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International Association for MicroAnalysis

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A NEWSLETTER FOR FORENSIC EXPERTS IN P-GSR ANALYSIS BY SEM/EDX

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Just Another Tool

Mythologies surround the forensic use of scanning electron microscope/energy dipersive x-ray (SEM/EDX) analysis: SEM/EDX analysis is expensive, time consuming, and requires operators who are part technician and part rocket scientist. As a SEM/EDX operator, I am flattered. However, the reality is that what was once an expensive research instrument has become an indispensable, practical tool for public safety - a powerful, mainstream forensic tool - just another tool. SEM/EDX is now a cost effective, client friendly, and reliable addition to the forensic arsenal. We must counter mystery with truth, ignorance with education: SEM/EDX analysis should take its rightful place in the full service crime lab.

Certainly, one benefit is that providing SEM/EDX analysis bestows immediate credibility on a crime lab. As the manager of a moderate-sized, local, forensic laboratory, I have occasionally encountered the supercilious and deprecating attitudes of forensic colleagues from larger labs. The attitudes always change to one of respect when they learn that the laboratory I represent provides SEM/EDX analysis. This newfound respect, while ego building, is predicated on the question "Why does any lab provide SEM/EDX analysis services?" Instead the question should be "Why don't all full service forensic labs have this tool?" Let's examine the facts.

I admit that SEM/EDX analysis is expensive. Our lab spent $216,000 in 1997 for a JEOL 5800, an Oxford Isis, a Hummel carbon evaporator, and a Haskris water-chilling unit. However, with proper maintenance, I expect this equipment to last at least fifteen years. Thus, the annualized cost for the purchase of this equipment is about $15,000. In addition, the analysis requires only $2,000 to $3,000 in consumables per year. Contrast this with the current must have "next big thing": STR analysis. The cost of a genetic analyzer, thermocycler, support equipment, operator training, and site remodeling pushes $150,000 with an unknown life span. (Remember all the monies spent on RFLP technologies only to require new PCR technologies within five years followed now by replacement with STR technologies. We have gone through all these in the last ten years! And now the talk is of SNPs and DNA chips.) Given the dubious estimate of a similar 15-year life span, the annualized cost for the purchase of the STR capital equipment is $10,000. In addition, the cost of reagents and kits for STR analyses is about $45,000 a year. Consequently, STR analysis costs roughly three times as much as SEM/EDX analysis. The reality is that SEM/EDX makes economic sense.

Cost is always an important consideration, but SEM/EDX is also client friendly. The analyses are relatively quick and nondestructive, with rapid turnaround, and produce understandable photographic results without numerous charts and graphs requiring extensive explanations. Non-technical persons easily understand the results. With the arrival of computer controlled motorized stages and automation software, once tedious analyses, such as primer residue, have become production work. Our lab can analyze and confirm the results of twelve sample stubs in a twenty-four hour period without a dedicated operator. We have run consecutive samples for days and weeks, stopping only to change filaments. Most cases get an overnight turnaround. None are left for more than a week.

Economics and speed are

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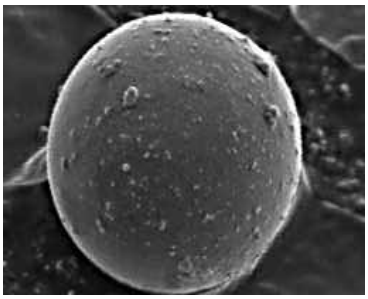
Just Another Tool, Continued



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nothing without reliable results. General SEM/EDX analyses have an impeccable scientific pedigree. SEM/EDX analysis has had and continues to have a distinguished and crucial role in industrial applications and materials research. The science in theory and practice is well established. This body of knowledge transfers well to the forensic sciences. In some cases, SEM/EDX analysis should be the forensic analysis of choice. For example, SEM/EDX primer residue results, though open to some interpretation, are qualitatively better than other elemental analyses such as AA or ICP-AES. The forensic SEM/EDX operator can target specific particles for elemental and image analysis. This data becomes key to an effective courtroom presentation. SEM/EDX analysis has proven itself to be the primer residue detection technique of choice time and time again.

