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Pyrotechnic Reaction Residue Particle Analysis

ABSTRACT: Pyrotechnic reaction residue particle (PRRP) production, sampling and analysis are all very similar to that for primer gunshot residue. In both cases, the preferred method of analysis uses scanning electron microscopy to locate suspect particles and then uses energy dispersive x-ray spectroscopy to characterize the particle's constituent chemical elements. There are relatively few times when standard micro-analytical chemistry performed on pyrotechnic residues may not provide sufficient information for forensic investigators. However, on those occasions, PRRP analysis provides a greatly improved ability to discriminate between materials of pyrotechnic origin and other unrelated substances also present. The greater specificity of PRRP analysis is the result of its analyzing a large number of individual micron-sized particles, rather than producing only a single integrated result such as produced using standard micro-analytical chemistry. For example, PRRP analyses are used to demonstrate its ability to successfully (1) discriminate between pyrotechnic residues and unrelated background contamination, (2) identify that two different pyrotechnic compositions had previously been exploded within the same device, and (3) establish the chronology of an incident involving two separate and closely occurring explosions.

KEYWORDS: forensic science, pyrotechnic reaction residues, scanning electron microscopy, x-ray energy dispersive spectroscopy

A pyrotechnic material (also called pyrotechnic composition) can be defined as:

A mixture of chemical elements and compounds that is capable of a self-contained and self-sustained exothermic chemical reaction, for the production of heat, light, gas, smoke or sound (1).

These are the reactive materials used to make safety matches, highway fusees (flares), fireworks, pyrotechnic special effects, many rocket propellants, and many devices used by the military such as those for signaling, delay trains, and obscurants.

In the course of pyrotechnic reactions, residues (reaction products) are generated. This is true whether the reaction is occurring in the primer of a cartridge in a gun, a pipe-bomb filled with a fireworks flash powder, or an incendiary device made from a highway fusee (flare). A large portion of the reaction products from nearly all pyrotechnic compositions are solids at room temperature. As such these reaction products are potentially available for collection and analysis using much the same methodology as used for primer gunshot residues (PGSR). In some cases, pyrotechnic reaction residue particle (PRRP) analysis is a useful adjunct to conventional micro-analytical chemistry by offering forensic information not otherwise provided (or not provided with a high confidence level).

Much of the current interest in PRRP analysis (2–4) originated with articles expressing concern that some fireworks might be capable of producing residue particles meeting the criteria for PGSR (5–7). PRRP production, sampling and analysis are all very similar to that of PGSR. Both types of residue originate from energetic chemical reactions that generate products that are initially melted and/or vaporized. These chemical reaction products are dispersed by the temporary and permanent gases from the reac-

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tion, and then solidify as tiny spheroidal particles deposited on objects in the area. For both PRRP and PGSR, sampling is generally accomplished using conductive carbon adhesive dots or tape. As with PGSR, the preferred method of analysis is using scanning electron microscopy (SEM) to locate suspect particles, and then using x-ray energy-dispersive spectroscopy (EDS) to characterize the suspect particle's constituent chemical elements.

Perhaps two general examples will make it more clear when and to what extent PRRP analysis may be helpful. On occasion, standard micro-analytical chemistry may fail to sufficiently discriminate between pyrotechnic residues and other unrelated substances also present on sampled items. These unrelated substances may preexist the pyrotechnic incident, they can be contributed during the course of the event but not be from the pyrotechnic composition, they may be deposited postevent, or any combination of the three. When unrelated materials are known to be present, often they can be successfully accounted for in the chemical analyses, although that may require additional or more complex analyses. However, when unrelated material is not known to exist or if it shares chemical species in common with the pyrotechnic composition, erroneous information may result. In that event, these unrelated residues could be incorrectly identified as part of the pyrotechnic residue, or those species in common could be missed because of being attributed to a nonpyrotechnic source. It is the morphologic specificity of PRRP (spheroidal particles in the range from about 0.5–20 μ m) that will generally allow the successful differentiation between PRRP and non-PRRP materials.

