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## Applications for Pyrotechnic Reaction Residue Particle Analysis - By K. L. Kosanke,<sup>a</sup> R. C. Dujay<sup>b</sup> and B. J. Kosanke<sup>c</sup>

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### Special Points of Interest:

- IAMA Acknowledgments
- IAMA Website: [www.iamaweb.com](http://www.iamaweb.com)
- Terrorist/Terrorism Investigation Software
- Upcoming Meetings

### Introduction

Pyrotechnic reaction residue particles (PRRP) are spheroidal particles ranging in size from about 0.5 to 20 microns that are produced during the functioning (burning or explosion) of pyrotechnic devices. One such pyrotechnic device is the primer of small arms ammunition. Accordingly, much of the same well-established methodology developed for the analysis of primer gunshot residue (PGSR) is also applicable to the analysis of PRRP from incendiary and explosive pyrotechnic devices. However, there are three important differences:

- The elements present in PRRP are generally different and more varied than in PGSR and are also among the most common elements in the Earth's crust.
- The elements present in PRRP tend to mostly be of relatively low atomic number, such that backscatter image contrast generally is not sufficient to help locate PRRP.
- The amount of PRRP produced is typically 100 to 100,000 times greater than for PGSR, facilitating the collection and analysis of ample numbers of PRRP.

The first two of these differences potentially makes performing PRRP analysis more difficult than PGSR analysis, whereas

the third generally makes it easier. More information about the production and analysis of PRRP was presented in a previous article in this publication,<sup>[1]</sup> as well as elsewhere,<sup>[2,3]</sup> and will not be repeated here.

There are two areas in which PRRP analysis may be especially helpful for the investigation of incidents involving pyrotechnic devices. On occasion, standard micro-analytical chemistry may fail to sufficiently discriminate between pyrotechnic residues and other unrelated substances also present on the items sampled. These unrelated substances may preexist the pyrotechnic incident, they may be contributed during the course of the event but not from the pyrotechnic composition, or they may be deposited post-event. When unrelated materials are known to be present, often they can be successfully accounted for during chemical analysis, although that may require additional or more complex analyses. When unrelated material is unknowingly present or if it shares chemical species in common with the pyrotechnic composition, erroneous information may result from standard chemical analyses. 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 the non-pyrotechnic source. As with PGSR, it is the morphologic specificity of

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PRRP that will generally allow the successful differentiation between PRRP and background 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.

### SEM / EDS Equipment Used

The SEM used in this work was a manually operated AMRAY 1000, recently remanufactured by E. Fjeld Co. (N. Billerica, MA, USA). 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 with a 7.6  $\mu\text{m}$  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  $\mu\text{m}$  final aperture is typically used with a condenser lens setting that produces a spot size estimated to be a little less than 0.4  $\mu\text{m}$  and providing approximately 3 nA specimen current.

In the spectra reproduced for this paper, the vertical scales are the square root of the number of counts per energy channel. Each spectrum was normalized to provide the same full-scale height for the largest X-ray peak. Also, while data was collected to nearly 20 keV, the 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. Often, 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.

### PRRP Example 1: Background Discrimination

When a pyrotechnic device is exploded on the ground, the force of the explosion will mobilize some of the soil particles, which will intermix with the pyrotechnic reaction

products and be deposited on remnants of the device and other objects in the area. In the case of a powerful explosion, a substantial amount of soil material will be mobilized and will mix with the PRRP that is produced; however, for the most part this mixing does not occur within individual particles. Rather a mixture of particle types is produced (i.e., most particles are either essentially pure PRRP or essentially unaltered soil particles). For the most part the temperature and duration of the explosion is not sufficient to melt the soil particles. Thus soil particles tend to maintain their generally non-spheroidal morphology, and the spheroidal morphology of PRRP serves well to successfully differentiate between PRRP and soil components. (There are also compositional differences that aid in the differentiation between soil particles and PRRP.<sup>[2,3]</sup>)

Because of the large degree of commonality between the chemical elements present in inorganic soil components and pyrotechnic compositions, combined with a somewhat similar range of solubilities and reactivities, conventional micro-analytical chemistry may fail to differentiate sufficiently between the two. Further, even a direct comparison between the samples collected from objects in the immediate area of the explosion (samples containing a mixture of soil particles and PRRP) and PRRP-free soil, may fail to produce fully definitive data on the nature of the pyrotechnic residues present. This is because, while PRRP is quite abundant compared with the amount of PGSR typically produced, the amount of PRRP present on sampled items after an explosion on the ground may be overwhelmed by the even larger amount of soil components 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 ounce) of a typical fireworks flash powder (70% potassium perchlorate and 30% aluminum powder). The reaction of this flash powder produces potassium chloride and aluminum oxide by the chemical equation:



The container was placed directly on the ground and exploded using an electric match that had been installed in the device, see Figure 1. Following the event, residues were collected from a previously cleaned surface of an object placed in the immediate area of the small explosion. The collecting surface, seen in Figure 1, was positioned

*(Continued on page 3)*

## Applications for Pyrotechnic Reaction Residue Particle Analysis.... Continued

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approximately 150 mm (6 inches) from the small explosive charge. The collecting surface was a small square of 1/8-inch tileboard, having a hard, thick and tightly laminated surface. Figure 2 presents a series of three EDS spectra collected from residues on this surface plus that from the unaffected ("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 post-explosion residue sample. This method integrates over a very large number of individual particles to produce an average EDS spectrum. In this spectrum the major peaks are from aluminum, silicon, sulfur, potassium, calcium and iron. However, 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 geologically distinct categories). The results embodied in spectra 1 and 2 are similar to what might be expected to be produced if conventional micro-analytical chemistry were performed on these two samples, with the exception that chemical species (ions) and not just the elements present would be determined.

The two sets of results (spectra 1 and 2) are quite similar to each other. In addition, even two samples of pristine dirt (both without the presence of PRRP) would be expected to present slight differences in the quantities of the elements that would be present. Accordingly, while it is likely that



Figure 1. 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.

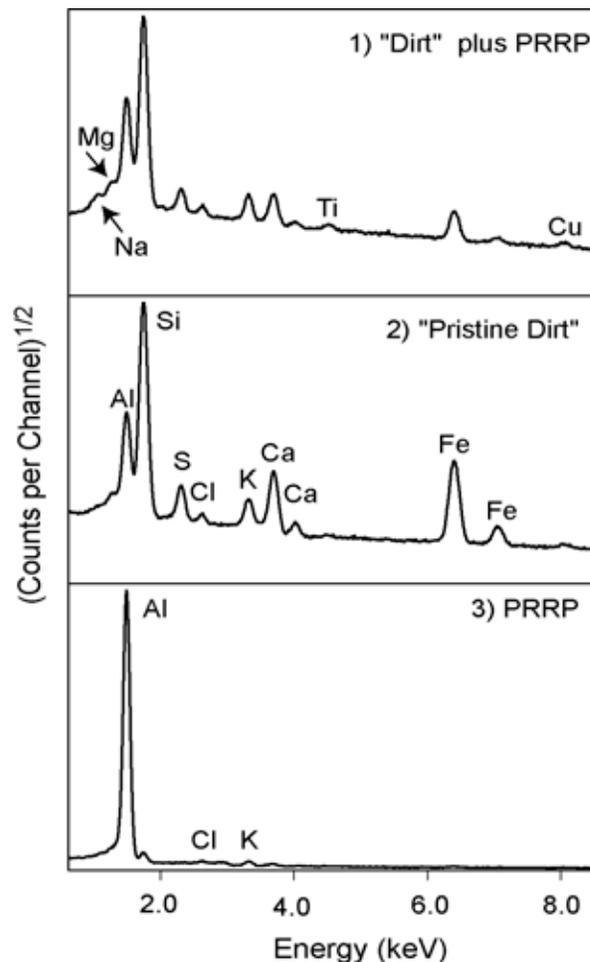


Figure 2. A collection of three composite spectra taken from samples pyrotechnic residues commingled with soil.

standard micro-analytical chemistry would reveal the presence of a trace amount of unreacted perchlorate ion from the flash powder, it seems unlikely that a statistically significant excess presence of aluminum ions would be found. To the contrary, by using the morphological criteria for PRRP, the presence of aluminum in the PRRP is unambiguous. This is presented as the third spectrum labeled "3) PRRP", which is a composite spectrum of 16 such particles.

Only an aluminum peak (from  $\text{Al}_2\text{O}_3$ ) from the flash powder reaction products is readily seen in the PRRP spectra. Potassium and chlorine peaks (from the KCl reaction product) are only weakly present in the composite spectrum. In this case, the virtual absence of the potassium and chlorine peaks is a result of the condensation

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## Applications for Pyrotechnic Reaction Residue Particle Analysis.... Continued

*(Continued from page 3)*

(sublimation) temperature of potassium chloride being moderate (1500 °C) compared with the high condensation (boiling) temperature of aluminum oxide (2980 °C). The effect is to cause the potassium chloride to tend to preferentially condense onto the surface of the relatively cool dirt particles that were mobilized as a result of the explosion. A thorough discussion of this phenomenon is beyond the scope of this paper, but this does help to make the point that a knowledge of the expected products of pyrotechnic reactions, and the physical and chemical properties of those products, can be important in 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 the two techniques can be expected to produce fairly unambiguous results.

