


เทคนิคการเตรียมตัวอย่างเพื่อการวิเคราะห์สารเคมีกำจัดศัตรูพืชกลุ่มออร์แกโนคลอรีน
ในขมิ้นชัน



นางสาวสุนิตรา จารุไพบูลย์

วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิทยาศาสตรมหาบัณฑิต

สาขาวิชาเคมี ภาควิชาเคมี

คณะวิทยาศาสตร์ จุฬาลงกรณ์มหาวิทยาลัย

ปีการศึกษา 2546

ISBN -974-17-5396-9

ลิขสิทธิ์ของจุฬาลงกรณ์มหาวิทยาลัย

SAMPLE PREPARATION TECHNIQUES FOR DETERMINATION
OF ORGANOCHLORINE PESTICIDES IN *Curcuma longa*.

Miss Sanitra Jarupaiboon

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

Department of Chemistry

Faculty of Science

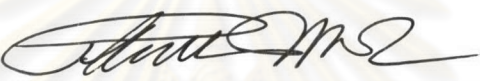
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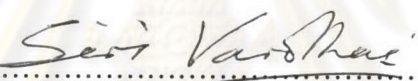
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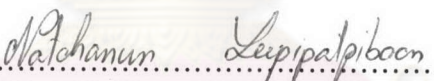
Thesis Title SAMPLE PREPARATION TECHNIQUES FOR
 DETERMINATION OF ORGANOCHLORINE PESTICIDES IN
 Curcuma longa
By Miss Sanitra Jarupaiboon
Field of Study Chemistry
Thesis Advisor Assistant Professor Natchanun Leepipatpiboon, Dr.rer.nat

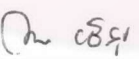
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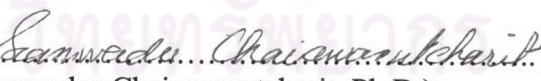

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สนิตรา จารุไพบูลย์: เทคนิคการเตรียมตัวอย่างเพื่อการวิเคราะห์สารเคมีกำจัดศัตรูพืช
กลุ่มออร์แกโนคลอรีนในขมิ้นชัน. (SAMPLE PREPARATION TECHNIQUES FOR
DETERMINATION OF ORGANOCHLORINE PESTICIDES IN *Curcuma longa*
อาจารย์ที่ปรึกษา: ผู้ช่วยศาสตราจารย์ ดร. ณัฐชนน ลีพิพัฒน์ไพบูลย์ 160 หน้า. ISBN
974-17-5396-9

งานวิจัยนี้เป็นการพัฒนาเทคนิคการเตรียมตัวอย่างให้เหมาะสมเพื่อการวิเคราะห์สารเคมี
กำจัดศัตรูพืชกลุ่มออร์แกโน คลอรีนในขมิ้นชัน โดยองค์ประกอบหลักในขมิ้นชันจะมีสารสีและ
กลิ่นของน้ำมันหอมระเหย ซึ่งรบกวนการหาปริมาณของสารเคมีกำจัดศัตรูพืชกลุ่มออร์แกโน
คลอรีนในขมิ้นชัน โดยใช้เทคนิค Gas chromatography โดยมี μ -electron capture detector เป็น
เครื่องตรวจวัด เทคนิคการเตรียมตัวอย่างแบบ multiresidue method สำหรับการวิเคราะห์หาปริมาณ
สารสารเคมีกำจัดศัตรูพืชกลุ่มออร์แกโน คลอรีน 17 ชนิด โดยใช้ solid phase extraction (SPE) ใน
ขั้นตอนการ clean up หลังจากการสกัดสารตัวอย่างด้วยสารละลายผสมของ เฮกเซน : ไดคลอโร
มีเทน ซึ่ง SPE ที่ใช้ประกอบด้วยซิลิกาเจล ฟลอริซิล และ โซเดียมแอนไฮดรัสซัลเฟต สำหรับค่าตัว
แปรของการตรวจสอบความถูกต้องของวิธีการวิเคราะห์ เป็นที่น่าพอใจดังนี้ ช่วงความเป็นเส้นตรง
ของการวิเคราะห์อยู่ในช่วงความเข้มข้น 1 – 500 นาโนกรัมต่อมิลลิกรัม ค่าขีดจำกัดต่ำสุดของ
วิธีการวิเคราะห์มีค่าอยู่ในช่วง 0.5 – 15.4 นาโนกรัมต่อกรัม และขีดจำกัดต่ำสุดในการหาปริมาณ
ของวิธีการวิเคราะห์มีค่าอยู่ในช่วง 1.4 – 51.2 นาโนกรัมต่อกรัม ความเที่ยงของวิธีการวิเคราะห์และ
การเตรียมตัวอย่างมีประสิทธิภาพดี โดยเมื่อเติมสารมาตรฐานในขมิ้นชันที่ระดับความเข้มข้น 5, 25
และ 125 นาโนกรัมต่อกรัม ให้ค่าสัมประสิทธิ์ความแปรผันของการวิเคราะห์น้อยกว่าร้อยละ 5.5
สำหรับค่าร้อยละของการคืนกลับอยู่ในช่วง 60.33 – 180.64 ผลวิเคราะห์ตัวอย่างขมิ้นชันที่มี
จำหน่ายในประเทศไทยจำนวน 6 ตัวอย่าง พบว่าตัวอย่างทั้งหมดมีการปนเปื้อนของดีดีที และ
อนุพันธ์ของดีดีทีในระดับความเข้มข้นต่ำและไม่เกินข้อกำหนดของ USP ดังนั้นวิธีการวิเคราะห์
และการเตรียมตัวอย่างในงานวิจัยนี้สามารถที่จะนำมาเป็นวิธีมาตรฐานในการวิเคราะห์หาปริมาณ
สารเคมีกำจัดศัตรู พืชกลุ่มออร์แกโนคลอรีนในขมิ้นชัน อีกทั้งยังเป็นประโยชน์ในการส่งเสริมการ
ส่งออกของผลิตภัณฑ์ขมิ้นชันของประเทศไทยไปยังประเทศทั้งใน เอเชียและยุโรปอีกด้วย

