

เอกสารวอร์นอีเทอร์เชื่อมกับ 1,4-ไดไฮโดรพรีดีนเพื่อเป็นฟลูออเรสเซนซ์คีโมเซ็นเซอร์ชนิดใหม่



นายวโรธร ไพรสุวรรณ

วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิทยาศาสตรมหาบัณฑิต

สาขาวิชาเคมี ภาควิชาเคมี

คณะวิทยาศาสตร์ จุฬาลงกรณ์มหาวิทยาลัย

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ลิขสิทธิ์ของจุฬาลงกรณ์มหาวิทยาลัย



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AZACROWN ETHER LINKED WITH 1,4-DIHYDROPYRIDINE AS NEW FLUORESCENCE
CHEMOSENSORS

Mr. Waroton Paisuwan

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Thesis Title AZACROWN ETHER LINKED WITH 1,4-DIHYDROPYRIDINE AS NEW FLUORESCENCE CHEMOSENSORS
By Mr. Waroton Paisuwan
Field of Study Chemistry
Thesis Advisor Anawat Ajavakom, Ph.D.
Thesis Co-Advisor Associate Professor Mongkol Sukwattanasinitt, Ph.D.

Accepted by the Faculty of Science, Chulalongkorn University in Partial Fulfillment of the Requirements for the Master's Degree





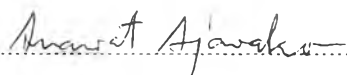
..... Dean of the Faculty of Science

(Professor Supot Hannongbua, Dr.rer.nat.)


THESIS COMMITTEE

..... Chairman

(Assistant Professor Varawut Tangpasuthadol, Ph.D.)

..... Thesis Advisor

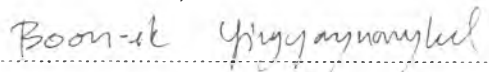
(Anawat Ajavakom, Ph.D.)

..... Thesis Co-Advisor

(Associate Professor Mongkol Sukwattanasinitt, Ph.D.)

..... Examiner

(Luxsana Dubas, Ph.D.)


..... External Examiner


(Assistant Professor Boon-ek Yingyongnarongkul, Ph.D.)

วโรธร ไพรสุวรรณ : เอซาคารานอีเทอร์เชื่อมกับ 1,4-ไดไฮโดรพิริดีนเพื่อเป็นฟลูออเรสเซนซ์เคมีเซ็นเซอร์ชนิดใหม่. (AZACROWN ETHER LINKED WITH 1,4-DIHYDROPYRIDINE AS NEW FLUORESCENCE CHEMOSENSORS) อ.ที่ปรึกษาวิทยานิพนธ์หลัก: อ. ดร.อนวัช อาชวาคม, อ.ที่ปรึกษาวิทยานิพนธ์ร่วม: รศ. มงคล สุขวัฒนาสินธิ์, 85 หน้า.

ฟลูออเรสเซนซ์เซ็นเซอร์ที่มีสมบัติการละลายน้ำที่ดีเป็นที่นิยมนำมาประยุกต์ใช้ในการตรวจวัดไอออนของโลหะในตัวทำละลายน้ำ งานวิจัยนี้จึงสังเคราะห์โมเลกุลที่ประกอบด้วยหน่วยของ 1,4-ไดไฮโดรพิริดีน (DHP) ทำหน้าที่เป็นหน่วยให้สัญญาณฟลูออเรสเซนซ์และหน่วยของเอซาคารานอีเทอร์ทำหน้าที่เป็นหน่วยจับที่มีสมบัติในการละลายน้ำที่ดี การสังเคราะห์อนุพันธ์ 1,4-ไดไฮโดรพิริดีนที่เชื่อมกับวงเอซาคารานอีเทอร์สามารถเตรียมได้ภายใน 3 ขั้นตอน เริ่มต้นจากการปดวแบบไซโคลไดรเมอไรเซชันของเบต้าอะมิโนอคริเลทได้เป็น Et-DHP-OH ต่อด้วยการเปลี่ยนหมู่ไฮดรอกซิลเป็นหมู่ทอซิลเลตผ่านปฏิกิริยาทอซิลเลชันได้เป็น Et-DHP-OTs ตามด้วยปฏิกิริยาการแทนที่ด้วยเอซาคารานอีเทอร์ได้เป็น Et-DHP-AC(1-3) จากการศึกษาสมบัติทางกายภาพเชิงแสงในน้ำมีลิลคิว พบว่าค่าการดูดกลืนแสงสูงสุดที่ความยาวคลื่น 367, 369, และ 362 นาโนเมตร และค่าการคายแสงสูงสุดที่ความยาวคลื่นเหมือนกันที่ 439 นาโนเมตรที่ประสิทธิภาพการคายแสง (ϕ_f) ที่ 0.41, 0.45, และ 0.46 ตามลำดับ พบว่า Et-DHP-AC(3) มีความจำเพาะกับทอง(III) ที่ความเข้มข้นต่ำสุด 50 μM ด้วยการระงับสัญญาณฟลูออเรสเซนซ์ซึ่งคาดว่าเกิดออกซิเดชันของวงไดไฮโดรพิริดีน กลายเป็นวงพิริดีเนียมตามผลที่แสดงใน $^1\text{H NMR}$ ยิ่งไปกว่านั้น Et-DHP-AC(1-3) ยังใช้เป็นฟลูออเรสเซนซ์เซ็นเซอร์ในตัวทำละลายผสมระหว่างเตตระไฮโดรฟิวแรนหรืออะซิโตไนโตรลกับน้ำมีลิลคิวซึ่งส่งผลทำให้สัญญาณฟลูออเรสเซนซ์เพิ่มขึ้น จากผลการศึกษาพบว่า Et-DHP-AC(2) แสดงการเพิ่มสัญญาณฟลูออเรสเซนซ์กับโครเมียม(III) อย่างจำเพาะเจาะจงมากที่สุดแล้วเกิดเป็นสารประกอบเชิงซ้อนด้วยอัตราส่วนระหว่าง Et-DHP-AC(2):Cr $^{3+}$ เท่ากับ 3:1 ในเตตระไฮโดรฟิวแรนกับน้ำมีลิลคิว (อัตราส่วนปริมาตร 1:1)