### PRRP Example 2: Use History

A pyrotechnic accident was thought to have been caused when an inappropriately powerful charge was exploded within a reusable stage appliance. The appliance was made using approximately a one foot length of 4-inch steel tubing having a 1/4-inch wall, welded to a steel base plate for stability (see Figure 3 for its basic construction). This type of appliance can be used to safely produce both an audible and visual effect when an appropriately sized and constructed pyrotechnic charge is exploded within it. In this case, the appliance was held as evidence, and several months after the incident the inside surface of the device was sampled and analyzed for PRRP. The result was that approximately equal numbers of two fairly distinct types of PRRP were found to be present. Their EDS spectra (composites from 8 particles each) are shown in Figure 4. There was a fairly consistent difference in the ratio of magnesium to aluminum between the two types of PRRP. However, the more notable difference was that one type of PRRP had an abundance of strontium present with relatively little titanium, while the other had no strontium and an abundance of titanium. It is important to note that less than 10% of the PRRP were found to contain roughly comparable quantities of both titanium and strontium, and for those PRRP the amount of both strontium and titanium were both relatively low.

Based on witness accounts and post-incident photography, it had been theorized that the stage appliance in question had been loaded with an inappropriately powerful ex-

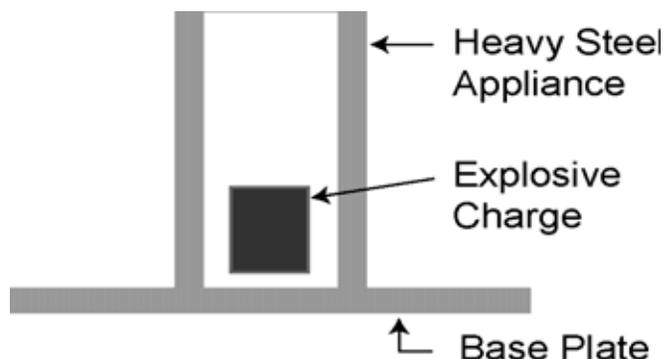


Figure 3. Illustration of the basic construction of the reusable stage appliance.

plosive charge rather than what was normally used in it. The identification of two distinctly different PRRP 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 producing substantial amounts of both titanium and strontium residues (in addition to the magnesium and aluminum), then the PRRP would have all been somewhat similar in composition, with the vast majority of PRRP containing substantial quantities of both titanium and strontium.

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

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

### PRRP Example 3: Incident Chronology

When an explosion occurs, if it is sufficiently violent 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 charges were exploded within less than 0.2 second of one another. One of the two explosive charges is seen close-up in the upper photograph of Figure 5, 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 ounce).

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## Applications for Pyrotechnic Reaction Residue Particle Analysis.... Continued

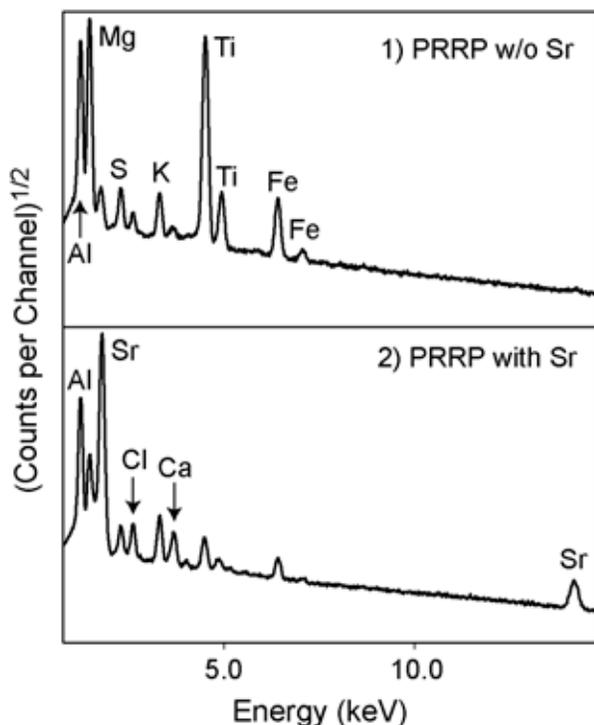


Figure 4. Two composite spectra taken from a stage appliance whose misuse was thought to have caused an accident.

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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 fuel rich flash powder (50% strontium nitrate and 50% magnesium). The distance between the two charges was approximately 0.5 m (20 inches). The demonstration was conducted in a research bay approximately 4 by 4 by 4 m (12 × 12 × 12 feet) in size, and configured such that one wall was completely removed and open to the outside environment. After exploding the two charges, so as not to provide too great an opportunity for additional PRRP deposition onto items within the room, the room was cleared of smoke using a large exhaust fan.

Once the air had cleared, fragments of the containment vessels (35mm-film cans, one made from black polyethylene and the other made from uncolored polyethylene) were collected and sampled for PRRP. Each PRRP specimen was taken from either the inside or outside surface of a containment vessel fragment. (The inside or outside orientation of the fragments was determined by their curvature, and which

of the two vessels produced the fragment was determined by their natural or black plastic color.) Thus a total of four types of specimens from the containment vessels were prepared. Also sampled was the midpoint of the light colored collecting surface seen in Figure 5 extending between the two steel blocks to which the explosive charges were attached. (The mostly white strip of collecting material was 1/8-inch tileboard. The collecting surface had been cleaned prior to the test. The pattern of small random spots seen in the upper photo of Figure 5, had been permanently imprinted within the surface coating by the manufacturer.) The midpoint PRRP specimen was prepared by contact using a conductive carbon dot.

In the analysis of the PRRP on the sampled items, it was obvious that two chemically different pyrotechnic explosives were involved. Thus, following somewhat the same scheme as in the “Use History” example above, PRRP and their spectra were divided into general categories. The composite spectra in Figure 6 are representative of the

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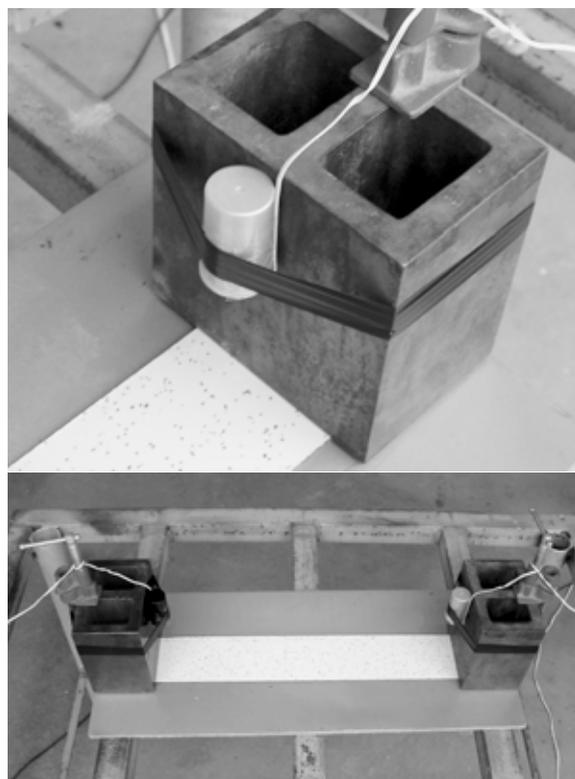


Figure 5. Photographs showing the setup of a demonstration using PRRP to reveal details of the course of an explosive event.

## Applications for Pyrotechnic Reaction Residue Particle Analysis.... Continued

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categories and are labeled to indicate their nature. These categories are “1) Pure Fireworks Flash”, “2) Mostly Fireworks Flash”, “3) Mostly Theatrical Concussion”, and “4) Pure Theatrical Concussion”. In this brief demonstration, only 25 PRRP from each of 5 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 PRRP found on the inner surfaces of the two containment vessels were radically different. In one case (black plastic), approximately 85 percent of the PRRP fell into category 4, having only the components of the theatrical concussion powder. In the other case (uncolored plastic), 70 percent of the PRRP fell into category 1, having only the components of the fireworks flash powder. Accordingly, it is obvious that the black film-can contained the theatrical concussion powder and the uncolored film-can contained the fireworks flash powder. Had the pyrotechnic explosives both been a single mixture of all of the ingredients, the normal variations in the ratios of the elements might account for a few particles producing relatively pure spectra, but this would amount to a few percent at most, and they would have been randomly distributed between the two specimens.

