Firearms discharge residues

Thesis

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FIREARMS DISCHARGE RESIDUES

BY

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Abstract

The three introductory chapters are intended to summarise the available knowledge prior to the experimental work and to provide useful background information for chemists, with little or no understanding of firearms, who are required to undertake the chemical aspects of firearms casework examination.

Chapter 1 provides a much condensed outline of the historical development of firearms and ammunition, with emphasis on ignition systems up to the development of the percussion primer and self contained ammunition. The second chapter presents a comprehensive literature search on the chemical nature of modern ammunition and firearms, with particular attention to priming compositions and projectiles. The final introductory chapter deals with the nature and properties of firearms discharge residues and outlines the most important developments in the search for a satisfactory detection and identification method.

The objective of the experimental work is to record experience gained from the 26 year terrorist campaign in Northern Ireland and to use such experience, coupled with further research and development, to substantially improve existing procedures for firearms and explosives residue detection.

The experimental work conducted is detailed in Chapter 4. Chapter 5 deals primarily with the Particle Analysis method for the detection and identification of firearms discharge residue, particular attention being directed towards the particle classification system and discharge residue from mercury fulminate and Sintox primed ammunition. Chapter 6 covers suspect handling procedures and

contamination avoidance and makes recommendations for permanent improvements in and regular monitoring of the environment in which suspects are sampled. Chapter 7 outlines the development of a method for the detection of the organic constituents of firearms discharge residues, which is compatible with existing inorganic firearms discharge residue and organic explosive residue detection techniques and which enables all suspects to be routinely screened for inorganic and organic firearms discharge residue and organic explosives residue. The final chapter summarises the conclusions, recommendations and comments arising from the work. .

Declaration

I hereby declare that this thesis is my own work and is based on research carried out at the Northern Ireland Forensic Science Laboratory between January 1985 and January 1997.

James Smyth Wallace
20/1/97

JAMES SMYTH WALLACE
20th January 1997

Dedication

To my wife Edna and my children, Carol and Ian

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I would like to express my sincere thanks to my supervisors, Dr D.A.Johnson of the Open University and Mr R.A.Hall - Director of the Northern Ireland Forensic Science Laboratory, for their helpful advice, guidance and encouragement throughout the period of this work and to Professor B. Caddy - Director of the Forensic Science Unit, University of Strathclyde for assistance in structuring the thesis.

I wish to acknowledge the help received from the Royal Ulster Constabulary; the graphics department for assistance with many of the illustrations and the CID, S.O.C.O., and uniformed officers for their co-operation during the course of this work.

My thanks are also due to all my colleagues whose assistance, dedication, and professionalism over the past 25 years has been a source of inspiration. Finally I would like to thank Mrs M Rodgers for typing the thesis and Mr. N. Leinster for his time, patience and expertise in use of computer software.

Glossary

Chemical

ACN	acetonitrile	IPA	isopropyl alcohol
DBP	dibutylphthalate	MC	methylcentralite
DDNP	diazodinitrophenol	MCE	mixed cellulose esters
DEGDN	diethyleneglycoldinitrate	MIBK	methylisobutylketone
DMF	dimethylformamide	NB	nitrobenzene
DNB	dinitrobenzene	NC	nitrocellulose
DNT	dinitrotoluene	NG	nitroglycerine
DPA	diphenylamine	PETN	pentaerythritoltetranitrate
nDPA	a nitro diphenylamine	PTFE	polytetrafluoroethylene
meDPA	methylethyldiphenylamine	RDX	cyclotrimethylenetrinitramine
EC	ethylcentralite	TNT	trinitrotoluene
EGDN	ethyleneglycoldinitrate		

Instrumental

FAAS	flameless atomic absorption spectrophotometry	HPLC/ PMDE	high performance liquid chromatography/ pendant mercury drop electrode
FTIR	fourier transfer infra red spectroscopy	NAA	neutron activation analysis
GC/MS	gas chromatography / mass spectrometry	SEM/ EDX	scanning electron microscopy/ energy dispersive x - ray analysis
GC/TEA	gas chromatography/ thermal energy analyser		

Firearms/ Ammunition

ACP	automatic Colt pistol	⊕	NATO specifications
AP	armour piercing	NCNM	non - corrosive, non - mercuric
FMJ	full metal jacket	P	parabellum
G	gauge (bore)	+P	higher pressure (velocity) ammunition
H & K	Heckler and Koch	Rem	Remington
HP bullet	hollow point bullet	Rev	revolver
I bullet	incendiary bullet	RNL bullet	round nosed lead bullet
JHP bullet	jacketed hollow point bullet	S &W	Smith and Wesson
Jkt	jacket	SMG	sub - machine gun

JSP bullet	jacketed soft point bullet	Spl	special
K	kurtz (short)	SWC bullet	semi - wad cutter bullet
L	long	T bullet	tracer bullet
.22 LR calibre	.22 long rifle calibre	TMJ bullet	total metal jacketed bullet
Mag	magnum	Win	Winchester
NATO	North Atlantic Treaty Organisation		

Miscellaneous

AFTE	Association of Firearm and Tool Mark Examiners	ARDS	automatic residue detection system
ATO	ammunition technical officer (Army)	CCI	Cascade Cartridge, Inc.
CID	Criminal Investigation Department	CDR	cartridge discharge residue
FDR	firearms discharge residue	FBI	Federal Bureau of Investigation (U.S.A.)
M level	major level	GSR	gunshot residue
T level	trace level	Mi level	minor level
RPG	rocket propelled grenade	NIFSL	Northern Ireland Forensic Science Laboratory
SOCO	scenes of crime officer		

Note:

1. Firearms related terms are defined in the Association of Firearm and Toolmark Examiners Glossary, Third edition 1994, Published by 'Available Business Printing, Inc.; 1519 South State Street, Chicago, Illinois 60605, U.S.A.
2. Ammunition details are given in 'Cartridge Headstamp Guide' by H.P. White and B.D. Munhall, Published by H.P. White Laboratory, Bel Air, Maryland, U.S.A.
3. 7000 grains (gn) = 1lb = 453.59237 g

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1 *HISTORICAL ASPECTS OF FIREARMS AND AMMUNITION*

1.1 FIREARMS AND AMMUNITION

1.1.1 Firearms

Hand held weapons preceded weapons designed to kill or incapacitate from a distance. Such weapons included wooden clubs and pointed sticks eventually leading to pointed stone tipped spears, and knives, daggers and swords made from wood or stone.

The desire to propel some form of missile through the air to kill or injure a foe originated with primitive man but this was probably not his initial objective. It is highly likely that the original reason was the necessity to hunt and kill dangerous animals for food and clothing and for obvious reasons a weapon capable of killing from a safe distance would be highly desirable.

The first projectiles were probably stones and pointed wooden sticks which were initially thrown by hand. These developed through various stages including flint tipped spears and arrows, eventually leading to propulsion using slings, throwing sticks, catapults, bows, etc., all of which gave the projectiles greater range, greater velocity and consequently greater wounding power.

A major development in man's armament was his discovery of and ability to work metal and this rapidly led to metal knives, daggers and swords and metal tipped spears and arrows. These were much superior to the wooden and stone weapons, and were used for many years until the development of a hand held weapon that surpassed all others and which had a pronounced effect on the history of man - THE FIREARM.

A firearm is a tool designed to discharge lethal projectiles from a barrel towards selected targets. It is the means of aiming and discharging the projectile and imparting stability to it.

In Northern Ireland the law defines a firearm as" ...... a lethal barrelled weapon of any description, from which any shot, bullet or other missile can be discharged ..... "(1).

This very broad definition does not specify the means of causing "the shot, bullet or other missile" to be discharged, but this may be by compressed air, by mechanical means, or by the rapid burning of a propellant composition. However, since a firearm operated by the burning of a propellant is by far the most common and potentially the most lethal, and because such a system lends itself to chemical analysis, only this will be considered.

In its simplest form a firearm could be a crude metal tube with one end packed with some form of propellant which on ignition produces enough gas pressure to discharge a projectile or projectiles with sufficient energy to cause human death. In its most complicated form it might be a well made and finely engineered tool capable of discharging and directing bullets on automatic fire up to a rate in the region of 1,500 rounds per minute over an accurate range of about 200 metres. Alternatively, it might be a high powered, highly accurate sniper rifle equipped with telescopic sights and capable of killing a selected target at a distance of 1,000 metres or more.

Firearms are relatively cheap, readily produced, reliable and deadly and find many uses amongst which are warfare, sport, self-defence, law enforcement and crime. It is the use of firearms in crime that demands the attention of the forensic scientist.

The most commonly used firearms in crime are pistols, revolvers and rifles up to and including 0.455" calibre, and shotguns, the most popular of which is the 'sawn off' 12 bore calibre. This discussion deals with these weapons although machine guns; larger calibre firearms; homemade firearms; air, spring and gas guns; imitation and replica firearms, are also encountered in crime, but to a much lesser extent.

Pistols and revolvers are usually described as handguns, and rifles and shotguns as shoulder guns, as this is their normal mode of use.

A revolver is a single barrelled handgun with a revolving cylinder (multiple chamber) which holds a number of rounds of ammunition (usually six). Each time the trigger is pulled the cylinder is mechanically rotated so that each successive round of ammunition is placed in the firing position, i.e. in line with the barrel. The spent cartridge cases are not ejected automatically but have to be removed manually.

A pistol is a single barrelled handgun in which the chamber is an integral part of the rear end of the barrel. A pistol can be either the single shot type, with manual or automatic ejection of the spent cartridge case or, much more commonly, the self-loading type. In self-loading pistols a number of rounds of ammunition are loaded into a magazine which is usually fitted into the hand grip of the weapon. Once the weapon is initially cocked and discharged a reloading mechanism, which is operated by the force of recoil or by gas pressure, extracts and ejects the spent cartridge case from the chamber and reloads the chamber with a live round of ammunition from the magazine. The process is repeated with each pull of the trigger until the ammunition is expended.

Rifles have a longer barrel than revolvers or pistols, and are usually more powerful and designed to shoot at targets at longer distances. Like pistols, they can be either the single shot type or the self loading type. Rifles use various methods for the ejection of spent cartridge cases including lever, bolt or pump action in manual operation, or recoil energy or gas pressure in automatic operation.

Shotguns can be either single or double barrel. The most common type is the design in which the barrel breaks forward on a hinge to expose the breech, into which live cartridges are inserted and from which spent cartridges are extracted. Some shotguns use either pump action, recoil energy or gas pressure reloading mechanisms.

The barrel of a firearm is a tube made of iron or steel. The inner surface of the barrels of revolvers, pistols and rifles contain a number of spiral grooves known as rifling. Hence they are known as rifled bore weapons. The rifling of the barrel grips the bullet and causes it to rotate thereby, preventing it wobbling or turning over in flight.

The raised area between two grooves is called a land and the calibre of a firearm is based on the diameter of the bore measured between two opposite lands (2). This over simplified definition of calibre gives a rough approximation of bullet diameter as the bullet is usually slightly bigger than the diameter of the bore. Calibre is usually given in inches or millimeters and common calibres for handguns are .22" (6 mm); .25" (6.35 mm); .32" (7.65 mm); .38"/.357" (9 mm); .45"; .455" and for rifles are .22" (6 mm); .223" and .30" (7.62 mm). There are many other calibres in existence. In fact the suitability of a round of ammunition for use with a particular firearm not only depends on the diameter of the bullet but also on the length and design of the cartridge case.

With very few exceptions the inner surface of shotgun barrels are smooth, hence they are called smooth bore weapons. The calibre of a shotgun is usually expressed as its bore or gauge the most common being 12, 16 and 20 bore with the 12 bore being by far the most popular. Bore refers to the number of lead balls of bore diameter which weigh 1 lb (3). Smaller diameter shotguns are usually described by the internal diameter of the barrel e.g .410".

1.1.2 Ammunition

A round of ammunition consists of a primer, propellant and bullet all of which are contained by a cylindrical shaped cartridge case (shell). Instead of a single bullet shotgun ammunition typically contains numerous spherical lead balls which are totally enclosed within the cartridge case. Shotgun cartridges are usually made of plastic with a metal base. Cartridges for rifled fireams are usually made of brass with the base of the bullet inserted into the neck of the cartridge case.

Figure 1.01 gives cross-section views of ammunition for rifled bore and smooth bore firearms.

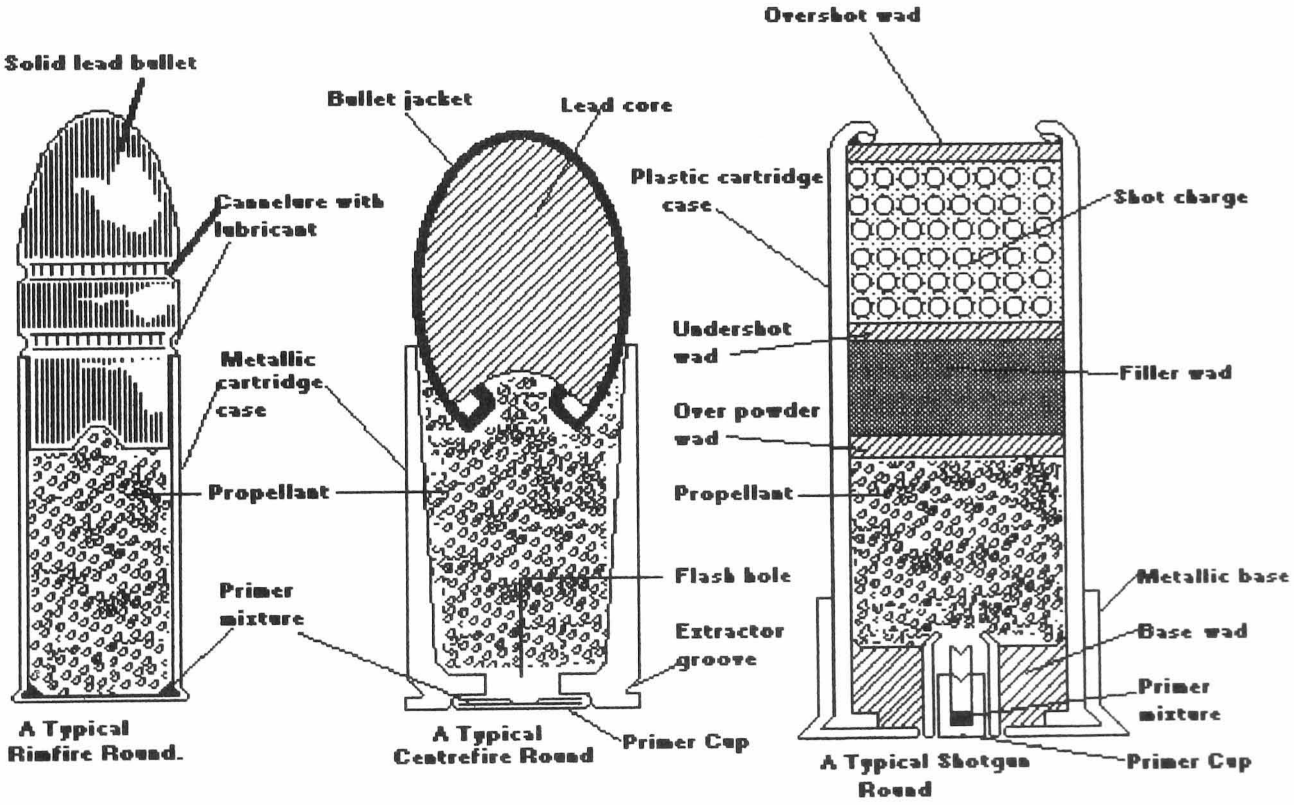


Figure 1.01 Typical ammunition types

1.1.3 Discharge of a firearm

The firing mechanism of a firearm consists of a mechanical device which causes a hammer to fly forward and deliver a blow to the firing pin when the trigger is pulled. In some firearms the hammer and firing pin are made in one piece. The firing pin goes through a small hole in the breech face and strikes the primer cup.

The primer cup contains a mixture of chemicals which sensitize each other to percussion and rapid burning, and consequently the primer burns rapidly producing a flame and a shower of hot particles that penetrates and ignites the propellant.

The burning of the propellant very rapidly produces a large volume of gases in a confined space accompanied by a substantial temperature and pressure rise. The resultant gas pressure forces the bullet away from the cartridge case and down the barrel of the firearm. The temperature and pressure rise also serves to cause the cartridge case to expand in the chamber, thereby effectively sealing the chamber to prevent any rearward escape of gas (obturation) which would lead to a reduction in pressure and consequently a reduction in bullet velocity.

The time span from the firing pin hitting the primer cup to the bullet leaving the gun is typically in the region of 0.01 to 0.03 seconds (4). Muzzle velocities range from approximately 600 feet per second for very low power handguns to approximately 3,500 feet per second for very powerful rifles. Temperatures and pressures inside a gun during discharge can be in the region of 3000°C (5) and 50,000 pounds per square inch (6).

1.2 HISTORY OF FIREARMS AND AMMUNITION

1.2.1 History of gunpowder

A mixture known as blackpowder revolutionised the art of warfare whenever it was applied to the propulsion of missiles. Blackpowder is a mixture of potassium nitrate (saltpetre), charcoal and sulphur in varying proportions, granulation and purity. A typical composition of a modern blackpowder is saltpetre 75%, charcoal 15% and sulphur 10% (7). A mixture of saltpetre, charcoal and sulphur with other ingredients was used in China and India in the eleventh century for incendiary and pyrotechnic purposes long before 'true' blackpowder was invented (8). History often deals in conjecture and opinion and it is not known for certain when and by whom blackpowder was invented, or when and by whom it was applied to the propulsion of a missile from a firearm. The composition of blackpowder was first recorded by an English Franciscan monk called Roger Bacon in 1249 but he did not apply it to the propulsion of a missile from a firearm. This use of blackpowder is usually credited to a German Franciscan monk called Berthold Schwartz in the early fourteenth century (9).

Whenever blackpowder was used as a propellant in guns it was commonly referred to as gunpowder. At first the ingredients were simply mixed together but the resulting gunpowder had a tendency to separate into its component parts when carried and it also absorbed moisture. Also the purity of the ingredients varied markedly and a combination of these factors led to early gunpowder being somewhat unreliable.

Improved methods of combining the chemicals evolved and by the fifteenth century a form known as 'corned' gunpowder had been developed in which the components were bonded together in small grains.

For many years experiments were conducted to determine the best composition of the mixture for use in firearms. Some examples of the formulas used at various times are :-

		% Saltpetre	*% Charcoal*	*% Sulphur*
c. 1252.	(Roger Bacon)	37.50	31.25	31.25
1350,	Arderne	66.6	22.2	11.1
1560,	Whitehorne	50.0	33.3	16.6
1560,	Bruxelles studies	75.0	15.62	9.38
1635,	British Government contract	75.0	12.5	12.5
1781,	Bishop Watson	75.0	15.0	10.0

(Other formulas are used for blasting purposes and for pyrotechnic devices)

Any marked deviation from the last two formulas produces gunpowder which has a slower burning rate or which burns with less vigorous effect (10).

Blackpowder was used as a firearms propellant until it was gradually replaced by smokeless propellants towards the end of the nineteenth century.

1.2.2 History of ignition systems

Ignition of the propellant was a major problem from the introduction of gunpowder in the early fourteenth century until the development of a percussion primer by a Scottish clergyman, the Reverend Alexander John Forsyth, in 1805 (11).

The first means of igniting the propellant was by placing a glowing twig or a hot wire into a touch hole at the rear of the barrel where it came into contact with the propellant. This direct method of ignition had many disadvantages; the firer needed to be near a fire, ignition was at the mercy of the wind and the rain and it was difficult to aim properly.

To overcome the lack of mobility the 'slow' match was developed. The match consisted of a piece of cord which had been soaked in a strong solution of potassium nitrate and then dried. Placed in the touch hole and lit the match would smoulder with a glowing end at the rate of about an inch a minute until it reached and ignited the propellant (12). Speed of ignition and dependence on weather conditions were serious disadvantages.

The first mechanical device to achieve ignition was the Matchlock which derived its name from the 'slow' match. The match was attached to the gun by a match holder and the action of the trigger lowered the glowing end of the match into a flashpan which contained loose gunpowder (priming powder). The powder in the pan was ignited (flashed) by the glowing match end and the flame was passed through a small barrel vent to ignite the main propellant charge. This was a major improvement in ignition systems as the time of discharge closely coincided with the pull of a trigger. Early matchlocks had an open flashpan and consequently a sudden gust of wind could remove the gunpowder from the flashpan. This was partly solved by fitting a cover over the flashpan, although the smouldering match system of ignition was still dependent on weather conditions.

The next major improvement in ignition systems was the Wheel-lock. It worked in the same way as the matchlock by conveying the flame from the gunpowder in the flashpan through a barrel vent to ignite the main propellant charge. However the ignition of the powder in the flashpan was achieved by sparks from flint stones or pyrites being held by a moving arm drawn down against a spring operated spinning serrated metal wheel (13). The spring for the wheel had to be tensioned with a key before firing each shot.

A more reliable and important variation of the principle of the wheel-lock was the Flintlock. In the wheel-lock sparks were produced by a grinding motion whereas in the flintlock system sparks were produced by a striking motion.

A cock or hammer with a piece of bevelled flint securely clamped to it and an L-shaped steel flashpan cover, called a frizzen, completed the spark making battery. When the hammer fell the flint struck the upper face of the hinged pan cover (forcing it open and exposing the gunpowder in the pan) and caused sparks which ignited the gunpowder in the pan. Again the flame from the gunpowder in the pan was directed through a small vent in the barrel causing the main propellant charge to ignite (14).

All the means of ignition, from the hot wire to the flintlock, were dependent to a greater or lesser extent on the weather conditions and none offered the reliability of ignition experienced with modern ammunition. However the flintlock was a very efficient mechanism and with the introduction of a waterproof flashpan in 1780 (15) the flintlock offered the shooter a reasonably reliable means of ignition under most weather conditions.

The flintlock was not without its faults. Misfires were not uncommon and since each piece of flint was serviceable for only 20 to 30 shots, the ignition system had to be efficiently maintained. The priming powder remained a potential weakness since wind or rain could dispose of it at the crucial moment of firing. Also the small time delay between pulling the trigger and the ignition of the main propellant charge was annoying. It took time for the flint to scrape along the frizzen and for the sparks to fall into and ignite the main propellant charge. The shooter had to make allowance for the delay especially when aiming at moving targets. A quicker and more reliable means of igniting the main propellant charge was needed (16).

According to many writers, the Reverend Alexander Forsyth studied a group of chemical compounds called metallic fulminates, whose existence had been known from the early seventeenth century. It was also known that they exploded with a flash when struck a sharp blow with a hard object. It was in 1805 that he applied this property of metallic fulminates to firearms ignition, thereby inventing the percussion system of ignition.

In 1807 he took out a patent on his invention by which a pivoted magazine deposited a few grains at a time of mercury fulminate into a touch hole in the barrel of the firearm. The mercury fulminate was detonated by a blow from the hammer of the firearm sending flame through the touch hole to ignite the propellant. 'Instant' ignition had been achieved.

The pivoted magazine was too complicated and subsequent development by other workers was geared towards a more convenient and efficient means of presenting the mercury fulminate to the firearm.

This led to several short-lived innovations including the tube lock, patch primers and the pill lock eventually leading to the percussion cap which proved to be the most efficient and practical way to package the primer. The development of the percussion cap - small waterproof copper cups - is credited to Joshua Shaw in 1816 (17). The cap was placed over a permanent hollow nipple, screwed into a flash hole in the gun barrel, and detonated by the crushing impact of the hammer.

1.2.3 History of bullets

The first projectiles to be discharged from any type of firearm were stones, feathered iron arrows and iron shot. These were discharged from cannons (18). The first hand held firearms had bore diameters between 1.5" and 2" and suitable small round stones were used as projectiles. The earliest use of lead in bullets would appear to have been about 1340 and these consisted of spherical lead bullets (19). Firearms of this era were large and heavy and from this time to the present day there has been a gradual reduction in bore size and weight. By the time the flintlock pistol came into use the spherical lead bullets were between 0.6" and 0.7" in diameter. Bullets of this type were used for many years in smooth bore muzzle loading firearms where the bullet did not have to be a tight fit in the bore.

Rifling of the bore was found to improve the accuracy and consequently the effective range of firearms, and was first applied to firearms by a German gunsmith called Augustin Kutler in 1520 (20).

The introduction of rifling coupled with the development of breech loading firearms focused attention on bullet design.

With rifled bore firearms the bullet had to be a tight fit otherwise it would not grip the rifling when discharged but, if it was too tight a fit, it was difficult to load the gun. The problem of bullet size created particular loading problems for rifled muzzle loaded firearms and for rifled breech loading firearms. A tight fitting spherical lead bullet was difficult to load, especially when the bore or chamber was dirty with fouling from previous shots.

The first attempts to solve the problem involved the use of a belt (driving band) around the lead ball. The bullets were cast in a mould and the lead belt was an integral part of the bullet. The spherical part was an easy fit in the bore and the belt was made large enough to fit the rifling. This bullet proved to be unsatisfactory as the belt caused the bullet to tilt after leaving the muzzle and it was very susceptible to the effects of the wind, causing poor accuracy.

During this period a large number of bullet designs were produced and tested, and it was found that an elongated bullet was much more efficient than a spherical one. The elongated bullet had greater weight for a given diameter and was more stable in flight. In 1855 General J. Jacob produced a cylindro-ogival bullet with four cast on lugs to engage the rifling. Another mechanically fitting bullet was produced by an English engineer called Joseph Whitworth who developed a hexagonal bored barrel and hexagonal shaped bullets. The hexagonal bullet had six flat portions along its cylindrical body which were given a twist corresponding to that of the rifling. Such mechanically fitting bullets were very difficult to manufacture and were soon found to be unnecessary. By this time the bullet diameter had been reduced to 0.45".

The first practical solution to the problem was developed in 1849 by Captain Minie of the French Army. He produced a cylindro-ogival bullet with a tapered hollow base containing a semi-spherical iron cup. When the gunpowder burnt the hot expanding gases forced the iron cup into the bullet which spread the bullet slightly so that its sides gripped the rifling. It was soon discovered that the same effect could be obtained without the iron cup and the Minie bullet was abandoned.

In 1863 William Ellis Metford produced a cylindro-conoidal bullet with a shallow depression in its base. The bullet was made of lead hardened with antimony and the cylindrical part was wrapped in a sheath of paper. The shape and design of this bullet resembles the modern bullet (21).

Another problem related to bullet design was the fact that the rifling could cause lead to be stripped from the bullet, resulting in 'leading' of the bore, which has a detrimental effect on accuracy by deforming the bullet and reducing the efficiency of the rifling. The use of antimony or tin to harden bullet lead dates from the early nineteenth century. The use of hardened rather than soft lead serves to reduce 'leading' of the bore and deformation of the bullet and also slightly reduces the extent of bullet deformation on hitting a target.

The use of hardened lead did not eliminate leading but it slightly reduced the extent of the problem. Lubrication of the bullet was found to significantly reduce the amount of leading by preventing the partial melting of the lead by heat due to friction. Lubricants such as tallow and beeswax were placed in annular grooves at the rear of elongated bullets.

The problems of 'leading' and bullet deformation were eventually eliminated by the use of a bullet jacket (envelope). Such a bullet was introduced in 1883 by a Major Rubin of the Swiss Army and consisted of a soft lead core covered with a copper jacket. This was an important step in bullet development since up to this time the rate of the rifling twist was limited by its effect on the unjacketed lead bullet. With this new bullet the rate of twist could be substantially increased and the rifling grooves could be made shallower.

A bullet jacket is normally harder than the bullet core material but soft enough to take up the rifling and not cause excessive wear to the barrel. Bullet jackets were for a long period made of cupro-nickel (80% copper, 20% nickel); gilding metal

(90-95% copper, 10-5% zinc) or steel which was coated with a softer metal to prevent barrel wear and rusting (22). In 1922, 1% to 2% of tin was added to the gilding metal because of its lubricating properties.

Unjacketed lead bullets are unsuitable for use in most modern self-loading firearms, the exception being 0.22" calibre rifles and pistols. With higher velocity firearms melting and fusing of the exposed lead surface can occur causing leading of the barrel, deformation of the bullet and a loss of accuracy of the firearm. Modern lubricated unjacketed lead bullets are usually confined to use in lower velocity revolvers and 0.22" calibre rimfire rifles and pistols, i.e. firearms with a muzzle velocity of less than about 1,200 feet per second. Another important factor influencing the use of unjacketed lead bullets is that they are more prone to 'feeding' problems in self-loading firearms due to the fact that the exposed part of the unjacketed lead bullet is more susceptible to damage than its jacketed equivalent.

The vast majority of modern bullet types are either completely or partially jacketed, usually with gilding metal, and are produced in a range of shapes, sizes, weights and designs depending on their intended use.

1.2.4 History of ammunition

The self-contained metallic cartridge is a relatively recent development in historical terms. Gunpowder has been in use as a firearms propellant for about six hundred and seventy years but the metallic cartridge is only about one hundred and fifty years old. The modern self-contained metallic cartridge was perfected about one hundred and twelve years ago and high velocity types with smokeless powders were developed about ninety two years ago (23).

Prior to the introduction of a self-contained cartridge, firearms were muzzle loaded by pouring a measured amount of gunpowder down the barrel followed by the bullet and then compacting the gunpowder/bullet combination by the use of a plunger and some sort of wad. Ignition of the gunpowder was accomplished separately. Obviously this system suffered several major disadvantages. Faster reloading in order to achieve greater firepower was desirable, the means of ignition was susceptible to weather conditions and it was necessary to carry items of equipment ancillary to the firearm, i.e. gunpowder, ignition powder (finely powdered gunpowder), bullets and ramming rod. Because of the long loading time the advantages of a self-contained ammunition package was evident early in the history of firearms and many attempts were made to produce such a package.

One of the earliest attempts to decrease loading time was a breech-loading match-lock firearm with the rear end of the barrel counterbored to give a larger diameter than the rest of the bore. A removable iron chamber complete with its own flashpan and loaded with gunpowder and bullet was inserted. Extra loaded insert chambers could be carried (24).

A paper cartridge was developed about 1550 and consisted of gunpowder and bullet wrapped in a cylindrical shaped paper package or a small paper bag of gunpowder attached by thread to the bullet. In use the bottom of the paper cartridge was torn open, usually with the teeth, and the gunpowder and bullet poured down the barrel from the muzzle end after placing a small amount of gunpowder in the flashpan. The paper was sometimes rammed down the barrel and used as a wad to prevent the bullet dropping out of the barrel.

Various designs of the paper cartridge were in general use by the middle of the seventeenth century and paper was used for cartridge manufacture for about 300 years.

Whenever the complete cartridge, including paper, was loaded into the firearm the paper cartridge case burnt when the charge was fired. However smouldering pieces of paper could remain in the barrel and on reloading an explosion could occur. This led to the introduction of a completely combustible paper cartridge, the paper having been nitrated prior to assembly. Nitrated animal intestines were also used in cartridges during this period. The paper cartridges caused problems in damp weather and the cartridges had to be carried in waterproof containers. Several attempts were made to waterproof paper cartridges using varnish but this did not achieve widespread acceptance.

The earliest example of a fully self-contained cartridge was produced by a Swiss engineer called Jean Samuel Pauly in 1808. This cartridge was loaded directly into the breech of a firearm, which was also developed by Pauly, and was fired by a needle piercing it.

An improved form of the cartridge was patented by Pauly in 1812. It consisted of a paper body rolled round the front portion of a rimmed brass base piece, the base of which had a central recess to contain the primer powder which was sealed with a small piece of gummed paper to retain it in position and protect it from moisture (25).

This was one of the most important developments in firearm history and is the earliest example of a fully self-contained centre fire cartridge. However the system did not gain widespread acceptance as it only applied to firearms of Pauly's design. It did establish the principle of a completely self-contained cartridge, i.e. a cartridge having its own means of ignition as an integral part.

Figure 1.02 gives a cross-section view of the Pauly cartridge.

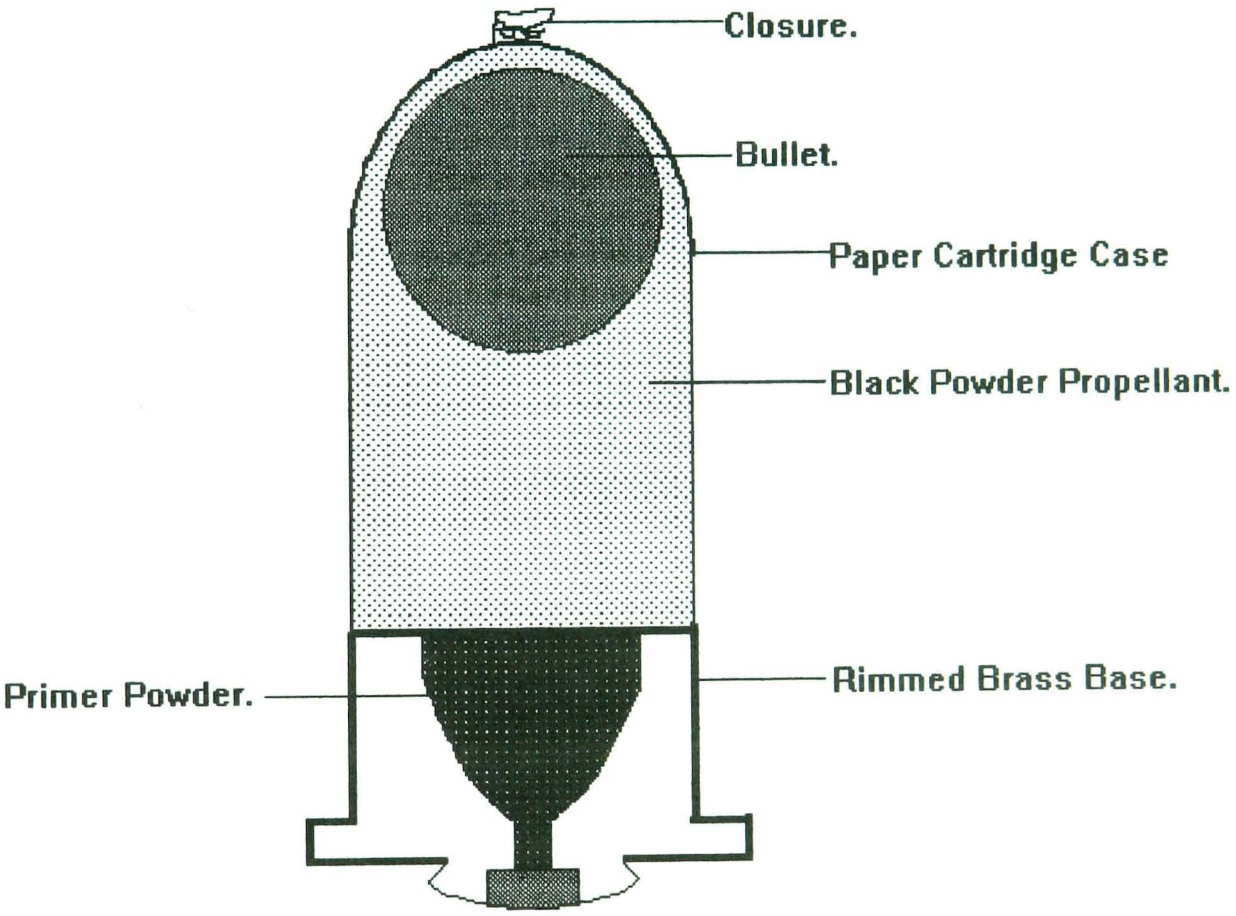


Figure 1.02 Pauly cartridge

The next cartridge with an integral primer was the Needle-Gun cartridge developed by a Prussian named Johann Nikolas Dreyse in 1831. In its original form it was made with either a paper or linen envelope and in its later form it was made entirely of paper. It has a flat base and was tied shut above the bullet which was contained in a sabot. There was a recess in the base of the sabot which contained a mercury fulminate paste. Ignition of the cartridge was accomplished by a long spring operated needle which had to penetrate the full length of the gunpowder charge to reach the mercury fulminate.

Figure 1.03 gives a cross-section view of the Dreyse cartridge.

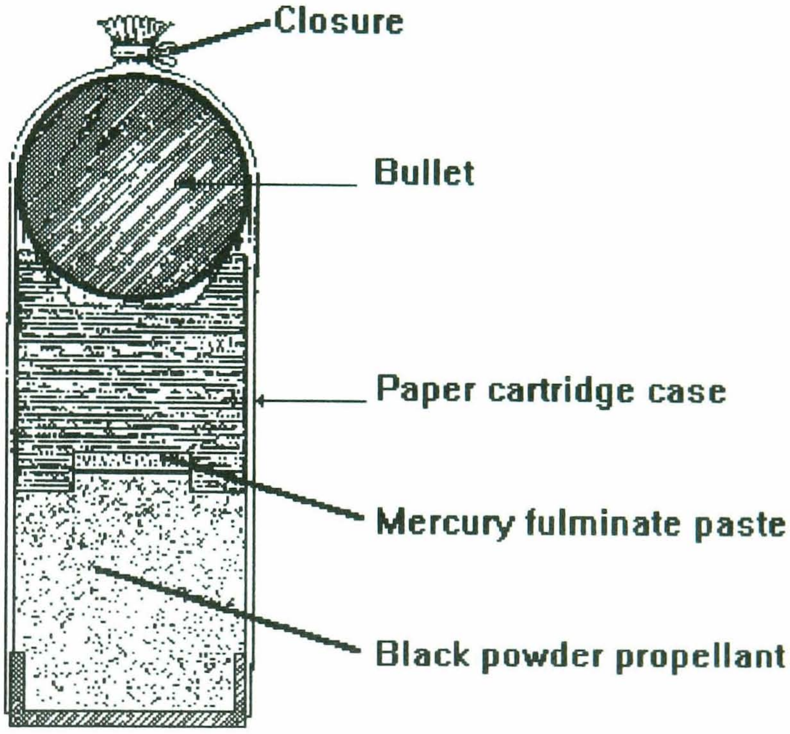


Figure 1.03 Dreyse cartridge

Further development of the needle gun concept led to a pasteboard cartridge case with the primer in the base portion in the form of a shallow metal foil cup containing a flanged percussion cap with its open end facing the base of the cartridge. A small hole was made through the centre of the base and metal foil cup and the cap was ignited by the penetration of a short firing needle.

An innovation not involving a conventional cartridge case was introduced by Joseph Rock Cooper in 1840.. This was a bullet with a charge of gunpowder placed in a cavity at the base of the bullet. Ignition was by means of an external percussion source. Development of this concept by other workers culminated in a conical shaped bullet with a charge of gunpowder in a base cavity which was closed by a cork plug and fitted with a priming system (26).

As there was nothing to prevent the rearward escape of gas and as the bullet itself had only about one fifteenth its weight in gunpowder charge, the bullet lacked power. Misfires were common and the system was abandoned about 1856.

Until 1846 all attempts to develop a satisfactory fully self-contained cartridge shared a serious disadvantage. None of them effectively sealed the chamber at the time of discharge and consequently there was a rearward escape of gas resulting in a reduction in the efficiency of the system. This problem was solved by the introduction of the metallic cartridge case which momentarily expands during the discharge process and seals the chamber.

The first recorded examples of fully self-contained completely metallic cartridges were the Pin-Fire cartridges of the early 1850's. These consisted of a thin copper cartridge case with a striker pin projecting radially from the base end. (Brass came into general use in the 1870's and replaced copper as the case material.) The striker pin was aimed at the priming composition but positioned just clear of it.

Figure 1.04 gives a cross-section view of the Pin-Fire cartridge.

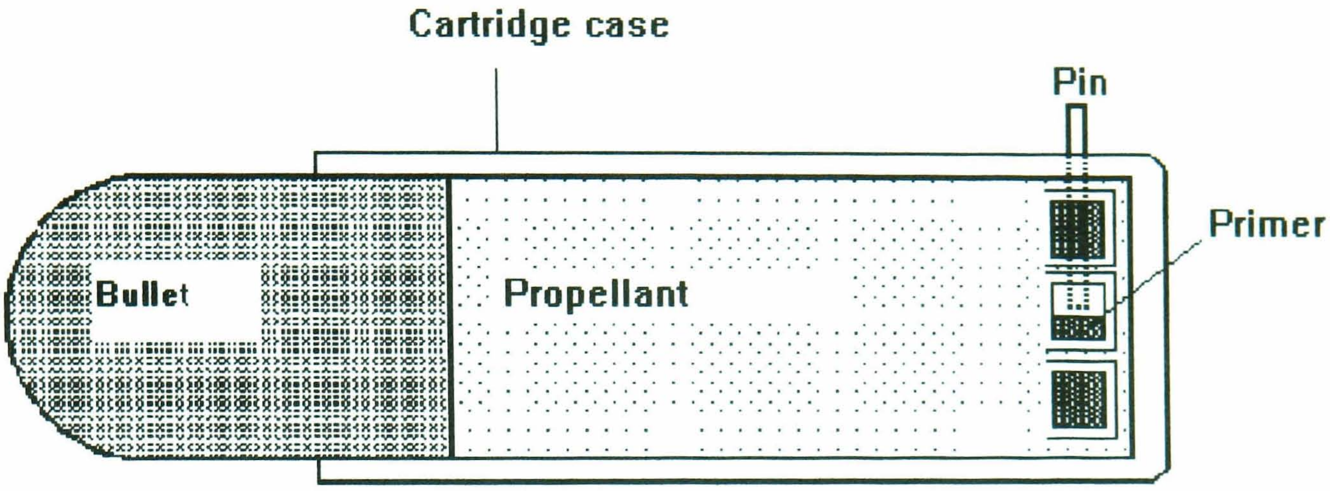


Figure 1.04 Pin-Fire cartridge

By effectively sealing the bore during discharge the Pin-Fire cartridges made breech loading a much more practical proposition and these cartridges were manufactured until the late 1930's. A major disadvantage of this system was that because of the projecting pin the cartridge could be loaded in one position only (27).

The next stage in cartridge evolution was the Rim-fire cartridge. The idea of a cartridge with a hollow rim to contain the priming composition was patented by a French gunsmith called Houllier in 1846 and developed by another French gunsmith called Flobert. The cartridge was originally produced with no gunpowder charge, the priming composition serving as both igniter and propellant.

In 1854 the American firm of Smith & Wesson developed the design by lengthening the case so that it could hold a charge of gunpowder. The rim-fire system became very popular and was manufactured in a range of calibres. A major advantage of the rim-fire cartridge was that it made possible the construction of firearms having a supply of cartridges housed in a magazine.

As firearms developed the trend was towards smaller calibre and greater power and range and it was found that the thin metal base of the rim-fire cartridge could not withstand the higher pressures involved. This was a disadvantage that could not be readily overcome and was one of the main reasons for the decline in popularity of the rim-fire cartridge. Other disadvantages of the rim-fire cartridge are unsuitability of design for modern firearm loading and ejection systems, the larger amount of priming composition that is required and the manufacturing inconvenience of ensuring an even spread of priming composition around the rim.

Rim-fire cartridges are still manufactured but only in the popular 0.22" calibre, and all other modern firearms ammunition is centre fire (central fire).

Centre fire cartridges were produced by Pauly in 1808 but it was not until 1854 that the firm of Smith & Wesson perfected and patented both the centre-fire and rim-fire metallic cartridge case. Since this time cartridge development has consisted of many small improvements, some resulting from advances in engineering and metallurgy, some from improvements in firearms design and some as a result of the development of modern smokeless propellants.

The modern cartridge evolved over this period to a very high standard of reliability. Ironically, serious attempts by reputable large munitions companies are now being made to perfect a completely combustible cartridge and/or caseless ammunition which would be suitable for use in modern firearms (28).

1.2.5 History of firearms

The history of firearms is long and complicated encompassing innovations and developments in AMMUNITION - from crude black powder muzzle loaders to modern brass cased, centre fire cartridges using smokeless propellant; in IGNITION SYSTEMS - from a glowing twig touching gunpowder through a simple flash hole in the barrel to a firing pin which strikes and crushes the priming mixture thereby instantly igniting the propellant charge; in MECHANICAL DEVELOPMENTS - from the simple metal tube attached to a stick to the finely machined high technology firearms which are capable of operating from single shot to fully automatic fire; in METALLURGY - from crude iron which withstood the pressure of the weak early powders, to high tensile metals which can withstand pressures in the tens of thousands of pounds per square inch.

Firearms were in general use in Europe for two centuries before the introduction of printing, consequently reliable accounts of early arms development are rare. The first firearms were cannon which fired large round stones, iron balls or a quantity of arrows and would appear to have been introduced into Europe from the Eastern Nations around 1300. Early cannon were small, and shot arrows weighing about half a pound, although very large cannon weighing about 4 tons and firing stone shot weighing in the region of 350 pounds were also produced.

The first handguns were really hand held cannons now known as cannon locks, the lock being the means of firing the gun. These obviously evolved from the early small cannon and were first used in Europe about 1324 (29). The cannon-lock had a cylindrical metal barrel, about 9 to 12 inches long, attached to a staff or pike. They were muzzle loaded with gunpowder, wad and round stones, metal balls or bolts (similar to crossbow bolts). In use, they were crudely aimed with one hand, the staff being held under the arm, and fired by a glowing twig or hot wire brought into contact with the gunpowder through a touch hole in the barrel.

Hand cannon developed through various stages. They were shortened and redesigned for use from horseback and were used in combination weapons where the weapon could either be used as a firearm, or for example, as a club or an axe. Many different designs of hand cannon were widely used for many years, until the middle of the fifteenth century when they were completely superceded by the Matchlock.

The Matchlock was developed about 1400 (30) and by mechanically carrying the fire to the priming it made possible the fitting of elementary aiming sights to the firearm. Early handguns consisted of a barrel secured to a wooden or metal arm but with the introduction of the matchlock musket, firearms had become much more sophisticated and had begun to resemble the modern rifle. Numerous variations of the matchlock were produced and were used for many years, until it was eventually superceded in the seventeenth century by the wheel lock and the flint lock.

The wheel-lock was developed about 1515 (31). This was an important development in firearms as, apart from dispensing with the need for a glowing match, the wheel-lock mechanism could be produced in any desired size which made possible the production of pistols which were small enough to be carried about the person. As with the hand cannon, combined wheel-lock weapons were produced where pistols were attached to weapons such as maces, swords and crossbows. The wheel-lock mechanism was intricate and subject to mechanical failures which were difficult to repair. This prompted a search for a simpler, more reliable mechanism resulting in the introduction of the Flint-lock.

The Flint-lock was developed about 1525 (32) and used a simpler and much more reliable mechanism than the wheel-lock. The flint-lock was used successfully until it was generally superseded by the percussion lock about the middle of the nineteenth century. A measure of the success of the flint-lock is demonstrated by the fact that until 1935, they were made in Germany and Belgium for export to Africa and Asia (33).

The percussion lock was developed in 1805, and by 1816 had evolved into a simple and reliable form. The percussion lock was the predecessor of the modern firearm and used a priming cap consisting of a small metal cup in the base of which was a dried paste containing mercury fulminate. This was placed over a permanent hollow nipple leading to the gunpowder so that the mercury fulminate paste would be crushed between the base of the cup and the nipple by the striking action of the hammer of the firearm. This produced a flame which passed through the hollow nipple and ignited the gunpowder.

The modern firearm employs the percussion principle but the percussion cap (primer) is an integral part of the round of ammunition.

The first practical repeating firearm was a revolver manufactured by Samuel Colt in 1835 (34). Up to this time the vast majority of firearms were single shot. This was a serious disadvantage as the firer was defenceless for a period of time whilst reloading. However the introduction of this revolver heralded the first practical multi-shot firearm. The revolver principle was not new, as flint-lock revolvers were produced prior to 1650 (35). However these were not a practical firearm as they were very prone to mechanical failure.

When a bullet leaves the muzzle of a firearm there is a recoil in the opposite direction to the travel of the bullet. Whilst the recoil is a nuisance it can be used to eject the spent cartridge case, load a live round of ammunition and cock the mechanism. This can also be achieved by using some of the gas generated during discharge.

In the self loading system the block or slide which moves backwards and forwards is stopped after each cycle and stays stopped until the trigger is pulled again. This mechanism can be modified so that the firearm continues to fire until either the ammunition is expended, or the trigger released. Some firearms incorporate a selector lever which allows them to deliver either a single shot, a burst of a preset number of shots, or to become fully automatic.

As early as 1718 there was a hand operated repeating gun, and in 1862 Dr Richard Gatling demonstrated a weapon of this kind which used revolving barrels. These weapons had severe limitations and it was not until 1884 that the first real fully automatic machine gun was patented by Sir Hiram Maxim. This was the first automatic firearm, and it was recoil operated (36). The development of the Maxim machine-gun focused attention on the development of self loading rifles and pistols.

The rifle evolved from the musket which was a long barrelled firearm with a fore end or forearm extending nearly to the muzzle. Dozens of designs of self loading rifles were produced. One of the first practical designs was developed in Austria by Mannlicher in 1885 and it was recoil operated (37).

The recoil operated self loading system was incorporated in the first successful multi- shot pistol which was designed by Hugo Borcharott, and marketed in 1893. George Luger modified the design and produced a highly successful pistol which was in production until 1942 (38).

Today, most self loading firearms are either recoil operated or gas operated, and progress since the production of the Maxim machine-gun has consisted mainly of a series of mechanical improvements resulting in the modern highly reliable self loading, semi or fully automatic firearms now employed throughout the World.

2 CHEMICAL ASPECTS OF FIREARMS AND AMMUNITION – LITERATURE SEARCH

2.1 AMMUNITION

2.1.1 Cartridge case

The cartridge case is designed to house the primer, propellant and to securely retain the bullet in the neck of the case. Cartridge case design is affected by various factors the most important being -

(a) The role of the ammunition.
(b) Type of weapon.
(c) Design of the bullet used.
(d) Type of ignition system, i.e. Boxer primed or Berdan primed.

The vast majority of cartridge cases are made of brass (approximately 70% copper and 30% zinc) but other materials such as steel-coated with either zinc, brass, gilding metal, copper, lacquer or blackened; copper; nickel plated brass; cupro-nickel (approximately 80% copper and 20% nickel); gilding metal (approximately 90% copper and 10% zinc); aluminium; teflon coated aluminium and plastic are also encountered.

Second in popularity to brass is steel. One specification for cartridge case steel is carbon 0.08% to 0.12%, copper 0.25%, manganese 0.6%, phosphorus 0.035%, sulphur 0.03% and silicon 0.12% (39).

Shotgun cartridges are usually plastic with a brass or coated steel base, but paper with a brass or coated steel base and all plastic shotgun cartridges are also encountered. 'All brass' shotgun cartridges are also known in older ammunition, and are also currently manufactured for reloading purposes. Some .410" calibre shotgun cartridges are all aluminium.

Apart from shotgun cartridges brass is by far the most common material used for the manufacture of cartridge cases. Experience has proved brass to be the most suitable as it is strong, sufficiently ductile, non-rusting, suited to drawing operations during manufacture, of reasonable weight, and readily available.

The strict specifications and quality control procedures for cartridge manufacture reflect the very important role the cartridge case plays in the discharge process. Apart from housing all the components of a round of ammunition in one package, a cartridge has to:-

1. Be safe to store, transport and use.
2. Seal against ingress of moisture and oil.
3. Consistently achieve the required ballistics performance even under very different climatic conditions.
4. Maintain performance after many years of storage.
5. Be sturdy enough to withstand rough treatment, especially on the battlefield.
6. Achieve moderate and consistent chamber pressures.
7. Function satisfactorily and reliably from belt and magazine fed firearms under sustained fire conditions.

8. Be relatively cheap and readily manufactured during periods of emergency such as war.

9. Retain the bullet for a period after ignition to allow the propelling gas pressure to build up sufficiently to achieve peak performance.

10. Effectively seal the chamber during discharge.

The type of brass used is very important to the manufacturing process and manufacturers carefully specify the quality of the brass to be used.

Four examples of specifications are as follows (40):-

(a) 68% to 74% copper and 32% to 26% zinc. Impurities must not exceed 0.2% nickel, 0.15% iron, 0.1% lead, 0.05% arsenic, 0.05% cadmium, 0.008% bismuth. Tin and antimony must be absent and there must not be more than a trace of any other impurity.

(b) 65% to 68% copper and 35% to 32% zinc with up to 0.2% nickel. There must be no individual impurity in excess of 0.1% and no more than 0.1% lead, 0.05% iron and 0.03% phosphorus.

(c) 70% copper and 30% zinc with not more than 0.25% of all other impurities combined.

(d) 72% to 74% copper and 28% to 26% zinc. Impurities must not exceed 0.1%, and there must not be more than 0.1% lead and 0.05% iron.

When a round of ammunition is discharged in a firearm the internal gas pressure, and to a much lesser extent the temperature rise, causes the cartridge case to expand tightly against the chamber walls. [This is an extremely important function of the cartridge case as this prevents the rearward escape of gas. Such an escape of gas would reduce the velocity of the projectile and consequently the efficiency of the firearm and could possibly cause a malfunction in the firearms mechanism.]

If the brass in the cartridge case is too soft it will not spring back from the chamber walls, which will probably make extraction of the spent cartridge case very difficult. If the brass is too hard the cartridge case could crack because it is too brittle and jam the firearms mechanism. When the brass in the cartridge case is of the correct hardness, it springs back to its near original dimensions and the spent cartridge case is readily extracted.

For higher velocity ammunition the hardness of the brass usually decreases from the base to the neck of the cartridge case. Cartridge cases for low velocity ammunition are normally made to a standard hardness along their entire length.

The base of a cartridge case must be strong enough to withstand ramming and extraction (this can happen numerous times to an individual round of ammunition during loading and unloading procedures) whilst the neck of the case must be strong enough to rigidly support the bullet yet flexible enough to expand and seal the chamber during discharge. High temperature discharge gases can raise the pressure inside the cartridge case to 40,000 pounds per square inch in a very short time period (41).

As the cartridge case is subjected to considerable stresses during loading, firing and extraction, case thickness as well as case hardness needs to be carefully controlled. There must be a sufficient thickness of metal at the base of a cartridge to sustain the severe back thrust that occurs during discharge. If the metal walls of the cartridge case are too thin, the extension of the cartridge case due to longitudinal stress may cause it to fracture or the wall to separate from the base. For these reasons the thickness of metal in a cartridge case is carefully controlled and decreases from the base to the neck. The need to keep the weight to a minimum is another factor that is taken into account at the design stage.

A large quantity of propellant is required for modern high velocity ammunition and this is accommodated by enlarging the diameter of the case over most of its length before markedly reducing the diameter at the forward end to accept the bullet. High velocity cartridge cases are tapered and necked in order to avoid extraction difficulties which would be experienced if cylindrical cases were used in firearms with high chamber pressures. Most low velocity cartridge cases are also slightly tapered.

The feeding and extraction mechanism of the firearm coupled with the type of ignition system dictates the design of the base of the cartridge case. Nearly all cartridge cases have the outside surface of the base indent stamped by the maker (headstamp). Information such as the makers initials, code or mark; year of manufacture (mainly military ammunition); calibre or other coded information are indent stamped into the base. It is sometimes possible, even for old ammunition, for a manufacturer to check their records and give the complete specification of a round of ammunition from the headstamp details.

The joint between the primer cup and the outside of the cartridge case base is sealed with lacquer to prevent the ingress of moisture and oil. The lacquer is sometimes colour coded as an aid to visual inspection during manufacture, and also sometimes to identify the type of bullet, e.g. ball, tracer, armour piercing. Sometimes the mouth of the case is internally varnished, just before inserting the bullet, to waterproof the joint and to provide resistance to the pressure of the propellant gases.

Bogus headstamps are sometimes encountered when a Government, for political or economic reasons, is supporting a rebel cause in another country by supplying the rebels with ammunition. For obvious reasons the source of supply is disguised. This can be done by omitting the headstamp or by using fake headstamps (42). It is not unknown for such ammunition to be headstamped in such a way as to attempt to place the blame for supply on some other Government.

2.1.2 Primer cups (caps)

Priming compositions for centre fire ammunition are housed in small metal cups which fit into a recess, called the primer pocket, in the centre of the base of the cartridge case. In rimfire ammunition the priming composition is housed inside the cartridge case in the hollow perimeter of the base.

The ideal primer cup metal should expand easily to provide a gas tight seal, be strong enough to withstand the blow from the firing pin and also strong enough to withstand the 'explosion' of the priming composition and the discharge gas pressure, even though it has been severely dented by the firing pin.

Primer cups are usually made of cartridge brass, although copper, nickel plated copper or brass, copper alloy, cupro nickel and zinc coated steel cups are also encountered. Primer cups for use with black powder were usually made of soft copper because of the weaker firing pin blow experienced with old black powder firearms, and the much lower pressures generated by black powder discharge. On the other hand smokeless powders typically give much higher pressures than black powder and are much harder to ignite. Smokeless powders require a much 'hotter' primer which needs a much stronger blow from the firing pin. Consequently soft copper cups are only suitable for use with low pressure ammunition.

Two specifications for primer cup metal are (a) 95% to 98% copper and 5% to 2%zinc with not more than 0.05% iron, 0.05% lead, 0.1% arsenic, 0.002% bismuth, 0.01% antimony and no more than a trace of any other impurity (43); (b) 72% to 74% copper and 28% to 26% zinc with the total impurities not exceeding 0.1% and not more than 0.1% lead and 0.05% iron (44).

There are two types of primers used in centre fire ammunition which differ only in physical design. In European countries, the Berdan primer design is preferred, whereas in Canada and the United States the Boxer primer design is favoured. The only difference between the two types is the design; the Berdan primer does not have an integral anvil, the anvil being part of the cartridge case, whereas the Boxer primer has its own anvil which is inserted into the primer cup.

Figure 2.01 illustrates the different primer systems.

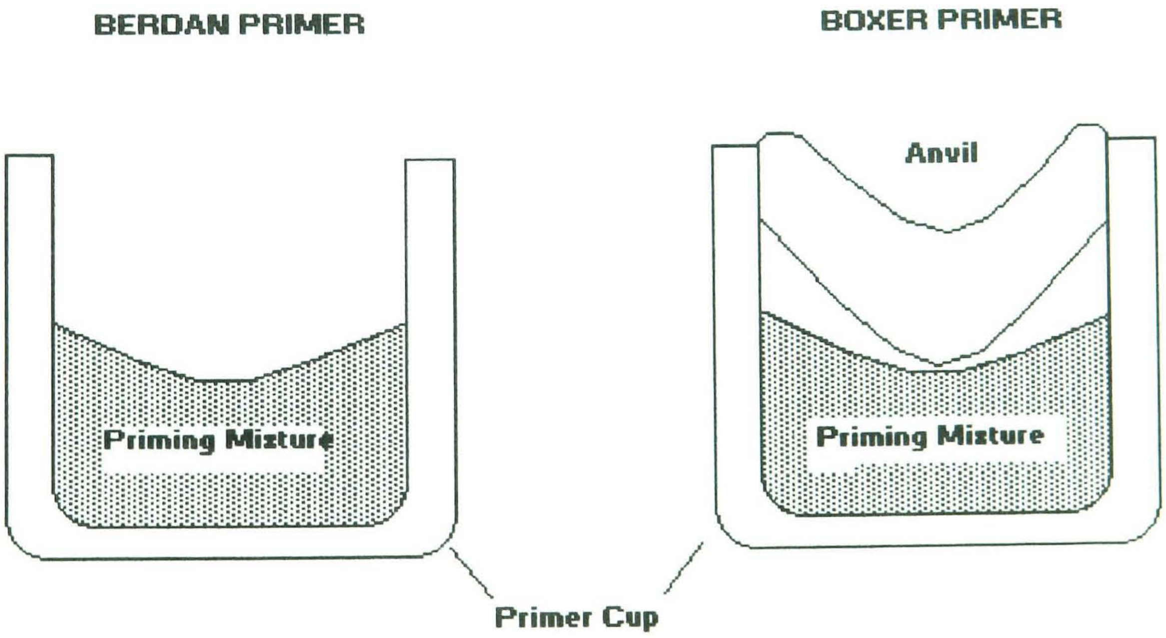


Figure 2.01 Berdan and Boxer Primers

The Berdan cup is varnished internally when empty, and after filling it is covered with a paper disc and then sealed with varnish.

Cupro nickel and copper alloy cups that are filled with a mercury fulminate based primer composition are closed with a tinfoil disc that is varnished on the side that is in contact with the primer composition. A varnish that is frequently used for this purpose is Shellac grade 1. After fitting, the cup annulus is coated with a clear or coloured varnish to prevent the ingress of moisture or oil.

Generally speaking primers for rifles differ in size, structure and amount of priming composition from those used for pistols and revolvers. Primer cups for use in rifles, pistols and revolvers range in size from 0.175" diameter to 0.210" diameter. For shotgun cartridges, the primer cup is typically in the range 0.240" diameter to 0.245" diameter.

Whilst pistol and revolver primer cups may have the same diameter as rifle primer cups, rifle primers have a greater cup metal thickness and contain larger amounts of priming composition which is accommodated by a longer primer cup length. The increased thickness of rifle primers is necessary because of the heavier blow they receive from the firing pin and the higher working pressures experienced. The larger amount of priming composition is necessary because of the larger amount of propellant used in rifle ammunition.

The weight of primer composition for pistol, revolver, rifle and shotgun ammunition can range from as little as 0.013g to as much as 0.352g depending on the calibre and type of ammunition, but is typically in the region of 0.05 g to 0.12 g.

2.1.3 Priming compositions

Priming compositions for firearms ammunition are mixtures which, when subjected to percussion, provide a sudden burst of flame that serves to ignite the propellant within the cartridge case. A priming composition must deliver a relatively large volume of hot gas and hot solid particles without the development of a detonating wave.

The ideal priming composition would consist of a cheap, readily available, relatively safe to handle, simple chemical compound of uniform granulation which when subjected to impact would undergo rapid, highly exothermic decomposition. The only compound to even approach these specifications is lead dinitroresorcinate; however, it is far too sensitive. In practice, no single chemical compound meets all the requirements of an ideal primer.

The next most desirable type of priming composition would be a mixture of compounds that, although individually non-explosive, sensitize each other to ignition and rapid burning. In fact, most priming compositions consist of mixtures of one or more initial detonating agents, with oxidizing agents, fuels, sensitizers and binding agents. The net effect of the additions is to dilute the initial detonating agent so as to convert its decomposition from detonation into rapid combustion. In some cases a single addition may serve two purposes, e.g. antimony sulphide acts as a fuel as well as a sensitizer to friction, and gum arabic acts as a fuel and a binding agent. The additions may also serve to increase the volume of gases produced per unit weight of priming composition, to prevent the gases from having too high a temperature, and to contribute incandescent solid particles to the decomposition products.

The sensitivity of priming compositions varies, but that of an individual composition can also be varied to some extent by careful control of the granulation of each of the ingredients. Sometimes this is more important than the proportions of the ingredients. Non-uniformity of composition due to physical separation caused by shaking, can cause great variations in sensitivity and even failure to function. The presence of a binding agent prevents such separation as well as fixing the composition in the desired position in the assembly.

The rate of burning, volume of gases, weight of solid particles produced and the duration of the flame are the major influences upon the efficient functioning of a priming composition. For a typical priming composition of 0.15g, the volume of gas at room temperature and pressure is in the order of 1.5cm^3. The percentage of the weight carried as incandescent particles by the hot gases will vary with the composition, but can be in the region of 70%. The incandescent particles are thought to promote ignition by thermal radiation. Flame bursts from various primers were found to have effective durations varying from 400 to 750 microseconds and total durations varying from 650 to 1500 microseconds (45).

Generally speaking small arms primers consist of an explosive, an oxidizer, a fuel and a frictionator. Other compounds act as sensitizers and binders.

EXPLOSIVES used include azides, fulminates, diazo compounds, nitro or nitroso compounds, e.g. lead or silver azide, mercury fulminate, lead styphnate, TNT and PETN (which also act as sensitizers).

OXIDIZERS used include barium nitrate, potassium chlorate, lead dioxide and lead nitrate.

FUELS used include antimony sulphide (which also acts as a frictionator), gum arabic (which also acts as a binding agent), calcium silicide (which also acts as a frictionator), nitrocellulose, carbon black, lead thiocyanate and powdered metals such as aluminium, magnesium, zirconium or their alloys.

FRICTIONATORS used include ground glass and aluminium powder (which also acts as a fuel).

SENSITIZERS used include tetracene, TNT and PETN.

BINDERS used include gum arabic, gum tragacanth, glue, dextrin, sodium alginate, rubber cement and karaya gum.

The quantity of oxidizer in the mixture is calculated to supply at least enough oxygen for the complete combustion of the primer, otherwise combustion products that are harmful to the firearm could be formed. The frictionators could be regarded as sensitizers as they sensitize the mixture to percussion. There may be more than one explosive, oxidizer, fuel and frictionator in a single priming composition and sometimes a dye is added as an identifying feature or as an aid in production. Sometimes no single primary explosive is present, the mixture itself being the primary explosive.

In 1805 the Reverend Alexander Forsyth used mercury fulminate as the basis of his primer composition, and from this time the percussion system developed into todays highly reliable, universally used, percussion primer compositions. This development which started in 1805, still continues today, and manufacturers are very reluctant to release details of their compositions. Consequently information on primer compositions and the chemical composition of ammunition is both sparse and fragmented in the literature.

It is accepted by most writers that the Reverend Alexander Forsyth's percussion priming composition was based on mercury fulminate. However there is some respected opinion which suggests that his composition was made up of wax coated pellets of potassium chlorate mixed with combustible materials, and that it was not until 1831 that mercury fulminate was widely used as the explosive ingredient in primer compositions (46, 47).

Early priming compositions consisted of mercury fulminate and potassium chlorate along with other ingredients. With the introduction of metallic cartridge cases about 1850, it was found that brass cartridge cases were unsuitable for use with priming compositions containing mercury fulminate as the brass was embrittled due to mercury amalgamation of the zinc. This made the spent cartridge case useless for reloading purposes, and reloading was essential for economic reasons. Initially the use of copper cartridge cases solved this problem. In 1869, Hobbs, by the use of internal varnishing of brass primer cups and brass cartridge cases, made the use of brass and mercury fulminate possible by preventing the direct contact of the brass surface with the primer mix.

Whenever black powder was used as a propellant a large amount of fouling was deposited on the inside of the barrel. On combustion, black powder produces 44% of its original weight as hot gases and 56% as solid residues in the form of dense white smoke (48). When smokeless powders were introduced between approximately 1870 and 1890, another major problem was encountered. Smokeless powders were harder to ignite than black powder; consequently, larger priming loads were necessary for smokeless powders. Higher pressures were experienced with smokeless powders, and smokeless powders on combustion produced much less fouling than black powder. The relatively clean surfaces remaining in the barrel interior after the combustion of smokeless powder became rusted, even when the gun was cleaned immediately after use.

The cause of the rusting was traced to the potassium chlorate used in the priming composition. Potassium chloride, formed after the combustion of potassium chlorate, was deposited inside the barrel; it then attracted atmospheric moisture and caused rapid rusting of the barrel interior. Gun cleaning mixtures were organic in nature and did not dissolve the potassium chloride; consequently despite cleaning immediately after use, salt particles trapped in the rifling and surface imperfections of the metal still caused rusting. Water proved to be efficient at removing all traces of the salt; however, it was then necessary, and very difficult, to ensure that all the water was removed from the gun, otherwise the water itself would cause rusting. The heavy residue left after the combustion of black powder substantially protected the metal surfaces from the effects of the salt, and to some extent from the effects of metallic mercury released after combustion of the primer.

The problems associated with the use of mercury fulminate and potassium chlorate led to a search for suitable alternatives, and the chemical reactions occurring within the cartridge case and the firearm were intensively studied. The objective of the study was to produce a satisfactory priming composition which was both non-corrosive and non-mercuric (NCNM).

Due to the need to reuse spent cartridge cases for economic reasons, there has been no mercury in United States military small arms primers manufactured since 1898. It was used to a later date (about 1930) in certain United States commercial primers. In 1898 the United States military adopted a non-mercuric primer composition, coded H-48 for use in the .30 Krag cartridge. The primer composition was: -

Potassium Chlorate	49.6%
Antimony Sulphide	25.1%
Sulphur	8.7%
Glass Powder	16.6%

During the First World War the non-mercuric primer mixture used was:-

Frankford Arsenal FH-42 (1910)	Potassium Chlorate	47.20%
	Antimony Sulphide	30.83%
	Sulphur	21.97%

It was discovered in 1911 that thiocyanate-chlorate mixtures were sensitive to impact, and this led to the Winchester Repeating Arms Company's 35-NF primer composition:-

Potassium Chlorate	53%
Antimony Sulphide	17%
Lead Thiocyanate	25%
TNT	5%

After a batch of damp sulphur and/or impure potassium chlorate (polluted with potassium bromate) caused 'dead' primers in millions of rounds of ammunition with Frankford Arsenals FH-42 primer mix this primer composition was abandoned. Frankford Arsenal adopted the Winchester Repeating Arms Company's 35-NF primer mix which was then standardised as FA-70 and was used in 0.45ACP and .30-06 ammunition throughout World War II and into the 1950's.

At this time a typical .22" calibre rimfire primer composition was the United States Cartridge Company's "NRA" which was:-

Potassium Chlorate	41.43%
Antimony Sulphide	9.53%
Copper Thiocyanate	4.70%
Ground Glass	44.23%

It would appear that the Germans were approximately 23 years ahead of the Americans in the production of non-corrosive primers, despite the fact that the German compositions were published in the open literature. This may have been due to patent rights.

The first non-corrosive primer was produced by the German firm of Rheinische-Westphalische Sprengstoff AG in 1901 (RWS).

The mixture was:-

Mercury Fulminate	39%
Barium Nitrate	41%
Antimony Sulphide	9%
Picric Acid	5%
Ground Glass	6%

(Barium Nitrate replaced Potassium Chlorate)

In 1910 the same firm produced the following .22" calibre rimfire priming composition:-

Mercury Fulminate	55%
Antimony Sulphide	11%
Barium Peroxide	27%
TNT	7%

The Swiss Army had also been using a non-corrosive primer mix since 1911 which was:-

Mercury Fulminate	40%
Barium Peroxide	25%
Antimony Sulphide	25%
Barium Carbonate	6%
Ground Glass	4%

It was not until 1927 that the first American commercial non-corrosive primers appeared on the market. Some of these are as follows (49):-

	Remington Kleanbore	***Western***	***Winchester Staynless***	***Peters Rustless***
Mercury Fulminate %	44.40	40.79	41.06	38.68
Barium Nitrate%	30.54	22.23	26.03	9.95
Lead Thiocyanate %	4.20	8.22	5.18	-
Ground Glass %	20.66	28.43	26.66	24.90
Lead Compound (?) %	-	-	-	25.91
Binder Gum%	0.20	0.33	0.58	0.56

Up to this time, primers had fallen into three categories, mercuric and corrosive, non-mercuric but corrosive, and mercuric but non-corrosive. Because of the disadvantages of mercury fulminate and potassium chlorate the main objective of primer development was to produce a primer with satisfactory ignition properties without the use of these two compounds. An early non-corrosive, non-mercuric priming composition used copper ammonium nitrate to replace mercury fulminate, and potassium nitrate to replace potassium chlorate.

The composition was :-

Copper Ammonium Nitrate	30-40%
Potassium Nitrate	42-25%
Sulphur	10-7%
Aluminium	18-28%

The first practical NCNM primer mixture with satisfactory ignition properties and good shelf life was produced by RWS in 1928. This type of primer was given the general name of "Sinoxyd" (Sinoxide/Sinoxid) and has the following general composition:-

Lead Styphnate	25-55%
Barium Nitrate	24-25%
Antimony Sulphide	0-10%
Lead Dioxide	5-10%
Tetracene	0.5-5%
Calcium Silicide	3-15%

Glass Powder	0-5%

This was the forerunner of all modern NCNM priming compositions. With very few exceptions, USA commercial primers became non-corrosive about 1931 but because of stringent US Government specifications for military ammunition which could not be met by the earlier versions of the new NCNM primer mixtures, it was not until the early 1950's that US military ammunition became non-corrosive. This was due to the fact that early NCNM commercial priming mixtures suffered erratic ignition and unsatisfactory storage stability, and as large quantities of small arms ammunition are stored as a war reserve, military ammunition must have unquestioned reliability and storage stability.

In the United Kingdom both commercial and military ammunition used primers that were both mecuric and corrosive, until the gradual changeover to NCNM primers which was completed during the mid-fifties and early sixties.

The explosive ingredient in "Sinoxyd" type primers is lead styphnate (lead trinitroresorcinate) which is very sensitive to static electricity, and fatalities have resulted from handling the dry salt. Preparation of the pure salt is difficult, and many patented preparations, including basic modifications, exist. Some claim special crystalline forms and/or reduced static electricity hazard. Explosive ingredient substitutes for lead styphnate were sought that would be easier to make, and safer to use. These included lead azide, diazonitrophenol, lead salts of many organic compounds, complex hypophosphite salts, picrate-clathrate inclusion compounds and pyrophoric metal alloys.

In 1935 lead azide was patented for use in priming mixtures in the following mix:-

Lead Azide	12%
Barium Nitrate	23%
Antimony Sulphide	20%
Calcium Silicide	10%
Tetracene	3%
Lead Dioxide	20%
Lead Thiocyanate	12%

In 1939 a primer mixture was patented that was identical to "Sinoxyd" except that diazonitrophenol was substituted for lead styphnate. Heat, humidity and copper have a detrimental effect on diazonitrophenol and it is no longer used in primer mixes. Normal lead styphnate has one lead atom per formula unit, whereas the basic form has two. A priming mixture using basic lead styphnate was patented in 1949 and consisted of:-

Basic Lead Styphnate	40%
Barium Nitrate	42%
Antimony Sulphide	11%
Nitrocellulose	6%
Tetracene	1%

Other substitutes for lead styphnate included lead salts of many organic compounds, none of which gained widespread acceptance.

It was not until 1954 that preparation of the pure compound, normal lead styphnate hydrate, was accomplished. Up to this time the impure salt (~93%) was used extensively.

Complex hypophosphite salts have been used successfully as substitutes for both lead styphnate and tetracene.

A 1939 patent gives the following composition:-

Lead Styphnate	33%
Calcium Hypophosphite	7%
Lead Nitrate	14%
Lead Thiocyanate	10%
Barium Nitrate	16%
Glass Powder	20%

When wet with water a reaction occurs between the calcium hypophosphite and the lead nitrate, producing a shock sensitive non-hygroscopic compound which incorporates both oxidizer and fuel.

In 1944 a patented rimfire priming mix included a triple salt, i.e. basic lead styphnate.lead styphnate.lead hypophosphite, in the following mix:-

Triple Salt	50%
Lead Nitrate	30%
Glass Powder	20%

In 1955, patents were issued for a nontoxic, lead-free, rimfire priming mixture which used the double salt, ferric styphnate.ferric hypophosphite, and for a glassless rimfire priming mixture using a triple salt, potassium styphnate.lead styphnate.lead hypophosphite, in the following unusual mixture:-

Triple Salt	10%
Lead Styphnate	36%
Barium Nitrate	50%
Tetracene	4%

About 1949 Frankford Arsenal manufactured an unusual priming mixture known as the P-4 primer (coded FA675):-

Stabilized Red Phosphorus	18%
Barium Nitrate	82%

Whilst this was a simple, relatively safe mixture, and was a satisfactory primer, it was discontinued after a very short period because of two major disadvantages. It was shown that copper, bismuth, silver, iron and nickel increased the oxidation rate of red phosphorus to acidic compounds. Primer cups had to be zinc plated to prevent contact with copper. The red phosphorus had to be of high purity, and it was necessary to remove the major impurities (iron and copper) from commercial red phosphorus before use, and to coat the purified material with up to 7.5% aluminium hydroxide which inhibited oxidation.

Although the P-4 primer was only in use for approximately 1 year, it was further improved in 1961 by coating the stabilized red phosphorus with PETN, RDX or TNT giving the following primer mix:-

Stabilized Red Phosphorus	25%
PETN, RDX or TNT	5%
Barium Nitrate	70%

However red phosphorus primers never achieved widespread use, presumably due to manufacturing difficulties.

In the early 1960's important advances were made in the development of safer, easier to make, cheaper and better substitutes for lead styphnate, which had been the main explosive ingredient in successful NCNM priming mixtures up to this time.

In 1962 Kenney applied for patents on many complex, basic lead picrate-clathrate inclusion compounds which did not have the static electricity hazard of lead styphnate. Of 44 compounds listed in his patent, monobasic lead picrate.lead nitrate.lead acetate was preferred for primers, although monobasic lead picrate.lead nitrate.lead hypophosphite; dibasic lead picrate.lead nitrate.lead acetate and monobasic lead picrate.lead nitrate.lead acetate.lead hypophosphite were also suitable. Glass was thought to damage the bore of the firearm and was considered by some to be undesirable.

A glassless rimfire mixture was:-

Any of the previous complex salts	46%
Barium Nitrate	50%
Tetracene	4%

In 1962, Staba applied for patents on the double salt, lead nitroaminotetrazole. lead styphnate, which become known as 'stabanate', and had much better thermal stability than lead styphnate.

A primer mix claimed to be superior to the lead styphnate based equivalent was:-

Stabanate	20%
Barium Nitrate	50%
Antimony Sulphide	15%
Tetracene	5%
Aluminium	10%

In 1966, Staba applied for a patent on certain forms of carbon that exhibit conchoidal fracture (very sharp, jagged concave edges) when shattered. A rimfire primer mix was:-

Lead Styphnate	20.00%
Stabanate	25.00%
Barium Nitrate	36.25%
Tetracene	3.00%
Karaya Gum	0.75%
Ground Anthracite coal	15.00%

Another of Staba's primer mixes was:-

Stabanate	48.5%
Tetracene	3.0%
PETN	14.0%
Aluminium	10.0%
Nitroaminoguanidine	23.0%
Karaya Gum	1.0%
Gum Arabic	0.5%

An interesting stage in the development of primer mixes was the use of pyrophoric metal alloys, first patented in 1936 and improved in 1964. These rare-earth alloys, as used in cigarette lighter flints, give a shower of sparks when lightly scraped. A typical pyrophoric alloy is 'misch metal' which has the following approximate composition:- cerium 50%, lanthanum 40% other rare earth elements 3% and iron 7%.

There are many patents listed in which the pyrophoric alloy replaces the function of both lead styphnate and tetracene.

One of the most sensitive mixtures was:-

Misch metal/Magnesium (80/20 alloy)	50%
Barium Nitrate	20%
Lead Dioxide	10%
Zirconium Powder	20%

Pyrophoric alloy primer mixtures never achieved widespread use, presumably because of their lack of sensitivity to percussion (50). There are hundreds of patents issued for priming compositions, a fact which illustrates the considerable experimentation in this area.

Examples of some of these are:-

Mercury Fulminate	20-50%
Barium Nitrate	19-45%
Lead Chromate	2-20%
Lead Sulphocyanide	3-25%
Zirconium Powder	2-30%
Glass Powder	30%

Basic Lead Trinitro-resorcinol	27%
Lead Dinitrophenylazide	13%

Potassium Nitrate	30%
Antimony Trisulphide	7%
Ground Glass	23%

Mercury Fulminate	33%
Thallium Nitrate	40%
Cobalt Nitrate	10%
Antimony Trisulphide	17%

Potassium Chlorate	85.0%
Asbestos Fibre	1.5%
Nitrotoluol	4.5%
Petroleum Gel	8.5%
Castor Oil	0.5%

Potassium Chlorate	48-53½%
Potassium Ferrocyanide	33⅓-36%
Glass Powder	13⅓-16%
Tetrazene	1-4%
Diazonitrophenol	12-18%

Barium Nitrate	25-40%
Antimony Trisulphide	8-18%
Lead Peroxide	15-25%
Calcium Silicide	8-20%

Tetrazene	4-7%
Diazonitrophenol	15-20%
Basic Lead Azide	6-12%
Barium Nitrate	20-30%
Lead Peroxide	12-20%
Ground Glass	20-28%

Lead Azide	20-25 oz
Powdered Glass	20-25 oz
Flake Aluminium	6-8 oz
Barium Nitrate	35-38.5 oz
Trinitrotoluol	0-25 oz
Canada Balsam or Cellulose Acetate	0-2.5 oz
m-Toluenesulphomethylamide	0-1 oz

Mercury Fulminate	65.0 g
Barium Nitrate	22.0 g
Antimony Sulphide	11.0 g
Hexogene	15.5 g
Barium Carbonate	1.5 g

Gum Arabic	30 g
Phosphorus Sulphide	15 g

Magnesium Carbonate	12 g
Calcium Carbonate	5 g
Potassium Chlorate	60 g

Mercury Fulminate	37.5%
Potassium Chlorate	37.5%
Antimony Sulphide	25.0%

Mercury Fulminate	25.9%
Potassium Chlorate	48.2%
Antimony Sulphide	3.7%
Ground Glass	22.2%

Mercury Fulminate	19.1%
Potassium Chlorate	33.3%
Antimony Sulphide	42.8%
Sulphur	2.4%
Mealed Powder	2.4%

Lead Trinitroresorcinol	40%
Tetracene	2%
Barium Nitrate	40%
Lead Oxide	3%
Calcium Silicate	11%
Powdered Glass	4%

Despite the search for alternatives to lead styphnate and the considerable experimentation with primer compositions, in the UK and the USA, the vast majority of modern ammunition contains "Sinoxyd" type primers with lead styphnate and barium nitrate together typically making up 60% to 80% of the total weight.

They also contain some of the following:-

Antimony Sulphide
Tetracene
Calcium Silicide
Lead Dioxide
Aluminium Powder
Ground Glass
Lead Hypophosphite
Lead Peroxide
Zirconium
Nitrocellulose
Pentaerythritol Tetranitrate
Gum Type Binder

Composition control is very stringent and ingredients are of analytical reagent quality.

Mercury fulminate/potassium chlorate based primer compositions are currently manufactured by some Eastern Bloc countries, although they also manufacture compositions based on lead styphnate.

Examples of some modern USA priming mixtures are (51):-

Normal Lead Styphnate	36%
Barium Nitrate	29%
Antimony Sulphide	9%
Lead Dioxide	9%
Tetracene	3%
Zirconium	9%
Pentaerythritol Tetranitrate	5%

Basic Lead Styphnate	39%
Barium Nitrate	40%
Antimony Sulphide	11%
Tetracene	4%
Nitrocellulose	6%

Normal Lead Styphnate	37%
Barium Nitrate	38%
Antimony Sulphide	11%
Tetracene	3%
Pentaerythritol Tetranitrate	5%
Nitrocellulose	6%

Normal Lead Styphnate	41%
Barium Nitrate	39%
Antimony Sulphide	9%
Calcium Silicide	8%
Tetracene	3%

Normal Lead Styphnate	43%
Barium Nitrate	36%
Calcium Silicide	12%
Tetracene	3%
Lead Peroxide	6%

Examples of some modern UK priming mixtures are (52):-

Lead Styphnate	35%
Tetracene	3%
Lead Peroxide	15%
Barium Nitrate	47%

Lead Styphnate	46%
Tetracene	4%
Barium Nitrate	25%
Antimony Sulphide	20%
Aluminium	5%

Lead Styphnate	44.2%
Tetracene	3.3%
Barium Nitrate	20.4%
Ground Glass	25.0%
Lead Hypophosphite	6.8%
Gum Arabic	0.3%

Lead Styphnate	38%
Tetracene	2%
Lead Peroxide	5%
Barium Nitrate	39%
Antimony Sulphide	5%
Calcium Silicide	11%

An interesting and extremely successful primer innovation was introduced by Eley and is known as Eleyprime. Instead of using lead styphnate, with its inherent safety and processing difficulties, they use calculated amounts of lead monoxide and styphnic acid which are much safer to process. At the end of the processing stage a drop of water is added to each individual primer which initiates a chemical reaction between the lead monoxide and the styphnic acid to form lead styphnate. The final product when dry is no different from a conventional primer.

