

Assessment of the Persistence and Transference of Inorganic Gunshot Residues

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CONTENTS

Decl	aration		v
Ackr	nowledge	ements	vi
List	of Figure	S	viii
List	of Tables		xi
List	of Abbre	viations and Symbols	. xv
Publ	ications	·	xvii
Conf	ference P	Proceedings	viii
Awa	rds and I	Honoraria	. xx
Abst	ract		.xxi
1.	INTROD	UCTION AND LITERATURE REVIEW	1
 1 1	Synor	ncic	2
1.1.			2
1.2.	Firear	m Background	3
	1.2.1.	Ammunition	3
	1.2.2.	Firearms	10
	1.2.3.	The Australian Firearms Environment	. 10
1.3.	Origir	ns of Gunshot Residue	. 13
	1.3.1.	Formation	.13
	1.3.2.	Composition	.14
	1.3.3.	Classification	.17
	1.3.4.	Primers	.19
	1.3.5.	Propellants	.21
1.4.	Meth	ods of GSR Analysis	. 26
1.5.	Sourc	es of 'GSR-like' particles	. 28
	1.5.1.	Cartridge operated industrial tools	.28
	1.5.2.	Brake pad residues	.32
	1.5.3.	Firework and other pyrotechnic residues	.36
	1.5.4.	Airbags and automotive pyrotechnic devices	.42
	1.5.5.	All sources of particles compositionally or morphologically similar to GSR	.45
1.6.	GSR T	ransfer	. 47
	1.6.1.	Primary Transfer	.48
	1.6.2.	Secondary and Further Transfer	.50
	1.6.3.	Cross-contamination	.52
1.7.	Persis	itence of GSR	. 60
	1.7.1.	General Persistence	.60
	1.7.2.	Persistence on Hands	.60
	1.7.3.	Persistence on other locations of an individual	.63
	1.7.4.	Persistence on Clothing	.64
1.8.	Statis	tical analysis of GSR evidence	. 66
	1.8.1.	GSR as Forensic Evidence	.66
	1.8.2.	Likelihood Ratios	.67

	1.8.3. 1 8 4	Bayesian Probability Bayesian Networks	68 70
1.9.	Kev R	esearch Stages	
2.01	1.9.1.	GSR distribution in the random Australian population	
	1.9.2.	Police GSR Background Survey	
	1.9.3.	Transfer during arrest	
	1.9.4.	GSR Distribution in cases of suicide	
	1.9.5.	Sub-particle morphology and composition of GSR	77
	1.9.6.	Statistical framework for the assessment of GSR evidence	78
2.	MATERI	ALS, INSTRUMENTATION, AND METHOD OPTIMISATION	
2.1.	Synop	sis	82
2.2.	Subje	ct Selection	
2.3.	Samp	e Handling	
	2.3.1.	Sample collection	
	2.3.2.	Control Samples	
	2.3.3.	Carbon Coating	
21	SEW-	:DS	88
2.7.	2/1	Instrument Operating Conditions	00
	2.4.1.	GSR Magnum Particle ID Software	90
2.5.	Syster	n Validation	
	2.5.1.	Plano Synthetic Particle Standard	
	2.5.2.	Instrument Comparison	
	2.5.3.	ENFSI Proficiency Test Performance	
	_		
2.6.	Concl	usions	109
2.6. 3.	Concl FIREARN	usions IS AND AMMUNITION ENCOUNTERED IN FORENSIC CASEWORK IN SOU	109 TH
2.6. 3. AUS	Concl FIREARN STRALIA	usions IS AND AMMUNITION ENCOUNTERED IN FORENSIC CASEWORK IN SOU	109 TH 111
2.6. 3. AUS 3.1.	Concle FIREARN GTRALIA Synop	usions IS AND AMMUNITION ENCOUNTERED IN FORENSIC CASEWORK IN SOU	109 TH 111 112
2.6. 3. AUS 3.1. 3.2.	Concle FIREARN STRALIA Synop Backg	usions IS AND AMMUNITION ENCOUNTERED IN FORENSIC CASEWORK IN SOU sis	109 TH 111 112 115
 2.6. 3. 3.1. 3.2. 3.3. 	Concle FIREARN STRALIA Synop Backg Quant	usions IS AND AMMUNITION ENCOUNTERED IN FORENSIC CASEWORK IN SOU sis round tifying gunshot residues in cases of suicide: implications for evaluation of	109 TH 111 112 115 of
2.6. 3. AUS 3.1. 3.2. 3.3. suic	Concle FIREARN STRALIA Synop Backg Quant ides and d	usions AS AND AMMUNITION ENCOUNTERED IN FORENSIC CASEWORK IN SOU sis round cifying gunshot residues in cases of suicide: implications for evaluation of criminal shootings	109 TH 111 112 115 of 117
2.6. 3. AUS 3.1. 3.2. 3.3. suic	Concle FIREARM STRALIA Synop Backg Quant ides and 3.3.1.	usions AS AND AMMUNITION ENCOUNTERED IN FORENSIC CASEWORK IN SOU asis round cifying gunshot residues in cases of suicide: implications for evaluation of criminal shootings Introduction	109 TH 111 112 115 of 117 117
2.6. 3. AUS 3.1. 3.2. 3.3. suic	Concle FIREARN STRALIA Synop Backg Quant ides and a 3.3.1. 3.3.2. 3.3.2.	Usions	109 TH 111 112 115 of 117 119 121
2.6. 3. AUS 3.1. 3.2. 3.3. suic	Concle FIREARM STRALIA Synop Backg Quant ides and 3.3.1. 3.3.2. 3.3.3. 3.3.4.	Usions	109 TH 111 112 115 of 117 117 119 121
2.6. 3. AUS 3.1. 3.2. 3.3. suic	Concle FIREARN STRALIA Synop Backg Quant ides and 3.3.1. 3.3.2. 3.3.3. 3.3.4.	Usions	109 TH 111 112 115 of 117 117 119 121 137
2.6. 3. AUS 3.1. 3.2. 3.3. suic	Concle FIREARM STRALIA Synop Backg Quant ides and 3.3.1. 3.3.2. 3.3.3. 3.3.4. Furthe	Usions	109 TH 111 112 115 of 117 117 119 121 137 140
2.6. 3. AUS 3.1. 3.2. 3.3. suic	Concle FIREARM STRALIA Synop Backg Quant ides and a 3.3.1. 3.3.2. 3.3.3. 3.3.4. Furtho 3.4.1.	Usions	109 TH 111 112 115 of 117 119 121 137 140 140
 2.6. 3. 3.1. 3.2. 3.3. suic 3.4. 3.5. 	Concle FIREARM STRALIA Synop Backg Quant ides and 3.3.1. 3.3.2. 3.3.3. 3.3.4. Furthe 3.4.1.	Usions	109 TH 111 112 115 of 117 117 119 137 137 140 143
2.6. 3. AUS 3.1. 3.2. 3.3. suic 3.4. 3.5.	Concle FIREARM STRALIA Synop Backg Quant ides and a 3.3.1. 3.3.2. 3.3.3. 3.3.4. Furtho 3.4.1. Furtho 3.5.1.	AS AND AMMUNITION ENCOUNTERED IN FORENSIC CASEWORK IN SOU Isis	109 TH 111 112 115 of 117 117 119 137 137 140 140 143 143
2.6. 3. AUS 3.1. 3.2. 3.3. suic 3.4. 3.5.	Concle FIREARM STRALIA Synop Backg Quant ides and 0 3.3.1. 3.3.2. 3.3.3. 3.3.4. Furtho 3.4.1. Furtho 3.5.1. 3.5.2.	AS AND AMMUNITION ENCOUNTERED IN FORENSIC CASEWORK IN SOU asis	109 TH 111 112 115 of 117 117 119 117 137 140 140 143 143 144
 2.6. 3. 3.1. 3.2. 3.3. suic 3.4. 3.5. 	Concle FIREARM STRALIA Synop Backg Quant ides and 3.3.1. 3.3.2. 3.3.3. 3.3.4. Furthe 3.5.1. 3.5.2. 3.5.3.	Usions	109 TH 111 112 115 of 117 117 117 137 140 140 143 143 143 145
 2.6. 3. 3.1. 3.2. 3.3. suic 3.4. 3.5. 	Concle FIREARM STRALIA Synop Backg Quant ides and 3.3.1. 3.3.2. 3.3.3. 3.3.4. Furtho 3.4.1. Furtho 3.5.1. 3.5.2. 3.5.3. 3.5.4.	AS AND AMMUNITION ENCOUNTERED IN FORENSIC CASEWORK IN SOU asis	109 TH 111 112 115 of 117 117 119 121 137 140 140 143 143 143 143 145 154
 2.6. 3. 3.1. 3.2. 3.3. suic 3.4. 3.5. 3.6. 	Concle FIREARM STRALIA Synop Backg Quant ides and 3.3.1. 3.3.2. 3.3.3. 3.3.4. Furtho 3.4.1. Furtho 3.5.1. 3.5.2. 3.5.3. 3.5.4. Concle	usions	109 TH 111 112 115 of 117 117 119 117 137 140 140 143 143 143 145 154 156
 2.6. 3. 3.1. 3.2. 3.3. suic 3.4. 3.5. 3.6. 4. 	Concle FIREARM STRALIA Synop Backg Quant ides and 3.3.1. 3.3.2. 3.3.3. 3.3.4. Furtho 3.5.1. 3.5.2. 3.5.3. 3.5.4. Concle SUB-PAR	USIONS	109 TH 111 112 115 of 117 117 117 117 117 117 117 117 117 117 115 117 115 117 115 117 115 117 117 115 117 117 117 117 117 117

4.2.	Back	ground	161
4.3.	Evalu	ation of the Sub-Surface Morphology and Composition of Gunshot Re	sidue
usin	g Focuss	ed Ion Beam Analysis	164
	4.3.1.	Introduction	164
	4.3.2.	Materials and methods	166
	4.3.3.	Results and Discussion	
	4.3.4.	Conclusions	
4.4.	Furth	er Exploration of the Weapon Memory Effect	184
	4.4.1.	Background	
	4.4.2.	Methods	
	4.4.3.	Results and Discussion	
	4.4.4.	Conclusions	
4.5.	FIB Pi	reparation for Alternate Analysis Techniques	190
	4.5.1.	Background	
	4.5.2.	Method	
	4.5.3.	Results and Discussion	
	4.5.4.	Conclusions	
46	Concl	usions	198
ч.о. г			201
э.			
5.1.	Synop	osis	202
5.2.	Back	ground	203
5.3.	A Stu	dy into the Distribution of Gunshot Residue Particles in the Random P	opulation.
			206
	5.3.1.	Introduction	
	5.3.2.	Materials and methods	
	5.3.3.	Results and discussion	
	5.3.4.	Conclusions	216
5.4.	Addit	ional Data	218
5.5.	Concl	usions	222
6.	GSR BA	CKGROUND IN THE POLICE POPULATION	225
с.1	Current Current		226
0.1.	Synop		220
6.2.	Back	ground	228
6.3.	Guns	hot Residue Background on Police officers: Considerations for seconda	ary
tran	ster in G	SR evidence evaluation	233
	6.3.1.	Introduction	
	6.3.2.	Iviaterials and methods	
	6.3.3.	Results and Discussion	
	6.3.4.		254
6.4.	Δ 1000 1000	unitions used by South Australian Police	255
	Amm	unitions used by South Australian Ponee	255
	6.4.1.	Background	
	6.4.1. 6.4.2.	Background Materials and Methods	
	6.4.1. 6.4.2. 6.4.3.	Background Materials and Methods Results and Discussion	

6.5.	GSR pi	resent on Special Units of the Police	265
	6.5.1.	Background	
	6.5.2.	Materials and Methods	
	6.5.3.	Results	
	6.5.4.	Conclusion	273
6.6.	Conclu	isions	275
7.	A FRAME	WORK FOR THE EVALUATION OF GSR EVIDENCE	277
7.1.	Synop	sis	278
7.2.	Backgi	round	279
	7.2.1.	Existing approaches to the evaluation of GSR evidence	
	7.2.2.	Challenges with a statistical model for GSR evidence evaluation	
	7.2.3.	Statistical approaches to GSR evidence evaluation	
	7.2.4.	A Bayesian Network approach to GSR evidence	
	7.2.5.	Proposed Bayesian Model	
7.3.	Mater	ials and methods	
	7.3.1.	Data Collection and Analysis	
7.4.	Result	s	294
	7.4.1	Node $B_1 - GSR$ Prevalence in the Random Population	294
	7.4.2.	Node $PB_1 - GSR$ Prevalence in the Police Population	
	7.4.3.	Node T ₁ – GSR Transfer During Arrest	
	7.4.4.	Node X – GSR Present	
7.5.	Discus	sion	302
7.6.	Conclu	isions	305
8	CONCLU	SIONS AND FUTURE WORK	307
0.1	Canal		200
ō.1.		ISIUIS	308
	0.1.1.	Sub particle composition and morphology of GSP	
	0.1.2. Q 1 2	GSR prevalence in the random population	012
	0.1.5. Q 1 /	GSR prevalence in the police population	
	0.1.4. 8 1 5	Secondary transfer during apprehension and arrest	216
	8.1.6	A logical framework for the assessment of GSR evidence	316
0.7	F		340
ð.2.	Future	e WORKS	
	ŏ.∠.⊥. o.a.a	Future Challenges in understanding GSK particle formation.	310
	ŏ.Z.Z.	Future Challenges for Statistical Evaluation of GSK	
Refe	erence List	t	322

DECLARATION

I certify that this thesis does not incorporate without acknowledgment any material previously submitted for a degree or diploma in any university; and that to the best of my knowledge and belief it does not contain any material previously published or written by another person except where due reference is made in the text.

Signed - Nick Lucas

Date - 17 June 2019

ACKNOWLEDGEMENTS

I remember once being asked what I would consider doing with my life when I finished my schooling. Having spent a fair amount of time considering the question, I surprised my audience by confidently responding that one day, I might like to be a palaeontologist. This was a little out of left field, partly because the previous student had said that he wanted to be a ninja turtle, and partly because I was four years old. When my kindergarten teacher asked me if I knew what a palaeontologist was, I patiently explained it to her, quietly questioning how one could claim to call themselves an educator without this vital knowledge. This anecdote tells you everything you need to know about me – I have a passion for the sciences, and I'm often quite tiresome to be around.

For as long as I can recall, science has been in my soul. Though I eventually cooled on my dreams of being a dinosaur hunter, it was not terribly long after that I developed an interest in forensic science. Over the intervening years, that initial morbid curiosity has blossomed into a passion, that has ultimately led me to where I sit now. On a journey this all-consuming, there is a long list of people to thank.

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What's next?

LIST OF FIGURES

Figure 2 - Cross-section of ammunition cartridges showing the differences between rimfire and centrefire4Figure 3 - Schematic of a shotgun cartridge in both shot and slug style.6Figure 4 - Main features of a revolver handgun (Smith and Wesson Model 63)7Figure 5 - Main features of a semi-automatic handgun (Smith and Wesson M&P.40)8Figure 6 - Extended Bayesian Network for evaluating GSR particle evidence as presented byBiedermann, Bozza and Taroni [148]72Figure 7 - BSE Images of Au/Nb/Ge/Si/C standard (Ardennes Analytique) demonstrating therelationship between brightness and Z.91Figure 8 - Particle classification categories as defined in the GSR Magnum software93Figure 10 - Percentage of cases by weapon type - Rimfire (RF) (n=36) and Centrefire (CF) (n=6)95Figure 11 - Cases involving 0.22 rimfire rifles ordered by action type - Bolt action (n=18), Lever
centrefire4Figure 3 - Schematic of a shotgun cartridge in both shot and slug style.6Figure 4 - Main features of a revolver handgun (Smith and Wesson Model 63)7Figure 5 - Main features of a semi-automatic handgun (Smith and Wesson M&P .40)8Figure 6 - Extended Bayesian Network for evaluating GSR particle evidence as presented byBiedermann, Bozza and Taroni [148]72Figure 7 - BSE Images of Au/Nb/Ge/Si/C standard (Ardennes Analytique) demonstrating therelationship between brightness and Z.91Figure 8 - Particle classification categories as defined in the GSR Magnum software93Figure 10 - Percentage of cases by weapon type - Rimfire (RF) (n=36) and Centrefire (CF) (n=6)95Figure 11 - Cases involving 0.22 rimfire rifles ordered by action type - Bolt action (n=18), Lever
Figure 3 - Schematic of a shotgun cartridge in both shot and slug style.6Figure 4 - Main features of a revolver handgun (Smith and Wesson Model 63)7Figure 5 - Main features of a semi-automatic handgun (Smith and Wesson M&P .40)8Figure 6 - Extended Bayesian Network for evaluating GSR particle evidence as presented byBiedermann, Bozza and Taroni [148]72Figure 7 - BSE Images of Au/Nb/Ge/Si/C standard (Ardennes Analytique) demonstrating therelationship between brightness and Z.91Figure 8 - Particle classification categories as defined in the GSR Magnum software93Figure 10 - Percentage of cases by weapon type - Rimfire (RF) (n=36) and Centrefire (CF) (n=6)95Figure 11 - Cases involving 0.22 rimfire rifles ordered by action type - Bolt action (n=18), Lever
Figure 4 - Main features of a revolver handgun (Smith and Wesson Model 63)
Figure 5 - Main features of a semi-automatic handgun (Smith and Wesson M&P .40)
Figure 6 - Extended Bayesian Network for evaluating GSR particle evidence as presented by Biedermann, Bozza and Taroni [148]
Biedermann, Bozza and Taroni [148].72Figure 7 - BSE Images of Au/Nb/Ge/Si/C standard (Ardennes Analytique) demonstrating the91relationship between brightness and Z.91Figure 8 - Particle classification categories as defined in the GSR Magnum software93Figure 9 - Plano SPS-5P-2 A-X02-Y03 Standard Particle Map95Figure 10 - Percentage of cases by weapon type - Rimfire (RF) (n=36) and Centrefire (CF) (n=6)rifles, Shotgun (n=12), Semi-automatic (SA) pistols (n=7), Revolvers (n=9) (70 cases).Figure 11 - Cases involving 0.22 rimfire rifles ordered by action type - Bolt action (n=18), Lever
Figure 7 - BSE Images of Au/Nb/Ge/Si/C standard (Ardennes Analytique) demonstrating the relationship between brightness and Z
relationship between brightness and Z
Figure 8 - Particle classification categories as defined in the GSR Magnum software
Figure 9 - Plano SPS-5P-2 A-X02-Y03 Standard Particle Map
Figure 10 - Percentage of cases by weapon type - Rimfire (RF) (n=36) and Centrefire (CF) (n=6) rifles, Shotgun (n=12), Semi-automatic (SA) pistols (n=7), Revolvers (n=9) (70 cases)
rifles, Shotgun (n=12), Semi-automatic (SA) pistols (n=7), Revolvers (n=9) (70 cases)
Figure 11 - Cases involving 0.22 rimfire rifles ordered by action type - Bolt action (n=18), Lever
action (n=3), Pump action (n=1) and unknown (n=13) (35 cases)
Figure 12 - Characteristic particles detected by percentage of cases (n=59)125
Figure 13 - Characteristic particles detected by percentage of stubs (n=195)
Figure 14 - Distribution of characteristic and consistent (excluding single-component) particle
numbers by case in those suicides involving 0.22 calibre rimfire ammunition (n=28)
Figure 15 - Percentage of cases containing either characteristic (Pb/Ba/Sb) particles and/or
consistent (excluding single-component) particle types (n=59)
Figure 16 - Average number of characteristic (Pb/Sb/Ba) GSR particles detected by post-firing
interval
Figure 17 - Average number of GSR particles (characteristic and consistent, excluding single-
component) detected by firing interval128
Figure 18 - Average number of characteristic (Pb/Sb/Ba) particles detected by post-firing
interval and victim location
Figure 19 - Average number of GSR particles (characteristic and consistent, excluding single-
component) detected by post-firing interval and victim location
Figure 20 - Average number of GSR particles (characteristic and consistent, excluding single-
component particles) detected by weapon type – Rimfire (RF) (n=28) and Centrefire (CF) (n=6)
rifles, shotguns (n=11), semi-automatic (SA) pistols (n=6) and revolvers (n-8)
Figure 21 - EDS spectrum of three-component particle from muzzle discharge using PMC
Zapper ammunition
Figure 22 - Secondary electron image and elemental map of three-component particles found
in muzzle discharge using PMC Zapper ammunition
Figure 23 - Separated elemental maps of three-component particle isolated from muzzle
discharge using PMC Zapper ammunition
Figure 24 - Percentage of cases involving 0.22 rimfire ammunition reporting particle types
containing antimony

Figure 25 - GSR cases involving rifles in South Australia between 2007 and 2016 separated by
action type
Figure 26 - Exemplar EDS spectrum of GSR from 0.22LR calibre PMC Zapper ammunition172
Figure 27 - EDS maps of FIB sectioned particle from 0.22 LR calibre PMC Zapper ammunition.
Figure 28 - Exemplar EDS spectrum of GSR from 0.40 American Eagle MFP ammunition 176
Figure 29 - EDS maps of FIB section particle from 0.40 American Eagle MFP ammunition 176
Figure 30 - Comparison of morphological differences between Sr only (top) and SrAI (bottom)
containing GSR particles originating from HMF ammunition (American Eagle 0.40 MFP)177
Figure 31 - Exemplar EDS spectrum of GSR from 0.40 Winchester Winclean ammunition178
Figure 32 - EDS maps of FIB sectioned particle from 0.40 Winchester Winclean ammunition.
Figure 33 - Exemplar EDS spectrum of GSR from 0.40 Federal Premium HST ammunition 179
Figure 34 - EDS maps of FIB sectioned particle from 0.40 Federal Premium HST ammunition 179
Figure 35 - Exemplar EDS spectrum of GSR from 7.62x39 calibre Norinco ammunition180
Figure 36 - EDS map of FIB sectioned particle from 7.62x39 Norinco (Steel Case)
Figure 37 - EDS Maps of FIB sections second particle from 7.62x39 Norinco (Steel Case) 181
Figure 38 – GSR Particle from 0.40 Federal Premium ammunition as observed prior to FIB
milling
Figure 39 – GSR Particle from 0.40 Federal Premium ammunition at stage tilt of 52°, prior to
deposition of the Pt cap
Figure 40 – The same GSR particle from 0.40 Federal Premium ammunition during the milling
process
Figure 41 – Section extracted from the GSR particle after it had been affixed to the Cu TEM
grid using Pt194
Figure 42 - Particle section following further thinning and polishing under the FIB
Figure 43 – Particle segment prepared from 0.22 PMC Zapper ammunition. Note the thicker
coating of platinum present on the top and sides of the segment
Figure 44 - Proposed Bayesian Network for the analysis of GSR results from Biedermann, Bozza
& Taroni [1]
Figure 45 – Backscatter image, elemental distribution X-ray map and EDS spectrum of a large
characteristic particle from the South Australian survey
Figure 46 - Subjects returning a positive GSR result grouped by declared GSR exposures
(hobbies, occupation, or other) and by gender. "All GSRs" is the sum of characteristic and
consistent particles detected
Figure 47 - Subjects returning a positive GSR result grouped by age
Figure 48 - Subjects returning a positive GSR result grouped by time since last handwashing.
Figure 49 - Proposed Bayesian Network for the analysis of GSR results from Biedermann, Bozza
& Taroni [1]
Figure 50 - Time between last handling a firearm and GSR sampling. (n=75)
Figure 51 - Comparison plot of Characteristic and Consistent particle types in the Random and
Police population
Figure 52 - Total GSR transfer efficiency grouped by quartile
Figure 53 - Box and whisker plot of transfer variability between quartiles

Figure 54 - Recovered cartridge case and expanded projectile from 0.40 S&W Federal Premium
Law Enforcement HST ammunition255
Figure 55 - Recovered fired cartridge case and projectile from 0.40 S&W Federal American
Eagle Cu-TMJ, Toxic metal free primed ammunition
Figure 56 - Firearm discharge into bullet recovery (water) tank. (left). Area of dominant hand
from which sample was collected (right)258
Figure 57 - Exemplar BES image and EDS spectrum of PbSbBa particle originating from 0.40
Federal Premium Law Enforcement ammunition
Figure 58 - Comparison BSE images and EDS spectra showing morphological differences
between Sr only (top) and SrAI (bottom) containing GSR particles originating from 0.40 Federal
American Eagle MFP ammunition
Figure 59 - SrAl flake originating from Federal American Eagle HMF Fired Cartridge Case 263
Figure 60 - Spent 'flashbang' stun grenades sampled for particulate residues
Figure 61 - Exemplar SrAl particle consistent with Federal American Eagle HMF ammunition
recovered from the glove of a STAR group officer
Figure 62 - Fragment of debris recovered from a Flashbang. Fragment measures approx. 4 μ m
by 2 μm
Figure 63 - An example Bayesian network for the assessment of GSR evidence and table
defining the nodes as presented by Biedermann, Bozza and Taroni [148] informed by data
collected by Cardinetti et al. [147]
Figure 64 - Extended Bayesian network for evaluating GSR particle evidence as presented by
Biedermann, Bozza and Taroni [148]
Figure 65 – A Bayesian Network for evaluating GSR particle evidence adapted from
Biedermann, Bozza and Taroni [148]
Figure 66 - Adapted Bayesian Network model for the assessment of GSR evidence created in
HUGIN Lite
Figure 67 - Segment of the adapted Bayesian Network evaluated as a part of this research . 294
Figure 68 – Probability distribution for observed characteristic GSR particles on the hands of
serving South Australian Police officers [237]
Figure 69 – Characteristic GSR particle percentage secondary transfer efficiency grouped by
quartile
Figure 70 - Probability distribution for observing one or more characteristic GSR particle
present as a result of secondary transfer from the hands of general duties police officers 299
Figure 71 - Probability distribution of characteristic GSR particles due to background
prevalence and secondary transfer due to contact with police

LIST OF TABLES

Table 1 - Categories and types of firearms as defined by the South Australian Firearms Act	
2015 [7]	12
Table 2 - GSR particle classification nierarchy as per ASTM E1588-17 [13]	18
Table 3 - GSR particle classification for Pb-free/non-toxic primer ammunitions as per ASTM	
	18
Table 4 - General composition of Sinoxid type primers [1].	19
Table 5 - Typical Composition of a Sintox type primer compound, relative composition and	_
purpose in the primer mix [1]2	20
Table 6 - Listing of commonly encountered organic compounds and their functions in	
propellant that may contribute to OGSR. [1, 10, 39-42]2	23
Table 7 - Comparison of Police Officer Characteristic GSR background studies conducted in	
Australia and Internationally7	′5
Table 8 - Particle sizes and numbers present on the Plano SPS-5P-2 A-X02-Y03 Particle	
Standard	94
Table 9 - Analysis of Plano standard at 0.5 μ m minimum particle size while changing pixels per	r
particle setting) 7
Table 10 - Analysis of Plano standard at 0.3 μ m minimum particle size while changing pixels	
per particle setting	98
Table 11 - Analysis of Plano standard at $0.3\mu m$ minimum particle size while changing pixels pe	٩r
particle setting, separated by particle size10)0
Table 12 - Calculated z-scores for FEI F50 at 0.3 μm min. particle size and varied pixels per	
particle)1
Table 13 - Performance of the Zeiss Evo 50 on Plano SPS-5P-A-X02-Y0310)2
Table 14 - Performance comparison of Plano Standard on the FEI F50 and Zeiss Evo 50 system	IS
)3
Table 15 - Particles present on the ENFSI GSR2014 sample and particles detected by the FEI	
Inspect F50)5
Table 16 - Calculated z-scores for the FEI Inspect F50 using ENFSI GSR2014 sample, running at	
0.3 µm minimum particle size and 2 pixels per particle)5
Table 17 - Particles present on the ENFSI GSR2014 sample and particles detected by the Zeiss	
Evo 50)6
Table 18 - Calculated z-scores for the Zeiss Evo 50)6
Table 20 - Post firing intervals by case	21
Table 21 - Weapon type and calibre frequency data	>>
Table 22 - Consistent particle type data	35
Table 22 – Number and percentage of firearm types used in GSR cases in South Australia	,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,
hetween 2007 and 2016	15
Table 24 – Weapon types and calibres observed in GSR cases in South Australia between 2007	7
and 2016	19
Table 25 Amounition product manufacturer/brand by firearm type as observed in South	۲Ö
Australia between 2007 and 2016	10
Australia between 2007 and 2016	ŧ9

Table 26 - Ammunition manufacturer prevalence across all firearm types as observed in GSR
cases in South Australia between 2007 and 2016
Table 27 - Separation of GSR results based on firearm type as observed in all cases involving
GSR analysis in South Australia between 2007 and 2016
Table 28 - Particle composition types separated by firearm type as observed in all case samples
involving GSR analysis in South Australia between 2007 and 2016.
Table 29 – Particle composition types observed in all case samples using rifles, separated by
ammunition type
Table 30 - Additional elements most frequently incorporated into GSR particles separated by
firearm type
Table 31 - Typical composition of a Sinovid type primer compound relative abundance and
numose in primer mix [1]
Table 32 - Typical Composition of a Sintax type primer compound, relative composition and
numore in the primer mix [1]
Table 22 Ammunition Tacted and Major Components present [70]
Table 33 - Ammunition rested and Wajor Components present [70]
Table 34 - Components Present in Cartridge Casing and Projectiles
Table 35 - Particle count from PIVIC 2apper ammunition barrel discharge sample (25% of
available stud area)
Table 36 - Comparison of particle types collected from barrel scraping, barrel discharge, and
breech scraping
Table 37 - Particle count from CCI Stinger ammunition barrel discharge sample (100% of
available stub area)
Table 38 - Comparison of particle types collected from barrel scraping, barrel discharge, and
breech scraping after firing CCI Stinger Ammunition
Table 39 - Comparison of .22LR CCI Stinger and .22LR PMC Zapper Barrel Discharge Residues
Table 40 - Collated particle data for characteristic and consistent particles 210
Table 41 - Collated particle data for single element particles 215
Table 42 - Collated particle data for characteristic, consistent and single element particles from
the NSW survey conducted by Hales [105]219
Table 43 - Comparison of observed particle frequencies separated by state 221
Table 44 - Comparison of Police Officer Characteristic GSR background studies conducted in
Australia and Internationally229
Table 45 - Set-up and Operating Conditions for SEM-EDS analysis
Table 46 - Self-reported time since last firing a firearm based on 76 surveys
Table 47 - Self-reported time since last hand-washing based on 76 surveys
Table 48 - Survey data separated based on last contact with a firearm and hand washing 243
Table 49 - Collected GSR particle data for samples collected from police officers' hands (n=76)
Table 50 - Comparison of Characteristic and Consistent particle types in the random
population and the police population
Table 51 - Comparison of results of Characteristic and Consistent particle types found on the
hands of officers who had handled a firearm <1hr before sampling with Cook [101] 246
Table 52 - Number of GSR particles, separated by particle type, for both officer and POI and
secondary transfer efficiency following mock arrest scenario

Table 53 – Particle summary for hand samples collected from the shooter's hands pre-firing,	
and post firing 0.40 S&W Federal American Eagle (TMF) and 0.40 S&W Federal Premium Law	
Enforcement ammunitions26	0
Table 54 - Particle summary of samples from gloves of STAR group officers26	9
Table 55 - Particle summary from 'flashbang' stun grenade samples	'1
Table 56 - Calculated probabilities for observing characteristic GSR on the hands of a POI due	
to random GSR background29	5
Table 57 - Calculated probabilities for observing characteristic GSR on the hands of a POI due	
to the secondary transfer from police during apprehension and arrest	9
Table 58 - Calculated probabilities for observing characteristic GSR on the hands of a POI due	
to the GSR background in the random population, and secondary transfer from police during	

LIST OF ABBREVIATIONS AND SYMBOLS

ACRONYM	DEFINITION
μm	micrometre /micron
Å	Ångstrom
AAS	Atomic Absorption Spectroscopy
AES	Auger Emission Spectroscopy
AFTE	Association of Firearms and Toolmark Examiners
ANZFSS	Australian and New Zealand Forensic Science Society
ASTM	American Society of Testing and Materials
BN	Bayesian Network
BSD	Backscatter detector
BSE	Backscattered electrons
cd	Candela
CPS	Counts Per Second
CSI	Crime Scene Investigator
dB	Decibels
DPA	Diphenylamine
EC	Ethyl Centralite
EDS / EDX	Energy dispersive X-ray spectroscopy
ENFSI	European Network of Forensic Science Institutes
ET	Everhart-Thornley
eV	Electron volts
FC	Faraday Cup
FCC	Fired Cartridge Case
FEG	Field Emission Gun
FFE	Forensic Firearms Examiner
FIB	Focussed Ion Beam
FSSA	Forensic Science South Australia
FWHM	Full width at half maximum
ga	Gauge
gGSR	Glass-containing Gunshot Residue
HMF	Heavy Metal Free
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
IED	Improvised Explosive Device
iGSR	Inorganic Gunshot Residue
ISO	International Organisation for Standardisation
JHP	Jacketed Hollow Point
keV	Kiloelectron volts
kPa	Kilopascals
LIBS	Laser Induced Breakdown Spectroscopy
LR	Likelihood Ratio
MC	Methyl Centralite
MFP	Metal Free Primer
mm	Milimetre

na	Nanoamp
NAA	Neutron Activation Analysis
NC	Nitrocellulose
NFDD	Noise-Flash Diversionary Devices
NG	Nitroglycerine
NSW	New South Wales
OEM	Original Equipment Manufacturer
oGSR	Organic Gunshot Residue
PETN	Petaerythritol tetranitrate
POI	Person of Interest
рх	pixels
RACI	Royal Australian Chemical Institute
SA	South Australia
SAG	Specialist Advisory Group
SAPOL	South Australia Police
SDD	Silicon Drift Detector
SDS	Safety Data Sheet
SE	Secondary Electrons
SED	Secondary Electron Detector
SEM	Scanning electron microscope/Scanning Electron Microscopy
SMANZFL	Senior Managers of Australian and New Zealand Forensic Laboratories
SOCG	Serious and Organised Crime Gangs
SPG TOU	State Protection Group Tactical Operations Unit
SPS	Synthetic Particle Standard
SS-BSED	Solid State-Backscattered Electron Detector
STAR	Special Tasks and Rescue
SWAT	Special Weapons and Tactics
SWGGSR	Scientific Working Group for GSR
TEAM	Texture and Elemental Analysis and Mapping
TEM	Transmission Electron Microscopy
TMF	Toxic Metal Free
TMJ	Total Metal Jacket
ToF-SIMS	Time of Flight Secondary Ion Mass Spectrometry
VICPOL	Victoria Police
XRF	X-Ray Fluorescence Spectroscopy
Z	Atomic Number
ZPP	Zirconium Potassium Perchlorate

PUBLICATIONS

The following is a list of publications by this author included as a part of this thesis.

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Lucas, N.; Cook, M.; Wallace, J.; Kirkbride, K. P.; Kobus, H., Quantifying gunshot residues in cases of suicide: Implications for evaluation of suicides and criminal shootings. *Forensic Science International* **2016**, *266*, pp 289-298.

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ABSTRACT

The interpretation of gunshot residue (GSR) evidence presents a number of challenges in evaluating the significance of a particular finding. Therefore, to ensure that evidence is placed in the appropriate context, a systematic approach to reviewing the factors that influence the significance of a GSR finding must be taken. To assist this, a framework assessing the numerous factors related to the deposition and persistence of GSR may serve as a useful tool to aid interpretation. The research performed in this thesis assesses a number of factors pertinent to GSR evidence evaluation in South Australia, and explores incorporating them into an evidential assessment framework.

To inform this framework, the type and nature of firearms frequently encountered in forensic casework in the region was ascertained through a comprehensive case-review. The frequency with which 0.22LR ammunition was encountered prompted further investigation of the impact of the weapon memory effect, and how two-component primed ammunition, such as many 0.22LR ammunitions, can create three-component GSR particles. Additional investigation of GSR generated from heavy metal free (HMF) ammunitions also provides valuable context for GSR assessments in a changing ammunition market. This information may be used to better inform GSR analysts of what may be expected in different case circumstances.

As a further means of assessing the significance of a particular GSR finding, one particularly important factor is the possibility that GSR present on a suspect is due to contamination from an unrelated incident or source, rather than the incident under investigation. Contributing to this are factors such as the likelihood of cross-contamination from police and the background level of GSR in the random population. These factors inform the possibility that an individual may be mistakenly included in an investigation as a result of a false positive error. To investigate this, surveys of the background prevalence of GSR in the random Australian population were performed. Similarly, the prevalence of GSR particles present on the hands of police officers, and the possibility that these may be transferred to a suspect during the process of arrest was also surveyed.

The results of these surveys were then assessed in the context of their contribution to an evidential framework that would allow for a greater understanding of the significance of a GSR test result. The calculated probabilities of observing GSR in each of the surveys was used to inform a Bayesian Network (BN) style approach to the assessment of GSR evidence. A BN

xxi

approach is particularly useful in the assessment of complex evidence, as the network structure allows for the model to be adapted as case conditions changed. The experimental data was used to inform a section of this framework, targeted at assessing the probability that an individual unrelated to an investigation would return a positive GSR test result as a consequence of the GSR background, or transfer from police. The findings of this assessment may be combined with relevant case information to better inform evaluations of the significance of GSR casework results in South Australia.

1.INTRODUCTION AND LITERATURE REVIEW

1.1. SYNOPSIS

Gunshot residue (GSR) is a valuable form of trace evidence in the investigation of firearms crime. It is distributed into the area around the firearm in the immediate aftermath of a firearm discharge, and therefore may be located on the shooter, any victims or bystanders, and on any surfaces in the vicinity. In this way, GSR may be used as evidence of a firearms association between persons and/or objects.

Part of the difficulty around the use of GSR evidence is related to interpretation of the significance of a finding. In order for the evidence to be valuable, the likelihood of observing a false positive (or type 1 error) or false negative (or type 2 error), as well as other factors that may influence the reliability of that finding must be understood. As with many forms of trace evidence, these factors represent numerous dynamic influences on the distribution, transfer, and collection of the evidence. To facilitate the interpretation of evidence, evidence dynamics must be considered at all stages of the forensic process, from crime scene, through analysis, to the court room. Particularly pertinent dynamic factors in the case of GSR analysis are conditions such as the GSR background in the random population, background on law enforcement, likelihood and extent of secondary and further transfer, and expected GSR distribution following firearm discharge must be considered.

This thesis represents a significant original contribution to knowledge through the further investigation of these factors informing a GSR test result, and establishing a framework informed by data to aid in the assessment of GSR evidence. These factors are particularly applicable to the Australian environment and jurisdictions, as a framework addressing these considerations has only been tentatively explored in this region.

1.2. FIREARM BACKGROUND

1.2.1. Ammunition

In order to understand the dynamics of gunshot residue creation, distribution, and transfer, first an elementary understanding of the nature and function of ammunition and firearms is required. At a fundamental level, most modern ammunitions consist of a simple explosive train packaged with a dense projectile, contained within a metal cartridge. The explosive train contains two components; a small quantity of shock sensitive primary explosive— the primer, and a larger quantity of less sensitive secondary explosive — the propellant or powder. A schematic representation of an ammunition cartridge representing the arrangement of these components can be observed in Figure 1.

Image removed due to copyright restriction.

Figure 1 - Ammunition cartridge in cross-section showing the principal components (Image courtesy of Google Images – used under fair use)

This two component system is an elegant solution to many of the problems involving the loading of firearms and the storage, transport, and maintenance of ammunitions. In the early days of firearms, the powder, primer, and projectile were handled separately and had to be introduced to the barrel of the firearm independently. This lack of a standard delivery system increased the likelihood of mishandling and misfiring, significantly impacting the reliability of firearms [1].

The secondary explosive component of the ammunition cartridge is more resistant to heat, shock, and friction, which reduces the chances of unexpected ignition. This renders the cartridges more robust, allowing them to be handled during transport without fear of accidental discharge. The trade-off in this circumstance however, is that being a secondary explosive, it requires a more substantial amount of energy to ignite. This is the role of the primer. The primary explosive component is shock sensitive, and therefore unstable enough to be initiated by a blow from the firing pin. This then initiates the secondary explosive, causing the explosive expansion of gases and particulates resulting in the projectile being expelled from the barrel.

Primer arrangements in commercial cartridges come in two arrangements - rimfire and centrefire. A diagram presenting the differences between the two arrangements can be observed in Figure 2.

Image removed due to copyright restriction.

Figure 2 - Cross-section of ammunition cartridges showing the differences between rimfire and centrefire (Image courtesy of Google Images – used under fair use)

Rimfire cartridges have the primer more or less evenly distributed around the rim at the base of the cartridge, and a strike from the firing pin to anywhere along the rim will initiate the primer. Cartridges of this type are easier and less expensive to make, but are restricted to lower calibres, most commonly 0.22 and below, as the base of the casing must be made of thinner brass to allow the firing pin to make adequate contact to initiate the primer. The larger powder charge required to fire a larger calibre bullet in a rimfire cartridge would likely result in distortion or critical failure of the casing, potentially leading to jamming, failure to fire, or catastrophic failure of the firearm. The requirement for a thinner casing makes these rounds unsuitable for larger calibres and less durable, therefore not well suited to military or law enforcement applications. However, their low cost per round means they are frequently seen in agricultural and amateur shooting contexts. Further, due to the lower cost of manufacture and process of distributing the primer into the case, anecdotal evidence suggests that a higher percentage of rimfire ammunition will fail to fire. While this percentage is still very low as a percentage of total rounds fired, it has led some more dedicated shooters to favour centrefire ammunition over rimfire [2, 3].

By comparison, centerfire ammunition contains the primer compound within a thin metallic primer cap located in the centre of the base of the cartridge. This allows the remainder of the cartridge to be thicker and more robust, as long as the firing pin impacts the thinner primer cap. Centerfire cartridges are more expensive and marginally more technically complicated to make, but their thicker construction allows them to be reliably made in larger and more diverse calibres. They are also more robust and resistant to damage, making them well suited for military, law enforcement and commercial applications [1].

Shotgun shells have a similar construction, with the projectile being replaced with either multiple small metal pellets (shot) or a single large slug. Shotgun shells are manufactured in full metal and full plastic arrangements, but the most commonly encountered construction consists of a thick metallic base containing the primer and powder, and a thinner plastic shell containing the shot or slug, and other components.

Image removed due to copyright restriction.

Figure 3 - Schematic of a shotgun cartridge in both shot and slug style. (Image courtesy of Google Images – used under fair use)

When discussing calibre of ammunition, measurements for pistol and rifle cartridges are reported as the internal diameter of the firearm barrel in fractions of an inch or in millimetres. In situations where two numbers are used, the second often refers to the overall length of the cartridge case. For example; 0.45 calibre ammunition is to be used in a firearm with a barrel measuring 0.45 of an inch (11.43mm) while 7.62x51mm NATO ammunition is for a barrel measuring 7.62mm (0.3 Inch), and the casing itself is 51mm long. When referring to shotguns, the shell size is measured in 'gauge' or 'bore' rather than calibre, with the gauge measurement representing the number of slugs with the same diameter as the bore could be made using 1 pound of lead [1]. Therefore, the lower the gauge, the larger the diameter of the barrel. For example, for the popular 12 gauge, or 12 bore shotgun, 12 lead balls of equal dimension to the barrel diameter have a weight of 1 pound.

Different manufacturers use a different combination of primers, propellants, projectiles, powders, and casings for their ammunition products meaning there is significant variation in the function of different ammunitions. From a practical GSR analysis perspective, this can impact the chemical composition of the resulting residues, as well as the amount and type of residues that are present.

1.2.2. Firearms

It is also instructive in the discussion of GSR to have a fundamental knowledge of the operation of firearms, and the core features of a firearm. Labelled schematics of two handguns, a revolver and a semi-automatic pistol can be seen in Figure 4 and Figure 5, respectively.

Image removed due to copyright restriction.

Figure 4 - Main features of a revolver handgun (Smith and Wesson Model 63 [4]) (Image supplied and annotated by author)

A revolver-style handgun possesses a cylinder with a number of chambers, each designed to accommodate a single ammunition cartridge. To discharge the firearm, the hammer is cocked and released to impact the base of the ammunition cartridge by pulling the trigger. The cylinder is then mechanically rotated to bring the next chamber into position. This mode of operation means that a revolver can be fired as many times as there are chambers before having to reload. From a firearms investigation perspective, revolvers are notable for retaining spent cartridge cases within the cylinder until such time that the cylinder is manually released, and the cases expelled by use of the ejector rod. From a GSR perspective, the need for a free-rotating cylinder means that there is a gap between the cylinder and the frame of the firearm. This allows a significant amount of residue to escape during discharge, increasing the amount of GSR that is released in the vicinity of the hands of the shooter [5].

Distinct from a revolver, Figure 5 presents a labelled schematic of a semi-automatic handgun. The semi-automatic designation indicates that with each pull of the trigger, a round is discharged, followed by the spent casing being automatically ejected via the ejection port, before the next live round is chambered. This is accomplished through the action of the slide, which operates under the force either recoil or gas pressure. This is also known as a 'selfloading' pistol. Rather than a cylinder containing the ammunition, semi-automatic pistols contain their ammunition within a magazine, often secured within the hand-grip, which is then fed to the barrel following each successive trigger pull.

Image removed due to copyright restriction.

Figure 5 - Main features of a semi-automatic handgun (Smith and Wesson M&P .40 [6]) (Image supplied and annotated by author)

n.b. The firearm in this image is unloaded. The red safety flag is visible in the ejection port, and the magazine well, which accommodates the ammunition magazine when loaded, is empty

Regions of the firearm most pertinent to the analysis of GSR are the muzzle or barrel, through which the majority of residues are expelled, along with the projectile, during firing. Like revolvers, any gaps in the frame of the firearm that can allow the release of the internal gasses and residues may be a rich source for the distribution of GSR. Particularly pertinent for semi-automatic firearms is the ejection port, which expels the spent cartridge casing following firing, and gaps in the frame around the trigger and slide [5]. Collectively, semi-automatic pistols and revolvers are often referred to as 'handguns', 'pistols' or in some situations 'short-arms'. This is to differentiate them from rifles and shotguns, which are often referred to collectively as 'long arms', and are generally fired from the shoulder, using both arms to control the firearm.

The basic function of rifles has many similarities to the operation of pistols, with the exception of the fact that rifles possess a longer, rifled barrel designed to make them more accurate over long distances. Therefore, they tend to be available in higher calibres than pistols. However, like pistols, they also fall into a number of broad classes depending on their configuration, type of action and style of operation. They can be configured to operate using either rimfire or centrefire ammunition, and can be manual, semi-automatic (self-loading), or automatic. Similarly, the method used for extraction of the spent cartridge can be via gas pressure or recoil energy as in semi-automatic pistols, or by lever, bolt, break, or pump action for manual extraction. Similar to rifles, shotguns have a long barrel, but it tends not to be rifled¹, making them less accurate over distance. However, they are designed to fire larger shells containing either a solid slug projectile, or shot, which is comprised of a number of smaller lead pellets. The advantage of shot is that the smaller pellets spread out over a short range, creating a wider cone of projectiles, which is particularly effective if attempting to hit small, fast moving targets. Historically, shotguns have been used to hunt birds and small game; however their flexibility and versatility has led to them being used as a close-quarters weapon in a number of military and law enforcement contexts. Much like rifles, the method for ejecting spent shells is varied and break, lever, bolt, revolver, pump, and semi and full automatic shotguns all see use.

From a firearms investigation perspective manual action rifles and shotguns contain spent cartridges until such time as they are manually removed, which potentially limits the recovery of casings and spent shells at a shooting scene. Regarding the distribution of GSR, the longer barrel disperses residues further from the shooter. Studies of GSR distribution patterns have indicated that regions of higher GSR particle concentration when firing rifles and shotguns are found on the support arm, face or hair, rather than on the hands [5]. However, in situations where there is a self-loading function, GSR tends to be distributed closer to the shooter from the ejection port, or breech of the firearm.

Fundamentally, the type, construction, and calibre of ammunition used in a firearm can influence the type of GSR that is generated, as well as how and where it is distributed into the environment. This can have an impact on where it may be detected on the shooter, or where else in the environment surrounding a firearms discharge that residues may be seen. The prevalence and popularity of the different types of firearms in a specific jurisdiction can also influence how frequently these firearms are encountered in forensic casework, as well as impacting factors such as the GSR background of a jurisdiction.

¹ Rifled barrelled shotguns, while rare, do exist, with this arrangement tending to be used to allow the firing of a slug over greater distances with increased accuracy. This does create a classification issue, due to the fact that in many classification systems, a smooth bore is considered the defining characteristic of a shotgun, while a rifled barrel is what defines a rifle.

1.2.3. The Australian Firearms Environment

Further informing the firearms environment in Australia is the legislation that is in place restricting the private ownership of firearms. In Australia, the individual states and territories are responsible for their own firearms legislation, however in all regions, it is similarly stringent. In the jurisdiction where the majority of this research took place, South Australia, the South Australia Firearms Act 2015 regulates the possession and use of automatic and selfloading firearms. Firearms are required to be registered, and all firearms users must be licenced and have a genuine reason to possess a firearm [7]. Under the Act, all firearms are divided into categories based on their means of operation, capacity and potential uses. For practical purposes, the higher the category, the more regulated and restricted the ownership and use of the firearm is. A summary of the categories as defined in the Act can be seen in Table 1. At the lowest level, are category A firearms. However, ownership of a firearm in this category still requires that documentation of a genuine reason for possessing a firearm is provided to the state or territory police, the firearm is registered and identifiable, and that it will be stored appropriately. Generally, the genuine reason for firearm ownership must be supported by documented evidence that the firearm is required for legitimate shooting club, hunting, or occupational uses. Collector licences are also issued for firearms in all categories with the exception of those in the prescribed category, with the requirement that category D firearms are rendered permanently inoperable. Due to the stringent regulations, category A firearms, the least strictly regulated, have a tendency to be among the most frequently encountered firearms in forensic casework and GSR analysis, simply due to their availability. While the specifics of firearms legislation differ between the other Australian States and Territories, the overall purpose is the same, with firearm ownership heavily controlled in all Australian jurisdictions.

Despite the stringent regulations, most recent estimates suggest that the number of firearms in civilian possession in Australia is 3,573,000, or between 14 and 16 firearms per 100 people [8]. Of these, the vast majority (93%) are registered firearms in the hands of an estimated 816,000 licenced owners [9]. However, the Australian Criminal Intelligence Commission also estimates that 260,000 of the 3,573,000 guns are illicit firearms, comprising 250,000 longarms, and 10,000 handguns [9].

The firearms environment in Australia is unique, and therefore distinct from other regions of the world in which detailed GSR research has been performed. For the purposes of firearms and GSR investigations, it is therefore most pertinent to consider the most frequently encountered firearms in an Australian context. The consequences of regulation and legislation has ensured that though handguns and other illicit firearms are encountered, the availability of category A type firearms, most notably shotguns and small calibre, rimfire rifles, results in them being some of the most frequently encountered firearms in a forensic context.

CATEGORY A	CATEGORY B	CATEGORY C	CATEGORY D	CATEGORY H	PRESCRIBED
	Muzzle loading	Self-loading rimfire	Self-loading rimfire rifles		
Air guns	firearms (not being	rifles with capacity 10	with capacity 10 rounds		Automatic firearms
	handguns)	rounds or less.	or more.		
Paint-ball firearms	Revolving chamber rifles	Self-loading shotguns with capacity 5 rounds or less	Self-loading centrefire rifles		Mortars, bazookas, rocket propelled grenades, and similar military firearms designed to fire explosive projectiles
Rim fire rifles (not being self-loading rifles)	Centre fire rifles (not self-loading)	Pump action shotguns with capacity 5 rounds or less	Self-loading shotguns with capacity 5 rounds or more	All handguns	Firearms designed to fire projectiles containing tear gas or any other lachrymatory substance
Shotguns (not being self-loading or pump action)	Multiple barrel centre fire rifles		Pump action shotguns with capacity 5 rounds or more		Firearms designed to appear as other objects
Break action combination shotguns and rimfire rifles	Break action shotguns and rifles				

Table 1 - Categories and types of firearms as defined by the South Australian Firearms Act 2015 [7]
1.3. ORIGINS OF GUNSHOT RESIDUE

1.3.1. Formation

When the trigger is pulled on a firearm, the hammer drops and strikes the firing pin, pushing it forward to impact the primer, causing it to detonate. This then delivers the required energy for the propellant to burn, initiating a reaction that causes a rapid spike in temperature and pressure and resulting in the expulsion both the projectile and the residues from the firearm. This initial explosion within the firearm reaches a sufficient temperature to exceed the vaporisation temperature of the heavy metallic elements within the cartridge– in excess of 1600°C [10]. As this vapour disperses away from the firearm, the vaporised components mix and mingle before condensing back into liquid droplets, eventually solidifying into the spheroidal, three-component particles that are considered most suggestive of a firearm origin [11]. The residues generated as a part of this process escape the firearm from the end of the barrel, the ejection port, and any other gaps in the frame of the firearm, such as the cylinder and trigger gaps in a revolver, or any other gaps around the slide or firing mechanism.

The initial defining work on GSR particle morphology by Basu [11] divides GSR particles into three broad categories.

- Type I particles were defined as small regular, nodular or irregular spheroids with uniform and even mingling of Pb, Ba, and Sb across the entire particle.
- Type II particles were defined as hollow-shaped, pitted or atypical and irregular, with larger protrusions and knobs about their surface. Elemental distribution in these particles is irregular and discontinuous, often with discrete regions containing Pb, Ba, or Sb across the particle.
- Type III particles were described as existing as an outer envelope, partially
 encompassing a spheroidal central core, referred to by Basu as a 'peeled
 orange' type particle [11]. The central core tends to be a uniform mixture of
 Sb and Ba, while the outer sheath is typically a thin layer of Pb.

Following this, research into the classification of GSR performed by Wolten et al. indicated that upwards of 70% of 'unique' (now called characteristic) GSR particles exhibited spheroidal morphology [12]. While the spheroidal morphology of GSR remains a consideration in evaluating a GSR sample, particles originating from primers have been observed to exhibit angular or irregular morphologies [5]. The current version of the ASTM notes that the

morphology of a particle should not be the only criterion for GSR identification [13], it remains an important consideration in evaluating if a particle originates from a firearm or an environmental source.

Further research on the morphology of GSR has indicated that particle morphology can be affected by collisions with surfaces while the particles are still in the liquid phase [14]. Surfaces that are in close proximity to a weapons discharge may present with particles that exhibit morphology more akin to a 'spattered droplet', with a flattened or distorted appearance rather than a spheroid [14]. Similarly, the morphology of GSR recovered from the inside of a fired cartridge case is affected by the proximity to the initial reaction, with irregular morphology being much more prevalent as compared to airborne GSR [15].

1.3.2. Composition

The resultant composition of GSR depends heavily on the components that are present in the primer and powder. These may vary slightly between manufacturers, ammunition batches, specific applications and types of ammunition [1].

Traditionally, GSR has been divided into two broad classes based on the components that are present within it – Organic GSR (oGSR) contains organic compounds such as nitroglycerine, nitrocellulose and other components of the powder charge, while Inorganic GSR (iGSR) contains of metallic elements such as Pb, Ba, and Sb, and typically originates from the primer charge. While both types of GSR may be forensically useful, the analysis of iGSR residues is particularly well established. Unless otherwise specified, the term GSR will be used in reference to iGSR through the remainder of this thesis.

The elements of primary concern in GSR analysis are lead – Pb, barium – Ba, and antimony – Sb. While a number of other metals may be present within GSR, these three are the primary components of the spheroidal three-component particle that is considered 'characteristic' of GSR, that is, they are rarely found together in particles from other sources. However, other elements used in the compounds that make up the primer, depending on its formulation, may be present, and include K, Ca, Hg, P, S, and Al, amongst others [1].

Other elements that may be incorporated into GSR particles may originate from:

The Projectile

The barrel of many rifles and handguns is rifled, with lands and grooves within the barrel causing the bullet to spin, stabilising its flight and imparting greater accuracy over range. Contact between the bullet and these grooves results in the production of striations on the bullets surface – a vital component of forensic ballistics – as well as the transfer of bullet surface material to the barrel. The material used in the projectile is typically a dense metal alloy, with or without a metal jacket. The most common of these arrangements is a Pb projectile with a either a partial or total Cu jacket, but lead hardened with antimony or tin, or other metals is also common in civilian applications [1]. Similarly, a jacket may or may not be present, or may be constructed of a variety of different materials. Brass, Al, and mild steel alloy jackets are also relatively common. Early work in the field conducted by Wolten and Nesbitt, has indicated that there is a clear differentiation between particles derived from the projectile, and particles originating from the primer [16]. They found that projectile particles primarily contain components from the surface of the projectile, including any coating or jacketing material, while primer derived particles contain both components of the primer and components from the projectiles. Further firing tests have indicated that whatever the outermost surface of the bullet comprises [17] will be found in relatively higher abundance in the resultant GSR [18], potentially allowing differentiation of the ammunition used based on the residues generated. Studies performed using blank cartridges, that is, cartridges that do not have a projectile, suggests that the projectile contributes a large number of Pb-only particles [19].

The Barrel

In addition to particulate residue present within the barrel due to the passage of the projectile, any other residues or contamination present in the barrel may contribute to the composition of GSR. Poorly maintained firearms may have rust or corrosion present within the barrel which will result in higher contributions from Fe or other compounds in the resulting residues [10]. Further, the metal that the firearm is constructed from can be identified at trace levels in GSR [20]. There has been some evidence to suggest that other substances, such as bluing or blacking agents applied to the firearm, may contribute to the elemental composition of GSR, and therefore may be of benefit in linking a weapon and its particulate residue [21].

Particles present in the barrel may include any residual GSR present from firings of previous ammunition, which is then expelled with subsequent discharges – a phenomenon known as the 'memory effect' [22] of firearms. Residues from previous firings of different ammunition may be retained in the barrel or firing mechanism and are distributed with GSR generated with subsequent firings [23] resulting in 'mixed composition' GSR that consists of elements from the two independent sources. Care in interpretation is then required, as atypical results may be observed, such as residues generated from Pb-free ammunition containing Pb. The memory effect has been found to be quite pronounced, with a number of careful and involved protocols to remove contamination failing to adequately address the issue [24].

The Primer Cap

In centrefire ammunition, the primer cap is the small metal cup set in the base of the cartridge that contains the primer compound. The cup is conventionally made of a thin cartridge brass or cupronickel alloy, but arrangements including Ni-plated Cu, Zn-plated steel, or plain Cu cups are not uncommon [1]. In some instances, thin Al foil is present to seal the primer within the cup, and this too may be present in the resulting residue. Direct analysis of the residues retained in the primer cup following discharge has allowed the primer type to be identified, but has also found that the composition of the priming cup contributes to the muzzle blast residues [25].

The Cartridge Case

The cartridge case contains the primer charge, propellant, and the projectile, and elements present in its construction are often found as components in GSR. The most common casing material is brass, resulting in Cu and Zn being present in the resulting residues. Even though brass is the most common, steel, polymer-coated, and other cartridge casing constructions exist, any of which may result in components being detectable at a trace level in GSR [1, 25, 26].

Additionally, although GSR recovered from cartridge cases may be useful for providing a sample for comparison against one recovered from a suspect, the composition may be slightly different, requiring caution in interpretation. Cartridge case residues possess a chemical composition very close to that of the primer composition, while airborne residues possess more contributions from the bullet, jacket, and firearm construction materials [15].

1.3.3. Classification

Early research into the formation and characterisation of gunshot residues conducted by Wolten el al. [27] proposed a standard classification scheme for GSR to facilitate easier analysis and categorisation of residues. These categories were:

Characteristic – (Formerly designated 'unique' particles). Characteristic particles were only observed in the residues left behind following the discharge of a firearm, and were therefore considered 'unique' to gunshot residues. It should be noted that the use of the term 'unique' to GSR has since been revised, due to the fact that particles of these compositions have been found, in rare cases, to originate from sources other than firearms. This classification originally included:

- Pb, Sb, Ba
- Ba, Ca, Si with trace levels of S
- Sb, Ba.

The characteristic category allowed for minor or trace contributions from other elements, listed by Wolten et al. as: Si, Ca, Al, Cu, Fe, S, P (rare), Zn (Only if Cu>Zn), Ni (rare – only with Cu and Zn), K, and Cl [12].

Consistent – (Formerly designated 'Indicative'). This classification included particles that were prevalent in, but not necessarily unique to gunshot residue. As such, their presence alone was not considered conclusive evidence of a firearm discharge, but they may be used as supportive evidence when detected alongside characteristic particles.

This classification originally included:

- Pb, Sb.
- Pb, Ba.
- Pb.
- Ba (if S exists at trace level, or is absent.)
- Sb (rare)

The particles were further assessed on the basis of their morphology, with the bulk of them, 70-100% exhibiting spheroidal or globular morphology, occasionally with some distortions, or as an agglomeration of small spheroids. Most of the particles of spheroidal morphology had diameters of between 0.5 and 5 μ m. The remainder exhibited irregular morphology, existing as flattened plates, with no indicative crystal structure and occupied various sizes between 1 and 100 μ m.

While the classification system proposed by Wolten et al. [12] was valuable, further research demonstrated that metal residues are more prevalent than was initially suspected, and these particles were less 'unique' to gunshot residue than first thought. This resulted in the reclassification and exclusion of some particles from consideration as GSR as the discipline has evolved.

The current ASTM method [13] maintains most of the terminology first proposed by Wolten, has re-arranged the classification categories as demonstrated in Table 2 below.

Table 2 Concentration metaleny as per Astra 21000 17 [15]				
'CHARACTERISTIC'	'CONSISTENT'	'COMMONLY ASSOCIATED'		
	Pb, Ba, Ca, Si	Pb,		
	Ba, Ca, Si	Sb,		
Pb, Ba, Sb	Sb, Ba	Ba (S may be present)		
Pb, Ba, Ca, Si, Sn	Pb, Sb			
	Ba, Al			
	Pb, Ba			

Table 2 - GSR particle classification hierarchy as per ASTM E1588-17 [13]

Non-toxic or Pb-free ammunition generates its own types of residues, and therefore has its own classification levels, presented in Table 3.

Table 3 - GSR particle classification for Pb-free/non-toxic primer ammunitions as per ASTM E1588-17
[13]

'CHARACTERISTIC'	'CONSISTENT'
Gd, Ti, Zn	Ti, Zn
Ga, Cu, Sn	Sr

The most recent addition to the ASTM classification scheme is the inclusion of PbBaCaSiSn particles as a part of the Characteristic class. These were included in 2017, based on research performed by Zeichner and Levin [28].

The ASTM acknowledges that this set of classifications is not exhaustive and that unusual or atypical combinations of elements may be observed in casework. In such situations, the standard recommends that the compositions of the particles observed be compared against 'case-specific known source items, such as fired cartridge cases (FCC), or ammunition/weapon test fire deposits' [13]. It should be noted that some early classification schemes employed the term 'unique' as a descriptor for particles that fit within the category described as characteristic under the ASTM. In some jurisdictions, this terminology has been preserved. While the use of this term is highly jurisdiction specific, most GSR analysts recommend against

the use of the word 'unique' as a descriptor for this particle type, as the particles in this classification are not truly 'unique' to GSR, and may originate from sources other than a firearm discharge.

1.3.4. Primers

The primer is the primary source of inorganic GSR residues, with some minor contributions originating from the firearm, primer cap, jacket, or the bullet itself [10] as previously discussed. Primers used in ammunition are a mix of different energetic compounds, the exact components and relative proportions of which tend to be proprietary information and vary between manufacturers and production batches. Generally speaking, the fundamental components of a primer compound include an explosive component, an oxidising agent and a fuel source [1, 5], with different sensitisers, binders, and stabilisers added to modify the physical and energetic properties of the compound.

Among the most common primer residues observed in forensic casework are those based on the 'Sinoxid' formulation, which contains lead styphnate as the explosive component, barium nitrate as the oxidising agent and antimony sulphide as a fuel source with some frictionating properties [1]. Additional minor compositional components include lead peroxide as an oxidising agent and corrosion inhibitor, tetrazene as a sensitiser, and calcium silicide serving as both a fuel and frictionator compound [1]. The general composition of Sinoxid-type primers and approximate concentrations of each component can be observed in Table 4.

Compound	Concentration	Purpose
Lead styphnate	25 % - 55%	Explosive
Barium Nitrate	24% - 25%	Oxidising Agent
Antimony Sulphide	0% - 10%	Fuel
Lead peroxide	5% - 10%	Oxidiser/Corrosion Inhibitor
Tetracene	0.5% - 5%	Sensitiser
Calcium Silicide	3% - 15%	Fuel / Frictionator
Powdered Glass	0% - 5%	Frictionator

Table 4 - General composition of Sinoxid type primers [1].

Ammunition containing priming compounds with compositions like this results in the formation of the spheroidal three-component Pb, Ba, Sb particles that are considered to be characteristic of gunshot residue. However, it should be noted that, while the Sinoxid formulation is a commonly encountered primer, it is not the only primer formulation available. In 0.22 calibre rimfire ammunitions in particular, some of the primers used omit Sb compounds altogether. Therefore, GSR originating from this primer is expected to produce more particles Pb and Ba, while complicating identification of three-component particles [29]. While these are common, particularly in Australian casework, some manufacturers still use formulations based on standard three-component compositions [1], meaning that care must be taken by the analyst when interpreting GSR evidence. This has an effect on the interpretation of evidence, as per the particle classifications established in the ASTM standard, particles that do not contain Sb cannot be considered characteristic of GSR, and are therefore fall into the consistent category, which may originate from other environmental sources. This has the effect of making the evidence appear comparatively weak.

Another primer formulation worthy of consideration is the broad class of primers known as 'Non-toxic', 'Sintox' or Heavy Metal Free (HMF) primers. The typical composition of a Sintox type primer can be seen in Table 5.

COMPOUND	CONCENTRATION	PURPOSE
Diazodinitrophenol (DDNP)	~15%	Explosive
Tetracene	~3%	Explosive
Zinc Peroxide	50%	Oxidiser
Powdered Titanium	5%	Fuel
Nitrocellulose	27%	Fuel

 Table 5 - Typical Composition of a Sintox type primer compound, relative composition and purpose in

 the primer mix [1]

Sintox primers were created to address the problem of repeated firing of ammunition containing Sinoxid type primers resulting in significant exposure to airborne Pb and other heavy metals that may pose a risk to the health of those exposed. This is of particular concern in situations where shooters are occupationally exposed through their need to regularly fire weapons or be in proximity to firing weapons, as is the case with firearms instructors or firing range personnel. This type of exposure has been shown to be a concern at both indoor [30] and outdoor firing ranges [31]. Environmental surveys conducted of indoor firing ranges indicated that under certain circumstances, the airborne Pb concentration could reach levels nearly five times the recommended occupational health and safety limits [32]. Further investigations into the effect of airborne Pb exposure from firing ranges on blood Pb concentration has shown that persistent exposure leads to a cumulative increase in blood Pb concentrations above set occupational limits [30]. Research has shown that the composition of the primer residue is accountable for a relatively small contribution to the elevation of blood Pb levels due to the low concentration of Pb present in the primer [33]. The primary contribution to airborne Pb levels is from the projectile itself, and is therefore heavily affected by the type of bullet used.

