ALPHA-GLUCOSIDASE INHIBITORS FROM *DENDROBIUM SCABRILINGUE*

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จุฬาลงกรณ์มหาวิทยาลัย
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สารที่มีฤทธิ์ยับยั้งเอนไซม์แอลฟา-กลูโคซิเดสจากเอื้องแซะ

ร.อ.เฉลิมพร สารกูลวัฒนา

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วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาเภสัชศาสตรมหาบัณฑิต
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เฉลิมพร สารกูลวัฒนา : สารที่มีฤทธิ์ยับยั้งเอนไซม์แอลฟา-กลูโคซิเดสจากเอื้องแซะ. (ALPHA-GLUCOSIDASE INHIBITORS FROM *DENDROBIUM SCABRILINGUE*) อ.ที่ปรึกษาวิทยานิพนธ์หลัก : รศ. ภก. ดร.บุญชู ศรีตุลารักษ์, อ.ที่ปรึกษาวิทยานิพนธ์ร่วม : ศ. ภก. ดร.กิตติศักดิ์ ลิขิตวิทยาวุฒิ

การศึกษาในเชิงพฤกษเคมีของสารสกัดหยาบด้วยเมทานอลของทั้งต้นเอื้องแซะ (วงศ์ Orchidaceae) พบว่า สามารถแยกสกัดสารบริสุทธิ์ชนิดใหม่ได้ 2 ชนิด ได้แก่ dendroscabrols A และ B ซึ่งเป็นสารกลุ่ม phenanthrene และ bisbibenzyl ตามลำดับ และนอกจากนี้ยังพบสารที่เคยมีการรายงานไว้แล้วอีก 8 ชนิด คือ (*Z*)-ferulic acid tetracosyl ester, (*E*)-ferulic acid tetracosyl ester, gigantol, batatasin III, coelonin, aloifol I, lusianthridin และ RF-3192C สารที่สกัดแยกมาได้ทุกชนิดได้รับการพิสูจน์โครงสร้างเคมีโดยใช้ข้อมูลหลักทางสเปกโทรสโกปี และถูกนำไปทดสอบฤทธิ์ยับยั้งเอนไซม์แอลฟา-กลูโคซิเดส Dendroscabrol B และ RF-3192C แสดงฤทธิ์ยับยั้งเอนไซม์แอลฟา-กลูโคซิเดสได้สูงที่สุด นอกจากนี้พบว่าสาร dendroscabrol A, gigantol, coelonin และ lusianthridin มีฤทธิ์ยับยั้งเอนไซม์นี้ได้ดี เมื่อเทียบกับ acarbose ซึ่งใช้เป็น positive control

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5976124033 : MASTER OF SCIENCE IN PHARMACY
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Chalermporn Sarakulwattana : ALPHA-GLUCOSIDASE INHIBITORS FROM *DENDROBIUM SCABRILINGUE* . ADVISOR: Assoc. Prof. Boonchoo Sritularak, Ph.D.,Prof. Kittisak Likhitwitayawuid, Ph.D.

Phytochemical study of the methanol extract of the whole plant of *Dendrobium scabrilingue* Lindl. (Orchidaceae) resulted in the isolation of a new phenanthrene and a new bisbibenzyl namely dendroscabrols A and B, respectively, along with eight known compounds, identified as (*Z*)-ferulic acid tetracosyl ester, (*E*)-ferulic acid tetracosyl ester, gigantol, batatasin III, coelonin, aloifol I, lusianthridin and RF-3192C. The structures of these isolated compounds were determined mainly by spectroscopic data. All isolates were examined for alpha-glucosidase inhibitory activity. Dendroscabrol B and RF-3192C exhibited the most potent activity. In addition, dendroscabrol A, gigantol, coelonin and lusianthridin displayed strong activity compared with the positive control acarbose.

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ABBREVIATIONS AND SYMBOLS

Acetone-d_6	=	Deuterated acetone
br s	=	Broad singlet (for NMR spectra)
°C	=	Degree celsius
CC	=	Column chromatography
$CDCl_3$	=	Deuterated chloroform
CH_2Cl_2	=	Dichloromethane
cm	=	Centimeter
^{13}C-NMR	=	Carbon-13 Nuclear Magnetic Resonance
1-D NMR	=	One-dimensional Nuclear Magnetic Resonance
2-D NMR	=	Two-dimensional Nuclear Magnetic Resonance
d	=	Doublet (for NMR spectra)
dd	=	Doublet of doublets (for NMR spectra)
δ	=	Chemical shift
DEPT	=	Distortionless Enhancement by Polarization Transfer
DMSO-d_6	=	Deuterated dimethylsulfoxide
ε	=	Molar absorptivity
ESI-MS	=	Electrospray Ionization Mass Spectrometry
EtOAc	=	Ethyl acetate
FCC	=	Flash Column Chromatography
g	=	Gram
Gal	=	Galactose
GF	=	Gel Filtration

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Glc	=	Glucose
HMBC	=	^{1}H-detected Heteronuclear Multiple Bond Correlation
HR-ESI-MS	=	High Resolution Electrospray Ionization Mass Spectrometry
^{1}H-NMR	=	Proton Nuclear Magnetic Resonance
HSQC	=	^{1}H-detected Heteronuclear Single Quantum Coherence
Hz	=	Hertz
IC_{50}	=	Concentration exhibiting 50% inhibition
IR	=	Infrared
J	=	Coupling constant
Kg	=	Kilogram
L	=	Liter
λ_{max}	=	Wavelength at maximal absorption
$[M+Na]^{+}$	=	Sodium-adduct molecular ion
$[M-H]^{-}$	=	Pseudomolecular ion
m	=	Multiplet (for NMR spectra)
MeOH	=	Methanol
mg	=	Milligram
µg	=	Microgram
min	=	Minute
mL	=	Milliliter
µL	=	Microliter
µM	=	Micromolar
mm	=	Millimeter

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mM	=	Millimolar
MS	=	Mass spectrum
MW	=	Molecular weight
m/z	=	Mass to charge ratio
N/A	=	Thai name not available
NA	=	No activity
nm	=	Nanometer
NMR	=	Nuclear Magnetic Resonance
NOESY	=	Nuclear Overhauser Effect Spectroscopy
ν_{max}	=	Wave number at maximal absorption
OEt	=	Ethoxy group
OMe	=	Methoxy group
Rha	=	Rhamnose
s	=	Singlet (for NMR spectra)
t	=	Triplet (for NMR spectra)
TLC	=	Thin Layer Chromatography
UV-VIS	=	Ultraviolet and Visible spectrophotometry
VLC	=	Vacuum Liquid Column Chromatography
Xyl	=	Xylose

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CHAPTER I

INTRODUCTION

Diabetes mellitus (DM) is a complex, chronic metabolic disorder requiring continuous medical care. The major symptom is hyperglycemia (high blood glucose level) which is due to problems with the hormone insulin, either its insufficient production or ineffective usage. Prolonged uncontrolled hyperglycemia can cause various serious complications such as kidney disease, retinopathy, cardiovascular disease which contribute to disability and mortality (Choudhury *et al.*, 2018).

DM can be classified into 4 types according to causes (American Diabetes Association, 2018) as follows.

1. Type 1 DM, which was called “insulin-dependent diabetes” or “juvenile-onset diabetes”, it is usually due to cellular-mediated autoimmune destruction of the β-cells in pancrease. The type 1 DM patients was normally absolute insulin deficiency and not typically obese.
2. Type 2 DM, which was called “noninsulin-dependent diabetes” or “adult-onset diabetes”. This form frequnctly encompasses individuals who have peripheral insulin resistance. At least the initial stage, and often throughout their lifetime, the insulin treatment may not necessary for these patients. The specific etiologies are not known but not detect the autoimmune destruction of β-cells.
3. Gestational DM, which is diagnosed by any degree of glucose intolerance that is first recognize during pregnancy, regardless of whether the symptom may be self-recoverd or persisted after the delivery.
4. Specific types of DM due to other causes i.e. diseases of the pancreas (such as cystic fibrosis and pancreatitis) and chemical-induced diabetes (such as long-term usage of glucocorticoid)

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The major type of DM is Type 2 DM which has been observed in more than 90% of the patients. Most patients with type 2 DM are overweight or obese. Overweight itself can cause insulin resistance. Others who are not obese may have an increased distribution of body fat predominantly in the abdominal region. The risk factors of type 2 DM increase with age, obesity, and lack of physical activity. Furthermore, type 2 DM often shows a strong genetic association, especially in first degree relatives (Choudhury *et al.*, 2018).

DM treatments are aimed at decreasing and controlling blood glucose to the desirable level. There are a lot of pharmacological agents currently used for that objective. The first line drugs for type 2 DM are oral anti-diabetic drugs. These drugs can be categorized based on their mechanisms of action (Chaudhury *et al.*, 2017).

- **Insulin secretagogues :** they lower blood glucose level by increasing insulin secretion in the pancreas. There are sevral groups of drugs divied by chemical structure and the target of action, i.e. sulfonylureas (glibenclamide, gliclazide), meglitinides (repaglinide), DPP-4 inhibitors (sitagliptin) and GLP-1 receptor agonists (Exenatide)
- **Insulin sensitisers :** they lower blood glucose level by improves peripheral tissues insulin sensitivity by activating insulin receptor expression and decrease gluconeogenesis in liver. There are two groups of drugs including biguanides (metformin) and thiazolidinediones (pioglitazone).
- **SGLT2 inhibitors :** they lower blood glucose level by blocking glucose reabsorption in the kidney resulting from the inhibition of sodium-glucose cotransporter. This is a new class of oral anti-diabetic agent and there are a few drugs including of canagliflozin, dapagliflozin and empagliflozin.
- **α-Glucosidase inhibitors** i.e. acarbose, miglitol

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α-Glucosidase is a digestive enzyme, secreted from small intestine epithelium, which is responsible for digestion of carbohydrates into monosaccharides prior to absorption. This enzyme has been a target of antidiabetic drug discovery. The class of these drugs is called α-glucosidase inhibitors. It can retard carbohydrate digestion and absorption by blocking the activity of α-glucosidase enzyme (Yin *et al.*, 2014).

There are several natural sources of α-glucosidase inhibitors. Initially, they were isolated from bacterial cultures. Their derivatives such as acarbose was isolated from *Actinoplanes* spp. and miglitol, a semisynthetic derivative of 1-deoxynojirimycin, from *Bacillus* and *Streptomyces* spp. (Kalra, 2014). Medicinal plants are important sources of α-glucosidase inhibitors from their secondary metabolites such as alkaloids, flavonoids, phenylpropanoids, phenols, quinones, steroids, terpenes and other classes of compounds (Yin *et al.*, 2014).

Recently, there have been reports about α-glucosidase inhibiting compounds including flavonoids, bibenzyls and phenanthrenes from many plants in the genus *Dendrobium*, for example *D. loddigesii* (Lu *et al.*, 2014a), *D. devonianum* (Sun *et al.*, 2014), *D. totile* (Limpanit *et al.*, 2016), *D. formosum* (Inthongkaew *et al.*, 2017) and *D. infundibulum* (Na Ranong *et al.*, 2018).

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Dendrobium is one of the largest genus in the Orchidaceae family. There are more than 1,500 species distributed throughout Asia and Australia. *Dendrobium* plants have been used as traditional medicines in Eastern Asia countries for a thousand years. For example, in China thirty *Dendrobium* species are collectively called “Shihu” and used as medicinal herbs for various purposes, such as nourishing the stomach, increasing production of body fluids or nourishing Yin (Cakova *et al.*, 2017). In Thailand, more than 150 *Dendrobium* species have been identified as follows (Office of the Forest Herbarium, 2014).

Dendrobium acerosum Lindl.	**กล้วยไม้มือนาง** Kluai mai mue nang
D. aciculare Lindl.	**เอื้องใบเข็ม** Ueang bai khem
D. acinaciforme Roxb.	**เอื้องยอดสร้อย** Ueang yot soi
D. aduncum Lindl.	N/A
D. albosanguineum Lindl.	**เอื้องตางัว** Ueang ta ngua
D. aloifolium (Blume) Rchb.f.	**เอื้องมณี** Ueang mani
D. anceps Sw.	N/A
D. angulatum Lindl.	N/A
D. anosmum Lindl.	**เอื้องสาย** Ueang sai
D. aphyllum (Roxb.) C.E.C.Fisch.	**เอื้องงวงช้าง** Ueang nguang chang
D. bellatulum Rolfe	**เอื้องแซะภู** Ueng sae phu
D. bensoniae Rchb.f.	N/A
D. bicameratum Lindl.	**เอื้องเข็ม** Ueang khem
D. bifarium Lindl.	N/A
D. bilobulatum Seidenf.	**กล้วยไม้ก้างปลา** Kluai mai kang pla
D. blumei Lindl.	N/A
D. brevimentum Seidenf.	N/A
D. brymerianum Rchb.f.	**เอื้องคำฝอย** Ueang kham foi
D. calicopis Ridl.	N/A
D. capillipes Rchb.f.	**เอื้องคำกิ่ว** Ueang kham kio
D. cariniferum Rchb.f.	**เอื้องกาจก** Ueang kachok
D. chittimae Seidenf.	**เอื้องจิตติมา** Ueang chittima
D. christyanum Rchb.f.	**เอื้องแซะภูกระดึง** Ueang sae phu kradueng
D. chrysanthum Lindl.	**เอื้องสายมรกต** Ueang sai morakot

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D. chrysotoxum Lindl.	**เอื้องคำ** Ueang kham
D. ciliatilabellum Seidenf.	**หวายเขาเขียว** Wai khao khiao
D. clavator Ridl.	N/A
D. compactum Rolfe ex Hackett	**เอื้องข้าวตอก** Ueang khao tok
D. compressum Lindl.	**หวายแบนตะนาวศรี** Wai baen tanao si
D. concinnum Miq.	**หางเปีย** Hang pia
D. confinale Kerr	N/A
D. cowenii P.O'Byrne & J.J.Vern.	N/A
D. crepidatum Lindl. & Paxton	**เอื้องสายน้ำเขียว** Ueang sai nam khiao
D. cretaceum Lindl.	N/A
D. crocatum Hook.f.	**เอื้องนางนวล** Ueang nang nuan
D. cruentum Rchb.f.	**เอื้องนกแก้ว** Ueang nok kaeo
D. crumenatum Sw.	**หวายตะมอย** Wai tamoi
D. crystallinum Rchb.f.	**เอื้องนางฟ่อน** Ueang nang fon
D. cumulatum Lindl.	**เอื้องสายสี่ดอก** Ueang sai si dok
D. curviflorum Rolfe	N/A
D. cuspidatum Lindl.	N/A
D. dantaniense Guillaumin	**เอื้องเข็ม** Ueang khem
D. delacourii Guillaumin	**เอื้องดอกมะขาม** Ueang dok ma kham
D. deltatum Seidenf.	N/A
D. denneanum Kerr	N/A
D. densiflorum Lindl.	**เอื้องมอนไข่** Ueang mon khai
D. denudans D.Don	**เอื้องสายจำปา** Ueang sai champa
D. devonianum Paxton	**เอื้องเมี่ยง** Ueang miang

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D. dickasonii L.O.Williams	**เอื้องเคี้ยะ** Ueang khia
D. dixanthum Rchb.f.	**เอื้องเทียน** Ueang thian
D. dixonianum Rolfe ex Downie	N/A
D. draconis Rchb.f.	**เอื้องเงิน** Ueang ngoen
D. elliottianum P.O'Byrne	**หวายเจดีย์** Wai chedi
D. ellipsophyllum Tang & F.T.Wang	**เอื้องทอง** Ueang thong
D. erostelle Seidenf.	N/A
D. erosum (Blume) Lindl.	N/A
D. eserre Seidenf.	N/A
D. exile Schltr.	**เอื้องเสี้ยน** Ueang sian
D. falconeri Hook.	**เอื้องสายวิสูตร** Ueang sai wisut
D. farmeri Paxton	**เอื้องมัจฉาณุ** Ueang matchanu
D. fimbriatum Hook.	**เอื้องคำน้อย** Ueang kham noi
*D. findlayanu*m Parish & Rchb.f.	**พวงหยก** Phuang yok
D. flexile Ridl.	N/A
D. formosum Roxb. ex Lindl.	**เอื้องเงินหลวง** Ueang ngoen luang
D. friedericksianum Rchb.f.	**เอื้องเหลืองจันทบูร** Ueang lueang chantabun
D. fuerstenbergianum Schltr.	**เอื้องแซะภูกระดึง** Ueang sae phukradueng
D. fychianum Bateman ex Rchb.f.	**หวายพม่า** Wai phama
D. garrettii Seidenf.	**หวายการ์เร็ต** Wai karet
D. gibsonii Paxton	**เอื้องคำสาย** Ueang kham sai
D. grande Hook.f.	**เอื้องแผงใบใหญ่** Ueang pheang bai yai
D. gratiotissimum Rchb.f.	**เอื้องกิ่งดำ** Ueang king dam
D. gregulus Seidenf.	**เอื้องมะต่อม** Ueang ma tom

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D. griffithianum Lindl.	**เอื้องมัจฉาณุ** Ueang matchanu
D. harveyanum Rchb.f.	**เอื้องคำฝอย** Ueang kham foi
D. hendersonii Hawkes & Heller	**หวายตะมอยน้อย** Wai tamoi noi
D. henryi Schltr.	**เอื้องสุริยัน** Ueang suriyan
D. hercoglossum Rchb.f.	**เอื้องดอกมะเขือ** Ueang dok ma kuea
D. heterocarpum Lindl.	**เอื้องสีตาล** Ueang si tan
D. hymenanthum Rchb.f.	**เอื้องน้อยกลีบบาง** Ueang noi klip bang
D. hymenopterum Hook.f.	N/A
D. incurvum Lindl.	N/A
D. indivisum (Blume) Miq. var. *indivisum*	**ตานเสี้ยนไม้** Tan sian mai
D. indivisum (Blume) Miq. var. *lampangense* Rolfe	N/A
D. indivisum (Blume) Miq. var. *pallidum* Seidenf.	**ก้างปลา** Kang pla
D. indragiriense Schltr.	N/A
D. infundibulum Lindl.	**เอื้องตาเหิน** Ueang ta hoen
D. intricatum Gagnep.	**เอื้องชมพู** Ueang chomphu
D. jenkinsii Wall. ex Lindl.	**เอื้องผึ้งน้อย** Ueang phueng noi
D. kanburiense Seidenf.	**หวายเมืองกาญจน์** Wai muang kan
D. keithii Ridl.	**หางเปีย** Hang pia
D. kentrophyllum Hook.f.	N/A
D. kontumense Gagnep.	**เอื้องเงินวิลาศ** Ueang ngoen wilat
D. kratense Kerr	N/A
D. lagarum Seidenf.	N/A

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D. lampongense J.J.Sm.	N/A
D. lamyaiae Seidenf.	N/A
D. leonis (Lindl.) Rchb.f.	**เอื้องตะขาบใหญ่** Ueang ta khap yai
D. lindleyi Steud.	**เอื้องผึ้ง** Ueang phueng
D. linguella Rchb.f.	N/A
D. lituiflorum Lindl.	**เอื้องสายม่วง** Ueang sai muang
D. lueckelianum Fessel & Wolff	N/A
D. mannii Ridl.	**เอื้องหางปลา** Ueang hang pla
D. metachilinum Rchb.f.	N/A
D. monticola Hunt & Summerh.	N/A
D. moschatum (Buch.-Ham.) Sw.	**เอื้องจำปา** Ueang champa
D. mucronatum Seidenf.	N/A
D. nanocompactum Seidenf.	N/A
D. nathanielis Rchb.f.	**เกล็ดนิ่ม** Klet nim
D. ochreatum Lindl.	**เอื้องตะขาบ** Ueang ta khap
D. oligophyllum Gagnep.	**ข้าวตอกปราจีน** Khao tok prachin
D. pachyglossum Parish & Rchb.f	**เอื้องขนหมู** Ueang khon mu
D. pachyphyllum (Kuntze) Bakh.f.	**เอื้องน้อย** Ueang noi
D. palpebrae Lindl.	**เอื้องมัจฉาณุ** Ueang matchanu
D. pandaneti Ridl.	N/A
D. panduriferum Hook.f.	N/A
D. parciflorum Rchb.f. ex Lindl.	**เอื้องดอกขาวใบแบน** Ueang dok khao bai baen
D. parcum Rchb.f.	**เอื้องก้านกิ่ว** Ueang kan kio

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D. parishii Rchb.f.	**เอื้องครั่ง** Ueang khrang
D. parvum Seidenf.	N/A
D. peguanum Lindl.	**หวายเปกู** Wai peku
D. pendulum Roxb.	**เอื้องไม้เท้าฤาษี** Ueang mai thao ruesi
D. perpaulum Seidenf.	**เอื้องข้าวตอกอินทนนท์** Ueang khao tok inthanon
D. planibulbe Lindl.	N/A
D. polyanthum Wall. ex Lindl.	**เอื้องสายประสาท** Ueang sai prasat
D. porphyrochilum Lindl.	**เอื้องเฉวียน** Ueang chawian
D. praecinctum Rchb.f.	**หวายภูหลวง** Wai phu luang
D. proteranthum Seidenf.	**หวายน้อยภูหลวง** Wai noi phu luang
D. pulchellum Roxb. ex Lindl.	**เอื้องคำตาควาย** Ueang kham ta khwai
D. pychnostachyum Lindl.	**เศวตสอดสี** Sawet sot si
D. rhodostele Ridl.	N/A
D. salaccense (Blume) Lindl.	**เอื้องใบไผ่** Ueang bai phai
D. sanguinolentum Lindl.	N/A
D. scabrilingue Lindl.	**เอื้องแซะ** Ueang sae
D. schilhaueri Ormerod & Pedersen	N/A
D. secundum (Blume) Lindl.	**เอื้องแปรงสีฟัน** Ueang preang si fan
D. senile Parish & Rchb.f.	**เอื้องชะนี** Ueang chani
D. setifolium Ridl.	N/A
D. signatum Rchb.f.	**เอื้องเค้ากิ่ว** Ueang khao kio
D. singaporense Hawkes & Heller	N/A
D. sinuatum (Lindl.) Lindl. ex Rchb.f.	N/A

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D. sociale J.J.Sm.	N/A
D. strongylanthum Rchb.f.	**เอื้องเย้าลม** Ueang yao lom
D. stuposum Lindl.	**เอื้องสาย** Ueang sai
D. subulatum (Blume) Lindl.	N/A
D. sukhakulii hort.	**หวายสุขะกุล** Wai sukhakun
D. sulcatum Lindl.	**เอื้องจำปาน่าน** Ueang champa nan
D. superbiens Rchb.f.	**หวายคิง** Wai khing
D. sutepense Rolfe ex Downie	**เอื้องมะลิ** Ueang mali
D. terminale Parish & Rchb.f	**เอื้องแผงโสภา** Ueang phaeng sopha
D. thyrsiflorum Rchb.f	**เอื้องมอนไข่ใบมน** Ueang mon khai bai mon
D. tortile Lindl.	**เอื้องไม้ตึง** Ueang mai tueng
D. trigonopus Rchb.f.	**เอื้องคำเหลี่ยม** Ueang kham liam
D. trinervium Ridl.	**เทียนลิง** Thian ling
D. truncatum Lindl.	N/A
D. umbonatum Seidenf.	N/A
D. unicum Seidenf.	**เอื้องครั่งแสด** Ueang krang saet
D. uniflorum Griff.	**เอื้องทอง** Ueang thong
D. venustum Teijsm. & Binn	**ข้าวเหนียวลิง** Khao niao ling
D. villosulum Lindl.	**กล้วยหญ้านา** Kluai ya na
D. viridulum Ridl.	N/A
D. wardianum R.Warner	**เอื้องมณีไตรรงค์** Ueang mani trairong
D. wattii (Hook.f.) Rchb.f.	**เอื้องแซะ** Ueang sae
D. williamsonii Day & Rchb.f.	N/A
D. xanthophlebium Lindl.	N/A

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D. ypsilon Seidenf. **เอื้องแบนปากตัด** Ueang baen pak tat

Dendrobium scabrilingue Lindl. has been found in Myanmar, Laos and Thailand. In Thailand it is known as "Ueang Sae (เอื้องแซะ)" and distributed in dry evergreen forests in northern, northeastern and eastern regions. *D. scabrilingue* is an epiphytic orchid with terete pseudobulbs 15 -30 cm in length, covered with black hairs. Its leaves are elliptic, 5 – 6 cm long, 1.5 – 2.5 cm wide. The flowers, 2.5 – 3 cm in diameter, arise near the apex of pseudobulb and are strongly fragrant. Sepals and petals are white to greenish white with greenish-yellow to orangish-yellow lip. The flowering period is during December to February (Vaddhanaphuti, 2005; The Botanical Garden Organization, 2008).

Prior to this investigation, there were no studies on the phytochemical constituents and biological activities of *D. scabrilingue*. So, I was interested in its chemical constituents and their α-glucosidase inhibitory activity. In the preliminary study, there were more than 80% α-glucosidase inhibition for the MeOH extract at 100 µg/ml. Then the crude extract was seoerated into 3 parts and found that only the ethyl acetate part showed a strong inhibition to α-glucosidase enzyme (> 90%) at the same concentration.

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The major objectives of this study were as follows.

1. To isolate and purify the chemical constituents from *Dendrobium scabrilingue*.

2. To determine the chemical structure of each isolated compound.

3. To evaluate each isolated compound for its α-glucosidase inhibitory activity.

Figure 1 *Dendrobium scabrilingue* Lindl.

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CHAPTER II

HISTORICAL

1. Chemical constituents of *Dendrobium* species

Dendrobium orchids have been reported as the source of several secondary metabolites. They can be categorized into several classes of chemical structures, such as bibenzyls, flavonoids, terpenoids and miscellaneous compounds (**Figures 2-5**).

Bibenzyls and their derivatives from *Dendrobium*, as shown in **Table 1**, are members of the stilbene group. The stilbenoid backbone is biosynthetically derived from one cinnamic acid-CoA and three malonyl-CoA units. Cinnamic acid, formed via shikimate pathway, is activated by hydroxylation to form 4-coumaroyl-CoA. Then, three malonyl-CoA is attached to the 4-coumaroyl-CoA by stilbene synthase enzyme and the extended structure is cyclized to form an unstable tetraketide intermediate, which is transformed to a stilbenoid (stilbenes, bibenzyls, phenanthrenes, 9,10-dihydro-phenanthrenes) or a chalcone, the precursor of flavonoids. The chalcone structure is further modified by glycosylation, methylation and hydroxylation to provide the variety of flavonoid structures (Dubrovina and Kiselev, 2017), as shown in **Table 2.**

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Terpenoid compounds, as shown in **Table 3**, are produced from C_5 unit called isoprene. Isoprene unit is created by two pathways including the mevalonic acid pathway and the methylerythritol phosphate pathway. Terpenoids can be classified by the number of C_5 isoprene units as hemiterpenes (1 unit), monoterpenes (2 units), sesquiterpenes (3 units), diterpenes (4 units), sesterterpenes (5 units), triterpenes (6 units), tetraterpenes (8 units), and polyterpenes (more than 9 units) (Schrader and Bohlmann, 2015).

Dendrobium plants produce not only these metabolites, but also several minor constituents including aliphatic compounds, benzoic acid derivatives, phenylpropanoids and fluorenones which are grouped together as miscellaneous compounds in **Table 4.**

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Table 1 Distribution of bibenzyls and derivatives in *Dendrobium* plants

Compounds	Plant	Plant part	Reference
Aloifol I [**1**]	*D. infundibulum*	Whole plant	Na Ranong *et al.*, 2018
	D. longicornu	Stem	Hu *et al.*, 2008b
	D. williamsonii	Whole plant	Yang *et al.*, 2017b
Amoenylin [**2**]	*D. amoenum*	Whole plant	Majumder *et al.*, 1999
	D. williamsonii	Whole plant	Yang *et al.*, 2017b
4,3′,4′-Trihydroxy-3,5-dimethoxybibenzyl [**3**]	*D. parishii*	Whole plant	Kongkatitham *et al.*, 2018
Batatasin [**4**]	*D. longicornu*	Stem	Hu *et al.*, 2008b
	D. plicatile	Stem	Yamaki and Honda, 1996
Batatasin III [**5**]	*D. aphyllum*	Stem	Yang *et al.*, 2015a
	D. cariniferum	Stem	Chen *et al.*, 2008c
	D. chrysotoxum	Whole plant	Li *et al.*, 2009c
	D. draconis	Stem	Sritularak *et al.*, 2011a
	D. formosum	Whole plant	Inthongkaew *et al.*, 2017
	D. gratiosissimum	Stem	Chen *et al.*, 2008b
	D. infundibulum	Whole plant	Na Ranong *et al.*, 2018
	D. loddigesii	Stem	Ito *et al.*, 2010b
	D. venustum	Whole plant	Sukphan *et al.*, 2014

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Table 1 (continued)

Compounds	Plant	Plant part	Reference
Brittonin A [**6**]	*D. secundum*	Stem	Sritularak *et al.*, 2011b
Chrysotobibenzyl [**7**]	*D. aurantiacum* var. *denneanum*	Stem	Yang *et al.*, 2006b
	D. capillipes	Stem	Phechrmeekha *et al.*, 2012
	D. chrysanthum	Stem	Yang *et al.*, 2006a
	D. chrysotoxum	Stem	Hu *et al.*, 2012
	D. nobile	Stem	Zhang *et al.*, 2007b
	D. pulchellum	Stem	Chanvorachote *et al.*, 2013
Chrysotoxine [**8**]	*D. aurantiacum* var. *denneanum*	Stem	Yang *et al.*, 2006b
	D. chrysanthum	Stem	Yang *et al.*, 2006a
	D. nobile	Stem	Zhang *et al.*, 2007b
	D. pulchellum	Stem	Chanvorachote *et al.*, 2013
Crepidatin [**9**]	*D. aurantiacum* var. *denneanum*	Whole plant	Liu *et al.*, 2009b
	D. capillipes	Stem	Phechrmeekha *et al.*, 2012
	D. chrysanthum	Stem	Yang *et al.*, 2006a

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Table 1 (continued)

Compounds	Plant	Plant part	Reference
Crepidatin [**9**] (continued)	*D. crepidatum*	Whole plant	Majumder and Chatterjee, 1989
	D. nobile	Stem	Zhang *et al.*, 2007b
	D. pulchellum	Stem	Chanvorachote *et al.*, 2013
Cumulatin [**10**]	*D. cumulatum*	Whole plant	Majumder and Pal, 1993
Dendrobin A [**11**]	*D. nobile*	Stem	Wang *et al.*, 1985; Ye and Zhao, 2002
3,3′-Dihydroxy-4,5-dimethoxy-bibenzyl [**12**]	*D. williamsonii*	Whole plant	Rungwichaniwat *et al.*, 2014
3,4′-Dihydroxy-5-methoxybibenzyl [**13**]	*D. amoenum*	Whole plant	Majumder *et al.*, 1999
3,4′-Dihydroxy-5,5′-dimethoxydihydro stilbene [**14**]	*D. nobile*	Stem	Hwang *et al.*, 2010
Erianin [**15**]	*D. chrysotoxum*	Stem	Hu *et al.*, 2012
Gigantol [**16**]	*D. aphyllum*	Whole plant	Chen *et al.*, 2008c
	D. aurantiacum var. *denneanum*	Whole plant	Liu *et al.*, 2009b
	D. brymerianum	Whole plant	Klongkumnuankarn *et al.*, 2015

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Table 1 (continued)

Compounds	**Plant**	**Plant part**	**Reference**
Gigantol [**16**]	*D. densiflorum*	Stem	Fan *et al.*, 2001
(continued)	*D. devonianum*	Whole plant	Sun *et al.*, 2014
	D. draconis	Stem	Sritularak *et al.*, 2011a
	D. gratiosissimum	Stem	Chen *et al.*, 2008b
	D. formosum	Whole plant	Inthongkaew *et al.*, 2017
	D. loddigesii	Whole plant	Ito *et al.*, 2010b
	D. longicornu	Stem	Hu *et al.*, 2008b
	D. nobile	Stem	Zhang *et al.*, 2007b
	D. officinale	Stem	Zhao *et al.*, 2018
	D. palpebrae	Whole plant	Kyokong *et al.*, 2018
	D. polyanthum	Stem	Hu *et al.*, 2009
	D. trigonopus	Stem	Hu *et al.*, 2008a
	D. venustum	Whole plant	Sukphan *et al.*, 2014
	D. wardianum	Stem	Zhang *et al.*, 2017
Gigantol-5-*O*-β-D-glucopyranoside [**17**]	*D. fimbriatum*	Stem	Xu *et al.*, 2017
4-Hydroxy-3,5,3′-trimethoxybibenzyl [**18**]	*D. nobile*	Stem	Ye and Zhao, 2002

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Table 1 (continued)

Compounds	Plant	Plant part	Reference
5-Hydroxy-3,4,3′,4′, 5′-pentamethoxy-bibenzyl [**19**]	*D. secundum*	Stem	Phechrmeekha *et al.*, 2012
3,4′-Dihydroxy-3′,4,5-trimethoxybibenzyl [**20**]	*D. infundibulum*	Whole plant	Na Ranong *et al.*, 2018
Isoamoenylin [**21**]	*D. amoenum*	Whole plant	Majumder *et al.*, 1999
Moscatilin [**22**]	*D. amoenum*	Whole plant	Majumder *et al.*, 1999
	D. aurantiacum var. *denneanum*	Stem	Yang *et al.*, 2006b
	D. brymerianum	Whole plant	Klongkumnuankarn *et al.*, 2015
	D. chrysanthum	Stem	Yang *et al.*, 2006a
	D. densiflorum	Stem	Fan *et al.*, 2001
	D. ellipsophyllum	Whole plant	Tanagornmeatar *et al.*, 2014
	D. formosum	Whole plant	Inthongkaew *et al.*, 2017
	D. gratiosissimum	Stem	Chen *et al.*, 2008b
	D. infundibulum	Whole plant	Na Ranong *et al.*, 2018
	D. loddigesii	Whole plant	Chen *et al.*, 1994; Ito *et al.*, 2010b
	D. longicornu	Stem	Hu *et al.*, 2008b

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Table 1 (continued)

Compounds	Plant	Plant part	Reference
Moscatilin [**22**] (continued)	*D. moscatum*	Whole plant	Majumder and Sen, 1987
	D. nobile	Stem	Miyazawa *et al.*, 1999; Yang *et al.*, 2007
	D. palpebrae	Whole plant	Kyokong *et al.*, 2018
	D. parishii	Whole plant	Kongkatitham *et al.*, 2018
	D. polyanthum	Stem	Hu *et al.*, 2009
	D. pulchellum	Stem	Chanvorachote *et al.*, 2013
	D. secundum	Stem	Sritularak *et al.*, 2011b
	D. wardianum	Stem	Zhang *et al.*, 2017
	D. williamsonii	Whole plant	Yang *et al.*, 2017b
Moscatilin diacetate [**23**]	*D. loddigesii*	Stem	Chen *et al.*, 1994
3,3′,4-Trihydroxy bibenzyl [**24**]	*D. longicornu*	Stem	Hu *et al.*, 2008b
3,3′,5-Trihydroxy bibenzyl [**25**]	*D. cariniferum*	Whole plant	Liu *et al.*, 2009a
3,5,4′-Trihydroxy bibenzyl [**26**]	*D. gratiosissimum*	Stem	Chen *et al.*, 2008b
4,5,4′-Trihydroxy-3,3′-dimethoxy bibenzyl [**27**]	*D. ellipsophyllum*	Whole plant	Tanagornmeatar *et al.*, 2014
	D. palpebrae	Whole plant	Kyokong *et al.*, 2018
	D. parishii	Whole plant	Kongkatitham *et al.*, 2018
	D. secundum	Stem	Sritularak *et al.*, 2011b

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Table 1 (continued)

Compounds	Plant	Plant part	Reference
Tristin [**28**]	*D. aphyllum*	Stem	Yang *et al.*, 2015a
	D. chrysotoxum	Stem	Hu *et al.*, 2012
	D. densiflorum	Stem	Fan *et al.*, 2001
	D. gratiosissimum	Stem	Chen *et al.*, 2008b
	D. longicornu	Stem	Hu *et al.*, 2008b
	D. officinale	Stem	Zhao *et al.*, 2018
	D. trigonopus	Stem	Hu *et al.*, 2008a
Dendromoniliside E [**29**]	*D. nobile*	Stem	Miyazawa *et al.*, 1999
Dendrophenol [**30**]	*D. candidum*	Stem	Li *et al.*, 2008
3,4-Dihydroxy-5,4′-dimethoxybibenzyl [**31**]	*D. candidum*	Stem	Li *et al.*, 2008
	D. signatum	Whole plant	Mittraphab *et al.*, 2016
	D. tortile	Whole plant	Limpanit *et al.*, 2016
	D. wardianum	Stem	Zhang *et al.*, 2017
	D. williamsonii	Whole plant	Yang *et al.*, 2017b
4,4′-Dihydroxy-3,5-dimethoxybibenzyl [**32**]	*D. candidum*	Stem	Li *et al.*, 2008
	D. ellipsophyllum	Whole plant	Tanagornmeatar *et al.*, 2014
	D. williamsonii	Whole plant	Yang *et al.*, 2017b
Loddigesiinol C [**33**]	*D. loddigesii*	Whole plant	Ito *et al.*, 2010b
3-*O*-Methylgigantol [**34**]	*D. candidum*	Stem	Li *et al.*, 2008
	D. plicatile	Stem	Yamaki and Honda, 1996

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Table 1 (continued)

Compounds	Plant	Plant part	Reference
Dendrocandin A [**35**]	*D. candidum*	Stem	Li *et al.*, 2008
	D. wardianum	Stem	Zhang *et al.*, 2017
Dendrocandin C [**36**]	*D. candidum*	Stem	Li *et al.*, 2009b
Dendrocandin D [**37**]	*D. candidum*	Stem	Li *et al.*, 2009b
Dendrocandin E [**38**]	*D. candidum*	Stem	Li *et al.*, 2009b
	D. parishii	Whole plant	Kongkatitham *et al.*, 2018
Dendrocandin B [**39**]	*D. candidum*	Stem	Li *et al.*, 2008
	D. signatum	Whole plant	Mittraphab *et al.*, 2016
	D. officinale	Stem	Yang *et al.*, 2015b
Dendrocandin T [**40**]	*D. officinale*	Stem	Yang *et al.*, 2015b
Dendrocandin U [**41**]	*D. officinale*	Stem	Yang *et al.*, 2015b
	D. wardianum	Stem	Zhang *et al.*, 2017
Dendrocandin V [**42**]	*D. wardianum*	Stem	Zhang *et al.*, 2017
Dendrocandin F [**43**]	*D. candidum*	Stem	Li *et al.*, 2009b
Dendrocandin G [**44**]	*D. candidum*	Stem	Li *et al.*, 2009b
Dendrocandin H [**45**]	*D. candidum*	Stem	Li *et al.*, 2009b
Dendrocandin I [**46**]	*D. candidum*	Stem	Li *et al.*, 2009b
	D. signatum	Whole plant	Mittraphab *et al.*, 2016
Dendrosinen A [**47**]	*D. sinense*	Whole plant	Chen *et al.*, 2014
Dendrosinen B [**48**]	*D. sinense*	Whole plant	Chen *et al.*, 2014
	D. infundibulum	Whole plant	Na Ranong *et al.*, 2018

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Table 1 (continued)

Compounds	Plant	Plant part	Reference
Dendrosinen C [**49**]	*D. sinense*	Whole plant	Chen *et al.*, 2014
Dendrosinen D [**50**]	*D. sinense*	Whole plant	Chen *et al.*, 2014
4-[2-(3-Hydroxyphenol)-1-methoxyethyl]-2,6-dimethoxy phenol [**51**]	*D. longicornu*	Stem	Hu *et al.*, 2008b
Nobilin A [**52**]	*D. nobile*	Stem	Zhang *et al.*, 2006
Nobilin B [**53**]	*D. nobile*	Stem	Zhang *et al.*, 2006
Nobilin C [**54**]	*D. nobile*	Stem	Zhang *et al.*, 2006
Nobilin D [**55**]	*D. nobile*	Stem	Zhang *et al.*, 2007b
Nobilin E [**56**]	*D. nobile*	Stem	Zhang *et al.*, 2007b
Densiflorol A [**57**]	*D. densiflorum*	Stem	Fan *et al.*, 2001
Longicornuol A [**58**]	*D. longicornu*	Stem	Hu *et al.*, 2008b
Trigonopol A [**59**]	*D. trigonopus*	Stem	Hu *et al.*, 2008a
Trigonopol B [**60**]	*D. chrysotoxum*	Stem	Hu *et al.*, 2012
	D. trigonopus	Stem	Hu *et al.*, 2008a
Crepidatuol A [**61**]	*D. crepidatum*	Stem	Li *et al.*, 2013
Crepidatuol B [**62**]	*D. crepidatum*	Stem	Li *et al.*, 2013
Loddigesiinol D [**63**]	*D. loddigesii*	Whole plant	Ito *et al.*, 2010b
Dencryol A [**64**]	*D. crystallinum*	Stem	Wang *et al.*, 2009
Dencryol B [**65**]	*D. crystallinum*	Stem	Wang *et al.*, 2009

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Table 1 (continued)

Compounds	Plant	Plant part	Reference
Dengraol A [**66**]	*D. gratiosissimum*	Stem	Chen *et al.*, 2008b
Dengraol B [**67**]	*D. gratiosissimum*	Stem	Chen *et al.*, 2008b
Dendrofalconerol A [**68**]	*D. falconeri*	Stem	Sritularak and Likhitwitayawuid, 2009
	D. signatum	Whole plant	Mittraphab *et al.*, 2016
	D. tortile	Whole plant	Limpanit *et al.*, 2016
Dendrofalconerol B [**69**]	*D. falconeri*	Stem	Sritularak and Likhitwitayawuid, 2009
Dendrowillol A [**70**]	*D. williamsonii*	Whole plant	Yang *et al.*, 2017b
Dendrosignatol [**71**]	*D. signatum*	Whole plant	Mittraphab *et al.*, 2016
Dendroparishiol [**72**]	*D. parishii*	Whole plant	Kongkatitham *et al.*, 2018
Coelonin [**73**]	*D. aphyllum*	Whole plant	Chen *et al.*, 2008e
	D. formosum	Whole plant	Inthongkaew *et al.*, 2017
	D. nobile	Stem	Yang *et al.*, 2007
9,10-Dihydromoscatin [**74**]	*D. polyanthum*	Stem	Hu *et al.*, 2009
9,10-Dihydrophenanthrene-2,4,7-triol [**75**]	*D. officinale*	Stem	Zhao *et al.*, 2018
	D. polyanthum	Stem	Hu *et al.*, 2009
4,5-Dihydroxy-2,3-dimethoxy-9,10-dihydrophenanthrene [**76**]	*D. ellipsophyllum*	Whole plant	Tanagornmeatar *et al.*, 2014
	D. sinense	Whole plant	Chen *et al.*, 2013

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Table 1 (continued)

Compounds	**Plant**	**Plant part**	**Reference**
Dendroinfundin A [**77**]	*D. infundibulum*	Whole plant	Na Ranong *et al.*, 2018
4,5-Dihydroxy-2,6-dimethoxy-9,10-dihydrophenanthrene [**78**]	*D. chrysotoxum*	Stem	Hu *et al.*, 2012
4,5-Dihydroxy-3,7-dimethoxy-9,10-dihydrophenanthrene [**79**]	*D. nobile*	Stem	Ye and Zhao, 2002
4,5-Dihydroxy-2-methoxy-9,10-dihydrophenanthrene (Orchinol) [**80**]	*D. nobile*	Stem	Zhang *et al.*, 2007a
	D. officinale	Stem	Zhao *et al.*, 2018
Lusianthridin [**81**]	*D. brymerianum*	Whole plant	Klongkumnuankarn *et al.*, 2015
	D. formosum	Whole plant	Inthongkaew *et al.*, 2017
	D. palpebrae	Whole plant	Kyokong *et al.*, 2018
	D. plicatile	Stem	Yamaki and Honda, 1996
	D. venustum	Whole plant	Sukphan *et al.*, 2014
7-Hydroxy-2,3,4-trimethoxy-9,10-dihydro-phenanthrene [**82**]	*D. hainanense*	Aerial part	Zhang *et al.*, 2018

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Table 1 (continued)

Compounds	Plant	Plant part	Reference
2,7-Dihydroxy-3,4,6-trimethoxy-9,10-dihydrophenanthrene [**83**]	*D. densiflorum*	Stem	Fan *et al.*, 2001
2,8-Dihydroxy-3,4,7-trimethoxy-9,10-dihydrophenanthrene [**84**]	*D. nobile*	Stem	Yang *et al.*, 2007
4,7-Dihydroxy-2,3,6-trimethoxy-9,10-dihydrophenanthrene [**85**]	*D. rotundatum*	Whole plant	Majumder and Pal, 1992b
Dendroinfundin B [**86**]	*D. infundibulum*	Whole plant	Na Ranong *et al.*, 2018
Ephemeranthol A [**87**]	*D. infundibulum*	Whole plant	Na Ranong *et al.*, 2018
	D. nobile	Stem	Yang *et al.*, 2007; Hwang *et al.*, 2010
	D. officinale	Stem	Zhao *et al.*, 2018
Ephemeranthol C [**88**]	*D. nobile*	Stem	Yang *et al.*, 2007; Hwang *et al.*, 2010
Erianthridin [**89**]	*D. nobile*	Stem	Hwang *et al.*, 2010
	D. formosum	Whole plant	Inthongkaew *et al.*, 2017
	D. plicatile	Stem	Yamaki and Honda, 1996
Flavanthridin [**90**]	*D. nobile*	Stem	Hwang *et al.*, 2010

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Table 1 (continued)