In conventional ammunition lead, antimony and barium are emitted when the ammunition is discharged. These three elements are undesirable from a health viewpoint and pose a major problem for firearms instructors in indoor firing ranges, as they are exposed to an unhealthy environment each working day. To solve this problem Dynamit Nobel AG developed a non-toxic primer composition called "Sintox". Lead styphnate is replaced by 2-diazo-4, 6-dinitrophenol (diazole) and the barium nitrate and antimony sulphide are replaced by a mixture of zinc peroxide and titanium metal powder.

The 'Sintox' primer mixture contains tetracene, diazole, zinc peroxide/titanium powder and nitrocellulose ball powder (53). The use of this primer coupled with a totally jacketed bullet (base also enclosed) entirely eliminates the health hazard problem.

CCI and Fiocchi produce lead free primers; Fiocchi substituted diazole for the lead compound, and CCI uses diazole, manganese$^{(iv)}$ oxide and aluminium (54).

The use of titanium as a replacement for calcium silicide in conventional Sinoxyd primers is currently being investigated by Dynamit Nobel.

Primers are not used exclusively for firearms ammunition, but have other uses which include blank cartridges, flare tripwires, mortars, pyrotechnic cartridges, hand grenades, ejector seat mechanisms and other jettison devices.

2.1.4 Propellants

Small arms ammunition propellants may be defined as "explosive materials which are formulated, designed, manufactured, and initiated in such a manner as to permit the generation of large volumes of hot gases at highly controlled, predetermined rates" (55).

Ideally a propellant would be a single, solid, non-toxic chemical compound that is stable, easy to store, easy to ignite, of compact mass etc; which is cheap and simple to prepare from readily available materials and which on combustion produces no smoke or solid residue, i.e. is completely converted into gas or gases. It must contain its own oxygen supply which is necessary for combustion in confined spaces, it must burn very rapidly as opposed to detonation, and it must have a satisfactory energy/weight relationship.

It is not surprising that no single chemical compound fulfils all these specifications. In practice propellants consist of a mixture of substances.

A propellant must fulfil the following general specifications:-

1. It should be capable of being manufactured simply, rapidly, with relative safety, at reasonable cost and from ingredients that are readily obtainable in time of war (military propellants).

2. It must be easy and safe to load, non-hygroscopic, and free from combustion products that are difficult to remove or injurious to the firearm or cartridge case.

3. It must give consistent performance under varying conditions of storage and climate, and it must not deteriorate with age (this is especially applicable to propellants for military use which can be stored as a war reserve for a long period of time). It must also not ignite when in the chamber of a very hot firearm for a considerable period of time. (This also applies to priming compositions.)

The energy/weight/bulk relationship of a propellant and the rate of delivery of the energy must be matched to the system, i.e. space available within the cartridge case and gun barrel, the bullet weight, pressure requirements and the required bullet velocity. Consequently a wide range of propellants are required to satisfy the varying ballistic requirements of a wide range of firearms and ammunition.

The burning rate is of extreme importance because if the propellant releases hot gases too quickly, it detonates, thereby destroying the gun and possibly causing injury to the firer. If it burns too slowly, it is inefficient, and the bullet will lack sufficient velocity. The burning rate can be controlled by the size and geometrical design of the individual grains. (An individual propellant particle is referred to as a grain (kernal) and grains (kernals) can be very small with simple geometries, or very large with complex geometries.).

Apart from the inherent burning characteristics of an individual propellant the burning rate can also be varied by the use of surface coatings (moderants) on individual grains of propellant.

Propellants are frequently referred to as gunpowder, powder charge or simply as 'charge' or 'powder'. However they are very rarely a true powder and are manufactured in a wide range of colours, shapes and sizes. Figure 2.02 illustrates some shapes.

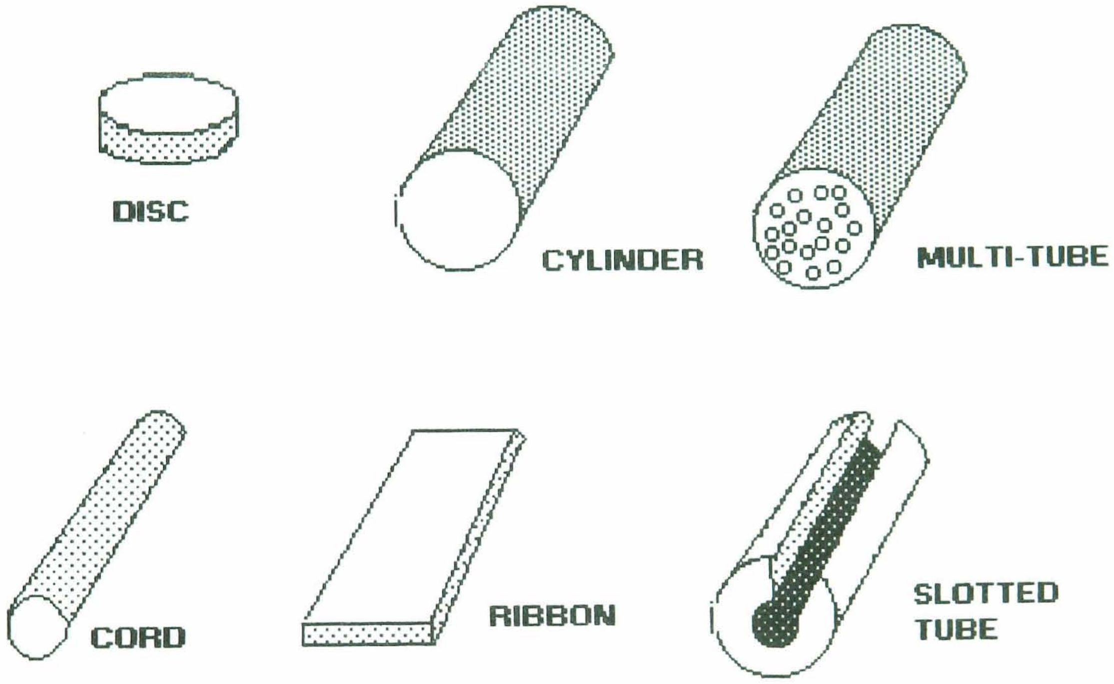


Figure 2.02 Propellant Shapes

It is critically important that propellant grains do not contain any non-uniformities such as cracks, pores and cavities, since this could cause internal grain burning, leading to detonation or excessive pressure.

The relationship between physical shape and burning rate is a complex one, dependent upon the characteristics of the propellant surface which affect the rate at which decomposition reactions occur, and also upon the characteristics of the environment above the propellant which affects the rate at which heat is transferred to the propellant surface to cause chemical breakdown. Both surface and gas phase theories are intimately related.

The process of delivery of propellant gases at a predetermined rate involves the selection of a propellant composition with the required burning rate at the operating pressure of the firearm, and then designing the propellant grains so that the necessary burning surface is available to provide the required mass rate of gas evolution, i.e. the necessary time/pressure relationship.

Since the introduction of smokeless powders in the period between 1870 and 1890, the use of black powder as a small arms ammunition propellant has substantially diminished. However black powder is still currently used as a propellant for some specialized purposes e.g. baton guns, punt guns, cable guns, signal flares and by black powder firearms enthusiasts. It is also used in blank rounds of various types, and in many other ammunition components designed for larger calibre guns.

Black powder is a mechanical mixture of charcoal, saltpetre (potassium nitrate) and sulphur in the typical proportion 15:75:10 respectively. The charcoal is the fuel, the saltpetre supplies the oxygen necessary for combustion in a confined space, and the sulphur is a binding agent which aids in holding the mixture together and to a much lesser extent also acts as a fuel. Black powder is black and granular in appearance and the burning rate is controlled by granulation size.

When black powder burns, the "initial portion" ignited undergoes a chemical reaction which results in the production of hot gases. The gases expand in all directions warming the next portion to the 'kindling' temperature. This then ignites producing more hot gases and raising the temperature of the next portion, and so on. As the black powder is confined in the cartridge case the pressure rises and the heat cannot escape, consequently,it is communicated rapidly throughout the mass. In a confined space the combustion becomes extremely rapid, consequently, the pressure rise is also extremely rapid.

Black powder burns to produce a dense white smoke which contains extremely small particles held temporarily in suspension by the hot combustion gases.

Analysis of the combustion products of a particular brand of black powder gave the following results (56):-

42.98% of its weight as gases, 55.19% solids and 1.11% water. Analysis of the solid products (% by weight) and of the gaseous products (% by volume) is as follows:-

Potassium Carbonate	61.03	Carbon Dioxide	49.29
Potassium Sulphate	15.10	Carbon Monoxide	12.47
Potassium Sulphide	14.45	Nitrogen	32.91
Potassium Thiocyanate	0.22	Hydrogen Sulphide	2.65
Potassium Nitrate	0.27	Methane	0.43
Ammonium Carbonate	0.08	Hydrogen	2.19
Sulphur	8.74		
Carbon	0.08		

Black powder can vary from brand to brand. Variations in percentage compositions between manufacturers are small, but different charcoals, types of saltpetre (purity), different moisture content etc., can result in different ballistic performances from basically similar mixtures. Owing to a temporary shortage of potassium nitrate during the first World War, sodium nitrate was used as a substitute. Ammonium nitrate has also been used as a substitute for potassium nitrate.

Brown powder (cocoa powder) represents the peak of development of black powder and was the most successful form of black powder exhibiting better burning characteristics. It was made in single perforated hexagonal or octagonal prisms. A partially burned brown charcoal made from rye straw was used which had colloidal properties and flowed under pressure, cementing the grains together. This made possible the manufacture of slow burning propellant containing little or no sulphur. A typical brown powder was brown charcoal 19%, saltpetre 78% and sulphur 3%. A sulphur free brown powder was brown charcoal 20% and saltpetre 80%.

A modern substitute for black powder is 'Pyrodex' It is safer to transport, store and use, and is cleaner burning than conventional black powder. Pyrodex incorporates both charcoal and sulphur but in much smaller proportions than in black powder, and potassium nitrate in addition to other ingredients. Pyrodex also contains potassium perchlorate, sodium benzoate and dicyandiamide (57).

Modern propellants for small arms ammunition almost exclusively contain plasticized cellulose nitrate (NC) as the major oxidizing ingredient. Various other chemicals are added for specific purposes:-

1. High energy oxidizing plasticizers such as nitroglycerine (NG) to increase performance;

2. fuel type plasticizers such as phthalates or urethane to improve physical and processing characteristics;

3. organic crystalline chemicals such as nitroguanidine to moderate the ballistic characteristics;

4. stabilizers such as diphenylamine, centralities or acardites (e.g. N,N'-diphenylurea), to increase chemical stability by combining with decomposition products;

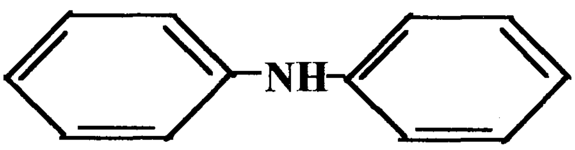
5. a range of inorganic additives such as chalk, graphite, potassium sulphate, potassium nitrate, barium nitrate, to improve ignitability, facilitate handling and minimize muzzle flash;

6. powdered metals are sometimes added to change thermal characteristics such as conductivity.

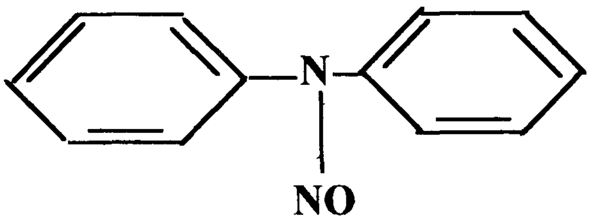
Some manufacturers also add coloured taggants to aid in identifying their product.

Propellants that contain nitrocellulose as the only oxidizer are referred to as single base and propellants that contain both nitrocellulose and nitroglycerine or other explosive plasticizers, as double base. Triple base propellants are produced when substantial quantities of an organic, energy producing, crystalline compound such as nitroguanidine are incorporated in double base propellants. Triple base propellants are unlikely to be encountered in small arms ammunition.

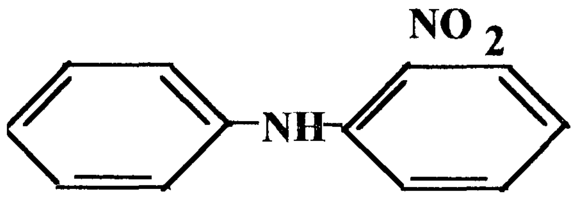
Stabilizers are necessary because nitrocellulose decomposes with age. The decomposition reaction yields dinitrogen tetraoxide which acts as an autocatalyst and accelerates the decomposition (58). Stabilizers act as dinitrogen tetraoxide scavengers, consequently shelflife is increased. A common stabilizer is diphenylamine or its nitro derivatives.



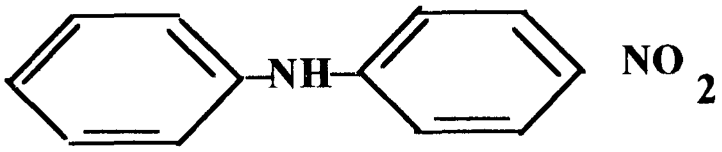
diphenylamine



N-nitrosodiphenylamine (diphenylnitrosamine)



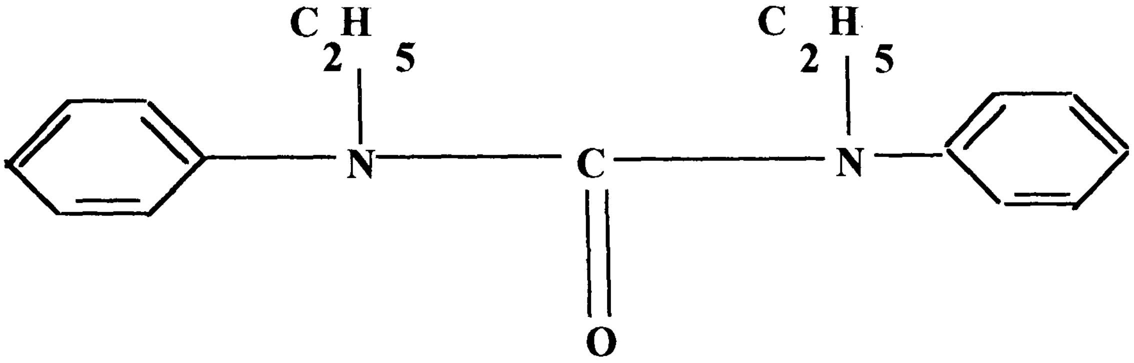
2-nitrodiphenylamine



4-nitrodiphenylamine

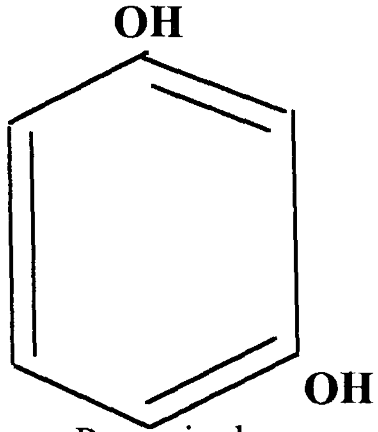
Diphenylamine is the most common stabilizer especially in single base powders. It has been suggested that diphenylamine is not a good stabilizer for double base propellants as it may hydrolyse NG (59).

Another common stabilizer is ethyl centralite, although sometimes methyl centralite is used (60). Methyl centralite is also used as a moderant to reduce the burning rate. Ethyl centralite is usually found in double base propellants.



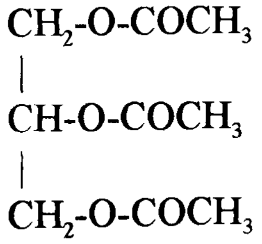
Ethyl Centralite (Smy-diethyl diphenylurea)

Resorcinol is also used as a stabilizer



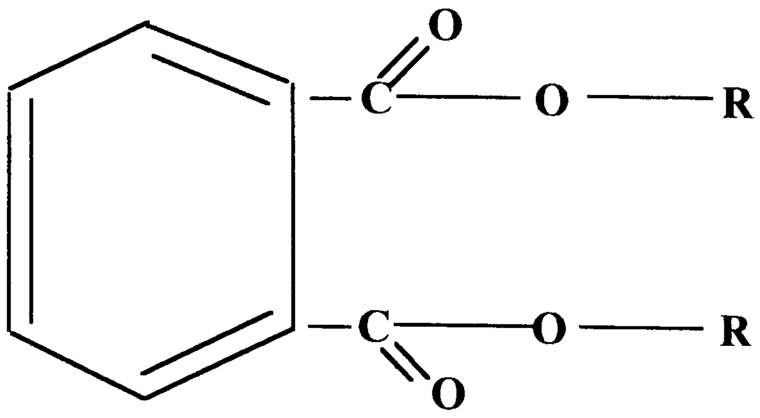
Resorcinol

Plasticizers add strength and flexibility to the propellant granules. Examples of some plasticizers used are (61, 62):-



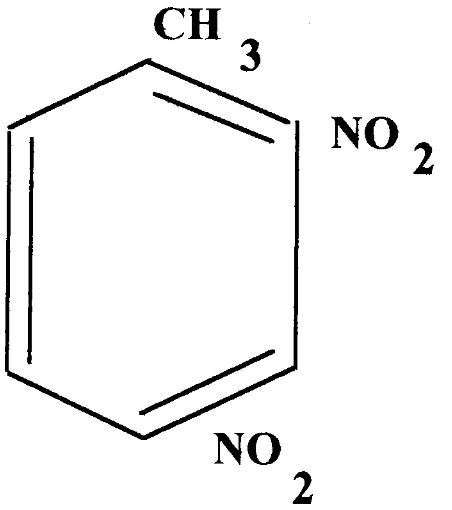
$$\begin{array}{l} CH_2\text{-O-COCH}_3 \\ | \\ CH\text{-O-COCH}_3 \\ | \\ CH_2\text{-O-COCH}_3 \end{array}$$

Triacetin (Glyceryl triacetate)

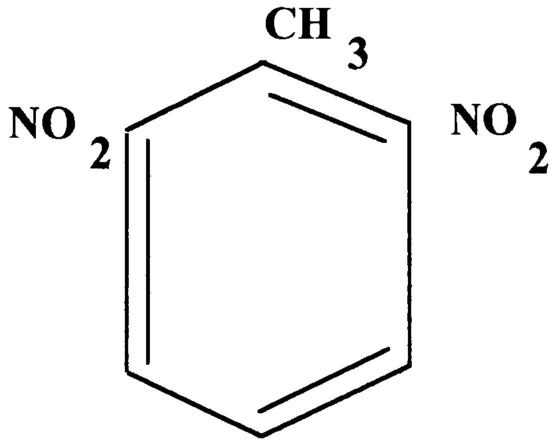


R is CH_3	dimethyl phthalate
R is C_2H_5	diethyl phthalate
R is C_4H_9	dibutyl phthalate

Muzzle flash suppressors include dinitrotoluene



2, 4 dinitrotoluene



2, 6 dinitrotoluene

Dinitrotoluene acts as a flash suppressor by reducing the heat of explosion. Nitroguanidine (picrite) is another flash suppressor which acts by producing nitrogen, thereby diluting the combustible muzzle gases. Examples of single and double base propellant compositions are given in Tables 2.01 and 2.02.

Smokeless powders leave relatively little solid residue on combustion and produce much less smoke than black powder. Combustion of smokeless powders produces primarily nitrogen, carbon monoxide, carbon dioxide, hydrogen and water vapour. The quantity of smokeless powder varies depending on the calibre, bullet weight/type, required pressure/velocity, space available within the cartridge/chamber etc. Ammunition for use in rifles contains propellant varying in weight from ~0.45 g (6.9 grains) for a .22" calibre to ~6.45 g (99.5 grains) for a .378" calibre. For pistols/revolvers the range can vary from ~0.06 g (0.9 grains) for a .25" calibre to ~1.72 g (26.5 grains) for a .44" Magnum calibre. For shotguns the range can vary from ~1.10 g (17.0 grains) for a 20 bore calibre to ~2.0 g (30.9 grains) for a 12 bore calibre.

SINGLE BASE PROPELLANTS (% COMPOSITION)

Nitrocellulose (NC)	89.0	99.0	97.7	90.0	79.0	85.0	87.0	96.25	94.25	94.0	98.0	99.4	92.4
Barium nitrate	6.0						6.0						
Potassium nitrate	3.0						2.0				0.25		
Starch	0.75												
Paraffin oil							4.0						
Diphenylamine	1.0	1.0	0.8	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	0.6	0.6
Dinitrotoluene				8.0		10.0							6.5
Methyl centralite								2.0	2.0	1.75			
Dibutyl phthalate				1.0		4.0			2.0	1.75			
Glyceryl triacetate					5.0								
Tin			0.75							0.8			
Graphite					0.2			With NC	With NC		0.5		0.5
Potassium sulphate			0.75					0.75	0.75	0.75			
Dye (aurine)	0.25												
Trinitrotoluene					15.0								

Table 2.01 Composition of some single base propellants

DOUBLE BASE PROPELLANTS (% COMPOSITION)

*added to basic composition

Nitrocellulose	77.45	52.15	56.50	59.65	85.45	59.40	58.00	76.5	89.4	79.25	51.5
Nitroglycerine	19.50	43.00	28.00	36.00	9.00	36.00	40.00	21.5	8.0	15.00	43.00
Diethylphthalate		3.00								3.50	3.25
Dibutylphthalate				0.40	0.40	0.40					
Diphenylphthalate					1.10						
Dinitrotoluene			11.00	0.35	0.65	0.55					
Potassium sulphate		1.25	1.50*						0.8		1.3
Potassium nitrate	0.75										
Ethyl centralite	0.60	0.60	4.50					2.0	1.0		1.00
Graphite	0.30			1.05	0.25	1.00	0.60			0.60	0.20*
Barium nitrate	1.40										
Candelilla wax			0.08*								
Methyl cellulose			0.50*								
Sodium sulphate				0.10	0.10	0.10	0.10			0.10	
Calcium carbonate				0.40	0.40	0.40	0.10			0.10	
Diphenylamine				1.00	1.00	1.00	0.50			0.50	
Water				0.50	0.90	0.55	0.40			0.60	
Methyl centralite									5.0*		
Tin									0.8		

Table 2.02 Composition of some double base propellants

Generally, about 700-1100 cm^3 of gas per gram is produced and flame temperature can range from e.g. 2000K for a cool propellant to 4000K for very hot propellants. Typical gas composition from double base propellants are carbon dioxide 28%, carbon monoxide 23%, hydrogen 8%, nitrogen 15% and water 26%.

Other ingredients that may be found in smokeless powders include:-

camphor, carbazole, cresol, diethyleneglycoldinitrate (DEGDN), dimethylsebacate, dinitrocresol, 2.4 dinitrodiphenylamine, PETN, TNT, RDX, acaroid resin, gum arabic, synthetic resins, aluminium, ammonium chlorate/oxalate/perchlorate, lead carbonate/salicylate/stearate, magnesium oxide, sodium aluminium fluoride, sodium carbonate/bicarbonate, petrolatum, dioctylphthalate, stannic oxide, potassium cyrolate.

The percentage of nitrogylcerine in double base propellants can range from as low as 5% to as high as 44%.

As a general rule only, rifle cartridges use single base propellant whereas pistol/revolver and shotgun cartridges use double base propellants. Rimfire rifle and revolver cartridges use either single or double base propellants.

Apart from firearms ammunition other propellant activated devices have numerous uses, e.g. to drive turbines, to move pistons, to eject pilots from jet planes, to shear bolts and wires, to operate vanes in rockets, to act as sources of heat in special operations, to operate pumps in missiles, to clear blocked drill bits underground, to start aircraft engines, to jettison stores from aircraft, and generally for systems that require well controlled sources of high force applied over relatively short periods of time. Propellants are also used in some blank cartridges.

2.1.5 Projectiles

The firearms act defines a firearm as "...a lethal barrelled weapon of any description, from which any shot, bullet or other missile can be discharged...".

This very loose definition leaves scope to cover almost every conceivable type of device that incorporates a tube through which any missile is projected. Could a blowpipe used to discharge poisoned darts be described as a firearm? It is 'gas' operated, has a barrel, and the projectile is designed to be lethal. Whilst the blowpipe may not be considered to be a lethal barrel, it is, as with a firearm the means of directing and discharging the projectile. It is the projectile that kills. Consequently great attention has been focused on projectile design, and there are many different types of projectiles available on the military and civilian markets.

For the purpose of this discussion, only conventional projectiles will be considered in detail. Conventional projectiles for firearms are bullets, pellets and slugs, each of which may differ from others of the same kind in size, shape, weight, composition and physical properties.

There is a wide range of firearms, and the choice of ammunition available presents a large number of gun/ammunition combinations. The reasons for such a variety of projectiles encompasses internal and external ballistics, nature of target and wound ballistics, all of which are beyond the scope of this discussion.

Bullets

Every bullet type is designed for a specific purpose and the range of bullet designs available for a single firearm can be substantial.

Figure 2.03 illustrates some different physical designs of round nose bullets (63).

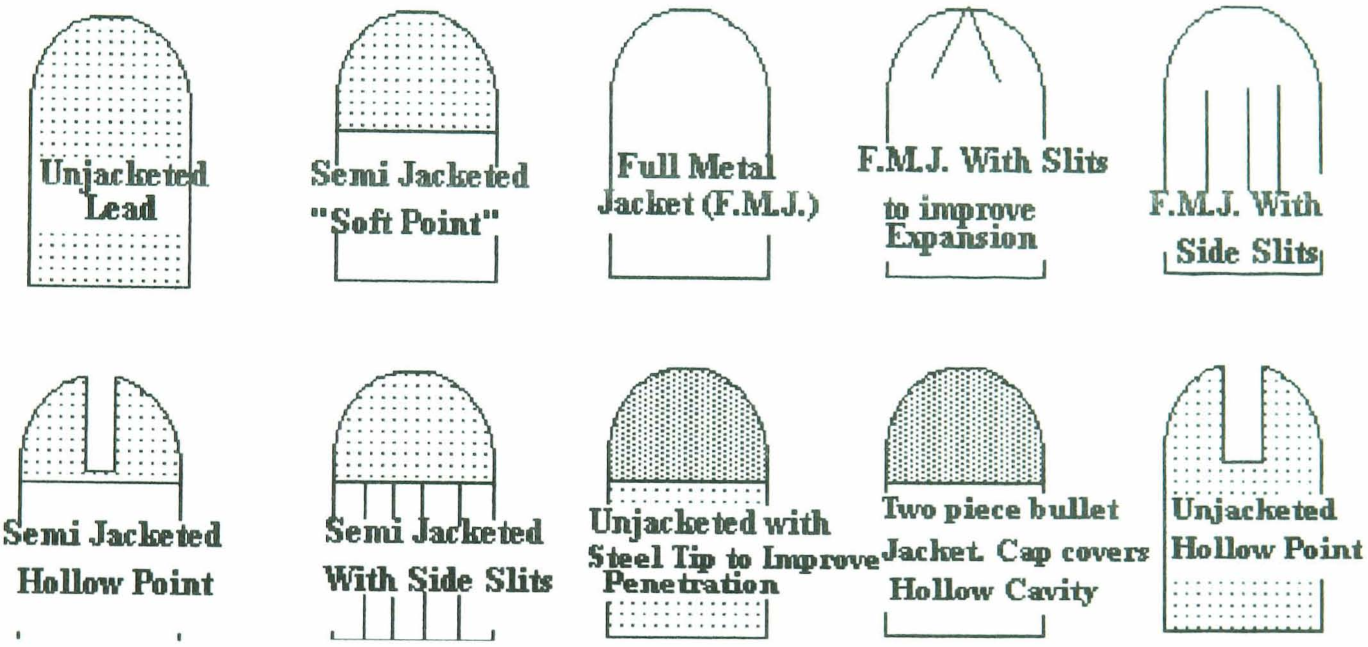


Figure 2.03 Designs of round nosed bullets

This only illustrates a variation of types within one design of bullet which is available in a range of calibres. Variations of types occur within other designs of bullet, e.g. truncated cone, cone or spire-point, spitzer, flat nose, semi-wadcutter, wadcutter and round ball, all of which are available in a range of calibres.

Even the design of the base of the bullet can vary (64). This is illustrated in Figure 2.04.

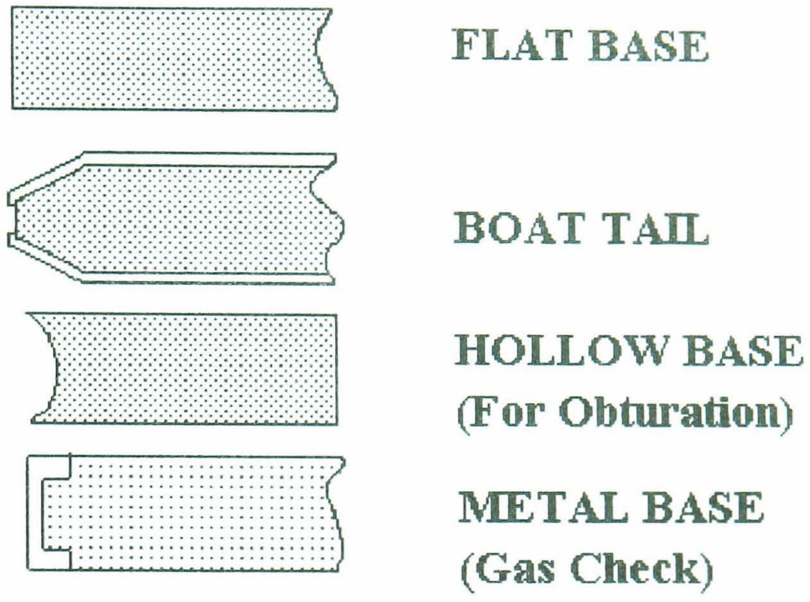


Figure 2.04 Bullet base designs

There is also a wide variety of bullet core/bullet jacket designs without even considering compositional differences. Bullets are either unjacketed, jacketed (envelope) or partially jacketed. Unjacketed bullets are usually confined to revolvers or low power pistols and rifles. Such bullets may have their surface coated with a very thin layer of copper or brass coloured material which is used as a lubricant, and for cosmetic reasons. This is referred to as a 'coat' or 'wash' and is not a bullet jacket in the conventional sense of the word. Unjacketed bullets are frequently lubricated with some form of wax or grease to prevent or reduce fouling in the barrel of the firearm.

Higher velocity bullets have to be either full or partly jacketed because an unjacketed lead bullet fired at high velocity can suffer deformation and have a detrimental amount of lead stripped from its surface by the rifling grooves. Such lead deposited inside the barrel has a pronounced effect on accuracy of subsequent shots. Unjacketed lead bullets are also prone to damage by the feeding mechanisms of self loading firearms.

In the majority of bullets the lead base is exposed to the hot propellant gases. This applies to unjacketed and jacketed bullets. Some bullets incorporate a gas check in the base to prevent erosion by the hot gases. Such erosion can upset the symmetry of the bullet and consequently the accuracy. The base of the bullet may be filled or covered with a substance, e.g. Alox base lubricant, that is unaffected by the temperature and pressure generated during discharge. Another method is to enclose the base with a shallow copper cup. Some bullets have the base enclosed by the jacket. Electroplated jackets usually cover the entire bullet and some soft point bullets with a nose of exposed lead, have a partial jacket which is usually closed at the base. Bullets that are totally enclosed by the jacket, including their base, are referred to as total metal jacketed bullets (TMJ).

Conventional bullets are referred to as ball loads, the word 'ball' originating from the use of round balls as projectiles in the early days of firearms development. Modern bullets are non-spherical projectiles for use in rifled barrels. Conventional bullets are designed either for penetrating power or stopping power (transfer of all energy on impact thereby rapidly stopping the human or animal target) or a combination of both. This is achieved by physical design and the selection of materials with suitable physical properties.

The bullet jacket material is almost always harder than the bullet core material, one exception being armour piercing bullet jackets. Bullet jacketing is done either by electroplating or, much more commonly, the jacket is manufactured separately from the bullet, the bullet then being forced into the jacket in a press. Another method is to pour molten lead into the jacket. The edges of the jacket are usually partly rolled over the base of the bullet or attached by some other physical means.

Whenever a jacketed bullet strikes a target it is possible for the core and jacket to separate, with a consequent reduction in penetration. To prevent such an occurrence a variety of crimps, folds, jacket geometries and melted core techniques are employed. Another method of interest to handloaders is the use of a product called Core-Bond which is a flux that removes surface oxides allowing molten lead to bond directly to the jacket. This allows a degree of alloying between the two metals which is claimed to provide bonding superior to that achieved by physical methods. Soldering of the jacket to the core has also been employed (65).

Bullet jacket materials include gilding metal; cupro-nickel; cupro-nickel coated steel; nickel; zinc, chromium or copper coated steel; lacquered steel; brass; nickel or chromium plated brass; copper; bronze; aluminium/aluminium alloy; nylon (nyclad) and cadmium coated steel (rare). Steel jackets are frequently coated both inside and outside as an anti-corrosion measure. Gilding metal is by far the most common bullet jacket material. Tin is claimed to have lubricating properties and is sometimes incorporated in bullet jacket material. The alloy is known as Lubaloy or Nobaloy and contains 90% Cu, 8% Zn and 2% Sn.

The thickness and hardness of the jacket can vary between the base and the nose of the bullet, the nose portion being thinner for better expansion on impact or thicker for greater penetration of the target. The way in which the jacket is physically attached to the core can vary. This depends on the desired effect of the bullet on the target, either the controlled expansion of the bullet, greater penetration of the bullet or the prevention of core and jacket separation.

Figure 2.05 illustrates some different physical designs (66)

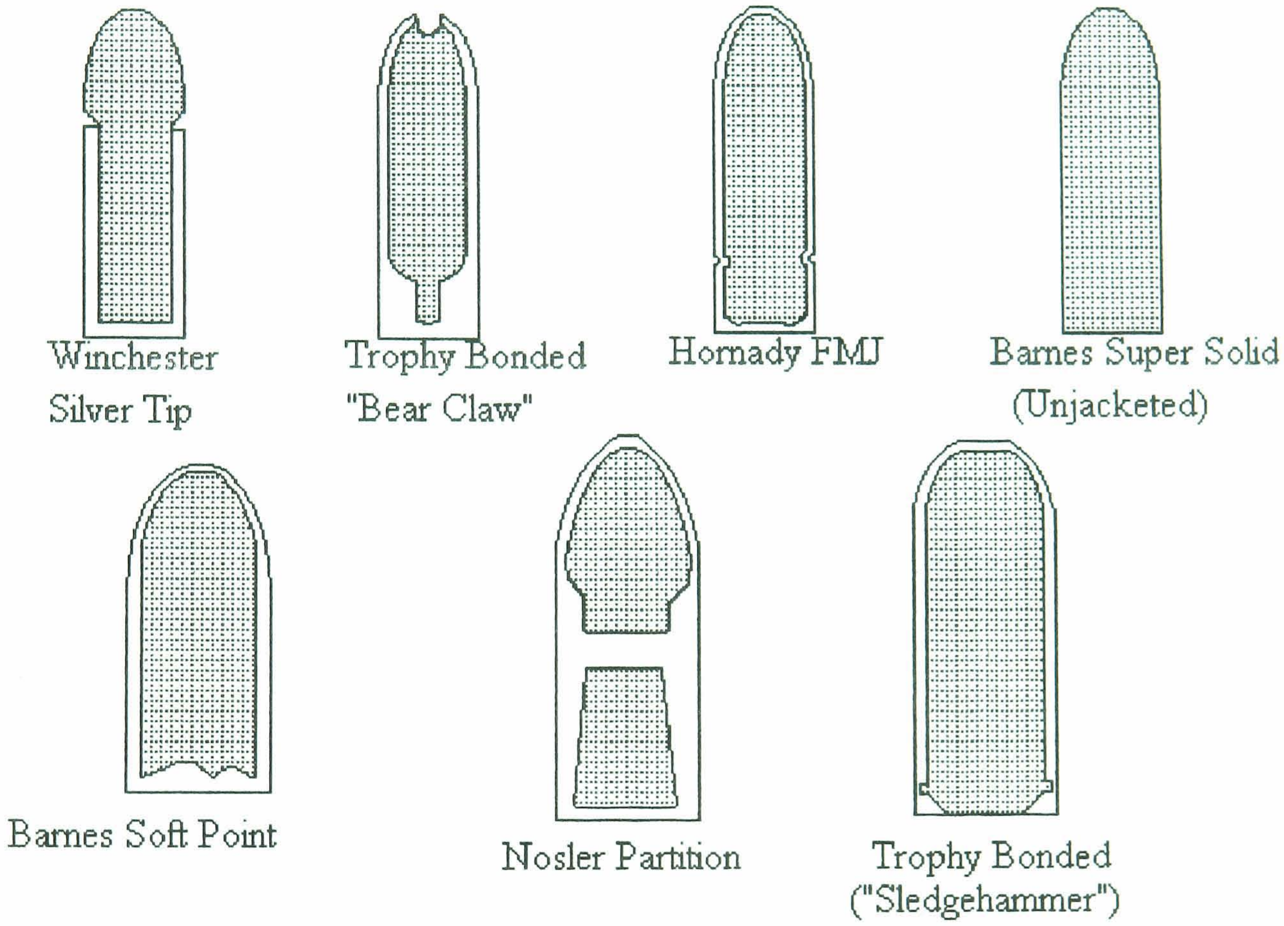


Figure 2.05 Bullet core/jacket designs

The core of the bullet can be made from a variety of materials, lead being by far the most common, but copper, brass, bronze, aluminium, steel (sometimes hardened by heat treatment), depleted uranium, zinc, iron, tungsten, rubber and various plastics, may also be encountered. Bullets with a lead core and a copper alloy jacket are by far the most common.

Sometimes a combination of bullet core materials is used to produce a hardness difference between the base and the nose (dual core bullets) e.g. jacketed bullets with a lead nose and a steel base, a steel nose and a lead base, or a soft lead nose and a hardened lead base.

Bullet lead can be either soft lead or lead hardened by antimony, by tin or by both. Mercury was also used to harden lead in the early days of bullet development. The quantity of alloying materials varies considerably, e.g. antimony <0.5% to as high as 12% but typically 2% to 5%; tin <0.5% to 10% but typically 3% to 5%. A larger amount of tin is required to give the same degree of hardness as that of antimony; consequently, for cost reasons, antimony is more frequently used.

Some jacketed bullets incorporate a small cavity in the nose which is filled with a different material from the bullet core. In some bullets the cavity is unfilled. Bullet tips are usually made from lighter weight material than that used for the core, e.g. plastic, aluminium, fibre, sodium carbonate, polycarbonate, nylon, paper, mild steel. Some soft point and unjacketed bullets employ a metal cap over the nose of the bullet either for increased penetration, to protect the nose from damage, or to improve feeding in self loading firearms. Copper, steel, aluminium and plastic caps are encountered.

Non-ball bullet types are often referred to as 'filled bullets' and are designed for special purposes, e.g. tracer, incendiary and armour piercing roles.

Armour Piercing Bullets

The most effective armour piercing bullets (AP) are usually confined to rifle bullets, velocity and range being important factors in armour piercing requirements. Some revolver and pistol ammunition is described as metal piercing but, although it would be effective against vehicle bodywork and some body armour, it would be ineffective against 'heavy' armour plate. Armour piercing bullets are, with very few exceptions, jacketed.

Armour piercing rifle bullets usually have a bullet tip filler (usually lead) which is designed to 'cushion' the effect of the impact of the armour piercing core; this is very hard and brittle and can break on impact without a 'cushioning' effect. The AP core is also frequently surrounded with a thin sheath of lead between the core and the bullet jacket. The AP core is usually hardened steel such as tungsten/carbon; tungsten/chromium; manganese/molybdenum; chromium/vanadium or chromium/molybdenum.

Two specifications for AP core material are:-

1. steel with 2% to 3% tungsten and 1.25% carbon;
2. steel with 3% to 4% tungsten, 1.1% carbon and a trace of manganese (67).

Tungsten carbide has also been used as an AP core and gives much superior penetration. It is produced by alloying tungsten with carbon, nickel, cobalt or other elements. Tungsten carbide is about twice as dense as steel and about 1.4 times denser than lead. It is non-magnetic and extremely hard. Analysis of such a core yielded the following results:-

Tungsten	93.90%
Carbon	1.65%
Titanium	1.55%
Nickel	1.55%
Iron	0.43%

+ residual trace elements.

A tungsten carbide AP bullet which also incorporates a lachrymal agent and a tungsten carbide AP core/tracer/tear gas bullet have been manufactured. A similar AP/tracer/tear gas bullet was also manufactured with a hardened steel, rather than a tungsten carbide core.

There is a Chinese tungsten carbide AP bullet with a discarding sabot which is very effective due to its very high velocity. The USA also produced an AP bullet with a discarding sabot using depleted uranium as the bullet core material (68).

Revolver and pistol metal piercing bullets are available in a range of calibres and designs. Some examples are:-

1. *KTW bullets:* the original design had a hard steel or tungsten steel core with a copper gas check and the current version is a solid brass or bronze bullet without a gas check. Both have a gliding metal half jacket and the exposed portion of the bullet is coated with green coloured Teflon.

2. *National bullets:* a sharply pointed solid steel bullet the base of which is contained within a brass cup.

3. *ABC (American Ballistic Company) bullets:* a solid steel pointed bullet without a jacket.

4. *Arcane bullets:* a conical shaped flat base bullet made from solid copper alloy (the .380" calibre is round nosed).

5. *THV bullets (Tres Haute Vitesse):* an unusually shaped bullet made of solid brass with 3 times more penetrating power than a conventional bullet due to its hardness, geometric design and high velocity.

Tracer Bullets

When fired a tracer bullet leaves a visible trace behind it so that the trajectory can be seen and the aim corrected if necessary. A tracer bullet has a cavity at the base of the bullet which is filled with a mixture of substances that are ignited by the propellant. The bullets are available in a range of rifle, pistol and revolver calibres, although they are more common in rifle calibres.

The tracer composition is frequently housed in a metal canister placed inside the base cavity. Copper, brass, gilding metal, gilding metal coated steel and copper coated steel canisters are known. Some compositions are placed into the hollow base cavity without the use of a canister. A typical tracer bullet consists of a gilding metal bullet jacket and a lead core with a base cavity containing the tracer composition. Paper discs and lead, steel or brass washers sealed with varnish are sometimes used to "seal' the base of the bullet.

Four examples of tracer compositions are (69):-

Barium Peroxide	86%	(Oxidant and colouring agent)
Magnesium Powder	12%	(Combustible material)
Acaroid Resin	2%	(Binder)

Strontium Nitrate	51.7%	(Oxidant and colouring agent)
Magnesium	33.3%	(Combustible material)
Polyvinylchloride	5.4%	
Phenol Formaldehyde (with yellow dye)	9.6%	

Magnesium Powder	38.0%	(Combustible material)
Beeswax	4.8%	
Strontium Nitrate	42.8%	(Oxidant and colouring agent)
Shellac	4.8%	

Chlorinated Rubber	4.8%
Magnesium Carbonate	4.8%

Magnesium	13%	(Combustible material)
Aluminium	3%	
Strontium Nitrate	73%	(Oxidant and colouring agent)
Iron	6%	
Resinous Material	5%	(Binder)

Dark or dim ignition tracer bullets are designed so as to 'ignite' some distance from the muzzle to avoid dazzling the firer and giving the firing position away. They are also known as delay tracer bullets. These bullets have a duller, slower burning igniter composition in the base above which is the bright burning tracer composition. One tracer igniter mixture consists of potassium permanganate and iron filings (70). Titanium metal is also used in some tracer compositions.

There are night tracer, day tracer and dim ignition tracer bullets, yellow, green, orange, red and white tracer colours are known and they can be manufactured in different degrees of luminosity. Tracer igniter compositions also have varying degrees of luminosity, i.e. bright for daytime use and dim or dark for night time use.

An explosive/tracer rifle bullet is known and consists of a copper alloy bullet jacket and a steel core. There is an explosive charge at the top of the cavity which consists of 40% PETN, 45% lead azide and 15% tetracene. Below the explosive charge is black powder or smokeless powder contained in a small metal cup followed by the tracer composition at the base.

Tracer bullets can also be used to indicate when the ammunition in a magazine or belt is nearly exhausted by putting tracer bullets in a known sequence with ball or other ammunition.

Incendiary Bullets

Incendiary bullets are used against flammable targets and have an incendiary composition in the nose portion which is ignited on impact. Incendiary bullets are usually encountered in rifle calibres.

The best known incendiary agent is magnesium which melts about 650°C and once melted is very easily ignited. Incendiary compositions capable of being easily ignited and which can evolve enough heat to melt the magnesium are used.

Some examples of incendiary compositions are (71):-

'Thermite'	Aluminium Powder	20%
	Iron Oxide (Hammerscale)	40%
	Barium Nitrate	35%
	Boric Acid	5%

This mixture is unlikely to be encountered in small arms ammunition. The following two mixtures are more likely to be encountered:-

Magnesium/Aluminium Alloy	50%
Barium Nitrate	50%

Barium Nitrate	32.0%
Barium Peroxide	53.3%
Magnesium	9.8%
Phenol Formaldehyde	4.9%

Phosphorus is another well known incendiary agent, and phosphorus or phosphorus based compositions have been used in both incendiary and tracer rounds. Because of manufacturing difficulties in working with phosphorus, such compositions have largely been replaced with magnesium type mixtures. However they are encountered in older ammunition and are still manufactured in some non-European countries. Whilst presenting manufacturing difficulties, phosphorus or phosphorus based compositions were effective both in incendiary and tracer roles. One such incendiary composition was a mixture of phosphorus and aluminium.

Other incendiary compositions used in older ammunition were potassium chlorate based with a mixture of potassium chlorate and mercury sulphocyanide as the 'priming' composition. Another older incendiary composition consisted of potassium nitrate, magnesium, aluminium and lead oxide (72,73). Multi-purpose filled bullets are also manufactured, e.g. armour piercing/incendiary, armour piercing/tracer, and spotter tracer bullets which leave a visible trace and produce a puff of smoke on impact. One such smoke charge is lead dioxide 85% and powdered aluminium 15%. There are also tracer/incendiary and armour piercing/incendiary/tracer bullets. An explosive charge or a lachrymal agent may also be incorporated in multi-purpose filled bullets.

Two World War II examples of multi-purpose filled bullets, are shown in Figure 2.06.

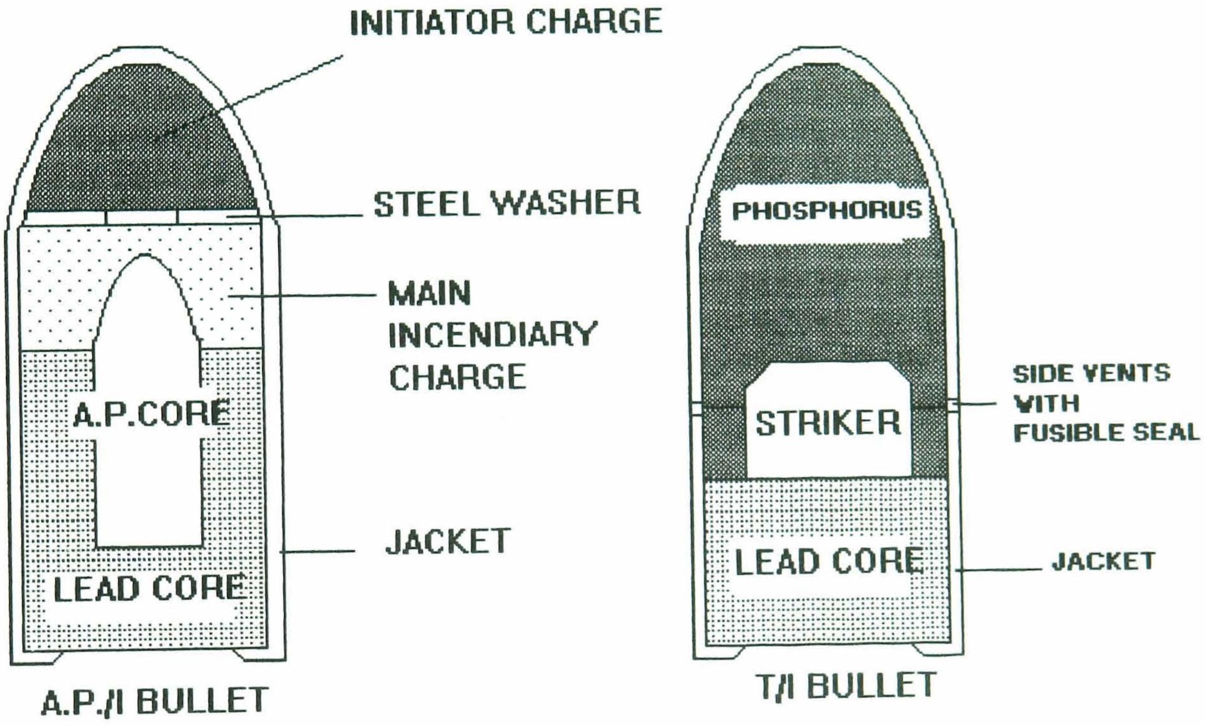


Figure 2.06 Multi-purpose filled bullet

In the armour piercing/incendiary bullet the initiator charge was a mixture of potassium chlorate, magnesium and antimony. The main incendiary composition was a mixture of magnesium, aluminium, barium chlorate, sulphur and nitrocellulose flake powder. The tracer/incendiary bullet had two side vents which were sealed with a fusible metal consisting of tin, bismuth and antimony. On passage down the barrel, the frictional heat melted the fusible seal and ignition of the phosphorus occurred on contact with the air, enabling the phosphorus to act as a tracer as well as an incendiary.

Shotgun Pellets and Slugs

Shotgun pellets (shot) are round metal balls available in various sizes, degrees of hardness and materials depending on their intended use. An unusual cubic shot load is manufactured in 12, 16 and 20 gauge and is claimed to give a faster opening pattern and reduced ricochet hazard than do round pellets.

In shotgun cartridges the pellets are contained within the cartridge case. The pellets in a cartridge are normally the same size and composition, but cartridges are available that are loaded with a range of different pellet sizes (Duplex).

Shotgun pellets can be soft shot, i.e. lead containing less than 0.5% alloying metal or hard shot (chilled shot) i.e. lead containing between 0.5% and 2.0% alloying metal or extra hard shot, i.e. lead containing 3% or more alloying metal or steel shot (iron shot) i.e. soft steel pellets. (Steel shot was introduced for environmental reasons, lead being toxic. However, steel shot has proved to give performance problems, leading to a search for a suitable non-toxic alternative. Bismuth shot is currently being evaluated and looks promising in terms of both performance and toxicity level.)

The pellets are sometimes graphite coated, copper plated or nickel plated. Plated shot is normally confined to hard or extra hard shot. The alloying metal is usually antimony, and lead pellets typically contain between 0% and 6% antimony, although up to 12% antimony has been encountered. Tungsten-polymer shot is also manufactured and, as the name implies, contain tungsten metal embedded in some sort of thermoplastic.

Sometimes a plastic buffer material (usually white granulated polyethylene) is mixed with the shot to help prevent distortion during discharge. The buffer material may be found in the wound channel in close range shootings.

Up to 4 or 5 wads can be present in a shotgun cartridge, although 2 or 3 is more common. The wads are used to provide a gas tight seal as the projectiles pass into the barrel, to separate the propellant from the pellets and to close the mouth of the cartridge case. Wads are usually made from paper or cardboard, plastic (range of colours), felt (which may or may not be waxed or greased), and occasionally cloth type material. Wads may be faced with waxed paper or by black or coloured glazed paper, and the overshot wad may be covered with a paper label containing printed information about the cartridge.

On discharge the wads are propelled from the muzzle, but due to their shape and weight they only travel a short distance unless carried by the wind. However, in close range shootings (usually less than 6') a wad or wads can be encountered in the wound.

In shotgun shootings, the wads can provide useful information. They can be chemically examined to provide some information about the primer and propellant types, and physical examination can sometimes reveal the calibre and give an indication of shot size. Wads from different manufactures can vary in composition, colour, thickness and design, and physical examination may reveal the identity of the cartridge. (Wads are also found in some rifle, revolver and pistol ammunition. There is never more than one wad and such ammunition is rare. Sometimes the wad is made of nitrocellulose but more commonly of glazed or waxed cardboard. Such wads are placed between the propellant and the bullet.)

Sometimes a single rifled slug, a single lead ball or a plated steel ball is loaded in shotgun cartridges. These are designed for heavy game at short range or for special purposes. Rifled slugs transform the shotgun into a makeshift short range rifle capable of considerable penetration and great stopping power at close range. Some slugs are made with hollow points to increase expansion on hitting the target. Special purpose ammunition is available for use in shotguns, e.g. large slugs for use against door locks and hinges for forced entry into premises and vehicles; saboted bullets and slugs; tear gas, smoke screen, flechette and rubber pellet loads for riot control. Special shotgun loads are also used by the military for bomb disposal work.

Several examples of special loads, are as follows. A single cylindrical frangible metal/ceramic slug which appears to be a mixture of iron and dental plaster is used in the "Shok-Lock" round which is designed for gaining entry to dwellings by destroying door locks. Sometimes a wad is attached to the base of a slug to provide a gas tight seal as it travels through the barrel of the shot gun.

Another unusual 12 bore shotgun round is the French "Silver Plus". It is an arrow shaped bullet fired from a shotgun and is claimed to give increased velocity, four times the accuracy and three times the penetration of a conventional lead slug. The bullet is surrounded by a two piece ring sleeve, the two halves of which fall away whenever the projective leaves the muzzle (74).

Handloading

Handloading (making ammunition from new component parts, i.e. cartridge case, primer, propellant and bullet) and reloading (making ammunition by reusing spent cartridge cases with new primers and propellant using either new bullets or home made bullets) are practiced by many firearms enthusiasts, especially in the USA. Reloading can substantially reduce the cost of ammunition, provide ammunition for firearms chambered for unusual or obsolete cartridges, and give scope for 'tailor made' designs. Both handloading and reloading use commercially available primers and propellants which are basically the same as those used in commercially manufactured ammunition.

Reloaders use either commercially available bullets, or make their own from commercially available lead alloy bars or a range of suitable scrap metal alloys. Two specifications for manufactured bullets for reloading purposes are:-

1. 90% lead, 6% antimony and 4% tin;
2. 83.5% lead, 11.5% antimony and 5% tin.

By using varying amounts of tin and/or antimony to harden lead, reloaders can control the hardness of their bullets. They can also cast composite bullets, one mould casting the nose portion and another mould casting the base portion. The two portions are then glued together using an epoxy glue.

Sources of scrap metal suitable for use in bullet making include old lead pipes, old cable sheathing, lead sheeting from old roofs, commercial lead wire, and scrap 'tin' such as pewter, high speed bearings, 50/50 bar solder (lead and tin) and basic white metal (92% tin and 8% antimony). The main sources for reloaders are wheelweight metal (approximately 90% lead, 1% tin and 9% antimony) and printing type metal of which there are five types ranging in composition from 62% to 94% lead, 3% to 15% tin and 3% to 23% antimony. Linotype is the most commonly used type metal (85% lead, 4% tin and 11% antimony).

Provided that the home made bullets meet the criteria for hardness and accuracy the reloader is not overly concerned with the exact composition. An alloy of 90% lead, 5% tin and 5% antimony is generally the favoured mix. Cast bullets may be further hardened by heat treating. For heat treating to be effective, there must be a small amount of arsenic in the alloy.

Other Projectile Types

Less common and unusual projectiles such as exploding bullets; saboted sub-calibre bullets; flare loads; wax, rubber, plastic and wooden bullets; frangible bullets; tear gas bullets and canisters; baton rounds; flechette cartridges; poisoned bullets;

multiple loads; shot loads for pistols and revolvers and other special purpose projectile types, are much less likely to be involved in forensic casework, although cases involving the use of such ammunition are occasionally encountered.

Exploding Bullets

Exploding bullets are not a recent concept. About the middle of the 19th Century two types of exploding rifle bullets were invented, one using a percussion cap and black powder and the other using mercury fulminate. Figure 2.07 illustrates both types.

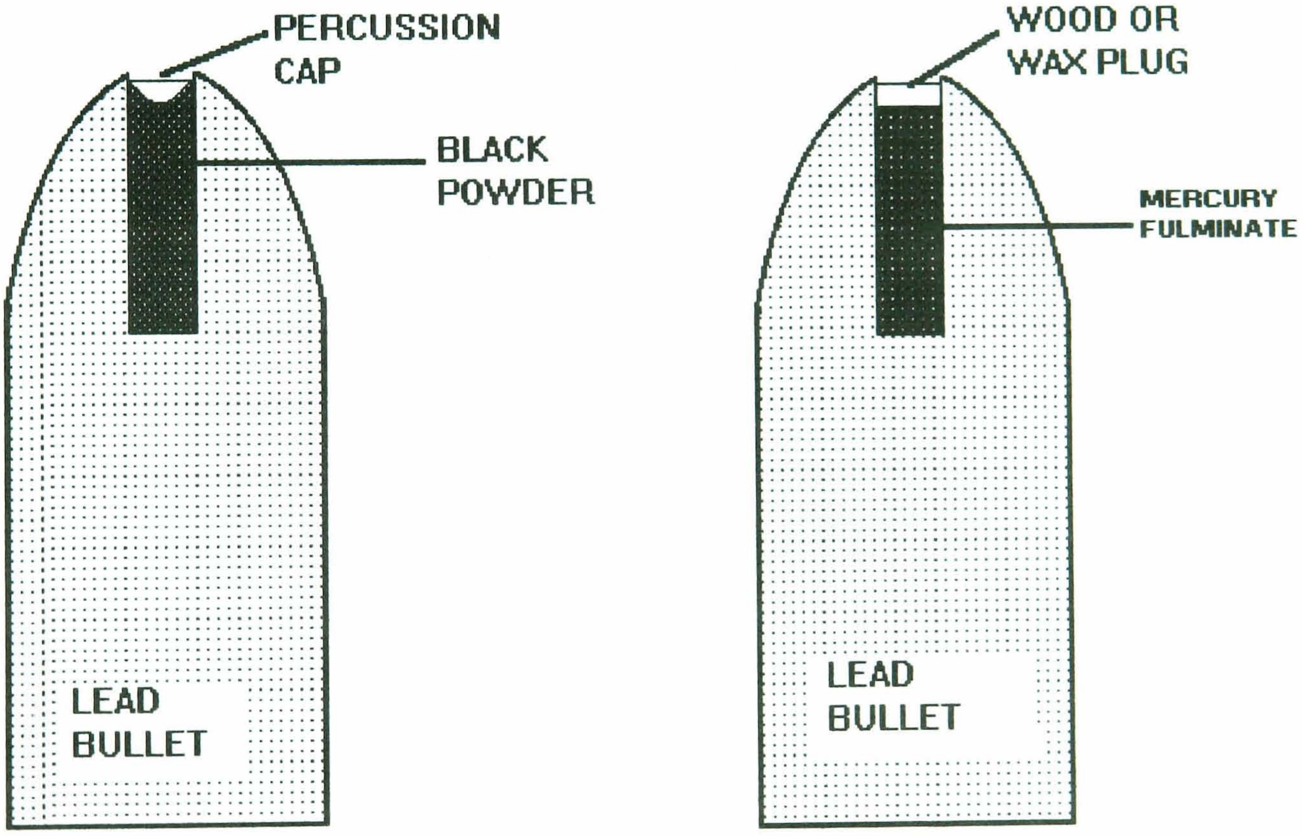


Figure 2.07 Exploding bullets

During World War II several types of exploding rifle bullets were developed containing an integral striking pin. One contained a firing pin inside a brass bushing. The bushing was free to slide backwards and forwards inside a copper cup which had a small hole in the forward end. On firing the pin moves back against the lead core. On impact it moves forward and the point protrudes through the hole in the forward end of the copper cup and detonates the explosive mixture. This is illustrated in Figure 2.08.

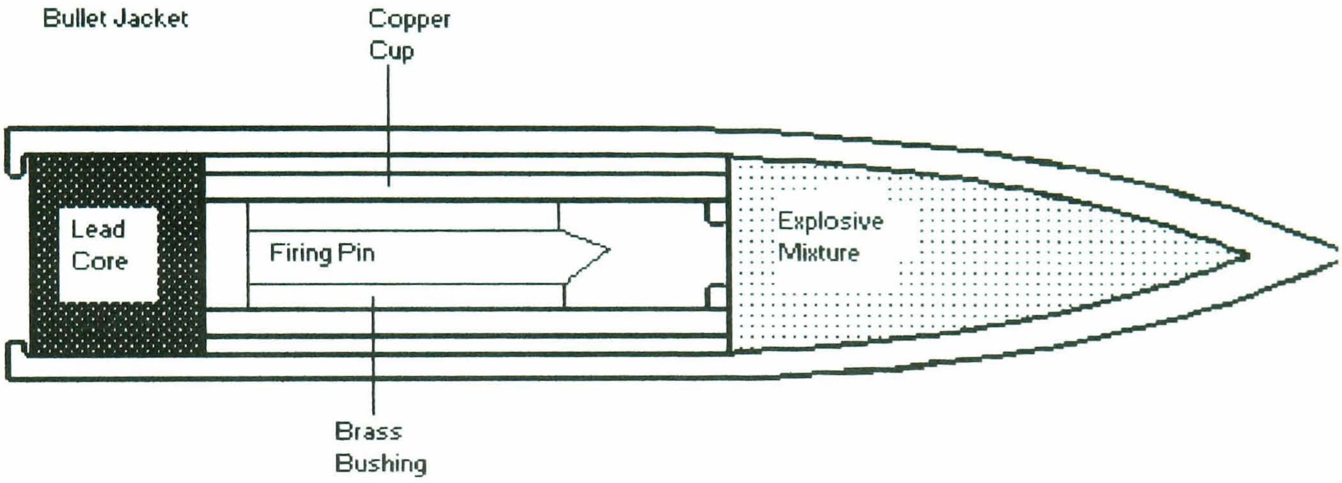


Figure 2.08 Exploding bullet

Another similar design contains a firing pin which on impact detonates 0.42g of mercury fulminate (42%) and potassium chlorate (58%) mixture. An exploding/incendiary bullet with its own firing pin was also developed, the incendiary component being 5 grains of white phosphorus and the explosive component being 7 grains of a mixture of lead styphnate, barium peroxide and calcium silicide. This is illustrated in Figure 2.09.

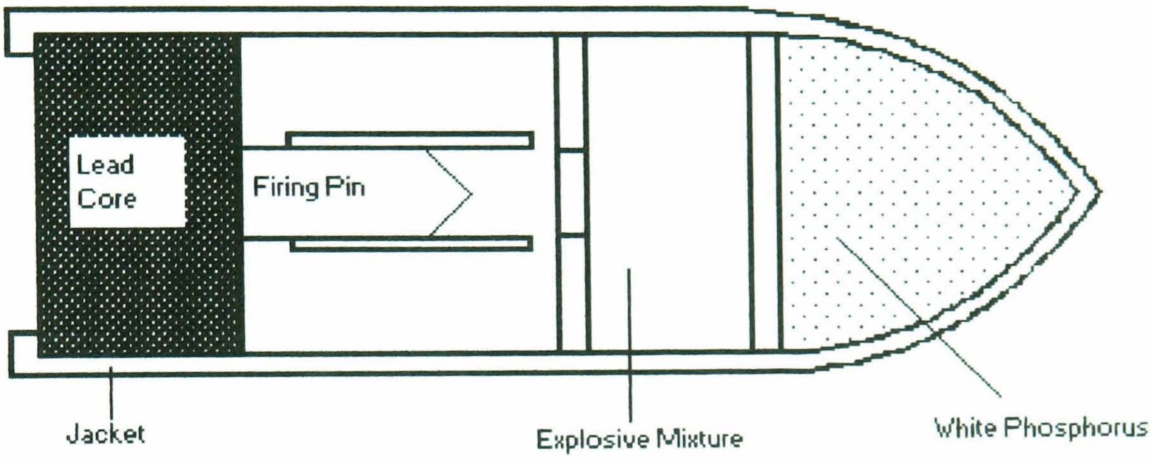


Figure 2.09 Exploding/incendiary bullet

A further design of exploding rifle bullet, without a firing pin, contained a primer with ~40 mg mercury fulminate in the nose of the bullet which on impact ignited compressed black powder and this in turn detonated a metal cup containing an explosive mixture of potassium chlorate (56%) and antimony sulphide (44%). PETN explosive has also been used in exploding rifle bullets.

Modern explosive bullets are available in the USA from Velet Cartridge Company and Bingham Limited (Exploder/Devastator). They are offered in a range of pistol and revolver calibres. The Velet exploding bullet is a normal semi jacketed hollow point bullet with black powder or Pyrodex as the explosive ingredient and with a primer cup sealing the cavity. Depending on the calibre a lead pellet may also be present in the cavity. On impact the primer cup ignites and detonates the explosive mixture. Velet also manufactured mercury filled projectiles. The Exploder bullet is basically the same as the Velet exploding bullet except that the lead shot is omitted (75).

The assassination attempt on President Regan involved the use of Devastator exploding bullets. These consisted of standard 0.22" LR calibre, copper coated lead hollow point bullets, modified by deep drilling to accept a tiny aluminium alloy canister containing about 24mg of lead azide. Nitrocellulose lacquer was used to seal the base of the canister. RDX explosive was originally used in these loads but was replaced by lead azide (76, 77).

Flare Loads

These loads contain some sort of pyrotechnic composition and can be used for signalling purposes, to provide a source of light for brief periods or as fireworks. They are mainly available in .38" Special and 9 mm Parabellum calibres or in 12 and 16 gauge shotgun cartridges (78, 79).

Wax Bullets

Wax bullets are used for mock dueling and fast draw contests, short range practice (training) and in blank ammunition. Rubber and plastic bullets are also used for short range practice. Such ammunition is usually powered by a pistol primer.

Wood Bullets

Wooden bullets are often used in blank ammunition where automatic weapon functioning is required. They can be lethal at short range if some form of bullet breakup device is not fitted to the muzzle of the firearm. Some wood bullets may be sabots for sub-calibre projectiles.

Saboted Sub-Calibre Bullets

A sabot (discarding sabot) is a lightweight plastic container that encloses the lower portion of a bullet. The sabot containing the bullet is seated into the neck of the cartridge case in much the same way as a conventional bullet. The sabot is used to produce very high bullet velocities when a smaller calibre bullet (smaller weight) is fired from a larger calibre barrel. Very high velocity results from firing a lighter bullet in a larger calibre gun, the sabot forming a sort of bushing between the barrel and the bullet and acting as a gas seal and a pusher plug.

The Remington "Accelerator" round is an example of this style of ammunition and is illustrated in Figure 2.10.

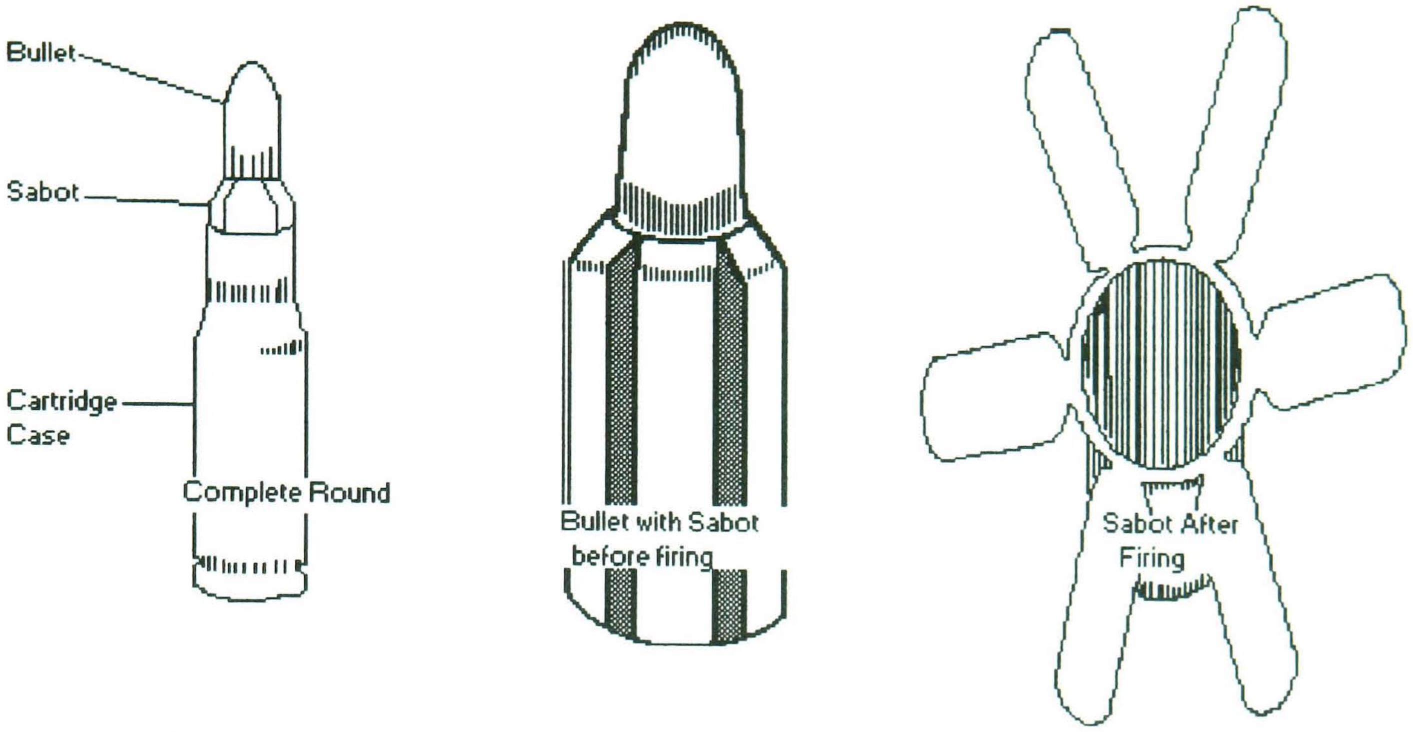


Figure 2.10 Remington "Accelerator" round

As the sabot and bullet move down the barrel together they both start to spin from the twist of the rifling. By the time the bullet and sabot leave the muzzle the spin rate can be in the region of 3,000 revolutions per second. The substantial centrifugal force generated opens the front end of the sabot. The sabot being lightweight and blunt separates from the bullet by air resistance about 18" from the muzzle. Sabots can travel up to 100 yards and due to the speed it is travelling at, it is a dangerous projectile.

The forensic implications of discarding sabots are the fact that the bullet will bear no rifling marks from the barrel of the weapon and one weapon could be used to fire a number of different sub-calibre projectiles (80, 81).

Multiple Loads

Ammunition has been manufactured with more than one bullet loaded inside the cartridge case. The idea is to increase the 'stopping power' of the ammunition. Up to four bullets loaded in tandem within a single cartridge case are known, the bullets being reduced in size and weight compared to a normal bullet loading. Such rounds of ammunition are rare. If a person was shot at close range with one round of such ammunition there would be a single entrance wound and more than one bullet could be recovered from the body (82, 83). A variation on this theme is stacking projectiles one over the other and then enclosing them in a normal gilding metal jacket (84). Of historical interest is the 7.62mm Nagant revolver ammunition. The bullet is contained completely within the cartridge case, the neck of which is coned inwards in front of the bullet (85).

Special Purpose Ammunition Types

The Glaser safety slug is a high velocity projectile with a copper jacket filled with very small lead shot pellets and sealed at the tip with a frangible plug (Teflon). On striking the target the pressure on the tip prevents it from fragmenting, but on penetrating the target the plug fragments causing the bullet to disintegrate, thereby releasing its shot charge. This substantially increases its stopping power by delivering all of its energy to the target, preventing over-penetration and substantially reducing ricochet hazard.

A bullet called Hydra-Shok is designed for increased expansion within the body (mushrooming) and thus causing greater tissue disruption than with conventional ammunition. The bullet is a hollow point with a central post. The central post diverts hydrostatic pressure as the bullet passes through soft tissue thereby assisting the mushrooming of the bullet (86). This is illustrated in Figure 2.11.

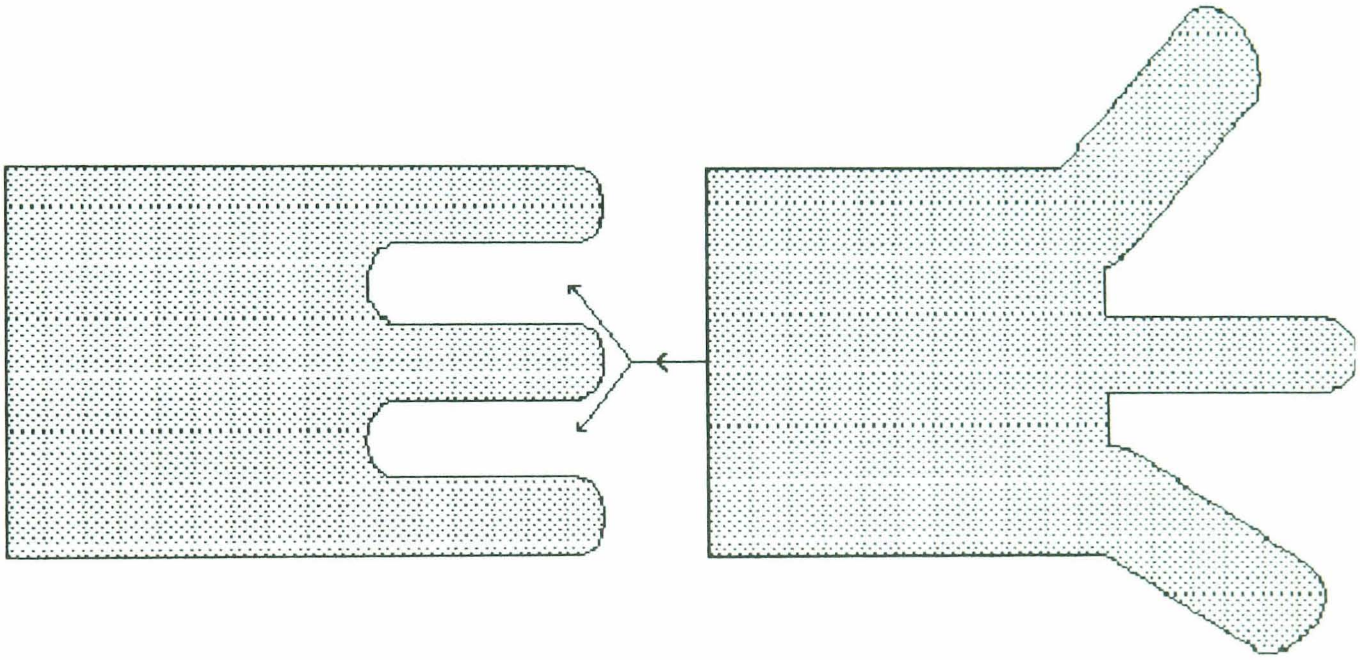


Figure 2.11 Hydra-Shok bullet

Another bullet design to assist bullet expansion is the insertion of a steel or lead ball into the cavity of a hollow point bullet, which on contact with the target is driven back into the cavity, thereby aiding expansion.

Ultra-Shock ammunition is a combination of a central post (a stainless steel screw) with a lead shot pellet over the head of the screw.

An unusual bullet design is the PMC Ultra-Mag ammunition. The bullet is machined from solid bronze and is a hollow tube with a plastic plug at the base to form a gas seal. It is essentially a tube travelling at high velocity and it slices a tubular section completely out of the centre of the wound channel, thereby creating a very destructive, permanent wound (87).

An aluminium bullet with a wounding capacity up to ten times greater than a conventional lead bullet has been developed for dealing with hijackers and hostage takers. It is claimed that it incapacitates quicker than ordinary bullets because of its lighter weight and consequent higher velocity. On hitting the target it reduces speed suddenly because of its light weight and does not pass through the body, thereby transferring all its kinetic energy to the target. The bullet has a self-lubricating nylon coating which is made to bond to the aluminium by first giving the metal some form of chemical coating (88).

Another round of ammunition developed specifically for law enforcement use is the BAT (Blitz-Action-Trauma) ammunition. It is designed for high 'stopping power' and for the projectile to have a short range of travel. The bullet is unjacketed and is made from solid copper alloy with a differing diameter hole all the way through the bullet from nose to base. This is illustrated in Figure 2.12.

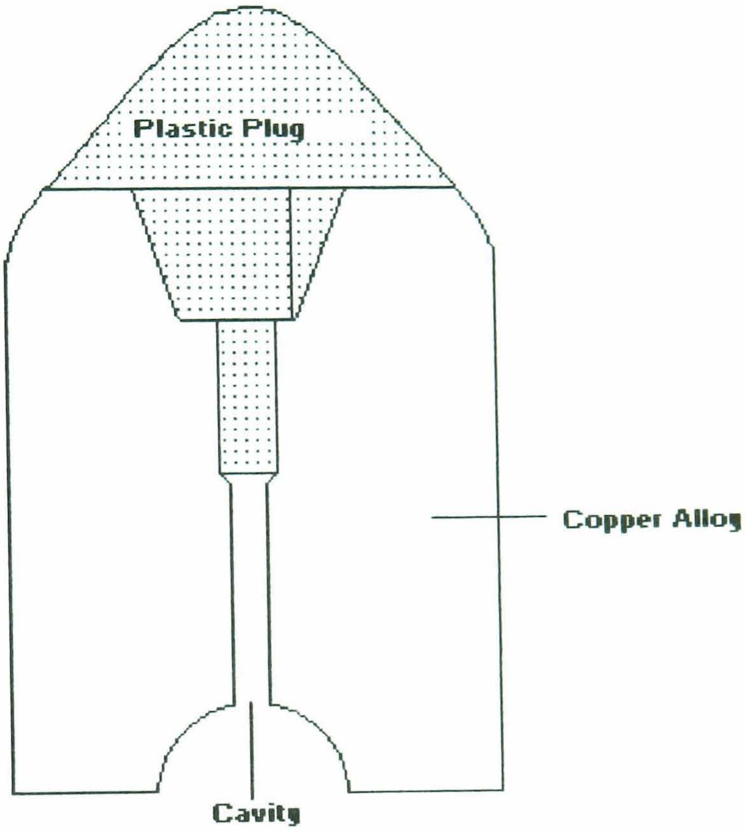


Figure 2.12 B.A.T. bullet

A plastic plug fills the larger hole and part of the smaller one. On firing the cap separates from the bullet due to gas pressure acting through the hole in the base. Because of the lighter weight of the bullet compared to a conventional bullet it loses velocity, and hence energy, much faster, and consequently is less of a risk to innocent persons should the bullet miss its target. Due to the larger nose cavity the bullet deforms rapidly in the target, substantially diminishing the risk of over penetration or ricochet, both of which could injure innocent bystanders (89).

A bullet specifically designed to shatter car windscreens and to retain sufficient energy to wound the occupant is manufactured under the name of Equaloy. It is designed not to exit from soft targets. The bullet had a semi-wadcutter profile, an aluminium alloy core, and is coated with white nylon (90).

MagSafe bullets are bullets with a gilding metal jacket filled with an epoxy resin which has No. 6 size hardened lead shot pellets embedded in it. Because of its substantially reduced weight, very high velocities are achieved. On impact the epoxy resin breaks up and releases the shot pellets, thereby transferring all the kinetic energy to the target (91). It also substantially reduces over penetration and ricochet hazards.

Splat multi-purpose ammunition (synthetic plastic loaded ammunition for training) uses a metal filled plastic material which is injection moulded into the shape of a bullet. The bullets are much lighter than conventional equivalents and consequently achieve much higher velocities. They are frangible on impact and virtually eliminate over penetration and ricochet hazards. Splat ammunition is available in a range of calibres and the mass, velocity and frangibility of the bullets can be varied to suit the intended purpose. The .38" Special aircraft load is designed to safeguard against perforation of aircraft windows and body panels whilst engaging hijackers; and other rounds are designed to penetrate car bodywork and injure the occupants. A 12 gauge shotgun slug is designed for attacking door locks and hinges to gain entry in hostage situations, and on doing so it completely disintegrates. Splat bullets produce wound cavities up to ten times larger than conventional lead bullets, do not over penetrate, and all the energy is transferred to the target (92).

Poisoned Bullets

The concept of a poison contained within a bullet is not new and has been experimented with over a long period of time.

As all poisons, in the quantities capable of being administered via a bullet, take some time to kill, the tactical use of such ammunition is of limited military or civilian use. However, it is an attractive concept for an assassin. If the bullet itself fails to kill, the backup system of the poison offers a twofold method of attack.

In 1892, Lagarde transmitted anthrax to animals by shooting them through their soft tissue with infected bullets.

A poisoned bullet containing about 38 mg of aconitin (a vegetable alkaloid: 4 mg is a lethal dose) was manufactured in the Soviet Union. Aconitin is prepared from the roots of a flowering plant known as Aconitum Napellus, three species of which grow wild in the USA and at least one in the Soviet Union. For centuries aconitin has been used to tip poisoned arrows. Figure 2.13 illustrates the Soviet design.

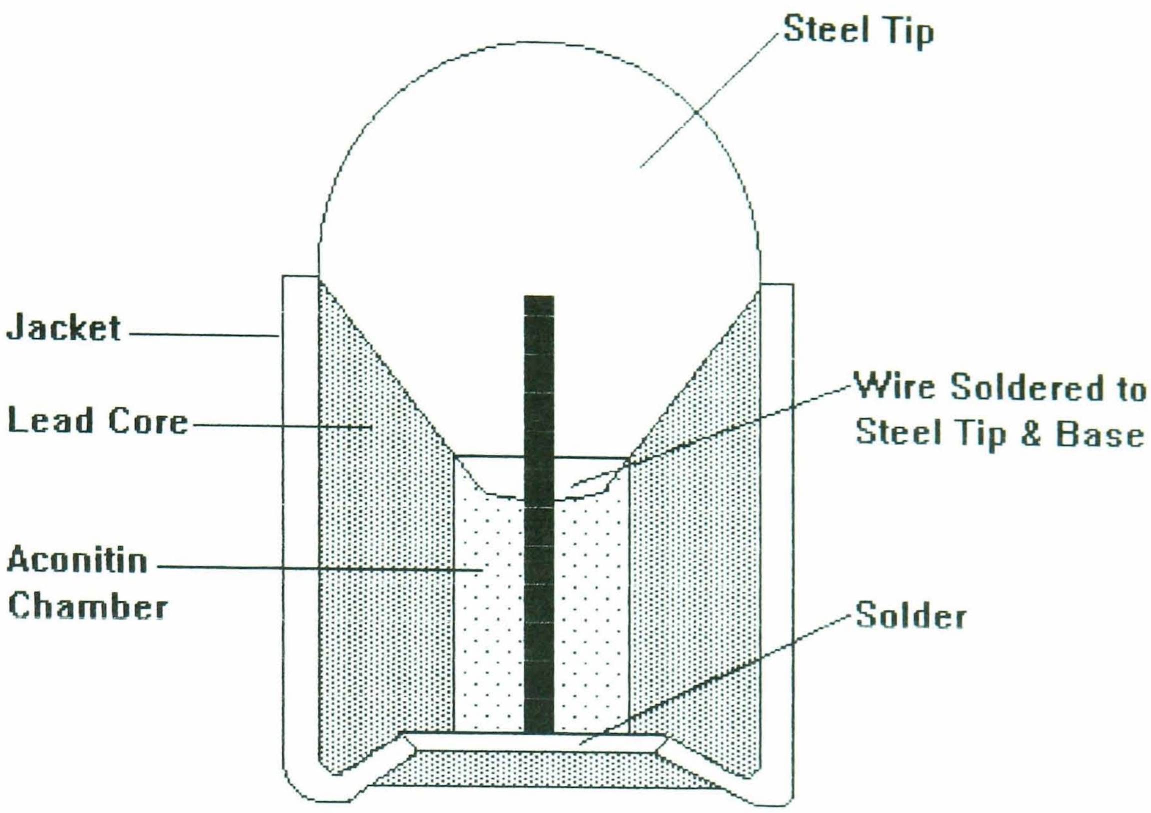


Figure 2.13 Soviet designed poisoned bullet

On impact the steel wedge is driven backwards, expanding the slotted jacket and lead core. The bullet fragments within the target releasing the poison.