Let's not overlook a primary concern of managers in the past: operator training and experience. The large capital investment and complexity of operation led many lab managers to assign a dedicated certified operator to each instrument. These highly trained technicians and scientists had to work with different kinds of sample media and instrument parameters for difficult analyses. This kind of experience took years to develop. Yet, improvements in instrument design and the routine nature of some forensic applications now require less training, experience, and versatility from operators. We should not underestimate the necessity for expert knowledge in the chemical and physical theory underlying SEM/EDX analysis. However, mastering scientific principles takes less time than achieving application skills. We can teach forensic production methods of the SEM/EDX to someone of average technical aptitude in a few days. Managers, thus, have greater flexibility in human resource decisions. Operator training is not the policy issue it once was.

It is time to overcome the archaic, philistine mythologies surrounding adopting SEM/EDX analysis. Forensic labs need every cost effective, client friendly, and reliable tool at their disposal to help reconstruct the truth. Lab managers must provide this indispensable capability to their scientists. Managers have no excuse not to do so.

Timothy C. Fallon
Crime Lab Manager
Bexar Co. Crime Laboratory

"The criminalist does not attempt identification except as a prelude to his real function - that of individualizing. The real aim of all forensic science is to establish individuality, or to approach it as closely as the present state of the science allows. Criminalistics is the science of individualization.."

- Paul L. Kirk; Taken from the Article "The Ontogeny of Criminalistics" 1963

P-GSR Detection on Clothing and in Automobiles

The methods for detecting primer gunshot residue (P-GSR) on the hands of individuals who have discharged a firearm have been well established in forensic laboratories the world over. Undoubtedly, the most efficient means for identifying the elemental primer components of discharged ammunition cartridges is by automated SEM/EDX analysis. However, little has been documented regarding the identification of P-GSR on the clothing of individuals suspected of discharging a firearm. Furthermore, even less has been documented with regard to the detection of P-GSR in the interior of vehicles.

In numerous criminal cases submitted to the Trace Evidence section, I have successfully identified P-GSR by automated SEM/EDX on the clothing of individuals suspected of discharging a firearm but whose hand samplings were negative for P-GSR particles. Additionally, I have successfully detected P-GSR in the interior of many automobiles involved in shooting incidents. The sampling technique used is the same as that for detecting P-GSR on the hands, with only slight modifications depending on the particular items in question.

The collection apparatus used to perform this analysis are commercially available sample vials with removable caps containing an SEM/EDX aluminum mount (stub) with double-sided adhesive carbon tape affixed. The questioned items are

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P-GSR Detection...Continued

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segmented into areas and the vial containers are appropriately labeled to reflect these sampling areas. The same dabbing technique used for sample collection from the hands is then applied to the segmented areas until the adhesiveness of the carbon tape is lost. A micro-thin coat of carbon is applied to the aluminum sampling stubs prior to analysis in order to improve electrical conductivity and ultimately improve P-GSR particle detection and identification.

The detected P-GSR particles are classified on the basis of morphology and elemental composition. These classifications are described as being either unique or indicative with respect to known manufactured primer ingredients, published articles, cases studies and relative environmental abundance. P-GSR particles identified as containing lead, barium and antimony, or containing barium and antimony are considered unique only to P-GSR, along with the appropriate morphology. Those particles containing lead and barium or lead and antimony are classified as being indicative of P-GSR but also found in other sources.

Positive results obtained by this method of detection and classification are reported as indicating a likely association that the questioned items were in contact with a discharged firearm or in close proximity to a discharged firearm. Obviously, it could be argued that the P-GSR particles were deposited days or perhaps months prior. Although this may be possible, it is not unreasonable to assume that P-GSR can be removed by general laundering of clothes or by repeated contact with automobile interiors. In addition, I am currently unaware of any study that establishes the exact time interval after which P-GSR particles can no longer be detected on the hands or on objects.