Compounds	Plant	Plant part	Reference
Hircinol [**91**]	*D. aphyllum*	Stem	Yang *et al.*, 2015a
	D. draconis	Stem	Sritularak *et al.*, 2011a
	D. formosum	Whole plant	Inthongkaew *et al.*, 2017
3-Hydroxy-2,4,7-trimethoxy-9,10-dihydrophenanthrene [**92**]	*D. nobile*	Stem	Yang *et al.*, 2007
3,4-Dimethoxy-1-(methoxymethyl)-9,10-dihydrophenanthrene-2,7-diol [**93**]	*D. hainanense*	Aerial part	Zhang *et al.*, 2018
2-Hydroxy-4,7-dimethoxy-9,10-dihydrophenanthrene [**94**]	*D. nobile*	Stem	Yang *et al.*, 2007
7-Methoxy-9,10-dihydrophenanthrene-2,4,5-triol [**95**]	*D. draconis*	Stem	Sritularak *et al.*, 2011a
2,5,7-Trimethoxy-4-methoxy-9,10-dihydrophenanthrene [**96**]	*D. formosum*	Whole plant	Inthongkaew *et al.*, 2017
	D. longicornu	Stem	Hu *et al.*, 2008b
Plicatol C [**97**]	*D. plicatile*	Stem	Honda and Yamaki, 2000
Rotundatin [**98**]	*D. rotundatum*	Whole plant	Majumder and Pal, 1992a

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Table 1 (continued)

Compounds	Plant	Plant part	Reference
2,5-Dihydroxy-3,4 dimethoxyphenanthrene [**99**]	*D. nobile*	Stem	Yang *et al.*, 2007
2,5-Dihydroxy-4,9-dimethoxyphenanthrene [**100**]	*D. nobile*	Stem	Zhang *et al.*, 2008b
	D. palpebrae	Whole plant	Kyokong *et al.*, 2018
2,8-Dihydroxy-3,4,7-trimethoxyphenanthrene [**101**]	*D. nobile*	Stem	Yang *et al.*, 2007
Epheranthol B [**102**]	*D. chrysotoxum*	Stem	Hu *et al.*, 2012
	D. plicatile	Stem	Yamaki and Honda, 1996
Fimbriol B [**103**]	*D. nobile*	Stem	Yang *et al.*, 2007; Hwang *et al.*, 2010
Flavanthrinin [**104**]	*D. brymerianum*	Whole plant	Klongkumnuankarn *et al.*, 2015
	D. nobile	Stem	Zhang *et al.*, 2008b
	D. parishii	Whole plant	Kongkatitham *et al.*, 2018
	D. venustum	Whole plant	Sukphan *et al.*, 2014
Loddigesiinol A [**105**]	*D. loddigesii*	Whole plant	Ito *et al.*, 2010b
	D. wardianum	Stem	Zhang *et al.*, 2017

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Table 1 (continued)

Compounds	Plant	Plant part	Reference
Nudol [**106**]	*D. formosum*	Whole plant	Inthongkaew *et al.*, 2017
	D. nobile	Stem	Yang *et al.*, 2007
	D. rotundatum	Whole plant	Majumder and Pal, 1992b
Plicatol A [**107**]	*D. nobile*	Stem	Yang *et al.*, 2007
	D. plicatile	Stem	Honda and Yamaki, 2000
Plicatol B [**108**]	*D. plicatile*	Stem	Honda and Yamaki, 2000
2,3,5-Trihydroxy-4,9-dimethoxyphenanthrene [**109**]	*D. nobile*	Stem	Yang *et al.*, 2007
3,4,8-Trimethoxyphenan-threne-2,5-diol [**110**]	*D. nobile*	Stem	Hwang *et al.*, 2010
Bulbophyllanthrin [**111**]	*D. nobile*	Stem	Yang *et al.*, 2007
Denthyrsinin [**112**]	*D. thyrsiforum*	Stem	Zhang *et al.*, 2005
5-Hydroxy-2,4-dimethoxyphenanthrene [**113**]	*D. loddigesii*	Whole plant	Ito *et al.*, 2010b
3-Hydroxy-2,4,7-trimethoxyphenanthrene [**114**]	*D. nobile*	Stem	Yang *et al.*, 2007

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Table 1 (continued)

Compounds	Plant	Plant part	Reference
Cypripedin [**115**]	*D. densiflorum*	Stem	Fan *et al.*, 2001
Densiflorol B [**116**]	*D. densiflorum*	Stem	Fan *et al.*, 2001
	D. venustum	Whole plant	Sukphan *et al.*, 2014
Denbinobin [**117**]	*D. moniliforme*	Stem	Lin *et al.*, 2001
	D. nobile	Stem	Yang *et al.*, 2007
	D. wardianum	Stem	Zhang *et al.*, 2017
Dendronone [**118**]	*D. chrysanthum*	Stem	Yang *et al.*, 2006a
	D. longicornu	Stem	Hu *et al.*, 2008b
Ephemeranthoquinone [**119**]	*D. plicatile*	Stem	Yamaki and Honda, 1996
5-Methoxy-7-hydroxy-9,10-dihydro-1,4-phenanthrenequinone [**120**]	*D. draconis*	Stem	Sritularak *et al.*, 2011a
	D. formosum	Whole plant	Inthongkaew *et al.*, 2017
Moniliformin [**121**]	*D. moniliforme*	Stem	Lin *et al.*, 2001
Moscatin [**122**]	*D. aphyllum*	Whole plant	Chen *et al.*, 2008e
	D. chrysanthum	Stem	Yang *et al.*, 2006a
	D. chrysotoxum	Whole plant	Li *et al.*, 2009c
	D. densiflorum	Stem	Fan *et al.*, 2001
	D. polyanthum	Stem	Hu *et al.*, 2009
Fimbriatone [**123**]	*D. nobile*	Stem	Zhang *et al.*, 2008b
	D. pulchellum	Stem	Chanvorachote *et al.*, 2013
Loddigesiinol B [**124**]	*D. loddigesii*	Whole plant	Ito *et al.*, 2010b

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Table 1 (continued)

Compounds	**Plant**	**Plant part**	**Reference**
Amoenumin [**125**]	*D. amoenum*	Whole plant	Veerraju *et al.*, 1989
Crystalltone [**126**]	*D. chrysotoxum*	Stem	Hu *et al.*, 2012
	D. crystallinum	Stem	Wang *et al.*, 2009
Chrysotoxol A [**127**]	*D. chrysotoxum*	Stem	Hu *et al.*, 2012
Chrysotoxol B [**128**]	*D. chrysotoxum*	Stem	Hu *et al.*, 2012
Confusarin [**129**]	*D. chrysotoxum*	Stem	Hu *et al.*, 2012
	D. formosum	Whole plant	Inthongkaew *et al.*, 2017
	D. nobile	Stem	Zhang *et al.*, 2008b
	D. officinale	Stem	Zhao *et al.*, 2018
2,6-Dihydroxy-1,5,7-trimethoxyphenanthrene [**130**]	*D. densiflorum*	Stem	Fan *et al.*, 2001
	D. palpebrae	Whole plant	Kyokong *et al.*, 2018
Dendrochrysanene [**131**]	*D. chrysanthum*	Stem	Yang *et al.*, 2006a
2,2′-Dihydroxy-3,3′,4,4′,7,7-hexamethoxy-9,9′,10,10′-tetrahydro-1,1′-biphenanthrene [**132**]	*D. nobile*	Stem	Yang *et al.*, 2007
2,2′-Dimethoxy-4,4′,7,7′-tetrahydroxy-9,9′,10,10′-tetrahydro-1,1′-biphenanthrene [**133**]	*D. plicatile*	Stem	Yamaki and Honda, 1996

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Table 1 (continued)

Compounds	Plant	Plant part	Reference
Flavanthrin [**134**]	*D. aphyllum*	Whole plant	Chen *et al.*, 2008c
Phoyunnanin C [**135**]	*D. venustum*	Whole plant	Sukphan *et al.*, 2014
Phoyunnanin E [**136**]	*D. venustum*	Whole plant	Sukphan *et al.*, 2014
Aphyllone [**137**]	*D. nobile*	Stem	Hwang *et al.*, 2010
9,10-Dihydro-aphyllone A-5-*O*-β-D-glucopyranoside [**138**]	*D. fimbriatum*	Stem	Xu *et al.*, 2017
(*S*)-2,4,5,9-Tetrahydroxy-9,10-dihydro-phenanthrene [**139**]	*D. fimbriatum*	Stem	Xu *et al.*, 2014
1,5,7-Trimethoxy-phenanthrene-2-ol [**140**]	*D. nobile*	Stem	Kim *et al.*, 2015
1,5-Dihydroxy-3,4,7-trimethoxy-9,10-dihydro-phenanthrene [**141**]	*D. moniliforme*	Whole plant	Zhao *et al.*, 2016
2,4,5,9*S*-Tetrahydroxy-9,10-dihydrophenanthrene-4-*O*-β-D-glucopyranoside [**142**]	*D. primulinum*	Whole plant	Ye *et al.*, 2016
Loddigesiinol G [**143**]	*D. loddigesii*	Stem	Lu *et al.*, 2014b
Loddigesiinol H [**144**]	*D. loddigesii*	Stem	Lu *et al.*, 2014b
Loddigesiinol I [**145**]	*D. loddigesii*	Stem	Lu *et al.*, 2014b
Loddigesiinol J [**146**]	*D. loddigesii*	Stem	Lu *et al.*, 2014b

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Table 1 (continued)

Compounds	**Plant**	**Plant part**	**Reference**
Dendropalpebrone [**147**]	*D. palpebrae*	Whole plant	Kyokong *et al.*, 2018
Dendrocandin P1 [**148**]	*D. officinale*	Stem	Zhao *et al.*, 2018
Dendrocandin P2 [**149**]	*D. officinale*	Stem	Zhao *et al.*, 2018

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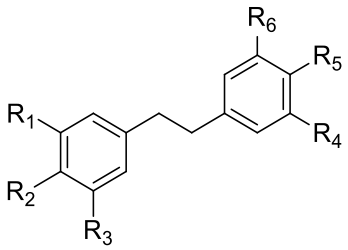

	R_1	R_2	R_3	R_4	R_5	R_6
[**1**] Aloifol I	OMe	OH	OMe	OH	H	H
[**2**] Amoenylin	OMe	OH	OMe	H	OMe	H
[**3**] 4,3′,4′-Trihydroxy-3,5-dimethoxybibenzyl	OMe	OH	OMe	H	OH	OH
[**4**] Batatasin	OMe	H	H	OH	H	OH
[**5**] Batatasin III	OH	H	OMe	H	H	OH
[**6**] Brittonin A	OMe	OMe	OMe	OMe	OMe	OMe
[**7**] Chrysotobibenzyl	OMe	OMe	OMe	OMe	OMe	H
[**8**] Chrysotoxine	OMe	OH	OMe	OMe	OMe	H
[**9**] Crepidatin	OMe	OMe	OMe	OMe	OH	H
[**10**] Cumulatin	OMe	OMe	OH	OH	OMe	OMe
[**11**] Dendrobin A	OH	OH	OMe	H	H	OMe
[**12**] 3,3′-Dihydroxy-4,5-dimethoxybibenzyl	OMe	OMe	OH	H	H	OH
[**13**] 3,4′-Dihydroxy-5-methoxybibenzyl	OH	H	OMe	H	OH	H
[**14**] 3,4′-Dihydroxy-5,5′-Dimethoxydihydrostilbene	OH	H	OMe	OMe	OH	H

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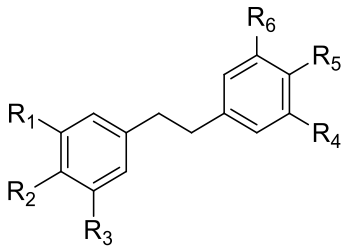
Figure 2 Structures of bibenzyls and derivatives previously isolated from *Dendrobium* plants



	R_1	R_2	R_3	R_4	R_5	R_6
[**15**] Erianin	OMe	OMe	OMe	H	OMe	OH
[**16**] Gigantol	OMe	H	H	H	OH	OMe
[**17**] Gigantol-5-*O*-β-D-glucopyranoside	OMe	H	OGlc	H	OH	OMe
[**18**] 4-Hydroxy-3,5,3′ trimethoxybibenzyl	OMe	OH	OMe	H	H	OMe
[**19**] 5-Hydroxy-3,4,3′,4′,5′ pentamethoxybibenzyl	OMe	OMe	OH	OMe	OMe	OMe
[**20**] 3,4′-Dihydroxy-3′,4,5-trimethoxybibenzyl	OMe	OMe	OH	H	OH	OMe
[**21**] Isoamoenylin	OMe	OMe	OMe	H	H	OH
[**22**] Moscatilin	OMe	OH	OMe	H	OH	OMe
[**23**] Moscatilin diacetate	OMe	OAc	OMe	H	OAc	OMe
[**24**] 3,3′,4-Trihydroxybibenzyl	OH	OH	H	H	H	OH
[**25**] 3,3′,5-Trihydroxybibenzyl	OH	H	OH	H	H	OH
[**26**] 3,5,4′-Trihydroxybibenzyl	OH	H	OH	H	OH	H
[**27**] 4,5,4′-Trihydroxy-3,3′-dimethoxybibenzyl	OMe	OH	OH	H	OH	OMe
[**28**] Tristin	OH	H	OH	H	OH	OMe
[**29**] Dendromoniliside E	OGlc	OGlc	OMe	H	OMe	H

Figure 2 (continued)

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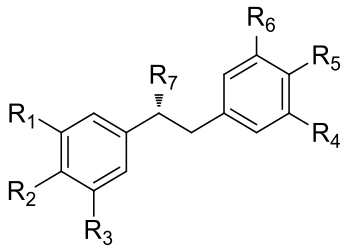
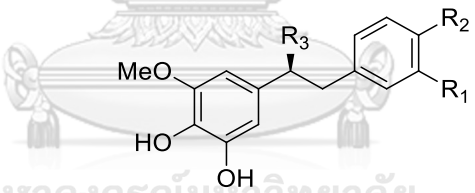


	R_1	R_2	R_3	R_4	R_5	R_6	R_7
[**30**] Dendrophenol	OMe	OH	OMe	OH	H	OH	H
[**31**] 3,4-Dihydroxy-5,4′-dimethoxybibenzyl	OH	OH	OMe	H	OMe	H	H
[**32**] 4,4′-Dihydroxy-3,5-dimethoxybibenzyl	OMe	OH	OMe	H	OH	H	H
[**33**] Loddigesiinol C	OMe	OH	OMe	H	OH	OMe	OMe
[**34**] 3-*O*-Methylgigantol	OMe	H	OH	OMe	OMe	H	H



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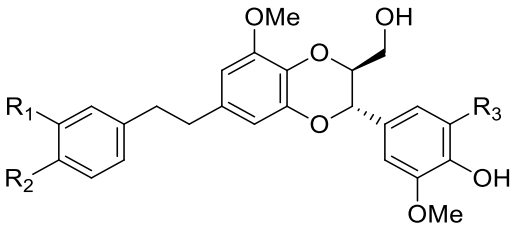
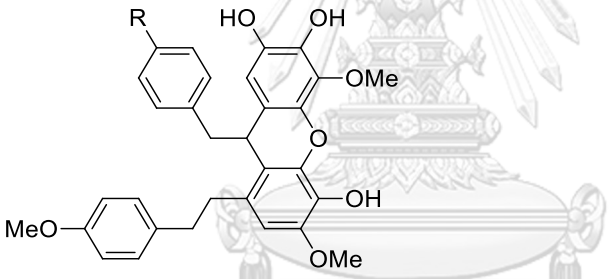
	R_1	R_2	R_3
[**35**] Dendrocandin A	H	OMe	OMe
[**36**] Dendrocandin C	H	OH	OMe
[**37**] Dendrocandin D	H	OH	OEt
[**38**] Dendrocandin E	OH	OH	H

Figure 2 (continued)

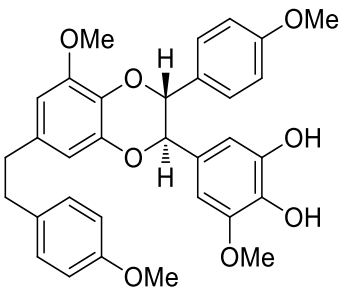


	R_1	R_2	R_3
[**39**] Dendrocandin B	H	OMe	OMe
[**40**] Dendrocandin T	OMe	OH	OMe
[**41**] Dendrocandin U	H	OH	OMe
[**42**] Dendrocandin V	H	OMe	H



[**43**] Dendrocandin F: R = OMe

[**44**] Dendrocandin G: R= OH



[**46**] Dendrocandin I

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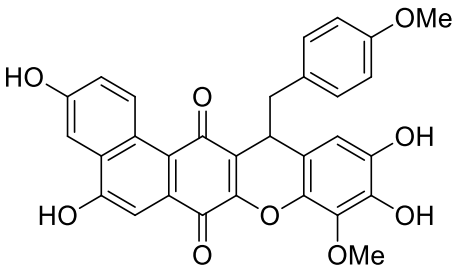
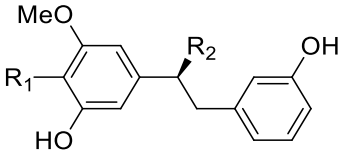


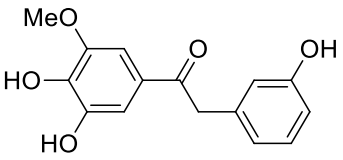
[**45**] Dendrocandin H



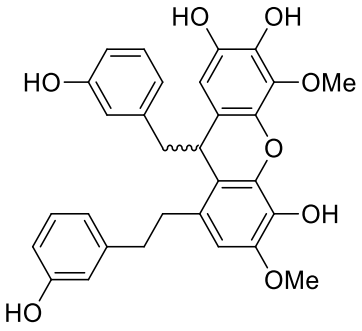
[**47**] Dendrosinen A: R_1 = OMe, R_2 = OH

[**48**] Dendrosinen B: R_1 = OH, R_2 = H

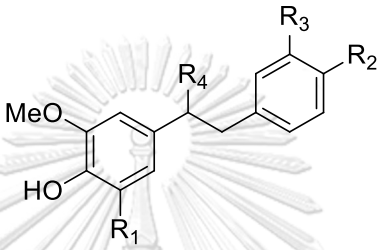
Figure 2 (continued)



[**49**] Dendrosinen C



[**50**] Dendrosinen D



	R_1	R_2	R_3	R_4
[**51**] 4-[2-(3-Hydroxyphenol)-1-methoxyethyl]-2,6-dimethoxyphenol	OMe	H	OH	OMe
[**52**] Nobilin A	OH	H	OMe	OMe
[**53**] Nobilin B	OMe	OH	OMe	OMe
[**54**] Nobilin C	OMe	OMe	OMe	OMe
[**55**] Nobilin D	OMe	OH	OMe	OH

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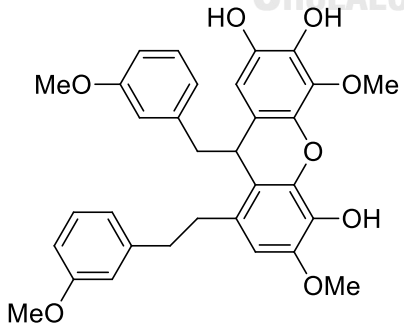
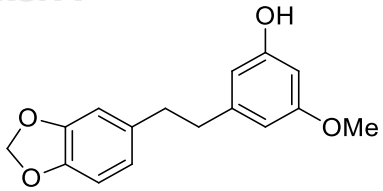
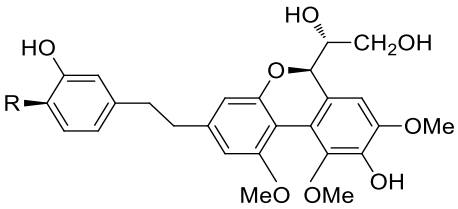


[**56**] Nobilin E



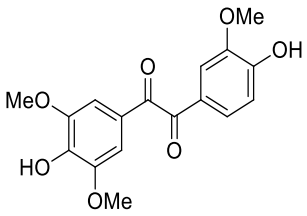
[**57**] Densiflorol A

Figure 2 (continued)

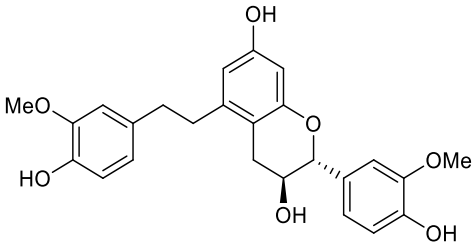


[**58**] Longicornuol A: R = H

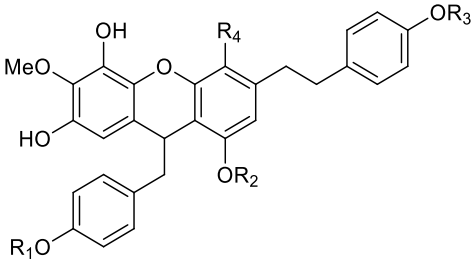
[**59**] Trigonopol A: R = OMe



[**63**] Loddigesiinol D

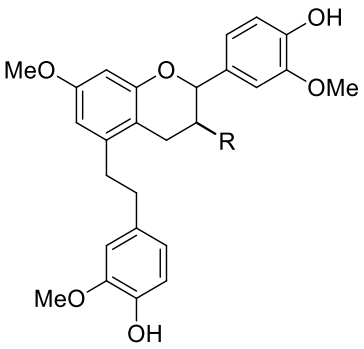


[**60**] Trigonopol B



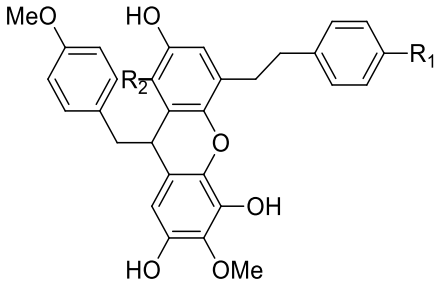
[**64**] Dencryol A: R_1 = OMe, R_2 =R_3 = OH, R_4 = H

[**65**] Dencryol B: R_1 = OH, R_2 = R_3 = OMe, R_4 = OH



[**61**] Crepidatuol A: R = H

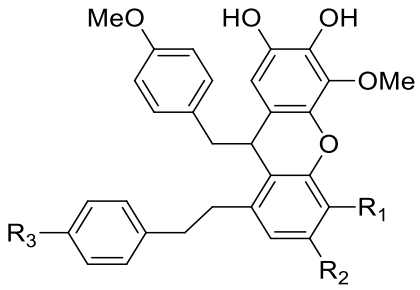
[**62**] Crepidatuol B: R = OH



[**66**] Dengraol A: R_1 = OH, R_2 = H

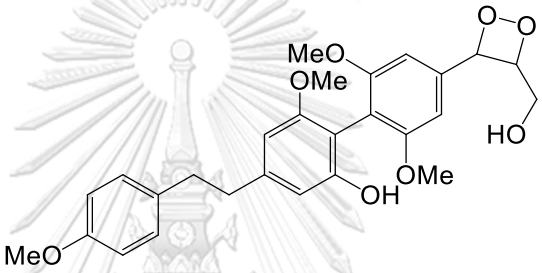
[**67**] Dengraol B: R_1 = R_2 = OMe

Figure 2 (continued)

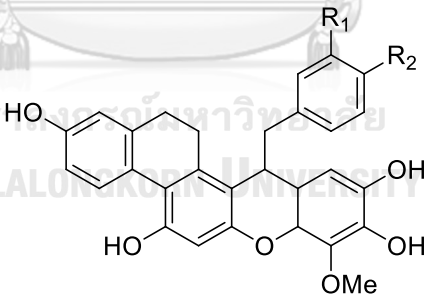


[**68**] Dendrofalconerol A: R_1 = OH, R_2 = R_3 = OMe

[**69**] Dendrofalconerol B: R_1 = H, R_2 = R_3 = OH



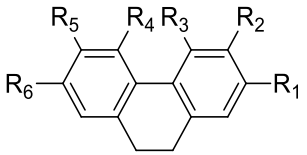
[**70**] Dendrowillol A



[**71**] Dendrosignatol: R_1 = H, R_2 = OMe

[**72**] Dendroparishiol: R_1 = OMe, R_2 = OH

Figure 2 (continued)



	R_1	R_2	R_3	R_4	R_5	R_6
[**73**] Coelonin	OH	H	OMe	H	H	OH
[**74**] 9,10-Dihydromoscatin	H	H	OH	OMe	H	OH
[**75**] 9,10-Dihydrophenanthrene-2,4,7-triol	OH	H	OH	H	H	OH
[**76**] 4,5-Dihydroxy-2,3-dimethoxy-9,10-dihydrophenanthrene	OMe	OMe	OH	OH	H	H
[**77**] Dendroinfundin A	OMe	OMe	OH	H	H	OMe
[**78**] 4,5-Dihydroxy-2,6-dimethoxy-9,10-dihydrophenanthrene	OMe	H	OH	OH	OMe	H
[**79**] 4,5-Dihydroxy-3,7-dimethoxy-9,10-dihydrophenanthrene	H	OMe	OH	OH	H	OMe
[**80**] 4,5-Dihydroxy-2-methoxy-9,10-dihydrophenanthrene (Orchinol)	OMe	H	OH	OH	H	H
[**81**] Lusianthridin	OMe	H	OH	H	H	OH
[**82**] 7-Hydroxy-2,3,4-trimethoxy-9,10-dihydro-phenanthrene	OMe	OMe	OMe	H	H	OH

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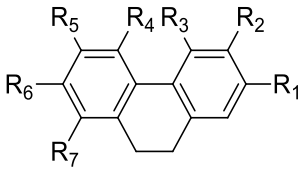
Figure 2 (continued)



	R_1	R_2	R_3	R_4	R_5	R_6	R_7
[**83**] 2,7-Dihydroxy-3,4,6-trimethoxy-9,10-dihydro phenanthrene	OH	OMe	OMe	H	OMe	OH	H
[**84**] 2,8-Dihydroxy-3,4,7-trimethoxy-9,10-dihydro phenanthrene	OH	OMe	OMe	H	H	OMe	OH
[**85**] 4,7-Dihydroxy-2,3,6-trimethoxy-9,10-dihydro phenanthrene	OMe	OMe	OH	H	OMe	OH	H
[**86**] Dendroinfundin B	OMe	OMe	OH	OH	H	H	OMe
[**87**] Ephemeranthol A	OH	H	H	OH	OMe	OMe	H
[**88**] Ephemeranthol C	OH	OH	OMe	OH	H	H	H
[**89**] Erianthridin	OH	OMe	OMe	H	H	OH	H
[**90**] Flavanthridin	OH	H	H	OMe	OH	OMe	H
[**91**] Hircinol	OH	H	OMe	OH	H	H	H
[**92**] 3-Hydroxy-2,4,7- trimethoxy-9,10-dihydrophenanthrene	OMe	OH	OMe	H	H	OMe	H
[**93**] 3,4-Dimethoxy-1-(methoxy methyl)-9,10-dihydro phenanthrene-2,7-diol	OH	H	H	OMe	OMe	OH	CH_2OMe

Figure 2 (continued)

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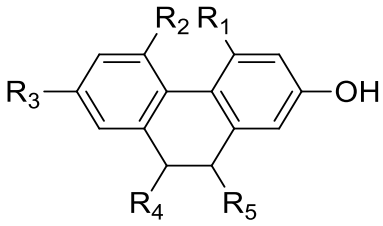

	R_1	R_2	R_3	R_4	R_5
[**94**] 2-Hydroxy-4,7-dimethoxy-9,10-dihydro phenanthrene	OMe	H	OMe	H	H
[**95**] 7-Methoxy-9,10-dihydrophenanthrene-2,4,5-triol	OH	OH	OMe	H	H
[**96**] 2,5,7-Trihydroxy-4-methoxy-9,10-dihydrophenanthrene	OMe	OH	OH	H	H
[97] Plicatol C	OMe	OH	H	OMe	OMe
[**98**] Rotundatin	OMe	OH	H	OH	OH

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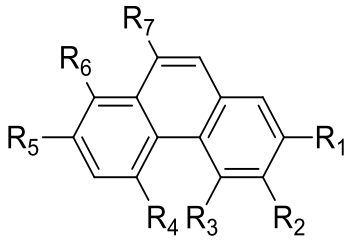
Figure 2 (continued)



	R_1	R_2	R_3	R_4	R_5	R_6	R_7
[**99**] 2,5-Dihydroxy-3,4-dimethoxyphenanthrene	OH	OMe	OMe	OH	H	H	H
[**100**] 2,5-Dihydroxy-4,9-dimethoxyphenanthrene	OH	H	OMe	OH	H	H	OMe
[**101**] 2,8-Dihydroxy-3,4,7-trimethoxyphenanthrene	OH	OMe	OMe	H	OMe	OH	H
[**102**] Epheranthol B	H	H	OMe	OH	OMe	H	H
[**103**] Fimbriol B	OH	OMe	OH	H	H	H	H
[**104**] Flavanthrinin	H	H	OMe	H	OH	H	H

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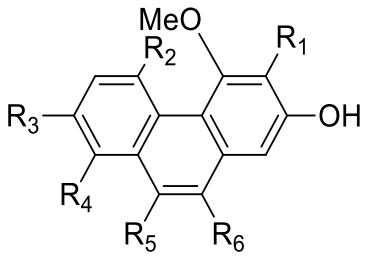
Figure 2 (continued)



	R_1	R_2	R_3	R_4	R_5	R_6
[**105**] Loddigesiinol A	H	OMe	H	H	OH	H
[**106**] Nudol	OMe	H	OH	H	H	H
[**107**] Plicatol A	H	OH	H	H	OMe	OMe
[**108**] Plicatol B	H	OH	H	H	H	H
[**109**] 2,3,5-Trihydroxy-4,9-dimethoxyphenanthrene	OH	OH	H	H	OMe	H
[**110**] 3,4,8-Trimethoxy phenanthrene-2,5-diol	OMe	OH	H	OMe	H	H

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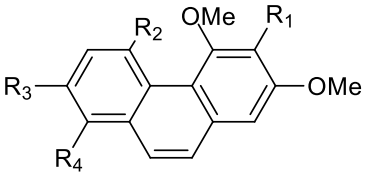
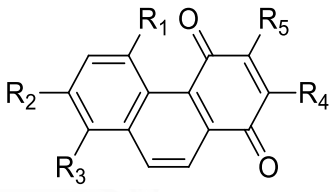
Figure 2 (continued)



	R_1	R_2	R_3	R_4
[**111**] Bulbophyllanthrin	OH	OH	H	H
[**112**] Denthyrsinin	OH	H	OH	OMe
[**113**] 5-Hydroxy-2,4-dimethoxy phenanthrene	H	OH	H	H
[**114**] 3-Hydroxy-2,4,7-trimethoxy phenanthrene	OH	H	OMe	H



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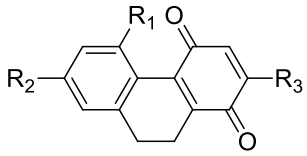
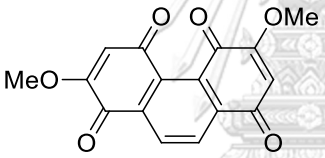
	R_1	R_2	R_3	R_4	R_5
[**115**] Cypripedin	H	OH	OMe	OMe	H
[**116**] Densiflorol B	H	OH	H	OMe	H
[**117**] Denbinobin	OH	OMe	H	H	OMe

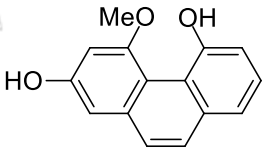
Figure 2 (continued)



	R_1	R_2	R_3
[**118**] Dendronone	OH	OMe	H
[**119**] Ephemeranthoquinone	H	OH	OMe
[**120**] 5-Methoxy-7-hydroxy-9,10-dihydro-1,4-phenanthrenequinone	OMe	OH	H



[**121**] Moniliformin



[**122**] Moscatin

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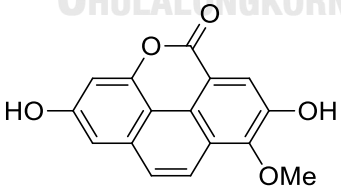
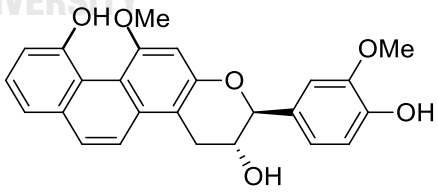
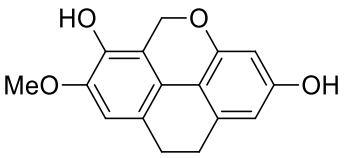


[**123**] Fimbriatone

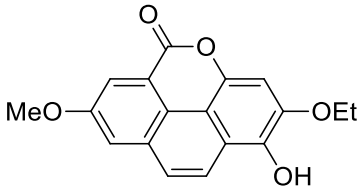


[**124**] Loddigesiinol B

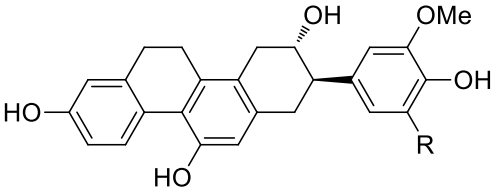
Figure 2 (continued)



[**125**] Amoenumin

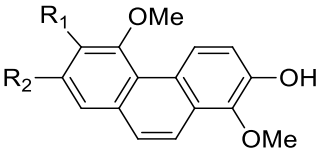


[**126**] Crystalltone



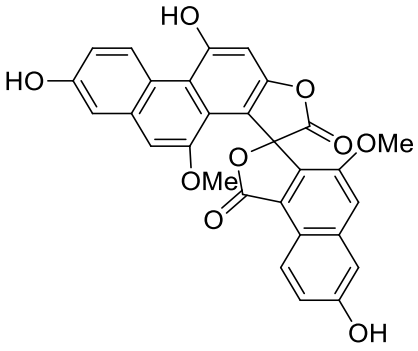
[**127**] Chrysotoxol A: R = H

[**128**] Chrysotoxol B: R = OMe



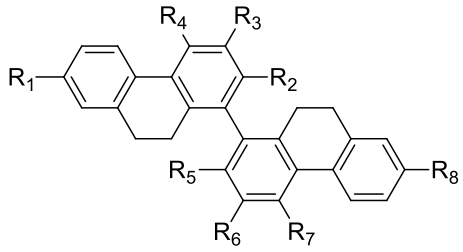
[**129**] Confusarin: R_1 = OMe, R_2 = OH

[**130**] 2,6-Dihydroxy-1,5,7-trimethoxy phenanthrene: R_1 = OH, R_2 = OMe



[**131**] Dendrochrysanene

Figure 2 (continued)



	R_1	R_2	R_3	R_4	R_5	R_6	R_7	R_8
[**132**] 2,2′-Dihydroxy-3,3′,4,4′, 7,7′-hexamethoxy-9,9′, 10,10′-tetrahydro-1,1′-biphenanthrene	OMe	OH	OMe	OMe	OH	OMe	OMe	OMe
[**133**] 2,2′-Dimethoxy-4,4′,7,7′-tetrahydroxy-9,9′,10, 10′-tetrahydro-1,1′-biphenanthrene	OH	OMe	H	OH	OMe	H	OH	OH
[**134**] Flavanthrin	OH	OH	H	OMe	OH	H	OMe	OH

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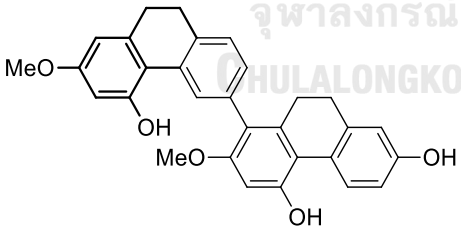
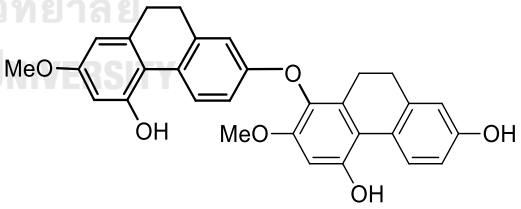
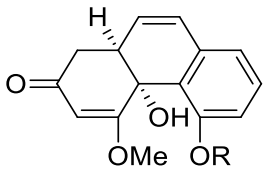


[**135**] Phoyunnanin C



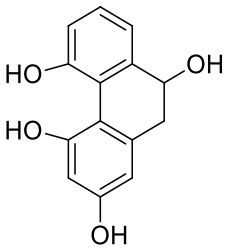
[**136**] Phoyunnanin E

Figure 2 (continued)

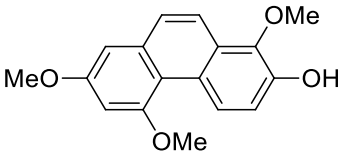


[**137**] Aphyllone: R = H

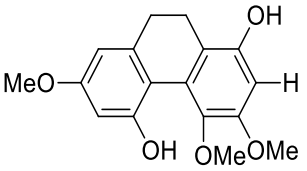
[**138**] 9,10-Dihydro-aphyllone A-5-*O*-β-D-glucopyranoside: R = Glc



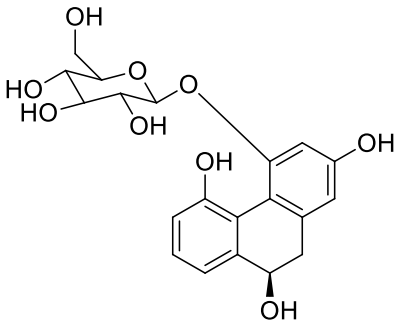
[**139**] (*S*)-2,4,5,9-Tetrahydroxy-9,10-dihydrophenanthrene



[**140**] 1,5,7-Trimethoxyphenanthren-2-ol

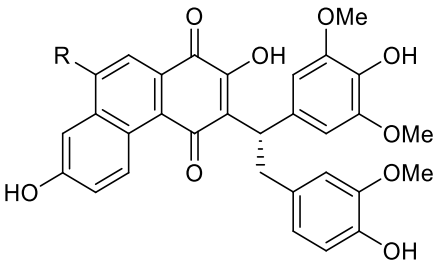


[**141**] 1,5-Dihydroxy-3,4,7-trimethoxy-9,10-dihydrophenanthrene



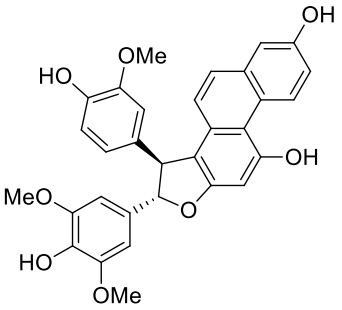
[**142**] 2,4,5,9*S*-Tetrahydroxy-9,10-dihydrophenanthrene 4-*O*-β-D-glucopyranoside

Figure 2 (continued)

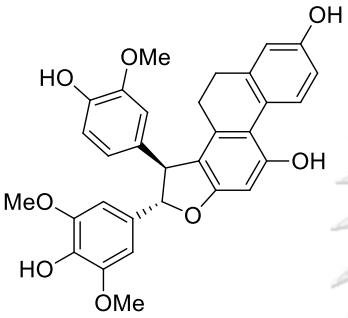


[**143**] Loddigesiinol G: R = H

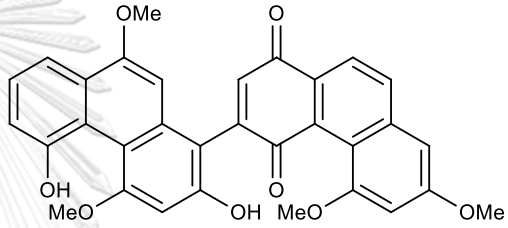
[**144**] Loddigesiinol H: R = OH



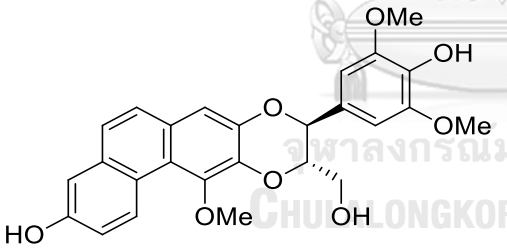
[**145**] Loddigesiinol I



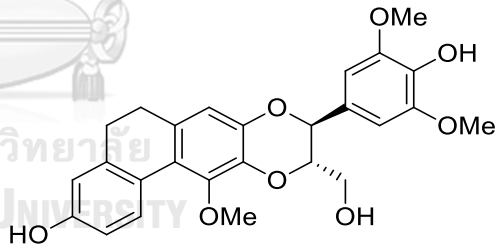
[**146**] Loddigesiinol J



[**147**] Dendropalpebrone



[**148**] Dendrocandin P1



[**149**] Dendrocandin P2

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Figure 2 (continued)

Table 2 Distribution of flavonoids in *Dendrobium* plants

Compounds	Plant	Plant part	Reference
Apigenin [**150**]	*D. crystallinum*	Stem	Wang *et al.*, 2009
	D. williamsonii	Whole plant	Rungwichaniwat *et al.*, 2014
5,6-Dihydroxy-4′-methoxyflavone [**151**]	*D. chrysotoxum*	Stem	Hu *et al.*, 2012
Luteolin [**152**]	*D. aurantiacum* var. *denneanum*	Whole plant	Liu *et al.*, 2009b
	D. ellipsophyllum	Whole plant	Tanagornmeatar *et al.*, 2014
	D. longicornu	Stem	Hu *et al.*, 2008b
Chrysoeriol [**153**]	*D. ellipsophyllum*	Whole plant	Tanagornmeatar *et al.*, 2014
6-*C*-(α-Arabinopyranosyl)-8-*C*-[(2-*O*-α-rhamnopyranosyl)-β-galactopyranosyl] apigenin [**154**]	*D. huoshanense*	Aerial part	Chang *et al.*, 2010
6-*C*-(α-Arabinopyranosyl)-8-*C*-[(2-*O*-α-rhamnopyranosyl)-β-glucopyranosyl] apigenin [**155**]	*D. huoshanense*	Aerial part	Chang *et al.*, 2010
Vicenin-2 [**156**]	*D. aurantiacum* var. *denneanum*	Stem	Xiong *et al.*, 2013

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Table 2 (continued)

Compounds	*Plant*	Plant part	Reference
(2*S*)-Homoeriodictyol [**157**]	*D. densiflorum*	Stem	Fan *et al.*, 2001
	D. ellipsophyllum	Whole plant	Tanagornmeatar *et al.*, 2014
Naringenin [**158**]	*D. aurantiacum* var. *denneanum*	Stem	Yang *et al.*, 2006b
	D. densiflorum	Stem	Fan *et al.*, 2001
	D. longicornu	Stem	Hu *et al.*, 2008b
(2*S*)-Eriodictyol [**159**]	*D. trigonopus*	Stem	Hu *et al.*, 2008a
	D. ellipsophyllum	Whole plant	Tanagornmeatar *et al.*, 2014
	D. tortile	Whole plant	Limpanit *et al.*, 2016
6‴-Glucosyl-vitexin [**160**]	*D. crystallinum*	Stem	Wang *et al.*, 2009
Isoschaftoside [**161**]	*D. huoshanense*	Aerial part	Chang *et al.*, 2010
Isoviolanthin [**162**]	*D. crystallinum*	Stem	Wang *et al.*, 2009
6-*C*-[(2-*O*-α-Rhamno-pyranosyl)-β-glucopyranosyl]-8-*C*-(α-arabinopyranosyl) apigenin [**163**]	*D. huoshanense*	Aerial part	Chang *et al.*, 2010

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Table 2 (continued)

Compounds	*Plant*	Plant part	Reference
6-*C*-(β-Xylopyranosyl)-8-*C*-[(2-*O*-α-rhamnopyranosyl)-β-glucopyranosyl] apigenin [**164**]	*D. huoshanense*	Aerial part	Chang *et al.*, 2010
Kaempferol [**165**]	*D. aurantiacum var. denneanum*	Stem	Yang *et al.*, 2006b
Kaempferol-3-*O*-α-L-rhamnopyranoside [**166**]	*D. secundum*	Stem	Phechrmeekha *et al.*, 2012
Kaempferol-3,7-*O*-di-α-L-rhamnopyranoside [**167**]	*D. secundum*	Stem	Phechrmeekha *et al.*, 2012
Kaempferol-3-*O*-α-L-rhamnopyranosyl-(1⟶2)- β-D-glucopyranoside [**168**]	*D. capillipes*	Stem	Phechrmeekha *et al.*, 2012
Kaempferol-3-*O*-α-L-rhamnopyranosyl-(1⟶2)-β-D-xylopyranoside [**169**]	*D. capillipes*	Stem	Phechrmeekha *et al.*, 2012
Quercetin-3-*O*-L-rhamnopyranoside [**170**]	*D. secundum*	Stem	Phechrmeekha *et al.*, 2012

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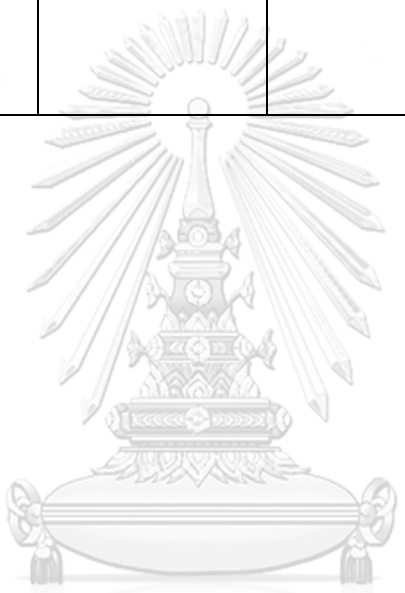
Table 2 (continued)

Compounds	*Plant*	Plant part	Reference
Quercetin-3-*O*-α-L-rhamnopyranosyl-(1′,2)-β-D-xylopyranoside [**171**]	*D. capillipes*	Stem	Phechrmeekha *et al.*, 2012
5-Hydroxy-3-methoxy-flavone-7-*O*-[β-D-apiosyl-(1⟶6)]-β-D-glucoside [**172**]	*D. devonianum*	Stem	Sun *et al.*, 2014



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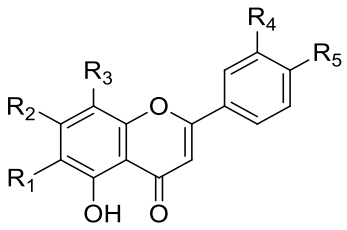