A German modification of the Soviet design replaced the aconitin with a glass ampoule containing an aqueous solution of hydrogen cyanide, which is faster acting than aconitin. On impact the steel striker is driven into the bullet, fragmenting the bullet and releasing the poison.

Figure 2.14 illustrates the German design.

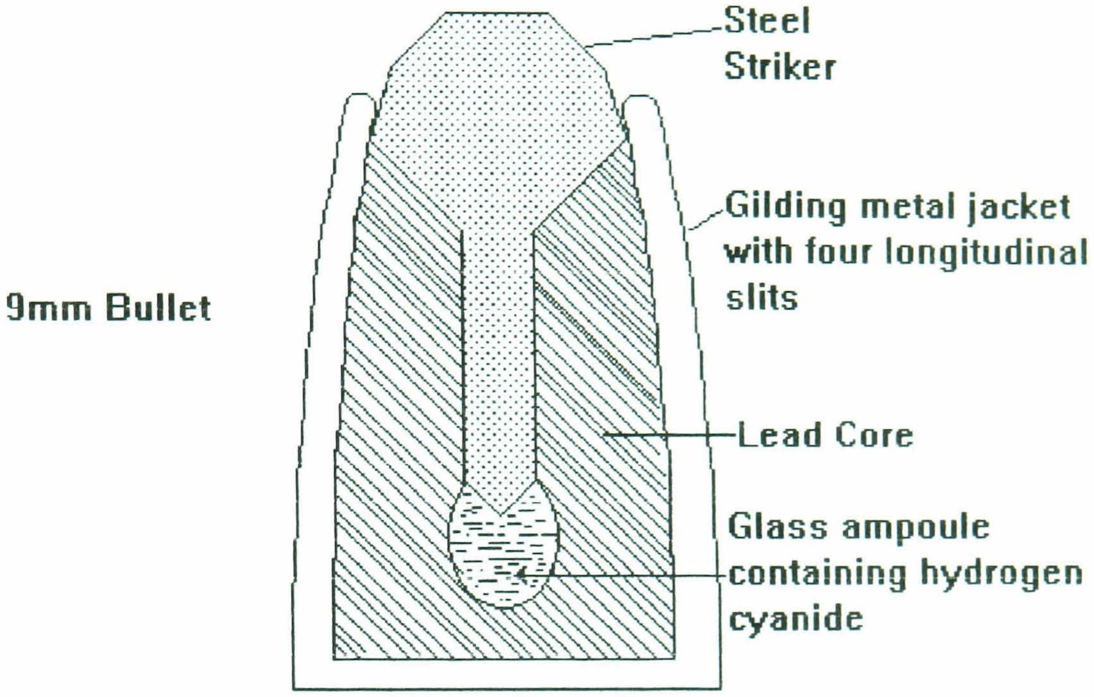


Figure 2.14 German designed poisoned bullet

Another German design was a jacketed bullet with a hollow in the nose. On impact the steel plug, squashed the glass ampoule against the lead core, causing the fluid to pour out of the nose cavity. This is illustrated in Figure 2.15.

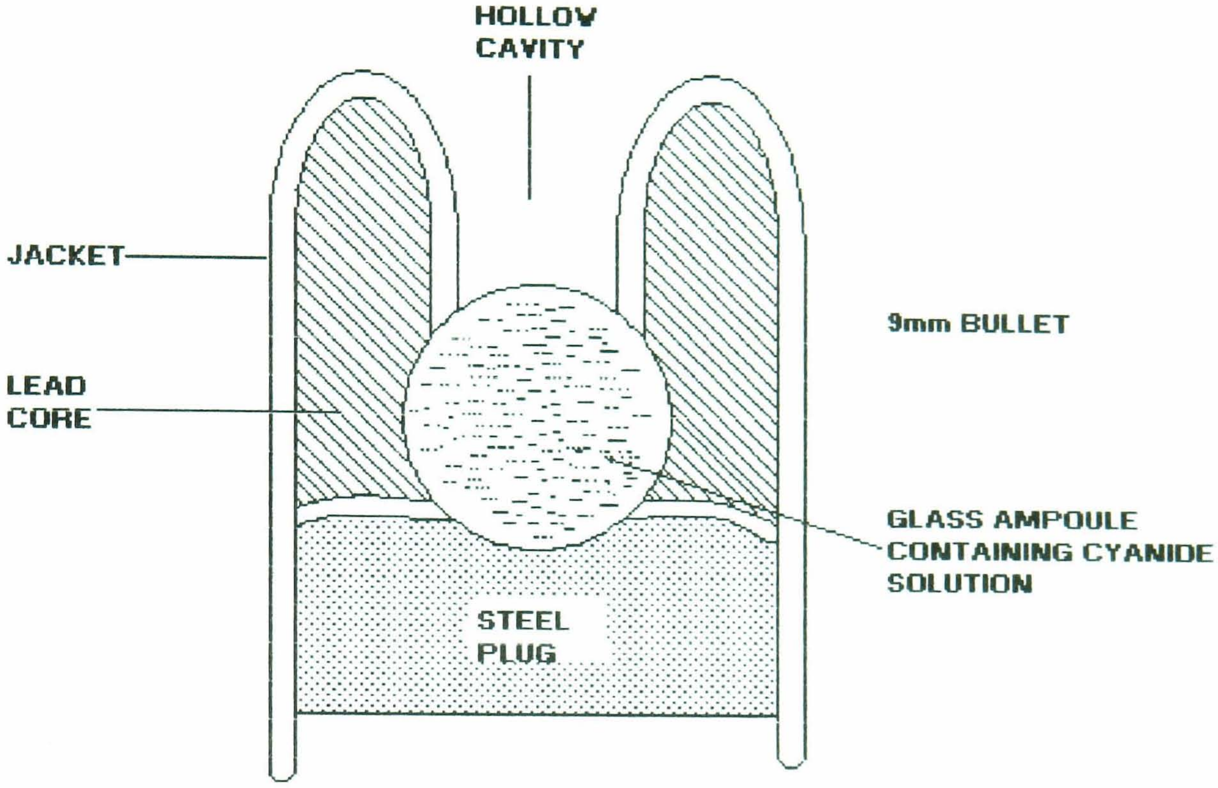


Figure 2.15 German designed poisoned bullet

In August 1978, a Bulgarian defector living in Paris was shot with a tiny pellet made of 90% platinum-irridium alloy which was only 1.7 mm in diameter. Two holes drilled in the sphere contained approximately 0.4 mg of ricin (a toxic protein derived from castor beans). This assassination attempt was unsuccessful. However, the same device was used successfully three weeks later to kill Georgi Markov in London. In this case the victim was stabbed in the thigh by an "umbrella" and injected with the pellet.

In 1975 a pistol was developed which was capable of accurately discharging a poisoned flechette over a range of 250 feet. Other dart launchers disguised as walking canes and umbrellas are also known. The flechette was coated with the shellfish poison saxitoxin. The launchers make very little noise and the toxin leaves no trace and has no known antidote. Another variation on this theme is a false 7.62 mm calibre rifle cartridge with a 'separable' bullet, the cartridge case being filled with anthrax spores or botulinal toxin and designed for attacks on people within confined spaces, by breaking on the walls or floor and releasing the 'poison' (93, 94).

A 7.9 mm calibre false cartridge containing poison gas is also known and is designed for the same purpose. A .320" calibre poison gas cartridge designed to be fired from a pistol has also been manufactured.

Shot Loads

Pistol and revolver cartridges can be loaded with lead shot pellets, primarily for short range vermin control. The shot can be totally enclosed within the cartridge case and sealed with a wad, or the shot may be contained in a paper, cardboard, plastic or wood sabot. A shot load/bullet combination is also manufactured in handgun calibres (95).

Baton Rounds

As the name implies, such rounds of ammunition incorporate projectiles that are designed to incapacitate rather than kill, and are used for riot control purposes. Either plastic or rubber bullets are used, and are usually very much larger than conventional bullets. Such bullets can be fatal if the weapon is not used in the correct manner. Much smaller plastic bullets, 5.56 mm calibre, have been used for riot control by the Israeli Army and fatalities have resulted from their use.

Frangible Bullets

These are usually confined to .22" calibre and are used in shooting galleries. They are designed to disintegrate on hitting the target and consequently will not ricochet. They consist of particles of iron or lead bonded in some synthetic material. One manufacturer describes a .22" short bullet as being lead or iron bonded with lanolin (96). Such bullets are also used for training purposes.

Frangible bullets have recently become popular in a range of calibres due to the trend towards lead free ammunition for indoor training purposes, the conventional lead bullet being replaced by a frangible non-toxic alternative coupled with the use of lead free primers. Details of some frangible ammunition is presented in Chapter 5.

Flechette Cartridge

These cartridges consist of small steel darts loaded into shotshells and rifle cartridges used by the military. They contain either single or multiple loads. A flechette with a sabot (Steyr Acr) has been developed and is claimed to have good penetrative ability and formidable wounding characteristics.

'Dardick Trounds'

These cartridges used a triangular plastic case with a primer in one end and the projectile in the other. They were developed for various civil and military applications but are no longer manufactured.

Tear Gas Bullets

Tear gas bullets are known to have been manufactured in a variety of calibres over the years. Israel currently makes a 7.62 mm NATO calibre tear gas bullet (97). Tear gas guns which fire large canisters of tear gas are used for riot control and hostage situations. Smoke producing canisters are also used for similar purposes.

2.1.6 Complementary components

Also present in ammunition is a range of lubricants (greases and waxes) usually confined to unjacketed bullets, and various sealers, varnishes and lacquers (often coloured) that are used for several purposes. These include prevention of the ingress of moisture or oil, anti-corrosion measures, to act as a visual aid in the manufacturing process, to provide additional resistance of the bullet to the pressure generated by the propellant gases, and colour coding to identify the type of ammunition. The bullet tip may also be coloured coded to identify the bullet type. A variety of sealing discs may also be present in different types of primers, some of which contain tin.

Bullet lubricants frequently contain mixtures of some of the following:-

beeswax, petroleum jelly, sheep tallow, Carnauba wax, molybdenum disulphide, lithium base grease, ceresin wax, powdered graphite, paraffin wax, Alox compound and Zokorite. The mixture may be dissolved or suspended in a fast drying solvent or applied directly whilst hot.

Coloured laquers and vanishes may incorporate metallic containing pigments.

2.1.7 Caseless ammunition

Current trends in firearms and ammunition are towards lighter and easier to carry firearms and ammunition. This includes the use of various plastics in the manufacture of firearms and some large, well known manufacturers are experimenting with the development of caseless ammunition (combustible cartridge cases and primers) which, if viable, will involve major design changes in the mechanism of firearms.

The disadvantages of caseless ammunition include the very high cost of totally new weapons and ammunition manufacturing systems, relative fragility and sensitivity of the ammunition, and difficulties with weapon maintenance and repair in the field.

Advantages of caseless ammunition include weight saving, a cost saving over brass cased ammunition, a material saving (copper is a critical material during wartime) and a higher cyclic rate of fire made possible because the extraction and ejection cycle of conventional firearms is no longer necessary (about 2,200 rounds per minute is possible).

The concept of caseless ammunition is not a recent one as such ammunition, for use in breechloading firearms, has been in circulation for well over one hundred years. The concept has been experimented with by different countries at various times, but with only limited success. Typical compositions consisted of nitrocellulose (12.6%N) 65%, kraft paper 15%, resin 20% and diphenylamine (added) 1%.
The German firm of Heckler & Koch has relatively recently solved many of the problems associated with caseless ammunition and produced a rifle, the HKGII to fire 4.73 x 33 mm calibre caseless ammunition.

Caseless ammunition has no cartridge case to serve as a heat-sink and barrier between the propellant and the hot chamber walls. The problem of "cook-off" i.e. the heat rather than the firing pin causing the cartridge to discharge, has always been a major disadvantage with caseless ammunition. Heckler & Koch have solved this problem by the use of a propellant that contains no nitrocellulose but is based on materials more commonly associated with explosives than with propellants. The composition is a commercial secret but the new propellant has an ignition temperature about 100 K greater than nitrocellulose based propellants (98,99).

Figure 2.16 illustrates cross-sectional view of the 4.73 x 33 mm H&K caseless ammunition.

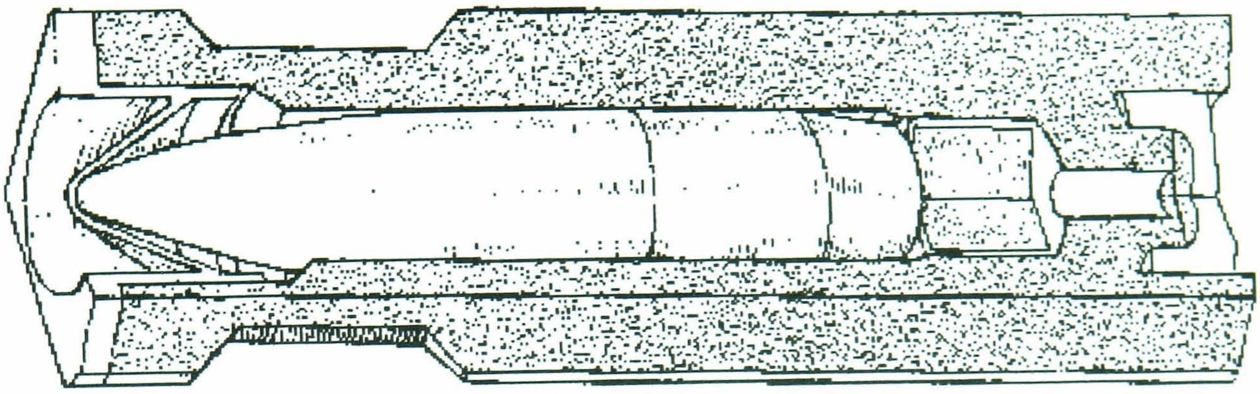


Figure 2.16 Heckler & Koch caseless ammunition

It is rumoured that Heckler & Koch have since abandoned the project.

2.2 FIREARMS

2.2.1 Construction materials

A range of different grades of steel are used in the manufacture of a single firearm. Chromium-molybdenum steel is the basic material for the modern firearms industry. It possesses good tensile strength, resists wear and has good machining properties. Most .22" rimfire calibre guns, shotguns and low pressure centrefire barrels are made from carbon steels. High pressure centrefire barrels for sporting use are normally made from chromium-molybdenum - vanadium steel.

The grades of steel used for the many different component parts involved in a single firearm can vary from manufacturer to manufacturer, but they all contain some of the following elements in order to achieve the desired metallic properties:- chromium, copper, manganese, molybdenum, nickel, phosphorus, silicon, tungsten and vandium. Aluminium alloys are also used in the manufacture of firearms (and telescopic sights) and contain some of the following elements:- chromium, copper, iron, magnesium, manganese, nickel, silicon and zinc (100). Springs used in firearms may contain beryllium and copper and some parts such as sights may be attached by brazing or silver soldering. Stainless steel is being increasingly used for the manufacture of firearms. Stainless steels range from 12% to 24% chromium with other additives such as molybdenum and nickel.

The trend is towards lightweight handguns and rifles and increasing use is being made of polymers (plastics) for the manufacture of parts such as stocks, grips, frames, magazines etc. Polymers have considerable advantages. They are lightweight, durable, inexpensive, non-corrosive and easily moulded to any required shape, thus eliminating the need for expensive tooling to machine a firearm to the desired shape.

A wide range of woods are used in the manufacture of solid wooden stocks for rifles and shotguns, one of the most popular being walnut. Laminated wooden stocks are also used, and consist of thin strips of wood that are impregnated with epoxy and compressed into a solid block.

Synthetic stocks are gradually replacing the traditional wooden stock due to their cheapness, ease of manufacture, lighter weight and wearability, and are claimed to be more stable and easier to maintain than wooden stocks. Materials such as nylon, polyurethane, fibreglass, Kevlar reinforced with fibreglass or carbon fibre, or thermoplastic resin reinforced with glass and ceramics may be used. The stocks may be hollow, or have a hollow filled with foam or solid. Such stocks may be surface treated with a coating such as polyurethane.

Grips may be made of wood, plastic, laminated phenolic resin or rubber, and rubber is also used for recoil pads. Customised or ornamental firearms may have grips made from ivory, mother of pearl or buffalo horns, and the firearm may have elaborate engraving some of which is inlaid with gold or silver. Such firearms are unlikely to be used in crime.

Homemade firearms are encountered in crime, and materials used in their construction varies markedly. Some of them are finished with paint, and flakes of paint transferred to the criminals clothing can be of considerable evidential value. The grips of some homemade firearms are metal and are sometimes covered in plastic adhesive tape. Plastic adhesive tape may also be used to tape magazines together for use with commercially manufactured firearms, so that the firearm can be reloaded rapidly. Fingerprints may be obtained from the tape and a comparison of tape ends with the ends of tape recovered from a suspect's home or workplace can yield very strong evidence if a physical match is obtained. The same applies to toolmark impressions from machine tools at a suspect's home or workplace.

2.2.2 Surface coatings

The reasons for treating the surface of a firearm is to get rid of reflections from bright surfaces which could dazzle the firer and/or reveal the firing position to the enemy or game, to improve the appearance of the firearm and more importantly to provide a degree of protection against corrosion.

Early firearms had no finish at all and rusted rapidly when exposed to blackpowder residues and atmospheric moisture. A process known as 'Browning' (artificial rusting) was the first attempt at a rust resistant finish and records on the process exist as early as 1637. The problem with Browning was stopping the rusting action. The early Browning finishes were known as 'Russetting'.

'Blueing' started to replace Browning by the 1800's although it originated much earlier as records exist dated 1719.

Both Browning and Blueing are essentially controlled artificial rusting processes using special oxidizing mixtures. The process consists of a number of stages:-

1. Degreasing (by using any suitable solvent).

2. Application of Browning of Blueing solution.

3. Rusting (at room temperature or in a steam oven).

4. Drying.

5. Scratching (removing loose rust with a wire brush or fine steel wool).

6. Repeating stages 2 to 5 from two to six times (usually three is sufficient) until desired colour is obtained.

7. Fixing (oiling, lacquering or waxing). Depending on the nature of the chemicals involved a further step may be necessary to neutralize or remove traces of the chemicals used before fixing.

There are hundreds of formulae for Browning/Blueing solutions, to the extent that it is not possible to give a typical example. However the majority of chemicals involved are given in Table 2.03 (101, 102).

Organic	***Inorganic***
Acetic acid	Ammonia; Ammonium carbonate, chloride, persulphate, sulphide
Acetone	***Antimony*** trichloride
Benzoin	***Arsenic***
Butyl alcohol	***Bismuth*** chloride, nitrate, oxychloride
Carbon	***Boron*** i.e. boric acid.
Chloroform	***Chromium*** i.e. chromic acid, oxide
Diethyl ether	***Copper*** chloride, oxalate, sulphate

Ethyl alcohol
Ethyl nitrate
Formic acid
Gallic acid
Oxalic acid
Picric acid
Tannic acid
Tartaric acid
Vaseline)
Amber Varnish)
)
Linseed oil) Fixing stage
Shellac)
)
Copal)
Mineral oil)

Iodine

Iron; Ferric acetate, bisulphate, chloride, permanganate, sulphate

Lead acetate, oxide

Manganese oxide, dioxide, peroxide, nitrate

Mercury i.e. Mercuric nitrate, chloride. Mercurous nitrate

Mineral acids i.e. Hydrochloric, Nitric, Sulphuric

Potassium bisulphate, chlorate, cyanide, dichromate, ferricyanide ferritartrate, iodide, nitrate, oxalate, permanganate

Selenium i.e. Selenious acid

Silver nitrate

Sodium chloride, dichromate, hydroxide, hyposulphite, nitrate

Sulphur

Tin i.e. Stannic chloride, oxalate. Stannous chloride

Zinc; chloride, nitrate, sulphate

Quartz sand, Glasspowder, Water

Table 2.03 Some Browning and Blueing ingredients

The process of Browning and Blueing can be very time consuming and labour intensive. Today, nearly all blueing (blackening) is done by the hot salt, black oxide process because of its speed and cheapness of materials. Hot salt blackening can vary in colour from a blue black to a deep black depending on the concentration of the chemical solutions, the temperature and the alloy content of the steel. The vast majority of firearms currently manufactured are finished by the use of a blackening solution containing sodium hydroxide, potassium nitrate and sodium nitrite in the typical ratio 65:25:10 respectively.

One of the best finishes for firearm steel is 'Phosphatizing' (Parkerizing) but few manufacturers offer this finish other than if required for military or police markets. The process deposits a crystalline layer of phosphates on the metal surface by immersion in a bath of iron, zinc or manganese dioxide and phosphoric acid. Of these, a manganese phosphate finish is preferred for military use.

After phosphatizing some firearms are then oil coated to provide extra protection against corrosion. Other protective coatings applied over the phosphate coating include electrostatic spray painting, epoxy, zinc chromate and Teflon. When the coating includes molybdenum disulphide or fluorocarbons such as Teflon, there is the added advantage of reduced friction between moving parts.

Some firearms are plated with anodized aluminium, nickel or chromium which gives durability and good looks, and some are made from stainless steel which is much less prone to rust than conventional steel. Electroless nickel coating is an alloy coating of 88% to 96% nickel and 4% to 12% phosphorus, which is produced by chemical (not electrical) reduction of nickel on to the metal surface.

Good care and maintenance of a firearm are the best protection against corrosion. There is a wide range of commercial gun cleaning and maintenance products available.

Bore cleaning products include an electrochemical cleaning device and numerous chemical cleaning mixtures, containing both organic and inorganic compounds, the compositions of which are commercial secrets.

Lubricating products range from light mineral oil to dry lubricants incorporating molybdenum disulphide, fluorocarbons (PTFE) and other synthetic lubricants

3 FIREARMS DISCHARGE RESIDUES

3.1 INTRODUCTION

Forensic firearms casework examination encompasses the following major areas of work.

Physical	***Chemical***
Examination of firearms, ammunition and associated items.	Examination of swabs and clothing from suspects for firearms discharge residues.
Examination of firearms and associated items for anything else of forensic interest e.g. blood, hairs, fibres, glass, fingerprints.	Examination of clothing and miscellaneous items i.e. identification of bullet holes, differentiating between entry and exit holes, angle of fire, range of fire.
Comparison macroscopy of spent bullets and spent cartridge cases.	Identification of bullet strike marks, firing points, weapon hides etc.,
Identification of weapon types from spent cartridge cases and spent bullets	Chemical comparison of bullet fragments and propellants.

← Serial number restoration →

← Examination of scenes of crime →

← Presentation of evidence in Courts of Law →

← Training of Police and Scenes of Crime Officers →

In addition, various research and development projects are undertaken and in Northern Ireland there is also an intelligence gathering aspect to the work (103, 104).

Firearms discharge residue work is an important aspect of the overall workload.

3.1.1 Firearms discharge residues

The gases, vapours and particulate matter formed by the discharge of ammunition in a firearm are collectively known as firearms discharge residue (FDR) or gunshot residue (GSR). Anything present in the ammunition may contribute along with a possible contribution from the firearm itself. The residue consists of both inorganic and organic constituents.

Inorganic constituents could originate from the primer mixture/cup/sealing disc/anvil, the cartridge case, inorganic additives to the propellant, the bullet core and jacket, metallic containing pigments in coloured lacquers/sealers/lubricants, inorganic trace impurities in any component, and also from the chamber/barrel interior/muzzle of the firearm in addition to inorganic debris already present inside the firearm before discharge. The main sources of inorganic discharge residue are the bullet and the primer.

Organic constituents could originate from the primer mixture, the propellant, sealers/lacquers/lubricants from the ammunition, and also from lubricants and organic debris already present inside the firearm before discharge. The main source of organic discharge residue is the propellant.

Firearms discharge residue consists of a complex heterogeneous mixture that is claimed to be mostly particulate in nature (105). Particulate matter can be detected on a suspect but the possibility of vapourised/gaseous products being adsorbed on to skin or clothing surfaces also exists.

Interior ballistics has been defined as the science which investigates the way in which the chemical energy stored in the propellant (and to a much lesser extent the primer) is released and converted into the kinetic energy of the projectile (106). The discharge of a round of ammunition in a firearm produces high temperature and high pressure for a very short period of time. A typical time period from the hammer or firing pin striking the primer to the bullet or shot exiting from the muzzle, is in the region of 0.03 seconds. Due to the time period and the nature of the discharge process, only partial mixing of the constituents occurs and this accounts for the very heterogeneous nature of firearms discharge residue.

When a round of ammunition is discharged in a firearm, in addition to the projectile(s), firearms discharge residue is emitted, mainly from the muzzle but also from cylinder gaps, ejection ports and other vents in the firearm. Some of this residue may be deposited on the skin, head hair and clothing of the firer.

In the vicinity of the muzzle the hot propellant gases ignite and emit light on contact with the surrounding air. This effect is called muzzle flash. (The bullet can also cause a flash of light whenever it strikes a target that has a hard abrasive nature (107, 108)). The high pressure propellant gases at the muzzle are released into the air causing considerable turbulence and producing a powerful blast shock. This effect is known as muzzle blast.

Muzzle blast residue can be deposited on the target if the muzzle to target distance is less than 3 feet (109, 110). Muzzle blast residue nearly always contains unburnt or partially burnt propellant and soot. If the firearm is fired perpendicular to the target, the resulting residue pattern will be in a roughly circular area around the entrance hole. The diameter of the circle and the density of residue depends on the distance between the muzzle and the target. The pattern size, shape and density enables range of fire to be determined, gives an indication of the angle of fire and very occasionally the propellant grains can be identified from physical appearance as originating from a specific type of ammunition and enable a tentative identification of weapon type. Chemical analysis of the grains can yield useful information for comparison purposes with suspect ammunition. However it must be borne in mind that a propellant can change, both in colour and chemical composition, because surface coatings may be blown or burnt off during discharge and the original grains may not all have a uniform composition.

High speed photographic techniques have shown that during the discharge process some smoke emerges from the muzzle ahead of the bullet and also that just after the bullet leaves the muzzle it is surrounded by a large cloud of smoke over a short distance (111).

The reason(s) for smoke emerging in front of the bullet could be due to (a) some of the discharge gases escaping in front of the bullet as the cartridge case neck begins to expand just before the bullet starts to move out of the case neck; (b) gases escaping past the bullet before the bullet completely engages the rifling; and (c) obturation not being absolutely complete, allowing gases to escape in front of the bullet through the rifling grooves.

The propellant gases expand rapidly on release into the atmosphere, accelerating to velocities much greater than that of the bullet and this accounts for the bullet being surrounded by a large cloud of smoke.

Due to these effects, FDR is deposited on to the surface of the bullet. Whenever the bullet penetrates the target, due to the wiping action of the target material, some of the FDR on the bullet surface is transferred to the perimeter of the entrance hole. This occurs irrespective of the range of fire. On passing through the target, some if not all of the lightly adhering residue is removed from the bullet surface and consequently is not present or is present to a much lesser extent on the perimeter of the exit hole. This allows identification of bullet holes and bullet strike marks and differentiation between entrance and exit holes, which can in turn yield useful information about angle of fire, possible firing points and relative positions of firer and target.

Detailed analysis of the perimeter residue often yields useful information e.g., whether the bullet is unjacketed or jacketed and the nature of the jacket material. Primer type may occasionally be inferred and the presence of strontium or magnesium indicates a tracer or incendiary bullet respectively.

Crimes involving the use of firearms are particularly serious and demand the fullest investigative effort. Thus it can be seen that the detection, identification and quantification of FDR provides significant evidence in several areas associated with incidents involving the use of firearms.

One of the most important, difficult and challenging aspects of the work is to connect a suspect to a firearm, or to an involvement with firearms, by the presence of FDR on the suspect's skin, head hair or clothing, or inside their dwelling or motor vehicle.

A satisfactory test for the identification of FDR on a suspected firer has been sought by forensic scientists for many years. A satisfactory test would be one that is simple, reliable, fast, inexpensive and conclusive. Until recently efforts have concentrated mainly on the detection of inorganic components of FDR and encompass qualitative and quantitative methods, culminating in the Particle Analysis Method which is the most informative method currently available. A brief outline of the most important developments follows.

3.1.2 Development of firearms discharge residue detection techniques

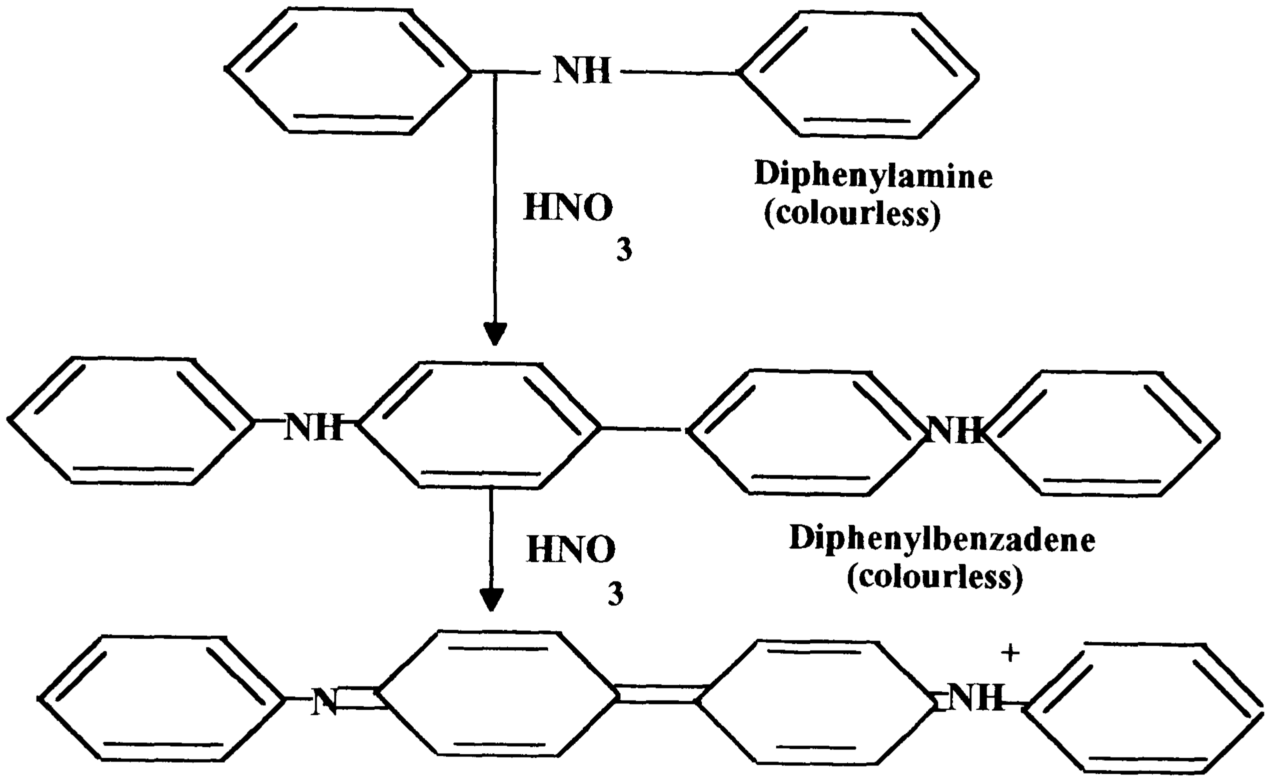
Paraffin test

The detection of nitrates and nitrites present in FDR using a colour reaction with diphenylamine in sulphuric acid was first applied to firearms related examinations about 1911, and in 1914 Doctor Iturrioz used paraffin wax as a lifting medium for propellant residue on clothing prior to treatment with diphenylamine/sulphuric acid reagent. The use of paraffin wax as a lifting medium led to the test being popularly referred to as the paraffin test.

In 1922 F.Benitez recorded this technique as a method for revealing the presence of propellant particles on the hands of a firer (112, 113, 114). In Mexico in 1931, T.Gonzales performed a modified version of Iturrioz's test using molten paraffin wax on the hands of a firer and in 1933 demonstrated the test in the USA. The test has also been referred to as the dermal nitrate test, the diphenylamine test and the Gonzales test (115, 116).

The test is conducted by applying melted paraffin wax to the back of a suspects hands. Using a brush the back of the hand is coated with paraffin which on cooling solidifies and can be peeled off the hand. The surface of the cast that has been in contact with the skin is treated with diphenylamine/sulphuric acid reagent by dropwise addition or spraying. The reagent produces a blue colour with individual particles of nitrates and nitrites.

The reaction sequence is as follows (117).



Quinoil Immonium Ion (blue)

The detection of dark blue spots was considered as indicative of the presence of nitrates and/or nitrites from FDR.

In 1935 the American Federal Bureau of Investigation pointed out that the test was not specific and had reservations about its use (118). Other evaluations of the technique proved it to be completely unreliable as an indicator of FDR. Common substances such as tobacco, tobacco ash, fertilizers, certain pharmaceuticals, certain paints and urine also give positive results (119,120). In addition a number of oxidising agents such as chlorates, bromates, iodates, permanganates, chromates, vanadates, molybdates, antimony (V) and ferric salts also give a reaction (121, 122).

At the Interpol conference in Paris in 1968 it was officially concluded that the Paraffin test should no longer be used (123).

Harrison and Gilroy method

In 1959 Harrison and Gilroy introduced a method based on the detection of the metallic containing components of FDR (124). The metallic components involved, namely, lead, antimony and barium, originate from the primer and the bullet (lead and antimony). The method is based on colorimetric spot tests and involves swabbing the suspects hands with cotton cloth damped with 0.1M hydrochloric acid. The swab is allowed to dry and is then tested with one or two drops of a 10% alcoholic solution of triphenylmethylarsonium iodide. The appearance of an orange ring indicates the presence of antimony.

The swab is then dried again and treated with two drops of freshly prepared 5% sodium rhodizonate solution to the centre of the orange ring. The development of a red colour indicates the presence of lead and/or barium. The swab is then dried a third time in the absence of strong light, and one or two drops of 1:20 hydrochloric acid are added to the red coloured area. A blue colour developed inside the orange ring is confirmation of the presence of lead. A red colour remaining in the centre confirms the presence of barium.

These tests were considered to be inconclusive, and the sensitivities of the colorimetric reagents used were not adequate to reliably detect the low concentrations found in actual firings (125, 126, 127, 128, 129).

Neutron Activation Analysis

Neutron Activation Analysis (NAA) is one of the most sensitive analytical techniques for many elements. A major breakthrough came in 1964 when NAA was applied to the quantitative detection of antimony and barium in FDR (130). (Antimony is the most valuable elemental indicator for FDR, lead and barium being more common in occupational and environmental surveys).

The method is based on the fact that when a sample is irradiated in a nuclear reactor for a specific length of time, atoms of some elements absorb neutrons. Nuclei that acquire an excess electron have a large excess of energy that is often released in the form of gamma rays. Nuclei with added neutrons are called radionuclides.

On placing the irradiated samples into a radio counter system capable of detecting and recording specific radiations it is possible to identify and quantify the elements of interest. If the elements emit gamma rays, the energy of the emissions and the decay lifetimes provides qualitative identification of the elements. The number of gamma rays per unit time versus the energy of the gamma rays is directly proportional to the amount of the element in the sample (131, 132, 133).

NAA is an excellent analytical tool which has and still is used successfully for the detection of barium and antimony in FDR. It has been applied to the detection of FDR on suspects, the identification of bullet holes in a variety of target materials, and range of fire estimations. Copper and mercury in FDR have also been determined (134).

NAA suffers from several major disadvantages for routine operation by most forensic laboratories. These include:-

(a) availability of and access to a nuclear reactor;

(b) high equipment costs and lack of trained staff;

(c) slow throughput of samples due to time required for irradiation, cooling and radiochemical separation;

(d) poor detection limit for lead (~ 10μg), a very important element in FDR work.

Much development work has been done on the NAA technique for FDR detection, but the inherent disadvantages of the technique led to a search for other more suitable quantitative methods. Despite its disadvantages NAA did much to increase our knowledge of quantitative aspects of the deposition and subsequent behaviour of FDR.

Many alternative techniques, both qualitative and quantitative, have been investigated either for screening purposes or as primary methods. Such techniques include Atomic Absorption, Molecular Luminescence, Electron Spin Resonance Spectrometry, X-ray analysis methods and Electroanalytical methods. Flameless Atomic Absorption Spectrophotometry (FAAS) is the technique that has almost completely replaced NAA.

Flameless Atomic Absorption Spectrophotometry

The FAAS method offers similar detection limits to NAA and is suitable for the determination of low levels of lead. Equipment costs are reasonable and the instrumentation is commonplace in many analytical laboratories. A large number of metallic elements, over a wide concentration range extending down to ultra trace level, can be analysed thus making the technique versatile and useful for other forensic applications as well as FDR detection. Apart from cost, the main advantages are simplicity, speed of analysis and 'in house' operation. One disadvantage of FAAS is the fact that it is not capable of simultaneous multi-element analysis.

In FAAS the sample is electrically heated to a high temperature thus breaking the chemical bonds and enabling individual atoms to float freely in the sample area. These ground state atoms are then capable of absorbing ultraviolet or visible radiation. The wavelength bands which each specific element can absorb are very narrow and different for every element. The desired element can be considered as being able to absorb only 'resonance lines' whose wavelength correspond to transitions from its ground state to some higher energy level.

A basic atomic absorption instrument includes a source of radiation, a system for placing sample atoms in the ground state, a monochromator to separate the resonance line of interest and finally a detector to measure the decrease in signal from the source when absorbing atoms are placed in the light beam. The magnitude of the decrease in signal is dependent on the amount of the element of interest in the sample.

Figure 3.01 illustrates the components of a basic flameless atomic absorption spectrophotometer.

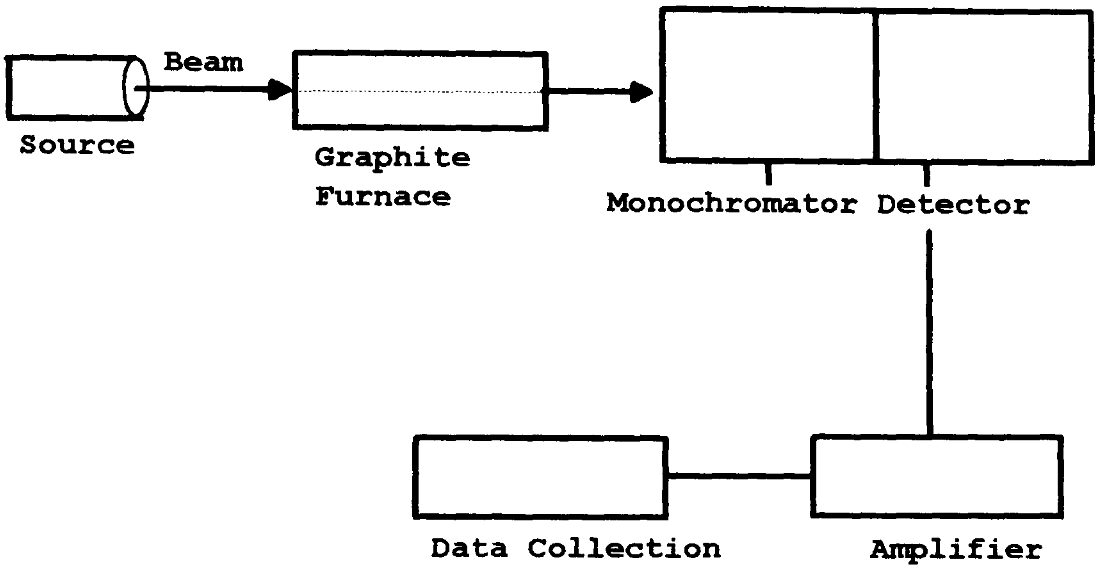


Figure 3.01 Basic components of a flameless atomic absorption spectrophotometer

FAAS is the most popular technique for the quantitative determination of elements associated with FDR i.e. lead, antimony, barium, copper and mercury. Other relevant elements have also been determined, and the use of FAAS for FDR detection is well documented in the literature (135, 136, 137, 138).

All bulk elemental analysis methods, such as NAA and FAAS, suffer from the serious disadvantage of lack of specificity in that the elements detected are not unique to FDR but also occur from occupational and environmental sources. Many surveys were carried out to determine background levels of lead, antimony and barium on the hands of people not involved with firearms. Some surveys also included copper and mercury. Both general and occupational data were gathered and threshold levels established for each of the elements. The threshold level may be defined as the level above which the results may be significant and correlate to the discharge of a firearm. The best that could be stated was that the levels detected were consistent with the discharge of a firearm but could not be taken as conclusive proof of the presence of FDR.

A more definitive method was sought resulting in the Particle Analysis Method which is claimed to conclusively identify FDR particles (139, 140, 141, 142, 143, 144).

Particle Analysis Method

This method employs a Scanning Electron Microscope equipped with elemental analysis capability (SEM/EDX) and combines details of the morphology and elemental composition of individual firearms discharge residue particles. A particle classification scheme was developed and is based on the elemental composition and morphology of individual FDR particles and is used to classify particles as either (a) of non-firearms origin (b) consistent with originating from the discharge of a firearm or (c) definitely from the discharge of a firearm. The ability to identify FDR particles uniquely and to distinguish them from environmental sources of lead, antimony and barium eliminates the threshold problem inherent in bulk elemental analysis.

In the SEM a beam of electrons is accelerated from a hot tungsten filament down a column by a high anode potential (up to 50kV), and by the use of three electromagnets (electromagnetic lenses) it is focused on the surface of the sample (specimen) which is mounted on a metal stub. The primary electron beam interacts with the elements at the surface of the sample causing, amongst other things, secondary electrons, backscattered electrons and characteristic X-rays to be emitted from the sample surface.

Figure 3.02 illustrates the different interactions.

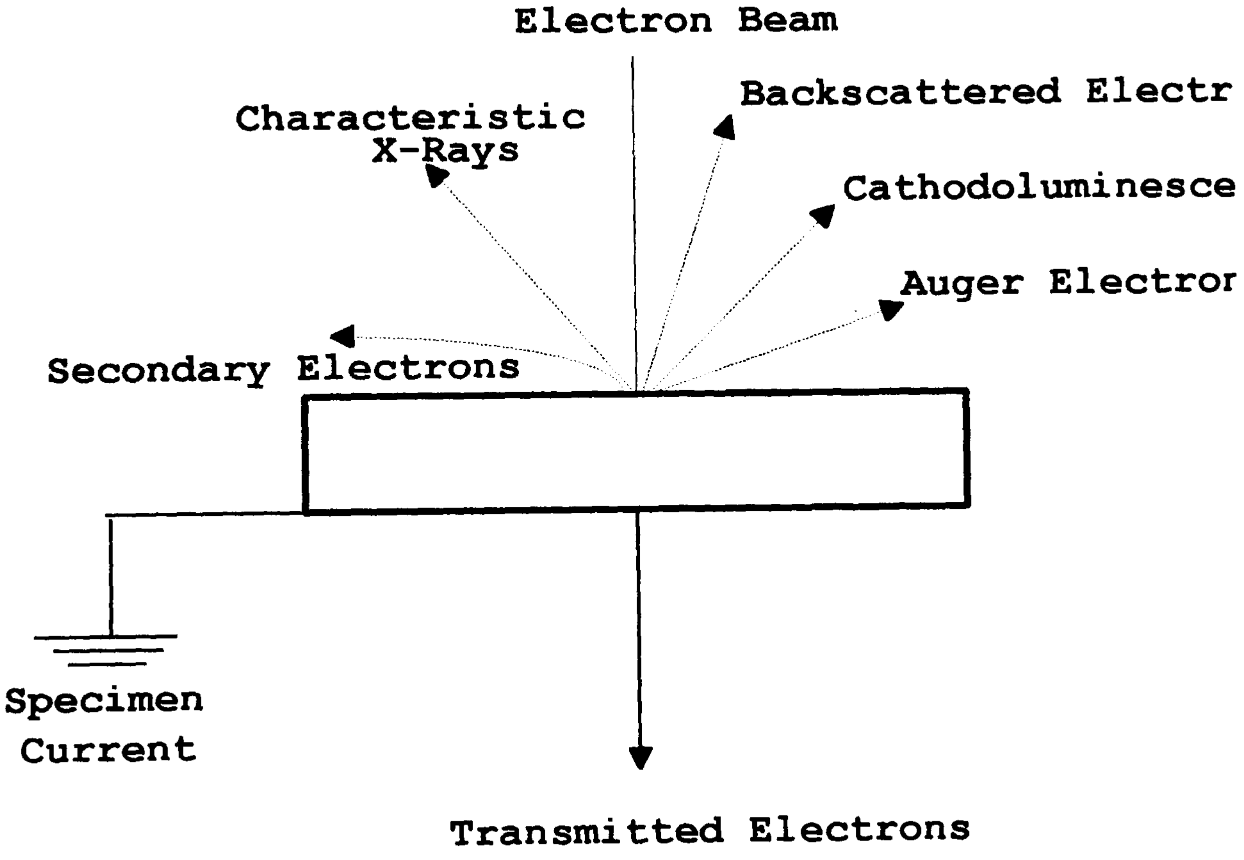


Figure 3.02 Electronic interaction with sample surface

These three effects are utilized in the Particle Analysis method as the surface of the sample is traversed (scanned) by the electron beam. The secondary electrons are used to view the sample, the backscattered electrons are used to identify likely FDR particles, and the X-rays are used to provide details of the elemental composition of the particles.

Scanning is achieved by the use of scanning (raster) coils which cause the primary beam to be electromagnetically deflected across a given area of the sample surface. The raster pattern of the beam is synchronized with the scanning pattern of the cathode ray tube.

The low energy secondary electrons (less than 50eV) ejected from the sample surface are attracted to a scintillator on the end of a perspex light guide, the other end of which is in contact with the window of a photomultiplier tube. The amplified signal is displayed on a cathode ray tube which records the image. The secondary electrons are attracted to the scintillator by a positively charged cage surrounding the end of the light guide. The number of electrons reaching the scintillator is dependent upon (a) the topography of the sample surface since this will influence whether or not a particular area on the sample surface is visible to the primary beam and detector and (b) the elemental nature of the sample surface as this will affect the energy of the secondary electrons and consequently their susceptibility to the cage potential. The topography is the most important of the two factors.

Backscattered electrons are those electrons which have undergone single or multiple scattering events, and escape back through the surface of the sample with energies greater than 50eV. Backscattered electrons travel in straight lines and because of their higher energies they are not attracted to the secondary electron detector. Backscattering increases as the atomic number of the sample increases and there is such a strong correlation with atomic number that the relationship forms the basis for a contrast mode in SEM.

FDR particles contain elements with high atomic number (heavy metals) and this fact is used to aid the search for FDR particles located amongst many other particles with similar morphology but from non-firearm related sources. The backscattered electron image is displayed on a separate screen. All particles containing heavy metals show stronger emission and appear as bright areas on the screen; only the bright particles are potential FDR and consequently only these particles need to be analysed. Without the aid of a backscattered image, all particles with similar morphology to FDR would need to be analysed.

Characteristic X-ray emission is one process by which an atom may stabilize itself following ionisation by the electron beam. When an electron from an inner atomic shell has been dislodged, an electron from an outer shell will replace it. The difference in energy between the initial and final state may be emitted as X-radiation. The various shells of an atom have discrete amounts of energy. It follows that their energy difference, emitted as X-radiation, is also a discrete quantity and is characteristic of the atom from which it was released. X-ray spectroscopy in the SEM, as used for FDR work, involves the identification of radiation of specific energies using a special detector, and can identify elements heavier than sodium.

Detection of lighter elements is not possible using a conventional detector due to the inefficiency of X-ray generation for the lighter elements.

Figure 3.03 illustrates the basic components of a SEM suitable for FDR work.

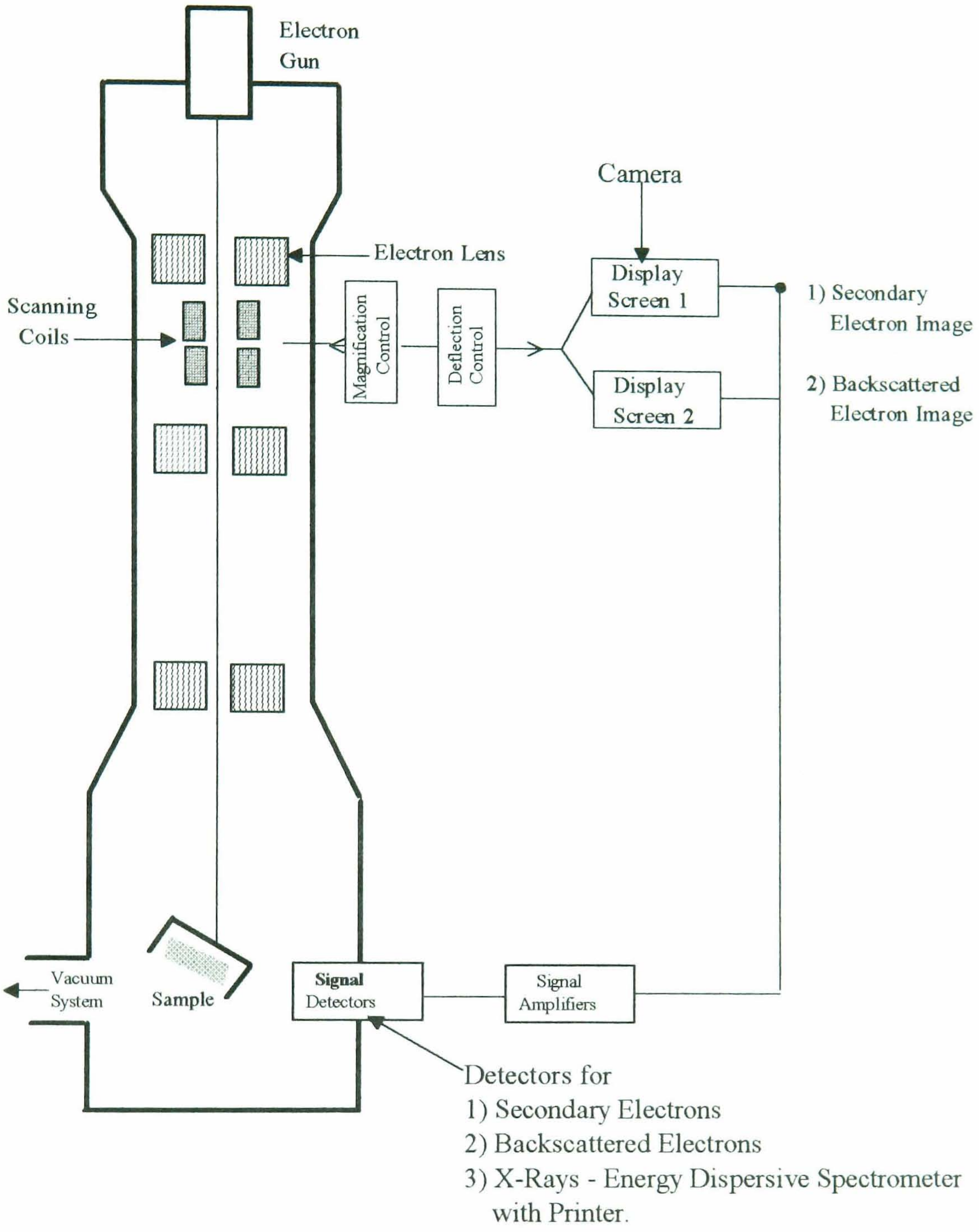


Figure 3.03 Basic components of a scanning electron microscope suitable for FDR detection.

Samples that do not conduct electricity and heat (insulators) cause problems in the SEM unless they can be made conductive by some means. Problems include charging and overhearing of the sample. Coating samples with a thin layer of conductive material helps to overcome the problems.

Samples for FDR examination are taken from a suspect's skin and/or clothing surfaces by a non-destructive sampling technique and prepared for examination in the SEM. FDR samples are coated with a very thin layer of carbon using either a vacuum coater or sputter coater before introduction to the SEM. The examination involves searching for FDR particles amongst many other particles from occupational and environmental sources, and is a labour intensive and time consuming task. FDR particles are recognised by a combination of morphology and elemental composition, and are classified as FDR using a scheme developed for the purpose. The classification system will be discussed at a later stage.

Particle analysis is the most informative method to date for the identification of FDR particles. It does however suffer from several major disadvantages including high cost of instrumentation and lengthy and tedious procedures requiring specialised staff. Since its introduction serious attempts have been made to solve the time problem. These include the use of backscattered electron images, automation of the search procedure and sample manipulation to pre-concentrate the sample prior to SEM examination (145, 146, 147, 148, 149, 150, 151).

Despite all the considerable improvements, the Particle Analysis method remains a lengthy and costly procedure. These disadvantages have renewed interest in the possibly of detecting the organic components of FDR, either as a primary method or as a screening technique.

3.1.3 Detection of organics in firearms discharge residue

Chromatographic techniques are the main methods that have been used to separate, detect and identify organic components of FDR (152, 153, 154, 155, 156, 157, 158, 159, 160, 161, 162). Other methods considered include Molecular Luminescence (163), Infrared Spectroscopy (164), Raman Spectroscopy (165), Electron Spin Resonance Spectrometry (166), Microchemical crystal tests (167, 168), Ultraviolet Spectroscopy/Nuclear Magnetic Resonance/Polarography (169).

Many of the organic constituents of FDR are explosive or explosive related compounds and much of the work already done on the detection of explosive residues can be extended to include FDR. Explosives and their residues are usually analysed using chromatographic techniques. Chromatography is the general name given to the methods by which two or more compounds in a mixture physically separate by distributing themselves between two phases (a) a stationary phase - which can be a solid or a liquid supported on a solid and (b) a mobile phase - either a gas or a liquid which flows continuously around the stationary phase. The separation of individual components results primarily from differences in their affinity for the stationary phase.

Of all the methods investigated for organics in FDR it would appear that High Performance Liquid Chromatography (HPLC) and Gas Chromatography with a Mass Spectrometer detector (GC/MS) are currently the most promising.

High Performance Liquid Chromatography

In liquid chromatography the mobile phase is a liquid. The stationary phase is a solid contained inside a long narrow tube called 'the column'. Separation of mixtures occurs during passage through the column. The separation can occur by different mechanisms depending on the type of material in the mixture and the column e.g. adsorption, exclusion, ion exchange, partition. Whatever the mechanism, the volume of mobile phase at which a component elutes is constant and characteristic of that component, for a given chromatographic system. Thus the retention volume (time) can be used for qualitative identification. If the detector response is related to the amount of component in the sample then the area under the chromatographic peak gives a measure of the quantity present.

The components in a mixture separate in the column and exit from the column at different times (retention times). As they exit the detector registers the event and causes the event to be recorded as a peak on the chromatogram. A wide range of detector types are available and include ultra violet adsorption, refractive index, thermal conductivity, flame ionisation, fluorescence, electrochemical, electron capture, thermal energy analyser, nitrogen-phosphorous. Other less common detectors include infra red, mass spectrometry, nuclear magnetic resonance, atomic absorption, plasma emission.

Chromatography is a very versatile technique offering a wide range of solid phase materials and detector types which can deal with very complex mixtures. In practice all materials and conditions used in the instrument are carefully chosen to match the type of sample mixture involved. This includes selection of stationary phase (chemical and physical properties); column type and length; sample pretreatment; operational temperatures, pressures and flow rates; physical and chemical nature of mobile phase; detector type etc., Detection to nanogram level is quite common and some systems can detect to picogram level using very small volumes of sample.

One of the major differences between normal Liquid Chromatography and High Performance Liquid Chromatography is the column. In liquid chromatography the solid phase consists of large porous particles (75 - 200 microns) packed into columns with internal diameters of 1-5 cm. Very low pressures are required to permit solvent flow (mobile phase) through the large particles in the column. Flow rates are very slow and separation times are long. HPLC uses narrower column diameters and solid phase particles that are much smaller and more uniform (3-10 microns). This leads to much larger back pressures than with liquid chromatography and high pressure pumps are required. However the column efficiency is increased 10-100 fold and separation times decreased compared to liquid chromatography. Separation methods employed in HPLC include normal phase, steric-exclusion, ion exchange, ion pair and reversed phase.

Gas Chromatography/Mass Spectrometry

A gas chromatograph is an apparatus consisting of an injection port connected to a column that has a detector at its outlet end. The column is contained in an oven that is electrically heated, either isothermally or at a programmed rate. A stream of inert carrier gas, usually helium, is introduced into the injection port and flows through the column and detector. The injection port is a heated region that is sealed from the outside environment by a silicone rubber septum through which the sample is injected using a hypodermic syringe. Individual sample components are vapourised and travel through the column at a rate dependent on their interaction with the material used to pack the column. The detector registers and records the output from the column. In this case the detector is a Mass Spectrometer.

Mass Spectrometers are sophisticated instruments which produce, separate and detect positively charged gas phase ions. (Negatively charged ions can also be investigated but the abundances of negative ions are 10 to 1000 times less than those of positive ions and negative ion mass spectrometry is less frequently used). Pure compounds can be identified by their characteristic ions and the result is nearly always unambiguous.

The Mass Spectrometer consists of an inlet system, an ionisation device, a mass analyser and an ion detector and the system is kept at a vacuum of about 10^{-4} to 10^{-7} Torr. (There are various options available for all four basic components which illustrates the versatility of the technique). The following describes an electron impact - quadrupole mass spectrometer.

Neutral gaseous molecules entering the ionisation area are bombarded with electrons to 'smash' the compound of interest to yield positively charged ions. Ionisation is often followed by a series of spontaneous competitive decomposition reactions (fragmentation) which produce additional ions. The instrument operates under a high vacuum to prevent the absorption of the charged particles by air molecules.

The ions are then extracted from the ion source, collimated and accelerated by an electric potential applied across a series of metal plates with exit apertures, before entering the quadrupole mass analyser. The quadrupole mass analyser consists of four circular parallel rods. The rods are electrically connected in diagonally opposite pairs and mass separation is achieved by applying a DC potential, positive to one pair and negative to the other and superimposing a radio frequency AC potential which differs in phase by 180° between the pairs of rods. The peak values of the AC voltage is greater than the DC voltage so that the 'positive' pair are sometimes negative and vice versa.

This produces a dynamic arrangement of electromagnetic fields resulting in certain ions taking a stable path through the analyser and reaching the detector, whereas other ions take unstable paths and are filtered out before reaching the detector. The detector consists of an electron multiplier which magnifies the current generated by the ions striking the detector by a factor of 10^6 to 10^9. The event is recorded by some form of data system. The mass spectrum is a plot of the electron multiplier output (intensity) versus the quadrupole electrical settings and produces a characteristic pattern of fragments for the compound under investigation.

Mass spectrometry on its own is not suitable for FDR work because generally speaking 'pure' compounds must be analysed. Samples for FDR examination taken from skin and clothing surfaces are complex mixtures containing many unpredictable contaminants from occupational and environmental sources as well as skin salts and lipids. The problem is solved by separation of the constituents using gas chromatography prior to introduction to the Mass Spectrometer.

Combined Gas Chromatography - Mass Spectrometry (GC-MS) takes advantage of the separating power of the gas chromatograph and the identification power of mass spectrometry. The gas chromatograph separates the components and provides retention time data and the mass spectrometer identifies the components. The combined instrumentation has the potential to provide very useful information in FDR casework.

Figure 3.04 illustrates a quadrupole mass spectrometer.

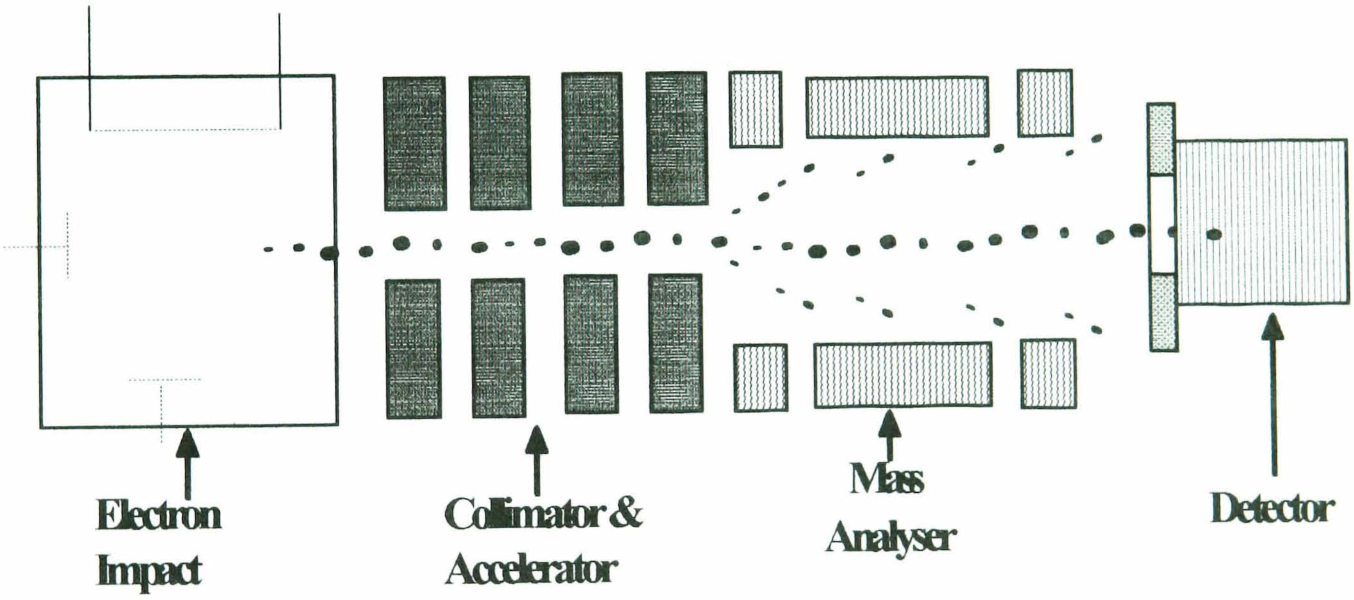


Figure 3.04 Quadrupole mass spectrometer

3.1.4 Properties of firearms discharge residue

Formation

The development of the Particle Analysis method for FDR detection and identification involved consideration of how the particles are formed and of their physical and chemical nature (170,171).

At the present time the exact mechanism(s) of formation of the particles can only be deduced from considerable practical experience and limited experimental work, most of which, if not all, has been concerned with the elemental content of FDR particles.

An extensive study of the nature of FDR particles was conducted at the Aerospace Corporation, California, USA in the mid-1970's. From firings involving a wide range of handguns and ammunition, extensive statistics were gathered about the size, shape and elemental composition of discharge particles. On the basis of these observations, several hypotheses have been suggested concerning the formation of the particles.

FDR is mostly particulate in nature. Unjacketed lead bullets produce residue in which greater than 70% of the particles are lead. Coated bullets give the same result, except that a substantial proportion of the lead particles contain copper from the coating material. With jacketed or semi-jacketed bullets the proportion of lead particles in the residue is greatly reduced. It was concluded that most of the lead in the residue comes from the bullet rather than from the primer. This has subsequently been confirmed by experiments involving the use of radioactive tracers (172).

The stability of the smaller lead particles under the SEM electron beam suggests that they consist of metallic lead rather than lead oxides. Considering that most of the lead particles originate from the bullet, coupled with their stability under the electron beam, leads to the conclusion that the vast majority consist of metallic lead.

Particles due to the discharge of a firearm can be loosely divided into two types, those originating from the bullet, and those originating from the primer. This is a broad general classification only and is not absolute. Particles classified as primer particles because of their elemental content are unlikely to contain metallic lead due to the fact that they were initially compounds and are unlikely to be reduced to the metal in the oxidising environment of the primer ignition.

The formation of particles is thought to proceed in the following manner. The hot, high pressure gases from the burning of the propellant (and primer) act initially on the exposed lead at the base of the bullet. The bullet then passes through the barrel and is subjected to strong frictional heating. This causes some of the bullet and bullet jacket material to be melted and vapourised as well as small fragments to be stripped from the bullet by the rifling. The metal vapours from the bullet mix, to some extent, with the vapours from the inorganic compounds of the primer and are emitted from the firearm through the muzzle and other gaps. They then condense into particles, some of which could be deposited on to the skin and clothing surfaces of the firer.

Discharge particles can be broadly classified into bullet particles and primer particles. This is not unexpected considering that whilst vapours are miscible, most inorganic compounds in the liquid and solid state cannot dissolve metals and vice versa. Upon solidification the metals would be expected to separate from the compounds and form separate particles.

Copper is found in both bullet and primer discharge particles if the bullet is coated or jacketed with copper or copper containing alloy. Discharge residue from ammunition with bare lead bullets shows a very marked decrease in the number of copper containing particles compared to ammunition with coated or jacketed bullets. This is strong evidence that the copper originates primarily from the bullet coating or jacket rather than from the primer cup assembly or the cartridge case. To explain why copper is found in both bullet and primer discharge particles it is assumed that some proportion of the metallic vapours become oxidised by oxygen or sulphur from the vapourised primer mixture and possibly by atmospheric oxygen outside the gun. Thus the primer (compound) particles contain a contribution from the bullet.

Experiments were conducted involving the addition of tracer compounds to the propellant, followed by an examination of the discharge particles to determine if any of the particles and which type contained the tracer (173). The tracer was found in primer particles only which supports the proposition that they should be found dissolved in the oxides and salts originating from the primer and not in the bullet particles. A similar experiment involving the coating of bullets with metals not normally associated with firearms ammunition, followed by examination of the discharge particles, supported the proposition that the bullet material makes a contribution to the primer particles.

It was also observed that the number of discharge residue particles on the firing hand decreased markedly as the bullet velocity increased. One possible explanation for this is that the greatly increased suction in the wake of the faster bullet causes more particles to be sucked out of the muzzle leaving fewer to exit from other vents and to be deposited on the hand. This explanation is supported by the observation that the number of bullet particles deposited is reduced slightly more as a function of velocity than is the number of primer particles. Primer particles are on average larger and are produced further away from the bullet than the bullet particles.

Very little is currently known about the formation and nature of organic discharge residue as the main thrust, until relatively recently, has been directed towards the inorganic content of discharge residue.

Morphology and size

Apart from various gases four types of particles have been detected in firearms discharge residue:-

1) Spherical particles

2) Irregular particles

3) Clusters of particles

4) Fragments of smokeless powder

As a general rule greater than 70% of the particles are spheroidal. They may be perfect spheres or they may be distorted in some way. Three-dimensional roundness is the basis for this classification. The surface of the particles may be smooth, fuzzy, scaly or have smaller spheres on their surface. Sometimes they are perforated, capped, broken or stemmed. The vast majority have diameters of less than 5 microns although they range in size from < 0.5 microns to greater than 32 microns (it is not practical to detect particles less than 0.5 microns in diameter).

The irregular particles constitute up to about 30% of the total particle population. The size varies over a wide range from < 1 micron to several hundred microns. The larger of the irregular particles sometimes have some small spheroidal particles attached to them. The irregular particles have the same compositions as the spheroidal particles.

None of the particles exhibit any features that would suggest a crystal or mineral origin, such as straight or sharp edges, and they are frequently squashed or flattened in appearance. FDR particles are not visibly crystalline although Diffraction Methods do give crystal patterns. Limited diffraction experiments on FDR particles have identified barium meta-antimonate $BaSb_2O_6$, lead oxy-sulphate $PbO.PbSO_4$, graphite, lead, lead sulphide PbS, antimony oxide Sb_2O_4 (tentative), a possible intermetallic compound Ba Pb_3 , lead oxide PbO, lead dioxide (peroxide) $Pb0_2$, tri lead tetraoxide $Pb_3 0_4$ and barium sulphide BaS, and there are probably others. The ability to identify the compounds present would further enhance the discriminating power of the method but, as yet, this is not a practical proposition.

Clusters consist of from five to several hundred spheroidal particles attached to each other in a similar fashion to a bunch of grapes. They occur infrequently and appear to be a product of high power or high velocity. Flakes of smokeless powder are few in number and are occasionally seen in promptly collected residue. Unlike the other three types of particles which are inorganic in nature, the flakes are organic although sometimes spheroidal particles are embedded in their surface. They range in size from about 50 microns to 1000 microns. Clusters and powder flakes are rarely seen in casework as they are usually relatively large in size and would be lost rapidly from skin and clothing surfaces.

Spheroidal particles are thought to result by rapid condensation from a vapour whereas the irregular particles may be produced by solidification of droplets of molten material that are flung against the inside surfaces of the firearm.

Composition

The identification of a particle as FDR is based on a combination of morphological and elemental composition criteria and also on the association of the particle with other particles present in the sample.

The following three particle compositions have thus far been observed only in FDR and are considered unique to it:-

Pb, Sb, Ba
Ba,Ca, Si, with a trace of S
Sb, Ba

The following five particle compositions are consistent with FDR but are not unique to it:-

Pb, Sb
Pb, Ba
Pb
Ba if S is absent or only a trace
Sb (rare)

The Pb, Sb and the Pb, Ba particles, although not unique, have been found in few occupational residues and are therefore considered to be fairly characteristic.

Any particle, unique or consistent, may also contain one or several of the following and only the following elements:- Si, Ca, Al, Cu, Fe, S, P (rare), Zn (only if Cu > Zn), Ni (rare, only with Cu, Zn), K, C1. The presence of some Sn is a possibility in obsolete ammunition.

The compositions, shapes, sizes, appearance and range of particle types should all be considered during interpretation. Particles that are individually consistent with FDR should not be found with otherwise similar particles that are in some way inconsistent with FDR.

The particle classification scheme is the basis of the Particle Analysis Method for FDR detection and identification. A revised particle classification scheme is presented in Chapter 5.

Other work has shown that the elemental composition can vary between the inside and the outside of a particle and using SEM and NAA the average amount of Ba and Sb per particle was determined to be in the region of 0.2 ng to 20 ng for Ba and 0.4 ng to 7 ng for Sb.

Deposition

There is evidence to suggest that the FDR on the firers hand is blasted on to the hand during the firing process and that residue settling from the atmosphere does not appear to contribute. Copious amounts of FDR issue from the muzzle but play a secondary role in hand deposits. The hand deposits are mainly emitted from openings around the breech and ejection port in self loading firearms and from the flash gap between the cylinder and barrel in revolvers (174). Such residues can also be deposited on to the face, head hair and clothing of the firer.

Many factors are thought to influence the amount and nature of FDR deposited on the firer. It should also be borne in mind that there can be a considerable variation in the amount recovered for analysis depending on the sampling method and on how efficiently it is used.

Such factors include:-

(a) the type of gun - a low success rate for FDR detection has been observed for casework involving .22" calibre revolvers and rifles, and for shotguns which are usually closed breech weapons; some rifles i.e. bolt action, require manual extraction of the spent cartridge case; consequently the design of the firearm can have an influence on the quantity of FDR deposited;

(b) the mechanical condition of the gun - a gun in poor mechanical condition is likely to have larger gaps in the firing mechanism, thereby allowing more FDR to escape;

(c) cleanliness of the gun - contact with a 'clean' gun is likely to produce less FDR than contact with a 'dirty' gun of the same type;

(d) type of ammunition - jacketed bullets produce substantially fewer FDR particles than unjacketed bullets. Primer type (size, composition, burning characteristics) can influence the number of primer particles produced. The temperature and pressure achieved by the burning of the propellant determines the power and velocity of the bullet which can influence the number of particles deposited;

(e) direction and force of air currents (wind) - tests involving similar firings with the same gun and ammunition batch, one series conducted indoors and the other outdoors, produced substantially less FDR on the firer in the case of the outdoor tests. This was thought to be due to the effect of the wind. Other climatic factors such as rain, humidity and temperature could also play a part;

(f) firing location and duration of exposure - firing from confined spaces e.g. doorways, small rooms, vehicle interiors, will tend to expose the firer to a more residue laden environment for a longer than normal period of time, consequently, it will take the FDR longer to disperse and the chances of FDR from the muzzle being deposited are increased; and

(g) nature of surface - skin condition (dry, moist, natural oils, amount of hair) and nature of clothing (smooth or rough) are thought to influence the quantity deposited.

Other factors include the position of the gun relative to the firer i.e., whether the arm is outstretched or closer to firer at time of discharge, whether firing is single or double handed, whether the firer is sitting, standing, kneeling or lying down will all affect the surface area available for deposition of FDR. Subsequent manipulation of the gun such as unloading, reloading, cleaning and picking up spent cartridge cases can yield more FDR than the actual shooting.

Quantity and Composition

Bulk elemental analysis methods have been employed by many workers to do quantitative studies of the amount of residue deposited on the firing hand.

Surveys based on indoor firings of a range of handgun/ammunition combinations and sampling of the firing hand using a variety of sampling techniques, followed by analysis using differing sample preparation/analytical methods, gave the following widely varying results for residue collected immediately after firing from 1 to 6 shots:-

Pb	0.45 μg to 326.0 μg	Average = 7.810 μg
Sb	0.01 μg to 10.1 μg	Average = 0.448 μg
Ba	0.02 μg to 13.7 μg	Average = 0.828 μg

There is very limited data available concerning the total discharge residue particle population from the firing of ammunition. Available data suggests wide variations in total particle population from repeat tests using the same gun and ammunition from the same batch.

A series of repeat tests involving the firing of a single round, one handed, with clean hands and sampling by a standardised procedure gave the following results. The mean of six determinations was 5,315 ± 3,622 (68%) for total particles and the mean for the percentage of primer particles was 50.5 ± 14.4 (29%). In another test series the mean of five determinations was 203 ± 81 (40%) for total particles and 65.4 ± 10 (15%) for the percentage of primer particles.

From a single firing of three types of .38" special calibre ammunition the following results, Table 3.01, for promptly collected residue were obtained using the Particle Analysis Method.

Cartridge	***Total Spherical Particles***	***Pb only***	***Ba only***	***Pb, Ba***	***Pb, Sb***	***Pb, Sb, Ba***	***Pb, Cu***	***Non-Spherical Particles***	***Powder Flakes***
125 Grain JHP Remington	142	35	35	34	13	19	6	13	18
158 Grain RNL Remington	2,664	2,086	66	367	39	106	0	28	4
158 Grain RNL Federal	4,551	4,162	0	101	95	193	0	0	3

Table 3.01 Particle population, one shot, same calibre

The vast majority of the total particle population was due to Pb only particles originating from the bullet in the case of the RNL (round nosed lead) ammunition whereas the JHP (jacketed hollow point) ammunition produced a much smaller proportion of Pb only particles because the base and side of the bullet are enclosed in the jacket, the only exposed lead being at the nose of the bullet (175). This partly explains the much larger particle population experienced with unjacketed bullets.

Repeated firings with the same gun do not necessarily yield progressively increasing levels of residue nor does the same gun/ammunition combination firing the same number of shots under apparent similar conditions necessarily yield comparable levels of residue (176).

It is known that FDR consists of gases and a heterogeneous mixture of particles that contain Pb, Sb and Ba either individually or in combination. The size of the particles can vary from < 1 micron to > 100 microns.

To explain anomalies in quantity and composition of FDR deposited under very similar conditions, it has been proposed that most of the mass of the elements detected is contained in a few large particles and that a large variation occurs in the number and composition of these large particles recovered from firing to firing. It has also been suggested that the skin becomes saturated with residue and that the blast from subsequent shots dislodges residue from previous shots.

It is obvious that the discharge process and the subsequent deposition of FDR on the firer are both subject to many factors, the overall result of which is an unpredictable amount of FDR being deposited. What is deposited, if anything, has a varying composition. The overall process is very random.

Distribution

Distribution may be defined as "the areas where FDR is deposited and the concentration in those areas".

It was thought that rifles and hand guns when fired in the normal manner would produce different distribution patterns on the firer, rifles being more likely to deposit FDR on the face than handguns. It was also thought that the distribution on the hands could determine whether or not the test subject had discharged or merely handled a gun. On firing a gun more residue is expected to be deposited on the back of the firing hand than on the palm.

Whilst these initial distribution patterns may be correct under ideal test conditions, loss of residue during the course of normal activity and redistribution by transfer from area to area complicate the issue. With the exception of suicides and dead suspects, the initial distribution pattern will almost certainly have altered markedly between the incident and sampling, which is typically several hours after the event. In practice the circumstances of the case dictate the areas to be examined for FDR.

There are many factors that influence the initial distribution pattern and most have been mentioned when discussing the quantity of FDR deposited. This coupled with the loss of residue with time and activity, and the fact that residues are readily transferred from area to area means that any interpretation based on distribution needs to be approached with caution. Distribution is of limited practical value, but it has been an important factor in several unusual cases.

Persistence

The persistence of FDR on the hands has been studied by many workers and whilst there are wide variations in the literature (1 hour to 24 hours) the majority opinion is in the region of 1 to 3 hours from the event to the sampling of the suspect (177, 178, 179). Beyond 3 hours it is unlikely that residue will be detected on the hands of live suspects.

Persistence on face, head hair and clothing has not been studied, but it is highly likely that FDR will persist on these areas for longer periods than on the hands. Persistence data from casework experience is presented in Chapter 5.

3.1.5 Sample collection

Some form of kit containing sampling materials is normally prepared, (either commercially, by police forces or by forensic laboratories) for the purpose of sampling suspects for FDR (180, 181, 182).

Kit design considerations include (a) avoiding contamination from external sources and cross contamination between sampling areas; (b) lifting efficiency; (c) matrix compatibility with subsequent laboratory procedures; (d) ease of use and preparation; and (e) cost, purity, availability of materials.

Hand sampling methods include:-

Cloth)
Cotton wool) and dilute acid SWABBING
Filter paper)

Dilute acid wash	WASHING
Paraffin 'glove'	FILM LIFTS
Film forming polymers	
Adhesive tape	ADHESIVE LIFTS
Adhesive stubs	

The analytical technique used strongly influences the type of sampling procedure. Sampling methods fall into two categories, destructive or non-destructive, depending on the effect of the sampling procedure on individual FDR particles, acids tending the break down the particles.

Whatever form of kit is used the lifting efficiency depends to a large extent on the care taken by the sampler.

3.2 OBJECTIVES OF EXPERIMENTAL WORK

The introductory chapters are mainly an in depth review of the available literature and as such do not necessarily represent my opinions or experience.

The purpose of the experimental work is to attempt to improve systems for firearms discharge residue detection and identification, with particular reference to the Northern Ireland situation. This will involve looking at all aspects including suspect handling, sampling, laboratory preparation, analysis techniques, interpretation and presentation of results in a court of law. There is very little information in the literature about suspect handling, contamination avoidance, interpretation of results and presentation of evidence in court. By taking an overview and a practical approach to all aspects of the system, it is hoped that by introducing improvements, however minor, in each area, these will have a cumulative effect, leading to a substantial overall improvement. This is a novel approach, the vast majority of the literature dealing with scientific methods of sampling, detection and identification, which whilst extremely important, are not the end product; the end product is the value and credibility of evidence given in court.

There are three main areas for consideration.

1. At the start of 1978 the Particle analysis method (140) replaced the Flameless Atomic Absorption bulk elemental method (138) as the firearms residue detection method in this laboratory. Since then the Particle Analysis method has been substantially improved by the use of a sample concentration - clean up procedure (149), the addition of a backscattered electron detector and the development of an automated residue detection system (145, 146). Despite these improvements the technique remains costly and labour intensive. Certain aspects of the system require further work, in particular, the particle classification scheme, discharge particles from mercury fulminate primed ammunition and discharge particles from new primer types (Sintox).

The validity of the particle classification scheme, section 3.1.4, will be tested by examining items that may produce similar particles, paying particular attention to blank cartridges, the main uses of which that are likely to be encountered in casework are cartridge tools and blank firing replica/imitation firearms.

The particle classification scheme is based on modern primed ammunition and consequently mercury fulminate primed ammunition is not included. Mercury containing particles from the discharge of mercury fulminate primed ammunition are rarely detected in casework. Discharge residue from such ammunition will be tested in an effort to provide an explanation for this. Discharge particles from Sintox primed ammunition will also be examined with a view to anticipating future problems the criminal use of this new ammunition may cause for the Particle Analysis method, range of fire estimations and the identification of bullets holes.

A major review of 23 years casework data shall also be undertaken for the following reasons: -

(a) to check the validity of the literature review on the chemistry of ammunition, much of which is based on information gathered over a 23 year period from numerous sources, many of which are of a non-scientific nature e.g. gun magazines, newspaper articles, manufacturers sales literature.

(b) to demonstrate the variations in and complexity of the basic item involved in firearms crimes, the round of ammunition, a detailed knowledge of which can aid both the physical and chemical investigation of scenes of crime and the subsequent laboratory examination.

(c) to record information gained from a terrorist campaign lasting 26 years, much of which will be of interest and benefit to the scientific community and will not be found published elsewhere.

(d) to clarify the list of accompanying elements in the particle classification scheme, and the levels at which they are found.

(e) to provide an insight into the types of mercury containing particles detected in casework.

2. With the emphasis on quality all systems will be explored, both internal and external, with a view to ensuring that they withstand close scrutiny from any source, that the possibility of cross contamination of suspects with explosives and/or firearms residue is minimised and that contamination risks within the laboratory are identified and minimised or eliminated.

3. The detection and identification of the organic constituents in firearms discharge residue has the potential to be used either as a screening technique or, much more likely, as a complementary technique to the Particle Analysis method. The Particle Analysis method has proved very satisfactory and has been well tried and tested in casework and court. The objective is to devise an efficient system for organic residue detection that is entirely compatible with the Particle Analysis method. As a suspect may need to be examined for both firearms and explosives residue the method must also be compatible with organic explosive residue detection techniques.

Summary of aims

1) Improve the particle classification scheme.

2) Explain the scarcity of discharge particles containing mercury.

3) Clarify the types of discharge particles containing mercury that have been detected in casework.

4) Gain information about recently introduced Sintox ammunition in anticipation of its use in crime.

5) Increase knowledge about the chemistry of firearms by reviewing 23 years casework results and related laboratory tests.

6) Improve suspect processing procedures and contamination avoidance measures.

7) Devise a method to enable organic FDR detection to be readily incorporated into our existing systems for inorganic FDR and organic explosives residue detection.

8) To substantially improve the overall system for firearms and explosives residue detection from the initial arrest of the suspect to the presentation of evidence in court.