In conclusion, P-GSR can be readily detected and identified on clothing and in automobile interiors with the same simple sampling techniques as those used for collecting P-GSR from the hands. The results obtained prove to be a valuable investigative tool in reconstructing the possible events of a firearms related crime and should not be overlooked by forensic scientists.

Michael V. Martinez, MSFS
Trace Evidence Analyst
Bexar Co. Crime Laboratory

"If evidence has been properly gathered and preserved, a mistake in interpretation may always be corrected. If the facts required for a correct interpretation are not preserved, the mistake is irreversible."

-Alan R. Moritz; Quoted in "The Pathology of Trauma" 2nd Edition, Edited by J.K. Mason, pg. 227

GSR Collection Kit Evolution

The West Virginia State Police Forensic Laboratory conducts automated gunshot residue particle analysis and classification on adhesive tape samples for all law enforcement agencies in the state. The CamScan system used today was the first one in the nation (1985) offering this capability.

The Trace Evidence section of the laboratory, after inquiring and assessing other collection kits, designed a three sample kit using ¾ inch carbon stubs labeled right hand, left hand, and face with conventional double sided tape (Scotch 665). An instruction sheet was developed along with a data sheet that asked about the time lapse between the shooting and collection, the subject's occupation, and activities before the collection. A search warrant to obtain these samples is deemed unnecessary because of the exigent circumstances associated with lose of GSR evidence over time and activity as well as the un-intrusive nature of the collection. In 1993, carbon conductive adhesive tapes (c-tape) were introduced into the kits. Using c-tape greatly reduced sample preparation and eliminated the question of cross contamination during any carbon-coating procedure. The brittle nature of the carbon stubs was overcome in 1994 when ¾ inch aluminum stubs were customized to replace them. In 1997, a purer grade of aluminum stock was adopted to remove the stray heavy metal alloy particles occasionally en-

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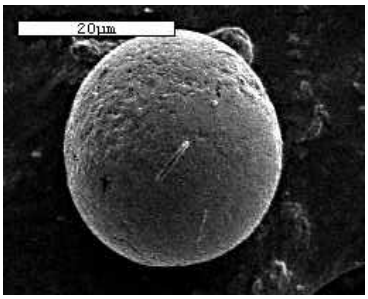
GSR Collection Kit Evolution…Continued



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countered.

The current kit and materials continue to perform efficiently for collection and examination. A sampling tip offered during instruction on collection techniques includes the flexing of the subject's skin folds of the knuckles and palms. By exposing the sheltered surface, which is normally wrinkled, trapped GSR particles may be recovered over longer time periods.

J. R. Giacalone, MSFS
Criminalist VI
West Virgina State
Police Forensic Laboratory

Unusual Firearm and Ammunition

A person came through our office as a suicide by GSW and as with all our GSW cases a gunshot residue kit was collected from the decedent's hands. Our forensic pathologist notified me that the weapon used was a "zip" gun and that he had seen a green halo around the entry site on the clothing so he requested that the GSR kit be analyzed. I analyzed the GSR kit using a SEM/EDS with Oxford Isis automated software and some interesting particles were found on the samples; 1)BaCaSi, 2) BaCaSiSn, 3)BaCaSiPb, 4) PbSnBa several of each type were found. The particles were mostly concentrated on the left hand sample and the majority of their morphologies were spherical. These were curious particles, what conclusion could I come to since some of particles were unique but rare and also in the presence of particles that are not usually considered GSR. I then requested that the "zip" gun, expended cartridge and ammunition be brought to our laboratory for examination (see Fig A and B).

The dimensions of the "zip" gun are ¼ inch wide, inside diameter and 17 inches long when the pipe with the cartridge is inserted into the T fitting shown. The ammunition is .25 caliber and made in the Czech Republic. After scraping the inside of the expended cartridge SEM/EDS analysis revealed particles of a similar elemental composition to that on the hand samples, confirming the hand sample source. Possibly the green halo seen on the clothing at the entry site was due to the diameter of the pipe being greater than that of the bullet, therefore the gun powder was able to get ahead of the bullet.