ภาควิชา.....เคมี..... ลายมือชื่อนิติ..... วรวิภา จารุไพบูลย์
สาขาวิชา.....เคมี..... ลายมือชื่ออาจารย์ที่ปรึกษา..... ณัฐชนน ลีพิพัฒน์ไพบูลย์
ปีการศึกษา.....2546..... ลายมือชื่ออาจารย์ที่ปรึกษาร่วม.....

447 24311 23: MAJOR CHEMISTRY

KEY WORDS: ORGANOCHLORINE PESTICIDES / *Curcuma longa* /

SAMPLE PREPARATION TECHNIQUES

SANITRA JARUPAIBOON: SAMPLE PREPARATION TECHNIQUES
FOR DETERMINATION OF ORGANOCHLORINE PESTICIDES IN
Curcuma longa. THESIS ADVISOR: Assist. Prof. NATCHANUN
LEEPIPATPIBOON, Dr.rer.nat, 160 pp. ISBN 974-17-5396-9

The purpose of this research is to develop a suitable sample preparation technique to analyse and determine the organochlorine pesticides in *Curcuma longa*. that contains colouring compounds and volatile oils which cause errors in the analysis of organochlorine pesticides in *Curcuma longa*. by using the GC- μ ECD. A multiresidue method for the determination of the 17 organochlorine pesticides was carried out by solid phase extraction (SPE) after extracting the sample with a mixture of hexane : dichloromethane (5:2). The extraction solutions were further cleaned up using mixed mode SPE that contained silica gel, florisil and anhydrous sodium sulfate. The method has been validated and achieved quantitative analysis down to their maximum residue limit. The linearity range from 1 – 500 ng/mL, method detection limit is in the range of 0.5 – 15.4 ng/g and its method quantitation limit is in the range of 1.4 – 51.2 ng/g. Intra – assay precision, intermediate precision and relative standard deviation were less than 5.5% for all compounds at 5, 25 and 125 ng/g. Recoveries obtained were generally in the range of 60.33 to 180.64%. The method was used to test in 3 Thai markets and 3 commercially – packed samples. All of them were contaminated with DDT and metabolite of DDT in low concentrations and not more than MRLs of USP regulations. The approach can be used as a standard method for quantitative analysis of organochlorine pesticides in *Curcuma longa*. Moreover, it can help boost the export of Thai *Curcuma longa*. to both Asian and European countries.

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Academic year.....2003..... Co-advisor's signature.....

ACKNOWLEDGEMENTS

First of all, I would like to express my deepest gratitude to my advisor, Assistant Professor Natchanun Leepipatpiboon, Dr.rer.nat. for her professionalism, discerning suggestions, forbearance, encouragement, and critical reading. My greatest thanks also go to the Department of Biology and National Research Center for Environmental and Hazardous Waste Management, Chulalongkorn University who financially supported for the GC instruments and we would like to express our research to Bureau of Quality and Safety of Food, Department of Medical Science who financially support for this thesis. My special thanks should also extend to the thesis committee who painstakingly gave their invaluable comments.