Note:

(a) For convenience the elements Pb, Sb, and Ba are referred to as the primary elements in FDR particles. Thus a single primary element particle would be termed Pb only, Sb only or Ba only but they could have, and typically do have, other elements present in the particle from the list of permitted additional accompanying elements.

(b) The terms major ,minor and trace level are defined as in reference 183 and when recording the analysis of a particle all the elements present are listed in order of descending peak height.

(c) Due to a terrorist bomb attack on the laboratory in September 1992 some material relating to this work was destroyed or lost. Fortunately the bulk of it was salvaged. However some details were lost and these will be mentioned in the text.

(d) Personnel providing information under 'Private Communications' cannot be identified for security reasons but may be contacted through the author should it become necessary.

4 EXPERIMENTAL PROCEDURES

4.1 INSTRUMENTATION

Scanning Electron Microscopy

SEM/EDX analyses were performed using a Camscan series 2 scanning electron microscope connected to a Link AN 100000 energy dispersive x-ray analyser. For automatic detection an automatic residue detection system (ARDS), developed at this laboratory, was employed the details of which are given in reference 146. For manual operation the following conditions apply.

- 29 mm working distance
- 1-1.25 x 100 μA emission
- x 1000 magnification
- 25 kV accelerating voltage
- 0° tilt and resolution and absorbed current set according to the microscope on which the examination is carried out.
- Brightness/contrast set on Fe particle until just visible on the backscattered image.

Swab and Deldrin samples for SEM/EDX examination were subjected to a concentration/clean up procedure, the apparatus for which is given in reference 149. All samples were carbon coated using a Biorad E6430 automatic vacuum controller before examination in the SEM.

Gas chromatography / Thermal energy analyser

Details of the GC/TEA system are as follows. The instrumentation is a Hewlett Packard 5890 GC with a Thermedics 543 Thermal Energy Analyser connected to a Hewlett Packard 3393A integrator. The system also incorporates a Hewlett Packard 7673A auto-injector. The TEA was modified according to the alterations detailed in reference 184. The conditions are:-

Column:	15M RTX 1 DM Silicon oil, 0.25 mm internal diameter
Initial temperature:	100° C for 3 minutes
First ramp:	35°C to 165°C for 3 minutes
Second ramp:	30°C to 195°C to final temperature 195°C for 3 minutes
Carrier gas:	Helium
Flow:	2.5 ml/min
Injection temperature:	198°C
Interface temperature:	250°C - TEA
Pyrolyser temperature:	800° - TEA
Injection volume:	5µl
Specificity:	All N- nitroso compounds plus organic and inorganic nitrites, some nitrates, certain alkyl C- nitroso compounds and certain C- nitro compounds.
Sensitivity:	Typically 100 pg in 5 µl injection
Linearity:	0.04 ng /µl to 0.6 ng /µl
Precision:	1 Standard deviation ± 0.01 mins. Coefficient of variation typically 0.15%.

High performance liquid chromatography/Pendant mercury drop electrode

The HPLC/PMDE instrumentation, materials, conditions and performance data are detailed in reference 185.

Gas chromatography/Mass spectrometry

The GC/MS instrumentation, materials and conditions are detailed in reference 184 and performance data for a typical analysis of 1 ng / µl standards is as follows.

Standard	Mean retention time (minutes)	Standard deviation	Coefficient of variation (%)
Camphor	2.580	0.008500	0.3290
NG	4.112	0.019050	0.4630
1,3 DNB	4.905	0.064280	1.3100
2,4 DNT	5.709	0.072230	1.2700
DPA	6.581	0.008869	0.1350
MC	8.213	0.015760	0.1920
EC	8.672	0.008180	0.0943

Flameless atomic absorption spectrophotometry

Lead, antimony, barium, mercury and copper determinations were done using the instrumentation, materials and methods detailed in reference 138. All other elements were analysed using the standard conditions as detailed in the instrument manual.

4.2 SAMPLING

Cartridge Cases

The interior of spent cartridge cases were sampled by dry swabbing with ~ 0.25 g of acrilan fibre for FAAS analysis (dry swabbing) or by double sided adhesive tape (Scotch pressure sensitive tape) on the end of a suitable diameter perspex rod (tape lift) for SEM/EDX analysis, the tape then being transferred to a SEM sample stub for examination.

FDR/Explosives residue

Unless stated otherwise, hands were tape lift sampled (adhesive stub) for inorganic FDR using a 13 mm diameter aluminium SEM sample stub with double sided adhesive tape attached (Scotch pressure sensitive tape). Sampling of the hands and face in the outdoor firing tests in Chapter 7 employed acrilan fibre damped with isopropanol (IPA)) as per the suspect sampling kit.

Unless stated otherwise, clothing was sampled using the apparatus detailed in Figures 7.01 and 7.02 (suction sampled).

4.3 EXPERIMENTAL TESTS

4.3.1 Blank cartridges

Test 1

A total of seventy nine different blank cartridges were discharged using appropriate firearms, which were cleaned before each discharge. The inside of the spent cartridge cases were tape lift sampled and the elemental composition of the discharge residue particles from the interior of the spent cartridge cases were determined by manual SEM/EDX examination (Figure 5.01 refers).

Test 2

Starting pistol blanks (numbers 1 to 7 inclusive Figure 5.01) were discharged in a .22" (6 mm) calibre 8 shot Igi Domino 308 starter revolver. The firing hand was tape lift sampled immediately after firing 8 of each type and the samples were manually examined by SEM/EDX to determine discharge particle types present (Table 5.02 refers).

Test 3

Two lots of 6 rounds each were fired from a .38" Special calibre Smith and Wesson revolver and the firing hand was tape lift sampled immediately after firing. Six rounds of Norma, .38" Special calibre ammunition with an unjacketed lead bullet were discharged in the first test and six rounds of Norma, .38" Special +P calibre ammunition with a full metal jacket in the second test. The samples were manually examined by SEM/EDX as per the starter pistol discharge residue procedure (Table 5.03 refers).

4.3.2 Toy caps

Test 1

The caps were discharged in appropriate toy guns which were cleaned prior to use. Sampling was achieved by holding an adhesive stub in close proximity to the repeated discharge of the caps to collect airborne residue. The samples were then manually examined by SEM/EDX and the size, shape and elemental content of the particles present were noted.

Test 2

The 'devil bombers' were initially examined visually with the aid of an optical microscope before the contents were tape lift sampled for manual examination by SEM/EDX to determine elemental content.

4.3.3 Matches

Seventeen different brand names of matches were examined, eleven of which were the safety type, the others being the strike anywhere type. For each brand the matches were repeatedly ignited in close proximity to an adhesive stub surface in order to collect airborne particles, the used striking surface was sampled by contact with an adhesive stub, and used match heads were mounted on stubs. The samples were manually examined by SEM/EDX to determine elemental content and the size and shape of particles present.

4.3.4 Flares

Two types of illuminating flares were examined both of which were the hand held type. After discharge outdoors, the inside of the launch tube was cut open and the interior sampled by tape lift. The samples were manually examined by SEM/EDX to determine morphology and elemental content of the discharge particles.

4.3.5 Fireworks

Test 1

Sampling of indoor fireworks was achieved by igniting the fireworks on a pyrex plate in close proximity to adhesive stubs. When all the fireworks had been used the surface of the plate was also sampled with an adhesive stub. The samples were analysed manually by SEM/EDX to determine the morphology and elemental content of the particles present.

Test 2

The debris from a large outdoor firework display was obtained and the wooden support frames and the remains of the firework casings were sampled by contact with adhesive stubs. The samples were analysed manually by SEM/EDX to determine the morphology and elemental content of the particles present.

4.3.6 Accompanying elements

Fourteen years (1979 - 1993) SEM/EDX results for FDR casework were reviewed and statistics compiled from cases in which at least one unique FDR particle was present on the item examined, the samples originating from skin or clothing surfaces of suspects. Greater than 20,000 particles were considered (unique and indicative) - Tables 5.04 and 5.05 refer.

4.3.7 Particles from handling ammunition

Test 1

The handler washed his hands, tape lift control samples from the hands were taken, then the box was opened and the ammunition emptied into the cupped hands of the handler, who discarded it and had his hands sampled again by adhesive stubs. The samples were then analysed manually using SEM/EDX and the morphology and elemental content of the particles were noted (Table 5.07 refers).

Test 2

The Geco 9 mm LUGER ammunition was loaded into a magazine and chambered through a Browning pistol, and the Geco .38 S &W ammunition was loaded into a Smith and Wesson revolver and unloaded (both firearms had been used one week previously and then cleaned in the normal way). The handling experiment was repeated (Table 5.08 refers)

4.3.8 *Bullet weight loss on firing*

A number of bullets were pulled, cleaned, weighed, reloaded and fired into a bullet recovery box containing clean cotton waste. The bullets were recovered, cleaned and weighed. Ten bullets of each type were used (Table 5.09 refers).

4.3.9 *Effect of water on FDR*

Test 1

Three separate, 13 round firings of 9 mmP calibre RG84 NATO ammunition using a Walther pistol were done and the firing hand was swabbed with acrilan fibre (~0.25g), damped with petroleum ether (120^{0}C-160^{0}C boiling range) in the first firing, acetone/water (80/20) in the second, and acetonitrile/water (80/20) in the third. The swab samples were left for 48 hours before processing for manual SEM/EDX examination, as per the processing procedure detailed in Appendix 3 (Table 5.10 refers).

Test 2

This test involved the single handed firing of 12 rounds of Samson ammunition from a .357" Magnum calibre Colt Python revolver followed by 20 rounds of Remington ammunition from a .25" ACP calibre Baby Browning pistol. The firing hand was sampled with ~ 0.25 g of acrilan fibre damped with petroleum ether (120°C - 160°C boiling range), then the swab was placed in a beaker containing 150 cm^3 of petroleum ether (120°C - 160°C boiling range) and subjected to ultrasonic agitation for 30 minutes. The beaker was covered and allowed to sit on a vibration free bench for 4 days in order to allow any large particles to settle to the bottom of the beaker. A 5 cm^3 portion was then removed from the top of the liquid and analysed for Pb by FAAS (138).

A 25 cm^3 volume of deionised water was placed in a plastic separating funnel and 25 cm^3 of the petroleum ether from the beaker was carefully pipetted into the separating funnel, taking care not to mix the two layers. This was allowed to sit undisturbed for 2 days on a vibration free bench before both layers were analysed for Pb by FAAS. The test was done in triplicate (Table 5.11 refers).

Test 3

The final test involved the single handed firing of 12 rounds of Samson ammunition from a .357" Magnum calibre Colt Python revolver followed by 20 rounds of Remington ammunition from a .25" Auto calibre Baby Browning pistol. The firing hand was sampled with acrilan fibre (~0.25g) damped with petroleum ether (120°C -

160^0C boiling range), then the swab was placed in a beaker containing 150 cm^3 of petroleum ether (120^0C - 160^0C boiling range) and subjected to ultrasonic agitation for 30 minutes. Two 50 cm^3 aliquots were removed and processed for manual FDR examination by SEM/EDX, as per the filtration procedure given in Appendix 3. Prior to carbon coating one of the samples, the membrane filter surface, on to which the sample had been filtered, was sprayed with water and allowed to air dry. This was repeated 6 times before the sample was carbon coated.

4.3.10 Bullet fragmentation

The front and rear outside surfaces of the jacket were suction sampled and the sample was processed for manual SEM/EDX examination as per the procedure detailed in Appendix 3. The morphology and elemental content of particles detected were noted

4.3.11 RPG7 rocket launcher

A test firing was conducted on an outdoor firing range, the security forces arranging the firing of the weapon, which was done remotely using electronic devices. The launcher was placed on two sandbag walls and anchored, and a tailors dummy dressed in a new laboratory coat and strapped to a chair simulated a person firing an RPG 7. The laboratory coat was suction sampled both before and after the discharge of the PG 7 rocket, the explosive charge in the rocket having previously been removed by the security forces.

After firing the front end and outside surface of the launcher were dry swabbed with ~0.25g acrilan fibre, as was the residue from the booster propellant and the primer.

The samples were prepared for manual SEM/EDX analyses using procedures given in Appendix 3 and the size, shape, appearance and elemental content of the particles present were recorded (Tables 5.12 and 5.13 refer).

4.3.12 Discharge residue from black powder ammunition

Test 1

Six types of old ammunition thought to contain black powder were selected. One of each type was debulleted and the propellant sampled using an adhesive stub. The samples were then manually analysed by SEM/EDX to confirm the presence of black powder (Table 5.14 refers).

Test 2

Six rounds of each type of ammunition in Table 5.14 were discharged in appropriate cleaned firearms, the firing hand was adhesive sampled and the samples were analysed manually for FDR particles using SEM/EDX (Table 5.15 refers).

4.3.13 Firearms coatings

Thirty firearms were selected at random and a scalpel was used to remove part of the surface coating on each firearm, the scrapings being placed on an adhesive stub for subsequent manual SEM/EDX examination to determine the elemental content of the surface coating (Table 5.16 refers).

4.3.14 Homogeneity of propellants

Test 1

A single round from a previously unopened box of Samson IMI .223" FMJ, batch number 239 - 84, was debulleted and the propellant removed. Twenty grains were individually weighed and dissolved in 50µl ethyl acetate and analysed by GC/MS as per the method detailed in reference 186.

Test 2

Ten different types of ammunition were selected from known batch lots (details lost in the explosion at the laboratory), one round of each was debulleted and the propellant sampled. For each type 5 grains were weighed before dissolving in 400 µl of ethyl acetate. One round of each type was discharged using an appropriate firearm, into a piece of clean white cloth at a muzzle to target distance of 9". Unburnt/partially burnt grains were removed from the cloth targets and in each test, a weight approximately equivalent to the unfired propellant was taken and dissolved in 400 µl of ethyl acetate. The samples were analysed by GC/MS using the method given in reference 186 (Table 5.17 refers).

4.3.15 Bullet hole perimeters

For all the tests the targets were a 6" square piece of clean white cloth stapled on to a 7" square piece of 1/8" thick plywood. The targets were attached to a plywood target board using 2x metal bulldog clips, and the target board was nailed to an adjustable pivoted wooden frame.

Test 1

A total of 63 single shots were fired from a range of 20 feet using a Browning Hi-Power pistol and Th 56, 9 mm 2 Z ammunition. The angle of fire was varied from 'straight on' 0^0 (10 shots) to 30^0 (10 shots right and 10 shots left) and 75^0 (10 shots right and10 shots left), the target being vertical. The remaining 13 shots were fired 'straight on' as follows, 1 shot with the target vertical, 6 shots with the target tilted backwards, 45^0 off the vertical, and 6 shots with the target tilted forwards, 45^0 off the vertical.

The appearance of the bullet damage was noted i.e. size, shape and bullet wipe pattern, and the bullet hole perimeter and surrounding area was tested for the presence of lead using the sodium rhodizonate test (187).

Test 2

A series of single shot firings were done, the range of fire being contact, 3", 6", 9", 12", 18", 24", 30" and 36". The firearms and ammunition involved were a Colt

Python .357" Magnum calibre revolver using Winchester .357 Mag 158 gn JSP bullet; Blazer.38 SPL +P 150 gn FMJ bullet and Remington .38 SPL 158 gn RNL bullet. The pistol was a Star SA .22LR calibre using Eley HV .22LR HP bullet; CCI Stinger .22LR copper washed HP bullet and Winchester .22LR Super X copper washed HP bullet. The rifle was an MI Carbine using WW 30 carbine FMJ; Norma US30 JSP and RA52 FMJ.

The targets were visually examined with the aid of an optical microscope and the diameter, propellant density, blackening etc., of the muzzle blast residue noted, before testing with sodium rhodizonate for the lead deposit pattern.

Test 3

This test involved the firing of 100 single shots under identical conditions (range 20 feet), using an Uzi SMG and RG85 ⊕ 9 mm2Z, FMJ ammunition, followed by a further series of single shot firings (169 shots) using a random selection of revolvers, pistols and rifles and a random selection of ammunition, encompassing unjacketed lead bullets, semi - jacketed lead bullets, copper washed lead bullets and full metal jacketed bullets. All bullet hole perimeters were tested for the presence of lead using the sodium rhodizonate test.

Test 4

A series of non - close range single shot firings were done at right angles to the target and from a range of 20 feet. The firearms were cleaned before use and three shots fired using the test ammunition, prior to shooting the target, to ensure no carry over of residue from ammunition previously used in the gun. All ammunition was wiped with a new dry paper tissue before use. The target cloth was analysed for Pb, Sb, Ba, Cu, Ni, and Hg by FAAS prior to use.

A 1" square piece of cloth containing the bullet hole was cut out and immersed in 10cm^3 20% v/v hydrochloric acid (MIBK saturated) overnight before analysis by FAAS. Primer and bullet jacket compositions were confirmed by manual SEM/EDX examination (Tables 5.18 and 5.19 refer).

4.3.16 Persistence

Test 1

A new laboratory coat was worn whilst 20 rounds of ammunition were fired from a .25" ACP calibre Browning pistol. The coat was promptly removed and sealed in a 500G plastic bag and stored, in an office, undisturbed for two years. The coat was then suction sampled and the sample processed for FDR examination using automated SEM/EDX (146).

Test 2

Twenty five rounds of ammunition were fired in each of three different calibre pistols. The firing hand was adhesive stub sampled immediately after each firing. The firings were repeated, but the firer was allowed to wipe his hands on clean dry paper kitchen roll for two minutes, in an effort to remove any FDR particles, prior to sampling.

The samples were then examined by automated SEM/EDX for the presence of FDR.

4.3.17 Antimony free primers

Test 1

Three firearms complete with magazines were dismantled in a clean environment, washed with water followed by acetone and then subjected to ultrasonic agitation for 30 minutes in 2% v/v hydrochloric acid. They were then rinsed with water, followed by acetone and allowed to air dry. During reassembly with light oiling, the parts of each firearm were sampled for FDR using a single dry ~ 0.25g acrilan swab. (The firearms used were all destined for disposal). Suitable ammunition for the firearms was selected and each round individually wiped with an x-ray medi-wipe tissue prior to each calibre being collectively sampled for FDR using a dry acrilan swab. Elaborate precautions were taken to ensure the cleanliness of the firearms and ammunition and after cleaning they were individually sealed in nylon bags prior to use. The ammunition selected was known, from previous experience, to have antimony free primers, and to have antimony in the bullet lead. Using SEM/EDX this was confirmed by dismantling a single round of each and analysing all the components for the presence of antimony. The six control swabs were analysed for FDR using automated SEM/EDX.

An outdoor range was used for the firing tests to avoid the possibility of contamination from airborne particles in an indoor range. Twenty rounds were fired from each firearm, the firer wearing a new disposable boiler suit and cleaning hands for each test. Using adhesive stubs each hand was sampled, both before and after firing. The samples were analysed for FDR using manual SEM/EDX and 50 to 60 particles were analysed in each test to give a random selection (Table 5.20 refers).

4.3.18 Analysis of a baton round

Test 1

A baton round was dismantled and the component parts analysed by manual SEM/EDX. The baton round was headstamped 12/77 FPL.

Test 2

Six rounds, headstamped 12/77 FPL, were fired in a Federal Riot Gun, followed by adhesive stub sampling of both hands and upper outer garment of the firer. The samples were analysed using manual SEM/EDX.

Test 3

A round of 7.62 mm x39 mm calibre Yugoslavian nny 82 ammunition was dismantled and all the components analysed using manual SEM/EDX. Organic constituents of the propellant were analysed using GC/MS (185).

4.3.19 Propellant analysis

Suspected propellant particles detected in casework were analysed by IR microspectrophotometry (FTIR) and GC/MS as per the method detailed in reference 186. Only samples in which nitrocellulose was detected by FTIR were submitted for GC/MS analysis (Tables 5.21 and 5.22 refer).

4.3.20 Frequency of occurrence of particles containing mercury

A series of indoor firings were conducted involving ammunition from seven different batch lots, six of which were known to contain mercury and the other a modern primer for comparison purposes. Two firearms were involved, a pistol and a revolver, and for each firing the gun was cleaned, loaded with six rounds of ammunition, the firers hands were washed and a control sample taken from the hand before the gun was fired single handed. The firing hand was sampled immediately using adhesive stub and the samples were examined by manual SEM/EDX. In most instances approximately 300 discharge particles per firing were examined. Firings number 1 to 3 were conducted using a .455" calibre Webley Mk VI revolver and firings number 4 to 7 using a 9 mmP calibre Browning Hi-Power pistol (Table 5.25 refers).

4.3.21 Distribution of mercury after discharge

Test 1

An unopened 100 round batch of mercury containing ammunition was obtained for testing purposes, namely 9 mmP calibre B↑E43. This ammunition was used for all the mercury distribution tests. Fifteen rounds were analysed for total mercury content by pulling the bullet and then dissolving the complete round, including the bullet in ~ 100 cm^3 of 50% $^{v}/_{v}$ nitric acid. After dissolution the samples were diluted to 500 cm^3 and analysed for Hg using the cold-vapour method (138). All quantitative mercury determinations were done by this method (Table 5.28 refers).

Test 2

Fifteen rounds were discharged in a 9 mmP calibre Colt pistol, which was destined for disposal, and the spent cartridge cases were individually dissolved in ~100 cm^3 of 50 $^{v}/_{v}$ nitric acid, diluted to 500 cm^3 and analysed for mercury (Table 5.29 refers).

Test 3

A wad of ten 15 cm diameter filter papers were attached to clean target boards and a shot fired through the wad from a range of approximately 12 feet. The portion containing the bullet hole was cut out from each of the filter papers, bulked, extracted into 50 cm^3 of 10 $^{v}/_{v}$ nitric acid and 1 cm^3 aliquots analysed for mercury. Ten single shot firings were done (Table 5.30 refers).

Test 4

A series of 10 x single shot firings were conducted into previously unused thermoplastic bullet recovery medium, a wad of filter paper acting as an intermediate target. The bullets were recovered, dissolved in ~70 cm^3 50 $^{v}/_{v}$ nitric acid, diluted to

500 cm^3 and analysed for mercury. The recovered bullets were very dirty, with a black deposit ingrained into the surface of the jacket (Table 5.31 refers)

Test 5

The pistol and magazine was dismantled, cleaned in 2% $^v/_v$ nitric acid and then acetone to remove the blueing and all traces from previous firings. The pistol was reassembled, very lightly oiled and used to test fire. A single shot was fired and the pistol and magazine immersed in 1 litre of 2% $^v/_v$ nitric acid for 1 hour, with frequent agitation, before an aliquot was removed for mercury analysis. The process was repeated a further three times (Table 5.32 refers).

Test 6

A 'firing tube' was made as illustrated in Figure 4.01 (destroyed in explosion at laboratory). Two sampling ports in the tube enable sampling of discharge residue. The test involved the sampling of discharge residue from the primer. A round of ammunition was debulleted and the propellant removed. The ends of the tube were sealed with rubber bungs, one of the bungs having a hole through it to accommodate the empty cartridge case with live primer and to hold it tightly in position. A one micron pore size membrane filter, a series of three liquid traps, each containing 50 cm^3 of 70% $^v/_v$ nitric acid, and a recycling pump (18 l/min capacity) were used to sample the primer discharge residue. Three separate primer firings were done by discharging the primer using a punch and hammer, the pump operating before discharge and for 30 minutes after discharge.

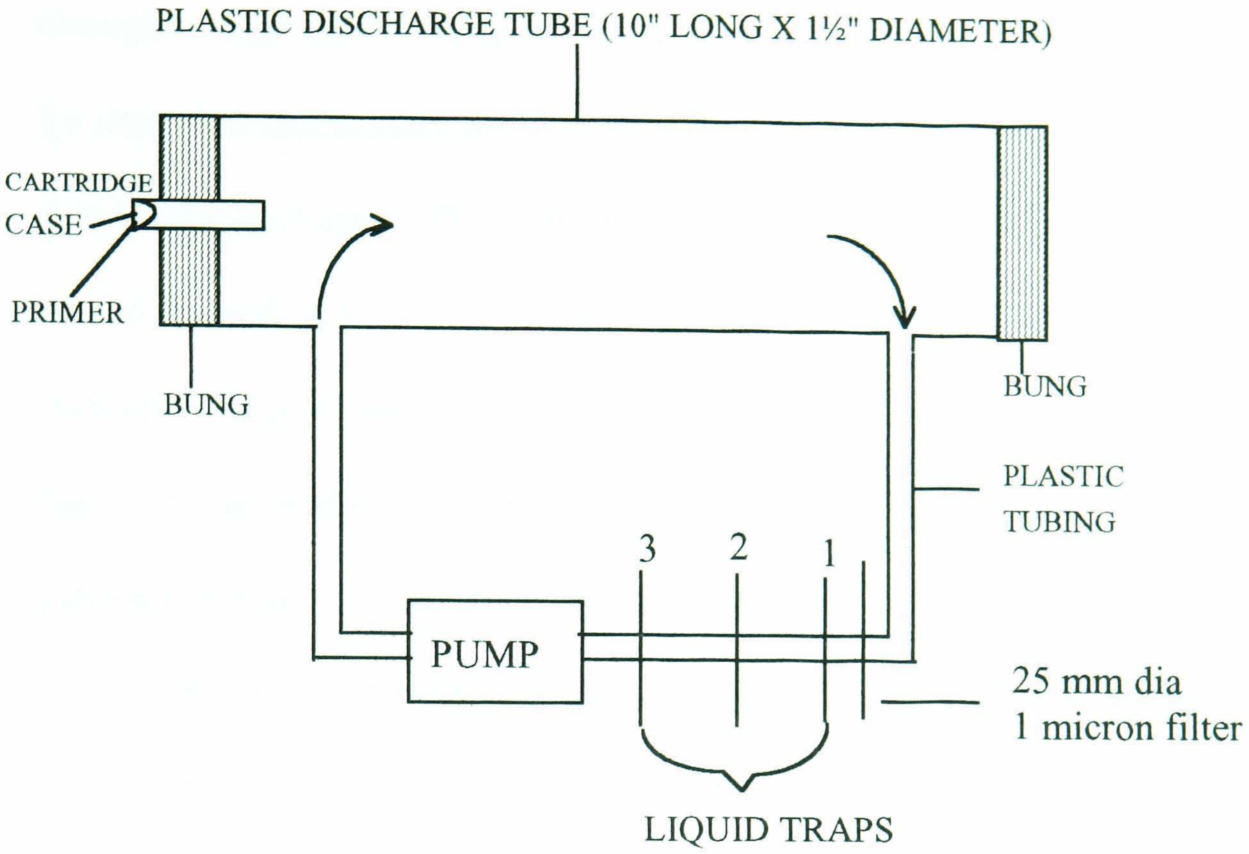
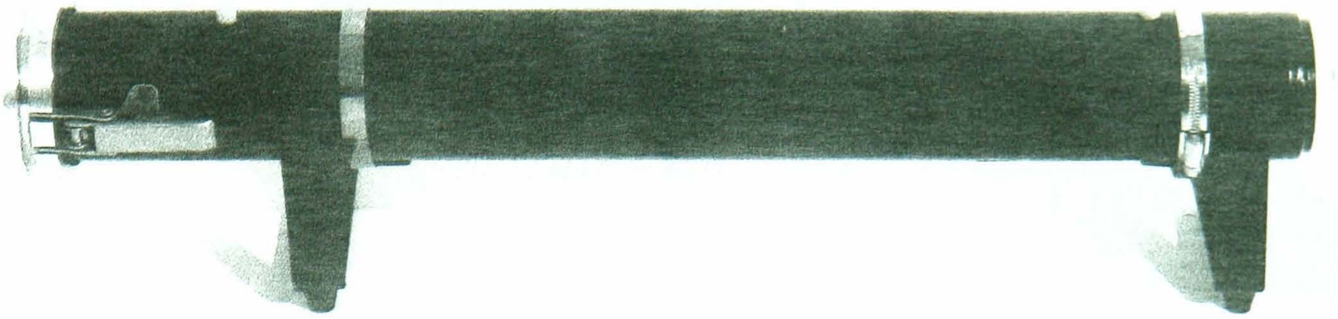


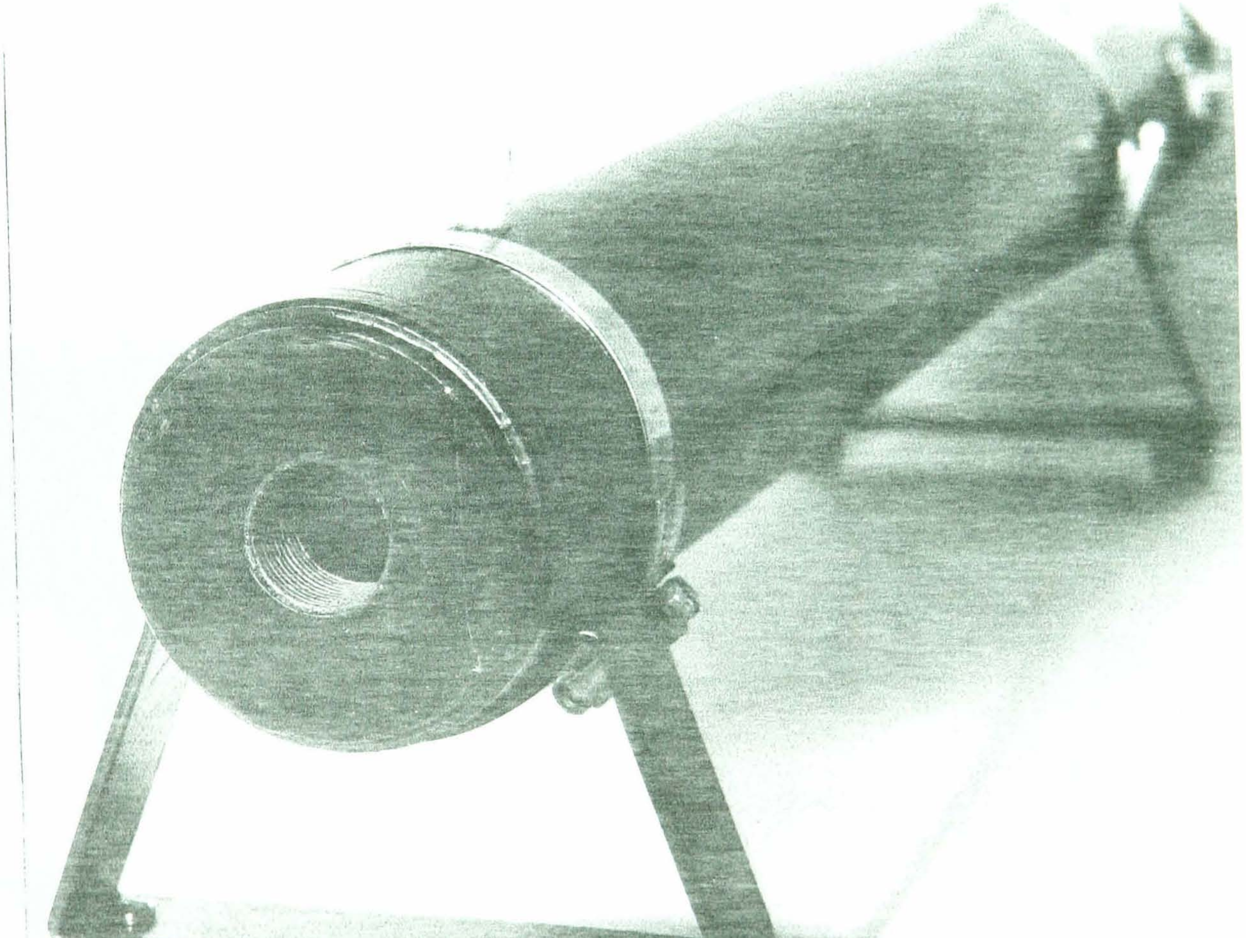
Figure 4.01 Primer discharge sampling system

The membrane filter, the liquid traps and the spent cartridge case were analysed for mercury. The filter was immersed in 50cm^3 of 50 % $^v/_v$ nitric acid for 2 hours before aliquots were analysed, the liquid traps were analysed directly and the spent cartridge case was dissolved in ~ 100cm^3 of 50 %$^v/_v$ nitric acid and diluted to 500 cm^3 before aliquots were taken (Table 5.33 refers).

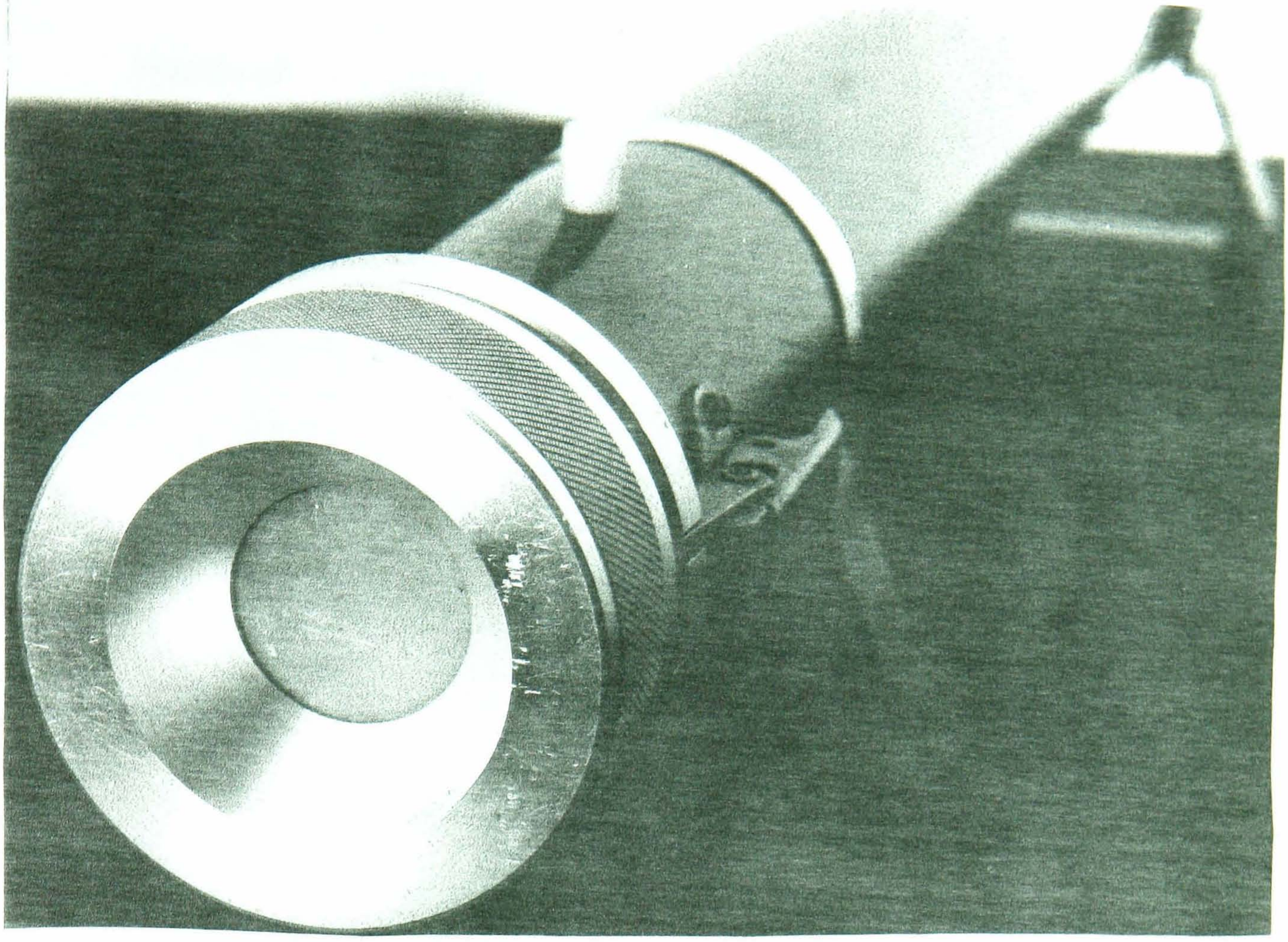
Test 7

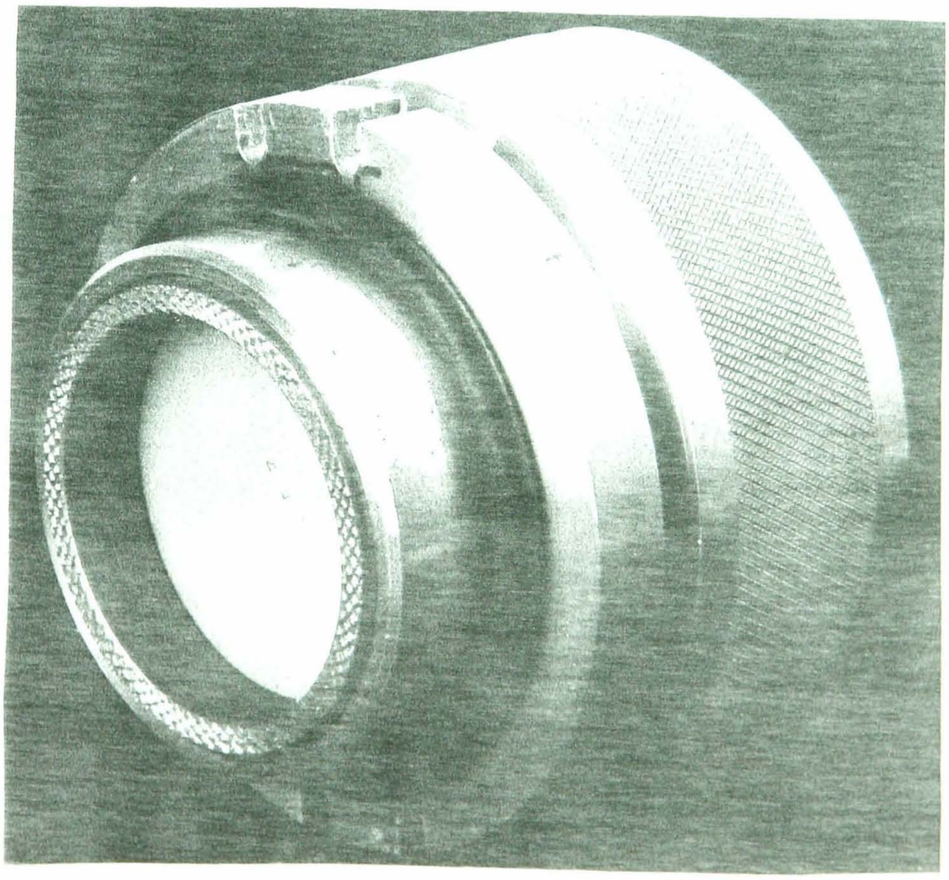
A metal firing tube was made as illustrated in Figure 4.02. For this apparatus a 12 bore calibre shotgun was modified in such a way that it acted as a trigger/firing pin mechanism to discharge a, manually loaded, single round of 9 mmP calibre ammunition through a 9 mm calibre barrel obtained from another pistol. The spent cartridge case remains in the chamber after discharge. The bullet exits the tube through a large rubber bung, the bullet damage to which is effectively self sealing. To determine the amount of mercury exiting from the muzzle, three separate single shot firings were done. The 'airborne' discharge residue exiting from the muzzle was passed through a 1 micron membrane filter followed by three in-line liquid traps each containing 50 cm^3 of 70 % $^{v}/_{v}$ nitric acid. The membrane filter retains particles that are detectable by SEM and the liquid traps retain the sub-micron particles/vapour. [It is not practical to routinely examine particles less than 1 micron in size in the SEM. Consequently, the sample concentration-clean up procedure (149) involves the use of a final filter of 1 micron pore size on to which the particles of interest are deposited].






MODEL 1908
HARRINGTON & RICHARDSON ARMS CO
WORCESTER, MASS. U.S.A.





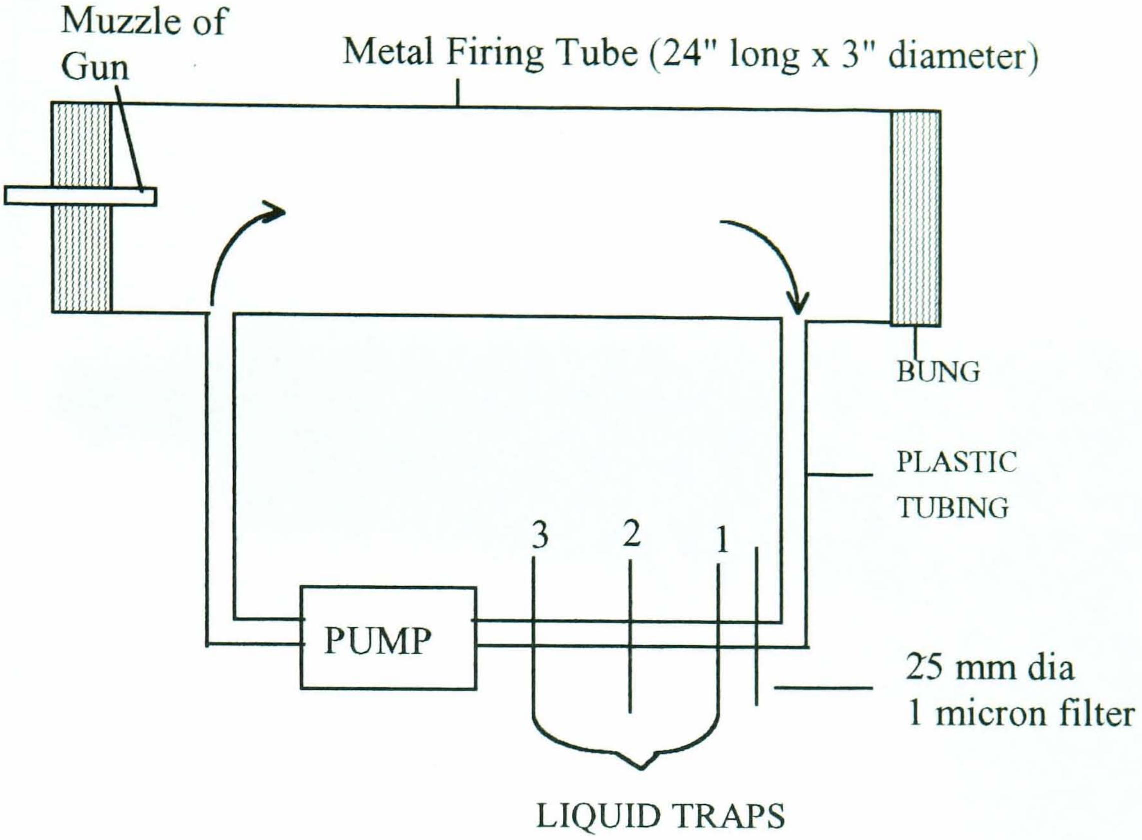


Figure 4.02 Muzzle discharge sampling system

The pump (18 l / min capacity) was operating before discharge and for 30 minutes after discharge. The filter and liquid traps were analysed for mercury as were rinsings from the firing tube. Aliquots from the liquid traps and rinsings were analysed directly and the filter was extracted as in the previous test (Table 5.34 refers).

Test 8

A 14" square perspex box (destroyed in explosion at laboratory) was constructed and the gun was fired using a string pull. The apparatus is illustrated in Figure 4.03. This test required a $1^1/_2$ " extension to be welded on to the barrel to enable the muzzle to be inserted through the rubber bung.

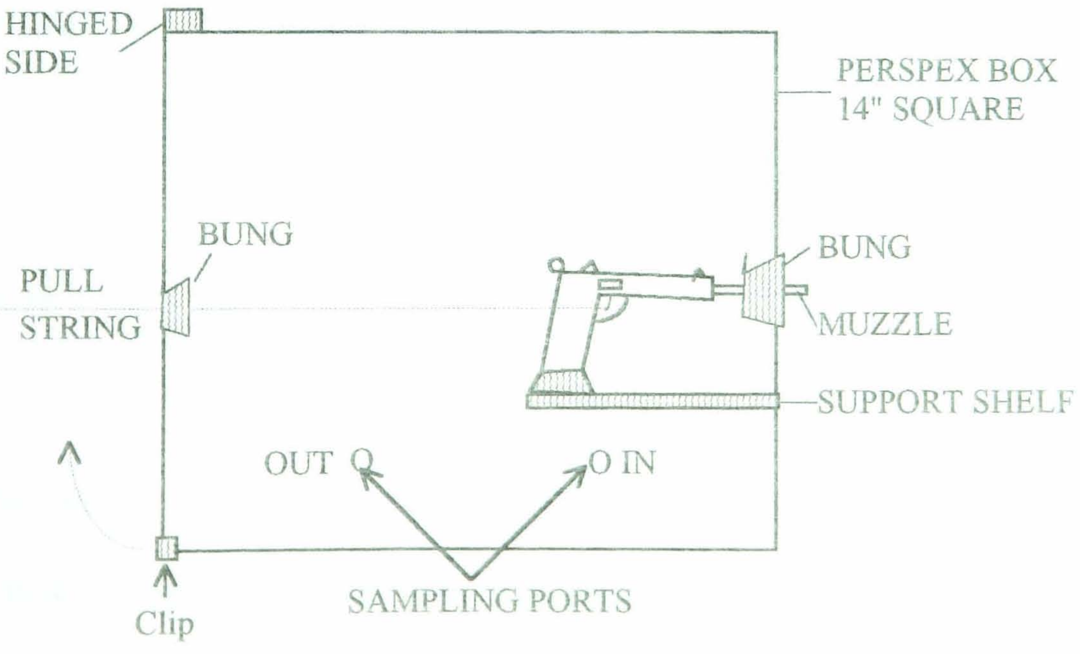


Figure 4.03 Sampling box for breech residue

A recycling pump (18 l / min capacity), an initial in line 25 mm diameter, 1 micron pore size membrane filter, followed by three in line liquid traps, each containing 50 cm^3 70%$^{v}/_{v}$ nitric acid, were used to sample the discharge residue. The pump was on prior to discharge and for 30 minutes after discharge. After suction sampling, the gun and spent cartridge case were removed and the inside surfaces of the box were swabbed four times with acrilan fibre damped with 70%$^{v}/_{v}$ nitric acid. The swabs were bulked, extracted with 50%$^{v}/_{v}$ nitric acid, made up to 500 cm^3 and analysed for mercury along with the filter and liquid traps. The test was done in triplicate (Table 5.35 refers).

Test 9

A series of 1, 3 and 7 round indoor firings were done, using the pistol in Figure 4.03 , with the firer wearing a clean laboratory coat and firing single handed (R) for each test. The tests were done in duplicate and controls were taken from the hands and coat prior to discharge. Sampling was achieved by swabbing the hands, face and head hair, as per the suspect sampling kit, and suction sampling the upper front surface of the coat (Table 5.38 refers).

Test 10

Seven rounds were fired using the ammunition and pistol involved in the mercury distribution tests. The firing hand was adhesive stub sampled and the particles were examined by manual SEM/EDX.

4.3.22 Loss of mercury from spent cartridge cases

Test 1

A quantity of .303" calibre RG7/54 ammunition was used, all of which was boxed and had the same batch lot number. Thirty rounds were analysed for mercury and a total of 120 rounds were fired. Thirty of the spent cartridge cases were dissolved in 50% $^{v}/_{v}$ nitric acid promptly after firing, made up to a known volume, and analysed for mercury. The remaining spent cartridge cases were stored in a well ventilated, non-heated area (gas cylinder store) and thirty were analysed for mercury after 3 days, another thirty after 10 days and the remainder after 69 days had lapsed. (Table 5.39 refers).

Test 2

Thirty rounds of the ammunition, as in Test 1, was discharged and the spent cartridge cases were stored in a fan assisted laboratory oven at 80^{0}C for three days before being analysed for mercury as in Test 1.

4.3.23 Lead free ammunition

Test 1

A round of .38" Special calibre, Smith and Wesson Nyclad ammunition was debulleted and the propellant removed. The primer was discharged and the bullet and spent cartridge case/primer cup were cut open. All components were analysed using manual SEM/EDX.

Test 2

A round of Geco .38" Special calibre ammunition with a metal piercing bullet, the base of which was enclosed, and a round of Geco .357" Magnum calibre ammunition with a metal piercing bullet, the base of which was exposed, were dismantled and the primer dissolved in 30%$^{v}/_{v}$ nitric acid and diluted to 500cm^3 , prior to analysis for Pb, Sb and Ba by FAAS (Table 5.40 refers).

Test 3

Lead free ammunition was debulleted, the propellant removed, the inside of the cartridge case was wiped with a tissue, and the cartridge case filled with ethyl acetate and allowed to sit for one hour, to extract the organic components of the primer. The primer extract and the propellant was analysed by GC/MS as per the method detailed in reference 185. Other rounds were dismantled, the primer discharged, and all the components analysed using manual SEM/EDX (Table 5.41 refers).

Test 4

Ten rounds of each type were discharged in appropriate firearms and the hands were sampled before and after firing, using adhesive stubs. The firearms involved were a 9 mmP calibre Walther pistol, a .357" Magnum calibre Ruger revolver and a .223" SA80 rifle, all of which were cleaned prior to use and 5 rounds of each ammunition fired through the firearm prior to the test firing. The samples were examined by manual SEM/EDX (Table 5.42 refers).

4.3.24 Suspect processing procedures

All suspect processing procedures were reviewed and all suspect processing premises in Northern Ireland were inspected. This took about 10 weeks to complete and involved discussions with cell block staff, custody officers, CID, SOCO, uniformed police and special police units involved in arrests. Suspect custody records were examined to determine typical time delays and reasons for delays.

As many details, views and opinions as possible were gathered about the processing of suspects from apprehension through to sampling for forensic evidence. Suspect processing premises were then inspected and recommendations made for improvements.

An important practical innovation resulting from the review is the introduction of an evidence protection kit, the development of which is now described.

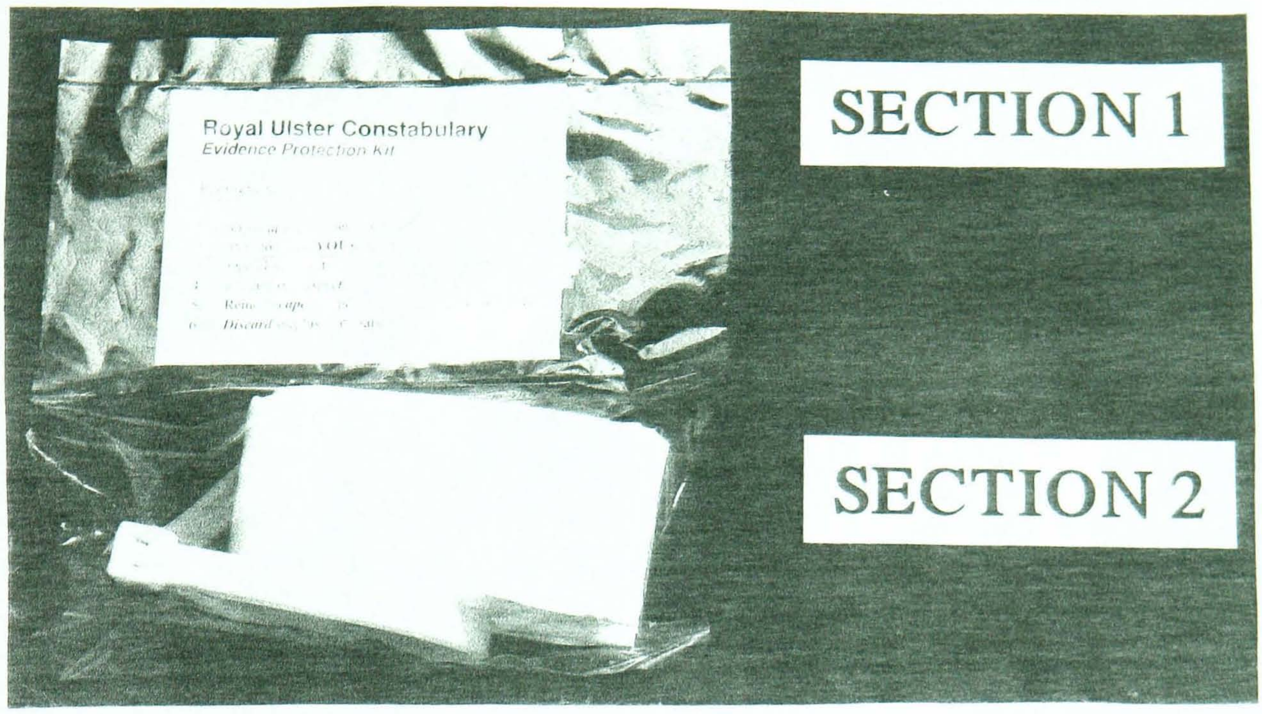
Design considerations

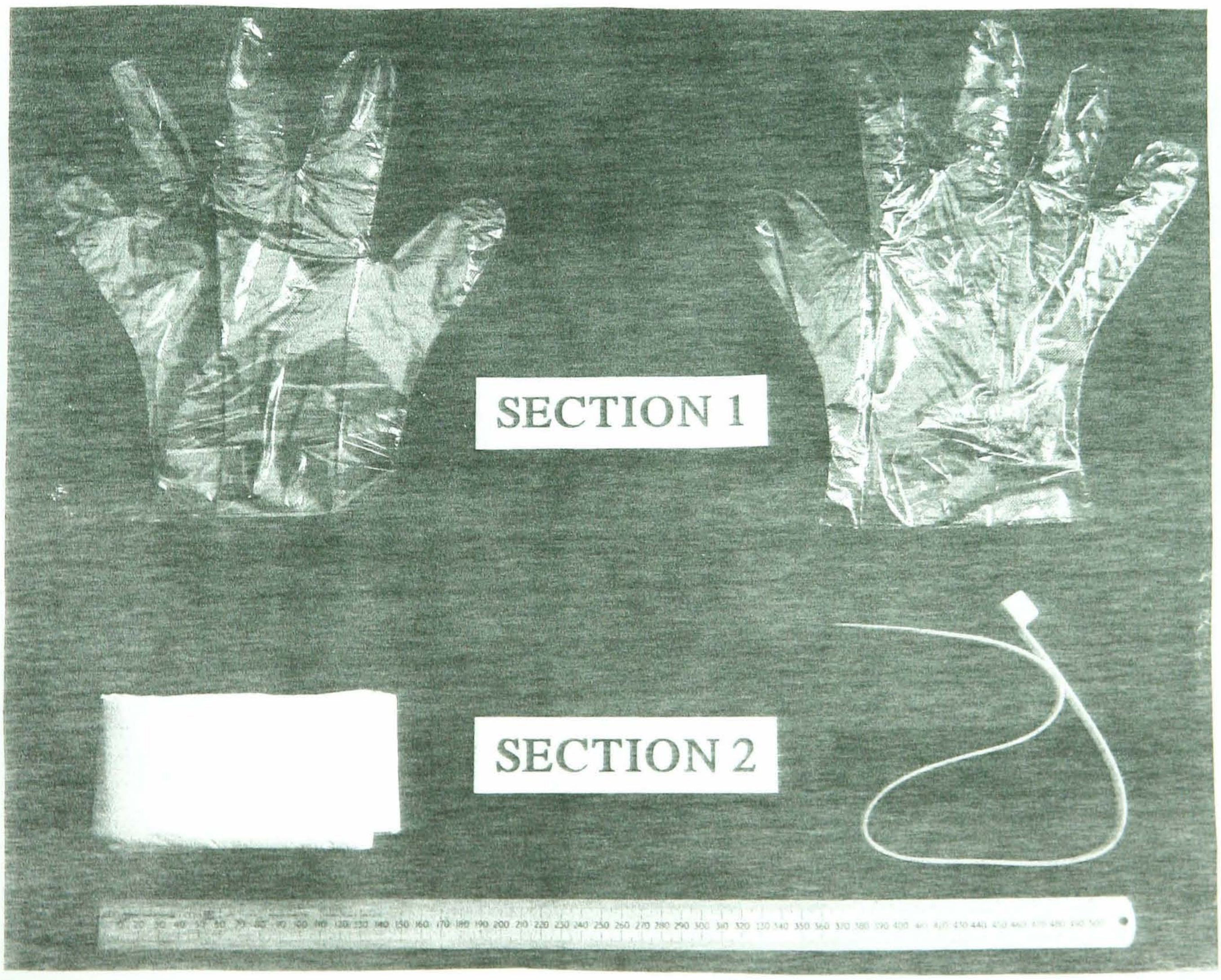
Many things had to be considered during the design stage including: time taken to apply kit; ease of preparation; cost; contamination avoidance during preparation and use; simplicity in use; efficiency of restraining strap; effect of using the kit on a range of evidence types e.g., hairs, fibres, glass, paint, body fluids, firearms and explosives residues; properties of materials used; size of kit; the value of using the kit versus the cost, time and effort in preparation and use; storage and issue of kits; legal implications; and quality assurance.

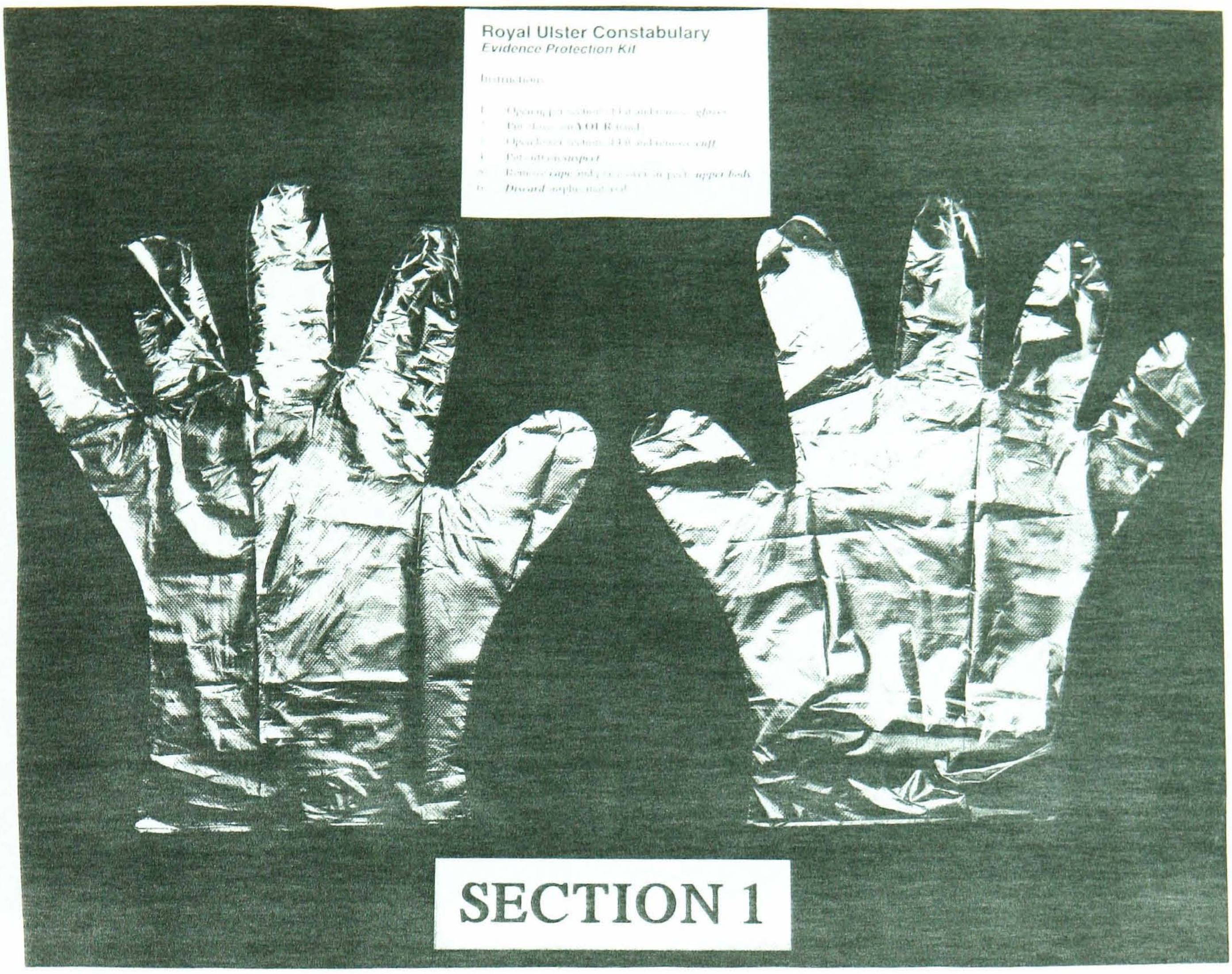
Evidence protection kit in use

The kit consists of a nylon bag made into two sections by the use of a heat sealer, the upper section containing a pair of large disposable polythene gloves and an instruction sheet, and the lower section containing a disposable plastic restraining strap (cuff) and an evidence protection cape. The instructions are in large print to enable them to be read in poor light conditions.

In use, the police officer opens the first section and puts on the polythene gloves. The second section is opened and the plastic cuff is used to secure the suspects wrists behind their back. The cape is then placed on the suspect. The use of the kit is illustrated in Figure 4.04.


Royal Ulster Constabulary
Evidence Protection Kit
SECTION 1
SECTION 2


SECTION 1
SECTION 2


Royal Ulster Constabulary
Evidence Protection Kit
SECTION 1


SECTION 2

Figure 4.04 Photographs of evidence protection kit

The cape has an elasticated waist and neck and a hood for the protection of the head hair. It is white in colour so that the suspect can be easily seen in poor light conditions, and the material is durable and 'breathable' with a reasonable degree of water resistance. The cape also covers the suspect's hands and is big enough to cover a large suspect wearing outdoor clothing. Another important advantage is that it protects the suspect's hands and upper clothing from the possibility of cross-contamination during the period between arrest and sampling.

When a suspect's clothing is seized for forensic examination, the cape is removed first and separately packaged. It is then submitted to the laboratory along with the clothing.

Some form of evidence protection garment that would cover the lower body as well (resembling a boiler suit/overalls) was also considered but was dismissed on the grounds that it would not be a practical proposition. If a suspect struggled, the time, effort and extra manpower required to force them into such a garment would almost certainly result in the loss of forensic evidence as well as increasing the risk of contamination.

Under certain circumstances it is essential that the arrest is made in an efficient manner, and as quickly as possible. Ease and speed of use are thus very important factors and the new kit is simpler and faster to use than the previous hand bagging kit. Nylon bags are used in preference to conventional plastic bags because nylon is approximately three times less permeable than plastic. Where this is not a major consideration, plastic bags would be adequate. Nylon suffers from the disadvantage of making a noise when handled or disturbed.

Possible improvements to the kit would be a suitable alternative for nylon, and transparent material for the cape to enable the suspect's hands to be visible, although the plastic restraining strap has been found, by police, to be effective. Despite the fact that the cuff is effective, a minority of police officers would prefer to be able to see the suspect's hands at all times.

The use of a plastic restraining strap rather than conventional non-disposable metal handcuffs has several advantages. Everything necessary for the arrest is contained in the kit and is known to be in a clean condition. The plastic restraining strap is disposable, thereby avoiding the possibility of any form of contamination being transferred from suspect to suspect; a possibility that exists with reusable handcuffs.

Bulk storage of kits is inside large nylon bags to prevent the possibility of the outside of the kit bags becoming contaminated, and strict procedures are in force to avoid the possibility of any form of contamination during preparation of the kits. The kit making premises are regularly checked for contamination and a random 2% sample of freshly prepared kits are submitted to the laboratory for examination. All kits have a batch number so that if a problem did arise the relevant batch could be withdrawn.

4.3.25 Sampling of clothing

Test 1

Two new laboratory coats were worn in separate firings, each involving the discharge of three rounds of Winchester .357 Magnum calibre ammunition in a Colt Python revolver. After firing each coat was removed, placed in a nylon bag and sealed. One of the coats was suction sampled three times in succession using the apparatus illustrated in Figure 7.01 and the other coat was swabbed three times in succession using cotton wool damped with acetone, as per the existing explosive residue sampling procedure. The membrane filters and swabs were individually extracted into acetone and analysed for NG using GC/TEA (Table 7.01 refers).

Test 2

A test was conducted involving the firing tube as previously described in Figure 4.02. Ammunition containing NG in the propellant, namely 9 mmP calibre RG 88 ⊕ , was discharged and the discharge residue sucked through in-line membrane filters of various pore sizes, housed in 25 mm diameter Deldrin filter holders, the suction rate being 108 l/ min. The apparatus is shown schematically in Figure 4.05.

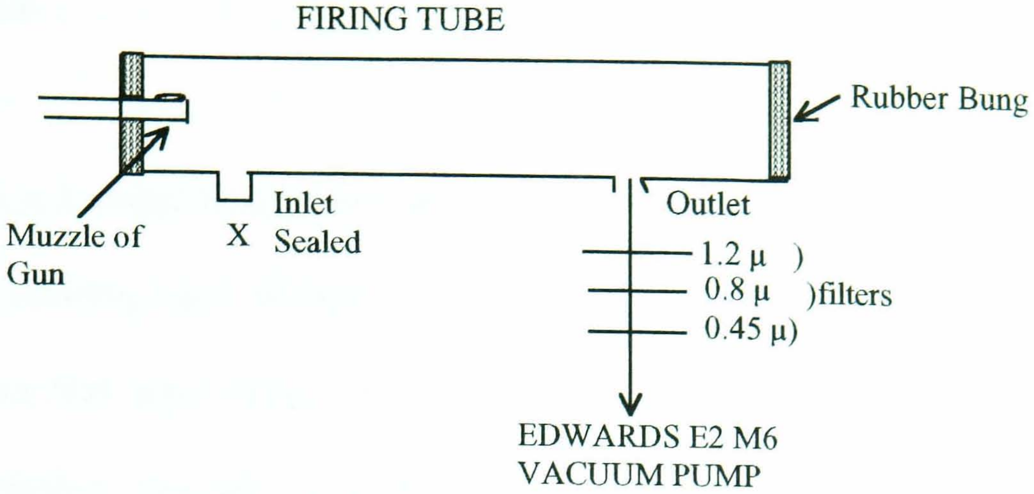


Figure 4.05 Apparatus for sampling discharge residue

Four x 1 shot firings were done using a combination of different in line pore size membrane filters which were extracted with acetone and analysed for NG using GC/TEA and confirmed by HPLC/PMDE (Table 7.02 refers).

Test 3

New laboratory coats were used throughout and control samples were taken from each coat prior to firing by suction sampling using a .45 micron pore size filter in a 25 mm diameter Deldrin holder. Four x 1 shot and four x 6 shot firings were done, using a Browning Hi-Power pistol and the ammunition as in Test 2, the coat being sealed in a nylon bag immediately after each firing. The coats were suction sampled using the combination of in-line pore size membranes and pump as in the previous experiment and using the suction sampling device as in Figure 7.01.

The membrane filters were individually extracted using acetone and analysed for NG by GC/TEA (Tables 7.03 and 7.04 refer).

Test 4

Four x 6 round firings were done using the gun and ammunition as in Test 3, the firer wearing a new laboratory coat each time. The coats were suction sampled, both before and after firing, using the revised sampling device Figure 7.02 and the membranes were extracted with acetone and analysed for NG using GC/TEA (Table 7.05 refers).

Test 5

Two jackets and two pullovers were washed, dried and worn in four firing tests, each involving the discharge of 12 rounds of Kynoch .38 S&W calibre ammunition in a revolver, the firer wearing one of the garments in each test firing. Before firing the garments were swabbed with cotton wool damped with acetone, as per the explosive residue sampling system, followed by suction sampling using the revised suction sampling device. The swabs and membrane filters were extracted with acetone and analysed for NG by GC/TEA and the filters were processed as in Appendix 3 and examined for FDR using automated SEM/EDX (Table 7.06 refers).

4.3.26 Suspect sampling kit

The kit is contained within a single nylon bag which has been divided into three compartments by the use of a heat sealer (Figure 4.06). The first part contains instructions on how to use the kit, an X-ray wipe (a large pre-wetted towelette in a foil sachet) and a ball-point pen (Figure 4.07). The second part contains a foil sachet) and a ball-point pen (Figure 4.07). The second part contains a disposable overall and a pair of large disposable polythene gloves, to be worn by the sampler (Figure 4.08). The third part contains the sampling materials in a rigid plastic container fitted with a lid that has a tamper proof seal, a spare lid for the container (to reseal after use), a nylon bag, two integrity labels, a police exhibit label, an X-ray wipe and an incident report form (Figure 4.09). The sampling materials are shown in detail in Figure 4.10.

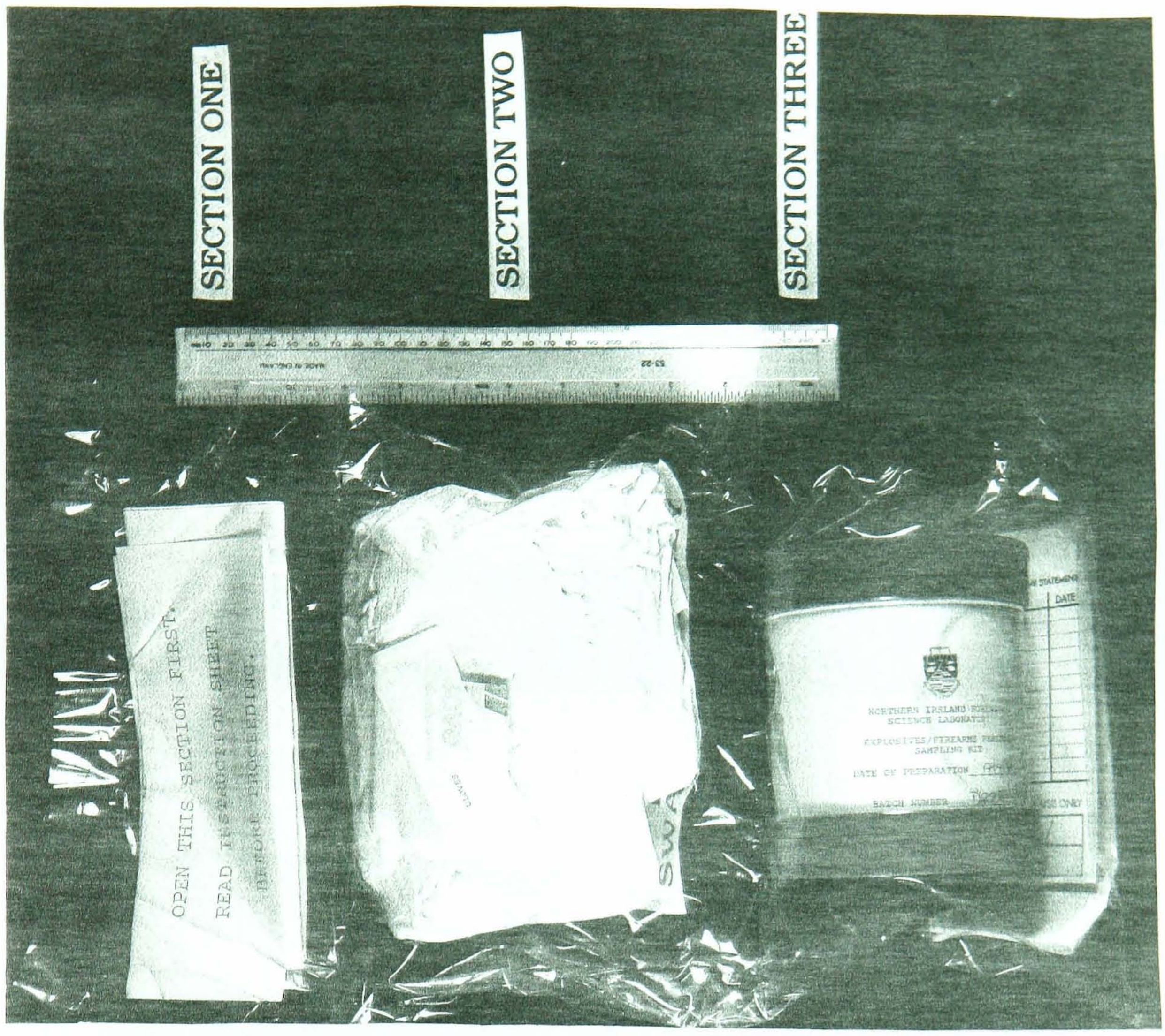
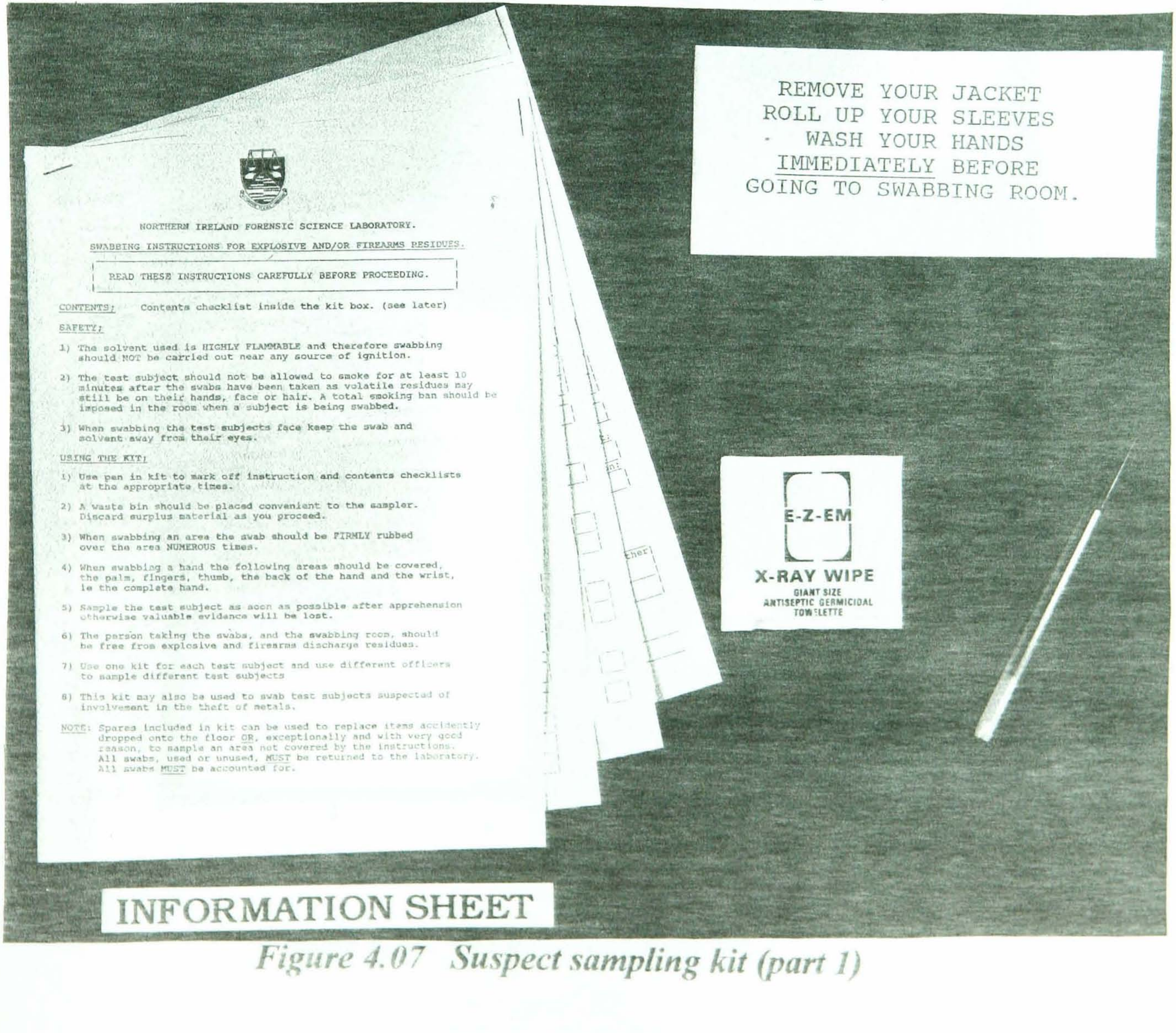


Figure 4.06 Suspect sampling kit (complete)



NORTHERN IRELAND FORENSIC SCIENCE LABORATORY.

SWABBING INSTRUCTIONS FOR EXPLOSIVE AND/OR FIREARMS RESIDUES.

READ THESE INSTRUCTIONS CAREFULLY BEFORE PROCEEDING.

CONTENTS; Contents checklist inside the kit box. (see later)

SAFETY;

1) The solvent used is HIGHLY FLAMMABLE and therefore swabbing should NOT be carried out near any source of ignition.

2) The test subject should not be allowed to smoke for at least 10 minutes after the swabs have been taken as volatile residues may still be on their hands, face or hair. A total smoking ban should be imposed in the room when a subject is being swabbed.

3) When swabbing the test subjects face keep the swab and solvent away from their eyes.

USING THE KIT;

1) Use pen in kit to mark off instruction and contents checklists at the appropriate times.

2) A waste bin should be placed convenient to the sampler. Discard surplus material as you proceed.

3) When swabbing an area the swab should be FIRMLY rubbed over the area NUMEROUS times.

4) When swabbing a hand the following areas should be covered, the palm, fingers, thumb, the back of the hand and the wrist, ie the complete hand.

5) Sample the test subject as soon as possible after apprehension otherwise valuable evidence will be lost.

6) The person taking the swabs, and the swabbing room, should be free from explosive and firearms discharge residues.

7) Use one kit for each test subject and use different officers to sample different test subjects

8) This kit may also be used to swab test subjects suspected of involvement in the theft of metals.

NOTE: Spares included in kit can be used to replace items accidently dropped onto the floor OR, exceptionally and with very good reason, to sample an area not covered by the instructions. All swabs, used or unused, MUST be returned to the laboratory. All swabs MUST be accounted for.

REMOVE YOUR JACKET
ROLL UP YOUR SLEEVES
WASH YOUR HANDS
IMMEDIATELY BEFORE
GOING TO SWABBING ROOM.