	R_1	R_2	R_3	R_4	R_5
[**150**] Apigenin	H	OH	H	H	OH
[**151**] 5,6-Dihydroxy-4′-methoxy-flavone	OH	H	H	H	OMe
[**152**] Luteolin	H	OH	H	OH	OH
[**153**] Chrysoeriol	H	OH	H	OMe	OH
[**154**] 6-*C*-(α-Arabinopyranosyl)-8-*C*-[(2-*O*-α-rhamnopyranosyl) -β-galactopyranosyl] apigenin	-Ara	OH	-Gal- Rha	H	OH
[**155**] 6-*C*-(α-Arabinopyranosyl)-8-*C*-[(2-*O*-α-rhamnopyranosyl) -β-glucopyranosyl] apigenin	-Ara	OH	-Glc- Rha	H	OH
[**156**] Vicenin-2	-Glc	OH	-Glc	H	OH

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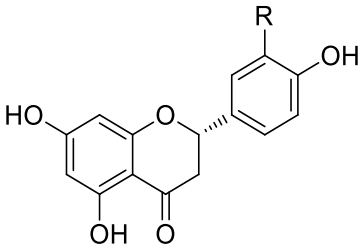
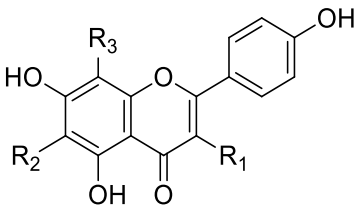


[**157**] (2*S*)-Homoeriodictyol: R = OMe

[**158**] Naringenin: R = H

[**159**] (2*S*)-Eriodictyol: R = OH

Figure 3 Structures of flavonoids previously isolated from *Dendrobium* plants



	R_1	R_2	R_3
[**160**] 6‴-Glucosyl-vitexin	H	H	-Glc-Glc
[**161**] Isoschaftoside	H	-Ara	-Glc
[**162**] Isoviolanthin	H	-Rha	-Glc
[**163**] 6-*C*-[(2-*O*-α-Rhamnopyranosyl)-β-glucopyranosyl]-8-*C*-(α-arabinopyranosyl) apigenin	H	-Glc-Rha	-Ara
[**164**] 6-*C*-(β-Xylopyranosyl)-8-*C*-[(2-*O*-α-rhamnopyranosyl)-β-glucopyranosyl] apigenin	H	-Xyl	-Glc-Rha
[**165**] Kaempferol	OH	H	H

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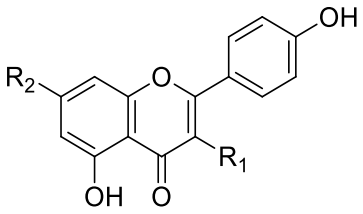
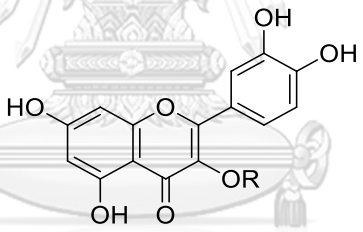
Figure 3 (continued)



	R_1	R_2
[**166**] Kaempferol-3-*O*-α-L-rhamnopyranoside	O-Rha	OH
[**167**] Kaempferol-3,7-*O*-di-α-L-rhamnopyranoside	O-Rha	O-Rha
[**168**] Kaempferol-3-*O*-α-L-rhamnopyranosyl (1⟶2)- β-D-glucopyranoside	O-Glc-Rha	OH
[**169**] Kaempferol-3-O-α-L-rhamnopyranosyl (1⟶2)-β-D-xylopyranoside	O-Xyl-Rha	OH



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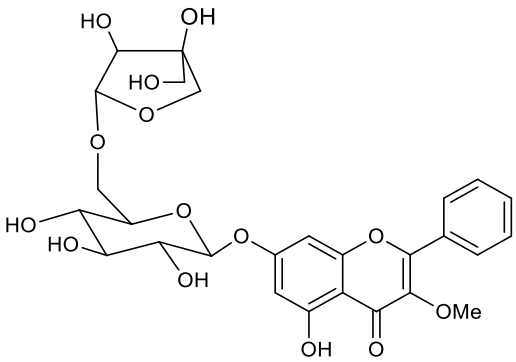
	R
[**170**] Quercetin-3-*O*-α-L-rhamnopyranoside	-Rha
[**171**] Quercetin-3-*O*-α-L-rhamnopyranosyl-(1⟶2) -β-D-xylopyranoside	-Xyl-Rha

Figure 3 (continued)



[**172**] 5-Hydroxy-3-methoxyflavone-7-*O*-[β-D-apiosyl-(1→6)]-β-D-glucoside

Figure 3 (continued)

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Table 3 Distribution of terpenoids in the *Dendrobium* plants

Compounds	Plant	Plant part	Reference
Dendronobiloside A [**173**]	*D. moniliforme*	Stem	Zhao *et al.*, 2003
	D. nobile	Stem	Zhao *et al.*, 2001; Ye and Zhao, 2002
Dendronobiloside B [**174**]	*D. nobile*	Stem	Zhao *et al.*, 2001; Ye and Zhao, 2002
Dendronobiloside C [**175**]	*D. nobile*	Stem	Zhao *et al.*, 2001; Ye and Zhao, 2002
Dendronobiloside D [**176**]	*D. nobile*	Stem	Zhao *et al.*, 2001; Ye and Zhao, 2002
Dendronobiloside E [**177**]	*D. nobile*	Stem	Zhao *et al.*, 2001; Ye and Zhao, 2002
Dendroside A [**178**]	*D. moniliforme*	Stem	Zhao *et al.*, 2003
	D. nobile	Stem	Zhao *et al.*, 2001; Ye and Zhao, 2002
Dendroside B [**179**]	*D. nobile*	Stem	Ye and Zhao, 2002; Zhao *et al.*, 2003
Dendroside C [**180**]	*D. moniliforme*	Stem	Zhao *et al.*, 2003
	D. nobile	Stem	Ye and Zhao, 2002
Dendroside D [**181**]	*D. nobile*	Stem	Ye and Zhao, 2002
Dendroside E [**182**]	*D. nobile*	Stem	Ye *et al.*, 2002
Dendroside F [**183**]	*D. moniliforme*	Stem	Zhao *et al.*, 2003
Dendroside G [**184**]	*D. nobile*	Stem	Ye *et al.*, 2002
Wardianumine A [**185**]	*D. wardianum*	Stem	Zhang *et al.*, 2017

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Table 3 (continued)

Compounds	**Plant**	**Plant part**	**Reference**
Aduncin [**186**]	*D. longicornu*	Stem	Hu *et al.*, 2008b
Amoenin [**187**]	*D. amoenum*	Whole plant	Dahmen and Leander, 1978
	D. williamsonii	Whole plant	Yang *et al.*, 2017a
Amotin [**188**]	*D. amoenum*	Whole plant	Majumder *et al.*, 1999
Dendrowillin A [**189**]	*D. williamsonii*	Whole plant	Yang *et al.*, 2017a
Dendrowillin B [**190**]	*D. williamsonii*	Whole plant	Yang *et al.*, 2017a
α-Dihydropicrotoxinin [**191**]	*D. amoenum*	Whole plant	Majumder *et al.*, 1999
	D. williamsonii	Whole plant	Yang *et al.*, 2017a
Picrotin [**192**]	*D. williamsonii*	Whole plant	Yang *et al.*, 2017a
Dendrobane A [**193**]	*D. moniliforme*	Stem	Bi *et al.*, 2004
Dendronobilin A [**194**]	*D. nobile*	Stem	Zhang *et al.*, 2007b
Dendronobilin B [**195**]	*D. wardianum*	Stem	Zhang *et al.*, 2007a
	D. nobile	Stem	Wang *et al.*, 2009; Meng *et al.*, 2017
Dendronobilin C [**196**]	*D. crystallium*	Stem	Wang *et al.*, 2009
Dendronobilin D [**197**]	*D. nobile*	Stem	Zhang *et al.*, 2007a
Dendronobilin E [**198**]	*D. nobile*	Stem	Zhang *et al.*, 2007a
Dendronobilin F [**199**]	*D. nobile*	Stem	Zhang *et al.*, 2007a
Dendronobilin G [**200**]	*D. nobile*	Stem	Zhang *et al.*, 2007a
Dendronobilin H [**201**]	*D. nobile*	Stem	Zhang *et al.*, 2007a

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Table 3 (continued)

Compounds	Plant	Plant part	Reference
Dendronobilin I [**202**]	*D. nobile*	Stem	Zhang *et al.*, 2007a
Dendronobilin J [**203**]	*D. nobile*	Stem	Zhang *et al.*, 2007a
Dendronobilin K [**204**]	*D. wardianum*	Stem	Fan *et al.*, 2013
Dendronobilin L [**205**]	*D. nobile*	Stem	Zhang *et al.*, 2007a
Dendronobilin M [**206**]	*D. nobile*	Stem	Zhang *et al.*, 2008a; Meng *et al.*, 2017
Dendronobilin N [**207**]	*D. nobile*	Stem	Zhang *et al.*, 2008a
Dendrowardol A [**208**]	*D. nobile*	Stem	Zhang *et al.*, 2008a
Dendrowardol B [**209**]	*D. nobile*	Stem	Zhang *et al.*, 2008a
Dendrowardol C [**210**]	*D. wardianum*	Stem	Fan *et al.*, 2013
Corchoionoside C [**211**]	*D. wardianum*	Stem	Fan *et al.*, 2013
Crystallinin [**212**]	*D. wardianum*	Stem	Fan *et al.*, 2013
Findlayanin [**213**]	*D. nobile*	Stem	Meng *et al.*, 2017
	D. polyanthum	Stem	Hu *et al.*, 2009
3-Hydroxy-2-oxodendrobine [**214**]	*D. findlayanum*	Whole plant	Qin *et al.*, 2011
Dendrobine [**215**]	*D. nobile*	Stem	Wang *et al.*, 1985 Meng *et al.*, 2017
(-)-(1*R*,2*S*,3*R*,4*S*,5*R*,6*S*,9*S*, 11*R*)-11-Carboxymethyl dendrobine [**216**]	*D. nobile*	Stem	Meng *et al.*, 2017
Dendromoniliside A [**217**]	*D. nobile*	Stem	Zhang *et al.*, 2007a

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Table 3 (continued)

Compounds	**Plant**	**Plant part**	**Reference**
Dendromoniliside B [**218**]	*D. moniliforme*	Stem	Zhao *et al.*, 2003
Dendromoniliside C [**219**]	*D. moniliforme*	Stem	Zhao *et al.*, 2003
Dendromoniliside D [**220**]	*D. moniliforme*	Stem	Zhao *et al.*, 2003
Asiatic acid [**221**]	*D. parishii*	Whole plant	Kongkatitham *et al.*, 2018

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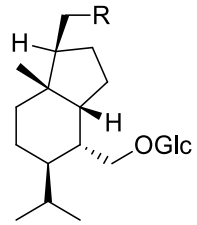
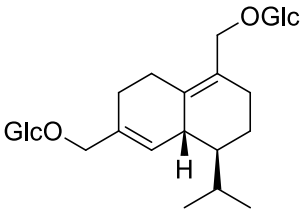
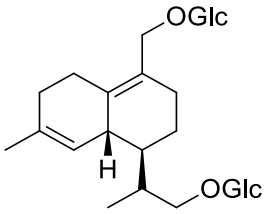


[**173**] Dendronobiloside A: R = OGlc

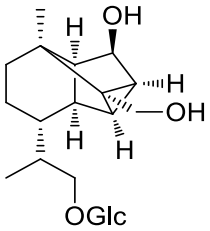
[**174**] Dendronobiloside B: R = OH



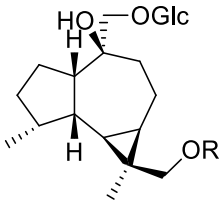
[**175**] Dendronobiloside C



[**176**] Dendronobiloside D



[**177**] Dendronobiloside E



[**178**] Dendroside A : R = H

[**179**] Dendroside B : R = Glc

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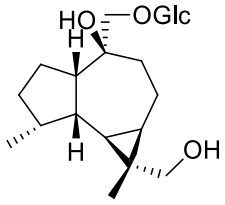
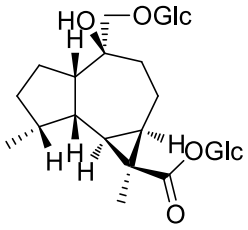
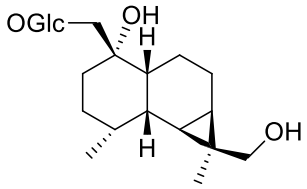


[**180**] Dendroside C: R = OH

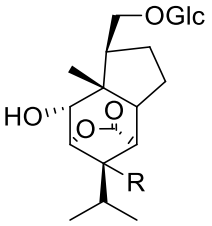
Figure 4 Structures of terpenoids previously isolated from *Dendrobium* plants



[**181**] Dendroside D

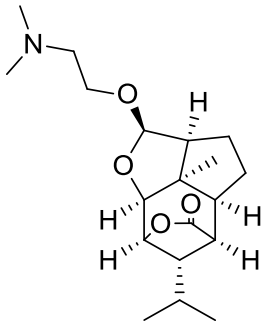


[**182**] Dendroside E



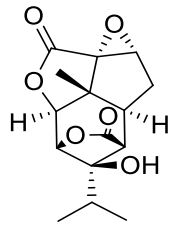
[**183**] Dendroside F: R = H

[**184**] Dendroside G: R = OH

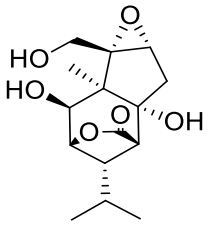


[**185**] Wardianumine A

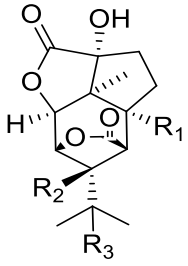
Figure 4 (continued)



[**186**] Aduncin



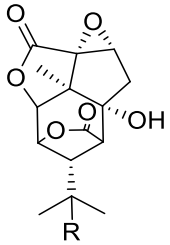
[**187**] Amoenin



[**188**] Amotin: R_1 = R_3 = H, R_2 = OH

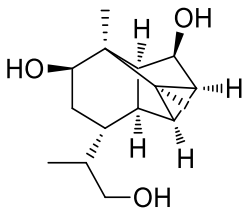
[**189**] Dendrowillin A: R_1 = R_3 = OH, R_2 = H

[**190**] Dendrowillin B: R_1 = R_2 = H, R_3 = OH

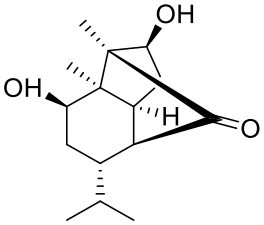


[**191**] α-Dihydropicrotoxinin: R = H

[**192**] Picrotin: R = OH

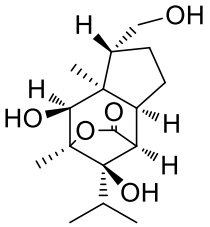


[**193**] Dendrobane A

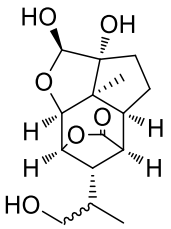


[**194**] Dendronobilin A

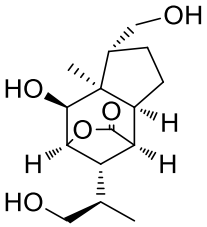
Figure 4 (continued)



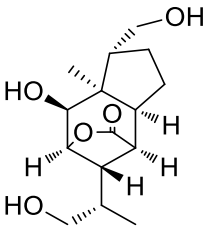
[**195**] Dendronobilin B



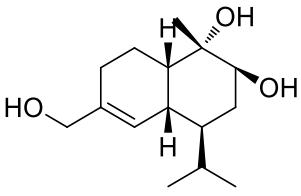
[**196**] Dendronobilin C



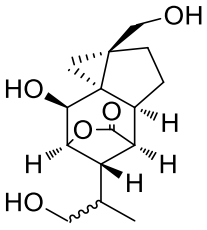
[**197**] Dendronobilin D



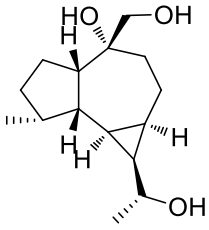
[**198**] Dendronobilin E



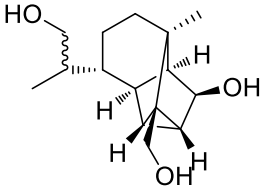
[**199**] Dendronobilin F



[**200**] Dendronobilin G

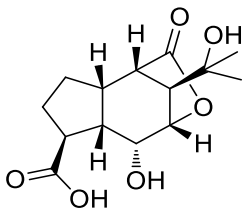


[**201**] Dendronobilin H

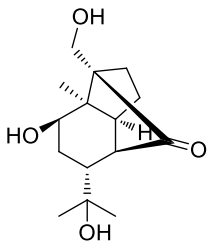


[**202**] Dendronobilin I

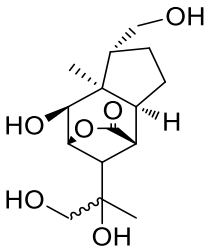
Figure 4 (continued)



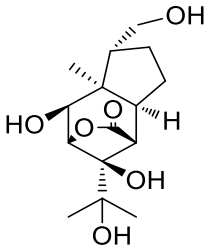
[**203**] Dendronobilin J



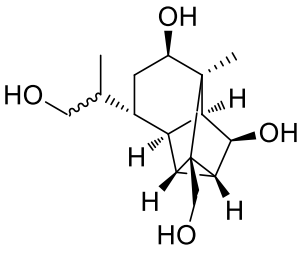
[**204**] Dendronobilin K



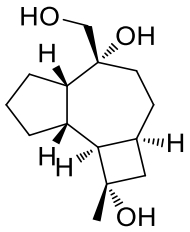
[**205**] Dendronobilin L



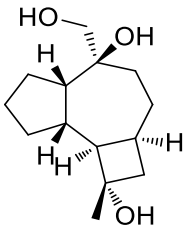
[**206**] Dendronobilin M



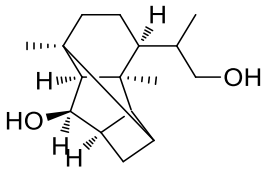
[**207**] Dendronobilin N



[**208**] Dendrowardol A



[**209**] Dendrowardol B



[**210**] Dendrowardol C

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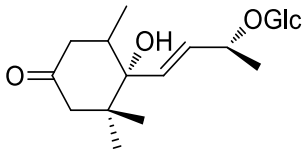
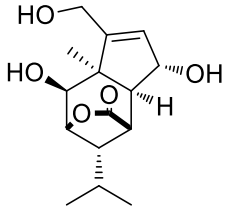
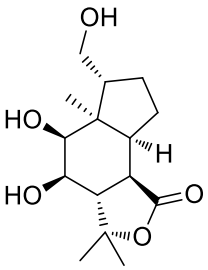
Figure 4 (continued)



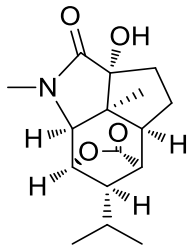
[**211**] Corchoionoside C



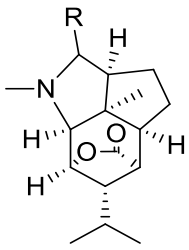
[**212**] Crystallinin



[**213**] Findlayanin

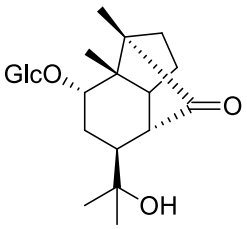


[**214**] 3-Hydroxy-2-oxodendrobine



[**215**] Dendrobine: R = H

[**216**] (-)-(1*R*,2*S*,3*R*,4*S*,5*R*,6*S*,9*S*,11*R*)-11-Carboxymethyldendrobine: R = CH_2COOH



[**217**] Dendromoniliside A

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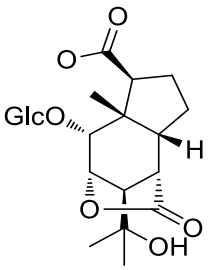
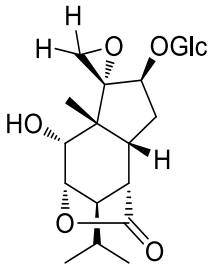
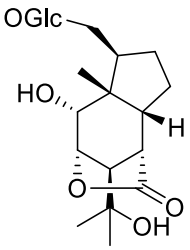


[**218**] Dendromoniliside B

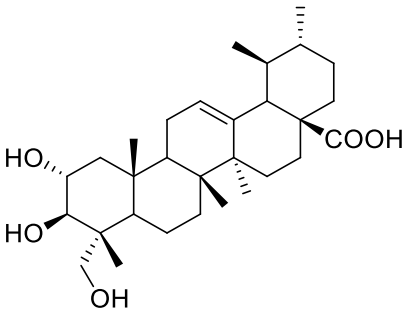


[**219**] Dendromoniliside C



[**220**] Dendromoniliside D

Figure 4 (continued)



[**221**] Asiatic acid

Figure 4 (continued)

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Table 4 Distribution of miscellaneous compounds in the *Dendrobium* plants

Category and Compound	**Plant**	**Plant part**	**Reference**
Aliphatic acid derivatives			
Aliphalic acids [**222**]	*D. clavatum* var. *aurantiacum*	Stem	Chang *et al.*, 2001
Aliphatic alcohols [**223**]	*D. clavatum* var. *aurantiacum*	Stem	Chang *et al.*, 2001
Malic acid [**224**]	*D. huoshanense*	Aerial part	Chang *et al.*, 2001
Dimethyl malate [**225**]	*D. huoshanense*	Aerial part	Chang *et al.*, 2010
Isopentyl butyrate [**226**]	*D. huoshanense*	Aerial part	Chang *et al.*, 2010
(-)-Shikimic acid [**227**]	*D. fuscescens*	Whole plant	Talapatra *et al.*, 1989
	D. huoshanense	Aerial part	Chang *et al.*, 2010
	D. longicornu	Stem	Hu *et al.*, 2008b
	D. pulchellum	Stem	Chanvorachote *et al.*, 2013
Decumbic acid [**228**]	*D. nobile*	Stem	Zhou *et al.*, 2016
Benzoic acid derivatives and phenolic compounds			
Salicylic acid [**229**]	*D. huoshanense*	Aerial part	Chang *et al.*, 2010
	D. williamsonii	Whole plant	Yang *et al.*, 2017b
p-Hydroxybenzoic acid [**230**]	*D. williamsonii*	Whole plant	Yang *et al.*, 2017b

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Table 4 (continued)

Category and Compound	**Plant**	**Plant part**	**Reference**
Gallic acid [**231**]	*D. longicornu*	Whole plant	Li *et al.*, 2009a
Syringic acid [**232**]	*D. crystallinum*	Stem	Wang *et al.*, 2009
Vanillic acid [**233**]	*D. crystallinum*	Stem	Hu *et al.*, 2012
	D. williamsonii	Whole plant	Li *et al.*, 2009a
Protocatechuic acid [**234**]	*D. nobile*	Stem	Ye and Zhao, 2002
3-Hydroxy-2-methoxy-5,6-dimethylbenzoic acid [**235**]	*D. crystallinum*	Stem	Wang *et al.*, 2009
Antiarol [**236**]	*D. chrysotoxum*	Stem	Hu *et al.*, 2012
Ethylhaematommate [**237**]	*D. longicornu*	Whole plant	Li *et al.*, 2009a
p-Hydroxybenz-aldehyde [**238**]	*D. devonianum*	Whole plant	Limpanit *et al.*, 2016
	D. falconeri	Stem	Yang *et al.*, 2017b
Vanillin [**239**]	*D. williamsonii*	Whole plant	Hu *et al.*, 2008b
Vanilloside [**240**]	*D. denneanum*	Stem	Pan *et al.*, 2012
Methyl 4-hydroxy-benzoate [**241**]	*D. williamsonii*	Whole plant	Hu *et al.*, 2012
Methyl β-orsellinate [**242**]	*D. longicornu*	Stem	Li *et al.*, 2009a

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Table 4 (continued)

Category and Compound	**Plant**	**Plant part**	**Reference**
Tachioside [**243**]	*D. denneanum*	Stem	Pan *et al.*, 2012
Dendroside [**244**]	*D. nobile*	Stem	Zhou *et al.*, 2017
Phenylpropanoids			
Alkyl 4′-hydroxy-*trans*-cinnamates [**245**]	*D. clavatum* var. *aurantiacum*	Stem	Chang *et al.*, 2001
Alkyl *trans*-ferulates [**246**]	*D. clavatum* var. *aurantiacum*	Stem	Chang *et al.*, 2001
Defuscin [**247**]	*D. aurantiacum* var. *denneanum*	Stem	Yang *et al.*, 2006b
	D. moniliforme	Stem	Bi *et al.*, 2004
n-Octacosyl ferulate [**248**]	*D. aurantiacum* var. *denneanum*	Stem	Yang *et al.*, 2006b
	D. moniliforme	Stem	Bi *et al.*, 2004
n-Triacontyl *p*-hydroxy-*cis*-cinnamate [**249**]	*D. moniliforme*	Stem	Bi *et al.*, 2004
Tetratriacontanyl-*trans*-*p*-coumarate [**250**]	*D. williamsonii*	Whole plant	Rungwichaniwat *et al.*, 2014
n-Docosyl *trans*-ferulate [**251**]	*D. longicornu*	Whole plant	Li *et al.*, 2009a
	D. williamsonii	Whole plant	Rungwichaniwat *et al.*, 2014
trans-Tetracosyl ferulate [**252**]	*D. tortile*	Whole plant	Limpanit *et al.*, 2016

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Table 4 (continued)

Category and Compound	**Plant**	**Plant part**	**Reference**
Ferulaldehyde [**253**]	*D. longicornu*	Whole plant	Li *et al.*, 2009a
Ferulic acid [**254**]	*D. secundum*	Stem	Sritularak *et al.*, 2011b
cis-Hexacosanoyl ferulate [**255**]	*D. tortile*	Whole plant	Limpanit *et al.*, 2016
2-(*p*-Hydroxyphenyl) ethyl *p*-coumarate [**256**]	*D. falconeri*	Stem	Sritularak and Likhitwitayawuid, 2009
Dihydroconiferyl dihydro-*p*-coumarate [**257**]	*D. formosum*	Whole plant	Inthongkaew *et al.*, 2017
	D. nobile	Stem	Zhang *et al.*, 2006
	D. williamsonii	Whole plant	Yang *et al.*, 2017b
1-[4-(β-D-Glucopyranosyloxy)-3,5-dimethoxyphenyl]-1-propanone [**258**]	*D. aurantiacum* var. *denneanum*	Stem	Xiong *et al.*, 2013
Coniferyl alcohol [**259**]	*D. trigonopus*	Stem	Hu *et al.*, 2008a
p-Hydroxyphenyl propionic methyl ester [**260**]	*D. aphyllum*	Whole plant	Chen *et al.*, 2008a
Phloretic acid [**261**]	*D. ellipsophyllum*	Whole plant	Tanagornmeatar *et al.*, 2014

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Table 4 (continued)

Category and Compound	**Plant**	**Plant part**	**Reference**
Dihydroconiferyl alcohol [**262**]	*D. longicornu*	Stem	Hu *et al.*, 2008b
Salidrosol [**263**]	*D. chrysotoxum*	Stem	Hu *et al.*, 2012
Shashenoside I [**264**]	*D. aurantiacum* var. *denneanum*	Stem	Xiong *et al.*, 2013
Syringin [**265**]	*D. aurantiacum* var. *denneanum*	Stem	Xiong *et al.*, 2013
Tetracosyl(*Z*)-*p*-coumarate [**266**]	*D. falconeri*	Whole plant	Sritularak and Likhitwitayawuid, 2009
Coumarins			
Ayapin [**267**]	*D. densiflorum*	Stem	Fan *et al.*, 2001
Coumarin [**268**]	*D. aurantiacum* var. *denneanum*	Stem	Yang *et al.*, 2006b
	D. clavatum var. *aurantiacum*	Stem	Chang *et al.*, 2001
Denthyrsin [**269**]	*D. thyrsiflorum*	Stem	Zhang *et al.*, 2005
Scoparone [**270**]	*D. densiflorum*	Stem	Fan *et al.*, 2001
	D. palpebrae	Whole plant	Kyokong *et al.*, 2018
	D. thyrsiflorum	Stem	Zhang *et al.*, 2005
	D. williamsonii	Whole plant	Yang *et al.*, 2017b
Scopoletin [**271**]	*D. densiflorum*	Stem	Fan *et al.*, 2001

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Table 4 (continued)

Category and Compound	**Plant**	**Plant part**	**Reference**
Lignans and neolignans			
Dehydrodiconiferyl alcohol-4-*O*-β-D-glucoside [**272**]	*D. chrysanthum*	Stem	Ye *et al.*, 2004
Balanophonin [**273**]	*D. williamsonii*	Whole plant	Yang *et al.*, 2017b
Episyringaresinol [**274**]	*D. chrysotoxum*	Stem	Hu *et al.*, 2012
	D. longicornu	Stem	Hu *et al.*, 2008b
	D. nobile	Stem	Zhang *et al.*, 2008b
Episyringaresinol 4′′-*O*- β-D-glucopyranoside [**275**]	*D. moniliforme*	Stem	Zhao *et al.*, 2003
(-)-(7*S*,8*R*,7′*E*)-4-Hydroxy-3,3',5,5'-tetramethoxy-8,4′-oxyneolign-7′-ene-7,9,9′-triol-7,9′-bis-*O*-β-D-glucopyranoside [**276**]	*D. aurantiacum* var. *denneanum*	Stem	Xiong *et al.*, 2013
Lyoniresinol [**277**]	*D. chrysanthum*	Stem	Ye *et al.*, 2004
(-)-Syringaresinol-4,4′-bis-*O*-β-D-glucopyranoside [**278**]	*D. aurantiacum* var. *denneanum*	Stem	Xiong *et al.*, 2013
Syringaresinol-4-*O*-D-monoglucopyranoside [**279**]	*D. aurantiacum* var. *denneanum*	Stem	Xiong *et al.*, 2013

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Table 4 (continued)

Category and Compound	**Plant**	**Plant part**	**Reference**
(-)-Medioresinol [**280**]	*D. loddigesii*	Whole plant	Ito *et al.*, 2010b
(-)-Pinoresinol [**281**]	*D. loddigesii*	Whole plant	Ito *et al.*, 2010b
Dendrolactone [**282**]	*D. nobile*	Stem	Zhou *et al.*, 2016
Erythro-1-(4-*O*-β-D-glucopyranosyl-3-methoxyphenyl)-2-[4-(3-hydroxypropyl)-2,6-dimethoxyphenoxy]-1,3-propanediol [**283**]	*D. longicornu*	Stem	Hu *et al.*, 2008b
Syringaresinol [**284**]	*D. secundum*	Stem	Sritularak *et al.*, 2011b
	D. williamsonii	Whole plant	Yang *et al.*, 2017b
Acanthoside B [**285**]	*D. chrysanthum*	Stem	Ye *et al.*, 2004
Liriodendrin [**286**]	*D. pulchellum*	Stem	Chanvorachote *et al.*, 2013
(-)-(8*R*,7′*E*)-4-Hydroxy-3,3′,5,5′-tetra-methoxy-8,4′-oxyneolign-7′-ene-9,9′-diol-4,9-bis-*O*-β-D-glucopyranoside [**287**]	*D. auranticum*	Stem	Li *et al.*, 2014

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Table 4 (continued)

Category and Compound	**Plant**	**Plant part**	**Reference**
(-)-(8*S*,7′*E*)-4-Hydroxy-3,3′,5,5′-tetramethoxy-8,4′-oxyneolign-7′-ene-9,9′-diol 4,9-bis-*O*-β-D-glucopyranoside [**288**]	*D. auranticum*	Stem	Li *et al.*, 2014
(-)-(8*R*,7′*E*)-4-hydroxy-3,3′,5,5′,9′-penta-methoxy-8,4′-oxyneolign-7′-ene-9-ol-4,9-bis-*O*-β-D-glucopyranoside [**289**]	*D. auranticum*	Stem	Li *et al.*, 2014
Fluorenones			
Denchrysan A [**290**]	*D. chrysotoxum*	Whole plant	Li *et al.*, 2009c
Dendroflorin [**291**]	*D. aurantiacum* var. *denneanum*	Stem	Yang *et al.*, 2006b
	D. brymerianum	Whole plant	Klongkumnuankarn *et al.*, 2015
	D. palpebrae	Whole plant	Kyokong *et al.*, 2018
Dengibsin [**292**]	*D. aurantiacum var. denneanum*	Stem	Yang *et al.*, 2006b
	D. chrysanthum	Stem	Yang *et al.*, 2006a
	D. chrysotoxum	Whole plant	Li *et al.*, 2009c

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Table 4 (continued)

Category and Compound	**Plant**	**Plant part**	**Reference**
Nobilone [**293**]	*D. brymerianum*	Whole plant	Klongkumnuankarn *et al.*, 2015
	D. nobile	Stem	Zhang *et al.*, 2007b
	D. palpebrae	Whole plant	Kyokong *et al.*, 2018
1,4,5-Trihydroxy-7-methoxy-9H-fluoren-9-one [**294**]	*D. chrysotoxum*	Whole plant	Chen *et al.*, 2008b
2,4,7-Trihydroxy-5-methoxy-9-fluorenone [**295**]	*D. chrysotoxum*	Stem	Yang *et al.*, 2004
2,4,7-Trihydroxy-1,5-dimethoxy-9-fluorenone [**296**]	*D. chrysotoxum*	Stem	Yang *et al.*, 2004
Denchrysan B [**297**]	*D. brymerianum*	Whole plant	Klongkumnuankarn *et al.*, 2015
	D. chrysanthum	Whole plant	Ye *et al.*, 2003

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Table 4 (continued)

Category and Compound	**Plant**	**Plant part**	**Reference**
Others			
3,6,9-Trihydroxy-3,4-dihydroanthracen-1-(2*H*)-one [**298**]	*D. chrysotoxum*	Stem	Hu *et al.*, 2012
Palmarumycin JC2 [**299**]	*D. crystallinum*	Stem	Wang *et al.*, 2009
Dehydrovomifoliol [**300**]	*D. loddigesii*	Whole plant	Ito *et al.*, 2010b
4-(2-Hydroxypropyl)-2(5H)-furanone [**301**]	*D. tortile*	Whole plant	Limpanit *et al.*, 2016
5,7-Dihydroxy-chromen-4-one [**302**]	*D. ellipsophyllum*	Whole plant	Tanagornmeatar *et al.*, 2014

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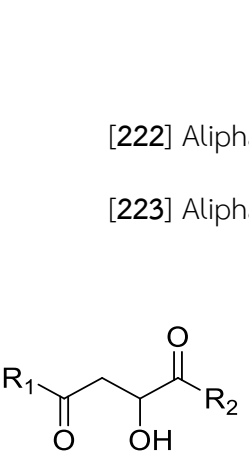
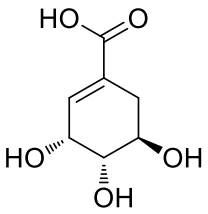


[**222**] Aliphatic acids: R = COOH, n = 19-31

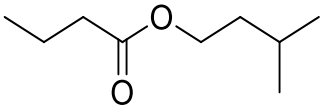
[**223**] Aliphatic alcohols: R = OH, n = 22-32

[**224**] Malic acid: R_1 = R_2 = OH

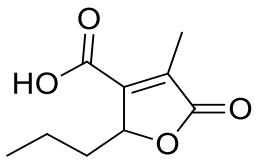
[**225**] Dimethyl malate: R_1 = R_2 = OMe



[**227**] (-)-Shikimic acid



[**226**] Isopentyl butyrate



[**228**] Decumbic acid

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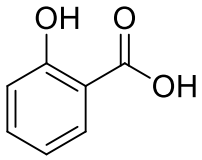
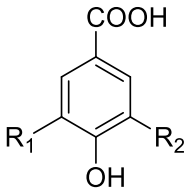


[**229**] Salicylic acid

Figure 5 Structures of miscellaneous compounds previously isolated from *Dendrobium* plants



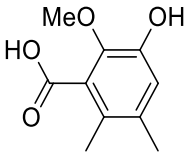
[**230**] *p*-Hydroxybenzoic acid: $R_1 = R_2 = H$

[**231**] Gallic acid: $R_1 = R_2 = OH$

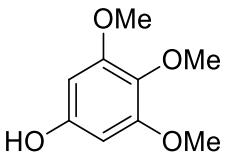
[**232**] Syringic acid: $R_1 = R_2 = OMe$

[**233**] Vanillic acid: $R_1 = H$, $R_2 = OMe$

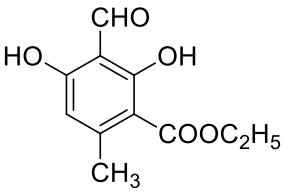
[**234**] Protocatechuic acid: $R_1 = H$, $R_2 = OH$



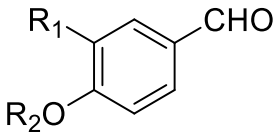
[**235**] 3-Hydroxy-2-methoxy-5,6-dimethylbenzoic acid



[**236**] Antiarol



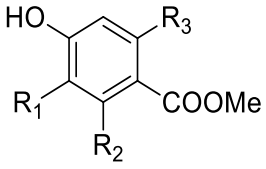
[**237**] Ethylhaematommate



[**238**] *p*-Hydroxybenzaldehyde: $R_1 = H$, $R_2 = H$

[**239**] Vanillin: $R_1 = OMe$, $R_2 = H$

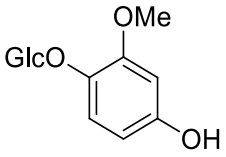
[**240**] Vanilloside : $R_1 = OMe$, $R_2 = -Glc$



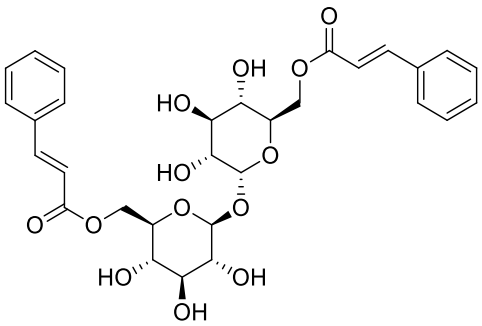
[**241**] Methyl 4-hydroxybenzoate $R_1 = R_2 = R_3 = H$

[**242**] Methyl β-orsellinate: $R_1 = R_3 = CH_3$, $R_2 = OH$

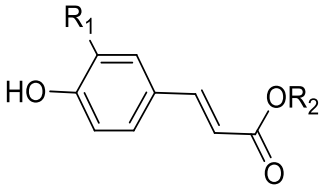
Figure 5 (continued)



[**243**] Tachioside



[**244**] Dendroside



[**245**] Alkyl 4′-hydroxy-*trans*-cinnamates: R_1 = H, R_2 = C_nH_{2n+1}, n =22-32

[**246**] Alkyl *trans*-ferulates: R_1 = OMe, R_2 = C_nH_{2n+1}, n = 18-28, 30

[**247**] Defuscin: R_1 = H, R_2 = $(CH_2)_{29}CH_3$

[**248**] *n*-Octacosyl ferulate: R_1 = OMe, R_2 = $(CH_2)_{27}CH_3$

[**249**] *n*-Triacontyl *p*-hydroxy-*cis*-cinnamate: R_1 = H, R_2 = $C_{30}H_{61}$

[**250**] Tetratriacontanyl-*trans*-*p*-coumarate: R_1 = H, R_2 = $(CH_2)_{33}CH_3$

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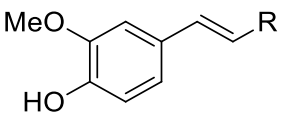


[**251**] *n*-Docosyl *trans*-ferulate:

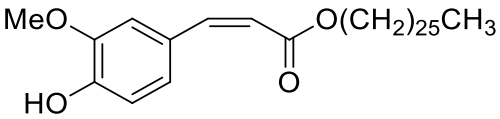
R = $COOCH_2(CH_2)_{20}CH_3$

[**252**] *trans*-Tetracosylferulate:

R = $COOCH_2(CH_2)_{22}CH_3$

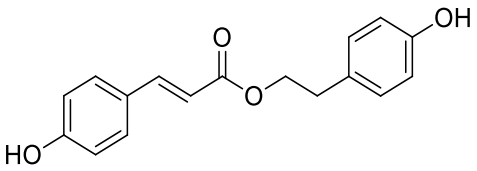
[**253**] Ferulaldehyde: R = CHO

[**254**] Ferulic acid: R = COOH

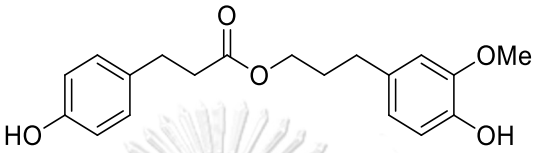


[**255**] *cis*-Hexacosanoyl ferulate

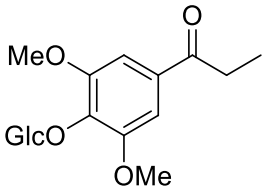
Figure 5 (continued)



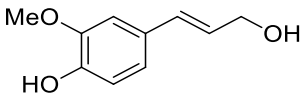
[**256**] 2-(*p*-Hydroxyphenyl) ethyl *p*-coumarate



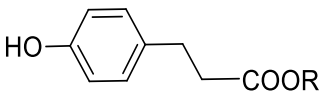
[**257**] Dihydroconiferyl dihydro-*p*-coumarate



[**258**] 1-[4-(β-D-glucopyranosyloxy)-3,5-dimethoxyphenyl]-1-propanone

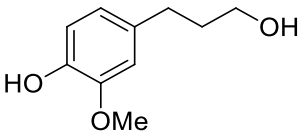


[**259**] Coniferyl alcohol

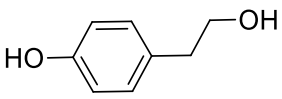


[**260**] *p*-Hydroxyphenyl propionic methyl ester: R = CH_3

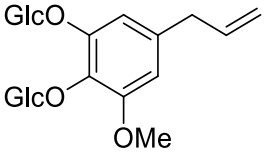
[**261**] Phloretic acid: R = H



[**262**] Dihydroconiferyl alcohol

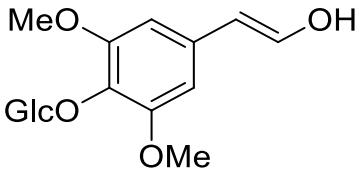


[**263**] Salidrosol

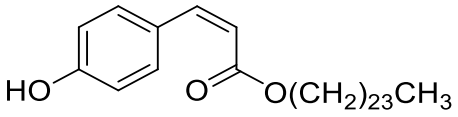


[**264**] Shashenoside I

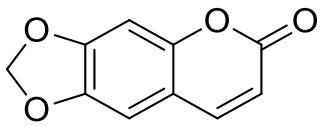
Figure 5 (continued)



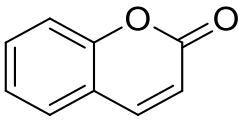
[**265**] Syringin



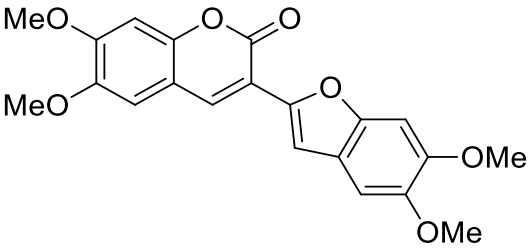
[**266**] Tetracosyl (*Z*)-*p*-coumarate



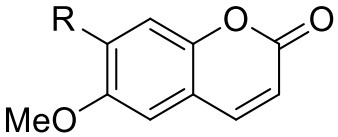
[**267**] Ayapin



[**268**] Coumarin



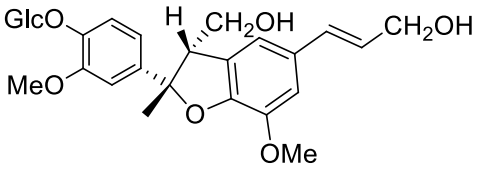
[**269**] Denthyrsin



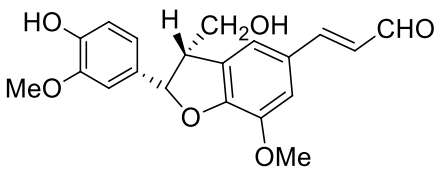
[**270**] Scoparone: R = OMe

[**271**] Scopoletin: R = OH

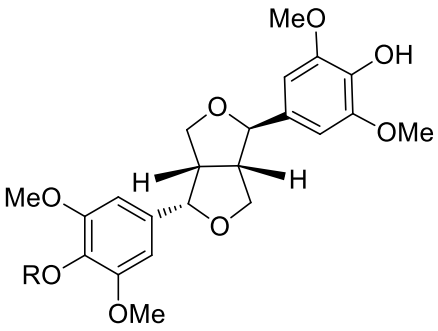
Figure 5 (continued)



[**272**] Dehydrodiconiferyl alcohol-4-*O*-β-D-glucoside

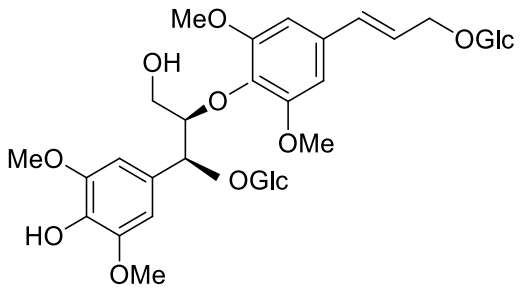


[**273**] Balanophonin

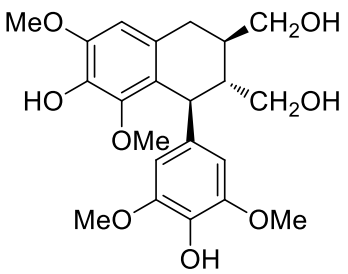


[**274**] Episyringaresinol: R = H

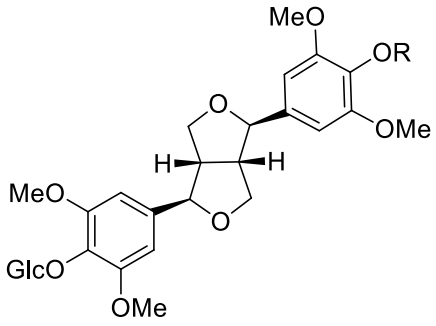
[**275**] Episyringaresinol 4″-*O*-β-D-glucopyranoside: R = Glc