Regardless, economic and safety considerations resulted in a push to substitute the use of Pb compounds in firearms primers, and lead to the adoption of a variety of 'non-toxic' or 'Pb-free' primers. One example of a common 'non-toxic' primer is the 'Sintox' formulation, which uses diazole (2-diazo-4,6-dinitrophenol) as the explosive component and a mixture of zinc peroxide and Ti metal as the oxidising agent and fuel source, respectively [34]. Other primers in the Pb-free and non-toxic families utilise different formulations that contain other elemental residues, such as Sr, Ti, Zn, or Al [35, 36] that are atypical to 'traditional' GSR. Certain specific munitions, designed for use by police in Europe, utilise Pb-free primers with the Sintox formulation that have been 'tagged' with specific elements (Gd, Ga, Ti) [37] to enable GSR to be directly linked to law enforcement [38].

The current ASTM standard [13] for the assessment of GSR defines particle types which may be considered as characteristic and consistent of GSR that originates from ammunition using 'non-toxic' primers as presented in Table 3. The ASTM standard acknowledges that beyond these classifications, additional elements may be incorporated into these particle types without precluding their classification as originating from a firearm source. The listed elements include AI, Si, P, S, CI, K, Ca, Fe, Ni, Cu, Zn, Zr and Sn [13].

While certain characteristic and consistent particles have been identified from non-toxic and Pb-free ammunitions, in some cases the residues are not significantly distinguishable from environmental particles. In these situations, a case-by-case approach, including an in depth examination of the firearms and ammunition under consideration is advocated to ensure that any residues that are recovered from the suspect may be definitively linked to the weapons and ammunition in question.

1.3.5. Propellants

Much like primers, the exact components of the propellant may vary between manufacturers, ammunition products and in some cases, between batches. The role of the propellant in the ammunition cartridge is to ignite when initiated by the primer, and react to generate the force which propels the projectile down the barrel.

An ideal propellant reacts predictably and readily on each initiation, is stable and not prone to degradation over time to ensure long-term performance. Further, different manufacturers

have individual recipes modifying the proportions and ingredients present to suit their product. To accomplish these goals, propellant mixes are typically a complex mix of compounds and other additives designed to modify the properties and performance of the powder [1, 10, 39]. While the energetic component of the mix may be considered the primary component of the mixture, it is far from the only important component. Other compounds are included as stabilisers, plasticisers, sensitisers, and flash suppressants. Well over 100 different organic compounds have been detected in propellants and are associated with GSR in the literature [40]. A collection of some of the more commonly encountered organic compounds associated with oGSR can be seen in Table 6.

Explosives	Stabilisers	Plasticisers	Flash Suppressants	Sensitisers
Ethylene Glycol dinitrate	Arkardite I (N,N'-diphenyl urea) Arkardite II (N'-Methyl-N N-	1,3-Benzenediol	Dinitrotoluene	Diethylene glycol dinitrate
Diethylene glycol dinitrate	diphenyl urea)	Camphor	2,2-Dinitro toluene	Tetracene
Nitrocellulose (NC)	Arkardite III (N'-Ethyl-N,N- dipheynl urea)	Diphenylamine	2,4-Dinitro toluene	Tetryl 2,4,6-Trinitrotoluene
Nitroglycerine (NG)	1,3-Benzenediol	2-nitrodiphenylamine	2,6-Dinitro toluene	(TNT)
1,2-Dinitroglycerine	Cresol	4-nitrodiphenylamine	3,4-Dinitro toluene	Petaerythritol tetranitrate (PETN)
1,3-Dinitroglycerine	Dinitro-ortho-cresol	4-nitrosodiphenylamine	2-Amino-4,6-dinitrotoluene	
2-Nitrotoluene	Diphenylamine (DPA)	N-nitrosodiphenylamine (NNDPA)	4-amino-2,6-dinitrotoluene	
3-Nitrotoluene	N-nitrosodiphenylamine (NNDPA)	Diethylene glycol dinitrate	2-Nitrotoluene	
4-Nitrotoluene	2-nitrodiphenylamine	1,3-Diacetyloxypropan-2-yl acetate	3-Nitrotoluene	
2,4,6-Trinitrotoluene	4-nitrodiphenylamine	Etylcentralite	4-Nitrotoluene	
Nitrobenzene	4-nitrosodiphenylamine	Methylcentralite	1,3-Diacetyloxypropan-2-yl acetate	
1,3-Dinitrobenzene	Etylcentralite	Butylcentralite	Nitroguanidine	
1,3,5-Trinitrobenzene	Methylcentralite	Methyl phthalate	Potassium Nitrate	
Petaerythritol tetranitrate (PETN)	Butylcentralite	Ethyl phthalate	Potassium Sulfate	
Octahydro-1,3,5,7-tetranitro- 1,3,5,7-tetrazocine (HMX) Hexahydro-1,3,5,7-trinitro-1,3,5-		Dimethyl phthalate		
triazine (RDX)		Diethyl phthalate		
		Dibutyl phthalate		
		Diamyl phthalate		
		Glyceryl triacetate		

Table 6 - Listing of commonly encountered organic compounds and their functions in propellant that may contribute to OGSR. [1, 10, 39-42]

The energetic or explosive component is the primary driver behind the reaction in the cartridge. When initiated by the primer, this component undergoes rapid combustion under confinement, generating large amounts of gas, and ultimately increasing the pressure within the firearm to the point that the projectile is propelled from the barrel. Most commonly, nitroglycerine (NG) and nitrocellulose (NC) are used for this role [1, 10]. Broadly, ammunitions may be classified as either single, double, or triple base, depending on their primary energetic components. Single base propellants rely on Nitrocellulose (NC) as their primary energetic component. Double base propellants contain NC, but also include Nitroglycerine (NG). Both single and double base propellants retain both NC and NG, but include Nitroguanidine as an additionally component. Triple base propellants are not used in small arms ammunitions, and are most frequently utilised in vehicle borne projectile weaponry, such as naval and tank cannons, or artillery.

Stabilisers are included to prolong the shelf life of the propellant, by neutralising decomposition products, and inhibiting further degradation of the explosive compound. They may also be included to increase the chemical stability of the energetic component. Commonly included for this purpose are, ethyl centralite, methyl centralite, or diphenylamine or its derivatives [1, 39, 41]. Plasticisers are compounds that are included to improve the handling characteristics of the propellant granules. The size and shape of propellant granules is important, as this will impact factors such as the burning rate of the powder, which contributes the predictability and consistency of the ballistic performance of the ammunition. Plasticisers assist this by modifying the physical characteristics of the propellant, allowing for propellants to be more readily worked into a specific shape, as well as assisting in retaining that shape. They may also serve the further purpose of acting as an additional fuel source during combustion. Commonly encountered plasticisers include phthalates as observed in Table 6. Flash suppressant compounds are included to reduce the size and brightness of the muzzle flash, which is desirable as excessive flash may result in making the shooter's position easier to identify, or compromising the shooter's vision. Sensitisers may be included as a component of either the primer or powder, and modify the reactivity of the compound. When present in the primer, they increase the sensitivity of the primer to percussive force, meaning they more reliably ignite upon impact of the hammer.

While some organic components of the propellant or primer may contribute to the elements present in iGSR particles, for the most part, residues generated from the organic components

are separated into the field of oGSR analysis. While a familiarity of the basics of the organic components that may be present in GSR is instructive, analysis of these components relies on different analytical and instrumental techniques and processes that are beyond the scope of this thesis.

1.4. METHODS OF GSR ANALYSIS

A number of different techniques have been used in the analysis of GSR. The 'gold standard' for GSR analysis since the publication of the Aerospace report in the late 1970s [12] has been scanning electron microscopy coupled with energy dispersive x-ray spectroscopy (SEM-EDS). SEM-EDS is valued as an analytical technique for GSR as it is capable of operating in backscattered electron mode (BSE), in which the brightness of a particle in the field of view is proportional to its atomic number (Z). This results in high contrast between high Z GSR particles, which appear bright in the field of view, and a low Z carbon tape background which appears darker. This makes the process of identifying GSR particles in a sample much easier. Once particles are identified, SEM-EDS is capable of assessing their chemical composition and morphology simultaneously, and therefore is highly selective for GSR. Many environmental sources independent of firearms discharge have been observed to generate residues that contain Pb, Ba and Sb as separate components, but incorporation of these three elements into a single spheroidal particle is still considered as a characteristic indication of GSR. As both composition and morphology are important to the identification of GSR, SEM-EDS is capable of providing the most unequivocal identification of GSR.

Historically, a variety of methods used in the analysis of metals have been applied to the instrumental analysis of GSR. These have included atomic absorption spectroscopy (AAS), Neutron activation analysis (NAA), Inductively coupled plasma mass spectrometry (ICP-MS), time-of-flight secondary ion mass spectrometry (TOF-SIMS) and X-ray fluorescence (XRF) [1, 10, 42, 43]. While these techniques are able to provide comprehensive elemental analysis of samples, the major limitation of these techniques has been that they preclude the examination of particle morphology. Therefore, such bulk analysis methods will return a positive indication of GSR in situations where Pb, Ba and Sb are detected in the same sample. However, in the identification and classification of GSR, it is not only the elemental composition that is of consequence, but the composition and morphology at the individual particle level. A sample containing discrete particles of Pb, Ba and Sb separately could potentially be assessed as positive for GSR by a bulk analysis method. However, it is possible that these three individual particle types may be present on an individual who has not necessarily fired a gun. Bulk analysis techniques therefore have the potential to increase the risk of type 1 (false positive) errors in analysis and should not be relied upon for comprehensive GSR analysis.

Initial investigations into the use of SEM-EDS for the analysis of GSR identified the need to assess both composition and morphology [11, 12, 44]. In these initial stages however, the technique was labour intensive, requiring an analyst to manually search the stub and identify any particles of potential interest. As the field has evolved along with technology, automated software packages that allowed for the automatic searching of the stub were introduced. These allowed for the system to search the stub and flag any particles of potential interest automatically, and required the operator only to review the results and re-acquire spectra for any particles of interest. As technology has progressed, SEM-EDS systems have been improved, allowing for the incorporation of faster detection systems (such as silicon drift detectors), and further improvements to control and analysis software [45].

For this reason, SEM-EDS has been considered by many practitioners to be the ideal technique for the detection, examination, and analysis of GSR [37]. As a technique, SEM-EDS has the further advantages of requiring a relatively small amount of sample preparation. Samples to be assessed for GSR can be collected directly from surfaces by way of an Al SEM pin stub topped with carbon adhesive tape. This can then be carbon coated to improve sample conductivity if required, before being loaded directly into an SEM. Further, this form of sample analysis is minimally damaging to the specimen, meaning that the sample can be retained for future analysis by SEM, or through a different technique if required. As it is the most frequently used technique for the analysis of GSR, the bulk of the research reported in this thesis has been conducted with SEM-EDS as the primary analytical technique.

1.5. SOURCES OF 'GSR-LIKE' PARTICLES

1.5.1. Cartridge operated industrial tools.

A number of industrial tools, including nail-guns, other industrial fasteners, and captive bolt pistols make use of a primer and propellant explosive cartridge as a means of operation. These cartridges are similar in construction of blank firearms cartridges; the projectile is absent, and the powder load is secured within the cartridge and initiated by a primer. These cartridges exist in both centrefire and rimfire arrangements and may utilise both two and three component primers. While cases where these tools are used as the primary weapon in a suicide or homicide have been documented [46, 47], the fact that they utilise the same cartridge composition as some firearms also presents the more important possibility that residues generated by these tools may be indistinguishable from GSR. Unlike other possible origins of 'GSR-like' particles, this source makes use of both an explosive primer and propellant that is compositionally and functionally identical to the type used in firearms. Practically, it is an ammunition cartridge sans projectile, and therefore could be expected to generate a similar primer particle population to a firearm discharge.

In initial work conducted on GSR, Wolten et al. investigated the possibility of cartridges used in industrial tools generating particles that could not be distinguished from GSR [48]. Two main styles of construction for such cartridges exist - 'wad' type or 'crimped' type. 'Wad' type construction has the appearance of a traditional ammunition cartridge, but with a paper or plastic 'wad' secured where the projectile would ordinarily be. In 'crimped' type construction, rather than replacing the projectile with a wad, the open end of the cartridge casing is crimped into a fluted cone. Both styles of cartridge operate by placing some type of industrial fastener (e.g., a nail, rivet, or staple) in the path of the cartridge. The cartridge is then discharged in a similar fashion to a firearm, with the delivery of a shock to the primer compound, initiating the propellant, which then drives the projectile. Wolten et al. used exemplars of both types of construction to drive a Zn-coated steel projectile, and then provided the samples blind to an experienced GSR examiner [48]. They concluded that although the discharge of cartridges from industrial tools can produce particles with spheroidal morphology, and with compositions similar to GSR, there were indicators suggestive of an alternative source. Notably, they specify that of the particles observed that did possess compositions similar to GSR, there were indications of a non-firearm origin such as high levels of Fe, or an anomalous Cu:Zn ratio (low Cu:High Zn) which are considered inconsistent with GSR. Finally, they note

that the percentage of spheroidal particles was low, and that the limited number of Pb-only and particularly, spheroidal Pb particles also indicated an alternative source. If familiar with these characteristics, it is likely that a trained examiner could exclude a firearms source. From an evidence evaluation standpoint, Wolten et al. suggested that the presence of substantial numbers of inconsistent particles should outweigh the evidential value of a small number of particles consistent with GSR [48].

Further studies into the composition of the residues generated from the discharge of cartridge actuated industrial tools conducted by Wallace & McQuillan [49] generally agreed with the conclusions of Wolten et al. in that these tools generated some particles that had the potential to be classified as consistent with, and characteristic of GSR [48]. Wallace and McQuillan however, considered a wider range of cartridges from different manufacturers, consistent with what was available in their specific jurisdiction [49]. Specific cartridges were found to produce both the three-component PbBaSb particles considered characteristic to GSR, as well as SbBa, BaCaSi, PbBa and Pb particles that would be considered consistent with GSR under the current ASTM definition [36]. Morphologically, the particles were found to be in the range of 1-12 μ m, and possessed both spheroidal and irregular morphology consistent with condensing from vapour [49]. These findings were later supported by subsequent investigations by Zeichner and Levin [28] and Garafano et al. [50].

However, while the residues originating from cartridge operated industrial tools were morphologically indistinguishable and exhibited some compositional similarity to GSR, a few key points of difference exist. In the analysis of their data, Wallace and McQuillan classified particles as inconsistent with GSR on a number of different criteria [49]. In agreement with Wolten et al. [48], they noted:

- Particles containing Zn in the absence of Cu, or Zn at levels higher than that of Cu.
- Low numbers of spheroidal Pb-containing and Pb-only particles.
- A significant presence of Fe in the particle population.

They also observed the following in particles, considered inconsistent with GSR:

- Particles containing Cr.
- Particles containing Mn.
- Particles containing Ni.
- Particles that exhibited a very high background.

It should be noted however that while particles containing Ni were classified inconsistent with GSR in this study, this runs contrary to the current ASTM classification guidelines, which allows

Ni to be incorporated into GSR without excluding the particle [36]. This study therefore excluded particles as inconsistent with GSR that would not necessarily be excluded under the current classification. Analysis of their results indicates that this only resulted in the exclusion of approximately 38 particles, representing approximately 0.9% of total particles found (1.2% of excluded particles), and would likely produce no significant impact on their interpretation.

Both Wolten et al. and Wallace and McQuillan noted that the bulk of the cartridge tool discharge particles contained Fe at either a major, minor or trace level [48, 49]. This is uncommon, but not unheard of in GSR, and is often the result of poorly maintained firearms affected by rust or steel-case ammunition [51]. Conversely, a similar study on cartridge operated industrial tools conducted by Garafano et al., noted 50% of the recovered characteristic particles in their survey were found to not contain any Fe, with the remainder only containing Fe at trace levels [50]. Under the ASTM classifications, this would not exclude any of these particles as characteristic of GSR. In this study however, the sample size was much smaller than that of the Wallace and McQuillan study, (6 particles from 7 samples originating from one type of cartridge versus 4237 particles from seven samples originating from seven types of cartridges). It should be further noted that the Wallace and McQuillan samples were collected following ten instances of discharge, while the Garafano et al. samples were collected following a single discharge [49, 50]. While the Garofano et al. study [50] provides further valuable data regarding the assessment of GSR, the broader scope of the Wallace and McQuillan study increases the confidence in their conclusions.

In their entirety, the criteria noted by Wallace and McQuillan resulted in the exclusion of nearly 75% of the overall particles detected, indicating that even if individual particles superficially indistinguishable from GSR were found in a sample, they could be excluded as being the result of a firearm discharge when considered in the context of other particles in the population [49]. The conclusion drawn by Garofano et al. is that while it is possible that particles indistinguishable from GSR will be generated from the discharge of cartridge operated industrial tools, careful examination should allow them to be excluded on the basis of other particles present in the sample, although they neglect to make reference to specific particle types that they observed in their analysis that permitted this [50]. Wallace and McQuillan support a similar approach, establishing clear exclusion criteria, advocating the consideration of particles in context, and warning against placing importance on small numbers of particles [49]. Both of these findings are in agreement with the prior study by Wolten et al., and echo the recommendation that the overall particle population be considered in any assessment of GSR [12]. All of the groups of researchers expressed particular interest in the fact that cartridge operated tool discharge exhibited a significant lack of Pb only particles with morphology typical of firearms discharge. This finding supports the suggestion that a greater number of particles of Pb present in GSR originate from the surface of the projectile, rather than from the primer itself [33]. This provides further information regarding the whole particle population which may be pertinent to a GSR examination.

While the structure and function of cartridge operated industrial tools means that there is a possibility that particles indistinguishable from GSR may be generated from their use, the probability of encountering an individual exposed to these tools is also of relevance. Over time, reliability and occupational health and safety concerns have resulted in a reduction in the popularity of this type of industrial fastener. More recent research has suggested that it has become harder to source cartridge operated industrial fasteners, with industry exhibiting a preference for pneumatic or electronically actuated nail guns [52]. This survey conducted on some of the currently commercially available industrial tool cartridges in Australia has indicated that the recommendations of Wolten et al. and Wallace and McQuillan still apply [48, 49]. Further, this study indicated that although residues originating from cartridges used in cartridge operated industrial tools can produce particles that are indistinguishable from particles generated from 0.22LR ammunition, the generation of significant quantities of these particles is unlikely. Analysis of the particles generated from discharge of cartridge operated industrial tools suggests that while there may be compositional similarities, because of the system of operation of cartridge operated tools, the morphology of the particles generated is likely to be different. Specifically, in evaluating over 40,000 particles from the hands of individuals who had just discharged several cartridges through a cartridge operated nail gun, only one particle (0.002%) was found to have Pb, Ba, and Sb present. However, this single particle had a morphology that was inconsistent with a firearm source, leading to its exclusion. Further, an abundant quantity of PbBa particles (21,170; 38.5%) and BaCaSi particles (3926; 7.1%), which fall into the consistent category were observed, but a majority of these also showed morphological inconsistency with GSR. It is thought that these inconsistencies in morphology may be attributable to the different mechanisms of operation of nail guns as compared to conventional firearms. Though not explored in detail, it is hypothesised that the internal pressures reached during the discharge of a nail gun are not as high as those experienced during firearm discharge, which may result in the difference in morphology. This study further indicated that although it is possible that currently commercially available industrial tool cartridges in Australia can theoretically produce particles similar to GSR, they

are likely to be small in number, and can be excluded on the basis of the wider particle population, morphology, or the inclusion of elements considered atypical in GSR.

Ultimately, the current state of the research suggests that although there is a small chance that cartridge operated industrial tools will generate particles indistinguishable from GSR, it is relatively unlikely that they will represent a major consideration for the assessment of GSR evidence. Pneumatic and electrically actuated nail guns are becoming more favoured by industry, making cartridge operated tools increasingly uncommon. Even when used, the likelihood of generating significant numbers of particles indistinguishable from characteristic GSR particles appears to be low. However, it is worth acknowledging that if a cartridge operated industrial tool is suspected of contributing to a particle population, there are some indicators that may support this hypothesis. A notable absence of spheroidal particles, specifically those consisting of Pb only, has been noted in particle populations originating from industrial tools. Similarly, these tools tend to generate populations of particles that contain significant numbers of Fe only, or Fe-containing particles as well as particles containing Zn in the absence of Cu, or with Cu at a level lower than that of Zn. These particle types are uncommon in GSR, and may be an indication that a cartridge operated tool may be a contributor. Finally, as in most situations, the context of the specific circumstances under investigation must be considered to determine if a cartridge operated industrial tool is relevant to the assessment.

1.5.2. Brake pad residues

Brake pads are the friction surfaces that are applied to the brake disc during the braking of a car, truck or motorcycle. Application of the brakes creates friction between the pads and the disc, resulting in the deceleration of the vehicle. The compounds utilised in the construction of all parts of the brake assembly, including the pads, calipers and discs vary depending on the type and manufacture of the vehicle, and the specific braking characteristics that are required. Asbestos was used as a friction compound in brakes until health concerns led to legislation banning its use in the 1980s. In the wake of the ban, a number of new friction compounds were implemented as replacements, including lead sulphide, antimony sulphide and barium sulfate [53]. Antimony trisulphide is often included as a solid lubricant, while barium sulphate is added to the mix to regulate the heat stability of the friction materials [54]. During the braking process, the temperature of a disc brake system in areas of the friction surface may reach temperatures in excess of 1500°C [53], which is comparable to the conditions around the formation of gunshot residues. At the same time, the wearing process may result in the

combination of components of the friction materials at high temperatures, which may result in the incorporation of elements relevant to GSR analysis into single particles [53].

Initial studies of particles originating from brake pads was conducted by Wolten et al., in which the hands of automotive brake mechanics were sampled and assessed for their particle populations [48]. They found small numbers of particles morphologically and compositionally consistent with GSR present on the hands of brake pad mechanics, specifically those with compositions PbBa, with other elements present. However, they note that the relative abundance of the other elements present in these particles (such as Fe, S, P, Ca) were at levels not frequently observed in GSR, and were therefore inconsistent with GSR. It is worth noting that at the time this study was performed, many vehicles still operated using leaded petrol, leading Wolten et al. to comment on PbBr particle types as being characteristic of exhaust from these vehicles [48]. Researchers in this case also observed that Pb-containing particles originating from exhaust were present in large numbers on the hands of mechanics, as well as anyone that had ongoing contact with vehicles, including truck drivers and other motorists. However, although Pb was abundant in these samples, the authors note that no particles relevant to GSR were noted in these samples [48]. In the time since, leaded petrol has been replaced in the vast majority of vehicles, which has further reduced environmental sources of Pb.

Garofano et al. performed further studies into the probability of observing particles indistinguishable from GSR by sampling the hands of mechanics, brake mechanics, and others with contact with vehicles, as well as sampling from disc brake hubs directly [50]. Their research found that BaSb particles may originate from automotive sources such as brake pads, and therefore could not be considered characteristic of GSR. Although some of the particles observed had irregular and flaky morphology allowing them to be differentiated from GSR, the authors noted that in some circumstances, particles indistinguishable from GSR were generated. Consequently, the classification of this particle type was revised, moving them from characteristic of GSR, to consistent with GSR. Garofano et al. [50] did however corroborate the analysis of Wolten et al. [48] that PbSbBa particles are characteristic of GSR, and were not observed in automotive sources [48]. Contrary to this however, a study performed by Cardinetti et al. found a small number of PbSbBa particles originating from the brake pads of an Audi A4 [55]. These particles contained minor amounts of Fe, but would not be compositionally excluded under the ASTM. However, morphologically, these particles appeared rough and flaky, and did not show complete incorporation of all three elements into a single spheroidal particle. To that end, general agreement was found in the suggestion of both Garofano et al. [50] and Wolten et al. [48] that both the compositional and morphological characteristics of the particles under consideration were important in the assessment of the origin of GSR particles.

A more comprehensive study of brake pads conducted by Torre et al. examined the relationship between brake pads and their residues, as well as the potential for misidentification of such residues as GSR [53]. Their study analysed samples of:

- 'Road dust' collected from the front rims of 40 different models of car,
- New brake linings that had been crushed.
- Samples stubbed from the surface of new brake linings
- Debris generated from new brake linings after being worn with a rotary grinding tool.

They were able to isolate several particles with sizes and elemental compositions both characteristic and consistent with GSR. However, similar to the observations of Garofano et al. [50] and Wolten et al. [48], a consideration of morphology indicated that particles generated from brake pads often presented as irregular, rough, or dusty, excluding it from classification as GSR. Elementally, the composition of the particles sampled from brake pads in the Torre study fell into two broad classes that the authors categorised as 'clean' and 'unclean' [53]. Clean particles were those that were compositionally similar to GSR, containing at least one primary element at a major level, with no extraneous elements inconsistent with GSR at a high level. All other particles were classed as unclean, in that they contained Pb, Sb and Ba, but also contained elements inconsistent with GSR. Much like the particles identified from cartridge operated industrial tools [49], the unclean particles that did contain Pb, Ba or Sb also contained Fe at a major level or other elements that would exclude these particles as originating from a firearms discharge. The Fe contamination was more abundant in samples of road dust, but was still present, albeit at a lower level in samples obtained from new brake pads. Mg was also commonly detected at minor and trace levels, which is not permissible in GSR under the ASTM definition[13]. These observations were in support of earlier work that identified particles originating from disc brake hubs as containing either Ba or Sb residues with a major Fe component along with minor Mg contribution [50]. In both of these studies, the particles recovered also possessed irregular morphology which is considered uncharacteristic of GSR.

Even in the case of the clean particles found in the Torre study, a number of the particles contained Mg at trace levels [53], which is rare in primer residues [1]. Further, S often occurred at a major level, which is typically inconsistent with GSR. Torre et al. make the point that while it is possible to find environmental particles that are indistinguishable from GSR on the basis of composition and size, they have a distinct morphology, and therefore a cautious interpretation of both composition and morphology is warranted. Further, they advocate for extreme caution in drawing conclusions to be followed when a cartridge case or firearm is not available for comparison. These suggestions echo the recommendations of Garofano et al. [50]

Torre et al. [53] also support the suggestion initially made by Wallace and McQuillan [49], that the terminology 'Primer discharge residue' replace the term 'GSR' [53]. This change in terminology incorporates residues generated from the discharge of blank cartridges, cartridge operated industrial tools, fireworks, and stud guns alongside those generated from a firearm discharge into the same category. This was originally considered under previous versions of the ASTM, where the terminology 'unique' was used to refer to PbSbBa and SbBa particles. These particles are not 'unique' to GSR, as they can originate from any discharge of a Pb, Sb, and Ba based priming compound, thus the term 'primer discharge residue' is more specific. However, the 'unique to GSR' category was revised to 'characteristic of GSR', rendering this change in terminology less important.

Following the United Nations declaration on the risk of lead exposure in 1996, many countries implemented legislation to reduce the use Pb-compounds in consumer products [56]. This resulted in a reduction in the use of Pb in brake pads and assemblies due to the potential health effects of Pb exposure. It would therefore be expected that as the numbers of older cars on the road decreases, the potential for brake pad residues containing Pb would also be reduced. The Torre et al. study sampled road dust from a number of different vehicles, but categorised their results based on country of origin, rather than make, model or year of manufacture, making an assessment of data based on year of manufacture impossible [53]. The studies conducted into the prevalence of GSR type residues originating from brake assemblies have been conducted primarily in Europe [50, 53] or the United States [48], and are not particularly representative of the environment present in Australia. Further, these studies were either conducted prior to, or relatively shortly after, regulation imposed to remove Pb-containg compounds as a component in brake pads. In order to provide a context around the background prevalence of GSR-like particles in road dusts and on vehicles, a study was conducted to analyse a number of samples from cars on Australian roads. Tucker et al.

performed a survey of commercially available brake pads and the hands of mechanics in an Australian context, in order to assess the relevance of more modern brake pads to the evaluation of GSR evidence [54]. This study assessed 75 brake pads, comprising both Original Equipment Manufacturer (OEM) and aftermarket pads to determine the prevalence of GSR relevant materials. Samples were first screened for elements of interest, resulting in a subsample of 12 pads which were identified for further analysis by SEM-EDS. Additionally, 11 samples were collected from the hands of mechanics that had recently handled brake pads or wheels, 3 samples directly from brake rotors, and 22 samples from wheel rims. Across the sample set, no particles characteristics of GSR were detected. Although a number of consistent particles were detected, specifically those containing Sb and Ba, the prevalence of Pb in all samples was low. This suggests that the restriction of Pb in consumer products has been successful, making the likelihood of observing PbSbBa particles from brake pads even more remote. Further, similar to previous studies on this topic, the morphology of the particles was often rough, angular and dusty, with very few spheroidal particles, or particles exhibiting a molten morphology. The authors conclude that the probability of observing particles originating from brake pads that are indistinguishable from GSR is low, perhaps even lower than suggested by previous studies. They do however agree with previous authors in the suggestion that a cautious and thorough review of particle morphology is necessary to avoid misclassification.

Taken as a whole, the literature suggests that although there is a remote possibility that brake pads may be a source of particles indistinguishable from GSR, it is unlikely that they will be observed frequently in case-work. Compositional and morphological differences are evident, allowing for these particles to be differentiated from those originating from a firearms discharge. Similarly, the wider particle population would provide indication that dust originating from brake pads may be relevant to the interpretation.

1.5.3. Firework and other pyrotechnic residues

Potential environmental sources of GSR-like particles also include fireworks and other pyrotechnic reaction residues. Fireworks and pyrotechnics utilise a similar composition to ammunition primers in order to ignite and detonate, containing a mixture of explosive compounds, fuels and oxidising agents. Further, metallic compounds are often included as a means of manipulating the size, colour, and characteristics of the display. Particles generated in the aftermath of a fireworks display have been exposed to conditions that are comparable to those seen in the discharge of a firearm. Ignition and reaction generates high temperatures, with components often under pressure, resulting in the distribution of vaporised elemental components into the atmosphere, which then mingle, agglomerate, and solidify on cooling.

Studies into the similarities between GSR and pyrotechnic reaction conducted by Kosanke et al. [57] indicate that the residues share the same means of production (energetic exothermic reaction), size domain, and gross morphology, but frequently differ in elemental composition compared to GSR. Specifically, pyrotechnic residues typically possess a wider range of different elements and elements of relatively low atomic number. Further, the authors of this study also note that pyrotechnic reaction residues are typically generated in numbers several orders of magnitude higher than typically observed in GSR. Taken together, data show that fireworks have the potential to generate a large quantity of particles that are morphologically similar and of a similar size to GSR, but may be differentiated on the basis of chemical composition. It is however, worthy of note that the differences in composition observed specify that the pyrotechnic reaction residues observed in this study contained lighter elements, such as Al, Ti, and Sr, and were therefore distinct from characteristic GSR containing Pb, Ba, and Sb [57]. However, since this study was conducted, there has been a rise in the popularity of nontoxic/Pb-free/Heavy metal free (HMF) ammunition formulations, in which lighter elements are more prevalent. Specifically, the current version of the ASTM considers Sr particles, and particles containing Ti and Zn as consistent with Pb-free or non-toxic primed ammunitions [13]. Due to this, the potential for fireworks to generate particles similar to GSR, specifically HMF or Pb-Free formulations, is worthy of further consideration.

Understanding the likelihood that particles considered characteristic of or consistent with GSR will be generated from fireworks informs the possibility that a false positive result will be recorded. A study performed by Garafano et al. considered a small number of samples (4) taken directly from the hands of pyrotechnic technicians and fireworks experts [50]. At least 11 characteristic particles were found from these samples, with 'many' (number not specified) particles of BaSb detected. While they were able to detect small numbers of both particles characteristic of, and consistent with GSR with comparable sizes to GSR, morphological examination precluded them from classification as GSR. The particles found were irregular, partially molten, and contained distinct zones of only Sb or Ba, with no significant integration seen [50]. The authors did not further specify the other particle types that were detected on the hands of the pyrotechnic technicians, so no further assessment of the relevance of the particles to Pb-Free or HMF ammunition can be made. Based on their research, the authors

concluded that PbSbBa particles were characteristic of GSR, but BaSb particles were present in other sources.

A more detailed study performed by Trimpe assessed the possibility of particles indistinguishable from GSR being generated from fireworks by analysing both unburned and burned samples [58]. A total of 148 different fireworks were analysed, with both the bulk unburned components and a sample collected from the burned remnants for each assessed by SEM-EDS. The results of this assessment indicated that of the 148 different firework samples, 117 (79%) contained at least one of the three elements (Pb, Sb, or Ba) of interest to GSR analysis. From this, 105 (71%) contained Ba, 46 (31%) contained Pb, and 9 (6%) contained Sb. Though not reported in detail, it is noted that K was almost always present, and Cl, S, Si, Mg and AI were frequently observed, while Ti, Sr, and Zn were only found occasionally. When morphology was considered, only 68 (46%) produced particles with spheroidal morphology in the 1-5 μ m range. Further assessment indicated that two of three elements were only present in 29 of the 148 fireworks (20%), and none of the firework samples assessed had all three components present. Of the 29 samples with two elements present, 11 could be excluded on the basis of the particle morphology, or the presence of magnalium (a 1:1 mix of Al_3Mg_2 and Al₂Mg₃). Ultimately, it was observed that only 18 of the 148 fireworks (12%), generated particles considered consistent with GSR on the basis of both composition and morphology. The fact that none of the samples assessed had all three components present in a single firework suggests that by themselves, none of the fireworks in this study had the ability to generate three-component particles characteristic of GSR. The results do suggest that ingredients containing all three components are used in the manufacture of fireworks, and therefore it is not beyond the realm of possibility that all three-components could be present in a single firework. This finding also suggests that a fireworks display, in which many different types of fireworks are detonated in close proximity, has the potential to generate a particle cloud that contains all three components as well as generating particles consistent with GSR.

This possibility was further explored in a study performed by Grima et al., in which a number of particles exhibiting elemental compositions and morphologies consistent with GSR were detected in samples collected from the environment around a fireworks display [59]. This study had the benefit of a sample size larger than the Garofano et al. [50] (Over 2000 particles identified from 20 samples with 141 particles containing an elemental composition similar to GSR). Further, unlike the Trimpe study [58], these samples were collected through environmental sampling over the course of a two-hour fireworks display. It would be expected therefore that the number of particles collected would be greater, and they are more likely to collect a wider population of particles. It is likely that the particles found in the Garofano et al. study were a result of handling fireworks prior to ignition [50], and those in the Trimpe study consisting of samples from handling fireworks in the aftermath of ignition [58]. The Grima et al. study collected residues from the particle cloud in the aftermath of multiple firework ignitions [59]. Studies of pre-ignition and post-ignition pyrotechnic residues using SEM-EDS has indicated compositional and morphological changes to residues occurring as a part of the process [60], suggesting that a direct comparison of the particles found by Garofano et al. [50] and Grima et al. is not valid. However, as both Grima et al. and Trimpe considered postignition residues, a comparison is more relevant. The particles in the Grima et al. study considered significant to GSR analysis represented a relatively small proportion of the total number of particles collected [59]. Of the more than two thousand particles collected, 141 particles were elementally profiled due to the presence of elements usually seen in GSR. The authors note in particular that in their sample, residues containing Ba, Sb, and Sr were frequently observed, while Pb, Ti, and Zn were comparatively less common. At the largest of three sites considered in this study, Ba-containing particles were present in over 50% of the samples collected, while Sb- and Sr-containing particles were both present in over 20% of the samples collected. Comparatively, Pb-containing particles were observed in approximately 12% of samples, while Ti- and Zn-containing particles were present in less than 10% of particles observed. From the same site, more than 75% of the particles containing heavy metals present in GSR contained K, while more than 80% contained S. This finding is consistent with previous studies performed by Mosher et al., which suggest that the prevalence of Bacontaining particles is due to the wide application of Ba compounds in pyrotechnic applications, while Pb and Sb compounds are comparatively less common [61]. This was also supported by the survey by Trimpe [58]. It was also noted by Grima et al. that Mg and Na were often present in fireworks particles, despite being infrequently seen in GSR [59]. Na was present in approximately 60% of particles, while Mg was observed in greater than 20% of particles. These two indicators can assist in discriminating residues generated from firearms and fireworks. When considering the composition of the observed particles, of the 141 particles identified as having similarities to GSR, 121 were excluded under the conditions of the ASTM standard, or on the basis of elements uncommon to GSR, leaving 12 particles that were considered to be 'GSR similar' on the basis of both morphology and composition. Of these 12, only 5 fell into the range of $0.5 - 5 \,\mu\text{m}$ typically seen in GSR and were thus deemed indistinguishable from GSR. While the authors do not specify exactly how many particles were

collected in total, the 5 particles indistinguishable from GSR represent 3.5% of the 141 particles with superficial similarity to GSR, and less than 0.5% of the overall particles collected, assuming a minimum of 2000 total particles. This suggests that while significant exposure to fireworks residues may result in the transfer of a small number of particles that are indistinguishable from GSR, the presence of a wider particle population that is inconsistent with GSR should enable an analyst to differentiate between true GSR and Fireworks residues. In order for a false positive identification to occur with regards to GSR particles, the small percentage of particles originating from fireworks, but compositionally and morphologically similar to GSR, would have to be selectively retained on the hands of an individual while the wider particle population is lost. This specific situation is relatively unlikely, as there is no recorded mechanism that suggests that particles with GSR composition and morphology would be selectively retained over other comparable residues. Ultimately however, Grima et al. did not detect any particles originating from fireworks that are considered characteristic of GSR, and found a small number of particles that had composition and morphology consistent with GSR. These particles represented a minor subset of the overall population [59]. This finding is consistent with what was previously reported by Trimpe [58].

A more recent study, conducted by Seyfang et al. [52], broadly agrees with the conclusions of Grima et al. [59]. Seyfang et al. evaluated the hands of fireworks technicians and observers, as well as spent firework components and surfaces less than 3m away from the firework launch area. A total of over 100,000 individual particles were identified using GSR search software across 20 total samples, with more than 67,000 being classified by the software. Of this sample, no particles characteristic of GSR were found. A total of 366 particles considered consistent with GSR were identified, representing 0.4% of the total particle population. Of these consistent particle types, the most frequently observed composition was BaCaSi, with BaAl being the next most common. When considering elements most pertinent to the analysis of GSR, in agreement with Grima et al. [59] Ba-containing particles were the most frequently observed in the survey (4.5% of the total particle population), owing in large part to a significant population of BaS particles. Pb-containing particles were the next most commonly observed (0.9%), with Sb-containing particles being relatively infrequently observed (0.08%). Further, it was noted that there were large numbers of particles containing either Fe or KCl, particularly on samples from firework technicians' hands. This supports the prior observations of Grima et al. [59] and is consistent with the use of KClO₄ as an oxidising agent in many fireworks [62]. Additionally, the presence of Mg, particularly in combination with Al was observed in a number of particles. Mg is not frequently present in primer mixes, but is seen in

fireworks, where it likely originates from the presence of magnalium. Magnalium is a mix of Al₃Mg₂ and Al₂Mg₃ and is used in fireworks to increase the brightness of colours, the production of bright white sparks [63], and a crackling effect [58]. Morphologically, most of the particles that exhibited compositional similarity to GSR could be excluded on the basis that they appeared angular or flaky, and lacked the spheroidal or globular morphology that indicated that they were the result of condensation. A small sub-set of the particles were reported as having rounded or spheroidal morphology, but could be excluded due to the fact Fe was present at a major level. The authors conclude that all three elements considered characteristic of GSR were present in the samples they observed, suggesting that there is at least a theoretical possibility that all three elements could be present in a single particle. However, as particles containing these characteristic elements represent a minor proportion of the overall particle population, the authors conclude that this would not be a likely occurrence. Similarly, the observed differences in morphology, inclusion of elements uncommon in primer GSR, and broader particle population suggest an overall agreement with the conclusions of Mosher et al. [61] and Grima et al. [59] While there is a possibility that fireworks could generate particles of similar composition and morphology to GSR, the likelihood of this occurring appears to be minimal. The possibility that a particle originating from fireworks would result in a false positive for an assessment of GSR is further reduced by the fact that a trained GSR examiner would be able to exclude such a particle on the basis of morphology, the presence of elements uncommon in primer GSR, or the wider particle population.

Overall, the current situation suggests that although fireworks may represent a theoretical source of particles indistinguishable from GSR, and, due to the presence of Pb, Sb, and Ba, cannot be entirely excluded as a source, by themselves, the probable impact of their contribution to the majority of assessments of GSR evidence is so minimal as to be virtually insignificant. That said, under specific circumstances, the consideration of their contribution is warranted. The types of compounds present in fireworks are highly specific to the desired pyrotechnic and visual effect, with some types, (such as the 'Crackering Ball' identified by Mosher et al. [61]) seeming to present an increased probability of generating characteristic particles. In the context of a professional fireworks display, particles generated by these few specific types of fireworks are likely to be a small subset of the overall particle population. However, in jurisdictions where fireworks are available for purchase by the general population, the chances of significant contact with specific types of firework are increased. This is less relevant in a jurisdiction such as Australia where fireworks and firework products

are not readily commercially available to the general public. However, fireworks are in some circumstances used to contribute pyrotechnic or explosive components to improvised explosive devices (IEDs) [64-66], and therefore may be a pertinent consideration in some investigations. If case circumstances indicate the presence of fireworks in a situation where GSR samples are to be taken, then their potential contribution is worthy of consideration. It is also worth noting the further research conducted by Grima et al. in their research into the awareness of operational police and emergency services personnel [59]. Their research indicates that these personnel are unaware of the potential problems that fireworks residue may cause for GSR analysis. In their study, they note that the large number of particles generated as a part of a fireworks display represents a particle population that may persist on hands for a long period. Police or emergency services personnel that are in close proximity to a fireworks display may inadvertently contaminate victims, persons of interest, or crime-scenes that are pertinent to a firearms investigation. This situation could result in samples being excluded, or producing a false negative error, in the event that particulate residues from fireworks contaminates a sample of 'true' GSR. To that end, although the probability of residues from fireworks producing a false positive error is thought to be comparatively low, there still exists a risk to the assessment of GSR evidence through potential of a false negative error.

1.5.4. Airbags and automotive pyrotechnic devices

There exists a number of potential sources of GSR-like residues within some newer consumer vehicles in the form of initiators for airbag systems and pyrotechnic seatbelt pre-tensioners. This equipment utilises a small pyrotechnic charge, initiated in an emergency as a means of deploying the airbag and tensioning the seatbelts to ensure the safety of passengers. In each case, an explosive primer is initiated following the activation of crash sensors indicating that a collision has occurred. In seatbelt pre-tensioners, the primer charge initiates a piston which then mechanically operates the seatbelt retractor [67]. Such systems have been found to be effective in reducing injury risk in vehicle collisions [67]. In airbags, a primer charge is used to trigger either a further chemical reaction resulting in the generation of a large volume of gas, or the release of a compressed gas to fill the airbag [68]. In some instances, a combination of the two systems is utilised in what is known as a 'hybrid' airbag system.

Berk performed a survey of the types of particles generated from the discharge of hybrid gas generation systems and the possibility of observing particles compositionally or morphologically indistinguishable from GSR [68]. To do so, the interior surfaces of airbags was sampled post-discharge using a standard SEM pin stub, before running the samples through GSR analysis. It was discovered that large numbers of Zr rich particles were observed which exhibited spheroidal morphology. This was attributed to the mix of Zr with KClO₄ to form Zirconium-Potassium Perchlorate (ZPP), which is used as an initiation charge as a part of the airbag inflator. There was also an observed presence of AlSi microfibers in samples collected from airbags. These fibres were easily differentiated from fabric fibres (such as nylon) due to the intensity of their BSE signal. Such fibres would be unlikely to be observed in residue from a firearms discharge, and therefore may serve as a marker for exclusion in GSR assessments [68]. Berk identified that a large percentage of the particles observed in the airbag population contained Co, Sr, Zr, Al and CuCo but were uncommonly observed on the clothing of individuals who had been present in a car where an airbag had not deployed [68].

A further particle type assessment to investigate the proportion of consistent and characteristic GSR indicated that airbags from a variety of makes and models were capable of generating residues that were compositionally and morphologically similar to GSR. However, in each case, they represented an incredibly minor proportion of the particle population (less than 0.3% consistent GSR), included elements atypical of GSR, and appeared in a wider population of particles uncommonly observed in GSR. In all instances where 'GSR-like' particles were observed, the majority of the wider particle population contained particles rich in Co or Zr. Though it is noted that some ammunition types contain Co, particularly as a component of the projectile jacketing material, it is comparatively rare as a component of primer or propellant material [69]. In this case, it was observed that up to 76% of the particles present in the wider particle population were rich in Co. Similarly, Zr-rich particles were detected in up to 87% of the particle population. Zr is not allowable in GSR and often results in exclusion. Berk also noted that particle populations from some manufacturers contained a high percentage of Al-rich (29% of total particle population) and Sr-rich (37%) particles. This may be of further relevance to GSR assessments if HMF ammunition has been used, as Sr and Al may be frequently observed in residues originating from these ammunitions [70]. This is particularly relevant in the case of Sr-rich particles, as these residues are now classified as consistent with GSR from Pb-free or non-toxic ammunitions under the ASTM [13].

Lafleche et al. performed a further study in Canada in 2018, considering 53 separate air-bags from 28 different makes and models of vehicle [71]. Their findings broadly agreed with Berk et al. and previous GSR studies on airbags [72], in that the particle populations identified contained K, Cl, Na, Si, Al, and Fe with other minor inclusions such as Zr, Sr, Cu, Co, Ti, Ca, Zn, Sn, Br and Mg [71]. A number of these elements are uncommon in GSR, and therefore could be excluded from consideration. Airbags from only two vehicles were identified to produce 'GSR-like' particles containing PbSbBa, a Chevrolet Silverado and a Honda Civic. Particles from both airbags exhibited spheroidal morphologies, and incorporated Pb, Sb and Ba, and were therefore superficially similar to GSR. However, of these two vehicles, all PbSbBa particles from the Civic incorporated Co, which is very infrequently observed in GSR, and served as a marker for exclusion. Similarly, all PbSbBa recovered from the Silverado included F, which is similarly uncommon in GSR. Both sets of PbSbBa particles recovered from either vehicle contained elevated levels of K and Mg. While some primer GSR particles from firearms have been observed to contain trace amounts of Mg [73], higher levels are associated with nonfirearm sources [54]. Repeated testing of airbags from the same manufacturers failed to identify additional characteristic particles from these sources, even when considering airbags manufactured in the same year, at the same facilities, and by the same manufacturer. Ultimately, the authors conclude that although there is a possibility of airbags generating particles with compositional and morphological similarity to GSR, they were only observed in a small percentage of the airbags tested (5%), and even then, were not observed in all airbags of the same type. Their conclusions are broadly in agreement with Berk in that only passengerside dashboard airbags were observed to generate the particles [72]. It was therefore recommended that in situations where GSR analysis is to be performed and the deployment of an airbag is a valid concern, a comparison sample from the airbag should be collected.

Considering the possibility of airbags of certain types generating particles that are compositionally or morphologically similar to GSR, the literature indicates that a minor possibility for type 1 errors does exist. However, similar to other potential sources of particles indistinguishable from GSR, the number of possible sources is low, and often additional indicators allowing particles to be distinguished from 'true' GSR are present. With specific regard to airbags, Co-rich and Zr-rich particles dominate the wider particle population, which Berk attributes to the use of Co (III) triammine trinitrate used as an oxidiser, in combination with sodium azide as a source of nitrogen gas. The presence of Zr-rich residues is suggested to originate from ZPP used as an initiator for the airbag. These conclusions were broadly shared by Lafleche et al. suggesting that the presence of Co and Zr rich particles in a wider population could indicate that a sample contains airbag contributions [71]. Other indicators, including elevated levels of Mg or K present in particles suspected of being GSR were also noted. If considering the possibility of observing residues consistent with Pb-free, non-toxic or HMF ammunitions, both authors report the presence of large numbers of Sr-rich particles. While the presence of indicators in the population may allow these to be excluded as originating from HMF GSR, the amount generated in an airbag deployment, coupled with the frequency of airbag deployments, suggests that this population may serve as a source for wide secondary or tertiary transfer of these particles from vehicles. This would further suggest that Sr particles originating from airbags would be more frequently encountered than the same particle type from shootings using HMF ammunition. This would then limit the significance of finding Sr particles in a firearms investigation, and perhaps suggest that they are over-valued as a particle consistent with HMF GSR.

Additionally, the study by Berk in 2008 indicated eight different manufacturers and ten different models of vehicle containing airbags that produced particles with compositional similarity to GSR [72]. The more recent study by Lafleche et al. in 2018 identified only two manufacturers and two models [71]. Only a single manufacturer (Chevrolet) was listed in both studies. This provides an indication that perhaps manufacturers are moving away from the inclusion of heavy metal-containing primers in airbags, possibly suggesting that the probability of observing these residues from airbags will diminish further over time.

1.5.5. All sources of particles compositionally or morphologically similar to GSR

In considering each of the possible sources of 'GSR-like' particles, or particles that are indistinguishable from GSR, a number of specific recommendations present themselves. In each case the elements considered characteristic of GSR - Pb, Sb and Ba - are present, and therefore the theoretical possibility of them being combined into a single particle exists. However, in most of the situations considered, the exact compositions of sources and circumstances under which particles are formed do not closely resemble the discharge of a firearm. This has the effect of producing a range of compositions of particles that provides an indication to the trained GSR examiner that the particles have originated from a source other than a firearm. Spheroidal, globular, or condensate-like morphology has been infrequently observed from alternative sources, but is often seen in 'true' GSR. Similarly, inclusions of elements considered atypical to GSR, such as Mg in the case of fireworks, high concentrations of Fe in the case of brake pads or cartridge operated industrial tools, or Co in airbag residues, may further suggest that a particle should not be considered GSR. Finally, the overall particle population in which suspected GSR particles appear may provide important context to the assessment. In each of the cases above, particles containing Pb, Ba or Sb were present in the minority, with a number of particles of differing elemental compositions representing the bulk of the samples considered in each case. Similar to atypical elemental inclusions, the composition of this wider particle population can provide evidence that the source may not be a firearm. Interestingly, this may be both due to what is present, and what is absent in this population. This was observed in the presence of large quantities of K-containing particles in the case of samples from fireworks, or the absence of Pb-containing particles in the case of samples from cartridge operated industrial tools.

Fundamentally, while these sources represent a theoretical possibility of producing particles that are compositionally or morphologically indistinguishable from GSR, that could then result in a false positive error, the probability of this occurring is low. However, in order to reduce the likelihood of one of these sources impacting a GSR assessment, a cautious, case-by- case approach should be taken. A GSR examiner must be aware of the possibility of these sources contributing to their assessment, but also the relative likelihood of that contribution.

1.6. GSR TRANSFER

The value of trace evidence lies in its ability to establish key associations which then contribute to the reconstruction of the circumstances surrounding the commission of a crime [74, 75]. In order for this to be successfully exploited, understanding the involvement and influence of all of the processes and decisions that encapsulate the entirety of the forensic science process is essential [76, 77]. The structure of the forensic science process incorporates considerations made at the crime scene, the collection of samples, the analysis and interpretation, and the ultimate presentation of this evidence in a courtroom. In this context, the sheer diversity of trace evidence types presents many avenues through which useful information to support an investigation may be obtained, provided that the significance of that evidence is interpreted appropriately [78]. However, facilitating this interpretation demands that the many external forces and influences on trace evidence are evaluated and considered. Collectively, these forces and influences have been defined by Chisum and Turvey as 'evidence dynamics', and has been defined to encapsulate 'any influence that adds, changes, relocates, obscures, contaminates, or obliterates physical evidence, regardless of intent' [79]. Practically, these dynamic influences include factors as diverse as the nature of the crime scene; climatic or weather effects; insect or animal activity; the activity of the offender, the victim, medical, police, and fire personnel; as well as sampling; evidence packaging, and storage considerations[79].

One particularly pertinent dynamic influence on all trace evidence, but especially GSR, is in the ability of evidence of this type to undergo multiple transfer events. Critical to the interpretation of this type of evidence, and its contribution to the reconstruction of is how and when these transfer events occur. For the assessment of GSR evidence, this means that the presence of GSR on an individual is not indicative of direct involvement in a firearm discharge. For this reason, when GSR is detected on a sample, rather than reporting this as an indication of definite firearm discharge, a result is often reported as a 'firearm association'. This is then accompanied by a contextual statement explaining the limitations of the finding [37]. It is recommended that this include a statement of the possibility of primary, secondary, and tertiary (or further) transfer, cross-contamination, and the significance of finding or not finding GSR on a surface.

1.6.1. Primary Transfer

Most frequently, primary transfer is defined as the transfer directly from the initial discharge of a firearm [37]. This therefore involves GSR present on the shooter's hands as a result of discharging the firearm [12, 80], but also extends to the presence of GSR on any victims, bystanders, or individuals who attend the scene shortly after a firearm discharge [81, 82]. Primary transfer therefore is significantly impacted by the circumstances and conditions of the initial firearms discharge, and the plume of residues distributed from the firearm. This can be influenced by the construction, calibre, and type of firearm that was used. High-speed photography studies performed initially by Schwoeble and Exline provided data related to the propagation of GSR plumes in the immediate aftermath of discharge [5]. Generally speaking, GSR is observed to be distributed from any gaps in the frame of the firearm, including the barrel, the ejection port, the trigger gap, or the cylinder gap (in revolvers). The authors observed that generally, for short arms (pistols and revolvers) the area of highest plume concentration was focussed on the fingers, hand, and wrist of the firing hand. This effect was more pronounced in revolvers, which release a significant cloud of residues from the cylinder gap in the vicinity of the shooting hand. With long arms, (rifles and shotguns), the highest concentration of the plume was observed in the area of the crook of the supporting arm, extending backwards to the shooting hand, face, and hair. These conclusions were broadly supported by Ditrich [83], who also observed substantial distribution from the breech and ejection port of the firearm when it was opened for reloading. They specifically noted that the distribution of particles and extent of primary deposition of GSR on the hands of a shooter when firing long arms is significantly impacted by when the chamber is opened to eject spent cartridges. This finding is particularly relevant to the Australian environment, where semiautomatic rifles and pistols are comparatively rare, with the bulk of firearms in circulation being manual action rifles and shotguns [9]. However, it should be noted that the propagation and evolution of a GSR plume is not only contingent on the firearm from which it is emanating, but is vulnerable to the influence of atmospheric conditions such as wind, by which it is ultimately distributed.

When considering primary transfer of GSR to bystanders, this may be influenced by the proximity of the bystanders to the firearm discharge. Fojtasek et al. mapped the spatial distribution of GSR in multiple directions around the discharge of a firearm in a controlled environment [81]. They note that GSR particle populations can be detected at distances of up to 10 meters from the firearm itself if fired indoors. If fired outdoors, the results were more difficult to predict, as the particle cloud is affected by the prevailing weather conditions. They
further note that the regions of highest particle concentration are not in the immediate vicinity of the discharge, but 3 - 4 meters forward of the firearm, likely due to the blast of residues expelled from the barrel during discharge. Interestingly, they also observed that the spatial distribution of GSR was concentrated forward and to the right of the shooter, coinciding with the side of the firearm on which the ejection port is located. Distribution in the direction of the projectile has also been investigated, with studies by Gerard et al., indicating that particles of GSR can be found almost 20m in the direction of projectile travel [84], suggesting that the particle cloud from the discharge is drawn with the projectile. This has been further supported by research indicating that GSR particles can routinely be detected adhering to the base of a discharged projectile [85]. In some situations, this has resulted in GSR being located around the projectile impact point, up to 800m away from where the projectile was fired [86]. A study by Greely and Weber further indicates that GSR distributed along the path of the projectile can be carried beyond a physical barrier, with GSR particles found distributed into the environment on the other side of windows that had been fired through [87]. Studies performed of primary transfer to bystanders in the immediate vicinity of a firearms discharge has indicated that the number of GSR particles present on a bystanders hands may be comparable to those present on a shooter, and therefore particle counts alone should not be relied on to distinguish a shooter from a bystander. [84, 88]. Taken together, it is clear that GSR is distributed across a significant area surrounding the firearm following discharge, making it likely that any bystanders in this area could therefore be exposed to primary transfer.

The area of the discharge plume however is not the only consideration for the possible primary transfer of GSR. A further study performed by Fojtášek and Kmjećc considered the 'settling rate' of GSR, in order to ascertain how long the particulate cloud persisted in the air after discharge [82]. Their research indicated that when firing a semi-automatic pistol, the maximum particle fallout, containing more than 50 individual characteristic particles, was observed between 1.5 - 2.5 minutes after the discharge. However, more than 5 characteristic particles were still detected in a population collected 6 - 6.5 minutes after firearm discharge. It was found that discharge from a revolver increases this interval, with the maximum particle fallout, containing more than 80 characteristic particles, observed 3.5 - 4 minutes after discharge. When considering particle size, the larger particles (6 - 10 μ m) represented the dominant proportion of the sample in the first 3 minutes, and were found to settle out first. Smaller particles (0 - 5 μ m) were predominant beyond this period. This

experimental finding was observed to agree with the theoretical modelling for the particle settling rate [89]. However, further research conducted by Schulteis et al. has suggested that in a still environment, this settling rate could be much longer than expected, with results from their survey indicating that GSR may still be present in the air three hours after initial discharge [90].

Based on the literature, it is clear that GSR is widely distributed in the aftermath of a firearms discharge, and is possibly suspended in the air for a significant period of time. This has the consequence of the primary transfer of GSR not being restricted to the shooter, but potentially being deposited also on the victim, any bystanders in the vicinity, and possibly any individuals arriving on the scene immediately following discharge. As there is no way to differentiate the residues distributed to the shooter by primary transfer, and those settling on other individuals, this can add a level of complexity to the interpretation of GSR evidence.

1.6.2. Secondary and Further Transfer

A further important consideration for the interpretation of GSR evidence is the phenomenon of secondary transfer. Secondary transfer involves GSR being present on a surface or individual that was not present during the initial discharge, as a result of contact with a firearm, surface or individual that has GSR present due to primary transfer. This then results in the spread of GSR particles beyond the initial firearms discharge incident. A major difficulty in the evaluation of secondary transfer is the nature of the transfer event is difficult to assess. The transfer event may not be wholly separate from the incident under investigation, in that the individual has had contact with the firearm, shooter, scene, victim or bystanders, and therefore the GSR present due to secondary transfer is not a controlled process, GSR may be transferred by any contact, even to individuals who have had no involvement with the incident, beyond a random contact with an individual or surface that has undergone primary transfer.

To investigate the dynamics of secondary transfer, French, Morgan and Davy analysed a set of scenarios designed to replicate real world conditions under which such transfer may take place [91]. In their study, they compared the GSR present on the hands of: an individual immediately after discharging a firearm; a second individual, separated from the discharge, who shook the hand of the shooter; and a different second individual, separated from the discharge, who handled the recently discharged firearm. In the first scenario, they detected between 206 and 443 total characteristic GSR particles, with the majority of particle sizes

between 1 - 2.99 µm. In the handshake scenario, they observed between 30 and 129 particles, with the majority of particles sized the same as the first scenario. In the firearm handling scenario, they observed between 14 and 50 particles, again, with the majority of particles between 1 - 2.99 µm. While in this case the shooter exhibited the largest particle counts, the authors also noted the significant variability in the GSR deposition between different firing occurrences, and therefore caution on extrapolating on this relationship [91]. Fundamentally however, in each instance investigated a GSR population was observed on an individual as a result of secondary transfer, with particle counts in a range that would be reported as significant. Additionally, the authors note that the size of the particle population on the individuals who had undergone secondary transfer was large enough to support potential tertiary, or quaternary transfer events [91]. A further study by French and Morgan investigated this possibility by extending the number of handshakes in the chain to two [92]. In this study, the shooter shook hands with a second individual who was not present at the original firearm discharge, who then shook hands with a third individual. Following this, between 21 - 44 GSR particles were detected on the secondary transfer subject, and 12 - 22 GSR particles were detected on the tertiary transfer subject. This represented a secondary transfer efficiency of between 6.4%-11%, and a tertiary transfer efficiency of between 40.9% -57.1%. The increase in transfer efficiency is a reflection of the fact that a smaller number of particles were transferred; therefore successive transfers resulted in a larger percentage of the population being transferred. While it was noted that the overall particle population diminishes with an increasing number of transfer events, in each case, the population present was at a level that would be reported as containing GSR [10, 93, 94]. Indeed, further studies of the potential transfers of particulate evidence have indicated that tertiary transfer, quaternary transfer, and beyond are possible, if there is a suitable initial particle population [95]. This further study also highlighted the possibility of inert surfaces serving as a 'reservoir', or secondary source for transfer. The rationale being that communally handled inert items (door handles, keyboards, tables for example) may become contaminated with GSR, and then act as intermediaries, allowing deposited material to spread to the next person who touches that item. Similar findings have been reported for other types of trace evidence, such as fibres [96] and glass [97]. Therefore, the possibility of a transfer chain, with multiple individuals experiencing secondary or further transfer events is a key concern in the evaluation of all trace evidence. This is particularly pertinent for GSR, where considerations of the context and specific case circumstances must form part of the interpretation of evidence.

1.6.3. Cross-contamination

While subsequent transfers from the incident under investigation are an important consideration, a further concern for the potential secondary or further transfer or GSR is related to the difficulty in establishing a definitive source of GSR. Although in some rare circumstances there may be markers present in GSR which establishes a link between the residues and a particular ammunition or firearm, this is the exception rather than the rule. To that end, while the presence of GSR is an indication that the particles have been generated as a consequence of firearm discharge, they cannot be conclusively linked to a specific firearm discharge. Therefore, the presence of GSR from firearms discharges other than the incident under investigation can further complicate the interpretation of GSR evidence. These events may be as a result of family members or relatives with firearms associations due to hobbies or occupations [98], or perhaps more significantly, transfer due to contact with police and law enforcement [73, 99-101]. In the context of GSR analysis, the importance of considering secondary transfer in these circumstances lies in the potential for an individual uninvolved in the firearms incident under investigation returning a positive result for GSR due to a secondary transfer event. This may be considered to be the probability of observing a type 1 (false positive) error. The consequences of an error of this type may result in the further investigation of individuals that are not involved in the incident in question, and potentially false conviction.

To assess the possibility of transfer of GSR to family members from individuals with an occupational firearms association, Brożek-Mucha collected 50 samples from the hands of those with an occupational firearms association (police, hunters, and foresters)[98]. The families of five hunters were identified and sampled for GSR during both the open hunting season and closed 'off' season. In the first case, 94% of police and 71% of the hunters were not observed to have characteristic GSR on their hands as a matter of course. With regard to hunters and their families, it was observed, perhaps unsurprisingly, that there was a correlation between the detection of GSR and the time of specimen collection. GSR was detected on samples from the family at higher levels in the open, or hunting season, than it was in the off season. Similarly, the highest GSR particle counts observed were in a situation where the hunter had been hunting the day before, and their child had been assisting with handling and reloading firearms. It was concluded that there is a strong correlation between the GSR being detected and a proximate firearms contact. With this in mind, while the possibility of secondary transfer from a family member with an occupational firearm association is a factor worthy of consideration in the assessment of GSR, the prevalence of

GSR in these environments is likely to be low, unless a specific, recent firearms contact has occurred [98].

The possibility of transfer from police or law enforcement has been well established, due to the fact that police and law enforcement carry firearms as a part of their job, they present a potential source of GSR that may then be transferred to a person of interest or individual in custody. Transfer in these environments may occur directly due to contact with police or law enforcement in situations of suspect apprehension or arrest, or police facilities and vehicles may serve as an inert reservoir, on which particles are retained until they undergo further transfer. In an investigation of the latter proposition, a study by Berk et al. investigated the possibility of GSR being present on a variety of surfaces in Chicago Police facilities and vehicles [102]. This study sampled from vehicle seats, tables, benches and restraining bars within police facilities. Ultimately, approximately 89% of the samples collected (178 samples negative from 201 collected) did not have characteristic GSR present. The remaining samples identified at least 1 particle characteristic of GSR present, with the majority of positive samples being collected from table-type surfaces within detention facilities. Restraining bars, and the seats of tactical vehicles also returned positive samples. Re-sampling of the positive areas indicated that the presence of GSR was not consistent, suggesting that the particle population is transient and readily transferred onwards, in agreement with French et al. [95]. Berk et al. concluded that although there was a small amount of GSR present, the potential for secondary contamination is relatively low [102]. The authors further recommended that persons of interest not be allowed to come into contact with table surfaces prior to GSR sampling, and that equipment (such as handcuffs or restraints) should be regularly cleaned and maintained to reduce or eliminate the possibility of cross-contamination. These results were supported by a similar study of Pittsburgh Police stations conducted by Ali et al. [103]. This study considered both the organic components (oGSR) as well as inorganic components (iGSR) and concluded that although the potential for secondary transfer for both exists, the potential is miniscule. In this study, across 32 samples collected from interview desks and chairs, holding cell restraint bars and benches, and the hands of individuals detained in police vehicles, only a single sample was found to contain a single characteristic GSR particle. Like Berk et al., Ali et al. conclude that although there is a potential risk of secondary transfer due to contact with police facilities and vehicles, the relative scarcity of GSR and the levels at which it was detected suggest that the likelihood of significant secondary particle transfer to the hands of a person of interest is low [102, 103].

