

# PYROTECHNIC RESIDUES ANALYSIS – DETECTION AND ANALYSIS OF CHARACTERISTIC PARTICLES BY SEM-EDS

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**ABSTRACT:** Many of the improvised explosive devices received at the Forensic Explosives Laboratory (FEL) contain pyrotechnic or home-made explosive mixtures as the explosive charge. Pyrotechnic residues contain a complex mixture of combustion products and unconsumed material. It has been reported that pyrotechnic residues comprise characteristic particles, similar to the formation of gunshot residue particles. In this study a number of pyrotechnic compositions were initiated, both confined and unconfined, and the residues analysed by scanning electron microscopy with energy dispersive spectroscopy for characteristic particles. Spheroid particles were observed in residues from both the unconfined and confined burning studies. Residues from the unconfined burning studies of flash powders and coloured fire comprised spheroid particles with an elemental composition characteristic of the original material, as did residues from the confined burning of the coloured fire compositions. Confined burning of the flash powders yielded residues whose spheroid particles were mainly comprised of elements from the metal fuel plus iron from the steel confinement pipe.

**KEY WORDS:** Forensic science; Pyrotechnic residues; Spheroid particles; SEM-EDS.

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## INTRODUCTION

Many of the improvised explosive devices received at the Forensic Explosives Laboratory (FEL) contain pyrotechnic or home-made explosive mixtures as the explosive charge, due to the ease by which they may be obtained. For devices which have been initiated the residues will comprise an often complex mixture of combustion products in addition to any unconsumed composition. In the absence of any unconsumed material determination of the original filling is carried out by interpretation of the combustion products alone. It has recently been reported that pyrotechnic residues also comprise characteristic particles that are formed at elevated temperatures and

pressures, similar to the production of gunshot residue particles [1]. These particles may also be found on the hands and clothing of persons who have fired and handled pyrotechnic devices. The aim of this study was to initiate a number of pyrotechnic compositions and to analyse the residues by scanning electron microscopy with energy dispersive spectroscopy (SEM-EDS) for characteristic particles.

## MATERIALS AND METHODS

### Pyrotechnic compositions

Four flash powders and three coloured fire compositions were tested. The flash powders comprised an inorganic oxidiser (potassium perchlorate, barium chlorate), a fuel (magnesium powder, acaroid resin) and a colour agent (barium chlorate – green, strontium carbonate – red, cryolite – amber). The white flash powder contained no additional colour agent. The coloured fire compositions comprised potassium perchlorate, potassium chlorate, acaroid resin and colour agent for the red and blue compositions and a mixture of potassium perchlorate, barium chlorate and acaroid resin for the green composition.

### Scanning electron microscopy with energy dispersive spectroscopy

SEM-EDS was performed on a JSM-5400 JEOL Scanning Electron Microscope with a PGT Energy Dispersive Spectrometer. All analyses were performed with an accelerating voltage of 20 kV. Samples were analysed on double sided adhesive carbon disks attached to aluminium stubs. Bulk analysis were performed in secondary electron imaging mode (SEI) with characterisation and photography of individual particles performed in backscatter electron imaging mode (BEI). Magnifications required to identify particles ranged from 50 × to over 1000 ×. Once a number of spheroid particles had been identified the remainder of the sample was not extensively searched.

### Unconfined burning of pyrotechnic compositions

Unconfined burning tests were performed in disposable aluminium foil dishes. Approximately 50mg of each composition was weighed into a clean dish and ignited with a bunsen flame. Residues were collected onto prepared SEM stubs by gently daubing the stub over the surface of the dish.

### Confined burning studies

Confined burning studies were performed in steel pipes (length 7.4 cm, outer diameter 4.2 cm, wall thickness 0.4 cm) with threaded end caps, washed and dried. Approximately 30 g of each pyrotechnic composition was used in the confined burning studies. The compositions were weighed into clean nylon tubing heat sealed at one end. An igniter safety fuse electric (ISFE) was inserted into the composition and the ends of the nylon tubing sealed around the ISFE legwires with adhesive tape. The filled tubing was placed into the pipe and the legwires threaded out through a small hole drilled in the centre of one of the end caps (diameter approximately 0.4 cm). The pyrotechnic compositions were sealed into nylon tubing prior to insertion into the steel pipe to prevent contamination of the screw threads with pyrotechnic material. Firings were performed in a sealed firing chamber. After firing the fragments were collected and sealed into clean nylon bags. A control firing was also performed using talcum powder in the place of the pyrotechnic composition. Residues were collected from the pipe fragments by gently brushing the inner surfaces with a clean stiff brush onto a sheet of clean paper.

## RESULTS

### Characterisation of the pyrotechnic compositions by SEM-EDS

Each type of composition comprised a fine mixture of the irregularly shaped individual components. Table I shows the elemental composition of each pyrotechnic powder.