[**276**] (-)-(7*S*,8*R*,7′*E*)-4-Hydroxy-3,3′,5,5′-tetramethoxy-8,4′-oxyneolign-7′-ene-7,9,9′-triol-7,9′-bis-*O*-β-D-glucopyranoside



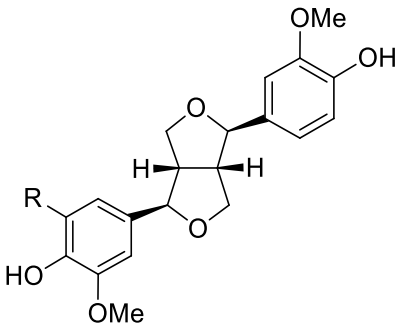
[**277**] Lyoniresinol



[**278**] (-)-Syringaresinol-4,4′-bis-*O*-β-D-glucopyranoside: R = Glc

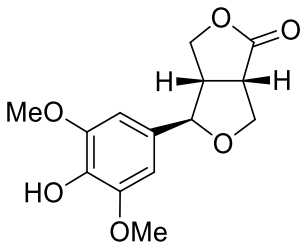
[**279**] Syringaresinol-4-*O*-D-monoglucopyranoside: R = H

Figure 5 (continued)

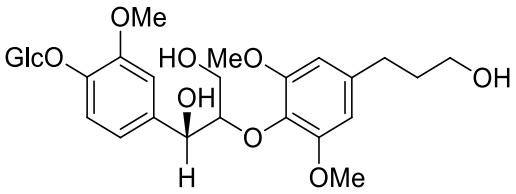


[**280**] (-)-Medioresinol: R = OMe

[**281**] (-)-Pinoresinol: R = H



[**282**] Dendrolactone



[**283**] *Erythro*-1-(4-*O*-β-D-glucopyranosyl-3-methoxyphenyl)-2-[4-(3-hydroxypropyl)-2,6-dimethoxyphenoxy]-1,3-propanediol

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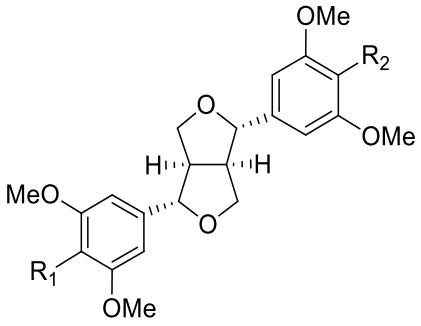
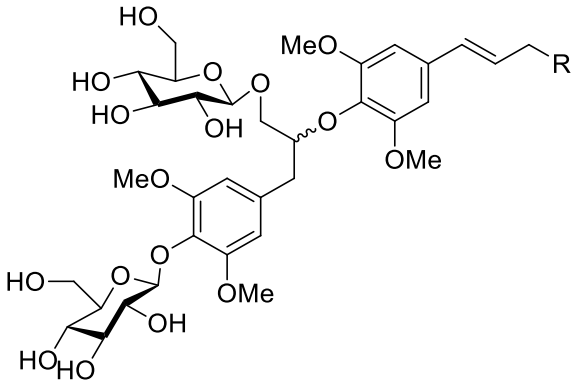
Figure 5 (continued)



	R_1	R_2
[**284**] Syringaresinol	OH	OH
[**285**] Acanthoside B	OGlc	OH
[**286**] Liriodendrin	OGlc	OGlc

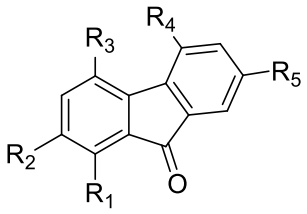


[**287**] (-)-(8*R*,7′*E*)-4-Hydroxy-3,3′,5,5′-tetramethoxy-8,4′-oxyneolign-7′-ene-9,9′-diol 4,9-bis-*O*-β-D-glucopyranoside: R = OH; 8*R*

[**288**] (-)-(8*S*,7′*E*)-4-Hydroxy-3,3′,5,5′-tetramethoxy-8,4′-oxyneolign-7′-ene-9,9′-diol 4,9-bis-*O*-β-D-glucopyranoside: R = OH; 8*S*

[**289**] (-)-(8*R*,7′*E*)-4-Hydroxy-3,3′,5,5′,9′-pentamethoxy-8,4′-oxyneolign-7′-ene-9-ol 4,9-bis-*O*-β-D-glucopyranoside: R = OMe; 8*R*

Figure 5 (continued)



	R_1	R_2	R_3	R_4	R_5
[**290**] Denchrysan A	H	OH	OH	OMe	OH
[**291**] Dendroflorin	OH	H	OH	OMe	OH
[**292**] Dengibsin	H	OH	OMe	OH	H
[**293**] Nobilone	H	OH	H	OMe	OH
[**294**] 1,4,5-Trihydroxy-7-methoxy-9*H*-fluoren-9-one	OH	H	OH	OH	OMe
[**295**] 2,4,7-Trihydroxy-5-methoxy-9-fluorenone	OMe	OH	OH	H	OH
[**296**] 2,4,7-Trihydroxy-1,5-dimethoxy-9-fluorenone	OMe	OH	OH	OMe	OH

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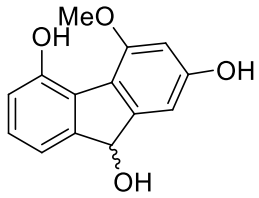
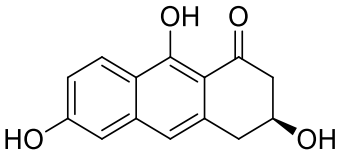
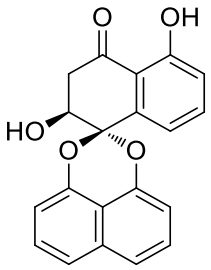


[**297**] Denchrysan B

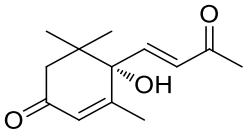
Figure 5 (continued)



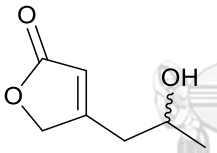
[**298**] 3,6,9-Trihydroxy-3,4-dihydroanthracen-1-(2*H*)-one



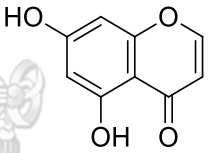
[**299**] Palmarumycin JC2



[**300**] Dehydrovomifoliol



[**301**] 4-(2-Hydroxypropyl)-2(5*H*)-furanone



[**302**] 5,7-Dihydroxy-chromen-4-one

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Figure 5 (continued)

2. Traditional uses and biological activities of *Dendrobium* species

A number of *Dendrobium* orchids have been used as folk medicines since antiquity. There have been numerous ancient records on their medicinal properties in many countries, for example Japan, India and especially in China. In traditional Chinese medicine (TCM), they are called with a common name "Shihu", which includes about thirty species of *Dendrobium,* such as *D. nobile*, *D. chrysotoxum*, *D. officinale*, *D. fimbriatum,* etc. Shihu is used for various purposes such as nourishing the stomach, enhancing production of body fluids, reducing fever, treatment of diabetes, used as immunomodulatory and anti-aging. Because of these various medicinal properties, researches on chemical constituents of *Dendrobium* orchids and their pharmacological activities have been increasing, particularly in age-related pathological conditions (Xu *et al.*, 2013; Cakova *et al.*, 2017).

Several scientific reports on *Dendrobium* plants have revealed many biological activities on age-related disorders of their secondary metabolites, for example, anticancer, neuroprotective, antidiabetic and immunomodulatory activities (Cakova *et al.*, 2017). In addition, some other biological activities such as anti-inflammatory, antiviral, antibacterial, antimalarial and antiplatelet activities, have also been reported (Teixeira da Silva and Ng, 2017).

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Regarding antidiabetic activity, some *Dendrobium* plants have been investigated for hypoglycemic activity. There were reports of crude extracts from *D. candidum* (Wu *et al.*, 2004) and *Dendrobium* mixture (Li and Deng, 2012) that could lower the blood glucose level in rodent model by various mechanisms. The polysaccharides from *D. huoshanense, D. officinale* and *D. nobile* also showed oral hypoglycemic effect in mice at dosages of 200, 100 and 50 mg/kg (Pan *et al.*, 2014). Furthermore, several secondary metabolites were isolated from these orchids and studied for biological activities related to antidiabetic activity. A major group of compound with hypoglycemic potential is the polyphenols with α-glucosidase

inhibitory activity. For example, loddigesiinols G-J [**143-146**] from *D. loddigesii* exhibited stronger inhibitory activity than the positive control *trans*-resveratrol (Lu *et al.*, 2014a). A flavonol glycoside [5-hydroxy-3-methoxy-flavone-7-*O*-β-D-apiosyl-(1-6)-β-D-glucoside] [**172**], and gigantol [**16**] from *D. devonianum* (Sun *et al.*, 2014), (2S)-eriodictyol [**159**], 3,4-dihydroxy-5,4′-dimethoxybibenzyl [**31**] and dendrofalconerol A [**68**] from *D. tortile* (Limpanit *et al.*, 2016), as well as confusarin [**129**] and 5-methoxy-7-hydroxy-9,10-dihydro-1,4-phenanthrenequinone [**120**] from *D. formosum* (Inthongkaew *et al.*, 2017) were also reported as inhibitors of α-glucosidase with higher potency than the drug acarbose.

Various compounds from *Dendrobium* species showed anticancer activity. For instance, moscatilin [**22**], gigantol [**16**], lusianthridin [**81**] and dendroflorin [**292**] from *D. brymerianum* exhibited cytotoxic activity against human lung cancer cell line (H46). Moreover, lusianthridin [**81**] and dendroflorin [**292**] displayed antimigratory activity (Klongkumnuankarn *et al.*, 2015). Bibenzyl derivatives from *D. signatum*, including dendrosignatol [**71**], 3,4-dihydroxy-5,4′-dimethoxybibenzyl [**31**], dendrocandin I [**46**], dendrofalconerol A [**68**] and dendrocandin B [**39**], were cytotoxic against human breast cancer, liver hepatocellular carcinoma and colorectal tumor cell lines (Mittraphab *et al.*, 2016).

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Some examples of biological activities of *Dendrobium* constituents on non-age-related disorders are as follows. Five compounds from methanolic extract of *D. venustum*, including densiflorol B [**116**], phoyunnanin E [**136**], batatasin III [**5**], gigantol [**16**], and phoyunnanin C [**135**], showed antimalarial activity. The first two compounds were more potent than others and the drugs dihydroartemisinin and mefloquine (Sukphan *et al.*, 2014). Besides, 2-(*p*-hydroxyphenyl) ethyl *p*-coumarate [**256**], a phenylpropanoid from *D. falconeri*, showed moderate anti-herpes simplex virus type 1 (HSV-1) activity when its viral plaque reduction effect was compared with

acyclovir (Sritularak and Likhitwitayawuid, 2009). Moscatin [**122**], moscatilin [**22**] and moscatilin diacetate [**23**], which are bibenzyls isolated derivatives from *D. loddigesii* stem showed anti-aggregation effect when tested on rabbit platelet (Chen *et al.*, 1994). Moscatilin [**22**] together with gigantol [**16**], homoeriodictyol [**157**], scoparone [**270**], and scopoletin [**271**] from *D. densiflorum* also potently inhibited rat platelet aggregation (Fan *et al.*, 2001).

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CHAPTER III

EXPERIMENTAL

1. Source of plant materials

The whole plants of *Dendrobium scabrilingue* Lindl. were purchased from Jatujak market, Bangkok, Thailand, in December 2015. Plant identification was done by Associate Professor Boonchoo Sritularak and comparison with the database of the Botanical Garden Organization. A voucher specimen (BS-DScab-122558) has been deposited at the herbarium of the Department of Pharmacognosy and Pharmaceutical Botany, Faculty of Pharmaceutical Sciences, Chulalongkorn University.

2. General techniques

2.1 Analytical thin-layer chromatography (TLC)

2.1.1 Normal-phase thin-layer chromatography

Technique : One-dimension ascending

Absorbent : Silica gel 60 F254 precoated plate (E. Merck)

Temperature : Laboratory temperature (30-35 ºC)

Detection : 1. Ultraviolet light at wavelengths of 254 and 365 nm.

2. Spraying with anisaldehyde reagent (*p*-anisaldehyde 15 g in ethanol 250 mL and concentrated sulfuric acid 2.5 mL) and heating at 105 ºC for 10 minutes.

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2.1.2 Reverse-phase thin-layer chromatography

Technique : One-dimension ascending

Absorbent : RP C-18 precoated on aluminum sheet (Anal Tech)

Temperature : Laboratory temperature (30-35 ºC)

Detection : Ultraviolet light at wavelengths of 254 and 365 nm.

2.2 Column chromatography (CC)

2.2.1 Vacuum liquid chromatography (VLC)

Adsorbent : Silica gel 60 (No. 1.07734.2500), size 0.063-0.200 mm (E. Merck)

Packing method : Dry packing

Sample loading : The sample was dissolved in a small volume of organic solvent, adsorbed by a small quantity of the adsorbent, dried and then gradually placed on top of the column.

Detection : Each fraction was examined by TLC under UV light at the wavelengths of 254 and 365 nm.

2.2.2 Flash column chromatography (FCC), normal phase

Adsorbent : Silica gel 60 (No. 1.09385.2500), size 0.040-0.063 mm (E. Merck)

Packing method : Wet packing

Sample loading : The sample was dissolved in a small volume of organic solvent, adsorbed by a small quantity of the adsorbent, dried and then gradually placed on top of the column.

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Detection : Fractions were examined as described in section 2.2.1

2.2.3 Flash column chromatography (FCC), reverse phase

Adsorbent : C-18 (No. 1.10167.1000), size 40-63 μm (E. Merck)

Packing method : Wet packing

Sample loading : The sample was dissolved in a small volume of organic solvent, and then gradually loaded on top of the column.

Detection : Fractions were examined as described in section 2.2.1

2.2.4 Gel filtration chromatography

Gel filter : Sephadex LH-20 particle size 25-100 μm (GE Healthcare)

Packing method : An appropriate organic solvent was used as the eluent. Gel filter was suspended in the eluent, left standing about 24 hours prior to use and then poured into the column and left to set tightly.

Sample loading : The sample was dissolved in a small volume of the eluent and then gradually distributed on top of the column.

Detection : Fractions were examined in a similar manner as described in section 2.2.1

2.3.5 Semi-preparative high-pressure liquid chromatography (HPLC)

Column : COSMOSIL $5C_{18}$-AR-II (10ID x 250 mm)

Flow rate : 3 ml/min

Mobile phase : Isocratic 50% methanol in water

Sample preparation : The sample was dissolved in a small volume of the eluent and filtered through Millipore filter paper before injection.

Injection volume : 1 ml

Pump : LC-8A (Shimadzu)

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Detector : SPD-10A UV-Vis Detector (Shimadzu)

Recorder : C-R6A Chromatopac (Shimadzu)

Temperature : Room temperature

2.3 Spectroscopy

2.3.1 Mass spectra

Mass spectra were recorded on a Bruker micro TOF mass spectrometer (ESI-MS) (Department of Chemistry, Faculty of Sciences, Mahidol University and Medical Research Center, Faculty of Medicine, Chulalongkorn University).

2.3.2 Ultraviolet (UV) spectra

UV spectra were recorded on a Milton Roy Spectronic 3000 Array spectrophotometer (Pharmaceutical Research Instrument Center, Faculty of Pharmaceutical Sciences, Chulalongkorn University).

2.3.3 Infrared (IR) spectra

IR spectra were recorded on a Perkin-Elmer FT-IR 1760X spectrophotometer (Scientific and Technology Research Equipment Center, Chulalongkorn University).

2.3.4 Proton and carbon-13 nuclear magnetic resonance (^{1}H and ^{13}C-NMR) spectra

^{1}H NMR (300 MHz) and ^{13}C NMR (75 MHz) spectra were recorded on a Bruker Avance DPX-300 FT-NMR spectrometer (Faculty of Pharmaceutical Sciences, Chulalongkorn University).

^{1}H NMR (500 MHz) and ^{13}C NMR (125 MHz) spectra were recorded on a Bruker Avance III HD 500 NMR spectrometer (Scientific and Technology Research Equipment Center, Chulalongkorn University).

Solvents for NMR spectra were deuterated acetone (acetone-d_6), deuterated dimethyl sulfoxide (DMSO-d_6) and deuterated chloroform ($CDCl_3$). Chemical shifts were reported in ppm scale using the chemical shift of the solvent as the reference signal.

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2.3.5 Optical rotation

Optical rotation was measured on a Perkin-Elmer 341 polarimeter (Pharmaceutical Research Instrument Center, Faculty of Pharmaceutical Sciences, Chulalongkorn University).

2.4 Solvents

All organic solvents employed throughout this work were of commercial grade and were redistilled prior to use.

3. Extraction and isolation

3.1 Extraction

The dried powdered whole plants of *D. scabrilingue* (4.8 kg) were macerated with 20 liters of methanol for 72 hours three times at room temperature. The organic solvent was evaporated by rotary evaporator to give 240 g of methanol crude extract, which was later screened for α -glucosidase inhibitory activity. The crude extract was suspended in water and partitioned with EtOAc and then *n*-butanol to give EtOAc extract (170 g), *n*-butanol extract (30 g), and aqueous extract (35 g). All three extracts were evaluated for α-glucosidase inhibitory activity. Only the EtOAc extract showed high activity (more than 90% inhibition at 100 µg/ml). The other extracts showed less than 50% inhibition of enzyme activity. Thus, the EtOAc extract was selected for further study (**Scheme 1**).

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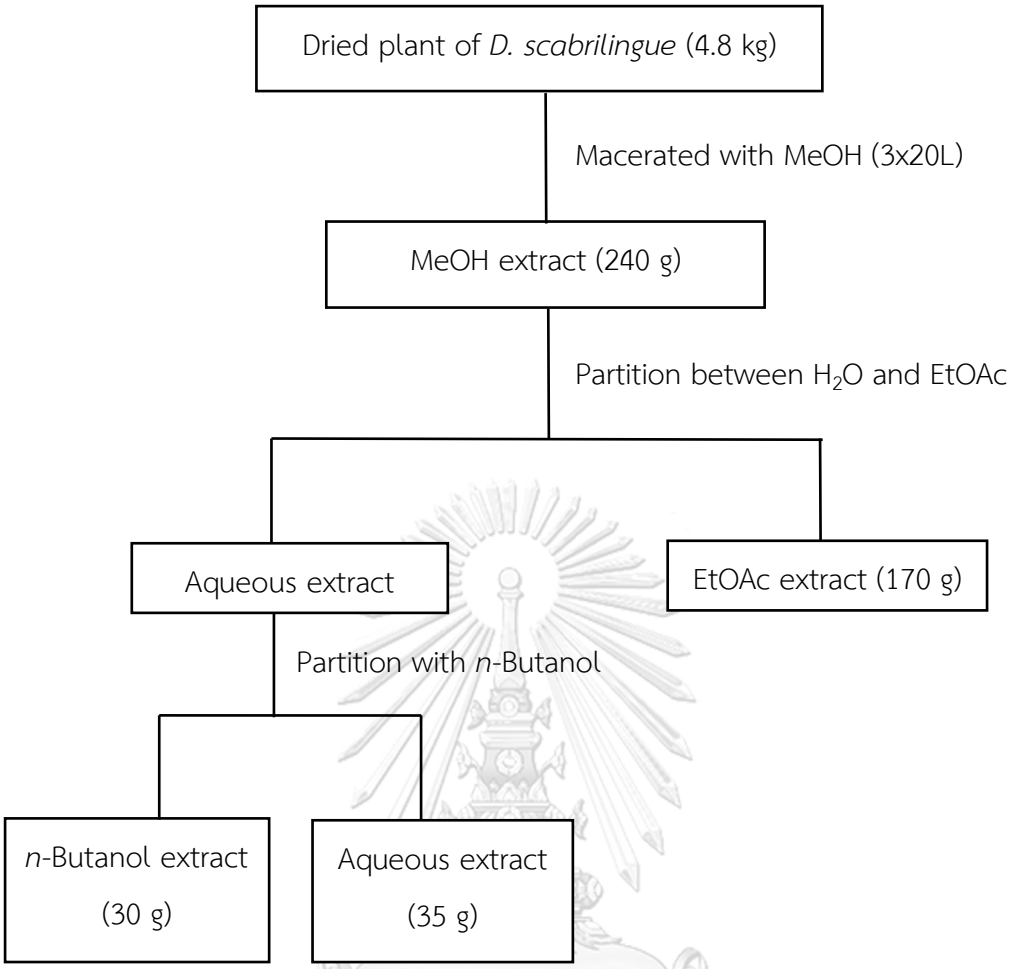

Scheme 1 Extraction of *Dendrobium scabrilingue* whole plants

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3.2 Isolation

EtOAc extract (100 g) was initially fractionated by vacuum liquid chromatography (VLC) as described in section 2.2.1. (**Scheme 2**). Silica gel was used as the stationary phase and a step gradient of hexane-EtOAc (1:0 to 0:1) as the mobile phase. The eluates (about 500 mL per fraction) were collected and examined by TLC (Silica gel, hexane-EtOAc = 1:1), then combined to give eight fractions (A-H).

3.2.1 Isolation of compounds DSC-3 [(*Z*)-ferulic acid tetracosyl ester] and DSC-4 [(*E*)-ferulic acid tetracosyl ester]

Fraction C (5.9 g) was further fractionated using silica gel FCC and eluted with gradient mixtures of hexane–CH_2Cl_2 (1:0 to 0:1) to give 18 fractions (C1-C18). Fraction C8 (1.9 g) was further separated on a similar column eluted with gradient mixtures of hexane–EtOAc (1:0 to 0:1) to give 8 fractions (C8a-C8h).

Fraction C8d (316 mg) was purified by gel filtration CC using Sephadex LH-20 in acetone to give 6 fractions (C8da-C8df). Fraction C8db (111 mg) was subjected to repeated silica gel FCC with hexane-EtOAc (97:3) as eluent to afford compounds DSC-3 (2 mg) and DSC-4 (45 mg). They were later identified as (*Z*)-ferulic acid tetracosyl ester and (*E*)-ferulic acid tetracosyl ester, respectively.

3.2.2 Isolation of compound DSC-1 (dendroscabrol A)

Fraction C8e (572 mg) was separated by silica gel FCC and eluted with hexane –CH_2Cl_2 (6:4) to give 4 fractions (C8ea-C8ed). Then fraction C8eb (42 mg) was purified on a Sephadex LH-20 column eluted with acetone to furnish compound DSC-1 (5 mg). This compound was characterized as a new structure and named dendroscabrol A.

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3.2.3 Isolation of compounds DSC-5 (gigantol) and DSC-6 (batatasin III)

Fraction D (8.2 g) was further fractionated by silica gel FCC, eluted with hexane–EtOAc (1:0 to 0:1 gradient) to provide 14 fractions (D1-D14).

Fraction D9 (1.5 g) was separated on a Sephadex LH-20 eluted with MeOH to give 3 fractions (D9a-D9c). Fraction D9b (1.1 g) was further separated by normal-phase silica gel FCC eluted with gradient mixtures of CH_2Cl_2-EtOAc (1:0 to 0:1) to yield compounds DSC-5 (48 mg) and DSC-6 (722 mg), which were identified as gigantol and batatasin III, respectively.

3.2.4 Isolation of compound DSC-7 (coelonin)

Fraction D10 (550 mg) was subjected to gel filtration CC using Sephadex LH-20 in MeOH to give 8 fractions (D10a-D10h). Fraction D10e was a pure compound DSC-7 (71 mg) and was identified as coelonin.

3.2.5 Isolation of compounds DSC-8 (aloifol I) and DSC-9 (lusianthridin)

Fraction D10c (145 mg) was further purified by silica gel FCC with CH_2Cl_2 as the mobile phase to afford DSC-8 (93 mg) and was identified as aloifol I. Fraction D10f (30 mg) was further purified by a similar column eluted with CH_2Cl_2 to provide compound DSC-9 (9 mg) and was identified as lusianthridin.

3.2.6 Isolation of compound DSC-10 (RF-3192C)

Fraction F (10.2 g) was fractionated by silica gel FCC and eluted with gradient mixtures of hexane–acetone (1:0 to 0:1) to give 14 fractions (F1-F14). Fraction F13 (1.8 g) was separated on a silica gel column with gradient mixture of CH_2Cl_2-MeOH (1:0 to 0:1) to provide 10 fractions (F13a-F13j)

Fraction F13h (100 mg) was purified by gel filtration CC using Sephadex LH-20 in MeOH to afford compound DSC-10 (11 mg), which was identified as RF-3192C.

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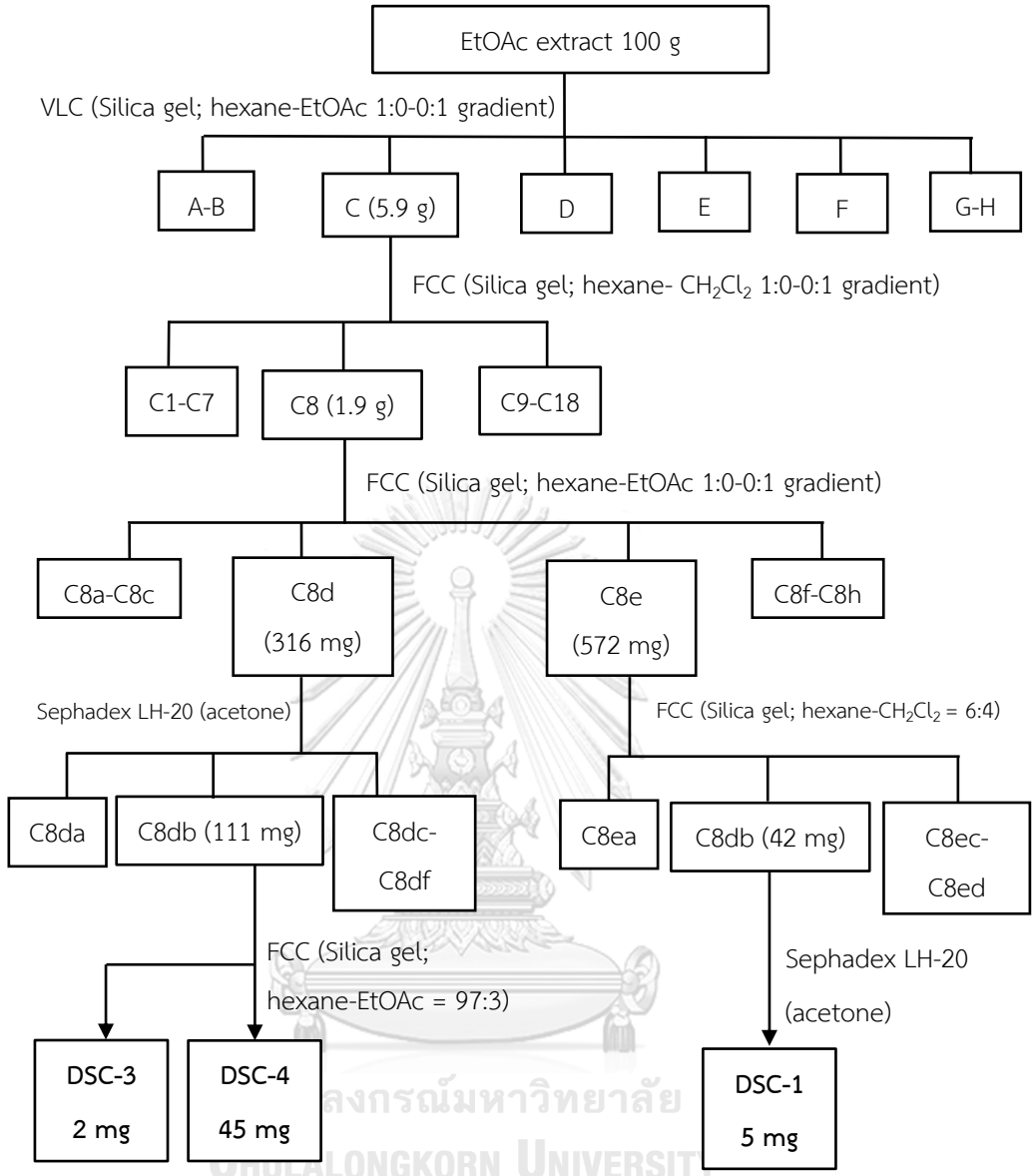
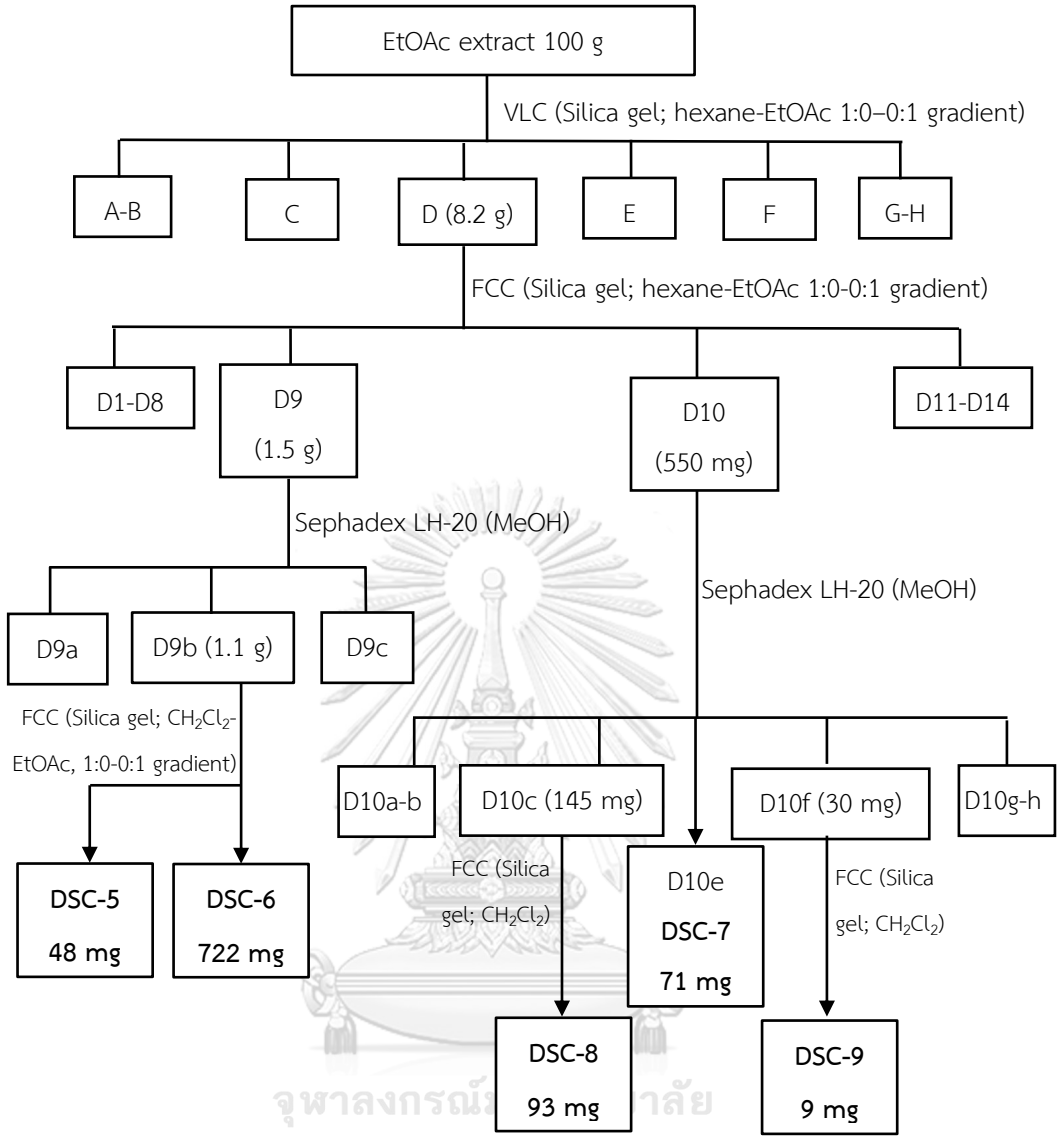
3.2.7 Isolation of compound DSC-2 (dendroscabrol B)

Fraction F13g (338 mg) was separated on a Sephadex LH-20 eluted with MeOH to give 12 fractions (F13ga-F13gl). Separation of fraction F13gf (69 mg) was performed by reverse phase FCC, eluted with gradient mixtures of H_2O-MeOH (1:0 to 0:1) to provide 8 fractions (F13gfa-F13gfh). Fraction F13gfc (17 mg) was further purified by semi-preparative HPLC, using C-18 column and H_2O-MeOH (1:1) as the mobile phase at the flow rate of 3 ml/min, to yield compound DSC-2 (3 mg). It was characterized as a new compound and named dendroscabrol B.



Scheme 2 Separation of fraction C from *Dendrobium scabrilingue* EtOAc extract



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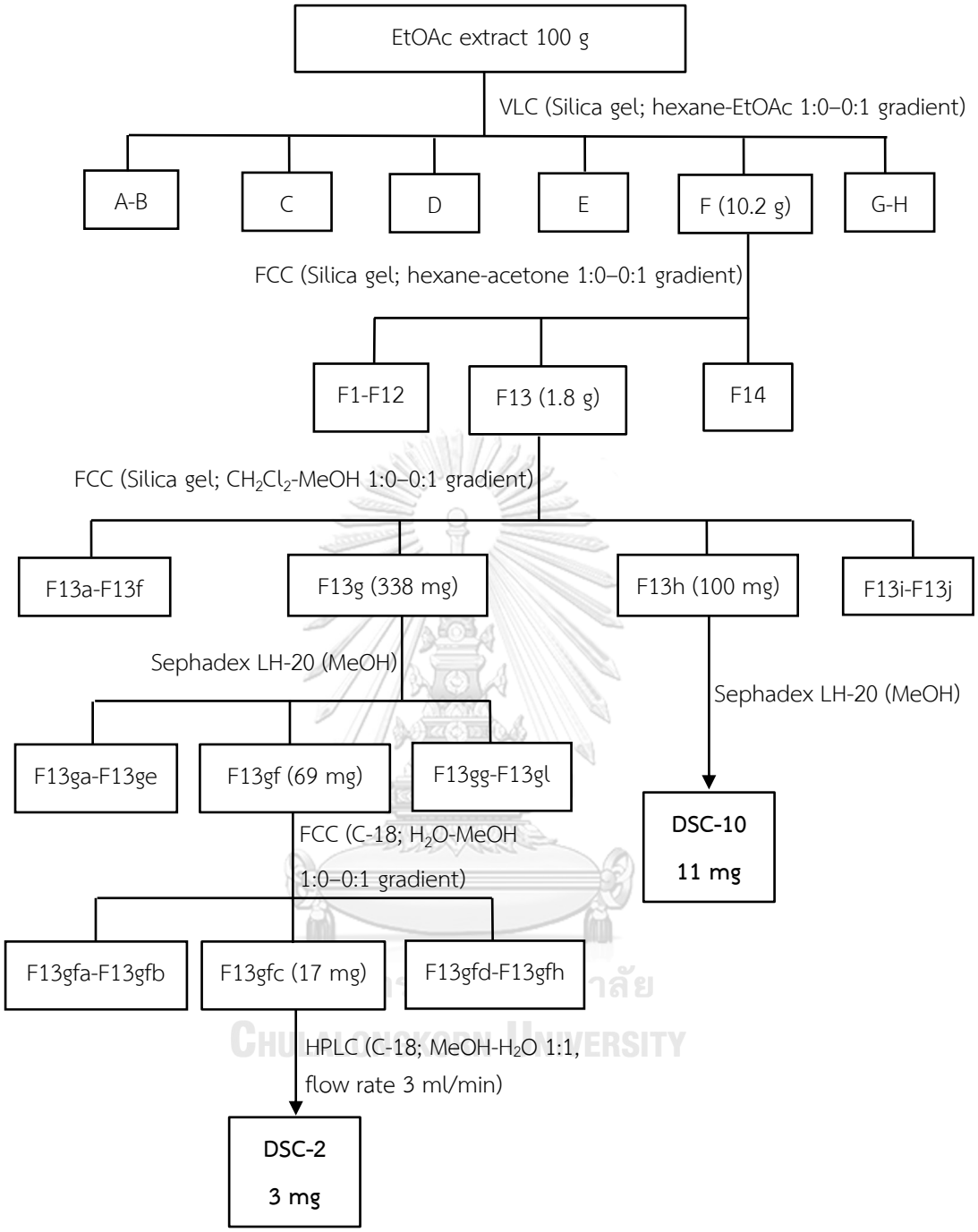
Scheme 3 Separation of fraction D from *Dendrobium scabrilingue* EtOAc extract



Scheme 4 Separation of fraction F from *Dendrobium scabrilingue* EtOAc extract

4. Physical and spectral data of isolated compounds

4.1 Compound DSC-1 (Dendroscabrol A)

Compound DSC-1 was obtained as a brown amorphous solid (5.0 mg, 0.00010% based on dried weight of whole plant). It was soluble in acetone and methanol.

HR-ESI-MS : $[M-H]^-$ ion at *m/z* 283.0973 ($C_{17}H_{15}O_4$); see **Figure 7**

UV : λ_{max} nm (log ε), in methanol: 220 (4.25), 260 (4.63), 285 (4.02), 345 (3.07), 360 (3.09); see **Figure 8**

FT-IR : ν cm^{-1} (film): 3468, 3007, 2956, 2924, 2852, 1729, 1616, 1473, 1286, 1223; see **Figure 9**

^{1}H NMR : δ ppm, 500 MHz, in acetone-d_6; see **Table 5, Figure 10**

^{13}C NMR : δ ppm, 125 MHz, in acetone-d_6; see **Table 5, Figure 11**

4.2 Compound DSC-2 (Dendroscabrol B)

Compound DSC-2 was obtained as a brown amorphous solid (3.0 mg, 0.00006% based on dried weight of whole plant). It was soluble in acetone and methanol.

HR-ESI-MS : $[M+Na]^+$ ion at *m/z* 523.1733 ($C_{30}H_{28}O_7Na$); see **Figure 15**

UV : λ_{max} nm (log ε), in methanol: 210 (4.60), 280 (3.25); see **Figure 16**

FT-IR : ν cm^{-1} (film): 3397, 2921, 2850, 1734, 1646, 1468, 1150; see **Figure 17**

Optical rotation : $[\alpha]_D^{20}$: +3.7 (*c* 0.1, MeOH)

^{1}H NMR : δ ppm, 500 MHz, in acetone-d_6; see **Table 6, Figure 18**

^{13}C NMR : δ ppm, 125 MHz, in acetone-d_6; see **Table 6, Figure 19**

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4.3 Compound DSC-3 [(*Z*)-ferulic acid tetracosyl ester]

Compound DSC-3 was obtained as a white amorphous solid (2.0 mg, 0.00004% based on dried weight of whole plant). It was soluble in acetone.

HR-ESI-MS : $[M+Na]^+$ ion at *m/z* 553.4207 ($C_{34}H_{58}O_4Na$); see **Figure 24**

^{1}H NMR : δ ppm, 300 MHz, in acetone-d_6; see **Table 7, Figure 25**

^{13}C NMR : δ ppm, 75 MHz, in acetone-d_6; see **Table 7, Figure 26**

4.4 Compound DSC-4 [(*E*)-ferulic acid tetracosyl ester]

Compound DSC-4 was obtained as a white amorphous solid (45.0 mg, 0.00094% based on dried weight of whole plant). It was soluble in chloroform.

HR-ESI-MS : $[M+Na]^+$ ion at *m/z* 553.4154 ($C_{34}H_{58}O_4Na$); see **Figure 29**

^{1}H NMR : δ ppm, 300 MHz, in $CDCl_3$; see **Table 8, Figure 30**

^{13}C NMR : δ ppm, 75 MHz, in $CDCl_3$; see **Table 8, Figure 31**

4.5 Compound DSC-5 (gigantol)

Compound DSC-5 was obtained as a brown amorphous solid (48.0 mg, 0.0010% based on dried weight of whole plant). It was soluble in acetone.

HR-ESI-MS : $[M+Na]^+$ ion at *m/z* 297.1111 ($C_{16}H_{18}O_4Na$); see **Figure 34**

^{1}H NMR : δ ppm, 300 MHz, in acetone-d_6; see **Table 9, Figure 35**

^{13}C NMR : δ ppm, 75 MHz, in acetone-d_6; see **Table 9, Figure 36**

4.6 Compound DSC-6 (batatasin III)

Compound DSC-6 was obtained as a brown amorphous solid (722.0 mg, 0.0150% based on dried weight of whole plant). It was soluble in acetone.

HR-ESI-MS : $[M+Na]^+$ ion at *m/z* 267.1051 ($C_{15}H_{16}O_3Na$); see **Figure 38**

^{1}H NMR : δ ppm, 300 MHz, in acetone-d_6; see **Table 10, Figure 39**

^{13}C NMR : δ ppm, 75 MHz, in acetone-d_6; see **Table 10, Figure 40**

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4.7 Compound DSC-7 (coelonin)

Compound DSC-7 was obtained as a brown amorphous solid (71.0 mg, 0.00148% based on dried weight of whole plant). It was soluble in acetone.

HR-ESI-MS : $[M+Na]^+$ ion at *m/z* 265.0845 ($C_{15}H_{14}O_3Na$); see **Figure 42**

^{1}H NMR : δ ppm, 300 MHz, in acetone-d_6; see **Table 11, Figure 43**

^{13}C NMR : δ ppm, 75 MHz, in acetone-d_6; see **Table 11, Figure 44**

4.8 Compound DSC-8 (aloifol I)

Compound DSC-8 was obtained as a brown amorphous solid (93.0 mg, 0.00194% based on dried weight of whole plant). It was soluble in acetone.

HR-ESI-MS : $[M+Na]^+$ ion at *m/z* 297.1107 ($C_{16}H_{18}O_4Na$); see **Figure 47**

^{1}H NMR : δ ppm, 300 MHz, in acetone-d_6; see **Table 12, Figure 48**

^{13}C NMR : δ ppm, 75 MHz, in acetone-d_6; see **Table 12, Figure 49**

4.9 Compound DSC-9 (lusianthridin)

Compound DSC-9 was obtained as a brown amorphous solid (9.0 mg, 0.00019% based on dried weight of whole plant). It was soluble in acetone.

HR-ESI-MS : $[M+Na]^+$ ion at *m/z* 265.0847 ($C_{15}H_{14}O_3Na$); see **Figure 51**

^{1}H NMR : δ ppm, 300 MHz, in acetone-d_6; see **Table 13, Figure 52**

^{13}C NMR : δ ppm, 75 MHz, in acetone-d_6; see **Table 13, Figure 53**

4.10 Compound DSC-10 (RF-3192C)

Compound DSC-10 was obtained as a yellowish brown amorphous powder (11.0 mg, 0.00023% based on dried weight of whole plant). It was soluble in DMSO.

HR-ESI-MS : $[M+Na]^+$ ion at *m/z* 403.0424 ($C_{20}H_{12}O_8Na$); see **Figure 55**

^{1}H NMR : δ ppm, 500 MHz, in DMSO-d_6; see **Table 14, Figure 56**

^{13}C NMR : δ ppm, 125 MHz, in DMSO-d_6; see **Table 14, Figure 57**

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5. Assay of α-glucosidase inhibitory activity

In this study, the evaluation of α-glucosidase inhibitory activity was based on the spectrophotometric measurement of the *p*-nitrophenol (*p*NP) level. *p*NP was released from hydrolysis of *p*-nitrophenyl-α-D-glucopyranoside (*p*-NPG), a synthetic substrate representing the α-linked terminal glucose of polysaccharide, catalyzed by α-glucosidase enzyme. The experiment was done at microscale *in vitro* in a 96-well plate by the following established protocols (Sun *et al.*, 2014; Inthongkaew *et al.*, 2017).

5.1 Materials and instruments

- *p*-Nitrophenyl-α-D-glucopyranoside (*p*NPG) (Sigma-Aldrich, USA)
- α-Glucosidase enzyme (Sigma-Aldrich, USA)
- Na_2CO_3 (Sigma-Aldrich, USA)
- Acarbose (Sigma-Aldrich, USA)
- Vortex mixer (Vortex-Genie2, Scientific industries)
- Ultrasonic bath (Transsonic 570/H, Elma)
- Incubator (BM500, Memmert)
- Microplate reader (CLARIOstar, BMG LABTECH)

5.2 Determination of α-glucosidase inhibitory activity

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Test sample was dissolved in DMSO-H_2O (1:1) by using vortex mixer and ultrasonic bath. Ten µl of sample solution were mixed with 40 µl of 0.1 U/ml α-glucosidase enzyme in a 96-well plate and then pre-incubated at 37 °C for 10 min. After that, 50 µl of 2 mM *p*NPG (substrate) were added, and the mixture was incubated at 37 °C for 20 min. Finally, the reaction was terminated by adding 100 µl of 1 mM Na_2CO_3 and the absorbance was measured at 405 nm by a microplate reader. The percentage of α-glucosidase enzyme inhibition was calculated by the following formula:

% inhibition of α-glucosidase enzyme = $[(A_{control} - A_{sample}) / A_{control}] \times 100$

$A_{control}$: Absorbance of 50% DMSO in H_2O (negative control).

A_{sample} : Absorbance of test sample or acarbose (positive control)

* The final concentration of DMSO in each well was not more than 5%.

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CHAPTER IV

RESULTS AND DISCUSSION

In this research, the dried and powdered whole plants of *Dendrobium scabrilingue* (4.8 kg) were extracted with MeOH to provide crude MeOH extract (240 g). The MeOH extract was evaluated for α-glucosidase inhibitory activity and showed more than 80 % inhibition at 100 µg/ml. This extract was suspended in water and partitioned with EtOAc and n-butanol, respectively. Only the EtOAc fraction exhibited strong α-glucosidase inhibitory activity with more than 90% inhibition at 100 µg/ml. The other fractions showed no activity (less than 50% inhibition at 100 µg/ml) Thus, the EtOAc part was further separated using several chromatographic techniques to give ten pure compounds including two new compounds (DSC-1 and DSC-2) and eight known compounds. Their structures were characterized using various spectroscopic techniques and then the isolated compounds were tested for α-glucosidase inhibitory activity.