In addition to offering help in differentiating between pyrotechnic and unrelated substances, there are times when PRRP analysis can be used to differentiate between pyrotechnic residues present on the same item, but contributed by different pyrotechnic sources, possibly at different times. With such information, insight might be gained into the cause and course of an incident that would not be available using standard analytical methods. This is because standard micro-analytical chemistry almost certainly would produce a single set of results representing the combination of the various different pyrotechnic residues present on a sampled item, whereas PRRP analysis can easily differentiate between the different pyrotechnic residue sources.

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Background

There are many similarities between PGSR and PRRP and their analysis.

- Their means of production is the same (from energetic chemical reactions).
- The morphology is the same (spheroidal particles approximately from 0.5 to 20 µm in diameter).
- The manner of deposition is the same (particles are projected and settle onto all surfaces in the area of the event).
- The manner of collection is the same (using conductive adhesive carbon dots).
- The manner of analysis is the same (an SEM to identify suspect particles and EDS to identify elements).

However, there are three important differences between PGSR and PRRPs and their analysis.

- The elements present in PRRPs are generally different and more varied than in PGSR.
- The elements present in PRRPs tend to mostly be of relatively low-atomic number.
- The amount of PRRPs produced is typically several orders of magnitude greater than that of PGSR.

The first two of these differences potentially makes performing PRRP analysis more difficult than PGSR analysis, whereas the third generally makes it much easier. A discussion of these differences and much additional information were presented in an earlier article in this journal (4) and will not be repeated here.

Many primers used in small arms contain approximately 20 mg of composition that is potentially available to produce detectable residues. Based on thermodynamic modeling a little more than half of these residues end up as solids (Webb R performed chemical reaction product modeling using the ICT thermodynamic free energy modeling code). (The burning of the smokeless powder contained in small arms ammunition is not the source of Pb/Sb/Ba containing particles associated with PGSR.) In strong contrast to PGSR, even an item as small as an M-80 explosion/gunfire simulator (i.e., a large firecracker) produces more than 2 g of solid residue (8), which is more than 200 times that produced by a small arms primer. The burning of Black Powder produces approximately 65% solid residues (9). Accordingly, burning a pound of powder produces nearly 300 g of solid residue, which is nearly 30,000 times that from a small arms primer. With the orders of magnitude greater quantities of detectable residues produced by pyrotechnics, finding sufficient quantities of PRRPs to characterize the nature (and to some extent, the course) of incidents involving pyrotechnics is much less of a problem than for PGSR. Figure 1 is an example of the very large number of PRRPs that can be found on surfaces in the vicinity of explosive pyrotechnic reactions. This is an electron micrograph of a small portion of a piece of plastic initially located approximately 0.5 m (20 in) from an explosion produced by 28 g (1 oz) of a common fireworks flash powder, in a relatively clean environment (i.e., in an area relatively free of dirt and other potential sources of particulate background).

Basic PRRP Methodology

As mentioned above, the basic methodology of PRRP analysis has much in common with PGSR analysis. First, samples are collected, generally from objects from the immediate area of a pyro-

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FIG. 1—An example of the number of pyrotechnic reaction residue particles that can be found after a small pyrotechnic explosion.

technic event, and also from persons and their clothing when such is available. In many cases samples can be collected directly on conductive carbon dots already mounted on the stubs commonly used for SEM work. However, in the case of sampling from rigid objects with curvature, sample collection is often facilitated by first attaching the carbon dot to the center of a narrow strip of Velostat (electrostatically conductive polyethylene film) cut to approximately 20×60 mm (approximately $3/4 \times 2.5$ in), and using one's finger (against the backside of the Velostat) to press the carbon dot attached to the film against the curved or irregular surface to be sampled. Then the sample-loaded carbon dot and film can be attached to the SEM stub using a second carbon dot and the excess Velostat film trimmed away.

Except when attempting to produce high-quality images, samples are generally not carbon or sputter coated. This is because the PRRPs and other particles collected are generally sufficiently conductive so as not to cause serious problems with charging. Moreover, as discrimination based on back-scatter contrast differences is rarely if ever useful in identifying PRRPs, the SEM is typically operated in the secondary electron mode. Accordingly minor problems with excessive contrast (flaring) can generally be tolerated.