Though the possibility of significant transfer from police facilities appears remote, the fact that GSR has been detected at low levels in both studies indicates that a source for GSR in these environments exists. Therefore, a further consideration is the amount of GSR present on the hands of individual police officers as they carry out their operational duties. To address this, a 1995 study conducted by Gialamas, Rhodes and Sugarman sampled the hands of 43 firearms carrying Californian police officers that had not recently fired their firearm [99]. The hands of participating officers were sampled using a standard GSR analysis stub at the end of shift. Subjects were also asked to answer a questionnaire regarding the type of firearm they carried, when it was last fired, when it was last removed from its holster, and when they last washed their hands. Of the collected samples, only three officers (7%) had characteristic GSR present on their hands. Of the remainder, 25 (58%) had no GSR (neither characteristic nor consistent particles) present on their hands, and 15 (35%) had only consistent particles present. Of the three officers with characteristic GSR on their hands, each individual had a single particle. Based on these results, the authors concluded that the potential for secondary transfer during arrest exists, but the low particle counts indicate the probability is low [99]. This finding is in agreement with the study by Berk et al. in police facilities [102]. Contrary to this finding however, a study by Pettersson collected a series of samples from crime-scene investigators (CSI) and police vehicles in Sweden [104]. All individuals had no contact with a firearm for at least 12 hours prior to sampling. Of the samples collected, approximately 25% were positive for characteristic GSR particles, with one sample having as many as 16 characteristic particles present. This same study found at least 25% of the samples from vehicles contained 6 or more particles of GSR. In a similar vein, Gerard et al. performed a series of surveys of the hands and equipment of Canadian uniformed and plain-clothes police officers [100]. Samples were collected from the hands of 66 police officers, 28 civilians who worked in police facilities, and 18 police vehicles. Of the collected samples, 60% of the police officers had at least one characteristic GSR particle on their hands. The police and civilians were surveyed about their exposure and handling of firearms. Of the police sample, 24% of the officers indicated that they had handled a firearm on the day of sampling. Of this cohort, 44% had GSR on their hands. Comparatively, 30% of the total police sample reported that they had not handled a firearm on the day of sampling, and only 10% of that subset had GSR present. Interestingly, none of the samples collected from the hands of civilians that shared police facilities were observed to contain GSR. This suggests that although contact with a firearm as a part of operational duties may result in a small amount of GSR being present on the hands of police, this particle population does not appear to accumulate in the environment in such a way that

it represents a source for further transfer. The authors note however that their results indicate a higher incidence of GSR on the hands of police than the Gialamas et al. study [99]. This may be attributable to jurisdictional differences resulting in a higher observed incidence of GSR in this study, or differences in sample collection and analysis [100]. Logically, it would be reasonable to assume that differences between jurisdictions in the issuing, handling and management of firearms may result in a difference of GSR background in different locations. However, neither Gialamas et al. nor Gerard et al. provide further comment on how firearms are handled and managed in their regions that allow this to be investigated further [99, 100]. These findings do suggest that regional differences will result in differences in GSR background that must be assessed.

In support of this, a study performed by Cook investigated the extent of GSR contamination deposited on police officer's hands immediately after receiving their firearm at the commencement of their shift [101]. In South Australia (SA) where this study was conducted, officers receive their firearm from an equipment officer, and must check, load, and secure their firearm prior to starting shift. This process requires that the officers manipulate and handle various components of the firearm, including the chamber, breech face and magazine well, bringing them into contact with surfaces likely to have a large GSR particle population present which could transfer to the officers' hands. From 33 samples, only 5 (15%) of the sample had no characteristic GSR present, 16 (48%) recorded between 1-10 particles, 9 (27%) between 10 and 100, and 6 (18%) had more than 100 characteristic particles present, up to a maximum of 610 particles. In total, 85% of the officers sampled had at least one characteristic particle present on their hands following receipt of their firearm. Obviously, it is suggested that officers wash their hands following receipt of their firearm to remove any GSR that may be present. Cook confirmed that vigorous handwashing was sufficient to remove any GSR present, as well as indicating that self-drying hand sanitising gel was also effective at removing GSR [101]. A survey of the textured grip of the firearm when stored in the officers' holster indicated low, but not absent, levels of characteristic GSR particles, suggesting that this may be a minor source for possible transfer. Cook acknowledges that it was expected that a large quantity of GSR would be deposited on the hands of officers when receiving their firearm, but the range of particle counts, from zero to 610, was unanticipated [101]. While it was reaffirmed that handwashing as an anti-contamination measure will address GSR present on the hands, this is only the case if all officers follow this policy religiously. If this is not the case, then there is the potential for a large population of GSR to be present on officers' hands as they perform their duties. A further survey to address this possibility was not conducted at the time. In the context of the other police GSR background studies, Cook's study further suggests that jurisdictional and regional differences in firearms handling and management may significantly impact the GSR background. The process that police officers must follow to receive their firearm in SA may be different from other jurisdictions, where firearms may be personally issued, kept with the officer off duty, or checked and loaded by someone else. This also suggests that specific surveys in individual regions to establish a GSR background may be valuable.

A further study into the GSR background on police performing different duties was conducted by Charles and Guesens [73], who investigated the risk of GSR transfer from special units of the police to arrested subjects. In this study, the researchers acknowledge that the sample set involved specialist tactical response units of the police that were expected to have an elevated GSR background due to the nature of their intense training and active use of many firearms. The units described appear to be analogous to the US Special Weapons and Tactics (SWAT), or the South Australian Special Tasks and Rescue (STAR), which are deployed in critical incidents requiring a higher level armed response. Charles and Geusens performed a variety of mock arrest scenarios, under what was designated 'high contamination' and 'low contamination' situations. The low contamination scenario involved police officers loading their firearm, before mock arresting, restraining, and frisking a person of interest (POI) for a period of five minutes. The high contamination scenario involved police officers wearing a tactical vest, bullet proof vest, and gloves used in firearms training, and then loading their firearm before simulating arrest and restraint of a POI as in the low contamination scenario. In each instance, POIs were dressed in single-use Tyvek coats. At the conclusion of the scenario, the hands of both the POI and the police officers were sampled for GSR, and the Tyvek coats were seized for future sampling. In the low contamination scenario, results indicated that in 25% of the cases modelled, GSR cross-contamination was detected on the officer's hands, and both the target's hands and their clothing. In 33% of the modelled cases, there was GSR present on the officer's hands, and the POI's clothing, but not their hands. A further 25% of cases showed no GSR present on the target at all. In the high contamination scenario, 58% of samples showed contamination of the police officers' gloves, and both the target's hands and clothing, while 42% of samples showed contamination of the officer's hands, and the target's clothing, with minimal or no contamination of the target's hands. Taken together, this indicates that in this study the clothing of the POIs was more contaminated than their hands on average. However, in the low contamination scenario, characteristic GSR was observed on the POI's hands in 42% of cases, compared to 92% in the high contamination scenario. Of particular interest in this

case was the fact that two different types of ammunitions are routinely used by these special units of the police. The first ammunition was operational ammunition that contained conventional three-component PbBaSb based primer. However, the other two ammunitions were used for training purposes, and contained Pb-free primer, with one ammunition having a Ti and Zn based primer, and the other having a Si, S, and K based primer. The training ammunition could not, under the ASTM guidelines, produce particles considered characteristic of GSR, however TiZn particles are considered to be consistent with a firearms origin [13]. The particle counts from the subjects in this study indicate that there were significant numbers of TiZn particles detected. In fact, of the 72 samples collected, only 5 samples (7%) did not have TiZn particles present. If this consideration is taken into account, in the low contamination scenario, 58% of subjects had TiZn particles present on their hands, and 75% of subjects had either TiZn particles or characteristic GSR, or both on their hands. In the high contamination scenario, all subjects had TiZn particles on their hands, resulting in all subjects having either characteristic GSR or TiZn particles or both on their hands. The authors note that as the prevalence of Pb-free primed ammunition is comparatively low in criminal shootings, the presence of particles originating from such a primer on a POI may serve as an indication that potential transfer from a member of the police has occurred. However, the results of this study further indicated that GSR is likely to persist on gloves and equipment used in firearms training. The authors conclude by suggesting that the risks of GSR cross-contamination from special units of the police must be clearly communicated and considered as a part of the case pre-assessment. Further, they indicate a further step for addressing this would be issuing the police with tagged ammunition, similar to that issued to German police [38]. This will then permit unequivocal indications of contamination or transfer from police resulting in an undisputed conclusion about the origin of the particles [73]. It is worth noting however, that the types of arrest that special units are involved in tend to be atypical events, as these units tend to be deployed in critical situations. Although an assessment of the GSR background and therefore the likelihood of secondary transfer or cross-contamination from these units is valuable, it does not represent a general rule by which the whole of the population of police officers and their activities can be assessed.

In Australia, studies of the GSR background on police have only been assessed in a limited fashion in some jurisdictions. In New South Wales (NSW), Hales reported on a survey of a small number of general duties police officers, State Protection Group Tactical Operations Unit (SPG TOU), Crime Scene Investigation (CSI) officers and Forensic Firearms Examiners (FFE) [105]. The hands of two armed, but non-firing general duties officers were sampled, with no particles considered characteristic, consistent or commonly associated with GSR detected. This survey could be considered comparable to the surveys conducted in the USA by Gialamas et al. [99], or Canada by Gerard et al. [100]. A further examination of 19 samples collected from the hands of SPG TOU officers was conducted, with five officers sampled while in the officer prior to conducting a security operation, six following a security operation, and eight following a firing range exercise. Although the eight individuals sampled after attending the firing range were observed to have higher particle counts present on their hands, with the exception of one officer, all of the samples SPG TOU officers had at least one characteristic particle present, regardless of the activities they were conducting before they were sampled. Interestingly, one of the highest characteristic particle counts was 230 particles found on the hands of an officer sampled prior to conducting a security operation. A further seventeen samples were collected from the hands of CSI officers. Of this population, 50% had at least one characteristic particle present on their hands. All of the CSI officers carry a firearm as a requirement of their operational duties. This survey could be considered comparable to that conducted by Pettersson [104]. The hands of three forensic firearm examiners were also sampled, and 100% of samples were observed to contain characteristic GSR.

Hales concludes that much of this survey is in line with expectations [105]. SPG TOU officers, like other special units of the police, undergo rigorous and frequent training with a number of different firearms and therefore would be expected to have a high background. This would suggest that in situations where these units are involved in the apprehension or arrest of POIs in a firearms investigation, a significant potential for cross-contamination by secondary transfer exists. This is in line with the conclusions drawn by Charles and Geusens [73]. Similarly, forensic firearms examiners also have routine and repeated contact with a variety of firearms, so their GSR background could be expected to be similarly high. Hales' survey indicates a moderate presence of GSR on the hands of CSI officers [105]. This is a potential risk if they are collecting samples to be assessed for GSR from either individuals or surfaces. However, Hales further reports that contamination control procedures are in place, including the use of gloves and disposable coveralls, and the collection of control samples from themselves before sampling an individual or item [105]. A particularly interesting consideration is in the general duties officers assessed in this survey. Although no GSR was detected on general duties officers, samples were only collected from two individuals. To that end, further surveys should be conducted to collect from a larger population in order to more fairly assess the GSR background on general duties officers.

The possibility of transfer in a variety of contexts is a major consideration for all types of trace evidence. In the context of GSR evidence interpretation, it is particularly pertinent to consider how GSR may be initially deposited on individuals in the proximity of a firearms discharge, as well as the numerous transfer events that may occur in the events that follow. The circumstances of secondary and further transfer are varied, encompassing everything from direct contact with a shooter and handling of objects that have been present at a shooting scene to experiencing cross-contamination due to contact with the police. To be confident in the integrity of a GSR test result, a number of factors related to deposition and transfer must be systematically considered in the assessment of the case.

1.7. PERSISTENCE OF GSR

1.7.1. General Persistence

Once GSR is transferred to a person or surface, the next concern for the forensic scientist is how long that GSR will persist on that surface before it is moved, lost, or otherwise unable to be detected. A GSR population is not infinite, and therefore represents a finite deposit which is lost over time. This consideration is of particular interest in cases of apprehension of suspected shooters, as it allows a context and time frame to be applied to the detection (or lack thereof) of GSR. The inorganic and metallic nature of iGSR particles means that they are not prone to significant environmental alteration or degradation, and as such, have the potential to persist on surfaces and in other environments for an extended period [5].Previous studies [106] have investigated the issue of the persistence of gunshot residue on the hands of suspects in order to ascertain the extent to which GSR particles may be lost following their deposition [107]. Studies have indicated that GSR may be undetectable after as little as 2 hours [106], or may persist for up to 24 hours [1] in live subjects, with some studies suggesting that persistence may be as long as 126 hours, or just over 5 days [108].

Persistence studies of GSR are at a significant disadvantage as a consequence of the limited reproducibility and lack of uniformity of deposition of GSR between shooters following a firearms discharge. This is compounded by poor reproducibility of particle numbers from shot to shot in test firings. Deposition of GSR on an individual is influenced by a number of factors, and therefore has a significant random element that is difficult to control experimentally, perhaps explaining the wide range of results obtained. Although it can be assumed that GSR has been deposited on the hands of the shooter in the immediate aftermath of a firearms discharge, it is not possible to quantify exactly how much is present at time zero, in order to then accurately determine how much is lost over time. As a consequence of this limitation, persistence studies are more valuable when viewed as guidance, rather than as a definitive rule.

1.7.2. Persistence on Hands

Due to the proximity of the hands to a discharging firearm, they are often considered a preferential target for GSR sampling [27, 43, 109]. Hence, an understanding of the persistence of GSR on hands is an important consideration if appropriate significance is to be applied to a GSR finding. The dynamics of GSR particle loss within the post-firing time frame also been

investigated, with the activity of the suspect being the major factor contributing to particle loss [5]. Jalanti et al. performed research into the persistence of GSR on the hands of a shooter following discharge of a single shot [106]. In all cases, they observed the greatest amount of GSR present on the firing hand, immediately after firearms discharge. However, beyond this point, at measured intervals of 2 to 6 hours, a lack of consistency was observed, with the number of particles present varying unpredictably due to the random nature of particles loss and transfer. While in most cases only a small amount of GSR was present on the hands after 6 hours, in one instance, more than 111 particles were still present. This finding further speaks to the inconsistent nature of GSR particle deposition and retention. The authors concluded that there was no observed variation in persistence based on the chemical nature or composition of the inorganic residues. A similar further study, performed by Schutz, Bonfanti and Desboeufs, also considered the discharge of a single round, and further observed exponential loss of GSR with time, independent of particle size, shape and chemical nature of the particles [110]. In this study, the authors noted a comparable persistence interval, with fewer than 5 total particles being detectable on the hands of the shooter after 4 hours. A similar persistence interval was noted by Brozek-Mucha, who also assisted particle persistence after a single round discharge [111]. This study assessed persistence in 30 minute increments up to a maximum of four hours. It was observed that the largest particle population was present in samples collected immediately after discharge, and that the bulk of this population was lost in the first 30 minutes. To inform their proposal on the statistical evaluation of GSR, Cardinetti et al., similarly measured persistence while increasing the number of shots discharged to 10 [55]. In their study, approximately 10% of the individuals sampled still had GSR observable on their hands 10 hours after firing. They did, however, observe that in the majority of cases the particle population was lost after 5 hours. Towards the upper end of the persistence scale, Rosenberg and Dockery used Laser-Induced Breakdown Spectroscopy (LIBS) to assess the persistence of GSR, and concluded that detectable amounts of GSR were present up to 126 hours post discharge [108]. In this study, the authors considered the Ba emission signal using LIBS, rather than assessing characteristic GSR particles using SEM-EDS as in the previous studies. This decreases the specificity for GSR itself, as Ba-containing particles are not specific to GSR, and have a number of environmental sources. The bulk of the literature indicates that GSR persists on hands for between 2-10 hours after discharge, assuming that no specific activity is undertaken to remove it.

Regarding removal, although movement of the hands as in ordinary, everyday activities has been shown to reduce particle persistence, specific activities targeted at removing GSR,

including rubbing, washing, or wiping hands, accelerates the rate of particle loss [112]. Work by Kilty indicated that washing hands, followed by wiping them dry is sufficient to remove almost all GSR present [112]. Rinsing under running water, and wiping with a cloth towel were found, separately, to reduce, but not entirely remove any GSR present. A similar result was observed for wiping hands on clothing, rubbing hands together, and putting hands in and out of the pockets. When considering particle persistence with time, detectable concentrations of Ba and Sb were observed nearly six hours after firearm discharge, with the subject allowed to conduct normal daily activities in this interval. It should however be noted that this study utilised NAA as its primary analytical technique, and was therefore not highly specific for GSR. A further study performed by Andrasko & Maehly broadly agrees with these conclusions, indicating that rinsing with low-pressure, running water for a few seconds, followed by wiping of the hand was sufficient to reduce, but not entirely remove GSR from the hands of a shooter [44]. However, thoroughly washing the hands was sufficient to remove GSR completely. This study further considered particle loss over time in the event that the hands were not immediately washed. In this case, the GSR particle population was found to decrease rapidly with time through the performance of normal activity and use of the hands. GSR was detected up to 3 hours after firing, but was absent by 5 hours post-firing. It was also observed that larger particles (greater than 10 μ m) were among the first to be lost, disappearing within the first 2 hours, leaving only particles less than 3 μ m present beyond this period. Unlike the work by Kilty, this work used SEM-EDS as the primary analytical technique, and is therefore more specific for GSR [112]. Again, significant variability in the methods and activities covered in these studies has made it difficult to create standard interpretations for the persistence of GSR particles on hands. More recently, Cook further indicated that in addition to washing of the hands, self-drying hand sanitising gel is effective at reducing the number of GSR particles collected from the hands of an individual with GSR present, without rinsing or otherwise wiping the hands [101]. The suspected mechanism is that the gel dries into a clear film, which seals in the GSR particles and prevents them from being collected. This is particularly pertinent, as these are offered as a hand-sanitisation option in police stations and other public buildings.

For most practical purposes, a persistence interval of 2-6 hours is considered sufficient for all GSR to be lost from the hands for forensic testing purposes. This has highlighted a need to promptly apprehend suspects, and take steps in order to preserve GSR evidence. The requirement to preserve this evidence has resulted in the practice of applying gloves or bags to the hands of suspects involved in shooting cases to prevent particle loss prior to sampling.

1.7.3. Persistence on other locations of an individual

As activity is a major factor in the loss of GSR from hands, and the hands are among the most involved and active parts of a person's body, alternative regions of GSR deposition and accumulation of particular interest. Zeichner and Levin established a protocol for the collection of GSR from hair using a tape lift [113]. The method showed no significant difference between the two hair types tested (Curly and straight), and was found to be comparable to methods involving swabbing and combing the hair [113]. Further, it was found that the persistence of GSR in the hair was greater than that on individuals' hands, with GSR observed 24 hours or more after firearm discharge, assuming that the hair had not been washed. This was then directly compared to the casework experience by the same researchers [114], who reported that the average post-discharge time frame while still recording positive results for GSR was 3.3 hours from hair, while only 2.7 hours from hands. They report further that in 18% of cases in a four year period that samples collected from hair returned a positive GSR result, when hand samples from the same individuals were negative. A similar study by Brozek-Mucha compared persistence on hands, clothing and hair, and similarly concluded that GSR persists for the longest time on hair, then clothing, and finally, hands [111]. The author noted that although the numbers of particles initially recorded on the hair was lower than was recorded on the hands, the persistence on the hair was higher. This supports the view that GSR is shed more quickly from hands than it is from other locations on the body.

Further investigating particle retention in different areas of the body, Scwartz and Zona considered the retention of GSR in nasal mucus [115]. While they reported that particle persistence may be as high as 48 hours post-discharge, and a significant number of particles were detected (>500 particles) they further note that this may be highly variable between individuals. Particle retention in the nose may be impacted by the health of the individual, climate, and particular geographic location [115]. The sample collection process was also marginally more complex than simple stubbing, with subjects blowing their nose into a collection substrate, followed by ashing of the substrate and recovery of the GSR to a polycarbonate filter, which was then directly analysed. The additional steps to this process increase the likelihood that particles will be lost, making a true assessment of the number of particles recovered difficult to ascertain. Additionally, the increase in handling increases analysis time, as well as potentially providing more opportunities for cross-contamination or pollution of the sample to occur.

1.7.4. Persistence on Clothing

Like the hands and hair, clothing has often been targeted as a location for GSR sampling. Clothing has the advantage of being washed less frequently than hands or hair, and if stored, can potentially retain GSR for an indefinite period, or until it is physically removed. Brozek-Mucha considered the persistence of GSR on clothing in comparison to persistence on hands and hair, and concluded that the approximate half-life of characteristic particles on clothing was approximately one hour, compared to approximately 30 minutes for hands, and 140 minutes for hair [111]. A further study by the same author assessed the deposition of GSR on cotton and leather targets, indicating that fewer GSR particles were recovered from the leather than from cotton [116]. It was suggested that this may be due to the complex and fibrous structure of the cotton better retaining GSR, while the smoother surface structure of the leather did not perform as well. Charles, Lannoy and Geusens contend that the persistence of GSR on clothing is contingent on the type of fabric from which that clothing is constructed[117]. Specifically, they note that the greater the sheddability of the fabric the lower the observed collection efficiency of GSR. In their study, they were able to recover between 3 and 5 times more GSR particles from the surface of a leather garment than from garments made of cotton and wool respectively. They suggest however, that this may be less a factor of the persistence of GSR on the garment itself, and more related to the collection efficiency from the garment, with high shed fabrics resulting in saturation of the sample stub more quickly. This limitation can be overcome by following the sampling protocol reported by Andrasko and Petersson, which uses a suction device to concentrate residues onto an adhesive stub [118]. The authors however note that because of the long persistence of GSR on clothing, it may be difficult to accurately determine when the GSR was deposited, and therefore is not suggestive of a recent firearm association in the same way that samples from hands are. This is further supported by the fact that comparable to hands, specific activities undertaken to remove GSR can be expected to significantly reduce the amount of GSR present on clothing. However some studies have indicated that GSR may persist, albeit at lower levels, even after conventional laundering. It is for these reasons that an assessment of GSR on clothing is often cautiously applied [119].

Persistence of GSR on individuals and surfaces is an important consideration in the evaluation of the significance of GSR evidence, as it speaks to the duration in which useful samples may be collected. This in turn, can inform the significance of the finding. For example, knowing the relatively limited persistence of GSR on hands suggests that collection of samples from hands more than a handful of hours after the suspected shooting may be of limited use. Conversely, the discovery of a large population of GSR on the hands of an individual suggests a relatively recent firearm association, given the limited persistence of GSR on hands. The same size population of GSR collected from an article of woollen clothing may hold less significance, as the collected GSR may be historic, and have persisted for a longer period. Additionally, the persistence of GSR on a surface renders it available to undergo transfer events, which represents another important factor in the assessment of GSR evidence.

1.8. STATISTICAL ANALYSIS OF GSR EVIDENCE

1.8.1. GSR as Forensic Evidence

The use of gunshot residue as evidence is not without its complications. While the systems of analysis and identification of GSR are well established, the more significant considerations in the assessment of GSR as forensic evidence are related to the interpretation of the significance of a finding. Often, the primary objective of a firearms investigation is to provide a link between a POI and a particular firearms discharge or incident. However, a positive result for GSR on an individual is not conclusive proof of their direct involvement with a particular shooting incident, and is only indicative of the fact that they possess a firearm association, that is have fired a firearm, were present during, or recently after the discharge of a firearm [81, 82], or handled a firearm or other object contaminated with GSR [91]. Similarly, a negative result is not conclusive proof that the individual did not fire a firearm – they may have quickly left the scene [82], washed their hands [112], changed their clothing, or shed the particles over the course of time or normal activities [12]. Ultimately, while the presence of GSR is suggestive of a firearms association of some kind, it does not provide a concrete indication of association with the firearms incident that is under investigation, just as the absence of GSR is not a concrete indication of a lack of involvement [1].

With these limitations in mind, in order to accurately represent the evidential value of a GSR result, forensic scientists need to apply a rigorous and logical process in their assessment of GSR evidence. Such a framework must address a multitude of factors which may include the potential for cross-contamination or pollution of the sample, loss of residue due to activity or the passage of time, as well as analytical and instrumental factors. These must be assessed on a case-by-case basis and interpreted in the context of what effect this may have on the final result. For example, the detection of a large amount of gunshot residue on the hands of a suspect who is a frequent recreational shooter is less significant than residue detected on the hands of a suspect who claims to have never used or handled a gun. The former is much more likely to have been exposed to the residues by innocent means, while the positive test result contravenes the latter's statement. Understandably, in order to make a full and informed assessment about the significance of a finding, the examiner must be familiar with the specific circumstances of the case they are examining, as well as the state of the literature informing the discipline. This presents something of a balancing act, with a GSR examiner needing

enough information to accurately present the significance of the evidence, while not being vulnerable to contextual bias [120, 121].

Historically, the interpretation of GSR evidence has been somewhat at the discretion of the examiner, contingent on their experience and understanding of the specific case circumstances. However, a significant weakness of this approach is that this introduces an inherent subjectivity into the process. This departure from objectivity has attracted criticism, toward all comparison methods, not just GSR, suggesting that research and data must underpin an objective approach to the assessment of evidence assessment methods [122]. In order to address this shortcoming, there has been a move in some jurisdictions to incorporate and strengthen an evaluative reporting framework to the assessment of all evidence types [123-125]. Such a framework provides a formal and structured means to evaluate forensic scientific findings in the context of their relative likelihoods given the case circumstances. By incorporating an assessment of the probabilities involved in the evaluation, this framework allows for the level of certainty (or uncertainty) in an assessment to be established. A strong evaluative reporting framework is also logically consistent, balanced, and transparent to the process followed to arrive at the results. In order to accomplish this, likelihood ratios are often employed.

1.8.2. Likelihood Ratios

In a forensic scientific context, a likelihood ratio (LR) represents the ratio of the probability of observing the evidence under consideration in the context of two competing hypotheses or propositions. These propositions are most frequently assigned the values of H_p or the prosecution proposition, and H_d , or the defence proposition. These are structured in the form represented by equation 1.

 $LR = \frac{Probability of evidence (E)given that the prosecution hypothesis is true}{Probability of evidence (E)given that defence hypothesis is true} (1)$

Which is often rendered in probability notation as:

$$LR = \frac{P(E|Hp)}{P(E|Hd)}$$
(2)

It should be noted that the terms designating the prosecution and defence propositions are rendered as H_p and H_d respectively. This is an acknowledgement of some models which refer to them as 'hypotheses'. For the purposes of this discussion, the term proposition is

considered more accurate, however the terms in equation 2 have been retained both for consistency with other models and to distinguish 'proposition' from 'probability'.

As can be seen from equations 1 and 2, the value of the LR will be greater than one when the probability of the results obtained for the analysis of evidence is more supportive of H_{p} and less than one when the probability of the results is more supportive of H_{d} . If the LR resolves to exactly one, then this is said to be 'neutral' in that the evidence offers equal support to both propositions. While this calculation results in a numerical value assigned to the LR, it is common practice in most disciplines to translate this into a verbal scale, typically ranging from 'extremely strong support against' to 'extremely strong support for' a proposition [123, 126]. As a tool, this allows a forensic scientist a means of contextualising the findings of their examination in the context of two competing propositions. However, this view has attracted some debate, with some research indicating that retaining the numerical scale leads to less confusion between the presentation of the expert and the message received by the jury [127]. This suggests that even carefully formulated statements of expert opinion may result in miscommunications and an understanding gap between the expert and the intended audience [128, 129]. Regardless, LRs have been widely explored and applied to a variety of evidence types, including glass [130, 131], fibres [132] handwriting [133, 134], fingerprints [135], DNA [136, 137], and voice analysis [138].

It is important to ensure that is clear that an expert is providing an assessment of the probability of the value of the results of their analysis of the evidence given the competing propositions and not the reverse [124]. It is the role of the court to assess the probability of propositions under consideration given the totality of the evidence that they have been presented. To that end, the expert should not offer an opinion on the probability of the propositions. In order to navigate this, it is often instructive to consider a thorough assessment of the probabilities that may inform these propositions.

1.8.3. Bayesian Probability

A powerful tool in the assessment of probabilities to inform a LR is the use of Bayesian probability, which allows for probability estimates to be updated based on changing conditions or observations. This system of probability assessment allows for the assessor to express a degree of subjective belief in a specific outcome, and incorporate that into a formal probability assessment. For the purposes of forensic science, this relates to how the understanding of the case under consideration is altered due to the results of the analysis of evidence [139]. This can be described by Bayes theorem, seen below.

$$\frac{P(Hp|E,C)}{P(Hd|E,C)} = \frac{P(E|Hp,C)}{P(E|Hd,C)} \times \frac{P(Hp|C)}{P(Hd|C)}$$
(3)

The equation can be read as the *a posteriori odds* are equal to the LR, multiplied by the *a priori* odds. The a priori, or prior, odds speak to the ratio of probabilities that each of the competing propositions is true, given a known context or set of circumstances (C). It should be noted that this expression is independent of the evidence under consideration. To that end, this can be appreciated as the prior probability ratio and represent the beliefs or understanding of this ratio given the case context, before the evidence is considered. Similarly, the a posteriori odds speak to the ratio of probabilities that each of the competing hypotheses is correct, given the observed evidence and context. This allows for the prior probabilities to be updated in light of the observations. Finally, the LR is rendered as the ratio of probabilities of observing evidence (E), given that each of the competing propositions is true and under a known context or set of circumstances (C). The difference between these two expressions may appear minor, but is a significant one. Indeed, it is important to appreciate what is being evaluated by each term. The a posteriori odds term assesses the probabilities of the propositions being true given the observed evidence and context. By this definition, it is clear that this is an evaluation that is performed by the court, as it is they who are responsible for considering the evidence and case context that have been presented, and then making a judgement in favour of one of the propositions. Additionally, only the court has access to the entire body of evidence pertinent to the case, and is therefore in a more informed position that the forensic scientist, who typically has a very narrow view. Conversely, the LR term assesses the probability of observing the evidence given the competing propositions and circumstances. This is the responsibility of the expert, or the forensic scientist to evaluate as they assess the evidence, evaluating its significance given the context and competing probabilities [139]. For this reason, the LR term is often used to reflect the strength of the evidence. However, evaluating the probabilities that inform the LR, a systematic and structural approach is often required. One strategy for approaching this is through the use of a Bayesian Network (BN), which is particularly useful for assessing complex evidence.

1.8.4. Bayesian Networks

At its most basic level, a Bayesian Network (BN) is a graphical model of decision making representing the conditional probabilities of a distributed set of variables. Each variable is presented as a 'node', which may then be linked to other nodes in the network by arrows that indicate the relationships between them. By plotting these nodes and their relationship between, a BN allows for a structured assessment of the probabilistic relationships between these variables. Individual nodes can be informed by specific experimental data, or estimated based on studies from literature. Due to their flexibility, BNs have been incorporated as a part of the assessment of a number of evidence types, including glass, fibres, DNA, and GSR [105, 139-144]. Proposals for the use of BNs as a means for interpreting evidence provide a structured system for the management of uncertainty in a forensic context. An advantage of the BN is that it allows for a structured and systematic sequence of thought, with the assumptions made presented clearly as components of the model [141, 142, 145]. The model is flexible enough to undergo revision as new information becomes available, or existing information changes.

With specific reference to the use of a BN approach to GSR, a review by Romolo and Margot identified some of the difficulties in adhering to a 'specific approach', instead, advocating for a 'case-by-case' approach that considers the many factors that may influence GSR evidence [107]. They further acknowledge that as a means of accomplishing this, utilising a Bayesian approach to evaluating evidence under competing propositions provides an elegant solution. The authors acknowledge that even a robust Bayesian approach is not likely to incorporate all of the issues that may be faced in assessing GSR, but does provide a useful structure for presenting this type of evidence to a court. They further specify that to be a robust framework, it must be supported and informed by research in order to inform a reliable probabilistic assessment of the strength of such evidence.

Following the suggestion of Romolo and Margot [107], Biedermann and Taroni proposed a joint framework for the evaluation of firearm and GSR evidence [146]. This further demonstrates the flexibility of this paradigm, in that multiple related evidence types can be incorporated to inform the evaluation of a set of propositions. Similarly, the BN can be expanded through the addition of nodes to account for increasingly complex scenarios and situations. As a means of assessing the probabilities of observing *n* GSR particles on an individual after a number of hours, Cardinetti et al . formulated a proposal for assessing the possible loss of GSR evidence based on a Poisson probability distribution [147]. In this study,

the authors performed two surveys – one of a non-firing population, and one of a population who had recently discharged a firearm – and assessed the amount of GSR that was present on their hands over time. These collected data were then tested for goodness of fit to a variety of probability distribution models, with view to using one such model as a means of informing the calculation of a LR for assessing GSR evidence. Ultimately, the authors concluded that the experimental data fit well with a Poission distribution, and propose a model based on this by which a LR for GSR evidence can be calculated. The authors acknowledge that while their proposed framework is valid, it could be further refined, as it fails to account for many of the variables impacting the assessment of GSR evidence. Regardless, Cardinetti et al. maintain that the proposed model provides a useful statistical model by which GSR evidence in casework can be evaluated.

This model was further explored by Biedermann, Bozza and Taroni, who incorporated the data presented by Cardinetti et al. into a Bayesian network [68]. The authors acknowledge that the prior research by Cardinetti et al. is valid, however suggested that a framework approach would allow expansion to incorporate further variables for more complex case assessment. Some of the additional variables proposed include those impacting the sample collected from the individual, including background content of GSR on the sampled individual's hands, as well as purely analytical concerns such as the lifting efficiency of the sample media, condition of the sample stub, and so on. From this it follows that there is a clear distinction between the number of particles present on the hands of an individual due to the discharge of a firearm, the total number of particles present, and the number observed on the sample. The framework suggested by Biedermann, Bozza and Taroni, and a key explaining the factors they propose, can be observed in Figure 6.



Figure 6 - Extended Bayesian Network for evaluating GSR particle evidence as presented by Biedermann, Bozza and Taroni [148].

The authors note however that the network in Figure 6 does not acknowledge that there are additional sources of uncertainty that may influence the calculation that are not accounted for. They propose that incorporating a BN approach allows for this framework to be systematically expanded, both to demonstrate the assumptions that underpin the conclusions, and as a way to evaluate the factors contributing to the conclusions drawn. While values for all nodes can be estimated mathematically based on a broad understanding of the case circumstances, or informed by literature, they may also be directly informed by experimental data [68]. Approaches have been made in this area, with Hales formulating a proof of concept network in tandem with that proposed by Biedermann, Bozza and Taroni [105]. Both Hales and Biederman, Bozza and Taroni note the available scope for the collection of further experimental data to better inform particular nodes to improve the quality of the assessment.

Charles and Nys report on their experiences of integrating a Bayesian approach to the reporting of GSR analysis results [139]. They acknowledge that performance of such an assessment presents some difficulties in the estimation of probabilities to inform the LR, and that additional experiments may be required to better model the situation under investigation. It is also noted that the assessment of the probabilities that inform the conclusions are based on estimates informed by literature and experience. To that end, the quality of any output is a reflection of the quality of the inputs that inform it. Practically, this demonstrates that the value in the BN paradigm for the assessment of GSR evidence is in the

systematic and logical approach required to perform the interpretation. Gauriot et al. [149] broadly agree with Charles and Nys [139], in that they acknowledge the difficulty and complexity of applying a systematic case-by-case approach to a Bayesian statistical GSR evaluation. Gauriot et al. applied statistical quantification models of GSR evidence based on a BN similar to that proposed by Biedermann, Bozza and Taroni, however they did so under a case-by-case style paradigm. Through modelling based on genuine case information, they report that these statistical models are highly sensitive to the values of their inputs, specifically noting that the arbitrary nature of background contamination values and activity levels makes it difficult to apply. Due to this ambiguity, Gauriot et al. suggest exercising extreme caution in applying a statistical approach to the evaluation of a quantitative claim based on GSR observations in casework [149].

While persistent progress has been made in utilising Bayesian networks [139, 146, 148, 150] as a means of ascribing a statistical measure in support of a particular hypothesis, the complexity and diversity of factors involved in the persistence and transference of GSR evidence renders a sound statistical model difficult to accurately and consistently apply [149]. That said, the structured nature of a Bayesian Network provides a useful framework upon which the potential contribution of the factors involved at each node to the overall value of the evidence may be evaluated. Particularly pertinent are evaluation of the nodes that inform the probabilities of observing GSR on an individual due to factors unrelated to the firearm discharge of interest. This incorporates the GSR background of a region, as well as possible instances of cross-contamination. The importance behind this is that these factors inform the possibility of a false positive (Type I) error.

1.9. KEY RESEARCH STAGES

The goal of the research addressed in this thesis was to contribute to a logical framework for the assessment of GSR evidence by further informing gaps in the current knowledge. On this basis, each stage of the research investigated a limitation in the assessment of GSR evidence, with view to incorporating it as a part of an evidential assessment framework. The data obtained could then be applied to informing the nodes of a Bayesian network type framework for the assessment of GSR evidence.

1.9.1. GSR distribution in the random Australian population

In order to be confident in an assessment of the significance of a GSR test result, an understanding of the prevalence of particles considered characteristic or consistent with GSR in the wider population must be considered. If such residues are widely observed in the random population, the relative significance of finding GSR particles on a person of interest is reduced. However, the reverse is true if GSR is rarely observed. The likelihood of observing GSR in a random population is likely to be contingent on a number of region dependent factors, including the popularity of firearms hobbies such as hunting or target shooting, the rate of private firearms ownership, and the probability of secondary or tertiary transfer occurring from same. To that end, a broad Australian based study of the prevalence of GSR on a random individual has yet to be conducted, although data from various jurisdictions may exist. The overall objective of this research stage was to assess the background GSR prevalence in an Australian context through a survey of the random population. These data will then directly inform a Bayesian network node related to the background prevalence in the population.

1.9.2. Police GSR Background Survey

While understanding the prevalence of GSR in the random population provides useful information related to the background of GSR in the general public, a further concern is the background level of GSR present on police officers. Police officers must routinely carry a firearm as a part of their operational duties, and therefore potentially possess a GSR background higher than the general public population. Studies in different jurisdictions indicate that this is the case for their police, and therefore police in Australia may also have a significant GSR background. This is potentially pertinent, as if a GSR population exists, it may pose a rich source for secondary contamination of facilities, vehicles or persons of interest.

Further, given the relatively low rates of private gun ownership, the abundance of GSR in the general Australian population is expected to be relatively low. Initial studies also indicate that the abundance of GSR-like residues originating from other sources in the general population is relatively low [50, 98, 151]. With this in mind, the most frequent contact that the general public are likely to have with armed personnel that are potentially contaminated with GSR is through interactions with law enforcement. Situations in which police officers handle their weapons, including receipt of their sidearm at the start of shift [101] and any time their firearm must be drawn or discharged in the execution of their regular duties has the potential to contaminate officers. While some research has been performed in this field, results have been varied. A summary and comparison of Police Officer GSR background studies can be seen in Table 7.

Police Duty Type	Australian Background		International Background	
	% frequency (n positive samples / n total samples)	Ref.	% frequency (n positive samples / n total samples)	Ref.
General Duties	0% (0/2)	[105]	7% (3/43)	[99]
	85% (28/33)	[101]	9.7% (3/31)	[98]
			60% (40/66)	[100]
Special Operations	95% (18/19)	[105]	75% (18/24) (PbSbBa Only)	[73]
CSI	50% (7/14)	[105]	25% (no data)	[104]

 Table 7 - Comparison of Police Officer Characteristic GSR background studies conducted in Australia

 and Internationally

Initial work has been done in evaluating the GSR background in the specific Australian police population, however this has either been performed on a limited sample size [105], or focussed around the receipt of firearms at start of shift [101]. This research has the opportunity to expand upon the initial survey of the general duties police population as conducted by Hales [105], in order to better inform the situation in the wider Australian environment. Cook's survey of the hands of police after receiving their firearm at start of shift provided useful information regarding how much GSR could be present after a firearm contact [101]. However, an equally pertinent question is if GSR is deposited on receipt of firearms at the start of shift, what amount of GSR persists on the hands of the police population throughout their shifts? If contamination control procedures are effective, it would be expected that the level of GSR background on the hands of serving officers would be comparable to that of the general public. This has not been thoroughly investigated in South Australia, and data from such a study will directly contribute to informing the possibility of cross-contamination or transfer during arrest. To that end, the objectives of this research stage include an evaluation of the GSR particle background observed on the hands of serving police. Cook has previously established that the receipt of firearms at start of shift results in a large number of GSR particles transferred to their hands [101]. This research followed up on this finding to determine if anti-contamination measures are effective in reducing the amount of GSR present on police officers hands, or if re-contamination events are occurring. If a significant GSR background exists on the hands of serving police officers, it is possible that this particle population could be transferred to persons of interest through the process of apprehension and arrest. This then has the potential to result in a false positive GSR test result. Also included was an evaluation of the ammunition products used by South Australian police. This was in order to determine the composition of GSR particles in the police population. This may be pertinent if there are any indicators in the GSR generated from these ammunitions that may serve as a specific indicator of possible cross-contamination between police and suspects.

1.9.3. Transfer during arrest

While some studies have been conducted regarding the person-to-person [91]and surface secondary transfer of iGSR [91], modelling the dynamics of GSR transfer between law enforcement and suspects is relatively under-developed, having only been investigated in a limited capacity [73],' particularly in an Australian context [105, 152]. In order to improve the evidential value of GSR evidence, an assessment of levels of GSR cross-contamination in Australian law enforcement contexts, identification of critical scenarios in which cross-contamination between law enforcement personnel is possible, and the dynamics of transfer between law enforcement and suspects would be invaluable. This thesis provides valuable information around the prevalence, persistence, and transference of iGSR in a law enforcement context and its impact on the use of GSR evidence as a whole.

In order to be confident in the integrity and relevance of GSR evidence, particularly if relatively low particle numbers are involved, an understanding of the potential for suspects to become contaminated through interactions with law enforcement prior to GSR samples being collected is necessary. Further modelling of the dynamics of GSR transfer in these critical interactions between law enforcement personnel and persons of interest addressed a knowledge gap and provided useful data in allowing forensic scientists to apply a logical framework for the assessment of GSR evidence. Modelling the extent of GSR transfer under conditions that would typically be encountered during arrest worked in concert with data previously obtained related to the GSR background on operational police to inform an assessment of the likelihood of secondary transfer occurring under arrest circumstances. These data then further informed a Bayesian network node related to the probability of a false positive due to GSR crosscontamination.

1.9.4. GSR Distribution in cases of suicide

In order to have a developed understanding of the significance of a particular GSR test result, it is instructive to have an understanding of what the expected findings would be. While some studies have been performed in this area, the majority of them have been conducted in other jurisdictions [88, 106, 111]. The Australian environment has distinct differences from other jurisdictions, notably an increased prevalence of 0.22 RF rifles and shotguns.

This research stage used historic cases of firearms suicide as a GSR case study of what expected GSR distribution may be. Suicide cases provide a unique opportunity to assess nonexperimental GSR distribution, as typically the shooter fires a single shot, remains at the scene, and has limited activity after the firearm discharge. For this reason, they may provide data related to the maximum expected GSR deposition under a real shooting event. An additional benefit of investigating these data was that they apply directly to further investigations of suicide. A review of these cases also provided additional valuable data, such as an indication of the types of firearms, ammunition products, which are available in the jurisdiction, as well as an understanding of the results observed when analyses of suicide cases are conducted.

1.9.5. Sub-particle morphology and composition of GSR

Initial surveys of firearms and ammunitions used in Australia indicate a prevalence of 0.22LR rimfire ammunitions. These ammunitions are particularly relevant to GSR investigations, as a significant portion of these ammunitions do not contain Sb-compounds in the primer, and therefore are expected to be incapable of producing characteristic PbSbBa GSR particles. However, the casework experience, as well as some previous studies [17] have indicated the presence of characteristic particles from these types of ammunition. While this may be attributable to the weapon memory effect, it was determined that further investigation of an alternative mechanism was warranted. To accomplish this, the sub-particle morphology and composition of GSR particles was explored.

Though initial explorations have been conducted into the sub-particle morphology and composition of GSR using a focussed ion beam (FIB), there are other ways that this technique can be exploited for the analysis of GSR. Sectioning of particles allows cross-sectioned slices to

be obtained which can then be analysed using alternative techniques (such as ToF-SIMS) to provide further information about GSR particle structure and composition.

This research stage sought to provide additional information surrounding the unexpected prevalence of three-component, characteristic GSR originating from two-component primed ammunition. By sectioning and mapping particles originating from these ammunitions, it was expected that further information as to the structure and formation of GSR would be obtained, which may assist in making judgements about the process behind their formation. Additionally, the inclusion of multiple different ammunition products, including those with Pb-Free, HMF, or non-toxic primers will provide insight into the features inherent in these particles.

1.9.6. Statistical framework for the assessment of GSR evidence

Previous work in the evaluation of a Bayesian network approach for the assessment of GSR evidence has suggested further research to produce more robust data as a means of informing the network [105, 139]. These data can involve a consideration of the type of firearm, time since discharge, ammunition used, GSR background and likelihood of cross-contamination. A number of these factors are highly dependent on an understanding of the specific case context. Others are more open to a more general assessment. Of specific interest to the research conducted as a part of this thesis are the factors that inform the possibility of GSR being present on an individual due to the background and possibility of cross-contamination during arrest. These factors speak to the probability that a randomly selected member of the public who has not had a recent firearm association, would test positive for GSR. As such, these findings could be used to better contextualise and explain the significance of GSR evidence. Of particular interest were the GSR background in the random metropolitan population in Australia, the background on the hands of serving police, and the possibility of transfer under arrest circumstances will be used to inform the network. This will then be used as a means to estimate the probability of observing a false positive error as a result of GSR exposures beyond the firearms discharge under investigation. It is hoped then that this will be able to inform future assessments of GSR evidence to increase the confidence in GSR results.

This thesis forms an original contribution to knowledge by providing a developed and detailed assessment of the factors informing a statistical framework for the assessment of GSR. This research is notable in two ways – it represents the largest sample size of a study of this type.

Secondly, all of the data were collected in the same region, and therefore are not as vulnerable to cross-jurisdictional differences. Ultimately, this research will contribute to a providing foundation for a logical framework for the assessment of GSR evidence in the context of Australian specific concerns. It will therefore assist in informing judgements of the significance of the GSR evidence obtained in the investigation of firearms crime.

2. MATERIALS, INSTRUMENTATION, AND METHOD OPTIMISATION

2.1. SYNOPSIS

Prior to commencement of this research, a new SEM-EDS system had been sourced for use as a GSR analysis instrument. An FEI Inspect F50 SEM-EDS system equipped with TEAM-EDAX elemental analysis software and GSR Magnum gunshot residue analysis package was commissioned and validated for GSR analysis. The ideal settings for optimum instrument performance in the analysis of GSR were determined through the use of a Plano standard, consisting of a known number and size distribution of three-component GSR particles deposited on a glassy carbon chip.

To verify the operation of the FEI F50 system, its performance in GSR analysis was compared against an existing SEM-EDS instrument used for forensic GSR analysis at Forensic Science SA in South Australia. Further, an ENFSI proficiency test sample (ENFSI GSR 2014) was used as a means to verify the instrument's operation, and compare it against other instruments in operating forensic laboratories world-wide.

Although the FEI F50 exhibited unsatisfactory performance in detecting particles of 0.5 μ m diameter, and questionable performance for particles 0.75 μ m and 0.8 μ m in diameter, it exhibits satisfactory performance for particles of diameters 1.0 μ m and above. The primary analysis instrument was seen to have comparable performance to the SEM-EDS system validated for GSR work in South Australia, and displayed comparable performance to other instruments in GSR analysis laboratories worldwide.

2.2. SUBJECT SELECTION

A significant part of the research discussed in this thesis was accomplished with the participation of volunteers, both members of the general public, and serving police officers. Ethics approval for this research was obtained for the collection of GSR samples from the hands, clothes, and other locations on their bodies. Approval was granted by the Flinders University Social and Behavioural Sciences Research Ethics Committee (SBSREC), project number 6780.

Prior to volunteers participating in the study, both an oral and written briefing was delivered, providing details regarding the purpose of the study, the specifics of what samples would be taken, and contact information for researchers. All participants were informed in detail of the goals and objectives of the research and were offered a written copy of the research study details for their information.

Participation in all stages of this research was strictly on an 'opt in' basis, and it was made clear to all participants that they would not be adversely affected in any way by choosing to either opt in, or opt out of the study. Steps were taken to ensure that this was abundantly clear with all subject groups, and this was communicated via a verbal address prior to seeking their cooperation, and in a subject information form provided in hard copy to all participants.

As a requirement of the ethics approvals, all participant information was de-identified and all participants remain anonymous. Though questionnaire responses and demographic information were collected at the time of sample collection and linked to the samples, no information identifying the volunteer was included. Consent forms were collected and stored separately from subject questionnaires, and were not linked in any way to the physical samples.

Members of the public who contributed samples to the survey conducted in chapter five were approached at random in public areas in three jurisdictions. Verbal and written informed consent was sought before their sample was provided.

Serving members of South Australian police who contributed samples to chapter six were approached while at work, with consent and permission of the South Australian Police commissioner. Members who were sampled were approached while attending a routine training course. Other samples collected for the purposes of identification and comparison of operational and training ammunitions were collected from the hands of South Australia Police (SAPOL) cadets as a part of a routine firearms qualification as a part of their training. In each case, the cohort was briefed in detail about the purpose of the study and how their samples would be handled before they were asked to volunteer. Volunteers then completed a written consent form before being directly sampled for GSR.

A review of GSR case files which contributed data to chapter 3 was conducted using coronial cases of suicide involving a firearm in South Australia between 1998 and 2014. Prior to accessing the case files, approval was sought and received by the State Coroner and FSSA. Case files were identified by case number only, and all details that could potentially identify the victim were redacted to preserve their anonymity. The only results of GSR analyses and sample request forms were extracted from the case file. A further review of case files between 1999 and 2014, to identify the types of ammunition and firearms used in all crimes in SA was performed following the same process under direct supervision from staff at Forensic Science SA.
2.3. SAMPLE HANDLING

The following describes the general sample collection and handling processes that were used throughout the research performed in this thesis. All processes and procedures that were used were informed by reference to the Scientific Working Group for GSR (SWGGSR) 'Guide to Primer GSR analysis by SEM-EDS' [37], as well as consultation with SAPOL and FSSA current operational procedure for the collection of GSR samples. Where pertinent to the discussion, specific deviations to sample handling or analytical procedure are described in the methods section of the appropriate chapter.

2.3.1. Sample collection

The bulk of sample collection for this research was conducted using pre-packaged GSR stubs consisting of a 12.5mm diameter aluminium SEM pin stub coated in a carbon-tape adhesive. Each stub is provided in a separate plastic sample container, keeping the adhesive tab isolated from the atmosphere until ready to use. Stubs were provided by Tri-Tech Forensics Inc. (North Carolina, USA). All samples were clearly labelled at the time of collection with an identifier that linked them to the current research, as well as the date and initials of the individual collecting the sample.

Where samples were collected directly from a subject's hands, face, or clothing, the stub was directly applied to the surface in question and 'dabbed' across the surface in a particular pattern until such time that the adhesive was exhausted, approximately 50-100 dabs, depending on the level of particulate matter present. Once completed, the stub was re-sealed and stored until analysis. When collecting GSR samples from hands, the stub was first applied to the area of the thumb, forefinger, and the webbing between the two fingers, before expanding out to include more of the hand. This sampling procedure was based on both the SWGGSR recommendations for sampling from human subjects [37], and previously established procedures for collecting GSR. Unless otherwise specified, when collecting a sample from an individual who was known to have recently discharged a firearm, two stubs were used, one for each hand. Each stub was used to sample both the back and front of the nominated hand. When collecting a sample from an individual who was not known to have recently discharged a firearm (e.g., background surveys), unless otherwise specified, a single stub was used for both hands. Collection from the individual was performed on the back of the nominated dominant hand first, then the dominant hand palm, then the non-dominant hand's back,

followed by the non-dominant hand's palm. The rationale behind this sampling process is to maximise the area surveyed, while keeping the total number of stubs analysed to a minimum.

Where samples were collected directly from fired cartridge cases (FCC), the cartridge cases were collected with gloved hands following discharge and placed in separate press-seal plastic exhibit bags. The bags were then transported to a laboratory where a clean wooden probe was used to carefully swab the inside of the fired cartridge case, before being rolled onto a new GSR stub. The wooden probe was then disposed of, and the cartridge case re-sealed in a plastic press-seal bag. Due to the possibility of cross-contamination, collection of samples from FCCs was performed in a separate laboratory space, physically separated from both the SEM room and the carbon coater room.

A similar process was followed when sampling for GSR directly from a firearm. A clean wooden probe was used to gently scrape the inside of the barrel or breech of the firearm, after it had been rendered safe following firing. The probe was then carefully rolled onto the surface of a new GSR stub. The stub was re-sealed, and the wooden probe discarded.

For long term storage, samples from different collection sessions were sealed into labelled, plastic, press-seal bags. Collected samples were stored in a different location to un-used stubs, and were separate from both the SEM-EDS room, and any laboratory preparation areas.

2.3.2. Control Samples

Control samples were employed throughout this research as a means of providing some indication of cross-contamination that may have been introduced into the process. Prior to collection of samples, a control sample was collected from the gloved hands of the individual performing the sample collection. Negative controls were employed during sample coating to ensure that there had been no cross-contamination between particulate matter in the chamber and the stubs that were being coated.

In some circumstances, an environmental control was employed as a means of ascertaining the possible level of GSR particle presence in the environment. This was achieved by exposing a fresh GSR stub in the vicinity of the activities being monitored. These were primarily employed during the recovery of GSR from FCCs to assess the potential for release or crosscontamination. When analysed by SEM-EDS, control samples were run first, before the remaining samples were assessed.

2.3.3. Carbon Coating

To improve the conductivity of the samples and prevent them from exhibiting charging artefacts in the SEM, a thin coat of carbon was applied to the GSR stubs prior to analysis. This process was conducted following the recommendations of the SWGGSR guide, which recommends carbon, as it does not interfere with the identification if elements of interest, and does not have a significant impact on the backscatter electron (BSE) signal that is used to identify GSR [37]. This process was performed using a Cressington 208 Carbon High vacuum carbon coater with rotary-planetary stage. The thickness of the carbon coat was estimated through use of a brass thickness monitor, and a coat of between 200 – 250 Ångström (20 - 25nm) was applied. To ensure that cross-contamination did not occur between samples in the carbon coater, each batch was processed with a fresh, unused GSR stub. The stub was then coated alongside the exhibit stubs, and analysed at the start of each analysis run in the SEM.

2.4. SEM-EDS

Scanning Electron Microscopy with Energy Dispersive X-Ray Spectroscopy (SEM-EDS) was used as the primary analytical technique. This technique was chosen on the basis that SEM-EDS has wide acceptance in the forensic scientific community as the 'gold standard' or 'optimum technique' for the analysis of GSR [12, 37]. To that end, a large number of operating forensic laboratories that perform GSR analysis use SEM-EDS as their primary GSR analysis technique.

Two SEM-EDS systems were used for analysis and data collection throughout the research conducted in this thesis. Gunshot residue particle searching was conducted using automated particle search software on each. The primary instrument used in this research was an FEI Inspect F50 with TEAM EDS system and GSR Magnum particle search software. The FEI F50 uses a Schottky field emission gun (FEG) for electron generation coupled with an Everhart-Thornley (ET) Secondary Electron Detector (SED) and Solid State Backscattered Electron Detector (SS-BSED). The EDS system operates with a 30mm² EDAX Apollo X silicon drift detector (SDD). The system operates under high vacuum. The system is complete with an eight-spot, motorised, programmable stage plate that allows for multiple sample stubs to be loaded for automated analysis. This system was validated for the analysis of GSR as discussed in the remainder of this chapter.

The secondary instrument (owned by Forensic Science SA, but made available for research) was a Zeiss Evo 50 SEM with Oxford EDX system and INCA particle search software (Inca Suite version 5.04, Issue 21a SP2). The Zeiss Evo 50 utilises a tungsten filament for electron generation coupled with an ET SED and a Qemscan backscattered electron detector (BSD). The EDS system operates with an Oxford Instruments X-Max^N 80mm² SDD. For GSR analysis, this system is run under 'constant vacuum' (i.e., not 'variable pressure' mode.) The sample chamber includes an eight spot, motorised, programmable stage. This system was a part of an operating GSR analysis laboratory attached to FSSA. As such, this system and its operating procedures had previously been validated for the analysis of GSR, and the details of its validation and operation have not been included as a component of this thesis. However, as it is a previously validated system, currently actively used for forensic GSR analysis, it was used as a basis of comparison to determine acceptable performance for the FEI Inspect F50 system.

While SEM-EDS formed the primary technique used for sample analysis, other analytical techniques were utilised for specialised analysis where appropriate. Where additional techniques have been used, full instrumental parameters and conditions are presented in the relevant chapter.

2.4.1. Instrument Operating Conditions

Instrumental operating conditions for the SEM-EDS systems used were established with reference to the ASTM Standard for Gunshot Residue Analysis by Scanning Electron Microscopy/Energy Dispersive X-Ray Spectrometry [13, 36, 153], as well as the SWGGSR Guide for Primer Gunshot Residue Analysis by Scanning Electron Microscopy/ Energy Dispersive X-Ray Spectrometry [37]. The ASTM standard underwent several revisions over the course of the research conducted as a part of this thesis. The majority of the changes were related to the particle classification scheme, and have been discussed in detail in chapter one. Where relevant, the specific version of the standard that is being referred to is noted.

Requirements from the ASTM pertaining to the operation of the instrument itself are:

- The SEM system must be capable of detecting particles down to at least 1.0 μm in diameter, of generating an accelerating voltage of at least 20kV.
- The EDS detector must be capable of acquiring a spectrum at 20eV per channel, with a minimum range of 0-15eV.
- Automated systems must be capable of acquiring X-ray spectra for a user specified collection time or set number of counts, and will be capable of recording and logging particle location coordinates. [13]

The SWGGSR guide further specifies that particles as low as 0.5 μ m in diameter should be able to be detected on the instrument [37]. However, this was revised in the latest version of the ASTM to be a minimum size of 1.0 μ m in diameter [13]. The FEI Inspect F50 operating conditions were established with reference to both the ASTM standard and the SWGGSR guide, as well as the recommendations of the instrument vendor.

The FEI F50 SEM-EDS is coupled with a micro-validation system that can be set up to perform a series of system checks that validate control and performance of the instrument prior to commencing a GSR analysis. These checks include verifying the operation of the SEM-EDS system, including control of beam settings, verifying the magnification is correct, and establishing that the stage movements are accurate. Importantly, it further performs an EDS

calibration on a Cu-Al standard, as well as verifying the EDS resolution at the full width at half of the maximum height (FWHM) of the Mn Kα peak. The micro-validator also performs a number of checks that verify the performance of the instrument, including the relationship between probe current and spot size, between spot size and counts per second (CPS), and between working distance and CPS. These checks establish that the instrument is performing as expected, prior to commencing a GSR run. The micro-validation process was run prior to commencing every GSR run. This ensured that all systems were functional, and ensured the reliability and reproducibility between analysis runs.

For the purposes of GSR analysis, the following settings were used. The system was set at an accelerating voltage of 25kV, in order to enhance the BSE signal at the cost of image quality. The final lens aperture was set to a 40 µm diameter for GSR operations. The typical setting for this instrument was at 30 µm, however it was recommended by the instrument vendor that GSR analysis would benefit from a larger final aperture, resulting in increased probe current to enhance the BSE signal at the expense of a reduced depth of field for imaging. EDS display resolution was set at 2048 channels at 10eV per channel to allow greater resolution and differentiation of neighbouring X-ray lines. EDS spectral range was set over 0 - 20keV. EDS Spectral acquisition conditions were set so that the initial EDS spectrum acquisition time (short) was 3 seconds live time, and the confirmation EDS spectrum acquisition time was 6 seconds live time.

2.4.2. GSR Magnum Particle ID Software

To streamline the labour intensive process of manually searching a sample stub to identify GSR particles of interest, many laboratories employ a software package that allows the system to automatically acquire data while unattended. Generally speaking, this software operates by controlling and coordinating the SEM system, the sample stage, the BSE detector and the EDS system to allow fully automated data collection. This permits a GSR analyst to define the parameters of a search across multiple sample stubs, and then leave the system to collect data for each sample. The system divides each stub into a series of 'frames', dependent on the magnification, and then scans each frame for particles of interest. When the software identifies a particle of interest, it will document its position on the stub in the form of stage coordinates, as well as collecting a thumbnail image to assess morphology and an EDS spectrum to assess composition. If the spectrum indicates particular elements of interest present in an individual particle it can then classify the particle based on its composition. The

analyst can then manually review this data before reporting. The software package used for this purpose on the FEI F50 Inspect Instrument was GSR Magnum (FEI/Eastern Analytical).

The automated particle search system operates in back-scattered electron mode, and was calibrated using a purpose-built BSE calibration standard. For the purposes of particle identification, the system uses this calibration as a means of assessing the relative atomic number (Z) of a particle based on its BSE signal. As the BSE signal increases with Z, heavier elements produce a greater BSE signal than lighter elements, and therefore appear to be brighter in the resulting image. To that end, C, with a low BSE signal, appears to be duller and darker in frame, while Au, with a much higher BSE signal, appears bright. This relationship can be observed in Figure 7. When calibrated, the system then uses the relative brightness of an area of the image as a means of quickly assessing where particles of interest may be located. Once they have been identified, the system can then position the beam and collect an EDS spectrum at this site. The BSE signal was calibrated using the GSR Magnum software such that particles containing high Z elements (e.g., – Au, Pb, Sb, Ba) appear bright against a low Z (i.e., – C) background.



Figure 7 - BSE Images of Au/Nb/Ge/Si/C standard (Ardennes Analytique) demonstrating the relationship between brightness and Z.

Z=32

Z = 41

Z=6

Z=14

The Zeiss Evo 50 system utilised a Gold/Cobalt/Rhodium/Carbon (Au/Co/Rh/C) standard (Micro-Analysis Consultants Ltd, United Kingdom). The FEI Inspect F50 instrument utilised a Gold/Niobium/Germanium/Silicon/Carbon (Au/Nb/Ge/Si/C) standard (Ardennes Analytique, sprl, Belgium) for this process. In each case, the BSE signal was calibrated using this standard prior to the commencement of each GSR analysis run.

The GSR Magnum software also permits a level of classification filtering based on the intensity of the BSE signal by allowing the user to define average Z thresholds. This reduces the number of middle Z element identifications by increasing the threshold for BSE signal required for the

Z=79

software to identify a particle. By default, this threshold is defined by the software to be between 25 (Mn) and 82 (Pb). For the purposes of this research, a large number of stubs with a significant number of particles present were searched, and to increase the efficiency of the analysis runs, the lower threshold was increased to 30 (Zn).

As a further component of this calibration, the system ascertains the ideal analysis conditions to ensure that there is sufficient BSE signal from the sample to appropriately characterise the particles present. For the purposes of GSR analysis, this system was set to achieve 50,000 counts on the Au standard based on the recommendation of the software and instrument vendor. This resulted in the selection of a spot size in the range of between 5 and 6, which provided a minimum probe current of 1na. It was found that these values provided the best balance of spatial resolution and signal from the particles for the software to run both smoothly and still detect the required number of particles.

With regard to particle classification, the GSR Magnum software allows the user to define a series of particle classifications based on the individual components detected in the EDS spectrum. The software further allows these classifications to be given a hierarchy, allowing particles of particular compositions to be grouped based on their significance. This is equally as useful in identifying particles of interest as it is in filtering particles of compositions of less significance. Practically, for GSR analysis, this classification hierarchy aligns with the ASTM standard for the analysis of GSR, allowing characteristic, consistent, and commonly associated particles to be defined and identified. The classifications can also be further expanded to include particles of common compositions from environmental sources that may interfere with analysis may be defined as a means of filtering them out. This includes particles originating from lighter flint (LaCe or mischmetal), jewellery (Ag, Au, AuCu), coinage (NiCu), cosmetics (Bi), pigments (BaS) and other interfering metals (Fe, Zn). The particle classifications defined in GSR Magnum as used for this thesis can be seen in Figure 8.

	Enabled	Class Names	Elements/Lines	KRatio Thresholds	Rank	Grou
		PbBaSb	PbL BaL SbL	0.02/1.00 0.04/1.00 0.03/1.00	Major	Non
2		BaSb	BaL SbL	0.04/1.00 0.03/1.00	Major	Non
3	~	PbSb	PbL SbL	0.05/1.00 0.05/1.00	Major	Non
4		Sb	SbL	0.10/1.00	Major	Non
5	✓	PbBa	PbL BaL	0.05/1.00 0.04/1.00	Major	Non
6	~	Ba	BaL	0.06/1.00	Major	Non
7	~	Pb	PbL	0.10/1.00	Major	Non
8	✓	PbClBr	PbL CIK BrK	0.10/1.00 0.02/1.00 0.02/1.00	Major	Nor
9	✓	Fe	FeK	0.16/1.00	Major	Non
10	✓	BaS	BaL SK	0.05/1.00 0.02/1.00	Major	Non
11	~	Ti	TiK	0.10/1.00	Major	Non
12	✓	PbTi	PbL TiK	0.05/1.00 0.02/1.00	Major	Non
13	✓	BaCaSi	BaL CaK SiK	0.08/1.00 0.02/1.00 0.02/1.00	Minor	Non
14	✓	LaCe	LaL CeL	0.07/1.00 0.08/1.00	Major	Nor
15	✓	PbSn	PbL SnL	0.10/1.00 0.06/1.00	Major	Non
16	✓	Sn	SnL	0.12/1.00	Major	Nor
17	✓	Au	AuL	0.27/1.00	Minor	Non
18	~	Ca	СаК	0.17/1.00	Major	Nor
19	~	Cu	CuK	0.14/1.00	Minor	Nor
20	✓	W	WL	0.12/1.00	Minor	Nor
21	v	Hg	HgL	0.15/1.00	Minor	Nor
22	~	Bi	BiL	0.13/1.00	Minor	Nor
23	✓	KCI	KK CIK	0.11/1.00 0.10/1.00	Minor	Non
24	✓	PbCa	PbL CaK	0.09/1.00 0.09/1.00	Minor	Nor
25	✓	Zn	ZnK	0.13/1.00	Minor	Nor
26	~	Ni	NiK	0.19/1.00	Minor	Nor
27	✓	NiCu	NiK CuK	0.17/1.00 0.17/1.00	Minor	Nor
28	✓	CuZn	CuK ZnK	0.10/1.00 0.10/1.00	Minor	Nor
29	✓	9ctAu	AuL CuK	0.10/1.00 0.22/1.00	Minor	Nor
30	~	18ctAu	AuL CuK	0.25/1.00 0.09/1.00	Minor	Nor
31	~	Ag	AgL	0.22/1.00	Minor	Nor
32	v	BaAl	Bal AlK	0.15/1.00.0.17/1.00	Minor	Nor

Figure 8 - Particle classification categories as defined in the GSR Magnum software

Following a GSR analysis run performed using GSR Magnum, all particles of interest were manually relocated on the stub, and their X-ray spectra were re-acquired to confirm their classification. Confirmation involved verifying that all identified elements were present in the spectrum and that peak identification was accurate, as well as confirming that there were no elements present that would preclude classification as GSR. In situations where manual reacquisition was impractical, such as in cases where large numbers of particles were detected on the stub, all spectra were reviewed, but only a representative sub-set of the identified particles were re-acquired.

