CHEMICAL CONSTITUENTS AND ANTIBACTERIAL ACTIVITY OF RHIZOMES FROM *Globba schomburgkii* Hook.f.

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A Thesis Submitted in Partial Fulfillment of the Requirements

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องค์ประกอบทางเคมีและการยับยั้งเชื้อแบคทีเรียของเหง้าจากต้นกระทือลิง *Globba schomburgkii* Hook.f.

น.ส.นฤมล เสือแก้ว

วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิทยาศาสตรมหาบัณฑิต
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Thesis Title	CHEMICAL CONSTITUENTS AND ANTIBACTERIAL ACTIVITY OF RHIZOMES FROM *Globba schomburgkii* Hook.f.
By	Miss Naruemon Suekaew
Field of Study	Chemistry
Thesis Advisor	Assistant Professor Dr. Tanatorn Khotavivattana
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นฤมล เสือแก้ว : องค์ประกอบทางเคมีและการยับยั้งเชื้อแบคทีเรียของเหง้าจากต้นกระทือลิง *Globba schomburgkii* Hook.f.. (CHEMICAL CONSTITUENTS AND ANTIBACTERIAL ACTIVITY OF RHIZOMES FROM *Globba schomburgkii* Hook.f.) อ.ที่ปรึกษาหลัก : ผศ. ดร.ธนธรณ์ ขอทวีวัฒนา, อ.ที่ปรึกษาร่วม : ดร.ชฎิล กุลสิงห์

งานวิจัยนี้ ได้นำสารสกัดไดคลอโคมีเทนของเหง้าจากต้นกระทือลิง *Globba schomburgkii* Hook.f. ไปแยกโดยใช้คอลัมน์โครมาโทกราฟีได้เป็นส่วนสกัดหยาบ 10 ส่วน คือ FA-FJซึ่งได้นำส่วนสกัดหยาบเหล่านั้นไปทดสอบฤทธิ์ยับยั้งเชื้อแบคทีเรีย (*S. aureus*) เบื้องต้นโดยใช้วิธีการ 96-well plate microdilution assay พบว่าส่วนสกัดหยาบ FE และ FG มีฤทธิ์ยับยั้งเชื้อแบคทีเรียได้ดีที่สุด ดังนั้นจึงนำส่วนสกัดหยาบ FE และ FG ไปแยกต่อได้เป็นส่วนย่อย 7 ส่วน (FE.A- FE.G) และ 13 ส่วน (FG.A-FG.M) ตามลำดับ จากนั้นนำส่วนย่อยทั้งหมดนี้ไปทดสอบฤทธิ์ยับยั้งเชื้อแบคทีเรีย พบว่ามีหลายส่วนย่อยที่มีฤทธิ์ยับยั้งเชื้อแบคทีเรียได้ดีกว่าส่วนสกัดหยาบ เช่น FE.B, FE.C, FE.D, FG.D, FG.G และ FG.H เป็นต้น นอกจากนี้ยังได้นำทั้งส่วนสกัดหยาบและส่วนสกัดย่อยทั้งหมดไปวิเคราะห์โดยใช้ gas chromatography hyphenated with mass spectrometry (GC-MS) เพื่อหาองค์ประกอบทางเคมีจากการวิเคราะห์ความสัมพันธ์ระหว่าง mass spectrum (experimental MS) ของสารตัวอย่างเทียบกับ retention index จากฐานข้อมูล NIST17 ทำให้สามารถคาดการณ์องค์ประกอบทางเคมีทั้งหมด 167 องค์ประกอบ ซึ่งมีองค์ประกอบทางเคมีที่สำคัญได้แก่ γ-bicyclohomofarnesal และ (*E*)-15,16-dinorlabda-8(17),11-dien-13-one (P2) และมีองค์ประกอบที่มีการรายงานฤทธิ์ทางชีวภาพ เช่น (*E*)-labda-8(17),12-diene-15,16-dial, methyl isocostate, zederone, alantolactone และ 8α,11-elemadiol นอกเหนือจากนี้ยังสามารถแยกสารประกอบบริสุทธิ์ได้ 2 สารประกอบ ได้แก่ petasol (P1) และ P2 จากการวิเคราะห์โดยใช้เทคนิค ^{1}H, ^{13}C, COSY, HSQC และ HMBC NMR

สาขาวิชา เคมี ลายมือชื่อนิสิต .....

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ลายมือชื่อ อ.ที่ปรึกษาร่วม .....

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Naruemon Suekaew : CHEMICAL CONSTITUENTS AND ANTIBACTERIAL ACTIVITY OF RHIZOMES FROM *Globba schomburgkii* Hook.f.. Advisor: Asst. Prof. Dr. Tanatorn Khotavivattana Co-advisor: Dr. Chadin Kulsing

Globba schomburgkii Hook.f. rhizomes were extracted with dichloromethane and fractionated using column chromatography to obtain 10 fractions (FA-FJ). The most active fractions, FE and FG, were further fractionated to give seven and thirteen sub-fractions, respectively. The fractions and sub-fractions were tested for antibacterial activity using 96-well plate microdilution method. The antibacterial activity of several sub-fractions (such as FE.B, FE.C, FE.D, FG.D, FG.G, and FG.H) were higher than that of the crude extract. Chemical compositions of all fractions and sub-fractions were analyzed by gas chromatography hyphenated with mass spectrometry (GC-MS), revealing 167 compounds identified according to match between the experimental MS and retention index data and that from the NIST17 library. The major volatile compounds were identified as γ-bicyclohomofarnesal and (*E*)-15,16-dinorlabda-8(17),11-dien-13-one (P2). Some of the compounds such as (*E*)-labda-8(17),12-diene-15,16-dial, methyl isocostate, zederone, alantolactone, and 8α,11-elemadiol have been reported to be biologically active. Lastly, two pure compounds isolated by column chromatography were characterized by ^{1}H, ^{13}C, COSY, HSQC and HMBC NMR techniques as petasol (P1) and P2.

Field of Study: Chemistry

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Student's Signature .....

Advisor's Signature .....

Co-advisor's Signature .....

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Naruemon Suekaew

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CHAPTER I
INTRODUCTION

1. Background and significance of research

Bacteria is a pathogen that causes many diseases in human, plants and animals. Since, bacteria are evolving over time to survive, this leads to the problem of bacteria becoming resistant to antibiotic. Centers for Disease Control and Prevention (CDC) has reported that in U.S., at least 2.8 million people were infected with antibiotic-resistant bacteria, and more than 35,000 people die due to bacterial infection each year. In Thailand, it has been reported that 38,481 deaths were caused by antibiotic-resistant bacterial infection in 2010. Globally, antimicrobial resistance has a trend to increase to 10 million deaths each year by 2050. Therefore, in the future, the existing antibacterial drugs might not be able to use to treat bacteria effectively anymore. As a result, the development of new antibacterial drugs is necessary to solve the problem caused by antibiotic-resistant bacteria. The extraction and separation of chemical constituents from plants is one of the promising methods to discover new antibacterial compounds, since traditional medicine uses various herbs to treat a wide range of ailments. Plants in the family of Zingiberaceae have been used in herbal remedies from past to present to treat bacterial diseases. The interesting plant is *Globba schomburgkii* Hook.f. or Kratue Ling, which the rhizomes have been reported to be used as a mixture of shampoo and soap for use in the treatment of itchy skin.

The Kratue Ling is a local plant of Saraburi Province. The flowers are typically used in Buddhist Lent day (Khaophansa). It is usually used to bundle into a bouquet to be offered to monks in Buddhist Lent day. Therefore, it is alternatively called as Khaophansa flower. Therefore, the Kratue Ling is an important economic plant of Saraburi Province and the Saraburi Provincial Public Health Office is also interested in

the development of the Kratue Ling into cosmetic products or medical supplies to promote the policy of Saraburi Province under the Herbarium City Development Project. The study of chemicals constituents and biological activity of Kratue Ling is very necessary. From the preliminary study of our research group was found that the crude extracts from the rhizomes of the Kratue Ling exhibited good antibacterial activity. When analyzed with gas chromatography-mass spectrometry (GC-MS) which is a technique used for separation and identification of the volatile compositions, more than 100 chemical volatile compositions which are mostly of terpenoids were identified. However, this research was not complete: the exact compounds that act as active to antibacterial activity still have not been identified. In addition, there were no report of pure compounds isolation for structural characterization by using reliable techniques.

This research aimed to study the bioactive compound in the crude extract from the rhizomes of *Globba schomburgkii* Hook.f. by using bioassay-guided fractionation technique. Furthermore, GC-MS technique is used for chemical composition analysis of fractions and sub-fractions that were obtained from column chromatography. The correlation between chemical compositions and antibacterial activity will be studied. This could serve as a prototype structure for future antibacterial drug discovery and development. The information on the chemical compounds to be used to determine the quality of the pharmaceutical products processed from this herb in the future.

2. Literature review

2.1 Bacterial infection and antibiotic resistant

Bacteria is the pathogen which is a major cause of various diseases such as skin infections, digestive system infection, and respiratory system infection.[1] Bacteria is typically classified according to gram color straining.[2] Gram positive bacteria are bacteria that retain the crystal violet stain. For example, *Staphylococcus aureus* (*S.*

aureus) is a pathogen that usually occurs on the skin,[3] and *Micrococcus luteus* (*M. luteus*) is a non-pathogenic organism but is a cause of shock after infection in the bloodstream for patients with immunodeficiency.[4] Gram negative bacteria are bacteria that do not retain the crystal violet stain. For example, *Psaeudomonas aeruginosa* (*P. aeruginosa*) is a pathogen causing disease in people with immunodeficiency such as cancer patients under chemotherapy.[5] Another example, *Escherichia coli* (*E. coli*) is a pathogen that causes urinary tract infections and inflammation.[6] According to centers for disease control and prevention (CDC), one of the biggest problems of global health is antibiotic-resistant bacteria nowadays. Since antibiotics are used frequently, the bacteria have been mutating to become resistant to most of the old drugs, and they have become ineffective for the treatment.[7] For example, a strain of *S. aureus* that is resistant to penicillin had been reported for first time in 1940.[8] In Thailand, 38,481 deaths have been reported to be caused by antibiotic-resistant bacteria in 2010.[9] Antimicrobial resistance has a trend to increase to 10 million deaths each year by 2050.[10] There are several mechanisms that can cause drug resistance in bacteria such as enzymatic modification of antibiotics, enzymatic degradation of antibiotics and efflux pumps.[10] Therefore, the development of antibiotic discovery is very important to help humanity.

2.2 Antibacterial drugs

Bacterial infection and antibiotic resistance are the big health problems in the world for a long time until now, so the antibacterial drug must be continuously discovered and developed. The first antibacterial drug is prontosil (**Figure 1.1a**) discovered by Gerhard Domagk in 1932.[11] The first natural antibiotic is penicillin (**Figure 1.1b**) that was used to help a lot of soldiers in World War II. Penicillin was extracted from *penicillium notatum* and can inhibit *S. aureus* that grew around it on an agar plate.[8] In this research we use streptomycin (**Figure 1.1c**), amoxicillin (**Figure 1.1d**), and ciprofloxacin (**Figure 1.1e**) as

antibiotic drug reference standards during antibacterial activity testing. First, streptomycin is a natural aminoglycoside antibiotic produced by the soil Actinomycete *Streptomyces griseus*. It is usually used by combination with another drug to treat septicemia, urinary tract and gastrointestinal tract infections, and numerous other infections caused by gram-positive and gram-negative bacteria. The mode of action of streptomycin is the inhibition of the synthesis of proteins used for cell wall production in the target bacteria.[12] However, it has been reported that some bacteria such as *Mycobacterium tuberculosis* has become resistant to streptomycin.[13] Second, amoxicillin is a broad-spectrum antibiotic drug in the penicillin group. It has been reported to have high inhibition against gram-negative bacteria. Recently, gram-negative bacteria can release *β*-lactamase, leading to the resistance towards amoxicillin.[14] In addition, it has been reported that 83.1% of *S. aureus* infection cases had become resistant to amoxicillin in hospitalized patients, and 73.9% in ambulatory patients.[15] Finally, Ciprofloxacin is an antibiotic in the fluoroquinolone group that can inhibit gram-negative bacteria such as urinary tract infections, skin and bone infections, and especially most active against *P. aeruginosa*.[16] In 1997, the mechanistic study of *Streptococcus pneumoniae* resistant to ciprofloxacin has been reported.[17] However, amoxicillin and ciprofloxacin are the common essential antibiotic recommended by the World Health Organization (WHO).[18] There are ninety antibacterial drugs that are commercialized in 1982-2002 and 79% of them were derived from natural product.[19] Therefore, natural molecules remain important sources for novel antibiotics. However, the difficulty in new antibacterial drug discovery from natural product by directly screening for antibacterial activity is the presence of large amounts of known compounds with the low contents of novel compounds.[20]

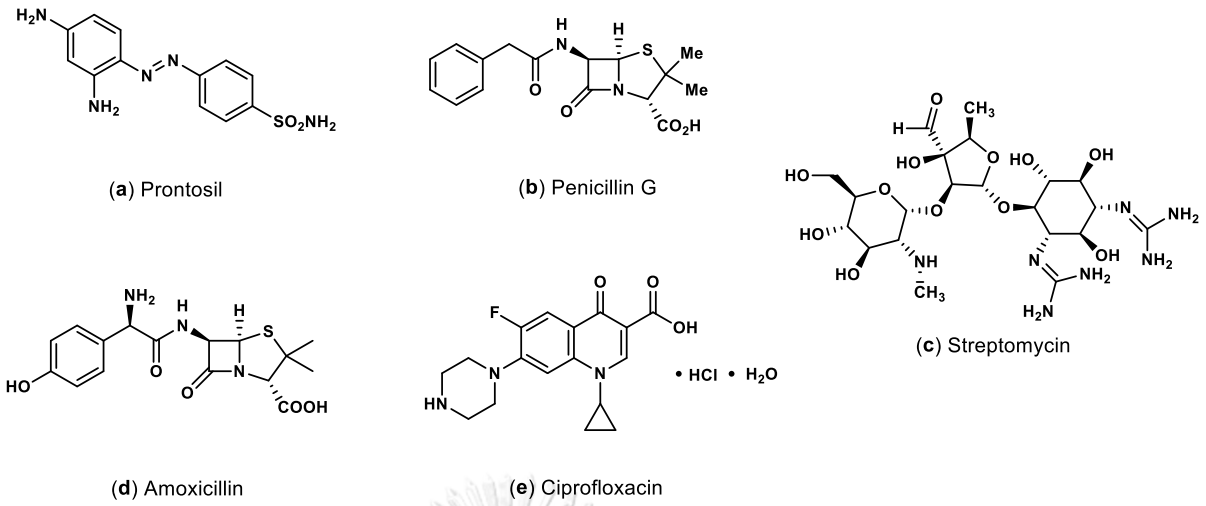
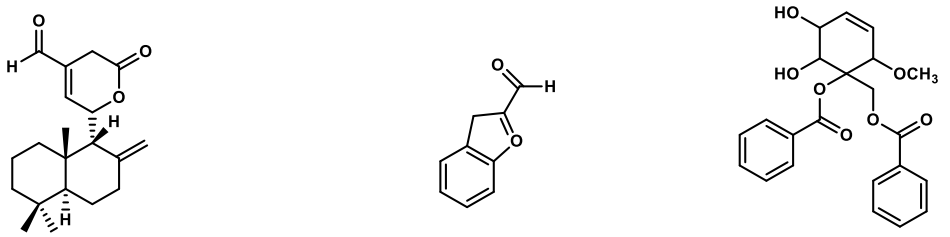


Figure 1.1: Chemical structures of antibacterial drugs

2.3 *Globba schomburgkii* Hook.f.

Globba spp. is the third most diverse genus of the zingiberaceae has been reported various chemical constituents and biological activity. Ethanolic extract of *G. bulbifera* tuber has been reported for protease inhibition and breast cancer inhibition.[21] *G. marantina* Linn. was used as traditional medicine such as asthma, rheumatoid arthritis, cough, cold, and snakebite.[22] The volatile constituents of the rhizomes and aerial parts of *G. sessiliflora* Sims were analyzed by GC-MS. Both the oils showed significant myorelaxant effect on isolated duodenum smooth muscles of Wistar rats. The oils exhibited moderate to good antioxidant activity in a dose-dependent manner.[23] Interestingly, *Globba* is a source of new chemical compounds to the future development of drug discovery. A new Globbanol A was isolated from *G. pendula* Roxb. rhizomes by using column chromatography.[24] Various terpenoids were reported to be the major constituents of *G. reflexa* Craib rhizomes.[25] A new naturally occurring of 16-oxo-(8)17-12-labdadien-15,11-olide (Figure 1.2a) and benzofuran-2-carboxaldehyde (Figure 1.2b) were isolated from *G. pendula*.[26] The structure of new compounds from rhizomes of *G. pendula* are shown in Figure 1.2.



(**a**) 16-Oxo-(8)17-12-labdadien-15,11-olide (**b**) Benzofuran-2-carboxaldehyde (**c**) Globbanol A

Figure 1.2: New compounds from *G. pendula*

Globba schomburgkii Hook.f. is a plant in *Globba* genus of the Zingiberaceae family and the most plentifully distributed in Southeast Asia, especially in Thailand.[27] More than 40 species of plants in *Globba* genus were found in Thailand.[25] The main morphological characteristics are 4 anthers appendages, yellow flowers, green inflorescences and separate branch.[28] The essential oil of *G. schomburgkii* has been extracted by hydrodistillation of whole plants and the chemical compositions of the essential oil has been studied.[29] In addition, our group also studied the volatile compositions from crude extracts obtained from rhizomes, stalks, leaves, and flowers of *G. schomburgkii*. The evaluation of the antibacterial activity of each extract revealed that the dichloromethane extract of rhizomes, which contained the highest amount of terpenoids, had the highest antibacterial activity.[30] Therefore, in this research, the crude dichloromethane extract of the rhizomes of *G. schomburgkii* will be further studied by using bioassay-guided fractionation technique and GC-MS analysis.

2.4 Bioassay-Guided Fractionation

Bioassay-guided fractionation is a method usually used in drug discovery research due to its potency to derive bioactive compounds from natural sources. In this process, the fractionation and biological activity testing are alternatively performed until pure compounds with high bioactivity are obtained.[31] A crude extract of the plants is typically fractionated using separation techniques such as chromatography or distillation to provide fractions. After evaluation of biological activity of these fractions, the most

bioactive fraction will be selected for further isolation to obtained sub-fractions. The process is repeated until pure compounds, which are expected to have high bioactivity, are isolated. The general bioassay-guided fractionation process is shown in **Figure 1.3**.

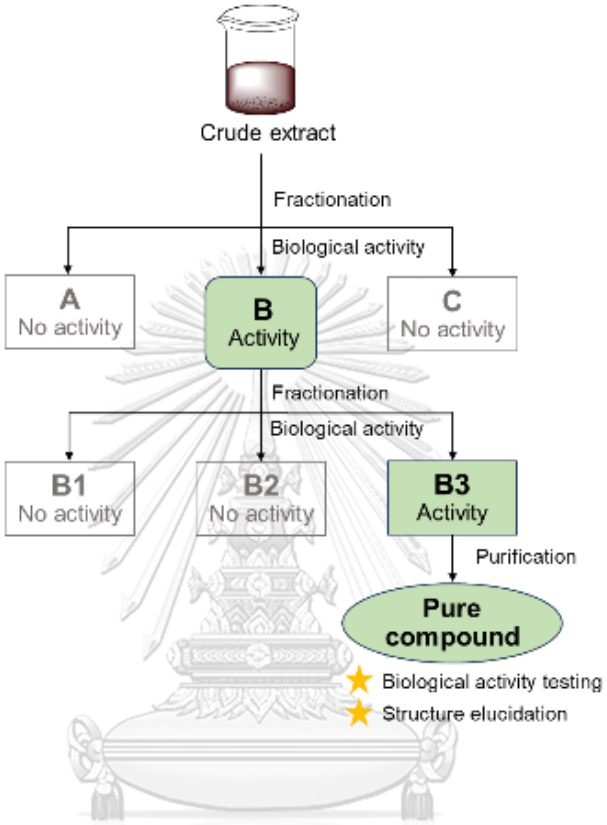


Figure 1.3: General process of bioassay-guided fractionation

A successful example for the bioassay guided fractionation for the discovery of new antibacterial agent from plants has been reported by Lianzhu Lin and co-workers in 2013.[32] In this work, a crude ethanol extract of *R. Serra* leaf was extracted with 4 solvents including petroleum ether (LPEL), ethyl acetate (LEAL), butanol (LBL), and water (LWL). LEAL was found to exhibit the highest antibacterial activity. This fraction was further fractionated into 9 sub-fractions (EA1-EA9) and it was found that EA4 gave the highest antibacterial activity. The isolation of EA4 revealed three phenolic compounds and four C-20 oxygenated *ent*-kauranes (effusanin E, lasiodin, rabdosichuanin D and a new compound namely effusanin F). Lasiodin was found to be

the most active antibacterial compound against *B. subtilis*, *B. cereus*, *S. aureus*, *L. monocytogenes*, and *P. aeruginosa* with MIC values of 16, 8, 16, 16, and 16 μg/mL, respectively. Moreover, effusanin E and effusanin F also exhibited moderate antibacterial activity.

2.5 Volatile chemical composition analysis by GC-MS

GC-MS is a useful analytical technique that has been used to obtain profiles of volatile and semi-volatile organic compounds in herb, where compounds are often identified according to MS library match and retention index data.[30, 33] The technique allows positive identification of compounds as well as quantitative analysis by construction of calibration curves. GC provides physical separation of components of a mixture mainly based on analyte boiling difference and interaction with the stationary phase while MS is used as the detector for the identification. There have been several reports of volatile chemical composition analysis with GC-MS. The chemical compositions of essential oils from *G. cernua* Baker, *G. marantina* L. and *G. ophioglossa* Wight were analyzed by using GC-FID and GC-MS.[34] The chemical compositions of the essential oil from *Dendranthema indicum* Var. *Aromaticum* was also studied by GC-MS analysis showing five categories of compounds including oxygenated monoterpenes, oxygenated sesquiterpenes, sesquiterpenes, fatty hydrocarbons and monoterpenes.[35] The chemical compositions of stem bark methanol extracts of *Khaya senegalensis* were isolated and analyzed using GC-MS, and the total ion chromatogram has seven teen peaks as shown in **Figure 1.4**.

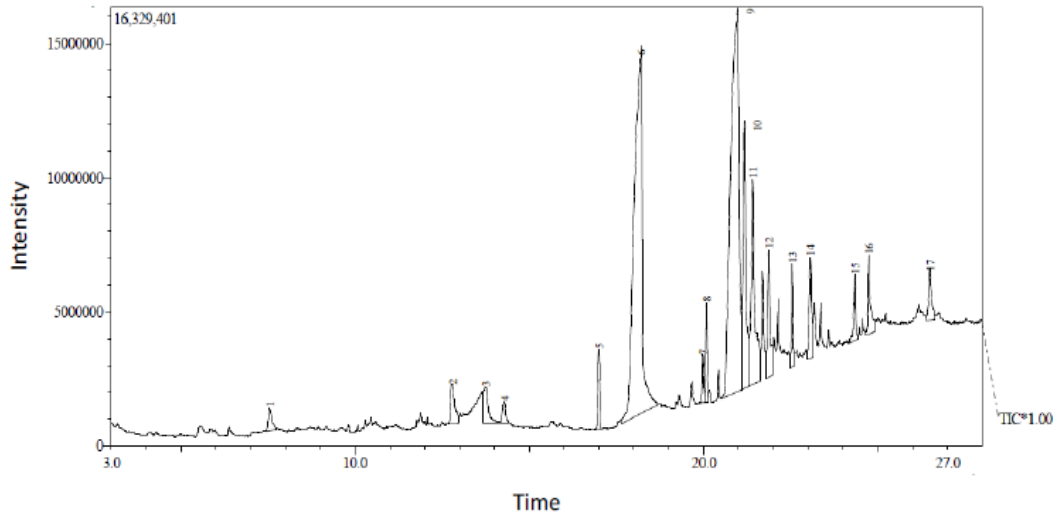


Figure 1.4: Total ion chromatogram of methanol extract of *K. senegalensis*.

3. Objectives

3.1 To analyze the chemical compositions in the rhizome extract of *Globba schomburgkii* Hook.f. by fractionation and GC-MS technique

3.2 To study the relation between chemical compositions and antibacterial activity

3.3 To isolate the bioactive chemical species by bioassay-guided fractionation and to elucidate their structures by spectroscopic technique

4. Scope of research

Crude dichloromethane extract of the rhizomes of *G. schomburgkii* was isolated using chromatographic technique into fractions, which will be tested for their antibacterial activity and GC-MS analysis. The comparison between antibacterial activity results and chemical compositions from GC-MS analysis to exhibit the relation between bioactivity and the quantity of significant chemical species. The fractions with high bioactivity were selected for further fractionation in order to obtain sub-fractions. This bioassay-guided fractionation was repeated until pure compounds were obtained. The pure compounds were characterized by ^{1}H, ^{13}C, COSY, HSQC, and HMBC NMR. The GC-MS analysis of pure compounds was used to predict the structure and compare with

NMR interpretation. Furthermore, the antibacterial activity of pure compounds was evaluated to compare with the crude extract. The overview of this research is shown in Figure 1.5

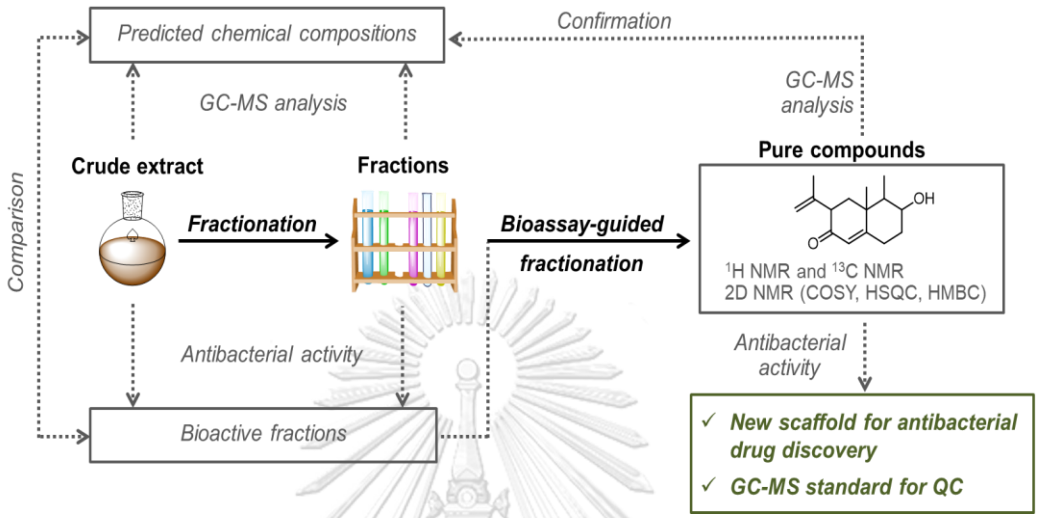


Figure 1.5: Overview scheme of bioassay-guided fractionation and chemical compositions of *Globba schomburgkii* Hook.f. rhizomes.

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CHAPTER II

EXPERIMENTS

1. Plant preparation and extraction

G. schomburgkii Hook.f. was obtained from a commercial grower (Roum Jai Kaset farm) in Phu Kham Chan subdistrict, Phra Phutthabat district, Saraburi Province, Thailand, in June 2018. The plant was authenticated at the Kasin Suvathabandhu Herbarium of the Department of Botany, Faculty of Science, Chulalongkorn University, Bangkok, Thailand. A voucher specimen (BCU 016011) was deposited in the herbarium.

All commercial-grade solvents from RCI Labscan (Samutsakorn, Thailand) were used to extract the plants. The air-dried *G. schomburgkii* Hook.f. rhizomes (2.92 kg) were ground and extracted twice with hexane (8.0 L each time) at room temperature. After filtration, the solid residue was further extracted with dichloromethane (9.0 L each time), filtered, and then, the solvent evaporated *in vacuo* to obtain a dark brown viscous dichloromethane extract (37.21 g).

2. Isolation and fractionation

All solvents for column chromatography from RCI Labscan (Samutsakorn, Thailand) were distilled before use. Column chromatography were monitored by thin-layer chromatography (TLC) using aluminium Merck TLC plates coated with silica gel 60 F_{254}. Normal phase column chromatography was performed using silica gel 60 (0.063-0.200 mm, 70-230 mesh ASTM, Merck, Darmstadt, Germany). Proton, carbon and two-dimensional nuclear magnetic resonance (^{1}H, ^{13}C, and 2D NMR) spectra were recorded on a Bruker Avance (III) 400WB spectrometer. Chemical shifts were expressed in parts per million (ppm), *J* values were in Hertz (Hz).

2.1 Bioassay-guided fractionation

The crude dichloromethane extract of the rhizomes was fractionated by silica gel column chromatography and eluted with gradually increasing polarity solvent, starting from 100% hexane to 100% AcOEt, and then to 100% MeOH to obtain ten fractions (**FA**–**FJ**) based on TLC analyses. These fractions were evaluated for their antibacterial activity against *Staphylococcus aureus* ATCC 6538P. Fractions **FE** and **FG** were the most active, both exhibiting minimum inhibitory concentration (MIC) of 400 μgmL^{-1} and minimum bactericidal concentration (MBC) of 800 μgmL^{-1}. Fraction **FE** was further separated by silica gel column chromatography and eluted with 5%–60% AcOEt in hexane, gradually increasing the polarity by adding 5% AcOEt and using 500 mL each time to provide 7 sub-fractions (**FE.A**–**FE.G**). Fraction **FG** was separated by silica gel column chromatography and eluted with 5%–70% AcOEt in hexane to obtain 13 sub-fractions (**FG.A**–**FG.M**).

2.2 Purification of pure compounds

Petasol (**P1**) was isolated from **FF** using column chromatography eluted with 33%–50% ethyl acetate in hexane, leading to 7 sub-fractions (**FF.A**–**FF.G**). Next, **FF.C** and **FF.D** were combined and further purified by preparative TLC using 10% acetone in dichloromethane as a mobile phase to provide **P1** (6.1 mg) as a yellow viscous oil. Another pure compound was isolated from **FG.B** by recrystallization with hexane, yielding (*E*)-15,16-dinorlabda-8(17),11-dien-13-one (**P2**) as a white solid (6.4 mg). Moreover, the compound **P3** was isolated from **FG.H** by Prep-TLC using 100% dichloromethane as a mobile phase. The NMR spectra were presented in **Appendix D**.

3. GC-MS analysis

The fractions and sub-fractions were dissolved in HPLC grade of ethyl acetate from RCI Labscan (Samutsakorn, Thailand). The concentration of the sample from 1 ppm to 4 ppm was used to analyze volatile compositions. The analyses of volatile compositions

were performed using GC/MS instrument (7890 A-7000, Agilent Technologies, Santa Clara, CA) with an HP-5MS column (30 m×0.25 mm i.d., 0.25 μm film thickness, J&W Scientific, USA) using ultra-high purity helium (99.999%) as the carrier gas at a constant flow rate of 1.0 mL min^{-1}, an injection volume of 1 μL (splitless) and injector temperature of 280 °C were used. The GC temperature program consisted of an initial temperature of 60 °C with an increase rate of 4°C min^{-1} to reach a final temperature of 320 °C, which was held for 5 min. MS ion source temperature was set at 230°C. The electron ionization was performed at 70 eV. The mass spectra were acquired over the mass range of 30–300 Da. Data acquisition and peak integration were performed using ChemStation software (Agilent). Data were further processed using Microsoft Excel 2017. Individual components were identified by comparison of both their experimental mass spectra and retention indices (RI) with the NIST 2017 (National Institute for Standards and Technology) database. Experimental RI values were calculated for all volatile compounds by using a homologous series of alkanes (C_7 - C_{30} purchased from Sigma–Aldrich, St. Louis, MO) recorded under the same operating conditions. Since the temperature programmed separation was performed, RI values were calculated according to

$$\mathrm{RI} = 100n + 100\left(\frac{t_{\mathrm{R}(i)} - t_{\mathrm{R}(n)}}{t_{\mathrm{R}(n+1)} - t_{\mathrm{R}(n)}}\right)$$

where $t_{R(i)}$ is retention time of peak *i*. $t_{R(n)}$ and $t_{R(n+1)}$ are retention time values of alkane standards with carbon numbers *n* and *n*+1, respectively, bracketing the peak *i* [33] Retention indices are applied in order to construct database with quality and reliability since the system is independent and long-term reproducible even with different places and times of experiments. Library searches are important to confirm peak identities obtained from the MS data. This is especially useful for the identification of isomeric

compounds, such as sesquiterpene hydrocarbons with either the same or similar mass spectra.

