\lambda_{\text{max}}$ (nm)(MeOH)(log $\epsilon$ )	224 (4.38), 271 (4.20), 315 (3.42)
FTIR(neat): $\nu$ (cm <sup>-1</sup> )	3433, 1660
$^1\text{H}$ NMR( $\text{CDCl}_3$ )( $\delta_{\text{ppm}}$ )(300 MHz) :	6.00 ( <i>brs</i> , 1H), 5.99 ( <i>s</i> , 1H), 5.23 ( <i>mt</i> , $J = 7.2$ Hz, 1H), 5.05 ( <i>mt</i> , $J = 6.9$ Hz, 1H), 4.04 ( <i>s</i> , 3H), 3.35 ( <i>d</i> , $J = 7.2$ Hz, 2H), 2.07 ( <i>m</i> , 4H), 1.80 ( <i>s</i> , 3H), 1.67 ( <i>s</i> , 3H), 1.59 ( <i>s</i> , 3H)
$^{13}\text{C}$ NMR( $\text{CDCl}_3$ )( $\delta_{\text{ppm}}$ ) (75 MHz):	169.98, 162.31, 138.72, 131.96, 123.79, 121.69, 105.98, 96.03, 93.71, 52.43, 39.70, 26.39, 25.64, 21.64, 17.67, 16.16
DEPT 90°	CH : 123.79, 121.69, 96.03
DEPT 135°	CH <sub>2</sub> : 39.70, 26.39, 21.64 CH <sub>3</sub> : 52.43, 25.64, 17.67, 16.16

EIMS (*m/z*) (% rel. int.) 320 (9), 219 (100), 197 (39), 165 (25), 69 (20)

**Fraction EF** Chromatogram characteristics on normal phase TLC with 50%CHCl<sub>3</sub>/Petrol showed none of well-separated spots under UV and ASA reagent. It was not further investigated.

**Fraction F** Chromatogram characteristics on normal phase TLC with 100%CHCl<sub>3</sub> showed three UV-active spots with the R<sub>f</sub> values of 0.45, 0.39 and 0.32. This fraction was separated by column chromatography over Sephadex LH 20. Elution was conducted with 100%MeOH. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford five subfractions, as shown in Table 18.

**Table 18** Subfractions obtained from the fraction F by column chromatography over Sephadex LH 20

Fraction	Weight (mg)	Physical appearance
FA	4.3	Yellow gum
FB	57.6	Yellow gum
FC	10.4	Yellow gum
FD	18.3	Yellow-brown gum
FE	24.2	Yellow-brown gum

**Fraction FA** Chromatogram characteristics on normal phase TLC with 50%CHCl<sub>3</sub>/Petrol showed no definite spot under UV and ASA reagent. Further investigation was then not carried out.

**Fraction FB** Chromatogram characteristics on normal phase TLC with 50%CHCl<sub>3</sub>/Petrol showed six UV-active spots with the R<sub>f</sub> values of 0.41, 0.37, 0.19, 0.16, 0.15 and 0.09. Therefore, it was not further investigated.

**Fraction FC** Chromatogram characteristics on normal phase TLC with 50%CHCl<sub>3</sub>/Petrol showed three UV-active spots with the R<sub>f</sub> values of 0.21, 0.11 and 0.10. Thus, it was not further investigated.

**Fraction FD** Chromatogram characteristics on normal phase TLC with 50%CHCl<sub>3</sub>/Petrol showed three UV-active spots with the R<sub>f</sub> values of 0.16, 0.13 and 0.08. The <sup>1</sup>H NMR data indicated the presence of GP7 as a major component. Further investigation was then not carried out.

**Fraction FE** Chromatogram characteristics on normal phase TLC with 50%CHCl<sub>3</sub>/Petrol showed none of well-separated spots under UV and ASA reagent. It was not further investigated.

**Fraction G** Chromatogram characteristics on normal phase TLC with 100%CHCl<sub>3</sub> showed eight UV-active spots with the R<sub>f</sub> values of 0.70, 0.45, 0.39, 0.32, 0.27, 0.21, 0.16 and 0.07. This fraction was separated by column chromatography over silica gel. Elution was conducted initially with 100%CH<sub>2</sub>Cl<sub>2</sub>, gradually enriched with MeOH and finally with 100%MeOH. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford eight subfractions, as shown in Table 19.

**Table 19** Subfractions obtained from the fraction G by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
GA	100%CH <sub>2</sub> Cl <sub>2</sub>	297.3	Yellow gum
GB	100%CH <sub>2</sub> Cl <sub>2</sub>	203.0	Yellow gum
GC	100%CH <sub>2</sub> Cl <sub>2</sub>	182.8	Yellow gum
GD	100%CH <sub>2</sub> Cl <sub>2</sub>	52.1	Yellow-brown gum
GE	100%CH <sub>2</sub> Cl <sub>2</sub>	238.3	Yellow-brown gum
GF	5-10%CH <sub>2</sub> Cl <sub>2</sub> /MeOH	148.2	Yellow-brown gum
GG	10-25%CH <sub>2</sub> Cl <sub>2</sub> /MeOH	834.1	Yellow-brown gum
GH	25-100%CH <sub>2</sub> Cl <sub>2</sub> /MeOH	108.7	Yellow-brown gum

**Fraction GA** Chromatogram characteristics on normal phase TLC with 100%CH<sub>2</sub>Cl<sub>2</sub> showed many UV-active spots. Therefore, it was not further investigated.

**Fraction GB** Chromatogram characteristics on normal phase TLC with 100%CH<sub>2</sub>Cl<sub>2</sub> showed three UV-active spots with the R<sub>f</sub> values of 0.47, 0.39 and 0.22.



This fraction was separated by column chromatography over Sephadex LH 20. Elution was conducted with 100%MeOH. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford six subfractions, as shown in Table 20.

**Table 20 Subfractions obtained from the fraction GB by column chromatography over Sephadex LH 20**

Fraction	Weight (mg)	Physical appearance
GB1	72.1	Yellow gum
GB2	44.3	Yellow gum
GB3	9.4	Yellow gum
GB4	33.4	Yellow-brown gum
GB5	51.3	Yellow-brown gum
GB6	7.0	Yellow-brown gum

**Fraction GB1** Chromatogram characteristics on normal phase TLC with 80%CHCl<sub>3</sub>/Petrol showed no definite spot under UV and ASA reagent. Further investigation was then not carried out.

**Fraction GB2** Chromatogram characteristics on normal phase TLC with 80%CHCl<sub>3</sub>/Petrol showed two UV-active spots with the R<sub>f</sub> values of 0.33 and 0.22. The <sup>1</sup>H NMR data indicated the presence of GP5 as a major component. Further investigation was then not carried out.

**Fraction GB3** Chromatogram characteristics on normal phase TLC with 80%CHCl<sub>3</sub>/Petrol showed no definite spot under UV and ASA reagent. Further investigation was then not carried out.

**Fraction GB4** Chromatogram characteristics on normal phase TLC with 80%CHCl<sub>3</sub>/Petrol showed one major UV-active spot with the same R<sub>f</sub> as GP7. Further investigation was then not carried out.

**Fraction GB5** Chromatogram characteristics on normal phase TLC with 80%CHCl<sub>3</sub>/Petrol showed two UV-active spots with the R<sub>f</sub> values of 0.33 and 0.21. This fraction was separated by column chromatography over silica gel. Elution was conducted initially with 10%EtOAc/Petrol, gradually enriched with EtOAc and

finally with 100%EtOAc. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford five subfractions, as shown in Table 21.

**Table 21 Subfractions obtained from the fraction GB5 by column chromatography over silica gel**

Fraction	Mobile phase	Weight (mg)	Physical appearance
GB51	10%EtOAc/Petrol	4.6	Yellow gum
GB52	10%EtOAc/Petrol	9.2	Yellow gum
GB53	10%EtOAc/Petrol	4.5	Yellow gum
GB54	10-30%EtOAc/Petrol	17.9	Yellow-brown gum
GB55	30-100%EtOAc/Petrol	8.5	Yellow-brown gum

**Fraction GB51** Chromatogram characteristics on normal phase TLC with 10%Acetone/Petrol showed no definite spot under UV and ASA reagent. Further investigation was then not carried out.

**Fraction GB52** Chromatogram characteristics on normal phase TLC with 10%Acetone/Petrol showed one UV-active spot with the  $R_f$  value of 0.28. It was named as **GP8**.

UV  $\lambda_{\max}$ (nm)(MeOH)(log  $\epsilon$ ) 236 (4.19), 254 (4.48), 285 (3.65), 328 (3.60)

FTIR(neat):  $\nu$  ( $\text{cm}^{-1}$ ) 3410, 1644

$^1\text{H}$  NMR( $\text{CDCl}_3$ )( $\delta_{\text{ppm}}$ )(300 MHz): 13.12 (*s*, 1H), 7.60 (*s*, 1H), 6.37 (*s*, 1H), 6.12 (*s*, 1H), 5.61 (*s*, 1H), 5.35 (*mt*,  $J = 7.5$  Hz, 1H), 5.22 (*brs*, 1H), 3.91 (*s*, 3H), 3.49 (*d*,  $J = 5.7$  Hz, 2H), 3.42 (*d*,  $J = 7.5$  Hz, 2H), 1.85 (*s*, 3H), 1.77 (*s*, 3H), 1.76 (*s*, 3H), 1.71 (*s*, 3H)

$^{13}\text{C}$  NMR( $\text{CDCl}_3$ )( $\delta_{\text{ppm}}$ ) (75 MHz): 180.69, 163.62, 162.17, 153.77, 147.75, 143.76, 134.34, 131.67, 130.14, 125.36, 122.89, 121.08, 117.10, 113.34, 107.31, 102.99, 94.36, 56.07, 28.39, 25.83, 25.63, 21.72, 17.86

DEPT 90°	CH : 122.89, 121.08, 117.10, 94.39
DEPT 135°	CH <sub>2</sub> : 28.39, 21.72
	CH <sub>3</sub> : 56.07, 25.83, 25.63, 17.86
EIMS ( <i>m/z</i> ) (% rel. int.)	410 (29), 394 (32), 376 (57), 354 (23), 294 (100), 251 (29), 234 (33), 219 (57), 203 (81), 197 (37), 177 (36), 165 (79), 149 (63), 85 (53), 71 (73), 69 (78)

**Fraction GB53** Chromatogram characteristics on normal phase TLC with 10%Acetone/Petrol showed no definite spot under UV and ASA reagent. Further investigation was then not carried out.

**Fraction GB54** Chromatogram characteristics on normal phase TLC with 10%Acetone/Petrol showed showed one UV-active spot with the  $R_f$  value of 0.22. It was named as **GP9**.

UV $\lambda_{\max}$ (nm)(MeOH)(log $\epsilon$ )	256 (4.46), 286 (3.68), 329 (3.60)
FTIR(neat): $\nu$ (cm <sup>-1</sup> )	3346, 1641
<sup>1</sup> H NMR(CDCl <sub>3</sub> )( $\delta_{\text{ppm}}$ )(300 MHz) :	13.32 ( <i>s</i> , 1H), 7.58 ( <i>s</i> , 1H), 6.43 ( <i>s</i> , 1H), 6.15 ( <i>brs</i> , 1H), 5.67 ( <i>brs</i> , 1H), 5.34 ( <i>mt</i> , $J = 7.5$ Hz, 1H), 5.28 ( <i>mt</i> , $J = 7.2$ Hz, 1H), 5.25 ( <i>mt</i> , $J = 6.6$ Hz, 1H), 3.52 ( <i>d</i> , $J = 6.6$ Hz, 2H), 3.46 ( <i>d</i> , $J = 7.2$ Hz, 2H), 3.41 ( <i>d</i> , $J = 7.5$ Hz, 2H), 1.85 ( <i>s</i> , 6H), 1.78 ( <i>d</i> , $J = 1.2$ Hz, 3H), 1.77 ( <i>s</i> , 3H), 1.75 ( <i>s</i> , 3H), 1.73 ( <i>s</i> , 3H)
<sup>13</sup> C NMR(CDCl <sub>3</sub> )( $\delta_{\text{ppm}}$ ) (75 MHz):	180.68, 160.34, 158.62, 152.56, 147.69, 143.67, 135.83, 134.39, 133.50, 130.08, 125.27, 122.33, 121.40, 121.09, 117.11, 113.38, 108.94, 105.22, 102.91, 28.47, 25.87, 25.84, 25.70, 22.02, 21.63, 17.95, 17.86
DEPT 90°	CH : 122.33, 121.40, 121.09, 117.11
DEPT 135°	CH <sub>2</sub> : 28.47, 22.02, 21.63
	CH <sub>3</sub> : 25.87, 25.84, 25.70, 17.95, 17.86

EIMS ( $m/z$ ) (% rel. int.)                      464 (58), 463 (50), 446 (25), 420 (36), 409 (50),  
 408 (46), 394 (37), 352 (68), 336 (25), 308 (26),  
 297 (31), 149 (44), 85 (55), 71 (79)

**Fraction GB55** Chromatogram characteristics on normal phase TLC with 10%Acetone/Petrol showed none of well-separated spots under UV and ASA reagent. It was not further investigated.

**Fraction GB6** Chromatogram characteristics on normal phase TLC with 80%CHCl<sub>3</sub>/Petrol showed none of well-separated spots under UV and ASA reagent. It was not further investigated.

**Fraction GC** Chromatogram characteristics on normal phase TLC with 100%CH<sub>2</sub>Cl<sub>2</sub> showed four UV-active spots with the R<sub>f</sub> values of 0.47, 0.39, 0.34 and 0.26. This fraction was separated by column chromatography over Sephadex LH 20. Elution was conducted with 100%MeOH. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford four subfractions, as shown in Table 22.

**Table 22** Subfractions obtained from the fraction GC by column chromatography over Sephadex LH 20

Fraction	Weight (mg)	Physical appearance
GC1	37.6	Yellow gum
GC2	92.2	Yellow gum
GC3	45.3	Yellow gum
GC4	4.3	Yellow-brown gum

**Fraction GC1** Chromatogram characteristics on normal phase TLC with 100%CHCl<sub>3</sub> showed no definite spot under UV and ASA reagent. Further investigation was then not carried out.

**Fraction GC2** Chromatogram characteristics on normal phase TLC with 100%CHCl<sub>3</sub> showed one UV-active spot with the R<sub>f</sub> value of 0.19. Because the <sup>1</sup>H NMR data indicated the presence of many compounds, it was not further investigated.

**Fraction GC3** Chromatogram characteristics on normal phase TLC with 100%CHCl<sub>3</sub> showed three UV-active spots with the R<sub>f</sub> values of 0.34, 0.26 and 0.17. This fraction was separated by column chromatography over silica gel. Elution was conducted with 20%EtOAc/Petrol. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford three subfractions, as shown in Table 23.

**Table 23** Subfractions obtained from the fraction GC3 by column chromatography over silica gel

Fraction	Weight (mg)	Physical appearance
GC31	24.3	Yellow gum
GC32	6.9	Colorless gum
GC33	10.3	Yellow gum

**Fraction GC31** Chromatogram characteristics on normal phase TLC with 10%EtOAc/Petrol showed two UV-active spots with the R<sub>f</sub> values of 0.48 and 0.39. Because the <sup>1</sup>H NMR data indicated the presence of many compounds, it was not further investigated.

**Fraction GC32** Chromatogram characteristics on normal phase TLC with 10%EtOAc/Petrol showed one UV-active spot with the R<sub>f</sub> values of 0.22. It was named as GP10.

UV λ <sub>max</sub> (nm)(MeOH)(log ε)	211 (4.10), 270 (3.73), 294 (3.09)
FTIR(neat):ν (cm <sup>-1</sup> )	3417
<sup>1</sup> H NMR(CDCl <sub>3</sub> )(δ <sub>ppm</sub> )(300 MHz):	7.04 ( <i>dd</i> , <i>J</i> = 8.0 and 2.1 Hz, 1H), 7.00 ( <i>d</i> , <i>J</i> = 2.1 Hz, 1H), 6.97 ( <i>d</i> , <i>J</i> = 8.0 Hz, 1H), 6.72 ( <i>s</i> , 2H), 5.16 ( <i>brs</i> , 1H), 5.04 ( <i>brs</i> , 1H), 3.96 ( <i>s</i> , 3H), 3.95 ( <i>s</i> , 6H)
<sup>13</sup> C NMR(CDCl <sub>3</sub> )(δ <sub>ppm</sub> ) (75 MHz):	147.72, 146.67, 145.01, 134.16, 134.09, 133.11, 120.03, 114.64, 109.69, 104.02, 56.44, 56.08
EIMS ( <i>m/z</i> ) (% rel. int.)	276 (100), 261 (18), 233 (35), 138 (10)

**Fraction GC33** Chromatogram characteristics on normal phase TLC with 10%EtOAc/Petrol showed none of well-separated spots under UV and ASA reagent. It was not further investigated.

**Fraction GC4** Chromatogram characteristics on normal phase TLC with 100%CHCl<sub>3</sub> showed none of well-separated spots under UV and ASA reagent. It was not further investigated.

**Fraction GD** Chromatogram characteristics on normal phase TLC with 100%CH<sub>2</sub>Cl<sub>2</sub> showed three UV-active spots with the R<sub>f</sub> values of 0.56, 0.47 and 0.37. Because the <sup>1</sup>H NMR data indicated the presence of many compounds, it was not further investigated.

**Fraction GE** Chromatogram characteristics on normal phase TLC with 100%CH<sub>2</sub>Cl<sub>2</sub> showed four UV-active spots with the R<sub>f</sub> values of 0.34, 0.30, 0.24 and 0.18. This fraction was separated by column chromatography over Sephadex LH 20. Elution was conducted with 100%MeOH. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford four subfractions, as shown in Table 24.

**Table 24 Subfractions obtained from the fraction GE by column chromatography over Sephadex LH 20**

Fraction	Weight (mg)	Physical appearance
GE1	101.6	Yellow gum
GE2	66.7	Yellow gum
GE3	66.7	Yellow gum
GE4	6.3	Yellow-brown gum

**Fraction GE1** Chromatogram characteristics on normal phase TLC with 20%EtOAc/Petrol showed no definite spot under UV and ASA reagent. Further investigation was then not carried out.

**Fraction GE2** Chromatogram characteristics on normal phase TLC with 20%EtOAc/Petrol showed none of well-separated spots under UV and ASA reagent. It was not further investigated.

**Fraction GE3** Chromatogram characteristics on normal phase TLC with 20%EtOAc/Petrol showed four UV-active spots with the  $R_f$  values of 0.34, 0.21, 0.12 and 0.05. This fraction was separated by column chromatography over silica gel. Elution was conducted with 20%EtOAc/Petrol. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford four subfractions, as shown in Table 25.

**Table 25** Subfractions obtained from the fraction GE3 by column chromatography over silica gel

Fraction	Weight (mg)	Physical appearance
GE31	29.7	Yellow gum
GE32	2.0	Yellow gum
GE33	2.2	Yellow gum
GE34	16.8	Yellow-brown gum

**Fraction GE31** Chromatogram characteristics on normal phase TLC with 20%EtOAc/Petrol showed many UV-active spots. Thus, it was not further investigated.

**Fraction GE32** Chromatogram characteristics on normal phase TLC with 20%EtOAc/Petrol showed one UV-active spot with the  $R_f$  value of 0.20. It was named as GP11.

UV  $\lambda_{\max}$ (nm)(MeOH)(log  $\epsilon$ )            241 (4.01), 257 (3.82), 317 (3.15), 359 (2.59)

FTIR(neat):  $\nu$  ( $\text{cm}^{-1}$ )                    3394, 1646

$^1\text{H NMR}(\text{CDCl}_3)(\delta_{\text{ppm}})(300 \text{ MHz})$ : 13.35 (*s*, 1H), 6.88 (*s*, 1H), 6.33 (*brs*, 1H), 6.25 (*s*, 1H), 6.00 (*brs*, 1H), 5.97 (*brs*, 1H), 5.28 (*mt*,  $J = 6.6 \text{ Hz}$ , 1H), 5.27 (*mt*,  $J = 6.0 \text{ Hz}$ , 1H), 5.03 (*mt*,  $J = 6.6 \text{ Hz}$ , 1H), 4.10 (*d*,  $J = 6.0 \text{ Hz}$ , 2H), 3.82 (*s*, 3H), 3.53 (*d*,  $J = 6.6 \text{ Hz}$ , 2H), 2.09 (*m*, 4H), 1.89 (*d*,  $J = 0.9 \text{ Hz}$ , 3H), 1.83 (*d*,  $J = 1.2 \text{ Hz}$ , 3H), 1.70 (*d*,  $J = 1.2 \text{ Hz}$ , 3H), 1.66 (*d*,  $J = 0.9 \text{ Hz}$ , 3H), 1.60 (*d*,  $J = 0.9 \text{ Hz}$ , 3H)

$^{13}\text{C}$ NMR( $\text{CDCl}_3$ )( $\delta_{\text{ppm}}$ ) (75 MHz):	182.39, 161.68, 155.81, 154.58, 154.00, 142.67, 138.76, 137.06, 135.69, 124.27, 123.72, 123.08, 121.26, 112.20, 104.08, 101.56, 98.53, 62.12, 39.70, 26.55, 26.38, 25.83, 25.65, 21.62, 18.24, 17.97
DEPT 90°	CH : 123.72, 123.08, 121.26, 101.56, 98.53
DEPT 135°	CH <sub>2</sub> : 39.70, 26.55, 26.38, 21.62 CH <sub>3</sub> : 62.12, 25.83, 25.65, 18.24, 17.97
EIMS ( $m/z$ ) (% rel. int.)	478 (59), 463 (21), 435 (43), 409 (100), 367 (26), 355 (37), 339 (26), 323 (25), 167 (29), 149 (96), 111 (23), 109 (27), 97 (35), 83 (52), 71 (63)

**Fraction GE33** Chromatogram characteristics on normal phase TLC with 20%EtOAc/Petrol showed one UV-active spot with the  $R_f$  value of 0.06. It was named as **GP12**.

UV $\lambda_{\text{max}}$ (nm)(MeOH)(log $\epsilon$ )	221 (4.09), 272 (3.16), 281 (3.15), 327 (2.59)
FTIR(neat): $\nu$ ( $\text{cm}^{-1}$ )	3363, 1657
$^1\text{H}$ NMR( $\text{CDCl}_3$ )( $\delta_{\text{ppm}}$ )(300 MHz) :	11.36 ( <i>s</i> , 1H), 6.76 ( <i>d</i> , $J = 10.2$ Hz, 1H), 6.69 ( <i>s</i> , 1H), 6.29 ( <i>s</i> , 1H), 6.26 ( <i>s</i> , 1H), 5.75 ( <i>d</i> , $J = 10.2$ Hz, 1H), 5.39 ( <i>brs</i> , 1H), 5.23 ( <i>mt</i> , $J = 7.2$ Hz, 1H), 5.04 ( <i>mt</i> , $J = 6.6$ Hz, 1H), 3.41 ( <i>d</i> , $J = 7.2$ Hz, 2H), 2.08 ( <i>m</i> , 4H), 1.80 ( <i>s</i> , 6H), 1.68 ( <i>s</i> , 3H), 1.59 ( <i>s</i> , 3H), 1.46 ( <i>s</i> , 6H)
$^{13}\text{C}$ NMR( $\text{CDCl}_3$ )( $\delta_{\text{ppm}}$ ) (75 MHz):	168.50, 162.60, 162.41, 160.08, 143.10, 142.24, 140.25, 136.45, 132.58, 132.25, 132.06, 123.58, 120.67, 116.22, 113.80, 110.76, 106.43, 100.52, 98.53, 77.21, 39.67, 27.70, 26.25, 25.65, 21.95, 17.70, 16.23
DEPT 90°	CH : 132.06, 123.58, 120.67, 116.22, 106.43, 100.52
DEPT 135°	CH <sub>2</sub> : 39.67, 26.25, 21.95



	CH <sub>3</sub> : 27.70, 25.65, 17.70, 16.23
EIMS ( <i>m/z</i> ) (% rel. int.)	478 (100), 463 (26), 409 (35), 393 (56), 355 (92), 219 (35), 203 (36), 165 (36), 149 (65), 135 (33), 83 (29), 81 (33)

**Fraction GE34** Chromatogram characteristics on normal phase TLC with 20%EtOAc/Petrol showed none of well-separated spots under UV and ASA reagent. It was not further investigated.

**Fraction GE4** Chromatogram characteristics on normal phase TLC with 20%EtOAc/Petrol showed none of well-separated spots under UV and ASA reagent. Because of low quantity, it was not further investigated.

**Fraction GF** Chromatogram characteristics on normal phase TLC with 100%CH<sub>2</sub>Cl<sub>2</sub> showed one UV-active spot with the R<sub>f</sub> value of 0.18. This fraction was separated by column chromatography over Sephadex LH 20. Elution was conducted with MeOH. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford four subfractions, as shown in Table 26.

**Table 26** Subfractions obtained from the fraction GF by column chromatography over Sephadex LH 20

Fraction	Weight (mg)	Physical appearance
GF1	18.3	Yellow gum
GF2	44.3	Yellow gum
GF3	69.3	Yellow gum
GF4	17.7	Yellow-brown gum

**Fraction GF1** Chromatogram characteristics on normal phase TLC with 20%EtOAc/Petrol showed no definite spot under UV and ASA reagent. Further investigation was then not carried out.

**Fraction GF2** Chromatogram characteristics on normal phase TLC with 20%EtOAc/Petrol showed two UV-active spots with the R<sub>f</sub> values of 0.27

and 0.22. Further purification was performed by precoated TLC, using 10%EtOAc/Petrol as a mobile phase (8 runs), to afford two bands.

Band 1 was obtained as a yellow gum in 10.3 mg. Chromatogram characteristics on normal phase TLC with 20%EtOAc/Petrol showed one UV-active spot with the  $R_f$  value of 0.27. Because the  $^1\text{H}$  NMR data indicated the presence of many compounds, it was not further investigated.

Band 2 was obtained as a yellow gum in 9.6 mg. Chromatogram characteristics on normal phase TLC with 20%EtOAc/Petrol showed one UV-active spot with the  $R_f$  value of 0.20. It was named as **GP13**.

$[\alpha]_D^{29}$	+60.01 (c = 0.01, $\text{CHCl}_3$ )
UV $\lambda_{\text{max}}$ (nm)(MeOH)(log $\epsilon$ ).	205 (3.82), 234 (2.23), 277 (2.01)
FTIR(neat): $\nu$ ( $\text{cm}^{-1}$ )	3404, 1655
$^1\text{H}$ NMR( $\text{CDCl}_3$ )( $\delta_{\text{ppm}}$ )(300 MHz) :	5.84 ( <i>brq</i> , $J = 1.2$ Hz, 1H), 5.20 ( <i>mt</i> , $J = 6.0$ Hz, 1H), 5.09 ( <i>m</i> , 3H), 4.15 ( <i>brd</i> , $J = 6.0$ Hz, 1H), 2.54 ( <i>brd</i> , $J = 13.5$ Hz, 1H), 2.36 ( <i>m</i> , 1H), 2.17 ( <i>m</i> , 1H), 2.16 ( <i>m</i> , 1H), 2.14 ( <i>m</i> , 1H), 2.13 ( <i>m</i> , 6H), 2.06 ( <i>m</i> , 2H), 2.04 ( <i>brt</i> , $J = 1.2$ Hz, 3H), 2.00 ( <i>m</i> , 2H), 1.95 ( <i>m</i> , 2H), 1.68 ( <i>brd</i> , $J = 0.9$ Hz, 3H), 1.64 ( <i>brd</i> , $J = 0.9$ Hz, 3H), 1.62 ( <i>s</i> , 3H), 1.59 ( <i>s</i> , 6H)
$^{13}\text{C}$ NMR( $\text{CDCl}_3$ )( $\delta_{\text{ppm}}$ ) (75 MHz):	196.45, 162.96, 138.54, 135.47, 134.98, 131.29, 126.85, 124.39, 124.17, 123.81, 120.73, 73.71, 43.62, 41.37, 39.85, 39.73, 39.69, 31.01, 26.77, 26.61, 26.47, 25.70, 20.32, 17.69, 16.29, 16.07, 16.01
DEPT 90°	CH : 126.85, 124.39, 124.17, 123.81, 120.73, 73.71, 43.62
DEPT 135°	CH <sub>2</sub> : 41.37, 39.85, 39.73, 39.69, 31.01, 26.77, 26.61, 26.47 CH <sub>3</sub> : 25.70, 20.32, 17.69, 16.29, 16.07, 16.01

**Fraction GF3** Chromatogram characteristics on normal phase TLC with 20%EtOAc/Petrol showed no definite spot under UV and ASA reagent. Further investigation was then not carried out.

**Fraction GF4** Chromatogram characteristics on normal phase TLC with 20%EtOAc/Petrol showed none of well-separated spots under UV and ASA reagent. Therefore, it was not further investigated.

**Fraction GG** Chromatogram characteristics on normal phase TLC with 100%CH<sub>2</sub>Cl<sub>2</sub> showed four UV-active spots with the R<sub>f</sub> values of 0.26, 0.18, 0.15 and 0.09. Because the <sup>1</sup>H NMR data indicated the presence of many compounds, it was not further investigated.

**Fraction GH** Chromatogram characteristics on normal phase TLC with 100%CH<sub>2</sub>Cl<sub>2</sub> showed none of well-separated spots under UV and ASA reagent. It was not further investigated.

**Fraction H** Chromatogram characteristics on normal phase TLC with 100%CHCl<sub>3</sub> showed five UV-active spots with the R<sub>f</sub> values of 0.28, 0.27, 0.21, 0.16 and 0.12. This fraction was separated by column chromatography over silica gel. Elution was conducted initially with 0.5%MeOH/CH<sub>2</sub>Cl<sub>2</sub>, gradually enriched with MeOH and finally with 100%MeOH. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford six subfractions, as shown in Table 27.

**Table 27** Subfractions obtained from the fraction H by column chromatography over silica gel.

Fraction	Mobile phase	Weight (mg)	Physical appearance
HA	0.5%MeOH/CH <sub>2</sub> Cl <sub>2</sub>	10.1	Yellow gum
HB	1.5%MeOH/CH <sub>2</sub> Cl <sub>2</sub>	6.5	Yellow gum
HC	1.5%MeOH/CH <sub>2</sub> Cl <sub>2</sub>	7.0	Yellow gum
HD	3%MeOH/CH <sub>2</sub> Cl <sub>2</sub>	46.4	Yellow-brown gum
HE	3-15%MeOH/CH <sub>2</sub> Cl <sub>2</sub>	93.8	Yellow brown gum
HF	30-100%MeOH/CH <sub>2</sub> Cl <sub>2</sub>	203.6	Yellow-brown gum

**Fraction HA** Chromatogram characteristics on normal phase TLC with 0.25%MeOH/CH<sub>2</sub>Cl<sub>2</sub> showed two UV-active spots with the R<sub>f</sub> values of 0.83 and 0.65. Because the <sup>1</sup>H NMR data indicated the presence of many compounds, it was not further investigated.

**Fraction HB** Chromatogram characteristics on normal phase TLC with 0.25%MeOH/CH<sub>2</sub>Cl<sub>2</sub> showed two UV-active spots with the R<sub>f</sub> values of 0.59 and 0.52. The <sup>1</sup>H NMR data indicated the presence of GP3 and GP4 as major components. Further investigation was then not carried out.

**Fraction HC** Chromatogram characteristics on normal phase TLC with 0.25%MeOH/CH<sub>2</sub>Cl<sub>2</sub> showed three UV-active spots with the R<sub>f</sub> values of 0.55, 0.52 and 0.46. The <sup>1</sup>H NMR data indicated the presence of GP3 and GP5 as major components. Further investigation was then not carried out.

**Fraction HD** Chromatogram characteristics on normal phase TLC with 0.25%MeOH/CH<sub>2</sub>Cl<sub>2</sub> showed three UV-active spots with the R<sub>f</sub> values of 0.48, 0.42 and 0.37. Because the <sup>1</sup>H NMR data indicated the presence of many compounds, it was not further investigated.

**Fraction HE** Chromatogram characteristics on normal phase TLC with 0.25%MeOH/CH<sub>2</sub>Cl<sub>2</sub> showed three UV-active spots with the R<sub>f</sub> values of 0.46, 0.38 and 0.33. Because the <sup>1</sup>H NMR data indicated the presence of many compounds, it was not further investigated.

**Fraction HF** Chromatogram characteristics on normal phase TLC with 0.25%MeOH/CH<sub>2</sub>Cl<sub>2</sub> showed four UV-active spots with the R<sub>f</sub> values of 0.38, 0.37, 0.33 and 0.24. Because the <sup>1</sup>H NMR data indicated the presence of many compounds, it was not further investigated.

**Fraction I** Chromatogram characteristics on normal phase TLC with 100%CHCl<sub>3</sub> showed two UV-active spots with the R<sub>f</sub> values of 0.07 and 0.05. This fraction was separated by column chromatography over silica gel. Elution was conducted initially with 100%CH<sub>2</sub>Cl<sub>2</sub>, gradually enriched with MeOH and finally with 100%MeOH. Fractions with the similar chromatogram characteristics were combined and

evaporated to dryness under reduced pressure to afford nine subfractions, as shown in Table 28.

**Table 28 Subfractions obtained from the fraction I by column chromatography over silica gel**

Fraction	Mobile phase	Weight (mg)	Physical appearance
IA	100%/CH <sub>2</sub> Cl <sub>2</sub>	19.3	Yellow gum
IB	100%/CH <sub>2</sub> Cl <sub>2</sub>	48.2	Yellow gum
IC	0.5%MeOH/CH <sub>2</sub> Cl <sub>2</sub>	23.2	Yellow gum
ID	2%MeOH/CH <sub>2</sub> Cl <sub>2</sub>	16.5	Yellow gum
IE	2%MeOH/CH <sub>2</sub> Cl <sub>2</sub>	13.5	Yellow gum
IF	3%MeOH/CH <sub>2</sub> Cl <sub>2</sub>	24.2	Yellow gum and white solid
IG	3-10%MeOH/CH <sub>2</sub> Cl <sub>2</sub>	57.2	Yellow-brown gum
IH	10-30%MeOH/CH <sub>2</sub> Cl <sub>2</sub>	206.3	Yellow -brown gum
II	30-100%MeOH/CH <sub>2</sub> Cl <sub>2</sub>	2340.9	Yellow-brown gum

**Fraction IA** Chromatogram characteristics on normal phase TLC with 0.25%MeOH/CH<sub>2</sub>Cl<sub>2</sub> showed two UV-active spots with the R<sub>f</sub> values of 0.71 and 0.68. Because the <sup>1</sup>H NMR data indicated the presence of many compounds, it was not further investigated.

**Fraction IB** Chromatogram characteristics on normal phase TLC with 0.25%MeOH/CH<sub>2</sub>Cl<sub>2</sub> showed three UV-active spots with the R<sub>f</sub> values of 0.68, 0.59 and 0.51. The <sup>1</sup>H NMR data indicated the presence of GP3 and GP4 as major components. Further investigation was then not carried out.

**Fraction IC** Chromatogram characteristics on normal phase TLC with 0.25%MeOH/CH<sub>2</sub>Cl<sub>2</sub> showed three UV-active spots with the R<sub>f</sub> values of 0.54, 0.51 and 0.17. The <sup>1</sup>H NMR data indicated the presence of GP4 and GP5 as major components. Further investigation was then not carried out.

**Fraction ID** Chromatogram characteristics on normal phase TLC with 0.25%MeOH/CH<sub>2</sub>Cl<sub>2</sub> showed one major UV-active spot with the same R<sub>f</sub> value as GP5. Further investigation was then not carried out.

**Fraction IE** Chromatogram characteristics on normal phase TLC with 0.25%MeOH/CH<sub>2</sub>Cl<sub>2</sub> showed one major UV-active spot with the same R<sub>f</sub> value as GP12. Further investigation was then not carried out.

**Fraction IF** Chromatogram characteristics on normal phase TLC with 0.25%MeOH/CH<sub>2</sub>Cl<sub>2</sub> showed two UV-active spots with the R<sub>f</sub> values of 0.29 and 0.20. The <sup>1</sup>H NMR data indicated the presence of GP10 and stigmasterol as major components. Further investigation was then not carried out.

**Fraction IG** Chromatogram characteristics on normal phase TLC with 0.25%MeOH/CH<sub>2</sub>Cl<sub>2</sub> showed three UV-active spots with the R<sub>f</sub> values of 0.29, 0.25 and 0.20. The <sup>1</sup>H NMR data indicated the presence of GP10 as a major component. Further investigation was then not carried out.

**Fraction IH** Chromatogram characteristics on normal phase TLC with 0.25%MeOH/CH<sub>2</sub>Cl<sub>2</sub> showed two UV-active spots with the R<sub>f</sub> values of 0.19 and 0.13. This fraction was separated by column chromatography over silica gel. Elution was conducted initially with 100%CH<sub>2</sub>Cl<sub>2</sub>, gradually enriched with MeOH and finally with 10%MeOH/CH<sub>2</sub>Cl<sub>2</sub>. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford four subfractions, as shown in Table 29.

**Table 29** Subfractions obtained from the fraction IH by column chromatography over silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
IH1	100%CH <sub>2</sub> Cl <sub>2</sub>	0.9	Yellow gum
IH2	100%CH <sub>2</sub> Cl <sub>2</sub>	30.9	Yellow gum
IH3	0.25%MeOH/CH <sub>2</sub> Cl <sub>2</sub>	103.9	Yellow gum
IH4	5-10%MeOH/CH <sub>2</sub> Cl <sub>2</sub>	8.4	Yellow-brown gum

**Fraction IH1** Chromatogram characteristics on normal phase TLC with 0.25%MeOH/CH<sub>2</sub>Cl<sub>2</sub> showed no definite spot under UV and ASA reagent. Further investigation was then not carried out.

**Fraction IH2** Chromatogram characteristics on normal phase TLC with 0.25%MeOH/CH<sub>2</sub>Cl<sub>2</sub> showed three UV-active spots with the R<sub>f</sub> values of 0.47, 0.39 and 0.34. Because the <sup>1</sup>H NMR data indicated the presence of many compounds, it was not further investigated.

**Fraction IH3** Chromatogram characteristics on normal phase TLC with 0.25%MeOH/CH<sub>2</sub>Cl<sub>2</sub> showed four UV-active spots with the R<sub>f</sub> values of 0.42, 0.34, 0.25 and 0.22. This fraction was separated by flash column chromatography over silica gel. Elution was conducted with 100%CHCl<sub>3</sub>. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford three subfractions, as shown in Table 30.

**Table 30** Subfractions obtained from the fraction IH3 by flash column chromatography over silica gel

Fraction	Weight (mg)	Physical appearance
IH31	15.9	Yellow gum
IH32	34.5	Yellow gum
IH33	39.7	Yellow gum

**Fraction IH31** Chromatogram characteristics on normal phase TLC with 0.25%MeOH/CH<sub>2</sub>Cl<sub>2</sub> showed one UV-active spot with the R<sub>f</sub> value of 0.22. Because the <sup>1</sup>H NMR data indicated the presence of many compounds, it was not further investigated.

**Fraction IH32** Chromatogram characteristics on normal phase TLC with 0.25%MeOH/CH<sub>2</sub>Cl<sub>2</sub> showed one UV-active spot with the R<sub>f</sub> value of 0.30. It was named as GP14.

UV λ<sub>max</sub>(nm)(MeOH)(log ε)                    223 (3.59), 276 (3.43), 319 (2.82)

FTIR(neat):ν (cm<sup>-1</sup>)                            3373, 1656

<sup>1</sup>H NMR(CDCl<sub>3</sub>)(δ<sub>ppm</sub>)(300 MHz) : 10.72 (*s*, 1H), 6.69 (*s*, 1H), 6.29 (*s*, 1H), 6.23 (*brs*, 1H), 5.59 (*brs*, 1H), 5.53 (*brs*, 1H), 5.25 (*mt*, *J* = 6.9 Hz, 1H), 5.20 (*mt*, *J* = 6.9 Hz, 1H), 5.05 (*mt*, *J* = 6.9 Hz, 1H), 3.57 (*d*, *J* = 6.9 Hz,

	4H), 2.13 ( <i>m</i> , 4H), 1.86 ( <i>s</i> , 3H), 1.83 ( <i>s</i> , 3H), 1.76 ( <i>s</i> , 3H), 1.67 ( <i>s</i> , 3H), 1.60 ( <i>s</i> , 3H)
$^{13}\text{C}$ NMR( $\text{CDCl}_3$ )( $\delta_{\text{ppm}}$ ) (75 MHz):	168.32, 163.20, 162.64, 158.74, 143.41, 141.95, 139.83, 139.36, 136.70, 135.86, 132.24, 123.63, 121.30, 120.15, 119.95, 111.18, 105.39, 100.90, 99.29, 39.65, 26.31, 25.76, 25.69, 23.92, 22.44, 17.99, 17.70, 16.35
DEPT 90°	CH : 123.63, 121.30, 120.15, 105.39, 100.90
DEPT 135°	CH <sub>2</sub> : 39.65, 26.31, 23.92, 22.44 CH <sub>3</sub> : 25.76, 25.69, 17.99, 17.70, 16.35
EIMS ( <i>m/z</i> ) (% rel. int.)	480 (71), 424 (44), 357 (73), 301 (57), 298 (100), 283 (51), 165 (43), 149 (68), 71 (33), 69 (60)

**Fraction IH33** Chromatogram characteristics on normal phase TLC with 0.25%MeOH/ $\text{CH}_2\text{Cl}_2$  showed two UV-active spots with the  $R_f$  values of 0.42 and 0.34. Because the  $^1\text{H}$  NMR data indicated the presence of many compounds, it was not further investigated.

**Fraction IH4** Chromatogram characteristics on normal phase TLC with 0.25%MeOH/ $\text{CH}_2\text{Cl}_2$  showed none of well-separated spots under UV and ASA reagent. It was not further investigated.

**Fraction II** Chromatogram characteristics on normal phase TLC with 0.25%MeOH/ $\text{CH}_2\text{Cl}_2$  showed two UV-active spots with the  $R_f$  values of 0.10 and 0.07. This fraction was separated by column chromatography over Sephadex LH 20. Elution was conducted with 100%MeOH. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford four subfractions, as shown in Table 31.



**Table 31 Subfractions obtained from the fraction II by column chromatography over Sephadex LH 20**

Fraction	Weight (mg)	Physical appearance
II1	737.3	Yellow gum
II 2	584.4	Yellow gum
II3	828.2	Yellow gum
II4	51.7	Yellow-brown gum

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

**Fraction II2** Chromatogram characteristics on normal phase TLC with 20%Acetone/Petrol showed two UV-active spots with the  $R_f$  values of 0.28 and 0.25. Because the  $^1\text{H}$  NMR data indicated the presence of many compounds, it was not further investigated.

**Fraction II3** Chromatogram characteristics on normal phase TLC with 20%Acetone/Petrol showed three UV-active spots with the  $R_f$  values of 0.30, 0.20 and 0.13. This fraction was separated by flash column chromatography over silica gel. Elution was conducted with 100% $\text{CHCl}_3$ . Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford four subfractions, as shown in Table 32.

**Table 32 Subfractions obtained from the fraction II3 by column chromatography over silica gel**

Fraction	Weight (mg)	Physical appearance
II31	14.3	Yellow gum
II32	42.9	Yellow gum
II33	529.3	Yellow gum
II34	169.3	Yellow-brown gum

**Fraction II31** Chromatogram characteristics on normal phase TLC with 20%Acetone/Petrol showed one UV-active spot with the  $R_f$  value of 0.30. Because the  $^1\text{H}$  NMR data indicated the presence of many compounds, it was not further investigated.

**Fraction II32** Chromatogram characteristics on normal phase TLC with 20%Acetone/Petrol showed three UV-active spots with the  $R_f$  values of 0.34, 0.30 and 0.25. This fraction was separated by flash column chromatography over silica gel. Elution was conducted with 10%Acetone/Petrol. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford three subfractions, as shown in Table 33.

**Table 33** Subfractions obtained from the fraction II32 by column chromatography over silica gel

Fraction	Weight (mg)	Physical appearance
II321	5.0	Yellow gum
II322	6.6	Yellow gum
II323	13.3	Yellow gum

**Fraction II321** Chromatogram characteristics on normal phase TLC with 20%Acetone/Petrol showed no definite spot under UV and ASA reagent. Further investigation was then not carried out.

**Fraction II322** Chromatogram characteristics on normal phase TLC with 20%Acetone/Petrol showed one UV-active spot with the  $R_f$  value of 0.34. It was named as **GP15**.

UV  $\lambda_{\text{max}}$ (nm)(MeOH)(log  $\epsilon$ )      243 (4.19), 273 (3.65), 317 (3.61)

FTIR(neat): $\nu$  ( $\text{cm}^{-1}$ )                      3400, 1651

$^1\text{H}$  NMR( $\text{CDCl}_3$ )( $\delta_{\text{ppm}}$ )(300 MHz) : 13.05 (*s*, 1H), 7.72 (*dd*,  $J = 7.5$  and 1.8 Hz, 1H),  
7.23 (*dd*,  $J = 7.5$  and 1.8 Hz, 1H ), 7.16 (*t*,  $J = 7.5$  Hz, 1H), 6.46 (*s*, 1H), 5.29 (*mt*,  $J = 6.6$  Hz,  
1H), 5.08 (*mt*,  $J = 6.6$  Hz, 1H), 3.40 (*d*,  $J = 6.6$

Hz, 2H), 2.07 (*m*, 2H), 1.99 (*m*, 2H), 1.82 (*s*, 3H), 1.65 (*s*, 3H), 1.58 (*s*, 3H)

$^{13}\text{C}$  NMR( $\text{CDCl}_3$ )( $\delta_{\text{ppm}}$ ) (75 MHz): 180.80, 162.80, 160.40, 155.00, 145.30, 145.00, 135.95, 131.28, 124.30, 123.49, 121.80, 121.40, 119.89, 116.09, 110.80, 102.80, 93.38, 39.73, 26.62, 25.51, 21.24, 17.52, 16.05

**Fraction II323** Chromatogram characteristics on normal phase TLC with 20%Acetone/Petrol showed none of well-separated spots under UV and ASA reagent. Thus, it was not further investigated

**Fraction II33** Chromatogram characteristics on normal phase TLC with 20%Acetone/Petrol showed three UV-active spots with the  $R_f$  values of 0.30, 0.20 and 0.15. This fraction was separated by flash column chromatography over silica gel. Elution was conducted with 10%Acetone/Petrol. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford five subfractions, as shown in Table 34.

**Table 34** Subfractions obtained from the fraction II33 by column chromatography over silica gel

Fraction	Weight (mg)	Physical appearance
II331	24.3	Yellow gum
II332	82.1	Yellow gum
II333	79.0	Yellow gum
II334	42.4	Yellow-brown gum
II335	52.3	Yellow-brown gum

**Fraction II331** Chromatogram characteristics on normal phase TLC with 30%Acetone/Petrol showed no definite spot under UV and ASA reagent. Further investigation was then not carried out.

**Fraction II332** Chromatogram characteristics on normal phase TLC with 30%Acetone/Petrol showed one UV-active spot with the  $R_f$

value of 0.30. This fraction was separated by column chromatography over Sephadex LH 20. Elution was conducted with 100%MeOH. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford three subfractions, as shown in Table 35.

**Table 35** Subfractions obtained from the fraction II332 by column chromatography over Sephadex LH 20

Fraction	Weight (mg)	Physical appearance
II3321	29.3	Yellow gum
II3322	11.3	Yellow gum
II3323	10.3	Yellow gum

**Fraction II3321** Chromatogram characteristics on normal phase TLC with 30%Acetone/Petrol showed no definite spot under UV and ASA reagent. Further investigation was then not carried out.

**Fraction II3322** Chromatogram characteristics on normal phase TLC with 30%Acetone/Petrol showed one UV-active spot with the  $R_f$  value of 0.30. It was named as GP16.

UV  $\lambda_{\max}$ (nm)(MeOH)(log  $\epsilon$ ) 239 (4.19), 253 (4.38), 312 (3.61), 351 (2.58)

FTIR(neat): $\nu$  ( $\text{cm}^{-1}$ ) 3366, 1646

$^1\text{H}$  NMR( $\text{CDCl}_3+\text{CD}_3\text{OD}$ )( $\delta_{\text{ppm}}$ )(300 MHz): 13.67 (*brs*, 1H), 6.76 (*s*, 3H), 6.25 (*d*,  $J = 1.8$  Hz, 1H), 6.18 (*d*,  $J = 1.8$  Hz, 1H), 5.25 (*brt*,  $J = 6.6$  Hz, 1H), 5.03 (*brt*,  $J = 6.6$  Hz, 1H), 4.09 (*d*,  $J = 6.6$  Hz, 2H), 3.78 (*s*, 3H), 2.07 (*m*, 2H), 2.00 (*m*, 2H), 1.82 (*s*, 3H), 1.60 (*s*, 3H), 1.54 (*s*, 3H)

$^{13}\text{C}$  NMR( $\text{CDCl}_3+\text{CD}_3\text{OD}$ )( $\delta_{\text{ppm}}$ ) (75 MHz): 181.85, 162.20, 157.03, 155.61, 143.17, 142.70, 137.41, 135.24, 131.10, 124.31, 123.37, 111.67, 103.21, 101.85, 97.93,

93.43, 61.28, 39.69, 26.54, 26.22, 25.49,  
17.53, 16.34

**Fraction II3323** Chromatogram characteristics on normal phase TLC with 30%Acetone/Petrol showed none of well-separated spots under UV and ASA reagent. Therefore, it was not further investigated

**Fraction II333** Chromatogram characteristics on normal phase TLC with 30%Acetone/Petrol showed three UV-active spots with the  $R_f$  values of 0.34, 0.30 and 0.25. This fraction was separated by column chromatography over Sephadex LH 20. Elution was conducted with 100%MeOH. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford four subfractions, as shown in Table 36.

**Table 36 Subfractions obtained from the fraction II333 by column chromatography over Sephadex LH 20**

Fraction	Weight (mg)	Physical appearance
II3331	13.3	Yellow gum
II3332	23.2	Yellow gum
II3333	4.3	Yellow gum
II3334	9.2	Yellow gum

**Fraction II3331** Chromatogram characteristics on normal phase TLC with 30%Acetone/Petrol showed no definite spot under UV and ASA reagent. Further investigation was then not carried out.

**Fraction II3332** Chromatogram characteristics on normal phase TLC with 30%Acetone/Petrol showed none of well-separated spots under UV and ASA reagent. Therefore, it was not further investigated.

**Fraction II3333** Chromatogram characteristics on normal phase TLC with 30%Acetone/Petrol showed one UV-active spot with the  $R_f$  values of 0.25. It was named as GP17.

UV $\lambda_{\max}$ (nm)(MeOH)(log $\epsilon$ )	238 (4.09), 251 (4.08), 312 (3.42), 353 (2.85)
FTIR(neat): $\nu$ (cm <sup>-1</sup> )	3371, 1643
<sup>1</sup> H NMR(CDCl <sub>3</sub> +CD <sub>3</sub> OD)( $\delta_{\text{ppm}}$ )(300 MHz)	:13.63 ( <i>brs</i> , 1H), 6.77 ( <i>s</i> , 1H), 6.26 ( <i>d</i> , <i>J</i> =2.1 Hz, 1H), 6.19 ( <i>d</i> , <i>J</i> = 2.1 Hz, 1H), 5.27 ( <i>mt</i> , <i>J</i> = 6.0 Hz, 1H), 4.09 ( <i>d</i> , <i>J</i> = 6.0 Hz, 2H), 3.79 ( <i>s</i> , 3H), 1.84 ( <i>s</i> , 3H), 1.69 ( <i>s</i> , 3H)
<sup>13</sup> C NMR(CDCl <sub>3</sub> +CD <sub>3</sub> OD)( $\delta_{\text{ppm}}$ ) (75 MHz)	: 181.93, 164.70, 164.00, 156.10, 155.63, 143.10, 137.30, 131.83, 123.30, 112.00, 101.87, 97.93, 92.60, 61.34, 26.34, 25.75, 18.10

**Fraction II3334** Chromatogram characteristics on normal phase TLC with 30%Acetone/Petrol showed none of well-separated spots under UV and ASA reagent. Thus, it was not further investigated

**Fraction II334** Chromatogram characteristics on normal phase TLC with 30%Acetone/Petrol showed one UV-active spot with the  $R_f$  values of 0.34. This fraction was separated by flash column chromatography over silica gel. Elution was conducted with 2%MeOH/CHCl<sub>3</sub>. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford three subfractions, as shown in Table 37.

**Table 37** Subfractions obtained from the fraction II334 by flash column chromatography over silica gel

Fraction	Weight (mg)	Physical appearance
II3341	13.4	Yellow gum
II3342	16.3	Yellow gum
II3343	6.4	Yellow gum

**Fraction II3341** Chromatogram characteristics on normal phase TLC with 30%Acetone/Petrol showed no definite spot under UV and ASA reagent. Further investigation was then not carried out.

**Fraction II3342** Chromatogram characteristics on normal phase TLC with 30%Acetone/Petrol showed one UV-active spot with the  $R_f$  value of 0.34. It was named as **GP18**.

UV $\lambda_{\max}$ (nm)(MeOH)(log $\epsilon$ )	231 (3.69), 281 (3.57), 324 (2.82)
FTIR(neat): $\nu$ ( $\text{cm}^{-1}$ )	3381, 1660
$^1\text{H}$ NMR( $\text{CDCl}_3$ )( $\delta_{\text{ppm}}$ )(300 MHz) :	11.30 ( <i>s</i> , 1H), 6.67 ( <i>s</i> , 1H), 6.47 ( <i>brs</i> , 1H), 6.27 ( <i>s</i> , 1H), 5.73 ( <i>brs</i> , 1H), 5.59 ( <i>brs</i> , 1H), 5.25 ( <i>mt</i> , $J = 7.2$ Hz, 1H), 5.22 ( <i>mt</i> , $J = 7.2$ Hz, 1H), 5.04 ( <i>mt</i> , $J = 6.9$ Hz, 1H), 3.55 ( <i>d</i> , $J = 7.2$ Hz, 2H), 3.39 ( <i>d</i> , $J = 7.2$ Hz, 2H), 2.09 ( <i>m</i> , 2H), 2.06 ( <i>m</i> , 2H), 1.84 ( <i>s</i> , 3H), 1.80 ( <i>s</i> , 3H), 1.75 ( <i>s</i> , 3H), 1.67 ( <i>s</i> , 3H), 1.58 ( <i>s</i> , 3H)
$^{13}\text{C}$ NMR( $\text{CDCl}_3$ )( $\delta_{\text{ppm}}$ ) (75 MHz):	168.70, 162.60, 162.34, 160.08, 143.43, 141.84, 139.82, 139.72, 136.18, 135.68, 132.12, 123.65, 120.76, 120.31, 110.91, 105.09, 100.33, 98.53, 39.68, 26.30, 25.77, 25.65, 23.88, 21.95, 17.98, 17.68, 16.22
DEPT 90°	CH : 123.65, 120.76, 120.31, 105.09, 100.33
DEPT 135°	CH <sub>2</sub> : 39.68, 26.30, 23.88, 21.95 CH <sub>3</sub> : 25.77, 25.65, 17.98, 17.68, 16.22

**Fraction II3343** Chromatogram characteristics on normal phase TLC with 30%Acetone/Petrol showed none of well-separated spots under UV and ASA reagent. Therefore, it was not further investigated

**Fraction II335** Chromatogram characteristics on normal phase TLC with 30%Acetone/Petrol showed none of well-separated spots under UV and ASA reagent. Thus, it was not further investigated

**Fraction II34** Chromatogram characteristics on normal phase TLC with 20%Acetone/Petrol showed none of well-separated spots under UV and ASA reagent. It was not further investigated.

**Fraction II4** Chromatogram characteristics on normal phase TLC with 20%Acetone/Petrol showed none of well-separated spots under UV and ASA reagent. It was not further investigated.

**Fraction J** Chromatogram characteristics on reverse phase TLC with 50%H<sub>2</sub>O/MeOH showed two UV-active spots with the R<sub>f</sub> values of 0.32 and 0.39. This fraction was separated by column chromatography over reverse phase silica gel. Elution was conducted initially with 50%H<sub>2</sub>O/MeOH, gradually enriched with MeOH and finally with 100%MeOH. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford three subfractions, as shown in Table 38.

**Table 38** Subfractions obtained from the fraction J by column chromatography over reverse phase silica gel

Fraction	Mobile phase	Weight (mg)	Physical appearance
JA	50%H <sub>2</sub> O/MeOH	413.4	Brown gum
JB	60-80%H <sub>2</sub> O/MeOH	132.5	Brown gum
JC	60-100%H <sub>2</sub> O/MeOH	216.3	Brown gum

**Fraction JA** Chromatogram characteristics on reverse phase TLC with 50%H<sub>2</sub>O/MeOH showed no definite spot under UV and ASA reagent. Further investigation was then not carried out.

**Fraction JB** Chromatogram characteristics on reverse phase TLC with 50%H<sub>2</sub>O/MeOH showed two UV-active spots with the R<sub>f</sub> values of 0.32 and 0.39. This fraction was separated by column chromatography over Sephadex LH 20. Elution was conducted with 100%MeOH. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford four subfractions, as shown in Table 39.



**Table 39** Subfractions obtained from the fraction JB by column chromatography over Sephadex LH 20

Fraction	Weight (mg)	Physical appearance
JB1	43.2	Yellow-brown gum
JB2	18.4	Yellow-brown gum
JB3	23.2	Yellow-brown gum
JB4	21.6	Yellow-brown gum

**Fraction JB1** Chromatogram characteristics on reverse phase TLC with 50% $\text{H}_2\text{O}$ /MeOH showed no definite spot under UV and ASA reagent. Further investigation was then not carried out.

**Fraction JB2** Chromatogram characteristics on reverse phase TLC with 50% $\text{H}_2\text{O}$ /MeOH showed two UV-active spots with the  $R_f$  values of 0.32 and 0.39. This fraction was separated by column chromatography over Sephadex LH 20. Elution was conducted with 100%MeOH. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford three subfractions, as shown in Table 40.

**Table 40** Subfractions obtained from the fraction JB2 by column chromatography over Sephadex LH 20

Fraction	Weight (mg)	Physical appearance
JB2-1	4.2	Yellow gum
JB2-2	2.1	Yellow gum
JB2-3	10.3	Yellow gum

**Fraction JB2-1** Chromatogram characteristics on reverse phase TLC with 50% $\text{H}_2\text{O}$ /MeOH showed no definite spot under UV and ASA reagent. Further investigation was then not carried out.

**Fraction JB2-2** Chromatogram characteristics on reverse phase TLC with 50% $\text{H}_2\text{O}$ /MeOH showed one UV-active spot with the  $R_f$  value of 0.32. It was named as **GP19**.

UV $\lambda_{\text{max}}$ (nm)(MeOH)(log $\epsilon$ )	236 (3.61), 253 (3.90), 273 (3.40), 312 (2.94), 365 (2.37)
FTIR(neat): $\nu$ ( $\text{cm}^{-1}$ )	3231, 1657
$^1\text{H}$ NMR( $\text{CD}_3\text{OD}$ )( $\delta_{\text{ppm}}$ )(300 MHz)	: 7.56 ( <i>d</i> , $J = 8.7$ Hz, 1H), 6.87 ( <i>d</i> , $J = 8.7$ Hz, 1H), 6.42 ( <i>d</i> , $J = 2.1$ Hz, 1H), 6.16 ( <i>d</i> , $J = 2.1$ Hz, 1H)
$^{13}\text{C}$ NMR( $\text{CD}_3\text{OD}$ )( $\delta_{\text{ppm}}$ )(75 MHz)	: 180.20, 166.00, 163.30, 158.00, 152.30, 146.30, 132.80, 115.40, 112.80, 112.10, 101.40, 97.80, 93.20

**Fraction JB2-3** Chromatogram characteristics on reverse phase TLC with 50% $\text{H}_2\text{O}$ /MeOH showed one UV-active spot with the  $R_f$  value of 0.39. The  $^1\text{H}$  NMR data indicated the presence of **GP20** as a major component. Further investigation was then not carried out.

**Fraction JB3** Chromatogram characteristics on reverse phase TLC with 50% $\text{H}_2\text{O}$ /MeOH showed one UV-active spot with the  $R_f$  values 0.39. This fraction was separated by column chromatography over Sephadex LH 20. Elution was conducted with 100%MeOH. Fractions with the similar chromatogram characteristics were combined and evaporated to dryness under reduced pressure to afford three subfractions, as shown in Table 41.

**Table 41** Subfractions obtained from the fraction **JB3** by column chromatography over Sephadex LH 20

Fraction	Weight (mg)	Physical appearance
JB3-1	1.4	Yellow gum
JB3-2	9.8	Yellow gum
JB3-3	7.2	Yellow gum

**Fraction JB3-1** Chromatogram characteristics on reverse phase TLC with 50% $\text{H}_2\text{O}$ /MeOH showed no definite spots under UV and ASA reagent. Further investigation was then not carried out.

**Fraction JB3-2** Chromatogram characteristics on reverse phase TLC with 50% $\text{H}_2\text{O}$ /MeOH showed one UV-active spot with the  $R_f$  value of 0.39. It was named as GP20.

UV $\lambda_{\text{max}}$ (nm)(MeOH)(log $\epsilon$ )	236 (3.60), 253 (3.72), 272 (3.43), 313 (2.94), 365 (2.41)
FTIR(neat): $\nu$ ( $\text{cm}^{-1}$ )	3231, 1655
$^1\text{H}$ NMR( $\text{CD}_3\text{OD}$ )( $\delta_{\text{ppm}}$ )(300 MHz)	: 7.44 ( <i>s</i> , 1H), 6.82 ( <i>s</i> , 1H), 6.28 ( <i>d</i> , $J = 2.4$ Hz, 1H), 6.15 ( <i>d</i> , $J = 2.4$ Hz, 1H)
$^{13}\text{C}$ NMR( $\text{CD}_3\text{OD}$ )( $\delta_{\text{ppm}}$ )(75 MHz)	: 179.73, 164.84, 162.93, 157.99, 153.77, 151.81, 143.38, 112.47, 107.80, 102.10, 101.94, 97.36, 93.30

**Fraction JB3-3** Chromatogram characteristics on reverse phase TLC with 50% $\text{H}_2\text{O}$ /MeOH showed none of well-separated spots under UV and ASA reagent. Therefore, it was not further investigated.

**Fraction JB4** Chromatogram characteristics on reverse phase TLC with 50% $\text{H}_2\text{O}$ /MeOH showed none of well-separated spots under UV and ASA reagent. Therefore, it was not further investigated.

**Fraction JC** Chromatogram characteristics on reverse phase TLC with 50% $\text{H}_2\text{O}$ /MeOH showed none of well-separated spots under UV and ASA reagent. Therefore, it was not further investigated.

**Fraction K** Chromatogram characteristics on reverse phase TLC with 50% $\text{H}_2\text{O}$ /MeOH showed none of well-separated spots under UV and ASA reagent. Therefore, it was not further investigated.

### 3. RESULTS AND DISCUSSION

The crude methanol extract from the twigs of *G. parvifolia* was separated by chromatographic methods to yield thirteen new compounds: two methyl benzoates (GP2 and GP7), five benzopyrans (GP1, GP3, GP4, GP5 and GP6), two depsidones (GP12 and GP14), three xanthenes (GP8, GP9 and GP11) and one benzocyclooctene (GP10) together with seven known compounds: one depsidone (GP18), five xanthenes (GP15, GP16, GP17, GP19 and GP20) and one cyclohexenone derivative (GP13). The structures were determined by analysis of 1D and 2D NMR spectroscopic data and comparison of the NMR data with those reported in literature.

#### 3.1 Methyl benzoate derivatives

##### 3.1.1 Compound GP7

Compound GP7 with the molecular formula  $C_{18}H_{24}O_5$  from EIMS ( $m/z$  320) (Figure 1) was isolated as a colorless gum. It exhibited UV (Figure 2) absorption bands at 224, 271 and 315 nm while hydroxyl and conjugated ester carbonyl absorption bands were found at 3433 and 1660  $cm^{-1}$ , respectively, in the IR spectrum (Figure 3). The  $^1H$  NMR spectrum (Figure 4) (Table 42) contained signals of one hydroxyl group ( $\delta_H$  5.99, *s*), one aromatic proton ( $\delta_H$  6.00, *s*), one geranyl unit [ $\delta_H$  5.23 (*mt*,  $J = 7.2$  Hz, 1H), 5.05 (*mt*,  $J = 6.9$  Hz, 1H), 3.35 (*d*,  $J = 7.2$  Hz, 2H), 2.07 (*m*, 4H), 1.80 (*s*, 3H) 1.67 (*s*, 3H) and 1.59 (*s*, 3H)] and one methoxyl group ( $\delta_H$  4.04, *s*). The carbon signal at  $\delta_C$  169.98 together with its HMBC correlation (Figure 8) with the methoxy protons confirmed the presence of the methyl ester group. The hydroxyl group ( $\delta_H$  5.99, 2-OH) and the geranyl unit were adjacent and located at C-2 ( $\delta_C$  162.31) and C-3 ( $\delta_C$  105.98), respectively, on the basis of HMBC correlations of 2-OH/C-1 ( $\delta_C$  93.71), C-2 and C-3 and those of H-7 ( $\delta_H$  3.35)/C-2, C-3 and C-4 ( $\delta_C$  162.31). Signal enhancement of 2-OH in the NOEDIFF experiment (Figure 9), upon irradiation of H<sub>2</sub>-7, supported the conclusion. The aromatic proton was attributed to H-5 according to  $^3J$  correlations with C-1 and C-3. Furthermore, a HMBC correlation between H-5 and the carbonyl carbon (C-17) together with the chemical-shift value of

C-1 established the attachment of the ester functionality at C-1. The assigned location of H-5, the ester moiety and the geranyl unit was further confirmed since irradiation of H-5 did not affect any proton signals in the NOEDIFF experiment. Because of no other signals were observed in the  $^1\text{H}$  NMR spectrum, the substituents at C-4 and C-6 were hydroxyl groups. Thus, GP7 was determined as methyl 2,4,6-trihydroxy-3-(3,7-dimethylocta-2,6-dienyl)benzoate, a new methyl benzoate derivative.

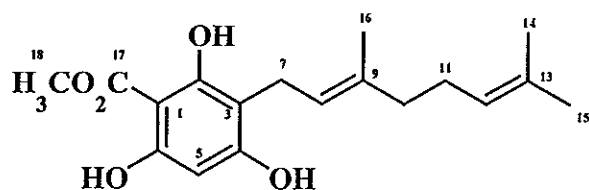


Table 42 The 300 MHz NMR data of compound GP7 in  $\text{CDCl}_3$

Position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$ (C-type)	HMBC correlations	NOE
1		93.71 (C)		
2-OH	5.99 ( <i>s</i> )	162.31 (C)	C-1, C-2, C-3	
3		105.98 (C)		
4-OH		162.31 (C)		
5	6.00 ( <i>s</i> )	96.03 (CH)	C-1, C-3, C-4, C-6, C-17	
6-OH		162.31 (C)		
7	3.35 ( <i>d</i> , 7.2)	21.64 ( $\text{CH}_2$ )	C-2, C-3, C-4, C-8	2-OH, H-8, H-16
8	5.23 ( <i>mt</i> , 7.2)	121.69 (CH)	C-4	H-7, H-10
9		138.72 (C)		
10	2.07 ( <i>m</i> )	39.70 ( $\text{CH}_2$ )	C-8, C-11, C-12	
11	2.07 ( <i>m</i> )	26.39 ( $\text{CH}_2$ )	C-9, C-10, C-13	
12	5.05 ( <i>mt</i> , 6.9)	123.79 (CH)		H-10, H-15
13		131.96 (C)		
14	1.59 ( <i>s</i> )	17.67 ( $\text{CH}_3$ )	C-12, C-13, C-15	H-11

Table 42 (Continued)

Position	$\delta_H$ ( <i>mult.</i> , $J_{Hz}$ )	$\delta_C$ (C-type)	HMBC correlations	NOE
15	1.67 ( <i>s</i> )	25.64 (CH <sub>3</sub> )	C-12, C-13, C-14	H-12
16	1.80 ( <i>s</i> )	16.16 (CH <sub>3</sub> )	C-8, C-9, C-10	H-7, H-11
17		169.98 (C=O)		
18-OCH <sub>3</sub>	4.04 ( <i>s</i> )	52.43 (CH <sub>3</sub> )	C-17	

### 3.1.2 Compound GP2

Compound **GP2** with the molecular formula C<sub>19</sub>H<sub>26</sub>O<sub>5</sub> from EIMS ( $m/z$  334) (**Figure 10**) was isolated as a colorless gum. Its UV (**Figure 11**) and IR (**Figure 12**) absorption bands were almost identical to those of **GP7**. The <sup>1</sup>H NMR spectrum (**Figure 13**) (**Table 43**) contained signals of one aromatic proton ( $\delta_H$  6.07, *s*), one geranyl unit [ $\delta_H$  5.16 (*mt*,  $J = 6.9$  Hz, 1H), 5.07 (*mt*,  $J = 6.9$  Hz, 1H), 3.26 (*d*,  $J = 6.9$  Hz, 2H), 2.00 (*m*, 4H), 1.76 (*s*, 3H) 1.64 (*s*, 3H) and 1.58 (*s*, 3H)] and two methoxyl groups [ $\delta_H$  4.03 (*s*) and 3.83 (*s*)]. The <sup>1</sup>H NMR data were similar to those of **GP7** except for the additional signal of the methoxyl group ( $\delta_H$  3.83) in **GP2**. The methoxyl group was located at C-4 ( $\delta_C$  164.15) on the basis of a HMBC correlation between the methoxy protons with C-4 (**Figure 17**). Signal enhancement of H-5 and H<sub>3</sub>-14 after irradiation at 4-OCH<sub>3</sub> in the NOEDIFF experiment (**Figure 18**) supported the assigned location of the methoxyl group. The remaining HMBC correlations were similar to those found in **GP7**. Thus, **GP2** was determined as methyl 2,6-dihydroxy-4-methoxy-3-(3,7-dimethylocta-2,6-dienyl)benzoate, a new methyl ether derivative of **GP7**.

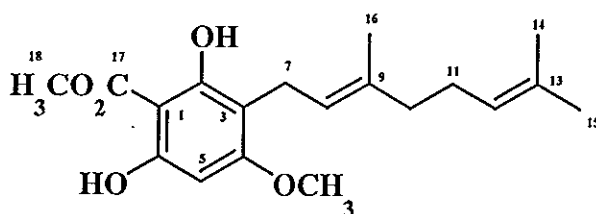


Table 43 The 300 MHz NMR data of compound GP2 in CDCl<sub>3</sub>

Position	$\delta_H$ ( <i>mult.</i> , <i>J</i> <sub>HHz</sub> )	$\delta_C$ (C-type)	HMBC correlations	NOE
1		109.00 (C)		
2-OH		158.60 (C)		
3		93.50 (C)		
3		164.15 (C)		
4-OCH <sub>3</sub>	3.83 ( <i>s</i> )	55.62 (CH <sub>3</sub> )	C-4	H-5, H-14
5	6.07 ( <i>s</i> )	91.57 (CH)	C-1, C-3, C-4, C-6	3-OCH <sub>3</sub>
6-OH		160.80 (C)		
7	3.26 ( <i>d</i> , 6.9)	21.46 (CH <sub>2</sub> )	C-2, C-3, C-4, C-8, C-9	
8	5.16 ( <i>mt</i> , 6.9)	122.53 (CH)		
9		134.82 (C)		
10	2.00 ( <i>m</i> )	39.79 (CH <sub>2</sub> )	C-11	
11	2.00 ( <i>m</i> )	26.75 (CH <sub>2</sub> )		
12	5.07 ( <i>mt</i> , 6.9)	124.48 (CH)		
13		131.37 (C)		
14	1.58 ( <i>s</i> )	17.66 (CH <sub>3</sub> )	C-12, C-13, C-15	
15	1.64 ( <i>s</i> )	25.67 (CH <sub>3</sub> )	C-12, C-13, C-14	
16	1.76 ( <i>s</i> )	16.04 (CH <sub>3</sub> )	C-8, C-9	
17		169.97 (C=O)		
18-OCH <sub>3</sub>	4.03 ( <i>s</i> )	52.41 (CH <sub>3</sub> )	C-17	

### 3.2 Benzopyran derivatives

#### 3.2.1 Compound GP1

Compound GP1 with the molecular formula C<sub>18</sub>H<sub>22</sub>O<sub>5</sub> from EIMS (*m/z* 318) (Figure 19) was isolated as a colorless gum. It exhibited UV absorption bands at 227, 254, 262, 278 and 333 nm (Figure 20), indicating the presence of a longer conjugation than that in GP2 and GP7. The IR absorption bands (Figure 21) were similar to those of GP7. The <sup>1</sup>H NMR spectrum (Figure 22) (Table 44) contained signals of one aromatic proton ( $\delta_H$  5.96, *s*), two olefinic protons of a chromene ring

[ $\delta_H$  6.65 (*dd*,  $J = 10.0$  and  $0.6$  Hz, 1H) and 5.41 (*d*,  $J = 10.0$  Hz, 1H)], one 4-methyl-3-pentenyl unit [ $\delta_H$  5.08 (*mt*,  $J = 7.0$  Hz, 1H), 2.06 (*m*, 2H), 1.69 (*m*, 2H), 1.66 (*s*, 3H) and 1.57 (*s*, 3H)], one methyl group ( $\delta_H$  1.39, *s*) and one methoxyl group ( $\delta_H$  4.03, *s*). The  $^1\text{H}$  NMR data were similar to those of GP7 except for the fact that signals of the geranyl substituent in GP7 were replaced by signals of two olefinic protons of the chromene ring, the 4-methyl-3-pentenyl unit and the methyl group in GP1.  $^3J$  HMBC correlations (Figure 27) between H-3 ( $\delta_H$  5.41) of the chromene ring with C-4a ( $\delta_C$  102.13), C-9 ( $\delta_C$  41.65) of the 4-methyl-3-pentenyl unit and C-15 ( $\delta_C$  27.12) of the quaternary methyl group established the attachment of the chromene ring at C-4a and C-8a ( $\delta_C$  161.00) with an ether linkage at C-8a. These also revealed the linkage of the 4-methyl-3-prenyl unit and H<sub>3</sub>-15 at C-2 of the chromene ring. Signal enhancement of H-4 ( $\delta_H$  6.65), H<sub>2</sub>-10 ( $\delta_H$  2.06) and H<sub>3</sub>-15 ( $\delta_H$  1.39), upon irradiation of H-3 in the NOEDIFF experiment (Figure 28), supported these assignments. In addition, H-8 showed a zig-zag coupling with H-4 in the COSY spectrum (Figure 25). Thus, GP1 was determined as 2-methyl-2-(4-methyl-3-pentenyl)-6-methylcarboxybenzopyran-5,7-diol, a new benzopyran derivative.

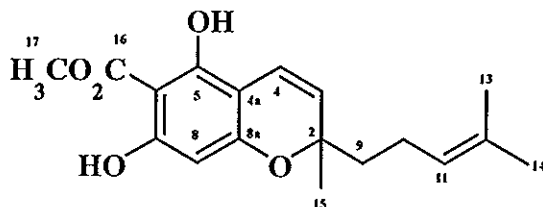


Table 44 The 300 MHz NMR data of compound GP1 in  $\text{CDCl}_3$

Position	$\delta_H$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_C$ (C-type)	HMBC correlations	NOE
2		80.22 (C)		
3	5.41 ( <i>d</i> , 10.0)	124.71 (CH)	C-2, C-4a, C-9, C-15	H-4, H-10, H-15
4	6.65 ( <i>dd</i> , 10.0, 0.6)	116.43 (CH)	C-2, C-4a, C-8a	H-3
4a		102.13 (C)		
5-OH		161.00 (C)		
6		93.37 (C)		



Table 44 (Continued)

Position	$\delta_H$ ( <i>mult.</i> , $J_{Hz}$ )	$\delta_C$ (C-type)	HMBC correlations	NOE
7-OH		161.00 (C)		
8	5.96 ( <i>brs</i> )	96.54 (CH)	C-4a, C-6, C-8a, C-16	
8a		161.00 (C)		
9	1.69 ( <i>m</i> )	41.65 (CH <sub>2</sub> )	C-2, C-3, C-10, C-11, C-15	
10	2.06 ( <i>m</i> )	22.61 (CH <sub>2</sub> )	C-9, C-11, C-12	H-9, H-11, H-13, H-15
11	5.08 ( <i>mt</i> , 7.0)	123.87 (CH)	C-13, C-14	H-10, H-14
12		131.83 (C)		
13	1.57 ( <i>s</i> )	17.61 (CH <sub>3</sub> )	C-11, C-12, C-14	H-10
14	1.66 ( <i>s</i> )	25.65 (CH <sub>3</sub> )	C-11, C-12, C-13	H-11
15	1.39 ( <i>s</i> )	27.12 (CH <sub>3</sub> )	C-2, C-3, C-9	H-3, H-10
16		169.79 (C=O)		
17-OCH <sub>3</sub>	4.03 ( <i>s</i> )	52.46 (CH <sub>3</sub> )	C-16	

### 3.2.2 Compound GP6

Compound GP6 with the molecular formula C<sub>18</sub>H<sub>24</sub>O<sub>6</sub> from EIMS ( $m/z$  336) (Figure 29) was isolated as a colorless gum. Its UV (Figure 30) and IR (Figure 31) absorption bands were almost identical to those of GP7. The <sup>1</sup>H NMR spectrum (Figure 32) (Table 45) contained signals of one aromatic proton ( $\delta_H$  6.00, *s*), one 4-methyl-3-pentenyl unit [ $\delta_H$  5.12 (*mt*,  $J = 7.2$  Hz, 1H), 2.11 (*m*, 2H), 1.60 (*m*, 2H), 1.68 (*s*, 3H) and 1.63 (*s*, 3H)], one oxymethine proton ( $\delta_H$  4.73, *t*,  $J = 8.7$  Hz, 1H), two methylene protons ( $\delta_H$  3.03, *d*,  $J = 8.7$  Hz, 2H), one methyl group ( $\delta_H$  1.29, *s*) and one methoxyl group ( $\delta_H$  4.03, *s*). The <sup>1</sup>H NMR data revealed the replacement of the olefinic proton signal of the chromene unit in GP1 with signal of the oxymethine ( $\delta_H$

4.73) and methylene ( $\delta_{\text{H}}$  3.03) protons in GP6.  $^3J_{\text{HMBC}}$  correlations (Figure 36) of the oxymethine proton/C-9 ( $\delta_{\text{C}}$  36.68) and C-15 ( $\delta_{\text{C}}$  22.72) as well as those of the methylene protons/C-2 ( $\delta_{\text{C}}$  73.70) and C-8a ( $\delta_{\text{C}}$  167.06) established the location of these protons at C-3 and C-4, respectively. The relative configuration of the methyl group at C-2 and the hydroxyl group at C-3 was assigned as *trans* since irradiation of H-3 enhanced the signal intensity of H<sub>3</sub>-15 in the NOEDIFF experiment (Figure 37). Thus, GP6 was determined as 2-methyl-2-(4-methyl-3-pentenyl)-6-methylcarboxy-3,4-dihydrobenzopyran-3,5,7-triol, a new dihydrobenzopyran derivative.

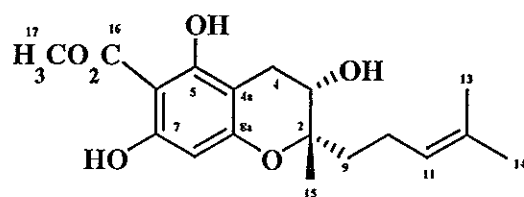


Table 45 The 300 MHz NMR data of compound GP6 in CDCl<sub>3</sub>

Position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$ (C-type)	HMBC correlations	NOE
2		73.70 (C)		
3	4.73 ( <i>t</i> , 8.7)	91.01 (CH)	C-9, C-15	H-4, H-15
4	3.03 ( <i>d</i> , 8.7)	26.65 (CH <sub>2</sub> )	C-2, C-3, C-4a, C-8a	H-3
4a		105.08 (C)		
5-OH		167.06 (C)		
6		93.00 (C)		
7-OH		167.06 (C)		
8	6.00 ( <i>s</i> )	90.83 (CH)	C-16	
8a		167.06 (C)		
9	1.60 ( <i>m</i> )	36.68 (CH <sub>2</sub> )	C-4a, C-6, C-8a	H-11
10	2.11 ( <i>m</i> )	21.93 (CH <sub>2</sub> )	C-11, C-12, C-15	H-11
11	5.12 ( <i>mt</i> , 7.2)	124.03 (CH)	C-9, C-11, C-12	H-14
12		132.20 (C)		
13	1.63 ( <i>s</i> )	17.65 (CH <sub>3</sub> )	C-14	H-10

Table 45 (Continued)

Position	$\delta_H$ ( <i>mult.</i> , <i>J</i> <sub>Hz</sub> )	$\delta_C$ (C-type)	HMBC correlations	NOE
14	1.68 ( <i>s</i> )	25.66 (CH <sub>3</sub> )	C-13	H-11
15	1.29 ( <i>s</i> )	22.72 (CH <sub>3</sub> )	C-2, C-3, C-9	H-3, H-4
16		169.82 (C=O)		
17-OCH <sub>3</sub>	4.03 ( <i>s</i> )	52.36 (CH <sub>3</sub> )	C-16	

### 3.2.3 Compound GP3

Compound GP3 with the molecular formula C<sub>28</sub>H<sub>42</sub>O<sub>2</sub> from EIMS (*m/z* 410) (Figure 38) was isolated as a yellow gum. It exhibited UV (Figure 39) absorption bands at 208, 227 and 298 nm while a hydroxyl absorption band was found at 3420 cm<sup>-1</sup> in the IR spectrum (Figure 40). The <sup>1</sup>H NMR spectrum (Figure 41) (Table 46) contained signals of one aromatic proton ( $\delta_H$  6.36, *brs*), one hydroxyl group ( $\delta_H$  4.35, *brs*), four methylene protons of a chroman ring [ $\delta_H$  2.67 (*t*, *J* = 6.6 Hz, 2H) and 1.75 (*m*, 2H)], one 4,8,12-trimethyltrideca-3,7,11-trienyl unit [ $\delta_H$  5.12 (*m*, 3H), 2.12 (*m*, 2H), 2.07 (*m*, 6H), 1.96 (*m*, 2H), 1.66 (*m*, 1H), 1.54 (*m*, 1H), 1.68 (*s*, 3H), 1.61 (*s*, 3H), 1.60 (*s*, 3H) and 1.58 (*s*, 3H)], two aromatic methyl groups [ $\delta_H$  2.13 (*s*, 6H)] and one quaternary methyl group [ $\delta_H$  1.26 (*s*, 3H)]. The presence of 4,8,12-trimethyltrideca-3,7,11-trienyl moiety was established by COSY (Figure 44), HMQC (Figure 45) and HMBC (Figure 46) correlations. The singlet aromatic proton at  $\delta_H$  6.36 was assigned as H-5, according to <sup>3</sup>*J* HMBC correlations of H-5 with C-4 ( $\delta_C$  22.29), C-7 ( $\delta_C$  125.82), C-8a ( $\delta_C$  145.70) and C-26 ( $\delta_C$  11.85). These also indicated the attachment of the methyl group ( $\delta_H$  2.13, H<sub>3</sub>-26) and the methylene protons ( $\delta_H$  2.67, H<sub>2</sub>-4) at C-6 ( $\delta_C$  121.65) and C-4a ( $\delta_C$  118.24), respectively. The HMBC cross peaks between the other methylene protons ( $\delta_H$  1.75, H<sub>2</sub>-3) of the chroman ring and C-4a, C-9 ( $\delta_C$  39.80) and C-25 ( $\delta_C$  24.10) constructed the chroman ring carrying the methyl group and 4,8,12-trimethyltrideca-3,7,11-trienyl moiety at C-2. These further indicated the formation of the chroman ring at C-4a and C-8a with an ether linkage at

C-8a. Irradiation of H<sub>2</sub>-4 in NOEDIFF (Figure 47) experiment affected signal intensity of H<sub>2</sub>-3, H-5 and H<sub>3</sub>-25. The location of the remaining aromatic methyl group was assigned at C-7 ( $\delta_C$  125.82) on the basis of the chemical-shift values of C-8 ( $\delta_C$  146.28) and C-8a which suggested the presence of two adjacent oxysubstituents. Thus, GP3 was determined as 2,6,7-trimethyl-2-(4,8,12-trimethyltrideca-3,7,11-trienyl)-3,4-dihydrobenzopyran-8-ol, a new dihydrobenzopyran derivative.

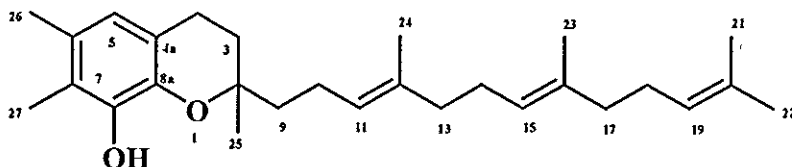


Table 46 The 300 MHz NMR data of compound GP3 in CDCl<sub>3</sub>

Position	$\delta_H$ ( <i>mult.</i> , $J_{Hz}$ )	$\delta_C$ (C-type)	HMBC correlations	NOE
2		75.23 (C)		
3	1.75 ( <i>m</i> )	31.44 (CH <sub>2</sub> )	C-2, C-4, C-4a, C-9, C-25	
4	2.67 ( <i>t</i> , 6.6)	22.29 (CH <sub>2</sub> )	C-2, C-3, C-4a, C-5, C-8a	H-3, H-5, H-25
4a		118.24 (C)		
5	6.36 ( <i>brs</i> )	112.16 (CH)	C-4, C-6, C-7, C-8a, C-26	H-4, H-26
6		121.65 (C)		
7		125.82 (C)		
8-OH	4.35 ( <i>brs</i> )	146.28 (C)		
8a		145.70 (C)		
9	1.66 ( <i>m</i> ) 1.54 ( <i>m</i> )	39.80 (CH <sub>2</sub> )	C-2, C-3, C-25	
10	2.07 ( <i>m</i> )	26.77 (CH <sub>2</sub> ) <sup>a</sup>	C-2, C-11, C-12	
11	5.12 ( <i>m</i> )	124.36 (CH) <sup>b</sup>	C-9	
12		135.09 (C) <sup>c</sup>		

Table 46 (Continued)

Position	$\delta_H$ ( <i>mult.</i> , $J_{Hz}$ )	$\delta_C$ (C-type)	HMBC correlations	NOE
13	2.12 ( <i>m</i> )	39.80 (CH <sub>2</sub> )		
14	2.07 ( <i>m</i> )	26.61 (CH <sub>2</sub> ) <sup>a</sup>	C-15, C-16	H-23
15	5.12 ( <i>m</i> )	124.42 (CH) <sup>b</sup>	C-13, C-23	
16		134.96 (C) <sup>c</sup>		
17	1.96 ( <i>m</i> )	39.72 (CH <sub>2</sub> )	C-15, C-16, C-23	
18	2.07 ( <i>m</i> )	26.77 (CH <sub>2</sub> ) <sup>a</sup>	C-19, C-20	H-21
19	5.12 ( <i>m</i> )	124.21 (CH) <sup>b</sup>	C-18	
20		131.25 (C)		
21	1.60 ( <i>s</i> )	17.68 (CH <sub>3</sub> )	C-19, C-20, C-22	H-18
22	1.68 ( <i>s</i> )	25.69 (CH <sub>3</sub> )	C-19, C-20, C-21	
23	1.58 ( <i>s</i> )	16.00 (CH <sub>3</sub> )	C-15, C-16, C-17	
24	1.61 ( <i>s</i> )	15.89 (CH <sub>3</sub> )	C-11, C-12, C-13	
25	1.26 ( <i>s</i> )	24.01 (CH <sub>3</sub> )	C-2, C-3, C-9	H-3, H-4
26	2.13 ( <i>s</i> )	11.85 (CH <sub>3</sub> )	C-5, C-6, C-7	H-5
27	2.13 ( <i>s</i> )	11.90 (CH <sub>3</sub> )	C-6, C-7, C-8	

<sup>a,b,c</sup> Chemical shifts with the same index may be interchanged.

### 3.2.4 Compound GP5

Compound GP5 with the molecular formula C<sub>27</sub>H<sub>40</sub>O<sub>2</sub> from EIMS ( $m/z$  396) (Figure 48) was isolated as a colorless gum. It exhibited UV (Figure 49) and IR (Figure 50) absorption bands almost identical to those of GP3. The <sup>1</sup>H NMR spectrum (Figure 51) (Table 47) contained signals of two *meta*-aromatic protons [ $\delta_H$  6.47 (*d*,  $J = 2.7$  Hz, 1H) and 6.37 (*d*,  $J = 2.7$  Hz, 1H)], four methylene protons of a chroman ring [ $\delta_H$  2.69 (*t*,  $J = 6.6$  Hz, 2H) and 1.76 (*m*, 2H)], one 4,8,12-trimethyltrideca-3,7,11-trienyl unit [ $\delta_H$  5.11 (*m*, 3H), 2.12 (*m*, 2H), 2.07 (*m*, 2H), 2.01 (*m*, 2H), 1.97 (*m*, 4H), 1.69 (*m*, 1H), 1.53 (*m*, 1H), 1.68 (*s*, 3H), 1.60 (*s*, 3H), 1.59 (*s*, 3H) and 1.58 (*s*, 3H)], one aromatic methyl group [ $\delta_H$  2.12 (*s*, 3H)] and one

oxyquaternary methyl group [ $\delta_{\text{H}}$  1.26 (*s*, 3H)]. The  $^1\text{H}$  NMR data were similar to those of GP3 except that signals for the aromatic proton and one of the aromatic methyl signals in GP3 were replaced by signals of two *meta*-aromatic protons ( $\delta_{\text{H}}$  6.47 and 6.37) in GP5. The higher field aromatic proton was attributed to H-5 according to its  $^3J$  HMBC correlations (Figure 55) with C-4 ( $\delta_{\text{C}}$  22.49), C-7 ( $\delta_{\text{C}}$  115.66), C-8a ( $\delta_{\text{C}}$  145.99) and C-26 ( $\delta_{\text{C}}$  16.00). The other *meta*-aromatic proton and the aromatic methyl group were then located at C-7 and C-6 ( $\delta_{\text{C}}$  127.36), respectively. Signal enhancement of both H-4 and H<sub>3</sub>-26 after irradiation of H-5 in the NOEDIFF experiment (Figure 56) supported above assignment. The remaining HMBC correlations and NOEDIFF results were similar to those of GP3. Thus, GP5 was determined as 2,6-dimethyl-2-(4,8,12-trimethyltrideca-3,7,11-trienyl)-3,4-dihydrobenzopyran-8-ol, a new dihydro-benzopyran derivative.

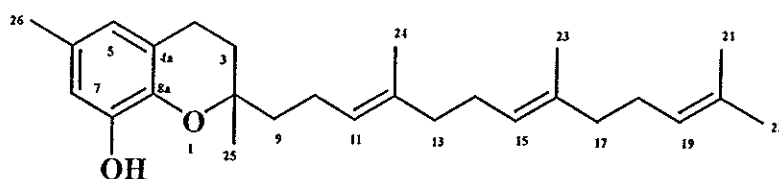


Table 47 The 300 MHz NMR data of compound GP5 in  $\text{CDCl}_3$

Position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$ (C-type)	HMBC correlations	NOE
2		75.34 (C)		
3	1.76 ( <i>m</i> )	31.38 ( $\text{CH}_2$ )	C-2, C-4, C-4a, C-9, C-25	
4	2.69 ( <i>t</i> , 6.6)	22.49 ( $\text{CH}_2$ )	C-2, C-3, C-4a, C-5, C-8, C-8a	H-5, H-25
4a		121.25 (C)		
5	6.37 ( <i>d</i> , 2.7)	112.61 (CH)	C-4, C-7, C-8a, C-26	H-4, H-26
6		127.36 (C)		

Table 47 (Continued)

Position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$ (C-type)	HMBC correlations	NOE
7	6.47 ( <i>d</i> , 2.7)	115.66 (CH)	C-5, C-8, C-8a, C-26	H-26
8-OH		147.75 (C)		
8a		145.99 (C)		
9	1.69 ( <i>m</i> ) 1.53 ( <i>m</i> )	39.69 (CH <sub>2</sub> ) <sup>a</sup>	C-2, C-3, C-10, C-25	
10	2.12 ( <i>m</i> )	22.18 (CH <sub>2</sub> )	C-2, C-11, C-12	H-24
11	5.11 ( <i>m</i> )	124.31 (CH) <sup>b</sup>	C-9	
12		135.13 (C) <sup>c</sup>		
13	1.97 ( <i>m</i> )	39.71 (CH <sub>2</sub> ) <sup>a</sup>	C-11, C-12, C-14	
14	2.07 ( <i>m</i> )	26.60 (CH <sub>2</sub> )		
15	5.11 ( <i>m</i> )	124.42 (CH) <sup>b</sup>	C-13, C-14	
16		134.97 (C) <sup>c</sup>		
17	1.97 ( <i>m</i> )	39.71 (CH <sub>2</sub> ) <sup>a</sup>	C-15, C-16, C-18	
18	2.01 ( <i>m</i> )	26.77 (CH <sub>2</sub> )	C-17	
19	5.11 ( <i>m</i> )	124.31 (CH) <sup>b</sup>	C-21, C-22	
20		131.25 (C)		
21	1.60 ( <i>s</i> )	15.87 (CH <sub>3</sub> )	C-19, C-20, C-22	
22	1.68 ( <i>s</i> )	25.69 (CH <sub>3</sub> )	C-19, C-20	H-19
23	1.59 ( <i>s</i> )	17.68 (CH <sub>3</sub> )	C-15, C-16	
24	1.58 ( <i>s</i> )	16.04 (CH <sub>3</sub> ) <sup>d</sup>	C-11, C-12	
25	1.26 ( <i>s</i> )	24.03 (CH <sub>3</sub> )	C-2, C-3, C-9	H-4, H-9, H-10
26	2.12 ( <i>s</i> )	16.00 (CH <sub>3</sub> ) <sup>d</sup>	C-5, C-6, C-7	H-5, H-7

<sup>a,b,c,d</sup> Chemical shifts with the same index may be interchanged.

