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SYNTHESIS OF ISOQUINOLINE ALKALOIDS FROM BARAKOL

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

Department of Chemistry

Faculty of Science

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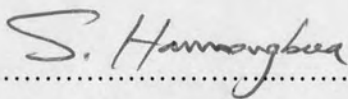
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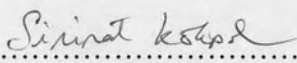
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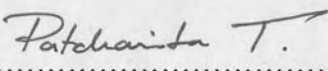
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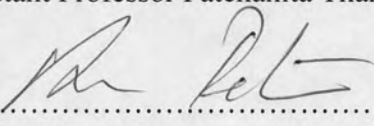
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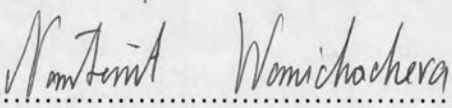
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บาราคอล, cassiarin A และ B เป็นสารประกอบที่มีฤทธิ์ทางชีวภาพสกัดได้จากใบของ *Cassia siamea* โดยที่บาราคอลมีลักษณะโครงสร้างโมเลกุลเป็นแบบ 3 วงเชื่อมติดกันคล้ายกับ สาร cassiarin A และ B ซึ่งพบสารนี้เป็นองค์ประกอบหลักในใบของ *Cassia siamea* ในขณะที่ cassiarin A และ B ที่มีฤทธิ์ต้านเชื้อ *Plasmodium falciparum* พบได้ปริมาณน้อย ดังนั้นการ เปลี่ยนบาราคอลเป็นสารอนุพันธ์ของไอโซควิโนลินแอลคาลอยด์ที่มีฤทธิ์ทางชีวภาพอย่าง cassiarin A และ B จึงได้เริ่มขึ้น ในการสังเคราะห์ cassiarin A เริ่มจากการเตรียมบาราคอลให้อยู่ในรูปของแอนไฮโดรบาราคอลคลอไรด์ที่มีความเสถียรมาทำปฏิกิริยากับแอมโมเนียไฮดรอกไซด์และทำปฏิกิริยาต่อกับกรดจะได้ผลิตภัณฑ์เป็น cassiarin A ในวิธีการเดียวกัน cassiarin B และอนุพันธ์ที่ตำแหน่งไนโตรเจนมีหมู่แทนที่แบบต่างๆ สังเคราะห์ได้จากปฏิกิริยาของแอนไฮโดรบาราคอลคลอไรด์กับเมทิล-4-อะมิโนบิวทิลเรตหรือสารประกอบแอมีนชนิดปฐมภูมิ อื่นๆ เช่น บิวทิลแอมีน, ไซโคลเฮกซิลแอมีน, แอนิลีน และ เบนซิลแอมีน เมื่อนำอนุพันธ์ของ cassiarin ทั้งหมดที่ได้จากการสังเคราะห์นำไปทดสอบฤทธิ์ต้านเชื้อ *Plasmodium falciparum* และฤทธิ์ ความเป็นพิษต่อเซลล์มะเร็ง พบว่าไม่มีฤทธิ์ทางชีวภาพ

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Barakol, cassiarins A and B are biological active compounds extracted from leaves of *Cassia siamea*. Barakol, with an unprecedented tricyclic core structure similar to cassiarins A and B, is a major constituent in leaves of *Cassia siamea*. Cassiarins A and B, which have been known to exhibit potent antiplasmodial activity against *Plasmodium falciparum*, could be extract from the natural source in low yield. Therefore, transformation of barakol to its bioactive isoquinoline alkaloids as cassiarins A and B has been investigated. Synthesis of cassiarin A started from a conversion of barakol to its more stable anhydrobarakol chloride, followed by a reaction with ammonium hydroxide and subsequently reacted with acid to form the cassiarin A. Similarly, cassiarin B and its derivative bearing different *N*-substituents was synthesized by the reaction of anhydrobarakol chloride and methyl-4-aminobutyrate or the other primary amine, such as butylamine, cyclohexylamine, aniline and benzylamine. All new cassiarin derivatives exhibited negative antiplasmodial activity against *Plasmodium falciparum* and cytotoxic activity against cancer cell.

