

องค์ประกอบทางเคมีของใบดาลีใบเล็ก (*Amoora gigantea* Pierre ex. Laness.)

นางสาวนิสากร กำจายกิตติกุล



วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิทยาศาสตรมหาบัณฑิต

ภาควิชาเคมี

บัณฑิตวิทยาลัย จุฬาลงกรณ์มหาวิทยาลัย

พ.ศ. 2539

ISBN 974-634-253-3

ลิขสิทธิ์ของบัณฑิตวิทยาลัย จุฬาลงกรณ์มหาวิทยาลัย

I 1701685X

CHEMICAL CONSTITUENTS OF THE LEAF OF
Amoora gigantea Pierre ex. Laness

Miss Nisakorn Kamjaikittikul

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

Department of Chemistry

Graduate School

Chulalongkorn University

1996

ISBN 974-634-253-3

Thesis Title CHEMICAL CONSTITUENTS OF THE LEAF OF
Amoora gigantea Pierre ex. Laness
By Miss Nisakorn Kamjaikittikul
Department Chemistry
Thesis Advisor Assistant Professor Dr. Amorn Petsom

Accepted by the Graduate School, Chulalongkorn University in partial
fulfillment of the Requirement for the Master's Degree.

Santi Thoongsawan
..... Dean of Graduate School
(Associate Professor Santi Thoongsawan, Ph.D.)

Thesis Committee

Siri Varothai Chairman
(Associate Professor Siri Varothai, Ph.D.)

Amorn Petsom Thesis Advisor
(Assistant Professor Amorn Petsom, Ph.D.)

Suparb Boonyaratavej Member
(Associate Professor Suparb Boonyaratavej)

Sophon Roengsumran Member
(Associate Professor Sophon Roengsumran, Ph.D.)

พิมพ์ต้นฉบับทัศน์อวิทยานิพนธ์ภาษาไทยในกรอบสีเขียวนี้เพียงแผ่นเดียว

นิสากร กำจายกิตติกุล : องค์ประกอบทางเคมีของใบตาเสือใบเล็ก

(CHEMICAL CONSTITUENTS OF THE LEAF OF *Amoora gigantea* Pierre ex. Laness)

อาจารย์ที่ปรึกษา : ผศ.ดร. อmor พेचรสม, 191 หน้า. ISBN 974-634-253-3

นำใบตาเสือใบเล็กที่แห้งและบดละเอียด มาสักด้วยตัวทำละลายเชกเซน ได้สิ่งสักดั่มวีลักษณะเป็นของเหลวหนืดสีน้ำตาลอ่อนเขียว นำออกที่เหลือมาสักดัดต่อตัวทำละลายเมทานอล ได้สิ่งสักดั่มคำนวณเขียว นำสิ่งสักดั่มมาทำการแยกด้วยวิธีคลัมน์ โครโนไฟฟ์ สามารถแยกสารได้ 10 ชนิด ซึ่งสามารถพิสูจน์ได้ทางสร้าง โดยอาศัยสมบัติทางกายภาพ ปฏิกิริยาทางเคมี และข้อมูลทางスペกโตรสโคปีได้สารต่างๆ ดังนี้ ของผสมไฮโดรคาร์บอนโซ่อุ่ง (C_{22-24} , C_{27-33}), เอสเทอร์โซ่อุ่งยาว, 5α -dammara-20(21),24-dien-3-one, ของผสมแอลกออลโซอลโซ่อุ่งยาว(C_{32-33}), 5α -dammara-20(21),24-dien-3 β -ol, ของผสมสเตอโรยด์ (stigmasterol, β -sitosterol), ของผสมสเตอโรยด์ไกลโคไซด์ (stigmasterol-3-O- β -D-glucopyranoside, β -sitosterol-3-O- β -D-glucopyranoside), และ tetratriacontanoic acid ($C_{34}H_{68}O_2$) ส่วนสารอีก 2 ชนิดที่เหลือไม่สามารถหาสูตรได้เนื่องจากสารที่แยกได้นั้นมีปริมาณน้อยมาก สำหรับงานวิจัยนี้นับว่าเป็นครั้งแรกที่ได้มีการรายงานองค์ประกอบทางเคมีของพืชชนิดนี้