In the search for and characterization of PRRPs on an object thought to have been exposed to the pyrotechnic event, primary attention is paid to those particles of the correct morphology. See Fig. 2 for some examples of the range of appearance of PRRPs. These suspect particles are then analyzed using x-ray EDS and the information archived. During this particle search process, usually a moderate number of nonspheroidal particles are also investigated using EDS. This helps to establish the components present in the background on that specific item. (It is possible that this background material may be different than that on other items and in bulk background samples taken from the area in general.) Analysis of the background components on individual items may thus be useful in helping to increase confidence in the identification and characterization of PRRPs in some cases.

Having found and characterized a sufficient quantity of PRRPs, it will almost always be possible to say something definitive about the nature of the pyrotechnic material involved. This is especially true when the analyst has a fairly thorough knowledge of pyrotechnic chemistry, both the materials used and their expected reaction products.



FIG. 2—Examples of a range of appearance of pyrotechnic reaction residue particles.

After a brief discussion of the instrument used in this study, a series of three examples are presented to illustrate some potentially useful capabilities of PRRP analysis.

SEM/EDS Equipment Used

The SEM used in this work was a manually operated AMRAY 1000, relatively recently remanufactured by E. Fjeld Co. (N. Billerica, MA). For this work, the instrument is most often used with an accelerating potential of 20 kV and operated in the secondary electron mode. The instrument provides software-driven digital imaging. The x-ray spectrometer is energy dispersive, using a Kevex Si(Li) detector (Thermo Electron Corporation, Scotts Valley, CA) with a 7.6-µm beryllium window, used in conjunction with an American Nuclear System model MCA 4000 multichannel analyzer and their Quantum-X software (version 03.80.20). For EDS work, a 200-µm final aperture was used with a condenser lens setting that produced a spot size estimated to be a little less than 0.4 µm and providing approximately 3 nA net specimen current. (Specimens imaged in the micrographs of Fig. 2 were sputter coated with gold and a 100-µm final aperture was used.)

In the spectra reproduced for this paper, the vertical scales are the square root of the number of counts per energy channel. The largest x-ray peak in each spectrum was normalized to the same full-scale height. Moreover, while data was collected to nearly 20 keV, the horizontal (energy) axis was truncated at a point a little above the last significant x-ray peak. Similarly, the portion of the spectrum below approximately 0.5 keV was not included. Also, generally several similar spectra have been added together to improve their individual statistical precisions. These procedures were used to more clearly display the points of interest in the spectra for this paper.

Example 1: Background Discrimination

When a pyrotechnic device is exploded on the ground, the force of the explosion will mobilize a substantial amount of soil material, some of which will be deposited on remnants of the device and other objects in the area. This process, however, does not result in a mixing of the soil and pyrotechnic components within individual particles. The temperature and duration of the explosion is not sufficient to vaporize or even melt the soil particles. As a result, intimate commingling of soil and pyrotechnic components does not occur. In addition, because they have not melted, soil particles tend to maintain their generally nonspheroidal morphology. Accordingly, the normal PRRP methodology works fairly well to differentiate successfully between PRRPs and soil material. There can, however, be a complication wherein some pyrotechnic reaction products may tend to collect on the surface of the mobilized soil particles within the vapor cloud of the explosion. This tends to occur when there is a large difference between the high temperature of an explosive fireball and much lower condensation temperatures of some of the reaction products. The example below is one case where this occurred. (While this is discussed briefly below, a more complete discussion is deferred until a future article.)

Because of the large degree of commonality between the chemical elements present in inorganic soil components and pyrotechnic compositions (3), combined with a somewhat similar range of solubilities and reactivities, conventional micro-analytical chemistry may fail to differentiate sufficiently between the two sources. Further, even a direct comparison between the samples collected from the immediate area of the explosion and PRRP-free soil may fail to produce fully definitive data on the nature of the pyrotechnic residues present. This is because, while PRRPs are quite abundant compared with the amounts of PGSR typically produced, the PRRPs present on sampled items after an explosion on the ground may be overwhelmed by the even larger amounts of soil collecting on those items.