In Table 1, the column labeled “Surface Total” is the total of the PRRP particles collected on the exteriors of the two containment vessels plus those from the midpoint of the collecting surface. Approximately 60 percent of the PRRP fell into the two “Pure” categories. 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 occurred within approximately 0.2 second.) Had the two explosions occurred at the same precise instant, there would have been more complete mixing of their respective fire-balls, and it is thought that fewer of the PRRP would have contained a mixture of the components of the two pyrotechnic explosives. Had the two explosions been more widely separated in time, it is thought that the number of mixed composition PRRP would have been even greater.

The PRRP on the outside surfaces of both film-cans is predominantly of the “pure flash powder” type. Further, the inside surfaces of the uncolored plastic film-can have no “pure theatrical concussion” PRRP. These observations are consistent with the fireworks flash powder exploding first.

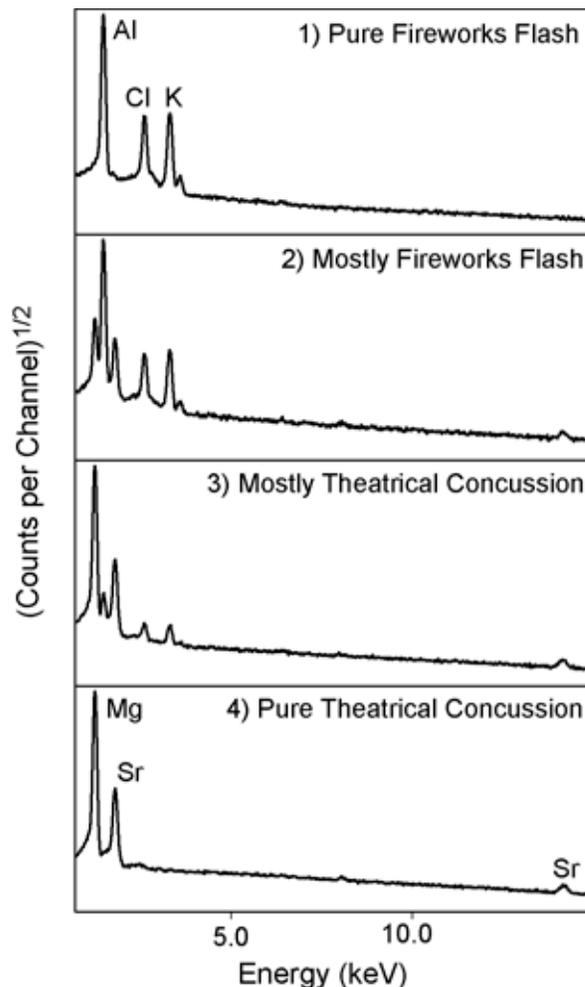


Figure 6. A series of four composite spectra representing the four categories of PRRP used in this analysis.

Based on this analysis, it can reasonably be concluded that:

- Two different types of pyrotechnic explosives were involved;
- The explosions occurred at nearly the same time but not simultaneously; and
- The fireworks flash powder exploded first.

### Conclusion

PRRP analysis, which only identifies the chemical elements present in pyrotechnic reaction residue, will not supplant micro-analytical chemistry, which has the ability

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## Applications for Pyrotechnic Reaction Residue Particle Analysis.... Continued

(Continued from page 6)

to identify more complex chemical species. However, there are times when PRRP analysis can provide information that is a useful adjunct to standard micro-analytical chemistry. There will also be occasions when PRRP analysis can provide information that is beyond the reach of standard chemistry. Accordingly, a more extensive knowledge of PRRP analytical techniques may be of use to the forensic analyst.

**Table 1. Numbers of Each of the Four Types of PRRP on Items Sampled.**

PRRP Category	Black Plastic		Clear Plastic		Surface	
	Outside	Inside	Outside	Inside	Midpoint	Total <sup>(a)</sup>
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

<sup>(a)</sup> This is the total of PRRP types for the initially exposed surfaces (i.e., the outsides of the two containment vessels plus the midpoint of the collecting surface).

### References

1. K. L. & B. J. Kosanke and R. C. Dujay, "Identification of Pyrotechnic Reaction Residue Particles". *IAMA Newsletter*, Vol. 3, No. 2002.
2. K. L. & B. J. Kosanke and R. C. Dujay, "Pyrotechnic Reaction Residue Particle Identification by SEM / EDS", *Journal of Pyrotechnics*, No. 13 (2001) pp 40–53.
3. K. L. Kosanke, R. C. Dujay, and B. K. Kosanke, "Characterization of Pyrotechnic Reaction Residue Particles by SEM/EDS", *Journal of Forensic Science*, Vol. 48, No. 3 ( 2003) pp 531–537.

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## Macro Imaging with a Digital Camera: Examination of a .22 Revolver – By Bryan Burnett

Digital cameras are supplanting film cameras in many areas of science. Investigators are now using these cameras for crime and accident scene imaging. Digital cameras attached to microscopes are now found in practically every laboratory in the US. Even in macro imaging applications, which is the main focus of this paper, these cameras will likely replace film cameras.

In a recent article, Burnett and Blaauw (2003) explored the capabilities of digital cameras for macro imaging applications. They found that the high resolution (5.8 megapixel), Peltier cooled Pixera 600CL camera is capable of taking images with extraordinary depth of field. Indeed, the depth of field that the Pixera 600CL with a good macro zoom lens produces, rivals or even exceeds that of the scanning electron microscope. And how is this possible? It is mainly due to the camera's exceptionally sensitive charged coupled device (CCD). The sensitivity that can be achieved by the CCD in this camera is so great, that images can be taken with the attached macro zoom lens stopped down past  $f\ 44$ . Along with this extraordinarily high  $f$ -stop comes an extraordinarily high depth of field.

There are actually two purposes for this paper. First, and foremost, I wish to show the remarkable capabilities for achieving a high depth of field in a digital camera and second, to locate apparent gunshot residue (GSR) deposits on a revolver.

A Smith and Wesson (S&W) Model 18 .22 revolver (Fig. 1) was used to demonstrate the depth of field capabilities of the Pixera 600CL coupled with a Computar MLH-10X ( $f\ 5.6$ ) or a Optem 18-108 ( $f\ 2.5$ ) macro zoom lens. The Pixera 600CL sensitivity was set to ISO 200. Generally, for images



Figure 1

to about 3X, the Computar lens was used (Figs. 1, 2 and 3A). For magnifications greater than 3X or so, the Optem macro zoom lens was used (Figs. 4 through 7). Illumination of the subject was with a 60 Watt-equivalent fluorescent bulb (high intensity lighting should be avoided because such lighting produces image hot spots). Most images were taken at greater than  $f\ 44$  with exposure times usually exceeding 1 second.

Figure 2



The image shown in Fig. 3A demonstrates the extraordinary depth of field that this camera system can achieve. Details on the muzzle, the rifling in the barrel and the firing pin of the .22 revolver can be seen. Figure 3B shows the distance that the Pixera 600CL/Computar MLH-10X macro zoom was from the .22 revolver when the image (Fig. 3A) was taken. Color loss or other distortion is not an issue with images taken at this high  $f$ -stop.

Apparent gunshot residue deposits were found at various locations on the breech face (Fig. 4, discoloration flanked by arrows) and the cylinder (Figs. 5, and 7). Probable remnants of gunpowder were found along the edge of the cylinder chambers that are closest to the breech face (Figs. 5 and 6: brown particles at arrows). In Fig. 6, the lighting was such that GSR debris shows as bright specks in the chamber.

Figure 4 reveals that the rifling in this revolver does not extend to the breech face. Instead in this area of the bore there are file-like ridges that run perpendicular to the axis of

(Continued on page 10)