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LIST OF ABBREVIATIONS

°C	:	degree Celcius
¹³ C-NMR	:	carbon-13 nuclear magnetic resonance spectroscopy
¹ H-NMR	:	proton nuclear magnetic resonance spectroscopy
AcOH	:	acetic acid
aq.	:	aqueous
AR	:	analytical reagent
b.p.	:	boiling point
CD ₃ OD	:	deuterated methanol
CDCl ₃	:	deuterated chloroform
cm ⁻¹	:	unit of wavenumber (IR)
conc.	:	concentration
d	:	doublet (NMR)
D ₂ O	:	dideuterium oxide
dd	:	doublet of doublet (NMR)
dec.	:	decompose
DMSO	:	dimethylsulfoxide
DMSO- <i>d</i> ₆	:	deuterated dimethylsulfoxide
equiv.	:	equivalent (s)
ESI-MS	:	electrospray ionization mass spectrometry
EtOAc	:	ethyl acetate
EtOH	:	ethanol
FT-IR	:	fourier transform infrared resonance spectroscopy
g	:	gram (s)
glac.	:	glacial
h	:	hour (s)
HMBC	:	heteronuclear multiple bond coherence
HR-ESI-MS	:	high resolution electrospray ionization mass spectrometry
HSQC	:	heteronuclear single quantum coherence
Hz	:	hertz (s)
IR	:	infrared resonance spectroscopy
<i>J</i>	:	coupling constant

kg	:	kilogram (s)
L	:	liter (s)
M	:	molar (s)
m	:	multiplet (NMR)
m.p.	:	melting point
<i>m/z</i>	:	mass per charge ratio
MeOH	:	methanol
mg	:	milligram (s)
min	:	minute
mL	:	milliliter (s)
mM	:	millimolar (s)
mmol	:	millimole (s)
mol	:	mole (s)
MS	:	mass spectroscopy
NH ₄ OAc	:	ammonium acetate
NMR	:	nuclear magnetic resonance spectroscopy
ppm	:	parts per million
q	:	quartet (NMR)
rt	:	room temperature
s	:	singlet (NMR)
t	:	triplet (NMR)
TEA	:	triethylamine
THF	:	tetrahydrofuran
v/v	:	volume per volume ratio
w/v	:	weight per volume ratio
δ	:	chemical shift
δ_H	:	proton chemical shift
δ_C	:	carbon-13 chemical shift
μL	:	microliter (s)

LIST OF ABBREVIATIONS

°C	:	degree Celcius
¹³ C-NMR	:	carbon-13 nuclear magnetic resonance spectroscopy
¹ H-NMR	:	proton nuclear magnetic resonance spectroscopy
AcOH	:	acetic acid
aq.	:	aqueous
AR	:	analytical reagent
b.p.	:	boiling point
CD ₃ OD	:	deuterated methanol
CDCl ₃	:	deuterated chloroform
cm ⁻¹	:	unit of wavenumber (IR)
conc.	:	concentration
d	:	doublet (NMR)
D ₂ O	:	dideuterium oxide
dd	:	doublet of doublet (NMR)
dec.	:	decompose
DMSO	:	dimethylsulfoxide
DMSO- <i>d</i> ₆	:	deuterated dimethylsulfoxide
equiv.	:	equivalent (s)
ESI-MS	:	electrospray ionization mass spectrometry
EtOAc	:	ethyl acetate
EtOH	:	ethanol
FT-IR	:	fourier transform infrared resonance spectroscopy
g	:	gram (s)
glac.	:	glacial
h	:	hour (s)
HMBC	:	heteronuclear multiple bond coherence
HR-ESI-MS	:	high resolution electrospray ionization mass spectrometry
HSQC	:	heteronuclear single quantum coherence
Hz	:	hertz (s)
IR	:	infrared resonance spectroscopy
<i>J</i>	:	coupling constant

kg	:	kilogram (s)
L	:	liter (s)
M	:	molar (s)
m	:	multiplet (NMR)
m.p.	:	melting point
<i>m/z</i>	:	mass per charge ratio
MeOH	:	methanol
mg	:	milligram (s)
min	:	minute
mL	:	milliliter (s)
mM	:	millimolar (s)
mmol	:	millimole (s)
mol	:	mole (s)
MS	:	mass spectroscopy
NH ₄ OAc	:	ammonium acetate
NMR	:	nuclear magnetic resonance spectroscopy
ppm	:	parts per million
q	:	quartet (NMR)
rt	:	room temperature
s	:	singlet (NMR)
t	:	triplet (NMR)
TEA	:	triethylamine
THF	:	tetrahydrofuran
v/v	:	volume per volume ratio
w/v	:	weight per volume ratio
δ	:	chemical shift
δ_{H}	:	proton chemical shift
δ_{C}	:	carbon-13 chemical shift
μL	:	microliter (s)