2.5. SYSTEM VALIDATION

Once instrument operating parameters had been defined to permit the system to perform GSR analyses, the reliability, reproducibility, and performance of the instrument was confirmed.

2.5.1. Plano Synthetic Particle Standard

As a means of verifying the performance of the automated GSR analysis, and testing the accuracy of particle identification and classification, the ASTM guideline recommends that a reference material containing particles of a known size and composition be regularly analysed [13]. While the standard specifies that this can be either a sample of GSR collected from a previously known source, in this case, a synthetic GSR Plano standard was used on both instruments. A Plano standard consists of an aluminium SEM pin stub mounted with a glassy carbon chip with number of deliberately precipitated particles characteristic of GSR (PbSbBa) in a range of sizes between 0.5 μ m and 10 μ m on its surface. Such synthetic particle standards are widely used as a batch analysis positive control and to monitor long term instrument performance drift.

The FEI Inspect F50 used a Plano GmbH, SPS-5P-2 A-X02-Y03 standard as a positive control, run at the start and end of every automated run. This standard contains 100 PbSbBa particles. The number of particles of each size present on this standard can be seen in Table 8. Similarly, the distribution of these particles across the surface of the stub can be observed in Figure 9.

Particle Size	Particles on Plano Standard
(μm)	(n)
2.4 μm	27
1.2 μm	26
0.8 μm	25
0.5 μm	22
Total	100

Table 8 - Particle sizes and numbers present on the Plano SPS-5P-2 A-X02-Y03 Particle Standard



Figure 9 - Plano SPS-5P-2 A-X02-Y03 Standard Particle Map

The Zeiss Evo 50 system utilises a Plano GmbH, SPS-A521-2(27C) standard as a positive control at the start and end of every automated run. This standard contains 43 particles, ranging in size from 1μ m to 5μ m. For an analysis to be considered valid, the automated particle search system had to identify 40 out of 43 particles at both the start and end of the run.

The GSR Magnum software offers a number of settings that allow the specific parameters of the search to be defined. When performing GSR analyses, there is always a trade-off between the time required for the run to complete, and the number of particles detected. A very thorough run can be performed, that identifies a large number of particles, down to the submicron scale, but such a run would take an impractically large amount of time per sample to complete. Similarly, a very rapid analysis can be performed, at the cost of reducing the number and size of the particles detected. As a means of balancing these factors, the GSR Magnum software provides the settings 'Minimum Size (μ m) of particles searched' and 'Minimum number of pixels per particle'. Taken together, these settings determine the search magnification and number of search fields that a sample is divided into for analysis. To that end, balancing these parameters is essential for ensuring efficient, effective and reliable analysis.

The minimum size of particles defines the smallest particles that the system will attempt to classify, and can be defined at any level down to a minimum value of 0.3 μ m. The smaller this value is, the higher the magnification required, and hence, the longer the analytical run will be. Conversely, the larger the value, the lower the required magnification, and the faster it will proceed.

'Minimum number of pixels per particle' determines how many pixels must be distinct from the background in both the x and y directions to be considered by the system to be counted as a particle. This setting can be given a value between 1 and 5, with 1 resulting in a search at the lowest magnification and with the fewest search fields, and 5 operating at the highest magnification and largest number of search fields. With this in mind, the speed of a search performed at 1 minimum pixel per particle will be the fastest, but due to the low magnification, particles of interest may be overlooked by the software. Further, the positioning of the beam during the scanning process may not be appropriately centred on the particle, resulting in the particle being misclassified or missed entirely. This results in a very short analysis time, however this comes with a lack of confidence in results and a high probability that particles present on the stub have been overlooked. Conversely, setting this to 5 increases the confidence in the results, increasing the likelihood that all particles above the size threshold present on the stub will be accurately detected and classified. However, the system achieves this by performing the run at much higher magnification, which divides the stub into more search fields and subsequently requires much more time to complete. The default value for this setting is 2, and is recommended by the software vendor.

In order to ascertain the impact that these settings had on the length and efficiency of the analysis, the Plano standard was analysed multiple times while varying the settings. Each analysis was performed in triplicate and averaged to provide a rough measure of performance. For the purposes of this analysis, particles were considered to be 'detected' if the system accurately indicated that a particle of interest was present at a mapped location and correctly assigned the appropriate classification. Particles were considered 'located but misclassified' if the system was able to indicate that a particle of interest was present at a mapped location, but the classification applied was inappropriate (e.g., classifying a PbSbBa particle as containing PbSb only), or the classification was listed as 'Unclassified'. The software indicates particles are 'unclassified' if they are located and mapped at a specific location, but the collected spectrum is of poor quality and the peaks corresponding to the elements present do not meet the required thresholds to be unequivocally classified. Per the ASTM standard, a

manual review must be performed by an analyst, involving re-location of the particles detected by the system followed by re-acquisition of the X-ray spectra, for GSR to be conclusively identified [13]. All particles placed in either category by the automated system were able to be appropriately classified following manual review of the results of each analysis. Therefore, following manual review these particles were then considered to have been identified as GSR. To that end, the sum of the particles detected and the particles located but misclassified represent the 'particles identified' category. The final category, particles missed, represents particles that were entirely overlooked by the system. A summary of the results of these analyses can be seen in Table 9.

Table 9 - Analysis of Plano standard at 0.5 μm minimum particle size while changing pixels per particle setting

Min. Part. Size (μm)	Pixels Per Particle	No. of fields	Run time (min)	Particles Detected (n)	Particles Located, but misclassified (n)	Total Particles Identified (n)	Particles missed (n)
	4	224	139	55	18	73	30
0.5	3	120	82	44	32	76	27
0.5	2	56	46	32	32	64	39
	1	12	9	11	24	35	68

n.b. 'No. of fields' refers to the number of analysis fields the software divides the search area into for analysis. The greater the number of fields, the higher the magnification.

Initial runs were conducted with the system set at a minimum particle size of 0.5 μ m, as this was the lower limit specified by the ASTM standard at the time of validation. Total analysis times range from 9 minutes at the least sensitive setting to just over two hours at the most sensitive considered. The results presented in Table 9 indicate that even at the most sensitive setting of 4 pixels per particle, although approximately 70% of the total particles were identified following manual review, 25% of that total had initially been misclassified, and a further 30% of the particles on the standard were missed entirely. Due to the generally poor performance of the instrument running at 1 - 4 pixels per particle, a run was not performed at 5 pixels per particle. Instead, in an attempt to increase the sensitivity of the system in order to ensure that more particles were detected and identified, this process was repeated, but setting the system to the minimum particle size setting of 0.3 μ m. The results of these sample runs can be seen in Table 10.

Min. Part. Size (μm)	Pixels Per Particle	No. of fields	Run time (min)	Particles Detected (n)	Particles Located, but Misclassified (n)	Total Particles Identified (n)	Particles missed (n)
	5	924	425	52	23	75	28
	4	598	341	54	18	71	32
0.3	3	340	192	53	15	69	34
	2	143	92	44	24	68	35
	1	42	47	36	18	54	49

Table 10 - Analysis of Plano standard at 0.3 µm minimum particle size while changing pixels per particle setting

n.b. 'No. of fields' refers to the number of analysis fields the software divides the search area into for analysis. The greater the number of fields, the higher the magnification.

As can be seen from Table 10, the higher sensitivity setting results in analysis times ranging from 42 minutes at the lowest minimum pixels per particle setting, to just over seven hours at the highest setting. This result was similarly troubling however, as even at the highest setting, which should have the greatest level of confidence in detection and classification, the system was still routinely overlooking particles that were known to be present on the standard.

In order to ascertain which particles were being overlooked, the data was separated out based on the size of the individual particles. The results of these analyses can be seen in Table 11. As a means of comparing the performance of different instruments and laboratories, instrument performance is converted into a z-score in accordance with the International Organisation for Standardisation (ISO) [154-156]. These z-scores are also the means by which different laboratories are compared when participating in 'round robin' style intralaboratory proficiency testing, such as those facilitated by the European Network of Forensic Science Institutes (ENFSI) [156]. The z-score is calculated for each particle size category by the formula:

$$z = \frac{x_p - x_a}{\sigma_t}$$

Where x_p represents the number of particles identified by the individual laboratory or instrument, x_a represents the actual number of particles present on the sample, and σ_t represents the target standard deviation. For ENFSI proficiency tests, satisfactory performance is defined as corresponding to z-scores between -2 and 0, questionable performance between -3 and -2, and unsatisfactory performance for values lower than -3 [156]. Target standard deviations are provided as an element of the ENFSI report for the analysis of proficiency test results, and are set to an upper limit of 10% of the assigned value. To further assess the performance of the FEI F50, z-scores were calculated for each particle size. These data can be seen in Table 12.

As can be seen from the results in Table 11, of the 53 particles above 1.0 μ m, greater than 90% were detected regardless of the settings. When considering the sub-micron particles, these data indicate that although there was a tendency for particles in the 0.8 μ m to be misclassified more frequently than the particles greater than 1 μ m in diameter, the majority of these particles were identified using everything but the least sensitive settings. Taken together, this indicates that in a majority of cases the system was capable of detecting more than 85% of the particles present on the Plano standard. However, in all cases the system had difficulty detecting particles at 0.5 μ m. The calculated z-scores present in Table 12 support this assessment. In all cases the system exhibits satisfactory performance, with z-scores between - 2 and 0 for particles of diameter 2.4 μ m and 1.2 μ m.

Min.					Detected -	Correctly	classified			Locate	d - Misclas	<u>sified</u>				Missed		
Part. Size (μm)	Pixels Per Particle	No. of fields	Run time (min)	2.4μm (n)	1.2μm (n)	0.8µm (n)	0.5μm (n)	Total	2.4μm (n)	1.2μm (n)	0.8µm (n)	0.5μm (n)	Total	2.4μm (n)	1.2μm (n)	0.8µm (n)	0.5μm (n)	Total
	5	924	425	27	26	0	0	53	0	0	21	0	21	0	0	4	22	26
	4	598	375	25	23	6	0	54	2	2	15	7	24	0	1	4	15	22
0.3	3	340	177	23	24	6	0	53	4	2	15	1	18	0	0	4	21	29
	2	143	78	24	19	2	0	45	3	6	17	0	24	0	1	6	22	31
	1	42	36	23	4	0	0	27	4	22	2	0	28	0	0	23	22	45

Table 11 - Analysis of Plano standard at 0.3µm minimum particle size while changing pixels per particle setting, separated by particle size

n.b. Data is based on three runs, averaged and rounded to the nearest whole particle.

Minimum					Target	
Particle	Pixels	Particle	Particles	Particles	Standard	
Size	per	Size	Identified	Present	Deviation	
(µm)	particle	(µm)	(x_p)	(x_a)	(σ_t) [156]	z-score
		2.4	27	27	0.6	0.0
	5	1.2	26	26	1.6	0.0
	J	0.8	21	25	2.6	-1.5
		0.5	0	22	2.5	-8.8
		2.4	27	27	0.6	0.0
	1	1.2	25	26	1.6	-0.6
	4	0.8	21	25	2.6	-1.5
		0.5	7	22	2.5	-6.0
	3	2.4	27	27	0.6	0.0
03		1.2	26	26	1.6	0.0
0.5		0.8	21	25	2.6	-1.5
		0.5	1	22	2.5	-8.5
		2.4	27	27	0.6	0.0
	2	1.2	25	26	1.6	-0.6
	2	0.8	19	25	2.6	-2.3
		0.5	0	22	2.5	-8.8
		2.4	27	27	0.6	0.0
	1	1.2	26	26	1.6	0.0
	T	0.8	2	25	2.6	-8.8
		0.5	0	22	2.5	-8.8

Table 12 - Calculated z-scores for FEI F50 at 0.3 μm min. particle size and varied pixels per particle.

n.b. Green cells indicate satisfactory performance (z-scores from -2 to 0), orange cells indicate questionable performance (z-scores from -3 to -2), and red cells indicate unsatisfactory performance (z-scores less than -3). Data is based on three runs, averaged and rounded to the nearest whole particle.

At all settings, the system displayed unsatisfactory performance for particles of diameter 0.5 μm. At the 1 pixel per particle setting, the FEI F50 exhibits unsatisfactory performance for particles below 1 μm. To that end, it was determined that this setting was inappropriate to use for further GSR analyses. At 2 pixels per particle, the system exhibits questionable performance for particles of diameter 0.8 μm. At the 3, 4 and 5 pixels per particle settings, the system exhibited satisfactory performance at all particle sizes 0.8 μm and above. However, from Table 11, it can be seen that analysis at these settings requires between approximately 80 minutes and seven hours. At 7.1 and 6.2 hours respectively, it was determined that the 5 and 4 pixels per particle settings were too time consuming to be used for future analysis, especially considering that there was no significant gain in performance observed at the higher settings. In order to ascertain if the performance issues were as a result of the system or the

Plano standard, a comparison between the FEI F50 instrument and the Zeiss Evo 50 instrument was conducted.

2.5.2. Instrument Comparison

Once the performance of the FEI F50 instrument had been explored and the best settings identified, it was identified that the performance of this instrument should be compared against the Zeiss Evo 50. This had a number of advantages, chief among them being the comparison of the performance of the new instrument against an instrument that was already used to perform GSR analyses. Further, it was surprising that the FEI F50 had such difficulty detecting 0.5 μ m particles on the Plano standard, so this comparison also served as a troubleshooting step to ascertain if the performance issues of the FEI F50 system were a result of the instrument, analyst, or the sample.

To compare the performance of the FEI F50 instrument against the Zeiss instrument, the Plano sample was run on both systems. To perform this comparison, the Plano SPS-5P-2 A-X02-Y03 sample stub was provided to a GSR analyst at FSSA, and they were asked to set up and run the Zeiss Evo 50 instrument as they normally would when performing a GSR analysis. It should be noted that this system also uses a Plano sample as a positive control, and therefore the analyst was familiar with the requirements for the use of a Plano standard, and the Zeiss system is capable of performing analysis of same. The results of these analyses can be seen in Table 13.

Run Number	Mag. (x)	No. of Fields	Area (mm²)	Run time (min)	Particles Identified
1	451	868	42.7	116.6	57
2	451	845	41.6	107.6	74
3	500	1261	41.6	172.8	67
4	550	1257	41.6	173.3	67

Table 13 - Performance of the Zeiss Evo 50 on Plano SPS-5P-A-X02-Y03

The analysis was initially performed twice as a means of verifying the variability of results. The first run identified 57 particles, the second 74 particles. In order to resolve this discrepancy, the sample was run two further times, at increased magnification, until a consistent identification was achieved. Both of the two further runs identified 67 particles in approximately 3 hours. This corresponded to identifying all of the particles greater than 1 μ m, and just over half (14) of the particles of 0.8 μ m. The analyst reported that the Zeiss system had similar difficulties identifying particles of 0.5 μ m diameter. A side-by-side comparison of the instrument results can be seen in Table 14.

Instrument	Min. Particle Size (μm)	Pixels Per Particle	Run Time (mins)	Particles Identified (n)
	0.2	3	177	75
FEIF50	0.5	2	78	71
Zeiss Evo 50			173	67

Table 14 - Performance comparison of Plano Standard on the FEI F50 and Zeiss Evo 50 systems

On the strength of this comparison, this indicated that the FEI F50 system performed marginally better than the Zeiss system; it identified more particles, on average, in a comparable analysis time when at the 3 pixels per particle setting. In fact, at the 2 pixels per particle setting, the FEI still exhibited better performance than the Zeiss instrument, in less than half the analysis time. This is unsurprising, as the FEI F50 is a much newer instrument, and uses a field emission gun (FEG) as compared to the Zeiss system's W-filament. However, this does indicate that the commissioned FEI system is at least as capable as the Zeiss system, a system which is already used to perform GSR analysis for casework. This comparison further indicated that in the interests of economising analysis time, it was not worth the relatively minor increase in sensitivity, at the cost of doubling the analysis time when running the system at 3 pixels per particle. These comparison data indicate that the FEI F50 system performs marginally better than an existing SEM-EDS system used for forensic GSR analysis, even when set at 2 pixels per particle. To that end, it was determined that by default, the FEI F50 system would be run at 0.3 µm minimum particle size, and 2 pixels per particle for all future GSR analyses, It was therefore concluded that at these settings, the FEI system was performing at a level that was acceptable for performing forensic GSR analysis, both in reference to the ASTM, and the performance of existing instruments already used for this purpose. However, as a final validation step, the performance of the FEI F50 was compared to other forensic GSR analysis instruments by analysing a GSR proficiency test sample.

2.5.3. ENFSI Proficiency Test Performance

Having determined that the FEI F50 system was performing to a comparable standard to an existing GSR analysis instrument, the next step was to determine how both instruments, as well as analytical processes, performed in comparision to a number of other operating forensic GSR analysis laboratories world-wide. To accomplish this, a previous proficiency test stub was obtained and analysed on the FEI F50 system.

The European Network of Forensic Science Institutes (ENFSI) conducts regular, 'round robin' style proficiency tests of a number of forensic science laboratories that perform GSR analysis. As a part of this proficiency test, all participating laboratories are provided with a sample with a known number of characteristic GSR particles of different sizes present on the surface. Laboratories are tasked with analysing the sample following standard laboratory procedure from GSR analysis, and reporting on their results. Once this is done, the results from different laboratories are interrogated and then circulated by ENFSI, and a comparison of different laboratories can be observed.

The sample provided for analysis is similar in construction to the Plano standard used as a positive control. The sample consists of a standard size 12.5 mm aluminum SEM stub with a glassy carbon chip mounted on its surface. In the middle of this chip is a region of interest, measuring $6x6 \text{ mm}^2$ which contains a deliberatly precipitated, known number of characteristic GSR particles. These particles range in size between 2 µm and 0.5 µm, and their size and location is mapped. The proficiency test used in this case was the ENFSI GSR 2014 test, as this was the most recently available GSR Proficiency test that could be obtained.

To avoid bias in the interpretation of the results, steps were taken to ensure that the analysis process mirrored the conditions of a proficiency test as much as possible. The analysis was performed blind, with the proficiency test stub initially provided with the instructions for the test only. The stub was analysed using SEM-EDS on the FEI Inspect F50 instrument, and the results intepreted and reported. Once this was completed, the results provided by ENFSI were made available, and compared to the reported results. With this in mind, it was up to the analyst to use the established GSR analysis procedure and their own knowledge in analysing, reviewing, and reporting on the GSR present on the stub before they were aware of the results. For the purposes of the ENFSI stub analysis, the FEI F50 system was run at a setting of 0.3 µm minimum particle size and 2 pixels per particle. As described above, this setting

sacrificed a small amount of sensitivity in detecting particles below 1.0 μ m, for a large reduction in analysis time.

The results obtained for the ENFSI GSR2014 proficiency test using the FEI Inspect F50 can be seen in Table 15, below.

Particle Size (μm)	Particles Expected (n)	Particles Identified (FEI Inspect F50) (n)
2.0	26	26
1.5	27	27
1.0	28	28
0.75	26	20
0.5	25	4
Total	132	105

Table 15 - Particles present on the ENFSI GSR2014 sample and particles detected by the FEI Inspect F50

Of the 132 particles present, 105 were detected and subsequently identified, indicating that the FEI Inspect F50 instrument was capable of detecting 79.5% of the particles present. Review of the results indicated that no particles had been mistakenly excluded by the analyst. As previously described, ENFSI uses z-scores as a means of comparing the performance of different laboratories. To that end, z-scores for each particle were calculated based on the results in Table 15, and can be seen in Table 16.

Table 16 - Calculated z-scores for the FEI Inspect F50 using ENFSI GSR2014 sample, running at 0.3 μm minimum particle size and 2 pixels per particle

Particle Size (μm)	Particles present (n)	Target s.d. [156]	Particles Identified (FEI Inspect F50) (n)	z-scores
2.0 μm	26	0.555	26	0
1.5 μm	27	1.012	27	0
1.0 µm	28	1.59	28	0
0.75 μm	26	2.6	20	-2.3
0.50 um	25	2.5	4	-8.4

n.b. Green cells indicate satisfactory performance (z-scores from -2 to 0), orange cells indicate questionable performance (z-scores from -3 to -2), and red cells indicate unsatisfactory performance (z-scores less than -3).

From the z-scores, in Table 16 it can be seen that based on the standards described by ENFSI, the analysis is considered 'satisfactory' for all particles greater than 1 μ m, 'questionable'

performance for particles in the 0.75 μ m size domain, and 'unsatisfactory' performance for particles of 0.50 μ m. This indicates similar results to those observed in the Plano analysis and benchmarking studies above. The ENFSI standard was also analysed using the Zeiss Evo 50. The results can be seen in Table 17.

Particle Size (μm)	Particles Expected (n)	Particles Identified (Zeiss Evo 50) (n)
2	26	26
1.5	27	27
1	28	28
0.75	26	14
0.5	25	0
Total	132	95

Table 17 - Particles present on the ENFSI GSR20	14 sample and particles detected by the Zeiss Evo 50
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From the table it can be seen that the Zeiss instrument was capable of detecting and subsequently identifying approximately 72% of the particles present. Following the same process of comparison of results based on z-scores, it can be seen in Table 18 that this instrument also achieved 'satisfactory' results for particles greater than 1 μ m, but 'unsatisfactory' results for both the 0.75 μ m and 0.50 μ m particle sizes.

Particle Size (µm)	Particles present (n)	Target s.d. [156]	Particles Identified (FEI Inspect F50) (n)	z-scores
2.0 μm	26	0.555	26	0
1.5 μm	27	1.012	27	0
1.0 µm	28	1.59	28	0
0.75 μm	26	2.6	14	-4.6
0.50 μm	25	2.5	0	-10

Table 18 -	Calculated	z-scores for	the Zeiss	: Evo 50
	culculated	2 300103 101		, LVC 30

n.b. Green cells indicate satisfactory performance (z-scores from -2 to 0), orange cells indicate questionable performance (z-scores from -3 to -2), and red cells indicate unsatisfactory performance (z-scores less than -3).

These results are comparable to the performance of the two instruments on the basis of their examination of the Plano SPS-5P-A-X02-Y03 standard analysed previously. Although it can be stated that the FEI Inspect F50 performs marginally better than the Zeiss system, both systems experience difficulty in detecting particles smaller than 1 μ m. However, this issue seems to be more frequently encountered with the particles on Plano-type samples, and both instruments are capable of routinely detecting particles under 1 μ m in case samples. It should be further

noted that while the FEI Inspect F50 system has demonstrated the ability to detect smaller particles on a routine basis, to do so requires significantly increased analysis time.

ENFSI provides a report, presenting the results of all laboratories that participated in the 2014 GSR proficiency test, which is not published publically. However, based on this report,

, it was noted that a large number of laboratories either do not analyse particles below 1.0 μ m, or exhibit unsatisfactory performance at this level. Taken together, a majority of the laboratories that participated in the ENFSI trial either did not analyse 0.5 μ m particles or showed unsatisfactory performance. Similarly, for 0.75 μ m particles, nearly half of laboratories either did not analyse particles of this size or did not perform at a satisfactory level. This suggests that either laboratories are using instruments that are not sensitive enough to identify particles of less than 1.0 μ m in diameter, or they are unable to validate their system in order to accurately report in this size domain. In either case, all of the laboratories that participated in this ENFSI proficiency test are forensic laboratories that routinely perform GSR analysis. Based on the ENFSI report the performance of the FEI Inspect F50 system is comparable in performance to the majority of other laboratories – exhibiting satisfactory performance in identifying particles above $1.0 \,\mu$ m, while having difficulty in identifying particles below 1.0 μm. It is worthy of note that although previous versions of the ASTM stipulated that SEM-EDS analysis systems for GSR analysis be capable of detecting particles down to at least 0.5 μ m in diameter [36], this has since been revised to down to at least 1.0 μm in diameter [13]. This change is possibly a response to ENFSI proficiency test results indicating that the majority of forensic GSR laboratories either cannot or do not, detect GSR particles below 1.0 µm.

In the context of this thesis, the purpose of this research is to collect data and develop statistics that may be used to inform case-evidence evaluation. Based on the results observed from the ENFSI GSR2014 proficiency test, the FEI Inspect F50 SEM-EDS with GSR Magnum particle analysis system utilised for this research performs at a level comparable to the majority of working forensic GSR analysis laboratories that contributed to this survey. Practically therefore, on an analytical level, it would be expected that if another lab were to analyse the experimental stubs collected as a part of this research, they would report broadly similar results. Although the system exhibits unsatisfactory performance for particles below $1.0 \,\mu\text{m}$, it is consistent with other forensic GSR analysis instruments globally, and is therefore fit for purpose.

2.6. CONCLUSIONS

The planning, experimental design and methods for this research project were established with specific reference to both the ASTM standard for the analysis of GSR[13, 36, 153] as well as the SWGGSR guide for GSR analysis [37]. This information was augmented through consideration of currently established sample collection, handling, and analysis procedure from both SAPOL and FSSA.

When considering collecting samples from human subjects, appropriate ethics approval was sought and all personal data and identifying information was redacted, keeping the samples collected anonymous. Review of case files was conducted under approval from the South Australian State Coroner, with certain information redacted to preserve victim anonymity.

The FEI Inspect F50 SEM-EDS system was validated for the analysis of GSR with reference to the ASTM standard for the analysis of GSR, and using a Plano synthetic particle standard (SPS-5P-A-X02-Y03). Despite persistent difficulties in achieving satisfactory performance on the Plano standard for particles below 1.0 μ m, the FEI Inspect F50 system performance was observed to be comparable to a Zeiss Evo-50 SEM-EDS system that is currently used for forensic GSR casework in South Australia. Similarly, an ENFSI GSR proficiency test was performed that indicated that the system performance was consistent with the majority of other GSR analysis instruments in working forensic GSR analysis laboratories around the world. Ultimately, settings were selected that provided satisfactory performance in identifying and classifying particles greater than 1.0 μ m in a suitable analysis time, offering comparable performance to other operating forensic GSR analysis laboratories.

3.FIREARMS AND AMMUNITION ENCOUNTERED IN FORENSIC CASEWORK IN SOUTH AUSTRALIA

3.1. SYNOPSIS

The amount and type of GSR formed and distributed following a firearm discharge is contingent on a number of variables. Among these are the type of ammunition and the type of firearm used. The type of primer composition of the ammunition used influences the composition and morphology of the particles formed [157], and the firearm used influences the spread of particles around the firearm [83]. Further, additional complexity to the chemistry of the resultant GSR particles may be introduced as a result of incorporation of additional elements from the projectile, its jacketing material, the cartridge material, or indeed elements from the firearm itself [10, 17, 18, 20, 25, 26]. Therefore, an assessment of GSR evidence may be supported by data which informs these variables, amongst others. This allows GSR examiners to have an understanding of what findings may be expected given the context of the case under investigation. The frequency of firearm and ammunition types in a particular jurisdiction is influenced, to some extent, by the firearms legislation of the specific jurisdiction and its impact on firearms and ammunition availability, among other factors. As a means of developing an understanding of the South Australian firearms environment, a comprehensive review of GSR analyses performed by FSSA in South Australia was performed, with the case circumstances and data interrogated for information that could be collated to inform future GSR analyses. Of specific interest were the types and frequency of firearms and ammunitions that were encountered in recent casework. Two sets of case files were interrogated for this purpose, the first encompassing suicide cases, before being expanded to cover all cases in which a GSR analysis was performed.

These data provided interesting insight into the South Australian firearms environment. Rifles were the most frequently encountered firearm type, with 0.22 calibre rimfire rifles being the most prevalent sub-classification. Shotguns were the next most frequently observed, with 12g variants being the most popular. Pistols were only encountered in a minority of cases, and showed much more diversity in calibre, with 0.22, 9mm and 0.357 calibre variants being relatively frequently observed. With regard to ammunition manufacturers, this survey indicated that Winchester brand ammunitions were the most frequently observed. It should be noted however, that the ammunition manufacturer was not recorded in a majority of the cases considered. With regard to the GSR particle compositions observed in this review, it was found that PbSbBaSi and PbSbBa particles were the most frequently observed particle types.

understanding that GSR generated reflects the components of the priming compound. The Sicomponent may be attributable to the presence of glassy GSR (gGSR), made up of the glass frictionator components of the priming compound encrusted with Pb and Ba from the primer. The significance of gGSR has been explored elsewhere [158-160]. The data also indicated that Si and Al were among the most frequently observed elements incorporated into characteristic GSR occurring in over 70% of the particles observed, with Cu and Fe being the next most frequently observed at 46% and 49% respectively.

In both sets of data, particles containing Sb as a component were observed in circumstances where they were unexpected based on the composition of the primer. A significant portion of 0.22LR rimfire ammunitions, seen frequently in these data, do not contain Sb as a component of the priming composition, and therefore are not expected to generate particles characteristic of GSR. These data indicate that this is not the case, and that characteristic, three component particles may be present in circumstances where only two component primers are used. Initial investigations into the mechanism behind this have suggested that Sb may be incorporated into these particles from the surface of the projectile, where Sb is used to harden the Pb. It is also possible that the weapon memory effect has resulted in Sb particles, or indeed PbSbBa particles being retained within the firearm from previous firings of three-component primed ammunition, and then distributed from the firearm with subsequent firings. However, these data also indicated that brands of rimfire primers containing Sb are not commonly encountered in forensic casework in SA, suggesting that this mechanism is not the most probable source of three-component particles from two component primers. Although the use of three-component primed ammunition could never be excluded based on the observation of a GSR particle population alone, these collected data suggest that the formation of three-component GSR from two-component primed ammunition with projectile inclusions must also be considered. Further investigation behind this phenomenon will work to inform the mechanisms behind GSR particle formation, and aid in interpretation of GSR evidence.

Ultimately, these surveys supported the view that 0.22LR rimfire rifles and 12g shotguns are among the most frequently encountered firearms in South Australian forensic GSR casework, in both cases of suicide and wider criminal casework. This is of particular interest as GSR research from other parts of the world tends to focus on the firearms that are most frequently encountered in their own jurisdictions. In many cases, this focus has been on handguns [116, 161], leaving circumstances involving 0.22 rifles and shotguns comparatively underrepresented in literature. Therefore, information obtained from these case reviews focussing on jurisdiction specific firearms and ammunitions can be used to better inform assessments of GSR evidence in South Australia.

3.2. BACKGROUND

In the context of this thesis, the study forming the basis of this chapter was performed as a means of better understanding the firearms environment in South Australia. While data exists for the types, calibres, and styles of firearms that are in circulation [8, 9, 162], only a small portion of these are encountered in forensic casework. Therefore, in order to address strategies that can be used to strengthen and support the assessment of GSR evidence, it is instructive to understand the types of cases, including the firearms and ammunition involved, that are most frequently encountered as a starting point.

In order to determine the significance of a GSR finding, there are a number of different variables that must be considered. One major variable that influences the persistence of GSR on the hands of a shooter is the amount of activity undertaken after the GSR has been deposited. Once deposited, GSR particles are relatively easily shed from the hands, and may be lost through regular activity including washing, wiping or rubbing hands, or activities conducted in the process of arrest [44, 112]. Initially, the study of suicide cases was considered as a means to assess GSR results while controlling for the activity level of the shooter. In the case of most instances of suicide, the shooter discharges a single shot, remains at the scene of the discharge, and does not undertake significant activity after firing. Under these conditions, it was assumed that they would therefore be exposed to the maximum amount of GSR settling. Additionally, the review of these cases allowed for the collection of GSR data from a variety of firearms and ammunition types that were typical of the mix of firearm types available in South Australia across the time period in question.

A further sample of all cases between 2007 and 2016 which involved a firearm and required GSR analysis were interrogated for their GSR results. By expanding the sample of case files to include all firearms cases involving GSR analysis, firearms that were rarely featured in suicide, but were more common in other firearms crime were captured. In this instance, cases were not filtered by the type of offence or the circumstances of the case. As a result, the data assessed in this case is likely to be more reflective of the types of firearms and ammunition used in all crime in South Australia in the time period in question. This sample set was also interrogated for GSR particle composition data to ascertain if particular compositions or elemental inclusions were more common from particular firearm types or ammunitions. The collected data could then serve as a foundation for the generation of a GSR case database which could then inform future assessments of GSR evidence.

In the context of an applied Bayesian network approach to the evaluation of GSR evidence, any data gained about the composition, distribution or transfer of GSR from different ammunitions and firearms can be used to inform the nodes related to assessing Hp. While these factors are highly dependent on the individual circumstances of the case, having an understanding of expectations based on the experience of the analyst can serve as a baseline to form the assessment that is to be performed.

Elements of the research conducted in this chapter were peer-reviewed and published [163]. This first appeared in:

N. Lucas, M. Cook, J. Wallace, K.P. Kirkbride, H. Kobus, Quantifying gunshot residues in cases of suicide: Implications for evaluation of suicides and criminal shootings, Forensic Science International **2016**, 266, pp. 289-298

For the purposes of this publication, the approximate contribution of each author was N. Lucas 50%, M. Cook 15%, H. Kobus 15%, K.P. Kirkbride 15%, J. Wallace 5%.

The full text of the publication has been incorporated in section 3.3 below.

Please note – Minor formatting amendments have been performed to the presentation of the publication to keep it consistent with the presentation of the thesis, however text and data remain unchanged from the published version.

This publication prompted a Letter to the Editor. It was originally published as:

A. Zeichner, Antimony content of inorganic gunshot residue (IGSR) produced by 0.22 caliber rimfire ammunition having free-antimony primer, Forensic science international **2017**, 270, pp. e26-e27

A response to this letter to the editor was then published [164]. It was published as:

N. Lucas, M. Cook, J. Wallace, K. Kirkbride, H. Kobus, Author's response-Letter to the Editor (FSI-D-16-00737), Forensic science international **2017**,270, pp. e28-e29.

For the purposes of this publication, the approximate contribution of each author was N. Lucas 55%, M. Cook 5%, H. Kobus 15%, K.P. Kirkbride 15%, J. Wallace 5%.

The full text of this publication has been incorporated in section 3.4.

3.3. QUANTIFYING GUNSHOT RESIDUES IN CASES OF SUICIDE: IMPLICATIONS FOR EVALUATION OF SUICIDES AND CRIMINAL SHOOTINGS

3.3.1. Introduction

Gunshot residue (GSR) is generated during the discharge of a firearm and originates from the ignition of the primer and propellant charge within the ammunition cartridge. These residues disperse as a plume from the barrel, breech, and any gaps in the frame of the firearm. As the plume cools, it condenses to form small particles that settle in the surrounding environment, on the shooter, and on any bystanders. The amount of residue initially deposited on the shooter is dependent on the type of ammunition and firearm used [83]. GSR is a valuable form of forensic trace evidence in firearms investigations.

The quantity and type of GSR particles transferred to the shooter immediately after discharge of a firearm is of fundamental importance to an accurate assessment of the value of GSR evidence. Once deposited, the persistence of GSR on a suspect then becomes important and may be influenced by a number of factors. GSR particles are easily shed from the hands, and may be lost through regular activity – putting hands in and out of pockets, washing, wiping or rubbing hands, or activities conducted in the process of arrest [112]. While an exact, quantitative model for transfer of GSR and evaluation of the evidence has been elusive thus far [149], it is commonly considered that the greater the amount of GSR detected on a suspect, the more likely that the GSR originated from a firing incident, rather than from secondary or tertiary transfer [165]. Understanding the mechanism behind particle loss allows for a more comprehensive understanding of the significance of a GSR test result. Taken together, the transfer and persistence of GSR on a suspect has been, and remains, a highly pertinent question for forensic scientists and, ultimately, the courts.

Although there is a significant body of knowledge in regards to transfer and persistence of GSR particles, especially primer derived particles ([88, 106, 111]), there are some substantial gaps, such as the quantity of deposition of GSR as a result of a single shot, and deposition from shotguns and 0.22 rimfire rifles, which are weapons that feature prominently in crime in Australia. One way of quantifying the transfer of GSR to the hands of a shooter, short of

setting-up test firings involving a range of firearms and a range of calibres, is to measure deposition of particles on victims of firearm-related suicides. Assuming that there is no question that the deceased discharged the firearm, measurement of particle deposition as a result of suicide allows many variations of shooting scenarios in a real world environment to be considered. The amount of activity undertaken after firing is a major variable affecting the persistence of GSR. In cases of suicide, the shooter typically fires a single shot, remains at the scene, and engages in no further activity after firing – remaining undisturbed beyond influences from the external environment. A collateral benefit of a survey of suicides is that the data are directly applicable to the investigations of suicide.

Demographic differences and variation in firearm regulations lead to significant differences in firearms availability and their use in suicide. In areas in which firearm control legislation is strict, such as the UK, the rate of suicide by firearms is significantly lower, with approximately 2% of total cases of suicide involving a firearm. There, the firearm type used tends to be a shotgun, (up to 70% of cases [166]), with rifles being the weapon of choice in less than 10% of cases; this seems to reflect particular firearm ownership regulations in the UK. By comparison, in the United States between 2010 and 2012 firearms were the most prevalent method of suicide, being used in over 50% of recorded cases [167]. A more detailed study conducted by Kohlmeier et al [161] indicated that handguns were the most prevalent firearm type, accounting for 78% of all suicide cases observed in the period of the study, with less than a quarter of all cases involving a rifle or shotgun. In Australia, where firearms regulation is relatively strict, but not as strict as in the UK, the number of cases of suicide involving firearms typically accounts for less than 10% of total cases of suicide in a given year [168]. Based on census data, of that figure, suicide by 'rifle, shotgun or larger firearm discharge' (where rifle refers predominantly to 0.22 calibre) accounts for a full 70% of all cases on both a national and state level [169].

A study of GSR deposition during suicide in Australia will therefore yield data mainly relating to deposition arising from discharge of shotguns and 0.22 calibre rifles which, as indicated above, represent a knowledge gap. The aim of this study was to examine case data from a statistically-significant number of shooting suicides in South Australia and extract data that could be used to model GSR transfer in suicides and criminal cases that involve single shots from firearms, in particular shotguns and 0.22 calibre firearms.

3.3.2. Materials and methods

Case Selection

Coronial cases of suicide involving a firearm occurring in South Australia between 1998 and 2014 were investigated for gunshot residue test results. The approval of the State Coroner was sought and received prior to case review. Identifying details were redacted in order to preserve the anonymity of victims. Only cases involving unambiguously self-inflicted gunshot wounds resulting in death were considered. Cases involving dyadic death (i.e., murder-suicide) or "suicide by cop" (i.e., forcing a confrontation with police to provoke the use of lethal force) were excluded. Information collected consisted of the GSR analysis results and details on the analysis request forms completed by the investigating officer. A total of 71 cases were identified and considered in this report.

Equipment

Samples were analysed by SEM-EDS at Forensic Science SA (FSSA) in accordance with operational procedure. Instrument operating parameters were established as per the American Society of Testing and Materials (ASTM) E1588-16 Standard guide for gunshot residue analysis by SEM/EDS [153]

Cases prior to 2006 were analysed using a Camscan MX2500 Series 4 SEM with EDitor EDX system and MX particle analysis software. Cases between 2006 and 2014 were analysed using a Zeiss Evo 50 SEM with Oxford EDX system and INCA Aztec particle analysis software.

The Camscan system brightness, contrast, and particle search system settings were calibrated through the use of a positive control, a stub known to have genuine GSR particles present, prior to every run.

The Zeiss automated particle search system brightness and contrast settings were calibrated through use of a Gold/Cobalt/Rhodium (Au/Co/Rh) standard.

A positive control for the Zeiss system, a synthetic particle standard (PLANO W. Plannet GmbH, Wetzlar, Germany, SPS-A521-2(27C)), consisting of accurately deposited particles of known size was analysed at the start and end of every sample run. The "particles" present on the standard are thin Pb/Sb/Ba films of sizes 1-5 μ m.

Data Analysis

The size and software classification of particles were reviewed in accordance with ASTM E1588-16. Particles were classified as "characteristic" of firearms origin or "consistent" with firearms origin in reference to this standard. In situations where a significantly large number of characteristic particles were identified on a stub, the analysis was suspended prior to completion. While the analysis was left incomplete, the percentage portion of the stub analysed was recorded. In situations where less than the whole sample stub was analysed, data were recorded based on the final reported results. In these situations, particle counts are likely to under-estimate the number of particles present. In situations where appropriate, these data have been highlighted.

We define the post-firing interval as the period between the estimated time of firearm discharge and the documented time of GSR sample collection. In circumstances where the post-firing interval was expressed as a range, the maximum value was taken for all further data treatment.

0.22 Rimfire ammunitions and Antimony

Samples collected to investigate the generation of three-component, characteristic particles from ammunition containing Sb-free primers were collected and analysed. Sample collection involved a single shot of PMC Zapper 0.22LR rimfire ammunition, known to use antimony-free primers, fired from a Ruger 10/22 rifle. The residue expelled from the barrel of the rifle was collected in a plastic catcher. Unburned propellant was removed, and the insides of the catcher sampled with a GSR collection stub mounted with adhesive carbon tape.

Prior to firing, the rifle was thoroughly cleaned using a cable pull through to clean the barrel, followed by cleaning with acetone. Blanks and controls were collected to verify that the firearm was adequately clean before firing.

Analysis of these samples was performed using a FEI F50 SEM system with GSR magnum particle analysis system and TEAM EDAX elemental mapping software. The particle search system brightness and contrast settings were calibrated through use of a Gold/Niobium/Germanium/Silicon/Carbon (Au/Nb/Ge/Si/C) standard (Eastern Analytical).

A positive control, Synthetic Particle Standard (PLANO W. Plannet GmbH, Wetzlar, Germany, SPS-5P-2A-X02-Y03), was analysed at the start and end of each run.
3.3.3. Results

General

The cases encompassed 67 male and 4 female victims. Of these, 62% (n=44) of victims were located within a structure or vehicle, 36.6% (n=26) were outside and exposed to the elements. The location of the victim was undocumented in 1.4% (n=1) of cases. The post-firing interval was unknown or undocumented in 40.8% of cases (n=29).

POST FIRING INTERVAL	FREQUENCY	%
Unknown	29	40.8
≤3 hours	19	26.8
3-6 hours	7	9.9
6-12 hours	10	14.1
>12 hours	6	8.5
Total	71	100.0

Table	19 -	Post	firing	interval	s bv	case
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The site of the entrance wound was most frequently the head (83.1% of cases), the chest in 8.5% and both sites in 2.8%. The entrance wound location was undocumented in 5.6% of cases.

Weapons and Calibre

As seen in Figure 10, rimfire rifles were the most frequently encountered weapon type, involved in over 50% of all cases. Shotguns were the next most common at nearly 17%, and revolvers accounted or approximately 13% of cases.



Figure 10 - Percentage of cases by weapon type - Rimfire (RF) (n=36) and Centrefire (CF) (n=6) rifles, Shotgun (n=12), Semi-automatic (SA) pistols (n=7), Revolvers (n=9) (70 cases). Note – One case for which firearm type was not recorded has been excluded.

The most frequently observed calibre of ammunition was 0.22, which was encountered in 39 of the 71 cases. Of these cases, 35 involved 0.22 rimfire rifle ammunition, two cases involved 0.22 centrefire rifle ammunition, and two cases involved 0.22 rimfire pistols.

WEAPONS AND CALIBRE							
RIFLE							
FREQUENCY %							
0.22	37	52.9					
.222	2	2.9					
.223	1	1.4					
.308	1	1.4					
.357	1	1.4					
Total Rifle	42	60.0					
	HANDGUN						
0.22	2	2.9					
.32	3	4.3					
9mm	3	4.3					
.357	5	7.1					
.38	2	2.9					
.40	1	1.4					
Total Handgun	16	22.9					
	SHOTGUN						
12 ga	10	14.3					
.410	2	2.9					
Total Shotgun	12	17.2					
ΤΟΤΑΙ	70*	100.0					

Table 20 - Weapon type and calibre frequency data

* One case, in which the firearm type and calibre was unknown has been excluded.

Given the frequency of 0.22 rifles used, further division based upon the action of the firearm was possible and results can be seen in Figure 11.



Figure 11 - Cases involving 0.22 rimfire rifles ordered by action type - Bolt action (n=18), Lever action (n=3), Pump action (n=1) and unknown (n=13) (35 cases) Note -Two cases using 0.22 ammunition were centrefire rifles and have been excluded)

Assessment of these data indicates that of the rifles that fired 0.22 rimfire ammunition, bolt action is the most prevalent, observed in over half of all cases involving rimfire rifles. No semiautomatic rifles were recorded; however in a relatively large number of rifle cases (37%) the action was not noted.

Gunshot Residue

Exclusions

Further interrogation of the data of all gunshot suicides identified a number of cases that had the potential to introduce bias to the analysis. These cases were removed prior to further consideration of the data. Cases of this type included those in which case notes indicated:

- Significant medical intervention or lifesaving efforts had occurred prior to GSR sampling.
- Transport, disturbance or significant movement of the victim had occurred prior to sampling.

- Victim was known to be exposed to the elements (particularly rain) for an extended period.
- Firing of the weapon occurred under atypical circumstances that were highly unlikely to be reflective of usual firing.

There were 12 cases that met one or more of these criteria, leaving 59 cases under consideration.

Characteristic particles

When classifying GSR particles for this work, the American Society for Testing and Materials (ASTM) standard [153] terminology of 'characteristic' of firearms origin (three-component lead, barium, antimony particles) was applied. Two-component particles are termed ''consistent' with firearms origin, while single element lead, barium or antimony particles are referred to as 'consistent' with a firearms origin only when they are found in the presence of two-component particles.

When reporting characteristic particle counts, the former UK Forensic Science Service [165] defines four categories of positive characteristic particle result - 1-3 particles (low), 4-12 particles (moderate), 13-50 particles (high) and greater than 50 particles (very high). These categories have been adopted for this study.

In this study, most GSR sampling kits used consisted of four sample stubs, with one stub intended to be used on the front and one on the back of each hand (i.e., left hand front, right hand front, left hand back, right hand back). While this is most often the case, in some situations, bloodstaining or other soiling of the hands prevented all stubs from being collected or analysed. After the time period reviewed in this study, two stub kits, with one stub for each hand have become the standard in our jurisdiction.

The data considered for this study involves 195 sample stubs over the 59 cases.



Figure 12 - Characteristic particles detected by percentage of cases (n=59)



Figure 13 - Characteristic particles detected by percentage of stubs (n=195)

Characteristic particles detected per case can be seen in Figure 13, while particles detected per stub can be seen in Figure 12. When assessing the prevalence of characteristic particles on the hands of victims of unambiguous suicide where characteristic particles might reasonably be expected (i.e., cases involving three-component primed centrefire ammunition), GSR sampling of the hands returned a 'nil detected' result in 10% of cases and a 'low' result (1-3 particles) in a further 20.7% of cases. In these cases exhibiting low or nil particle recovery, there was no observed correlation between the time elapsed between deaths and sampling

and the number of particles recovered. When considering sample stubs, 33% of stubs returned no characteristic particles, and a further 32% returned only 1-3 characteristic particles.

As expected, the cases (approx. 85%) in which no characteristic particles were detected were primarily instances in which 0.22 rimfire ammunition and a rifle were used, but not exclusively. Given that most 0.22 rimfire ammunition contains primers that do not contain antimony sulphide [170], but may contain antimony as a component of the projectile, particles consistent with firearms origin (i.e., those containing Pb/Ba, Pb/Sb, Sb/Sn, Pb/Ba/Si, Ba/Al, Sb/Sn/Pb, Sn/Ba/Pb, Ba/Si/Ca) should be the ones most frequently encountered in shootings involving this type of ammunition. Considering just those suicides that involved 0.22 calibre ammunition and counting characteristic particles were detected in 64% of cases where characteristic particles should not arise from primer (i.e., cases involving 0.22 rimfire ammunition). Surprisingly, in 3 cases (5%) no particles of any type were detected. With respect to all the cases examined and including all types of ammunition, 5% of cases and 9% of cases involved detection of nil or few GSR particles, respectively (Figure 15). The cases and particle findings in relation to 0.22 rimfire ammunition are discussed further in a separate section below.



Figure 14 - Distribution of characteristic and consistent (excluding single-component) particle numbers by case in those suicides involving 0.22 calibre rimfire ammunition (n=28)



Figure 15 - Percentage of cases containing either characteristic (Pb/Ba/Sb) particles and/or consistent (excluding single-component) particle types (n=59)

Post-Firing Interval and GSR Recovery

When particle recovery relative to the time since firing was considered, the greatest average recovery of characteristic particles is observed when the sample was collected promptly after firing, as displayed in Figure 16. Across all case files, the post-firing interval was undocumented in 23 of the 59 cases.



Figure 16 - Average number of characteristic (Pb/Sb/Ba) GSR particles detected by post-firing interval *Cases for which post-firing interval was undocumented (n=23) were excluded.

Two critical factors in assessing retention and persistence of GSR in living subjects are the amount of residue that is initially deposited on the subject, and the subsequent loss of particles caused by physical disturbance and loss of particles caused by activity. As a consequence, it is suggested that samples should be collected from the shooter within 2-4 hours of firing [5]. In suicide cases however, the victims are known not to have undertaken any activity in the interval between firing the fatal shot and sampling, therefore any correlation between particle numbers and time since death must be due to other phenomena. If activity of the subject is excluded as the contributing factor, apparent decrease in GSR particle recovery versus time in suicides must be due to another phenomenon, assuming that GSR was deposited on the victim as a consequence of firing. Three alternatives were identified. Firstly, that the effects of time-dependent physical changes to the body post-mortem (e.g., cooling, drying, and suspension of circulation) reduces the collection capability of the sampling stub, or reduces the availability of particles. Secondly, that environmental exposure (e.g., exposure to wind, rain, etc.) reduces the number of particles present on the deceased. This will be further investigated in future research. Finally, post-mortem changes to the body might reduce efficiency of the sample collection, and that may be responsible for low particle recovery. Whatever the mechanism, the prevalence of negative GSR results in cases of suicide provides further support for the understanding that negative GSR results cannot reliably be taken as an indication of no association with firearms.



Figure 17 - Average number of GSR particles (characteristic and consistent, excluding singlecomponent) detected by firing interval.

*Cases for which post-firing interval was undocumented (n=23) were excluded. One case which exhibited excessively high numbers of consistent particles in the 12+ hour category was an outlier and was removed.

To assess the effect that environmental exposure may have had on particle recovery, average particle recoveries were assessed against time since firing and victim location as displayed in Figure 18.



Figure 18 - Average number of characteristic (Pb/Sb/Ba) particles detected by post-firing interval and victim location.

*Cases in which post-firing interval or location were undocumented were excluded (n=23)



Figure 19 - Average number of GSR particles (characteristic and consistent, excluding singlecomponent) detected by post-firing interval and victim location *Cases for which the post-firing interval or location were undocumented were excluded.

One case, which exhibited excessively high consistent particle (8694 Sb/Pb) counts in the 12+ hour, outside category was an outlier and removed.

When all data independent of post-firing interval were considered, there was no significant observed difference between GSR particle recovery from a subject that was found inside or outside.

However, while average recovery from victims that were outside remained consistent over time, It was observed that average characteristic GSR particle recovery from victims that were discovered inside falls off rapidly beyond 3 hours since firing. While this variation might arise as a result of environmental differences, further examination of the data suggests that firearm type might also have an influence on the number of initially deposited particles and particles detected over time. A higher proportion of shotguns and revolvers tended to be used outside. In this instance, sample size does not allow for a higher resolution breakdown based on weapon type at this level.

A broader analysis of the effect of weapon type can be seen in the average particle count by firearm in Figure 20. It was observed that cases involving revolvers had a higher likelihood of producing a significant number of characteristic particles on the victim's hands. This is in support of existing knowledge regarding the distribution of GSR from revolvers, with a significant quantity of GSR escaping the weapon from the cylinder gap, which is closer to the hands of the shooter [83]. The long arms - shotguns and rifles - exhibit much lower average numbers of characteristic particles, with rimfire rifles generating the lowest overall average. It should be noted however that cartridge calibre also plays a role, and as 0.22 rimfire ammunition is among the smallest calibre ammunition, the amount of primer from which to generate GSR particles is also smaller.



Figure 20 - Average number of GSR particles (characteristic and consistent, excluding singlecomponent particles) detected by weapon type – Rimfire (RF) (n=28) and Centrefire (CF) (n=6) rifles, shotguns (n=11), semi-automatic (SA) pistols (n=6) and revolvers (n-8).

From Figure 20, it can be seen that the rimfire rifles appear to generate the lowest average characteristic particle counts, while centrefire revolvers generate the highest. This is in support of current knowledge that characteristic particles are less commonly observed in 0.22 calibre ammunitions due to the lack of antimony in the primer – this is covered in more detail later. When consistent particle counts are considered, for those firearms that can be expected to produce characteristic particles, shotguns and semi-automatic pistols produce the lowest overall particle recovery, while revolvers maintain position as the highest. Typically, GSR will only be released in the proximity of the hands of a long arm (i.e., rifle or shotgun) shooter during the ejection and re-chambering cycle, or through gaps in the firearm or firing mechanism. Given Australia's restriction of semi-automatic firearms, the bulk of the long arms involved in these cases were manual action, (bolt, lever, or pump), in which a round must be manually re-chambered by the shooter in each firing cycle. In cases of suicide usually only one round is fired, minimising the release of GSR from the ejection port or firing mechanism to be deposited on the hands. Due to the difficulty in positioning a long arm (such as a shotgun or rifle) to administer a self-inflicted gunshot wound, long arms used in suicide are often awkwardly held or positioned, such that the victim is holding the barrel, or manipulating the trigger by other means. This atypical positioning of the firearm and hands relative to the regular discharge of a firearm may explain the high rate of low or no characteristic particles observed. Further, it is possible that any GSR detected on the victim may originate from the barrel or other gaps in the mechanism that would not typically settle on the shooter during normal firing. In the cases considered in this study, the positioning of the firearm relative to the victim was not clear from the case notes.

Characteristic particles from Sb free primers in 0.22 rimfire ammunitions

With 0.22 rimfire ammunition being used in over 50% of the suicide cases examined, and given its prevalence in crime in Australia, further analysis of residues from this ammunition type were examined in detail. In cases involving 0.22 rimfire ammunition, the detection of characteristic particles originating from this ammunition is unexpected, due to the fact that most of this ammunition contains primers that do not contain antimony sulphide [170]. However, the projectile itself is unjacketed lead hardened with antimony, which may then be incorporated into GSR particles [171]. From this data set, of the cases involving only rimfire rifles at least one characteristic particle was detected in 64% of the cases.

Though the ammunition type under consideration was undocumented in many cases (n=12), Winchester variants featured prominently (n=15), with Winchester Super X as the most

prevalent (n=7). Winchester ammunitions are known to use antimony-free primers [17]. A single instance involving Remington ammunition was observed. Interestingly, this case involving Remington ammunition involved a single shot from a bolt action rifle and resulted in the detection of no characteristic particles. However Pb/Ba, Pb/Sb, and Sb particles were detected, indicating the presence of all three elements in the sample. Remington ammunitions have been documented as using a three-component primer [171]. However, there are also some Remington variants, specifically Remington UMC variants that contain a lead-only primer that would similarly result in the detection of no characteristic particles and no particles found, the identity of the ammunition variant is undocumented, making positive identification impossible.

Given the small proportion of 0.22 rimfire ammunitions that contain antimony as a component of their primer, the relatively frequent observation of characteristic particles from ammunition of this type could be either attributable to incorporation of antimony from particles generated from the surface of the projectile during firing, or from contamination of the barrel due to previous firings of ammunition that does contain antimony. This is known as the weapon memory effect, and has been found to be a contributing factor in the generation of GSR that incorporates atypical elements [22, 172]. As there is no way of verifying the history of the weapons used in the cases considered, tracing the origin of the characteristic particles observed is not possible in this instance. However, given the relatively low prevalence of Sbcontaining primers in the 0.22 calibre ammunition population, significant contribution due to the weapon-memory effect can be expected to be unlikely.

Contribution of antimony from the surface of the projectile has been investigated previously by Zeichner [17], who concluded that the probability of a considerable amount of antimony in GSR from ammunition containing antimony-free primers was relatively low. The higher proportion of characteristic particles generated from 0.22 rimfire ammunition in our study indicates that either the probability of antimony from the projectile being incorporated into a three component particle is higher than estimated by Zeichner, or the population of victims involved in this study exhibited a bias towards ammunitions using antimony-containing primers despite the rarity of this ammunition in the Australian market, which is not likely.

In order to test the former proposition, muzzle discharge from a rifle firing a single shot of ammunition known to use antimony-free primers was collected and analysed for GSR. GSR collected in this fashion was found to contain a significant proportion of three-component particles with a significant concentration of antimony per particle, as well as a number of particles classified as Sb only. A sample collected in this fashion returned 150 three-component particles in less than 10% of the overall stub area. Examples of these three-component particles were elementally mapped using a FEI F50 SEM EDS system to verify the distribution of the elements within the particle – that is, to ascertain that the three-component particles found were indistinguishable from GSR generated from antimony-containing primers.



Figure 21 - EDS spectrum of three-component particle from muzzle discharge using PMC Zapper ammunition



Figure 22 - Secondary electron image and elemental map of three-component particles found in muzzle discharge using PMC Zapper ammunition



Figure 23 - Separated elemental maps of three-component particle isolated from muzzle discharge using PMC Zapper ammunition. (Yellow = Antimony, Green = Barium, Red = Lead)

Our findings, both from the test firings and the survey, suggest that three-component particles can be encountered relatively frequently, which is quite different to Zeichner's conclusion that Sb-containing particles are encountered only rarely in residues from antimony-free ammunition. Zeichner et al collected scrapings from the breech and muzzle of the firearm after firing, while our samples were derived from muzzle discharge. This suggests that muzzle discharge is more likely to incorporate particles from the bullet surface than those retained on the firearm and that characteristic particles formed from known Sb-free primers are likely to be more prevalent in muzzle discharge. As a consequence, our findings are of relevance in the examination of suicides and deposits on shooting victims and perhaps the findings of Zeichner et al are only applicable to particles retained by a firearm.

Particles consistent with firearms origin

The most frequently occurring two-component particle consistent with GSR was Pb/Sb and these particles were also found in the greatest abundance; Ba/Pb were the second most prevalent particle detected. This observation may be due to the fact that the bulk of cases under consideration involved the use of unjacketed 0.22 calibre long rifle ammunition, where antimony is often used to increase the hardness of lead [1] as discussed above. As antimony-free primers are likely in the majority of cases, Ba/Pb or Pb/Ba/Si particles are consistent with this source.

Controlizion	FREQ.	MAX.	MEAN.
COMPOSITION	(NO. CASES)	(NO. PARTICLES)	(NO. PARTICLES)
Sb/Ba	25	449	15
Sb/Pb	53	8694	297
Ba/Pb	41	1306	59
Sb/Sn	6	49	2
Pb/Ba/Si	27	2210	47
Ba/Al	3	14	1
Sb/Sn/Pb	24	36	3
Sn/Ba/Pb	1	1	0
Ba/Si/Ca	3	458	11
Pb	57	21796	1195
Sb	47	236	13
Ва	8	67	1
Total Consistent	295	35316	1644

Table 21 - Consistent particle type data

As shown in Figure 24 despite the low likelihood of antimony being present in the primer, particles containing antimony were detected in the bulk of the cases considered, with 86% of cases recording lead/antimony particles. Interestingly, particles classified as only containing antimony were observed in 82% of cases; in the majority of these cases the Sb can only have arisen from the projectile. As described above, the generation of Pb/Sb and Sb-only particles from ammunitions known to use a lead only primer is possible. The mechanism by which antimony-only particles might be produced from an alloy of lead that contains only a few percent of antimony is not obvious however.





When addressing single element, consistent particles containing antimony, it should be noted that particles identified as such have been categorised by particle analysis software based on a short (2 sec) initial particle scan. In practice, when assessing characteristic or consistent particles for casework, the initial classification is verified by the analyst relocating the particle in question and conducting a longer live-time analysis. In this instance, what are classified as single-element antimony particles may in fact be misclassified Pb/Sb or Sb/Ba particles. Further, there is no verification that the elemental assignments of single-component particles are correct. The calcium K α line (3.690keV) and antimony L α line (3.604keV) are close enough that some overlap may occur resulting in misidentification and poor categorisation of the element peak. Similarly, the fact that these particles were not verified means a non-firearm, environmental source of antimony cannot be ruled out –fabrics and other consumer products may contain antimony and therefore the potential to result in false positives for GSR [173]. For this reason, the individual particles are considered in the context of the overall particle population as a means of reducing the chances of false positives.

3.3.4. Conclusions

The main aim of this study was to examine the GSR particle populations on a significant number of individuals who had recently discharged firearms covering a wide range of types, in particular, shotguns and 0.22 calibre weapons. Whilst it was expected at the outset that studying suicides would be a simple model for GSR transfer dynamics in general, it also produced some unexpected findings in regards to GSR persistence under circumstances where activity of the shooter would not diminish particle numbers detected. Therefore, in addition to contributing data that assists in modelling particle transfer and persistence in criminal shootings in general, this study provided some new, specific findings in regards to evaluation of suicide GSR evidence.

It was expected that victims of suicide by firearm would exhibit high GSR particle counts, an abundance of characteristic particles when ammunition with Pb, Ba, Sb-containing primer was used, and moreover, that particle counts on suicide victims would represent a maximum that would be applicable to shooters involved in firearms crime. The combination of the barrel being directed at the shooter, the use of a single shot, lack of action after firing and persistence of the shooter at the scene all should maximise deposited GSR and minimise GSR loss. This study however, indicates that in cases where the victim had unequivocally used ammunition with Pb, Ba, Sb-containing primer a significant number of cases (9% in cases involving shotguns and 10% in cases involving other centre-fire firearms) resulted in the detection of very few (less than 3) or no characteristic GSR particles. Furthermore, in these cases many stubs (47.4% in cases involving shotguns and 33% in cases involving other firearms) without characteristic particles were observed. These findings suggest that caution should be exercised when evaluating cases in which characteristic particles are expected but not found. Consideration of consistent particles and a case-by-case assessment are required in order for GSR results in suicides to be adequately evaluated. It further indicates that under certain circumstances hand stubs alone cannot be reliably used to ascertain if a particular case is a suicide, murder or accidental discharge, which is a finding that supports existing knowledge [174].

The particle types detected are influenced to a certain extent by the formulation of the primer used in the shooting, but not absolutely. While three-component primers are common, 0.22 rimfire ammunition frequently does not contain antimony in the primer, and some primer formulations containing only lead (Remington UMC) [1] reduces the availability of other key elements to be incorporated into particle types. In cases specifically involving 0.22 rimfire ammunition, characteristic particles were unexpected but were observed in 64.3% of cases, with an average number of 13 particles found. It was also found that in 85.6% of cases involving 0.22 rimfire ammunition particles of Sb/Pb composition were detected, likely originating from the projectile. A test firing revealed that three-component, 'characteristic' particles may be generated in significant numbers in muzzle discharge from 0.22 rimfire ammunitions with antimony-free primers, which explains the observation of characteristic particles in suicides and has implications in regards to the examination of residues on victims of shooting. Additionally, particles classified as containing only antimony were observed in 82% of cases; the test firing confirmed that these types of particle are produced by ammunition with antimony-free primer. However, as these were not reacquired and confirmed, some may have been misclassified two-component particles, or particles containing elements with an antimony line overlap.

Given the lack of activity after firing in cases of suicide, it was expected that persistence of GSR on the victim would be relatively stable over time. Our data suggest differently, with maximum mean recovery of particles being observed in cases where the post firing interval was less than 3 hours and fewer particles detected after longer time intervals, independent of body location and firearm type. This finding supports the accepted knowledge that absence of GSR particles on a suspect cannot be used as definitive proof that a firearm was not discharged.

General data collected from cases of suicide support the understanding that rifles and shotguns are amongst the most prevalent firearms used in suicide in South Australia with 0.22 rimfire ammunition being the most commonly encountered ammunition type. No semi-automatic rifles were observed, and the most prevalent action type was bolt action (52.8% of cases).

In regards to limitations of this study, it is recognised that data such as calibre, firearm type and post-firing interval were based on initial submissions by the sampling officer. As such, there was no ability to verify or validate this information. This is particularly important in the case of post-firing intervals, which were often recorded as a range reflecting an estimate by the first officer on scene, rather than through verified time of death. Further, reliance upon investigator information resulted in most data being incomplete in some fashion, with a high proportion of information unknown or undocumented, making full examination of all cases impossible.

----- End of Publication-----

3.4. FURTHER PUBLICATIONS

The manuscript originating from this work attracted a level of attention once it was published, with one commentator producing a letter to the editor of Forensic Science International to offer a critique our conclusions [175]. A response, also published in Forensic Science International [164], allowed for the provision of additional context behind our conclusions, and the position on the issue to be clarified.

3.4.1. Publication - Author's Response - Letter to the Editor (FSI-D-16-00737)

We appreciate the opinions of the commentator in regards to what might be an important paradox in regards to the composition of inorganic gunshot residue (IGSR) particles, which we mentioned in our recent article.

The broad purpose of our article was to investigate the deposition of GSR in situations where movement of the shooter post-firing was minimal. Suicide cases were used as a means to investigate this. Within this broader purpose, we noticed some puzzling results that we felt obliged to report. In short, we hoped that readers would find the following informative in regards to suicides (and possibly shootings in general):

A significant fraction of suicides involving a firearm that we investigated did not result in the detection of IGSR on the deceased;

In a significant fraction of cases involving 0.22 calibre ammunition where we knew that the ammunition involved not contain Sb in the primer (and a number of other cases where we suspect Sb was not present in the primer) 3-component, characteristic particles (i.e., Sb, Pb, and Ba-containing particles) were found.

We think that the author of the letter to the editor may have not appreciated the point we are trying to make. We did not set out to refute the weapon memory effect, but given the brands of 0.22 calibre ammunition used in the suicides and the ammunition preferences in South Australia, we did suggest that the weapon-memory effect might not be the major factor behind the presence of antimony-containing particles we observed in the suicide survey.

In regards to the rimfire ammunition used in the suicides we investigated, 15 out of 29 cases involved Winchester brand ammunition (known to not contain antimony in the primer).

Therefore, in these cases 3-component particles were not expected. However, in the cases studied, 8 of the 15 cases involving Winchester ammunitions returned at least one 3-component, characteristic particle. An average of 8 characteristic, 3-component particles were observed, and the maximum number of such particles observed was 93. It was felt that this finding was significant enough to warrant comment and further investigation.