# Macro Imaging with a Digital Camera: Examination of a .22 Revolver ...Continued

Figures 3A and 3B

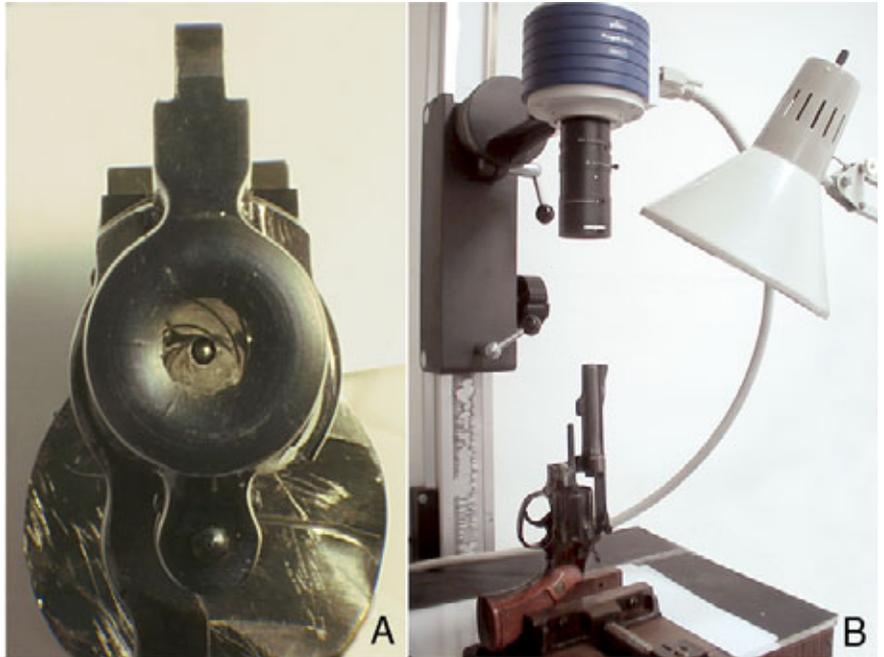


Figure 4

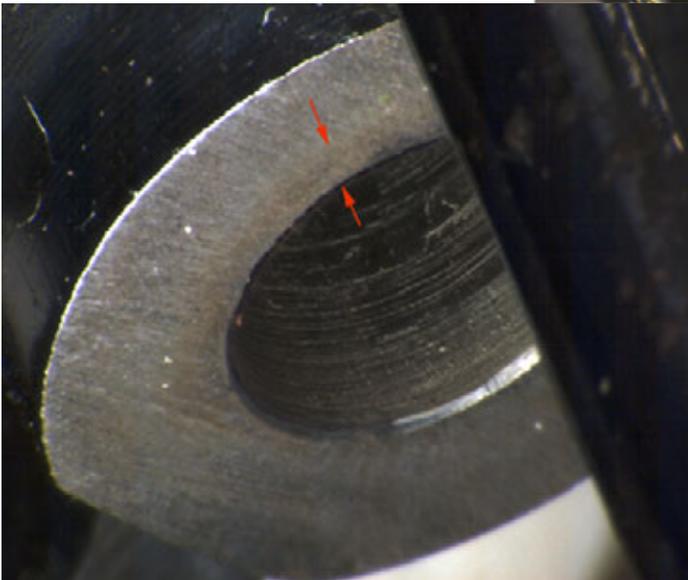
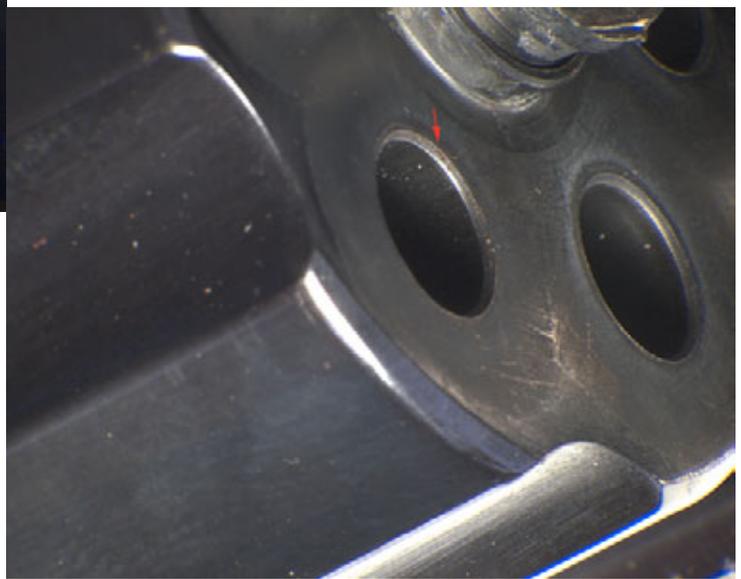


Figure 5



## Macro Imaging with a Digital Camera: Examination of a .22 Revolver ...Continued

Figure 6



Figure 7



(Continued from page 8)

the barrel. Gunshot residue is likely to deposit in these groves.

The nose of a Remington brass-coated bullet is seen in the right chamber in Fig. 7. Above this bullet relatively large GSR particles can be seen. The chamber on the left is empty and is backlit with a fiber optic light. There is a heavy ribbon-like deposit of apparent GSR on the wall of the chamber (Fig.7, arrow). This material was likely deposited following the departure of each bullet while it was within millimeters of the casing during the firing process.

The Pixera 600CL digital camera coupled with either the Computar or Optem macro zoom lens presents a broad range of low magnifications with large depth of field. This system essentially allows for much better documentation of small three-dimensional evidence as well as the potential for providing a more precise record of collection areas on evidence. The stereo microscope, as an intermediate optical system for documenting sampling of evidence, in many cases can now be eliminated - it just cannot match the documentation potential of the system described in this paper. Dr. Jozef Lebedzik noted, upon reading an earlier version of this paper, that this Pixera camera /macro zoom lens system fills "the gap between macro photography/viewing and SEM."

I wish to thank Jozef Lebedzik and Steven Blaauw for their valuable comments on previous versions of this manuscript.

### Reference

Burnett, B.R. and Blaauw, S. 2003. Macro imaging with digital cameras. (in press) *Microscopy Today*. July/August issue.

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## Clear Adhesive Tape Analysis Using Polarizing Light Techniques: The “Megascop”- By Brad Rogers

### Introduction

Polymer analysis often entails a complex and quite lengthy procedure. For example, clear adhesive tape contains at minimum the polymer backing and adhesive. Each of these layers may be analyzed by several techniques, including polarizing light microscopy (PLM), Fourier Transform Infrared Microscopy (FTIR) and perhaps pyrolysis with the aid of a Gas Chromatograph with a Mass Selective Detector. Instruments such as these are expensive and through lean budget periods, most organizations are forced to pass over laboratory instrumentation purchases. A simple and very economical method used to compare clear adhesive tape or other clear polymer sheets, such as plastic sandwich type baggies, is the Polarized Light Megascop. The ‘megascop’ utilizes principles established with fiber analysis and polarized light microscopy, simply on a larger scale.

were used to provide elevation for this setup (See Figure 2). Samples are then inserted between the polarizer and analyzer and rotated to observe differences.

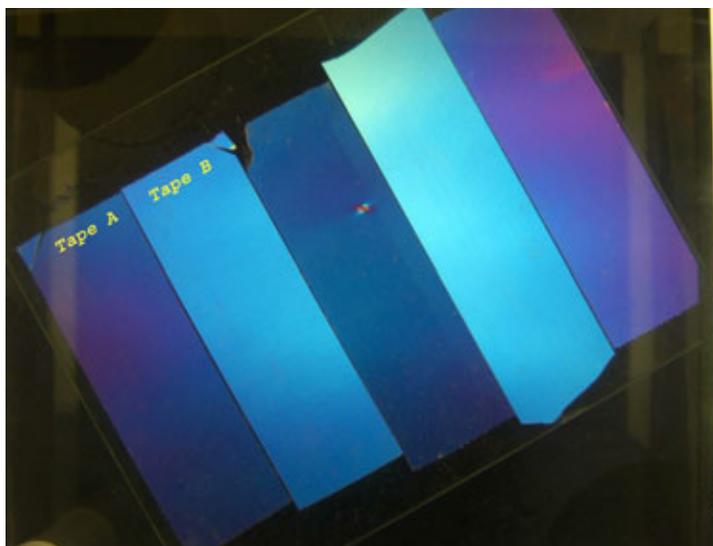


Figure 1: Interference colors of five (5) pieces of Scotch 3M 3750 tape collected from five different units within the author's laboratory.

### Megascop Construction [1]

The megascop is comprised of a plate of frosted glass (for light dispersion) secured above a light source. It is important that the frosted glass be placed high enough to disperse the light completely without viewing the individual bulbs through the glass. Additionally, two large polarizers (polarizer and analyzer) are required for the megascop. Each is made by placing one sheet of 12” x 12” polarizing film (full wave) between two glass plates. To prevent injury, the plate edges may be covered with black electrical tape. The polarizer is placed on top of the frosted glass and the analyzer is elevated and rotated 90 degrees relative to the lower polarizer; film canisters

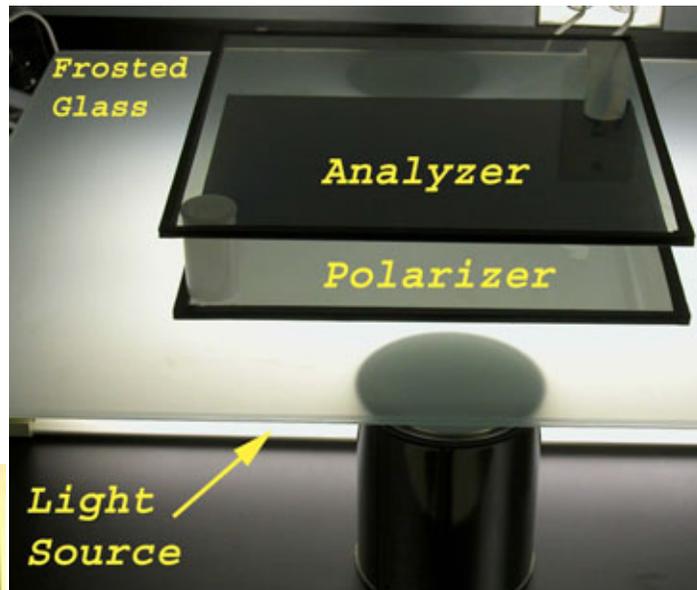


Figure 2: Megascop Setup

### Infrared Analysis

Fourier Transform Infrared (FTIR) analysis is a mainstay in forensic polymer analysis and provides more specific chemical information about polymer composition than optical microscopy [2]. For instance, the sub-generic classes of nylon 6 and nylon 6,6 may be distinguished with FTIR (Figure 3) whereas differentiation with other techniques, such as Polarized Light Microscopy, melting point and solubilities, are less discriminating.