### 3.2.5 Compound GP4

Compound GP4 was isolated as a colorless gum. It showed the molecular ion at  $m/z$  426 ( $C_{28}H_{42}O_3$ ) from EIMS (Figure 57), which was higher than that of GP3 by one oxygen atom. Its UV (Figure 58) and IR (Figure 59) absorption bands were almost identical to those of GP3. The  $^1H$  NMR spectrum (Figure 60) (Table 48) contained signals of one hydroxyl group ( $\delta_H$  4.44, *brs*), four methylene protons of a chroman ring [ $\delta_H$  2.32 (*m*, 1H), 2.11 (*m*, 1H), 1.68 (*m*, 2H)], one 4,8,12-trimethyltrideca-3,7,11-trienyl unit [ $\delta_H$  5.14 (*m*, 1H), 5.10 (*m*, 2H), 2.11 (*m*, 2H), 2.07 (*m*, 4H), 2.00 (*m*, 4H), 1.59 (*m*, 2H), 1.68 (*s*, 3H), 1.60 (*brs*, 3H) and 1.59 (*s*, 6H)], two aromatic methyl groups [ $\delta_H$  2.20 (*s*, 3H) and 2.19 (*s*, 3H)] and one oxyquarternary methyl group [ $\delta_H$  1.26 (*s*, 3H)]. The  $^1H$  NMR data were similar to those of GP3 except for the disappearance of the aromatic proton in GP4. The EIMS data revealed the presence of an additional hydroxyl group in GP4. HMBC correlations (Figure 64) of H<sub>2</sub>-4 ( $\delta_H$  2.32 and 2.11)/C-4a ( $\delta_C$  117.28) and C-5 ( $\delta_C$  115.17) and those of H<sub>3</sub>-26 ( $\delta_H$  2.20)/C-4a, C-5 and C-6 ( $\delta_C$  126.89) established the linkage of the methyl group at C-5. The other aromatic methyl group ( $\delta_C$  2.19) was attached to C-8 ( $\delta_C$  122.22) on the basis of HMBC correlations of H<sub>3</sub>-27/C-7 ( $\delta_C$  144.72), C-8 and C-8a ( $\delta_C$  145.86) and the chemical-shift values of C-6, C-7 and C-8a. Signal enhancement of H<sub>3</sub>-27 after irradiation of H<sub>3</sub>-25 in the NOEDIFF experiment (Figure 65) supported above assignment. Thus, GP4 was determined as 2,5,8-trimethyl-2-(4,8,12-trimethyltrideca-3,7,11-trienyl)-3,4-dihydrobenzopyran-6,7-diol, a new dihydrobenzopyran derivative.

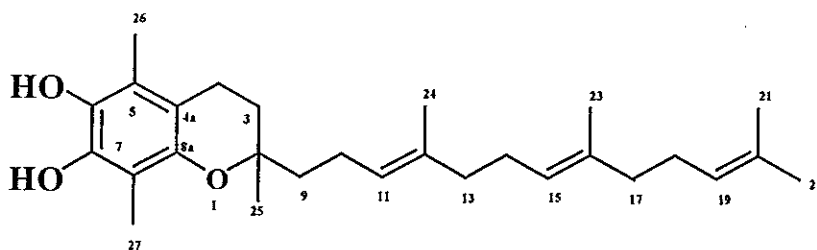




Table 48 The 300 MHz NMR data of compound GP4 in CDCl<sub>3</sub>

Position	$\delta_H$ ( <i>mult.</i> , $J_{Hz}$ )	$\delta_C$ (C-type)	HMBC correlations	NOE
2		74.96 (C)		
3	1.68 ( <i>m</i> )	31.27 (CH <sub>2</sub> )	C-4, C-4a, C-25	
4	2.32( <i>m</i> ) 2.11( <i>m</i> )	20.62 (CH <sub>2</sub> )	C-2, C-3, C-4a, C-5	H-9, H-25
4a		117.28 (C)		
5		115.17 (C)		
6-OH		126.89 (C)		
7-OH	4.44 ( <i>brs</i> )	144.72 (C)	C-7, C-8	
8		122.22 (C)		
8a		145.86 (C)		
9	1.59 ( <i>m</i> )	39.82 (CH <sub>2</sub> )	C-3, C-10, C-25	
10	2.11( <i>m</i> )	22.20 (CH <sub>2</sub> )	C-11, C-12	
11	5.10 ( <i>m</i> )	124.24 (CH)	C-9, C-13, C-24	
12		135.20 (C)		
13	2.00 ( <i>m</i> )	39.72 (CH <sub>2</sub> )	C-11, C-12, C-14, C-15, C-24	
14	2.07 ( <i>m</i> )	26.76 (CH <sub>2</sub> )	C-13, C-15, C-16	
15	5.14 ( <i>m</i> )	124.18 (CH)	C-13, C-17, C-23	
16		134.97 (C)		
17	2.00 ( <i>m</i> )	39.72 (CH <sub>2</sub> )	C-15, C-18, C-19, C-23	
18	2.07 ( <i>m</i> )	26.65 (CH <sub>2</sub> )	C-16, C-19, C-20	
19	5.10 ( <i>m</i> )	124.39 (CH)	C-17, C-18, C-20, C-21	

Table 48 (Continued)

Position	$\delta_H$ ( <i>mult.</i> , $J_{Hz}$ )	$\delta_C$ (C-type)	HMBC correlations	NOE
20		131.24 (C)		
21	1.60 ( <i>brs</i> )	17.68 (CH <sub>3</sub> )	C-19, C-20, C-22	
22	1.68 ( <i>s</i> )	25.69 (CH <sub>3</sub> )	C-19, C-20, C-21	
23	1.59 ( <i>s</i> )	15.85 (CH <sub>3</sub> ) <sup>a</sup>	C-15, C-16, C-17	
24	1.59 ( <i>s</i> )	16.00 (CH <sub>3</sub> ) <sup>a</sup>	C-11, C-12, C-13	
25	1.26 ( <i>s</i> )	23.83 (CH <sub>3</sub> )	C-2, C-3, C-9	H-3, H-4, H-9, H-27
26	2.20 ( <i>s</i> )	11.98 (CH <sub>3</sub> ) <sup>b</sup>	C-4a, C-5, C-6	
27	2.19 ( <i>s</i> )	12.25 (CH <sub>3</sub> ) <sup>b</sup>	C-7, C-8, C-8a	

<sup>a,b</sup> Chemical shifts with the same index may be interchanged.

### 3.3 Depsidone derivatives

#### 3.3.1 Compound GP18

Compound GP18 was isolated as a yellow gum. It exhibited UV (Figure 66) absorption bands at 231, 281 and 324 nm while the hydroxyl and conjugated carbonyl absorption bands were found at 3381 and 1660  $\text{cm}^{-1}$ , respectively, in the IR spectrum (Figure 67). The <sup>1</sup>H NMR spectrum (Figure 68) (Table 49) contained signals of one chelated hydroxyl group ( $\delta_H$  11.30, *s*), two aromatic protons [ $\delta_H$  6.67 (*s*, 1H) and 6.27 (*s*, 1H)], one geranyl unit [ $\delta_H$  5.22 (*mt*,  $J = 7.2$  Hz, 1H), 5.04 (*mt*,  $J = 6.9$  Hz, 1H), 3.39 (*d*,  $J = 7.2$  Hz, 2H), 2.09 (*m*, 2H), 2.06 (*m*, 2H), 1.80 (*s*, 3H), 1.67 (*s*, 3H) and 1.58 (*s*, 3H)] and one prenyl unit [ $\delta_H$  5.25 (*mt*,  $J = 7.2$  Hz, 1H), 3.55 (*d*,  $J = 7.2$  Hz, 2H), 1.84 (*s*, 3H) and 1.75 (*s*, 3H)]. The chemical-shift values of the chelated hydroxy proton ( $\delta_H$  11.30) and the ester carbonyl carbon ( $\delta_C$  168.70) indicated that GP18 had a depsidone chromophore. In the HMBC spectrum (Figure 72), the chelated hydroxy proton, which was located at the peri-position to the lactone carbonyl group, showed correlations with C-1 ( $\delta_C$  162.60), C-2 ( $\delta_C$  110.91) and C-11a ( $\delta_C$  98.53). The geranyl unit was then linked at C-2 on the basis of HMBC correlations between H<sub>2</sub>-12 ( $\delta_H$

3.39)/C-1, C-2 and C-3 ( $\delta_C$  162.34). The aromatic proton ( $\delta_H$  6.27) was located at C-4 ( $\delta_C$  100.33) according to the HMBC correlations of H-4/C-2, C-3, C-4a ( $\delta_C$  160.08) and C-11a. The remaining aromatic proton at  $\delta_H$  6.67 was attributed to H-6 due to its HMBC correlations with C-5a ( $\delta_C$  141.84), C-7 ( $\delta_C$  143.43), C-8 ( $\delta_C$  136.18) and C-9a ( $\delta_C$  139.82).  $^3J$  HMBC correlations between the methylene protons [H<sub>2</sub>-22 ( $\delta_H$  3.35)] and C-8 and C-9a established the attachment of the prenyl unit at C-9 ( $\delta_C$  135.68). According to the chemical-shift values of C-3, C-7 and C-8, these carbons carried hydroxyl substituents. Signal enhancement of H-6 in the NOEDIFF experiment (Figure 73), upon irradiation of H-4, supported above assignment. Thus, GP18 was 1,3,7,8-tetrahydroxy-9-(3-methylbut-2-enyl)-2-(3,7-dimethylocta-2,6-dienyl)dibenzo-*[b,e]*[1,4]dioxepin-11-one (garcidepsidone B), previously isolated from leaves of *Garcinia parvifolia* (Xu, 2000).

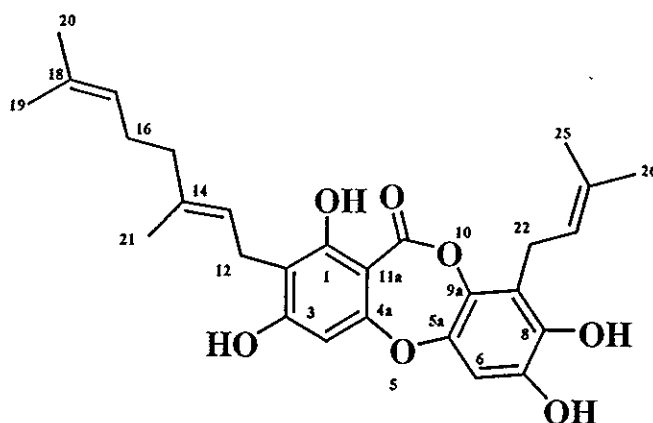


Table 49 The 300 MHz NMR data of compound GP18 in CDCl<sub>3</sub>

Position	$\delta_H$ ( <i>mult.</i> , $J_{Hz}$ )	$\delta_C$ (C-type)	HMBC correlations	NOE
1-OH	11.30 ( <i>s</i> )	162.60 (C)	C-1, C-2, C-11a	
2		110.91 (C)		
3-OH		162.34 (C)		
4	6.27 ( <i>s</i> )	100.33 (CH)	C-2, C-3, C-4a, C-11a	H-6

Table 49 (Continued)

Position	$\delta_{\text{H}}$ ( <i>mult.</i> , J <sub>Hz</sub> )	$\delta_{\text{C}}$ (C-type)	HMBC correlations	NOE
4a		160.08 (C)		
5a		141.84 (C) <sup>a</sup>		
6	6.67 ( <i>s</i> )	105.09 (CH)	C-5a, C-7, C-8, C-9a	H-4
7-OH		143.43 (C) <sup>a</sup>		
8-OH		136.18 (C) <sup>b</sup>		
9		135.68 (C)		
9a		139.82 (C) <sup>b</sup>		
11		168.70 (C=O)		
11a		98.53 (C)		
12	3.39 ( <i>d</i> , 7.2)	21.95 (CH <sub>2</sub> )	C-1, C-2, C-3, C-13, C-14	H-13, H-21
13	5.22 ( <i>mt</i> , 7.2)	120.76 (CH)	C-12, C-15, C-21	
14		139.72 (C)		
15	2.09 ( <i>m</i> )	39.68 (CH <sub>2</sub> )	C-13, C-14, C-16, C-17, C-21	
16	2.06 ( <i>m</i> )	26.30 (CH <sub>2</sub> )	C-14, C-16, C-17	
17	5.04 ( <i>mt</i> , 6.9)	123.65 (CH)	C-15, C-16, C-19, C-20	
18		132.12 (C)		
19	1.58 ( <i>s</i> )	17.68 (CH <sub>3</sub> )	C-17, C-18, C-20	
20	1.67 ( <i>s</i> )	25.65 (CH <sub>3</sub> )	C-17, C-18, C-19	
21	1.80 ( <i>s</i> )	16.22 (CH <sub>3</sub> )	C-13, C-14, C-15	
22	3.55 ( <i>d</i> , 7.2)	23.88 (CH <sub>2</sub> )	C-8, C-9a, C-23, C-26	H-23, H-25
23	5.25 ( <i>mt</i> , 7.2)	120.31 (CH)	C-26, C-25	H-22, H-26
24		136.18 (C)		
25	1.84 ( <i>s</i> )	17.98 (CH <sub>3</sub> )	C-23, C-24, C-26	
26	1.75 ( <i>s</i> )	25.77 (CH <sub>3</sub> )	C-23, C-24, C-25	

<sup>a,b</sup> Chemical shifts with the same index may be interchanged.

### 3.3.2 Compound GP14

Compound GP14 with the molecular formula  $C_{28}H_{32}O_7$  from EIMS ( $m/z$  480) (Figure 74) was isolated as a yellow gum. It exhibited UV (Figure 75) and IR (Figure 76) absorption bands similar to those of GP18. The  $^1H$  NMR spectrum (Figure 77) (Table 50) contained signals of one chelated hydroxyl group ( $\delta_H$  10.72, *s*), two aromatic protons [ $\delta_H$  6.69 (*s*, 1H) and 6.29 (*s*, 1H)], three hydroxyl groups [ $\delta_H$  6.23 (*brs*, 1H), 5.59 (*brs*, 1H) and 5.53 (*brs*, 1H)], one prenyl unit [ $\delta_H$  5.25 (*mt*,  $J = 6.9$  Hz, 1H), 3.57 (*d*,  $J = 6.9$  Hz, 2H), 1.83 (*s*, 3H) and 1.76 (*s*, 3H)] and one geranyl unit [ $\delta_H$  5.20 (*mt*,  $J = 6.9$  Hz, 1H), 5.05 (*mt*,  $J = 6.9$  Hz, 1H), 3.57 (*d*,  $J = 6.9$  Hz, 2H), 2.13 (*m*, 4H), 1.86 (*s*, 3H), 1.67 (*s*, 3H) and 1.60 (*s*, 3H)]. The  $^1H$  NMR data were similar to those of GP18 except for the differences in the HMBC correlations (Figure 81) observed in left hand ring.  $^3J$  HMBC correlations of the aromatic proton  $\delta_H$  6.29/C-4 ( $\delta_C$  111.18) and C-11a ( $\delta_C$  99.29) established the attachment of the aromatic proton at C-2 ( $\delta_C$  100.90). The location of the geranyl group at C-4 was confirmed by  $^3J$  HMBC correlations of the methylene protons ( $\delta_H$  3.57)/C-3 ( $\delta_C$  162.64) and C-4a ( $\delta_C$  158.74). Signal enhancement of H<sub>2</sub>-12 ( $\delta_H$  3.57), H-13 ( $\delta_H$  5.20) and H<sub>3</sub>-21 ( $\delta_H$  1.86) in the NOEDIFF experiment (Figure 82) after irradiation of H-6 ( $\delta_H$  6.69) confirmed the close proximity of H-6 and the geranyl group. The remaining HMBC correlations and NOEDIFF results were similar to those found in GP18. Therefore, GP14 was determined as 1,3,7,8-tetrahydroxy-9-(3-methylbut-2-enyl)-4-(3,7-dimethylocta-2,6-dienyl)dibenzo[*b,e*][1,4]dioxepin-11-one, a new depsidone derivative.

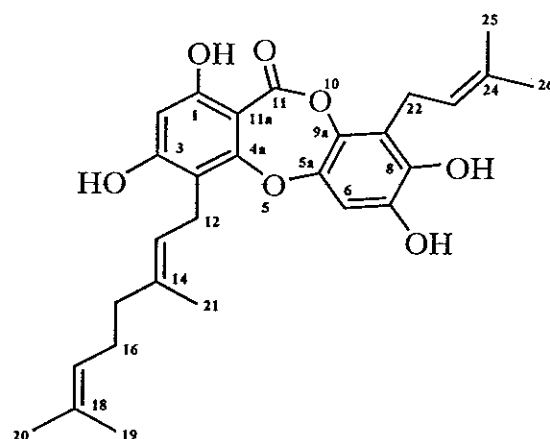


Table 50 The 300 MHz NMR data of compound GP14 in CDCl<sub>3</sub>

Position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$ (C-type)	HMBC correlations	NOE
1-OH	10.72 ( <i>s</i> )	163.20 (C)	C-1, C-2, C-11, C-11a	
2	6.29 ( <i>s</i> )	100.90 (CH)	C-4, C-11a, C-12	
3-OH	6.23 ( <i>brs</i> )	162.64 (C)	C-2, C-3, C-4	
4		111.18 (C)		
4a		158.74 (C)		
5a		143.41 (C)		
6	6.69 ( <i>s</i> )	105.39 (CH)	C-5a, C-7, C-8, C-9a	H-12, H-13, H-21
7-OH	5.53 ( <i>brs</i> )	141.95 (C)	C-6, C-7, C-8	
8-OH	5.59 ( <i>brs</i> )	139.83 (C)	C-7, C-8, C-9	
9		119.95 (C)		
9a		135.86 (C)		
11		168.32 (C)		
11a		99.29 (C)		
12	3.57 ( <i>d</i> , 6.9)	22.44 (CH <sub>2</sub> )	C-3, C-4, C-4a, C-13, C-14, C-21	H-6, H-13, H-21
13	5.20 ( <i>mt</i> , 6.9)	121.30 (CH)	C-4, C-12, C-15, C-21	H-6, H-12, H-15

Table 50 (Continued)

Position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$ (C-type)	HMBC correlations	NOE
14		139.36 (C)		
15	2.13 ( <i>m</i> )	39.65 (CH <sub>2</sub> )	C-13, C-14, C-16, C-17	
16	2.13 ( <i>m</i> )	26.31 (CH <sub>2</sub> )	C-14, C-15, C-17, C-18	
17	5.05 ( <i>mt</i> , 6.9)	123.63 (CH)	C-19	H-15, H-16, H-20
18		132.24 (C)		
19	1.60 ( <i>s</i> )	17.70 (CH <sub>3</sub> )	C-17, C-18, C-20	H-16
20	1.67 ( <i>s</i> )	25.69 (CH <sub>3</sub> )	C-17, C-18	H-17
21	1.86 ( <i>s</i> )	16.35 (CH <sub>3</sub> )	C-13, C-14, C-15	H-6, H-12, H-16
22	3.57 ( <i>d</i> , 6.9)	23.92 (CH <sub>2</sub> )	C-8, C-9a, C-22, C-23	H-23, H-25
23	5.25 ( <i>mt</i> , 6.9)	120.15 (CH)	C-22, C-25, C-26	H-22, H-26
24		136.70 (C)		
25	1.83 ( <i>s</i> )	17.99 (CH <sub>3</sub> )	C-23, C-24, C-26	H-22
26	1.76 ( <i>s</i> )	25.76 (CH <sub>3</sub> )	C-23, C-24, C-25	H-23

### 3.3.3 Compound GP12

Compound GP12 with the molecular formula C<sub>28</sub>H<sub>30</sub>O<sub>7</sub> from EIMS ( $m/z$  478) (Figure 83) was isolated as a yellow gum. The UV (Figure 84) and IR (Figure 85) absorption bands indicated the presence of the depsidone chromophore. The <sup>1</sup>H NMR spectrum (Figure 86) (Table 51) contained signals of one chelated hydroxyl proton ( $\delta_{\text{H}}$  11.36, *s*), two aromatic protons [ $\delta_{\text{H}}$  6.69 (*s*, 1H) and 6.29 (*s*, 1H)], two hydroxyl groups [ $\delta_{\text{H}}$  6.26 (*brs*, 1H) and 5.39 (*brs*, 1H)], two olefinic protons of a dimethyl chromene ring [ $\delta_{\text{H}}$  6.76 (*d*,  $J = 10.2$  Hz, 1H), 5.75 (*d*,  $J = 10.2$  Hz, 1H) and 1.46 (*s*, 6H)] and one geranyl unit [ $\delta_{\text{H}}$  5.23 (*mt*,  $J = 7.2$  Hz, 1H), 5.04 (*mt*,  $J = 6.6$  Hz, 1H),

3.41 (*d*,  $J = 7.2$  Hz, 2H), 2.08 (*m*, 4H), 1.80 (*s*, 3H), 1.68 (*s*, 3H) and 1.59 (*s*, 3H)]. The  $^1\text{H}$  NMR data were similar to those of **GP18** except for the fact that signals of the prenyl substituent in **GP18** were replaced by signals of the dimethylchromene ring in **GP12**. The HMBC correlations (Figure 90) between olefinic proton, H-22 ( $\delta_{\text{H}}$  6.76), with C-8 ( $\delta_{\text{C}}$  136.45), C-9a ( $\delta_{\text{C}}$  132.58) and C-24 ( $\delta_{\text{C}}$  77.21) and those of H-23 with C-9 ( $\delta_{\text{C}}$  113.80) and C-24, established the attachment of the chromene ring at C-9 with an ether linkage at C-8. The remaining HMBC correlations and NOEDIFF results were similar to those found in **GP18**. Thus, **GP12** was determined as 1,3,7-trihydroxy-2-(3,7-dimethylocta-2,6-dienyl)-24,24-dimethyl-2H-pyran[9,8-*b*]dibenzo[*b,e*][1,4]dioxepin-11-one, a new depsidone derivative.

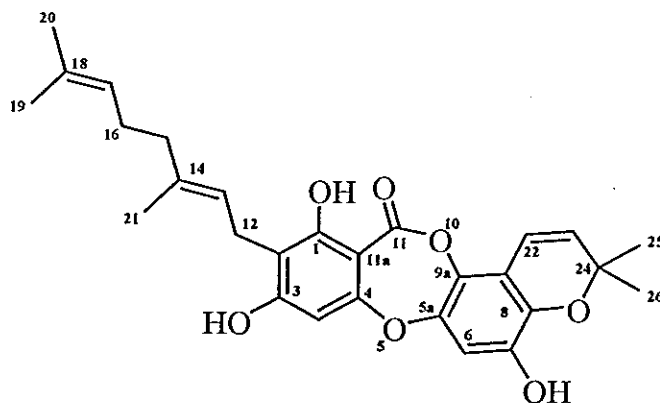


Table 51 The 300 MHz NMR data of compound **GP12** in  $\text{CDCl}_3$

Position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$ (C-type)	HMBC correlations	NOE
1-OH	11.36 ( <i>s</i> )	162.41 (C)	C-1, C-2, C-11a	
2		110.76 (C)		
3-OH	6.26 ( <i>brs</i> )	162.60 (C)		
4	6.29 ( <i>s</i> )	100.52 (CH)	C-1, C-2, C-3, C-4a, C-11a	H-6
4a		160.08 (C)		
5a		142.24 (C)		



Table 51 (Continued)

Position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$ (C-type)	HMBC correlations	NOE
6	6.69 ( <i>s</i> )	106.43 (CH)	C-5a, C-7, C-8, C-9a, C-24	H-4
7-OH	5.39 ( <i>brs</i> )	143.10 (C)		
8		136.45 (C)		
9		113.80 (C)		
9a		132.58 (C)		
11		168.50 (C)		
11a		98.53 (C)		
12	3.41 ( <i>d</i> , 7.2)	21.95 (CH <sub>2</sub> )	C-1, C-2, C-3, C-13, C-14	H-13, H-21
13	5.23 ( <i>mt</i> , 7.2)	120.67 (CH)		
14		140.25 (C)		
15	2.08 ( <i>m</i> )	39.67 (CH <sub>2</sub> )	C-14, C-16	
16	2.08 ( <i>m</i> )	26.25 (CH <sub>2</sub> )	C-14, C-15	
17	5.04 ( <i>mt</i> , 6.6)	123.58 (CH)		H-15, H-20
18		132.25 (C)		
19	1.59 ( <i>s</i> )	17.70 (CH <sub>3</sub> )	C-17, C-18, C-20	
20	1.68 ( <i>s</i> )	25.65 (CH <sub>3</sub> )	C-17, C-18, C-19	
21	1.80 ( <i>s</i> )	16.23 (CH <sub>3</sub> )	C-13, C-14, C-15	
22	6.76 ( <i>d</i> , 10.2)	116.22 (CH)	C-8, C-9a, C-24	H-23
23	5.75 ( <i>d</i> , 10.2)	132.06 (CH)	C-9, C-24	H-22, H-25, H-26
24		77.21 (C)		
25	1.46 ( <i>s</i> )	27.70 (CH <sub>3</sub> )	C-23, C-24, C-26	
26	1.46 ( <i>s</i> )	27.70 (CH <sub>3</sub> )	C-23, C-24, C-25	

### 3.4 Xanthone derivatives

#### 3.4.1 Compound GP9

Compound GP9 with the molecular formula  $C_{28}H_{32}O_6$  by EIMS ( $m/z$  464) (Figure 91) was isolated as a yellow gum. It exhibited UV (Figure 92) absorption bands of a xanthone chromophore at 256, 286 and 329 nm while the hydroxyl and conjugated carbonyl absorption bands were found at 3346 and 1641  $cm^{-1}$ , respectively, in the IR spectrum (Figure 93). The  $^1H$  NMR spectrum (Figure 94) (Table 52) contained signals of one chelated hydroxyl group ( $\delta_H$  13.32, *s*), one aromatic proton ( $\delta_H$  7.58, *s*), three prenyl units: unit 1 [ $\delta_H$  5.28 (*mt*,  $J = 7.2$  Hz, 1H), 3.46 (*d*,  $J = 7.2$  Hz, 2H), 1.85 (*s*, 3H) and 1.78 (*d*,  $J = 1.2$  Hz, 3H)], unit 2 [ $\delta_H$  5.25 (*mt*,  $J = 6.6$  Hz, 1H), 3.52 (*d*,  $J = 6.6$  Hz, 2H), 1.85 (*s*, 3H) and 1.73 (*s*, 3H)] and unit 3 [ $\delta_H$  5.34 (*mt*,  $J = 7.5$  Hz, 1H), 3.41 (*d*,  $J = 7.5$  Hz, 2H), 1.77 (*s*, 3H) and 1.75 (*s*, 3H)] and three hydroxyl groups [ $\delta_H$  6.43 (*s*), 6.15 (*brs*) and 5.67 (*brs*)]. The  $^{13}C$  NMR (Figure 95) (Table 52), DEPT (Figure 96) and HMQC (Figure 97) data indicated that GP9 consisted of 15 quaternary, 4 methine, 3 methylene and 6 methyl carbons. The location of all substituents was established by HMBC data (Figure 98) as follows. The chelated hydroxyl group was placed at C-1 ( $\delta_C$  158.62), a peri-position of the xanthone carbonyl group, and gave a  $^2J$  cross peak with C-1 and  $^3J$  ones with C-2 ( $\delta_C$  108.94) and C-9a ( $\delta_C$  102.91).  $^3J$  HMBC correlations between the methylene protons [H<sub>2</sub>-11, ( $\delta_H$  3.46)] of the prenyl unit 1 and C-1 and C-3 ( $\delta_C$  160.34) and those between the methylene protons [H<sub>2</sub>-16, ( $\delta_H$  3.52)] of the prenyl unit 2 and C-3 and C-4a ( $\delta_C$  152.56) established the attachment of the prenyl units I and II at C-2 and C-4 ( $\delta_C$  105.22), respectively. The singlet aromatic proton at  $\delta_H$  7.58 was attributed to H-8 on the basis of the chemical-shift value and  $^3J$  HMBC correlations of H-8/C-6 ( $\delta_C$  147.69), C-9 ( $\delta_C$  180.68) and C-10a ( $\delta_C$  143.67). The prenyl unit 3 was linked at C-7 due to HMBC correlations between the methylene protons [H<sub>2</sub>-21 ( $\delta_H$  3.41)] with C-6, C-7 ( $\delta_C$  125.27) and C-8 ( $\delta_C$  117.11). According to the chemical-shift values of C-3 and C-6, these carbons carried hydroxyl substituents. Furthermore, HMBC correlations of the hydroxy proton ( $\delta_H$  6.43)/C-2, C-3 and C-4 and those of the

hydroxy proton ( $\delta_H$  6.15)/C-5, C-6 and C-7 established the linkage of these hydroxyl groups at C-3 and C-6, respectively. Thus, the remaining hydroxy proton at  $\delta$  5.67 belonged to the C-5 hydroxyl group. Signal enhancement in the NOEDIFF experiment of H<sub>2</sub>-11 and H-17 upon irradiation of 3-OH (Figure 100) and that of H<sub>2</sub>-21 after irradiation of H-8 (Figure 99) supported the assigned location of all prenyl substituents. Therefore, GP9 was determined as 1,3,5,6-tetrahydroxy-2,4,7-tri(3-methylbutyl-2-enyl)xanthone, a new tetraoxygenated xanthone.

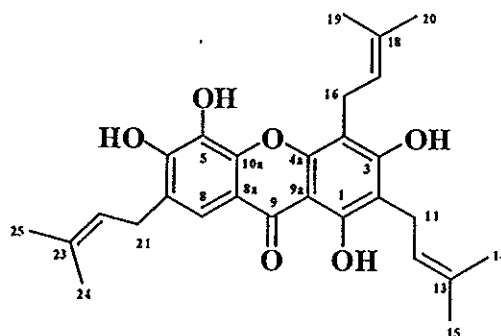


Table 52 The 300 MHz NMR data of compound GP9 in CDCl<sub>3</sub>

Position	$\delta_H$ ( <i>mult.</i> , $J_{Hz}$ )	$\delta_c$ (C-type)	HMBC correlations	NOE	
1-OH	13.32 ( <i>s</i> )	158.62(C)	C-1, C-2, C-9a	H-11, H-12, H-17	
2		108.94 (C)			
3-OH	6.43 ( <i>s</i> )	160.34 (C)	C-2, C-3, C-4		
4		105.22 (C)			
4a		152.56 (C)			
5-OH	5.67 ( <i>brs</i> )	130.08 (C)			
6-OH	6.15 ( <i>brs</i> )	147.69 (C)	C-5, C-6, C-7		
7		125.27 (C)			
8	7.58 ( <i>s</i> )	117.11 (CH)	C-6, C-9, C-10a, C-21		H-21, H-22
8a		113.38 (C)			
9		180.68 (C=O)			
9a		102.91 (C)			

Table 52 (Continued)

Position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$ (C-type)	HMBC correlations	NOE
10a		143.67 (C)		
11	3.46 ( <i>d</i> , 7.2)	21.63 (CH <sub>2</sub> )	C-1, C-2, C-3, C-12, C-13	1-OH, 3-OH, H-14
12	5.28 ( <i>mt</i> , 7.2)	121.40 (CH)		3-OH, H-11, H-15
13		135.83 (C)		
14	1.85 ( <i>s</i> )	17.95 (CH <sub>3</sub> )	C-12, C-13, C-15	
15	1.78 ( <i>d</i> , 1.2)	25.84 (CH <sub>3</sub> )	C-12, C-13, C-14	
16	3.52 ( <i>d</i> , 6.6)	22.02 (CH <sub>2</sub> )	C-3, C-4, C-4a, C-17, C-18	3-OH, H-17, H-19
17	5.25 ( <i>mt</i> , 6.6)	122.33 (CH)		
18		133.50 (C)		
19	1.85 ( <i>s</i> )	17.95 (CH <sub>3</sub> )	C-17, C-18, C-20	
20	1.73 ( <i>s</i> )	25.70 (CH <sub>3</sub> )	C-17, C-18	
21	3.41 ( <i>d</i> , 7.5)	28.47 (CH <sub>2</sub> )	C-6, C-7, C-8, C-22, C-23	H-8, H-22, H-24
22	5.34 ( <i>mt</i> , 7.5)	121.09 (CH)	C-21	H-8, H-21, H-25
23		134.39 (C)		
24	1.75 ( <i>s</i> )	17.86 (CH <sub>3</sub> )	C-22, C-23, C-25	
25	1.77 ( <i>s</i> )	25.87 (CH <sub>3</sub> )	C-22, C-23, C-24	

### 3.4.2 Compound GP8

Compound GP8 with the molecular formula C<sub>24</sub>H<sub>26</sub>O<sub>6</sub> by EIMS ( $m/z$  410) (Figure 101) was isolated as a yellow gum. The UV (Figure 102) and IR (Figure 103) absorption bands were similar to those of GP9, indicating that GP8 had a xanthone chromophore. The <sup>1</sup>H NMR spectrum (Figure 104) (Table 53) contained signals of one chelated hydroxyl group ( $\delta_{\text{H}}$  13.12, *s*), two hydroxyl groups [ $\delta_{\text{H}}$  6.12 (*s*)

and 5.61 (*s*)], two aromatic protons [ $\delta_H$  7.60 (*s*) and 6.37 (*s*)], two prenyl units: unit 1 [ $\delta_H$  5.22 (*brs*, 1H), 3.49 (*d*,  $J = 5.7$  Hz, 2H), 1.85 (*s*, 3H) and 1.71 (*s*, 3H)] and unit 2 [ $\delta_H$  5.35 (*mt*,  $J = 7.5$  Hz, 1H), 3.42 (*d*,  $J = 7.5$  Hz, 2H), 1.76 (*s*, 3H) and 1.77 (*s*, 3H)] and one methoxyl group ( $\delta_H$  3.91, *s*). Its  $^1\text{H}$  NMR data were similar to those of GP9 except that one of the prenyl signals in GP9 was replaced by signal of a singlet aromatic proton at  $\delta_H$  6.37 in GP8. This proton was attributed to H-2 due to HMBC correlations of H-2/C-3 ( $\delta_C$  163.62), C-4 ( $\delta_C$  107.31), and C-9a ( $\delta_C$  102.99) (Figure 108). The additional methoxyl group was located at C-3 on the basis of a HMBC correlation of 3-OCH<sub>3</sub>/C-3. The enhancement observed between H-2 and the methoxy protons in the NOEDIFF experiment (Figure 109) confirmed the assignment. The remaining HMBC correlations and NOEDIFF results were similar to those found in GP9. Thus, GP8 as determined as 1,5,6-trihydroxy-3-methoxy-4,7-di(3-methylbutyl-2-enyl)xanthone, a new tetraoxygenated xanthone.

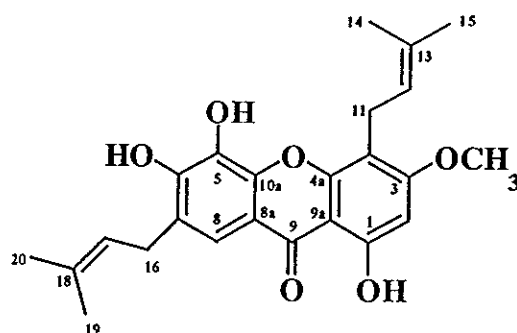


Table 53 The 300 MHz NMR data of compound GP8 in CDCl<sub>3</sub>

Position	$\delta_H$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_C$ (C-type)	HMBC correlations	NOE
1-OH	13.12 ( <i>s</i> )	162.17(C)	C-1, C-2, C-9a	
2	6.37 ( <i>s</i> )	94.36 (CH)	C-1, C-3, C-4, C-9a	3-OCH <sub>3</sub>
3		163.62 (C)		
3-OCH <sub>3</sub>	3.91 ( <i>s</i> )	56.07 (CH <sub>3</sub> )	C-3	
4		107.31 (C)		
4a		153.77 (C)		
5-OH	5.61 ( <i>s</i> )	130.14 (C)		

Table 53 (Continued)

Position	$\delta_H$ ( <i>mult.</i> , $J_{Hz}$ )	$\delta_C$ (C-type)	HMBC correlations	NOE
6-OH	6.12 ( <i>s</i> )	147.75 (C)		
7		125.36 (C)		
8	7.60 ( <i>s</i> )	117.10 (CH)	C-6, C-9, C-10a, C-16	H-16, H-17
8a		113.34 (C)		
9		180.69 (C=O)		
9a		102.99 (C)		
10a		143.76 (C)		
11	3.49 ( <i>d</i> , 5.7)	21.72 (CH <sub>2</sub> )	C-3, C-4, C-4a, C-12, C-13	H-12, H-14
12	5.22 ( <i>brs</i> )	122.89 (CH)		
13		131.67 (C)		
14	1.85 ( <i>s</i> )	17.86 (CH <sub>3</sub> )	C-12, C-13, C-15	H-11
15	1.71 ( <i>s</i> )	25.63 (CH <sub>3</sub> )	C-12, C-13, C-14	
16	3.42 ( <i>d</i> , 7.5)	28.39 (CH <sub>2</sub> )	C-6, C-7, C-8, C-17, C-18	H-8, H-17, H-19
17	5.35 ( <i>mt</i> , 7.5)	121.08 (CH)	C-19, C-20	H-8, H-16, H-20
18		134.34 (C)		
19	1.76 ( <i>s</i> )	17.86 (CH <sub>3</sub> )	C-17, C-18, C-20	
20	1.77 ( <i>s</i> )	25.83 (CH <sub>3</sub> )	C-17, C-18, C-19	

### 3.4.3 Compound GP20

Compound GP20 was isolated as a yellow gum. It exhibited UV (Figure 110) absorption bands of a xanthone chromophore at 236, 253, 272, 313 and 365 nm while the hydroxyl and conjugated carbonyl absorption bands were found at 3231 and 1655  $\text{cm}^{-1}$ , respectively, in the IR spectrum (Figure 111). The  $^1\text{H}$  NMR spectrum (Figure 112) (Table 54) contained signals of two singlet aromatic protons ( $\delta_H$  7.44 and 6.82 and two *meta*-coupled aromatic protons [ $\delta_H$  6.28,  $J = 2.4$  Hz and 6.15,  $J = 2.4$  Hz].

The  $^{13}\text{C}$  NMR (Figure 113) (Table 54) and HMQC (Figure 114) data indicated that compound GP20 consisted of 13 carbons: 9 quarternary and 4 methine carbons. The aromatic proton at  $\delta_{\text{H}}$  7.44 was attributed to H-8 on the basis of the chemical-shift value and HMBC correlations (Figure 115) of H-8/C-6 ( $\delta_{\text{C}}$  153.77), C-7 ( $\delta_{\text{C}}$  143.38), C-9 ( $\delta_{\text{C}}$  179.73) and C-10a ( $\delta_{\text{C}}$  151.81). The aromatic proton at  $\delta_{\text{H}}$  6.82 gave  $^2J$  cross peaks with C-6 and C-10a and  $^3J$  ones with C-7 and C-8a ( $\delta_{\text{C}}$  112.47), indicating its attachment at C-5 ( $\delta_{\text{C}}$  102.10). Two *meta*-coupled aromatic protons ( $\delta_{\text{H}}$  6.15 and 6.28) were assigned as H-2 and H-4, respectively, according to HMBC cross peaks of H-2/C-1 ( $\delta_{\text{C}}$  162.93), C-3 ( $\delta_{\text{C}}$  164.84), C-4 ( $\delta_{\text{C}}$  93.30) and C-9a ( $\delta_{\text{C}}$  101.94) and those of H-4/C-2 ( $\delta_{\text{C}}$  97.36), C-3, C-4a ( $\delta_{\text{C}}$  157.99), C-9 ( $\delta_{\text{C}}$  179.73) and C-9a. The chemical-shift values of C-3, C-6 and C-7 suggested the substituents to be hydroxyl groups. Thus, GP20 was determined as 1,3,6,7-tetrahydroxyxanthone (norathyriol), isolated from *Hypericum sampsonii* (Don, 2004).

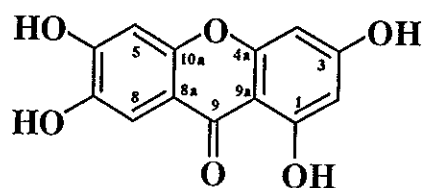


Table 54 The 300 MHz NMR data of compound GP20 in  $\text{CD}_3\text{OD}$

Position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$ (C-type)	HMBC correlations
1-OH		162.93 (C)	
2	6.15 ( <i>d</i> , 2.4)	97.36 (CH)	C-1, C-3, C-4, C-9a
3-OH		164.84 (C)	
4	6.28 ( <i>d</i> , 2.4)	93.30 (CH)	C-2, C-3, C-4a, C-9, C-9a
4a		157.99 (C)	
5	6.82 ( <i>s</i> )	102.10 (CH)	C-6, C-7, C-8a, C-9, C-10a
6-OH		153.77 (C)*	
7-OH		143.38 (C)	
8	7.44 ( <i>s</i> )	107.80 (CH)	C-6, C-7, C-9, C-10a

Table 54 (Continued)

Position	$\delta_H$ ( <i>mult.</i> , $J_{Hz}$ )	$\delta_C$ (C-type)	HMBC correlations
8a		112.47 (C)	
9		179.73 (C=O)	
9a		101.94 (C)	
10a		151.81 (C)*	

\* interchangeable

### 3.4.4 Compound GP19

Compound GP19 was isolated as a yellow gum. The UV (Figure 116) and IR (Figure 117) absorption bands were similar to those of GP20. The  $^1H$  NMR spectrum (Figure 118) (Table 55) contained signals of two *ortho*-coupled aromatic protons [ $\delta_H$  7.56,  $J = 8.7$  Hz and 6.87,  $J = 8.7$  Hz] and two *meta*-coupled aromatic protons [ $\delta_H$  6.42,  $J = 2.1$  Hz and 6.16,  $J = 2.1$  Hz]. The  $^1H$  NMR data and HMBC correlations (Figure 120) of ring B were similar to those of GP20. The *ortho*-coupled aromatic protons at  $\delta_H$  7.56 and  $\delta_H$  6.87 were attributed to H-8 and H-7, respectively, on the basis of the chemical-shift value of H-8 and HMBC correlations of H-8/C-6 ( $\delta_C$  152.30), C-9 ( $\delta_C$  180.20) and C-10a ( $\delta_C$  146.30) and those of H-7/C-6 and C-8a ( $\delta_C$  112.10). Thus, GP19 was determined as 1,3,5,6-tetrahydroxyxanthone, isolated from *Hypericum scabrum* (Tanaka, 2004).

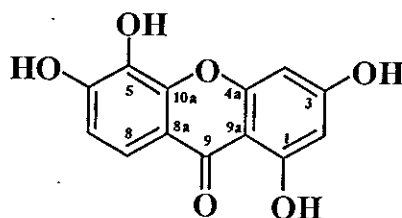




Table 55 The 300 MHz NMR data of compound GP19 in CD<sub>3</sub>OD

Position	$\delta_H$ ( <i>mult.</i> , $J_{Hz}$ )	$\delta_C$ (C-type)	HMBC correlations
1-OH		163.30 (C)	
2	6.16 ( <i>d</i> , 2.1)	97.80 (CH)	C-1, C-3, C-4, C-9a
3-OH		166.00 (C)	
4	6.42 ( <i>d</i> , 2.1)	93.20 (CH)	C-2, C-3, C-4a, C-9a
4a		158.00 (C)	
5-OH		132.80 (C)	
6-OH		152.30 (C)	
7	6.87 ( <i>d</i> , 8.7)	112.80 (CH)	C-5, C-6, C-8a, C-10a
8	7.56 ( <i>d</i> , 8.7)	115.40 (CH)	C-6, C-9, C-10a
8a		112.10 (C)	
9		180.20 (C=O)	
9a		101.40 (C)	
10a		146.30 (C)	

### 3.4.5 Compound GP17

Compound GP17 was isolated as a yellow gum. The UV (Figure 121) and IR (Figure 122) absorption bands were similar to those of GP20. The <sup>1</sup>H NMR spectrum (Figure 123) (Table 56) contained signals of one chelated hydroxyl group ( $\delta_H$  13.63, *s*), three aromatic protons [ $\delta_H$  6.77, (*s*), 6.26 (*d*,  $J = 2.1$  Hz) and 6.19 (*d*,  $J = 2.1$  Hz)], one prenyl unit [ $\delta_H$  5.27 (*mt*,  $J = 6.0$  Hz, 1H), 4.09 (*d*,  $J = 6.0$  Hz, 2H), 1.84 (*s*, 3H) and 1.69 (*s*, 3H)] and one methoxyl group ( $\delta_H$  3.79, *s*). The <sup>1</sup>H NMR data were similar to those of GP20 except for the replacement of the H-8 signal in GP20 with the prenyl signal in GP17. The appearance of the methylene protons of the prenyl group at lower field together with the HMBC correlations (Figure 126) of H<sub>2</sub>-11/C-7 ( $\delta_C$  143.10), C-8 ( $\delta_C$  137.30) and C-8a ( $\delta_C$  112.00) supported above conclusion. The additional methoxyl group was located at C-7 on the basis of a HMBC correlation of 7-OCH<sub>3</sub>/C-7. The remaining HMBC correlations were almost identical to those found

in GP20. Thus, GP17 was determined as 1,3,6-trihydroxy-7-methoxy-8-(3-methylbutyl-2-enyl)xanthone (dulxanthone D) isolate from *Garcinia mangostana* (Nilar, 2005).

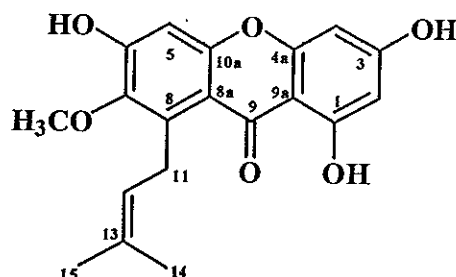


Table 56 The 300 MHz NMR data of compound GP17 in CDCl<sub>3</sub> + CD<sub>3</sub>OD

Position	$\delta_H$ (mult., J <sub>Hz</sub> )	$\delta_C$ (C-type)	HMBC correlations
1-OH	13.63 ( <i>brs</i> )	164.00 (C)	
2	6.19 ( <i>d</i> , 2.1)	97.93 (CH)	C-1, C-3, C-4, C-9a
3-OH		164.70 (C)	
4	6.26 ( <i>d</i> , 2.1)	92.60 (CH)	C-2, C-3, C-4a, C-9a
4a		156.10 (C)	
5	6.77 ( <i>s</i> )	101.87 (CH)	C-6, C-7, C-8a, C-9, C-10a
6-OH		155.63 (C)	
7		143.10 (C)	
7-OCH <sub>3</sub>	3.79 ( <i>s</i> )	61.34 (CH <sub>3</sub> )	C-7
8		137.30 (C)	
8a		112.00 (C)	
9		181.93 (C=O)	
9a		101.87 (C)	
10a		155.63 (C)	
11	4.09 ( <i>d</i> , 6.0)	26.34 (CH <sub>2</sub> )	C-7, C-8, C-8a, C-12, C-13
12	5.27 ( <i>mt</i> , 6.0)	123.30 (CH)	
13		131.83 (C)	
14	1.84 ( <i>s</i> )	18.10 (CH <sub>3</sub> )	C-12, C-13, C-15
15	1.69 ( <i>s</i> )	25.75 (CH <sub>3</sub> )	C-12, C-13, C-14

### 3.4.6 Compound GP16

Compound GP16 was isolated as a yellow gum. The UV (Figure 127) absorption bands at 239, 253, 312 and 351 nm and IR absorption bands at 3366 and 1646  $\text{cm}^{-1}$  (Figure 128) indicated that GP16 had a xanthone chromophore. The  $^1\text{H}$  NMR spectrum (Figure 129) (Table 57) contained signals of one chelated hydroxyl group ( $\delta_{\text{H}}$  13.67, *s*), three aromatic protons [ $\delta_{\text{H}}$  6.76 (*s*), 6.25 (*d*,  $J = 1.8$  Hz) and 6.18 (*d*,  $J = 1.8$  Hz)], one geranyl unit [ $\delta_{\text{H}}$  5.25 (*brt*,  $J = 6.6$  Hz, 1H), 5.03 (*brt*,  $J = 6.6$  Hz, 1H), 4.09 (*d*,  $J = 6.6$  Hz, 2H), 2.07 (*m*, 2H), 2.00 (*m*, 2H), 1.82 (*s*, 3H), 1.60 (*s*, 3H) and 1.54 (*s*, 3H)] and one methoxyl group ( $\delta_{\text{H}}$  3.78, *s*). The  $^1\text{H}$  NMR data were similar to those of GP17 except for the replacement of the prenyl signal in GP17 with one geranyl signal in GP16. The chemical-shift values of methylene protons (H<sub>2</sub>-11) and their HMBC correlations (Figure 132) with C-7 ( $\delta_{\text{C}}$  143.17), C-8 ( $\delta_{\text{C}}$  137.41) and C-8a ( $\delta_{\text{C}}$  111.67) established the attachment of the geranyl group at C-8. Signal enhancement of H<sub>2</sub>-11 and H-12 in the NOEDIFF experiment (Figure 133) upon irradiation of the methoxy protons confirmed above assignment. Thus, GP16 was determined as rübraxanthone, previously isolated from latex of *Garcinia parvifolia* (Patthalung, 1988).

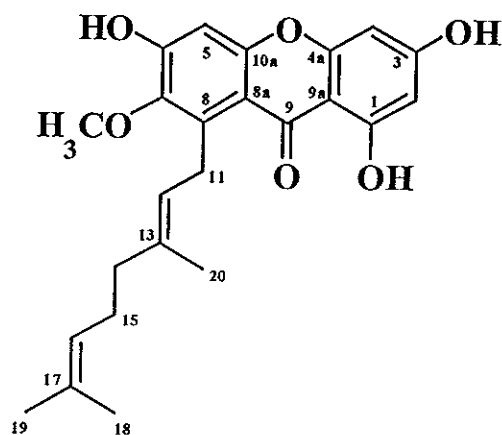


Table 57 The 300 MHz NMR data of compound GP16 in CDCl<sub>3</sub> + CD<sub>3</sub>OD

Position	$\delta_H$ ( <i>mult.</i> , J <sub>Hz</sub> )	$\delta_C$ (C-type)	HMBC correlations	NOE
1-OH	13.67 ( <i>brs</i> )	162.20 (C)	C-1, C-2, C-9a	
2	6.18 ( <i>d</i> , 1.8)	97.93 (CH)	C-1, C-3, C-4, C-9a	
3-OH		157.03 (C)		
4	6.25 ( <i>d</i> , 1.8)	93.43 (CH)	C-2, C-3, C-4a, C-9a	
4a		157.03 (C)		
5	6.76 ( <i>s</i> )	101.85 (CH)	C-6, C-7, C-8a, C-9, C-10a	
6-OH		142.70 (C)		
7		143.17 (C)		
7-OCH <sub>3</sub>	3.78 ( <i>s</i> )	61.28 (CH <sub>3</sub> )	C-7	H-11, H-12, H-20
8		137.41 (C)		
8a		111.67 (C)		
9		181.85 (C=O)		
9a		103.21 (C)		
10a		155.61 (C)		
11	4.09 ( <i>d</i> , 6.6)	26.22 (CH <sub>2</sub> )	C-7, C-8, C-8a, C-12, C-13	H-12, H-20
12	5.25 ( <i>brt</i> , 6.6)	123.37 (CH)	C-8, C-11, C-14, C-20	H-11, 7-OCH <sub>3</sub>
13		135.24 (C)		
14	2.00 ( <i>m</i> )	39.69 (CH <sub>2</sub> )	C-12, C-13, C-15, C-16, C-20	
15	2.07 ( <i>m</i> )	26.54 (CH <sub>2</sub> )	C-13, C-14, C-16, C-17	
16	5.03 ( <i>brt</i> , 6.6)	124.31 (CH)	C-15, C-18, C-19	
17		131.10 (C)		
18	1.54 ( <i>s</i> )	17.53 (CH <sub>3</sub> )	C-16, C-17, C-19	H-14, H-19
19	1.60 ( <i>s</i> )	25.49 (CH <sub>3</sub> )	C-16, C-17, C-18	
20	1.82 ( <i>s</i> )	16.34 (CH <sub>3</sub> )	C-12, C-13, C-14	H-11, 7-OCH <sub>3</sub>

### 3.4.7 Compound GP11

Compound GP11 with the molecular formula  $C_{29}H_{34}O_6$  by EIMS ( $m/z$  478) (Figure 134) was isolated as a yellow gum. The UV (Figure 135) and IR (Figure 136) absorption bands were similar to those of GP16, indicating that GP11 had a xanthone chromophore. The  $^1H$  NMR spectrum (Figure 137) (Table 58) contained signals of one chelated hydroxyl group ( $\delta_H$  13.35, *s*), two singlet aromatic protons ( $\delta_H$  6.88 and 6.25), two hydroxyl groups [ $\delta_H$  6.33 (*brs*) and 5.97 (*brs*)], one prenyl unit [ $\delta_H$  5.27 (*mt*,  $J = 6.0$  Hz, 1H), 4.10 (*d*,  $J = 6.0$  Hz, 2H), 1.83 (*d*,  $J = 1.2$  Hz, 3H) and 1.70 (*d*,  $J = 1.2$  Hz, 3H)], one geranyl unit [ $\delta_H$  5.28 (*mt*,  $J = 6.6$  Hz, 1H), 5.03 (*mt*,  $J = 6.6$  Hz, 1H), 3.53 (*d*,  $J = 6.6$  Hz, 2H), 2.09 (*m*, 4H), 1.89 and (*d*,  $J = 0.9$  Hz, 3H), 1.66 (*d*,  $J = 0.9$  Hz, 3H) and 1.60 (*d*,  $J = 0.9$  Hz, 3H)] and one methoxyl group ( $\delta_H$  3.82, *s*). The  $^1H$  NMR data were similar to those of GP17 except for the replacement of one aromatic proton signal ( $\delta_H$  6.26) in GP17 with the geranyl signal in GP11. The geranyl group was located at C-4 ( $\delta_C$  104.08) on the basis of HMBC correlations (Figure 141) between methylene protons, H<sub>2</sub>-11, with C-3 ( $\delta_C$  138.76), C-4 and C-4a ( $\delta_C$  154.00). The remaining HMBC correlations were almost identical to those found in GP17. Thus, GP11 was determined as 1,3,6-trihydroxy-7-methoxy-8-(3-methylbutyl-2-enyl)-4-(3,7-dimethylocta-2,6-dienyl)xanthone, a new tetraoxygenated xanthone.

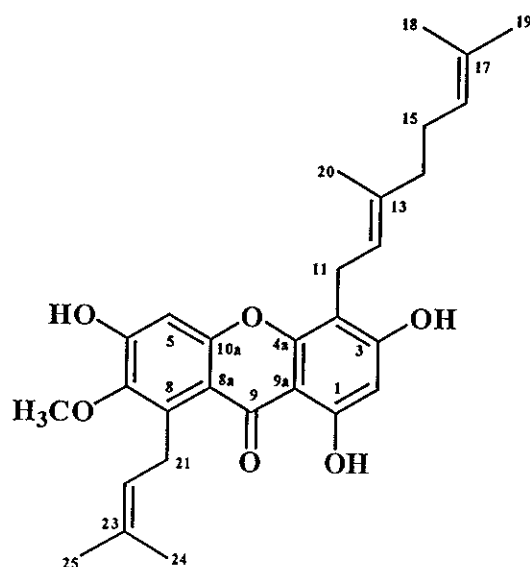


Table 58 The 300 MHz NMR data of compound GP11 in CDCl<sub>3</sub>

Position	$\delta_H$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_c$ (C-type)	HMBC correlations	NOE
1-OH	13.35 ( <i>s</i> )	161.68 (C)	C-1, C-2, C-9a	
2	6.25 ( <i>s</i> )	98.53 (CH)	C-1, C-9a	
3-OH	5.97 ( <i>brs</i> )	138.76 (C)	C-3	
4		104.08 (C)		
4a		154.00 (C)		
5	6.88 ( <i>s</i> )	101.56 (CH)	C-6, C-7, C-10a	
6-OH	6.33 ( <i>brs</i> )	155.81 (C)		
7		142.67 (C)		
7-OCH <sub>3</sub>	3.82 ( <i>s</i> )	62.12 (CH <sub>3</sub> )	C-7	H-21, H-24
8		137.06 (C)		
8a		112.20 (C)		
9		182.39 (C=O)		
9a		104.08 (C)		
10a		154.58 (C)		
11	3.53 ( <i>d</i> , 6.6)	21.62 (CH <sub>2</sub> )	C-3, C-4, C-4a, C-12 C-13	H-12, H-20
12	5.28 ( <i>mt</i> , 6.6)	121.26 (CH)	C-14	H-11, H-14
13		138.76 (C)		
14	2.09 ( <i>m</i> )	39.70 (CH <sub>2</sub> )	C-15, C-20	H-12
15	2.09 ( <i>m</i> )	26.38 (CH <sub>2</sub> )	C-14, C-20	H-16
16	5.03 ( <i>mt</i> , 6.6)	123.72 (CH)		H-19
17		124.27 (C)		
18	1.60 ( <i>d</i> , 0.9)	17.97 (CH <sub>3</sub> )	C-16, C-17	
19	1.66 ( <i>d</i> , 0.9)	25.65 (CH <sub>3</sub> )	C-16, C-17, C-18	
20	1.89 ( <i>d</i> , 0.9)	18.24 (CH <sub>3</sub> )	C-12, C-13, C-14	H-11, H-14
21	4.10 ( <i>d</i> , 6.0)	26.55 (CH <sub>2</sub> )	C-7, C-8, C-8a, C-22, C-23	7-OCH <sub>3</sub> , H-22, H-24

Table 58 (Continued)

Position	$\delta_H$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_C$ (C-type)	HMBC correlations	NOE
22	5.27 ( <i>mt</i> , 6.0)	123.08 (CH)	C-8, C-25	
23		135.69 (C)		
24	1.83 ( <i>d</i> , 1.2)	18.24 (CH <sub>3</sub> )	C-22, C-23, C-25	H-21, H-25
25	1.70 ( <i>d</i> , 1.2)	25.83 (CH <sub>3</sub> )	C-22, C-23, C-24	H-22, H-24

### 3.4.8 Compound GP15

Compound GP15 was isolated as a yellow gum. It exhibited UV (Figure 142) absorption bands of a xanthone chromophore at 243, 273 and 317 nm while the hydroxyl and conjugated carbonyl absorption bands were found at 3400 and 1651  $\text{cm}^{-1}$ , respectively, in the IR spectrum (Figure 143). The  $^1\text{H}$  NMR spectrum (Figure 144) (Table 59) contained signals of one chelated hydroxyl group ( $\delta_H$  13.05, *s*), one singlet aromatic proton ( $\delta_H$  6.46), three aromatic protons of 1,2,3-trisubstituted benzene [ $\delta_H$  7.72 (*dd*,  $J = 7.5$  and 1.8 Hz), 7.23 (*dd*,  $J = 7.5$  and 1.8 Hz), 7.16, (*t*,  $J = 7.5$  Hz)] and one geranyl unit [ $\delta_H$  5.29 (*mt*,  $J = 6.6$  Hz, 1H), 5.08 (*mt*,  $J = 6.6$  Hz, 1H), 3.40 (*d*,  $J = 6.6$  Hz, 2H), 2.07 (*m*, 2H), 1.99 (*m*, 2H), 1.82 (*s*, 3H), 1.65 (*s*, 3H) and 1.58 (*s*, 3H)]. The HMBC correlations (Figure 147) between the chelated hydroxy proton and C-2 ( $\delta_C$  110.80) together with those between the methylene protons (H<sub>2</sub>-11,  $\delta_H$  3.40) and C-1 ( $\delta_C$  160.40), C-2 and C-3 ( $\delta_C$  162.80) established the attachment of the geranyl group at C-2. The singlet aromatic proton ( $\delta_H$  6.46) was then attribute to H-4 according to its HMBC cross peaks with C-2, C-3, C-4a ( $\delta_C$  155.00), C-9 ( $\delta_C$  180.80) and C-9a ( $\delta_C$  102.80). The remaining aromatic protons of the 1,2,3-trisubstituted benzene at  $\delta_H$  7.23, 7.16 and 7.72 were assigned as H-6, H-7 and H-8, respectively, on the basis of the chemical-shift value of H-8, their multiplicities and HMBC correlations. The substituents at C-3 and C-5 were hydroxyl groups according to chemical-shift values of C-3 and C-5 (Figure 145) (Table 59). Thus, GP15 was

determined as mangostinone, previously isolated from green fruit of *Garcinia mangostana* (Suksamran, 2002).

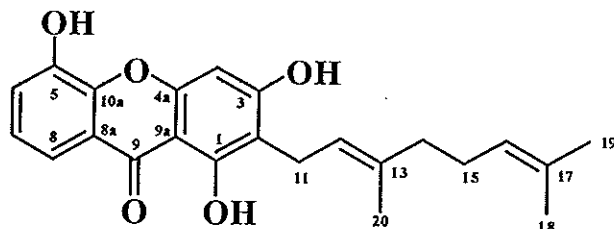


Table 59 The 300 MHz NMR data of compound GP15 in  $CDCl_3$

Position	$\delta_H$ (mult., $J_{Hz}$ )	$\delta_C$ (C-type)	HMBC correlations	NOE
1-OH	13.05 (s)	160.40 (C)	C-1, C-2, C-9a	
2		110.80 (C)		
3-OH		162.80 (C)		
4	6.46 (s)	93.38 (CH)	C-2, C-3, C-4a, C-9, C-9a	
4a		155.00 (C)		
5		145.30 (C)		
6	7.23 (dd, 7.5, 1.8)	119.89 (CH)	C-8, C-10a	
7	7.16 (t, 7.5)	123.49 (CH)	C-5, C-8, C-8a	
8	7.72 (dd, 7.5, 1.8)	116.09 (CH)	C-6, C-9, C-10a	
8a		121.40 (C)		
9		180.80 (C)		
9a		102.80 (C)		
10a		145.00 (C)		
11	3.40 (d, 6.6)	21.24 (CH <sub>2</sub> )	C-1, C-2, C-3, C-12, C-13	H-12, H-20
12	5.29 (mt, 6.6)	121.80 (CH)	C-2, C-11, C-14, C-20	H-11, H-14
13		135.95 (C)		
14	1.99 (m)	39.73 (CH <sub>2</sub> )	C-12, C-13, C-15, C-20	



Table 59 (Continued)

Position	$\delta_H$ ( <i>mult.</i> , $J_{Hz}$ )	$\delta_C$ (C-type)	HMBC correlations	NOE
15	2.07 ( <i>m</i> )	26.62 (CH <sub>2</sub> )	C-13, C-14, C-16, C-17	H-14, H-18
16	5.08 ( <i>mt</i> , 6.6)	124.30 (CH)	C-14, C-15, C-18, C-19	
17		131.28 (C)		
18	1.65 ( <i>s</i> )	25.51 (CH <sub>3</sub> )	C-16, C-17, C-19	H-15
19	1.58 ( <i>s</i> )	17.52 (CH <sub>3</sub> )	C-16, C-17, C-18	H-16
20	1.82 ( <i>s</i> )	16.05 (CH <sub>3</sub> )	C-12, C-13, C-14	H-11, H-14

### 3.5 Benzocyclooctene derivative

#### 3.5.1 Compound GP10

Compound GP10 with the molecular formula C<sub>15</sub>H<sub>16</sub>O<sub>5</sub> from EIMS ( $m/z$  276) (Figure 148) was isolated as a colorless gum. It exhibited UV (Figure 149) absorption bands at 211, 270 and 294 nm while a hydroxyl absorption band was found at 3471 cm<sup>-1</sup> in the IR spectrum (Figure 150). The <sup>13</sup>C NMR (Figure 152) (Table 60) and DEPT (Figure 153) data revealed the presence of 7 quaternary, 5 methine and 3 methyl carbons. These results together with the molecular formula established a benzocyclooctene skeleton. The <sup>1</sup>H NMR spectrum (Figure 151) (Table 60) contained signals of three protons of 1,2,4-trisubstituted benzene [ $\delta_H$  7.04 (*dd*,  $J = 8.0$  and 2.1 Hz, 1H), 7.00 (*d*,  $J = 2.1$  Hz, 1H) and 6.97 (*d*,  $J = 8.0$  Hz, 1H)], two identical protons [ $\delta_H$  6.72 (*s*, 2H)] and three methoxyl groups [ $\delta_H$  3.96 (*s*, 3H) and 3.95 (*s*, 6H)]. The protons of the 1,2,4-trisubstituted benzene at  $\delta_H$  7.00, 7.04 and 6.97 were assigned as H-7, H-9 and H-10, respectively, on the basis of HMBC correlations (Figure 155) and their multiplicities. The methoxyl group ( $\delta_H$  3.96) was linked at C-8 ( $\delta_C$  146.67) due to a HMBC correlation with C-8. Two identical protons of the eight-membered ring were attributed to H-1 and H-6 due to HMBC correlations of H-1/C-2 ( $\delta_C$  147.72), C-6a ( $\delta_C$  134.09) and C-10a ( $\delta_C$  133.11) and those of H-6/C-5 ( $\delta_C$

147.72), C-6a and C-10a. Furthermore, HMBC cross peaks of the methoxy protons ( $\delta_{\text{H}}$  3.95)/C-2 and C-5 established the linkage of the methoxyl groups at C-2 and C-5. According to the chemical-shift values of C-3 ( $\delta_{\text{C}}$  145.01) and C-4 ( $\delta_{\text{C}}$  134.09), these carbons carried hydroxyl substituents. Signal enhancement of H-7, H-10, 2-OCH<sub>3</sub> and 5-OCH<sub>3</sub> in the NOEDIFF experiment (Figure 156) upon irradiation of H-1 and H-6 supported above assignment. Thus, GP10 was determined as 2,5,8-trimethoxy-3,4-dihydroxybenzocyclooctene, a new benzocyclooctene derivative.

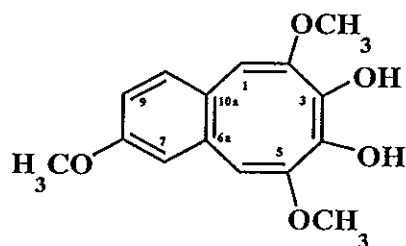


Table 60 The 300 MHz NMR data of compound GP10 in CDCl<sub>3</sub>

Position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$ (C-type)	HMBC correlations	NOE
1	6.72 ( <i>s</i> )	104.02 (CH)	C-2, C-6a, C-10a	2-OCH <sub>3</sub> , H-10
2		147.72 (C)		
2-OCH <sub>3</sub>	3.95 ( <i>s</i> )	56.44 (CH <sub>3</sub> )	C-2	
3-OH		134.16 (C) *		
4-OH		145.01 (C)		
5		147.72 (C)		
5-OCH <sub>3</sub>	3.95 ( <i>s</i> )	56.44 (CH <sub>3</sub> )	C-5	
6	6.72 ( <i>s</i> )	104.02 (CH)	C-5, C-6a, C-10a	5-OCH <sub>3</sub> , H-7
6a		134.09 (C)*		
7	7.00 ( <i>d</i> , 2.1)	109.69 (CH)	C-6a, C-8, C-9, C-10a	
8		146.67 (C)		
8-OCH <sub>3</sub>	3.96 ( <i>s</i> )	56.08 (CH <sub>3</sub> )	C-8	
9	7.04 ( <i>dd</i> , 8.0, 2.1)	120.03 (CH)	C-7, C-10a	
10	6.97 ( <i>d</i> , 8.0)	114.64 (CH)	C-6a, C-8, C-9, C-10a	
10a		133.11 (C)		

\* interchangeable

### 3.6 Cyclohexanone derivative

#### 3.6.1 Compound GP13

Compound **GP13** was isolated as a colorless gum. It exhibited UV (Figure 157) absorption bands at 205, 234 and 277 nm while the hydroxyl and conjugated carbonyl absorption bands were found at 3404 and 1655  $\text{cm}^{-1}$ , respectively, in the IR spectrum (Figure 158). The  $^1\text{H}$  NMR spectrum (Figure 159) (Table 61) contained signals of one olefinic proton  $\delta_{\text{H}}$  5.84 (*brq*,  $J = 1.2$  Hz, 1H), two nonequivalent methylene protons [ $\delta_{\text{H}}$  2.54 (*brd*,  $J = 13.5$  Hz, 1H) and 2.16 (*m*, 1H)], one oxymethine proton  $\delta_{\text{H}}$  4.15 (*brd*,  $J = 6.0$  Hz, 1H), one methine proton  $\delta_{\text{H}}$  2.14 (*m*, 1H), one vinylic methyl group [ $\delta_{\text{H}}$  2.04 (*brt*,  $J = 1.2$  Hz, 3H)] and one geranyl-geranyl unit [ $\delta_{\text{H}}$  5.20 (*mt*,  $J = 6.0$  Hz, 1H), 5.09 (*m*, 3H), 2.36 (*m*, 1H), 2.17 (*m*, 1H), 2.13 (*m*, 6H), 2.06 (*m*, 2H), 2.00 (*m*, 2H), 1.95 (*m*, 2H), 1.68 (*brd*,  $J = 0.9$  Hz, 3H), 1.64 (*brd*,  $J = 0.9$  Hz, 3H), 1.62 (*s*, 3H) and 1.59 (*s*, 6H)]. The  $^1\text{H}$ - $^1\text{H}$  COSY (Figure 162) and HMBC correlations (Figure 164) supported the presence of the geranyl-geranyl unit. HMBC correlations of the olefinic proton (H-2,  $\delta_{\text{H}}$  5.84)/C-4 ( $\delta_{\text{C}}$  73.71) and C-6 ( $\delta_{\text{C}}$  41.37) and those of the methine proton H-5 ( $\delta_{\text{H}}$  2.16-2.11)/C-1 ( $\delta_{\text{C}}$  196.45) and C-4 established a cyclohexenone moiety. The vinylic methyl was located at C-3 due to its  $^3J$  HMBC correlations with C-2 and C-4. The geranyl-geranyl unit was linked at C-5 on the basis of HMBC correlations of nonequivalent methylene protons, H<sub>2</sub>-8 ( $\delta_{\text{H}}$  2.36 and 2.17)/C-1, C-4, C-5 ( $\delta_{\text{C}}$  43.62) and C-6 ( $\delta_{\text{C}}$  41.37). The relative configuration of C-4 and C-5 was assigned as *cis* since irradiation of H-4 enhanced the signal intensity of H-5 in the NOEDIFF experiment (Figure 165). The coupling-constant value of 6.0 Hz between H-4 and H-5 supported this orientation. Therefore, **GP13** was determined as (2*E*,6*E*,10*E*)-(+)-4 $\beta$ -hydroxy-3-methyl-5 $\beta$ -(3,7,11,15-tetramethylhexadeca-2,6,10,14-tetraenyl)cyclohex-2-en-1-one, previously isolated from *Garcinia cowa* (Wahyuni, 2004). **GP13** had the same relative configuration as the previously isolated compound since they gave similar specific rotation [**GP13**:  $[\alpha]_{\text{D}} = +60.01$  ( $c = 0.01$ ,  $\text{CHCl}_3$ ) and the previously isolated compound:  $[\alpha]_{\text{D}} = +50$  ( $c = 0.01$ ,  $\text{CHCl}_3$ )].

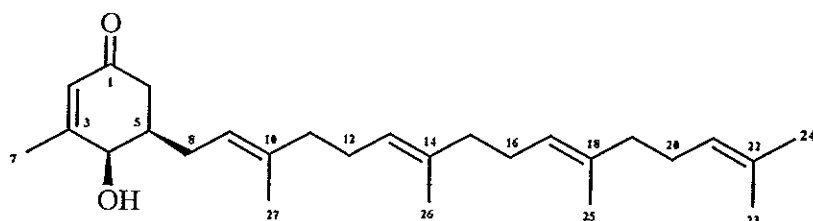


Table 61 The 300 MHz NMR data of compound GP13 in  $\text{CDCl}_3$

Position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$ (C-type)	HMBC correlations	NOE
1		196.45 (C=O)		
2	5.84 ( <i>brq</i> , 1.2)	126.85 (CH)	C-4, C-6, C-7	H-7
3		162.96 (C)		
4	4.15 ( <i>brd</i> , 6.0)	73.71 (CH)		H-5, H-7, H-8
5	2.14 ( <i>m</i> )	43.62 (CH)	C-1, C-4, C-8, C-9	H-4, H-6
6	2.54 ( <i>brd</i> , 13.5)	41.37 (CH <sub>2</sub> )	C-1, C-4, C-5, C-8	
	2.16 ( <i>m</i> )			
7	2.04 ( <i>brt</i> , 1.2)	20.32 (CH <sub>3</sub> )	C-2, C-3, C-4	
8	2.36 ( <i>m</i> )	31.01 (CH <sub>2</sub> )	C-1, C-4, C-5, C-6, C-9	H-5, H-9, H-27
	2.17 ( <i>m</i> )			
9	5.20 ( <i>mt</i> , 6.0)	120.73 (CH)	C-8, C-11, C-27	H-11
10		138.54 (C)		
11	2.06 ( <i>m</i> )	39.85 (CH <sub>2</sub> ) <sup>a</sup>	C-12, C-13	H-9, H-13
12	2.13 ( <i>m</i> )	26.47 (CH <sub>2</sub> ) <sup>b</sup>	C-10, C-11, C-13, C-14, C-26	
13	5.09 ( <i>m</i> )	123.81 (CH) <sup>c</sup>	C-12, C-15, C-26	
14		135.47 (C) <sup>d</sup>		
15	1.95( <i>m</i> )	39.73 (CH <sub>2</sub> ) <sup>a</sup>	C-13, C-14, C-16, C-26	H-13, H-17
16	2.13 ( <i>m</i> )	26.77 (CH <sub>2</sub> ) <sup>b</sup>	C-14, C-15, C-18	
17	5.09 ( <i>m</i> )	124.17 (CH) <sup>c</sup>	C-15, C-16, C-19, C-25	

Table 61 (Continued)

Position	$\delta_{\text{H}}$ ( <i>mult.</i> , $J_{\text{Hz}}$ )	$\delta_{\text{C}}$ (C-type)	HMBC correlations	NOE
18		134.98 (C) <sup>d</sup>		
19	2.00 ( <i>m</i> )	39.69 (CH <sub>2</sub> ) <sup>a</sup>	C-18, C-20, C-25	
20	2.13 ( <i>m</i> )	26.61 (CH <sub>2</sub> ) <sup>b</sup>	C-18, C-19, C-22	
21	5.09 ( <i>m</i> )	124.39 (CH) <sup>c</sup>	C-19, C-20, C-23,	H-24
22		131.29 (C)	C-24	
23	1.62 ( <i>s</i> )	17.69 (CH <sub>3</sub> )	C-21, C-22, C-24	
24	1.68 ( <i>brd</i> , 0.9)	25.70 (CH <sub>3</sub> )	C-21, C-22, C-23	
25	1.59 ( <i>s</i> )	16.01 (CH <sub>3</sub> ) <sup>e</sup>	C-17, C-18, C-19	
26	1.59 ( <i>s</i> )	16.07 (CH <sub>3</sub> ) <sup>e</sup>	C-13, C-14, C-15	
27	1.64 ( <i>brd</i> , 0.9)	16.29 (CH <sub>3</sub> )	C-9, C-10, C-11	

<sup>a,b,c,d,e</sup> Chemical shifts with the same index may be interchanged

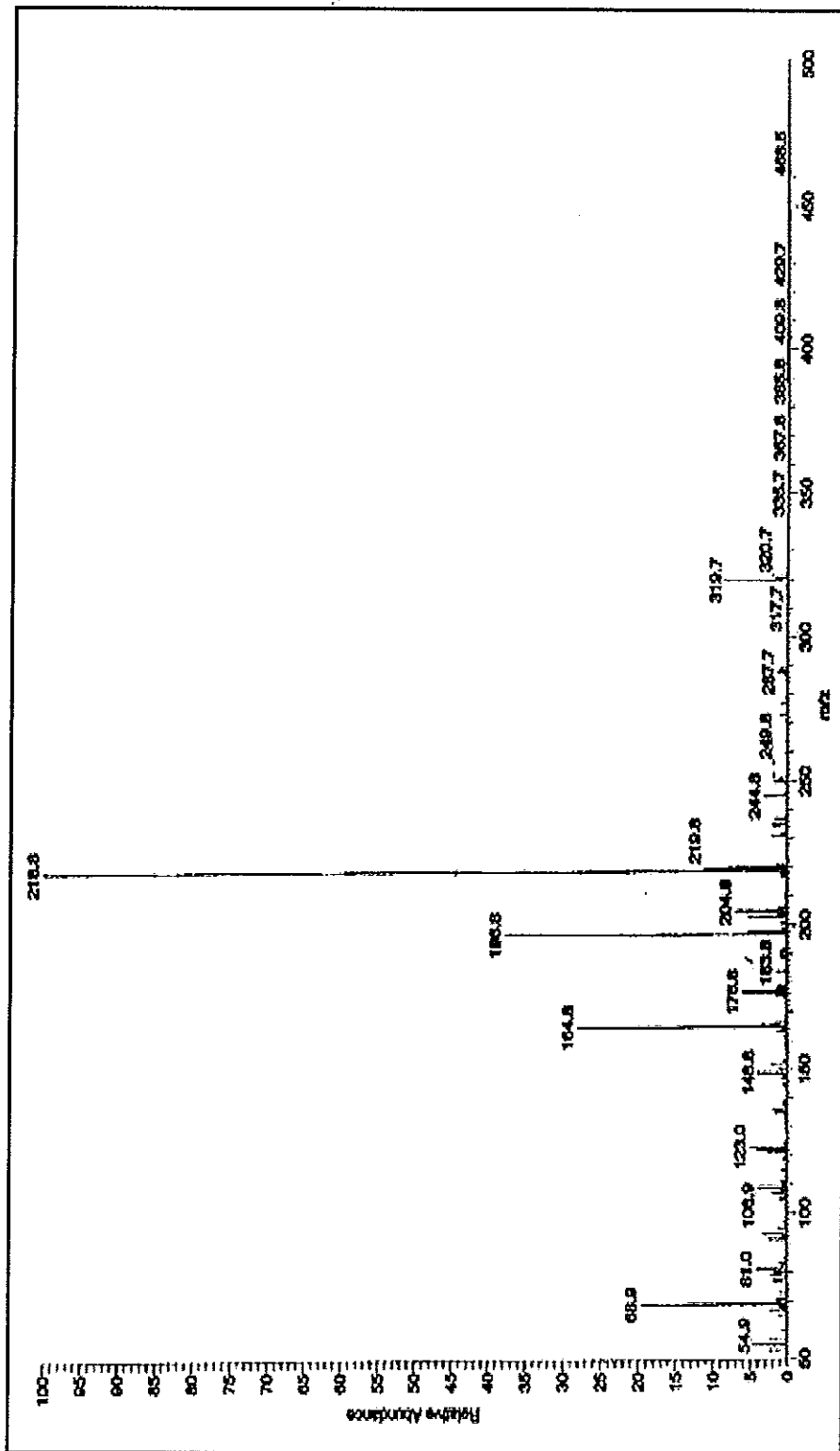


Figure 1 Mass spectrum of compound GP7

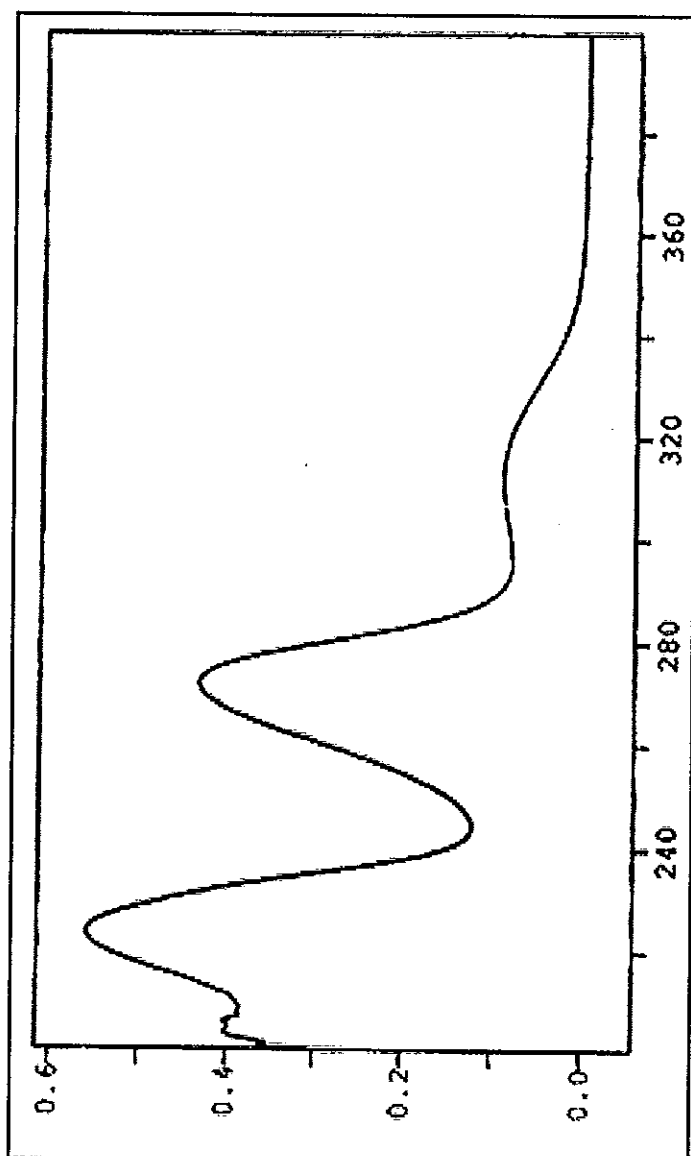


Figure 2 UV (MeOH) spectrum of compound GP7

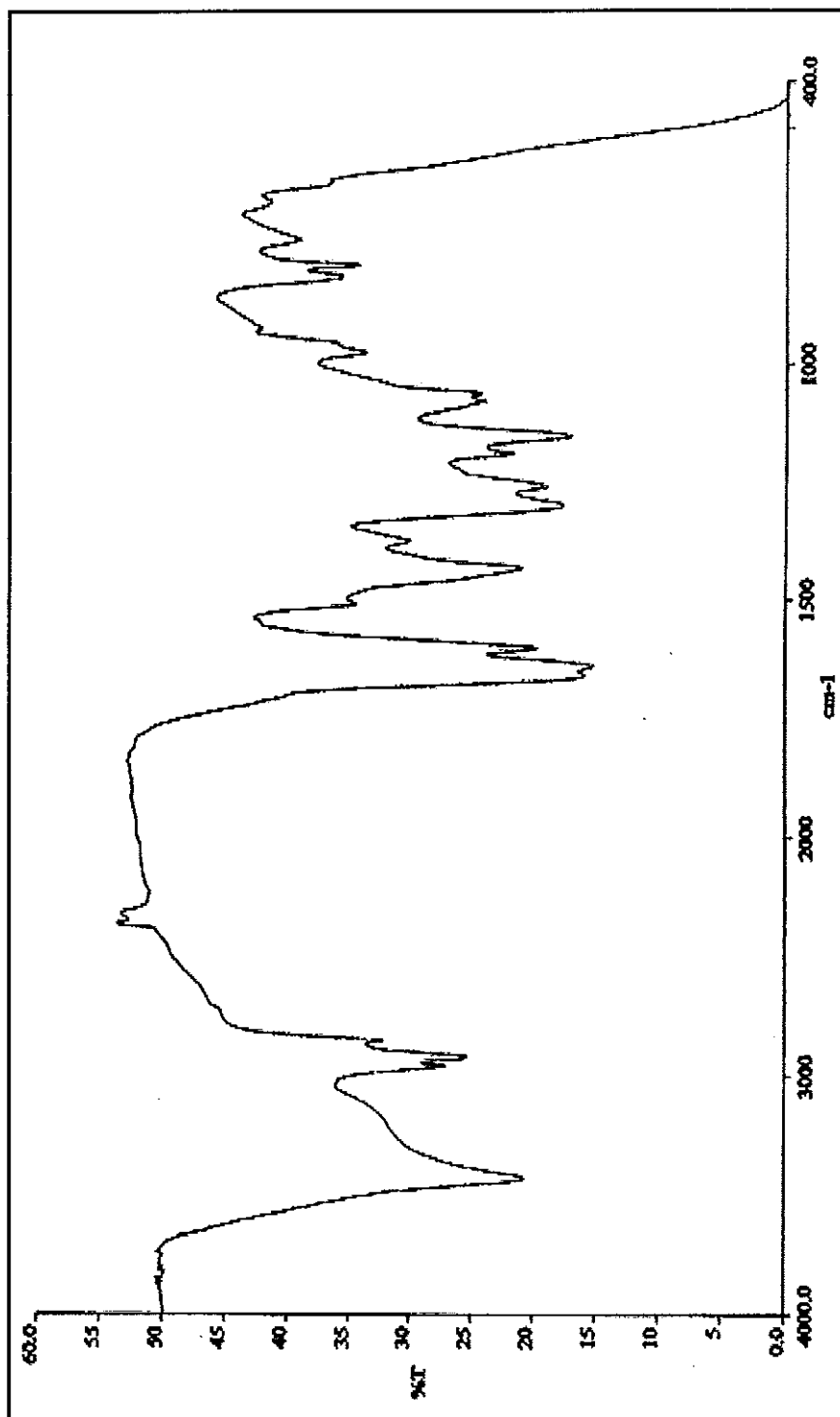


Figure 3 FT-IR (neat) spectrum of compound GP7



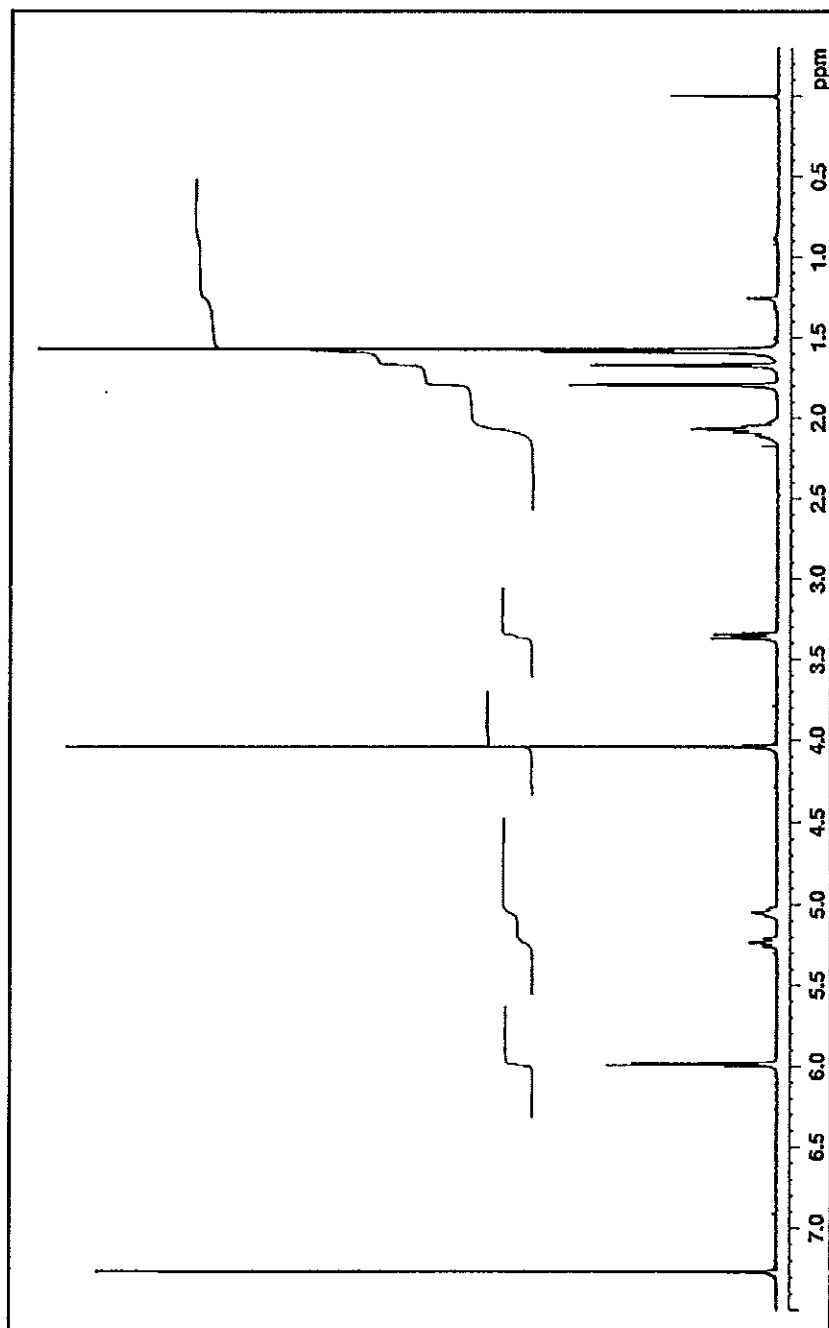


Figure 4  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound GP7

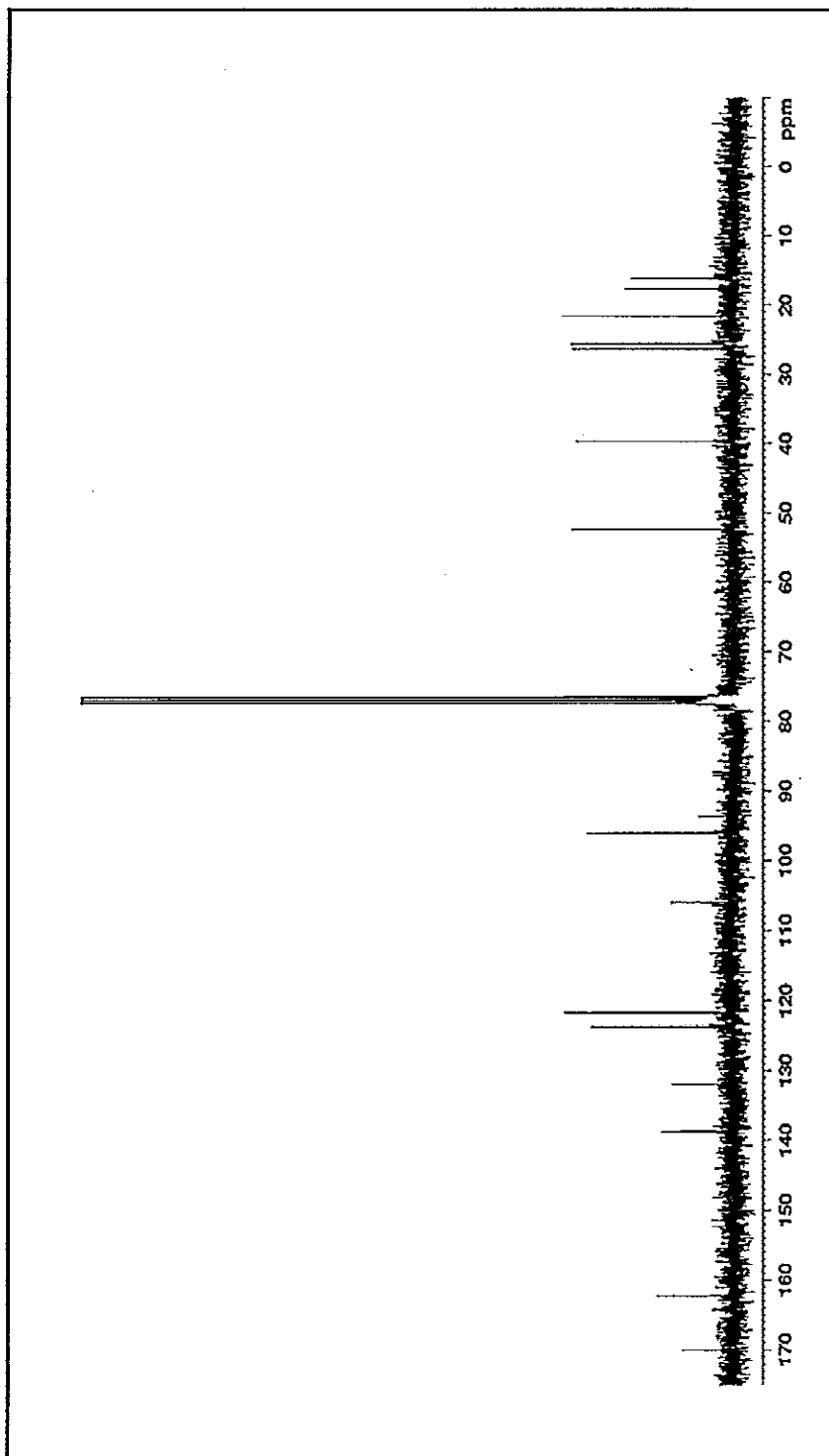


Figure 5  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound GP7

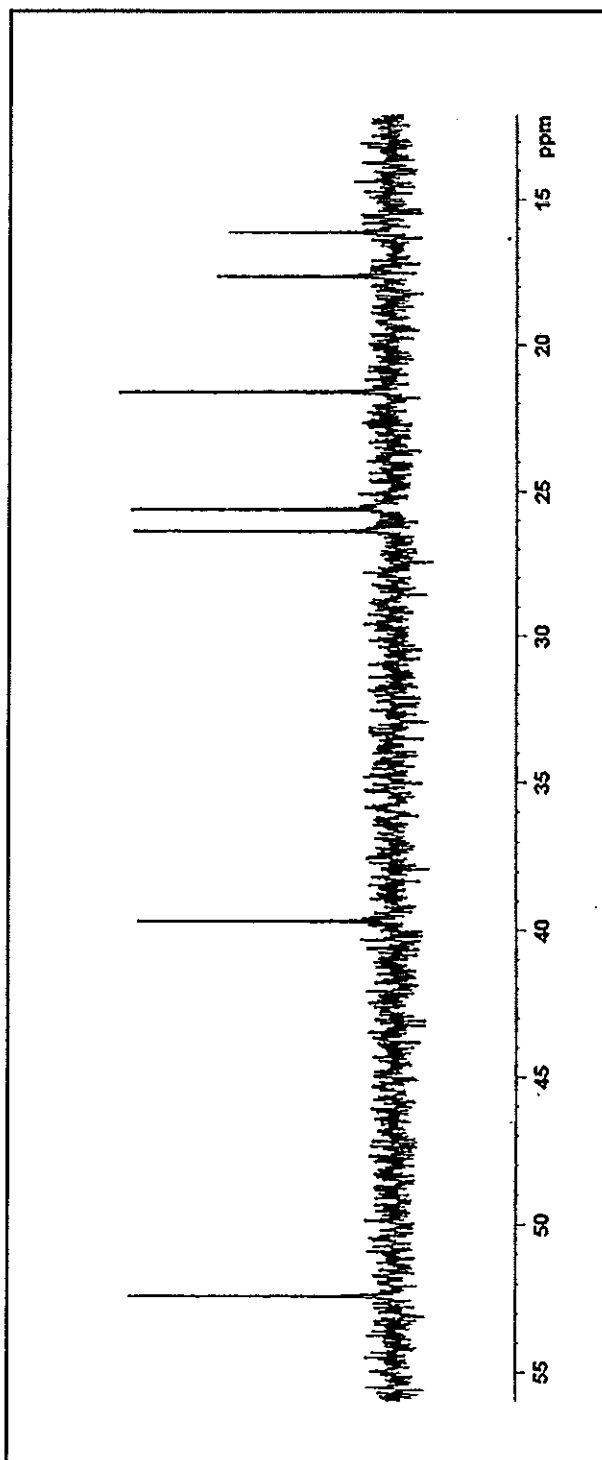


Figure 6 DEPT 90° spectra of compound GP7

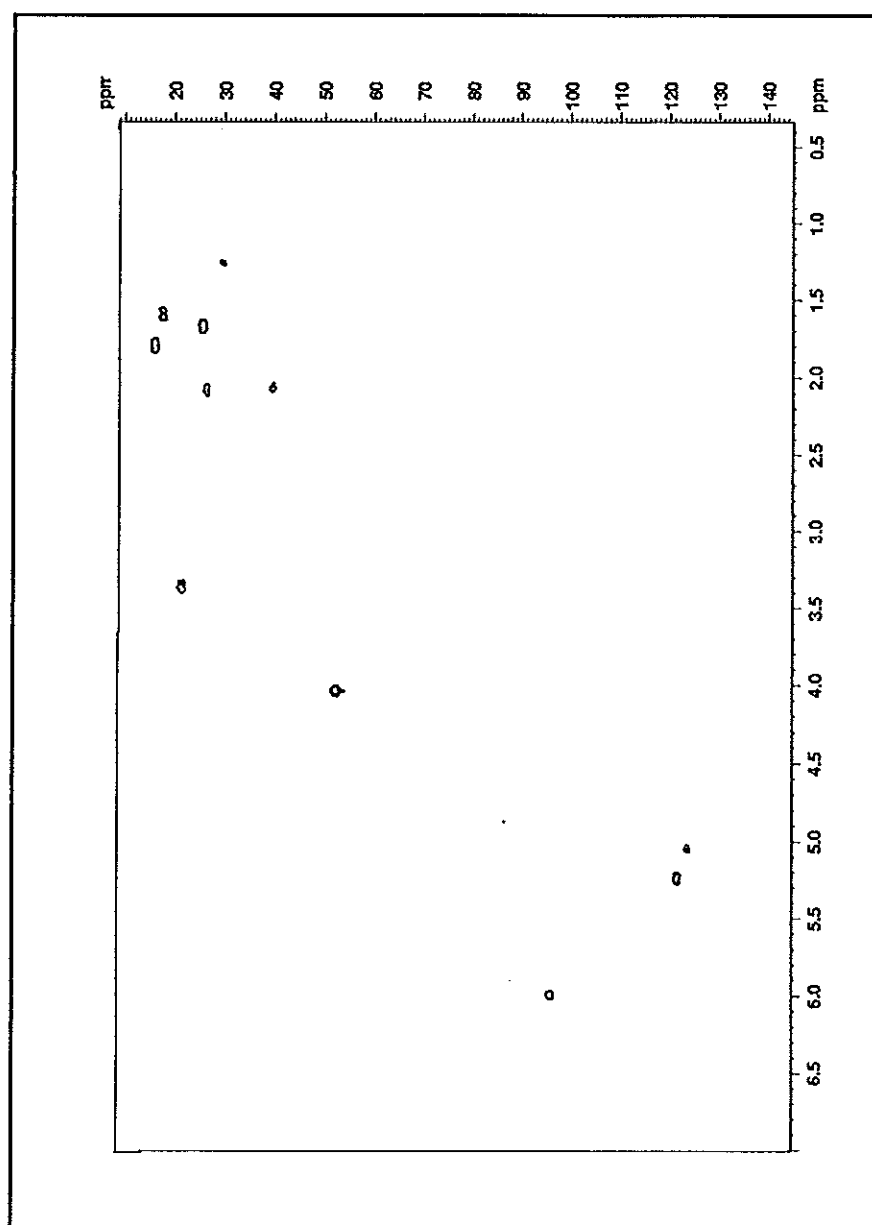


Figure 7 2D HMQC spectrum of compound GP7

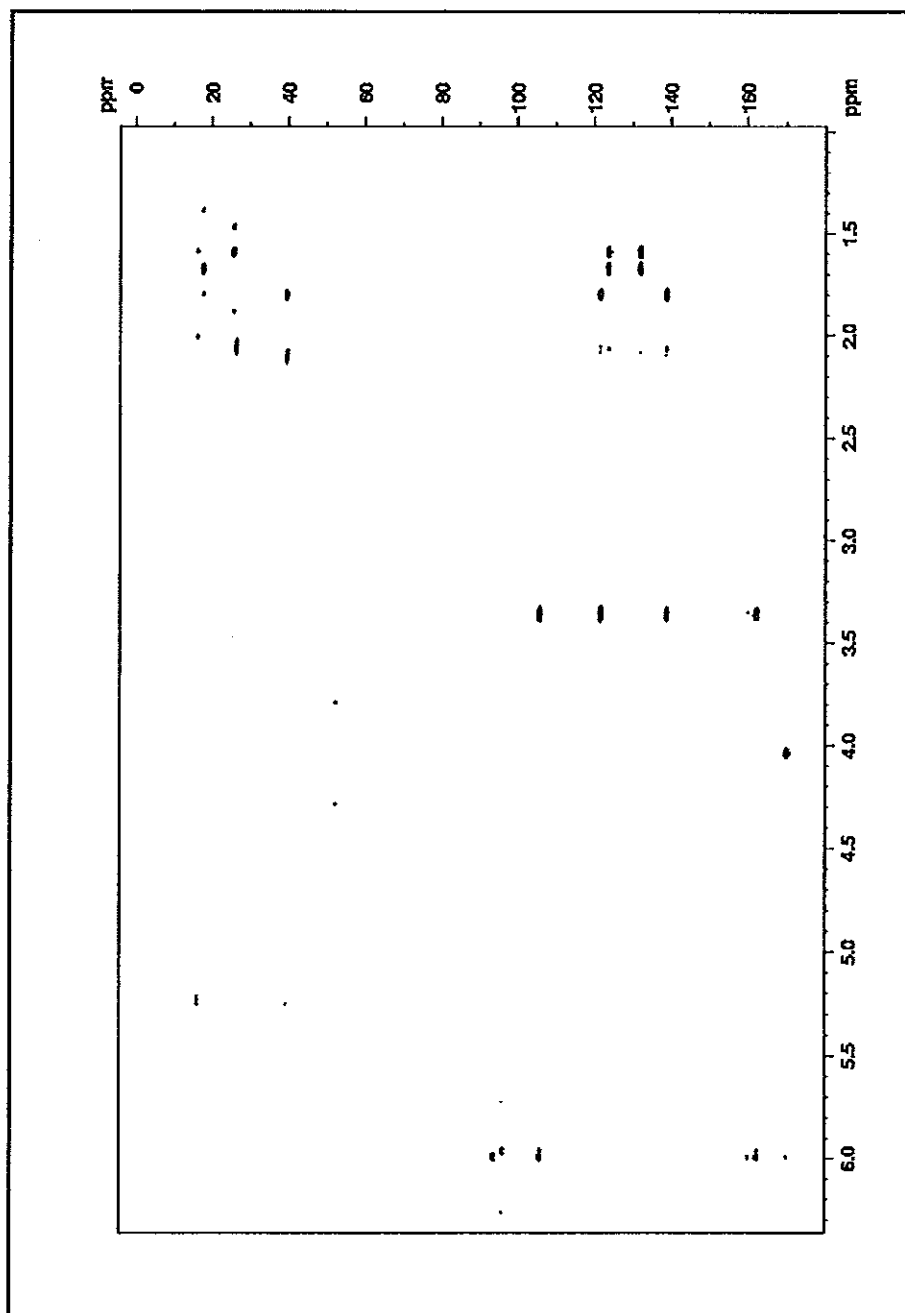
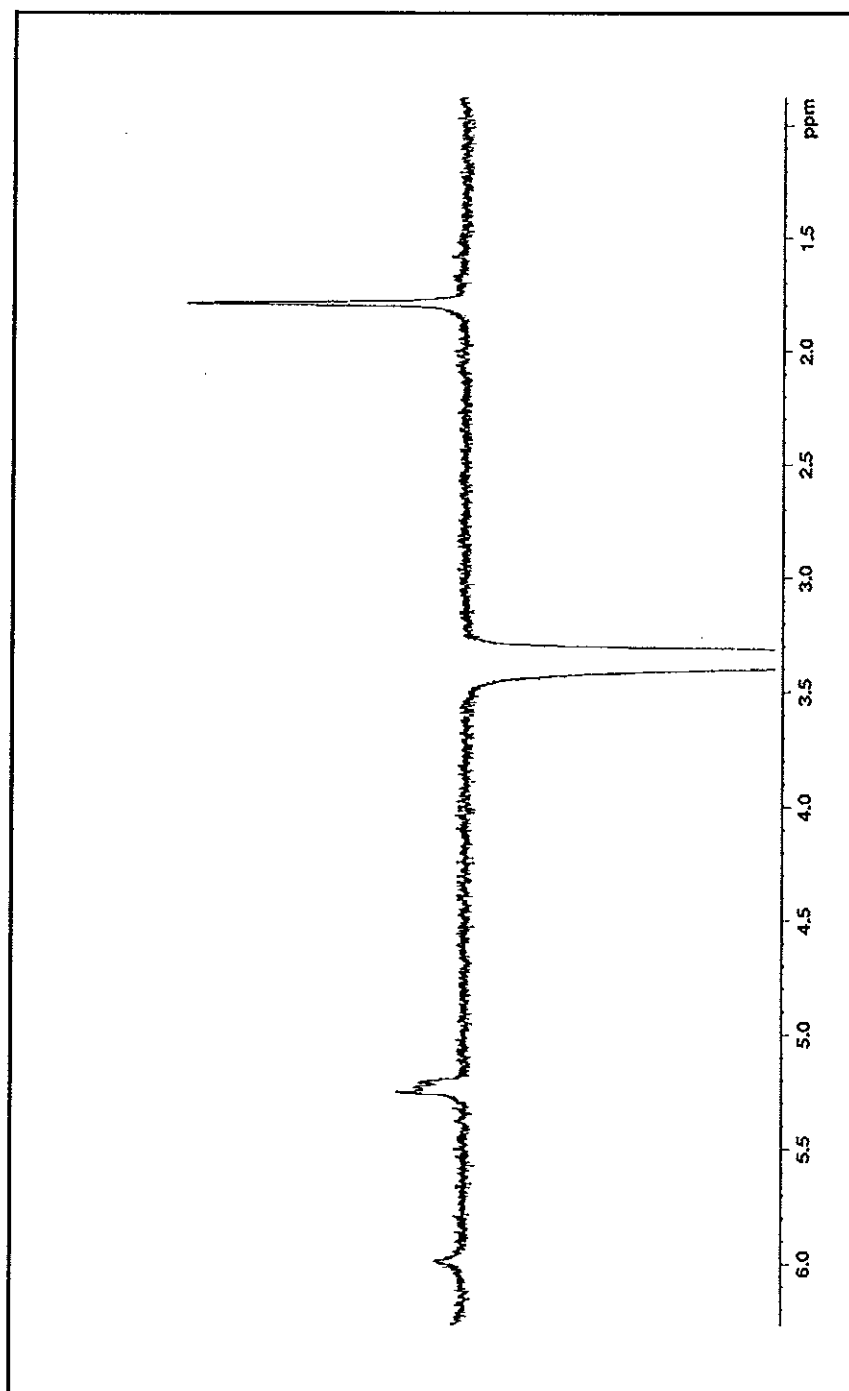