In South Australia, the majority of the 0.22 ammunition market is held by Winchester, followed by CCI (ammunition that also does not use antimony-containing primer) therefore we believe that it is reasonable to conclude that the ammunition used in the cases where the ammunition is unknown is also most likely to involve ammunition in which the primers were antimony-free. We wish to acknowledge that we do not have hard quantitative data for market shares, that premise is based upon advice from local police and an ammunition vendor. Due to the market share held by Winchester and CCI, if a different ammunition was used in the firearm prior to its involvement in the suicides, then it is also more likely that the ammunition used previously also had antimony-free primers. In order to add to these findings, we presented results from a single controlled test firing. We were cognizant of the fact that we had only carried out one controlled test, and we understood that we cannot rule out that possibility that the suicide victims had fired different ammunition before taking their life; therefore we expressed our conjectures with what we believe to be an appropriate level of caution under the circumstances. Nowhere did we dogmatically rule out the involvement of the weapon-memory effect, instead we sought to offer some explanation and in the sentence "As a consequence, our findings are of relevance in the examination of suicides and deposits on shooting victims and perhaps the findings of Zeichner et al. are only applicable to particles retained by a firearm." the word "perhaps" is salient.

In response to the commentator's questions we can provide some additional information that might be helpful to them and other readers.

All of the reported suicide cases were investigated by a laboratory accredited under ISO-IEC17025 in accordance with ASTM guidelines. Per these guidelines, automatic software classification of particles was manually verified by reacquiring the particles before reporting. While for the test firing sample not all 0.22cal GSR particles containing Sb were analysed manually (there were too many), many were, and the presence of Sb was confirmed. The automated search printout was not used as the sole criterion for identification of elements and classification of particles. In regards to collection of GSR from the test firing, the firearm that was used (Ruger 10/22) was stripped and cleaned as described, and then 20 rounds of Sb-free (in regards to both primer and projectile) ammunition were fired through the barrel. A test round of the same ammunition was then fired into a catcher, which was sampled for GSR. Particles collected did not contain antimony. This constituted the control – it gave the expected result from the ammunition used. A sample was collected from the interior of the catcher as a blank, to verify that the catcher was free of any significant contamination. Then, a single round of PMC Zapper ammunition was fired and muzzle discharge was collected and sampled.

After an additional 20 rounds of PMC Zapper ammunition had been fired through the firearm the region around the ejection port of the firearm was sampled, and the following particles were found: 6x PbBa; 21x PbSb; 1x BaSb; 1x Sb only; and 120x Pb only. No 3-component PbSbBa particles were detected. This is in significant contrast to the particle population collected from the muzzle discharge, where 150 PbBaSb-containing particles were found in approximately 10% of the stub surface (the search automatically terminated at 10% due to the number of particles detected).

The correspondent concludes by writing "In summary, the impression that one may get from the study, is that only because of the contribution of antimony from a projectile and not taking into account the possible contribution of the memory effect, there will be no significant difference in the antimony content of the resulted IGSR population between discharge of .22 caliber rimfire ammunition containing antimony in the primer and the ammunition having free-antimony primer". We have raised this as a possibility, and therefore suggested a caution in regards to evaluation of IGSR analytical findings, and it would appear that further research is warranted. The overall suggestion that we wished to express to our readers was that (as indicated in the conclusion to the article) "The particle types detected are influenced to a certain extent by the formulation of the primer used in the shooting, but not absolutely."

-----End of Publication-----

3.5. FURTHER CASE SURVEY

3.5.1. Background

For a more complete picture of the types of firearms involved in firearms crime, a review of all cases in which a GSR analysis was performed were considered. Specific consideration was given to the same general categories as in the previous survey of suicide cases. Specifically, data such as firearm type, firearm action, ammunition type and manufacturer, presence or absence of GSR, and GSR composition were considered. Due to the complexity of the case information, and the diverse sample types that were collected, a meaningful assessment of the post-firing interval as it pertains to GSR findings could not be performed. Unlike the previous review of suicide cases, in this survey, no cases were excluded, and therefore this data set includes any samples that were collected and analysed, regardless of their source. To that end, the sample set includes samples collected from spent ammunition cartridges and from surfaces of firearms, which are virtually assured to have characteristic particles present. Similarly, the sample set also includes a number of samples collected from persons, vehicles, clothing, and other surfaces collected under diverse circumstances and conditions. As a result, there is no clear pattern to the case circumstances, including the proximity of the individual or surface to a firearm discharge, the time since discharge, or other case context relevant to a GSR assessment. For these reasons, the scope of the assessment of GSR was limited in two ways. First, the absolute particle counts present on each sample were not considered. Without the appropriate context regarding the circumstances surrounding the collection of the sample, suspected time since discharge, activity of the source, among other things, it was concluded that little meaningful information could be extracted from these data. Secondly, the total particle population present on the stub was not considered, as devoid of context, a meaningful assessment of the overall particle population could not be performed. Rather, the focus of this data collection was on the presence of characteristic GSR, the relative abundance of the three characteristic elements, and the presence of other elements observed in the particle. In situations where the case circumstances suggested that it was warranted, namely the use of 0.22LR rimfire ammunitions, if present and reported, consistent PbBaSi, and PbSbSi particles were also noted.

These collected data further informs the types of firearms and ammunitions, as well as the particle compositions that they generate, that are commonly encountered in forensic GSR casework in South Australia. This case review and data collection formed the foundation of a

GSR case database, which could be built upon to assess observations and trends as it pertains to the GSR assessments performed by FSSA.

3.5.2. Materials and Methods

Case Selection

A further sample of cases in South Australia in which a firearm was involved, covering the period between 2007 and 2016 were reviewed and interrogated for their GSR test results. Cases that had been considered in the previous survey were excluded, however cases of suicide occurring between 2014 and 2016, and cases that had been excluded in the previous survey were included in this data. As before, appropriate approvals were gained prior to interrogation of the data, and all identifying details were redacted to preserve the anonymity of the individuals involved. The samples considered in these cases had been collected from a variety of surfaces, as deemed pertinent to the case under consideration, but included the hands of persons, clothing, vehicles, firearms, impact points, cartridges, and other surfaces of interest. The information collected from the case files included the firearm type, calibre, action, cartridge headstamp, ammunition brand or manufacturer, and any other pertinent notes. As with the previous survey, this information was not available in all cases, as the data collected was from the analysis request forms completed by the investigating officer.

A total of 295 cases, encompassing 455 separate GSR samples were identified and considered in this study.

Equipment

As previously reported, all samples were analysed by SEM-EDS at FSSA in accordance with operational procedure. Instrument operating parameters were established as per the American Society of Testing and Materials (ASTM) E1588-16 Standard guide for gunshot residue analysis by SEM/EDS [153]. All cases covered in the period of this survey were analysed using a Zeiss Evo 50 SEM with Oxford EDX system and INCA Aztec particle analysis software. The automated particle search system brightness and contrast settings were calibrated through use of a Gold/Cobalt/Rhodium (Au/Co/Rh) standard.

A positive control for the Zeiss system, a synthetic particle standard (PLANO W. Plannet GmbH, Wetzlar, Germany, SPS-A521-2(27C)), consisting of accurately deposited particles of known size was analysed at the start and end of every sample run. The "particles" present on the standard are thin PbSbBa films of sizes $1 - 5 \mu m$.

Data Analysis

Where it was recorded, demographic data pertaining to firearm type, calibre, and action was collated. Similarly, when it was available, the specific ammunition manufacturer and headstamp information was also noted. This information was not uniformly available for all cases.

With regard to the specific GSR results, classification of particles present on the sample stubs was performed and reviewed in accordance with the contemporaneous ASTM guidelines at the time the analysis was performed. To that end, the automatic classification had been reviewed by an experienced GSR examiner, and the particles had been reacquired and had their composition verified. The characteristic GSR considered as a part of this review was that which had been recorded and reported in the finalised case file.

The core focus of this survey was on the composition of characteristic GSR observed. To that end, the absolute count of particles present on the sample stub was not recorded, rather the number of different particle compositions present on the sample, and the elements of that composition were recorded. This included the relative abundance of each of the three characteristic elements (Pb, Sb, Ba), along with the presence and relative level of other elements present in the particles. Levels of all elements were recorded and reported as either a major component (>30% of the strongest peak), minor component (between 10% and 30%), or trace (<10%), as previously described by Wallace and McQuillian [49].

3.5.3. Results

Firearms and calibre

The type of firearm involved was recorded in approximately 73% of cases. A complete breakdown of the firearm types reported can be seen in Table 23.

FIREARM TYPE	N	%
Rifle	99	33.6
Shotgun	70	23.7
Revolver	13	4.4
Pistol	32	10.8
Other	2	0.7
Not Recorded/Not applicable	79	26.8
TOTAL	295	100.0

Table 22 – Number and percentage of firearm types used in GSR cases in South Australia betweer
2007 and 2016

From Table 23, it can be seen that the most frequently observed firearm type across all cases in this period was rifles, which featured in 99 (34%) of the total cases. Distinct from the previous survey, in this instance cases in which the firearm was not recorded represented nearly 27% of the total cases considered. With this in mind, frequencies were considered as a percentage of the number of cases in which firearms were recorded. Rifles were observed in 45.8% of the cases where the firearm was recorded. Shotguns were the next most represented observed in 70 (32.4%) of cases, and revolvers and pistols representing a combined 45 (20.8%) of cases. The relative prevalence of these firearms are consistent with both the previous review of cases of suicide and research on the use of firearms in the Australian population by both the general public and serious and organised crime gangs (SOCG) [162]. These observations support the view that the use of firearms in situations that result in investigations involving GSR analysis correlates with the general availability of firearms in the wider population. It should be noted that in two cases (<1%), a firearm recorded as 'other' was noted. In one case, this represented a 12g 'Powerhead', (an underwater firearm used in shark fishing) was used. The other represented a home-made firearm or pyrotechnic device that was not otherwise categorised. Overall, the type of firearm involved was not recorded or deemed not applicable in approximately 27% of the cases considered in this survey.

Given the observed prevalence of rifles in this study, as before, a more detailed breakdown of the rifle data was considered, categorising them by action type. A presentation of these data can be seen in Figure 25. From the collected information, it was noted that of the entire rifle population, approximately 35% of rifles used rimfire ammunition while 13% used centrefire ammunition. The remaining 52% of the population did not have this information recorded.





From the data in Figure 25, it can be seen that the most frequently observed situation were cases in which the specific action of the firearm was not recorded. However, compared to the data in the previous study, the relative abundance of each action type, in situations where they were recorded, was comparable. In both instances, bolt action rifles were the most prevalent, followed by lever action. In the all cases survey, semi-automatic rifles were the next most frequently observed, while these were not encountered at all in the suicide study. In both instances, pump action rifles were the least frequently observed. Beyond the specific firearm type used, it is also instructive to consider the range of calibres encountered in casework in this period. A breakdown of the calibre type, separated by firearm can be seen in Table 24.

WEAPONS AND CALIBRE	N	%			
Rifle					
0.22 80 36.4					
22-250	1	0.5			
0.222	4	1.8			
0.223	4	1.8			
0.27	2	0.9			
0.303	2	0.9			
0.308	1	0.5			
0.44	1	0.5			
7.62	3	1.4			
30-30	2	0.9			
Total Rifle	100	45.6			
HANDGUN					
5.56	2	0.9			
0.22	13	5.9			
0.32	5	2.3			
9mm	14	6.4			
0.357	7	3.2			
0.38	5	2.3			
0.4	2	0.9			
0.44	1	0.5			
0.45	1	0.5			
Total Handgun	50	22.7			
Shotgi	UN				
12g	36	16.4			
0.410	7	3.2			
Not Recorded	27	12.3			
Total Shotgun	70	31.8			
TOTAL	220*	100.0			

Table 23 – Weapon types and calibres observed in GSR cases in South Australia between 2007 and2016

Number of recorded firearms totals more than the number of cases as a number of cases involved the use of more than one firearm.

From the data presented in Table 24, some clear trends are evident. Again, consistent with the data collected in the previous review of suicide cases, the most frequently observed firearm calibre was 0.22 rifles, with over 36% of the total cases reviewed involving a firearm of this type. Next most popular was 12 ga shotguns, which featured in 16.4% of cases. In the handgun category, 9mm (6.4%) and 0.22 (5.9%) handguns were observed with similar frequency, while in the suicide case review, 0.357 handguns (7.1%) were the most frequently encountered.

Independent of the calibre of firearm involved, in some instances, the specific ammunition product used was noted by the investigating officer. To inform the prevalence of different

ammunition product brands and manufacturers observed in the South Australian market, a breakdown of these products separated by firearm type can be seen in Table 25.

AMMUNITION MANUFACTURER	Ν	%				
RIFLE	RIFLE					
Winchester	26	11.9				
CCI	3	1.4				
Remington	2	0.9				
Sellier & Bellot	2	0.9				
Total Rifle Recorded	33	15.1				
Not Recorded	67	30.6				
Total Rifle	100	45.7				
Handgun						
Winchester	8	3.7				
Remington	1	0.5				
PMC	2	0.9				
ODL	3	1.4				
CBC	1	0.5				
Federal	2	0.9				
Luger	1	0.5				
Fiocchi	1	0.5				
Wolf	1	0.5				
Total Handgun Recorded	20	9.1				
Not recorded	29	13.2				
Total Handgun	49	22.4				
Shotgun						
Winchester	11	5.0				
Eley-Kynoch	1	0.5				
Remington	1	0.5				
Super 6	1	0.5				
Diana	1	0.5				
Kent	1	0.5				
Fiocchi	1	0.5				
Sellier & Bellot	1	0.5				
Federal	2	0.9				
RC1	1	0.5				
Total Shotgun Recorded	21	9.6				
Not Recorded	49	22.4				
Total Shotgun	70	32.0				
Total Ammunition Brand Recorded	74	33.8				
Total Unrecorded	145	66.2				
TOTAL	219	100.0				

Table 24 - Ammunition product manufacturer/brand by firearm type as observed in South Australiabetween 2007 and 2016

When considered independent of firearm type, the relative prevalence or popularity of different ammunition products in the South Australian consumer firearms market may be seen, as presented in Table 26.

	Ν	%
Winchester	45	60.8
Federal	4	5.4
Remington	4	5.4
CCI	3	4.1
Sellier & Bellot	3	4.1
ODL	3	4.1
PMC	2	2.7
Fiocchi	2	2.7
Luger	1	1.4
CBC	1	1.4
Wolf	1	1.4
Eley-Kynoch	1	1.4
Super 6	1	1.4
Diana	1	1.4
Kent	1	1.4
RC1	1	1.4
ΤΟΤΑΙ	74	100.0

Table 25 - Ammunition manufacturer prevalence across all firearm types as observed in GSR cases in
South Australia between 2007 and 2016

OVERALL AMMUNITION MANUFACTURER PREVALENCE

Reports of ammunition brand were based on the information recorded by the investigating officer, and therefore may not necessarily be wholly accurate. For example, in one instance, a shotgun ammunition was recorded as 'Champion' brand, which is actually a Federal product line. While the data in Table 26 indicates that Winchester ammunitions are the most frequently encountered in South Australian casework, a cautious interpretation must be applied. It may be the case that Winchester is better labelled, more distinctive, or more recognisable than other ammunitions, and therefore is more likely to be recorded. Likewise, it is possible that this ammunition is more appealing to consumers for other unquantified reasons, such as pricing or availability. It is similarly important to note that the ammunition brand was unrecorded in approximately 66% of cases. Regardless, these figures indicate that Winchester ammunition products are relatively frequently encountered in this jurisdiction.

Gunshot Residue

Of the 456 GSR samples collected and analysed across 295 cases, 338 (74%) were determined to have characteristic GSR particles present, with the remaining 118 (26%) having no characteristic GSR present. If considering the 329 samples for which a firearm was recorded, these results can be further separated based on firearm type, as seen in Table 27.

	CHARACTERISTIC GSR PRESENT		NO CHARACTERISTIC		
	Ν	%	n	%	
Rifles	103	73.0	38	27.0	
Shotguns	80	75.5	26	24.5	
Handguns	73	89.0	9	11.0	
TOTAL	256	77.8	73	22.2	

Table 26 - Separation of GSR results based on firearm type as observed in all cases involving GSRanalysis in South Australia between 2007 and 2016.

In total, the composition of 732 particles was recorded across all 456 samples. The division of particles observed and the type noted separated by firearm type can be seen in Table 28.

Table 27 - Particle composition types separated by firearm type as observed in all case samplesinvolving GSR analysis in South Australia between 2007 and 2016.

	ALL DATA		Rı	RIFLE		HANDGUN		SHOTGUN	
	Ν	%	Ν	%	Ν	%	Ν	%	
PbSbBa	165	22.5	23	10.4	59	39.9	39	24.8	
PbSbBaSi	353	48.2	93	41.9	67	45.3	94	59.9	
PbBaSi	209	28.6	103	46.4	22	14.9	23	14.6	
PbSbSi	5	0.7	3	1.4	0	0	1	0.6	
PERCENTAGE OF TOTAL	732	100.0	222	30.3	148	20.2	157	21.4	

Percentage column represents the percentage of particles in each firearm category. Firearm categories do not total 100% as the all data column captures cases for which a firearm type was not recorded.

As can be seen from Table 28, the most frequently observed particle composition across all of the particle types was PbSbBaSi, which represented nearly 50% of the total particles recorded. The contribution of Si to these particles in this case was not investigated in detail, but it may be attributable to the presence of gGSR, where glass frictionator from the primer compound is incorporated into GSR particles [159, 160] This general trend persists for the individual firearm categories, with this particle type being the most frequently observed in both the handgun and shotgun categories. A notable exception is in the case of particles originating from rifles, in which the largest particle type category was PbBaSi particles. This is likely due to the contribution of 0.22LR rimfire ammunitions which lack Sb in the primer compound. To further assess this, the particle composition data from all cases involving rifles was further separated based on the ammunition type used. This comparison can be seen in Table 29.

	CENT	REFIRE	Rim	IFIRE	NOT RECORDED		
	Ν	%	Ν	%	Ν	%	
PbSbBa	4	21.1	4	4.6	15	12.9	
PbSbBaSi	11	57.9	29	33.3	53	45.7	
PbBaSi	1	5.3	54	62.1	48	41.4	
PbSbSi	3	15.8	0	0.0	0	0.0	
Sub-Total	19	100.0	87	100	116	100	
PERCENTAGE OF ALL RIFLE PARTICLES		8.6		39.2		52.3	

 Table 28 – Particle composition types observed in all case samples using rifles, separated by ammunition type.

Percentage of all rifle cases totals to 100.1% due to rounding.

From Table 29, it can be seen that for most particles, the ammunition type they originated from was not recorded in a majority of cases. For consistency, data was only recorded in either the rimfire or centrefire category if the ammunition type was clearly recorded. Based on this information, it can be seen that centrefire ammunition was only observed in approximately 8.6% of cases, while rimfire ammunitions were much more frequently observed, present in almost 40% of cases. Most evident from the data in Table 29 are the differences particle composition types observed between centrefire and rimfire ammunitions. In the case of centrefire ammunitions, particle types containing Sb represent nearly 80% of the total particles observed originating from this ammunition type. By comparison, in the case of rimfire ammunitions, over 62% of the particles that were observed did not contain Sb. Conversely, nearly 38% of the particles were observed to contain Sb. This series of findings is consistent with the observations in the previous review of suicide cases. This suggests that although in most instances GSR produced is compositionally consistent with the components of the primer and therefore lacks Sb, characteristic particles are still observed, either due to the weapon memory effect, or some other mechanism.

In considering additional elements that were frequently observed incorporated into particles of GSR, data was again separated based on reported firearm type. This can be seen in Table 30.

	Si		AI		Mg		Fe		Ni		Sn		Cu		Zn	
	N	%	N	%	N	%	N	%	N	%	N	%	N	%	N	%
Rifle	101	87	87	75	20	17	64	55	1	1	3	3	58	50	20	17
Shotgun	118	75	139	89	34	22	81	52	1	1	1	1	47	30	7	4
Handgun	89	60	94	64	9	17	66	45	3	2	8	5	77	52	33	22
All Data	564	77	523	71	133	18	358	49	12	2	31	4	338	46	117	16

 Table 29 - Additional elements most frequently incorporated into GSR particles separated by firearm type

Percentage column represents the percentage of particles in which the element was observed at any level in each firearm category. The n observed in each firearm category does not equal the all data total as the all data column captures cases for which a firearm type was not recorded.

Most frequently observed incorporated into GSR particles were Si and Al, which were each seen in over 70% of the particles noted, and were roughly evenly distributed at major, minor and trace levels. Next most common were Cu and Fe, which were observed incorporated into approximately half of the particle types observed. It should be noted however that in the majority of cases, these elements were only present at a trace level. The other incorporated elements were all observed in less than 20% of the total particle types. In considering trends observed on the basis of firearm type, both Cu and Zn were observed much less frequently in particle types originating from shotguns. This is most likely a result of elemental contributions from the cartridge brass in rifle and handgun ammunition. Cu was observed much more frequently than Zn, suggesting that cartridge brass is not the sole contributor, and that elements of the projectile jacketing material or wash may also be contributors. Particle types originating from cases involving shotguns also incorporated AI more frequently than other firearms. Si-containing particles were most frequently encountered in cases involving a rifle, and least frequently observed in cases involving handguns. The frequency with which Si containing particles were observed was of particular interest, given that one explanation for its presence may be the incorporation of glass frictionator into the particle matrix. Recent research has explored the phenomena and possible usefulness of glassy GSR (gGSR) to firearm investigations [52, 158-160]. It is worth remembering that the basis of this assessment is based around particle composition types rather than individual particles. To that end, for the purposes of these data if a GSR sample contained many particles with a similar compositional elements in similar proportions, it was only recorded once. Conversely, if a sample was observed to contain many particles with different components and with different proportions, an entry was recorded for each distinct particle type. For this reason, this data is most useful for assessing general trends, rather than the specifics of each case.

Atypical GSR Findings

From Table 28 and Table 29, it can be seen that a small number of PbSbSi particles with no Ba present were observed. The majority of these particles (3/5) were observed in a sample collected from a spent cartridge, which was determined to be a Sellier & Bellot 7.62 centrefire rifle ammunition. It was also observed that each of these particles had some level of Hg present, which suggests that the cartridge contained an antiquated, mercury fulminate based primer compound. Priming compositions of this type have long since been discontinued, due to the toxic and corrosive properties of Hg, and are unlikely to be frequently encountered. Of the remaining two PbSbSi particles, one was identified from a spent 9mm cartridge case, identified as ODL 'Greenpoint' training ammunition, and the final one was present on a sample collected from clothing, alongside other particles with characteristic composition. This suggests that particles of this composition are infrequently encountered, and seem to be more frequently observed in situations where an uncommon ammunition has been used.

One further case of note was a series of characteristic GSR particles originating from a 0.38 Revolver loaded with centrefire Winchester ammunition that were observed to contain Se at a moderate level. Other components included Mg at a high level, and Al and P at a moderate level. It was ultimately determined that the Se originated from selenous acid used in gun bluing solutions. This phenomenon has been observed Europe as well as Australia has been explored in detail by Romolo et al [21]. This finding speaks to the incorporation of elements present on the firearm into the resultant GSR particles.

3.5.4. Conclusions

This case review provided some broader data about the different types of firearms and ammunition that are used in crime in South Australia. All cases in which a GSR analysis was performed between 2007 and 2016 were interrogated for their GSR results. This data showed similar broad trends as the assessment of suicide cases, with the observed frequency of firearm types generally correlating with their availability under Australian firearms legislation. For this reason, rifles were the most frequently encountered, followed by shotguns, and then handguns. As it pertains ammunition types, the findings of this survey of cases suggests that 0.22LR rimfire rifle ammunitions are among the most frequently encountered ammunitions types. Further information was able to be collected relating to the brands and manufacturers of ammunition that are most commonly encountered in this jurisdiction. While the specific brand of ammunition used was unrecorded in the majority of cases, in situations where the manufacturer was noted, Winchester brand ammunitions were encountered in over 60% of cases. Federal and Remington were observed in just over 5% of cases, with a variety of other brands occurring more infrequently. However, it was unable to be determined on the basis of these data if this is due to Winchester brands being more recognisable and therefore more prone to being recorded, if they are more readily available, or if there are economic, functional, or other reasons that these ammunitions are preferred. Regardless, Winchester brand ammunitions were observed to be frequently encountered in casework.

Further, in a number of cases, particles from rimfire ammunitions were observed to contain Sb, which is frequently absent in the primer mix of 22LR rimfire ammunitions. This phenomenon was also seen in the suicide case data. These findings support the notion that characteristic particles may be encountered in situations where they are unexpected due to the primer composition of the ammunition. While these data suggest that this observation is not restricted to cases of suicide, it does not provide any further information as to the origin of these particles. It is possible that they are retained in the firearm due to the weapon memory effect, or that Sb is incorporated into these particles from the surface of the projectile.

A small number of particles with odd, or atypical compositions were observed. This included one case observing GSR particles containing minor levels of Hg, likely originating from an antiquated Hg-containing primer mixture. Further, one set of GSR particles were observed to contain minor levels of Se, which was determined to be a result of a gun-bluing solution containing selenous acid being used on the firearm. These findings further reinforce the importance of a structured, case-by-case approach in assessing GSR evidence.

3.6. CONCLUSIONS

A comparison of the data collected from a specific case survey of suicides, as well as a survey of all cases involving GSR analysis provided useful data pertaining to the use of firearms in South Australia. Across both surveys, it was evident that rifles were the most frequently encountered firearm type in all circumstances, with rimfire rifles being the most frequently observed variant. Shotguns were encountered in approximately a third of all GSR cases, but less than a fifth of suicides. The popularity of these firearm types is most likely due to their classification within Australian firearm legislation making them more readily available. Conversely, handguns were encountered relatively infrequently, and only featured in approximately 23% of both suicide cases and all other GSR cases, which is similarly a likely reflection of the difficulty in obtaining them and their general availability.

With regard to ammunition manufacturers, the results of both studies indicate that Winchester-made ammunitions feature prominently. While it is unclear if this is a function of the ammunition's cost, reliability, availability, or simply the fact that it is recognisable, the popularity of these ammunition types provides useful information in informing GSR analysis. This is particularly relevant as it has been well documented that many 0.22LR Winchester ammunitions make use of an Sb-free primer formulation [17]. Due to the popularity and prevalence of 0.22LR ammunitions with Sb-free primers, the presence of three-component, characteristic GSR particles was higher than would be anticipated in both sets of data. While it could not be ascertained as a part of these surveys if this was a result of the weapon memory effect, or of incorporation of Sb into these particles by other means, further investigation to identify the possible source would be valuable. This would improve the understanding of when particular particle compositions may be expected, and further inform the phenomena behind GSR particle formation.
4.SUB-PARTICLE COMPOSITION AND MORPHOLOGY OF GSR

4.1. SYNOPSIS

Research reported in the previous chapter indicated that the number of three-component particles generated from two-component primed ammunition was higher than would be expected given the prevalence of 0.22LR ammunitions without Sb in the priming compound. This finding was consistent across both a review of GSR results from suicide cases between 1998 and 2014, as well as a wider survey of all forensic GSR casework between 2007 and 2016. While it had been suggested that this finding was a result of the weapon memory effect [175], the relative scarcity of three-component primed 0.22LR rimfire ammunitions observed in surveys of this jurisdiction suggested that another mechanism may be responsible.

To that end, the goal of this study was twofold. First, to assess the internal structure and composition of GSR originating from different types of ammunition, including considering nontoxic primer formulations, to determine if there were differences that could be used as a means of identifying the ammunition that produced the particles, or differentiating particles generated by different ammunitions. In addition, it was hoped that further information about the source of Sb observed in three-component particles from two-component primed ammunitions could be obtained. Secondly, a Focussed Ion Beam (FIB) has previously been used as a means of preparing thin sections of sample which can then be analysed using alternative techniques. Techniques that can benefit from this form of sample preparation include Transmission Electron Microscopy (TEM), Auger Emission Spectroscopy (AES) and Time of Flight – Secondary Ion Mass Spectrometry (ToF-SIMS). Each of these techniques could then be applied to Sb-containing GSR samples in order to obtain additional detail about their composition. Specifically, application of these techniques could help identify the oxidation state of the Sb, and ascertain if it exists as metallic Sb, or as another compound, such as a sulphate, carbonate, or oxide form. This would then assist in ascertaining if any of these techniques could assist to distinguish elements of GSR that originate from the primer, and those that originate from the projectile, the weapon memory effect, or other components of the firearm.

Samples of GSR obtained from different ammunitions, including a selection of heavy metal free (HMF) and non-toxic primer formulations were first identified using SEM-EDS and a dedicated GSR analysis package. Particles identified by this method were then cross-sectioned using a FIB to reveal their internal structure. The cross-sectioned particles were then reacquired using SEM-EDS, and X-ray mapped to assess sub-particle composition. Despite numerous attempts, a suitable thin section of a sample to be used for further analysis by an alternative technique was not able to be prepared. The fragility of the particle matrix meant that, once sectioned, the prepared samples were fragile and vulnerable to damage or loss during transportation. That said, some types of GSR, most notably glassy GSR (gGSR) were more robust, and were able to be better withstand the extraction and transport process. This suggests that this technique may be more successfully applied to some particle types than others. This study forms a preliminary study that suggests that there is the potential that additional information may be obtained from GSR in some cases. All of the samples originating from different ammunitions exhibited some particles with distinct internal morphologies and compositions, which, with further research, could be used as a potential means of differentiating GSR from different ammunitions, or aiding in identifying the ammunition from which the GSR was generated.

Results of the FIB study provided further information pertaining to the mechanism of three component particles being generated in situations where they would be unexpected such as when two-component primed ammunitions have been used. The cross-section of a three-component particle obtained from 0.22 LR PMC Zapper two-component primed ammunition indicated that Sb present in the particle was co-located with other elements also found on the surface of the projectile, namely Pb and Cu. Firing studies also indicated that three component particles from this ammunition were more frequently observed in barrel discharge residues than they were in samples collected from barrel or breech scrapings. Taken together, this suggests that incorporation of Sb from the surface of the projectile is one mechanism by which three-component particles are generated from two-component primed ammunition. While this does not discount the weapon memory effect as a possible contributor to these particles present in the population, it does provide further indication of the complexities behind GSR particle formation.

Further exploration of the impact of the weapon memory effect was conducted through the use of two different ammunitions. Both ammunitions used a two-component primer that did not contain Sb, however one ammunition, CCI Stinger, had a Pb projectile while the other, PMC Zapper, had a hardened projectile containing both Pb and Sb. Both projectiles were coated in a Cu wash, and were contained within CuZn cartridge casings. Samples were collected of both the muzzle discharge, as well as scrapings of the barrel and breech. It was observed that particles containing Sb were at noticeably higher levels following discharge of PMC Zapper. However, Sb was not totally absent in the CCI Stinger sample, which suggests

that the weapon memory effect still makes some contribution to the overall particle population.

4.2. BACKGROUND

In the context of this thesis, this study was performed as a means of further investigating the findings collected in the review of suicide and other GSR cases, previously discussed in chapter 3. In the previous study, it was noted that despite the fact that a majority of 0.22LR calibre ammunitions do not typically include Sb as a component of the priming compound, there was a higher than expected number of three-component characteristic particles observed. Previous research had suggested that the presence of three-component particles in cases where two component primed ammunition had been used was likely attributable to the weapon memory effect, with the probability of Sb being incorporated from the surface of the projectile being low [17]. This indicated that any three-component particles detected were a result of particles generated by previous discharges of three-component primed ammunition being retained within the firearm and being distributed with subsequent firings.

The weapon memory effect is well established as a mechanism by which particles from previous firings are retained and redistributed in subsequent firings [22, 172]. However the previous study, at least one characteristic particle was detected in 64% of cases, and 82% of cases returning single element Sb particles [163]. At the same time, nearly 41% (15/37) of the ammunitions involved were Winchester brand, known to use two component primers, and only 2.7% (1/37) of the ammunitions were confirmed to use three component primers. This is reflective of the availability of the different ammunition products in Australian market, with the vast majority using two component primers, with no Sb present in the priming compound [17]. For the weapon memory effect to explain these observations, it relies on the assumption that the majority of firearms under consideration had discharged three-component primed ammunition, which is comparatively uncommon in the Australian market, before being reloaded with a more common two-component primed ammunition. This scenario seemed unlikely, given the observations of the previous study, combined with the relative scarcity of three-component primed 0.22 LR rimfire ammunitions in the Australian market. As proposed in the previous study, it seemed that a more likely explanation for the presence of Sb in GSR originating from two component primed ammunition was the incorporation of Sb from the surface of the projectile, where it is used to harden the Pb [1]. The previous study had used SEM-EDS and X-ray mapping as a means of demonstrating that Sb present in GSR was colocated with Pb, supporting the hypothesis that the Sb had originated from the Sb/Pb used in the surface of the projectile. As a means of further testing this hypothesis, the use of a FIB was proposed. This approach allowed the internal structure of the particles to be mapped, as a means of determining exactly where the Sb was located. The FIB was also to be used to further prepare samples from GSR to be analysed using alternative techniques, such as ToF-SIMS or AES, as a means of more conclusively identifying the origin of the Sb.

As a further point of investigation, other research had indicated that investigating the subparticle morphology of GSR, particularly as it pertains to heavy metal free (HMF) or Pb-free ammunitions may be a fruitful line of inquiry to enhance GSR investigations [176]. Although initial research had been performed in this arena [177, 178], this line of inquiry had not been continued. Previous work by Basu had established the heterogeneous distribution of Pb, Sb and Ba in both the surface and sub-surface morphology of GSR particles [11]. Basu further proposed that the formation process was as a result of thermodynamic differences between the elements, principally the differences in melting and boiling points of the elements that may be present in GSR [11, 107]. This was further explored by Nunziata and Donghi, who proposed that electrostatic attraction of Pb nanoparticles, coupled with the overall instability of Pb droplets was responsible for the formation of Pb nodules in GSR particles [179]. It can therefore be predicted that similar relationships exist between other elements commonly encountered in GSR, including those originating from HMF or Pb-free ammunitions, creating the possibility that distinct particle morphology at the surface and sub-surface level may be observed. As the FIB was to be used for the investigation of the origin of Sb, it was thought that this could be extended to cross-sectioning further particles from HMF and Pb-free ammunitions, to provide further data about their sub-particle morphology and composition. It was further hoped that compositional or morphological features characteristic of a particular ammunition could be identified.

This information could then potentially aid in identification of a particular ammunition or perhaps narrow the field of possible sources of GSR.

With regard to a Bayesian network system of assessing GSR evidence, any additional information obtained about the GSR originating from a particular ammunition can be used to inform nodes related to the firing condition or circumstances around discharge. The probability of observing a particular type of GSR may be linked to a number of different factors, including the availability and popularity of a particular ammunition within the jurisdiction, and the likelihood of observing particles originating from that ammunition. As it pertains to 0.22LR ammunitions, if three-component particles can be generated from two-component primed ammunition through incorporation of Sb from the surface of the projectile,

and three component particles are observed in the case sample, then the evaluation of the evidence must be updated to include the possibility that a 0.22LR two-component primed ammunition may have been used. Similarly, if there are particular sub-particle morphological and compositional features that are limited to a particular group or type of ammunitions, and said particles are observed on a case sample, there is an increased likelihood that one of these ammunitions has been used. In this way, obtaining further information about GSR originating from specific ammunition types can further inform a Bayesian network type approach to the evaluation of GSR evidence.

Elements of the research conducted in this chapter were peer-reviewed and published [180]. The publication first appeared in:

Lucas, N.; Seyfang, KE.; Plummer, A.; Cook, M.; Kirkbride, KP.; Kobus, H. Evaluation of the Sub-Surface Morphology and Composition of Gunshot Residue using Focussed Ion Beam Analysis. *Forensic Science International*, **2019**, 297, pp. 100-110.

For the purposes of this publication, the approximate contribution of each author was N. Lucas 55%, KE Seyfang 15%, A Plummer 5%, M. Cook 5%, K. P. Kirkbride 10%, H. Kobus 10%.

The full text of the publication has been incorporated in section 4.3 below.

Please note – Minor formatting amendments have been performed to the presentation of the publication to keep it consistent with the presentation of the thesis, however text and data remain unchanged from the published version.

4.3. EVALUATION OF THE SUB-SURFACE MORPHOLOGY AND COMPOSITION OF GUNSHOT RESIDUE USING FOCUSSED ION BEAM ANALYSIS

4.3.1. Introduction

Gunshot residue (GSR) particles are formed through a series of complex, high temperature and pressure interactions in the immediate aftermath of a firearm discharge. Under firing conditions, temperatures may reach levels in excess of 2000°C, with pressures reaching levels of 9500 kPa [11]. GSR particles have been observed to be composed of elements originating from the ammunition's primer, as well as inclusions from the cartridge case, projectile, firearm, or previous firings [10, 27, 107]. Identifying the provenance of suspected GSR particles, including differentiating GSR from non-GSR, provides valuable information able to contribute to criminal investigations. Prior research conducted on particles similar to GSR, generated from explosive residues, has demonstrated that different explosive compositions are capable of generating particles that can be differentiated based on their internal morphology [157]. Therefore, the composition of the primer in ammunition may also influence both the composition and morphology of GSR particles.

While firearm primers tend to have similar major components, an example of which can be seen in Table 1, individual ammunition manufacturers modify the composition of their primers and powders based on their individual needs and requirements.

Table 30 - Typical composition of a Sinoxid type primer compound, relative abundance and purpose in
primer mix [1]

COMPOUND	CONCENTRATION	PURPOSE
Lead Styphnate	25 % - 55%	Explosive
Barium Nitrate	24% - 25%	Oxidising Agent
Antimony Sulphide	0% - 10%	Fuel
Lead Dioxide	5% - 10%	Oxidiser/Corrosion Inhibitor
Tetracene	0.5% - 5%	Sensitiser
Calcium Silicide	3% - 15%	Fuel / Frictionator
Powdered Glass	0% - 5%	Frictionator

An example such a change of requirements is the transition to lead or heavy metal free primers. Repeated firing of ammunition containing Sinoxid type primers can result in significant exposure to airborne lead and other heavy metals that may pose a risk to the health of those exposed [30, 31, 33, 181]. To address these concerns, steps have been made to remove lead compounds from a variety of ammunition [70]. An example of one such primer is 'Sintox' type primers, containing titanium and zinc compounds. A typical composition can be seen in Table 32.

COMPOUND	CONCENTRATION	PURPOSE
Diazodinitrophenol (DDNP)	~15%	Explosive
Tetracene	~3%	Explosive
Zinc Peroxide	50%	Oxidiser
Powdered Titanium	5%	Fuel
Nitrocellulose	27%	Fuel

 Table 31 - Typical Composition of a Sintox type primer compound, relative composition and purpose

 in the primer mix [1]

Alternative heavy metal free primer compositions have used strontium salts of dinitrodihydroxydiazobenzene as explosive compounds, coupled with strontium sulfate or oxalate as passivating compounds, alongside components such as tetracene and zinc peroxide [182].Changing primer formulations impacts the types, compositions, and morphologies of particles that may be observed in casework. To ensure that GSR evidence is analysed and placed in the appropriate context, ongoing investigation of primer composition and the particle types generated, using a variety of techniques, is essential.

In practice, the most well-established technique for the analysis of GSR particles is scanning electron microscopy with energy dispersive x-ray microanalysis (SEM-EDS). While this technique allows the assessment of both morphology and composition of GSR particles, it is limited in that only the surface features of the particle are observable. Focussed ion beam (FIB) systems have the capability to cross-section small particles, permitting their internal morphology to be observed. The technique has been well established in the semiconductor industry, but has also seen use in materials science and micro- and nanoscale fabrication applications [183-186]. This technique has also been explored for forensic applications, and shown promise as a means of obtaining additional information from GSR particles [178, 187, 188].

Work conducted by Niewöhner and Wenz [177] on the application of the focussed ion beam to gunshot residues showed that each of the four different ammunition products in their study produced particles with different internal morphologies. Further work has indicated that the use of FIB for GSR analysis has utility in providing additional information to the assessment of this type of evidence [187]. Additional recent research has suggested that assessing the internal structure of GSR particles, particularly those originating from non-toxic primers, may be of benefit in providing additional information to GSR investigations [176].

Another potential contribution to the composition of GSR particles is the phenomenon known as the 'weapon memory effect'. The 'weapon memory effect' describes the circumstances in which particles retained within the firearm from previous firings are ejected alongside, or incorporated into, particles generated from subsequent firings. This effect has been demonstrated to result in the formation of mixed primer composition particles – particles that contain elements that are known to be absent in the primer mix of the ammunition used [23, 172, 176]. Recent work has further explored the origin of mixed primer composition particles, suggesting that particles retained from previous firings are incorporated into the structure of new particles generated in subsequent firings [189]. This results in particles with distinct compositional domains becoming visible using EDS mapping.

This study seeks to build upon the existing knowledge of the different types of particles and their internal morphologies that has previously been explored. An expanded mix of ammunition, including heavy metal-containing and lead-free variants has been included to investigate the impact of different primer formulation on the particle types formed. This research set out to assess the internal morphology and composition of GSR particles originating from a variety of different ammunition products, in an attempt to determine if differentiation of ammunition based on internal structure was possible.

4.3.2. Materials and methods

Sample Collection

All samples collected for the purposes of this research were collected using 12.5mm diameter Aluminium SEM pin stubs coated in double sided carbon-tape adhesive (Tri-Tech Forensics Inc. North Carolina, USA).

Representative GSR samples were collected from a selection of different ammunition, chosen to include exemplars of non-toxic primed and traditional three-component primed ammunition in a variety of calibres. A complete list of the ammunition used, the typical GSR particle types and components of their primers, cartridge and projectile can be seen in Table 33 and Table 34.

AMMUNITION TYPE	Ватсн	CALIBRE	Primer
PMC Zapper	22-D-794	0.22 Long Rifle	Ba, Pb,
Winchester Winclean (HMF)	SL10	0.40 S&W	Ba, Al,
American Eagle	VA27A57	0 10 58.11/	Sr Al
(Toxic Metal Free Primer)	V4224J7	0.40 3& W	<i>31, A</i> 1
Federal Premium HST	C19V26	0.40 S&W	Pb, Sb, Ba
Norinco	-	7.62 x 39mm	Pb, Sb, Ba

Table 32 - Ammunition Tested and Major Components present [70]

AMMUNITION	CARTRIDGE CASING	PROJECTILE TYPE
PMC Zapper	Brass (Cu, Zn)	Pb (Sb, Cu traces)
Winchester Winclean (HMF)	Brass (Cu, Zn)	Pb, Brass Enclosed Base (Cu, Zn)
American Eagle (MFP)	Brass (Cu, Zn)	Cu TMJ, Pb core.
Federal Premium HST	Ni coated Brass (Cu, Zn)	Hydra-Shok (Cu jacketed HP with Pb core)
Norinco	Steel Case (Fe)	Cu FMJ, Pb core.

Table 33 - Components Present in Cartridge Casing and Projectiles

n.b.: HMF = Heavy Metal Free, MFP = Metal Free Primer, TMJ = Total Metal Jacket, HP = Hollow Point, FMJ = Full metal Jacket. HST is a brand indication, and does not represent an initialism that can be defined.

PMC Zapper ammunition (22 Long Rifle rimfire) was included in the survey in order to investigate the generation of three-component particles from a two-component primed ammunition. PMC Zapper was included as analysis of unfired primer has demonstrated that it only contains Pb and Ba compounds, but has small quantities of Sb in the projectile, which is included to harden the lead of the projectile[1, 160]. Three-component particles generated from two-component primers have been observed, and their possible origins have been previously investigated [17, 163]. It has been hypothesised that the Sb incorporated into three component particles originates from the surface of the projectile. FIB milling and mapping of the interior of one such particle was used as a technique to investigate if internal composition could provide additional information that would allow the source of the Sb to be determined.

To address this possibility, a Ruger 77/22 bolt action, 0.22 calibre rifle was thoroughly cleaned. Ten rounds of PMC Zapper 0.22LR ammunition was discharged through the firearm to condition the barrel. A wooden probe was used to collect a sample from the inside of the barrel, which was then rolled onto an SEM stub. The breech was then also sampled with a wooden probe, and recovered to an SEM stub. A plastic (PET) catcher, fashioned from a clean, dry water bottle of approximate volume 1.25L was then affixed to the barrel using cloth adhesive tape. A further three rounds were discharged through the catcher. The holes in the catcher were sealed with cloth tape, and the catcher was removed from the barrel and sealed. The catcher was then transported back to the laboratory, where it was cut open, and the inside surfaces directly sampled with an SEM stub. Ultimately, the particles used for the FIB analysis were present on the sample from the inside of the catcher.

Samples from additional ammunition with different primer compositions were then collected as a means of investigating how primer composition may impact sub-particle morphological features. Particular focus was given to ammunition that have particular significance in the local jurisdiction.

Winchester WinClean was included as a primer composition that lacked both Pb and Sb, but did contain Ba, to serve as a basis of comparison to other primer formulations.

American Eagle MFP 0.40 S&W is the standard issue ammunition used in firearms training for SA Police (SAPOL). Similarly, Federal Premium HST 0.40 S&W was included as it is the standard issue operational ammunition carried by SAPOL. All 0.40 calibre ammunition was fired using a 0.40 Smith and Wesson Military and Police (M&P) Semi-Automatic Handgun.

Norinco 7.62x39mm is a Chinese made, steel-cased ammunition with a Copper FMJ projectile. Although in most markets it is no longer available or sold, it is of particular interest in the Australian context, as stockpiles of this ammunition are still routinely encountered in firearms casework, particularly in situations involving outlaw motorcycle gangs [190]. A similar situation may also be relevant beyond Australia. The Norinco ammunition was fired through a Norinco SKS Self-loading rifle.

In each case, the firearm was conditioned by discharging six rounds of the nominated ammunition through the firearm into a bullet recovery (water) tank. Following this, samples were taken from the hands of the shooter using an adhesive SEM pin stub. The shooter's hands were washed thoroughly before commencing each sample run, and a blank sample of their hands was collected prior to handling and discharging the firearm.

Cartridge casings were collected to verify the components that were detected in the hand residues. Residues from the casings were recovered to an SEM Stub prior to analysis using a wooden probe.

Equipment

All collected samples were analysed by SEM-EDS using an Inspect F50 Scanning Electron Microscope (FEI Inc., Oregon, USA) equipped with an EDS detector (EDAX Inc., New Jersey, USA.) Instrument operating parameters were established as per the American Society of Testing and Materials (ASTM) E1588-10-17 Standard guide for gunshot residue analysis by SEM/EDS [153].

Automated particle search was conducted using the GSR Magnum Particle analysis system (FEI Inc., Oregon USA), and TEAM elemental analysis software (EDAX Inc., New Jersey, USA).

The GSR Magnum system brightness and contrast settings were calibrated through use of a Gold/Niobium/Germanium/Silicon/Carbon (Au/Nb/Ge/Si/C) standard (Ardennes Analytique, sprl. Belgium).

A positive control for the FEI system, a synthetic particle standard (PLANO W. Plannet GmbH, Wetzlar, Germany) consisting of accurately deposited particles of known size was analysed at the start and end of every sample run. The 'particles' deposited on the glassy carbon surface of this standard are thin Pb/Sb/Ba films of sizes 0.5-10 μ m.

The Focussed Ion Beam used in this study was an FEI DualBeam Helios Nanolab 600 SEM/FIB system hosted at the Australian Microscopy and Microanalysis Research Facility (AMMRF) Facility at Adelaide Microscopy, University of Adelaide, South Australia.

Particle Analysis

ASTM E1588-10-17 was used to classify detected particles as either 'characteristic of' or 'consistent with' a firearms origin.

Particles of interest were first found and visualised using the FEI F50 SEM operating in backscattered electron mode (BSE), and images captured in both SE and BSE mode. Representative spectra of the whole particles were collected using the TEAM elemental analysis software. The locations of these particles were mapped so that they could be examined in greater detail later using EDS.

Several particles were identified *via* SEM for sectioning by FIB. Particles were selected based on composition and morphology, and were chosen to be representative of the population of particles originating from the ammunition. To make this assessment, information about the primer composition, cartridge and projectile were obtained. All samples were processed through GSR particle analysis software to ascertain the number of particles in each classification, and then a selection was made based on particle composition, size, shape, and the region of the stub in which it was located. Composition of particles was determined by re-acquisition of the particles and a 30 second EDS scan of the bulk of the particle. Compositional elements were compared against known components of the ammunition based on literature and cartridge recovery samples.

Particle size was a selection criterion for particles that were to be sectioned using the FIB process. Particles that are large are easy to find in the FIB instrument, but take a long time to section and polish for mapping. Smaller particles are harder to locate, but are faster to section. For the purposes of this analysis, features within the particles were the primary interest, so the particles that were selected ranged in size between 5µm and 50µm in diameter.

The shape of the particle was considered as a means of ensuring the FIB process could proceed unhindered. Non-spheroidal particles, or those with protrusions or odd shapes were excluded, as this made it difficult to accurately cross-section across the bulk of the particle. Selected particles were spheroidal in nature, with every attempt made to section across the bulk of the particle.

The region of the stub was a concern as the area around the particles of interest impacted the ability of the FIB to access the particle. For example, particles resting in a depression on the stub, or partially obscured by other features of the stub were excluded due to the technical difficulty in accessing them with the FIB instrument.

Particles were relocated on the stub using the FEI Dualbeam Instrument, and images collected in secondary electron mode. The stage was then tilted to an angle of 52° to expose the side of the particle, and confirm that the particle was accessible for cross-sectioning.

The particles of interest were cross-sectioned by milling using a Ga liquid metal ion source (LMIS). Prior to sectioning, a 2µm thick layer of Pt was deposited over the location to be cut. The Pt is deposited using a Pt gas injection system, and serves a number of purposes in FIB analysis. First, it provides a conductive layer in proximity to the freshly milled surface, which serves to minimise or prevent the appearance of artefacts of the FIB process. One such artefact is the 'curtaining' or 'waterfall' effect, which is characterised by pronounced lines visible on the milled surface [183, 191]. The Pt layer also confers a level of protection and stability to the surface of the sample to allow it to withstand the milling process. It should be noted that the Pt cap is visible in the figures of the cross-sectioned particles. It appears as a region around the top surface of the particle that appears bright in the BSE image, but shows low x-ray counts in the resultant maps.

FIB operational parameters used were determined based on the advice of an experienced operator. A beam current of up to 21nA was used to mill the bulk of the particle mass away and expose the interior of the particle. Once the surface was milled, it was polished using a reduced beam current of approximately 2.8nA. Using the SEM within the Dualbeam instrument, images were collected before, during and after the milling process.

After the particles had been cross-sectioned using the FIB, their EDS spectra were re-acquired using the FEI F50 SEM instrument. A stage tilt of 45° was used to permit clear imaging of the exposed surface. Images were collected in both SE and BSD mode. To assess the internal composition of the particle, an elemental x-ray map was collected using TEAM software.

4.3.3. Results and Discussion





Figure 26 - Exemplar EDS spectrum of GSR from 0.22LR calibre PMC Zapper ammunition (Inset - SE Image)



Figure 27 - EDS maps of FIB sectioned particle from 0.22 LR calibre PMC Zapper ammunition. (L to R - Backscattered electron image, Ba map, Sb map, Pb map, Cu map)

Figure 26 shows the EDS spectrum and whole particle secondary electron image from a three component particle, approximately 5µm in diameter, generated from 0.22 calibre rimfire PMC Zapper Ammunition. This particle was collected on an SEM stub from the interior of the plastic catcher. Figure 27 shows the same particle after cross-sectioning, and the collected EDS maps. In Figure 26, the particle is seen perpendicularly from the stub surface. The particle was then cut perpendicularly, but in Figure 27 the view is at a 45° angle to the surface of the stub and the surface of the cut to allow the cross-section to be seen.

PMC Zapper only contains Sb in the projectile, where it is used to harden the Pb. In this case, like many 0.22 rimfire ammunition, PMC Zapper does not use antimony sulphide as a component of the primer, but does have Pb- and Ba- containing compounds. This composition has been documented previously by Seyfang et al.[160]. Analysis of primers taken from unfired PMC Zapper cartridges using SEM-EDS, showed only Cu compounds present in addition to Ba and Pb. This ammunition does however have antimony present as a component of the projectile, where antimony is used to harden the lead. The particle pictured in Figure 26 was recovered from barrel residues, and therefore may be expected to include components of the projectile that have been retained in the barrel. It is also therefore possible that the composition of particles from the barrel discharge is different to that of particles distributed from the ejection port of a firearm. Previous work conducted by Zeichner et al. [17] has indicated that Sb enrichment on a projectile's surface results in only a small probability of finding this element in GSR from ejection port residues.

From the SE image in Figure 26, it can be seen that morphologically the particle exhibited a pockmarked and irregular surface appearance. In cross-section, observable in Figure 27, the internal structure of the particle appears to be regular and uninterrupted, free of voids or cavities. Compositionally, the Pb is present toward the centre, and appears to make up the bulk of the particle. Although it is present, Ba is at minor to trace levels across the particle. Based on the BSE signal, Ba appears to be richest in regions where Pb is absent and Sb is diffuse. Where it is present, Sb is seen in higher concentrations in discrete regions of the particle. This can be observed at the top of the particle in a small 'cap' and towards the bottom right of the particle; it appears that the bulk of the central Pb deposit does not contain Sb. It is also notable that Sb and Ba are co-located as trace deposits in regions where Pb is absent. Regions of the particle that have Sb present also show the presence of Cu and Pb. There are numerous sources of Cu within this ammunition, including as a component of the primer, within the cartridge brass, and as a Cu wash applied to the surface of the projectile. The absence of Zn in the spectrum of the full particle, or the EDS maps, suggests that the source of Cu is not the cartridge brass. Similarly, Cu compounds in the primer would be predicted to co-locate with other elements in the primer, such as the Ba and Pb. Taken together, the fact that the Cu appears to be most concentrated into areas in which Sb and Pb are also located suggests that the Sb present in these particles has originated from the surface of the projectile. Increases in the number of particles of Pb when using non-jacketed ammunitions (compared to jacketed ammunition) has previously been reported by Wolten [2], Wallace [1], and Udey et al [18].

However, it has also been documented that materials from previous firings, such as condensed vapours or particles can be retained in the barrel or on surfaces of the weapon, only to then be deposited in subsequent firings. This phenomena is known as the weapon memory effect, and has been described in detail elsewhere [22, 23, 172, 189, 192]. In this instance, steps were taken to minimise the impact of the weapon memory effect. The firearm was thoroughly cleaned before use, and was then conditioned with several rounds of the PMC Zapper

ammunition before samples were taken. The stub on which this particle was located reported in excess of 200 three-component particles in approximately 25% of the stub area. A complete breakdown of the particles detected can be seen in Table 35.

PARTICLE TYPES	Particles Present (n)	% OF CLASSIFIED	% TOTAL PARTICLES
CH	IARACTERISTIC OF G	SR	
PbSbBa	200	1.4	1.0
C	ONSISTENT WITH GS	R	
BaSb	106	0.7	0.5
PbSb	1161	8.0	5.6
PbBa	335	2.3	1.6
BaCaSi	4	0.03	0.02
Total Consistent	1606	11.0	7.7
	d with GSR (S ingi	LE ELEMENT PARTI	CLES)
Pb	5244	36.0	25.2
Sb	509	3.5	2.5
Ва	353	2.4	1.7
Total Commonly Associated	6106	41.9	29.4
Total Other Classified Particles*	6650	45.7	32.0
Total Classified	14562		
Total Unclassified	6211		
Total Particles	20773		

Table 34 - Particle count from PMC Zapper ammunition barrel discharge sample (25% of available stub
area).

*The 'Other Classified particles' category includes any particles identified that are not pertinent to GSR analysis. This includes, but is not limited to, particles such as Fe, Au, Ag, BaS, Bi, Zr.

As a means of ascertaining the extent of the contribution of the weapon memory effect to these data, first, it is important to note that the firearm was thoroughly cleaned prior to collection of GSR. Second, as it is known that even thorough cleaning cannot completely remove GSR from previous firings from within the barrel [193], the firearm was further conditioned with the discharge of 10 rounds of PMC Zapper through the firearm. The rationale behind this was to displace particles in areas that cleaning might not reach and to 'load' the barrel with particles originating from the ammunition of interest, such that any particles retained from previous discharges would represent a smaller percentage of the total residues. A sample was collected directly from the barrel using a wooden probe and compared against the discharge residues. Following this, three rounds were discharged through a plastic catcher, with the inside of the catcher being sampled directly using an SEM stub. Finally, a scraping from the breech was collected using a wooden probe. A comparison between the results from the three samples can be seen in Table 36.

PARTICLE TYPES	PARTICLES PRESENT (BARREL SCRAPE) (100% Stub Analysed)	Particles Present (Barrel Discharge) (25% Stub Analysed)	PARTICLES PRESENT (BREECH SCRAPE) (100% STUB ANALYSED)
	CHARACTERISTIC OF C	iSR	
PbSbBa	3	200	0
	CONSISTENT WITH G	SR	
BaSb	2	106	0
PbSb	19	1161	53
PbBa	110	335	16
BaCaSi	8	4	0
Total Consistent	139	1606	69
COMMONLY ASSOC	IATED WITH GSR (SING	ILE ELEMENT PARTIC	cles)
Pb	1095	5244	1087
Sb	2	509	20
Ва	68	353	5
Total Commonly Associated	1165	6106	1112
Total Other Classified Particles*	32	6650	332
Total Classified	1339	14562	1513
Total Unclassified	933	6211	355
Total Particles	2272	20773	1868

 Table 35 - Comparison of particle types collected from barrel scraping, barrel discharge, and breech scraping.

*The 'Other Classified particles' category includes any particles identified that are not pertinent to GSR analysis. This includes, but is not limited to, particles such as Fe, Au, Ag, BaS, Bi, Zr.

While the possibility that some of the particles present on the discharge stub originate from previous firings cannot be excluded, the conditioning step, coupled with the number of particles detected on the post-discharge stub suggests that the proportion of particles originating from PMC Zapper ammunition would outnumber those retained from previous firings. This indicates that a significant number of three component GSR particles are generated from this ammunition. The selection of the particle to cross-section was targeted to ensure that it was typical of the population of three component particles present and was therefore most likely to have originated from the PMC Zapper ammunition used in test firings rather than from previous firings. However, even with these controls in place, we acknowledge the possibility that the particle that was sectioned is a three-component particle retained from a previous firing and collected coincidentally.

0.40 Smith and Wesson American Eagle MFP

When considering the 0.40 calibre American Eagle MFP ammunition, two main types of particles were observed. The first were uniform, regular spheroids that contained both Sr and Al. A trace amount of Cu was also present in many of the particles. Figure 28 and Figure 29 show a representative particle of this classification approximately 40 µm diameter. In cross-section, it can be seen from Figure 29 that the interior of the particle is smooth and uninterrupted, with neither voids, nor regions of high concentrations of specific elements present. It should be noted that the region visible in Figure 29 that appears as a bright crescent shape in the BSE image is the Pt deposit, which was deposited as a part of the FIB process.



Figure 28 - Exemplar EDS spectrum of GSR from 0.40 American Eagle MFP ammunition (Inset - SE Image)



Figure 29 - EDS maps of FIB section particle from 0.40 American Eagle MFP ammunition (L to R - Backscattered electron image, Al map, Sr map, Cu map)

The second particle type were rough, irregular spheroids, with some exhibiting cracking or fissures on their surface. Compositionally, these were primarily composed of Sr, with no observable traces of other elements from the primer, firearm or cartridge. A comparison of particles of similar sizes can be seen in Figure 30.



Figure 30 - Comparison of morphological differences between Sr only (top) and SrAl (bottom) containing GSR particles originating from HMF ammunition (American Eagle 0.40 MFP)

A similar observation of cracked, fissured particles was observed by Oommen et al. in their study of Speer Lawman Cleanfire ammunition [70], but in that work the particles contained Sr and Al. In the current study, however, it appears that the presence of these two elements within a single particle tends to result in a more rounded, smooth, and uniform appearance. When Al is absent, the particles generated tend to display a more irregular and cracked appearance at their surface.

0.40 Smith and Wesson WinClean

Particles originating from the 0.40 Winchester Winclean ammuntion exhibited an amorphous, and 'bubbly' morphology which can be observed in Figure 31 and Figure 32. The internal morphology of these particles exhibited high porosity.



Figure 31 - Exemplar EDS spectrum of GSR from 0.40 Winchester Winclean ammunition (Inset - SE Image)



Figure 32 - EDS maps of FIB sectioned particle from 0.40 Winchester Winclean ammunition. (L to R - Backscattered electron image, Al map, Si map, Ba map, Cu map)

The particle that was cross-sectioned was approximately 10 µm in diameter. The regular spheroidal shape of the bulk of the particle suggests that the particle is solidified foam, or the solid particle matrix condenses from the vapour or liquid phase. In this case, Ba and Al are co-located throughout the bulk of the particle, with small amounts of Si also present throughout. Additionally, a small amount of Cu is visible in both the spectrum in Figure 31 and the maps in Figure 32. The fact that the Cu appears localised to small nodules on the surface of the particles, and is not co-located with any other elements (especially Zn) suggests that these small inclusions originate from the Cu jacket of the projectile rather than the brass cartridge case. This further speaks to the possibility of incorporation of elements present only in the projectile into the GSR particles that are generated.

0.40 Federal Premium HST



Figure 33 - Exemplar EDS spectrum of GSR from 0.40 Federal Premium HST ammunition





Figure 34 - EDS maps of FIB sectioned particle from 0.40 Federal Premium HST ammunition (L to R - Backscattered electron image, Ba map, Sb map, Pb map)

In Figure 33, the collected spectrum and SE image of a GSR particle originating from 0.40 S&W calibre Federal Premium ammunition are shown. The particle is approximately 50 µm in diameter. The inset SE image shows that although the particle has some surface cracking, it is spheroidal, with small nodules evident. The particle was identified as having 'characteristic' GSR particle composition (Pb, Ba, and Sb). In Figure 34, the heterogeneous distribution of elements within the cross-section of the particle can be observed. When considering the EDS maps collected from the cross section, it can be seen that Ba and Sb are co-located, and appear to make up the bulk of the particle. The Pb, however, is located in discrete regions throughout the particle, and does not appear to be uniformly incorporated into the particle. The presence of this Pb as spheres embedded in a Ba and Sb matrix suggests that the nodules of Pb condense first, followed by the Ba and Sb condensing around them to form the final particle.

In this ammunition there are two potential sources of Pb – the Pb compounds used in the primer, and alloyed Pb in the core of the projectile. In this case, the core of the projectile, including the base, is encapsulated in a Cu jacket, making it unlikely that the Pb core of the

projectile would be exposed to either friction in the barrel, or the burning powder. Therefore, it is comparatively unlikely that the Pb observed in this particle is derived from the core of the projectile. This indicates that the small nodules of Pb visible in this particle are derived from the Pb compounds in the primer, suggesting that either the primer formulation, or the conditions of the firearm discharge have allowed the Pb to separate and condense independently of Sb and Ba. An alternative possibility is that the small Pb particles are already present in the firearm due to the weapon memory effect, and are incorporated into the final particle during the firing process.

7.62 x 39mm Norinco Steel Case



Figure 35 - Exemplar EDS spectrum of GSR from 7.62x39 calibre Norinco ammunition





Figure 36 - EDS map of FIB sectioned particle from 7.62x39 Norinco (Steel Case) (L to R - Backscattered electron image, Ba map, Sb map, Pb map, Fe map, Cu map)

Particles originating from Norinco ammunition exhibited interesting morphological and compositional features that have not been widely observed or reported in GSR from other sources. As can be observed in Figure 35, this ammunition generated particles that meet the requirements for 'characteristic' morphology and composition under the ASTM standard. The

exemplar particle was approximately 15 µm in diameter, and contained Pb and Sb at a major level, with trace amounts of Ba, along with trace quantities of Fe and Cu. In Figure 36, the internal morphology of the particle can be observed. From this cross-section it is evident that the Pb-rich and the Sb-rich regions do not appear to co-locate. Interestingly, it appears as though small nodules of Sb are captured in a Pb-containing matrix. While not as pronounced, this separation of components was visible on the surface of the particles, with a number of particles exhibiting a mottled or speckled appearance. Although the spectrum in Figure 35 indicates that Ba is present at trace levels in the whole particle, upon sectioning, a region of high Ba concentration was observed inside the particle with small nodules of Ba present on the underside of the particle.

Trace concentrations of Fe were present in a number of the particles observed, showing some co-location with Ba. Trace levels of Cu were observed homogeneously across the entire particle. While the particle type observed in Figure 36 was commonly observed in this sample, other types were observed. Figure 37 shows a particle that exhibits some of the features observed in the previous particle type, notably the mottled-type particle containing discrete regions of Pb and Sb, however this was observed to be embedded in a larger primarily Bacontaining matrix.





The presence of both types of these particles suggests complex interactions at play in the formation of these GSR particles. The appearance of the PbSb particle embedded in the Ba matrix as observed in Figure 37 suggests that Pb and Sb coalesce first, occupying discrete regions within the same spheroidal particle, then allowing the two component particle to be absorbed into a still molten Ba matrix. However, the particle pictured in Figure 36 indicates that Ba may be similarly captured within a PbSb droplet. In either case, particles of this particular composition and morphology were only observed in the Norinco ammunition, suggesting that the specific components of the primer itself and/or the idiosyncrasies of the discharge pressure and temperature result in the formation of particles of this type.

4.3.4. Conclusions

Several samples from different ammunition, including heavy metal free variants, were observed to generate particle populations with distinct compositions and morphologies. The use of FIB-sectioning coupled with EDS-mapping was useful in identifying distinct compositional domains in a cross-sectioned GSR particle that provide further information to the particle formation. In some cases, different components of the ammunition were seen to co-locate with each other. In three-component primed 0.40 calibre Federal ammunition, Ba and Sb were seen to co-locate, while these same elements occupied discrete regions in the particles from three-component primed 7.62x39 calibre Norinco ammunition. When considering heavy metal-free or non-toxic variants of different compositions, particle types ranged from solid spheroidal, SrAl-containing particles to porous, but regularly shaped, BaAl-containing particles. These observed differences between ammunition suggest, at least tentatively, that sub-particle morphology and composition may be used to provide additional information to forensic GSR analysis.

This study also provided additional support for the mechanism behind three component particles being observed in situations that involve only two-component primed ammunition. Previous work has indicated that even with thorough cleaning, the weapon memory effect can have an impact on the particles generated [194, 195]. In the work described here, controls were implemented to minimise the impact of the weapon memory effect, and yet FIB sectioning and EDS mapping still indicated mixed composition particles were present. These particles exhibited compositional elements that were not present in the primer mix, but had possible origins elsewhere in the ammunition. The origin of these elements, based on their location and composition was determined to be most likely originating from the surface of the projectile in a number of cases. Given the controls used to address the memory effect, the significant number of these three-component particles observed when using two-component primed ammunition are more than would be expected if they were retained from previous firings.