Furthermore, I would like to specially thank all members of the Separation and Chromatography Research Group for their invaluable suggestions. Moral supports and cheerful willingness of my friends are truly appreciated.

Finally, I am eternally grateful to my beloved parents and all my dearest family members for their enormously ever-willing support. Thanks are also extended to everyone who has contributed to the success of this thesis.

ศูนย์วิทยทรัพยากร
จุฬาลงกรณ์มหาวิทยาลัย

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 จุฬาลงกรณ์มหาวิทยาลัย

LIST OF ABBREVIATIONS AND SYMBOLS

<i>Curcuma Longa.</i>	Curcumin, Turmeric
MRMs	Multi residue methods
SRMs	Single residue methods
AOAC	Association of Official Analytical Chemists
USP	United States of Pharmacopeial Convention, Inc.
CDFA	California Department of Food and Agriculture
GC	Gas Chromatography
μ -ECD	micro – Electron Capture Detector
HPLC	High Performance Liquid Chromatography
TID	Thermoionic Detector
NPD	Nitrogen Phosphorous Detector
FPD	Flame Photometric Detector
FID	Flame Ionization Detector
GPC	Gel Permeation Chromatography
ELCD	Hall Electrolytic Conductivity Detector
MSPD	Matrix Solid – Phase Dispersion
MS	Mass Spectrometer Detector
MIP	Molecularly Imprinted Polymer
AED	Atomic Emission Detector
SIM	Selective Ion Monitoring
IA	Immunoassay
OCPs	Organochlorine Pesticides
OPPs	Organophosphate Pesticides
α -BHC, HCH	alpha-1,2,3,4,5,6-Hexachlorocyclohexane
β -BHC, HCH	beta-1,2,3,4,5,6-Hexachlorocyclohexane
γ -BHC, HCH	gamma-1,2,3,4,5,6-Hexachlorocyclohexane, lindane

Heptachlor	1,4,5,6,7,10,10-heptachloro-4,7,8,9-tetrahydro-4,7-endomethyleneindene
Alachlor	2-chloro-2',6'-diethyl-N-(methoxymethyl)acetanilide
Aldrin	1,2,3,4,10,10-hexachloro-1,4,4a,5,8,8a-hexahydro-1,4:5,8-Dimethanonaphthalene
O,P'-DDE	1,1-Dichloro-2-(o-chlorophenyl)-2-(p-chlorophenyl) ethylene
α -endosulfan	6,7,8,9,10,10-hexachloro-1,5,5a,6,9,9a-hexahydro-6,9-methano-2,4,3-benzodioxathiepin 3-oxide
Dieldrin	1,2,3,4,10,10-Hexachloro-6,7-epoxy-1,4,4a,5,6,7,8,8a-octahydro-1,4-endo-exo-5,8-dimethanonaphthalene
P,P'-DDE	1,1-Dichloro-2,2-bis(p-chlorophenyl)ethylene; 2,2-bis(p-chlorophenyl)-1,1-dichloroethene
O,P'-DDD	1,1-Dichloro-2,2-bis(2,4'-dichlorophenyl)ethane
Endrin	1,2,3,4,10,10-hexachloro-6,7-epoxy-1,4,4a,5,6,7,8,8a-octahydro-1,4-endo-endo-5,8-dimethano-naphthalene
β -endosulfan	6,7,8,9,10,10-hexachloro-1,5,5a,6,9,9a-hexahydro-6,9-methano-2,4,3-benzodioxathiepin 3-oxide
P,P'-DDD	1,1-Dichloro-2,2-bis(p-chlorophenyl)ethane
O,P'-DDT	1-(2-Chlorophenyl)-1-(4-chlorophenyl)-2,2,2-trichloroethane
Carbophenothion	p-chlorophenylmercaptomethyl dithiophosphate
Endosulfan-sulfate	6,7,8,9,10,10-hexachloro-1,5,5a,6,9,9a-hexahydro-6,9-Methano-2,4,3-benzodioxathiepin
P,P'-DDT	1,1,1-Trichloro-2,2-bis(p-chlorophenyl)ethane
Methoxychlor	1,1,1-trichloro-2,2-bis(p-methoxyphenyl)ethane
ppm	part per million
ppb	part per billion
mL	milliliter (s)
g	gram (s)
mm	milliliter
μ m	micrometer
nm	nanometer
i.d.	internal diameter
r^2	correlation coefficient

LOD	Limit Of Detection
LOQ	Limit of Quantitation
MDL	Method Detetion Limit
SQL	Method Quantification Limit



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