Figure 8 2D HMBC spectrum of compound GP7



**Figure 9** NOE difference spectrum of compound GP7 after irradiation at  $\delta_{\text{H}} 3.35$  ( $\text{H}_2\text{-7}$ )

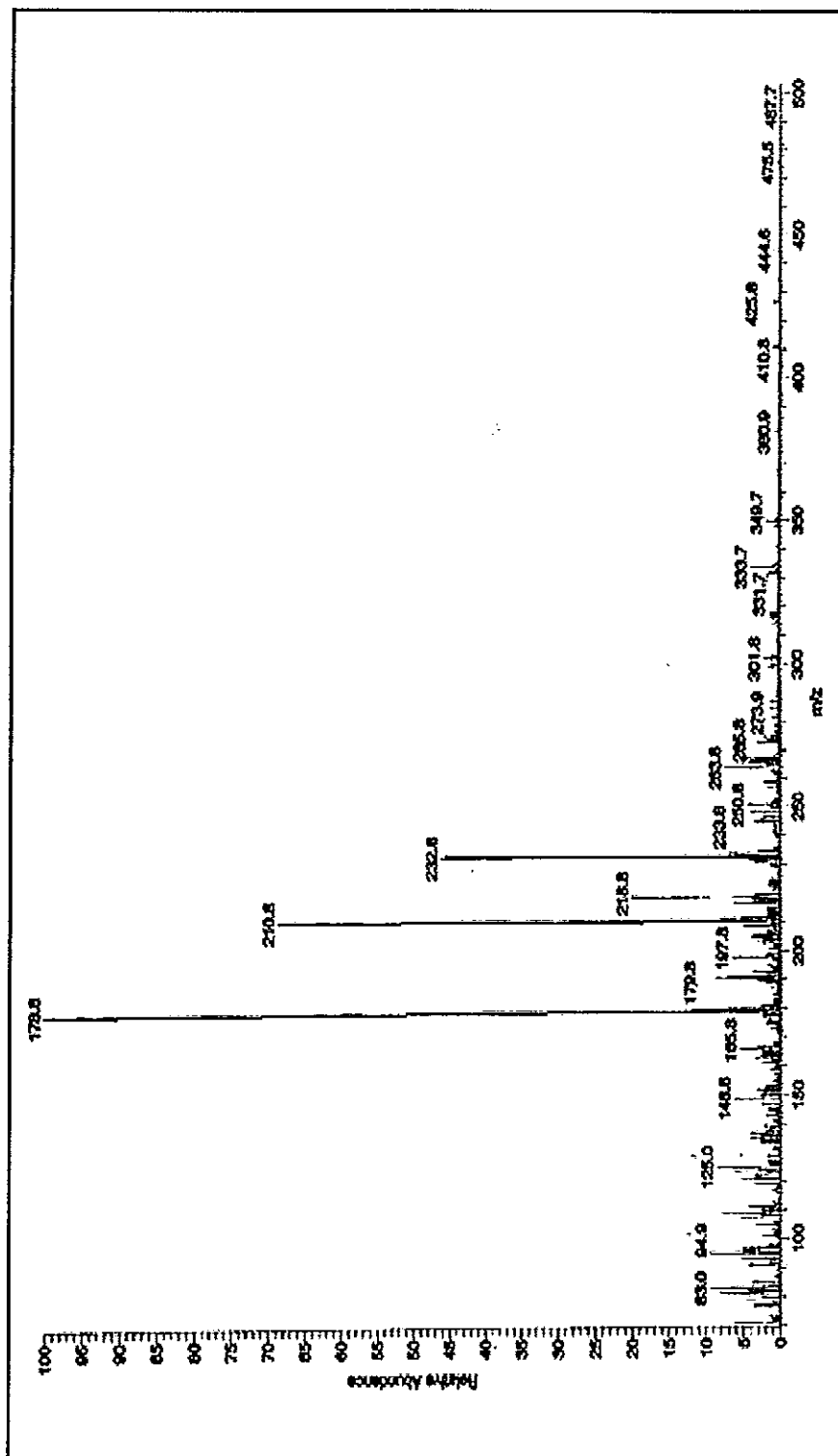


Figure 10 Mass spectrum of compound GP2

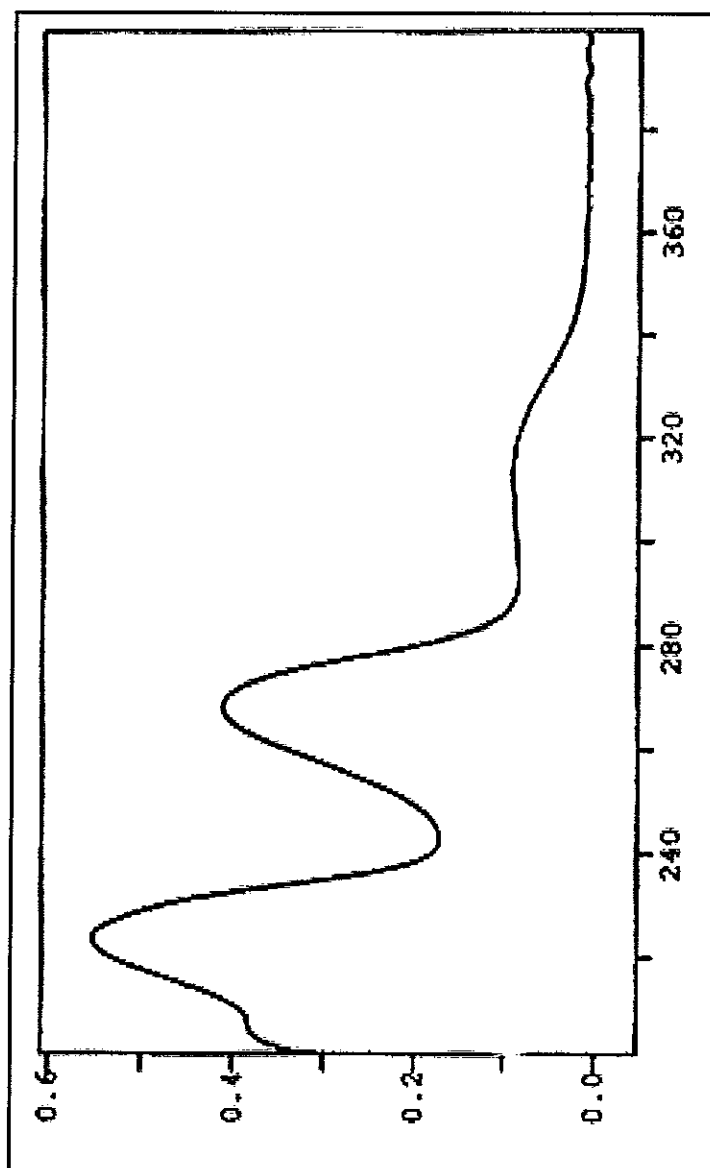


Figure 11 UV (MeOH) spectrum of compound GP2



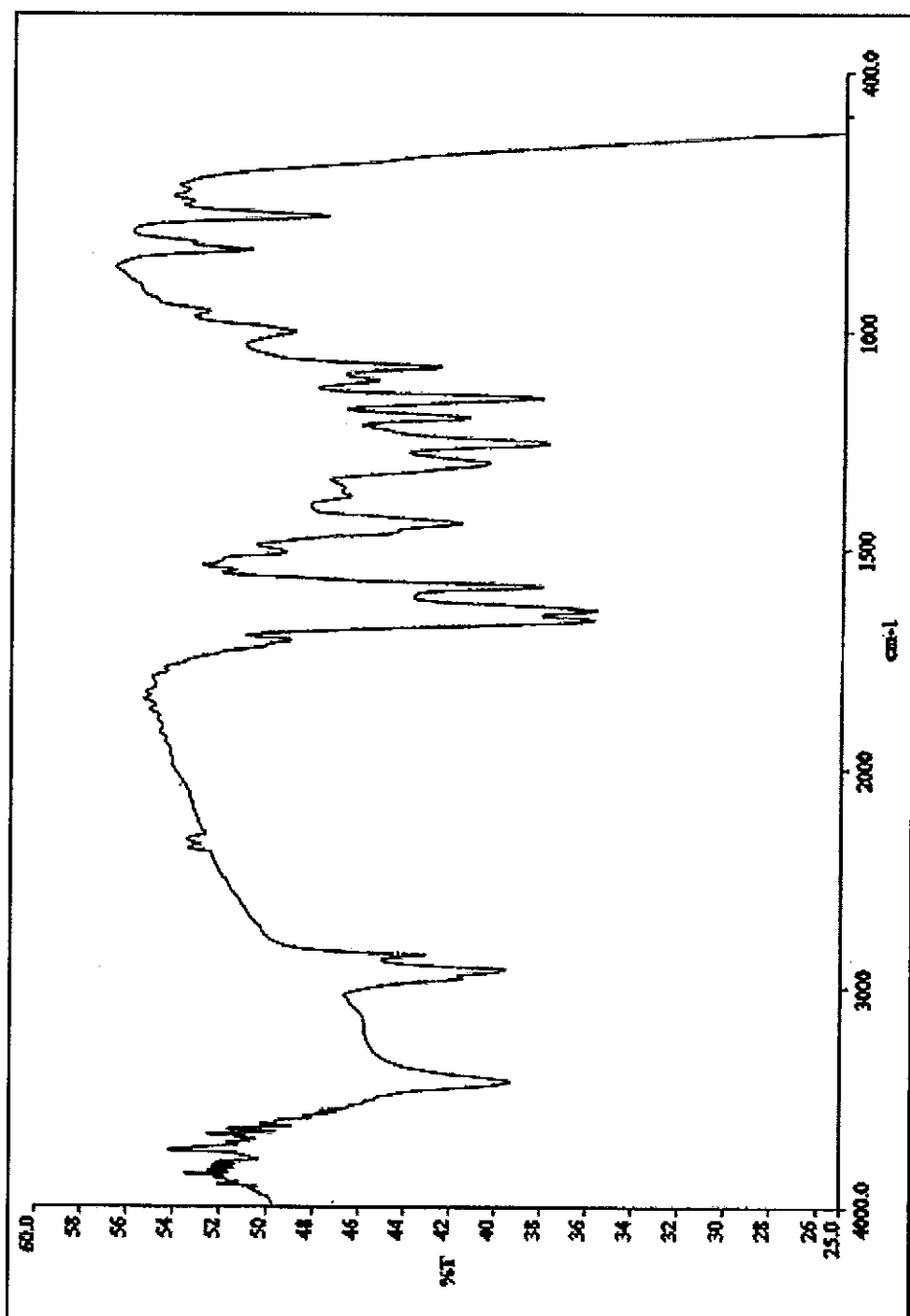


Figure 12 FT-IR (neat) spectrum of compound GP2

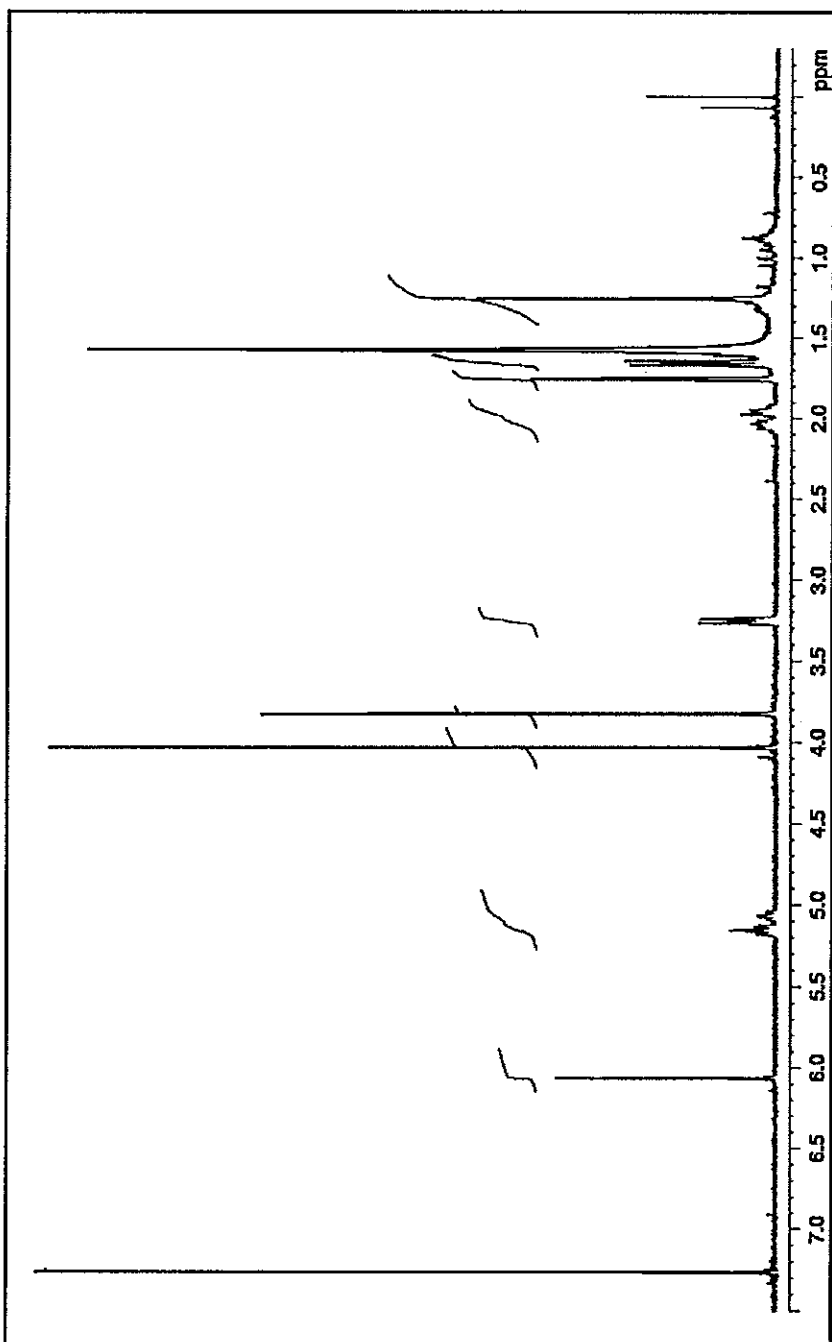


Figure 13  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound GP2

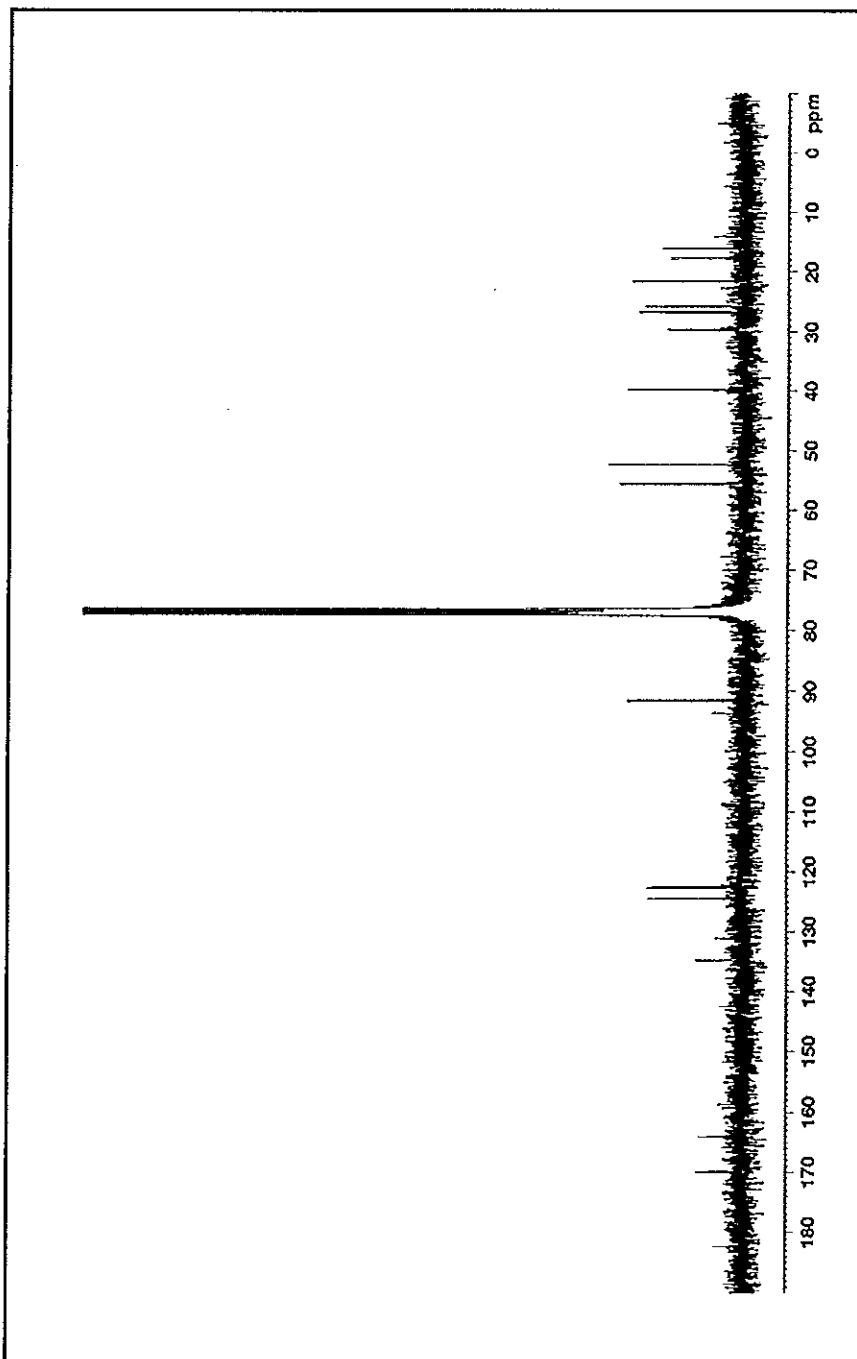


Figure 14  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound GP2

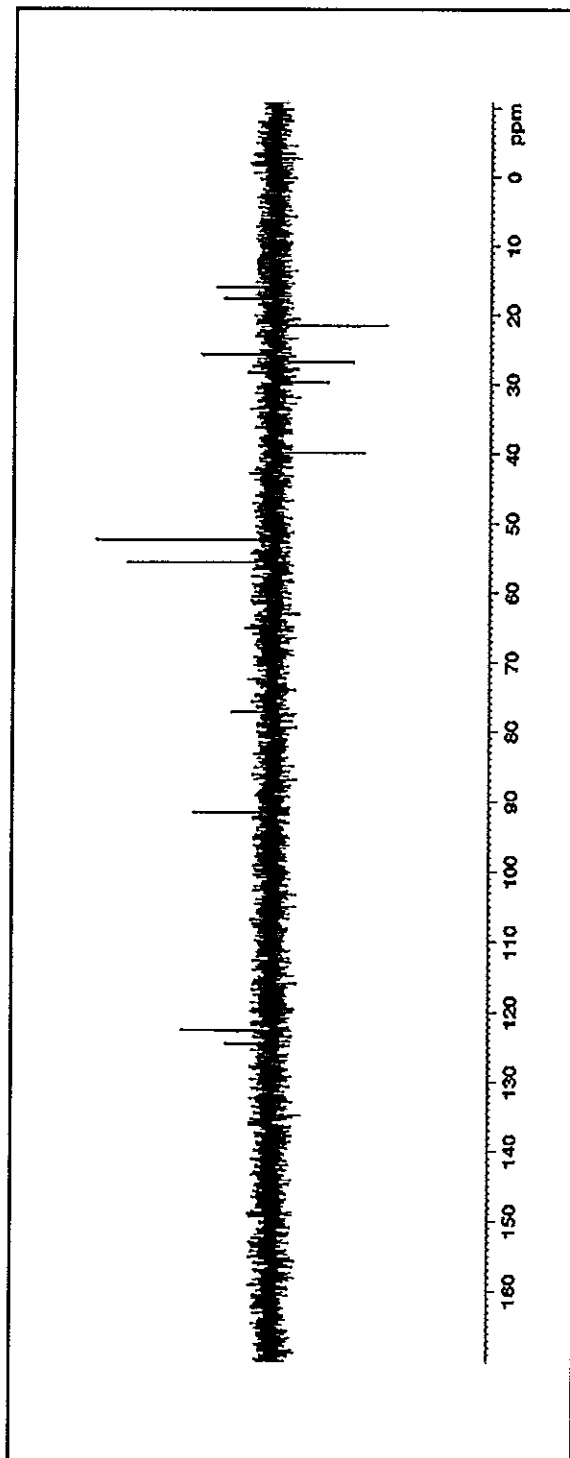


Figure 15 DEPT  $^{135}\text{C}$  spectra of compound GP2

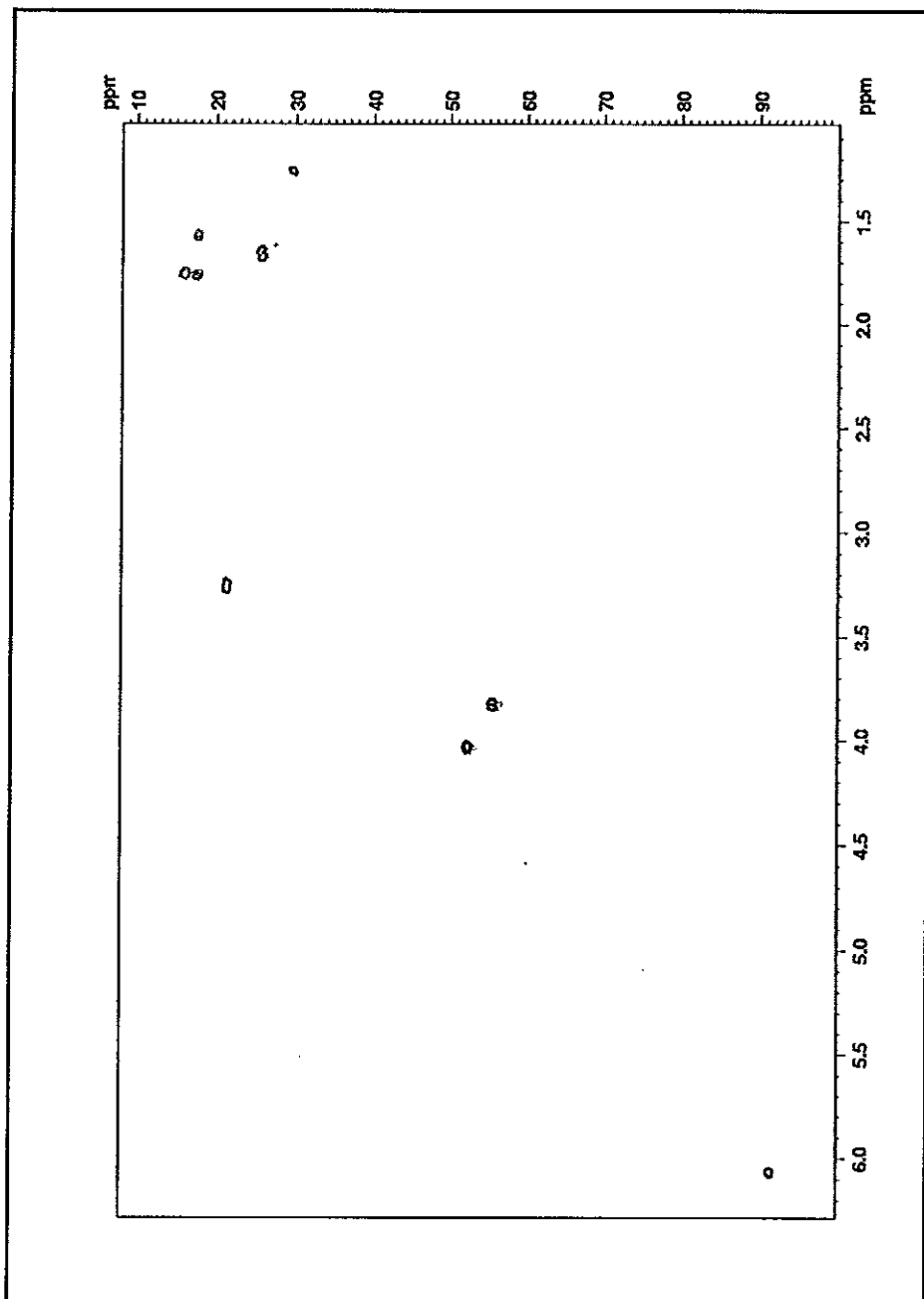


Figure 16 2D HMQC spectrum of compound GP2

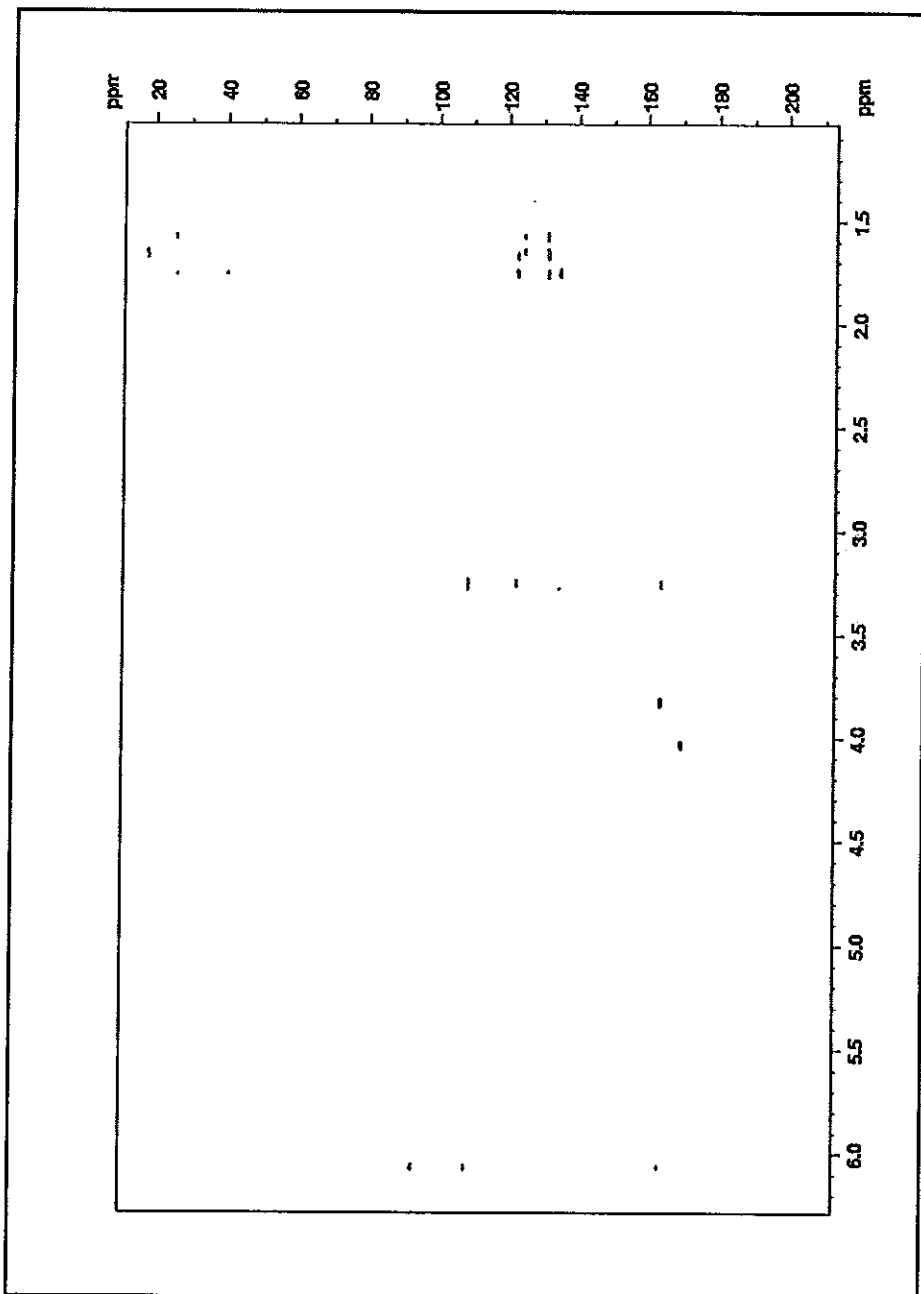


Figure 17 2D HMBC spectrum of compound GP2

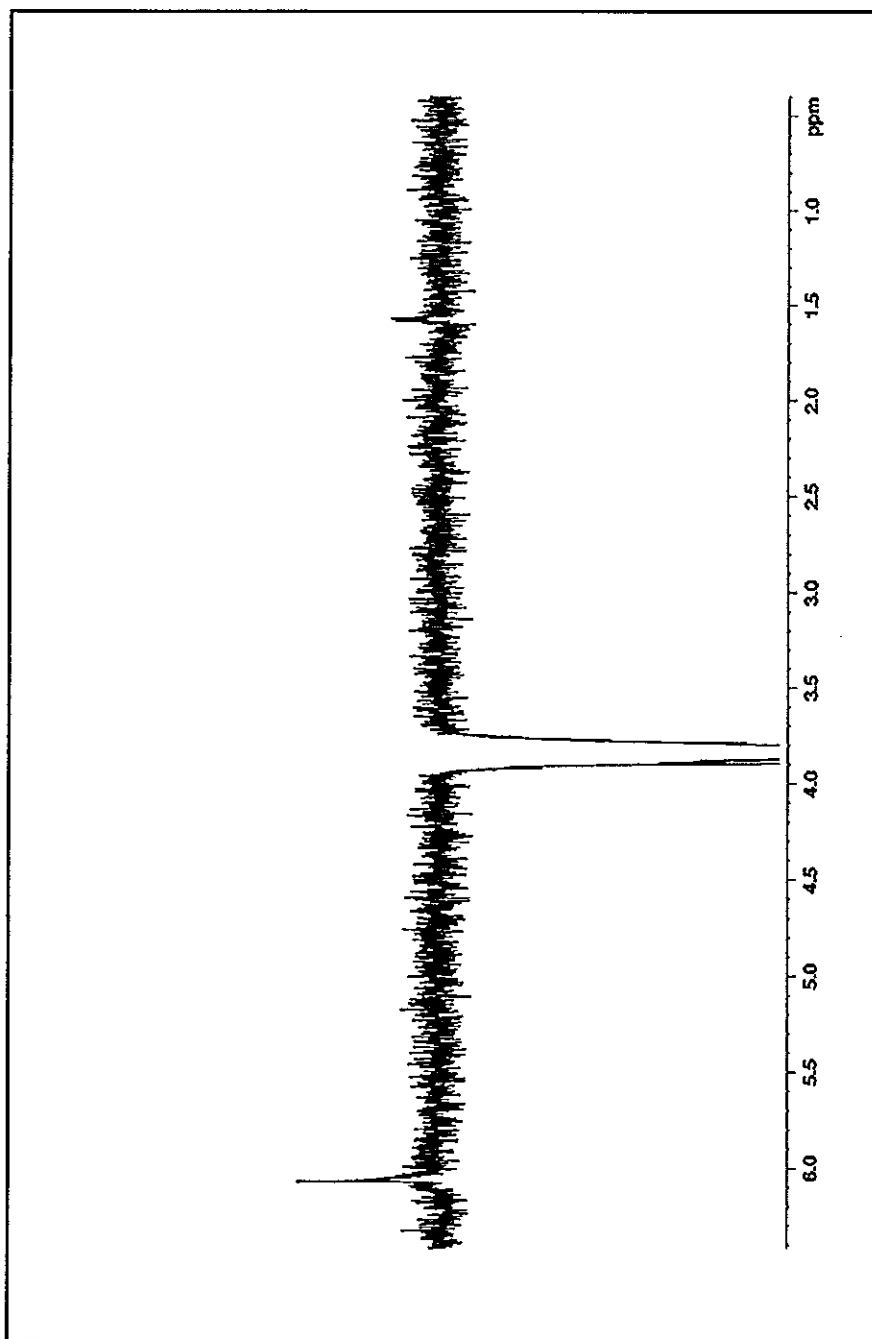


Figure 18 NOE difference spectrum of compound GP2 after irradiation at  $\delta_{\text{H}} 3.83$  (4-OCH<sub>3</sub>)

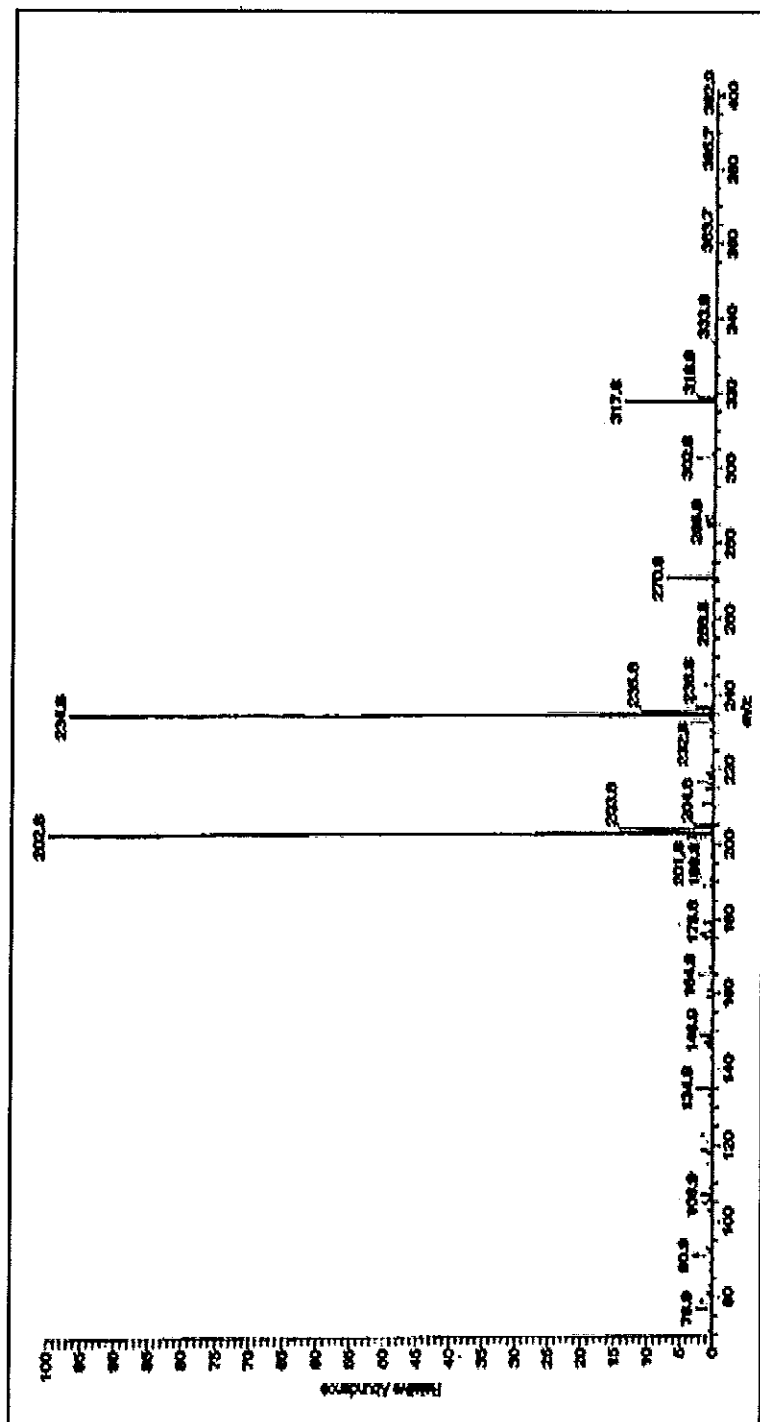


Figure 19 Mass spectrum of compound GP1



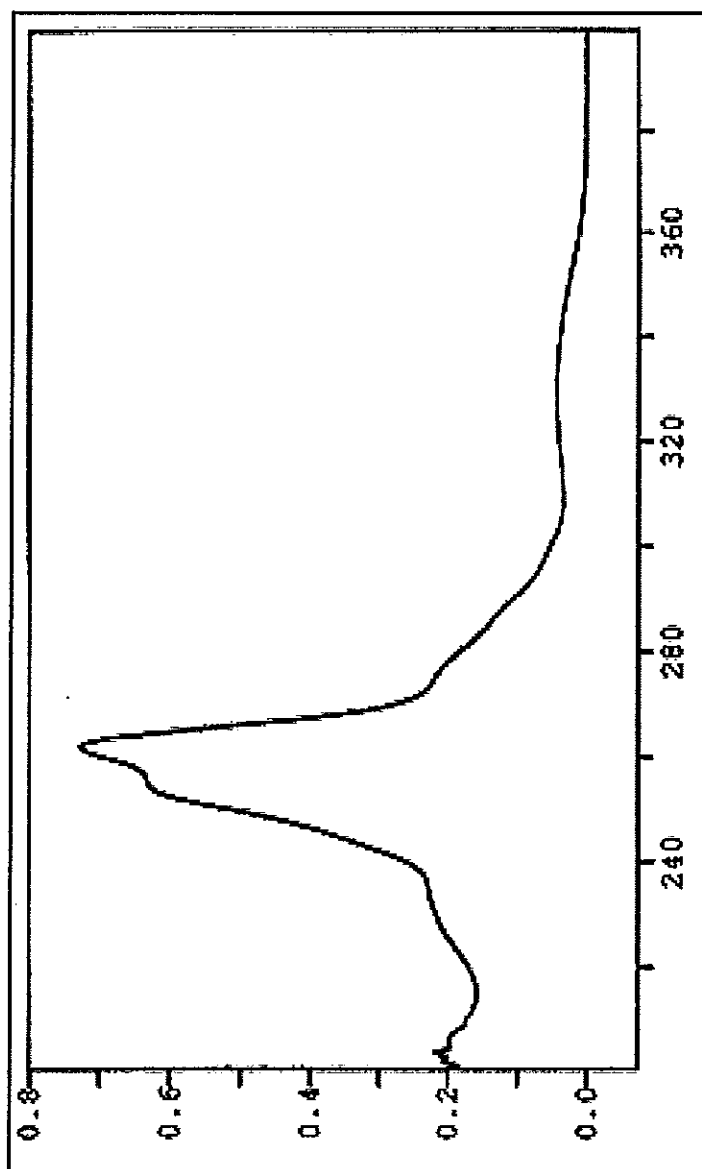


Figure 20 UV (MeOH) spectrum of compound GP1

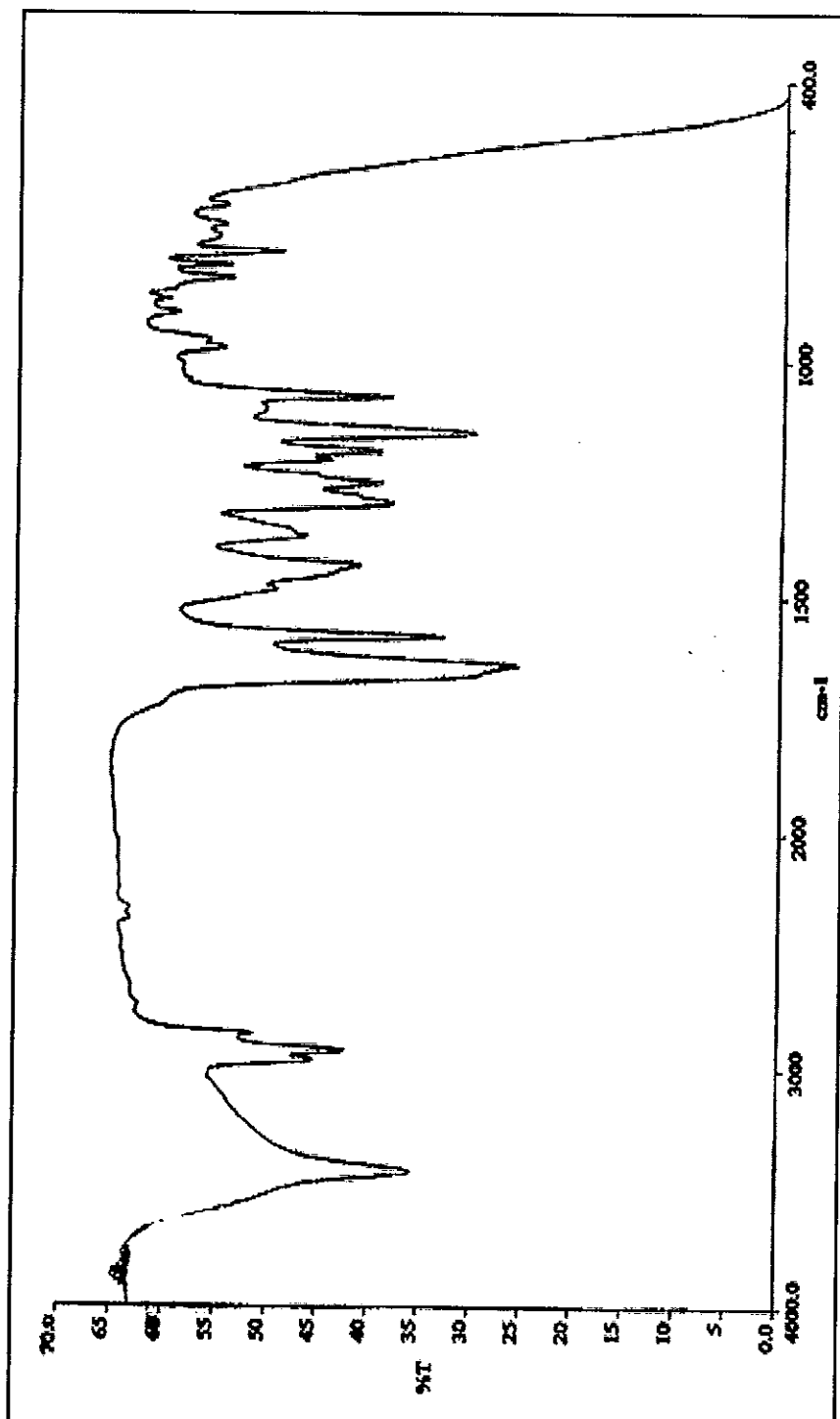


Figure 21 FT-IR (neat) spectrum of compound GP1

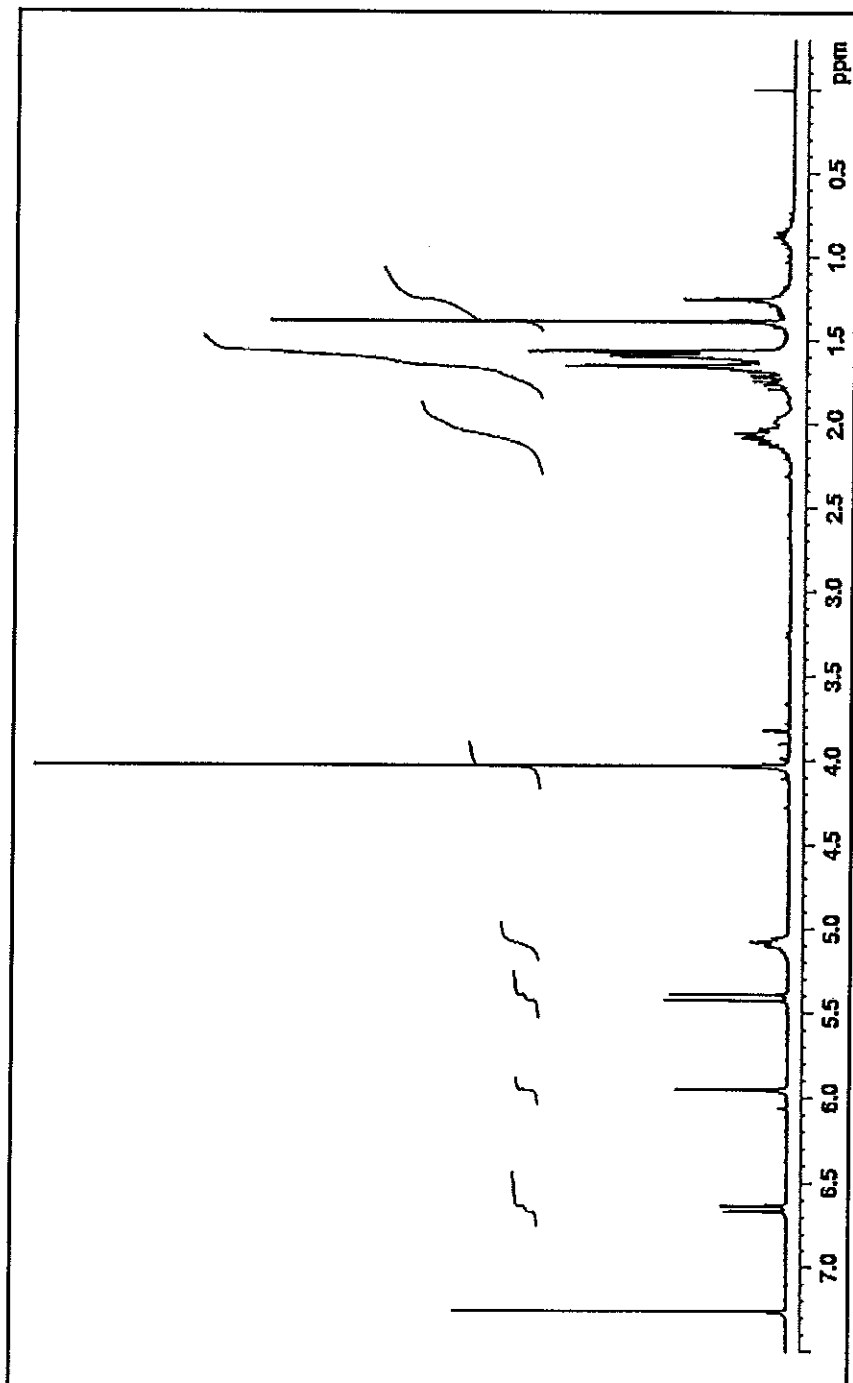


Figure 22  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound GP1

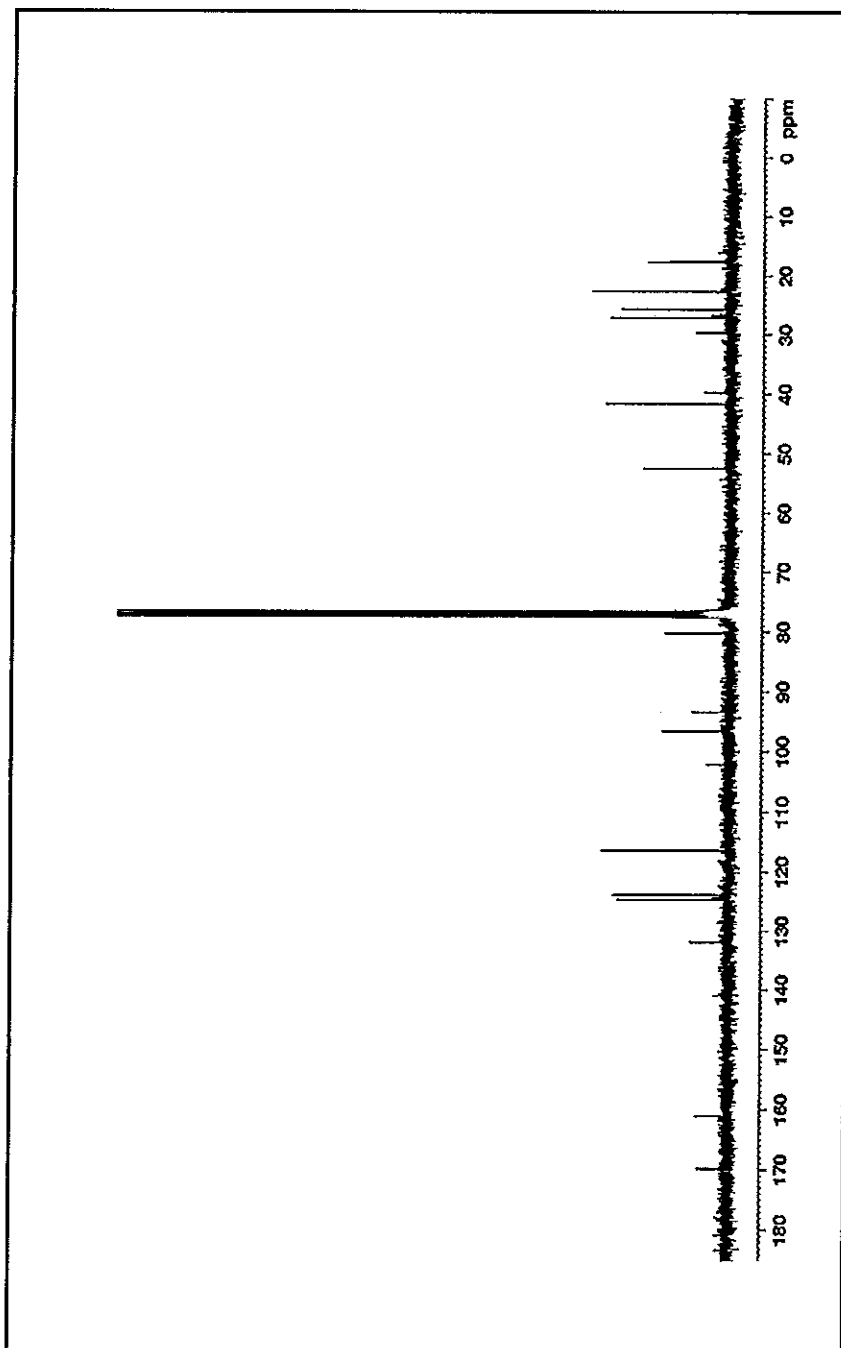


Figure 23  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound GPI

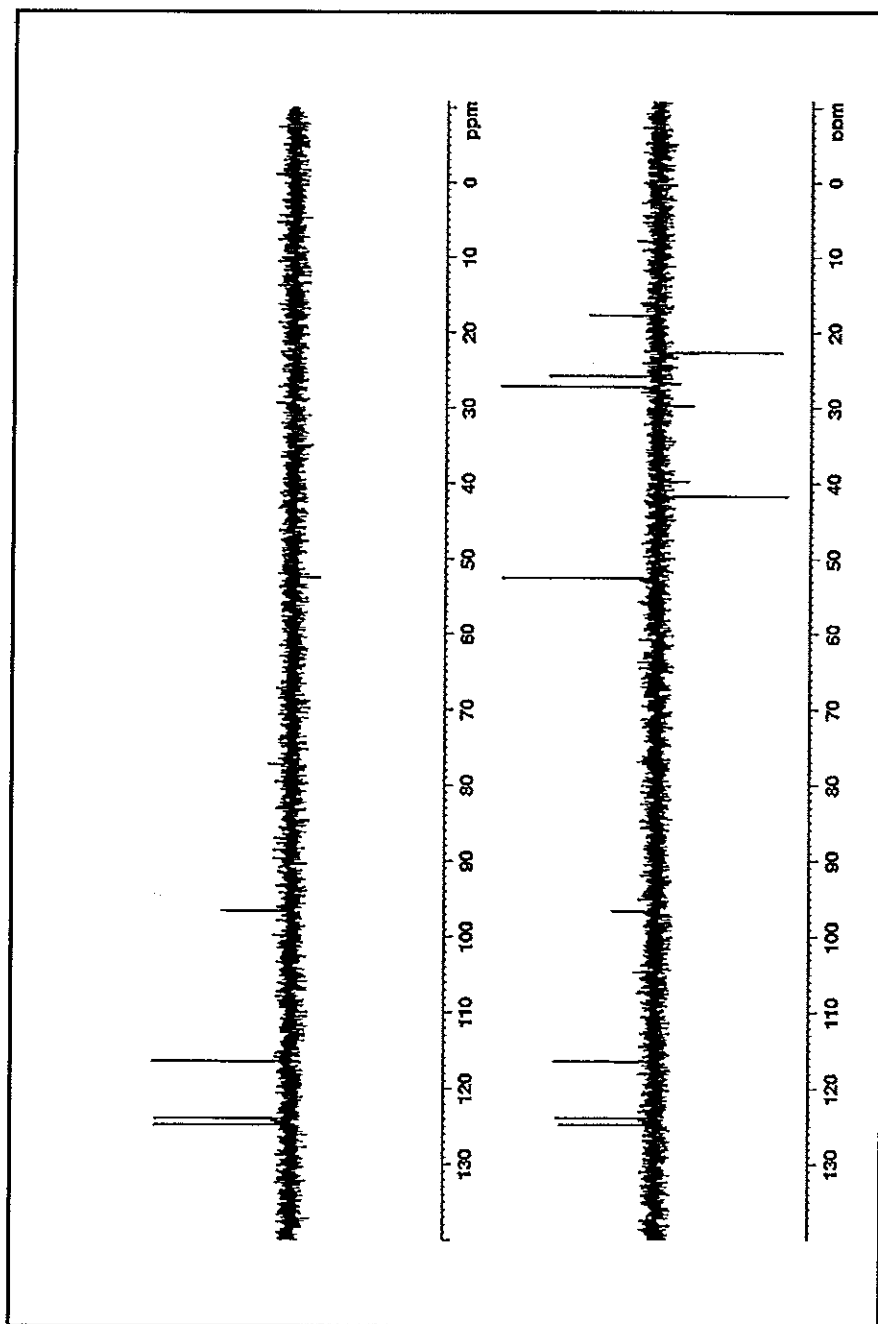


Figure 24 DEPT  $90^\circ$  and  $135^\circ$  spectra of compound GPI

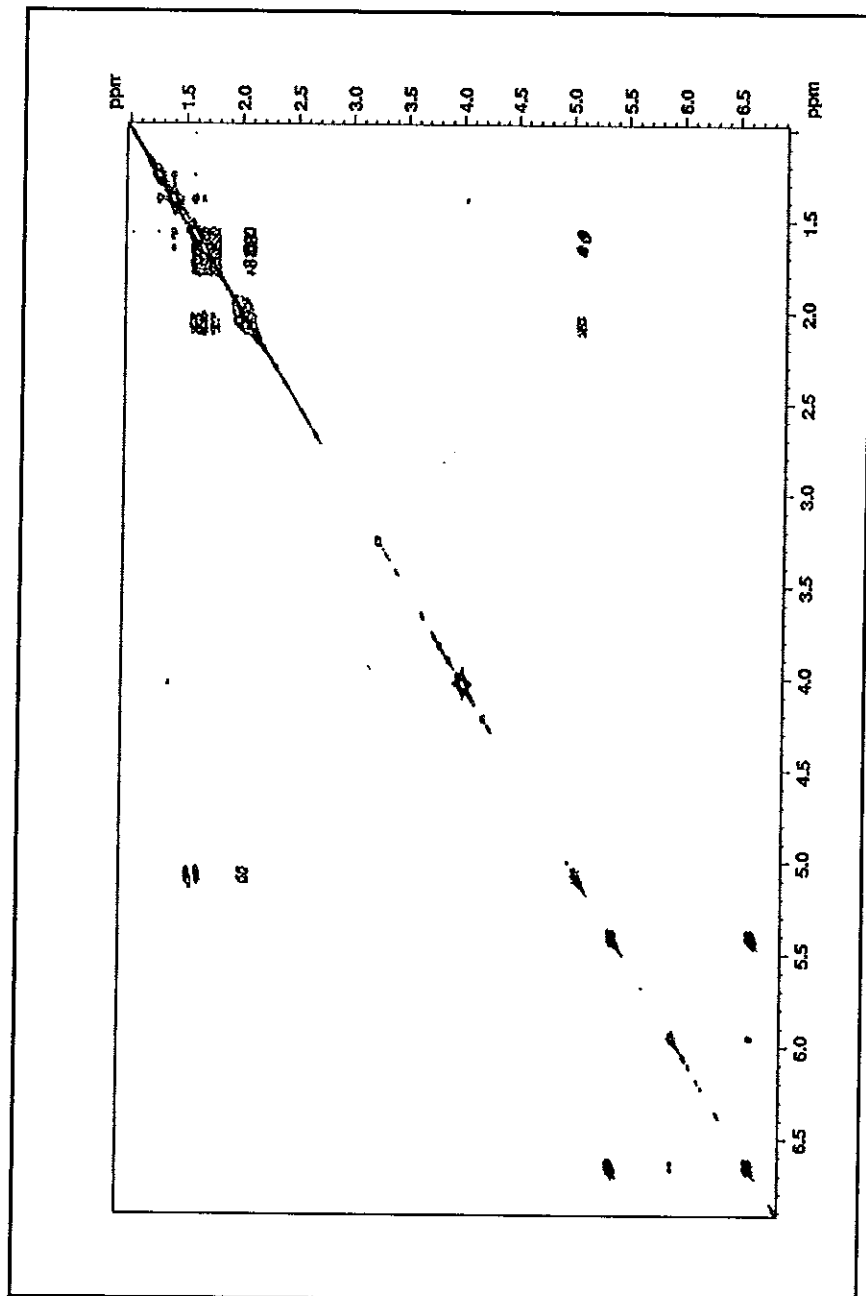


Figure 25  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of compound GP1

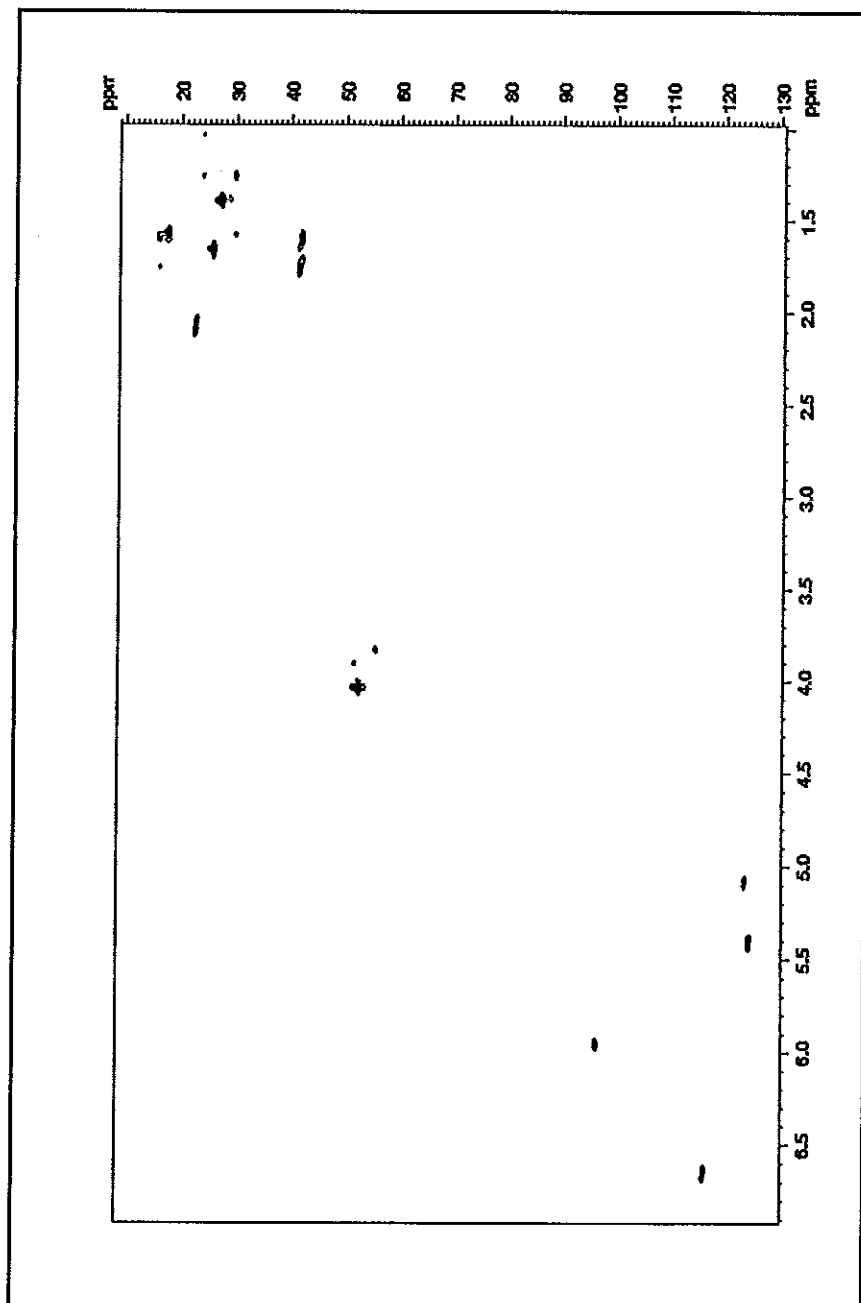


Figure 26 2D HMQC spectrum of compound GPI

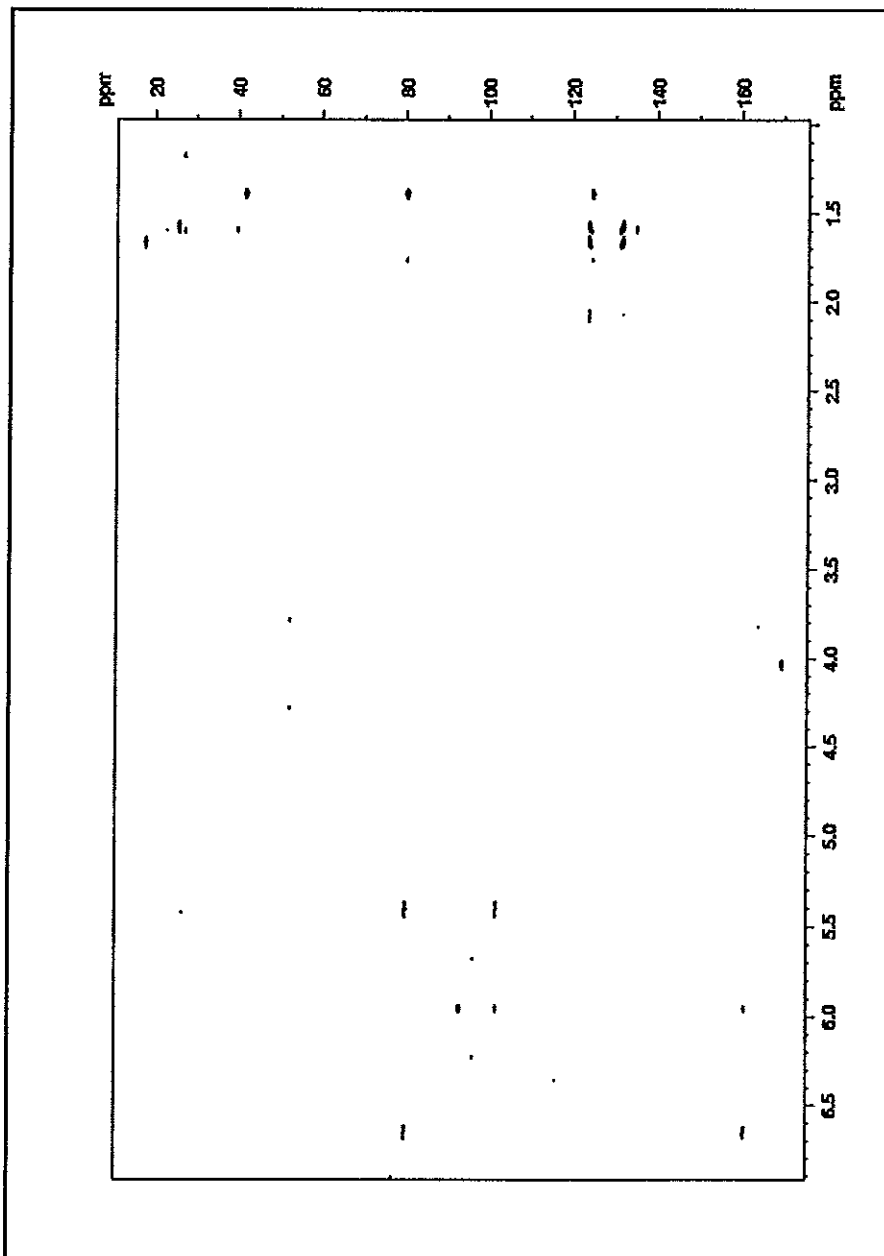


Figure 27 2D HMBC spectrum of compound GP1



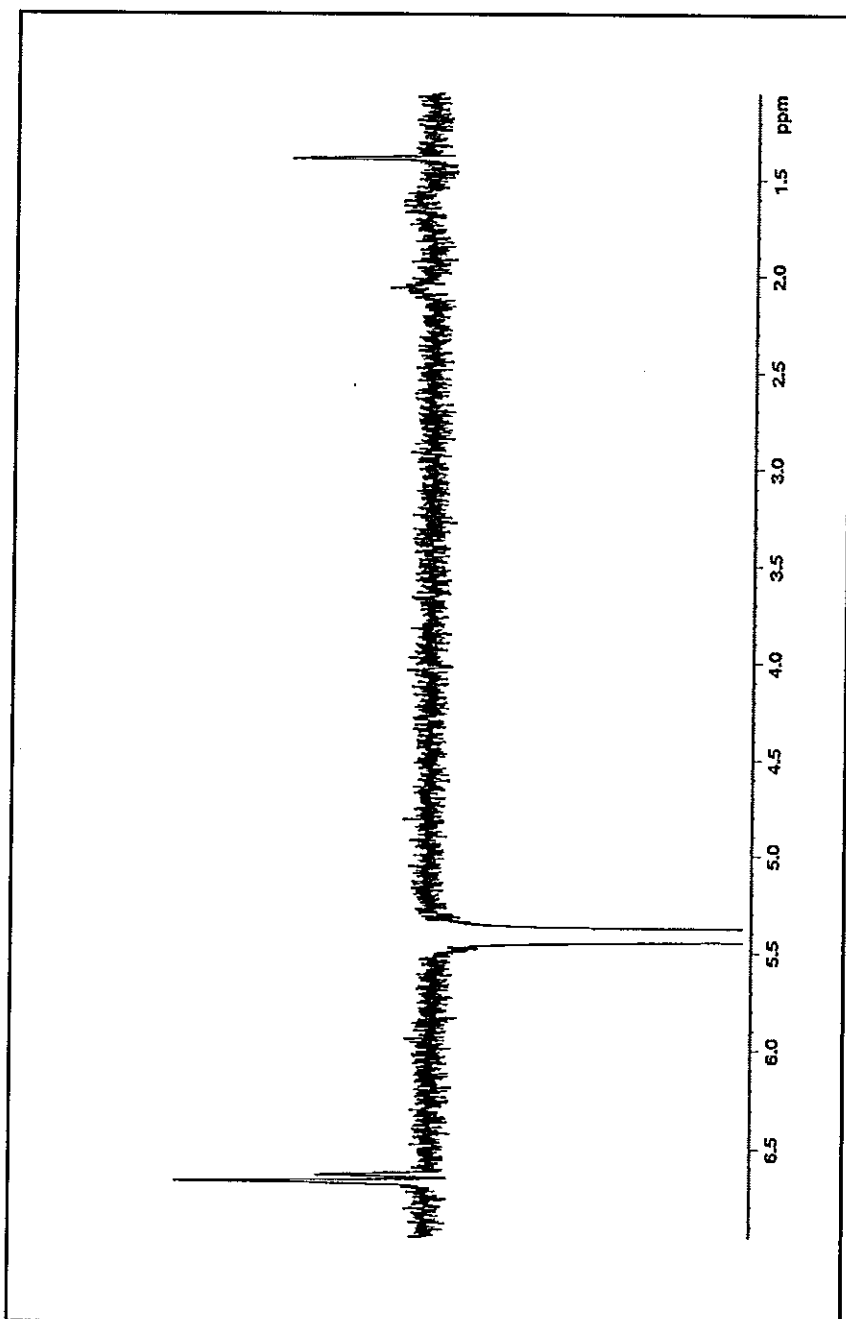


Figure 28 NOE difference spectrum of compound GPI after irradiation at  $\delta_{\text{H}} 5.41$  (H-3)

