

TABLE OF CONTENTS

	page
Title page	i
Approval page	ii
Acknowledgments	iii
Abstract	iv
List of tables	xi
List of figures	xiii
Abbreviations and Symbols	xv
Chapter 1 INTRODUCTION	
1.1 Methamphetamine/Amphetamine	1
1.2 Diagnosis of MA abuse	5
1.3 Hair analysis	6
1.4 Solid-phase microextraction (SPME)	11
1.5 Gas chromatography–mass spectrometry	13
1.6 Method validation	16
1.7 The scope and aims of this research	18
Chapter 2 MATERIALS AND METHODS	
2.1 Apparatus and chemicals	19
2.1.1 Apparatus	19
2.1.2 Chemicals	20
2.2 Preparation of solutions	21
2.2.1 Preparation of standard solutions	21
2.2.1.1 Stock standard solutions	21

ลิขสิทธิ์มหาวิทยาลัยเชียงใหม่

Copyright © by Chiang Mai University
All rights reserved

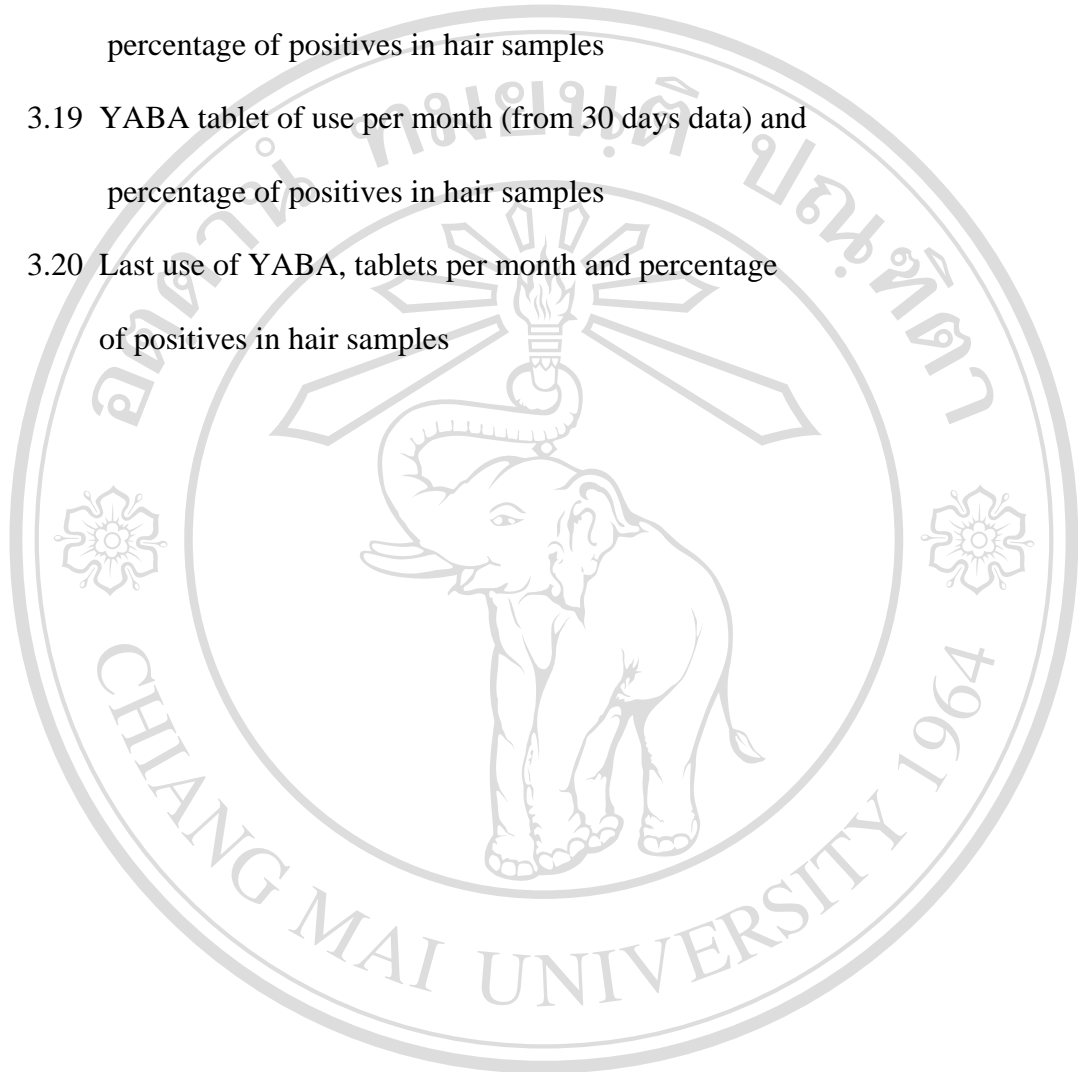
2.2.1.2 Working standard solutions	21
2.2.1.3 Internal standard solution	21
2.2.2 Potassium carbonate	22
2.2.3 Hydrochloric acid	22
2.3 GC–MS Conditions	22
2.4 Optimization of SPME conditions	23
2.4.1 Optimization of total volume of preparation	23
2.4.2 Optimization of potassium carbonate concentration	23
2.4.3 Comparison of SPME fibers	24
2.4.4 Optimization of incubation time	24
2.4.5 Optimization of extraction time	24
2.4.6 Optimization of desorption time	25
2.4.7 Determination of carry over effect	25
2.5 Validation of the analytical method by HS-SPME-GC-MS	25
2.5.1 Linearity	26
2.5.2 Accuracy and precision	26
2.5.3 Limit of detection and limit of quantitation	27
2.6 Analysis of hair samples from drug abusers	28
2.6.1 Subjects	28
2.6.2 Sample preparation	28
2.6.3 Analytical procedure	29
2.7 Statistical analysis	29
Chapter 3 RESULTS	
3.1 Suitable GC-MS conditions for determination of AM and MA	30