A demonstration was conducted using a small polyethylene container (a common 35-mm film can) filled with approximately 28 g (1 oz) of a typical fireworks flash powder (70% potassium perchlorate and 30% aluminum powder). The container was placed directly on the ground and exploded using an electric match that had been installed in the device (see Fig. 3). Following the event, residues were collected from the previously cleaned surfaces of objects placed in the immediate area of the small explosion. One collecting surface, seen in Fig. 3, was positioned approximately 150 mm (6 in) from the explosive charge. (The collecting surface used was a small piece of 1/8-in tileboard with a hard, thick and tightly laminated surface. The collecting surface had been cleaned prior to the test.) Figure 4 presents a series of three EDS spectra, two collected from residues on those surfaces plus that from the unaffected dirt ("Pristine Dirt").

The top spectrum, labeled "(1) Dirt plus PRRP" is a composite of two EDS spectra taken at low magnification, scanning over relatively large portions of a postexplosion residue sample. This integrates over a very large number of individual particles. In the



FIG. 3—Setup for background discrimination demonstration, showing a 35-mm film can of fireworks flash powder and a collection surface attached to a heavy metal support.



FIG. 4—A collection of three composite spectra taken from samples of pyrotechnic residues commingled with soil.

spectrum the major peaks are from aluminum, silicon, sulfur, potassium, calcium, and iron. When examined more closely, minor peaks from sodium, magnesium, chlorine, titanium, and copper can be seen to be present as well. The second spectrum, labeled "(2) Pristine Dirt," is a composite of the spectra from 16 individual soil component grains. (Based on the different assemblages of constituent elements present, the individual soil grains fell into four reasonably distinct geologic categories.)

The results embodied in spectra 1 and 2 are somewhat similar to what would be expected to be produced if conventional microanalytical chemistry were performed on these two samples, with the exception that chemical species (ions) and not just the elements present would be determined. These two spectra are quite similar to each other. Especially, considering that any two samples of pristine dirt (both without the presence of PRRPs) would be expected to present slight differences in the quantities of the elements found to be present. Accordingly, while it is likely that standard micro-analytical chemistry would reveal the presence of some unreacted and slightly soluble perchlorate ion from the flash powder, in this case it seems unlikely that a statistically significant excess presence of aluminum ions would be found. To the contrary, by using the morphological criteria for PRRPs, the presence of aluminum in the PRRPs is unambiguous. This is presented as the third spectrum labeled "(3) PRRP," which is a composite spectrum of 16 such particles. Another potential problem for results from micro-analytical chemistry is that trace quantities of other species might be found that are unrelated to the explosive event, but which might be incorrectly interpreted as originating from the pyrotechnic composition. (For example, chlorate ions could originate from weed-killer, and nitrate, sulfate, ammonium, and potassium ions could originate from fertilizer.) However, if background soil samples are also analyzed, they are likely to also have the same suspect ions present in varying amounts.

In this case, in the PRRP spectrum only an aluminum peak (from Al₂O₃) from the flash powder reaction products is readily seen. Substantial potassium and chlorine peaks (from the KCl reaction product) are essentially not seen in the composite spectrum but are seen weakly in some of the individual particle spectra. In this case, the virtual absence of the potassium and chlorine peaks is a result of the condensation (sublimation) temperature of KCl being moderate (1500°C) compared with the high temperature of the fireball and the condensation (boiling) temperature of aluminum oxide (2980°C). The effect is to cause the KCl to tend to condense preferentially onto the surface of the relatively cool dirt particles that were mobilized as a result of the explosion. As mentioned above, a thorough discussion of this phenomenon is beyond the scope of this paper, but this does help to make the point that knowledge of the expected products of pyrotechnic reactions, and the physical and chemical properties of those products, can be important in correctly interpreting PRRP results.

Note that in this example, neither micro-analytical chemistry nor PRRP analysis were thought to be likely to yield completely definitive results about the nature of the pyrotechnic explosive involved. Rather, it is the combination of information produced by both techniques that can be expected to produce fairly unambiguous results.