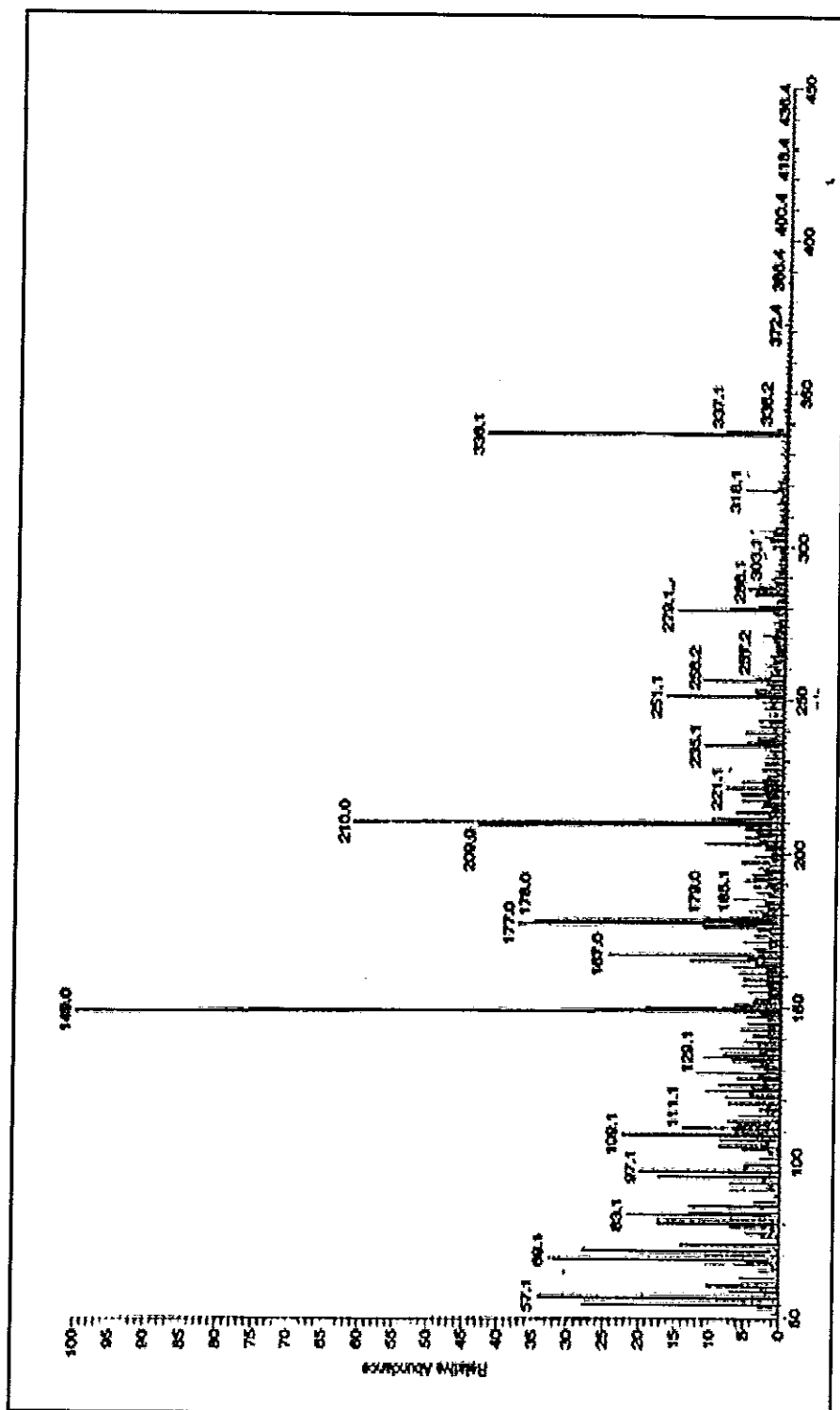


Figure 29 Mass spectrum of compound GP6

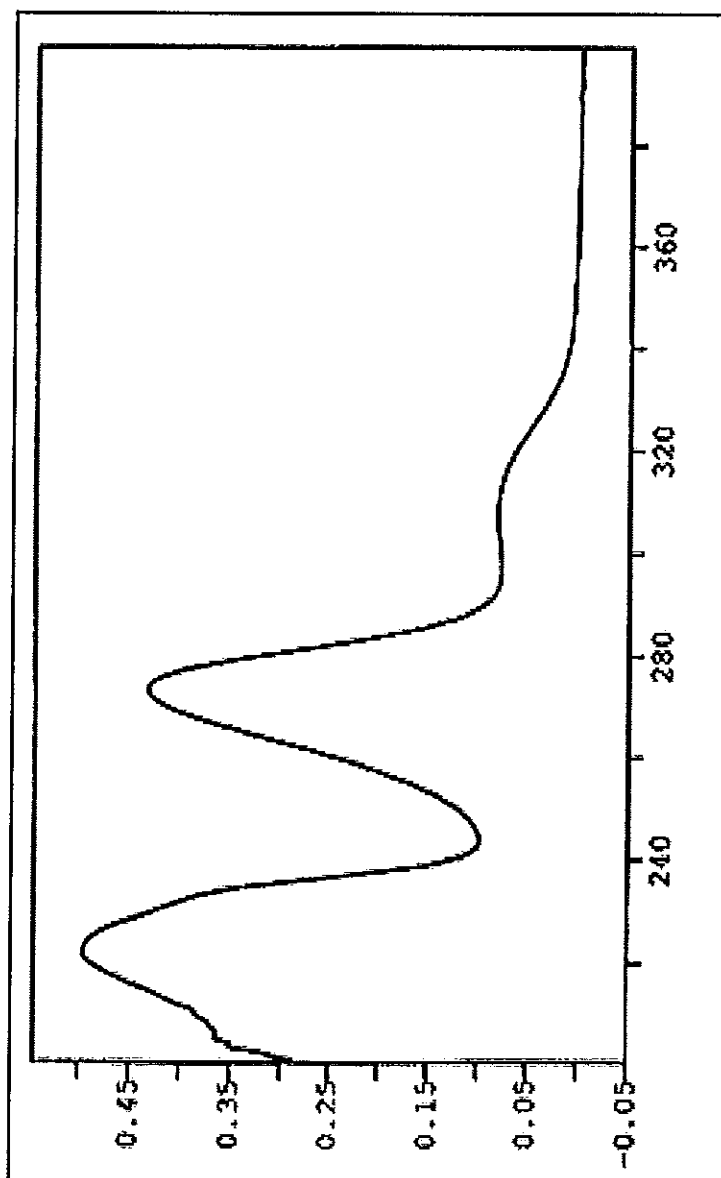


Figure 30 UV (MeOH) spectrum of compound GP6

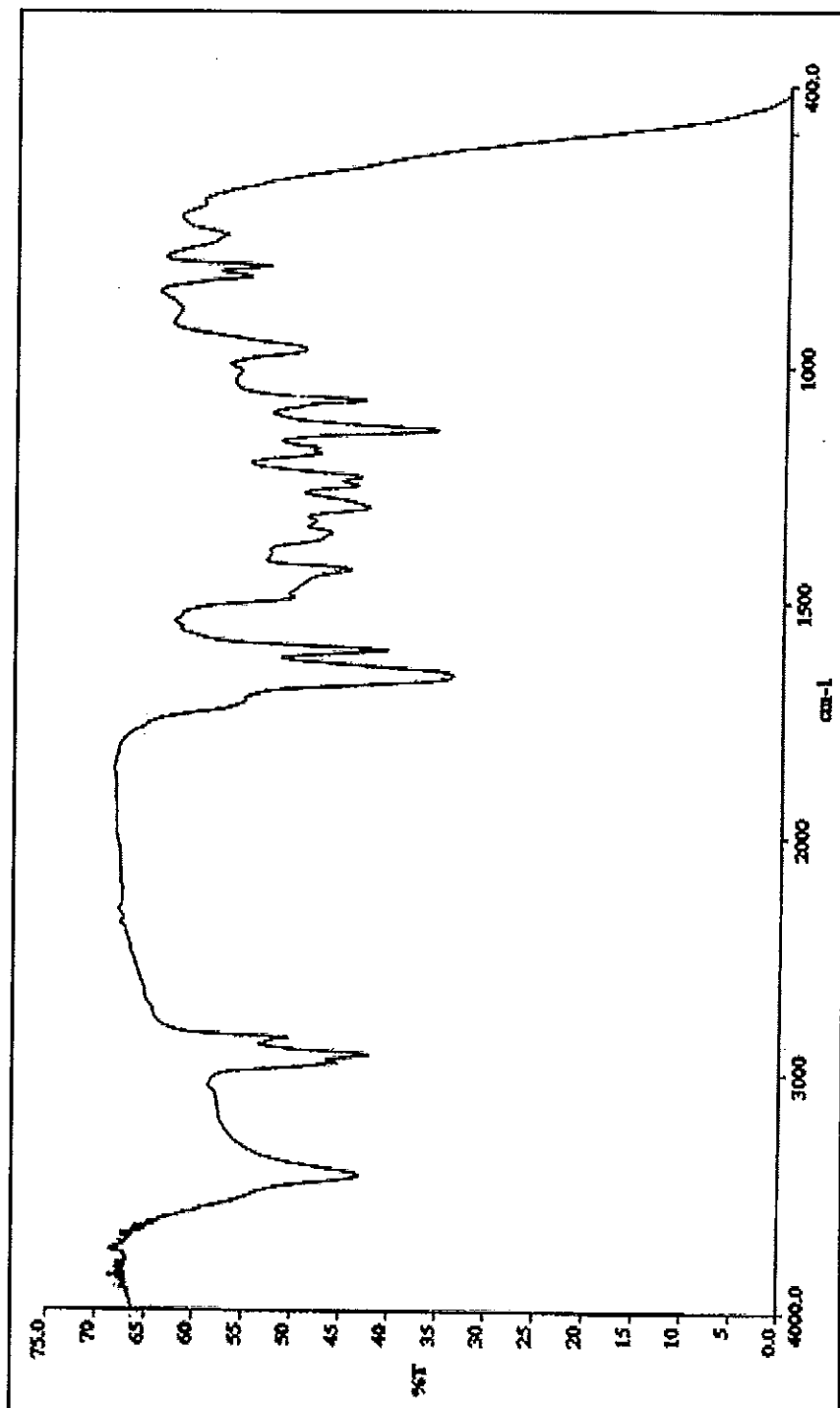


Figure 31 FT-IR (neat) spectrum of compound GP6

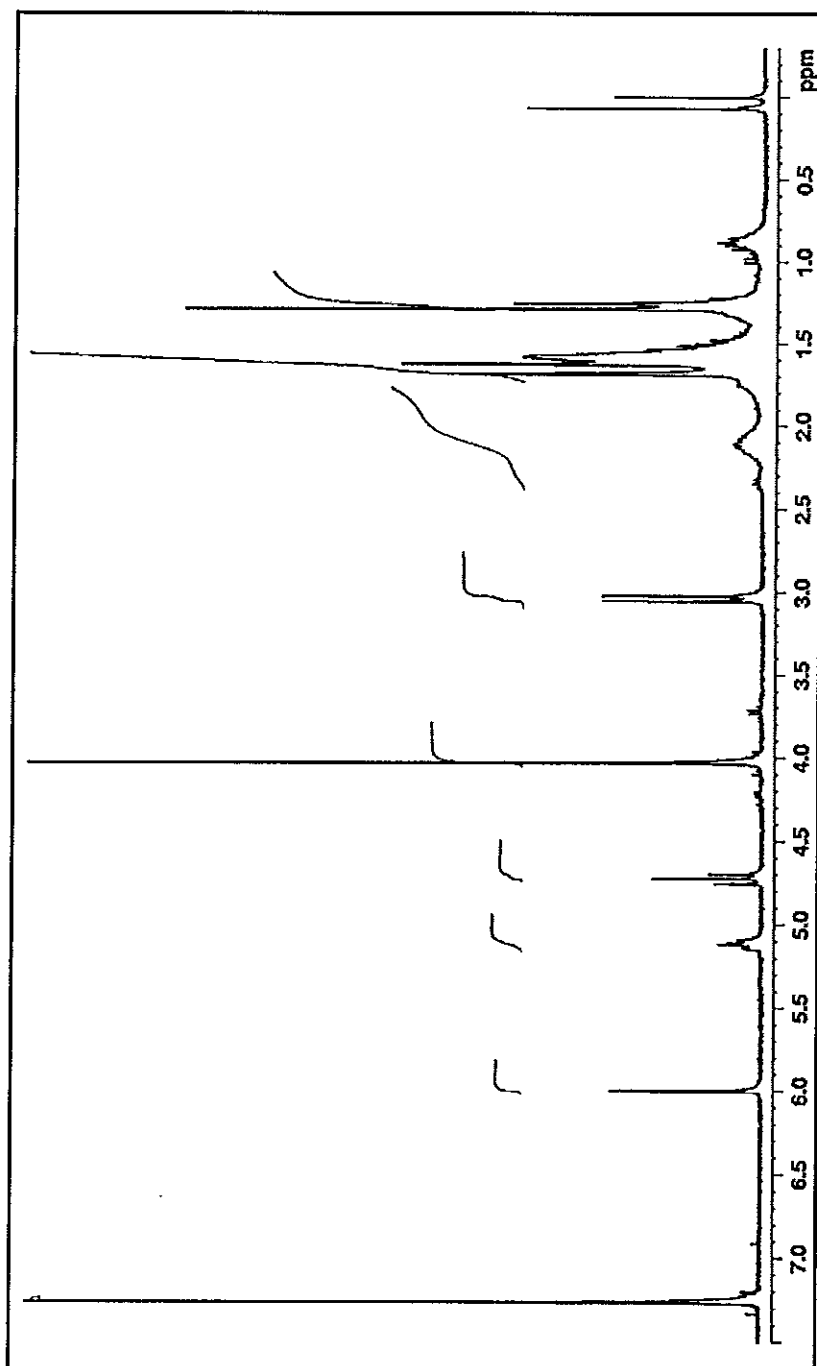


Figure 32  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound GP6

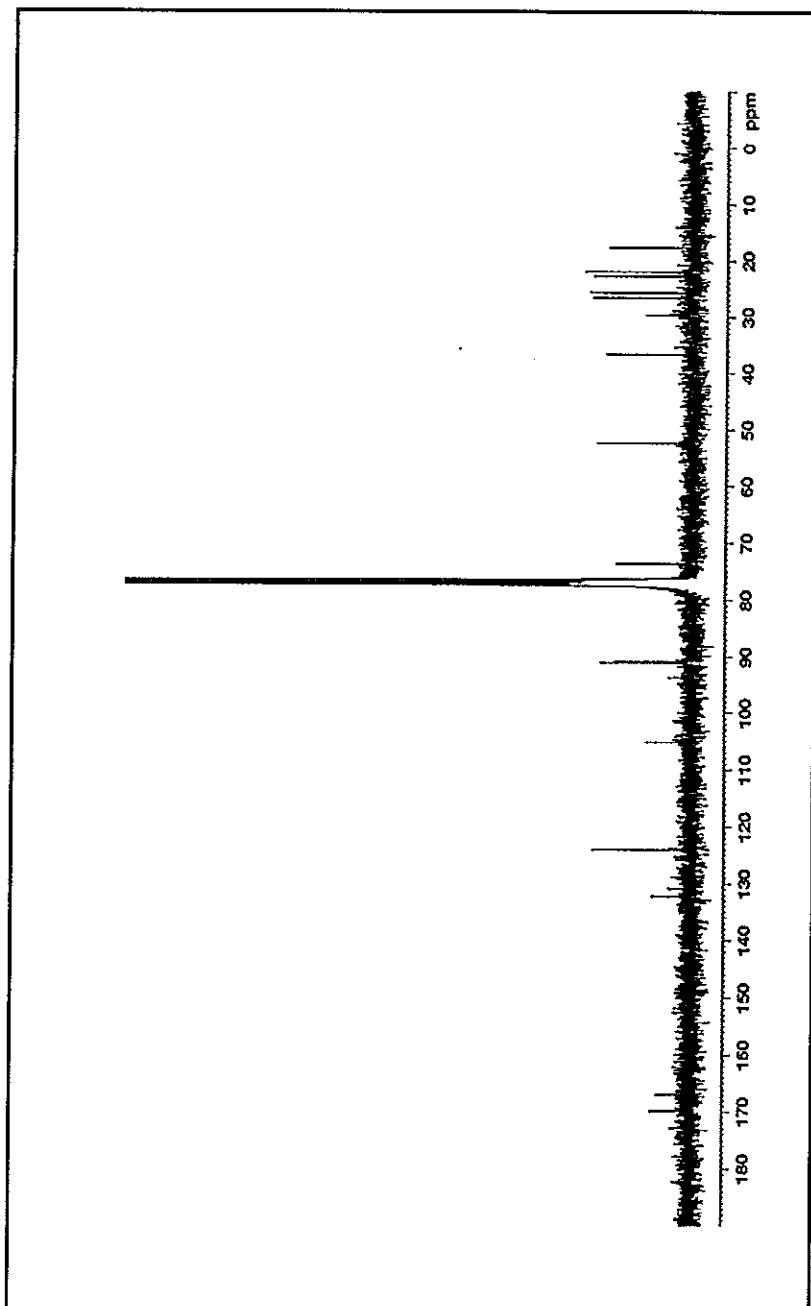


Figure 33  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound GP6

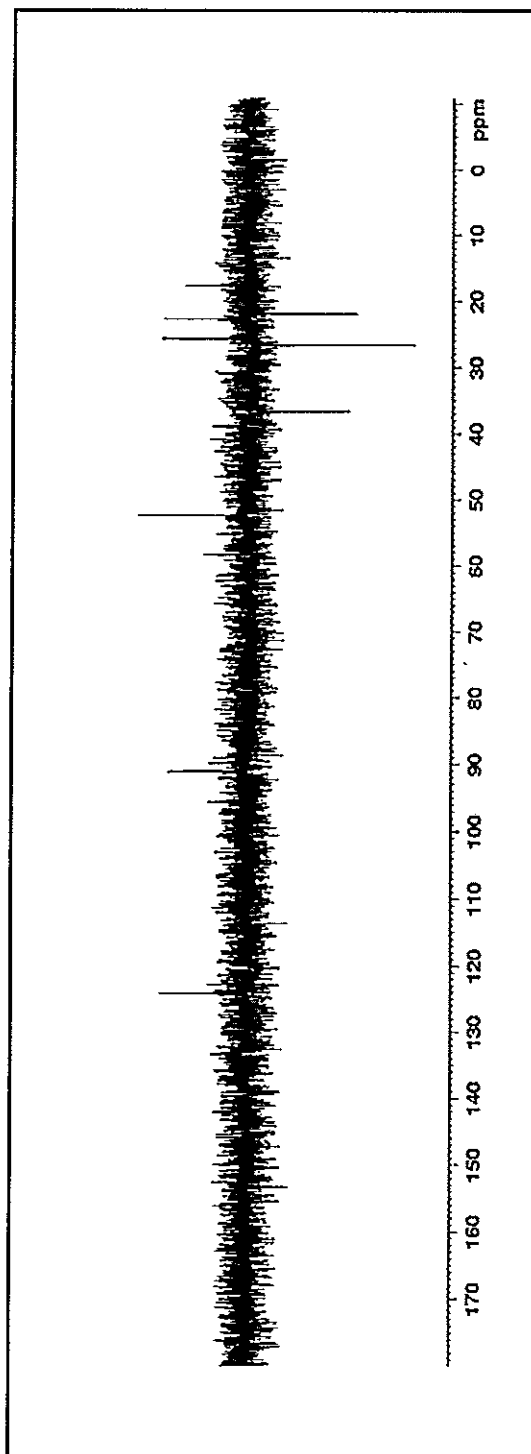


Figure 34 DEPT 135° spectra of compound GP6

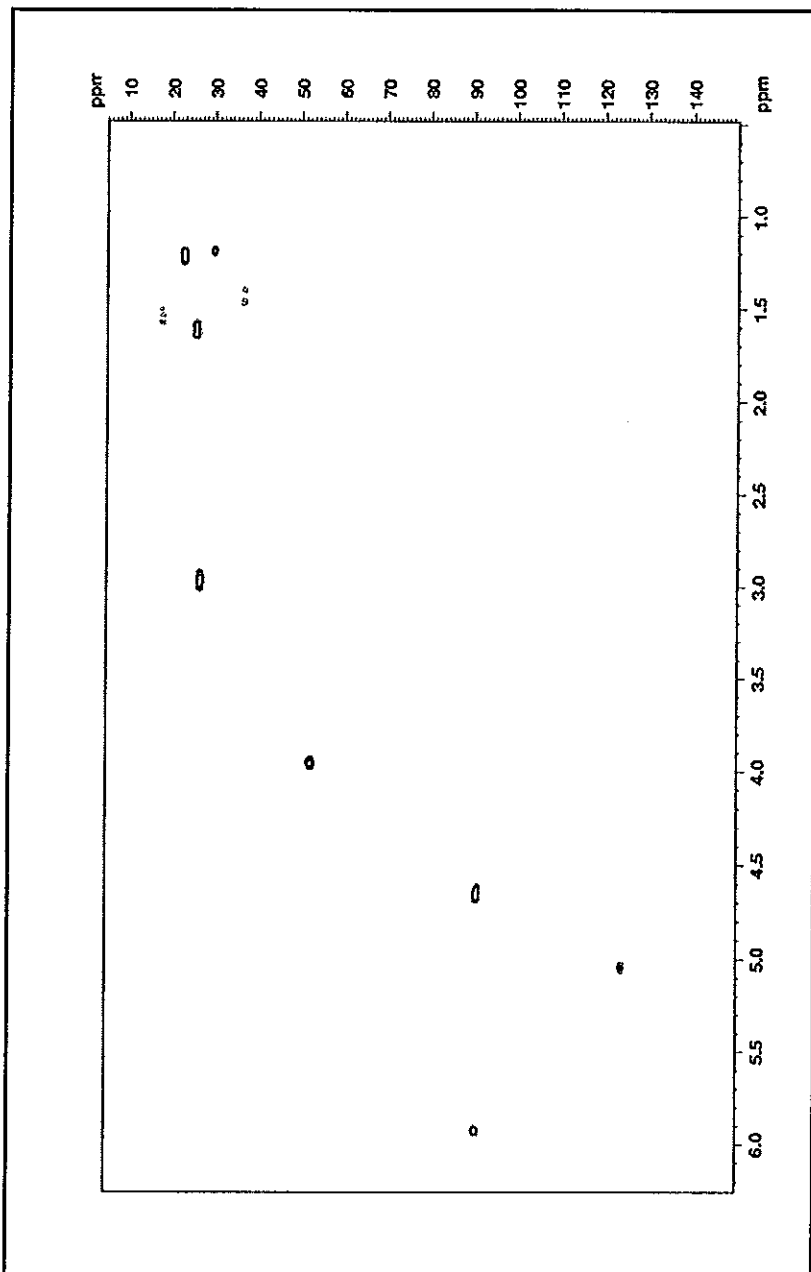


Figure 35 2D HMQC spectrum of compound GP6



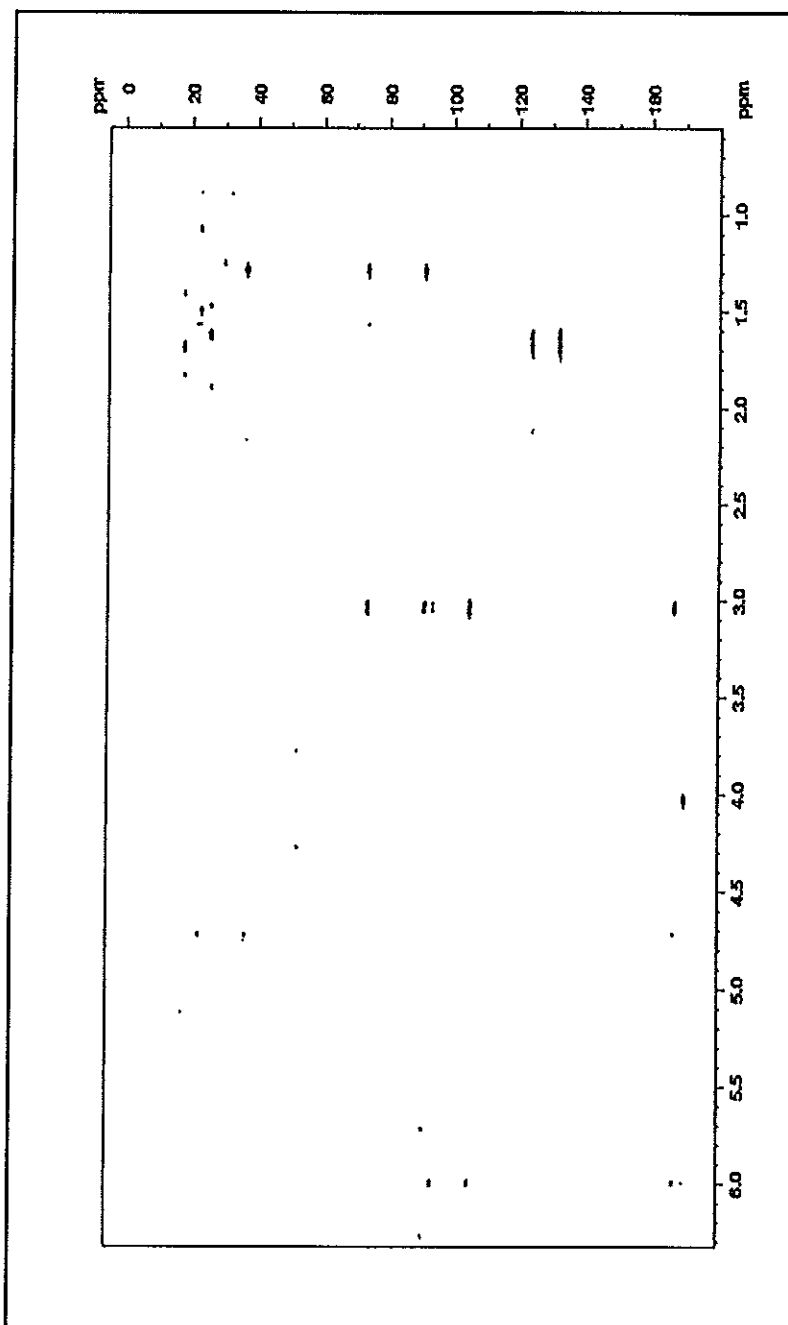


Figure 36 2D HMBC spectrum of compound GP6

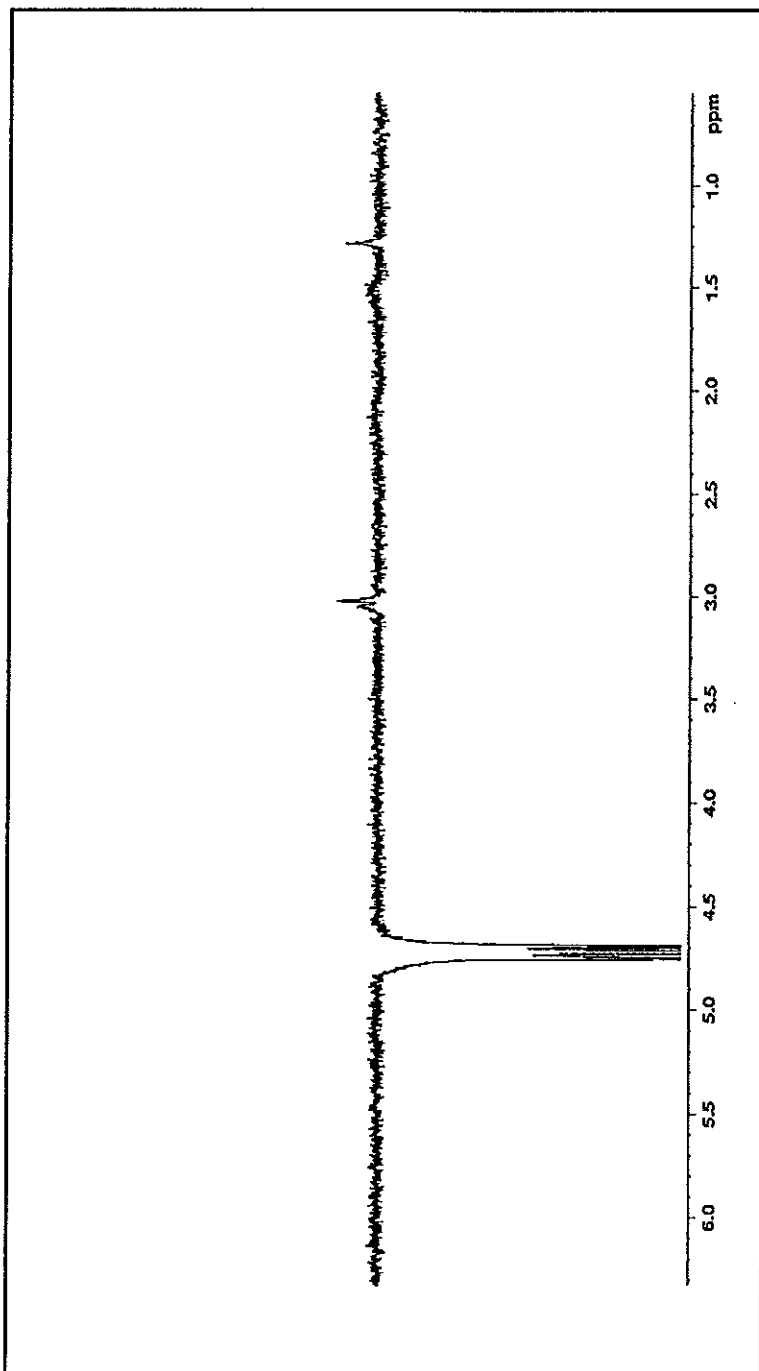


Figure 37 NOE difference spectrum of compound GP6 after irradiation at  $\delta_H$  4.73 (H-3)

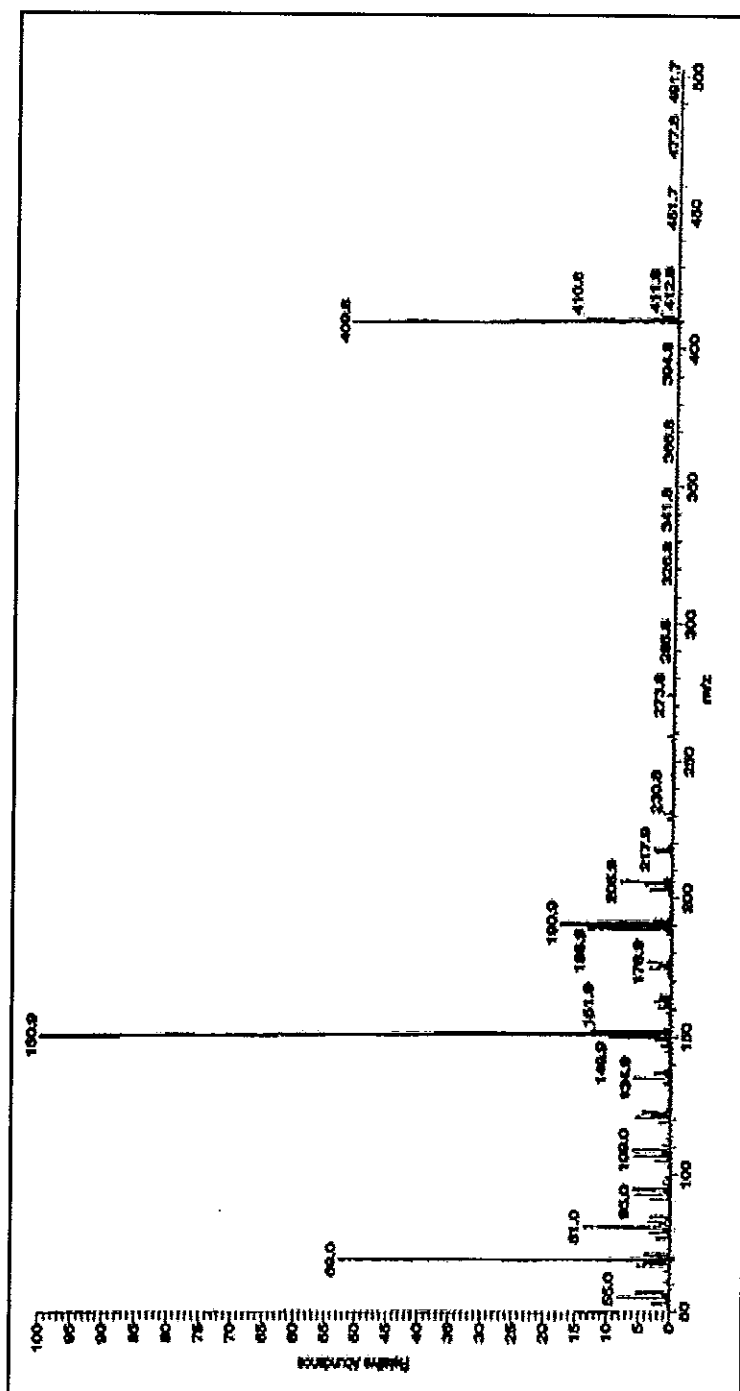


Figure 38 Mass spectrum of compound GP3

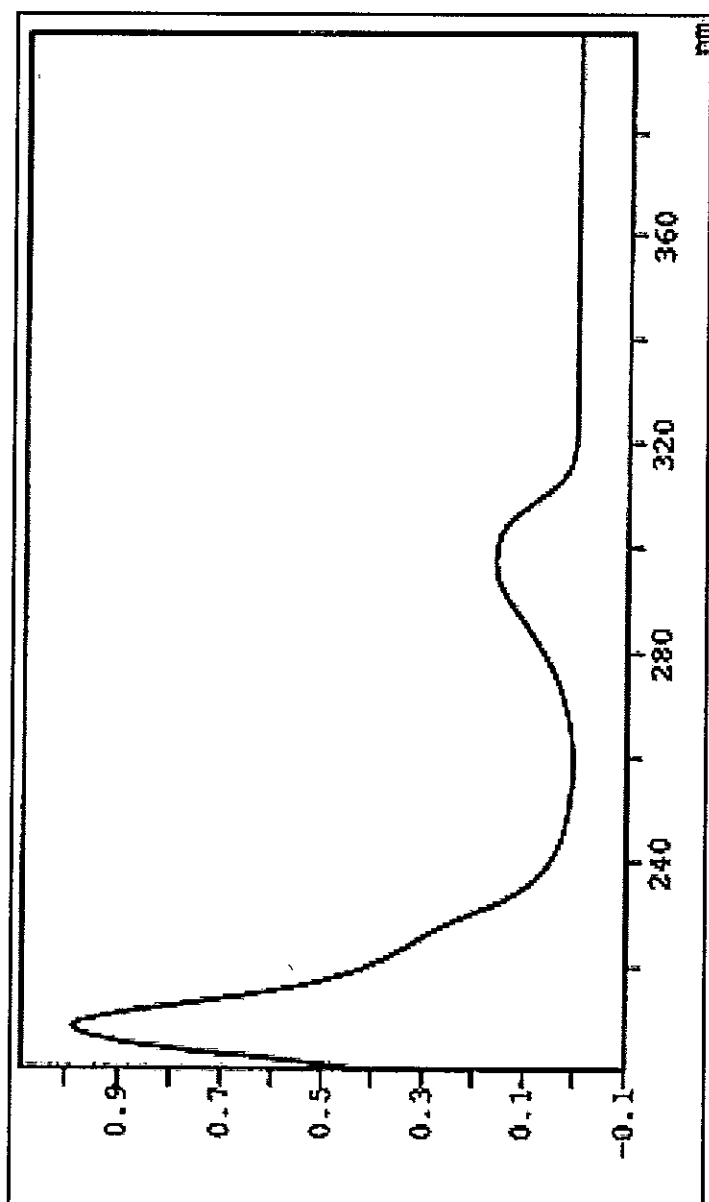


Figure 39 UV (MeOH) spectrum of compound GP3

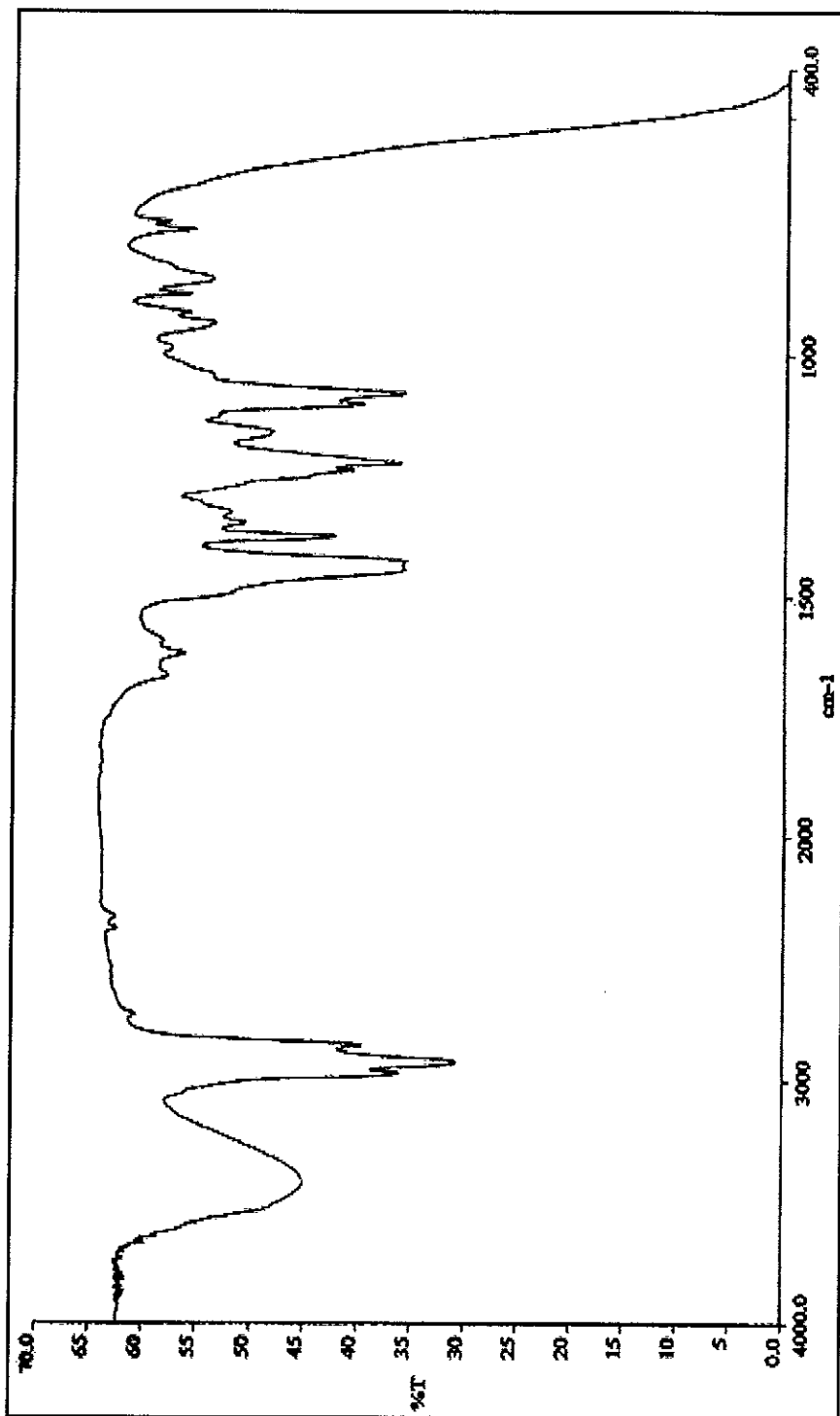


Figure 40 FT-IR (neat) spectrum of compound GP3

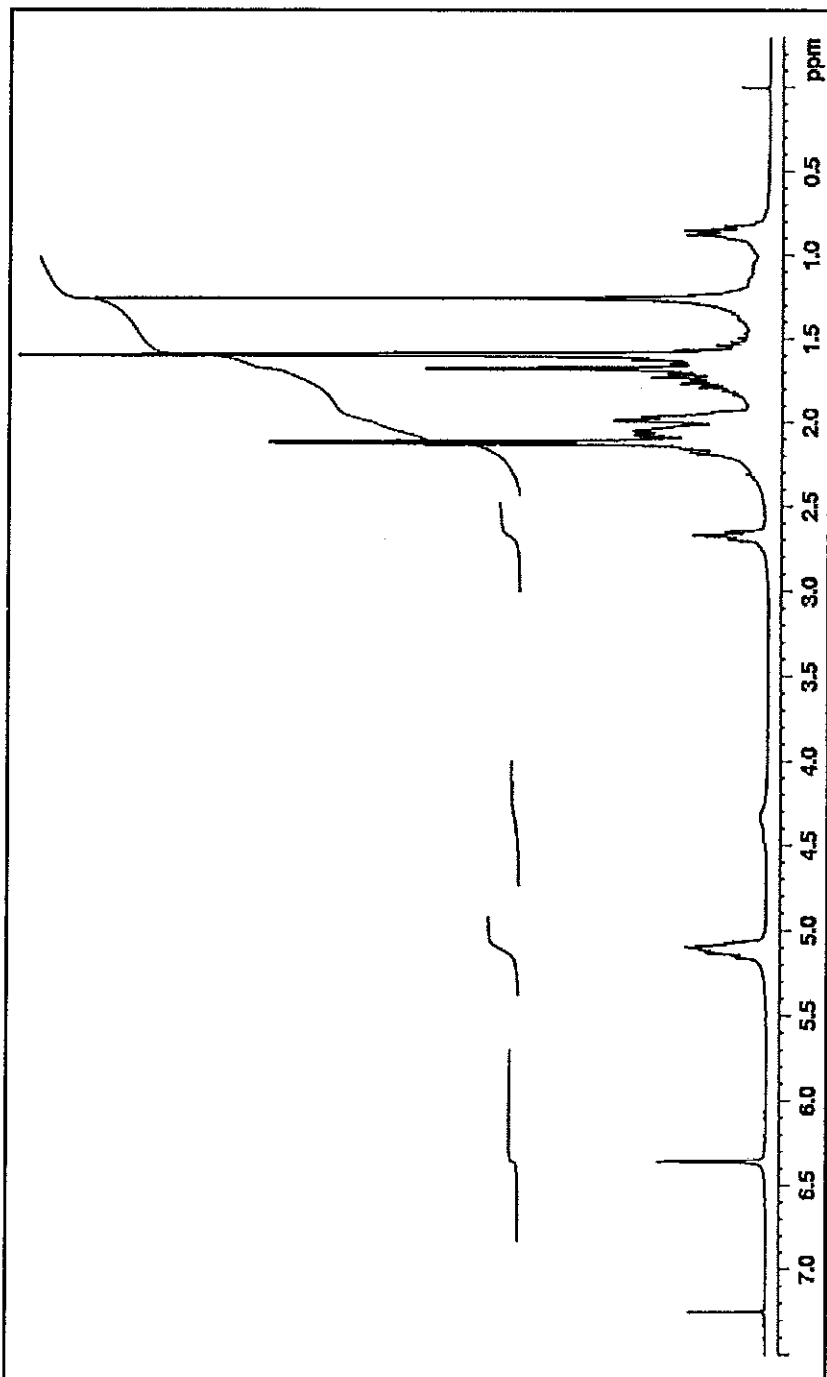


Figure 41  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound GP3

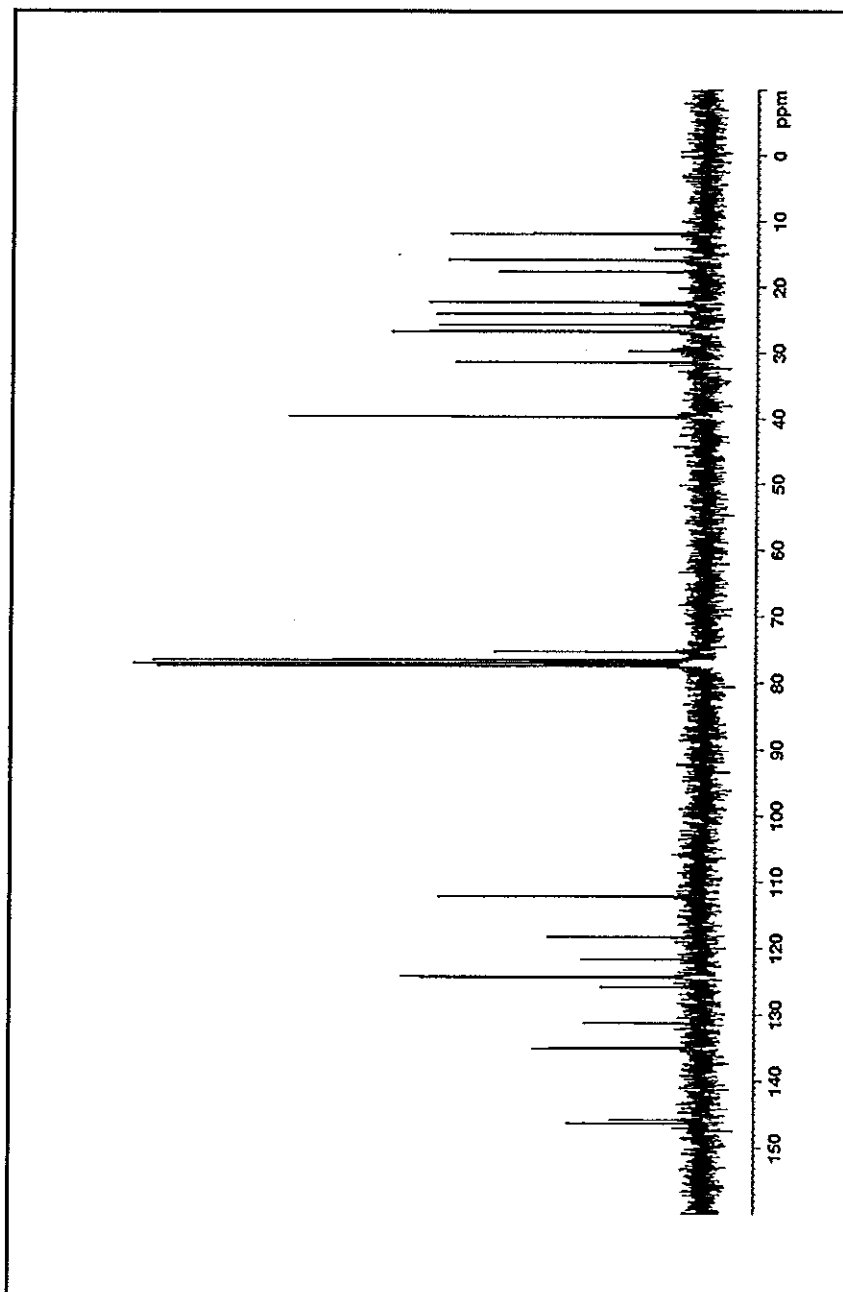
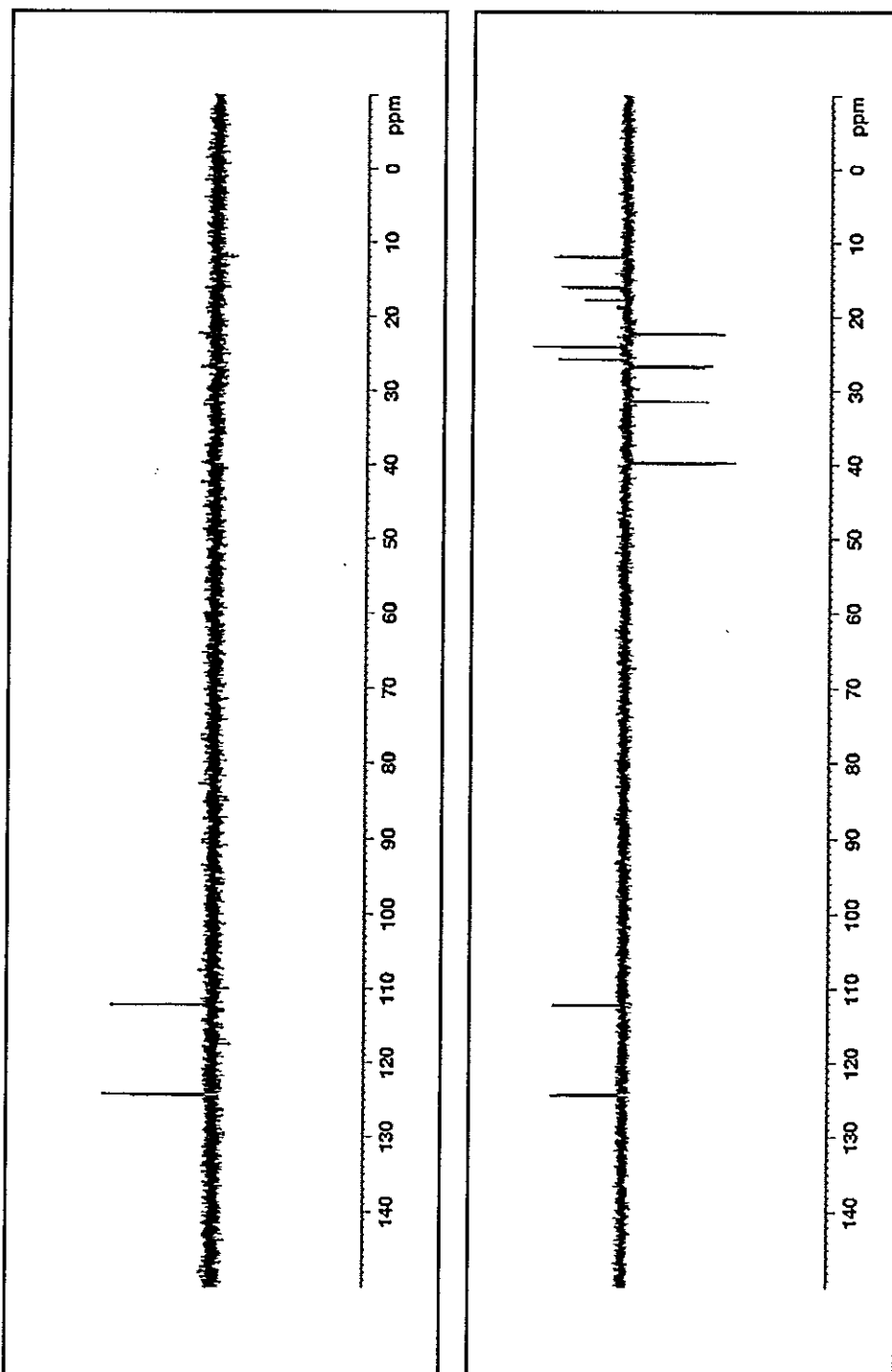
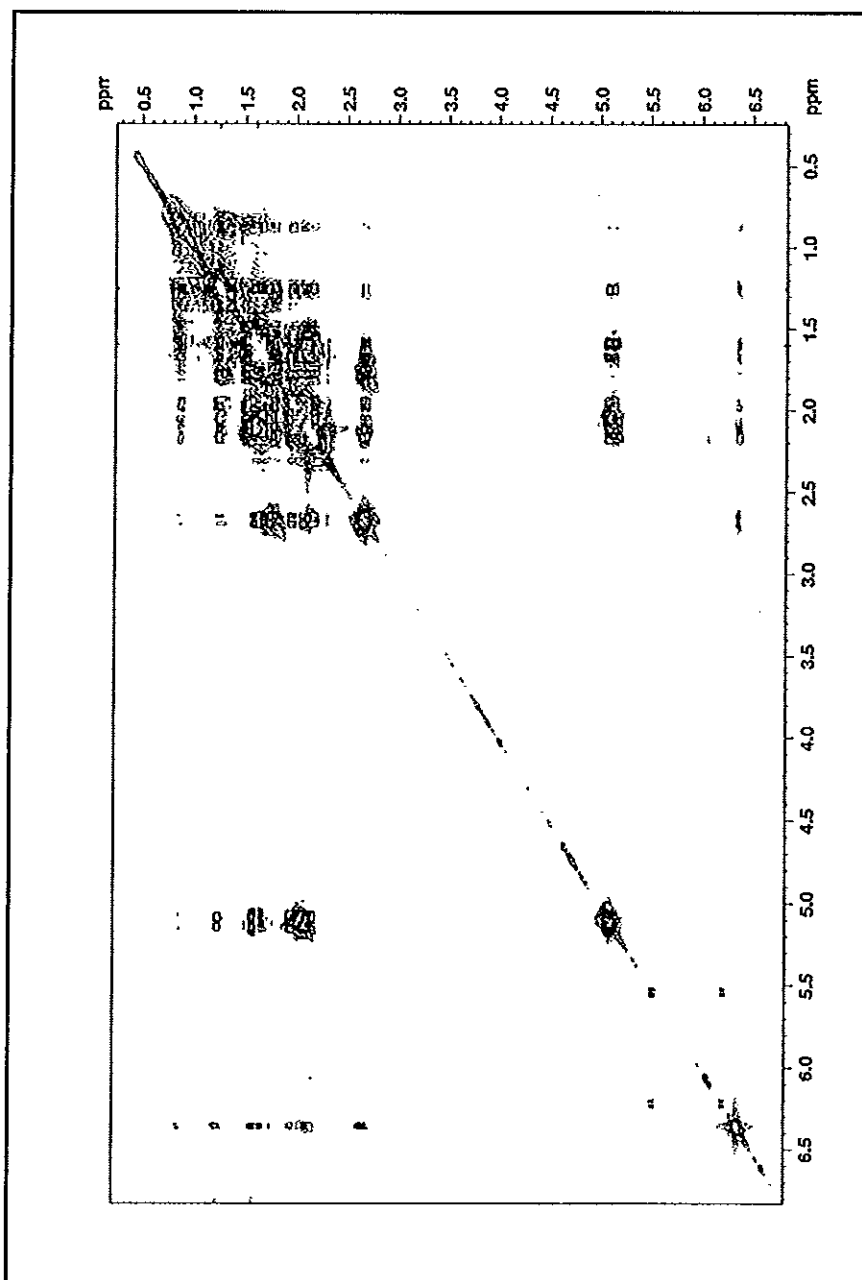


Figure 42  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound GP3

Figure 43 DEPT  $90^\circ$  and  $135^\circ$  spectra of compound GP3



Figure 44  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of compound GP3

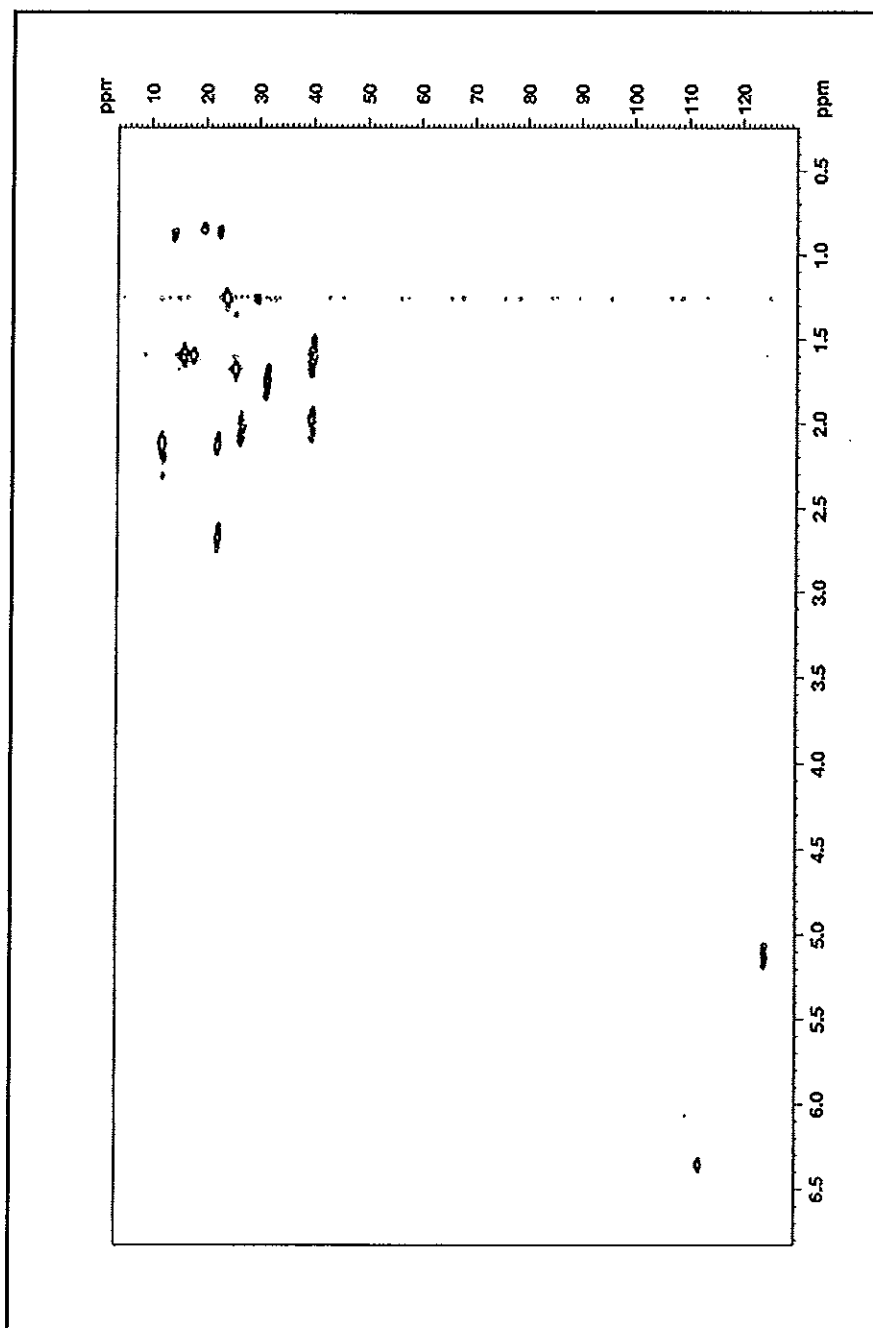


Figure 45 2D HMQC spectrum of compound GP3

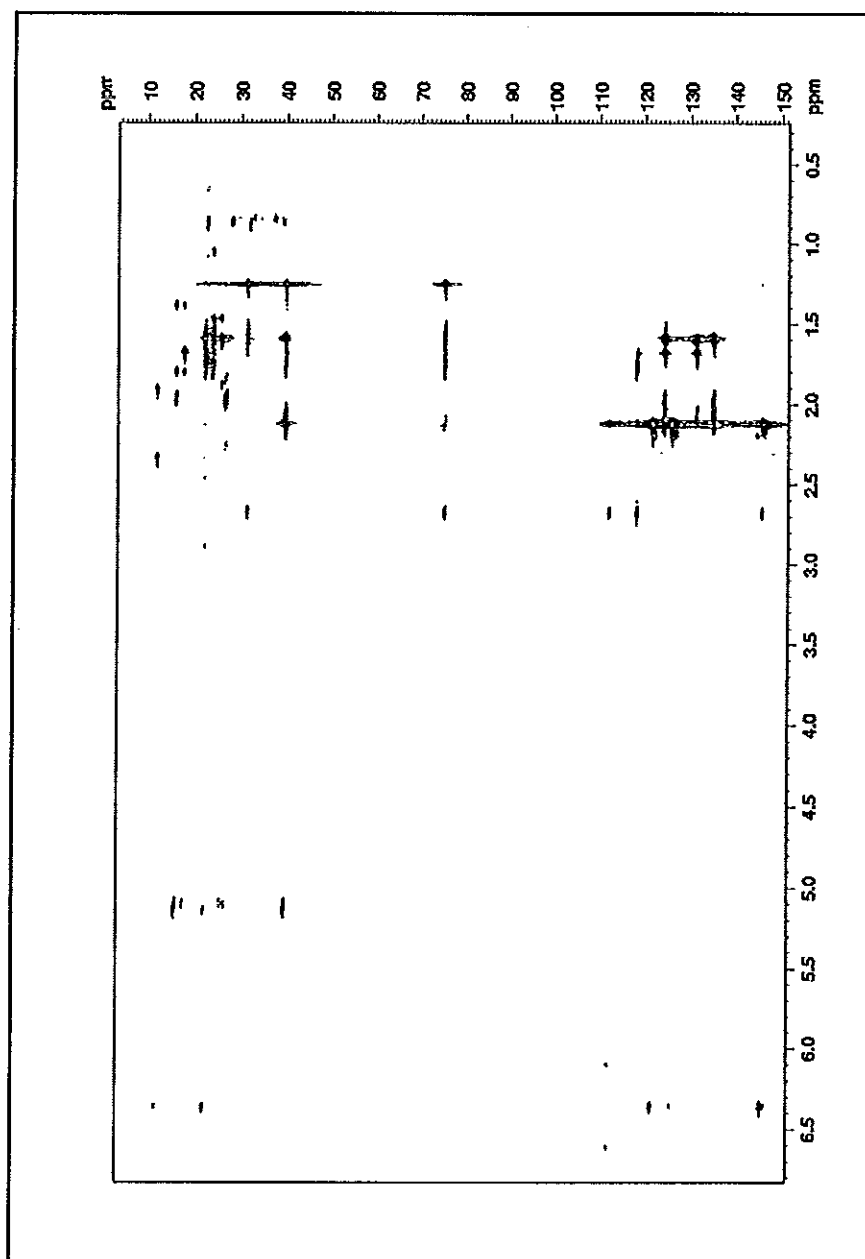


Figure 46 2D HMBC spectrum of compound GP3

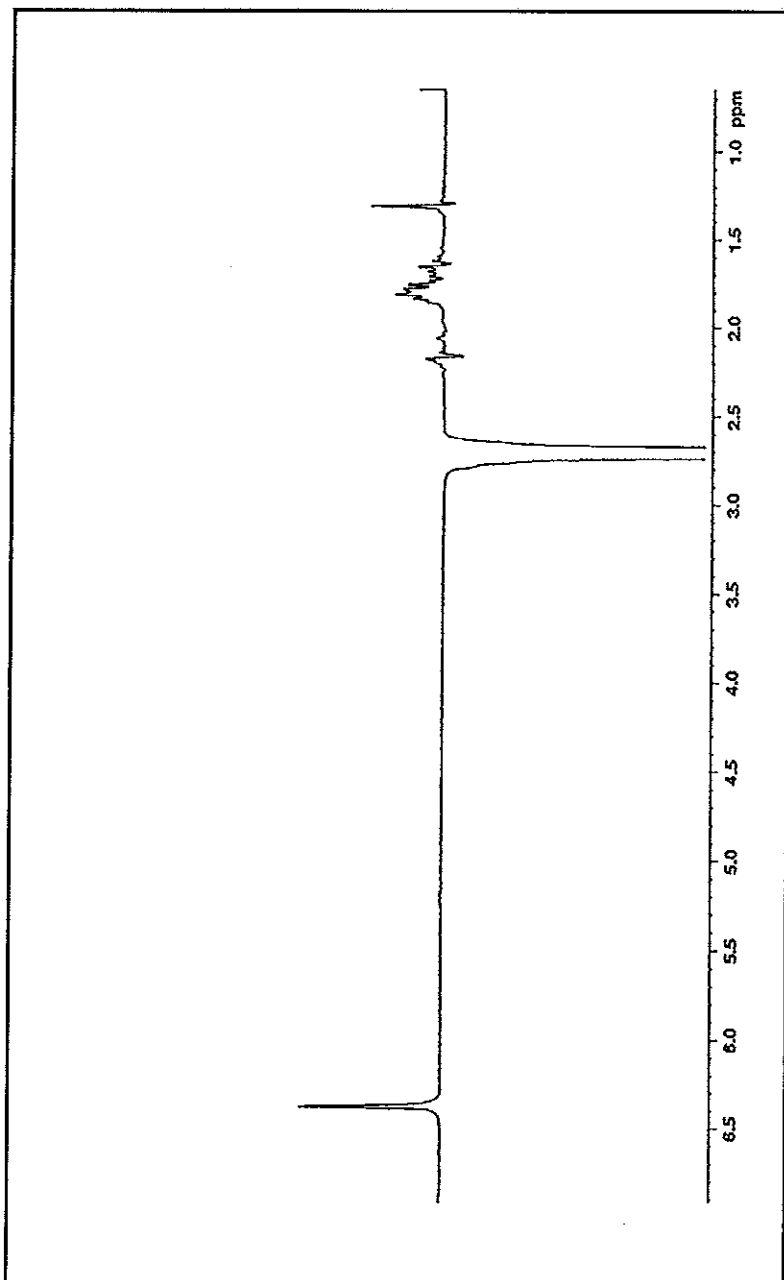


Figure 47 NOE difference spectrum of compound GP3 after irradiation at  $\delta_H$  2.67 (H-4)

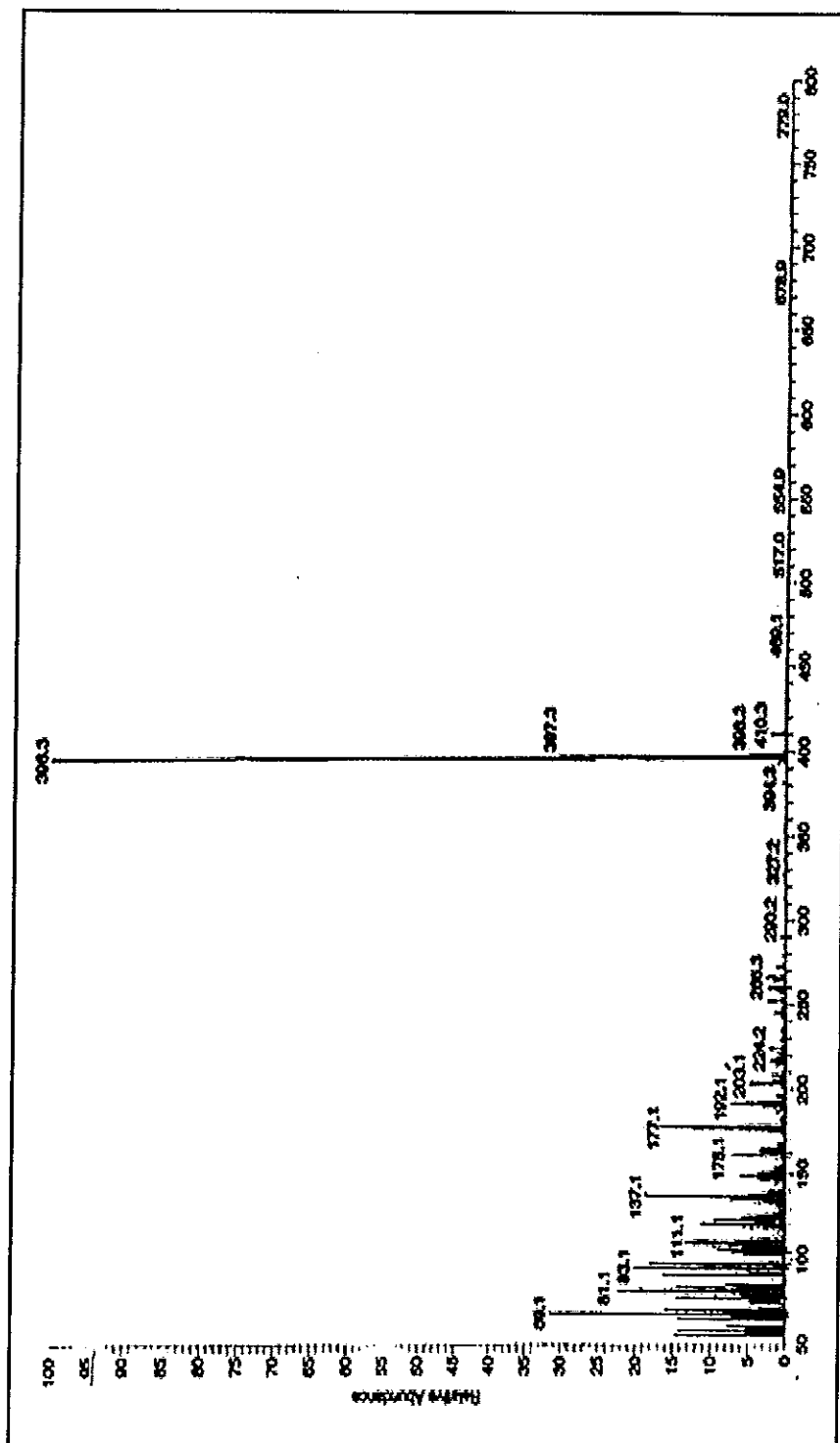


Figure 48 Mass spectrum of compound GP5

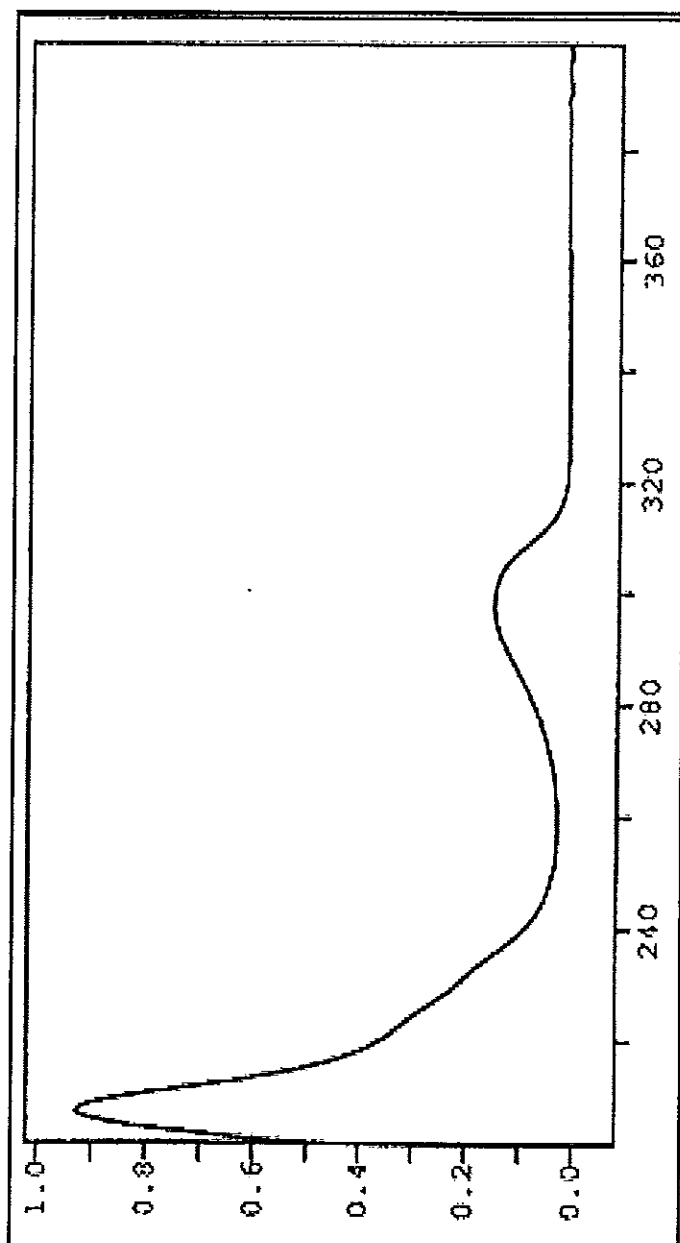


Figure 49 UV (MeOH) spectrum of compound GP5

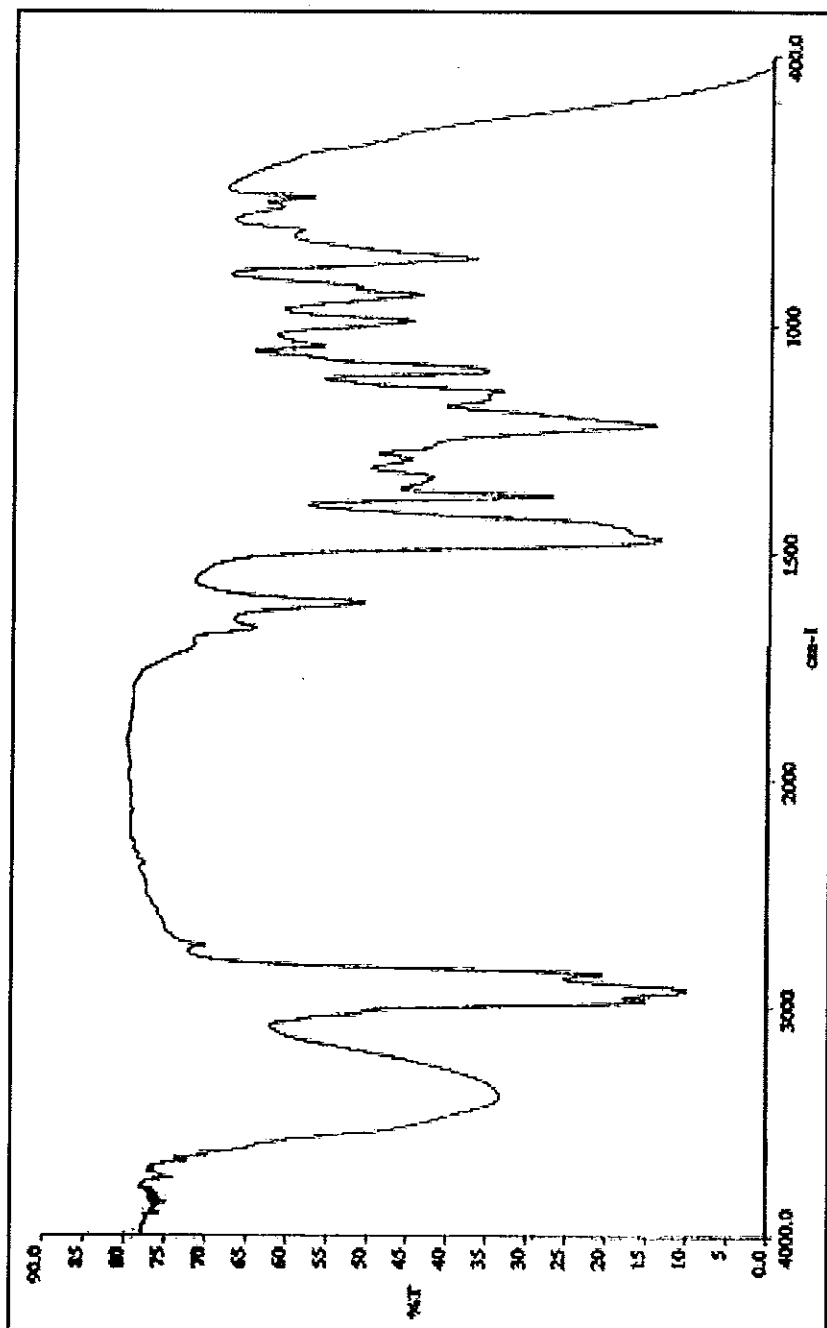


Figure 50 FT-IR (neat) spectrum of compound GP5

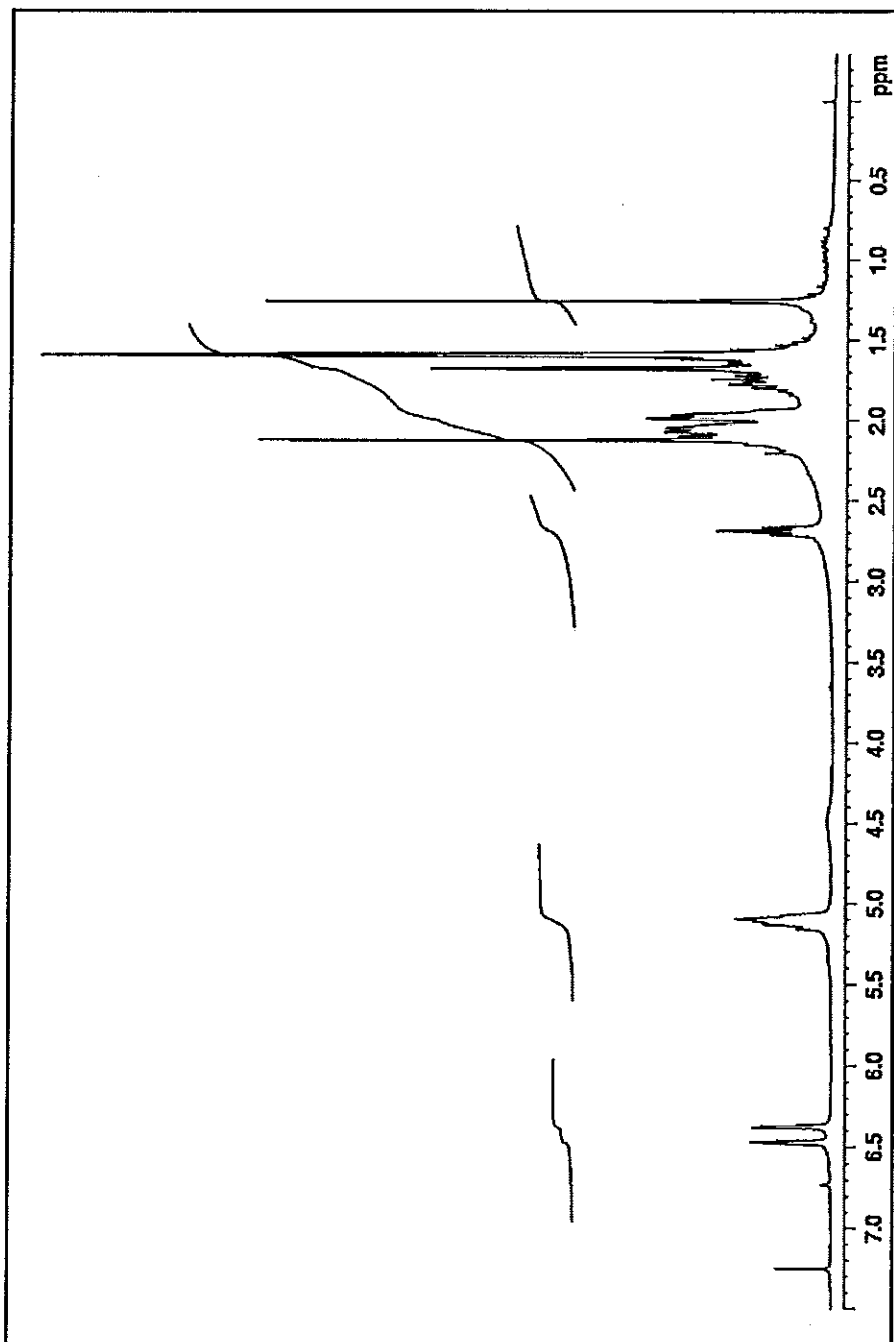


Figure 51  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound GP5



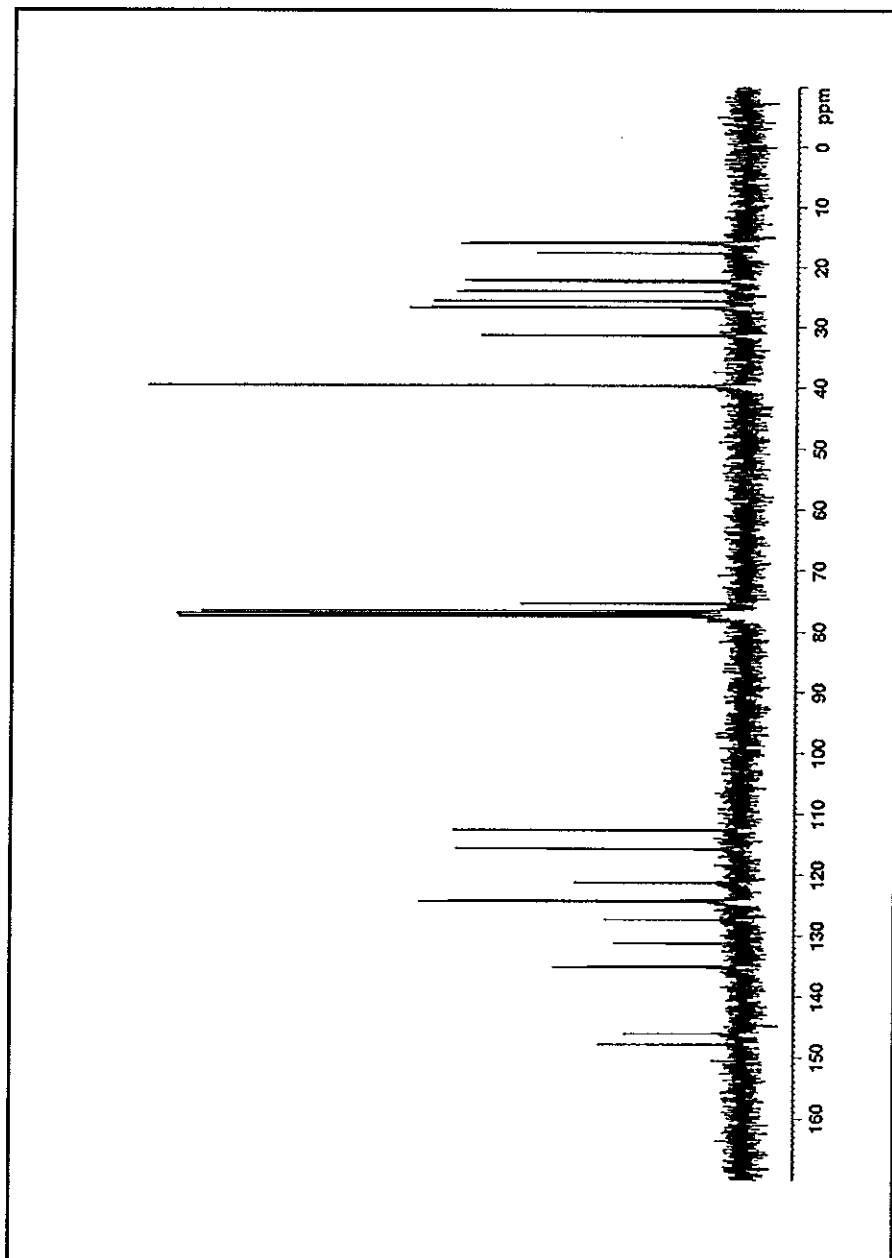


Figure 52  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound GP5

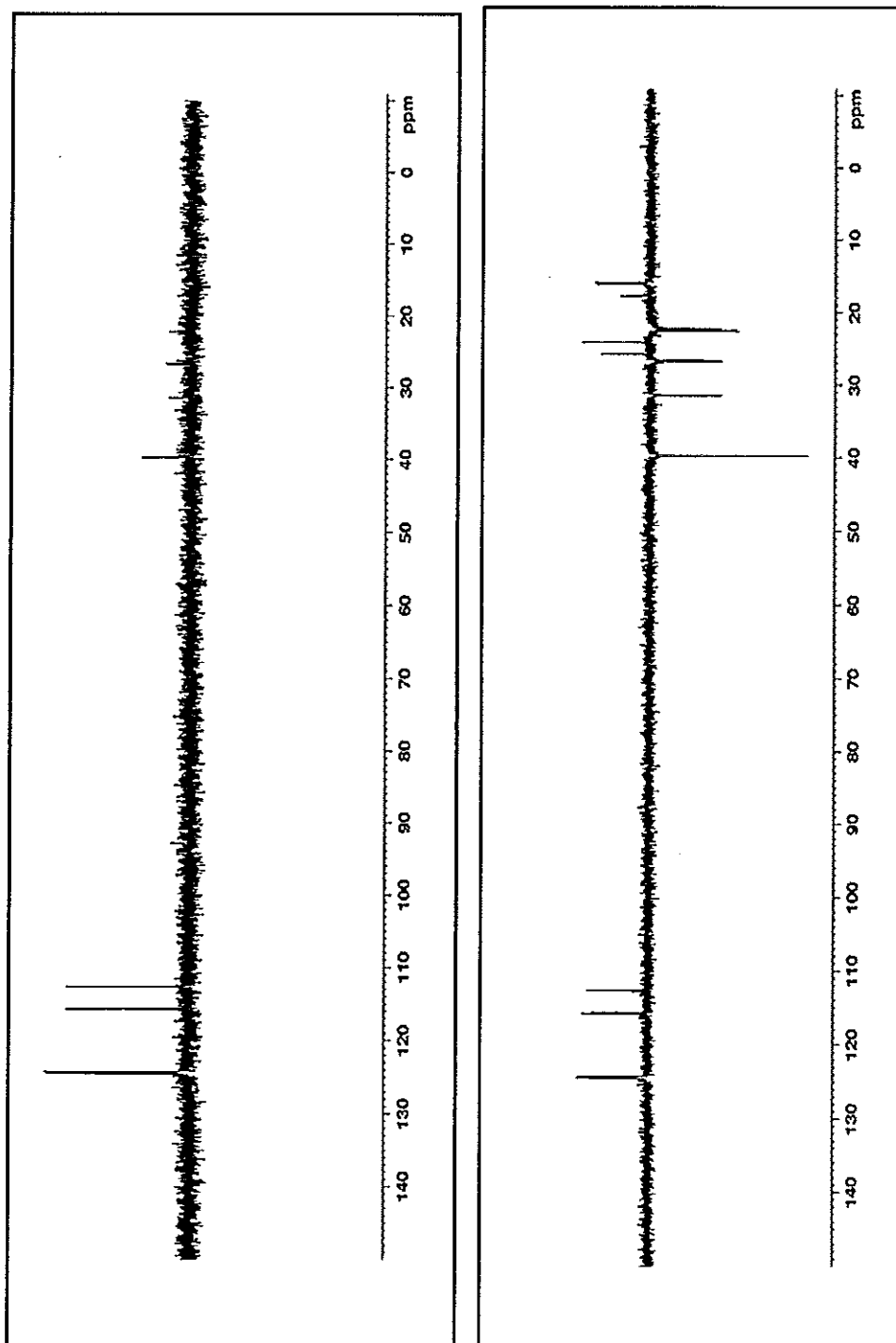


Figure 53 DEPT  $90^\circ$  and  $135^\circ$  spectra of compound GP5

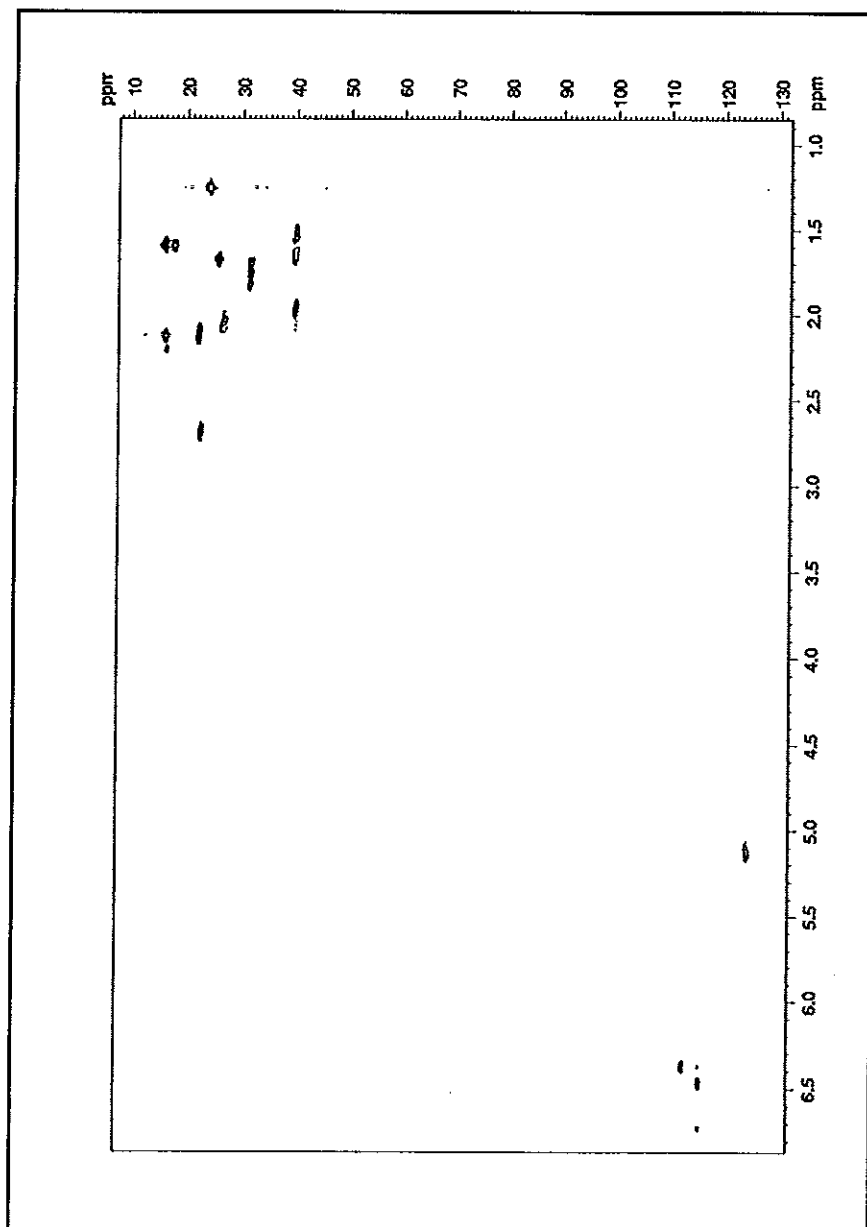


Figure 54 2D HMQC spectrum of compound GP5

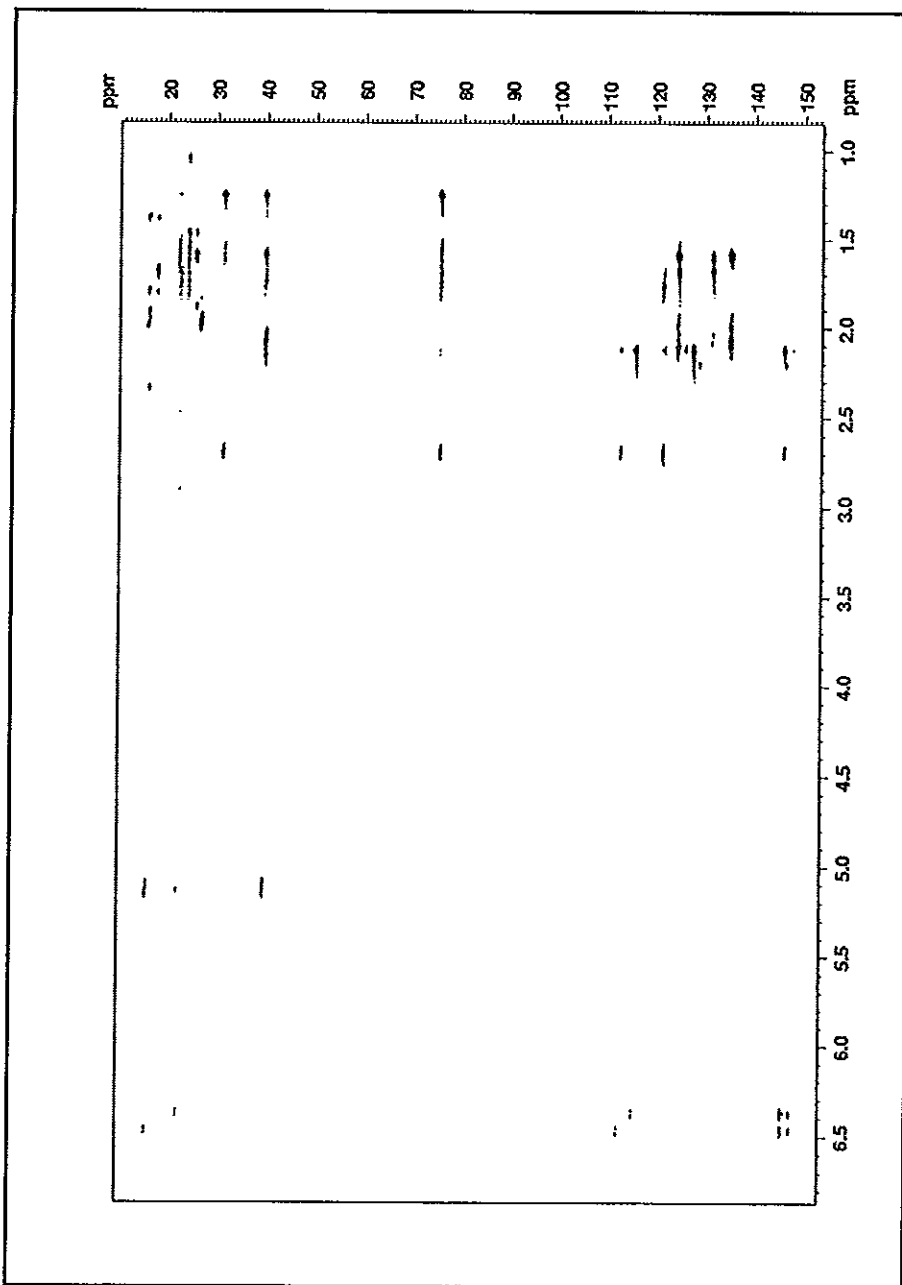


Figure 55 2D HMBC spectrum of compound GP5

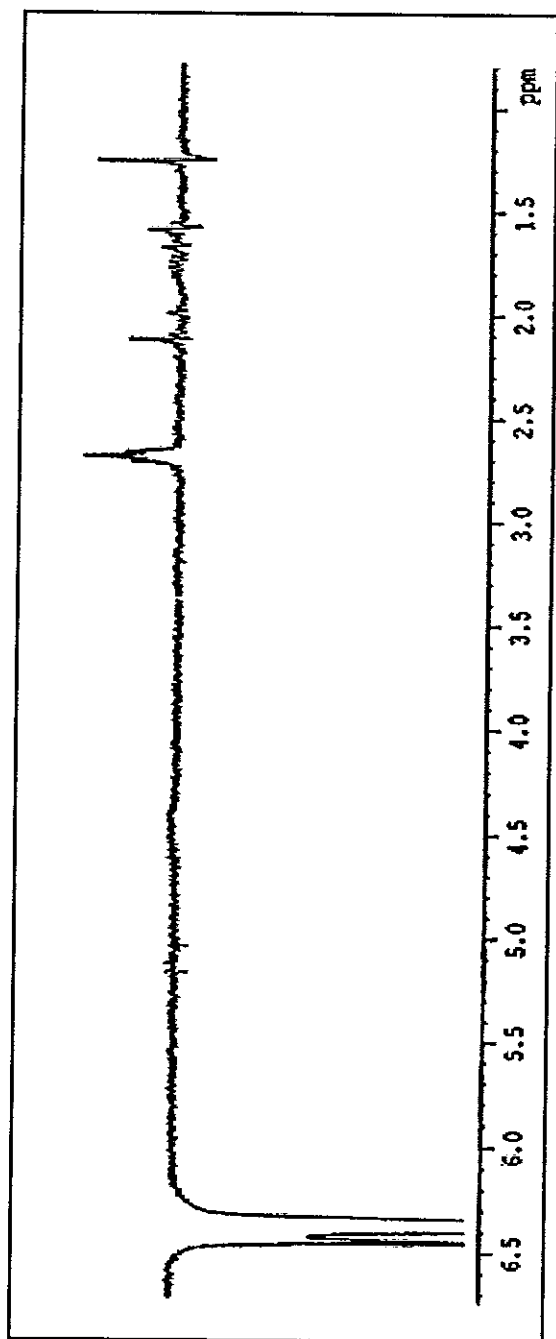


Figure 56 NOE difference spectrum of compound GP5 after irradiation at  $\delta_{\text{H}} 6.37$  (H-5)

