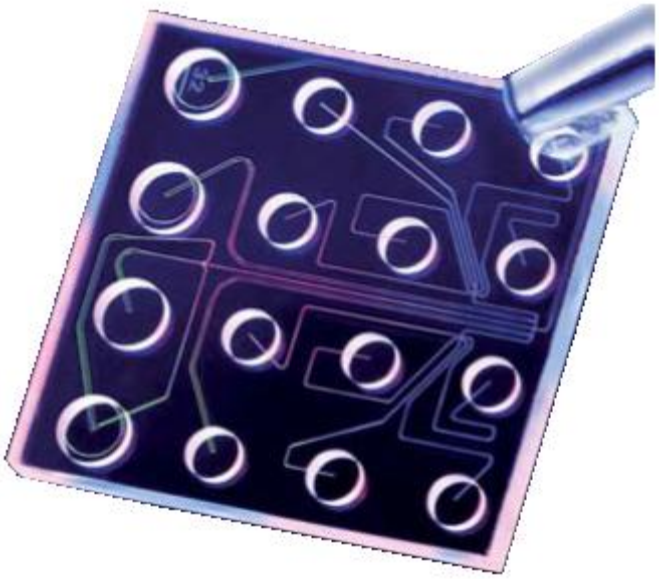
The analysis of amphetamine-type stimulants using microchip capillary electrophoresis

Aimee Lloyd



A thesis submitted for the

Degree of Doctor of Philosophy (Science)

University of Technology, Sydney

July, 2013

Simplicity is the ultimate sophistication

Leonardo da Vinci

Certificate of authorship and originality

I certify that the work in this thesis has not previously been submitted for a degree nor has it been submitted as part of the requirements for a degree except as fully acknowledged in the text.

I also certify that the thesis has been written by me. Any help I received in my research work and the preparation of the thesis itself has been acknowledged. In addition, I certify that all the information sources and literature used are indicated in the thesis.

Aimee Lloyd

31st July 2013

Acknowledgements

Many people have supported and guided me throughout this mammoth project. It has been an incredible journey and I would like to thank each and every one of you. The insightful discussions, constructive advice and feedback has greatly influenced my personal and intellectual development and opened my mind to new ideas. I am grateful for the funding sources that allowed me carry out this research and attend various meetings and conferences.

To my supervisors, Claude Roux and Philip Doble, thank you for your invaluable advice, patience and support throughout my thesis. I am indebted to you both for giving me the chance to embark on such an exciting research project and for the unique opportunities along the way. Claude, my sincere appreciations go to you for your understanding and ongoing support despite your many other commitments. You always believed in me and encouraged me to shape my own interests and ideas, not only with my project but in Forensic Science. Philip, the insightful advice and discussions you imparted on me have enabled me to grow intellectually. Thank you for persuading me to pursue a PhD and guiding me through my research.

A big thank you to Lucas, my mentor, for motivating me throughout my postgraduate studies; you inspired me to develop as a researcher. I admire your positive outlook and ability to smile despite the situation. As an honorary supervisor, you were always keen to lend a hand - even extending your expert technical services all the way to NZ.

To the illicit drug and clan lab teams of ESR, in particular Matthew Russell, I am really appreciative of the invaluable opportunity to research in collaboration with you. Matt, thank you for your generous support; I have greatly benefited from your interest and commitment to my field work studies.

To Pierre Esseiva, Pierre Margot, Oliver Ribaux and colleagues of the University of Lausanne, I am forever indebted to you all for the opportunity to grow and gain insight into Forensic Science in Switzerland. Thank you to the Australian Government

for awarding me with an Endeavour Postgraduate Award to make this exchange possible and to Agilent Technologies for providing and transporting the equipment.

To my fellow postgraduate students and support staff of whom I had the pleasure of working with. Thank you for providing such a positive and enjoyable research environment; I will always have great memories from our excursions to conferences and beyond. A special thanks to Marie for being such a caring and dependable friend – our morning coffees, mixed with advice and suggestions, got me through the tough times.

To my loving boyfriend Nic, thank you for believing in me and keeping me positive – especially during the latter stages. You inspired me to get over the final hurdles and always took time to cheer me up. Thank you for your patience and the hours you spent proof-reading drafts of my thesis.

Last but not least, to my Mum, Dad and sister Hannah: you have always supported, encouraged and believed in me in all my endeavours. Your guidance and persistent help has enabled me to realise my full potential. This thesis would not have been possible without your unconditional love and support.

Table of contents

List of figures

List of tables

Abbreviations

AFP	Australian Federal Police
AIC	Australian Institute of Criminology
AMP	amphetamine
ATR-FTIR	attenuated total reflection – fourier transform infrared
ATS	amphetamine-type stimulants
CE	capillary electrophoresis
CMC	critical micellar concentration
CNS	central nervous system
CZE	capillary zone electrophoresis
DNA	deoxyribonucleic acid
DTAF	5-([4,6-dichlorotriazin-2-yl]amino)fluorescein
ED	electrochemical detection
EMCDDA	European Monitoring Centre for Drugs and Drug Addiction
EOF	electroosmotic flow
ESI	electrospray ionisation
ESR	institute of environmental science and research limited
FITC	fluorescein isothiocyanate isomer I