1. Structure determination of isolated compounds

1.1 Structure elucidation of compound DSC-1

Compound DSC-1 was isolated as a brown amorphous solid. The HR-ESI mass spectrum (**Figure 7**) showed a pseudomolecular ion $[M-H]^-$ at *m/z* 283.0973 (calculated for $C_{17}H_{15}O_4$, 283.0970), suggesting the molecular formula $C_{17}H_{16}O_4$. The IR spectrum (**Figure 8**) exhibited absorption bands for hydroxyl (3468 cm^{-1}) and aromatic ring (2924, 1616 cm^{-1}). The UV maximal absorption peaks (**Figure 9**) at 220, 260, 285, 345 and 360 nm suggested the presence of a phenanthrene core structure (Ito *et al.*, 2010a).

The ^{1}H-NMR spectrum (**Figure 10** and **Table 5**) showed the presence of six aromatic proton at δ_H 7.16 (1H, *s*, H-1), 7.21 (1H, *dd*, *J* = 9.5, 3.0 Hz, H-6), 7.34 (1H, *d*, *J* = 3.0 Hz, H-8), 7.56 (1H, *d*, *J* = 9.0 Hz, H-10), 7.60 (1H, *d*, *J* = 9.0 Hz, H-9) and 9.36 (1H, *d*, *J* = 9.5 Hz, H-5), one hydroxyl proton at δ_H 8.28 (1H, *s*, 2-OH), together with

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three methoxy groups at δ_H 3.93 (3H, *s*, 7-OMe), 3.97 (3H, *s*, 4-OMe) and 4.00 (3H, *s*, 3-OMe). The two *ortho*-coupled doublet signals of H-9 and H-10 protons suggested the presence of a phenanthrene nucleus.

The ^{13}C NMR spectrum (**Figure 11** and **Table 5**) displayed seventeen carbon signals, including three methoxyl carbons at δ_C 55.5 (7-OMe), 60.1 (4-OMe) and 61.2 (3-OMe), two equivalent methine carbons at δ_C 109.7 (C-1, C-8). The other thirteen carbons can be classified as four methine carbons at δ_C 117.3 (C-6), 127.3 (C-9), 127.8 (C-10), 128.8 (C-5) and eight aromatic quaternary carbons at δ_C 119.0 (C-4a), 125.0 (C-4b), 130.5 (C-10a), 134.3 (C-8a), 142.9 (C-3), 150.1 (C-2), 152.3 (C-4), 158.1 (C-7), and assigned by HSQC correlations (**Figure 12**). These ^{1}H and ^{13}C NMR data indicated that DSC-1 was a phenanthrene compound.

On ring A, H-8 showed three-bond correlation to C-9 (δ_C 127.3) in HMBC spectrum (**Figure 13**). The ^{1} H-NMR spectrum showed an ABM splitting system consisting of two doublets at δ_H 7.34 (1H, *d*, *J* = 3.0 Hz, H-8) and 9.36 (1H, *d*, *J* = 9.5 Hz, H-5) and a double doublet at 7.21 (1H, *dd*, *J* = 9.5, 3.0 Hz, H-6). The first methoxy group which resonated at δ_H 3.93 (3H, *s*, 7-OMe) could be located at C-7 as evident from the NOESY correlations (**Figure 14**) with H-6 and H-8. For ring B, H-1 (δ_H 7.16, *s*) showed HMBC correlation with C-10 (δ_C 127.8) and a NOESY cross-peak with H-10. The second methoxy group which resonated at δ_H 3.97 (3H, *s*, 4-OMe) was placed at C-4 based on its NOESY correlation with H-5 (δ_H 9.36). The hydroxy group (δ_H 8.28) was located at C-2 (δ_C 150.1) since it exhibited NOESY correlation with H-1 (δ_H 7.16). This placed the third methoxy group (δ_H 4.00) at C-3 (δ_C 149.2), which was confirmed by the absence of NOESY correlation between 3-OMe (δ_H 4.00) and H-1 (δ_H 7.16) and the appearance of HMBC correlation peaks between C-3 (δ_C 149.2) with 3-OMe (δ_H 4.00) and H-1 (δ_H 7.16).

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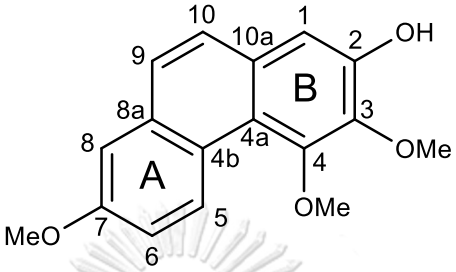
Based on the above spectral data, DSC-1 was characterized as a new phenanthrene derivative, and its structure was similar to that of nudol [**106**]. Its structure was established as 2-hydroxy-3,4,7-trimethoxyphenanthrene and named dendroscabrol A [**303**].



dendroscabrol A [**303**]

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Table 5 ^{1}H NMR 500 MHz and ^{13}C NMR 125 MHz spectral data of compound DSC-1 (in acetone-d_6)

Position	δ_H (mult., *J* in Hz)	δ_C	HMBC (correlation with ^{1}H)
1	7.16 (*s*)	109.7	10, 2-OH
2	-	150.1	1, 2-OH
3	-	142.9	1, 3-OMe, 2-OH
4	-	152.3	4-OMe
4a	-	119.0	1, 5, 10
4b	-	125.0	6, 8, 9
5	9.36 (*d,* 9.5)	128.8	-
6	7.21 (*dd,* 9.5, 3.0)	117.3	8
7	-	158.1	5, 7-OMe
8	7.34 (*d,* 3.0)	109.7	6, 9
8a	-	134.3	5, 10
9	7.60 (*d,* 9.0)	127.3	8
10	7.56 (*d,* 9.0)	127.8	1
10a	-	130.5	9
3-OMe	4.00 (*s*)	61.2	-
4-OMe	3.97 (*s*)	60.1	-
7-OMe	3.93 (*s*)	55.5	-
2-OH	8.28 (*s*)	-	-

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1.2 Structure elucidation of compound DSC-2

Compound DSC-2 was isolated as a brown amorphous solid. The HR-ESI mass spectrum (**Figure 15**) showed a sodium-adduct molecular ion $[M+Na]^+$ at *m/z* 523.1733 (calculated for $C_{30}H_{28}O_7Na$ 523.1732), suggesting the molecular formula $C_{30}H_{28}O_7$. The IR spectrum (**Figure 17**) exhibited absorption bands for hydroxyl (3397 cm^{-1}), aromatic ring (2921, 1646 cm^{-1}), methylene (1468 cm^{-1}) and ether (1246 cm^{-1}). The UV maximal absorption peaks (**Figure 16**) at 210 and 280 nm suggested the presence of a bibenzyl skeleton (Zhang *et al.*, 2007b).

The ^{1}H-NMR spectrum (**Figure 18** and **Table 6**) showed the presence of seven aliphatic protons at δ_H 2.68 (1H, *dd, J* = 13.0, 7.0 Hz, H-8), 2.77 (1H, *m,* H-8), 2.83 (2H, *m,* H-8′), 2.83 (1H, *m,* H-7′), 2.97 (1H, *m,* H-7′) and 4.23 (1H, *dd, J* = 7.0, 5.5 Hz, H-7), eleven aromatic protons at δ_H 6.18-7.10 and two groups of methoxy protons at δ_H 3.77 (3H, *s,* 1′-OMe) and 3.82 (3H, *s,* 1-OMe). The ^{13}C NMR spectrum (**Figure 19** and **Table 6**) displayed thirty carbon signals including three methylene carbons at δ_C 46.4 (C-8), 38.0 (C-8′), 34.4 (C-7′) and a methine carbon at 38.8 (C-7), which were paired with aliphatic protons by HSQC correlations (**Figure 20**). The other carbon signals could be classified as those of two methoxy carbons at δ_C 55.5 (1′-OMe), 61.4 (1-OMe) and twenty four aromatic carbons at δ_C 100.2 (C-2′), 109.8 (C-4), 111.1 (C-6′), 113.8 (C-12, C-12′), 116.2 (C-10′), 117.1 (C-4′), 117.5 (C-10), 117.6 (C-5), 120.4 (C-14′), 121.7 (C-14), 129.3 (C-13), 130.2 (C-13′), 136.8 (C-1), 137.7 (C-2), 139.6 (C-6), 140.1 (C-3), 140.9 (C-9), 141.8 (C-5′), 144.2 (C-9′), 155.0 (C-3′), 157.7 (C-11), 158.4 (C-11′) and 159.7 (C-1′).

Comparison of ^{1}H and ^{13}C NMR spectra of DSC-2 with those of dendrosinen D [**50**], a bisbibenzyl derivative found in *Dendrobium sinense* (Chen et al. 2014), displayed similarity in their structures of rings A, B and B′ except for ring A′. DSC-2 had a proton signal of H-2′ (1H, *d, J* = 2.5 Hz) while dendrosinen D had a hydroxy

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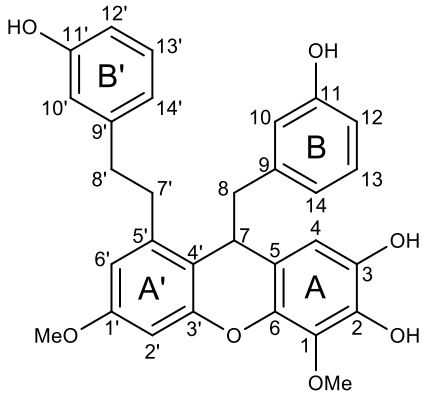
group at this position. H-2′ showed correlations with C-6′ (δ_C 111.1) and C-4′ (δ_C 117.1) in the HMBC spectrum (**Figure 21**). The HMBC correlations from H-7 (δ_H 4.23, 1H, *dd, J* = 7.0, 5.5 Hz) to C-4 (δ_C 109.8), C-5 (δ_C 117.6), C-6 (δ_C 139.6), C-3′ (δ_C 155.0) and C-4′ (δ_C 117.1) demonstrated that rings A and A′ were connected with a methane carbon bridge and an ether linkage. On ring A, the H-4 proton (δ_H 6.25) showed HMBC correlations with C-2 (δ_C 137.7), C-6 (δ_C 139.6) and C-7 (δ_C 38.8), and a NOESY interaction with H-7. On ring A′, the signal of H-6′ (δ_H 6.63, *d,* J = 2.5 Hz) was assigned by its HMBC correlation with C-4′ (δ_C 117.1) and C-7′ (δ_C 34.4). The methoxy group was placed at C-1′ (δ_C 159.7) from NOESY (**Figure 22**) interactions with H-2′ (δ_H 6.49, *d, J* = 2.5 Hz) and H-6′ (δ_H 6.63, *d,* J = 2.5 Hz). On rings B and B′, there were proton signals for two 1,3-disubstituted aromatic rings at δ_H 6.18 (1H, br *d, J* = 7.5 Hz, H-14), 6.21 (1H, *t, J* = 1.5 Hz, H-10), 6.57 (1H, *dd, J* = 7.5, 1.5 Hz, H-12), 6.90 (1H, *t, J* = 7.5 Hz, H-13) for ring B, and 6.66 (1H, *dd, J* = 7.5, 1.5 Hz, H-12′), 6.75 (1H, *br d*, *J* = 7.5 Hz, H-14′), 6.77 (1H, *t, J* = 1.5 Hz, H-10′), and 7.10 (1H, *t, J* = 7.5 Hz, H-13′) for ring B′. The assignments of H-10 and H-14 were obtained from their HMBC correlations with C-8 (δ_C 46.4), whereas H-10′ and H-14′ were assigned based on their HMBC correlations with C-8′ (δ_C 38.0). The *ortho*-coupled aromatic protons of rings B and B′ were also identified by the ^{1}H-^{1}H COSY experiment (**Figure 23**). Since, the carbon at C-7 was chiral, this compound displayed an optical rotation value of +3.7 (*c* 0.1), when measured in methanol at 20°c.

Based on the above spectral data, DSC-2 was characterized as a new bisbibenzyl derivative. It was named dendroscabrol B [**304**].



dendroscabrol B [**304**]

Table 6 ^{1}H NMR 500 MHz and ^{13}C NMR 125 MHz spectral data of compound DSC-2 (in acetone-d_6)

Position	δ_H (mult., *J* in Hz)	δ_C	HMBC (correlation with ^{1}H)
1	-	136.8	1-OMe
2	-	137.7	4
3	-	140.1	4
4	6.25 (*s*)	109.8	7
5	-	117.6	7, 8
6	-	139.6	4, 7
7	4.23 (*dd,* 7.0, 5.5)	38.8	4, 8
8	2.77 (*m*), 2.68 (*dd,* 13.0, 7.0)	46.4	7, 10, 14
9	-	140.9	8, 13
10	6.21 (*t,* 1.5)	117.5	8, 12, 14
11	-	157.7	13
12	6.57 (*dd,* 7.5, 1.5)	113.8	10, 14
13	6.90 (*t,* 7.5)	129.3	-
14	6.18 (*br d*, 7.5)	121.7	8, 10, 12

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Table 6 (continued)

Position	δ_H (mult., *J* in Hz)	δ_C	HMBC (correlation with ^{1}H)
1′	-	159.7	2′, 6′, 1′-OMe
2′	6.49 (*d,* 2.5)	100.2	6′
3′	-	155.0	2′, 7
4′	-	117.1	2′, 7, 8, 7′
5′	-	141.8	7′, 8′
6′	6.63 (*d,* 2.5)	111.1	2′, 7′
7′	2.97 (*m*), 2.83 (*m*)	34.4	6′, 8′
8′	2.83 (*m*)	38.0	7′, 10′, 14′
9′	-	144.2	7′, 13′
10′	6.77 (*t,* 1.5)	116.2	8′, 12′, 14′
11′	-	158.4	13′
12′	6.66 (*dd,* 7.5, 1.5)	113.8	10′, 14′
13′	7.10 (*t,* 7.5)	130.2	-
14′	6.75 (*d,* 7.5)	120.4	10′, 12′
1-OMe	3.82 (*s*)	61.4	-
1′-OMe	3.77 (*s*)	55.5	-

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1.3 Structure determination of compound DSC-3

Compound DSC-3 was isolated as a white amorphous solid. The HR-ESI mass spectrum (**Figure 24**) showed a sodium-adduct molecular ion $[M+Na]^+$ at *m/z* 553.4207 (calculated for $C_{34}H_{58}O_4Na$ 553.4232), suggesting the molecular formula $C_{34}H_{58}O_4$

The ^{1}H and ^{13}C-NMR spectra (**Figures 25, 26** and **Table 7**) showed the characteristic signals of a feruloyl moiety including an ABM splitting system of three aromatic protons at δ_H 6.81 (*d, J*=8.1 Hz, H-5), 7.22 (*dd, J*=8.1, 1.8 Hz, H-6) and 7.83 (*d, J*=1.8 Hz, H-2), two *cis*-olefinic protons (*J*=12.9 Hz) at δ_H 6.85 (H-7) and 5.79 (H-8), one methoxy group at δ_H 3.85 (s), δ_C 55.3 and an oxycarbonyl carbon at δ_C 166.2 (C-9). The aliphatic alcohol part was demonstrated by the signals of an oxygenated-methylene group at δ_H 4.11 (*t, J*=6.6, H-1′), δ_C 63.8 (C-1′), long-chain methylenes at δ_H 1.30-1.64 (H-2′ - H-23′), δ_C 22.4-31.8 (C-2′ - C-23′) and a terminal methyl at δ_H 0.89 (*t, J*=6.6, H-24′), δ_C 13.5 (C-24′). Furthermore, the protonated carbons were paired with their coresponding protons by the HSQC experiment (**Figure 27**).

In the HMBC spectrum (**Figure 28**) the signal of an oxycarbonyl carbon (C-9) displayed cross peaks to those of methine proton (H-7) and methylene protons (H-1′). The methoxy protons showed a correlation peak with C-3, confirming the assignment.

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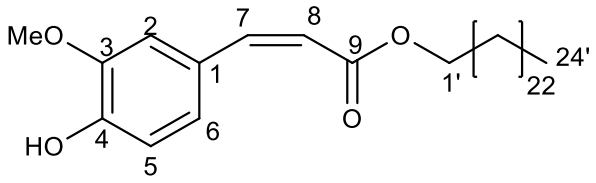
Based on the above spectral data, and a comparison of ^{1}H and ^{13}C-NMR spectra with a previous report (Yin *et al.*, 2008), DSC-3 was identified as (*Z*)-ferulic acid tetracosyl ester [**305**]. This compound has earlier been found in the twigs of *Garuga forrestii* (Burseraceae) (Yin *et al.*, 2008) and the bark of *Zanthoxylum pistaciiflorum* (Rutaceae) (Chen et al., 2004).



(*Z*)-ferulic acid tetracosyl ester [**305**]

Table 7 NMR spectral data of compound DSC-3 (in acetone-d_6) and (*Z*)-ferulic acid tetracosyl ester (in $CDCl_3$)

Position	Compound DSC-3		(*Z*)-ferulic acid tetracosyl ester*	
	δ_H (mult., *J* in Hz)	δ_C	δ_H (mult., *J* in Hz)	δ_C
1	-	126.9	-	127.0
2	7.83 (*d,* 1.8)	114.0	7.06 (*d,* 1.6)	109.3
3	-	148.7	-	146.8
4	-	148.9	-	147.9
5	6.81 (*d,* 8.1)	114.4	6.90 (*d,* 8.1)	114.7
6	7.22 (*dd*, 8.1, 1.8)	125.5	7.03 (*dd*, 8.1, 1.6)	123.0
7	6.85 (*d,* 12.9)	143.4	6.78 (*d,* 13.9)	144.6
8	5.79 (*d,* 12.9)	116.0	5.83 (*d,* 13.9)	115.6
9	-	166.2	-	167.4
1′	4.11 (*t*, 6.6)	63.8	4.19 (*t*)	64.6
2′-23′	1.35-1.65 (*m*)	22.4-31.8	1.33-1.74 (*m*)	22.7-31.9
24′	0.89 (*t*, 6.6)	13.5	0.87 (*t,* 6.3)	14.1
3-OMe	3.85 (*s*)	55.3	3.90 (*s*)	55.9

*Yin *et al.*, 2008

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1.4 Structure determination of compound DSC-4

Compound DSC-4 was isolated as a white amorphous solid. The HR-ESI mass spectrum (**Figure 29**) showed a sodium-adduct molecular ion $[M+Na]^+$ at *m/z* 553.4154 (calculated for $C_{34}H_{58}O_4Na$ 553.4232), suggesting the same molecular formula $C_{34}H_{58}O_4$, as compound DSC-3.

The ^{1}H and ^{13}C-NMR spectra (**Figure 30, 31** and **Table 8**) showed close similarity with DSC-3 spectra, including a feruloyl fragment, three aromatic protons with an ABM splitting system and a methoxy group, as well as the aliphatic alcohol part, with an oxygenated-methylene group, long-chain methylenes and a terminal methyl. The only difference was the coupling constant of the olefinic protons at positions 7 and 8. The ^{1}H-NMR spectrum of DSC-4 displayed the doublet signals (*J* = 15.9 Hz) of H-7 and H-8 at δ_H 7.58 and 6.27, respectively indicating *trans* configuration of the double bond in the feruloyl fragment.

Its NMR assignments were based on the HSQC (**Figure 32**) and HMBC (**Figure 33**) spectra. Therefore, DSC-4 was geometrical isomer of DSC-3 and was thus identified as (*E*)-ferulic acid tetracosyl ester [**252**]. It was previously isolated from the twigs of *Garuga forrestii* (Burseraceae) (Yin *et al.*, 2008) and *Dendrobium tortile* (Limpanit *et al.*, 2016).

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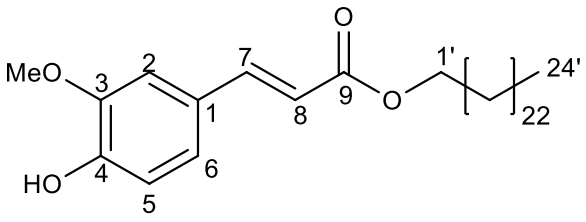

(*E*)-ferulic acid tetracosyl ester [**252**]

Table 8 NMR spectral data of compound DSC-4 and (*E*)-ferulic acid tetracosyl ester (in $CDCl_3$)

Position	Compound DSC-4		(*E*)-ferulic acid tetracosyl ester*	
	δ_H (mult., *J* in Hz)	δ_C	δ_H (mult., *J* in Hz)	δ_C
1	-	127.0	-	127.0
2	7.01 (*br s*)	109.3	7.06 (*d,* 1.6)	109.3
3	-	146.7	-	146.8
4	-	147.9	-	147.9
5	6.89 (*d,* 8.1)	114.7	6.90 (*d,* 8.1)	114.7
6	7.04 (*br d,* 8.1)	123.0	7.03 (*dd,* 1.6, 8.1)	123.0
7	7.58 (*d,* 15.9)	144.6	7.60 (*d,* 16.2)	144.6
8	6.27 (*d,* 15.9)	115.6	6.28 (*d,* 15.9)	115.6
9	-	167.4	-	167.4
1′	4.16 (*t*, 6.9)	64.6	4.19 (*t*)	64.6
2′-23′	1.30-1.70 (*m*)	22.7-31.9	1.33-1.74 (*m*)	22.7-31.9
24′	0.85 (*t*, 6.9)	14.1	0.87 (*t,* 6.3)	14.1
3-OMe	3.89 (*s*)	55.9	3.90 (*s*)	55.9

*Yin *et al.*, 2008

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1.5 Structure determination of compound DSC-5

Compound DSC-5 was isolated as a brown amorphous solid. The HR-ESI mass spectrum (**Figure 34**) showed a sodium-adduct molecular ion $[M+Na]^+$ at *m/z* 297.1111 (calculated for $C_{16}H_{18}O_4Na$ 297.1103), suggesting the molecular formula $C_{16}H_{18}O_4$

The ^{1}H and ^{13}C-NMR spectra (**Figure 35, 36** and **Table 9**) showed the characteristic signals of a bibenzyl derivative including two methylene carbons at δ_C 38.2 (C-α), 37.1 (C-α') and δ_H 2.80 (4H, *m,* H-α, H-α'), two methoxy groups at δ_H 3.79, 3.70 and δ_C 54.5, 55.3 (for 3′-OMe and 5-OMe, respectively), as well as two sets of aromatic protons at δ_H 6.24 (*t, J*=2.1 Hz, H-4), 6.29 (*t, J*=2.1 Hz, H-6) and 6.31 (*t, J*=2.1 Hz, H-2) for ring A (1, 3, 5 trisubstitutions) and ring B (1, 3, 4 trisubstitutions) at δ_H 6.65 (*dd, J*=7.8, 1.8 Hz, H-6′), 6.72 (*d, J*=7.8 Hz, H-5′) and 6.79 (*d, J*=1.8 Hz, H-2′).

The NOESY spectrum (**Figure 37**) displayed cross peaks from 5-OMe protons (δ_H 3.70) to H-6 (δ_H 6.29, *t, J*=2.1 Hz) and H-4 (δ_H 6.24, *t, J*=2.1 Hz). Thus, this methoxy group should be located at C-5 and the other substitution group at C-3 should be hydroxy. For ring B, there was only one NOE correlation peak from 3′-OMe (δ_H 3.79) to H-2′ (δ_H 6.79, *d, J*=1.8 Hz), confirming the position of 3′-OMe.

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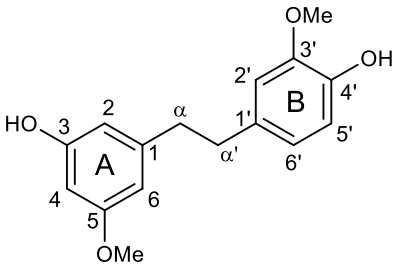
Based on the above spectral data, along with comparison of its ^{1}H and ^{13}C-NMR spectra with a previous report (Chen *et al.*, 2008d), DSC-5 could be identified as gigantol [**16**]. This bibenzyl compound has frequently been found in *Dendrobium* plants, for example, *D. brymerianum* (Klongkumnuankarn *et al.*, 2015), *D. devonianum* (Sun *et al.*, 2014), *D. draconis* (Sritularak *et al.*, 2011a), *D. formosum* (Inthongkaew *et al.*, 2017), *D. loddigesii* (Ito *et al.*, 2010a), *D. officinale* (Zhao *et al.*, 2018), *D. palpebrae* (Kyokong *et al.*, 2018) and *D. venustum* (Sukphan *et al.*, 2014).



Gigantol [**16**]

Table 9 NMR spectral data of compound DSC-5 and gigantol (in acetone-d_6)

Position	Compound DSC-5		gigantol*	
	δ_H (mult., *J* in Hz)	δ_C	δ_H (mult., *J* in Hz)	δ_C
1	-	144.6	-	144.5
2	6.31 (*t,* 2.1)	108.1	6.33 (*dd,* 2.0, 2.0)	107.9
3	-	158.4	-	158.2
4	6.24 (*t,* 2.1)	98.9	6.26 (*dd,* 2.0, 2.0)	98.7
5	-	161.0	-	160.8
6	6.29 (*t,* 2.1)	105.5	6.30 (*dd,* 2.0, 2.0)	105.3
α	2.80 (*m*)	38.2	2.79 (*s*)	37.9
α′	2.80 (*m*)	37.1	2.78 (*s*)	36.9
1′	-	133.3	-	133.1
2′	6.79 (*d,* 1.8)	114.7	6.80 (*d,* 2.0)	114.6
3′	-	147.2	-	147.0
4′	-	144.4	-	144.2
5′	6.72 (*d,* 7.8)	112.1	6.74 (*d,* 8.0)	111.9
6′	6.65 (*dd,* 7.8, 1.8)	120.8	6.66 (*dd,* 8.0, 2.0)	120.6
3′-OMe	3.79 (*s*)	54.5	3.78 (*s*)	54.3
5-OMe	3.70 (*s*)	55.3	3.69 (*s*)	55.2

*Chen *et al.*, 2008d

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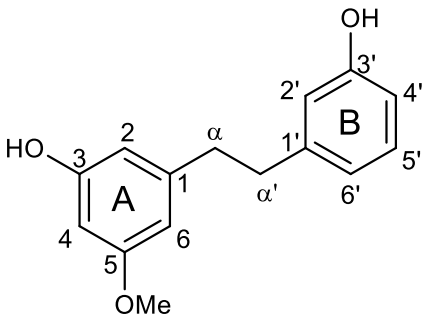
1.6 Structure determination of compound DSC-6

Compound DSC-6 was isolated as a brown amorphous solid. The HR-ESI mass spectrum (**Figure 38**) showed a sodium-adduct molecular ion $[M+Na]^+$ at *m/z* 267.1051 (calculated for $C_{15}H_{16}O_3Na$ 267.0997), suggesting the molecular formula $C_{15}H_{16}O_3$

The ^1H and ^{13}C-NMR spectra (**Figures 39, 40** and **Table 10**) showed the characteristic signals of a bibenzyl derivative including two methylene carbons at δ_C 37.7 (C-α), 37.4 (C-α') and δ_H 2.80 (4H, *m,* H-α, H-α'), a methoxy group at δ_H 3.72 and δ_C 54.4 (5-OMe), as well as three aromatic proton signals at at δ_H 6.26 (*t, J*=2.1 Hz, H-4), 6.33 (*br s*, H-6) and 6.35 (*br s*, H-2). These were very similar to the NMR signals of ring A of compound DSC-5. Furthermore, the NOESY spectrum (**Figure 41**) displayed a cross peak from 5-OMe protons to H-4 (δ_H 6.26, *t,* 2.1) and H-6 (δ_H 6.33, br s). For ring B, there were four aromatic protons at δ_H 6.66 (*dd, J*=7.8, 1.8 Hz, H-4′), 6.70 (*br d, J*=7.8 Hz, H-6′), 6.73 (*br s*, H-2′) and 7.10 (*t, J*=7.8 Hz, H-5′).

Based on the above spectral data, and comparison of the ^1H and ^{13}C-NMR spectra with a previous report (Chen *et al.*, 2008d), DSC-6 could be identified as batatasin III [**5**]. This compound has frequently been reported as a component of *Dendrobium* plants, for instance *D. aphyllum* (Yang *et al.*, 2015a), *D. draconis* (Sritularak *et al.*, 2011a), *D. formosum* (Inthongkaew *et al.*, 2017), *D. infundibulum* (Na Ranong *et al.*, 2018) and *D. venustum* (Sukphan *et al.*, 2014).



batatasin III [**5**]

Table 10 NMR spectral data of compound DSC-6 (in acetone-d_6) and batatasin III (in $CDCl_3$)

Position	Compound DSC-6		batatasin III*	
	δ_H (mult., *J* in Hz)	δ_C	δ_H (mult., *J* in Hz)	δ_C
1	-	144.2	-	144.4
2	6.35 (*br s*)	107.9	6.34 (*dd,* 1.4, 1.4)	108.2
3	-	158.4	-	156.4
4	6.26 (*t,* 2.1)	98.9	6.27 (*dd,* 1.4, 1.4)	99.3
5	-	161.0	-	160.7
6	6.33 (*br s*)	105.4	6.29 (*dd,* 1.4, 1.4)	106.9
α	2.80 (*m*)	37.7	2.80 (*m*)	37.3
α′	2.80 (*m*)	37.4	2.81 (*m*)	36.9
1′	-	143.5	-	143.4
2′	6.73 (*br s*)	115.3	6.64 (*dd,* 2.4, 2.4)	115.4
3′	-	157.4	-	155.4
4′	6.66 (*dd,* 7.8, 2.1)	112.7	6.67 (*dd,* 8.0, 2.4)	112.9
5′	7.10 (*t*, 7.8)	129.2	7.12 (*dd,* 8.0, 8.0)	129.3
6′	6.70 (*br d*, 7.8)	119.5	6.74 (*d,* 8.0)	120.8
5-OMe	3.72 (*s*)	54.4	3.73 (*s*)	55.2

*Chen *et al.*, 2008d)

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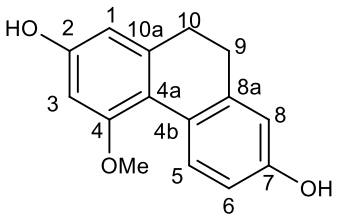
1.7 Structure determination of compound DSC-7

Compound DSC-7 was isolated as a brown amorphous solid. The HR-ESI mass spectrum (**Figure 42**) showed a sodium-adduct molecular ion $[M+Na]^+$ at *m/z* 265.0845 (calculated for $C_{15}H_{14}O_3Na$ 265.0841), suggesting the molecular formula $C_{15}H_{14}O_3$

The ^1H and ^{13}C-NMR spectra (**Figure 43, 44** and **Table 11**) showed signals of four aliphatic protons at δ_H 2.65 (*s,* H-9, H-10) and two methylene carbons at δ_C 29.9 (C-9) nad 30.5 (C-10), suggesting the presence of dihydrophenanthrene nucleus. The five aromatic proton signals can separated into two groups, comprising a pair of doublet signals at δ_H 6.39 (*d*, *J*=2.1 Hz, H-1) and 6.47 (*d*, *J*=2.1 Hz, H-3), as well as signals of an ABM system at δ_H 6.69 (*dd*, *J*=9.0, 2.4 Hz, H-6), 6.71 (*br s,* H-8), and 8.07 (*d*, *J*=9.0 Hz, H-5). There were also signals of a methoxy group at δ_H 3.84 (*s*) and δ_C 54.8 (4-OMe).

The NOESY spectrum (**Figure 45**) confirmed the location of the methoxy group by the cross peak from δ_H 3.84 (4-OMe) to 6.47 (*d*, *J*=2.1 Hz, H-3). Furthermore, confirmation of a dihydrophenanthrene structure was obtained from the correlation peaks from H-1 (δ_H 6.39, *d*, *J*=2.1 Hz) to C-4a (δ_C 115.4) and C-10 (δ_C 30.5), H-3 (δ_H 6.47, *d*, *J*=2.1 Hz) to C-4a, H-5 (δ_H 8.07, *d*, *J*=9.0 Hz) to C-4a and C-8a (δ_C 139.1), as well as from H-8 (δ_H 6.39, *br s*) to C-4b (δ_C 124.8) and C-9 (δ_C 29.9) in HMBC spectrum (**Figure 46**).

Based on the above spectral data, and comparison with a previous report (Rueda *et al.*, 2014), DSC-7 could be identified as coelonin [**73**]. This dihydrophenanthrene derivative have earlier been found in some *Dendrobium* plants, including *D. aphyllum* (Chen *et al.*, 2008e), *D. formosum* (Inthongkaew *et al.*, 2017) and *D. nobile* (Yang *et al.*, 2007).



coelonin [**73**]

Table 11 NMR spectral data of compound DSC-7 (in acetone-d_6) and coelonin (in $CDCl_3$)

Position	Compound DSC-7		coelonin*	
	δ_H (mult., *J* in Hz)	δ_C	δ_H (mult., *J* in Hz)	δ_C
1	6.39 (*d*, 2.1)	107.3	6.26 (*d*, 2.5)	108.4
2	-	156.4	-	155.4
3	6.47 (*d*, 2.1)	98.3	6.30 (*d*, 2.5)	100.1
4	-	157.8	-	158.3
4a	-	115.4	-	114.8
4b	-	124.8	-	125.2
5	8.07 (*d*, 9.0)	129.0	8.13 (*d*, 8.4)	128.6
6	6.69 (*dd*, 9.0, 2.4)	112.6	6.62 (*dd*, 8.3, 2.7)	112.2
7	-	155.1	-	154.8
8	6.71 (*br s*)	114.1	6.61 (*d*, 2.6)	113.8
8a	-	139.1	-	139.8
9	2.65 (*s*)	29.9	2.59 (*s*)	30.1
10	2.65 (*s*)	30.5	2.59 (*s*)	30.8
10a	-	140.4	-	138.7
4-OMe	3.84 (*s*)	54.8	3.67 (*s*)	54.2

*Rueda *et al.*, 2014

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1.8 Structure determination of compound DSC-8

Compound DSC-8 was isolated as a brown amorphous solid. The HR-ESI mass spectrum (**Figure 47**) showed a sodium-adduct molecular ion $[M+Na]^+$ at *m/z* 297.110 7 (calculated for $C_{16}H_{18}O_4Na$ 297.1103), suggesting the molecular formula $C_{16}H_{18}O_4$

The ^1H and ^{13}C-NMR spectra (**Figures 48, 49** and **Table 12**) showed signals of methylene carbons at δ_C 37.7 (C-α), 37.9 (C-α') and δ_H 2.80 (*m,* H-α, H-α') similar to the earlier isolated bibenzyl derivatives, DSC-5 and DSC-6. The signals of aromatic protons integrated for 2 protons at δ_H 6.48 (*s,* H-2, H-6) and methoxy group at δ_H 3.76 (6H, *s,* 3-OMe, 5-OMe) suggested symmetrical substitution on ring A. Corresponding ^{13}C NMR signals were observed at δ_C at 105.9 (C-2, C-6), 147.6 (C-3, C-5) and 55.7 (3-OMe, 5-OMe). These assignments were supported by the NOESY spectrum (**Figure 50**) which presented the cross peaks from methoxy protons at δ_H 3.76 (*s,* 3-OMe, 5-OMe) to aromatic protons at δ_H 6.48 (*s,* H-2, H-6).

For ring B, there were four aromatic proton signals at δ_H 6.65 (*dd, J*=8.1, 2.4 Hz, H-4′), 6.68 (*br d, J*=8.1 Hz, H-6′), 6.70 (*br d*, *J*=2.4 Hz H-2′) and 7.07 (*t, J*=8.1 Hz, H-5′) displaying the same splitting pattern as ring-B of DSC-6 compound, suggesting the identical substituted aromatic ring.

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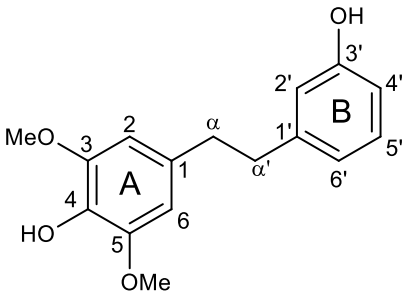
Based on the above spectral data, and comparison of its ^1H and ^{13}C-NMR spectra with a previous report (Juneja *et al.*, 1987), DSC-8 could be identified as aloifol I [**1**]. Several *Dendrobium* plants have also been found to produce this compound, including *D. infundibulum* (Na Ranong *et al.*, 2018), *D. longicornu* (Hu *et al.*, 2008b) and *D. williamsonii* (Yang *et al.*, 2017b).



aloifol I [**1**]

Table 12 NMR spectral data of compound DSC-8 (in acetone-d_6) and aloifol I (in $CDCl_3$)

Position	Compound DSC-8		aloifol I*	
	δ_H (mult., *J* in Hz)	δ_C	δ_H (mult., *J* in Hz)	δ_C
1	-	132.3	-	132.8
2	6.48 (*s*)	105.9	6.27 (*s*)	105.4
3	-	147.6	-	146.8
4	-	134.1	-	132.9
5	-	147.6	-	146.8
6	6.48 (*s*)	105.9	6.27 (*s*)	105.4
α	2.80 (*m*)	37.7	2.75 (*m*)	36.7
α′	2.80 (*m*)	37.9	2.75 (*m*)	37.7
1′	-	143.6	-	143.3
2′	6.70 (*br d,* 2.4)	115.5	6.62 (*dd,* 9.0, 2.5)	115.2
3′	-	157.4	-	155.9
4′	6.65 (*dd,* 8.1, 2.4)	112.7	6.62 (*dd,* 9.0, 2.5)	112.9
5′	7.07 (*t,* 8.1)	129.2	7.03 (*t*, 9.0)	129.2
6′	6.68 (*br d*, 8.1)	119.7	6.62 (*dd,* 9.0, 2.5)	120.5
3-OMe	3.76 (*s*)	55.7	3.72 (*s*)	56.2
5-OMe	3.76 (*s*)	55.7	3.72 (*s*)	56.2

*Juneja *et al.*, 1987

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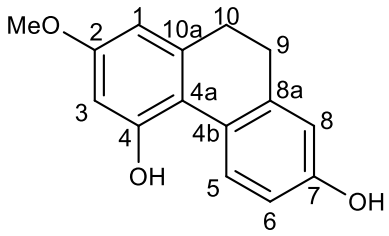
1.9 Structure determination of compound DSC-9

Compound DSC-9 was isolated as a brown amorphous solid. The HR-ESI mass spectrum (**Figure 51**) showed a sodium-adduct molecular ion $[M+Na]^+$ at *m/z* 265.0847 (calculated for $C_{15}H_{14}O_3Na$ 265.0841), suggesting the molecular formula $C_{15}H_{14}O_3$

Comparison of ^1H and ^{13}C-NMR spectra (**Figures 52, 53** and **Table 13**) of DSC-9 with those of DSC-7 showed very similar splitting pattern and almost chemical shift values, including a pair of doublet signals at δ_H 6.37 (*d*, *J*=2.4 Hz, H-1) and 6.44 (*d*, *J*=2.4 Hz, H-3), as well as signals of an ABM system at δ_H 6.70 (*dd*, *J*=9.3, 3.0 Hz, H-6), 6.71 (*br s,* H-8), and 8.23 (*d*, *J*=9.3 Hz, H-5), suggesting they are isomers with crucial difference in the position of the methoxy group at δ_H 3.74 (*s*) and δ_C 54.4 (2-OMe).

The signal of methoxy protons at δ_H 3.74 (*s,* 2-OMe) displayed NOE correlations to δ_H 6.37 (*d*, *J*=2.4 Hz, H-1) and 6.44 (*d*, *J*=2.4 Hz, H-3) in the NOESY spectrum (**Figure 54**), supporting the location on C-2 of the methoxy.

Based on the above spectral data, along with comparison of to a previous report (Guo *et al.*, 2007), DSC-9 could be identified as lusianthridin [**81**]. This dihydrophenanthrene has previously been isolated from other *Dendrobium* plants i.e. *D. brymerianum* (Klongkumnuankarn *et al.*, 2015), *D. formosum* (Inthongkaew *et al.*, 2017), *D. palpebrae* (Kyokong *et al.*, 2018), *D. plicatile* (Yamaki and Honda, 1996) and *D. venustum* (Sukphan *et al.*, 2014).



lusianthridin [**81**]

Table 13 NMR spectral data of compound DSC-9 and lusianthridin (in acetone-d_6)

Position	Compound DSC-9		Lusianthridin*	
	δ_H (mult., *J* in Hz)	δ_C	δ_H (mult., *J* in Hz)	δ_C
1	6.37 (*d,* 2.4)	105.0	6.37 (*d,* 2.6)	106.0
2	-	158.4	-	159.3
3	6.44 (*d,* 2.4)	100.7	6.44 (*d,* 2.6)	101.6
4	-	155.1	-	155.9
4a	-	114.9	-	115.9
4b	-	125.0	-	125.9
5	8.23 (*d,* 9.3)	129.0	8.22 (*d,* 7.5)	129.9
6	6.70 (*dd,* 9.3, 3.0)	112.6	6.68 (*dd,* 7.5, 2.7)	113.5
7	-	155.2	-	156.1
8	6.71 (*br s*)	114.1	6.69 (*m*)	115.0
8a	-	138.9	-	139.8
9	2.67 (*m*)	29.8	2.67 (*m*)	30.8
10	2.67 (*m*)	30.6	2.67 (*m*)	31.5
10a	-	140.5	-	141.4
2-OMe	3.74 (*s*)	54.4	3.74 (*s*)	55.3

*Guo *et al.*, 2007

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1.10 Structure determination of compound DSC-10

DSC-10 was isolated as a yellowish brown amorphous powder. The HR-ESI mass spectrum (**Figure 55**) showed a sodium-adduct molecular ion $[M+Na]^+$ at *m/z* 403.0424 (calculated for $C_{20}H_{12}O_8Na$ 403.0430), suggesting the molecular formula $C_{20}H_{12}O_8$

The ^{1}H-NMR spectrum (**Figure 56** and **Table 14**) displayed the signals of 5 hydroxyl protons at δ_H 13.83 (1H, *s,* 8-OH), 12.54 (1H, *s,* 8′-OH), 11.30 (1H, *s,* 6′-OH), 10.68 (1H, *s,* 6-OH) and 8.11 (1H, *d, J*=1.5 Hz, 3′-OH), 5 aromatic protons at δ_H 7.45 (1H, *d, J*=2.0 Hz, H-5), 7.11 (1H, *d, J*=2.0 Hz, H-5′), 6.47 (1H, *d, J*=2.0 Hz, H-7′), 6.35 (1H, *d, J*=2.0 Hz, H-7) and 5.88 (1H, *s,* H-2) and 2 methylene protons at δ_H 3.22 (1H, *d, J*=16.5 Hz, H-2′) and 3.41 (1H, *dd, J*=16.5, 1.5 Hz, H-2′).

The ^{13}C NMR spectrum (**Figure 57 and Table 14**), supplemented with the HSQC spectrum (**Figure 58**), could indicate signals of two carbonyl carbons at δ_C 198.6 (C-1′) and 189.8 (C-1), five strongly polarized aromatic carbons at δ_C 171.5 (C-3), 164.8 (C-8), 164.7 (C-6′), 163.9 (C-8) and 162.4 (C-6), seven quaternary carbons at δ_C 146.1 (C-4′), 134.7 (C-4a′), 130.4 (C-4a), 121.6 (C-4), 112.1 (C-3′), 109.1 (C-8a′) and 107.5 (C-8a), five methine carbons at δ_C 109.0 (C-5′), 105.0 (C-7′), 104.9 (C-5), 104.7 (C-7) and 98.8 (C-2), and one methylene carbon at 49.2 (C-2′).

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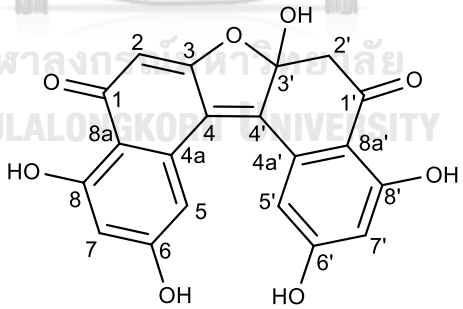
From the HMBC spectrum (**Figure 59**), Two napthalenone units could be inferred. The first one showed cross peaks from C-1 carbonyl carbon (δ_C 189.8) to H-2 (δ_H 5.88, 1H, *s*), from H-2 to C-3 (δ_C 171.5), C-4 (δ_C 121.6) and C-8a (δ_C 107.5), from C-8a to 8-OH (δ_H 13.83, 1H, *s*), H-7 (δ_H 6.35, 1H, *d, J*=2.0 Hz) and H-5 (δ_H 7.45, 1H, *d, J*=2.0 Hz) from H-5 to C-7 (δ_C 104.7), C-4 (δ_C 121.6) and C-6 (δ_C 162.4), from 6-OH to C-6, C5 (δ_C 104.9) and C-7. The other fragment signals showed cross peaks from C-1′ carbonyl carbon (δ_C 198.6) to H-2′ (δ_H 3.22, 1H, *d, J*=16.5 Hz and 3.41, 1H, *dd, J*=16.5, 1.5 Hz) methylene protons, from H-2′ to C-3′ (δ_C 112.1), C-4′ (δ_C 146.1) and

C-8a′ (δ_C 109.1), from C-8a′ to 8-OH′ (δ_H 12.54, 1H, *s*), H-7′ (δ_H 6.47, 1H, *d, J*=2.0 Hz) and H-5′ (δ_H 7.11, 1H, *d, J*=2.0 Hz), from H-5′ to C-7′ (δ_C 105.0), C-4′ (δ_C 146.1) and C-6′ (δ_C 164.7), from 6′-OH (δ_H 11.30, 1H, *s*) to C-6′, C-5′ (δ_C 109.0) and C-7′. A NOESY experiment (**Figure 60**) confirmed the positions of the hydroxy groups. NOE correlations of 8-OH with H-7, 6-OH with H-5 and H-7 were observed for the first fragment. Similar correlations were found between 8′-OH and H-7′, 6′-OH and H-5′/ H-7′. Furthermore, NOESY dipolar coupling between H-5 and H-5′, along with between 6-OH and 6′-OH supported the alignment of these two napthalenone fragments.