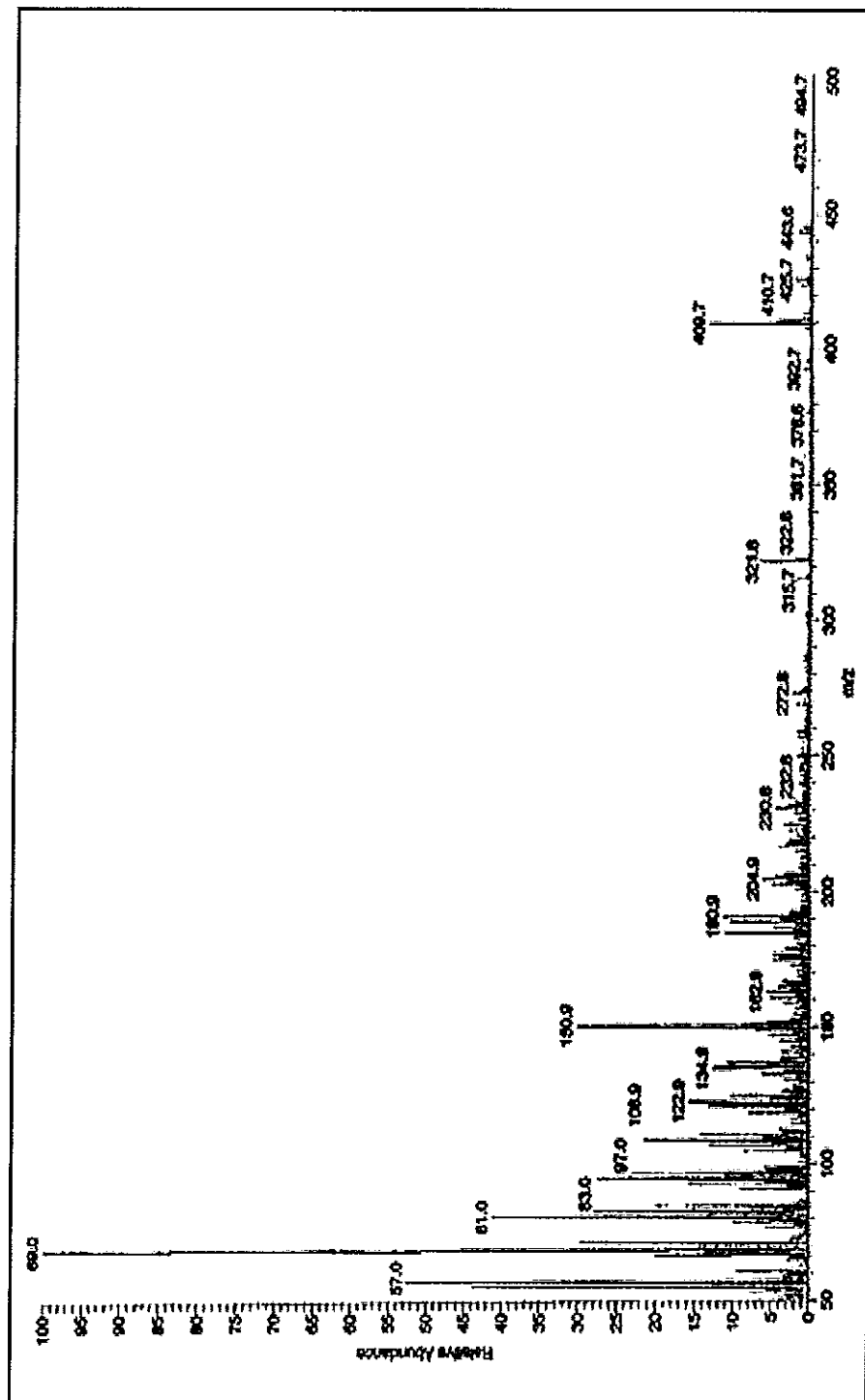


Figure 57 Mass spectrum of compound GP4

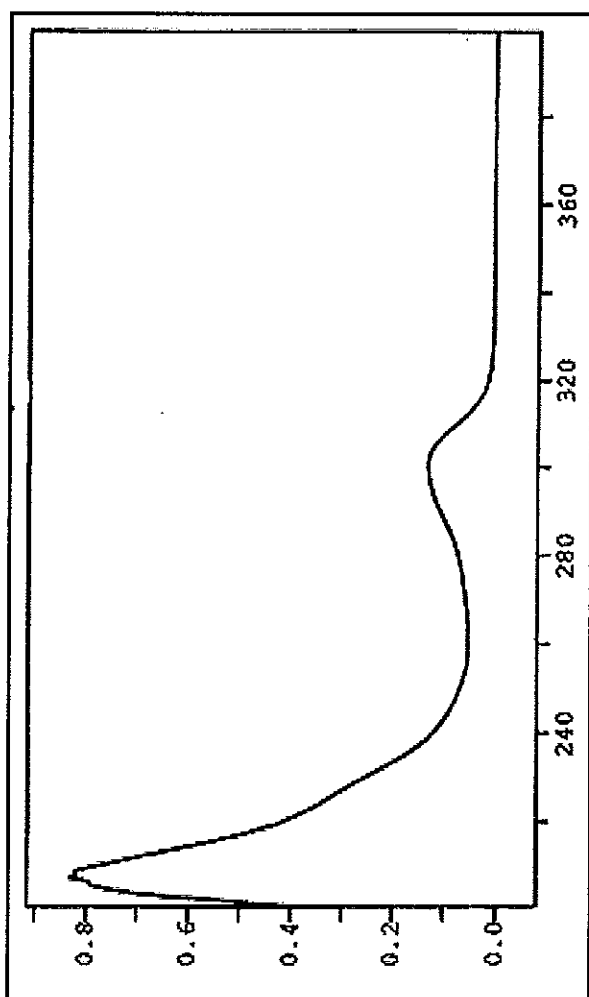


Figure 58 UV (MeOH) spectrum of compound GP4

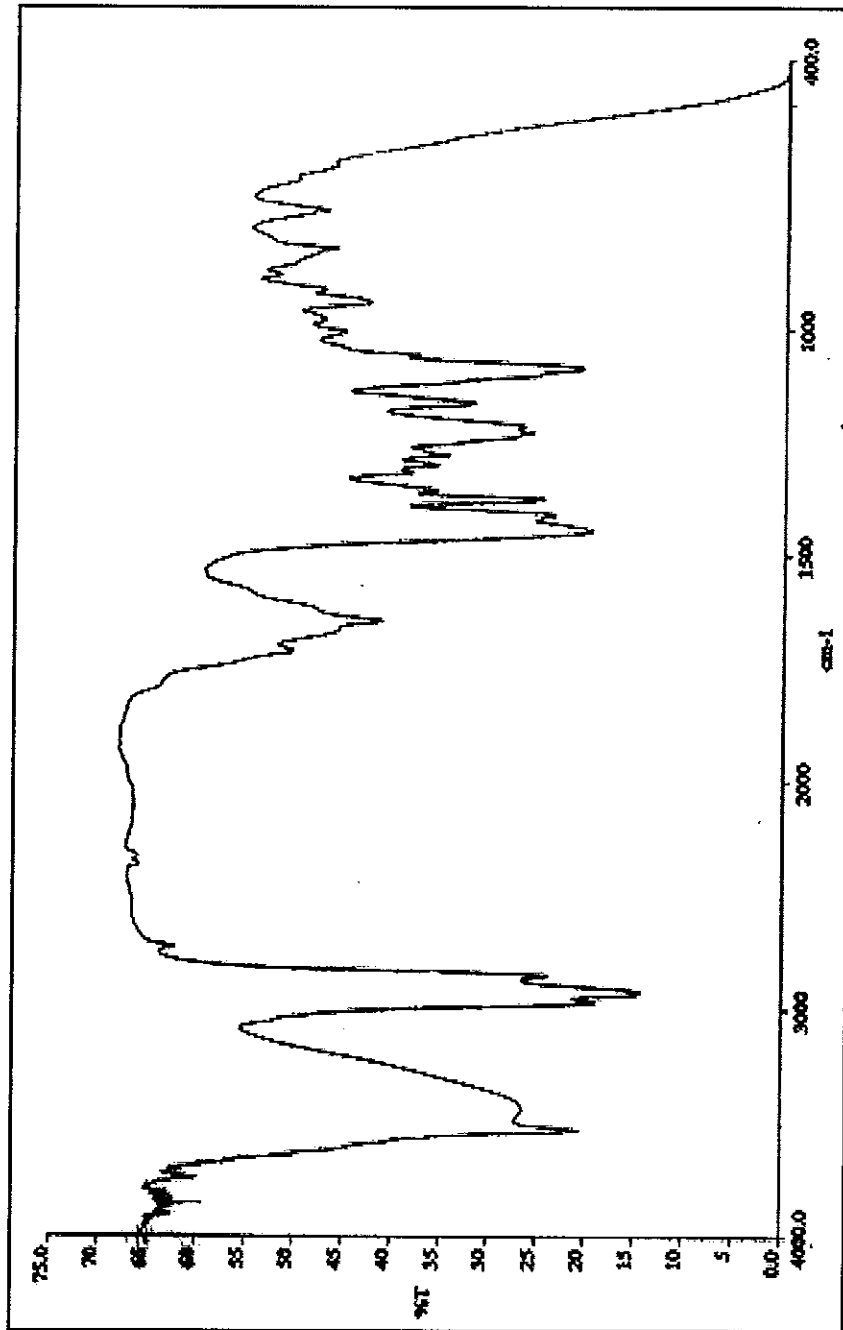


Figure 59 FT-IR (neat) spectrum of compound GP4



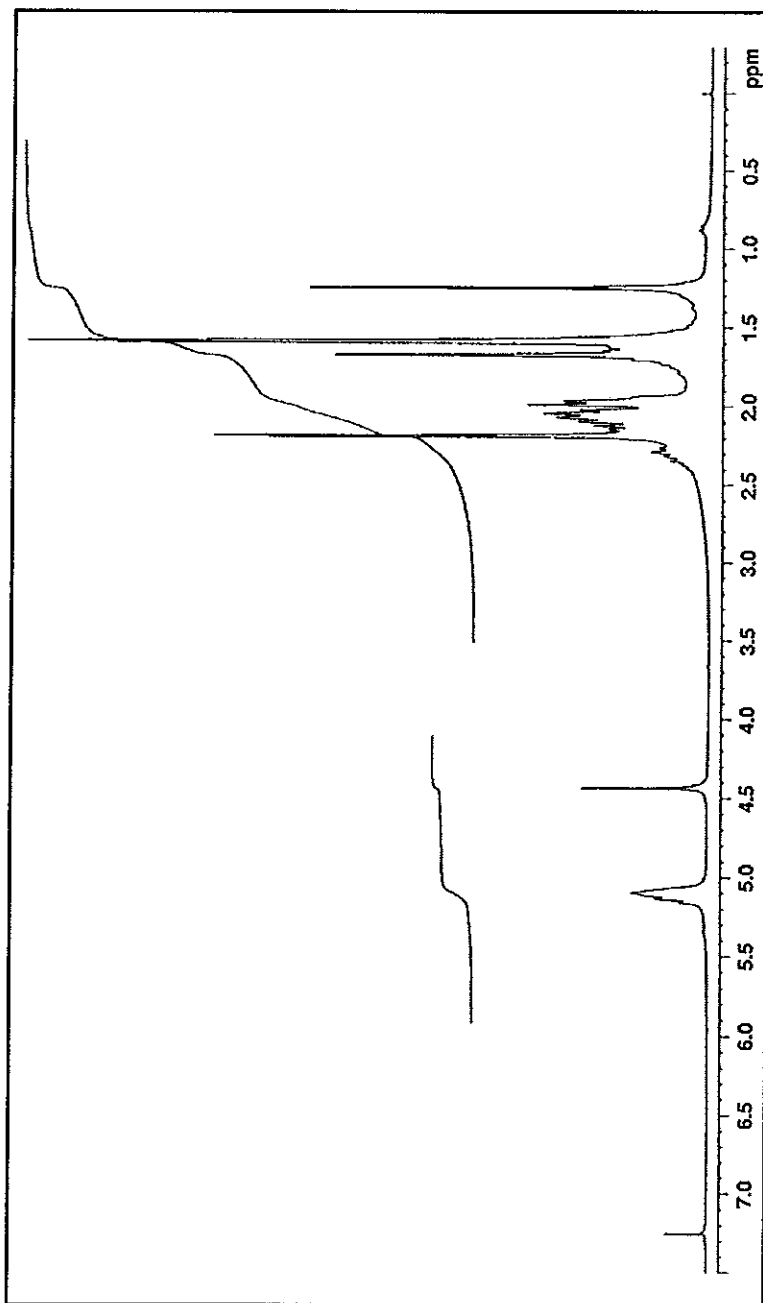


Figure 60  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound GP4

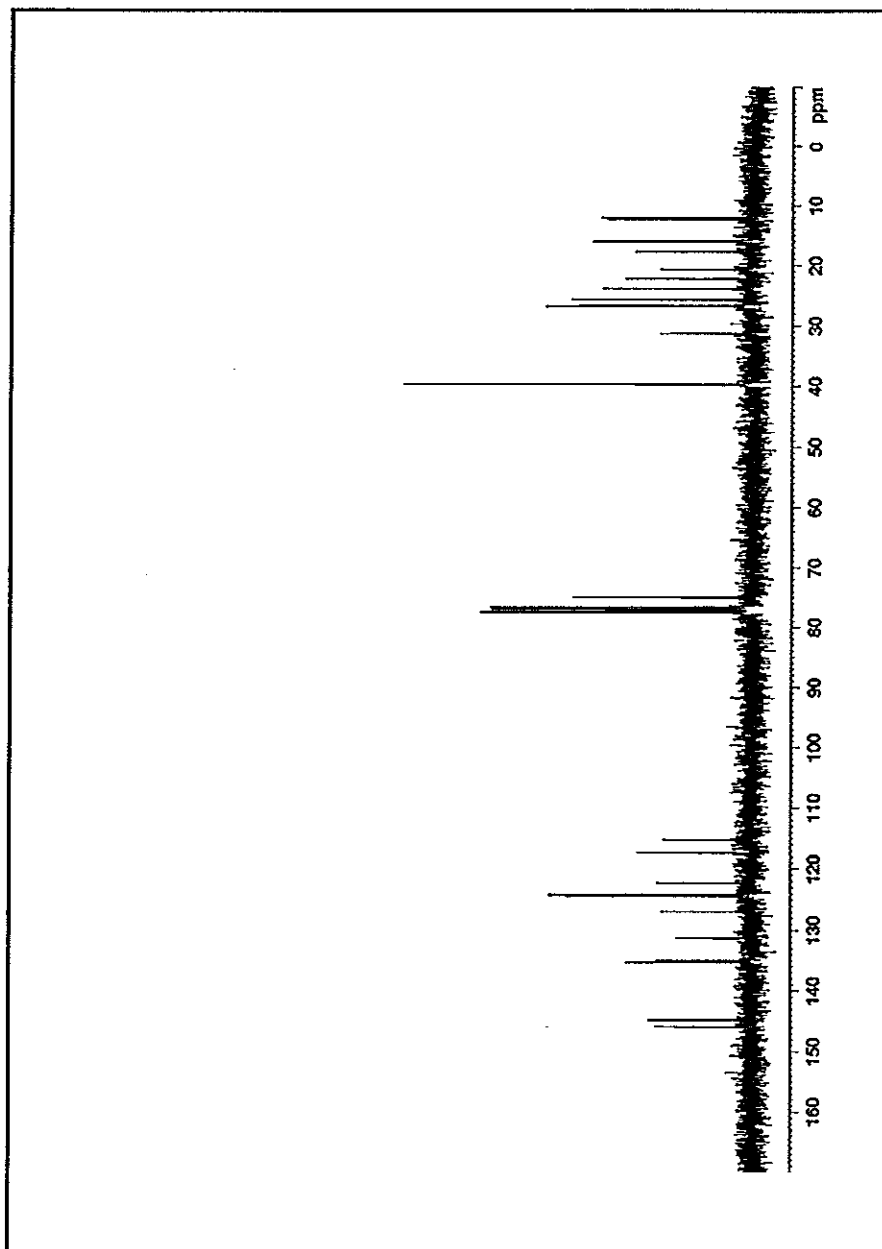


Figure 61  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound GP4

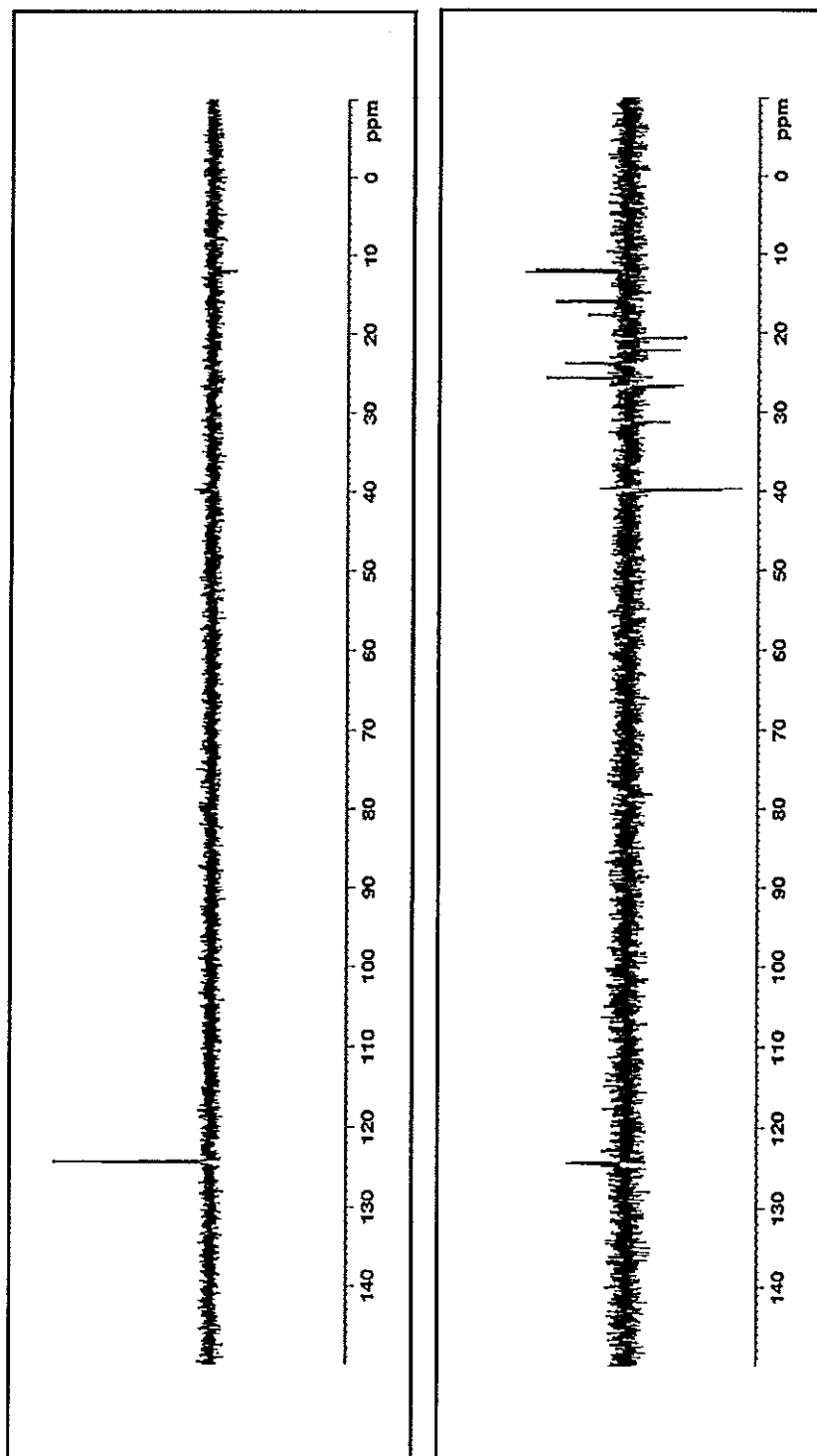


Figure 62 DEPT 90° and 135° spectra of compound GP4

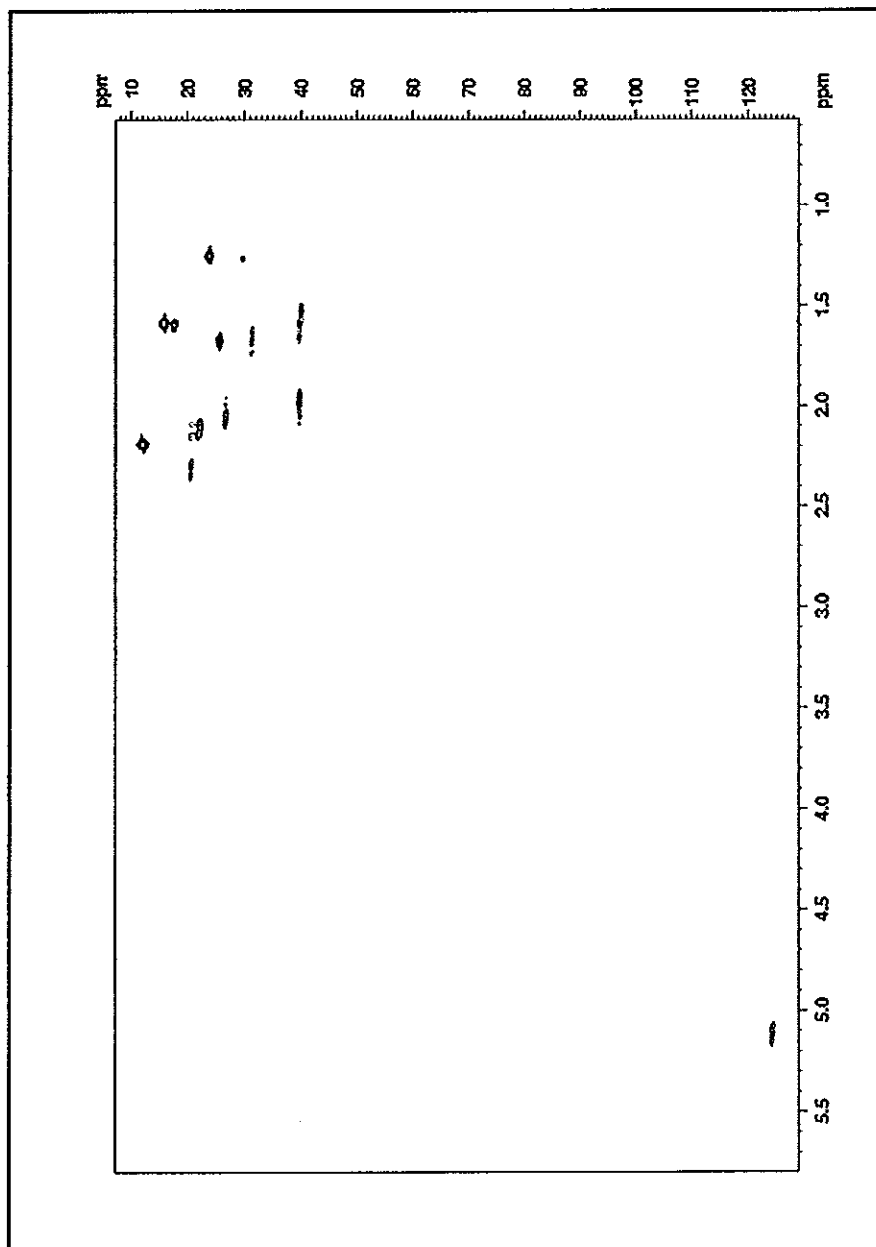


Figure 63 2D HMQC spectrum of compound GP4

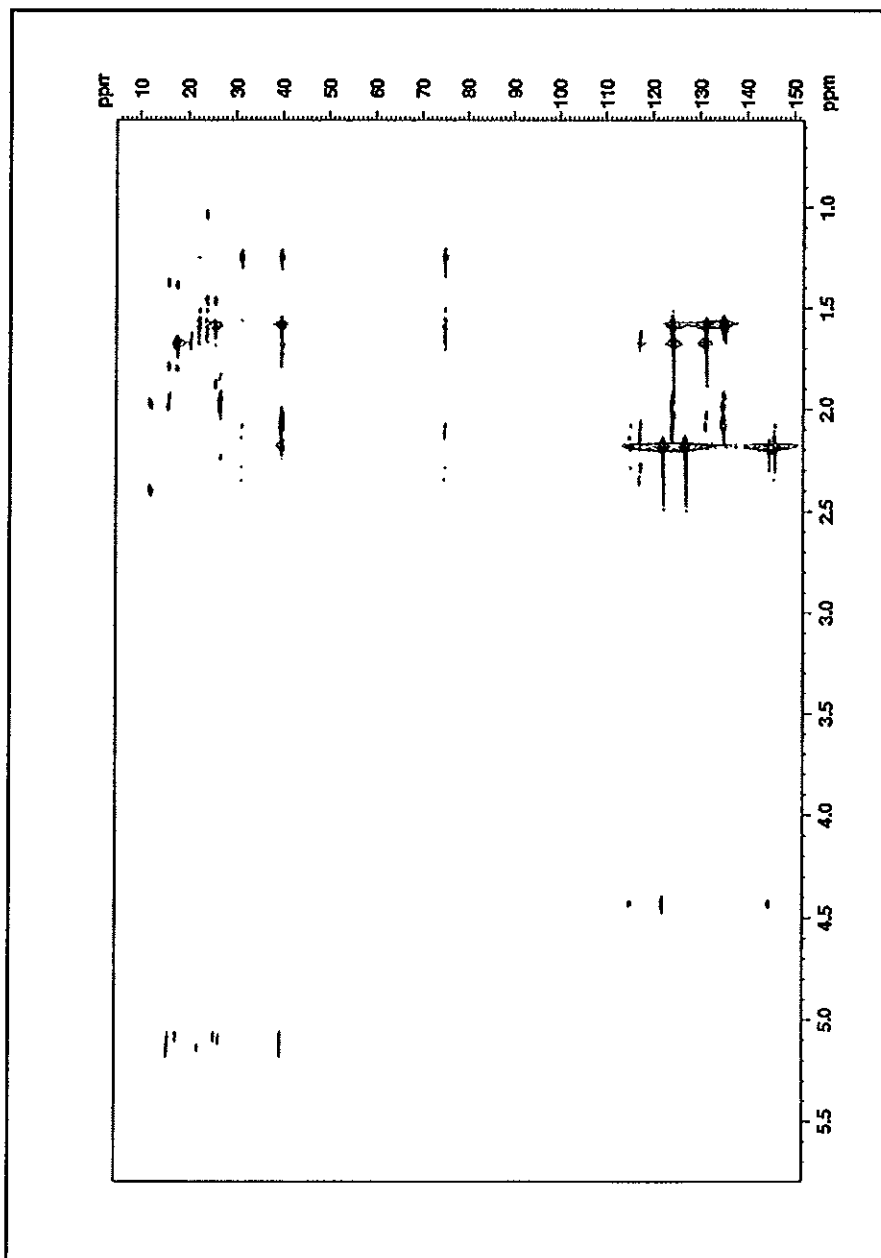


Figure 64 2D HMBC spectrum of compound GP4

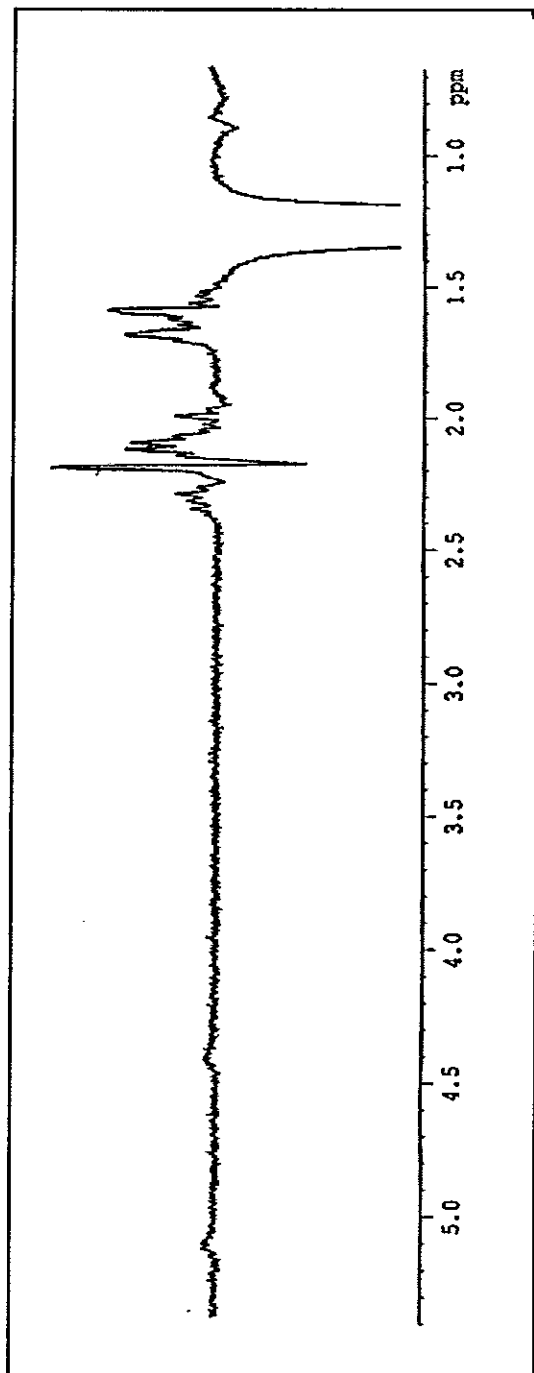


Figure 65 NOE difference spectrum of compound GP4 after irradiation at  $\delta_{\text{H}} 1.26$  ( $\text{H}_3\text{-25}$ )

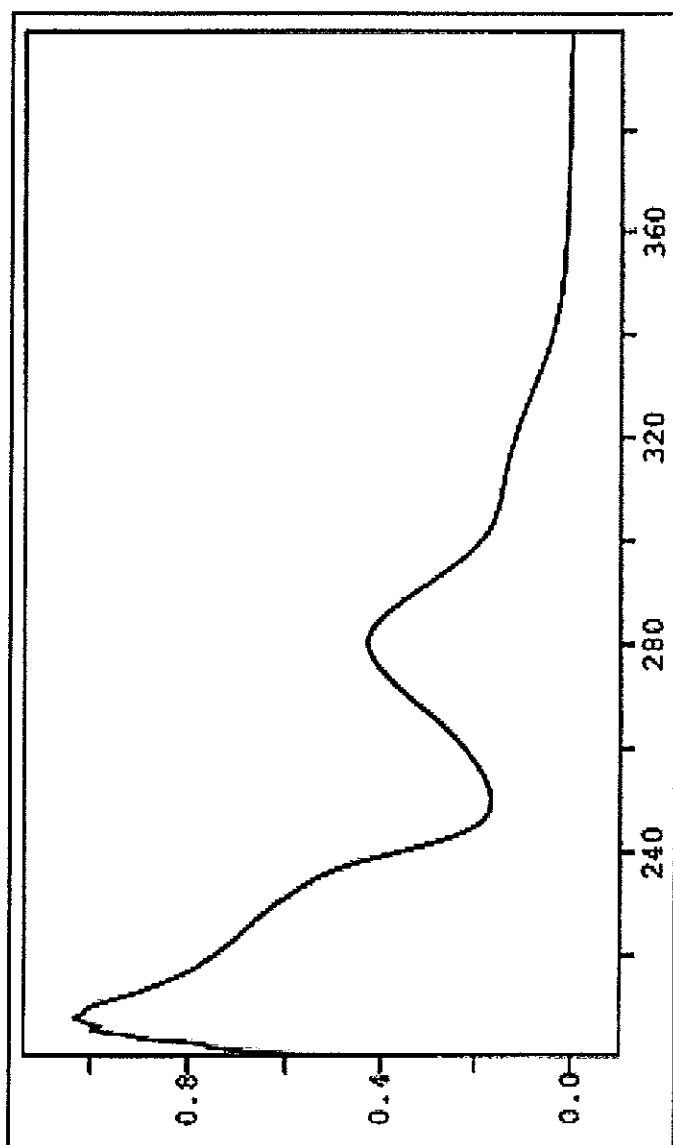


Figure 66 UV (MeOH) spectrum of compound GP18

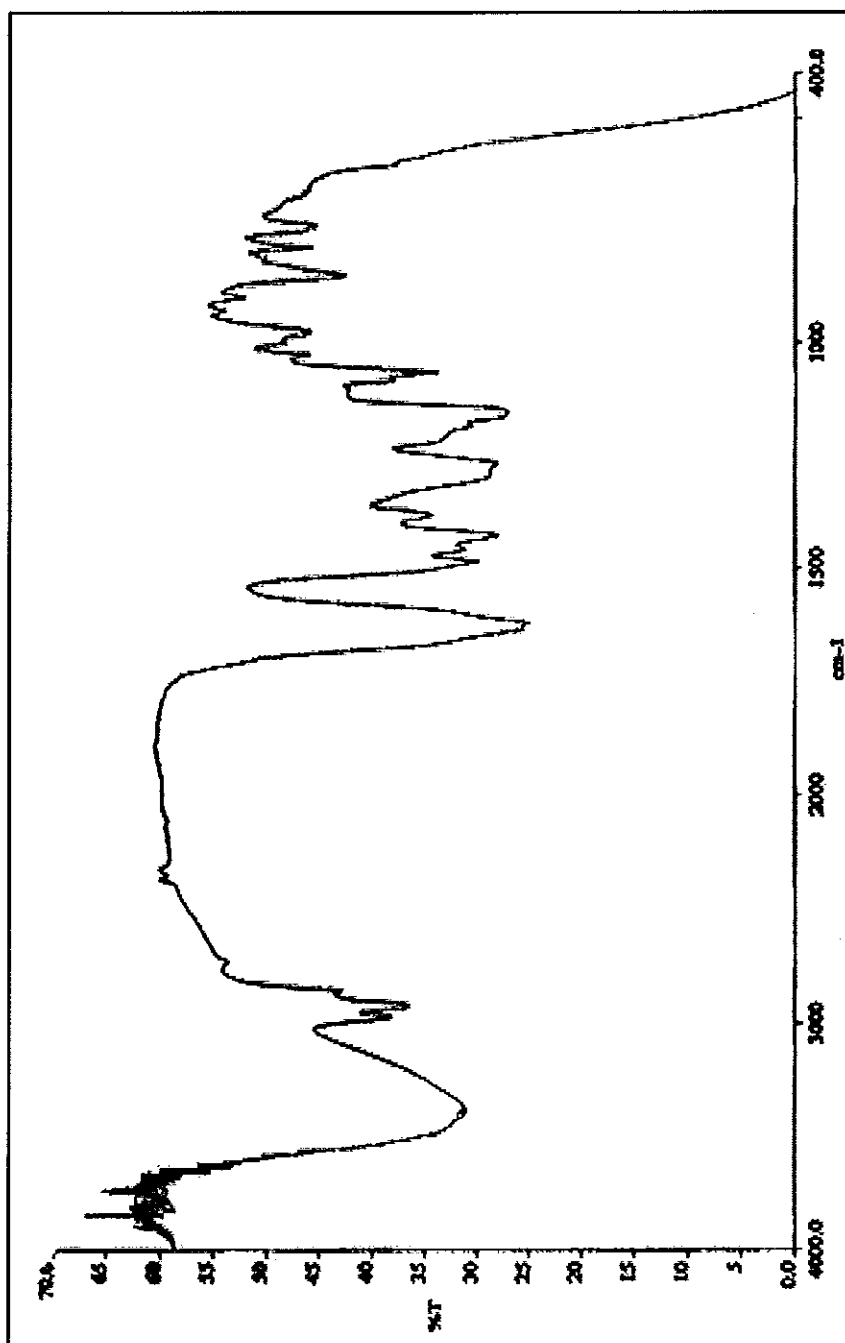


Figure 67 FT-IR (neat) spectrum of compound GP18



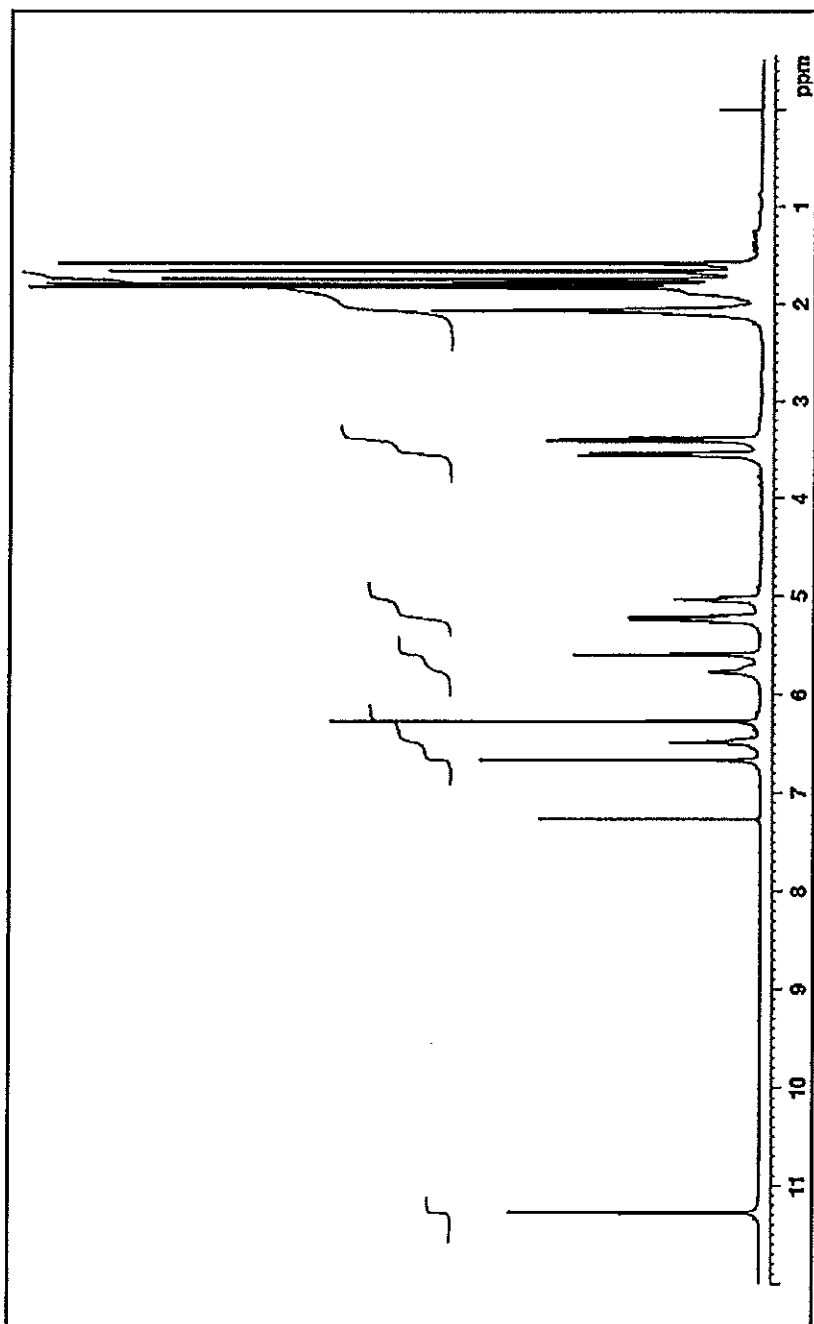


Figure 68  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound GP18

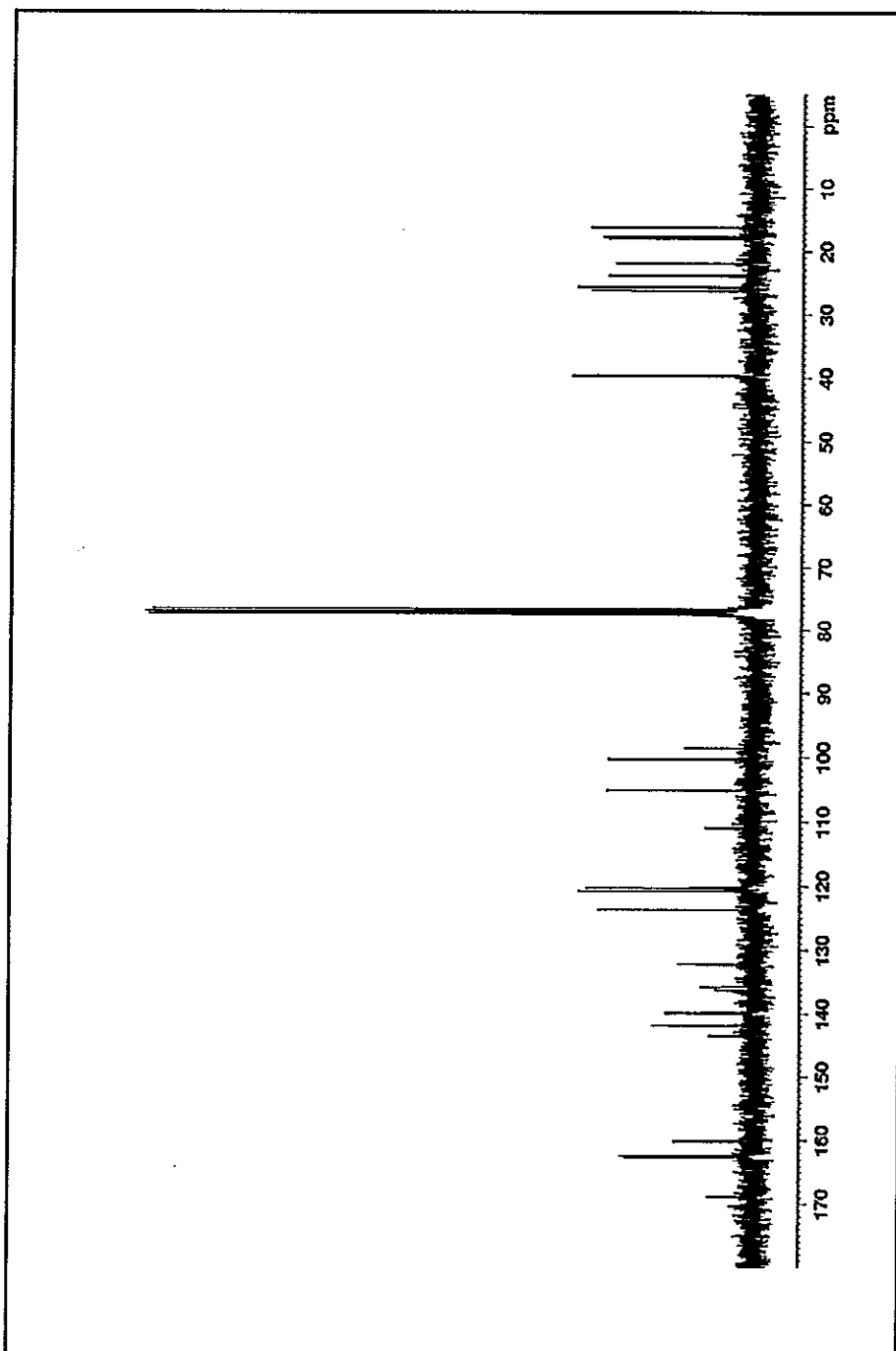


Figure 69  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound GP18

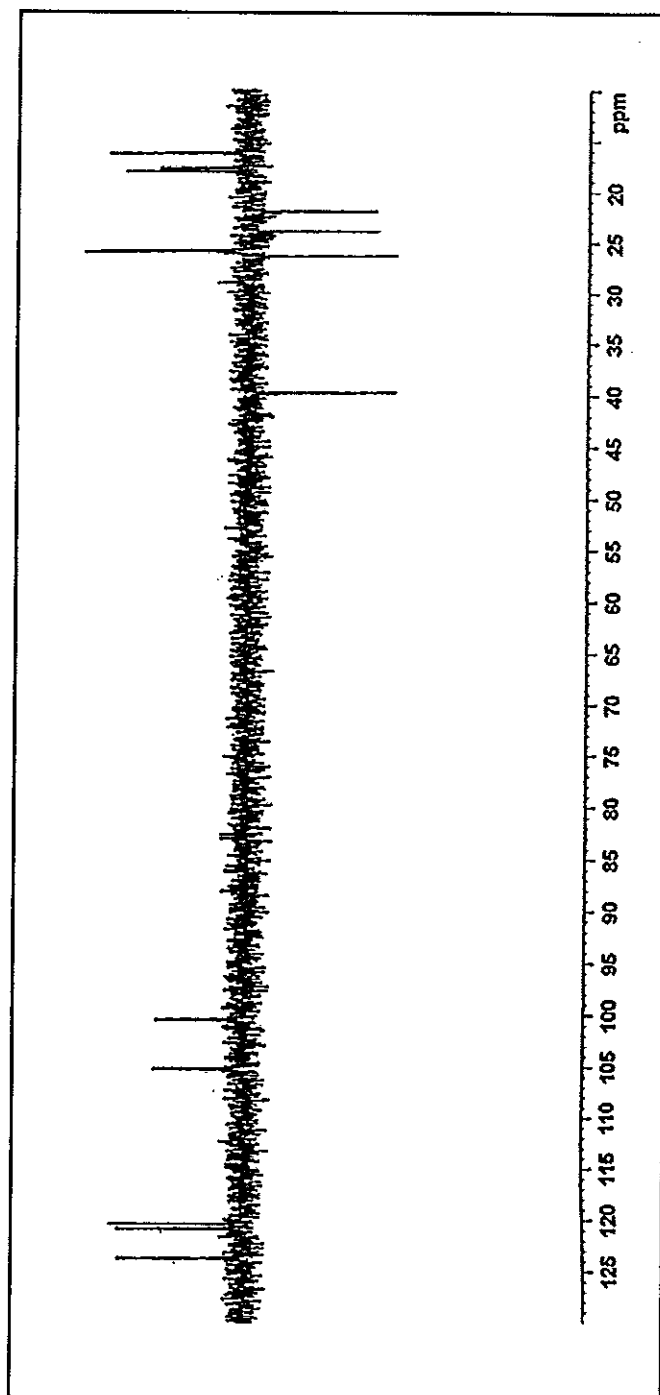


Figure 70 DEPT 135° spectra of compound GP18

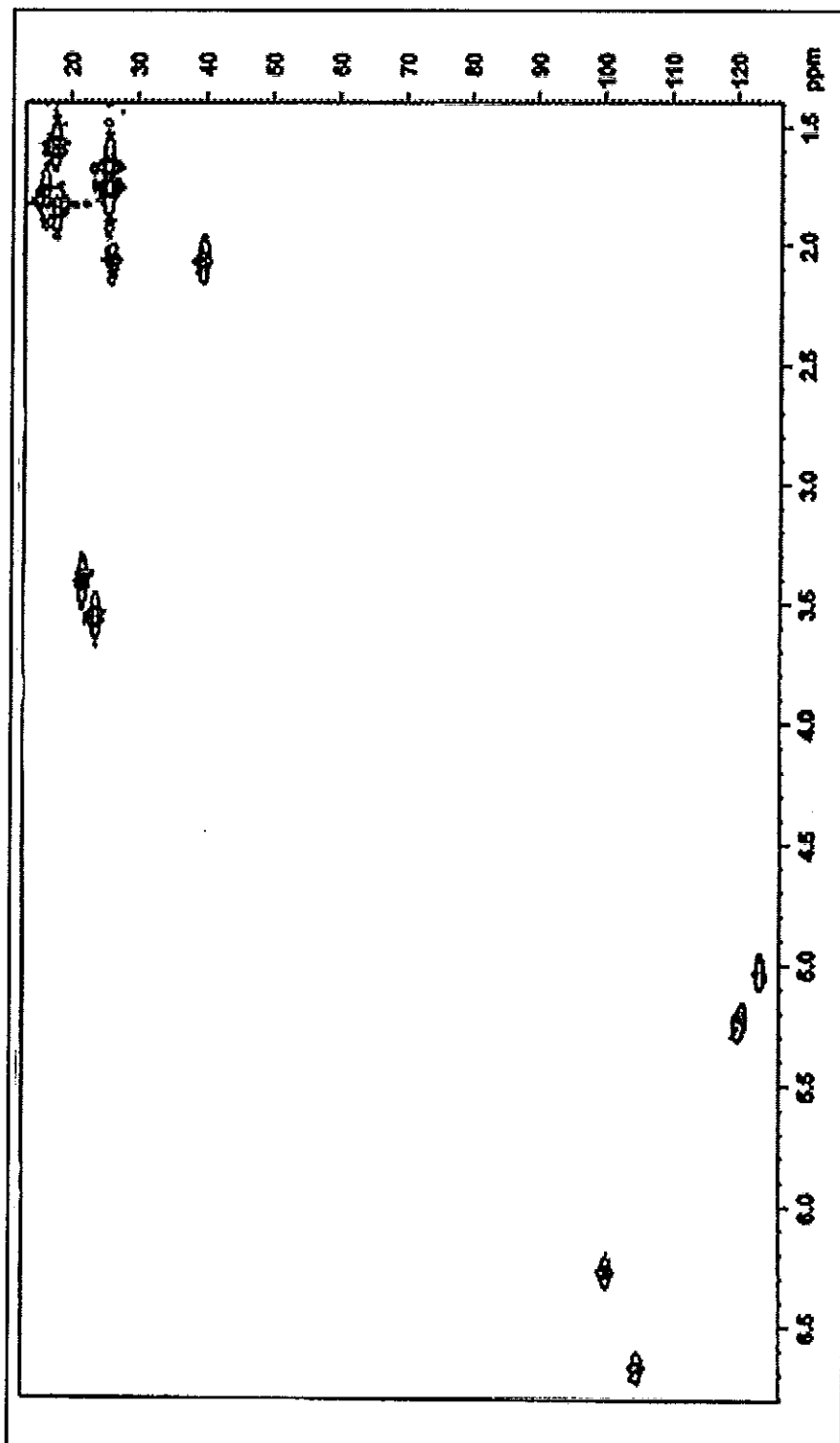


Figure 71 2D HMQC spectrum of compound GP18

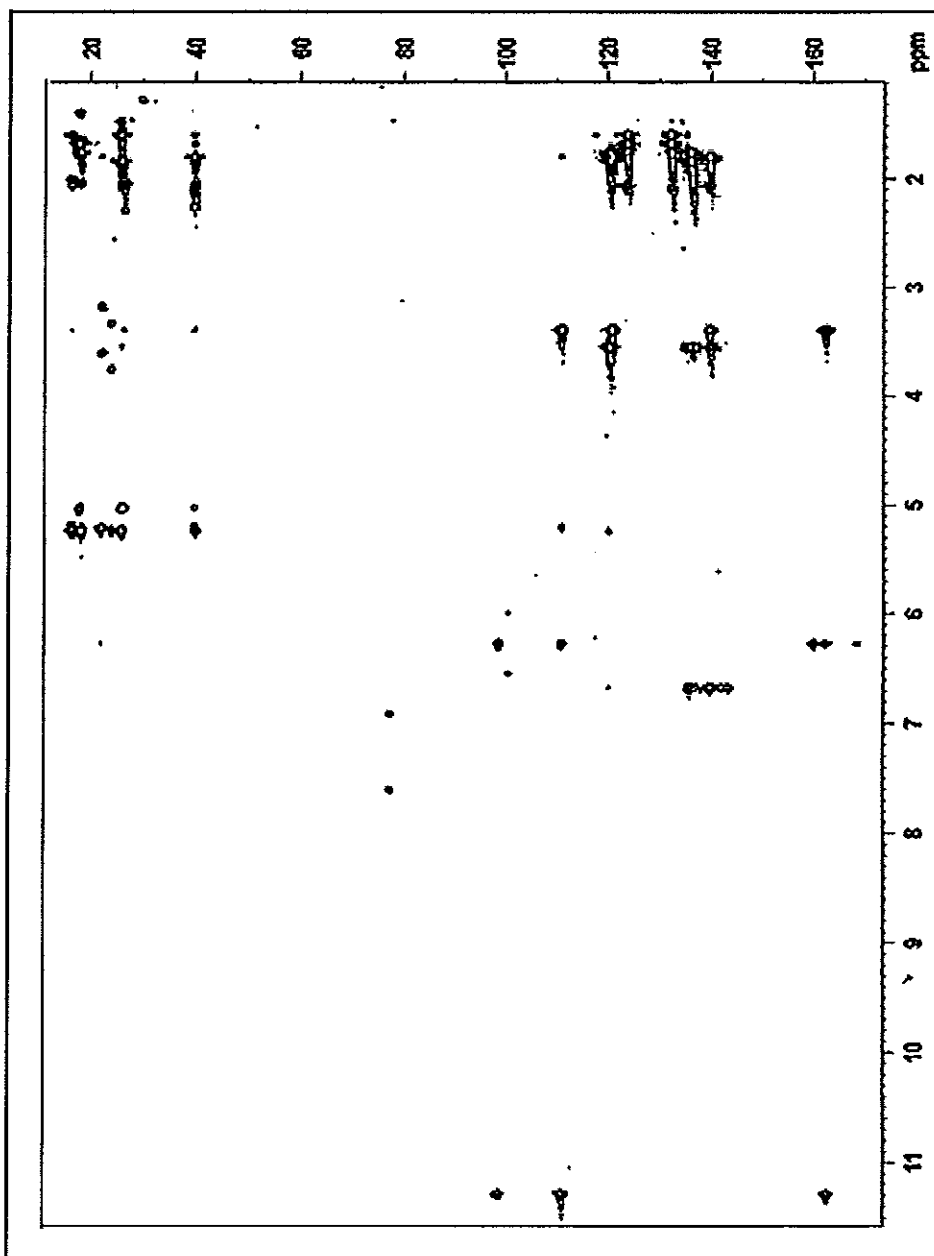


Figure 72 2D HMBC spectrum of compound GP18

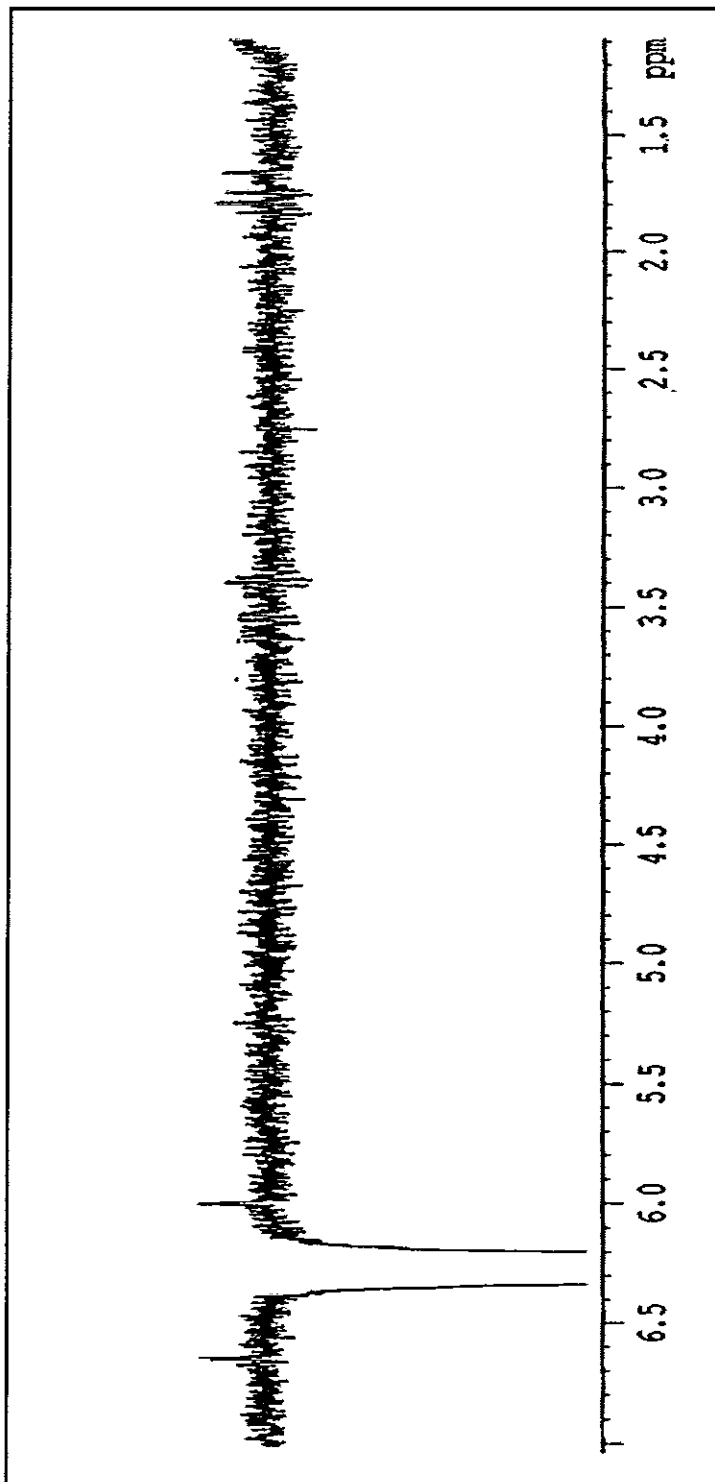


Figure 73 NOE difference spectrum of compound GP18 after irradiation at  $\delta_{\text{H}} 6.27$  (H-4)

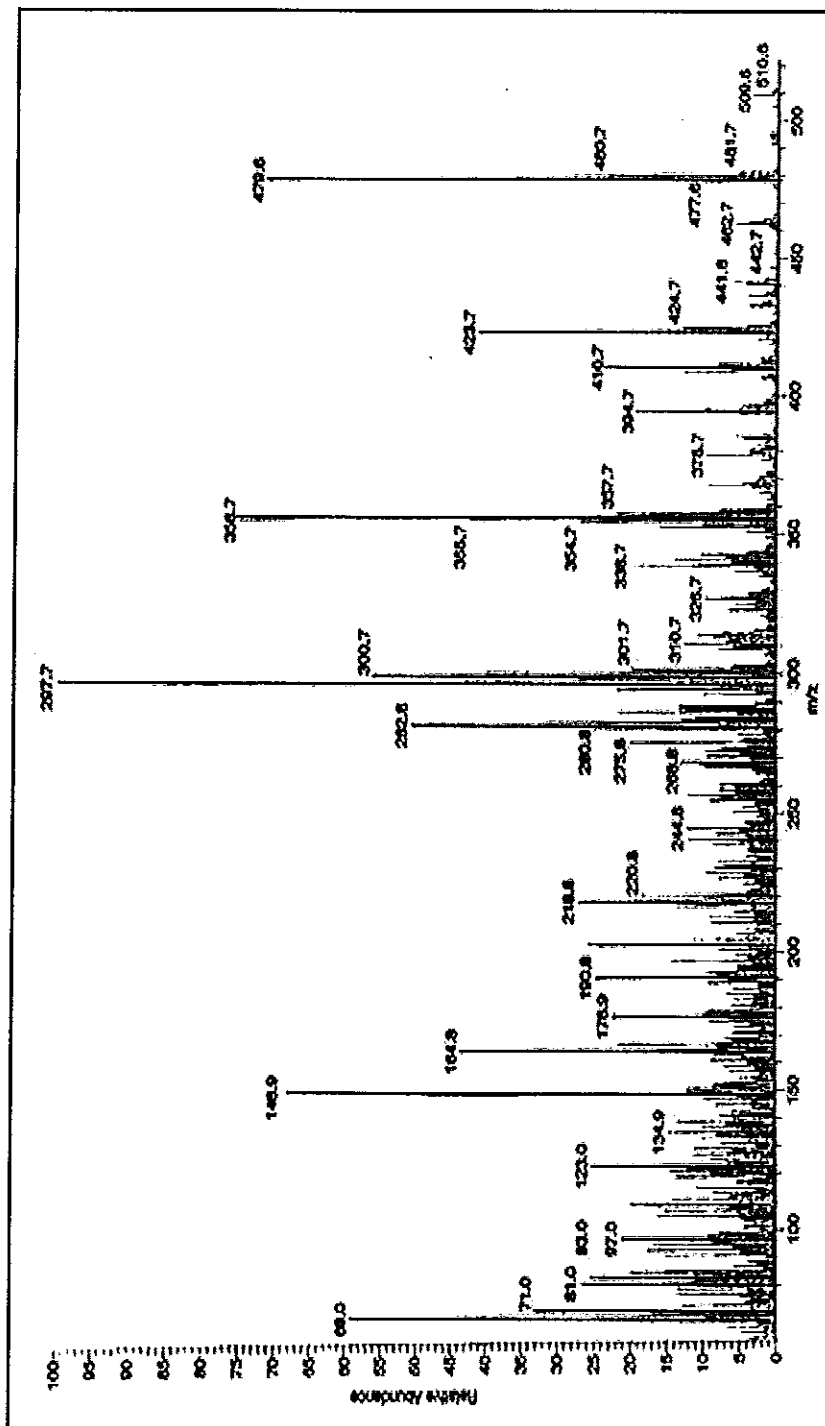


Figure 74 Mass spectrum of compound GP14

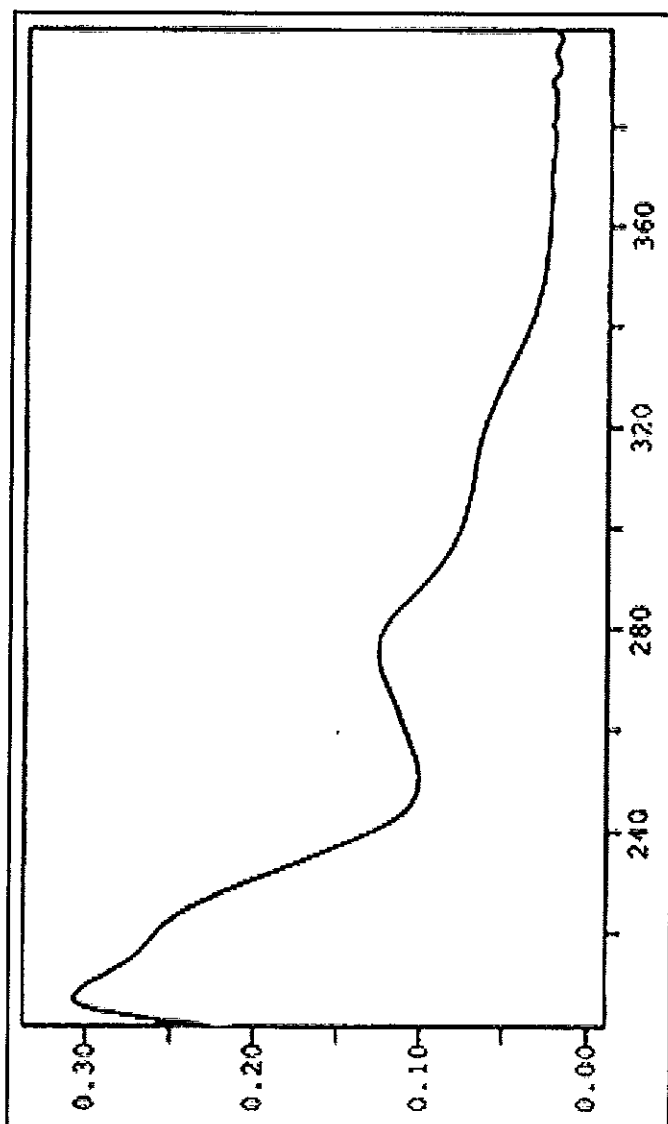


Figure 75 UV (MeOH) spectrum of compound GP14



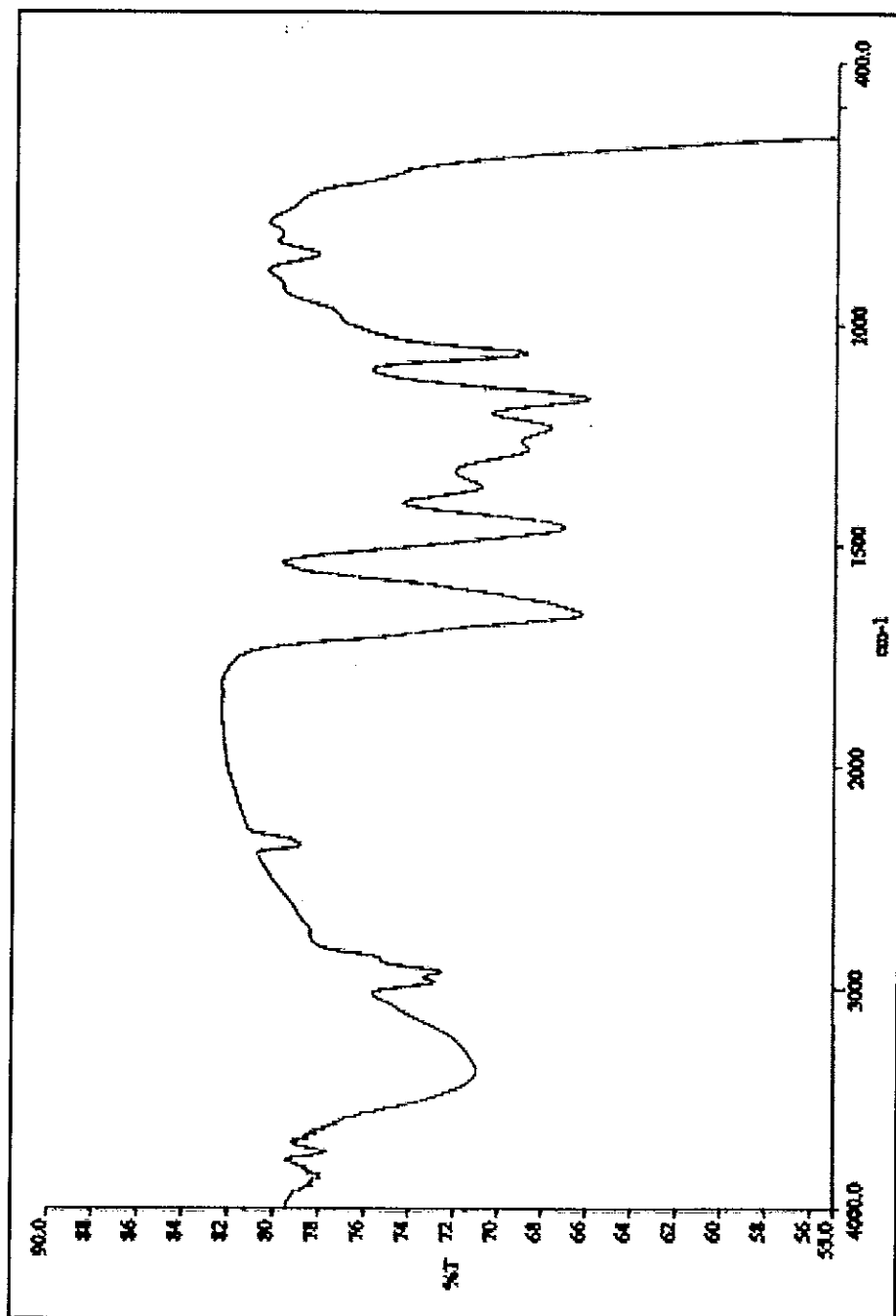


Figure 76 FT-IR (neat) spectrum of compound GP14

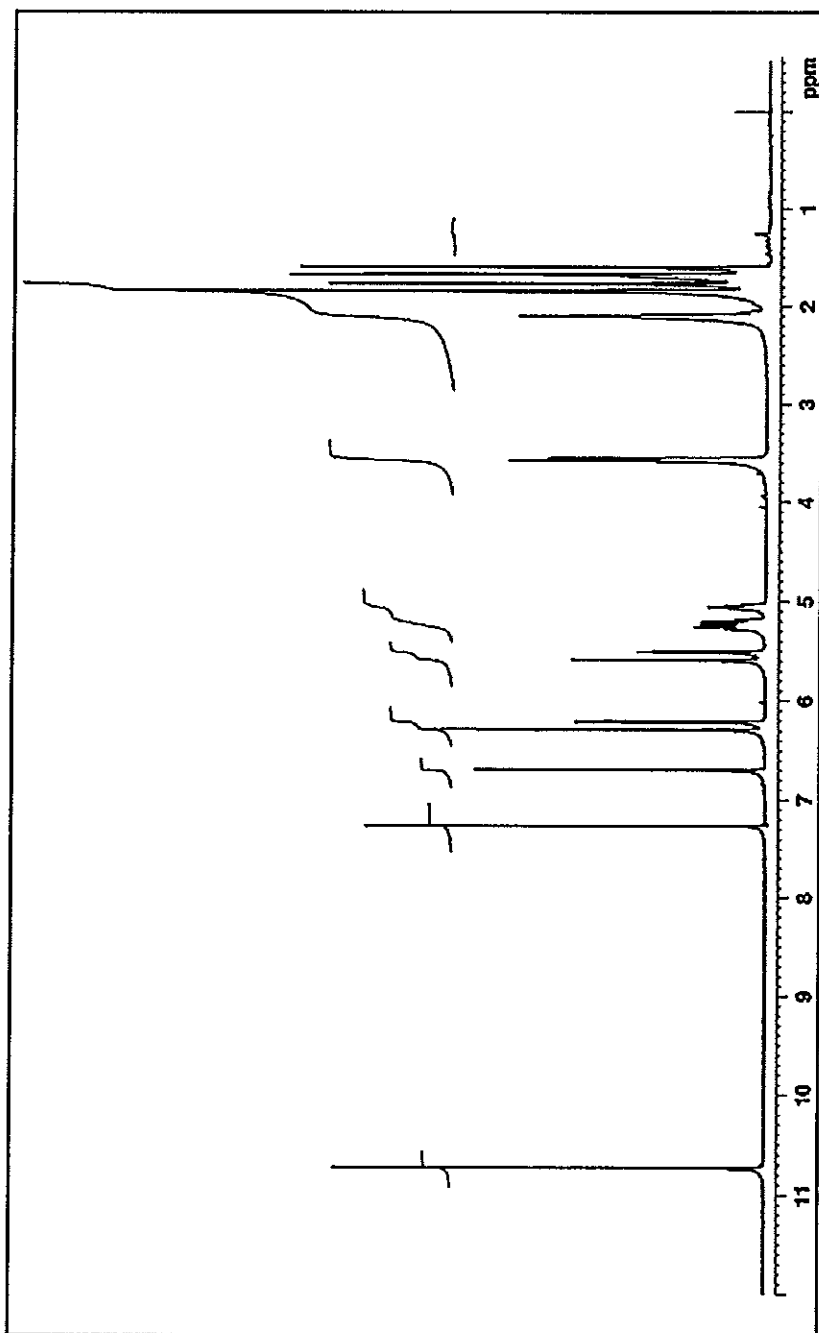


Figure 77  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound GP14

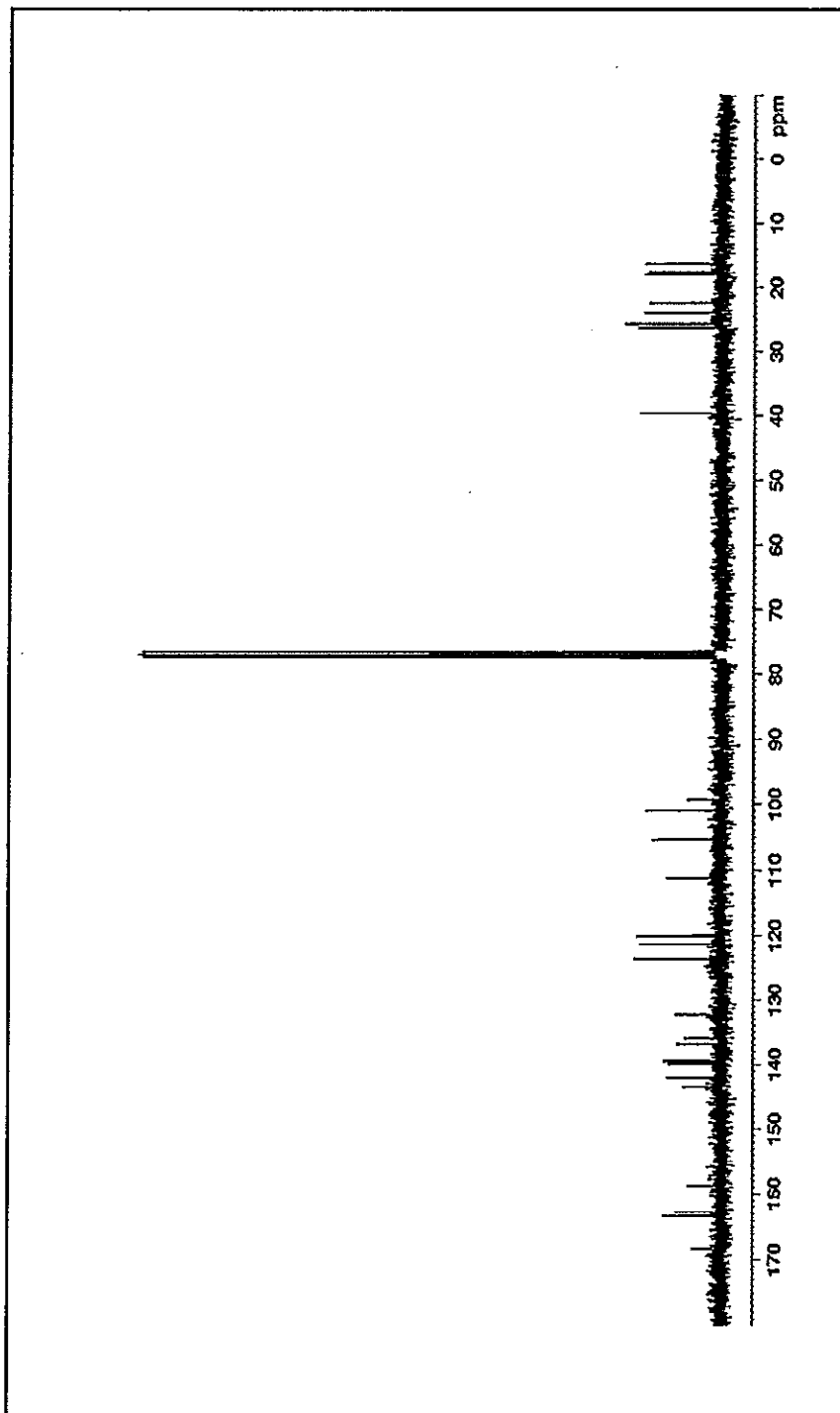


Figure 78  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound GP14

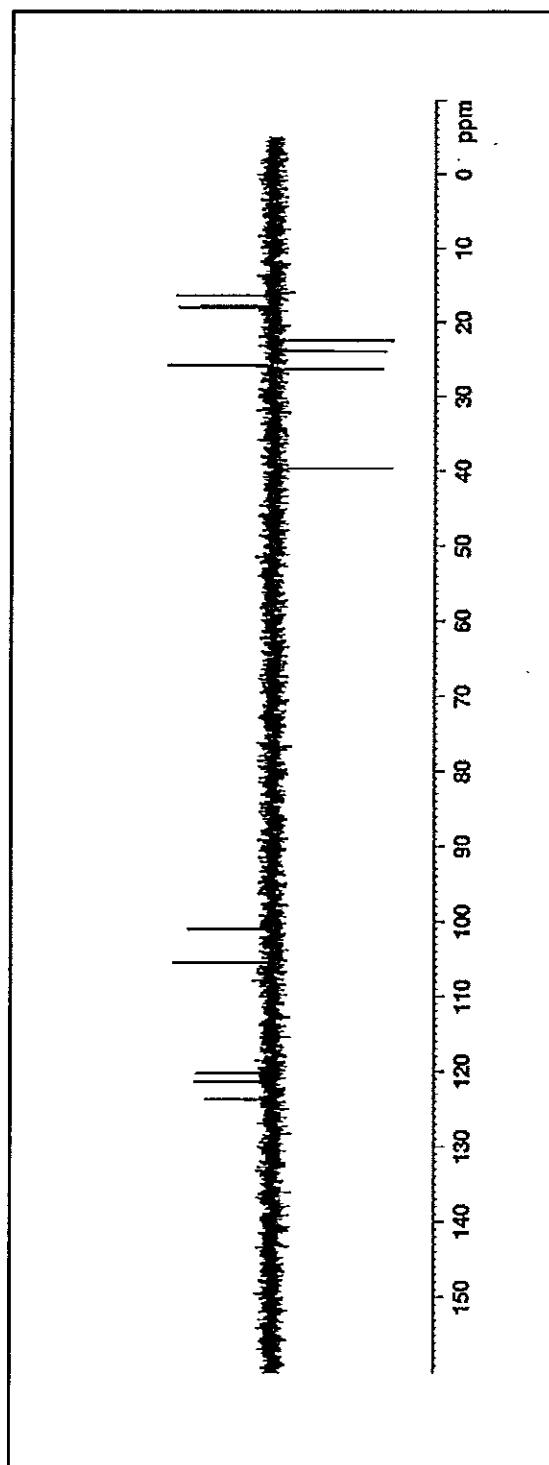


Figure 79 DEPT 135° spectra of compound GP14

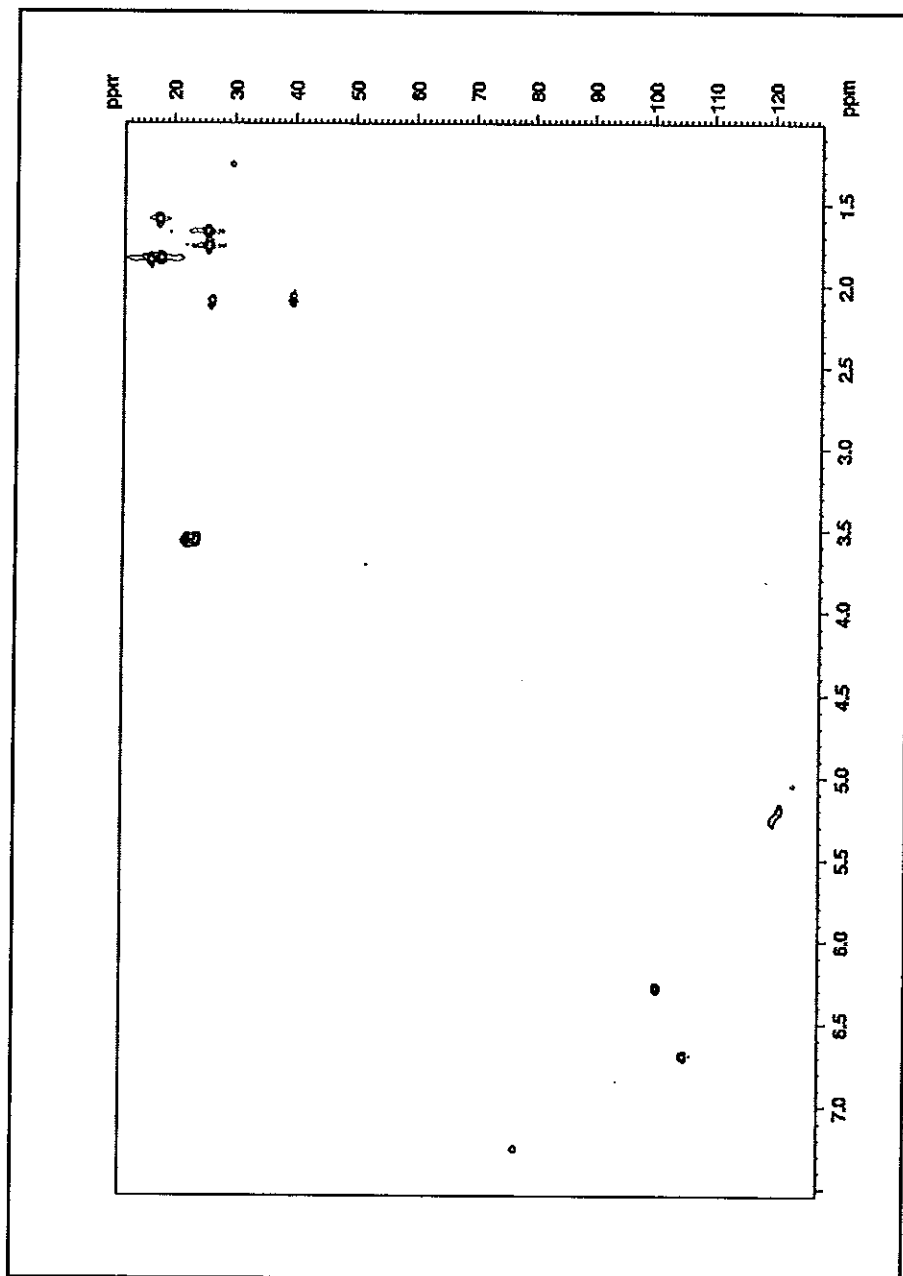


Figure 80 2D HMQC spectrum of compound GP14

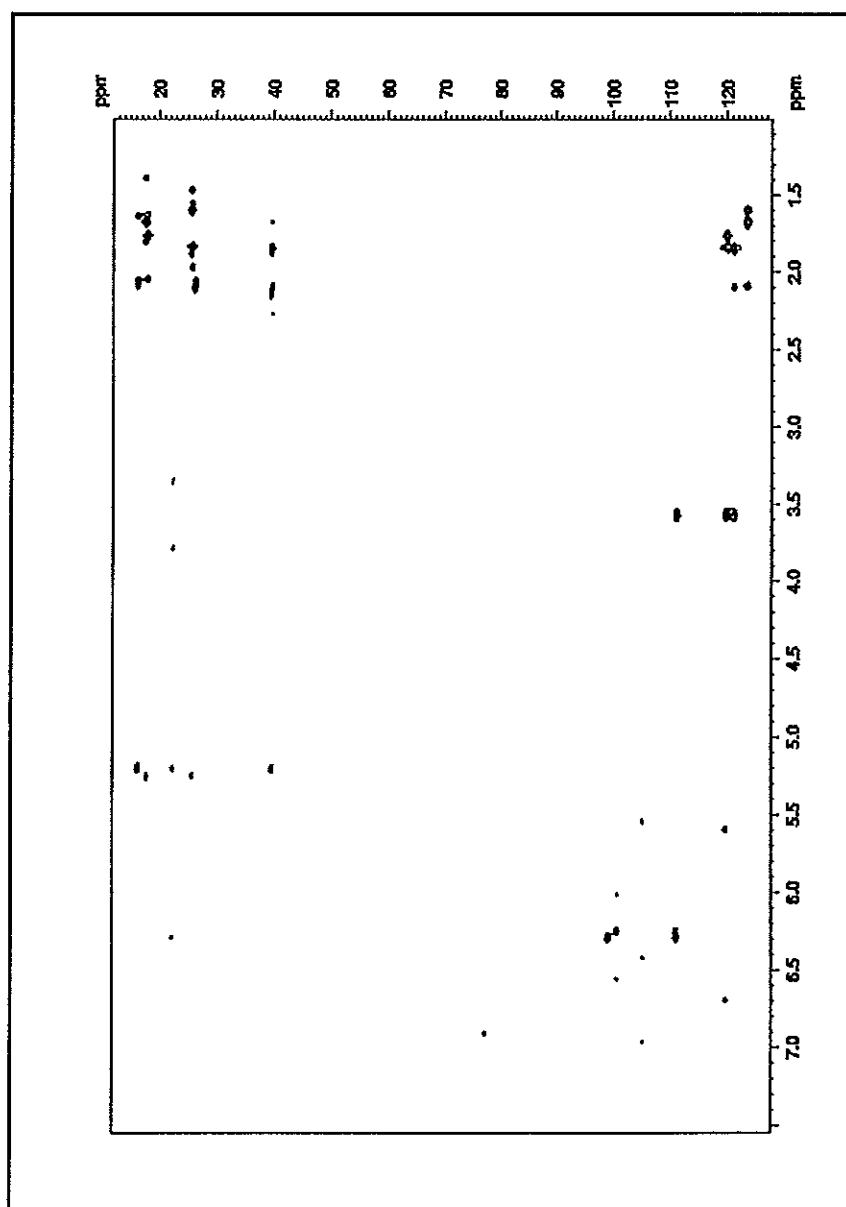


Figure 81 2D HMBC spectrum of compound GP14

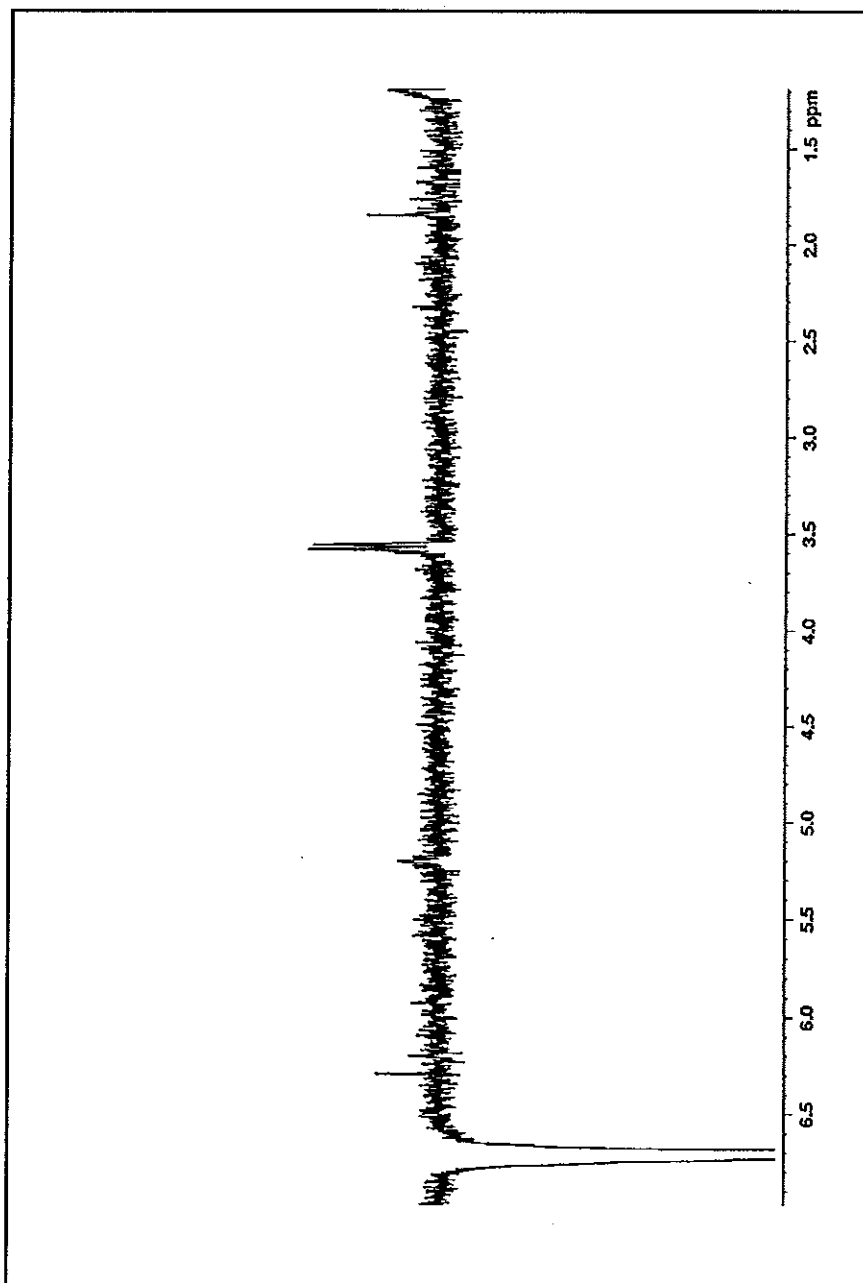


Figure 82 NOE difference spectrum of compound GP14 after irradiation at  $\delta_{\text{H}} 6.67$  (H-6)