Example 2: Use History

A pyrotechnic accident was thought to have been caused when a reusable performance appliance was loaded with an inappropriately powerful explosive charge. The appliance was made using approximately a 1-ft length of 102-mm- (4-in-) diameter steel tubing having a 6-mm (1/4-in) wall, welded to a thick steel base plate for stability (see Fig. 5 for its basic construction). This type of appliance can be used in stage productions (i.e., entertainment) to produce both an audible and visual effect by exploding an appropriately sized and constructed pyrotechnic charge within it. Following the incident in question, the appliance was held as evidence and after several months the inside surface of the device was sampled and analyzed for PRRPs. The result was that approximately equal numbers of two fairly distinct types of PRRPs were found to be present. Their EDS spectra (composites from eight particles each) are shown in Fig. 6. One type of PRRP had an



FIG. 5—An illustration of the basic construction of a reusable performance appliance used to produce visual and audible effects.



FIG. 6—Two composite spectra taken from a performance appliance whose misuse was thought to have caused an accident.

abundance of strontium present with relatively little titanium and aluminum, while the other had no strontium and an abundance of titanium and aluminum. It is important to note that less than 10% of the PRRPs were found to contain roughly comparable quantities of strontium, titanium, and aluminum.

Based on witness accounts and postincident photography, it had been theorized that the performance appliance in question had been loaded with an inappropriately powerful explosive charge rather than what was normally used in it. The identification of two distinctively different PRRPs supported the theory that there had been a recent change in the type of pyrotechnic explosive used in the appliance. Had a single type of pyrotechnic charge been used, one principally producing both titanium and strontium residues (in addition to the magnesium and aluminum), then the PRRPs would have all been somewhat similar in composition, with the vast majority of PRRPs containing substantial and approximately equal quantities of aluminum, strontium, and titanium.

Armed with the fairly definitive information about the use history of the performance appliance, employees acknowledged that both red color-producing (strontium-containing) fireworks star shells and much more powerfully explosive titanium fireworks salutes had been exploded within the device.

In this case, PRRP analysis produced results that would not have been possible with conventional methods. Had the samples from the stage appliance been analyzed using conventional microanalytical chemistry, only a single combined set of results would have been produced.

Example 3: Incident Chronology

When an explosion occurs, if the blast or thermal output is sufficient and there are other explosive devices in the area, it is possible for the first explosion to initiate secondary explosions. To simulate such an event on a small scale, a demonstration was staged wherein two relatively small pyrotechnic charges were exploded within 0.2 sec of one another. One of the two explosive charges is seen close-up in the upper photograph of Fig. 7, and the pair of charges can be seen in the lower photograph at opposite ends of the setup. Each of the explosive charges was approximately 28 g (1 oz). One was a standard fireworks flash powder (70% potassium perchlorate and 30% aluminum powder). The other was a common theatrical concussion powder, a type of fuelrich flash powder (50% strontium nitrate and 50% magnesium). The distance between the two charges was approximately 0.5 m (20 in). The demonstration was conducted in a research bay approximately $4 \times 4 \times 4$ m ($12 \times 12 \times 12$ ft) in size, and configured such that one wall was completely removed and open to the outside environment. After exploding the two charges, the room was cleared of smoke using a large exhaust fan.

Once the air had cleared, fragments of the containment vessels (35-mm film cans, one made from black polyethylene and the other made from uncolored polyethylene) were collected and mounted for PRRP analysis. Each specimen was made using three small pieces of the same type (color) plastic, each of which were mounted with the same inside up or outside up orientation. (The inside/outside orientation of the fragments was determined by their curvature.) Thus a total of four specimens of the containment vessels were prepared. Also sampled was the midpoint of the light-colored collecting surface, seen in Fig. 7, extending between the two steel blocks to which the explosive charges were attached. (The mostly white strip of collecting material was 1/8-in tileboard, which has a hard, thick, and water-resistant coating bonded to the base material.) The collecting surface had been cleaned prior to the test. The pattern of small random spots seen in the upper photo of Fig. 7 had been permanently imprinted within the surface coating by the manufacturer. This midpoint PRRP specimen was



FIG. 7—Photographs showing the setup for a demonstration in which pyrotechnic reaction residue particles are used to discover details of the course of an explosive event.



FIG. 8—A series of four composite spectra representing the four categories of pyrotechnic reaction residue particles used in this analysis.

prepared by contact using the carbon dot and Velostat method described above.