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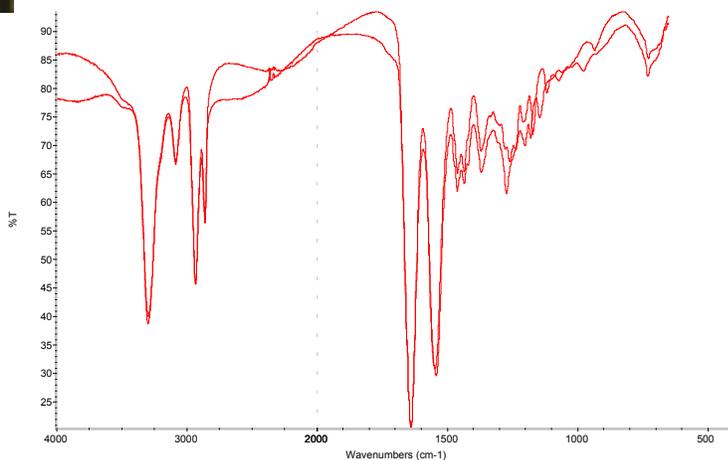


Figure 3: Infrared spectra of Nylon 6 and Nylon 6,6.

## Clear Adhesive Tape Analysis Using Polarizing Light Techniques: The “Megascope”...Continued

(Continued on page 11)

In this study, FTIR analysis was performed on several pieces of tape collected from different sections within the author’s laboratory. All tape samples collected were from standard rolls of two inch 3M 3750 tape. Figures 4 & 5, showing the FTIR spectra of adhesive and polymer backing are virtually indistinguishable.

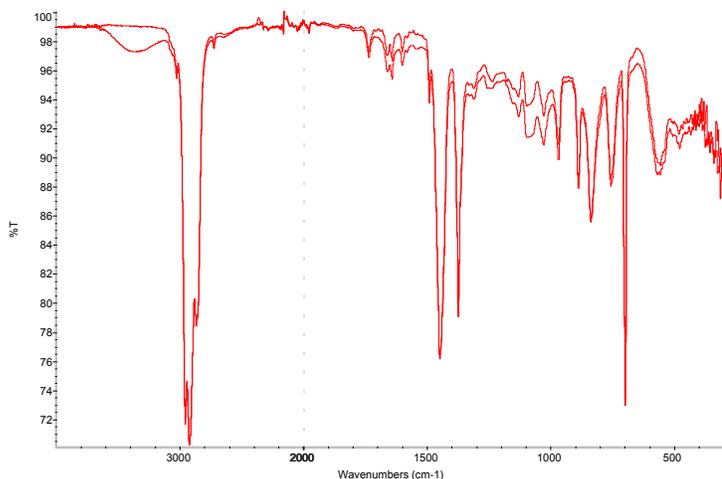


Figure 4: Infrared spectra of adhesive from 3M 3750 tape (Figure 1A & 1B).

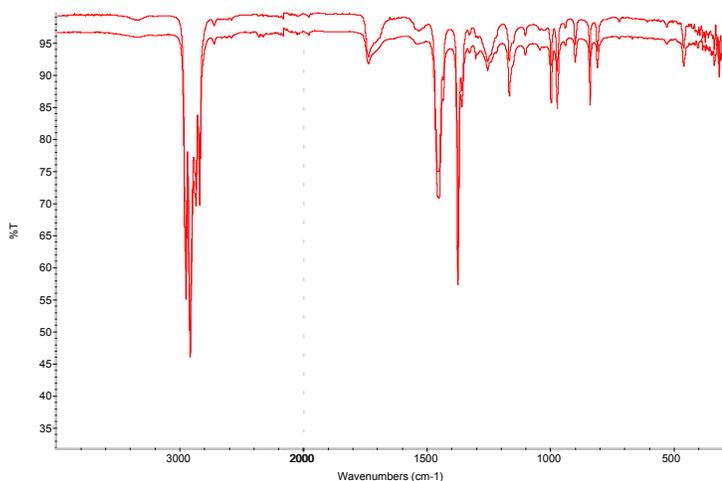


Figure 5: Infrared spectra of polymer backing from 3M 3750 tape (Figure 1A & 1B).

Although infrared analysis is chemically specific, the optical characteristics obtained through the use of polarized light are invaluable. As stated by Palenik [3], there are four basic optical properties (refractive index, isotropic refractive index, birefringence and sign of elongation) that can be determined for any fiber. In effect, these sections of tape are analyzed with the Polarized Light Megascope like fibers are analyzed with the Polarized Light Microscope.

### Megascope Analysis

The megascope has a practical association to polarized light microscopy of fibers, but on a macroscopic scale. The Polarized Light Microscope (PLM) is used to determine the generic class of synthetic fibers through observations of interference colors and the calculation of birefringence. The interference colors are seen when comparing clear adhesive tapes with the megascope as well.

Interference colors differ widely on some pieces of tape (Figure 1), while they are similar in other pieces of tape. The interference colors of the different tapes may yield important information, but of equal if not more importance is the extinction point/angle. Plane polarized light is said to vibrate parallel to one particular direction [4]. If the tape is oriented so that one of its principle refractive indices is parallel to the vibrational direction of the polarizer, then all emerging light is absorbed by the analyzer, which is rotated 90 degrees to the polarizer [5], thus producing a black image (the extinction point). Extinction points of fibers, for instance, coincide with the length of the fiber, whereas extinction points of clear tapes do not necessarily coincide with the length of the tape. This extinction point is observed using the megascope. The angle between the extinction point and the length of the tape may be measured. By viewing the tapes side-by-side and comparing the extinction points, real differences may be viewed in the tape analysis.

Fiber birefringence may be calculated by plotting the interference color and fiber thickness on a Michel-Levy chart. Minor diameter variations in two fibers can result in excluding one sample from a known source of fibers. Variations in thickness of the fiber will result in slight variations of the interference color. Some thickness variation can also be observed on a single strip of tape, with a corresponding change in interference color. However, minor thickness variations in tape analysis may not be as crucial, as noted above, as differing extinction angles.

One question then, is do thickness variations in tape alter extinction angles? In Figure 6A, five strips of tape (taken from the same roll, collected at ten feet intervals) were layered in the same direction. The extinction point of all five pieces coincide at the same position. Figure 6B shows an induced difference of extinction. Three pieces of tape were placed on top of each other at differing angles. Extinction of all three pieces is never observed at any rotation.

Sequencing of plastic bags is also possible. Great differences may occur between bag manufacturers, while within the same box of sandwich bags, the order of bags as they were cut from the roller may be observed by comparing manufacturing marks (Figure 7) [6].

(Continued on page 13)

## Clear Adhesive Tape Analysis Using Polarizing Light Techniques: The “Megascop”...Continued

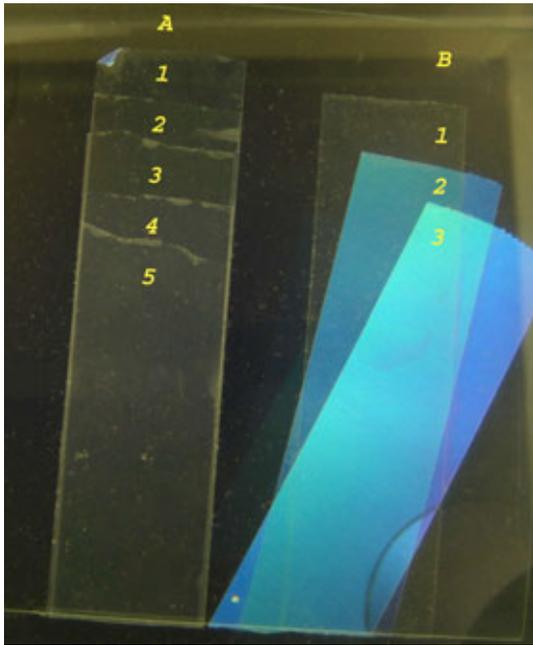


Figure 6: Comparison of thickness and extinction.