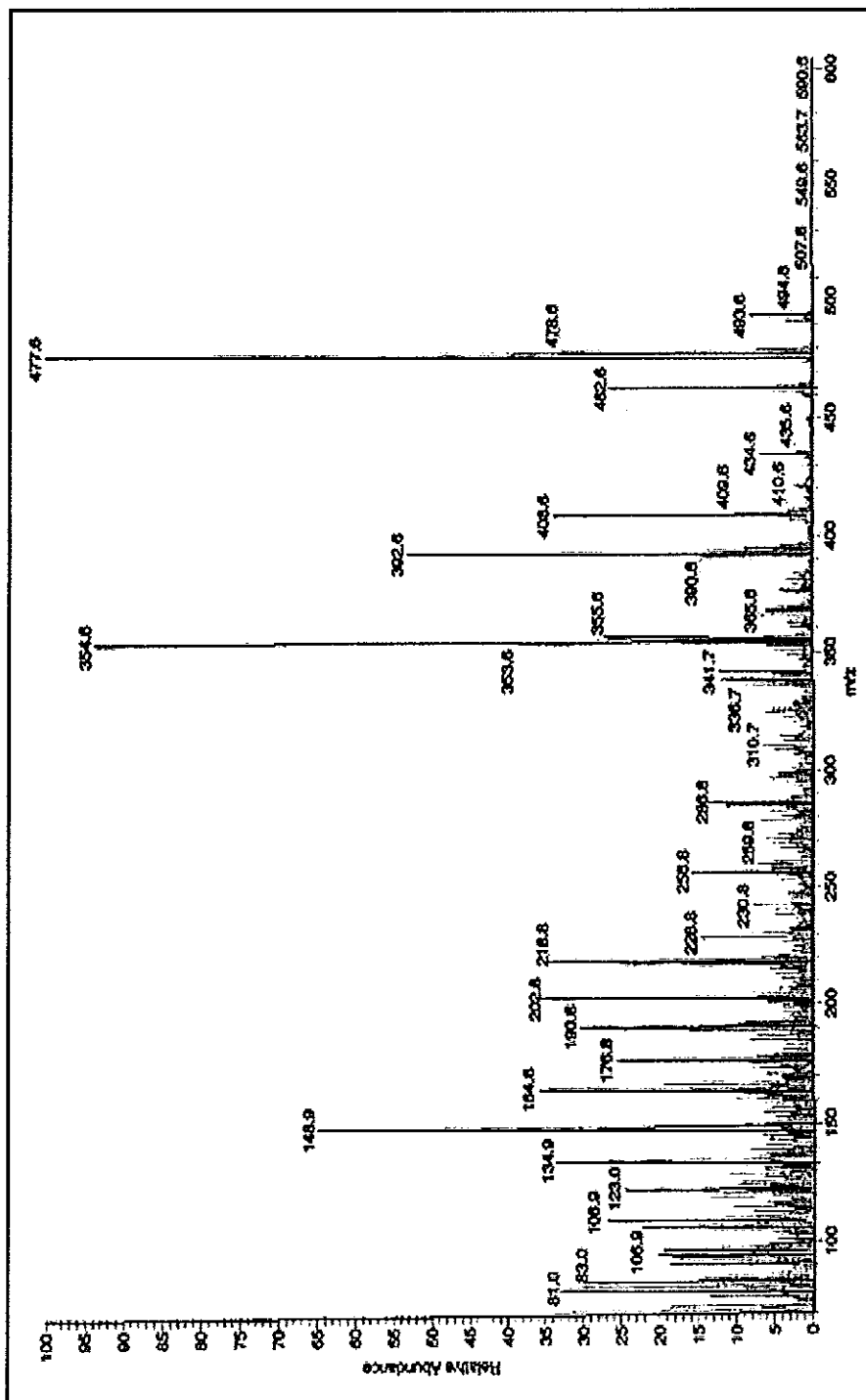


Figure 83 Mass spectrum of compound GP12



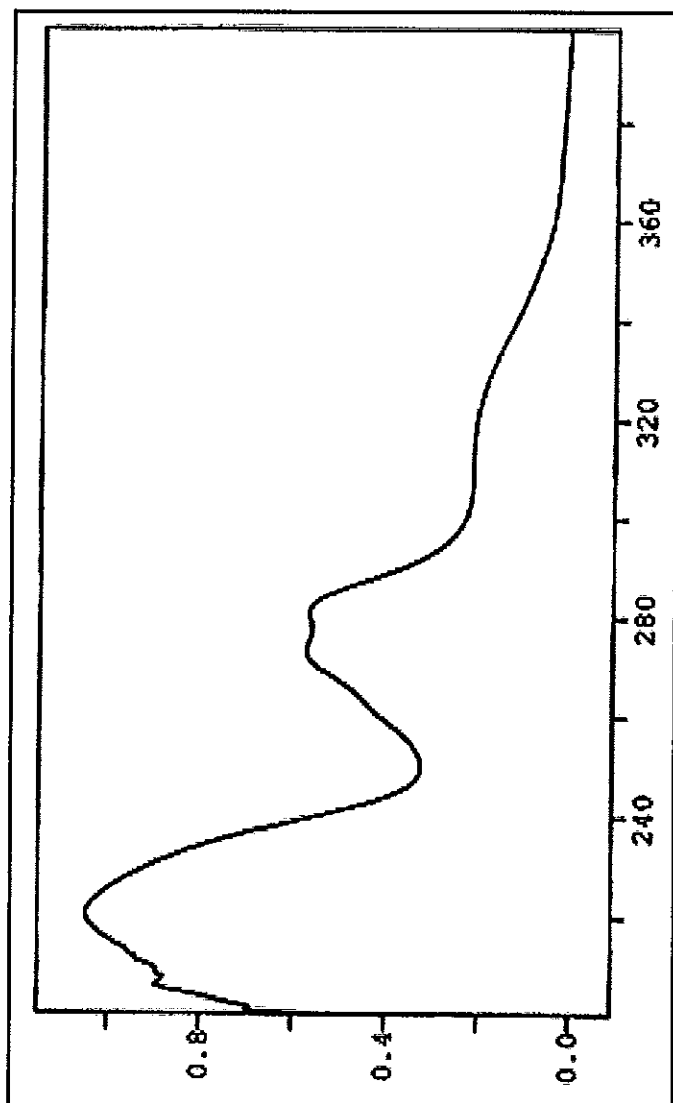


Figure 84 UV (MeOH) spectrum of compound GP12

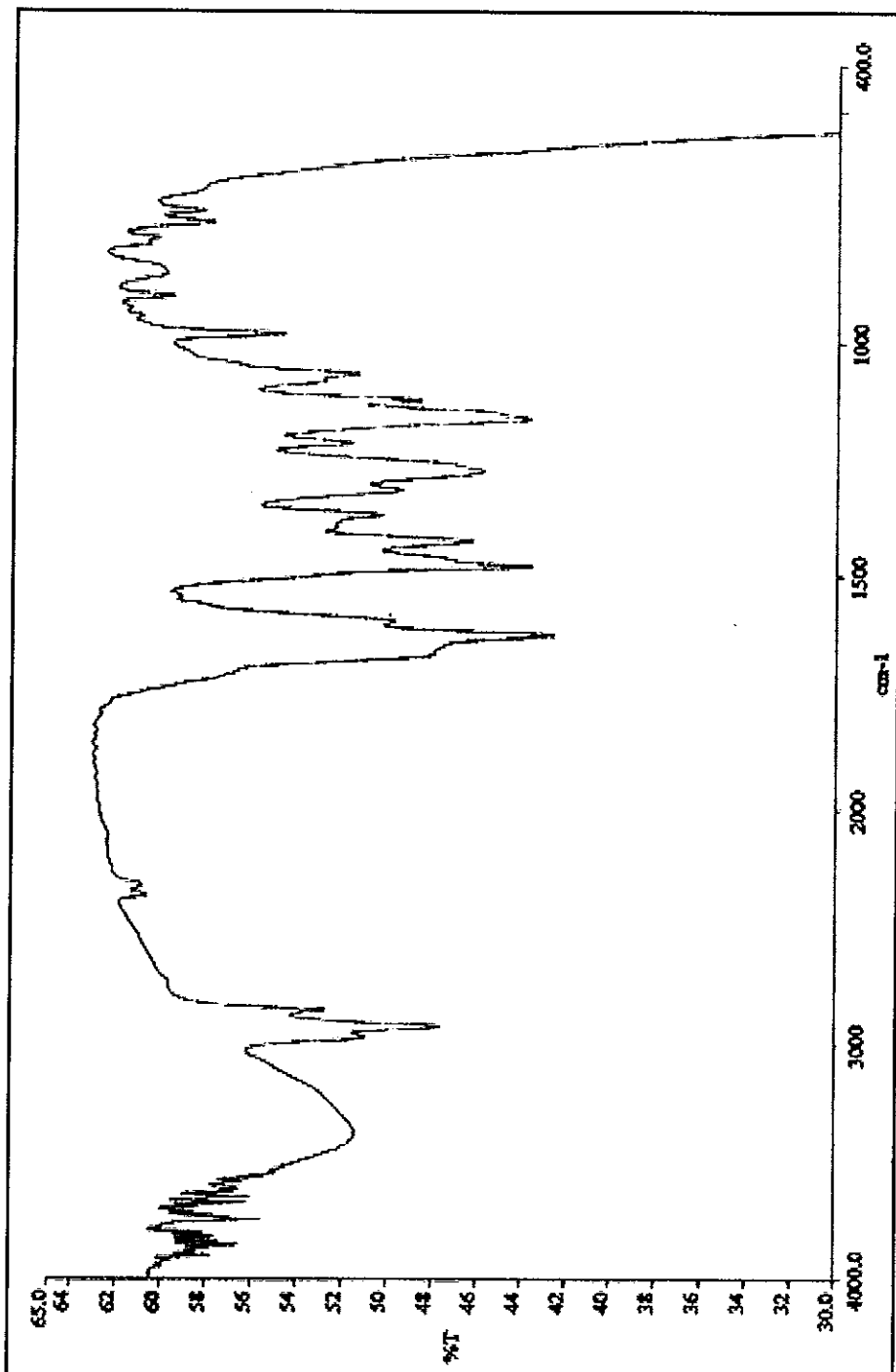


Figure 85 FT-IR (neat) spectrum of compound GP12

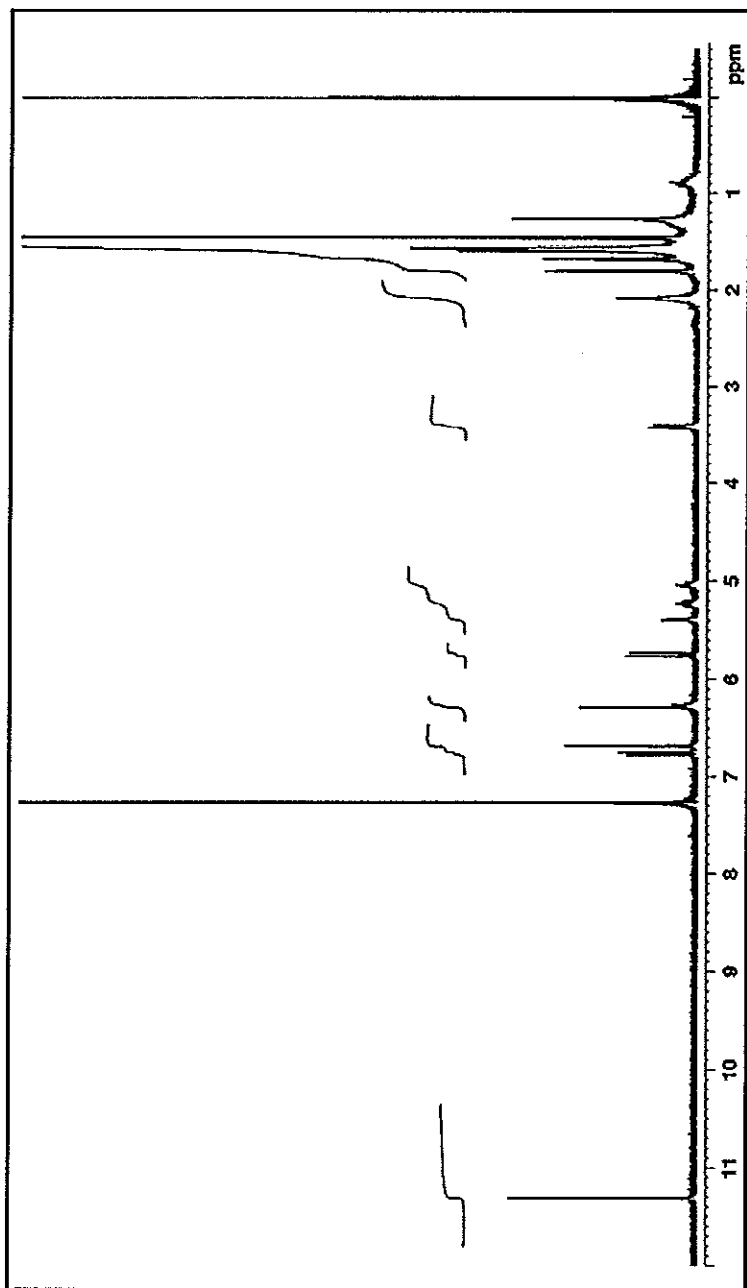


Figure 86  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound GP12

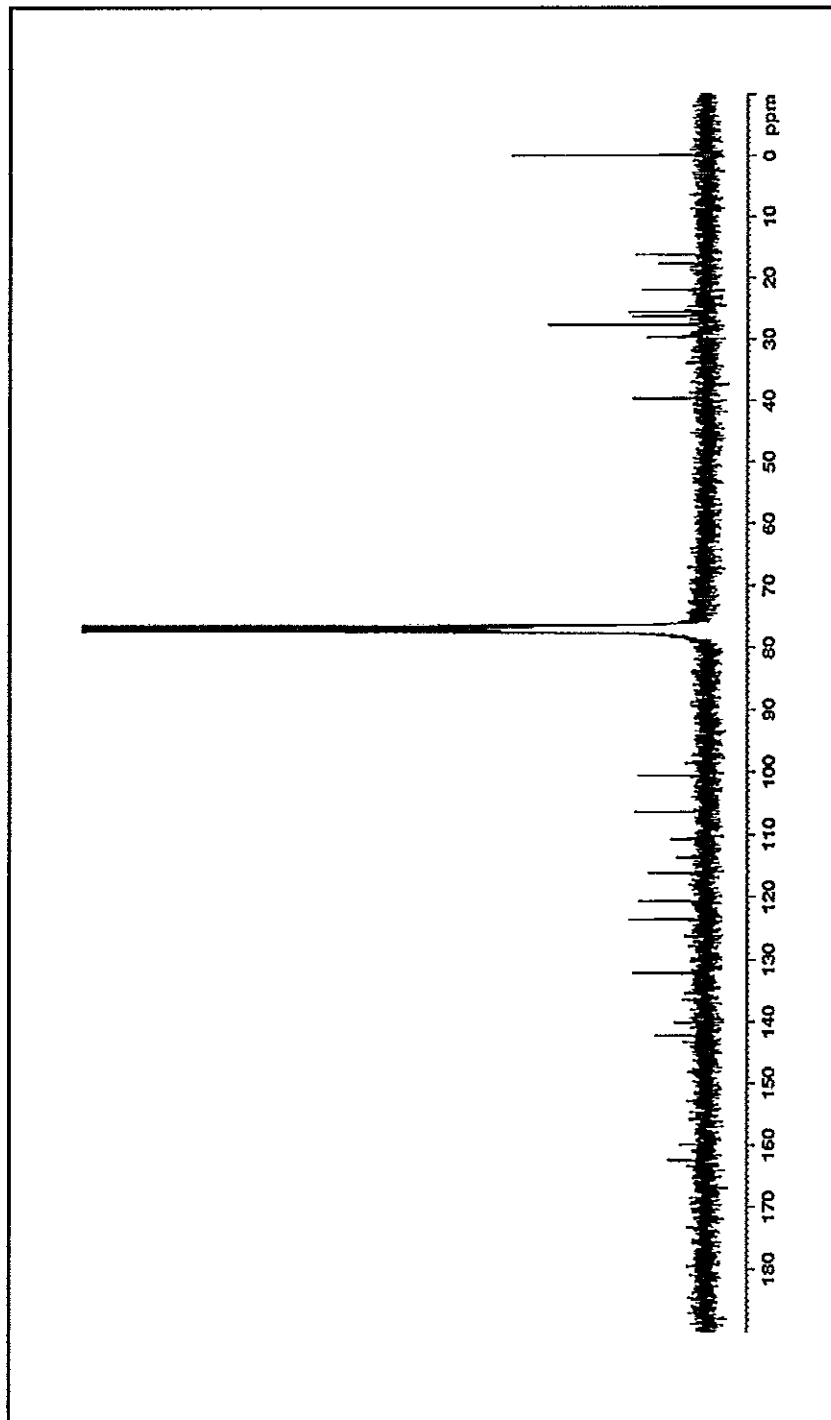


Figure 87  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound GP12

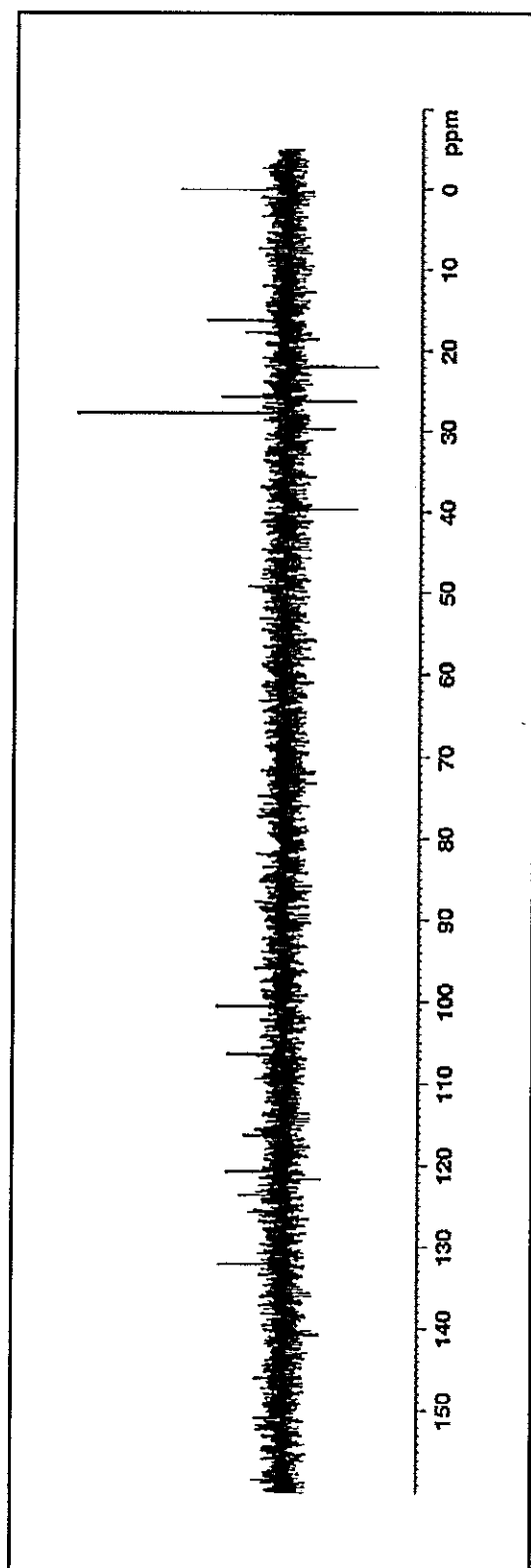


Figure 88 DEPT 135° spectra of compound GPI2

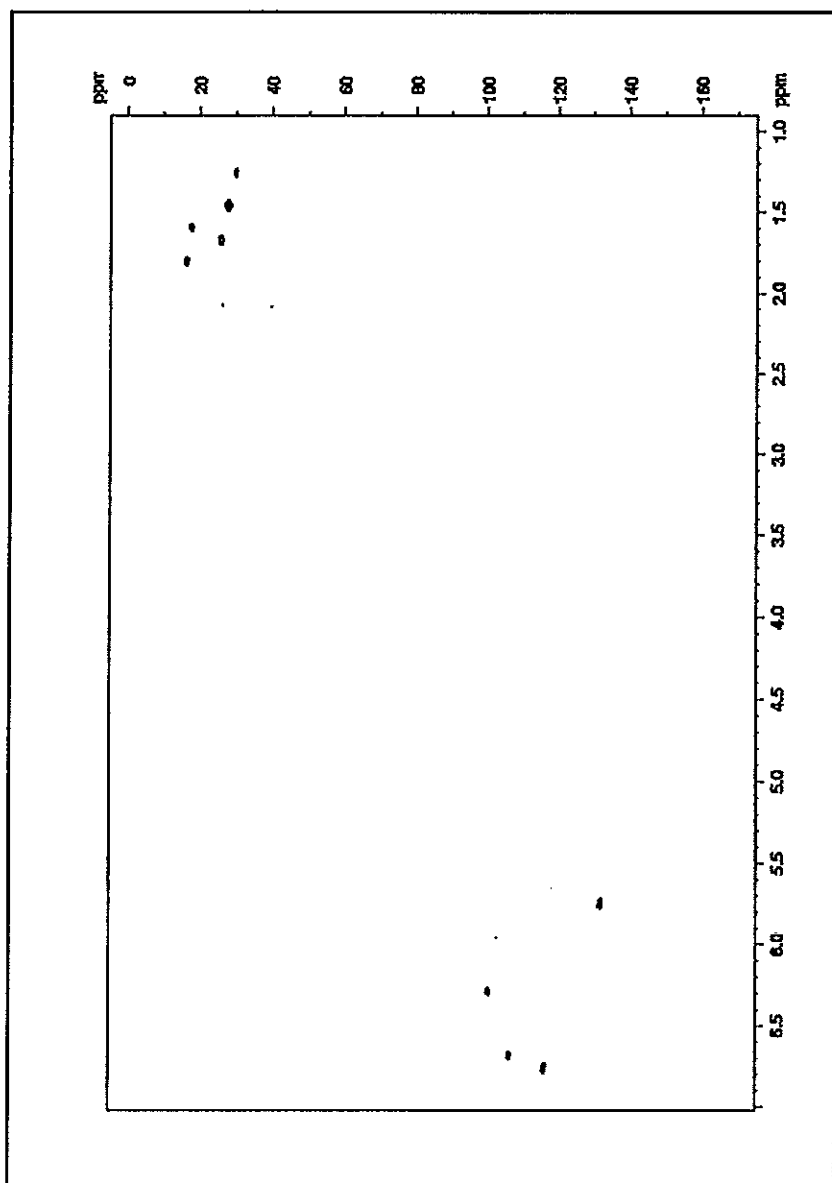


Figure 89 2D HMQC spectrum of compound GP12

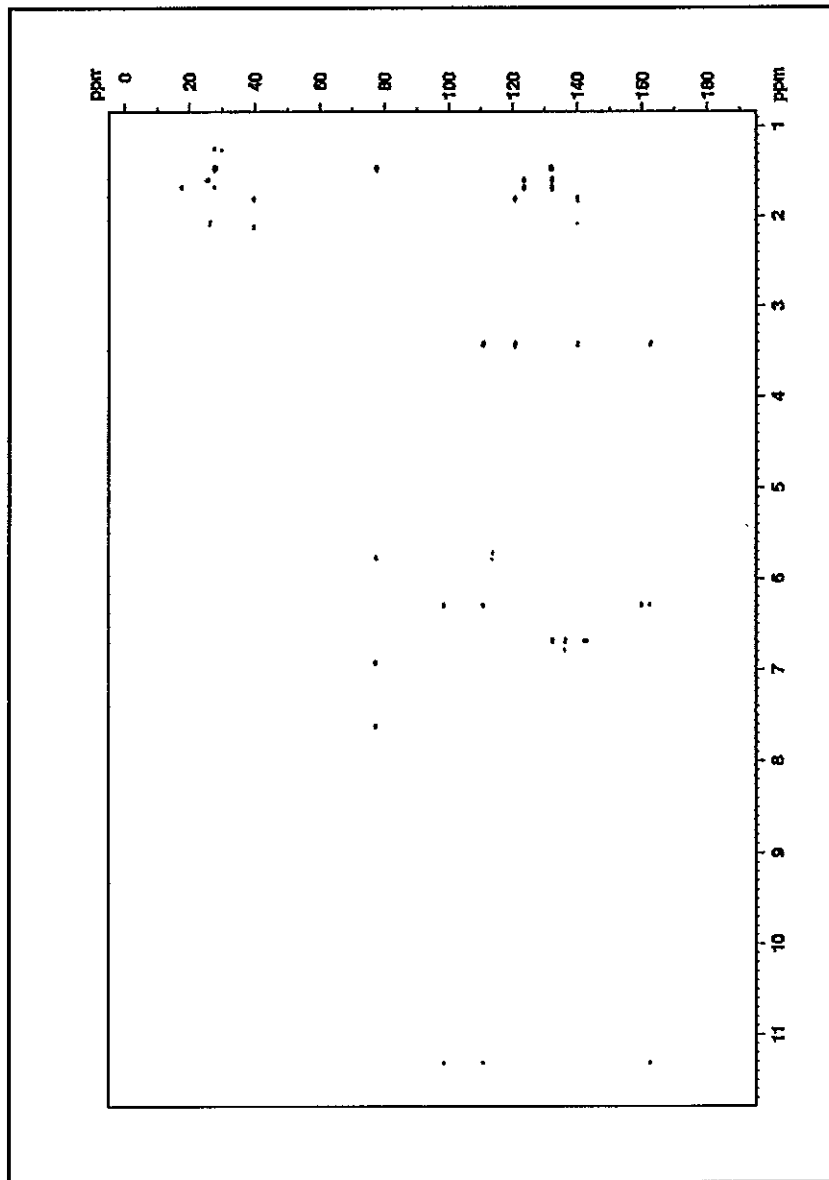


Figure 90 2D HMBC spectrum of compound GP12

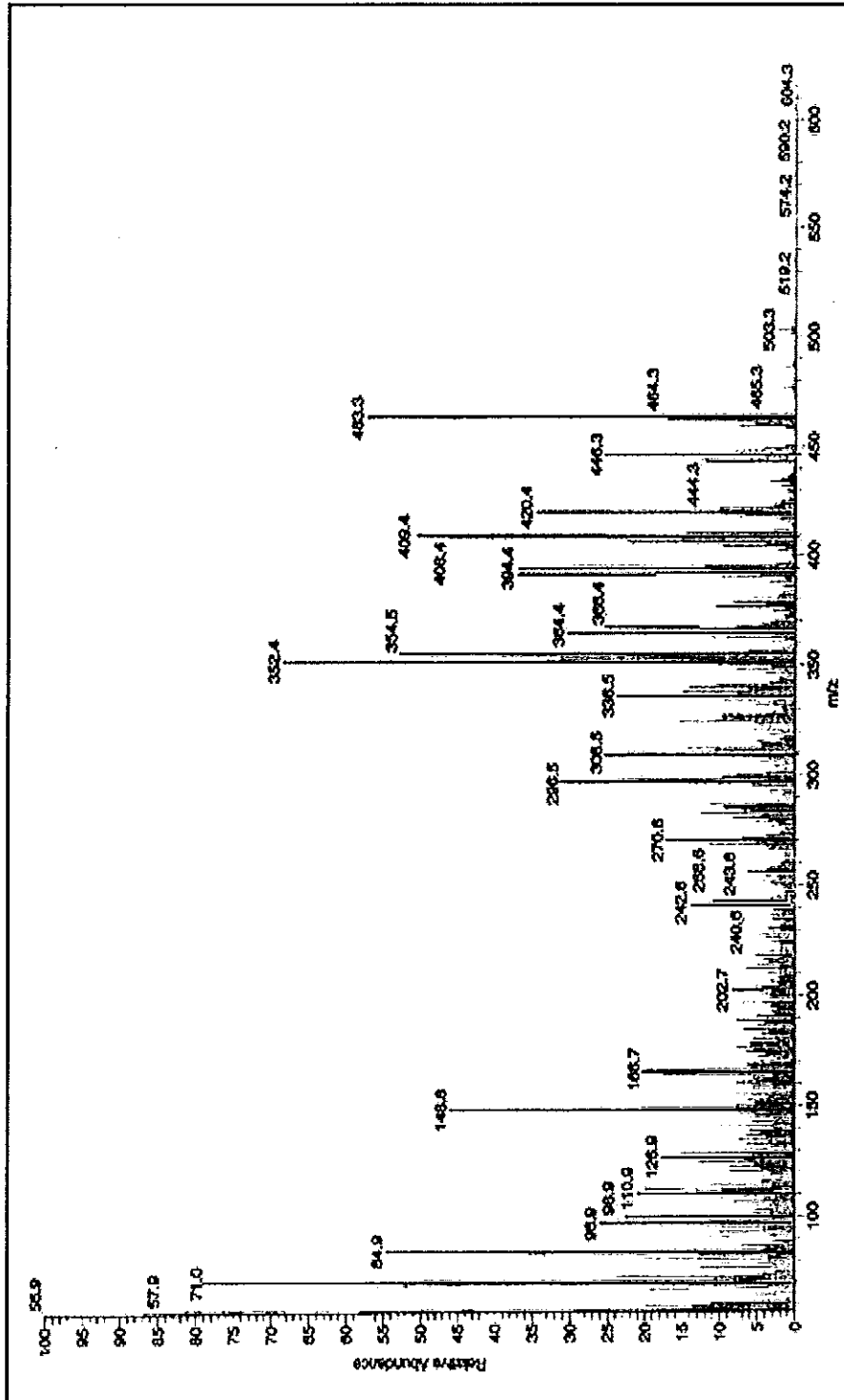


Figure 91 Mass spectrum of compound GP9



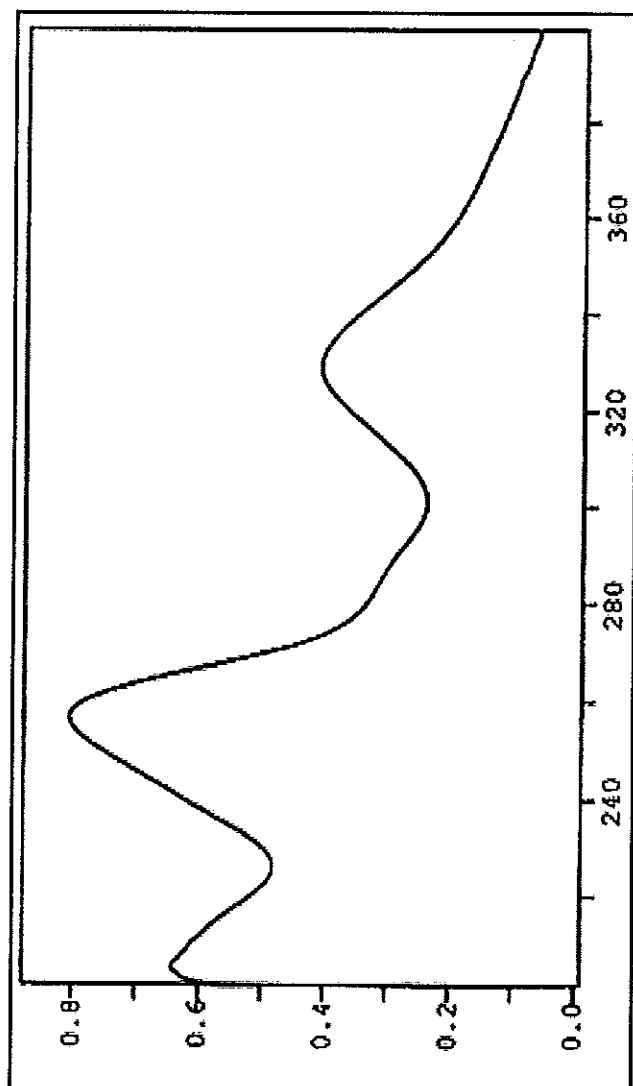


Figure 92 UV (MeOH) spectrum of compound GP9

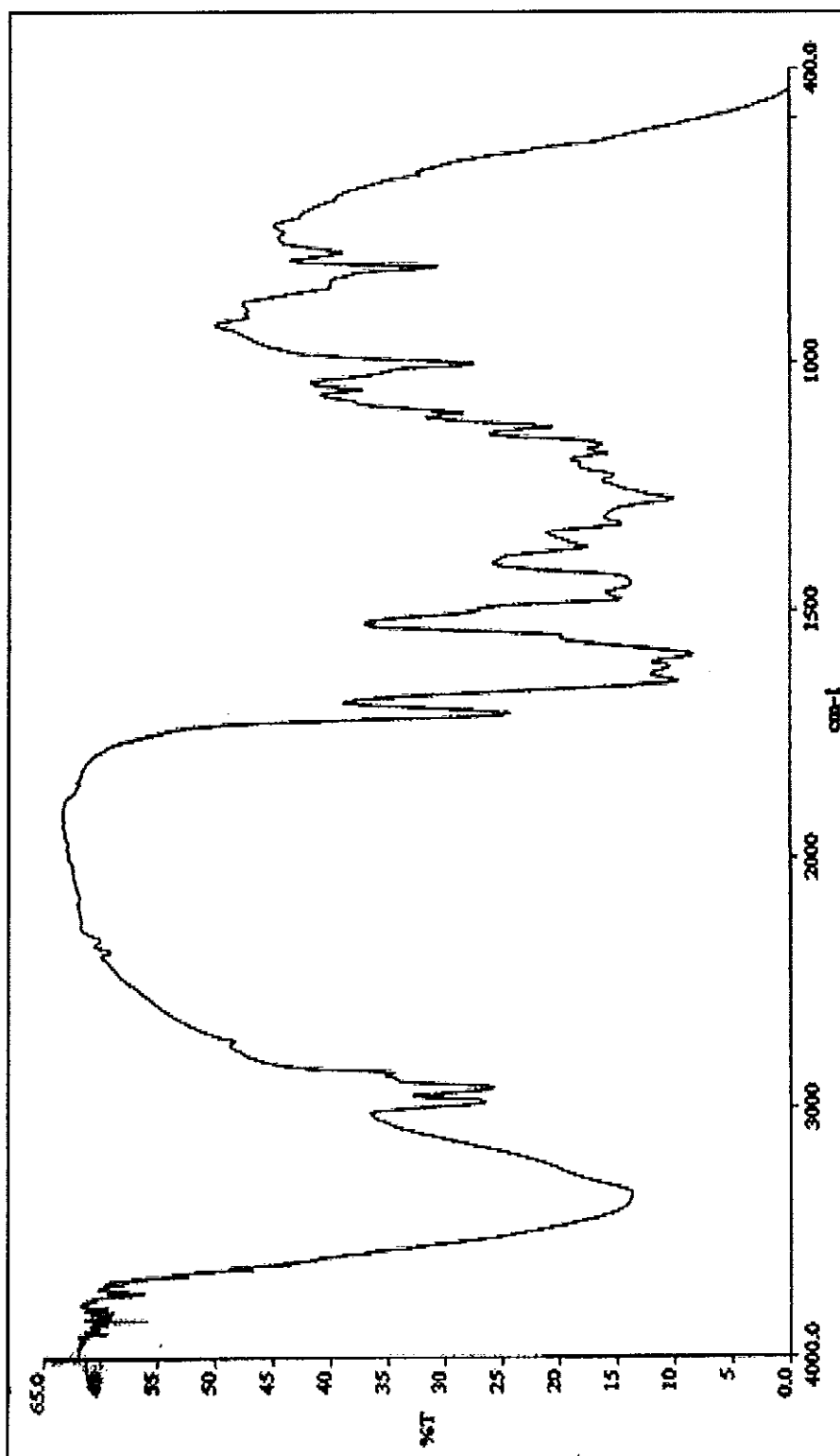


Figure 93 FT-IR (neat) spectrum of compound GP9

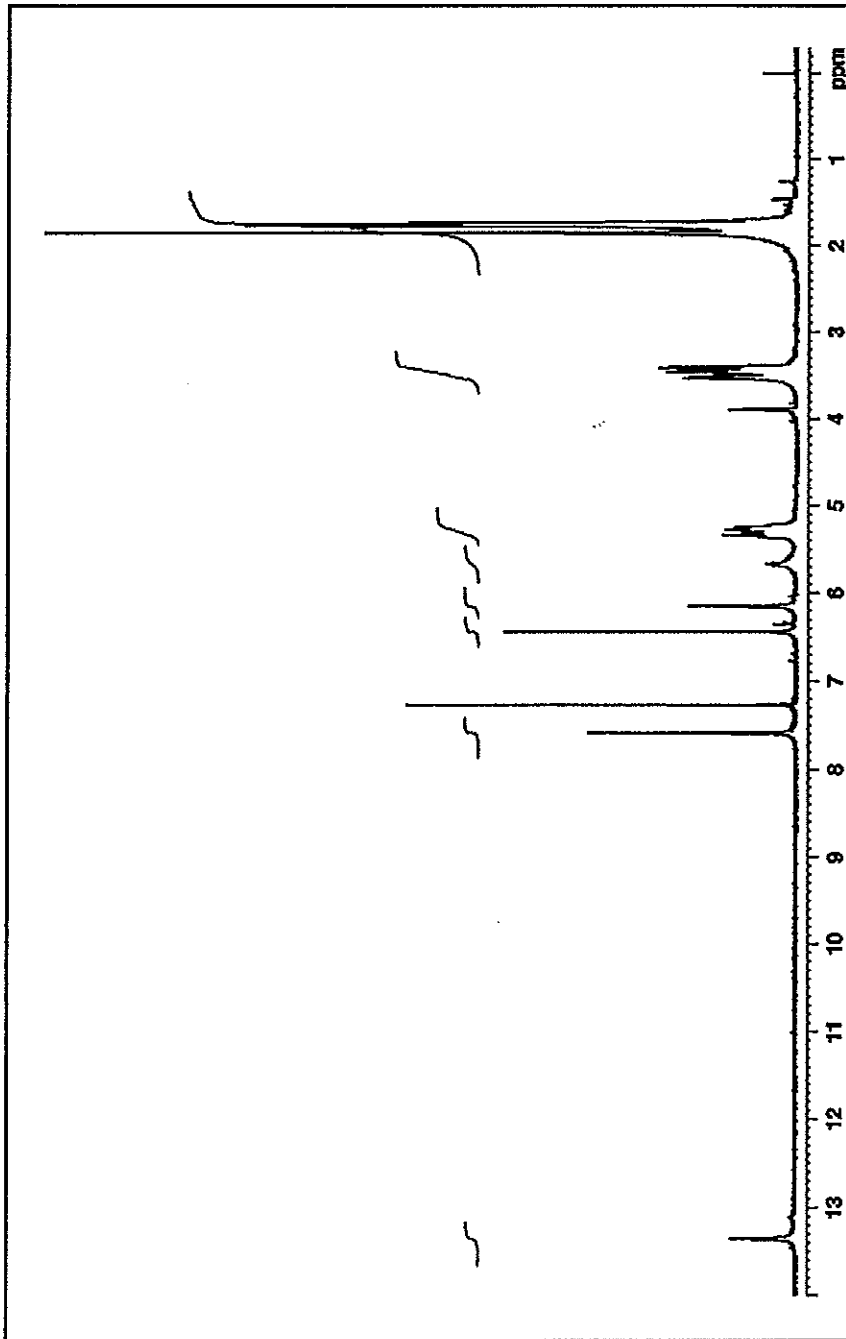


Figure 94  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound GP9

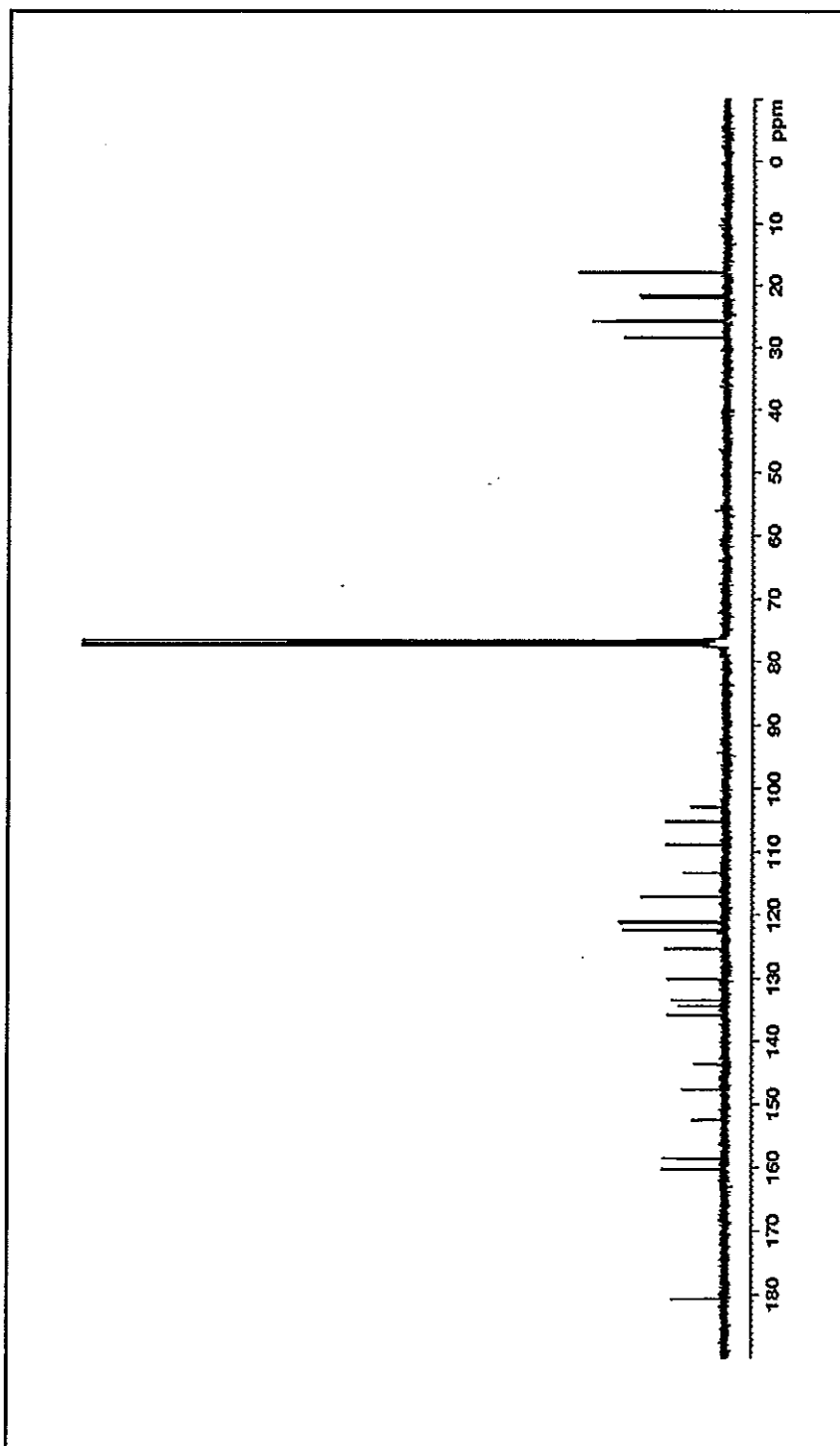


Figure 95  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound GP9

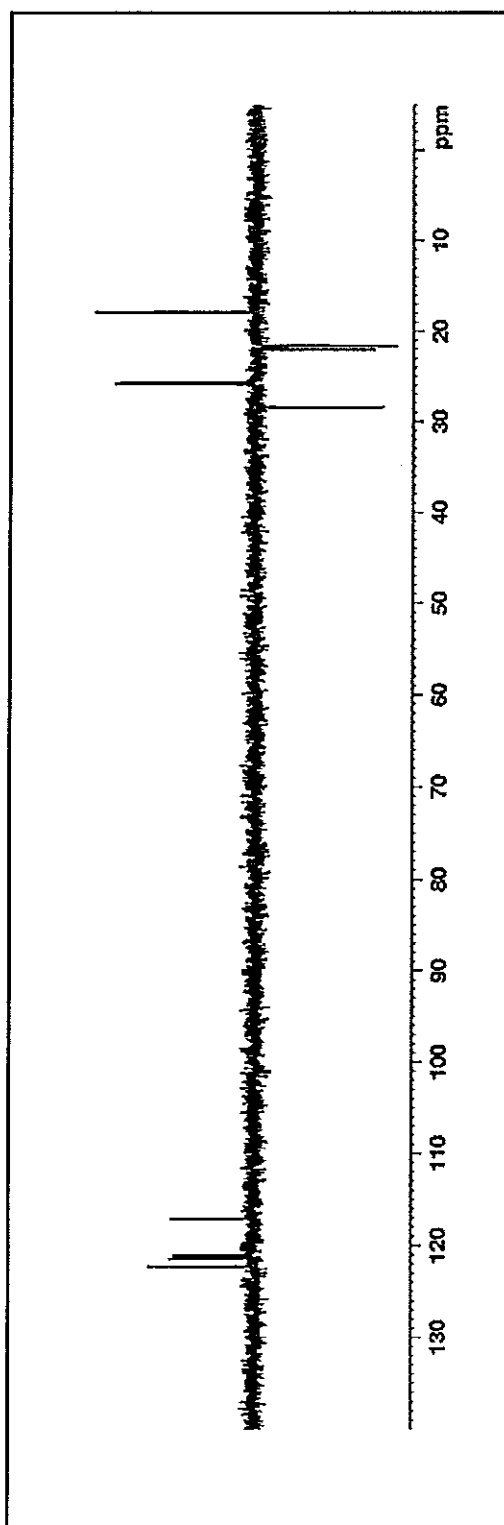


Figure 96 DEPT 135° spectra of compound GP9

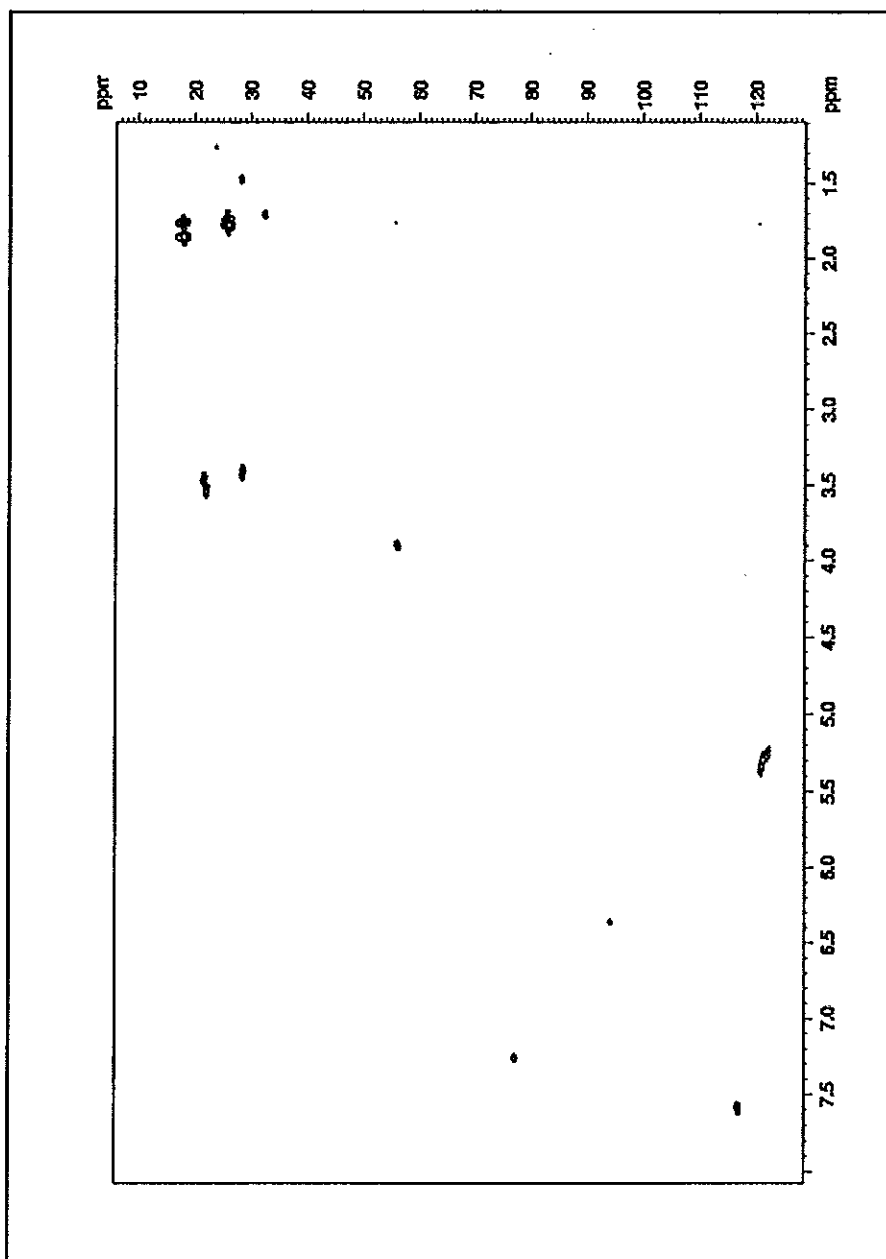


Figure 97 2D HMQC spectrum of compound GP9

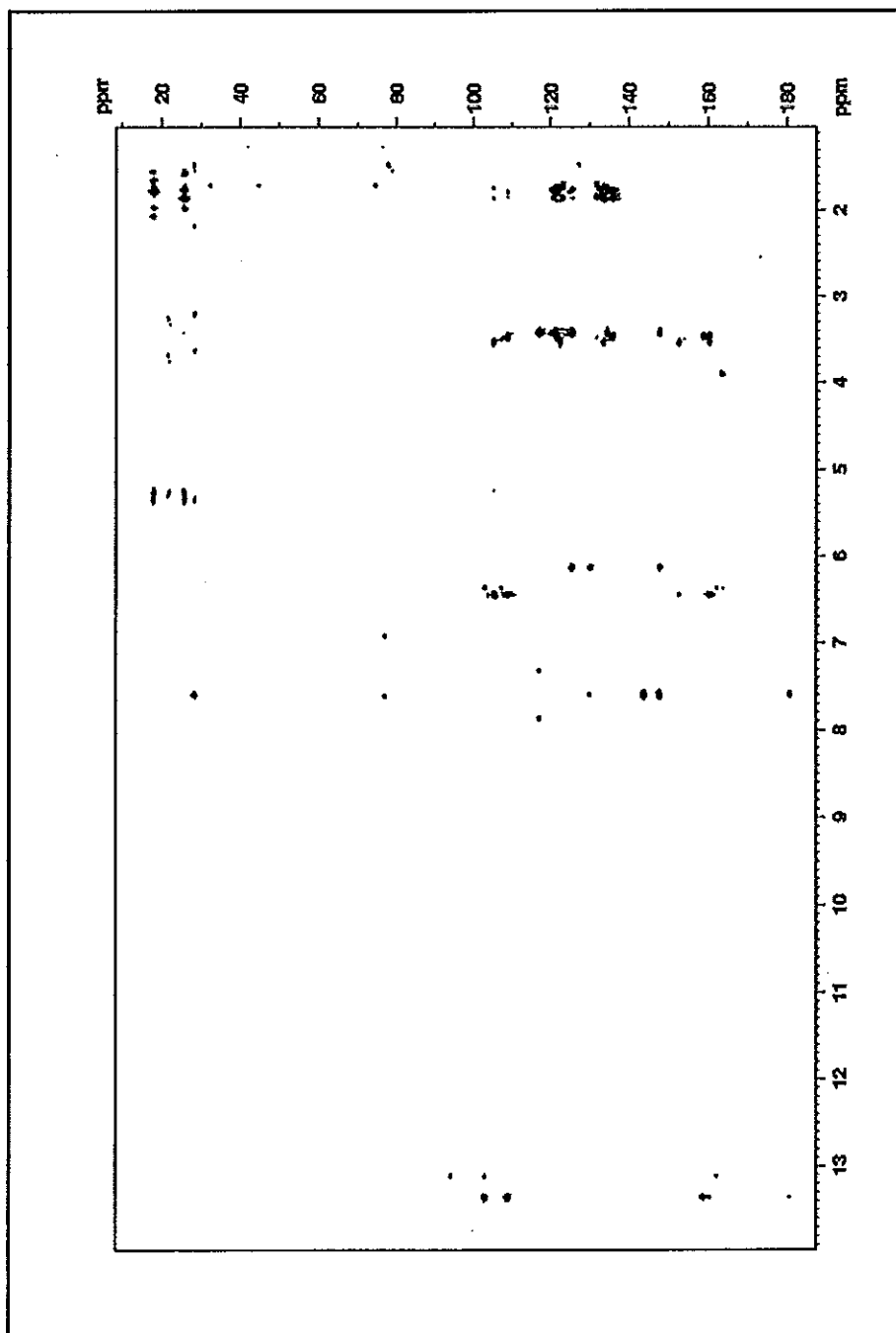


Figure 98 2D HMBC spectrum of compound GP9

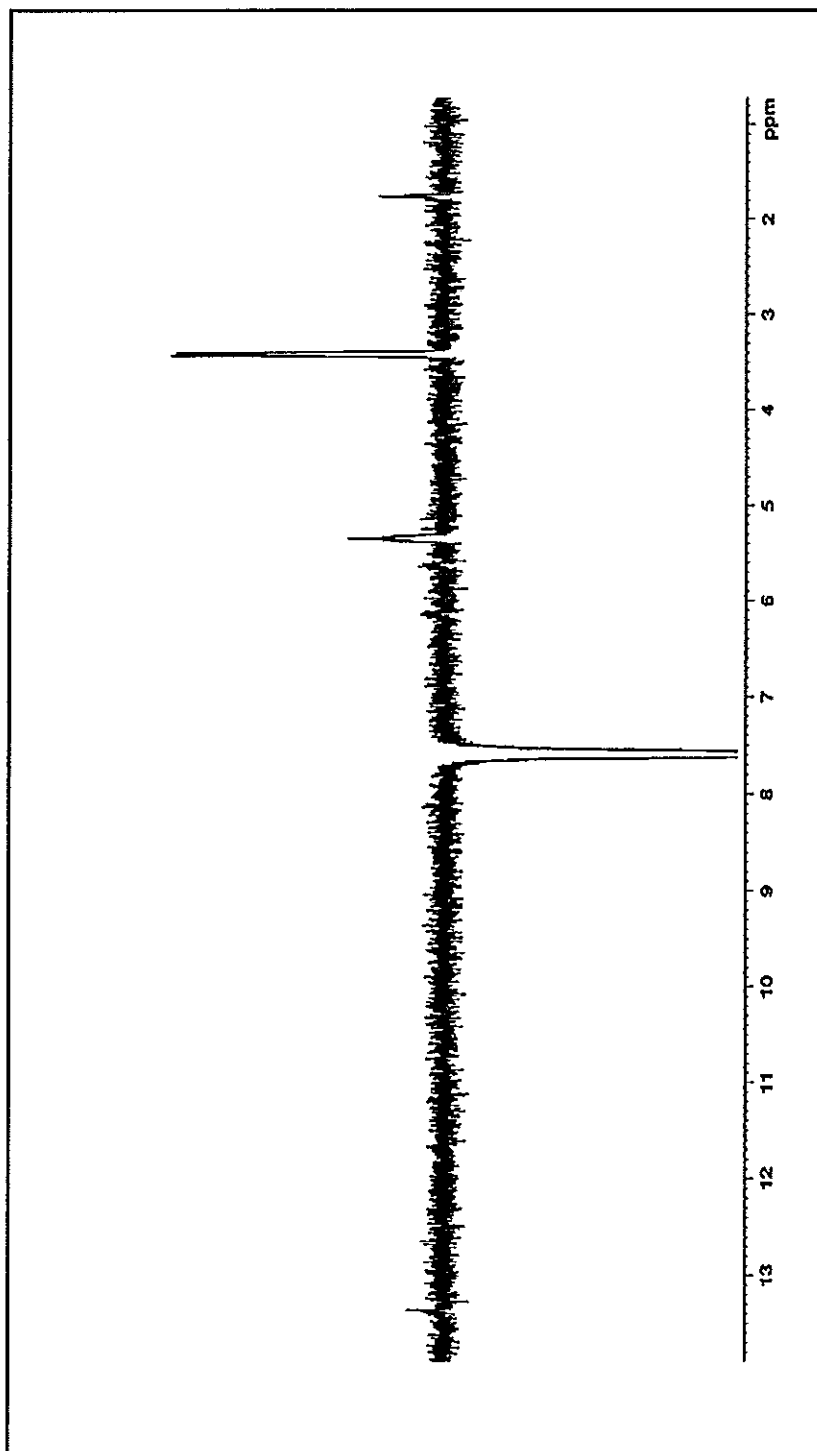


Figure 99 NOE difference spectrum of compound GP9 after irradiation at  $\delta_{\text{H}} 7.58$  (H-8)



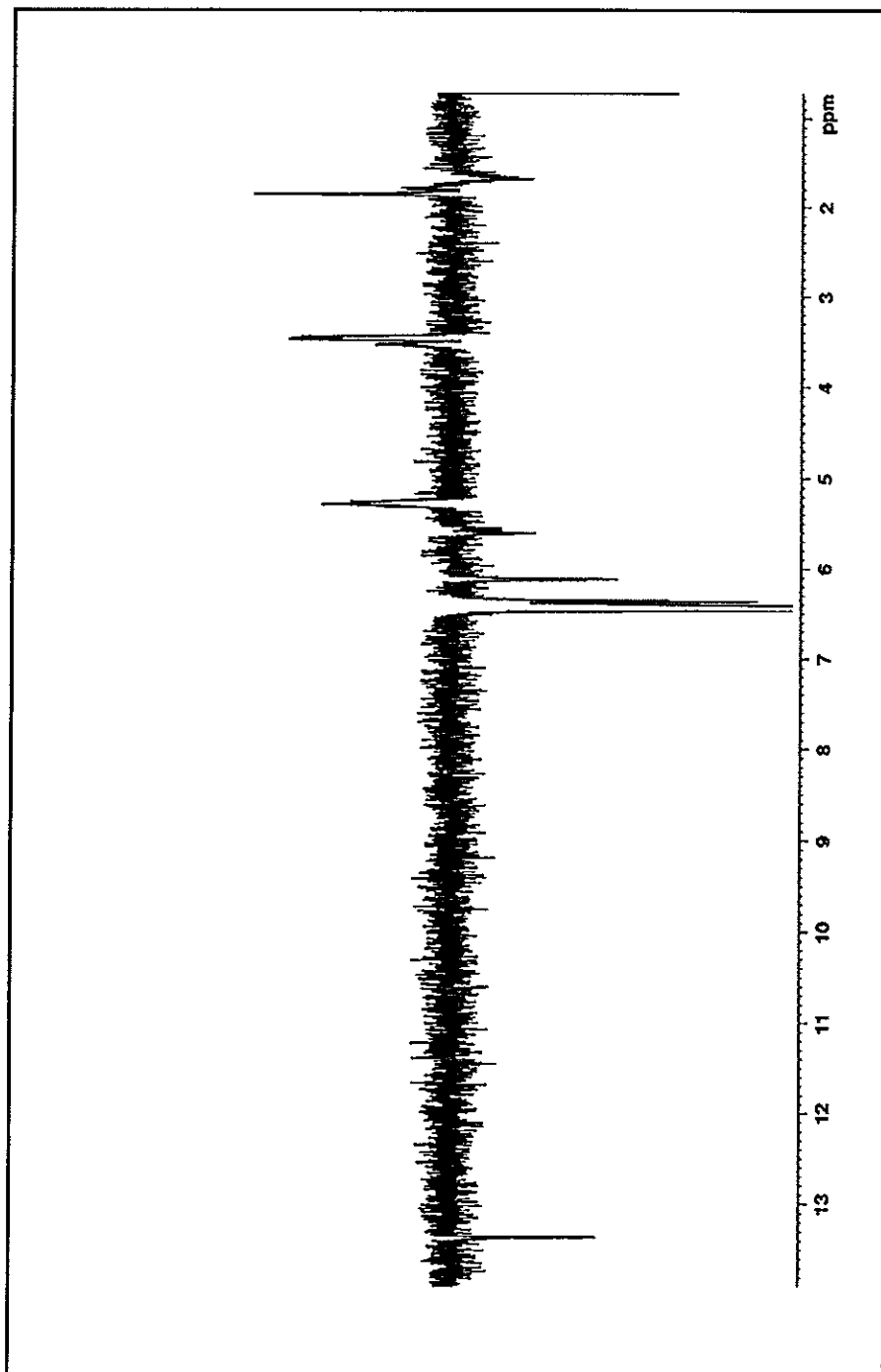


Figure 100 NOE difference spectrum of compound GP9 after irradiation at  $\delta_{\text{H}} 6.43$  (3-OH)

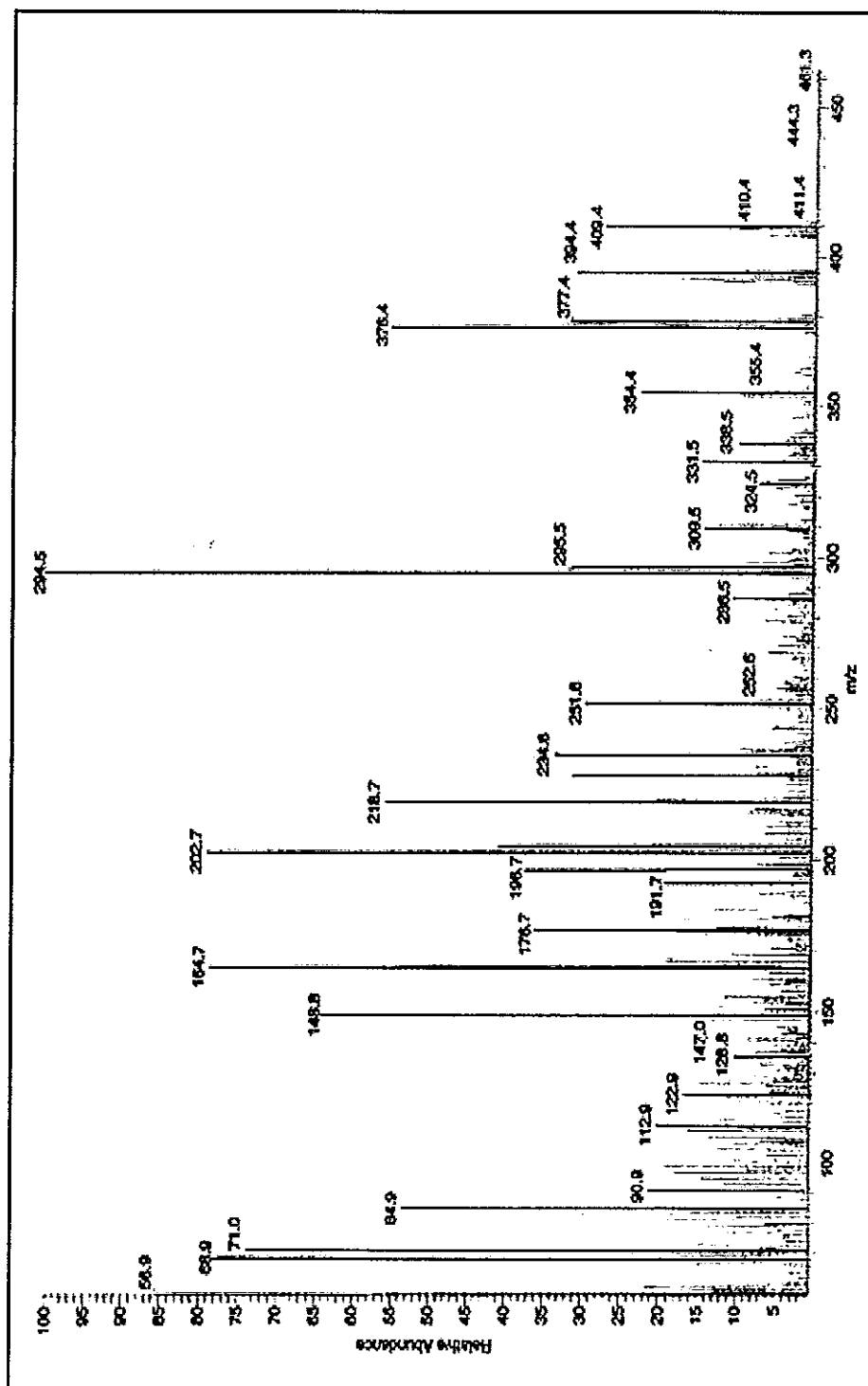


Figure 101 Mass spectrum of compound GP8

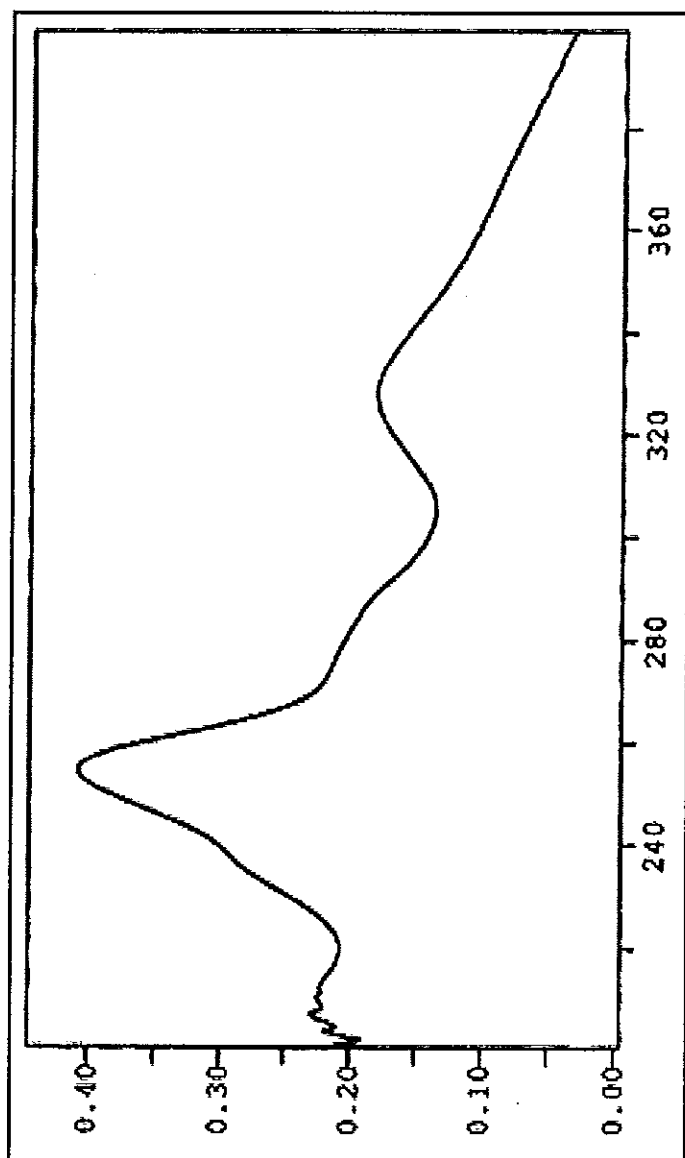


Figure 102 UV (MeOH) spectrum of compound GP8

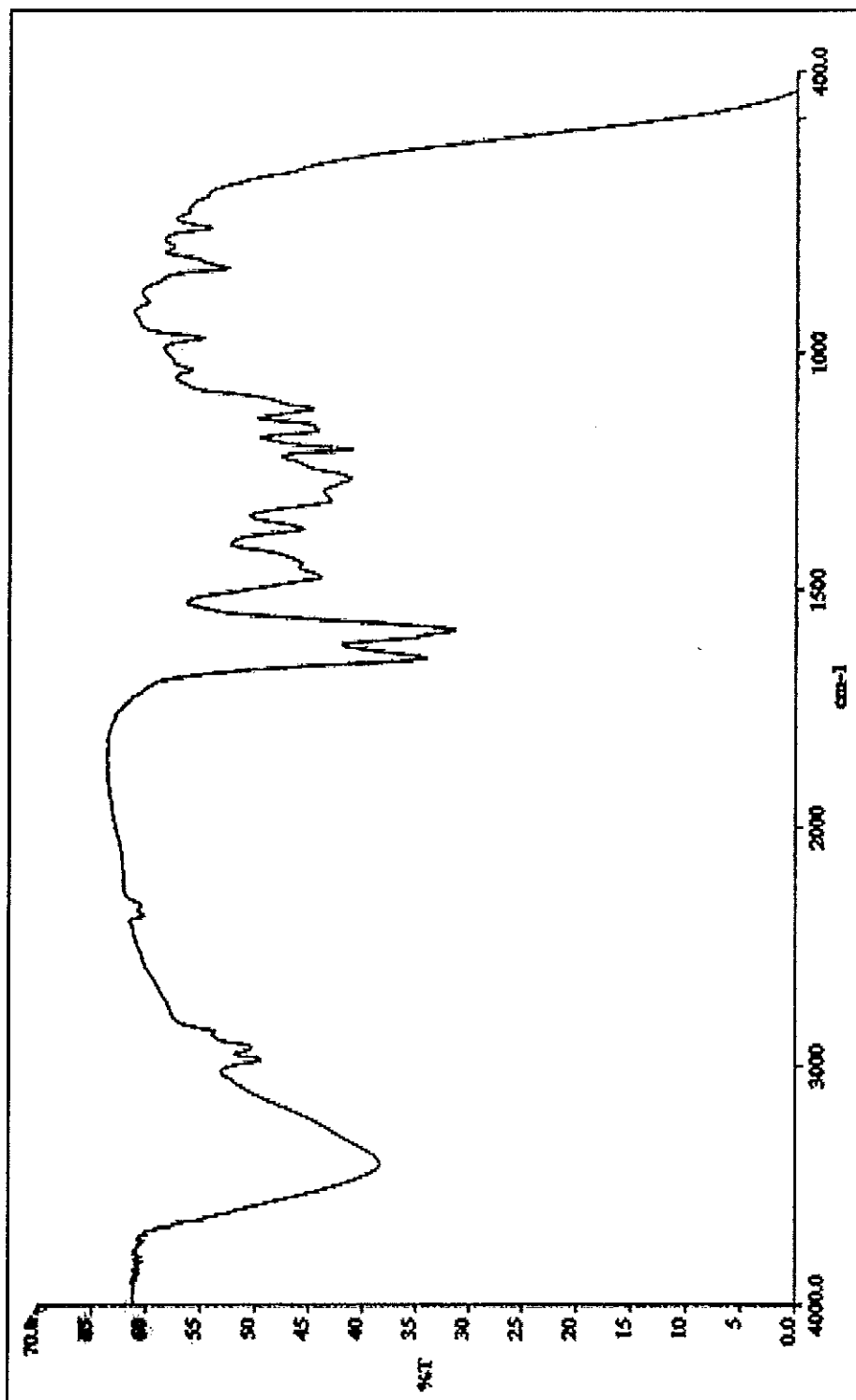


Figure 103 FT-IR (neat) spectrum of compound GP8

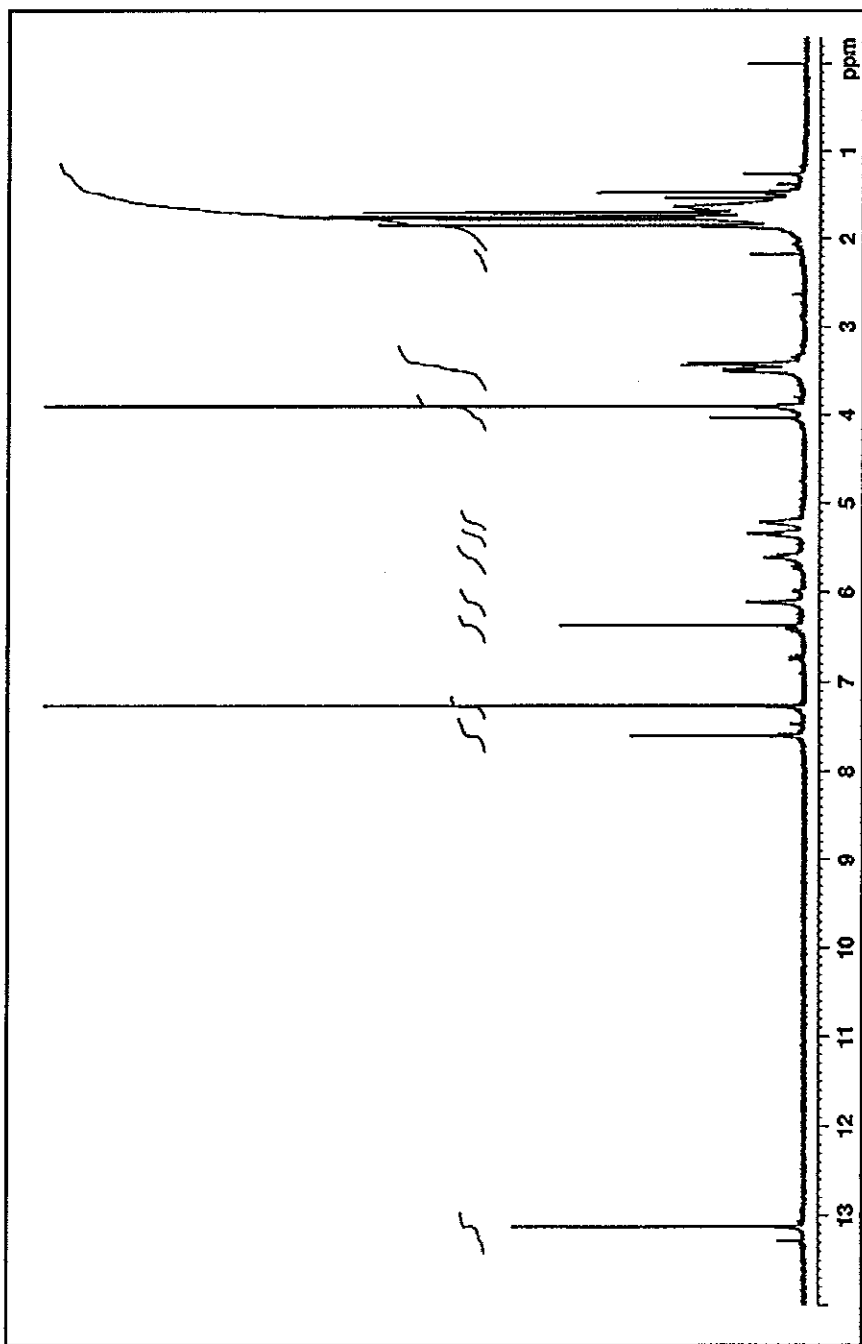


Figure 104  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound GP8

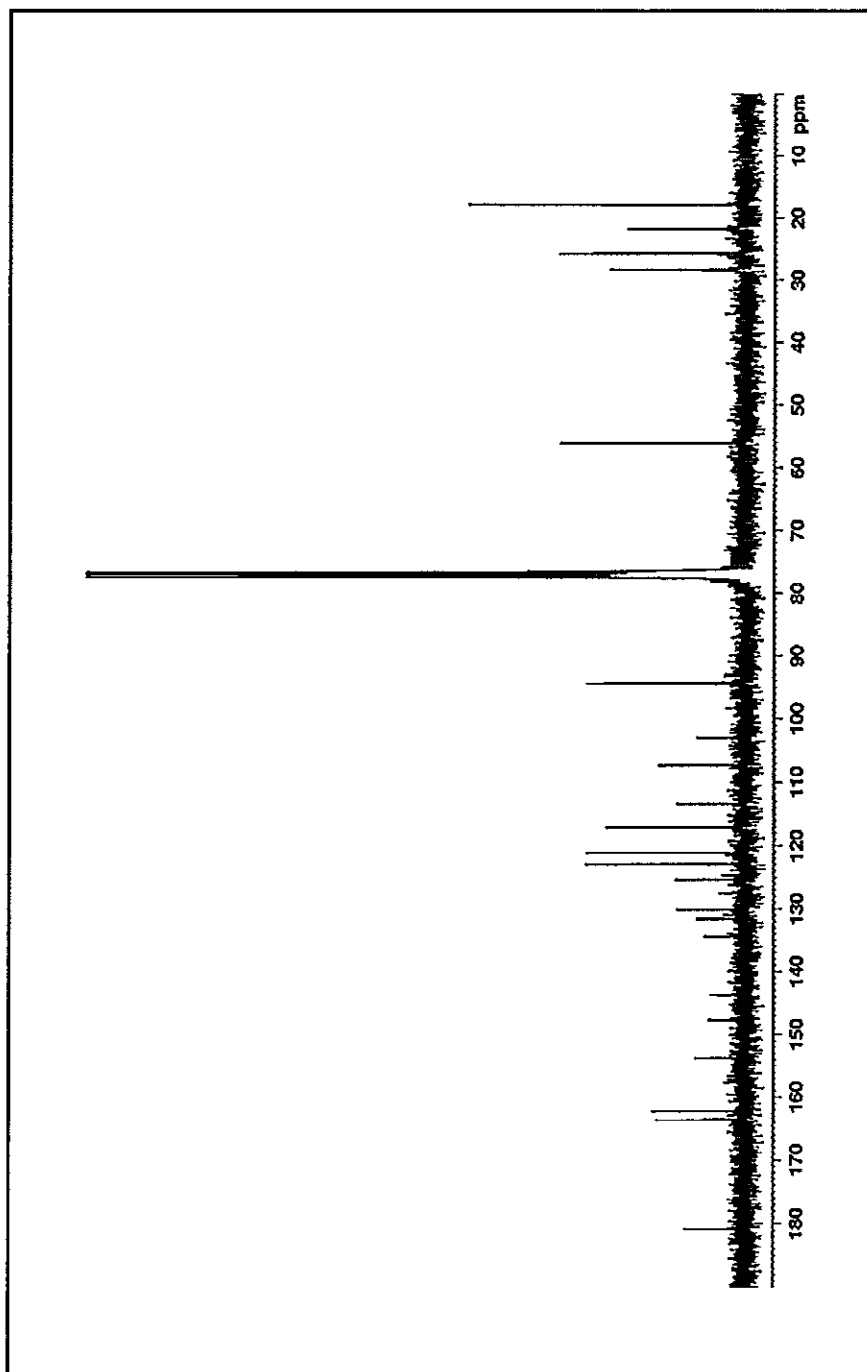


Figure 105  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound GP8

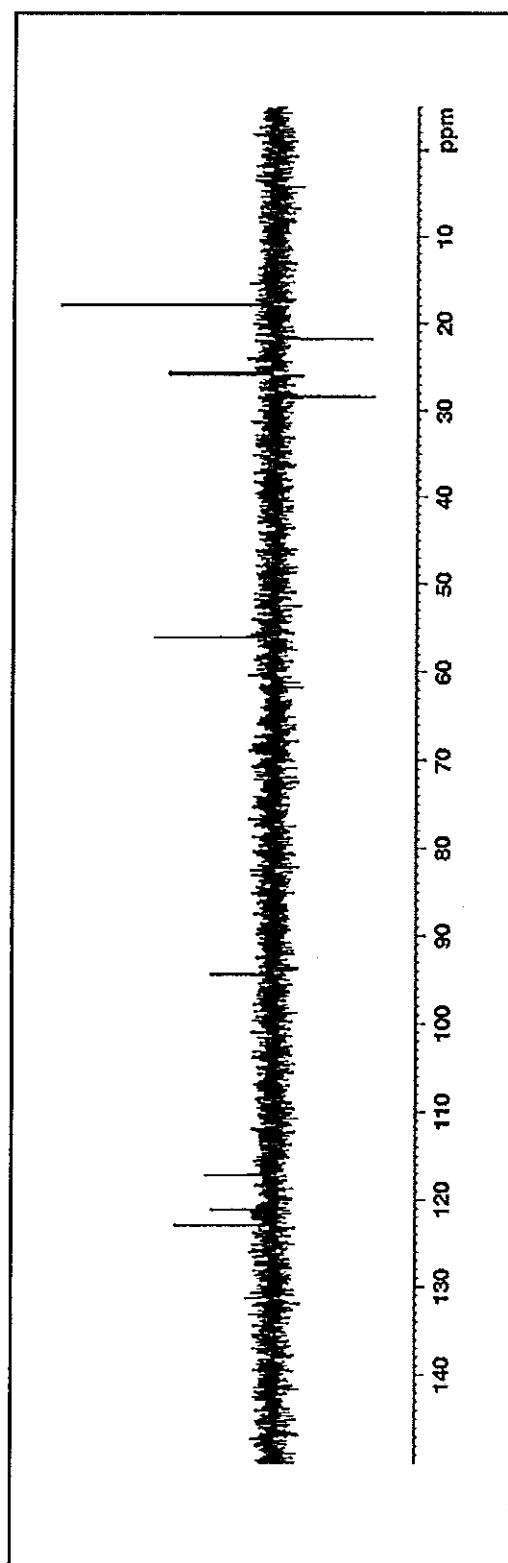


Figure 106 DEPT 135° spectra of compound GP8

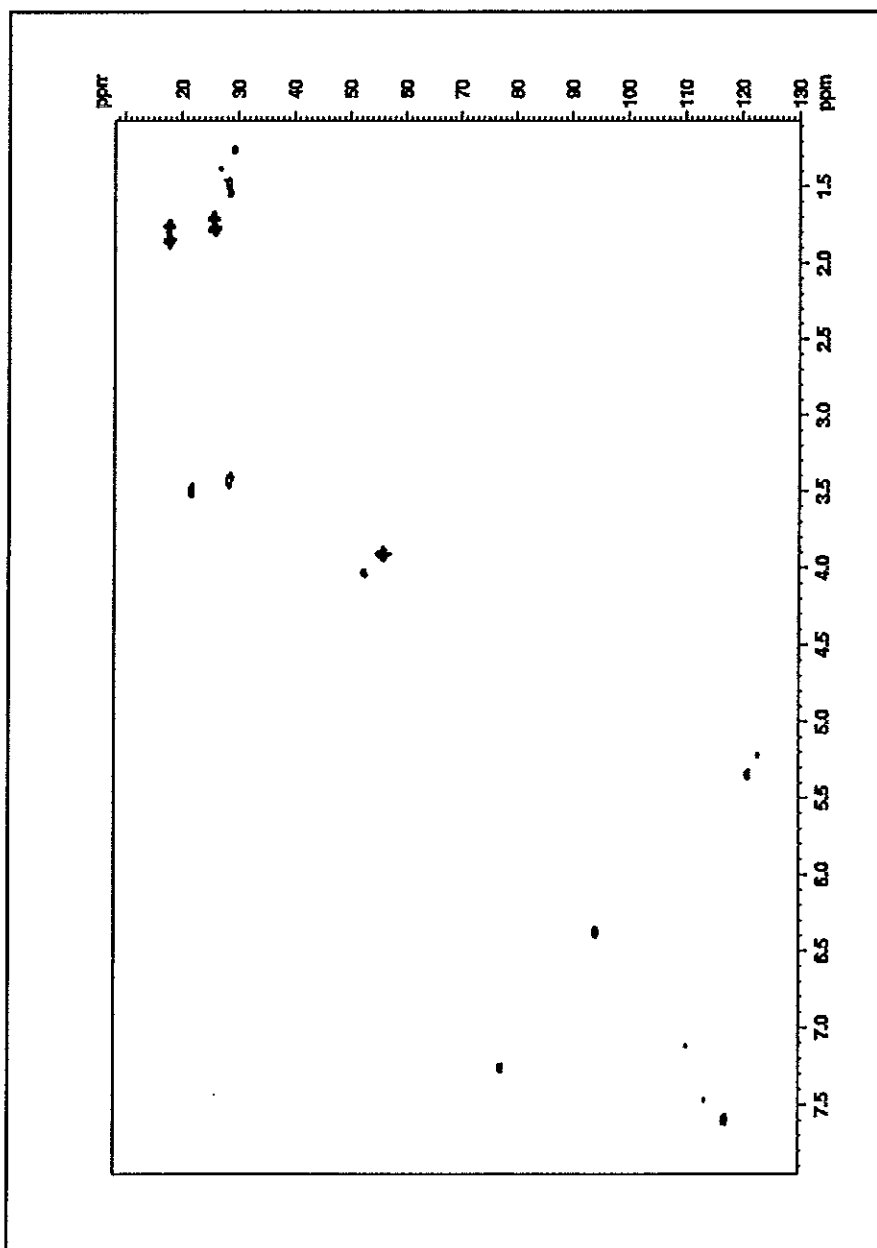


Figure 107 2D HMQC spectrum of compound GP8



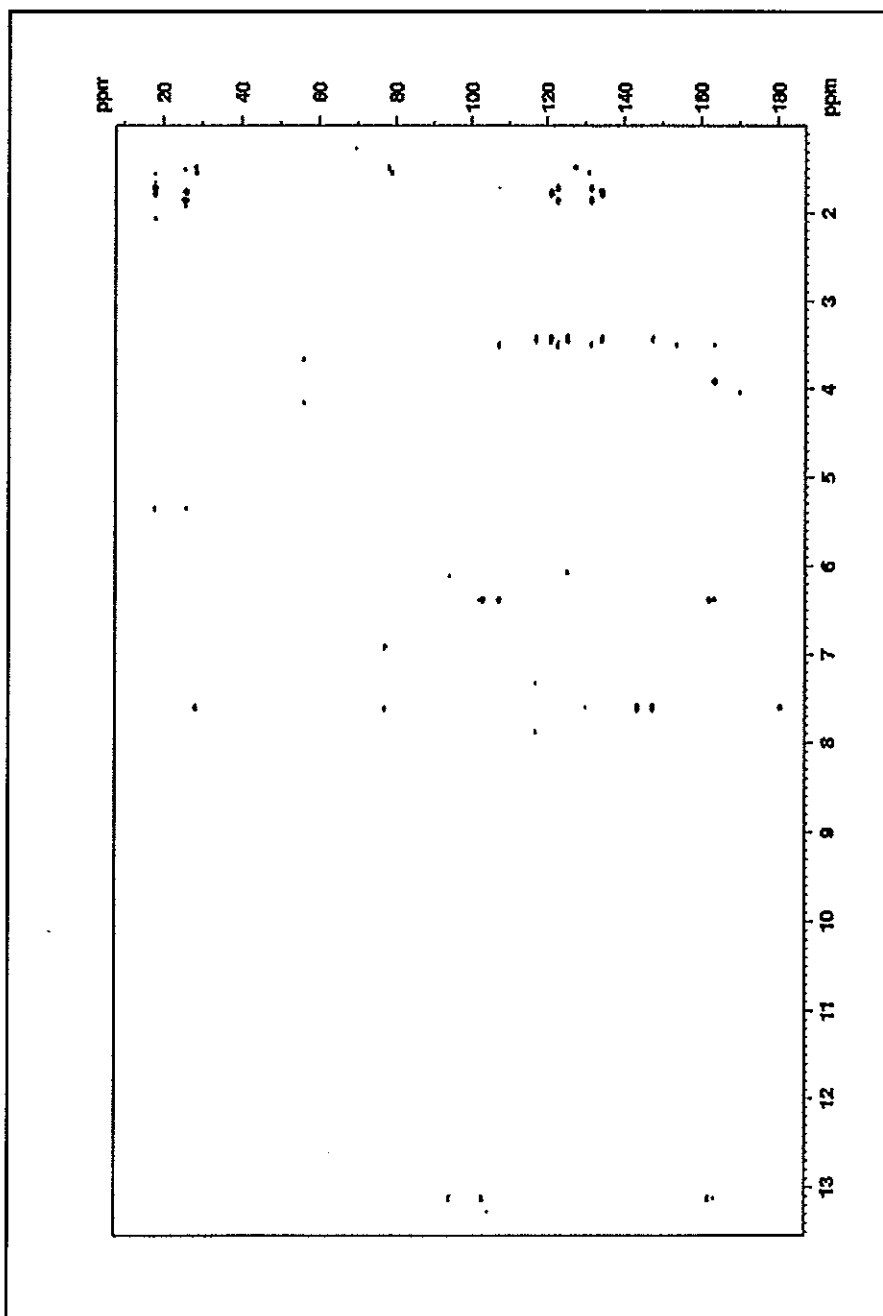


Figure 108 2D HMBC spectrum of compound GP8

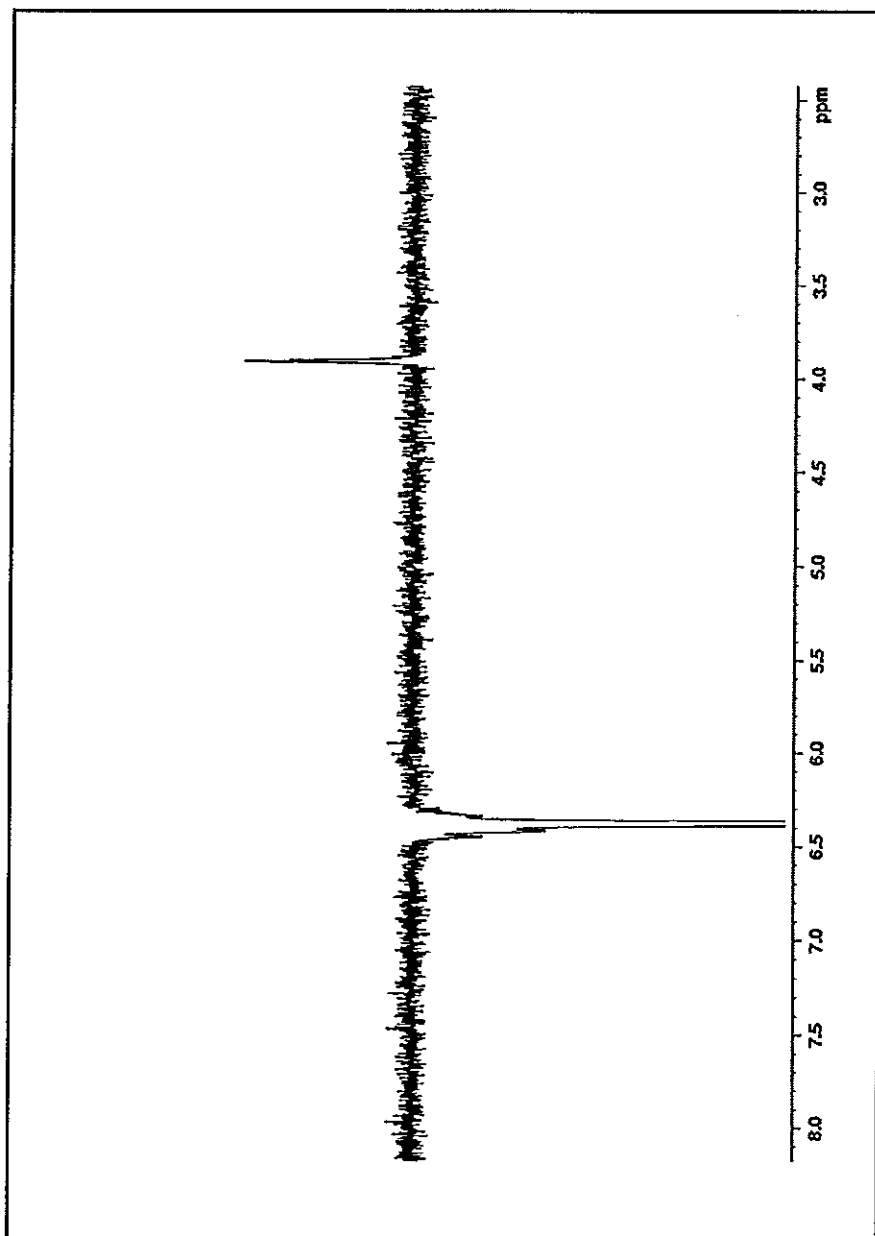


Figure 109 NOE difference spectrum of compound GP8 after irradiation at  $\delta_{\text{H}} 6.37$  (H-2)

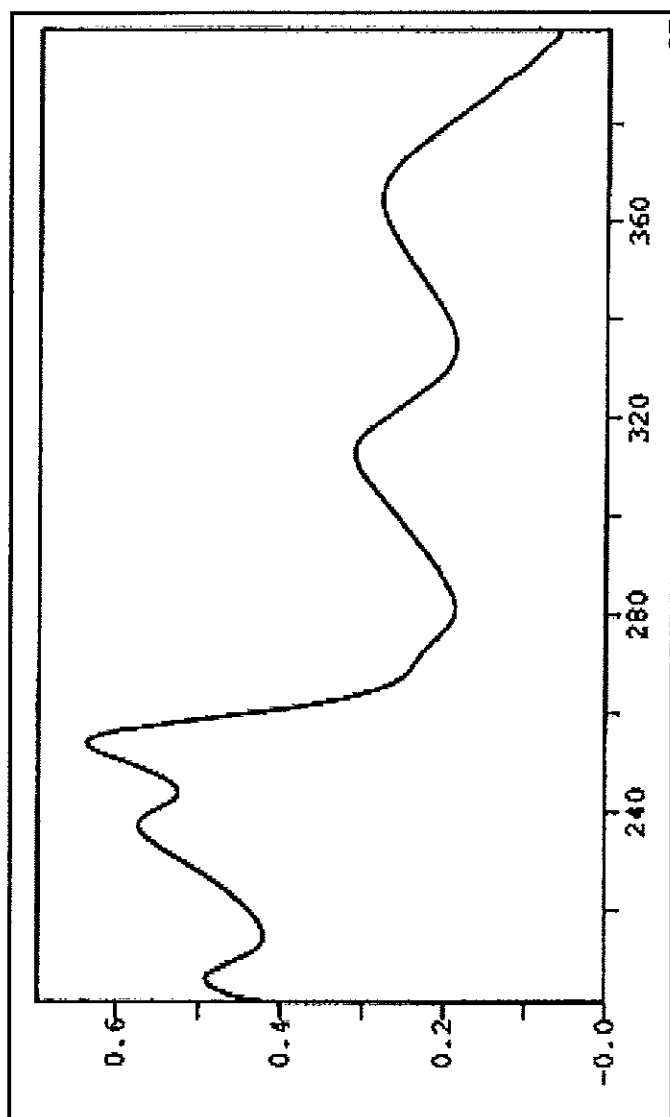


Figure 110 UV (MeOH) spectrum of compound GP20

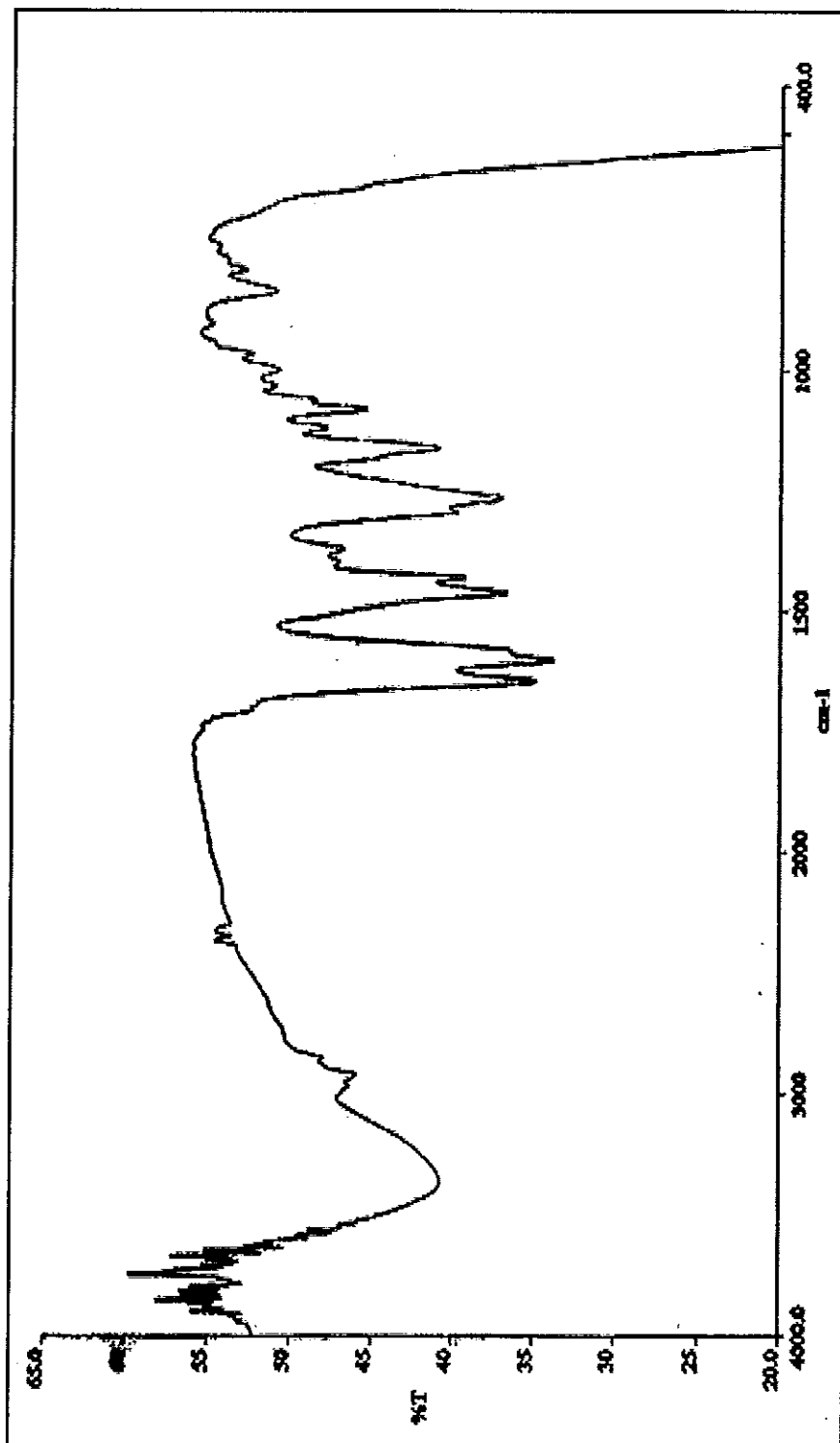


Figure 111 FT-IR (neat) spectrum of compound GP20

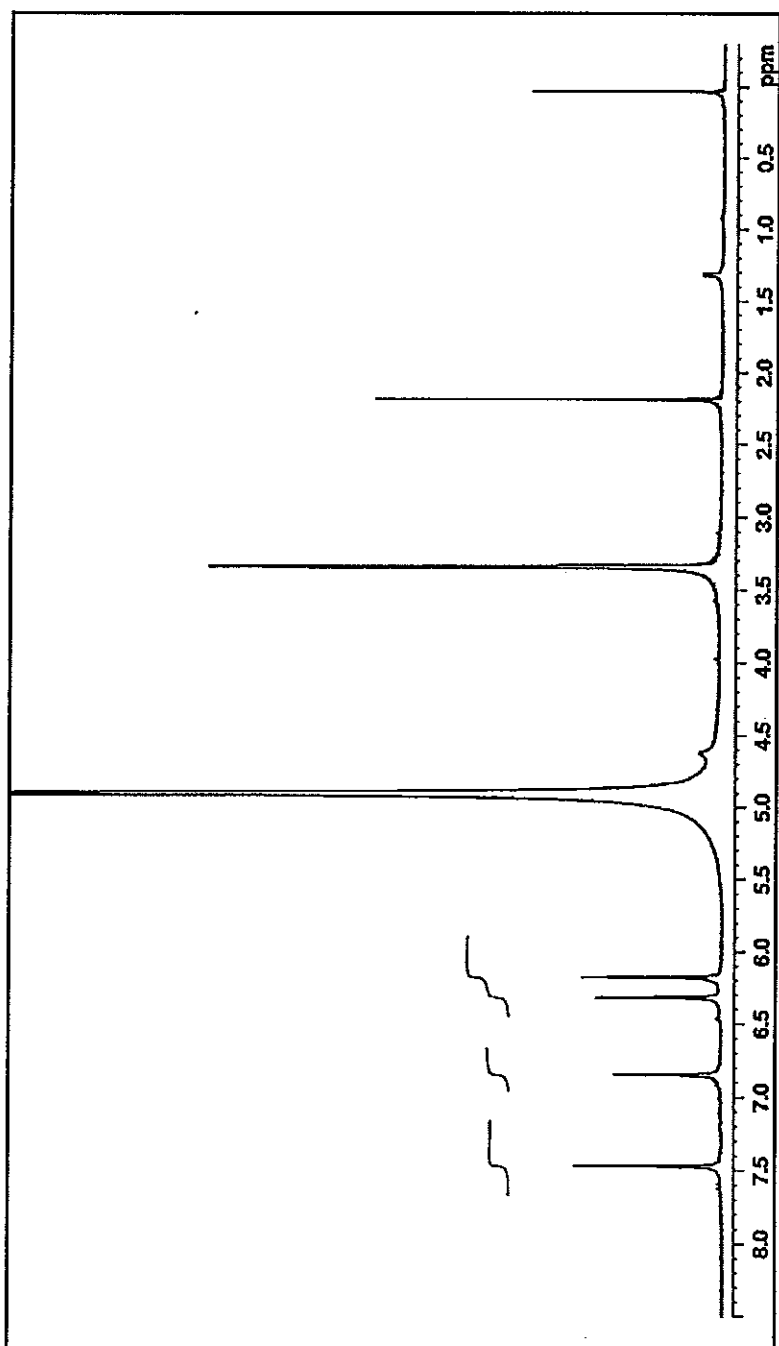


Figure 112  $^1\text{H}$  NMR (300 MHz) ( $\text{CD}_3\text{OD}$ ) spectrum of compound GP20

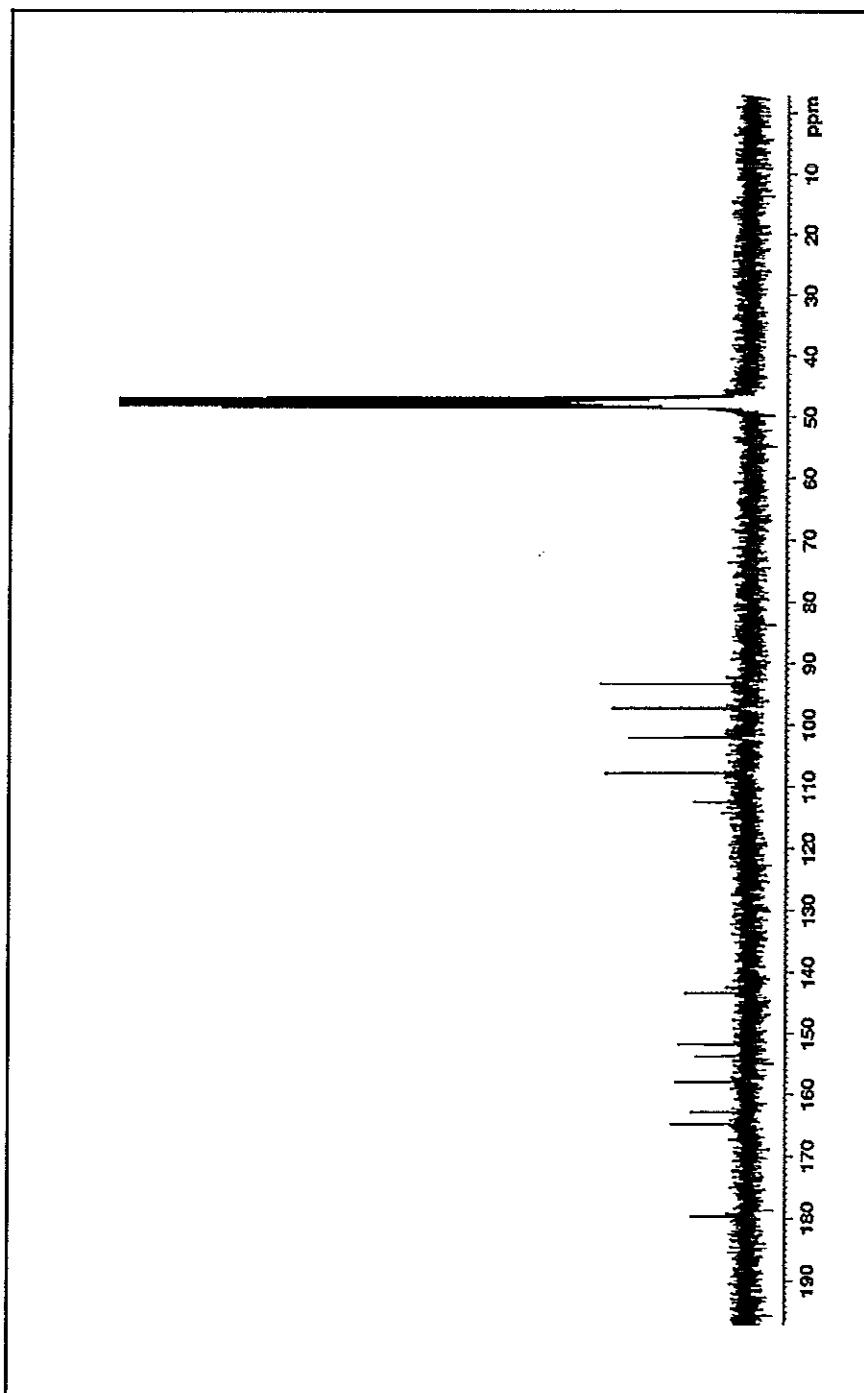


Figure 113  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CD}_3\text{OD}$ ) spectrum of compound GP20

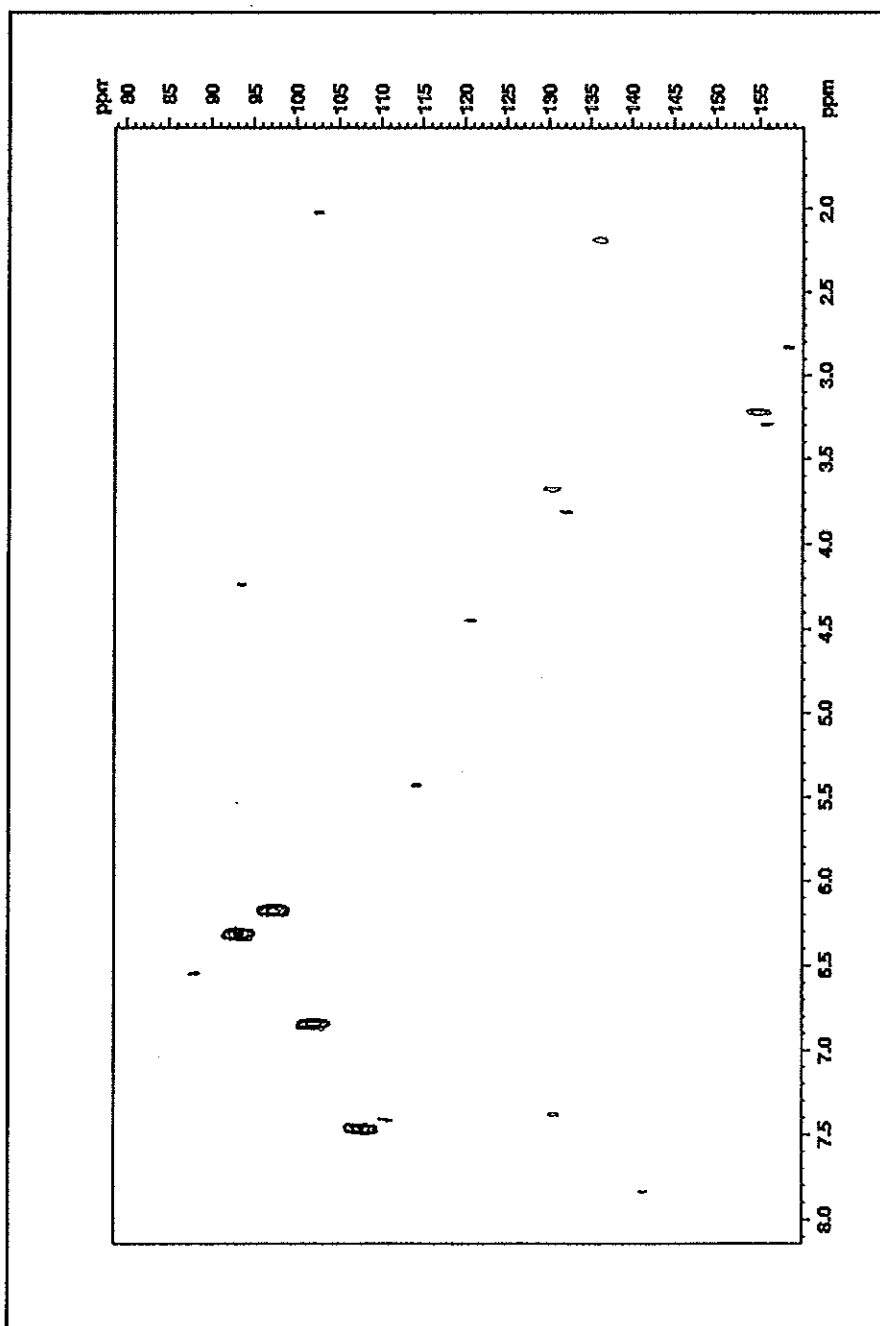


Figure 114 2D HMQC spectrum of compound GP20

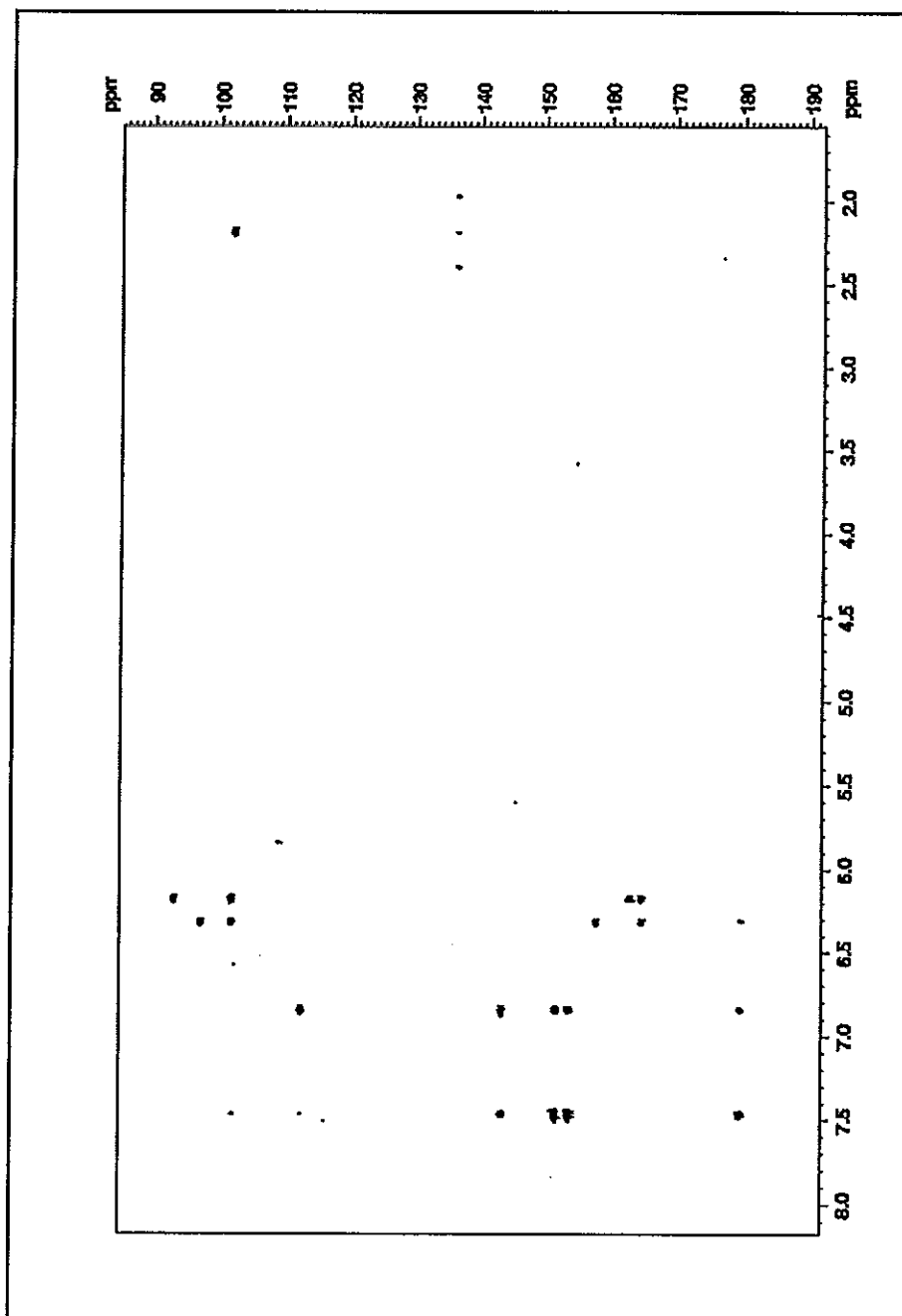


Figure 115 2D HMBC spectrum of compound GP20



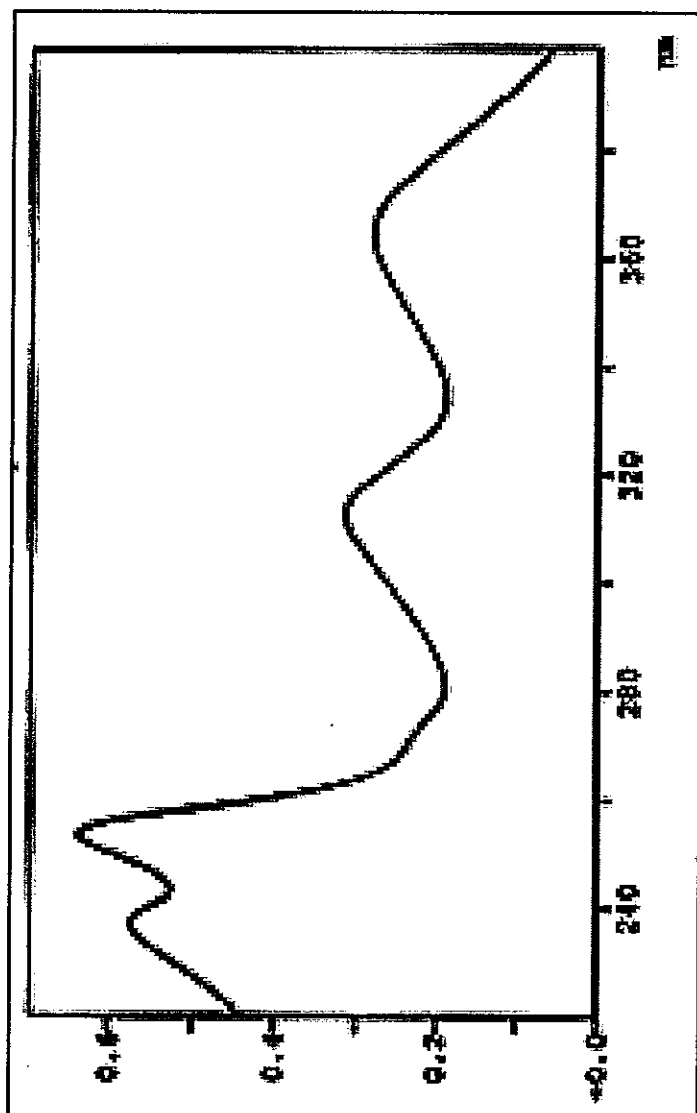


Figure 116 UV (MeOH) spectrum of compound GP19

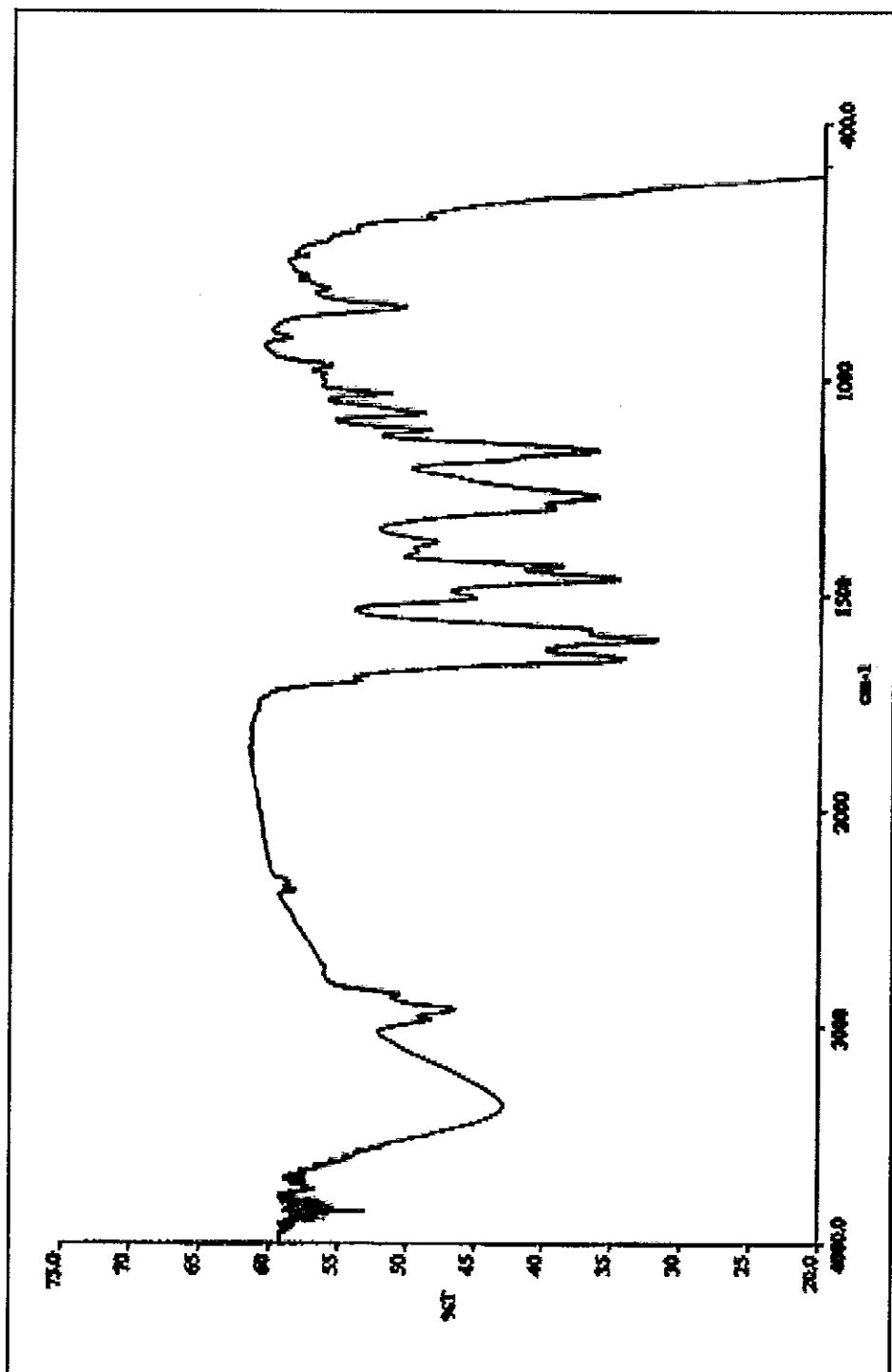


Figure 117 FT-IR (neat) spectrum of compound GP19

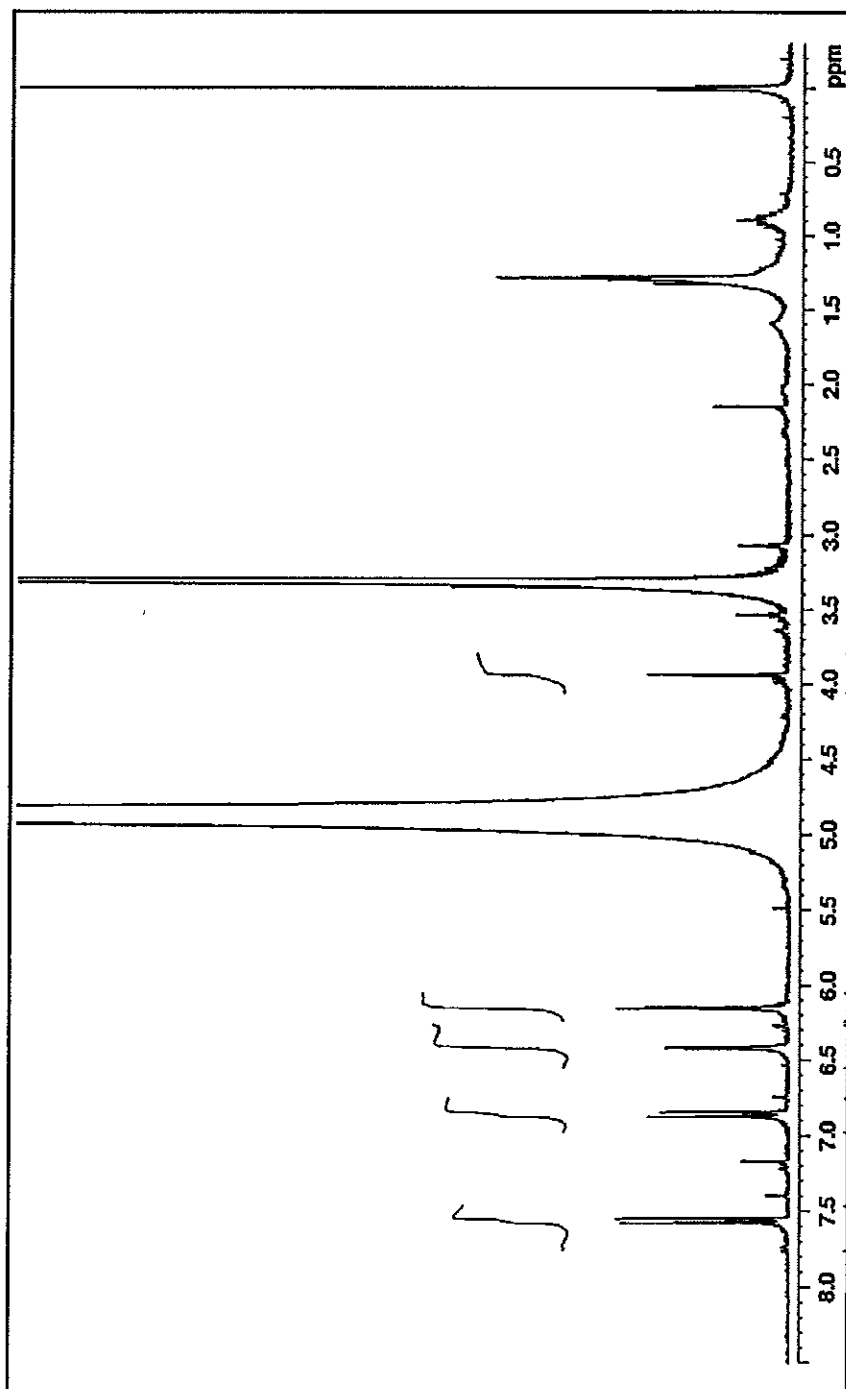


Figure 118  $^1\text{H}$  NMR (300 MHz) ( $\text{CD}_3\text{OD}$ ) spectrum of compound GP19