TABLE I. ELEMENTAL COMPOSITIONS OF THE INTACT PYROTECHNIC POWDERS

Composition	Elements detected by SEM-EDS
White flash powder	Magnesium, chlorine, potassium
Amber flash powder	Magnesium, chlorine, potassium
Red flash powder	Magnesium, strontium, chlorine, potassium
Green flash powder	Magnesium, chlorine, barium
Red coloured fire	Chlorine, potassium, strontium
Green coloured fire	Chlorine, potassium, barium
Blue coloured fire	Chlorine, potassium, copper

### Unconfined burning pyrotechnic residues

Results for the unconfined burning residue analyses are shown in Table II. Bulk analysis of each residue showed then to have an elemental composition characteristic of the intact powder. In the majority of compositions the residues predominantly comprised irregular particles. In most cases spheroid particles were detected whose elemental compositions were also characteristic of the original powder.

TABLE II. ANALYSIS OF UNCONFINED BURNING PYROTECHNIC RESIDUES

Composition	Bulk elemental analysis	Spheroid particle elemental analysis
White flash powder	Mg, Cl, K	Cl, K, Mg
Amber flash powder	Mg, Cl, K	Mg, Al, Si
Red flash powder	Mg, Cl, K, Sr	Mg, Cl, K, Sr
Green flash powder	Mg, Cl, Ba	Cl, Ba Cl, Ba, Mg
Red coloured fire	Cl, K, Sr	Cl, K Sr, Cl Cl, K, Sr
Blue coloured fire	Cl, K, Cu	Cl, K
Green coloured fire	Ba, K, Cl	Cl, Ba, K

### Confined burning pyrotechnic residues

Confined burning tests were performed in steel pipes. In the majority of firings the end cap through which the legwires of the ISFE exited was fragmented with the remainder of the pipe remaining intact. Firings of the green flash powder and red coloured fire produced minimal damage to the pipes, gases were vented through the whole in the end cap and the pipe remained intact. These firings produced large quantities of residues and it was concluded that partial firings had occurred. For the remainder of the firings the residues consisted of a fine powder coating the inside of the pipe. SEM-EDS bulk analysis of the residues, summarised in Table III, showed them to have elemental compositions characteristic of the original material plus iron originating from the steel pipe. Spheroid particles were detected in the residues from all firings. Those arising from the coloured fire compositions had elemental compositions characteristic of the unburned material. Those particles detected in the flash powder residues were comprised predominantly of magnesium from the fuel and iron from the steel pipe.

TABLE III. ANALYSIS OF CONFINED BURNING PYROTECHNIC RESIDUES

Composition	Bulk elemental analysis	Spheroid particle elemental analysis
White flash powder	Mg, Fe, Cl, K	Mg Fe, Mg
Amber flash powder	Mg, Fe, Cl, K	Fe Fe, Mg
Red flash powder	Mg, Fe, Cl, K, Sr	Mg, Fe, Sr Mg, Fe, Cl, K
Green flash powder	Mg, Cl, Ba	Mg Mg, Cl, Ba
Red coloured fire	Cl, K, Sr	Cl, K, Sr
Blue coloured fire	Cl, K, Fe, Cu	Cl, K, Cu
Green coloured fire	Ba, Cl, Fe, K, Si	Cl, Ba, K, Fe

## DISCUSSION

The pyrotechnic powders studied were of two types – flash powders and coloured fire compositions. Spheroid particles were detected in all residues from the burning of the seven studied pyrotechnic compositions. Spheroid particles detected in the unconfined burning residues had elemental compositions, on the whole, which were characteristic of the original material. However unconfined burning of the blue coloured fire composition gave rise to spheroid particles from which copper was absent despite copper being detected in the bulk residue. Confined burning of the coloured fire compositions also gave rise to spheroid particles whose elemental composition was characteristic of the original material, and copper was this time detected in the spheroids in the blue coloured fire residue. Confined burning of the flash powders gave rise to spheroid residue particles whose compositions were predominantly magnesium and iron. Other elements present in the original powder were only detected, if at all, in the spheroids at a low level. The predominance of magnesium/iron spheroid particles in only the confined flash powder firings can possibly be explained by the different reaction temperatures of the flash powder and coloured fire composition. The spheroid particles are formed from vaporised material which condenses into droplets on cooling. For iron to be incorporated into the spheroid residue particles reaction temperatures need to be reached which will vaporise iron particles produced during the deflagration. Coloured fire compositions have a lower reaction temperature than the flash powders which may not be sufficient to vaporise the iron particles. Evidence for this is supported by the few spheroid particles detected in the green flash powder firing, and in particular no spheroid particles containing iron were detected. The green flash powder is

thought to have undergone only a partial firing and therefore the high temperature of the other flash powder firings was not reached. It has been shown in this study that characteristic spheroid particles can be detected in pyrotechnic residues. In the absence of unconsumed material these particles, in combination with other chemical analyses i.e. CE, IC, may aid the forensic scientist in the identification of the original pyrotechnic material.

References:

1. Giacolone J. R., Forensic fireworks analysis and their residues by scanning electron microscopy/energy-dispersive spectroscopy, *Scanning* 1998, vol. 20, pp. 172–173.