However, this finding is interpreted cautiously, and does not discount the contribution of the weapon memory effect to the detection of mixed composition particles, especially in casework where the prior usage of the firearm is not known. While the precise mechanism behind the formation of these particles has been difficult to ascertain, recent research has suggested that their formation may be due to incorporation of existing particles into the pre-solidified particle matrix. If this is the case, then the same mechanism that allows the incorporation of existing

particles from previous firings to be incorporated into new particles would theoretically also allow for incorporation of particles generated from other parts of the firing process. To that end, a cautious, case-by-case approach to the assessment of GSR is still encouraged.

It should be noted that there are difficulties inherent in evaluating evidence at the single particle level. This research has highlighted the highly variable and heterogeneous nature of GSR particles, and therefore any assessment or conclusions based on single particle composition or morphology must be treated with utmost caution and preferably involve the evaluation of the wider population of particles collected from the crime scene or person of interest. Although in this case care was taken in selecting the particles to ensure that they were representative of the wider population, this study still only considered individual particles. While surface features present in the cross-sectioned particles were observed in a number of others in the sample, it was impractical to cross-section large numbers of them in this study. Despite this, as a proof-of concept for the ammunition selected, the use of FIB on GSR particles did allow the internal morphology of GSR to be assessed, thereby gaining additional information about the samples and perhaps, more fundamentally, indicating that some ammunition-specific characteristics are present in GSR. It is evident that different primer compositions and different ammunition can produce particles that exhibit characteristics that have distinct sub-particle features, compositions and morphologies, information which can potentially assist discriminating ammunition products and enhancing the overall quality of GSR evidence.

----- End of Publication-----

4.4. FURTHER EXPLORATION OF THE WEAPON MEMORY EFFECT

4.4.1. Background

In a previous chapter, the possibility of 0.22LR ammunitions that lack Sb components in the primer, but generate three-component PbSbBa particles, was tentatively explored. This was initiated due to the observation that in a large number (64%) of suicide cases in which 0.22LR ammunitions were used, three-component particles were observed. This was at a number greater than would be expected, given that the majority of this ammunition contains primers that do not incorporate Sb components, specifically Sb₂S₃ as a component of the primer [170]. With no source of Sb present in the primer mix, it was thought that the only way three-component particles like this could be present due to a discharge of this ammunition is due to the weapon memory effect - GSR from previous discharges of three component ammunition retained in the firearm, and then expelled with subsequent discharge.

Prior to collecting GSR samples to be sectioned using FIB, a survey was conducted to assess the extent of the weapon memory effect under the proposed testing conditions. The objective of this survey was to establish how readily particles originating from the ammunition could be identified, compared to those that may have been retained in the firearm due to the weapon memory effect. The results of the FIB-sectioning discussed in section 4.3 indicated that in particles originating from two-component primed ammunition, Sb was observed co-located with Cu, the mostly likely source of which is the wash over the surface of the projectile. However, further exploration of the influence of the weapon memory effect, and how it may impact the distribution of particles was investigated.

4.4.2. Methods

To assess the potential contribution of the weapon memory effect, and to test the hypothesis that three component PbSbBa particles were generated through incorporation of Sb from the surface of the projectile, an additional experiment was performed.

A sample of 0.22LR CCI Stinger ammunition was collected. Stinger uses a primer based on a two component formulation and therefore like PMC Zapper, does not contain Sb as a component of the priming compound. Both ammunitions also have a CuZn cartridge, and both have a Cu wash over the surface of the projectile. Unlike PMC Zapper however, CCI Stinger does not include Sb as a component of the projectile (H. Wrobel 2016, personal communication, 5th October),. It was thought that by replicating the process performed with PMC Zapper using CCI Stinger, this would allow the types of particles observed from the barrel, breech and discharge residues to be assessed.

As previously, a cleaned Ruger 77/22 bolt action, 0.22 calibre rifle was loaded with ten rounds of CCI Stinger 0.22LR ammunition. This was then discharged through the firearm to condition the barrel. A wooden probe was used to collect a sample from the inside of the barrel, and the breech, before being recovered to an SEM stub. A plastic (PET) catcher, fashioned from a clean, dry water bottle of approximate volume 1.25L was then affixed to the barrel using cloth adhesive tape. A further three rounds were discharged through the catcher. The holes in the catcher were sealed with cloth tape, and the catcher was removed from the barrel and sealed. The catcher was then transported back to the laboratory, where it was cut open, and the inside surfaces directly sampled with an SEM stub.

All stubs were then analysed by SEM-EDS using an Inspect F50 Scanning Electron Microscope (FEI Inc., Oregon, USA) equipped with an EDS detector (EDAX Inc., New Jersey, USA.) Instrument set up and operating parameters were as previously established and described in Section 4.3.2 above

4.4.3. Results and Discussion

If particles retained within the firearm attributable to the weapon memory effect are as persistent and prevalent as previously documented [22, 23, 172, 189, 192], then it would be expected that a comparable contribution from the memory effect would be observed under similar circumstances.

Although the precise history of the use of this firearm was not specifically known, its use is comparable to the use of a randomly selected firearm in Australia. It is used to fire commercially available ammunitions, and therefore the proportion of three-component primed and two-component primed 0.22LR ammunition used in the firearm would be expected to be comparable to that observed in the wider market.

In each case, 10 rounds of the ammunition had been discharged through the firearm prior to sample collection. This step was taken to condition the barrel, generating residues from the ammunition of interest to ensure that they represented the bulk of the residues present when samples were collected.

Results obtained from the sample collected from the propellant catcher can be observed in Table 37

PARTICLE TYPES	Particles Present (n)	% OF CLASSIFIED	% TOTAL PARTICLES
Сн	ARACTERISTIC OF G	SR	
PbSbBa	28	0.6	0.4
Co	NSISTENT WITH GS	R	
BaSb	18	0.4	0.3
PbSb	197	4.0	2.8
PbBa	1013	20.5	14.2
BaCaSi	0	0.0	0.0
Total Consistent	18	0.4	0.3
COMMONLY ASSOCIATE	d with GSR (Sing	LE ELEMENT PARTI	CLES)
Pb	3336	67.6	46.9
Sb	3	0.1	0.0
Ва	188	3.8	2.6
Total Commonly Associated	3527	71.5	49.6
Total Other Classified Particles*	151	3.1	2.1
Total Classified	4934		
Total Unclassified	2176		
Total Particles	7110		

Table 36 - Particle count from CCI Stinger ammunition barrel discharge sample (100% of available stub
area).

*The 'Other Classified particles' category includes any particles identified that are not pertinent to GSR analysis. This includes, but is not limited to, particles such as Fe, Au, Ag, BaS, Bi, Zr.

From Table 37, it can be seen that even though CCI Stinger ammunition does not have Sb in its primer or in the projectile, there are still some PbSbBa particles present in the barrel discharge residues. Also worthy of note is the presence of other Sb-containing particle types, including PbSb particles, BaSb particles and Sb only particles. This finding suggests that the weapon memory effect does provide a contribution to the resultant discharge residues, even in the event that the firearm has been thoroughly conditioned. However, there is an abundance of particles with composition reflective of the primer and projectile of the ammunition used, with a large number of PbBa particles observed, and a significant quantity of particles classified as Pb-only. This offers further support of the position that conditioning the firearm ensures that the bulk of the residues observed originate from the ammunition most recently discharged. Further investigation of the distribution of particle types was considered by comparing a breech and barrel scraping to the collected discharge residues. This comparison can be seen in Table 38.

PARTICLE TYPES	PARTICLES PRESENT (BARREL SCRAPE) (100% STUB ANALYSED)	PARTICLES PRESENT (BARREL DISCHARGE) (100% STUB ANALYSED)	PARTICLES PRESENT (BREECH SCRAPE) (100% STUB ANALYSED)
	CHARACTERISTIC OF G	SR	
PbSbBa	0	28	0
	CONSISTENT WITH G	SR	
BaSb	0	18	0
PbSb	2	197	53
PbBa	322	1013	16
BaCaSi	2	0	0
Total Consistent	326	1228	69
COMMONLY ASSOC	IATED WITH GSR (SING	ILE ELEMENT PARTIC	CLES)
Pb	710	18	1087
Sb	0	197	20
Ва	115	1013	5
Total Commonly Associated	825	3527	1112
Total Other Classified Particles*	67	151	332
Total Classified	1218	4934	1513
Total Unclassified	1118	2176	355
Total Particles	2336	7110	1868

 Table 37 - Comparison of particle types collected from barrel scraping, barrel discharge, and breech scraping after firing CCI Stinger Ammunition.

*The 'Other Classified particles' category includes any particles identified that are not pertinent to GSR analysis. This includes, but is not limited to, particles such as Fe, Au, Ag, BaS, Bi, Zr.

Evident from the barrel scraping after firing CCI Stinger ammunition is a smaller proportion of Sb containing particles, but without any PbSbBa particles observed. Similarly, in the sample collected from the breech of the firearm, a small number of PbSb and Sb only particles are observed, but no PbSbBa particles. The most abundant population in the case of the barrel residues is Pb-only, PbBa and Ba only particles, which reflect the bulk composition of the primer and projectile in CCI Stinger ammunition. At the breech, the largest population was Pbonly particles, with a small number of PbSb and even fewer PbBa observed. In each case, the largest population of particles can be attributed to those that could be generated from the surface of the projectile. If it is considered that the presence of Sb in any residue is an indication of the weapon memory effect, this indicates that a small, but significant percentage of residues from previous discharges is retained on the surfaces of the firearm, even many discharges later. This is in line with established knowledge of the topic [172, 189]. A direct comparison between the results observed from CCI stinger and PMC Zapper can be seen in Table 39

PARTICLE TYPES	CCI STINGER PARTICLES PRESENT (BARREL DISCHARGE)	PMC ZAPPER Particles Present (Barrel Discharge)
C+	IARACTERISTIC OF GSR	
PbSbBa	28	200
C	ONSISTENT WITH GSR	
BaSb	18	106
PbSb	197	1161
PbBa	1013	335
BaCaSi	0	4
Total Consistent	1228	1606
COMMONLY ASSOCIATE	ED WITH GSR (SINGLE ELEM	MENT PARTICLES)
Pb	3336	5244
Sb	3	509
Ва	188	353
Total Commonly Associated	3527	6106
Total Other	151	6650
Total Classified	4934	14562
Total Unclassified	2176	6211
Total Particles	7110	20773

Table 38 - Comparison of .22LR CCI Stinger and .22LR PMC Zapper Barrel Discharge Residues

The 'Other Classified particles' category includes any particles identified that are not pertinent to GSR analysis. This includes, but is not limited to, particles such as Fe, Au, Ag, BaS, Bi, Zr.

When compared against CCI Stinger, it can be seen that all particle populations that include Sb as a component show a significant increase when using PMC Zapper. This indicates that the inclusion of Sb as a component of the projectile has an observable impact in the number of particles in the overall population that contain Sb, including PbSbBa particles. However, the sample from CCI Stinger still produced some particles that contained Sb, including PbSbBa particles. This suggests that the weapon memory effect still has some influence over the types and number of particles observable in the final population. To that end, this suggests that the mechanisms underpinning particle formation are inherently complex, and must be considered as a part of GSR evidence evaluation.

4.4.4. Conclusions

These results indicate that the presence of Sb as a component of the projectile results in an observable increase in the number of particles in the population that contain Sb, including PbSbBa particles. While it is possible that some of the contribution is still attributable to particles that have been retained within the firearm from previous firings, due to the weapon memory effect, these data indicate that this is not the only mechanism. Considered in combination with the observations from the particles cross-sectioned by FIB, this suggests that incorporation of Sb from the surface of the projectile may contribute to the generation of PbSbBa particles from two-component primed ammunition.

4.5. FIB PREPARATION FOR ALTERNATE ANALYSIS TECHNIQUES

4.5.1. Background

A further consideration for the use of the FIB to assess GSR particles is the usefulness of the technique in other arenas to prepare samples for further analysis. The utility of the FIB system lies in the fact that it allows individual sections of a sample to be visualised before initiating sample preparation. For this reason, FIB has been useful in applications requiring ultrathin sample sections or very surface sensitive techniques. With these considerations in mind, FIB sample preparation has been applied to the preparation of thin sections for Transmission Electron Microscopy (TEM), Auger Electron Spectroscopy (AES) and Time-of-flight Secondary Ion Mass Spectrometry (ToF-SIMS). A further goal of this research was to prepare cross-sectioned samples by FIB, to allow AES or ToF-SIMS to be used to assess internal particle composition.

AES has an analysis depth of approximately 5nm, making it very surface sensitive. FIB has been used to assist these applications by milling away a surface, or assessing small features of a specific sample. Such applications have been explored by Scheithauer [185]. Even more surface sensitive are techniques such as ToF-SIMS, which have an approximate analysis depth of only 1 nm. ToF-SIMS also has the benefit of allowing for the analysis of a broader range of elements and compounds to be analysed, and has lower limits of detection [170]. As a technique, ToF-SIMS has been applied to GSR analysis at a surface level, with surface etching used to assess the top few layers of the surface [170]. While these results are promising, preparing a thin slice in cross-section would allow for more detail about the elements and compounds present throughout the particle to be obtained.

Advances in technological capability have permitted further development of FIB sample preparation techniques. One such technique is known as the 'lift out' technique, in which a small sub-sample of the material to be analysed is removed from the bulk, and then affixed to a Cu grid to be taken for further analysis. [196-198]. This sample preparation technique utilises the Pt cap applied to the top of the sample being analysed. In standard milling applications, the Pt cap serves to create a conductive surface closest to the milled area, minimise artefacts as a result of the milling process, and stabilise the sample surface to allow it to withstand the milling process. The Pt cap also provides a solid surface to weld a probe to the surface of the

sample in order to allow *in situ* movement or manipulation of the sample. This then provides the opportunity for a small slice of the sample to be extracted and prepared for further analysis. This method of sample preparation was attempted on GSR particles as a part of this study, but it was found that even at low current, the structural integrity of the GSR particle matrix was compromised, resulting in difficulty in successfully thinning and transporting the sample.

4.5.2. Method

The sample lift-out procedure was attempted on particles originating from three-componentprimed, 0.40 Federal Premium ammunition. The rationale behind attempting the process on particles originating from this ammunition was based on a number of considerations. First, this ammunition had been observed to generate relatively large (10 - 50 μ m) regular particles containing all three components. The size and shape of these particles resulted in them sitting clearly above the carbon tape, and allowed easier access for the milling and extraction procedure.

Prior sectioning of particles from this ammunition suggested that they were solid throughout, without significant voids or cavities internally that would complicate the extraction and thinning process. Particles generated from ammunition, such as the Winclean ammunition as observed in Figure 31, exhibited a significant number of internal voids within the particle. These voids had the potential to render the sample collected by sectioning more fragile, and more difficult to successfully thin, extract, and affix to the sample holder for transport. Though the surface of the particles generated from the Federal Premium ammunition displayed some surface cracking, the interior of the particle appeared mostly solid.

Finally, the interior of these particles was observed to be non-homogenous, with regions of high Pb embedded in a Ba- and Sb- containing matrix (as seen in Figure 33, above). As previously mentioned, this separation of components indicates that either the Pb-compounds present in the primer separate and condense first, with the Sb and Ba compounds condensing around them, or that small particles of Pb present in the barrel of the firearm due to the weapon memory effect are incorporated into the final particle as a part of the process. It was hoped that through sectioning and further analysis, further information about the source of these components could be determined.

4.5.3. Results and Discussion

A particle selected for the process can be seen in Figure 38 below. The particle is between 25 μ m and 30 μ m in diameter, and shows the same cracked and fissured surface as was present in other particles of this type. The green guidelines displayed in Figure 38 indicate the size of the slice that was to be removed, representing an initial slice of approximately 6 μ m thickness. It was initially hoped that this slice could be liberated from the particle and affixed to a TEM analysis grid, before being thinned to an appropriate thickness.



Figure 38 – GSR Particle from 0.40 Federal Premium ammunition as observed prior to FIB milling. The stage was then repositioned and tilted display the particle from the side. This view can be seen in Figure 39. A clear view of the particle at this angle is important, as it shows that all sides of the particle are readily accessible, and there is nothing to obstruct the attachment, milling, and lifting out of the particle by the FIB.


Figure 39 – GSR Particle from 0.40 Federal Premium ammunition at stage tilt of 52°, prior to deposition of the Pt cap



Figure 40 – The same GSR particle from 0.40 Federal Premium ammunition during the milling process. The platinum cap can be observed towards the top of frame. Towards the bottom of frame, the section currently being milled can be observed.

The particle during milling can be observed in Figure 40. In the cross-section, nodules, similar to the nodules of Pb that were observed to be present previously, can be observed. Also apparent in the image is the void in the particle at the bottom left of frame. This section of the

particle was embedded in the tape, and there was no evidence at the surface level of the particle that this void was present. While it was identified at this point that this void could pose a problem to the further sample preparation, it was hoped that the use of caution during thinning would allow the section between the void and the Pt cap to be retained. Ultimately, a section was extracted from this particle, and was moved and affixed to the copper grid for further analysis. This can be observed in Figure 41.



Figure 41 – Section extracted from the GSR particle after it had been affixed to the Cu TEM grid using Pt.

During the process of milling and extraction of the particle, the void, present towards the bottom left of frame in Figure 41, expanded, and was widened, stretching across the centre of the particle. Numerous other fissures are visible in the slice, indicating that the particle matrix is relatively fragile, and prone to cracking.



Figure 42 - Particle section following further thinning and polishing under the FIB.

Figure 42 shows further thinning and preparation of the particle section. The fissure from the previous image been milled away. Visible in this image towards the top of the particle is noticeable thinning of the top section. A number of holes and voids have opened, making it likely that the top part of the particle would also be lost. Towards the middle left of frame, a void is visible in a nodule of Pb, indicating that the integrity of this structure is also beginning to be compromised.

At this point in the process, further thinning and polishing of the surface was suspended. It was estimated that the resultant thickness of the particle was in the range of 1 μ m. In this case, extraction and transport of a slice of the GSR particle was unsuccessful, as the section was lost in-transit between the FIB instrument used for sectioning and the SEM that was to be used for elemental mapping.

A further attempt was made with a different type of ammunition, and with additional precautions taken to stabilise the GSR particle and section for transport. A thicker platinum cap was applied in an attempt to ensure further stability during thinning and lifting, as well as providing a stronger attachment point to the sample holder. A particle segment prepared in this fashion from 0.22 PMC Zapper ammunition can be seen in Figure 43.



Figure 43 – Particle segment prepared from 0.22 PMC Zapper ammunition. Note the thicker coating of platinum present on the top and sides of the segment.

Despite these efforts, additional samples could not be extracted for further analysis. In one instance, it was affixed to the Cu TEM grid, but did not remain attached, and was no longer present following transport. In the second instance, the sample was thinned and welded to the needle in order to be lifted out, but the weld failed, resulting in loss of the sample prior to it being affixed to the TEM grid.

One particle type that formed a notable exception to this was glass-containing GSR (gGSR), due to the stability and hardness of the glass matrix, which withstood the milling well enough to be extracted. Segments from these particles were extracted and further analysis was performed. The results of this study have been reported by Seyfang et al. [159].

4.5.4. Conclusions

While use of the FIB was successful in preparation of cross-sections of numerous GSR particles, attempts to prepare thin-sections for further analysis was observed to be problematic. The GSR particle matrix was fragile and did not endure the FIB milling process well. This resulted in difficulty in attaching the resultant segment to the TEM grid, and retaining it during transport, causing multiple fragments to be lost prior to further analysis. A notable exception was glassy GSR (gGSR) which proved to be more robust, and was successfully re-analysed using other techniques.

4.6. CONCLUSIONS

The available evidence suggests that a complex combination of factors can influence the formation and distribution of elements within GSR particles, with some differences in subparticle morphology and composition being observed consistently between different ammunition products. The results of this research suggest that additional information may be obtained about GSR particles from their internal composition and morphology. Observable compositional and morphological differences were noted between the ammunition types considered in this study, with at least one ammunition – Norinco – generating particles that had a highly distinctive internal morphology. Therefore, in some circumstances, if there is enough evidential value to be obtained, it is possible that this type of analysis could add to an investigation. The usefulness of this approach will be contingent on further research into the variation of particle morphology and composition between ammunition types, and collaboration and knowledge sharing of the results. Ultimately, although it certainly seems possible that this technique could be used further to obtain additional information about all types of GSR, the practical considerations required for this technique mean that it is unlikely to be particularly useful in a routine case-work context.

Due to the limitations of the FIB system, not all particles are appropriate for individual crosssectioning and in order for a particle to be cross-sectioned, or to apply the lift-out technique, the particle must be accessible to allow the relatively complex manipulation required. This means that extraction and analysis can be hindered by the surface topography of the stub, the quantity, type, and size of other particles present, and how the individual particle is positioned on the stub. In a casework context, the analyst does not often have the ability to be selective with the particles that they need to analyse. Further, the time, cost, and complexity of the processes required mean that for most forensic laboratories, it would be impractical to conduct this analysis in a routine manner. While it appears possible that a FIB system can be used to thin and prepare a sample for further analysis using different analytical techniques, the success of this process is highly dependent on the sample matrix. Glass containing GSR (gGSR) appeared to be the most robust to the preparation.

The preceding chapters have addressed a number of factors that may influence source-level (or indeed, sub-source level) assessments. In the wider context of this thesis, understanding the significance of and differences between propositions evaluated at the source or the activity level are paramount to ensuring GSR is placed in its appropriate context. The ENFSI guideline on evaluative reporting states that,

'Source level propositions are adequate in cases where there is no risk that the court will misinterpret them in the context of the alleged activities in the case' [124]

In the case of GSR, purely evaluating evidence in the context of source level propositions has, resulted in some severe miscarriages of justice in situations where identifying the evidence as GSR in the absence of any contextual activity level evaluation has been determined to be prejudicial [199-201]. This suggests that further consideration of GSR evidence in the context of activity level propositions adds important value and context to the evidence, allowing it to be more appropriately considered by the court. Of course, in order for an activity level proposition to be considered, the forensic scientist must first evaluate the evidence in the context of source level propositions [202]. That is, it must first be determined that a trace is GSR before the significance of that finding can be evaluated at the activity level. The previous chapters have explored factors influencing source level interpretation, including the influence of things such as primer chemistry, projectile composition, and weapon memory effect, to support GSR analysts in more accurate source level assessments. However, equally important is supporting activity level assessments through more comprehensive studies on factors such as GSR background, potential for cross-contamination, and extent of transfer. These will be addressed in the following chapters.

Sub-Particle Composition and Morphology of GSR | Chapter 4

5. THE PRESENCE OF GSR IN THE RANDOM POPULATION

5.1. SYNOPSIS

Fundamentally, the value of a GSR test result of residues collected from the hands of a person of interest as a part of an investigation is linked to the GSR background in a population. The background prevalence of GSR particles directly informs the significance of a GSR test result. If the background prevalence of GSR in the random population were to be high, the value of detecting GSR in a forensic context is reduced, as it becomes difficult to separate the GSR background from GSR deposited as a consequence of the incident under investigation. However, if the background were to be low, this suggests that GSR particles are less frequently encountered in the random population, and therefore finding them on the hands of a person of interest is more significant.

Factors that may influence the GSR background of a region include the rate of private firearms ownership, popularity of firearms hobbies, and proportion of the population serving in the police or defence forces. Although some studies have been performed in Europe [50, 98], the difference in firearms environment in Australia means a detailed local study is more pertinent to GSR evidence assessments in Australia.

To assess the background prevalence, a multi-jurisdictional survey of the hands of random individuals was performed. Samples were collected by approaching subjects in public places, and a questionnaire was completed related to their personal firearms use, as well as their potential exposure to GSR via secondary transfer. Ultimately, 309 individuals comprising 120 from Victoria, 169 from South Australia participated in the study. Data from an earlier survey of 20 individuals from New South Wales was later used as a basis of comparison across states. Samples collected from all participating individuals were analysed in each state according to established GSR analysis protocols. From the results, it was ascertained that the frequency of particles characteristic of GSR in the random population was approximately 0.3%, with only 3 particles being detected on a single individual. With respect to consistent particle types, the most prevalent were PbSb and PbBa with an observed frequency of 3.8%, with the least frequently encountered particle type being SbBa at 1.4%. These results suggest that GSR particle types are still infrequently encountered in the random population, and therefore their evidential value is upheld. The results of this survey will inform a framework for the assessment of GSR evidence.

5.2. BACKGROUND

In the context of this thesis, the research from this chapter works to inform one of the nodes of a proposed Bayesian network for the assessment of GSR evidence. From the previously discussed framework, reproduced here in Figure 44, nodes representing various factors that can inform an assessment of GSR evidence can be seen.



Figure 44 - Proposed Bayesian Network for the analysis of GSR results from Biedermann, Bozza & Taroni [1]

One of the major considerations in the ultimate evaluation of GSR evidence requires an understanding of the background prevalence of GSR, corresponding to node B in Figure 44. This node specifically relates to the likelihood of observing GSR particles in a sample collected from the hands of a randomly selected member of the general public. If the background prevalence of particles indistinguishable from GSR in the random population is high, then the relative value of GSR evidence is weakened. High background prevalence means that GSR particles are commonly encountered in the wider environment, and therefore finding them on the hands of an individual when conducting a firearms investigation has less significance. Conversely, if the background prevalence of GSR particles in the random population is low, the opposite is true, which results in a relative increase in the significance of any GSR found as a part of a firearms or shooting investigation. The background of GSR in a population can be influenced by a number of factors. Prior research conducted by Ditrich [83] demonstrated that different firearms produce different patterns of distribution of GSR during firing, depending on the type and style of operation. These particles are then able to settle on the shooter [111], nearby surfaces [203], and any bystanders [88], as well as being retained in the firearm. It has previously been observed that GSR can pass between individuals and items through direct

contact in the form of secondary transfer [91], and tertiary transfer [92]. With these considerations in mind, the GSR background in the wider population is likely to be impacted by the firearms culture of the jurisdiction. That is, the greater the prevalence and use of firearms in a particular area, the more likely that a random individual will encounter a surface, individual, or object which may result in transfer. It could therefore be expected that the regions with a higher proportion of individuals with firearms hobbies (such as hunting or target shooting), higher private firearm ownership, or a greater number of individuals employed in the defence or police forces would exhibit a higher background of GSR. To investigate this, some studies have been conducted of targeted populations, assessing the GSR background in populations of hunters and their families [98], as well as in police populations [73, 99]. Police populations have attracted particular focus due to their involvement with firearms and shooting investigations, where the possibility of cross-contamination is a concern.

While the information collected pertaining to these targeted populations is of value in compiling a detailed picture of the GSR background, a broad study, investigating the prevalence of GSR in the random population has not been conducted. This study was designed to address a number of factors pertaining to the GSR background. First, direct sampling of the hands of individuals allowed for an assessment of the prevalence of characteristic and consistent GSR in the random population. Specifically, conducting a wide GSR population study for the first time in Australia will permit a further assessment of how frequently consistent GSR particles are encountered. The fact that two-component, consistent GSR has sources other than firearms discharge has been well documented. However, the prevalence of these particles is still worthy of a robust assessment. This is particularly the case in Australia where 0.22LR rimfire ammunitions are more prevalent. The survey will also provide the opportunity for the collection of demographic data from the Australian population, including a reflection of the number of individuals who report that they have firearms hobbies, or other occupational firearms exposures. Of further consideration will be the potential for secondary transfers due to contact with family members and the home environment. All of this information can be used to inform assessments of the significance of GSR evidence.

The research within this chapter addresses a multi-jurisdictional survey of the random prevalence of GSR in the Australian context. GSR hand samples were collected in Victoria and South Australia, with a small number of additional samples from New South Wales used as a point of comparison.

Elements of the research conducted in this chapter were peer-reviewed and published. This first appeared in:

N. Lucas, H. Brown, M. Cook, K. Redman, T. Condon, H. Wrobel, K.P. Kirkbride, H. Kobus, A study into the distribution of gunshot residue particles in the random population, *Forensic Science International* **2016**, 262, pp. 150-155.

For the purposes of this publication, the approximate contribution of each author was N. Lucas 30%, H. Brown 10%, M. Cook 10%, K. Redman 10%, T. Condon 10%, H. Wrobel 10%, K. P. Kirkbride 10%, H. Kobus 10%.

The full text of the publication has been incorporated in section 5.3 below.

Please note – Minor formatting amendments have been performed to the presentation of the publication to keep it consistent with the presentation of the thesis, however text and data remain unchanged from the published version.

5.3. A STUDY INTO THE DISTRIBUTION OF GUNSHOT RESIDUE PARTICLES IN THE RANDOM POPULATION

5.3.1. Introduction

When considering the impact and value of gunshot residues (GSR) as forensic trace evidence, the likelihood of a suspect producing a positive GSR analysis result without having direct exposure to a firearm is a major consideration. Therefore, the random prevalence of GSR and 'GSR-like' residues in the wider population is a highly pertinent question when considering the probative value of such evidence. This raises the possibility of applying a probabilistic interpretation to GSR results. Biedermann et al. ([148, 150]) have proposed a Bayesian approach to GSR evidence. They state in order to evaluate competing hypotheses (usually that a person has discharged a firearm and that the person has not) two important factors must be addressed - (i) the number of GSR particles expected and (ii) the probability of random occurrence of GSR. The first factor is difficult to address, as it depends on a number of variables, including firearm and ammunition. However, tests with the questioned weapon and ammunition can be undertaken for each case situation. The second question can be addressed by undertaking random GSR sampling in the general population.

Previous population studies, conducted in Europe (Italy [50] and Poland [98]), are most applicable to the countries in which they were conducted. Differences in firearms laws and availability in different countries may be expected to result in differences in the background prevalence of GSR in the wider population. This may be considered of particular relevance in Australia, where a comparatively greater proportion of firearms encountered in casework are of the 0.22 rimfire variety or 12 gauge shotguns. GSR originating from ammunitions of this type may present significant challenges in the identification of detected particles: many 0.22 calibre rimfire primers produce PbBa residues and many 12 gauge ammunitions produce predominantly BaAl particles with relatively few 3-component particles being detected. With that in mind, a survey specific to the Australian environment is warranted in order to accurately assess the prevalence of GSR and 'GSR-like' particles at random in Australia.

5.3.2. Materials and methods

Research reported in this paper

This survey was an initiative carried out on behalf of the Criminalistics Specialist Advisory Group (SAG) of the Senior Managers of Australian and New Zealand Forensic Laboratories (SMANZFL).

Sample collection and analysis was carried out independently in two states, Victoria and South Australia, by accredited forensic science laboratories - the Victoria Police Forensic Services Centre (VPFSC) and Forensic Science SA (FSSA).

The initial results from 41 of the 120 subjects sampled in Victoria were reported by Condon in Honours work conducted at Deakin University.

Subject selection

Samples were collected from volunteer members of the public at two different geographical locations in Victoria, a metropolitan shopping centre (Preston) and a regional market (Geelong), and nine metropolitan locations in Adelaide. Appropriate ethics and privacy approvals were obtained. Questionnaires were used to establish, amongst other information, whether the volunteer had any association with firearms. Minor differences existed between the questionnaires used in the two states, but the same relevant information was collected. Both questionnaires requested the age group and hand preference of volunteers, as well as asking them to disclose if they had handled or fired a firearm in the last 5 (Victoria) or 4 (South Australia) hours, if they had any hobbies related to firearms, and when they had last washed their hands. Both questionnaires asked if volunteers worked in professions that may result in GSR or 'GSR-like' particle exposure (i.e., police, military, pyrotechnic technicians, building, automotive or agricultural industries). However the Victorian questionnaire required volunteers to nominate if they were a member of a list of specified professions, while the South Australian questionnaire asked volunteers to declare their profession.

The Victorian survey further asked if the volunteer worked with or regularly handled metals that may be present in GSR (i.e., Pb, Ba, Sb, Zn, Cu, Si, and Al) or if members of the volunteer's household worked in high exposure risk industries, or had firearms hobbies. The South Australian survey asked volunteers to nominate what they had been doing with their hands for the last 4 hours. Anonymity of participants was ensured. Samples were collected from a total of 289 subjects - 120 in Victoria and 169 in South Australia.

Sample Collection

Sample collection was performed using each laboratory's documented GSR procedures, which ensured that the survey replicated the way in which case samples would be collected. Samples were collected on standard 12.5mm SEM pin stubs held in closed plastic vials. The stubs used in Victoria were covered with transparent double sided adhesive tape and those used in South Australia had double sided carbon adhesive tape.

There were minor variations in the sampling procedure used in the two states. In Victoria, a separate stub was used for each hand and more extensive dabbing conducted over the top of the thumb, forefinger and web, followed by the palm and underneath the thumb and forefingers. In South Australia, one stub was used to sample both hands, starting with the volunteer's nominated favoured hand. After removing the paper backing to expose the adhesive surface, the stub was dabbed around the forefinger, followed by the webbing and then the thumb. Following sampling the stubs were returned to the vials.

Samples were carbon coated prior to analysis by SEM-EDS using automated particle searching software.

Instrumentation and Analysis

The Victorian instrument was a CamScan Apollo SEM with Genesis EDS system. The South Australian instrument was a Zeiss Evo 50 SEM with Oxford EDS system.

Each laboratory used different set up and monitoring procedures, but all analyses were performed in accordance with each institution's validated, documented and accredited procedures for GSR analysis, ensuring that samples were exposed to the same conditions that case-samples would experience. This included measures to prevent laboratory contamination.

VPFSC: The backscatter signal set-up standard was gold, niobium, germanium, silicon, carbon (Au, Nb, Ge, Si, C) and Faraday cup. (Eastern analytical).The positive control was prepared in house by sampling the back of a hand immediately after the discharge of a Smith and Wesson 0.38 revolver.

FSSA: A Gold/Cobalt/Rhodium/Carbon (Au/Co/Rh/C) standard (Micro-Analysis Consultants Ltd, United Kingdom) was used to set up brightness and contrast settings to allow for the automated particle search system to detect particles based on the signal generated from the backscatter detector. A SPS-Synthetic Particle Specimen (Plano GmbH, SPS-A521-2(27C)) standard was used as a positive control at the start and end of every automated run. It is a silicon chip with 43 precipitated PbSbBa particles, ranging in size from 1µm to 5µm. For a valid analysis, the automated particle search system had to identify 40 out of 43 particles at both the start and end of the run.

At the end of each automated run, particles classified as GSR were manually reviewed by an analyst in order to assess their morphology and composition before being excluded or confirmed as GSR. Although data acquisition conditions were not identical across the two laboratories, both instruments were capable of reliably detecting gunshot residue particles down to a size of 0.5µm and, therefore, their performance can be considered to be equivalent for all practical purposes in regards to this survey.

5.3.3. Results and discussion

The following particle classification, based upon the ASTM standards [36] and protocols of the forensic laboratories that carried out the analysis, was used:

- The three element combination (PbSbBa) was considered characteristic of a GSR origin.
- Two element combinations (e.g., PbSb, PbBa, SbBa) were considered consistent with a GSR origin.
- Au, Fe, LaCe, etc. were considered common environmental particles.

Data from subject information sheets revealed that six of the subjects in Victoria (5.0% of Victorian subjects) declared personal firearms hobbies – including hunting, sports shooting, or membership of a rifle/pistol club. With regard to occupational exposure to GSR, four of the sampled subjects (3.3%) worked in an industry with potential GSR exposure (one a police officer and three subjects listing their industry as hunting/shooting). Twelve subjects (10%) declared a member of their household participated in a firearm related hobby and/or worked in an industry with exposure to firearms, including one bullet factory worker. Of the 120 subjects sampled in Victoria, 16 (13.3%) of subjects declared a potential exposure to GSR particles through a combination of hobbies, employment or members of their household. All subjects declared they had not fired a firearm within the preceding five hours.

South Australian subject information sheets revealed 13 subjects (7.7% of South Australian subjects) with nominated firearms hobbies, one subject (0.6%) declared a potential occupational exposure to GSR (a member of the Australian Air Force reserve), and one (0.6%) subject declared they had handled, but not fired, a firearm in the previous four hours. Of the 169 South Australian subjects, 15 (8.3%) declared a potential exposure to GSR particles through hobbies, occupational exposure or handling a firearm.

Considering the samples collected from all 289 subjects across both jurisdictions, 32 (11.1%) subjects declared they or a member of their household had some association with firearms and therefore exhibited the potential to be contaminated with GSR.

Characteristic and Consistent Particles

PARTICLE TYPE	Frequency % of participants. Total (Vic/SA)	NO OF SUBJECTS TOTAL (VIC/SA)	Max. (No. of Particles) A	Min. (if DETECTED)A (NO. OF PARTICLES)	Avga (no. of particles)
PbSbBa	0.3% (0.0% / 0.6%)	1 (0/1)	3	3	3.0
PbSb	3.8% (0.0%/ 6.5%)	11 (0/11)	5	1	1.5
PbBa	3.8% (0.0% / 6.5%)	11 (0/11)	3	1	1.4
SbBa	1.4% (0.0%/ 2.4%)	4 (0/4)	1	1	1.0

Table 39 - Collated particle data for characteristic and consistent particles

^a Of participants with nominated particle type found.

Three subjects of those reported in table 1 in South Australia recorded simultaneous detection of PbSb and PbBa particles, while one subject recorded simultaneous detection of PbSb and SbBa particles. In each case particle numbers were low, (less than 5) and the subjects declared no firearms hobbies or exposure. This is of significance if a bulk analysis technique, such as neutron activation analysis (NAA) or atomic absorption spectroscopy (AAS), was to be used, as simultaneous detection of the above particle types may result in a false positive for GSR.

The most significant particles are the three characteristic particles found on one subject. This was in the South Australian survey, on a female subject with no declared firearms hobbies or contacts. The subject reported her occupation as a retail worker, and had spent the four hours prior to sampling at work. The sampling information sheet revealed she had washed her

hands 60 minutes prior to sampling. Two of the particles are large (~30 μ m) and the third smaller (~2 μ m). The large particles appear to be agglomerates of smaller particles, and it is possible the small particle is a fragment from one of the larger ones. While the morphology does not exclude the particles from consideration as GSR under the ASTM standard, the agglomerate morphology is not convincing of GSR origin. The X-ray spectrum shows clearly defined peaks for Pb, Sb, Ba together with Cl and Al - a GSR particle composition that is also not excluded under the ASTM Standard Guide as GSR. However, an abundance of sodium and chlorine in the particles, and their large size (~30 μ m) is atypical for GSR. Figure 1, below, shows the thumbnail image of one of the large particles and its associated X-ray spectrum as produced by the automated GSR search software.



Figure 45 – Backscatter image, elemental distribution X-ray map (Red – Sb, Green – Ba, Blue, Pb) and EDS spectrum of a large characteristic particle from the South Australian survey With regard to two component 'consistent' particles, PbSb and PbBa combinations occurred with comparable frequencies (both on 11 subjects and only in South Australia). The latter combination is relevant to GSR from 0.22 rimfire ammunition that does not contain Sb as a component of the primer. The PbSb combination is consistent with projectile derived particles as lead is often alloyed with a small amount of antimony to increase hardness [1]. The presence of Cu can enhance this identification as projectiles can be coated with copper wash.

There was no observable correlation between those individuals with declared firearms occupations or hobbies and detection of consistent GSR particles. In Victoria, of 16 subjects declaring possible exposure to potential sources of GSR, no particles characteristic of, or

consistent with, GSR were found. In South Australia, of 15 subjects declaring a potential firearms exposure, only one subject tested positive for two-component GSR particles.



Figure 46 - Subjects returning a positive GSR result grouped by declared GSR exposures (hobbies, occupation, or other) and by gender. "All GSRs" is the sum of characteristic and consistent particles detected.

As seen in figure 2, subjects declaring no GSR exposures returned a comparably larger number of positive GSR results. However, this may be attributable to a significant difference in the sample sizes (258 with no declared hobbies, 31 with declared hobbies). Male subjects returned a significantly higher number of positive results, despite comparable sample sizes (male n =153, female n =136).



Figure 47 - Subjects returning a positive GSR result grouped by age

Figure 3 shows the breakdown of positive GSR results grouped by age. Those in the group 25-39 returned a positive GSR analysis result more frequently than those in the 40-55 age group, despite the fact that the sample sizes were comparable (25-39 n = 93, 40-55 n = 91).



Figure 48 - Subjects returning a positive GSR result grouped by time since last handwashing.

Interestingly, as can be seen in figure 4, those who declared they had washed their hands within the last hour returned a comparatively larger number of positive results. However, the time since last hand washing was self-reported, and there was no way for researchers to accurately verify this interval.

PARTICLE TYPE	Frequency (% of participants.) Total (Vic/SA)	NO. SUBJECTS TOTAL (VIC/SA)	Max. (No. of Particles)	Min. (if detected) (No. of particles	Avga (no. of particles)
Pb	54.3%	157	6223	1	50.9
	(53% / 56%)	(63/ 94)			
Sb	32.2%	93	70	1	3.2
	(39% /27%)	(47 / 46)			
Ва	1.7%	5		5 1	
	$(4.2\% / n/a^*)$	(5 /	5		1.8
	(/././././	n/a*)			

Table 40 - Collated particle data for single element particles

Single Element Particles

^a Of participants with nominated particle type found.

*n.b. – Ba was not a particle classification in the South Australian survey.

There was extensive occurrence of particles containing Pb or Sb with large numbers on some individuals. For example, over 6,000 Pb particles were found on one subject in South Australia, a plumber. Another seven individuals had Pb particles ranging in number between 50 and 500. The most Sb particles on a single individual was 70 with another 5 subjects having between 10 and 50 particles on their samples. Thus it can be seen these single element particles are quite widespread in the random population and can be quite abundant on certain individuals.

In the absence of any two or three component particles, these particles would not be reported or considered in relation to GSR. However, of the subjects on which single element particles were detected, 57% (n= 13) exhibited simultaneous detection of two element particle combinations, including the subject reporting in excess of 6000 particles of lead. Under these conditions, single element particles would be recorded alongside two element combinations as consistent with a GSR origin.

Similar to the situation with the South Australian population, in the Victorian population, samples collected from three individuals were found to contain all three elements (Pb, Ba, and Sb) but only as single element particles. This again emphasises the importance of the capability of SEM-EDS in identifying particles that contain all the critical elements and demonstrates the strong possibility for false positive results when total elemental analysis techniques, such as AAS, are used.

Environmental Particles

Not surprisingly, large numbers of environmental particles were found in the survey, including jewellery (e.g., AuCu, Ag. Ni), pigments (e.g., BaS, Bi), mischmetal (e.g., lighter flint Ce, La, Fe), brass (CuZn), coinage (CuNi) and solder (PbSn). This agrees with casework experience.

In the Victorian survey, over 37,000 particles were found on 120 participants, with approximately 45% of them remaining unclassified ("unclassified" refers to particles which do not contain elements typically associated with firearm discharges and are therefore not classified by the automated process). The South Australian data classified over 15,500 particles across 169 participants.

5.3.4. Conclusions

This study has shown the three-component particles that are classified characteristic of GSR, are not common in the general population - the frequency measured approximates that arrived at by Brożek-Mucha [98] in her survey of selected populations of users and non-users of firearms. Of 289 subjects in this study, only one subject was detected with three-component particles on their hands. The three, three-component particles detected were unable to be excluded as GSR under the current ASTM definition, and would therefore have been reported as characteristic of GSR if they were to be found in case samples in the jurisdiction. However, the limited number of characteristic particles on the individual concerned, together with the absence of consistent particles on the subject would have led to a cautious interpretation of the evidential significance of these particles.

In regards to quantitative approaches to evidence evaluation, the overall frequency of threecomponent particles was 0.3%, indicating that while the coincidence frequency of characteristic particles in the random population used in this study is not zero, it is close to zero. As the likelihood ratio is quite sensitive to this coincidence frequency and inversely proportional to it, the detection of small numbers of three-component particles on suspects can still be valuable evidence if the case circumstances are appropriate [147]. There were sufficient two-component particles found in the random population to support the current, cautious approach in interpreting GSR results involving particles of this type. This is of particular relevance to incidents involving 0.22 calibre firearms, which are prevalent in Australia. Despite a number of subjects declaring an association with firearms either through work, hobbies or members of their household that may have increased their chances of exposure to GSR, there was no significant correlation between this group and the detection of GSR. The bulk of subjects on which consistent particles were detected declared no association with firearms.

The detection of three-component, characteristic particles on a female in the general population who declared no firearms exposures with recently washed hands was unexpected, and due to the nature of sample collection, their source cannot be definitively determined. If these particles are not of GSR origin, when evaluated in the context of other particles detected on this individual, there is no indication that the characteristic particles have originated from brake components, fireworks, or other previously described non-GSR sources. Further work aimed at establishing the source of the particles, including the use of focussed ion beam techniques following previously established protocols [204] will be carried out.

----- End of Publication-----

5.4. ADDITIONAL DATA

Although the publication addresses data collected primarily in Victoria and South Australia, a smaller sample set was collected by researchers in New South Wales. Data from an additional 20 sample stubs had been provided in an early stage of the project.

Ultimately, this sample set was excluded from the published data, based on a number of key considerations. First, the data that were provided originating from these additional samples was provided in a different format, with limited contextual information, making it impossible to verify that the same system of sample collection and analysis had been followed. No information was provided that detailed from where samples originated, or the particulars of the individuals from whom the samples had been collected. Additionally, it was unclear if the stubs represented samples collected from 20 different individuals, or from 10 individuals, with one stub used for each hand, or if a different sampling protocol had been followed. Finally, the form in which the data had been provided meant that it was impossible to extrapolate individual person frequencies from the data alone. For these reasons, these data were excluded from the data considered in the publication.

However, following publication, it was discovered that the data from the additional 20 sample stubs were originally analysed and reported by Hales as a component of her doctoral thesis [105]. From this, it was determined that the samples collected from these individuals had been collected from the hands of members of the general public at a shopping centre and a university, and had been analysed in accordance with established GSR analysis procedure in the New South Wales. In this case, four samples were collected from each individual, with a separate stub used for the front and back of both left and right hands. Data however were reported on a per subject frequency basis. While the differences were such that the NSW data was not incorporated into the Victorian and South Australian data set, the subject selection, sample collection and data analysis bore enough similarity to the existing study that the NSW data could be used as a point of comparison. To that end, the data collected from the NSW samples can be observed in Table 42, and a comparison of the particle type frequencies separated by state can be seen in Table 43.

Particle Type	Frequency % of participants. Total (NSW)	No of subjects ^{Total} (NSW)	Max. (No. of particles) ^a	Min. (if detected) ^a (No. of particles)	Avg ^a (no. of particles)	
CHARACTERISTIC						
PbSbBa	0.0%	0	0	0	0	
Consistent						
PbSb	30%	6	5	1	2.8	
PbBa	10%	2	1	1	1.5	
SbBa	0%	0	0	0	0	
SINGLE ELEMENT						
Pb	45%	9	5	1	1.7	
Sb	40%	8	5	1	2.6	
Ва	10%	2	2	1	1.5	

Table 41 - Collated particle data for characteristic, cor	sistent and single element particles from the
NSW survey conducted	by Hales [105].

From the NSW only data in Table 42, it is important to note that no characteristic particles were detected on any of the samples in this jurisdiction. This finding was initially reported by Hales [105], and further corroborates the findings from the Victorian and South Australian data, in suggesting that this particle type is rare in situations that a firearm has not been handled or discharged. The NSW data, however, exhibit some differences from what was observed in the other jurisdictions, with higher reported frequencies of two component particle types. This was most evident in the case of PbSb particles, which had a frequency of 3.8% in the wider sample set, but reported a 30% frequency in the samples collected in NSW. It was acknowledged that one subject indicated that they had performed activities that may have impacted results, in that they had recently checked the tire pressure of their car and filled their vehicle with leaded petrol. Although this individual did have particles considered consistent with GSR present on their hands, Hales concluded that they were not at a significantly higher level than the rest of the sample set [105]. Rigorous anti-crosscontamination measures were taken in the sampling, preparation and analysis stages of this research, suggesting that the chances of the increased particle numbers being a result of cross-contamination of the samples is highly unlikely. However, all of the samples were collected at the same location, presenting the possibility that these particle types were elevated within this specific environment. Regardless, in agreement with the data collected from the other states, although a larger percentage of individual samples tested in NSW had this particle type present, the numbers of particles observed were low. This data therefore continues to support the assessment that low numbers of consistent particles are not uncommon on the hands of members of the general public. However, significantly elevated

numbers, the appearance of multiple different particle types, or the appearance of consistent particles alongside characteristic particles are still of relevance to forensic GSR analysis.

When considering the single element classified particles, the frequencies reported in NSW are comparable with the rest of the data set, with the combined Victorian and South Australian data reporting frequencies for Pb, Sb, and Ba particles of 54%, 32% and 1.7% respectively. The NSW data follows this general trend, with 45% of the samples having Pb particles present, 40% having Sb particles, and 10% having Ba particles. The relative prevalence of these particle types is preserved in the NSW data set, when compared against the results reported in the combined Victoria and South Australian data. Particles of Pb were observed to be the most prevalent, occurring in almost 50% of the samples analysed in both cohorts, while Ba particles were relatively uncommon. That said, it must be acknowledged that the data collected was from a single location, and from a limited sample set (20 individuals). Therefore, it should not be used to draw specific conclusions about the jurisdiction in which they were collected, and should only be used in a broad sense.

As previously discussed, a large proportion of the samples from NSW had PbSb particles present. It is noteworthy that these were detected on the hands of 6 subjects from a pool of 20 individuals, compared to 11 subjects from 169 subjects in SA, and zero of 120 subjects in Victoria. A side by side comparison of the observed particle type frequencies in each state can be seen in Table 43. This further suggests that although the presence of consistent particle types on the hands of members of the public is not uncommon, the frequency of individual particles types may be contingent on population and location. Further, the NSW data was collected many years prior to the Victorian and SA data, suggesting that perhaps particle frequency may also have changed over time. These factors make extrapolating further meaning from individual particle classes difficult, which may impact the assessment of GSR originating from two-component primed ammunition.

Particle Type	Frequency % of participants (Vic) n=120	Frequency % of participants (SA) n=169	Frequency % of participants (NSW) n=20			
CHARACTERISTIC						
PbSbBa	0%	0.6%	0.0%			
CONSISTENT						
PbSb	0%	6.5%	30%			
PbBa	0%	6.5%	10%			
SbBa	0%	2.4%	0%			
SINGLE ELEMENT						
Pb	53%	56%	45%			
Sb	39%	27%	40%			
Ва	4.2%	_*	10%			

Table 42 - Comparison of observed particle frequencies separated by state

*n.b. – Ba was not a particle classification in the South Australian survey.

While in the original Victorian and SA data it was suggested that the overall frequency of both PbSb and PbBa particles was comparable, the addition of the NSW data suggests that PbSb is the most prevalent overall with PbBa not far behind. Overall, SbBa remains the least frequently observed particle class in the random population. With regard to the single element particles, viewed in isolation, the NSW data in Table 42 supported the particle distribution observed in Table 41. Pb remains the most prevalent single element particle type, followed by Sb and lastly Ba.

Overall, although the NSW data were not originally included in the sample set, it does provide some valuable further context with which to further evaluate the results of the survey. Specifically, it further supports the suggestion that the presence of three-component characteristic GSR is uncommon in the random population, confirming the utility of these particles in the assessment of firearm crime. Additionally, the results observed with regard to consistent particle types demonstrate differences between both the South Australian and Victorian data, suggesting that the prevalence of these particle types may differ in individual locations. Some general trends were observed, with PbSb particles being observed relatively frequently in the consistent class, and SbBa particles the least frequently observed. With regard to single element particles, Pb particles were the most commonly observed, and Ba particles the least.

5.5. CONCLUSIONS

The data represent the results of surveys conducted across three states of Australia, with residue samples collected from the hands of 309 members of the Australian general public. It represents the largest study of its kind conducted to date anywhere, not just Australia. Ultimately, the frequency of observing characteristic GSR on the hands of randomly selected members of the public was determined to be 0.3% of the population surveyed. The frequency of observing particles consistent with a firearm origin on the hands of the same individuals was determined to be 13.8%. Both of these findings support the understanding that characteristic GSR is uncommonly observed in situations not related to a firearms incident, and therefore maintains value as a form of trace evidence in the investigation of firearms crime.

A state-by-state consideration of the particle types observed indicates some trends in the data. There were significant differences between specific particle classes observed in the consistent category. This was most evident when considering PbSb particles, which had a 0% frequency in Victoria, 6.5% in South Australia, and 30% in NSW. This suggests that the incidence of these particle types may exhibit variations in prevalence between different environments and populations. This also supports the classification of these particle types in the consistent category, as originating from relatively common non-firearm sources.

It is worthy of note that all of the participants considered were approached in metropolitan or near-metropolitan areas. In Australia, the bulk of the population is resident in metropolitan areas, and therefore this background evaluation is most applicable for those residing within these regions. As in many countries, firearm ownership is more common in rural areas, where hunting and agricultural applications are more commonplace. It is therefore possible that the increased prevalence of firearms in these regions could translate into an elevated background of GSR on the hands of people resident in these regions. Although some previous research has been done in Europe in evaluating the GSR background specifically among groups of hunters [98], a further investigation of the GSR background in regional or rural Australian populations would further develop this picture.

When considering a Bayesian network for the assessment of GSR evidence, the results of this survey relate directly to the node 'B', representing the background content of GSR likely to be present on an individual's hands. This background node represents the probability of

observing GSR, or particles indistinguishable from GSR from any background sources. This includes any recreational or occupational exposures to GSR, as well as exposure to nonfirearm sources - including brake pads, fireworks, and cartridge operated industrial tools, among others. Further assessing the prevalence of 'GSR-like' particles originating from these sources will permit a more context-specific breakdown of the probability of observing GSR-like particles from these sources. Specific studies in pursuit of this goal have been conducted by Wolten et al. [48], Wallace and McQuillan [49], Garofano et al. [50], Torre et al [53], and more recently and specifically relevant to the Australian context, Tucker et al.[54] and Seyfang et al. [160]. These studies contribute useful data and speak to the frequency and probability of observing particles indistinguishable from GSR from a non-firearm source. These data may then be useful in GSR case evaluation when considering case-specific circumstances, such as evaluating a GSR result from the hands of a mechanic who may have GSR present alongside particles originating from brake pads. Practically however, the data from the current study provides an understanding of how frequently these particles appear in the random population. This can then be used to inform a general evaluation of a GSR result, where no specific exposures to sources of GSR similar particles exist.

6.GSR BACKGROUND IN THE POLICE POPULATION

6.1. SYNOPSIS

Beyond considering the GSR background in the random population, considering the GSR background on the hands of firearms-carrying police officers is also pertinent in the assessment of GSR evidence. If a large GSR population exists on the hands of serving police, this may represent a population that could be transferred to a person of interest through the process of apprehension or arrest.

A study performed by Cook [101] indicated that a significant population of GSR particles are transferred to the hands of police officers upon receipt of their firearms at start of shift. These deposits persist throughout the day, they may serve as a significant source of GSR particles that could undergo secondary transfer to other officers, surfaces, or persons of interest to an investigation. Further results in this field have shown significant variation, suggesting that jurisdictional specific studies will provide stronger, jurisdictionally relevant data [73, 98-100, 105].

In the work described in this chapter, police officers were sampled for GSR at random points throughout their shift. When compared to the random population, it was found that the GSR background on police officers was higher, with nearly 8% of the officers sampled returning at least one characteristic GSR particle. Further, 75% of officers had at least one particle considered consistent with GSR. Despite this, the overall particle counts were relatively small, with a maximum of 12 characteristic particles observed on one suspect. Considered as a whole, this suggests that although the GSR background on police is higher than the random population, the size of the particle population is only marginally larger.

Prior research has also considered the extent of secondary transfer that may occur between police and individuals under arrest situations [73, 205]. For the purposes of this survey, the extent of transfer from police was assessed under conditions of apprehension and arrest. As anticipated, the results showed significant variation, as particle transfer is strongly influenced by the amount and nature of the contact or activity. However, transfer experiments did indicate that the most frequently occurring amount of transfer being less than 25% of the available particle population. Taken together with the amount of background GSR observed on the hands of police, this suggests that in the event that secondary transfer to an arrested or detained individual does occur, the amount of potential transfer is likely to be low. This indicates that although the possibility of secondary transfer from law enforcement is worthy

of consideration, and taking steps towards mitigation, it is not a major or overwhelming concern.

Assessment of the composition of the types of ammunition routinely used by South Australia Police (SAPOL) was conducted to ascertain the types of residues they may generate. Operational ammunition was found to contain standard three-component primers, and produce a particle population that is consistent with a number of other ammunition products with this primer composition. With this in mind, there was nothing observable in the particle population that was distinct to GSR from SAPOL operational ammunition. Training ammunition however was a heavy metal free (HMF) variant that generated large numbers of particles containing Sr and SrAI. This finding was subsequently applied to case-work where the presence of an unexpected population of these particles identified a potential incidence of crosscontamination between an arrested individual and a member of a special operations unit of the police. To further assess this, the gloves of a number of officers from special units of the police were sampled, to ascertain the types of particles present. At the same time, other pyrotechnic devices to which they were routinely exposed, in the form of flashbang stun grenades, were also assessed to ascertain the particle types present. It was determined that flashbangs are capable of generating particles that are compositionally consistent with GSR, but also contain components that may indicate that they have originated from a non-firearms source. Numerous particles of all types - flashbang, standard primed ammunition, and HMF ammunition – were detected on the gloves of special units of the police, indicating that they present a significant risk of cross-contamination or transfer if involved in direct contact with a person of interest to an investigation. For this reason, it is important for the GSR analyst to be aware of the involvement of special unit officers in cases where GSR evidence is under assessment.

6.2. BACKGROUND

In the previous chapter, the GSR background in the random population was assessed as a means of evaluating the B node of a proposed Bayesian network for the assessment of GSR evidence. The proposed network is reproduced in Figure 44, below.



Figure 49 - Proposed Bayesian Network for the analysis of GSR results from Biedermann, Bozza & Taroni [1]

In the previous evaluation of the elements of node 'B', an assessment of the general GSR background in Australian metropolitan areas was performed. This informs the probability of observing GSR particles present on the hands of an individual selected at random from the population, even if they have not had a recent firearm association. A further factor that can inform this is the possibility of cross-contamination of the individual in question due to other contacts or exposures in the environment. It should be noted, that while the network in Figure 49 only indicates a node for the background content of GSR, it could be expanded to include contamination events, informed by the time and nature of the event as with node Y. It is possible that multiple contamination events could be incorporated, and therefore multiple nodes representing these events may be evaluated.

While a variety of possible contamination events may be pertinent to the assessment of GSR evidence, one of particular importance is the potential for GSR cross-contamination due to contact with police or law enforcement. This is particularly important, as while other cross-contamination events may occur to a POI in a firearms investigation, if they are to be sampled for GSR, it is certain that some contact with police or law enforcement will occur. Therefore,
understanding both the GSR background on the hands of police officers, and the potential and dynamics of transfer under the conditions of apprehension or arrest is valuable in assessing the probability of a significant amount of transfer occurring. While this is not a novel assertion, with numerous studies and surveys having been conducted of the levels of GSR on operational police and their work environment, significant variation in the results has been observed. A comparison and summary of figures obtained from literature can be seen in Table 44.

	AUSTRALIAN BACKGRO	UND	INTERNATIONAL BACKGROUND		
Ρομίζε Ομιτν Τύρε	% FREQUENCY		% FREQUENCY		
	(N POSITIVE SAMPLES / N	REF.	(N POSITIVE SAMPLES / N	REF.	
	TOTAL SAMPLES)		TOTAL SAMPLES)		
	0% (0/2)	[105]	7% (3/43)	[99]	
General Duties	050/ (20/22)	[101]	9.7% (3/31)	[98]	
	0570 (20/55)	[101]	60% (40/66)	[100]	
Special Operations	95% (18/19)	[105]	75% (18/24)	[73]	
			(PbSbBa Only)		
C (1)	50% (7/14)	[105]	25% (no data)	[104]	
CSI			· ·		

 Table 43 - Comparison of Police Officer Characteristic GSR background studies conducted in Australia and Internationally

From the literature, it can be seen that the GSR background on the hands of police varies significantly. This is likely to be influenced by the specific ways that firearms are handled and managed in different regions, as well as the specific duties that police are required to undertake. To support this, from Table 7, it can be seen that special operations units of the police, both in Australia and internationally report a significantly higher GSR background than general duties officers [73, 105]. This is not unexpected, as special operations units of the police typically undertake rigorous training with a number of different firearms, and therefore may be anticipated to have a larger GSR background than general duties officers. Similarly, the type of activities undertaken with firearms and the way that firearms are managed will impact the GSR background observed. Cook's survey of the hands of general duties police officers following receipt of their firearm at the start of shift shows significantly elevated particle counts [101] when compared to similar surveys of general duties police conducted in Europe during their shifts [98], and in the USA at the end of shift [99]. To that end, jurisdiction and region specific surveys that can assess the GSR background in the context of the local firearms environment are essential to constructing an accurate picture of GSR prevalence.

While a limited survey of Australian general duties officers was conducted in Australia by Hales [105], it seems clear that there is a need to expand this sample set to further increase the reliability of the observations. Additionally, although Cook's study also indicated that the elevated number of GSR particles deposited on the hands of general duties police was completely removed by following anti-contamination procedures, including handwashing [101], given the extent of the particle population observed, a follow up survey in the same jurisdiction is warranted. In the event that anti-contamination procedures are overlooked, there is the potential that these particles persist on the hands of general duties officers, and may then be further transferred to police vehicles, facilities, or other individuals in the process of apprehension or arrest. A survey of the hands of general duties officers at random points throughout their shift in the same jurisdiction will give a better understanding of the general GSR background on the hands of police officers in this region.

The GSR background present on the hands of serving police represents a particle population that may be a source of secondary transfer to other individuals or surfaces that that officer comes into contact with. However, this is only one factor that may impact the probability of GSR contamination to a specific individual through secondary or further transfer. It has been documented that GSR is able to undergo secondary and tertiary transfer to individuals that were not present during a firearm discharge through physical contact [91, 92]. Additional studies of particulate evidence have indicated that transfer through up to five individuals is possible [95]. One of the most critical situations to consider is the possibility of secondary transfer of GSR to the hands of an individual or POI to a shooting investigation during the process of apprehension or arrest. If GSR is transferred from the hands of a police officer, or from police vehicles or facilities in these circumstances, there is the possibility of a false positive error occurring, in that an individual returns a positive GSR test, when the GSR present is from a situation other than the incident under investigation. Under these circumstances, this may result in the focus of the investigation being diverted, or potentially a false conviction. In either case, this presents a challenge to the integrity and reliability of GSR evidence. To avoid this, both the extent of the GSR background on the hands of police officers, and the possibility and potential for transfer must be understood. To that end, modelling the type and extent of GSR transfer under 'worst case scenario' conditions will allow further understanding of the type and amount of transfer that may be expected. When combined with the GSR background on the hands of officers, this can be used to assess the likelihood of observing a significant amount of GSR transfer during arrest.

Beyond evaluating the likelihood of cross-contamination due to secondary or further transfer occurring, it is worth considering if it is possible for a GSR analyst to identify if such a transfer has occurred based on the nature of the GSR on the sample alone. Due to the difficulty in conclusively identifying the specific source of a particular population of GSR, it is difficult to determine if a cross-contamination event may have occurred. While research has informed specific circumstances in which contamination events may be more likely [73, 92, 105], this relies on the GSR analyst being familiar with the specific context and circumstances of the case. While this is an important consideration in any GSR evaluation, it still requires that the GSR analyst make an informed assessment of what may have occurred, without having any evidence or conclusive indication that cross-contamination has had an impact on the sample. In some jurisdictions, specifically marked or tagged ammunition is used to allow possible contamination to be readily identified. Some police forces in Europe, notably in Germany, use two HMF, tagged ammunitions that generate GdTiZn particles and GaCuSn particles respectively [13, 206]. These ammunitions have limited commercial availability and use, and therefore the presence of particles of these compositions on anyone other than police can serve as an indication that cross-contamination has occurred. Elsewhere, ytterbium and neodymium have been used to tag ammunition primers as a means of identifying the source of GSR [207], with some success. However, cost, reliability, and organisational reasons have limited the number of police forces that utilise this tagged ammunition for operational purposes [73]. Charles and Geusens indicated in their survey of special units of the police that the presence of TiZn particles consistent with Pb-free ammunitions may serve as an indicator of possible contamination from police [73]. Although these are not specifically tagged ammunitions, a number of police forces utilise HMF ammunition for training purposes, due to occupational health and safety concerns. The authors note that HMF ammunitions exhibit limited market penetration in Belgium [172], and the a similar situation is reflected in the Australian market. With this in mind, an assessment of the ammunition products used by SAPOL, in order to determine if there are any markers in either operational or training ammunition that maybe used in a similar fashion will assist in future assessment of GSR.

The research presented within this chapter addresses four core objectives.

- A survey of the hands of general duties police officers at random points throughout their shifts to better develop a picture of the GSR background on the hands of general duties police officers in South Australia.
- Performing transfer-modelling experiments in order to better understand to what extent GSR may be transferred from the hands of police officers to POIs during the process of apprehension or arrest.
- A survey of the ammunition products routinely used by SAPOL in order to determine their composition, and if there are any indicators that could suggest potential crosscontamination from police should they be observed in a GSR sample from a POI to an investigation.
- A survey of the equipment and other prominent sources of pyrotechnic residues used by special units of the police.

Elements of the research conducted in this chapter were peer-reviewed and published. This first appeared in:

Lucas, N.; Cook, M.; Kirkbride, KP.; Kobus, H., Gunshot Residue Background on Police Officers: Considerations for secondary transfer in GSR Evidence Evaluation. *Forensic Science International*, **2019**, 297, pp. 293-301

For the purposes of this publication, the approximate contribution of each author was: N. Lucas 60%, M. Cook 20%, K.P. Kirkbride 10%, H. Kobus 10%. The full text of the publication has been incorporated in section 6.3 below.

Please note – Minor formatting amendments have been performed to the presentation of the publication to keep it consistent with the presentation of the thesis, however text and data remain unchanged from the published version.