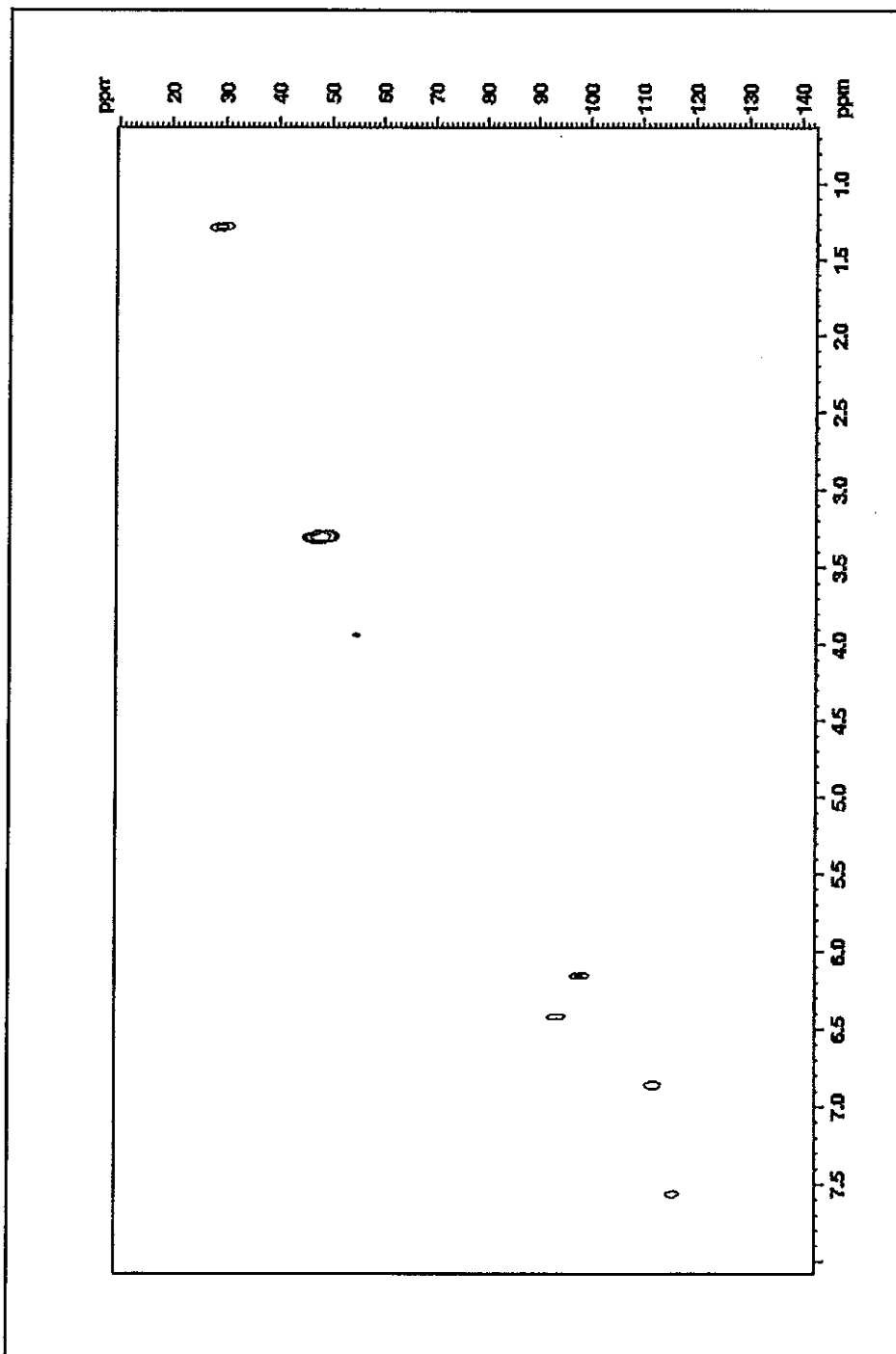


Figure 119 2D HMQC spectrum of compound GP19

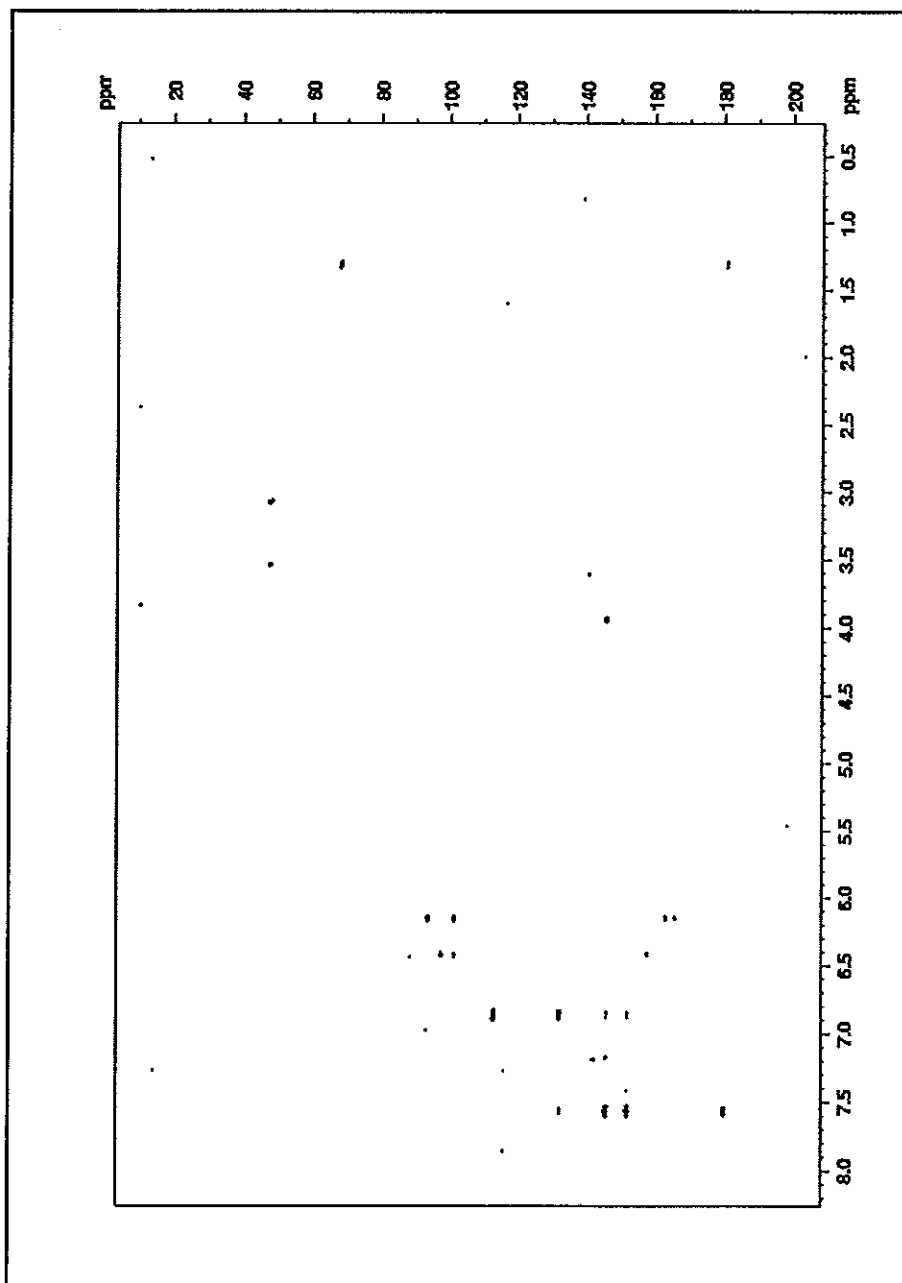


Figure 120 2D HMBC spectrum of compound GP19

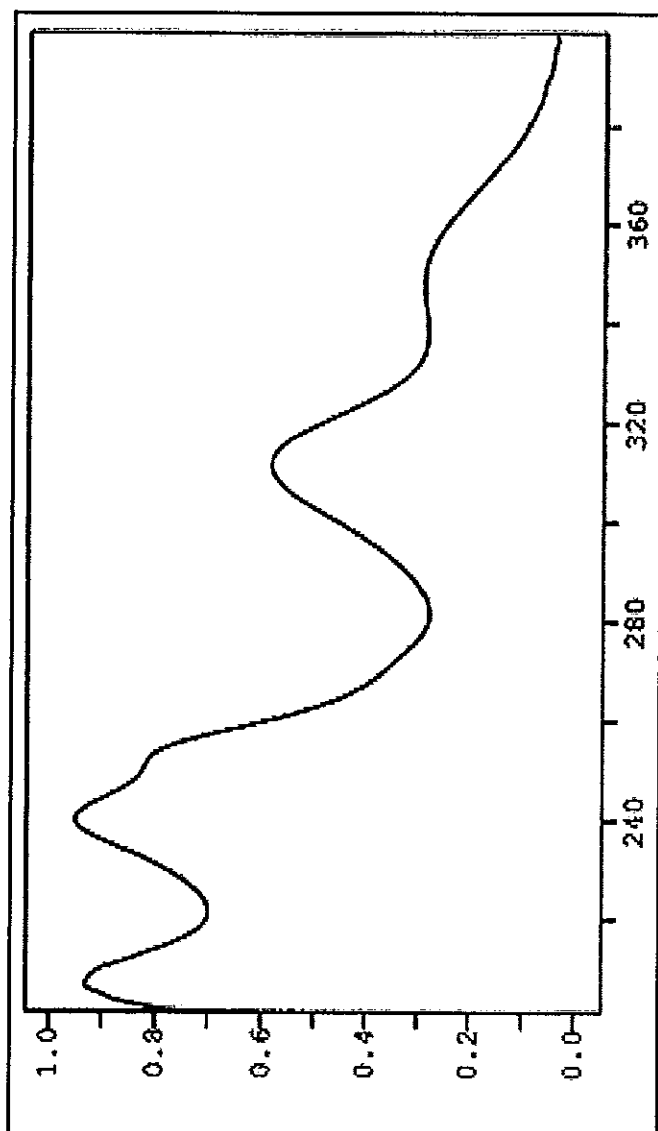


Figure 121 UV (MeOH) spectrum of compound GP17

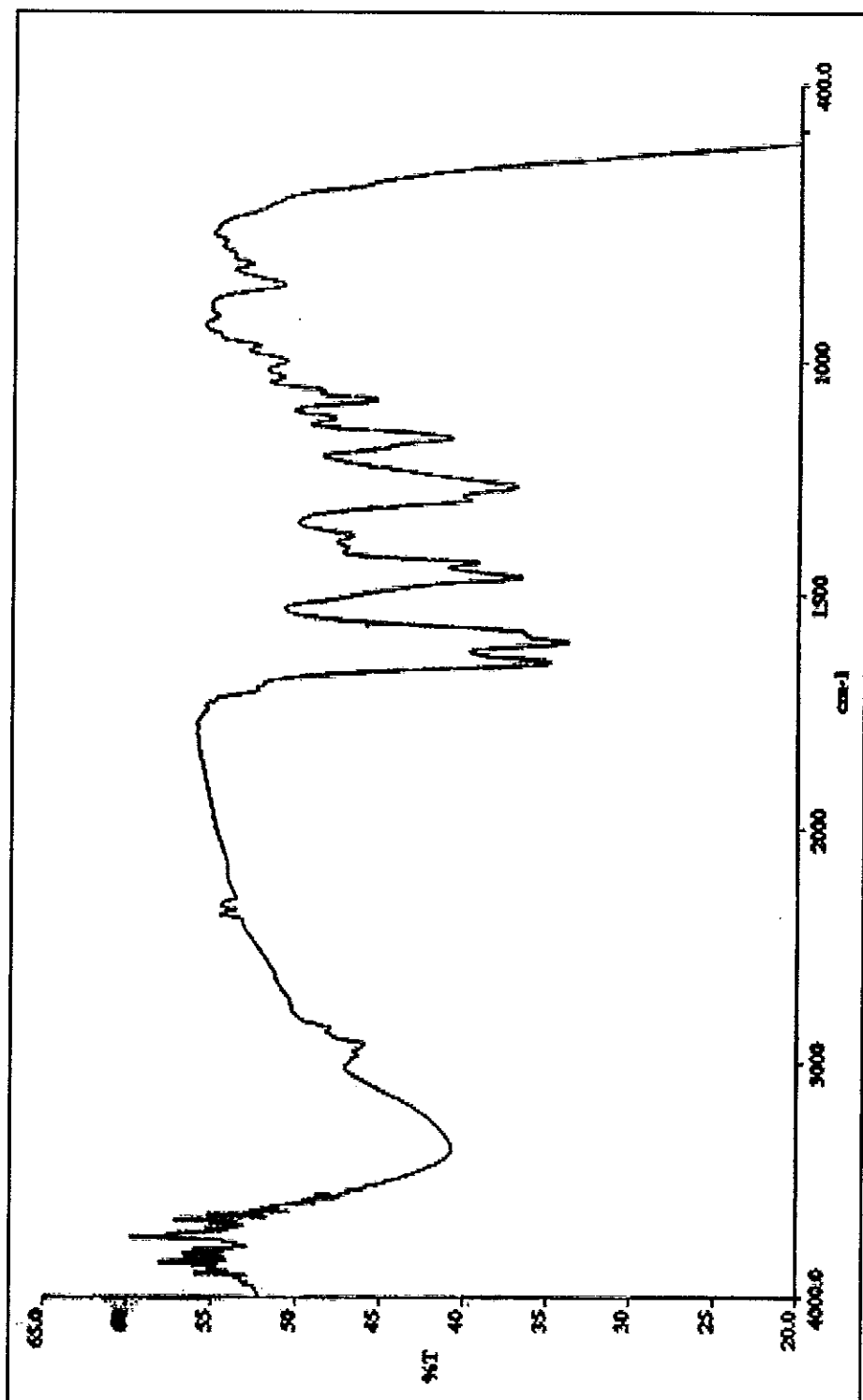


Figure 122 FT-IR (neat) spectrum of compound GP17

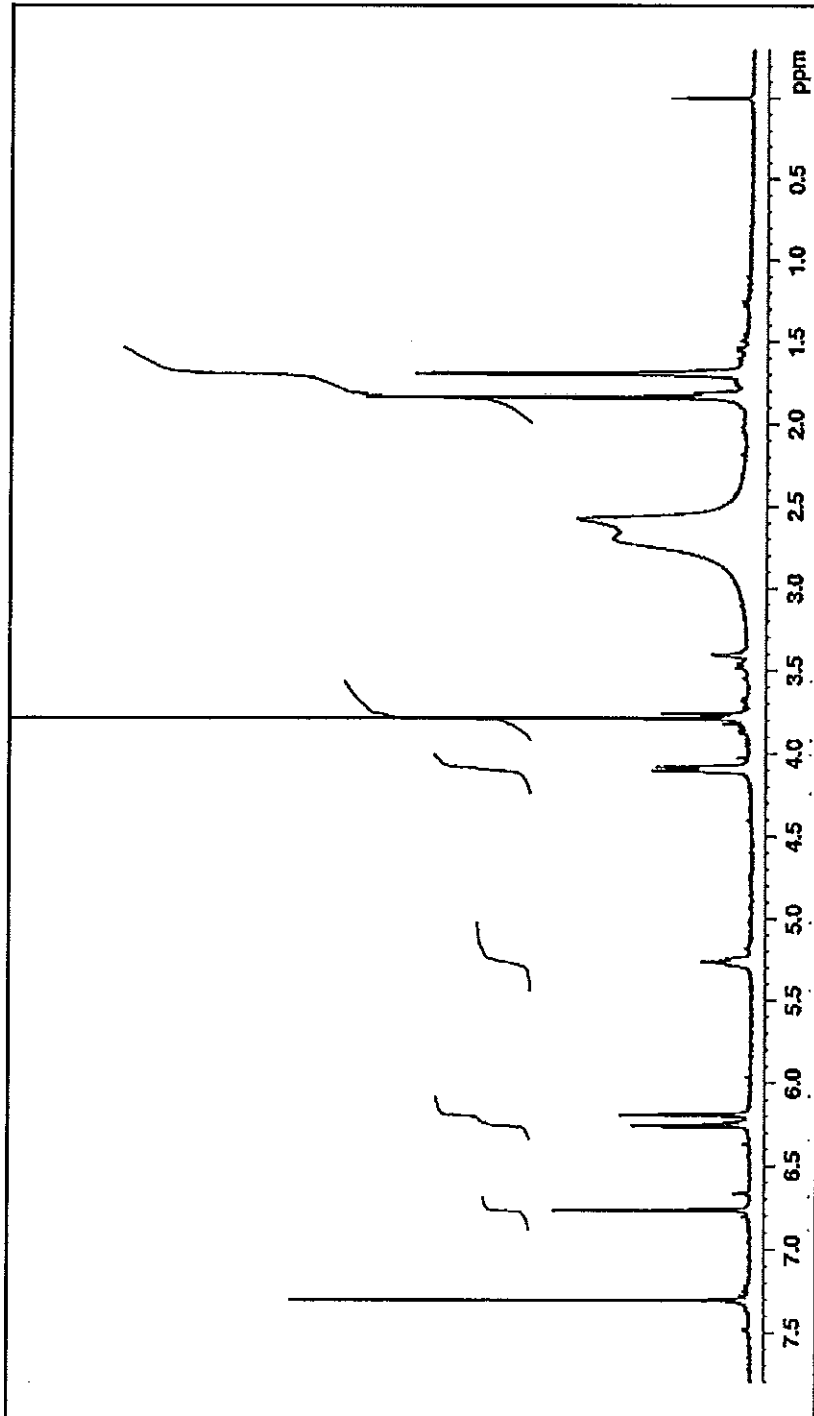


Figure 123  $^1\text{H}$  NMR (300 MHz) ( $\text{CD}_3\text{Cl}+\text{CD}_3\text{OD}$ ) spectrum of compound GPI7



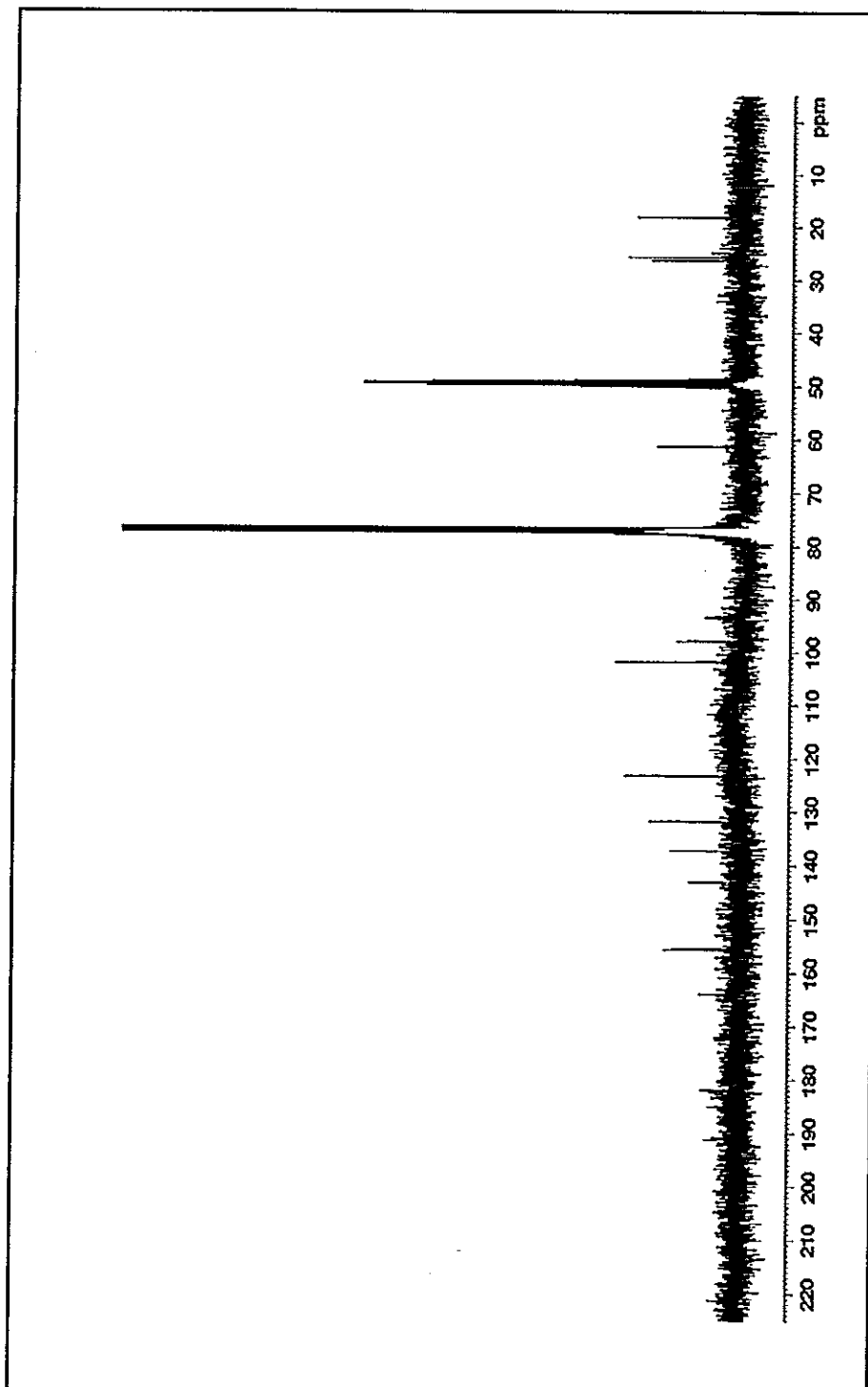


Figure 124  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CD}_3\text{Cl}$ - $\text{CD}_3\text{OD}$ ) spectrum of compound GP17

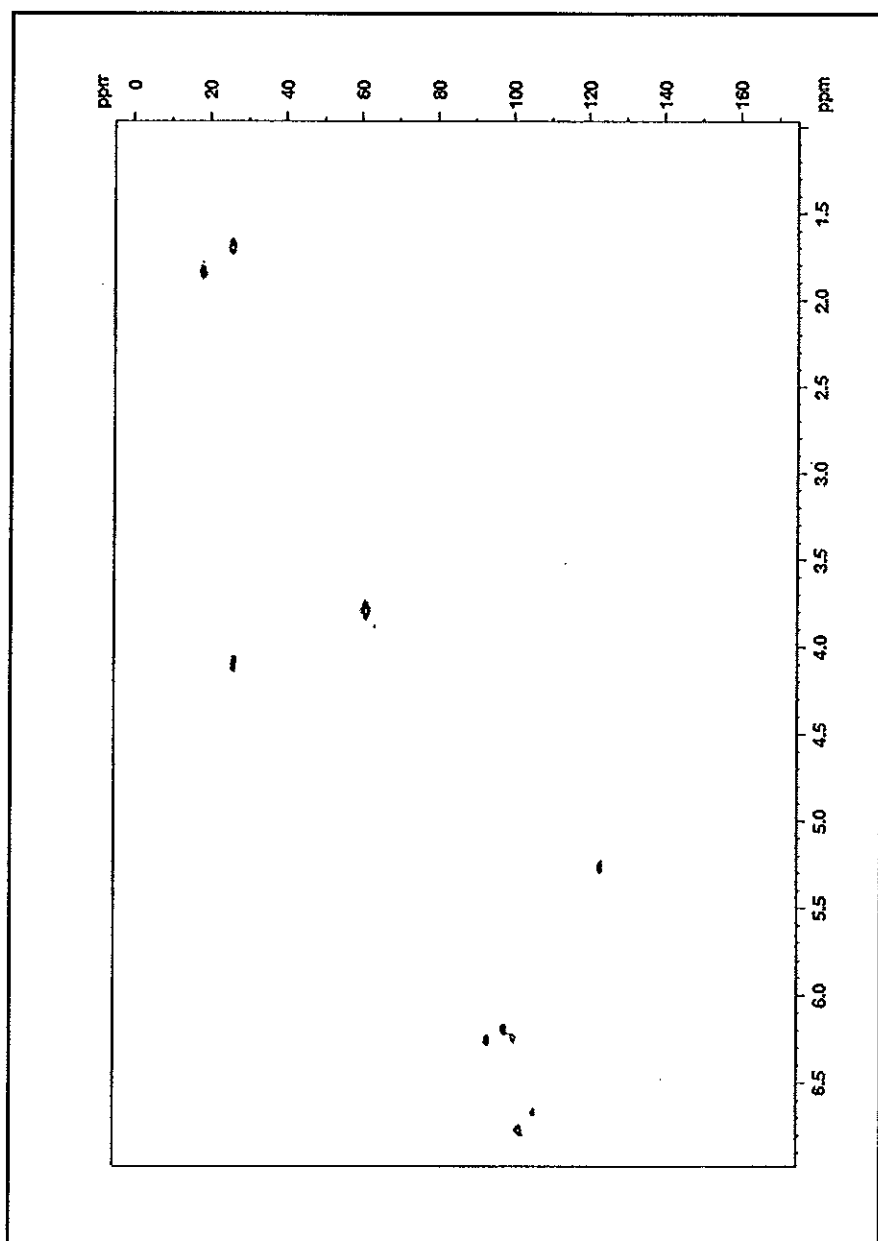


Figure 125 2D HMQC spectrum of compound GP17

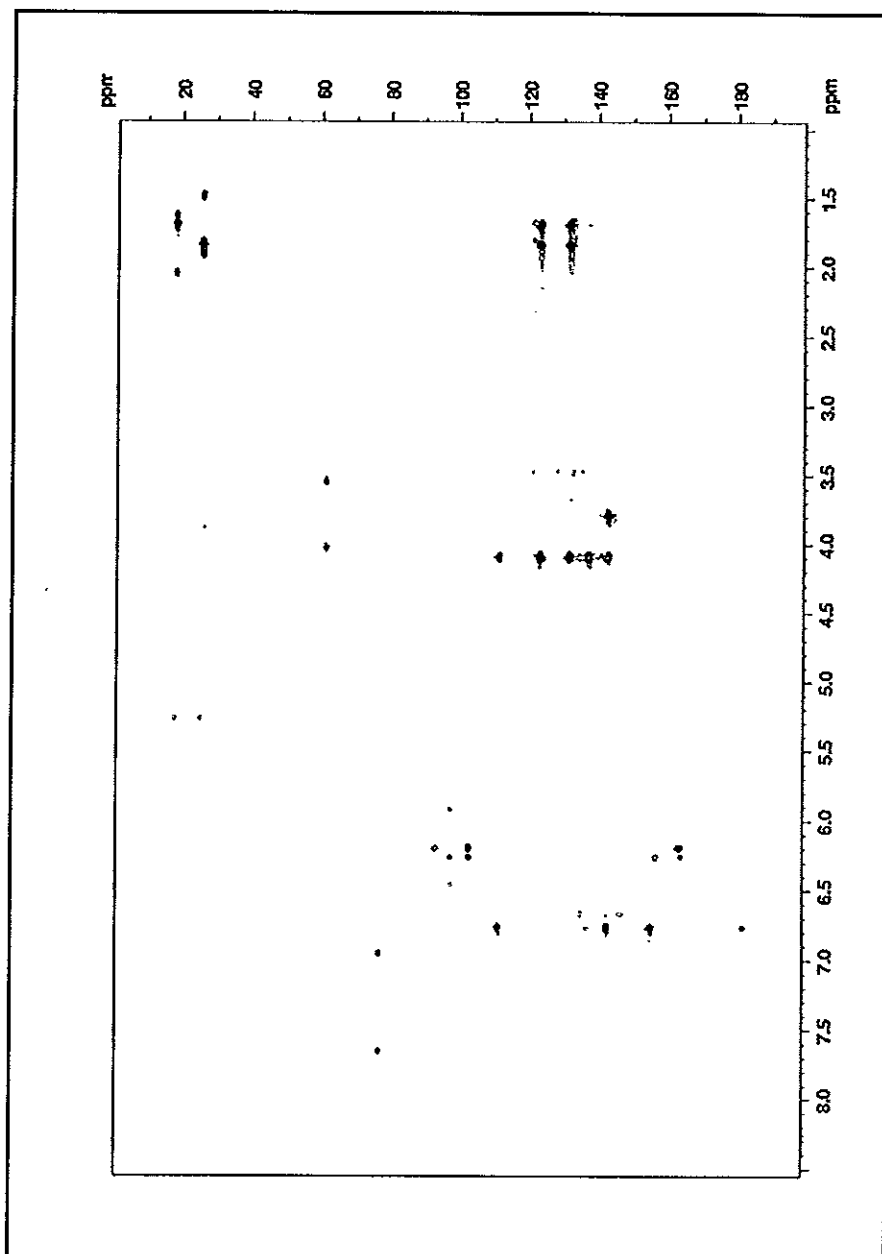


Figure 126 2D HMBC spectrum of compound GP17

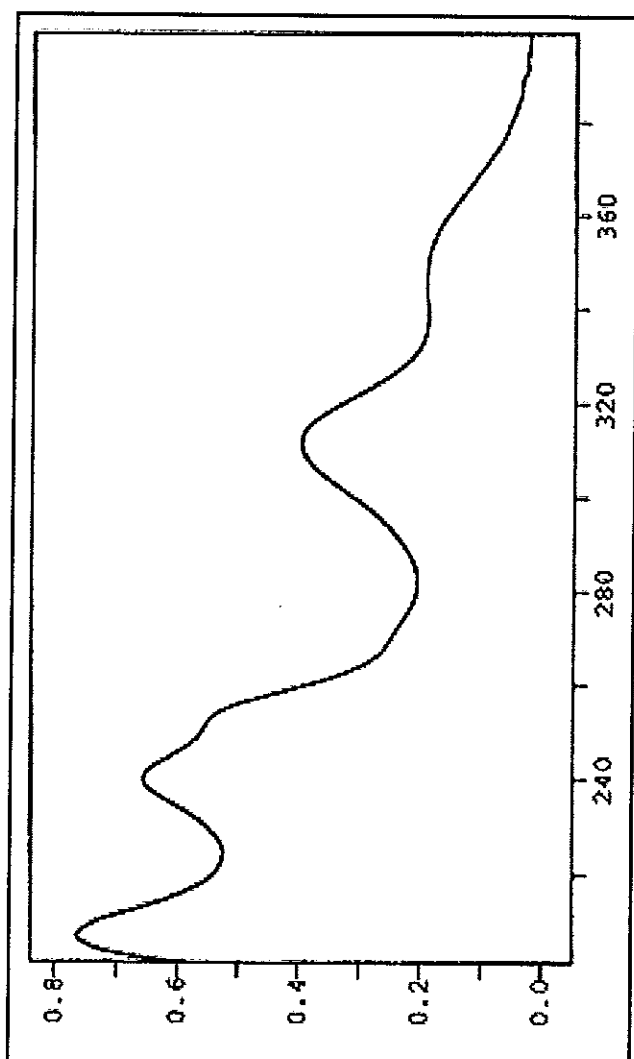


Figure 127 UV (MeOH) spectrum of compound GP16

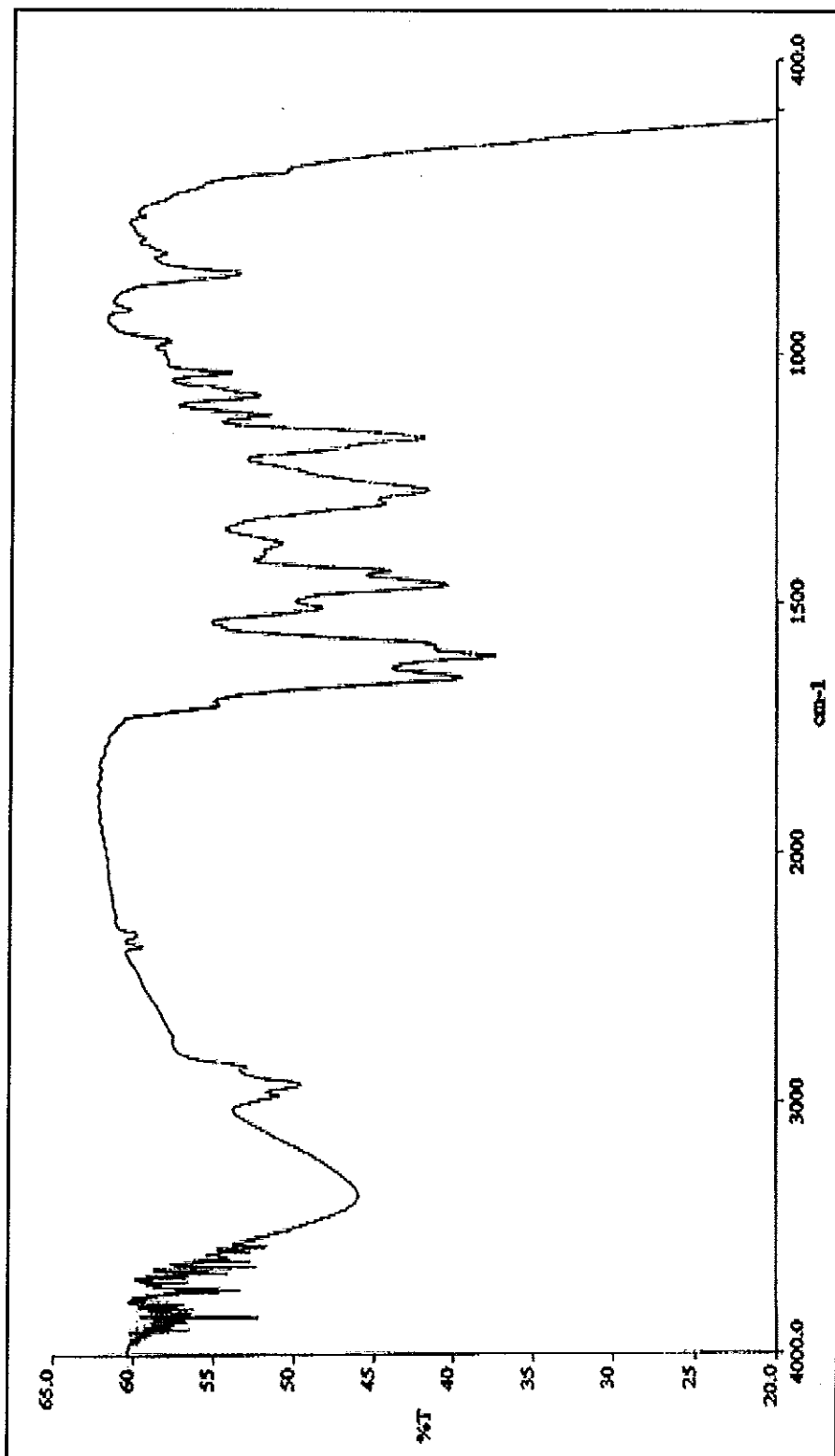


Figure 128 FT-IR (neat) spectrum of compound GP16

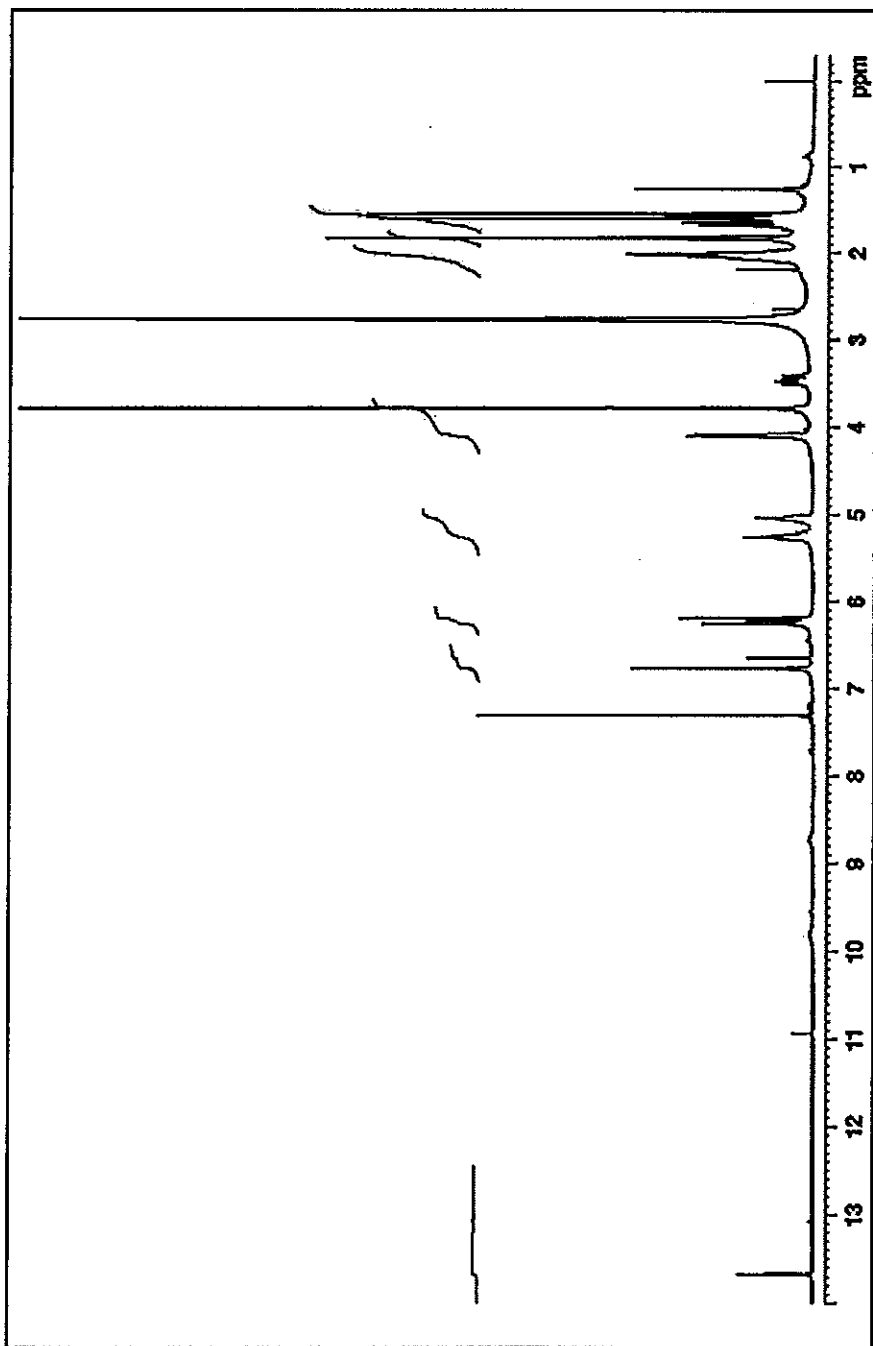


Figure 129  $^1\text{H}$  NMR (300 MHz) ( $\text{CD}_3\text{Cl}+\text{CD}_3\text{OD}$ ) spectrum of compound GP16

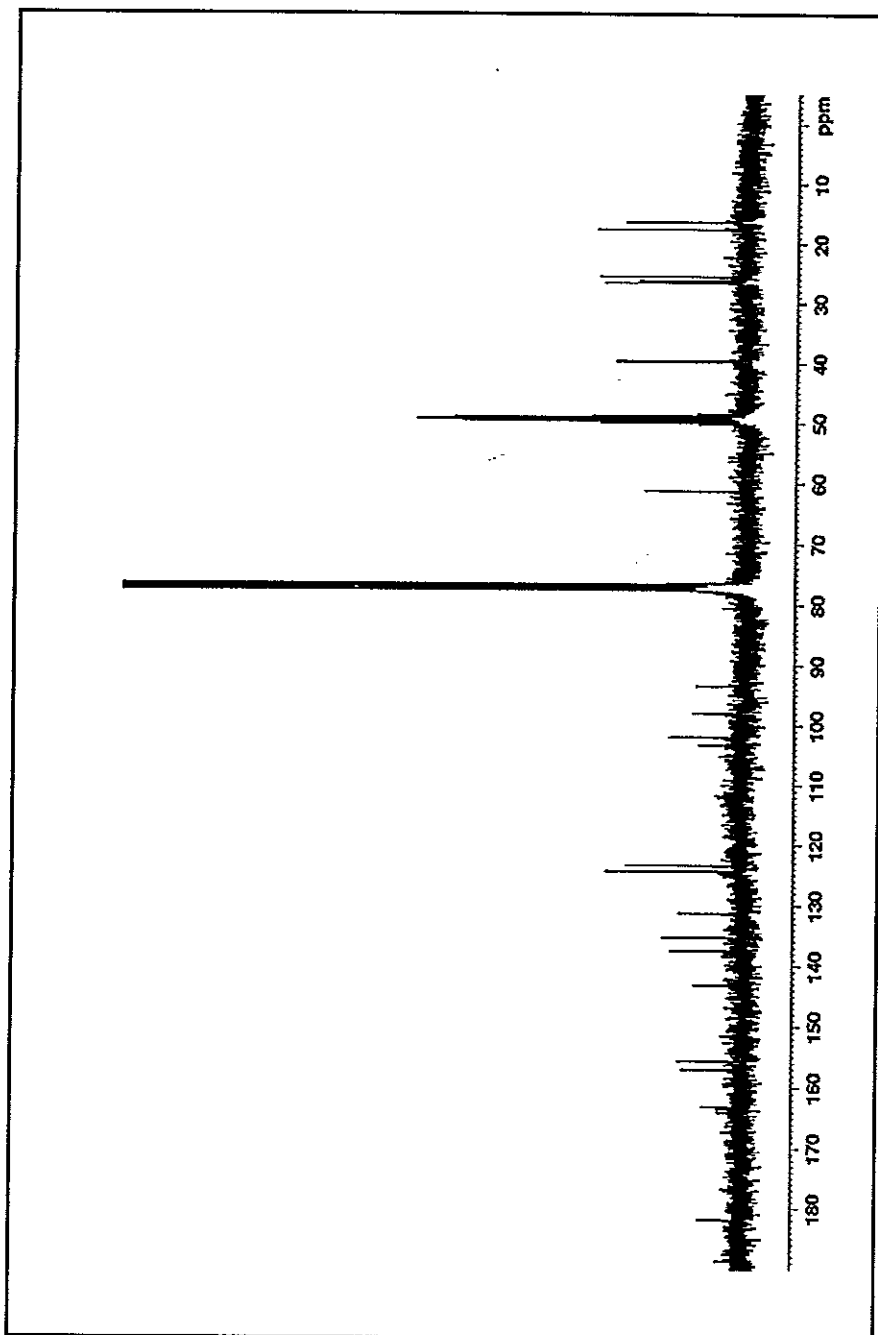


Figure 130  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CD}_3\text{Cl}+\text{CD}_3\text{OD}$ ) spectrum of compound GP16

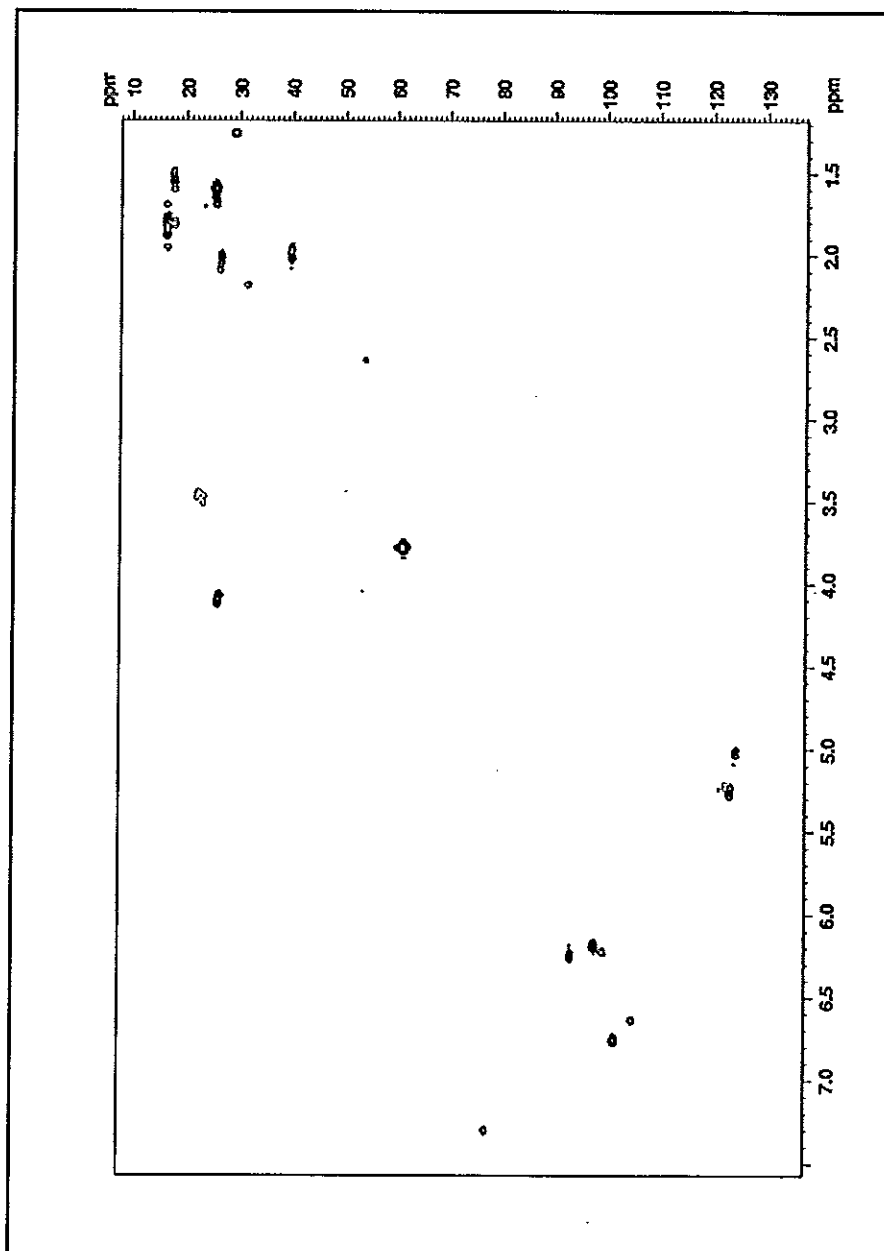


Figure 131 2D HMQC spectrum of compound GP16



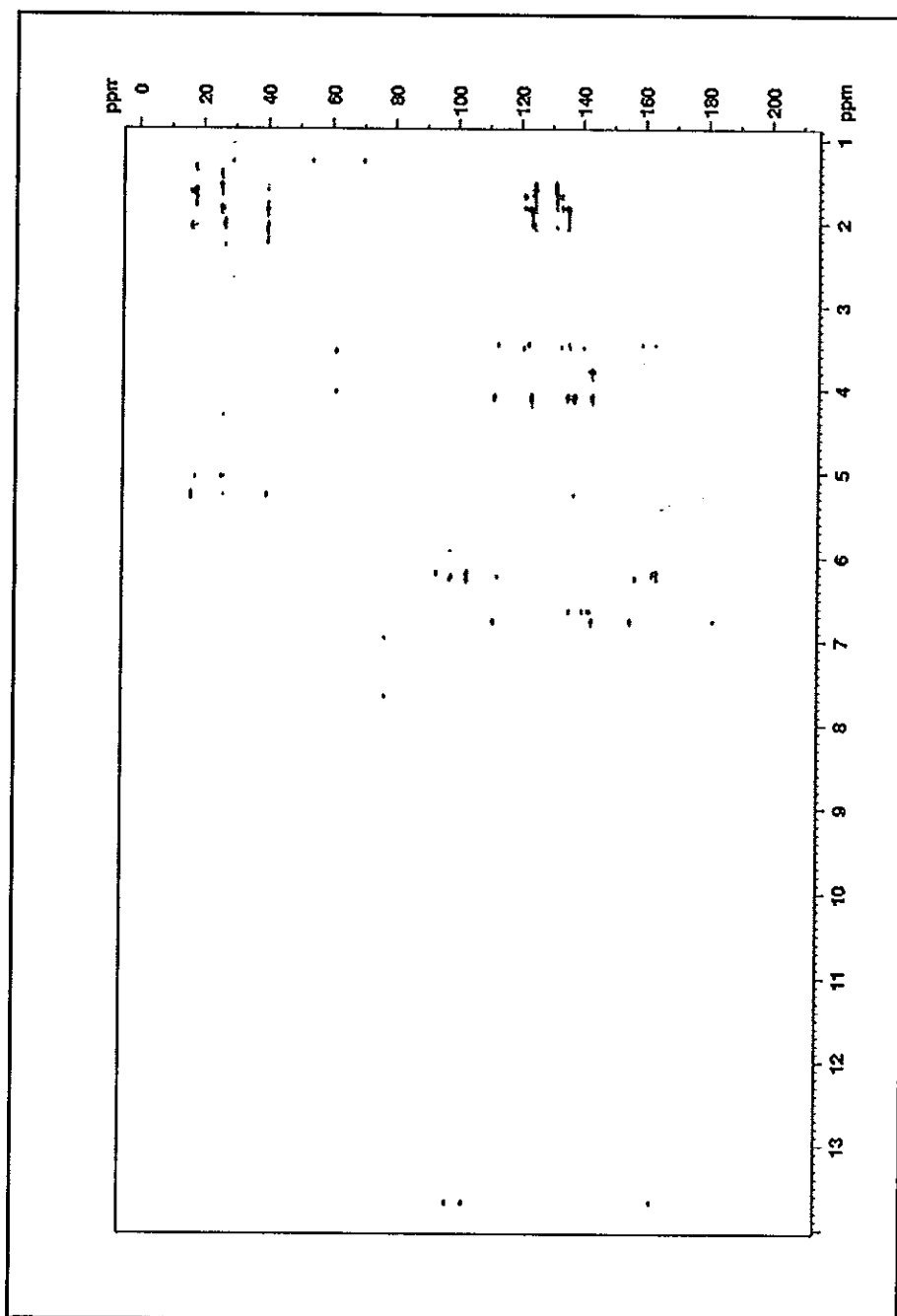


Figure 132 2D HMBC spectrum of compound GP16

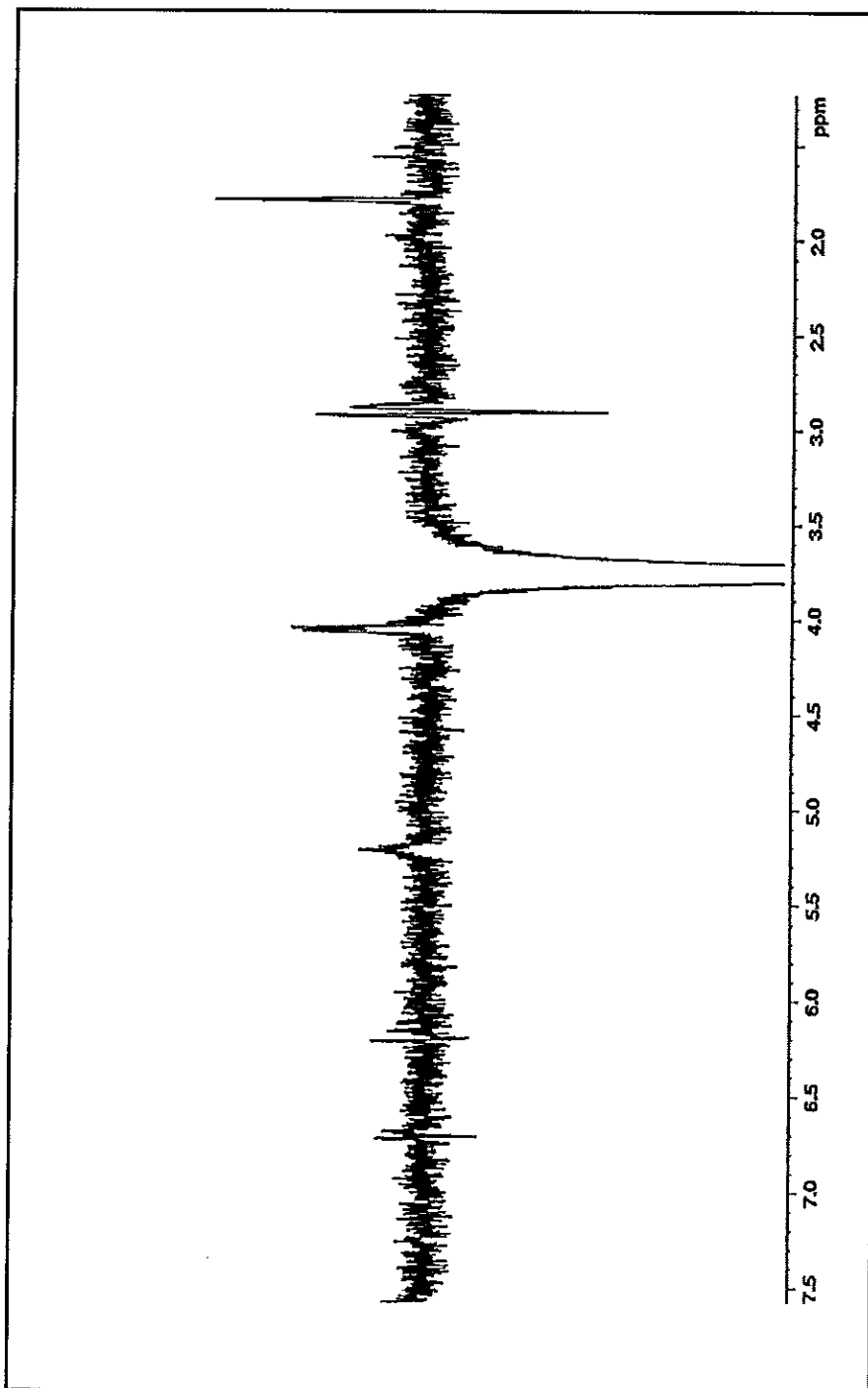


Figure 133 NOE difference spectrum of compound GP16 after irradiation at  $\delta_{\text{H}} 3.78$  (7-OCH<sub>3</sub>)

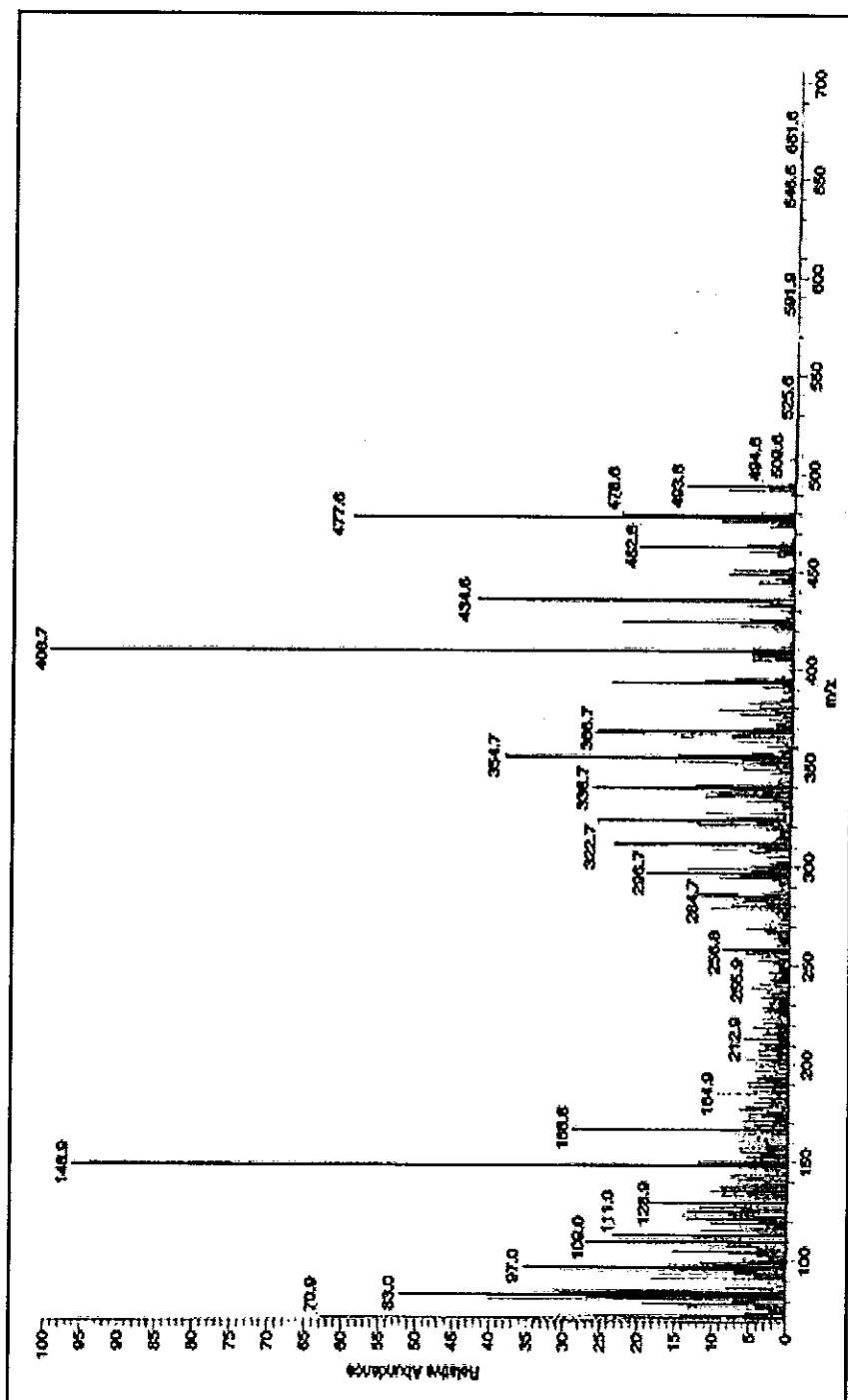


Figure 134 Mass spectrum of compound GPII

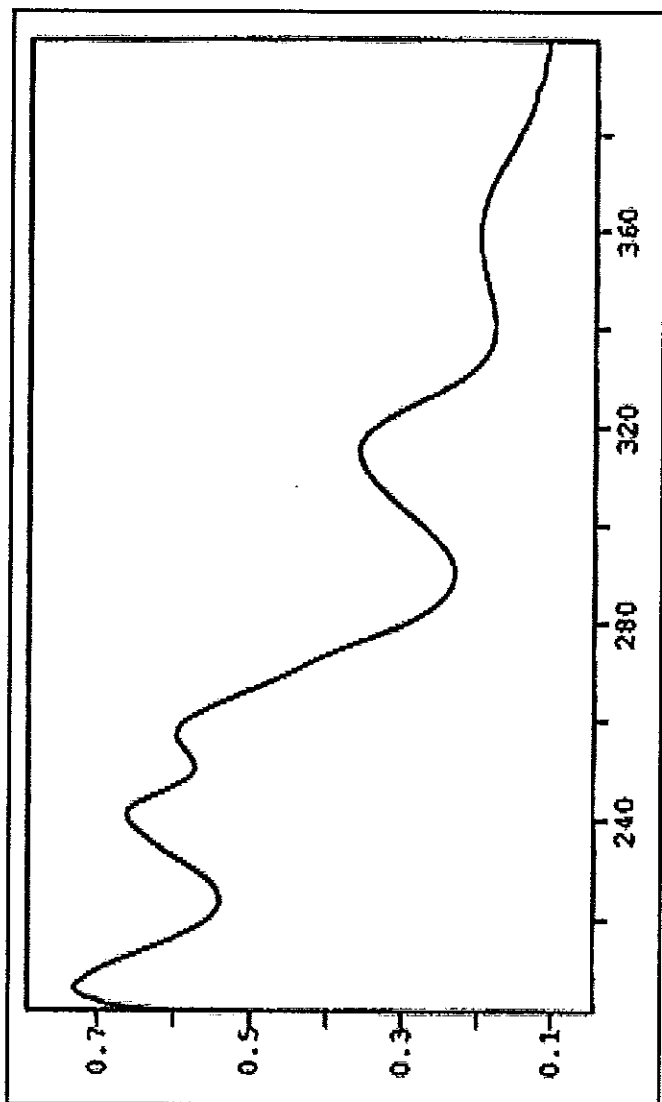


Figure 135 UV (MeOH) spectrum of compound GP11

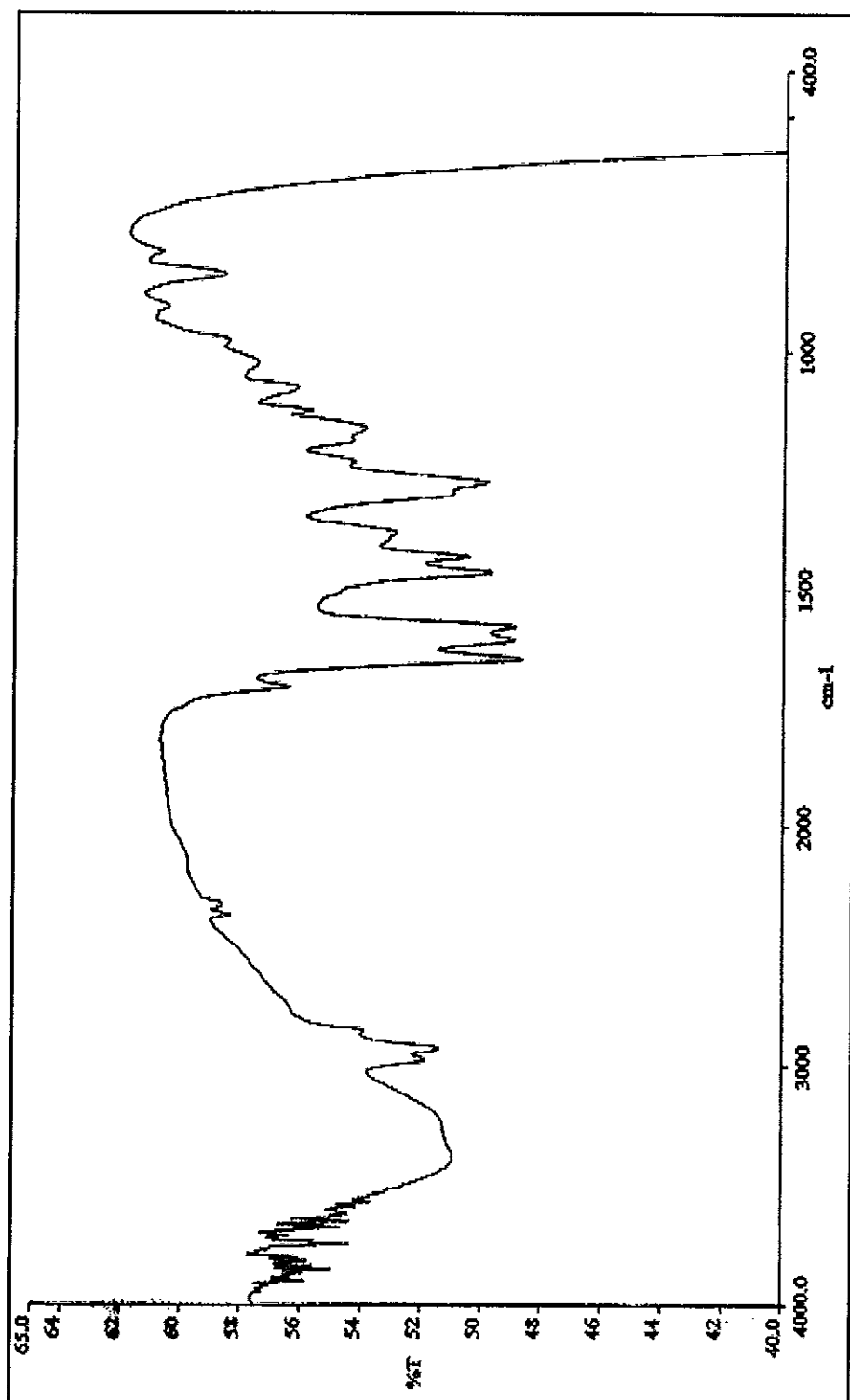


Figure 136 FT-IR (neat) spectrum of compound GP11 .

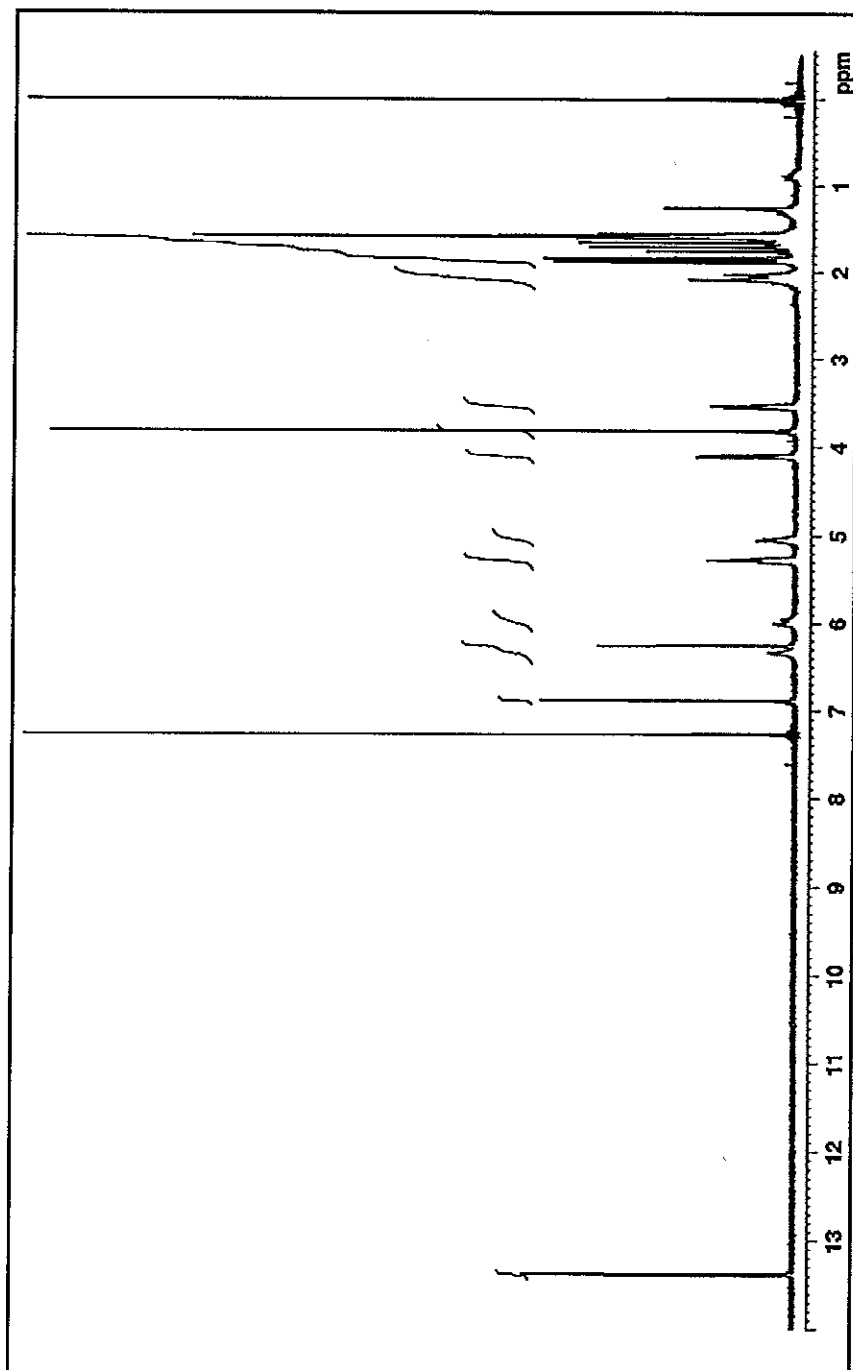


Figure 137  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound GP11

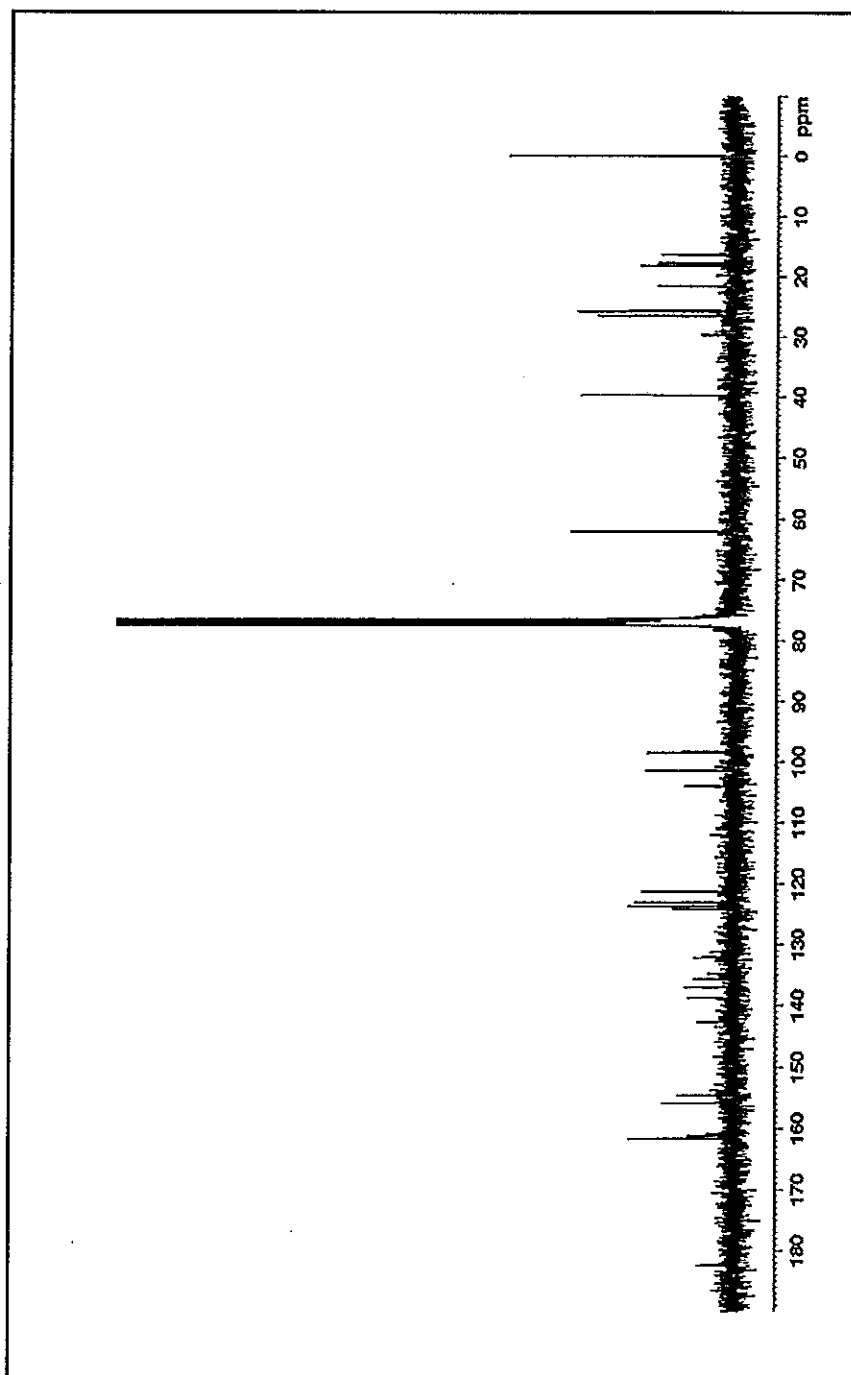


Figure 138  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound GP11

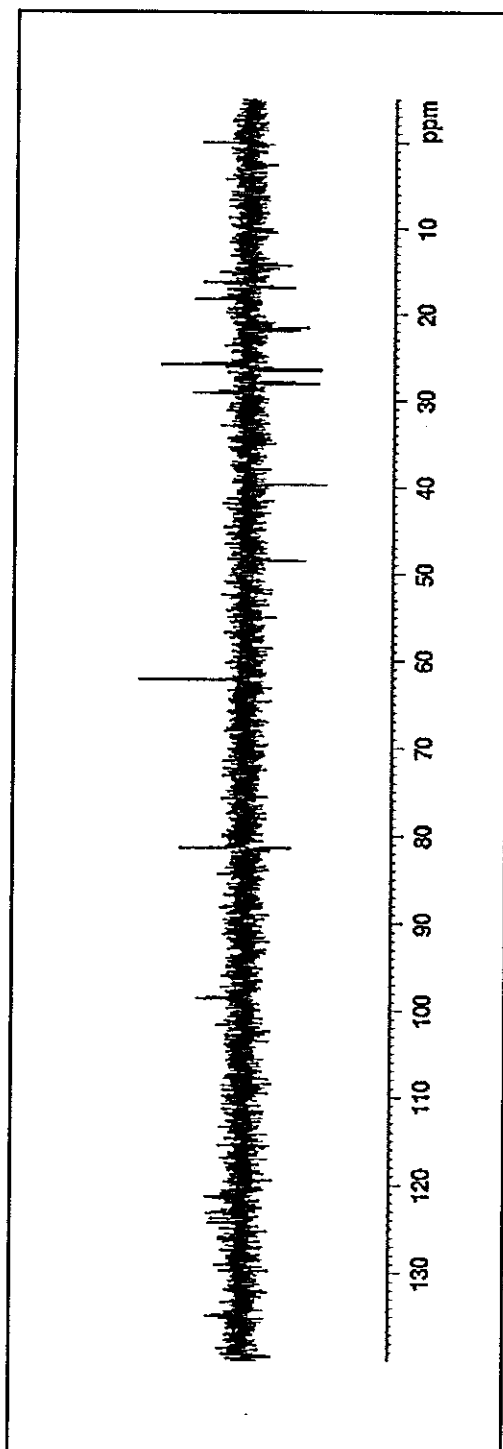


Figure 139 DEPT 135° spectra of compound GP11



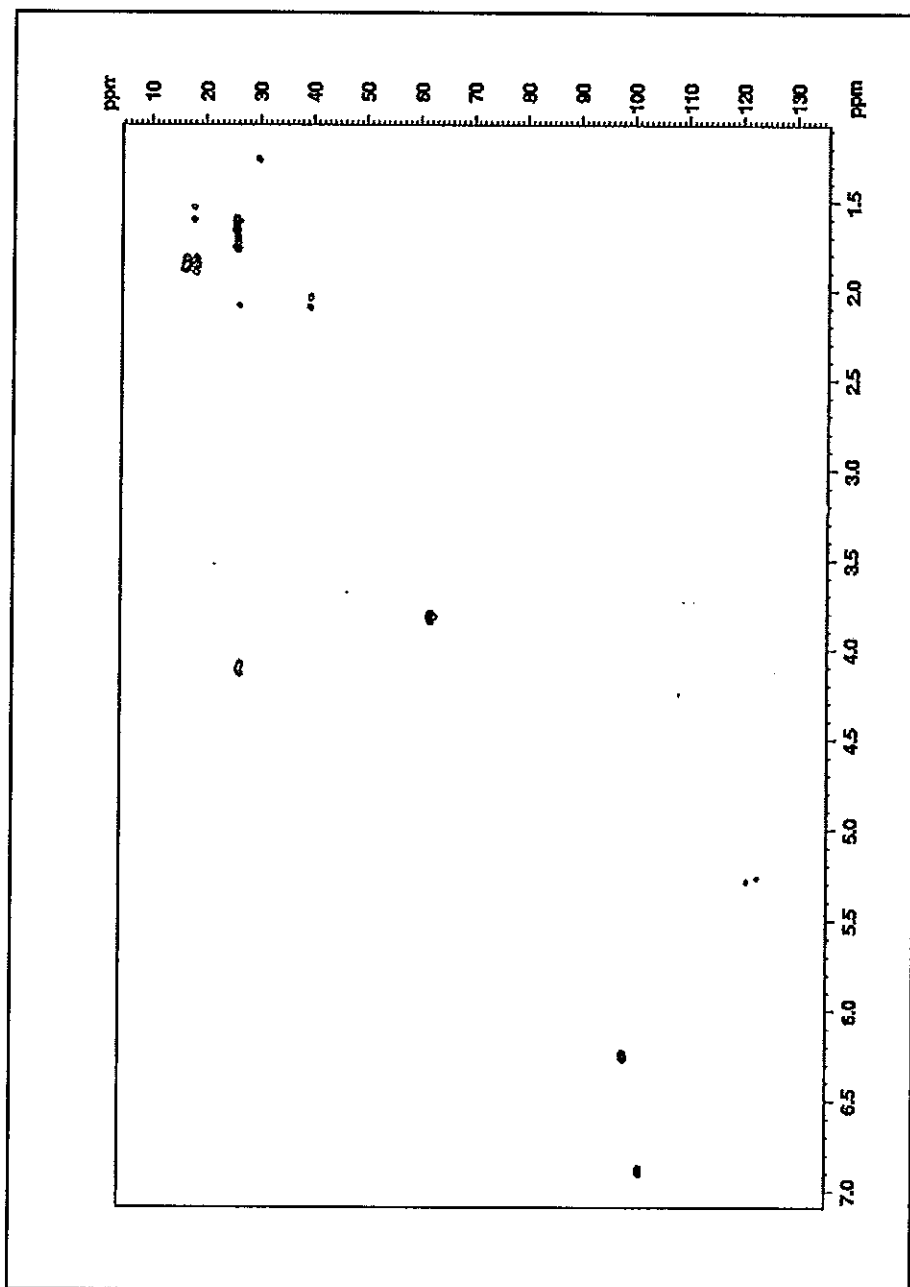


Figure 140 2D HMQC spectrum of compound GPI I

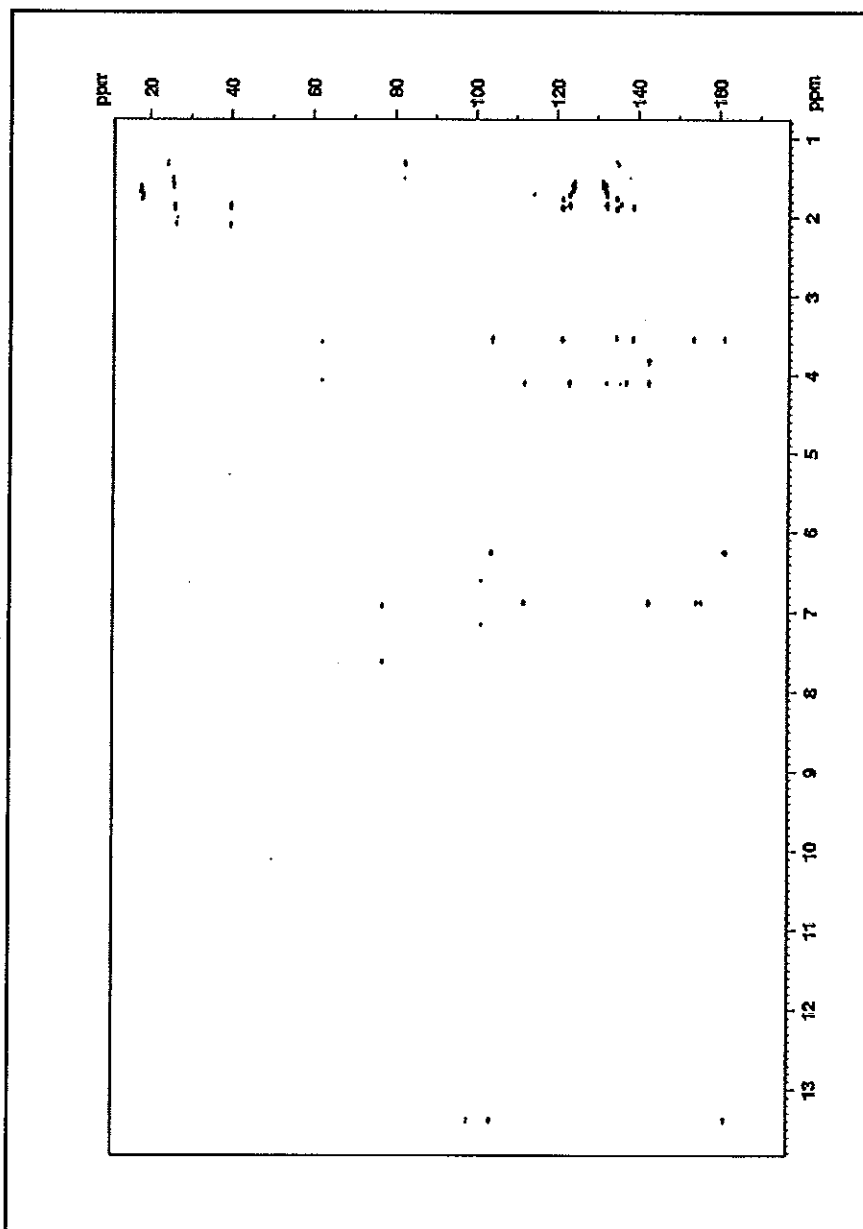


Figure 141 2D HMBC spectrum of compound GP11

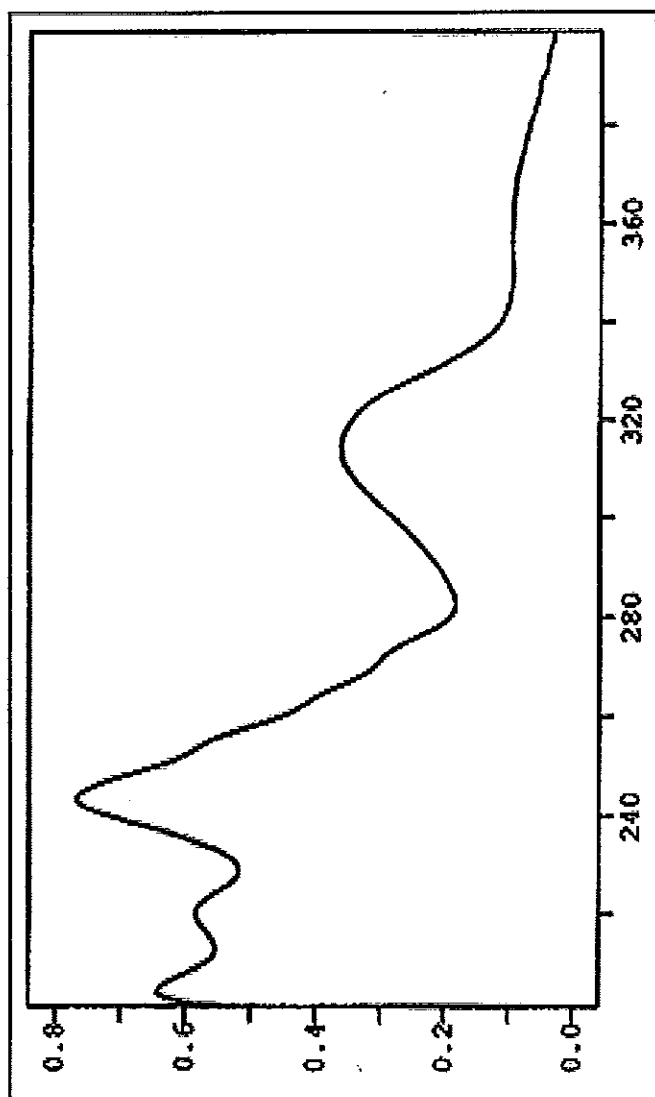


Figure 142 UV (MeOH) spectrum of compound GP15

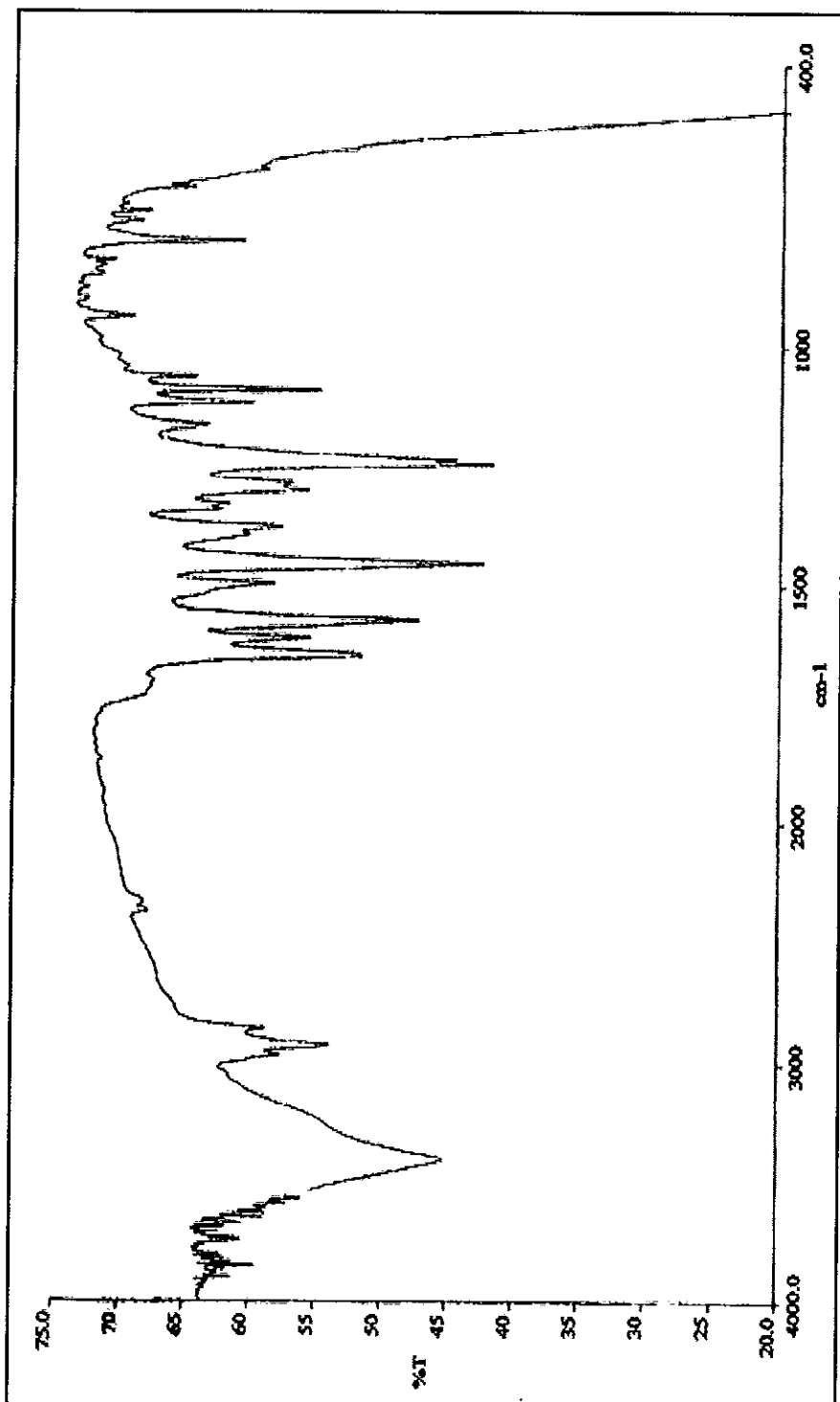


Figure 143 FT-IR (neat) spectrum of compound GP15

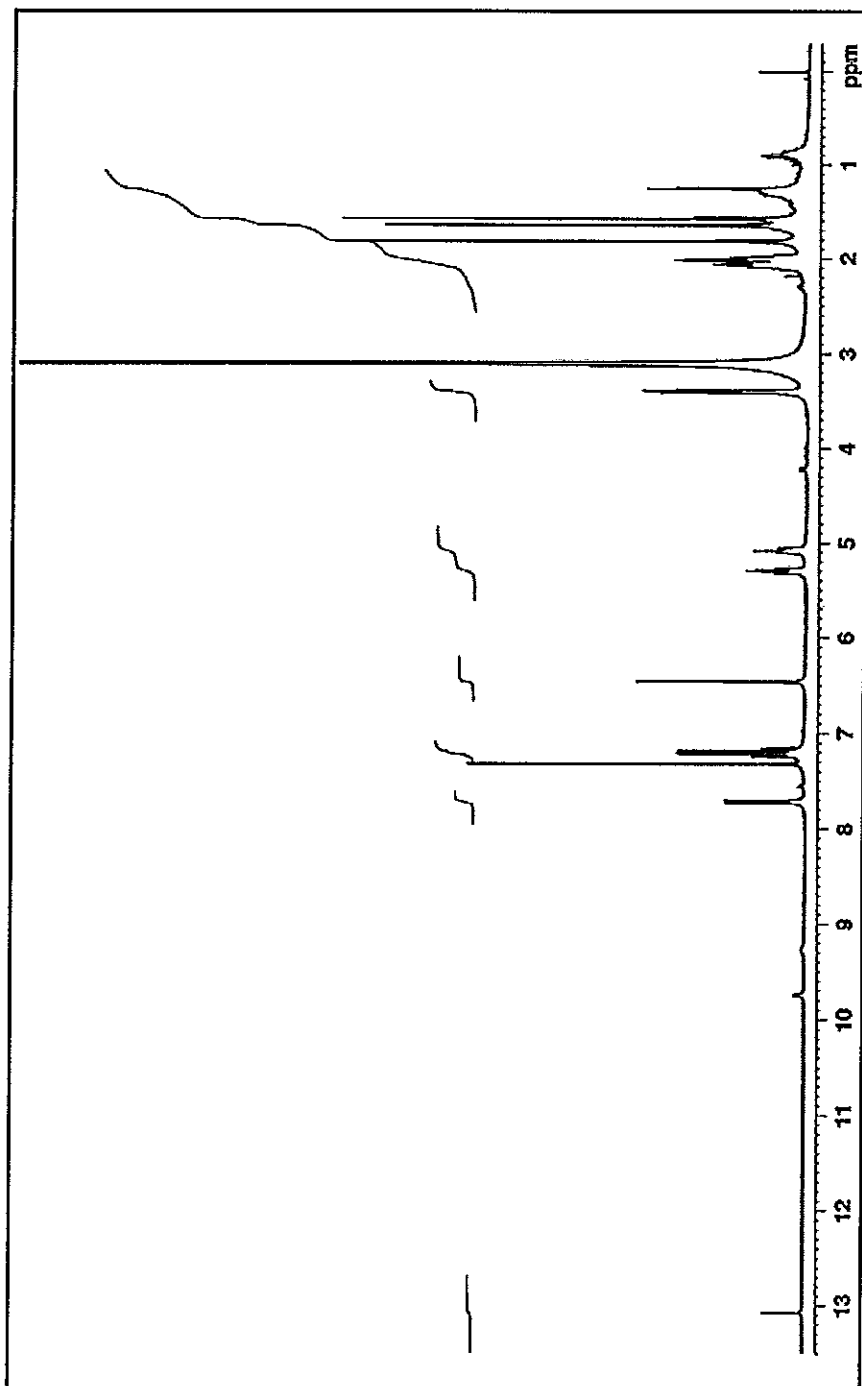


Figure 144  $^1\text{H}$  NMR (300 MHz) ( $\text{CD}_3\text{Cl}+\text{CD}_3\text{OD}$ ) spectrum of compound GP15