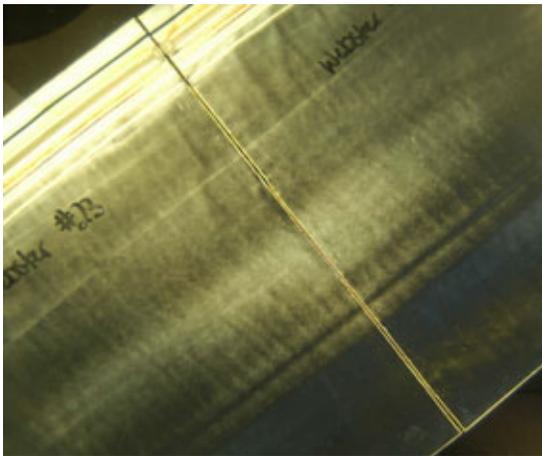


Figure 7: Sequencing marks observed in plastic sandwich type baggie comparison.

(Continued on page 12)

### Conclusion

Fiber and clear adhesive tape analysis share similar methods of forensic analysis. Polarized light techniques are used to identify and/or characterize the polymer in both situations. Whereas with fiber analysis, the birefringence is sought for identification and the extinction point may be less interesting, observation of the extinction angle with a megascop is very interesting. These observations form a direct relationship to the manufacturing marks [7]. Megascop analysis is inexpensive yet the resulting discrimination, is in some cases, superb

when compared to more expensive equipment. The comparison of spectra in Figures 4 & 5 shows little difference, but by using the megascop, the two tapes are easily differentiated (Tapes A & B, Figure 1). This technique is simple and enlightening yet sensitive and very meaningful!

### Acknowledgement

Mel Hett, formerly of the Oklahoma State Bureau of Investigation, is one of the initial developers of the “megascop”. Mel’s knowledge and expertise in trace analysis is sincerely missed.

### References

1. Hett, M.R., A Case Involving Comparison of Plastic Trash Bags, Presented at Southwestern Association of Forensic Scientists Meeting, Spring 1980.
2. Tungol, M.W., Bartick, E.G., Montaser, A., Forensic Examination of Synthetic Textile Fibers by Microscopic Infrared Spectrometry, In: Practical Guide to Infrared Microspectrometry. Humecki, H.J. editor. 1995. 245-285.
3. Palenik, S.J., Microscopical Examination of Fibers, In: Forensic Examination of Fibers. Robertson, J., Grieve, M. editors. 1999. 153-177.
4. Bloss, F.D., Optical Crystallography, Mineralogical Society of America. 1999.
5. McCrone, W.C., McCrone, L.B., Delly, J.G., Polarized Light Microscopy, McCrone Research Institute. 1984.
6. von Bremen, U.G., Blunt, L.K.R. Physical Comparison of Plastic Garbage Bags and Sandwich Bags, Journal of Forensic Sciences, JFSCA, V28, No. 3 July 1983, 644-654.
7. Smith, Jenny. PLM Examinations of Clear Packing Tape and Determination of Monoaxial or Biaxial Orientation. ITER/MICRO-2002 Abstract. The MICROSCOPE (2002) 50.

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## Nose or Base Strike— By Bryan Burnett

During a recent homicide trial in San Diego, testimony was given concerning the ballistics of the fatal .22 bullet. There was evidence of a second strike on a portion of bullet's mushroomed surface (Fig. 1A). It was alleged that the missile was tumbling when it struck the second object and that this second strike was from the direction of the base toward the nose of the bullet. In this case, the direction of the bullet impact on the second surface had important implications for the crime scene reconstruction. As can be seen in Fig. 1A, the direction of the second impact is at about a 45° angle from the central axis of the bullet and there is a peculiar direction change of the tool marks in a portion of the strike zone. A careful examination of the bullet's second impact surface suggests that the direction of this strike was toward the base of the bullet (i.e., from left to right in Fig. 1A).

This case was reported previously (1). In that paper, evidence was provided which supported a scenario that the copper-coated .22 bullet went through window glass prior to hitting the victim. Both the mushrooming of the bullet as well as the second impact surface (Fig. 1A) were likely the result of the bullet's interaction with window glass (1). Regardless of the identity of the second impact surface, a distinctive feature associated with the bullet's second impact defect attests to the orientation of the bullet when it struck that second surface.

Figure 1B is an enlargement of a portion of the second strike area shown on the victim's bullet in Fig. 1A. In this region of the victim's bullet are a series of apparent overlapping laminae. The surface of each lamina has tool-like

*(Continued on page 15)*

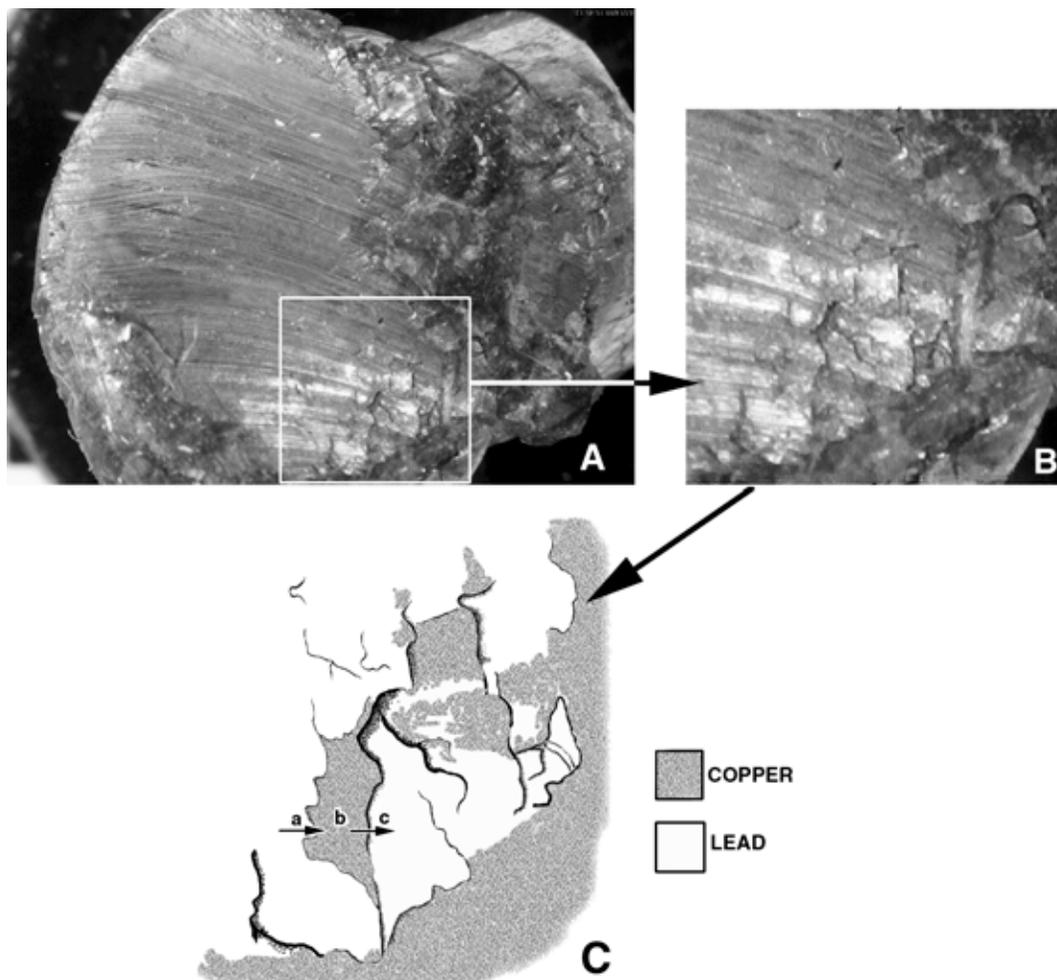


Figure 1. The questioned .22 bullet. A: The second impact area showing the region (box) of apparent lamina. B: An enlargement of the box area in A. C: Drawing of the region showing the distribution of copper (uniform stipple) from the .22 bullet coating.

## Nose or Base Strike....Continued

*(Continued from page 14)*

marks and in this sequence of the laminal surfaces are remnants of the copper coating from the .22 bullet surface. Figure 1C is a drawing of this area which maps the distribution of copper (uniform stippled areas). In one part of this area, the overlapping lamina-like surfaces are particularly well defined. Copper alternates with lead on the laminal surfaces. Arrows in Fig. 1C indicate the direction of the overlap. The surface of layer "a," overlaps surface "b." The surface area of "b" is copper coated. Surface "b" overlaps surface "c." The laminal surfaces marked "a" and "c" do not have copper associated.