Figure 4.07 Suspect sampling kit (part 1)

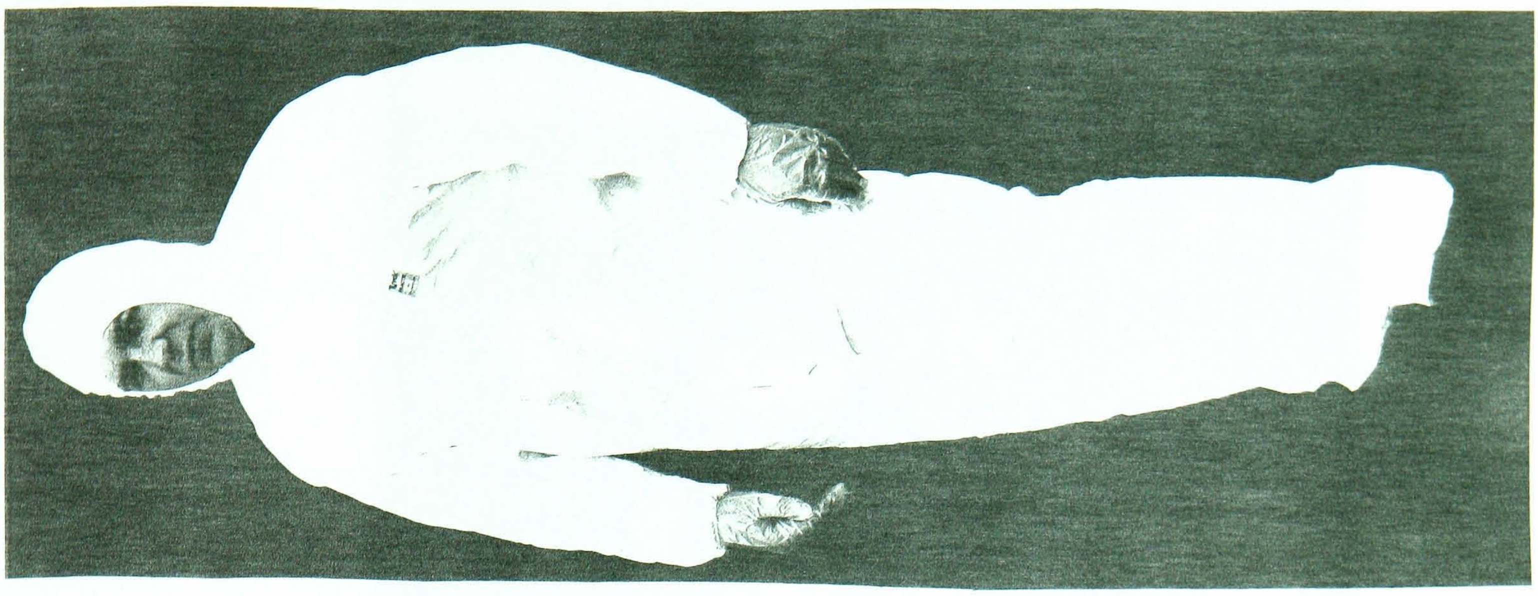


Figure 4.08 Suspect sampling kit (part 2)

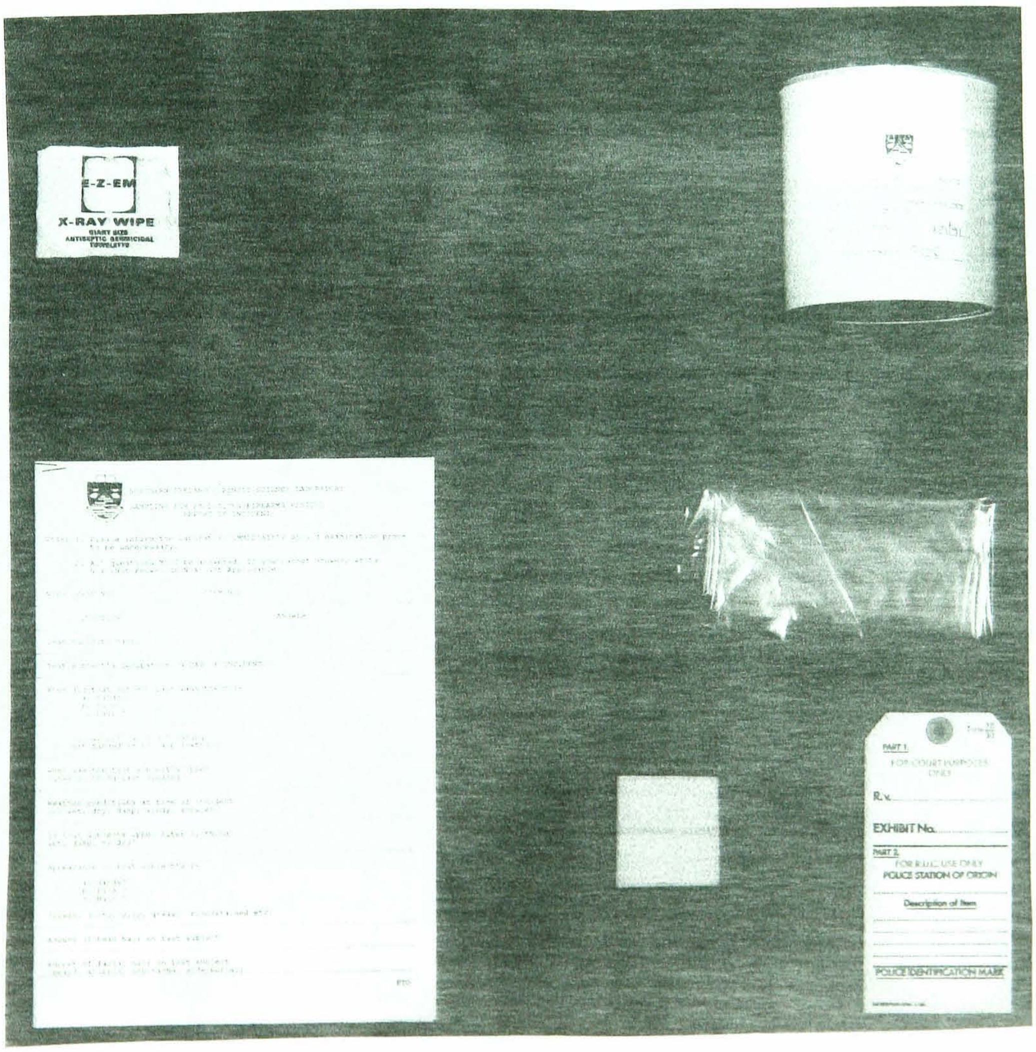


Figure 4.09 Suspect sampling kit (part 3)

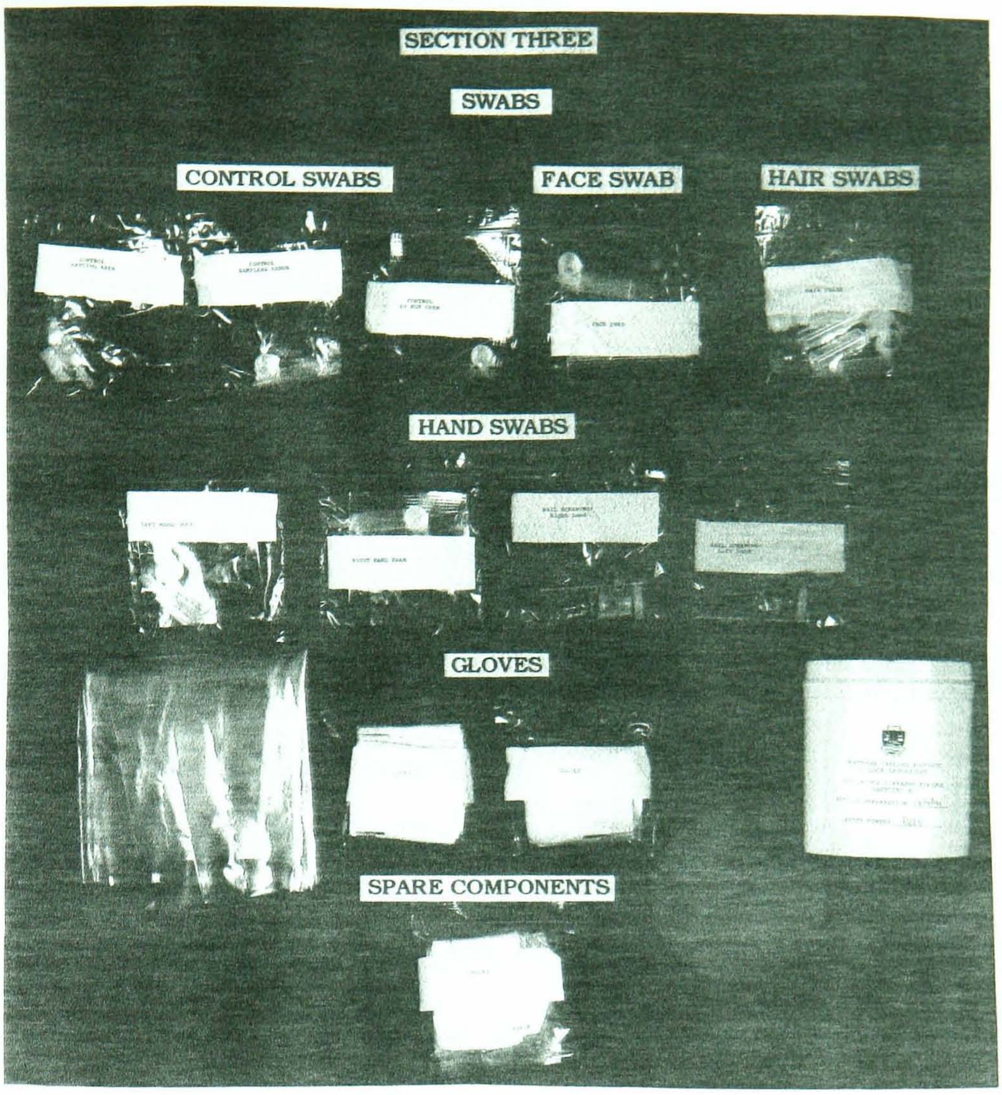


Figure 4.10 Suspect sampling kit materials

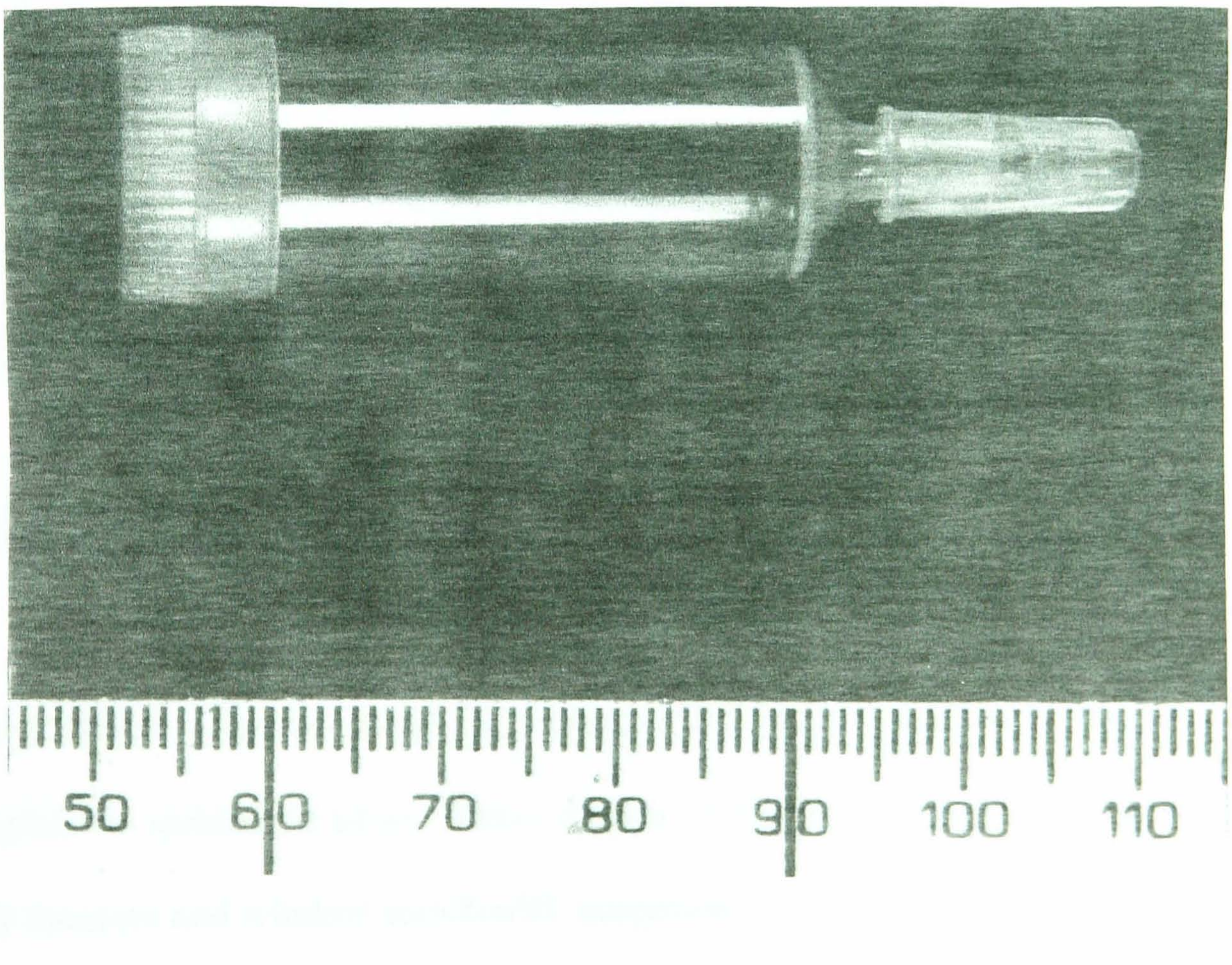


Figure 4.11 Plastic tube used in kits

The kit contains comprehensive instructions. The sampler and the swabbing room must be free from explosives and firearms discharge residues. The sampler's gloved hands and the work surface are swabbed to provide control samples of the sampler and the sampling environment.

The suspect's hands and nails, the face and finally the head hair are then sampled. The sampling material is a piece of acrilan fibre, ~0.25 g, damped with 1.5 cm^3 analar grade isopropanol and contained in a sealed polythene-lined metal foil sachet. A sample is taken by firmly rubbing the area numerous times. It is then placed inside a plastic tube, sealed with a cap and put into a labelled self-seal bag. The plastic tube (Figure 4.11) and the acrilan fibre are used because they are an integral part of the subsequent laboratory sample preparation procedure. Nail scrapings are taken using sharp wooden sticks and transferred directly onto lint in the appropriate glass vial. Gloves are changed between sampling areas. When all samples have been taken, they are returned to the container which is resealed with the unused lid. This is placed inside the unused nylon bag which is sealed with the integrity labels, and it is later sealed across the signed integrity labels using transparent adhesive tape in a manner specified under laboratory exhibit packaging instructions.

4.3.27 General purpose sampling kit

Apart from suspects, there are many other requirements for firearms and/or explosives residue sampling. These include sampling of alleged firing points (e.g., car interiors and window surrounds), suspected bullet holes and bullet strike marks, suspected weapon hides, and of vehicle interiors (transport of explosives), suspected explosives hides, dwelling interiors (possible explosives/bomb making or storage), seats of explosions (to identify explosives type). For these purposes, a revised general purpose sampling kit has been introduced because the suspect sampling kit is expensive and difficult to prepare and it would be a very wasteful practice to use it to sample such areas. The general purpose kit is used mainly for scenes of crime work and in such circumstances the sampler will already be wearing a disposable overall and gloves. Hand washing facilities may, or may not, be available but the hands will be cleaned with the x-ray wipe provided for the purpose in the kit. The swabbing method and materials are the same as in the suspect sampling kit. The control swabs are the same except that for firearms residue examination the environmental control is taken from the object or area being swabbed, care being taken to avoid areas likely to be contaminated with the substance of interest. The object of this control is to determine background levels of elements or compounds of interest. In some cases, it is not possible to take an environmental control as the area could potentially be saturated with residues.

The kit is contained in a heat sealed nylon bag which holds a rigid plastic container with a tamper proof lid, an X-ray wipe, a ball-point pen in a self-seal plastic bag and a spare lid to reseal the container after use. The contents are shown in detail in Figure 4.12. In its use, and all other aspects, i.e., security, contamination avoidance and self-containment, the general purpose kit closely resembles the suspect sampling kit. The only major differences are that the areas sampled are not predetermined, but are at the discretion of the sampler, and it is substantially cheaper than the suspect sampling kit and its preparation is less complex. It replaces the two previous general purpose sampling kits, one of which was used for firearms residue sampling and the other for explosives residue sampling. In order to avoid any confusion between the suspect sampling kit and the general purpose sampling kit, the rigid plastic containers are different in size and colour and are clearly labelled, and only one of the kits contains a disposable overall.

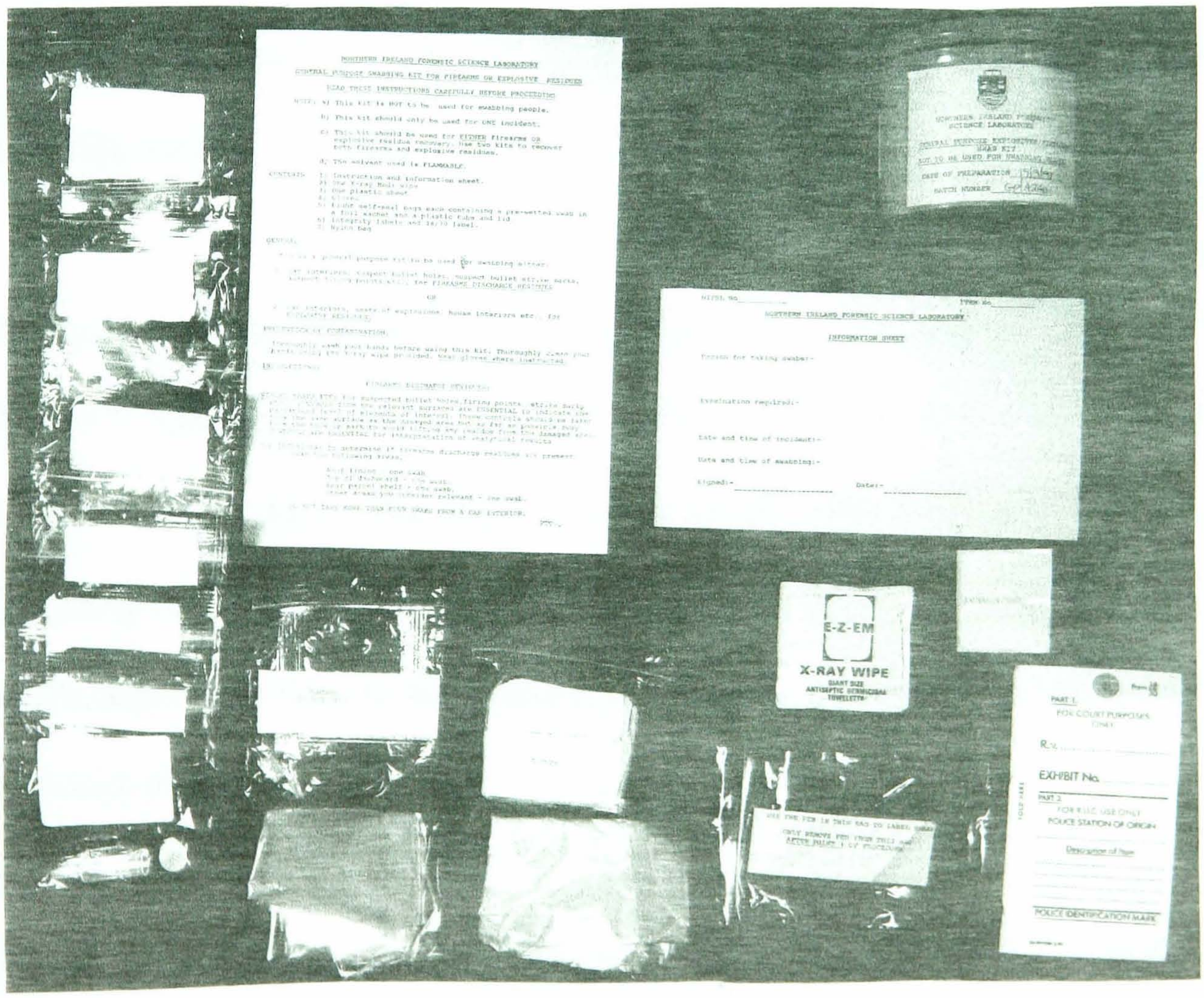


Figure 4.12 General purpose sampling kit

4.3.28 Development of a method for organic FDR detection

A slurry mix of the sample and Chromosorb 104 was sucked into a 1 mm I.D. PTFE tube containing Amberlite XAD - 4. Organic explosive residues and organic FDR are selectively retained on the support material. The cleaned organic residues were then eluted from the tube using acetonitrile - water (25:12, $^{v}/_{v}$) as per the method of Lloyd (159, 160).

4.3.29 Development of the SPE system

Commercial reversed phase (C18) and aminopropyl (NH_2) 100 mg 1.5 ml SPE columns were compared with 1.5 ml SPE columns prepared in the laboratory which contained 40 mg Chromosorb 104 - Amberlite XAD-4 (3:1). The ability of the different SPE materials to extract and recover FDR from acetonitrile was assessed using the Millilab workstation.

100 µl of an isopropanol solution containing 10 ng/µl each of NG, 1,3-DNB, 2,4-DNT, DPA, EC and MC, diluted 1:9 with water, was used to simulate organic FDR. The standard solution was added to the three different SPE columns according to the procedure recommended in reference185. To improve the binding of the organic residues to the aminopropyl support the mixed standard was diluted 1:19 with hexane.

The organic FDR was recovered from the SPE columns in 300 µl of acetonitrile, analysed by HPLC-PMDE and GC-MS, and the recovery calculated. The experiments were performed twice to obtain an average recovery (Table 7.07 refers).

4.3.30 Efficiency of Millilab extraction

Three new cotton laboratory coats were suction sampled for a period of 5 minutes each. A 400 μl volume of methanol containing 10 ng of NG, 1,3-DNB, 2,4-DNT, DPA, EC and MC were added to each Deldrin holder to simulate the recovery of organic FDR from clothing. Organic FDR were extracted from the three Deldrin units and cleaned-up and concentrated by SPE using the Millilab workstation. To test the system for carryover, clean Deldrin units and SPE tubes (blanks) were extracted after each sample. The extracted organic FDR and blanks were analysed by HPLC/PMDE and GC/MS (Table 7.08 refers).

4.3.31 Efficiency of swab extraction

Test 1

An isopropyl alcohol (IPA) standard containing 10 ng/ul each of NG, 1,3-DNB, 2,4-DNT, DPA, EC and MC was used to simulate organic FDR extracted from an acrilan swab in a syringe barrel (sample tube, Figure 4.11). After 2 minutes the sample was extracted through a 1 micron membrane filter, by centrifugation at 3000 rpm for 1 minute. The IPA extract was analysed by HPLC/PMDE and GC/MS (Table 7.09 refers).

Test 2

Test 1 was repeated with the SPE stage included (Table 7.10 refers).

4.3.32 Recovery and analysis of FDR from clothing

Test 1

Three different items of clothing with varying retentive properties for FDR were doped with residues from a Colt Python .357" Magnum calibre revolver using double based Winchester .357" calibre ammunition, by wearing each garment and firing six shots in still air conditions. The garments were removed and packaged immediately after the tests and suction sampled within 1 hour of firing. The recovered residues were extracted and analysed for organic and inorganic FDR using GC/TEA, GC/MS and SEM/EDX. Precautions were taken to ensure that no contamination of garments with FDR from other sources occurred. Samples of air within the room where the shooting took place and samples from the hands and clothing of the firer were taken prior to performing the shootings (Table 7.11 refers).

Test 2

Two new laboratory coats were each doped with residues from a single shot using the same revolver and ammunition as in Test 1. Precautions were taken to avoid contamination from extraneous sources. The garments were suction sampled and the samples were extracted and analysed for FDR as in Test 1 (Table 7.12 refers).

4.3.33 Current method for organic FDR detection

Five different types of firearms were selected, a rifle, a pistol, a revolver, a shotgun and a submachine gun, and each firearm was duplicated, so that one would be used to fire single based ammunition and the other used to fire double based ammunition. The firearms were stripped and cleaned in the normal manner and 10 rounds of ammunition, of the type to be used in the subsequent outdoor test firing of the firearm, were discharged in each firearm. This was done to ensure no carry over of residue from previous firings. The outside of each firearm was then cleaned using an x-ray medi wipe, prior to the firearm being sealed in a plastic bag whilst awaiting test firing outdoors

Test 1

In order to get appropriate ammunition a selection of available ammunition had to be examined in order to determine if it was single or double based. This was done by taking a single round from previously unopened boxes and quantitatively analysing the propellant by GC/MS (Table 7.14 refers).

Test 2

The test firings were done on different days at two outdoor firing ranges. The firearms and ammunition used are given in Table 4.01

Firearms	***Ammunition***	
	Double-based	***Single-based***
Beretta Model 12 SMG 9 mmP	FC 9 mm LUGER	GECO 9 mm LUGER
Mossberg pump action 12-bore shotgun	12-bore Winchester	12-bore RWSIGECO ROTTWEIL
S&W Model 19 revolver .357" Magnum	PMC .357 mag	LAPUA .357 mag
Browning High-Power pistol 9 mmP	FC 9 mm LUGER	GECO 9 mm LUGER
Colt AR-15 rifle .223"	HP84 5.56	LAPUA .223 REM

Table 4.01 Firearms and ammunition for outdoor tests

The first series of tests involved the firing of 6 shots from each firearm using single based ammunition with the firer wearing a clean laboratory coat. The firers hands and face were swabbed and the coat was removed and packaged immediately after firing for subsequent suction sampling in the laboratory. Control samples were taken from the coats prior to use. The test was repeated using double based ammunition. Elaborate precautions were taken to avoid contamination.

The second series of tests involved the same firearms and the same sampling procedures, but only the single based ammunition was used to fire 2, 6 and 10 shots. In the first series of tests it was a cold, wet and windy day with the wind blowing from right to left across firers and for the second series the weather was good but a strong wind was blowing into the face of the firers. The samples were analysed by GC/TEA with HPLC/PMDE confirmation of positives, by GC/MS and SEM/EDX. (Tables 7.15 and 7.16 refer).

5 *FIREARMS DISCHARGE RESIDUES - INORGANIC – Results and Discussion*

5.1 PARTICLE CLASSIFICATION

5.1.1 Blank cartridges

Doubts initially arose about the particle classification scheme when this laboratory experienced a case involving the examination of a suspects upper outer clothing for firearms discharge residue, in which the only discharge particle types detected were Pb only and Ba, Ca, Si; the Ba, Ca, Si type were considered to be unique to the discharge of a firearm at the time. The size and appearance of the particles were consistent with firearms discharge residue; however, none of the other particle types were detected and test firing of similar ammunition to that used in the incident (same calibre and headstamp) produced the complete range of particle types. In an effort to explain this anomaly it was decided to investigate the suspects occupation as a possible source of the particles. Enquiries revealed that the suspect was employed as a general labourer on a building site and that cartridge operated industrial tools (stud guns) were used on the site. Consequently it was necessary to investigate discharge residue particles from all types of cartridge operated industrial tools used in Northern Ireland, in order to establish if this was the source of the particles detected on the suspect. The results of the study (183) neither proved nor disproved that the use of a cartridge tool accounted for the particles on the suspect.

Two other similar cases were subsequently experienced and as a consequence it was decided to act on the side of safety by reclassifying Ba, Ca, Si particles as indicative, rather than unique, to the discharge of a firearm.

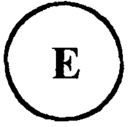
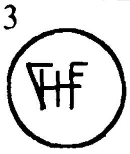
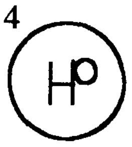
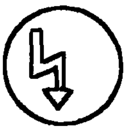
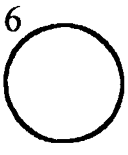
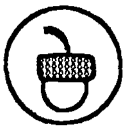
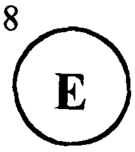
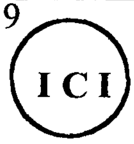
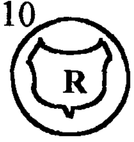
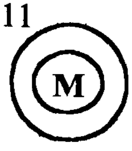
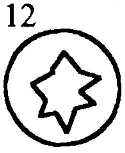
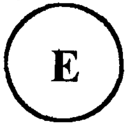
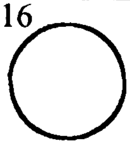
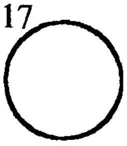
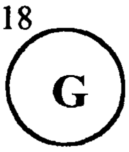
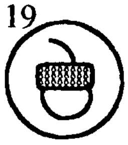
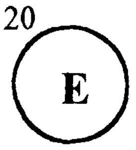
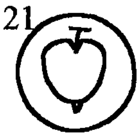
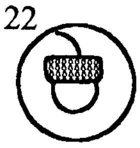
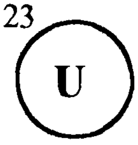
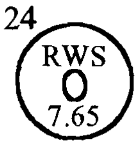
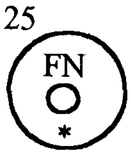
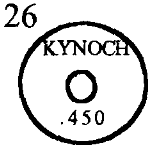
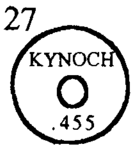
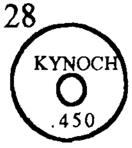
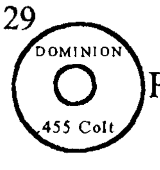
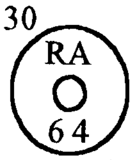
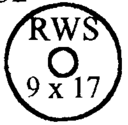
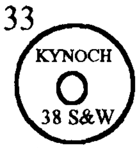
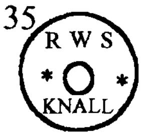
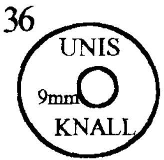
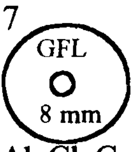
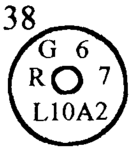
In order to clarify the situation and to test the validity of the particle classification scheme it was necessary to consider other possible sources of particles that may have a similar elemental composition and morphology to firearms discharge residue particles. As the formation of firearms discharge residues particles involves high temperatures and the particles have the appearance of condensing from a vapour or melt, it was decided to study blank cartridges other than those used in cartridge tools. A limited range of toy caps, matches, signal flares and fireworks were also examined as these, when used, involve high temperatures and may contain one or more of the elements lead, antimony or barium, elements associated with firearms ammunition.

A blank cartridge could be defined as 'a percussion initiated cartridge that does not have a bullet or missile that is designed to be lethal'. Blank cartridges usually consist of a metal cartridge case containing either a primer and propellant or a primer only. They can be crimp closed or closed by using a sealing wad or wax. Blank cartridges have many uses, some of which are given in Table 5.01.

Training (e.g. weapon training, gun dogs)	Humane killers (captive bolt type)
Signalling (e.g. starting pistol)	Saluting cartridges (ceremonial)
Cartridge tools (stud guns)	Bird scarers
Grenade launching	Target launching
Antenna erecting	Line throwing
Engine starters	Mortar ignition
Fright guns (personal protection)	Balloon cable cutting
Gas guns (personal protection)	Spotting charges for practice bombs and mines
Film and Theatre	Artillery shell training adapters
'Quick draw' contests	Cleaning industrial furnaces

Table 5.01 Uses of Blank Cartridges

A range of blank cartridges were examined to determine the elemental content of the primer. Particular attention was given to blank cartridges designed for use in starting pistols and replica firearms as these sources are the most likely to be encountered in casework. Results are given in Figure 5.01

1  .22" Pb, Sb, Ba (Al, Cu, Fe, Si)	2 .22" MAGNETIC Pb, Sb, Ba (Al, Cu, Si)	3 .22" Pb, Sb, Ba (Al, Cu, Si, Zn)	4 .22" Pb, Sb, Ba (Al, Cu, Si)
5  .22" Pb, Sb, Ba (Al, Cu, Fe, Si, Zn)	6  .22" PLASTIC Pb, Sb, Ba (Al, Cl, Cu, Fe, K, P, Zn)	7  .22" Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, K, P, Si)	8 .22" Pb, Ba (Al, Cu, Fe, K, P, S, Si, Zn)
9 .22" Pb, Ba, Hg (Al, Cl, Cu, Fe, K, Si)	10 .22" Pb, Ba (Al, Ca, Cu, K, P, Si, Zn)	11 .22" Pb, Sb, Ba (Al, Cl, Cu, Fe, K, Si, Zn)	12  .22" Pb, Sb, Hg (Al, Cu, Fe, K, S, Si, Zn)
13 .22" Pb, Ba, Hg (Al, Cl, Cu, Fe, K, S, Si, Zn)	14  .22" Pb, Sb, Ba,Hg (Al, Cl, Cu, Fe, K, S, Si, Zn)	15  .22" Pb, Ba (Al, Ca, Cl, Cu, Fe, Si, Zn)	16  .22" Pb, Hg (Al, Ca, Cl, Cu, Fe, K, S, Si, Zn)
17  .22" Pb, Ba, Hg (Al, Ca, Cl, Cu, Fe, K, P, Si, Zn)	18 .22" Sb, Hg (Al, Cl, Cu, Fe, K, Si, Zn)	19  .22" Pb, Ba (Al, Cu, Fe, K, Si, Zn)	20 .22" Pb, Ba (Al, Cu, Si)
21  .22" Pb, Sb, Ba (Al, Cu, Fe, Si)	22  .22" Pb, Sb, Ba (Al, Cu, Si)	23 .22" Pb (Al, Cu, Fe, Si, Zn)	24  7.65 mm Pb, Sb, Ba (Al, Cu, Fe, Si, Zn)
25  7.65 mm Sb, Ba, Hg (Al, Cl, Cu, Fe, K, S, Si, Sn, Zn)	26 .450" Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, K, S, Si Zn)	27 .455" Pb, Sb, Ba (Al, Ca,Cl, Cu, Fe, K, S, Si, Zn)	28 .450" Pb, Hg (Al, Cl, Cu, Fe, K, S, Si, Z
29 .455" Colt Pb, Sb, Ba (Al, Cl, Cu, Fe, K, S, Si, Zn)	30 .450" ACP Pb, Ba (Al, Cl, Cu, K, S, Si, Zn)	31 .380" Pb, Sb, Ba (Al, Cl, Cu, K, S, Si Zn)	32  9 mm MAGNETIC Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, K, Si, Zn
33 .380" S&W Pb, Ba (Al, Cu, Fe, K, S, Si Zn)	34 .380" S&W Pb, Ba (Al, Ca, Cu, Fe, K, Si, Zn)	35  9 mm Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, K, S, Si, Zn)	36  9 mm Pb, Sb, Ba (Al, Cl, Cu, Fe, K, S, Si)
37  8 mm Pb, Sb, Ba (Al, Cl, Cu, Fe, Si)	38  7.62 mm Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, K, S, Si, Zn)	39 .38" SPL Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, K, S, Si Zn	40  6.35 mm Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, K, S, Si, Zn)

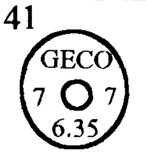
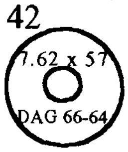
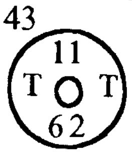
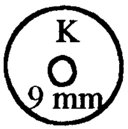
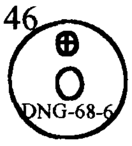
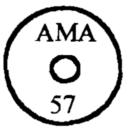
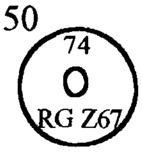
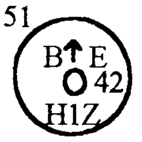
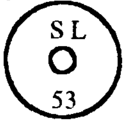
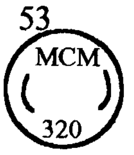
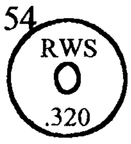
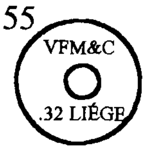
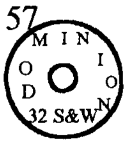
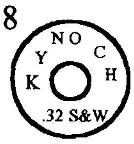
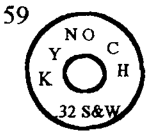
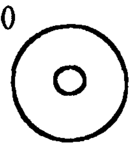
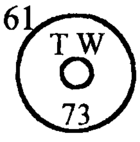
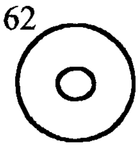
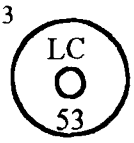
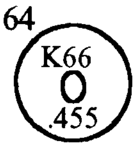
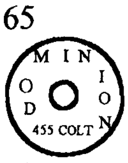
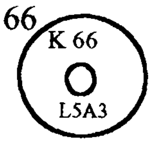
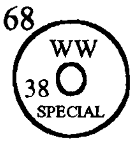
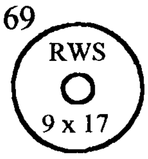
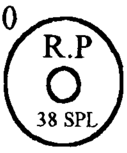
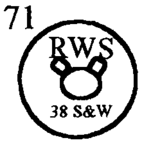
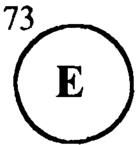
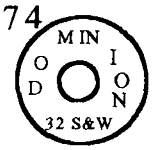
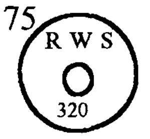
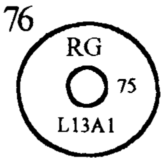
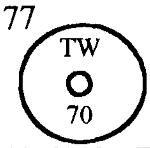
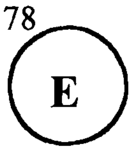
41  6.35 mm Pb, Sb, Ba (Al, Cu, Fe, K, Si)	42  7.62 mm PLASTIC Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, Si)	43  7.5 mm Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, K, Si, Zn)	44  9 mmP Pb, Sb, Ba (Al, Cu, Cl, Fe, Si, Zn)
45  9 mmP Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, K Si, Zn)	46  9 mmP Pb, Sb, Ba (Al, Cu, Fe, K, Si, Zn)	47  9 mmP Pb, Sb, Ba (Al, Ca, Cu, Fe, Si, Zn)	48  9 mm Sb, Ba, Hg (Al, Cu, Fe, K, S, Si, Zn)
49  .303" Pb, Sb (Al, Cl, Cu, Fe, K, Si, Zn)	50  .303" Pb, Ba (Al, Ca, Cl, Cu, Fe, K,Si)	51  .303" Pb, Sb, Hg (Al, Ca, Cl, Cu, Fe, K, S, Si, Zn)	52  .30-06 Pb, Sb, Ba (Al, Cl, Cu, Fe, K, Si)
53  .320" Sb, Hg (Al, Cl, Cu, K, S, Si, Zn)	54  .320" Pb, Sb, Ba,Hg (Al, Cl, Cu, Fe, K, S, Si, Zn)	55  .320" Pb (Al, Cl, Cu, Fe, K, S, Si, Zn)	56  .320" Pb, Sb, Ba (Al, Cl, Cu, Fe, K, Si)
57  .320" S&W Pb, Ba (Al, Ca, Cl, Cu, Fe, K, Si, Zn)	58  .320" S&W Pb, Ba, Hg (Al, Ag, Ca, Cl, Cu, Fe, K, S, Si, Zn)	59  .320"S&W Pb, Sb, Ba (Al, Cl, Cu, Fe, K, S,Si, Zn)	60  .320" Sb, Hg (Al, Cl, Cu, Fe, K, S, Si, Zn)
61  .223" Pb, Sb, Ba (Al, Cl, Cu, Fe, K, S, Si, Zn)	62  .30-06 PLASTIC Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, S, Si, Zn)	63  .30-06 Pb, Sb, Ba (Al, Ca, Cl, K, Cu, Fe, S, Si, Zn)	64  .455" Hg (Al, Ag, Cl, Cu, K, S, Si, Zn)
65  .455" COLT Pb, Ba (Al, Ca, Cl, Cu, Fe, K, Si, Zn)	66  .450" ACP Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, K, S, Si Zn)	67  9 mmP Pb, Sb, Ba (Al, Cl, Cu, Fe, K, Si, Zn)	68  .38 SPL Pb, Sb, Ba (Al, Cl, Cu, Fe, K, S, Si, Zn)
69  .380" Pb, Ba (Al, Ca, Cl, Cu, Fe, K, S, Si, Zn)	70  .38" SPL Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe,K, S, Si, Zn)	71  .38"S&W Pb, Ba (Al, Ca, Cl, Cu,Fe, K, S, Si Zn)	72  8 mm Pb, Sb, Ba (Al, Cl, Cu, Fe, K, S, Si, Zn)
73  .22" LR Pb, Ba (Al,Cl, Cu, Fe, K, S, Si Zn)	74  .32" S&W Pb, Ba (Al, Ca, Cl, Cu, Fe, K, S, Si, Zn)	75  .320" Pb, Ba (Al, Ca, Cl, Cu, Fe, K, S, Si, Zn)	76  7.62 mm Pb, Ba (Al, Ca, Cl, Cu, Fe, K, S, Si, Zn)
77  .223" Pb, Sb, Ba (Al, Cl, Cu, Fe, K , S, Si, Zn)	78  .22" LR Pb, Ba (Al, Cl, Cu, Fe, Si, Zn)	79  .303" SPL Pb, Sb, Hg (Al, Cl, Cu, Fe, K, S, Si Zn)	

Figure 5.01 Residue in spent cartridge case (blanks)

[Comparison of Figure 5.01 with Figures 5.03 and 5.04 reveals that blank cartridges are similar in composition to live ammunition and may be expected to produce discharge particles with similar composition to those originating from firearms discharge].

Discharge residue particles from starter pistol blanks were examined for comparison with discharge residue particles originating from firearms ammunition. Table 5.02 gives the starting pistol discharge residue particles classified according to their consistency with FDR particles.

Blank No	***Pb, Sb, Ba***	***Pb, Sb***	***Pb, Ba***	***Sb with S***	***Ba only***	***Pb only***
1	103	ND	2	ND	2	ND
2	96	14	7	ND	ND	ND
3	116	4	ND	5	ND	ND
4	126	8	ND	ND	ND	6
5	80	7	8	10	ND	ND
6	133	ND	ND	ND	ND	ND
7	99	ND	ND	3	ND	ND

ND = none detected

Table 5.02 Starting pistol discharge residue

The particles detected were all in the size range 1 micron to 19 microns. Both spherical and irregular particles were found and all had the appearance of having partially or wholly condensed from a vapour or melt, and all exhibited some degree of curvature. They did not exhibit any degree of crystallinity and their surface details were smooth, irregular, cratered or nodular. The physical characteristics of the particles were indistinguishable from FDR particles.

Individually the starting pistol discharge residue particles could not be distinguished from FDR particles, either by physical appearance, size range or elemental composition, which includes the additional accompanying elements. It is a reasonable assumption that discharge residue particles from any blank cartridge could be confused with FDR. Mercury containing particles could also be produced from the use of blanks incorporating mercury fulminate (see Figure 5.01 for mercury containing blanks).

When the particles were considered as a group three distinct differences between firearms and starting pistol discharge residue particles were noted.

(1) The ratio of indicative to unique particles is markedly different from that of firearms discharge. From firearms residue casework statistics, based on cases with at least one particle in the unique category, the ratio of indicative to unique particles is approximately 35:1. For starting pistol discharge residue particles the overall ratio is in the region of 1:10.

This abundance of unique discharge particles suggests a much more homogeneous mixture of discharge residue, which is not surprising considering that the blanks were rimfire primed, the chemicals are contained within a relatively small volume compared to firearm ammunition i.e. a more intimate mixture contained in a smaller cartridge case. In addition there is no bullet involved to complicate the issue by producing a large number of bullet particles, which would make a substantial contribution to the total number of particles in the indicative category.

It is interesting to note that blank number 6, Table 5.02, produced Pb, Sb, Ba unique particles only, and also that it had a plastic cartridge case. Considering that 8 rounds were fired and that a primer composition consists of a mixture of chemical compounds, this result tends to suggest that the mixture was initially homogeneous and that the discharge gases/vapours were intimately mixed prior to condensation of the particles. This trend is noticeable throughout Table 5.02, particularly for blanks numbered 1, 6 and 7.

(2) Unlike firearms discharge residue, the discharge residue from the blank cartridges contained very few lead only particles, which is not unexpected as there is no lead bullet involved and any lead only particles detected must originate from the primer. The few lead only particles detected all originated from the discharge of blank cartridge number 4. A tentative explanation for this could be the relative proportion/total quantity/burning rate/grain size of the lead compound or compounds or the degree of uniformity of the priming mixture.

(3) Unlike firearms discharge residue, each of the blank cartridges produced a limited range of discharge particle types.

There are several things to be considered when comparing the ratio of indicative to unique particles in firearms casework and starting pistol discharge residue. Like with like is not being compared in that samples of starter pistol discharge residue were taken immediately after firing whereas in casework the vast majority of the suspects were apprehended between 1 and 4 hours after the shooting incident. In laboratory tests one has a high degree of confidence in the origin of the particles in the indicative category, whereas in casework, one cannot be sure of the origin of some of the particles, particularly the single primary element ones. In order to compare like with like a further experiment was conducted involving promptly collected FDR. Results are given in Table 5.03.

	Unjacketed Bullet ***.38 Special Calibre***		***Jacketed Bullet*** ***.38 Special +P calibre***	
Particle Type	***Number***	***Approximate %***	***Number***	***Approximate %***
Pb, Sb, Ba	39	17.0	29	27.0
Sb, Ba	None	-	None	-
Ba, Ca, Si	3	1.5	None	-
Pb, Sb,	42	18.0	16	15.0
Pb, Ba	6	3.0	44	40.0
Pb only	138	59.0	14	13.0
Sb only	6	3.0	1	0.5
Ba only	None	-	4	4.0
Ratio Indicative/ Unique	5:1		4:1	

Table 5.03 Particle types from promptly collected firearms discharge residue

[Examination of the copper and zinc relationship revealed that for the unjacketed bullet, 94% of the particles contained copper only and 6% contained both copper and zinc, with copper > zinc. For the jacketed bullet, 90% of the particles contained copper only and 10% contained both copper and zinc, with copper > zinc in 9.5% and copper = zinc in 0.5%.]

As can be seen from Table 5.03, the proportion of indicative particles exceeds the proportion of unique particles, even for promptly collected firearms discharge residue. The higher proportion of indicative particles detected in casework is almost certainly due to particles from non-firearms sources, particularly single primary element ones, meeting the criteria of the classification scheme.

It is interesting to note that the firing of ammunition with an unjacketed bullet produced more lead only particles than similar ammunition with a jacketed bullet, which is consistent with the findings of the Aerospace Corporation work (140). A surprising result was the number of particles containing copper from the firing of the unjacketed bullets. This is inconsistent with their findings and is difficult to explain, the only obvious source of copper being the cartridge case/primer cup. They concluded that these sources did not appear to make a significant contribution to the elemental composition of the discharge particles. Little significance can be attached to this finding as it is based on a particular gun/ammunition combination and very limited experimental data.

Starting pistols/blank firing imitation firearms normally have a hardened steel blockage in the barrel to prevent them from being converted to fire bulleted ammunition. Firearms and firearms ammunition are designed so that the maximum pressure is reached when the bullet has travelled a considerable distance up the barrel (like an expanding chamber). Thus the nature of the discharge process differs between firearms and blank firers and this could account for the homogeneous character of the discharge gases and vapours from blank cartridges. In a firearm the vast majority of the discharge residue emerges from the muzzle whereas blank firers have a small vent, usually at the top, to emit the discharge residue. Because of the smaller fixed volume available to the discharge residue gases and vapours and the venting mechanism in blank firers it is likely that more uniform temperatures and pressures are attained and better mixing occurs, leading to an abundance of Pb, Sb, Ba particles and a limited range of particle types.

Whatever the reason there is no doubt that the discharge of blank cartridges produces a much higher ratio of unique to indicative particles than the discharge of firearms ammunition. As a consequence of the work on blank cartridges, discharge residue particles that were previously referred to as firearms discharge residue are now referred to as cartridge discharge residue (CDR) throughout the U.K.

5.1.2 Toy caps

Six different brand names of caps designed for use in toy guns were examined, two of which were the paper roll type, the others being the individual plastic cup type that is placed on the 'anvil' of the toy gun.

Analysis of discharge particles revealed that both spherical and irregular particles were present, approximately 1in 12 being spherical. The particle size range was from 3 μm to 160 μm. The elements detected were Al, Ca, Cl, Cu, Fe, K, Mg, P, Pb, S, Sb, Si, Ti and Zn with Ca, Cl, K, P, Pb and Si being the major elements. Antimony and lead did not occur together and none of the samples examined would be confused with FDR particles as their elemental profile differed. A small proportion of the particles containing either lead or antimony met the criteria for 'single' element FDR particles.

At the time the tests were conducted, children in Northern Ireland played with 'devil bombers', which consisted of a solid mixture rolled up in a piece of waxed paper. When thrown with force against a hard object they exploded creating a loud bang. Visual examination of the contents revealed a mixture of wood like material (cellulose) and sand like material (silicate). Elemental analysis of the mixture showed Ag, Si at major level, Al at minor level and K, Cl at trace level. Its exact composition was not determined but it would appear that cellulose was a fuel, silicate was a frictionator, a silver compound (azide or fulminate?) was the explosive and potassium chlorate was the oxidizer.

5.1.3 Matches

Analysis of particles originating from the use of matches revealed that only a very small number of spherical particles were present, the majority being very irregular. The elements detected were Al, Ca, Cl, Cr, Fe, K, Mg, Mn, P, S, Sb, Si and Zn, with K, Cl, P, S and Si being the major components. Antimony was detected in only two of the seventeen types. None of the samples examined would be confused with FDR particles as both their morphology and elemental content differed.

5.1.4 Flares

Flares have several uses including signalling and illumination and there are several means of launching, including hand held, rocket and specifically designed pistols (e.g. Verey pistol). The use of flares in Northern Ireland is very limited, the security forces using them occasionally. They have in rare instances been used illegally. Analysis of two hand held types showed that the vast majority of the discharge particles were irregular with several large flakes present. Elemental analysis revealed the presence of Ca, Cu, Fe, Mg, Na, Ti, Zn in one of the flares with Mg, Na at major level, and Al, Ba, Cl, Fe, K in the other with Al, K, Cl at major level. Their morphology and composition was such that they would not be confused with FDR particles.

The flares examined were the only ones used by the Security Forces at the time. A brief review of the literature (188, 189, 190) on pyrotechnics/flares indicates that Pb and Sb compounds are infrequently used and when used do not occur together. Barium compounds are frequently used, particularly in signal flares. From the literature it is apparent that residues from flares could not be confused with FDR as the elements Pb, Sb and Ba would be accompanied by other elements that would clearly indicate a non-FDR source.

5.1.5 Fireworks

In Northern Ireland only indoor type fireworks can be purchased without a special licence. Analysis of particles originating from the use of indoor type fireworks showed only a few spherical particles, the majority being large irregular shaped flakes. The elements Al, Ba, Cl, Cr, Fe, K, S and Sb were detected, all of which were at a major level.

Analysis of particles originating from the use of outdoor fireworks revealed that the majority of the particles were irregular, many were crystalline and many large flakes were present. A small proportion of the particles were spherical and physically resembled FDR particles. Elemental analysis showed the presence of Al, As, Ba, Ca, Cl, Cu, Fe, K, Mg, Na, Pb, S, Sb, Si, Sr, Ti, Zn and Zr. None of the particles detected would be confused with FDR particles as the primary FDR elements were always accompanied by elements that were clearly of non-FDR source.

In conclusion, lead, antimony and barium may be encountered in pyrotechnics, in both fireworks and flares. Lead and antimony were present in toy caps but were not found occurring together. Antimony only was detected in matches. None of these sources should be confused with FDR particles as their morphology and/or elemental content differs. (The text on toy caps, matches, flares and fireworks represents the conclusions of the work conducted, as the details and results were lost in the explosion at the laboratory in September 1992).

5.1.6 Accompanying elements

From casework statistics the unique particles (those containing the combination Pb, Sb and Ba, and those containing Sb and Ba) occur in the ratio 7:3 respectively. Approximate percentages for indicative particles are Pb only 55%; Pb, Sb 20%; Pb, Ba 8%; Sb only 7%; Ba, Ca, Si 5%; Ba only 5%.

Table 5.04 gives an indication of the levels of the primary elements in each particle type.

Particle Type	***Element***	***~% Major***	***~% Minor***	***~% Trace***
Pb, Sb, Ba	Pb	61	39	-
	Sb	39	31	30
	Ba	64	31	5
Sb, Ba	Sb	-	12	88
	Ba	100	-	-
Ba, Ca, Si	Ba	93	7	-
	Ca	28	55	17
	Si	34	52	14
Pb, Ba	Pb	95	5	-
	Ba	38	57	5
Pb, Sb	Pb	66	34	-
	Sb	55	40	5
Sb only	Sb	92	8	-
Ba only	Ba	100	-	-
Pb only	Pb	95	5	-

Table 5.04 Elemental level per particle type

Table 5.05 gives an indication of the levels of accompanying elements in each particle type and is the basis for note C. in the Particle Classification Scheme - Table 5.06.

Element	*Pb, Sb, Ba*	*Sb, Ba*	*Ba, Ca, Si*	*Pb, Ba*	*Pb, Sb*	*Sb only*	*Ba only*	*Pb only*
Al	47.0 19.5 M 19.5 Mi 8.0 T	Zero	69.0 17.0 M 41.5 Mi 10.5 T	35.0 Zero M 30.5 Mi 4.5T	53.0 1.0 M 24.5 Mi 27.5 T	88.5 Zero M 21.0 Mi 67.5 T	87.5 Zero M 55.0 Mi 32.5 T	63.0 Zero M 26.0 Mi 37.0 T
Ca	86.0 33.0 M 53.0 Mi Zero T	100.0 87.0 M 13.0 Mi Zero T	100.0	97.0 73.0 M 20.0 Mi 4.0 T	15.5 1.0 M 10.0 Mi 4.5 T	16.0 6.5 M 3.0 Mi 6.5 T	91.0 3.0 M 25.0 Mi 63.0 T	69.0 9.5 M 37.0 Mi 22.5 T
Cl	43.5 Zero M 26.5 Mi 17.0 T	93.0 Zero M 11.5 Mi 81.5 T	69.0 Zero M 20.5 Mi 48.5 T	36.0 0.5 M 28.0 Mi 7.5 T	37.5 1.0 M 6.5 Mi 30.0 T	84.0 Zero M 26.0 Mi 58.0 T	92.5 Zero M 12.5 Mi 80.0 T	34.0 Zero M 11.5 Mi 22.5 T
Cr	less than 0.5 (all trace)	Zero	Zero	3.0 Zero M 2.5 Mi 0.5 T	1.0 (all trace)	Zero	less than 0.5 (all trace)	1.5 (all trace)
Cu	96.0 Zero M 52.5 Mi 43.5 T	99.0 Zero M 50.0 Mi 49.0 T	90.0 Zero M 7.0 Mi 83.0 T	99.0 Zero M 30.0 Mi 69.0 T	100.0 Zero M 22.5 Mi 77.5 T	89.0 Zero M 6.5 Mi 82.5 T	47.5 Zero M 2.5 Mi 45.0 T	76.5 3.5 M 18.5 Mi 54.5 T
Fe	94.5 1.5 M 57.5 Mi 35.5 T	100.0 Zero M 93.0 Mi 7.0 T	79.5 3.5 M 41.5 Mi 34.5 T	99.5 Zero M 91.5 Mi 8.0 T	73.0 Zero M 36.5 Mi 36.5 T	92.0 Zero M 14.5 Mi 77.5 T	37.5 Zero M 20.0 Mi 17.5 T	64.5 Zero M 22.0 Mi 42.5 T
K	58.0 Zero M 52.5 Mi 5.5 T	Zero	65.5 Zero M 27.5 Mi 38.0 T	47.5 Zero M 35.5 Mi 12.0 T	36.0 1.0 M 26.0 Mi 9.0 T	24.0 Zero M 9.5 Mi 14.5 T	89.0 Zero M 4.0 Mi 85.0 T	50.0 Zero M 14.0 Mi 36.0 T
Mg	1.0 (all trace)	Zero	14.0 (all trace)	28.0 Zero M 19.5 Mi 8.5 T	20.0 (all trace)	9.5 (all trace)	Zero	12.0 (all trace)

Element	*Pb, Sb, Ba*	*Sb, Ba*	*Ba, Ca, Si*	*Pb, Ba*	*Pb, Sb*	*Sb only*	*Ba only*	*Pb only*
Na	4.0 (all trace)	Zero	Zero	1.0 (all trace	zero	0.5 (all trace)	Zero	1.0 (all trace)
P	Zero M 3.0 1.5 Mi 1.5 T	Zero	Zero M 14.0 3.5 Mi 10.5 T	Zero M 6.0 3.0 Mi 3.0 T	7.5 (all trace)	Zero M 15.0 2.0 Mi 13.0 T	Zero	Zero M 23.0 10.0 Mi 13.0 T
S	31.5 M 67.0 35.5 Mi Zero T	70.0 (all minor)	59.0 (all trace)	81.0 M 85.5 4.5 Mi Zero T	10.5 M 37.5 25.5 Mi 1.5 T	14.5 M 81.0 40.5 Mi 26.0 T	85.0 (all trace)	24.0 M 27.0 3.0 Mi Zero T
Si	20.5 M 97.0 64.5 Mi 12.0 T	81.5 M 100.0 18.5 Mi Zero T	100.0	76.0 M 99.0 23.0 Mi Zero T	12.0 M 95.5 66.5 Mi 17.0 T	11.5 M 100.0 30.5 Mi 58.0 T	5.0 M 25.5 3.0 Mi 17.5 T	12.0 M 83.5 45.0 Mi 26.5 T
Ti	5.0 (all minor)	Zero	1.0 (all trace)	4.5 (all trace)	Zero M 7.0 2.0 Mi 5.0 T	Zero M 16.0 1.0 Mi 15.0 T	Zero M 3.5 0.5 Mi 3.0 T	Zero M 10.0 3.0 Mi 7.0 T
Zn	Zero M 7.5 5.0 Mi 2.5 T	18.0 (all minor)	14.0 (all trace)	Zero M 27.5 21.0 Mi 6.5 T	7.5 (all trace)	7.0 (all trace)	Zero M 7.5 1.0 Mi 6.5 T	Zero M 7.0 4.5 Mi 2.5 T

M = Major
Mi = Minor
T = Trace

Table 5.05 Percentage occurrence of certain accompanying elements in unique and indicative particles

The work serves to illustrate the heterogeneous nature of firearms discharge residue particles and to clarify the types of particles detected.

5.1.7 Particle classification scheme

The original particle classification scheme (140) has been revised based on casework experience, research work on blank cartridges etc., and a detailed analysis of 14 years casework results.

The current particle classification scheme used in Northern Ireland is given in Table 5.06. The indicative particles are numbered in tentative order of decreasing significance.

Unique[C]		**Indicative**[C]
Pb, Sb, Ba	(1)	Ba, Ca, Si[A]
Sb, Ba	(2)	Pb, Ba
	(3)	Pb, Sb
	(4)	Sb only (with S)
	(5)	Ba only[A]
	(6)	Sb only (without S)
	(7)	Pb only
	(8)	Pb, Sb, Ba, absent[B]

A. S absent or acceptable at trace level only when Ba is present at a major level.

B. Particles containing no Pb, Sb or Ba may be considered indicative if they have the correct morphology, are composed entirely from the permitted accompanying elements and are accompanied by a range of indicative/unique particles. Such particles are very rarely encountered.

C. Any of the particles listed may also include some of the following:-

Al, Ca, Si, S (unless specifically excluded). *MAJOR, MINOR or TRACE*

Cl, Cu, Fe, K, P, Zn - only if Cu also present and Cu > Zn. *MINOR or TRACE*

Co, Cr, Mg, Mn, Na, Ni, Ti (typically none present occasionally one, rarely two). *TRACE ONLY*

The presence of Sn suggests mercury fulminate primed ammunition.

Table 5.06 Particle Classification Scheme

The classification scheme is based on discharge residue particles from modern primed brass cased ball ammunition. It is only applied rigidly when no other information is available. When a gun, ammunition, spent cartridge case or bullet is recovered it can be examined to determine elemental composition and likely discharge residue particle composition.

The classification scheme has to be flexible in order to encompass the wide range of different primer/cartridge case/propellant/bullet combinations. For example, zinc coated steel cased ammunition gives iron and zinc at major levels in the discharge particles; firearms with rusted barrel interiors or the use of steel jacketed bullets can produce discharge particles with iron at major level; primers containing lead hyphophosphite can give discharge particles with phosphorus at major level; ammunition with black powder can produce discharge particles with K and S at major level. Because of these and other variables the classification scheme has to be flexible.

It must be stressed that the classification scheme is intended as a general guide and is only applied rigidly when there is nothing recovered for comparison purposes.

NOTE:

Firearms discharge residue particles have been noted in a wide range of shape, size and appearance. The shape and appearance is particularly important in the indicative category to aid the differentiation between occupational/environmental particles. Particles originating from the bullet/bullet jacket are sometimes encountered. These are usually clearly identifiable and are not included in the particle classification scheme.

5.2 CASEWORK RELATED TESTS

5.2.1 Particles from handling ammunition

Particles on the outside surface of newly acquired, unopened ammunition were examined in order to determine if the ammunition was contaminated with discharge residue in the factory. Results are given in Table 5.07. Nothing of significance was detected on the control samples.

Ammunition	*Pb only*	*Sb only*	*Pb, Sb*	*Brass*	*Observations*
GECO 9 mm Luger. Brass jacketed bullet	Large Number	None	None	Large number	All the brass particles were irregular shaped whereas the Pb only particles were a mixture of irregular and spherical. The Pb only particles contained some or all of Al, Ca, Cl, Cu, S, Si, Ti, Zn at minor or trace level. A few Fe particles were present. Unusual particles detected were Bi, P, Al major, Si minor, Ca, Fe trace: Cr, Fe major, Si, Zn trace: Zn major, Fe, Cu minor, Si trace: Si, Al, Fe major, Ca minor, Mn Zn trace.
GECO .32 S&W long Unjacketed Pb bullet	Large Number	None	Very Small Number	Small Number	All the brass particles were irregular shaped whereas the Pb only particles were a mixture of irregular and spherical. The Pb only particles contained some or all of Al, Ca, Cl, Cu, S, Si at minor or trace level. The few Pb, Sb particles were spherical. Unusual particles detected were Fe, Cr major, Ni minor, Mn trace: Ti major, Fe, Si minor, Al trace.
GECO .32 S&W Unjacketed Pb bullet	None	Very Small Number	Large Number	None	Numerous Pb, Sb particles and a few Sb only were detected. No other particle types were detected. The particles were mainly spherical. All particles contained Sn and Ti at minor or trace levels in addition to Ca, Cu, Fe, S, Si at minor or trace level.
GECO .38 Special Unjacketed Pb bullet	Large Number	None	None	None	Numerous Pb only particles and a few Fe particles detected. No other particle types detected. The particles were a mixture of irregular and spherical shapes. All the Pb only particles contained Sn and Ti at minor or trace level in addition to S, Si.
GECO .38 S&W Unjacketed Pb bullet	None	None	None	None	None of the particles contained Pb, Sb or Ba. A large number of predominately irregular particles were detected containing some or all of the following: Al, Ca, K, Fe, Si, Ti at major, minor or trace level, Cr, Mg at minor or trace level, Cl, Cu at trace level.
LAPUA 9 mm Luger Cu jacketed bullet	Small Number	Very Small Number	Small Number	Very Small Number	All the brass particles were irregular whereas the Pb only, Sb only and Pb, Sb particles were all spherical. There were numerous particles containing some or all of the following: Al, Ca, Cl, Cr, Fe, Mg at major, minor or trace level, K, Ni at minor or trace level, Cu, Ti at trace level. Unusual particles detected were Fe, P, Si major, Ca minor, Cl, Cu trace; Fe, Cr, Cl major, Si trace.
LAPUA .38 SPL. Unjacketed Pb bullet	Large Number	None	Very Small Number	Large Number	All the brass particles were irregular whereas the Pb only particles were a mixture of irregular and spherical. The Pb, Sb particles were spherical. Several Fe particles were detected. Unusual particles detected were Zn major, S, Si minor, Ca, Cr, Fe, trace; Cr, Fe major, Si minor, S trace.
LAPUA .357 MAG. Brass case and primer cup. Half Cu jacket, Pb H.P bullet	Very Small Number	None	Large Number	Very Small Number	All the brass particles were irregular as were the Pb only particles. The Pb, Sb particles were a mixture of irregular and spherical and accompanying elements were Ca, Cu, S, Si at minor or trace level.

Table 5.07 Particles on new ammunition

The majority of the Pb only, Sb only and Pb, Sb particles that were spherical would be classified as indicative of FDR. However, they were accompanied by particles whose morphology was inconsistent, and only a limited range of particle types were present. No unique FDR particles were detected.

A further test was conducted to determine whether or not ammunition that had been previously loaded in a firearm would have FDR on its surface. Results are given in Table 5.08. Nothing of significance was detected on the control samples.

Ammunition	*Pb, Sb, Ba*	*Sb, Ba*	*Ba, Ca, Si*	*Pb, Ba*	*Pb, Sb*	*Pb only*	*Sb only*	*Ba only*
GECO 9 mm LUGER	37	1	None	6	24	>100	10	None
GECO .38 S&W	18	2	2	1	>100	>100	3	1

Table 5.08 Particles on unloaded ammunition

Table 5.08 shows that the complete range of FDR types can be deposited from handling ammunition that has been in a firearm. It is reasonable to assume that the same applies to handling magazines, or ammunition that has been in a magazine. Particles similar to those detected in Table 5.07 were also present. The presence of FDR on a suspects hands could arise from handling ammunition that had been chambered in a firearm or from handling spent cartridge cases, a gun or a magazine. Consequently the presence of FDR on the hands does not prove that the suspect fired a gun, but does infer **recent** involvement with firearms or related items.

5.2.2 Bullet weight loss on firing

A test was conducted to determine the weight loss of some bullets after discharge. Results are given in Table 5.09.

Bullet Type	*Weight Range Before Firing (g)*	*Weight Range After Firing (g)*	*Weight Loss Range (g)*	*Weight Loss Range (%)*	*Average weight loss (g)*	*Average Weight Loss (%)*
9 mmP Blazer FMJ (Base enclosed)	7.4322→ 7.4741	7.4206→ 7.4630	0.0111→ 0.0125	0.1485→ 0.1678	0.0119	0.1592
9 mmP RG FMJ (Base exposed)	7.5166→ 7.6288	7.4951→ 7.6033	0.0176→ 0.0284	0.2316→ 0.3770	0.0224	0.2942
.380 REV R.P Unjacketed Pb	9.3935→ 9.5412	9.3535→ 9.4936	0.0271→ 0.0476	0.2862→ 0.4989	0.0364	0.3850
W-SUPER-W .38 SPL +P Unjacketed Pb	10.2137 → 10.2679	10.1214 → 10.1962	0.0625→ 0.1051	0.6104→ 1.0277	0.0840	0.8490

Table 5.09 Bullet weight loss on firing

From the limited experimental data it would appear that, as expected, the full metal jacketed bullets lose less than the soft unjacketed bullets. The FMJ bullet with its base enclosed lost less than its equivalent with its base exposed. This is also predictable as the exposed base is subject to erosion during discharge. The .38 SPL +P unjacketed bullet showed a marked increase in loss. Again this is predictable as the bullet travels at a considerably higher velocity (pressure) than the .380 revolver bullet and is consequently subjected to greater stress. Barrel length and rate of rifling twist may be amongst other contributing factors. The three sources of weight loss are erosion of the base by the hot propellant gases, engraving of the outside surface by the rifling of the barrel and friction. It has been noted in casework that fired bullets with exposed bases frequently have powdered lead at the base area. Also noted on some occasions are embedded propellant grains or indentations caused by the grains, in the base of the bullet.

Whilst the weight loss may appear to be insignificant in terms of the total weight of the bullet, it is not insignificant in terms of its potential to produce a large number of discharge residue particles originating from the bullet. This work supports the proposition that the bullet makes a contribution to the discharge particle population.

5.2.3 Effect of water on FDR

It has been observed that in casework involving examination of damp or wet clothing for FDR, the success rate is very low. Possible explanations are that the particles are chemically attacked by the water, that the water removes the particles by physical disturbance e.g. washed away by rain, that the water moves the particles further into the fabric of the garment and the sampling procedure fails to recover them, or that all the cases submitted just happen to be negative. Laboratory experience and casework details make the last two options unlikely. In an attempt to clarify the situation several tests were conducted. The first test involved sampling of the firing hand immediately after firing using the same swabbing material but three different solvents, two of which had water added to them. Results are presented in Table 5.10.

Solvent	*Particles Detected*
Petroleum Ether	11 x Pb, Sb, Ba; 3 x Sb, Ba; 34 x Pb, Sb; 3 x Pb, Ba
Acetone-Water	58 x Pb, Sb, Ba; 46 x Pb, Sb; 4 x Pb, Ba
Acetonitrile-Water	6 x Pb, Sb, Ba; 8 x Pb, Sb

Table 5.10 Effect of water in the swabbing solvent

Given the random nature of FDR deposition and particle recovery there is insufficient evidence to draw any conclusions from this test, although the presence of water does not appear to have a noticeable detrimental effect.

The next test involved the distribution of lead between two solvents, namely petroleum ether and water, in an attempt to determine the effect of water on the level of lead in FDR, lead being chosen because it is present in FDR at a much higher level than either antimony or barium.

Before use both the petroleum ether and deionised water were analysed for Pb with negative result. The results are given in Table 5.11.

Sample	*Test No.*	*Initial (ng)*	*48 Hours (ng)*	*Difference (ng)*
Petroleum	1	2700	1900	800
Ether	2	1900	275	1625
Layer	3	4025	3750	275
Water	1	None	None	None
Layer	2	None	100	100
	3	None	50	50

Table 5.11 Lead distribution between layers

Again there is insufficient evidence to draw conclusions, and the test results are difficult to explain. The lost lead from the petroleum ether layer could have been adsorbed on to the surface of the separating funnel and/or concentrated at the petroleum ether/water interface. A small amount of Pb did enter the water layer in tests 2 and 3 but none in test 1. This could be due to a small proportion of the discharge residue containing a water soluble lead compound or a small number of insoluble Pb containing particles finding their way into suspension in the water layer.

A further test involved repeatedly treating a sample with water prior to carbon coating for manual SEM/EDX examination, with a duplicate, untreated sample acting as a control.

When examined, both samples had a high concentration of particles encompassing the complete range of particle types. The sample treated with water did not show any noticeable difference.

There is nothing to indicate that water has a significant chemical effect on the particles. It is likely that water, in the form of rain, would substantially decrease particle population by physical disturbance.

5.2.4 Bullet fragmentation

As a result of a terrorist attack on a motor vehicle, in which the terrorists used 7.62 mm x 39 mm calibre, Yugoslavian nny 82 ammunition, the driver was shot dead. A large number of bullets struck the car, and the interior of the car and the clothing of the deceased suffered severe bullet fragmentation damage. An item of clothing worn by the deceased was examined for FDR, not as a requirement of the case but to gain background knowledge of the types of particles originating from bullet fragmentation.

Examination revealed that the sample contained both spherical and irregular particles, although the vast majority were spherical. The spherical particles could originate from the considerable heat generated when a high velocity bullet strikes a hard surface, such as vehicle glass or bodywork (107).

Numerous Pb, Sb particles were detected accompanied by Cu, Zn particles, Fe particles, and Pb only particles. The Pb, Sb; Cu, Zn; and Pb only particles probably originated from the bullets and the Fe particles probably originated from the car bodywork. No unique FDR particles or other FDR particle types were detected.

If it was required to examine a person for FDR who had been subjected to bullet fragmentation, the presence of such large numbers of particles originating from fragmentation would make the task very difficult. In this instance no unique FDR particles were detected. However it is possible that all types of FDR particles could be carried on the surface of the bullets, and this possibility would have to be carefully considered in this type of examination.

5.2.5 RPG7 rocket launcher

The Soviet RPG7 anti tank rocket launcher using a PG7 tank rocket has been used during the terrorist campaign in Northern Ireland. It is a long weapon which sits on top of the shoulder when in use and 'exhausts' to the rear of the firer. After incidents in which the RPG7 was used, the laboratory was requested to examine swabs and clothing from suspects, for discharge residue from the launcher. Due to the fact that the 'exhaust' from the weapon emerges a considerable distance to the rear of the firer, and the mechanism involved in use, it was considered unlikely that residue would be present on the firer. To determine whether or not it was worthwhile examining swabs and clothing from suspects, a test was conducted and discharge residue particles from the RPG 7 were examined. Discharge residues detected on the 'firer' of the RPG 7 are given in Table 5.12. Nothing of significance was detected on the control sample.

Size μ	*Shape*	*Major*	*Minor*	*Trace*	*No. Particles*	*Comments*
3.0	Sphere	Pb	Si	Cu, Ca, Cl, Al	7	2 x Fe, S, trace
3.0	Triangle	Zr, Si	Ca	Fe, K, Cu, Cl	2	From primer?
2.0 x 5.0	Oval	Pb, Si	Ca, K, Al, Fe	Cu, Cl, Ti, Mg	1	
2.0	Sphere	Cu, Pb	Sb, Fe, Zn	Al, Si, Cl, K	1	
10.0	Spherical	Pb	Ba, Cr, Fe, Ca	Cu, Si, Al	1	
3.5	Sphere	Pb, Ca	Si, Fe	Al, K, Mg, P, Cu	10	
2.0	Spherical	Sb, Sn	Cu	Fe, Si, Cl, S, Al, Mg	3	Tin present
1.5	Spherical	Pb, Si, Ca	Cl, Fe, Ti, Mg, K	Al, Cu, Zn	1	
2.5	Spherical	Pb, Ca	Ba, Si, Cl, Fe, K	Al, Mg, Cu, P, Zn	2	
1.5	Oval	Pb	Cr, Ti, Ca, Si, Zn	Cu, Cl, K, Al, Na	1	Zn > Cu
3.0	Sphere	Pb, Fe	Ca, Si, Al, Cl	Ti, Cu, K, Mg	4	
2.5	Oval	Pb, Cl	Ca, Si, Cu, Fe, K	Al, Zn, Ba, Mg	6	1 x Fe major
3.0	Double sphere	Pb, Si	Ba, Ca, Fe, Zn	Al, Mg, K, Cu, Cl	1	Zn > Cu
4.0	Spherical	Pb, Ca,	Si, K, Cu, Fe	Al, Mg	21	
1.5	Sphere	Sb	Fe, Cl, Si, Al	S, K, Cu, P, Mg	3	
2.5	Oval	Pb	Ba, Si, Ca, Fe, K	Al, Zn, Cu, Mg	2	Zn > Cu
1.5	Spherical	Pb	Ca, Cr, Si, Fe	K, Cu, Zn, Al, Mg	1	
3.5	Oval	Zn	Fe	Al, K, Si	3	Zn only
3.0	Spherical	Fe	Si	Cr, Al, P, Ti	6	

Table 5.12 Discharge residue from RPG7 rocket launcher

Discharge residue particles remaining in the launcher were also examined and the results are given in Table 5.13.

Sample	*Size μ*	*Shape*	*Major*	*Minor*	*Trace*	*No. Particles*	*Comment*
Booster Propellant	3.5	Diamond	Pb	K, Ca, Co, Fe, Zn	Cu, Si, Al, P, Mn	6	Zn > Cu
	3.0	Spherical	Si, S, Pb, K, Ti	Fe, Ca, Co, Sr	Cu, Zn	2	Sr from tracer?
	7.0	Spherical	K	Si, Pb, Ca, Fe, Co	Zn	1	Zn only
Front End and outside	5.0	Rounded	Ba, Ti	-	-	5	Ti from paint?
	3.5	Globular	Si, Ti	S, K, Ca, Sr	Fe, Cu, Zn	8	Sr from tracer?
Primer	2.0	Spherical	Ba, K, S	Al, Si, Fe, Cu	Pb	14	
	3.0	Sphere	Ba, K, S, Cr	Al, Si, Ca, Cu	Zn, Pb	2	
	2.5	Spherical	Zr	Si, K, Ca	Fe, Cu	2	Zr powder?
	1.5	Sphere	Al, Si, S, Pb, K	Ba, Fe, Cu	-	5	
	4.0	Sphere	Co, Si, Pb	K, Ca, Fe	Cu	2	Co from propellant?
	1.5	Spherical	Pb, S, K	Al, Si	Fe, Cu	11	
	2.5	Spherical	S, Pb, Co	Al, Si, K, Ca	Fe, Cu, Zn	6	Co from propellant?
	4.5	Oblong	S, Pb, Co	Al, Si, Fe	Cu, Zn	5	Co from propellant?
	3.0	Irregular	Co	-	K, Ca	4	Co from propellant?
	12.0	Irregular	Si, Ba	Cu, Sr, Al, Si	K, Ca, Fe	1	Sr from tracer?
	3.0	Spherical	Hg	K, Ca, Sb, Fe	Cu	3	From primer?

Table 5.13 Residue in discharged warhead

The PG7 rocket is known to contain the following:- black powder; a mix containing RDX explosive, hydrocarbon wax and an orange dye; PETN explosive; a mercury fulminate primer containing Zr (see particles in Table 5.13); an ignition powder containing barium nitrate, barium peroxide, magnesium, phenol-formaldehyde; a tracer composition containing strontium nitrate, magnesium, polyvinylchloride, phenol-formaldehyde with a yellow dye; an initial propellant charge containing NC, NG, EC, DBP and a rocket propellant containing NC, NG, TNT (with some DNT), DPA, EC, DBP, hydrocarbon and salts of Pb and Co. In addition the assembly contains steel, Al, and Sn coated Cu parts, and is painted an olive drab colour with black markings.

Apart from the possibility of residue being deposited on the firer in the act of firing there is also the possibility that subsequent handling of the launcher could yield distinctive residue. It is worthwhile examining the suspect for such residue.

5.2.6 Discharge residue from black powder ammunition

In previous casework in which the majority of the FDR particles contained K and S, frequently at high levels, it was thought that the ammunition responsible probably contained black powder. In most of the cases the type of ammunition was not known, whereas in others the sampling and analysis of the residue from the interior of the spent cartridge cases, confirmed the presence of black powder.

This posed the question "does K and S always occur, often at high level, in discharge residue particles from ammunition loaded with black powder"? In other words, from the presence and levels of K and S can it be accurately predicted when black powder is used?

A selection of old ammunition was tested to confirm that the propellant was black powder. Results of a representative selection of analyses are presented in Table 5.14.

Ammunition	*Major*	*Minor*	*Trace*	*Comment*
UMC 32-20	K, Pb, S	-	-	
	Pb, S, K	-	Si	numerous
	Pb, S	K	Si	
	Pb, S, K	-	Si, Zn	
	Pb, S	-	K, Si, Zn	
	Hg, K	-	Si, Cu, Zn	
	K, Hg	-	Cu, Si	
	Pb, S	-	K, Si	
RWS .320	K	-	Pb, S, Cu	
	K	S	Pb, Si	
	K	-	Pb, Cu	
	S, K	-	Pb, Si	
	Pb, S, K	-	Si, Cu	numerous
No headstamp 297/230 Morris	K	-	S	
	S	-	K	numerous
	K, S	-	Cu	
RWS .380	Pb, S, K	-	Si	numerous
	S, Sb, K	-	Si	
	Pb, S	K	Si	
RWS .450	S, Sb, K	-	Si	
	Pb, S	K	Si	
	Pb, S, K	-	Si	numerous
	S, Sb, K	-	Si	
	Pb, S, K	-	Cu, Zn, Si	
Eley London .450	K, Pb, S	-	Si	
	Pb, S, K	-	Si	numerous
	K, Sb	-	S, Si	
	K, S	Pb	Si	
	S, Sb	K	Si	

Table 5.14 Analysis of unburnt black powder

It is interesting to note the presence of Pb, Sb and Hg in some of the analyses. The Hg is almost certainly from the primer whereas the Pb and Sb could originate from two sources, the base of the bullet or the primer. However, if they originate from the bullet it would be expected that they would occur together and that the Pb level would be significantly greater than the Sb level. This suggests that the Pb and Sb also originates from the primer.

Discharge residue particles from black powder ammunition were then examined. Table 5.15 gives representative results.

Ammunition	*Size μ*	*Shape*	*Major*	*Minor*	*Trace*	*Comments*
UMC 32-20	5.0	Oval	Pb, S	-	Si	Numerous
	4.0	Kidney	Pb, S, Ba, Sb	-	Si, Fe, Cu	No Hg detected
	3.5	Sphere	Pb, S	-	Si, Cu, Sb	
	2.0	Oval	Pb, S, Si, Ba	Ca	K, Fe, Cu	(overall high
	2.0	Oval	Sb, Si	Pb, S	Ba, Cu, Fe	S, trace K)
RWS .320	8.0	Irregular	Pb, S	-	Si, Cu	Numerous
	4.0	Oval	K, S	-	Cu	
	3.0	Oval	Pb, S, K	Cl, Si, Ca	Fe, Cu, Ba	(Overall high
	5.0	Sphere	Sb, Pb, S, Fe	Cl, K	Si, Cu	K and S)
No headstamp 297/230 Morris	1.5	Oval	Pb, S	-	Si, Ti, Fe, Ca, Cu	Numerous
	4.0	Irregular	Fe	Cr	Ni	
	3.0	Oval	Pb, S	-	Si, Sb, Ti, K, Fe	
	3.0	Sphere	Fe, Pb, S, K	Ca	Si, Ti, Cu	(Overall high
	8.0	Oval	K, Si, Ba, Ca, Pb, S	Fe	Cu, Ti	K and S)
RWS .380	7.0	Oval	Pb, S	-	Si, Ca, K, Fe, Ti	Numerous
	5.0	Spherical	Pb, S, K, Ca	-	Si, Ti, Fe, Cu	
	3.5	Oval	Pb, S, Ba	Sb, K, Si	Fe, Cu	
	7.0	Kidney	Ba, Si, Ca	-	K, Fe	(Overall high
	3.0	Triangle	Pb, S	Sb, Si	K, Fe, Cu	S, low K)
RWS .450	10.0	Irregular	K, S	-	-	Numerous
	3.0	Spherical	Pb, S, K, Cl	Si	Fe, Cu	
	5.0	Oval	Cl, K	-	Si	
	12.0	Oval	Sb, Ba, Pb, S	Si	K, Cu, Fe	(Overall high
	12.0	Irregular	S, Pb, K, Fe	Cl	Cu, Zn	K and S)
Eley London .450	4.5	Spherical	K, S	-	-	Numerous
	2.0	Oval	S, Pb, Sb	Ba	Fe, Cu, Cl	
	8.0	Irregular	Pb, S, Cl	K, Ca, Si	Fe, Cu	
	2.5	Oval	S, Pb, K	-	Cl, Si	(Overall high
	2.0	Oval	S, Sb, Ba	Cl	K, Cu, Fc	K and S)

Table 5.15 Discharge particles from black powder ammunition

As antimony sulphide is widely used in primer compositions, S is frequently present in discharge residue particles and can occur at major, minor or trace level (see Table

5.05). Consequently the occurrence of S at major level is not an accurate indicator of the use of black powder. The particles should be considered as a group and it is clear that the frequent occurrence of both K and S at high level is strongly indicative of black powder. However, as can be seen from Table 5.15 the use of black powder does not necessarily yield overall high levels of K. Potassium does not normally occur at major level in FDR particles (see Table 5.05) and its presence at major level in any of the particles suggests the use of black powder.

A potential problem arises whenever black powder ammunition is used in close range shootings, in that the particulate matter deposited in the vicinity of the bullet hole is nondescript and does not resemble propellant. Consequently its significance may not be realised and it is also difficult to see on dark surfaces. If found it should be examined for K and S for confirmation of black powder.

5.2.7 Firearms coatings

The surface coatings of a random selection of firearms were examined and the results are given in Table 5.16.

Firearm	***Surface Appearance***	***Major***	***Minor***	***Trace***
Steyr Grand Rapide .308 Win	Grey, Matt	Fe, Mn, P	Ca, S	Si
FAL Rifle 7.62 x 51 mm	Black, Gloss	Si, Cl	P, Ca, Si, Ba, Ca	S, K, Fe, Ti, Na
Brno Mod 38 .22 Rimfire	Black, Gloss	Fe	-	Mn, S, Si
Sig Manurhin .243 Win	Grey, Matt	Mn, Fe, Ca	K	P, Si, S, Cu
FNC Rifle .223 Rem	Black, Matt	Fe	-	Mn
H&K MP5 SMG 9 mmP	Blue, Matt	Si	-	Mg
Webley Vulcan Air Rifle .22	Black, Gloss	Fe	-	Mn, S
Beretta O/U Shotgun 12 G	Black, Gloss	Fe	-	Mn, Cr, Ca, Si
Beretta 302 S/A Shotgun 12 G	Black, Gloss	S	Ni	Fe
Colt AR-15 Rifle .223 Rem	Grey, Matt	Fe, Mn, P	Ca, Cl, K, S	-
Sterling SMG 9 mmP	Black, Gloss	Si, Fe	P, S, Mn, Cl, Zn	K
MI Garand Rifle .30-06	Grey, Matt	Fe	Cr, K	S, Si
Baikal S/B Shotgun 12 G	Black, Gloss	Fe	-	S, Cl, Si
Aya D/B Shotgun 12 G	Black, Gloss	Fe	Mn	K
Gardone O/U Shotgun 12G	Black, Gloss	Fe	Mn, Si	S, Cl
Steyr 1904 Rifle 7.9 mm	Black, Gloss	Fe, Cl, K	Si	Mn, Cu, S
Walther Pistol .380 ACP	Black, Gloss	Fe	Mn, K, S, Si	Ca, Cl
S&W Mod.59 Pistol 9 mmP	Black, Gloss	Al	S, Cl, Si	K, Ca, Fe, Ni, Zn
S&W 15-4 Revolver .38 SPL	Black, Gloss	Fe	-	S
Browning Pistol 9 mmP	Black, Matt	Fe	-	K, Cl, Ca
Ruger Speed Six .357 Mag	Black, Gloss	Fe	-	Cr
Ingram SMG 9 mmP	Black, Matt	P, Zn	Fe, Sb, Ca, K	Si, S, Cl, Cu
Sussex Armoury Replica	Black, Matt	Si, Ba, S	Mg, Al	Fe, Cu
Webley & Scott .38 S&W	Black, Matt	Fe	S, K, Cl, Ca	Si, Cu
Colt Pistol .45 ACP	Black, Matt	Fe	S, Al	Cr, Mn, Cu, Cl, K
MI Carbine .30	Black, Matt	Si, S, Fe	Ca, Cl, K, Al	Ni, Mn, Cu
Franchi S/A Shotgun 12 G	Black, Gloss	Fe	Cl, S	K, Cu, Si
Webley & Scott S/B Shotgun 12G	Black, Gloss	Fe	-	Si, S, Cl, K, Ca, Cu
Lee Enfield Rifle .303	Black, Matt	Fe, Ca	K, S, Cl, Si	Cu, Zn
AR-180 Rifle .233 Rem	Green, Matt	P, Mn, Fe	-	Ca, Cu

Table 5.16 Firearm surface coatings

The elements detected in Table 5.16 do not fully reflect the wide range of elements mentioned in section 2.2.2.

It is possible that such coatings could, on rare occasions, make a contribution to the elemental content of some of the particles, or be deposited directly on to the hands from handling the firearm. Surface coatings have the potential to make a contribution, particularly from the cylinder gap area of revolvers and from the muzzle area of any type of firearm. These are the 'exterior' areas subjected to the hot propellant gases, which may erode the surface coating. Mixtures containing selenium are used to repair surface coatings of firearms, and particles containing selenium are occasionally encountered in casework. Such particles can provide useful additional evidence. Home-made firearms are frequently painted black using household paints. Such coatings can flake and leave paint flakes on the hands or clothing, particularly pocket interiors, and this can provide very useful evidence.

5.2.8 Homogeneity of propellants

To investigate the feasibility of conducting chemical comparisons between propellants detected in casework and suspect ammunition, it was decided to determine variations from grain to grain in a single round of ammunition and then to compare burnt (discharged) and unburnt propellants, to determine what, if any, difference was caused by the discharge process.

Analysis of 20 propellant grains from a single round of ammunition gave the following results. Diphenylamine and three dialkylphthalates were detected in all 20 grains whereas NG was detected in only 2 of the grains. Peak area ratios of DPA to each phthalate varied widely for each grain. This, and the fact that NG was detected in only 2 out of 20 grains in a single round of this particular ammunition shows a considerable compositional difference between grains of propellant. It is a possibility that this is a blended propellant. Further work would need to be done in this area to determine grain to grain and batch to batch variations in composition, in order to determine the feasibility of chemical comparisons in each particular instance.