4. Antibacterial activity determination

Staphylococcus aureus ATCC 6538P, *Micrococcus luteus* ATCC 4698, *Escherichia coli* ATCC 25922, and *Pseudomonas aeruginosa* Strain Boston 41501 ATCC 27853 were purchased from Biomedia (Thailand). The analytical grade (AR.) of dimethyl sulfoxide (DMSO) from RCI Labscan (Samutsakorn, Thailand) was used to dissolve the crude sample. Mueller Hinton Broth (MHB) from Himedia was used to sample dilution. Streptomycin, amoxicillin and ciprofloxacin from TCI chemicals (Tokyo, Japan) were used as a commercial standard drug. The bacterial cell viability was measured by using resazurin sodium salts form TCI chemicals (Tokyo, Japan). The living bacteria cells were oxidized the blue color of resazurin to purple color of resofurin is shown in **Figure 2.1**.[36]

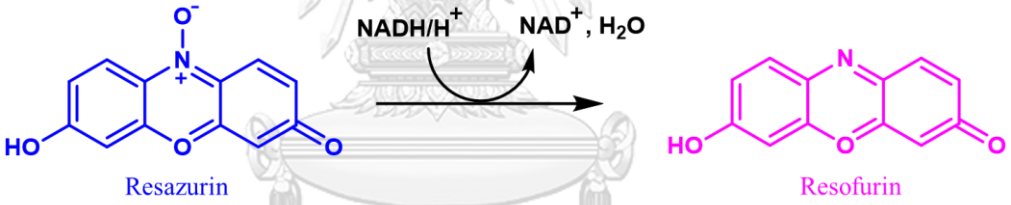


Figure 2.1: Reduction of resazurin to produce resofurin

4.1 The minimum inhibitory concentrations (MIC)

The minimum inhibitory concentrations (MIC) for each fraction/sub-fraction against *Staphylococcus aureus* ATCC 6538P, *Micrococcus luteus* ATCC 4698, *Escherichia coli* ATCC 25922, and *Pseudomonas aeruginosa* Strain Boston 41501 ATCC 27853 were determined using 96-well plate microdilution method modified from the Manual of Antimicrobial Susceptibility Testing, American Society for Microbiology.[37] Briefly, 800 μgmL^{-1} solution of each crude extract was prepared by dissolving the crude extracts in the 5% v/v solution of dimethyl sulfoxide (DMSO) in Mueller Hinton Broth (MHB). Serial two-fold dilutions of crude extract samples were performed to obtain the concentrations

of 800, 400, 200, 100, 50, 25, 12.5, and 6.25 μgmL^{-1} in 50 μL of 5% v/v DMSO/MHB. Streptomycin, amoxicillin and ciprofloxacin were used as a control group. Four to five isolated colonies of bacteria were selected and cultured in MHB at 37°C for 4–18 h. The turbidity of the bacterial culture was determined by comparison with 0.5 McFarland standard (HiMedia, Dindori, Nashik, India). Then, the bacterial cell density was adjusted with MHB to 10^6 $CFUmL^{-1}$. Within 15 min of adjusting the inoculum, 50 μL of the bacterial suspension were added into each well of the 96-well plates containing 50 μL of either the crude extract solutions or control group solutions. This resulted in the final bacterial concentration of 5×10^5 $CFUmL^{-1}$ in the total volume of 100 μL of 5% v/v DMSO/MHB. The 5×10^5 $CFUmL^{-1}$ bacterial solution in 5% v/v DMSO/MHB was used as a positive control group. A solution of 5% DMSO/MHB was used as a negative control group. After the addition of bacterial cells, the microplates were wrapped with parafilm to prevent the dehydration and then, incubated at 37°C for 24 h. At the 24-h post inoculation, the MIC values were investigated by adding 30 μL of 0.015% w/v resazurin aqueous solution into each well and incubating at 37°C for 1 h. The lowest concentrations of crude extracts with no color change of blue resazurin were scored as MIC values.

4.2 The minimum bactericidal concentration (MBC)

The minimum bactericidal concentration (MBC) was determined by plating directly the content of each well with a concentration at the MIC and one with higher than the MIC value. Streak of the contents from the MIC well to the higher concentration on Mueller Hinton Agar (MHA) was then incubated at 37°C for 24 h. The MBC value was determined when there is no colony growth from the directly plated contents of the directly plated contents of the wells.

CHAPTER III

RESULTS & DISCUSSIONS

1. Bioassay-guided fractionation

1.1 Fractionation of crude extract

The crude dichloromethane extract of *G. schomburgkii* Hook.f. rhizomes was isolated by column chromatography to provide ten fractions (**FA-FJ**) as shown in **Figure 3.1**. These fractions were evaluated for their antibacterial activity against *S. aureus*. Fractions **FA** and **FB** were inactive against *S. aureus*, probably due to the major components in these fractions consist of compounds with low polarity such as long chain hydrocarbons. Fractions **FE** and **FG** were the most active against *S. aureus*. Strangely, fraction **FF** was inactive, even though it is the fraction between **FE** and **FG** that were the most active fractions. This suggests that the major components of these fractions are different as can be roughly visualized by the TLC analysis as shown in **Figure 3.1**. The most active fractions, **FE** and **FG** (MIC = 400 μg mL^{-1} and MBC = 800 μg mL^{-1}), were selected for further isolation.

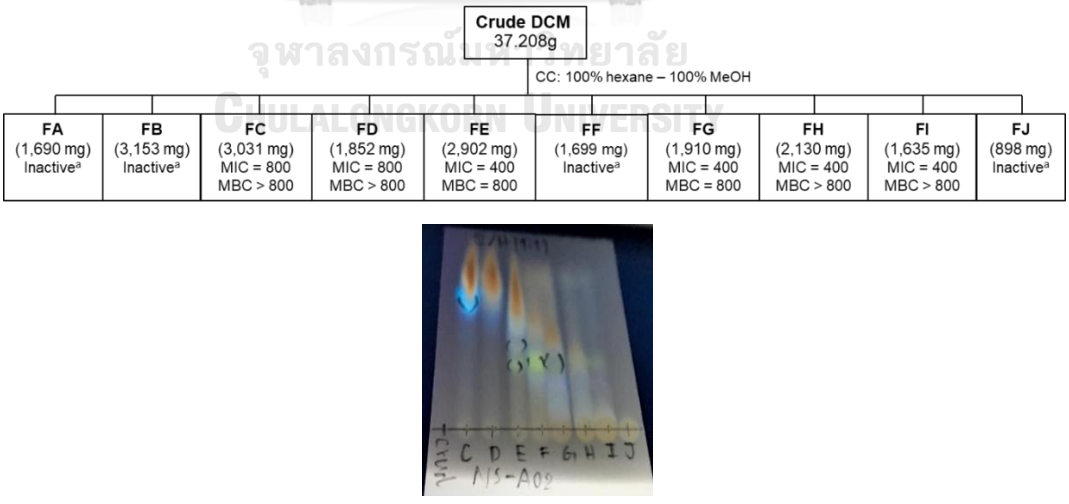


Figure 3.1: Bioassay-guided fractionation of **crude DCM** extract of *G. schomburgkii* and their biological activities against *S. aureus*; MIC and MBC were reported in the unit of μg mL^{-1}. [a]Inactive means MIC > 800 μg mL^{-1}.

1.2 Fractionation of fraction FE

Fraction **FE** was isolated to give seven sub-fractions (**FE.A-FE.G**) which were tested for their antibacterial activity (**Figure 3.2**). The most active sub-fraction **FE.D** (MIC = 200 μg mL^{-1} and MBC = 200 μg mL^{-1}) was further separated. However, the isolation was difficult due to the streaking of the compounds in column chromatography as can be visualized by the TLC analysis (**Figure 3.2**). Moreover, the UV inactivity of the compound hindered the separation even further, and hence we cannot isolate any pure compound from this fraction.

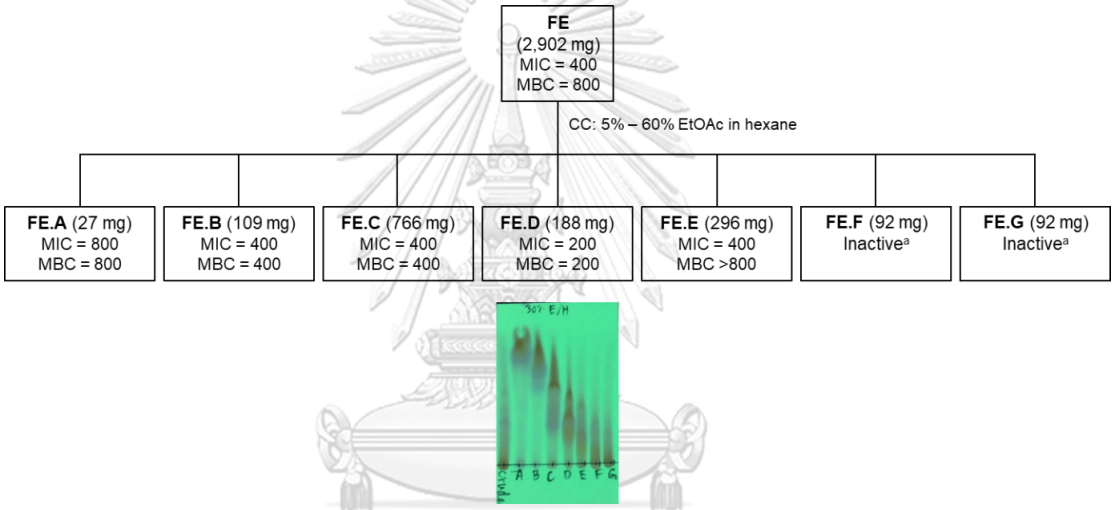


Figure 3.2: **FE** Fractionation and TLC data

1.3 Fractionation of fraction FF

Although fraction **FF** is inactive against *S. aureus*, but since it is next to the other two most active fractions, **FE** and **FG**; therefore, we also attempted to isolate compounds in this fraction. The fractionation by column chromatography gave seven sub-fractions as shown in **Figure 3.3**. According to the TLC analysis, both **FF.C** and **FF.D** showed bright yellow spots under UV irradiation; therefore, these two fractions were combined for further isolation. The fraction **FF.CD** was selected to further isolation into two sub-fractions, including **FF.CDA** and **FF.CDB**. Lastly, **FF.CDB** was purified by Prep-TLC to provide **P1** as a yellow viscous oil (6.1 mg). Using spectroscopic techniques such as

^{1}H, ^{13}C, and 2D NMR (**Appendix D**), the chemical structure of **P1** was identified as petasol when compared with the literature values [38] as tabulated in **Table1**. The sub-fractions were not evaluated for their antibacterial activity since fraction **FF** itself is inactive against *S. aureus*.

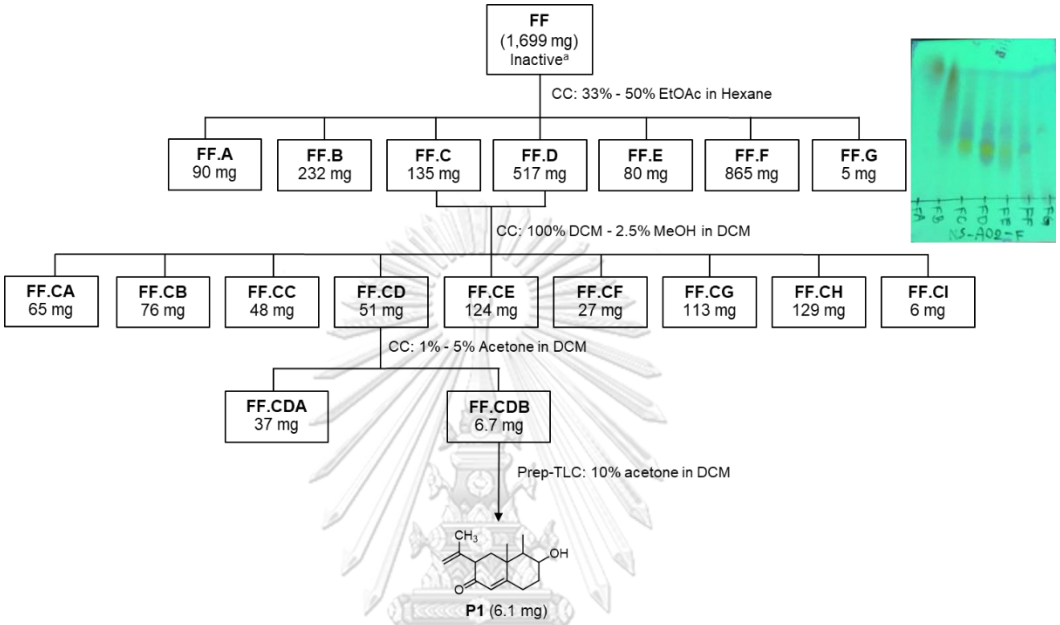


Figure 3.3: FF Fractionation and TLC data; **P1** purification

Table 3.1: Chemical shift comparison between petasol (**P1**) and reference

^{13}C NMR			^{1}H NMR	
Carbon position	P1[a]	Reference[b]	P1[c]	Reference[b]
C-8	198.8	198.6	-	-
C-10	167.7	167.6	-	-
C-11	143.6	143.4	-	-
C-9	124.6	124.4	5.78 (1H, s)	5.77 (1H, d, *J* = 1.8 Hz)
C-12	114.5	114.3	4.99 (1H, s) 4.82 (1H, s)	4.98 (1H, br.t, *J* = 1.5 Hz) 4.82 (1H, br.s)
C-3	71.3	71.1	3.62 (1H, td, *J* = 10.8, 4.4 Hz)	3.62 (1H, td, *J* = 10.7, 4.4 Hz)

C-4	50.5	50.30	1.34 (1H, s)	1.33 (1H, m)
C-7	50.5	50.26	3.11 (1H, dd, *J* = 14.4, 4.6 Hz)	3.10 (1H, dd, *J* = 14.3, 4.7 Hz)
C-6	41.9	41.7	2.00 (1H, d, *J* = 4.6 Hz) 1.88 (1H, s)	2.02 (1H, dd, *J* = 13.1, 4.8 Hz) 1.87 (1H, t, *J* = 13.6 Hz)
C-5	39.9	39.7	-	-
C-2	35.3	35.2	2.16 (1H, dd, *J* = 8.5, 3.7 Hz) 1.45 (1H, dd, *J* = 14.0, 4.5 Hz)	2.16 (1H, m) 1.44 (1H, m)
C-1	31.1	30.9	2.43 (1H, dd, *J* = 14.2, 5.0 Hz) 2.37 – 2.35 (1H, m)	2.45 (1H, tdd, *J* = 15.0, 5.1, 2.0 Hz) 2.34 (1H, ddd, *J* = 15.0, 4.9, 2.7 Hz)
C-13	20.2	20.0	1.74 (3H, s)	1.74 (3H, br.s)
C-14	17.4	17.2	1.18 (3H, s)	1.17 (3H, s)
C-15	10.6	10.4	1.08 (3H, d, *J* = 6.7 Hz)	1.08 (3H, d, *J* = 6.7 Hz)
[a]100 MHz in $CDCl_3$, [b]$CDCl_3$, [c]400 MHz in $CDCl_3$				

1.4 Fractionation of fraction FG

The fractionation of **FG** afforded thirteen sub-fractions. Crystallization occurred when the sub-fraction **FG.B** was collected during column chromatography. **FG.B** was recrystallized in hexane to provide **P2** as a white crystal (6.4 mg). Using spectroscopic techniques (**Appendix D**), the structure of **P2** was identified as (*E*)-15,16-dinorlabda-8(17),11-dien-13-one. The proton and carbon chemical shifts were compared with the literature reported values [39] as tabulated in **Table 3.2**.

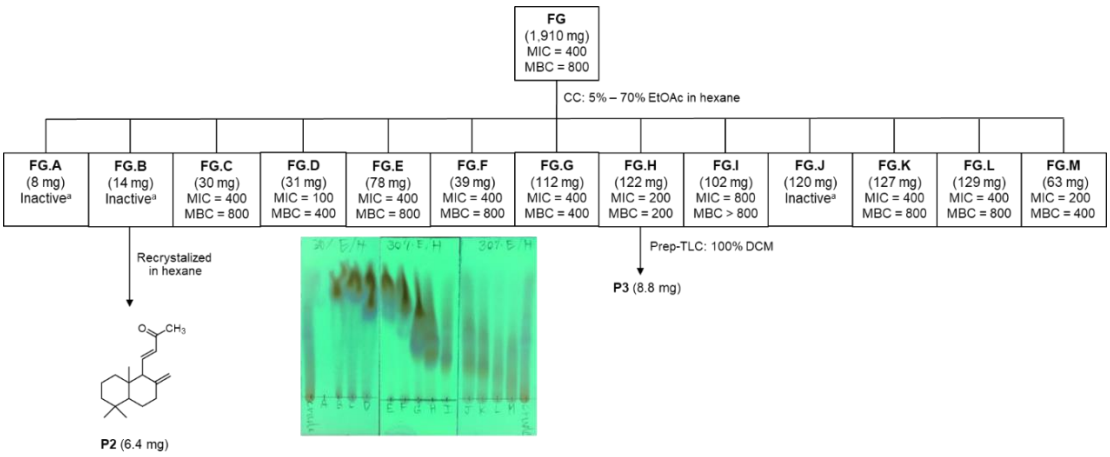


Figure 3.4: Fractionation and TLC data; **P2** and **P3** purification

Table 3.2: Chemical shift comparison between (*E*)-15,16-dinorlabda-8(17),11-dien-13-one (**P2**) and reference

^{13}C NMR			^{1}H NMR	
Carbon position	P2[a]	Reference[a]	P2[b]	Reference[b]
C-13	198.2	197.9	-	-
C-8	148.8	148.5	-	-
C-11	146.7	146.5	6.87 (1H, dd, *J* = 15.9, 10.3 Hz)	6.87 (dd, *J* = 15.8, 10.3 Hz)
C-12	133.7	133.5	6.07 (1H, d, *J* = 15.9 Hz)	6.07 (d, *J* = 15.8 Hz)
C-17	108.8	108.6	4.40 (1H, s) 4.79 (1H, s)	4.41 (d, *J* = 1.6 Hz) 4.79 (d, *J* = 1.6 Hz)
C-9	61.0	60.8	-	-
C-5	54.7	54.4	-	-
C-3	42.3	42.0	-	-
C-7	41.0	40.9	-	-
C-10	39.5	39.3	-	-
C-1	36.8	36.6	-	-
C-4	33.7	33.6	-	-

C-18	33.7	33.6	0.84 (3H, s)	0.85 (s)
C-14	27.3	27.2	2.27 (3H, s)	2.27 (s)
C-6	23.4	23.2	-	-
C-19	22.1	21.9	0.89 (6H, s)	0.89 (s)
C-2	19.2	19.0	-	-
C-20	15.3	15.1	0.89 (6H, s)	0.90 (s)
[a]100 MHz in $CDCl_3$, [b]400 MHz in $CDCl_3$				

The preliminary screening of sub-fractions **FG.A-FG.M** against *S. aureus* demonstrated that **FG.H** was the most active sub-fraction (MIC and MBC = 200 μg mL^{-1}). However, the attempt to isolate pure compound in this sub-fraction was rather unsuccessful. We managed to isolate a compound (**P3**); however, we were unable to purify it from impurities and the structural elucidation was ambiguous. The ^{1}H and ^{13}C NMR (**Appendix D**) showed the peak at 0.75, 0.82, 0.89, 440, 4.86, and 9.40 ppm and 14.56, 21.88, 24.78, 148.23, and 193.91 ppm, respectively, suggesting that this compound should contain four methyl groups, a germinal alkene, and an aldehyde group. This correlates well with the GC-MS analysis of **P3**, demonstrating that this is a mixture between α-kessyl acetate (**A**) and (*E*)-15,16-dinorlabda-8(17),11-dien-13-one (**B**) (**Figure 3.5**). However, ^{1}H and ^{13}C NMR spectra of **P3** does not fully match with those of **B** reported in the literature [40, 41] (**Table 3.3**).

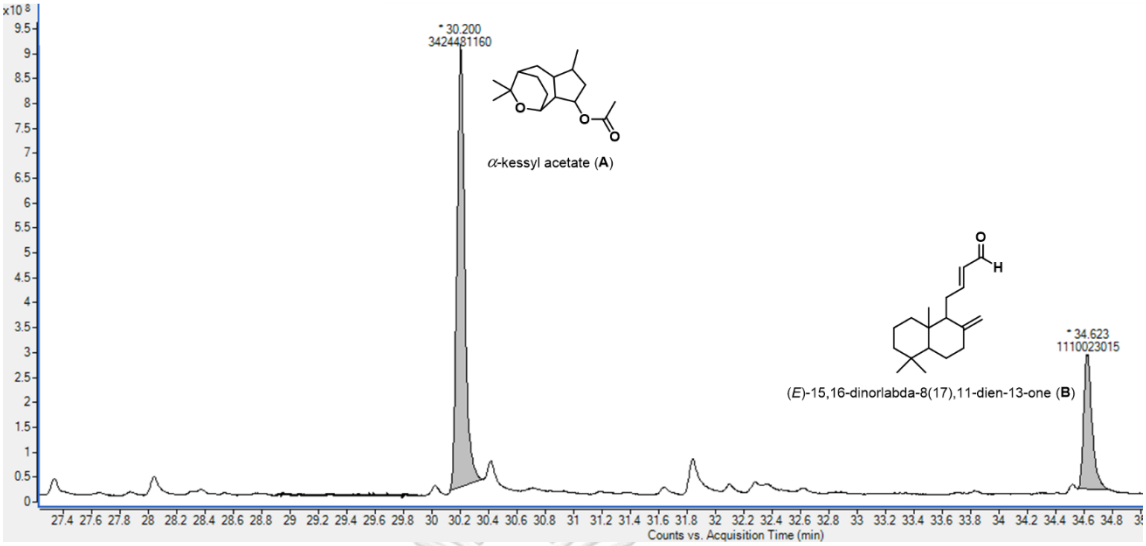


Figure 3.5: The ion-chromatogram of **P3**

Table 3.3: Chemical shift comparison between **P3** and (*E*)-15,16-dinorlabda-8(17),11-dien-13-one (**B**) reference

^{13}C NMR			^{1}H NMR	
Carbon position	P3[a]	Reference[c]	P3[b]	Reference[b]
C-10	14.6	14.4	0.75 (3H,s)	0.75 (3H,s)
C-4	21.9	21.7	0.82 (3H,s)	0.83 (3H,s)
C-4	33.7	33.5	0.89 (3H,s)	0.90 (3H,s)
C-3	42.2	42.0	1.11 (1H,s)	1.09 ($1H_{ax}$,ddd, J = 13, 13, 4 Hz)
C-5	55.6	55.4	1.13 (1H,s)	1.13 ($1H_{ax}$,dd, J = 13, 2.5 Hz)
C-1	39.4	39.2	1.20 (1H,s)	1.20 ($1H_{ax}$,ddd, J = 13, 13, 4 Hz)
C-6	24.3	24.1	1.37 (2H,d, J = 4.1 Hz)	1.36 ($1H_{ax}$,dddd, J = 13, 13, 13, 4 Hz)
C-3	42.2	42.0	1.44 (1H,s)	1.43 ($1H_{eq}$, ddd, br, J = 13, 3, 3 Hz)
C-2	19.5	19.3	1.53 (1H,s)	1.53 ($1H_{eq}$,ddd, br, J = 13, 4, 3 Hz)
C-2	19.5	19.3	1.58 (2H,d, J = 3.5 Hz)	1.59 ($1H_{ax}$, ddddd, J = 13, 13, 13, 4, 3 Hz)
C-1	39.4	39.2	1.71 (1H,s)	1.72 ($1H_{eq}$,d, br,. J = 13 Hz)
C-6	24.3	24.1	1.76 (1H,s)	1.75 ($1H_{eq}$, dddd, J = 13, 5, 2.5, 2.5 Hz)

C-9	56.6	55.9	1.92 (2H,d, *J* = 11.2 Hz)	1.90 ($1H_{ax}$, dd, br, *J* = 12, 4 Hz.)
C-7	38.0	37.9	2.00 (1H,d, *J* = 5.3 Hz)	2.01 ($1H_{ax}$, ddd, br, *J* = 13, 13, 5 Hz.)
C-7	38.0	37.9	2.38 (1H,s)	2.39 ($1H_{eq}$, ddd, *J* = 13, 4, 2 Hz)
C-11	29.7	27.9	2.43 (1H,s)	2.43 (1H, dddd, *J* = 16, 11, 6.5, 1.5 Hz)
C-11	29.7	27.9	2.56 (2H,dd, *J* = 6.0, 3.0 Hz)	2.55 (1H,dddd, *J* = 16, 6.5, 4, 1.5 Hz)
C-17	108.0	108.0	4.40 (1H,s)	4.46 (1H, dd, *J* = 1.5, 1 Hz)
C-17	108.0	108.0	4.86 (1H,s)	4.86 (1H,dd, *J* = 1.5, 1 Hz)
C-13	135.9	133.4	-	6.10 (1H,dddd, *J* = 16, 8, 1.5, 1.5 Hz)
C-12	159.5	160.2	6.70 (1H,t, *J* = 6.5 Hz)	6.82 (1H,ddd, *J* = 16, 6.5, 6.5 Hz)
CHO	193.9	194.2	9.40 (1H,s)	9.45 (1H,d, *J* = 8 Hz)
C-8	148.2	147.8	-	-
C-10	39.8	39.6	-	-
C-4	33.7	33.5	-	-
[a]100 MHz, in $CDCl_3$, [b]400 MHz in $CDCl_3$, [c] 270 MHz with DEPT in $CDCl_3$				

2. GC-MS Analysis of fractions and sub-fractions

2.1 Total ion chromatograms of DCM extract and FA-FJ

The ion chromatograms from GC-MS analysis of **crude DCM** extract and **FA-FJ** are shown in **Figure 3.6.** The major volatile components were identified as γ-bicyclohomofarnesal and **P2** in most of the fractions. Note that **P1** was identified by GC-MS as an unknown with the experimental retention index (RI) = 2026. This compound can only be found in **FE** and **FF**.

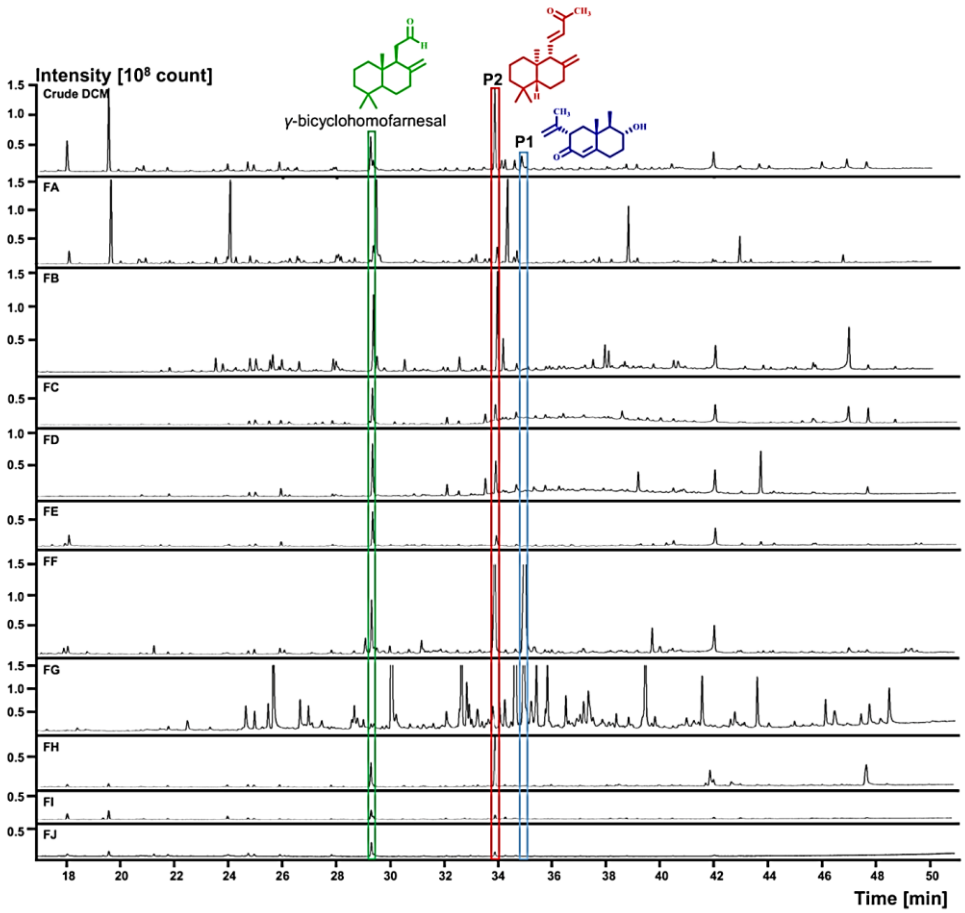


Figure 3.6: Total ion chromatograms of **DCM** extract and **FA-FJ** from *G. schomburgkii* rhizomes

2.2 Selected volatile composition of crude DCM and FA-FJ

The volatile compounds of crude **DCM** and **FA-FJ** were selected to investigate their quantity in each fraction as shown in **Table 3.4**. From the table, the compounds were categorized into four groups including oxygenated monoterpene (OM), sesquiterpene hydrocarbon (SH), oxygenated sesquiterpene (OS), and diterpene. The groups with the highest peak area percentages in the fractions are as followed: oxygenated monoterpene (3.9%) in crude **DCM**, sesquiterpene hydrocarbon (36.3%) in **FA**, oxygenated sesquiterpene (18.5%) in **FB**, and diterpene (51.9%) in **FH**. **FA** contains the highest sesquiterpene hydrocarbon content correlated with the group of the least polar obtained in the first fraction from the column chromatography. **FB** has the highest

content of oxygenated sesquiterpenes which are more polar than that in **FA**. However, 13-nor-eremophil-1(10)-en-11-one which should have been found in **FB** was only identified in crude **DCM**. This indicates either false positive identification of this compound in the crude (due to compound coelution) or the compound loss during the separation with column chromatography. On the other hand, some volatile compounds such as longifolenaldehyde were identified in **FA-FJ** (not in crude **DCM**). This suggests that crude **DCM** contains more coeluting peaks in GC-MS hindering these compound identification. This can be solved by analysis of the compounds after fractionation reducing the number of co-eluting peaks. In addition, some compounds were identified in both crude DCM and the fractions which are (*E*)-15,16-dinorlabda-8(17),11-dien-13-one (**P2**). Moreover, the average match score for all the identified compounds was 688 which can be considerably low. However, our chemical identification was respectable due to the confirmation by RI with the average difference (from the literature data) of 7 According to the peak area analysis, recoveries of the compounds identified in both the crude and the fractions were within the range of 12-137%, see the related data provided in **Appendix A (Table A.1)**.

Table 3.4: Selected volatile composition of crude **DCM** and **FA-FJ**

No.	Compound[a][b]	%Percentage of total peak area (%Recovery)[c]										
		DCM	FA	FB	FC	FD	FE	FF	FG	FH	FI	FJ
Oxygenated monoterpene												
1	Linderol	3.9	-	1.4	3.3	0.7	0.5	0.1	-	0.8	3.1	-
2	2-Hydroxycineol	-	-	-	-	-	0.9	-	-	-	-	-
Sesquiterpene hydrocarbon												
4	Cyperene	7.2	4.3	-	-	-	-	-	-	1.4	12.7	6.3
5	*cis*-β-Copaene	-	-	0.1	-	-	-	-	-	1.8	-	-
6	Guaia-6,9-diene	19.1	31.1	-	-	-	-	-	-	-	18.0	-
7	Selina-5,11-diene	0.2	-	-	-	-	-	-	-	-	3.0	-

8	Himachala-2,4-diene	0.4	0.9	-	-	-	-	-	-	-	-	11.4
Oxygenated sesquiterpene												
9	Caryophyllene oxide	0.4	2.1	2.1	-	-	-	-	-	-	-	-
10	Boronia butenal	-	0.9	0.2	-	-	-	-	-	-	-	-
11	Humulene epoxide II	-	1.9	0.8	-	-	-	-	-	-	-	-
12	13-nor-Eremophil-1(10)-en-11-one	1.9	-	-	-	-	-	-	-	-	-	-
13	Longifolenaldehyde	-	2.4	2.1	1.4	1.3	0.9	0.3	1.2	1.0	2.8	5.4
14	Caryophylladienol II	-	-	2.2	-	-	-	-	-	-	-	-
15	Pogostole	0.2	0.7	1.5	1.4	-	-	-	-	-	-	-
16	Neointermedeol	0.3	-	2.3	-	-	-	-	-	-	-	-
17	(–)-Isolongifolol, methyl ether	1.9	1.6	1.6	1.1	2.6	3.0	0.5	1.3	1.2	-	-
18	Cyperenone	0.5	-	1.5	-	-	-	-	-	-	-	-
19	3-Hydroxy-5,6-epoxy-β-ionone	-	-	-	-	-	-	-	0.9	-	-	-
20	Isolongifolol	-	1.1	2.1	0.3	0.6	-	-	0.8	-	-	-
21	α-Cyperone	0.4	1.5	-	0.7	-	-	-	-	-	-	-
22	Dehydrovomifoliol	-	-	-	-	-	-	1.7	-	-	-	-
23	Dehydrofukinone	-	-	2.1	-	-	-	-	-	-	-	-
24	8α-Hydroxy-Eremophila-1(10),11-dien-9-one	-	-	-	-	-	1.8	-	-	-	-	-
25	Alantolactone	-	-	-	2.8	3.9	-	-	1.8	-	2.0	-
26	β-Cyclocostunolide	-	-	-	-	-	-	-	1.5	-	-	-
Diterpene												
27	γ-Bicyclohomofarnesal	6.3	2.9	11.1	14.2	18.5	24.2	5.2	20.9	-	18.7	28.8
28	(*E*)-15,16-Dinorlabda-8(17),12-dien-14-al	-	1.5	0.4	-	-	0.3	-	-	0.8	-	-
29	(*E*)-15,16-Dinorlabda-8(17),11-dien-13-one (**P2**)	19.2	5.4	15.3	7.2	12.9	7.6	20.9	28.2	39.0	9.6	10.4
30	Androst-5-en-4-one	-	-	-	0.9	-	-	-	-	-	-	-

31	Epimanool	-	0.3	0.2	-	0.8	0.6	0.3	2.6	0.8	-	-
32	Isoretinene a	-	-	-	-	-	1.4	-	-	-	-	-
33	Sclareol	0.5	-	-	-	-	1.3	2.6	0.1	-	-	-
Oxygenated monoterpene [%]		3.9	-	1.7	4.7	0.7	1.4	0.1	-	0.8	3.1	-
Sesquiterpene hydrocarbon [%]		26.9	36.3	0.1	-	-	-	-	-	3.2	33.7	17.7
Oxygenated sesquiterpene [%]		5.6	12.2	18.5	7.7	8.4	5.7	2.5	7.5	2.2	4.8	5.4
Diterpene [%]		26.0	10.1	27.0	22.3	32.2	35.4	29.0	51.8	51.9	28.3	39.2
Percentage of constituents identified		62.4	58.6	47.3	34.7	41.8	42.5	31.6	59.3	58.1	69.9	62.3
Number of identified constituents		28	25	45	21	10	13	19	19	11	11	5
Number of unidentified constituents		31	40	38	45	25	34	41	45	25	17	14
[a] Tentative identification with match score above 600. [b] Linear straight-chain hydrocarbons were excluded from the analysis. [c] -, Not identified.												