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สาขาวิชา เคมี
ปีการศึกษา 2556

ลายมือชื่อนิสิต 

ลายมือชื่อ อ.ที่ปรึกษาวิทยานิพนธ์หลัก 

ลายมือชื่อ อ.ที่ปรึกษาวิทยานิพนธ์ร่วม 

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WAROTON PAISUWAN: AZACROWN ETHER LINKED WITH 1,4-DIHYDROPYRIDINE AS NEW FLUORESCENCE CHEMOSENSORS. ADVISOR: ANAWAT AJAVAKOM, Ph.D., CO-ADVISOR: ASSOC. PROF. MONGKOL SUKWATTANASINITT, Ph.D., 85 pp.

Water soluble fluorescent sensors are of interest for sensing the metal ion in aqueous media. The target molecules possessing a DHP moiety as fluorophore and azacrown ring as a receptor and water soluble moiety were synthesized. The synthetic preparation of the series of 1,4-dihydropyridine (DHP) derivatives linked with various sizes of azacrown ring involves 3 steps of reaction. Firstly, Et-DHP-OH can be obtained from the cyclotrimerization of β -amino acrylates. Then, the hydroxyl group of Et-DHP-OH was tosylated followed by the substitution with the azacrown ether (n=1-3) to afford the corresponding Et-DHP-AC(1-3). According to the investigation results of photophysical property in milliQ water, these Et-DHP-AC(1-3) exhibited the absorption maxima at 367, 369, and 362 nm and the similar emission peak at 439 nm with the fluorescence quantum efficiencies (ϕ_f) of 0.41, 0.45, and 0.46, respectively. The Et-DHP-AC(3) showed selective fluorescence quenching by gold(III) with the the lowest detectable concentration of 50 μ M. The fluorescence quenching occurred through the oxidation reaction of the DHP into a pyridinium ring also confirmed by the ^1H NMR results. Moreover, when Et-DHP-AC(1-3) were used as sensors in THF or acetonitrile and milliQ water, they gave fluorescence enhancement signals. As a result, Et-DHP-AC(2) was found to demonstrate the best selective enhancement with chromium(III) that formed complexation with the ratio of Et-DHP-AC(2):Cr $^{3+}$ equal to 3:1 in THF/milliQ water (v/v=1:1).

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Student's Signature *Naroton Paisuwan*

Advisor's Signature *Anawat Ajavakom*

Co-Advisor's Signature *Mongkol Sukwattanasinitt*

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LIST OF ABBREVIATIONS

ACN	acetonitrile
Ar	aryl group
a.u.	arbitrary unit
calcd	calculated
^{13}C NMR	carbon-13 nuclear magnetic resonance
CDCl_3	deuterated chloroform
CH_2Cl_2	methylene chloride
DHP	1,4-dihydropyridine
D_2O	deuterium oxide
d	doublet (NMR)
dd	doublet of doublet (NMR)
ESI	electrospray ionization mass spectrometry
EtOAc	ethyl acetate
equiv	equivalent (s)
FT-IR	fourier transform infrared spectroscopy
g	gram (s)
^1H NMR	proton nuclear magnetic resonance
Hz	hertz
HRMS	high resolution mass spectrum
h	hour (s)
IR	Infrared
J	coupling constant
m	multiplet (NMR)
mg	milligram (s)
mL	milliliter (s)
mM	millimolar
mmol	millimole (s)

m/z	mass per charge
M.W.	molecular weight
M	molar
MHz	megahertz
nm	nanometer (s)
q	quartet (NMR)
R	alkyl group
rt	room temperature
s	singlet (NMR)
t	triplet (NMR)
THF	tetrahydrofuran
TLC	thin layer chromatography
UV	ultraviolet
δ	chemical shift
$^{\circ}\text{C}$	degree Celsius
μL	microliter (s)
μM	micromolar (s)
ϵ	molar absorptivity
λ	wavelength
Φ	quantum yield
% yield	percentage yield