A number of .22 bullets that were mushroomed by being fired through window glass (and caught in a cotton box) were clamped on their bases in a ViseGrip™ pliers and struck, nose leading, at approximately 30° angle on a smooth concrete surface. The result of one of these simulated bullet strikes is shown in Fig. 2. The bullet (Fig. 2A) showed laminal structures on the trailing edge of the impact zone on the bullet (Fig. 2B). The front margin of the strike area (Fig. 2A) is uniform with no laminae.

The formation of trailing-edge laminae on the bullet is likely associated with a lead bullet ricochet. As shown by diagram (Fig. 3A), the bullet hit the object at an angle. The forward force of the bullet impact tends to push the bullet's nose into the object's surface with the base of the bullet rotating away from that object. The lead in contact with the strike surface flows toward the base of the bullet as it engages that surface. Small wavelets of lead that are apparently created during this process flow in the opposite direction of the bullet travel while the bullet engages the surface. As the bullet base rotates on this surface more wave-

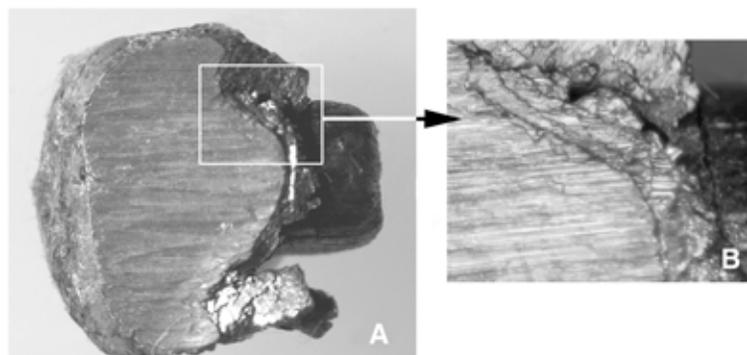


Figure 2. Test bullet that was hit at approximately a 30° angle on a concrete surface. A: The .22 bullet showing a region of laminal formation (box). B: An enlargement of the boxed area on Fig. 2A showing the distinctive laminae on the trailing edge of the strike zone.

lets are created. Thus, the trailing edge of the bullet impact area is left with the impression of laminae as shown in Fig. 2B.

The presence of laminal features on the margin of the strike zone of a bullet is an indicator the direction of that strike. When the second impact zone was created, the .22 bullet in question likely struck near its nose.

### Reference

1. Burnett, B.R. 2000. A shot through the window. J Forensic Sci 2001;46(2)379-385.

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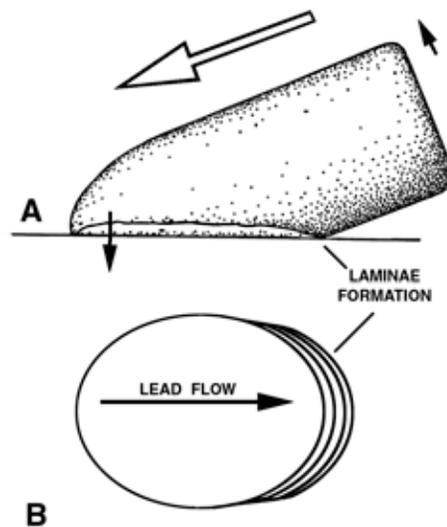
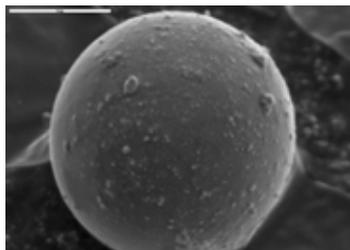


Figure 3. A: Diagram of a lead bullet in the process of a ricochet strike on a surface. White arrow: the direction of the strike. Black arrows: direction of rotation of the bullet. B: Diagram of the bullet's contact surface showing the probable lead flow during contact with a surface.

### Unusual Crystallization- A Case Study



Lead, Barium and Antimony  
IAMA Collection

Several years ago during a routine case analysis of primer gunshot residue (P-GSR), I came across several unusual crystals on the surface of the P-GSR stubs. Further inspection of the crystals by energy dispersive x-rays analysis (EDX) revealed that the crystal composition was identified as lead and potassium chloride (Figures 1 and 2). In addition to finding lead and potassium chloride crystals, P-GSR particles consisting of lead, barium and antimony were also detected on the P-GSR stubs collected from the hands of the subject.

The only information obtained regarding specifics of the case reveal that the individual was apprehended shortly after a shooting incident and the subjects hands were wet prior to collection. Unfortunately, No additional information was obtained.

#### Conclusion

Additional information into the unusual crystal formation was well documented by J. Goldstein et al<sup>1</sup>. and states that the crystal formation can be caused by "extreme environments". It is this authors speculation that the crystal formation was initiated by the slow vacuum increase when the wet sample stubs were placed into the sputter coater (~10<sup>-8</sup> torr) and then continued when placed into the SEM (~10<sup>-7</sup> torr). The reasons for the crystal formation or source of wetness of the hands are still unclear. It is possible that the P-GSR sampling stubs were wet from sweat or water. Further research is needed to duplicate the phenomenon.

#### Reference

1. J. Goldstein, D. Newbury et al., *Scanning Electron Microscopy and X-Ray Microanalysis*, Second Edition. Plenum Press, New York 1994. (Pg. 259).

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**In its famous paradox, the equation of money and excrement, psychoanalysis becomes the first science to state what common sense and the poets have long known - that the essence of money is in its absolute worthlessness.**

*Norman O. Brown*

#### Instrumentation

The P-GSR sampling stubs were sputter coated with carbon prior to analysis. The carbon coated sampling stubs were then placed into an automated stage JEOL 5800 scanning electron microscope (SEM) equipped with an Oxford energy dispersive x-ray detector (EDX) using Oxford ISIS software.

A Tri-Tech, Inc. P-GSR collection kit containing four sampling stubs labeled "Right Back, Right Palm, Left Back and Left Palm" were used on the subjects hands.

Figure 1

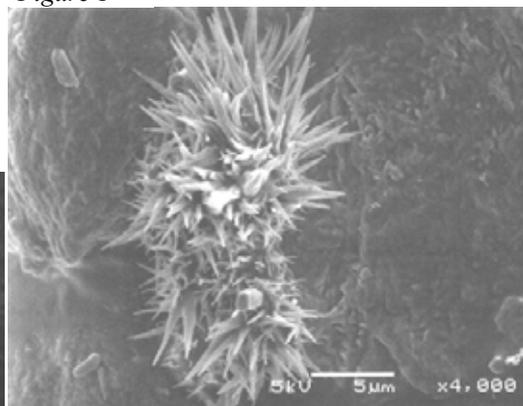
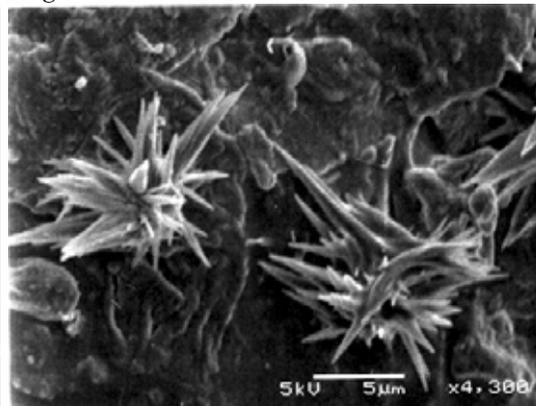
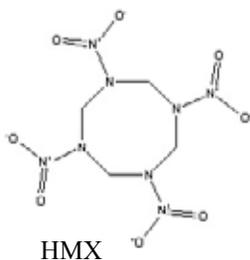


Figure 2

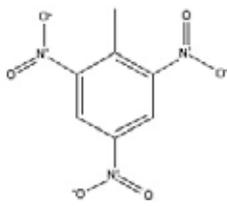


Lead, Barium and Antimony  
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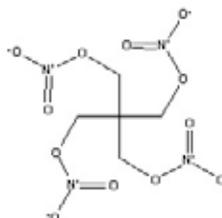
## Terrorist/Terrorism Investigation Software



HMX



TNT



PETN

The latest innovation from Forensic Products and Services, Inc.

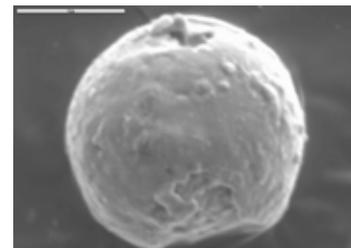
The phone rings, you pick it up and someone says "I found these chemicals, but I don't know what are they used for??" Now the search starts. Reference book after reference book. Hours upon hours of time looking through these reference books looking for these chemicals trying to figure out what they can be used for. Or try this one: You are on a Hazmat call out and you find several chemicals hidden in suspects home. Your cell phone is not working, but you still need to figure what these chemicals can be used for.

Introducing our new, revolutionary software designed to help law enforcement with **bombing** or **terrorist investigations**. The software is made so that you do not have to spend valuable hours researching information on the materials needed to make **explosives** or **chemical weapons**. No more valuable time lost looking-up information in book after book after book.