A further test explored compositional differences between fired and unfired propellant grains. Results are given in Table 5.17 (ND = not detected).

Sample	***NG***	***EC***	***DPA***	***nDPA 1***	***nDPA 2***	***Tri Butyl Phos.***	***PHTHALATES***			
							P1	***P2***	***P3***	***P4***
A. Fired	Minor	Major	ND	ND	ND	ND	Trace	Major	ND	Minor
A.Unfired	Major	Major	Trace	ND	ND	ND	Trace	Trace	Minor	Trace
B.Fired	Minor	Trace	Major	Trace	Trace	ND	ND	Trace	Trace	Trace
B.Unfired	Minor	Trace	Major	Trace	Trace	ND	ND	Major	Trace	Minor
C.Fired	Trace	ND	Trace	ND	ND	ND	ND	ND	ND	ND
C.Unfired	Minor	Trace	Major	Trace	Trace	ND	ND	Minor	Minor	Minor
D.Fired	Minor	Trace	Major	Trace	Trace	ND	ND	Minor	Trace	Trace
D.Unfired	Minor	ND	Major	Trace	Trace	ND	ND	Minor	ND	Minor
E.Fired	Minor	Major	Minor	ND	ND	ND	ND	Major	ND	Trace
E.Unfired	Minor	Major	Minor	ND	ND	ND	ND	Major	ND	Trace
F.Fired	Minor	Minor	Major	Trace	Trace	ND	ND	Minor	ND	Trace
F.Unfired	Minor	Trace	Major	Trace	Trace	ND	ND	Major	ND	Major
G.Fired	Minor	Trace	Major	Trace	Trace	ND	ND	Major	ND	Minor
G.Unfired	Minor	Trace	Major	Trace	Trace	ND	ND	Minor	ND	Trace
H.Fired	Minor	Minor	Major	Trace	Trace	ND	ND	Major	ND	Trace
H.Unfired	Minor	Trace	Major	Trace	Trace	ND	ND	Major	ND	Trace
I. Fired	Major	Major	Trace	ND	ND	ND	ND	Major	ND	Trace
I. Unfired	Major	Major	Trace	ND	ND	Trace	ND	Major	ND	Trace
J. Fired	Minor	Major	ND	ND	ND	Trace	ND	Major	ND	Trace
J. Unfired	Minor	Major	ND	ND	ND	ND	ND	Minor	ND	Trace

Table 5.17 Comparison of fired and unfired propellants

Some ingredients such as EC may be present as a surface coating, as opposed to an integral component, and may be blown or burnt off during discharge. It is clear from this work that whatever the reasons for compositional differences, whether qualitative or quantitative, any interpretation based on compositional data needs to be approached with caution, particularly when only a small quantity is available for comparison. Any conclusive interpretations would need to be supported by a massive amount of background data.

5.2.9 Bullet hole perimeters

The sodium rhodizonate test for lead is routinely used in many forensic laboratories for confirmation of bullet damage and range of fire determinations. In a number of cases the test failed to indicate the presence of lead on the perimeter of holes that had a distinct bullet wipe. The bullets involved in these cases were all copper jacketed (FMJ). As the test is routinely used in this laboratory, the negative findings from known bullet holes caused concern. It was decided to instigate a project to assess the reliability of the sodium rhodizonate test for lead, and the validity of lead as an indicator of bullet damage. Also investigated was the bullet wipe pattern for shots fired from different angles, and the dependence of close range residue patterns on the ammunition used.

The first test involved single shot firings at a fixed distance, using the same gun and ammunition type, but varying the angle of the target and the angle of the firer to the target. A 'straight on' (0°) shot will produce a uniform circular hole and wipe, whereas a shot fired from an angle will produce an elongated bullet wipe and a somewhat irregular hole. One of the questions to be tested was 'does the size and position of the elongated wipe reliably indicate the angle of fire'?

Test results revealed that 62 of the 63 bullet hole perimeters gave a positive rhodizonate test for lead and it was concluded that the size of wipe produced by the bullet increases as the angle of fire increases e.g. a shot fired from 75° (left or right) will produce a larger wipe than a shot fired from 30° (left or right). Shots fired straight on at the target only produced an elongated wipe whenever the target was tilted.

There was a definite tendency for the size and position of the wipe to be reproducible for repeated firings under identical conditions, but in a few instances it varied markedly, without apparent reason. As the size and position of the wipe depends on the relative positions and attitudes of the firer and target, any conclusions about the direction of fire need to be very carefully considered.

The second test involved a series of single shot close range firings using a revolver, a pistol and a rifle, but varying the ammunition used. The objective was to determine the influence of the type of firearm and the type of ammunition on the muzzle blast residue pattern deposited on the target in close range shootings.

Results indicated that in each case the diameter and density of the unburnt propellant patterns were similar using the same gun but different ammunition. There were variations in the soot (blackening) deposits with different ammunition. Contact shots were very similar irrespective of the ammunition. All gave positive rhodizonate tests for lead.

Whilst it is always desirable to use the actual gun and the same ammunition type to do range tests for comparison with casework items, if the ammunition type is unknown and the firearm type is known, it is still possible to give a reasonable estimate of range. If both the gun and ammunition type are unknown i.e. at one extreme it could be a low power handgun and at the other it could be a high power rifle, then in these instances it is only possible to state that there is evidence of a close range shooting, give the upper limits for a handgun and a rifle and then give a rough estimate for each couched in terms such as 'not more than' and 'not less than'.

A final test was conducted to determine the reliability of the sodium rhodizonate test as an indicator of bullet damage.

This revealed that approximately 99% of the ammunition used in the pistols and revolvers gave positive rhodizonate tests on the perimeter of the bullet hole and approximately 94% of the ammunition used in the rifles gave positive rhodizonate tests. These results indicate that the sodium rhodizonate test for lead is reliable, and that lead is a good indicator of bullet damage and close range shootings. The results are better than those experienced in casework due to the fact that, in casework, many bullet hole perimeters are bloodstained and the blood could disturb the perimeter residues and have a masking effect, thereby hindering the removal of residue for testing. Despite this the test is effective for the vast majority of cases. The lower success rate with rifles is difficult to explain, but may well be a result of some of the lightly adhering residue on the bullet surface being lost due to the higher velocity (wind disturbance) before the bullet strikes the target.

Alternative tests for the identification of bullet holes and testing for close range shootings will need to be devised as the use of lead free ammunition increases (191).

Tests were also conducted to determine if it was possible to identify the bullet jacket material from examination of the bullet hole perimeter. The ammunition used is given in Table 5.18 and the test results are presented in Table 5.19. Nothing of significance was detected on the control samples of target cloth.

Test No	*Ammunition*			*Primer*
1	9 mmK Hirtenberg	Ni Jkt	FMJ	Pb, Ba
2	9 mmK Sako	Cu Jkt	FMJ	Pb, Sb
3	9 mmK W-W	Cu Jkt	FMJ	Pb, Sb, Ba
4	9 mmK Federal	Cu Jkt	FMJ	Pb, Sb, Ba
5	9 mmP VPT42	Cu Jkt	FMJ	Pb, Sb, Ba
6	9 mmP VPT43	Cu Jkt	FMJ	Pb, Sb, Ba
7	9 mmP VPT44	Cu Jkt	FMJ	Pb, Sb, Ba
8	9 mmP 11 52	Cu Jkt	FMJ	Sb, Hg
9	9 mmP K52	Cu Jkt	FMJ	Pb, Sb, Ba
10	9 mmP S044	Cu Jkt	FMJ	Pb, Sb, Ba
11	9 mmP GECO 80-59	Cu Jkt	FMJ	Pb, Sb, Ba
12	9 mmP Norma	Cu Jkt	FMJ	Pb, Sb, Ba
13	9 mmP REM-UMC	Cu Jkt	FMJ	Pb, Sb, Ba
14	9 mmP RG55	Cu Jkt	FMJ	Pb, Sb, Hg
15	9 mmP DI43	Cu Jkt	FMJ	Pb, Ba
16	9 mmP RG56	Cu Jkt	FMJ	Pb, Sb, Hg
17	9 mmP RG57	Cu Jkt	FMJ	Pb, Sb, Hg
18	9 mmP WRA	Cu Jkt	FMJ	Pb, Sb, Ba
19	.45 ACP R-P	Cu Jkt	FMJ	Pb, Sb, Ba
20	.45 ACP W-W	Cu Jkt	FMJ	Pb, Sb, Ba
21	.45ACP SF57	Cu Jkt	FMJ	Sb, Hg
22	.45ACP WRA. Co	Cu Jkt	FMJ	Pb, Sb, Ba, Hg
23	.303 R↑L49	Cu Jkt	FMJ	Pb, Sb, Hg
24	7.62 NATO RG70	Cu Jkt	FMJ	Pb, Sb, Ba
25	.223 HP	Cu Jkt	FMJ	Pb, Sb, Ba
26	.223 Norma	Cu Jkt	FMJ	Pb, Sb, Ba
27	.223 IVI70	Cu Jkt	FMJ	Pb, Sb
28	.223 RA69	Cu Jkt	FMJ	Pb, Sb, Ba
29	.223 RA65	Cu Jkt	FMJ	Pb, Sb, Ba
30	.30MI Norma	Steel Jkt	JSP	Pb, Sb, Ba
31	.30MI R-P	Steel Jkt	JSP	Pb, Sb, Ba
32	.30MI W-W	Cu Jkt	FMJ	Pb, Sb, Ba
33	.30MI W-W	Cu Jkt	JHP	Pb, Sb
34	.30MI VE-F	Cu Jkt	FMJ	Sb, Hg
35	.30MI VE-N	Cu Jkt	FMJ	Sb, Hg
36	.455 Dominion	Pb	Unjacketed	Pb, Sb, Ba, Hg
37	.455 Kynoch	Pb	Unjacketed	Pb, Sb, Ba

Test No	Ammunition			Primer
38	.455 K62	Cu Jkt	FMJ	Pb, Sb, Ba, Hg
39	.357 W-W	Cu Jkt	JHP	Pb, Sb, Ba
40	.357 R-P	Cu Jkt	JSP	Pb, Sb
41	.357 W-W	Pb	SWC	Pb, Sb, Ba
42	.357 R-P	Pb	SWC	Pb, Sb, Ba
43	.38 S&W R↑L39	Ni Jkt	FMJ	Pb, Sb, Hg
44	.38 S&W Norma	Pb	Unjacketed	Pb, Sb, Ba, Hg
45	.38 S&W REM-UMC	Pb	Unjacketed	Pb, Sb, Ba, Hg
46	.38 S&W Browning	Pb	Unjacketed	Pb, Sb, Ba
47	.38S&W GECO	Pb	Unjacketed	Pb, Sb, Ba

Table 5.18 Bullet hole perimeter test ammunition

Sample No. Jkt. Material	***Pb ng***	***Sb ng***	***Ba ng***	***Cu ng***	***Ni ng***	***Hg ng***	***Comments***
1 Ni	2600	None	160	600	None	None	No Ni, low Pb and Ba
2 Cu	>10000	None	500	1150	None	None	No Sb. Ba present
3 Cu	>10000	38	1690	1500	3700	None	High Ni
4 Cu	>10000	None	None	None	None	None	Pb only detected
5 Cu	8300	None	850	>5000	None	None	No Sb
6 Cu	>10000	None	560	4075	2850	None	No Sb, High Ni
7 Cu	9350	30	410	4650	None	None	Pb and Ba present
8 Cu	9000	None	650	>5000	None	63	No Sb. Ba present
9 Cu	3950	None	520	>5000	None	None	No Sb.
10 Cu	8875	30	520	4750	None	None	
11 Cu	4400	20	1460	2200	None	None	
12 Cu	>10000	46	670	2600	None	None	
13 Cu	>10000	36	700	2550	None	None	
14 Cu	>10000	33	540	4175	2000	160	Ba present, high Ni
15 Cu	>10000	None	830	>5000	1900	None	High Ni
16 Cu	9500	41	580	4100	None	176	Ba present
17 Cu	3850	66	130	3100	None	286	Ba present
18 Cu	3830	75	400	>5000	None	None	
19 Cu	>10000	167	2000	>5000	1725	None	High Ni
20 Cu	>10000	137	1470	3275	None	None	
21 Cu	5150	102	650	4890	2300	>500	Pb and Ba present, high Ni
22 Cu	9220	None	>2000	>5000	None	>500	No Sb
23 Cu	>10000	52	None	>5000	None	>500	
24 Cu	>10000	None	390	>5000	None	None	No Sb
25 Cu	2150	None	160	>5000	None	None	No Sb, low Pb and Ba
26 Cu	5450	None	450	2490	None	None	No Sb
27 Cu	5000	None	740	1850	2100	None	No Sb, Ba present, high Ni
28 Cu	3375	29	450	1400	None	None	
29 Cu	4900	None	>2000	4750	2250	None	No Sb, high Ni
30 Steel	2950	None	1270	>5000	2150	None	No Sb, high Ni
31Steel	>10000	55	1050	>5000	2150	None	High Ni
32 Cu	8800	30	>2000	>5000	None	None	
33 Cu	5900	32	720	>5000	None	None	Ba present

Sample No. Jkt. Material	***Pb ng***	***Sb ng***	***Ba ng***	***Cu ng***	***Ni ng***	***Hg ng***	***Comments***
34 Cu	4700	>200	900	>5000	None	155	Pb and Ba present
35 Cu	850	>200	550	>5000	None	>500	Pb and Ba present
36 Pb	>10000	>200	>2000	3950	None	>500	
37 Pb	>10000	>200	>2000	3325	None	None	
38 Cu	>10000	>200	>2000	>5000	2850	>500	High Ni
39 Cu	>10000	None	240	2200	None	None	No Sb
40 Cu	7300	None	200	1100	1600	None	No Sb, Ba present, high Ni
41 Pb	>10000	>200	520	None	None	None	
42 Pb	>10000	None	835	None	3675	None	No Sb, high Ni
43 Ni	>10000	None	390	>5000	3250	>500	No Sb, Ba present, high Ni, Cu
44 Pb	>10000	>200	1830	>5000	2850	>500	High Ni
45 Pb	>10000	174	1770	3300	4750	>500	High Ni
46 Pb	>10000	174	700	900	None	None	
47 Pb	>10000	130	1370	1675	None	None	

Table 5.19 Elemental levels on perimeter of bullet hole

The residue on the surface of a discharged bullet appears to originate from the base of the bullet itself, from the primer and from inorganic additives to the propellant. Firings numbered 8, 21, 34 and 35 had lead free primers yet lead was detected on the perimeter of the bullet holes. Ammunition with barium free primers gave barium on the perimeter.

Only one of the two nickel jacketed bullets, number 43, gave nickel on the perimeter of the bullet hole. Nickel was frequently detected from non-nickel coated bullets. This is a surprising result which demonstrates that the presence of nickel cannot be used to identify the use of a nickel jacketed bullet. The origin of the nickel is unknown but it may have originated from the primer cup coating.

It is interesting to note that in all tests in which mercury was present in the primer, it was detected on the perimeter of the bullet hole. The unjacketed lead bullets all gave a large quantity of lead on the perimeter, although this was not confined to unjacketed bullets. The copper results were similarly confusing.

Overall the possibility of determining the bullet jacket material from the residue around the bullet hole does not appear to be feasible using FAAS. However FAAS reliably detects elements associated with firearms discharge on the perimeter of the bullet hole and is a very useful method for confirming bullet damage.

5.2.10 Persistence

An obvious trend over the past 26 years is the decreasing percentage of Northern Ireland casework that is positive for FDR. During this period substantial improvements have been made in the efficiency of sampling and in the sensitivity of the detection techniques. Despite this the downward trend continued. Our success rate decreased from approximately 35% at the start of the terrorist campaign in 1969 to about 6% (excluding suicides and dead suspects) in 1994. The reason for the current limited success rate is not the detection system but rather the careful planning of terrorist incidents and the precautions they take to prevent leaving any type of forensic evidence at a scene or on their person. Coupled with this is the unfavourable behaviour of firearms discharge residue particles once they are deposited on a suspect. The particles are small and lightly adhering, and as such can become airborne again and be transferred from surface to surface by physical contact. They are lost rapidly from the hands, an order of magnitude in the first hour, and consequently the detection of residue on the hands suggests very recent contact (excluding suicides and dead suspects). They persist longer on clothing surfaces, the length of time depending on the nature of the material and the extent of physical disturbance of the garment. The particles are chemically stable. This was confirmed by an experiment involving an FDR contaminated garment which was packaged, sealed and stored for two years. FDR particles were readily detected on the garment surface after the lengthy storage period.

Another persistence experiment involved prompt sampling of the firing hand after firing. Numerous FDR particles were detected on the firing hand. The experiment was repeated but the firer was allowed to dry wipe his hands on tissue, in an effort to remove any FDR particles, prior to sampling, Very few particles were detected and it was concluded that FDR particles can easily be removed from the hands, even by dry rubbing.

Statistics gathered from 15 years casework results gave the following persistence data. Figure 5.02 illustrates the situation for suspects whose hands, face and head hair was sampled for FDR, resulting in the detection of particles on all or some of the samples. Suspects are rarely apprehended immediately. The data is based on 410 positive swab kits and excludes suicides and dead suspects.

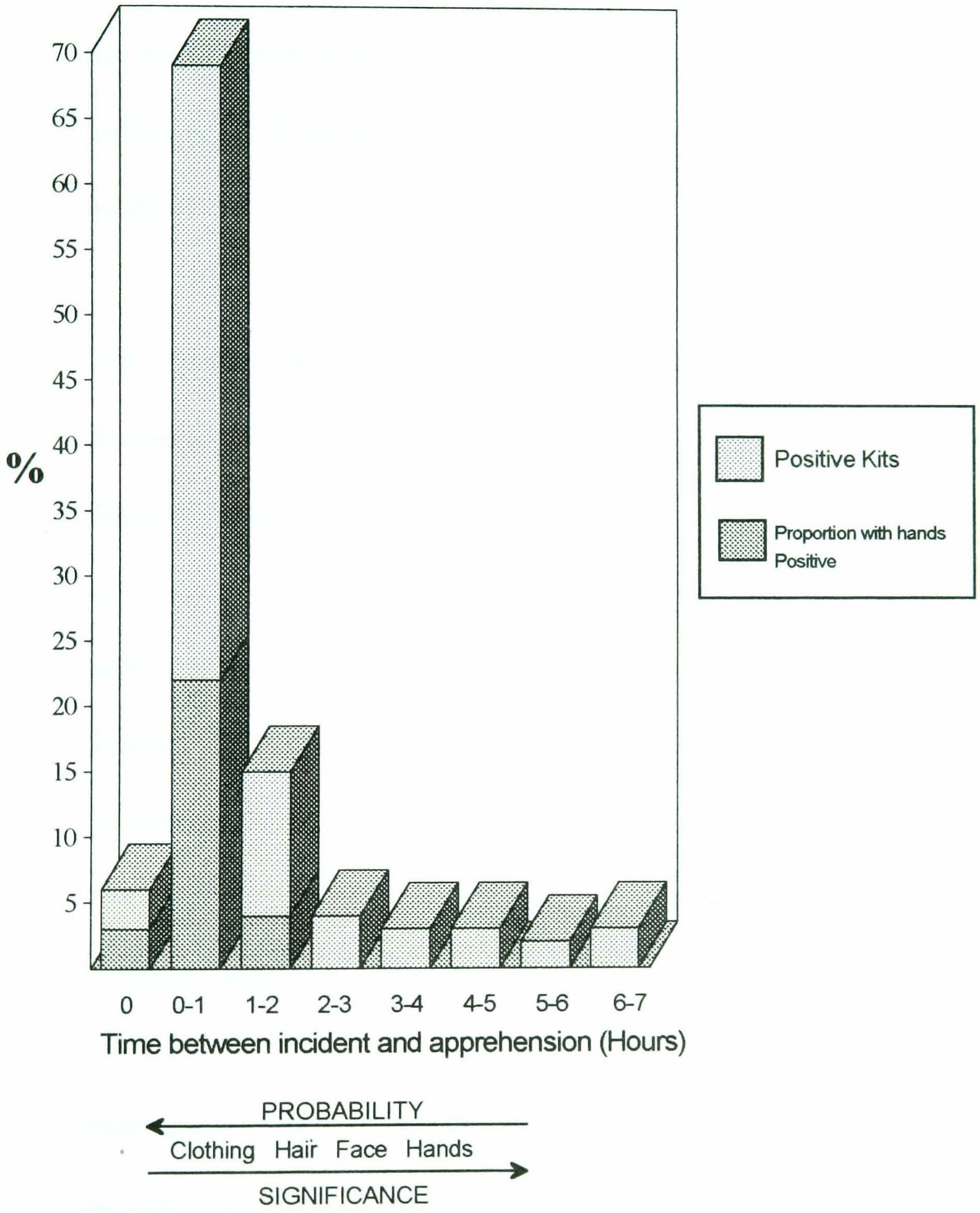


Figure 5.02 Persistence of firearms discharge residue

It is difficult to produce valid persistence data for clothing other than to say that it is our most fruitful sampling area. As stated previously, persistence on clothing depends on the nature of the material and the degree of physical disturbance the garment suffers. FDR will remain indefinitely on clothing if the clothing is undisturbed.

Suspects have been known to either abandon or destroy clothing worn at the time of the incident and change into 'clean' clothing. Consequently it is often not known for certain if the clothing submitted to the laboratory was the clothing worn during the incident.

FDR has been detected on the clothing of a suspect up to 6 days after an incident, but the history of the clothing was not known; consequently the residue could have been deposited since the original incident. On the other hand residue found on a garment could have originated from an incident prior to the incident under investigation. This highlights the problems encountered when interpreting positive results on clothing. Residue detected on the hands, face or head hair, because of known persistence, can be assumed to be recently deposited whereas on clothing (particularly pocket interiors) it is difficult to link it to a specific shooting incident. However it is still valuable evidence requiring an explanation from the suspect.

FDR is not found on the hands if the time between the incident and apprehension exceeds two hours (the suspects hands must be protected immediately they are apprehended). The police are instructed not to sample the hands if the two hours are exceeded but to take the face and head hair samples as normal. FDR has been detected on the face up to 5 hours and on the head hair up to 7 hours after an incident.

5.2.11 Antimony free primers

As mentioned previously it was noted in casework that Yugoslavian 7.62 mm x 39 mm calibre nny 82 ammunition, produces discharge particles containing barium, despite the fact that there is no barium in the primer, although it is present in the propellant. This indicates that the propellant can make a contribution to the elemental content of the discharge particles. In some incidents involving the use of ammunition with antimony free primers, discharge particles containing antimony have been detected on suspects. The possibilities are that the antimony originated from the bullet, that the particles were due to contamination of the gun or ammunition from some previous firing, that the particles originated from some other incident in which the suspects were involved or that the suspects had been exposed to contamination between apprehension and sampling.

To clarify the situation it was decided to investigate the possibility that the antimony originated from the bullet.

Discharge residue particles originating from ammunition with Sb free primers and Sb hardened bullets were examined for the presence of Sb. Results are given in Table 5.20. Antimony was not detected on the control samples from the firearms and ammunition or on the hand control samples.

Firearm	*Ammunition Headstamp*	*Ammunition Details*	*Pb, Sb, Ba*	*Ba, Ca, Si*	*Pb, Ba*	*Pb, Sb*	*Ba only*	*Pb only*
.22LR Walther Pistol	U	Unjacketed. Brass wash on bullet. Pb only primer	ND	ND	ND	20	ND	36
9 mmP Star pistol	DI44	FMJ. Cu washed Fe jacket. Pb, Ba primer	7	19	15	2	ND	15
.30 MI Winchester carbine	LC68	FMJ. Cu washed Fe jacket. Pb, Ba primer	18	ND	12	2	6	22

ND = not detected

Table 5.20 Discharge particles from ammunition with antimony free primers

These results confirm casework observations and illustrate that the antimony in the bullet can make a substantial contribution to the discharge particle compositions. This is important as it has previously been assumed that ammunition with antimony free primers would not produce discharge particles containing antimony.

5.2.12 Analysis of a baton round

As a consequence of a case in which the suspect alleged that the FDR on his person originated from contact with the inside of a police vehicle, in which baton guns had previously been fired, it was necessary to conduct a detailed examination of the baton round and the crime ammunition.

Antimony was detected in the residue particles on the suspect and it was known that the baton round does not have Sb in the primer. However it was required to prove that there was no antimony in any part of a baton round. Analysis of the baton round revealed that the cartridge case was Al with trace amounts of Fe and Ag, and it was painted black with a white band. Analysis of the black paint showed the presence of Al, Ag, Br, Cl, Cr, Fe, K, Ni, S whereas the white paint revealed Ti only. The discharged primer residue gave Pb and Ba at major level, Al and Si at minor level and Sn and Cu at trace level. The primer cup was Sn with a trace of Cu and Ag, and the red lacquer on the exterior surface of the cup contained Si and Sn. The black powder propellant was housed in a plastic casing on which was painted a green dot. Analysis of the propellant revealed K and S at major level and Si and Fe at trace level, and the green paint gave Pb, S, K at major level, Cr, Cl at minor level and Ba, Ti, Fe at trace level. The plastic baton itself had Cl, Fe, Ba at major level and Ca, Si at trace level on its outside surface, and Cl at major level and Ca at trace level on its inside surface. The end cap was painted cream and analysis showed the presence of S, Si, Ca at major level with Fe, K, Ag, Al and Br at trace level. The lacquer on the cap showed Al, Br at major level, with Ag, Si, S, Ca, Fe at trace level.

No antimony was detected anywhere in the baton round. Test firing the baton gun and sampling the firer also failed to reveal the presence of antimony in the discharge particles. Tin was present in some of the discharge particles.

The crime ammunition was 7.62 x 39 mm calibre Yugoslavian nny 82. Analysis of the components gave the following results.

Propellant: Single base with DPA, EC, a phthalate plasticiser, camphor.

Unburnt - Si, Pb at major level, Ca, Cu, S at minor level, Ba, Fe, K at trace level

Burnt - K and S at major level

Cartridge case and primer cup: Cu with a trace of Zn.

Discharge primer residue: Sb, Sn, S, Cl, Hg, Cu, Zn, Fe, K, Ca)
(inside of primer cup)
) Elements listed
: Sb, Sn, K, Cl, Cu, S, Zn, Fe, Hg) in descending
(inside cartridge case)) order

Bullet jacket: Cu at major level with Zn at minor level and Fe, Cl at trace level

Bullet Core: Pb with trace levels of Sb, Si

Lacquer (primer): Pb at major level, Ti, Cr, Si at minor level, Fe, Mn, Cu, Cl, K, Ca at trace level

Black sealant between bullet and cartridge case: Cu, Pb, Cl at major level; Zn, S at minor level; Si, Sb, Fe, K, Ca at trace level

It is interesting to note that the primer appears to be based on mercury fulminate, antimony sulphide and potassium chlorate i.e. mercuric and corrosive, and that the ammunition was manufactured in 1982. There is no Pb or Ba in the primer yet discharge particles from this ammunition frequently contain Pb, Sb and Ba. The Pb and Ba must come from other components in the ammunition and/or from contamination in the firearm. Tin was also frequently present in the discharge particles and originates from the tin foil disc used to seal the primer cup in mercury fulminate primers.

The absence of Sb in the baton round discharge particles proved that the residue on the suspect did not originate from this source.

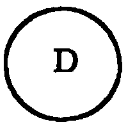
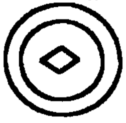
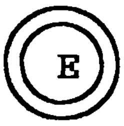
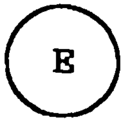
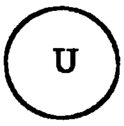
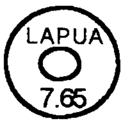
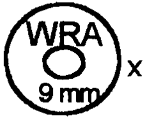
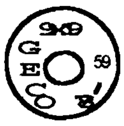
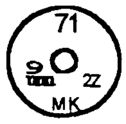
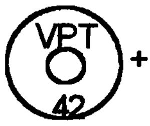
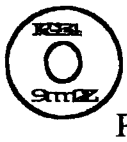
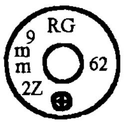
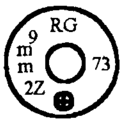
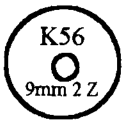
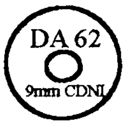
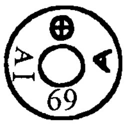
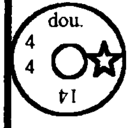
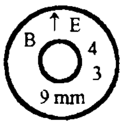
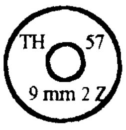
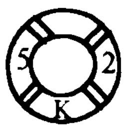
5.3 ANALYSIS OF AMMUNITION

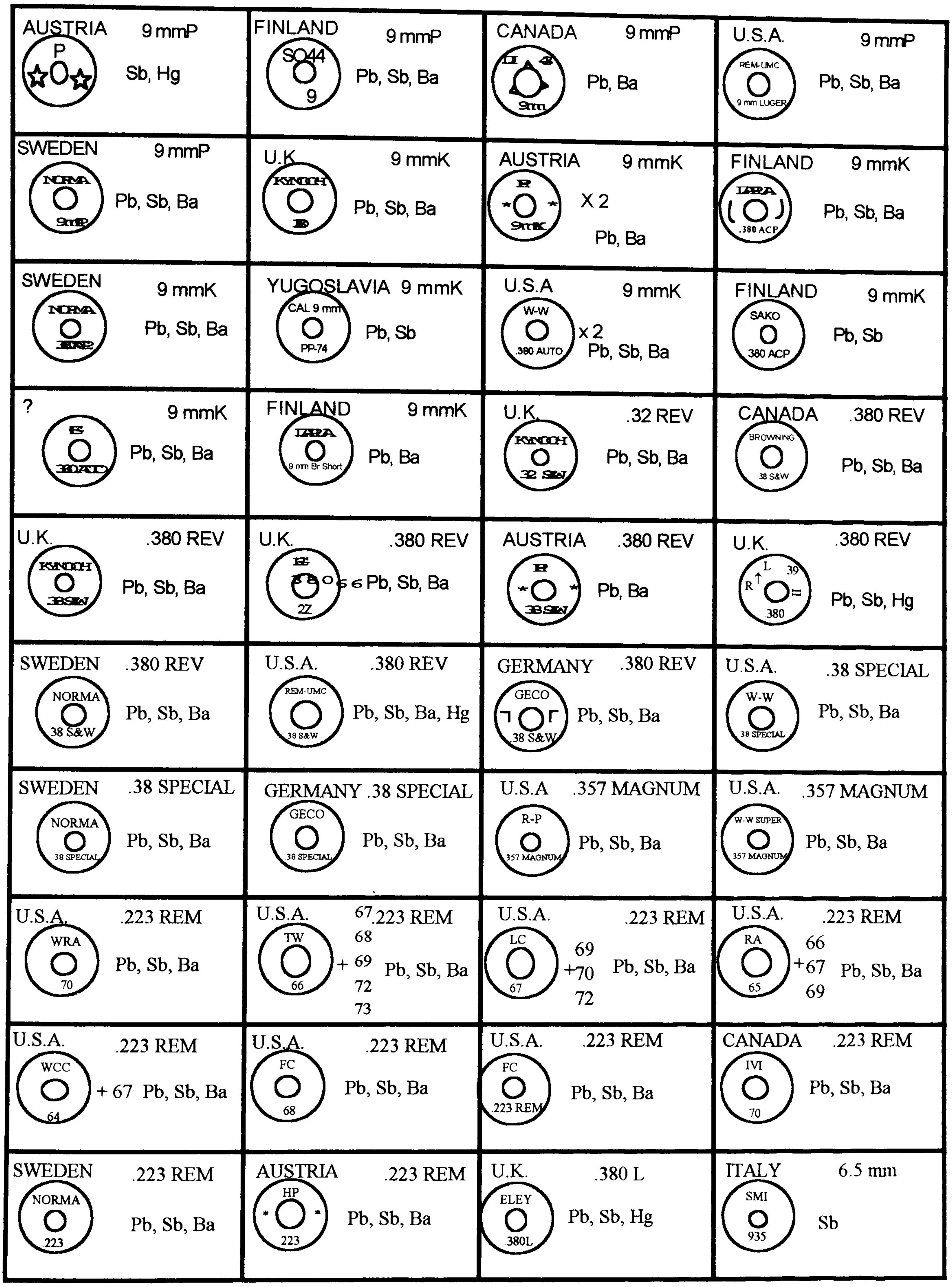
From the beginning of the terrorist campaign in 1969 to the end of April 1994 a total of 16,381 firearms, 82,168 spent cartridge cases and 1,667,115 rounds of ammunition have been recovered by the security forces, along with numerous miscellaneous firearms related items. The recovered firearms consist of 905 machine guns, 630 carbines, 4,871 rifles, 3,816 pistols, 3,414 revolvers, 2,196 shotguns and 549 miscellaneous firearms. Calibres range from .22" up to and including .50"/12.7 mm. During this period there were 10,995 shooting incidents and analysis of some of the recovered ammunition is discussed in this section.

5.3.1 Primer types

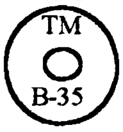
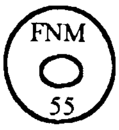
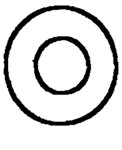
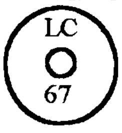
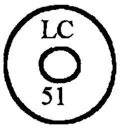
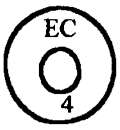
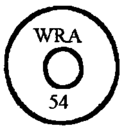
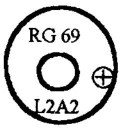
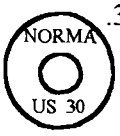
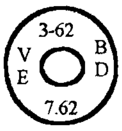
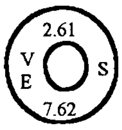
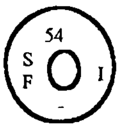
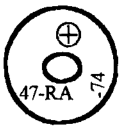
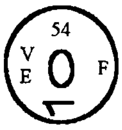
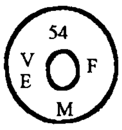
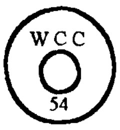
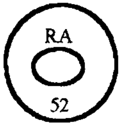
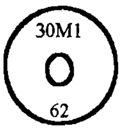
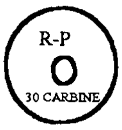
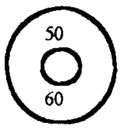
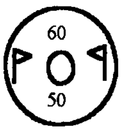
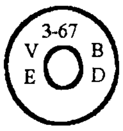
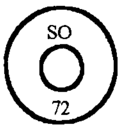
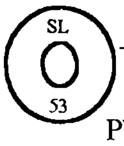
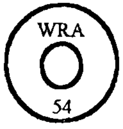
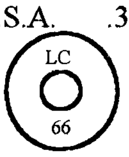
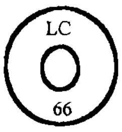
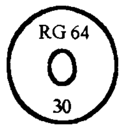
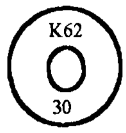
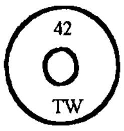
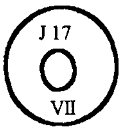
The interior of the spent cartridge case is routinely examined whenever FDR is detected on a suspect, to determine the type of primer involved in the incident. Figures

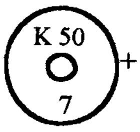
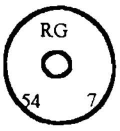
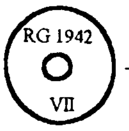
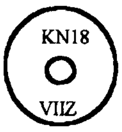
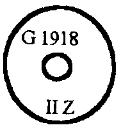
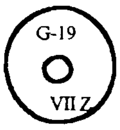
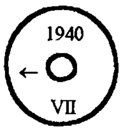
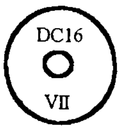
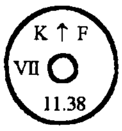
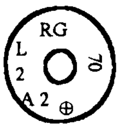
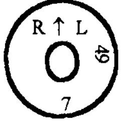
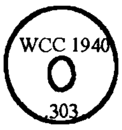
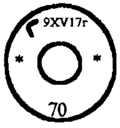
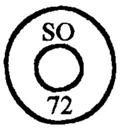
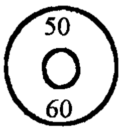
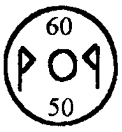
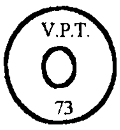
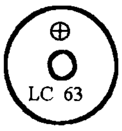
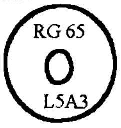
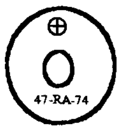
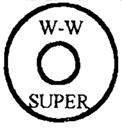
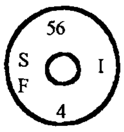
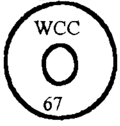
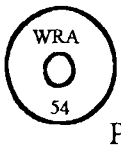
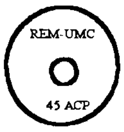
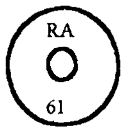
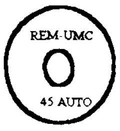
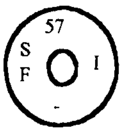
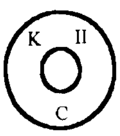
5.03 and 5.04 illustrate primer types as determined by FAAS and SEM/EDX examination respectively.

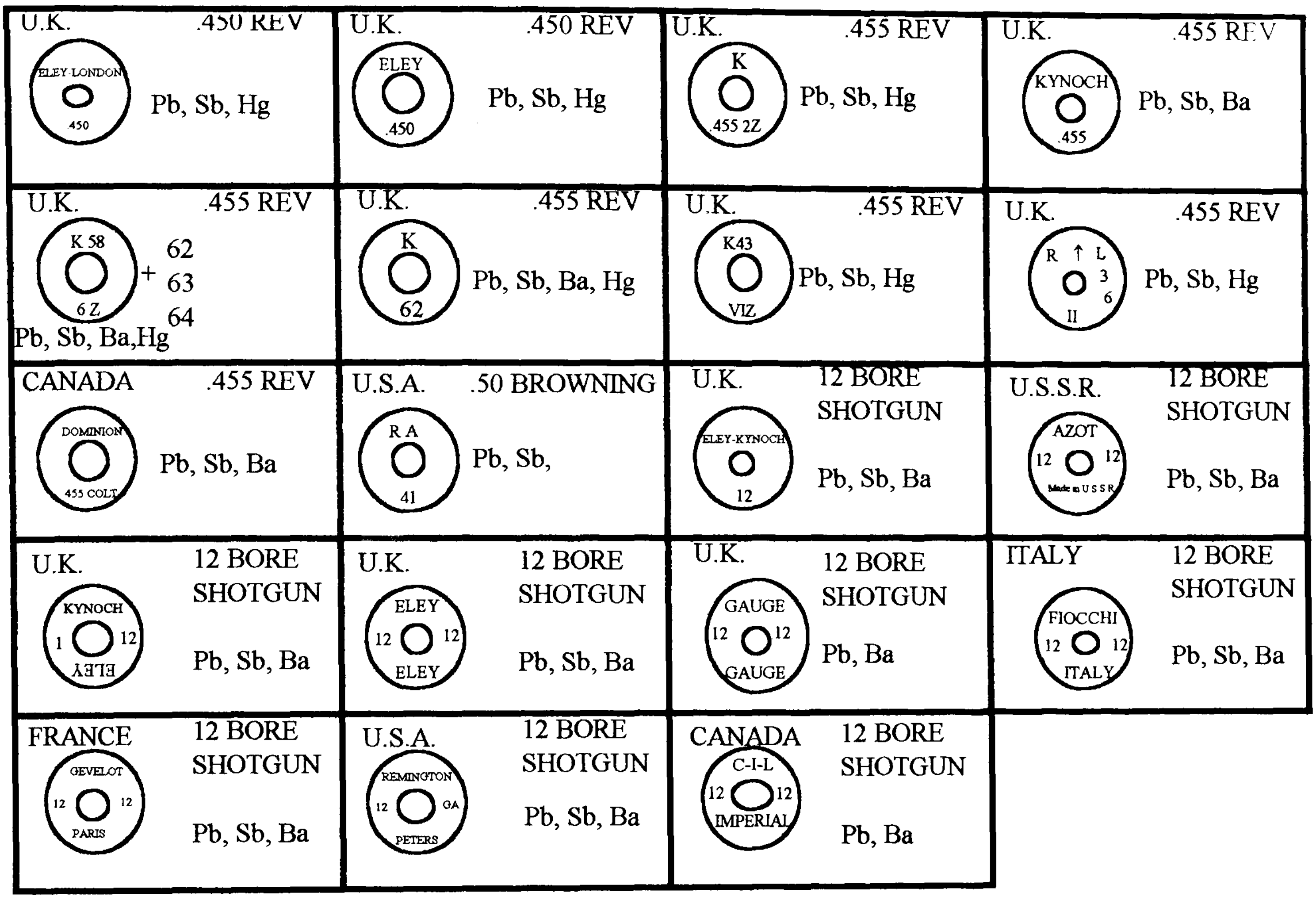
CANADA .22 LR  Pb, Ba	U.S.A. .22 LR  Pb, Ba	U.K. .22 LR  Pb, Ba	U.K. .22 LR  Pb, Ba
U.S.A. .22 LR  Pb, Sb, Ba	U.K. .22LR  Pb, Sb	U.S.A. .22 LR  Pb	FINLAND .32 ACP  Pb, Sb, Ba
U.K. .32 ACP  Pb, Sb, Ba	U.S.A. .32 ACP  Pb, Sb, Ba	FINLAND .32 ACP  Pb, Sb, Ba	CZECHOSLOVAKIA .32 ACP  Pb, Ba
AUSTRIA .32 ACP  Pb, Sb, Ba	SWEDEN .32 ACP  Pb, Sb, Ba	BELGIUM .32 ACP  Pb, Sb, Ba	GERMANY .32 ACP  Pb, Sb, Ba
U.S.A.. 9 mmP  x 2 Pb, Sb, Ba	CANADA 9 mmP  Pb, Sb, Ba	GERMANY 9 mmP  + 31-60 2-61 80-59 Pb, Sb, Ba	PORTUGAL (FOR ICI) 9 mmP  Pb, Sb, Ba
FINLAND 9 mmP  + 43, 44 Pb, Sb, Ba	U.K. 9 mmP  +55→59 Pb, Sb, Hg	U.K. 9 mmP  Pb, Sb, Ba, Hg	U.K. 9 mmP  +74→76 Pb, Sb, Ba
U.K. 9 mmP  Pb, Sb, Ba, Hg	U.K 9 mmP  + 58, 59 Pb, Sb, Ba	U.K. 9 mmP  Pb, Sb, Ba	YUGOSLAVIA 9 mmP  + 50, 52 Sb, Hg
U.S.A 9 mmP  Pb, Sb, Ba	CANADA 9 mmP  Pb, Sb, Ba	CANADA 9 mmP  Pb, Ba	CANADA 9 mmP  Pb, Sb, Ba
BELGIUM 9 mmP  + 68, 71 Pb, Sb, Ba	HOLLAND 9 mmP  Pb, Sb, Ba	CZECHOSLOVAKIA 9 mmP  Pb, Sb, Hg	U.K. 9 mmP  Pb, Sb, Hg
U.K. 9 mmP  Pb, Sb, Hg	ISRAEL 9 mmP  Pb, Ba, Sb, Hg	FRANCE 9 mmP  Ba, Sb, Hg	SWEDEN 9 mmP  Pb, Sb, Ba



AUSTRIA 9 mmP Sb, Hg	FINLAND 9 mmP Pb, Sb, Ba	CANADA 9 mmP Pb, Ba	U.S.A. 9 mmP Pb, Sb, Ba
SWEDEN 9 mmP Pb, Sb, Ba	U.K. 9 mmK Pb, Sb, Ba	AUSTRIA 9 mmK X 2 Pb, Ba	FINLAND 9 mmK Pb, Sb, Ba
SWEDEN 9 mmK Pb, Sb, Ba	YUGOSLAVIA 9 mmK Pb, Sb	U.S.A 9 mmK x 2 Pb, Sb, Ba	FINLAND 9 mmK Pb, Sb
? 9 mmK Pb, Sb, Ba	FINLAND 9 mmK Pb, Ba	U.K. .32 REV Pb, Sb, Ba	CANADA .380 REV Pb, Sb, Ba
U.K. .380 REV Pb, Sb, Ba	U.K. .380 REV Pb, Sb, Ba	AUSTRIA .380 REV Pb, Ba	U.K. .380 REV Pb, Sb, Hg
SWEDEN .380 REV Pb, Sb, Ba	U.S.A. .380 REV Pb, Sb, Ba, Hg	GERMANY .380 REV Pb, Sb, Ba	U.S.A. .38 SPECIAL Pb, Sb, Ba
SWEDEN .38 SPECIAL Pb, Sb, Ba	GERMANY .38 SPECIAL Pb, Sb, Ba	U.S.A .357 MAGNUM Pb, Sb, Ba	U.S.A. .357 MAGNUM Pb, Sb, Ba
U.S.A. .223 REM Pb, Sb, Ba	U.S.A. .223 REM 67 68 + 69 72 73 Pb, Sb, Ba	U.S.A. .223 REM 69 +70 72 Pb, Sb, Ba	U.S.A. .223 REM 66 +67 69 Pb, Sb, Ba
U.S.A. .223 REM + 67 Pb, Sb, Ba	U.S.A. .223 REM Pb, Sb, Ba	U.S.A. .223 REM Pb, Sb, Ba	CANADA .223 REM Pb, Sb, Ba
SWEDEN .223 REM Pb, Sb, Ba	AUSTRIA .223 REM Pb, Sb, Ba	U.K. .380 L Pb, Sb, Hg	ITALY 6.5 mm Sb

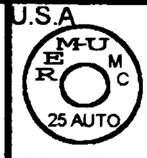
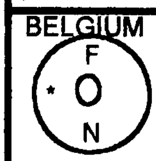
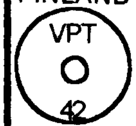
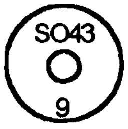
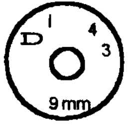
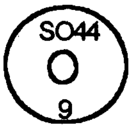
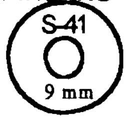
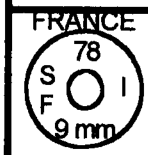
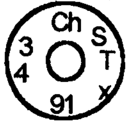
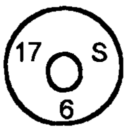
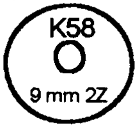
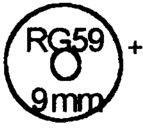
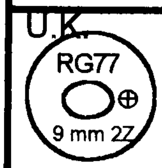
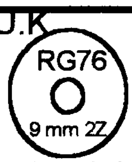
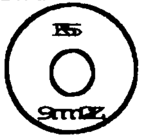
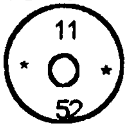
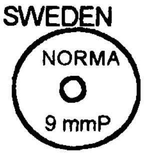
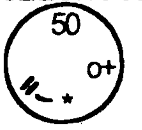
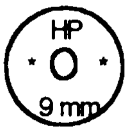
ITALY 7 mm CARCANO  TM B-35 Sb, Hg	ITALY 7 mm CARCANO  SMI 9-35 Sb	ITALY 7.35 mm  TM B-35 Sb, Hg	PORTUGAL 8 mm MAUSER  FNM 55 Pb, Sb, Hg
UNKNOWN 8 mm MAUSER  No Headstamp Sb, Hg	U.S.A. 30M1 CARBINE  LC 67 +68 69 Pb, Ba	U.S.A. 30M1 CARBINE  LC 51 +52 Pb, Sb, Ba	U.S.A. .30M1 CARBINE  EC 4 Pb, Sb, Ba, Hg
U.S.A. .30M1 CARBINE  WW 30 CARBINE Pb, Sb, Ba	U.S.A. .30M1 CARBINE  WRA 54 Pb, Sb, Ba	U.K. .30M1 CARBINE  RG 69 ⊕ L2A2 +70 Pb, Sb, Ba	SWEDEN (FOR U.S.A.) .30M1 CARBINE  NORMA US 30 Pb, Sb, Ba
FRANCE .30M1 CARBINE  3-62 V E B D 7.62 +3-67 Sb, Hg	FRANCE .30M1 CARBINE  3.59 V E N 7.62 +1.61 Sb, Hg	FRANCE .30M1 CARBINE  2.61 V E S 7.62 +2.67 Sb, Hg	FRANCE .30M1 CARBINE  54 S F I - Sb, Hg
NORWAY .30M1 CARBINE  ⊕ 47-RA .74 Pb, Sb, Ba	FRANCE .30M1 CARBINE  54 V E F Sb, Hg	FRANCE .30M1 CARBINE  54 V E F M Sb, Hg	U.S.A. .30M1 CARBINE  W C C 54 Pb, Sb, Ba
U.S.A. .30M1 CARBINE  RA 52 Pb, Sb, Ba	U.S.A. .30M1 CARBINE  30M1 62 Pb, Ba	U.S.A. .30M1 CARBINE  R-P 30 CARBINE Pb, Sb, Ba	U.S.S.R. .30-06  50 60 Sb, Hg
U.S.S.R. .30-06  60 50 Sb, Hg	FRANCE .30-06  3-67 V E B D Sb, Hg	FINLAND .30-06  SO 72 Pb, Sb, Ba	U.S.A. .30-06  SL 53 +54 57 Pb, Sb, Ba
U.S.A. .30-06  WRA 54 Pb, Sb, Ba	U.S.A. .30-06  LC 66 Pb, Ba	U.S.A. .30-06  LC 66 Pb, Sb	U.K. .30-06  RG 64 30 Pb, Sb, Ba
U.K. .30-06  K62 30 Pb, Sb, Ba	U.S.A. .30-06  42 TW Pb, Sb	U.S.A. .303  WRA 1941 Pb, Hg	U.S.A. .303  WRA 1943 .303 Pb, Sb, Hg
U.S.A. .303  WRA 303 BRITISH Pb, Sb, Ba	U.S.A. .303  WCC 1940 .303 Pb, Sb, Hg	U.K. .303  J 17 VII Sb, Hg	U.K. .303  K18 VIIZ Pb, Sb, Hg

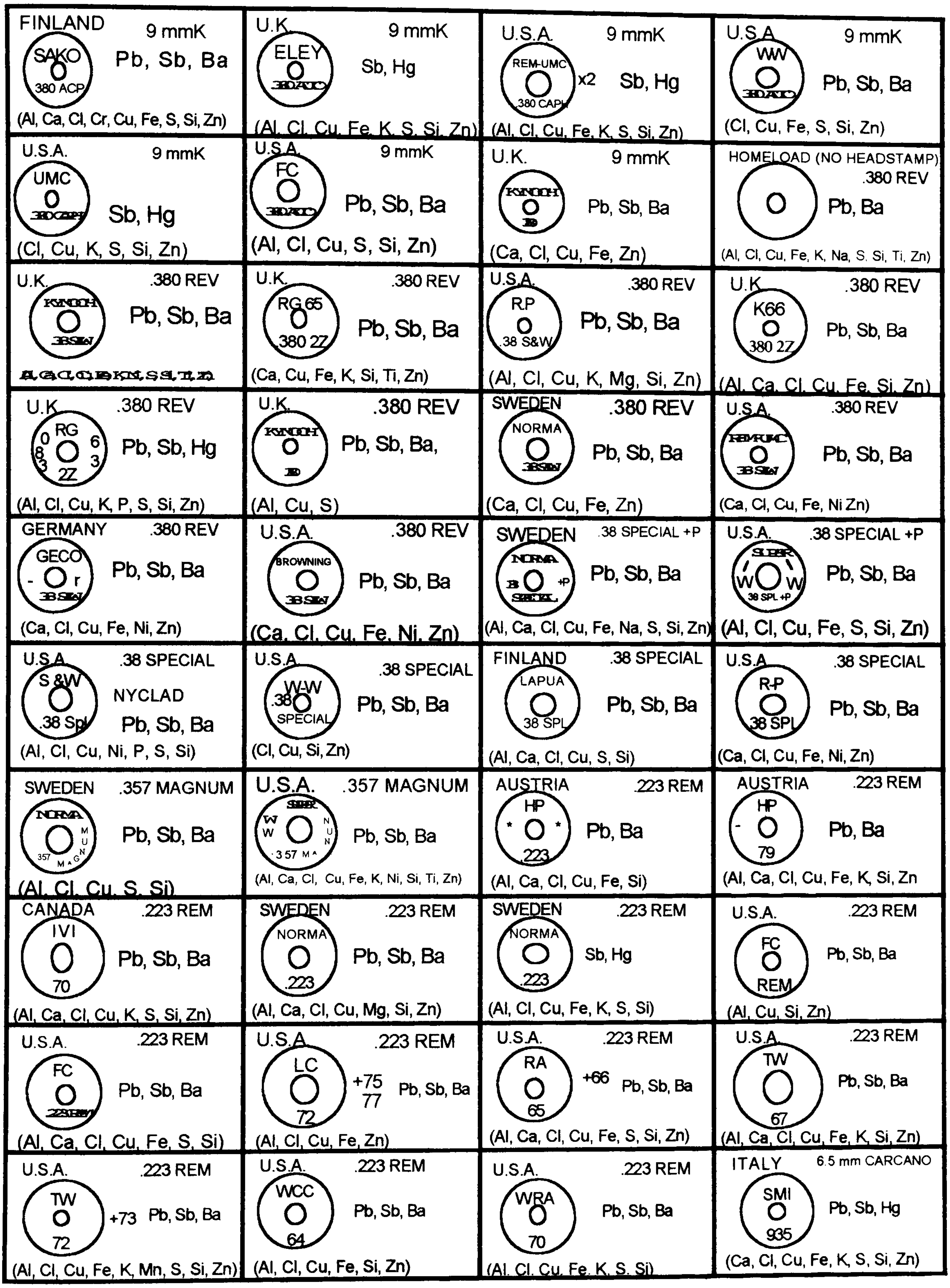
U.K. .303  52 +55 56 59 Pb, Sb, Hg	U.K. .303  Pb, Sb, Hg	U.K. .303  Pb, Sb, Hg	U.K. .303  +44 Pb, Sb, Hg
U.K. .303  Sb, Hg	U.K. .303  Pb, Sb, Hg	U.K. .303  Sb, Hg	U.K. .303  Pb, Sb, Ba, Hg
U.K. .303  Pb, Sb, Ba, Hg	CANADA .303  Pb, Ba, Hg	CANADA .303  Sb, Hg	PORTUGAL .303  Pb, Sb, Hg
INDIA .303  Sb, Hg	CZECHOSLOVAKIA .303  Sb, Hg	BELGIUM .303  Sb, Hg	U.K. .303  Pb, Sb, Ba
U.K. .303  Pb, Sb, Hg	U.S.A. .303  Pb, Sb, Ba, Hg	SYRIA 7.62 x 39 mm  Sb, Hg	FINLAND 7.62 X 39 mm  Pb, Sb, Ba
U.S.S.R 7.62 x 39 mm  Sb, Hg	U.S.S.R 7.62 x 39 mm  Sb, Hg	FINLAND 7.62 X 39 mm  Pb, Sb, Ba	U.S.A. 7.62 X 51 mm  Pb, Sb, Ba
U.S.A. 7.62 X 51 mm  Pb, Sb, Ba	U.K. 7.62 X 51 mm  66→70 +72, 73, 75 Pb, Sb, Ba	U.K. 7.62 X 51 mm (Tracer)  Pb, Sb, Ba	NORWAY 7.62 X 51 mm  Pb, Sb, Ba
U.S.A. .308 WIN. MAG.  Pb, Sb, Ba	FRANCE .45 ACP  +57 Sb, Hg	U.S.A. .45 ACP  + 69 71 Pb, Sb, Ba	U.S.A. .45 ACP  + 65 66 Pb, Sb, Ba
U.S.A. .45 ACP  Pb, Ba, Hg	U.S.A. .45 ACP  Pb, Sb, Hg	U.S.A. .45 ACP  Pb, Sb, Ba	U.S.A. .45 ACP  Pb, Sb, Ba
U.S.A. .45 ACP  Pb, Sb, Ba	FRANCE .45 ACP  Pb, Sb, Hg	U.S.A. .45 ACP  Pb, Sb, Ba	U.K. .450 REV  Pb, Sb, Hg



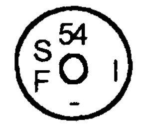
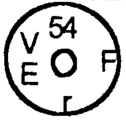
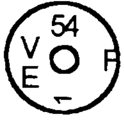
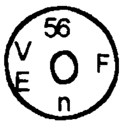
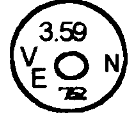
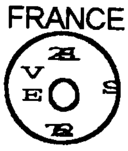
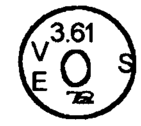
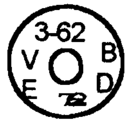
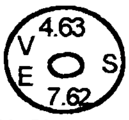
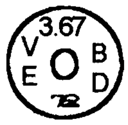
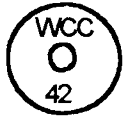
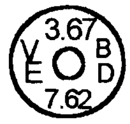
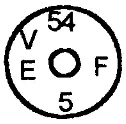
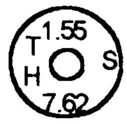
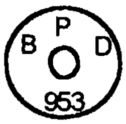
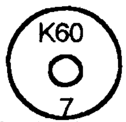
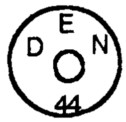
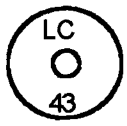
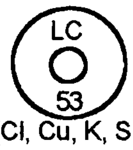
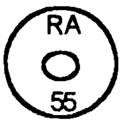
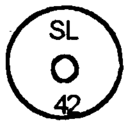
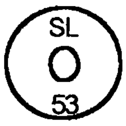
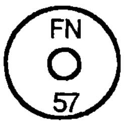
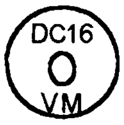
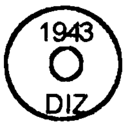
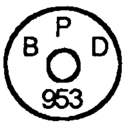
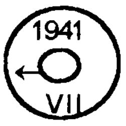
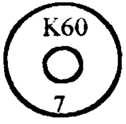
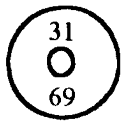
U.K. .450 REV ELEY·LONDON 450 Pb, Sb, Hg	U.K. .450 REV ELEY .450 Pb, Sb, Hg	U.K. .455 REV K .455 2Z Pb, Sb, Hg	U.K. .455 REV KYNOCH .455 Pb, Sb, Ba
U.K. .455 REV K 58 6 Z + 62 63 64 Pb, Sb, Ba,Hg	U.K. .455 REV K 62 Pb, Sb, Ba, Hg	U.K. .455 REV K43 VIZ Pb, Sb, Hg	U.K. .455 REV R ↑ L 3 6 II Pb, Sb, Hg
CANADA .455 REV DOMINION 455 COLT Pb, Sb, Ba	U.S.A. .50 BROWNING R A 41 Pb, Sb,	U.K. 12 BORE SHOTGUN ELEY-KYNOCH 12 Pb, Sb, Ba	U.S.S.R. 12 BORE SHOTGUN AZOT 12 12 Made in USSR Pb, Sb, Ba
U.K. 12 BORE SHOTGUN KYNOCH 1 12 ELEY Pb, Sb, Ba	U.K. 12 BORE SHOTGUN ELEY 12 12 ELEY Pb, Sb, Ba	U.K. 12 BORE SHOTGUN GAUGE 12 12 GAUGE Pb, Ba	ITALY 12 BORE SHOTGUN FIOCCHI 12 12 ITALY Pb, Sb, Ba
FRANCE 12 BORE SHOTGUN GEVELOT 12 12 PARIS Pb, Sb, Ba	U.S.A. 12 BORE SHOTGUN REMINGTON 12 GA PETERS Pb, Sb, Ba	CANADA 12 BORE SHOTGUN C-I-L 12 12 IMPERIAL Pb, Ba	

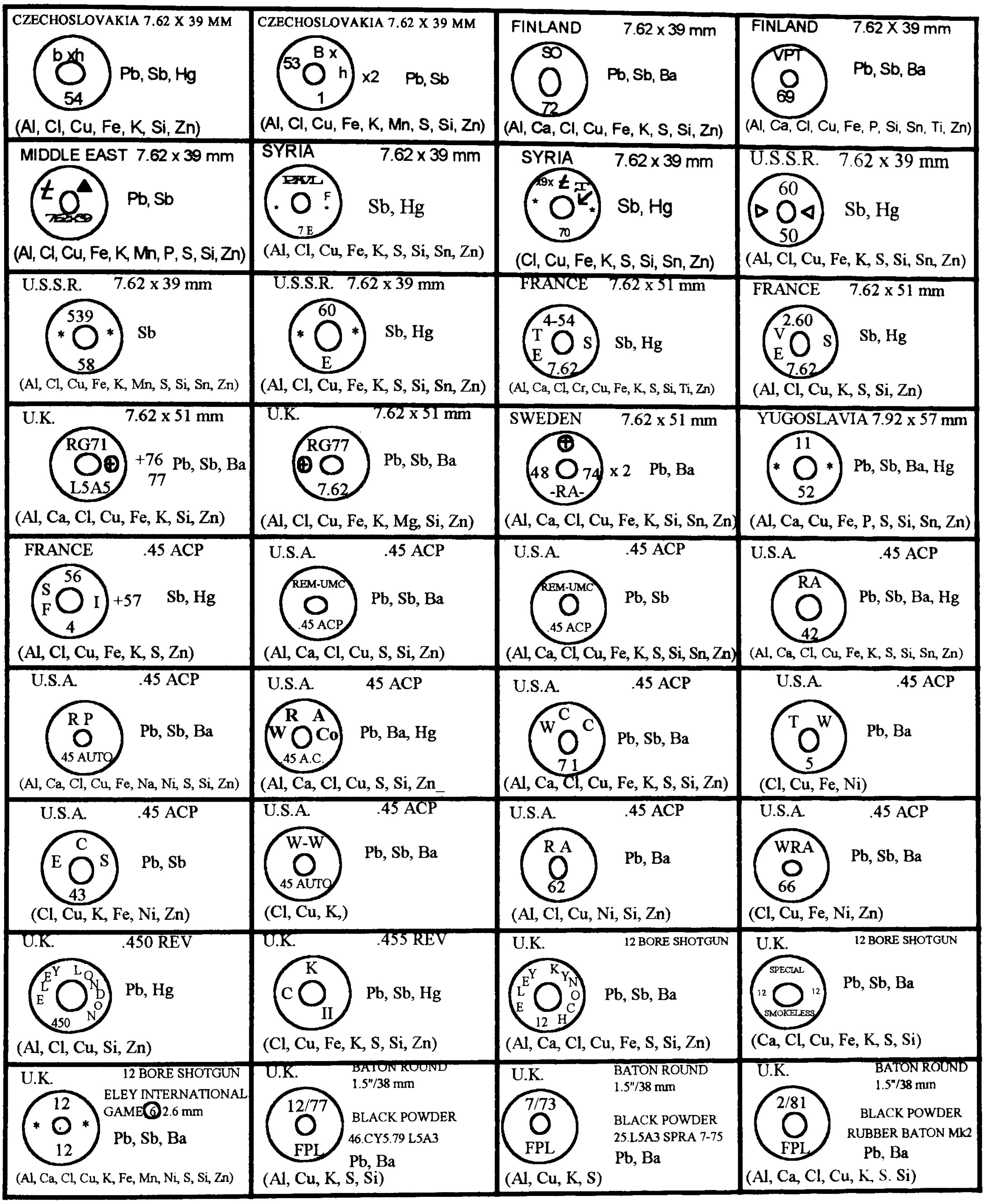
Figure 5.03 FAAS analysis of spent cartridge cases

U.S.A .22 LR  Pb, Ba (Cu, Si, Na)	U.S.A .25 ACP  Pb, Sb, Ba (Al, Ca, Cl, Cu, S, Si)	BELGIUM .320 REV  Pb, Ba (Cl, Cu, K, S, Si, Zn)	GERMANY .320 REV  Sb, Hg (Al, Cl, Cu, Fe, K, S, Si, Zn)
AUSTRIA .32 ACP  Pb, Ba (Ca, Cl, Cu, Fe, K, Si, Zn)	U.S.A. .32 ACP  Pb, Sb, Ba (Al, Cu, S, Si)	U.K. .32 ACP  Pb, Sb, Ba (Al, Cl, Cu, K, Si, Zn)	GERMANY .32 ACP  Pb, Sb, Ba (Al, Cl, Cu, S, Si)
BELGIUM .32 ACP  Pb, Ba (Cl, Cu, Ni, Zn)	CZECHOSLOVAKIA .32 ACP  Pb, Sb (Ca, Cl, Cu, Fe, Zn)	ITALY .32 ACP  Pb, Sb, Ba (Ca, Cl, Cu, Fe, Ni, Zn)	CANADA .32 ACP  Sb, Hg (Ca, Cl, Cu, Fe, Zn)
U.S.A. .32 ACP  Pb, Sb, Ba, Hg (Ca, Cu, Fe, Zn)	U.S.A. .32 ACP  Pb, Sb, Ba, (Ca, Cl, Cu, Fe, Ni, Zn)	SWEDEN .32 ACP  Pb, Sb, Ba (Ca, Cl, Cu, Fe)	FINLAND 9 mmP  + 43 44 Pb, Ba (Al, Ca, Cl, Cu, Fe, P, S, Si, Zn)
FINLAND 9 mmP  Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, Si, Zn)	CANADA 9 mmP  Pb, Ba (Al, Ca, Cl, Cu, Mg, Si, Zn)	FINLAND 9 mmP  Pb, Sb, Ba (Al, Ca, Cl, Cu, K, Si)	FINLAND 9 mmP  Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, Si, Zn)
FRANCE 9 mmP  Sb, Hg (Al, Cl, Cu, Fe, K, S, Si, Zn)	GERMANY 9 mmP  Pb, Sb, Hg (Al, Cl, Cu, Fe, K, P, S, Si, Sn, Zn)	GERMANY 9 mmP  Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, K, Mn, S, Si, Sn, Ti, Zn)	GERMANY 9 mmP  Pb, Ba (Al, Ca, Cl, Cu, Fe, K, P, Si)
GERMANY 9 mmP  Pb, Sb, Hg (Cl, Cu, K, Si, Zn)	U.K. 9 mmP  Pb, Sb, Ba Al, Ca, Cl, Cu, Fe, K, Si Zn)	U.K. 9 mmP  +62 Pb, Sb, Hg (Al, Ca,Cl, Cu, Fe, K, S, Si, Zn)	U.K. 9 mmP  Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, K, S, Si, Zn)
U.K. 9 mmP  Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, S, Si, Zn)	U.K. 9 mmP  79 +83 85 Pb, Sb, Ba Al, Ca, Cl, Cr, Cu, Fe, K, Si, Zn)	U.S.A. 9 mmP  Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, Si, Zn)	U.S.A. 9 mmP  Pb, Sb, Ba (Ca, Cl, Cu, S, Si, Zn)
U.S.A. 9 mmP  Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, K, S, Si, Zn)	U.K. 9 mmP  + 58 Pb, Sb, Ba (Ca, Cl, Cu, Fe, K, Si, Zn)	YUGOSLAVIA 9 mmP  Pb, Sb, Hg (Al, Ca, Cl, Cu, Fe, K, Mg, S, Si, Zn)	GERMANY 9 mmP  Pb, Sb, Ba (Cl, Cu, Ni)
SWEDEN 9 mmP  Pb, Sb, Ba (Ca, Cl, Cu, Fe, Ni, Zn)	CZECHOSLOVAKIA 9 mmP  Pb, Sb, Hg (Al, Ca, Cl, Cu, K, Fe, S, Si, Zn)	AUSTRIA 9 mmK  x 2 Pb, Ba (Al, Ca, Cl, Cu, Fe, K, Si Zn)	FINLAND 9 mmK  Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, Si, Zn)



FINLAND 9 mmK SAKO 380 ACP Pb, Sb, Ba (Al, Ca, Cl, Cr, Cu, Fe, S, Si, Zn)	U.K. 9 mmK ELEY 380 AUTO Sb, Hg (Al, Cl, Cu, Fe, K, S, Si, Zn)	U.S.A. 9 mmK REM-UMC 380 CAP x2 Sb, Hg (Al, Cl, Cu, Fe, K, S, Si, Zn)	U.S.A. 9 mmK WW 380 AUTO Pb, Sb, Ba (Cl, Cu, Fe, S, Si, Zn)
U.S.A. 9 mmK UMC 380 CAP Sb, Hg (Cl, Cu, K, S, Si, Zn)	U.S.A. 9 mmK FC 380 AUTO Pb, Sb, Ba (Al, Cl, Cu, S, Si, Zn)	U.K. 9 mmK KYNOCH 38 Pb, Sb, Ba (Ca, Cl, Cu, Fe, Zn)	HOMELOAD (NO HEADSTAMP) .380 REV Pb, Ba (Al, Cl, Cu, Fe, K, Na, S, Si, Ti, Zn)
U.K. .380 REV KYNOCH 38 S&W Pb, Sb, Ba Al, Ca, Cl, Cu, Fe, K, Mn, S, Si, Ti, Zn	U.K. .380 REV RG 65 380 2Z Pb, Sb, Ba (Ca, Cu, Fe, K, Si, Ti, Zn)	U.S.A. .380 REV R P .38 S&W Pb, Sb, Ba (Al, Cl, Cu, K, Mg, Si, Zn)	U.K. .380 REV K66 380 2Z Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, Si, Zn)
U.K. .380 REV RG 0 8 3 6 3 2Z Pb, Sb, Hg (Al, Cl, Cu, K, P, S, Si, Zn)	U.K. .380 REV KYNOCH 38 Pb, Sb, Ba, (Al, Cu, S)	SWEDEN .380 REV NORMA 38 S&W Pb, Sb, Ba (Ca, Cl, Cu, Fe, Zn)	U.S.A. .380 REV REM-UMC 38 S&W Pb, Sb, Ba (Ca, Cl, Cu, Fe, Ni Zn)
GERMANY .380 REV GECO - r 38 S&W Pb, Sb, Ba (Ca, Cl, Cu, Fe, Ni, Zn)	U.S.A. .380 REV BROWNING 38 S&W Pb, Sb, Ba (Ca, Cl, Cu, Fe, Ni, Zn)	SWEDEN .38 SPECIAL +P NORMA 38 +P SPECIAL Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, Na, S, Si, Zn)	U.S.A. .38 SPECIAL +P SUPER W W 38 SPL +P Pb, Sb, Ba (Al, Cl, Cu, Fe, S, Si, Zn)
U.S.A. .38 SPECIAL S &W .38 Spl NYCLAD Pb, Sb, Ba (Al, Cl, Cu, Ni, P, S, Si)	U.S.A. .38 SPECIAL W-W .38 SPECIAL Pb, Sb, Ba (Cl, Cu, Si, Zn)	FINLAND .38 SPECIAL LAPUA 38 SPL Pb, Sb, Ba (Al, Ca, Cl, Cu, S, Si)	U.S.A. .38 SPECIAL R-P 38 SPL Pb, Sb, Ba (Ca, Cl, Cu, Fe, Ni, Zn)
SWEDEN .357 MAGNUM NORMA 357 MAGN Pb, Sb, Ba (Al, Cl, Cu, S, Si)	U.S.A. .357 MAGNUM SUPER W W N U N 357 MA Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, K, Ni, Si, Ti, Zn)	AUSTRIA .223 REM HP * * .223 Pb, Ba (Al, Ca, Cl, Cu, Fe, Si)	AUSTRIA .223 REM HP - 79 Pb, Ba (Al, Ca, Cl, Cu, Fe, K, Si, Zn
CANADA .223 REM IVI 70 Pb, Sb, Ba (Al, Ca, Cl, Cu, K, S, Si, Zn)	SWEDEN .223 REM NORMA .223 Pb, Sb, Ba (Al, Ca, Cl, Cu, Mg, Si, Zn)	SWEDEN .223 REM NORMA .223 Sb, Hg (Al, Cl, Cu, Fe, K, S, Si)	U.S.A. .223 REM FC REM Pb, Sb, Ba (Al, Cu, Si, Zn)
U.S.A. .223 REM FC 223 REM Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, S, Si)	U.S.A. .223 REM LC 72 +75 77 Pb, Sb, Ba (Al, Cl, Cu, Fe, Zn)	U.S.A. .223 REM RA 65 +66 Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, S, Si, Zn)	U.S.A. .223 REM TW 67 Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, K, Si, Zn)
U.S.A. .223 REM TW 72 +73 Pb, Sb, Ba (Al, Cl, Cu, Fe, K, Mn, S, Si, Zn)	U.S.A. .223 REM WCC 64 Pb, Sb, Ba (Al, Cl, Cu, Fe, Si, Zn)	U.S.A. .223 REM WRA 70 Pb, Sb, Ba (Al, Cl, Cu, Fe, K, S, Si)	ITALY 6.5 mm CARCANO SMI 935 Pb, Sb, Hg (Ca, Cl, Cu, Fe, K, S, Si, Zn)

FRANCE .30 M1 CARBINE  Sb, Hg (Al, Cl, Cu, Fe, K, S, Si, Sn, Zn)	FRANCE .30 M1 CARBINE  Pb, Sb, Ba (Ca, Cl, Cu, Fe, K, Si)	FRANCE .30 M1 CARBINE  Sb, Hg (Al, Cl, Cu, Fe, K, Ni, S, Si, Zn)	FRANCE .30 M1 CARBINE  Sb, Hg (Al, Cl, Cu, Fe, K, S, Si, Zn)
FRANCE .30M1 CARBINE  Sb (Al, Cl, Cu, Fe, K, S, Si)	FRANCE .30M1 CARBINE  Sb, Hg (Cl, Cu, Fe, K, S, Si, Zn)	FRANCE .30 M1 CARBINE  [illegible] (Al, Ca, Cl, Cu, Fe, K, Si, Zn)	FRANCE .30 M1 CARBINE  Sb, Hg (Al, Cl, Cr, Cu, Fe, K, S, Si, Zn)
FRANCE .30M1 CARBINE  Sb (Al, Cl, Cu, K, S, Si)	FRANCE .30M1 CARBINE  Sb, Hg (Al, Cl, Cu, Fe, K, S, Si, Zn)	FRANCE .30M1 CARBINE  Pb, Sb, Hg (Al, Cl, Cu, Fe, K, S, Si, Zn)	FRANCE .30M1 CARBINE  Pb, Sb, Hg (Al, Ca, Cl, Cu, Fe, K, S, Si, Zn)
FRANCE .30M1 CARBINE  Sb, Ba, Hg (Al, Ca, Cl, Cu, Fe, K, S, Si, Zn)	SWEDEN .30M1 CARBINE  Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, K, S, Si, Zn)	U.S.A. .30M1 CARBINE  Pb, Sb, Ba (Al, Cl, Cu, Fe, K, S, Si, Zn)	U.S.A. .30M1 CARBINE  Pb, Sb, Ba (Al, Cl, Cu, Fe, S, Si, Zn)
FRANCE .30M1 CARBINE  Sb, Hg (Ca, Cl, Cu, Fe, K, Si, Zn)	FRANCE .30M1 CARBINE  Pb, Sb, Ba (Ca, Cl, Cu, Fe, K, Si)	FRANCE .30-06  Sb, Ba, Hg (Al, Cl, Cu, Fe, K, S, Si, Zn)	FRANCE .30-06  Pb, Sb, Ba, Hg (Al, Ca, Cl, Cu, Fe, K, S, Zn)
FRANCE .30-06  Pb, Sb, Ba, Hg (Al, Ca, Cl, Cu, Fe, K, Mg, S, Si, Zn)	ITALY .30-06  Pb, Sb, Hg (Cl, Cu, Fe, K, S, Si, Zn)	U.K. .30-06  Pb, Sb, Hg (Al, Cl, Cu, Fe, K, S, Si, Zn)	U.S.A. .30-06  Pb, Sb (Al, Cl, Cu, K, S, Si, Zn)
U.S.A. .30-06  Pb, Sb (Al, Cl, Cu, Fe, K, S, Si, Zn)	U.S.A. .30-06  Pb, Sb (Al, Cl, Cu, Fe, K, S, Si, Zn)	U.S.A. .30-06  Sb (Cl, Cu, K, S, Zn)	U.S.A. .30-06  Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, Si, Zn)
U.S.A. .30-06  Pb, Sb, Hg (Al, Cl, Cu, Fe, K, S, Si, Zn)	U.S.A. .30-06  Pb, Sb, Ba (Al, Cl, Cu, Fe, K, S, Si, Zn)	BELGIUM .303  Sb, Hg (Al, Cl, Cu, Fe, K, S, Si, Sn, Zn)	CANADA .303  Sb, Hg (Al, Cl, Cu, K, S, Si, Zn)
CANADA .303  Pb, Ba (Al, Ca, Cl, Cu, Fe, Si Zn)	ITALY .303  Pb, Sb, Hg (Al, Cl, Cu, Fe, K, S, Si, Zn)	U.K. .303  Pb, Sb, Hg (Cl, Cu, K, S)	U.S.A. .303  +41 Pb, Sb (Al, Cl, Cu, K, Na, S, Zn)
PORTUGAL .303  Sb, Hg (Cl, Cu, Fe, K, S, Si, Sn, Zn)	U.K. .303  Pb, Sb (Al, Cl, Cu, Fe, K, S, Si)	CZECHOSLOVAKIA .303  Sb, Hg (Al, Cl, Cu, Fe, K, S, Si, Sn, Zn)	CHINA 7.62 x 39 mm  +70 Sb, Hg (Al, Cl, Cu, Fe, K, P, S, Si, Sn, Zn)



CZECHOSLOVAKIA 7.62 X 39 MM Pb, Sb, Hg (Al, Cl, Cu, Fe, K, Si, Zn)	CZECHOSLOVAKIA 7.62 X 39 MM x2 Pb, Sb (Al, Cl, Cu, Fe, K, Mn, S, Si, Zn)	FINLAND 7.62 x 39 mm Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, K, S, Si, Zn)	FINLAND 7.62 X 39 mm Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, P, Si, Sn, Ti, Zn)
MIDDLE EAST 7.62 x 39 mm Pb, Sb (Al, Cl, Cu, Fe, K, Mn, P, S, Si, Zn)	SYRIA 7.62 x 39 mm Sb, Hg (Al, Cl, Cu, Fe, K, S, Si, Sn, Zn)	SYRIA 7.62 x 39 mm Sb, Hg (Cl, Cu, Fe, K, S, Si, Sn, Zn)	U.S.S.R. 7.62 x 39 mm Sb, Hg (Al, Cl, Cu, Fe, K, S, Si, Sn, Zn)
U.S.S.R. 7.62 x 39 mm Sb (Al, Cl, Cu, Fe, K, Mn, S, Si, Sn, Zn)	U.S.S.R. 7.62 x 39 mm Sb, Hg (Al, Cl, Cu, Fe, K, S, Si, Sn, Zn)	FRANCE 7.62 x 51 mm Sb, Hg (Al, Ca, Cl, Cr, Cu, Fe, K, S, Si, Ti, Zn)	FRANCE 7.62 x 51 mm Sb, Hg (Al, Cl, Cu, K, S, Si, Zn)
U.K. 7.62 x 51 mm +76 77 Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, K, Si, Zn)	U.K. 7.62 x 51 mm Pb, Sb, Ba (Al, Cl, Cu, Fe, K, Mg, Si, Zn)	SWEDEN 7.62 x 51 mm x 2 Pb, Ba (Al, Ca, Cl, Cu, Fe, K, Si, Sn, Zn)	YUGOSLAVIA 7.92 x 57 mm Pb, Sb, Ba, Hg (Al, Ca, Cu, Fe, P, S, Si, Sn, Zn)
FRANCE .45 ACP +57 Sb, Hg (Al, Cl, Cu, Fe, K, S, Zn)	U.S.A. .45 ACP Pb, Sb, Ba (Al, Ca, Cl, Cu, S, Si, Zn)	U.S.A. .45 ACP Pb, Sb (Al, Ca, Cl, Cu, Fe, K, S, Si, Sn, Zn)	U.S.A. .45 ACP Pb, Sb, Ba, Hg (Al, Ca, Cl, Cu, Fe, K, S, Si, Sn, Zn)
U.S.A. .45 ACP Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, Na, Ni, S, Si, Zn)	U.S.A. 45 ACP Pb, Ba, Hg (Al, Ca, Cl, Cu, S, Si, Zn_	U.S.A. .45 ACP Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, K, S, Si, Zn)	U.S.A. .45 ACP Pb, Ba (Cl, Cu, Fe, Ni)
U.S.A. .45 ACP Pb, Sb (Cl, Cu, K, Fe, Ni, Zn)	U.S.A. .45 ACP Pb, Sb, Ba (Cl, Cu, K,)	U.S.A. .45 ACP Pb, Ba (Al, Cl, Cu, Ni, Si, Zn)	U.S.A. .45 ACP Pb, Sb, Ba (Cl, Cu, Fe, Ni, Zn)
U.K. .450 REV Pb, Hg (Al, Cl, Cu, Si, Zn)	U.K. .455 REV Pb, Sb, Hg (Cl, Cu, Fe, K, S, Si, Zn)	U.K. 12 BORE SHOTGUN Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, S, Si, Zn)	U.K. 12 BORE SHOTGUN Pb, Sb, Ba (Ca, Cl, Cu, Fe, K, S, Si)
U.K. 12 BORE SHOTGUN ELEY INTERNATIONAL GAME⑥2.6 mm Pb, Sb, Ba (Al, Ca, Cl, Cu, K, Fe, Mn, Ni, S, Si, Zn)	U.K. BATON ROUND 1.5"/38 mm BLACK POWDER 46.CY5.79 L5A3 Pb, Ba (Al, Cu, K, S, Si)	U.K. BATON ROUND 1.5"/38 mm BLACK POWDER 25.L5A3 SPRA 7-75 Pb, Ba (Al, Cu, K, S)	U.K. BATON ROUND 1.5"/38 mm BLACK POWDER RUBBER BATON Mk2 Pb, Ba (Al, Ca, Cl, Cu, K, S. Si)

Figure 5.04 SEM/EDX analysis of spent cartridge cases

5.3.2 Propellant analysis

The samples were grains of propellant recovered from materials shot at close range, mainly clothing from injured persons. Consequently, in the vast majority of instances, the type of ammunition is not known. It must be noted that grains found may not necessarily be representative of the bulk, as composition can vary from grain to grain, and also that these are 'discharged' propellant and in the act of discharge surface coatings can be blown or burnt off. Tables 5.21 and 5.22 give the propellant compositions detected over a 4 year period (1990-1993).

