Explosive Residue Analysis: Evaluation and Optimisation of Sampling, Storage and Cleanup Protocols

Nopporn Song-im

A thesis submitted for the degree of Doctor of Philosophy in Applied Science

University of Canberra

June 2011

ABSTRACT

For the detection and identification of explosive residues, surfaces of interest are typically sampled by swabbing or solvent wash. The current use of sequential swabbing protocols that separately target organic and inorganic compounds is under threat due to the increased use of improvised organic/inorganic explosive mixtures. An optimised swabbing procedure, using commercially available skin cleansing alcohol wipes, was developed to maximize the collection of both organic and inorganic explosive residues in a single step. Using six representative compounds (TNT, RDX, PETN, TATP, ammonium nitrate, and sodium chlorate) and four different substrates (glass, plastic, aluminium foil and laminate), the collection efficiency of the alcohol wipes was compared to the results obtained using conventional cotton swabs, polyester swabs, and a direct solvent wash (used as a control). The alcohol wipes demonstrated better overall performance in the recovery of both the organic and inorganic compounds from all test surfaces.

The compositions of a mixture of three organic solvents (acetone, acetonitrile and methanol) and water for a single-step solvent extraction of both organic and inorganic representative compounds from the alcohol wipes was investigated. In addition, the study included the evaluation and optimisation of a subsequent solid-phase extraction (SPE) clean-up procedure for the wipes extracts utilising several solid-phase extraction cartridges (both commercially available and prepared in-house).

The application of a polyester alcohol wipe as a universal swab, followed by extraction with 60% v/v methanol/water and clean-up with a Nexus SPE cartridge, was proposed as the final optimised protocol for the combined recovery and clean-up of organic and inorganic explosive residues.

The stability of the six representative compounds on polyester wipes and in 60% v/v methanol/water extracts, stored over 30 days in clear and amber glass vials at three different temperatures, was assessed in order to establish storage recommendations in conjunction with the final proposed protocol. The retention of all six target compounds on a glass surface at two different storage temperatures was included for an estimation of the maximum time that the explosive material could still be detected and recovered from a stored exhibit.

The results from the stability study suggested that, after sampling, the wipes should be stored in a dark and low temperature environment. Also, after processing using the proposed protocol, the extracts should be stored in a similar fashion. The results from the retention study of the six target compounds on the glass substrate suggested that exhibits should be stored at the lowest temperature possible to minimise the loss of any TNT or TATP (or similar target compounds) that might be contained within the sample.

ACKNOWLEDGEMENTS

Completion of this research would not have been possible without the significant assistance and outstanding support of a number of individuals and organizations.

I owe enormous thanks to my great supervisors, Professor Chris Lennard and Dr Sarah Benson. Without the support of my application to the university from Chris, and the initial thoughts of Sarah to outline the scope of the research, this thesis would not exist and my passion to gain expertise in the field of forensic chemistry would have come to an abrupt end. Thank you very much for your encouragement, guidance and your effort to review this thesis within a short period of time. It has been a privilege to be your student.

I must thank my parents for their absolute support in all the endeavours I have chosen.

I would like to acknowledge the support from Forensic and Data Centres at the Australian Federal Police (AFP), and the assistance and advice given by the staff of the Chemical Criminalistics team at the AFP.

Thanks are due to technical staff, both past and present, at the Faculty of Applied Science, University of Canberra, especially to Melissa Clark, Richard Carne and Dr Ruben Ramirez. Without their help, this research would not have been completed on time.

As a first time user of capillary electrophoresis, my special thanks go to Dr Cameron Jones at the University of Tasmania for his guidance regarding technical issues, and to Dr Tamsin Kelly for access to the instrument.

Cheers to my colleagues at the National Centre for Forensic Studies, both past and present, for making it such a great and friendly environment. Carolyn, Sam and Aidan deserve special thanks for lending me their helping hands to collect background swabs.

During the first month in Canberra, life would not have been easy without the help from Ron Foster and also I would like to thank Dr Dennis McNevin for help in commuting to the University at that period of time. Thanks also to Nicholas Coppel and housemates at number 52 for a great living environment. Heartfelt thanks to my good friends, Dr Stephen Wild, Dr Royston Gustuvson and Dr Peter McCabe who offered advice and insights on life during the PhD and how to manage personal issues.

All work and no play make Jack a dull boy. I am extremely grateful to everyone in Sydney Roadrunners especially James and Luke who provided me an exhilarating experience at high speed every time I fled from frustrating laboratory work.

Finally, on a more personal level, I would like to express my deep appreciation for the sustaining support from two extra-special men who have been my great mates, Tom and Steve, their encouragement kept me going through all the tough times. Thank you.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

LIST OF ABBREVIATIONS

AN	Ammonium nitrate
BGE	Background electrolyte
CE	Capillary electrophoresis
DFLEX	Diffusive flammable liquid extraction device
DNB	m-Dinitrobenzene
DNN	1,3-Dinitronaphthalene
DSTL	Defence Science and Technology Laboratory, UK
EGDN	Ethyleneglycol dinitrate
FINEX	Forensic International Network for Explosives Investigation
FT-IR	Fourier transform infrared spectroscopy
GC-ECD	Gas chromatography/electron capture detector
GC-MS	Gas chromatography/mass spectrometry
GC-TEA	Gas chromatography/thermal energy analyser detector
HMTD	Hexamethylene triperoxide diamine
HMX	High melting explosive or Her Majesty's Explosive (1,3,5,7-tetranitro-1,3,5,7-tetraazacyclooctane or octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine)
HPLC	High performance liquid chromatography
IC	Ion chromatography
IMS	Ion mobility spectrometry
IR	Infrared spectroscopy
IS	Internal standard
NATA	National Association of Testing Authorities
NG	Nitroglycerine
NMR	Nuclear magnetic resonance spectrometry
NT	Nitrotoluene
PETN	Pentaerythritol tetranitrate
PTFE	Poly(tetrafluoroethylene)
RCMP	Royal Canadian Mounted Police
RDX	Research Department Explosive or Royal Demolition Explosive (1,3,5-trinitro-1,3,5-triazacyclohexane or hexahydro-1,3,5-trinitro-1,3,5-triazine)
SEM-EDX	Scanning electron microscopy/energy dispersive X-ray analysis
SFE	Supercritical fluid extraction
SPE	Solid phase extraction

SPME	Solid phase microextraction
TATP	Triacetone triperoxide
TLC	Thin-layer chromatography
TNT	2,4,6-Trinitrotoluene
TRIS	Tris(hydroxymethyl)aminomethane
XRD	X-ray diffraction
XRF	X-ray fluorescence