2.3 Selected volatile composition of FE and FE.A-FE.G

The volatile compositions of **FE** and their sub-fraction **FE.A- FE.G** were shown in **Table 3.5**. The volatile compounds were categorized into three groups including oxygenated sesquiterpene (OS), oxygenated diterpene (OD), and miscellaneous. The fractions with the highest contents of the compounds in these groups were **FE.G** (43.2%), **FE.F** (45.4%), and **FE.A** (21.3%). However, antibacterial activities of these sub-fractions (**FE.A-FE.G**) were lower than **FE.D** as show by the lower MIC and MBC values in **Table 3.7**. The major volatile compounds of **FE.D** were found to be 4,8-β-epoxy-caryophyllene (9.8% of the total peak area), (*E*)-labda-8(20),13-diene-15,19-diol (6.6%), methyl isocostate (5.3%), retinal (3.0%), isoretinene a (2.4%), and (*E*)-labda-8(17),12-diene-15,16-dial (2.4%). In addition, (*E*)-labda-8(17),12-diene-15,16-dial has been reported to exhibit potent antifungal activity.[42] Methyl isocostate was reported to display high cytotoxicity against various cancer cell lines.[43] This suggests that these two

compounds might be important for the higher bioactivity of **FE.D**. Recoveries of the compounds identified in both the crude and the fractions were within the range of 0-106%, see the related data provided in **Appendix A (Table A.2)**. In addition, the average match score for all the identified compounds was 667 with the average RI difference of 11 for the data in **Table 3.5**.

Table 3.5: Selected volatile composition of **FE** and **FE.A-FE.G**

No.	Compound [a][b]	Percentage of total peak area [%][c]							
		FE	FE.A	FE.B	FE.C	FE.D	FE.E	FE.F	FE.G
Oxygenated sesquiterpene									
1	4,8-β-Epoxy-caryophyllane	-	-	-	-	9.8	-	-	-
2	13-nor-Eremophil-1(10)-en-11-one	-	3.3	-	-	-	-	-	-
3	γ-Costol	-	3.2	-	-	-	-	-	-
4	β-Costol	-	-	-	-	-	14.4	-	43.2
5	Methyl isocostate	-	-	-	-	5.3	-	-	-
6	Methyl valerate	-	-	-	-	1.4	-	-	-
7	8α-Hydroxy-eremophila-1(10),11-dien-9-one	1.8	0.2	-	-	-	-	-	-
8	Valerenic acid	-	-	0.2	2.1	0.4	-	-	-
9	Corymbolone	-	-	0.3	-	0.9	-	-	-
10	6-(1-Hydroxymethylvinyl)-4,8a-dimethyl-3,5,6,7,8,8a-hexahydro-1H-naphthalen-2-one	-	-	0.2	-	0.4	0.8	-	-
Oxygenated diterpene									
11	γ-Bicyclohomofarnesal	24.2	22.3	-	-	-	-	33.4	-
12	(*E*)-15,16-Dinorlabda-8(17),11-dien-13-one	7.6	10.1	0.2	6.2	-	2.0	-	11.5
13	Coronarin E	-	3.1	-	-	-	-	-	-
14	Isoretinene a	1.4	-	1.8	-	2.4	-	-	-
15	Sclareol	1.3	-	-	-	-	3.4	12.0	-
16	(*E*)-Labda-8(20),13-diene-15,19-diol	-	-	-	6.7	6.6	-	-	-
17	(*E*)-Labda-8(17),12-diene-15,16-dial	-	-	-	-	2.4	-	-	-
18	Retinal	-	1.1	-	3.7	3.0	-	-	-
19	7α,17α-Dimethyl-5β-androstane-3α,17β-diol	-	-	2.4	-	-	-	-	-
20	Calusterone	-	-	34.4	-	-	-	-	-
Miscellaneous									

21	Methyl tridecyl ketone	-	-	2.3	-	-	-	-	-
22	Hexadecanoic acid	-	-	-	5.5	-	-	-	-
23	Ethyl palmitate	-	9.4	-	-	-	-	-	-
24	2-*cis*,*cis*-9,12-Octadecadienyloxyethanol	-	-	-	4.3	-	-	-	-
25	Ethyl linolenate	-	6.9	-	-	-	-	-	-
26	Ethyl octadecanoate	-	1.9	-	-	-	-	-	-
27	Eicosyl acetate	-	1.7	-	-	-	-	-	-
28	Docosyl acetate	-	1.4	-	-	-	-	-	-
Oxygenated sesquiterpene [%]		1.8	6.7	0.7	2.1	18.2	15.2	-	43.2
Oxygenated diterpene [%]		34.5	36.6	38.8	16.6	14.4	5.4	45.4	11.5
Miscellaneous [%]		-	21.3	2.3	9.8	-	-	-	-
Percentage of constituents identified		36.3	64.6	41.8	28.5	32.6	20.6	45.4	54.7
Number of identified constituents		13	21	13	6	10	4	2	2
Number of unidentified constituents		34	31	23	14	36	28	7	13

[a] Tentative identification with match score above 600. [b] Linear straight-chain hydrocarbons were excluded from the analysis. [c] -, Not identified.

2.4 Selected volatile composition of FG and FG.A-FG.M

The volatile compositions of **FG** and there sub-fractions **FG.A- FG.M** were provided in **Table 3.6**. From the table, the volatile compound was categorized into three groups of monoterpene, sesquiterpene, and diterpene which were mostly contained in the fractions **FG.E** (31.1%), **FG.H** (57.5%), and **FG.M** (82.7%). From the antibacterial screening, **FG.D** and **FG.H** exhibited greatest antibacterial activity as indicated by the lower MIC and MBC, respectively, in **Table 3.6**. Noticeably, (*E*)-15,16-dinorlabda-8(17),11-dien-13-one (**P2**) and γ-bicyclohomofarnesal were found as major components in both **FG.D** and **FG.H** (29.6% and 12.6%, respectively for **FG.D**, and 11.6% and 4.1%,

respectively for **FG.H**). Nevertheless, these two major components were also observed in the other sub-fractions with the lower antibacterial activity. Therefore, these major components might not be responsible for the antibacterial activity. On the other hand, α-kessyl acetate and zederone were only found in **FG.H** in high percentages (24.9% and 9.1%, respectively). Although no biological activity has been reported for α-kessyl acetate, zederone has been demonstrated to exhibit antibacterial [44], and antioxidant activities. [45] Likewise, some major components such as clovanediol, ledene oxide-(I), alantolactone, and 8α,11-elemadiol showed significant correlation with the antibacterial activity in each sub-fraction supported by the data in **Table 3.7**. Moreover, there are several bioactivities reported for alantolactone including antimicrobial, anti-inflammatory, and anti-proliferative effects on several cancer cell lines.[46] 8α, 11-Elemadiol has also been reported to exhibit antifungal activity against eczema fungi and insecticidal activity. [47] Recoveries of the compounds identified in both the crude and the fractions were within the range of 10-160%, see the related data provided in **Appendix A (Table A.3)** In addition, the average match score for all the identified compounds was 682 with the average RI difference of 8 for the data in **Table 3.6.**

Table 3.6: Selected volatile composition of **FG** and **FG.A-FG.M**

No.	Compound[a][b]	Percentage of total peak area [%][c]												
		FG	FG.B	FG.C	FG.D	FG.E	FG.F	FG.G	FG.H	FG.I	FG.J	FG.K	FG.L	FG.M
Monoterpene														
1	*trans*-Ascaridol glycol	-	-	-	-	-	-	0.1	0.3	-	-	1.4	-	-
2	(+)-Pinanediol	-	-	-	-	-	-	1.5	-	-	-	-	-	-
3	Sobrerol	-	-	-	-	7.5	-	-	2.4	-	-	12.8	-	-
4	1,2,4-Trihydroxy menthane	-	-	-	-	-	-	-	0.9	-	-	-	-	-
5	Sobrerol 8-acetate	0.6	-	-	-	23.6	-	-	-	-	-	-	-	-
Sesquiterpene														
6	(±)-Gymnomitrene	-	-	-	3.0	-	-	-	-	-	-	-	-	-

7	Caparratriene	-	-	-	-	-	-	-	-	-	1.1	-	-	-
8	4,8-β-Epoxy-caryophyllane	-	-	-	-	-	-	-	-	1.6	-	-	-	-
9	9-Cedranone	-	-	1.2	-	-	-	-	-	-	-	-	-	-
10	Longifolenaldehyde	1.2	-	5.9	-	-	-	-	0.3	3.1	1.6	-	-	0.9
11	Neointermedeol	-	-	-	-	-	-	-	-	-	4.5	-	-	-
12	(*Z*)-6,7-Dihydrofarnesol	-	-	-	-	-	-	-	-	-	-	-	3.2	-
13	(–)-Isolongifolol, methyl ether	1.3	0.2	1.2	1.4	-	-	-	1.4	1.7	-	2.2	-	4.5
14	Aromadendrene oxide-(2)	-	-	-	-	1.8	-	-	-	-	-	-	-	-
15	Isolongifolol	0.8	0.1	0.8	0.2	0.8	-	0.1	-	1.2	1.7	-	-	-
16	8α,11-Elemadiol	-	-	-	0.8	-	-	-	4.7	-	-	2.6	-	-
17	β-Costol	-	-	-	-	1.7	-	0.8	-	-	-	-	-	-
18	Dehydrovomifoliol	-	-	-	-	-	-	1.4	-	-	-	9.2	-	-
19	Saussurea lactone	-	-	-	1.3	-	-	0.9	-	-	-	-	-	-
20	α-Kessyl acetate	-	-	-	-	-	-	-	24.9	-	-	-	-	-
21	Cryptomeridiol	-	-	-	-	-	-	-	-	15.1	3.3	4.8	0.5	-
22	Cyperadione	-	-	-	-	0.4	6.0	0.2	0.6	-	-	-	-	-
23	*iso*-Longifolol acetate	-	-	-	1.7	-	-	-	-	-	-	-	-	-
24	8,14-Cedrandiol	-	-	-	-	-	-	-	-	-	2.9	-	-	-
25	Clovanediol	-	-	-	1.9	-	-	-	6.3	4.9	-	-	-	-
26	Ledene oxide-(I)	-	-	-	4.5	-	-	-	8.5	1.5	-	-	-	-
27	Corymbolone	-	-	-	0.5	-	-	-	0.7	-	-	-	-	-
28	Alantolactone	1.8	0.1	6.9	1.9	0.6	1.5	-	1.0	0.3	-	-	-	-
29	Clovanediol, monoacetate	-	-	-	-	1.5	-	-	-	-	-	-	-	-
30	β-Cyclocostunolide	1.5	-	-	2.4	-	-	-	-	-	-	-	-	-
31	Zederone	-	-	-	-	-	-	-	9.1	-	-	-	-	-
Diterpene														
32	Cembrene	-	-	-	-	-	-	-	-	-	-	3.2	-	-
33	Sandaracopimaradiene	-	1.9	-	-	-	-	-	-	-	-	-	2.2	-
34	γ-Bicyclohomofarnesal	20.9	4.6	20.2	12.6	29.4	46.6	13.0	4.1	9.1	33.6	23.5	30.4	20.8

35	(*E*)-15,16-Dinorlabda-8(17),12-dien-14-al	-	-	-	-	-	-	-	-	-	-	-	-	1.4
36	(*E*)-15,16-Dinorlabda-8(17),11-dien-13-one	28.2	-	28.5	29.6	9.4	14.8	11.2	11.6	14.3	12.8	12.1	39.6	58.2
37	Epimanool	2.6	14.0	2.8	-	-	-	-	0.2	0.2	-	-	0.7	1.4
38	Sclareolide	-	-	2.4		-	-	-	-	-	-	-	-	-
39	Larixol	0.6	0.1	0.7	0.5	0.4	1.2	-	-	0.9	2.0	0.5	0.7	0.9
40	(*E*)-Labda-8(20),13-diene-15,19-diol	-	0.8	2.9	-	-	-	-	-	-	-	-	-	-
Monoterpene [%]		0.6	-	-	-	31.1	-	1.6	3.6	-	-	14.2	-	-
Sesquiterpene [%]		6.6	0.4	16.0	19.6	6.8	7.5	3.4	57.5	29.4	15.1	18.8	3.7	5.4
Diterpene [%]		52.3	21.4	57.5	42.7	39.2	62.6	24.2	15.9	24.5	48.4	39.3	73.6	82.7
Percentage of constituents identified		59.5	21.8	73.5	62.3	77.1	70.1	29.2	77	53.9	63.5	72.3	77.3	88.1
Number of identified constituents		19	12	19	21	17	11	19	23	14	14	18	13	13
Number of unidentified constituents		45	18	42	41	27	32	41	43	37	31	22	17	18

[a] Tentative identification with match score above 600. [b] Linear straight-chain hydrocarbons were excluded from the analysis.
[c] -, Not identified.

The fractions were tested for antibacterial activity with four bacteria stains to compare with the three commercial drugs as shown in **Table 3.7**. **FG.D** and **FE.D** were the most active against *S. aureus* (MIC = 100 μg mL^{-1}) and *M. luteus* (MIC = 50 μg mL^{-1}), respectively. All of these fractions were not active to *E. coli* and *P. aeruginosa* which were the gram-negative bacteria. This may be due to their outer membranes differing from the gram-positive bacteria (*S. aureus* and *M.luteus*). **FE.D** and **FG.H** were the most active against *S. aureus* (MBC = 200 μg mL^{-1}). **FE.D** was also the most active against *M. luteus* (MBC = 200 μg mL^{-1}). For pure compounds, **P1** and **P2** were not active to all four bacteria strains.

Table 3.7: Antibacterial activity of sub-fractions of *G. schomburgkii* Hook.f. rhizomes

Sample	MIC[a] [μg mL^{-1}]				MBC[b] [μg mL^{-1}]	
	S. aureus	*M. luteus*	*E. coli*	*P. aeruginosa*	*S. aureus*	*M. luteus*
Crude DCM	800	400	>800	>800	>800	>800
FE	400	200	>800	>800	800	800
FE.A	800	800	>800	>800	800	>800
FE.C	400	100	>800	>800	400	800
FE.D	200	50	>800	>800	200	200
FE.E	400	200	>800	>800	>800	800
FG	400	400	>800	>800	800	>800
FG.D	100	400	>800	>800	400	>800
FG.G	400	800	>800	>800	400	>800
FG.H	200	400	>800	>800	200	>800
FG.M	200	400	>800	>800	400	>800
P1	>800	>800	>800	>800	-	-
P2	>800	>800	>800	>800	-	-
Streptomycin	12.5	1.5625	12.5	50	-	-
Amoxicillin	<0.195	0.2	25	200	-	-
Ciprofloxacin	0.625	2.5	<0.04	2.5	-	-

[a] Minimum inhibitory concentration (MIC) against four kinds of bacteria including *Staphylococcus aureus* ATCC 6538P, *Micrococcus luteus* ATCC 4698, *Escherichia coli* ATCC 25922, *Pseudomonas aeruginosa* Strain Boston 41501 ATCC 27853 [b] Minimum bactericidal concentration (MBC) against four kinds of bacteria including *Staphylococcus aureus* ATCC 6538P, *Micrococcus luteus* ATCC 4698, *Escherichia coli* ATCC 25922, *Pseudomonas aeruginosa* Strain Boston 41501 ATCC 27853 (All the experiments were performed in triplicate.)

CHAPTER IV

CONCLUSION

The rhizomes of *Globba schomburgkii* Hook.f. was extracted with dichloromethane to provide crude extract, which was isolated by silica gel column chromatography to provide ten fractions (**FA - FJ**). These fractions were evaluated for antibacterial activity to provide MIC and MBC of each fraction against various strains of bacteria. The chemical composition of each fraction was examined by GC - MS. The major volatile compositions were identified as γ-bicyclohomofarnesal and (*E*)-15,16-dinorlabda-8(17),11-dien-13-one (**P2**). The fractions with highest antibacterial activity, **FE** and **FG**, were further separated to give sub-fractions of **FE.A - FE.G** and **FG.A - FG.M**, respectively. The antibacterial activity of these sub-fractions were measured, and the sub-fractions with the highest antibacterial activity were **FE.D** (MIC and MBC = 200 μgmL^{-1}), **FG.D** (MIC = 100 μgmL^{-1}, MBC = 400 μgmL^{-1}), and **FG.H** (MIC and MBC = 200 μgmL^{-1}). The first pure compound, petasol (**P1**), was isolated using Prep-TLC technique and another pure compound, **P2**, was crystallized from **FG.B**. The chemical structures of these pure compounds were verified by NMR techniques including ^{1}H, ^{13}C, COSY, HSQC, and HMBC. However, these two pure compounds did not show any antibacterial activity. Nevertheless, GC-MS results from this study can be used as GC standard for qualitative control of medical supplies from *G. schomburgkii* Hook. f. Active sub-fraction might be applied to pharmaceutical development. For example, active sub-fraction might be used as active ingredients in soap to treat itchy skin in the future.

Appendix A

Percentage of recovery

Table A.1: Percentage recovery of FA-FJ

Number	Compound	Recovery [%]											
		NIST RI	FA	FB	FC	FD	FE	FF	FG	FH	FI	FJ	Total
Oxygenated monoterpene													
1	Linderol	1166	-	3.0	6.9	0.9	1.0	0.1	-	1.2	3.5	-	16.6
Sesquiterpene hydrocarbon													
2	Cyperene	1399	2.7	-	-	-	-	-	-	1.1	7.8	2.1	13.7
3	Guaia-6,9-diene	1443	7.4	-	-	-	-	-	-	-	4.1	-	11.5
4	Selina-5,11-diene	1447	-	-	-	-	-	-	-	-	65.9		65.9
5	Himachala-2,4-diene	1462	10.2	-	-	-	-	-	-	-	-	68.8	79.0
Oxygenated sesquiterpene													
6	Caryophyllene oxide	1581	23.8	44.5	-	-	-	-	-	-	-	-	68.3
7	Pogostole	1655	15.9	63.6	57.0	-	-	-	-	-	-	-	136.5
8	Neointermedeol	1660	-	65.0	-	-	-	-	-	-	-	-	65.0
9	(-)-Isolongifolol, methyl ether	1672	3.8	7.1	4.7	6.8	12.3	1.2	3.5	3.6	-	-	43.1
10	Cyperenone	1687	-	25.4	-	-	-	-	-	-	-	-	25.4
11	7-Isopropenyl-1,4α-dimethyl-4,4a,5,6,7,8-hexahydro-3H-naphthalen-2-one	1755	17.0	-	14.3	-	-	-	-	-	-	-	31.3
Diterpene													
12	γ-Bicyclohomofarnesal	1809	2.1	14.9	18.4	14.6	30.0	3.8	17.0	10.3	13.0	11.0	135.1
13	(*E*)-15,16-Dinorlabda-8(17),11-dien-13-one	1994	1.3	6.8	3.1	3.3	3.1	5.0	7.5	11.6	2.2	1.3	45.2
14	Sclareol	2227	-	-	-	-	20.3	23.7	1.0	-	-	-	45.0
Oxygenated monoterpene [%]			0	3.0	6.9	0.9	1.0	0.1	0	1.2	3.5	0.0	
Sesquiterpene hydrocarbon [%]			20.3	0	0	0	0	0	0	1.1	77.8	70.9	
Oxygenated sesquiterpene [%]			60.6	205.6	76.0	6.8	12.3	1.2	3.5	3.6	0	0	
Diterpene [%]			3.4	21.7	21.4	18.0	53.3	32.5	25.6	21.9	15.2	12.3	

Table A.2: Percentage recovery of FE.A-FE.G

Number	Compound	Recovery [%]								
		NIST RI	FE.A	FE.B	FE.C	FE.D	FE.E	FE.F	FE.G	Total
Oxygenated sesquiterpene										
1	Eremophila-1(10),11-dien-9-one, 8α-hydroxy-	1864	0.1	-	-	-	-	-	-	0.1
Oxygenated diterpene										
2	γ-Bicyclohomofarnesal	1809	0.9	-	-	-	-	11.9	-	12.7
3	(*E*)-15,16-Dinorlabda-8(17),11-dien-13-one	1994	1.2	0.1	21.5	-	2.7	-	4.8	30.3
4	Isoretinene a	2198	-	4.8	-	11.1	-	-	-	15.9
5	Sclareol	2227	-	-	-	-	26.7	79.5	-	106.2
Oxygenated sesquiterpene [%]			0.1	0	0	0	0	0	0	
Oxygenated diterpene [%]			2.1	4.9	21.5	11.1	29.4	91.4	4.8	

Table A.3: Percentage recovery of FG.A-FG.M

No.	Compound[a]	Recovery [%]													
		NIST RI	FG.B	FG.C	FG.D	FG.E	FG.F	FG.G	FG.H	FG.I	FG.J	FG.K	FG.L	FG.M	Total
Monoterpene															
1	Sobrerol 8-acetate	1529	-	-	-	160.6	-	-	-	-	-	-	-	-	160.6
Sesquiterpene															
2	Longifolenaldehyde	1631	-	7.7	-	-	-	-	1.6	13.8	8.4	-	-	2.5	34.0
3	(-)-Isolongifolol, methyl ether	1672	0.1	1.4	1.7	-	-	-	6.9	7.0	-	11.3	-	11.4	73.8
4	Isolongifolol	1738	0.1	1.6	0.4	4.1	-	0.7	-	8.0	13.4	-	-	-	68.1
5	Bohlmann k2631	1916	0.0	6.0	1.7	1.4	1.7	-	3.5	0.9	-	-	-	-	43.5
6	β-Cyclocostunolide	1983	-	-	2.6	-	-	-	-	-	-	-	-	-	17.9
Diterpene															
7	γ-Bicyclohomofarnesal	1809	0.2	1.5	1.0	5.7	4.6	3.6	1.3	2.3	10.1	7.5	9.8	3.3	50.9
8	(*E*)-15,16-Dinorlabda-8(17),11-dien-13-one	1994	-	1.6	1.7	1.4	1.1	2.3	2.6	2.7	2.9	2.9	9.5	6.8	35.4
9	Epimanool	2056	3.9	1.7	-	-	-	-	0.5	0.4	-	-	1.8	1.8	10.1
10	Larixol	2264	0.1	1.8	1.4	2.7	4.1	-	-	8.0	20.9	5.5	7.9	4.9	57.4
Monoterpene [%]			0	0	0	160.6	0	0	0	0	0	0	0	0	
Sesquiterpene [%]			0.2	16.8	6.5	5.4	1.7	0.7	12.0	29.7	21.7	11.3	0.0	13.9	
Diterpene [%]			4.2	6.6	4.0	9.8	9.7	6.0	4.4	13.5	33.9	15.9	29.0	16.8	

Appendix B

Volatile chemical composition from GC-MS analysis

Table B.1: Volatile chemical composition in dichloromethane extract of rhizomes (crude DCM)

Peak	Name	Class[a]	Match	NIST RI	Exp. RI	RT	%Area	Area (count·s)
1	(+)-2-Bornanone	OM	836	1144	1144	9.953	0.5	12377631
2	Linderol	OM	887	1166	1166	10.625	3.9	92621141
3	Bornyl acetate	OM	810	1285	1285	14.403	0.5	12629410
4	Unknown	Unknown			1333	15.907	0.3	7091549
5	Cyprotene	SH	666	1345	1353	16.542	0.4	9173888
6	2,4-Patchouladiene	SH	623	1351	1362	16.807	0.2	4526314
7	Unknown	Unknown			1389	17.658	0.2	5489821
8	Cyperene	SH	813	1399	1399	17.981	7.2	170507495
9	Selina-5,11-diene	SH	655	1447	1443	19.319	0.2	4819708
10	Guaia-6,9-diene	SH	724	1443	1450	19.525	19.1	450962025
11	Himachala-2,4-diene	SH	675	1462	1462	19.886	0.4	8651111
12	Aristolochene	SH	820	1487	1484	20.564	1.2	27276812
13	Unknown	Unknown			1488	20.690	0.3	7084764
14	Valencene	SH	797	1492	1492	20.820	1.1	25957858
15	Unknown	Unknown			1506	21.214	0.3	6339253
16	Unknown	Unknown			1523	21.704	0.9	20733859
17	Epoxycyperene	OS	654	1542	1527	21.834	0.2	3884454
18	Caryophyllene oxide	OS	723	1581	1581	23.408	0.4	8405218
19	Himbaccol	OS	650	1591	1591	23.670	0.2	5319770
20	13-nor-Eremophil-1(10)-en-11-one	OS	650	1629	1627	24.678	1.9	45578830
21	Unknown	Unknown			1635	24.900	1.5	34448652
22	Neointermedeol	OS	699	1660	1653	25.421	0.3	6398306
23	Pogostole	OS	660	1655	1657	25.520	0.2	4160755
24	(−)-Isolongifolol, methyl ether	OS	680	1672	1669	25.852	1.9	44323180
25	Unknown	Unknown			1681	26.170	0.8	18610448
26	Acorenone B	OS	602	1701	1690	26.426	0.3	6878323
27	Cyperenone	OS	604	1687	1693	26.500	0.6	13546105
28	Unknown	Unknown			1740	27.763	0.3	6760686
29	α-Cyperone	OS	673	1755	1744	27.868	0.4	10111485
30	Eremophilone	OS	659	1756	1747	27.948	0.5	12920046
31	γ-Bicyclohomofarnesal	OS	846	1809	1796	29.239	6.3	149405105
32	Unknown	Unknown			1857	30.783	0.4	8476629
33	Unknown	Unknown			1870	31.091	0.4	10526115
34	Unknown	Unknown			1907	32.010	0.6	13268421

35	Unknown	Unknown			1925	32.441	0.5	12972160
36	Unknown	Unknown			1943	32.891	0.5	12122312
37	Unknown	Unknown			1950	33.045	0.4	9104526
38	Unknown	Unknown			1966	33.431	0.6	15023680
39	(*E*)-15,16-Dinorlabda-8(17),11-dien-13-one	OD	712	1994	1983	33.837	19.2	453673507
40	Ethyl palmitate	Misc.	667	1993	1994	34.109	1.8	42388760
41	Unknown	Unknown			2015	34.595	2.0	47331005
42	Unknown	Unknown			2026	34.857	3.1	74097195
43	Unknown	Unknown			2034	35.030	0.3	5959953
44	Unknown	Unknown			2062	35.683	0.4	9036048
45	Unknown	Unknown			2091	36.334	0.4	8856264
46	Coronarin E	OD	621	2136	2121	37.005	0.4	9249777
47	Unknown	Unknown			2140	37.434	0.3	7943404
48	Ethyl linoleate	Misc.	667	2162	2166	38.016	0.6	13855353
49	Unknown	Unknown			2217	39.126	0.8	20087782
50	Sclareol	OD	611	2227	2243	39.662	0.5	12340724
51	Unknown	Unknown			2279	40.414	1.2	27544576
52	Unknown	Unknown			2354	41.967	4.3	102807923
53	Unknown	Unknown			2403	42.956	1.2	28556996
54	Unknown	Unknown			2439	43.662	1.1	25635037
55	Unknown	Unknown			2457	44.020	0.7	16255089
56	Unknown	Unknown			2560	45.983	1.7	41240054
57	Unknown	Unknown			2610	46.910	2.1	50659877
58	Unknown	Unknown			2650	47.638	1.8	42932534
59	Unknown	Unknown			2806	50.414	0.3	7900323

[a]MH = monoterpene hydrocarbon, OM = oxygenated monoterpene, SH = sesquiterpene hydrocarbon, OS = oxygenated sesquiterpene, DH = diterpene hydrocarbon, OD = oxygenated diterpene, TH = triterpene hydrocarbon, Misc = Miscellaneous, Unknown = match score < 600 or DRI > 20.

Table B.2: Volatile chemical composition in fraction **FA**

Peak	Name	Class[a]	Match	NIST RI	Exp. RI	RT	%Area	Area (count·s)
1	Unknown	Unknown			1279	14.255	0.3	6127154
2	Cyprotene	SH	649	1345	1354	16.610	0.6	10255072
3	Cyperene	SH	814	1399	1400	18.071	4.3	75577609
4	Unknown	Unknown			1413	18.456	0.3	4767410
5	Unknown	Unknown			1444	19.396	0.1	2313144
6	Guaia-6,9-diene	SH	739	1443	1451	19.621	31.1	551110650
7	Himachala-2,4-diene	SH	636	1462	1463	19.972	0.9	16200064
8	Aristolochene	SH	799	1487	1485	20.647	2.1	36756104
9	Unknown	Unknown			1490	20.786	0.3	4855755
10	Valencene	SH	805	1492	1494	20.903	1.5	26006896
11	Unknown	Unknown			1519	21.655	0.6	11409375
12	Unknown	Unknown			1524	21.800	1.0	17428323
13	Unknown	Unknown			1529	21.923	0.5	7991944
14	Unknown	Unknown			1549	22.502	0.4	6811715
15	Unknown	Unknown			1553	22.644	0.5	8357858
16	Caryophyllene oxide	OS	827	1581	1583	23.501	2.1	37582547
17	Boronia butenal	OS	651	1584	1598	23.935	0.9	15930160
18	Humulene epoxide II	OS	793	1606	1609	24.253	1.9	33449543
19	Longifolenaldehyde	OS	658	1631	1628	24.777	2.4	42674234
20	Unknown	Unknown			1637	25.005	1.6	28462270
21	Pogostole	OS	624	1655	1654	25.492	0.7	11730264
22	Unknown	Unknown			1671	25.960	0.4	7012305
23	Unknown	Unknown			1676	26.096	0.6	11176514
24	(−)-Isolongifolol, methyl ether	OS	664	1672	1682	26.250	1.6	28360154
25	Unknown	Unknown			1692	26.527	1.5	26085876
26	Unknown	Unknown			1694	26.595	0.6	9902013
27	Unknown	Unknown			1701	26.774	1.0	17110922
28	Isolongifolol	OS	617	1738	1725	27.421	1.1	19463355
29	Unknown	Unknown			1746	27.976	1.2	21944465
30	α-Cyperone	OS	700	1755	1749	28.059	1.5	26583688
31	Unknown	Unknown			1753	28.148	1.3	22518427
32	Unknown	Unknown			1764	28.453	1.3	23122388
33	Unknown	Unknown			1779	28.829	0.3	5613244
34	γ-Bicyclohomofarnesal	OS	766	1809	1798	29.341	2.9	50775905
35	Unknown	Unknown			1860	30.897	1.3	23562232

36	Unknown	Unknown			1902	31.936	0.7	12642593
37	Unknown	Unknown			1909	32.111	0.5	8266950
38	Unknown	Unknown			1927	32.543	0.7	12545060
39	Unknown	Unknown			1946	33.005	1.4	24580282
40	(*E*)-15,16-Dinorlabda-8(17),12-dien-14-al	OD	629	1958	1967	33.498	1.5	27302862
41	(*E*)-15,16-Dinorlabda-8(17),11-dien-13-one	OD	699	1994	1985	33.939	5.4	96454870
42	Hexadecanyl acetate	Misc.	677	2009	2013	34.598	1.9	33139697
43	Unknown	Unknown			2018	34.709	3.7	64805299
44	Epimanool	OD	603	2056	2049	35.424	0.3	5472951
45	*cis*-10-Heptadecenoic acid	Misc.	620	2073	2087	36.303	0.8	13449468
46	Unknown	Unknown			2093	36.444	0.8	14392385
47	2-*cis*,*cis*-9,12-Octadecadienyloxyethanol	Misc.	637	2120	2130	37.249	0.7	13230901
48	Unknown	Unknown			2143	37.542	1.9	34174883
49	Unknown	Unknown			2212	39.042	0.4	7805965
50	Octadecanol acetate	Misc.	667	2208	2218	39.178	1.6	28399366
51	Unknown	Unknown			2282	40.528	0.4	6385076
52	Unknown	Unknown			2289	40.676	0.6	11365427
53	Unknown	Unknown			2358	42.075	0.5	8275968
54	Unknown	Unknown			2374	42.411	0.4	7675427
55	Eicosyl acetate	Misc.	601	2409	2412	43.178	0.4	7385799
56	Docosyl methyl ether	Misc.	625	2428	2423	43.379	0.9	15707727
57	Unknown	Unknown			2501	44.910	0.3	5936837
58	Ethylhexyl phthalate	Misc.	600	2529	2548	45.801	0.4	7816823
59	Unknown	Unknown			2552	45.875	0.4	6746435
60	Unknown	Unknown			2575	46.297	0.3	4430630
61	Unknown	Unknown			2627	47.268	0.4	6683802
62	Unknown	Unknown			2701	48.615	0.3	5258145
63	Unknown	Unknown			2746	49.394	0.4	6440852
64	Unknown	Unknown			2801	50.368	0.9	15069547
65	Unknown	Unknown			2810	50.513	0.6	11028709

[a]MH = monoterpene hydrocarbon, OM = oxygenated monoterpene, SH = sesquiterpene hydrocarbon, OS = oxygenated sesquiterpene, DH = diterpene hydrocarbon, OD = oxygenated diterpene, TH = triterpene hydrocarbon, Misc = Miscellaneous, Unknown = match score < 600 or ΔRI > 20.