KEY: Tables 5.21 and 5.22

NC	Nitrocellulose	DNT	Dinitrotoluene
NG	Nitroglycerine	TNT	Trinitrotoluene
MC	Methylcentralite	DPA	Diphenylamine
EC	Ethylcentralite	nDPA	a nitrodiphenylamine
DBP	Dibutylphthalate	meDPA	Methylethyldiphenylamine
√	Detected	-	Not Detected

Number of Shooting Incidents	*DPA*	*NDPA*	*EC*	*MC*	*DBP*	*DNT*	*Comments*
40	√	-	-	-	-	-	
10	√	-	√	-	-	-	
5	√	-	-	-	-	-	+ Camphor
4	√	√	√	-	-	-	
4	√	-	-	√	-	-	
4	√	-	-	-	√	√	
3	√	-	-	-	-	√	+ Naphthalene in one of the samples
2	√	-	√	-	-	√	
2	-	-	-	-	-	-	Only Camphor detected
2	√	-	-	-	-	√	+ Camphor (Benzene and Naphthalene also detected in one sample)
2	√	√	-	-	-	-	+ Sulphur
2	√	-	-	√	-	√	
2	√	x2	-	-	√	-	Tributylphosphate also detected in one sample
1	-	x2	√	-	√	√	
1	-	-	-	-	√	√	+ Cresol
1	√	√	-	-	-	√	+ Camphor and Naphthalene
1	√	√	√	-	√	-	
1	√	√	√	-	-	-	+ Camphor
1	-	-	√	√	-	-	
1	√	√	-	-	-	-	+ ME DPA
1	-	x2	√	-	√	√	+ Naphthalene
1	√	x2	√	-	√	-	
1	-	-	√	-	-	-	

Table 5.21 Analysis of single base propellants (NC present)

Number of Shooting Incidents	***DPA***	***nDPA***	***EC***	***MC***	***DBP***	***DNT***	***Comments***
20	√	-	-	-	-	-	Two nDPA's also detected in one sample
12	-	-	√	-	-	-	Naphthalene also detected in one sample
12	√	-	√	-	-	-	
11	√	-	-	-	-	√	Sulphur, TNT, Tributylphosphate also detected, occurring separately in three samples
10	√	-	√	-	-	√	
5	√	-	-	-	√	√	
5	√	-	-	-	√	-	
4	√	-	√	√	-	√	Napthalene also detected in one sample
3	√	-	√	-	√	√	Two nDPA's also detected in one sample
2	√	√	-	-	√	√	
2	√	√	-	-	-	-	
2	√	√	-	-	-	√	
2	√	-	√	√	-	-	
1	-	√	√	-	-	-	
1	√	√	√	√	√	√	
1	√	√	√	-	-	-	Tributylphosphate also detected
1	√	-	√	-	√	-	
1	-	-	-	-	-	-	Only Camphor detected
1	-	-	-	-	√	-	
1	-	-	-	-	√	√	
1	-	√	-	-	√	-	
1	-	-	√	√	√	-	
1	-	-	√	-	√	-	
1	-	-	√	-	-	√	
1	√	-	-	√	-	√	

Table 5.22 Analysis of double base propellants (NC, NG present)

5.3.3 Miscellaneous components

Over the past 23 years it has been necessary to examine numerous components of ammunition as a consequence of the requirement of the particular case. Table 5.23 was compiled from casework records and illustrates the variation in, and complexity of, ammunition. It should be noted that 'coating' means either a plating or wash and that some cartridge cases and bullet jackets are coated both externally and internally, whereas others are only coated externally.

Description	*Observations*
.22, Eley, standard velocity, primer	Pb, Ba, (Al, Ca, Cu, P, Si)
.22 tracer bullet	Pb core with Ba, Sr, Mg containing tracer composition
German military bullet (no detail)	Brass coated Fe jacket/Fe core
12 bore, metal base of case	Many are Sn coated Fe e.g. Eley
Silvalube bullet (Mountain & Sawden)	Al coated Pb (trace Sb, Cu, Fe, Si)
9 mmP, MEN-83-25, primer	Pb, Sb, Ba (Al, Ca, Cl, Cu, Fe, K, Si, Sn, Zn)
9 mmP, OXO43, bullet	Brass coated Fe jacket/Fe core
9 mmP, K582Z, bullet	Brass coated Fe jacket/Pb core
9 mP, Ch (Belgian), bullet	Brass coated Fe jacket/Pb sheath, Fe core
9 mP, SFI78, bullet	Brass jacket/Pb core (trace Sb)
9 mP, 1X*51.2, bullet	Ni coated Fe jacket/Fe core with Pb sheath
9 mmP, Mauser, bullet	Ni (trace Cu) coated Fe (trace Mn) jacket/Pb core (trace Sb)
.30M1, Norma triclad, bullet	Cu/Ni coated Fe jacket/Pb core (trace Sb)
.38 S&W, Kynoch, bullet	Unjacketed Pb (trace Sb)
.38 Equaloy, bullet	Al bullet; outside skin (Al, Ti, major: Cl, Fe, P, S, trace), inside Al only
.38SPL, Winchester Silvertip, bullet	Al jacket/Pb core
.38SPL, W-W Lubaloy, bullet	Cu jacket/Pb core
.38SPL, W-W, bullet	Unjacketed Pb (trace Sb)
.38SPL, Kynoch, bullet	Unjacket Pb (trace Sn)
.357 Mag, KTW metal piercing bullet	Homogeneous brass with green plastic (Teflon) coating containing Al, Cr, Ti, major: Ca, Cl, Cu, K, S, Si, Zn trace
.357 Mag, W-W Super, bullet	Solid brass with exposed top portion coated with green plastic (Teflon) containing Cr, Ti
.450, Eley, bullet	Unjacketed Pb
.450, Kynoch, bullet	Unjacketed Pb (trace Sb)
.455, Kynoch, bullet	Unjacketed Pb
7.62 NATO, RAUFOSS, bullet	Brass jacket/Pb core (trace Sb)
7.62 NATO, 47-RA-77, tracer bullet	Cu jacket/Sr and Fe at tail end: Pb sheath, Fe core at nose end
.308 WIN, PMC, primer	Pb, Sb, Ba (Al, Ca, Cl, Cu, K, S, Si)
7.9 mm, Mauser, bullet	Cu/Ni coated Fe jacket/Pb core (trace Sb)
.30-06, SL53, AP bullet	Brass jacket/Pb sheath, Fe core (trace Mn)
12 bore, Eley International, primer	Pb, Sb, Ba (Al, Ca, Cl, Cu, K, Fe, Mn, Ni, S, Si, Zn) **Note:** Fe frequently at major level

Description	***Observations***
12.7 mm, Russian 188/83 AP/I bullet	Cu jacket (trace Zn)/Pb sheath, Fe core: incendiary powder contained Mg, Al, Ba
7.62 NATO, L5A3, tracer bullet	Tracer composition contained Cl, Cu, Sr (trace Al, Ba, Bi, Ca, Fe, K, Ni, S, Si, Zn)
.50, tracer bullet	Tracer composition contained Ba with a trace of S
.223, 84.SF, SFM, round	Frangible bullet with Cu/Sn, Al case; Pb, Sb, Ba primer, single base propellant (DPA)
.223 NATO, RORG88, ROTA round	Frangible bullet with Cu/Si/W, brass case, Pb, Sb, Ba primer, double base DPA, DBP
.25 AUTO, REM-UMC, round	Cu jacket/Pb core, brass case, Ni coated brass primer cup
.25 AUTO, R-P, round	Cu jacket/Pb core, double base propellant
.32 AUTO, GECO LT, round	Ni coated brass jacket/Pb core, brass case
.320, RWS, round	Unjacketed Pb bullet, brass case, Cu primer cup, black powder
.32-20, UMC, round	Lubricated unjacketed Pb bullet, brass case, Cu primer cup, black powder
.297-.230, no headstamp, round	Unjacketed Pb bullet, brass case, Cu primer cup, black powder with fibre wad
9 mK, *HP*, round	Fe jacket/Pb core (trace Si, Fe); brass case, primer cup and anvil; Pb, Ba primer
9 mmP, CCI.NR, round	Cu jacket/Pb core (trace Sb, Fe, Cu, Al, Si), Al coated steel case, Ni coated brass primer cup. Propellant contains K and S; Pb, Sb, Ba primer
9 mmP, ELEY 83, round	Ni coated brass primer cup, brass case (trace Al), Pb, Sb, Ba primer
9 mmP, SBP, round	Fe jacket/Pb core (trace Si, Fe); brass primer cup, anvil and case; Pb, Ba primer (trace Sn)
9 mmP, B↑E43, round	Cu jacket/Pb core, brass case
9 mmP, *11.50.0, round	Ni coated Fe jacket (trace Zn)/Fe core, brass primer cup and case; Pb, Sb, Hg primer
9 mmP, NATO, RG85 round	Brass jacket/Pb core (trace Si), brass primer cup and case
9 mmP, W-W, round	Ni coated brass jacket/Pb core (trace Si), brass case, Ni coated brass primer cup
9 mmP, 12* 49x51, round	Fe jacket/Fe core with Pb sheath, brass case
9 mmP, SFM-THV, round	Solid brass bullet, brass case; Pb, Ba primer
9 mmP, SANDIA, round	Brass jacket/Pb core (trace Si), brass case; Pb, Sb, Ba primer
9 mmP, R.P, round	Cu jacket/Pb core, brass case, Ni coated brass primer cup

Description	***Observations***
9 mmP, GECO*, round	Brass coated Fe jacket/Pb core, brass case, Ni coated brass primer cup
9 mmP NATO, RG84.2Z, round	Cu jacket/Pb core, brass case and primer cup
9 mmP NATO, FFV88, round	Cu coated Fe jacket/Pb core, brass case and primer cup
9 mmP NATO, FNM84-12, round	Cu coated Fe jacket/Pb core, brass case and primer cup
9 mmP, NORMA round	Cu coated Fe jacket/Pb core, brass case, Ni coated brass primer cup
9 mmP, WIN, round	Cu jacket/Pb core, brass case, Ni coated brass primer cup
9 mmP, S&B, round	Ni coated Fe jacket/Pb core, brass case, Ni coated brass primer cup; Pb, Sb, Ba primer (trace Sn)
.380 AUTO, W-W, round	Brass jacket/Pb core (trace Si); brass case, primer cup and anvil; Pb, Sb, Ba primer
.380 REV, R↑L45.2Z, round	Cu coated Fe jacket/Pb core, brass case and primer cup
.380 REV, K66.2Z, round	Cu jacket/Pb core, brass case and primer cup
.38 S&W, Kynoch, round	Unjacketed Pb bullet (trace Sb), brass case and primer cup
.38 S&W, Kynoch, round	Lubricated unjacketed Pb bullet, brass case, Ni coated brass primer cup
.38 SPL, SBW, round	Unjacketed Pb bullet (trace Al, Ca, Si); brass case, primer cup and anvil, Pb, Sb, Ba primer
.38 SPL, S&W, round	Pb bullet fully coated with plastic (Teflon), Ni coated brass case
.38 SPL, LAPUA, round	Lubricated unjacketed Pb bullet, brass case and primer cup
.38 SPL, W-W, round	Cu jacket/Pb core (trace Sb), Ni coated brass case and primer cup
.38 SPL, NORMA, round	Cu jacket/Pb core, brass case, Ni coated brass primer cup
.38 SPL, R.P, round	Lubricated unjacketed Pb bullet, Ni coated brass case
.38 SPL, CCI.NR, round	Lubricated unjacketed Pb bullet (trace Sb), Al case (trace Cu, Fe, Mn)
.38 SPL +P, W SUPER W, round	Al jacket/Pb core, Ni coated brass case
.38 SPL +P, W-SUPER-W, round	Lubricated unjacketed Pb bullet, Ni coated brass case and primer cup, brass anvil
.38 SPL +P, CCI.NR, round	Cu jacket/Pb core (trace Sb), Al case, brass primer cup (trace Fe); Pb, Sb, Ba primer
.38 SPL +P, SFM-THV, round	Solid brass bullet, brass case; Pb, Sb, Ba primer

Description	*Observations*
.357 MAG, CCI.NR, round	Al case, Ni coated brass primer cup; Pb, Sb, Ba primer
.357 MAG, NORMA, round	Brass jacket/Pb core; brass case, primer cup and anvil; Pb Sb, Ba primer
.357 MAG, W-W SUPER, round	Unjacketed Pb bullet (trace Si); brass case, primer cup and anvil; Pb, Sb, Ba primer
.357 MAG, W-W SUPER, round	Brass jacket/Pb core (trace Sb), Ni coated brass case (trace Al)
.357 MAG, W-W SUPER, round	Cu jacket/Pb core, Ni coated brass case and primer cup
.45ACP, WRA68, round	Cu coated Fe jacket/Pb core, brass case and primer cup
.45ACP, WCC73, round	Cu coated Fe jacket/Pb core, brass case and primer cup
.45ACP, SFI4.56, round	Brass jacket/Pb core, brass case and primer cup
.45ACP, FN45*, round	Cu jacket/Pb core, brass case and primer cup
.45ACP, RA68, round	Cu coated Fe jacket/Pb core, Ni coated brass case and primer cup
.45ACP, R.P, round	Cu jacket/Pb core, Ni coated brass case
.45ACP, W-W, round	Cu jacket/Pb core, brass case
.30 Mauser, Kynoch, round	Cu jacket/Pb core ('K' marked on base), Brass case
.30MI, DAG.VL, round	Cu coated Fe jacket/Pb core, brass case
7.9 STEYER, no headstamp, round	Ni coated Fe jacket/Pb core, brass case
.223, FN79, AP round	Cu jacket/Steel penetrator with Pb sheath, brass case
.223, FNB83, round	Cu jacket/Pb core with steel tip, brass case and primer cup
.223, TW72, round	Cu jacket/Pb core, brass case and primer cup
.223, LC72, round	Cu jacket/Pb core, brass case and primer cup
7.62 x 39, VPT73, round	Cu jacket/Pb core, brass case
7.62 x 39, BXN51, round	Cu coated Fe jacket/Steel core with Pb sheath, lacquered steel case, brass primer cup
7.62 NATO, RG84, round	Cu jacket/Pb core, brass case
7.62 NATO, 47-RA-74, round	Cu jacket/Pb core, brass case
7.62 NATO, FN78, round	Cu jacket/steel penetrator with Pb at base, brass case
7.62 x 51, FLB78, round	Brass jacket (trace Ni)/Pb core, brass (trace Al) case and primer cup; Pb, Sb, Ba primer
7.62 x 51, 89-070, AP round	Cu coated Fe jacket/hardened steel core with base enclosed in Al cup; brass case and primer cup

Description	*Observations*
.30-06, K53, round	Cu coated Fe jacket/Pb core, brass case and primer cup
.30-06, K58, round	Cu coated Fe jacket/Pb core, brass case and primer cup
.30-06, DM42, round	Cu coated Fe jacket/Pb core, brass case and primer cup
.30-06, FA54, round	Cu coated Fe jacket/Pb core, brass case and primer cup
7.62 NATO, 12-RA-78, tracer bullet	Cu coated Fe jacket/Pb nose, tracer composition contains Sr, Mg, Cl, tracer igniter composition contains Sr, Cu with minor Zn, base enclosed with Cu disc
.38 SPL, WCC, primer	Pb, Sb, Ba (Al, Ca, Cu, Fe, K, Mn, Ni, S, Si, Ti, Zn)
.357 MAG, FEDERAL, primer	Pb, Sb, Ba (Ca, Cu, K, Fe, Mn, Ni, S, Si, Ti, Zn)
.357 MAG, HP, Primer	Pb, Sb, Ba (Ca, Cu, K, Fe, Ni, S, Si, Ti, Zn)
9 mmP NATO, RG83, primer	Pb, Sb, Ba (Al, Ca, Cu, Fe, S, Si, Ti, Zn)
.32ACP, S&B primer	Pb, Sb, Ba (Ca, Cu, Fe, K, Mn, Ni, P, S, Si, Ti, Zn)
7.65 mm, GECO, primer	Pb, Sb, Ba (Al, Ca, Cu, Fe, K, Mn, Ni, S, Si, Sn, Ti, Zn)
.30MI, DAG, primer	Pb, Sb, Ba, (Ca, Cu, Fe, K, Mn, P, S, Si, Ti, Zn)
.30MI, WINCHESTER, primer	Pb, Sb, Ba (Al, Ca, Cu, Fe, K, Mn, S, Si, Ti, Zn)
TW 72, .223" calibre ammunition	Cu, Zn jacket/Pb core (trace Al); Cu, Zn primer cup; Pb, Sb, Ba primer; double base propellant with DPA, DNT, a phthalate plasticiser.
WRA70, .223" calibre ammunition	Cu, Zn jacket/Pb core (trace Sb); Cu, Zn primer cup; Pb, Sb, Ba primer; double base propellant with DPA, DNT, a phthalate plasticiser.
LC 72, .223" calibre ammunition	Cu (trace Al) jacket/Pb core (trace Al); Cu, Zn primer cup; Pb, Sb, Ba primer; double base propellant with DPA, DNT, a phthalate plasticiser.
FNB 83, .223" calibre ammunition	Cu, Zn jacket/Pb core (trace Al), Fe (trace Al) penetrator; Cu, Zn primer cup; Pb, Sb, Ba primer; double base propellant with DPA, DNT, a phthalate plasticiser.

Table 5.23 Analysis of ammunition components

5.3.4 Interpretation of ammunition analysis

The quantity of ammunition analysed provides a good database from which to draw general conclusions. The literature review presented in Chapter 2 is, in the main, supported by Figures 5.03 and 5.04 and Tables 5.21, 5.22 and 5.23.

Brass is the most popular material for cartridge cases and primer cups, with steel being the second most widely used material for cartridge case manufacture. Soft copper primer cups encountered were all from old ammunition containing black powder propellant.

Copper alloy bullet jackets are by far the most common and coated Fe jackets are also frequently employed. Lead is by far the most common bullet core material and is often hardened with Sb, but not as often as originally presumed, Sb occurring in only 25% of the lead bullets examined. Only one of the bullets examined was hardened by Sn. Some combination of the elements Ba, Sr, Mg, Fe and Cl were present in tracer bullets.

The review suggests that DPA is the most common stabiliser in single base propellants, whereas EC is the most common in double base. In fact Tables 5.21 and 5.22 show that DPA and/or its derivatives occurs in the majority of propellants, ~94.5% of single base and ~82.5% of double base. Ethyl centralite and DPA frequently occur together, whereas EC on its own is only found in ~2.0% of single base and ~14.5% of double base. It must be remembered that these are 'discharged' propellants, their origin is largely unknown and the figures are local to Northern Ireland; consequently the percentages must be viewed with caution. Methyl centralite occurs in ~8% of the propellants and according to the literature it may be used either as a plasticiser or moderant. It always occurred accompanied by other plasticisers and is more likely to be included as a moderant rather than as a plasticiser. Tributylphosphate was detected in 3 propellants and it is not mentioned in the literature. Its function is uncertain but it is used as a plasticiser in certain industrial processes. On the other hand many of the compounds mentioned in the review as possible constituents of propellants were not detected in the propellants analysed. However it must be borne in mind that Tables 5.21 and 5.22 represent only a small selection of propellants, which have been discharged and which are local to Northern Ireland.

The majority of the propellants analysed were from kneecapping incidents involving the use of handguns. A total of 194 propellant samples were analysed of which 92 were single based. This throws some doubt on the general rule stated in Chapter 2, namely, "rifle cartridges use single base propellants whereas pistol, revolver and shotgun cartridges use double base propellant. Rimfire rifle and revolver cartridge uses either single or double base propellant". Rimfire cartridges and rifles are rarely used in kneecappings.

Ammunition recovered in Northern Ireland covers a time span of > 50 years of ammunition manufacture, and many residues remaining in the spent cartridge case have been analysed. The residue examined does not necessarily originate exclusively from the primer, as a contribution could be made by the propellant or the exposed base of the bullet. However the residue appears to reflect the primer type in the majority of instances. A more satisfactory way to determine primer type is to remove and open the spent primer cup and examine the inside using SEM/EDX. This is not practical in casework as it would mean the destruction of evidence and would be time consuming, tedious and in the vast majority of instances, unnecessary. A more satisfactory method is to remove the bullet and propellant from a live round, discharge the primer and then sample the spent cartridge case interior. However in casework the actual spent cartridge cases involved in the incident are sampled, as it cannot be assumed that ammunition of the same calibre and headstamp will have the same composition.

Information obtained from visits to various munitions factories suggests that manufacturers will use whatever is available at the time, from whatever source, to complete an order, provided that it meets the required ballistics performance and produces no residues that are injurious to the gun. During the war years shortage of materials meant many variations in materials used in manufacture. For these reasons it is unwise to make assumptions about ammunition components and composition, even for the same calibre and manufacturer, as they could vary from batch to batch. The differences between ammunition with the same headstamp can be seen in Table 5.23 for Winchester Western in .38 SPECIAL and .357 MAGNUM calibres and in Figure 5.04 for .30 M1 calibre VE 54 F1 and VE 2-61 S.

The analysis of 'primers' supports the statement that Communist Bloc countries frequently use mercury fulminate primers and it is also worth noting that the same applies to ammunition manufactured in France, at least for the time period involved. According to the literature there is no mercury in U.S. military ammunition since 1898, but it was used to a later date (~1930) in some U.S. commercial primers. Whilst the ammunition data in Figures 5.03 and 5.04 strongly supports this, there are some anomalies, namely, .30-06 calibre SL42, .30M1 calibre EC4, .303" calibre WRA 41 and 43 and WCC 1940, and .45 ACP calibre RA 42, all of which had mercury present and were manufactured during war years.

A detailed summary of primer types encountered over a 13 year period (1975-1987) is given in Table 5.24 which represents the examination of 1,300 spent cartridge cases, involving 310 different headstamps and 58 manufacturers, and is based on casework results, some of which are included in Figures 5.03 and 5.04 and Table 5.23.

Calibre	*Pb/Sb/Ba*	*Pb/Ba*	*Pb/Sb*	*Sb/Hg*	*Pb/Ba/Hg*	*Pb/Ba/Sb/Hg*	*Pb/Sb/Hg*	*Pb*	*Sb*	*Pb/Hg*	*Ba/Sb/Hg*	*Number of headstamps*
.22 LR	2	37	4	-	-	-	-	5	-	-	-	7
.32 ACP	42	7	3	-	-	-	-	-	-	-	-	10
9 mmP	103	15	3	26	-	7	31	-	-	-	3	58
9 mmK	46	34	7	8	-	-	-	-	-	-	-	28
.380 Rev	18	4	-	-	-	2	2	-	-	-	-	10
.38 Special	12	-	-	-	-	-	-	-	-	-	-	5
.223	278	3	11	1	-	-	-	3	-	-	-	25
8 mm Mauser	-	-	-	1	-	-	8	-	-	-	-	2
.30 MI	34	12	1	34	-	32	10	-	27	-	2	31
.30-06	49	2	18	4	-	2	5	-	1	-	2	21
.303	4	1	3	15	1	6	28	-	-	2	-	34
7.62 x 39 mm	16	-	9	33	-	-	-	4	3	-	-	14
7.62 x 51 mm	54	3	1	2	-	-	-	-	-	-	-	20
.45 ACP	61	3	1	1	2	1	2	-	3	-	-	16
.450 Rev	-	-	-	-	-	-	9	-	-	3	-	3
.455 Rev	3	1	-	-	-	11	9	-	-	-	-	11
12 Bore	24	7	3	-	-	-	-	-	-	-	-	10
Miscellaneous	3	-	2	-	-	1	-	2	1	1	-	5
Total	749	129	66	125	3	62	104	14	35	6	7	310
~ %	57.5	10.0	5.0	9.5	<0.5	5.0	8.0	1.0	3.0	0.5	0.5	

Table 5.24 Analysis of spent cartridge cases for Pb, Sb, Ba, Hg

Primers could be grouped into 6 categories (1) corrosive and mercuric (potassium chlorate and mercury fulminate), (2) non-corrosive and mercuric (barium nitrate replaced potassium chlorate), (3) corrosive and non-mercuric (lead styphnate replaced mercury fulminate), (4) non-corrosive, non-mercuric (modern Sinoxyd type), (5) unusual/miscellaneous primer compositions and (6) recent non-toxic primers (Sintox).

The fact that the spent cartridge cases in Table 5.24 were not analysed for K or Cl to indicate potassium chlorate makes interpretation difficult. Nevertheless Table 5.24 does support the history of primer development as outlined in Chapter 2.

Category (6) Sintox primers, can be excluded from consideration as they were introduced at a later date and their use has not yet been encountered in casework. For category (1) mercury would be present and barium would be absent. From Table 5.24 ~76.5% of mercury containing primers are corrosive. For category (2) both mercury and barium would be present. Therefore ~23.5% of mercury containing primers are non-corrosive.

For category (3) mercury and barium would be absent and lead would be present. Modern type primers would be Pb, Sb, Ba, and Pb, Ba. On this basis a somewhat speculative breakdown of primer types involved in casework during this period is:-

~67.5% modern

~24.0% mercury fulminate

~6.0% non-mercuric but corrosive

~2.5% miscellaneous

This also supports the history of primer development.

These figures do not reflect the current situation. As a consequence of terrorist organisations on both sides acquiring large arms consignments, since March 1988 the IRA and related groups frequently use the 7.62 x 39 mm calibre AKM type rifle with Yugoslavian nny 82 ammunition whereas the loyalists groups use the 7.62 x 39 mm calibre VZ 58 P rifle with Chinese 351/73 ammunition.

Analysis of the nny 82 ammunition, as previously detailed, shows that it uses a mercuric, corrosive primer. Analysis of the Chinese 351/73 ammunition revealed that it has a Cu coated Fe jacketed bullet with a Fe core and a Pb tip, the cartridge case is steel with a brown coloured lacquered finish, a brass primer cup, and the propellant is single base with DPA, 2 x nitrodiphenylamines, camphor, and contains no inorganic additives, and the discharged primer composition is Sb, K, Cl, Hg, Sn, S, Fe, Mn, P, Zn and Pb in descending order (Pb, Sb, Hg type).

The frequent use of both types of ammunition in recent years has substantially increased the proportion of shooting incidents involving the use of mercury fulminate primed ammunition. It has also substantially increased the proportion of shooting incidents involving the use of single base propellant. It is worth noting that both propellants contain camphor.

5.4 AMMUNITION CONTAINING MERCURY

The particle classification scheme, developed as described in reference 140, did not include mercury fulminate primed ammunition, which is frequently encountered in Northern Ireland, and is currently manufactured in some Eastern Bloc countries.

In casework in which discharge residue particles were detected, and in which the ammunition involved is known to contain mercury, very few, if any, of the particles contained mercury. This has been noted over many years and in numerous cases. Possible reasons for this could be the volatility of mercury and its compounds, or decomposition of the mercury fulminate and the loss of mercury through amalgamation with zinc in the primer cup/cartridge case. It is not uncommon, when firing old ammunition with mercury fulminate primers, for some of the cartridge cases to crack, due to embrittlement of the brass caused by mercury amalgamating with the zinc.

In order to clarify the situation regarding mercury containing ammunition a series of experiments were conducted and casework statistics gathered.

5.4.1 ***Frequency of occurrence***

To determine the frequency of occurrence of Hg containing particles in FDR, promptly collected residue from the discharge of mercury fulminate primed ammunition was examined. Results are given in Table 5.25. Nothing of significance was detected on the controls taken from the firing hand.

In the first firing a small proportion of the particles also contained one of the following elements, Co (trace), Mg (trace), Mn (trace), Ni (trace) and P (minor and trace).

The particle types containing mercury that were detected in firings 1 to 7 (Table 5.25) are as listed in Table 5.26.

Ammunition	***Particle Types***												***Total Number Particles***
	Pb, Sb, Ba	***Sb, Ba***	***Ba, Ca, Si***	***Pb, Sb,***	***Pb, Ba***	***Pb only***	***Sb only***	***Ba only***	***Brass***	***Fe Major***	***Others***	***Hg***	
1. Unjacketed K.455 2Z	1	None	None	218	2	45	1	1	2	17	3	16	306
Pb, Sb, Ba, Hg	<0.5%	-	-	71.0%	0.5%	14.5%	<0.5%	<0.5%	0.5%	5.5%	1.0%	5.0%	
2. FMJ (Cu) K64 6Z	22	1	None	30	1	88	1	3	6	33	11	None	196
Pb, Sb, Ba	11.0%	0.5%	-	15.0%	0.5%	45.0%	0.5%	1.5%	3.0%	17.0%	5.5%	-	
3. FMJ (Cu) K58 6Z	7	None	None	193	4	36	3	3	8	33	None	8	295
Pb, Sb, Ba, Hg	2.0%	-	-	65.0%	1.0%	12.0%	1.0%	1.0%	2.5%	11.0%	-	2.5%	
4. FMJ (Cu) RG56.9 mm 2Z	8	None	2	13	8	139	None	1	9	99	15	1	295
Pb, Sb, Ba, Hg	2.5%	-	0.5%	4.5%	2.5%	47.0%	-	<0.5%	3.0%	33.5%	5.0%	<0.5%	
5. FMJ (Cu) RG59.9 mm 2Z	11	1	1	24	20	103	2	None	25	81	27	3	298
Pb, Sb, Ba, Hg	3.5%	<0.5%	<0.5%	8.0%	6.5%	34.5%	0.5%	-	8.0%	27.0%	9.0%	1.0%	
6. FMJ (Cu) RG55.9 mm 2Z	14	None	3	42	9	101	None	1	30	63	23	5	291
Pb, Sb, Ba, Hg	5.0%	-	1.0%	14.0%	3.0%	34.5%	-	<0.5%	10.0%	21.5%	8.0%	1.5%	
7. FMJ (Cu) K56.9 mm 2Z	24	2	10	39	27	84	1	1	35	52	19	1	295
Pb, Sb, Ba, Hg	8.0%	1.0%	3.5%	13.0%	9.0%	28.5%	<0.5%	<0.5%	12.0%	18.0%	6.5%	<0.5%	

Table 5.25 Occurrence of mercury particles

Major	*Minor*	*Trace*	*Particle Type*
Pb	Si, Sb	Cl, K, Cu, Fe, Hg	Pb, Sb, Hg
Pb, Sb	-	Cu, K, Si, Cl, Zn, Hg, Fe	Pb, Sb, Hg
3 x Pb	Si	Cl, Sb, Cu, Hg	Pb, Sb, Hg
Pb	Sb, K, Cl, Si, Al	Cu, Zn, Hg	Pb, Sb, Hg
Pb	Sb, Si, Al	Cu, Hg	Pb, Sb, Hg
Pb	Sb, Cl, K, Si	Cu, Fe, Zn, Al, Hg	Pb, Sb, Hg
Pb, S, Sb, K, Cl	Si, P, Al, Cu	Hg, Fe	Pb, Sb, Hg
Sb, Pb, S	Cu, Si, Al, K, Cl	Fe, Zn, Hg	Pb, Sb, Hg
Si, Al	K, Ca, Fe	Hg	Hg only
Pb, Cl	Sb, K, Si	Cu, Co Hg	Pb, Sb, Hg
Pb, S	Sb, Si	Cu, Fe, Hg	Pb, Sb, Hg
Pb, S, Cl, K, Sb	Si	Cu, Hg	Pb, Sb, Hg
Pb, S	Si, Sb, Cl	K, Mg, Cu, Fe, Hg	Pb, Sb, Hg
2 x Pb, S, Sb	Cu	Cl, Hg	Pb, Sb, Hg
Pb, Cl, Sb	K	Cu, Si, Fe, Hg	Pb, Sb, Hg
Pb, S, Sb	Cl, K	Cu, Fe, Hg	Pb, Sb, Hg
Pb, S	Sb, Cu	Si, K, Hg, Fe, Cl	Pb, Sb, Hg
Pb, S, Sb	Cu	Si, Hg, Fe, Cl	Pb, Sb, Hg
2 x Pb	Sb	Cu, Cl, K, Fe, Hg	Pb, Sb, Hg
Pb	Sb, Hg	Cu, Si	Pb, Sb, Hg
2 x Pb, Sn	Cu, Zn	Hg, Si	Pb, Hg (Sn)
Ba, Ca, Si	Pb, S	Cl, K, Cu, Hg	Pb, Ba, Hg
Pb, S, Sb, Ba	Si, K, Cl, Cu	Zn, Fe, Hg	Pb, Sb, Ba, Hg
Pb, S, Sb, Ca, Cu	Ba, Cl, K, Si, Fe	Zn, Hg	Pb, Sb, Ba, Hg
Pb	Si, Sb, Hg	Cl, Cu	Pb, Sb, Hg
Pb, S	Sb, Hg, Cu	Si, Zn, Fe	Pb, Sb, Hg
Pb, Sb	Hg, Cl	Si, Fe, Cu, Zn	Pb, Sb, Hg
Pb, S, Sb	Hg	Cu, Si, Cl	Pb, Sb, Hg
Pb, S, Sb	K, Cl	Cu, Hg	Pb, Sb, Hg

Table 5.26 Mercury containing particles

As can be seen from Table 5.25, even with promptly collected residue, the proportion of mercury containing particles is very low. It is interesting to note that firing 1, the ammunition with the unjacketed bullet, produced fewer Pb only particles than expected but did produce a large proportion of Pb, Sb particles. This suggests that the majority of the Pb, Sb particles originated from the bullet rather than from the primer.

On other occasions mercury containing ammunition has been test fired, the hands sampled immediately, the samples analysed by SEM/EDX, and mercury was not detected in any of the particles. One such test involved the firing of ammunition which, according to the sampling of the interior of the spent cartridge case, had Sb and Hg in the primer. No Hg was detected in any of the discharge particles but Pb and Ba were detected in some particles. The Pb is thought to originate from the base of the bullet (FMJ) and the Ba from inorganic additives to the propellant. This supports the proposition that anything present in a round of ammunition can make a contribution to the composition of the discharge residue particles. The presence of Sn in any of the discharge particles is a strong indication that the primer contains mercury, the Sn originating from the tinfoil disc used to seal mercury fulminate primer cups..

5.4.2 Mercury containing particles in casework

Particles containing mercury are relatively rare in the environment, the only non-cartridge source previously detected being from dental fillings. A brief search of the literature on the uses of mercury revealed that it is used for: fungicides/bactericides, amalgamation, catalysts, special solders (along with Pb and Sn), dental preparations, electrical apparatus, electrolytic preparation of chlorine and sodium hydroxide, government/commercial laboratory use, paint (anti-fouling, mildew proofing), paper pulp (slime inhibitor), pharmaceuticals (ointments, antiseptics, diuretics), manufacture of thermometers and barometers, timber preservation, photography and in the manufacture of mercury fulminate. A representative selection of mercury containing particles detected in casework involving the use of mercury fulminate primed ammunition is presented in Table 5.27 and is included to demonstrate the variety of compositional types.

Major	*Minor*	*Trace*	*Particle Type*
Si, Pb, Fe	Ca, K, Cl	Cu, Hg	Pb, Hg
Sn	Cu	Zn Hg	Hg only
Sb, Sn, Cl, S	-	Fe, Cu, Hg	Sb, Hg
Hg	Cu, Zn	Cl, K, Ca	Hg only
Hg, Cu, Zn	Sb, Sn	Cl	Sb, Hg
Hg	-	Cu, Zn	Hg only
Hg	-	Cu, Zn, Si, Cl	Hg only
Hg	Cl, K	Sb, Cu	Sb, Hg
Hg, S, Sb	K, Cl, Cu	Si	Sb, Hg
Hg,	Cl, K, Cu, Sb	Zn, Si	Sb, Hg
Sb	Cu, Hg, Cl	-	Sb, Hg
Sb, Hg	Cl, Cu	K, Zn	Sb, Hg
Hg	Sb, Cu, Cl, K	Zn, Si	Sb, Hg
Sb, Cu, Hg	Cl	K	Sb, Hg
Sb, Cl, Cu, S, Hg	K, Si	Zn, Al	Sb, Hg
K, Cl	Hg, S, Cu	Sb, Si	Sb, Hg
Si, Pb	Ca, Cl	Al, P, Hg, K, Ti	Pb, Hg
Hg	Ca, Si	Al, K, Fe, Cu, Zn, Ti, Mg	Hg only
K	Hg, S, Sb, Cu	Si, Zn	Sb, Hg
Sb, Cl	K, Hg, S, Cu	Si, Al, Zn	Sb, Hg
Si	Ca, Hg, Ag, Al	Fe, Cu	Dental Filling
Pb, Sb, Hg, Ca	Si, Cu, Cl, Fe, Al	K, Mg, Cr, Ti	Pb, Sb, Hg
Hg, Sb	Cu	K, Cl, Zn	Sb, Hg
Sb	Hg, Cu	Cl, Si	Sb, Hg
Cu, Cl	Hg, K, Sb, Zn	-	Sb, Hg
Cu, Hg, Sb	K, Cl	Zn	Sb Hg
Cu, S, Sb	K, Cl	Zn, Al, Hg	Sb, Hg
Hg, Cu	Zn, K, Cl	Si, Fe	Hg only
Hg, Cl	Cu, Zn	K, Si	Hg only
Cu, Zn	Cl, Sb, Pb, Hg	K, Si	Pb, Sb, Hg
Cu	-	Cl, Hg, Pb, Zn	Pb, Hg
Hg, Pb, Cu, Sb, Cl	-	Zn	Pb, Sb, Hg
Hg, S, Cl, K, Sb	Cu	-	Sb, Hg
Hg	-	Cl, K, Sb	Sb, Hg
Cl, K, Hg	Sb	Cu	Sb, Hg
Sb, Hg, S	Cu	Fe, Cl	Sb, Hg
Sb, Hg, S	K	Ba, Fe	Sb, Ba, Hg
Sb	K, Hg, S, Cl	-	Sb, Hg

Major	*Minor*	*Trace*	*Particle Type*
Sb, Sn	Cu	Hg, Cl	Sb, Hg
Sb	S, Sn	Cu, Hg, Al	Sb, Hg
Cl, K	Hg, S, Sb, Cu	Si, Zn	Sb, Hg
Cl, K, S, Hg, Sb	Cu, Si	Al, Zn	Sb, Hg
Sb, K, Cl, Pb, S	Cu, Al, Hg	-	Pb, Sb, Hg
Pb, S, K, Sb, Cl	Al, Cu	Si, P, Hg, Zn	Pb, Sb, Hg
Cu, Cl, Zn, Hg, Pb, S	K, Sb	-	Pb, Sb, Hg
Pb, S	Sb, Cl, K, Si, Hg	Cu, Zn, Fe	Pb, Sb, Hg
Pb, Sb	Hg, Cu	Si, Al	Pb, Sb, Hg
Hg, Si, Ca	Fe, Cu, Zn, K, Al, Mg, Cl	-	Hg only
Sb, Si	S, Cl, K, Ti, Al, P, Fe	Hg, Cu, Zn	Sb, Hg
S, Pb, Hg, Sb, Si	Fe, Al, K	Cu, Cl, Mg	Pb, Sb, Hg
Hg, S	Al, Si, K, Ca, Fe, Cu	-	Hg only
Pb, Hg, Sb, Ca	Si, Cu, Cl, Fe, Al	K, Mg, Cr, Ti	Pb, Sb, Hg
Pb, Hg, Cu, Sb	K, Cl, Ba	Al, Zn	Pb, Sb, Ba, Hg
Cu, Sb	Pb, Hg	Cl, K, Al	Pb, Sb, Hg
Sb, Cl, Cu, S, Hg	K, Si	Zn, Al	Sb, Hg
Hg	Si, Ca	Al, Fe, Cr, K, Cu, Zn	Hg only
Hg	Cl, K, Cu, Sb	Zn, Si	Sb, Hg
Hg, S	Si	Ca, Ti, Mg, Fe, Cu	Hg only
Pb, Sb	Ca, Hg, Cl, Cu	K, Fe	Pb, Sb, Hg
Hg, Ca	Si, Al	K, Fe, Cu, Zn	Hg only

Table 5.27 Casework particles containing mercury

5.4.3 Distribution of mercury after discharge

A specific ammunition type was examined to determine the total mercury content. Results are given in Table 5.28.

Sample Number	*Mercury µg*	*Average µg*
1	3850	4070
2	3500	
3	3900	
4	4150	
5	4200	
6	4550	
7	4450	
8	4150	
9	4050	
10	3750	
11	4000	
12	4450	
13	4500	
14	3950	
15	3600	

Table 5.28 Total mercury content

Spent cartridge cases, originating from the same ammunition type, were examined to determine the amount of Hg remaining in the cartridge case. Results are given in Table 5.29.

Sample Number	*Mercury μg*	*Average μg*
1	490	533
2	710	
3	555	
4	470	
5	465	
6	695	
7	550	
8	405	
9	680	
10	520	
11	510	
12	545	
13	460	
14	510	
15	425	

Table 5.29 Mercury remaining in spent cartridge case

Using the same ammunition type, the amount of mercury deposited on the perimeter of a bullet hole was then determined. Results are given in Table 5.30.

Sample number	*Mercury μg*	*Average μg*
1	1.32	2.15
2	2.95	
3	3.00	
4	1.85	
5	1.51	
6	2.10	
7	2.23	
8	2.17	
9	2.35	
10	1.97	

Table 5.30 Mercury in bullet wipe

The amount of mercury remaining on the bullet after it had passed through a target was then determined. Results are presented in Table 5.31.

Sample number	***Mercury µg***	***Average µg***
1	11.6	16.78
2	20.8	
3	10.2	
4	20.8	
5	13.9	
6	17.7	
7	16.3	
8	17.7	
9	14.2	
10	14.6	

Table 5.31 Mercury remaining on bullet

The amount of mercury remaining in the gun after discharge was then determined. The results are given in Table 5.32.

Sample number	***Mercury µg***	***Average µg***
1	5.7	6.5
2	7.1	
3	6.8	
4	6.3	

Table 5.32 Mercury remaining in gun

These tests give an estimate of the total amount of mercury in a complete round of ammunition prior to discharge (4070 µg), the amount deposited on the bullet (2.15 + 16.78 = 18.93 µg), the amount remaining in the spent cartridge case (533 µg) and the amount remaining in the gun (6.5 µg). This gives a difference of ~3511 µg (85%) of mercury released into the environment during the discharge process. In order to determine what proportion of this would be detectable by SEM/EDX a further set of experiments were devised. The initial test involved the examination of residue from the discharge of a primer, in a primed cartridge case (no propellant or bullet present), to determine what proportion of the mercury would be detectable in 1 micron or greater, particulate form. Results are given in Table 5.33.

Test No	*Cartridge Case (μg)*	*Filter (μg)*	*Liquid Trap 1 (μg)*	*Liquid Trap 2 (μg)*	*Liquid Trap 3(μg)*
1	969.0	51.6	225.0	23.6	8.1
2	1030.0	50.9	271.0	30.3	9.7
3	941.0	73.0	277.0	28.2	7.9

Table 5.33 Mercury levels from primer discharge

Average results indicate that approximately 24% of the mercury remains in the spent case/primer cup, approximately 7% of the mercury was recovered from the liquid traps and only 1.5% of the mercury was present on the filter. Approximately 68% of the mercury appears to be present as large particulate matter, which must have been deposited on the interior of the firing tube.

The mercury concentration remaining in the spent cartridge case was considerably higher than previously experienced. This test did not reproduce the conditions experienced during the discharge of a round of ammunition, where much higher temperatures and pressures are attained, plus the possible suction effect in the wake of the bullet. To simulate actual conditions a further test was devised.

This test involved the discharge of a complete round of ammunition and facilitated the examination of discharge residue exiting the muzzle of the firearm. The primary objective of the examination was to determine what proportion of the mercury containing discharge residue particulate matter is likely to be detected by SEM/EDX. Results are given in Table 5.34

Test No	Inside Tube (μg)	Filter (μg)	Liquid Trap 1 (μg)	Liquid Trap 2 (μg)	Liquid Trap 3 (μg)
1	444.0	171.0	786.0	6.92	3.66
2	521.0	194.0	919.0	10.70	4.10
3	403.0	165.0	641.0	6.12	4.11

Table 5 .34 Mercury levels in muzzle discharge residue

The proportion of the discharge residue issuing from the muzzle that has the potential to be detectable by SEM is 12.1%, 11.7% and 13.5% respectively. These figures are remarkably reproducible given the scope for experimental error in this type of experiment. The distribution between the filter and the liquid traps i.e. between particulate and vapour; is 17.7%, 17.2% and 20.2% respectively on the filter.

Finally, breech discharge residue was examined to investigate the occurrence of Hg containing particles that would be SEM/EDX detectable. Results are given in Table 5.35.

Test No	Inside Box (μg)	Filter (μg)	Liquid Trap 1 (μg)	Liquid Trap 2 (μg)	Liquid Trap 3 (μg)
1	0.68	2.05	2.56	0.51	0.11
2	0.50	3.01	2.97	0.55	0.11
3	0.57	2.73	2.55	0.73	0.19

Table 5.35 Mercury in breech discharge residue

Averaging the results it would appear that in the region of 6.60 μg of mercury exits via the breech (ignoring spent cartridge case), of which approximately 48% is particulate (approximately 40% was retained on the filter).

Table 5.36 gives a quantitative summary of the measured distribution of mercury after discharge. Percentages are based on an initial amount of 4070 μg.

Distribution	***μg Mercury***	***~%***
Spent Cartridge	533.00	13.10
Bullet Wipe	2.15	0.05
Spent Bullet	16.78	0.40
Residue in Gun	6.50	0.16
Exits Muzzle	1412.00	34.86
Exits Breech	6.60	0.16
Totals	1977.03	48.73

Table 5.36 Distribution of mercury after discharge

A percentage recovery of 48.73 is disappointing. The variation in the amount of mercury initially present in the ammunition (see Table 5.28) is probably due to deterioration of the mercury fulminate over a long period, the ammunition having been manufactured in 1943. The variation would also be a major factor in accounting for the varying amounts remaining in the spent cartridge case. Ideally a recently manufactured batch lot of ammunition should have been used but none was available, and to acquire some in the U.K. in a pistol calibre would be very difficult. From the start it was known that the variation in mercury levels in the ammunition would introduce large errors in the percentage recovery, somewhere in the region of ±16%. However the objective was to explain the scarcity of mercury containing particles from discharge residue, the primary concern being the recovery of particulate matter using a 1 micron pore size membrane filter.

The data confirms that, as expected, the vast majority of residue exits from the muzzle, only 12.4% of which was detected on the membrane filter and 55.5% in the liquid traps, the balance probably remaining in the apparatus. The most important aspect is the discharge residue exiting from the breech, as it is some of this residue that is likely to be deposited on the firer. Only 6.6 μg of mercury was detected (excluding spent cartridge case) of which 39.1% was detected on the membrane filter and 52.0% in the liquid traps, the balance presumably remaining in the firing box. The presence of the spent cartridge case in the firing box may have caused, or contributed to, the amount of mercury remaining in the box.

The primer only discharge test produced interesting results, only approximately 1.5% of the mercury being retained by the 1 micron filter. The spent cartridge cases had a significantly higher level of mercury than when they were discharged normally in a firearm. This is thought to be due to the very substantial temperature and pressure difference between the discharge of non-bulleted and bulleted ammunition, coupled with the absence of a possible suction effect in the wake of a bullet. The physical disturbance of extraction and ejection of the spent cartridge case and the shock it suffers on striking the ground could dislodge mercury containing particles from its surface. In the test only the inside surface of the cartridge case was exposed to discharge residue due to the fact that the outer surface was surrounded by a rubber bung, whereas in normal use in a firearm the outside surface would also be exposed to a residue laden environment.

The low concentration of mercury on the filter is surprising given that only the initial source, the primer, was involved plus the smaller volume of the firing tube, compared to the other experiments, which should have increased sampling efficiency. This tends to suggest that the majority of the mercury was contained in large particles that were deposited on the inside of the firing tube, the particles being too large to be circulated by the pump.

The primer is the origin of the mercury, the average amount being 4070 µg which is equivalent to approximately 5775 µg mercury fulminate. This is burnt at high temperature and pressure, the vast majority of it being expelled with force from the muzzle and away from the firer. The pistol used in the tests works on the blow back principle. This involves the use of gas pressure to move the slide to the rear, thereby extracting the spent cartridge case from the chamber. The ejector then expels the spent cartridge case through the ejection port and the slide moves forward again under spring pressure and, in doing so, chambers another round from the magazine. It is the blow back 'gases' that are the most likely to be deposited on the firer under normal circumstances. Table 5.37 gives a comparison between quantities detected on the filter and liquid traps.

	FILTER		***LIQUID TRAPS***	
	Amount Hg µg	***%***	***Amount Hg µg***	***%***
Primer Discharge	58.50	17	293.60	83
Muzzle Residue	176.70	18	793.90	82
Breech Residue	2.60	43	3.43	57

Table 5.37 Quantitative comparison between filter and liquid traps

There is remarkable agreement between the primer discharge and muzzle residue, filter to liquid ratios. The breech residue shows a marked difference which is difficult to explain, the filter retaining a much higher percentage of the mercury. It is possible that the filter picked up some larger particles, with high mercury content, from the ejection of the spent cartridge case. This is unlikely and would need to occur in all three tests.

There was debris inside the firing tubes after discharge of the primers and bullets. It was a sooty deposit which contained particulate matter. All the filters were dirty after use, the residue being grey/black in colour, and no large particles were noted on the surface of the filter on visual examination. There are three distinct forms of residue involved, large particles that were not transported through the sampling system, smaller particles that were transported through the sampling system and which are greater than 1 micron in size and vapour/sub-micron particles that passed through the filter and were retained in the liquid traps.

It is obvious that there is some form of concentration effect of the particulate matter in the breech emission. When a round of ammunition is discharged in a firearm there is considerable pressure generated, which acts in all direction including rearwards. It is the rearward pressure of the cartridge case acting on the breech face which drives the slide backwards. This backward pressure may also tend to contain the bulk of the primer particles, which are at the start of the discharge process, and create a zone of discharge residue, travelling rearwards, which is rich in particles originating from the primer. Whatever the reason the residue exiting from the breech has a higher concentration of small detectable particles than the muzzle exhaust.

From the tests it is clear that a large amount of mercury is present in vapour/sub-micron particle form and is consequently not detectable by SEM. A large proportion of the mercury is also present in large particulate matter, which is very unlikely to be deposited on the firer as it exits from the muzzle and travels a considerable distance away from the firer. Only 2.6 μg mercury that exited from the breech and possibly 0.55 μg present in the sampling box (which may have originated from the spent cartridge case) is potentially detectable on the firer. In practice only a small proportion of this will be deposited on the firer as it will disperse in all directions. Given the low percentage of mercury containing particles detected in promptly collected residue from indoor firings under favourable laboratory conditions, it is not surprising that very few mercury containing particles are detected in casework.

The nature of the tests performed is such that large experimental errors are involved. Care was taken to ensure that all the apparatus was airtight and all manipulations, extractions, washings etc., were done as carefully as possible. Despite the care taken a low percentage recovery of mercury resulted. Some mercury was probably lost by adsorption /absorption on to / in to the metal (firing tube), rubber (bungs and gaskets), plastic (tubing) and glass (liquid traps). Some may have been lost through the pistol after discharge, as the pistol would not be airtight. The rinsing of the firing tube, after discharge and suction sampling, was difficult and probably very inefficient and no rinsing of the glass tubing in the liquid traps or plastic connecting tubing was attempted. The possibility of particles present in the wake of the bullet exiting the firing tube along with the bullet cannot be excluded. It is likely that a small amount of mercury would have been lost from the surface of the bullet into the bullet recovery medium.

The pump used was not very powerful (18 l/min). This was by choice, as a more powerful pump could possibly have cause larger particles to enter the sampling system, which would not reflect a real life situation, as the larger particles would not remain airborne for long.

Despite the experimental errors it is obvious that a high percentage (86%) of the mercury is released into the atmosphere, the majority via the muzzle. The majority (88%) that exits the muzzle is not detectable by SEM. A very small percentage exits via the breech (0.16%) of which only ~40% is detectable by SEM.

Indications are that the majority of the mercury is present in sub-micron particulate/vapour form and in large particulate form which is unlikely to be deposited on the firer. All these observations account for the scarcity of discharge residue particles containing mercury. The scarcity of mercury containing discharge particles on the firer has been confirmed by other workers (192).

A surprising result was the amount of mercury remaining on the bullet after it had passed through a wad of filter paper and the bullet recovery medium. Mercury was also readily detectable in the bullet hole perimeter tests (Tables 5.18 and 5.19).

The possibility of relating bullet damage to a particular round of ammunition by chemical comparison of the deposit on the bullet hole perimeter with the deposit on the spent bullet should be investigated.

It would be desirable to repeat the experiments using recently manufactured batch lots of ammunition and testing for Pb, Sb, Ba in addition to Hg. It would also be desirable to examine a range of ammunition and handguns, rifles being difficult, although not impossible to accommodate because of size and pressure generated on discharge. Unfortunately time did not permit. An improved design of apparatus would incorporate the choice of a closed or open breech firing mechanism with bullet recovery included in the design, such as shown schematically in Figure 5.05, volumes of both sampling areas to be kept to a minimum.

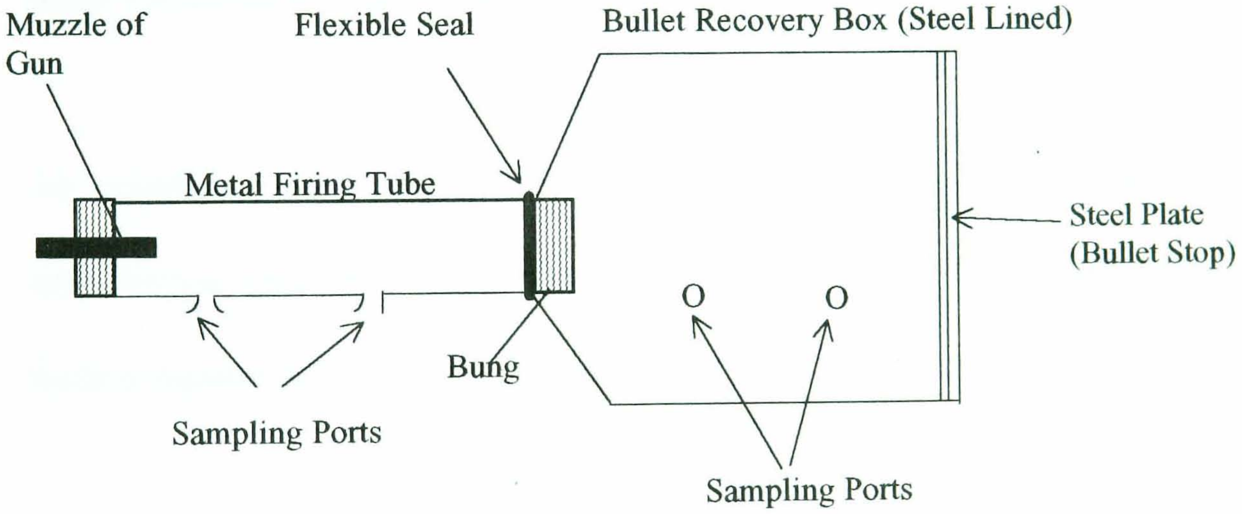


Figure 5.05 Improved residue sampling apparatus

It would also be desirable to investigate the use of acidified potassium permanganate as a means of absorbing the mercury vapour, which can then be titrated with dithizone for quantitative analysis.

To finalise the series of tests on the distribution of mercury a further test was conducted to determine the amount of mercury deposited on the firer. The results are given in Table 5.38. No mercury was detected in the control samples.

Number Shots	***ng Mercury***				
	R Hand	***L Hand***	***Face***	***Hair***	***Clothing***
1	87	None	None	None	None
1	40	None	22	None	70
3	56	None	20	None	68
3	25	None	None	None	79
7	66	None	29	None	88
7	170	11	42	44	60

Table 5.38 Amount of mercury deposited on firer

The results demonstrate the random nature of FDR deposition, however, the firing hand was positive in all firings.

An ancillary test involved examining discharge residue particles from the ammunition used in the mercury distribution tests. Approximately 260 particles were examined of which 8 contained mercury (~3%) at minor or trace level.

This number of particles is unlikely to account for the levels of mercury on the firing hand in the previous test. The possibility that some non-SEM detectable mercury is deposited on the firer cannot be excluded.

5.4.4 Loss of mercury from spent cartridge cases

The rate of loss of mercury from spent cartridge cases was examined to determine if this would assist in estimating the time of discharge. Results are presented in Table 5.39.

Time Interval	*Weight Range μg Hg*	*Average μg Hg*
Unfired	3813-5938	4952
Immediate	710-860	787
3 days	655-905	789
10 days	710-890	780
69 days	444-631	537

Table 5.39 Loss of mercury with time

As can be seen the rate of loss of mercury is too slow to be of practical value in estimating time of discharge. Again there is wide variation in the amount of mercury initially present which is reflected in the variation in the amount remaining in the spent cartridge case. The amount remaining in the spent cartridge case immediately after firing (~16%) is roughly in agreement with that determined in the distribution experiment (~13%). The slow rate of loss of mercury (~32% in 69 days) coupled with the wide variation in amounts initially present excludes the mercury level as a useful indicator of the time of discharge.

A further test involved storing spent cartridge cases for 3 days at 80^0 C.The elevated temperature greatly increased the rate of loss of mercury, the weight range being 351-432 μg, the average being 378 μg. This suggests that climatic conditions (temperature) would be amongst the many factors involved in the rate of loss of mercury from the spent cartridge case.

5.5 *LEAD FREE AMMUNITION*

Airborne lead, in both particulate and vapour form, arising from the discharge of ammunition, can be a health hazard to those regularly exposed over a long period of time. People, such as firearms training instructors, are at risk as lead has a cumulative effect in the body, eventually leading to serious illness. Antimony and barium are also undesirable from a health viewpoint, but their levels in discharging residue are considerably lower than the lead level. Despite elaborate indoor range extraction and ventilation systems, which do much to reduce the risk, continuous exposure over the years is undesirable and many organisations monitor blood lead levels (and hearing) of staff involved.

The first serious attempt to solve the problem at source was in 1978 when Smith & Wesson encased the entire lead bullet in a coating of black nylon (Nyclad ammunition). This substantially reduced the amount of lead released into the atmosphere from the discharge process, greater than 60% reduction when compared to the 'cleanest' of conventional lead bulleted ammunition. As mentioned previously, most of the lead originates from the bullet, the lead in the primer making a much smaller contribution.

Analysis of a round of .38 Special calibre, Smith & Wesson Nyclad ammunition revealed that, the cartridge case and primer cup are Ni plated brass, no inorganic additives to the propellant were detected, the primer composition contains Pb, Sb and Ba, the bullet core is Sb hardened Pb and the bullet jacket contains traces of Ca, Co, Ti and P.

The Northern Ireland Forensic Science Laboratory has been involved in the assessment of such ammunition for the police and tests were conducted to compare lead levels using Nyclad and conventional ammunition. Unfortunately the tests results did not survive the explosion at the laboratory, but the overall conclusion was that the claims made relating to the reduction of lead were more than justified. (Other tests involving the examination of the perimeter of bullet holes in cloth caused by Nyclad bullets revealed that Co was repeatedly and readily detected by FAAS).

The next stage in the development of environmentally friendly ammunition was when Geco produced ammunition with a totally jacketed bullet and a primer composition containing much reduced Pb, Sb and Ba levels. An example of such ammunition was compared with its nearest equivalent to indirectly assess its effectiveness. Results are given in Table 5.40.

Ammunition details	***Pb μg***	***Sb μg***	***Ba μg***
Geco .38" Special, Metal piercing bullet with ENCLOSED base	4.2	1.4	0.06
Geco .357" Magnum, Metal piercing bullet with EXPOSED base	91.5	130.0	4.4

Table 5.40 Geco primer analysis

The .38" Special ammunition with the bullet base enclosed coupled with the substantially lower levels of Pb, Sb and Ba in the primer, will obviously give much lower levels of Pb, Sb and Ba in the discharge products than the .357" Magnum calibre ammunition.

The nylon coated bullets from Smith & Wesson and the Geco total metal jacket bullet/new primer composition, whilst effective in markedly reducing the lead levels, did not totally eliminate the problem. In 1983, Dynamit Nobel introduced 9 mmP calibre ammunition with a total metal jacketed bullet and a Pb, Sb, Ba free primer. The new primer type was called Sintox. A typical Sintox primer composition contains 15% diazodinitrophenol (DDNP) and 3% tetracene as the 'explosive' ingredients, 50% zinc peroxide as the oxidiser, 5% of 40 micron size titanium metal powder, and 27% nitrocellulose as propellant powder (193). As expected other munitions manufacturers eventually introduced similar ammunition, some with primers that were lead free but containing Sb and Ba and others Pb, Sb and Ba free. The objective was to produce ammunition which performed satisfactorily in every way and did not produce any toxic product on discharge.

A selection of 'lead free' ammunition was dismantled and analysed and the results are given in Table 5.41

Ammunition	*Cartridge Case*	*Primer Cup*	*Bullet*	*Propellant*	*Primer*
9 mmP IVI 92 ⊕ Greenshield TM (1)	Brass	Ni plated brass	Frangible - powdered Cu in caprolactam binder. A number of alkane nitriles and long chain alcohols also detected.	NG, EC, trace DPA, K, S also detected	Sb, Ba, S (trace Fe, K, Cl, Si, Cu, Zn Na, Ca) Dinitrophenol
CCI 9 mm LUGER Speer-Lawman CF (2)	Brass (trace Fe)	Ni plated brass	Lead core (trace Cu, Fe, Sb) Total Cu jacket.	NG, EC, trace DPA, K, S also detected	Sr, Ti S (trace Ba, Fe, Ca, K, Cu, Zn Cl) Dinitrophenol, EC, DPA (trace DNT, a nitro DPA, a phthalate plasticiser)
CCI NR 9 mm LUGER BLAZER (3)	Al (trace Cu, Mn)	Ni plated brass	Lead core (trace Al, Fe) Total Cu jacket	NG, EC, trace DPA, K, S also detected	Sr, S, Al, Fe (trace Ca, Cl, Ti, Cu, Si, Mg, Zn) EC, DPA (trace DNT, a phthalate plasticiser, a nitro DPA, benzyl butyl phthalate plasticiser)
GFL 9 mm LUGER FIOCCHI (4)	Brass	Brass	Lead core, Al base cup Full brass jacket	NG, EC K, Cl (trace Cu, Zn, Cl, K, Ca, Fe, S, Ti, Mg, P) also detected	Sb, Ba, S (trace Al, Si, Fe, Cu) dinitrophenol
✧—✧ 9 mm LUGER DELTA (5)	Brass (trace Al)	Ni plated brass	Frangible - powder Cu with a trace of W in caprolactam binder. A number of alkane nitriles also detected.	NG, EC, trace DPA K, S also detected	Sb, Ba, S (trace W, Cu, Ca, Fe, Si, Zn, Al, Mg) Dinitrophenol, NG and a number of N-alkanes
SINTOX 9 mm LUGER (6)	Brass	Ni plated brass (trace Fe)	Lead core (trace Fe, Cu, Sb) Brass (trace Fe) base cup. Full brass coated Fe jacket (trace Al)	NG, DPA, a phthalate plasticiser (trace DNT and a nitro DPA) K, Ca (trace S) also detected	Zn, Ti (minor S, trace Cl, Al, Cu, Ca, Fe, K, Si, Na) Dinitrophenol, a methyl propyl phenol, DPA, a phthalate plasticiser, a trace of DNT

Ammunition	*Cartridge Case*	*Primer Cup*	*Bullet*	*Propellant*	*Primer*
9 x 19 SX DAG 88 (7)	Brass (trace Al)	Ni plated brass	Solid brass with a plastic filled hollow point nose	NG, DPA, a nitro DPA, a phthalate plasticiser (trace DNT and other phthalate plasticisers) K, trace S also detected	Zn, Ti (trace Cu, Ca, K, S, Fe, Si) NG, DPA, a phthalate plasticiser, a methyl propyl phenol, trace DNT
WIN 9 mm LUGER DELTA (8)	Brass	Ni plated brass	Fangible - powdered Cu with minor W in caprolactam binder. A number of alkane nitriles also detected.	NG, EC, trace DPA K, S also detected	Sb, Ba, S (trace Fe) Dinitrophenol (trace DPA, EC)
CCI NR 38 SPL +P BLAZER (9)	Al (trace Cu, Mn)	Ni plated brass	Lead core (trace Fe) Total Cu jacket	NG, EC, trace DPA K, S also detected	Sr (trace Na, S, Al, Ca, K, Ti) Dinitrophenol, EC, DPA (trace DNT, 2 x nitro DPA, a phthalate plasticiser)
WIN 9 mm LUGER Winchester Super X (10)	Brass (trace Fe)	Ni plated brass (trace Fe)	Lead core (trace Al, Fe) Brass (trace Fe) base plate Full Cu jacket (trace Fe, Zn)	NG, DPA, a nitro DPA, a phthalate plasticiser (trace DNT and EC) K, S, Ca also detected	Zn, Ti (minor S, Ca, K, Si, Mg, Al, trace Fe, Cu) A series of N-alkanes with phthalate plasticisers
M&S 38 SPECIAL (11)	Brass (trace Al)	Brass	Frangible - powdered Cu, Al in caprolactam binder. A number of alkane nitriles also detected.	NG, EC, 2 x nitro DPA (trace DNT, glycol dilaurate plasticiser) K, trace S also detected	Pb, Sb, Ba, Al (trace S, Ca, Cu, Fe, Si) NG and a number of N-alkanes
.223 IVI ⊕ 91 (12)	Brass (trace Fe, Al)	Ni plated brass (trace Fe, Al)	Frangible - powdered Cu, Al in caprolactam binder. A number of alkane nitriles and long chain alcohols also detected.	NG, DPA, a phthalate plasticiser with trace EC, DNT, a nitro DPA K, S, Ca also detected	Sb, Ba, S, Ca (trace Al, Cl, Cu, K, Fe) NG, a phthalate plasticiser and a number of N-alkanes
HP .223 5.56 CF (13)	Brass (trace Fe)	Ni plated brass	Plastic core Full Fe jacket, copper coated	NG, DPA, a phthalate plasticiser (trace DNT, EC, a nitro DPA) Ca, K, S, Cl also detected	Sr (minor Ti, trace Cu, Ca, S, Zn, K, Fe) Dinitrophenol, DPA, EC, a phthalate plasticiser (trace DNT, methyl propyl phenol)

Table 5.41 Analysis of lead free ammunition

The analysis of the organic components in the primers could suffer contamination from the propellant, although the results suggest that this is not the case. Dinitrophenol is probably a breakdown product of DDNP arising from the conditions used in GC/MS.

Environmentally friendly ammunition was initially introduced for training purposes in indoor firing ranges, but it is now gaining acceptance as conventional ammunition (193). The use of such ammunition has yet to be encountered in casework in Northern Ireland but it is highly likely that it will be in the future. This will pose major problems for existing methodology in the characterisation of discharge particles by the Particle Analysis method, in range of fire determination and in the identification of bullet holes. Some workers are already addressing the problems (191, 193, 194, 195, 196, 197).

Anticipating its use in casework it was decided to look at the nature of discharge particles from lead free ammunition. Typical results are presented in Table 5.42. Nothing of significance was detected on the control samples.

Ammunition	Typical Particle Types			Comment
	Major	Minor	Trace	
Numbers 1, 4, 5, 8 and 12 Table 5.41 Sb, Ba primed	S, Sb, Ba, Cu	-	K, Cl, Si	Size range 1 to 14 microns Large number of Sb, Ba particles No. 5 produced a number of particles containing tungsten Mainly irregular particles
	S, Sb, Ba	-	K, Cl, Si, Cu	
	Cu, Zn	S	K, Sb, Ba, Si	
	S, Cl, Sb, Ba	Cu	Si, Na	
	Ba, S, Cl	-	Cu, K, Si, Ca	
	Sb, S	-	Cu	
	Ba	Sb, S	Si	
	Sb, Ba	S	Cu	
	Sb	-	-	
	Cu, S, Ba, Sb	Zn, Si	Fe	
Numbers 2, 3, 9 and 13 Table 5.41 Sr, Ti primed	Sr	S, Ca	Ti, Cu, K	Size range 1 to 10 microns Large number of Sr, Ti particles and Sr only particles detected A few Ti only particles were noted Mixture of irregular, spherical and oval particles
	Sr, Ti, S	Ca, K	-	
	Sr	-	S, Ca, K, Ti, Zn	
	Sr, S	Ti, Cu	K	
	Cl, Sr, Ti	S, Cu, K	-	
	Sr, Al	-	Ca, K, S	
	Sr	-	-	
	Ca, Ti, Si, S	Zn	Cl, Cu, Al	
Numbers 6, 7 and 10 Table 5.41 Zn, Ti primed	S, Zn	-	Cl, Al, Cu	Size range 1 to 6 microns Large number of Zn, Ti particles A substantial proportion of Zn only and Ti only were detected Mostly spherical and oval particles
	Zn, Ti	Ca,	Cu, S	
	Ti, Ca	Zn	S, Cu, Si	
	Ti	-	Zn, Ca, K	
	Zn, Ti	S, Al	-	
	Zn, Ti	-	S, Na	
	Ti	Ca	-	
	Zn	Ti	Ca	
	Zn	-	-	
	Zn	-	Cu	

Table 5.42 Discharge particle types from lead free ammunition

Ammunition with primers containing Sb, Ba should not pose a problem, as discharge particles produced contain Sb and Ba, particles classified as unique to the discharge of a cartridge. However the other primer types will cause problems. A study of the discharge particles from Sintox ammunition concluded that morphology is an essential identification criterion and that spherical particles composed mainly of titanium and zinc can be used to identify discharge particles from Sintox ammunition (193). [It is reported that Dynamit Nobel have replaced calcium silicide with titanium in Sinoxid type primers. Consequently, titanium as well as Pb, Sb and Ba may be encountered in discharge particles.]

It would be desirable to examine environmental and occupational particles containing one or more of the elements Sr, Ti and Zn and to compare them with discharge particles, in order to establish whether or not it is possible to differentiate the FDR particles. If not the Particle Analysis method may still be a useful tool to provide supporting evidence, particularly if organic propellant constituents are also detected on the suspect.

6 *SUSPECT PROCESSING PROCEDURES -*

Discussion and Recommendations

6.1 INTRODUCTION

The sequence of events associated with FDR examination is usually as follows: the initial incident, apprehension of suspects, transporting of suspects to police station, sampling of suspects at police station (swabs of hands, face, head hair and seizure of clothing), submission of items to the laboratory, sampling of clothing at the laboratory, sample preparation, analysis of samples, interpretation of results, preparation of statement of witness report, and the presentation of forensic evidence in court.

With any process involving sampling, if the sampling is poor or incorrect, then all subsequent manipulations, observations and conclusions can be devalued or meaningless. It was decided to review the complete process, starting with the processing of suspects.(The review was done prior to the development of a method for organic FDR detection, in the hope that improvements could be identified and implemented in suspect processing procedures, thereby increasing the chances of obtaining positive results in most areas of forensic work, including the existing inorganic FDR system and any new organic FDR system). Procedures for dealing with suspects from the time of apprehension to the time of forensic sampling were considered with a view to identifying any weaknesses in the system that could result in contamination and/or loss of any type of forensic evidence. Recommendations designed to eliminate those weaknesses should then, if implemented, increase the chances of obtaining forensic evidence and/or strengthen the value of evidence obtained.

There are two very important factors involved in the processing of suspects for forensic evidence, namely:-

1. time delays which substantially diminish the chances of obtaining evidence.
2. contamination risks which substantially diminish the value of evidence obtained

6.2 TIME DELAYS

Time delays greatly reduce the chances of obtaining positive results, a fact that is well documented in the scientific literature e.g. reference (176, 177). This applies to a greater or lesser extent to nearly all types of forensic evidence originating from the examination of suspects. Firearms and explosives residue in particular are lost very rapidly with the passage of time (90% of firearms residue is lost from the hands in the first hour after firing, even during the course of normal activity). Suspects must be sampled as a matter of urgency.

Reasons for delays include the use of civilian scenes of crime officers (SOCO), lack of availability of any SOCO, lack of availability of other trained samplers and materials, organisational problems and geographical locations.

The use of civilian SOCO for swabbing causes problems whenever the suspect does not consent. When this happens the permission to swab must be given by an officer of at least Superintendent rank before samples can be taken, by force if necessary. A civilian SOCO still cannot take the samples as they can only be taken by a police officer. This causes lengthy delays as a trained police officer must then be tasked. Civilian SOCO's should not be used for this purpose.

Large time delays can occur in certain cases because of geographical locations. Suspects should be taken to the nearest sampling location irrespective of divisional boundaries. After sampling they could then be transferred to the appropriate premises if necessary. Although this may generate extra paperwork and inconvenience, obtaining evidence should be the paramount consideration.

At each sampling location there should be trained personnel on site at all times. To this end CID officers should be trained in the complete sampling procedures for suspects and consideration should be given to the training of relevant uniformed police. The more trained personnel available at any location, the greater the chances of reducing time delays to a minimum.

At each sampling location all the necessary sampling materials should be readily available at all times. A clean store should be provided at each sampling location for storing materials required for the forensic sampling of suspects. This would avoid the possibility of kits or packaging materials becoming contaminated whilst stored at other locations or carried in vehicles.

A conveniently situated modest sized (~120 sq. ft) store would suffice and it should be smooth, light coloured, lino floored with formica type shelving, non-heated and dry. (Bear in mind that some sampling kits contain flammable solvents). The store should be lockable and under the direct control of an appointed officer and used ***exclusively*** for the storage of suspect sampling materials for use at that location only. There must be a strict accounting system for all materials.

This is the most obvious solution to the time problem and should be given very serious consideration. Having fully trained samplers permanently on site, 24 hours a day every day, coupled with all the necessary sampling materials permanently on site, is by far the most sensible and effective way of solving many of the time delays.

6.3 CONTAMINATION AVOIDANCE

The major problem associated with trace evidence is the possibility of cross transfer to the suspect from some unrelated source. In Northern Ireland there must be a greater contamination risk than in the rest of the UK, due to the relative abundance of firearms and explosives. Contamination risks have been exaggerated out of all proportion and all our problems in this area stem from the difficulty in providing basic facts, knowledge and statistics to demonstrate that cross contamination is unlikely to have occurred. This statement is based on court experience and the fact that the vast majority of suspects examined for firearms and explosive residues are negative. If contamination is such a problem it is certainly not reflected in laboratory results. None of this is meant to suggest complacency. These 'negative' suspects are ideal 'controls' and consequently provide the ammunition with which to hit back at the frequently used defence of cross contamination. This defence is very often invalid but hard to refute completely. To resolve this situation accurate casework statistics on computer should be compiled for use in court.

In multi - suspect cases the suspects should be separated immediately and remain separated at all times. Suspects are apprehended and conveyed to the sampling location in police vehicles. On arrival they may initially be individually placed in a room to await SOCO examination, or they may go to the cell block to be 'processed' in the custody office, prior to being placed in a cell or room before SOCO examination. (Processing includes recording details about the prisoners and their arrest, photography of prisoners and the removal of personal items from them. Fingerprinting, if required, takes place after the SOCO examination as does the medical examination). If the SOCO examination takes place after the processing procedure, then the possibility of cross contamination is increased because all suspects are individually taken, one after the other, into the same room where they probably sit in the same chair and lean on the same desk top during the processing stage.

Ideally suspects should go directly to a room and be under *constant* supervision until sampling is complete. They should then go to the custody office for processing. It would not be desirable for the custody officer to visit the prisoners at this stage, as this would involve him going from room to room thereby creating a cross contamination risk. Obviously if only one suspect is involved, then the problem is not as severe, but there is still the possibility of cross contamination from suspects in other cases.

It is most important that all suspects be kept separated and supervised at all times between arrest and sampling. On arrival at the sampling location they should individually be taken directly to a room and be under constant supervision until sampling is completed. This would avoid the risk of cross contamination during the processing stage.

In some cases the requirement for forensic sampling may not be known until after the suspect has been processed and placed in a cell. It is important to avoid contamination risks in the cell blocks for this reason and also to exclude the possibility of a residue-free suspect becoming contaminated as a direct result of police custody.

To minimise the risks of contamination in the cell blocks, the following procedures are recommended:

(a) Custody Offices

The chairs used to seat the suspects should be the smooth plastic covered type with no breaks in the surface. The chair and desk top should be cleaned between suspects using an X-ray medi-wipe. This simple, fast and cheap procedure will substantially diminish cross contamination risk.

(b) Cells

Cells are often furnished with a bed that contains a mattress and pillow, both of which are covered with a heavy duty plastic material. Disposable type paper sheets are usually provided, which may or may not be used by the suspect. The sheet type paper covers are disposable and changed for each suspect, but the blanket/s, whilst dry cleaned at frequent regular intervals, are not so changed. Clean blankets should be provided for each suspect in addition to the disposable pillow and mattress covers. This would avoid any cross contamination risk and would also be beneficial as regards hygiene.

Cells should ideally be cleaned between each suspect or, if this is not a practical proposition, at frequent regular intervals. When this is done, both sides of the pillow and mattress should be cleaned using a clean damp cloth and disinfectant.

(c) Clothing Containers

Clothing containers are plastic type covers with zip closure and are designed to hold some, or all of a prisoners clothes. One container is placed on the wall outside each cell. If they are non-disposable there is a strong likelihood of all forms of contamination being concentrated and spread by their use.

All such containers should be disposable and changed between suspects.

(d) Sampling Rooms

A single clean room to be used exclusively for the forensic sampling of suspects has often been proposed. Whilst the idea has some merit I feel that it is not a practical proposition. Such a room would concentrate all contamination problems in one area, cause a risk of fibre cross contamination and cause lengthy time delays in multi-suspect cases. However a number of such rooms would be the ideal situation.

The rooms should be conveniently situated and should be furnished and decorated in such a way as to avoid porous or rough surfaces, such as carpet or chairs upholstered with cloth material. The following type of room is suggested.

Furniture	-	A suitable sized table with a smooth, white formica type top.
	-	Chairs to be light coloured with smooth plastic covered padding.

- Medium sized waste bin lined with a disposable plastic bin bag.
- A wall mounted shelf ~3' long and 9" deep, lined with smooth white formica type material.

Decoration - Light coloured, smooth surface lino type flooring.

- Smooth surface, white, half tiled walls from floor level. Remainder of wall to be painted with white paint.

All sampling rooms should be no smoking areas. Because of the light coloured decor, dirt would be readily seen and the smooth surfaces would facilitate easy cleaning. The tiles, floor, table, shelf, chairs and bin should be cleaned at frequent regular intervals, preferably after each suspect sampling. Cleaners should have dedicated cleaning equipment and protective clothing.

6.4 EVIDENCE PROTECTION KIT

One potential major improvement in suspect handling lies in the use of a kit, specifically designed to retain evidence on a suspect. It has for a long time been a source of annoyance and frustration that, at the time of apprehension of a suspect, there are currently no effective measures available to secure potentially crucial evidence. Some police forces throughout the World use bags, usually plastic ones, to cover the hands of persons suspected of firing a gun. The use of plastic bags can have a detrimental effect on FDR, causing the hands to sweat, which in turn causes most of the FDR to be removed from the hands on to the surface of the bag. Apart from protection of the hands, no effort has been made to protect other suspect sampling sites.

Existing systems fail to prevent the loss of evidence from the most significant areas during the period between apprehension and sampling, as well as providing many opportunities for cross contamination to occur. If one could prevent the loss of evidence and confine any contamination risk to the initial arrest procedure, the advantages would be enormous.

A kit for the protection of a suspect's hands before sampling for firearms and/or explosives residue has been used in Northern Ireland for many years. The kit involved placing the suspect's hands inside paper bags to prevent the removal of residue prior to sampling. In use it was cumbersome, the bags having to be applied before securing the suspects arms. Unfortunately, suspects are rarely apprehended until several hours after the incident, and due to the rate of loss of such traces from the hands, the success rate was very low. The suspect's upper outer garment proved to be a much more fruitful area for the detection of firearms and explosives residue. This led to some thought being directed towards a means of protecting both the upper outer garment and hands prior to sampling, in an effort to reduce the rate of loss of all types of forensic evidence. Consequently, a kit for use by the police during apprehension of the suspect was designed and commercially produced, and has been operational since early 1993 without problems. Details of the kit have been published (198) and the kit is described in Chapter 4.

6.5 COMMENT

The single most important factor in the avoidance of contamination problems is good housekeeping. This applies to all aspects of trace residue examination procedures, and includes police vehicle interiors, custody offices, cells, sampling rooms and samplers premises.

The use of SOCO to sample suspects should be reviewed. The fact that SOCO are involved in the examination of scenes of crime in which firearms and explosives are employed and also in the sampling of suspects for trace levels of same, raises cross contamination possibilities and is a source of concern. In practice the sampling kit employs elaborate contamination avoidance procedures; consequently the risk of contamination of the suspect by the sampler is extremely remote. However the perception of risk could cause problems. The examination of scenes should be divorced from the sampling of suspects to overcome this difficulty.

It is highly desirable for a forensic science laboratory engaged in such work, to frequently monitor the sampling procedures and sampling environment used by the police in order to be in a position to demonstrate the validity of laboratory results. This would involve an ongoing commitment of laboratory resources, such as regular monitoring of sampling locations, samplers premises and vehicles and kit making premises and products, for the presence of FDR and explosives residue, in addition to the compilation of computer records of all casework involving the examination of suspects for either FDR or explosive residue.

The benefit is the ability to effectively refute cross contamination allegations in court, thereby increasing the confidence level of everyone involved, in both the laboratory results and interpretations based on the results.

A further improvement in contamination avoidance, instigated by the police, is the compulsory use of a contamination avoidance kit by anyone entering the scene of a crime. I was approached to design such a kit and my initial thoughts are:- a suitable sized nylon bag divided into two sections by the use of a heat sealer, the first (upper) section containing instructions, an x-ray medi-wipe and a pair of large size disposable plastic gloves and the second (lower) section containing a large size disposable overall with integral hood and overshoes. A demonstration kit was prepared and is currently being considered by the police.

In conclusion, it is my opinion that laboratory results should not be considered in isolation but should be interpreted with the knowledge that established and reliable contamination avoidance procedures are in operation and that they are monitored on a regular basis.

Note:

Most of the recommendations contained in this Chapter have been accepted and implemented by the police, including the provision of stores, refurbishment and provision of suspect processing rooms, use of evidence protection kit and regular monitoring of premises for firearms and explosives residue.

7 FIREARMS DISCHARGE RESIDUE - ORGANIC - Results and Discussion

7.1 INTRODUCTION

There are several reasons why the detection of organic constituents of FDR was worthy of investigation. The Particle Analysis method is tedious and the speed of analysis is very slow, thereby often not meeting all the needs of the police, who frequently require a fast answer at an early stage in an investigation. The detection of organics is much faster, particularly for multi - suspect cases involving a large number of samples, and it was hoped that the time problem could be partly solved by screening for organics prior to inorganic examination, enabling preliminary results to be available for the police investigation.

If the organic system was as sensitive, if not more so, than the inorganic system and if there was a very good correlation between the detection of organic and inorganic FDR, then it would be sensible to use organic analysis to determine which samples are worthy of lengthy SEM/EDX examination. The vast majority of samples examined by the laboratory are negative for inorganic FDR. Our current screening technique is automated SEM/EDX (145,146), which is a lengthy procedure. Positive samples are confirmed by manual examination, also a lengthy procedure.

The majority of terrorist shooting incidents in Northern Ireland involve the use of single base propellants and as the main thrust to date in organic FDR analysis had been based on the detection of NG from double base propellants, using techniques such as GC/TEA and HPLC/PMDE, it was obvious that GC/ MS was the instrumentation most likely to satisfy the requirement of detecting constituents from single base propellant. From the start, it was realised that the chief problem was the low concentration of detectable constituents present in single base propellants which, after combustion, would be present at extremely low levels in the discharge residue. To increase the chances of success it was decided to optimise all aspects of the system, starting with decreasing the suspect sampling time by the police and increasing the efficiency of sampling techniques used by the police and the laboratory. From the outset, this work was planned so that all subsequent development work could be based on optimised and standardised sampling techniques.