FTIR	fourier transform infrared
GC	gas chromatography
HPLC	high performance liquid chromatography
Hypo	hypophosphorus
IMS	ion mobility spectrometry
LC-MS	liquid chromatography-mass spectrometry
LED	light-emitting diode
LIF	laser-induced fluorescence
LOC	lab-on-a-chip
LOD	limit of detection
LOQ	limit of quantification
MA	methamphetamine
MCE	microchip capillary electrophoresis
MDA	3,4-methylenedioxyamphetamine
MDEA	3,4-methylenedioxyethylamphetamine
MDMA	3,4-methylenedioxymethamphetamine
3,4-MDP-2-P	3,4-methylenedioxyphenyl-2-propanone
MDPBP	3’,4’-methylenedioxy-α-pyrrolidinobutiophenone

4-MEC	4-methylethcathinone
MEKC	micellar electrokinetic chromatography
4-MMC	4-methylmethcathinone
MS	mass spectra
MS	mass spectrometry
NFSTC	National Forensic Science Technology Centre
NIR	near infra-red
OPA	o-phthalaldehyde
P2P	phenyl-2-propanone
PSE	pseudoephedrine
RSD	relative standard deviation
SDS	sodium dodecyl sulfate
SWGDRUG	scientific working group for the analysis of seized drugs
µ-TAS	µ-total analytical systems
TLC	thin layer chromatography
UN	United Nations
UNODC	United Nations Office on Drugs and Crime
β-PEA	β-phenethylamine

Abstract

The illicit drug trade, dominated by sophisticated trans-national criminal organisations, has put increasing demands on law enforcement bodies. Timely information concerning illegal activity is required to effectively combat the illicit drug problem. Rapid, if not real-time, identification tools would help direct investigators with sampling procedures and safety precaution measures at drug-related crime scenes. In addition to enhancing work-flow processes, for example the creation of *rapid laboratories* or intelligence units, a major focus rests on the miniaturisation of existing analytical techniques, predominantly spectroscopic-based, in order to create field portable tools for this purpose. Currently available techniques such as colour tests, Raman and infra-red spectrometers often have limitations associated with specificity, portability and sample preparation requirements. The diverse nature of exhibits present challenges for the in-field detection of controlled drugs and pre-cursors.

An emerging area of research, lab-on-a-chip (LOC), with its ability to integrate multiple functions on a microchip, has shown promising applications for in-field testing. The aim of this project was to evaluate a commercial portable microchip capillary electrophoresis (MCE) platform, the Agilent Bioanalyzer 2100, for the analysis of amphetamine-type stimulants (ATS). This device, although designed for the analysis of biological molecules, holds significant potential for the analysis of inorganic ions, explosives and illicit drugs. This project focused on developing and optimising a rapid, simple and inexpensive separation method. The method was adapted for the analysis of a wide range of casework exhibits including liquids, tablets and powders in order to test its in-field capabilities. The prospects, challenges and applications are discussed. This research has highlighted MCE as a competitive platform for the screening of ATS and has demonstrated its potential use in forensic drug analysis.

List of publications and presentations

Lloyd, A., Russell, M., Blanes, L., Doble, P. and Roux, C. Lab-on-a-chip screening of methamphetamine and pseudoephedrine in samples from clandestine laboratories. *Forensic Science International*. 2013; 228(1-3): 8-14.

Lloyd, A., Blanes, L., Beavis, A., Roux, C. and Doble, P. A rapid method for the in-field analysis of amphetamines employing the Agilent Bioanalyzer. *Analytical Methods*. 2011; 3(7): 1535-1539.

Lloyd, A., Russell, M., Somerville, R., Doble, P. and Roux, C. The use of portable microchip electrophoresis for the screening and comparative analysis of synthetic cathinone seizures. Submitted to *Forensic Science International (manuscript ID: FSI-S-13-01367).*

Lloyd, A., Russell, M., Blanes, L., Doble, P. and Roux, C. Rapid screening for pseudoephedrine and methamphetamine in clandestine laboratory samples using the Agilent 2100 Bioanalyzer [oral presentation]. 2012; *The 21st International Symposium on the Forensic Sciences of the Australian and New Zealand Forensic Science Society (ANZFSS) Hobart, Tasmania, Australia.*

Lloyd, A., Doble, P., Roux, C., Esseiva, P. and Delémont, O. Comparison between microchip capillary electrophoresis and capillary electrophoresis-mass spectrometry for the detection of amphetamines [oral presentation]. 2011; *19th International Association of Forensic Sciences (IAFS) world meeting, Funchal, Madeira, Portugal.*

Lloyd, A., Blanes, L., Beavis, A., Roux, C. and Doble, P. Analysis of amphetamine analogues using the portable Bioanalyzer 2100 lab-on-a-chip [poster presentation]. 2010; *17th International Symposium on Capillary electroseparation techniques (ITP 2010), Baltimore, Maryland, United States.*

Lloyd, A., Blanes, L., Beavis, A., Roux, C. and Doble, P. Analysis of amphetamine-type stimulants using the portable Bioanalyzer 2100 lab-on-a-chip [poster presentation].

2010; *The 20th International Symposium on the Forensic Sciences of the Australian and New Zealand Forensic Science Society (ANZFSS), Sydney, NSW, Australia*.