An interesting case of gunshot residue, revealing another profile of ammunition infrequently seen and an unusual deposition of gun powder on the clothing possibly due to the mechanics of a "zip" gun.

Debra K. Kowal
Senior Criminalist
Los Angeles Co. Coroner's Office

"I think that a particle must have a separate reality independent of the measurements. That is an electron has spin, location and so forth even when it is not being measured. I like to think that the moon is there even if I am not looking at it."

-Albert Einstein

Fig. A
"Zip" Gun with Detonated Cartridge in Place

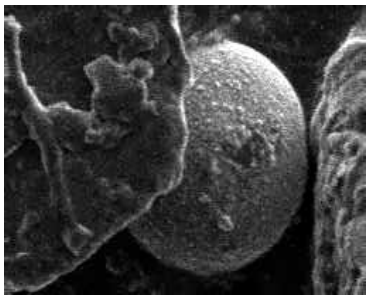
Fig. B
.25 Caliber Ammunition from the Czech Republic

In Theory...

This section of the newsletter is dedicated to short theoretical information specifically related to scientific theory, scientific information, or scientific trivia as it related to P-GSR. Look for this section in the next edition of IAMA

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 that of the editors or other contributors.



I asked a simple question and received the simple answer, "NO". The answer was in response to a question posed to a firearms examiner in our lab. "Have you seen any lead-free ammunition in any case work recently?" To be honest I was not surprised.

There has been a "scare" of sorts regarding SEM/EDX and lead-free ammunition. The question is "Can SEM/EDX detect Primer Gunshot Residue (P-GSR) from lead-free ammunition?" Perhaps. Granted, much work must be done before we can raise our hands in victory or lower our arms in defeat. Obviously, the future of SEM/EDX analysis with regard to lead-free ammunition should be looked at very closely.

Newer automated EDX systems suited for P-GSR do allow a great deal of operator control. We have the ability to define the type of particle analyzed in an automated environment. This includes size, shape, percent composition and classification. Let's not forget that SEM/EDX systems allow for a low limit of detection and the ability to analyze morphology. Unfortunately, very few brands of lead-free ammunitions have been defined under these terms for SEM/EDX and they are continually evolving. This is the problem.

In order to address this concern, our laboratory has decided to undertake a lead-free ammunition study. I intend to define consistencies in the components of commercially available lead-free primers and will be able to define the parameters for lead-free P-GSR analysis. The experience will no doubt raise awareness as well as new questions regarding our analytical approach and interpretation of lead-free P-GSR by SEM/EDX. However, there is no reason to suspect that SEM/EDX will be left completely for a new analytical technique in response to lead-free ammunition.

It has been suggested that identification of organic components from the propellant and primer be used instead of SEM/EDX. I believe that a combination of techniques, identifying for organic composition and elemental composition, will be the most effective way to analyze for lead-free ammunition. But there still is a lot to be said with regard to the identification of morphology and composition of P-GSR particles by SEM/EDX alone.

Much has been said about the use of lead-free ammunition and the simple truth is that I have not seen such a trend in my region. It is understood that there are regional differences in the prevalence of lead-free ammunition. However, when I do start to see lead-free ammunition in case work, I will be able to give the same valuable information to the investigation as I already do for lead based ammunitions.

James D. Garcia, MSFS
Sr. Trace Evidence Analyst
Bexar Co. Crime Lab

"We have a criminal jury system which is superior to any in the world; and its efficiency is only marred by the difficulty of finding twelve men every day who don't know anything and can't read."