ศูนย์วิทยทรัพยากร
จุฬาลงกรณ์มหาวิทยาลัย

ภาควิชา ๑๗๙
สาขาวิชา ๑๗๙
ปีการศึกษา ๒๕๓๘

ลายมือชื่อนิสิต น.ส.กร คงกระพัน
ลายมือชื่ออาจารย์ที่ปรึกษา ดร. พ.ศ.
ลายมือชื่ออาจารย์ที่ปรึกษาร่วม

C725264 : MAJOR CHEMISTRY SCIENCE
KEY WORD: *Amoora gigantea*/CHEMICAL CONSTITUENTS

NISAKORN KAMJAIKITTAKUL : CHEMICAL CONSTITUENTS OF THE LEAF
OF *Amoora gigantea* Pierre ex. Laness

THESIS ADVISOR : ASSIS. PROF. AMORN PETSOM, Ph.D. 191 pp. ISBN 974-634-253-3

Dried and ground leaf of *Amoora gigantea* Pierre ex. Laness were extracted with hexane, giving a greenish-brown sticky liquid. The residue was further extracted with methanol, giving a greenish-black crude extract. The both crude extracts then were separated by column chromatography and ten compounds were obtained. These isolated compounds were established on the basis of physical properties and spectral evidences. Eight compounds can be identified, they were a mixture of long chain hydrocarbons (C_{22-24} , C_{27-33}), long chain ester (Octadecyl eicosanoate($C_{38}H_{76}O_2$)), 5α -dammara-20(21), 24-dien-3-one, a mixture of long chain alcohol (C_{32-33}), 5α -dammara-20(21), 24-dien-3 β -ol, a mixture of steroid (stigmasterol and β -sitosterol), a mixture of steroid glycoside (stigmasterol-3-O- β -D-glucopyranoside and β -sitosterol-3-O- β -D-glucopyranoside), and tetratriacontanoic acid($C_{34}H_{68}O_2$). The other two substances cannot be identified due to the limited amount. This is the first report ever known about the chemical constituents of this particular plant species.

ศูนย์วิทยบรพยากร
จุฬาลงกรณ์มหาวิทยาลัย

ภาควิชา.....เคมี
สาขาวิชา.....เคมี
ปีการศึกษา.....๒๕๓๘

ลายมือชื่อนิสิต.....Nisakorn Kamjaikittakul
ลายมือชื่ออาจารย์ที่ปรึกษา.....Dr. ket
ลายมือชื่ออาจารย์ที่ปรึกษาร่วม.....

ACKNOWLEDGEMENT



The author would like to express her deepest sincere gratitude and appreciation to Assistant Professor Dr. Amorn Petsom, her major advisor for his guidance, continued interest and encouragement throughout the course of this research. She would like to express her appreciation to all of staff members, Associate Professor Dr. Siri Varothai, Associate Professor Dr. Sophon Roengsumran, and Associate Professor Suparb Boonyaratavej, Department of Chemistry, Faculty of Science, Chulalongkorn University for their comments, guidances and valuable suggestions. Thanks are extended to the Graduate School and Department of Chemistry, Faculty of Science, Chulalongkorn University for the financial support and to the staffs of the Scientific and Technology Research Equipment Centre, Chulalongkorn University for giving services on sample analysis. Moreover, she would like to thank Mr. Kularb Joijaroen for providing the plants and thank Institute of Biotechnology and Genetic Engineering, Chulalongkorn University for elemental analysis.

Finally, she wishes to express her grateful appreciation to her parents for their love, encouragement and cheerfulness through her graduate study.