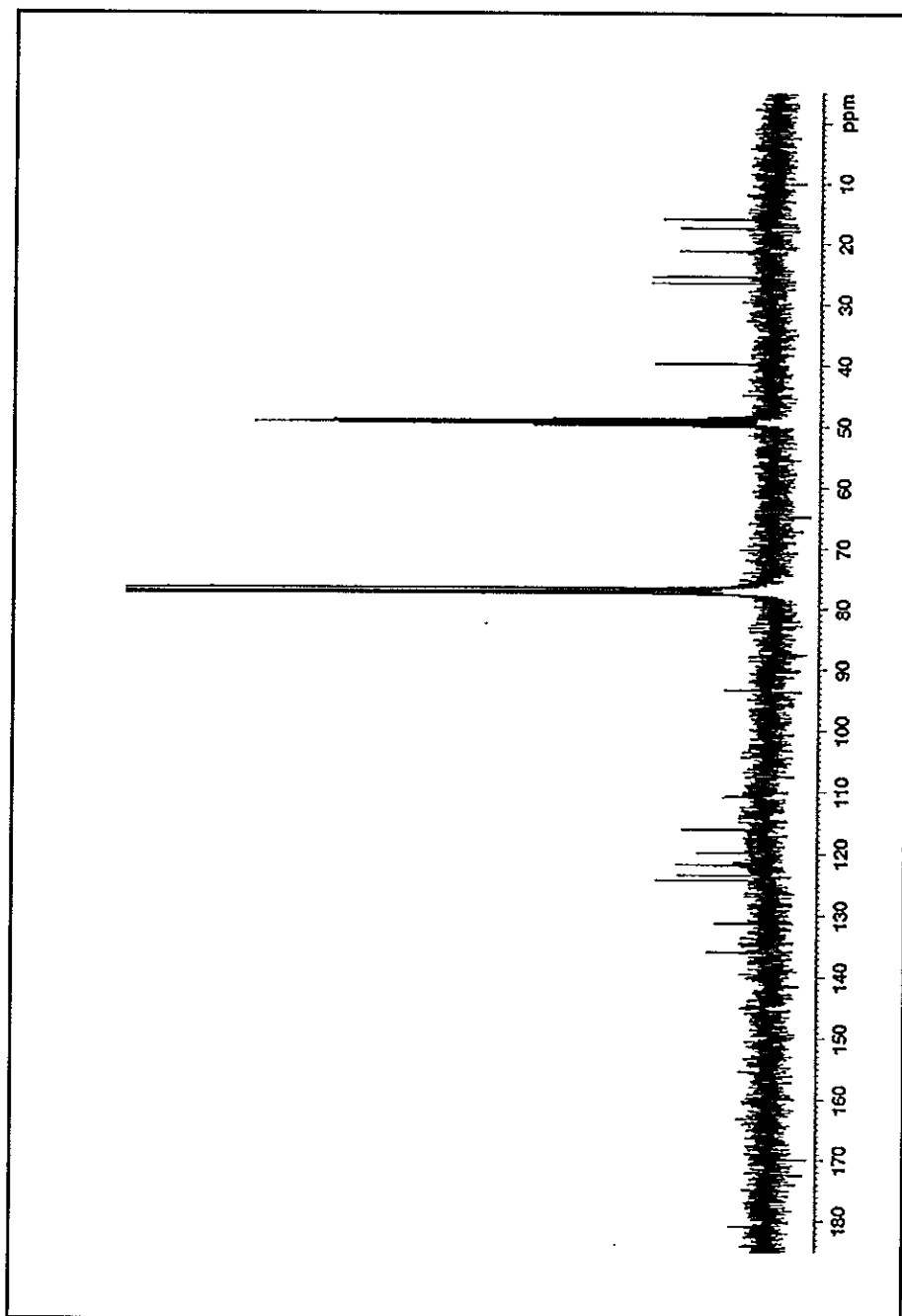


Figure 145  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CD}_3\text{Cl}+\text{CD}_3\text{OD}$ ) spectrum of compound GP15

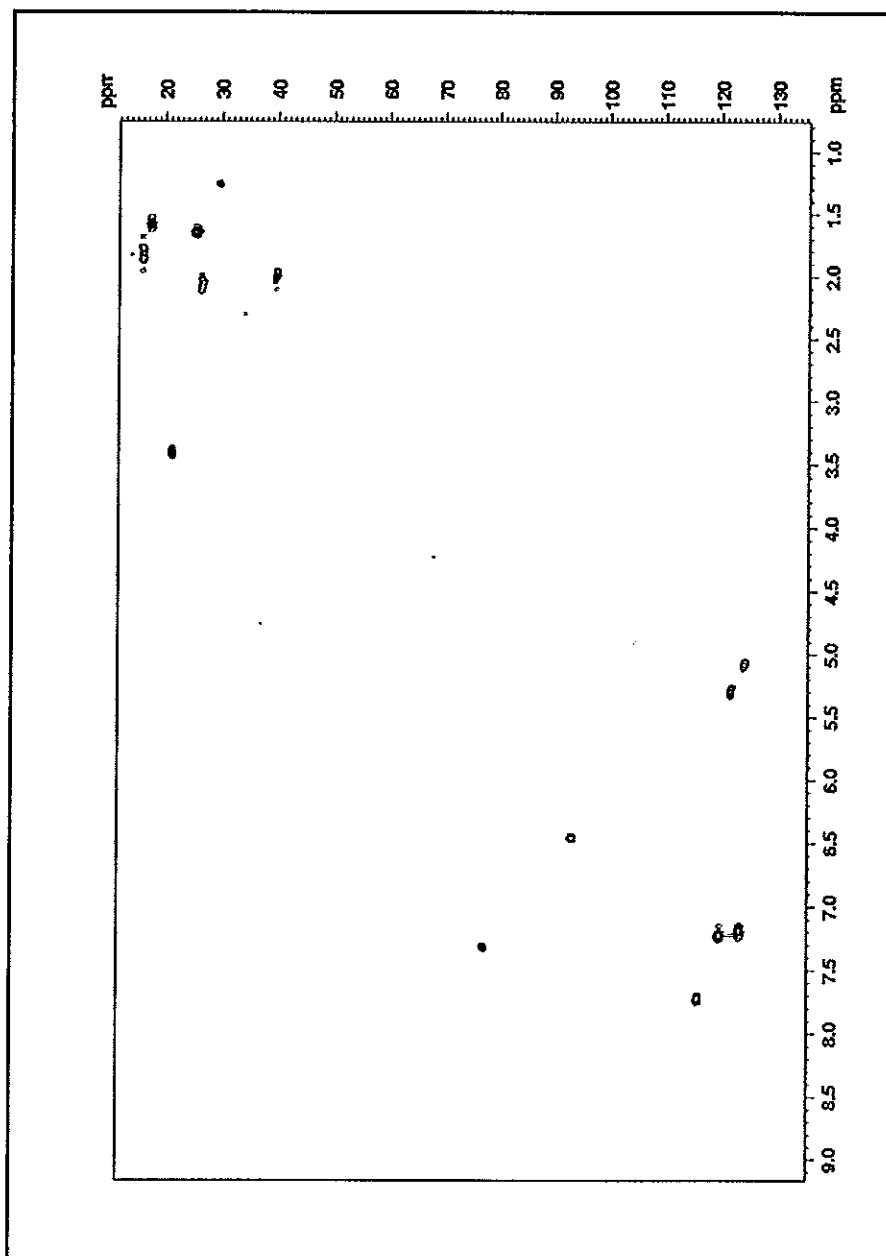


Figure 146 2D HMQC spectrum of compound GP15

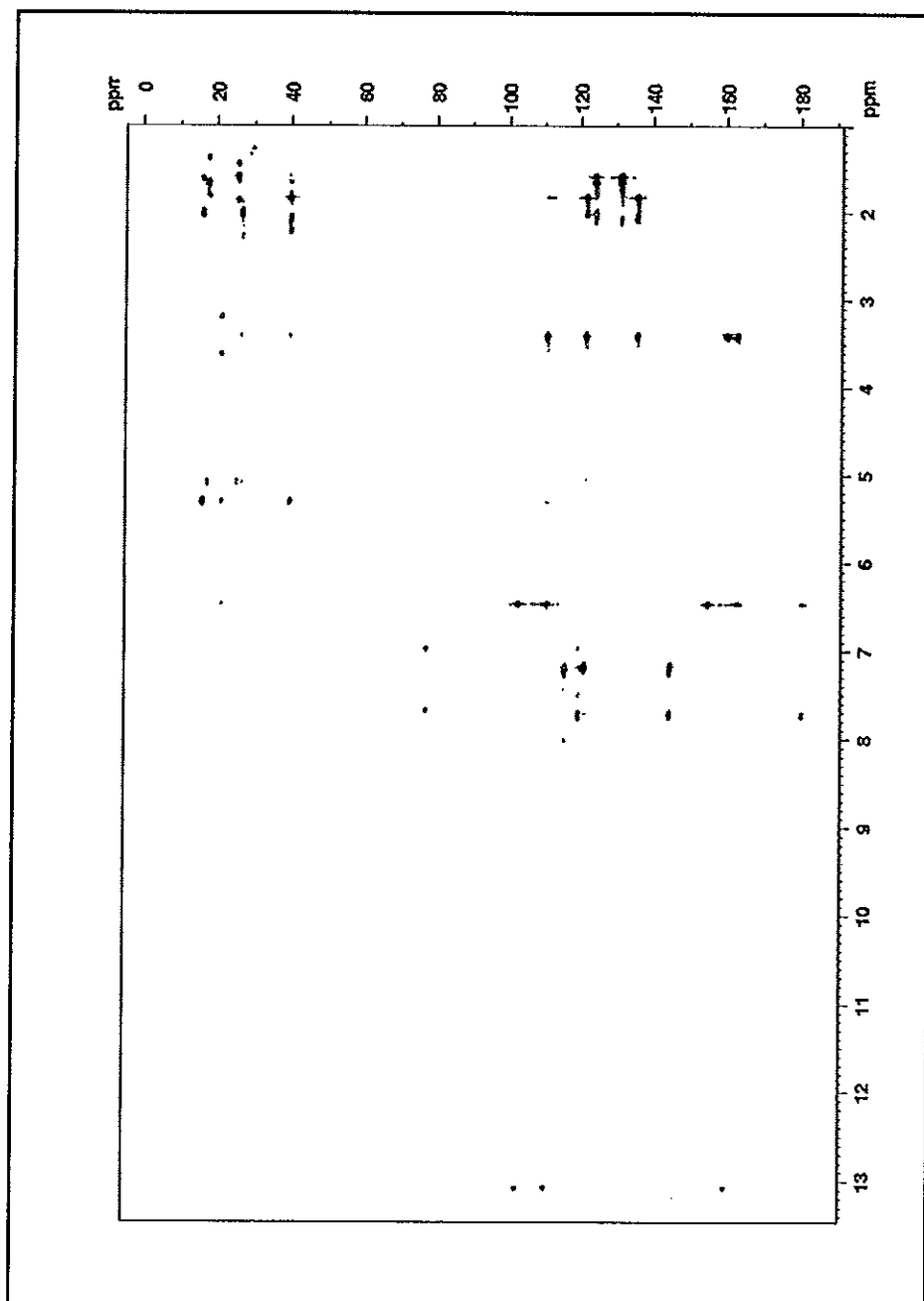


Figure 147 2D HMBC spectrum of compound GP15



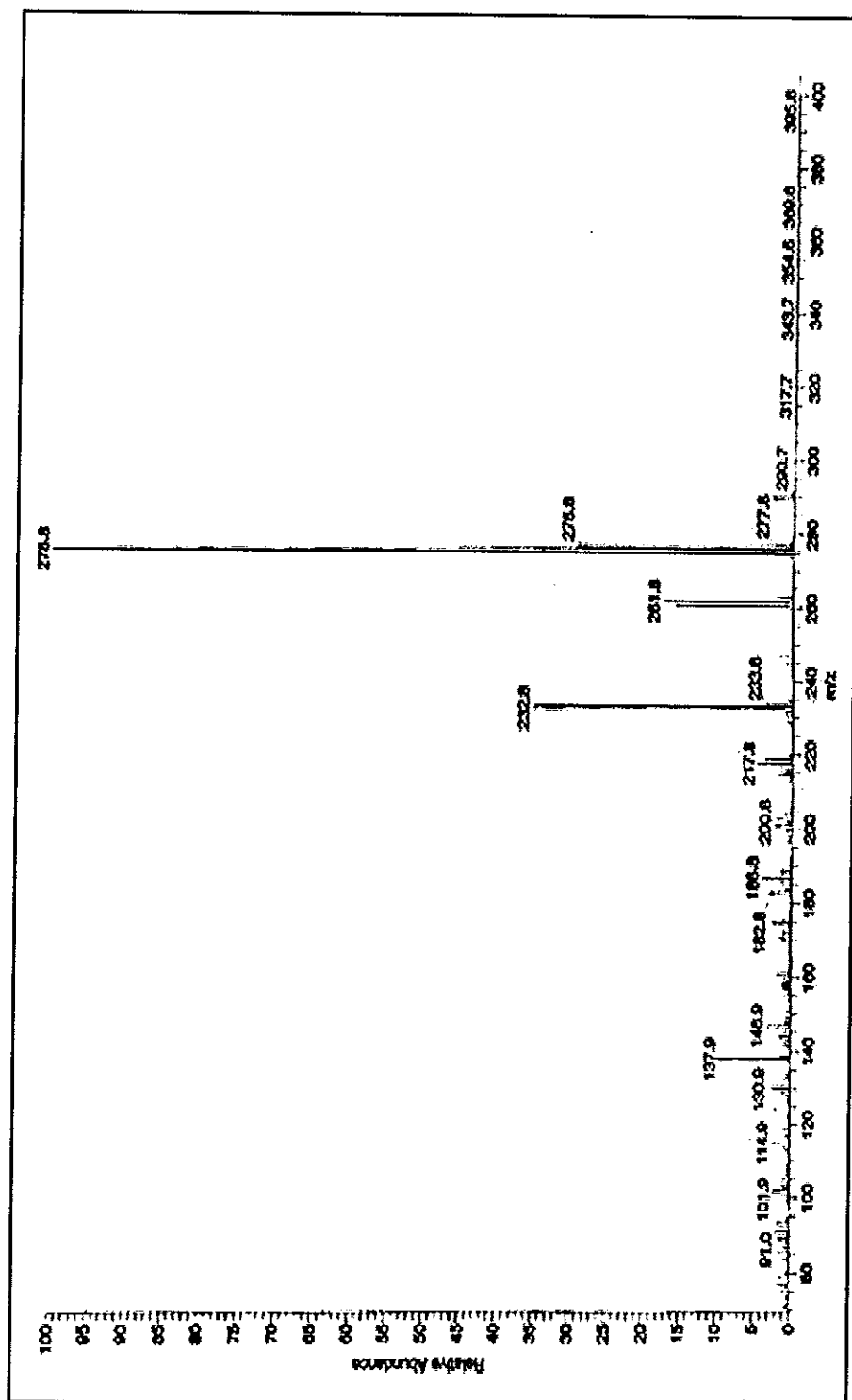


Figure 148 Mass spectrum of compound GPI0

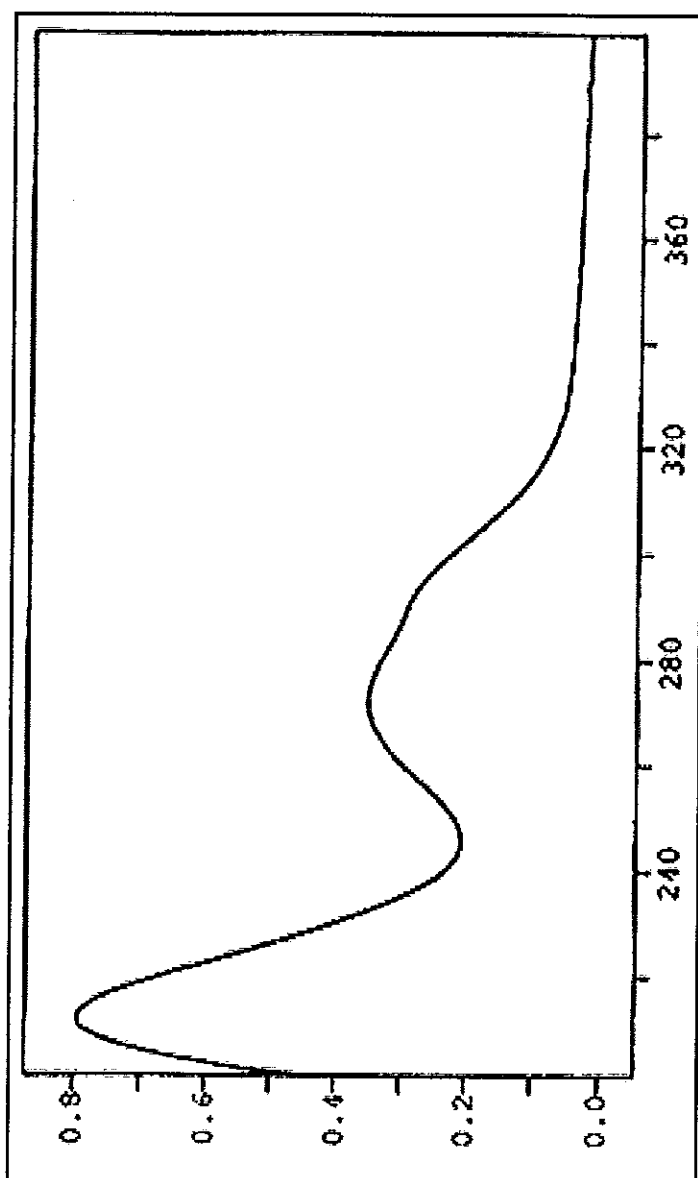


Figure 149 UV (MeOH) spectrum of compound GP10

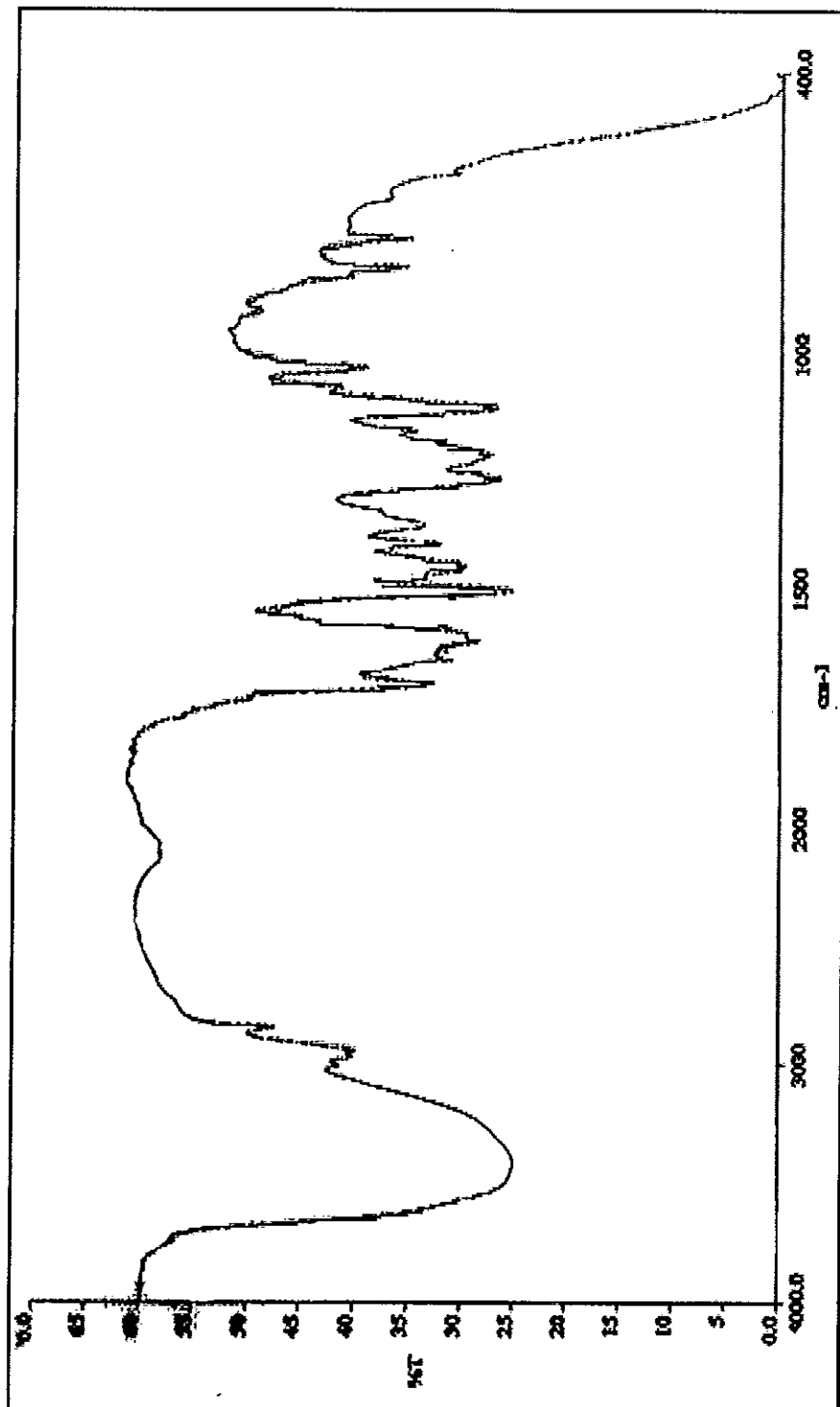


Figure 150 FT-IR (neat) spectrum of compound GP10

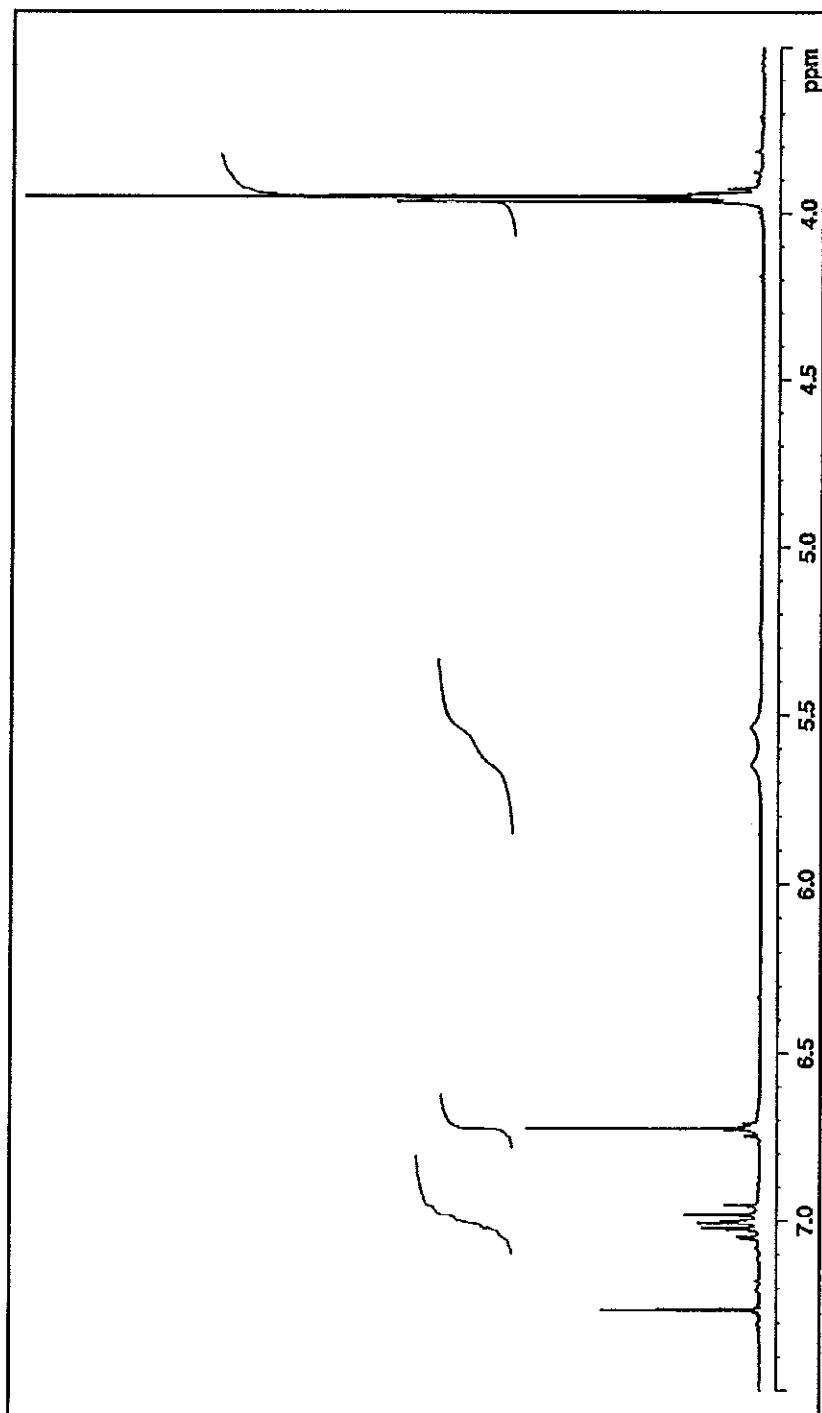


Figure 1S1  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound GP10

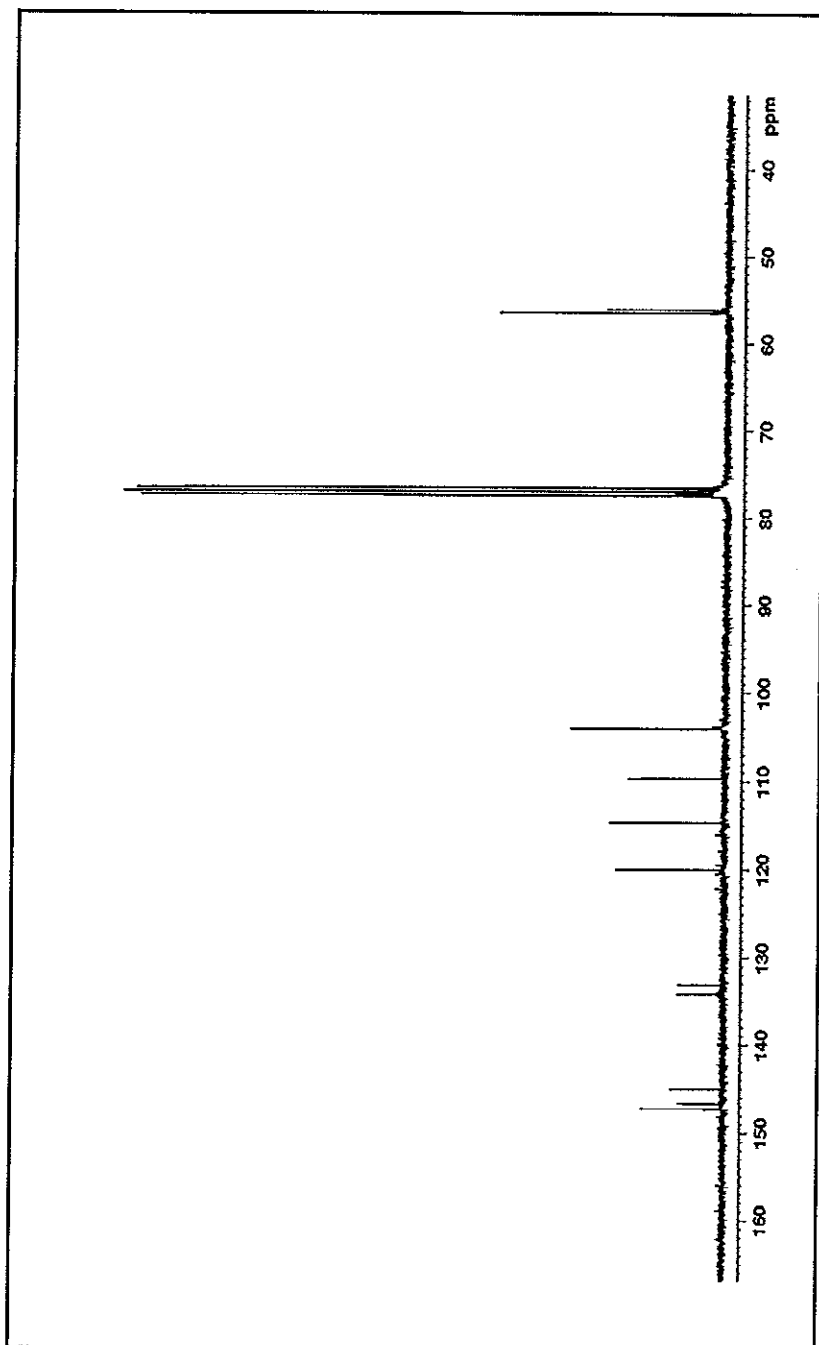


Figure 152  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound GP10

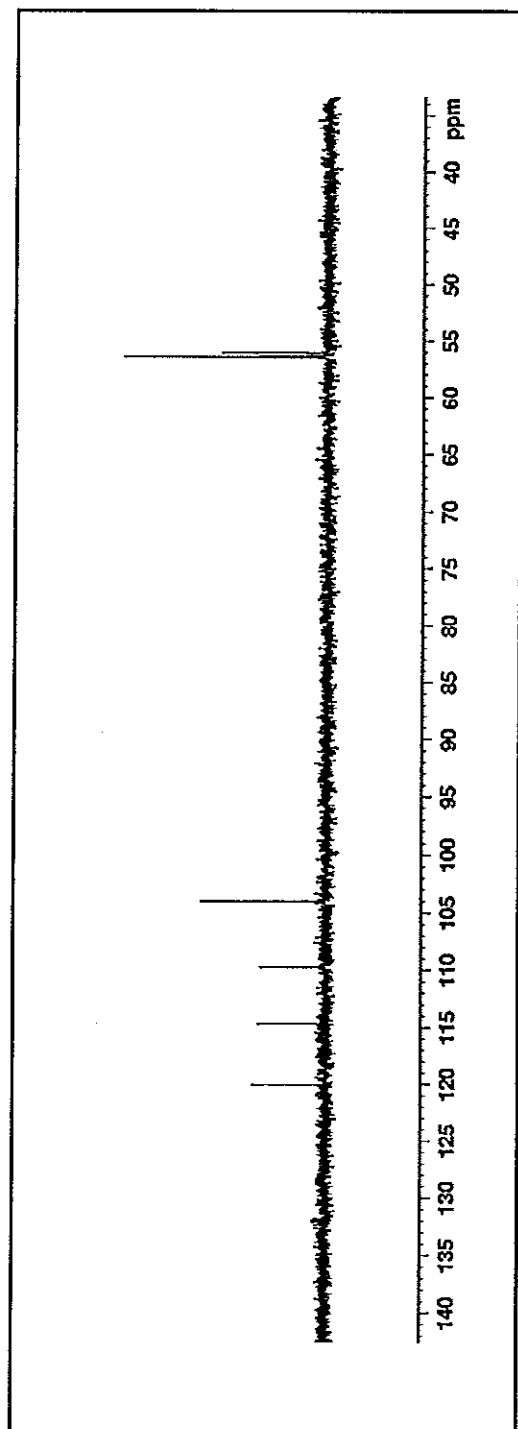


Figure 153 DEPT 90° spectra of compound GP10

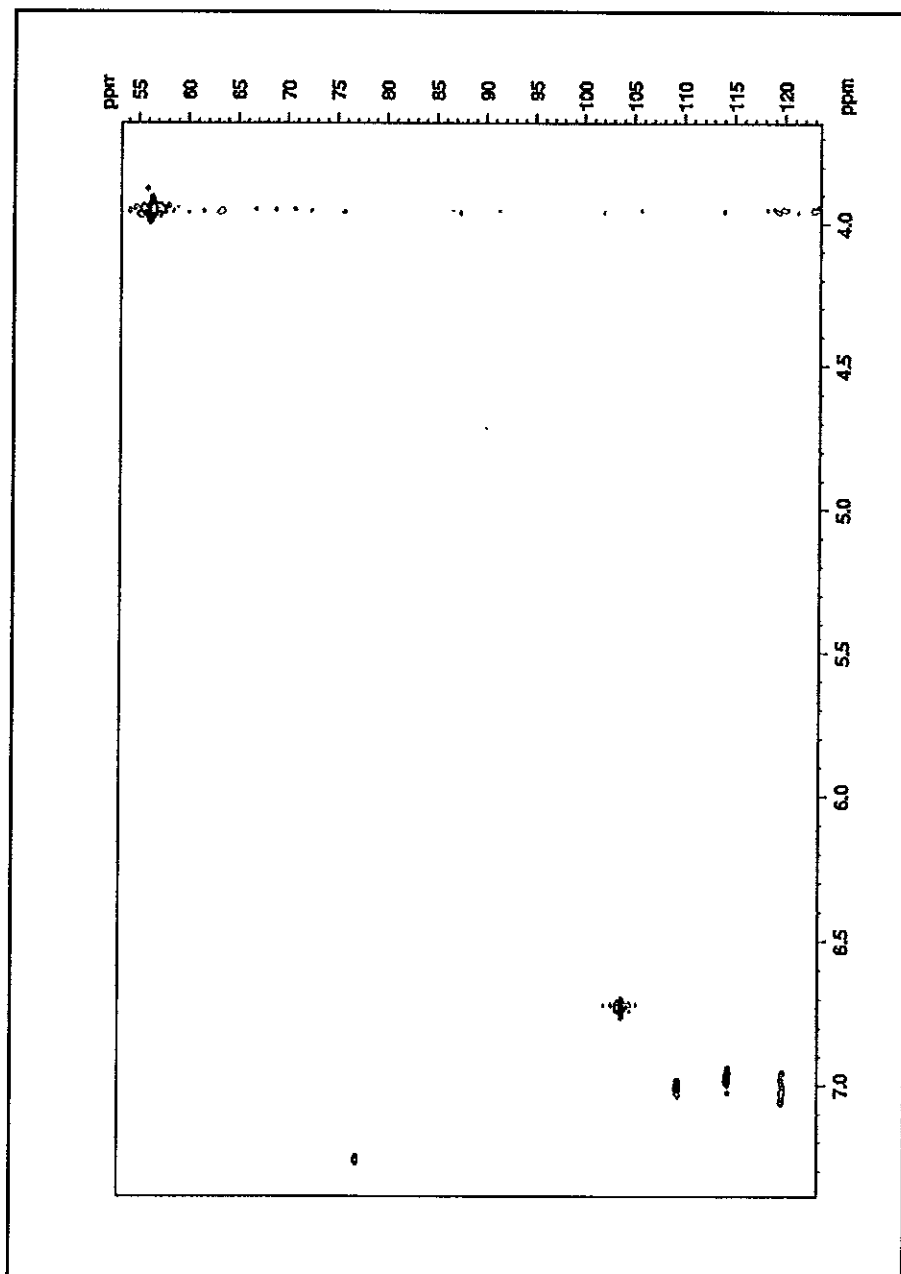


Figure 154 2D HMQC spectrum of compound GP10

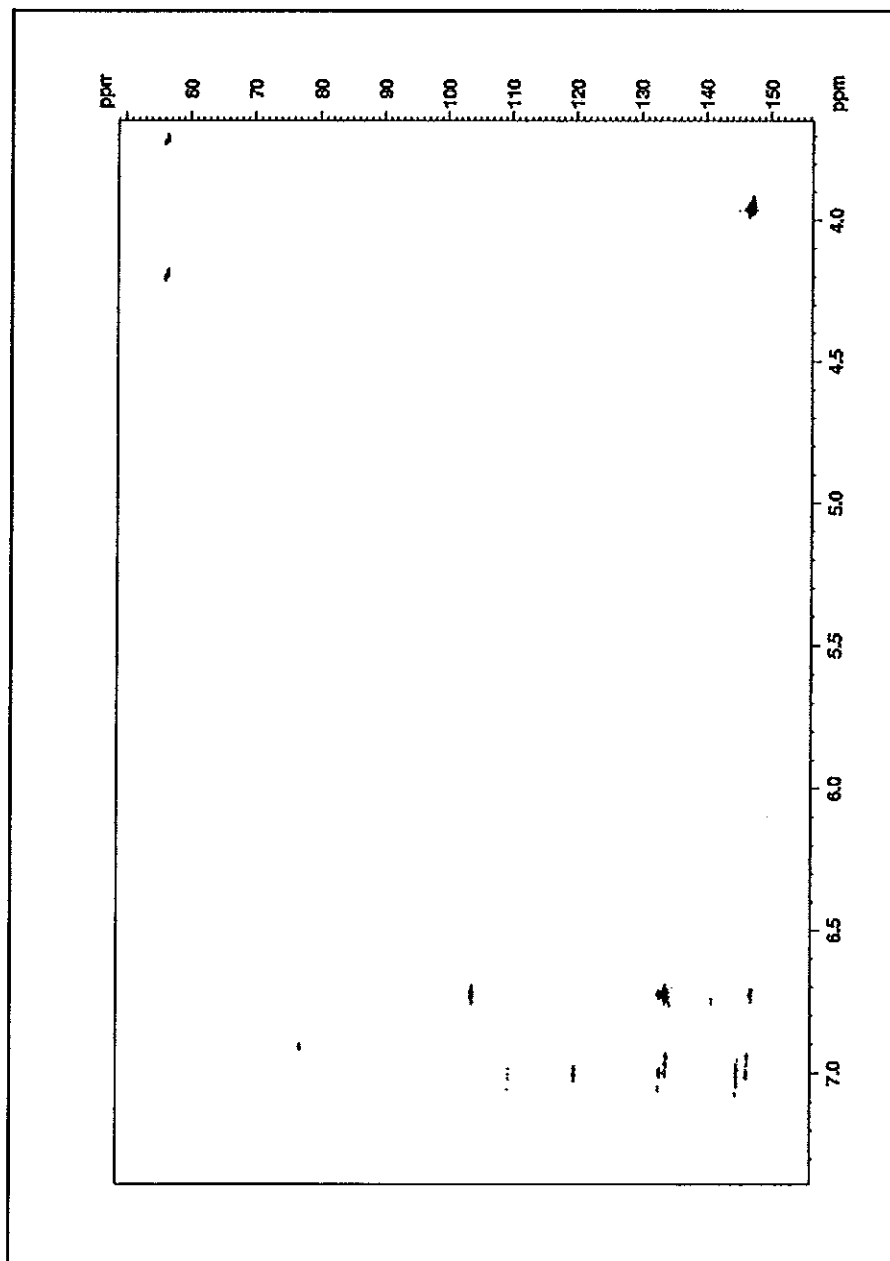


Figure 155 2D HMBC spectrum of compound GPI0



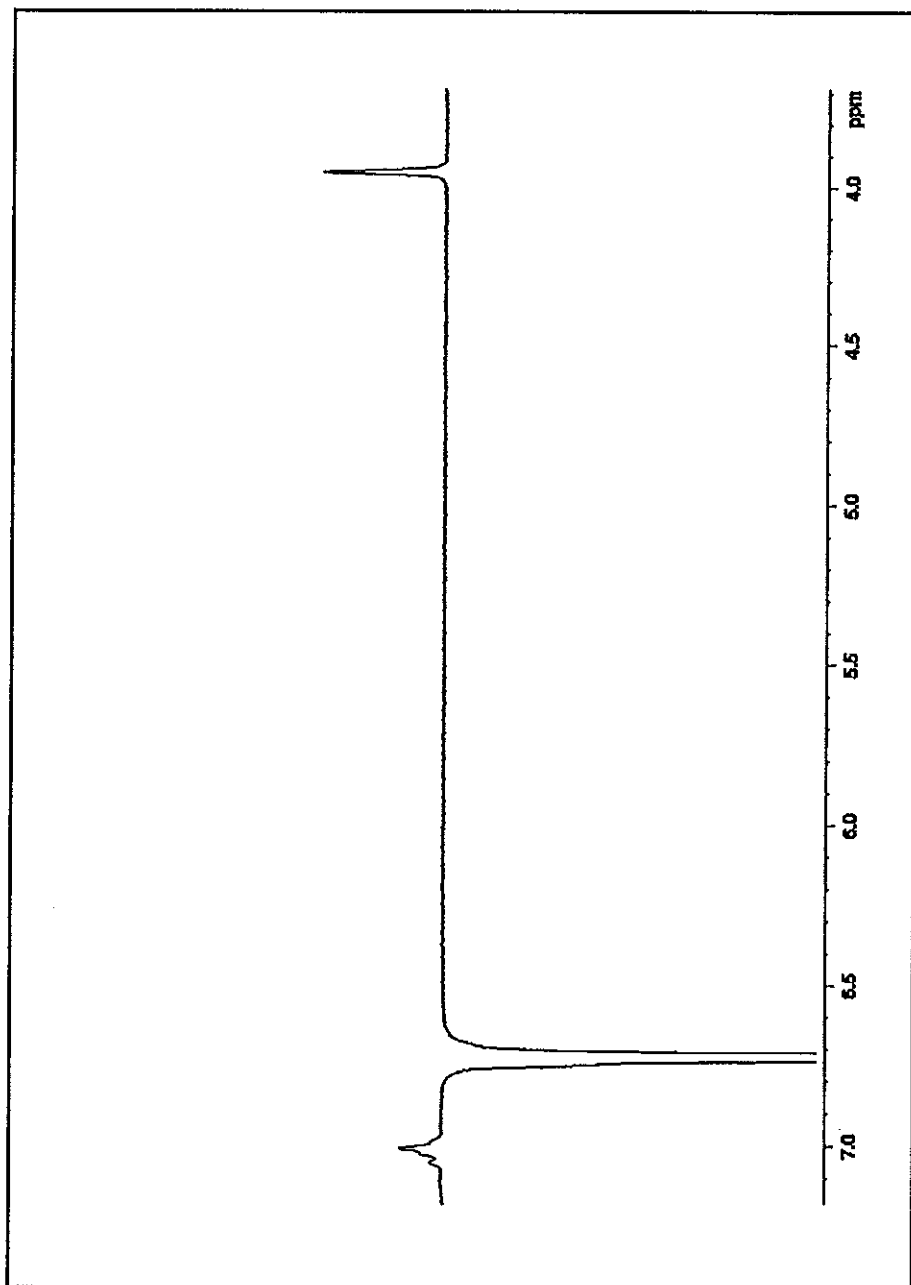


Figure 156 NOE difference spectrum of compound GP10 after irradiation at  $\delta_{\text{H}}$  6.72 (H-1 and H-6)

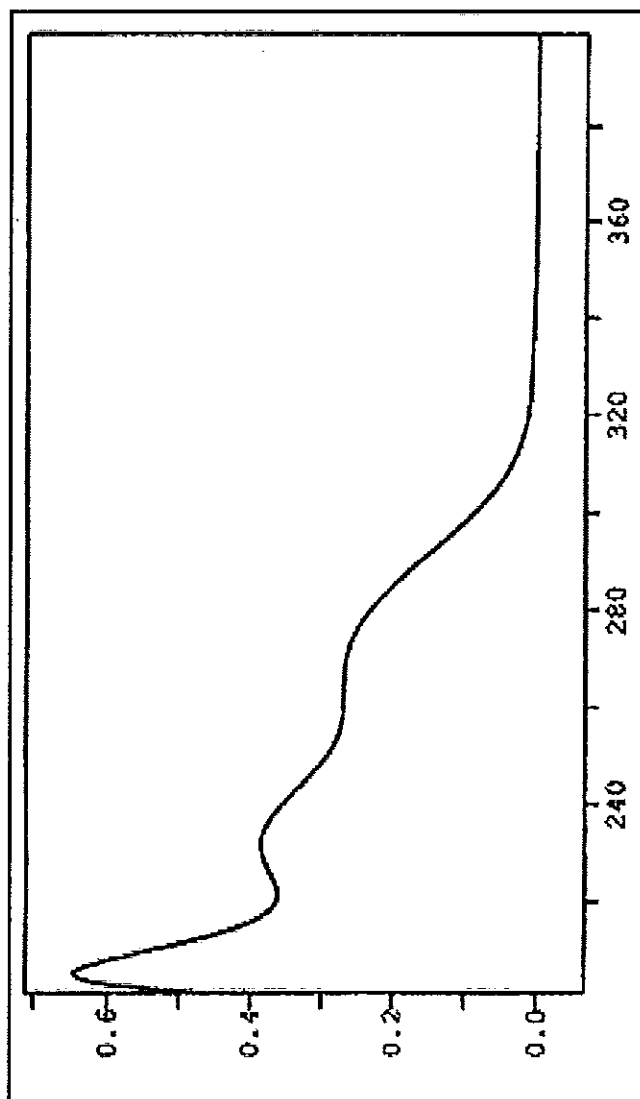


Figure 157 UV (MeOH) spectrum of compound GP13

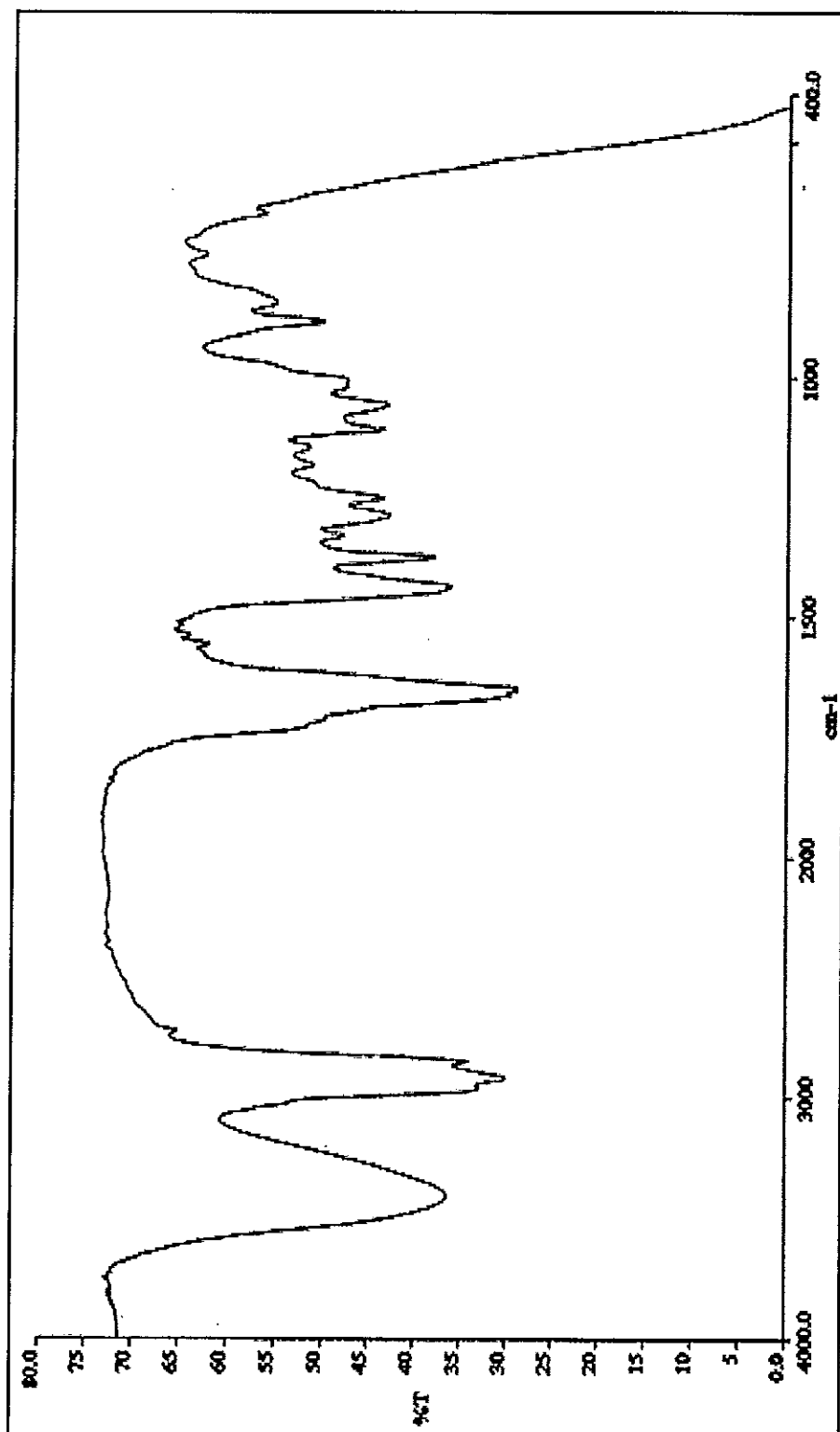


Figure 158 FT-IR (neat) spectrum of compound GP13

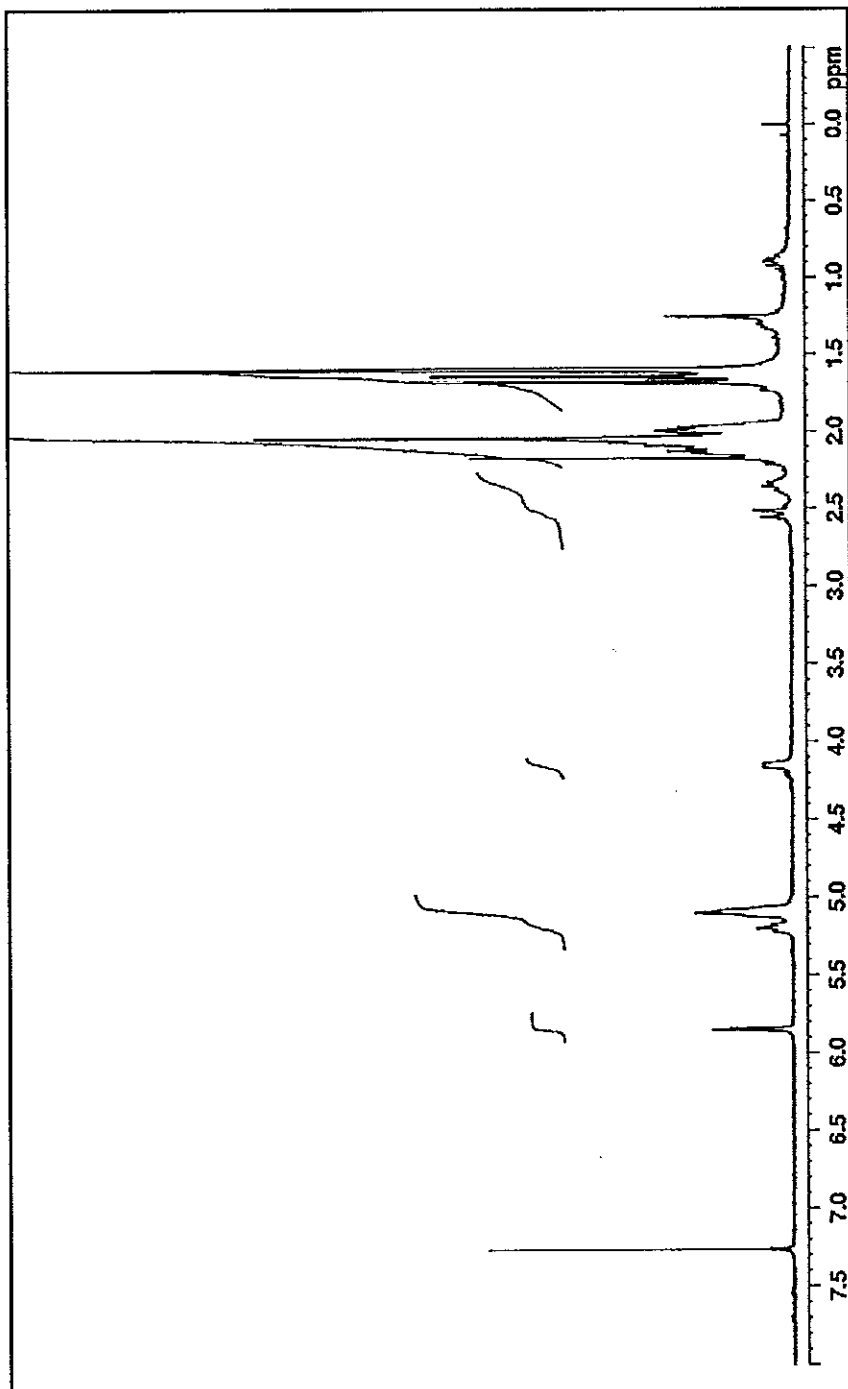


Figure 159  $^1\text{H}$  NMR (300 MHz) ( $\text{CDCl}_3$ ) spectrum of compound GP13

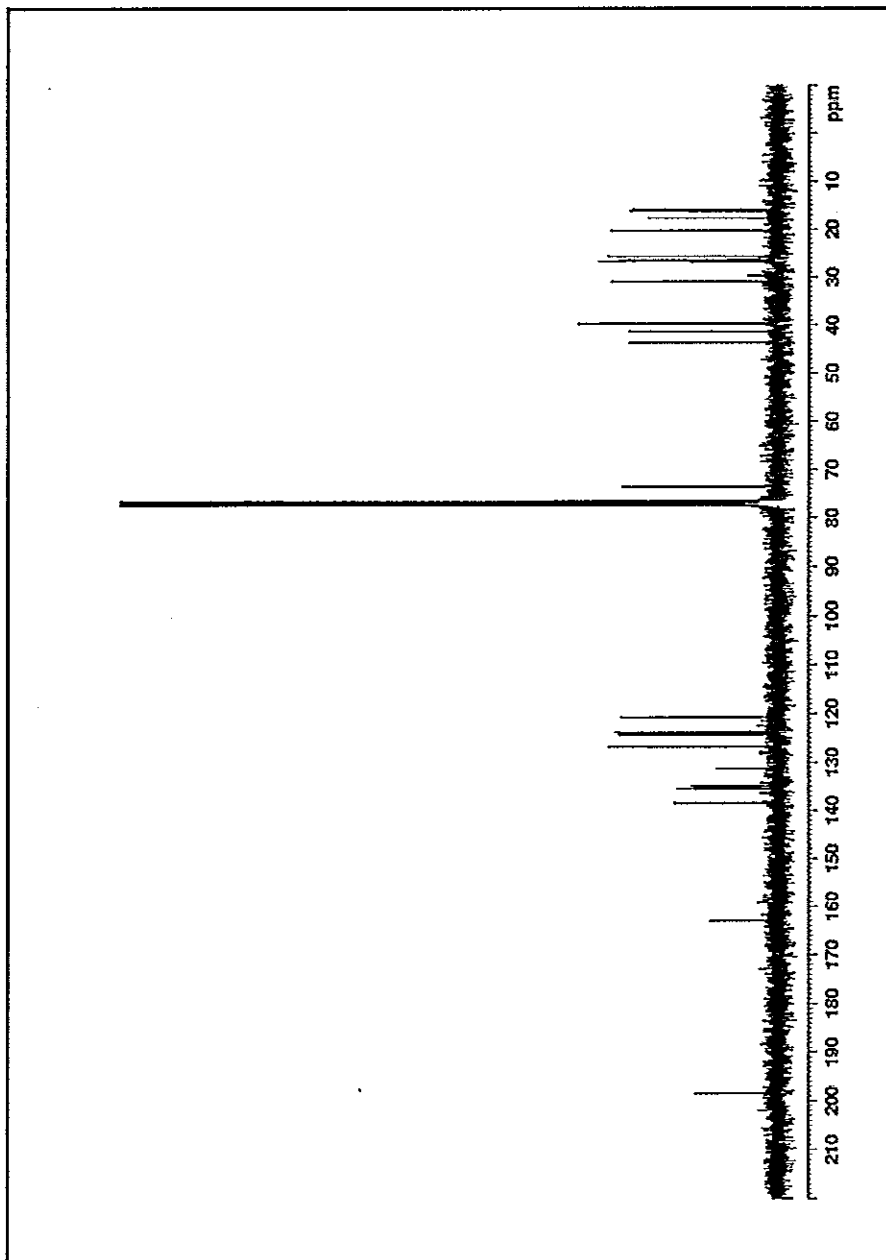


Figure 160  $^{13}\text{C}$  NMR (75 MHz) ( $\text{CDCl}_3$ ) spectrum of compound GPI3

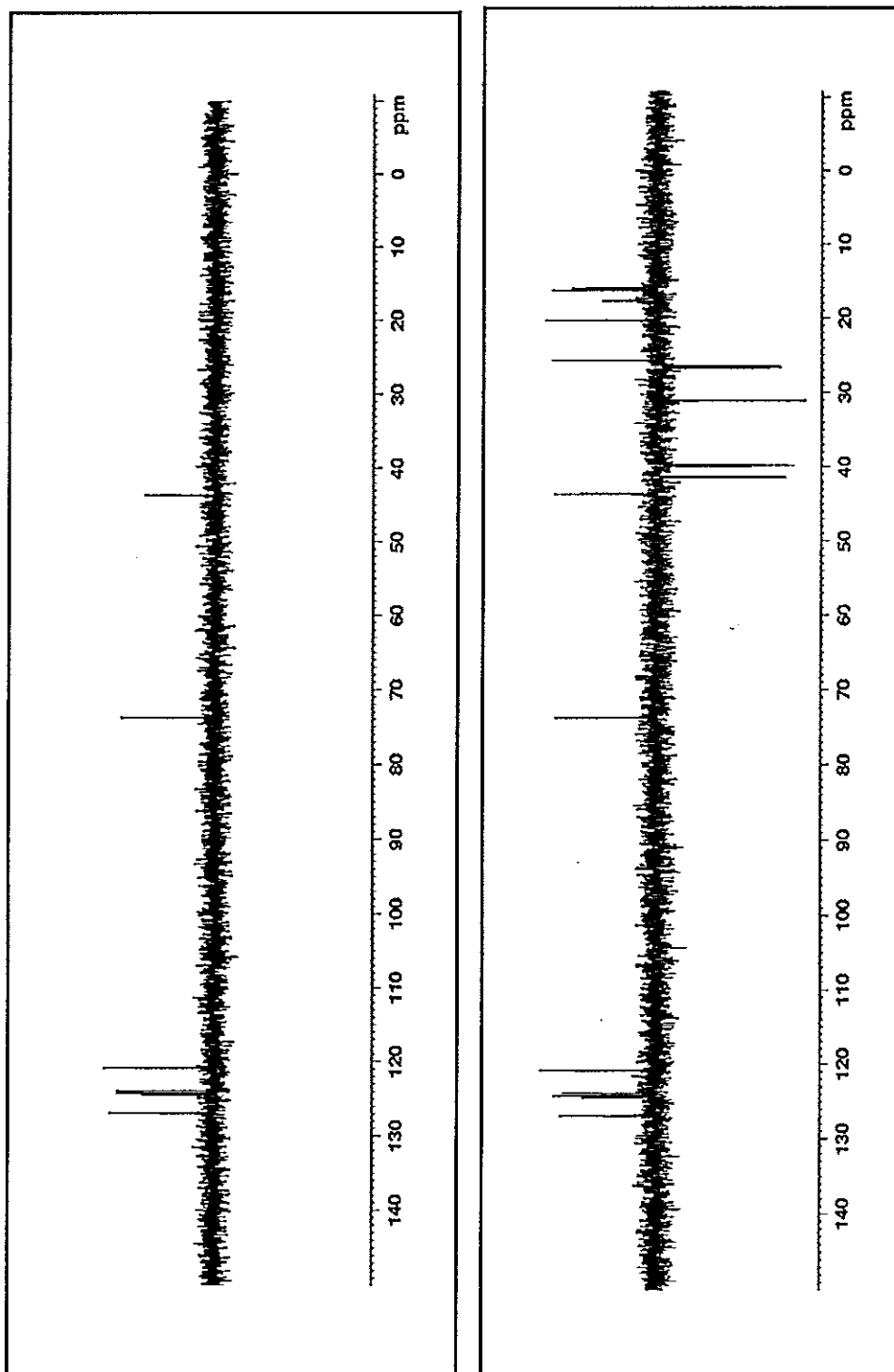


Figure 161 DEPT  $90^\circ$  and  $135^\circ$  spectra of compound GP13

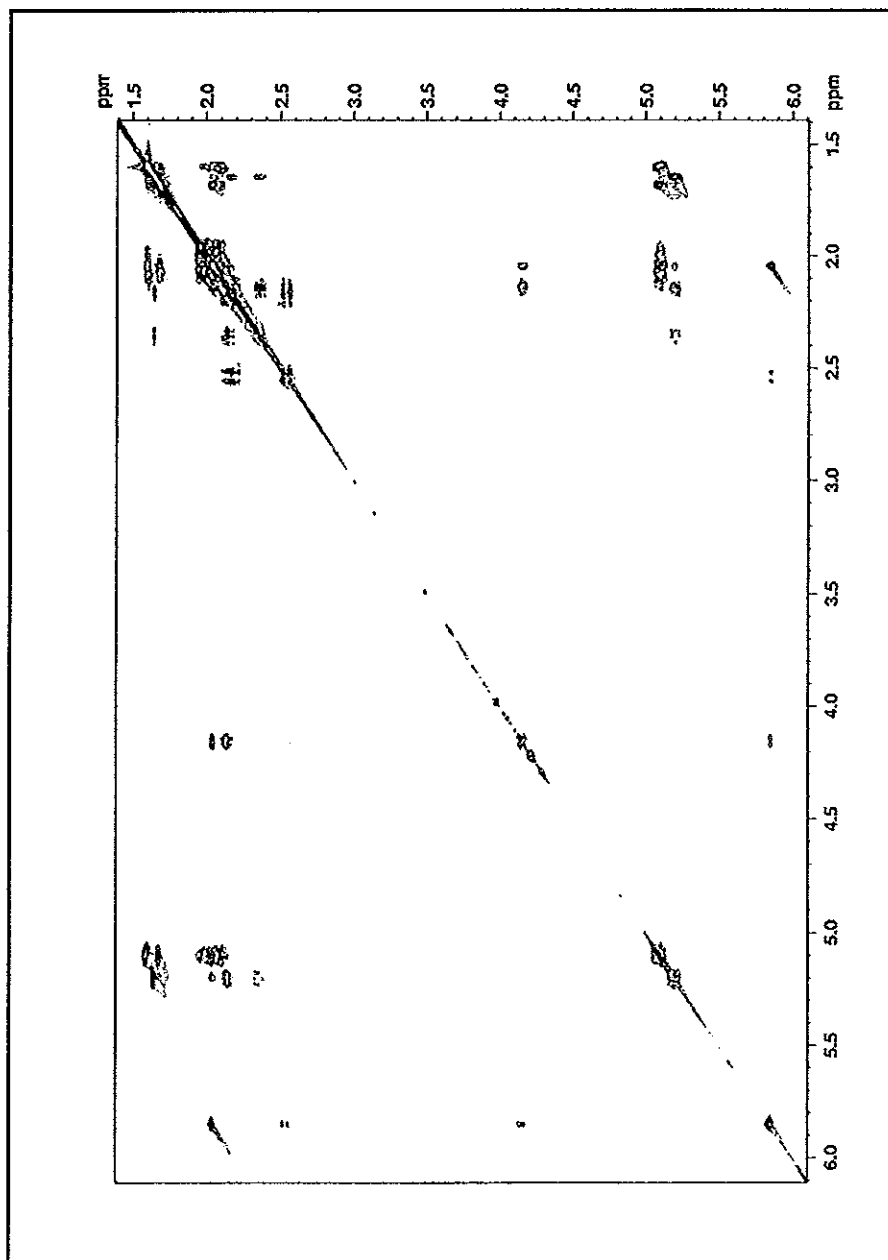


Figure 162  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of compound GP13

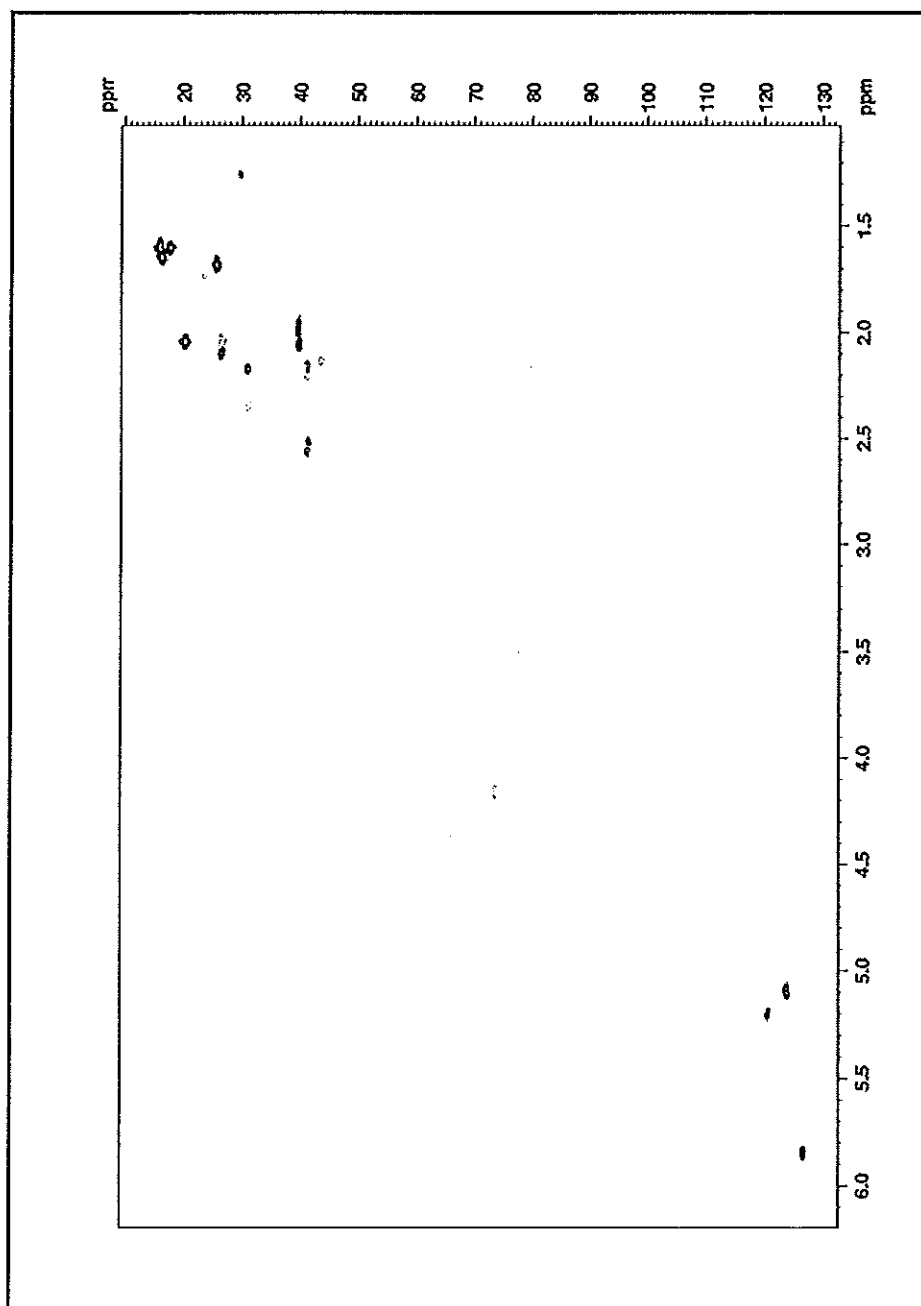


Figure 163 2D HMQC spectrum of compound GP13



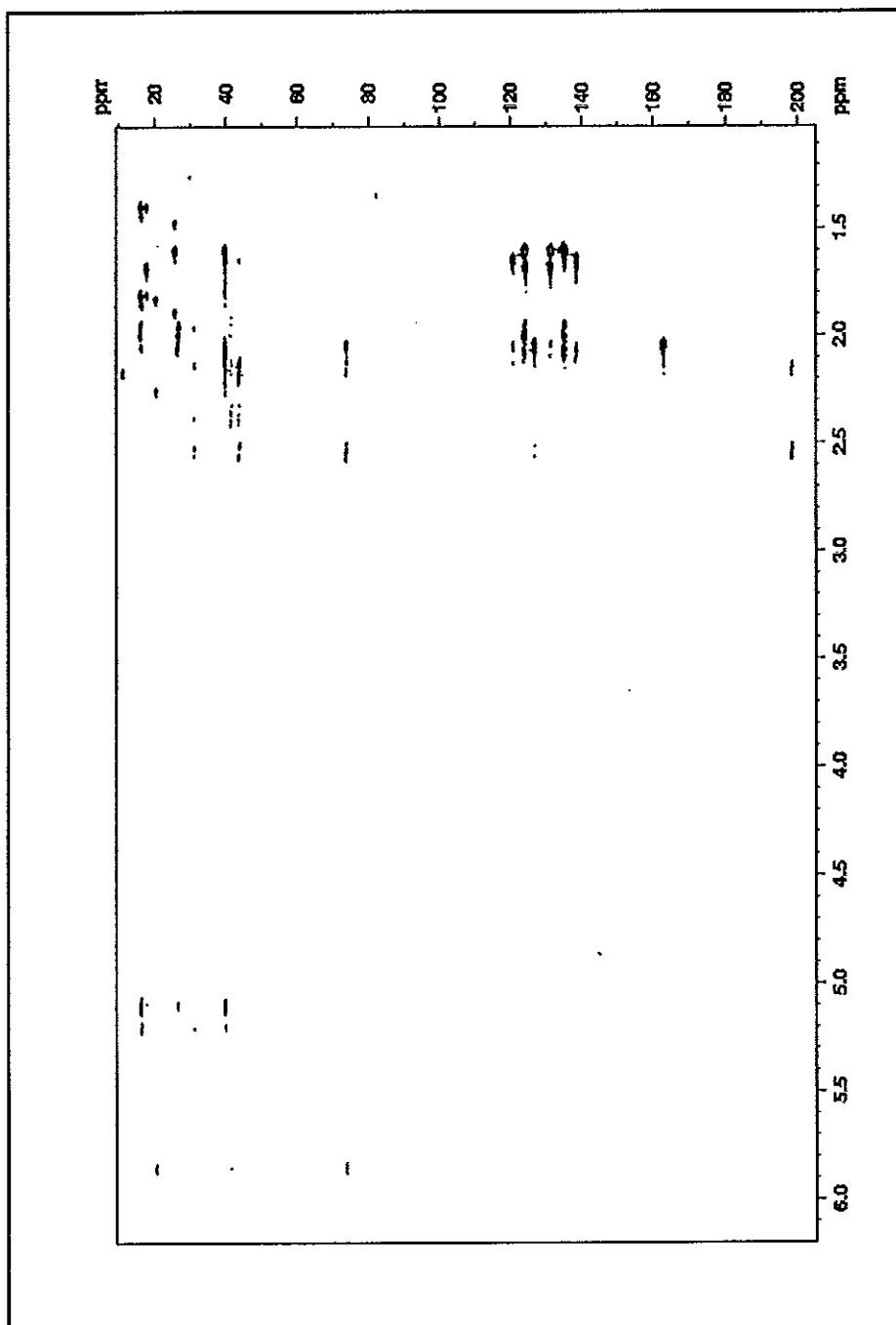


Figure 164 2D HMBC spectrum of compound GP13

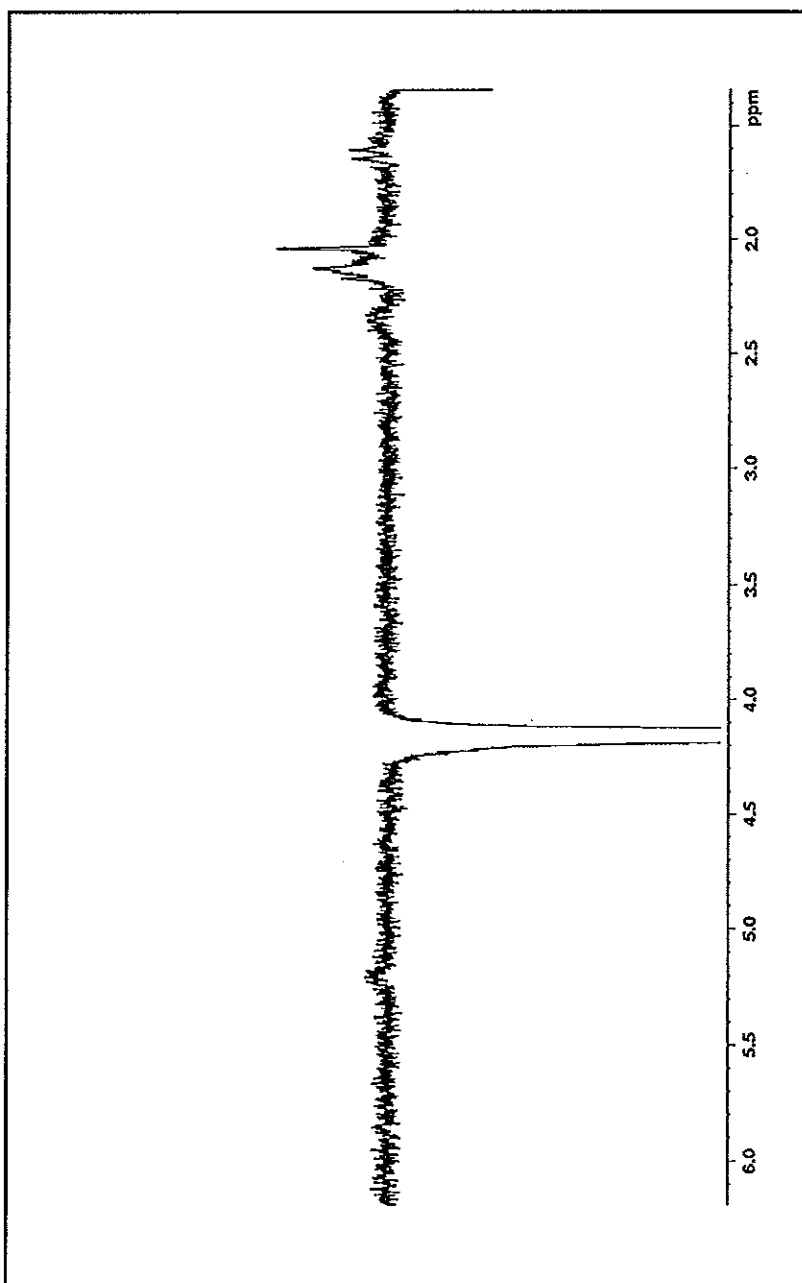


Figure 165 NOE difference spectrum of compound GPI3 after irradiation at  $\delta_{\text{H}} 4.15$  (H-4)

## REFERENCE

- Abe, F., Nagafuji, S., Okabe, H., Higo, H. and Akahane, H. 2003. "Trypanocidal constituents in plants 2. Xanthenes from the stem bark of *Garcinia subelliptica*", *Bio. Pharm. Bull.* 26(12), 1730-1733.
- Abe, F., Nagafuji, S., Okabe, H., Akahane, H., Estrada-Muniz, E., Huerta-Reyes, M. and Reyes-Chilpa, R. 2004. "Trypanocidal constituents in plants 3. Leaves of *Garcinia intermedia* and heartwood of *Calophyllum brasiliense*", *Bio. Pharm. Bull.* 27(1), 141-143.
- Ali, S., Goundar, R., Sotheeswaran, S., Beaulieu, C. and Spino, C. 2000. "Benzophenones of *Garcinia pseudoguttifera* (Clusiaceae)", *Phytochemistry.* 53, 281-284.
- Baggett, S., Protiva, P., Mazzola, E.P., Yang, H., Ressler, E.T., Basile, M.J., Weinstein, I.B. and Kennelly, E.J. 2005. "Bioactive benzophenones from *Garcinia xanthochymus* fruits", *J. Nat. Prod.* 68(3), 354-360.
- Chanmahasathien, W., Li, Y., Satake, M., Oshima, Y., Ishibashi, M., Ruangrunsi, N. and Ohizumi, Y. 2003a. "Prenylated xanthenes from *Garcinia xanthochymus*", *Chem. Pharm. Bull.* 51(11), 1332-1334.
- Chanmahasathien, W., Li, Y., Satake, M., Oshima, Y., Ruangrunsi, N. and Ohizumi, Y. 2003b. "Prenylated xanthenes with NGF-potentiating activity from *Garcinia xanthochymus*", *Phytochemistry.* 64(5), 981-986.
- Chen, J.J., Chen, I.S. and Duh, C.Y. 2005. "Cytotoxic xanthenes and biphenyls from the root of *Garcinia liniifera*", *Planta Med.* 71(2), 196-201.

- Chiang, Y.-M., Kuo, Y.-H., Oota, S. and Fukuyama, Y. 2003. "Xanthenes and benzophenones from the stems of *Garcinia multiflora*", *J. Nat. Prod.* 66(8), 1070-1073.
- Deachathai, S., Mahabusarakam, W., Phongpaichit, S. and Taylor, W.C. 2005. "Phenolic compounds from the fruit of *Garcinia dulcis*", *Phytochemistry.* 66(19), 2368-2375.
- Don, M.-J., Huang, Y.-J., Huang, R.-L. and Lin, Y.-L. 2004. "New phenolic principles from *Hypericum sampsonii*", *Chem. Pharm. Bull.* 52(7), 866-869.
- Farombi, E.O., Akanni, O.O. and Emerole, G.O. 2002. "Antioxidant and scavenging activities of flavonoid extract (Kolaviron) of *Garcinia kola* seeds", *Pharm. Bio.* 40(2), 107-116.
- Han, Q.-B., Lee, S.-F., Qiao, C.-F., He, Z.-D., Song, J.-Z., Sun, H.-D. and Xu, H.-X. 2005. "Complete NMR assignments of the antibacterial biflavonoid GB1 from *Garcinia kola*", *Chem. Pharm. Bull.* 53(8), 1034-1036.
- Hay, A.-E., Helesbeux, J.-J., Duval, O., Labaied, M., Grellier, P. and Richomme, P. 2004a. "Antimalarial xanthenes from *Calophyllum caledonicum* and *Garcinia vieillardii*", *Life Sciences.* 75(25), 3077-3085.
- Hay, A.-E., Aumond, M.-C., Mallet, S., Dumontet, V., Litaudon, M., Rondeau, D. and Richomme, P. 2004b. "Antioxidant xanthenes from *Garcinia vieillardii*", *J. Nat. Prod.* 67(4), 707-709.
- Herath, K., Jayasuriya, H., Ondeyka, J.G., Guan, Z., Borris, R.P., Stijfhoorn, E., Stevenson, D., Wang, J., Sharma, N., MacNaul, K., Menke, J.G., Ali, A., Schulman, M.J. and Singh, S.B. 2005. "Guttiferone I, a new prenylated benzophenone from *Garcinia humilis* as a Liver X Receptor Ligand", *J. Nat. Prod.* 68(4), 617-619.

- Huang, Y.-L., Chen, C.-C., Chen, Y.-J., Huang, R.-L. and Shieh, B.-J. 2001. "Three xanthenes and a benzophenone from *Garcinia mangostana*", *J. Nat. Prod.* 64(7), 903-906.
- Ilyas, M., Parveen, M., Shafiullah. and Ahmad, S.M. 2002. "A novel chalcone from *Garcinia nervosa*", *J. Chem. Res, Synop.* 5, 231-233.
- Ito, C., Itoigawa, M., Miyamoto, Y., Onoda, S., Rao, K.S., Mukainaka, T., Tokuda, H., Nishino, H. and Furukawa, H. 2003a. "Polyprenylated benzophenones from *Garcinia assigu* and their potential cancer chemopreventive activities", *J. Nat. Prod.* 66(2), 206-209.
- Ito, C., Itoigawa, M., Takakura, T., Ruangrunsi, N., Enjo, F., Tokuda, H., Nishino, H. and Furukawa, H. 2003b. "Chemical constituents of *Garcinia fusca* : Structure elucidation of eight new xanthenes and their cancer chemopreventive activity", *J. Nat. Prod.* 66(2), 200-205.
- Jantan, I., Pizar, M.M., Idris, M.S., Taher, M. and Ali, R.M. 2002. "In vitro inhibitory effect of rubraxanthone isolated from *Garcinia parvifolia* on platelet-activating factor receptor binding", *Planta Med.* 68(12), 1133-1134.
- Komguem, J., Meli, A.L., Manfouo, R.N., Lontsi, D., Ngounou, F.N., Kuete, V., Kamdem, H.W., Tane, P., Ngadjui, B.T., Sondengam, B.L. and Connolly, J.D. 2005. "Xanthenes from *Garcinia smeathmannii* (Oliver) and their antimicrobial activity", *Phytochemistry.* 66(14), 1713-1717.
- Kosela, S., Hu, L.-H., Yip, S.-C., Rachmatia, T., Sukri, T., Daulay, T.S., Tan, G.-K., Vittal, J.J. and Sim, K.-Y. 1999. "Dulxanthone E : a pyranoxanthone from the leaves of *Garcinia dulcis*", *Phytochemistry.* 52, 1375-1377.

- Kosela, S., Hu, L.-H., Rachmatia, T., Hanafi, M. and Sim, K.-Y. 2000. "Dulxanthones F-H, three new pyranoxanthones from *Garcinia dulcis*", *J. Nat. Prod.* 63, 406-407.
- Lakshmi, C., Kumar, K.A. and Dennis, T.J. 2002. "Polyprenylated benzophenones from *Garcinia indica*", *J. Indian. Chem. Soc.* 79(12), 968-969.
- Lannang, A.M., Komguem, J., Ngninzeko F.N., Tangmouo, J.G., Lontsi, D., Ajaz, A., Choudhary, M.I., Ranjit, R., Devkota, K.P. and Sondengam, B.L. 2005. "Bangangxanthone A and B, two xanthones from the stem bark of *Garcinia polyantha* Oliv", *Phytochemistry.* 66(19), 2351-2355.
- Mahabusarakam, W., Chairerk, P. and Taylor, W.C. 2005. "Xanthones from *Garcinia cowa* Roxb. latex", *Phytochemistry.* 66(10), 1148-1153.
- Merza, J., Aumond, M.-C., Rondeau, D., Dumontet, V., Le Ray, A.-M., Seraphin, D. and Richomme, P. 2004. "Prenylated xanthones and tocotrienols from *Garcinia virgata*", *Phytochemistry.* 65(21), 2915-2920.
- Nguyen, L.H.D. and Harrison, L.J. 2000. "Xanthones and triterpenoids from the bark of *Garcinia vilersiana*", *Phytochemistry.* 53, 111-114.
- Nilar and Harrison, L.J. 2002. "Xanthones from the hartwood of *Garcinia mangostana*", *Phytochemistry.* 60(5), 541-548.
- Nilar., Nguyen, L.H.D., Venkatraman, G., Sim, K.Y. and Harrison, L.J. 2005. "Xanthones and benzophenones from *Garcinia griffithii* and *Garcinia mangostana*", *Phytochemistry.* 66(14), 1718-1723.
- Parveen, M., Ilyas, M., Mushfiq, M., Busudan, O.A. and Muhaisen, H.M.H. 2004. "A new biflavonoid from leaves of *Garcinia nervosa*", *Nat. Prod. Res.* 18(3), 269-275.

- Pattalung, P.N., Wiriyachitra, P., Ongsakul, M. 1988. "Antimicrobial activities of rubraxanthone isolated from *Garcinia parvifolia* (Miq.) Miq", *J. Sci. Soc. Thailand.* 14(1), 67-71.
- Rukachaisirikul, V., Kaewnok, W., Koysomboon, S., Phongpaichit, S. and Taylor, W.C. 2000a. "Caged-Tetraprenylated xanthenes from *Garcinia scortechinii*", *Tetrahedron.* 56(43), 8539-8543.
- Rukachaisirikul, V., Adair, A., Dampawan, P., Taylor, W.C. and Turner, P.C. 2000b. "Lanostanes and friedolanostanes from the pericarp of *Garcinia hombroniana*", *Phytochemistry.* 55, 183-188.
- Rukachaisirikul, V., Pailee, P., Hiranrat, A., Tuchinda, P., Yoosook, C., Kasisit, J., Taylor, W.C. and Reutrakul, V. 2003a. "Anti-HIV-1 protostane triterpenes and digeranylbenzophenone from trunk bark and stems of *Garcinia speciosa*", *Planta Med.* 69, 1141-1146.
- Rukachaisirikul, V., Ritthiwigrom, T., Pinsa, A., Sawangchote, P. and Taylor, W.C. 2003b. "Xanthenes from the stem bark of *Garcinia nigrolineata*", *Phytochemistry.* 64(6), 1149-1156.
- Rukachaisirikul, V., Naklue, W., Sukpondma, Y. and Phongpaichit, S. 2005a. "An antibacterial biphenyl derivative from *Garcinia bancana* MIQ", *Chem. Pharm. Bull.* 53(3), 342-343.
- Rukachaisirikul, V., Saelim, S., Karnsomchoke, P. and Phongpaichit, S. 2005b. "Friedolanostanes and lanostanes from the leaves of *Garcinia hombroniana*", *J. Nat. Prod.* 68(8), 1222-1225.
- Rukachaisirikul, V., Phainuphong, P., Sukpondma, Y., Phongpaichit, S. and Taylor, W.C. 2005c. "Antibacterial caged-tetraprenylated xanthenes from the stem bark of *Garcinia scortechinii*", *Planta Med.* 71, 165-170.

- Rukachaisirikul, V., Tadpetch, K., Watthanaphanit, A., Saengsanae, N. and Phongpaichit, S. 2005d. "Benzopyran, biphenyl, and tetraoxygenated xanthone derivatives from the twigs of *Garcinia nigrolineata*", *J. Nat. Prod.* 68(8), 1218-1221.
- Sherley, G., Kosela, S., Kardono, L.B.S., Hanafi, M. and Harrison, L.J. 2004. "Identification of cytotoxic and antibacterial pyranoxanthenes from *Garcinia porrecta*", *New Technologies & Medicine.* 5(1), 39-43.
- Smitinand, T. 2001. "Thai Plant Names", The Forest Herbarium, The Royal Forest Department.
- Soemiati, A., Kosela, S., Hanafi, M. and Harrison, L.J. 2004. "Elucidation and cytotoxic L1210 evaluation of prenylated pyranoxanthenoids from the indonesian *Garcinia dulcis* fruit (Guttiferae) n-hexane extract", *New Technologies & Medicine.* 5(4), 389-393.
- Sukpondma, Y., 2005. "Chemical constituents from the fruits of *Garcinia scortechinii* and *Garcinia hanburyi*", *Ph. D. Thesis.* Prince of Songkla University, Thailand.
- Sukpondma, Y., Rukachaisirikul, V. and Phongpaichit, S. 2005a. "Antibacterial caged-tetraprenylated xanthenes from the fruits of *Garcinia hanburyi*", *Chem. Pharm. Bull.* 53(7), 850-852.
- Sukpondma, Y., Rukachaisirikul, V. and Phongpaichit, S. 2005b. "Xanthone and sesquiterpene derivatives from the fruits of *Garcinia scortechinii*", *J. Nat. Prod.* 68(7), 1010-1017.
- Suksamrarn, S., Suwannapoch, N., Ratananukul, P., Aroonlerk, N. and Suksamrarn, A. 2002. "Xanthenes from the green fruit hulls of *Garcinia mangostana*", *J. Nat. Prod.* 65(5), 761-763.



- Suksamrarn, S., Suwannapoch, N., Phakhodee, W., Thanuhiranlert, J., Ratananukul, P., Chimnoi, N. and Suksamrarn, A. 2003. "Antimycobacterial activity of prenylated xanthenes from the fruits of *Garcinia mangostana*", *Chem. Pharm. Bull.* 51(7), 857-859.
- Tanaka, N., Takaishi, Y., Shikishima, Y., Nakanishi, Y., Bastow, K., Lee, K.-H., Honda, G., Ito, M., Takeda, Y., Kodzhimatov, O.K. and Ashurmetov, O. 2004. "Prenylated benzophenones and xanthenes from *Hypericum scabrum*", *J. Nat. Prod.* 67, 1870-1875.
- Thoison, O., Fahy, J., Dumontet, V., Chiaroni, A., Riche, C., Tri, M.V. and Sevenet, T. 2000. "Cytotoxic prenylxanthenes from *Garcinia bracteata*", *J. Nat. Prod.* 63, 441-446.
- Thoison, O., Cuong, D.D., Gramain, A., Chiaroni, A., Hung, N.V. and Sevenet, T. 2005. "Further rearranged prenylxanthenes and benzophenones from *Garcinia bracteata*", *Tetrahedron.* 61, 8529-8535.
- Vieira, L.M.M., Kijjoa, A., Silva, A.M.S., Mondranondra, I.-O., Kengthong, S., Gales, L., Damas, A.M. and Herz, W. 2004a. "Lanostanes and friedolanostanes from the bark of *Garcinia speciosa*", *Phytochemistry.* 65, 393-398.
- Vieira, L.M.M., Kijjoa, A., Wilairat, R., Nascimento, M.S.J., Gales, L., Damas, A.M., M.S.Silva, A., Mondranondra, I.-O. and Herz, W. 2004b. "Bioactive friedolanostanes and 11(10<sup>+</sup> 8)-Abeolanostanes from the bark of *Garcinia speciosa*", *J. Nat. Prod.* 67, 2043-2047.
- Wahyuni, F.S., Byrne, L.T., Dachriyanus, Dianita, R., Jubaha, J., Lajis, N.H. and Sargent, M.V. 2004. "A new ring-reduced tetraprenyltoluquinone and a prenylated xanthone from *Garcinia cowa*", *Aust. J. Chem.* 57(3), 223-226.

- Weng, J.-R., Lin, C.-N., Tsao, L.-T. and Wang, J.-P. 2003a. "Novel and anti-inflammatory constituents of *Garcinia subelliptica*", *Chemistry—A European Journal*. 9(9), 1958-1963.
- Weng, J.-R., Lin, C.-N., Tsao, L.-T. and Wang, J.-P. 2003b. "Terpenoids with a new skeleton and novel triterpenoids with anti-inflammatory effects from *Garcinia subelliptica*", *Chemistry—A European Journal*. 9(22), 5520-5527.
- Whitmore, T.C. 1973. "Tree flora of Malaya", Vol. II, Hong Kong : Wing Tai Cheung Printing CO. Ltd.
- Williams, R.B., Hoch, J., Glass, T.E., Evans, R., Miller, J.S., Wisse, J.H. and Kingston, D.G.I. 2003. "A novel cytotoxic guttiferone analogue from *Garcinia macrophylla* from the Surinam rainforest", *Planta Med.* 69(9), 864-866.
- Williams, R.B., Hoch, J., Glass, T.E., Evans, R., Miller, J.S., Wisse, J.H. and Kingston, D.G.I. 2005. "A novel cytotoxic guttiferone analogue from *Garcinia macrophylla* from the Surinam rainforest", *Planta Med.* 71(4), 354-356.
- Xu, Y.-J., Chiang, P.-H., Lai, Y.-H., Vittle, J.J., Wu, X.-H, Tan, B.K.H., Imiyabir, Z. and Goh, S.-H. 2000. "Cytotoxic prenylated depsidonees from *Garcinia parvifolia*", *J. Nat. Prod.* 63, 1361-1363.
- Xu, Y.-J., Lai, Y.-H., Imiyabir, Z. and Goh, S.-H. 2001. "Xanthones from *Garcinia parvifolia*", *J. Nat. Prod.* 64(9), 1191-1195.

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