Table B.3: Volatile chemical composition in fraction **FB**

Peak	Name	Class[a]	Match	NIST RI	Exp. RI	RT	%Area	Area (count·s)
1	Linderol	OM	887	1166	1166	10.696	1.4	42514524
2	Cyprotene	SH	663	1345	1354	16.612	0.2	4648350
3	2,4-Patchouladiene	SH	668	1351	1362	16.880	0.2	4786511
4	Unknown	Unknown			1374	17.253	0.1	2667099
5	*cis*-β-Copaene	SH	668	1432	1450	19.589	0.1	3611887
6	Unknown	Unknown			1490	20.779	0.1	4050854
7	Unknown	Unknown			1513	21.478	0.2	6896829
8	Unknown	Unknown			1524	21.789	0.6	19704937
9	Diepicedrene-1-oxide	OS	631	1551	1553	22.643	0.2	6673235
10	*trans*- Nerolidol	OS	706	1564	1565	22.969	0.1	3481096
11	Espatulenol	OS	653	1576	1578	23.352	0.1	3257530
12	Caryophyllene oxide	OS	852	1581	1583	23.500	2.1	63758957
13	Himbaccol	OS	830	1591	1592	23.758	1.3	38716986
14	Boronia butenal	OS	714	1584	1598	23.934	0.2	7356776
15	Humulene epoxide II	OS	663	1606	1609	24.245	0.8	23681644
16	Unknown	Unknown			1614	24.369	0.1	4258858
17	Unknown	Unknown			1620	24.544	0.4	11681285
18	Longifolenaldehyde	OS	654	1631	1628	24.772	2.1	63505605
19	β-Acorenol	OS	622	1649	1633	24.902	0.1	2465678
20	Caryophylladienol II	OS	667	1637	1636	24.991	2.2	66293307
21	Muurolol T	OS	697	1642	1643	25.182	0.3	9773644
22	Pogostole	OS	717	1655	1655	25.518	1.5	46184651
23	Neointermedeol	OS	793	1660	1659	25.620	2.3	71323550
24	epi-γ-Eudesmol	OS	633	1662	1667	25.845	0.5	15745109
25	(−)-Isolongifolol, methyl ether	OS	684	1672	1671	25.949	1.6	49097982
26	Unknown	Unknown			1682	26.248	0.5	15714452
27	Cyperenone	OS	821	1687	1694	26.593	1.5	47543452
28	Unknown	Unknown			1714	27.126	0.1	3970088
29	Unknown	Unknown			1718	27.231	0.1	4448557
30	Unknown	Unknown			1742	27.863	1.7	51636612
31	Isolongifolol	OS	629	1738	1746	27.968	2.1	63360893
32	Unknown	Unknown			1754	28.187	0.3	8179849
33	Squamulosone	OS	620	1771	1771	28.627	0.2	4802397
34	γ-Bicyclohomofarnesal	OS	857	1809	1798	29.348	11.1	342665461
35	Dehydrofukinone	OS	867	1817	1804	29.484	2.1	64895456

36	Unknown	Unknown			1815	29.755	0.5	16418215
37	Unknown	Unknown			1845	30.513	1.8	56441382
38	8,14-Cedrandiol	OS	632	1876	1859	30.877	0.4	11982964
39	Di-*sec*-butyl phthalate	Misc.	688	1855	1868	31.089	0.1	3643168
40	Valerenic acid	OS	615	1871	1872	31.197	0.2	6688397
41	Unknown	Unknown			1879	31.367	0.2	6487219
42	Unknown	Unknown			1902	31.943	0.5	16145570
43	Unknown	Unknown			1908	32.103	0.5	15762244
44	Unknown	Unknown			1927	32.541	2.3	69601809
45	Unknown	Unknown			1933	32.692	0.1	4168568
46	Unknown	Unknown			1946	33.000	0.2	5421765
47	Sandaracopimaradiene	DH	620	1960	1952	33.136	0.4	13554537
48	Dibutyl phthalate	Misc.	798	1965	1962	33.385	0.7	21135444
49	(*E*)-15,16-Dinorlabda-8(17),12-dien-14-al	OD	646	1958	1966	33.493	0.4	11071098
50	(*E*)-15,16-Dinorlabda-8(17),11-dien-13-one	OD	769	1994	1986	33.952	15.3	469264572
51	Ethyl palmitate	Misc.	726	1993	1996	34.202	4.6	140017326
52	Unknown	Unknown			2001	34.319	0.2	6757544
53	Unknown	Unknown			2018	34.704	1.2	38127370
54	Unknown	Unknown			2036	35.129	0.3	10692547
55	Epimanool	OD	655	2056	2049	35.422	0.2	6743801
56	Unknown	Unknown			2065	35.798	0.5	16692333
57	Unknown	Unknown			2071	35.921	0.4	12928927
58	Thunbergol	OD	612	2073	2076	36.041	0.3	10092236
59	*cis*-10-Heptadecenoic acid	Misc.	671	2073	2087	36.291	0.7	20753405
60	Unknown	Unknown			2094	36.464	0.5	14588832
61	Oleic Acid	Misc.	601	2141	2129	37.240	0.4	12065531
62	Unknown	Unknown			2143	37.539	1.3	39635451
63	Linoleic acid ethyl ester	Misc.	809	2162	2163	37.977	3.3	100201750
64	Ethyl Oleate	Misc.	785	2173	2169	38.118	2.4	74591863
65	Unknown	Unknown			2175	38.245	0.3	8494217
66	Ethyl octadecanoate	Misc.	658	2195	2196	38.716	0.7	20963576
67	Unknown	Unknown			2211	39.037	0.3	10190612
68	Unknown	Unknown			2225	39.317	0.3	8997266
69	Unknown	Unknown			2247	39.782	0.7	21252443
70	Unknown	Unknown			2282	40.534	1.3	39744566
71	Eicosanol	OD	622	2281	2289	40.685	1.3	40485907
72	Unknown	Unknown			2302	40.947	0.3	9844096
73	Unknown	Unknown			2358	42.081	3.5	108298989

74	Eicosyl acetate	Misc.	642	2409	2412	43.181	0.5	14232770
75	Unknown	Unknown			2447	43.853	0.6	17045388
76	Unknown	Unknown			2461	44.134	0.3	10533040
77	Unknown	Unknown			2508	45.052	0.4	10958428
78	Unknown	Unknown			2543	45.705	0.8	23137674
79	Phthalic acid, hept-3-yl octyl ester	Misc.	641	2537	2548	45.795	0.4	11737426
80	Unknown	Unknown			2614	47.034	7.3	224322699
81	1-Methylene-5α-androstan-3α-ol-17-one	OD	605	2649	2653	47.742	0.6	18695532
82	Borneol palmitate	Misc.	620	2720	2709	48.744	0.3	10358797
83	Sebacic acid, di(2-octyl) ester	Misc.	737	2814	2809	50.510	1.7	52199196

[a]MH = monoterpene hydrocarbon, OM = oxygenated monoterpene, SH = sesquiterpene hydrocarbon, OS = oxygenated sesquiterpene, DH = diterpene hydrocarbon, OD = oxygenated diterpene, TH = triterpene hydrocarbon, Misc = Miscellaneous, Unknown = match score < 600 or ΔRI > 20.

Table B.4: Volatile chemical composition in fraction **FC**

Peak	Name	Class[a]	Match	NIST RI	Exp. RI	RT	%Area	Area (count·s)
1	Linderol	OM	878	1166	1166	10.699	3.3	59102050
2	*p*-Cymenol	OM	601	1183	1187	11.319	0.1	2398178
3	Berbenone	OM	607	1205	1209	12.033	0.1	2080940
4	Bornyl formate	OM	622	1226	1228	12.631	0.2	3220578
5	Unknown	Unknown			1274	14.098	0.3	5903676
6	Bornyl acetate	OM	730	1285	1286	14.471	0.4	7554524
7	Unknown	Unknown			1354	16.616	0.2	3918181
8	Unknown	Unknown			1451	19.602	0.3	4988635
9	Unknown	Unknown			1490	20.780	0.2	3285121
10	Unknown	Unknown			1514	21.488	0.2	2841001
11	Unknown	Unknown			1524	21.797	0.5	9373770
12	Longifolenaldehyde	OS	681	1631	1628	24.774	1.4	25759912
13	Unknown	Unknown			1636	24.986	2.3	40453331
14	Pogostole	OS	694	1655	1655	25.510	1.4	25397061
15	Unknown	Unknown			1667	25.843	0.2	3159048
16	Unknown	Unknown			1671	25.942	1.6	28770946
17	(−)-Isolongifolol, methyl ether	OS	637	1672	1681	26.244	1.1	19967294
18	β-Nootkatol	OS	642	1712	1709	26.983	0.4	6834618
19	Unknown	Unknown			1718	27.233	0.6	10766853
20	(*Z*)-α-Atlantone	OS	602	1717	1728	27.489	0.7	12573853
21	Unknown	Unknown			1742	27.859	1.4	24437028
22	Isolongifolol	OS	650	1738	1746	27.973	0.3	5030882
23	α-Cyperone	OS	601	1755	1759	28.315	0.7	12626046
24	Santalcamphor	OS	603	1774	1765	28.460	0.6	10140313
25	Unknown	Unknown			1794	29.221	0.7	12420632
26	γ-Bicyclohomofarnesal	OS	851	1809	1798	29.338	14.2	254126194
27	Nootkatone	OS	672	1808	1804	29.489	0.3	5507534
28	Cyperadione	OS	717	1845	1831	30.164	0.9	15862914
29	Unknown	Unknown			1844	30.503	0.5	8616325
30	Unknown	Unknown			1859	30.873	0.2	3454510
31	Unknown	Unknown			1878	31.350	0.3	5118008
32	Unknown	Unknown			1900	31.911	0.4	7271124
33	Alantolactone	OS	662	1916	1908	32.102	2.8	50079639
34	Unknown	Unknown			1927	32.543	1.3	23095228
35	Unknown	Unknown			1951	33.135	0.3	5027977

36	Unknown	Unknown			1968	33.523	3.9	69364167
37	Unknown	Unknown			1977	33.745	0.8	14165641
38	(*E*)-15,16-Dinorlabda-8(17),11-dien-13-one	OD	707	1994	1984	33.927	7.2	129155550
39	Unknown	Unknown			1992	34.105	0.5	9450716
40	Ethyl palmitate	Misc.	604	1993	1996	34.201	0.8	14575770
41	Unknown	Unknown			2001	34.321	0.7	13070761
42	Unknown	Unknown			2017	34.700	2.2	39908496
43	Androst-5-en-4-one	OD	629	2030	2048	35.415	0.9	16262557
44	Unknown	Unknown			2065	35.785	1.2	21454265
45	Unknown	Unknown			2087	36.297	1.1	19580218
46	Unknown	Unknown			2093	36.441	2.2	39098087
47	Unknown	Unknown			2128	37.212	0.5	9746613
48	Unknown	Unknown			2169	38.112	0.6	11387561
49	Unknown	Unknown			2174	38.232	0.4	7454622
50	Unknown	Unknown			2192	38.626	3.0	54074789
51	Unknown	Unknown			2201	38.814	0.4	7107341
52	Unknown	Unknown			2220	39.224	0.7	12822506
53	Unknown	Unknown			2236	39.554	1.4	24651316
54	Unknown	Unknown			2260	40.056	1.8	31927765
55	Unknown	Unknown			2282	40.531	1.0	18350015
56	Unknown	Unknown			2291	40.707	0.5	9380289
57	Unknown	Unknown			2303	40.962	0.6	10893765
58	Unknown	Unknown			2358	42.075	7.4	132095803
59	Unknown	Unknown			2462	44.140	0.2	4350207
60	Unknown	Unknown			2522	45.302	0.8	14983797
61	Unknown	Unknown			2543	45.705	3.1	54804949
62	Unknown	Unknown			2563	46.078	0.5	8242223
63	Unknown	Unknown			2589	46.562	0.4	7533327
64	Unknown	Unknown			2613	47.015	6.7	120210572
65	Unknown	Unknown			2653	47.745	6.4	114100591
66	Borneol palmitate	Misc.	637	2720	2709	48.750	1.4	24727517

[a]MH = monoterpene hydrocarbon, OM = oxygenated monoterpene, SH = sesquiterpene hydrocarbon, OS = oxygenated sesquiterpene, DH = diterpene hydrocarbon, OD = oxygenated diterpene, TH = triterpene hydrocarbon, Misc = Miscellaneous, Unknown = match score < 600 or ΔRI > 20.

Table B.5: Volatile chemical composition in fraction **FD**

Peak	Name	Class[a]	Match	NIST RI	Exp. RI	RT	%Area	Area (count·s)
1	Linderol	OM	775	1166	1166	10.693	0.7	10450717
2	Unknown	Unknown			1354	16.613	0.4	6202478
3	Unknown	Unknown			1490	20.783	0.6	8963307
4	Unknown	Unknown			1524	21.797	0.7	10498570
5	Longifolenaldehyde	OS	650	1631	1628	24.761	1.3	18064622
6	Unknown	Unknown			1636	24.992	1.7	24505849
7	(−)-Isolongifolol, methyl ether	OS	670	1672	1670	25.939	2.6	37363137
8	Unknown	Unknown			1676	26.102	0.5	7025237
9	Unknown	Unknown			1682	26.262	0.5	7362644
10	Unknown	Unknown			1742	27.852	0.8	10872899
11	Isolongifolol	OS	669	1738	1746	27.970	0.6	8822219
12	Unknown	Unknown			1754	28.176	0.4	5617201
13	γ-Bicyclohomofarnesal	OS	846	1809	1798	29.335	18.5	264177521
14	Unknown	Unknown			1859	30.879	0.6	8562495
15	Unknown	Unknown			1872	31.190	0.3	4404325
16	Alantolactone	OS	668	1916	1908	32.099	3.9	56467773
17	Unknown	Unknown			1927	32.540	1.5	21525583
18	Unknown	Unknown			1946	32.999	0.4	6043330
19	Cembrene	DH	610	1939	1951	33.128	0.3	3640881
20	Unknown	Unknown			1968	33.523	5.5	79016166
21	Unknown	Unknown			1977	33.757	0.5	6482301
22	(*E*)-15,16-Dinorlabda-8(17),11-dien-13-one	OD	690	1994	1985	33.939	12.9	184859662
23	Unknown	Unknown			1996	34.213	1.0	14331586
24	Unknown	Unknown			2018	34.703	3.7	53586527
25	Epimanool	OD	628	2056	2046	35.350	0.8	11729811
26	Unknown	Unknown			2064	35.776	1.9	27709730
27	Unknown	Unknown			2087	36.293	1.7	23970061
28	Unknown	Unknown			2161	37.930	0.7	10228225
29	Unknown	Unknown			2220	39.221	6.8	98015147
30	Unknown	Unknown			2282	40.528	1.3	18442716
31	Unknown	Unknown			2357	42.072	7.9	112771046
32	Unknown	Unknown			2406	43.058	0.5	6777516
33	Unknown	Unknown			2442	43.767	15.1	215982970
34	Unknown	Unknown			2467	44.251	0.9	13298201
35	Unknown	Unknown			2653	47.733	2.3	33113957

[a]MH = monoterpene hydrocarbon, OM = oxygenated monoterpene, SH = sesquiterpene hydrocarbon, OS = oxygenated sesquiterpene, DH = diterpene hydrocarbon, OD = oxygenated diterpene, TH = triterpene hydrocarbon, Misc = Miscellaneous, Unknown = match score < 600 or ΔRI > 20.

Table B.6: Volatile chemical composition in fraction **FE**

Peak	Name	Class[a]	Match	NIST RI	Exp. RI	RT	%Area	Area (count·s)
1	Unknown	Unknown			1101	8.687	0.3	2457077
2	Unknown	Unknown			1121	9.294	0.3	2476843
3	Linderol	OM	680	1166	1166	10.684	0.5	4607226
4	2-Hydroxycineol	OM	732	1228	1223	12.453	0.9	7264443
5	Unknown	Unknown			1332	15.935	0.5	3955611
6	Unknown	Unknown			1380	17.433	1.0	8199472
7	Unknown	Unknown			1395	17.910	2.1	17459196
8	Unknown	Unknown			1400	18.062	7.5	63010845
9	2,4-Di-tert-butylphenol	OS	698	1519	1513	21.470	0.9	7238588
10	Unknown	Unknown			1524	21.787	0.8	6509005
11	Unknown	Unknown			1600	24.000	1.3	10515621
12	Longifolenaldehyde	OS	633	1631	1628	24.761	0.9	7280954
13	Unknown	Unknown			1636	24.983	1.7	14058940
14	(−)-Isolongifolol, methyl ether	OS	615	1672	1670	25.936	3.0	25344453
15	Unknown	Unknown			1741	27.846	0.9	7417090
16	γ-Bicyclohomofarnesal	OD	852	1809	1798	29.326	24.2	202978322
17	Unknown	Unknown			1824	30.004	0.6	5112505
18	Unknown	Unknown			1855	30.771	0.6	5126266
19	Unknown	Unknown			1859	30.876	0.6	5315638
20	8α-hydroxy-Eremophila-1(10),11-dien-9-one	OS	613	1864	1874	31.252	1.8	14768222
21	Unknown	Unknown			1900	31.899	0.5	4110866
22	Unknown	Unknown			1926	32.524	1.0	8420349
23	Unknown	Unknown			1945	32.981	0.3	2681107
24	(*E*)-15,16-Dinorlabda-8(17),12-dien-14-al	OD	617	1958	1958	33.286	0.3	2925360
25	(*E*)-15,16-Dinorlabda-8(17),11-dien-13-one	OD	680	1994	1984	33.924	7.6	64161928
26	Unknown	Unknown			2000	34.309	0.5	4446480
27	Unknown	Unknown			2016	34.679	1.1	8939994
28	Epimanool	OD	626	2056	2048	35.397	0.6	4931921
29	Unknown	Unknown			2064	35.776	0.4	3028403
30	Unknown	Unknown			2070	35.902	0.4	3369829
31	Unknown	Unknown			2075	36.025	0.8	6599714
32	Unknown	Unknown			2090	36.371	2.6	21880382
33	Unknown	Unknown			2106	36.722	1.8	15364890
34	Isoretinene a	OD	608	2198	2188	38.531	1.4	11389432
35	Unknown	Unknown			2223	39.292	0.9	7339998

36	Sclareol	OD	617	2227	2245	39.745	1.3	11031451
37	Unknown	Unknown			2268	40.241	1.8	15115334
38	Unknown	Unknown			2281	40.512	2.9	24138571
39	Unknown	Unknown			2357	42.056	13.0	109074189
40	Unknown	Unknown			2406	43.049	1.3	10752626
41	Unknown	Unknown			2441	43.748	2.5	21233910
42	Unknown	Unknown			2466	44.235	1.6	13790267
43	Unknown	Unknown			2543	45.693	0.9	7424826
44	Ethylhexyl phthalate	Misc.	619	2529	2547	45.770	1.0	8165859
45	Unknown	Unknown			2652	47.718	1.1	9532925
46	Unknown	Unknown			2751	49.490	1.2	10212996
47	Unknown	Unknown			2762	49.690	1.1	8909545

[a]MH = monoterpene hydrocarbon, OM = oxygenated monoterpene, SH = sesquiterpene hydrocarbon, OS = oxygenated sesquiterpene, DH = diterpene hydrocarbon, OD = oxygenated diterpene, TH = triterpene hydrocarbon, Misc = Miscellaneous, Unknown = match score < 600 or ΔRI > 20.

Table B.7: Volatile chemical composition in fraction **FF**

Peak	Name	Class[a]	Match	NIST RI	Exp. RI	RT	%Area	Area (count·s)
1	Unknown	Unknown			1158	10.437	0.3	16546202
2	Linderol	OM	753	1166	1166	10.690	0.1	5375538
3	*trans*-Ascaridol glycol	OM	816	1272	1269	13.932	0.3	15455114
4	2-Methyl-5-(propan-2-ylidene)cyclohexane-1,4-diol	OM	696	1322	1311	15.254	0.2	8912426
5	(+)-Pinanediol	OM	623	1316	1316	15.423	0.5	24639249
6	Unknown	Unknown			1320	15.534	0.1	5663515
7	Unknown	Unknown			1333	15.954	1.0	47923765
8	Limonene glycol	OM	686	1321	1339	16.142	0.3	13393478
9	Unknown	Unknown			1395	17.917	0.5	25907886
10	Unknown	Unknown			1400	18.062	0.7	35468269
11	Carvone hydrate	OM	695	1424	1423	18.776	0.2	10130305
12	Unknown	Unknown			1496	20.983	0.1	3705540
13	Unknown	Unknown			1506	21.254	0.8	37977730
14	Unknown	Unknown			1524	21.778	0.2	11744475
15	Unknown	Unknown			1542	22.299	0.2	7596535
16	Longifolenaldehyde	OS	617	1631	1628	24.758	0.3	13027330
17	Unknown	Unknown			1636	24.980	0.5	24413205
18	(−)-Isolongifolol, methyl ether	OS	644	1672	1670	25.932	0.5	24644484
19	Unknown	Unknown			1676	26.090	0.2	11669572
20	Unknown	Unknown			1741	27.840	0.3	12589176
21	Unknown	Unknown			1773	28.681	0.2	9662057
22	Dehydrovomifoliol	OS	799	1800	1789	29.101	1.7	83504706
23	γ-Bicyclohomofarnesal	OS	842	1809	1798	29.326	5.2	255923969
24	Unknown	Unknown			1801	29.421	0.2	9766950
25	Unknown	Unknown			1805	29.520	0.4	21971621
26	Unknown	Unknown			1824	30.004	0.6	31788172
27	Unknown	Unknown			1837	30.318	0.1	5652755
28	Unknown	Unknown			1853	30.715	0.3	15959736
29	Unknown	Unknown			1871	31.181	1.0	50537376
30	Unknown	Unknown			1881	31.433	0.1	6739357
31	Corymbolone	OS	629	1899	1889	31.628	0.2	9393025
32	Unknown	Unknown			1900	31.893	0.2	10100557
33	Unknown	Unknown			1908	32.081	0.2	7531308
34	Dihydrocolumellarin	OS	629	1909	1926	32.521	0.3	12342517

35	Unknown	Unknown			1945	32.981	0.1	7162665
36	Unknown	Unknown			1960	33.335	0.3	13281167
37	(*E*)-15,16-Dinorlabda-8(17),11-dien-13-one	OD	606	1994	1984	33.917	20.9	1022431970
38	Unknown	Unknown			2001	34.324	0.3	14424608
39	Unknown	Unknown			2017	34.685	0.3	14653947
40	Unknown	Unknown			2033	35.055	42.8	2094878082
41	Unknown	Unknown			2047	35.387	0.9	42345393
42	Valerenic acid, n-butyl ester	OS	609	2065	2064	35.773	0.2	11484549
43	Epimanool	OD	601	2056	2075	36.032	0.3	16881234
44	Unknown	Unknown			2127	37.200	0.6	30235540
45	Unknown	Unknown			2173	38.213	0.2	7664338
46	Unknown	Unknown			2200	38.802	0.1	6017723
47	Sclareol	OD	651	2227	2245	39.751	2.6	128179678
48	Unknown	Unknown			2259	40.035	0.8	38022617
49	Unknown	Unknown			2281	40.506	0.2	11942596
50	Unknown	Unknown			2357	42.060	3.1	151843807
51	Gibberellic acid	OD	625	2393	2405	43.036	0.5	24312799
52	Unknown	Unknown			2448	43.872	0.2	8883504
53	Unknown	Unknown			2466	44.223	0.2	8588742
54	Unknown	Unknown			2542	45.687	0.1	5248721
55	Piperanine	Misc.	706	2625	2615	47.046	0.4	21807594
56	Unknown	Unknown			2652	47.715	0.3	14126867
57	Unknown	Unknown			2732	49.151	0.4	18094311
58	Unknown	Unknown			2745	49.376	0.4	19356571
59	Unknown	Unknown			2756	49.570	0.2	7671218
60	Piperine	Misc.	638	2884	2890	51.872	5.4	264103025

[a]MH = monoterpene hydrocarbon, OM = oxygenated monoterpene, SH = sesquiterpene hydrocarbon, OS = oxygenated sesquiterpene, DH = diterpene hydrocarbon, OD = oxygenated diterpene, TH = triterpene hydrocarbon, Misc = Miscellaneous, Unknown = match score < 600 or ΔRI > 20.

Table B.8: Volatile chemical composition in fraction FG

Peak	Name	Class[a]	Match	NIST RI	Exp. RI	RT	%Area	Area (count·s)
1	Unknown	Unknown			1391	18.405	0.2	25590271
2	Unknown	Unknown			1502	21.798	0.2	22280723
3	Sobrerol 8-acetate	OM	621	1529	1526	22.498	0.6	81733624
4	Unknown	Unknown			1555	23.339	0.1	13423732
5	Unknown	Unknown			1612	24.988	0.8	111847208
6	Longifolenaldehyde	OS	664	1631	1630	25.493	1.2	170805276
7	Unknown	Unknown			1637	25.691	6.0	849918515
8	(−)-Isolongifolol, methyl ether	OS	681	1672	1673	26.674	1.3	183705691
9	3-Hydroxy-5,6-epoxy-β-ionone	OS	641	1692	1684	26.988	0.9	129279971
10	Unknown	Unknown			1689	27.124	0.1	20532973
11	Shyobunol	OS	614	1701	1702	27.484	0.4	58252654
12	Unknown	Unknown			1716	27.845	0.1	15946451
13	Unknown	Unknown			1745	28.603	0.3	37218870
14	Isolongifolol	OS	717	1738	1748	28.693	0.8	107688564
15	Unknown	Unknown			1752	28.807	0.4	50286435
16	Unknown	Unknown			1761	29.029	0.3	43762884
17	Unknown	Unknown			1775	29.408	0.2	22922908
18	Unknown	Unknown			1787	29.728	0.1	16629463
19	Unknown	Unknown			1793	29.885	0.2	21373801
20	γ-Bicyclohomofarnesal	OD	666	1809	1800	30.073	20.9	2956054320
21	Unknown	Unknown			1807	30.243	0.4	53484229
22	Isopropyl myristate	Misc.	669	1827	1827	30.755	0.2	25207079
23	Dehydrosaussurea lactone	OS	607	1838	1839	31.038	0.1	11893160
24	Unknown	Unknown			1847	31.248	0.4	50913246
25	Unknown	Unknown			1862	31.624	0.2	31011034
26	Unknown	Unknown			1881	32.108	0.7	100417612
27	Unknown	Unknown			1903	32.665	7.0	989714579
28	Alantolactone	OS	675	1916	1911	32.863	1.8	248576360
29	Unknown	Unknown			1915	32.955	0.7	98331467
30	7,9-Di-tert-butyl-1-oxaspiro(4,5)deca-6,9-diene-2,8-dione	OD	788	1923	1919	33.044	0.3	35568695
31	Methyl palmitate	Misc.	662	1926	1928	33.257	1.2	162655429
32	Unknown	Unknown			1935	33.430	0.2	34032919
33	Unknown	Unknown			1941	33.578	0.1	16876348
34	Unknown	Unknown			1945	33.667	0.2	31708508

35	Unknown	Unknown			1948	33.753	0.1	13741830
36	Unknown	Unknown			1952	33.830	0.9	131368339
37	Dibutyl phthalate	Misc.	789	1965	1963	34.102	1.2	164844651
38	β-Cyclocostunolide	OS	602	1983	1971	34.299	1.5	205356796
39	Unknown	Unknown			1981	34.530	0.2	33809141
40	(*E*)-15,16-Dinorlabda-8(17),11-dien-13-one	OD	758	1994	1987	34.690	28.2	3987309400
41	Unknown	Unknown			1993	34.838	0.1	13985638
42	Unknown	Unknown			2012	35.276	1.6	232582734
43	Unknown	Unknown			2020	35.467	3.4	480575635
44	Unknown	Unknown			2035	35.800	0.3	44481604
45	Epimanool	OD	730	2056	2038	35.877	2.6	372804728
46	Unknown	Unknown			2068	36.561	1.3	186384001
47	Unknown	Unknown			2072	36.669	0.1	19995462
48	Unknown	Unknown			2085	36.968	0.4	50207221
49	Unknown	Unknown			2091	37.088	0.3	48432991
50	Unknown	Unknown			2096	37.221	1.1	157298869
51	Unknown	Unknown			2104	37.390	2.4	335894245
52	Unknown	Unknown			2111	37.563	0.2	26231634
53	Unknown	Unknown			2127	37.908	0.2	23669968
54	Unknown	Unknown			2138	38.161	0.2	34973931
55	Unknown	Unknown			2205	39.736	0.1	18676887
56	Octadecanol acetate	Misc.	676	2208	2208	39.862	0.5	75200165
57	Sclareol	OD	628	2227	2224	40.531	0.1	14119675
58	Unknown	Unknown			2236	41.036	0.3	47567223
59	Unknown	Unknown			2242	41.314	0.2	26665103
60	Unknown	Unknown			2246	41.477	0.1	14032901
61	Unknown	Unknown			2249	41.610	2.7	377350059
62	Unknown	Unknown			2274	42.661	0.3	45729659
63	Larixol	OD	693	2264	2278	42.824	0.6	89832494
64	Unknown	Unknown			2287	43.178	0.1	19505089

[a]MH = monoterpene hydrocarbon, OM = oxygenated monoterpene, SH = sesquiterpene hydrocarbon, OS = oxygenated sesquiterpene, DH = diterpene hydrocarbon, OD = oxygenated diterpene, TH = triterpene hydrocarbon, Misc = Miscellaneous, Unknown = match score < 600 or ΔRI > 20.