Based on an initial brief analysis of the PRRPs on the sampled items, it was fairly obvious that two chemically different pyrotechnic explosives were involved. Thus, following somewhat the same scheme as in the "Use History" example above, PRRP spectra were divided into four categories. The composite spectra in Fig. 8 are representative of the four categories: "(1) Pure Fireworks Flash," "(2) Mostly Fireworks Flash," "(3) Mostly Theatrical Concussion," and "(4) Pure Theatrical Concussion." In this brief demonstration, only 25 PRRPs from each of five samples were analyzed. (In an actual investigation, the number of samples and the number of particles per sample would be adjusted to produce results with the desired level of confidence.) Table 1 is a summary of the numbers of PRRP in each category found on each type specimen. Note that the PRRPs found on the inner surfaces of the two containment vessels were radically different. Of the PRRPs on the inside surface of the black plastic fragments, approximately 85% were category 4, having only the components of the theatrical concussion powder. Of the PRRPs on the inside surface of the clear plastic fragments, 70% were category 1, having only the components of the fireworks flash powder. Accordingly, it is obvious that the black film can must have contained the theatrical concussion powder and the clear film can must have contained the fireworks flash powder. Had the pyrotechnic compositions (explosive charges) both been a single mixture of all of the ingredients, the PRRPs on the inside surfaces of both containers would have been essentially the same and the vast majority would have been a mixture of all the individual components.

Given the geometry of the demonstration setup, it can be expected that there will be somewhat of a tendency for each exploding charge to project its PRRPs outward toward the other charge. However, note that the PRRPs found on the outside surfaces of both containers were mostly category 1, having only the components of fireworks flash powder. Note further that while 40% of the PRRPs on the outside surface of the black container were category 1, the outside surface of the clear container had no PRRPs of category 4 (pure concussion powder components). This is consistent with the flash powder charge having exploded first. This is simply because, if the flash powder charge exploded first, its container would no longer be present in the immediate area to collect PRRPs from the concussion powder charge when it explodes.

In Table 1, the column labeled "Surface Total" is the total of the PRRPs collected on the exteriors of the two containment vessels plus those from the midpoint of the collecting surface. Note that approximately 40% of the PRRPs fell into the two "Mixed" categories (categories 2 and 3). This is an indication that two distinct explosions took place, but with only a brief delay between the two. (In this demonstration the two explosions were made to occur within approximately 0.2 sec.) From other demonstrations using the same charges and setup, it has been observed that

- Had the two explosions occurred at the same instant, there would have been more complete mixing of their respective fireballs, and roughly 70% of the PRRPs would have contained a significant mixture of the components of the two pyrotechnic explosives.
- Had the two explosions been more widely separated in time (even with as little as 0.2 sec between the explosions), there would have been no mixing of the fireballs and only a little mixing because of the remobilization of previously deposited PRRPs, resulting in roughly 10% of the PRRPs having contained a mixture of the components of the two pyrotechnic explosives.

Accordingly, in this case, an analysis of PRRPs has shown that two different types of explosive charges were involved, that the two explosions took place, that the flash powder charge exploded

TABLE 1—Numbers of each of the four types of pyrotechnic reaction residue particles (PRRPs) on items sampled.

PRRP Category	Black Plastic		Clear Plastic			
	Outside	Inside	Outside	Inside	Surface Midpoint	Surface Total
(1) Pure fireworks flash	10	2	15	18	3	28
(2) Mostly fireworks flash	7	1	6	5	2	15
(3) Mostly theatrical concussion	4	1	4	2	7	15
(4) Pure theatrical concussion	4	21	0	0	13	17

first, and that the two explosions were only very slightly separated in time. With a careful series of chemical analyses of the fragments (both containers and both the inside and outside surfaces) and other exposed surfaces in the area, most but not all of the above conclusions might have been drawn, but with a lesser degree of confidence.

Conclusion

PRRP analysis, which only identifies the chemical elements present in the residue, will not supplant micro-analytical chemistry, which has the ability to identify chemical species. However, there are times when PRRP analysis can provide information that is a useful adjunct to normal micro-analytical chemistry. There will also be occasions when PRRP analysis can provide information that is beyond the reach of standard micro-analytical chemistry. Accordingly, a more extensive knowledge of PRRP analytical techniques may be of use to the forensic analyst, in providing useful information to investigators.

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