This software is specifically made for use in identifying chemicals that can be used to make explosives or chemical weapons. This software is simple and easy to use. Made to work on a laptop or a desktop computer, it contains over 450 various formulations of explosives and chemical weapons. Everything from the ordinary to the ones most commonly used by terrorists. You have the ability to search by common name, synonym, ingredients, use, etc. Not only will it tell you what the chemical can be used to make, but it will also identify the other chemicals necessary to complete the manufacturing process.

This software utilizes a database developed by a forensic chemist with over 24 years of experience in explosives analysis and who is currently one of the main instructors for the Weapons of Mass Destruction training program at the Transportation Technology Center, Inc. Emergency Response Training Center northeast of Pueblo. This facility was designated by the Colorado State Legislature as the official State of Colorado Domestic Preparedness training facility.

A free demo can be downloaded be visiting <http://www.cripkit.com/products/ttis.html>



Lead, Barium and Antimony  
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**Wherever he steps, whatever he touches, whatever he leaves even unconsciously, will serve as silent witness against him.**

**Not only his fingerprints or his footprints, but his hair, the fibers from his clothes, the glass he breaks, the tool marks he leaves, the paint he scratches, the blood or semen he leaves or collects—all of these things and more bear mute witness against him. This is evidence that does not forget. It is not confused by the excitement of the moment. It is not absent because human witnesses are. It cannot perjure itself. It cannot be wholly absent. Only its interpretation can error. Only human failure to find it, study and understand it, can diminish its value.**

## Acknowledgements

*We have added this section of the newsletter to show our appreciation for those individuals who have contributed much of their time and energy in the success of this newsletter.*

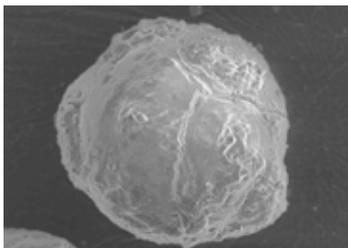
**Brad Rogers:** *Oklahoma State Bureau of Investigation.* Thank you Brad for your tireless efforts in rounding up articles for the newsletter.

**Jamie Crippin:** *Western Law enforcement Training Center.* Thanks Jamie for auditing the IAMA account.

*Crime Investigation, second edition, Paul L. Kirk (deceased), edited by John I. Thornton (1974), p.2. (Quoted also in "Footwear Impression Evidence" by William J. Bodziak, at page 1)*

## From The Bench

*"FROM THE BENCH" is a section of the newsletter intended to provoke conversation, address new concerns, express opinions and, hopefully, provide insight into the new and old. Therefore, the opinions of the contributors are not necessarily those of the editors or other contributors.*



Lead, Barium and Antimony  
IAMA Collection

"What is Forensic Science?" a simple question we have all had to address at some point in our careers. Recently, media exposure has placed much attention in the field of Forensic Science. With this newly found popularity, I find it interesting how quickly the traditional natural sciences have embraced Forensic Science as a long time integral part of their studies. Traditional scientists seem to use the term "Forensics" as a loose fitting hat that can be worn, removed or passed around at a whim. Of course we all know better...

As I flick through the channels on the television, I find myself frequently immersed in those fascinating educational broadcasts. It may be the dynasty of the Egyptian Pharaohs, the sinking of the Titanic, or the life of a Gladiator, and regardless of the episode, the term "Forensics" always appears and is used by the "scientific authorities" as an important tool in their investigative findings. It seems to me that "Forensics" is an interesting term to be applied to ancient history. I guess it would be better to ask the question, "What is Forensics (plural)?"

Well, according to the Merriam-Webster Dictionary:

Forensic (singular) is a term that was coined in 1659 and is most commonly used as an adjective<sup>1</sup> to describe:

**1** : belonging to, used in, or suitable to courts of judicature or to public discussion and debate

**2** : relating to or dealing with the application of scientific knowledge to legal problems  
<forensic medicine> <forensic science> <forensic pathologist> <forensic experts>.

Forensic, when used as a noun<sup>2</sup>, was first coined in 1814 and refers to:

**1** : an argumentative exercise

**2** : plural but singular or plural in construction : the art or study of argumentative discourse .

Although I have not found any specific definition of "Forensics", my assumption based on the application of the term when and how it is most commonly used as a noun, seems to have evolved into another American colloquialism. Or as the British would rightfully say, another American bastardization of the Queen's English. Nevertheless, this new derivation would imply (adjective) the use of scientific knowledge of or used in legal proceedings or in public forum.

Based on this, I can't help but wonder why the dynasty of the Egyptian Pharaohs would need a legal proceeding with judge and jury, or even why this would be an argumentative exercise if it's based upon proven scientific methodologies.

I believe that what my favorite educational television channels are referring to when the scientific authorities use the term "Forensics" is simply that the same established scientific methodology and techniques used in the fields of Forensic Science have been incorporated into developing a plausible explanation of the past events in history. I suppose it's easier just to say "Forensics", besides it sounds better and the masses can relate.

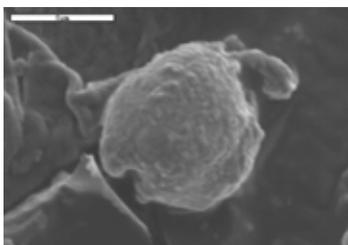
Which just goes to prove that in order for mankind to effectively communicate with one another, we must clearly define our words more specifically. Realistically, it sure would be nice if all of our national funding sources would give as much flexibility to fighting crime as they do to institutions and organizations researching in the name of "Forensics".

<sup>1</sup>adjective: any class of words used to modify a noun or other substantive by limiting, qualifying or specifying.

<sup>2</sup>noun: a word used to name a person, place, thing quality, or action.

**Mastering the lawless  
science of our law,--  
That codeless myriad of  
precedent,  
That wilderness of  
single instances.**

*Aylmer's Field*



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**FYI!** - *A reminder of upcoming events:*

**TWGFEX: the Technical Working Group for Fire and Explosions.  
Third Annual TWGFEX Symposium  
For Fire and Explosion Debris Analysis and Scene Investigation**

November 19th through November 23rd, 2003

TWGFEX is hosting a symposium comprising workshops, lectures, verbal presentations of current research, and discussion sessions. The general theme of the 2003 Symposium is **“Raising the Bar – From Opinions to Facts.”** Topics will be relevant to fire and explosion investigations, at the scene and/or in the laboratory. The symposium will be held at:

Hawthorn Suites at the Orlando International Airport  
7450 Augusta National Drive  
Orlando, FL 32822  
(407) 438-2121  
<http://www.orlandosuites.com>

The TWGFEX Chairs Meeting will be held on November 17th and TWGFEX Business/Committee meetings will be held on November 18th & 19th, preceding the Symposium. Registration information, a call for papers and posters, and vendor information is available below (in Adobe PDF format). For your convenience, registration information can be submitted online at: <http://www.ncfs.org/twgfex/twgfex2003.html>

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**2003 Meeting - Columbus, OH - October 18-24, 2003  
MAFS 2003 FALL MEETING**

Hyatt Regency, Greater Columbus Convention Center, Columbus, Ohio  
Local Arrangements Co-chairs: Jennifer Duvall and Diane Larson  
Program Chair: G. Michele Yezzo  
Sail into Columbus ... and see what there is to discover!  
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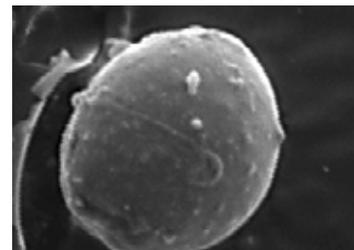
**The Forensic Labs in the Fort Worth Area  
would like to invite you to  
“The City Where the West Begins”  
SWAFS 2003 Training Conference 2003 in  
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November 3rd-6th**

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800-333-3333  
[www.radisson.com/ftworthtx](http://www.radisson.com/ftworthtx)

The conference will be held at the Radisson Plaza Hotel, 20 minutes from DFW Airport, walking distance to The Bass Hall, Barnes and Noble Bookstore, two multi-screen AMC theatres and many other unique shops and restaurants. The Fort Worth Trolley provides easy access to the Historic Stockyards and the Art District.

A nightly room rate of \$80.00/single-\$170.00/quad has been secured. The final agenda will be provided with the registration packet sent to all SWAFS members in the next few months.

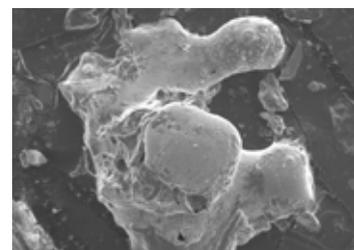
For more information contact: Michelle O’Neal,  
Tarrant County Medical Examiners Office  
817-920-5700 ext 163



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**Every great advance in  
science has issued from  
a new audacity of  
imagination.**

*John Dewey*



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