3.1.1 GC-MS conditions	30
3.2 Optimization of SPME conditions	38
3.2.1 Optimization of total volume of solution preparation	38
3.2.2 Optimization of potassium carbonate concentration	39
3.2.3 Comparison of SPME fibers	40
3.2.4 Optimization of incubation time	42
3.2.5 Optimization of extraction time	43
3.2.6 Optimization of desorption time	44
3.2.7 Determination of carry over effect	45
3.2.8 Optimal condition for SPME in this experiment	45
3.3 Method validation	46
3.3.1 Linearity	46
3.3.2 Accuracy and precision	46
3.3.3 Limit of detection and limit of quantitation	47
3.4 Analysis of hair samples from drug abusers	52
3.4.1 Comparison of drug abuse history and the hair analysis for MA	53
3.4.2 Correlation between MA concentration in hair samples and the number of YABA tablets used	59
Chapter 4 DISCUSSION AND CONCLUSION	
4.1 Discussion	62
4.2 Conclusion	67
RERERENCES	68
APPENDIX	76
VITA	79

LIST OF TABLES

Table	page
1.1 Physical properties of MA	2
2.1 Working standard solution preparation	21
3.1 GC-MS parameter	30
3.2 Retention time and selected ion monitoring of amphetamine derivatives and ketamine	34
3.3 Comparison of different total volume	38
3.4 Comparison of potassium carbonate concentration	39
3.5 Comparison of PDMS and PDMS/DVB fibers on amphetamine derivatives and ketamine analysis	40
3.6 Comparison of incubation time	42
3.7 Comparison of extraction time	43
3.8 Comparison of desorption time	44
3.9 Determination of carry over effect	45
3.10 Optimal condition for SPME	45
3.11 Linearity and correlation coefficient of AM	48
3.12 Linearity and correlation coefficient of MA	48
3.13 Accuracy and precision of AM and MA	50
3.14 Limit of detection and limit of quantitation of AM and MA	50
3.15 Descriptive analysis of YABA abuse history in the study group	54
3.16 Analysis of hair samples in 3 sections	56