Table B.9: Volatile chemical composition in fraction **FH**

Peak	Name	Class[a]	Match	NIST RI	Exp. RI	RT	%Area	Area (count·s)
1	1,2-Dimethyl-4-ethylbenzene	MH	840	1085	1077	6.656	2.1	19941493
2	Linderol	OM	765	1166	1166	10.681	0.8	7344776
3	Unknown	Unknown			1200	11.735	0.4	3848334
4	Unknown	Unknown			1285	14.456	0.3	2403027
5	Cyperene	SH	692	1399	1399	18.049	1.4	13124883
6	*cis*-β-Copaene	SH	679	1432	1450	19.584	1.8	17252555
7	Unknown	Unknown			1523	21.775	0.7	6423971
8	Longifolenaldehyde	OS	647	1631	1627	24.743	1.0	9457615
9	Unknown	Unknown			1635	24.971	1.0	9911717
10	(—)-Isolongifolol, methyl ether	OS	605	1672	1670	25.923	1.2	11032809
11	Unknown	Unknown			1682	26.247	0.3	2412681
12	Unknown	Unknown			1741	27.834	0.9	8309362
13	Unknown	Unknown			1758	28.290	0.3	2552607
14	γ-Bicyclohomofarnesal	OS	848	1809	1797	29.310	11.3	106990420
15	Dehydrosaussurea lactone	OS	614	1838	1846	30.540	0.5	4555759
16	Unknown	Unknown			1858	30.857	0.3	2771918
17	Unknown	Unknown			1899	31.877	0.3	3109971
18	Unknown	Unknown			1907	32.078	0.4	3702093
19	Unknown	Unknown			1925	32.506	0.1	1293373
20	Unknown	Unknown			1936	32.771	0.3	2712542
21	Unknown	Unknown			1950	33.107	0.4	3392951
22	(*E*)-15,16-Dinorlabda-8(17),12-dien-14-al	OD	721	1958	1957	33.270	0.8	7748305
23	(*E*)-15,16-Dinorlabda-8(17),11-dien-13-one	OD	738	1994	1984	33.914	39.0	367980239
24	Unknown	Unknown			2000	34.296	0.5	4727525
25	Unknown	Unknown			2035	35.101	0.3	3090624
26	Unknown	Unknown			2069	35.887	0.3	3108533
27	Epimanool	OD	626	2056	2075	36.013	0.8	7827307
28	Unknown	Unknown			2168	38.087	0.6	5634386
29	Unknown	Unknown			2183	38.417	0.5	4280112
30	Unknown	Unknown			2281	40.497	0.5	4480211
31	Unknown	Unknown			2342	41.751	1.3	12526851
32	Unknown	Unknown			2349	41.902	8.6	80915780
33	Unknown	Unknown			2356	42.035	2.0	18722132
34	Unknown	Unknown			2388	42.694	1.4	13388243
35	Unknown	Unknown			2459	44.100	0.3	2828081

36	Unknown	Unknown			2651	47.705	17.4	164796816

[a]MH = monoterpene hydrocarbon, OM = oxygenated monoterpene, SH = sesquiterpene hydrocarbon, OS = oxygenated sesquiterpene, DH = diterpene hydrocarbon, OD = oxygenated diterpene, TH = triterpene hydrocarbon, Misc = Miscellaneous, Unknown = match score < 600 or ΔRI > 20

Table B.10: Volatile chemical composition in fraction **FI**

Peak	Name	Class[a]	Match	NIST RI	Exp. RI	RT	%Area	Area (count·s)
1	1,3-Dimethyl-2-ethylbenzene	MH	816	1080	1077	6.656	4.7	17274283
2	Linderol	OM	793	1166	1166	10.681	3.1	11255422
3	Bornyl acetate	OM	672	1285	1285	14.453	1.1	3880496
4	Cyperene	SH	773	1399	1399	18.043	12.7	46293420
5	Selina-5,11-diene	SH	744	1447	1443	19.371	3.0	10884089
6	Guaia-6,9-diene	SH	696	1443	1450	19.578	18.0	65351604
7	Valencene	SH	684	1492	1493	20.875	1.1	3974980
8	Unknown	Unknown			1523	21.775	1.8	6457825
9	Longifolenaldehyde	OS	628	1631	1627	24.743	2.8	10205954
10	Unknown	Unknown			1635	24.962	2.3	8334368
11	Unknown	Unknown			1670	25.923	1.8	6515829
12	Unknown	Unknown			1681	26.234	0.8	2869633
13	Unknown	Unknown			1741	27.828	1.5	5569578
14	γ-Bicyclohomofarnesal	OS	838	1809	1797	29.310	18.7	68177926
15	Unknown	Unknown			1843	30.475	1.1	4129473
16	Alantolactone	OS	620	1916	1907	32.074	2.0	7149168
17	Unknown	Unknown			1936	32.774	0.9	3300102
18	Unknown	Unknown			1944	32.962	0.8	2853661
19	Unknown	Unknown			1973	33.643	1.0	3462306
20	(*E*)-15,16-Dinorlabda-8(17),11-dien-13-one	OD	733	1994	1984	33.905	9.6	34896741
21	Unknown	Unknown			2121	37.070	1.0	3574763
22	Unknown	Unknown			2173	38.192	1.3	4582995
23	Unknown	Unknown			2200	38.787	1.2	4365240
24	Unknown	Unknown			2281	40.497	1.3	4596912
25	Unknown	Unknown			2356	42.038	2.8	10098931
26	Unknown	Unknown			2405	43.027	1.7	6166133
27	Unknown	Unknown			2542	45.678	0.7	2491115
28	Unknown	Unknown			2652	47.718	1.4	5033736

[a]MH = monoterpene hydrocarbon, OM = oxygenated monoterpene, SH = sesquiterpene hydrocarbon, OS = oxygenated sesquiterpene, DH = diterpene hydrocarbon, OD = oxygenated diterpene, TH = triterpene hydrocarbon, Misc = Miscellaneous, Unknown = match score < 600 or ΔRI > 20.

Table B.11: Volatile chemical composition in fraction **FJ**

Peak	Name	Class[a]	Match	NIST RI	Exp. RI	RT	%Area	Area (count·s)
1	Unknown	Unknown			1333	15.972	5.6	6623980
2	Unknown	Unknown			1354	16.610	1.3	1549906
3	Cyperene	SH	665	1399	1400	18.052	6.3	7424792
4	Himachala-2,4-diene	SH	630	1462	1450	19.584	11.4	13503889
5	Unknown	Unknown			1506	21.260	4.0	4755658
6	Unknown	Unknown			1524	21.778	3.5	4129372
7	Longifolenaldehyde	OS	610	1631	1627	24.743	5.4	6344206
8	Unknown	Unknown			1635	24.965	4.3	5118002
9	Unknown	Unknown			1670	25.926	4.0	4756728
10	Unknown	Unknown			1676	26.080	1.1	1314380
11	Unknown	Unknown			1741	27.831	3.7	4401649
12	γ-Bicyclohomofarnesal	OS	822	1809	1797	29.307	28.8	33984555
13	Unknown	Unknown			1837	30.306	0.7	882958
14	Unknown	Unknown			1908	32.081	1.3	1527480
15	Unknown	Unknown			1945	32.968	1.8	2160424
16	(*E*)-15,16-Dinorlabda-8(17),11-dien-13-one	OD	605	1994	1984	33.914	10.4	12319712
17	Unknown	Unknown			2000	34.303	1.6	1849436
18	Unknown	Unknown			2016	34.669	2.5	2980428
19	Unknown	Unknown			2356	42.047	2.1	2458925

[a]MH = monoterpene hydrocarbon, OM = oxygenated monoterpene, SH = sesquiterpene hydrocarbon, OS = oxygenated sesquiterpene, DH = diterpene hydrocarbon, OD = oxygenated diterpene, TH = triterpene hydrocarbon, Misc = Miscellaneous, Unknown = match score < 600 or ΔRI > 20.

Table B.12: Volatile chemical composition in fraction FE.A

Peak	Name	Class[a]	Match	NIST RI	Exp. RI	RT	%Area	Area (count·s)
1	β-Copaene	SH	703	1432	1451	19.782	0.1	43430657
2	Caparratriene	SH	705	1493	1489	20.957	0.2	98274792
3	Sesquicineole	OS	632	1516	1524	21.971	0.4	194588441
4	Unknown	Unknown			1553	22.812	0.4	152525780
5	13-nor-Eremophil-1(10)-en-11-one	OS	705	1629	1628	24.960	3.3	1424822902
6	(—)-Isolongifolol, methyl ether	OS	696	1672	1670	26.131	0.8	342104840
7	3-Hydroxy-5,6-epoxy-β-ionone	OS	664	1692	1682	26.455	0.3	109979062
8	Unknown	Unknown			1685	26.544	0.2	73503613
9	γ-Costol	OS	729	1752	1742	28.051	3.2	1380760345
10	Unknown	Unknown			1752	28.326	0.1	38138416
11	Unknown	Unknown			1759	28.504	2.0	846387130
12	Unknown	Unknown			1771	28.834	0.1	44109823
13	γ-Bicyclohomofarnesal	OD	610	1809	1800	29.583	22.3	9638059740
14	Saussurea lactone	OS	637	1806	1815	29.981	0.2	102892534
15	8α-hydroxy-Eremophila-1(10),11-dien-9-one	OS	672	1864	1844	30.693	0.2	95868410
16	Unknown	Unknown			1859	31.075	0.3	148056148
17	iso-Longifolol acetate	OS	782	1850	1870	31.346	0.8	358776509
18	Unknown	Unknown			1894	31.963	0.3	134588823
19	Unknown	Unknown			1926	32.730	2.4	1049252039
20	Unknown	Unknown			1951	33.328	0.9	395687102
21	Unknown	Unknown			1966	33.682	0.4	167335687
22	Unknown	Unknown			1972	33.833	0.1	64466687
23	(*E*)-15,16-Dinorlabda-8(17),11-dien-13-one	OD	763	1994	1985	34.148	10.1	4376936540
24	Ethyl palmitate	Misc.	688	1993	1996	34.410	9.4	4079997360
25	Unknown	Unknown			2011	34.764	0.2	69184676
26	Unknown	Unknown			2016	34.875	0.4	171060953
27	Unknown	Unknown			2035	35.319	0.5	206041177
28	Unknown	Unknown			2051	35.686	0.2	107756868
29	Unknown	Unknown			2075	36.222	0.3	125274397
30	Unknown	Unknown			2088	36.524	0.2	104393958
31	Unknown	Unknown			2094	36.666	0.2	100396145
32	Coronarin E	OD	845	2136	2122	37.301	3.1	1333869866
33	Unknown	Unknown			2144	37.766	0.4	193687835
34	Unknown	Unknown			2152	37.948	0.2	71086057
35	Unknown	Unknown			2164	38.207	9.9	4285283110

36	Ethyl linolenate	Misc.	754	2169	2169	38.330	6.9	2991468720
37	Unknown	Unknown			2188	38.746	0.7	302954498
38	Ethyl octadecanoate	Misc.	751	2195	2195	38.900	1.9	806765252
39	Unknown	Unknown			2200	39.008	1.9	822082976
40	Unknown	Unknown			2210	39.218	1.1	480372122
41	Unknown	Unknown			2282	40.731	6.2	2685037310
42	Unknown	Unknown			2356	42.247	0.2	104594586
43	Ethyl icosanoate	Misc.	678	2395	2395	43.055	0.1	62954887
44	Eicosyl acetate	Misc.	781	2409	2411	43.360	1.7	734725175
45	Retinal	OD	668	2466	2466	44.439	1.1	454752167
46	Unknown	Unknown			2546	45.977	0.5	197023914
47	Villosin	OD	685	2575	2575	46.519	0.6	250610865
48	Docosyl acetate	Misc.	749	2611	2612	47.197	1.4	612957735
49	Unknown	Unknown			2751	49.694	0.3	146414610
50	Unknown	Unknown			2762	49.885	0.3	122656678
51	Unknown	Unknown			2799	50.541	0.5	202770030
52	Unknown	Unknown			2828	51.034	0.4	151642732

[a]MH = monoterpene hydrocarbon, OM = oxygenated monoterpene, SH = sesquiterpene hydrocarbon, OS = oxygenated sesquiterpene, DH = diterpene hydrocarbon, OD = oxygenated diterpene, TH = triterpene hydrocarbon, Misc = Miscellaneous, Unknown = match score < 600 or ΔRI > 20.

Table B.13: Volatile chemical composition in fraction FE.B

Peak	Name	Class[a]	Match	NIST RI	Exp. RI	RT	%Area	Area (count·s)
1	Unknown	Unknown			1467	20.278	2.0	270572872
2	Unknown	Unknown			1635	25.151	2.8	384156045
3	2-Pentadecanone	Misc.	721	1698	1698	26.896	2.3	315586285
4	Isolongifolol	OS	695	1738	1745	28.132	0.7	92476115
5	Unknown	Unknown			1797	29.515	1.8	249189236
6	Unknown	Unknown			1844	30.687	0.2	22988092
7	Valerenic acid	OS	630	1871	1851	30.875	0.2	30289176
8	Unknown	Unknown			1877	31.534	0.4	61529125
9	Corymbolone	OS	680	1899	1882	31.645	0.3	44058632
10	Unknown	Unknown			1899	32.089	0.4	56153196
11	Alantolactone	OS	680	1916	1908	32.295	0.5	64580692
12	Unknown	Unknown			1911	32.376	0.2	20592547
13	6-[1-(Hydroxymethyl)vinyl]-4,8a-dimethyl-3,5,6,7,8,8a-hexahydro-2(1H)-naphthalenone	OS	616	1909	1925	32.715	0.2	25191770
14	Columellarin	OS	664	1958	1967	33.710	0.8	104932771
15	(*E*)-15,16-Dinorlabda-8(17),11-dien-13-one	OD	619	1994	1984	34.129	0.2	23771424
16	Unknown	Unknown			1995	34.379	0.3	47129494
17	Unknown	Unknown			2000	34.505	0.2	24853760
18	Unknown	Unknown			2016	34.875	1.0	134803823
19	Unknown	Unknown			2044	35.516	0.2	34222950
20	Unknown	Unknown			2063	35.963	0.6	86921119
21	Unknown	Unknown			2069	36.096	0.3	41966977
22	*cis*-10-Heptadecenoic acid	Misc.	602	2073	2082	36.385	0.1	18511776
23	Unknown	Unknown			2091	36.610	0.5	61724739
24	Unknown	Unknown			2127	37.393	0.2	27700506
25	Isoretinene a	OD	657	2198	2190	38.792	1.8	245593603
26	Unknown	Unknown			2356	42.263	7.3	997728305
27	Unknown	Unknown			2455	44.226	0.3	39900101
28	Unknown	Unknown			2499	45.083	0.2	30810047
29	Unknown	Unknown			2543	45.906	9.9	1362472745
30	Unknown	Unknown			2612	47.194	1.2	158474368
31	7α,17α-Dimethyl-5β-androstane-3α,17β-diol	OD	648	2656	2652	47.931	2.4	323303127
32	Unknown	Unknown			2756	49.780	25.2	3448132030
33	Calusterone	OD	679	2750	2768	49.996	34.4	4713660360
34	Unknown	Unknown			2793	50.437	0.3	44461202

35	Squalene	TH	621	2832	2828	51.034	0.4	55736460
36	Unknown	Unknown			2858	51.546	0.3	40840531

[a]MH = monoterpene hydrocarbon, OM = oxygenated monoterpene, SH = sesquiterpene hydrocarbon, OS = oxygenated sesquiterpene, DH = diterpene hydrocarbon, OD = oxygenated diterpene, TH = triterpene hydrocarbon, Misc = Miscellaneous, Unknown = match score < 600 or ΔRI > 20.

Table B.14: Volatile chemical composition in fraction FE.C

Peak	Name	Class[a]	Match	NIST RI	Exp. RI	RT	%Area	Area (count·s)
1	Unknown	Unknown			1491	21.012	0.8	11622144
2	Unknown	Unknown			1670	26.131	1.2	17039006
3	Unknown	Unknown			1797	29.515	11.5	168664778
4	Unknown	Unknown			1811	29.879	1.2	17891974
5	Valerenic acid	OS	631	1871	1854	30.948	2.1	30192172
6	Unknown	Unknown			1858	31.050	1.0	15366416
7	Hexadecanoic acid	Misc.	645	1968	1965	33.673	5.5	79830970
8	(*E*)-15,16-Dinorlabda-8(17),11-dien-13-one	OD	606	1994	1984	34.132	6.2	90712033
9	Unknown	Unknown			1989	34.243	5.3	77488130
10	Unknown	Unknown			2006	34.650	1.8	26721319
11	2-*cis*,*cis*-9,12-Octadecadienyloxyethanol	Misc.	643	2120	2138	37.646	4.3	62936077
12	Agatadiol	OD	605	2266	2280	40.694	6.7	98020387
13	Unknown	Unknown			2356	42.260	21.4	313580734
14	Unknown	Unknown			2404	43.224	1.5	21993299
15	Unknown	Unknown			2441	43.958	11.9	174841276
16	Retinal	OD	620	2466	2465	44.429	3.7	54639049
17	Unknown	Unknown			2479	44.691	1.3	19626901
18	Unknown	Unknown			2542	45.890	4.0	57869665
19	Unknown	Unknown			2773	50.085	6.4	94293906
20	Unknown	Unknown			2828	51.034	2.1	30755150

[a]MH = monoterpene hydrocarbon, OM = oxygenated monoterpene, SH = sesquiterpene hydrocarbon, OS = oxygenated sesquiterpene, DH = diterpene hydrocarbon, OD = oxygenated diterpene, TH = triterpene hydrocarbon, Misc = Miscellaneous, Unknown = match score < 600 or DRI > 20.

Table B.15: Volatile chemical composition in fraction FE.D

Peak	Name	Class[a]	Match	NIST RI	Exp. RI	RT	%Area	Area (count·s)
1	Unknown	Unknown			1301	15.134	1.2	42981776
2	Unknown	Unknown			1379	17.594	1.7	61019056
3	Unknown	Unknown			1395	18.087	1.2	43090578
4	4,8-β-Epoxy-caryophyllane	OS	644	1421	1402	18.293	9.8	355803955
5	Unknown	Unknown			1409	18.531	0.9	33571962
6	Unknown	Unknown			1593	23.986	0.7	26482098
7	Unknown	Unknown			1652	25.620	0.6	21857178
8	Unknown	Unknown			1670	26.128	0.5	17392219
9	Unknown	Unknown			1711	27.247	1.6	58041379
10	Unknown	Unknown			1733	27.814	0.4	13839694
11	Unknown	Unknown			1736	27.906	2.3	84672589
12	Methyl isocostate	OS	625	1792	1797	29.521	5.3	191480638
13	Unknown	Unknown			1813	29.928	0.4	15437484
14	Valerenic acid, methyl ester	OS	637	1828	1820	30.101	1.4	52685853
15	Unknown	Unknown			1848	30.801	8.0	292368628
16	Unknown	Unknown			1854	30.958	0.8	30812917
17	Valerenic acid	OS	633	1871	1868	31.312	0.4	13965745
18	Unknown	Unknown			1871	31.386	0.8	27834555
19	Corymbolone	OS	647	1899	1889	31.821	0.9	33126938
20	6-(1-Hydroxymethylvinyl)-4,8a-dimethyl-3,5,6,7,8,8a-hexahydro-1H-naphthalen-2-one	OS	612	1909	1908	32.308	0.4	15958482
21	Unknown	Unknown			1984	34.129	0.6	23576805
22	Unknown	Unknown			1989	34.246	1.1	41564216
23	Unknown	Unknown			2006	34.638	1.4	52271459
24	Unknown	Unknown			2014	34.835	1.8	64076299
25	Unknown	Unknown			2025	35.091	2.3	83790836
26	Unknown	Unknown			2037	35.350	1.0	36187651
27	Unknown	Unknown			2058	35.846	2.4	88141755
28	Unknown	Unknown			2086	36.478	0.6	20565382
29	Unknown	Unknown			2089	36.558	1.3	47282550
30	Unknown	Unknown			2103	36.878	5.4	194519299
31	Isoretinene a	OD	642	2198	2215	39.322	2.4	86784604
32	Unknown	Unknown			2223	39.483	5.7	207383650
33	Reynosin	OS	707	2266	2269	40.460	6.6	238623123
34	Unknown	Unknown			2272	40.531	0.9	32831038

35	Unknown	Unknown			2281	40.716	3.6	130954539
36	Unknown	Unknown			2356	42.263	10.6	383942859
37	Unknown	Unknown			2390	42.935	0.4	15452301
38	(*E*)-Labda-8(17),12-diene-15,16-dial	OD	690	2383	2403	43.203	2.4	86615188
39	Retinal	OD	652	2466	2466	44.433	3.0	110722150
40	Unknown	Unknown			2542	45.887	0.7	27177379
41	Unknown	Unknown			2565	46.316	0.6	20760954
42	Unknown	Unknown			2616	47.265	0.8	27502927
43	Unknown	Unknown			2651	47.909	2.2	79048835
44	Unknown	Unknown			2752	49.715	0.7	27047764
45	Unknown	Unknown			2774	50.097	1.4	51897128
46	Unknown	Unknown			2828	51.031	0.6	22368779

[a]MH = monoterpene hydrocarbon, OM = oxygenated monoterpene, SH = sesquiterpene hydrocarbon, OS = oxygenated sesquiterpene, DH = diterpene hydrocarbon, OD = oxygenated diterpene, TH = triterpene hydrocarbon, Misc = Miscellaneous, Unknown = match score < 600 or DRI > 20.

Table B.16: Volatile chemical composition in fraction FE.E

Peak	Name	Class[a]	Match	NIST RI	Exp. RI	RT	%Area	Area (count·s)
1	Unknown	Unknown			1330	16.050	1.2	30693893
2	Unknown	Unknown			1338	16.287	0.7	19504844
3	Unknown	Unknown			1395	18.078	1.0	26922659
4	Unknown	Unknown			1400	18.241	2.5	67692400
5	Unknown	Unknown			1422	18.916	0.4	10419599
6	Unknown	Unknown			1670	26.122	0.5	14103686
7	Unknown	Unknown			1736	27.903	2.8	75109705
8	β-Costol	OS	608	1778	1797	29.518	14.4	381966898
9	Unknown	Unknown			1824	30.184	6.1	163347221
10	Unknown	Unknown			1848	30.788	1.2	31978928
11	Unknown	Unknown			1851	30.874	0.3	9138654
12	Unknown	Unknown			1871	31.377	0.9	23092691
13	6-(1-Hydroxymethylvinyl)-4,8a-dimethyl-3,5,6,7,8,8a-hexahydro-1H-naphthalen-2-one	OS	626	1909	1889	31.821	0.8	21336470
14	Unknown	Unknown			1895	31.981	1.3	34740564
15	(*E*)-15,16-Dinorlabda-8(17),11-dien-13-one	OD	620	1994	1984	34.117	2.0	53923595
16	Unknown	Unknown			2026	35.097	3.8	100341943
17	Unknown	Unknown			2126	37.375	2.6	68849978
18	Unknown	Unknown			2188	38.731	0.9	25032563
19	Unknown	Unknown			2215	39.332	1.4	36668795
20	Sclareol	OD	676	2227	2245	39.945	3.4	91675480
21	Unknown	Unknown			2273	40.540	9.3	248401442
22	Unknown	Unknown			2293	40.971	2.6	67936260
23	Unknown	Unknown			2356	42.263	22.3	593608318
24	Unknown	Unknown			2396	43.070	1.0	26291412
25	Unknown	Unknown			2441	43.946	1.5	39810435
26	Unknown	Unknown			2466	44.433	2.4	64437679
27	Unknown	Unknown			2474	44.602	0.4	10474774
28	Unknown	Unknown			2480	44.722	1.0	27502743
29	Unknown	Unknown			2542	45.890	0.4	10303685
30	Unknown	Unknown			2637	47.650	2.1	55820944
31	Unknown	Unknown			2651	47.903	7.9	208651685
32	Unknown	Unknown			2828	51.031	0.7	17573835

[a]MH = monoterpene hydrocarbon, OM = oxygenated monoterpene, SH = sesquiterpene hydrocarbon, OS = oxygenated sesquiterpene, DH = diterpene hydrocarbon, OD = oxygenated diterpene, TH = triterpene hydrocarbon, Misc = Miscellaneous, Unknown = match score < 600 or DRI > 20.

Table B.17: Volatile chemical composition in fraction **FE.F**

Peak	Name	Class[a]	Match	NIST RI	Exp. RI	RT	%Area	Area (count·s)
1	γ-Bicyclohomofarnesal	OD	687	1809	1797	29.518	33.4	160886704
2	Unknown	Unknown			1804	29.685	5.5	26719644
3	Unknown	Unknown			1984	34.120	6.8	32769743
4	Sclareol	OD	614	2227	2245	39.948	12.0	57708261
5	Unknown	Unknown			2356	42.250	30.3	145731402
6	Unknown	Unknown			2405	43.237	3.4	16241689
7	Unknown	Unknown			2546	45.971	2.1	10198971
8	Unknown	Unknown			2561	46.254	3.6	17151240
9	Unknown	Unknown			2828	51.034	3.0	14227635

[a]MH = monoterpene hydrocarbon, OM = oxygenated monoterpene, SH = sesquiterpene hydrocarbon, OS = oxygenated sesquiterpene, DH = diterpene hydrocarbon, OD = oxygenated diterpene, TH = triterpene hydrocarbon, Misc = Miscellaneous, Unknown = match score < 600 or DRI > 20.

Table B.18: Volatile chemical composition in fraction **FE.G**

Peak	Name	Class[a]	Match	NIST RI	Exp. RI	RT	%Area	Area (count·s)
1	Unknown	Unknown			1635	25.142	0.6	3978307
2	Unknown	Unknown			1670	26.128	1.9	13353440
3	β-Costol	OS	606	1778	1797	29.518	43.2	302495142
4	Unknown	Unknown			1880	31.614	0.5	3538669
5	Unknown	Unknown			1925	32.702	1.3	8777382
6	(*E*)-15,16-Dinorlabda-8(17),11-dien-13-one	OD	620	1994	1983	34.111	11.5	80381689
7	Unknown	Unknown			2017	34.909	1.9	13097190
8	Unknown	Unknown			2047	35.599	1.2	8357538
9	Unknown	Unknown			2074	36.210	1.6	11370752
10	Unknown	Unknown			2190	38.786	1.6	11093418
11	Unknown	Unknown			2356	42.247	29.3	204752351
12	Unknown	Unknown			2404	43.227	1.6	11524826
13	Unknown	Unknown			2465	44.420	0.8	5771475
14	Unknown	Unknown			2617	47.293	0.9	6059466
15	Unknown	Unknown			2828	51.031	2.2	15158399

[a]MH = monoterpene hydrocarbon, OM = oxygenated monoterpene, SH = sesquiterpene hydrocarbon, OS = oxygenated sesquiterpene, DH = diterpene hydrocarbon, OD = oxygenated diterpene, TH = triterpene hydrocarbon, Misc = Miscellaneous, Unknown = match score < 600 or DRI > 20.

Table B.19: Volatile chemical composition in fraction **FG.A**

Peak	Name	Class[a]	Match	NIST RI	Exp. RI	RT	%Area	Area (count·s)
1	Unknown	Unknown			1030	7.201	1.7	7087224
2	Unknown	Unknown			1058	7.984	1.8	7497829
3	Unknown	Unknown			1064	8.138	0.7	2836009
4	Acetophenone	Misc.	614	1065	1069	8.292	1.8	7252793
5	Unknown	Unknown			1104	9.279	2.0	8231572
6	Unknown	Unknown			1120	9.775	10.3	42197589
7	Unknown	Unknown			1602	24.695	5.6	22879031
8	Unknown	Unknown			1675	26.723	1.7	6997414
9	Unknown	Unknown			1752	28.804	2.3	9481495
10	13-Tetradecenyl acetate	Misc.	651	1805	1807	30.234	3.8	15822094
11	Unknown	Unknown			1836	30.973	1.8	7413999
12	Unknown	Unknown			1861	31.614	2.6	10683482
13	Palmityl acetate	Misc.	626	2009	2012	35.270	24.8	102000373
14	Unknown	Unknown			2060	36.382	2.8	11319746
15	Unknown	Unknown			2067	36.546	4.5	18725914
16	Unknown	Unknown			2101	37.332	3.1	12783117
17	Unknown	Unknown			2139	38.167	3.8	15626559
18	Octadecanol acetate	Misc.	727	2208	2208	39.877	15.7	64482176
19	Unknown	Unknown			2236	41.055	1.2	4896554
20	Unknown	Unknown			2275	42.691	5.7	23652672
21	Unknown	Unknown			2285	43.123	2.4	9707928

[a]MH = monoterpene hydrocarbon, OM = oxygenated monoterpene, SH = sesquiterpene hydrocarbon, OS = oxygenated sesquiterpene, DH = diterpene hydrocarbon, OD = oxygenated diterpene, TH = triterpene hydrocarbon, Misc = Miscellaneous, Unknown = match score < 600 or ΔRI > 20

Table B.20: Volatile chemical composition in fraction FG.B

Peak	Name	Class	Match	NIST RI	Exp. RI	RT	%Area	Area (count·s)
1	Unknown	Unknown			1601	24.692	0.2	68021403
2	Unknown	Unknown			1631	25.512	0.1	19477602
3	Unknown	Unknown			1638	25.715	0.1	29568598
4	(-)-Isolongifolol, methyl ether	OS	634	1672	1673	26.683	0.2	60020814
5	Isolongifolol	OS	657	1738	1748	28.702	0.1	17661107
6	γ-Bicyclohomofarnesal	OD	712	1809	1800	30.070	4.6	1399648970
7	Unknown	Unknown			1847	31.248	0.1	24172847
8	Unknown	Unknown			1862	31.624	0.3	102644994
9	Unknown	Unknown			1874	31.941	0.1	21981665
10	Unknown	Unknown			1883	32.154	1.1	335360527
11	Alantolactone	OS	608	1916	1912	32.875	0.1	27407373
12	Unknown	Unknown			1920	33.066	0.1	16408234
13	Unknown	Unknown			1928	33.273	1.8	548067527
14	Unknown	Unknown			1936	33.448	0.2	71553531
15	Pimaradiene	DH	696	1960	1954	33.880	1.9	578993423
16	Valerenic acid, n-propyl ester	OS	605	1971	1964	34.139	0.1	21190349
17	Dibutyl phthalate	Misc.	753	1965	1968	34.237	0.8	234492340
18	(*E*)-15,16-Dinorlabda-8(17),11-dien-13-one	OD	590	1994	1998	34.949	71.2	21474836470
19	Unknown	Unknown			2013	35.291	0.4	118238909
20	Unknown	Unknown			2019	35.448	0.2	48933293
21	Unknown	Unknown			2022	35.516	0.1	36356210
22	Epimanool	OD	721	2056	2039	35.908	14.0	4209461680
23	Unknown	Unknown			2112	37.584	0.2	61830377
24	Unknown	Unknown			2127	37.917	0.3	82640041
25	Unknown	Unknown			2139	38.173	0.2	53192227
26	Unknown	Unknown			2171	38.882	0.5	163287582
27	Octadecanol acetate	Misc.	652	2208	2208	39.859	0.2	66191610
28	Agatadiol	OD	626	2266	2250	41.625	0.8	230791847
29	Larixol	OD	625	2264	2269	42.420	0.1	22148864
30	Unknown	Unknown			2274	42.667	0.1	28317556

[a]MH = monoterpene hydrocarbon, OM = oxygenated monoterpene, SH = sesquiterpene hydrocarbon, OS = oxygenated sesquiterpene, DH = diterpene hydrocarbon, OD = oxygenated diterpene, TH = triterpene hydrocarbon, Misc = Miscellaneous, Unknown = match score < 600 or ΔRI > 20.

Table B.21: Volatile chemical composition in fraction FG.C

Peak	Name	Class[a]	Match	NIST RI	Exp. RI	RT	%Area	Area (count·s)
1	Unknown	Unknown			1355	17.270	0.1	16295266
2	Unknown	Unknown			1391	18.402	0.1	20554112
3	Unknown	Unknown			1502	21.786	0.2	27586795
4	Unknown	Unknown			1526	22.495	0.6	93511754
5	Unknown	Unknown			1555	23.336	0.1	19089494
6	Unknown	Unknown			1612	24.988	0.8	119901804
7	9-Cedranone	OS	619	1618	1630	25.487	1.2	183080541
8	Longifolenaldehyde	OS	639	1631	1637	25.685	5.9	888261350
9	(-)-Isolongifolol, methyl ether	OS	683	1672	1673	26.674	1.2	185082003
10	Unknown	Unknown			1684	26.988	0.9	132440420
11	Unknown	Unknown			1702	27.478	0.4	57644454
12	Unknown	Unknown			1716	27.839	0.1	12129336
13	Unknown	Unknown			1744	28.591	0.3	40422954
14	Isolongifolol	OS	715	1738	1748	28.683	0.7	109516786
15	Unknown	Unknown			1752	28.794	0.4	55193147
16	Unknown	Unknown			1761	29.029	0.3	43382475
17	Unknown	Unknown			1775	29.411	0.1	19515788
18	Unknown	Unknown			1787	29.725	0.1	12955035
19	Unknown	Unknown			1793	29.876	0.1	17777133
20	γ-Bicyclohomofarnesal	OD	657	1809	1800	30.073	20.2	3039584630
21	Unknown	Unknown			1807	30.234	0.4	54401185
22	Isopropyl myristate	Misc.	644	1827	1827	30.758	0.1	19466797
23	Unknown				1838	31.032	0.1	10991894
24	8α-hydroxy-Eremophila-1(10),11-dien-9-one	OS	624	1864	1847	31.248	0.2	28832354
25	Unknown	Unknown			1861	31.617	0.2	31764603
26	Unknown	Unknown			1881	32.104	0.7	111544480
27	Alantolactone	OS	663	1916	1903	32.662	6.9	1036605167
28	Unknown	Unknown			1911	32.860	1.8	270419804
29	Unknown	Unknown			1915	32.952	0.6	90564457
30	7,9-Di-tert-butyl-1-oxaspiro(4,5)deca-6,9-diene-2,8-dione	OD	829	1923	1919	33.041	0.2	35514635
31	Methyl palmitate	Misc.	682	1926	1928	33.254	1.1	173178287
32	Unknown	Unknown			1935	33.424	0.2	37294279
33	Unknown	Unknown			1941	33.572	0.1	19872841
34	Unknown	Unknown			1944	33.658	0.3	37829492

35	Unknown	Unknown			1948	33.750	0.1	21215251
36	Unknown	Unknown			1951	33.824	1.1	162773065
37	Dibutyl phthalate	Misc.	784	1965	1963	34.102	1.3	188315886
38	Unknown	Unknown			1971	34.290	1.6	234392297
39	Unknown	Unknown			1980	34.524	0.3	41197812
40	(*E*)-15,16-Dinorlabda-8(17),11-dien-13-one	OD	753	1994	1987	34.681	28.5	4289749790
41	Unknown	Unknown			1993	34.838	0.1	15752056
42	Unknown	Unknown			2011	35.264	1.7	257868348
43	Unknown	Unknown			2020	35.464	3.6	547725227
44	Unknown	Unknown			2035	35.797	0.3	45292591
45	Epimanool	OD	723	2056	2038	35.871	2.8	419355629
46	Linoleyl methyl ketone	Misc.	670	2075	2067	36.555	1.5	222077257
47	Unknown	Unknown			2072	36.663	0.2	26790211
48	*cis*-10-Heptadecenoic acid	Misc.	624	2073	2085	36.956	0.3	47534674
49	Unknown	Unknown			2090	37.085	0.5	68941419
50	Unknown	Unknown			2096	37.211	1.2	178202557
51	Sclareolide	OD	633	2089	2104	37.393	2.4	365193979
52	Unknown	Unknown			2111	37.563	0.2	37238226
53	Unknown	Unknown			2127	37.908	0.2	31924419
54	Unknown	Unknown			2138	38.151	0.2	32008851
55	Octadecanol acetate	Misc.	646	2208	2205	39.733	0.2	29684622
56	Unknown	Unknown			2208	39.859	0.6	93579868
57	Unknown	Unknown			2235	41.027	0.4	65900861
58	Unknown	Unknown			2242	41.295	0.1	21397526
59	Agatadiol	OD	632	2266	2249	41.606	2.9	435764389
60	Larixol	OD	678	2264	2278	42.815	0.7	109318548
61	Unknown	Unknown			2287	43.172	0.1	20545021

[a]MH = monoterpene hydrocarbon, OM = oxygenated monoterpene, SH = sesquiterpene hydrocarbon, OS = oxygenated sesquiterpene, DH = diterpene hydrocarbon, OD = oxygenated diterpene, TH = triterpene hydrocarbon, Misc = Miscellaneous, Unknown = match score < 600 or DRI > 20.