6.3. GUNSHOT RESIDUE BACKGROUND ON POLICE OFFICERS: CONSIDERATIONS FOR SECONDARY TRANSFER IN GSR EVIDENCE EVALUATION

6.3.1. Introduction

Gunshot residue (GSR) is a form of trace evidence which contributes to the investigation of firearms crime. GSR consists of particles generated from the ammunition primer, propellant, projectile and the firearm itself, which deposit in the immediate aftermath of a firearm discharge [11],[16]. These particles settle in the environment around the firearm, including on the hands of the shooter, the victim, and any other surfaces in the immediate vicinity [83],[81]

In order for GSR evidence to be given appropriate consideration in court, some assessment of the significance of the finding must be undertaken. A major factor influencing this assessment is the likelihood that the GSR particles present on a person of interest (POI) are truly associated with the firing event under investigation, or whether the particles are present due to other factors, such as secondary transfer or even tertiary transfer. Research has indicated that GSR particles may be present on the hands of a POI for a number of reasons unrelated to discharging a firearm directly. These prior studies have indicated that particles may be present on the hands and clothing of people that were bystanders to a firearm discharge [88], entered a scene shortly after a firearm discharge [82], or had handled a recently fired firearm [91]. Further, modelling of particle transfer has suggested that both secondary and tertiary transfer of GSR particles through physical contact, is possible [91],[92]. To ensure that GSR evidence is given appropriate weighting in court, the possibility that a person who is unrelated to the incident under investigation returning a positive GSR test result must be understood. This is analogous to assessing the possibility of a type I, or false positive error, occurring and is of particular concern if the evidence is to be considered reliable and not misleading to the court.

One particular concern about the potential for false positive errors is the possibility that a POI may have GSR on them as a result of their contact with police. Due to the fact that police officers routinely carry a firearm as a requirement of their work, there exists the possibility that they may have a high background level of GSR on their person, which may then pass to

POIs through contact with officers or subsequently in police custody. Studies conducted into the amount of GSR present on police officers, or police facilities and vehicles being a source of GSR contamination, have shown varied results, likely owing to differences in sampling methodology and jurisdictional variation. A study conducted by Berk et al. [102] indicated that the police vehicles and facilities in their study had a low, but possible likelihood of retaining GSR that has the potential to be passed to POIs. In the Berk et al. study, 201 samples were collected from surfaces and restraining bars in police facilities, as well as from police vehicles. Analysis of these 201 samples indicated that 178 of the samples (89%) were free of characteristic GSR particles. The remaining samples returned 56 characteristic particles between them. In total, 34 of these particles (61%) were collected from table-like surfaces within police facilities, with 20 particles (36%) collected from restraining bars used to secure suspects. The remaining 2 particles were found in tactical vehicles. A similar study conducted by Ali et al. [208] surveyed the presence of both the organic and inorganic components of GSR, originating from the propellant and the primer respectively, in police facilities. This study collected seventy samples from a variety of locations in four police stations. A single sample (1.4%) had characteristic GSR present with a further 7 samples (10%) reported as having at least one consistent particle present. The authors note however that only a single characteristic particle was detected, and was therefore deemed to be below the reporting limit for this study. Similarly, the maximum number of consistent particles present was 3 particles on one sample, with all others reporting a single particle of consistent GSR. With regard to the organic components, the only component detected on the test samples out of the six components considered was ethylcentralite (EC). The authors report that EC was present in a majority of samples, but was detected at a quantifiable concentration in only two samples (2.9%). No samples were observed to have both organic and inorganic components present on them simultaneously. This led the authors to conclude although the possibility of secondary transfer of both components from contact with police facilities exists, the possibility of transfer is remote. Conversely, a study conducted in Sweden conducted by Pettersson indicated much higher levels of GSR contamination present in police vehicles, with 1 in 4 of the vehicles tested returning a significant positive result [104]. Police in different jurisdictions have different firearms, protocols and systems of operation, as well as being exposed to regionallyspecific circumstances which may influence findings. As a result, while findings from these studies provide useful context for the possibility of a POI that is transported or detained being exposed to GSR, their results are not easily comparable. As a result, a more consistent and jurisdictionally-specific approach is required.

GSR contamination originating from police facilities and vehicles is not the only consideration that is relevant in assessing the possibility of secondary contamination of POIs. Research in multiple jurisdictions has indicated that there is a possibility of police officers carrying GSR contamination on their person. As to whether potential sources of contamination actually result in contamination, Charles and Guesens [73] performed a variety of mock arrest scenarios, under both 'high contamination' and 'low contamination' situations. The low contamination scenario involved police officers loading their firearm, before mock arresting, restraining and frisking a POI for a period of five minutes. The high contamination scenario involved police officers wearing a tactical vest, bullet proof vest, and gloves used in firearms training, then loading their firearm and simulating arrest and restraint of a POI in the low contamination scenario. In each instance, POIs were dressed in single-use Tyvek coats. At the conclusion of the scenario, the hands of both the POI and the police officers were sampled for GSR, and the Tyvek coats were seized for future sampling. To provide a basis for comparison for any GSR detected on the hands or clothes, primer from the ammunitions used by the police officers was sampled from fired cartridge cases and recovered to a GSR stub for analysis. Three ammunition types were of relevance to this study. Two ammunitions used for training purposes which had Pb-free primers - one containing Ti and Zn components in the primer, and one with Si, S and K components. The third ammunition was operational ammunition with a three-component PbSbBa based primer. That research indicated that transfer did take place to a level that could not be ignored. In the low contamination scenario, results indicated that in 25% of the cases modelled, GSR cross-contamination was detected on the officer's hands, and both the target's hands and clothing. In 33% of the modelled cases, there was GSR on the officer's hands, and the POIs clothing, but not their hands. A further 25% of cases showed no GSR present on the target at all. In the high contamination scenario, 58% of samples showed contamination of the police officers' gloves, and both the target's hands and clothing, while 42% of samples showed contamination of the officer's hands, and the target's clothing, with minimal or no contamination of the target's hands. In each case, both TiZn and PbSbBa particles were observed, indicating that contamination remains from both training and operational situations. Taken together, this indicates that in this study, the clothing of the POIs was more contaminated than their hands on average. Further, TiZn particles were observed more frequently than PbSbBa particles, indicating that the participating officers were more routinely exposed to GSR from their training ammunition than from operational ammunition [73]. The authors note that as the prevalence of Pb-free

primed ammunition is comparatively low in criminal shootings, the presence of these particles on a person of interest may serve as an indication for potential police cross-contamination. However, the researchers acknowledge that this sample set involved special units of the police that were known to be highly contaminated due to the nature of their intense training. That research specifically highlighted that GSR is likely to persist on gloves and equipment used in firearms training [73]. The types of arrest that special units are involved in are unusual events, as these units tend to be deployed in critical situations. Although an assessment of the GSR contamination on these units is valuable, it does not represent a general rule by which the whole of the population of police officers and their activities can be assessed.

When considering general duty police officers who do not frequently fire a gun, other studies have been conducted to assess the extent of contamination present in this broader population. Gialamas et al. [99] assessed the number of characteristic particles present on the hands of firearms-carrying, but non-firing police officers at the end of their rostered shift. In that survey, it was found that nearly 60% of the officers sampled had no GSR collected from their hands, and although a small number (7%) of officers sampled did return a positive, they had no more than one characteristic particle present. The remainder had small numbers of particles consistent with GSR on their hands [99]. Conversely, Cook [101] recently reported that the receipt of firearms at the start of shift can result in significant contamination of the hands of officers. Of the 33 officers that were sampled for GSR contamination in that study, 22 (66%) recorded at least one characteristic particle, with 6 officers (18%) having more than 100 characteristic particles present. That same study indicated that this contamination could be addressed through the washing of hands or the use of self-drying hand gel, with particle numbers dropping significantly after use. However, small amounts of GSR were observed to persist beyond hand washing. That study indicates that although procedures exist to minimise the extent of GSR contamination on the hands of police officers, there is still a chance that it occurs.

This article performs an assessment of the GSR background that may be present on the hands of non-firing, but firearms-carrying police officers in their day to day duties. The research was performed in two stages in the same jurisdiction (South Australia) as the Cook study [1]. The first was a survey of firearms-carrying police officers, to determine how much GSR was present on their hands at a randomly selected point throughout their shifts. The previous research performed by Cook [101] indicates that the receipt of firearms at the beginning of shift represents a contamination event that could potentially increase the background level of GSR present on the hands of officers. If a high background GSR level exists, this provides an increased population of GSR particles that could then possibly be transferred to the hands of a POI during the process of arrest. However, while a quantity of GSR may be deposited at the beginning of their shifts, washing of hands, time and activities undertaken during their day-to-day duties would reduce the amount of GSR present, possibly to a level comparable to the random population [209]. To that end, first stage of this study aimed to assess the background level of GSR present on the hands of on-duty police officers. The second stage of this study was to model the extent of GSR transfer during the process of arrest. If the GSR background present on police represents the population of particles that has the potential to be transferred to a suspect, these transfer experiments were designed to assess what proportion of that population is likely to be transferred. Taken together, these data will inform the possibility of cross-contamination between police and POIs in situations where a firearm has not been discharged, but the arrest is performed by firearms-carrying police officers.

6.3.2. Materials and methods

Sample Collection

All samples used in this study were collected from the hands of volunteer South Australia Police (SAPOL) officers serving in various capacities in Adelaide, South Australia.

All sample collection was performed using 12.5mm diameter aluminium SEM pin stub coated in double sided carbon-tape adhesive (Tri-Tech Forensics Inc. North Carolina, USA). In each instance of sample collection, the stub was dabbed directly on the surface of the subject's hands until the adhesive was exhausted, which was after approximately 50 dabs.

The standard issue, duty firearm for serving officers is the Smith and Wesson Military and Police (M&P) .40 semi-automatic hand gun. The operational ammunition used with this firearm is .40 S&W Federal Premium Law Enforcement Tactical, 165 grain HST ammunition, and is known to contain lead, barium and antimony in the primer mix [210]. The HST ammunition uses a nickel-plated brass cartridge, and has a semi-copper jacketed, lead core, hollow point projectile.

Police Officer Hand Background Survey

GSR samples were collected from SAPOL officers during their attendance at routine training courses held at a central police patrol base. All officers involved in this study routinely carry a firearm as a part of their work duties, and all officers had received a firearm, following the procedure detailed by Cook [101], between 1 and 12 hours prior to sample collection.

Volunteers were asked to fill in a short survey prior to their participation, which provided information including:

- Time since last handling a firearm
- Time since last discharging a firearm
- Time since last hand-washing
- Details of any hobbies related to firearms
- Nominated dominant hand

In this context, 'handling a firearm' was defined as anything that officers completed in the execution of their duties that involved contact with a firearm, including handling their standard-issue sidearm, handling firearms exhibits, or any other contact with a firearm. Most

commonly, this represented the procedure for checking, loading and securing their firearm prior at the start of their shift, described in detail by Cook [101].

A standard GSR sample stub was used to sample hands directly. A single stub was used to sample both hands. The sample stub was dabbed across the surface of the hands, starting with the nominated dominant hand. In total, 76 officers contributed samples to this portion of the study.

GSR Transfer Studies

GSR transfer studies were conducted using Police cadet volunteers attending routine firearms training at the police training facility. All participants were instructed to wash their hands thoroughly, ensuring coverage of both the front and back of the hands, and then dry them completely. The product used to perform the handwashing was 'D-Lead' Dry or wet skin cleaner with abrasives (Esca Tech Inc. USA), a product designed specifically to remove metal traces from the hands.

The pool of volunteer officers was divided into pairs, with one volunteer being designated the 'officer' and one the 'POI'. A blank sample was collected from the hands of the 'POIs' prior to further involvement in the study. The 'officers' were physically separated from the POI group, and issued their standard duty firearm. They then entered the firing range and each participant discharged two rounds of operational ammunition. They were then instructed to leave the firing range and mock-arrest the 'POIs'. The mock arrest involved making physical contact with the POI, placing them in a wrist lock, and then restraining their hands behind their back. The POIs were instructed to offer resistance to their apprehension, and continual contact was maintained until their hands were sampled. Physical contact between the pair was maintained for a period of 5 minutes. At the conclusion of this period, the hands of both hands, starting with the nominated dominant hand. In all cases, the 'POI' member of the pair was sampled first. Samples were labelled and coded as a set so it was clear which officer had apprehended which POI. In total, 42 officers, separated into 21 pairs, participated in this phase of the study.

Equipment

All collected samples were analysed by SEM-EDS using an Inspect F50 Scanning Electron Microscope (FEI Inc., Oregon, USA) equipped with an EDS detector (EDAX Inc., New Jersey, USA.) in accordance with established operational procedure. Instrument operating parameters were established as per the ASTM E1588-17 Standard guide for gunshot residue analysis by SEM/EDS [153]. Automated particle search was conducted using the GSR Magnum Particle analysis system (FEI Inc., Oregon USA), and TEAM elemental analysis software (EDAX Inc., New Jersey, USA). Further operating parameters for the SEM-EDS system can be seen in Table 45.

The GSR Magnum system brightness and contrast settings were calibrated through use of a Gold/Niobium/Germanium/Silicon/Carbon (Au/Nb/Ge/Si/C) standard (Ardennes Analytique, sprl, Belgium).

A positive control for the FEI system, a synthetic particle standard (PLANO W. Plannet GmbH, Wetzlar, Germany,) consisting of accurately deposited particles of known size was analysed at the start and end of every sample run. The 'particles' deposited on the glassy carbon surface of this standard are thin PbBaSb films of sizes 0.5-10 μ m diameter.

PARAMETER	Setting
Accelerating Voltage	25kV
Working Distance	10mm
Emission Current	~110µA
Magnification	486x
Min. Particle Size	0.5µm
Dwell Time	10µs

Table 44 - Set-up and Operating Conditions for SEM-EDS analysis

Data Analysis

ASTM E1588-17 was used as a basis to classify detected particles as either 'characteristic' or 'consistent with' a firearms origin.

Particles deemed 'characteristic' were those with compositions containing PbBaSb or PbBaCaSiSn. Particles reported as 'consistent' particle types included PbBaCaSi, BaCaSi, SbBa, PbBa, BaAl, Pb, Sb and Ba. Morphologically, particles falling into these classifications are noncrystalline, tend to be spheroidal, and range between 0.5 µm and 5 µm in diameter. In situations where 'All GSRs' are reported, this value is the sum of the 'characteristic' and 'consistent' categories.

As this study was primarily concerned with ammunition known to use a three-component primer, "lead-free/non-toxic" particle types were not considered for this study.

All particles classified as characteristic GSR by the automated software were reacquired and had their classification manually reviewed prior to reporting of the overall data.

6.3.3. Results and Discussion

Police Officer Background Study

Survey Responses

Based on the subject surveys collected prior to sampling the following demographic data were obtained.

	N	%
<1 month	4	5.3
1-3 months	20	26.3
3-6 months	17	22.4
6+ months	31	40.8
No Data	4	5.3
TOTAL	76	100

Table 45 - Self-reported time since last firing a firearm based on 76 surveys

n.b. Figures total to more than 100% due to rounding

As can be seen from Table 46, approximately 40% of responses indicated that the officer had last fired a firearm more than 6 months prior to sampling. In most cases, responses indicated that this coincided with the last routine firearm re-qualification that they took part in. The shortest interval between firing a firearm and sampling was 18.5 hours. The longest reported interval was 18 months. Four subjects presented incomplete surveys with no answer in this category and therefore were unable to be classified based on time since last firearm discharge. However, it is known that discharge of a firearm is not the only mechanism by which a person might have GSR present on their person. To that end, the questionnaire also requested data regarding the interval between last handling a firearm and the GSR Sample collection. These data can be seen in Figure 50.



Figure 50 - Time between last handling a firearm and GSR sampling. (n=75)

From Figure 50, it can be seen that the majority of subjects had last handled a firearm between 1-3 hours immediately prior to sampling, with nearly 65% of those sampled falling in this interval. In most cases, this corresponded with the start of their shift. Approximately 16% of samples were collected from officers less than one hour after they had received their firearm. The shortest interval between handling a firearm and sampling was approximately 5 minutes. The longest interval was reported as 12 hours. One subject provided an incomplete survey, and therefore was not included in this data set. As it is known that GSR present on the hands can be removed through handwashing, it was pertinent to note when subjects in this study had last washed their hands prior to a GSR sample being collected. The data from these survey responses can be seen in Table 3.

WASHED HANDS	Ν	%
<30 min	0	0.0
30 min - 1hr	17	22.4
1-2hrs	29	38.2
2-3hrs	16	21.1
>3hrs	13	17.1
No data	1	1.3
ΤΟΤΑΙ	76	100.1

Table 46 - Self-reported time since last hand-washing based on 76 surveys

n.b. Figures total to more than 100% due to rounding

It was expected that respondents who reported washing their hands after handling a firearm would exhibit lower levels of GSR. To that end, data were separated based on those respondents who reported that they had most recently handled a firearm (i.e., after they last washed their hands), and those who most recently washed their hands, (i.e., after they last handled a firearm). When comparing the data sets for washing hands and handling firearms, approximately 54% of subjects reported that they had received or handled a firearm after they last washed their hands, while 46% reported that they had washed their hands since they had received or handled a firearm.

	N (TOTAL)	N (GSR Positive)	Frequency (%)	TOTAL PARTICLES	Median (No. of Particles)	Mean (No. of Particles)	SD
Handled firearm after washing hands	41	31	75.1	788	11	19	21.4
Washed hands after handling firearm	35	34	97.1	252	5	7	6.9

Table 47 - Survey data separated based on last contact with a firearm and hand washing.

As can be seen in Table 48, both groups have a high number of individuals returning samples positive for GSR. However, the overall number of particles, and median number of particles detected are higher in the group that handled their firearm after they had washed their hands. This is perhaps unsurprising, as it is well documented that thorough handwashing will remove GSR particles present on the hands [106, 112]. The group that reported that they had washed their hands after last handling their firearm still produced samples that had GSR present, but moreover only one individual out of 35 did not have GSR present. This suggests either particles are persisting beyond reported hand-washing, or there are low-level re-exposures to GSR, either from surfaces, or the officers' own clothing, that have occurred between hand-washing and sampling.

Interestingly, when considering further survey data, those reporting firearm hobbies amounted to approximately 7% of the overall sample set. These hobbies were self-declared as target shooting (n=2), hunting (n=2) and farming and agricultural uses (n=1). This is marginally lower than the reported frequency of firearms hobbies observed in the general population, as reported in previous studies [209]. This indicates that despite the requirement to carry a firearm as a part of their job, police officers considered in this research did not have a higher incidence of non-occupational firearms exposure when compared to the broader population. This then suggests that GSR detected on their hands as a part of this study is more likely to originate from their occupational exposure, rather than private exposure.

GSR Results

The collected particle data from all samples collected from the hands of police officers can be seen in Table 49.

		NUMBER OF				
	FREQUENCY	PARTICIPANTS				
	(%)	(N)	ΜΑΧ	MEDIAN	MEAN	SD
		CHARACT	TERISTIC			
PbSbBa	7.9	6	12	0	0.2	1.4
		Consis	STENT			
BaSb	52.6	40	14	1	1.5	2.6
PbSb	17.1	13	6	0	0.4	1.1
PbBa	7.9	6	13	0	0.3	1.5
BaCaSi	29.0	22	67	0	1.4	7.8
BaAl	19.7	15	2	0	0.2	0.4
		SINGLE E	LEMENT			
Pb	56.6	43	60	1	3.2	8.2
Ва	61.8	47	9	1	1.5	1.8
Sb	40.8	31	39	0	1.6	4.9

Table 48 - Collected GSR particle data for samples collected from police officers' hands (n=76)

Ultimately, one of the major considerations for this survey was to compare the background level of GSR in a police population against the background level of GSR observed in the random population. If the background level in the police population is many times higher than the background in the random population, then this represents a source of particles that could potentially be transferred during contact between. Previous research has reported the results of our random population survey [209]. Direct comparison of the two data sets can be seen in Figure 51 and Table 50.



Figure 51 - Comparison plot of Characteristic and Consistent particle types in the Random and Police population.

When considering a more detailed breakdown of the data obtained in each study, the individual particle types detected in each study were compared. It should be noted that the previous random prevalence study was conducted under a more limited classification scheme, and only considered three consistent particle types [209]. The collected data can be seen in Table 506.

						MEANA (NO. OF	SD ^ª (No. of
		FREQUENCY	NO. OF SUBJECTS	ΜΑΧ	MIN	PARTICLES)	PARTICLES)
Dandam	PbBaSb	0.3	1	3	3	3.0	-
Kanuom (n-200)	PbSb	3.8	11	5	1	1.5	0.4
(N=289) PbBa [209] SbBa	PbBa	3.8	11	3	1	1.4	0.3
	SbBa	1.4	4	1	1	1.0	0.1
	PbBaSb	7.9	6	12	1	2.8	1.4
Police	PbSb	17.1	4	6	1	2.2	1.1
(n=76)	PbBa	7.9	2	13	1	3.3	1.5
	SbBa	52.6	12	14	1	2.8	2.6

Table 49 - Comparison of Characteristic and Consistent particle types in the random population andthe police population

^a of participants with the nominated particle type found.

Ultimately, the overall frequency for officers having at least one three-component characteristic particle on their hands was 7.9%, which is comparable to the 7% finding reported by Gialamas et al.[99]. When considering these results in the context of the previous work by Cook [101], the prior study reported that of the 33 subjects tested immediately following receipt of their firearm, only 15% returned no characteristic GSR particles, with the remainder returning at least one characteristic particle. Narrowing the sample set in the current study to consider only officers that had handled their firearm in the hour prior to sampling allowed for direct comparison between the data sets. In the current study, 12 participants met these requirements. The comparison of particle types of interest between the studies can be seen in Table 51.

CHARACTERISTIC PARTICLE TYPES **NO. OF SUBJECTS WITH** MEAN OF ALL FREQUENCY SD NOMINATED PARTICLE MAX. MEDIAN PARTICIPANTS (%) TYPE PRESENT ASSESSED Cook [101] 142.4 (n=33) 84.8 28 610 6.0 63.8 3.4 16.7 2 0.0 1.0 Current (n=12) 12 **CONSISTENT PARTICLE TYPES NO. OF SUBJECTS WITH** FREQUENCY MEDIAN SD NOMINATED PARTICLE MAX. MEAN (%) TYPE PRESENT Cook [101] 160.3 (n=33) 97.0 32 722 13.0 76.6

17

2.0

 Table 50 - Comparison of results of Characteristic and Consistent particle types found on the hands of officers who had handled a firearm <1hr before sampling with Cook [101].</th>

As can be seen from Table 51, both the frequency of officers with Characteristic GSR present on their hands, and the number of particles detected when positive, are lower in the current study. Though the reduction in frequency is less pronounced for the consistent particle types, it can still be observed that the overall number of particles detected is lower than was observed previously. Fewer subjects were observed to have characteristic particles on their hands, and the number of particles detected was lower overall than were observed in Cook's study [101]. This is despite the fact that all subjects across both studies had the same GSR exposure on receipt of their firearm at the start of their shift. This observed reduction in both the number of subjects with GSR on their hands and the number of particles present between the studies suggests that anti-contamination measures that are in place for officers after receipt of their firearm may be effective at minimising GSR retained on hands. These anticontamination measures have included education campaigns about GSR cross-contamination, as well as posters and reminders that officers should wash their hands thoroughly after receipt of their firearm. However, it should be noted that 12 characteristic particles were detected on

10

Current (n=12)

83.3

5.2

4.5

one subject, which is a particle count higher than 64% of the subjects in Cook's study. This speaks to the possibility of GSR contamination still persisting on some subjects at a level that may facilitate secondary transfer.

Interestingly, subject questionnaires indicated that all of the 12 subjects with GSR particles detected on their hands reported that they had not washed their hands since receipt of their firearm. If this is accurate, this means that the mechanical removal accomplished by handwashing may not have been responsible for the reduction in particles observed between this study and the study by Cook. This presents a number of possibilities to explain these observations. Firstly, two possible options for hand-washing are available for police officers in this situation. The first is soap and water, where GSR particles are removed by the running water and the mechanical action of lathering and drying of the hands. The second option is self-drying hand sanitising gel, which is made available in police equipment rooms as an alternative for hand cleaning. This hand-wash has been observed to be effective at preventing GSR particles from being collected from the hands during sampling. This alternative was previously explored by Cook [101]. To that end, it is possible that self-drying hand wash, made available to officers at receipt of their firearm, was used, and the officers did not report this as 'washing their hands'. The questionnaire did not ask respondents to specify how handwashing was performed, nor define what constituted handwashing. However, the fact that some GSR was detected indicates that if the hand-wash was used, it was not completely effective at removing GSR. A second option is that the prior study sampled subjects immediately after the receipt of the firearm, while the current subject group had handled a firearm in the past hour. This time interval between handling and sampling could account for the reduction in particle numbers observed. How long GSR persists on hands once it has been deposited has been widely studied in literature [106, 112, 114, 211]. Literature reports significant variability in the time periods that GSR persists on hands, depending on the circumstances of deposition, and activities undertaken after GSR is deposited. However, it is generally accepted that the retention of GSR on hands is poor, with most particles being shed in less than two hours [106]. It is therefore possible that in this study, any GSR that was deposited at receipt of firearm was lost from the hands prior to sampling. Finally, time since last handwashing was self-reported, and therefore the subjects may have been mistaken about the time of last hand-washing. Unfortunately, the nature of this survey prevents a direct cause from being established.

The data collected in this study indicated that the washing of hands after a GSR contact reduced the prevalence of all particle types. However, the general trend for observed levels of

GSR particles present on police officers was still non-zero, even after hand washing, and GSR particles were observed to be present on the hands of police officers at a level higher than that of the random population. Data indicate that GSR particles were present on the hands of officers who had reported washing their hands between receipt of their firearm and sampling, which suggests that either the handwashing was not efficient, or that additional GSR contact events that increase the background level of GSR on the hands occurred after the hands were washed. GSR particles are not known to decompose or degrade of their own accord, and once formed, the particles can theoretically persist on surfaces until they are moved or dislodged by some external force. Cook's prior finding suggested that the receipt of firearm at start of shift represents a significant GSR transfer event [101]. Further, other surveys that have indicated that police facilities and vehicles also have GSR present [102],[104]. It is therefore reasonable to suggest that particles lost from the hands in the aftermath of a GSR contact are redistributed into the environment and represent the possibility of a source for secondary or tertiary transfer events. This then speaks to the increased likelihood of GSR observed on police officers as compared to the random population. Taken as a whole, these data present the possibility that contact between a POI and a police officer carrying GSR particles originating from contact with their duty firearm, and potential recontamination events as a part of their work, can result in transfer of GSR to the POI, potentially resulting in a type I (false positive) error. To determine whether the possibility this occurring is significant or not, further transfer experiments were undertaken to ascertain the extent to which GSR may transfer from a police officer to a person of interest through the activities of apprehension and arrest.

GSR Transfer Studies

Samples were collected from the hands of the volunteers playing the 'POI' prior to their mock arrest to assess the background level of GSR. As there was no GSR detected on their hand samples prior to contact with the 'officer' in their mock arrest scenario, it was assumed that all GSR detected after the mock arrest originated from the contact between 'officer' and 'POI'. It was also assumed that the number of particles initially present on the 'officer' as a result of firing two rounds of ammunition could be expressed as the sum of the number of particles detected on the 'officer' sample and the number of particles on the 'POI' sample after mock arrest. Transfer was assessed as a ratio of the number of particles present on the sample collected from the 'POI' and the number calculated to be present on the 'officer' after firing two rounds of ammunition, and was expressed as a percentage transfer efficiency. This method for reporting particle transfer has been used previously by French et al. [91].

The results of this can be seen in Table 52. A lack of consistency was observed in the number of particles transferred to the POI. The maximum number of characteristic GSR particles detected was 308 present on the hands of one of the police officers, while samples from 10 officers had less than 10 characteristic particles on their hands, with two having none detected at all. This range of particle counts was observed despite the fact that all officers were known to have discharged the same number of rounds from the same firearm under the same conditions. With regards to the transfer between officer and POI, in six cases, no characteristic particles were detected on the POI, despite the fact that the officer arresting them was observed to have GSR present on their sample after 'arrest'. Further, there was one instance of an officer returning no characteristic particles on their hand sample; their arrested POI returned 8 characteristic particles. It is possible in this case that all of the GSR present on the officer's hands has either been transferred to the POI, or otherwise dislodged through the process of arrest. Alternatively, there is the possibility that GSR was present on the officer, but it was not collected on the sample stub. This serves as a reminder that the collection of a GSR sample is a survey only, and does not represent the entirety of GSR that may be present on an individual. Further, this reinforces the notion that the absence of GSR on a sample collected from an individual does not conclusive suggest that there has been no firearm association. Regardless, between both characteristic and consistent particles in all cases some secondary transfer was observed.

		OFFICER			POI		
			-				
			ALL				EFFICIENCY °
PAIR	CHARACTERISTIC	CONSISTENT	GSR	CHARACTERISTIC	CONSISTENT	ALL GSR	(%)
1	46	299	345	7	8	15	4.2
2	7	27	34	7	20	27	44.3
3	5	14	19	0	5	5	20.8
4	0	6	6	0	18	18	75.0
5	7	4	11	4	9	13	54.2
6	0	8	8	8	7	15	65.2
7	14	47	61	28	2	30	33.0
8	9	20	29	1	1	2	6.5
9	21	82	103	5	2	7	6.4
10	11	14	25	4	4	8	24.2
11	4	6	10	0	14	14	58.3
12	3	5	8	21	75	96	92.3
13	11	22	33	15	95	110	76.9
14	2	29	31	5	14	19	38.0
15	18	61	79	1	12	13	14.1
16	97	206	303	3	4	7	2.3
17	5	47	52	1	18	19	26.8
18	25	57	82	13	21	34	29.3
19	36	133	169	0	8	8	4.5
20	308	1162	1470	0	12	12	0.8
21	233	732	965	0	5	5	0.5
Median	11	29	34	4	9	14	26.8

Table 51 - Number of GSR particles, separated by particle type, for both officer and POI and secondary transfer efficiency following mock arrest scenario.

^a Secondary transfer efficiency was calculated by dividing the number of all GSR particles present on the POI by the sum of all GSR present on the officer and the POI, multiplied by 100. This method for reporting secondary transfer efficiency has previously been reported by French and Morgan [92]

Though the median transfer efficiency was calculated to be approximately 27% across the whole sample set, it can be seen from the individual transfer pairs in Table 52 that there is variation in the secondary transfer efficiency, ranging from 0.5% of the total particles transferred, through to 92.3% of the total particles transferred. This is, to some extent, a factor of the variation inherent in the quantity of GSR deposited initially as a result of the firearm discharge, as well as variability in the activity related to the transfer incident. Functionally controlling for this is not only difficult in an experimental capacity, but doing so means that the experimental conditions are unlikely to be reflective of the real-world conditions of transfer. However, considering these data as a whole and grouping the secondary transfer efficiency into quartiles provides useful information. As can be seen from Figure 52, a greater number of observations were observed in the first quartile of transfer,

representing transfer between 0-25% of the overall particle population. This suggests that the most transfer is of a small percentage of the GSR present on the contaminated person. That said, it should be acknowledged that like many GSR studies, there is significant variation in the amount of GSR initially deposited on an individual, and the amount of GSR that is then transferred. The variation in the data can be observed in Figure 53.



Figure 52 - Total GSR transfer efficiency grouped by quartile.

Figure 51 demonstrates that in nearly 50% of the cases modelled in this study, less than 25% of the available particle population was transferred. It can be inferred from this result that in the contact modelled, most observed outcome was only a small amount of particle transfer. Conversely, greater than 75% particle transfer was observed in less than 10% of cases. In this instance, this would suggest that in this study, it was more probable that a small percentage of particle transfer occurred. However, transfer modelling is vulnerable to many random variables that may influence the result, and Figure 51 does not provide any information regarding the variability within the quartile divisions. To demonstrate this, a box and whisker plot displaying the variability between the quartiles, as well as the maximum and minimum transfer percentages can be observed in Figure 53.



Figure 53 - Box and whisker plot of transfer variability between quartiles.

Similar to the findings reported in prior research, the results of this study represent a 'worst case scenario' of a POI being arrested and sampled for GSR immediately after a police officer has discharged a firearm and are therefore likely to overestimate the extent of transfer between officers and POIs under typical 'real-life' arrest situations. For practical purposes, the interval between the time of firing and the time of arrest was short. This limitation has been previously acknowledged in similar studies, such as that conducted by Charles and Geusens [73], and French et al. [91].

The results, however, do provide some insight into the extent of secondary transfer due to physical contact. French and Morgan's prior study reported between 6.4% and 11% secondary transfer observed following a handshake between a person who had recently fired a gun and a non-involved second party [92]. The current results, however, show much more variability than this. This suggests that the extent of secondary transfer can be influenced by a number of factors, including the duration of contact and level of activity during contact amongst others. In this case, the activities that were undertaken as a part of the contact – a wrist-lock and temporary restraint - represent more involved actions than a handshake, which may have resulted in the increased variation observed. These considerations make the extent of secondary transfer due to physical contact complex to model, with the results presented here intended to provide a starting point to assess the worst possible case.

The likelihood of a false positive due to contact between police and members of the public is not only based on the number of particles on the hands of the police officers. It is also contingent on the quantity of particles transferred through secondary contact. When the results of the background levels of GSR on police officers survey are considered in the context of the likely extent of transfer, it provides further information around the possible outcomes in a real world situation. Based on the particle transfer data collected through mock arrest scenarios, it was observed that the most observed outcome resulted in less than 25% of the initial particle population being transferred to the subject. This was observed in almost 50% of cases involving brief contact between subjects. Conversely, these data indicated that more than 75% of the initial particle population were transferred in less than 10% of modelled mock arrest situations. When considering the limited amount of GSR particle contamination present on police detected in the first phase of this study, this suggests that if the most contaminated police officer had contact with a POI, the most likely outcome is that fewer than 3 particles would be transferred to the POI. Even if the least likely scenario of greater than 75% transfer is considered, this would result in 8-12 particles transferred to the subject. It should be noted, however, that the extent of secondary transfer based on physical contact is likely to be influenced by the type, intensity, and duration of physical contact. In this study, while the contact was longer than may have been expected in handcuffing, subjects were relatively compliant, designed to mimic the brief physical restraint and handcuffing of a subject in the process of arrest. Longer contact, or contact involving more physical movement or struggling may be expected to change the dynamics of particle transfer. Regardless, this factor should be considered when interpreting GSR evidence, particularly in cases were only a small number of GSR particles has been detected. While this is instructive in the context of this study, a more detailed statistical model may be considered as a means of evaluating the weight of evidence in such cases.

This study makes no specific comment or calculation on the probabilities from this data that could be used to inform a likelihood ratio or Bayesian network approach to the evaluation of the significance of this data. However, incorporation of background levels of GSR on the hands of police, and the magnitude of transfer through the processes of arrest could be used to inform this approach.

6.3.4. Conclusions

This study set out to assess the GSR background present on the hands of firearms-carrying police officers, and through mock-arrest experiments, determine the possible extent of secondary transfer to a POI. Survey data were collected from police officers for whom firing their duty firearm was a relatively rare event, with most officers reporting that the last time that they discharged a firearm it was as a part of routine firearms requalification. Despite this, characteristic GSR particles were detected on the hands of approximately 8% of the police officers sampled, compared to 0.3% of the random population. While some officers in this study possessed a much higher background level of GSR particles than the random population, on average, the number of particles present was comparatively small, with fewer than 3 characteristic or consistent particles being present on their hands on average. The findings presented here suggest while the prospect of secondary transfer to POIs from the hands of non-firing police officers is not negligible, it is also not a major concern in evaluation of GSR evidence where large numbers of particles are involved. However, the data suggest a cautious, case-by-case approach be used in the assessment of GSR evidence, particularly if small numbers of particles are being considered.

----- End of Publication-----

6.4. AMMUNITIONS USED BY SOUTH AUSTRALIAN POLICE

6.4.1. Background

South Australian Police (SAPOL) make use of two different ammunitions for the majority of their operational and training purposes. These ammunitions were sampled for the purposes of understanding the types of GSR that they may generate. The operational ammunition sample was 0.40 S&W Federal Premium Law Enforcement Tactical, 165 grain HST ammunition. Safety data sheets from the manufacturer indicated that this ammunition contains Pb, Ba and Sb in the primer mix [210]. The HST ammunition uses a Ni-plated brass cartridge, and has a partial Cu-jacketed, Pb core, hollow point projectile. This construction is designed to facilitate expansion of the projectile upon hitting a target. This has the dual purpose of increasing the stopping power of the round, and reducing the likelihood of over-penetration of the target which is likely to result in collateral damage. An example of a recovered fired cartridge case (FCC) and fired projectile, demonstrating projectile expansion, can be seen in Figure 54.



Figure 54 - Recovered cartridge case and expanded projectile from 0.40 S&W Federal Premium Law Enforcement HST ammunition. (Image supplied by author) The training ammunition was 0.40 S&W Federal American Eagle, 165 grain total metal jacket (TMJ), ammunition with toxic metal-free primer. As the primer does not contain toxic metals, safety data sheets did not report the elements present in the primer. This ammunition consists of a brass cartridge case, and has a Cu TMJ, Pb core projectile. TMJ construction means that the Pb core of the projectile is completely coated with Cu (including the base of the projectile). This is distinct from full metal jacket (FMJ) ammunition in which the base of the Pb core is visible [1]. This complete encapsulation of the projectile prevents it from expanding on impact with a target, but also limits the amount of Pb from the surface of the projectile that is released into the air. Due to this, TMJ projectiles are often featured in ammunitions used for target shooting at an indoor range, to aid in limiting the inhaled Pb exposure to range staff. For this reason, this ammunition is used at the SAPOL training range for routine firearms qualifications. An example of a recovered FCC and fired projectile can be seen in Figure 55.



Figure 55 - Recovered fired cartridge case and projectile from 0.40 S&W Federal American Eagle Cu-TMJ, Toxic metal free primed ammunition. (Image supplied by author)

A further rationale behind the sampling of these ammunitions is supported by work conducted by Charles and Geusens [73]. In their study, they observed TiZn particles transferred to the hands and clothing of arrested individuals after contact with special units of the police. It was determined that these particles originated from training ammunitions used by these special units, which used a Ti and Zn based toxic metal-free (TMF), or 'green' primer compound to minimise airborne Pb exposure during training. Use of such primer compounds was determined to be relatively uncommon in criminal shootings, but TiZn particles originating from these primers were consistently observed on the hands of police sampled in their study. To that end, it was suggested that the presence of TiZn particles could serve as a *de facto* indicator of cross-contamination from police sources. The civilian use of ammunitions containing HMF or TMF primers is comparably infrequent in Australia, with such ammunitions rarely observed in criminal casework, but comparatively frequently used in police training. If a particle composition similar to the TiZn particles observed by Charles and Geusens [73] can be identified originating from the ammunitions used by SAPOL, this particle type could serve a similar role as an indicator of cross-contamination. This will then better inform assessments of GSR evidence in situations where cross-contamination due to secondary transfer is thought to have occurred. To further develop an understanding of the types of residues that may be generated by this type of ammunition, samples of training and operational ammunitions were collected, both from the hands of shooters immediately after discharge and from fired cartridge cases as a means of comparison.

6.4.2. Materials and Methods

Sample Collection

All samples collected were collected using 12.5mm diameter Al SEM-pin stubs coated in double-sided carbon tape adhesive (Tri-Tech Forensics Inc. North Carolina, USA.)

Representative GSR samples for this stage of the study were collected from each of the two frequently used SAPOL ammunitions. The shooter was asked to thoroughly wash and dry their hands before a blank hand sample was taken.

Two rounds of 0.40 S&W Federal American Eagle TMJ with toxic metal free primers (Lot V427457) were fired through a Walther P99 handgun into a bullet recovery (water) tank. Immediately following this, the hands of the shooter were sampled directly using an SEM stub. The setup and stubbed area of the dominant hand can be seen in Figure 56. The spent cartridge cases were recovered using tweezers, and sealed in a clean, press-seal exhibit bag. The projectile was recovered from the water tank and retained in a separate press-seal bag.



Figure 56 - Firearm discharge into bullet recovery (water) tank. (left). Area of dominant hand from which sample was collected (right). (n.b. Image is to demonstrate process and setup only. The firearm pictured is a Ruger 0.22 Mk 2 handgun, and not the firearms used in testing).

The shooter was again asked to thoroughly wash and dry their hands and a blank sample was collected. Two rounds of 0.40 S&W Federal Premium Law Enforcement HST (three component primers) (Lot C19V26) was discharged through a Glock 23 handgun into a bullet recovery tank. GSR samples were immediately collected from the hands, and the spent cartridge cases and projectiles were recovered and packaged as previously described.

Comparison samples were collected from FCC for each ammunition. Recovery from FCC was completed separately, with all surfaces and tools cleaned between samples, and PPE changed in between in order to minimise cross-contamination. To recover the sample, the inside of each casing was gently scraped with a clean wooden probe, which was then rolled onto the surface of a fresh GSR stub.

All stubs were carbon coated using a Cressington 208 High vacuum carbon coater to an approximate thickness of 20-25nm (200- 250Å), as indicated by the colour change of a brass thickness monitor stub.

Sample Analysis

All collected samples were analysed by SEM-EDS using an FEI Inspect F50 Scanning Electron Microscope (FEI Inc., Oregon, USA) equipped with an EDS detector (EDAX Inc., New Jersey, USA.). Instrument set-up, calibration and operating parameters were as described in section 6.3.3, above.

As recommended in the ASTM E1588-17 for the assessment of GSR by SEM-EDS [13], a representative sample of particles classified as 'characteristic' of GSR were re-acquired and had their classification manually reviewed prior to reporting. Multiple detections of the same particle, where present, were corrected to ensure the accuracy of the characteristic particle count. The spectra of particles in the 'consistent' category were reviewed, but were not manually reacquired. A selection of the spectra for particles in the 'other classified particles' category were reviewed to confirm their categorisation was accurate, but they were similarly not re-acquired, nor were they corrected for multiple hits. For this reason, particle counts in this category should be considered indicative only.

6.4.3. Results and Discussion

The particle analysis results for the samples collected from the hands of the shooter after

discharging different ammunitions can be seen in Table 53.

Table 52 – Particle summary for hand samples collected from the shooter's hands pre-firing, and post firing 0.40 S&W Federal American Eagle (TMF) and 0.40 S&W Federal Premium Law Enforcement ammunitions.

	SAMPLE NAME						
PARTICLE TYPE		FEDERAL	FEDERAL				
	PRE-FIRING	AMERICAN	PREMIUM LAW				
		EAGLE	ENFORCEMENT				
	CHARACTERIS	TIC					
PbSbBa	0	0	75				
CONSISTENT							
BaSb	1	6	19				
PbSb	3	1	18				
PbBa	1	0	5				
BaCaSi	1	0	1				
BaAl	0	0	53				
Sr	0	47	8				
Total Consistent	6	54	104				
0		CIATED					
Pb	3	10	42				
Ва	0	0	25				
Sb	0	0	5				
Total Commonly Associated	3	10	72				
От	HER CLASSIFIED F	ARTICLES					
SbS	10	1	6				
BaS	25	15	43				
PbTi	0	0	0				
Ti	4	0	3				
Fe	73	29	151				
Sn	2	4	20				
Au	0	0	15				
AuCu	1	2	2				
Bi	30	10	39				
KCI	20	8	1				
Cu	11	58	18				
Zn	19	3	2				
Ni	1	2	5				
NiCu	0	1	0				
CuZn	12	66	31				
LaCe	6	4	1				
Unclassified	96	2	42				
Total	337	271	648				

The results for the hand blank sample collected from the shooter's hands prior to firing indicate that it is not entirely free of particles, with a small number of particles consistent with and commonly associated with GSR present on the sample. This is despite the fact that their hands had been thoroughly washed and dried immediately prior to sampling. This was expected due to the fact that the facility in which the sampling was performed was routinely used for the discharge of firearms using a variety of ammunitions, and hence, represented quite a high cross-contamination risk. The population can also be seen to contain a variety of common environmental particles including BaS, Fe, Zn, as well as lighter flint (LaCe), and jewellery metals (AuCu).Particles of these types were observed consistently across all samples collected.

After accounting the particle population present on the pre-firing hand sample, it can be seen that the most significant contributions to the particle population originating from the Federal Premium Law Enforcement ammunition are particles characteristic of GSR containing PbSbBa with spheroidal morphology. Additional contributions can be seen in the consistent category, with more BaSb, PbSb and PbBa particles observed. Most notably, there is an observed increase in BaAl-containing particles, which were not observed in the pre-firing hand sample. This suggests that AI is present as a component of the priming compound, which was supported by both comparison with a fired cartridge case sample and SDS data from this manufacturer [210]. Regarding the construction of the ammunition, it is known that the projectile is encased in a partial Cu jacket, and the cartridge case is constructed of Ni-plated brass. Particles containing all of these elements were observed in the hand sample collected following discharge, at a level higher than that which was observed in the pre-firing hand sample. A large number of the particles with characteristic composition observed exhibited a nodular or 'bumpy' appearance. These nodules appeared brighter under BSE, suggesting the presence of higher Z elements, and EDS analysis of the nodules indicated the presence of Pb. An exemplar particle and the whole particle EDS spectrum can be seen in Figure 57.



Figure 57 - Exemplar BES image and EDS spectrum of PbSbBa particle originating from 0.40 Federal Premium Law Enforcement ammunition.

Interestingly, a small number of Sr-containing particles were also observed on the sample collected from the shooter's hands. Review of the particles indicated that they had spheroidal morphologies consistent with GSR. However, the sample collected from the FCC did not indicate any Sr-containing residues. As there was no indication of Sr in the FCC, nor in the SDS data for this ammunition [210], it was concluded that the presence of these particles suggested cross-contamination from the previous firing of toxic metal free ammunition.

In considering the toxic metal-free ammunition, accounting for the particle population observed on the pre-firing hand sample, it can be seen that the largest contributions to the population attributable to Federal American Eagle ammunitions are Sr-containing particles, as well as Cu-containing and CuZn particles. The latter components are consistent with the construction of the ammunition, with the Cu-TMJ being the likely source of Cu-containing particles, and the brass (CuZn) casing. The population of Sr-containing particles represented an approximately equal mix of Sr-only particles, and SrAl particles. Both particle types exhibited spheroidal morphology consistent with firearm discharge; however the surface of the particle appeared different based on composition. A side-by-side comparison of the two particle types can be seen in Figure 58.



Figure 58 - Comparison BSE images and EDS spectra showing morphological differences between Sr only (top) and SrAI (bottom) containing GSR particles originating from 0.40 Federal American Eagle MFP ammunition.

Analysis of samples collected from fired cartridge cases from this ammunition indicated the presence of large Sr and SrAl-containing flakes with a disrupted or 'splattered' appearance, consistent with impacting a surface while still molten [212, 213]. An example of a SrAl-containing flake recovered from the fired cartridge case can be seen in Figure 59.



Figure 59 - SrAl flake originating from Federal American Eagle HMF Fired Cartridge Case

No other particles or fragments of interest were identified on either the hand stub or sample from the FCC. In this case, a SDS for the ammunition was unavailable to confirm the components based on the listed formulation, but it appears from the collected data that the primer compound contains Sr- and Al-compounds. It is important to note that under the ASTM guideline, Sr-containing particles are considered to only be consistent with GSR originating from Pb-free or non-toxic ammunitions. This is a reflection of their relatively common environmental sources, including airbags [71] and fireworks [57, 58], amongst others. However, identifying these particles originating from SAPOL training ammunitions still provides further information that can inform the evaluation of GSR evidence. Despite the existence of other sources of Sr and SrAI particles, identifying police training ammunition as another source of such particles may allow these particles to serve as an indication of possible cross-contamination from police, in the same fashion that TiZn particles were identified by Charles and Geusens [73].

6.4.4. Conclusions

An analysis of the two primary ammunition types used by South Australian Police (SAPOL) was performed through comparing the residues deposited on the hands of the shooter after discharge and those collected from fired cartridge cases (FCC). The primary operational ammunition, 0.40 S&W Federal Premium Law Enforcement HST, was seen to contain a three component primer, and produced characteristic PbSbBa particles. These particles exhibited spheroidal morphology, with a nodular type appearance. The further presence of a number of BaAl particles also indicated that the primer mix contained Al. This was supported by safety data sheets for the ammunition composition provided by the manufacturer.

Standard training ammunition, 0.40 S&W Federal American Eagle, with toxic metal-free primers was observed to generate a number of Sr-containing and SrAl-containing particles. No other particles containing elements characteristic to this ammunition were observed. Although Sr- and SrAl-containing particles have other environmental sources, identifying that these particles may also originate from police training ammunition provides valuable information that can further inform the assessment of GSR evidence.
6.5. GSR PRESENT ON SPECIAL UNITS OF THE POLICE

6.5.1. Background

A broad survey of GSR persisting on special units of the police was not initially performed alongside the survey of general duties officers, due to the fact that the extent of their firearms associations and elevated GSR background has been well documented [73, 105]. It was therefore thought that little additional information could be gained by further surveys of this particular group. However, following an incident in which a GSR analyst at FSSA detected abnormally large numbers of Sr- and SrAl-containing particles on a GSR stub collected from the hands of two individuals suspected of a firearms offence, this was revisited. In this case, the suspected firearms used were two 12 ga shotguns, and neither Sr- nor SrAl-particles were consistent with the ammunition. Although particles of this type were present alongside a number of characteristic and consistent particles on samples collected from the hands of the POIs, the atypical particle population was noted by the analyst. As it had recently been identified that Sr-containing particles are generated by SAPOL training ammunitions, this indicated to the analyst that perhaps cross-contamination between police and suspects had occurred. This prompted the analyst to seek further information about the specific case circumstances. It was at this point that it was discovered that the Special Tasks and Rescue (STAR) group, a special operations group of SAPOL, had been involved in the arrest of these individuals. To further investigate the possibility that STAR group officers had been then source of some of these particles, samples were collected from gloves belonging to STAR group officers. It was thought that in line with the findings of Charles and Geusens, GSR particle populations persist on the equipment of special units of the police [73].

Much like the special units of the police assessed in the Charles and Geusens study [73], the STAR group in South Australia participate in rigorous training involving multiple different firearms. As a result of this, they have exposure to the same training and operational ammunitions used by general duties officers, but on a more frequent basis. In addition to training and operational ammunitions, special units of the police in SA are exposed to another source of particulate pyrotechnic residues originating from stun-grenades. Stun-grenades, also known as 'Flashbangs' or 'Noise Flash Diversionary Devices' (NFDD), are used as a means of non-lethal distraction or temporary incapacitation in situations where a tactical advantage is required. To accomplish this, these devices are capable of generating an intense sound and

bright light, designed to temporarily deafen and blind individuals in the vicinity. Typically, the pyrotechnic mixture used contains a primer compound, and a mix of energetic components designed to create a loud explosion and accompanying bright flash. Devices used by police are capable of generating sounds between 170 - 180 decibels (dB), and a flash intensity of between 3 - 6 million Candela (cd) [214]. The composition of the primer and explosive may include primer compounds that are present in ammunition, as well as other components such as Al, Mg and ammonium or potassium perchlorate. As they use pyrotechnic primers, it is possible that these generate a population of particles that may also be present on special units of the police. To assess this, additional samples were collected from spent Rheinmetall Mk-13 BTV-1 Flash Bang Grenades, as used by special units of the police.

6.5.2. Materials and Methods

Sample Collection

Samples were collected from the surface of gloves belonging to STAR group officers. Collection was completed using standard SEM-pin stubs as previously described, and dabbing them over the surface of the gloves until the adhesive was exhausted.

Two spent Rheinmetall Mk-13 BTV-1 Flash-Bang stun grenades were collected after a routine training exercise and stored in separate press-seal exhibit bags until sampling. The devices can be seen in Figure 60.



Figure 60 - Spent 'flashbang' stun grenades sampled for particulate residues. (Top – side view. Bottom – Base view)

The two devices were of the same make and manufacture, but were distinct in the method of operation. Both have a steel body, and have pyrotechnic flash and sound charges contained within the body of the device. On the left hand side Figure 60, is a '6 bang' variant, which detonates with six separate sound and flash charges from the base of the body. This was sampled by applying a clean GSR stub to the inside of the base of a spent device. On the right

hand side of Figure 60 is a '2 bang' variant, which releases two sound and flash charges from within the sides of the device. This was sampled by applying a stub directly to the body of the device in the area around the holes pictured.

Sample Analysis

All collected samples were analysed by SEM-EDS using a FEI Inspect F50 Scanning Electron Microscope (FEI Inc., Oregon, USA) equipped with an EDS detector (EDAX Inc., New Jersey, USA.). Instrument set-up, calibration and operating parameters were as previously described in section 6.3.3. As performed previously, a representative sample of particles classified as 'characteristic' of GSR were re-acquired and had their classification manually reviewed prior to reporting. Multiple detections of the same particle, where present, were corrected to ensure the accuracy of the characteristic particle count. The spectra of particles in the 'consistent' category were reviewed, but were not manually reacquired. A selection of the spectra for particles in the 'other classified particles' category were reviewed to confirm their categorisation was accurate, but they were similarly not re-acquired, nor were they corrected for multiple hits. For this reason, particle counts in this category should be considered indicative only.

Due to the significant quantity of particles present on the sample stubs collected from the STAR group gloves, analysis time was capped at 500 minutes.

6.5.3. Results

A comparison of the particle count results for samples collected from each of five sets of gloves used by STAR group officers can be seen in Table 54.

DARTICIE TVDE	SAMPLE NAME				
	STAR 1	STAR 2	STAR 3	STAR 4	STAR 5
CHARACTERISTIC					
PbSbBa	4	20	54	10	13
	Cons	ISTENT			
BaSb	13	25	15	17	4
PbSb	1	42	42	16	5
PbBa	0	7	80	1	106
BaCaSi	82	180	5	69	8
BaAl	15	105	2	46	19
Sr	100	140	53	19	37
Total Consistent	211	499	197	168	179
C	OMMONLY	ASSOCIATE	D		
Pb	12	25	412	9	107
Ва	36	127	37	73	23
Sb	412	43	16	27	7
Total Commonly Associated	460	195	465	109	137
Οτι	HER CLASSI	FIED PARTIC	CLES		
SbS	6	17	5	24	1
BaS	589	2560	21	1026	42
PbTi	1	2	1	0	0
Ti	27	179	1	175	7
Fe	854	2347	243	1878	224
Sn	48	45	3	8	5
Au	2	7	30	7	8
AuCu	0	3	0	0	1
Bi	2	27	23	23	20
KCI	11	49	0	48	50
Cu	88	196	44	3	50
Zn	35	19	5	72	12
Ni	23	99	7	6	27
NiCu	1	72	0	0	0
CuZn	77	95	22	2	44
FeCrNi	31	49	72	30	6
Zr	31	53	4	56	3
LaCe	0	0	0	8	0
Unclassified	125	1119	192	165	107
Total	2663	7737	1438	3885	942
% of stub surface analysed	45.9	33.2	16.8	29.8	13.0

Table 53 - Particle summary of samples from gloves of STAR group officers.

From Table 54 it can be seen that restricting the analysis time to 500 minutes resulted in less than the total stub area being analysed. In each case, between 13% and 46% of the stub was assessed for the particle population present. A large and diverse particle population was observed across all samples, with particles consistent with primers from both operational ammunition (PbBaSb, BaAl, PbSb, PbBa, BaSb) and training ammunition (Sr and SrAl) observed. An exemplar SrAl particle consistent with SAPOL training ammunition can be seen in Figure 61. Further, jacketing and cartridge materials (Cu, CuZn) were also observed in high numbers. Large numbers of environmental type particles, including as BaS, Fe, LaCe, and FeCrNi were also observed.



Figure 61 - Exemplar SrAl particle consistent with Federal American Eagle HMF ammunition recovered from the glove of a STAR group officer

Consistent across all samples was the large number of particles that were present. This indicates either that the gloves are high-persistence surfaces, retaining particulates for a long time after they are deposited, or they are routinely in contact with a variety of firearms, ammunitions and other particulate residues. This suggests that they may represent a source of a variety of particles, including GSR, that may undergo secondary transfer to a suspect through the process of arrest, as suggested by French et al. [95] and by Charles and Geusens [73].

A particle summary detailing the particles observed on the samples collected from the two 'flashbang' stun grenades can be seen in Table 55.

	SAMPLE NAME		
PARTICLE TYPE			
	2 BANG	6 BANG	
CHARACTERIS	тіс		
PbSbBa	3	1	
CONSISTEN	CONSISTENT		
BaSb	2	1	
PbSb	1	1	
PbBa	76	34	
BaCaSi	71	13	
BaAl	323	13	
Sr	0	0	
Total Consistent	473	62	
COMMONLY ASSO	CIATED		
Pb	2169	1221	
Ва	176	124	
Sb	0	0	
Total Commonly Associated	2345	1345	
OTHER CLASSIFIED PARTICLES			
BaS	248	403	
PbTi	46	8	
Ti	72	43	
Fe	196	41	
Sn	7	1	
КСІ	634	256	
Cu	8	5	
Zn	138	73	
Ni	2	0	
CuZn	7	1	
FeCrNi	6	0	
CrNi	2	1	
Zr	5	95	
Total	4387	2575	

Table 54 - Parti	icle summary from	'flashbang'	stun grenade	samples
			Brain Breinaac	Samples

From Table 55, it can be seen that a small number of particles with compositions characteristic and consistent with GSR were detected on the samples. While particles characteristic of GSR were observed, it is unclear if these have originated from the device itself, or are present by virtue of secondary or further transfer. The nature of the use of these devices is that they are carried alongside firearms, and then thrown into what is potentially a heavily contaminated environment. It would therefore be unsurprising if some of the particles detected originated from firearms, and had been retained in some areas of the surface until sampled. However, the particle types present provide some indication of the composition of the pyrotechnic mixture. Comparatively few particles containing Sb as a component were observed both in the consistent and commonly associated category, when compared to the larger numbers of PbBa, BaCaSi and BaAl particles. This suggests that of the elements most relevant to the examination of GSR, the primer composition of these flashbangs is most likely to contain Pb- and Bacontaining compounds, with Sb-containing compounds being absent.

With regard to the wider particle population, most evident is the large number of Pb particles counted. Although counted as separate individual particles, a large number of 'flakes' embedded with small Pb containing nodules were observed. An example can be seen in Figure 62.



Lsec: 30.0 0 Cnts 0.000 keV Det: Octane Pro Det

Figure 62 - Fragment of debris recovered from a Flashbang. Fragment measures approx. 4 μm by 2 $\mu m.$

As can be seen in Figure 62, a large number of small Pb-containing nodules are scattered across the surface of these flakes. The origin of these flakes could not be ascertained, and it could not be determined if they exist as a feature of the manufacturing process, or produced during discharge of the device. The nodules are regularly shaped, sub-micron spheroids, showing no signs of impact disruption, suggesting that their presence is less likely to be attributable to the random conditions of primer discharge. A large number of such flakes were present in both samples, accounting for the large Pb particle count.

Common in flash powders are the combination of an oxidising agent and a powdered metallic fuel, with powdered AI, Mg and Zr often included as components. In this case, the observed presence of a large population of particles containing K and Cl is suggestive that a chlorate- or

perchlorate-based explosive compound may be used. Further, the particle population showed Zr was present, however Mg, or Mg-containing particles were absent. Although not marked as its own category, Al-containing particles were observed, most notably as an inclusion into the BaAl particles previously discussed. Though specific manufacturer or SDS information for this particular brand of device could not be found, comparison with other common devices of this type, for both civilian [215] and police or military applications [216] provided additional compositional information . Both KClO₄ and KClO₃ along with powdered Al and Mg are frequently present. With regard to elements of primary interest to GSR, compounds such as barium nitrate, barium chromate and lead styphnate are often present in these pyrotechnic formulations. While a further assessment of the composition of the primer and explosive compounds contained within these devices would be supported by performing a comparison of an undischarged sample, it was deemed too difficult and high risk to attempt to do so for the purposes of this study.

Fundamentally, it appears as though the flashbangs used by special units of the police operate using a Pb- and Ba-based primer that could contribute to the generation of particles compositionally consistent with GSR. It also appears that they are a rich source of Pbcontaining particles. Most notable was the appearance of sub-micron Pb-containing spheroidal particles adhering to small metal fragments, which were observed frequently in flashbang samples. Additional components, including K- and Cl-containing particles indicating the use of a potassium chlorate or perchlorate based explosive compound, and other metallic particles were also observed.

6.5.4. Conclusion

It was observed in this case that Sr and SrAl particles originating from training ammunition had been transferred to the hands of persons of interest who had contact with a special operations unit of the police. These particles were not explained by the firearms and ammunition types used by the individuals in this case. Further investigation indicated that high numbers of threecomponent GSR, GSR from training ammunition, and numerous other particles are retained on the gloves of the SAPOL STAR group. Either the gloves represent a high-persistence surface, or they are frequently exposed to various sources of GSR. Either way, they represent a potential source of particles that may undergo secondary transfer to other surfaces or individuals. Regardless, it was observed in casework that Sr and SrAl particles with morphology consistent with GSR were observed on the hands of individuals apprehended as a part of an investigation under conditions where such particles were unexpected. To that end, much like the TiZn particles that were observed in large numbers on the hands, gloves and equipment of the special operations group sampled in Charles and Geusens survey [73], the Sr and SrAI particles in this case served as an indication that cross-contamination had occurred.

Review of particles present on spent 'flashbang' stun grenades suggests that they operate using a PbBa- based primer, with a chlorate or perchlorate based explosive, and several metallic components. This suggests that they possess the potential to generate particles with compositions consistent with GSR and therefore may contribute to a particle population that could be retained on the gloves or equipment of special units of the police, and therefore undergo further transfer. Flashbang particles, along with GSR from conventional primed ammunition and HMF ammunition were observed on the gloves of the special units of the police assessed in this study, suggesting that their gloves and equipment represent a significant cross-contamination risk. For this reason, it is important for a GSR analyst to know if these units have been involved in the arrest or apprehension of persons of interest to a firearms investigation.

6.6. CONCLUSIONS

A GSR background study conducted on firearms-carrying SAPOL officers was conducted as a means to determine the extent of GSR present on their hands under routine circumstances. Characteristic GSR particles were detected on the hands of 8% of the officers sampled, which compares to only 0.3% of the wider public population. However, the amount of GSR present was relatively low, with fewer than three characteristic or consistent particles present on average. This suggests that despite the findings of Cook [101], which indicated that receipt of their firearms at the beginning of their shift represents a significant transfer of GSR to the officers hands, this is well controlled through existing contamination control measures. While GSR is observed more frequently on the hands of police than of the general public, it is at levels that suggests it is not a major concern with regards to cross-contamination of persons of interest in shooting investigations arising through contact with police.

To test this suggestion, transfer modelling experiments to investigate the extent of GSR transfer between an officer and another individual in the process of apprehension or arrest was investigated. It was found that transfer was highly variable, however, the most probable outcome was less than 25% of the overall particle population undergoing secondary transfer. The conditions for these experiments were intentionally representative of the 'worst case scenario' in which the officer had recently discharged a firearm, the individual was free of GSR, and the contact occurred relatively soon after a firearms discharge. Therefore the conditions are likely to overestimate the amount of GSR transferred.

Representative samples 0.40 S&W Federal Premium Law Enforcement HST ammunition, used as the standard operational ammunition in SAPOL firearms were collected. Analysis of the GSR produced by this ammunition did not indicate any atypical or unexpected elements that could serve to identify these particles as originating from SAPOL ammunition. The presence of BaAl particles indicates the inclusion of Al in the primer mix, which was supported by manufacturer's SDS data [210]. While Al-containing primers are not unique to SAPOL ammunition, they are similarly not ubiquitous in all ammunitions, and therefore may serve as a point of differentiation in some circumstances. With the exception of the inclusion of Cu or Cu-containing particles, likely due to the partial Cu jacket, and CuZn the particle population generated was as would be expected from three-component primed ammunition. However, the 0.40 S&W American Eagle HMF primed ammunition used by SAPOL for training was found to generate significant numbers of Sr and SrAl containing particles. Although most general duties officers would not retain these particles on their hands or uniforms for an extended period, it was found that a particle population containing these particles persists on the gloves and equipment of Special Tasks and Rescue (STAR) group officers. These officers are part of a special operations group of the police which conducts rigorous firearms training with a large and diverse number of firearms. Similarly, they are also exposed to other pyrotechnic residues through the use of flashbang style stun grenades, which were also assessed in this study. Although STAR group officers are not involved in the arrest or apprehension of many individuals, it seems prudent that their involvement in the arrest of individuals who are then sampled for GSR should be noted in records. Such records would be of value in the interpretation of GSR results due to the elevated risk of GSR contamination. As HMF ammunitions represent a relatively small segment of the consumer market in South Australia, but are routinely used in training by police, an atypical population of Sr or SrAl containing particles may be used as an indicator that some cross-contamination may have occurred, similar to the role that TiZn particles played in a similar Belgian survey [73].

7.A FRAMEWORK FOR THE EVALUATION OF GSR EVIDENCE

7.1. SYNOPSIS

Factors that influence the evaluation of GSR evidence were determined experimentally, and reported in previous chapters. Of particular consideration were factors informing the denominator of the likelihood ratio equation, that is, factors influencing the probability of observing characteristic GSR particles on an individual that has had no involvement with the firearms incident under investigation. The factors considered in this evaluation included the GSR background in the random Australian population, the GSR background on the hands of general duties police officers, and the likelihood and extent of cross-contamination due to secondary transfer as a result of contact during apprehension and arrest. The cumulative impact of these factors was considered as a means to estimate the probability of observing GSR on an individual who has had no firearms association, that could lead to a false positive result.

A model incorporating these factors based on a Bayesian Network (BN) is explored in this chapter. The limitations and challenges of performing a BN style assessment of factors related to the incident under investigation was also explored. The proposed model is not without its limitations, and is not intended to represent an inflexible rule which should be broadly applied in all situations. Rather, this is intended to function as a framework which may be used as a foundation upon which more detailed, case-by-case evaluation may be performed. A further use of these findings may also be in better contextualising GSR evidence, and therefore supporting evaluative assessments of evidential significance of GSR in Australian casework.

7.2. BACKGROUND

7.2.1. Existing approaches to the evaluation of GSR evidence

While the ASTM guide is prescriptive regarding sample preparation, instrumental requirements and calibration, and the definition of GSR particles [13], it makes no comment on the interpretation of the significance of the evidence once found. Specifically, the method notes that the ASTM guide,

...cannot replace knowledge, skill, or ability acquired through appropriate education, training, and experience and should be used in conjunction with sound professional judgement. [13]

As a result, it has been left to individual jurisdictions to define their own evaluation processes for the significance of GSR evidence, based on their specific requirements and challenges. Some laboratories restrict the reporting of GSR evidence to the presence or absence of GSR only, and provide no statement to the significance of this finding based on the circumstances of the specific case under consideration. Under this paradigm, detection of a single characteristic particle may be considered sufficient to report that the sample was 'positive for GSR' [93, 217]. However, this approach has attracted criticism for its failure to provide enough information to be sufficiently clear to avoid misinterpretation by the triers of fact [199]. This has been particularly problematic in situations where small numbers of particles have been located, and the significance of this finding, or possibility of cross-contamination from contact with law enforcement has not been adequately communicated. In these cases, reporting the presence of GSR without a statement of the significance of the finding has the potential to be prejudicial, resulting in high profile cases in which the integrity of GSR evidence has been questioned, such as in the cases of Barry George, Dwaine George, and Ross Monaghan in the UK [199-201]. As a result, standardised terminology for the framing of GSR evidence assessment may be used, based on a 'formal' approach as described by Romolo and Margot [107]. This approach incorporates specific wording into the report that communicates the limitations associated with the finding, whether positive or negative, based on the categories defined by the ASTM [37]. While these caveats do accurately capture the limitations and considerations taken by the examiner in the reporting of GSR, they have the possible consequence of resulting in GSR evidence being under-valued or misinterpreted. As a result, some sources have viewed GSR evidence as of limited value in supporting an investigation [218], and GSR analysis viewed as flawed, or being discontinued by some laboratories [219]. Consequently, a strictly 'formal' approach to the assessment of GSR evidence has been viewed as problematic [10], particularly in situations where ammunitions that do not produce characteristic GSR, such as HMF ammunitions, or Sb-free primers are concerned [107]. Romolo and Margot in particular criticised the limitations of such a formal approach, and advocated that GSR analysts adopt a more fruitful case-by-case approach [107].