3.17 Last use of YABA and percentage of MA positives in hair samples	56
3.18 YABA tablet use per month (from 3 months data) and percentage of positives in hair samples	57
3.19 YABA tablet of use per month (from 30 days data) and percentage of positives in hair samples	57
3.20 Last use of YABA, tablets per month and percentage of positives in hair samples	58



ลิขสิทธิ์มหาวิทยาลัยเชียงใหม่
Copyright© by Chiang Mai University
All rights reserved

LIST OF FIGURES

Figure	page
1.1 AM and MA structure	2
1.2 Metabolic pathway of MA	4
1.3 Diagram illustrating hair growth sequences	8
1.4 Possible model for drug incorporation into hair	9
1.5 Schematic view of the headspace SPME apparatus	12
1.6 The GC-MS system	15
1.7 Schematic view of the ion source based on electron impact ionization and the quadrupole mass filter typically found in a GC-MS instrument	15
1.8 Electron Impact Ionization	16
3.1 Scan mode chromatogram of mixed standard solutions of amphetamine derivatives and ketamine using HS-SPME-GC-MS	31
3.2 Scan mode chromatogram of AM standard and mass spectrum of AM by using scan mode	32
3.3 Scan mode chromatogram of MA standard and mass spectrum of MA by using scan mode	33
3.4 SIM mode chromatogram of mixed standard solutions of amphetamine derivatives and ketamine using HS-SPME-GC-MS	35
3.5 SIM mode chromatogram of AM standard and mass spectrum of AM by using SIM mode	36

3.6 SIM mode chromatogram of MA standard and mass spectrum of MA by using SIM mode	37
3.7 Comparison of different total volumes of preparation	38
3.8 Comparison of potassium carbonate concentration	39
3.9 SIM chromatogram of mixed standard solutions of amphetamine derivatives and ketamine using PDMS and PDMS/DVB fiber with HS-SPME-GC-MS	41
3.10 Comparison of incubation time	42
3.11 Comparison of extraction time	43
3.12 Comparison of desorption time	44
3.13 Linearity of AM	49
3.14 Linearity of MA	49
3.15 Overlay chromatogram of AM at 1.5, 2.0, 2.5 ng/mg and negative hair	51
3.16 Overlay chromatogram of MA at 0.25, 0.3, 0.5 ng/mg and negative hair	51
3.17 Chromatogram of an MA negative hair sample	55
3.18 Chromatogram of an MA positive hair sample	55
3.19 Correlation between the concentration in section 1 of hair and the number of YABA tablets used per month from 30 days data	59
3.20 Correlation between the concentration in section 1 of hair and the number of YABA tablets used per month from 3 months data	60
3.21 Correlation between the concentration in section 2 of hair and the number of YABA tablets used per month	60
3.21 Correlation between the concentration in section 3 of hair and the number of YABA tablets used per month	61

ABBREVIATIONS AND SYMBOLS

AM	amphetamine
CV	coefficient of variation
EI	electron ionization
eV	electron volts
GC	gas chromatography
GC-MS	gas chromatography-mass spectrometry
h	hour
HP-5MS	95% dimethyl-5% diphenylpolysiloxane
HS	headspace
INS	internal standard
LOD	limit of detection
LOQ	limit of quantitation
MA	methamphetamine
MDA	methylenedioxyamphetamine
MDMA	methylenedioxymethamphetamine
MDE	methylenedioxyethylamphetamine
MeOH	methanol
mg	milligram
min	minute
ml	milliliter
MS	mass spectrometry

MW	molecular weight
m/z	mass to charge ratio
ng	nanogram
No	number
RIA	radioimmunoassay
RR	relative recovery
RT	retention time
r^2	correlation coefficient
SIM	selected ion monitoring
SPME	solid phase microextraction
TIC	total ion chromatogram
μg	microgram
μl	microliter
$^{\circ}\text{C}$	degrees Celsius

ลิขสิทธิ์มหาวิทยาลัยเชียงใหม่
 Copyright© by Chiang Mai University
 All rights reserved