Table B.22: Volatile chemical composition in fraction FG.D

Peak	Name	Class[a]	Match	NIST RI	Exp. RI	RT	%Area	Area (count·s)
1	*p*-Cymene	MH	729	1025	1028	7.130	0.1	12854459
2	Camphol	OM	675	1167	1167	11.260	0.1	10113744
3	Unknown	Unknown			1355	17.274	0.1	11180125
4	Unknown	Unknown			1374	17.850	0.1	11299992
5	Unknown	Unknown			1378	17.979	0.2	17069520
6	Unknown	Unknown			1390	18.380	10.2	1053394794
7	Unknown	Unknown			1401	18.707	0.3	31910625
8	Unknown	Unknown			1421	19.311	0.1	9704998
9	Unknown	Unknown			1449	20.171	0.6	64658076
10	(±)-Gymnomitrene	SH	614	1452	1462	20.574	3.0	307276208
11	Unknown	Unknown			1484	21.259	0.4	41762869
12	Unknown	Unknown			1492	21.502	1.2	123895795
13	Unknown	Unknown			1502	21.789	0.2	21546355
14	Unknown	Unknown			1526	22.498	0.8	81536079
15	Unknown	Unknown			1555	23.327	0.2	16203679
16	Unknown	Unknown			1601	24.667	0.6	57370711
17	Unknown	Unknown			1621	25.222	0.7	68416012
18	Unknown	Unknown			1630	25.497	0.5	48371493
19	Unknown	Unknown			1638	25.694	1.1	112242779
20	(-)-Isolongifolol, methyl ether	OS	666	1672	1673	26.671	1.4	141270713
21	Unknown	Unknown			1679	26.840	0.7	75421172
22	Unknown	Unknown			1684	26.985	0.5	46474961
23	Unknown	Unknown			1719	27.913	0.2	16639571
24	8α,11-Elemadiol	OS	703	1745	1735	28.354	0.8	83047424
25	Unknown	Unknown			1740	28.480	0.7	69821083
26	Unknown	Unknown			1744	28.582	0.2	21291680
27	Isolongifolol	OS	642	1738	1747	28.671	0.2	22924331
28	β-Santanol acetate	OS	646	1797	1788	29.741	0.3	35336338
29	γ-Bicyclohomofarnesal	OD	769	1809	1800	30.067	12.6	1291693288
30	Saussurea lactone	OS	677	1806	1807	30.240	1.3	131816874
31	iso-Longifolol acetate	OS	646	1850	1864	31.679	1.7	171427594
32	Clovanediol	OS	789	1885	1872	31.883	1.9	191081057
33	Corymbolone	OS	660	1899	1890	32.329	0.5	47724766
34	Ledene oxide-(I)	OS	698	1890	1903	32.672	4.5	464392185
35	Alantolactone	OS	686	1916	1911	32.863	1.9	195391753

36	Unknown	Unknown			1928	33.266	0.5	48936502
37	Unknown	Unknown			1941	33.568	1.2	119355016
38	Unknown	Unknown			1948	33.753	0.2	24671813
39	Unknown	Unknown			1951	33.818	0.6	59336985
40	Dibutyl phthalate	Misc.	630	1965	1963	34.095	1.3	138569364
41	β-Cyclocostunolide	OS	715	1983	1970	34.287	2.4	249459008
42	Unknown	Unknown			1976	34.422	0.4	36041653
43	(*E*)-15,16-Dinorlabda-8(17),11-dien-13-one	OD	747	1994	1986	34.672	29.6	3045571470
44	Unknown	Unknown			2011	35.257	0.4	40488697
45	Unknown	Unknown			2020	35.458	1.1	113797134
46	Unknown	Unknown			2031	35.707	0.5	55003830
47	Unknown	Unknown			2038	35.865	0.2	19096103
48	Unknown	Unknown			2041	35.954	0.2	16682399
49	Valerenic acid, n-butyl ester	OS	600	2065	2067	36.543	0.4	39047916
50	Unknown	Unknown			2079	36.826	1.0	100838424
51	Unknown	Unknown			2124	37.834	0.4	42064372
52	Unknown	Unknown			2129	37.945	2.8	292243759
53	Unknown	Unknown			2150	38.429	0.2	19388387
54	Unknown	Unknown			2162	38.700	0.2	22564192
55	Ethyl linolenate	Misc.	677	2169	2170	38.873	0.9	95584155
56	2-Hydroxy-1-(hydroxymethyl)ethyl (9E,12E,15E)-9,12,15-octadecatrienoate	Misc.	631	2161	2176	39.002	0.4	39721912
57	Unknown	Unknown			2189	39.295	0.3	29139301
58	Unknown	Unknown			2210	39.979	1.1	108224052
59	Unknown	Unknown			2232	40.870	2.7	278347807
60	Unknown	Unknown			2242	41.308	0.4	38980232
61	Unknown	Unknown			2260	42.063	0.5	54922589
62	Larixol	OD	677	2264	2279	42.836	0.5	52851800

[a]MH = monoterpene hydrocarbon, OM = oxygenated monoterpene, SH = sesquiterpene hydrocarbon, OS = oxygenated sesquiterpene, DH = diterpene hydrocarbon, OD = oxygenated diterpene, TH = triterpene hydrocarbon, Misc = Miscellaneous, Unknown = match score < 600 or DRI > 20.

Table B.23: Volatile chemical composition in fraction **FG.E**

Peak	Name	Class[a]	Match	NIST RI	Exp. RI	RT	%Area	Area (count·s)
1	Unknown	Unknown			1120	9.778	0.2	8691629
2	Camphol	OM	695	1167	1167	11.267	0.2	10623789
3	exo-2-Hydroxycineole	OM	642	1224	1224	13.060	0.2	9988680
4	2-Methylbenzothiazole	Misc.	810	1281	1293	15.304	1.1	54000658
5	Sobrerol	OM	648	1388	1390	18.374	7.5	363654018
6	Unknown	Unknown			1401	18.719	1.0	48784142
7	β-Copaene	SH	603	1432	1452	20.275	0.4	20156622
8	Unknown	Unknown			1462	20.574	0.5	23703853
9	Unknown	Unknown			1492	21.484	1.9	92505338
10	Unknown	Unknown			1501	21.776	6.8	330570969
11	Unknown	Unknown			1511	22.060	0.6	28628689
12	Sobrerol 8-acetate	OM	636	1529	1525	22.470	23.6	1149281066
13	Unknown	Unknown			1554	23.299	0.5	25427643
14	Unknown	Unknown			1562	23.546	0.5	23104094
15	Unknown	Unknown			1612	24.985	0.5	22139467
16	13-nor-Eremophil-1(10)-en-11-one	OS	608	1629	1630	25.490	0.3	16655765
17	Unknown	Unknown			1638	25.700	0.6	28362067
18	Aromadendrene oxide-(2)	OS	693	1678	1673	26.671	1.8	89594372
19	Unknown	Unknown			1684	26.985	0.3	14872262
20	Isolongifolol	OS	652	1738	1735	28.354	0.8	40397862
21	Unknown	Unknown			1744	28.597	0.4	21053593
22	Unknown	Unknown			1752	28.800	0.2	11280731
23	Unknown	Unknown			1771	29.312	0.2	11610621
24	β-Costol	OS	688	1778	1788	29.750	1.7	81388161
25	γ-Bicyclohomofarnesal	OD	665	1809	1800	30.070	29.4	1434359072
26	Unknown	Unknown			1807	30.243	0.4	19323422
27	Unknown	Unknown			1812	30.372	0.3	12770633
28	Unknown	Unknown			1827	30.739	0.8	36895471
29	Cyperadione	OS	601	1845	1851	31.343	0.4	19549671
30	Unknown	Unknown			1904	32.675	0.9	43831992
31	Alantolactone	OS	639	1916	1911	32.856	0.6	27825732
32	Unknown	Unknown			1928	33.266	0.5	23334451
33	Unknown	Unknown			1952	33.840	1.0	49075807
34	Dibutyl phthalate	Misc.	694	1965	1964	34.123	0.7	32084096
35	Clovanediol,monoacetate	OS	716	1982	1971	34.299	1.5	72889775

36	(*E*)-15,16-Dinorlabda-8(17),11-dien-13-one	OD	766	1994	1986	34.669	9.4	458514397
37	Unknown	Unknown			1995	34.888	0.2	12197671
38	Unknown	Unknown			2012	35.270	0.5	23506553
39	Unknown	Unknown			2151	38.444	0.5	23348614
40	Unknown	Unknown			2171	38.897	0.2	7519193
41	Unknown	Unknown			2182	39.144	0.2	11974989
42	Unknown	Unknown			2208	39.877	0.3	14928556
43	Unknown	Unknown			2220	40.389	0.2	7581548
44	Larixol	OD	611	2264	2279	42.852	0.4	21924419

[a]MH = monoterpene hydrocarbon, OM = oxygenated monoterpene, SH = sesquiterpene hydrocarbon, OS = oxygenated sesquiterpene, DH = diterpene hydrocarbon, OD = oxygenated diterpene, TH = triterpene hydrocarbon, Misc = Miscellaneous, Unknown = match score < 600 or DRI > 20.

Table B.24: Volatile chemical composition in fraction **FG.F**

Peak	Name	Class[a]	Match	NIST RI	Exp. RI	RT	%Area	Area (count·s)
1	Camphol	OM	659	1167	1167	11.263	0.3	12420320
2	Unknown	Unknown			1270	14.552	0.2	6788737
3	Unknown	Unknown			1370	17.733	0.5	19336298
4	Unknown	Unknown			1390	18.377	3.2	120657289
5	Unknown	Unknown			1397	18.587	0.9	31770338
6	Unknown	Unknown			1401	18.722	1.6	57881984
7	β-Copaene	SH	631	1432	1452	20.282	0.6	20903334
8	Unknown	Unknown			1491	21.474	0.2	9274283
9	Unknown	Unknown			1526	22.501	0.5	20075915
10	Unknown	Unknown			1630	25.490	0.6	21387219
11	Unknown	Unknown			1638	25.709	1.2	44431336
12	Unknown	Unknown			1673	26.677	2.8	103454866
13	Unknown	Unknown			1684	26.988	0.6	22349217
14	Unknown	Unknown			1735	28.360	0.4	14008405
15	Unknown	Unknown			1744	28.594	0.9	32598335
16	Unknown	Unknown			1752	28.797	0.3	11105209
17	Unknown	Unknown			1771	29.303	0.3	12068082
18	γ-Bicyclohomofarnesal	OD	691	1809	1800	30.064	46.6	1731286539
19	(*Z*)-7-Hexadecenal	Misc.	608	1798	1807	30.240	0.6	22280919
20	Unknown	Unknown			1827	30.742	0.6	22191442
21	Cyperadione	OS	663	1845	1851	31.343	6.0	223953089
22	Unknown	Unknown			1861	31.611	0.3	11718137
23	Unknown	Unknown			1874	31.938	2.1	79884271
24	Alantolactone	OS	621	1916	1903	32.659	1.5	55554456
25	Unknown	Unknown			1912	32.869	0.2	6935319
26	Unknown	Unknown			1915	32.958	0.1	4826635
27	7,9-Di-tert-butyl-1-oxaspiro(4,5)deca-6,9-diene-2,8-dione	OD	668	1923	1919	33.041	0.3	9811680
28	Methyl palmitate	Misc.	647	1926	1928	33.263	1.9	69117272
29	Unknown	Unknown			1935	33.427	0.4	13613690
30	Unknown	Unknown			1951	33.827	1.2	44039882
31	Dibutyl phthalate	Misc.	761	1965	1963	34.095	1.6	58333501
32	Unknown	Unknown			1980	34.512	0.4	15636543
33	(*E*)-15,16-Dinorlabda-8(17),11-dien-13-one	OD	799	1994	1986	34.666	14.8	549077075
34	Unknown	Unknown			2011	35.264	1.1	42741578

35	Unknown	Unknown			2020	35.461	0.8	31573171
36	Unknown	Unknown			2038	35.877	0.4	13222104
37	Unknown	Unknown			2077	36.783	0.4	13841684
38	Unknown	Unknown			2093	37.156	0.5	16977512
39	Unknown	Unknown			2171	38.888	0.4	15619674
40	Unknown	Unknown			2182	39.138	0.7	25723161
41	Unknown	Unknown			2208	39.859	0.7	25606300
42	Unknown	Unknown			2274	42.661	0.3	10607970
43	Larixol	OD	636	2264	2279	42.836	1.2	43418893

[a]MH = monoterpene hydrocarbon, OM = oxygenated monoterpene, SH = sesquiterpene hydrocarbon, OS = oxygenated sesquiterpene, DH = diterpene hydrocarbon, OD = oxygenated diterpene, TH = triterpene hydrocarbon, Misc = Miscellaneous, Unknown = match score < 600 or DRI > 20.

Table B.25: Volatile chemical composition in fraction **FG.G**

Peak	Name	Class[a]	Match	NIST RI	Exp. RI	RT	%Area	Area (count·s)
1	Unknown	Unknown			1126	9.966	0.2	26730729
2	Camphol	OM	723	1167	1167	11.257	0.1	19489926
3	3,7-Dimethyl-1,5-octadiene-3,7-diol	OM	760	1190	1190	11.966	0.3	38370384
4	Unknown	Unknown			1218	12.869	0.1	15460112
5	*trans*-Ascaridol glycol	OM	664	1272	1270	14.540	0.1	18390322
6	2-Methylbenzothiazole	Misc.	837	1281	1293	15.298	1.1	151487860
7	Unknown	Unknown			1302	15.581	0.1	14217487
8	Unknown	Unknown			1311	15.868	0.1	14723425
9	(+)-Pinanediol	OM	719	1316	1317	16.041	1.5	195098362
10	Unknown	Unknown			1320	16.149	3.5	468184198
11	Unknown	Unknown			1373	17.841	0.4	56559085
12	Unknown	Unknown			1378	17.976	0.3	35512871
13	Unknown	Unknown			1390	18.374	1.4	181314110
14	Unknown	Unknown			1400	18.697	0.4	50615058
15	β-Copaene	SH	627	1432	1452	20.275	0.2	20361591
16	Unknown	Unknown			1525	22.479	0.4	58856795
17	Unknown	Unknown			1543	23.000	0.2	28858648
18	Unknown	Unknown			1554	23.314	0.3	40477784
19	Unknown	Unknown			1630	25.490	0.3	41132314
20	Unknown	Unknown			1637	25.688	0.5	60057866
21	Unknown	Unknown			1673	26.668	0.8	102778810
22	Unknown	Unknown			1684	26.985	0.5	64902380
23	Unknown	Unknown			1716	27.845	0.1	18941661
24	Isolongifolol	OS	633	1738	1719	27.913	0.1	15389062
25	Unknown	Unknown			1735	28.347	0.1	12336204
26	Unknown	Unknown			1744	28.582	0.3	44306356
27	Unknown	Unknown			1752	28.788	0.1	9747205
28	β-Costol	OS	634	1778	1774	29.386	0.8	100211293
29	Dehydrovomifoliol	OS	825	1800	1789	29.765	1.4	180753170
30	Unknown	Unknown			1793	29.879	0.1	11426108
31	γ-Bicyclohomofarnesal	OD	676	1809	1800	30.061	13.0	1730698291
32	Unknown	Unknown			1807	30.249	1.1	144360943
33	Saussurea lactone	OS	664	1806	1814	30.409	0.9	113756794
34	Unknown	Unknown			1826	30.724	0.5	71676925
35	Unknown	Unknown			1840	31.075	0.1	8610161

36	Cyperadione	OS	622	1845	1850	31.328	0.2	21681222
37	Unknown	Unknown			1874	31.938	0.4	47936266
38	Unknown	Unknown			1887	32.271	0.1	11743623
39	Unknown	Unknown			1895	32.450	0.1	14665750
40	Unknown	Unknown			1903	32.653	0.7	87714947
41	Unknown	Unknown			1915	32.946	0.1	10664383
42	7,9-Di-tert-butyl-1-oxaspiro(4,5)deca-6,9-diene-2,8-dione	OD	649	1923	1918	33.029	0.1	13029600
43	Methyl palmitate	Misc.	617	1926	1927	33.239	0.4	52472033
44	Unknown	Unknown			1935	33.424	0.1	11337063
45	Unknown	Unknown			1940	33.559	0.2	20729040
46	Unknown	Unknown			1947	33.729	0.1	19966051
47	Unknown	Unknown			1951	33.815	0.3	40274841
48	Dibutyl phthalate	Misc.	618	1965	1962	34.083	1.0	132615509
49	Unknown	Unknown			1971	34.296	0.2	25270706
50	(*E*)-15,16-Dinorlabda-8(17),11-dien-13-one	OD	646	1994	1983	34.586	11.2	1492109933
51	Unknown	Unknown			2004	35.082	0.2	24631204
52	Unknown	Unknown			2020	35.455	0.1	18959212
53	Unknown	Unknown			2033	35.757	50.6	6742427140
54	Unknown	Unknown			2047	36.077	0.8	107700472
55	Valerenic acid, *n*-butyl ester	OS	601	2065	2067	36.543	0.1	19730438
56	Unknown	Unknown			2128	37.942	0.2	24187172
57	Unknown	Unknown			2170	38.876	0.3	35258717
58	*cis*-9,10-Methyleneoctadecanoic acid methyl ester	Misc.	604	2212	2207	39.853	0.2	24923110
59	Sclareol	OD	690	2227	2223	40.519	0.9	115480877
60	Unknown	Unknown			2278	42.821	0.4	52002633

[a]MH = monoterpene hydrocarbon, OM = oxygenated monoterpene, SH = sesquiterpene hydrocarbon, OS = oxygenated sesquiterpene, DH = diterpene hydrocarbon, OD = oxygenated diterpene, TH = triterpene hydrocarbon, Misc = Miscellaneous, Unknown = match score < 600 or DRI > 20.

Table B.26: Volatile chemical composition in fraction FG.H

Peak	Name	Class[a]	Match	NIST RI	Exp. RI	RT	%Area	Area (count·s)
1	Unknown	Unknown			1219	12.894	0.2	36202667
2	*trans*-Ascaridol glycol	OM	759	1272	1287	15.095	0.3	60876388
3	2-Methylbenzothiazole	Misc.	650	1281	1293	15.307	0.1	33245734
4	Unknown	Unknown			1304	15.646	0.2	43497071
5	Unknown	Unknown			1317	16.047	0.1	20309846
6	Unknown	Unknown			1320	16.152	1.0	234643978
7	Unknown	Unknown			1332	16.515	0.2	40014738
8	Unknown	Unknown			1338	16.713	0.1	24749604
9	2-Hydroxycineole, acetate	OM	639	1344	1347	17.015	0.2	34232568
10	Unknown	Unknown			1355	17.246	0.1	15641653
11	Unknown	Unknown			1373	17.834	0.6	127563576
12	Sobrerol	OM	656	1388	1377	17.964	2.4	541494224
13	Unknown	Unknown			1382	18.127	0.5	102337011
14	Unknown	Unknown			1390	18.380	8.4	1881077935
15	Unknown	Unknown			1401	18.701	0.2	50392372
16	Unknown	Unknown			1417	19.200	0.1	16712103
17	Unknown	Unknown			1421	19.311	0.3	70683222
18	Unknown	Unknown			1431	19.644	0.1	13985075
19	Unknown	Unknown			1462	20.565	0.2	36919583
20	1,2,4-Trihydroxy menthane	OM	812	1487	1477	21.034	0.9	201257750
21	Unknown	Unknown			1501	21.770	0.1	22204865
22	Kessane	OS	621	1537	1525	22.458	0.7	153794172
23	5,5-Dimethyl-4-(3-oxobutyl)dihydro-2(3H)-furanone	OM	744	1558	1550	23.197	0.8	178314130
24	Unknown	Unknown			1611	24.951	0.2	51032200
25	Longifolenaldehyde	OS	622	1631	1630	25.487	0.3	69440883
26	Unknown	Unknown			1637	25.678	0.4	81242462
27	(-)-Isolongifolol, methyl ether	OS	651	1672	1673	26.665	1.4	305875147
28	Unknown	Unknown			1684	26.979	0.3	69055814
29	Unknown	Unknown			1697	27.337	0.2	40367939
30	Blumenol C	OS	677	1711	1702	27.475	0.3	59994008
31	Unknown	Unknown			1710	27.688	0.8	172492525
32	3-Hydroxy-1a,3,6,6-tetramethylhexahydro-2-oxacyclopropa[d]naphthalen-5-one	OS	655	1716	1719	27.913	0.6	136891033
33	8α,11-Elemadiol	OS	770	1745	1735	28.351	4.7	1042884038

34	Unknown	Unknown			1743	28.569	0.7	154217100
35	Unknown	Unknown			1753	28.822	0.3	65173690
36	Unknown	Unknown			1760	28.998	0.2	55513379
37	Unknown	Unknown			1789	29.771	0.4	84722012
38	γ-Bicyclohomofarnesal	OD	751	1809	1800	30.061	4.1	905349025
39	α-Kessyl acetate	OS	705	1813	1808	30.258	24.9	5562596550
40	Unknown	Unknown			1826	30.718	0.4	95506412
41	Cyperadione	OS	640	1845	1845	31.201	0.6	140866743
42	Unknown	Unknown			1864	31.679	0.1	21636084
43	Unknown	Unknown			1867	31.762	0.1	29129622
44	Clovanediol	OS	877	1885	1872	31.879	6.3	1418278995
45	Ledene oxide-(I)	OS	696	1890	1889	32.317	8.5	1894764115
46	Unknown	Unknown			1896	32.490	0.1	17157381
47	Alantolactone	OS	660	1916	1903	32.656	1.0	221496236
48	Corymbolone	OS	634	1899	1908	32.770	0.7	161995897
49	Unknown	Unknown			1927	33.239	0.3	57676102
50	Unknown	Unknown			1940	33.544	0.2	40232408
51	Unknown	Unknown			1954	33.889	0.3	68931341
52	Unknown	Unknown			1962	34.092	0.9	192036709
53	Unknown	Unknown			1970	34.287	0.1	23372748
54	Dodecenyl succinic anhydride	Misc.	633	1966	1979	34.490	0.1	25655358
55	(*E*)-15,16-Dinorlabda-8(17),11-dien-13-one	OD	783	1994	1986	34.666	11.6	2597152410
56	Unknown	Unknown			2009	35.217	0.2	47476641
57	Zederone	OS	629	2010	2030	35.695	9.1	2026474563
58	Unknown	Unknown			2046	36.071	0.2	53973322
59	Epimanool	OD	706	2056	2050	36.160	0.2	41485180
60	Unknown	Unknown			2077	36.771	0.1	32152902
61	Unknown	Unknown			2088	37.020	0.2	46782504
62	Unknown	Unknown			2142	38.241	0.1	25987144
63	Unknown	Unknown			2152	38.478	0.2	39575881
64	Unknown	Unknown			2224	40.528	0.3	58181170
65	Unknown	Unknown			2249	41.603	0.2	49274390
66	Unknown	Unknown			2277	42.772	0.6	125934245

[a]MH = monoterpene hydrocarbon, OM = oxygenated monoterpene, SH = sesquiterpene hydrocarbon, OS = oxygenated sesquiterpene, DH = diterpene hydrocarbon, OD = oxygenated diterpene, TH = triterpene hydrocarbon, Misc = Miscellaneous, Unknown = match score < 600 or DRI > 20.

Table B.27: Volatile chemical composition in fraction **FG.I**

Peak	Name	Class[a]	Match	NIST RI	Exp. RI	RT	%Area	Area (count·s)
1	Unknown	Unknown			1074	8.416	0.1	4388091
2	Camphol	OM	606	1167	1167	11.260	0.1	9150590
3	Unknown	Unknown			1201	12.318	0.2	12541868
4	Unknown	Unknown			1335	16.636	0.1	9009021
5	Unknown	Unknown			1355	17.264	0.2	16104537
6	Unknown	Unknown			1370	17.730	0.5	36130885
7	Unknown	Unknown			1377	17.964	0.5	35115510
8	Unknown	Unknown			1390	18.377	21.8	1640929994
9	4,8-β-Epoxy-caryophyllane	OS	617	1421	1401	18.707	1.6	121674854
10	Unknown	Unknown			1421	19.323	0.4	27135403
11	Unknown	Unknown			1438	19.835	0.1	11241470
12	Unknown	Unknown			1448	20.155	0.1	8086777
13	Unknown	Unknown			1452	20.263	0.1	8844085
14	Unknown	Unknown			1462	20.584	0.5	34588951
15	Unknown	Unknown			1478	21.071	0.5	39395577
16	Unknown	Unknown			1526	22.488	1.3	95613029
17	Unknown	Unknown			1600	24.652	0.9	66626637
18	Longifolenaldehyde	OS	642	1631	1630	25.490	3.1	232912782
19	Unknown	Unknown			1637	25.691	1.0	77915060
20	β-Eudesmol	OS	694	1649	1651	26.079	0.4	32116697
21	(-)-Isolongifolol, methyl ether	OS	677	1672	1673	26.662	1.7	124677956
22	Unknown	Unknown			1684	26.979	0.5	38368797
23	Unknown	Unknown			1710	27.694	0.5	35293563
24	Isolongifolol	OS	654	1738	1735	28.347	1.2	93994528
25	Unknown	Unknown			1744	28.588	0.3	22249431
26	Unknown	Unknown			1752	28.788	0.2	17890743
27	Unknown	Unknown			1775	29.398	0.2	17624877
28	γ-Bicyclohomofarnesal	OD	740	1809	1800	30.061	9.1	688109548
29	Unknown	Unknown			1807	30.234	8.8	658306714
30	Cryptomeridiol	OS	752	1826	1815	30.456	15.1	1132949527
31	Unknown	Unknown			1826	30.718	0.3	21639727
32	Clovanediol	OS	846	1885	1872	31.873	4.9	369606611
33	Unknown	Unknown			1882	32.135	1.6	116871795
34	Ledene oxide-(I)	OS	646	1890	1889	32.311	1.5	114989599
35	Unknown	Unknown			1903	32.650	0.3	25346865

36	Alantolactone	OS	644	1916	1912	32.875	0.3	21822834
37	Unknown	Unknown			1928	33.260	0.3	22671550
38	Unknown	Unknown			1940	33.559	0.4	27995557
39	Unknown	Unknown			1948	33.744	0.5	38048368
40	Unknown	Unknown			1972	34.317	0.4	27403457
41	(*E*)-15,16-Dinorlabda-8(17),11-dien-13-one	OD	781	1994	1986	34.660	14.3	1077419898
42	Unknown	Unknown			2012	35.270	0.4	29733226
43	Unknown	Unknown			2020	35.452	0.9	67222553
44	Unknown	Unknown			2030	35.698	0.4	27433498
45	Unknown	Unknown			2034	35.788	0.1	11213428
46	Epimanool	OD	623	2056	2037	35.862	0.2	16759966
47	Unknown	Unknown			2067	36.549	0.3	23848975
48	Unknown	Unknown			2077	36.783	0.4	27989369
49	Unknown	Unknown			2096	37.208	0.3	20234495
50	Unknown	Unknown			2150	38.432	0.2	17150460
51	Larixol	OD	664	2264	2278	42.821	0.9	66098549

[a]MH = monoterpene hydrocarbon, OM = oxygenated monoterpene, SH = sesquiterpene hydrocarbon, OS = oxygenated sesquiterpene, DH = diterpene hydrocarbon, OD = oxygenated diterpene, TH = triterpene hydrocarbon, Misc = Miscellaneous, Unknown = match score < 600 or DRI > 20.

Table B.28: Volatile chemical composition in fraction FG.J

Peak	Name	Class[a]	Match	NIST RI	Exp. RI	RT	%Area	Area (count·s)
1	Unknown	Unknown			1027	7.121	0.5	11258942
2	Unknown	Unknown			1044	7.577	0.5	11259422
3	Nonanal	Misc.	766	1104	1106	9.337	2.5	59616762
4	Unknown	Unknown			1132	10.163	0.9	22138763
5	Camphol	OM	636	1167	1167	11.267	0.4	8880015
6	Unknown	Unknown			1281	14.897	0.3	6316655
7	Unknown	Unknown			1355	17.270	0.8	19995904
8	Unknown	Unknown			1391	18.389	3.2	75271085
9	Patchoulene	SH	642	1467	1452	20.260	0.5	10837947
10	Caparratriene	SH	636	1493	1491	21.468	1.1	26674905
11	Unknown	Unknown			1526	22.507	2.2	52420749
12	Unknown	Unknown			1600	24.664	1.4	33247509
13	Unknown	Unknown			1622	25.265	1.1	26232117
14	Longifolenaldehyde	OS	641	1631	1630	25.487	1.6	38194820
15	Unknown	Unknown			1638	25.706	3.2	76602442
16	Neointermedeol	OS	611	1660	1673	26.668	4.5	105881586
17	Unknown	Unknown			1679	26.840	0.6	14397908
18	Unknown	Unknown			1684	26.982	0.9	21345629
19	Unknown	Unknown			1692	27.207	0.6	14326576
20	Unknown	Unknown			1710	27.700	0.5	11638796
21	Isolongifolol	OS	670	1738	1735	28.347	1.7	40529347
22	Unknown	Unknown			1744	28.591	0.8	19079406
23	Unknown	Unknown			1754	28.853	0.3	7799417
24	γ-Bicyclohomofarnesal	OD	877	1809	1800	30.061	33.6	792351593
25	Unknown	Unknown			1807	30.243	0.3	7621681
26	Cryptomeridiol	OS	860	1826	1813	30.394	3.3	78216708
27	Unknown	Unknown			1861	31.614	0.4	9407340
28	8,14-Cedrandiol	OS	627	1876	1874	31.929	2.9	67752531
29	Unknown	Unknown			1885	32.200	0.8	19603418
30	Unknown	Unknown			1903	32.656	1.9	45963162
31	Unknown	Unknown			1914	32.937	0.5	11327878
32	7,9-Di-*tert*-butyl-1-oxaspiro(4,5)deca-6,9-diene-2,8-dione	OD	686	1923	1919	33.041	0.6	14953525
33	Unknown	Unknown			1924	33.174	0.5	10852800
34	Unknown	Unknown			1928	33.273	0.4	10467625

35	Unknown	Unknown			1948	33.738	0.8	19967957
36	Unknown	Unknown			1960	34.025	0.5	12644344
37	Unknown	Unknown			1966	34.188	1.1	26190002
38	(*E*)-15,16-Dinorlabda-8(17),11-dien-13-one	OD	746	1994	1986	34.672	12.8	302979588
39	Unknown	Unknown			2020	35.458	3.3	78222421
40	Unknown	Unknown			2077	36.786	0.6	13562310
41	Coronarin E	OD	623	2136	2124	37.840	0.3	7693736
42	Unknown	Unknown			2136	38.118	1.4	32772741
43	Unknown	Unknown			2154	38.515	0.8	19879328
44	Unknown	Unknown			2215	40.167	0.6	14980396
45	Larixol	OD	661	2264	2278	42.827	2.0	46628652

[a]MH = monoterpene hydrocarbon, OM = oxygenated monoterpene, SH = sesquiterpene hydrocarbon, OS = oxygenated sesquiterpene, DH = diterpene hydrocarbon, OD = oxygenated diterpene, TH = triterpene hydrocarbon, Misc = Miscellaneous, Unknown = match score < 600 or DRI > 20.