The ability to detect organic constituents in FDR from ammunition with single base propellant was the ultimate goal. If this proved impossible, the investigation would at least clarify the situation and improve the detection method for NG, which could serve as a useful complementary technique to SEM/EDX. If particles in the 'indicative' category were accompanied by NG, it would substantially raise the significance level of such particles.

In the Northern Ireland Forensic Science Laboratory two separate sections dealt with the detection of firearms and explosives residue. The trace analysis work of the firearms section and the explosives section are now done in one laboratory, called the microchemistry laboratory. Each section had two sampling kits, one for the sampling of suspects and one for the sampling of miscellaneous items, and each section had a different method for sampling clothing from suspects. It was decided to look at the possibility of using the same kit to sample for both firearms and explosives residue and to investigate the feasibility of a joint procedure for sampling clothing. Both the kit and clothing sampling procedures would have to be compatible with existing residue detection systems, primarily organic in the case of explosives, and inorganic in the case of firearms. It was also necessary to ensure that the organic components of FDR could be accommodated using existing instrumentation and that a single sample could be analysed for both firearms and explosives residue. In the past separate samples would have been taken, one for FDR and one for explosives residue. The desirability of routinely screening all terrorists suspects for both FDR (inorganic and organic) and explosives residue was also borne in mind.

A joint sampling procedure for clothing was devised and the four previous sampling kits were reduced to two, a joint kit for suspects and one for miscellaneous items.

7.2 *SAMPLING OF CLOTHING*

In this section, the development of a clothing sampling method, common to both explosives and firearms residues, is described.

Previous experiments on clothing demonstrated that suction sampling is considerably more efficient for the removal of FDR than either adhesive lifting or swabbing techniques. The experiments were conducted by repeated sampling of FDR contaminated clothing, the samples then being analysed for Pb, Sb and Ba by FAAS. Recovery using adhesive tape was ~20% clothing ~37% hands, swabbing was ~25% clothing ~70% hands, and suction sampling was ~65% clothing, (hands not examined). (Suction sampling is also suitable for recovery of FDR from head hair). As a consequence this laboratory has, since 1979, been suction sampling clothing for FDR using the apparatus in Figure 7.01 attached to an Edwards E2M6 vacuum pump (pumping rate ~ 108 l/min).

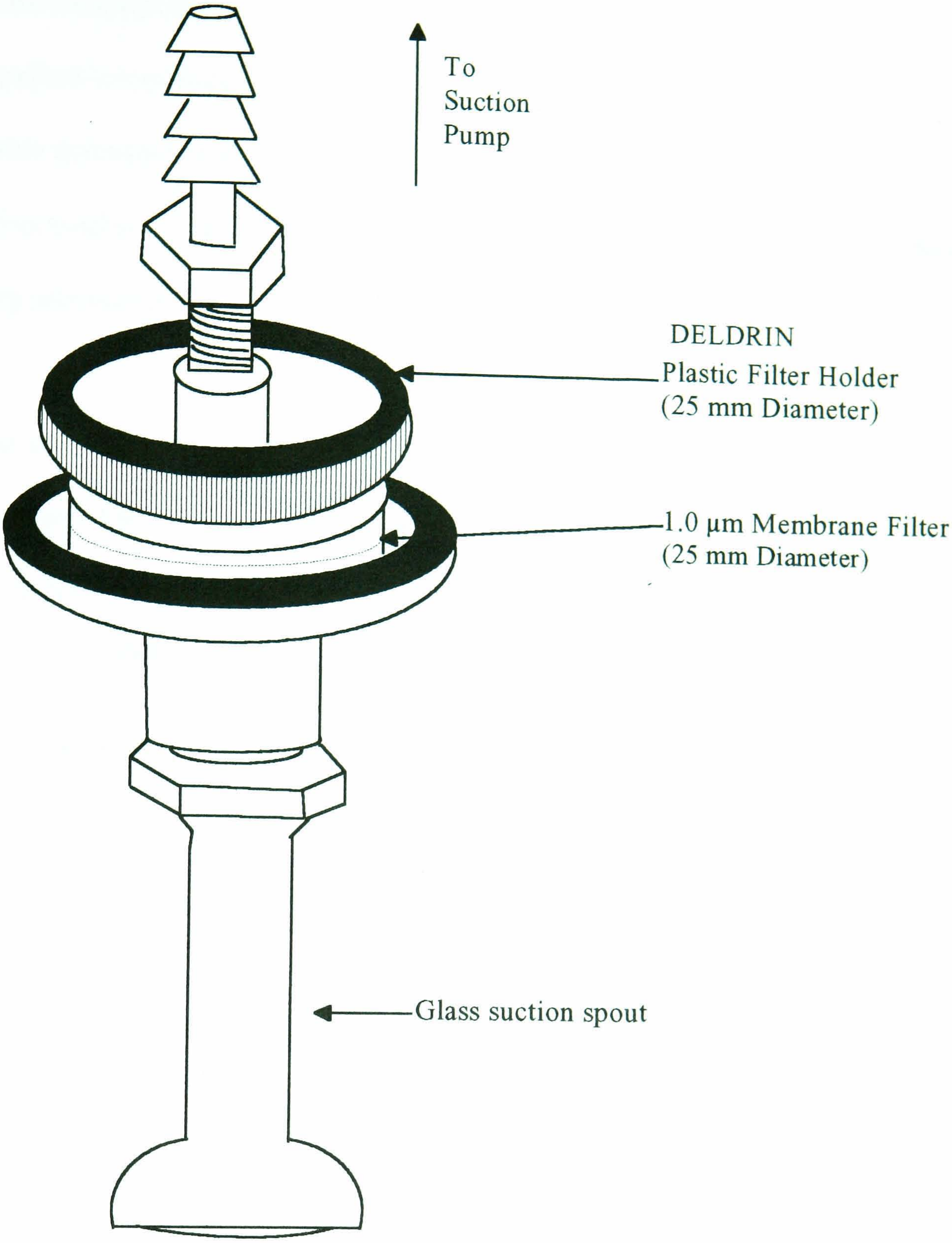


Figure 7.01 Suction sampling device

Previously, when a garment was to be examined for both firearms and explosives residue, certain areas were swabbed with cotton wool damped with acetone to collect explosives residue and other areas were suction sampled for FDR. It was decided to investigate the possibility of using suction sampling for both types of residue and to look at the existing suction system with a view to improving its efficiency. As many of the propellant components are explosive or explosive related compounds, it is a reasonable assumption that experimental results with organic FDR would also apply to explosives residue. A series of experiments were devised to compare and test clothing sampling procedures.

The first step was to directly compare existing procedures, namely, suction sampling and swabbing. Table 7.01 gives comparison data. Control samples taken from the coats prior to firing were negative.

Sample	***NG (ng)***	***2,4-DNT (ng)***
Swab 1	272	ND
Swab 2	110	ND
Swab 3	26	ND
Membrane 1	876	17
Membrane 2	58	ND
Membrane 3	ND	ND

ND = not detected

Table 7.01 Recovery of organic FDR from clothing

The results reveal that NG was detected on all three swab samples and that it was not present on the third suction sample. The initial suction sampling removes a high proportion of the NG whereas the second swabbing shows a substantial level of NG (~40%) when compared to the initial swab sample. Suction sampling detected 2,4, DNT, admittedly at a low level, but swabbing failed to reveal its presence.

It was concluded that suction sampling is more efficient than swabbing and the results suggest that suction sampling may also be suitable for explosive residue sampling. In order to confirm this a further test was conducted involving doping a new laboratory coat with a mixture of explosive compounds and then suction sampling it. The mixture contained a known concentration of RDX, PETN, EGDN, NG, NB, TNT, 2,3 DNT, 2,4 DNT, 2,6 DNT, DPA, EC and MC and was sprayed on to the garment and the solvent allowed to evaporate before sampling. The suction sample was submitted to the explosives laboratory for analysis using a combination of GC/TEA, HPLC/PMDE and GC/MS techniques. The explosives section concluded that suction sampling is suitable for the recovery of explosives residue from clothing (199)

As suction sampling is superior to other sampling techniques, such as adhesive lifting and swabbing, it was decided to try and optimise the existing suction sampling system. To this end the effect of the membrane pore size on the efficiency of recovery of NG was investigated. Results are given in Table 7.02.

Test Number	***Pore Size (Microns)***	***NG (ng)***
1	1.2	8660
	0.8	355
	0.45	17
2	0.8	7400
	0.45	200
3	1.2	9050
	0.8	570
4	1.2	8100
	0.45	315

Table 7.02 Membrane pore size experiment on muzzle residue

Whilst the results were not definitive, it is clear that substantial amounts of NG passed through both the 1.2 micron and 0.8 micron filters, although the initial filter retained the bulk in each test. [There were no membrane filters with a pore size less than 0.45

microns in the laboratory at the time of the test. It would have been interesting to repeat the experiment incorporating smaller pore size filters, such as 0.2 micron.] It is suspected that NG is present as both vapour and particulate matter, the particulate matter having a wide size range

In an attempt to clarify the situation and to validate the system the experiment was repeated on clothing worn whilst firing. Results are given in Tables 7.03 and 7.04. No NG was detected on the control samples.

Test Number	***Pore Size (Microns)***	***NG (ng)***
1	1.2	100
	0.8	ND
	0.45	ND
2	0.8	160
	0.45	5
3	1.2	90
	0.8	3
4	1.2	200
	0.45	20

ND = not detected

Table 7.03 Membrane pore size experiment on clothing (1 shot)

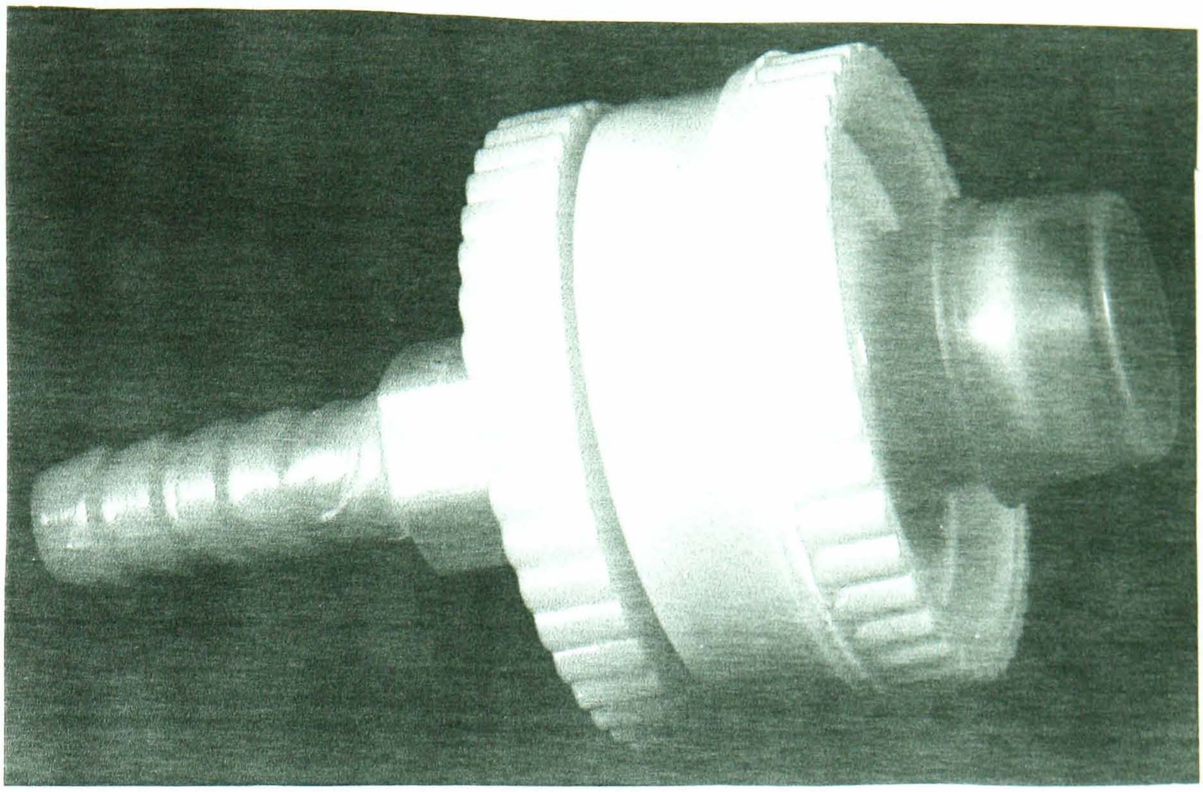
Test Number	***Pore Size (Microns)***	***NG (ng)***
1	1.2	320
	0.8	5
	0.45	ND
2	0.8	480
	0.45	15
3	1.2	270
	0.8	8
4	1.2	335
	0.45	17

ND = not detected

Table 7.04 Membrane pore size experiment on clothing (6 shot)

As the 1.2 micron and the 0.8 micron filters allowed a significant amount of NG through, it was decided to introduce the .45 micron filter coupled with a more powerful pump (Edwards E2 M12 - pumping rate ~ 240 l/min), the increase in pump capacity to enable two suction lines to be run to two sampling areas, thereby improving our sampling facilities. (Later experience proved the 0.45 micron MCE membrane to be affected by the solvents used in the organic FDR procedure and it was changed to a 0.5 micron PTFE membrane, which has since proved to be entirely satisfactory).

The glass disposable spout on the suction sampling apparatus was originally introduced to facilitate the sampling of pocket interiors, but with experience it was decided that the glass spout was not necessary as it restricted the suction area, reduced the efficiency of removal and increased the time taken to sample a garment. Consequently, the revised sampling device is as shown in Figure 7.02.



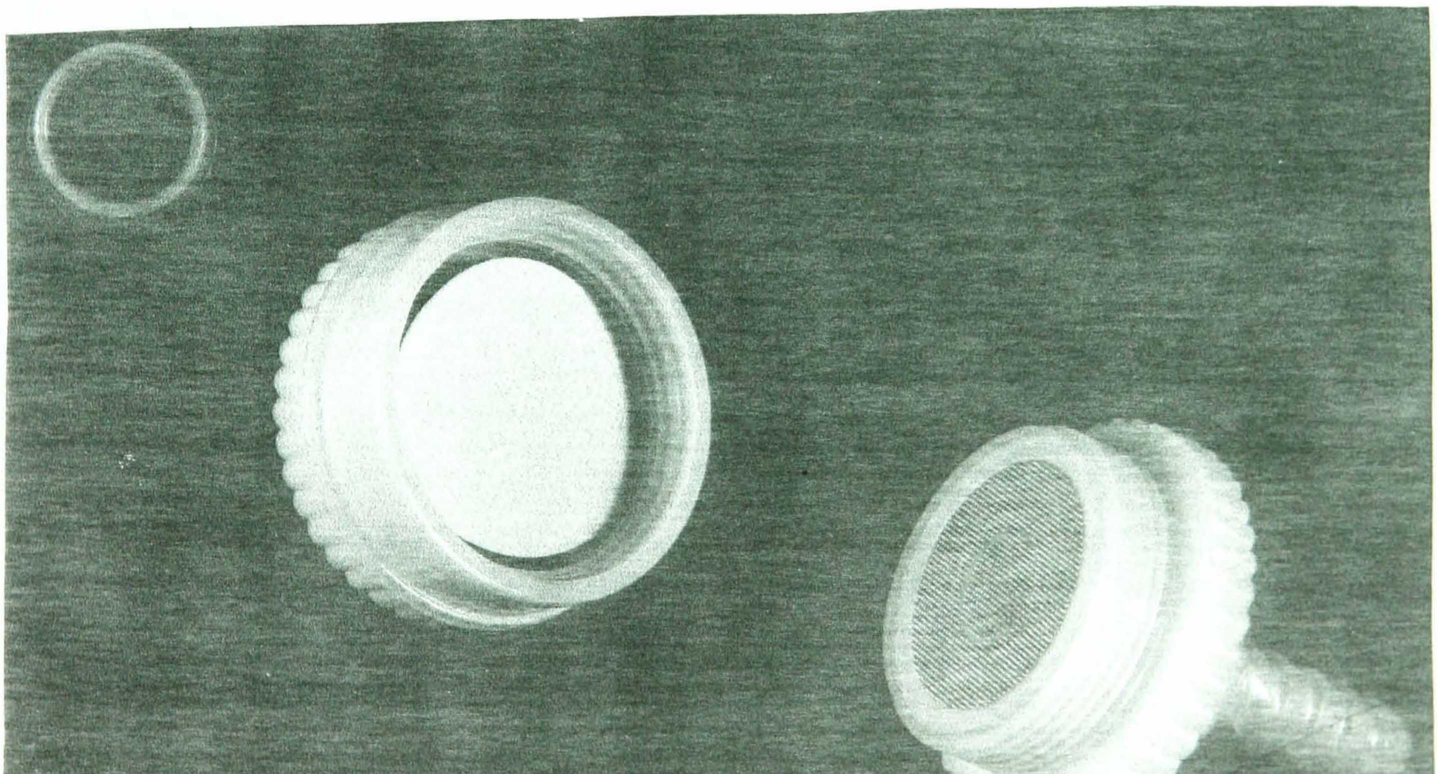


Figure 7.02 Revised suction sampling device

The revised suction sampling device was tested by comparison with the previous device. Results are presented in Table 7.05. No NG was detected on the suction samples taken from the coats prior to use.

Test Number	*NG (ng)*
1	720
2	660
3	670
4	615

Table 7.05 NG recovered from clothing (6 shot)

This represents a good recovery of NG when compared to Table 7.04 and illustrates that the device is effective. However in this instance only one membrane filter was present in the suction line, consequently like is not being compared with like. The improved recovery may be due to a combination of factors.

A final test was conducted, using everyday clothing, to simulate a 'real life' situation. The test was designed to verify the superiority of suction sampling over swabbing and also to check that the entire system, including inorganic particle detection, was satisfactory. The test involved using both sampling techniques on the same FDR contaminated garment and alternating the order of sampling.

Results are given in Table 7.06. No NG or inorganic FDR particles were detected on the control samples.

	Sampling procedure	*NG (ng) Suction*	*NG (ng) Swabbing*	*Inorganic particles (suction sample)*			
				Pb, Sb, Ba	*Sb, Ba*	*Pb, Sb*	*Pb, Ba*
Jacket 1	Swabbed first, then suction sampled	1080	495	8	2	8	7
Pullover 1	Swabbed first, then suction sampled	915	810	13	1	24	2
Jacket 2	Suction sampled first, then swabbed	1555	ND	27	None	16	None
Pullover 2	Suction sampled first, then swabbed	2670	Trace	15	None	7	4

ND = not detected

Table 7.06 Comparison of swab and suction sampling techniques

The results show that suction sampling removes nearly all the detectable NG present whereas swabbing removes considerably less. The swabbing prior to suction sampling probably had a detrimental effect on the number of inorganic particles recovered.

7.3 SAMPLING KITS

The previous method for sampling a suspects skin surfaces for explosive residue was swabbing using cotton wool damped with acetone, and the previous firearms method involved swabbing with acrilan fibre damped with petroleum ether. The two previous general purpose kits for sampling miscellaneous items, used the same techniques and materials, except that dry swabbing was used in the firearms kit. When a suspect had to be examined for both types of residue, swabs from the palm of the hand plus nail scrapings were taken for explosives residue and the back of the hands were swabbed for FDR, as was the face and head hair. This was necessary as the use of one sampling technique excluded the subsequent use of the other on the same area, and caused confusion and considerable inconvenience as two types of kit had to be used to sample one suspect. The firearms kit was incompatible with laboratory preparation procedures for explosives residue, and vice versa. Similar problems were encountered in the sampling of clothing.

The analytical methods currently used by this laboratory are chromatography (GC/TEA: HPLC/PDME) for explosives residues and the particle analysis method (SEM/EDX) for firearms discharge residues. The latter method involves the detection and identification of individual firearms discharge residue particles; any sampling technique must therefore be non-destructive.

At the kit design stage many factors had to be considered, including lifting efficiency, compatibility with existing laboratory techniques, and the avoidance of cross-contamination between sampling areas and possible contamination from other sources, e.g., the sampler, and the sampling room. Ease of use and preparation, the cost, purity and availability of materials, and the shelf life, are all important, as well as the health and safety aspects. Finally, it needs to be acceptable to courts of law and to other scientific examiners. Previous kits suffered a limited shelf life due to evaporation of the solvents. To solve this problem the new kits use a pre - damped acrilan swab in a sealed metal foil sachet. Joint sampling kits were designed, tested and prepared in the laboratory before issue to the police. Details of the revised sampling kits and procedures have been published (200) and the kit is described in Chapter 4.

7.3.1 Clothing examination

As discussed in section 7.2, a single sampling technique for clothing that satisfies the requirements of both types of residue has now been devised. The entire outside front surface (one sample) and pocket interiors (one sample) of a suspect's upper outer garment is routinely sampled for firearms and/or explosives residues by laboratory staff. In addition, other areas and other garments may be sampled depending on the nature and circumstances of the case. Control samples are also taken, one from the outside of the nylon packaging bag containing the garment and a combined control from the sampler, the work surface and the air in the sampling room.

The apparatus currently used is shown in Figure7.02. It consists of a 25 mm diameter Deldrin filter holder (Gelman product No 1109) with one of the nylon hose nipples removed. The filter used is a 25 mm diameter, 0.5 micron pore size Fluoropore membrane filter (Millipore catalogue No FHLP 025 00). An autosampler cup cap is used to seal the apparatus, both before and after use. In use, the filter holder is attached to an Edwards E2 M-12 vacuum pump.

7.3.2 Discussion

At the initial stage the suspect sampling kit was designed to be as self-contained and secure as practical. Everything needed for the procedure, including packaging and labelling materials for submission to the laboratory, is in the kit. This approach lessens the possibility of contamination as all materials involved are under the control of the laboratory, and it also makes the kits more user friendly. Security is enhanced by the issue of the kit in a sealed nylon bag, and by the swab sample container having a tamper proof seal and the requirement for its return in a similar fashion. All swabs are in a sealed metal foil sachet and all swabs, used or unused, must be returned to the laboratory. All kits have a laboratory code number and logo on them and are only issued against a signature. As a consequence, laboratory records show how many kits are issued, when and to whom. Incoming kits are checked against the issue records. Integrity labels, included in the kit, are used initially to seal the nylon packaging bag used for submission of the kit to the laboratory.

At the design stage the assumption was made that both the sampler and sampling room were contaminated. The avoidance of the possibility of contamination is improved by the procedures used. The kit contains three control swabs, one of which is left untouched by the sampler and serves as a control of the kit materials; another serves as a control of the sampler, and the third serves as a control of the sampling environment. Cross-contamination between sampling areas is avoided by the changing of the sampler's gloves between samples. Polythene gloves are used because other types of disposable gloves have a lubricant present which can contaminate the samples and cause problems during a search for firearms discharge residue particles in the scanning electron microscope. The kit is sealed inside a nylon bag which is less permeable than a plastic bag, and a quantity of kits are issued inside a larger sealed nylon bag from where they are removed only as required, after which the bag is resealed. The overall is worn by the sampler to avoid the possibility of any transfer of residue on their clothing to the suspect or work surface. The work surface is cleaned before use, and in addition, a plastic 'tablecloth' is used as a clean work area.

The kit contains a detailed incident report form. The kit instructions are signed and dated by the sampler as a true and accurate record of the process, and are designed to be retained by the sampler as contemporaneous notes for use in court. (Documentation used in the kits is included in Appendix 1). The kit is suitable for both destructive and non-destructive testing, and can also be used for sampling suspects in cases involving the theft of metals.

The revised suspect sampling kit and general purpose sampling kit have now been operational in casework for four years during which time no problems have been encountered, either in the use of the kits or in subsequent laboratory preparation and analysis procedures.

Suspect sampling time is in the region of 30 to 40 minutes, and the removal and packaging of the suspect's outer garments takes a further 5 minutes. The kits are more costly and time consuming to prepare than the equivalent previous kits, but as two new kits replace four previous kits, the overall cost and preparation time is less. None of the components of either kit is re-used. Cost of materials (January 1992) is £8.96 per suspect sampling kit and £4.97 per general purpose sampling kit.

Precautions to prevent cross-contamination, incorporated in the new suspect sampling kit, play a significant role in negating cross-contamination claims arising from the sampling procedure. Quality of the kits is controlled by strict preparation and packaging procedures. It could be necessary to demonstrate in court that all reasonable precautions have been taken during manufacture to avoid contamination occurring, and also that no evidence of contamination was found. To this end, there are clearly defined written procedures to be followed during preparation and packaging stages, including the use of hand washing, disposable overalls, disposable gloves and cleaning processes for certain components. Additionally, swab samples are taken daily from personnel, protective wear and accommodation (tables, benches, chairs) and the completed kits are subjected to random sampling. Analytical results are retained for possible future use. A swab and a plastic tube are included in every kit as a kit (materials) control, and is left untouched by the sampler.

Suction sampling of clothing for firearms discharge residue examination has been used by this laboratory since 1979. As discussed in section 7.2, the suction sampling apparatus described was modified from that previously used to increase its efficiency and to make it compatible with explosives residue examination. Such sampling is efficient and easy to use but suffers from the disadvantage that the Deldrin filter holder is too expensive to be regarded as disposable. Consequently, a rigorous washing procedure is necessary to ensure no carry over of organic or inorganic residues. This does not pose a problem as all traces of firearms and explosives residues can be removed by rinsing under running water, soaking overnight, first, in 2% v/v Decon, secondly in 2% v/v hydrochloric acid, and thirdly in 2% v/v bleach, followed by rinsing in de-ionised water and then in acetone. Apparatus blanks and control samples are routinely taken, whose results testify to the efficiency of the washing procedure.

The overall result of unification of procedures is a modest saving of time and money, but, more importantly, both police and laboratory staff have benefited from the simplification of the system.

7.4 DEVELOPMENT OF A METHOD FOR ORGANIC FDR DETECTION

The requirements of a method for detecting organic FDR were that it would not be detrimental to the existing particle analysis method, that the results would be meaningful and that it should integrate into the two individual existing systems for the detection of explosives and firearms residues, thereby creating a system which could, if desired, routinely analyse all samples for organic explosives and organic and inorganic FDR. Due to the fact that a large proportion of our casework involves the use of ammunition with single base propellants, it was desirable to investigate the possibility of using GC/MS to detect constituents of propellant that are not detectable using our existing explosives residue analysis system. Such constituents include DPA, MC, EC, camphor and phthalates. Because these constituents are originally present in relatively small amounts (typically 0.5 to 2.0%), compared to the level of NG in double base propellants, it would be necessary to optimise all aspects of the system, i.e. the extraction and clean-up/concentration procedures in addition to the instrumental parameters.

As stated previously, the existing techniques used for explosives residue detection of nitro compounds would be suitable for the detection of similar compounds in propellant. Initially the method of Lloyd (159, 160) was employed but it was found that the clean-up/concentration procedure was tedious, lengthy and not robust enough for routine application with a large FDR caseload. Another major disadvantage was the back flushing procedure for the recovery of inorganic particles for SEM/EDX analysis. It was found that after back flushing, a substantial number of inorganic particles remained in the acrodisc which was used for suction sampling clothing. This was determined by cutting open acrodiscs after back flushing and examining their interior by SEM/EDX. As the main consideration in the development of an organic FDR method was that it must not be detrimental to the existing particle analysis method, the back flushing procedure was unacceptable, and it was therefore necessary to develop our own systems (185).

Although there is extensive literature on the identification and detection of propellants, little work has been devoted to the combined analysis of organic and inorganic FDR recovered from hands and clothing in forensic casework. The analysis of organic FDR has concentrated on the detection of NG and 2,4-DNT by HPLC/PMDE. The HPLC-PMDE system requires a clean up and concentration of samples containing organic residues for optimum performance.

The technique developed by Lloyd for the clean-up and concentration of organic FDR and explosive residues was assessed. The technique was found to be laborious, time consuming and unsuitable for the large number of samples processed by this laboratory; consequently, the extraction system has been adapted and optimised. The revised suction sampling apparatus was coupled to an automated robotic system (the Millilab 1A workstation) for the extraction and clean-up/concentration of the organic FDR using a solid phase extraction (SPE) system containing Chromosorb 104 and Amberlite XAD-4. A sensitive gas chromatography - mass spectrometry (GC-MS) method was developed for the analysis of DPA, EC, MC, camphor and phthalates. An existing HPLC-PMDE system was adapted so that the automated deoxygenation and injection of samples for the detection of NG and 2,4-DNT was achieved. GC-MS and HPLC-PMDE analysis were performed on fractions of the same extract. The system has been applied to routine firearms casework for a trial period to assess its evidential value.

Details of the method are given in reference 185 and the following is an assessment of the performance of the method.

7.5 *DEVELOPMENT OF THE SPE SYSTEM*

The Millilab 1A workstation is a personal computer controlled automated robotic system which performs sample extraction from filters and SPE devices according to user defined programmes. This was used to compare the efficiency of different SPE materials, as manual error is substantially reduced. The results are listed in Table 7.07.

Organic FDR	***Recovery (%)***		
	SPE support material		
	Chromosorb-Amberlite	C_{18}	***Aminopropyl***
NG	95	47	5
1,3-DNB	96	36	9
2,4-DNT	96	35	9
DPA	98	42	7
EC	95	39	2
MC	96	32	5

Table 7.07 Average recovery of 10 ng/µl standard containing organic FDR by SPE on the Millilab workstation

Note: The relative standard deviation of the percentage recovery of the organic residues from the Chromosorb 104-Amberlite XAD-4 SPE columns ranged from 3.5% for 1,3-DNB to 5.5% for DPA.

It was demonstrated that the recovery of residues from the Chromosorb 104-Amberlite XAD-4 SPE column prepared in the laboratory was more efficient (greater than 95%) compared to the commercial C_{18} (32-47%) and aminopropyl SPE columns (2-9%). This confirms the work of Lloyd (201) who found that Chromosorb 104 and Amberlite XAD-4 were the most efficient supports for the recovery of organic explosive residues from relatively polar solvents. To reduce the minimum volume required to elute the organic residues from the 1.5-ml SPE columns prepared in the laboratory, 40 mg of support material was used. Using these columns a 1.4-ml extract from the Deldrin unit is cleaned and concentrated to 300 μl.

The Chromosorb 104-Amberlite XAD-4 SPE column prepared in the laboratory allows full automation of the extraction process on the Millilab workstation. Subsequent experiments were performed using these SPE columns.

7.5.1 Efficiency of Millilab extraction

The efficiency of extraction of organic FDR from the Deldrin unit and the subsequent SPE clean-up and concentration using the Millilab workstation were now assessed. The results are listed in Table 7.08. All blanks were negative.

Organic FDR	*Extraction efficiency (%)*
NG	78
1,3-DNB	72
2,4-DNT	74
DPA	57
EC	60
MC	67

Table 7.08 Recovery of organic FDR from Deldrin/SPE units using the Millilab workstation

It was found that the recovery of organic FDR was reduced when the Deldrin filter unit was used (57-78% recovery compared to 95% recovery from the SPE columns). This may be explained by the presence of garment fibres and debris recovered with the FDR. The more material present within the Deldrin unit, the more difficult it is to extract the FDR with a given volume of acetonitrile (total extract 1.4 ml). Using a greater volume of acetonitrile poses problems with the subsequent 1:9 dilution of extracts from SPE. The Millilab workstation is limited to using 160 mm x 10 mm tubes for dilution (a total workable volume of 14 ml).

When examining "dirty" garments a number of Deldrin units may be required to cover the entire surface as a result of the fluoropore filter becoming clogged with material, hence reducing the suction efficiency. An attempt to use a 20 µm prefilter to prevent clogging was abandoned because this resulted in reduced recovery of inorganic FDR.

7.5.2 Efficiency of swab extraction

Swabs were examined to determine the efficiency of recovery of organic FDR, both without and with the SPE clean - up step. Results are presented in Tables 7.09 and 7.10 respectively which represent average recoveries from four tests.

Organic FDR	***Recovery %***
NG	94
1,3-DNB	97
2,4-DNT	100
DPA	98
EC	88
MC	101

Table 7.09 Average recovery of 10 ng/μl standard from a swab (filter)

Organic FDR	***Recovery %***
NG	88
1,3-DNB	82
2,4-DNT	80
DPA	93
EC	81
MC	94

Table 7.10 Average recovery of 10 ng/μl standard from a swab (filter and SPE)

This represents a very satisfactory recovery of organic FDR from swabs. The lower recovery from clothing is thought to be due to debris from the clothing inhibiting the extraction of organics. The larger volume of the Deldrin unit compared to the swab extraction apparatus, i.e. different volume to solvent ratios, may be another factor. It must also be borne in mind that pure compounds may not necessarily reflect the behaviour of actual organic FDR.

7.5.3 *Recovery and analysis of FDR from clothing*

The efficiency of the technique to recover and detect FDR from different types of clothing worn during the firing of six rounds of ammunition from a revolver was assessed. Results are given in Table 7.11. No organic or inorganic FDR was detected in the control samples.

Garment	*Organic FDR (ng)*				*Inorganic FDR (No. of particles)*			
	NG	*2,4-DNT*	*DPA*	*EC*	*Pb, Sb, Ba*	*Sb, Ba*	*Pb, Sb*	*Pb, Ba*
Laboratory coat	976	39	4.6	1	3	2	71	3
Sweatshirt	1,273	39	7.4	2.2	30	5	175	13
Woollen jumper	730	10	1.7	0.5	1	-	34	6

Table 7.11 Analysis of FDR recovered from clothing (six shots)

FDR were recovered from all garments, with the sweatshirt and the laboratory coat giving better recovery than the woollen jumper. In all cases organic and inorganic FDR were easily identified. It was assumed at the start of the experiment that the woollen jumper would have the best retention of FDR but this was not reflected in the results. It is suggested that a reason for this could be the suction sampling procedure which works best on flat/tight weave garments, such as the laboratory coat and the sweatshirt.

The suction sampling and analysis techniques were repeated to determine if organic and inorganic FDR could be detected on clothing worn during the firing of one round of ammunition from a revolver. The results are listed in Table 7.12.

	Organic FDR (ng)			***Inorganic FDR (No. of particles)***			
	NG	***DPA***	***EC***	***Pb, Sb, Ba***	***Sb, Ba***	***Pb, Sb***	***Pb, Ba***
Laboratory coat 1	775	3.5	2.6	2	1	1	-
Laboratory coat 2	910	8.7	4.3	3	-	7	14

Table 7.12 Analysis of FDR recovered from clothing (one shot)

Detectable quantities of organic and inorganic residues were recovered from the laboratory coats. The amounts of NG, DPA and EC detected were well above the detection limits of the systems. A smaller number of inorganic FDR particles were recovered from the laboratory coats compared to the garments doped with six shots.

7.5.4 Survey of clothing submitted to the laboratory for FDR examination

For a trial period of three months, clothing submitted to the laboratory for inorganic FDR analysis was also examined for organic FDR. Organic residues detected during the trial period were not used as evidence in criminal proceedings. Thirteen different firearm related incidents (cases F1-F13) with a total of 186 exhibits were examined. One case F1 accounted for 100 exhibits. The positive results are listed in Table 7.13.

Case	*Clothing*	*Organic FDR (ng)*		*Inorganic FDR (No. of particles)*			
		NG	*2,4-DNT*	*Pb, Sb, Ba*	*Sb, Ba*	*Pb, Sb*	*Pb, Ba*
F1	(a) Upper front body/cuffs	124	93	-	-	-	-
F1	(b) Pockets	300	4	-	-	1	-
F3	(a) General outer/body	124	-	-	-	-	-
F3	(b) General outer/body	2,068	4	-	-	-	-
F8	(a) Front pocket	1,685	-	-	-	1	-
F13	(a) Mask	-	-	1	1	1	1

Table 7.13 Survey of clothing submitted to the laboratory for FDR analysis

Only one exhibit, mask (a) in case F13, was positive for inorganic FDR, although no organic FDR was detected on this item. The indicative inorganic particles Pb, Ba was detected in 17 exhibits from four cases but, in the absence of any unique inorganic particles, they were reported as negative.

Unfortunately it was not possible to analyse for the presence of DPA, EC, MC, camphor and phthalates in the extracts from the 186 items as they were destroyed in a terrorist explosion at the laboratory prior to GC/MS analysis.

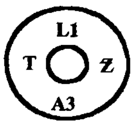
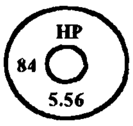
On the basis of this work there is poor correlation between the detection of inorganic and organic FDR in casework, and organic detection appears to be more sensitive than the detection of inorganics. However, one of the cases examined (F1) was atypical and consisted of 100 exhibits; consequently there is insufficient data to draw firm conclusions about the value of organic FDR detection. Nevertheless the results are very encouraging.

7.6 CURRENT METHOD FOR ORGANIC FDR DETECTION

Whilst automation of the extraction and SPE stages has advantages it was unsuitable for many of our cases as it was very time consuming (40 minutes per sample), requiring overnight running, and the Millilab could only accommodate twelve Deldrins per run. This is a disadvantage whenever there is an urgent case to be done, perhaps in the middle of the night, with the police wanting a verbal report prior to interviewing suspects. It was decided not to opt for an automatic process, but to do the sample manipulations manually.

It was also decided to use GC/TEA in preference to HPLC/PMDE for several reasons. GC/TEA is more specific for nitro groups, more suited to automation, faster, safer and simpler to use and does not require a sample clean up/concentration step, thereby providing better quantification as there is less sample manipulation. As a consequence a modified analytical scheme was devised the details of which are given at the end of this chapter.

To evaluate the apparent greater sensitivity of organic FDR relative to inorganic FDR it was decided to conduct outdoor firing tests. For this purpose it was necessary to identify suitable ammunition as containing either single or double based propellant. Propellant from new boxed ammunition was analysed. Quantitative rather than qualitative analysis was done as the information, apart from being of possible interest to the current tests, could be of use for future tests. The results are presented in Table 7.14.

Sample	***Calibre***	***NG %***	***DNT%***	***DPA%***	***EC%***	***A nitro DPA***	***Other***
	7.62 x 39 mm	-	-	0.8	-	Present	Camphor 1.1%
	7.62 x 39 mm	-	-	0.7	3.3	Present	
	.223"	11.1	-	0.96	-	-	Large phthalate and glycol peak
	.223"	9.4	0.1	0.43	-	-	Large phthalate peak
	.223"	9.6	-	0.62	-	Present	Large phthalate peak
	.223"	6.8	-	0.01	1.3	-	MC 4.3%
	.223"	5.8	-	0.6	-	Present	Large phthalate peak
	.223"	4.92	0.24	0.5	0.08	Present	Large phthalate peak
	.223"	-	-	0.45	5.7	Present	
	.357" Magnum	-	-	0.46	0.42	Present	Large phthalate
	.357" Magnum	-	-	0.83	-	Present	
	357" Magnum	6.33	-	0.73	-	Present	Large phthalate peak
	.357" Magnum	6.5	-	0.79	-	-	Large phthalate peak
	.357" Magnum	-	0.01	0.64	2.32	Present	
	12 Bore	9.4	0.08	0.32	-	-	Large phthalate peak
	12 Bore	-	-	0.54	0.4	-	Large phthalate peak

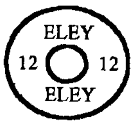
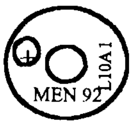
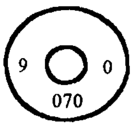
Sample	Calibre	NG %	DNT%	DPA%	EC%	A nitro DPA	Other
	12 Bore	-	0.11	0.52	-	Present	
	12 Bore	-	0.06	1.27	-	-	MC 0.021%
	12 Bore	-	-	1.0	1.43	-	
	12 Bore	-	-	0.85	-	Present	
	12 Bore	-	1.91	0.47	-	Present	
	12 Bore	-	0.02	0.53	-	Present	
	9 mmP	18.1	0.1	0.29	-	-	Large phthalate peak
	9 mmP	14.2	-	-	-	-	Large phthalate peak
	9 mmP	12.5	0.03	0.33	0.03	Trace	Small phthalate peak
	9 mmP	-	-	0.81	-	Present	
	9 mmP	15.7	-	0.17	0.03	-	
	9 mmP	15.0	-	0.24	-	-	Small phthalate peak
	9 mmP	10.4	0.05	0.26	-	-	Phthalate peak
	9 mmP	12.5	0.03	0.3	-	-	
	9 mmP	-	-	1.08	0.05	-	

Table 7.14 Quantitative analysis of propellant

Two outdoor firing tests were conducted and the firers were examined for the presence of organic FDR. Results of the organic analysis of the first series of tests are presented in Table 7.15 and those of the second series in Table 7.16.

Firearm	***Sample***	***GC/TEA***	***GC/MS***
SMG	Swab kits 6 shot single based	-	-
Shotgun		-	-
Revolver		-	-
Pistol		-	-
Rifle		NG 14 ng L.Hand	-
SMG	Coats 6 shot single based	NG 320 ng	DPA 144 ng
Shotgun		-	-
Revolver		-	EC 51 ng
Pistol		-	-
Rifle		-	-
SMG	Swab kits 6 shot double based	-	-
Shotgun		-	-
Revolver		-	-
Pistol		NG 48 ng L.Hand	-
Rifle		-	-
SMG	Coats 6 shot double based	NG 500 ng	DPA 120 ng
Shotgun		NG 300 ng	-
Revolver		-	-
Pistol		NG 240 ng	-
Rifle		NG 300 ng	DPA 66 ng

(-) Nothing detected

Table7.15 Outdoor firings with single and double based propellants

Firearm	***Sample***	***GC/TEA***	***GC/MS***
	Swab Kits		
SMG	2, 6, 10 shot	-	-
Shotgun	2, 6, 10 shot	-	-
Revolver	2, 6, 10 shot	-	-
Pistol	2 shot, R.Hand	NG 29 ng	-
Pistol	6, 10 shot	-	-
Rifle	2, 10 shot	-	-
Rifle	6 shot, R.Hand	NG 200 ng	EC 3 ng
	Coats		
SMG	2, 6 shot	-	-
SMG	10 shot	NG 680 ng	DPA 150 ng
Shotgun	2, 6, 10 shot	-	-
Revolver	2, 6, 10 shot	-	-
Pistol	2 shot	-	DPA 36 ng
Pistol	6, 10 shot	-	-
Rifle	2, 10 shot	-	-
Rifle	6 shot	-	EC 33 ng

(-) Nothing detected

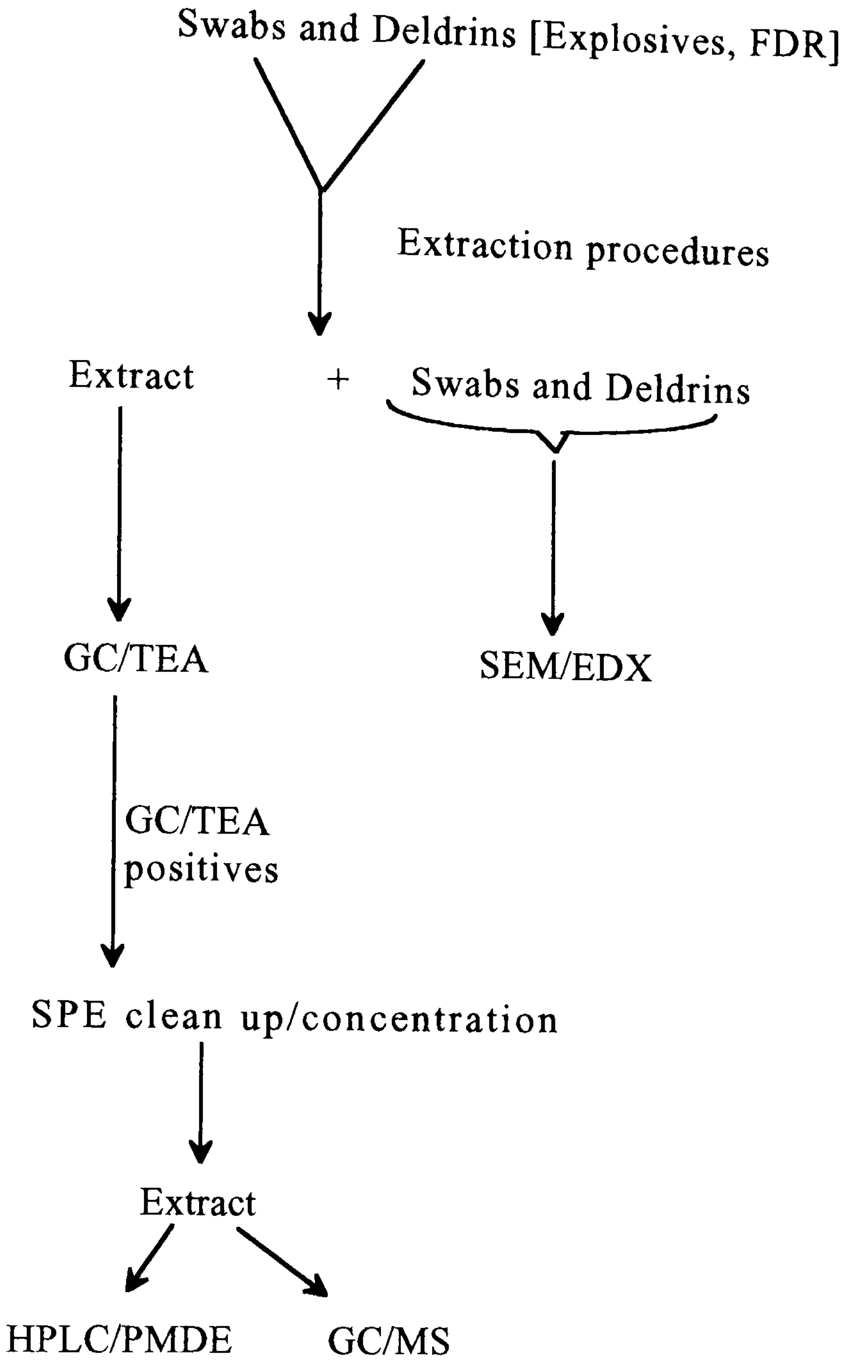
Table7.16 Outdoor firings with single based propellants

Elaborate precautions were taken to prevent cross contamination, yet despite this NG was detected on some of the samples originating from the discharge of single based propellants. Obviously contamination did occur, the source of which is unknown, but it is more likely to have occurred as a consequence of being in a firing range rather than from a flaw in the procedures. These tests were conducted outdoors with a strong wind blowing, and in the first series it was also raining. The conditions were not ideal for a heavy deposition of FDR. Hands and face were sampled immediately after firing and then the laboratory coat was removed and packaged i.e. sampling of the firer was prompt.

Under these firing conditions, unique and characteristic inorganic FDR particles were readily detected on all of the samples, both swab and suction. This result conflicts with the previous work which suggested that organics are more readily detected than inorganics (see Tables 7.11, 7.12 and 7.13). There does not appear to be good correlation between inorganic and organic FDR detection and this may be due to differing retention properties of both types of residue.

It is noteworthy that throughout the course of this work, organic residue originating from the discharge of single based propellants has rarely been detected. [EC was detected in FDR from single based propellants in LAPUA .223" and .357" Magnum calibres (Tables 7.15 and 7.16); however, EC occurs at a relatively high concentration (5.7% and 2.32% respectively) in both these propellants.] It was concluded that, due to the low concentration of detectable constituents in single based propellants, it was not possible to reliably detect them in discharge residue deposited on the skin and clothing surfaces of a firer using current instrumentation and techniques. There appears to be good correlation between the detection of NG and other organic constituents of propellant, such as DPA and EC.

On the basis of this work the following system has been adopted:-



Organic FDR detection is a useful additional technique, despite the fact that single base propellant constituents cannot be reliably detected. The current system uses GC/TEA as a rapid screening technique for NG and 2,4-DNT, and only positive samples need to be subjected to the SPE clean up/concentration procedure. The method is flexible in that the extract can be analysed by a range of analytical instrumentation. There does not appear to be good correlation between the detection of organic and inorganic FDR, which emphasises the need for combined instrumentation i.e. GC/TEA, GC/MS and SEM/EDX. Details of the method devised are given in Appendix 3.

8 SUMMARY, COMMENTS AND CONCLUSIONS

8.1 OVERVIEW

Apart from giving the background to the project, the three introductory chapters also serve as useful background reading for anyone undertaking the chemical aspects of firearms casework examination. To the best of my knowledge no other single source of such information is available. Casework statistics and tests strongly support the literature review in Chapter 2, except for a few minor areas which are mentioned in the text.

The experimental work attempted to improve all stages in the detection and identification of FDR. The main aspects are as follows:-

Scene examination	1. Improved and simplified sampling kit
	2. Design of scene contamination avoidance kit
Arrest and processing of suspects	3. Speeding up of the suspect sampling procedure
	4. Use of a protective cape to prevent loss of forensic evidence and improve contamination avoidance
	5. .Improved and simplified sampling kit
	6. Improved contamination avoidance in sampling locations
Laboratory sample preparation	7. Improved sampling of clothing
	8. Improved contamination detection and avoidance
	9. Amalgamation of explosives residues and FDR systems
Laboratory analysis methods	10. Development of a method for the detection of organic FDR
	11. Development of a system that has much greater flexibility and readily facilitates the routine screening of all suspects for both explosives and inorganic/organic FDR

Interpretation	12. Extended knowledge about the nature, properties and behaviour of inorganic FDR and, to a lesser extent, organic FDR
	13. Updated and more technically correct particle classification scheme
	14. Improved overall knowledge regarding the chemistry of firearms
Court Evidence	15. The production of statistics from casework on contamination risk
	16. Regular monitoring of sampling locations, samplers premises and kit making premises

The failure to reliably detect organic constituents from single based propellants is disappointing, but at least the situation is clarified and the recovery of detectable constituents from double based propellant has been improved. The detection of organic FDR from double based propellants is a useful addition to the overall capability in this area and has the potential to substantially increase the significance of particles in the 'indicative' category of the particle classification scheme. The ability to routinely screen all terrorist suspects, both swabs and clothing, for explosives residue and organic and inorganic FDR is a major addition to our capabilities, but is obviously only applicable in countries with a terrorist problem. However, the means of incorporating organic FDR into the inorganic FDR system will be of interest to all involved in the detection of FDR.

The correlation between the detection of organic and inorganic FDR appears to be bad, based on the results to date. It would be desirable to test this in casework over a period of several years for clarification in a 'real world' situation. Based on results to date, the Particle Analysis method remains the preferred method for the detection of FDR and is enhanced by the detection of organic FDR.

Whilst the detection of organic FDR is a useful addition, it does lengthen the time taken to analyse a sample and involves additional sophisticated instrumentation, which is expensive to operate and maintain and which requires specialised staff. The objective of a simple, fast, conclusive and reliable method for the detection of FDR has yet to be achieved, indeed the more instrumentation involved the greater the chances of instrumental breakdown somewhere in the system. The examination of a suspect for FDR is a costly process, both in terms of money and time, and it is essential that there is close liaison between the police investigating officer and the forensic scientist, to determine the value of doing an examination based on the facts in each case.

Further work needs to be done to investigate occupational/environmental particles containing Sr, Ti, Zn, either individually or in combination, in anticipation of the use of Sintox primers in crime. Another area worthy of attention is the detailed examination of spent bullets and bullet hole perimeters for the presence of both organic and inorganic residue as this may provide useful information about the ammunition involved in the crime.

Appendix 1 - Documentation in sampling kits

SWABBING INSTRUCTIONS FOR EXPLOSIVE/AND OR FIREARMS RESIDUES.

CONTENTS: Contents checklist inside the kit box. (see later)

SAFETY:

1) The solvent used is HIGHLY FLAMMABLE and therefore swabbing should not be carried out near any source of ignition.

2) The test subject should not be allowed to smoke for at least 10 minutes after the swabs have been taken as volatile residues may still be on their hands, face or hair. A total smoking ban should be imposed in the room when a subject is being swabbed.

3) When swabbing the test subjects face keep the swab and solvent away from their eyes.

USING THE KIT:

1) Before use check that the seal is intact on the securitainor. If not, use another kit and return faulty kit to the laboratory.

2) Use pen in kit to mark of instruction and contents checklists at the appropriate times.

3) A waste bin should be placed convenient to the sampler. Discard surplus material as you proceed.

4) When swabbing an area the swab should be FIRMLY rubbed over the area NUMEROUS times.

5) When swabbing a hand the following areas should be covered, the palm, fingers, thumb, back of the hand and the wrist, i.e. the complete hand.

6) Sample the test subject as soon as possible after apprehension otherwise valuable evidence will be lost.

7) The person taking the swabs, and the swabbing room, should be free from explosive and firearms discharge residues.

8) Use one kit for each test subject and use different officers to sample different test subjects.

9) This kit may also be used to swab test subjects suspected of involvement in the theft of metals.

NOTE: Spares included in kit can be used to replace items accidentally dropped onto the floor OR, exceptionally and with very good reason, to sample in an area not covered by the instructions. All swabs, used or unused, MUST be returned to the laboratory. All swabs MUST be accounted for.

PREPARATION:

Tick boxes as you proceed

1) Using the x-ray medi wipe accompanying these instructions, thoroughly clean the work surface on which the kit is to be placed and then discard the medi wipe. ☐
2) Open the second section of the nylon bag, remove the self seal bag labelled GLOVES and put gloves on. ☐
3) Remove the disposable overalls, and put them on. ☐
4) Open the third section containing the kit, remove the kit box and x-ray medi wipe from the bag and place on the work surface. ☐
5) Remove your gloves and discard them. ☐
6) CLEAN YOUR HANDS THOROUGHLY, USING THE X-RAY MEDI WIPE PROVIDED. ☐
7) Open the kit, DISCARD THE LID, remove the self seal bag labelled gloves, and place TWO gloves on EACH of your hands. ☐
8) Remove the plastic sheet from the kit and spread on the examination table. DO NOT PLACE KIT CONTAINER ON THE SHEET ☐
9) Assemble the components from the kit on the sheet and check them against the contents list. ☐
10) Remove and discard your outer gloves only and put on a new pair. ☐

PROCEDURE:

CONTROL SWABS:

This kit contains 3 control swabs. One is used to take a control swab from the samplers gloved hands. The second control swab is used to swab the work surface on which the kit is placed.

The third control is LEFT UNTOUCHED.

1) Remove the sachet from the bag labelled CONTROL - SAMPLERS HANDS, open the sachet by tearing at nick in foil and remove the pre-wetted swab. Shake the swab to remove excess solvent. ☐
2) Using this swab, SWAB THE BACK AND PALM OF BOTH GLOVED HANDS ☐
3) Place the swab in the small plastic tube and seal the tube by fitting the lid. Replace the tube in the original labelled bag. ☐
4) Remove the sachet from the bag labelled CONTROL-SAMPLE AREA, open the sachet and remove the pre-wetted swab. Shake the swab to remove the excess solvent. ☐

5) Using this swab, SWAB THE WORK SURFACE ON WHICH THE KIT IS PLACED ☐

6) Repeat (3). ☐

7) LEAVE THE OTHER CONTROL SWAB UNTOUCHED. ☐

8) DISCARD YOUR OUTER PAIR OF GLOVES AND PUT ON A NEW PAIR. ☐

HAND SWABBING:

1) Whilst holding the test subjects right wrist; visually examine the right hand for particulate matter. ☐
If anything of significance is found scrape it into the appropriate nail scraping vial, then reseal the vial, and continue with the hand swabbing procedure.

DO NOT TAKE NAIL SCRAPING SAMPLES AT THIS STAGE

2) Remove the sachet from the bag labelled RIGHT HAND, open the sachet and remove the pre wetted swab. Shake the swab to remove the excess solvent. ☐

3) Swab the test subjects right hand as thoroughly and as rapidly as possible, covering the entire area including the wrist. ☐

4) Place the swab in the plastic tube and seal it by fitting the lid. Replace the tube in the original labelled bag. ☐

5) DISCARD OUTER GLOVES AND PUT ON A NEW PAIR ☐

6) Repeat 1 to 4 for the left hand using the swab labelled LEFT HAND. ☐

7) DO NOT DISCARD THE GLOVES ON COMPLETION OF THE LEFT HAND SWABBING, THEY MUST BE WORN DURING THE NAIL SCRAPING PROCEDURE. ☐

NAIL SCRAPINGS:

1) If the test subjects finger nails are dirty remove the dirt from one hand with the stick provided and place the dirt in the vial. Replace the stick in the vial and replace the vial in the pre labelled self seal bag. ☐

2) Repeat for other hand ☐

3) DISCARD OUTER GLOVES ☐

FACE SWAB:

1) PUT ON NEW OUTER GLOVES ☐

2) Using the swab from the container labelled FACE SWAB, swab the test subjects face, using the same technique as for taking hand swabs. Swab the cheeks first, then work your way round the chin, finishing with the subjects forehead. ☐

3) Place swab in plastic tube and seal. Replace tube in the original labelled self seal poly bag. ☐

4) DISCARD OUTER GLOVES. ☐

HAIR SWABS:

1) PUT ON NEW OUTER GLOVES ☐

2) WITHOUT DISCARDING THE OUTER GLOVES BETWEEN EACH SWAB take 4 swabs from the test subjects hair, using the swabs labelled HAIR SWABS. ☐

3) Swab from the crown of the head towards the front, covering the total area with each swab. <u>NB</u> Rub swab vigorously into the head. Place each swab in an individual tube and seal. Place the tubes in the labelled self seal poly bag. ☐

4) DISCARD OUTER AND INNER GLOVES. ☐

<u>PACKAGING</u>:

1) Write the test subjects name on all the self seal poly bags. ☐

2) Pack all samples and spares into the plastic container and seal with the NEW, UNUSED lid. ☐

3) Write test subjects name, and the date on the outside of the kit container. ☐

4) Place kit inside unused nylon bag supplied and seal the bag with labels supplied. Sign labels and seal bag with sellotape. Sellotape 38/30 label to outside of the bag. ☐

5) Discard all surplus materials. ☐

6) Complete information sheet and submit it, with the kit to the Laboratory <u>AS QUICKLY AS POSSIBLE</u>. ☐

7) Complete the following section and retain instructions for your records. ☐

Test Subjects name:

Time of commencement:

Date, and exact location of sampling:

Time of completion:

Nature of incident:

Were samples taken for firearms or explosive residues or both?

SAMPLE TAKEN (√ or X)

Control samplers hands	***Control sampling area***	***Right hand (swab)***	***Left hand (swab)***	***Right hand (nail)***	***Left hand (nail)***	***Face (swab)***	***Head hair (swabs)***	***Other***

If "other" swab taken specify and record reason:

Record reason for <u>not</u> taking samples:

I certify that all samples taken from ______________________________ were in strict accordance with the instructions.

Signed _____________________ Date ____________

SAMPLING FOR EXPLOSIVES/FIREARMS RESIDUE

REPORT OF INCIDENT

Note: 1. Please inform the laboratory IMMEDIATELY should examination prove to be unnecessary.

2. ALL questions MUST be answered. If you cannot answer, write N/K (Not Known) or N/A (Not Applicable).

NIFSL CASE NO: ITEM NO:

QUESTION	ANSWER
Test subject's name.	
Test subject's occupation ON DAY OF INCIDENT.	
When did test subject last wash their:- a. Hands? b. Face? c. Hair?	
Is test subject (a) right handed, (b) left handed or (c) ambidextrous?	
When was the test subject's upper outer clothing last cleaned?	
Weather conditions at time of incident i.e.: wet, dry, damp, windy, snow etc.	
Appearance of test subject's:- a. Hands? b. Face? c. Hair? (clean, dirty, oily, greasy, bloodstained etc.)	

PTO

READ THESE INSTRUCTIONS CAREFULLY BEFORE PROCEEDING

QUESTION	**ANSWER**
Amount of head hair on test subject.	
Amount of facial hair on test subject. (beard, stubble, moustache, side burns.)	
Date and time of:- a. Incident? b. Apprehension? c. Sampling?	
What laboratory examination is required? Please tick ONE ONLY	EXPLOSIVES FIREARMS BOTH
Nature of explosive suspected?	
Was the test subject protected prior to sampling using OFFICIAL ISSUE protection kit.	YES/NO
Was the test subject protected by other means. If so, WHAT? (Include unused sample)	YES/NO
Was protection put on the subject immediately on apprehension? If not, WHEN?	YES/NO
Was test subject injured by gunshot? If so, WHERE?	

PTO

QUESTION	ANSWER
Was test subject (a) indoors or (b) outdoors when shot? Did injury appear to result from a close range shooting.	YES/NO
Is there any evidence of bullet fragmentation	
a. to test subject's body?	YES/NO
b. to test subject's clothing?	YES/NO
c. in area of shooting incident?	YES/NO
Number of shots thought to have been fired by test subject?	
Type of firearm thought to have been used by test subject?	
Total number of shots fired during the incident?	
Is it possible that the test subject through a hobby or occupation could legally have discharged a firearm prior to the shooting incident?	
If YES give details under comments below	YES/NO

Person required to be contacted if further information is required:

PLEASE PRINT NAME ....

STATION ....

TEL NO ....

EXTN ....

PTO

QUESTION	**ANSWER**

COMMENTS:

DETAILS OF INCIDENT:

Signed: ______________________________

Dated: ______________________________

EXPLOSIVE/FIREARMS RESIDUE KIT CONTENTS CHECKLIST

TICK BOXES AS YOU PROCEED

2 x pair of gloves in self sealed poly bag } ALREADY USED ☐
1 x 24" square plastic sheet } ☐

1 x self sealed poly bag labelled "GLOVES" containing 5 x pairs of plastic gloves. ☐

1 x self sealed poly bag labelled "CONTROL SAMPLERS HANDS" containing 1 x pre wetted swab in a foil sachet, 1 x plastic tube and lid. ☐

1 x self sealed poly bag labelled "CONTROL SAMPLING AREA" containing 1 x pre wetted swab in a foil sachet, 1 x plastic tube and lid. ☐

1 x self sealed poly bag labelled "CONTROL DO NOT OPEN" containing 1 x pre wetted swab in a foil sachet, 1 x plastic tube and lid. ☐

1 x self sealed poly bag labelled "RIGHT HAND SWAB" containing 1 x pre wetted swab in a foil sachet, 1 x plastic tube and lid. ☐

1 x self sealed poly bag labelled "LEFT HAND SWAB" containing 1 x pre wetted swab in a foil sachet, 1 x plastic tube and lid. ☐

1 x self sealed poly bag labelled "NAIL SCRAPING RIGHT HAND" containing 1 x nail scraper, 1 x squat vial. ☐

1 x self sealed poly bag labelled "NAIL SCRAPING LEFT HAND" containing 1 x nail scraper, 1 x squat vial. ☐

1 x self sealed poly bag labelled "FACE SWAB" containing 1 x pre wetted swab in a foil sachet, 1 x plastic tube and lid. ☐

1 x self sealed poly bag labelled "HAIR SWAB" containing 4 x pre wetted swabs in foil sachets, 4 x plastic tubes and lids. ☐

1 x self sealed poly bag labelled "SPARES" containing gloves, swabs, plastic tubes/lids, and self seal poly bags. ☐

GENERAL PURPOSE SWABBING KIT FOR FIREARMS AND/OR EXPLOSIVES RESIDUE

RETAIN THESE INSTRUCTIONS FOR YOUR RECORDS

NOTE: THIS KIT IS NOT TO BE USED FOR SWABBING PEOPLE

CONTENTS

Upper Section
1) Instructions
2) X-ray medi wipe
3) Gloves

Lower Section
1) Sealed kit containing:
   i. Gloves
   ii. One plastic sheet
   iii. Seven self-seal bags each containing a pre-wetted swab in a foil sachet and a plastic tube and lid.
   iv. One bag as above labelled "Control Do Not Open".
2) X-ray medi wipe
3) Spare lid for kit container
4) 2 x Integrity labels and a 38/30 label
5) Nylon bag
6) Ballpoint pen
7) Information sheet

SAFETY The solvent used is HIGHLY FLAMMABLE and therefore swabbing should not be carried out near any source of ignition.

USES:

This is a general purpose swabbing kit to be used for the recovery of:

A) FIREARMS DISCHARGE RESIDUES EXCLUSIVELY.

B) FIREARMS DISCHARGE AND/OR EXPLOSIVE RESIDUES.

C) EXPLOSIVE RESIDUES EXCLUSIVELY.

THE FOLLOWING INSTRUCTIONS OUTLINE THE USE OF THIS KIT IN EACH OF THE ABOVE CATEGORIES

A) RECOVERY OF FIREARM DISCHARGE RESIDUES EXCLUSIVELY

i) STRIKE MARKS ETC.;

For suspected bullet holes, firing points, strike marks etc., CONTROLS from the relevant surfaces are ESSENTIAL to indicate the background level of elements of interest. These controls should be taken from the same surface as the damaged area but as far as possible away from the hole or mark to avoid lifting any residue from the damaged area. CONTROLS are ESSENTIAL for interpretation of analytical results.

ii) DISCHARGE OF A FIREARM INSIDE A VEHICLE;

To determine if firearms discharge residues are present swab the following areas:

Roof lining - one swab.
Top of dashboard - one swab.
Rear parcel shelf - one swab.
Other areas you consider relevant - one swab.

DO NOT TAKE MORE THAN FOUR SWABS FROM THE CAR INTERIOR.

B) RECOVERY OF FIREARMS DISCHARGE AND/OR EXPLOSIVE RESIDUES

i) TRANSPORTATION OF FIREARMS AND/OR EXPLOSIVES INSIDE A VEHICLE:

To determine if firearm discharge and/or explosive residues are present swab the following areas:

Front passenger seat - one swab.
" " floor - one swab.
Rear seat - one swab.
" floor - one swab.
Boot area - one swab.
Other areas you consider relevant - one swab.

ii) PROPERTY

Use this kit to swab property and hides where it is thought firearms and/or explosives may have been stored or used.

C) RECOVERY OF EXPLOSIVE RESIDUES EXCLUSIVELY

SEATS OF EXPLOSIONS:

To determine the type of explosive used in an explosion the seat of the explosion and suitable surrounding areas may be swabbed using this kit.

USE <u>ONE KIT</u> FOR EACH OF THE ABOVE CATEGORIES OR AREA WITHIN A CATEGORY

PROCEDURE

Before use check the seal is intact on the kit container.
If not, use another kit and return the faulty kit to the laboratory.

1) If a suitable surface is available, use the x-ray wipe accompanying these instructions to thoroughly clean the surface on which the kit is to be placed and then discard the wipe.

2) Remove self seal bag labelled "gloves" and put gloves on.

3) Open the lower section containing the kit, remove the kit container and X-ray wipe from the bag, and place on the cleaned work surface.

4) Remove your gloves and discard them.

5) USING THIS X-RAY WIPE, CLEAN YOUR HANDS THOROUGHLY and discard wipe.

6) Open the kit, DISCARD THE LID, and place on the cleaned surface.

7) Remove the self seal bag labelled gloves and place TWO gloves on EACH of your hands.

8) Remove plastic sheet, open and place on the cleaned surface. DO NOT PLACE KIT CONTAINER ON THE SHEET.

9) Remove contents of kit and place on sheet, and then remove the pen from the nylon bag and place on sheet.

10) Remove and discard your OUTER gloves only and put on a new pair.

11) Remove one swab from a sachet, shake the swab to remove excess solvent.

12) Thoroughly swab the outer surface of both gloves hands.
(Note: Keep these gloves for taking the first "sample" swab.)

13) Place the swab in the plastic tube, fit the lid and replace the tube in the original self-seal bag and label the bag.

14) To take a swab, repeat 11), thoroughly swab surface and repeat 13).

15) Change your OUTER pair of gloves for each subsequent swab you take, and repeat 14).

PACKAGING

1) Replace both used and unused swabs in the kit container, together with the swab marked CONTROL DO NOT OPEN.
All swabs, used or unused, MUST be returned to the laboratory.
All swabs MUST be accounted for.

2) Complete the information sheet and place it in the kit container.
(This is important to aid processing of kit)

3) Seal with the new unused lid and fill in details on container label, place in new nylon bag supplied, sign and attach integrity labels TO BAG and seal bag with sellotape as normal exhibit packaging procedures. Complete and sign 38/30 label and SELLOTAPE to outside of the bag.
Deliver to the Laboratory AS QUICKLY AS POSSIBLE.

4) Discard all other materials.

COMPLETE THE FOLLOWING SECTION AND RETAIN WITH INSTRUCTIONS FOR YOUR RECORDS

Nature of incident:

Exact location of sampling:

Item swabbed, (house/car etc.)

Kit Serial No)______________
)
NIFSL No __________ Item No ___________ Date of preparation)______________
(From label on kit)

Date of sampling: ___________ Time of commencement: _______ Time of completion:_______________

Description and order of samples:

1)

2)

3)

4)

5)

6)

7)

I certify that all samples taken from____

were in strict accordance with these instructions.

Signed____________________________

Date____________________________

NIFSL NO ______________________ ITEM NO ____________________

GENERAL PURPOSE SWAB KIT INFORMATION SHEET

Reason for taking swabs:-

Examination required:-

Was any ATO action taken:-
(not inc seats of explosions)

Were any firearms discharged at the scene:-
(Details)

Date/time of incident:

Date/time of swabbing:-

Signed: ____________________________

Date: ______________________

Appendix 2 Contamination avoidance - Statement of witness reports

CONTAMINATION AVOIDANCE

There are three distinct stages in the examination of items for FDR and explosives residue, namely; sampling, sample preparation and analysis. During all stages elaborate contamination avoidance procedures are employed, some of which were introduced during the course of this work, in addition to the introduction of further control samples. 'Blanks' were also introduced to serve as a check on the apparatus, materials and reagents used, and the possibility of carry over from sample to sample. If contamination did occur, not only would it be identified at an early stage, but the procedure during which it occurred and the location at which it occurred would also be identified.

STATEMENT OF WITNESS REPORTS

As a result of an initiative throughout the U.K. forensic science service to produce more detailed, easier to understand and informative statement of witness reports (S.O.W.), the following style of report has been adopted for reporting cartridge discharge residue (CDR) casework.

The content of the S.O.W. report falls into 4 main categories following the introductory part:- 1). what was done, 2) results, 3) comments; and 4) conclusion. Recommended wording of S.O.W. reports is similar to the following:-

Category 1 "Items were examined for particles originating from the discharge of a cartridge, as used in firearms or blank firing devices. The identification of discharge residue particles is based on a combination of elemental composition and morphology and the particles fall into two groups, namely, those which can be conclusively identified as cartridge discharge residue and those which, whilst indicative of cartridge discharge, also arise from a limited number of occupational or environmental sources".

Category 2 "X particles conclusively identified as cartridge discharge residue and Y particles indicative of cartridge discharge residue were detected on item ...."

"Y particles indicative of cartridge discharge residue were detected on item ...., but none were found that could be conclusively identified as cartridge discharge residue."

"Nothing of significance was detected on item ...."

Category 3 "Deposition and distribution of cartridge discharge residue is a random process depending on numerous factors including environmental conditions, type and condition of firearm, type of ammunition, orientation of firer. Persistence depends on the type of surface, activity after firing and environmental conditions. Transfer can occur from surface to surface, and the ease of transfer will affect the persistence.

Due to the factors involved, the absence of cartridge discharge residue cannot exclude someone from involvement in a firearms related incident. The presence of cartridge discharge residue should be interpreted in conjunction with all other available information."

Category 4 "The information supplied to me in this case, along with the results, strongly supports/supports/does not support the proposition that the test subject was involved with firearms."

Minor alterations will have to be made to categories 1 and 2 to include organic FDR.

Appendix 3 Current method for FDR detection

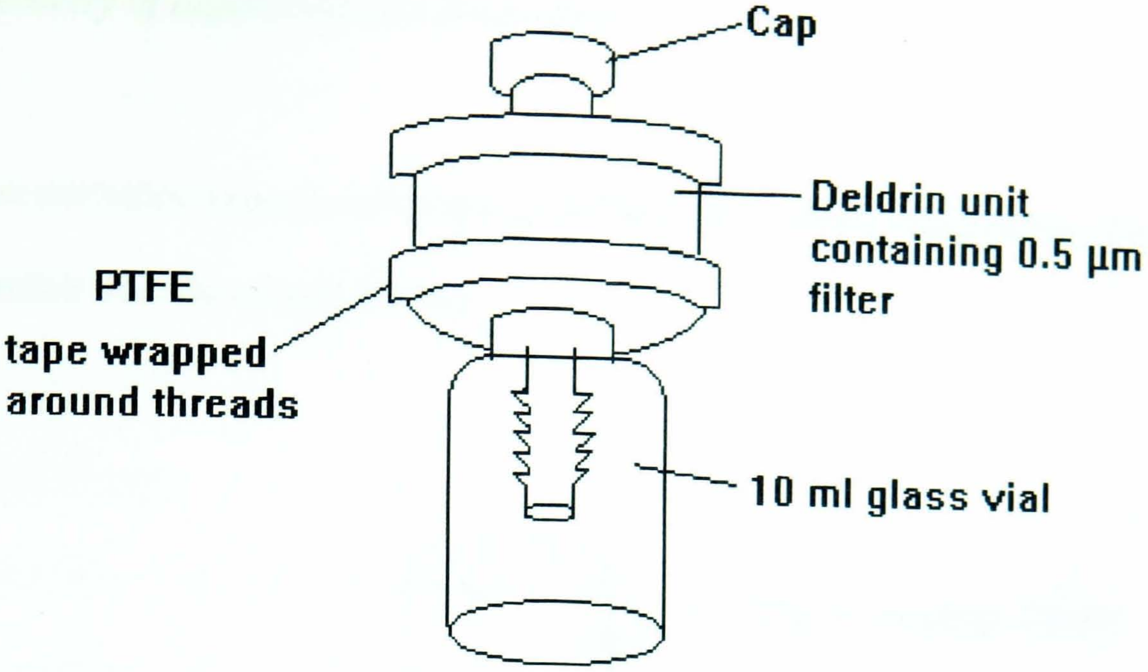
Sampling of clothing

Clothing is sampled by suction using the revised suction sampling device (Figure 7.02). Samples taken are stored at -20°C until required. The Deldrins are reusable and are washed according to the procedure given in reference 200.

At least 10 minutes should be spent suction sampling a garment. Contamination detection measures include a dry acrilan swab from the outside of the exhibit bag prior to opening and a suction sampling device to sample the air, bench and the samplers hands and sleeves immediately before the examination (combined control sample). [The suction sampling apparatus can also be used for monitoring the laboratory premises for FDR/explosive residue contamination, by air sampling at different locations.] Contamination avoidance measures include hand washing and the wearing of clean disposable laboratory coats, the use of disposable paper sheets on the work bench and the washing of the worktop with 2% $^{v}/_{v}$ hydrochloric acid, followed by 2% $^{v}/_{v}$ bleach, followed by acetone, both before and after an examination.

Recovery of organic residue from Deldrin unit

The extraction apparatus is prepared as illustrated, all the components clearly labelled with the sample number.



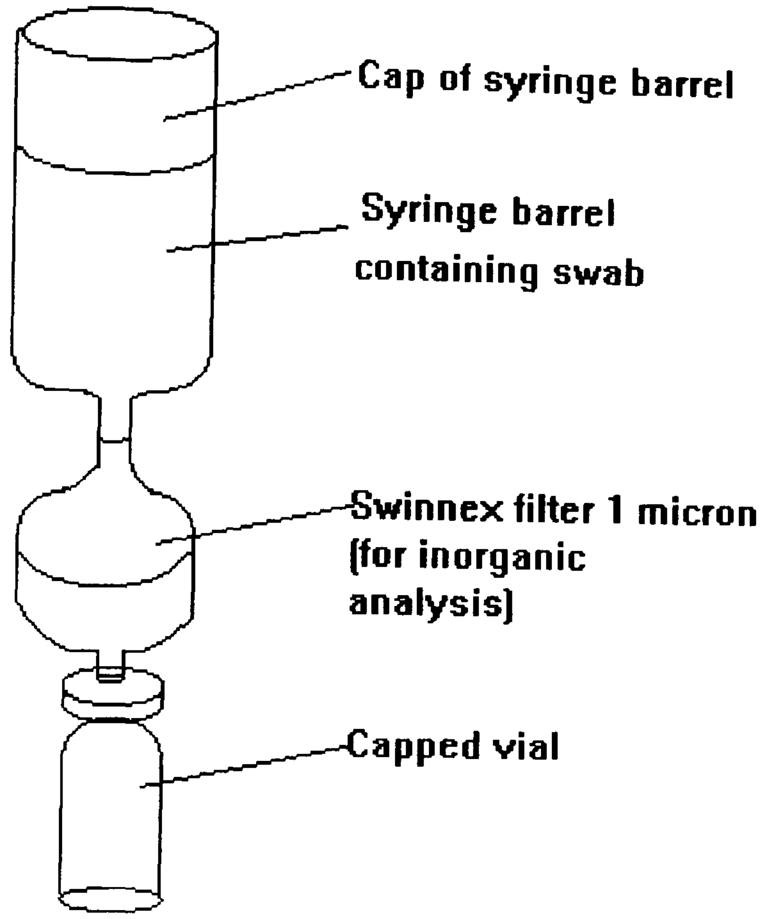
Apparatus for the extraction of organics from Deldrin

500 µl of isopropanol containing 0.6 ng/ul of 1.3 dinitrobenzene (internal standard) is pipetted into the Deldrin and allowed to sit for 2 minutes. Then 500 µl of diethyl ether is pipetted into the Deldrin and allowed to sit for 5 minutes before the apparatus is centrifuged at 1500 rpm for 2 minutes. A further 500 µl of ether is added, allowed to sit for 5 minutes, and again centrifuged at 1500 rpm for 2 minutes. The lid is replaced on the Deldrin unit and the unit retained for inorganic FDR analysis. The organic extract is transferred to a 2 ml CV glass vial for GC-TEA analysis, after which positive samples are cleaned up and concentrated by SPE and analysed by HPLC/PMDE for confirmation and then by GC/MS. The extracts are stored at -20°C whilst awaiting analysis.

Control swabs are taken from the work bench and from the operator during the organic extraction procedure for Deldrins and swabs.

Recovery of organic residue from swab

The extraction apparatus is prepared as illustrated, all the components clearly labelled with the sample number.



Apparatus for the extraction of organics from swab

750 µl of ether containing 0.4 ng/µl 1,3 DNB (internal standard) is pipetted into the syringe barrel and allowed to sit for 2 minutes before centrifugation at 3000 rpm for 1 minute. The extract is then analysed by GC/TEA, after which positive samples are cleaned up and concentrated by SPE and analysed by HPLC/PMDE for confirmation and then by GC/MS. The Swinnex and syringe barrel containing the swab are

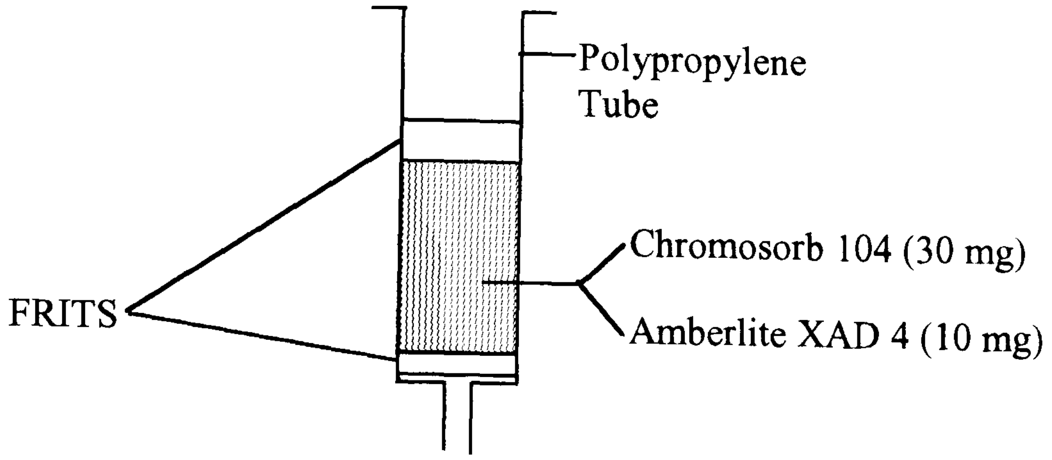
packaged and retained for inorganic FDR analysis. The extracts are stored at -20°C whilst awaiting analysis and prior to extraction the swabs are stored at -20°C.

NOTE:

The purpose of the in line 1 μm filter is to retain any inorganic particles removed from the swab by the organic extraction. The filter and filter holder are an integral part of a subsequent concentration/clean up procedure for inorganic FDR as outlined in reference 149. The filter unit consists of a 13 mm diameter Swinnex disposable filter holder containing a 13 mm diameter, 1 μm pore size, fluoropore membrane filter (Millipore FALP 01300).

Solid phase extraction procedure

Chromosorb 104, mesh size 125-150 µm and Amberlite XAD-4 are the SPE materials and prior to use both materials are prepared and cleaned according to the procedure recommended in reference 202. 40 mg of a 3:1 mixture of Chromosorb 104: Amberlite XAD-4 is packed between frits into 1.5 ml SPE tubes as illustrated. The tube is clearly labelled with the sample number.



Solid phase extraction tube

The SPE tubes are used in conjunction with a Visiprep D-L™ vacuum chamber complete with disposable flow control valve liners.

The SPE tubes are rinsed with 1.5 ml of acetonitrile (ACN) immediately prior to use in order to eliminate possible contaminants. The tubes are then conditioned with 1.5 ml deionised water to activate the sites in the support material. To ensure that the sites remain active sufficient water is retained to just cover the top frit. Organic extracts from swabs and Deldrins are blown down to near dryness in an atmosphere of nitrogen, redissolved in 100 µl of ACN and diluted 1:10 with deionised water (deionised water reduces the affinity of organic FDR and explosives residue for ACN and IPA, permitting binding to the solid phase). The samples are then passed through the SPE tube at a rate no greater than 4 ml per minute. The SPE tube is then washed with deionised water and allowed to dry.

The internal standard and any organic FDR/explosives residue are then eluted from the SPE tube with 300 µl ACN into tapered 1.1 CTVG vials, the extract now being ready for HPLC/PMDE and GC/MS analysis. The internal standard should be present at a concentration of approximately 1 ng/µl.

Recovery of inorganic FDR from Deldrin

After extraction of organic FDR/explosives residue, the 0.5 µm filter is removed from the Deldrin unit and placed in a 150 ml glass beaker. The filter holder interior and cap interior are rinsed with petroleum ether (120°C to 160°C boiling range) into the same beaker and the volume made up to approximately 20 ml, after which the beaker is ultrasonicated for 20 minutes and the contents allowed to settle.

The suspension is then filtered through a concentration/clean up system (149) consisting of two in line 13 mm diameter filters i.e. a 25 µm wire mesh initial coarse filter and a final 1 µm fluoropore filter (Millipore FALP 01300), each of which are housed in a Swinnex filter holder (Millipore SX 0001300). After filtration the 1 µm filter is placed on a 13 mm diameter aluminium SEM sample stub using double sided adhesive tape, and carbon coated using a Biorad E6430 automatic vacuum controller after which it is analysed by SEM/EDX for the presence of inorganic FDR.

The Deldrin filter holders and glassware are reused after cleaning using the procedure given in reference 200.

Recovery of inorganic FDR from swab

After extraction of organic FDR/explosives residue, the swab is placed in a glass bottle and 75 ml of dimethylformamide (DMF) added. The DMF is allowed sufficient time to dissolve the acrilan fibre and the particle suspension is filtered through the same concentration/clean up procedure as already described, using the Swinnex holder involved in the initial organic FDR/explosives residue extraction. The 1 µm filter is then placed on a stub, carbon coated and examined by SEM/EDX.

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