-Mark Twain

Acknowledgements

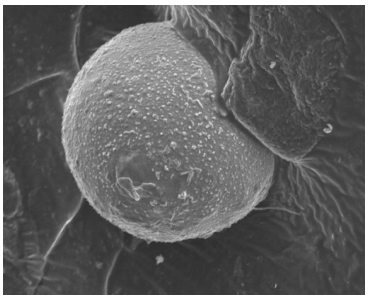
We have added this section of the newsletter to show our appreciation for those individuals who have contributed much of there time and energy in the success of this newsletter.

Jay W. Walker, Jr.: *Tri-Tech,Inc.* Thank you Jay for the great IAMA logo and newsletter publcation.

Henry Hollyday: *Bexar Co. QA/QC Manager.* Thanks Henry for the time you spent in the great editing of the IAMA newsletter.

Articles and Books of Interest...

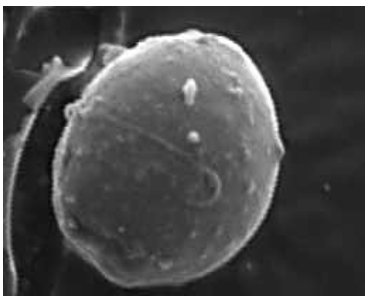
This section of the newsletter is dedicated to articles and books pertaining to P-GSR and SEM/EDX analysis. Further additions will be added in upcoming issues. If you would like to contribute to this section please contact us and we will add them to this section.



1. The Particle Atlas Vol.3, 2nd Ed. Particle No. 512 p.758 Ann Arbor Science Publishers 1973 by W.C. McCrone and J.G. Delly
2. Forensic Science Handbook, Volume II; by Saferstein (selected topics).
3. Instrumental Methods of Analysis, 7th Ed. Chapters 13 "X-RAY METHODS" by Willard, Merritt, Dean, and Settle
4. Forensic Science An Introduction to Criminalistics Chapter 14 "Toolmarks and Firearms" by DeForest, Gaensslen, and Lee
5. Scanning Electron Microscopy; Applications to Materials and Device Science; Chapter # 1, by P. R. Thornton
6. The Scanning Electron Microscope; Part 1, The Instrument; by C. W. Oatley, F. R. S.
7. "Gunshot Residue Analysis - A Review", JFS 1997;42(4):553-570 by Meng, H H et. al.
8. "Survey of Gunshot Residue Analysis in Forensic Science Laboratories" JFS 1990;35(5):1087-1095 by DeGaetano, D. et. al.
9. "Evaluation of instrumental Parameters for Automated Scanning Electron Microscopy/Gunshot Residue Particle Analysis" JFS 1991;36(2):331-342 by Germani, M. S.
10. "Semi-Automatic Detection of Gunshot Residue (GSR) by Scanning Electron Microscopy and Energy Dispersive X-Ray Analysis (SEM/EDX)" Scanning Electron Microscopy 1982;I:107-114 by Gansau and Becker
11. "A Field Kit for Sampling Gunshot Residue Particles" JFS 1982;27(3):671-676 by Tassa, M. et. al.
12. "Suicide Reconstruction by Glue -Lift of Gunshot Residue" JFS 1984;29(3):843-864 by Basu, S. et. al.
13. "Fundamental Studies of Gunshot Residue Deposition" JFS 1997;42(4):571-581 by Basu, S. et. al.
14. "Detection of Gunshot Residues on Hands by Scanning Electron Microscopy" JFS 1977;22(2):279-287 by Andrasko, J. and Maehly, A. C.
15. "Gunshot Residue Particles Formed By Using Different Types of Ammunition in the same Firearm" JFS 1991;36(4):1020-1026 by Zeichner, A. et. al.
16. "The Form of Gunshot Residue is Modified by Target Impact" JFS 1989;34(4):808-822 by Burnett
17. "Analysis of Primer Residue form CCI Blazer® Lead Free Ammunition by Scanning Electron Microscopy/Energy Dispersive X-Ray" JFS 1995;40(1):27-30 by Harris
18. "Identification of Ammunition from Gunshot Residues and Other Cartridge Related Materials- A Preliminary Model Using .22 Caliber Rimfire Ammunition" JFS 1998;43(2):324-328 by Wrobel et. al.
19. "The Identification of Gunshot Residue Particles from Lead-Free Sintox Ammunition" JFS 1994;39 (2):532-536 by Gunaratnam and Himberg
20. "The Contribution of Trace Elements from Smokeless Powder to Post Firing Residues" JFS 1998;43 (1);90-96 by Miyauchi et. al.
21. "American Lead-Free 9mm-p Cartridges" AFTE Journal 1995;27(2):142-149 by Haag
22. "Particle Analysis for the Detection of Gunshot Residue. I: Scanning Electron Microscopy/Energy Dispersive X-Ray Characterization of Hand Deposits from Firing" JFS 1979;24:409-422 by Wolten et. al.
23. "Particle Analysis for the Detection of Gunshot Residue. II: Occupational and Environmental Particles" JFS 1979;24(2):423-430 by Wolten et.al..
24. "Particle Analysis for the Detection of Gunshot Residue. III: The Case Record" JFS 1979;24(4):864-869 by Wolten et.al.
25. "On the Mechanism of Gunshot Residue Particle Formation" JFS 1980;25(3):533-545 by Wolten and Nesbitt