Table B.29: Volatile chemical composition in fraction FG.K

Peak	Name	Class[a]	Match	NIST RI	Exp. RI	RT	%Area	Area (count·s)
1	*p*-Cymene	MH	777	1025	1027	7.115	0.3	19838585
2	Unknown	Unknown			1099	9.137	0.2	15812406
3	Unknown	Unknown			1122	9.830	0.1	7591791
4	Unknown	Unknown			1128	10.021	0.2	10519008
5	Unknown	Unknown			1145	10.552	0.2	11241603
6	Camphol	OM	716	1167	1167	11.257	0.3	22451378
7	Carvomenthenal	OM	601	1229	1226	13.131	0.9	59986875
8	Neral	OM	607	1240	1259	14.195	0.7	48833366
9	*trans*-Ascaridol glycol	OM	812	1272	1287	15.095	1.4	95456769
10	4-Methylphenyl isothiocyanate	Misc.	673	1293	1293	15.286	2.1	145139384
11	Unknown	Unknown			1300	15.514	0.3	18591680
12	Unknown	Unknown			1310	15.828	0.6	40572402
13	Unknown	Unknown			1373	17.828	1.5	99471118
14	Sobrerol	OM	817	1388	1378	17.979	12.8	862647997
15	Unknown	Unknown			1390	18.368	11.4	767717061
16	Unknown	Unknown			1421	19.326	0.6	41486175
17	α-Himachalene	SH	620	1449	1452	20.272	0.5	31684099
18	Unknown	Unknown			1470	20.821	0.5	33336157
19	Unknown	Unknown			1491	21.471	0.3	21678030
20	*cis*-Sesquisabinene hydrate	OS	602	1543	1526	22.491	0.5	32849340
21	Unknown	Unknown			1630	25.490	0.7	44454736
22	Unknown	Unknown			1637	25.688	0.8	52317370
23	(-)-Isolongifolol, methyl ether	OS	662	1672	1672	26.658	2.2	150188093
24	Unknown	Unknown			1684	26.976	0.5	34510167
25	8α,11-Elemadiol	OS	754	1745	1735	28.347	2.6	176048620
26	α-Vetivol	OS	619	1756	1744	28.579	0.5	35028004
27	Unknown	Unknown			1752	28.807	0.2	15560758
28	Dehydrovomifoliol	OS	641	1800	1788	29.750	9.2	620490187
29	γ-Bicyclohomofarnesal	OD	743	1809	1800	30.058	23.5	1585462661
30	Unknown	Unknown			1806	30.221	0.3	21748833
31	Cryptomeridiol	OS	894	1826	1813	30.385	4.8	326875349
32	Unknown	Unknown			1850	31.334	1.0	66244690
33	Unknown	Unknown			1902	32.647	0.9	59180182
34	Unknown	Unknown			1928	33.254	0.3	21210403
35	Cembrene	DH	675	1939	1951	33.827	3.2	217648450

36	(*E*)-15,16-Dinorlabda-8(17),11-dien-13-one	OD	798	1994	1986	34.660	12.1	820064680
37	Unknown	Unknown			2011	35.248	0.4	23661736
38	Unknown	Unknown			2037	35.858	0.5	30591742
39	Unknown	Unknown			2047	36.080	0.5	35131842
40	Larixol	OD	666	2264	2278	42.833	0.5	32538355

[a]MH = monoterpene hydrocarbon, OM = oxygenated monoterpene, SH = sesquiterpene hydrocarbon, OS = oxygenated sesquiterpene, DH = diterpene hydrocarbon, OD = oxygenated diterpene, TH = triterpene hydrocarbon, Misc = Miscellaneous, Unknown = match score < 600 or DRI > 20.

Table B.30: Volatile chemical composition in fraction FG.L

Peak	Name	Class[a]	Match	NIST RI	Exp. RI	RT	%Area	Area (count·s)
1	Unknown	Unknown			1003	6.428	0.3	12210323
2	*p*-Cymene	MH	780	1025	1027	7.118	0.8	30973718
3	Unknown	Unknown			1101	9.189	0.2	9633450
4	Unknown	Unknown			1120	9.772	0.5	21290460
5	Unknown	Unknown			1128	10.024	0.2	8694937
6	Unknown	Unknown			1144	10.536	3.7	143016957
7	Camphol	OM	705	1167	1167	11.257	0.6	22642337
8	Unknown	Unknown			1259	14.207	0.3	10990816
9	4-Methylphenyl isothiocyanate	Misc.	700	1293	1293	15.283	5.0	193497454
10	Unknown	Unknown			1355	17.252	0.3	12924609
11	Unknown	Unknown			1374	17.847	0.3	12754321
12	Unknown	Unknown			1390	18.380	2.6	102598556
13	β-Copaene	SH	639	1432	1452	20.272	0.7	27544318
14	Unknown	Unknown			1491	21.465	0.4	17392632
15	Unknown	Unknown			1526	22.504	0.6	23428084
16	Unknown	Unknown			1630	25.493	0.6	22455298
17	Unknown	Unknown			1637	25.688	1.2	46750740
18	(*Z*)-6,7-Dihydrofarnesol	OS	623	1668	1673	26.668	3.2	126409504
19	Unknown	Unknown			1684	26.979	0.7	28815622
20	Unknown	Unknown			1744	28.585	1.1	41042730
21	γ-Bicyclohomofarnesal	OD	736	1809	1800	30.064	30.4	1185730131
22	Cryptomeridiol	OS	701	1826	1813	30.397	0.5	19747332
23	Unknown	Unknown			1877	32.015	0.5	19445440
24	Unknown	Unknown			1903	32.656	0.9	33200959
25	Sandaracopimaradiene	DH	625	1960	1953	33.864	2.2	84282782
26	(*E*)-15,16-Dinorlabda-8(17),11-dien-13-one	OD	808	1994	1986	34.660	39.6	1541747017
27	Isopropyl palmitate	Misc.	649	2023	2027	35.627	0.6	23140417
28	Epimanool	OD	643	2056	2037	35.862	0.7	27381526
29	Coronarin E	OD	690	2136	2124	37.843	0.4	17108378
30	Larixol	OD	647	2264	2279	42.849	0.7	28728134

[a]MH = monoterpene hydrocarbon, OM = oxygenated monoterpene, SH = sesquiterpene hydrocarbon, OS = oxygenated sesquiterpene, DH = diterpene hydrocarbon, OD = oxygenated diterpene, TH = triterpene hydrocarbon, Misc = Miscellaneous, Unknown = match score < 600 or DRI > 20.

Table B.31: Volatile chemical composition in fraction **FG.M**

Peak	Name	Class[a]	Match	NIST RI	Exp. RI	RT	%Area	Area (count·s)
1	*p*-Cymene	MH	663	1025	1027	7.112	0.2	9460155
2	Unknown	Unknown			1101	9.189	0.3	11225813
3	Unknown	Unknown			1127	10.012	0.1	5352212
4	Unknown	Unknown			1144	10.542	0.4	16427718
5	endo-Borneol	OM	709	1167	1167	11.248	0.3	11148509
6	2-Methylbenzothiazole	Misc.	708	1281	1294	15.326	0.5	23225740
7	Unknown	Unknown			1300	15.523	0.2	8381717
8	Unknown	Unknown			1355	17.258	0.4	16471447
9	Unknown	Unknown			1391	18.389	1.4	60123477
10	Unknown	Unknown			1491	21.459	0.4	16281983
11	Unknown	Unknown			1527	22.516	0.6	27795496
12	Longifolenaldehyde	OS	617	1631	1630	25.493	0.9	40348932
13	Unknown	Unknown			1638	25.700	1.4	61529821
14	(-)-Isolongifolol, methyl ether	OS	676	1672	1673	26.668	4.5	198107144
15	Unknown	Unknown			1684	26.985	0.9	39033884
16	γ-Costol	OS	637	1752	1744	28.588	0.8	37051870
17	γ-Bicyclohomofarnesal	OD	852	1809	1800	30.067	20.8	923170789
18	Unknown	Unknown			1878	32.021	1.0	44760765
19	Unknown	Unknown			1903	32.656	0.7	29378382
20	Unknown	Unknown			1928	33.273	0.3	13994174
21	(*E*)-15,16-Dinorlabda-8(17),12-dien-14-al	OD	664	1958	1953	33.867	1.4	62447616
22	(*E*)-15,16-Dinorlabda-8(17),11-dien-13-one	OD	794	1994	1986	34.666	58.2	2579123380
23	Unknown	Unknown			2020	35.461	0.3	14505193
24	Geranyl linallol	OD	615	2034	2037	35.862	0.5	22112095
25	Epimanool	OD	700	2056	2053	36.219	1.4	61519209
26	Coronarin E	OD	654	2136	2124	37.843	0.3	11512589
27	Unknown	Unknown			2142	38.250	0.2	10197052
28	Unknown	Unknown			2152	38.472	0.3	13354987
29	Unknown	Unknown			2244	41.397	0.1	5511111
30	Unknown	Unknown			2249	41.613	0.4	16281949
31	Larixol	OD	663	2264	2279	42.849	0.9	41819616

[a]MH = monoterpene hydrocarbon, OM = oxygenated monoterpene, SH = sesquiterpene hydrocarbon, OS = oxygenated sesquiterpene, DH = diterpene hydrocarbon, OD = oxygenated diterpene, TH = triterpene hydrocarbon, Misc = Miscellaneous, Unknown = match score < 600 or DRI > 20.

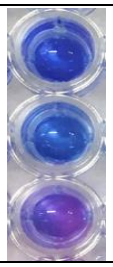
Appendix C

Antibacterial evaluation

C1) Minimum inhibitory concentration (MIC) against *S. aureus* of all fractions

C1.1) MIC of crude DCM and fraction FA-FJ

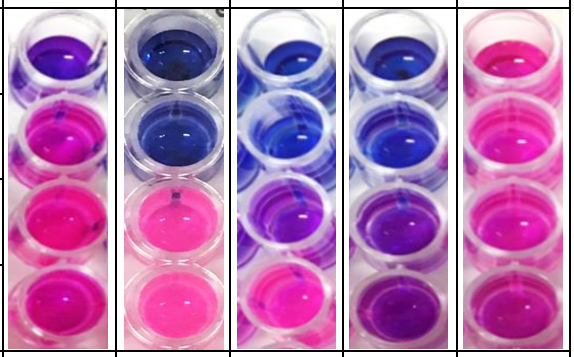
Concentration	Negative control	Positive control
5% DMSO in MHB		
2.5% DMSO in MHB		
1.25% DMSO in MHB		
0.63% DMSO in MHB		

Concentration	Streptomycin
25 μg mL^{-1} in H_2O	
12.5 μg mL^{-1} in H_2O	
6.25 μg mL^{-1} in H_2O	
MIC result	12.5 μg mL^{-1}

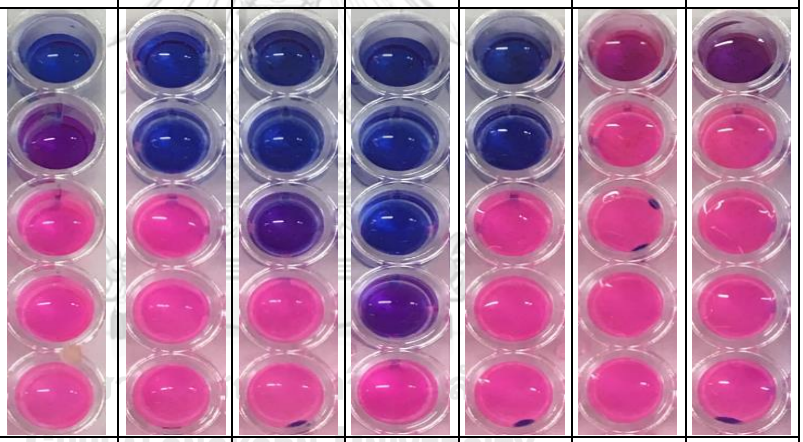
Concentration	Amoxicillin
1.563 μg mL^{-1} 0.312% DMSO in H_2O	
0.781 μg mL^{-1} 0.156% DMSO in H_2O	
0.391 μg mL^{-1} 0.078% DMSO in H_2O	
0.195 μg mL^{-1} 0.039% DMSO in H_2O	
MIC result	<0.195 μg mL^{-1}

Concentration	Ciprofloxacin
1.25 μg mL^{-1} in H_2O	
0.625 μg mL^{-1} in H_2O	
0.312 μg mL^{-1} in H_2O	
0.156 μg mL^{-1} in H_2O	
MIC result	0.625 μg mL^{-1}

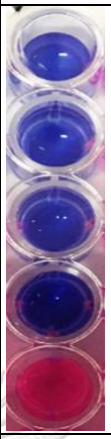
Concentration	Crude DCM	FA	FB	FC	FD	FE
800 μg mL^{-1} 5% DMSO in MHB						
400 μg mL^{-1} 5% DMSO in MHB						
200 μg mL^{-1} 5% DMSO in MHB						
100 μg mL^{-1} 5% DMSO in MHB						
MIC result	800 μg mL^{-1}	>800 μg mL^{-1}	>800 μg mL^{-1}	800 μg mL^{-1}	800 μg mL^{-1}	400 μg mL^{-1}

Concentration	FF	FG	FH	FI	FJ
800 µg mL^{-1} 5% DMSO in MHB					
400 µg mL^{-1} 5% DMSO in MHB					
200 µg mL^{-1} 5% DMSO in MHB					
100 µg mL^{-1} 5% DMSO in MHB					
MIC result	>800 µg mL^{-1}	400 µg mL^{-1}	400 µg mL^{-1}	400 µg mL^{-1}	>800 µg mL^{-1}

C1.2) MIC of sub-fraction FE.A-FE.G

Concentration	FE.A	FE.B	FE.C	FE.D	FE.E	FE.F	FE.G
800 µg mL^{-1} 5% DMSO in MHB							
400 µg mL^{-1} 5% DMSO in MHB							
200 µg mL^{-1} 5% DMSO in MHB							
100 µg mL^{-1} 5% DMSO in MHB							
50 µg mL^{-1} 5% DMSO in MHB							
MIC result	800 µg mL^{-1}	400 µg mL^{-1}	400 µg mL^{-1}	200 µg mL^{-1}	400 µg mL^{-1}	>800 µg mL^{-1}	>800 µg mL^{-1}

C1.3) MIC of sub-fraction FG.A-FG.M

Concentration	FG.A	FG.B	FG.C	FG.D	FG.E	FG.F	FG.G	FG.H
800 µg mL^{-1} 5% DMSO in MHB 400 µg mL^{-1} 5% DMSO in MHB 200 µg mL^{-1} 5% DMSO in MHB 100 µg mL^{-1} 5% DMSO in MHB 50 µg mL^{-1} 5% DMSO in MHB								
MIC result	>800 µg mL^{-1}	>800 µg mL^{-1}	400 µg mL^{-1}	100 µg mL^{-1}	400 µg mL^{-1}	400 µg mL^{-1}	400 µg mL^{-1}	200 µg mL^{-1}

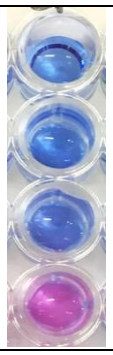
Concentration	FG.I	FG.J	FG.K	FG.L	FG.M	P1	P2
800 µg mL^{-1} 5% DMSO in MHB 400 µg mL^{-1} 5% DMSO in MHB 200 µg mL^{-1} 5% DMSO in MHB 100 µg mL^{-1} 5% DMSO in MHB 50 µg mL^{-1} 5% DMSO in MHB							
MIC result	800 µg mL^{-1}	>800 µg mL^{-1}	400 µg mL^{-1}	400 µg mL^{-1}	200 µg mL^{-1}	>800 µg mL^{-1}	>800 µg mL^{-1}

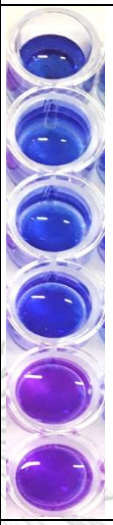
C2.) Minimum inhibitory concentration (MIC) against *M. luteus* of the selected fractions

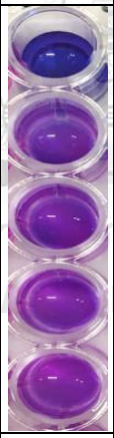
Concentration	Negative control	Positive control
5% DMSO in MHB		
2.5% DMSO in MHB		
1.25% DMSO in MHB		
0.63% DMSO in MHB		

Concentration	Streptomycin
3.13 μg mL^{-1} in H_2O	
1.56 μg mL^{-1} in H_2O	
0.78 μg mL^{-1} in H_2O	
MIC result	1.56 μg mL^{-1}

Concentration	Amoxicillin
0.4 μg mL^{-1} 5% DMSO in MHB	
0.2 μg mL^{-1} 2.5% DMSO in MHB	
0.1 μg mL^{-1} 1.25% DMSO in MHB	
0.05 μg mL^{-1} 0.63% DMSO in MHB	
MIC result	0.2 μg mL^{-1}

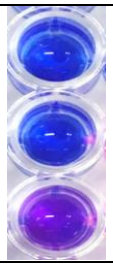
Concentration	Ciprofloxacin
10 μg mL^{-1} in H_2O	
5 μg mL^{-1} in H_2O	
2.5 μg mL^{-1} in H_2O	
1.25 μg mL^{-1} in H_2O	
MIC result	2.5 μg mL^{-1}

Concentration	Crude DCM	FE	FE.A	FE.C	FE.D	FE.E	FG
800 µg mL^{-1} 5% DMSO in MHB 400 µg mL^{-1} 5% DMSO in MHB 200 µg mL^{-1} 5% DMSO in MHB 100 µg mL^{-1} 5% DMSO in MHB 50 µg mL^{-1} 5% DMSO in MHB 25 µg mL^{-1} 5% DMSO in MHB							
MIC result	400 µg mL^{-1}	200 µg mL^{-1}	800 µg mL^{-1}	100 µg mL^{-1}	50 µg mL^{-1}	200 µg mL^{-1}	400 µg mL^{-1}

Concentration	FG.D	FG.G	FG.H	FG.M	P1	P2
800 µg mL^{-1} 5% DMSO in MHB 400 µg mL^{-1} 5% DMSO in MHB 200 µg mL^{-1} 5% DMSO in MHB 100 µg mL^{-1} 5% DMSO in MHB 50 µg mL^{-1} 5% DMSO in MHB						
MIC result	400 µg mL^{-1}	800 µg mL^{-1}	400 µg mL^{-1}	400 µg mL^{-1}	>400 µg mL^{-1}	>400 µg mL^{-1}

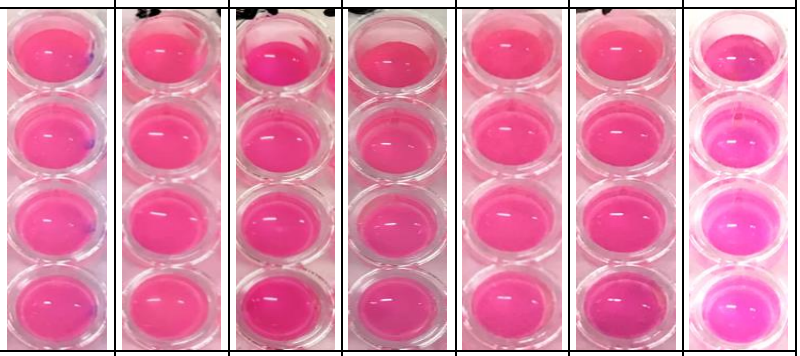
C3.) Minimum inhibitory concentration (MIC) against *E. coli* of the selected fractions

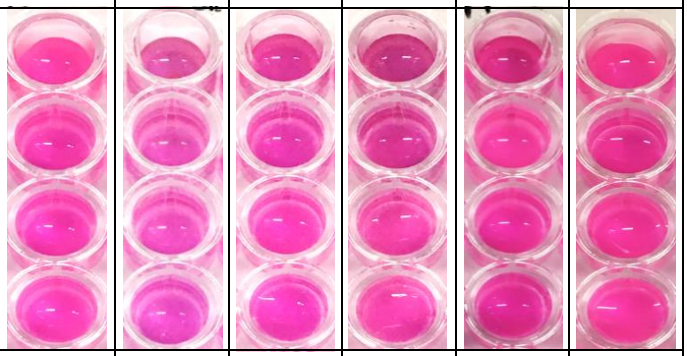
Concentration	Negative control	Positive control
5% DMSO in MHB		
2.5% DMSO in MHB		
1.25% DMSO in MHB		
0.63% DMSO in MHB		

Concentration	Streptomycin
25 μg mL^{-1} in H_2O	
12.5 μg mL^{-1} in H_2O	
6.25 μg mL^{-1} in H_2O	
MIC result	12.5 μg mL^{-1}

Concentration	Amoxicillin
50 μg mL^{-1} 5% DMSO in MHB	
25 μg mL^{-1} 2.5% DMSO in MHB	
12.5 μg mL^{-1} 1.25% DMSO in MHB	
6.25 μg mL^{-1} 0.63% DMSO in MHB	
MIC result	25 μg mL^{-1}

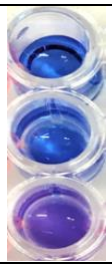
Concentration	Ciprofloxacin
0.313 μg mL^{-1} in H_2O	
0.156 μg mL^{-1} in H_2O	
0.67 μg mL^{-1} in H_2O	
0.04 μg mL^{-1} in H_2O	
MIC result	<0.04 μg mL^{-1}

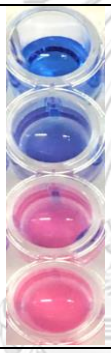
Concentration	Crude DCM	FE	FE.A	FE.C	FE.D	FE.E	FG
800 μg mL^{-1} 5% DMSO in MHB							
400 μg mL^{-1} 5% DMSO in MHB							
200 μg mL^{-1} 5% DMSO in MHB							
100 μg mL^{-1} 5% DMSO in MHB							
MIC result	>800 μg mL^{-1}	>800 μg mL^{-1}	>800 μg mL^{-1}	>800 μg mL^{-1}	>800 μg mL^{-1}	>800 μg mL^{-1}	>800 μg mL^{-1}

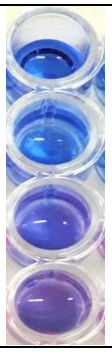
Concentration	FG.D	FG.G	FG.H	FG.M	P1	P2
800 µg mL^{-1} 5% DMSO in MHB						
400 µg mL^{-1} 5% DMSO in MHB						
200 µg mL^{-1} 5% DMSO in MHB						
100 µg mL^{-1} 5% DMSO in MHB						
MIC result	>800 µg mL^{-1}	>800 µg mL^{-1}	>800 µg mL^{-1}	>800 µg mL^{-1}	>800 µg mL^{-1}	>800 µg mL^{-1}

C4.) Minimum inhibitory concentration (MIC) against *P. aeru*ginosa of the selected fractions

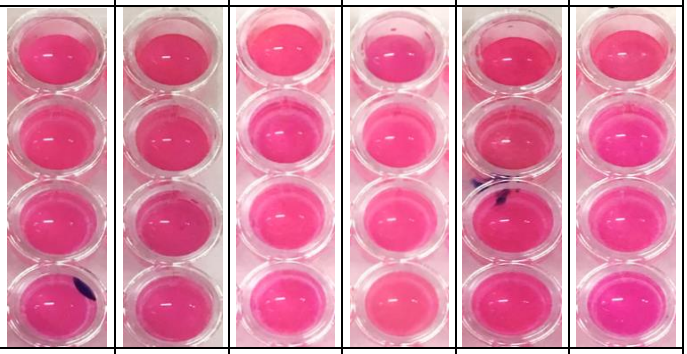
Concentration	Negative control	Positive control
5% DMSO in MHB		
2.5% DMSO in MHB		
1.25% DMSO in MHB		
0.63% DMSO in MHB		

Concentration	Streptomycin
100 µg mL^{-1} in H_2O	
50 µg mL^{-1} in H_2O	
25 µg mL^{-1} in H_2O	
MIC result	50 µg mL^{-1}

Concentration	Amoxicillin
200 µg mL^{-1} 5% DMSO in MHB	
100 µg mL^{-1} 2.5% DMSO in MHB	
50 µg mL^{-1} 1.25% DMSO in MHB	
25 µg mL^{-1} 0.63% DMSO in MHB	
MIC result	200 µg mL^{-1}

Concentration	Ciprofloxacin
5 µg mL^{-1} in H_2O	
2.5 µg mL^{-1} in H_2O	
1.25 µg mL^{-1} in H_2O	
0.63 µg mL^{-1} in H_2O	
MIC result	**2.5** µg mL^{-1}

Concentration	Crude DCM	FE	FE.A	FE.C	FE.D	FE.E	FG
800 µg mL^{-1} 5% DMSO in MHB							
400 µg mL^{-1} 5% DMSO in MHB							
200 µg mL^{-1} 5% DMSO in MHB							
100 µg mL^{-1} 5% DMSO in MHB							
MIC result	>800 µg mL^{-1}	>800 µg mL^{-1}	>800 µg mL^{-1}	>800 µg mL^{-1}	>800 µg mL^{-1}	>800 µg mL^{-1}	>800 µg mL^{-1}

Concentration	FG.D	FG.G	FG.H	FG.M	P1	P2
800 µg mL^{-1} 5% DMSO in MHB						
400 µg mL^{-1} 5% DMSO in MHB						
200 µg mL^{-1} 5% DMSO in MHB						
100 µg mL^{-1} 5% DMSO in MHB						
MIC result	>800 µg mL^{-1}	>800 µg mL^{-1}	>800 µg mL^{-1}	>800 µg mL^{-1}	>800 µg mL^{-1}	>800 µg mL^{-1}

Appendix D

NMR spectra

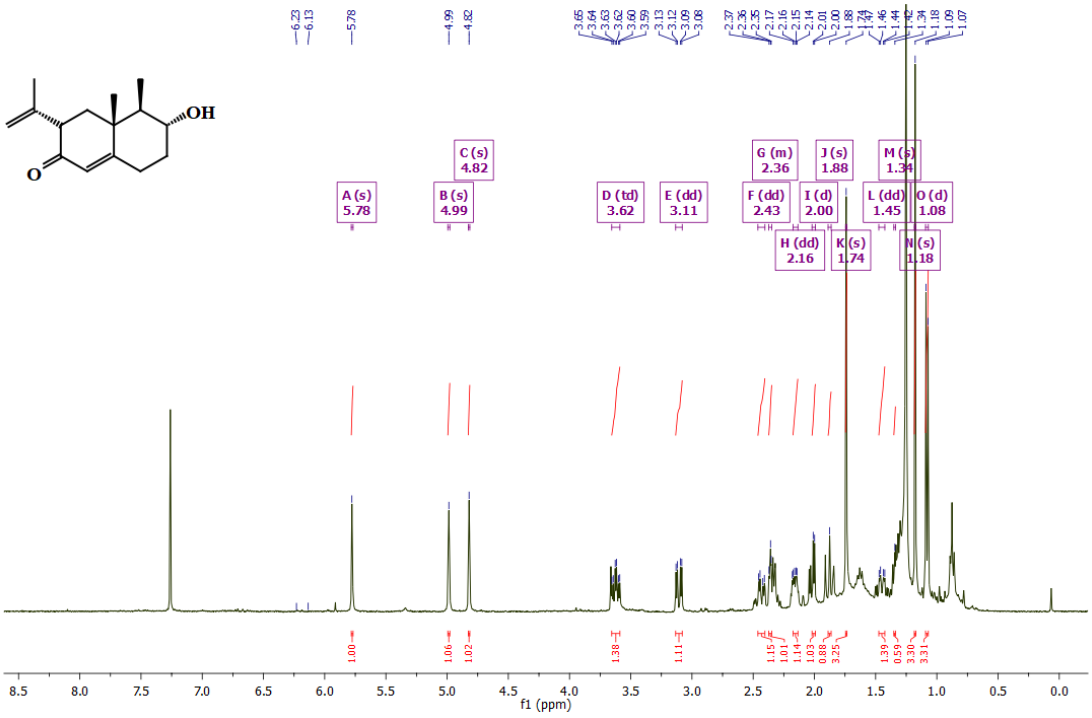


Figure D.1: ^{1}H NMR spectrum of petasol (**P1**) ($CDCl_3$, 400 MHz)

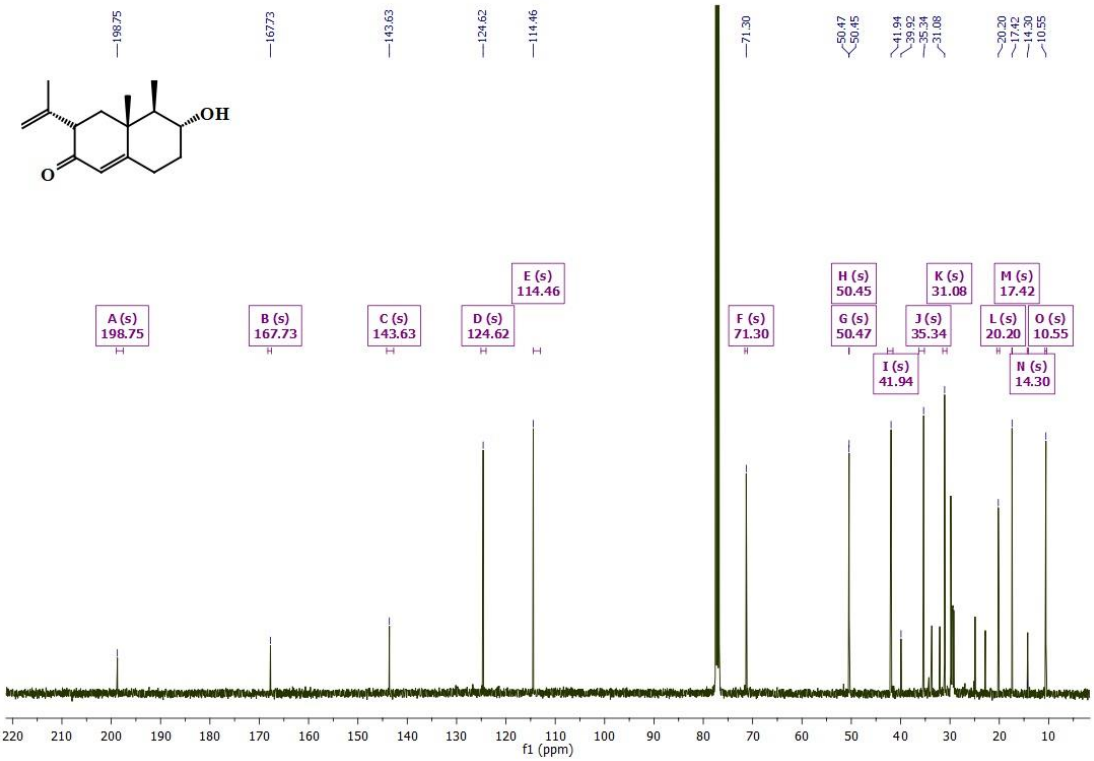


Figure D.2: ^{13}C NMR spectrum of petasol (**P1**) ($CDCl_3$, 100 MHz)

