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HOLLOW FIBER LIQUID PHASE MICROEXTRACTION FOR THE
DETERMINATION OF GLYPHOSATE AND AMINOMETHYLPHOSPHONIC ACID

Miss Montra Piriya Pittaya

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for the Degree of Master of Science Program in Chemistry

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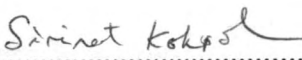
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By Montra Piriypittaya
Field of Study Chemistry
Thesis Advisor Assistant Professor Natchanun Leepipatpiboon, Dr.,rer.nat
Thesis Co-advisor Assistant Professor M.L. Siripastr Jayanta

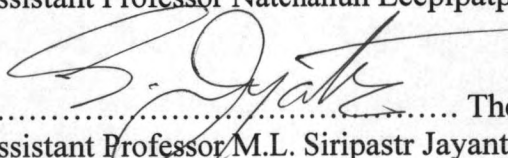
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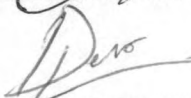
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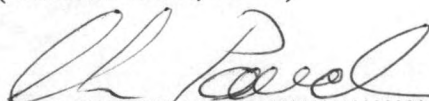
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(Associate Professor Sirirat Kokpol, Ph.D.)

..... Thesis Advisor
(Assistant Professor Natchanun Leepipatpiboon, Dr.,rer.nat)

..... Thesis Co-advisor
(Assistant Professor M.L. Siripastr Jayanta)

..... Member
(Luxana Dubas, Ph.D.)

..... Member
(Associate Professor Ura Pancharoen, D.Eng., Sc.D)

มนตรา พิริยะพิทยา: การสกัดระดับจุลภาคในเฟสของเหลวด้วยเส้นใยกลวงสำหรับการวัดไกลโฟเซตและกรดอะมิโนเมทิลฟอสฟอนิก. (HOLLOW FIBER LIQUID PHASE MICROEXTRACTION FOR THE DETERMINATION OF GLYPHOSATE AND AMINOMETHYPHOSPHONIC ACID) อ.ที่ปรึกษา: ผศ. ดร. ณัฐชนันต์ ลิปิพัฒน์ไพบูลย์, อ.ที่ปรึกษาร่วม: ผศ. มล. สิริพัศตร์ ไชยันต์ 88 หน้า.

เทคนิคการสกัดระดับจุลภาคด้วยเส้นใยกลวงเหมาะสำหรับการวิเคราะห์สารกำจัดวัชพืชไกลโฟเซตและสารอนุพันธ์หลักกรดอะมิโนเมทิลฟอสฟอนิก เมื่อใช้ Aliquat 336 เป็นสารตัวพา ประจุบวกเข้มข้น 0.20 โมลาร์ ในตัวทำละลายไดเอทิลอีเทอร์ ทำหน้าที่เป็นเฟสของเหลวพวยอยู่ในเส้นใย และใช้สารละลายโพแทสเซียมคลอไรด์ 1.0 โมลาร์ 20.0 ไมโครลิตรบรรจุในเส้นใยกลวง นำไปแช่ในสารละลายตัวอย่างปริมาตร 20.0 มิลลิลิตร ปรับพีเอชให้เป็น 9.0 ทิ้งไว้ 60 นาที จากนั้นทำวิเคราะห์ปริมาณในเฟสดัวยับ ด้วยโครมาโทกราฟีแบบของเหลวสมรรถนะสูงที่เชื่อมต่อกับระบบเปลี่ยนอนุพันธ์ผ่านคอลัมน์แยก การสกัดด้วยวิธีนี้ให้ค่าแฟกเตอร์การเพิ่มความเข้มข้นถึง 814 เท่า และ 141 เท่า สำหรับไกลโฟเซตและกรดอะมิโนเมทิลฟอสฟอนิกตามลำดับ จัดจำกัดค่าสุดของการวิเคราะห์ไกลโฟเซตและกรดอะมิโนเมทิลฟอสฟอนิกในน้ำมีค่า 0.3 และ 1.5 ไมโครกรัมต่อลิตร ตามลำดับ สำหรับค่าเบี่ยงเบนมาตรฐานสัมพัทธ์ของไกลโฟเซตและกรดอะมิโนเมทิลฟอสฟอนิกที่ความเข้มข้น 5 ไมโครกรัมต่อลิตร (ทำซ้ำ 10 ครั้ง) อยู่ที่ร้อยละ 11.86 และ 7.33 ตามลำดับ เทคนิคเยื่อของเหลวพวยที่ใช้สารตัวพาเป็นสื่อกลางแสดงให้เห็นถึงความสามารถในการเพิ่มความเข้มข้นของสารของการสกัด มีความเที่ยงสูง ข้อดีของเทคนิคการสกัดแบบนี้คือ ใช้ตัวทำละลายอินทรีย์ในปริมาณน้อยมาก ราคาถูก ง่าย ไม่มีอันตรายต่อสิ่งแวดล้อม และเทคนิคนี้สามารถนำมาใช้กับการตรวจหาสารทั้งสองชนิดในน้ำได้ดังนี้