Based on the above spectral data, and comparison of its ^{1}H and ^{13}C-NMR spectra with a previous report (Yoshida *et al.*, 1993), DSC-10 could be identified as RF–3192C [**306**]. This compound was firstly reported from *Chaetomella circinoseta* strain RF3192, a soil fungus (Yoshida *et al.*, 1993), and then from *Graphostroma* sp., a deep-sea fungus (Niu *et al.*, 2018). This is the first report of a dinapthalenone from *Dendrobium* plant. It might have been produced by an endophytic fungus since there was a previous report of an endophytic fungus *Aspergillus* sp. in mangrove that produced a few dinaphthalenone derivatives (Xiao *et al.*, 2015).



RF–3192C [**306**]

Table 14 NMR spectral data of compound DSC-10 and RF–3192C (in DMSO-d_6)

Position	Compound DSC-10		RF–3192C*		DSC-10 HMBC
	δ_H (mult., *J* in Hz)	δ_C	δ_H (mult., *J* in Hz)	δ_C	(correlation with ^{1}H)
1	-	189.8	-	190.3	2
2	5.88 (*s*)	98.8	5.84 (*s*)	99.3	-
3	-	171.5	-	172.0	2
4	-	121.6	-	122.1	5
4a	-	130.4	-	130.8	5
5	7.45 (*d,* 2.0)	104.9	7.51 (*d,* 2.0)	105.3	7, 6-OH
6	-	162.4	-	162.9	5, 7, 6-OH
7	6.35 (*d,* 2.0)	104.7	6.36 (*d,* 2.2)	105.1	5, 8-OH
8	-	163.9	-	164.4	7, 8-OH
8a	-	107.5	-	107.9	2, 5, 7, 8-OH
1′	-	198.6	-	199.1	2′
2′	3.22 (*d,* 16.5)	49.2	3.23 (*d,* 16.8)	49.6	3′-OH
	3.41 (*dd,* 16.5, 1.5)		3.41 (*d,* 16.8)		
3′	-	112.1	-	112.6	2′
4′	-	146.1	-	146.6	5′
4a′	-	134.7	-	135.1	5′
5′	7.11 (*d,* 2.0)	109.0	7.10 (*d,* 2.0)	109.4	7′, 6′-OH
6′	-	164.7	-	165.2	5′, 7′, 6′-OH
7′	6.47 (*d,* 2.0)	105.0	6.46 (*d,* 2.2)	105.3	5′, 8′-OH
8′	-	164.8	-	165.2	7′, 8′-OH
8a′	-	109.1		109.5	2′, 5′, 7′, 8′-OH

*Yoshida *et al.*, 1993

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Table 14 (continued)

Position	Compound DSC-10		RF–3192C*		DSC-10 HMBC
	δ_H (mult., *J* in Hz)	δ_C	δ_H (mult., *J* in Hz)	δ_C	(correlation with ^1H)
6-OH	10.68 (*s*)	-	10.71 (*s*)	-	-
8-OH	13.83 (*s*)	-	13.86 (*s*)	-	-
3′-OH	8.11 (*d,* 1.5)	-	-	-	-
6′-OH	11.30 (*s*)	-	11.34 (*s*)	-	-
8′-OH	12.54 (*s*)	-	12.57 (*s*)	-	-

*Yoshida *et al.*, 1993

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2. Evaluation of α-glucosidase inhibitory activity

In the preliminary screening for α-glucosidase inhibitory activity, the crude methanol extract of *Dendrobium scabrilingue* demonstrated 84.87% inhibition at 100 µg/mL. The extract was then partitioned to give EtOAc, butanol and aqueous extracts, all of which were examined for α-glucosidase inhibitory activity. Only the EtOAc extract still showed strong inhibition of α-glucosidase enzyme (96.55% inhibition). Thus, the EtOAc extract was selected for further study (**Table 15)**.

Table 15 Screening test for α-glucosidase inhibitory activity of *D. scabrilingue* extracts (at 100 µg/mL)

Extracts	%Inhibition of α-Glucosidase
Methanol	84.87
EtOAc	96.55
n-Butanol	24.87
aqueous	14.35
Acarbose at 1 mg/mL (Positive control)	67.50

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The EtOAc extract was separated by vacuum liquid chromatography to afford 8 fractions (A-H). Each fraction was tested for α-glucosidase inhibitory activity at 100 µg/mL. Most of them displayed potent inhibitory activity, except fractions A and H, as shown in **Table 16.**

Table 16 Screening test for α-glucosidase inhibitory activity of fractions obtained from EtOAc extract (at 100 µg/mL)

Extracts	%Inhibition of α-Glucosidase
A	32.98
B	91.11
C	99.45
D	99.53
E	90.52
F	98.39
G	95.26
H	35.24
Acarbose at 1 mg/mL (Positive control)	67.50

Fractions C, D and F were chosen for investigation of their active compounds based on their most potent inhibitory activity. Each fraction afforded several pure compounds as described in **Chapter III.** The isolated compounds were evaluated for α-glucosidase inhibitory activity, and their IC_{50} values were calculated, shown in **Table 17.**

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Table 17 IC_{50} values for α-glucosidase inhibitory activity of the isolated compounds from *Dendrobium scabrilingue*

Compounds	IC_{50} (μM)
Dendroscabrol A (DSC-1)	96.2±12.0
Dendroscabrol B (DSC-2)	9.4±0.7
(*Z*)-Ferulic acid tetracosyl ester (DSC-3)	NA
(*E*)-Ferulic acid tetracosyl ester (DSC-4)	NA
Gigantol (DSC-5)	103.1±0.8
Batatasin III (DSC-6)	NA
Coelonin (DSC-7)	131.4±6.6
Aloifol I (DSC-8)	NA
Lusianthridin (DSC-9)	112.9±5.3
RF-3192C (DSC-10)	7.3±0.4
Acarbose (Positive control)	1,076.4±30.6

The results demonstrated the most potent inhibitors as RF-3192C (IC_{50} = 7.3±0.4 μM) and dendroscabrol B (IC_{50} = 9.4±0.7 μM). The other strong inhibitors include dendroscabrol A (IC_{50} = 96.2±12.0 μM), gigantol (IC_{50} = 103.1±0.8 μM), lusianthridin (IC_{50} = 112.9±5.3 μM) and coelonin (IC_{50} = 131.4±6.6 μM) as compared with acarbose, the positive control. It is interesting to note that the bisbibenzyl derivative dendroscabrol B has about 11- to 14- fold stronger inhibitory activity than the bibenzyl monomers, such as gigantol, coelonin and lusianthridin.

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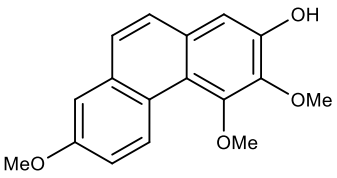
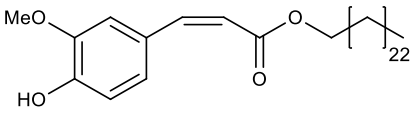
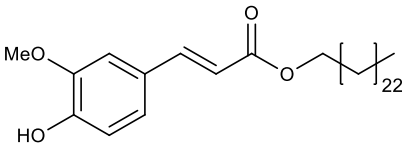


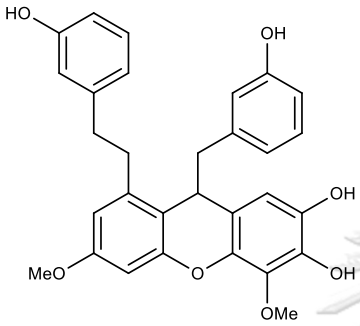
dendroscabrol A [**303**]



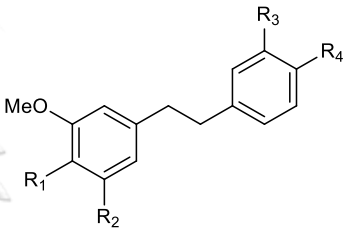
(*Z*)-ferulic acid tetracosyl ester [**305**]



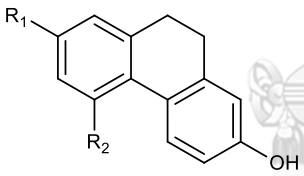
(*E*)-ferulic acid tetracosyl ester [**252**]



Dendroscabrol B [**304**]



	R1	R2	R3	R4
gigantol [**16**]	H	OH	OMe	OH
batatasin III [**5**]	H	OH	OH	H
aloifol I [**1**]	OH	OMe	OH	H



	R1	R2
coelonin [**73**]	OH	OMe
lusiantridin [**81**]	OMe	OH

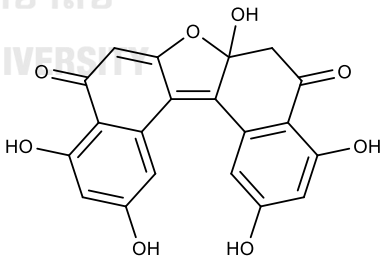


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Figure 6 Structures of isolated compounds from *Dendrobium scabrilingue*

CHAPTER V

CONCLUSION

In this study, ten pure compounds were isolated from the EtOAc fraction of the methanol extract of *Dendrobium scabrilingue* whole plant (Orchidaceae) by several chromatographic methods. Two of them were characterized as new compounds: a phenanthrene derivative named dendroscabrol A and a bisbibenzyl named dendroscabrol B. The rest were known compounds and were identified as (*Z*)-ferulic acid tetracosyl ester, (*E*)-ferulic acid tetracosyl ester, gigantol, batatasin III, coelonin, aloifol I, lusianthridin and RF-3192C (suspected endophytic fungus metabolite). All of the isolated compounds were examined for α-glucosidase inhibitory activity. The relatively large molecules, RF-3192C and dendroscabrol B, exhibited the most potent inhibitory activity against α-glucosidase enzyme. Although, bibenzyl and phenanthrene derivatives, including dendroscabrol A, gigantol, lusianthridin and coelonin, were also active, they were less potent than the bisbibenzyl dendroscabrol B by 11- to 14-fold. All active compounds had stronger inhibitory activity than acarbose, a widely used hypoglycemic drug. This result might provide potential lead compounds from a natural source for the development of new anti-diabetic drugs.

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APPENDIX

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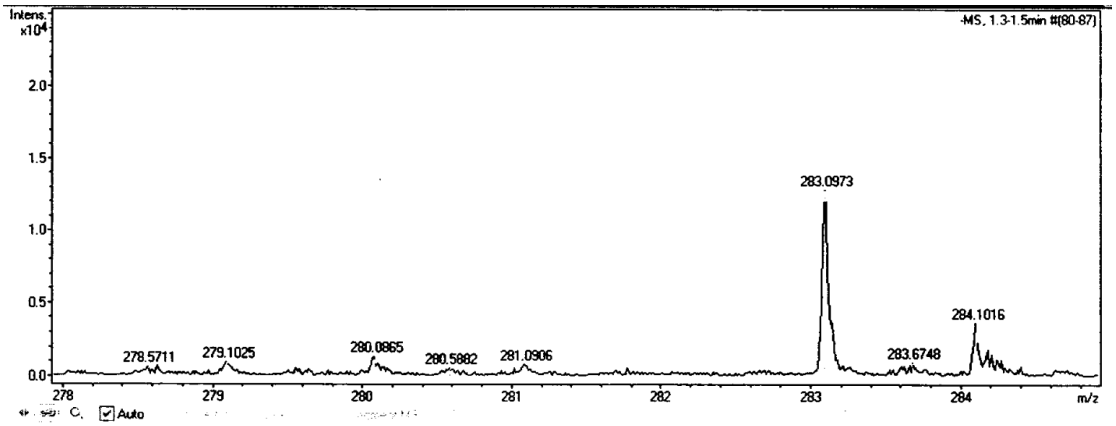


Figure 7 Mass spectrum of compound DSC-1

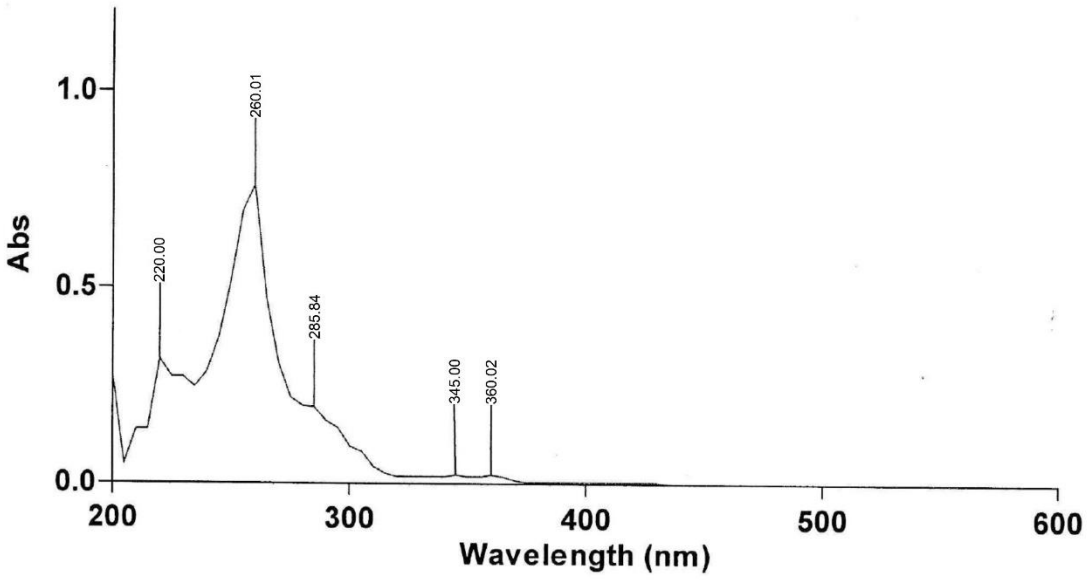


Figure 8 UV spectrum of compound DSC-1

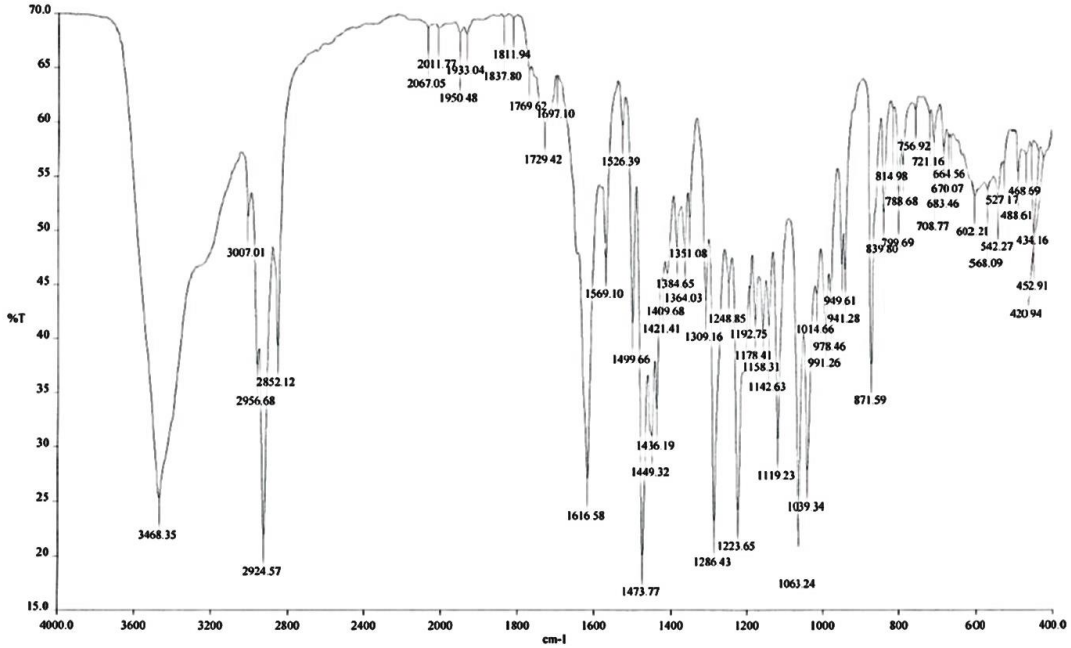


Figure 9 IR spectrum of compound DSC-1

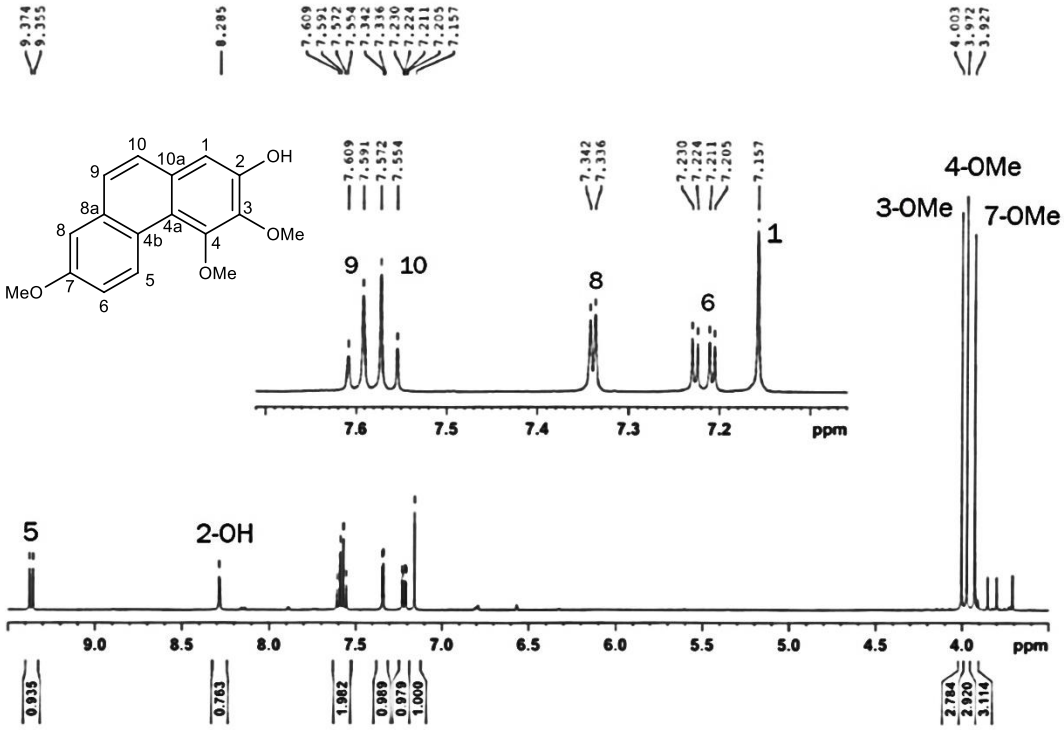


Figure 10 ^{1}H-NMR (500 MHz) spectrum of compound DSC-1 (in acetone-d_6)

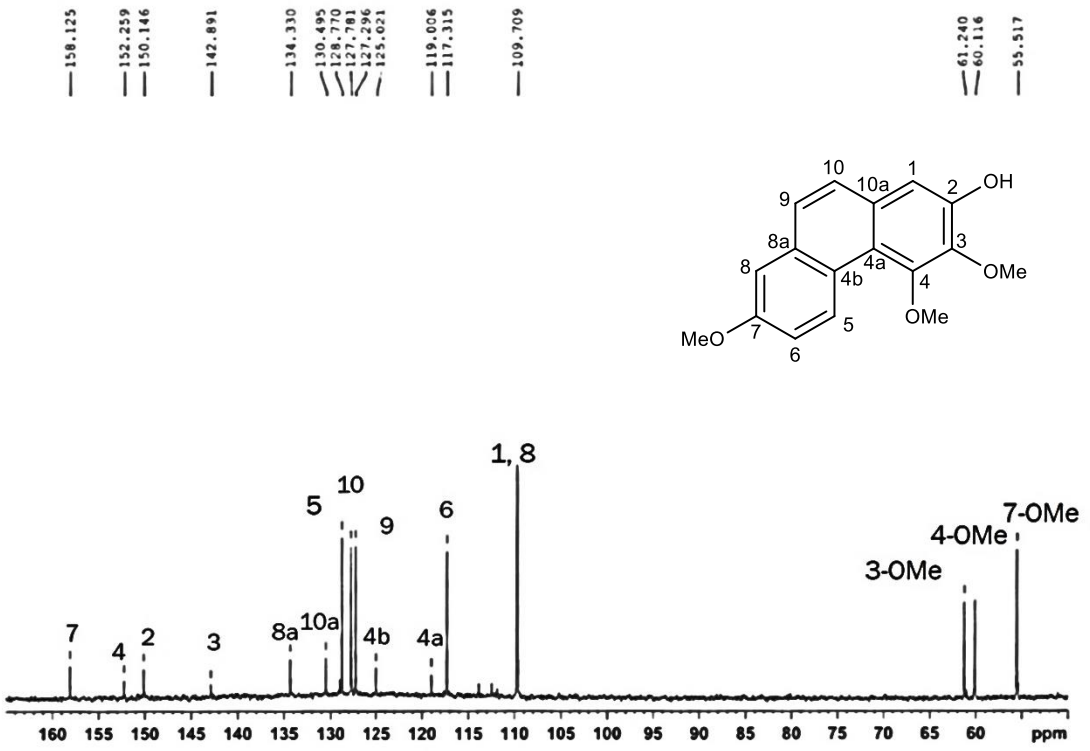


Figure 11 ^{13}C-NMR (125 MHz) spectrum of compound DSC-1 (in acetone-d_6)

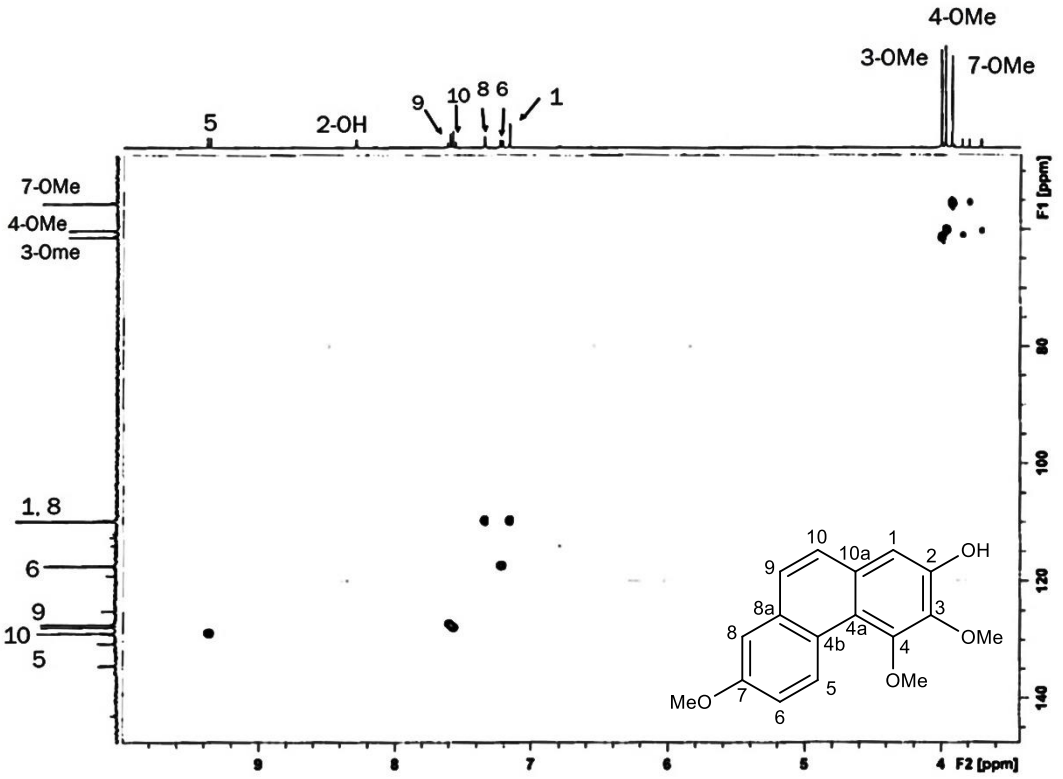


Figure 12 HSQC spectrum of compound DSC-1 (in acetone-d_6)

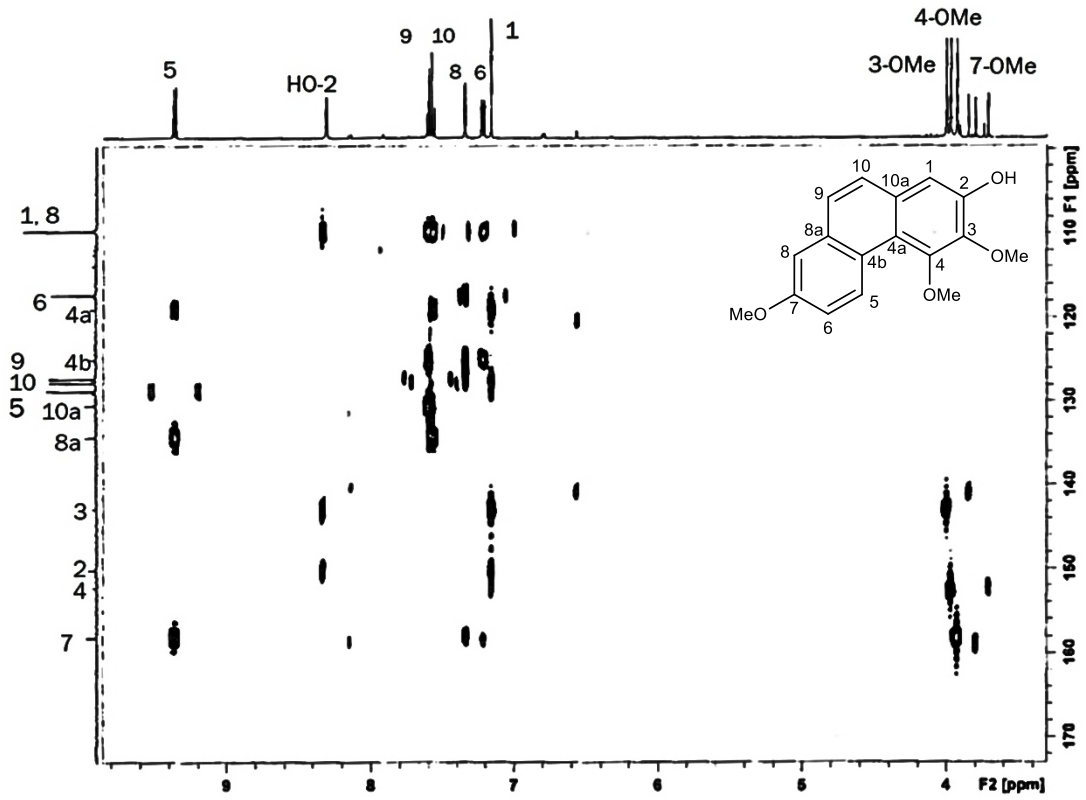


Figure 13 HMBC spectrum of compound DSC-1 (in acetone-d_6)

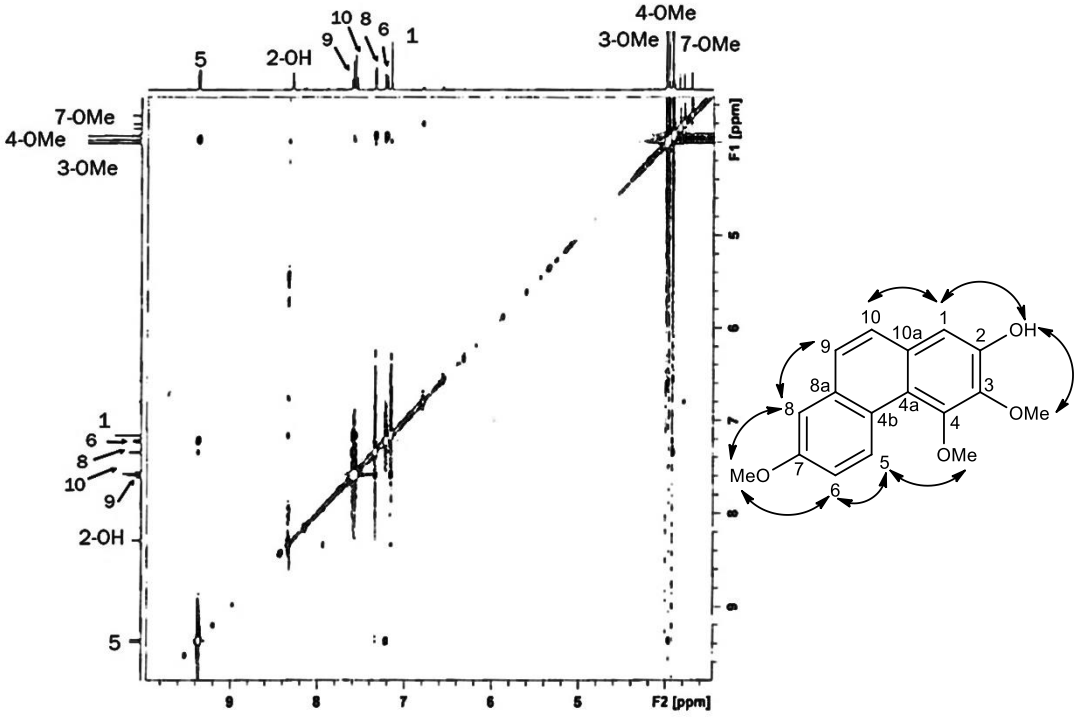


Figure 14 NOESY spectrum of compound DSC-1 (in acetone-d_6)

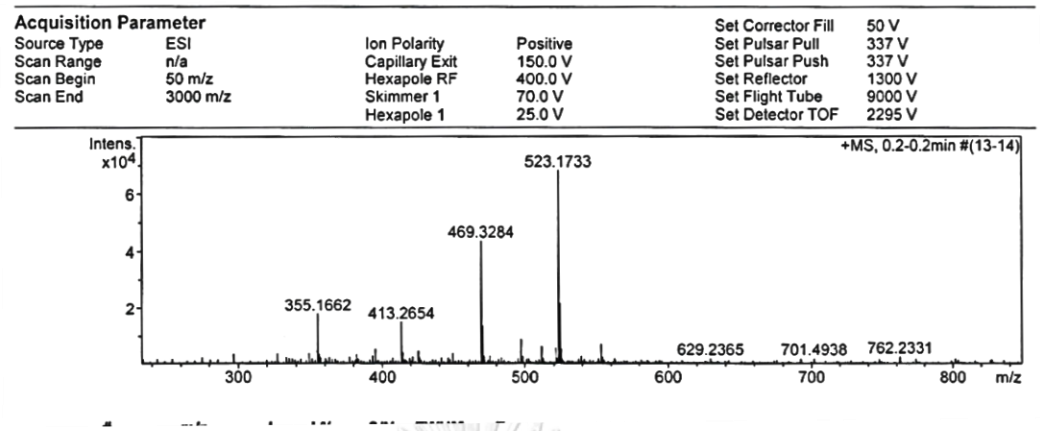


Figure 15 Mass spectrum of compound DSC-2

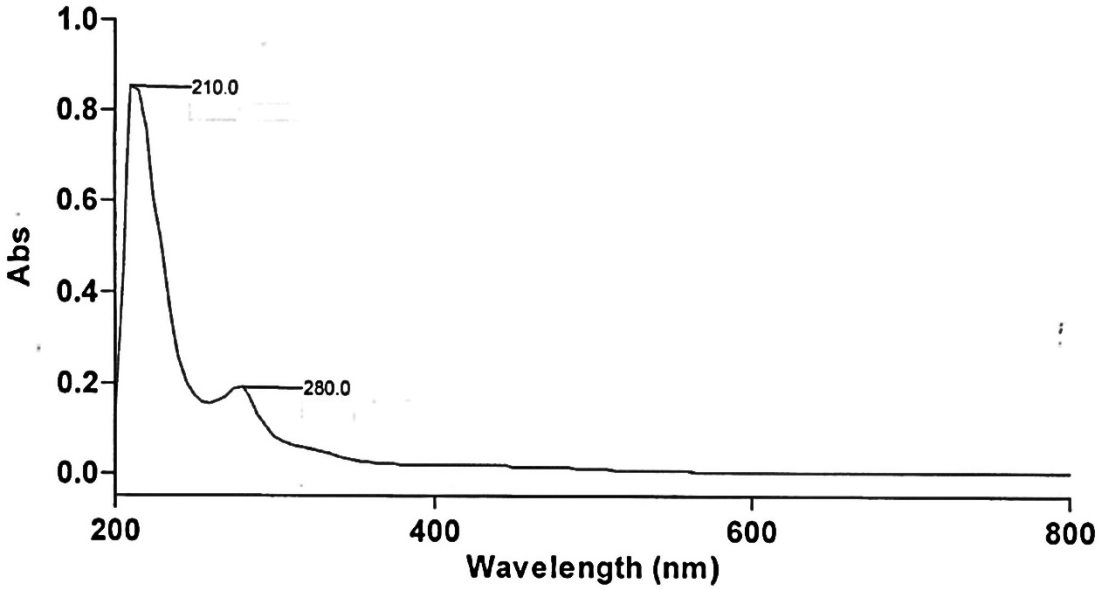


Figure 16 UV spectrum of compound DSC-2

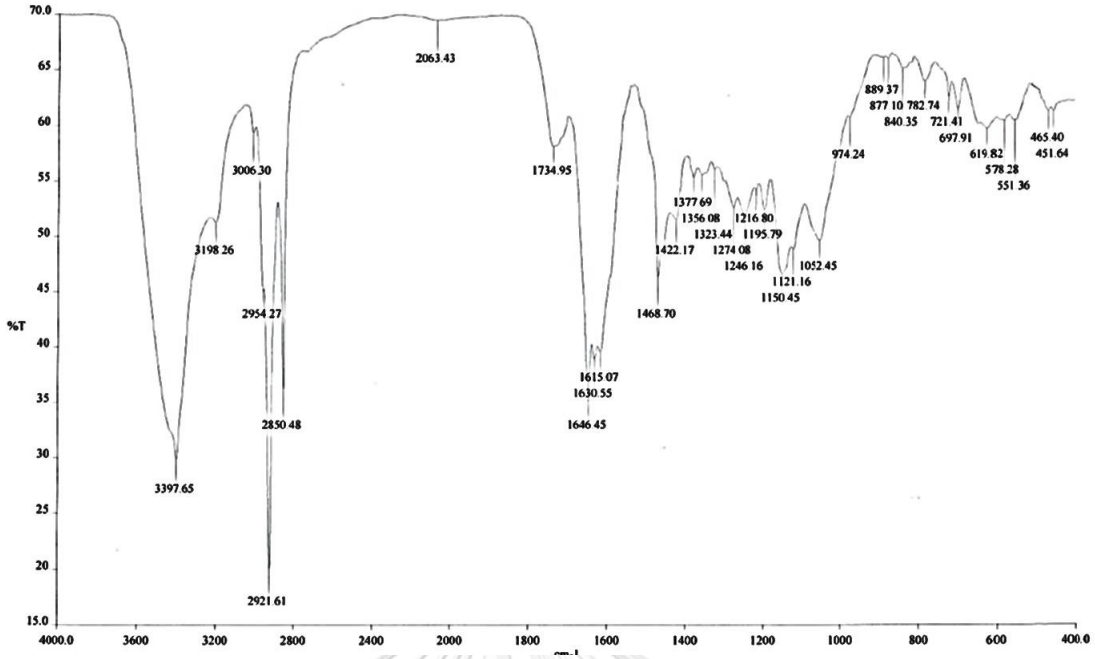


Figure 17 IR spectrum of compound DSC-2

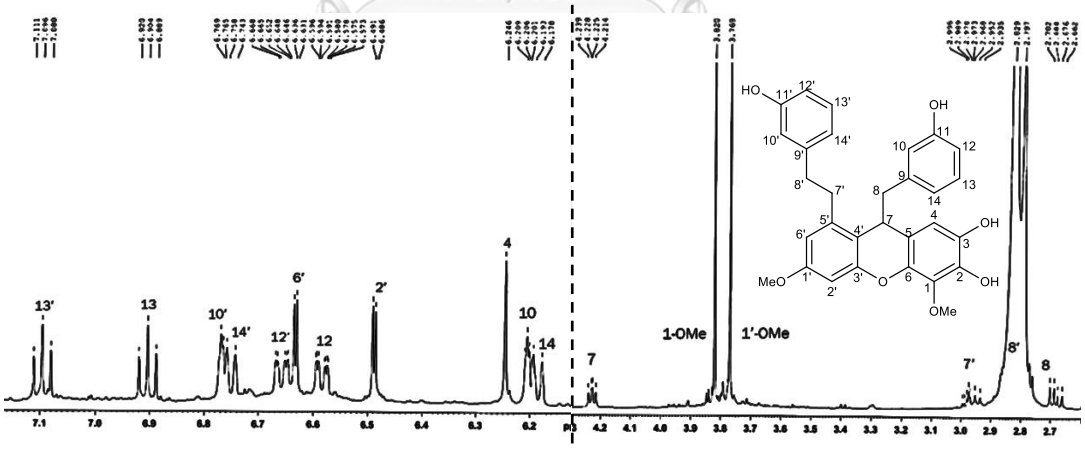


Figure 18 ^{1}H-NMR (500 MHz) spectrum of compound DSC-2 (in acetone-d_6)

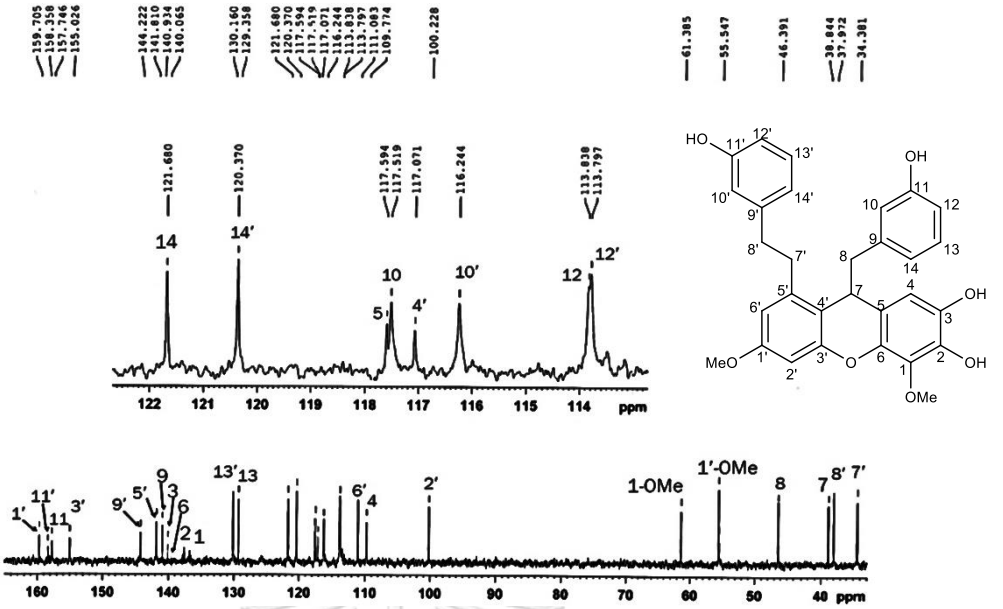


Figure 19 ^{13}C-NMR (125 MHz) spectrum of compound DSC-2 (in acetone-d_6)

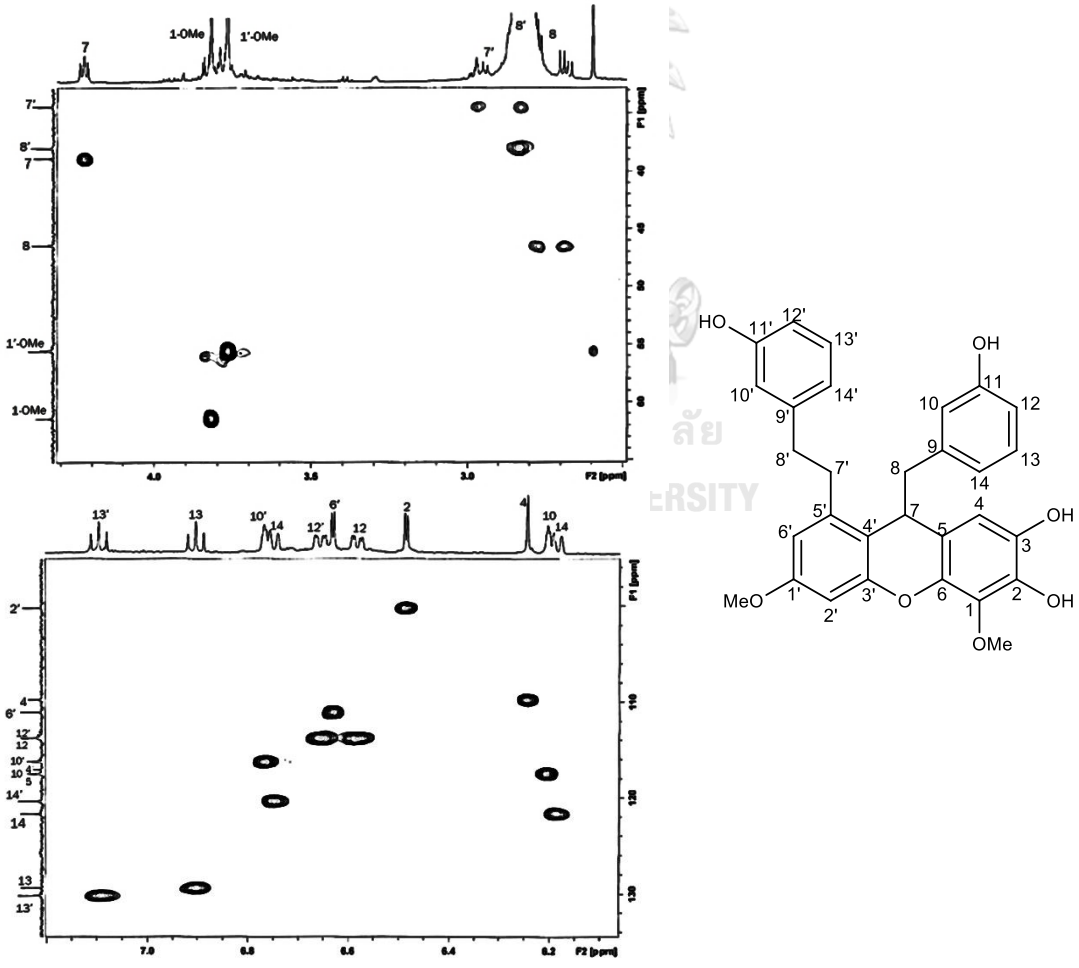


Figure 20 HSQC spectrum of compound DSC-2 (in acetone-d_6)

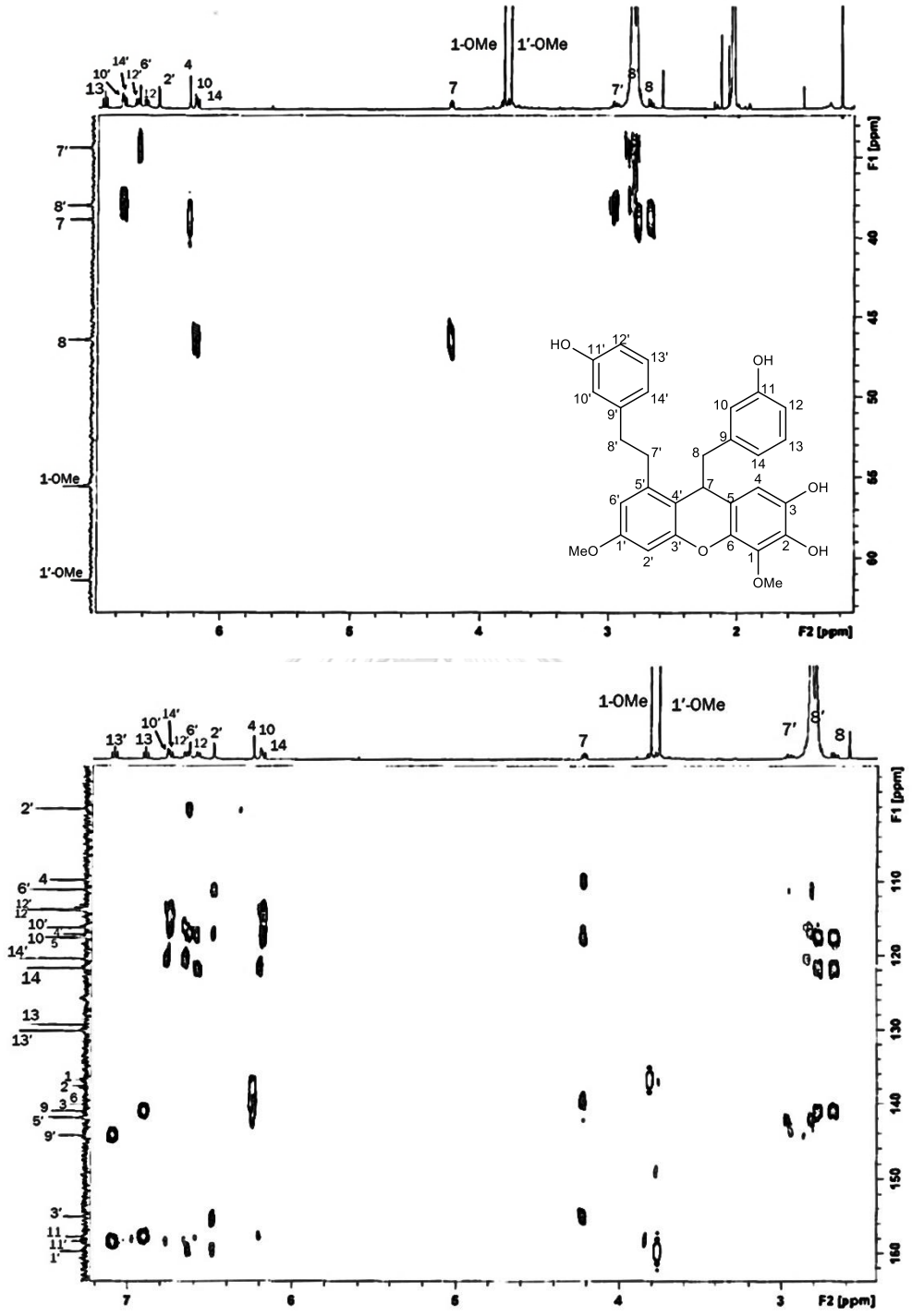


Figure 21 HMBC spectrum of compound DSC-2 (in acetone-d_6)