As a means of addressing the limitations of the 'formal' approach, in some jurisdictions, the experience and expertise of the examiner is relied upon to fill the gap. Under this paradigm, it is left to the analyst to consider the specific circumstances of the case in question, interpret the GSR evidence in the context of their wider experience, and present the significance of the finding on this basis. However, inherent in the expert's assessment is a level of subjectivity, based on the specific experiences of the examiner and which factors they deem to be the most significant. Over-reliance on the opinion of an expert without a clear and systematic explanation of the specific factors that underpin the reasoning leading to the conclusion is similarly vulnerable to misinterpretation, as is the case with the 'formal' process, or worse - manipulation on behalf of the expert. The inability of the expert to explain and justify their reasoning underpinning a GSR assessment has also drawn criticism in some high-profile cases, such as Australia's Eastman case [220].

As a means of addressing the limitations of other approaches to the assessment of GSR evidence, a further approach that has been widely adopted is a 'case-by-case' assessment, in which the specific factors relevant to the matter under investigation are evaluated and tested by the expert prior to evaluating the significance of the finding. This may take the form of performing test-firing case ammunition with recovered firearms (if available), as a means of addressing the contribution of the weapon memory effect on the particle population, and then comparing this with the recovered GSR sample [172, 217]. More common is comparison of the exhibit GSR sample with a sample recovered from a fired cartridge case (FCC), recovered firearm, or sample from the victim's clothing. While this approach strengthens the ability to apply the findings directly to the circumstances under investigation, it also significantly increases the amount of time and resources required, as well as adding complexity to the analysis. Similarly, in situations where a suitable comparison sample cannot be obtained, communicating the significance of the findings falls back on the opinion and experience of the expert, or in the caveats of the 'formal' approach.

In order to strengthen the ability of experts to communicate the significance of GSR evidence, it is clear that there is a need for a structured, robust, and objective method for the interpretation of GSR evidence. As a means of addressing this, work has been conducted in

some jurisdictions to incorporate and strengthen an evaluative reporting framework to the assessment of all evidence types [123-125], or to apply a Bayesian Network (BN) approach to interpretation [139, 148, 150]. Either such framework is capable of providing a formal, structured, and systematic means of evaluating scientific findings in the context of their relative likelihoods given the case circumstances. In the context of GSR evaluation, cautious application of such a framework allows for transparency of the assumptions made by the expert, which can be underpinned by research and data, and can provide an objective and consistent framework for the assessment of the evidence. Even so, such an approach is not without its own limitations and challenges.

7.2.2. Challenges with a statistical model for GSR evidence evaluation

As an evidence type, GSR faces a number of challenges with the application of a rigorous statistical model for the evaluation of GSR evidence. Typically, the strength of a statistical model is contingent on the ability of the analyst to use this model in order to make strong and sound predictions about the nature of the evidence. For example, the application of statistical evaluation of DNA evidence at the source level is well established [136, 137, 143, 221-223]. In performing this evaluation, the DNA analyst has knowledge of both the random population frequency of the particular profile, as well as the fact that the questioned profile is characteristic of the individual from which it originated. These factors are also static, and are assumed to be unchanging in a particular population over time. Combined, these factors allow the strength of evidence at the source level to be presented as a random match probability of the form "The probability that a randomly selected individual, unrelated to the POI, would share this DNA profile is estimated to be one in 10 million". Unlike DNA, GSR and other types of trace evidence face complications at the source level, and further challenges at the activity level. The nature of GSR particle formation, results in a lack of consistency in the composition and morphology of GSR particles, even if originating from the same ammunition, which complicates source level evaluations. Similarly, at the activity level, there are significant challenges associated with establishing a static random population frequency, and are influenced by numerous factors that impact the deposition, prevalence, persistence, and transfer of the original source. The influence of these factors significantly increases the complexity of applying a statistical model to the evaluation of GSR evidence.

These complexities are a feature of many trace evidence classes, including glass and fibres, and are not inherent to GSR. A likelihood-ratio (LR) framework has been well established for

its applications in the evaluation of forensic evidence [224, 225]. At a fundamental level, the LR represents the relationship:

$$LR = \frac{P(E|Hp)}{P(E|Hd)}$$

where the numerator represents the probability (P) of observing the evidence (E) given the hypothesis of the prosecution (H_p), is true, and the denominator represents the probability of observing the same evidence, given that the hypothesis of the defence (H_d) is true. The LR represents a comparison of relative likelihood of two hypotheses, given the observations of the case in question. Consequently, values of the LR greater than one favour H_p , while values less than one favour H_d . Care must be taken in assigning and evaluating these hypotheses, in order to ensure a fair comparison [226]. The challenge in informing the LR calculation, however, is developing an understanding of the relative probabilities of observing a particular value for the evidence under both H_p and H_d . In evaluating GSR evidence in this context, numerous challenges exist.

In order to develop a robust, statistical model for the evaluation of GSR, the challenges associated with it as an evidence type must be addressed. The random factors that influence the deposition of GSR in the aftermath of a firearms discharge result in the inability to accurately establish a 'base rate' for the initial deposition. The type and amount of GSR deposited on an individual involved with a firearms incident may be influenced by the firearm [5, 83], ammunition type and number of rounds discharged [29, 69, 70, 116, 206, 227], and the location from which the sample is collected (e.g., hands, hair, or clothing) [43, 44, 110, 113, 114]. A further complexity exists in the fact that deposition of GSR is not restricted to the individual who discharged the firearm, but is distributed as a plume influenced by atmospheric conditions, and may also be found on victims, bystanders, and the wider environment [81, 87, 90, 116]. Statistical assessments of the significance of GSR evidence have been further complicated by the introduction of HMF-primed ammunition [70, 228], with the probabilities of encountering particles generated by these ammunitions being comparatively underresearched. Given these limitations, even the most robust of statistical models would be unable to reliably predict the expected amount and type of GSR on a shooter, or conclusively differentiate a shooter from a bystander. This means that constructing a predictive model allowing a definitive initial type and quantity of GSR deposited on the individual responsible for discharging the firearm, based solely on the available case-context represents a significant challenge.

Beyond the factors influencing the deposition of GSR, the level of persistence is similarly challenging to accurately gauge. Rates of loss are subject to the influence of random factors, including surface type [88, 229], and the type and level of activity undertaken between particle deposition and sampling [112]. To that end, the rate of particle loss cannot be definitively quantified. While extent of particle loss can be modelled, applying the findings to a casework context would require a detailed knowledge of all activity undertaken by the POI between firing and sampling, which is relatively unlikely in casework. A further complication is the possibility of a further, unrelated GSR exposure event between the incident in question and the time of sampling. Currently, consistently and conclusively differentiating GSR particles deposited as a result of the incident in question and those possibly deposited as a result of a further unrelated event is not possible. This means that GSR deposition from a later event cannot be excluded as a factor that may influence GSR interpretation.

Finally, a further important consideration in the construction of statistical models is the fact that the utility of the model is contingent on the data that informs it. Advocates of a statistical method tout the usefulness of using graphical models, such as Bayesian networks, for their flexibility and versatility to be modified based on the specific circumstances being modelled [142, 148, 150, 230]. Critics however, urge caution in an overzealous application of this approach, warning that the previously discussed complexities of GSR as an evidence type make a unified model for quantitating GSR evidence highly complex [149]. Further, when determining what modifications to the network are required based on the circumstances being modelled, there is an inherent level of subjectivity in which modifications are chosen [149]. However, Gallidabino, Biedermann and Taroni, provide a worthy reminder that 'every inferential model cannot be anything other than an approximation to the complexities of the real world' [231]. While constructing these models presents a challenge, they may still be a useful tool in the assessment of GSR evidence provided they are applied cautiously and with careful consideration of their limitations.

It is clear that there are a number of limitations associated with factors informing the statistical assessment of GSR evidence. Most notable amongst them is the lack of consistency and highly random nature of the distribution, persistence, and possible transfer of GSR in the aftermath of a firearm discharge. In the final analysis, the combination of these factors results in a situation that GSR examiners possess no practical means to accurately determine the type or amount of GSR that could be expected to be present on an individual if that individual was

involved in the firearms incident in question. This makes assigning a probability that can inform the $P(E|H_{o})$ expression of the LR equation particularly challenging.

7.2.3. Statistical approaches to GSR evidence evaluation

Initial studies into informing a likelihood ratio-based approach for the purposes of GSR evaluation have focussed on informing the significance of a finding informed by test-firing data and further modelling. Cardinetti et al. proposed a statistical model for the evaluation of GSR evidence at the activity level that could inform the probabilities of detecting n GSR particles on a shooter or non-shooter at some time (t) after the firearm discharge [147]. In this case, experimental data informing the model for GSR on shooters' hands was collected directly by performing a number of test firings in which police volunteers fired a number of rounds, before being sampled for GSR at different intervals (2, 3, 4, 5, 6, 8, and 10 h) after firing. Further samples were collected from the hands of police officers that had not recently handled firearms, across the same time intervals, to be used as a model for the GSR present on nonshooters' hands. The fact that the non-shooters in this instance were police officers, and were therefore expected to have a higher GSR background, was intentional to avoid favouring the prosecution hypothesis in the evaluation. The combined data were then used to inform a framework for assessing the probability of observing characteristic particles on both a shooter and a non-shooter, at some time t, after the initial discharge, and thereby informing a LR. To do this, the authors performed goodness of fit testing of the experimentally collected data against a number of probability distribution models. A Poisson distribution was selected based on the fact that it describes the discrete probability distribution of rare events. The authors argue that given the amount of GSR produced in a firearm discharge, observation of n GSR particles at time t after the discharge event should be considered a rare event, and thus a Poisson distribution is a rational choice for these types of data. To assess the suitability of this assignment, the authors performed an assessment of fit for experimental data to the Poisson distribution using a chi-squared test. It was found that the Poisson distribution showed a good, but not perfect, fit with the experimental data, with the experimental data departing more from the distribution at t=2h. This suggests that perhaps the observation of GSR present on an individual at 2h (and presumably below) should not be considered a 'rare' event that can be modelled with the Poisson distribution. The authors acknowledge that other distributions may also fit the experimental data, or fit the data better (such as a normal distribution), but this would also have the effect of favouring the prosecutor's hypothesis, and would therefore unfairly disadvantage the defendant. As it stands, the choice of the distribution in this study was found to underestimate the likelihood ratio, and therefore be the most favourable to the

defendant. The distribution for this study is defined using a Poisson distribution, $P_{\lambda}(n)$, which was defined by:

$$P_{\lambda}(n) = e^{-\lambda} \frac{\lambda^n}{n!}$$

Such that λ is equal to the mean number of GSRs detected on a sample stub collected from the shooter, and provides a probability distribution of finding *n* GSRs.

Therefore, for the assessment of the likelihood ratio, V(n),

$$V(n) = e^{\mu - \lambda} \left(\frac{\lambda}{\mu}\right)^n$$

Where λ is the mean number of GSR particles on a stub collected from a shooter, while μ represents the mean number of GSR particles present on a stub collected from a non-shooter. Under the conditions in which a case assessment would be conducted, it would be expected that λ is greater than μ , which results in the equation above simplifying to an increasing exponential function of n. That is, as the number of GSR particles detected on a POI increases, the more support exists for H_p . It should be noted that this distribution is favourable to the defence, and therefore can be considered as providing an underestimation, or conservative estimation of the likelihood ratio, and is hence more resistant to potential accusations of prosecutorial bias. From the collected experimental data and the selected probability distribution, their model was used to evaluate the probability of observing n GSR particles on both a shooter and non-shooter. They found that under this model the value of the LR was greater than 1 (favourable to H_o) if two or more GSR particles were detected on the hands after more than two hours. Further, the value of the LR increases significantly in situations where more than 3 GSR particles are detected after more than 2 hours. This implies that the probability of observing 3 or more GSR particles on the hands of a non-shooter is negligible, and therefore this result is much more probable if the individual has had a recent firearm association. Additionally, under this model, it was found that detection of a single characteristic GSR particle after six or more hours also favoured H_p. The authors acknowledge that this assessment considers a relatively narrow selection of factors that may impact a GSR assessment, and to be further confident in results, additional factors such as GSR distribution, persistence and likelihood of contamination should be assessed. Fundamentally, this paper informs the assessment of GSR evidence using a likelihood ratio, by comparing the expected prevalence of GSR particles present on a shooter to that of a non-shooter. This information

can then be used to directly inform the numerator and denominator of a LR calculation respectively. This model, informed by experimental data, does provide a limited assessment of the probability of observing GSR on an individual's hands in light of the two competing hypotheses. However, its singular focus and lack of consideration of other factors influencing the distribution of GSR mean that the findings are not broadly applicable to casework in a variety of situations.

7.2.4. A Bayesian Network approach to GSR evidence

Bayesian Networks (BNs) have been proposed as a means of addressing the lack of consistency in approaches to GSR evidence evaluation. Bayesian networks have historically been explored as a more formalised and logical structure to the evaluation of forensic evidence [232]. This approach has been applied to a variety of forensic evidence types, including DNA [143], fibres [233] and glass [130, 131, 140]. Notable among these are the physical trace evidence types, which exhibit similar limitations of GSR evidence, in that it is difficult to establish a 'base rate' for the initial deposition, and persistence and transfer are key considerations which further complicates assessment of the value of evidence.

Fundamentally, a BN is a graphical model, consisting of a series of 'nodes', where each node represents an event or circumstance for which a set of conditional probabilities can be calculated [230]. The relationship between these nodes can then be demonstrated through use of a series of connections, linking individual nodes into a network. In this way, the number, type, and direct relationships of each node can be clearly visualised, transparently presenting all of the factors considered in the interpretation, and allowing them to be logically evaluated. Further, population of the individual probability tables allows data from multiple sources to be incorporated into the assessment in a systematic fashion. An example network, constructed by Biedermann, Bozza & Taroni [148] to model the experimental data collected by Cardinetti et al. [147] has been reproduced in Figure 63.



Node	Description	States
н	The suspect has shot a firearm (H_p) ,	H_p, H_d
	the suspect has not shot a firearm (H_d)	
S	Experimental setting	s = A, B
Т	Time interval (hours) between firearm	t = 2, 3, 4, 5, 6, 8, 10
	discharge and collection of GSR particles	
Y	Number of GSR particles detected on a sample	$y=0,1,2,\ldots,15$
	taken from the surfaces of the suspect's hands	

Figure 63 - An example Bayesian network for the assessment of GSR evidence and table defining the nodes as presented by Biedermann, Bozza and Taroni [148] informed by data collected by Cardinetti et al. [147]

A core strength of the application of a Bayesian approach to evidence assessment, rather than a frequentist paradigm, is that the Bayesian approach allows an initial evaluation of the probability of an outcome to be updated based on new information. This new information may take the form of additional information received from investigators, or further specifics about the incident under investigation. Though the statistical model proposed by Cardinetti et al. showed promise, the authors acknowledge that the statistical data could be improved by addressing additional variables to more accurately calculate the LR. As a means of approaching this, Biedermann, Bozza and Taroni took the existing data from the Cardinetti et al. study, and incorporated it into a Bayesian network as an alternative structure for performing a probability evaluation, as presented in Figure 63. However, Biedermann, Bozza and Taroni further suggest that the advantage of this approach is in its flexibility, which allows for additional nodes to be considered and incorporated into the probability calculation as required. This resulted in the proposition of an expanded network that can be seen in Figure 64.



Figure 64 - Extended Bayesian network for evaluating GSR particle evidence as presented by Biedermann, Bozza and Taroni [148].

As with any approach, there are limitations to the usefulness of a BN type assessment. Various authors acknowledge that incorporating experimental data into a network also incorporates any sources of uncertainty related to that experimental data [142, 148, 150, 230]. In short, the amount of uncertainty in the evaluation of the LR is contingent upon the cumulative uncertainty from in the data of the individual nodes. However, missing data, or additional data informing the distribution of a particular node, may be accounted for by incorporating additional nodes, informed by experimental data, into the network. In Australia, as a means of addressing this, work on constructing a BN for the evaluation of GSR evidence was conducted by Hales [105], in which further surveys and testing were performed to begin to develop data to inform some of the nodes that contribute to the network. Despite this, the BN approach to the evaluation of the likelihood ratio remains only as strong as the data that informs it. Consistent across various models of a BN approach to evaluating GSR evidence has been the call for more research, and the collection of a substantial enough library of data to ensure that the nodes are well informed [105, 146-150, 234]. The importance of this has been further underlined by Gauriot et al. who note that the BN statistical approach is, to some extent, informed by the subjective choices made by the examiner who constructs the network for GSR evaluation [149]. These authors further caution that the complexity of rendering a statistical model for the evaluation of GSR evidence, coupled with the complexity of the factors influencing the transfer and persistence of GSR ensures that a degree of arbitrariness will be inherent in any conclusions drawn from a statistical model. The value in the use of a BN

approach to the evaluation of evidence is in its ability to clearly define the factors being considered in the interpretation, and the values that they have been assigned. Further, the data underpinning the nodes can be specified, ensuring that all assumptions of the model are communicated. In this way, such a model transparently presents what is being considered in the evidence evaluation.

7.2.5. Proposed Bayesian Model

A slightly modified framework, adapted from that proposed by Biedermann, Bozza, and Taroni [148], used to underpin the discussion of the data collected for this thesis is presented in Figure 65.



Figure 65 – A Bayesian Network for evaluating GSR particle evidence adapted from Biedermann, Bozza and Taroni [148].

The flexibility of a BN approach means that the framework presented in Figure 65 can be expanded to incorporate factors beyond those currently displayed. Additionally, in adapting the framework, factors such as pre-sampling condition of the sample stub have been excluded, while nodes describing the extent of GSR present on the POIs hands due to non-incident related factors have been expanded. Fundamentally, the nodes informing this network model can be separated into three broad categories – Analytical, incident-related, and background factors. These have been colour-coded yellow, blue, and green respectively in Figure 65.

The first category represents factors directly related to the collection and analysis of the GSR sample, and encompass the yellow nodes R, O, I, C, S, α , and β in Figure 65. They can include

the reliability and efficiency of the chosen analytical technique (such as the performance of the instrument) and factors related to the collection of the sample. In this example network, nodes such as α and β may be resolved to incorporate additional factors which inform node S. In this instance, they represent factors that could influence the efficiency of the sampling process, such as differences in the collection of different sample media [229], adhesive on the media [235], or sampling location [113]. Similarly, any number of nodes can be modified contingent on the specific circumstances of the case being considered. Node F, for example, could be further expanded to account for individual factors such as the firearm type [83, 236], ammunition type [70, 206], number of rounds fired [5], and so on. For brevity and ease of display, these have not been included in this network for every node they could inform. Unlike the other nodes in this network, the nodes in this category represent factors that, to some extent, can be influenced by the analyst. This is evident in the fact that the chosen analytical technique, instrumental settings, sampling media, and collection method can theoretically be changed. While practically, individual laboratories may be limited in their ability to modify these, incorporating these nodes into the network model ensures that they are considered, and explained if appropriate.

The second category represents factors related incident under investigation, and are represented by the blue nodes T, H, F, and Y in Figure 65. These nodes represent the factors influencing the probability that GSR will be observed on an individual, based on the specific context and circumstances of the case. Taken together, they influence the amount of GSR that is present on the POI as a result of the incident under investigation, prior to sampling being conducted. It is worthy of noting that the absence of source-level considerations in the current network displayed in Figure 65 should not be taken as an indication that no source level assessment has been performed. Node 'F', which captures the specifics of the conditions of the firearm discharge, represents a simplified presentation of what may be a highly complex evaluation incorporating many elements of source-level interpretation. In many ways, this node could be expanded into its own network, containing further nodes to address factors like primer and projectile composition, weapon memory effect, firearm construction, amongst others. The combination of nodes at T, H and F is then considered cumulatively at node Y. As these factors are highly specific to the circumstances around the firearm discharge under investigation, it is beyond the ability of the analyst to directly evaluate them or influence them in any way. However, data to form estimates informing these nodes may be collected through careful test firings with the specific firearms and ammunition used in the incident under investigation, or additional modelling based on research from the literature. For example,

research into the distribution of GSR originating from different ammunitions and firearms can be used to evaluate what findings may be expected under conditions similar to those experienced in the incident under investigation. Caution here is warranted, even under the best case scenario, it is unlikely that all of the specific circumstances of the incident under investigation will be known, or will be reproducible. To that end, any GSR assessment of these nodes represents an estimate, rather than a certainty. In framing this model, the two proposed hypotheses, represented at node H, are:

Hp: 'The POI has had some involvement in the firearm incident under investigation'Hd: 'The POI has had no involvement in the firearm incident under investigation'

These are activity level hypotheses, in that they propose to evaluate the activities of an individual involved. It is important to note that involvement has specifically not been defined as discharging a firearm, and may therefore incorporate discharging a firearm, being present while a firearm was discharged, attending a scene shortly after a firearm discharge, or handling a firearm, spent cartridge cases, or other items contaminated with GSR. Typically, the pertinent hypotheses are determined by the circumstances of the case, and the claims put forward by the prosecutor and defence. However, in evaluating the significance of the evidence, caution must be applied to ensure that the hypotheses under consideration are appropriate [226]. Under the proposed model in Figure 65, in the event that H_d is supported (i.e., node $H = H_d$), it would be expected that node Y would resolve that the probability of observing GSR is zero. This can be better understood as representing the situation in which an individual truly had no involvement with the incident under investigation, the probability of observing GSR present on them as a result of the circumstances under investigation is zero.

The final category contains background-related factors, which are represented in Figure 65 by nodes PB1, T1, B1, and X. These nodes represent factors influencing the probability of observing GSR on an individual as a result of factors unrelated to the incident under investigation, and encapsulate the likelihood of GSR being present as a result of random background, as well as the possibility of cross-contamination from law enforcement. While, like the incident-related nodes, it is beyond the ability of the analyst to directly influence these nodes, they can be assessed more readily. For example, the GSR background of a population can be evaluated using random man studies as a way to assess the probability that a randomly selected individual from that population will have GSR present on them. Intuitively, it can be understood that the presence of GSR on a POI's hands comprises that which has been deposited from the firearms incident under investigation, the contribution of any GSR background, and any GSR that may have been transferred due to contact with law enforcement during apprehension and arrest. Contextualising this in terms of the presented BN, it can be seen that node P is equal to the sum of the values at node X and node Y. In the event that H_d is supported, and node Y resolves that the probability of observing GSR is zero, it follows that any GSR that may be present is as a result of the factors informing node X. Therefore, in a true false positive scenario in which a POI is apprehended and sampled for GSR, but has had no involvement with the incident under investigation, the value of node X is directly related to the denominator of the LR, in that it represents the probability of observing characteristic GSR on an individual, given the fact that H_d is true. Even when not incorporated directly into a BN framework for the assessment of evidence, developing an understanding of the probability of observing a certain amount of GSR on an individual as a result of factors unrelated to the incident in question provides valuable information to the analyst as a means of establishing the significance of a finding. The nodes in this category have been the primary interest behind this thesis. It is however, worth noting that at this stage, these nodes have been evaluated in broad consideration of the Australian police and population. To that end, this network model considered characteristic particles only. The work performed in Chapter 3 and Chapter 4 indicated that characteristic particles can be generated from ammunitions using two component primers where Sb is present in the projectile. However, it could be foreseen that there are some situations where an ammunition does not produce characteristic particles, and instead generates a population of consistent particles only. It is further acknowledged that in other jurisdictions, specific consideration of particles generated from other primer formulations, including HMF primed ammunition, may be more pertinent, and the proposed network may be less appropriate in these areas. With this in mind, the proposed network structure does not claim to be an exhaustive model that is suitable for all GSR evidence evaluations, but rather a step on the path of strengthening evaluation of GSR evidence.

This chapter draws together previous research conducted in assessing some of these factors, and combines them into a BN model to assist in the interpretation of GSR evidence. Of specific interest are the nodes related to the probability of observing GSR on an individual that has had no involvement in a firearm incident, as this can be seen to be analogous to the probability of observing a false positive. To accomplish this, factors including the GSR background in the random population, GSR background in the police population, and dynamics and probability of transfer were evaluated. However this model serves only as a tool to assist in the interpretation of GSR evidence, and not as a strict metric on which all evidence should be judged. It is important that evidential findings be placed in the appropriate context, and that a rigorous case-by-case assessment is used to ensure that all GSR findings are assessed appropriately.

7.3. MATERIALS AND METHODS

7.3.1. Data Collection and Analysis

Construction of the network model was programmed using HUGIN Lite, Version 8.6 (<u>http://www.hugin.com</u>), a software package designed for the construction and evaluation of Bayesian networks.

The adapted network present in Figure 65 was incorporated into the HUGIN Lite software, and can be observed in Figure 66.



Figure 66 - Adapted Bayesian Network model for the assessment of GSR evidence created in HUGIN Lite.

All data informing the network were collected as described in the previous chapters and publications. In all cases, ASTM E1588-17 [13] was used to classify detected particles as either 'characteristic' or 'consistent with' a firearms origin.

The research conducted as a part of this chapter has primarily been focussed at the factors informing node X from Figure 65, that is, the likelihood of observing GSR unrelated to the incident under investigation on the hands of an individual as a result of the background prevalence of GSR in the random population, combined with any secondary transfer due to

direct contact with law enforcement. This section of the adapted network can be seen in Figure 67.



Figure 67 - Segment of the adapted Bayesian Network evaluated as a part of this research Data informing the 'GSR Background in the Random Population' node (B1) were calculated on the basis of the observations and data collected and reported in detail in chapter five, and presented in a previous publication [209].

Data informing the 'Police Background' node (PB1) was based on observations and data collected and reported on in detail in chapter six, and presented in a previous publication [237].

Data informing the 'Transfer' node (T1) was based on observations and data collected and reported on in detail in chapter six, and presented in a previous publication [237].

7.4. **RESULTS**

7.4.1. Node B₁ – GSR Prevalence in the Random Population

Values at this node represent the probability that a randomly selected member of the random population will have particles of characteristic GSR present on their hands. These data therefore may be used as a prediction of the prevalence of characteristic GSR on an average member of the Australian metropolitan population.

From this previous survey, it was observed that characteristic GSR particles were present on a single individual in a sample of 289 individuals across two Australian jurisdictions. This single individual had three characteristic particles recovered from their hands. It should further be noted, that the data used for this research makes no judgement on the origin of these particles, only that they would be considered characteristic of GSR under the ASTM, and the

subject from whom they were collected declared no firearms contact. These data were then used to calculate the probabilities that inform the B_1 node.

If the probability of observing particle numbers was calculated on the basis of the raw observed particle frequency data alone, then the probability of observing 3 particles is 1/289 (0.35%). However, this means that the probability of a random individual having less than three, but more than nil, characteristic particles present on their hands is zero. It is known that GSR particles are lost from the surfaces on which they are deposited over time. Given the fact that three characteristic particles were observed on the hands of one individual, it can be inferred that had they not been collected as a part of the GSR sampling, at some time later this individual would have had two or one characteristic GSR particles on their hands. Conversely, it can be inferred that at some time before the GSR sample was collected from this individual, that they had a larger particle population on their hands, up to whatever the maximum number of particles that was originally transferred. However, without specific knowledge of what this number of particles was, a maximum value cannot be predicted. For the purposes of this model, in the absence of other evidence, it was assumed that the three particles detected on this individual represented the maximum value. Therefore, in incorporating these data into the model, it was inferred that the probability of observing any number of GSR particles up to the maximum number observed (in this case, 3) was equal. While this is likely to over-estimate the probability of observing characteristic GSR in the random population, it does so in favour of H_d. That is, it results in a marginal increase in the probability of observing characteristic GSR on the hands of a randomly selected member of the Australian population, which then informs the denominator of the likelihood ratio calculation.

Therefore, with this in mind, the probability values for characteristic GSR prevalence in the random Australian population that can be used to inform node B_1 in the BN can be seen in Table 56.

N(GSR PRESENT)	PROBABILITY
0	98.962
1	0.346
2	0.346
3	0.346
4	0.000
5	0.000

Table 55 - Calculated probabilities for observing characteristic GSR on the hands of a POI due torandom GSR background.

From Table 56, it can be seen that there is a small, but non-zero, probability of detecting a small number of characteristic GSR particles on an individual randomly selected from the Australian population who has no declared firearm exposures. While these data suggest that the most probable outcome is that nil characteristic GSR particles will be detected on an individual, this suggests that caution should be used in the interpretation of GSR examination results in situations where small numbers of characteristic particles are detected.

7.4.2. Node PB₁ – GSR Prevalence in the Police Population

Values at this node represent the probability that a randomly selected, firearm carrying, general duties police officer will have some number of characteristic GSR particles present on their hands. This then forms a potential particle population that could then undergo secondary transfer to the hands of a POI.

From the previous survey of police officers, it was determined that characteristic GSR particles were observed on the hands of six serving police officers in South Australia from a total sample population of 76 officers. The maximum number of particles observed on a single individual was 12, with the remaining five individuals only having a single characteristic particle present each. As expected, by virtue of the fact that police officers carry a firearm as a part of their operational duties, characteristic GSR particles were observed comparatively more frequently on officers hands than they were in the random population. To that end, these data were then used to calculate the probabilities that inform the PB₁ node.

As with the random prevalence survey, when calculating the probability of observing a number of characteristic GSR particles, the potential contribution of particle loss over time was incorporated. In this instance however, there were additional members of the sample population who were observed to have a single characteristic GSR particle on their hands. Therefore, the calculated probability was evaluated by incorporating the probability of observing a single particle as a result of particle loss from a larger population, as well as the probability of observing a single particle based on the collected data.

The probability distribution for characteristic GSR particle prevalence on the hands of police officers that can be used to inform node PB_1 can be seen in Figure 68.



Figure 68 – Probability distribution for observed characteristic GSR particles on the hands of serving South Australian Police officers [237].

7.4.3. Node T₁ – GSR Transfer During Arrest

Values at this node represent the probability of observing some number of characteristic GSR particles present on the hands of an individual as a result of transfer due to contact with nonfiring, but firearm carrying, law enforcement, comparable to undergoing brief detainment and arrest. It is pertinent to note at this juncture that this assumes that there has been no use or manipulation of the firearm by the police officer undertaken in the process of arrest. Understandably, if the police have handled, or discharged their firearm shortly prior to contact with a POI, the likelihood of cross-contamination is much higher, as it is more probable that the officer will have a significant amount of GSR present on their person which could then be transferred. This node therefore aims to assess the likelihood of transfer which may occur under ordinary conditions, where the only handling of the firearm by the police which has occurred is as a result of receipt of their firearm at the start of the shift. In order to assess this, this node has been informed by the previous node, PB₁, as the amount of GSR transferred is contingent on the population present to begin with. Therefore, data informing the 'Transfer' node was based on observations and data collected and reported on in detail in chapter six, and presented in a previous publication [237].

Secondary transfer modelling was conducted under mock-arrest conditions designed to replicate the conditions of arrest, where transfer between a police officer and a POI may occur. As expected, there was a significant amount of variability observed in secondary

transfer efficiency, with the maximum amount transferred being 92% of the total particle population, and the minimum amount being 0.5%, with a median transfer efficiency of approximately 27%. This variability suggests that it is likely that the extent of secondary transfer is heavily contingent on the type, duration, and nature of the contact. However, these data can still usefully inform a model to assess the probability of secondary transfer under arrest conditions. The distribution of the probability of percentage secondary transfer efficiency, based on the observed data, can be seen in Figure 69.





The data collected related to the observed secondary transfer efficiency as presented in Figure 69 were then used in conjunction with the police background data (Node PB₁), as presented in Figure 68, as a means of evaluating the probability informing node T₁. The results of this evaluation can be seen in Table 58 - Calculated probabilities for observing characteristic GSR on the hands of a POI due to the GSR background in the random population, and secondary transfer from police during apprehension and arrest.Table 57 and Figure 70.

n(GSR Present)	Probability
0	9.97E-01
1	9.51E-04
2	5.87E-04
3	3.64E-04
4	2.98E-04
5	2.40E-04
6	1.82E-04
7	1.49E-04
8	1.16E-04
9	6.62E-05
10	4.96E-05
11	3.31E-05
12	1.65E-05
13	0.0

 Table 56 - Calculated probabilities for observing characteristic GSR on the hands of a POI due to the secondary transfer from police during apprehension and arrest.



Figure 70 - Probability distribution for observing one or more characteristic GSR particle present as a result of secondary transfer from the hands of general duties police officers.

Table 57 and Figure 70 display the probability of characteristic GSR particles being observed on the hands of an individual as a result of secondary transfer from a general duties police officer in South Australia. It can be observed that the probability of observing one or more characteristic GSR particles under these conditions is low, and the most probable outcome is that no particles are detected (P=0.997). This may be attributable to the low probability of observing a large population of characteristic particles on the hands of serving police (at node PB₁), or the highly variable extent of transfer, which is contingent on the type and duration of contact.

7.4.4. Node X – GSR Present

Values at node X therefore represent the probability of observing some number of characteristic particles on the hands of an individual as a result of the combined factors of nodes PB₁, T₁ and B₁. This therefore represents the probability of observing characteristic GSR particles on the individual as a result of the GSR background in the random population, combined with the impact of GSR particles transferred from the hands of police officers under the conditions of apprehension and arrest. The calculated probability values at this node can be observed in Table 58 and Figure 71.

Table 57 - Calculated probabilities for observing characteristic GSR on the hands of a POI due to theGSR background in the random population, and secondary transfer from police during apprehensionand arrest.

n(GSR Present)	Probability
0	9.69E-01
1	1.35E-02
2	9.10E-03
3	5.03E-03
4	1.17E-03
5	8.66E-04
6	6.22E-04
7	4.38E-04
8	2.87E-04
9	1.69E-04
10	1.02E-04
11	5.14E-05
12	1.76E-05
13	5.79E-07
14	2.32E-07
15	5.79E-08
16	0.0


Figure 71 - Probability distribution of characteristic GSR particles due to background prevalence and secondary transfer due to contact with police

From the collected data presented in Table 58 and Figure 71, the theoretical maximum of observed characteristic particles is 15, which represents the situation in which three particles are present due to the random GSR background, while 12 particles are present due to secondary transfer from the hands of police. Under the conditions of this study, the probability of this outcome was evaluated to be extremely unlikely (P=5.79x10⁻⁸). On a practical level, this predicts that this outcome could be expected once in 25 million iterations of these conditions. The combined data indicate that the most probable outcome is zero particles being observed (P=0.969). It can further be inferred therefore that the probability of observing one or more particles on an individual as a result of either background population or secondary transfer from police is low (P=0.031), suggesting that this could be expected to occur from approximately 3% of individuals sampled. Comparatively, two or more particles could be expected from 1.8% of individuals sampled, while 3 or more particles could be expected from less than 1%. In context, these data support the view that, while highly improbable, the possibility of observing GSR particles on an individual as a result of the random GSR background and transfer from law enforcement exists, and should therefore be considered as an element of GSR evidence evaluation.

7.5. DISCUSSION

The constructed model operates under a number of assumptions. Transfer, in particular, was modelled under worst case scenario conditions. That is, the 'officer' had discharged a firearm very shortly before contact between the two parties. Additionally, the contact between the two was relatively prolonged, with the POI being restrained over an extended period before being mock-handcuffed. These conditions are unlikely to be truly representative of real world circumstances in which GSR evaluation would be considered. However, these conditions ensured that an adequate population of GSR particles was present in order to model the dynamics of secondary transfer and to minimise stochastic effects. The model therefore assumes that the dynamics of secondary transfer for smaller numbers of GSR particles are comparable to that of the larger populations modelled. The likely impact of this on the wider model is that the extent of secondary transfer that may have occurred between the parties.

Similarly, the GSR background in the random population was collected under specific circumstances, and is therefore somewhat limited. Specifically, the survey sample was collected in two Australian capital cities and surrounding regions, both metropolitan areas, meaning that the sample is representative of a population which has comparatively low private civilian firearms ownership and use [8, 9]. The bulk of the Australian population is resident in metropolitan areas and it may be reasonably expected that these data would therefore be applicable to the majority of firearms investigations. However, it could be predicted that regional areas, where firearms are more frequently owned and used for hunting or agricultural purposes, would exhibit a different and perhaps higher background. To that end, although the random GSR particle background data are likely to be useful for a large number of GSR evidence evaluations in Australia, it cannot be claimed to be broadly representative for all populations. Likewise, it would be expected that the observed background values would not be representative of other international populations, particularly those which have significantly higher private civilian firearms ownership.

Finally, this model assumes that the hands of the POI are either sampled or protected to minimise particle loss (by using nitrile gloves for example) shortly after they are arrested. Failure to adhere to this introduces further considerations related to additional particle loss due to time, or the possibility of additional cross-contamination due to contact with police facilities or vehicles. Additionally, it can be inferred that the application of gloves will preserve GSR, either on the hands, or the gloves themselves, but this distribution has not been modelled as a part of this work.

Despite the fact that the survey of the characteristic GSR background on the hands of serving police officers indicated that the probability of observing GSR was greater than that of the random population, when secondary transfer is considered this did not translate to a significantly increased probability of observing GSR on the hands of a POI who has had contact with police. The dynamics of particle transfer are highly contingent on the type, nature, and duration of the contact. Although having a larger particle population in the first place increases the likelihood that more particles will undergo transfer (by the very nature of the fact that they are available to be transferred), the variable nature of the dynamics of transfer makes this far from certain.

It is further pertinent to acknowledge that this forms a preliminary work in utilising experimentally collected data to inform a BN approach to the assessment of GSR evidence. It is therefore recognised that this model is not exhaustive, nor is it intended to be the only tool used in the assessment of GSR evidence. The importance of careful consideration of the factors related to the incident under investigation (those informing node Y in Figure 65), especially those informing source-level evaluations must not be overlooked. As was discussed in Chapters 3 and 4, there is often significant complexity in performing source level evaluations, which may be contingent on diverse factors such as primer composition, projectile composition, the type and structure of the firearm amongst others. The influence and significance of these factors on the resultant GSR is highly dependent on the circumstances and context of each case. To that end, although the proposed network can be used to support GSR evidence evaluation, it should be used in conjunction with a cautious case-by-case approach.

However, at its core, the proposed network seeks to inform the denominator of the likelihood ratio evaluation, that is, they inform the probability of observing some number of GSR particles on the hands of an individual, given that they exhibited no involvement in the situation under investigation. Factors informing this assessment, including the background prevalence of GSR in civilian and police populations, would be expected to be relatively enduring. The current research therefore provides some important context on the probability of observing a false positive GSR test as a result of both background and law enforcement contact.

The purpose of this model was to contribute towards a framework for evidence assessment that allows for some estimate of the likelihood of obtaining a false positive result in GSR analysis, and the impact this that may have on a Bayesian interpretation of the value of said evidence. Even if not applied to a BN style interpretation of GSR evidence, the calculated probabilities, supported by the experimental data, allow a GSR analyst to better contextualise the significance of a finding.

7.6. CONCLUSIONS

A structured network-type approach was used to inform the evaluation of GSR evidence. A proposed Bayesian Network adapted from that proposed by Biedermann, Bozza & Taroni was used as a means of illustrating factors which may be considered as a part of a GSR evaluation [148]. Of particular interest were those nodes which may be used to inform the probability of observing GSR on an individual who has had no involvement with the matter under investigation. This data may then be used to inform the denominator of the likelihood ratio. To accomplish this, data collected from previous surveys, including the characteristic GSR background in the random Australian population, the background on the hands of serving police officers, and the estimated extent of secondary transfer under apprehension and arrest conditions, were used to inform the nodes pertinent to this assessment.

It was noted that the probability of observing large amounts of characteristic GSR particles on an individual as a result of the random background and secondary transfer from law enforcement was low. The most probable outcome was zero particles being observed on a POIs hands (P=0.969) as a result of this activity, with the probability of observing one or more particles being P=0.031. Practically, this suggests that one or more GSR particles being present on an individual as a result of these factors would be expected to be observed from approximately 3% of individuals from which a GSR sample was collected. Further, 3 or more characteristic particles as a result of these factors would be expected from less than 1% of individuals.

Regardless, although there is promise in applying a Bayesian probabilistic approach to the evaluation of GSR evidence, great care must be applied to assessing the factors related specifically to the incident under investigation. Even if these factors are assessed under the best possible circumstances, additional refinement may be needed to be reflective or representative of the circumstances of the particular incident. Therefore, any such evaluation represents an estimate only, rather than a definitive rule. Despite this, these data may be used to support an evaluative process for the assessment of GSR evidence in Australia.

A Framework for the Evaluation of GSR Evidence | Chapter 7

8.CONCLUSIONS AND FUTURE WORK

8.1. CONCLUSIONS

The ultimate goal of the research conducted as a part of this thesis was to investigate various approaches to provide additional evidential value to GSR evidence assessment. This objective was addressed by sequentially assessing a number of key factors related to the assessment of GSR evidence in South Australia.

GSR casework performed in South Australia between 1998 and 2016 was reviewed, with data related to firearm types, ammunition products, and GSR results collated and assessed. These data were then used to understand the most frequently encountered firearms, ammunitions and particle types in routine casework. As a part of this review, it was observed that three-component, characteristic particles were observed with a frequency that was unexpected, given the more frequently encountered ammunition types. This observation directed further investigation of the mechanisms underpinning GSR particle formation, and the influence of the weapon memory effect.

These further investigations took the form of targeted test-firings using different ammunition products as a means of evaluating how different components of the primer and projectile were distributed into the resultant GSR. Further exploration of this was performed by collecting GSR particles from a number of different ammunition products, including HMF varieties, and cross sectioning the particles using a focussed ion beam (FIB). The sub-particle compositional and morphological features were then assessed using x-ray mapping. The FIB was also used to prepare thin-sections of GSR particles to be analysed using alternative techniques.

A series of additional surveys and experiments were conducted as a means of further supporting an evaluative assessment or further statistical evaluation of GSR evidence. To that end, the random prevalence of GSR in the random Australian population was assessed by collecting samples from the hands of members of the public in three different Australian metropolitan jurisdictions. A similar survey assessing the background prevalence of GSR on the hands of randomly selected SA police officers was performed and compared to the random population survey. Finally, the potential dynamics of secondary transfer from police officers to the hands of a member of the public under mock apprehension and arrest circumstances was modelled. The combination of these factors speaks to the probability of observing a false positive GSR result, and can therefore be utilised to evaluate the denominator of the likelihood ratio equation. Therefore, these three sets of observations were incorporated into a section of Bayesian network model for the evaluation of evidence.

8.1.1. Firearms encountered in forensic casework in South Australia

A comprehensive review of cases in South Australia involving GSR analysis allowed for several interesting trends to be observed. This study provided data regarding the frequency that different firearms and ammunitions were encountered in forensic casework. A review of 71 suicide cases occurring in South Australia between 1998 and 2014 were interrogated for their GSR data, as well as the firearms and ammunition used. The most frequently observed firearm type in these cases were rifles, followed by handguns and shotguns. The most prevalent calibre was 0.22LR which featured in more than half of the cases considered. With regard to GSR findings, it was observed that over 47% of the cases considered produced GSR results of low probative value, in that either little or no characteristic GSR was located, despite the fact that it was known that the individual sampled had discharged a firearm.

To develop a more comprehensive picture of the types of firearms encountered in all crimetypes in South Australia, a further review of all cases in which a GSR analysis was performed between 2007 and 2016 was conducted. Rifles were the most frequently encountered firearm type, followed by shotguns, and then handguns, which generally correlates with their availability under Australian firearms legislation. In this survey, GSR particle type data were also interrogated to discover how frequently additional elements were incorporated into GSR particle compositions. It was observed that on average, Si was present in approximately 77% of particles observed, appearing the most frequently in particles originating from rifles, of which 87% had some level of Si present. Further, Al was similarly frequently observed, occurring in 71% of particles on average, but most frequently observed in particles originating from shotguns, at 89%. Fe and Cu were seen at some level, on average, in approximately 50% of the particles. The Si finding was of particular interest, given recent exploration into the usefulness of glassy GSR (gGSR) [52, 130, 158-160].

In both sets of data, 0.22LR rimfire ammunitions were among the most frequently encountered ammunition types. It is known that the majority of rimfire ammunition primer contains only Pb and Ba, and this is the case for Winchester rimfire ammunition, which was the most frequently encountered rimfire ammunition involved the shooting cases examined. Despite this, three-component, characteristic GSR particles were observed more frequently than would be expected in shootings that involved rimfire firearms. Conventionally, observations such as these have been attributed to the weapon memory effect, and the GSR from previous firings of three-component primed ammunition retained within the firearm being distributed in subsequent firings with two component primed ammunition. However, the data suggest that usage of rimfire ammunition with PbBaSb-containing primer was rare in the shooting events and it would be unusual if previous a substantial number of previous shooting events would involve ammunition with 3-component primers. This suggests that there was some other mechanism responsible for the detection of relatively high numbers of particles containing Sb.

8.1.2. Sub-particle composition and morphology of GSR

While it was known that GSR particles from firings of previous ammunitions can contribute to the particle population of subsequent findings via the weapon memory effect, the data collected from the review of GSR cases in South Australia had suggested that the process behind the formation of some particles is more complex. One proposed mechanism was the incorporation of Sb (or Sb and Pb) from the surface of the projectile into Ba (or PbBa) particles generated from two-component primed ammunition. To investigate this, a number of further experiments were conducted to investigate the impact of the weapon memory effect, and how that may impact the composition of GSR particles.

Test firings were conducted using a 0.22LR rimfire rifle, with two different ammunitions. The first, CCI Stinger, was known to possess a two-component primer compound, but had a Pb only projectile, and therefore did not contain Sb in any part of the ammunition cartridge. The second, PMC Zapper, also possessed a two-component primer, but had a Pb and Sb containing projectile. Both ammunitions were fired from a cleaned and conditioned firearm, and particles from the barrel and breech were captured and analysed. When comparing the barrel residues from both ammunitions, PMC Zapper was observed to generate many times more particles containing Sb than the CCI Stinger ammunition. The CCI Stinger ammunition did generate a small number particles containing Sb, suggesting that even with cleaning and conditioning, the weapon memory effect can still make a contribution to a particle population. However, these findings suggest that there are other complexities in the formation of GSR particles that may exert a greater influence than the weapon-memory effect that must be considered as a part of GSR evidence evaluation.

As a means of further evaluating this hypothesis, a focussed ion beam (FIB) was used to crosssection GSR particles with view to applying different analytical techniques in order to identify the source of the Sb in these particles. This process was also applied to GSR originating from a number of different ammunition products, including HMF ammunitions and different primer formulations as a means of investigating if sub-particle morphology could provide additional data about the ammunitions from which they originated. Although the process of cross-sectioning of the GSR particles was ultimately successful, the resultant thin-sections were fragile and difficult to manipulate in order to secure them for transport and further analysis. A notable exception to this were particles of glassy GSR (gGSR), in which the glass matrix was hardier and more resilient, making securing the thin-sections for further analysis more successful. The results of this have been reported on elsewhere [159]. Despite this, the cross-sectioned particles were able to be imaged and x-ray mapped, to obtain additional information about the sub-particle morphology and composition of the GSR particles.

In the case of 0.22 PMC Zapper ammunition, the cross-section revealed that the elements of interest were not homogenously distributed throughout the particle. Regions of higher concentration of Pb were observed in the centre of the particle, while Ba was more sparsely distributed across the particle. Sb, where it was present, was observed to be co-located with Cu, and was observed in discrete regions across the particle. The absence of Zn observed in the particle suggested that the source of the Cu in these regions was from the Cu wash over the projectile, rather than the cartridge brass. While the impact of the weapon-memory effect could not be entirely excluded, these observations support the notion that the Sb originated from the surface of the projectile.

The opportunity arose to use FIB to examine GSR arising from the discharge of a small range of high calibre ammunition. With regard to the sub-particle morphology and composition of GSR originating from other ammunition types, the particles observed ranged from a solid, with a homogenous mix of elements (0.40 American Eagle MFP) to sponge-like, with many internal voids (0.40 Winchester Winclean). In some cases, heterogeneous distribution of the component elements was observed, ranging from distinct nodules of Pb distributed in a Ba and Sb matrix (0.40 Federal Premium HST), to more complex separation of all three components in a distinct arrangement (7.62x39 Norinico). While this survey considered a comparatively narrow selection of ammunition products, the results suggest that there were observable morphological and compositionally differences on the sub-particle level that could serve to provide additional information to a GSR evidence evaluation.

While use of a FIB process could be utilised to create thin sections of GSR particles that could then be used for more detailed characterisation of their internal composition using alternative

techniques. It does appear however, that the success of this process may be highly contingent on the matrix and internal structure of the GSR particle. More robust matrices (such as the glass component of gGSR) and those particles with solid interiors withstand the sectioning and transportation process much better, while fissured particles or those with internal voids are less amenable to this technique. Although FIB-sectioning can be applied successfully, there is a chance that application of this technique will result in samples being lost. Further, at this stage the sectioning process is time consuming and inconsistent, which can also result in sections being lost. Although the practicalities of using FIB for the analysis of GSR, including the time, cost, and limitations of the technique, suggest that it has relatively narrow casework applications, this research suggests that it exists as an option that can be used to obtain additional information from GSR in some cases and for future research. Further exploration of the weapon-memory effect indicated that while GSR retained within the firearm from previous discharges contributes to the particle population of subsequent discharges, it is evident that this is not the sole explanation nor perhaps even the most relevant explanation. Test firings and FIB cross-sectioning suggests that under some circumstances, elements from the surface of the projectile are incorporated into particles of GSR. This is particularly important in situations where 0.22LR rimfire is used, as the lack of Sb compounds in the primer of many of these ammunitions has resulted in situations where observing three-component, characteristic GSR is unexpected, as it is ostensibly logical that GSR particles derived from these primers do not generate these particles. As revealed in the surveys of GSR cases in South Australia, cases involving 0.22LR ammunitions are among the most frequently encountered. With this in mind, these findings have the potential to better contextualise observations involving 0.22LR ammunitions that are frequently encountered in South Australian casework.

8.1.3. GSR prevalence in the random population

As a means of assessing the prevalence of particles indistinguishable from GSR particles in the randomly selected Australian population, samples were collected from the hands of 309 individuals across three Australian jurisdictions in the largest survey of its kind to date. Ultimately, three particles indistinguishable from characteristic GSR were detected across all samples, representing a frequency of 0.3%, while particles that would be identified as consistent with GSR were observed with a frequency of 13.8%. These data further underscore the current understanding of the prevalence of GSR-like particles in the random population, in that characteristic particles are very infrequently observed, even in situations where firearms associations have been declared. This therefore supports the view that if particles having a composition characteristic of GSR are observed on the hands of an individual, then it is

probable that that individual has had a relatively recent firearm association, or close contact with an individual who had a recent firearm association. In short, this understanding of the background frequency of particles indistinguishable from GSR enhances the ability of forensic scientist to contextualise GSR evidence, and subsequently draw more reliable conclusions, informed by data, about the significance of a finding. Understanding the relative frequency of GSR in the population allows analysts to better contextualise and communicate the strength of their findings. For example, if questioned about the probability of observing GSR on the hands of a random individual in court, these data allow a frequency to be applied, rather than simply stating that such an observation is unlikely. In this fashion, the findings reinforce the value of finding GSR evidence on a POI to an investigation. Secondly, the collected frequency data can be incorporated into a more formal statistical assessment of the value of GSR evidence using a Bayesian Network (BN) type approach.

The data however, are not without their limitations. One particular drawback was that this survey was conducted over a number of years, during which time the firearms market has evolved. None of the surveys considered particles generated from HMF, green, or Pb-free ammunitions. As these ammunition products gain popularity in the firearms market, future population studies should be performed, to similarly provide context and inform the significance of a GSR finding. A further limitation of the data is the fact that these surveys were performed in Australian metropolitan areas only. The majority of the Australian population live within metropolitan areas, and the bulk of firearms crime occurs in these regions, therefore it would be anticipated that this data would be useful in informing the majority of GSR casework. However, it cannot be stated that these conclusions are applicable in all circumstances. It should be further noted that it would be expected that the prevalence of firearms ownership and use might be higher in rural and regional areas. To that end, it may be anticipated that the GSR background in these areas may be higher than was observed in this survey. Similarly, it would be anticipated that the findings of this survey would not necessarily be applicable internationally in regions where private firearms ownership, firearms hobbies, or military or police service is more frequent, where the GSR background may be higher. Fundamentally, these data collected as a part of this survey have value in two key ways. Firstly, understanding the prevalence of GSR in the random population allows for a GSR examiner to perform an informal evaluation of the value of GSR evidence.

8.1.4. GSR prevalence in the police population

Previous surveys of particles on the hands of police officers immediately after receiving their firearm at the start of shift indicated that this represents a significant source of GSR particle contamination [101]. Besides this, it would be expected that the GSR background on the hands of police officers would be higher than that of the random population, due to the fact that they routinely handle, wear, and use firearms as a part of their job. While similar surveys have been performed in different international jurisdictions, policy and procedure around how firearms are stored, used, and handled may differ. Therefore, a survey of the hands of serving South Australian Police (SAPOL) officers was performed. Unlike the previous survey by Cook [101], the samples informing this survey were collected from police officers at various points across the day shift, in order to ascertain if GSR persisted within the population.

The results of the survey indicated that as expected, the frequency of GSR on the hands of police (8% of samples had particles indistinguishable from characteristic GSR present) was higher than the random population (0.3% of samples had characteristic GSR present). However, where GSR was observed on police officers, it was seen in small amounts, with approximately 3 particles being the average observation, and a maximum of 12 observed on a single officer. This suggests that although GSR may be present on the hands of police, it does not represent a major population from which significant amounts of secondary transfer may occur. While this may not be a major concern, it is still not a negligible result, suggesting that this is a factor that should be considered in GSR evidence evaluation.

The value in this finding as it pertains to better contextualising GSR evidence is two-fold. First, it provides a useful counter-point to the previous study by Cook, in demonstrating that although receipt of their firearm results in transfer of significant quantities of GSR, these levels are not maintained across the officers' shifts. This indicates that anti-contamination measures (such as washing hands after receipt of firearm) are effective, and that re-contamination, such as by handling the firearm throughout the shift, is not occurring. Therefore, although a randomly selected SAPOL officer may have GSR present on them, it is not likely to be a large population. Secondly, the collected data forms one part of a model for cross-contamination, or secondary transfer to a POI due to contact with police. In order to model GSR on a general duties police officer must first be modelled. In this fashion, data collected from this survey may be combined with estimates of secondary transfer, to better inform the probability of GSR being deposited on a POI during apprehension and arrest by a typical SAPOL officer.

Secondary transfer experiments and the results obtained are discussed more fully in the next section.

Further data were collected directly from two of the types of ammunition most frequently used by SAPOL officers – HMF Training ammunition and a three-component primed operational ammunition. This served the dual purpose of developing further understanding of the types of GSR generated by these ammunitions, and, with regard to the HMF ammunition, potentially using specific particle types as a *de facto* indicator of possible cross-contamination from police. In this case, it was identified that that operational ammunition generated a particle population that was consistent with the discharge of a conventional three-component primed ammunition, and was therefore not observed to generate any particles that could be considered characteristic of this particular ammunition. The HMF training ammunition demonstrated an absence of particle-types considered characteristic of HMF GSR, but was observed to produce Sr- and SrAI- containing particles. These have relatively abundant non-firearm sources, and therefore are only considered consistent with HMF GSR. However, identifying SAPOL training ammunition as an additional source of these particles suggests that locating particles of this type in a case sample should be viewed with caution in interpretation.

Special units of the police were observed to have many different particle types retained on their gloves and equipment. These included conventional three-component GSR, HMF GSR consistent with SAPOL training ammunition, and indications of particles consistent with those generated by flash-bang grenades, along with many other non-GSR particle types. This suggests that, consistent with previous work performed overseas [73], the equipment of special units of SAPOL may pose a significant cross-contamination risk if they come into contact with a POI. As a result of this, the involvement of these units in an arrest requiring a GSR analysis is an important consideration in GSR evidence evaluation. However, given the relative scarcity of HMF ammunitions and flash-bangs in civilian populations, identification of particles originating from one of these sources on a case sample may serve as a *de facto* indicator that cross-contamination may have occurred.

8.1.5. Secondary transfer during apprehension and arrest

Once an estimate for the GSR background on the hands of SAPOL officers was established, an assessment of how much of this background may undergo secondary transfer to the hands of a POI given activities conducted during apprehension and arrest. The median transfer efficiency observed under these conditions was 27%, but significant variability was observed, with the minimum transfer representing 0.5% of the total particle population transferred and the maximum value observed 92.3%. However, transfer of less than 25% of the particle population was the most frequently observed, suggesting that the most probable outcome is transfer of a comparatively small portion of the particle population. It must be acknowledged that the variation observed in the data is a reflection of the random factors influencing the initial deposition of GSR. Similarly, as well as the quality and amount of GSR that exists as a population to be transferred, it is likely that the amount of secondary transfer is highly likely to be dependent on the type, duration, and nature of the physical contact. The combination of these factors means that the use of these values to inform a predictive model of how GSR may undergo secondary transfer represents an informed estimate only.

From a holistic perspective, it has been well documented that secondary transfer of GSR can occur when two individuals come into physical contact, such as by shaking hands [91] or through direct contact with police [73]. The conditions of the contact in this survey were intentionally designed to model transfer of GSR from SAPOL ammunitions and firearms, if SAPOL apprehension and arrest procedures were followed. While the results can only be claimed to represent an estimate of the most probable extent of secondary transfer that may occur under these conditions, they may still be used to inform a probabilistic interpretation of GSR evidence.

8.1.6. A logical framework for the assessment of GSR evidence

An ongoing challenge in the assessment of GSR evidence has been in the shortage of methodology with which to evaluate and express the significance of a particular finding. Various approaches to address this have been explored and applied, including reporting only the presence or absence of GSR and avoiding expressing any statement of significance, providing detailed expressions of the limitations of GSR evidence, or relying solely on the experience of the expert to describe the significance of the finding. While each of these approaches has strengths and limitations, it is clear that a more structured and robust framework for the assessment of GSR evidence would strengthen the ability of experts to express the significance of their findings. Such a framework would allow for all of the analyst's

assumptions and considerations to be transparently described, and informed and supported by data. While various statistical approaches have been explored, one particularly promising model is a Bayesian Network informing a likelihood ratio-based approach.

One of the overarching goals of this thesis has therefore been to collect data that can be used to inform this type of assessment of GSR evidence. It has been well documented that the distribution and persistence of GSR on a POI involved in a shooting is highly specific to the circumstances of that case, and may be contingent on factors such as the ammunition, firearm, number of rounds discharged, atmospheric conditions, activity undertaken post-shooting, time since the incident, amongst others. These are therefore best assessed in the context of a specific case, with testing performed with the case firearm and ammunition as appropriate. In this research, primarily of interest have been those factors which have the potential to generate a false positive result, in which GSR may be observed on an individual who has had no involvement in the shooting incident under investigation. The combined probabilities of these factors producing a positive GSR test result therefore inform the denominator of the likelihood ratio equation. To that end, the previous evaluations of the random background prevalence of GSR, the police background prevalence of GSR, and the extent of secondary transfer under apprehension and arrest were incorporated into a Bayesian Network for the assessment of GSR.

When combined and systematically evaluated under this model, it was observed that the most probable outcome of the random background and secondary transfer from law enforcement under arrest was that zero particles would be present on a POI (P=0.969). It can therefore be inferred that the probability of observing one or more particles of GSR is low (P=0.031). Similarly, the probability of observing two or more particles (P=0.0179) was more than three (P=0.009), and continuing to reduce from there. Practically, this suggests that it could be expected that more than 3 particles as a result of random prevalence and secondary transfer would be observed in less than 1% of cases. Collectively, this suggests that although improbable, there is a possibility of small amounts of GSR being present on a POI as a result of these factors, and therefore these factors should be considered as an element of GSR evidence evaluation.

Ultimately, the research performed in this thesis has provided a solid foundation for a logical framework for the assessment of GSR evidence, specifically in the Australian environment.

8.2. FUTURE WORKS

8.2.1. Future Challenges in understanding GSR particle formation.

It is well understood that the process by which GSR particles are formed is inherently at the mercy of a number of random factors. These can include primer formulation, propellant formulation, firearm type, barrel length, and history of the use of the firearm, amongst others. It seems clear however, that the reaction responsible for the generation is highly complex. It seems that it is not only the weapon memory effect that is responsible for the particle population, but incorporation of further elements from the projectile is a further concern. The more thoroughly we understand the process by which GSR particles are formed, the better the judgements that can be made about the significance of particular findings. HMF ammunition increasing in popularity and availability poses further challenges for the GSR analyst, as changing primer formulation will produce flow-on effects to the type, composition and morphology of the particles formed.

Although the work conducted to assess the internal morphology of GSR particles from various sources has shown promise, there is much further work to be done. Although initial indications are that in some circumstances the internal morphology and composition of some GSR particles is distinct, depending on the type of ammunition from which it originated it is far from being conclusive. However, further research conducted using different ammunition types and compilation of the resultant data into a shared database may allow this to be exploited in casework in the future.

Glassy GSR (gGSR) is glass-containing, characteristic GSR particles, incorporate the glass frictionator used in lieu of antimony sulphide in some 0.22 rimfire ammunitions [158]. Research has indicated that the elemental and isotopic composition of the glass frictionator remains consistent through the firing process. Further, use of a variety of analytical techniques on gGSR shows promise for brand discrimination based on this information [160]. Incorporation of this sort of data into an evidence evaluation framework has the potential to significantly strengthen forensic GSR examinations, particularly at the source and sub-source levels.

8.2.2. Future Challenges for Statistical Evaluation of GSR

As many different evidence types work towards a unified standard for evaluative reporting [123, 234, 238], there are still many challenges to be faced. This is particularly evident for many types of trace evidence, where base-rate frequencies, random prevalence, persistence, and transfer must be considered in the overall evaluation for each evidence type. As it pertains specifically to GSR, there are still gaps in knowledge that need to be addressed. The increasing prominence of HMF-primed ammunitions, new primers and polymer coated projectiles [26] are further changing the face of GSR analysis. These ammunitions generate a different population of particles, departing from the PbSbBa characteristic composition that has been well established and thoroughly researched since the 1970s. In doing so, the nature of GSR evidence must be re-evaluated, and an understanding of other products that may generate particles similar to HMF GSR, or new primers must be established. Further, random prevalence studies addressing the frequency of these new particle types in the population will need to be performed in order to establish the likelihood of encountering such particles on a 'random man'. In Australia, there have been moves in regions to reduce the amounts of Pb used in ammunition, particularly Pb shot for hunting waterfowl in wetlands, due to its significant environmental impact [239, 240]. Internationally however, some regions are legislating-out the use of Pb-containing ammunition entirely, opting to require that non-Pb containing ammunitions be used for all hunting [241]. As legislation and regulation catch up, it is likely that Pb-free, HMF and other ammunition products will increase in market share. As this occurs, it is probable that these ammunition types will be more frequently encountered in casework. Subsequently, further exploration of strategies for incorporating source-level interpretation into a similar framework will strengthen the ability of GSR examiners to more confidently make source-level assessments.

The evidential value of an assessment framework would be similarly further strengthened by the incorporation of organic GSR (oGSR) residues and other particle types (such as glassy GSR – gGSR) into the framework. To do so, tandem analysis methods for the joint sampling and detection of oGSR and iGSR should be further explored. This work is currently being undertaken by a variety of approaches [242-244]. Cumulatively, a number of these factors indicate that there is no shortage of work to be done in strengthening the future evaluation of GSR evidence. It is clear that addressing the wider goals of a Bayesian framework for the assessment of GSR evidence is no small task. To that end, databases, assessment frameworks, and transfer and persistence modelling continues to be a significant focus in improving GSR evidence around the world [238, 245]. While the findings of some of these studies and surveys

is likely to be jurisdiction-specific, strengthening ties and information sharing in the international GSR analysis community will ensure that the assessments of GSR evidence will continue to be refined, strengthened, and improve over time.

However, developing the model and process and collecting the data that informs the statistical evaluation of evidence only represents part of the battle. Lay-people's understanding of the relative strength of the framing of forensic scientist's conclusions has been tested [246-249], with concerns identified with both a numerical and a verbal scale [129, 250, 251]. Fundamentally, the usefulness of any data informing an evaluation of the significance of evidence is inexorably linked to the ability of a lay-audience to understand it, and to do so in the appropriate context. Even the very best model serves the forensic scientist poorly if it cannot be understood by the triers of fact. Particularly important in this consideration is the issue that the triers of fact are privy to the totality of the evidence in a particular matter, and therefore, any statement of the significance of an individual piece of evidence must be appropriately considered in that broader context. For example, can a LR = 10^{-15} offering very strong support for a particular piece of DNA evidence under one paradigm [222] be fairly considered by a lay jury against an LR = 250 offering very strong support for a particular piece of glass evidence [252]? In short, as movement toward a unified framework for evaluative reporting progresses, it is incumbent upon forensic scientists as experts to ensure that statements of evidential significance continue to be appropriately understood in context.

A logical framework for the assessment of GSR evidence has been underpinned by the findings of this work. However, this does not suggest that the work is complete. A fundamental benefit to the use of a Bayesian network structure for the assessment of evidence is that the network can be expanded, contracted, or modified as required by the specific circumstances of the case. Where the BN approach does produce the most value is as a tool that lends a structured approach to the evaluation of the multitude of factors contributing to a statement of the evidential value of a particular finding. By providing a structured and systematic approach, the expert is able to explain in a transparent fashion how they have arrived at their conclusion. Although this will still require a variety of strong data sets to inform the network, the structure itself allows for evidential value to be better contextualised and explained. As more research is performed to expand and develop the network structure through the addition of new nodes and improving the data informing existing nodes , it is anticipated that this approach will be refined, resulting in stronger outcomes for GSR evidence.

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