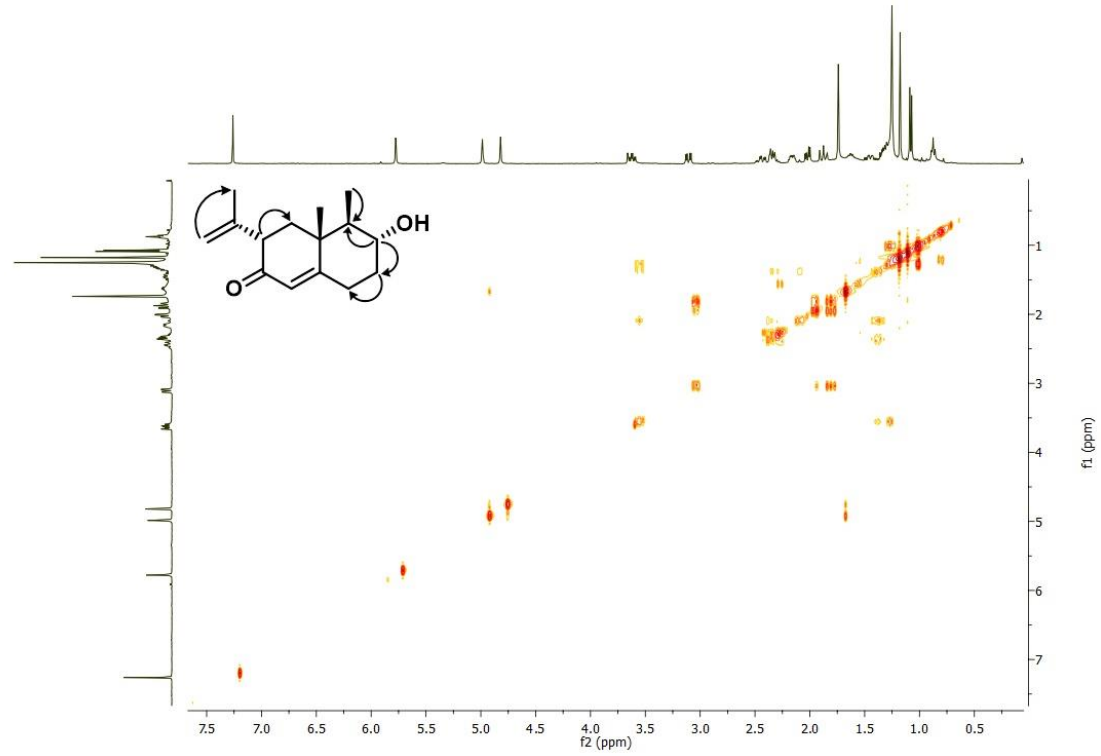


Figure D.3: COSY NMR spectrum of petasol (**P1**) in $CDCl_3$

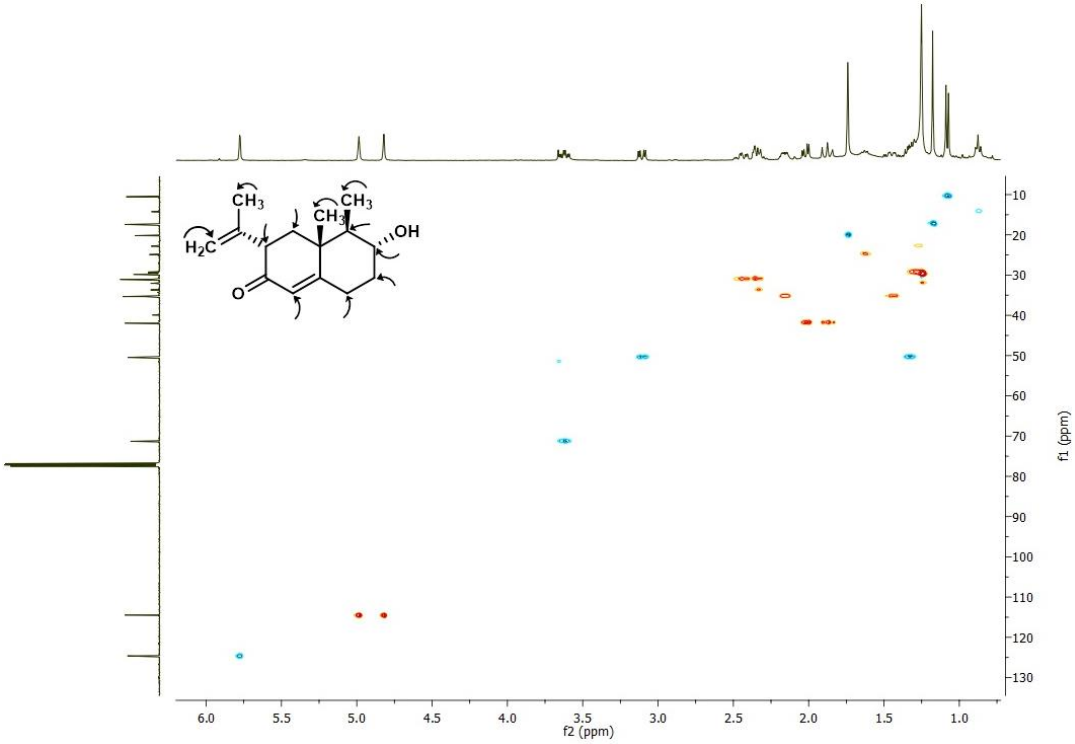


Figure D.4: HSQC NMR spectrum of petasol (**P1**) in $CDCl_3$

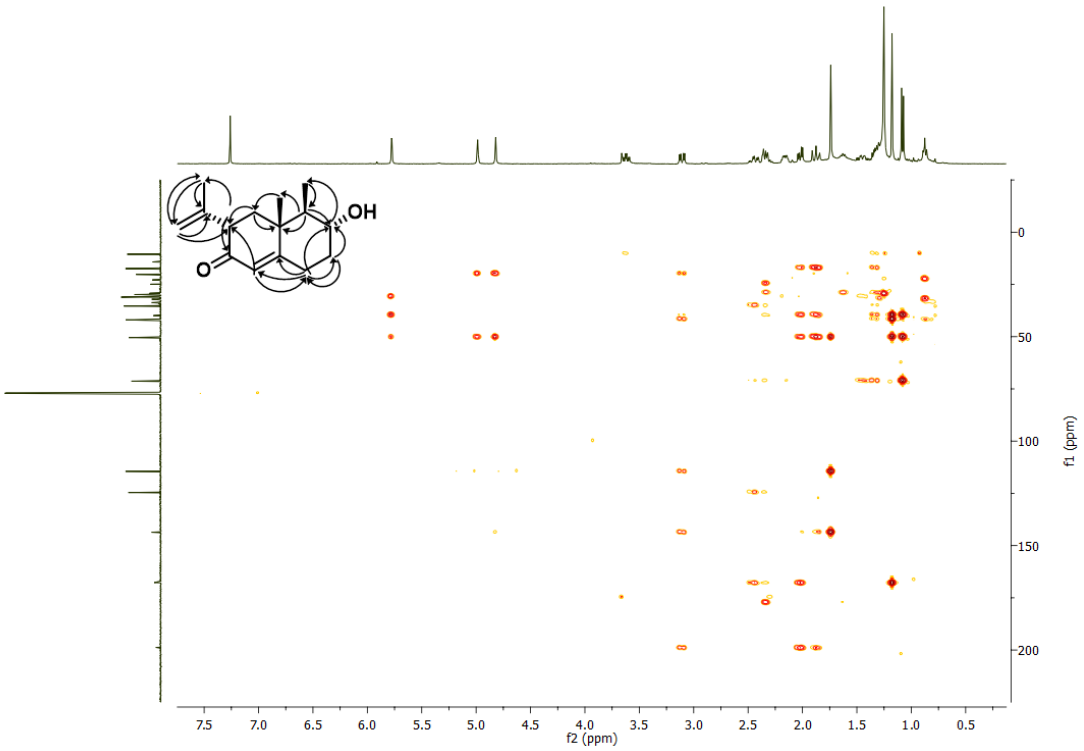


Figure D.5: HMBC NMR spectrum of petasol (**P1**) in $CDCl_3$

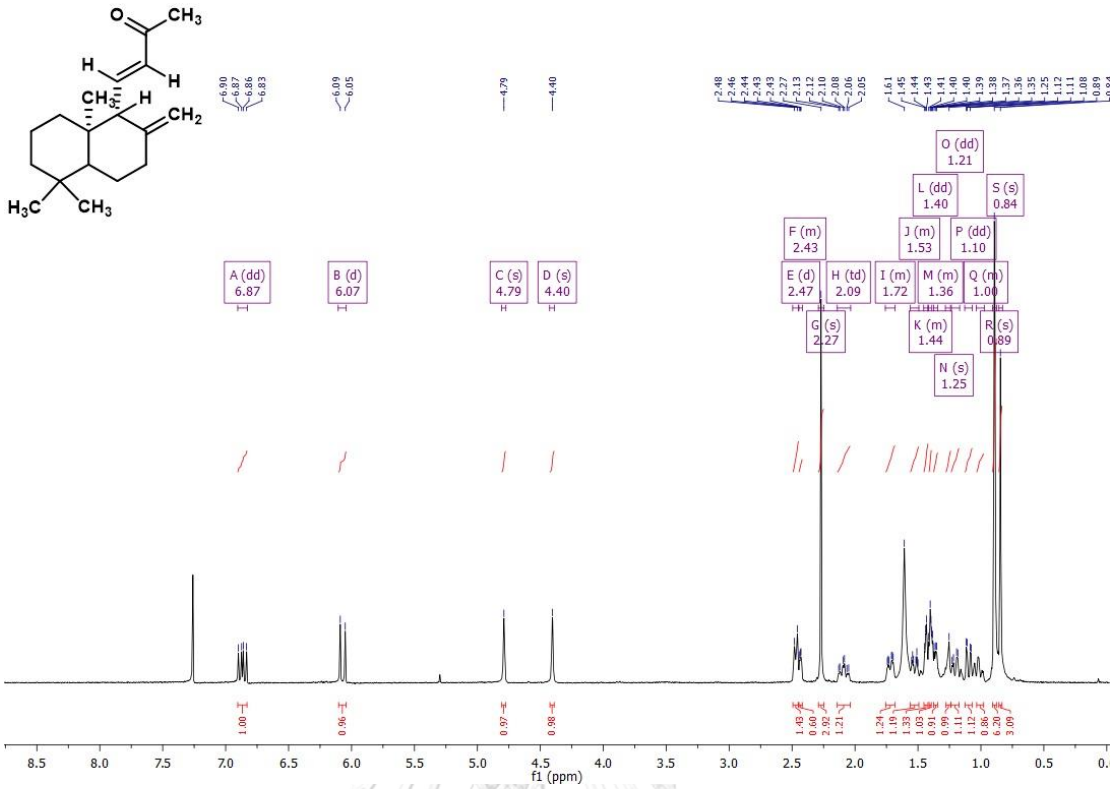


Figure D.6: ^{1}H NMR spectrum of (*E*)-15,16-dinorlabda-8(17),11-dien-13-one (**P2**) ($CDCl_3$, 400 MHz)

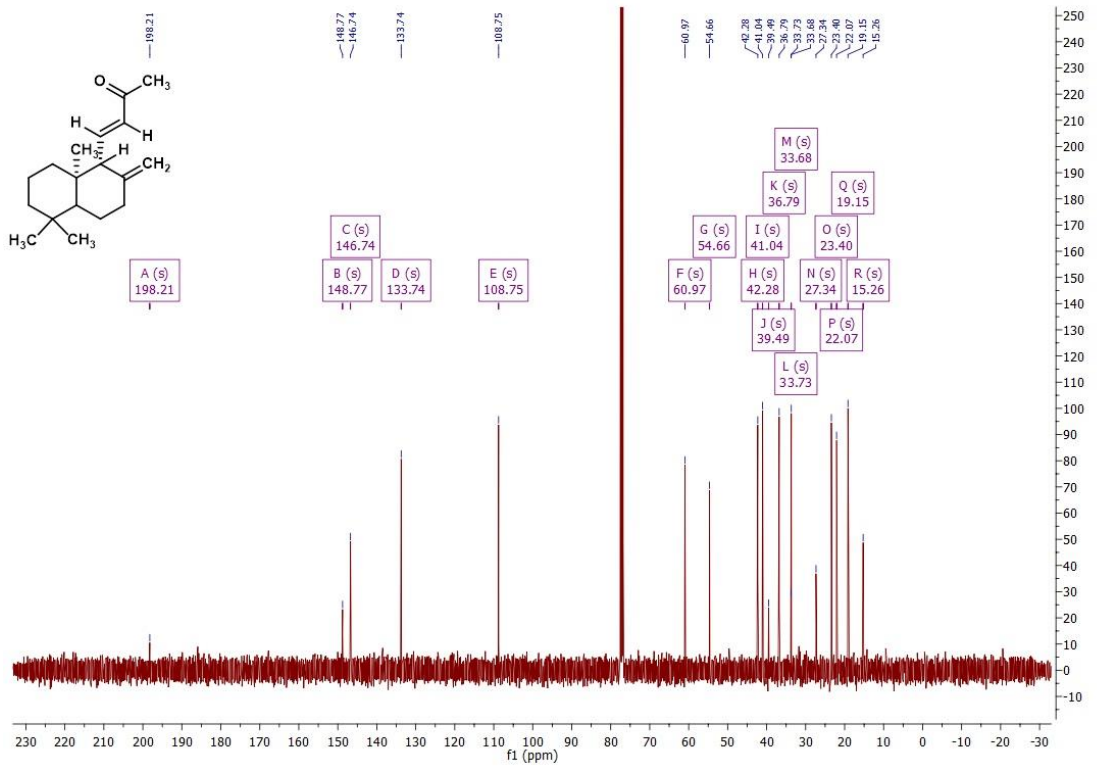


Figure D.7: ^{13}C NMR spectrum of (*E*)-15,16-dinorlabda-8(17),11-dien-13-one (**P2**) ($CDCl_3$, 100 MHz)

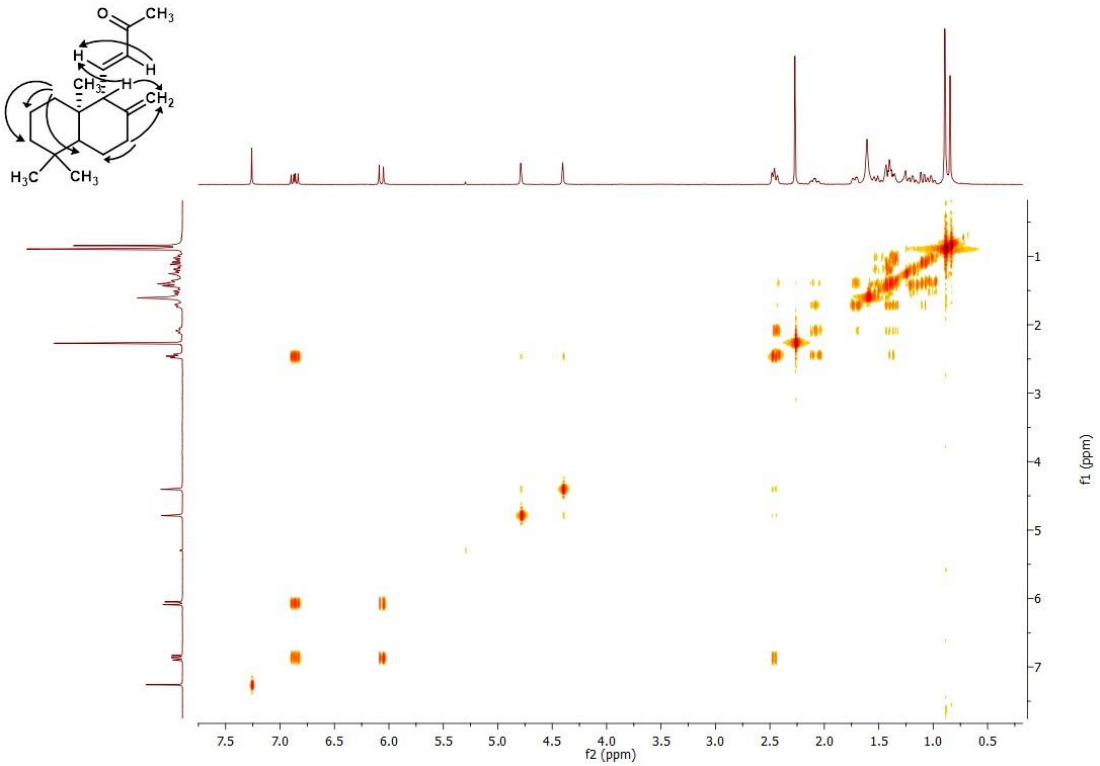


Figure D.8: COSY NMR spectrum of (*E*)-15,16-dinorlabda-8(17),11-dien-13-one (**P2**) in $CDCl_3$

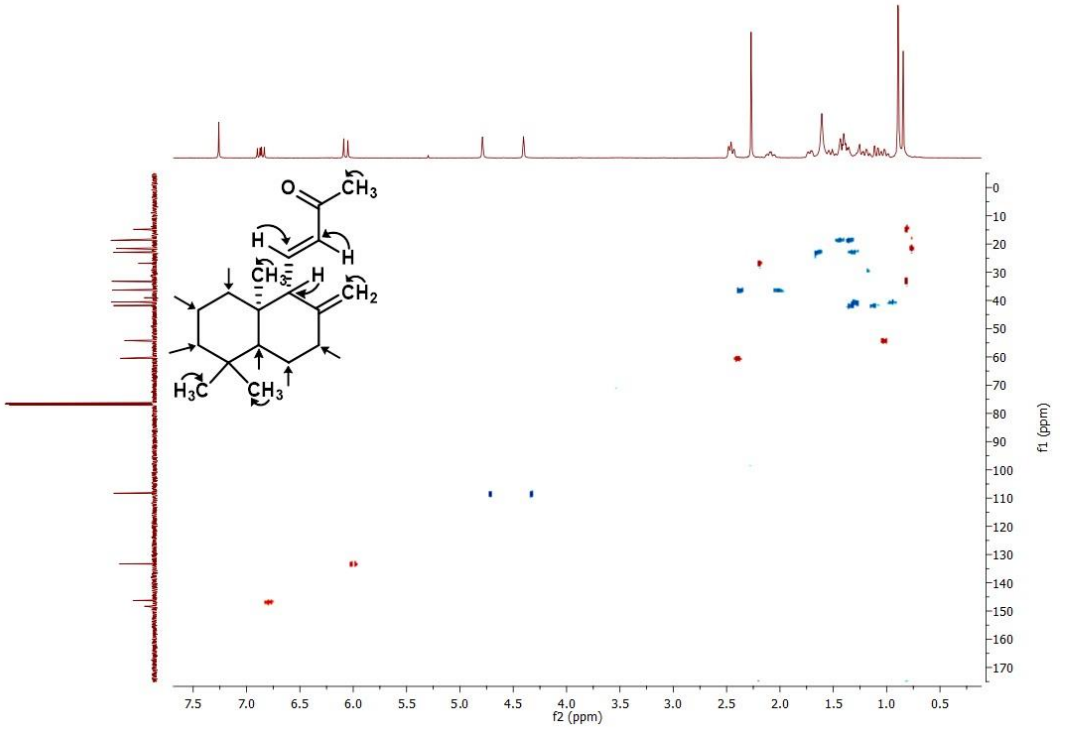


Figure D.9: HSQC NMR spectrum of (*E*)-15,16-dinorlabda-8(17),11-dien-13-one (**P2**) in $CDCl_3$

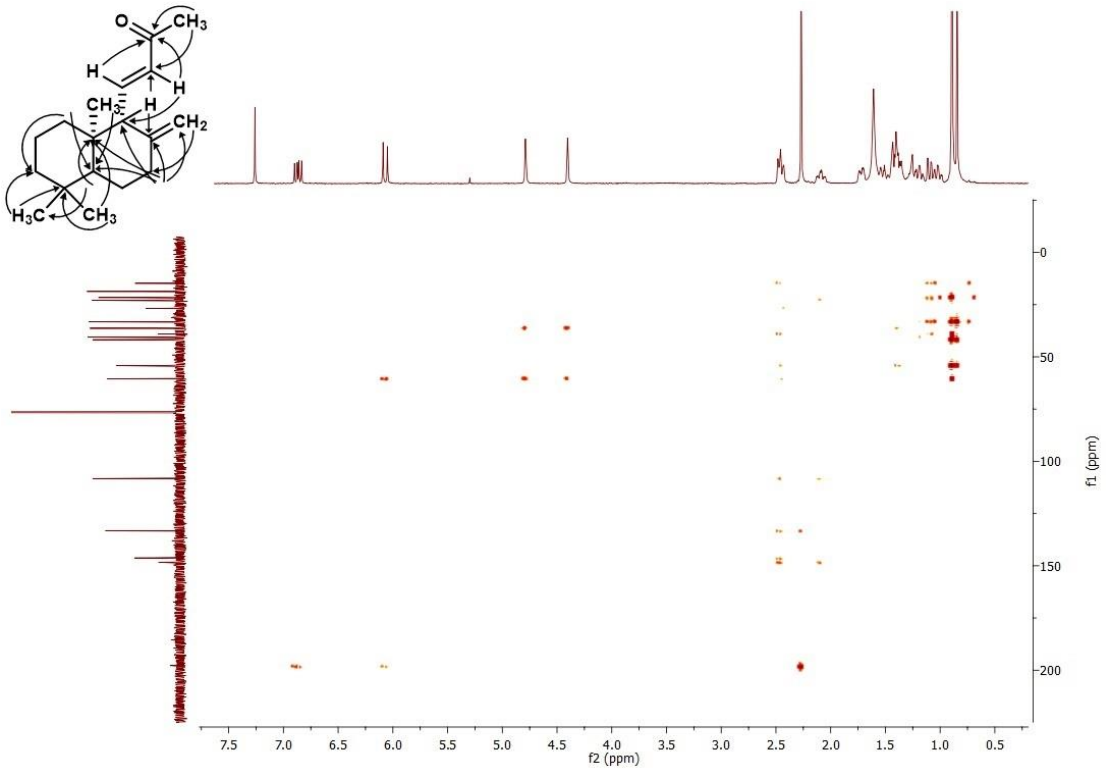


Figure D.10: HMBC NMR spectrum of (*E*)-15,16-dinorlabda-8(17),11-dien-13-one (**P2**) in $CDCl_3$

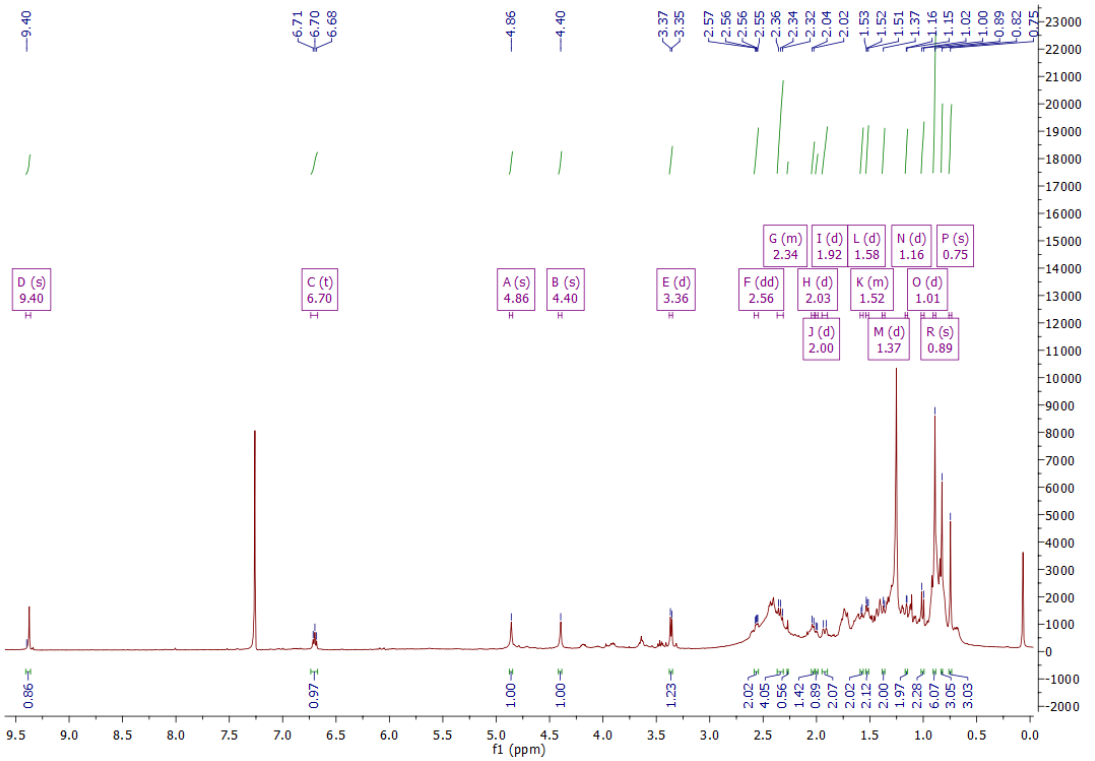


Figure D.11: ^{1}H NMR spectrum of **P3** ($CDCl_3$, 400 MHz)

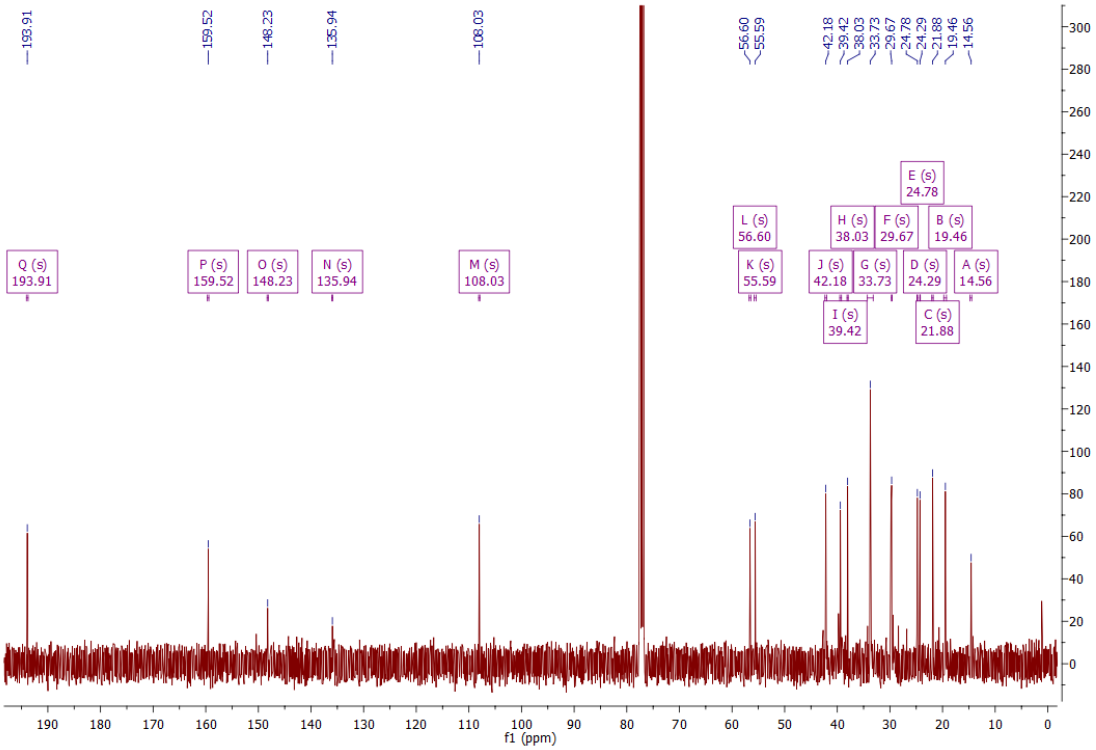


Figure D.12: ^{13}C NMR spectrum of **P3** ($CDCl_3$, 100 MHz)

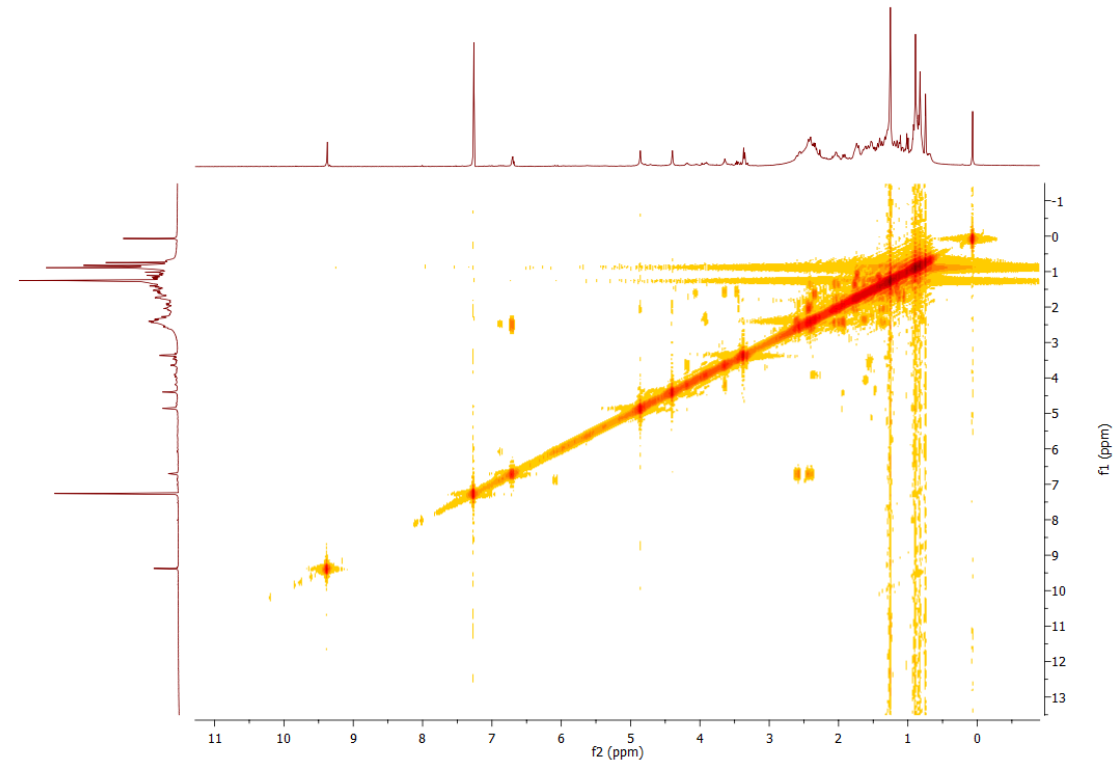


Figure D.13: COSY NMR spectrum of **P3** in $CDCl_3$

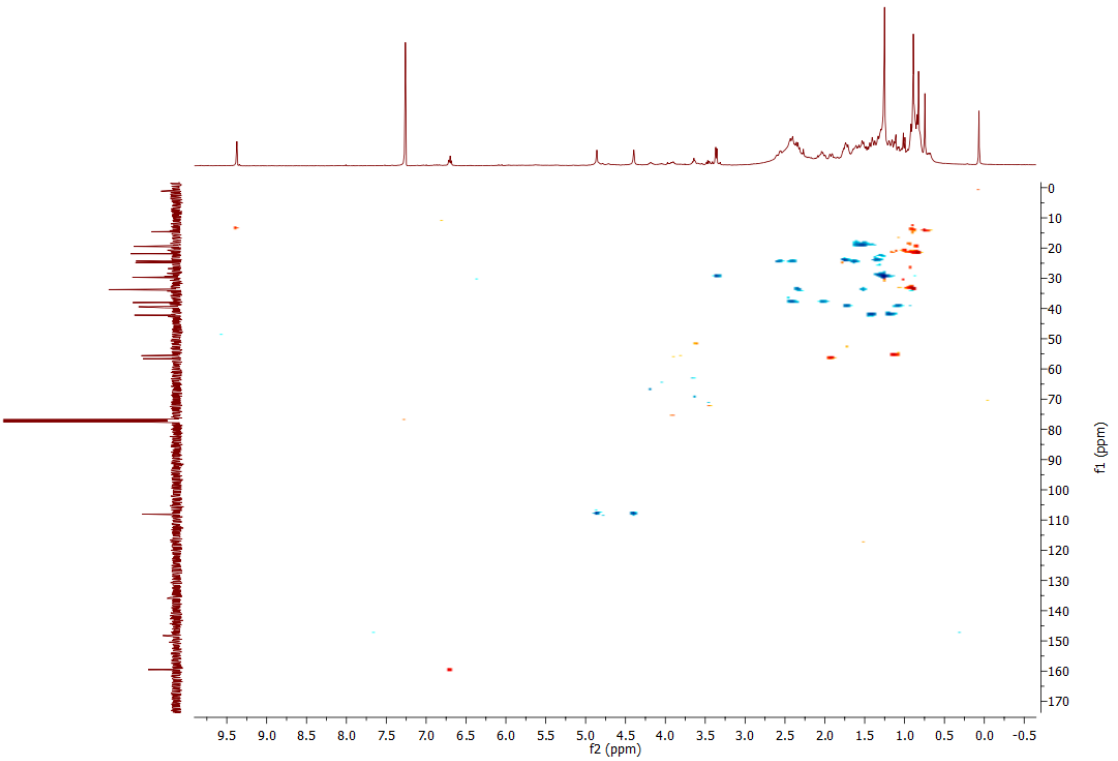


Figure D.14: HSQC NMR spectrum of **P3** in $CDCl_3$

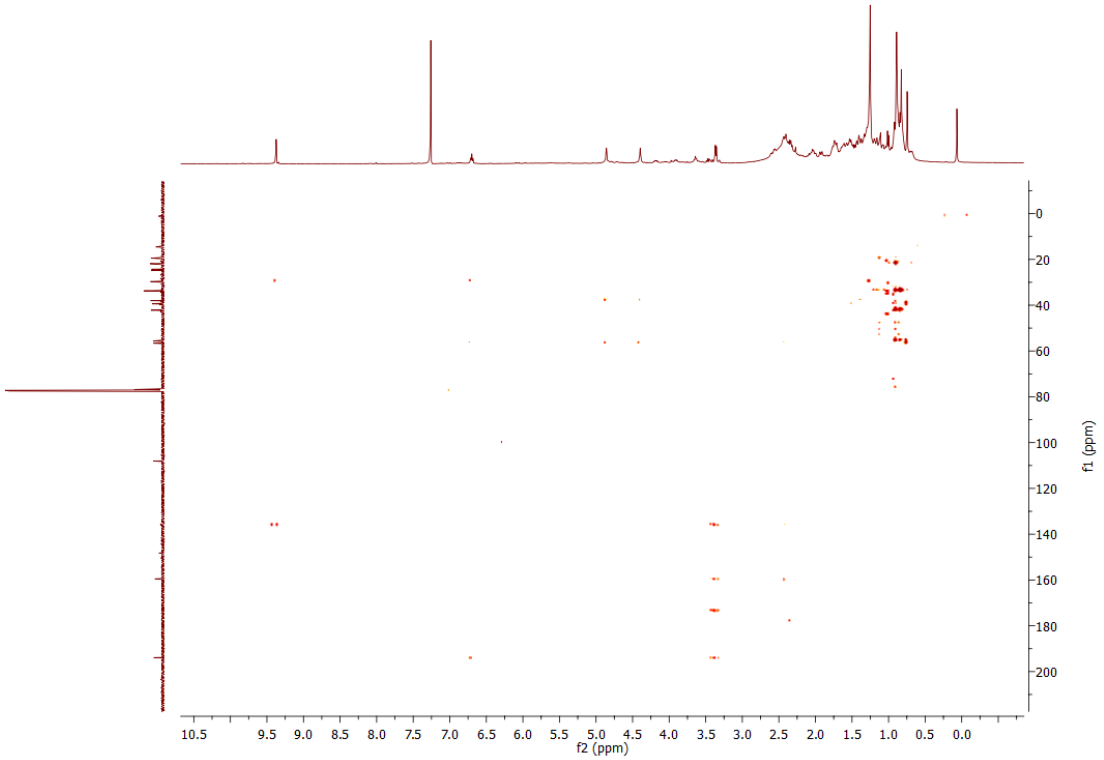


Figure D.15: HMBC NMR spectrum of **P3** in $CDCl_3$

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