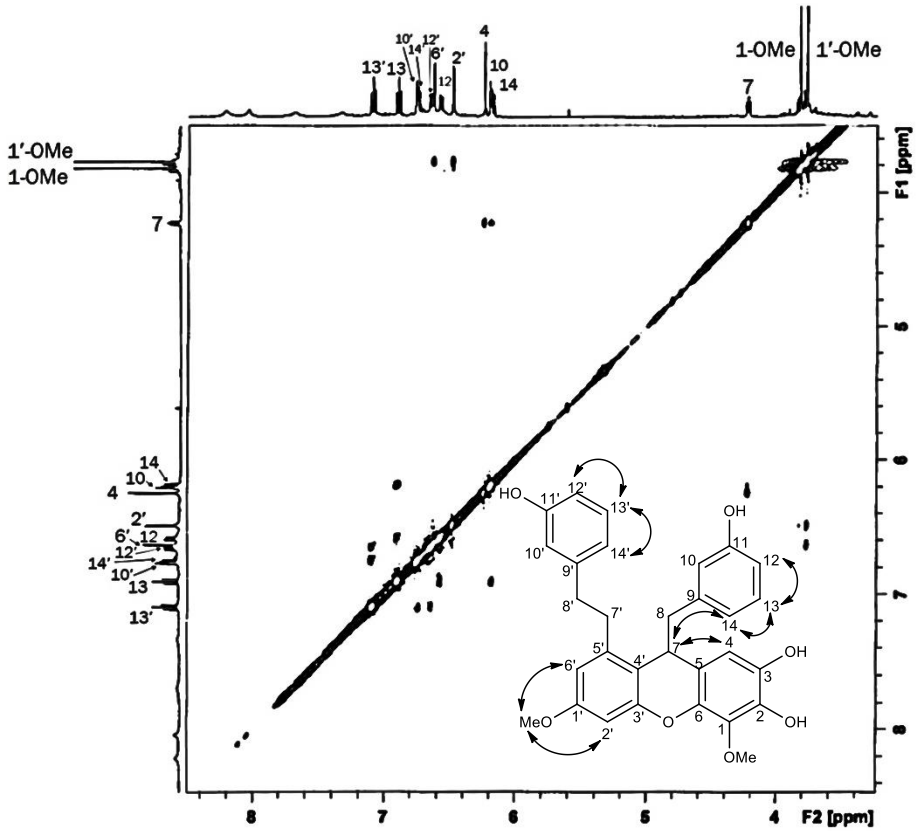


Figure 22 NOESY spectrum of compound DSC-2 (in acetone-d_6)

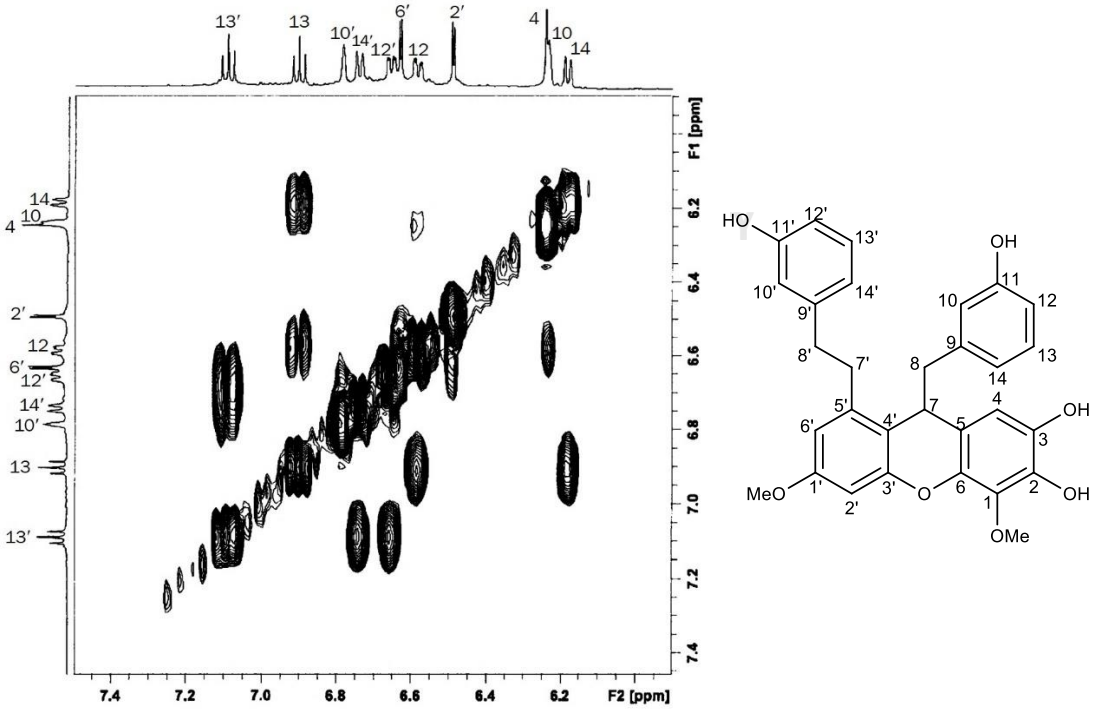


Figure 23 COSY spectrum of compound DSC-2 (in acetone-d_6)

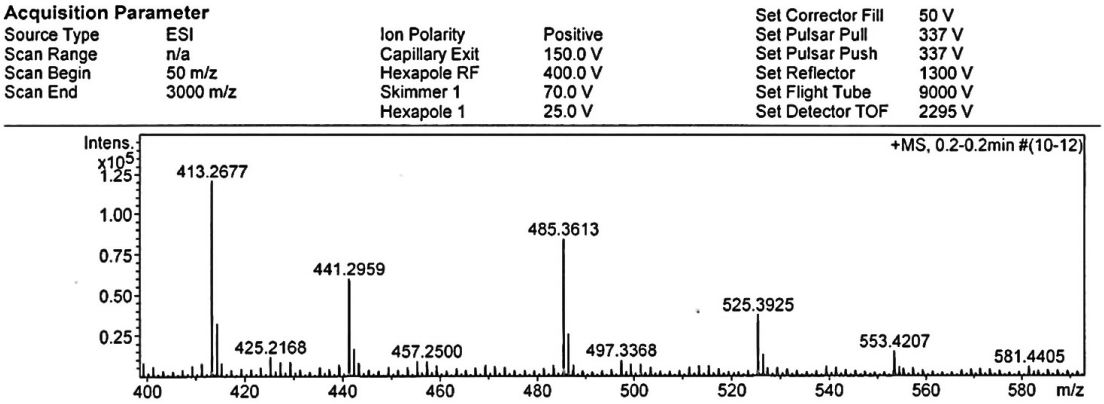
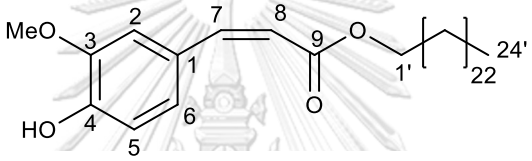


Figure 24 Mass spectrum of compound DSC-3



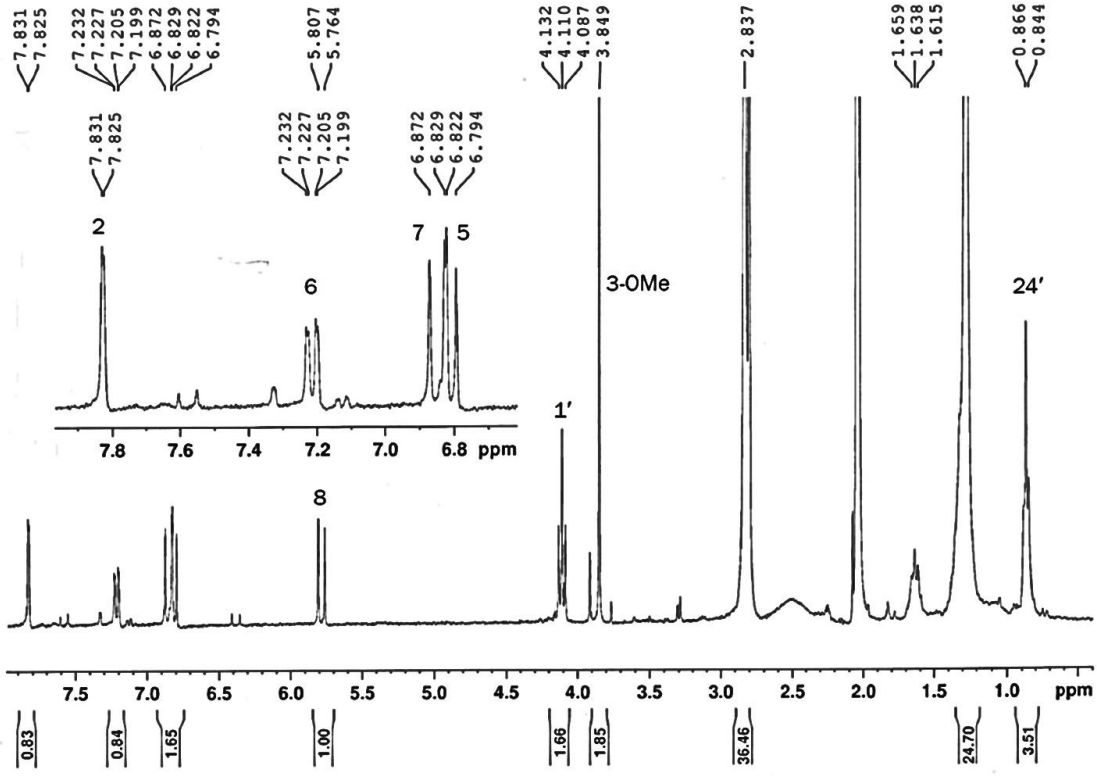


Figure 25 ^{1}H-NMR (300 MHz) spectrum of compound DSC-3 (in acetone-d_6)

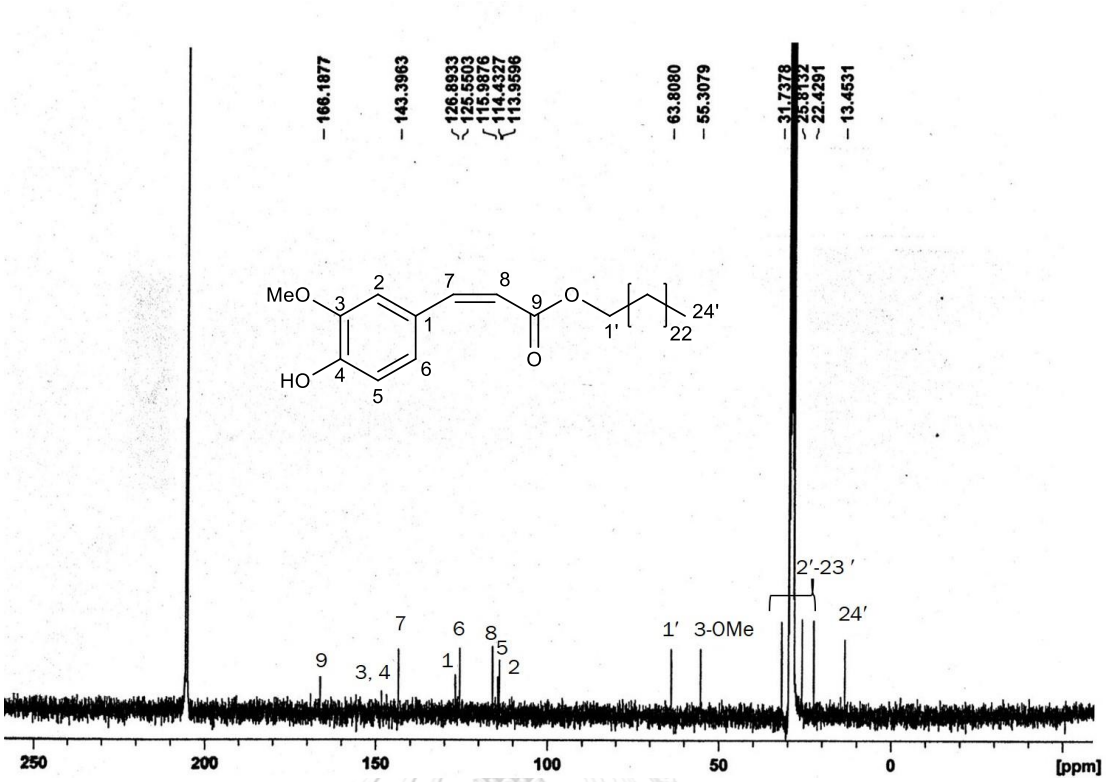


Figure 26 ^{13}C-NMR (75 MHz) spectrum of compound DSC-3 (in acetone-d_6)

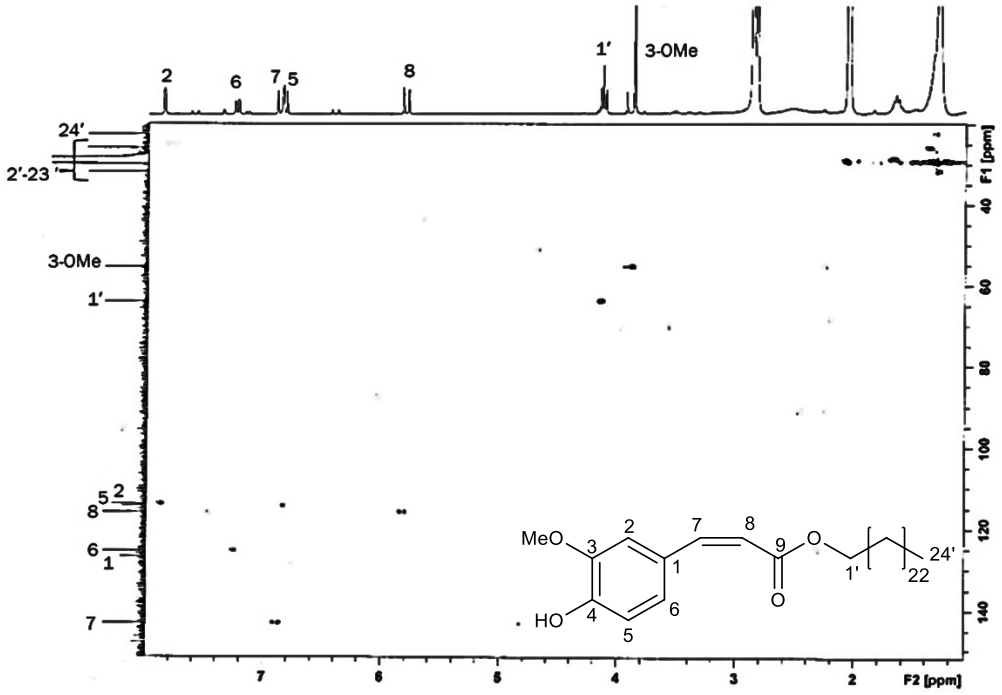


Figure 27 HSQC spectrum of compound DSC-3 (in acetone-d_6)

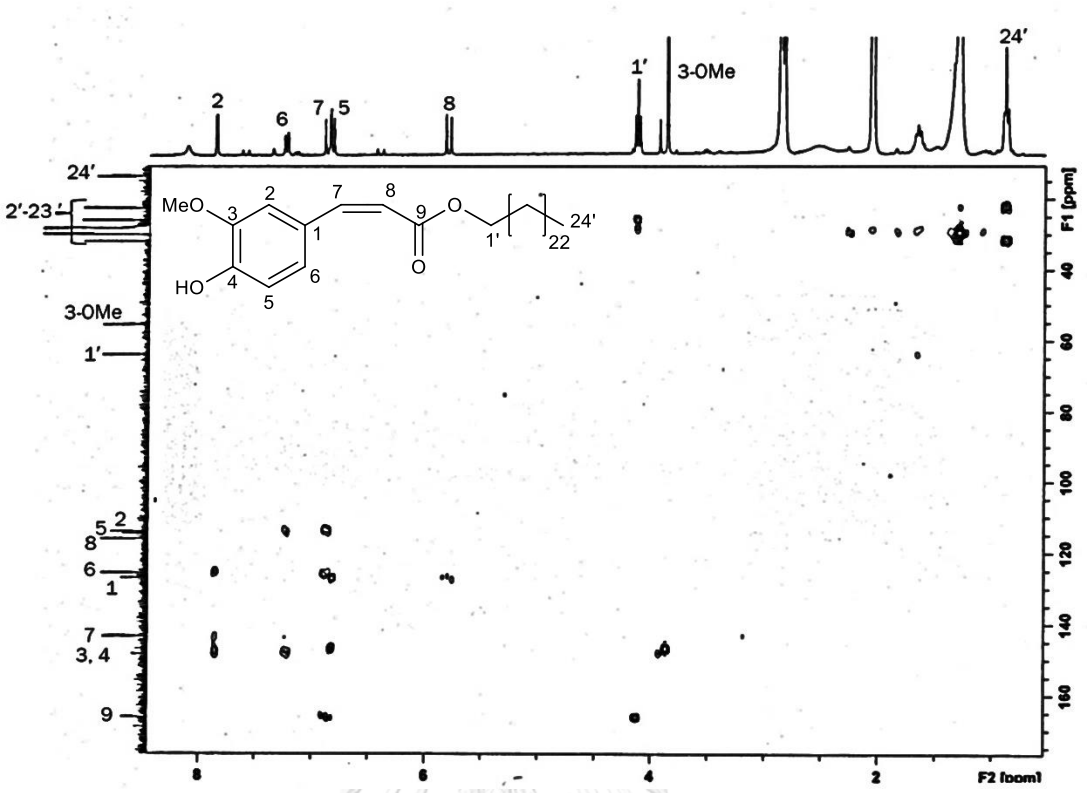


Figure 28 HMBC spectrum of compound DSC-3 (in acetone-d_6)

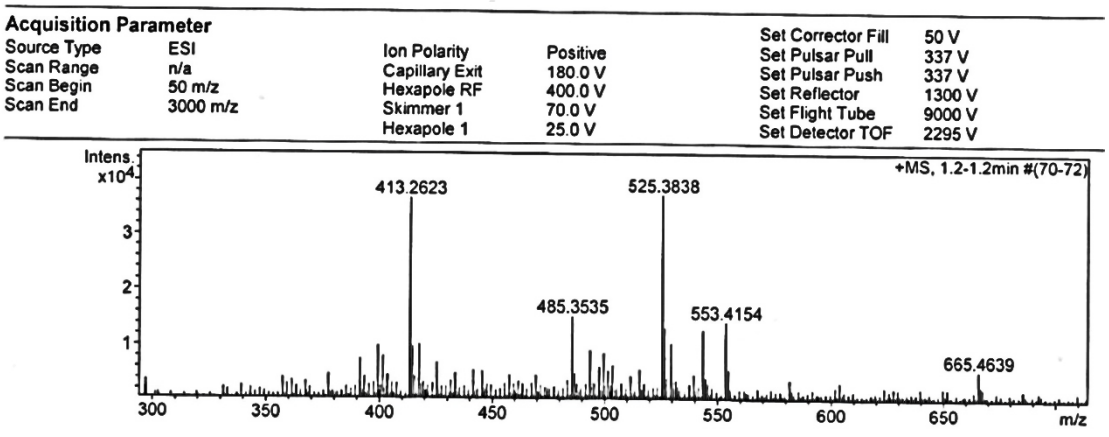


Figure 29 Mass spectrum of compound DSC-4

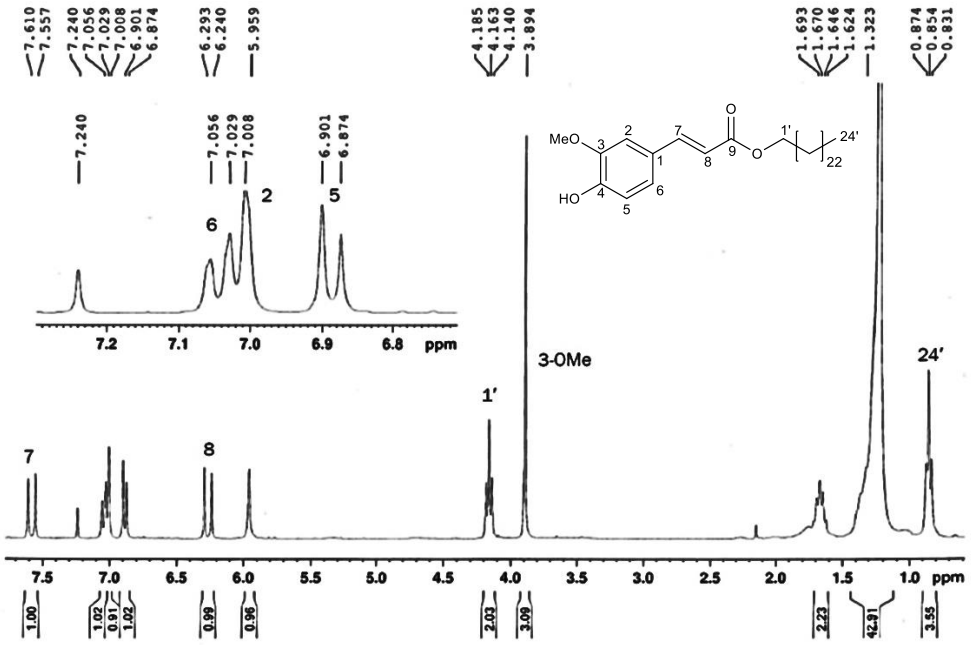


Figure 30 ^{1}H-NMR (300 MHz) spectrum of compound DSC-4 (in $CDCl_3$)

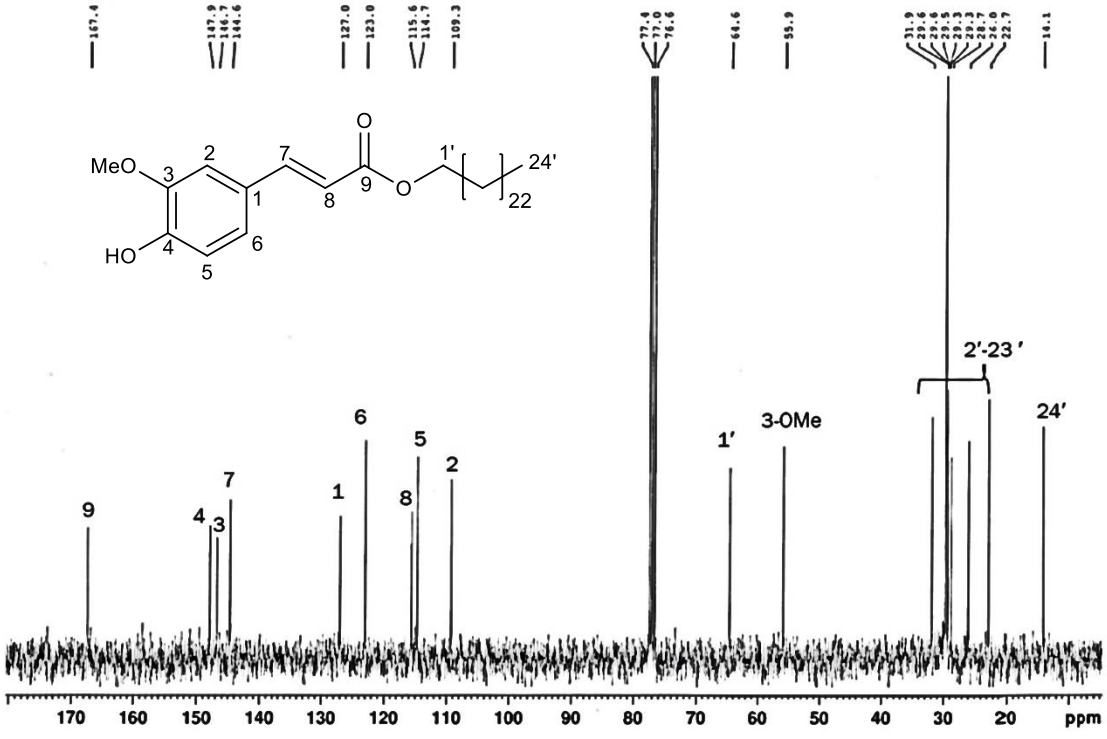


Figure 31 ^{13}C-NMR (75 MHz) spectrum of compound DSC-4 (in $CDCl_3$)

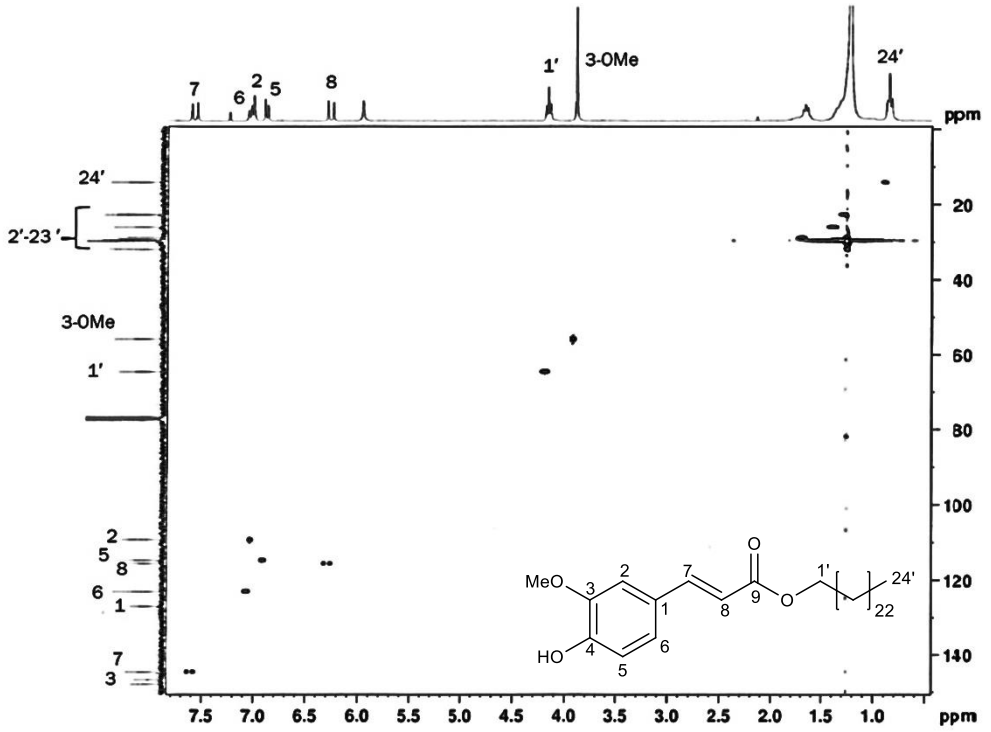


Figure 32 HSQC spectrum of compound DSC-4 (in $CDCl_3$)

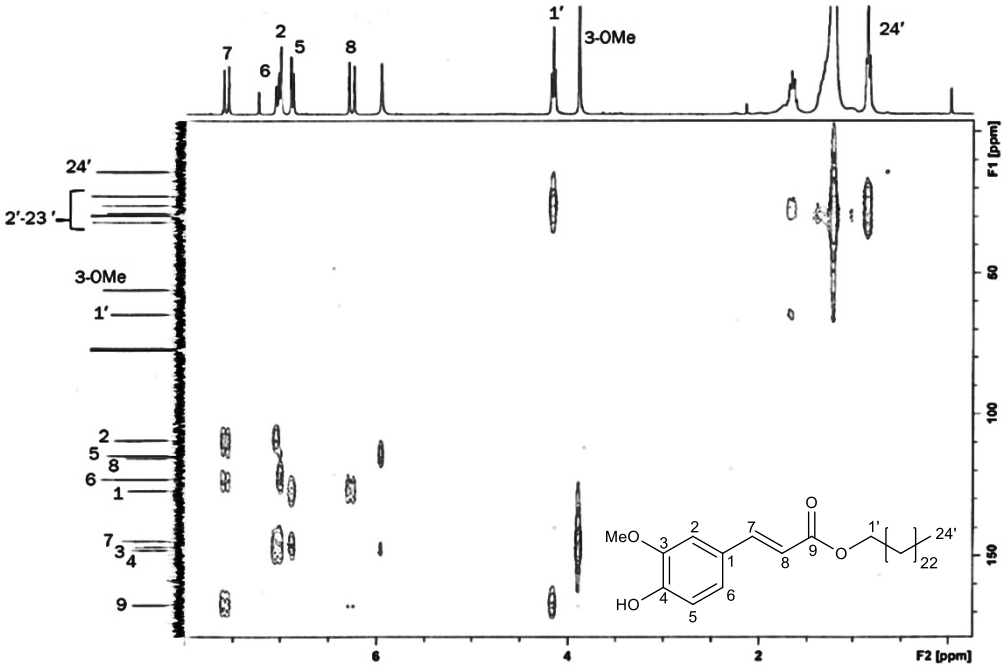


Figure 33 HMBC spectrum of compound DSC-4 (in $CDCl_3$)

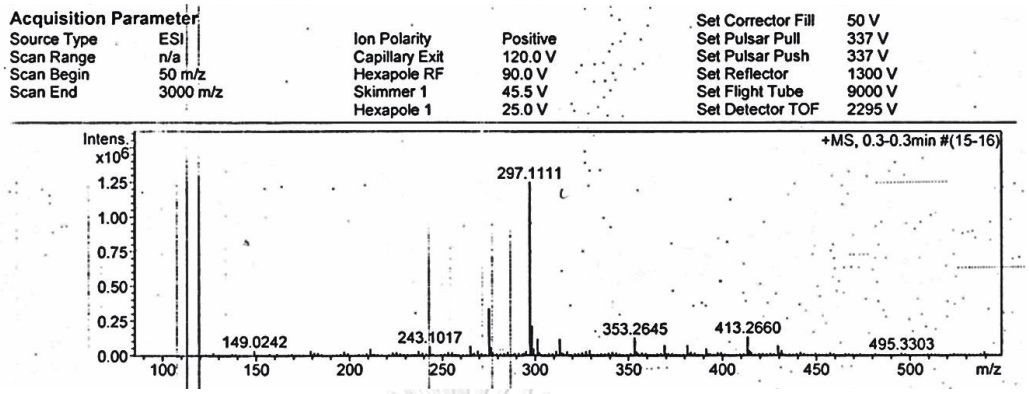


Figure 34 Mass spectrum of compound DSC-5

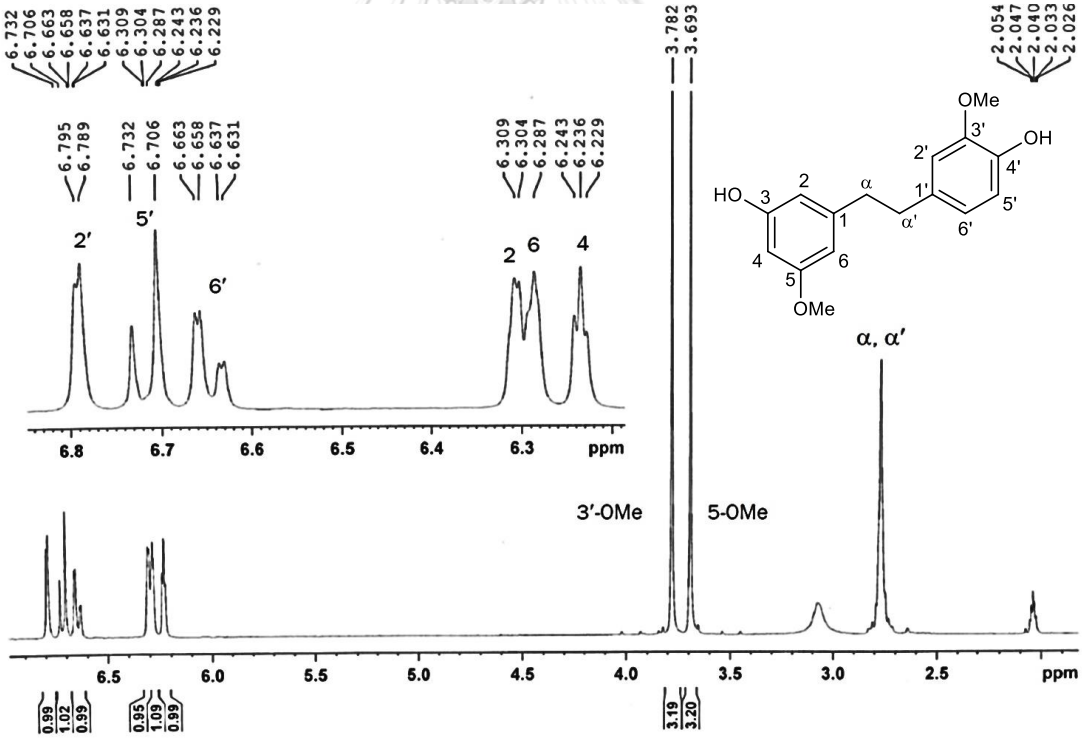


Figure 35 ^{1}H-NMR (300 MHz) spectrum of compound DSC-5 (in acetone-d_6)

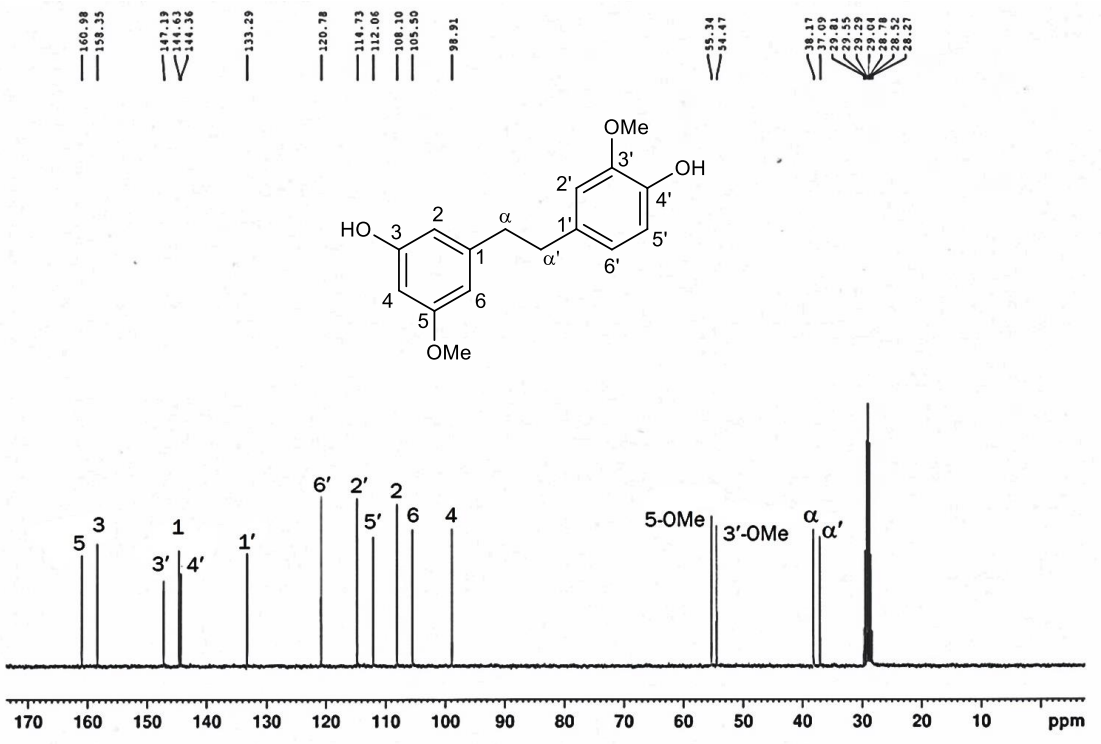


Figure 36 ^{13}C-NMR (75 MHz) spectrum of compound DSC-5 (in acetone-d_6)

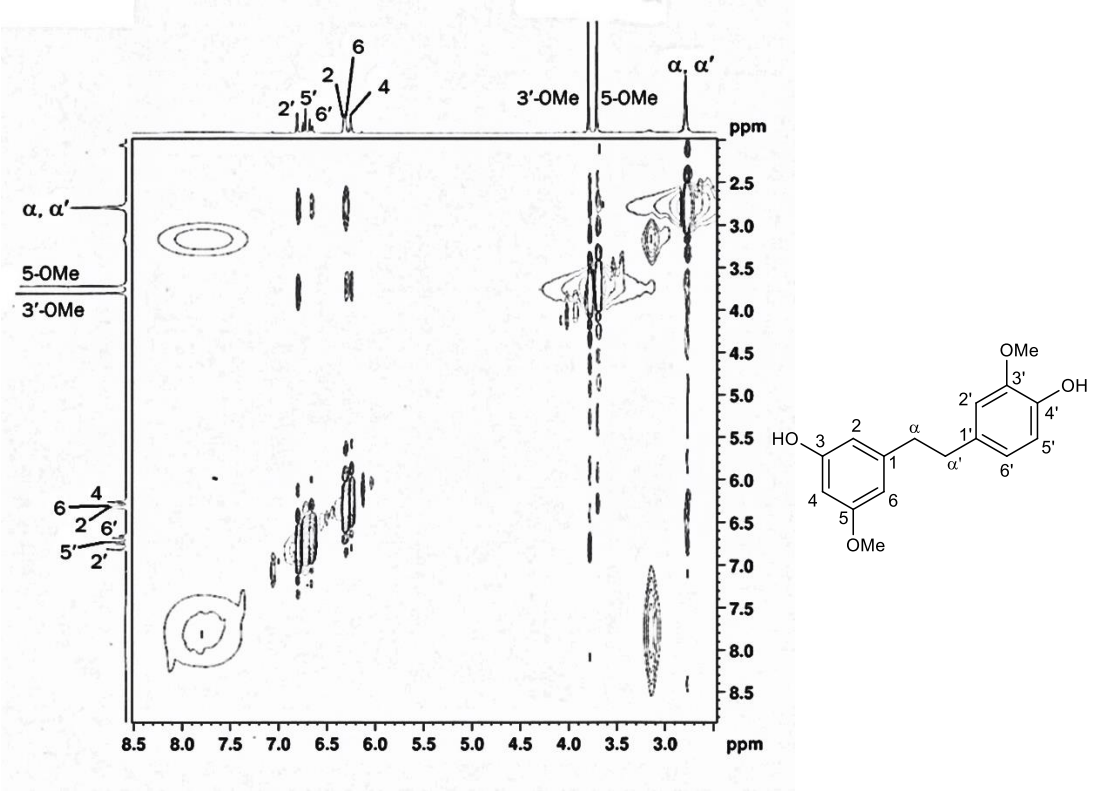


Figure 37 NOESY spectrum of compound DSC-5 (in acetone-d_6)

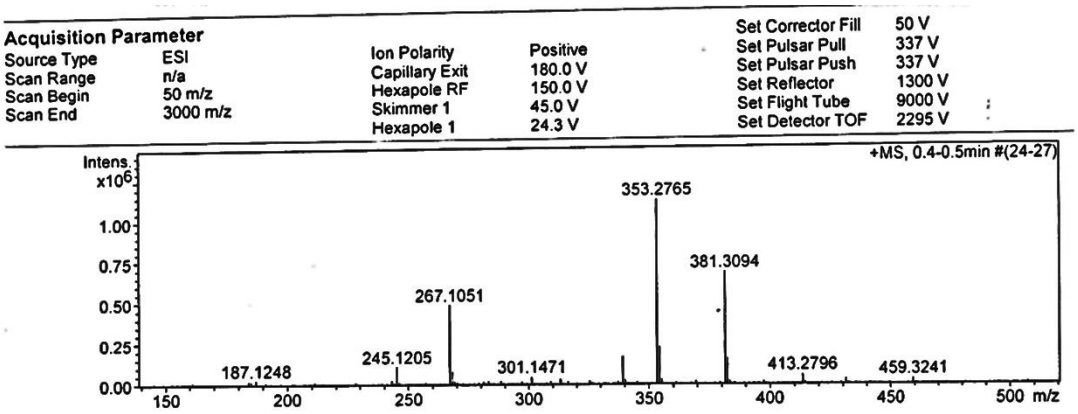


Figure 38 Mass spectrum of compound DSC-6

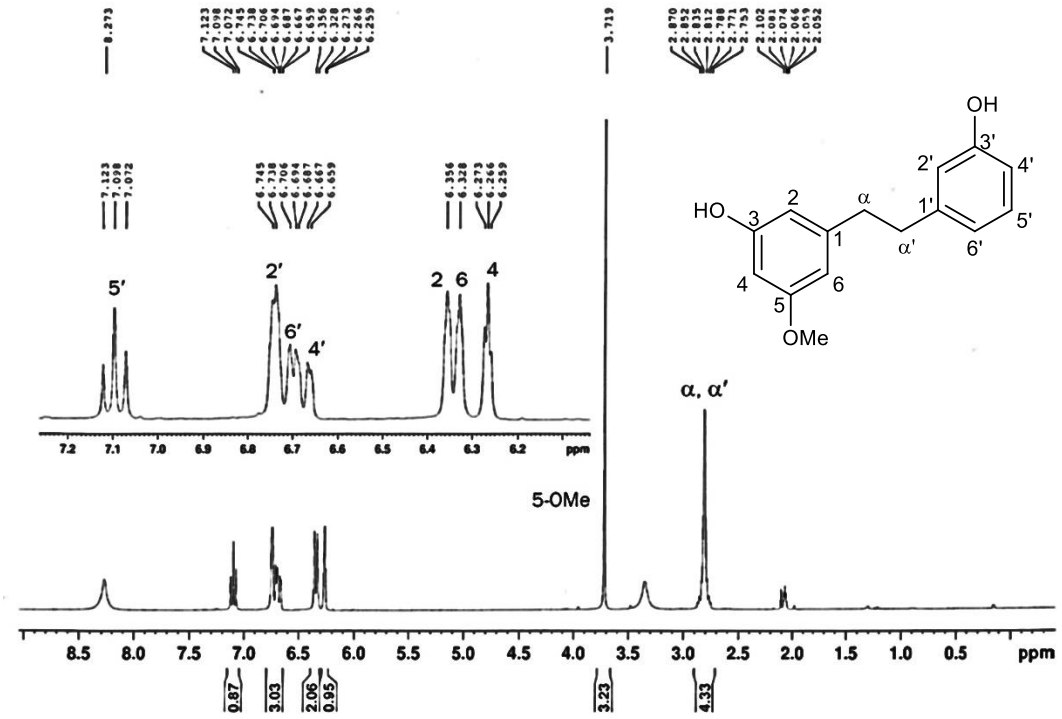


Figure 39 ^{1}H-NMR (300 MHz) spectrum of compound DSC-6 (in acetone-d_6)

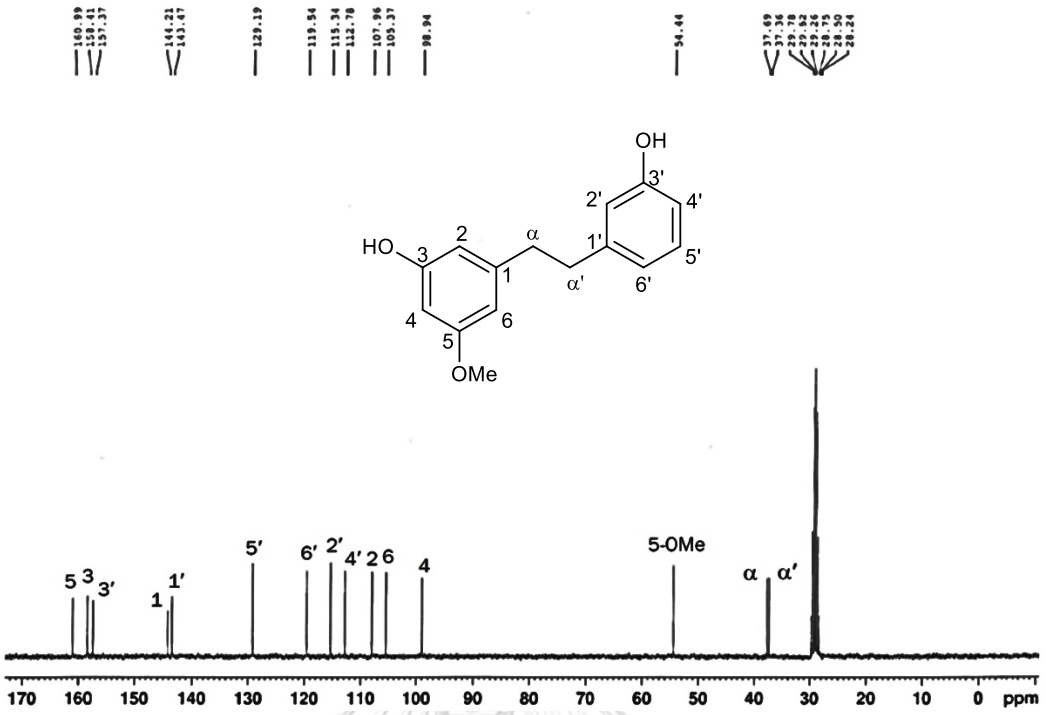


Figure 40 ^{13}C-NMR (75 MHz) spectrum of compound DSC-6 (in acetone-d_6)