CONTENTS

	Pages
Abstract in Thai.....	iv
Abstract in English.....	v
Acknowledgement.....	vi
List of Tables.....	x
List of Figures.....	xii
List of Schemes.....	xv
List of Abbreviations.....	xvi

Chapter

I INTRODUCTION.....	1
-General Characterization of the Plants in the Family of Meliaceae.....	2
-Research in the Biological and Pharmacological Activity	
Screening of Some Meliaceous Plants.....	3
-General Characterization of the Plants in “Amoora” Genus.....	6
-Research Studies in Chemical Constituents on the Plants	
in “Amoora” Genus.....	7
-Characterization and Utilization for Medicine of <i>A. gigantea</i> Pierre... ..	13
II EXPERIMENTS AND RESULTS	26
-Plant Materials.....	26
-Instruments and Equipments.....	26
-Chemical Reagents.....	28
-Physical Separation Techniques.....	28
-Colour Tests.....	29
-Extraction.....	30

	Pages
-Isolation of Chemical Constituents of the leaf of <i>Amoora gigantea</i> Pierre ex. Laness.	32
-Separation of Crude Hexane Extract by Column Chromatography..	32
-Separation of Crude Methanol Extract by Column Chromatography. 34	
-Purification and Properties of the Eluted Compounds by Column Chromatography.....	36
-Purification and Properties of Compound <u>1</u>	36
-Purification and Properties of Compound <u>2</u>	37
-Purification and Properties of Compound <u>3</u>	38
-Purification and Properties of Compound <u>4</u>	40
-Purification and Properties of Compound <u>5</u>	41
-Purification and Properties of Compound <u>6</u>	43
-Purification and Properties of Compound <u>7</u>	45
-Purification and Properties of Compound <u>8</u>	46
-Purification and Properties of Compound <u>9</u>	47
-Purification and Properties of Compound <u>10</u>	48
 III RESULTS AND DISCUSSION	49
-Structural Elucidation of the Isolated Compounds from the leaf of <i>Amoora gigantea</i> Pierre ex. Laness.....	49
-Structural Elucidation of Compound <u>1</u>	49
-Structural Elucidation of Compound <u>2</u>	53
-Structural Elucidation of Compound <u>3</u>	55
-Structural Elucidation of Compound <u>4</u>	66
-Structural Elucidation of Compound <u>5</u>	69
-Structural Elucidation of Compound <u>6</u>	79
-Structural Elucidation of Compound <u>7</u>	83
-Structural Elucidation of Compound <u>8</u>	86

	Pages
-Structural Elucidation of Compound <u>9</u>	89
-Structural Elucidation of Compound <u>10</u>	91
IV CONCLUSION.....	93
REFERENCES.....	96
APPENDIX.....	105
VITA.....	191

ศูนย์วิทยทรัพยากร
จุฬาลงกรณ์มหาวิทยาลัย

LIST OF TABLES

Tables	Pages
1 The Biological Activity Screening of Some Meliaceous Plants.....	3
2 Pharmacological Activities of the Meliaceous Plants.....	4
3 Summary of Research in Chemical Constituents of Some <i>Amoora</i> Species.....	11
4 The Results of the Column Chromatographic Separation of Crude Hexane Extract (Fraction I) by Column Chromatography.....	32
5 The Results of the Column Chromatographic Separation of Crude Methanol Extract (Fraction II) by Column Chromatography.....	34
6 The IR Absorption Band Assignments of Compound <u>1</u>	49
7 Retention Times of Standard Long Chain Aliphatic Hydrocarbons (C ₂₄ -C ₂₆ , C ₂₈ -C ₃₃) and Compound <u>1</u> with Number of Carbons.....	51
8 Saturated Long Chain Aliphatic Hydrocarbons Found in Compound <u>1</u>	52
9 The IR Absorption Band Assignments of Compound <u>2</u>	53
10 The IR Absorption Band Assignments of Compound <u>3</u>	55
11 The Data Comparison of ¹ H-NMR Spectrum of 5 α -dammara-20(21), 24-dien-3-one and Compound <u>3</u>	57
12 The Data Comparison of ¹³ C-NMR Spectrum of 5 α -dammara-20(21), 24-dien-3-one and Compound <u>3</u>	58
13 The Chemical Shift of ¹³ C- ¹ H Correlation Assignments of Compound <u>3</u>	60
14 The Chemical Shift of ¹³ C- ¹ H COLOC Assignments of Compound <u>3</u> . .	62