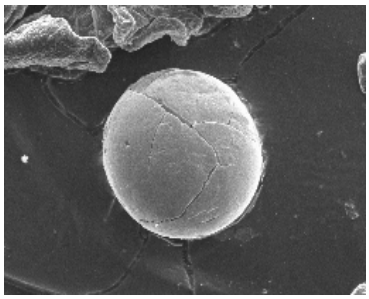
"The great tragedy of science - the slaying of a beautiful hypothesis by an ugly fact."

-Thomas Huxley



In Regard To...

This section of the newsletter is dedicated to letters to the editor(s), responses, comments, questions, clarifications, and/or suggestions to any information published in recently released IAMA newsletters. Please keep your comments in good taste and please be as concise as possible.



In the first IAMA newsletter, we asked if you could provide us with information as to how your laboratory analyzes P-GSR. However, only a few laboratories contacted us with this information. Remember, the purpose for this questionnaire is to collect and distribute the results among us all. The findings may serve to enhance or confirm your existing methods.

- How many P-GSR cases per year.
- Type of SEM in your laboratory.
- Does your SEM have an automated stage?
- EDX manufacture & crystal used. (i.e. beryllium, silicon-lithium, germanium, etc.)
- Microanalysis software and version used.
- Accelerating voltage, bias current, % deadtime & working distance.
- What elemental calibration standard do you use?
- Do you use an imaging standard, positive and negative controls?
- Do you carbon sputter coat your samples?
- Do you set a limit on the minimum number of P-GSR particles con-

You can contact Gary Lawrence at (501) 227-5747 or e-mail glawrence@aristotle.net, James Garcia or Mike Martinez at (210) 335-4115, Fax (210) 335-4101 or e-mail jdgarcia@co.bezar.tx.us or mmartinez@co.bexar.tx.us

"In looking at a drop of water under the microscope, we find there are twice as many H's as O's"

-Funny Science Quotes by Kids

FYI!

A reminder for upcoming events in San Antonio:

SCANNING 2000 will be held at The Sheraton Four Points Riverwalk North Hotel in **San Antonio, TX** on **May 9-12, 2000**. Exhibit set-up days are Monday, May 8 and Tuesday, May 9. Short Courses will be offered beginning Tuesday, May 9 through Friday, May 12. You can find additional Information by visiting http://www.scanning-fams.org/

Southwestern Association for Forensic Scientist (SWAFS) will be held at the beautifully historic Camberley Gunter Hotel in **San Antonio, TX** on **November 5-8, 2001** at a phenomenal rate of $70.00 per night. You can find more information about SWAFS by visiting http://www.cripkit.com/swafs/swafs.html

For additional information or reservation information please contact Gustavo De Leon at the Bexar County Criminal Investigation Laboratory at (210) 335-4148.