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KEY WORD: SUPPORTED LIQUID MEMBRANE/ HOLLOW FIBER MEMBRANE/ GLYPHOSATE/ AMINOMETHYPHOSPHONIC ACID/ ALIQUAT 336/ CARRIER-MEDIATE

MONTRA PIRIYAPITTAYA: HOLLOW FIBER LIQUID PHASE MICROEXTRACTION FOR THE DETERMINATION OF GLYPHOSATE AND AMINOMETHYPHOSPHONIC ACID. THESIS ADVISOR: ASST. PROF. NATCHANUN LEEPIPATPIBOON, Dr.,rer.nat, THESIS CO-ADVISOR: ASST. PROF. M.L. SIRIPASTR JAYANTA, 88 pp.

A hollow fiber based microextraction suitable for the determination of the herbicide glyphosate and its main metabolite aminomethylphosphoric acid was developed. A solution of 0.20 M Aliquat 336, a cationic carrier, in di-n-hexyl ether was used as the membrane support liquid. The membrane lumen was filled with 20.0 µL of 1.0 M potassium chloride before dipping in a 20.0-mL of sample solution adjusted to pH 9.0. After a 60-minute extraction, the acceptor phase was analyzed by high-performance liquid chromatography with post-column derivatization. The enrichment factors obtained for glyphosate and aminomethylphosphonic acid were 814 and 141, respectively. The method detection limits were 0.3 µg/L for glyphosate and 1.5 µg/L for aminomethylphosphonic acid. Percent relative standard deviations (n=10) of glyphosate and aminomethylphosphonic acid at 5 µg/L spiked level, were 11.86 and 7.33%, respectively. In general, the developed carrier-mediated supported-liquid membrane technique demonstrated high enrichment capability with good reproducibility. The technique consumed very little organic solvent, inexpensive, simple, and environmental friendly. The method was tested with ground water samples and proved to be feasible for the determination of both analytes in ground water.

Department.....Chemistry..... Student's signature..... Montra Piriypittaya.....
Field of study.....Chemistry..... Advisor's signature..... Natchanun Leepipatpiboon.....
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LIST OF ABBREVIATIONS AND SYMBOLS

°C	degree centigrade
%RSD	percent relative standard deviation
α	fractional composition
μg	microgram (s)
μL	microliter (s)
μm	micrometer (s)
ADI	acceptable daily intake
AMPA	aminomethylphosphonic acid
AOAC	Association of Analytical Communities
CE	capillary electrophoresis
cm	centimeter (s)
DAG	diacetonketogulonic acid
DEHPA	diethylhexyl phosphoric acid
EDTA	ethylenediamine tetracetate
EE	extraction efficiency
EF	enrichment factor
EPA	U.S. Environmental Protection Agency
EPSP	5-Enolpyruvylshikimic acid-3-phosphate synthase
FAO	Food and Agricultural Organization
FLD	fluorescence detector
FMOC-Cl	9-fluorenylmethyl chloroformate
FPD	flame photometric detector
g	gram (s)
GC	gas chromatography
GC-MS	gas chromatography-mass spectrometry
HF-LPME	hollow fiber-liquid phase microextraction
HFM	hollow fiber membrane
HPLC	high performance liquid chromatography

HPLC-ESIMS	high performance liquid chromatography-electrospray ionization mass spectrometry
HPLC-MS	high-performance liquid chromatography mass spectrometry
K_a	soil adsorption coefficient
KCl	potassium chloride
K_{ex}	equilibrium constant
kg	kilogram (s)
L	liter (s)
LLE	liquid-liquid extraction
$\log K_{ow}$	octanol-water coefficient
Lu	light Unit
M	molar
m^3	cubic meter (s)
MCL	maximum contaminant level
MDL	method detection limit
mg	milligram (s)
min	minute (s)
mL	milliliter (s)
mm	millimeter (s)
MMLLE	microporous membrane liquid liquid extraction
MQL	method quantitation limit
ng	nanogram (s)
NPD	nitrogen phosphorous detector
OPA	<i>o</i> -phthalaldehyde
PP	polypropylene
ppb	part per billion
ppm	part per million
PTFE	polytetrafluoroethylene
PVDF	polyvinylidene difluoride
R^2	correlation coefficient
S/N	signal to noise
SD	standard deviation
SLM	supported liquid membrane
SPE	solid-phase extraction

TFAA	trifluoroacetic anhydride
TFE	trifluoroethanol
TOPT	tri-octyl phosphine oxide
UV	ultraviolet
vis	visible
WHO	World Health Organization
wt/v	weight by volume