Tables	Pages
15 The Comparison of Melting Points Between 5α -dammara-20(21), 24-dien-3-one With Compound <u>3</u>	63
16 The IR Absorption Band Assignments of Compound <u>4</u>	67
17 Retention Time of Standard Long Chain Aliphatic Primary Alcohols (C =14, 16, 18, 20, 22) and Compound <u>4</u>	68
18 Various Type of Saturated Long Chain Aliphatic Primary Alcohols Found in Compound <u>4</u>	69
19 The IR Absorption Band Assignments of Compound <u>5</u>	70
20 The Comparison of ^1H -NMR Spectra of 5α -dammara-20(21), 24-dien-3 β -ol and Compound <u>5</u>	71
21 The Comparison of ^{13}C -NMR Spectra of 5α -dammara-20(21), 24-dien-3 β -ol and Compound <u>5</u>	72
22 The Chemical Shift of ^{13}C - ^1H Correlation Assignments of Compound <u>5</u>	73
23 The Chemical Shift of ^{13}C - ^1H COLOC Assignments of Compound <u>5</u> . 75	75
24 The Comparison of Melting Points Between 5α -dammara-20(21), 24-dien-3 β -ol With Compound <u>5</u>	76
25 The IR Absorption Band Assignments of Compound <u>6</u>	79
26 Retention Times of the Mixture of Three Standard Steroids and Compound <u>6</u>	81
27 Various Type Steroids Found in Compound <u>6</u>	82
28 The IR Absorption Band Assignments of Compound <u>7</u>	83
29 Various Type of Steroid Glycosides Found in Compound <u>7</u>	85
30 The IR Absorption Band Assignments of Compound <u>8</u>	86
31 The IR Absorption Band Assignments of Compound <u>9</u>	89
32 The IR Absorption Band Assignments of Compound <u>10</u>	91

LIST OF FIGURES

Figures	Pages
1 The Leaf of <i>Amoora gigantea</i> Pierre ex. Laness.....	14
2 Organic Compounds Found in the Seeds of <i>Amoora rohituka</i> W&A. (syn. <i>Aphanamixis polystachya</i> Parker).....	15
3 Organic Compounds Found in the Stem Barks of <i>Amoora rohituka</i> W&A. (syn. <i>Aphanamixis polystachya</i> Parker).....	18
4 Organic Compounds Found in the Roots of <i>Amoora rohituka</i> W&A. (syn. <i>Aphanamixis polystachya</i> Parker).....	21
5 Organic Compounds Found in the Fruits of <i>Amoora rohituka</i> W&A.. (syn. <i>Aphanamixis polystachya</i> Parker).....	22
6 Organic Compounds Found in the Heartwoods of <i>Amoora wallachi</i>	22
7 Organic Compounds Found in the Stem Barks of <i>Amoora rohituka</i> Wall	23
8 Organic Compounds Found in the Fruits of <i>Amoora grandifolia</i> (syn. <i>Aphanamixis grandifolia</i>).....	23
9 Organic Compounds Found in the Seeds of <i>Amoora grandifolia</i> (syn. <i>Aphanamixis grandifolia</i>).....	24
10 The IR Spectrum of Compound <u>1</u>	106
11 The Gas-Liquid Chromatogram of Standard Long Chain Aliphatic Hydrocarbons (C ₂₄ -C ₂₆ , C ₂₈ -C ₃₃).....	107
12 The Gas-Liquid Chromatogram of Compound <u>1</u>	108
13 The Calibration Curve of Standard Long Chain Aliphatic Hydrocarbons (C ₂₄ -C ₂₆ , C ₂₈ -C ₃₃).....	109
14 The IR Spectrum of Compound <u>2</u>	110
15 The ¹ H-NMR Spectrum of Compound <u>2</u>	111
16 The Mass Spectrum of Compound <u>2</u>	112