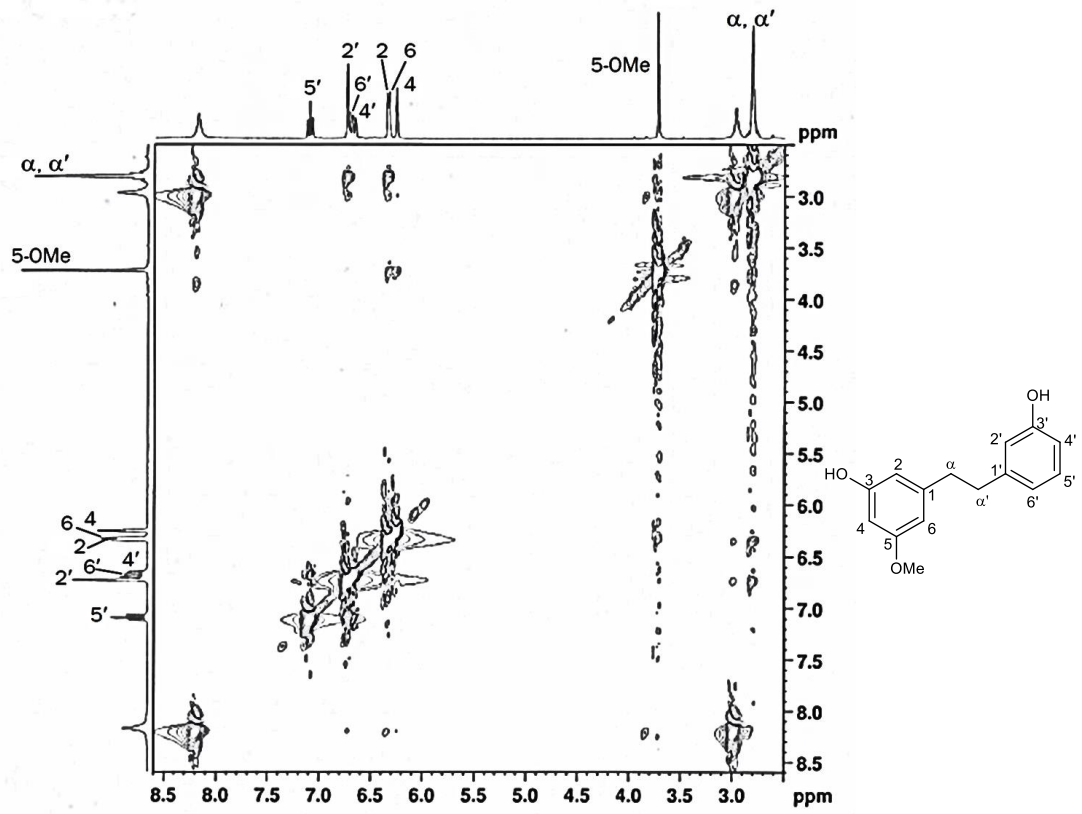


Figure 41 NOESY spectrum of compound DSC-6 (in acetone-d_6)

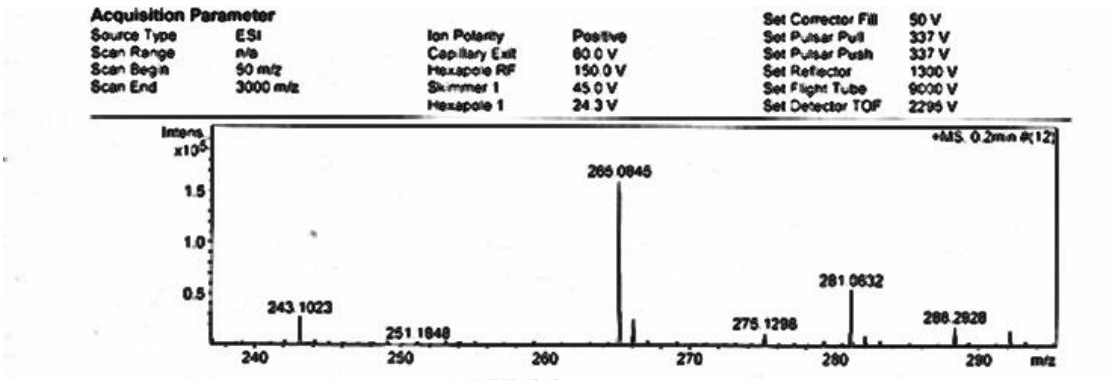


Figure 42 Mass spectrum of compound DSC-7

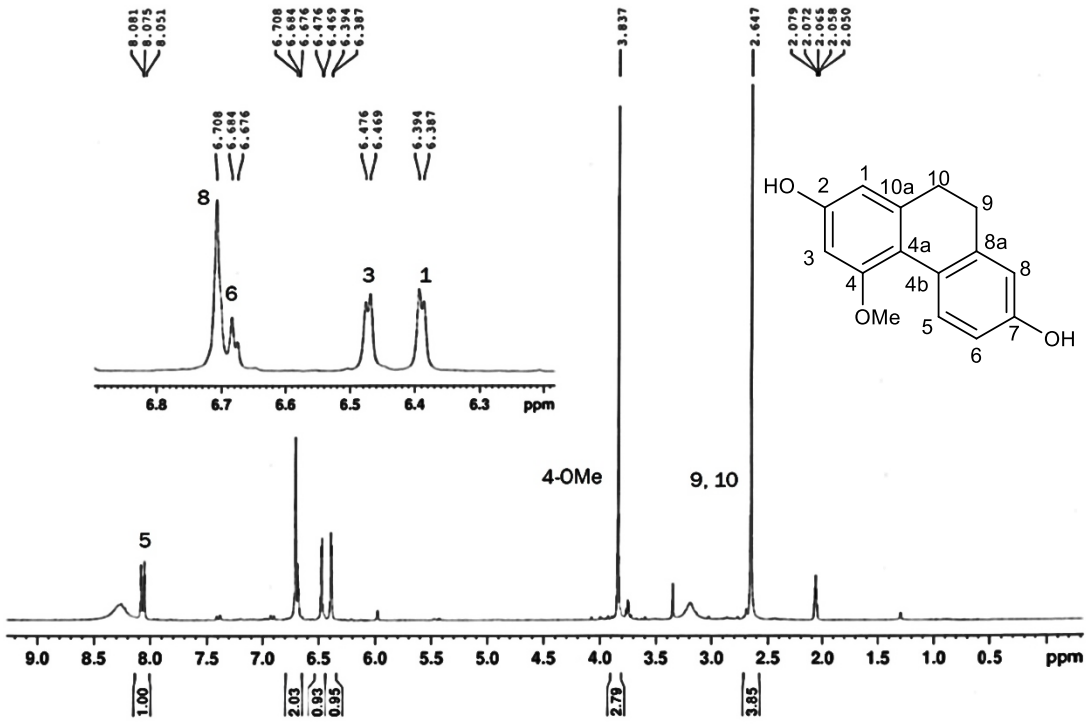


Figure 43 ^{1}H-NMR (300 MHz) spectrum of compound DSC-7 (in acetone-d_6)

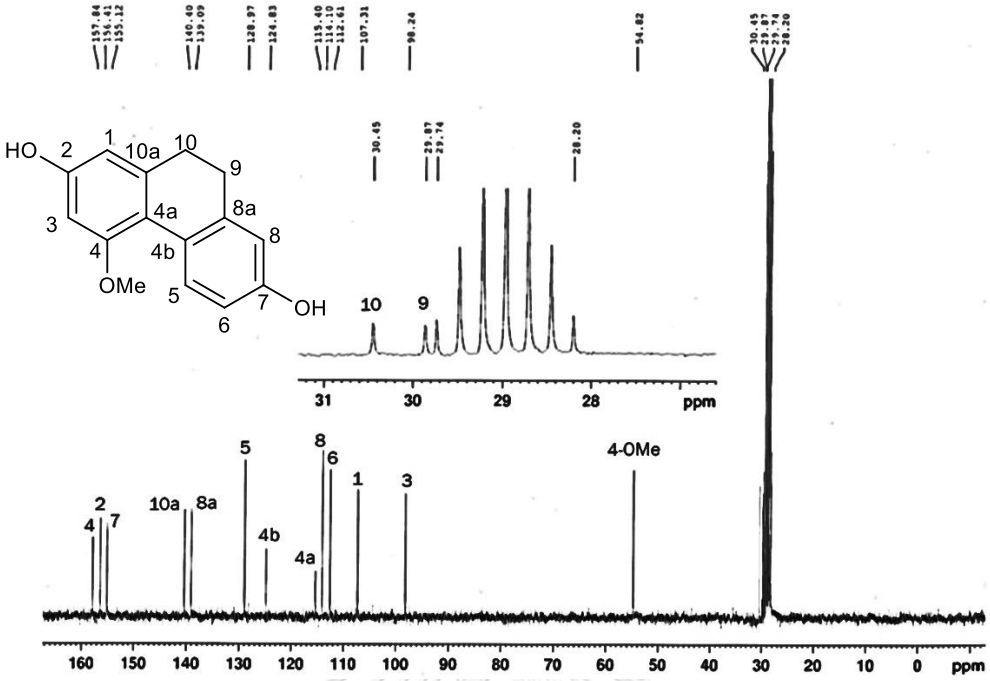


Figure 44 ^{13}C-NMR (75 MHz) spectrum of compound DSC-7 (in acetone-d_6)

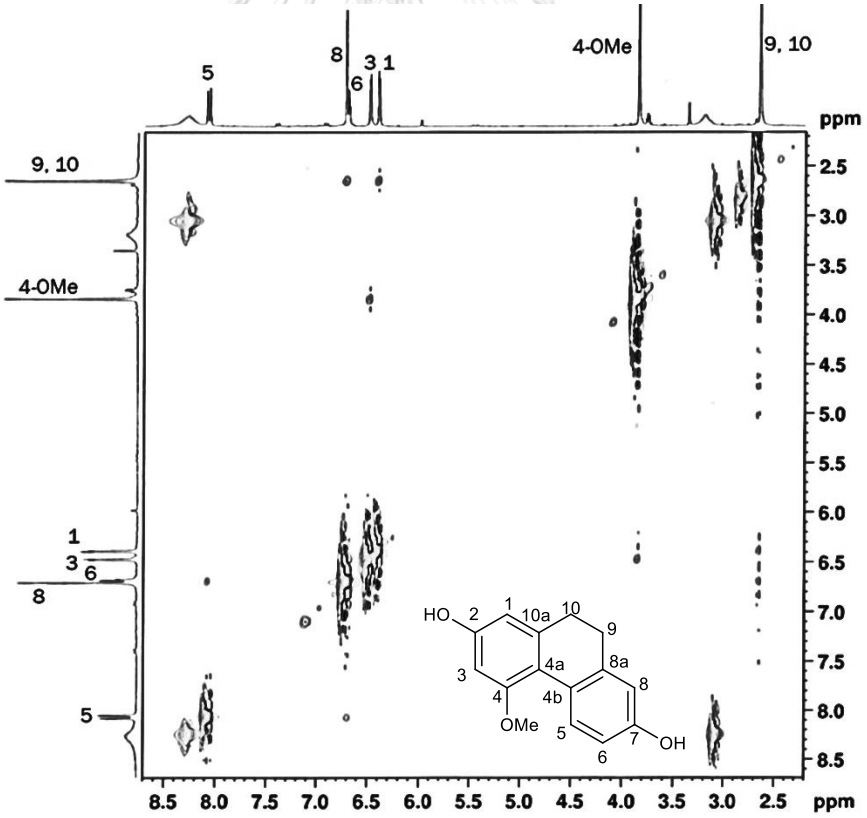


Figure 45 NOESY spectrum of compound DSC-7 (in acetone-d_6)

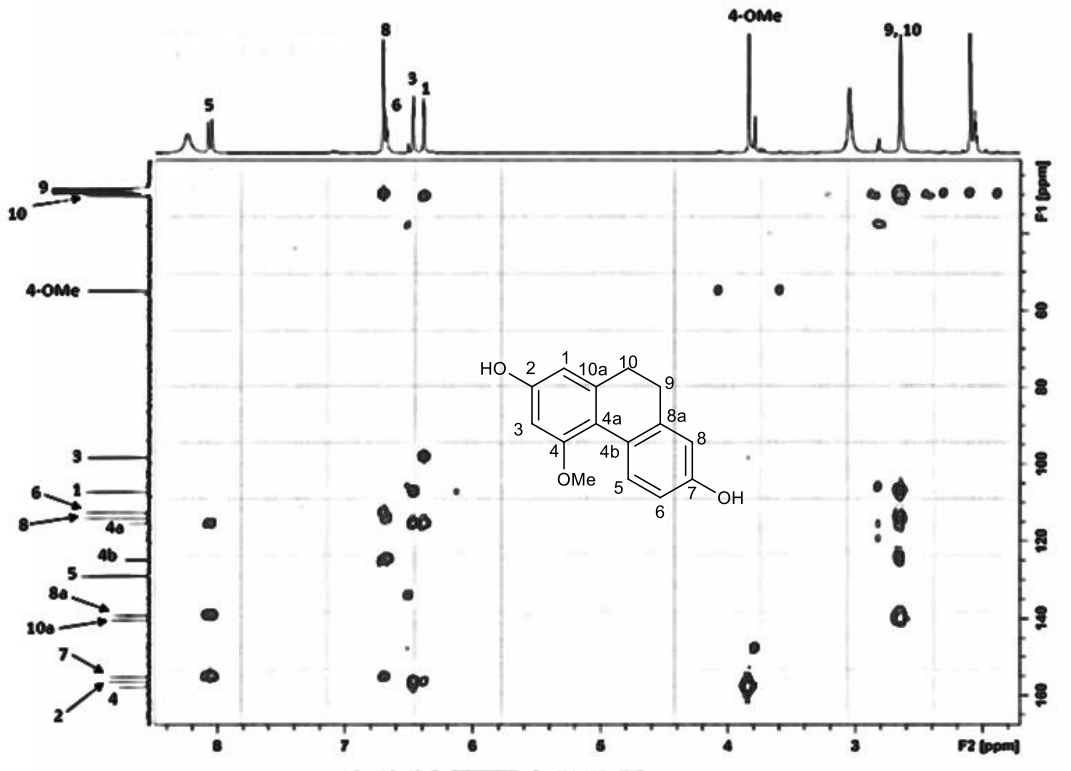


Figure 46 HMBC spectrum of compound DSC-7 (in acetone-d_6)

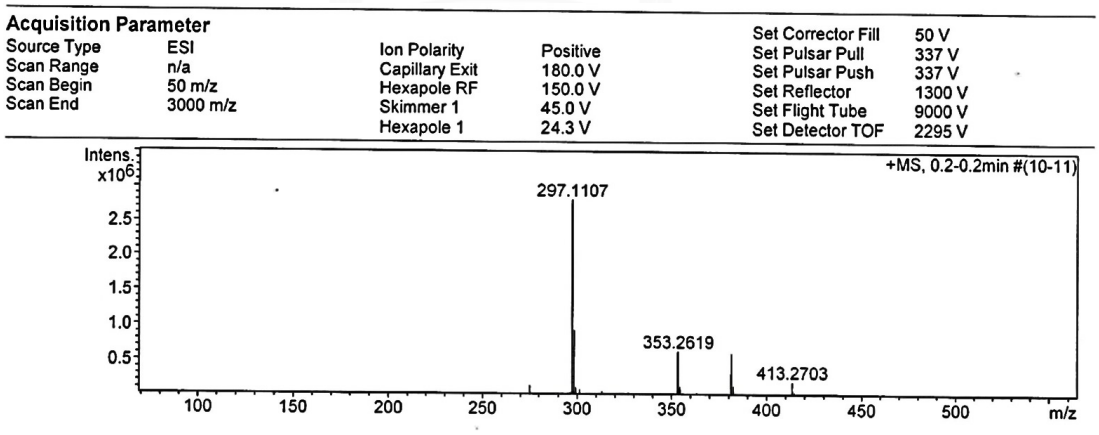


Figure 47 Mass spectrum of compound DSC-8

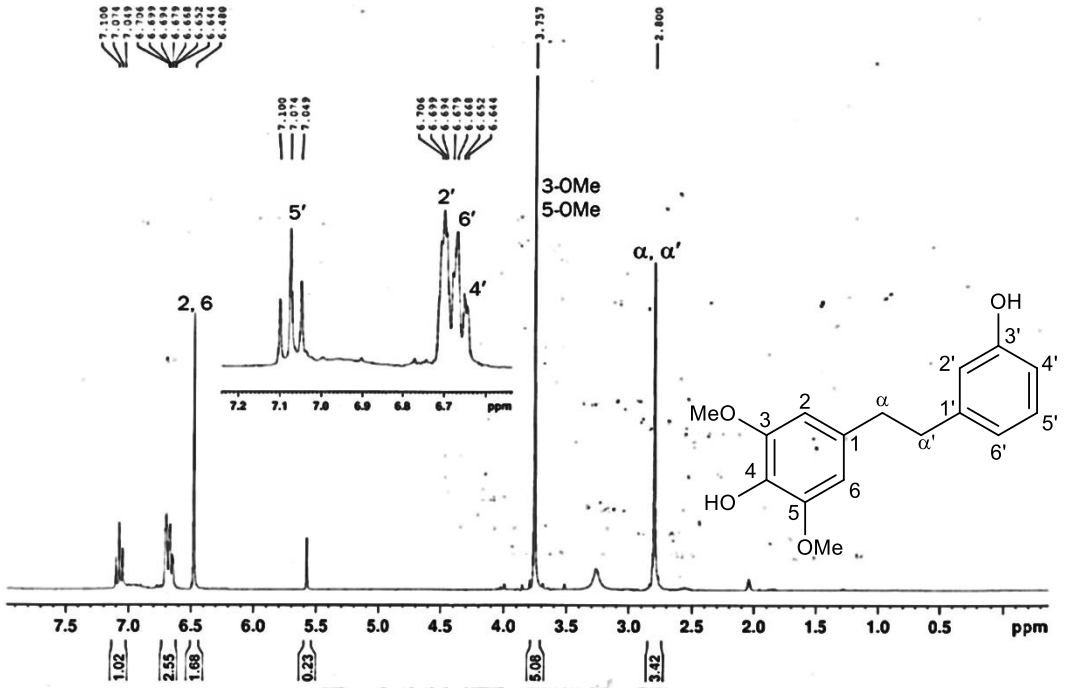


Figure 48 ^{1}H-NMR (300 MHz) spectrum of compound DSC-8 (in acetone-d_6)

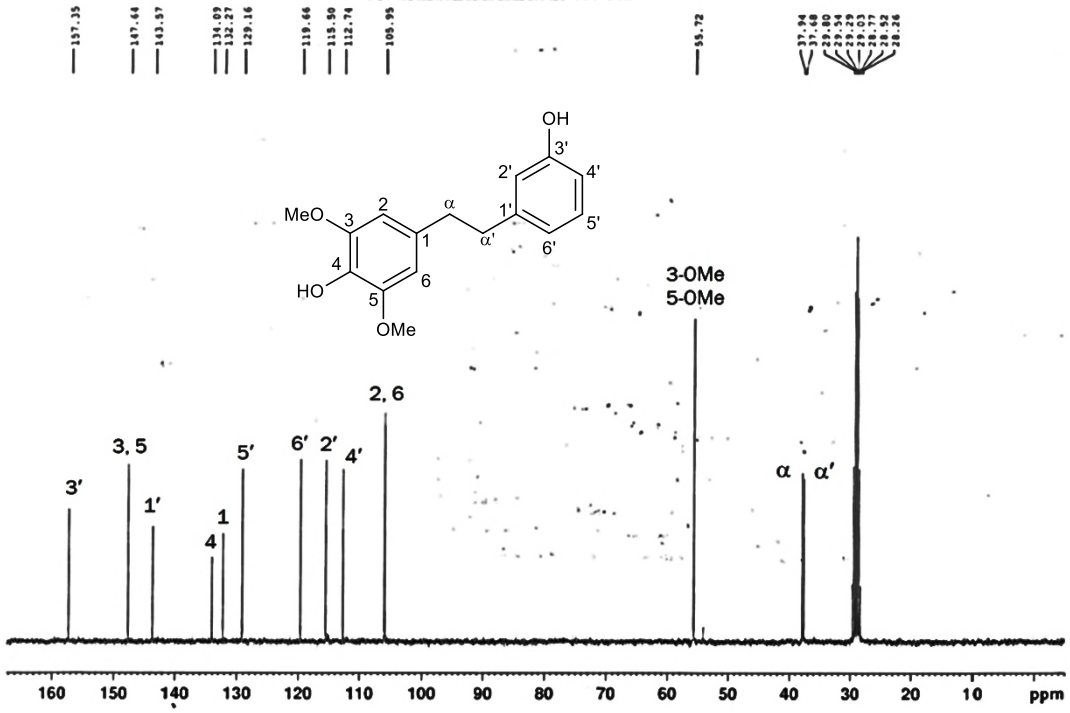


Figure 49 ^{13}C-NMR (75 MHz) spectrum of compound DSC-8 (in acetone-d_6)

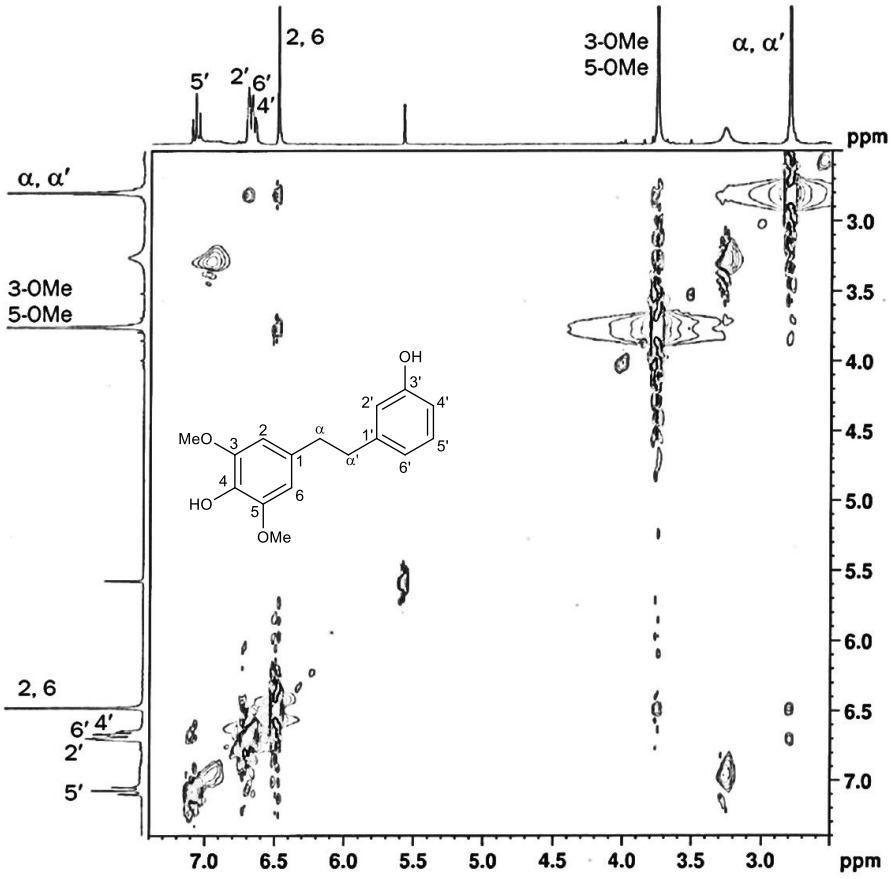


Figure 50 NOESY spectrum of compound DSC-8 (in acetone-d_6)

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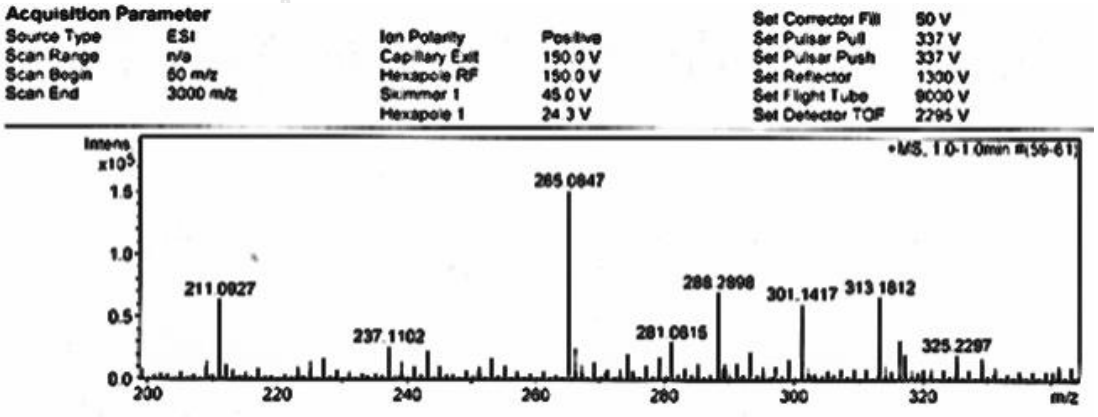

Figure 51 Mass spectrum of compound DSC-9

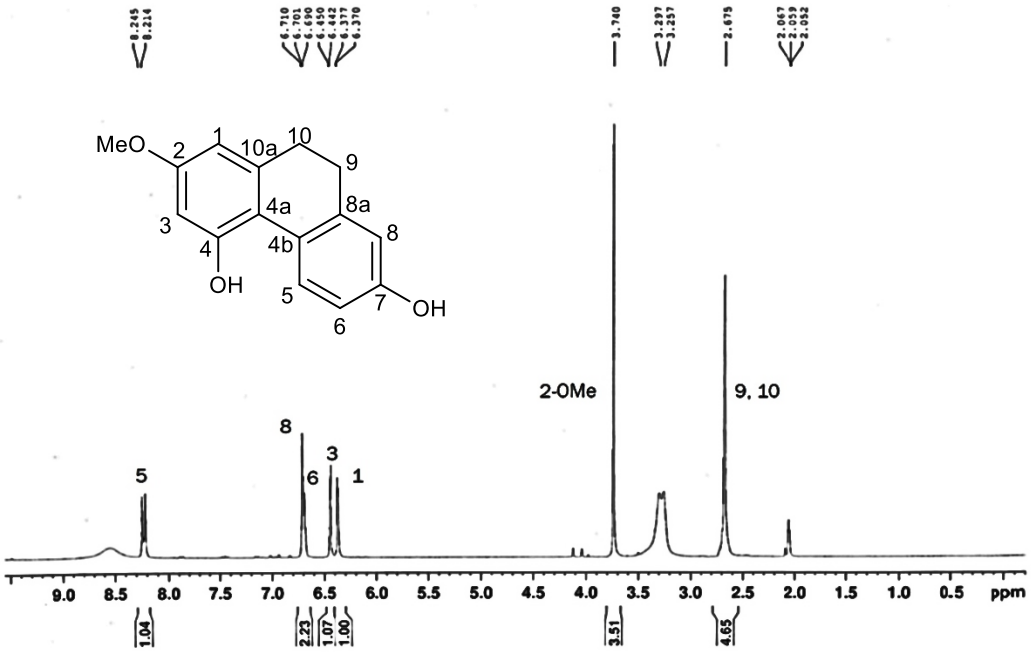

Figure 52 ^{1}H-NMR (300 MHz) spectrum of compound DSC-9 (in acetone-d_6)

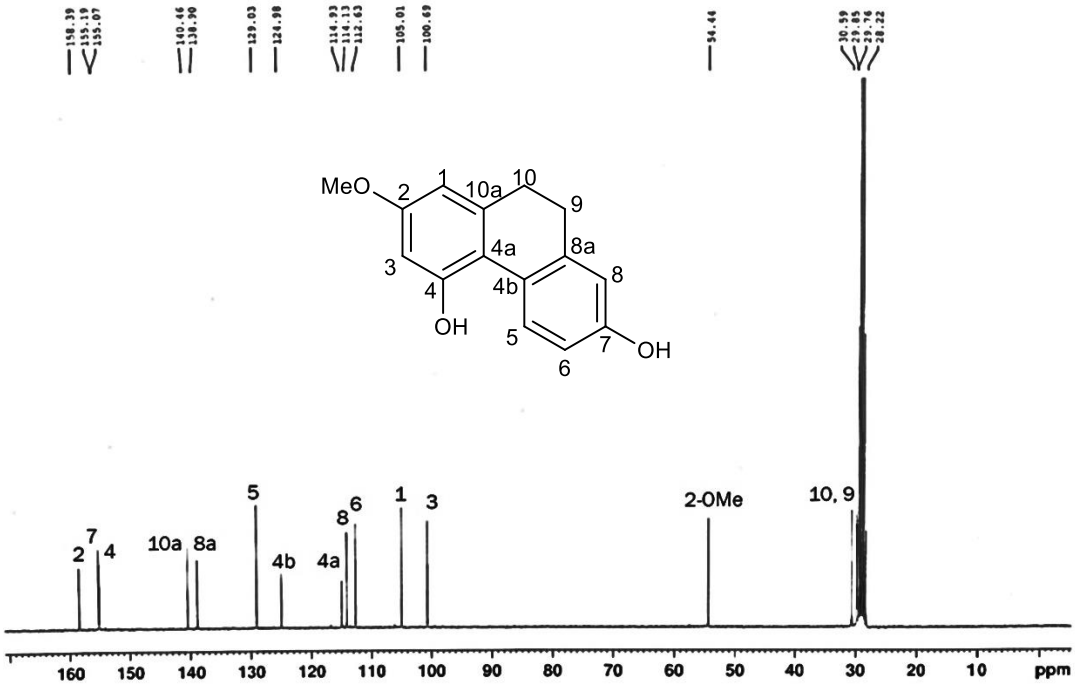

Figure 53 ^{13}C-NMR (75 MHz) spectrum of compound DSC-9 (in acetone-d_6)

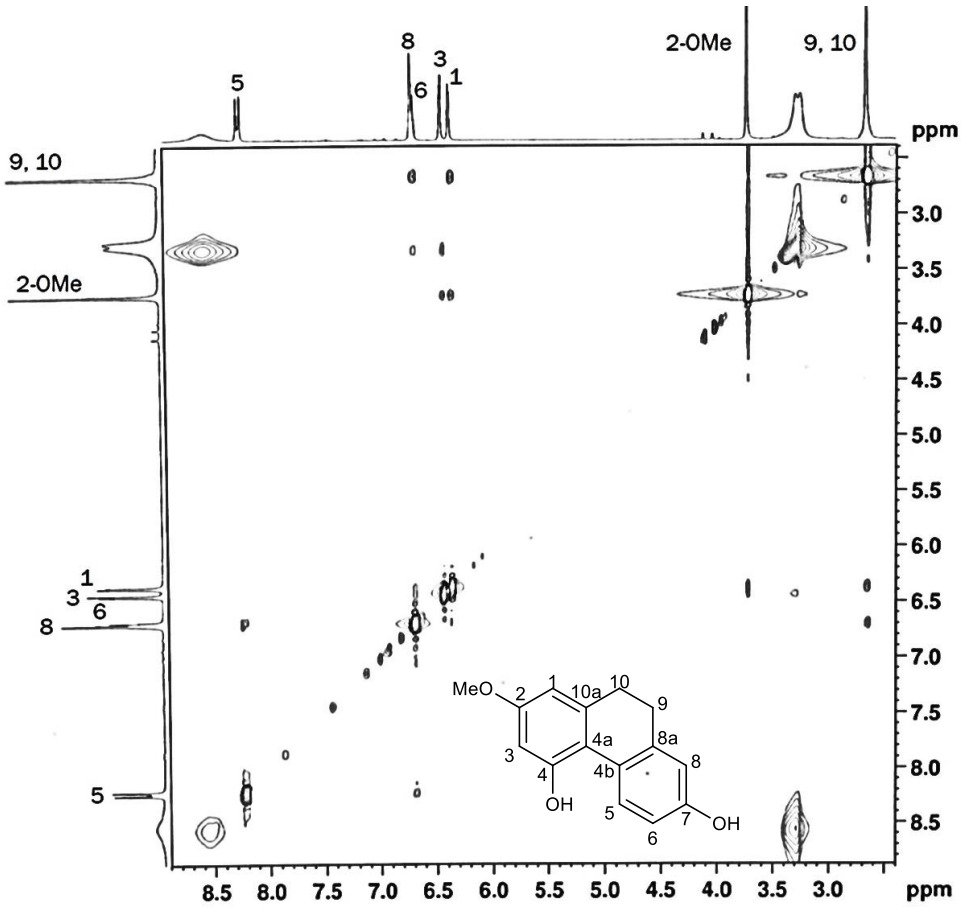

Figure 54 NOESY spectrum of compound DSC-9 (in acetone-d_6)

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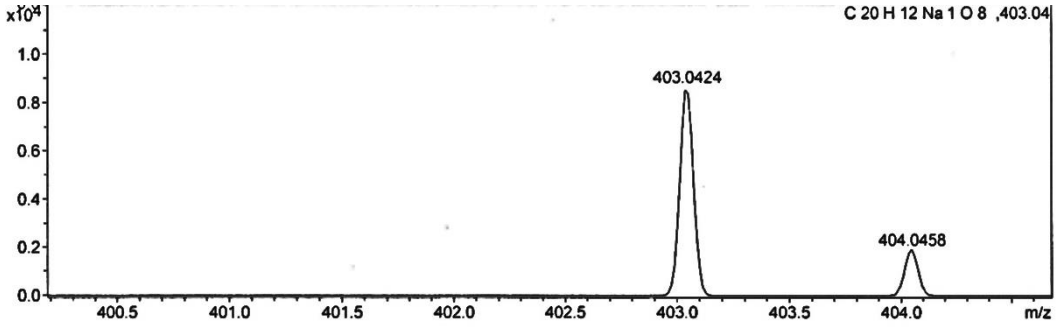

Figure 55 Mass spectrum of compound DSC-10

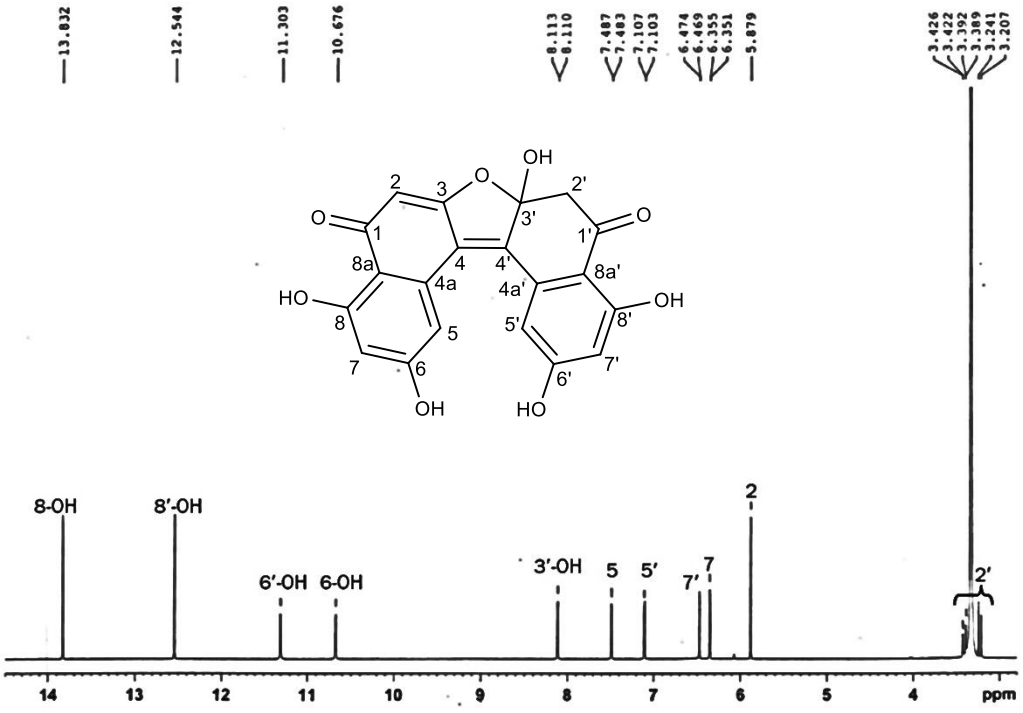

Figure 56 ^{1}H-NMR (500 MHz) spectrum of compound DSC-10 (in DMSO-d_6)

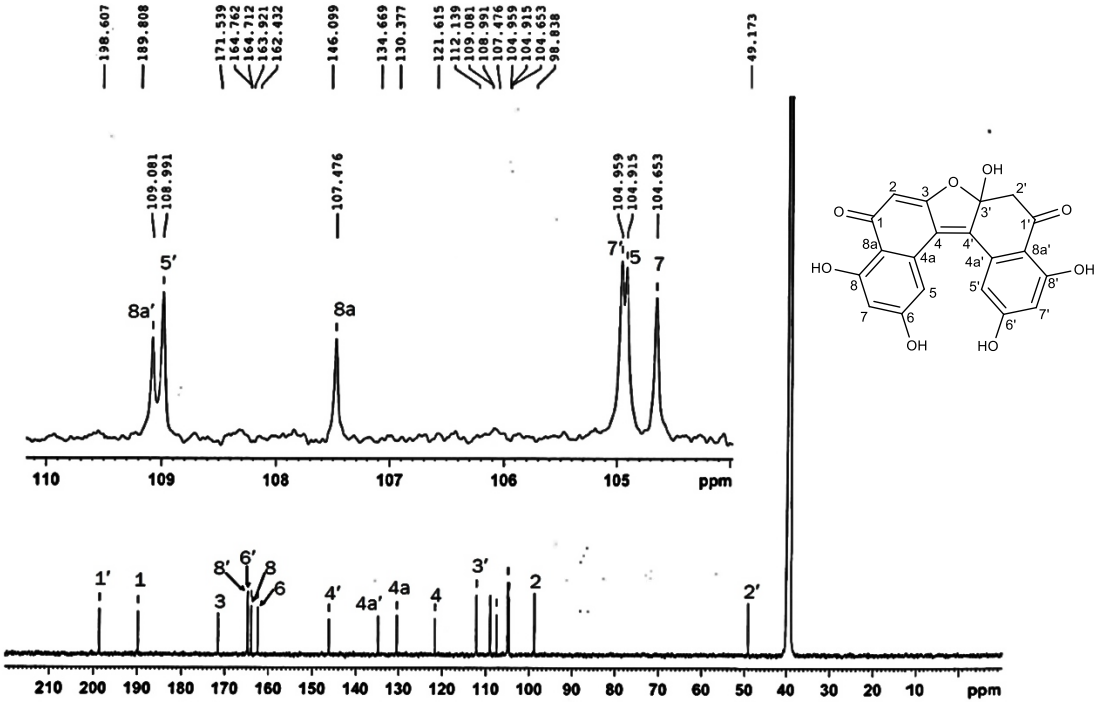

Figure 57 ^{13}C-NMR (125 MHz) spectrum of compound DSC-10 (in DMSO-d_6)

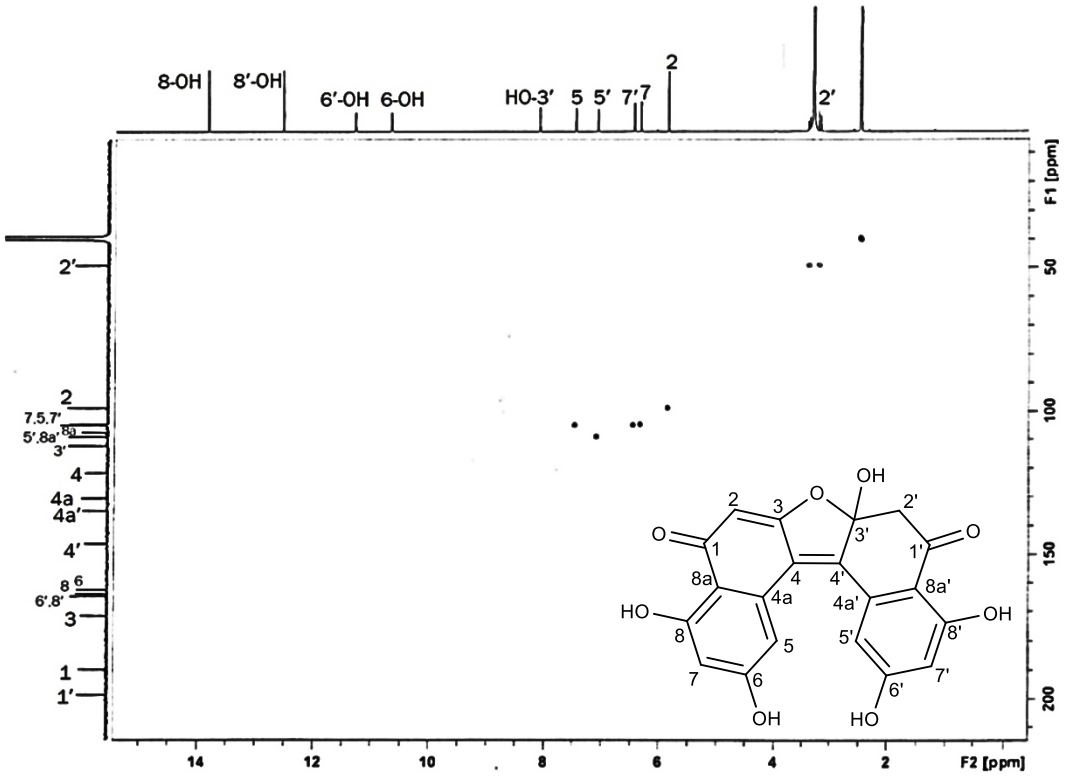

Figure 58 HSQC spectrum of compound DSC-10 (in DMSO-d_6)

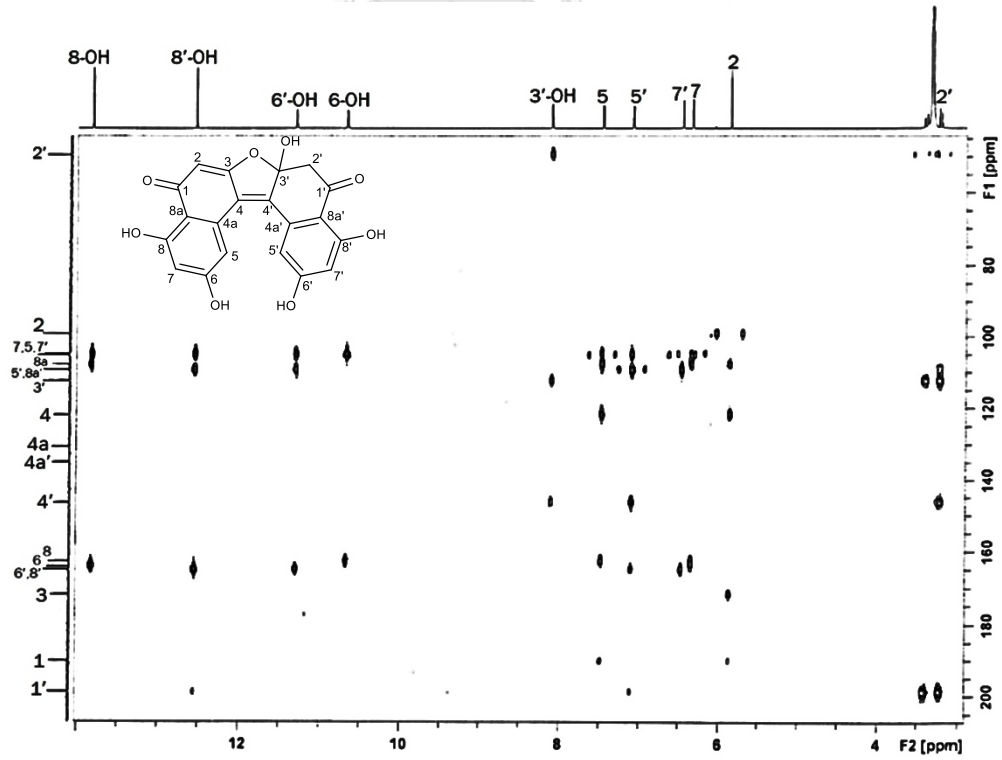

Figure 59 HMBC spectrum of compound DSC-10 (in DMSO-d_6)

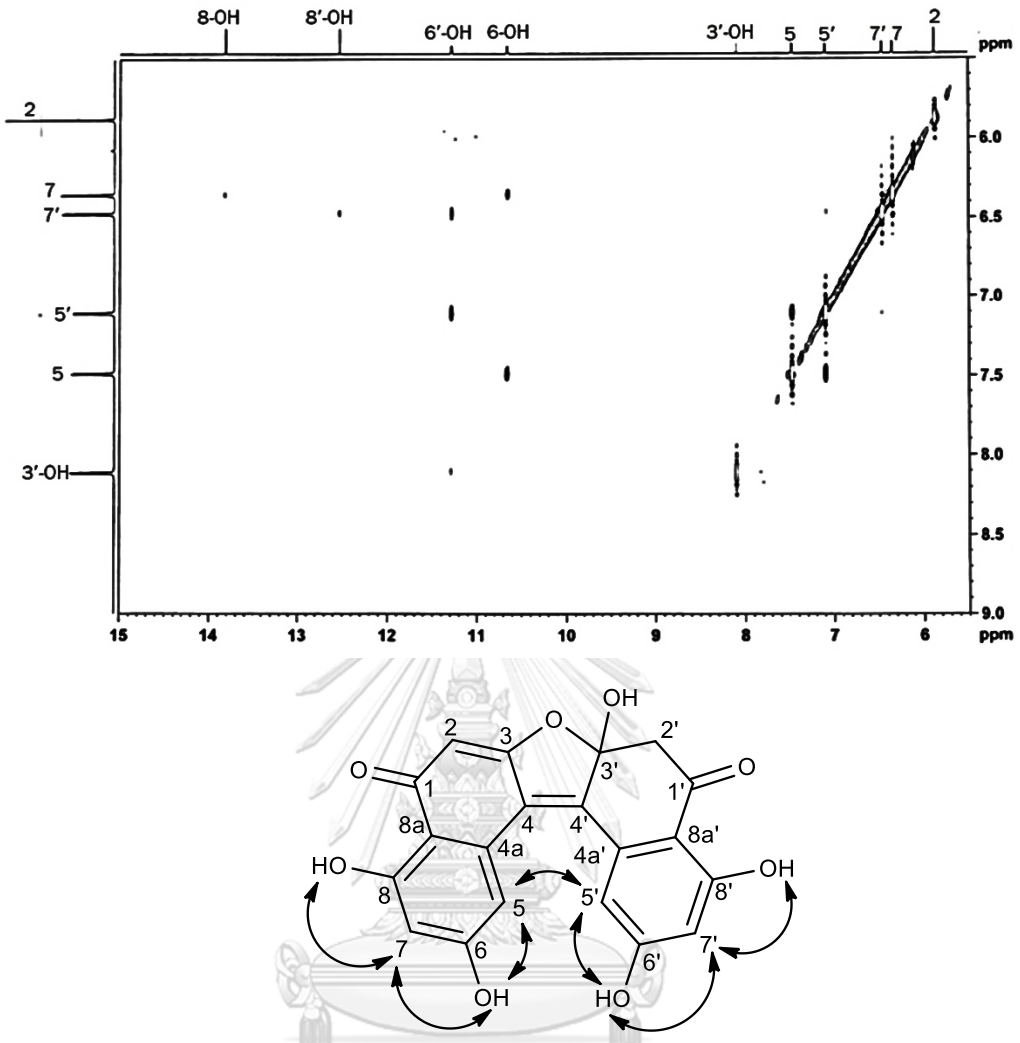

Figure 60 NOESY spectrum of compound DSC-10 (in DMSO-d_6)

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