Figures	Pages
17 The Mass Spectrum of Compound <u>2</u> Compared With the Mass Spectrum of Eicosanoic Acid, Octadecyl Ester ($C_{38}H_{76}O_2$).....	113
18 The IR Spectrum of Compound <u>3</u>	114
19-22 The 1H -NMR Spectrum of Compound <u>3</u>	115
23-24 The ^{13}C -NMR Spectrum of Compound <u>3</u>	119
25-26 The DEPT 90 and DEPT 135 ^{13}C -NMR Spectrum of Compound <u>3</u> ... 121	
27-28 1H - 1H COSY of Compound <u>3</u>	123
29-31 ^{13}C - 1H Correlation of Compound <u>3</u>	125
32-34 1H - 1H NOESY of Compound <u>3</u>	128
35-37 ^{13}C - 1H COLOC of Compound <u>3</u>	131
38 The Mass Spectrum of Compound <u>3</u>	134
39 The IR Spectrum of Compound <u>4</u>	135
40 The 1H -NMR Spectrum of Compound <u>4</u>	136
41 The ^{13}C -NMR Spectrum of Compound <u>4</u>	137
42 The Gas-Liquid Chromatogram of Standard Long Chain Aliphatic Primary Alcohols($C = 14, 16, 18, 20, 22$).....	138
43 The Gas-Liquid Chromatogram of Compound <u>4</u>	139
44 The Calibration Curve of Standard Long Chain Aliphatic Primary Alcohols($C = 14, 16, 18, 20, 22$).....	140
45 The IR Spectrum of Compound <u>5</u>	141
46-49 The 1H -NMR Spectrum of Compound <u>5</u>	142
50 The ^{13}C -NMR Spectrum of Compound <u>5</u>	146
51-53 The DEPT 90 and DEPT 135 ^{13}C -NMR Spectrum of Compound <u>5</u> ... 147	
54-58 1H - 1H COSY of Compound <u>5</u>	150
59-60 ^{13}C - 1H Correlation of Compound <u>5</u>	155
61-65 1H - 1H NOESY of Compound <u>5</u>	157
66-68 ^{13}C - 1H COLOC of Compound <u>5</u>	162
69 The Mass Spectrum of Compound <u>5</u>	165

Figures	Pages
70 The IR Spectrum of Compound <u>6</u>	166
71-75 The ¹ H-NMR Spectrum of Compound <u>6</u>	167
76-77 The ¹³ C-NMR Spectrum of Compound <u>6</u>	172
78-80 The DEPT 90 and DEPT 135 ¹³ C-NMR Spectrum of Compound <u>6</u>	174
81 The Gas-Liquid Chromatogram of Three Standard Steroids.....	177
82 The Gas-Liquid Chromatogram of Compound <u>6</u>	178
83 The Mass Spectrum of Compound <u>6</u>	179
84 The IR Spectrum of Compound <u>7</u>	180
85 The ¹ H-NMR Spectrum of Compound <u>7</u>	181
86 The ¹³ C-NMR Spectrum of Compound <u>7</u>	182
87 The Mass Spectrum of Compound <u>7</u>	183
88 The IR Spectrum of Compound <u>8</u>	184
89 The Mass Spectrum of Compound <u>8</u>	185
90 The Mass Spectrum of Compound <u>8</u> Compared With Mass Spectrum of Tetratriacontanoic Acid (C ₃₄ H ₆₈ O ₂).....	186
91 The IR Spectrum of Compound <u>9</u>	187
92 The mass Spectrum of Compound <u>9</u>	188
93 The IR Spectrum of Compound <u>10</u>	189
94 The Mass Spectrum of Compound <u>10</u>	190
95 All Isolated Substances from the Leaf of <i>Amoora gigantea</i> Pieere ex. Laness.	95

LIST OF SCHEMES

Schemes	Pages
1 Extraction of the Leaf of <i>Amoora gigantea</i> Pierre ex. Laness.	31
2 The Possible Mass Fragmentation Pattern of Compound <u>3</u>	64
3 The Possible Mass Fragmentation Pattern of Compound <u>5</u>	77
4 The Possible Mass Fragmentation Pattern of Compound <u>8</u>	88

LIST OF ABBREVIATIONS

TMS	Tetramethylsilane
Hz	Hertz
ppm	part per million
δ	chemical shift
s	singlet (NMR)
d	doublet (NMR)
t	triplet (NMR)
q	quartet (NMR)
m	multiplet (NMR)
dd	doublet of doublet
J	coupling constant
cm^{-1}	unit of wave number
M^+	molecular ion
m/e	mass to charge ratio
M.W.	molecular weight
ν_{max}	the wavelength at maximum absorption
br	broad
s	strong
m	medium
w	weak
%	percent
R_t	Retention time in gas chromatography
conc.	concentration
syn.	synonym